Supplementary Material (ESI) for Chemical Communications

Supporting Information

Application of Iron (II)-Bis(isonitrile) Catalysts in the Asymmetric Transfer Hydrogenation of Aromatic and Pyridyl Ketones

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1. General:

All the reactions were carried out under nitrogen atmosphere. All reagents, unless otherwise specified were taken from commercial sources and used without further purification. THF, diethyl ether and toluene were distilled over sodium/benzophenone. Isoproponol was dried over sodium. Pyridyl ketones were synthesized as reported in literature.[1, 2] GC conversions for the reactions were determined relative to decane as an internal standard. ^{1}H NMR, ^{13}C NMR and ^{31}P NMR spectra were obtained on a Bruker Advance 300 (300 MHz) and Bruker Advance 300 (75.5 MHz) spectrometer respectively. Mass analysis was performed using a Varian Mat 311 A. IR spectrums were recorded using an ATI Mattson Genesis Series FT-IR. Chiral HPLC was performed on a Kontron Instruments 325 System (HPLC 335 UV detector, λ = 254 nm, Chiracel OD-H, OJ and AS respectively served as chiral stationary phase.

2. Synthesis of Iron (II)-Bis(isonitrile) complexes 2b:

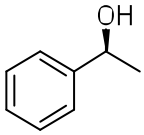
To $FeCl_2.4H_2O$ (1 equiv.) in dry MeOH was added a solution of bis(isonitrile) ligand **1b** (2 equiv.) dissolved in minimum amount of dichloromethane. A reddish solution was obtained that was stirred for 12 h at room temperature upon which no **1b** could be detected by TLC. The solvent was removed under pressure and residue was washed with hexane thrice. Yellowish orange colored solid was obtained after drying under vacuum, yield: 56 %. $[\alpha]^{20}_D$ + 120.8 (c = 1.0, $CHCl_3$). IR (KBr): υ 2963, 2165, 1473, 1439, 1247, 1131, 1020, 988 cm^{-1}. ^1H NMR (CD_2Cl_2, 400 MHz): δ 0.5 – 1.4 (m, 18H), 3.3 – 4.9(m, 6H), 7.4 – 7.70 (m, 3H), 7.72 – 7.9 (m, 1H), 7.92 – 8.19 (m, 1H). ^{31}P NMR (CD_2Cl_2, 162 MHz): δ 20.19 (bs). MS (LSI-MS), m/z (rel. intensity): $[M-Cl]^+$ 843.2. HRMS: calcd. for $C_{40}H_{58}O_6N_4P_2FeCl$ $[M-Cl]^+$: 843.287, found: 843.289.

3. Synthesis of Fe•$(2b)_2(SnCl)_3$:

To a solution of complex **2b** (50 mg, 0.057 mmol) in chloroform (37 mL) was added $SnCl_2$•$2H_2O$ (128 mg, 0.57 mmol). After stirring the mixture for 12 h, the excess of $SnCl_2$•$2H_2O$ was removed by filtration. The solution was concentrated to a small volume and again excess of $SnCl_2.2H_2O$ was removed by filtration. This step was repeated 3 to 4 times. The remaining solvent was then removed under pressure to obtain a yellowish compound. Crystals suitable for X-ray studies were obtained from MeOH/pentane.

4. Typical procedure for the iron catalyzed asymmetric transfer hydrogenation:

In a 10 mL Schlenk tube, the iron complex **2b** (5mol %, 15 mg, 0.0169 mmol), KO*t*Bu (10 equiv., 19 mg, 0.169 mmol) and *iso*-propanol (1.7 mL, for 0.2 M concentration of substrate) were stirred under nitrogen at room temperature for 5 minutes. The substrate (20 equiv.) was added to this mixture and stirred for the time period mentioned in the tables. Conversion and enantiomeric excess of the products were determined by GC using decane as internal standard and chiral HPLC. The products were also identified by ^1H NMR and ^{13}C NMR spectroscopy and the data obtained matches with literature values. Absolute configurations were determined by comparison of the signs of rotation of the isolated products with literature values.

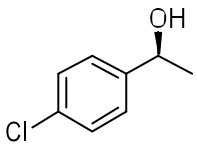


(*S*)-1-phenylethanol[3,4]

The title compound was prepared according to the general procedure as described above in 90% conversion. The product was analyzed by HPLC to

determine the enantiomeric excess 64% ee (OJ: heptane/isopropanol (95/5), 1mL/min, 254 nm); t_r (major) = 10.90 (*S*); t_r (minor) = 12.49 (*R*) min.

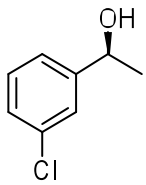
^{1}H-NMR (300 MHz; $CDCl_3$): δ 7.20-7.4 (m, 5H), 4.86 (q, *J* = 6.6, 12.8 Hz, 1H), 2.29 (s, 1H), 1.48 (d, *J* = 6.3 Hz, 3H). ^{13}C NMR ($CDCl_3$, 75 MHz): δ25.17, 70.36, 125.44, 127.46, 128.24, 145.88.



(*S*)-1-(4-chlorophenyl)ethanol[3,4]

94% conversion, 60% ee (OD-H: heptane/isopropanol (99/1), 0.5 mL/min, 254 nm); t_r (minor) = 45.81(*R*), t_r (major) = 52.46 (*S*) min.

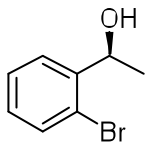
^{1}H-NMR (300 MHz; $CDCl_3$): δ 7.20-7.34 (m, 4H), 4.83 (q, *J* = 6.6, 12.8 Hz, 1H), 2.34 (s, 1H), 1.43 (d, *J* = 6.3 Hz, 3H). ^{13}C NMR ($CDCl_3$, 75 MHz): δ 25.25, 69.69, 126.82, 128.59, 133.02, 144.26.



(*S*)-1-(3-chlorophenyl)ethanol[3,4]

>99% conversion, 67% ee (OJ: heptane/isopropanol (99/1), 1 mL/min, 254 nm); t_r (major) = 19.85 (*S*), t_r (minor) = 23.78 (*R*) min.

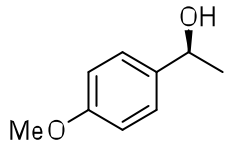
^{1}H-NMR (300 MHz; $CDCl_3$): δ 7.34-7.39 (m, 1H), 7.19-7.30 (m, 3H), 4.84 (q, *J* = 6.6, 12.9 Hz, 1H), 2.16 (s, 1H), 1.46 (d, *J* = 6.4 Hz, 3H). ^{13}C NMR ($CDCl_3$, 75 MHz): δ 25.23, 69.79, 123.56, 125.64, 127.53, 129.80, 134.36, 147.87.



(*S*)-1-(2-bromophenyl)ethanol[3,4]

60% conversion, 67% ee (OD-H: heptane/isopropanol (99/1), 0.5 mL/min, 254 nm); t_r (minor) = 40.82 (*R*), t_r (major) = 45.08 (*S*) min.

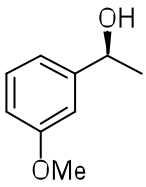
^{1}H-NMR (300 MHz; $CDCl_3$): δ 7.57 (dd, J = 1.6, 7.6 Hz, 1H), 7.50 (dd, J = 1.06, 7.9 Hz, 1H), 7.29-7.38 (m, 1H), 7.08-7.16 (m, 1H), 5,21 (q, *J* = 6.6, 12.8 Hz, 1H), 2.24 (s, 1H), 1.47 (d, *J* = 6.3 Hz, 3H). ^{13}C NMR ($CDCl_3$, 75 MHz): δ 23.59, 69.19, 121.72, 126.69, 127.87, 128.78, 132.66, 144.63.



(*S*)-1-(4-methoxyphenyl)ethanol[3,4]

50% conversion, 58% ee (OD-H: heptane/isopropanol (99/1), 1 mL/min, 254 nm); t_r (minor) = 29.31 (*R*), t_r (major) = 33.09 (*S*) min.

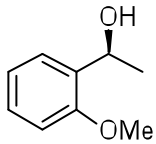
^{1}H-NMR (300 MHz; $CDCl_3$): δ 7.25-7.32 (m, 2H), 6.84-6.91 (m, 2H), 4.83 (q, *J* = 6.6, 12.8 Hz, 1H), 3.79 (s, 3H), 2.07 (s, 1H), 1.46 (d, *J* = 6.3 Hz, 3H). ^{13}C NMR ($CDCl_3$, 75 MHz): δ 25.04, 55.30, 69.93, 113.84, 126.69, 138.07, 158.95.



(*S*)-1-(3-methoxyphenyl)ethanol[3,4]

93% conversion, 54% ee (OD-H: heptane/isopropanol (99/1), 1 mL/min, 254 nm); t_r (minor) = 31.08 (*R*), t_r (major) = 37.42 (*S*) min.

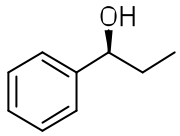
^{1}H-NMR (300 MHz; $CDCl_3$): δ 7.22-7.30 (m, 1H), 6.90-6.96 (m, 2H), 6.80 (ddd, *J* = 8.20, 2.54, 1.06 Hz, 1H), 4.84 (q, *J* = 6.5, 12.8 Hz, 1H), 3.80 (s, 3H), 2.20 (bs, 1H), 1.47 (d, *J* = 6.3 Hz, 3H). ^{13}C NMR ($CDCl_3$, 75 MHz): δ 25.16, 55.22, 70.29, 110.91, 112.86, 117.72, 129.53, 147.65, 159.75.



(*S*)-1-(2-methoxyphenyl)ethanol[3,4]

56% conversion, 52% ee (OD-H: heptane/isopropanol (99/1), 1 mL/min, 254 nm); t_r (major) = 19.28 (*S*), t_r (minor) = 21.32 (*R*) min.

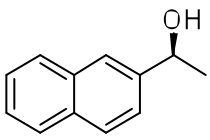
^{1}H-NMR (300 MHz; $CDCl_3$): δ 7.34 (dd, *J* = 1.65, 7.4 Hz, 1H), 7.21-7.30 (m, 1H), 6.93-7.01 (m, 1H), 6.85-6.92 (dd, *J* = 0.8, 8.2 1H), 5.09 (q, *J* = 6.6, 12.9 Hz, 1H), 3.86 (s, 3H), 2.7 (bs, 1H), 1.51 (d, *J* = 6.6 Hz, 3H). ^{13}C NMR ($CDCl_3$, 75 MHz): δ 22.88, 55.27, 66.51, 110.43, 120.81, 126.11, 128.30, 133.46, 156.55.



(*S*)-1-phenylpropan-1-ol[3,4]

73% conversion, 64% ee (OD-H: heptane/isopropanol (99/1), 1 mL/min, 254 nm); t_r (minor) = 16.39 (*R*), t_r (major) = 19.06 (*S*) min.

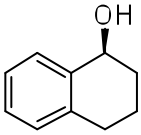
^{1}H-NMR (300 MHz; $CDCl_3$): δ 7.22-7.40 (m, 5H), 4.57 (t, *J* = 6.6 Hz, 1H), 2.11 (s, 1H), 1.65-1.91 (m, 2H), 0.92 (t, *J* = 7.4 Hz, 3H). ^{13}C NMR ($CDCl_3$, 75 MHz): δ 10.18, 31.89, 76.01, 126.02, 127.49, 128.41, 144.64.



(*S*)-1-(naphthalen-2-yl)ethanol[3,4]

84% conversion, 64% ee (OJ: heptane/isopropanol (90/10), 1 mL/min, 254 nm); t_r (major) = 14.34 (*S*), t_r (minor) = 18.43 (*R*) min.

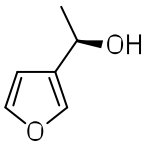
^{1}H-NMR (300 MHz; $CDCl_3$): δ 7.78-7.88 (m, 4H), 7.43-7.54 (m, 3H), 5.06 (q, *J* = 6.3, 12.9 Hz, 1H), 2.04 (s, 1H), 1.58 (d, *J* = 6.3 Hz, 3H). ^{13}C NMR ($CDCl_3$, 75 MHz): δ 25.17, 70.55, 123.83, 123.85, 125.83, 126.18, 127.71, 127.96, 128.34, 132.94, 133.34, 143.21.



(*S*)-1,2,3,4-tetrahydronaphthalen-1-ol[4]

62% conversion, 46% ee (OD-H: heptane/isopropanol (99/1), 1 mL/min, 254 nm); t_r (minor) = 15.91 (*R*), t_r (major) = 17.33 (*S*) min.

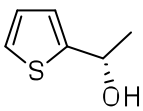
^{1}H-NMR (300 MHz; $CDCl_3$): δ 7.41-7.46 (m, 1H), 7.18-7.24 (m, 2H), 7.08-7.15 (m, 1H), 4.77 (t, *J* = 5.2 Hz, 1H), 2.65-2.9 (m, 2H), 1.7-2.09 (m, 5H). ^{13}C NMR ($CDCl_3$, 75 MHz): δ 18.83, 29.27, 32.28, 68.15, 126.19, 127.59, 128.69, 129.03, 137.14, 138.82.



(*R*)-1-(furan-3-yl)ethanol[5]

>99% conversion, 30% ee (OJ: heptane/isopropanol (99/1), 1 mL/min, 254 nm); t_r (minor) = 21.05 (*S*), t_r (major) = 23.41 (*R*) min.

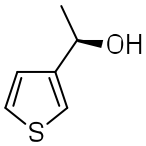
^{1}H-NMR (300 MHz; $CDCl_3$): δ 7.35-7.40 (m, 1H), 6.32 (dd, 1H, *J* = 3.2 and 1.2 Hz), 6.22 (d, 1H, *J* = 3.2 Hz), 4.88 (q, 1H, *J* = 6.4 Hz), 2.30 (br s, 1H), 1.54 (d, 3H, *J* = 6.8 Hz); ^{13}C NMR (75 MHz, $CDCl_3$): δ = 157.6, 141.8, 110.1, 105.0, 63.5, 21.2.



(*S*)-1-(2-Thienyl)ethanol[5]

70% conversion, 53% ee (OJ: heptane/isopropanol (99/1), 1 mL/min, 254 nm); t_r (major) = 25.72 (*S*), t_r (minor) = 33.38 (*R*) min.

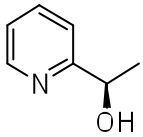
^{1}H-NMR (300 MHz; $CDCl_3$): δ 7.19-7.24 (m, 1H), 6.91-6.99 (m, 2H), 5.07 (q, *J* = 6.3, 12.9 Hz, 1H), 2.94 (s, 1H), 1.56 (d, *J* = 6.4 Hz, 3H). ^{13}C NMR ($CDCl_3$, 75 MHz): δ 25.25, 66.11, 120.22, 124.37, 126.66, 149.99.



(*R*)-1-(3-Thienyl)ethanol[5]

36% conversion, 62% ee (OJ: heptane/isopropanol (99/1), 1 mL/min, 254 nm); t_r (major) = 25.08 (*R*), t_r (minor) = 30.51 (*S*) min.

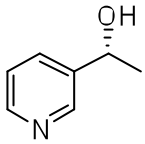
^{1}H-NMR (300 MHz; $CDCl_3$): δ 7.29-7.31 (m, 1H), 7.18-7.20 (m, 1H), 7.09-7.10 (m, 1H), 4.97 (q, *J* = 6.8 Hz, 1H), 1.89 (s, 1H), 1.52 (d, *J* = 6.8 Hz), ^{13}C NMR ($CDCl_3$, 75 MHz): δ 147.4, 126.1, 125.7, 120.2, 66.5, 24.5.



(*R*)-1-(pyridin-2-yl)ethanol[6]

85% conversion, 41% ee (OD-H: heptane/isopropanol (99/1), 1 mL/min, 254 nm); t_r (major) = 18.66 (*R*), t_r (minor) = 21.61 (*S*) min.

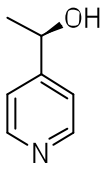
^{1}H-NMR (300 MHz; $CDCl_3$): δ 8.54 (d, *J* = 5.3 Hz, 1H), 7.68-7.72 (m, 1H), 7.28 (d, *J* = 9.3 Hz, 1H), 4.90 (q, *J* = 6.4 Hz, 1H), 4.34 (s, 1H), 1.5 (d, *J* = 6.4 Hz, 3H). ^{13}C NMR ($CDCl_3$, 75 MHz): δ 24.19, 69.01, 119.82, 122.22, 136.87, 148.09, 163.25.



(*R*)-1-(pyridin-3-yl)ethanol[6]

95% conversion, 61% ee (OJ: heptane/isopropanol (90/10), 1 mL/min, 254 nm); t_r (major) = 7.21 (*R*), t_r (minor) = 9.33 (*S*) min.

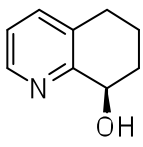
^{1}H-NMR (300 MHz; $CDCl_3$): δ 8.38 (d, *J* = 2.4 Hz, 1H), 8.29 (dd, *J* = 1.3, 4.6 Hz, 1H), 7.64-7.75 (m, 1H), 7.15-7.24 (m, 1H), 4.85 (q, *J* = 6.4 Hz, 1H), 4.6 (bs, 1H), 1.44 (d, *J* = 6.4 Hz, 3H). ^{13}C NMR ($CDCl_3$, 75 MHz): δ 25.19, 67.45, 123.58, 133.60, 141.84, 146.99, 147.96.



(*S*)-1-(pyridin-4-yl)ethanol[6]

99% conversion, 55% ee (AS-H: heptane/isopropanol (90/10), 1 mL/min, 254 nm); t_r (minor) = 17.74 (*R*), t_r (major) = 31.69 (*S*) min.

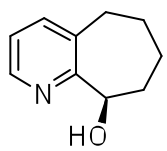
^{1}H-NMR (300 MHz; $CDCl_3$): δ 8.42 (d, *J* = 4.5 Hz, 1H), 7.27 (d, *J* = 4.5 Hz, 2H), 4.86 (q, *J* = 6.4 Hz, 1H), 4.13 (s, 1H), 1.46 (d, *J* = 6.4 Hz, 3H). ^{13}C NMR ($CDCl_3$, 75 MHz): δ 25.10, 68.53, 120.56, 149.40, 155.53.



(*R*)-8-hydroxy-5, 6, 7, 8-tetrahydroquinoline[1]

80% yield, 91% ee (OD-H: heptane/isopropanol (99/1), 1 mL/min, 254 nm); t_r (major) = 15.94 (*R*), t_r (minor) = 22.73 (*S*) min.

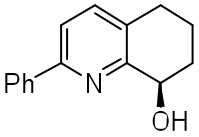
^{1}H-NMR (300 MHz; $CDCl_3$): δ 8.30-8.29 (m, 1H), 7.31 (dd, *J* = 7.7, 0.6 Hz, 1H), 7.01 (dd, *J* = 7.7, 4.8 Hz, 1H), 5.15 (s, 1H), 4.68 (dd, *J* = 7.2, 5.4 Hz, 1H), 2.79-2.60 (m, 2H), 2.16-2.06 (m, 1H), 1.98-1.65 (m, 3H). ^{13}C NMR ($CDCl_3$, 75 MHz): δ 158.03, 146.46, 137.05, 131.90, 122.19, 67.85, 31.03, 28.44, 18.84.



(*R*)-9-hydroxy-6, 7, 8, 9-tetrahydro-5H-cycloheptapyridine[1]

83% conversion, 83% ee (AS-H: heptane/isopropanol (99/1), 0.5 mL/min, 254 nm); t_r (major) = 21.46 (*R*), t_r (minor) = 27.12 (*S*) min.

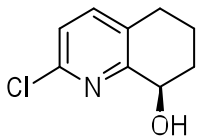
^{1}H NMR ($CDCl_3$, 300 MHz): δ 1.03 – 1.20(m, 1H), 1.22 – 1.40 (m, 1H), 1.65 – 1.82 (m, 1H), 1.83 – 2.03 (m, 2H), 2.06 – 2.19 (m, 1H), 2.53 – 2.73(m, 2H), 4.66 (dd, 1H, *J* = 2.4, 11 Hz), 5.81 (s, 1H), 7.02 (dd, 1H, *J* = 4.9, 7.5 Hz), 7.30 – 7.37 (m, 1H), 8.24 (dd, 1H, *J* = 1.5, 4.9 Hz). ^{13}C NMR ($CDCl_3$, 75 MHz): δ 27.03, 29.01, 34.32, 36.31, 72.17, 122.08, 135.67, 137.20, 144.35, 160.93.



(*R*)-8-hydroxy-2-phenyl-5, 6, 7, 8-tetrahydroquinoline[1]

98% yield, 72% ee (OD-H: heptane/isopropanol (99/1), 1 mL/min, 254 nm); t_r (minor) = 12.54 (*S*), t_r (major) = 31.41 (*R*) min.

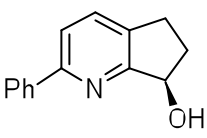
^{1}H NMR ($CDCl_3$, 300 MHz): δ 1.72 – 1.93(m, 2H), 1.95 – 2.11(m, 1H), 2.30 – 2.45 (m, 1H), 2.76 – 2.94 (m, 2H), 4.37 (s, 1H), 4.73 (dd, 1H, *J* = 5.3, 9.2 Hz), 7.37 – 7.52 (m, 4H), 7.57 (d, 1H, *J* = 8.0 Hz), 7.97 – 8.03 (m, 2H). ^{13}C NMR ($CDCl_3$, 75 MHz): δ 19.72, 28.02, 30.67, 69.20, 119.19, 126.69, 128.74, 128.88, 137.76, 138.92, 154.22, 157.65.



(*R*)-8-hydroxy-2-chloro-5, 6, 7, 8-tetrahydro-2-quionoline[1b,2]

93% yield, 84% ee (OJ: heptane/isopropanol (99/1), 0.5 mL/min, 254 nm); t_r (major) = 25.48 (*R*), t_r (minor) = 30.0 (*S*) min.

^{1}H NMR ($CDCl_3$, 300 MHz): δ 1.6 – 1.84 (m, 2H), 1.85 – 2.01 (m, 1H), 2.05 – 2.2 (m, 1H), 2.57 – 2.8 (m, 2H), 3.8 (s, 1H), 4.62 (dd, 1H, *J* = 4.0, 16.3 Hz), 7.05 (d, 1H, *J* = 8.0 Hz), 7.31 (d, 1H, *J* = 8.0 Hz). ^{13}C NMR ($CDCl_3$, 75 MHz): δ 18.03, 16.8, 29.3, 67.4, 75.7, 121.9, 129.6, 138.9, 147.3, 157.6.



(*R*)-2-phenyl-7-hydroxy- 6, 7-dihydro-5H-cyclopentapyridine[1]

89% yield, 52% ee (OD-H: heptane/isopropanol (99/1), 1 mL/min, 254 nm); t_r (major) = 32.41 (*R*), t_r (minor) = 57.29 (*S*) min.

^{1}H-NMR (300 MHz; $CDCl_3$): δ 7.94-7.90 (m, 2H), 7.54 (q, *J* = 7.6 Hz, 2H), 7.46-7.35 (m, 3H), 5.25 (t, *J* = 6.7 Hz, 1H), 4.80-4.72 (m, 1H), 3.04-2.94 (m, 1H), 2.82-2.71 (m, 1H), 2.50

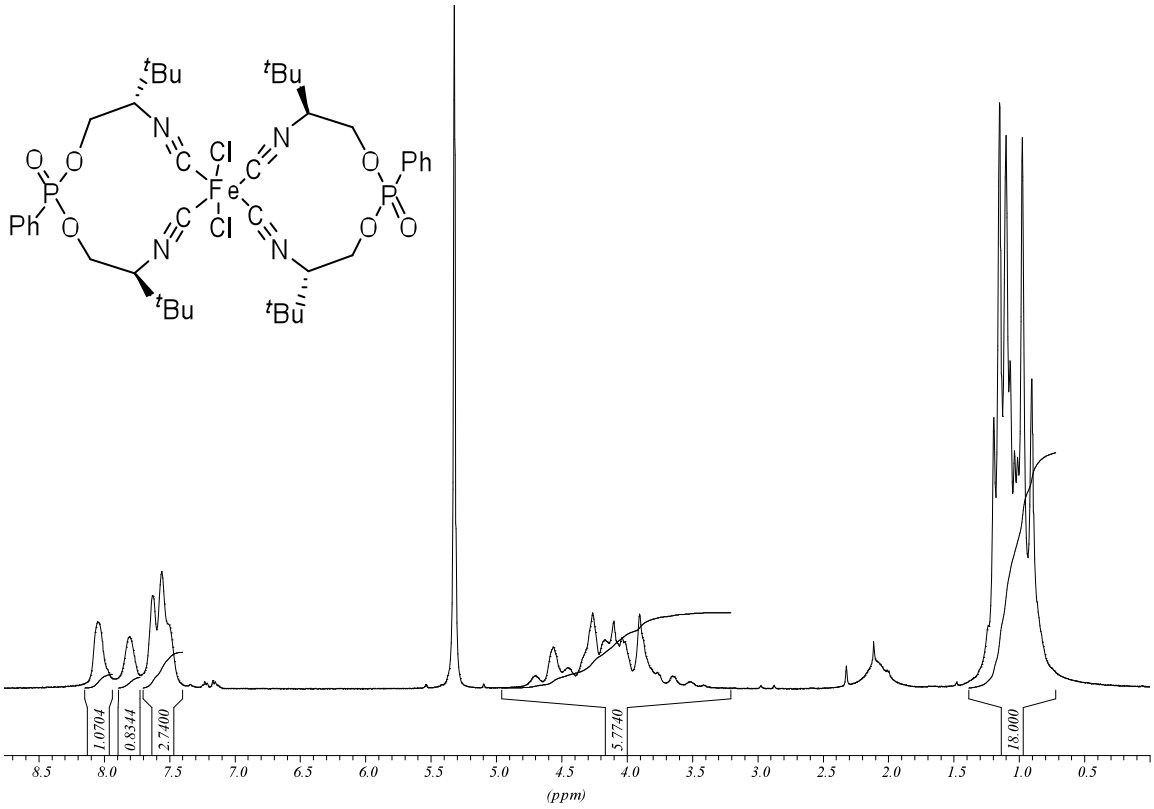
(m, 1H), 2.05 (m, 1H). ^{13}C-NMR (75.5 MHz; $CDCl_3$): δ 164.96, 156.51, 139.48, 134.98, 133.83, 128.67, 127.12, 120.13, 74.58, 33.13, 27.29.

5. References:

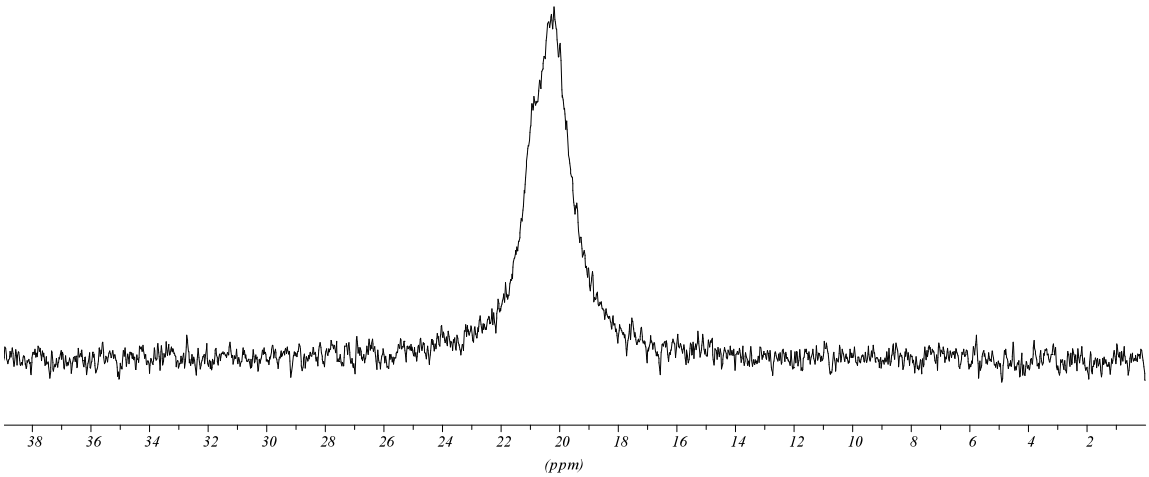
1) a) S. Kaiser, S. P. Smidt and A. Pfaltz, *Angew. Chem. Int. Ed*., 2006, **45**, 5194. b) Y. Xie, H. Huang, W. Mo, X. Fan, Z. Shen, N. Sun, B. Hu and X. Hu, *Tetrahedron: Asymmetry*, 2009, **20**, 1425.
2) A. I. Meyers and G. Garcia-Munoz, *J. Org. Chem.*, 1964, **29**, 1435.
3) a) M. L. Kantam, J. Yadav, S. Laha, P. Srinivas, B. Sreedhar, and F. Figueras, *J. Org. Chem.* 2009, **74**, 4608. b) K. Junge, B. Wendt, D. Addis, S. Zhou, S. Das, and M. Beller, *Chem. Eur. J*., 2010, **16**, 68.
4) a) S. Zeror , J. Collin , J. Fiaud and L. A. Zouioueche, *Journal of Molecular Catalysis A*: *Chemical,* 2006, **256**, 85. b) J. S. Yadav, B. V. S. Reddy, C. Sreelakshmi and A. B. Rao, *Synthesis*, 2009, **11**, 1881.
5) a) X. Zhang, Y. Wu, F. Yu, F. Wu, J. Wu and A. S. C. Chan, *Chem. Eur. J.,* 2009, **15**, 5888. b) T. Ohkuma, M. Koizumi, M. Yoshida and R. Noyori, *Org. Lett.*, 2000, **2**, 1749.
6) K. Uwai, N. Konno, S. Kitamura, S. Ohta and M. Takeshita, *Chirality*, 2005, **17**, 494.

6. ^{1}H NMR, ^{13}C NMR and ^{31} P NMR Spectra:

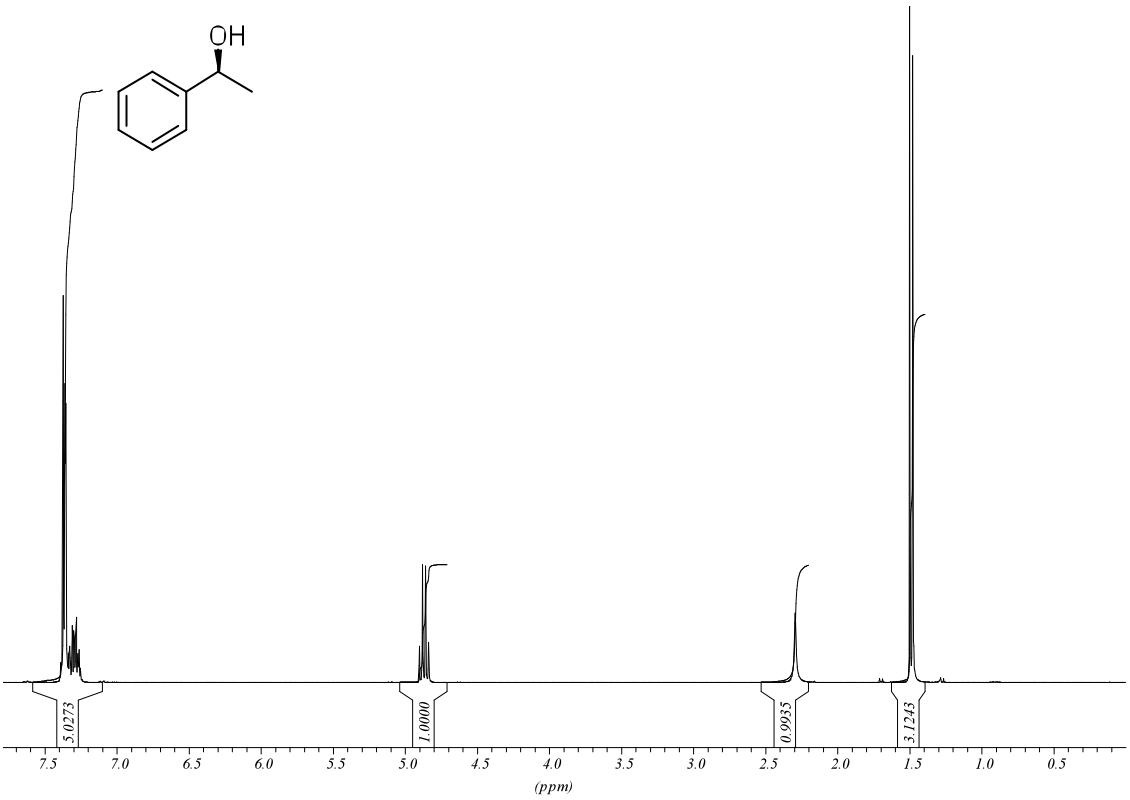
Complex 2b: ^{1}H NMR

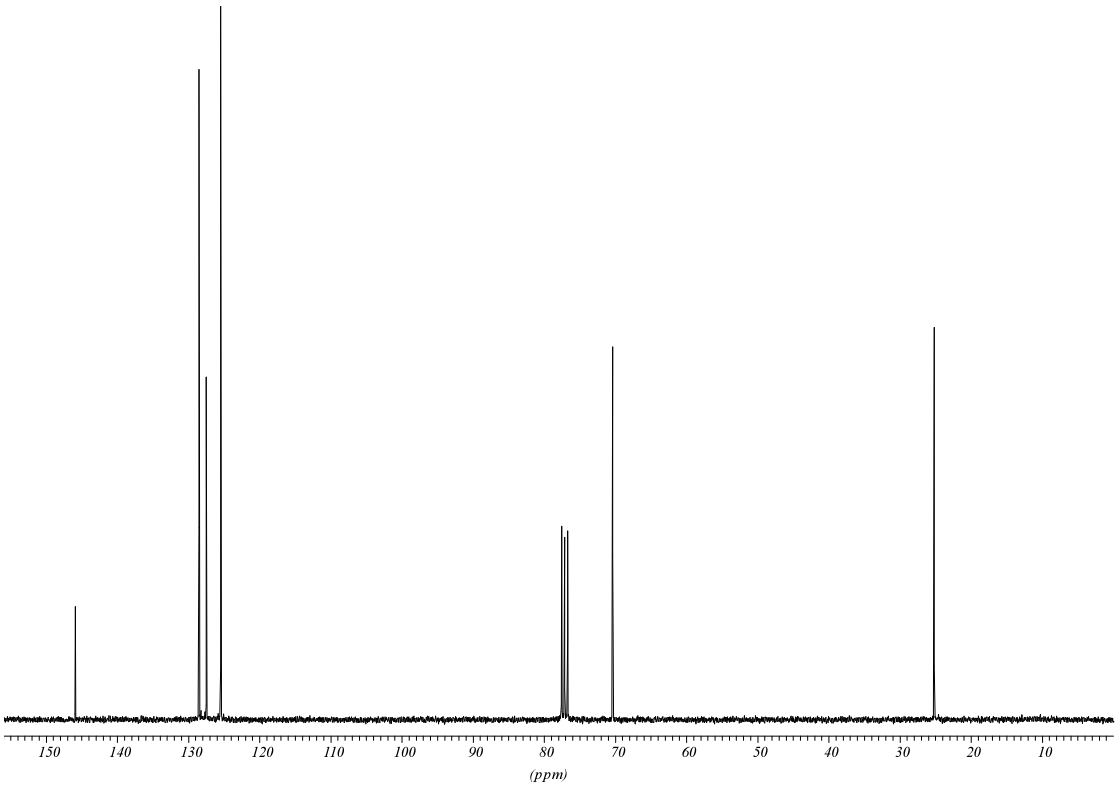


^{31} P NMR

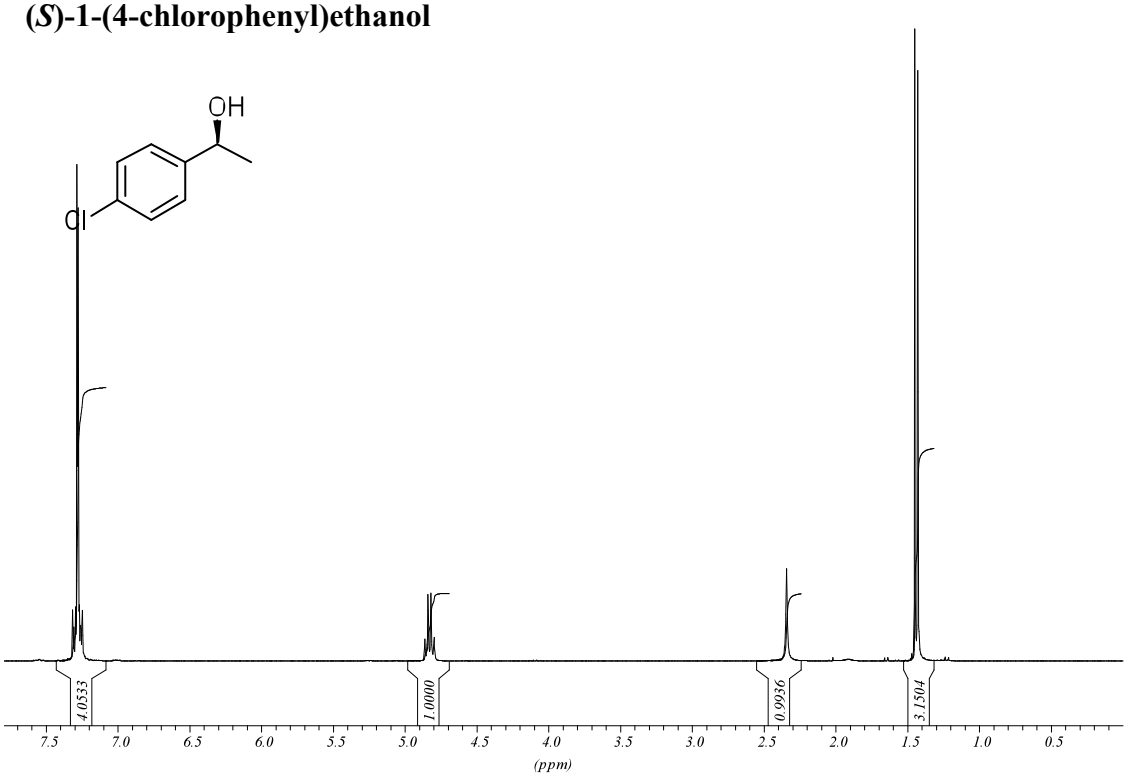


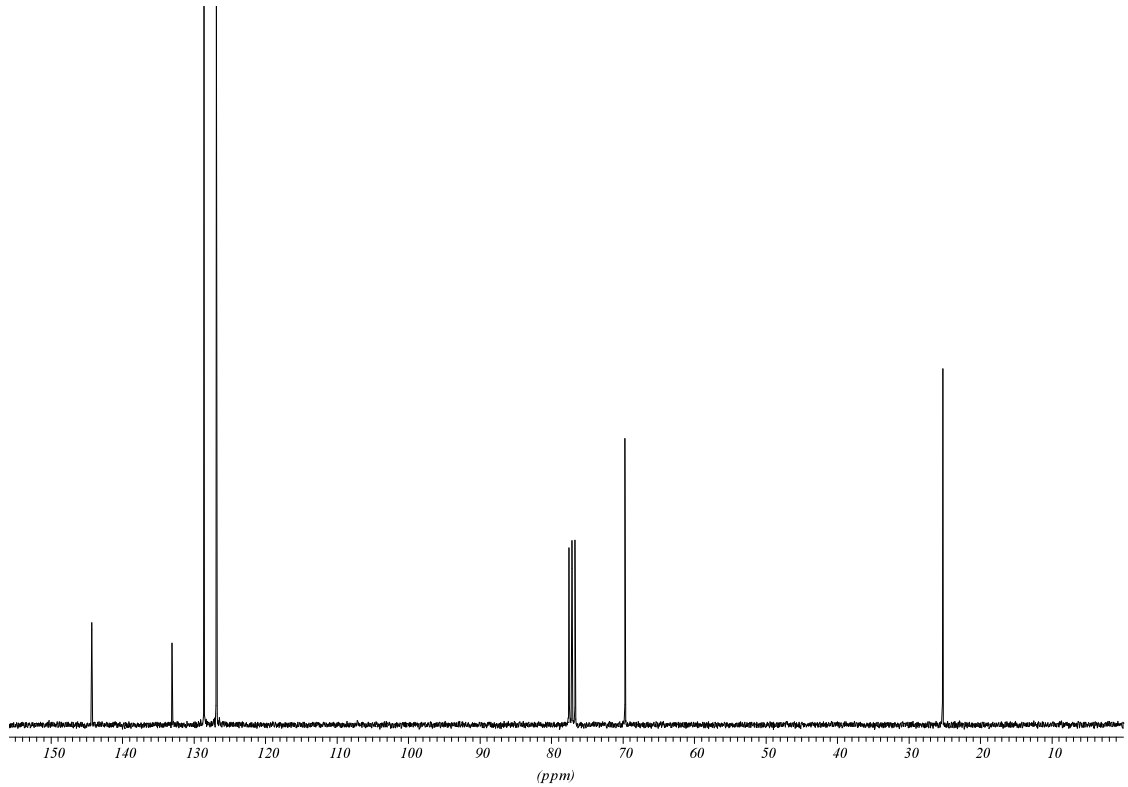
(*S*)-1-phenylethanol



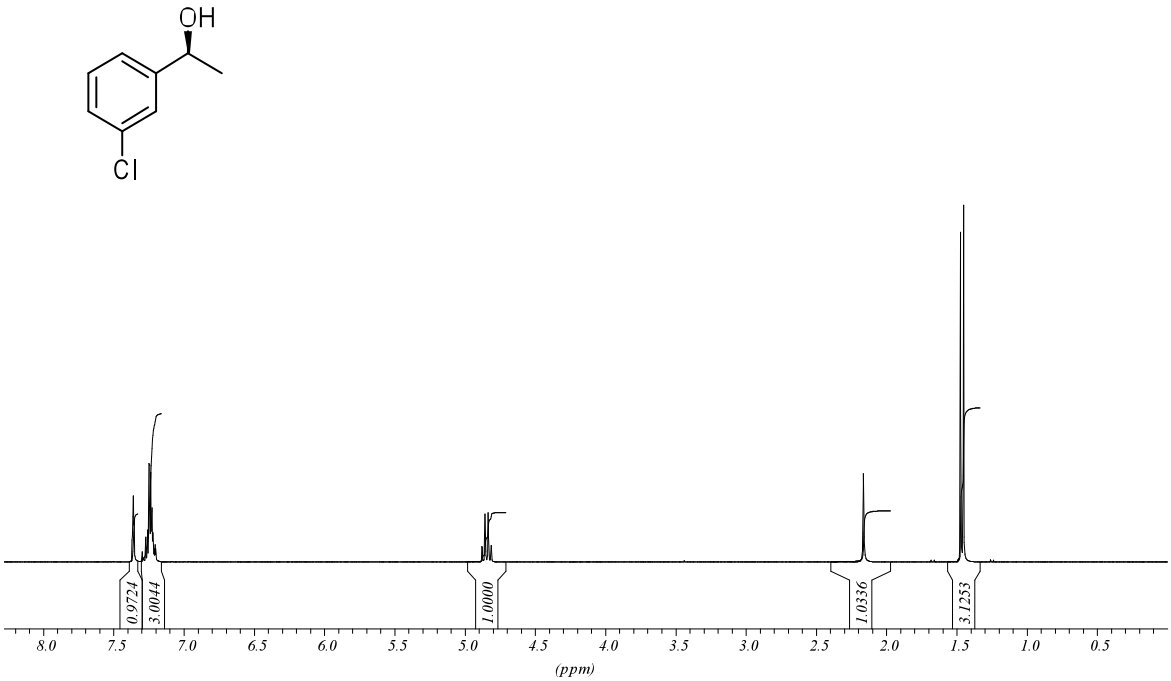


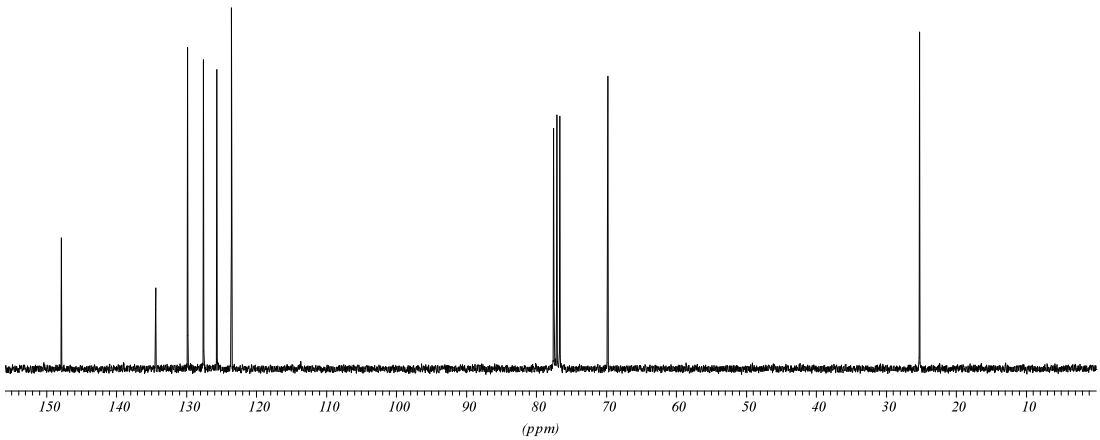
(*S*)-1-(4-chlorophenyl)ethanol



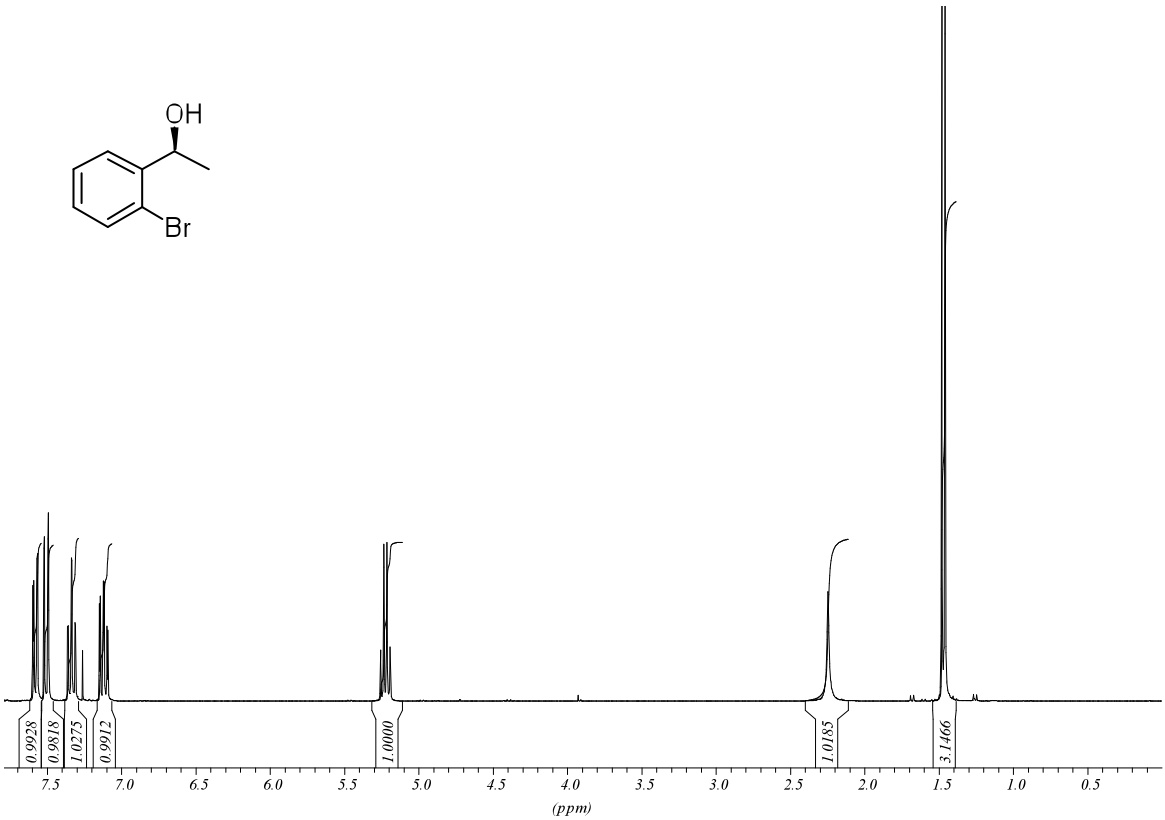


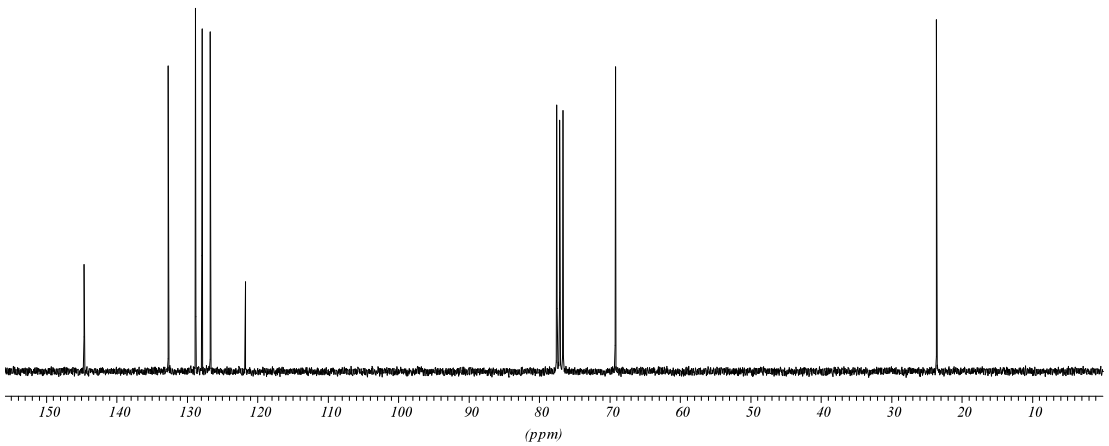
(*S*)-1-(3-chlorophenyl)ethanol



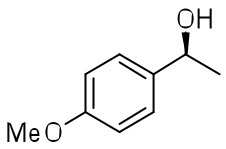


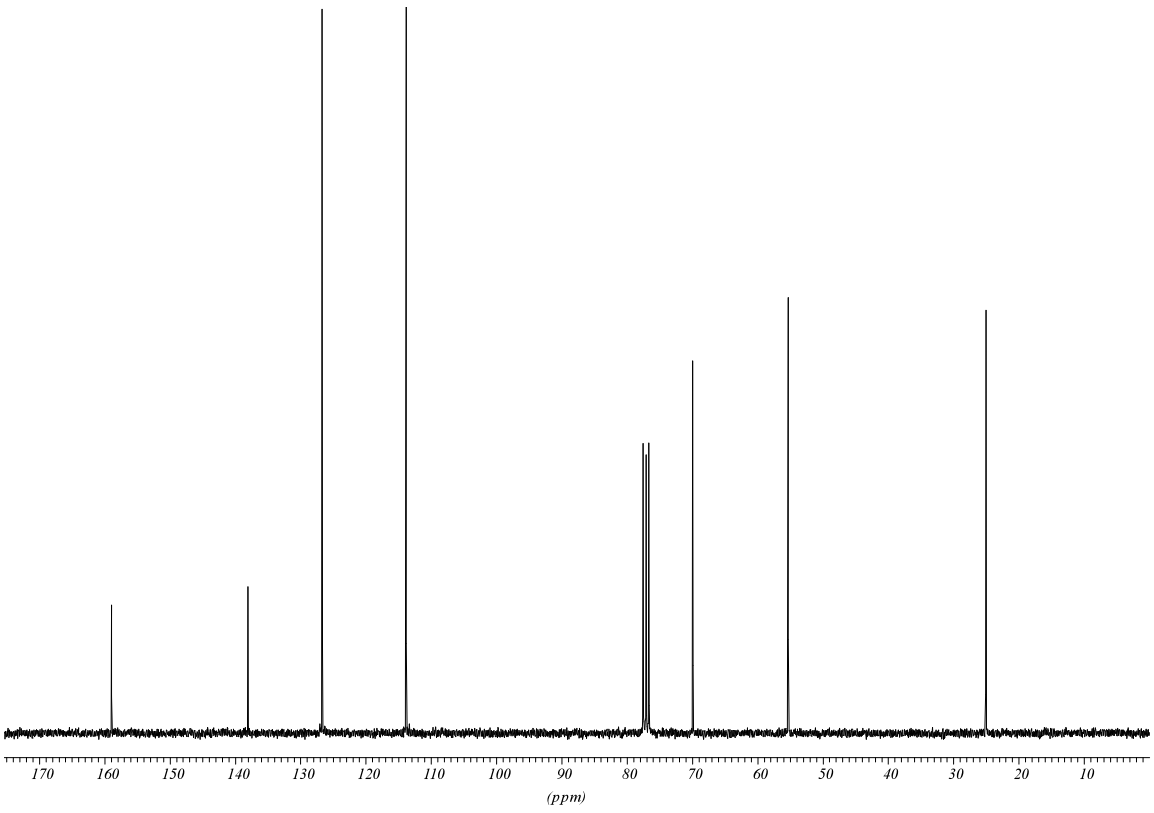
(*S*)-1-(2-bromophenyl)ethanol



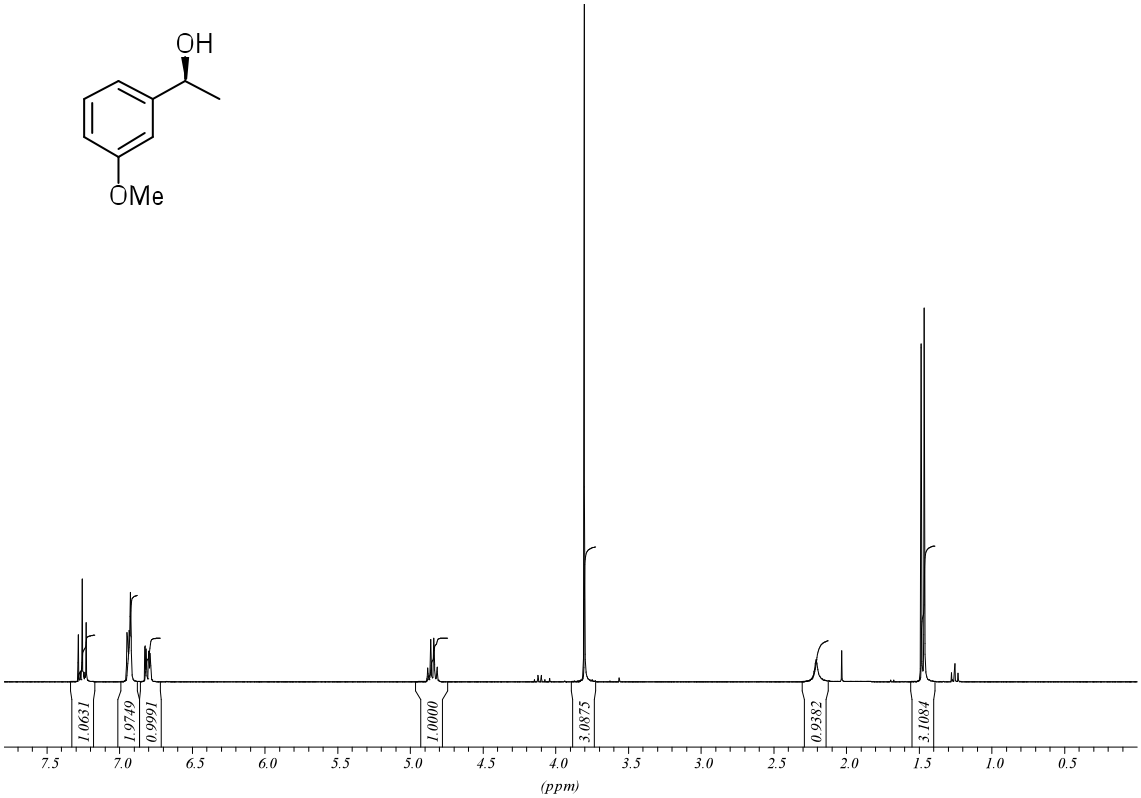


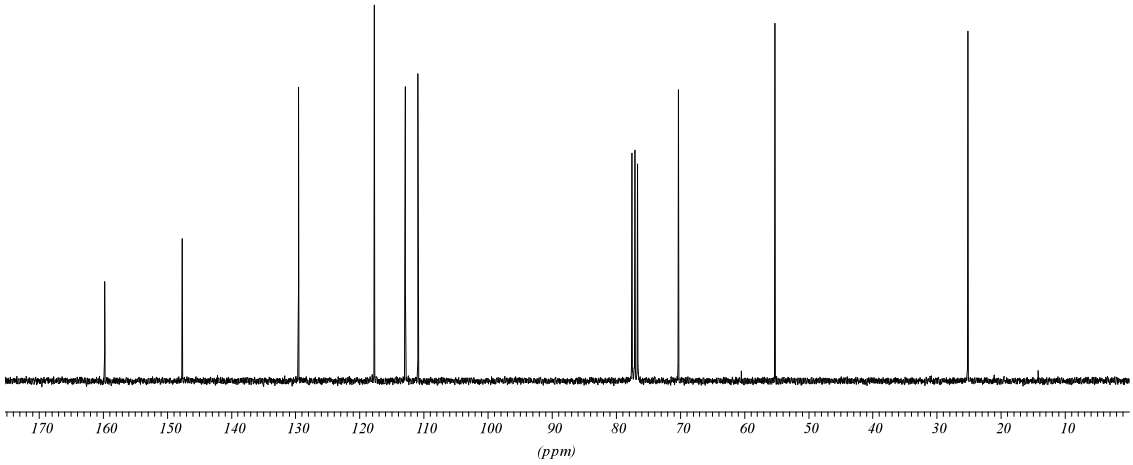
(*S*)-1-(4-methoxyphenyl)ethanol



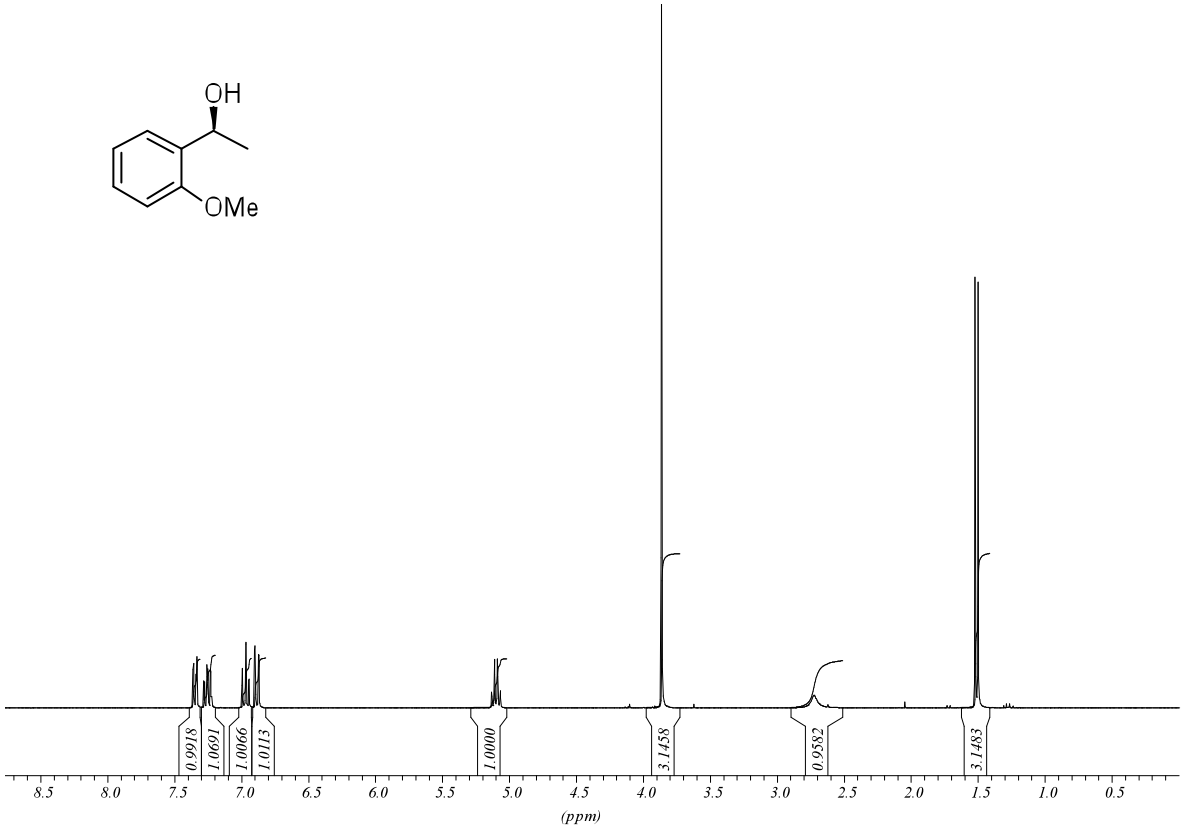


(*S*)-1-(3-methoxyphenyl)ethanol





(*S*)-1-(2-methoxyphenyl)ethanol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OMe
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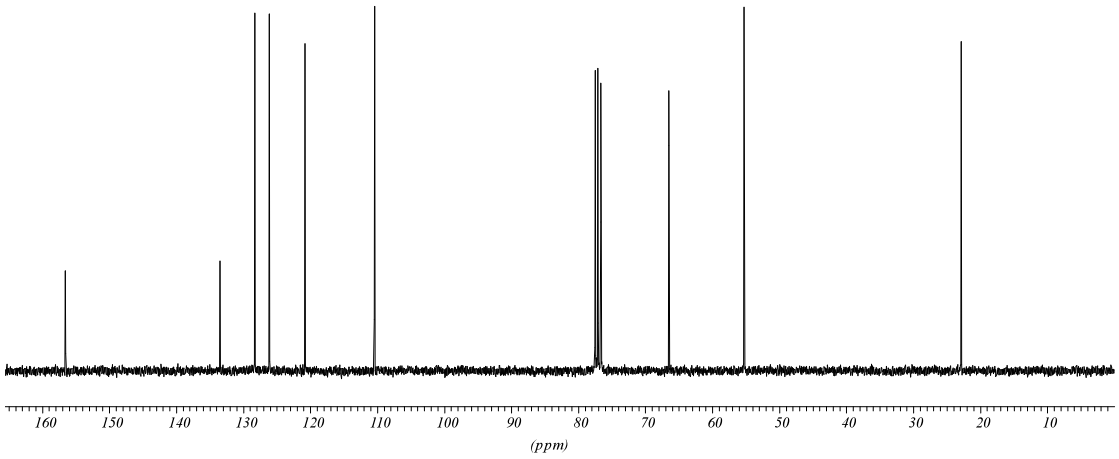
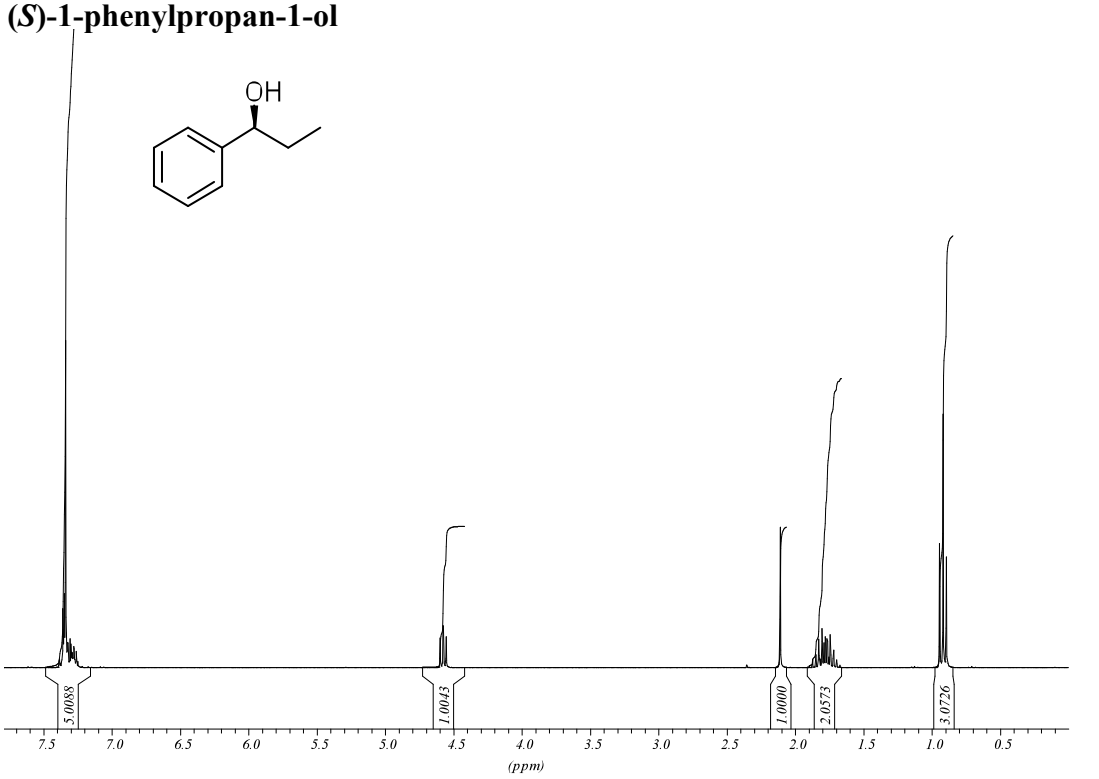
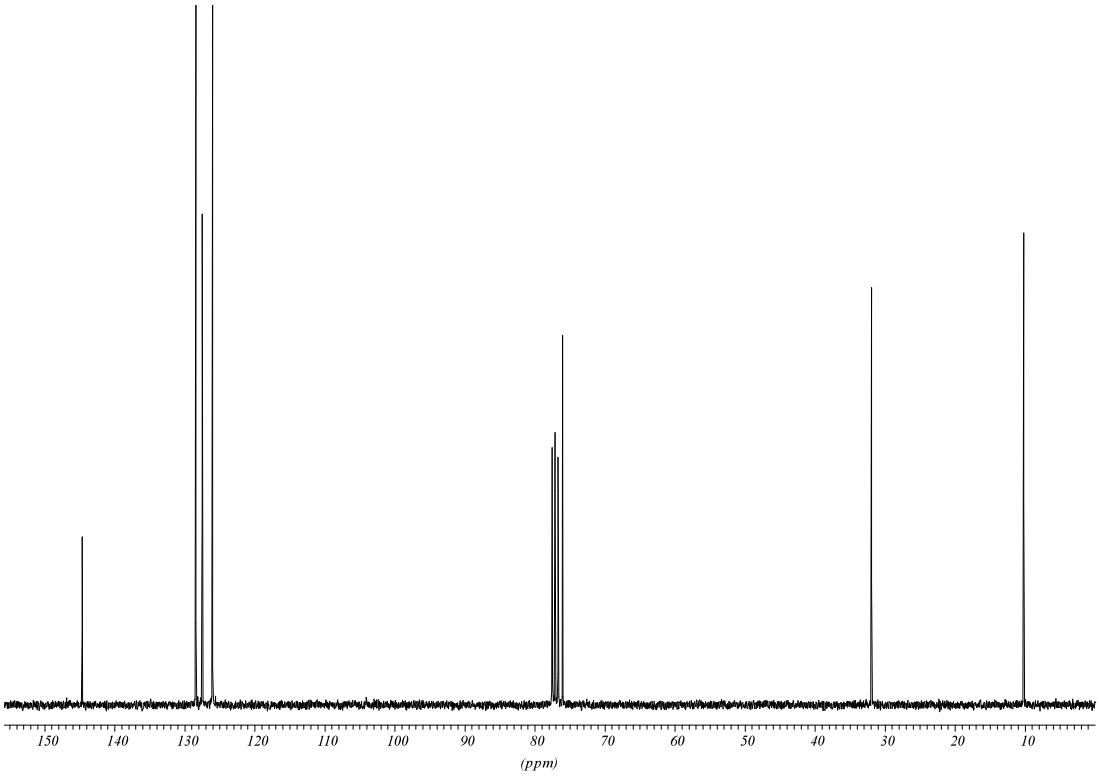
1.0691
1.0066
1.0113
1.0000
3.1458
0.9582
3.1483
8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5
(ppm)

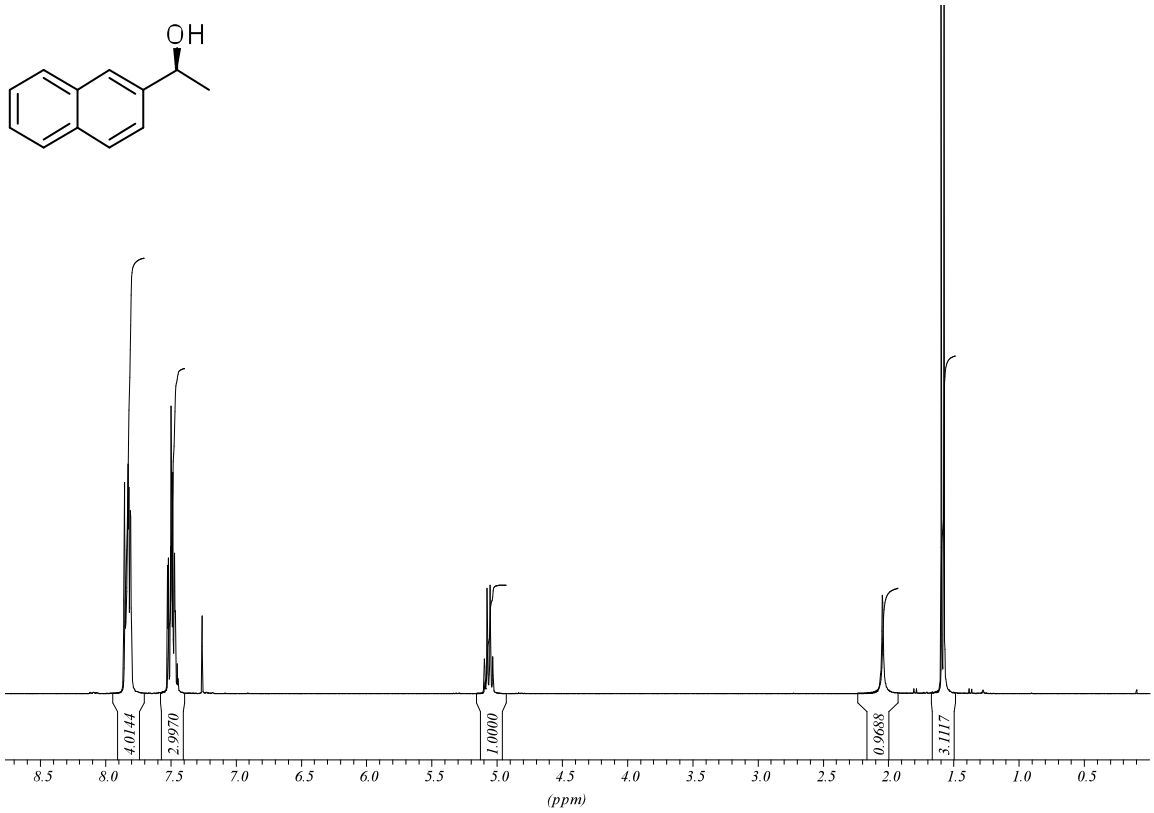

160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10
(ppm)

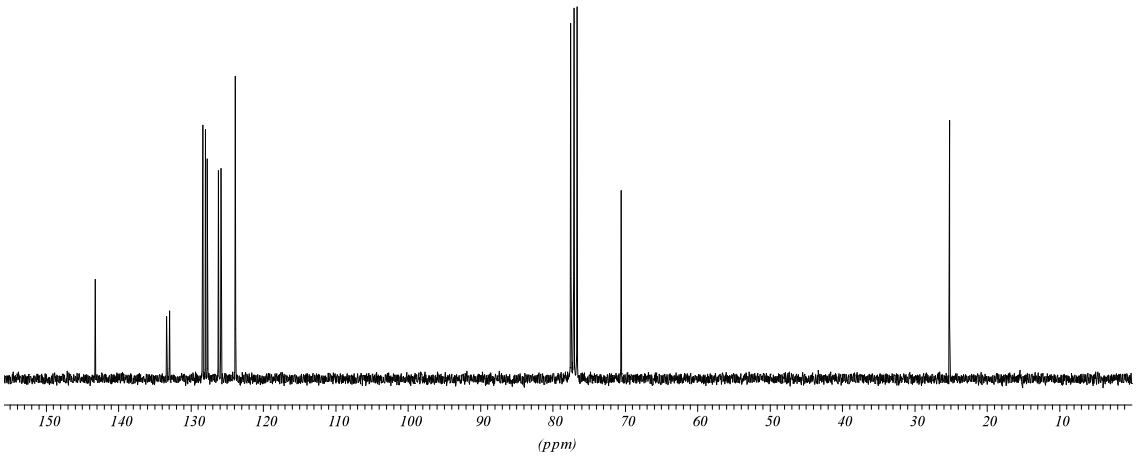
(*S*)-1-phenylpropan-1-ol



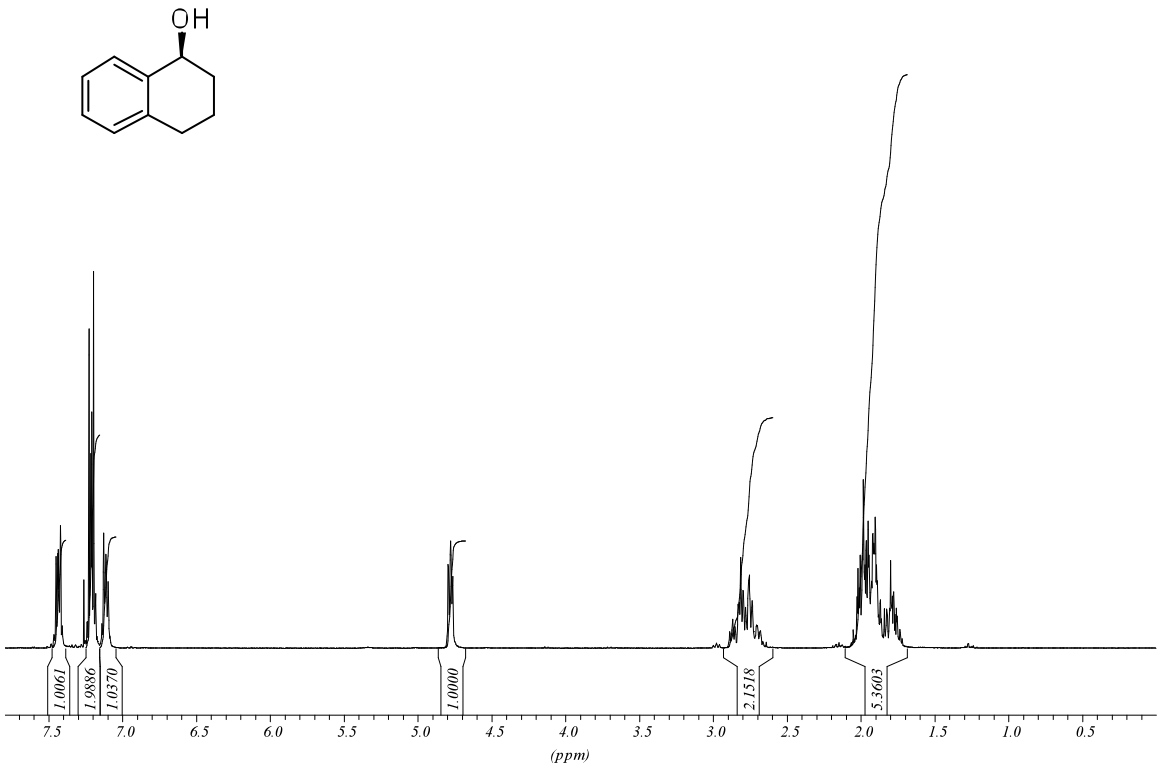


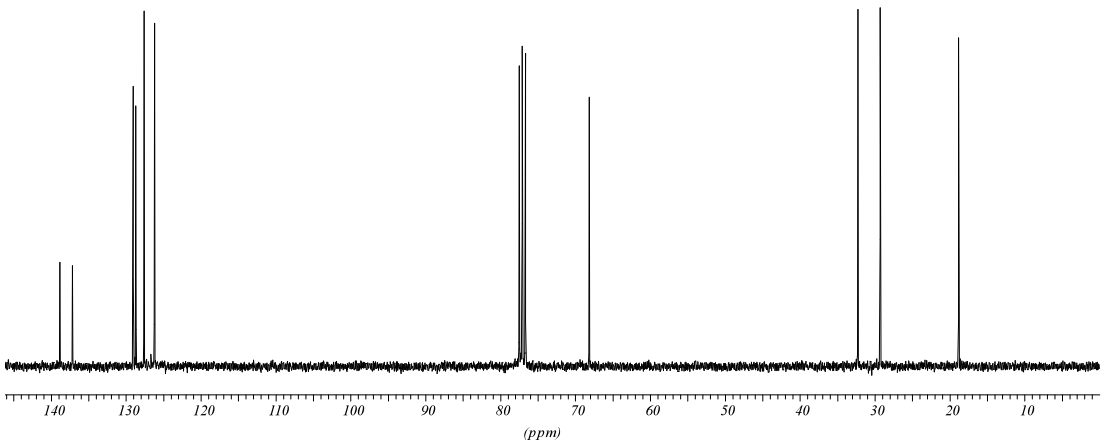
(*S*)-1-(naphthalen-2-yl)ethanol



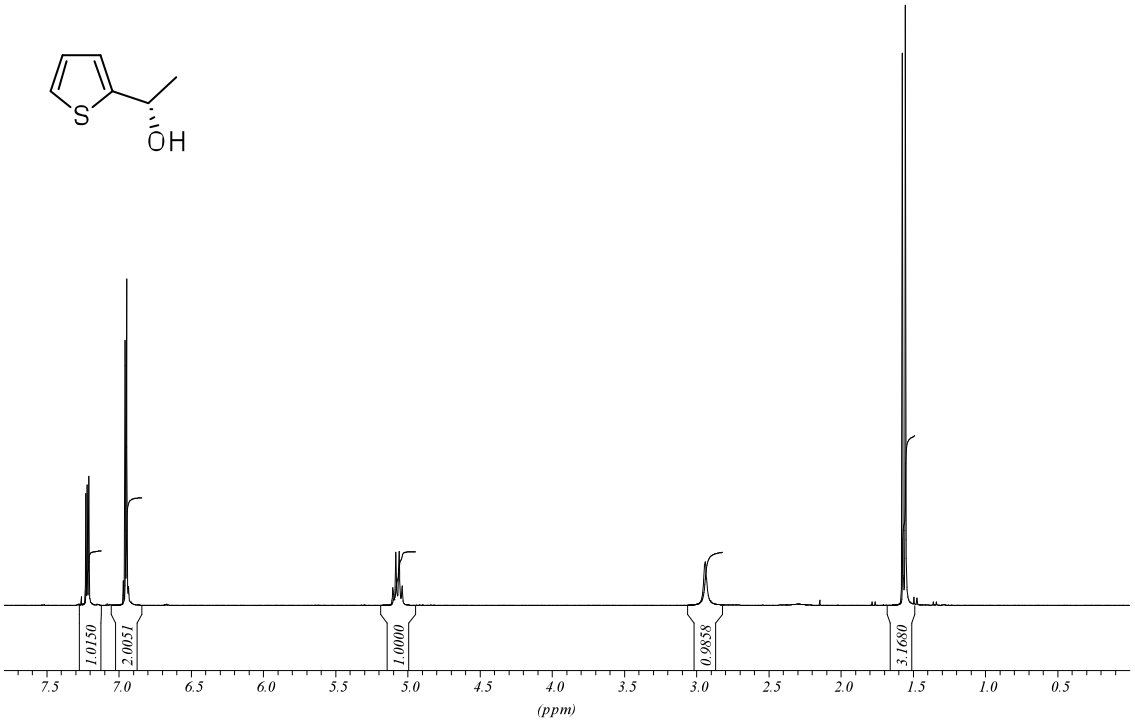


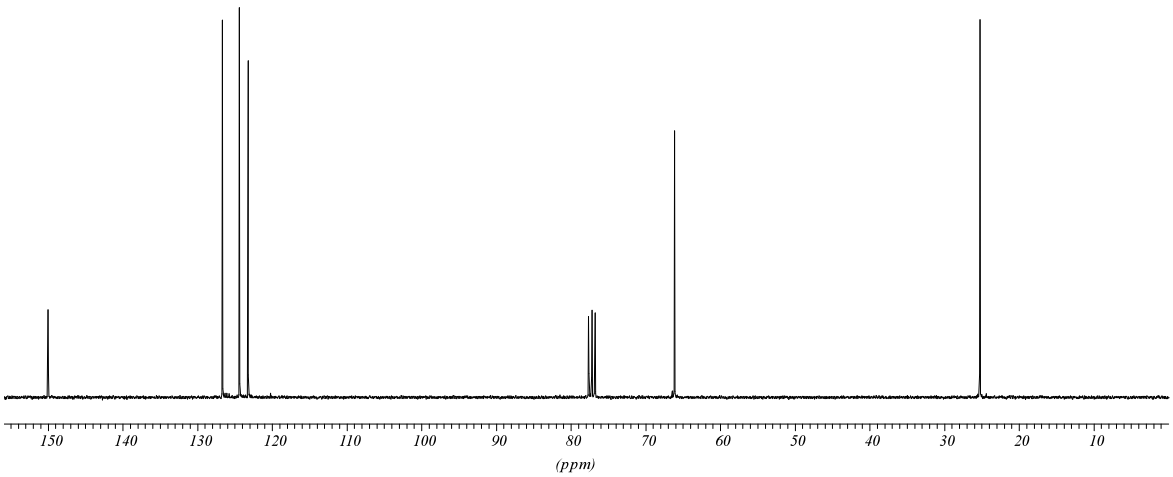
(*S*)-1,2,3,4-tetrahydronaphthalen-1-ol



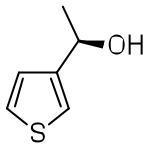


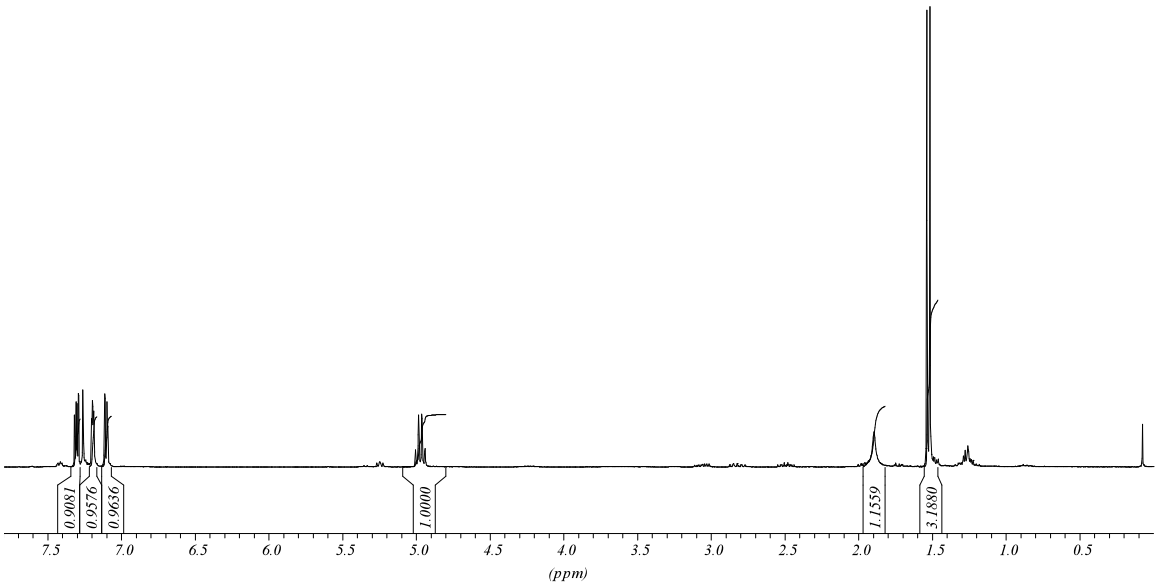
(*S*)-1-(2-Thienyl)ethanol

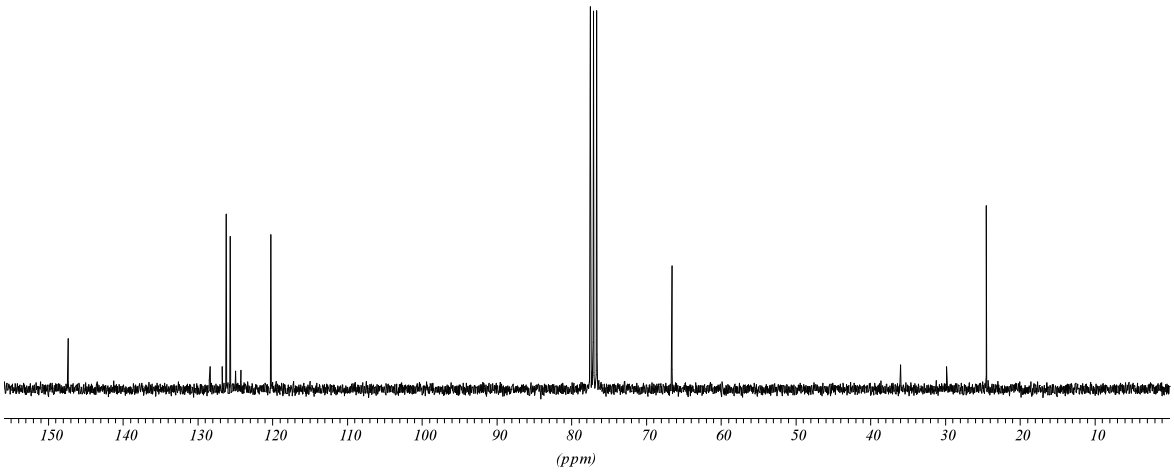




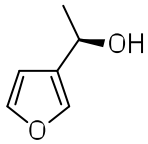
(*R*)-1-(3-Thienyl)ethanol

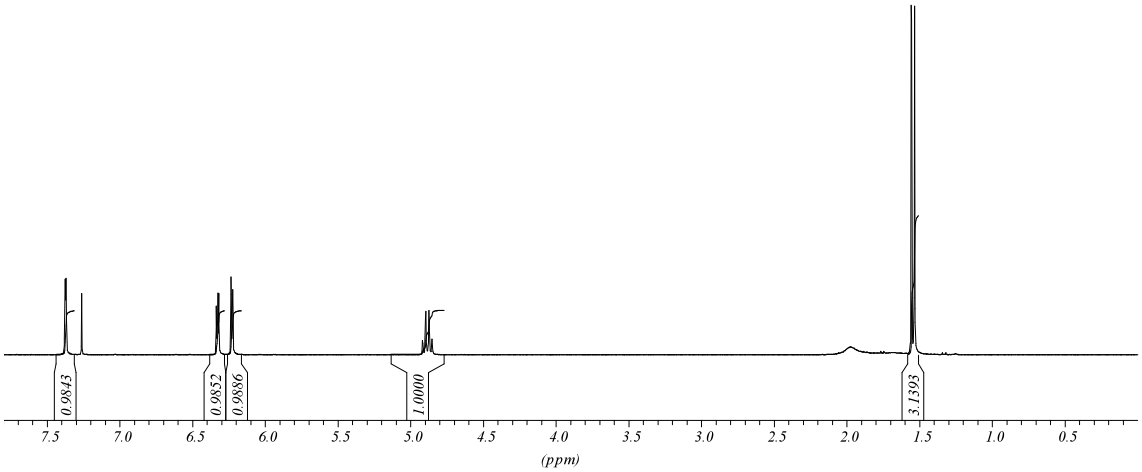


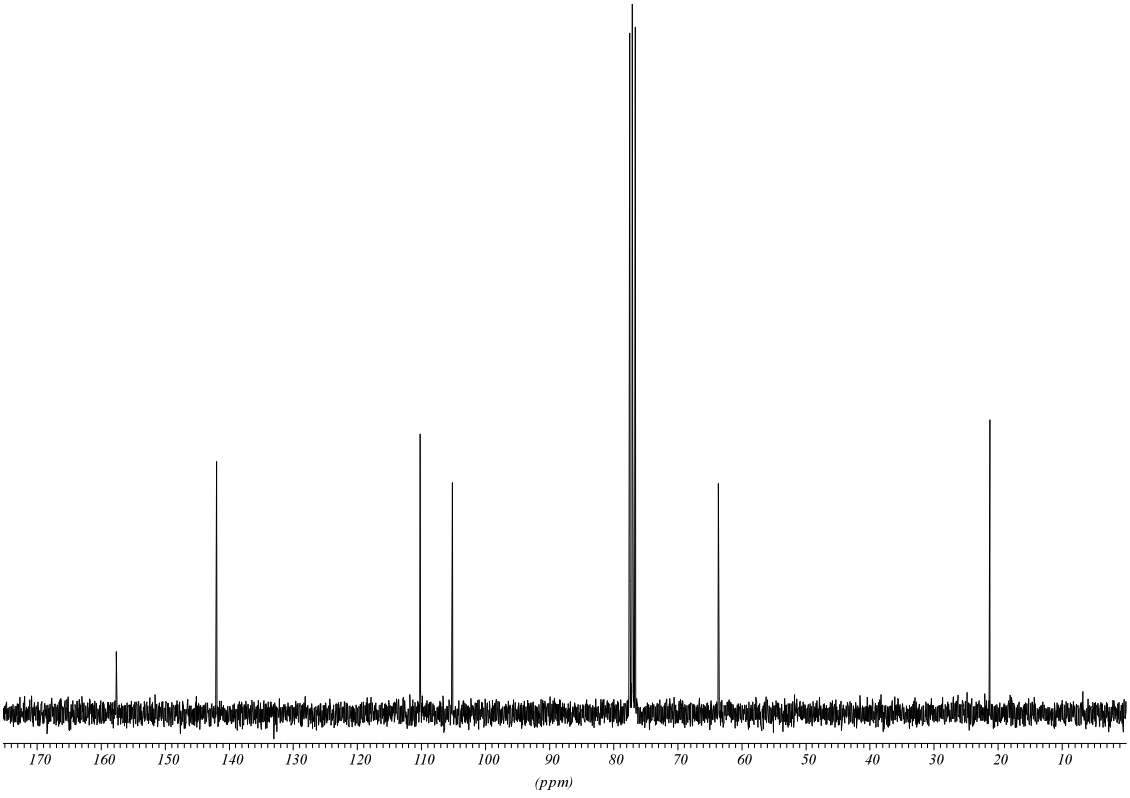




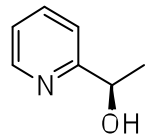
(*R*)-1-(furan-3-yl)ethanol

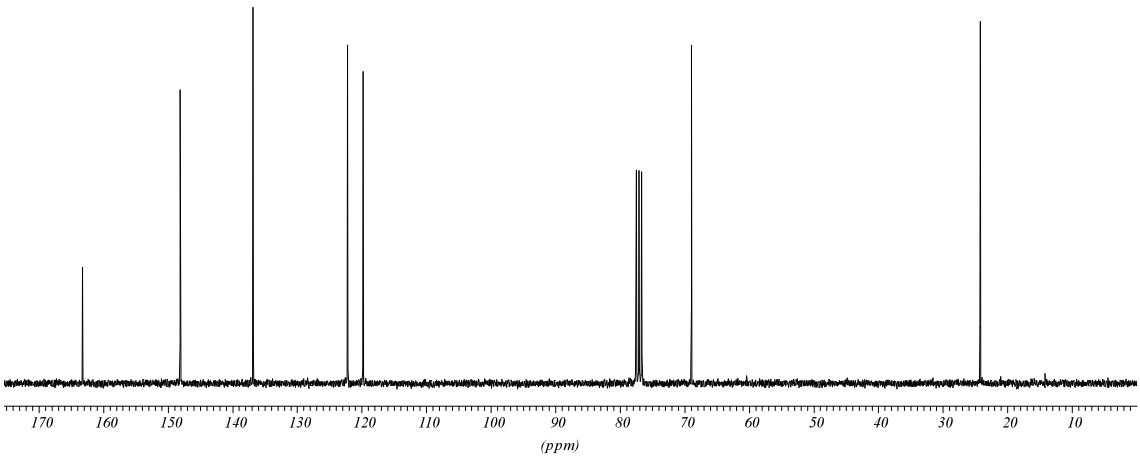




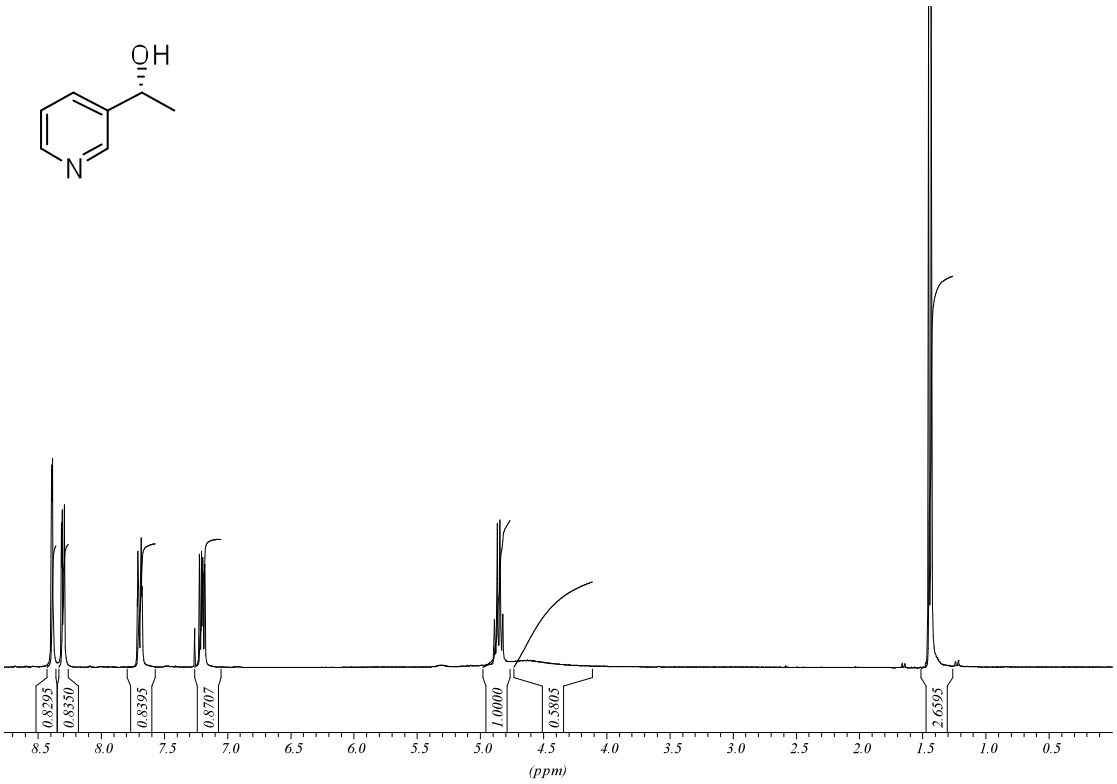


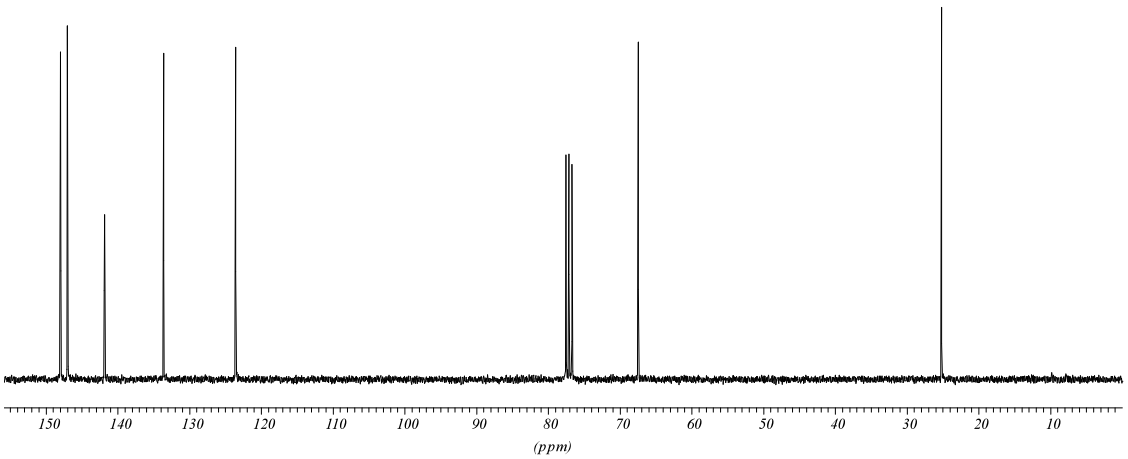
(*R*)-1-(pyridin-2-yl)ethanol





(*R*)-1-(pyridin-3-yl)ethanol





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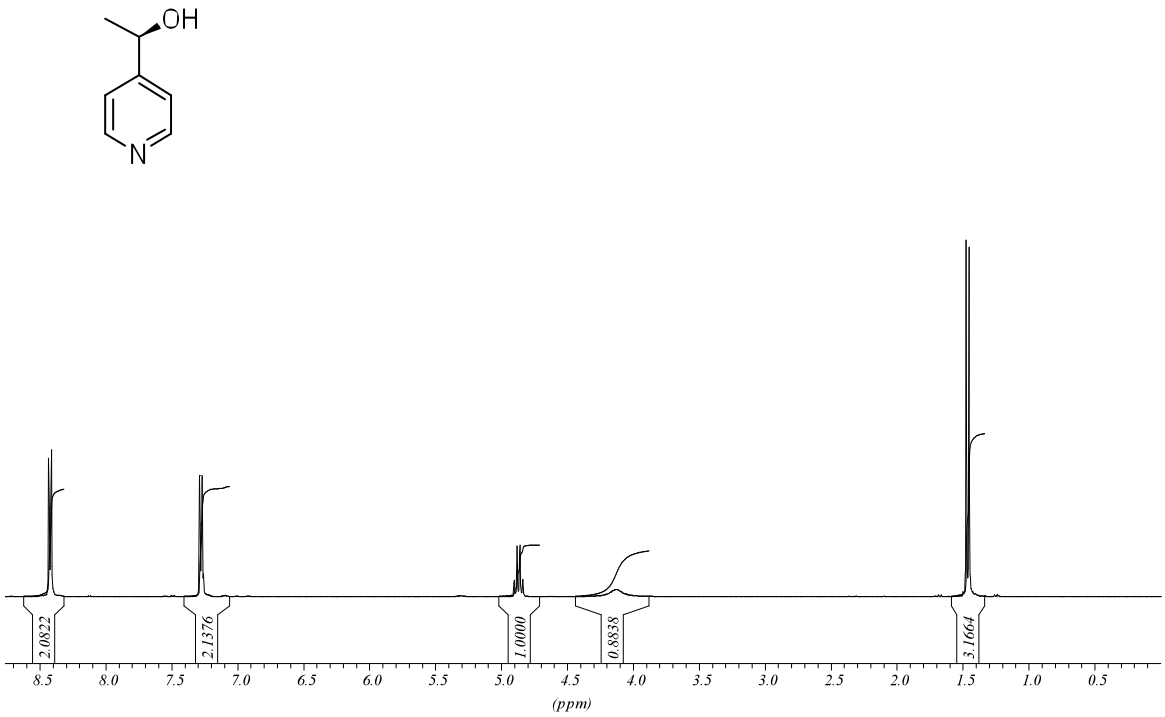
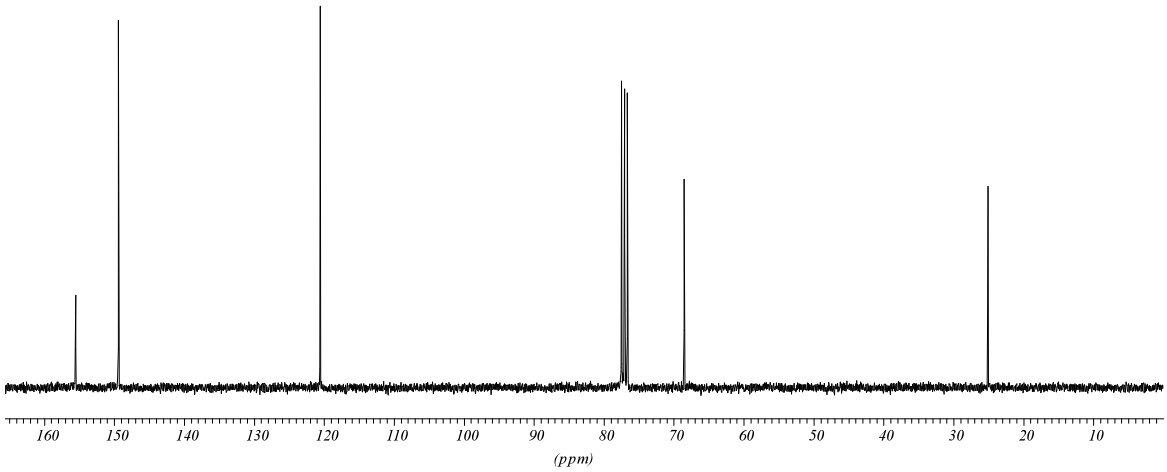
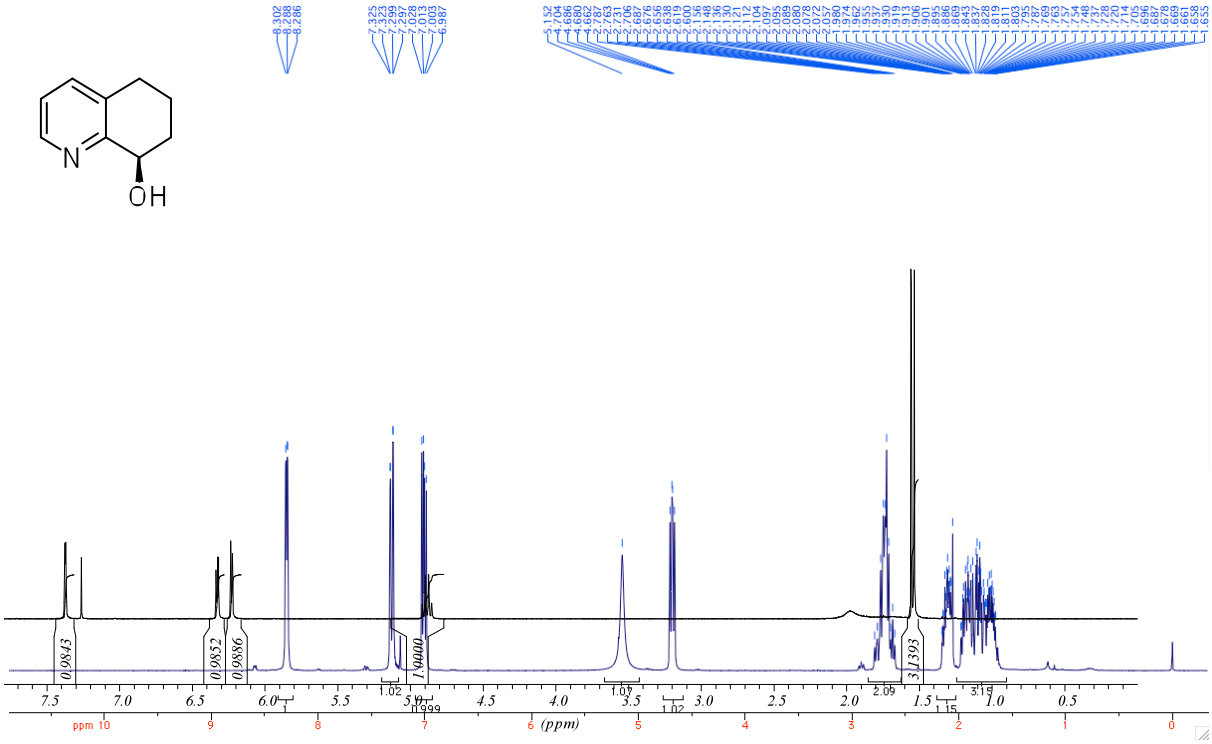
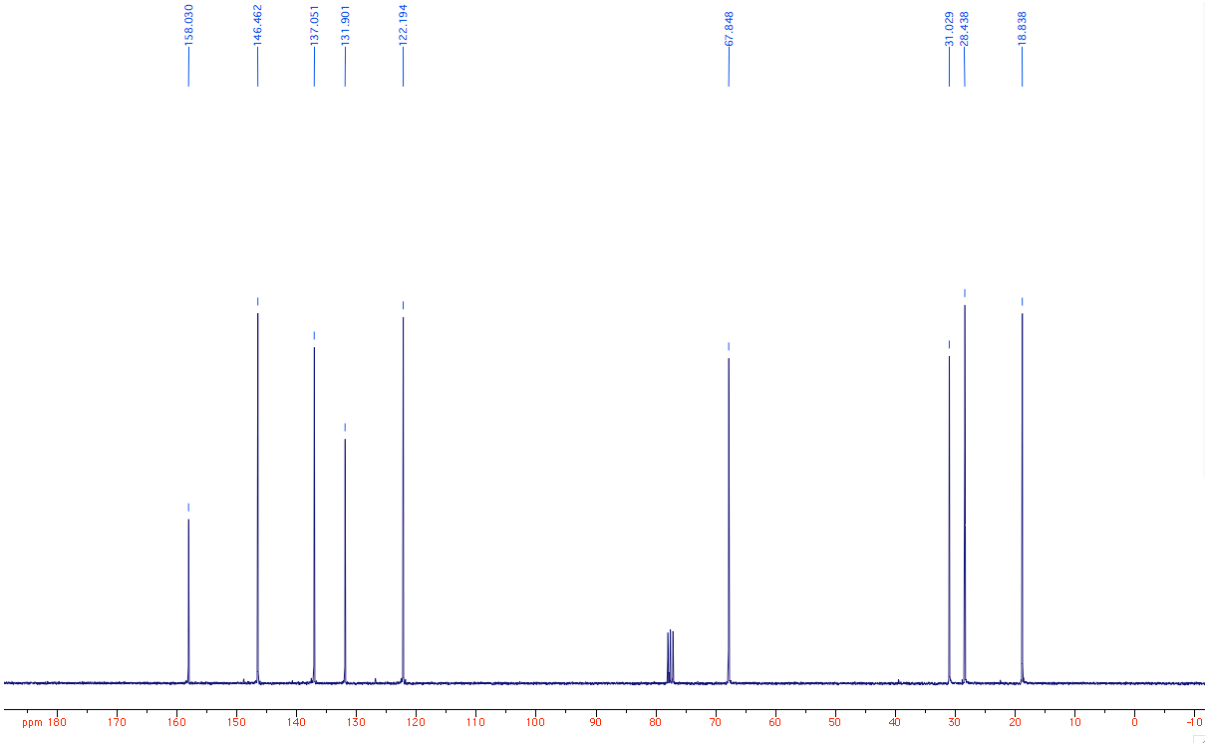


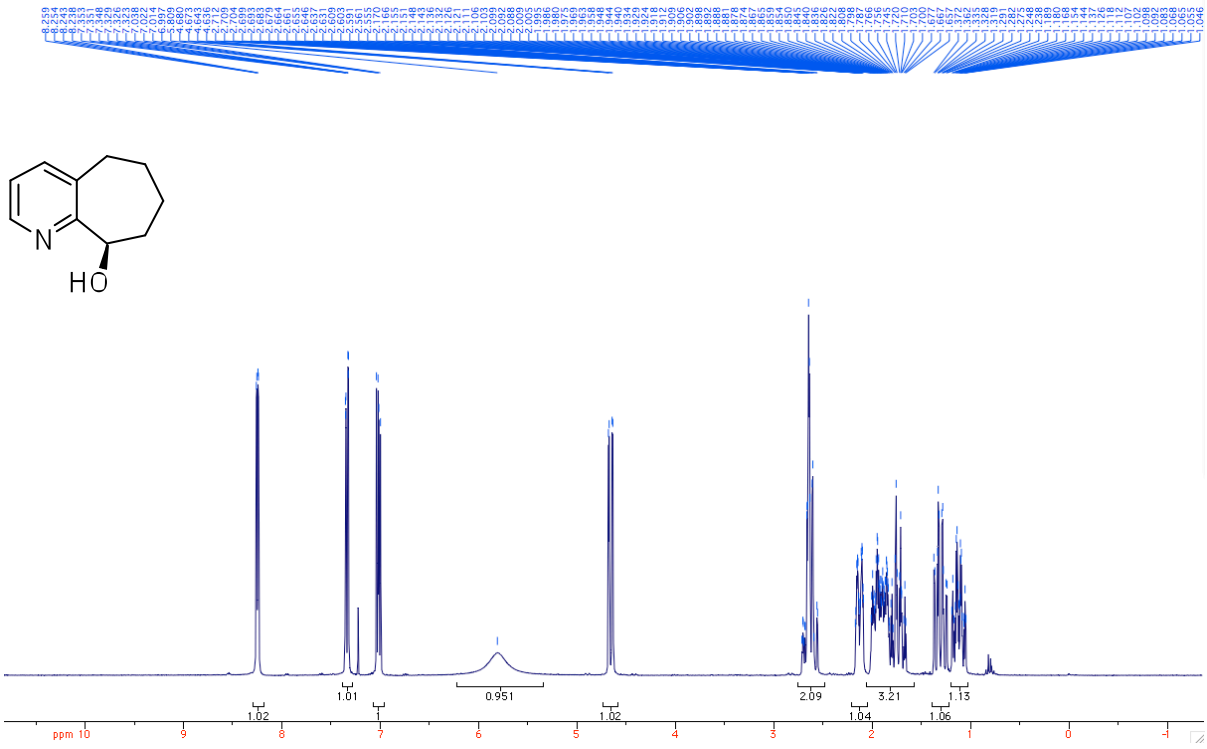


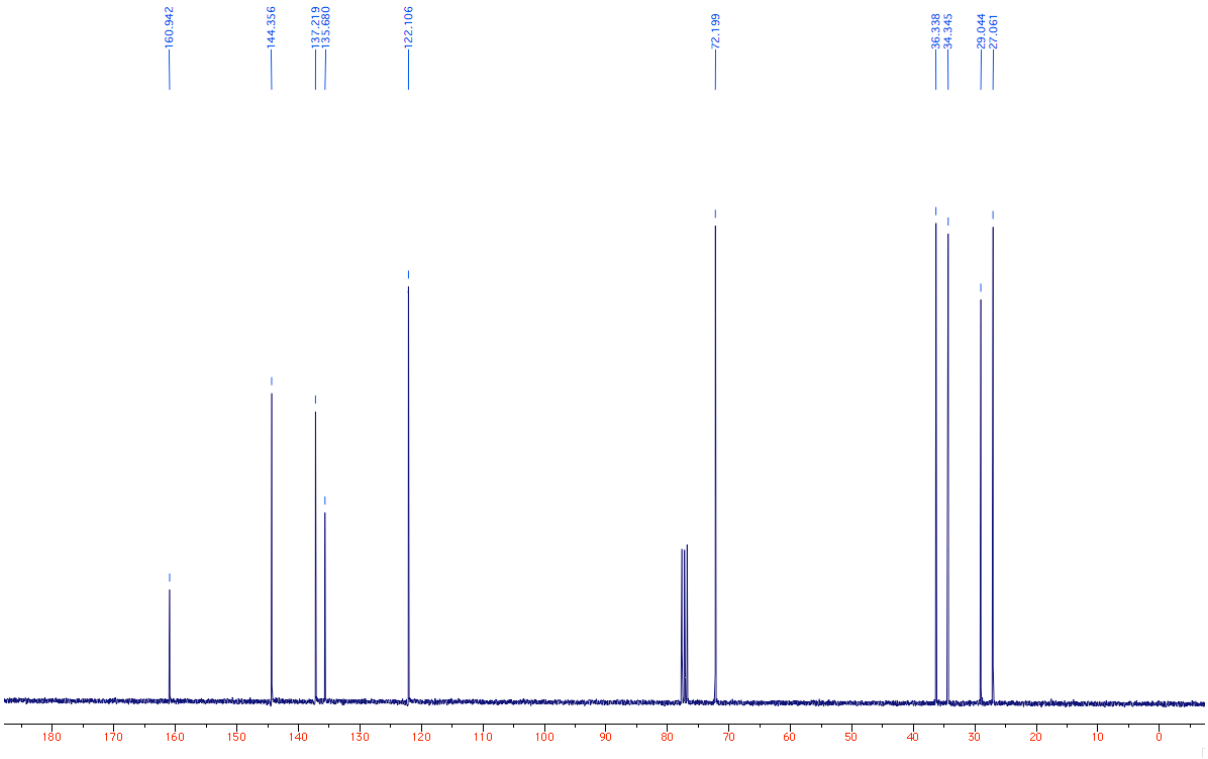
(*R*)-8-hydroxy-5, 6, 7, 8-tetrahydroquinoline



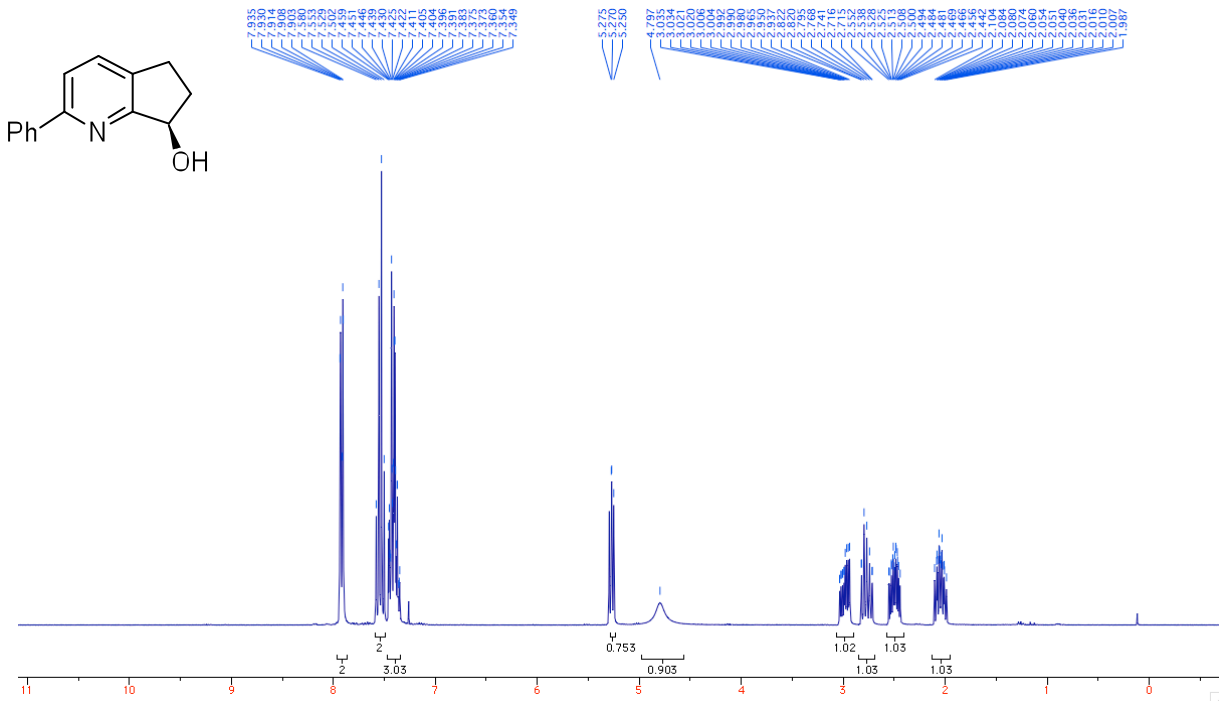


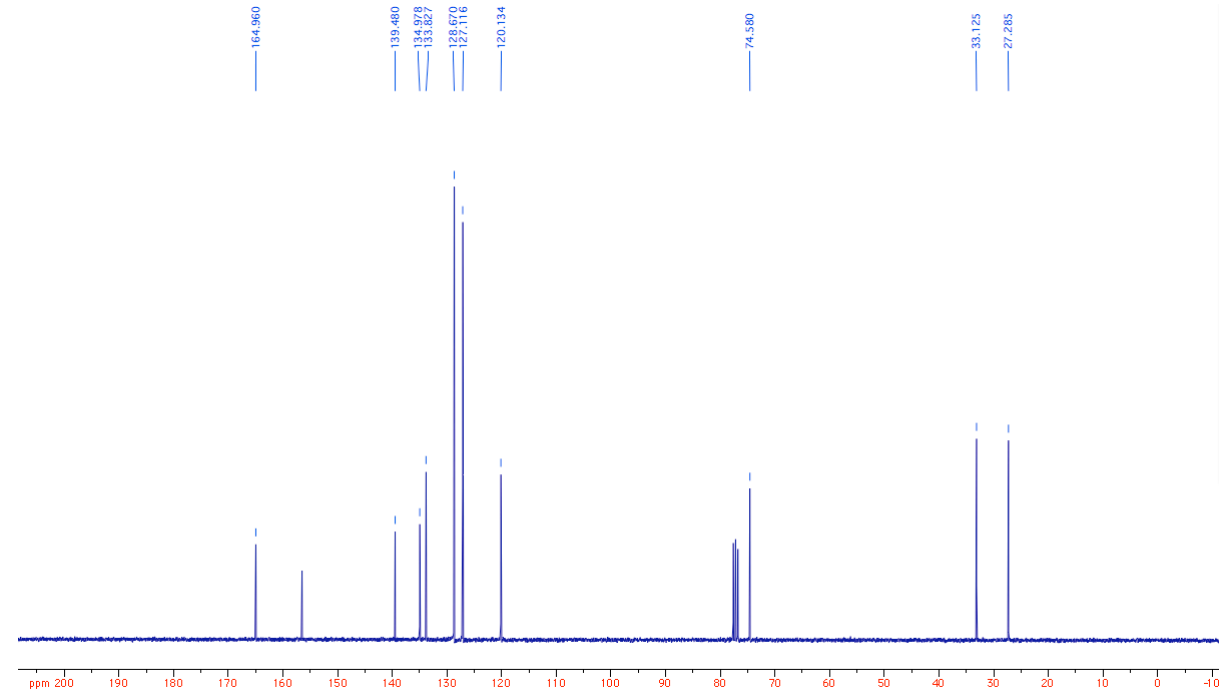
(*R*)-9-hydroxy-6, 7, 8, 9-tetrahydro-5H-cycloheptapyridine



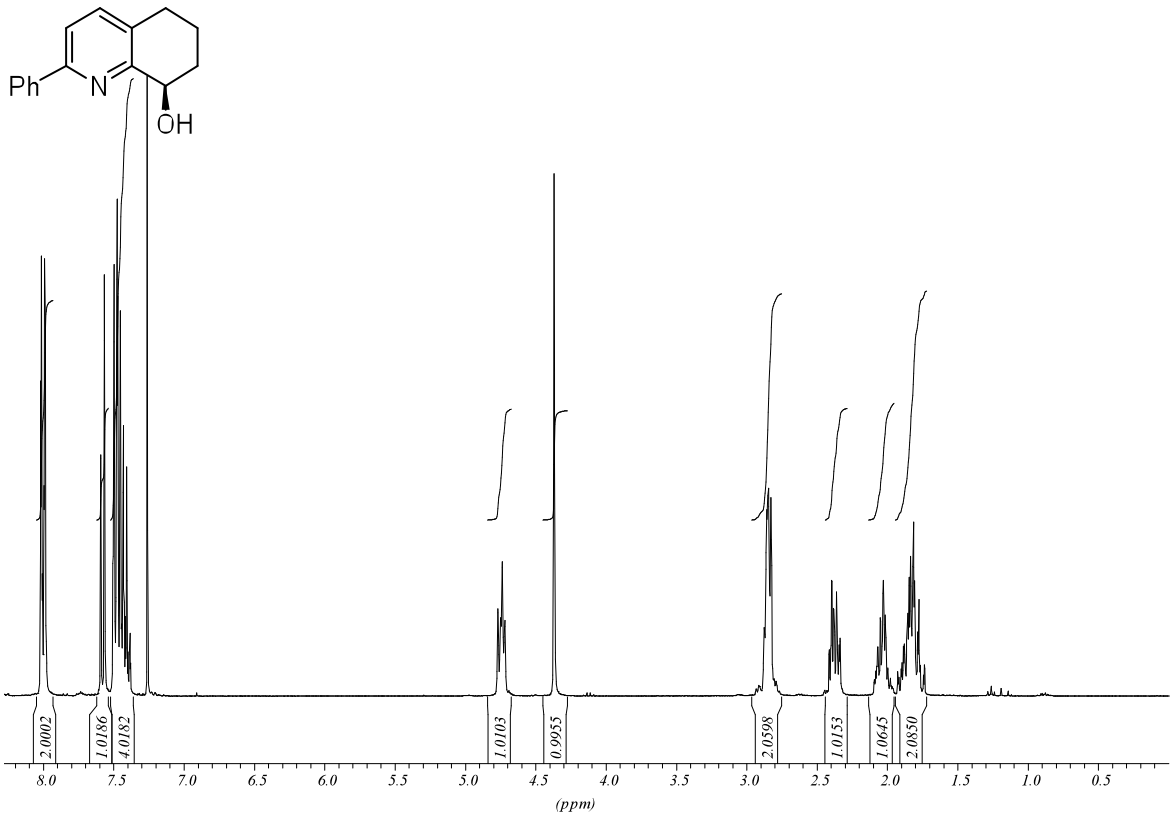


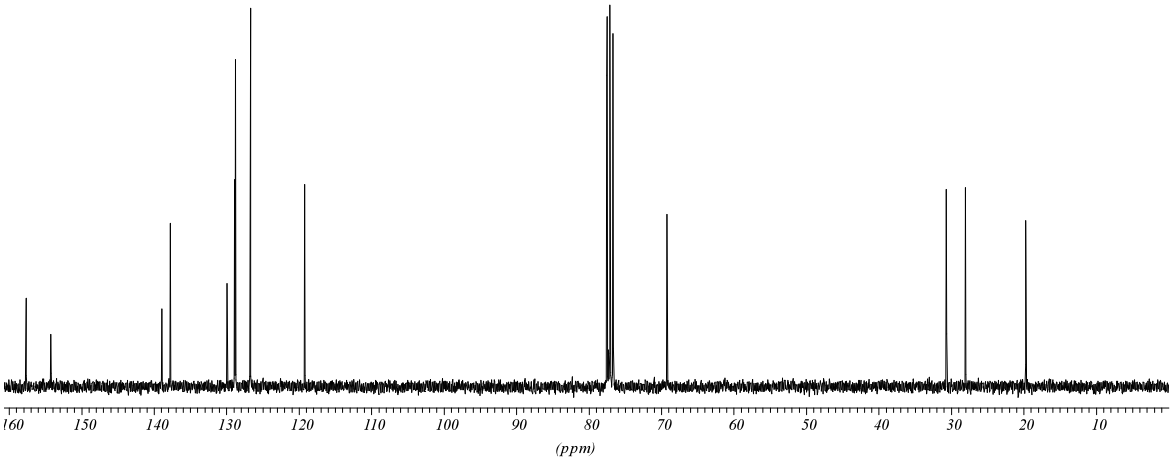
(*R*)-2-phenyl-7-hydroxy- 6, 7-dihydro-5H-cyclopentapyridine



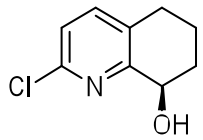


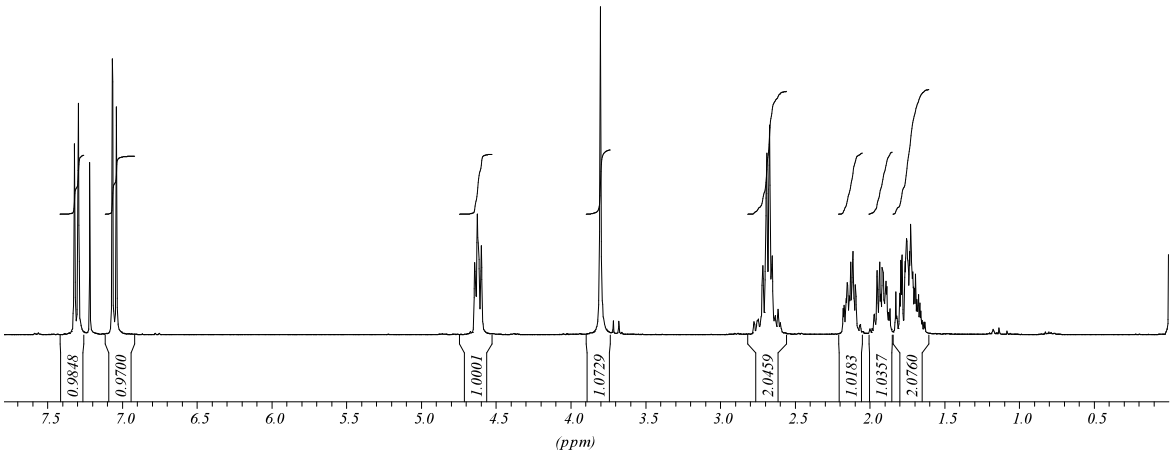
(*R*)-8-hydroxy-2-phenyl-5, 6, 7, 8-tetrahydroquinoline

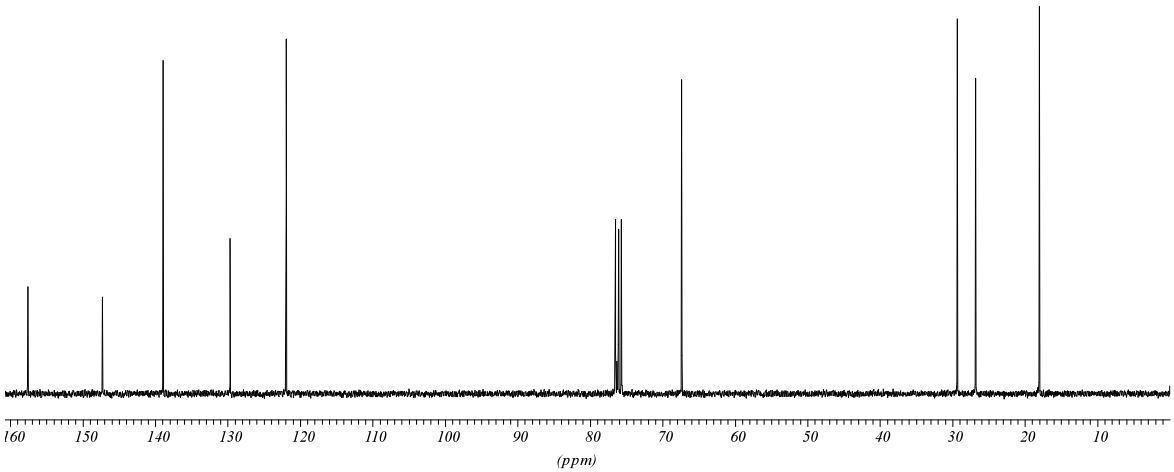




(*R*)-8-hydroxy-2-chloro-5, 6, 7, 8-tetrahydro-2-quionoline



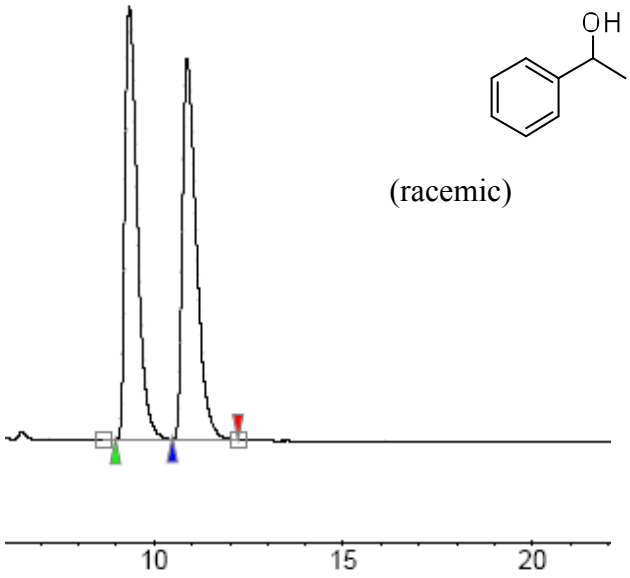




7. HPLC DATA:

1-phenylethanol

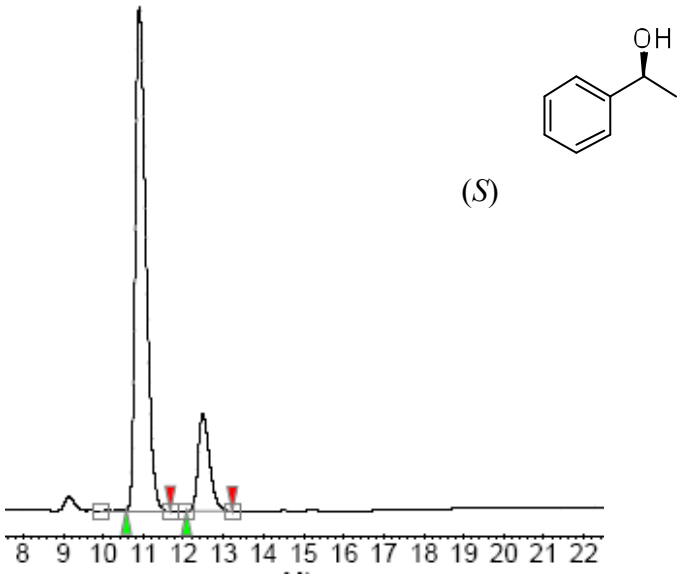
OJ: heptane/isopropanol (95/5, 1mL/min, 254 nm



(racemic)

Peak results :

Index	Name	Time [Min]	Quantity [% Area]	Height [mAU]	Area [mAU.Min]	Area % [%]
1	UNKNOWN	9,35	49,95	44,7	16,8	49,950
2	UNKNOWN	10,88	50,05	39,3	16,9	50,050
Total			100,00	84,0	33,7	100,000



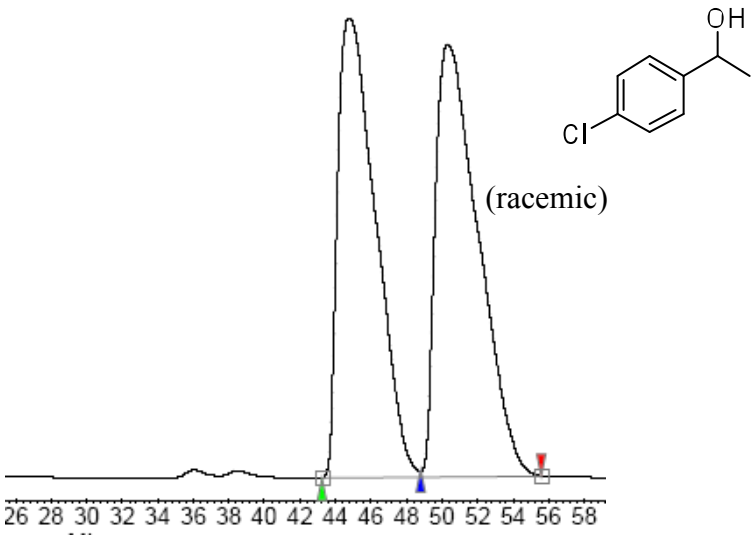
(*S*)

Peak results :

Index	Name	Time [Min]	Quantity [% Area]	Height [mAU]	Area [mAU.Min]	Area % [%]
1	UNKNOWN	10,90	82,95	439,7	135,1	82,946
2	UNKNOWN	12,49	17,05	84,7	27,8	17,054
Total			100,00	524,4	162,9	100,000

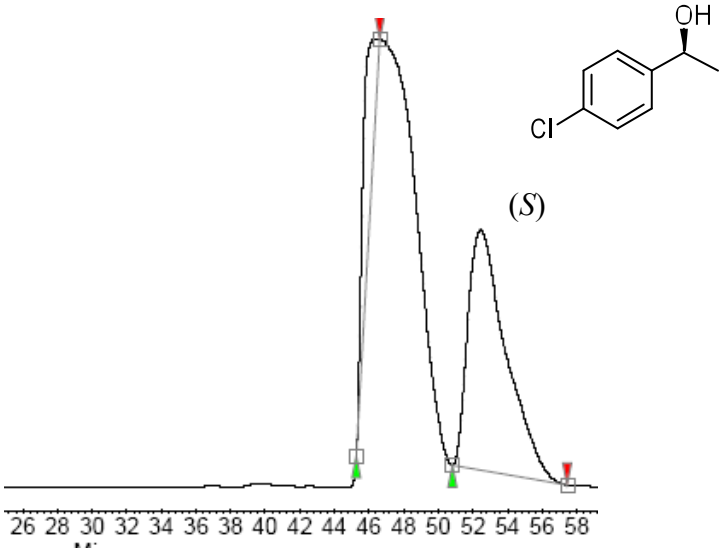
1-(4-chlorophenyl)ethanol

OD-H: heptane/isopropanol (99/1), 0.5 mL/min, 254 nm



Peak results :

Index	Name	Time [Min]	Quantity [% Area]	Height [mAU]	Area [mAU.Min]	Area % [%]
1	UNKNOWN	44,76	48,46	1330,6	3291,8	48,456
2	UNKNOWN	50,34	51,54	1252,7	3501,6	51,544
Total			100,00	2583,3	6793,5	100,000

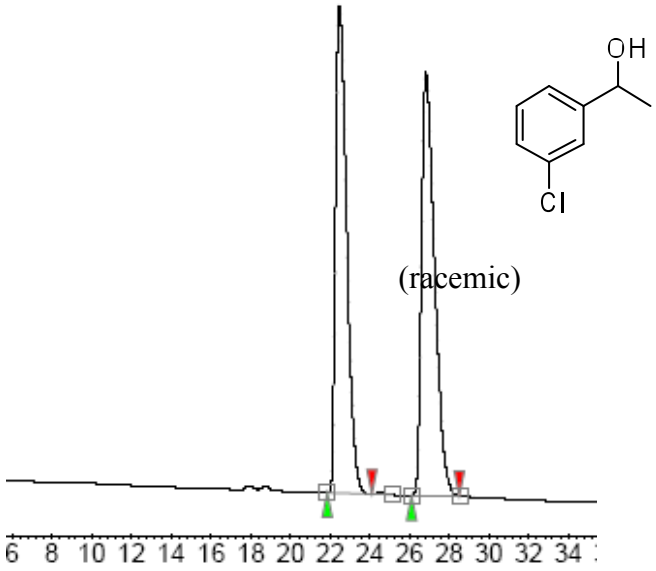


Peak results :

Index	Name	Time [Min]	Quantity [% Area]	Height [mAU]	Area [mAU.Min]	Area % [%]
1	UNKNOWN	45,81	20,14	674,4	491,3	20,137
2	UNKNOWN	52,46	79,86	765,7	1948,5	79,863
Total			100,00	1440,0	2439,8	100,000

1-(3-chlorophenyl)ethanol

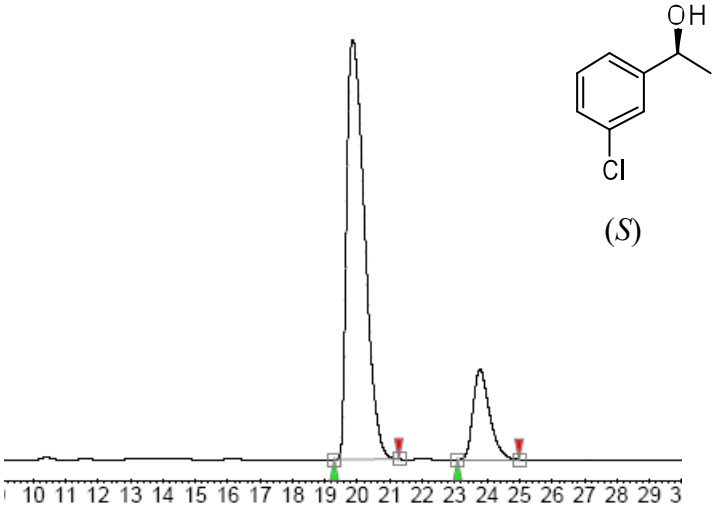
OJ: heptane/isopropanol (99/1), 1 mL/min, 254 nm



(racemic)

Peak results :

Index	Name	Time [Min]	Quantity [% Area]	Height [mAU]	Area [mAU.Min]	Area % [%]
1	UNKNOWN	22,47	49,22	858,6	554,2	49,220
2	UNKNOWN	26,82	50,78	747,3	571,8	50,780
Total			100,00	1606,0	1126,0	100,000



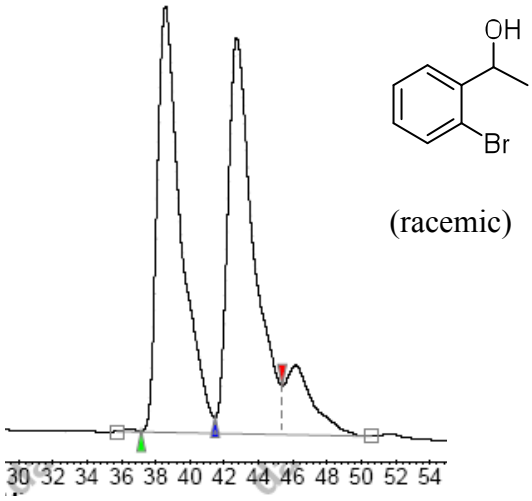
(*S*)

Peak results :

Index	Name	Time [Min]	Quantity [% Area]	Height [mAU]	Area [mAU.Min]	Area % [%]
1	UNKNOWN	19,85	83,47	1063,4	677,7	83,473
2	UNKNOWN	23,78	16,53	229,2	134,2	16,527
Total			100,00	1292,7	811,9	100,000

1-(2-bromoophenyl)ethanol

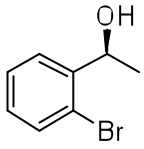
OD-H: heptane/isopropanol (99/1), 0.5 mL/min, 254 nm



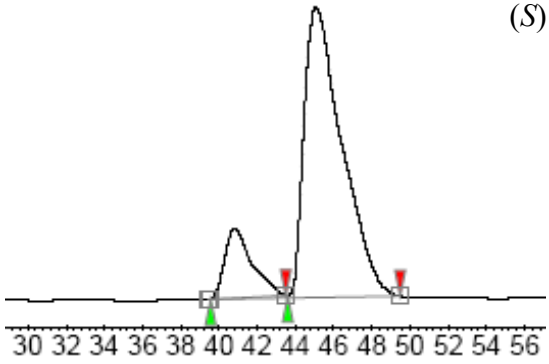
(racemic)

Peak results :

Index	Name	Time [Min]	Quantity [% Area]	Height [mAU]	Area [mAU.Min]	Area % [%]
1	UNKNOWN	38,57	49,92	482,3	784,0	49,917
2	UNKNOWN	42,73	50,08	447,9	786,6	50,083
Total			100,00	930,2	1570,6	100,000



(*S*)

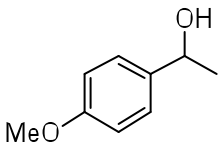


Peak results :

Index	Name	Time [Min]	Quantity [% Area]	Height [mAU]	Area [mAU.Min]	Area % [%]
2	UNKNOWN	40,82	14,73	258,8	430,2	14,733
1	UNKNOWN	45,08	85,27	1083,5	2489,9	85,267
Total			100,00	1342,4	2920,2	100,000

1-(4-methoxyphenyl)ethanol

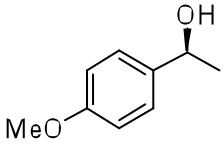
OD-H: heptane/isopropanol (99/1), 1 mL/min, 254 nm



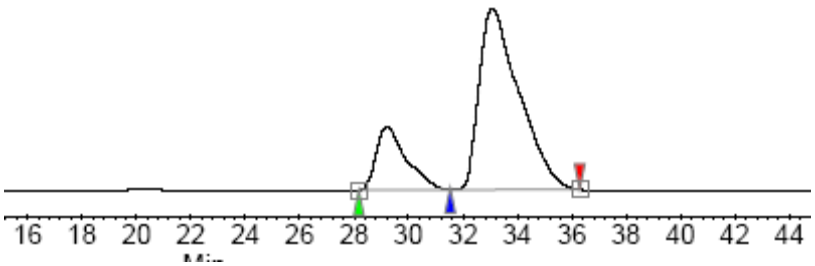
(racemic)

Peak results :

Index	Name	Time [Min]	Quantity [% Area]	Height [mAU]	Area [mAU.Min]	Area % [%]
1	UNKNOWN	26,43	49,53	91,1	133,5	49,531
2	UNKNOWN	29,77	50,47	78,1	136,0	50,469
Total			100,00	169,1	269,6	100,000



(*S*)

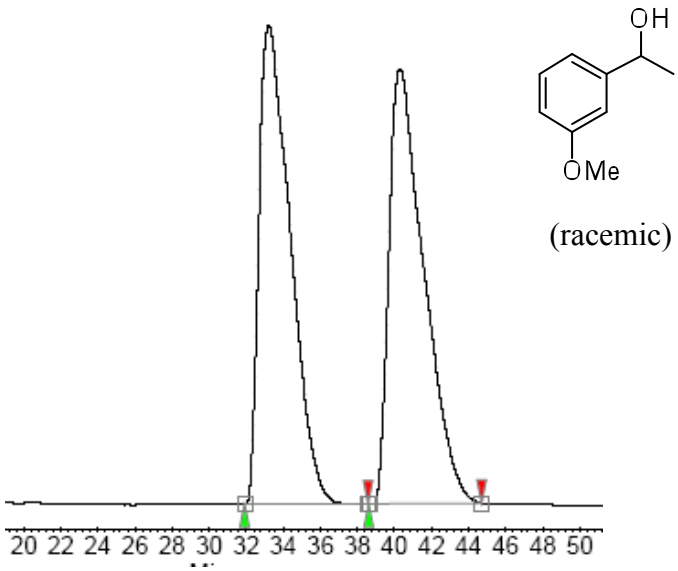


Peak results :

Index	Name	Time [Min]	Quantity [% Area]	Height [mAU]	Area [mAU.Min]	Area % [%]
1	UNKNOWN	29,21	20,91	227,7	289,9	20,912
2	UNKNOWN	33,09	79,09	643,2	1096,5	79,088
Total			100,00	870,9	1386,4	100,000

1-(3-methoxphenyl)ethanol

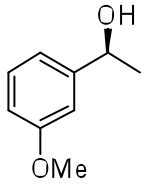
OD-H: heptane/isopropanol (99/1), 1 mL/min, 254 nm



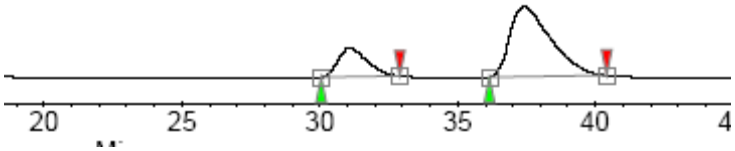
(racemic)

Peak results :

Index	Name	Time [Min]	Quantity [% Area]	Height [mAU]	Area [mAU.Min]	Area % [%]
1	UNKNOWN	33,22	49,70	1027,4	2012,1	49,702
2	UNKNOWN	40,29	50,30	933,9	2036,2	50,298
Total			100,00	1961,3	4048,3	100,000



(*S*)

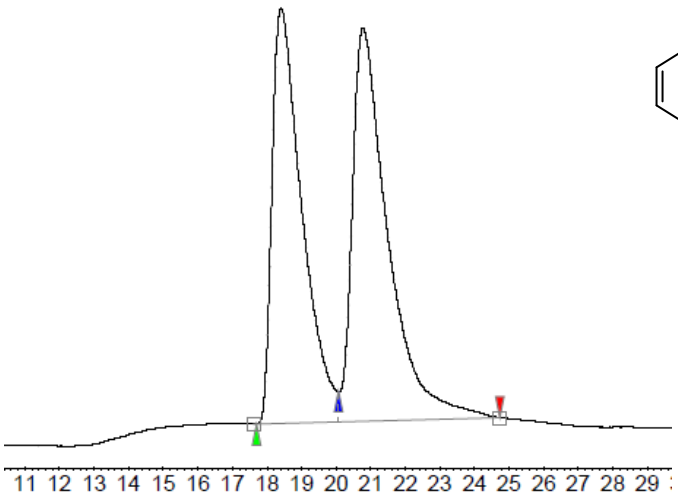


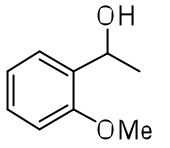
Peak results :

Index	Name	Time [Min]	Quantity [% Area]	Height [mAU]	Area [mAU.Min]	Area % [%]
1	UNKNOWN	31,08	22,17	41,1	50,2	22,174
2	UNKNOWN	37,42	77,83	102,7	176,2	77,826
Total			100,00	143,8	226,4	100,000

1-(2-methoxyphenyl)ethanol

OD-H: heptane/isopropanol (99/1), 1 mL/min, 254 nm

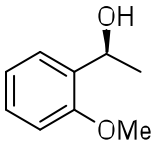




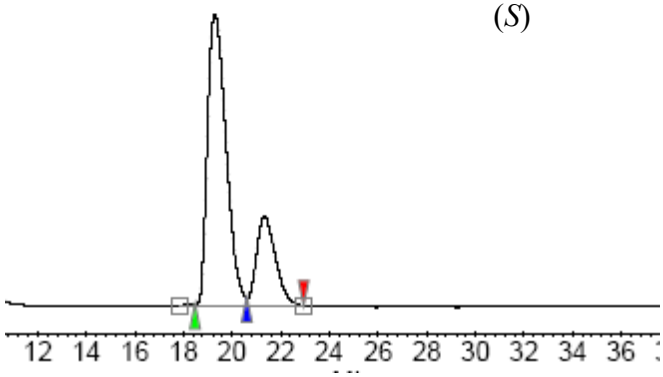
(racemic)

Peak results :

Index	Name	Time [Min]	Quantity [% Area]	Height [mAU]	Area [mAU.Min]	Area % [%]
1	UNKNOWN	18,41	46,96	569,8	570,6	46,962
2	UNKNOWN	20,78	53,04	539,7	644,5	53,038
Total			100,00	1109,5	1215,1	100,000



(*S*)

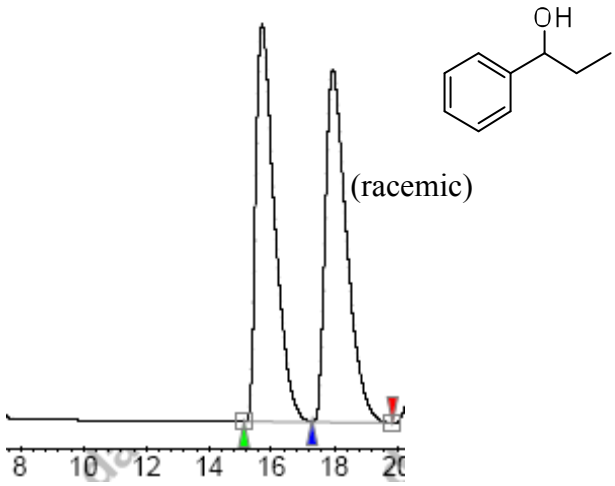


Peak results :

Index	Name	Time [Min]	Quantity [% Area]	Height [mAU]	Area [mAU.Min]	Area % [%]
1	UNKNOWN	19,28	75,94	1083,4	960,9	75,939
2	UNKNOWN	21,32	24,06	334,9	304,5	24,061
Total			100,00	1418,4	1265,4	100,000

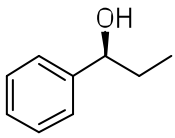
1-phenylpropan-1-ol

OD-H: heptane/isopropanol (99/1), 1 mL/min, 254 nm

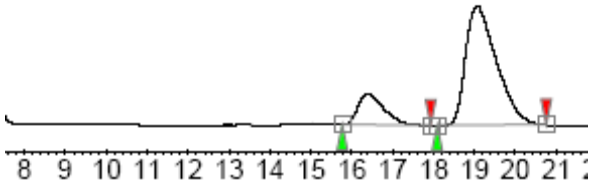


Peak results :

Index	Name	Time [Min]	Quantity [% Area]	Height [mAU]	Area [mAU.Min]	Area % [%]
1	UNKNOWN	15,70	49,96	104,3	72,4	49,960
2	UNKNOWN	17,95	50,04	92,7	72,5	50,040
Total			100,00	197,0	144,9	100,000



(*S*)

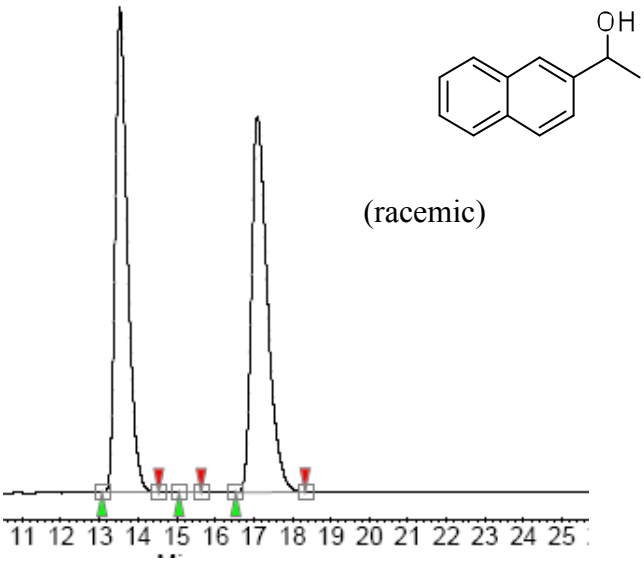


Peak results :

Index	Name	Time [Min]	Quantity [% Area]	Height [mAU]	Area [mAU.Min]	Area % [%]
1	UNKNOWN	16,39	17,86	98,1	70,5	17,863
2	UNKNOWN	19,06	82,14	370,9	324,3	82,137
Total			100,00	469,1	394,8	100,000

1-(naphthalen-2-yl)ethanol

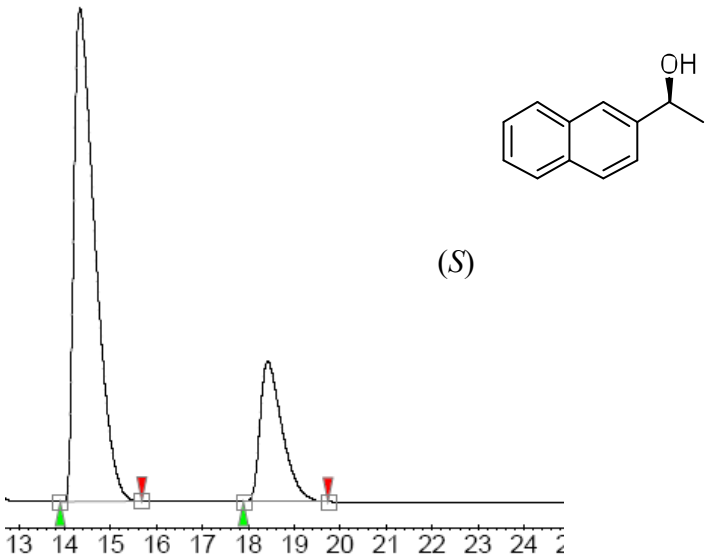
OJ: heptane/isopropanol (90/10), 1 mL/min, 254 nm



(racemic)

Peak results :

Index	Name	Time [Min]	Quantity [% Area]	Height [mAU]	Area [mAU.Min]	Area % [%]
1	UNKNOWN	13,53	49,79	212,3	78,2	49,792
2	UNKNOWN	17,10	50,21	164,1	78,9	50,208
Total			100,00	376,4	157,1	100,000



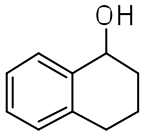
(*S*)

Peak results :

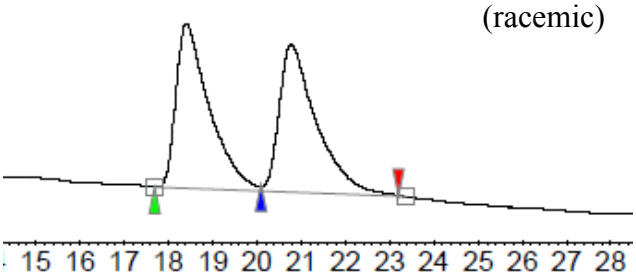
Index	Name	Time [Min]	Quantity [% Area]	Height [mAU]	Area [mAU.Min]	Area % [%]
1	UNKNOWN	14,34	77,02	1035,1	551,4	77,023
2	UNKNOWN	18,43	22,98	294,2	164,5	22,977
Total			100,00	1329,3	715,9	100,000

1,2,3,4-tetrahydronaphthalen-1-ol

OD-H: heptane/isopropanol (99/1), 1 mL/min, 254 nm

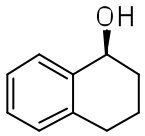


(racemic)

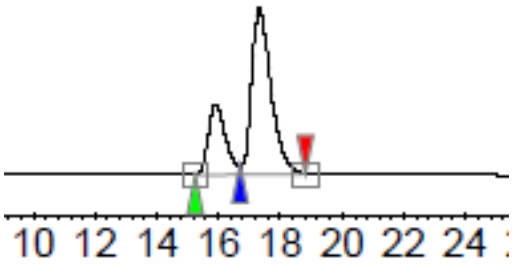


Peak results :

Index	Name	Time [Min]	Quantity [% Area]	Height [mAU]	Area [mAU.Min]	Area % [%]
1	UNKNOWN	18,41	49,84	21,7	19,0	49,844
2	UNKNOWN	20,78	50,16	19,5	19,1	50,156
Total			100,00	41,2	38,0	100,000



(*S*)

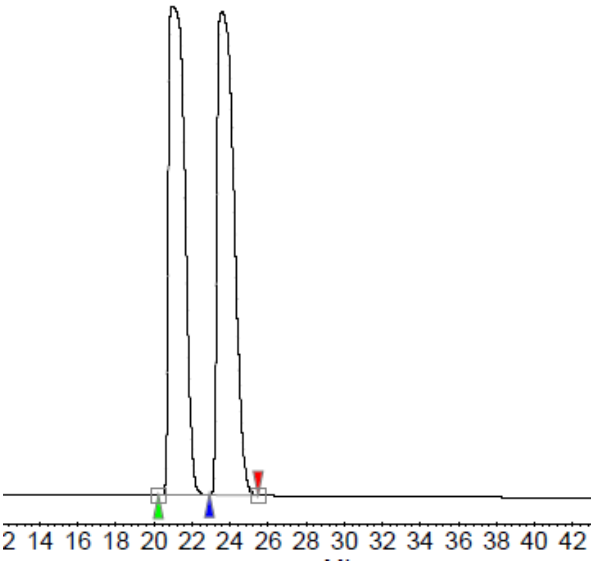


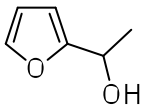
Peak results :

Index	Name	Time [Min]	Quantity [% Area]	Height [mAU]	Area [mAU.Min]	Area % [%]
1	UNKNOWN	15,91	27,10	151,5	97,9	27,101
2	UNKNOWN	17,33	72,90	357,8	263,2	72,899
Total			100,00	509,3	361,1	100,000

1-(furan-2-yl)ethanol

OJ: heptane/isopropanol (99/1), 1 mL/min, 254 nm

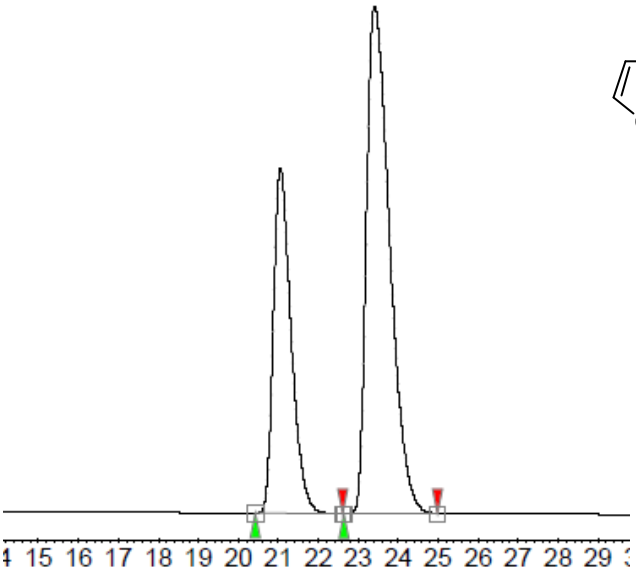


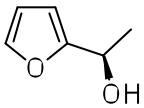


(racemic)

Peak results :

Index	Name	Time [Min]	Quantity [% Area]	Height [mAU]	Area [mAU.Min]	Area % [%]
1	UNKNOWN	20,98	49,37	1241,4	1220,7	49,370
2	UNKNOWN	23,58	50,63	1228,7	1251,8	50,630
Total			100,00	2470,1	2472,5	100,000





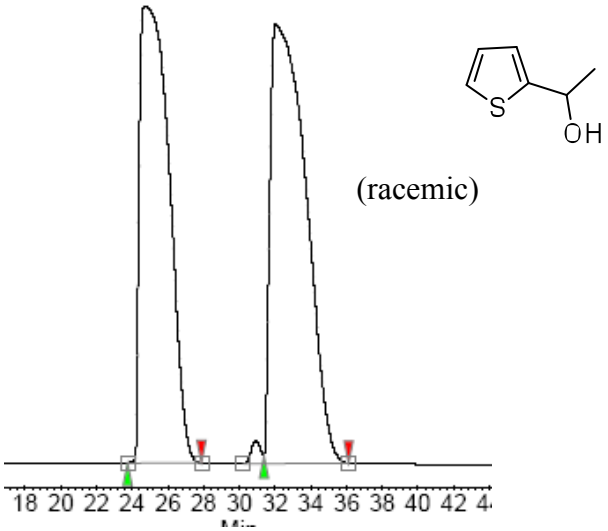
(*R*)

Peak results :

Index	Name	Time [Min]	Quantity [% Area]	Height [mAU]	Area [mAU.Min]	Area % [%]
1	UNKNOWN	21,05	34,23	625,0	321,9	34,229
2	UNKNOWN	23,41	65,77	919,3	618,5	65,771
Total			100,00	1544,3	940,4	100,000

1-(thiophen-2-yl)ethanol

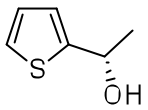
OJ: heptane/isopropanol (99/1), 1 mL/min, 254 nm



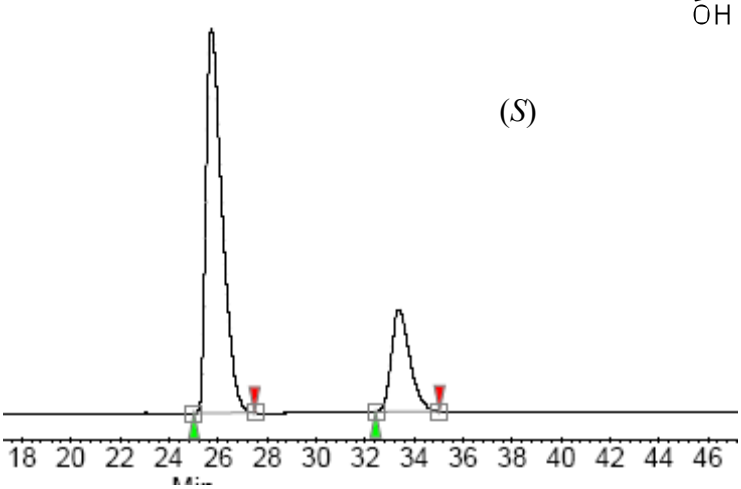
(racemic)

Peak results :

Index	Name	Time [Min]	Quantity [% Area]	Height [mAU]	Area [mAU.Min]	Area % [%]
1	UNKNOWN	24,69	46,14	1684,9	3203,5	46,137
2	UNKNOWN	32,04	53,86	1617,1	3739,9	53,863
Total			100,00	3301,9	6943,4	100,000



(*S*)

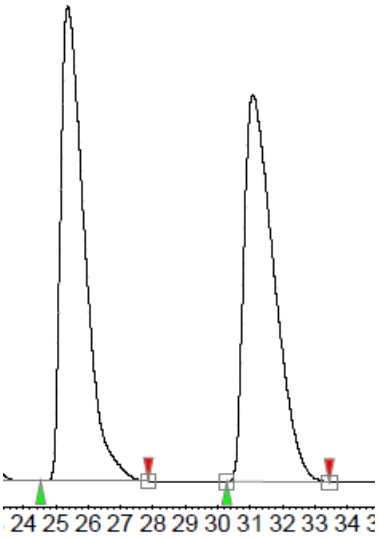


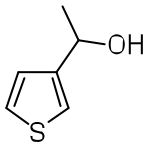
Peak results :

Index	Name	Time [Min]	Quantity [% Area]	Height [mAU]	Area [mAU.Min]	Area % [%]
1	UNKNOWN	25,72	76,64	766,3	583,2	76,638
2	UNKNOWN	33,38	23,36	203,7	177,8	23,362
Total			100,00	969,9	761,0	100,000

1-(3-Thienyl)ethanol

OJ: heptane/isopropanol (99/1), 1 mL/min, 254 nm

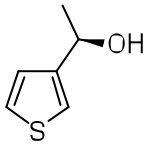




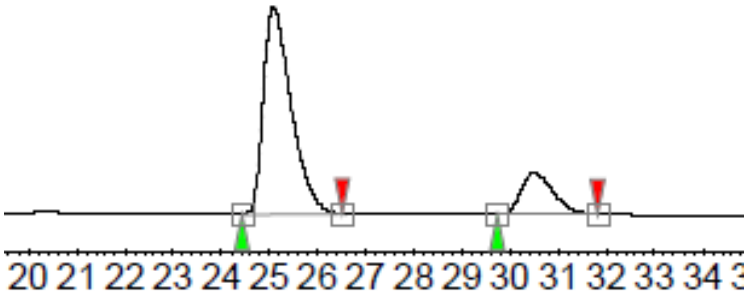
(racemic)

Peak results :

Index	Name	Time [Min]	Quantity [% Area]	Height [mAU]	Area [mAU.Min]	Area % [%]
1	UNKNOWN	25,37	49,40	142,1	121,1	49,402
2	UNKNOWN	31,09	50,60	115,7	124,1	50,598
Total			100,00	257,8	245,2	100,000



(*R*)

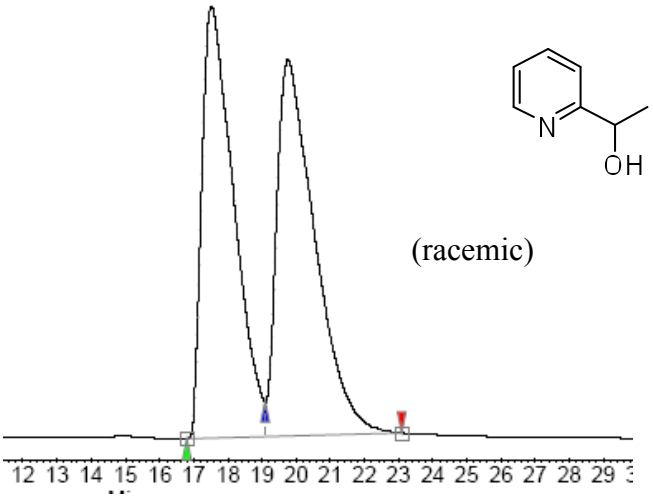


Peak results :

Index	Name	Time [Min]	Quantity [% Area]	Height [mAU]	Area [mAU.Min]	Area % [%]
1	UNKNOWN	25,08	81,13	501,5	336,7	81,130
2	UNKNOWN	30,51	18,87	101,5	78,3	18,870
Total			100,00	603,0	415,0	100,000

1-(pyridin-2-yl)ethanol

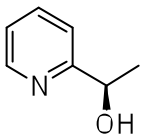
OD-H: heptane/isopropanol (99/1), 1 mL/min, 254 nm



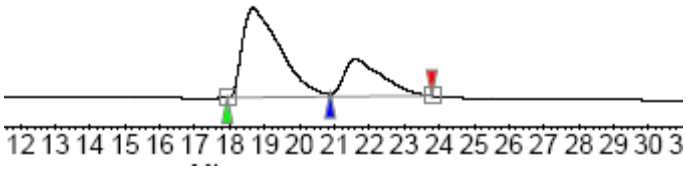
(racemic)

Peak results :

Index	Name	Time [Min]	Quantity [% Area]	Height [mAU]	Area [mAU.Min]	Area % [%]
1	UNKNOWN	17,52	48,93	582,5	631,3	48,927
2	UNKNOWN	19,76	51,07	508,4	659,0	51,073
Total			100,00	1090,8	1290,2	100,000



(*R*)

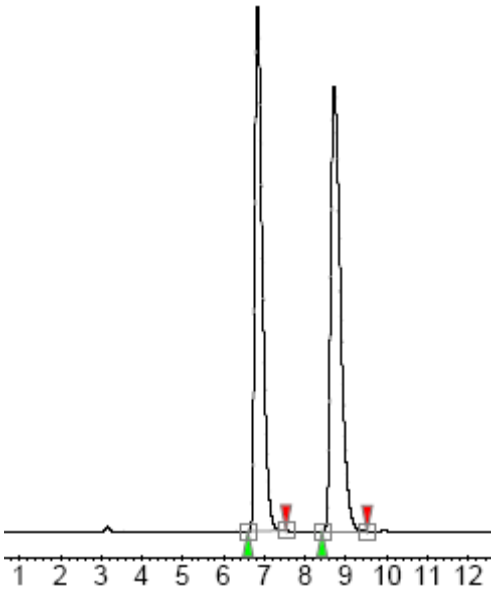


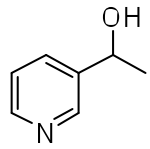
Peak results :

Index	Name	Time [Min]	Quantity [% Area]	Height [mAU]	Area [mAU.Min]	Area % [%]
1	UNKNOWN	18,66	70,43	313,6	394,3	70,432
2	UNKNOWN	21,61	29,57	130,2	165,5	29,568
Total			100,00	443,8	559,8	100,000

1-(pyridin-3-yl)ethanol

OJ: heptane/isopropanol (90/10), 1 mL/min, 254 nm

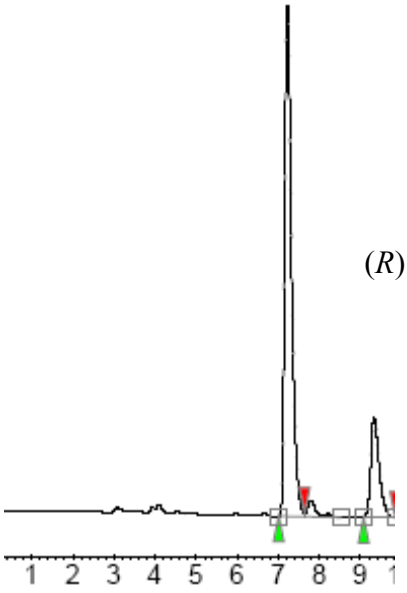


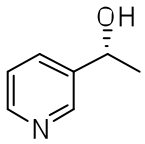


(racemic)

Peak results :

Index	Name	Time [Min]	Quantity [% Area]	Height [mAU]	Area [mAU.Min]	Area % [%]
1	UNKNOWN	6,83	48,32	1048,8	229,1	48,315
2	UNKNOWN	8,71	51,68	888,0	245,1	51,685
Total			100,00	1936,8	474,2	100,000





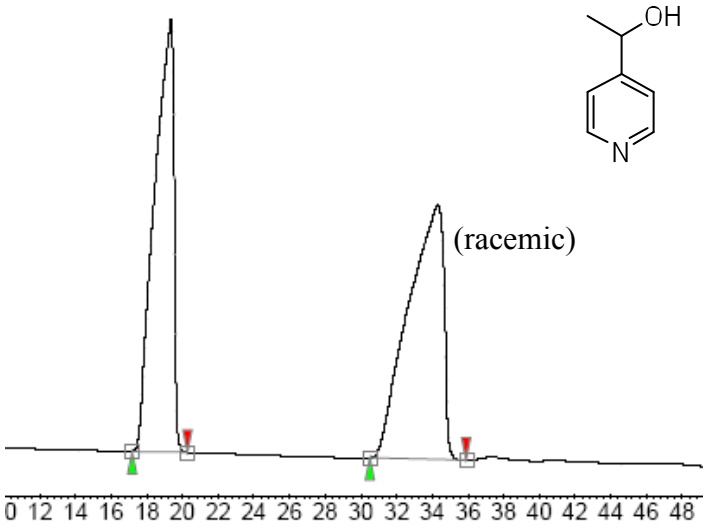
(*R*)

Peak results :

Index	Name	Time [Min]	Quantity [% Area]	Height [mAU]	Area [mAU.Min]	Area % [%]
1	UNKNOWN	7,21	80,61	1266,7	249,0	80,607
2	UNKNOWN	9,33	19,39	250,2	59,9	19,393
Total			100,00	1516,9	308,9	100,000

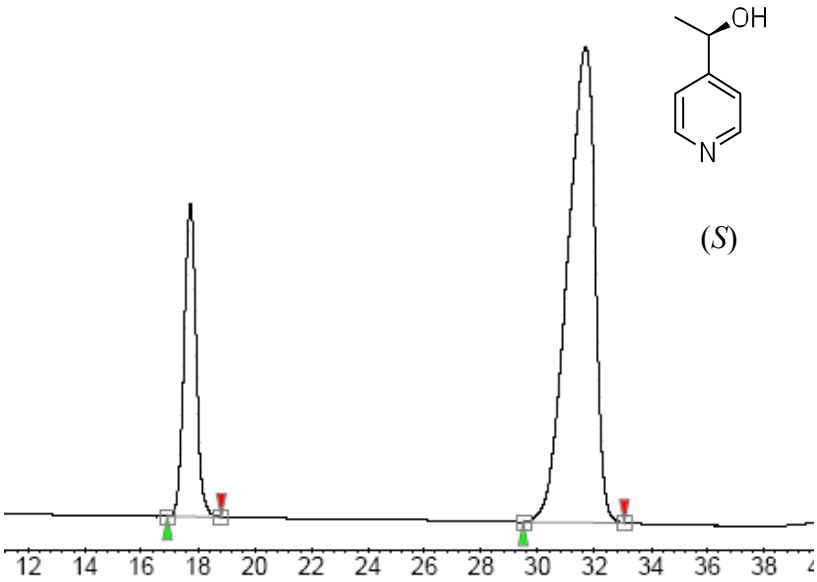
1-(pyridin-4-yl)ethanol

AS-H: heptane/isopropanol (90/10), 1 mL/min, 254 nm



Peak results :

Index	Name	Time [Min]	Quantity [% Area]	Height [mAU]	Area [mAU.Min]	Area % [%]
1	UNKNOWN	19,35	48,39	492,8	623,5	48,389
2	UNKNOWN	34,32	51,61	289,0	665,1	51,611
Total			100,00	781,8	1288,6	100,000

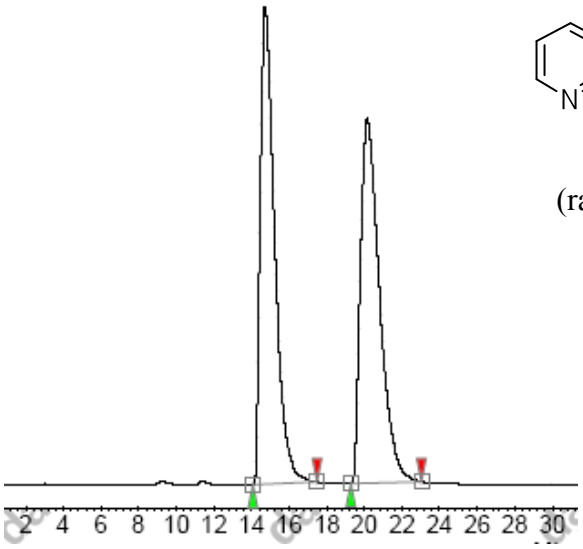


Peak results :

Index	Name	Time [Min]	Quantity [% Area]	Height [mAU]	Area [mAU.Min]	Area % [%]
1	UNKNOWN	17,74	22,40	95,2	47,0	22,404
2	UNKNOWN	31,69	77,60	145,0	162,8	77,596
Total			100,00	240,3	209,7	100,000

8-hydroxy-5, 6, 7, 8-tetrahydroquinoline

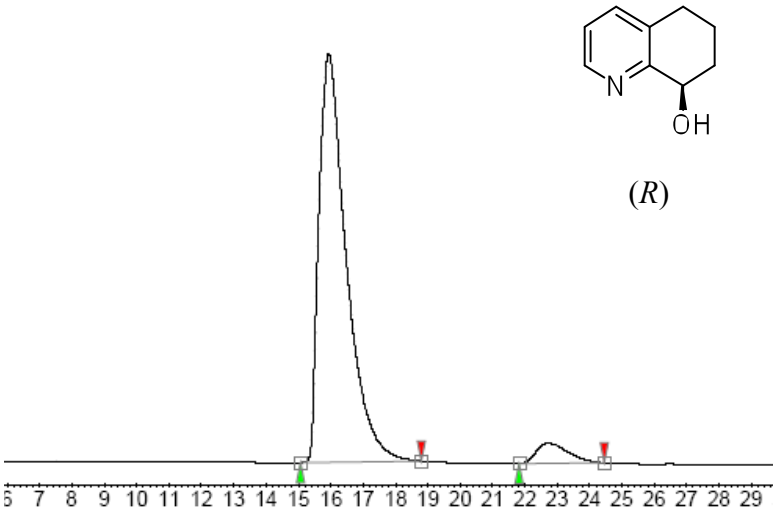
OD-H: heptane/isopropanol (99/1), 1 mL/min, 254 nm



(racemic)

Peak results :

Index	Name	Time [Min]	Quantity [% Area]	Height [mAU]	Area [mAU.Min]	Area % [%]
1	UNKNOWN	14,73	49,97	342,6	306,4	49,969
2	UNKNOWN	20,15	50,03	261,4	306,8	50,031
Total			100,00	604,1	613,2	100,000



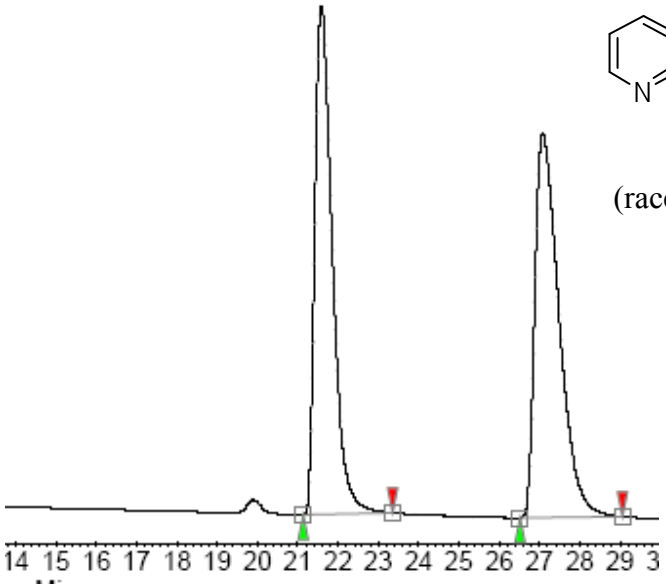
(*R*)

Peak results :

Index	Name	Time [Min]	Quantity [% Area]	Height [mAU]	Area [mAU.Min]	Area % [%]
1	UNKNOWN	15,94	94,56	600,7	587,4	94,563
2	UNKNOWN	22,73	5,44	29,0	33,8	5,437
Total			100,00	629,7	621,2	100,000

9-hydroxy-6, 7, 8, 9-tetrahydro-5H-cycloheptapyridine

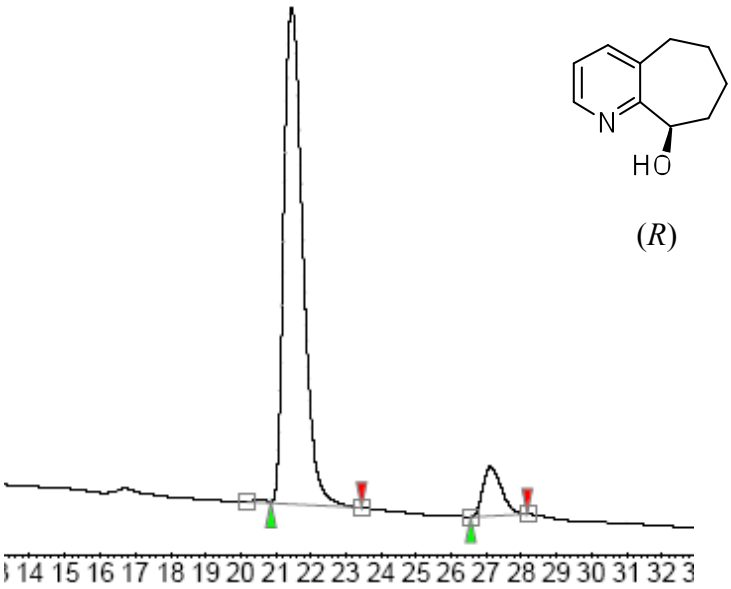
AS-H: heptane/isopropanol (99/1), 0.5 mL/min, 254 nm



(racemic)

Peak results :

Index	Name	Time [Min]	Quantity [% Area]	Height [mAU]	Area [mAU.Min]	Area % [%]
1	UNKNOWN	21,58	48,92	812,9	408,2	48,925
2	UNKNOWN	27,08	51,08	615,3	426,1	51,075
Total			100,00	1428,1	834,3	100,000



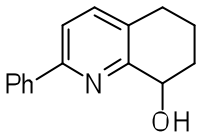
(*R*)

Peak results :

Index	Name	Time [Min]	Quantity [% Area]	Height [mAU]	Area [mAU.Min]	Area % [%]
1	UNKNOWN	21,46	91,17	289,6	181,4	91,167
2	UNKNOWN	27,12	8,83	28,8	17,6	8,833
Total			100,00	318,4	199,0	100,000

8-hydroxy-2-phenyl-5, 6, 7, 8-tetrahydroquinoline

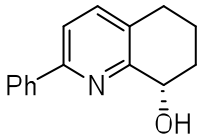
OD-H: heptane/isopropanol (99/1), 1 mL/min, 254 nm

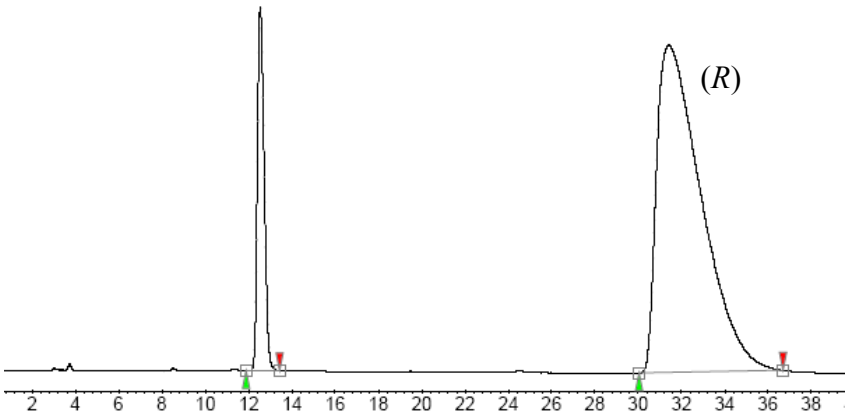


(racemic)

Peak results :

Index	Name	Time [Min]	Quantity [% Area]	Height [mAU]	Area [mAU.Min]	Area % [%]
1	UNKNOWN	12,90	50,34	1110,5	489,6	50,338
2	UNKNOWN	31,13	49,66	267,1	483,0	49,662
Total			100,00	1377,6	972,6	100,000



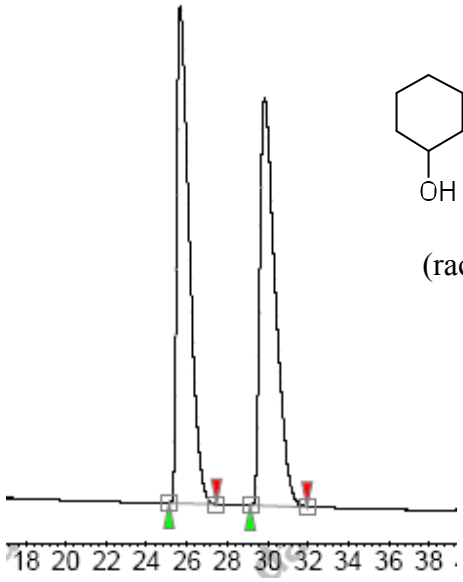


Peak results :

Index	Name	Time [Min]	Quantity [% Area]	Height [mAU]	Area [mAU.Min]	Area % [%]
1	UNKNOWN	12,54	14,31	415,5	148,7	14,306
2	UNKNOWN	31,41	85,69	373,8	890,6	85,694
Total			100,00	789,3	1039,3	100,000

8-hydroxy-2-chloro-5, 6, 7, 8-tetrahydro-2-quionoline

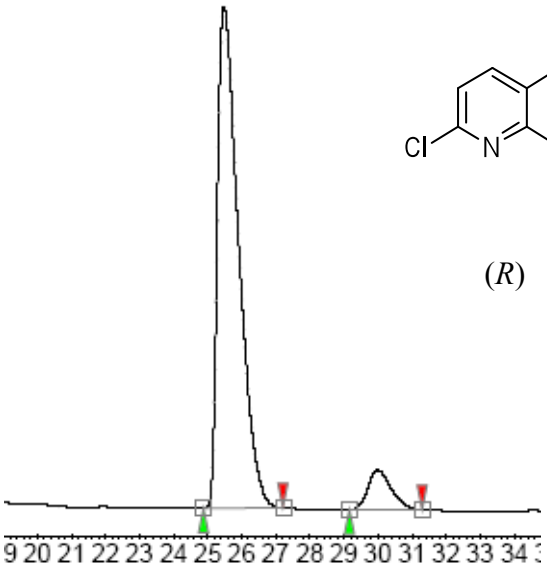
OJ: heptane/isopropanol (99/1), 0.5 mL/min, 254 nm



(racemic)

Peak results :

Index	Name	Time [Min]	Quantity [% Area]	Height [mAU]	Area [mAU.Min]	Area % [%]
1	UNKNOWN	25,67	49,84	401,6	287,8	49,841
2	UNKNOWN	29,84	50,16	328,9	289,6	50,159
Total			100,00	730,5	577,4	100,000



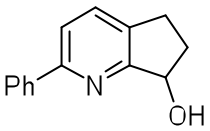
(*R*)

Peak results :

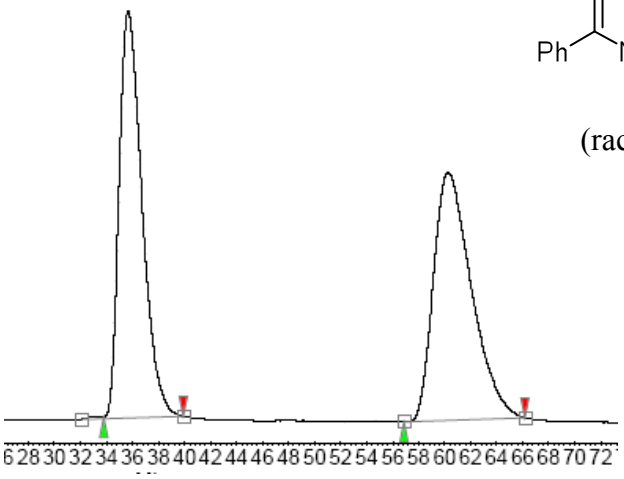
Index	Name	Time [Min]	Quantity [% Area]	Height [mAU]	Area [mAU.Min]	Area % [%]
1	UNKNOWN	25,48	91,96	319,1	228,9	91,959
2	UNKNOWN	30,00	8,04	25,1	20,0	8,041
Total			100,00	344,1	248,9	100,000

2-phenyl-7-hydroxy- 6, 7-dihydro-5H-cyclopentapyridine

OD-H: heptane/isopropanol (99/1), 1 mL/min, 254

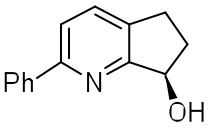


(racemic)



Peak results :

Index	Name	Time [Min]	Quantity [% Area]	Height [mAU]	Area [mAU.Min]	Area % [%]
1	UNKNOWN	35,66	49,76	174,8	356,1	49,763
2	UNKNOWN	60,25	50,24	106,3	359,5	50,237
Total			100,00	281,2	715,6	100,000



(*R*)

Peak results :

Index	Name	Time [Min]	Quantity [% Area]	Height [mAU]	Area [mAU.Min]	Area % [%]
1	UNKNOWN	32,41	76,19	598,7	1091,9	76,191
2	UNKNOWN	57,29	23,81	119,1	341,2	23,809
Total			100,00	717,8	1433,2	100,000

checkCIF/PLATON report

No syntax errors found. **CIF dictionary** **Interpreting this report**

Datablock: j001

Bond precision: C-C = 0.0200 A Wavelength=0.71073

Cell: a=11.1616(18) b=21.663(5) c=11.9040(16)
alpha=90 beta=101.547(17) gamma=90
Temperature: 297 K

	Calculated	Reported
Volume	2820.1(9)	2820.1(9)
Space group	P 21	P 21
Hall group	P 2yb	P 2yb
Moiety formula	C40 H58 Cl6 Fe N4 O6 P2 Sn2	C40 H58 Cl6 Fe N4 O6 P2 Sn2
Sum formula	C40 H58 Cl6 Fe N4 O6 P2 Sn2	C40 H58 Cl6 Fe N4 O6 P2 Sn2
Mr	1258.82	1258.82
Dx,g cm-3	1.482	1.482
Z	2	2
Mu (mm-1)	1.515	1.515
F000	1264.0	1264.0
F000’	1264.60	
h,k,lmax	13,26,14	13,26,13
Nref	5290[10283]	9649
Tmin,Tmax	0.775,0.886	0.698,0.809
Tmin’	0.761	

Correction method= ANALYTICAL

Data completeness= 1.82/0.94 Theta(max)= 25.300

R(reflections)= 0.0554(4594) wR2(reflections)= 0.1286(9649)

S = 0.832 Npar= 550

The following ALERTS were generated. Each ALERT has the format
test-name_ALERT_alert-type_alert-level.
Click on the hyperlinks for more details of the test.

Alert level A
PLAT029_ALERT_3_A _diffrn_measured_fraction_theta_full Low 0.94

Author Response: There is a missing cusp of data due to data collection by rotation around the spindle axis only.

PLAT213_ALERT_2_A Atom Cl3 has ADP max/min Ratio ..... 5.40 prola

Author Response: The Cl atoms, attached to Sn, show positional disorder, which could not be resolved in the case of Cl1, Cl3 and Cl5.

PLAT242_ALERT_2_A Check Low Ueq as Compared to Neighbors for Sn1

Author Response: Sn1 is the central atom, around which the Cl1 to Cl3 are disordered.

PLAT242_ALERT_2_A Check Low Ueq as Compared to Neighbors for Sn2

Author Response: Sn1 is the central atom, around which the Cl1 to Cl3 are disordered.

PLAT242_ALERT_2_A Check Low Ueq as Compared to Neighbors for C3

Author Response: Sn1 is the central atom, around which the Cl1 to Cl3 are disordered.

PLAT242_ALERT_2_A Check Low Ueq as Compared to Neighbors for C36

Author Response: Sn1 is the central atom, around which the Cl1 to Cl3 are disordered.

PLAT234_ALERT_4_A Large Hirshfeld Difference C30 -- C31 .. 0.34 Ang.

Author Response: the benzene rings C8 - C13 and C28 - C33 are oscillating, with the biggest displacement at the atoms C10, C11 and C30, C31 respectively.

Alert level B

PLAT220_ALERT_2_B Large Non-Solvent C Ueq(max)/Ueq(min) ... 4.25 Ratio
PLAT220_ALERT_2_B Large Non-Solvent Cl Ueq(max)/Ueq(min) ... 4.40 Ratio
PLAT241_ALERT_2_B Check High Ueq as Compared to Neighbors for O4
PLAT241_ALERT_2_B Check High Ueq as Compared to Neighbors for C10
PLAT241_ALERT_2_B Check High Ueq as Compared to Neighbors for C12
PLAT241_ALERT_2_B Check High Ueq as Compared to Neighbors for C30
PLAT242_ALERT_2_B Check Low Ueq as Compared to Neighbors for C16

Author Response: Sn1 is the central atom, around which the Cl1 to Cl3 are disordered.

PLAT234_ALERT_4_B Large Hirshfeld Difference O6 -- C34 .. 0.25 Ang.

Author Response: the benzene rings C8 - C13 and C28 - C33 are oscillating, with the biggest displacement at the atoms C10, C11 and C30, C31 respectively.

PLAT234_ALERT_4_B Large Hirshfeld Difference C31 -- C32 .. 0.29 Ang.

Author Response: the benzene rings C8 - C13 and C28 - C33 are oscillating, with the biggest displacement at the atoms C10, C11 and C30, C31 respectively.

PLAT234_ALERT_4_B Large Hirshfeld Difference C36 -- C37 .. 0.26 Ang.

Author Response: the benzene rings C8 - C13 and C28 - C33 are oscillating, with the biggest displacement at the atoms C10, C11 and C30, C31 respectively.

Alert level C

PLAT026_ALERT_3_C Ratio Observed / Unique Reflections too Low .... 48 Perc.
PLAT213_ALERT_2_C Atom Cl1 has ADP max/min Ratio ..... 3.70 prola

Author Response: The Cl atoms, attached to Sn, show positional disorder, which could not be resolved in the case of Cl1, Cl3 and Cl5.

PLAT213_ALERT_2_C Atom Cl5 has ADP max/min Ratio ..... 3.60 prola

Author Response: The Cl atoms, attached to Sn, show positional disorder, which could not be resolved in the case of Cl1, Cl3 and Cl5.

PLAT213_ALERT_2_C Atom C10 has ADP max/min Ratio ..... 3.10 prola

Author Response: The Cl atoms, attached to Sn, show positional disorder, which could not be resolved in the case of Cl1, Cl3 and Cl5.

PLAT213_ALERT_2_C Atom C19 has ADP max/min Ratio ..... 3.40 prola

Author Response: The Cl atoms, attached to Sn, show positional disorder, which could not be resolved in the case of Cl1, Cl3 and Cl5.

PLAT222_ALERT_3_C Large Non-Solvent H Ueq(max)/Ueq(min) ... 3.38 Ratio
PLAT241_ALERT_2_C Check High Ueq as Compared to Neighbors for O6
PLAT242_ALERT_2_C Check Low Ueq as Compared to Neighbors for P2

Author Response: Sn1 is the central atom, around which the Cl1 to Cl3 are disordered.

PLAT242_ALERT_2_C Check Low Ueq as Compared to Neighbors for C8

Author Response: Sn1 is the central atom, around which the Cl1 to Cl3 are disordered.

PLAT242_ALERT_2_C Check Low Ueq as Compared to Neighbors for C13

Author Response: Sn1 is the central atom, around which the Cl1 to Cl3 are disordered.

PLAT242_ALERT_2_C Check Low Ueq as Compared to Neighbors for C23

Author Response: Sn1 is the central atom, around which the Cl1 to Cl3 are disordered.

PLAT242_ALERT_2_C Check Low Ueq as Compared to Neighbors for C28

Author Response: Sn1 is the central atom, around which the Cl1 to Cl3 are disordered.

PLAT242_ALERT_2_C Check Low Ueq as Compared to Neighbors for C29

Author Response: Sn1 is the central atom, around which the Cl1 to Cl3 are disordered.

PLAT331_ALERT_2_C Small Average Phenyl C-C Dist. C28 -C33 1.36 Ang.
PLAT342_ALERT_3_C Low Bond Precision on C-C Bonds (x 1000) Ang .. 20
PLAT234_ALERT_4_C Large Hirshfeld Difference Sn1 -- Cl1 .. 0.18 Ang.

Author Response: the benzene rings C8 - C13 and C28 - C33 are oscillating, with the biggest displacement at the atoms C10, C11 and C30, C31 respectively.

PLAT234_ALERT_4_C Large Hirshfeld Difference Sn2 -- Cl5 .. 0.18 Ang.

Author Response: the benzene rings C8 - C13 and C28 - C33 are oscillating, with the biggest displacement at the atoms C10, C11 and C30, C31 respectively.

PLAT234_ALERT_4_C Large Hirshfeld Difference P2 -- O5 .. 0.16 Ang.

Author Response: the benzene rings C8 - C13 and C28 - C33 are oscillating, with the biggest displacement at the atoms C10, C11 and C30, C31 respectively.

PLAT234_ALERT_4_C Large Hirshfeld Difference P2 -- O6 .. 0.18 Ang.

Author Response: the benzene rings C8 - C13 and C28 - C33 are oscillating, with the biggest displacement at the atoms C10, C11 and C30, C31 respectively.

PLAT234_ALERT_4_C Large Hirshfeld Difference O4 -- C27 .. 0.21 Ang.

Author Response: the benzene rings C8 - C13 and C28 - C33 are oscillating, with the biggest displacement at the atoms C10, C11 and C30, C31 respectively.

PLAT234_ALERT_4_C Large Hirshfeld Difference N2 -- C15 .. 0.17 Ang.

Author Response: the benzene rings C8 - C13 and C28 - C33 are oscillating, with the biggest displacement at the atoms C10, C11 and C30, C31 respectively.

PLAT234_ALERT_4_C Large Hirshfeld Difference N2 -- C20 .. 0.16 Ang.

Author Response: the benzene rings C8 - C13 and C28 - C33 are oscillating, with the biggest displacement at the atoms C10, C11 and C30, C31 respectively.

PLAT234_ALERT_4_C Large Hirshfeld Difference N3 -- C21 .. 0.17 Ang.

Author Response: the benzene rings C8 - C13 and C28 - C33 are oscillating, with the biggest displacement at the atoms C10, C11 and C30, C31 respectively.

PLAT234_ALERT_4_C Large Hirshfeld Difference C14 -- C15 .. 0.22 Ang.

Author Response: the benzene rings C8 - C13 and C28 - C33 are oscillating, with the biggest displacement at the atoms C10, C11 and C30, C31 respectively.

PLAT234_ALERT_4_C Large Hirshfeld Difference C15 -- C16 .. 0.20 Ang.

Author Response: the benzene rings C8 - C13 and C28 - C33 are oscillating, with the biggest displacement at the atoms C10, C11 and C30, C31 respectively.

PLAT234_ALERT_4_C Large Hirshfeld Difference C16 -- C19 .. 0.22 Ang.

Author Response: the benzene rings C8 - C13 and C28 - C33 are oscillating, with the biggest displacement at the atoms C10, C11 and C30, C31 respectively.

PLAT234_ALERT_4_C Large Hirshfeld Difference C28 -- C29 .. 0.19 Ang.

Author Response: the benzene rings C8 - C13 and C28 - C33 are oscillating, with the biggest displacement at the atoms C10, C11 and C30, C31 respectively.

PLAT234_ALERT_4_C Large Hirshfeld Difference C28 -- C33 .. 0.22 Ang.

Author Response: the benzene rings C8 - C13 and C28 - C33 are oscillating, with the biggest displacement at the atoms C10, C11 and C30, C31 respectively.

PLAT234_ALERT_4_C Large Hirshfeld Difference C32 -- C33 .. 0.23 Ang.

Author Response: the benzene rings C8 - C13 and C28 - C33 are oscillating, with the biggest displacement at the atoms C10, C11 and C30, C31 respectively.

PLAT234_ALERT_4_C Large Hirshfeld Difference C36 -- C39 .. 0.25 Ang.

Author Response: the benzene rings C8 - C13 and C28 - C33 are oscillating, with the biggest displacement at the atoms C10, C11 and C30, C31 respectively.

PLAT775_ALERT_1_C Suspect X-Y Cont in CIF: FE1 -- CL3 .. 4.05 Ang.
PLAT775_ALERT_1_C Suspect X-Y Cont in CIF: CL3 -- FE1 .. 4.05 Ang.
PLAT790_ALERT_4_C Centre of Gravity not Within Unit Cell: Resd. # 1
C40 H58 Cl6 Fe N4 O6 P2 Sn2

Alert level G

REFLT03_ALERT_4_G Please check that the estimate of the number of Friedel pairs is correct. If it is not, please give the correct count in the _publ_section_exptl_refinement section of the submitted CIF.
From the CIF: _diffrn_reflns_theta_max 25.30
From the CIF: _reflns_number_total 9649
Count of symmetry unique reflns 5290
Completeness (_total/calc) 182.40%
TEST3: Check Friedels for noncentro structure
Estimate of Friedel pairs measured 4359
Fraction of Friedel pairs measured 0.824
Are heavy atom types Z>Si present yes
PLAT301_ALERT_3_G Note: Main Residue Disorder ..... 2.00 Perc.
PLAT791_ALERT_4_G The Model has Chirality at C2 (Verify) .... S
PLAT791_ALERT_4_G The Model has Chirality at C15 (Verify) .... S
PLAT791_ALERT_4_G The Model has Chirality at C22 (Verify) .... S
PLAT791_ALERT_4_G The Model has Chirality at C35 (Verify) .... S

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7 **ALERT level A** = In general: serious problem
10 **ALERT level B** = Potentially serious problem
33 **ALERT level C** = Check and explain
6 **ALERT level G** = General alerts; check

2 ALERT type 1 CIF construction/syntax error, inconsistent or missing data
24 ALERT type 2 Indicator that the structure model may be wrong or deficient
5 ALERT type 3 Indicator that the structure quality may be low
25 ALERT type 4 Improvement, methodology, query or suggestion
0 ALERT type 5 Informative message, check

Publication of your CIF in IUCr journals

A basic structural check has been run on your CIF. These basic checks will be run on all CIFs submitted for publication in IUCr journals (*Acta Crystallographica, Journal of Applied Crystallography, Journal of Synchrotron Radiation*); however, if you intend to submit to *Acta Crystallographica Section C* or *E*, you should make sure that full publication checks are run on the final version of your CIF prior to submission.

Publication of your CIF in other journals

Please refer to the *Notes for Authors* of the relevant journal for any special instructions relating to CIF submission.

PLATON version of 13/08/2009; check.def file version of 12/08/2009

Datablock j001 - ellipsoid plot