Electronic Supplementary Material (ESI) for ChemComm.

Asymmetric synthesis of functionalized cyclohexanes bearing five stereocenters *via* a one-pot organocatalytic Michael/Michael/1,2-addition sequence

Pankaj Chauhan,[a] Gregor Urbanietz,[a] Gerhard Raabe[b] and Dieter Enders[a]*

[a] Institut of Organic Chemistry, RWTH Aachen University Landoltweg 1, 52074 Aachen (Germany)
E-mail: enders@rwth-aachen.de

Contents

General Methods and Materials:

All reactions were performed in oven-dried glassware. Analytical TLC were performed using SIL G-25 UV254 from MACHERY NAGEL and visualized either with ultraviolet radiation at 254 nm. ^{1}H and ^{13}C NMR spectra were recorded at ambient temperatures on a Varian Innova 600 or Varian Innova 400 instrument with tetramethylsilane as the internal standard. Chemical shifts for ^{1}H-NMR and ^{13}C-NMR are reported in parts per million (ppm), with coupling constants reported in Hertz (Hz). The following abbreviations are used for spin multiplicity: s = singlet, br s = broad singlet, d = doublet, dd = doublet of doublet, t = triplet and m = multiplet. Mass spectra were acquired on a Finnigan SSQ7000 (EI 70 eV) spectrometer, high resolution mass spectra (HRMS) on a Finnigan MAT 95 and high resolution ESI spectra on a ThermoFisher Scientific LTQ-Orbitrap XL. IR spectra were taken on a PerkinElmer Spectrum 100 FT-IR Spectrometer. Elemental analyses were performed with a Vario EL elemental analyzer. Analytical HPLC were carried out either on a Hewlett-Packard 1050 Series instrument or Agilent 1100 instrument using chiral stationary phases. Optical rotation values were measured on a Perkin-Elmer 241 polarimeter.

Starting materials and reagents were purchased directly from commercial suppliers and used without further purifications. All solvents used as reaction medium were distilled before the use. The nitroalkenes **2**[1] and α,α-dicyano-olefins **3**[2] were synthesized using known literature procedures. The chiral squaramides **I** and **II** were also synthesized using known literature procedures.[3]

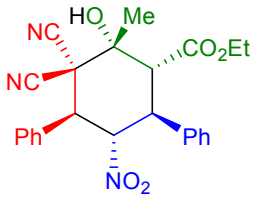
General Procedure for the organocatalytic one-pot Michael/Michael/1,2-additon reactions:

In a 10 mL round bottom flask equipped with a magnetic stirring bar, the nitroalkene **2** (1.0 equiv., 0.5 mmol) and catalyst **I** or **II** (1 mol%) were dissolved in CH_2Cl_2 (1.25 mL) and stirred 5 minutes at room temperature followed by the addition of the β-keto ester **1** (1.0 equiv. 0.5 mmol). After stirring the reaction mixture at room temperature for 24 hours the α,α-dicyano-olefins **3** (1.2 equiv., 0.6 mmol) and TBD (20 mol%; 0.1 M in CH_2Cl_2) were added subsequently and stirred for another 24 hours at room temperature. The crude product

[1] (a) Organic Syntheses, Coll. *Vol. 1,* p.413 (**1941**); *Vol. 9,* p.66 (**1929**); (b) B. M. Trost and C. Müller, *J. Am. Chem. Soc.* 2008, **130**, 2438.
[2] Y. Lv, X. Yan, L. Yan, Z. Wang, J. Chen, H. Deng, M. Shao, H. Zhang and W. Cao, *Tetrahedron,* 2013, **69**, 4205.
[3] J. P. Malerich, K. Hagihara and V. H. Rawal, *J. Am. Chem. Soc.,* 2008, **130,** 14416.

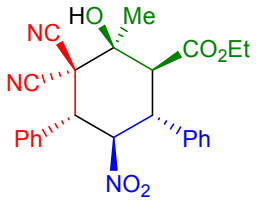
was directly purified by flash column chromatographies (first *n*-hexane/EtOAc = 9:1, then *n*-hexane/EtOAc = 4:1) to afford the polysubstituted cyclohexanes **4a-4p**.

Analytic data:



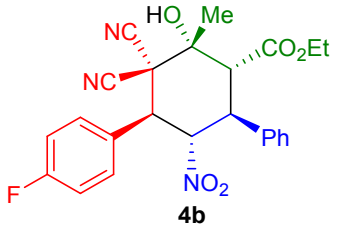
4a

Compound **4a** was synthesized by catalyst **I** and isolated as a white solid (156 mg, 72%); Mp = 208-210 °C; $[\alpha]_{24}^{D}$ = +20.4 (c = 0.5, $CHCl_3$); >30:1 dr; 99% ee (major diastereomer); HPLC (major diastereomer): tR 6.35 min (major) 8.97 min (minor), 230 nm, *n*-heptane/EtOH, 9:1, 1.00 mL/min, Chiralpak AS column; IR (Capillary): 3814, 3417, 2983, 2451, 2275, 2176, 2057, 1976, 1890, 1705, 1563, 1458, 1343, 1211, 1109, 1060, 1016, 898, 847, 754, 696 cm^{-1}; ^1H NMR(600 MHz, $CDCl_3$, major diastereomer): δ = 7.54 (br s, 2H, ArH), 7.43-7.42 (m, 3H, ArH), 7.36-7.31 (m, 3H, ArH), 7.25 (br s, 2H, ArH), 5.42 (t, *J* = 11.7 Hz, 1H, CH), 4.62 (s, 1H, OH), 4.46 (d, *J* = 11.7 Hz, 1H, CH), 3.96 (t, *J* = 12.0 Hz, 1H, CH), 3.91-3.87 (m, 2H, CH_2), 3.31 (d, *J* = 12.6 Hz, 1H, CH), 1.72 (s, 3H, CH_3), 0.78 (t, *J* = 7.2 Hz, 3H, CH_3); ^{13}C NMR (151 MHz, $CDCl_3$, major diastereomer): δ = 171.9, 133.6, 130.8, 130.3 (2C), 129.4 (4C), 129.2 (4C), 112.7, 111.3, 89.1, 73.7, 62.2, 52.3, 51.2, 47.3, 46.4, 25.7, 13.4 ppm; MS (ESI, pos): m/z (%) 456.1 (100) $[M+Na]^+$; Anal. Calcd for $C_{24}H_{23}N_3O_5$: C, 66.50; H, 5.35; N, 9.69. Found: C, 66.26; H, 5.34; N 9.75.



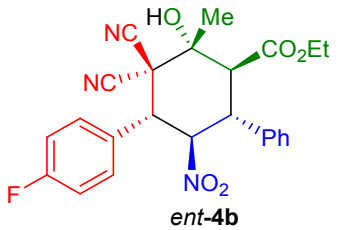
ent-**4a**

Compound *ent*-**4a** was synthesized by catalyst **II** and isolated as a white solid (151 mg, 70%); >30:1 dr; 96% ee (major diastereomer); HPLC (major diastereomer): tR 6.34 min (minor) 9.13 min (major), *n*-heptane/EtOH, 9:1, 1.00 mL/min, Chiralpak AS column.



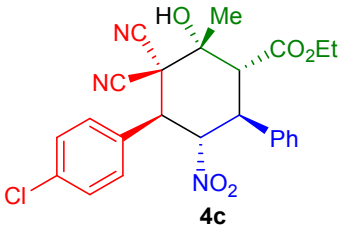
4b

Compound **4b** synthesized by catalyst **I** and isolated as a white solid (172 mg, 76%); Mp = 170-171°C; $[\alpha]_{24}^{D}$ = +20.4 (c = 0.5, $CHCl_3$); >30:1 dr; 99% ee (major diastereomer); HPLC (major diastereomer): tR 8.79 min (major), 11.44 min (minor), *n*-heptane/ethanol, 9:1, 0.70 mL/min, Chiralpak-AS column; IR (Capillary): 3826, 3427, 2984, 2646, 2480, 2179, 2034, 1985, 1899, 1704, 1606, 1565, 1513, 1459, 1343, 1231, 1108, 1016, 897, 841, 758, 695 cm^{-1}; ^1H NMR(400 MHz, $CDCl_3$, major diastereomer): δ = 7.56-7.52 (m, 2H, ArH), 7.34-7.24 (m, 5H, ArH), 7.14-7.09 (m, 2H, ArH), 5.36 (t, *J* = 11.6 Hz, 1H, CH), 4.69 (s, 1H, OH), 4.47 (d, *J* = 12.0 Hz, 1H, CH), 4.03-3.83 (m, 3H, CH, CH_2), 3.29 (d, *J* = 12.4 Hz, 1H, CH), 1.71 (s, 3H, CH_3), 0.77 (t, *J* = 7.2 Hz, 3H, CH_3); ^{13}C NMR (101 MHz, $CDCl_3$, major diastereomer): δ = 171.7, 163.7 (*J* = 25.1 Hz), 133.5, 129.2, 126.7, 126.6, 116.7, 116.5, 112.6, 111.2, 89.2, 73.7, 62.3, 52.3, 51.2, 46.7, 46.3, 25.7, 13.3 ppm; MS (ESI, pos): m/z 474.1 $[M+Na]^+$; Anal. Calcd for $C_{24}H_{22}FN_3O_5$: C 63.85; H 4.91; N 9.31; found: C 63.96; H 4.82; N 9.35.



***ent*-4b**

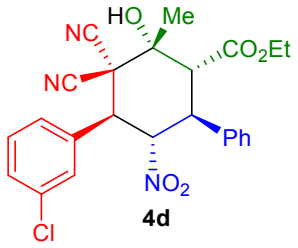
Compound *ent*-**4b** synthesized by catalyst **II** and isolated as a white solid (174 mg, 77%); Mp >30:1 dr; 97% ee (major diastereomer); HPLC (major diastereomer): tR 8.93 min (minor), 11.61 min (major), *n*-heptane/ethanol, 9:1, 0.70 mL/min, Chiralpak-AS column.



4c

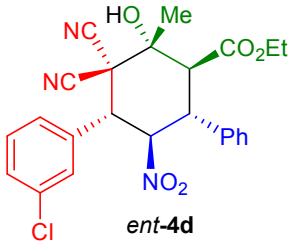
Compound **4c** was synthesized by catalyst **I** and isolated as a white solid (167 mg, 72%); Mp = 179-181 °C; $[\alpha]_{24}^{D}$ = +28.0 (c = 0.5, $CHCl_3$); >30:1 dr; 99% ee (major diastereomer); HPLC (major diastereomer): tR 13.77 min (major), 16.75 min (minor), *n*-heptane/ethanol,

97:3, 1.00 mL/min, Chiralcel-OD column. IR (Capillary): 3827, 3440, 2987, 2644, 2443, 2213, 2066, 1979, 1904, 1710, 1564, 1492, 1457, 1371, 1215, 1100, 1016, 897, 837, 763, 694 cm^{-1}; ^{1}H NMR(600 MHz, MeOH-d_4, major diastereomer): δ = 7.61-7.60 (m, 2H, ArH), 7.46-7.44 (m, 2H, ArH), 7.32-7.27 (m, 5H, ArH), 5.56 (t, *J* = 11.4 Hz, 1H, CH), 4.40 (d, *J* = 11.4 Hz, 1H, CH), 4.09 (t, dd, *J* = 12.6, 11.4 Hz, 1H, CH), 3.90 (q, *J* = 7.1 Hz, 2H, CH_2), 3.34 (d, *J* = 12.6 Hz, 1H, OH), 3.30 (t, *J* = 1.7 Hz, 1H, CH), 1.74 (s, 3H, CH_3), 0.9 (t, *J* = 6.9 Hz, 3H, CH_3); ^{13}C NMR (151 MHz, MeOH-d_4, major diastereomer): δ = 168.4, 135.8, 135.3, 130.7, 128.9, 128.8, 128.6, 128.5, 128.3, 112.9, 112.3, 89.5, 73.3, 60.9, 53.5, 52.4, 46.9, 45.4, 23.6, 12.7 ppm; MS (ESI, pos): m/z 490.11560 $[M+Na]^+$; Anal. Calcd for $C_{24}H_{22}ClN_3O_5$: C 61.61; H 4.74; N 8.98; found: C 61.60; H 4.90; N 8.78.



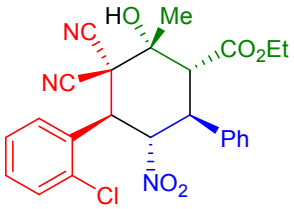
4d

Compound **4d** was synthesized by catalyst **I** and isolated as a white solid (172 mg, 74%); Mp = 203-204 °C; $[\alpha]_{24}^{D}$ = +25.0 (c = 0.5, $CHCl_3$); >30:1 dr; 99% ee (major diastereomer); HPLC (major diastereomer): tR 11.95 min (major), 16.99 min (minor), *n*-heptane/ethanol, 9:1, 0.50 mL/min, Chiralpak-AS column; IR (Capillary): 3421, 3070, 3035, 2983, 2938, 2651, 2322, 2167, 2064, 1983, 1951, 1886, 1703, 1596, 1565, 1461, 1375, 1339, 1293, 1248, 1215, 1190, 1095, 1166, 1014, 967, 912, 888, 866, 846, 607, 770, 741, 695 cm^{-1}; ^{1}H NMR(600 MHz, $CDCl_3$, major diastereomer): δ = 7.54 (s, 1H, ArH), 7.46-7.33 (m, 6H, ArH), 7.25 (br s, 2H, ArH), 5.37 (t, *J* = 11.7 Hz, 1H, CH), 4.67 (s, 1H, OH), 4.44 (d, *J* = 12.0 Hz, 1H, CH), 3.98-3.87 (m, 3H, CH, CH_2), 3.30 (d, *J* = 12.6 Hz, 1H, CH), 1.72 (s, 3H, CH_3), 0.78 (t, *J* = 7.2 Hz, 3H, CH_3); ^{13}C NMR (151 MHz, $CDCl_3$, major diastereomer): δ = 171.7, 135.3, 133.4, 132.7, 130.7, 130.6, 129.3, 127.8, 127.9, 112.4, 111.7, 89.0, 73.7, 62.3, 52.3, 50.8, 46.9, 46.3, 25.6, 13.4 ppm; MS (ESI, pos): m/z 490.20959 $[M+Na]^+$; Anal. Calcd for $C_{24}H_{22}ClN_3O_5$: C 61.61; H 4.74; N 8.98; found: C 61.62; H 4.47; N 8.81.



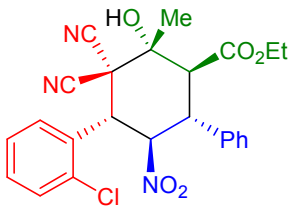
ent-**4d**

Compound *ent*-**4d** was synthesized by catalyst **II** and isolated as a white solid (175 mg, 75%); >30:1 dr; 98% ee (major diastereomer); HPLC (major diastereomer): tR 11.98 min (minor), 17.60 min (major), *n*-heptane/ethanol, 9:1, 0.50 mL/min, Chiralpak-AS column.



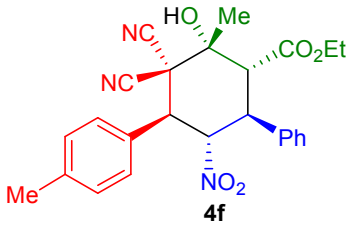
4e

Compound **4e** was synthesized by catalyst **I** and isolated as a white solid (164 mg, 70%); Mp = 187-188°C; $[\alpha]_{24}^{D}$ = +35.2 (c = 0.5, $CHCl_3$); >30:1 dr; 99% ee (major diastereomer); HPLC (major diastereomer): tR 14.26 min (major), 17.72 min (minor), *n*-heptane/*i*-PrOH, 95:5, 0.70 mL/min, Chiralcel-OD column; IR (Capillary): 3435, 3071, 3035, 2986, 2649, 2292, 2190, 2067, 1980, 1890, 1828, 1711, 1561, 1469, 1372, 1340, 1280, 1219, 1197, 1114, 1022, 956, 914, 865, 840, 805 755 698 cm^{-1}; ^1H NMR (600 MHz, $CDCl_3$, major diastereomer): δ = 7.80-7.78 (m, 1H, ArH), 7.50-7.40 (m, 1H, ArH), 7.39-7.25 (m, 7H, ArH), 5.52 (d, *J* = 11.4 Hz, 1H, CH), 5.36 (t, *J* = 11.7 Hz, 1H, CH), 4.68 (s, 1H, OH), 4.04 (t, *J* = 11.7 Hz, 1H, CH), 3.92-3.85 (m, 2H, CH_2), 3.32 (d, *J* = 12.6 Hz, 1H, CH), 1.73 (s, 3H, CH_3), 0.79 (t, *J* = 7.2 Hz, 3H, CH_3); ^{13}C NMR (151 MHz, $CDCl_3$, major diastereomer): δ = 171.4, 136.1, 133.5, 131.2, 130.9, 129.2, 128.7, 128.0, 127.7, 112.7, 110.7, 89.1, 73.8, 62.2, 52.4, 50.2, 46.5, 41.6, 25.4, 13.3 ppm; MS (ESI, pos): m/z 468.1309 $[M+H]^+$; HRMS Calcd for $[C_{24}H_{22}ClN_3O_5+H]^+$: 468.1321 found: 468.1298.



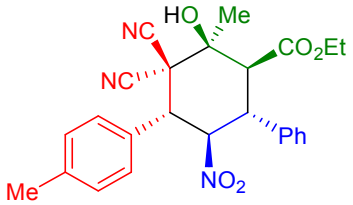
ent-**4e**

Compound *ent*-**4e** was synthesized by catalyst **II** and isolated as a white solid (180 mg, 77%); >30:1 dr; 98% ee (major diastereomer); HPLC (major diastereomer): tR 14.79 min (minor), 17.82 min (major), *n*-heptane/*i*-PrOH, 95:5, 0.70 mL/min, Chiralcel-OD column.



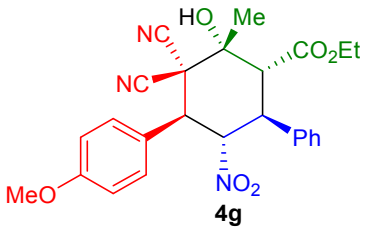
4f

Compound **4f** was synthesized by catalyst **I** and isolated as a white solid (189 mg, 84%); Mp = 200-201 °C; $[\alpha]_{24}^{D}$ = +26.4 (c = 0.5, $CHCl_3$); >30:1 dr; 99% ee (major diastereomer); HPLC (major diastereomer): tR 15.50 min (minor), 20.23 min (major), *n*-heptane/*i*-PrOH, 95:5, 0.7 mL/min, Chiralpak-AD column; IR (Capillary): 3436, 3034, 2987, 2287, 2183, 2067, 1987, 1911, 1804, 1707, 1614, 1564, 1515, 1457, 1374, 1340, 1192, 1115, 1062, 1018, 967, 896, 832, 775, 696 cm^{-1}; ^1H NMR(600 MHz, $CDCl_3$, major diastereomer): δ = 7.43 (d, *J* = 7.2 Hz, 2H, ArH), 7.34-7.22 (m, 7H, ArH), 5.41 (t, *J* = 11.7 Hz, 1H, CH), 4.66 (s, 1H, OH), 4.44 (d, *J* = 11.4 Hz, 1H, CH), 3.98 (t, *J* = 11.7 Hz, 1H, CH), 3.91-3.86 (m, 2H, CH_2), 3.31 (d, *J* = 12.0 Hz, 1H, CH), 2.34 (s, 3H, CH_3), 1.72 (s, 3H, CH_3), 0.78 (t, *J* = 7.2 Hz, 3H, CH_3); ^{13}C NMR (151 MHz, $CDCl_3$, major diastereomer): δ = 171.8, 140.3, 133.7, 130.1, 130.0, 129.2, 129.1, 128.7, 127.7, 112.7, 111.4, 89.2, 73.6, 62.2, 52.3, 51.3, 47.0, 46.3, 25.6, 21.1, 13.3 ppm; MS (ESI, pos): m/z 470.1 $[M+Na]^+$; Anal. Calcd for $C_{25}H_{25}N_3O_5$: C 67.10; H 5.63; N 9.39; found: C 66.84; H 5.63; N 9.46.



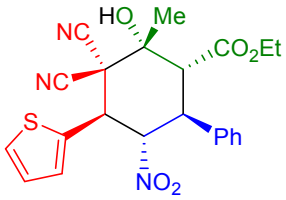
ent-**4f**

Compound *ent*-**4f** was synthesized by catalyst **II** and isolated as a white solid (193 mg, 86%); >30:1 dr; 96% ee (major diastereomer); HPLC (major diastereomer): tR 15.15 min (major), 19.66 min (minor), *n*-heptane/*i*-PrOH, 95:5, 0.7 mL/min, Chiralpak-AD column.



4g

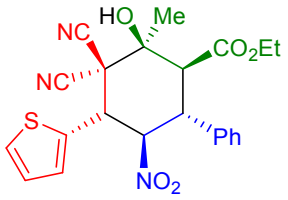
Compound **4g** was synthesized by catalyst **I** and isolated as a white solid (185 mg, 80%); Mp = 218-220 °C; $[\alpha]_{24}^{D}$ = +24.6 (c = 0.5, $CHCl_3$); >30:1 dr; 99% ee (major diastereomer); HPLC (major diastereomer): tR 11.76 min (minor), 14.63 min (major), *n*-heptane/*i*-PrOH, 9:1, 0.70 mL/min, Chiralpak-AD column; IR (Capillary): 3869, 3444, 3060, 2982, 2841, 2647, 2287, 2225, 2190, 2074, 1994, 1983, 1816, 1704, 1612, 1563, 1512, 1459, 1376, 1347, 1254, 1188, 1116, 1064, 1021, 962, 924, 894, 836, 763, 701, 676; ^{1}H NMR(600 MHz, $CDCl_3$, major diastereomer): δ = 7.47 (d, *J* = 7.8 Hz, 2H, ArH), 7.34-7.25 (m, 5H, ArH), 6.93 (d, *J* = 9.0 Hz, 2H, ArH), 5.38 (t, *J* = 11.7 Hz, 1H, CH), 4.66 (s, 1H, OH), 4.43 (d, *J* = 12.0 Hz, 1H, CH), 3.98 (t, *J* = 11.7 Hz, 1H, CH), 3.90-3.86 (m, 2H, CH_2), 3.79 (s, 3H, CH_3), 3.30 (d, *J* = 12.0 Hz, 1H, CH), 1.71 (s, 3H, CH_3), 0.78 (t, *J* = 7.2 Hz, 3H, CH_3); ^{13}C NMR (151 MHz, $CDCl_3$, major diastereomer): δ = 171.7, 160.8, 133.7, 130.2, 129.2, 129.1, 122.5, 114.7 112.8, 111.5, 89.3, 73.6, 62.1, 55.2, 52.3, 51.5, 46.7, 46.3, 25.6, 13.3 ppm; MS (EI): m/z 463.4 $[M+1]^+$; HRMS Calcd for $[C_{25}H_{25}N_3O_6+H]^+$: 464.1816 found: 464.1802.



4h

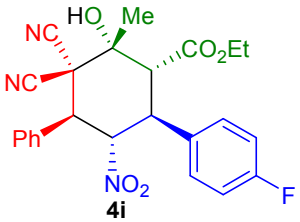
Compound **4h** was synthesized by catalyst **I** and isolated as a white solid (175 mg, 80%); Mp = 188-189 °C; $[\alpha]_{24}^{D}$ = +28.4 (c = 0.5, $CHCl_3$); >30:1 dr; 99% ee (major diastereomer); HPLC (major diastereomer): tR 12.68 min (major), 14.41 min (minor), *n*-heptane/ethanol, 9:1, 0.50 mL/min, Chiralcel-OD column; IR (Capillary): 3863, 3422, 3075, 3037, 2983, 2656, 2289, 2190, 2112, 1980, 1910, 1705, 1563, 1497, 1458, 1373, 1340, 1218, 1192, 1149, 1111, 1015, 970, 892, 849, 754, 703 cm^{-1}; ^{1}H NMR (600 MHz, $CDCl_3$, major diastereomer): δ = 7.39 (d, *J* = 4.8 Hz, 1H, ArH), 7.34-7.30 (m, 4H, ArH), 7.25-7.21 (m, 2H, ArH), 7.05 (t, *J* = 4.2 Hz, 1H, ArH), 5.27 (t, *J* = 11.7 Hz, 1H, CH), 4.80 (d, *J* = 11.4 Hz, 1H, CH), 4.65 (s, 1H, OH), 3.97 (d, *J* = 11.7 Hz, 1H, CH), 3.94-3.83 (m, 2H, CH_2), 3.31 (d, *J* = 12.0 Hz, 1H, CH), 1.72 (s, 3H, CH_3), 0.77 (t, *J* = 7.2 Hz, 3H, CH_3); ^{13}C NMR (151 MHz, $CDCl_3$, major

diastereomer): δ = 171.7, 133.5, 132.3, 129.3, 129.2, 128.0, 127.9, 127.8 127.7, 127.4, 112.7, 111.3, 90.8, 73.7, 62.2, 52.2, 51.7, 46.4, 43.5, 25.7, 13.3 ppm; MS (ESI, pos): m/z 440.1 $[M+1]^+$; Anal. Calcd for $C_{22}H_{21}N_3O_5S$: C 60.12; H 4.82; N 9.56, found: C 59.97; H 4.88; N 9.39.



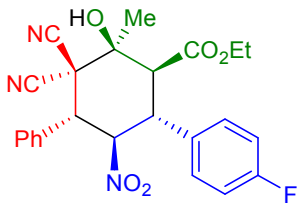
***ent*-4h**

Compound *ent*-**4h** was synthesized by catalyst **II** and isolated as a white solid (178 mg, 81%); >30:1 dr; 99% ee (major diastereomer); HPLC (major diastereomer): tR 12.65 min (minor), 14.31 min (major), *n*-heptane/ethanol, 9:1, 0.50 mL/min, Chiralcel-OD column;



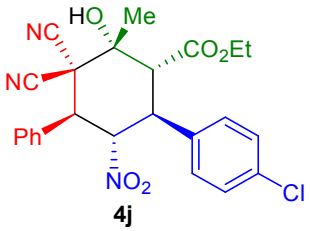
4i

Compound **4i** was synthesized by catalyst **I** and isolated as a white solid (167 mg, 74%); Mp = 197-199 °C; $[\alpha]_{24}^{D}$ = + 20.2 (c = 0.5, $CHCl_3$); >30:1 dr; 99% ee; HPLC (major diastereomer): tR 5.73 min (major), 6.47 min (minor), *n*-heptane/ethanol, 97:3, 0.70 mL/min, Chiralpak-IC column; IR (Capillary): 3408, 2985, 2278, 2095, 1891, 1703, 1607, 1564, 1511 1457, 1358, 1217, 1109, 1014, 900, 825, 735, 699 cm^{-1}; ^1H NMR(600 MHz, $CDCl_3$, major diastereomer): δ = 7.54 (br s, 2H, ArH), 7.47-7.39 (m, 3H, ArH), 7.25 (br s, 2H, ArH), 7.05 (s, 2H, ArH), 5.37 (t, *J* = 11.4 Hz, 1H, CH), 4.62 (s, 1H, OH), 4.47 (d, *J* = 12.0 Hz, 1H, CH), 4.01 (t, *J* = 12.0 Hz, 1H, CH), 3.93 (dd, *J* = 13.2, 7.2 Hz, 2H, CH_2), 3.29 (d, *J* = 12.6 Hz, 1H, CH), 1.72 (s, 3H, CH_3), 0.86 (t, *J* = 7.2 Hz, 3H, CH_3); ^{13}C NMR (151 MHz, $CDCl_3$, major diastereomer): δ = 171.5, 163.7, 162.0, 130.6, 130.4, 129.5, 129.4, 128.9, 116.4, 116.2, 112.6, 111.2, 89.2, 73.71, 62.3, 52.4, 51.2, 47.2, 45.5, 25.6, 13.5 ppm; MS (ESI, pos): m/z 474.1 $[M+Na]^+$; Anal. Calcd for $C_{24}H_{22}FN_3O_5$: C 63.85; H 4.91; N 9.31, found: C 63.74; H 4.99; N 9.18.



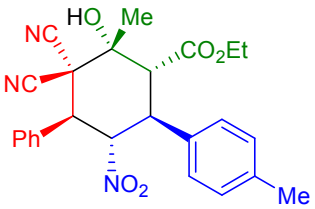
ent-**4i**

Compound *ent*-**4i** was synthesized by catalyst **II** and isolated as a white solid (169 mg, 75%); >30:1 dr; 98% ee; HPLC (major diastereomer): tR 5.70 min (minor), 6.36 min (major), *n*-heptane/ethanol, 97:3, 0.70 mL/min, Chiralpak-IC column.



4j

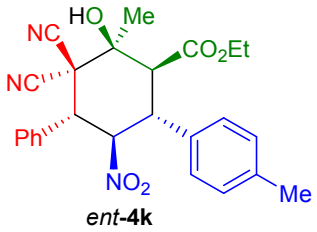
Compound **4j** was synthesized by catalyst **I** and isolated as a white solid (170 mg, 73%); Mp = 173-175 °C; $[\alpha]_{24}^{D}$ = +11.5 (c = 0.5, $CHCl_3$); >30:1 dr, 99% ee; HPLC (major diastereomer): tR 6.33 min (major), 9.54 min (minor), *n*-heptane/ethanol, 9:1, 1.00 mL/min, Chiralpak-AS column; IR (Capillary): 3812, 3452, 2983, 2648, 2456, 2228, 2180, 2059, 1987, 1915, 1710, 1561, 1492, 1457, 1371, 1295, 1206, 1091, 1015, 953, 893, 824, 731, 697 cm^{-1}; ^1H NMR(400 MHz, $CDCl_3$, major diastereomer): δ = 7.52 (br s, 2H, ArH), 7.42–7.41 (m, 3H, ArH), 7.33-7.32 (m, 2H, ArH), 7.24-7.21 (m, 2H, ArH), 5.35 (t, *J* = 11.8 Hz, 1H, CH), 4.57 (s, 1H, OH), 4.44 (d, *J* = 12.0 Hz, 1H, CH), 4.01-3.91 (m, 3H, CH, CH_2), 3.27 (d, *J* = 12.4 Hz, 1H, CH), 1.71 (s, 3H, CH_3), 0.86 (t, *J* = 6.9Hz, 3H, CH_3); ^{13}C NMR (101 MHz, $CDCl_3$, major diastereomer): δ = 171.5, 135.2, 132.2, 130.5, 130.4, 129.5, 129.4, 128.9, 112.6, 111.2, 89.0, 73.7, 62.4, 52.2, 51.2, 47.2, 45.7, 25.6, 13.5 ppm; MS (ESI, pos): m/z 490.1 $[M+Na]^+$; Anal. Calcd for $C_{24}H_{22}ClN_3O_5$: C 61.61; H 4.74; N 8.98, found: C 61.44; H 4.89; N 8.84.



4k

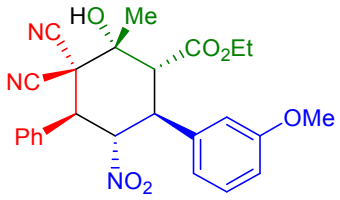
Compound **4k** was synthesized by catalyst **I** and isolated as a white solid (152 mg, 68%); Mp = 160-162 °C; $[\alpha]_{24}^{D}$ = +13.8 (c = 0.5, $CHCl_3$); >30:1 dr; 99% ee; HPLC (major

diastereomer): tR 8.48 min (major), 11.66 min (minor), *n*-heptane/ethanol, 9:1, 0.70 mL/min, Chiralpak-AS column; IR (Capillary): 3465, 2981, 2929, 2646, 2286, 2066, 1988, 1902, 1701, 1558, 1516, 1458, 1372, 1220, 1014, 1058, 1019, 899, 849, 810, 734, 699 cm^{-1}; ^1H NMR(400 MHz, $CDCl_3$, major diastereomer): δ = 7.64-7.47 (m, 2H, ArH), 7.51-7.37 (m, 3H), 7.13 (br s, 4H, ArH), 5.38 (t, *J* = 11.7 Hz, 1H, CH), 4.62 (s, 1H, OH), 4.44 (d, *J* = 12.0 Hz, 1H, CH), 4.03-3.81 (m, 3H, CH, CH_2), 3.28 (d, *J* = 12.4 Hz, 1H, CH), 2.30 (s, 3H, CH_3), 1.70 (s, 3H, CH_3), 0.80 (t, *J* = 7.2 Hz, 3H, CH_3); ^{13}C NMR (101 MHz, $CDCl_3$, major diastereomer): δ = 171.9, 139.1, 130.8, 130.5, 130.3, 129.8, 129.3, 112.7, 111.3, 89.2, 73.7, 62.2, 52.4, 51.2, 47.3, 46.0, 25.7, 21.1, 13.4 ppm; MS (ESI, pos): m/z 470.2 $[M+Na]^+$; HRMS Calcd for $[C_{25}H_{25}N_3O_5+Na]^+$: 470.1686, found: 470.1690.



***ent*-4k**

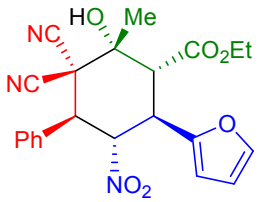
Compound *ent*-**4k** was synthesized by catalyst **II** and isolated as a white solid (161 mg, 72%); >30:1 dr; 99% ee; HPLC (major diastereomer): tR 8.56 min (minor), 11.64 min (major), *n*-heptane/ethanol, 9:1, 0.70 mL/min, Chiralpak-AS column.



4l

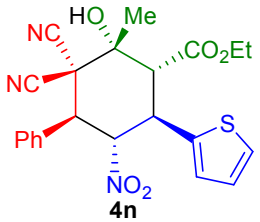
Compound **4l** was synthesized by catalyst **I** and isolated as a white solid (162 mg, 70%); Mp = 189-190 °C; $[\alpha]_{24}^{D}$ = +11.9 (c = 0.5, $CHCl_3$); >30:1 dr; 98% ee; HPLC (major diastereomer): tR 7.50 min (minor), 10.76 min (major), *n*-heptane/*i*-PrOH, 9:1, 0.70 mL/min, Chiralpak-IC column; IR (Capillary): 3744, 3449, 2972, 2843, 2642, 2274, 2179, 2044, 1988, 1889, 1718, 1599, 1561, 1457, 1373, 1262, 1197, 1106, 1037, 950, 857, 803, 739, 700 cm^{-1}; ^1H NMR(400 MHz, $CDCl_3$, major diastereomer): δ = 7.54 (d, *J* = 3.2 Hz, 2H, ArH), 7.42-7.40 (m, 3H, ArH), 7.28-7.24 (m, 1H, ArH), 6.83 (dd, *J* = 8.0, 2.4 Hz, 2H, ArH), 6.75 (br s, 1H, ArH), 5.40 (t, *J* = 11.8 Hz, 1H, CH), 4.63 (s, 1H, OH), 4.44 (d, *J* = 12.0 Hz, 1H, CH), 3.97-3.89 (m, 3H, CH, CH_2), 3.77 (s, 3H, CH_3), 3.29 (d, *J* = 12.4 Hz, 1H, CH), 1.70 (s, 3H,

CH_3), 0.82 (t, J = 7.2 Hz, 3H, CH_3); ^{13}C NMR (101 MHz, $CDCl_3$, major diastereomer): δ = 171.7, 160.0, 135.1, 130.7, 130.3, 130.2, 129.3, 128.9, 114.2, 112.7, 111.3, 89.0, 73.7, 62.2, 55.3, 52.3, 51.1, 47.3, 46.3, 25.6, 13.4 ppm; MS (ESI, pos): m/z 486.2 $[M+Na]^+$; Anal. Calcd for $C_{25}H_{25}N_3O_6$: C 64.79; H 5.44; N 9.07, found: C 64.56; H 5.69; N 8.92.



4m

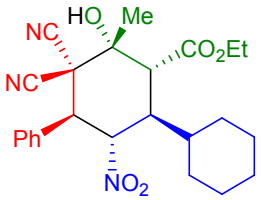
Compound **4m** was synthesized by catalyst **I** and isolated as a white solid (159 mg, 75%); Mp = 189-190 °C; $[\alpha]_{24}^{D}$ = +26.2 (c = 0.5, $CHCl_3$); >30:1 dr; 99% ee; HPLC (major diastereomer): tR 9.05 min (major), 13.22 min (minor), *n*-heptane/*i*-PrOH, 7:3, 0.50 mL/min, Chiralpak-AS column; IR (Capillary): 3881, 3745, 3433, 2983, 2647, 2424, 2272, 2165, 2079, 1989, 1946, 1888, 1708, 1565, 1458, 1365, 1214, 1112, 1016, 927, 881, 810, 739, 698 cm^{-1}; ^1H NMR(400 MHz, $CDCl_3$, major diastereomer): δ = 7.53-7.52 (m, 2H, ArH), 7.45 (d, J = 1.62 Hz, 1H, ArH), 7.42-7.41 (m, 3H, ArH), 6.62 (dd, J = 3.2, 2.0 Hz, 1H, ArH), 6.20 (d, J = 2.8 Hz, 1H, ArH), 5.50 (t, J = 11.6 Hz, 1H, CH), 4.62 (s, 1H, OH), 4.41 (d, J = 12.4 Hz, 1H, CH), 4.12 (t, J = 12.0 Hz, 1H, CH), 4.05-4.02 (m, 2H, CH_2), 3.43 (d, J = 12.0 Hz, 1H, CH), 1.71 (s, 3H, CH_3), 1.03 (t, J = 7.2 Hz, 3H, CH_3); ^{13}C NMR (101 MHz, $CDCl_3$, major diastereomer): δ = 171.8, 146.8, 143.9, 130.6, 130.3, 129.4, 129.3, 128.9, 112.3, 111.3, 110.4, 110.2, 87.0, 73.5, 62.4, 51.1, 50.5, 46.9, 40.1, 25.5, 13.6 ppm; MS (CI, Methane): m/z 423.6 $[M]^+$; Anal. Calcd for $C_{22}H_{21}N_3O_6$: C 62.41; H 5.00; N 9.92, found: C 62.49; H 4.93; N 9.85.



4n

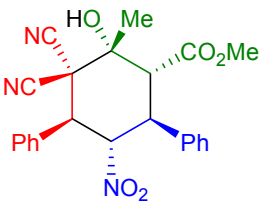
Compound **4n** was synthesized by catalyst **I** and isolated as a white solid (149 mg, 68%); Mp = 208-210 °C; $[\alpha]_{24}^{D}$ = +15.0 (c = 0.5, $CHCl_3$); >30:1 dr, 99% ee (major diastereomer); HPLC (major diastereomer): tR 12.80 min (major), 16.13 min (minor), *n*-heptane/ethanol, 9:1, 0.50 mL/min, Chiralcel-OD column; IR (Capillary): 3832, 3434, 2983, 2412, 2181, 2071, 1975, 1889, 1705, 1565, 1456, 1341, 1201, 1111, 1015, 855, 697 cm^{-1}; ^1H NMR(400 MHz, $CDCl_3$, major diastereomer): δ = 7.52 (br s, 2H, ArH), 7.43-7.41 (m, 3H, ArH), 7.29 (d,

J = 4.8 Hz, 1H, ArH), 6.96-6.91 (m, 2H, ArH), 5.33 (t, J = 11.6 Hz, 1H, CH), 4.58 (s, 1H, OH), 4.43 (d, J = 12.0 Hz, 1H, CH), 4.35 (t, J = 11.7 Hz, 1H, CH), 4.06-3.96 (m, 2H, CH_2), 3.28 (d, J = 12.4 Hz, 1H, CH), 1.71 (s, 3H, CH_3), 0.94 (t, J = 7.2 Hz, 3H, CH_2); ^{13}C NMR (101 MHz, $CDCl_3$, major diastereomer): δ = 171.5, 136.1, 130.5, 130.4, 129.4, 127.9, 127.1, 126.5, 112.5, 111.3, 90.2, 73.7, 62.5, 53.7, 51.1, 47.4, 41.7, 25.6, 13.5 ppm; MS (EI): m/z 440.4 $[M+1]^+$; HRMS (ESI): calcd for $[C_{22}H_{21}N_3O_5S+Na]^+$: 462.1094, found: 462.1098.



4o

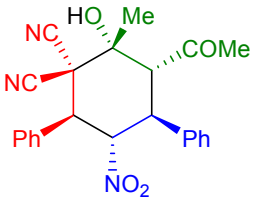
Compound **4o** was synthesized by catalyst **I** and isolated as a white solid (152 mg, 69%); Mp = 68 °C; $[\alpha]_{24}^D$ = +49.4 (c = 0.5, $CHCl_3$); >30:1 dr; 99% ee (major diastereomer); HPLC (major diastereomer): tR 6.17 min (major) 8.11 min (minor), *n*-heptane/*i*-PrOH, 8:2, 0.7 mL/min, Chiralpak AS column; IR (Capillary): 3949, 3784, 3466, 2930, 2857, 2658, 2529, 2321, 2074, 1999, 1929, 1714, 1560, 1453, 1370, 1184, 1107, 1018, 962, 862, 794, 739, 698 cm^{-1}; ^1H NMR(600 MHz, $CDCl_3$, major diastereomer): δ = 7.48 (br s, 2H, ArH), 7.43-7.41 (m, 3H, ArH), 5.15 (t, J = 11.7 Hz, 1H, CH), 4.46 (s, 1H, OH), 4.39-4.31 (m, 2H, CH_2), 4.30 (d, 1H, J = 12.0 Hz, CH), 2.99 (d, J = 12.6 Hz, 1H, CH), 2.87 (t, J = 12.0 Hz, 1H, CH), 1.82-1.76 (m, 2H, CH_2), 1.78-1.64 (m, 3H, CH_2, CH), 1.63 (s, 3H, CH_3) 1.39 (t, J = 7.2 Hz, 3H), 1.25-1.06 (m, 6H, CH_2); ^{13}C NMR (151 MHz, $CDCl_3$, major diastereomer): δ = 173.2, 131.0, 130.2, 129.2, 112.5, 111.4, 85.9, 73.3, 62.6, 50.8, 48.6, 47.0, 43.5, 40.1, 28.6, 28.5, 27.0, 26.9, 26.1, 25.6, 14.1 ppm; MS (EI): m/z 439.2 $[M]^+$; HRMS (ESI): calcd for $[C_{24}H_{29}N_3O_5+Na]^+$: 462.1999, found: 462.1998.



4p

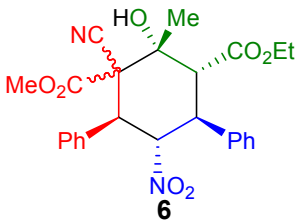
Compound **4p** was synthesized by catalyst **I** and isolated as a white solid (144 mg, 69%); Mp = 213-215 °C; $[\alpha]_{24}^D$ = +24.1 (c = 0.5, $CHCl_3$); >30:1 dr; 99% ee (major diastereomer);

HPLC (major diastereomer): tR 14.21 min (minor), 16.50 min (major), *n*-heptane/*i*-PrOH, 95:5, 1.00 mL/min, Chiralpak-AD column; IR (Capillary): 3431, 2958, 2587, 2202, 2079, 1972, 1895, 1714, 1564, 1498, 1449, 1352, 1217, 1113, 995, 905, 845, 738, 696 cm^{-1}; ^1H NMR(600 MHz, $CDCl_3$, major diastereomer): δ = 7.56 (d, *J* = 2.4 Hz, 2H, ArH), 7.44-7.33 (m, 3H, ArH), 7.36-7.31 (m, 3H, ArH), 7.26 (br s, 2H, ArH), 5.44 (t, *J* = 11.4 Hz, 1H, CH), 4.56 (s, 1H, OH), 4.48 (d, *J* = 12.0 Hz, 1H, CH), 4.01 (t, *J* = 12.0 Hz, 1H, CH), 3.40 (s, 3H, CH_3), 3.35 (d, *J* = 12.0 Hz, 1H, CH), 1.72 (s, 3H, CH_3); ^{13}C NMR (151 MHz, $CDCl_3$, major diastereomer): δ = 172.0, 133.6, 130.7, 130.3, 129.4, 129.3, 129.2, 112.6, 111.3, 89.0, 73.7, 52.7, 52.6, 51.2, 47.2, 46.2, 25.7 ppm; MS (ESI, pos): m/z 420.2 $[M+H]^+$; Anal. Calcd for $C_{23}H_{21}N_3O_5$: C 65.86; H 5.05; N 10.02, found: C 65.62; H 5.20; N 9.82.



4q

Compound **4q** was synthesized by catalyst **I** and isolated as a white solid (144 mg, 71%); Mp = 190-192 °C; $[\alpha]_{24}^{D}$ = +73.2 (c = 0.5, $CHCl_3$); >30:1 dr; 99% ee (major diastereomer); HPLC (major diastereomer): tR 25.42 min (major), 29.55 min (minor), *n*-heptane/ethanol, 97:3, 1.00 mL/min, Chiralpak-AS column; IR (Capillary): 3885, 3418, 2982, 4648, 2324, 2215, 2163, 2085, 2024, 1971, 1887, 1707, 1559, 1496, 1456, 1366, 1216, 1171, 1116, 1064, 948, 905, 830, 755, 697 cm^{-1}; ^1H NMR(400 MHz, $CDCl_3$, major diastereomer): δ = 7.55 (d, *J* = 3.6 Hz, 2H, ArH), 7.43-7.41 (m, 4H, ArH), 7.35-7.24 (m, 4H, ArH), 5.49 (t, *J* = 11.8 Hz, 1H, CH), 4.92 (s, 1H, OH), 4.46 (d, *J* = 12.0 Hz, 1H, CH), 3.90 (t, *J* = 12.0 Hz, 1H, CH), 3.52 (d, *J* = 12.4 Hz, 1H, CH), 1.73 (s, 3H, CH_3), 1.66 (s, 3H, CH_3); ^{13}C NMR (101 MHz, $CDCl_3$, major diastereomer): δ = 211.5, 133.6, 130.7, 130.3, 129.7, 129.4, 129.3, 128.9, 127.9, 112.8, 111.2, 88.9, 74.2, 56.0, 51.4, 47.0, 46.7, 34.1, 25.4 ppm; MS (EI): m/z 403.4 $[M]^+$; HRMS (ESI): calcd. for $[C_{23}H_{21}N_3O_4+Na]^+$: 426.1424, found: 426.1420.



6

Compound **6** was synthesized by catalyst **I** and isolated as a white solid (156 mg, 67%); Mp = 100-102 °C; $[\alpha]_{24}^{D}$ = +18.0 (c = 0.4, $CHCl_3$); 1.6:1 dr; 99% ee (both diastereomers); HPLC: tR 22.49 min (minor), 25.15 min (major) (major diastereomer), HPLC: tR 12.02 min (minor), 15.63 min (major) (minor diastereomer), *n*-heptane/i-PrOH, 9:1, 0.70 mL/min, Chiralpak-IC column; IR (Capillary): 3835, 3461, 2982, 2653, 2324, 2179, 2101, 2012, 1973, 1893, 1728, 1603, 1556, 1494, 1453, 1373, 1241, 1185, 1115, 1020, 951, 906, 832, 762, 700, 659 cm^{-1}; ^1H NMR(600 MHz, $CDCl_3$, major diastereomer): δ = 7.45-7.25 (m, 10H, ArH), 5.94 (t, *J* = 11.7 Hz, 1H, CH), 4.71 (s, 1H, OH), 4.39 (d, *J* = 12.0 Hz, 1H, CH), 3.96-3.80 (m, 3H, CH, CH_2), 3.73 (s, 3H, CH_3), 3.73 (t, *J* = 12.6 Hz, 1H, CH), 1.43 (s, 3H, CH_3), 0.76 (t, *J* = 6.9 Hz, 3H, CH_3); ^1H NMR(600 MHz, $CDCl_3$, minor diastereomer): δ = 7.45-7.25 (m, 10H, ArH), 5.44 (t, *J* = 11.7 Hz, 1H, CH), 4.56 (d, *J* = 12.0 Hz, 1H, CH), 4.50 (s, 1H, OH), 4.15 (t, *J* = 12.0 Hz, 1H, CH), 3.96-3.80 (m, 2H, CH_2), 3.50 (s, 3H, CH_3), 3.46 (d, *J* = 12.6 Hz, 1H, CH) 1.52 (s, 3H, CH_3), 0.86 (t, *J* = 6.9 Hz, 3H, CH_3); ^{13}C NMR (151 MHz, $CDCl_3$, major and minor diastereomer): δ = 173.2, 170.2, 167.1, 165.8, 135.2, 135.1, 132.2, 132.0, 129.5, 129.4, 129.0, 128.9, 128.8, 128.7, 128.0, 116.5, 115.4, 91.1, 89.2, 73.7, 72.3, 62.0, 61.7, 61.5, 59.9, 54.0, 53.9, 53.8, 51.6, 47.9, 46.5, 46.1, 45.8, 25.4, 25.2, 13.6, 13.4 ppm; MS (ESI, pos): m/z 489.2 $[M+Na]^+$; Anal. Calcd for $C_{25}H_{26}N_2O_7$: C 64.37; H 5.62; N 6.01; found: C 64.62; H 5.44; N 5.81.

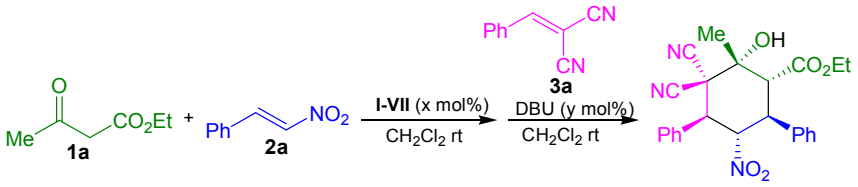
Procedure for the organocatalytic one-pot Michael/Knoevenagel condensation/Michael/1,2-addition reaction:

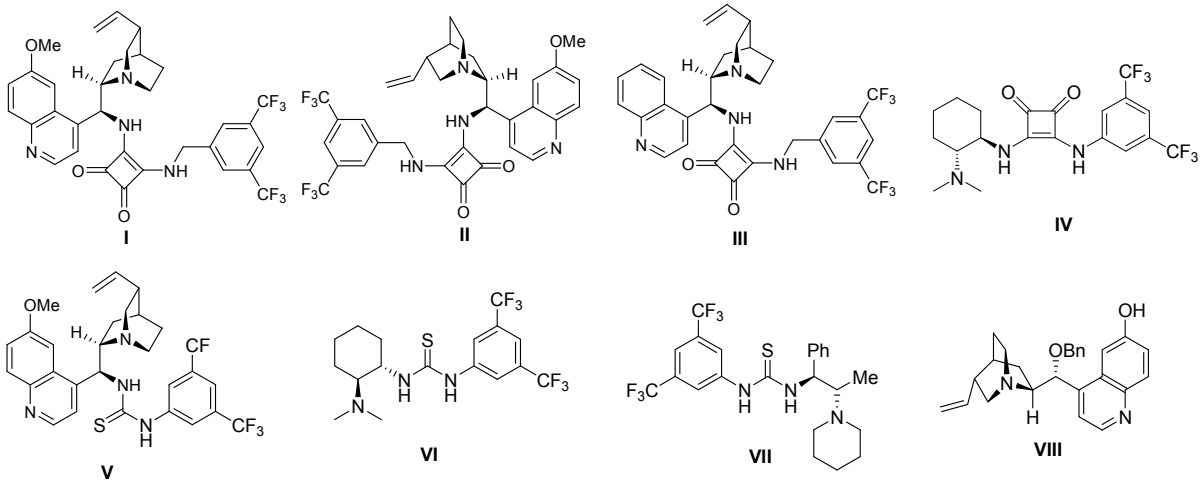
In a 50 mL round bottom flask equipped with a magnetic stirring bar, the (*E*)-β-nitrostyrene **2a** (1.0 equiv., 0.5 mmol) and catalyst **I** (1 mol%) were dissolved in CH_2Cl_2 (1.25 mL) and stirred 5 minutes at room temperature followed by the addition of the β-keto ester **1a** (1.0 equiv. 8 mmol, 1.041g). After stirring the reaction mixture at room temperature for 24 hours the benzaldehyde (1.2 equiv., 0.6 mmol), malononitrile (1.2 equiv., 0.6 mmol) and TBD (40 mol%; 0.1 M in CH_2Cl_2) were added subsequently and stirred for another 24 hours at room temperature. The crude product was directly purified by flash column chromatography (first *n*-hexane/EtOAc = 9:1, then *n*-hexane/EtOAc = 4:1) to afford the cyclohexane **4a** (69% yield).

Procedure for the gram-scale one-pot Michael/Michael/1,2-addition reaction:

In a 100 mL round bottom flask equipped with a magnetic stirring bar, the (*E*)-β-nitrostyrene **2a** (1.0 equiv., 8 mmol, 1.19 g) and catalyst **I** (1 mol%) were dissolved in CH_2Cl_2 (10.0 mL) and stirred 5 minutes at room temperature followed by the addition of the β-keto ester **1a** (1.0 equiv. 8 mmol, 1.04g). After stirring the reaction mixture at room temperature for 24 hours the α,α-dicyano-olefins **3a** (1.2 equiv., 9.6 mmol, 1.48 g) and TBD (20 mol%; 0.1 M in CH_2Cl_2) were added subsequently and stirred for another 24 hours at room temperature. The crude product was directly purified by flash column chromatography (first *n*-hexane/EtOAc = 9:1, then *n*-hexane/EtOAc = 4:1) to afford the cyclohexane **4a** (2.32 g, 67% yield).

Table: Screening of catalysts.[a]

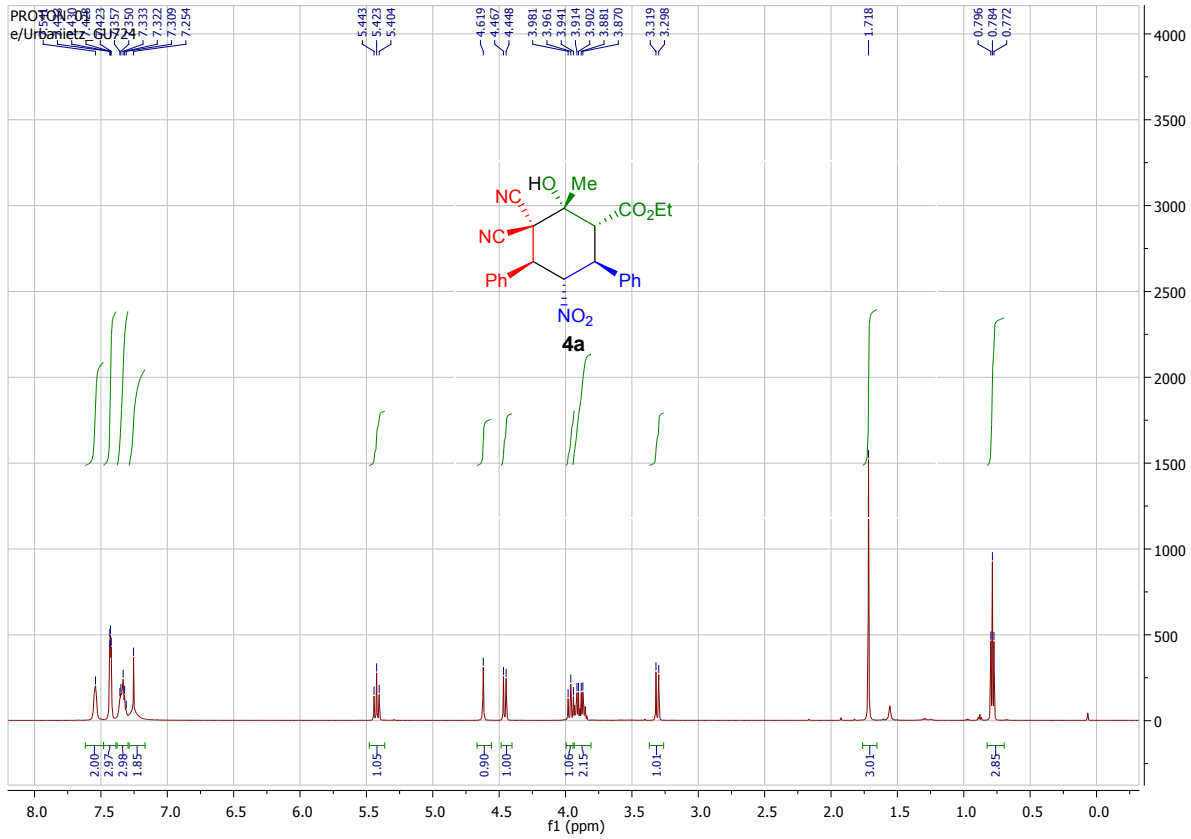


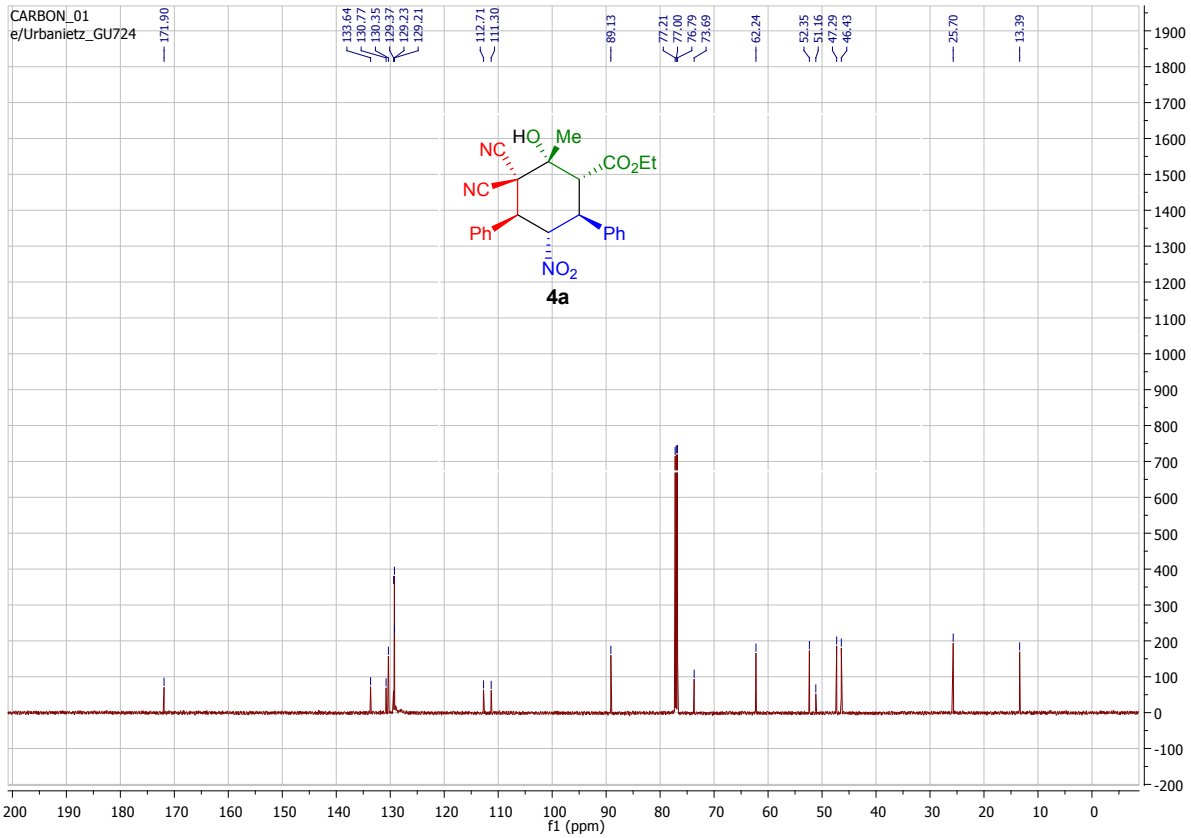


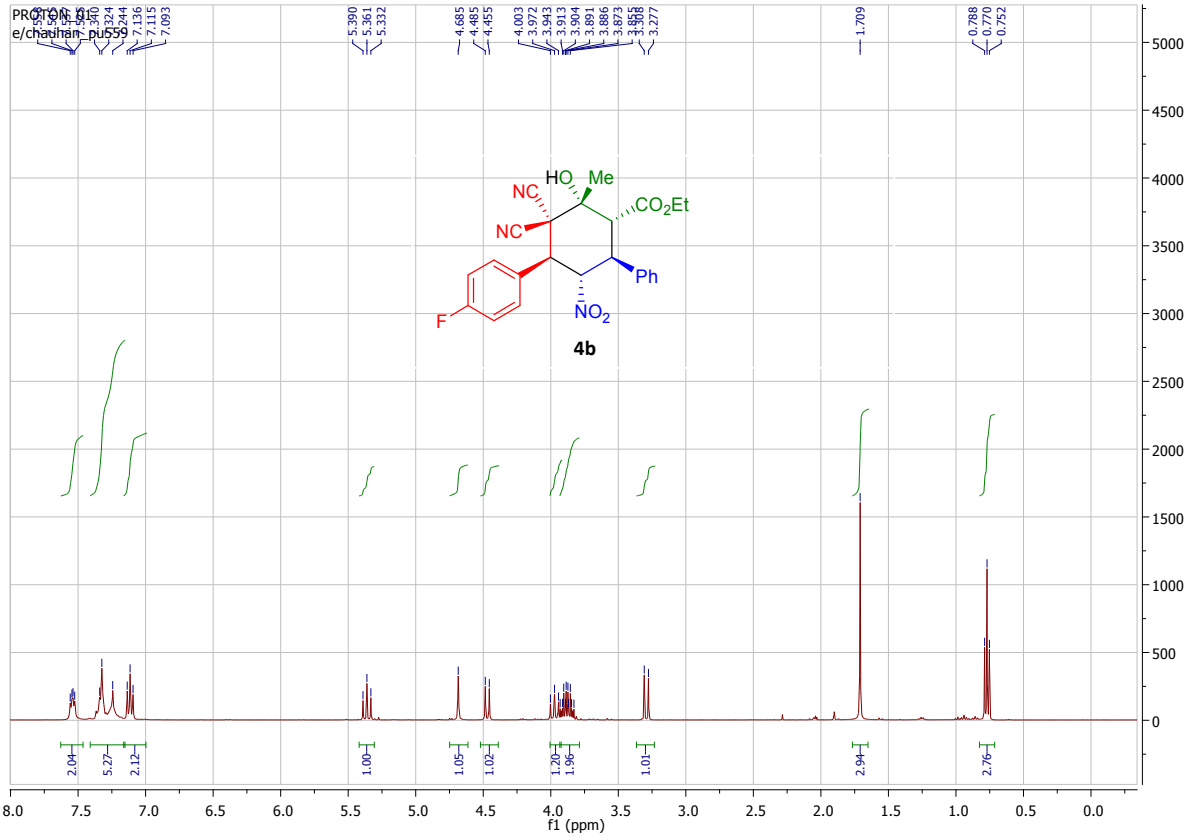
entry	catalyst (x mol%)	DBU (y mol%)	Time (h)	Yield (%)[b]	ee (%)[c]
1	**I** (2)	100	15+4	70	99
2	**II** (2)	100	15+4	71	-96 [d]
3	**III (2)**	100	15+4	69	95
4	**IV** (2)	100	24+4	67	-87 [d]
5	**V** (2)	100	60+4	59	89
6	**VI** (2)	100	36+4	61	-85 [d]
7	**VII** (2)	100	60+4	58	-25 [d]
8	**VIII** (2)	100	18+4	69	96
9	**I** (2)	10	15+24	54	99
10	**I** (1)	10	24+24	53	99

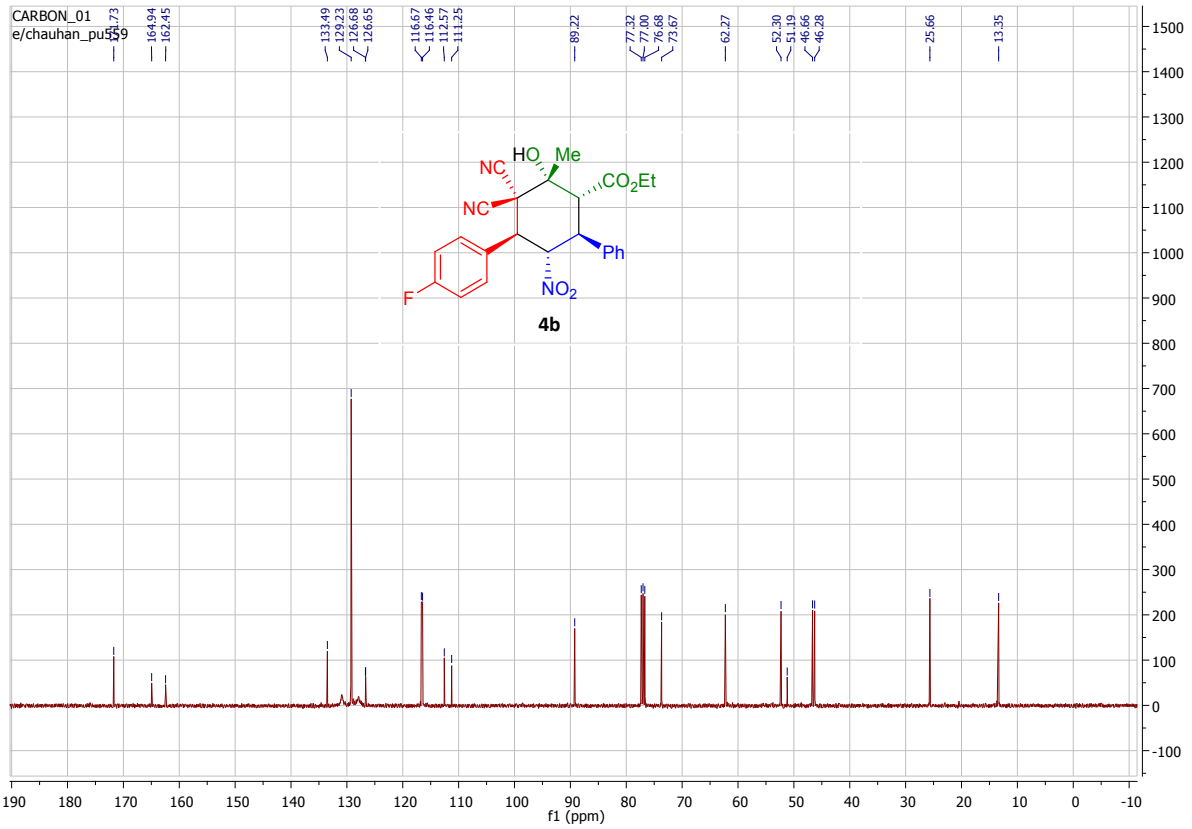
[a] Reaction conditions: 0.2 mmol of **1a**, 0.2 mmol of **2a**, X mmol of **I-VIII**, 0.24 mmol of **3a** and y mol% of base. [b] Yield of isolated **4a** after column chromatography [c] Enantiomeric excess of the major diastereomer (>30:1 dr) determined by HPLC analysis on achiral stationary phase. [d] The negative sign indicates the ee value of opposite enantiomer.

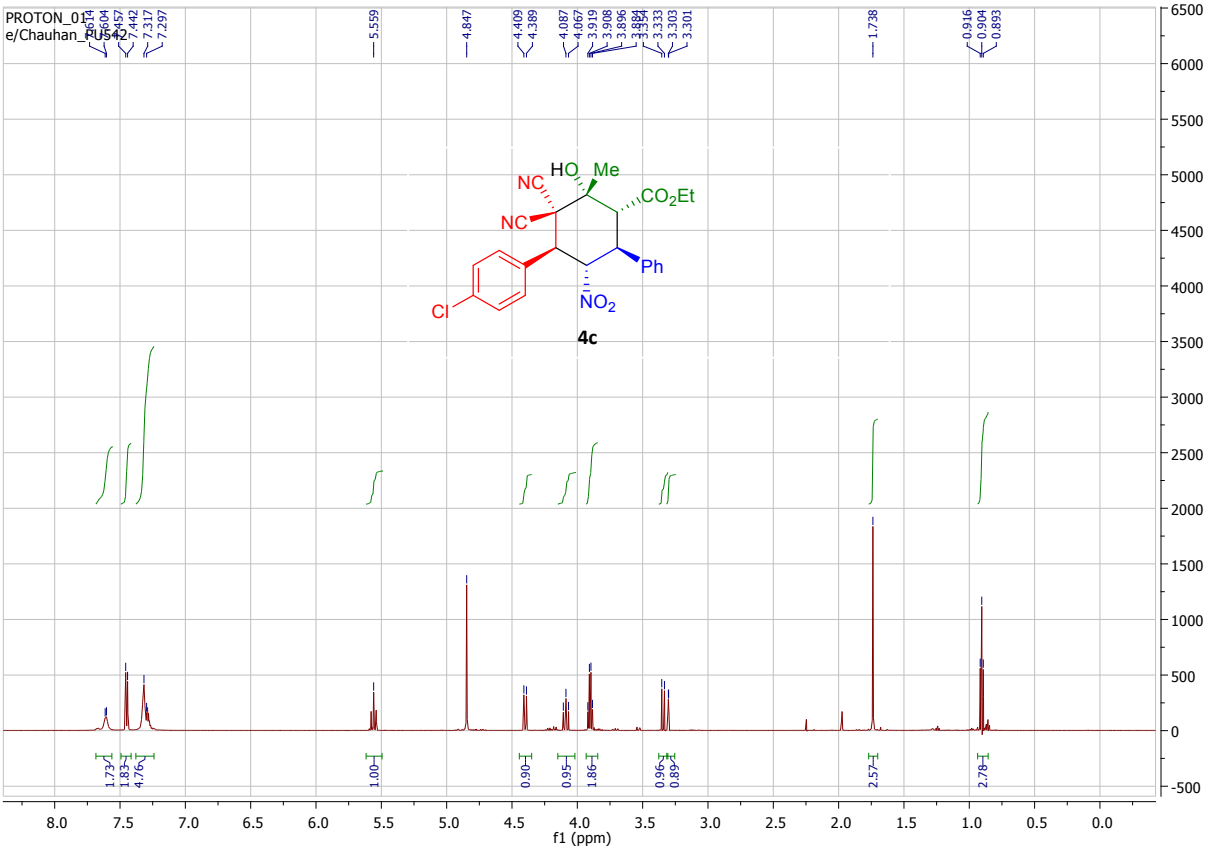
NMR Spectra:

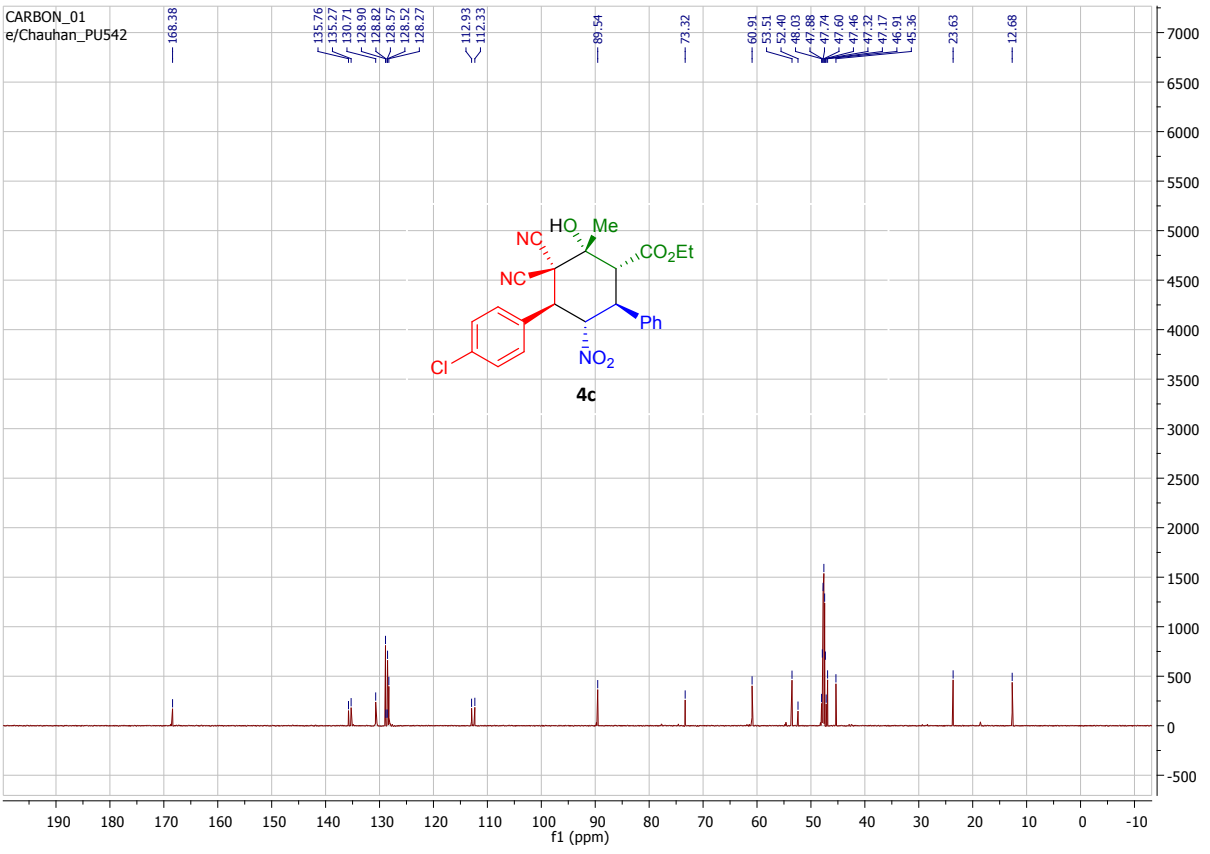

PROTON_01
e/Urbanietz_GU724
HO
Me
NC
CO2Et
NC
Ph
Ph
NO2
4a
f1 (ppm)

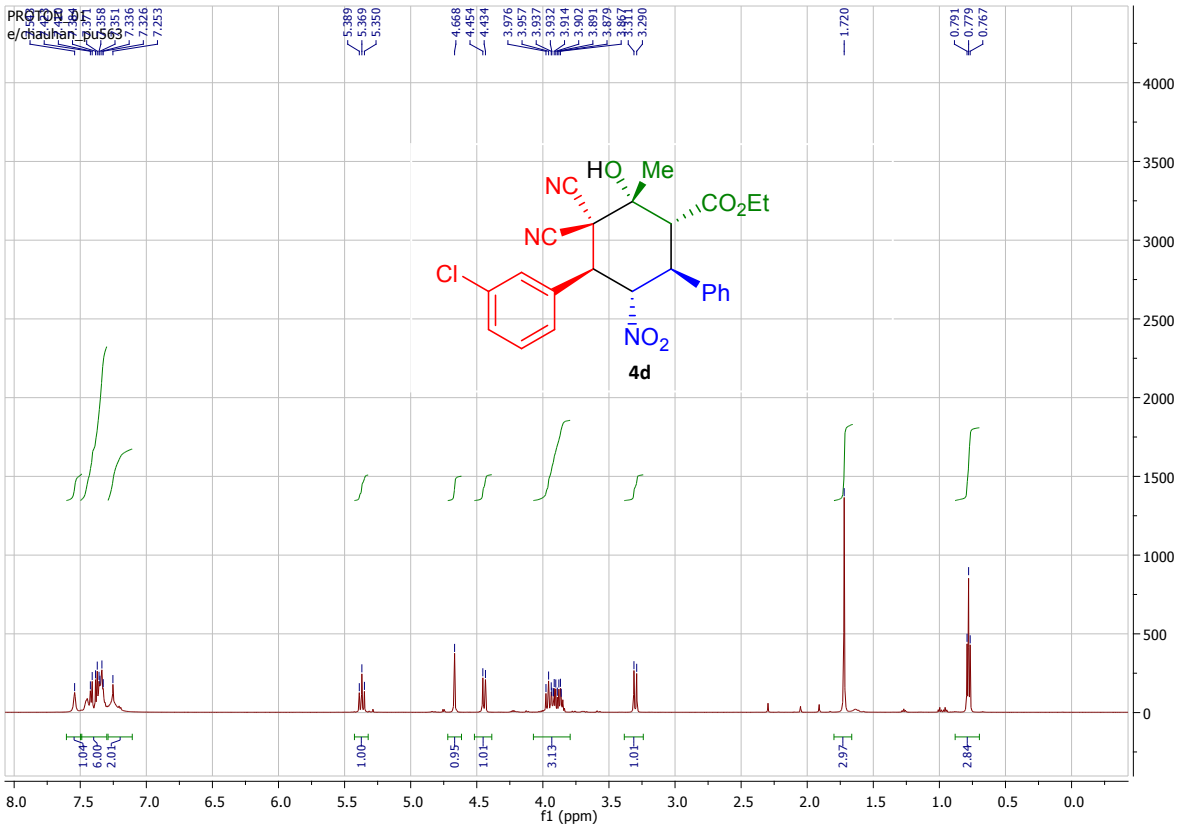

CARBON_01
e/Urbanietz_GU724
HO
Me
NC
CO2Et
NC
Ph
Ph
NO2
4a
f1 (ppm)

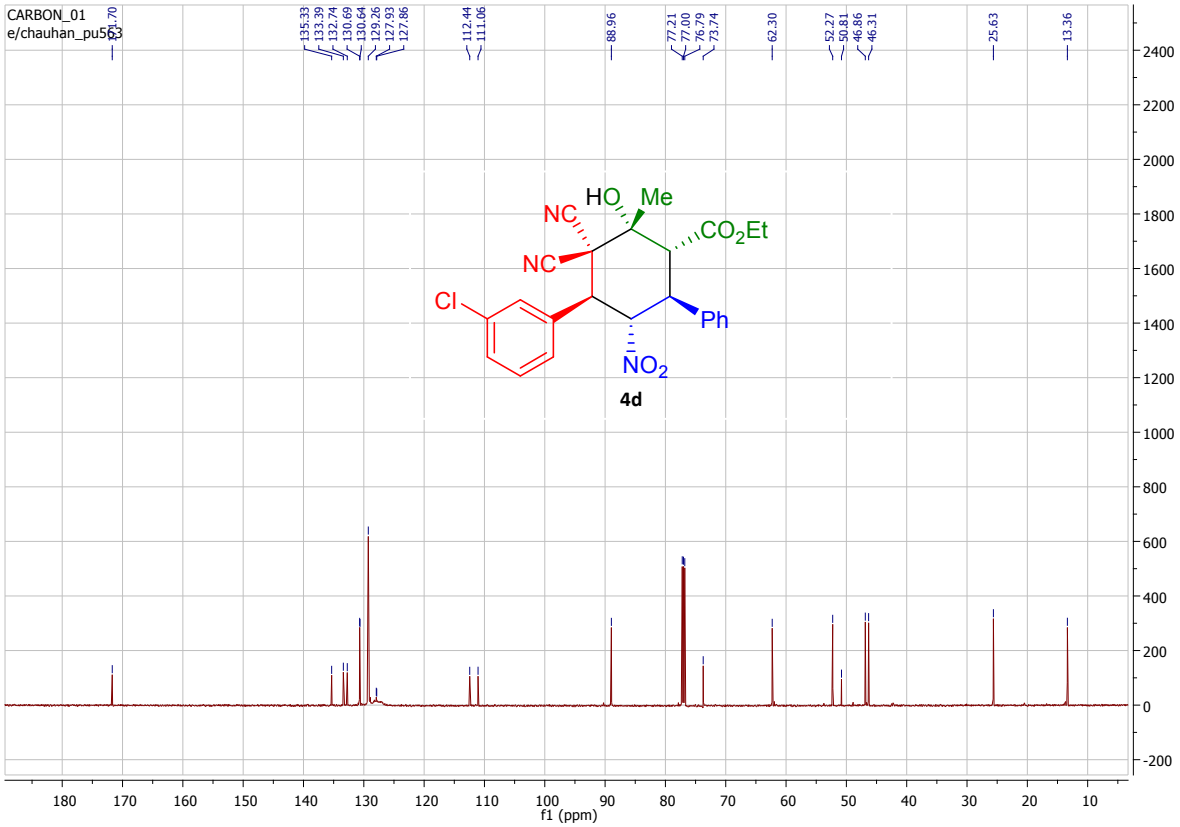

PROTON_01
e/chauhan_pu559
7.340
7.324
7.244
7.136
7.115
7.093
5.390
5.361
5.332
4.685
4.485
4.455
4.003
3.972
3.943
3.913
3.904
3.891
3.886
3.873
3.855
3.808
3.277
1.709
0.788
0.770
0.752
HO
Me
NC
NC
CO2Et
Ph
NO2
F
4b
2.04
5.27
2.12
1.00
1.05
1.02
1.20
1.96
1.01
2.94
2.76
f1 (ppm)

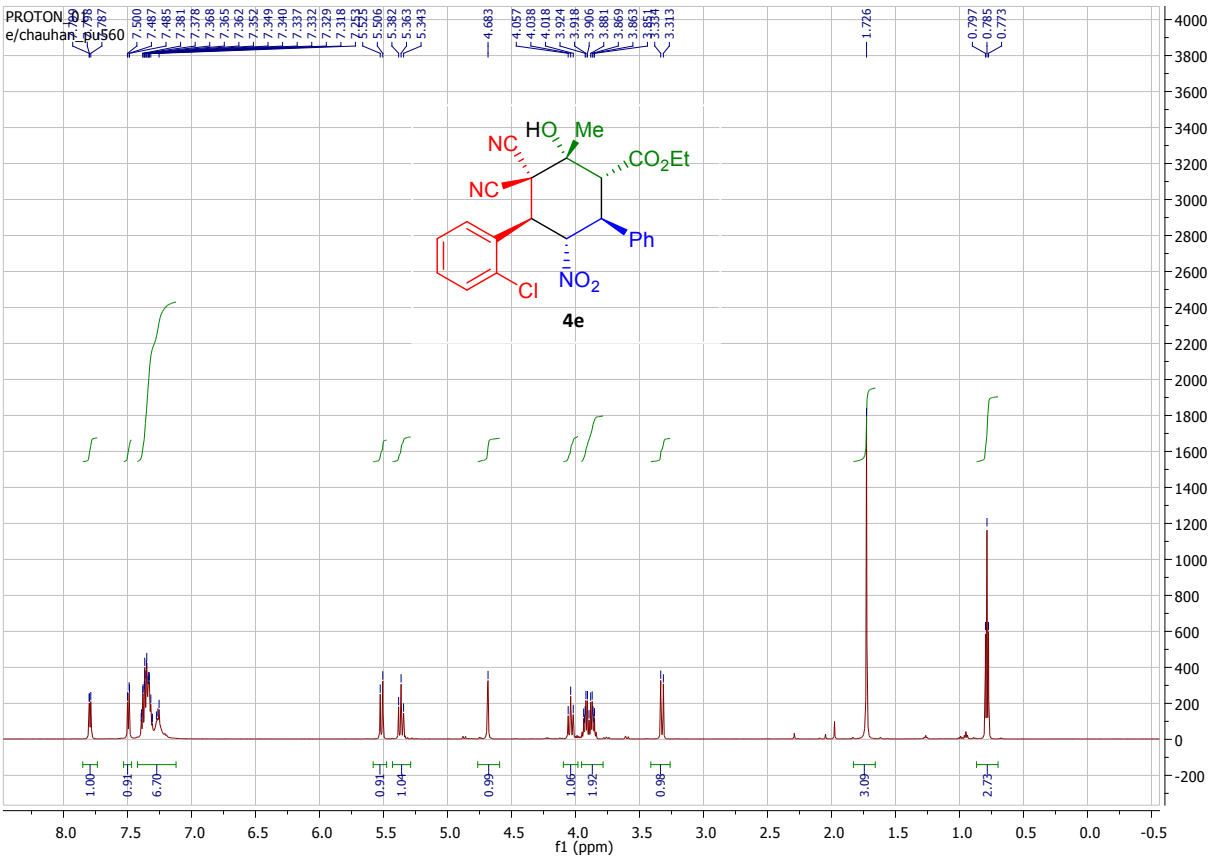

CARBON_01
e/chauhan_pu559
168.73
164.94
162.45
133.49
129.23
126.68
126.65
116.67
116.46
112.57
111.25
89.22
77.32
77.00
76.68
73.67
62.27
52.30
51.19
46.66
46.28
25.66
13.35
HO
Me
NC
NC
CO2Et
Ph
NO2
F
4b
f1 (ppm)

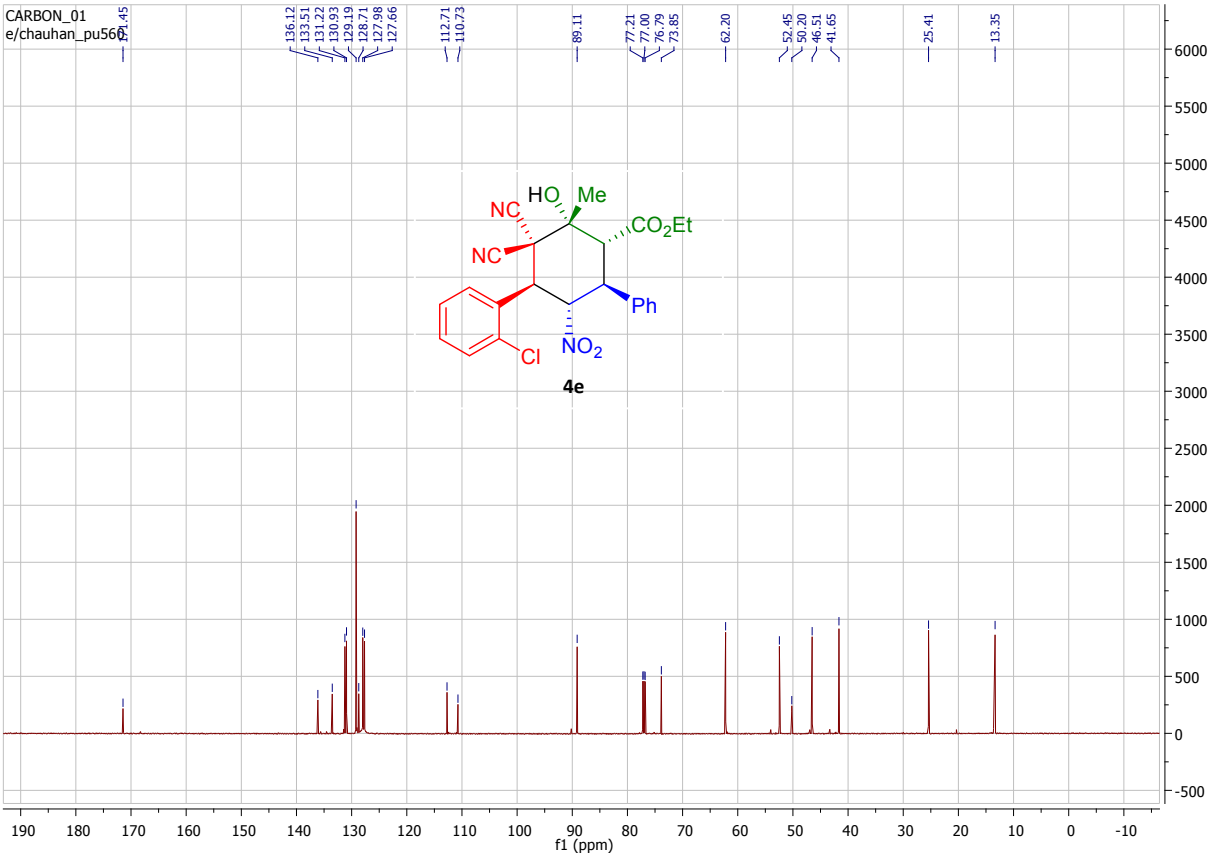

PROTON_01
e/Chauhan_PU542
5.559
4.847
4.409
4.389
4.087
4.067
3.919
3.908
3.896
3.884
3.354
3.333
3.303
3.301
1.738
0.916
0.904
0.893
HO
Me
NC
CO2Et
NC
Ph
NO2
Cl
4c
f1 (ppm)

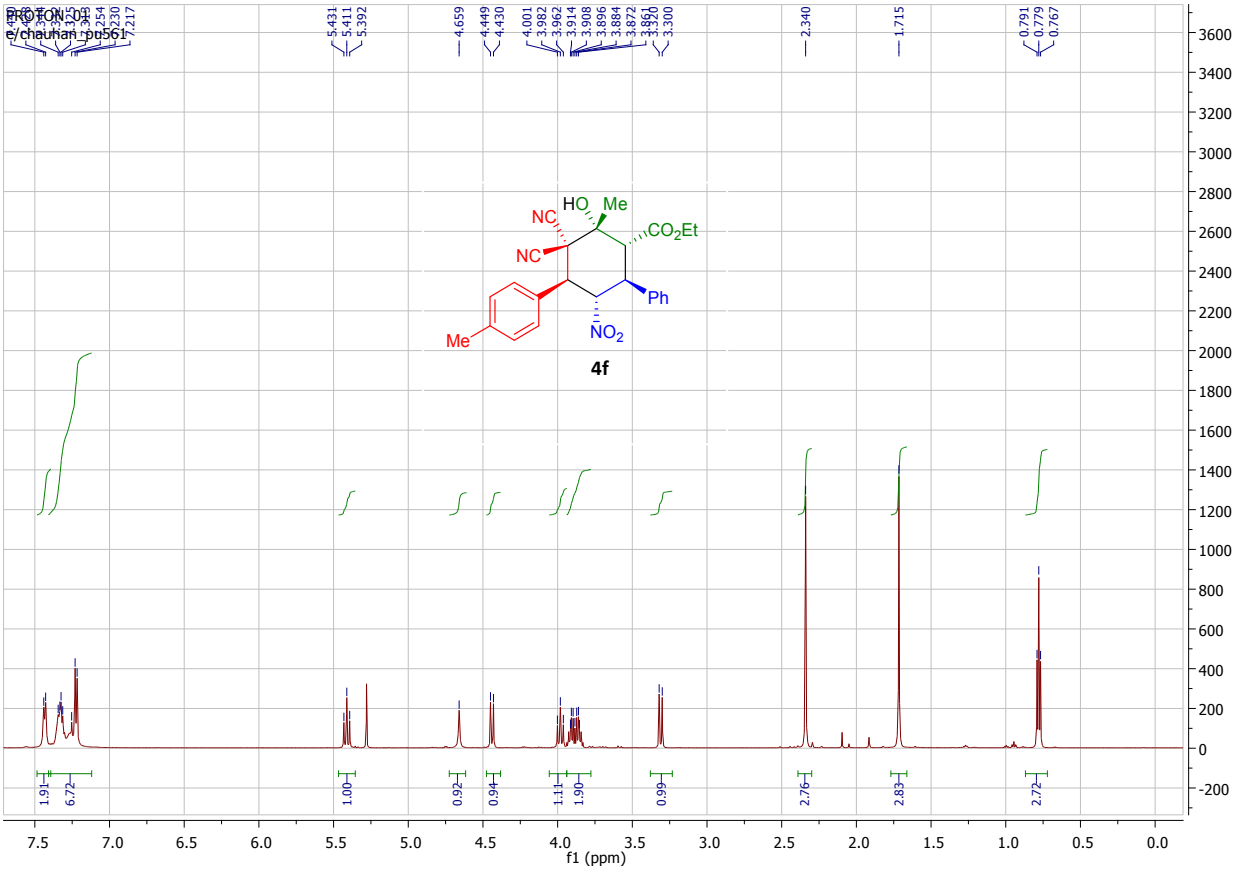

CARBON_01
e/Chauhan_PU542
168.38
135.76
135.27
130.71
128.90
128.82
128.57
128.52
128.27
112.93
112.33
89.54
73.32
60.91
53.51
52.40
48.03
47.88
47.74
47.60
47.46
47.32
47.17
46.91
45.36
23.63
12.68
HO
Me
NC
CO2Et
NC
Ph
NO2
Cl
4c
f1 (ppm)

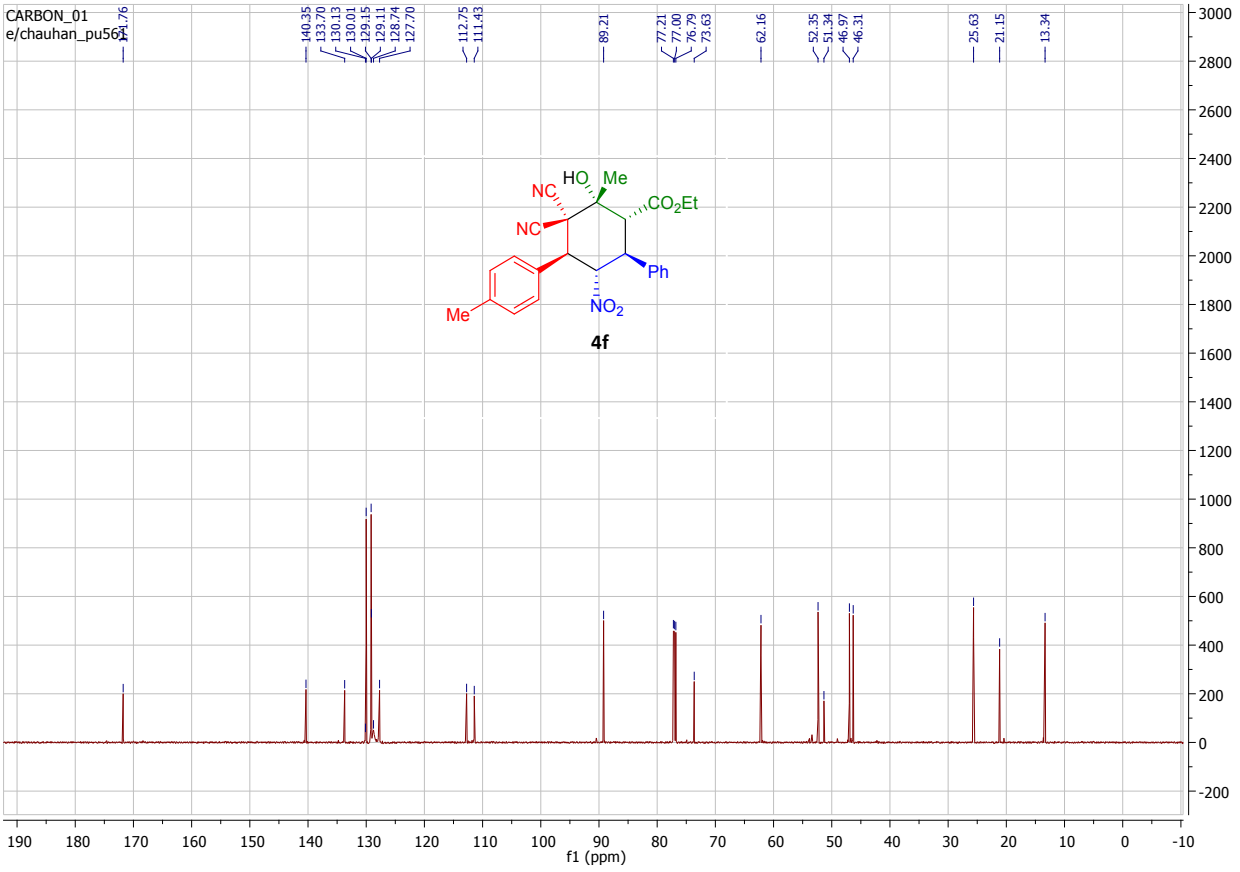

PROTON_01
e/chauhan_pu563
HO
Me
NC
CO2Et
NC
Cl
Ph
NO2
4d
f1 (ppm)

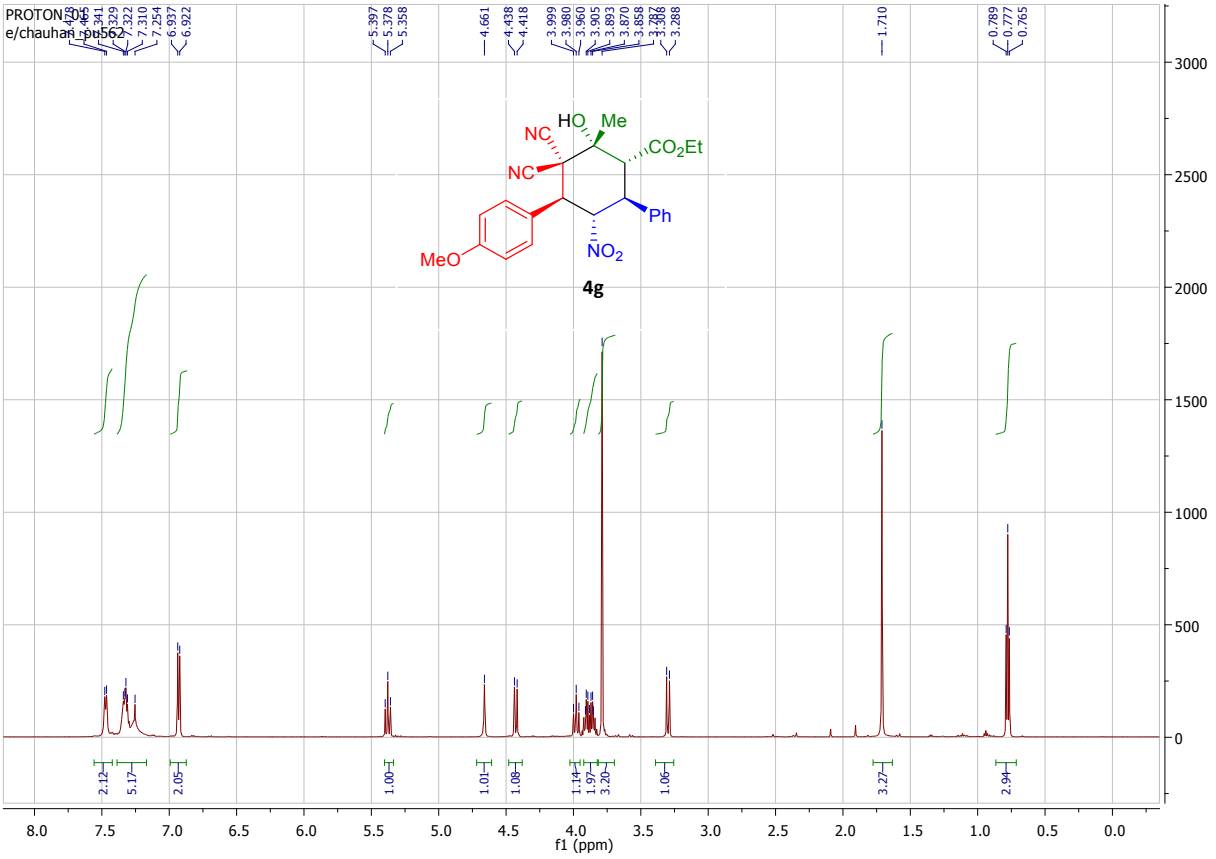

CARBON_01
e/chauhan_pu563
HO
Me
NC
CO2Et
NC
Cl
Ph
NO2
4d
f1 (ppm)

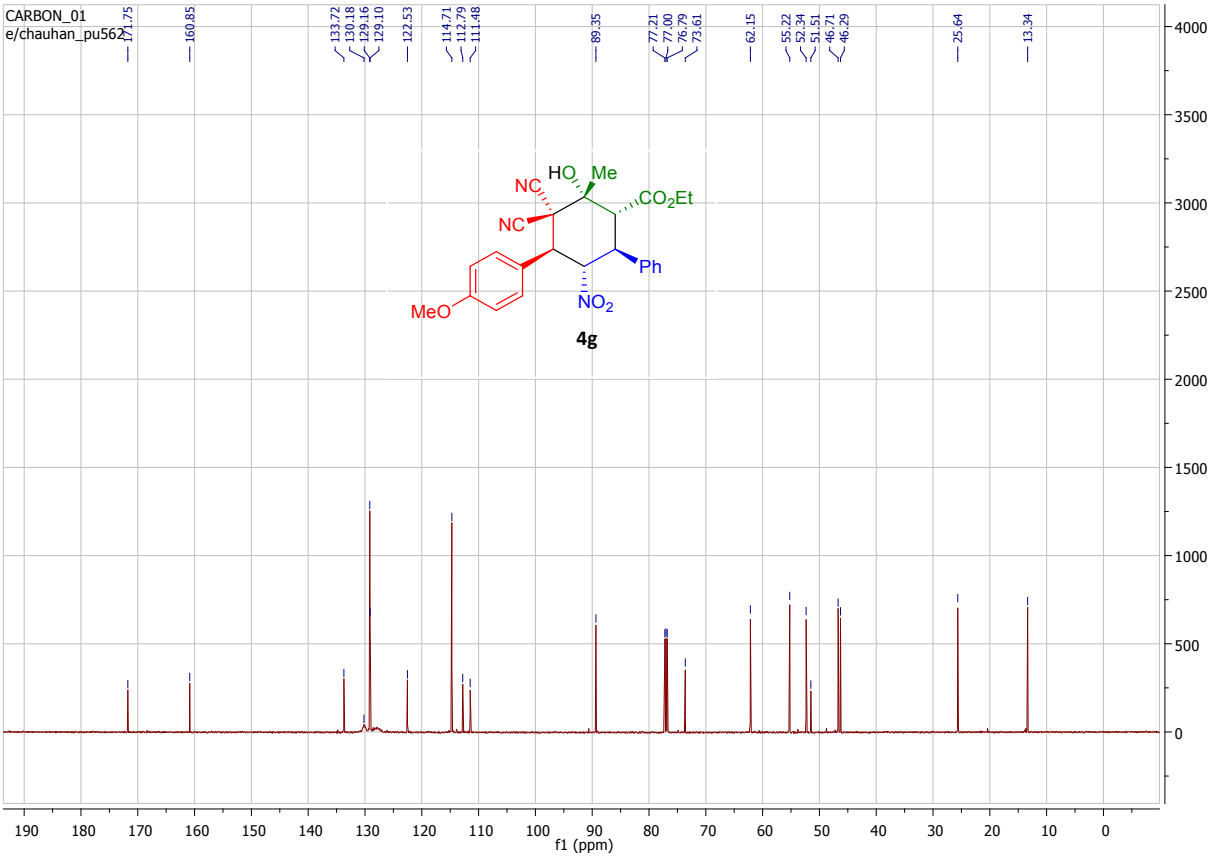

PROTON_01
e/chauhan_pu560
HO
Me
NC
CO2Et
NC
Ph
NO2
Cl
4e
f1 (ppm)

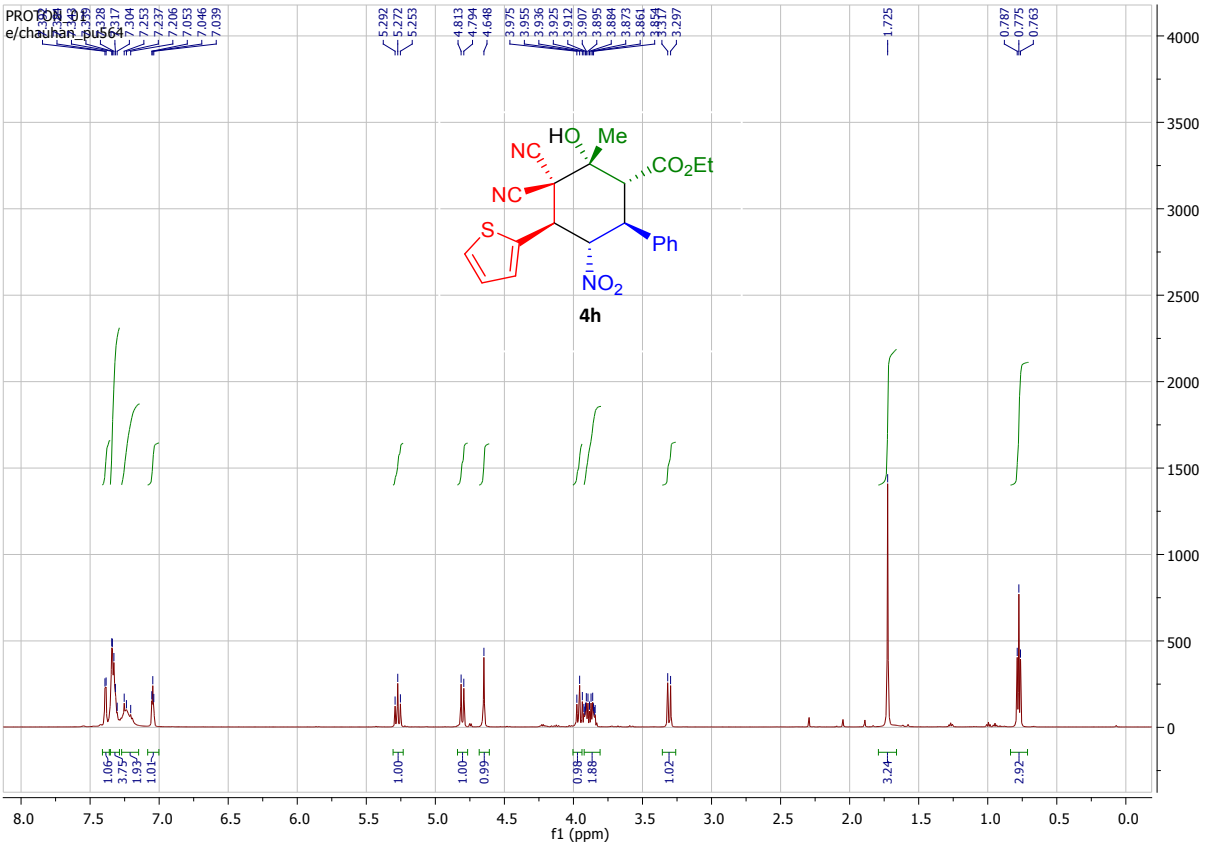

CARBON_01
e/chauhan_pu560
HO
Me
NC
CO2Et
NC
Ph
NO2
Cl
4e
f1 (ppm)

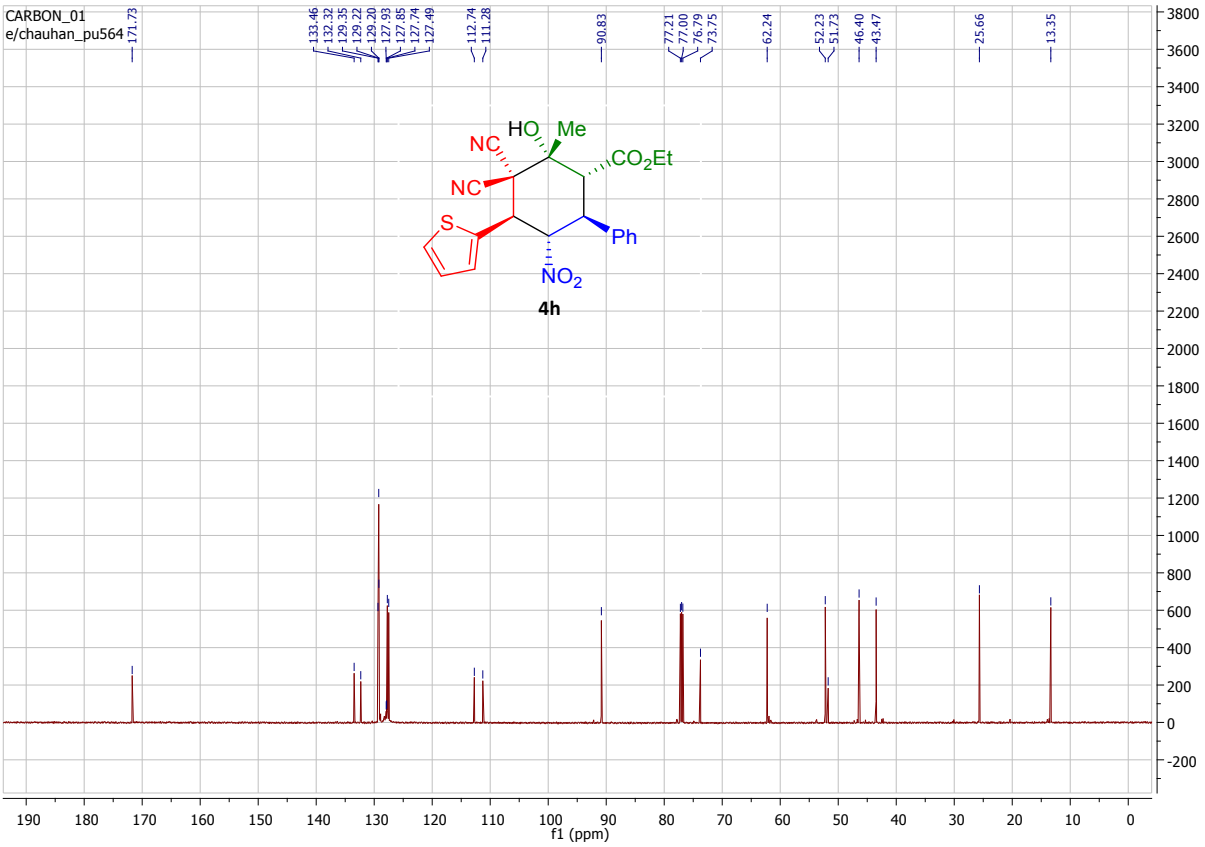

PROTON_01
e/chauhan_pu561
HO
Me
NC
CO2Et
NC
Ph
Me
NO2
4f
f1 (ppm)

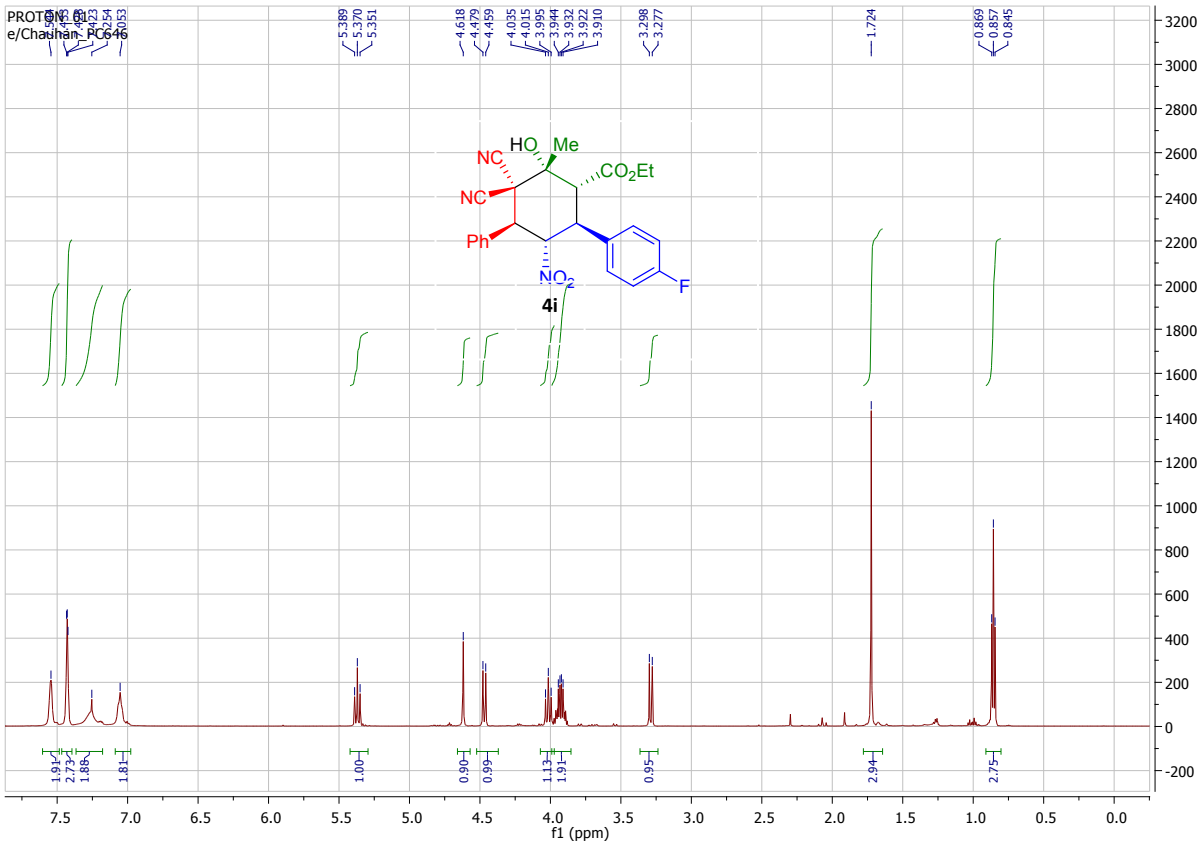

CARBON_01
e/chauhan_pu561
HO
Me
NC
CO2Et
NC
Ph
Me
NO2
4f
f1 (ppm)

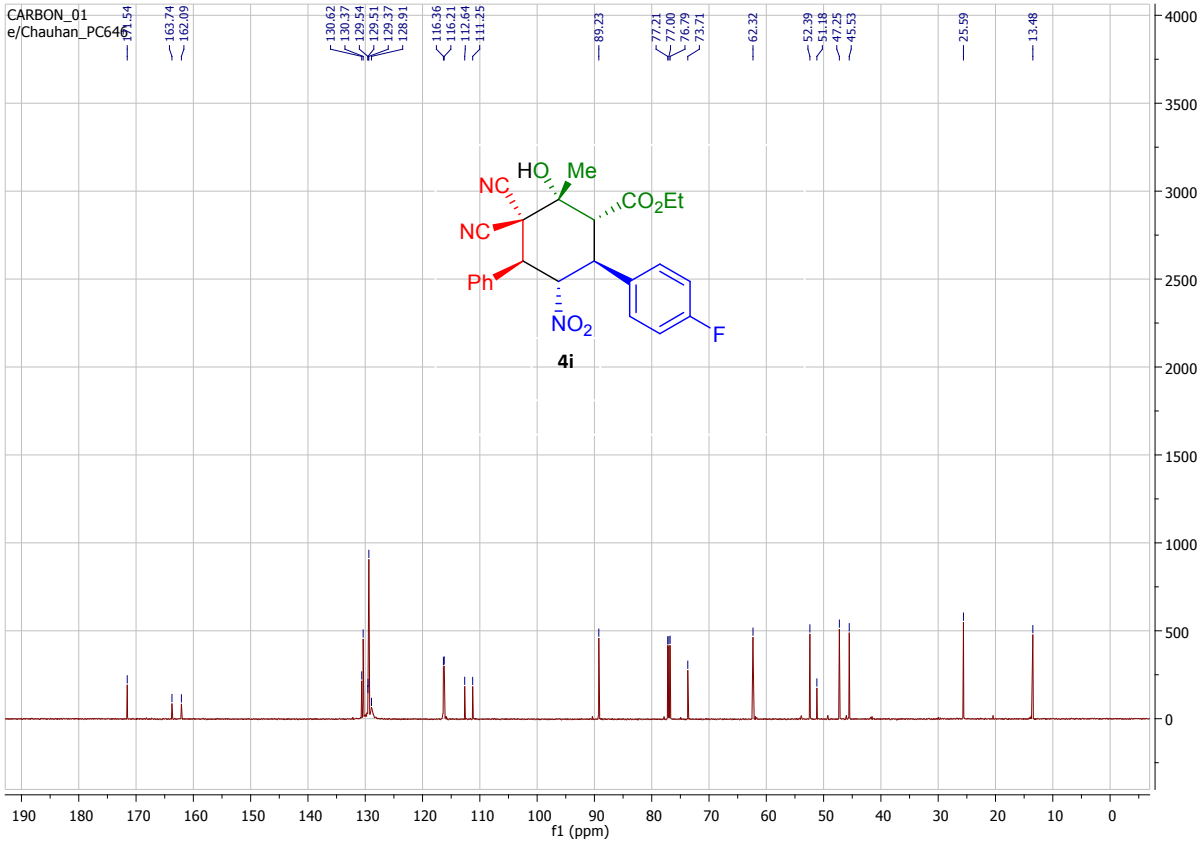

PROTON_05
e/chauhan_pu562
7.341
7.329
7.322
7.310
7.254
6.937
6.922
5.397
5.378
5.358
4.661
4.438
4.418
3.999
3.980
3.960
3.905
3.893
3.870
3.858
3.288
1.710
0.789
0.777
0.765
HO
Me
NC
NC
CO2Et
Ph
NO2
MeO
4g
2.12
5.17
2.05
1.00
1.01
1.08
1.14
1.97
3.20
1.06
3.27
2.94
f1 (ppm)

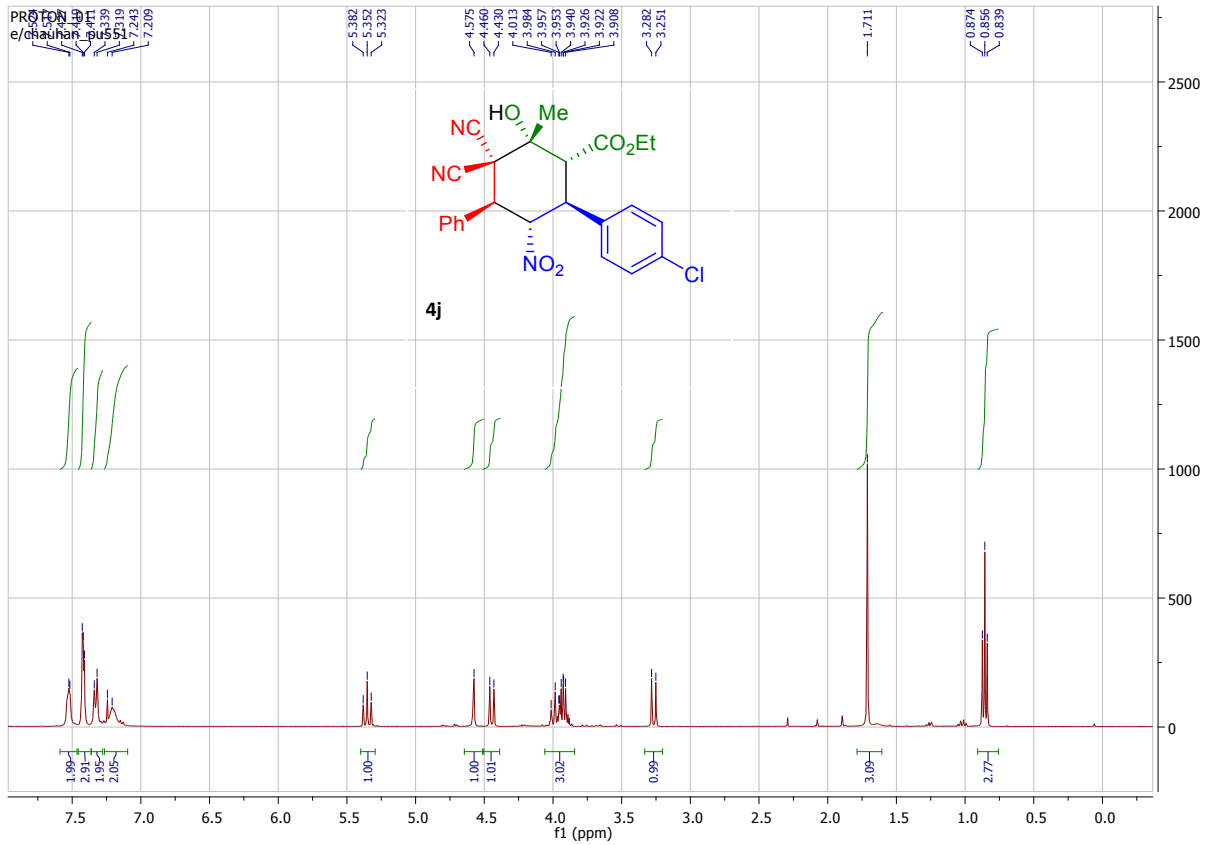

CARBON_01
e/chauhan_pu562
171.75
160.85
133.72
130.18
129.16
129.10
122.53
114.71
112.79
111.48
89.35
77.21
77.00
76.79
73.61
62.15
55.22
52.34
51.51
46.71
46.29
25.64
13.34
HO
Me
NC
NC
CO2Et
Ph
NO2
MeO
4g
f1 (ppm)

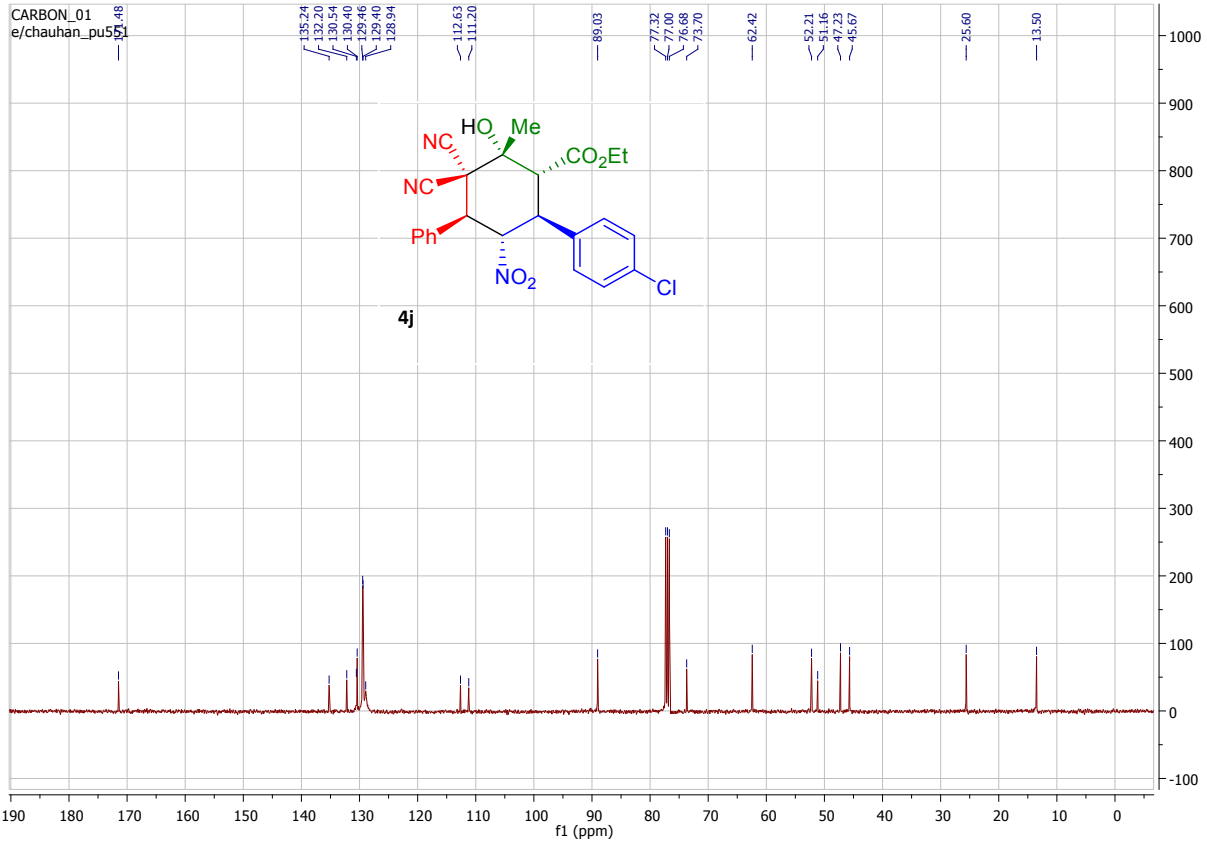

PROTON_01
e/chauhan_pu564
HO
Me
NC
CO2Et
NC
S
Ph
NO2
4h
f1 (ppm)

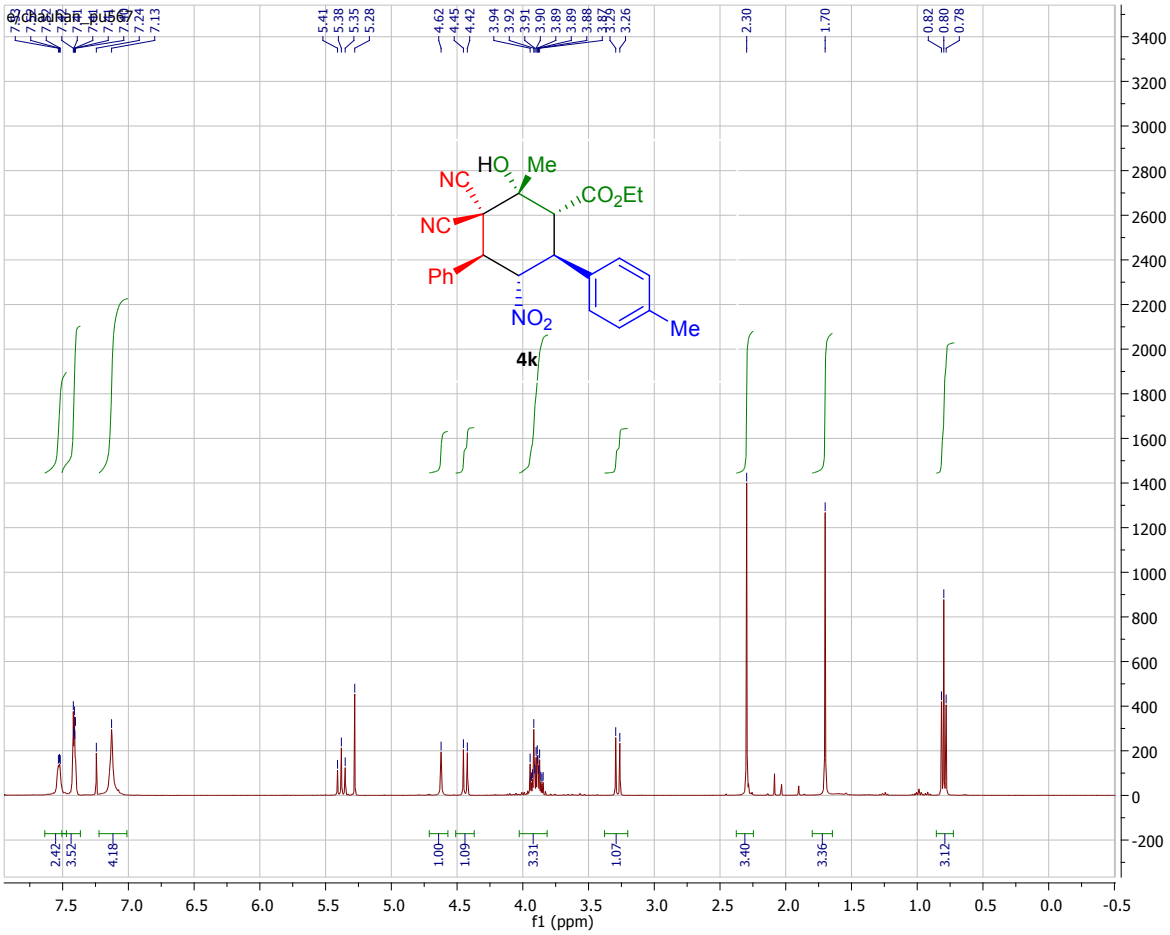

CARBON_01
e/chauhan_pu564
HO
Me
NC
CO2Et
NC
S
Ph
NO2
4h
f1 (ppm)

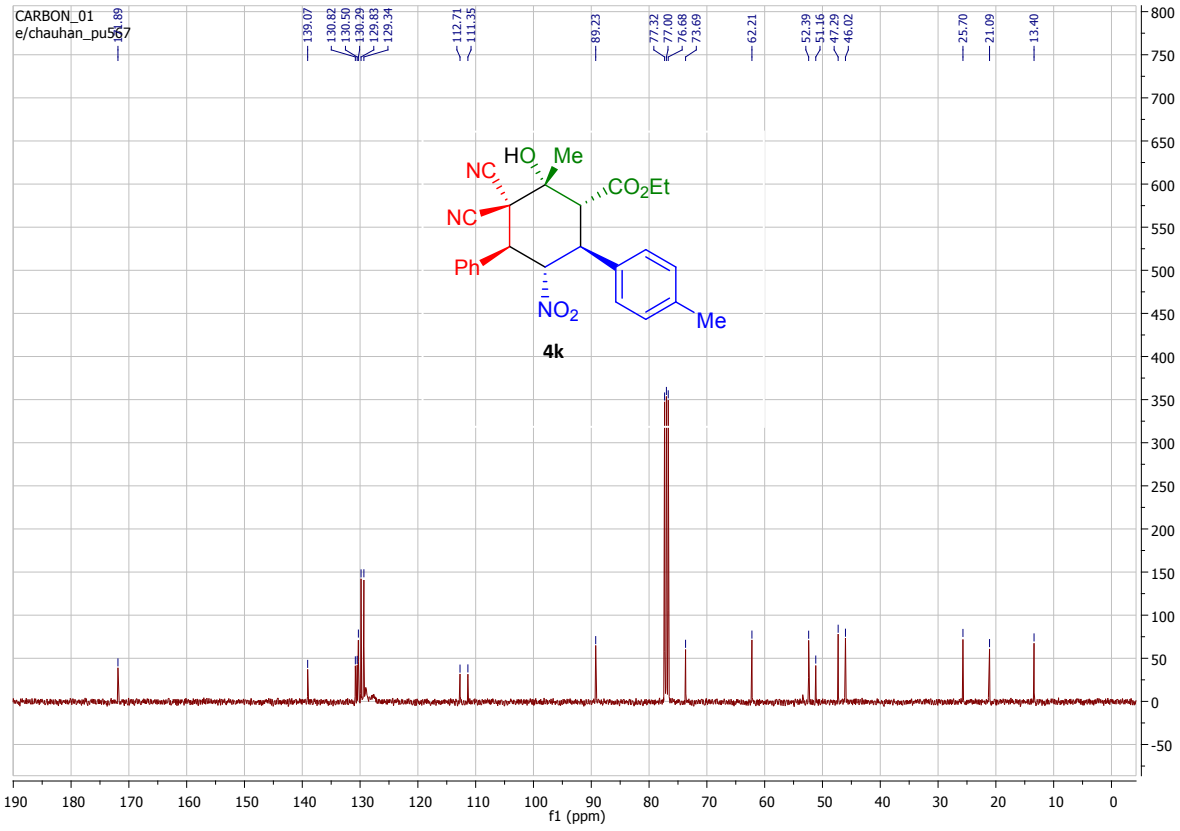

PROTON_01
e/Chauhan_PC646
HO
Me
NC
CO2Et
NC
Ph
NO2
F
4i
f1 (ppm)

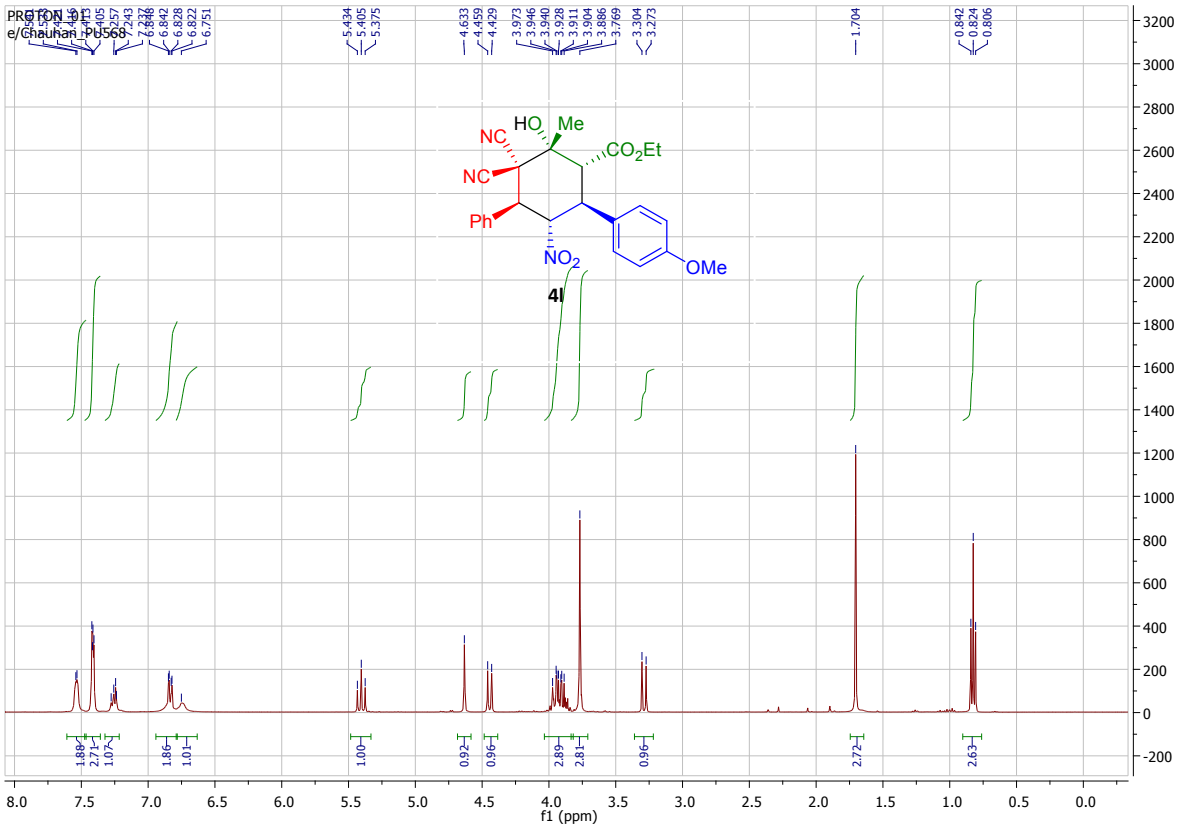

CARBON_01
e/Chauhan_PC646
HO
Me
NC
CO2Et
NC
Ph
NO2
F
4i
f1 (ppm)

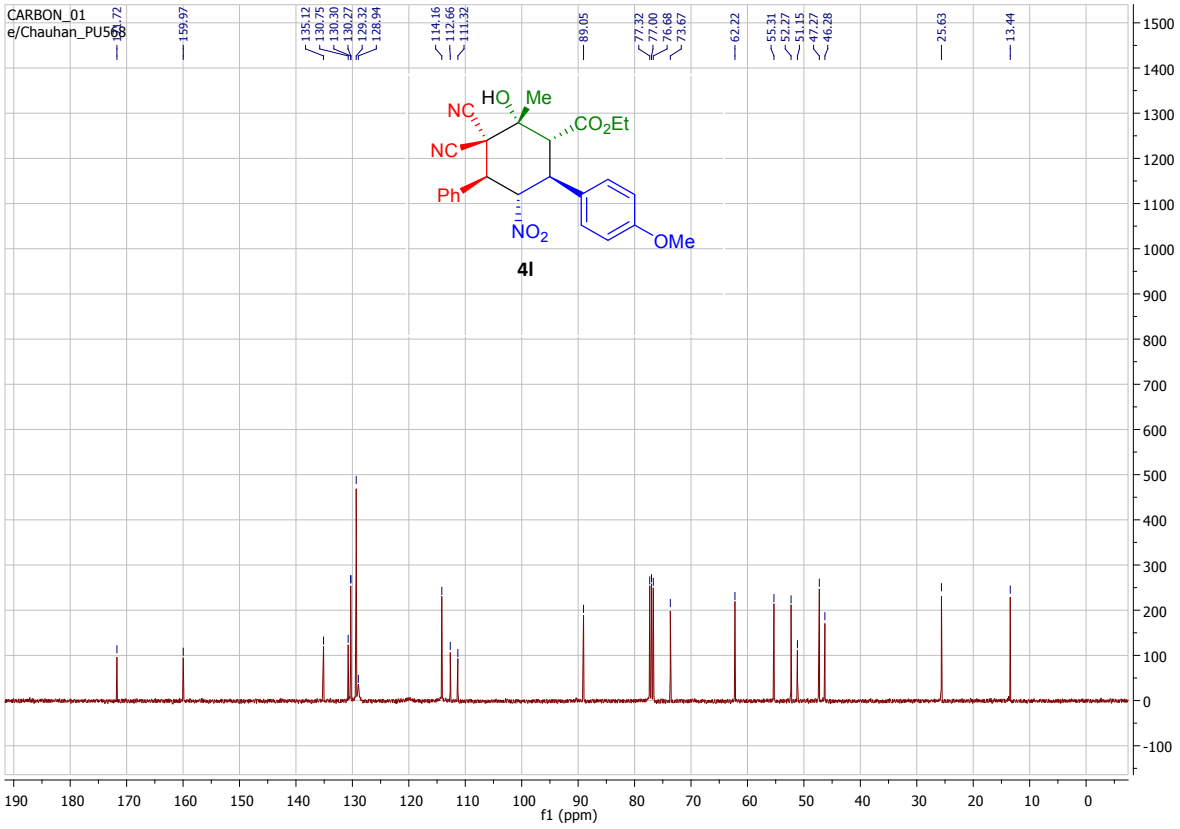

PROTON_01
e/chauhan_pu551
HO
Me
NC
CO2Et
NC
Ph
NO2
Cl
4j
f1 (ppm)

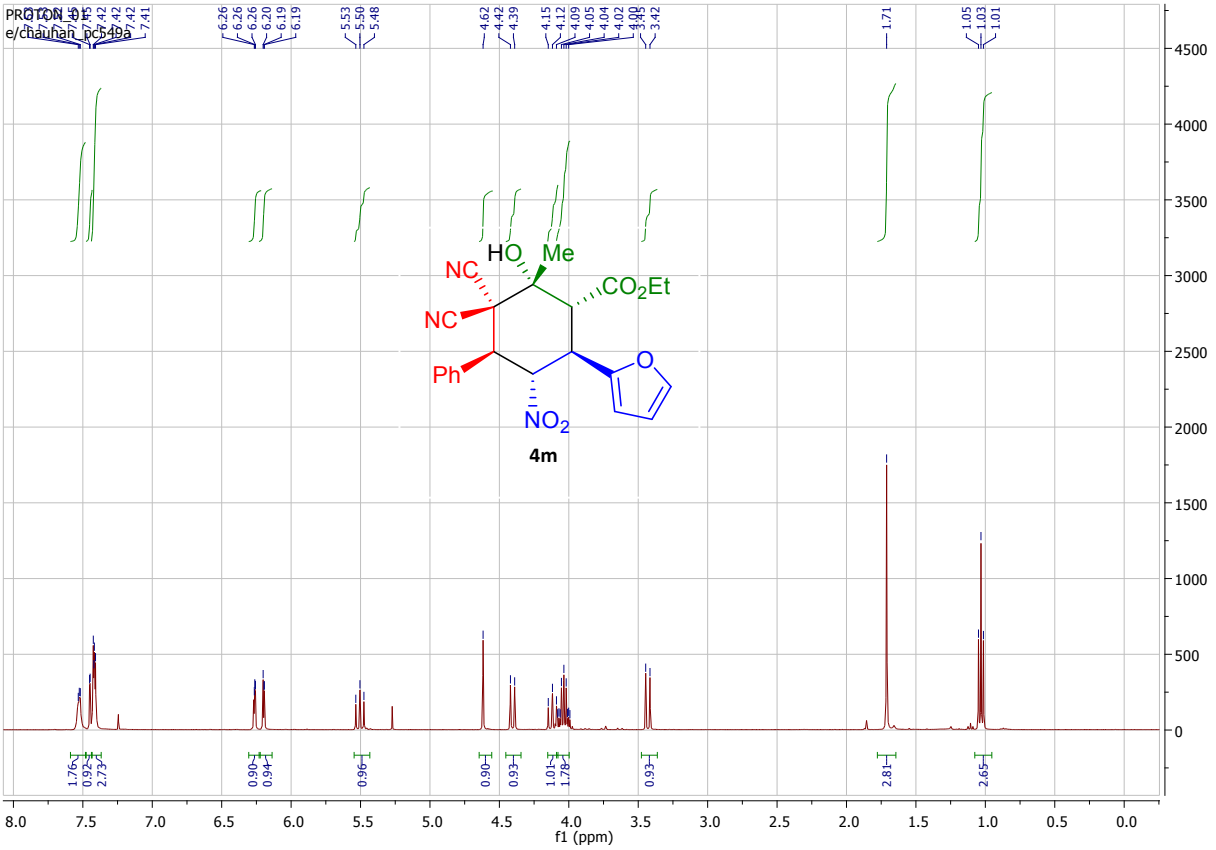

CARBON_01
e/chauhan_pu551
HO
Me
NC
CO2Et
NC
Ph
NO2
Cl
4j
f1 (ppm)

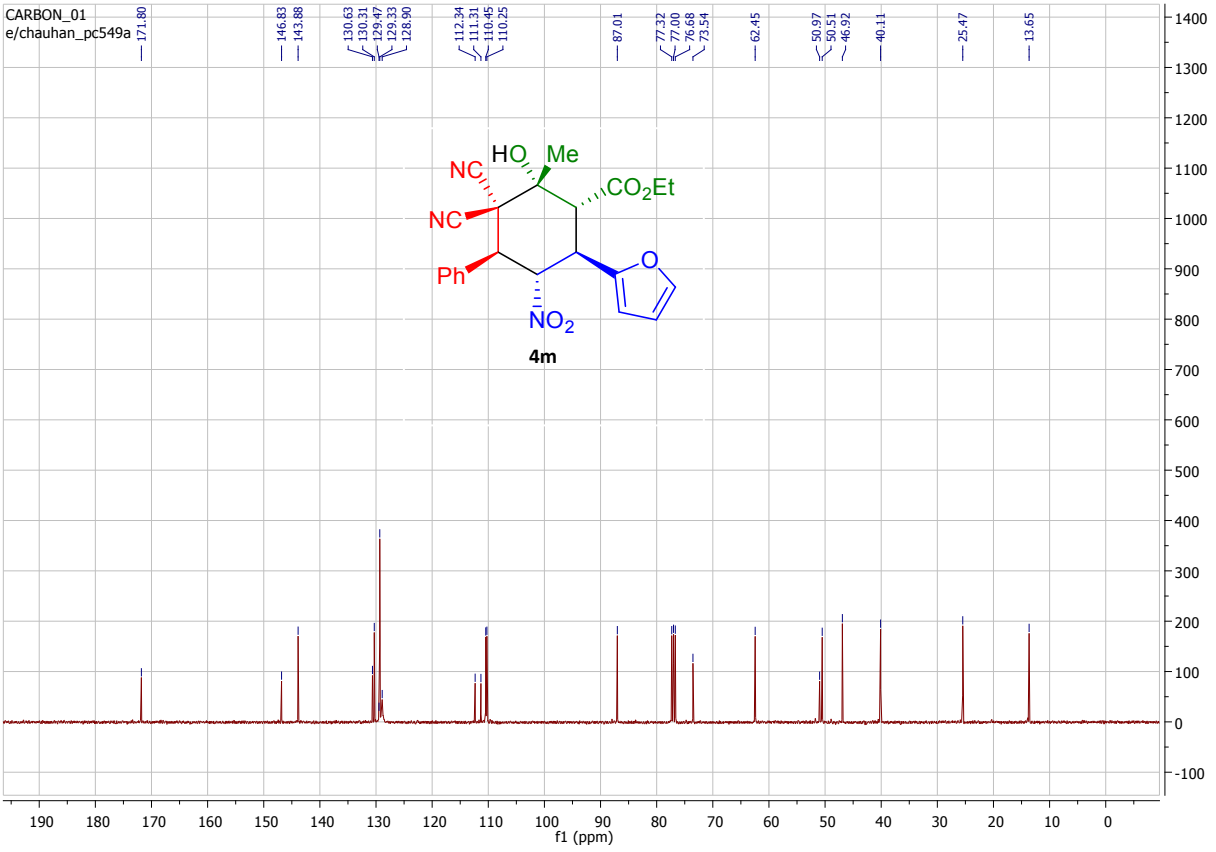

e/chauhan_pu567
HO
Me
NC
CO2Et
NC
Ph
NO2
Me
4k
f1 (ppm)

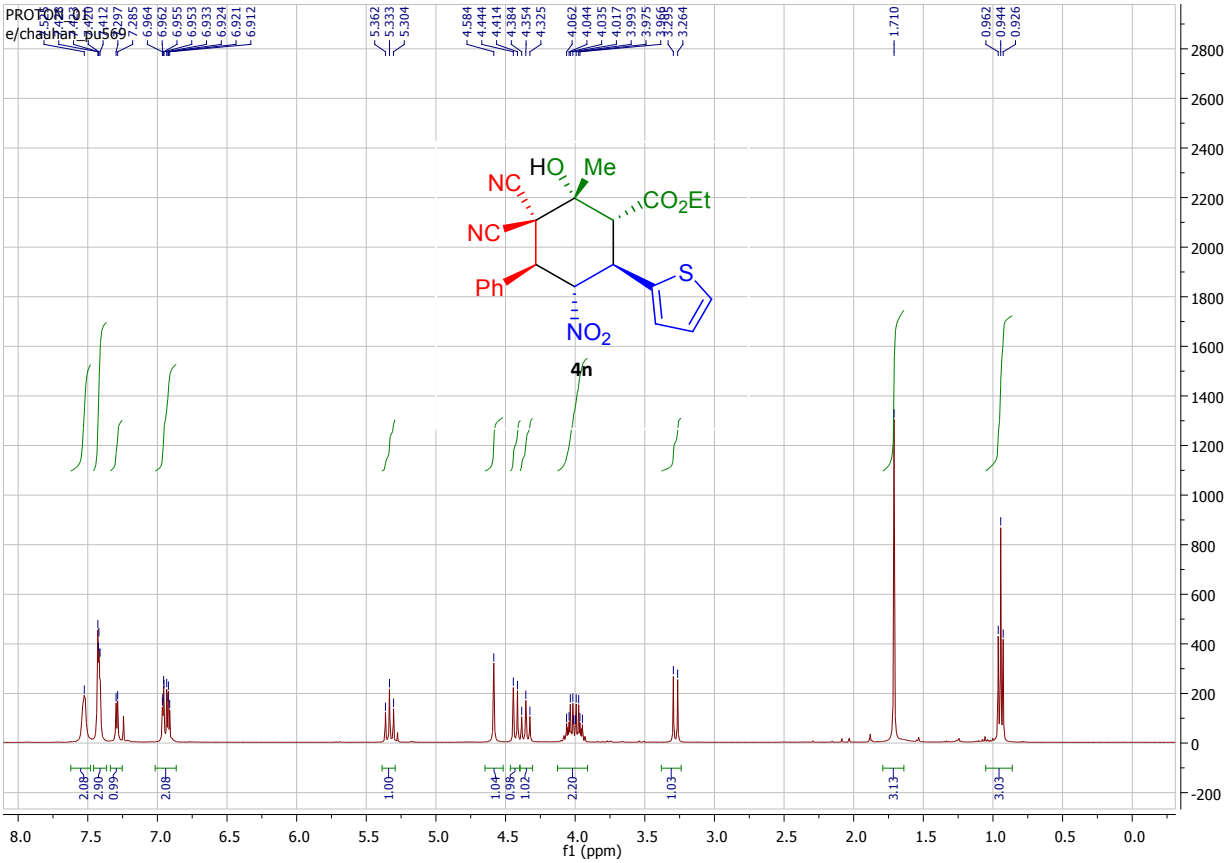

CARBON_01
e/chauhan_pu567
HO
Me
NC
CO2Et
NC
Ph
NO2
Me
4k
f1 (ppm)

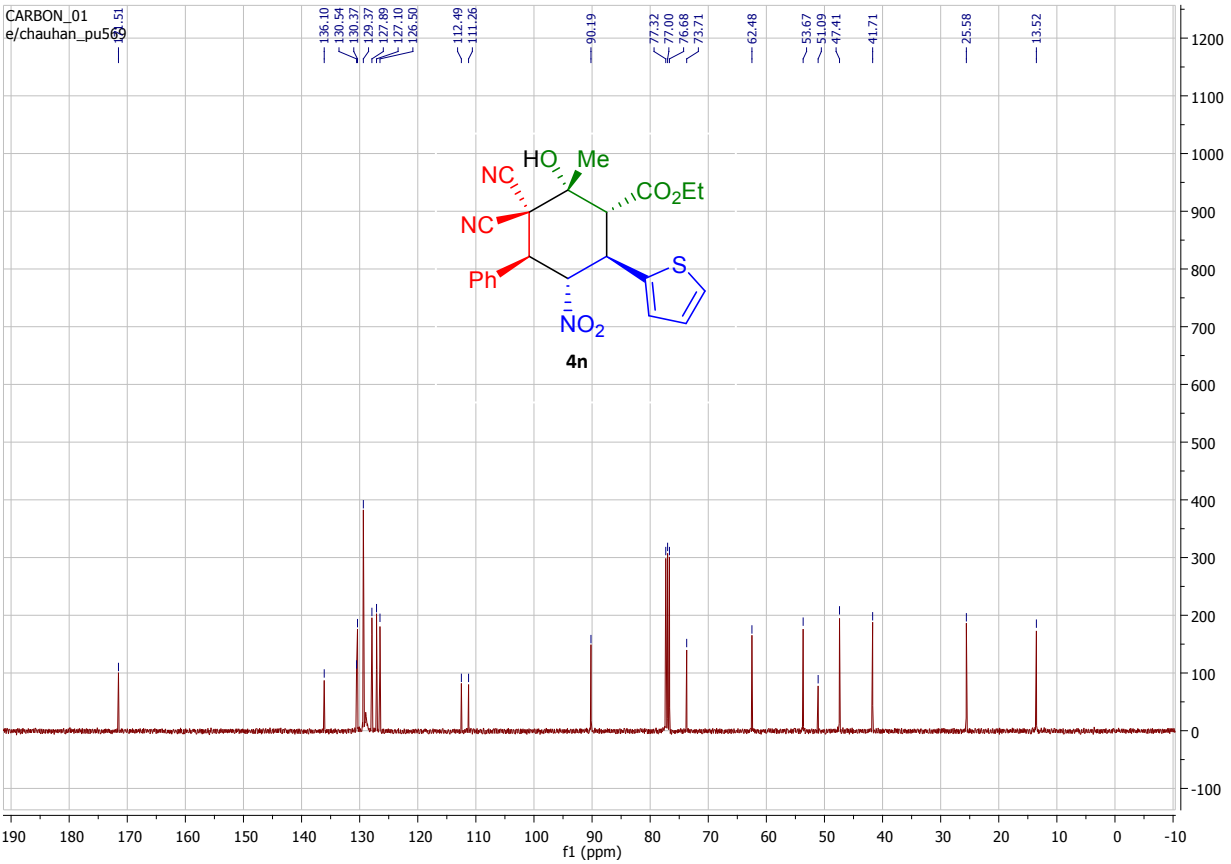

PROTON_01
e/Chauhan_PU568
HO
Me
NC
CO2Et
NC
Ph
NO2
OMe
4l
f1 (ppm)

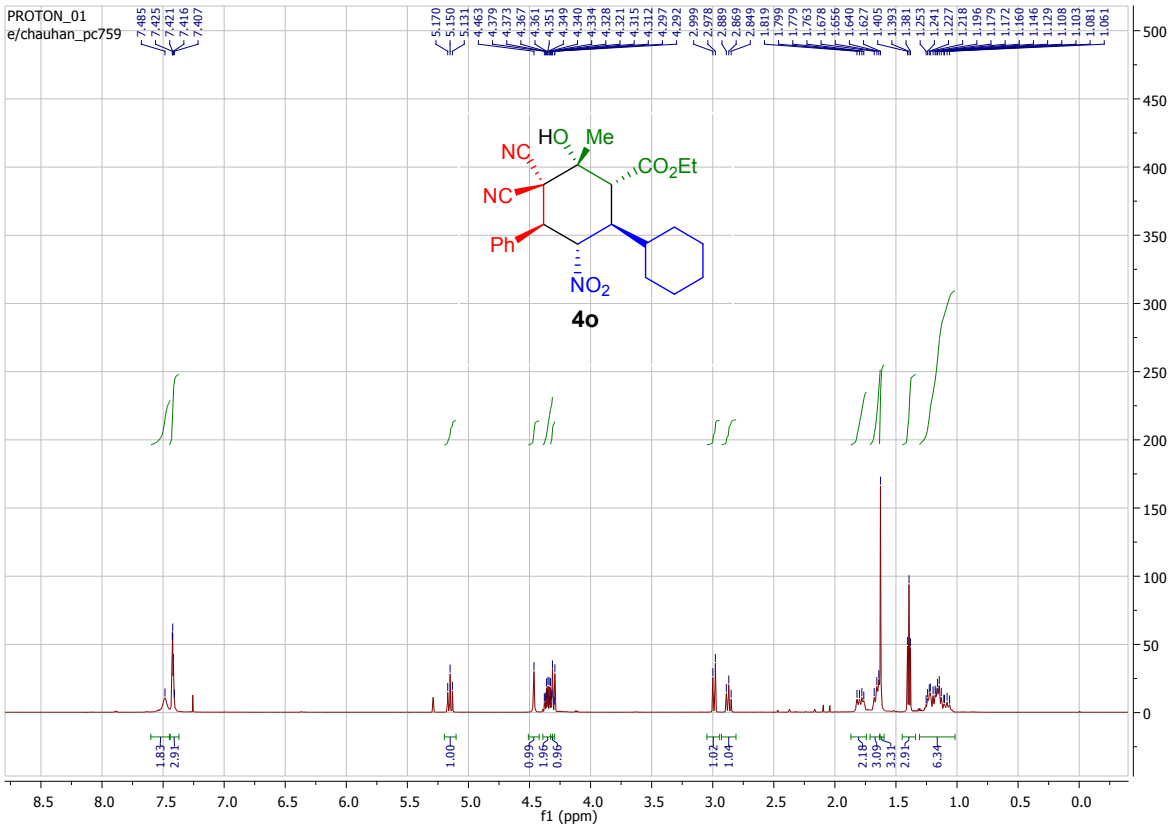

CARBON_01
e/Chauhan_PU568
HO
Me
NC
CO2Et
NC
Ph
NO2
OMe
4l
f1 (ppm)

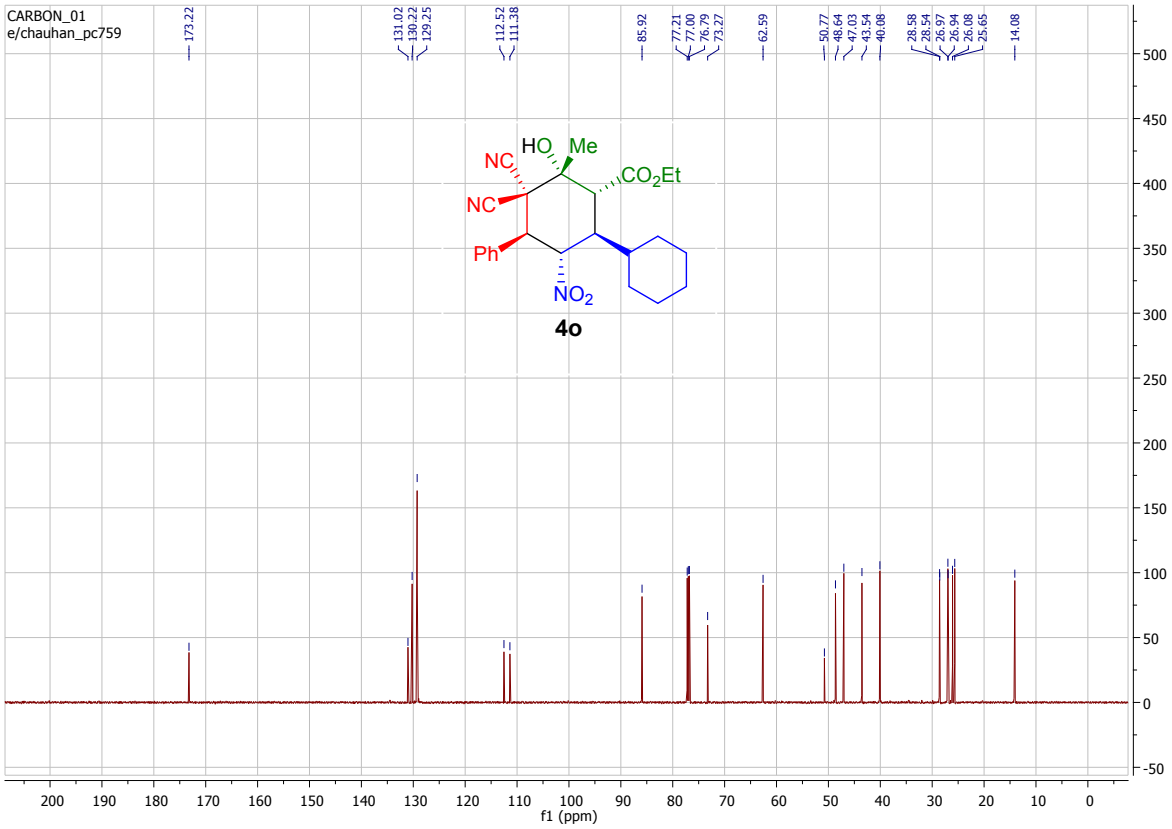

PROTON_01
e/chauhan_pc549a
HO
Me
NC
CO2Et
NC
Ph
O
NO2
4m
f1 (ppm)

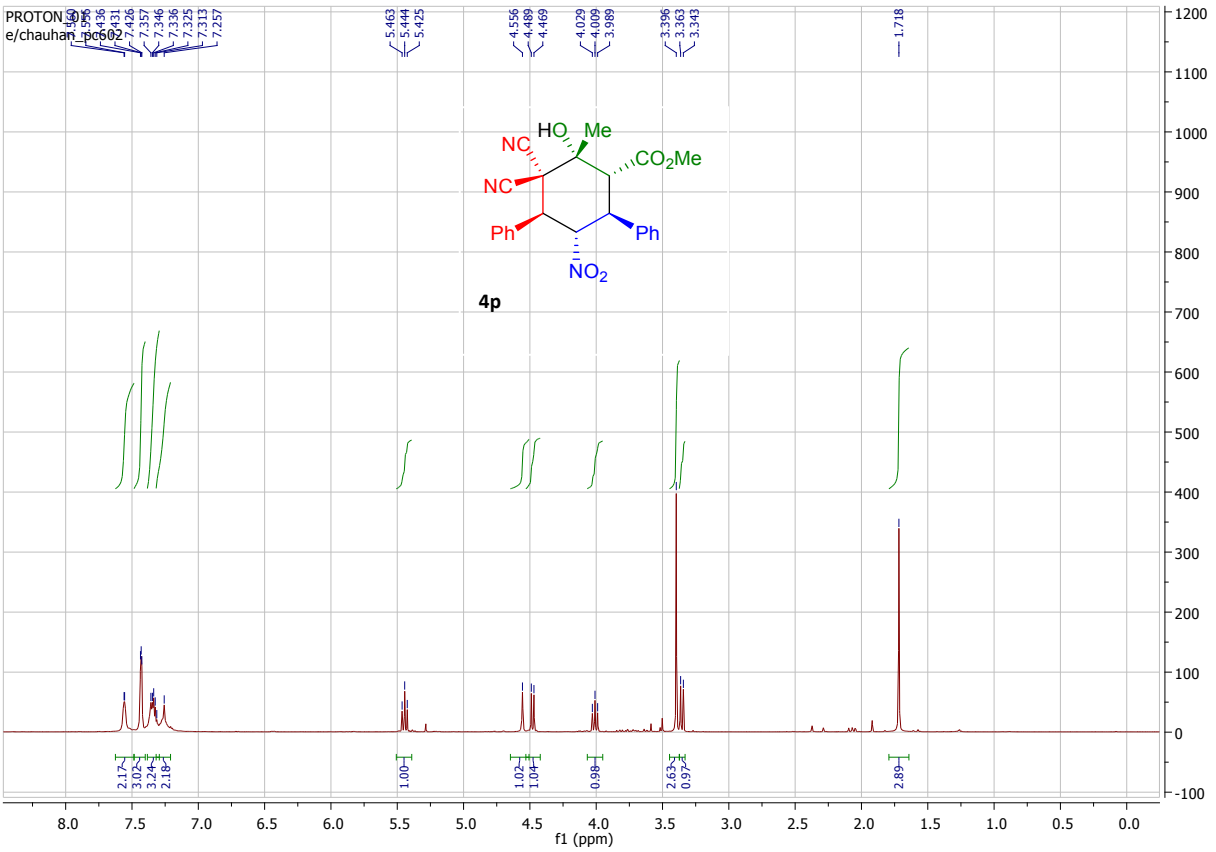

CARBON_01
e/chauhan_pc549a
HO
Me
NC
CO2Et
NC
Ph
O
NO2
4m
f1 (ppm)

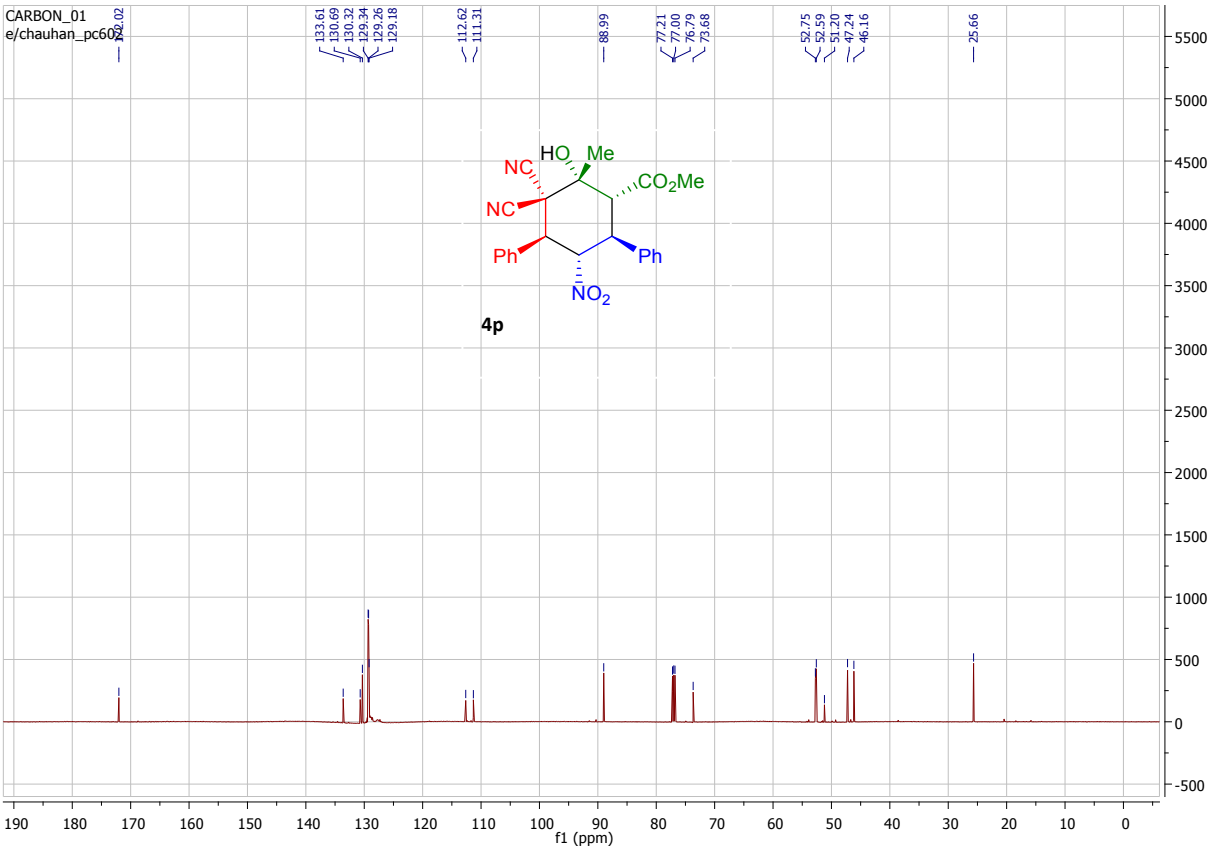

PROTON_01
e/chauhan_pu569
HO
Me
NC
CO2Et
NC
Ph
S
NO2
4n
f1 (ppm)

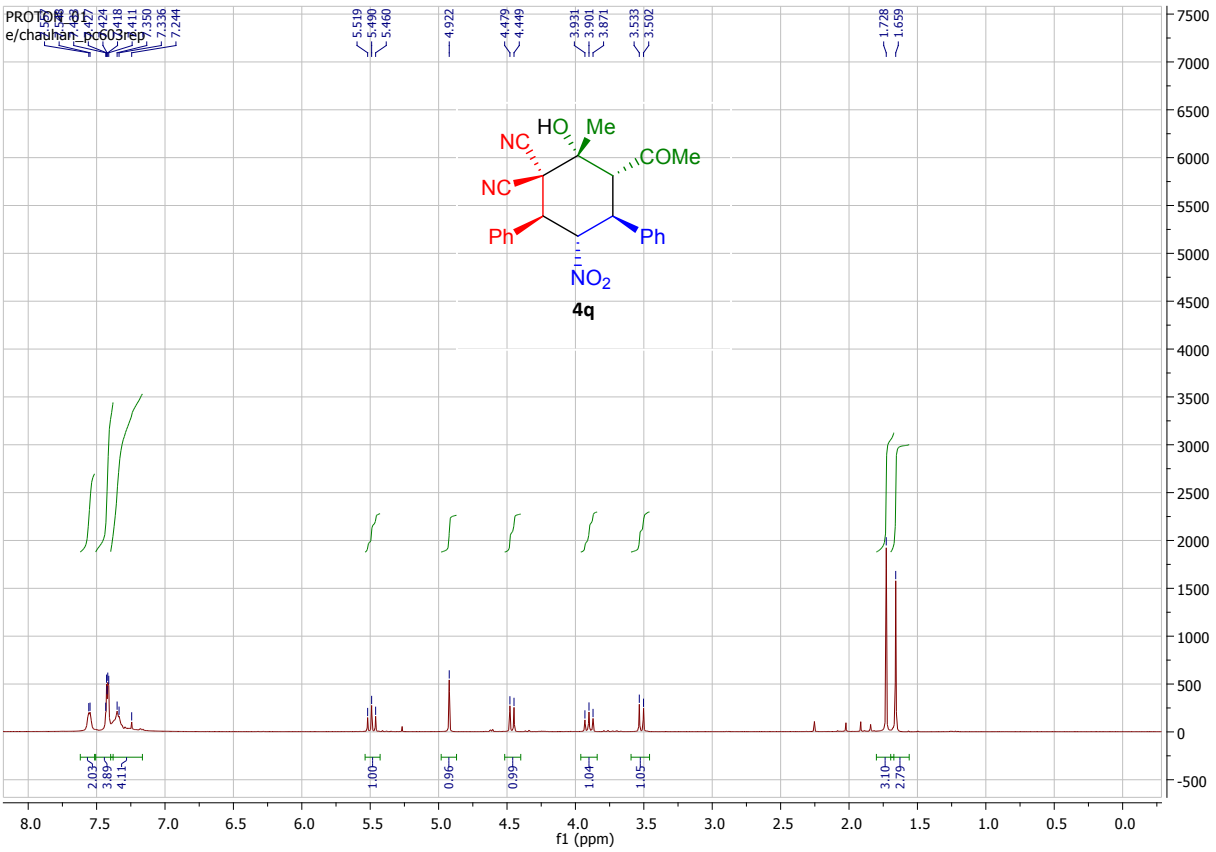

CARBON_01
e/chauhan_pu569
HO
Me
NC
CO2Et
NC
Ph
S
NO2
4n
f1 (ppm)

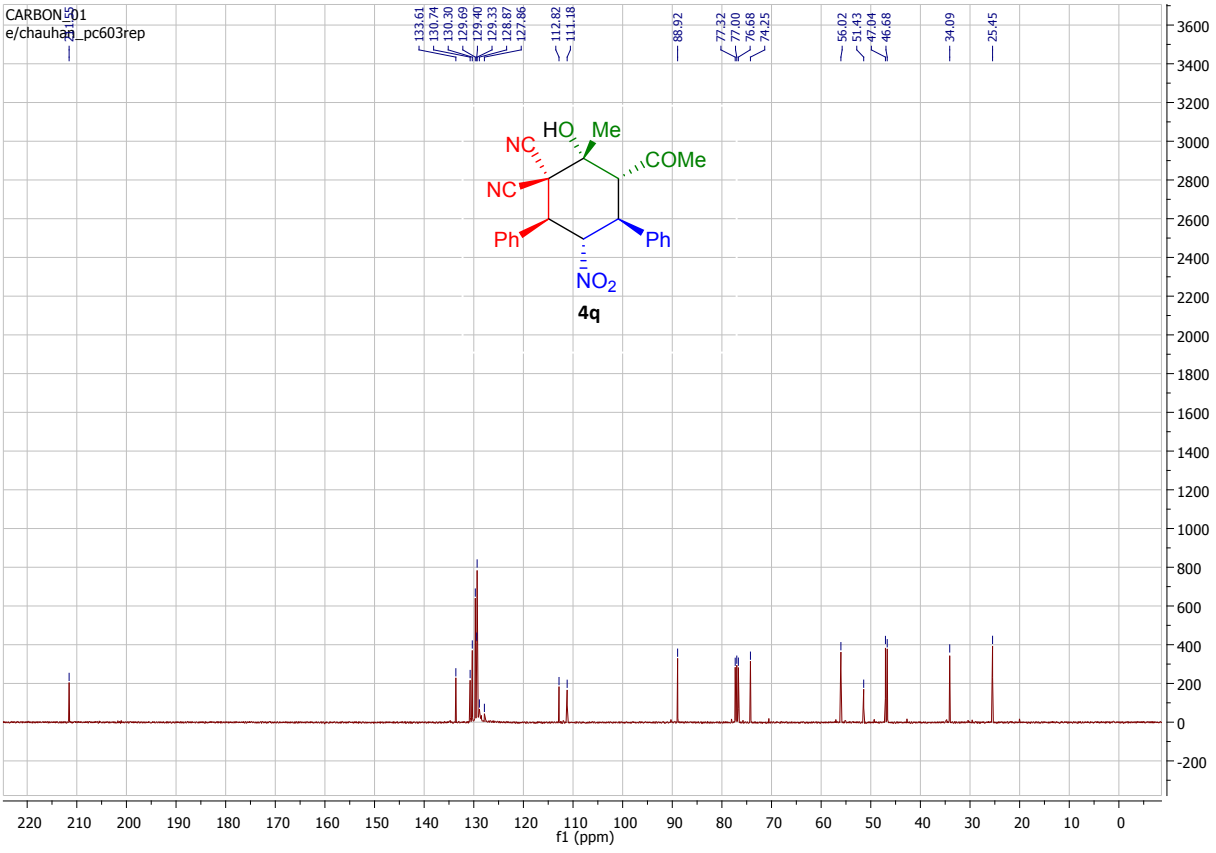

PROTON_01
e/chauhan_pc759
HO
Me
NC
CO2Et
NC
Ph
NO2
4o
f1 (ppm)

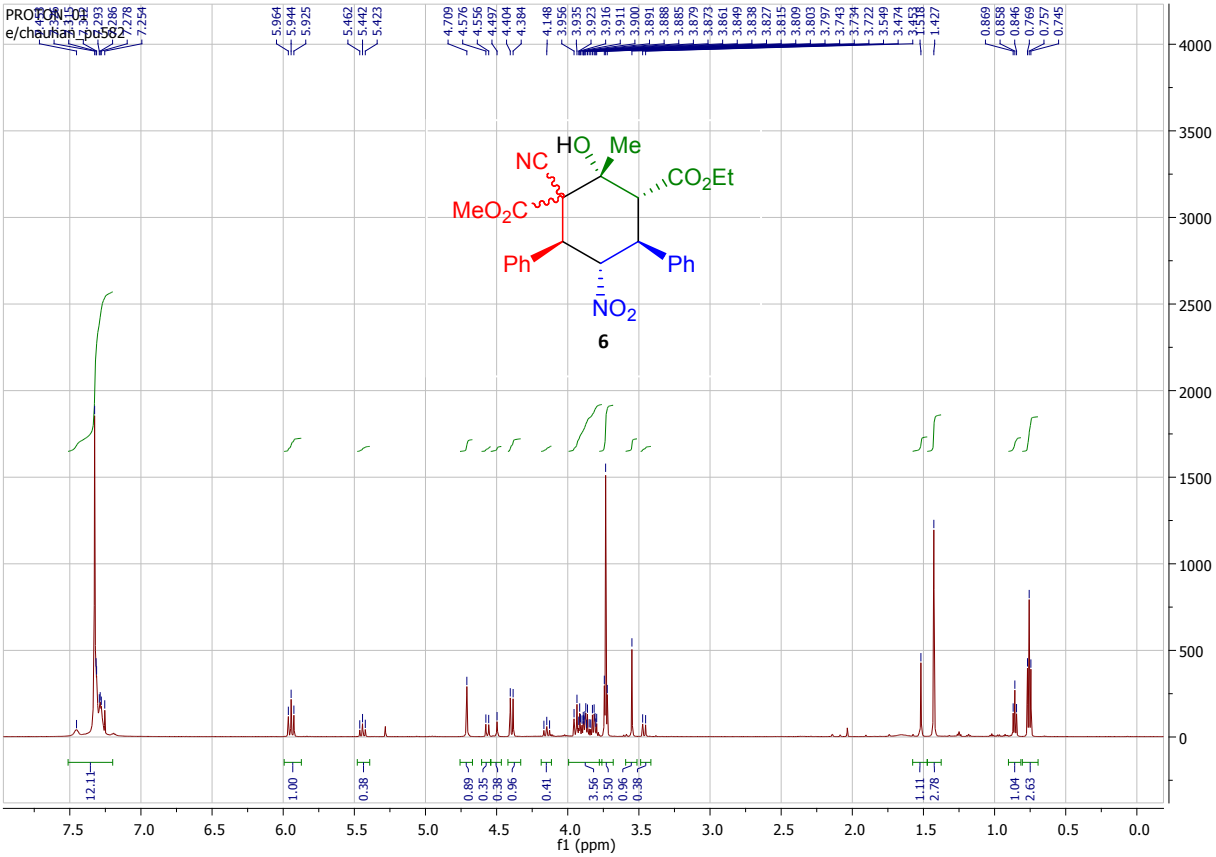

CARBON_01
e/chauhan_pc759
HO
Me
NC
CO2Et
NC
Ph
NO2
4o
f1 (ppm)

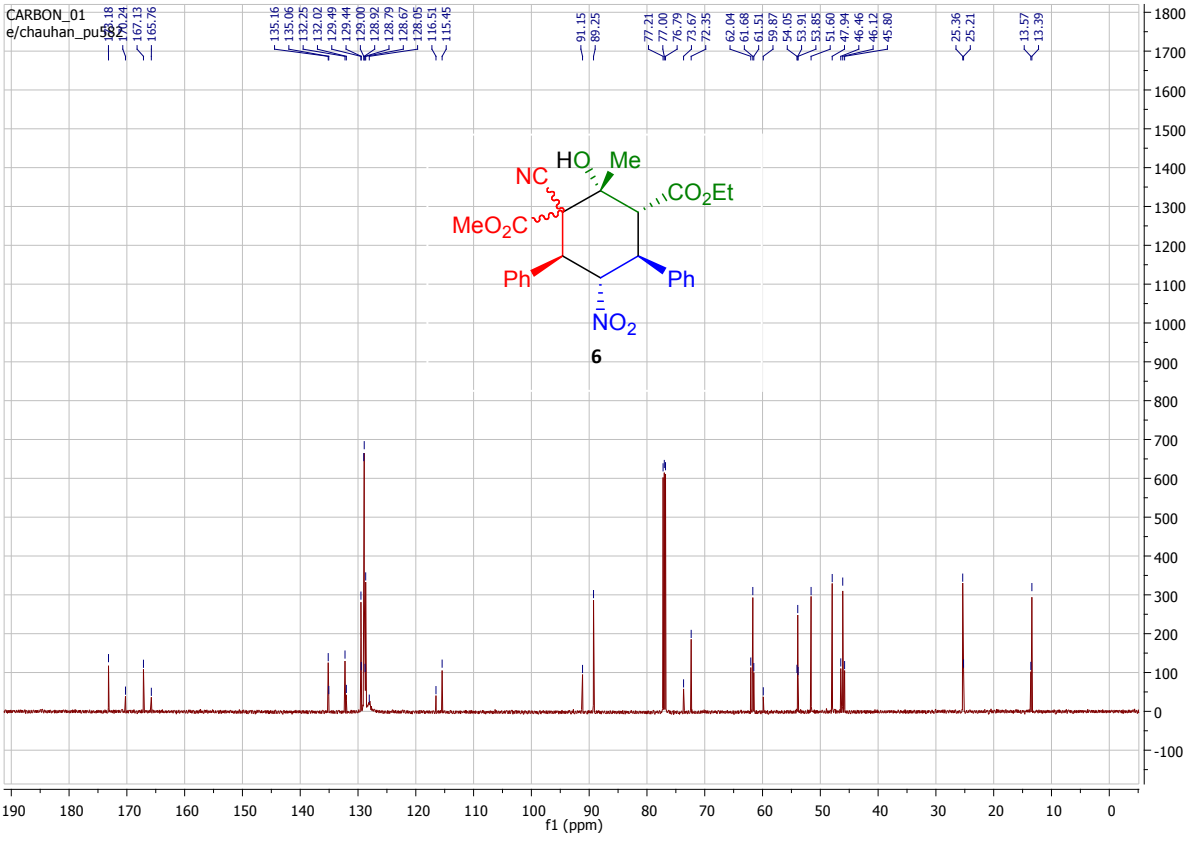

PROTON_01
e/chauhan_pc602
5.463
5.444
5.425
4.556
4.489
4.469
4.029
4.009
3.989
3.396
3.363
3.343
1.718
HO
Me
NC
CO2Me
NC
Ph
Ph
NO2
4p
f1 (ppm)


CARBON_01
e/chauhan_pc602
22.02
133.61
130.69
130.32
129.34
129.26
129.18
112.62
111.31
88.99
77.21
77.00
76.79
73.68
52.75
52.59
51.20
47.24
46.16
25.66
HO
Me
NC
CO2Me
NC
Ph
Ph
NO2
4p
f1 (ppm)


PROTON_01
e/chauhan_pc603rep
HO
Me
NC
COMe
NC
Ph
Ph
NO2
4q
f1 (ppm)


CARBON_01
e/chauhan_pc603rep
HO
Me
NC
COMe
NC
Ph
Ph
NO2
4q
f1 (ppm)


PROTON_02
e/chauhan_pu582
HO
Me
NC
CO2Et
MeO2C
Ph
Ph
NO2
6
f1 (ppm)


CARBON_01
e/chauhan_pu582
HO
Me
NC
CO2Et
MeO2C
Ph
Ph
NO2
6
f1 (ppm)

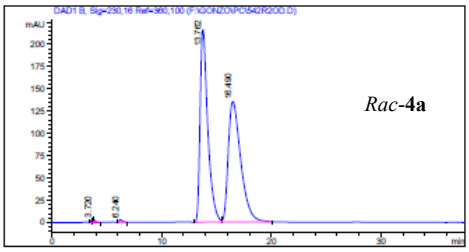
HPLC Chromatograms:

Sample Name: PC 542 rac
Data file: F:\GONZO\PC\542R2OD.D
Sample Info: Laufmittel: n-Heptan/EtOH 97:3;
Die Probe ist in LM/EtOH gelöst.

Säule: DAICELOD.M
Säuleninfo: Chiralcel OD (250x4,6)mm
Operator: Analytik Labor AKEN

Injektion Time: 11:00:54
Injektion Date: 09.09.2013

Instrument Conditions:	At Start	At Stop
Temperature in°C:	30.0°C	30.0°C
Pressure in bar:	26.7	27.1
Flow in ml/min:	1.00	1.00



Rac-**4a**

#	Ret. Time (min)	Width	Height (mAU)	Area (mAU*s)	Area %
1	3.51	0.12	1.77	13.81	0.06
2	3.72	0.14	3.26	29.91	0.14
3	3.85	0.17	2.78	34.97	0.16
4	6.24	0.29	2.95	58.88	0.28
5	13.76	0.73	216.18	10520.42	49.23
6	16.49	1.20	135.32	10714.12	50.13
Total				21372.11	100.00

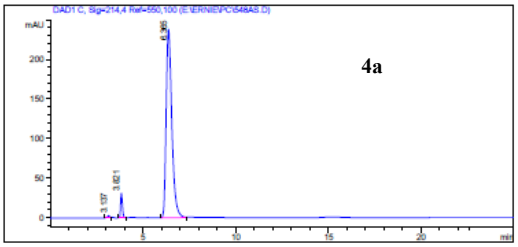
Sample Name: PC 548
Data file: E:\ERNIE\PC\548AS.D
Sample Info: Laufmittel: n-Heptan/EtOH 9:1;
Die Probe ist in DCM/LM gelöst

Säule: DAICELAS.M
Säuleninfo: Chiralpak AS (250 x 4.6)mm 10µ

Operator: Analytik Labor AKEN

Injektion Time: 13:01:01
Injektion Date: 09.09.2013

Instrument Conditions:	At Start	At Stop
Temperature in°C:	30.0	30.0
Pressure in bar:	30.6	31.8
Flow in ml/min:	1.0	1.0



4a

#	Ret. Time (min)	Width	Height (mAU)	Area (mAU*s)	Area %
1	3.14	0.13	3.16	28.75	0.54
2	3.82	0.11	31.44	213.47	4.02
3	6.36	0.32	238.55	5072.51	95.44
Total				5314.73	100.00

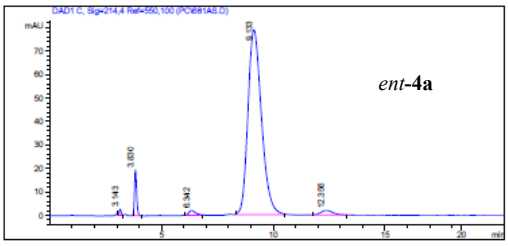
Sample Name: PC 681
Data file: D:\ERNIE\PC\681AS.D
Sample Info: Laufmittel: n-Heptan/EtOH 9:1;
Die Probe ist in DCM/LM gelöst

Säule: DAICELAS.M
Säuleninfo: Chiralpak AS (250 x 4.6)mm 10µ

Operator: Analytik Labor AKEN

Injektion Time: 14:19:13
Injektion Date: 12.02.2014

Instrument Conditions:	At Start	At Stop
Temperature in°C:	30.0	30.0
Pressure in bar:	32.2	31.6
Flow in ml/min:	1.0	1.0



ent-**4a**

#	Ret. Time (min)	Width	Height (mAU)	Area (mAU*s)	Area %
1	3.14	0.10	2.86	20.29	0.56
2	3.83	0.10	19.60	129.61	3.60
3	6.34	0.25	2.11	44.22	1.23
4	9.13	0.64	78.80	3330.01	92.58
5	12.36	0.45	1.93	72.66	2.02
Total				3596.79	100.00

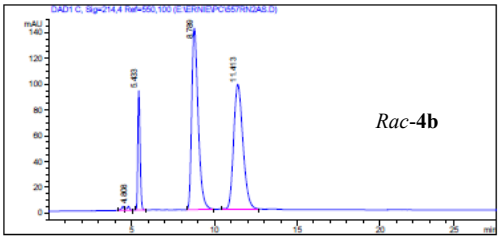
Sample Name: PC 557 rac neu
Data file: E:\ERNIE\PC\557RN2AS.D
Sample Info: Laufmittel: n-Heptan/IP 9:1;
Die Probe ist in DCM/LM gelöst

Säule: DAICELAS.M
Säuleninfo: Chiralpak AS (250 x 4.6)mm 10µ

Operator: Analytik Labor AKEN

Injektion Time: 10:59:52
Injektion Date: 01.10.2013

Instrument Conditions:	At Start	At Stop
Temperature in°C:	30.0	30.0
Pressure in bar:	22.4	22.0
Flow in ml/min:	0.7	0.7



Rac-**4b**

#	Ret. Time (min)	Width	Height (mAU)	Area (mAU*s)	Area %
1	4.47	0.17	3.15	37.17	0.43
2	4.81	0.16	3.16	36.81	0.43
3	5.43	0.15	92.61	894.56	10.43
4	8.79	0.41	140.84	3807.09	44.40
5	11.41	0.61	96.86	3798.22	44.30
Total				8573.85	100.00

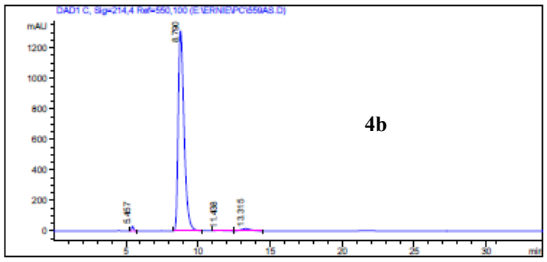
Sample Name: PC 559
Data file: E:\ERNIE\PC\559AS.D
Sample Info: Laufmittel: n-Heptan/EtOH 9:1;
Die Probe ist in DCM/LM gelöst

Säule: DAICELAS.M
Säuleninfo: Chiralpak AS (250 x 4.6)mm 10µ

Operator: Analytik Labor AKEN

Injektion Time: 13:05:26
Injektion Date: 07.10.2013

Instrument Conditions:	At Start	At Stop
Temperature in°C:	30.0	30.0
Pressure in bar:	22.0	22.2
Flow in ml/min:	0.7	0.7



4b

#	Ret. Time (min)	Width	Height (mAU)	Area (mAU*s)	Area %
1	5.46	0.15	30.87	294.17	0.76
2	8.79	0.44	1311.32	37212.43	96.68
3	11.44	0.67	4.24	241.33	0.63
4	13.32	0.61	15.50	742.47	1.93
Total				38490.39	100.00

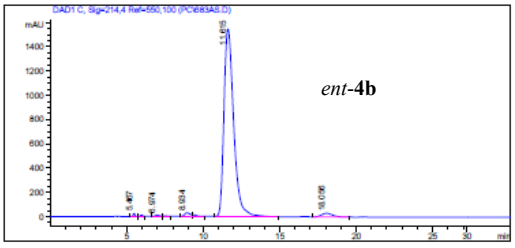
Sample Name: PC 683
Data file: D:\ERNIE\PC\683AS.D
Sample Info: Laufmittel: n-Heptan/EtOH 9:1;
Die Probe ist in DCM/LM gelöst

Säule: DAICELAS.M
Säuleninfo: Chiralpak AS (250 x 4.6)mm 10µ

Operator: Analytik Labor AKEN

Injektion Time: 15:30:06
Injektion Date: 10.02.2014

Instrument Conditions:	At Start	At Stop
Temperature in°C:	30.0	30.0
Pressure in bar:	21.4	21.3
Flow in ml/min:	0.7	0.7



ent-**4b**

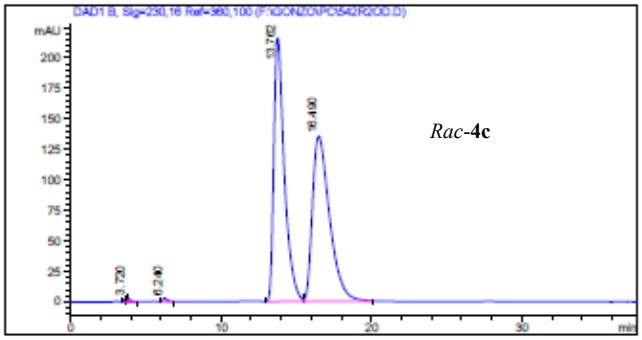
#	Ret. Time (min)	Width	Height (mAU)	Area (mAU*s)	Area %
1	5.47	0.14	27.06	258.99	0.36
2	5.94	0.21	14.78	210.37	0.29
3	6.97	0.33	14.93	364.23	0.50
4	7.50	0.32	7.11	171.90	0.24
5	8.93	0.42	32.38	937.68	1.30
6	9.36	0.33	16.39	386.45	0.53
7	11.61	0.68	1547.55	68358.58	94.57
8	18.06	0.70	31.36	1592.19	2.20
Total				72280.40	100.00

Sample Name: PC 542 rac
Data file: F:\GONZO\PC\542R2OD.D
Sample Info: Laufmittel: n-Heptan/EtOH 97:3;
Die Probe ist in LM/EtOH gelöst.

Säule: DAICELOD.M
Säuleninfo: Chiralcel OD (250x4,6)mm
Operator: Analytik Labor AKEN

Injektion Time: 11:00:54
Injektion Date: 09.09.2013

Instrument Conditions:	At Start	At Stop
Temperature in°C:	30.0°C	30.0°C
Pressure in bar:	26.7	27.1
Flow in ml/min:	1.00	1.00



Rac-**4c**

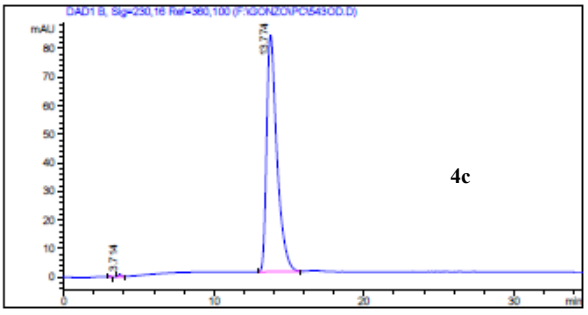
#	Ret. Time (min)	Width	Height (mAU)	Area (mAU*s)	Area %
1	3.51	0.12	1.77	13.81	0.06
2	3.72	0.14	3.26	29.91	0.14
3	3.85	0.17	2.78	34.97	0.16
4	6.24	0.29	2.95	58.88	0.28
5	13.76	0.73	216.18	10520.42	49.23
6	16.49	1.20	135.32	10714.12	50.13
Total				21372.11	100.00

Sample Name: PC 543
Data file: F:\GONZO\PC\543OD.D
Sample Info: Laufmittel: n-Heptan/EtOH 97:3;
Die Probe ist in LM/EtOH gelöst.

Säule: DAICELOD.M
Säuleninfo: Chiralcel OD (250x4,6)mm
Operator: Analytik Labor AKEN

Injektion Time: 13:51:47
Injektion Date: 09.09.2013

Instrument Conditions:	At Start	At Stop
Temperature in°C:	30.0°C	30.0°C
Pressure in bar:	26.7	27.1
Flow in ml/min:	1.00	1.00



4c

#	Ret. Time (min)	Width	Height (mAU)	Area (mAU*s)	Area %
1	3.04	0.12	0.36	2.57	0.06
2	3.71	0.16	0.96	11.53	0.29
3	13.77	0.72	82.64	3952.43	99.64
Total				3966.52	100.00

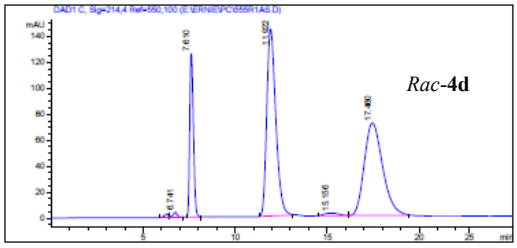
Sample Name: PC 555 rac
Data file: E:\ERNIE\PC\555R1AS.D
Sample Info: Laufmittel: n-Heptan/EtOH 9:1;
Die Probe ist in DCM/LM gelöst

Säule: DAICELAS.M
Säuleninfo: Chiralpak AS (250 x 4.6)mm 10µ

Operator: Analytik Labor AKEN

Injektion Time: 15:17:30
Injektion Date: 07.10.2013

Instrument Conditions:	At Start	At Stop
Temperature in°C:	30.0	30.0
Pressure in bar:	15.8	16.0
Flow in ml/min:	0.5	0.5



Rac-**4d**

#	Ret. Time (min)	Width	Height (mAU)	Area (mAU*s)	Area %
1	6.27	0.23	2.90	45.14	0.38
2	6.74	0.23	4.06	64.63	0.55
3	7.61	0.22	126.05	1765.82	14.94
4	11.92	0.52	143.78	4900.99	41.47
5	15.16	0.69	1.87	103.13	0.87
6	17.46	1.01	71.25	4937.68	41.78
Total				11817.39	100.00

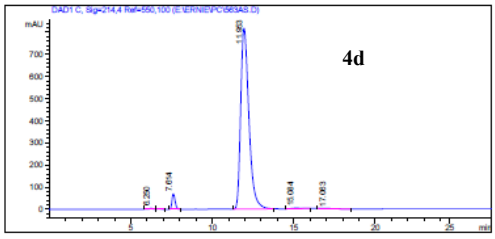
Sample Name: PC 563
Data file: E:\ERNIE\PC\563AS.D
Sample Info: Laufmittel: n-Heptan/EtOH 9:1;
Die Probe ist in DCM/LM gelöst

Säule: DAICELAS.M
Säuleninfo: Chiralpak AS (250 x 4.6)mm 10µ

Operator: Analytik Labor AKEN

Injektion Time: 08:13:45
Injektion Date: 08.10.2013

Instrument Conditions:	At Start	At Stop
Temperature in°C:	30.0	30.0
Pressure in bar:	15.6	15.4
Flow in ml/min:	0.5	0.5



4d

#	Ret. Time (min)	Width	Height (mAU)	Area (mAU*s)	Area %
1	6.25	0.24	4.67	81.22	0.27
2	6.72	0.21	2.23	32.30	0.11
3	7.61	0.21	69.29	920.49	3.09
4	11.95	0.54	815.83	28549.85	95.86
5	15.08	0.58	2.48	117.38	0.39
6	17.06	0.95	1.40	80.10	0.27
Total				29781.35	100.00

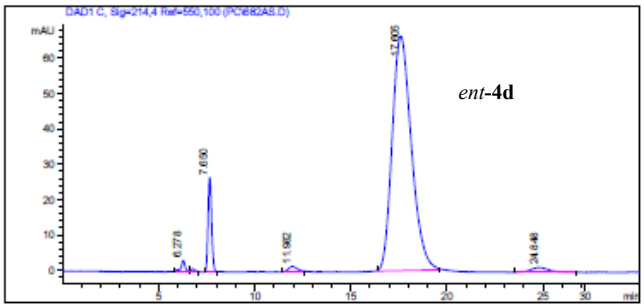
Sample Name: PC 682
Data file: D:\ERNIE\PC\682AS.D
Sample Info: Laufmittel: n-Heptan/EtOH 9:1;
Die Probe ist in DCM/LM gelöst

Säule: DAICELAS.M
Säuleninfo: Chiralpak AS (250 x 4.6)mm 10µ

Operator: Analytik Labor AKEN

Injektion Time: 13:23:37
Injektion Date: 12.02.2014

Instrument Conditions:	At Start	At Stop
Temperature in°C:	30.0	30.0
Pressure in bar:	16.0	16.2
Flow in ml/min:	0.5	0.5



ent-**4d**

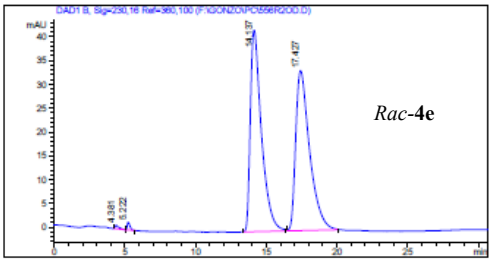
#	Ret. Time (min)	Width	Height (mAU)	Area (mAU*s)	Area %
1	6.28	0.23	3.21	49.96	0.97
2	6.75	0.18	0.89	13.06	0.25
3	7.65	0.20	26.70	341.78	6.61
4	11.98	0.41	1.49	49.62	0.96
5	17.60	0.94	66.37	4622.62	89.39
6	24.85	1.14	1.38	94.38	1.83
Total				5171.42	100.00

Sample Name: PC 556 rac
Data file: F:\GONZO\PC\556R2OD.D
Sample Info: Laufmittel: n-Heptan/IP 95:5;
Die Probe ist in LM/EtOH/DCN gelöst.

Säule: DAICELOD.M
Säuleninfo: Chiralcel OD (250x4,6)mm
Operator: Analytik Labor AKEN

Injektion Time: 08:31:47
Injektion Date: 27.09.2013

Instrument Conditions:	At Start	At Stop
Temperature in°C:	30.0°C	30.0°C
Pressure in bar:	19.9	20.9
Flow in ml/min:	0.70	0.70



Rac-**4e**

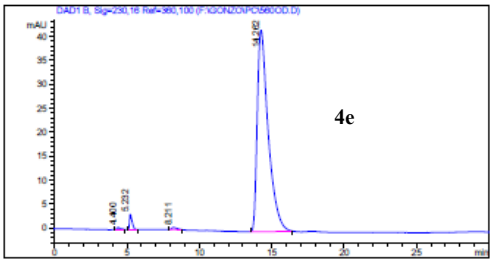
#	Ret. Time (min)	Width	Height (mAU)	Area (mAU*s)	Area %
1	4.38	0.22	0.80	13.45	0.29
2	5.22	0.21	1.64	21.99	0.47
3	14.14	0.81	42.29	2305.14	49.65
4	17.43	1.02	33.56	2302.48	49.59
Total				4643.06	100.00

Sample Name: PC 560
Data file: F:\GONZO\PC\560OD.D
Sample Info: Laufmittel: n-Heptan/IP 95:5;
Die Probe ist in LM/EtOH/DCN gelöst.

Säule: DAICELOD.M
Säuleninfo: Chiralcel OD (250x4,6)mm
Operator: Analytik Labor AKEN

Injektion Time: 14:56:47
Injektion Date: 27.09.2013

Instrument Conditions:	At Start	At Stop
Temperature in°C:	30.0°C	30.0°C
Pressure in bar:	20.3	20.5
Flow in ml/min:	0.70	0.70



4e

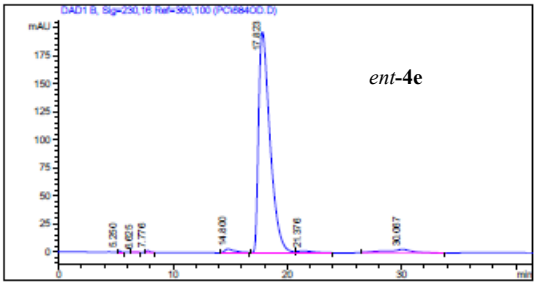
#	Ret. Time (min)	Width	Height (mAU)	Area (mAU*s)	Area %
1	4.40	0.25	0.47	8.02	0.35
2	5.23	0.19	3.19	40.91	1.80
3	8.21	0.36	0.45	11.94	0.53
4	14.26	0.78	41.99	2213.69	97.32
Total				2274.57	100.00

Sample Name: PC 684
Data file: D:\GONZO\PC\684OD.D
Sample Info: Laufmittel: n-Heptan/IP 95:5;
Die Probe ist in EtOH/DCM/LM gelöst.

Säule: DAICELOD.M
Säuleninfo: Chiralcel OD (250x4,6)mm
Operator: Analytik Labor AKEN

Injektion Time: 06:48:19
Injektion Date: 05.02.2002

Instrument Conditions:	At Start	At Stop
Temperature in°C:	30.0°C	30.0°C
Pressure in bar:	18.8	18.6
Flow in ml/min:	0.70	0.70



ent-**4e**

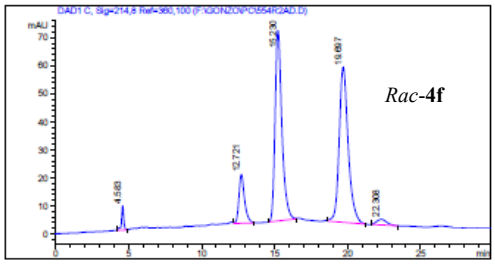
#	Ret. Time (min)	Width	Height (mAU)	Area (mAU*s)	Area %
1	5.25	0.17	1.59	19.21	0.13
2	6.63	0.24	0.30	5.55	0.04
3	7.78	0.24	1.76	30.50	0.21
4	14.80	0.87	3.41	220.36	1.51
5	17.82	1.05	197.05	13710.62	93.76
6	21.38	1.13	1.75	167.88	1.15
7	30.07	1.86	3.05	469.25	3.21
Total				14623.36	100.00

Sample Name: PC 554 rac
Data file: F:\GONZO\PC\554R2AD.D
Sample Info: Laufmittel: n-Heptan/IP 95:5;
Die Probe ist in LM/EtOH/DCM gelöst.

Säule: DAICELAD.M
Säuleninfo: Chiralpak AD (250x4,6)mm
Operator: Analytik Labor AKEN

Injektion Time: 09:08:53
Injektion Date: 09.10.2013

Instrument Conditions:	At Start	At Stop
Temperature in°C:	30.0°C	30.0°C
Pressure in bar:	15.7	16.1
Flow in ml/min:	0.70	0.70



Rac-**4f**

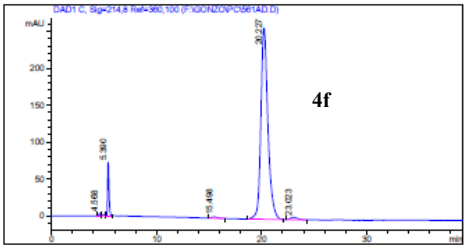
#	Ret. Time (min)	Width	Height (mAU)	Area (mAU*s)	Area %
1	4.58	0.15	8.74	91.37	1.74
2	12.72	0.40	17.52	466.82	8.89
3	15.23	0.49	68.03	2210.69	42.11
4	19.70	0.64	55.47	2383.20	45.40
5	22.31	0.57	2.06	97.22	1.85
Total				5249.30	100.00

Sample Name: PC 561
Data file: F:\GONZO\PC\561AD.D
Sample Info: Laufmittel: n-Heptan/IP 95:5;
Die Probe ist in LM/EtOH/DCM gelöst.

Säule: DAICELAD.M
Säuleninfo: Chiralpak AD (250x4,6)mm
Operator: Analytik Labor AKEN

Injektion Time: 10:08:00
Injektion Date: 11.10.2013

Instrument Conditions:	At Start	At Stop
Temperature in°C:	30.0°C	30.0°C
Pressure in bar:	16.2	16.5
Flow in ml/min:	0.70	0.70



4f

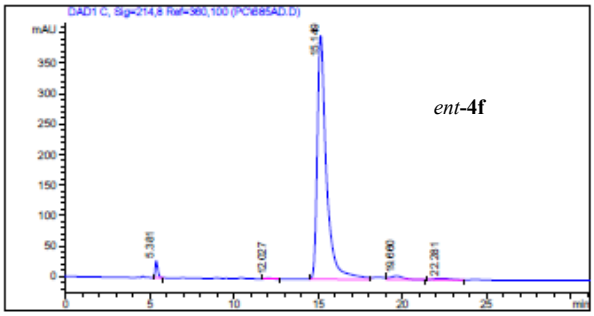
#	Ret. Time (min)	Width	Height (mAU)	Area (mAU*s)	Area %
1	4.57	0.17	2.86	30.80	0.24
2	4.92	0.13	1.67	15.34	0.12
3	5.39	0.14	72.26	683.91	5.27
4	15.50	0.53	1.75	69.09	0.53
5	20.23	0.70	258.44	12038.20	92.71
6	23.02	0.59	3.05	147.42	1.14
Total				12984.77	100.00

Sample Name: PC 685
Data file: D:\GONZO\PC\685AD.D
Sample Info: Laufmittel: n-Heptan/IP 95:5;
Die Probe ist in EtOH/DCM/LM gelöst.

Säule: DAICELAD.M
Säuleninfo: Chiralpak AD (250x4,6)mm
Operator: Analytik Labor AKEN

Injektion Time: 01:53:08
Injektion Date: 06.02.2002

Instrument Conditions:	At Start	At Stop
Temperature in°C:	30.0°C	30.0°C
Pressure in bar:	16.3	17.0
Flow in ml/min:	0.70	0.70



ent-**4f**

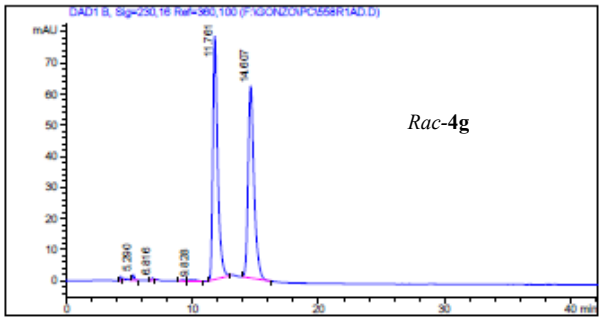
#	Ret. Time (min)	Width	Height (mAU)	Area (mAU*s)	Area %
1	5.38	0.16	28.15	288.06	1.80
2	12.03	0.35	1.44	35.24	0.22
3	15.15	0.55	400.11	15219.53	94.96
4	19.66	0.73	5.90	326.02	2.03
5	22.28	0.81	2.30	158.38	0.99
Total				16027.23	100.00

Sample Name: PC 558 rac
Data file: F:\GONZO\PC\558R1AD.D
Sample Info: Laufmittel: n-Heptan/IP 9:1;
Die Probe ist in EtOH/LM gelöst.

Säule: DAICELAD.M
Säuleninfo: Chiralpak AD (250x4,6)mm
Operator: Analytik Labor AKEN

Injektion Time: 13:40:41
Injektion Date: 04.12.2013

Instrument Conditions:	At Start	At Stop
Temperature in°C:	30.0°C	30.0°C
Pressure in bar:	20.0	20.4
Flow in ml/min:	0.70	0.70



Rac-**4g**

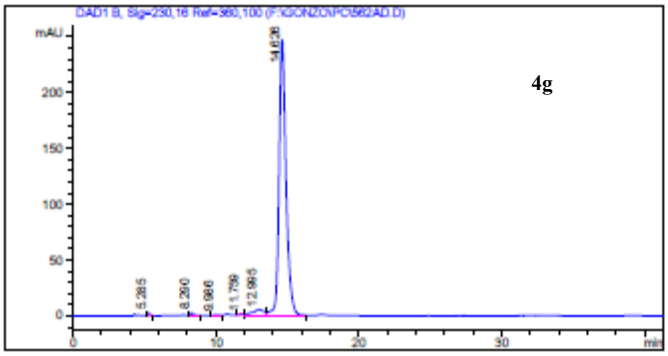
#	Ret. Time (min)	Width	Height (mAU)	Area (mAU*s)	Area %
1	4.30	0.13	1.35	12.08	0.29
2	5.29	0.16	1.88	20.77	0.50
3	6.82	0.20	0.82	10.73	0.26
4	9.24	0.34	0.40	9.33	0.22
5	9.83	0.54	0.47	19.40	0.46
6	11.76	0.39	78.19	2083.47	49.81
7	14.61	0.49	61.75	2027.39	48.47
Total				4183.15	100.00

Sample Name: PC 562
Data file: F:\GONZO\PC\562AD.D
Sample Info: Laufmittel: n-Heptan/IP 9:1;
Die Probe ist in EtOH/LM gelöst.

Säule: DAICELAD.M
Säuleninfo: Chiralpak AD (250x4,6)mm
Operator: Analytik Labor AKEN

Injektion Time: 14:26:17
Injektion Date: 04.12.2013

Instrument Conditions:	At Start	At Stop
Temperature in°C:	30.0°C	30.0°C
Pressure in bar:	20.2	20.5
Flow in ml/min:	0.70	0.70



4g

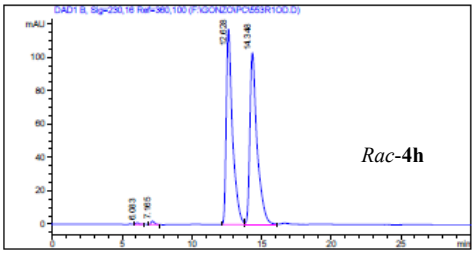
#	Ret. Time (min)	Width	Height (mAU)	Area (mAU*s)	Area %
1	5.28	0.14	2.57	24.36	0.28
2	8.29	0.26	2.38	40.07	0.46
3	9.99	0.35	0.67	16.66	0.19
4	11.76	0.27	0.70	11.52	0.13
5	12.99	0.80	5.44	319.81	3.64
6	14.63	0.50	247.02	8375.73	95.31
Total				8788.14	100.00

Sample Name: PC 553 rac
Data file: F:\GONZO\PC\553R10D.D
Sample Info: Laufmittel: n-Heptan/EtOH 9:1;
Die Probe ist in LM gelöst

Säule: DAICELOD.M
Säuleninfo: Chiralcel OD (250x4,6)mm
Operator: Analytik Labor AKEN

Injektion Time: 13:34:46
Injektion Date: 16.12.2013

Instrument Conditions:	At Start	At Stop
Temperature in°C:	30.0°C	30.0°C
Pressure in bar:	13.4	13.9
Flow in ml/min:	0.50	0.50



Rac-**4h**

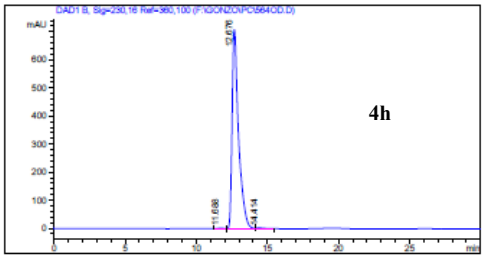
#	Ret. Time (min)	Width	Height (mAU)	Area (mAU*s)	Area %
1	6.08	0.26	1.05	18.55	0.25
2	7.16	0.25	2.00	33.14	0.45
3	12.63	0.45	116.98	3660.92	49.31
4	14.35	0.52	102.81	3712.31	50.00
Total				7424.93	100.00

Sample Name: PC 564
Data file: F:\GONZO\PC\564OD.D
Sample Info: Laufmittel: n-Heptan/EtOH 9:1;
Die Probe ist in LM gelöst

Säule: DAICELOD.M
Säuleninfo: Chiralcel OD (250x4,6)mm
Operator: Analytik Labor AKEN

Injektion Time: 07:47:17
Injektion Date: 17.12.2013

Instrument Conditions:	At Start	At Stop
Temperature in°C:	30.0°C	30.0°C
Pressure in bar:	13.3	13.6
Flow in ml/min:	0.50	0.50



4h

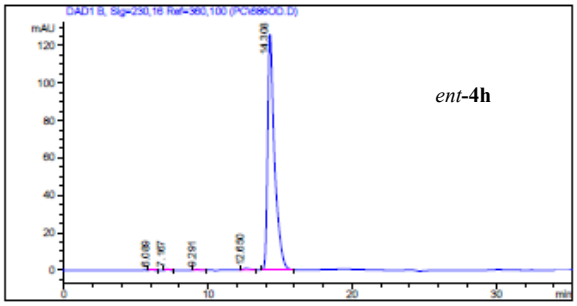
#	Ret. Time (min)	Width	Height (mAU)	Area (mAU*s)	Area %
1	11.69	0.39	2.14	56.13	0.25
2	12.68	0.46	709.15	22451.00	99.23
3	14.41	0.53	3.14	117.71	0.52
Total				22624.84	100.00

Sample Name: PC 686
Data file: D:\GONZO\PC\686OD.D
Sample Info: Laufmittel: n-Heptan/EtOH 9:1;
Die Probe ist in EtOH/DCM/LM gelöst.

Säule: DAICELOD.M
Säuleninfo: Chiralcel OD (250x4,6)mm
Operator: Analytik Labor AKEN

Injektion Time: 13:30:33
Injektion Date: 12.02.2014

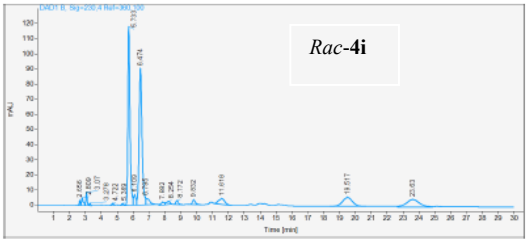
Instrument Conditions:	At Start	At Stop
Temperature in°C:	30.0°C	30.0°C
Pressure in bar:	12.4	12.4
Flow in ml/min:	0.50	0.50



ent-**4h**

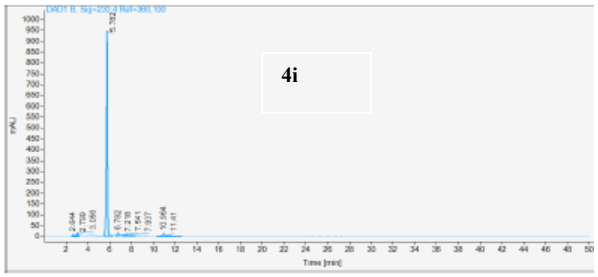
#	Ret. Time (min)	Width	Height (mAU)	Area (mAU*s)	Area %
1	6.09	0.21	0.50	7.51	0.18
2	7.17	0.23	0.65	10.18	0.24
3	9.29	0.32	0.28	7.10	0.17
4	12.65	0.41	0.74	22.03	0.52
5	14.31	0.48	125.83	4201.21	98.90
Total				4248.04	100.00

Sample name: PC 647 rac
Data file: C:\SNOOPY\PC\647R3IC.D
Description: Laufmittel: n-Heptan/EtOH 97:3; Die Probe ist im LM/DCM gelöst.
Injection date: 12/3/2013 2:05:13 PM
Aoq. Analysis method: CHIRALPAKIC1-6LNP.M
Column: Chiralpak IC, (150 x 4,6) mm, 5µ, SN: IC00CD-QF015
Pressure at start: 25 bar Start flow: 0.700 ml/min Column oven: 29.98 °C



RT [min]	Type	Area%	Area	Height	Width [min]
2.66	BV	0.48	15.75	3.40	0.07
2.81	VV	1.28	42.34	5.25	0.12
3.07	VV	1.48	48.72	8.74	0.08
3.28	VB	0.31	10.32	1.70	0.09
4.72	BB	0.37	12.21	1.45	0.12
5.37	BB	0.38	12.39	1.32	0.14
5.73	BV	32.13	1059.33	118.08	0.14
6.11	VV	2.35	77.43	6.84	0.17
6.47	MF	34.03	1121.97	90.08	0.21
6.80	FM	2.35	77.53	4.31	0.30
7.88	MF	1.00	32.89	1.89	0.29
8.25	FM	0.96	31.66	1.87	0.28
8.77	BB	0.96	31.63	2.66	0.18
9.83	BB	1.33	43.93	2.98	0.22
11.62	BB	3.07	101.38	3.75	0.39
19.52	BB	7.89	260.03	5.79	0.70
23.63	MM	9.64	317.93	5.16	1.03
	Sum	100.00	3297.45		

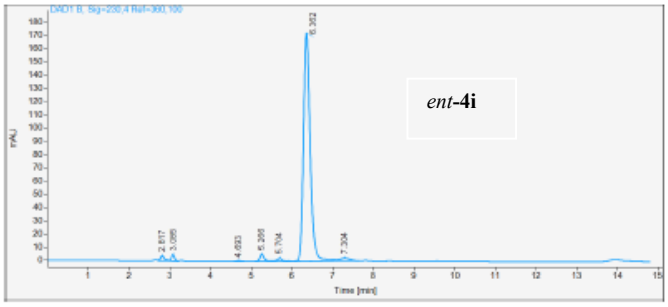
Sample name: PC 646
Data file: C:\SNOOPY\PC\PC 646 IC.D
Description: Laufmittel: n-Heptan/EtOH 97:3; Die Probe ist in DCM/EtOH/LM gelöst.
Injection date: 12/4/2013 8:18:04 AM
Aoq. Analysis method: CHIRALPAKIC1-6LNP.M
Column: Chiralpak IC, (150 x 4,6) mm, 5µ, SN: IC00CD-QF015
Pressure at start: 22 bar Start flow: 0.700 ml/min Column oven: 29.99 °C



Name PC 646

RT [min]	Type	Area%	Area	Height	Width [min]
2.64	BV	0.49	39.49	9.08	0.07
2.80	VV	0.57	45.79	5.98	0.11
3.05	VB	1.19	95.29	18.39	0.08
5.78	VV	87.02	6985.18	947.07	0.11
6.78	VB	2.41	193.44	13.60	0.23
7.22	BV	0.78	62.94	6.17	0.16
7.54	VB	1.35	108.02	9.30	0.18
7.94	BV	1.09	87.52	7.09	0.19
10.96	BV	2.96	237.32	13.58	0.26
11.41	VB	2.15	172.22	9.40	0.27
	Sum	100.00	8027.21		

Sample name: PC 687
Data file: C:\SNOOPY\PC\PC 687 IA.D
Description: Laufmittel: n-Heptan/EtOH 97:3 Die Probe ist in EtOH/LM gelöst.
Injection date: 2/12/2014 1:48:36 PM
Aoq. Analysis method: CHIRALPAKIC1-6LNP.M
Column: Chiralpak IC, (150 x 4,6) mm, 5µ, SN: IC00CD-QF015
Pressure at start: 22 bar Start flow: 0.700 ml/min Column oven: 29.99 °C



Name PC 687

RT [min]	Type	Area%	Area	Height	Width [min]
2.82	VV	1.36	28.79	4.11	0.10
3.09	VV	1.24	26.14	4.94	0.08
4.69	BB	0.17	3.64	0.56	0.10
5.27	BV	2.21	46.68	5.88	0.12
5.70	VB	1.03	21.78	2.65	0.12
6.36	BV	91.93	1943.50	172.30	0.17
7.30	VB	2.06	43.65	2.41	0.27
	Sum	100.00	2114.18		

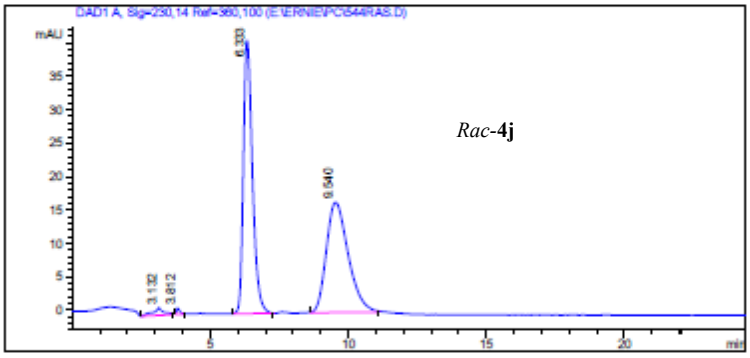
Sample Name: PC 544 rac
Data file: E:\ERNIE\PC\544RAS.D
Sample Info: Laufmittel: n-Heptan/EtOH 9:1;
Die Probe ist in DCM/LM gelöst

Säule: DAICELAS.M
Säuleninfo: Chiralpak AS (250 x 4.6)mm 10µ

Operator: Analytik Labor AKEN

Injektion Time: 09:54:59
Injektion Date: 14.10.2013

Instrument Conditions:	At Start	At Stop
Temperature in°C:	30.0	30.0
Pressure in bar:	29.9	30.1
Flow in ml/min:	1.0	1.0



Rac-**4j**

#	Ret. Time (min)	Width	Height (mAU)	Area (mAU*s)	Area %
1	3.13	0.32	1.17	30.53	1.63
2	3.81	0.12	0.94	8.12	0.43
3	6.33	0.35	40.94	931.00	49.55
4	9.54	0.80	16.52	909.10	48.39
Total				1878.76	100.00

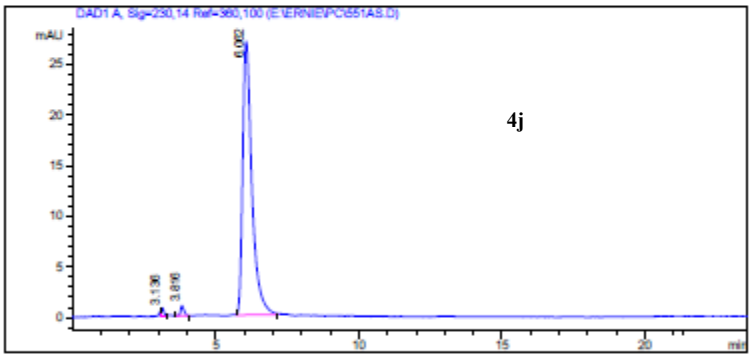
Sample Name: PC 551
Data file: E:\ERNIE\PC\551AS.D
Sample Info: Laufmittel: n-Heptan/EtOH 9:1;
Die Probe ist in DCM/LM gelöst

Säule: DAICELAS.M
Säuleninfo: Chiralpak AS (250 x 4.6)mm 10µ

Operator: Analytik Labor AKEN

Injektion Time: 10:22:49
Injektion Date: 14.10.2013

Instrument Conditions:	At Start	At Stop
Temperature in°C:	30.0	30.0
Pressure in bar:	30.1	30.0
Flow in ml/min:	1.0	1.0



4j

#	Ret. Time (min)	Width	Height (mAU)	Area (mAU*s)	Area %
1	3.14	0.09	0.81	5.23	0.84
2	3.82	0.12	1.01	8.35	1.35
3	6.06	0.33	27.03	605.66	97.81
Total				619.23	100.00

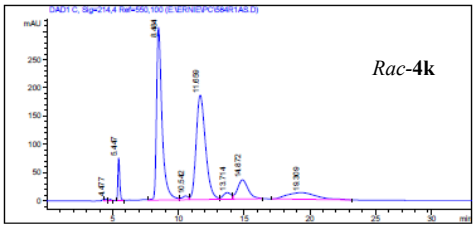
Sample Name: PC 584 rac
Data file: E:\ERNIE\PC\584R1AS.D
Sample Info: Laufmittel: n-Heptan/EtOH 9:1;
Die Probe ist in DCM/LM gelöst

Säule: DAICELAS.M
Säuleninfo: Chiralpak AS (250 x 4.6)mm 10µ

Operator: Analytik Labor AKEN

Injektion Time: 14:13:25
Injektion Date: 13.12.2013

Instrument Conditions:	At Start	At Stop
Temperature in°C:	30.0	30.0
Pressure in bar:	22.6	21.6
Flow in ml/min:	0.7	0.7



Rac-**4k**

#	Ret. Time (min)	Width	Height (mAU)	Area (mAU*s)	Area %
1	4.48	0.16	3.63	40.18	0.17
2	4.82	0.17	2.89	34.00	0.15
3	5.45	0.15	75.53	733.06	3.15
4	8.48	0.43	305.46	8907.90	38.25
5	10.54	0.42	6.76	217.29	0.93
6	11.66	0.75	184.59	9137.15	39.24
7	13.71	0.51	11.58	494.19	2.12
8	14.87	0.78	33.89	1903.92	8.18
9	19.31	1.82	11.70	1818.07	7.81
Total				23285.75	100.00

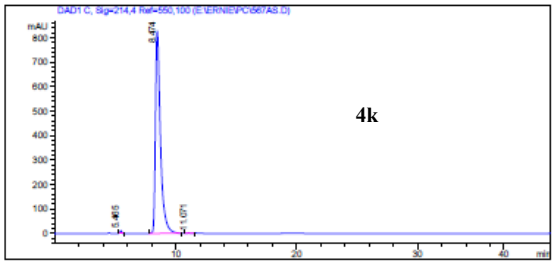
Sample Name: PC 567
Data file: E:\ERNIE\PC\567AS.D
Sample Info: Laufmittel: n-Heptan/EtOH 9:1;
Die Probe ist in DCM/LM gelöst

Säule: DAICELAS.M
Säuleninfo: Chiralpak AS (250 x 4.6)mm 10µ

Operator: Analytik Labor AKEN

Injektion Time: 14:46:57
Injektion Date: 13.12.2013

Instrument Conditions:	At Start	At Stop
Temperature in°C:	30.0	30.0
Pressure in bar:	21.6	21.8
Flow in ml/min:	0.7	0.7



4k

#	Ret. Time (min)	Width	Height (mAU)	Area (mAU*s)	Area %
1	5.46	0.14	11.72	108.95	0.45
2	8.47	0.43	827.49	23890.94	99.47
3	11.07	0.28	0.92	19.24	0.08
Total				24019.13	100.00

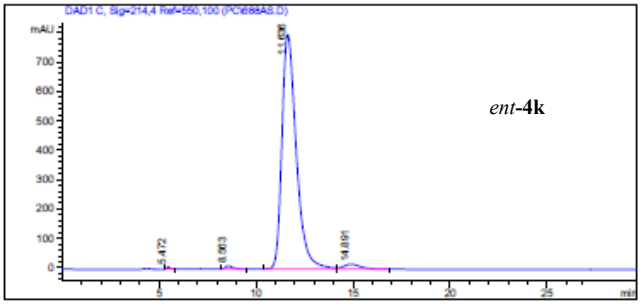
Sample Name: PC 688
Data file: D:\ERNIE\PC\688AS.D
Sample Info: Laufmittel: n-Heptan/EtOH 9:1;
Die Probe ist in DCM/LM gelöst

Säule: DAICELAS.M
Säuleninfo: Chiralpak AS (250 x 4.6)mm 10µ

Operator: Analytik Labor AKEN

Injektion Time: 16:02:24
Injektion Date: 10.02.2014

Instrument Conditions:	At Start	At Stop
Temperature in°C:	30.0	30.0
Pressure in bar:	21.3	21.3
Flow in ml/min:	0.7	0.7



ent-**4k**

#	Ret. Time (min)	Width	Height (mAU)	Area (mAU*s)	Area %
1	5.47	0.16	8.31	93.15	0.22
2	8.56	0.39	10.67	288.03	0.68
3	11.64	0.79	797.76	41164.37	96.61
4	14.89	0.74	17.14	1061.93	2.49
Total				42607.48	100.00

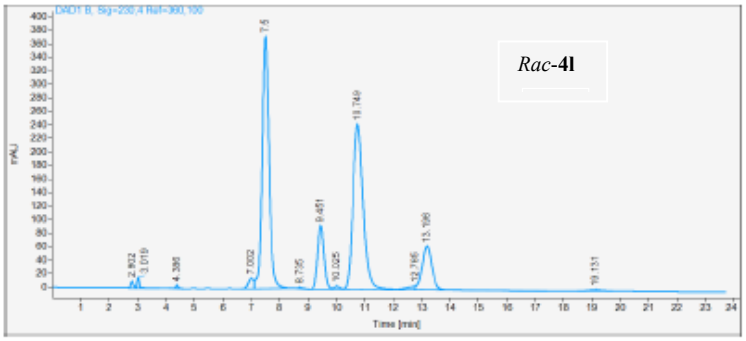
Sample name: PC 550 rac

Data file: C:\SNOOPY\PC\PC 550 RAC1IC.D
Description: Laufmittel: n-Heptan/IP 9:1; Die Probe ist in DCM/EtOH/LM gelöst.

Injection date: 11/4/2013 3:12:40 PM
Aeq. Analysis method: CHIRALPAK\IC1-6LNP.M

Column: Chiralpak IC, (150 x 4,6) mm, 5µ, SN: IC00CD-QF015

Pressure at start: 23 bar **Start flow:** 0.700 ml/min **Column oven:** 29.99 °C



Name PC 550 rac

RT [min]	Type	Area%	Area	Height	Width [min]
2.80	VV	0.39	63.53	9.02	0.11
3.02	VB	0.51	81.60	15.37	0.08
4.39	BB	0.16	26.37	4.95	0.08
7.00	BV	1.29	206.98	14.98	0.21
7.50	VB	38.85	6249.43	372.48	0.26
8.74	BB	0.15	23.81	1.51	0.25
9.45	BV	9.03	1452.94	93.23	0.24
10.03	VV	0.41	65.64	4.29	0.23
10.75	VV	38.10	6128.25	244.10	0.39
12.78	MF	0.81	129.78	5.91	0.37
13.20	FM	9.90	1592.11	64.71	0.41
19.13	BB	0.40	64.71	1.94	0.54
	Sum	100.00	16085.15		

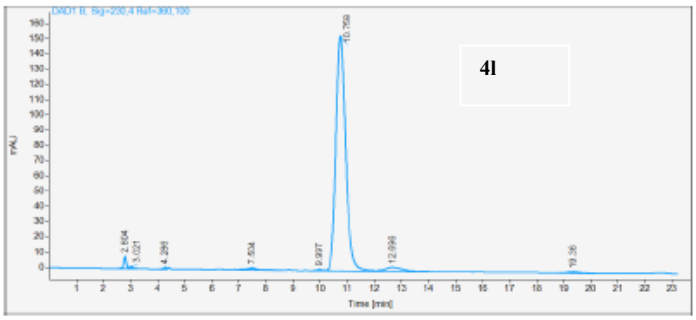
Sample name: PC 568

Data file: C:\SNOOPY\PC\PC 568IC.D
Description: Laufmittel: n-Heptan/IP 9:1; Die Probe ist in DCM/EtOH/LM gelöst.

Injection date: 11/4/2013 3:42:36 PM
Aeq. Analysis method: CHIRALPAK\IC1-6LNP.M

Column: Chiralpak IC, (150 x 4,6) mm, 5µ, SN: IC00CD-QF015

Pressure at start: 23 bar **Start flow:** 0.700 ml/min **Column oven:** 30 °C



Name PC 568

RT [min]	Type	Area%	Area	Height	Width [min]
2.80	BV	1.29	52.80	7.70	0.11
3.02	VV	0.23	9.29	1.62	0.09
4.29	BV	0.17	6.95	1.01	0.10
7.50	BB	0.72	29.23	1.19	0.34
10.00	BV	0.24	9.83	0.66	0.24
10.76	VV	93.70	3825.40	154.02	0.38
12.70	VB	2.92	119.24	2.58	0.70
19.36	BB	0.73	29.86	0.99	0.49
	Sum	100.00	4082.60		

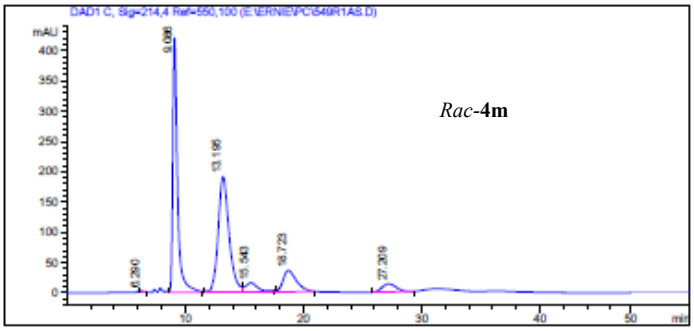
Sample Name: PC 549 rac
Data file: E:\ERNIE\PC\549R1AS.D
Sample Info: Laufmittel: n-Heptan/IP 7:3;
Die Probe ist in DCM/LM gelöst

Säule: DAICELAS.M
Säuleninfo: Chiralpak AS (250 x 4.6)mm 10µ

Operator: Analytik Labor AKEN

Injektion Time: 10:14:32
Injektion Date: 09.12.2013

Instrument Conditions:	At Start	At Stop
Temperature in°C:	30.0	30.0
Pressure in bar:	19.2	19.6
Flow in ml/min:	0.5	0.5



Rac-**4m**

#	Ret. Time (min)	Width	Height (mAU)	Area (mAU*s)	Area %
1	6.29	0.25	3.36	60.68	0.21
2	9.09	0.41	420.22	11622.67	40.13
3	13.19	0.96	190.86	12241.63	42.27
4	15.54	0.90	15.07	1143.25	3.95
5	18.72	1.01	35.32	2794.53	9.65
6	27.21	0.99	13.32	1099.26	3.80
Total				28962.02	100.00

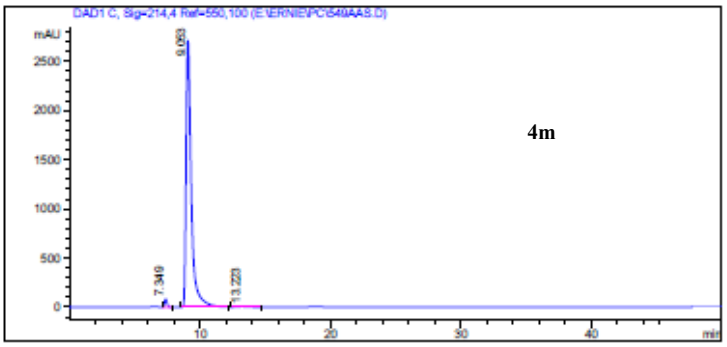
Sample Name: PC 549a
Data file: E:\ERNIE\PC\549AAS.D
Sample Info: Laufmittel: n-Heptan/IP 7:3;
Die Probe ist in DCM/LM gelöst

Säule: DAICELAS.M
Säuleninfo: Chiralpak AS (250 x 4.6)mm 10µ

Operator: Analytik Labor AKEN

Injektion Time: 11:12:16
Injektion Date: 09.12.2013

Instrument Conditions:	At Start	At Stop
Temperature in°C:	30.0	30.0
Pressure in bar:	19.4	19.4
Flow in ml/min:	0.5	0.5



4m

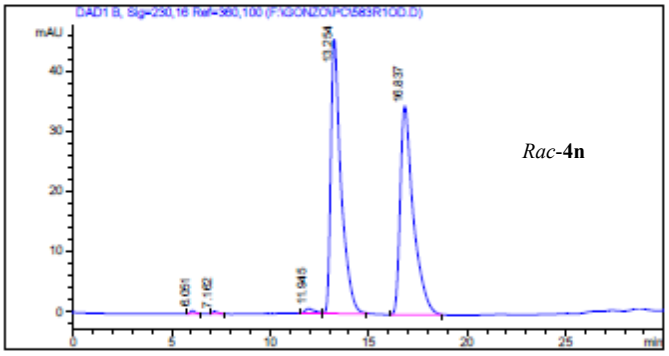
#	Ret. Time (min)	Width	Height (mAU)	Area (mAU*s)	Area %
1	7.35	0.20	81.83	1040.55	1.35
2	9.05	0.41	2711.45	75591.83	98.07
3	13.22	0.76	7.15	450.49	0.58
Total				77082.87	100.00

Sample Name: PC 583 rac
Data file: F:\GONZO\PC\583R1OD.D
Sample Info: Laufmittel: n-Heptan/EtOH 9:1;
Die Probe ist in LM/EtOH/DCM gelöst.

Säule: DAICELOD.M
Säuleninfo: Chiralcel OD (250x4,6)mm
Operator: Analytik Labor AKEN

Injektion Time: 08:43:35
Injektion Date: 14.10.2013

Instrument Conditions:	At Start	At Stop
Temperature in°C:	30.0°C	30.0°C
Pressure in bar:	12.9	13.0
Flow in ml/min:	0.50	0.50



Rac-**4n**

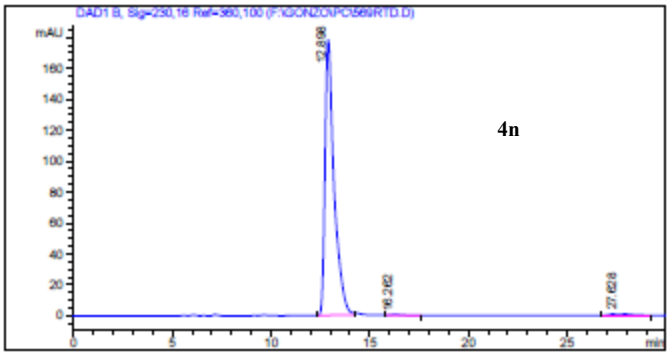
#	Ret. Time (min)	Width	Height (mAU)	Area (mAU*s)	Area %
1	6.05	0.24	0.52	8.04	0.24
2	7.16	0.25	0.40	6.94	0.21
3	11.95	0.49	0.79	26.80	0.81
4	13.25	0.52	45.58	1646.46	49.94
5	16.84	0.68	34.69	1608.78	48.79
Total				3297.02	100.00

Sample Name: PC 569 r
Data file: F:\GONZO\PC\569RTD.D
Sample Info: Laufmittel: n-Heptan/EtOH 9:1;
Die Probe ist in LM gelöst

Säule: DAICELOD.M
Säuleninfo: Chiralcel OD (250x4,6)mm
Operator: Analytik Labor AKEN

Injektion Time: 08:18:30
Injektion Date: 17.12.2013

Instrument Conditions:	At Start	At Stop
Temperature in°C:	30.0°C	30.0°C
Pressure in bar:	13.3	13.7
Flow in ml/min:	0.50	0.50



4n

#	Ret. Time (min)	Width	Height (mAU)	Area (mAU*s)	Area %
1	12.90	0.46	178.26	5698.97	98.02
2	16.26	0.41	0.57	19.03	0.33
3	27.63	0.81	1.40	96.33	1.66
Total				5814.34	100.00

AK Enders - Analytische HPLC

Sample Name: PC 758 + 759
Data file: D:\ERNIE\PC\758759AS.D
Sample Info: Laufmittel: n-Heptan/IP 8:2;
Die Probe ist in LM gelöst

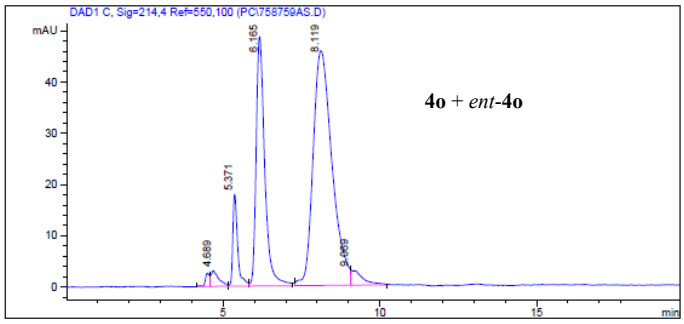
Agilent Technologies

Säule: DAICELAS.M
Säuleninfo: Chiralpak AS (250 x 4.6)mm 10µ

Operator: Analytik Labor AKEN

Injektion Time: 14:12:37
Injektion Date: 12.05.2014

Instrument Conditions:	At Start	At Stop
Temperature in°C:	30.0	30.0
Pressure in bar:	25.2	23.8
Flow in ml/min:	0.7	0.7



#	Ret. Time (min)	Width	Height (mAU)	Area (mAU*s)	Area %
1	4.50	0.14	2.54	23.58	0.74
2	4.69	0.23	3.09	53.89	1.69
3	5.37	0.16	17.96	193.26	6.05
4	6.16	0.28	48.67	921.92	28.87
5	8.12	0.70	45.82	1923.12	60.23
6	9.07	0.42	3.05	77.03	2.41
Total				3192.81	100.00

AK Enders - Analytische HPLC

Sample Name: PC 758
Data file: D:\ERNIE\PC\758AS.D
Sample Info: Laufmittel: n-Heptan/IP 8:2;
Die Probe ist in LM gelöst

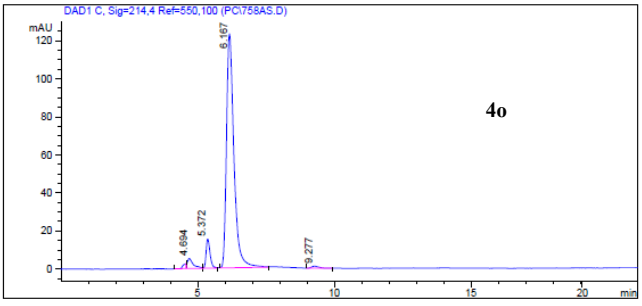
Agilent Technologies

Säule: DAICELAS.M
Säuleninfo: Chiralpak AS (250 x 4.6)mm 10µ

Operator: Analytik Labor AKEN

Injektion Time: 13:20:13
Injektion Date: 12.05.2014

Instrument Conditions:	At Start	At Stop
Temperature in°C:	30.0	30.0
Pressure in bar:	24.6	24.2
Flow in ml/min:	0.7	0.7



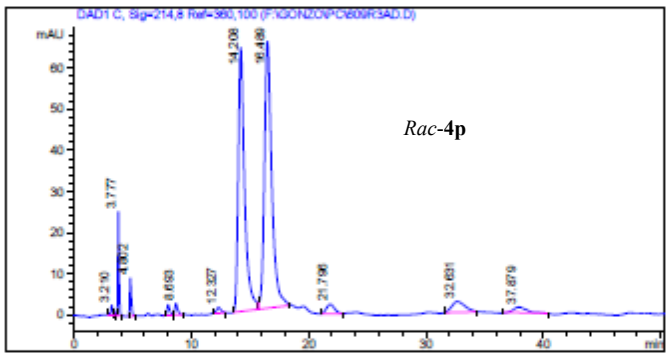
#	Ret. Time (min)	Width	Height (mAU)	Area (mAU*s)	Area %
1	4.58	0.14	2.50	21.43	0.83
2	4.69	0.25	5.30	78.65	3.04
3	5.37	0.16	15.33	153.41	5.92
4	6.17	0.29	123.18	2317.03	89.42
5	9.28	0.29	1.09	20.71	0.80
Total				2591.24	100.00

Sample Name: PC 609 rac
Data file: F:\GONZO\PC\609R3AD.D
Sample Info: Laufmittel: n-Heptan/IP 95:5;
Die Probe ist in LM/DCM gelöst

Säule: DAICELAD.M
Säuleninfo: Chiralpak AD (250x4,6)mm
Operator: Analytik Labor AKEN

Injektion Time: 12:06:11
Injektion Date: 09.12.2013

Instrument Conditions:	At Start	At Stop
Temperature in°C:	30.0°C	30.0°C
Pressure in bar:	29.6	30.3
Flow in ml/min:	1.00	1.00



Rac-**4p**

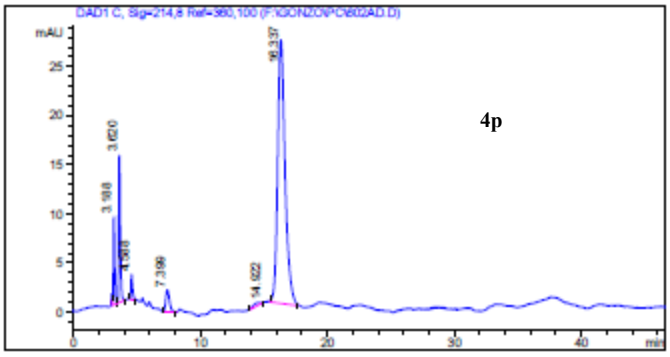
#	Ret. Time (min)	Width	Height (mAU)	Area (mAU*s)	Area %
1	3.21	0.15	2.48	26.87	0.44
2	3.44	0.11	1.00	7.71	0.13
3	3.78	0.11	25.29	182.59	2.99
4	4.80	0.14	9.29	86.38	1.41
5	8.02	0.24	2.62	42.28	0.69
6	8.69	0.26	2.80	49.26	0.81
7	12.33	0.38	1.39	39.34	0.64
8	14.21	0.55	64.15	2420.32	39.62
9	16.49	0.65	64.89	2829.28	46.31
10	21.80	0.61	2.34	113.57	1.86
11	32.63	0.93	2.68	210.05	3.44
12	37.88	1.26	1.35	101.63	1.66
Total				6109.29	100.00

Sample Name: PC 602
Data file: F:\GONZO\PC\602AD.D
Sample Info: Laufmittel: n-Heptan/IP 95:5;
Die Probe ist in LM/DCM gelöst

Säule: DAICELAD.M
Säuleninfo: Chiralpak AD (250x4,6)mm
Operator: Analytik Labor AKEN

Injektion Time: 12:57:45
Injektion Date: 09.12.2013

Instrument Conditions:	At Start	At Stop
Temperature in°C:	30.0°C	30.0°C
Pressure in bar:	30.1	30.4
Flow in ml/min:	1.00	1.00



4p

#	Ret. Time (min)	Width	Height (mAU)	Area (mAU*s)	Area %
1	3.19	0.11	8.97	64.28	4.69
2	3.62	0.14	15.04	136.13	9.93
3	4.59	0.13	2.57	23.17	1.69
4	7.40	0.31	2.24	46.35	3.38
5	14.92	3.58	0.06	12.35	0.90
6	16.34	0.60	26.88	1089.02	79.42
Total				1371.30	100.00

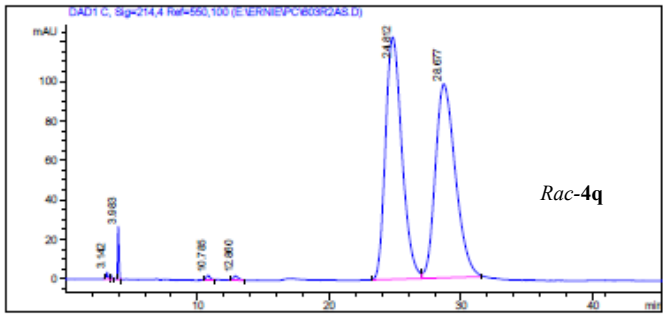
Sample Name: PC 603 rac
Data file: E:\ERNIE\PC\603R2AS.D
Sample Info: Laufmittel: n-Heptan/EtOH 97:3;
Die Probe ist in DCM/LM gelöst

Säule: DAICELAS.M
Säuleninfo: Chiralpak AS (250 x 4.6)mm 10µ

Operator: Analytik Labor AKEN

Injektion Time: 15:51:08
Injektion Date: 09.12.2013

Instrument Conditions:	At Start	At Stop
Temperature in°C:	30.0	30.0
Pressure in bar:	29.1	28.4
Flow in ml/min:	1.0	1.0



#	Ret. Time (min)	Width	Height (mAU)	Area (mAU*s)	Area %
1	3.14	0.14	3.51	36.40	0.17
2	3.41	0.10	2.36	17.56	0.08
3	3.98	0.11	26.68	183.67	0.86
4	10.78	0.27	2.29	46.83	0.22
5	12.86	0.33	2.15	58.52	0.27
6	24.81	1.12	122.66	10620.38	49.71
7	28.68	1.33	98.17	10402.10	48.69
Total				21365.45	100.00

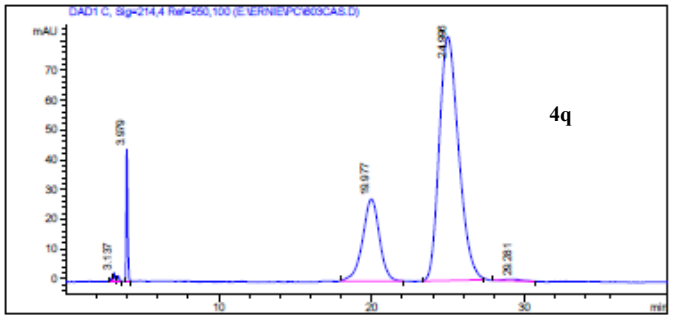
Sample Name: PC 603 c
Data file: E:\ERNIE\PC\603CAS.D
Sample Info: Laufmittel: n-Heptan/EtOH 97:3;
Die Probe ist in DCM/LM gelöst

Säule: DAICELAS.M
Säuleninfo: Chiralpak AS (250 x 4.6)mm 10µ

Operator: Analytik Labor AKEN

Injektion Time: 16:43:08
Injektion Date: 09.12.2013

Instrument Conditions:	At Start	At Stop
Temperature in°C:	30.0	30.0
Pressure in bar:	28.8	28.6
Flow in ml/min:	1.0	1.0



#	Ret. Time (min)	Width	Height (mAU)	Area (mAU*s)	Area %
1	3.00	0.08	1.31	7.64	0.08
2	3.14	0.10	2.90	20.39	0.21
3	3.42	0.13	1.93	17.22	0.18
4	3.98	0.11	44.40	308.43	3.17
5	19.98	0.92	27.59	2115.04	21.73
6	25.00	1.18	81.90	7219.44	74.18
7	29.28	1.35	0.55	44.61	0.46
Total				9732.77	100.00

Sample name: PC 582 O

Data file: C:\SNOOPY\PC\582O\C.D

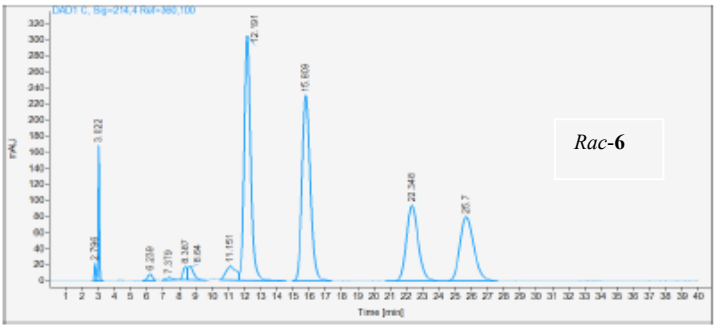
Description: Laufmittel: n-Heptan/IP 9:1;
Die Probe ist im LM/DCM gelöst.

Injection date: 12/19/2013 1:26:53 PM

Aeq. Analysis method: CHIRALPAK\C1-6LNP.M

Column: Chiralpak IC, (150 x 4,6) mm, 5µ, SN: IC00CD-QF015

Pressure at start: 24 bar **Start flow:** 0.700 ml/min **Column oven:** 30.02 °C



Rac-**6**

Name PC 582 O

RT [min]	Type	Area%	Area	Height	Width [min]
2.80	VV	0.47	133.89	20.67	0.10
3.02	VB	2.87	820.76	168.63	0.08
6.24	BV	0.61	173.52	8.18	0.29
7.38	BB	0.37	104.58	3.15	0.50
8.39	BV	0.93	266.84	15.72	0.26
8.64	VB	1.50	428.45	16.41	0.38
11.15	BV	2.61	745.63	16.89	0.65
12.19	VB	28.94	8270.34	304.32	0.42
15.81	BB	28.39	8114.51	230.44	0.54
22.35	BB	16.65	4758.00	93.88	0.79
26.70	BB	16.67	4763.91	79.60	0.92
	Sum	100.00	28580.43		

Sample name: PC 582

Data file: C:\SNOOPY\PC\582X\C.D

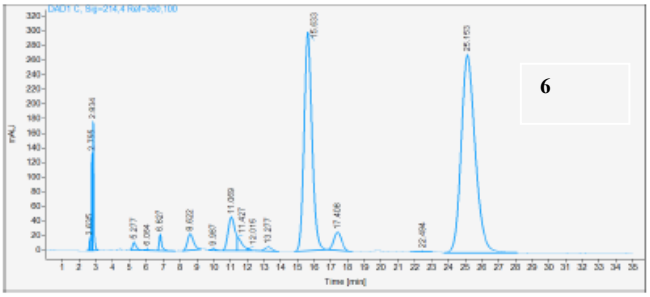
Description: Laufmittel: n-Heptan/IP 9:1;
Die Probe ist im LM/DCM gelöst.

Injection date: 12/13/2013 9:10:29 AM

Aeq. Analysis method: CHIRALPAK\C1-6LNP.M

Column: Chiralpak IC, (150 x 4,6) mm, 5µ, SN: IC00CD-QF015

Pressure at start: 24 bar **Start flow:** 0.700 ml/min **Column oven:** 29.98 °C



6

RT [min]	Type	Area%	Area	Height	Width [min]
2.64	BV	0.20	62.86	15.55	0.06
2.75	VV	1.68	522.50	131.73	0.06
2.83	VB	3.10	963.22	174.85	0.08
5.28	VB	0.47	146.53	10.01	0.21
6.06	BB	0.11	32.79	1.61	0.27
6.83	BB	0.74	230.07	21.39	0.16
8.62	BB	1.67	519.62	21.33	0.38
9.99	BB	0.12	38.39	1.86	0.32
11.07	MF	4.17	1292.53	45.23	0.48
11.43	MF	1.13	350.60	19.09	0.23
12.02	FM	0.11	33.37	3.42	0.16
13.28	BB	0.54	166.49	5.28	0.48
15.63	BB	32.32	10028.27	298.68	0.52
17.41	BB	2.55	790.30	24.29	0.51
22.49	MM	0.21	65.46	1.26	0.87
25.15	BB	50.87	15781.22	270.62	0.90
	Sum	100.00	31024.23		