Organocatalytic Asymmetric Michael Addition of α-Aryl Cyclopentanones to Nitroolefins for Construction of Adjacent Quaternary and Tertiary Stereocenters

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Supporting Information

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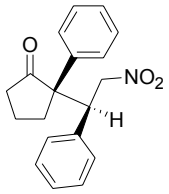
1. General Remarks

^{1}H NMR spectra were recorded on a VARIAN Mercury 300 MHz spectrometer in chloroform-d. Chemical shifts are reported in ppm with the internal TMS signal at 0.0 ppm as a standard. The data are reported as (s = singlet, d = doublet, t = triplet, q = quartet, m = multiplet or unresolved, brs = broad singlet, coupling constant(s) in Hz, integration). ^{13}C NMR spectra were recorded on a VARIAN Mercury 75 MHz spectrometer in chloroform-d. Chemical shifts are reported in ppm with the internal chloroform signal at 77.0 ppm as a standard. Commercially obtained reagents were used without further purification. All reactions were monitored by TLC with silica gel-coated plates. Diastereomeric ratios were determined from crude ^{1}H NMR or HPLC analysis. Enantiomeric ratios were determined by HPLC, using a chiralpak AS-H column, a chiralpak AD-H column with hexane and *i*-PrOH as solvents.

Catalyst **I-VII** were prepared according to literature report.[1] α-Aryl Cyclopentanones were prepared according to the literature procedure.[2] The racemic adducts were attained by using DABCO (1.2 eq.) as the base. The absolute (2*S*,3*S*)-configuration of **3ad** and (2*S*,3*S*,4*S*)-**7** were determined unequivocally according to the X-ray diffraction analysis, and those of other adducts were deduced on the basis of these results.

2. General procedure for asymmetric Michael Addition Reaction of α-substituted cyclopentanones to Nitroolefins catalyzed by organocatalysts (I-d)

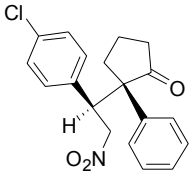
The catalyst **I-d** (13.5 mg, 0.02 mmol) was added to a vial containing α-substituted cyclopentanone (0.2 mmol) and nitroolefins (0.21 mmol) in CH_2Cl_2 (0.45 mL) at the indicated temperature, TLC analysis indicated completion of the reaction after about 12-20h. Then the reaction mixture was concentrated *in vacuo* to obtain the crude product. The crude product was purified by flash silica gel chromatography to afford the product.



(3aa)

(2*S*, 3*S*)-2-(2-Nitro-1-phenyl-ethyl)-2-phenyl-cyclopentanone (table 2, entry 1):

The title compound was prepared according to the general procedure as described above in 90% yield. $[\alpha]^{25}_{D}$ +65.8 (*c* 1.24, $CHCl_3$); m.p 106.5 ^{o}C; ^{1}H NMR ($CDCl_3$, TMS, 300 MHz) δ 1.55-1.68 (m, 1H), 1.75-1.84 (m, 1H), 1.96-2.14 (m, 2H), 2.19-2.36 (m, 2H), 4.19-4.23 (dd, *J* = 3.0 and 12.3 Hz, 1H), 4.48-4.56 (t, *J* = 12.9 Hz, 1H), 4.86-4.92 (dd, *J* = 4.3 and 12.9 Hz, 1H), 7.09 (brs, 2H), 7.17-7.26 (m, 3H), 7.31-7.39 (m, 5H); ^{13}C NMR ($CDCl_3$, TMS, 75 MHz) δ 18.4, 31.5, 37.6, 49.5, 58.9, 76.5, 128.1, 128.6, 129.4, 129.9, 134.6, 135.9, 217.1; IR (KBr) ν: 3019, 2972, 2894, 2400, 1732, 1555, 1515, 1437, 1378, 1216, 1046, 928, 757 cm^{-1}; HRMS Calcd. for $C_{19}H_{19}NO_3$: 309.1365, found: 309.1363; dr > 99:1, determined by the crude ^{1}HNMR; 92% ee, determined by chiral HPLC analysis (Chiralcel AD-H, *i*-propanol/hexane = 1/99, flow rate 0.25 mL/min, λ = 254 nm): t_{minor} = 78.7 min, t_{major} = 86.3 min. Enantioenriched compounds (>99% ee) can be easily obtained by direct crystallization of the crude products from methanol.

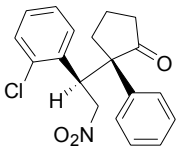


(3ab)

(2*S*, 3*S*)-2-[1-(4-chloro-phenyl)-2-Nitro-ethyl]-2-phenyl-cyclopentanone (table 2, entry 2):

The title compound was prepared according to the general procedure as described above in 88% yield. $[\alpha]^{25}_{D}$ +76.5 (*c* 1.02, $CHCl_3$); m.p 86 ^{o}C; ^{1}H NMR ($CDCl_3$, TMS, 300 MHz) δ 1.58-1.64 (m, 1H), 1.78-1.88 (m, 1H), 1.99-2.06 (m, 2H), 2.23-2.32 (m, 2H), 4.12-4.17 (dd, *J* = 3.9 and 12.6 Hz, 1H), 4.40-4.48 (t, *J* = 12.6 Hz, 1H), 4.88-4.94 (dd, *J* = 3.9 and 12.9 Hz, 1H), 6.97-7.00 (m, 2H), 7.24-7.26 (m, 3H), 7.32-7.42 (m, 4H); ^{13}C NMR ($CDCl_3$, TMS, 75 MHz) δ 17.9, 31.3, 37.1, 48.6, 58.4,

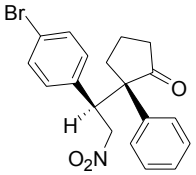
75.9, 127.6, 128.3, 128.4, 129.1, 130.8, 133.7, 134.0, 216.5; IR (KBr) ν: 3015, 2972, 2887, 2396, 1739, 1591, 1556, 1515, 1420, 1395, 1210, 1042, 933, 754 cm^{-1}; HRMS Calcd. for $C_{19}H_{18}ClNO_3$: 343.0975 , found: 343.0973; dr > 99:1, determined by the crude ^{1}HNMR; 94% ee, determined by chiral HPLC analysis (Chiralcel AS-H, *i*-propanol/hexane = 10/90, flow rate 0.5 mL/min, λ = 210 nm): t_{minor} = 38.7 min, t_{major} = 30.5 min.



(3ac)

(2*S*, 3*S*)-2-[1-(2-chloro-phenyl)-2-Nitro-ethyl]-2-phenyl-cyclopentanone (table 2, entry 3):

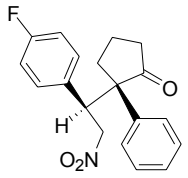
The title compound was prepared according to the general procedure as described above in 92% yield. $[\alpha]^{25}_D$ +108.8 (*c* 1.28, $CHCl_3$); m.p 76 ^oC; ^{1}H NMR ($CDCl_3$, TMS, 300 MHz) δ 1.52-1.60 (m, 1H), 1.82-1.92 (m, 1H), 2.23-2.36 (m, 4H), 4.28-4.32 (t, *J* = 12.6 Hz, 1H), 4.66-4.70 (m, 1H), 5.66-5.69 (m, 1H), 6.42-6.44 (m, 1H), 7.03-7.08 (m, 1H), 7.18 (m, 3H), 7.39 (m, 4H); ^{13}C NMR ($CDCl_3$, TMS, 75 MHz) δ 17.9, 33.6, 37.5, 42.8, 58.7, 76.9, 126.6, 128.7, 128.8, 129.1, 129.4, 130.4, 133.3, 134.4, 135.7, 218.0; IR (KBr) ν: 3019, 2976, 2894, 2396, 1727, 1555, 1524, 1478, 1377, 1213, 1050, 933, 754 cm^{-1}; HRMS Calcd. for $C_{19}H_{18}ClNO_3$: 343.0975, found: 343.0974; dr > 99:1, determined by the crude ^{1}HNMR; 94% ee, determined by chiral HPLC analysis (Chiralcel AS-H, *i*-propanol/hexane = 10/90, flow rate 0.5 mL/min, λ = 210 nm): t_{minor} = 19.1 min, t_{major} = 22.8 min.



(3ad)

(2*S*, 3*S*)-2-[1-(4-Bromo-phenyl)-2-Nitro-ethyl]-2-phenyl-cyclopentanone (table 2, entry 4):

The title compound was prepared according to the general procedure as described above in 94% yield. $[\alpha]^{25}_{D}$ +95.7 (*c* 1.02, $CHCl_3$); m.p 108 ^{o}C; ^{1}H NMR ($CDCl_3$, TMS, 300 MHz) δ 1.57-1.63 (m, 1H), 1.81-1.83 (m, 1H), 2.00-2.09 (m, 2H), 2.22-2.35 (m, 2H), 4.10-4.15 (dd, *J* = 3.9 and 11.7 Hz, 1H), 4.38-4.47 (t, *J* = 12.6 Hz, 1H), 4.87-4.93 (dd, *J* = 3.9 and 13.8 Hz, 1H), 6.90-6.93 (d, *J* = 8.7 Hz, 2H), 7.25 (s, 2H), 7.31-7.41 (m, 5H); ^{13}C NMR ($CDCl_3$, TMS, 75 MHz) δ 17.9, 31.3, 37.1, 48.7, 58.3, 75.9, 122.0, 127.7, 128.3, 129.1, 131.2, 131.4, 133.7, 134.5, 216.5; IR (KBr) ν: 3015, 2976, 2439, 1739, 1556, 1493, 1373, 1213, 929, 762 cm^{-1}; HRMS Calcd. for $C_{19}H_{18}BrNO_3$: 387.0470, found: 387.0471; dr > 99:1, determined by the crude ^{1}HNMR; 94% ee, determined by chiral HPLC analysis (Chiralcel AS-H, *i*-propanol/hexane = 10/90, flow rate 0.5 mL/min, λ = 210 nm): t_{minor} = 41.5 min, t_{major} = 32.3 min. Enantioenriched compounds (>99% ee) can be easily obtained by direct crystallization of the crude products from methanol.

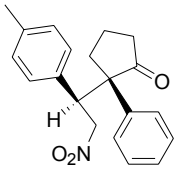


(3ae)

(2*S*, 3*S*)-2-[1-(4-Fluoro-phenyl)-2-Nitro-ethyl]-2-phenyl-cyclopentanone (table 2, entry 5):

The title compound was prepared according to the general procedure as described above in 83% yield. $[\alpha]^{25}_{D}$ +62.2 (*c* 0.85, $CHCl_3$); m.p 86 ^{o}C; ^{1}H NMR ($CDCl_3$, TMS, 300 MHz) δ 1.58-1.64 (m, 1H), 1.83-1.90 (m, 1H), 2.01-2.13 (m, 2H), 2.24-2.38 (m, 2H), 4.16-4.21 (dd, *J* = 3.0 and 11.7 Hz, 1H), 4.42-4.51 (t, *J* = 12.9 Hz, 1H), 4.89-4.94 (dd, *J* = 2.7 and 12.6 Hz, 1H), 6.96-7.07 (m, 4H), 7.28-7.43 (m, 5H); ^{13}C NMR ($CDCl_3$, TMS, 75 MHz) δ 18.3, 31.6, 37.5, 48.8, 58.5, 76.5, 115.4, 115.6, 128.0, 128.6, 129.4, 131.4, 131.5, 134.2, 216.9; IR (KBr) ν: 3016, 2973, 2883, 2400, 1736, 1599, 1556, 1509, 1478, 1420, 1210, 1046, 929, 761 cm^{-1}; HRMS Calcd. for $C_{19}H_{18}FNO_3$: 327.1259, found: 327.1265; dr > 99:1, determined by the crude ^{1}HNMR; 95% ee, determined by chiral HPLC analysis (Chiralcel AS-H,

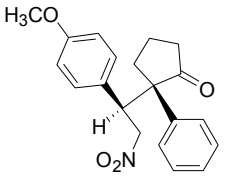
i-propanol/hexane = 10/90, flow rate 0.5 mL/min, λ = 210 nm): t_{minor} = 37.4 min, t_{major} = 30.5 min.



(3af)

(2*S*, 3*S*)-2-(2-Nitro-1-*p*-tolyl-ethyl)-2-phenyl-cyclopentanone (table 2, entry 6):

The title compound was prepared according to the general procedure as described above in 90% yield. $[\alpha]^{25}_{D}$ +65.0 (*c* 1.15, $CHCl_3$); m.p 82 °C; ^{1}H NMR ($CDCl_3$, TMS, 300 MHz) δ 1.50-1.68 (m, 1H), 1.75-1.86 (m, 1H), 1,98-2.15 (m, 2H), 2.21-2.28 (m, 2H), 2.30 (s, 3H), 4.12-4.18 (dd, *J* = 3.9 and 12.6 Hz, 1H), 4.43-4.51 (t, *J* = 12.9 Hz, 1H), 4.81-4.87 (dd, *J* = 4.2 and 13.2 Hz, 1H), 6.92-6.95 (d, *J* = 8.1 Hz, 2H), 7.06-7.08 (d, *J* = 7.5 Hz, 2H), 7.38 (brs, 5H); ^{13}C NMR ($CDCl_3$, TMS, 75 MHz) δ 18.4, 21.3, 31.6, 37.6, 49.1, 58.9, 76.5, 128.1, 128.4, 129.3, 129.7, 132.6, 134.7, 137.8, 217.2; IR (KBr) ν: 3013, 2969, 2899, 2393, 1741, 1558, 1509, 1439, 1383, 1211, 1038, 931, 758 cm^{-1}; dr > 99:1, determined by the crude ^{1}HNMR; 95% ee, determined by chiral HPLC analysis (Chiralcel AS-H, *i*-propanol/hexane = 10/90, flow rate 0.5 mL/min, λ = 210 nm): t_{minor} = 44.7 min, t_{major} = 25.0 min.

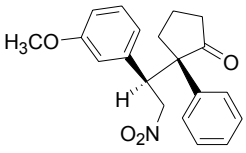


(3ag)

(2*S*, 3*S*)-2-[1-(4-Methoxy-phenyl)-2-Nitro-ethyl]-2-phenyl-cyclopentanone (table 2, entry 7):

The title compound was prepared according to the general procedure as described above in 87% yield. $[\alpha]^{25}_{D}$ +76.5 (*c* 1.15, $CHCl_3$); m.p 140 °C; ^{1}H NMR ($CDCl_3$, TMS, 300 MHz) δ 1.55-1.68 (m, 1H), 1.82-1.86 (m, 1H), 2.01-2.18 (m, 2H), 2.25-2.41 (m, 2H), 3.82 (s, 3H), 4.15-4.21 (dd, *J* = 3.9 and 12.0 Hz, 1H), 4.44-4.53 (t, *J* = 12.6 Hz, 1H), 4.84-4.90 (dd, *J* = 4.2 and 13.2 Hz, 1H), 6.82-6.85 (d, *J* = 8.7 Hz,

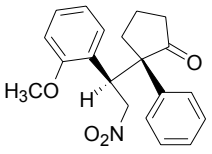
2H), 7.00-7.03 (d, *J* = 9.0 Hz, 2H), 7.42 (brs, 5H); ^{13}C NMR ($CDCl_3$, TMS, 75 MHz) δ 18.6, 31.7, 37.8, 49.0, 55.6, 59.2, 76.8, 114.1, 127.8, 128.2, 128.6, 129.5, 131.1, 134.9, 159.5, 217.4; IR (KBr) ν: 3015, 2976, 2400, 1739, 1552, 1513, 1423, 1375, 1252, 1220, 1036, 929, 754 cm^{-1}; HRMS Calcd. for $C_{20}H_{21}NO_4$: 339.1471, found: 339.1468; dr > 99:1, determined by the crude ^{1}HNMR; 95% ee, determined by chiral HPLC analysis (Chiralcel AD-H, *i*-propanol/hexane = 10/90, flow rate 0.5 mL/min, λ = 210 nm): t_{minor} = 21.3 min, t_{major} = 25.8 min.



(3ah)

(2*S*, 3*S*)-2-[1-(3-Methoxy-phenyl)-2-Nitro-ethyl]-2-phenyl-cyclopentanone (table 2, entry 8):

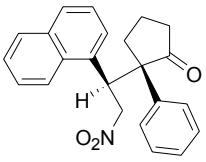
The title compound was prepared according to the general procedure as described above in 93% yield. $[\alpha]^{25}_D$ +42.5 (*c* 0.85, $CHCl_3$); m.p 58 °C; ^{1}H NMR ($CDCl_3$, TMS, 300 MHz) δ 1.50-1.60 (m, 1H), 1.72-1.75 (m, 1H), 1.93-2.08 (m, 2H), 2.14-2.32 (m, 2H), 3.67 (s, 3H), 4.05-4.11 (dd, *J* = 3.6 and 12.3 Hz, 1H), 4.35-4.44 (t, *J* = 12.9 Hz, 1H), 4.78-4.84 (dd, *J* = 4.5 and 13.5 Hz, 1H), 6.50 (brs, 1H),6.56-6.59 (d, *J* = 7.8 Hz, 1H), 6.70-6.74 (dd, *J* = 2.1 and 8.1 Hz, 1H), 7.08-7.14 (t, *J* = 7.8 Hz, 1H), 7.31 (m, 5H); ^{13}C NMR ($CDCl_3$, TMS, 75 MHz) δ 18.4, 31.8, 37.6, 49.3, 55.4, 58.7, 76.4, 113.5, 115.7, 122.1, 128.1, 128.5, 129.3, 129.5, 134.5, 137.3, 159.5, 217.1; IR (KBr) ν: 3015, 2970, 2934, 2887, 2400, 1735, 1603, 1554, 1517, 1498, 1424, 1389, 1220, 1038, 925, 767 cm^{-1}; HRMS Calcd. for $C_{20}H_{21}NO_4$: 339.1471, found: 339.1472; dr > 99:1, determined by the crude ^{1}HNMR; 94% ee, determined by chiral HPLC analysis (Chiralcel AD-H, *i*-propanol/hexane = 10/90, flow rate 0.4 mL/min, λ = 210 nm): t_{minor} = 24.7 min, t_{major} = 27.9 min.



(3ai)

(2*S*, 3*S*)-2-[1-(2-Methoxy-phenyl)-2-Nitro-ethyl]-2-phenyl-cyclopentanone (table 2, entry 9):

The title compound was prepared according to the general procedure as described above in 85% yield. $[\alpha]^{25}_{D}$ +135.8 (*c* 0.59, $CHCl_3$); m.p 85 ^{o}C; ^{1}H NMR ($CDCl_3$, TMS, 300 MHz) δ 1.46-1.53 (m, 1H), 1.62-1.90 (m, 1H), 2.03-2.13 (m, 2H), 2.15-2.30 (m, 2H), 3.75 (s, 3H), 4.36-4.44 (t, *J* = 12.9 Hz, 1H), 4.50-4.55 (dd, *J* = 3.3 and 11.7 Hz, 1H), 5.13-5.17 (m, 1H), 6.49-6.52 (d, *J* = 7.2 Hz, 1H), 6.68-6.73 (t, *J* = 7.8 Hz, 1H), 6.78-6.81 (d, *J* = 8.1 Hz, 1H), 7.11-7.19 (m, 3H), 7.27 (brs, 3H); ^{13}C NMR ($CDCl_3$, TMS, 75 MHz) δ 17.8, 29.6, 32.7, 37.4, 41.1, 55.6, 58.6, 76.3, 111.1, 120.1, 124.4, 127.9, 128.2, 128.6, 128.8, 130.1, 134.3, 157.9, 217.9; IR (KBr) ν: 3014, 2985, 2404, 1727, 1552, 1521, 1490, 1420, 1377, 1212, 1054, 1027, 929, 758 cm^{-1}; dr > 99:1, determined by the crude ^{1}HNMR; 94% ee, determined by chiral HPLC analysis (Chiralcel AS-H, *i*-propanol/hexane = 10/90, flow rate 0.5 mL/min, λ = 210 nm): t_{minor} = 19.6 min, t_{major} = 22.3 min.

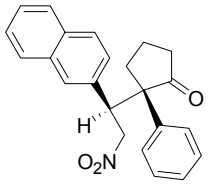


(3aj)

(2*S*, 3*S*)-2-(1-Naphthalen-1-yl-2-Nitro-ethyl)-2-phenyl-cyclopentanone (table 2, entry 10):

The title compound was prepared according to the general procedure as described above in 89% yield. $[\alpha]^{25}_{D}$ +42.2 (*c* 1.49, $CHCl_3$); m.p 40 ^{o}C; ^{1}H NMR ($CDCl_3$, TMS, 300 MHz) δ 1.40-1.53 (m, 1H), 1.62-1.80 (m, 1H), 1.90-2.05 (m, 2H), 2.10-2.35 (m, 2H), 4.42-4.51 (t, *J* = 12.3 Hz, 1H), 5.00-5.05 (dd, *J* = 3.3 and 12.0 Hz, 1H), 5.59-5.65 (dd, *J* = 3.9 and 12.9 Hz,, 1H), 6.65-6.67 (d, *J* = 7.2 Hz, 1H), 7.21-7.30 (m, 3H), 7.40 (brs, 3H), 7.49-7.52 (t, *J* = 7.8 Hz, 1H), 7.57-7.60 (t, *J* = 7.8 Hz, 1H), 7.74-7.77 (d, *J* = 8.1 Hz, 1H), 7.83-7.85 (d, *J* = 8.1 Hz, 1H), 8.34-8.37 (d, *J* = 8.4 Hz, 1H); ^{13}C NMR ($CDCl_3$, TMS, 75 MHz) δ 17.6, 29.6, 33.7, 36.9, 40.7, 53.4, 58.3, 77.4, 123.3, 124.3, 125.6, 125.8, 126.4, 128.3, 128.4, 128.8, 132.5, 132.7, 133.1, 133.9, 217.9; IR (KBr) ν: 3011, 2976, 2397, 1727, 1556, 1513, 1424, 1213, 1040,

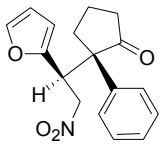
925, 754 cm^{-1}; dr > 99:1, determined by the crude ^{1}HNMR; 96% ee, determined by chiral HPLC analysis (Chiralcel AS-H, *i*-propanol/hexane = 10/90, flow rate 0.5 mL/min, λ = 210 nm): t_{minor} = 32.8 min, t_{major} = 41.9 min.



(3ak)

(2*S*, 3*S*)-2-(1-Naphthalen-2-yl-2-Nitro-ethyl)-2-phenyl-cyclopentanone (table 2, entry 11):

The title compound was prepared according to the general procedure as described above in 95% yield. $[\alpha]^{25}_D$ +78.6 (*c* 1.08, $CHCl_3$); m.p 105 ^oC; ^{1}H NMR ($CDCl_3$, TMS, 300 MHz) δ 1.50-1.59 (m, 1H), 1.72-1.84 (m, 1H), 2.01-2.16 (m, 2H), 2.18-2.38 (m, 2H), 4.32-4.37 (dd, *J* = 3.6 and 12.0 Hz, 1H), 4.58-4.67 (t, *J* = 12.9 Hz, 1H), 4.96-5.02 (dd, *J* = 3.9 and 12.9 Hz, 1H), 7.17-7.20 (d, *J* = 8.1 Hz, 1H), 7.40 (brs, 5H), 7.48 (brs, 3H), 7.74-7.80 (m, 3H); ^{13}C NMR ($CDCl_3$, TMS, 75 MHz) δ 18.0, 31.5, 37.2, 49.2, 58.7, 76.2, 126.2, 127.4, 127.5, 127.8, 127.9, 128.2, 128.7, 129.0, 132.7, 132.8, 133.1, 134.2, 216.8; IR (KBr) ν: 3015, 2980, 2891, 2435, 2400, 1727, 1603, 1552, 1521, 1485, 1424, 1381, 1210, 1046, 925, 762 cm^{-1}; HRMS Calcd. for $C_{23}H_{21}NO_3$: 359.1521, found: 359.1524; dr > 99:1, determined by the crude ^{1}HNMR; 94% ee, determined by chiral HPLC analysis (Chiralcel AS-H, *i*-propanol/hexane = 10/90, flow rate 1 mL/min, λ = 210 nm): t_{minor} = 38.7 min, t_{major} = 22.1 min.

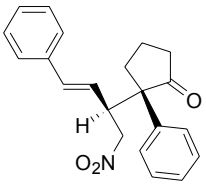


(3al)

(2*S*, 3*S*)-2-(1-Furan-2-yl-2-Nitro-ethyl)-2-phenyl-cyclopentanone (table 2, entry 12):

The title compound was prepared according to the general procedure as described above in 93% yield. $[\alpha]^{25}_D$ +10.6 (*c* 0.99, $CHCl_3$); ^{1}H NMR ($CDCl_3$, TMS, 300 MHz) δ 1.50-1.60 (m, 1H), 1.73-1.80 (m, 1H), 1.89-2.02 (m, 2H), 2.14-2.24 (m, 1H),

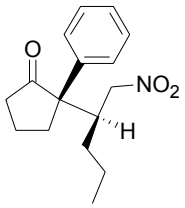
2.41-2.46 (m, 1H), 4.27-4.50 (m, 3H), 6.06 (s, 1H), 6.23 (s, 1H), 7.20-7.38 (m, 6H); ^{13}C NMR ($CDCl_3$, TMS, 75 MHz) δ 18.3, 30.2, 36.9, 43.9, 58.8, 74.2, 109.6, 110.6, 127.1, 128.2, 129.2, 134.4, 142.1, 150.2, 216.3; IR (KBr) ν: 3019, 2973, 2400, 1731, 1560, 1427, 1377, 1217, 1046, 929, 754 cm^{-1}; HRMS Calcd. for $C_{17}H_{17}NO_4$: 299.1158, found: 299.1161; dr = 98:2 , determined by the crude ^{1}HNMR; 94% ee, determined by chiral HPLC analysis (Chiralcel AS-H, *i*-propanol/hexane = 10/90, flow rate 0.5 mL/min, λ = 210 nm): t_{minor} = 34.1 min, t_{major} = 24.7 min.



(3am)

(2*S*, 3*S*)-2-(1-Nitromethyl-3-phenyl-allyl)-2-phenyl-cyclopentanone (table 2, entry 13):

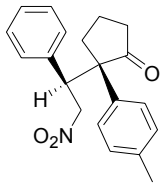
The title compound was prepared according to the general procedure as described above in 89% yield. $[\alpha]^{25}_D$ +41.7 (*c* 1.01, $CHCl_3$); m.p 80 °C; ^{1}H NMR ($CDCl_3$, TMS, 300 MHz) ^{1}H NMR ($CDCl_3$, TMS, 300 MHz) δ 1.62-1.78 (m, 1H), 1.82-1.96 (m, 1H), 2.15-2.38 (m, 3H), 2.56-2.62 (m, 1H), 3.63-3.69 (t, *J* = 8.4 Hz, 1H), 4.02-4.10 (t, *J* = 11.4 Hz, 1H), 4.63-4.68 (dd, *J* = 3.3 and 12.0 Hz, 1H), 5.93-6.01 (dd, *J* = 9.0 and 15.6 Hz, 1H), 6.52-6.57 (d, *J* = 15.6 Hz, 1H), 7.26-7.48 (m, 10H); ^{13}C NMR ($CDCl_3$, TMS, 75 MHz) δ 18.5, 31.4, 37.5, 48.0, 58.2, 76.7, 123.6, 126.7, 127.4, 128.1, 128.2, 128.7, 129.4, 134.9, 135.9, 136.3, 217.0; IR (KBr) ν: 3015, 2980, 2895, 2400, 1735, 1556, 1521, 1474, 1424, 1213, 1042, 929, 770 cm^{-1}; dr > 99:1, determined by the crude ^{1}HNMR; 96% ee, determined by chiral HPLC analysis (Chiralcel AS-H, *i*-propanol/hexane = 10/90, flow rate 0.5 mL/min, λ = 210 nm): t_{minor} = 52.0 min, t_{major} = 41.0 min.



(3an)

(*S*)-2-((*S*)-1-nitropentan-2-yl)-2-phenylcyclohexanone

The catalyst **I-d** (13.5 mg, 0.02 mmol) was added to a vial containing α-phenyl cyclopentanone (0.2 mmol) and aliphatic nitroolefin (0.21 mmol) in CH_2Cl_2 (0.45 mL) at room temperature about 30 h. Then the reaction mixture was concentrated *in vacuo* to obtain the crude product. The crude product was purified by flash silica gel chromatography in 50% yield. $[\alpha]^{25}_D$ +30.6 (*c* 0.2, $CHCl_3$); ^{1}H NMR ($CDCl_3$, TMS, 300 MHz) ^{1}H NMR ($CDCl_3$, TMS, 300 MHz) δ 1.12-1.38 (m, 5H), 1.66-1.71 (m, 2H), 1.91-2.04 (m, 3H), 2.09-2.20 (m, 1H), 2.12-2.26 (m, 1H), 2.53-2.56 (m, 1H), 2.91 (brs, 1H), 3.83-3.89 (dd, *J* = 7.2 and 12.6 Hz, 1H), 4.13-4.18 (m, 1H), 7.20-7.32 (m, 3H), 7.38-7.41 (m, 2H); ^{13}C NMR ($CDCl_3$, TMS, 75 MHz) δ 14.4, 18.5, 21.5, 28.3, 29.5, 33.2, 37.4, 44.5, 59.9, 127.5, 128.2, 129.3, 135.8, 217.8; IR (KBr) ν: 3059, 2961, 2926, 2874, 1732, 1544, 1495, 1462, 1382, 1260, 1151, 1083, 1024, 799, 753cm^{-1}; dr > 99:1, determined by the crude ^{1}HNMR; 80% ee, determined by chiral HPLC analysis (Chiralcel OD-H, *i*-propanol/hexane = 5/95, flow rate 0.5 mL/min, λ = 210 nm): t_{minor} = 17.3 min, t_{major} = 16.1 min.

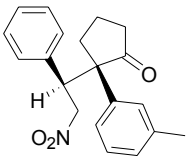


(3ba)

(2*S*, 3*S*)-2-(2-Nitro-1-phenyl-ethyl)-2-*p*-tolyl-cyclopentanone (table 2, entry 14):

The title compound was prepared according to the general procedure as described above in 88% yield. $[\alpha]^{25}_D$ +65.2 (*c* 1.24, $CHCl_3$); ^{1}H NMR ($CDCl_3$, TMS, 300 MHz) ^{1}H NMR ($CDCl_3$, TMS, 300 MHz) δ 1.42-1.62 (m, 1H), 1.65-1.78 (m, 1H), 1.85-2.04 (m, 2H), 2.12-2.27 (m, 2H), 2.29 (s, 3H), 4.06-4.11 (m, 1H), 4.37-4.46 (t, *J* = 12.6 Hz, 1H), 4.80-4.85 (m, 1H), 7.00 (brs, 2H), 7.11-7.18 (m, 7H); ^{13}C NMR ($CDCl_3$, TMS, 75 MHz) δ 18.0, 20.9, 31.4, 37.1, 49.1, 58.2, 76.2, 127.7, 127.8,

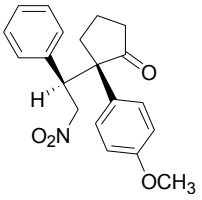
128.2, 129.5, 129.7, 130.9, 135.6, 138.1, 216.9; IR (KBr) ν: 3015, 2969, 2891, 2432, 2397, 1727, 1552, 1513, 1427, 1381, 1217, 1042, 925, 754 cm^{-1}; HRMS Calcd. for $C_{20}H_{21}NO_3$: 323.1521, found: 323.1524; dr > 99:1, determined by the crude ^{1}HNMR; 91% ee, determined by chiral HPLC analysis (Chiralcel AS-H, *i*-propanol/hexane = 10/90, flow rate 0.5 mL/min, λ = 210 nm): t_{minor} = 43.1 min, t_{major} = 22.1 min.



(3ca)

(2*S*, 3*S*)-2-(2-Nitro-1-phenyl-ethyl)-2-*m*-tolyl-cyclopentanone (table 2, entry 15):

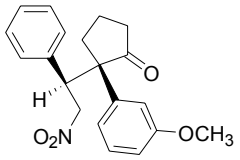
The title compound was prepared according to the general procedure as described above in 91% yield. $[\alpha]^{25}_D$ +100.1 (*c* 0.50, $CHCl_3$); m.p 70 ^oC; ^{1}H NMR ($CDCl_3$, TMS, 300 MHz) ^{1}H NMR ($CDCl_3$, TMS, 300 MHz) δ 1.57-1.75 (m, 1H), 1.77-1.82 (m, 1H), 1.95-2.12 (m, 2H), 2.21-2.30 (m, 2H), 2.37 (s, 3H), 4.15-4.20 (dd, *J* = 3.3 and 12 Hz, 1H), 4.45-4.54 (t, *J* = 12.6 Hz, 1H), 4.83-4.89 (dd, *J* = 3.9 and 13.8 Hz, 1H), 7.05-7.07 (m, 2H), 7.13-7.16 (m, 3H), 7.26-7.30(m, 4H); ^{13}C NMR ($CDCl_3$, TMS, 75 MHz) δ 18.1, 21.6, 31.3, 37.2, 49.1, 58.5, 76.1, 124.7, 127.8, 128.2, 128.4, 128.8, 128.9, 129.6, 134.1, 135.5, 138.7, 216.8; IR (KBr) ν: 3023, 2972, 2891, 2432, 2404, 1731, 1559, 1521, 1478, 1424, 1381, 1210, 1046, 925, 762 cm^{-1}; HRMS Calcd. for $C_{20}H_{21}NO_3$: 323.1521, found: 323.1520; dr > 99:1, determined by the crude ^{1}HNMR; 90% ee, determined by chiral HPLC analysis (Chiralcel AS-H, *i*-propanol/hexane = 10/90, flow rate 0.5 mL/min, λ = 210 nm): t_{minor} = 32.4 min, t_{major} = 21.9 min.



(3da)

(2*S*, 3*S*)-2-(4-Methoxy-phenyl)-2-(2-Nitro-1-phenyl-ethyl)-cyclopentanone (table 2, entry 16):

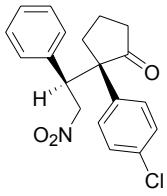
The title compound was prepared according to the general procedure as described above in 93% yield. $[\alpha]^{25}_{D}$ +86.9 (*c* 0.82, $CHCl_3$); ^{1}H NMR ($CDCl_3$, TMS, 300 MHz) ^{1}H NMR ($CDCl_3$, TMS, 300 MHz) δ 1.42-1.60 (m, 1H), 1.62-1.69 (m, 1H), 1.90-2.02 (m, 2H), 2.10-2.21 (m, 2H), 3.76 (s, 3H), 4.04-4.08 (m, 1H), 4.38-4.46 (t, *J* = 12.3 Hz, 1H), 4.83-4.87 (m, 1H), 6.83-6.86 (t, *J* = 9 Hz, 2H), 6.96-6.99 (m, 2H), 7.18-7.20(m, 5H); ^{13}C NMR ($CDCl_3$, TMS, 75 MHz) δ 18.3, 31.9, 37.4, 49.4, 55.5, 58.1, 76.5, 114.6, 125.9, 128.0, 128.5, 129.3, 129.8, 135.9, 159.6, 217.2; IR (KBr) ν: 3018, 2972, 2891, 2397, 1731, 1602, 1559, 1511, 1479, 1421, 1213, 1042, 929, 758 cm^{-1}; HRMS Calcd. for $C_{20}H_{21}NO_4$: 339.1471, found: 339.1469; dr > 99:1, determined by the crude ^{1}HNMR; 90% ee, determined by chiral HPLC analysis (Chiralcel AS-H, *i*-propanol/hexane = 10/90, flow rate 1.0 mL/min, λ = 210 nm): t_{minor} = 30.0 min, t_{major} = 19.2 min.



(3ea)

(2*S*, 3*S*)-2-(3-Methoxy-phenyl)-2-(2-Nitro-1-phenyl-ethyl)-cyclopentanone (table 2, entry 17):

The title compound was prepared according to the general procedure as described above in 90% yield. $[\alpha]^{25}_{D}$ +85.9 (*c* 0.71, $CHCl_3$); m.p 85 ^{o}C; ^{1}H NMR ($CDCl_3$, TMS, 300 MHz) δ 1.56-1.72 (m, 1H), 1.76-1.82 (m, 1H), 1.96-2.15 (m, 2H), 2.21-2.35 (m, 2H), 3.81 (s, 3H), 4.16-4.21 (dd, *J* = 4.2 and 12.6 Hz, 1H), 4.48-4.57 (t, *J* = 12.3 Hz, 1H), 4.83-4.89 (dd, *J* = 4.2 and 13.2 Hz, 1H), 6.88-6.96 (m, 3H), 7.08-7.10 (m, 2H), 7.26-7.35(m, 4H); ^{13}C NMR ($CDCl_3$, TMS, 75 MHz) δ 18.2, 31.4, 37.2, 49.1, 55.3, 58.6, 76.1, 113.1, 114.0, 119.3, 127.9, 128.2, 129.6, 129.9, 135.5, 135.9, 159.9, 216.7; IR (KBr) ν: 3019, 2972, 2895, 2397, 1735, 1600, 1556, 1521, 1424, 1217, 1046, 929, 754 cm^{-1}; HRMS Calcd. for $C_{20}H_{21}NO_4$: 339.1471, found: 339.1475; dr > 99:1, determined by the crude ^{1}HNMR; 91% ee, determined by chiral HPLC analysis (Chiralcel AS-H, *i*-propanol/hexane = 10/90, flow rate 1.0 mL/min, λ = 210 nm): t_{minor} = 27.3 min, t_{major} = 19.3 min.

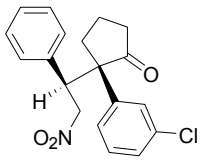


(3fa)

(2*S*, 3*S*)-2-(4-Chloro-phenyl)-2-(2-Nitro-1-phenyl-ethyl)-cyclopentanone (table 2, entry 18):

The title compound was prepared according to the general procedure as described above in 89% yield. $[\alpha]^{25}_{D}$ +73.1 (*c* 1.01, $CHCl_3$); ^{1}H NMR ($CDCl_3$, TMS, 300 MHz) δ 1.56-1.66 (m, 1H), 1.79-1.88 (m, 1H), 2.01-2.17 (m, 2H), 2.22-2.35 (m, 2H), 4.11-4.16 (dd, *J* = 4.2 and 12.3 Hz, 1H), 4.45-4.53 (t, *J* = 12.3 Hz, 1H), 4.88-4.94 (dd, *J* = 4.5 and 13.5 Hz, 1H), 7.01-7.04 (m, 2H), 7.26-7.31 (m, 5H), 7.36-7.39(m, 2H); ^{13}C NMR ($CDCl_3$, TMS, 75 MHz) δ 18.2, 31.9, 37.5, 49.1, 58.1, 76.0, 128.2, 128.5, 129.3, 129.4, 129.6, 133.2, 134.5, 135.2, 216.8; IR (KBr) ν: 3019, 2976, 2891, 2439, 2400, 1731, 1599, 1556, 1525, 1424, 1377, 1217, 1046, 929, 762 cm^{-1}; HRMS Calcd. for $C_{19}H_{18}ClNO_3$: 343.0970, found: 343.0969; dr > 99:1, determined by the crude ^{1}HNMR; 91% ee, determined by chiral HPLC analysis (Chiralcel AS-H, *i*-propanol/hexane = 10/90, flow rate 0.5 mL/min, λ = 210 nm): t_{minor} = 35.6 min, t_{major} = 28.4 min.

.

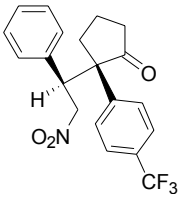


(3ga)

(2*S*, 3*S*)-2-(3-Chloro-phenyl)-2-(2-Nitro-1-phenyl-ethyl)-cyclopentanone (table 2, entry 19):

The title compound was prepared according to the general procedure as described above in 95% yield. $[\alpha]^{25}_{D}$ +54.5 (*c* 1.09, $CHCl_3$); m.p 107.6 ^{o}C; ^{1}H NMR ($CDCl_3$, TMS, 300 MHz) δ 1.56-1.68 (m, 1H), 1.80-1.88 (m, 1H), 2.01-2.18 (m, 2H), 2.24-2.33 (m, 2H), 4.13-4.18 (dd, *J* = 4.2 and 12.3 Hz, 1H), 4.47-4.55 (t, *J* = 12.6 Hz, 1H), 4.85-4.91 (dd, *J* = 3.9 and 13.2 Hz, 1H), 7.03-7.06 (m, 2H), 7.26-7.36 (m, 7H); ^{13}C NMR ($CDCl_3$, TMS, 75 MHz) δ 18.1, 31.5, 37.4, 48.9, 58.2, 75.8, 126.0, 127.9,

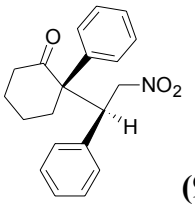
128.1, 128.3, 128.4, 129.5, 130.2, 134.9, 135.0, 136.8, 216.3; IR (KBr) ν: 3019, 2973, 2890, 2432, 2398, 1735, 1556, 1521, 1424, 1213, 1046, 925, 758 cm^{-1}; dr > 99:1, determined by the crude ^{1}HNMR; 94% ee, determined by chiral HPLC analysis (Chiralcel AS-H, *i*-propanol/hexane = 10/90, flow rate 0.5 mL/min, λ = 210 nm): t_{minor} = 45.8 min, t_{major} = 33.5 min.



(3ha)

(2*S*, 3*S*)-2-(2-Nitro-1-phenyl-ethyl)-2-(4-trifluoromethyl-phenyl)-cyclopentanone (table 2, entry 20):

The title compound was prepared according to the general procedure as described above in 90% yield. $[\alpha]^{25}_D$ +35.3 (*c* 1.16, $CHCl_3$); m.p 65.5 ^oC; ^{1}H NMR ($CDCl_3$, TMS, 300 MHz) δ 1.50-1.63 (m, 1H), 1.70-1.82 (m, 1H), 2.03-2.36 (m, 4H), 4.09-4.15 (dd, *J* = 3.9 and 12.0 Hz, 1H), 4.38-4.47 (t, *J* = 12.3 Hz, 1H), 4.78-4.84 (dd, *J* = 4.5 and 13.5 Hz, 1H), 6.95-6.97 (m, 2H), 7.19-7.21 (m, 3H), 7.44-7.46 (d, *J* = 8.1 Hz, 2H), 7.58-7.61 (d, *J* = 8.7 Hz, 2H); ^{13}C NMR ($CDCl_3$, TMS, 75 MHz) δ 18.3, 31.8, 37.6, 49.1, 58.5, 75.8, 125.93, 125.98, 128.3, 128.5, 128.6, 129.6, 134.9, 139.1, 216.5; IR (KBr) ν: 3018, 2976, 2891, 2428, 2400, 1736, 1556, 1525, 1420, 1324, 1213, 1170, 1131, 1069, 1050, 925, 758 cm^{-1}; dr > 99:1, determined by the crude ^{1}HNMR; 95% ee, determined by chiral HPLC analysis (Chiralcel AS-H, *i*-propanol/hexane = 10/90, flow rate 0.5 mL/min, λ = 210 nm): t_{minor} = 29.9 min, t_{major} = 20.9 min.

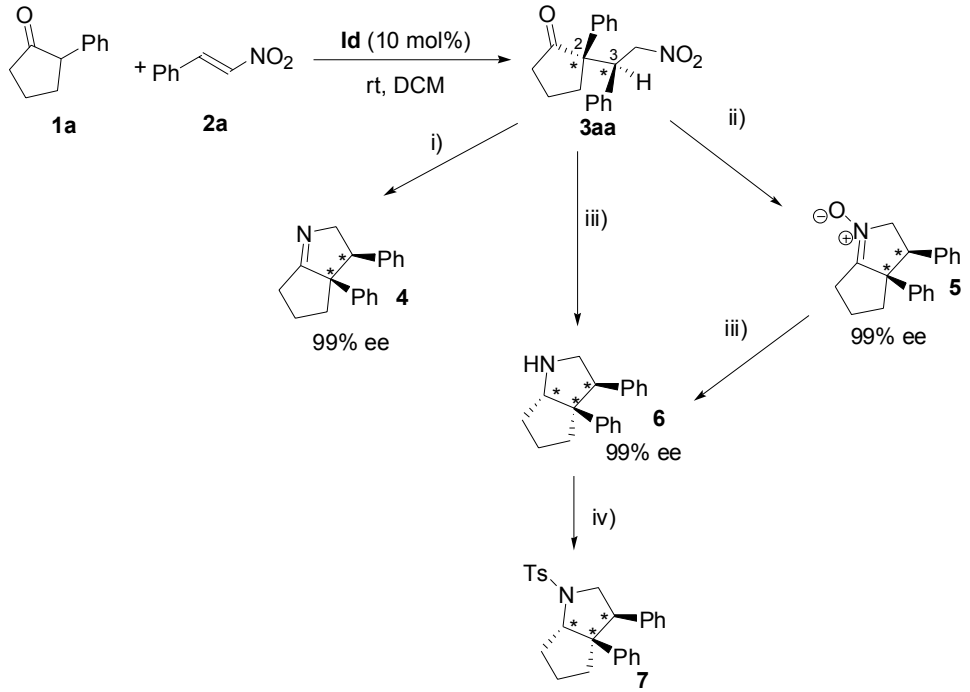


(9)

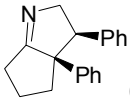
(*S*)-2-((*S*)-2-nitro-1-phenylethyl)-2-phenylcyclohexanone

The catalyst **I-d** (13.5 mg, 0.02 mmol) was added to a vial containing α-phenyl cyclohexanone (0.2 mmol) and nitroolefin (0.21 mmol) in $CHCl_3$ (0.45 mL) at 50 °C about 30 h. Then the reaction mixture was concentrated *in vacuo* to obtain the crude product. The crude product was purified by flash silica gel chromatography in 20% yield. $[\alpha]^{25}_D$ +25.2 (*c* 0.34, $CHCl_3$); ^{1}H NMR ($CDCl_3$, TMS, 300 MHz) δ 1.50-1.63 (m, 2H), 1.70-1.95 (m, 3H), 2.05-2.15 (m, 1H), 2.20-2.30 (m, 1H), 2.35-2.50 (m, 1H), 4.02-4.20 (m, 2H), 5.08-5.11 (d, *J* = 10.8 Hz, 1H), 6.70-6.73 (d, *J* = 6.6 Hz, 1H), 6.79-6.81 (d, *J* = 4.8 Hz, 2H), 7.08-7.18 (m, 3H), 7.24-7.31 (m, 3H); ^{13}C NMR ($CDCl_3$, TMS, 75 MHz) δ 21.3, 29.1, 29.9, 37.5, 40.4, 49.4, 59.6, 127.98, 128.1, 128.6, 128.8, 129.1, 130.3, 135.8, 136.0, 213.4; IR (KBr) ν: 3060, 3029, 2925, 2855, 1703, 1551, 1469, 1449, 1377, 1308, 1278, 1119, 1086, 802, 759 cm^{-1}; The dr was > 99:1, determined by the crude ^{1}HNMR; 84% ee, determined by chiral HPLC analysis (Chiralcel AS-H, *i*-propanol/hexane = 10/90, flow rate 0.5 mL/min, λ = 210 nm): t_{minor} = 26.6 min, t_{major} = 28.2 min.

3. Synthetic Transformation of the Michael adduct 3aa

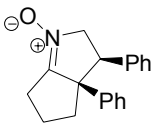


Conditions: i) Zn/HCl, 40 °C, 78% yield; ii) Pd/C, H_2 (20 atm), 50°C, 10 h, 60% yield; iii) Pd/C, H_2 (40 atm), 80°C, 30 h, 63% yield; iv) TsCl/Et_3N/DCM, 91% yield.



(4)

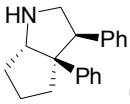
3,3a-Diphenyl-2,3,3a,4,5,6-hexahydro-cyclopenta[*b*]pyrrole:[3] A 100mL round bottom flask with well-stirring was filled with Zinc powder 945 mg (15 mmol, 15eq.), and optical pure **3aa** 309.4 mg (1.0 mmol), then EtOH (9.5 mL) was added. After the mixture was kept at 40 °C for 10 min, 6.4 mL 4 M HCl aq. was added dropwise over 2 h. The contents were stirred at 40 °C for 24 h. To maintain the pH value of the mixture between 0-1, additional 4N HCl aq. was added. After the reaction was completed to afford the final product (monitored by TLC), EtOH was removed under reduced pressure, 3N NaOH aq. (20 mL) was added, and then the solution was stirred for 5 min. After diluted with 15 mL CH_2Cl_2, the mixture was filtrated through a plug of Celite. The layers were separated. The acquired aqueous layer was extracted with CH_2Cl_2, and then the combined organic layers were washed with brine and dried over anhydrous $MgSO_4$ and concentrated. The crude oil was purified by flash chromatography on silica gel to afford the product **4** (204.4 mg, 78% yield). $[\alpha]^{25}_D$ +369.8 (*c* 0.44, $CHCl_3$); ^{1}H NMR ($CDCl_3$, TMS, 300 MHz) δ 1.83-2.01 (m, 2H), 2.13-2.18 (m, 1H), 2.45-2.51 (m, 1H), 2.59 (brs, 2H), 3.67-3.73 (m,, 1H), 4.24-4.32 (m, 1H), 4.38-4.45 (m, 1H), 6.67-6.76 (m, 4H), 7.03-7.15 (m, 5H); ^{13}C NMR ($CDCl_3$, TMS, 75 MHz) δ 25.3, 25.6, 37.6, 59.3, 69.0, 69.3, 126.8, 126.9, 127.8, 127.9, 128.4, 128.6, 137.6, 138,2, 192.2; IR (KBr) ν: 3019, 2973, 2899, 2428, 2400, 1667, 1601, 1522, 1497, 1422, 1216, 1047, 928, 758 cm^{-1}; HRMS Calcd. for $C_{19}H_{19}N$: 261.1517, found: 261.1521; >99% ee, determined by chiral HPLC analysis (Chiralcel AD-H, *i*-propanol/hexane = 10/90, flow rate 0.25 mL/min, λ = 210 nm): t_{major} = 21.7 min.



(5)

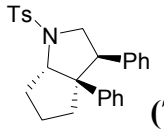
3,3a-Diphenyl-2,3,3a,4,5,6-hexahydro-cyclopenta[*b*]pyrrole 1-oxide:[4]

A suspension of Pd/C (20 mg) and **3aa** (200 mg) in MeOH (5 mL) was stirred at 50℃ under 20 atm hydrogen atmosphere. After being stirred for 10 h, the mixture was filtrated through a pad of Celite and the filtration was concentrated in vacuo, the residue was purified by column chromatography on silica gel to afford the desired product **5** (178 mg, 60% yield). $[\alpha]^{25}_{D}$ +10.5 (*c* 1.0, $CHCl_3$); ^{1}H NMR ($CDCl_3$, TMS, 300 MHz) δ 1.80-1.86 (m, 1H), 1.96-2.08 (m, 1H), 2.13-2.22 (m, 1H), 2.43-2.54 (m, 2H), 2.73-2.82 (m, 1H), 4.02-4.09 (dd, *J* = 8.1 and 11.4 Hz, 1H), 4.23-4.30 (dd, *J* = 8.1 and 12.3 Hz, 1H), 4.74-4.82 (t, *J* = 12.3 Hz, 1H), 6.68-6.77 (m, 4H), 7.07-7.15 (m, 5H); ^{13}C NMR ($CDCl_3$, TMS, 75 MHz) δ 22.5, 26.4, 38.9, 55.1, 65.9, 70.7, 127.5, 127.7, 128.4, 128.6, 135.3, 137.7, 158.6; IR (KBr) ν: 3019, 2976, 2891, 2430, 2400, 1521, 1477，1423, 1216, 1046, 928, 904, 758 cm^{-1}; HRMS Calcd. for $C_{19}H_{19}NO$: 277.1467, found: 277.1468.



(6)

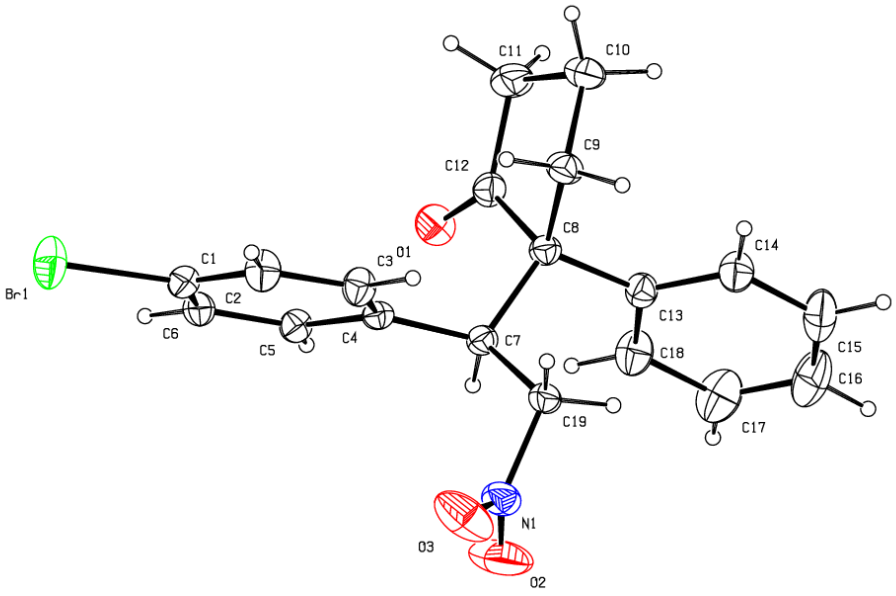
3,3a-Diphenyl-2,3,3a,4,5,6-hexahydro-cyclopenta[*b*]pyrrole: A suspension of Pd/C (30 mg) and **3aa** (300 mg) in MeOH (6 mL) was stirred at 80℃ under 40 atm hydrogen atmosphere. After being stirred for 30 h, the mixture was filtrated through a pad of Celite and the filtration was concentrated in vacuo, the residue was purified by column chromatography on silica gel to afford the desired product **6** (255 mg, 63% yield, d.r. > 99:1). $[\alpha]^{25}_{D}$ +65.4 (*c* 0.44, $CHCl_3$); ^{1}H NMR ($CDCl_3$, TMS, 300 MHz) δ 1.75-2.04 (m, 4H), 2.12-2.16 (m, 1H), 2.40-2.45 (m, 1H), 3.23-3.29 (m, 1H), 3.36-3.39 (m, 2H), 4.45 (brs, 1H), 4.57 (m, 1H), 6.59-6.62 (d, *J* = 7.2 Hz, 2H), 6.96-7.08 (m, 7H); ^{13}C NMR ($CDCl_3$, TMS, 75 MHz) δ 23.7, 34.1, 38.7, 50.3, 56.1, 63.6, 68.7, 126.3, 126.8, 127.8, 128.1, 128.5, 128.6, 137.4, 144.5; IR (KBr) ν: 3450，3019, 2976, 2891, 2426, 2400, 1521, 1477，1423, 1216, 1046, 929, 758 cm^{-1}; HRMS Calcd. for $C_{19}H_{21}N$: 263.1674, found: 263.1744.



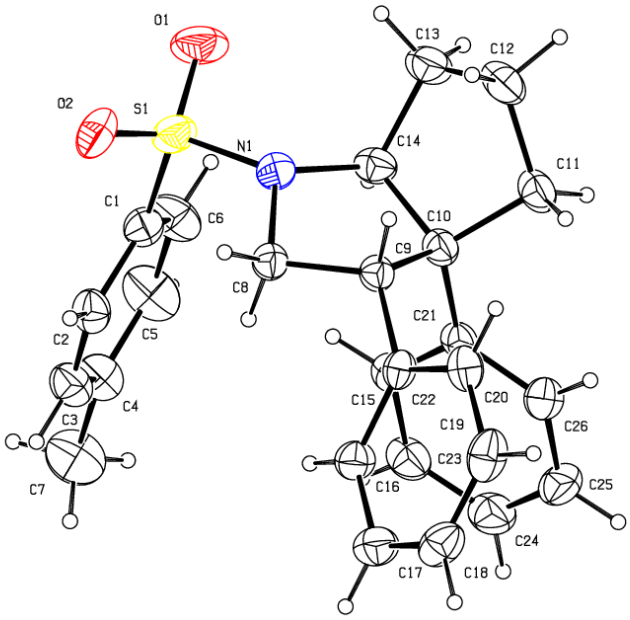
(7)

3,3a-Diphenyl-1-(toluene-4-sulfonyl)-octahydro-cyclopenta[*b*]pyrrole: To a magnetically stirred solution of **6** (263 mg, 1 mmol) in dry DCM (5 ml), Et_3N (0.42 ml, 3 mmol) was aded, then TsCl (285 mg, 1.5 mmol) in 1 mL DCM was added slowly. After the reaction was completed to afford the final product (monitored by TLC), DCM was removed in vacuo, the residue was purified by column chromatography on silica gel to afford the desired product **7** (431 mg, 91% yield). $[\alpha]^{25}_D$ +60.9 (*c* 1.48, $CHCl_3$); m.p. 216.5 ^oC; ^1H NMR ($CDCl_3$, TMS, 300 MHz) δ 1.61-1.64 (m, 1H), 1.80-1.89 (m, 1H), 1.90-2.02 (m, 1H), 2.04-2.20 (m, 1H), 2.29-2.35 (m, 2H), 2.58 (s, 3H), 3.30-3.34 (m, 1H), 3.39-3.41 (m, 1H), 3.71-3.76 (dd, *J* = 6.3 and 8.1 Hz, 1H), 4.21-4.23 (d, *J* = 7.2 Hz, 1H), 5.96-5.98 (d, *J* = 7.5 Hz, 2H), 6.41-6.44 (d, *J* = 7.8 Hz, 2H), 6.70-6.75 (t, *J* = 7.8 Hz, 2H), 6.93-6.98 (t, *J* = 7.8 Hz, 3H), 7.03-7.08 (d, *J* = 7.2 Hz, 1H), 7.49-7.52 (d, *J* = 7.8 Hz, 2H), 7.91-7.94 (d, *J* = 8.1 Hz, 2H); ^{13}C NMR ($CDCl_3$, TMS, 75 MHz) δ 21.9, 23.5, 35.3, 37.3, 52.3, 52.5, 63.6, 70.3, 126.3, 127.2, 127.6, 127.8, 128.4, 128.7, 130.3, 133.6, 136.0, 143.8, 144.2; IR (KBr) ν: 3019, 2976, 2895, 2428, 2400, 1599, 1520, 1475, 1423, 1374, 1213, 1162, 1047, 928, 877, 769，669 cm^{-1}; HRMS Calcd. for $C_{26}H_{27}NO_2S$: 417.1762, found: 417.1844; 99% ee, determined by chiral HPLC analysis (Chiralcel AS-H, *i*-propanol/hexane = 10/90, flow rate 1.0 mL/min, λ = 210 nm): t_{major} = 36.9 min.

4. The absolute configuration of (2*S*,3*S*)-3ad and (2*S*,3*S*,4*S*)-7 were determined by X-ray diffraction analysis



Crystal data for (2*S*,3*S*)-**3ad**: $C_{19}H_{18}BrNO_3$, M_r = 388.25, T = 293 K, Orthorhombic, space group $P2_12_12_1$, a = 8.367(4), b = 13.132(6), c = 16.078(8) Å, V = 1766.6(15) Å^3, Z = 4, 3435 unique reflections, final R_1 = 0.0354 and wR_2 = 0.0922 for 2914 observed [I>2σ(I)] reflections. CCDC 768128 contains the supplementary crystallographic data for this paper. These data can be obtained free of charge via www.ccdc.cam.ac.uk/conts/retrieving.html (or from the Cambridge Crystallographic Data Centre, 12, Union Road, Cambridge CB21EZ, UK; fax: (+44) 1223-336-033; or deposit@ccdc.cam.ac.uk).

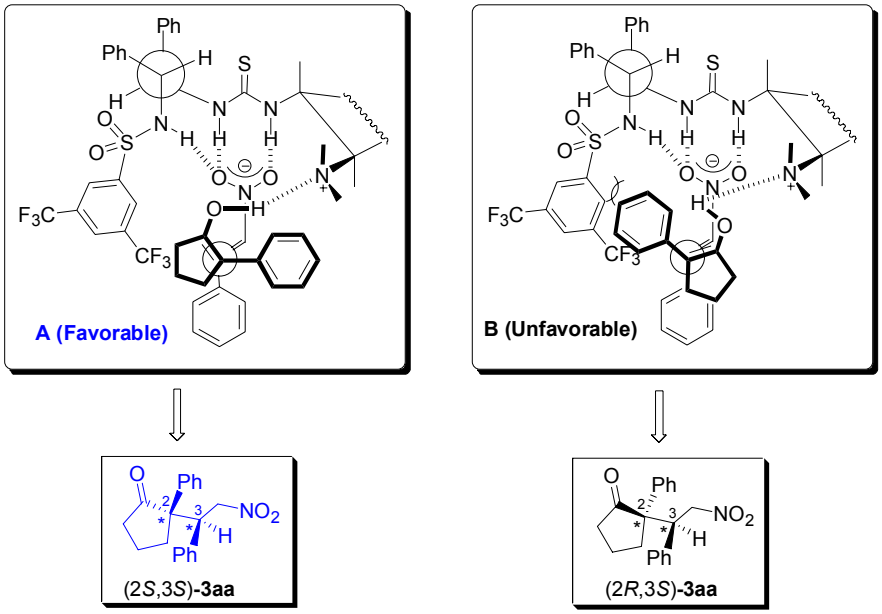


Crystal data for (2*S*,3*S*,4*S*)-**7**: $C_{26}H_{27}NO_2S$, M_r = 417.55, T = 293 K, Orthorhombic, space group $P2_12_12_1$, a = 7.4773(6), b = 11.8993(9), c = 25.2455(19) Å, V = 2246.2(3) Å^3, Z = 4, 4657 unique reflections, final R_1 = 0.0364 and wR_2 = 0.0787 for 3641 observed [I>2σ(I)] reflections. CCDC 768129 contains the supplementary crystallographic data for this paper. These data can be obtained free of charge via www.ccdc.cam.ac.uk/conts/retrieving.html (or from the Cambridge Crystallographic Data Centre, 12, Union Road, Cambridge CB21EZ, UK; fax: (+44) 1223-336-033; or deposit@ccdc.cam.ac.uk).

5. Proposed transition-state models through synergistic dual activation

Based on the absolute configuration of (2*S*,3*S*)-**3ad** and previous studies[6], a plausible dual acitivation model accounting for the obsevered selectivity of the addition of α-phenyl cyclopentanones to nitroolefins in the presence of **I-d** as a catalyst is shown in Figure 2, in which the thiourea and sulfonamide moiety interact through hydrogen bonding with a nitro group of the nitroolefins and enhances their electrophilicity while the neighboring tertiary amine deprotonates the α-proton of α-phenyl cyclopentanones

and enhances their nucleophilicity simultaneously. Compared with the extremly slow reaction rate exhibited in the single base-catalyzed racemic version of this Michael addition,[7] the synergistic activation of both nitroolefins and α-phenyl cyclopentanones through the bifunctional catalyst **I-d** might be responsible for the significantly enhanced reactivity. To avoid the highly steric congestion imposed by the aryl ring of **I-d** and the phenyl group of **1**, the Michael addition would proceed predominantly via the favored transition state **A** giving the adduct **3aa** in (2*S*,3*S*)-configuration, which is compatible with the experimental results. Nevertheless, the real catalytic mechanism still needs further investigation.



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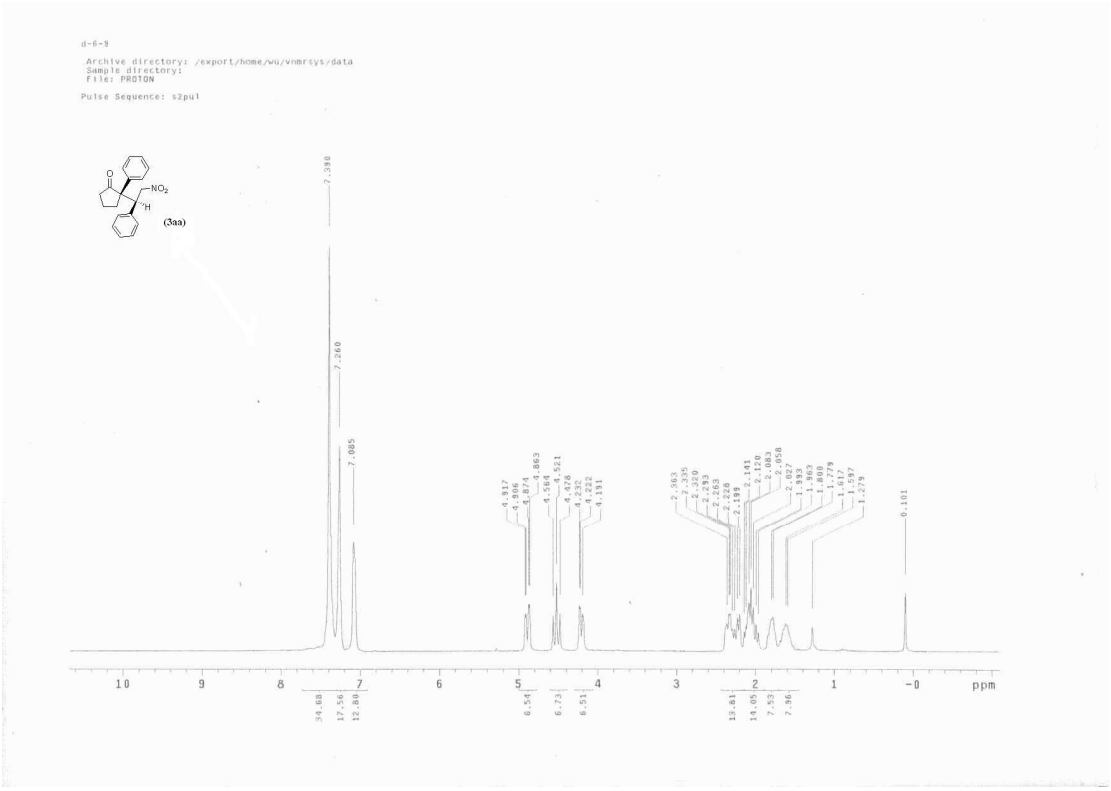
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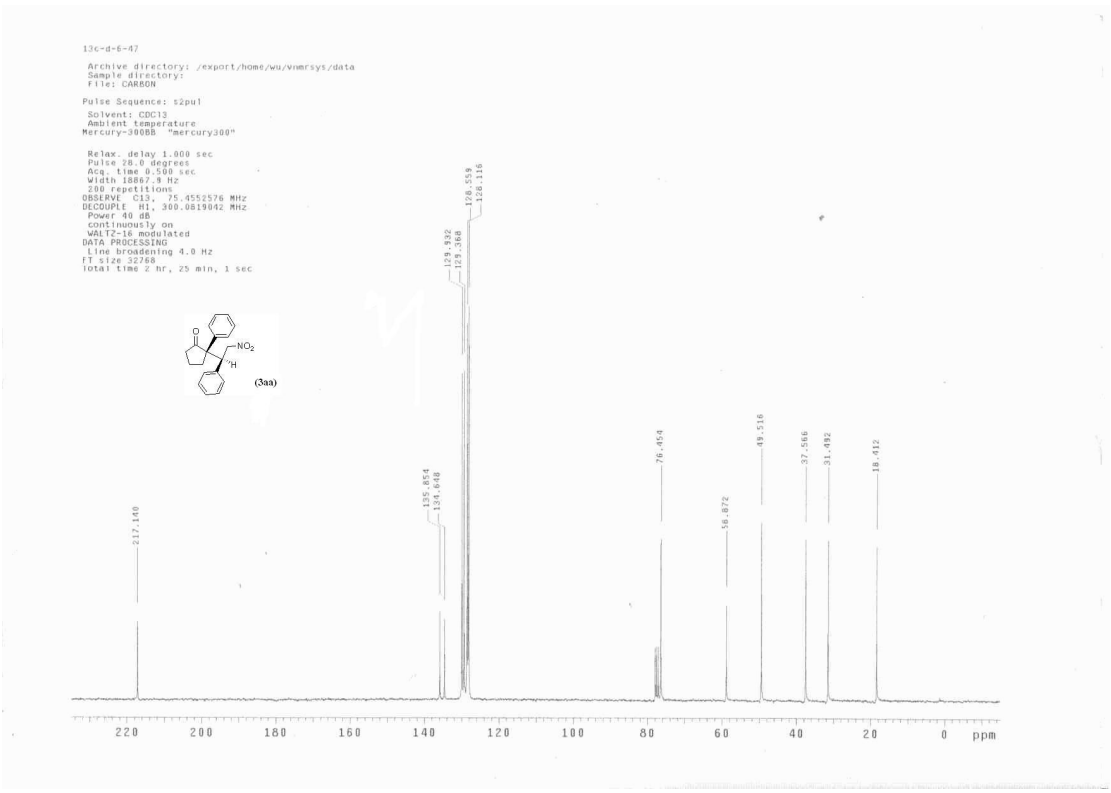
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continuously on
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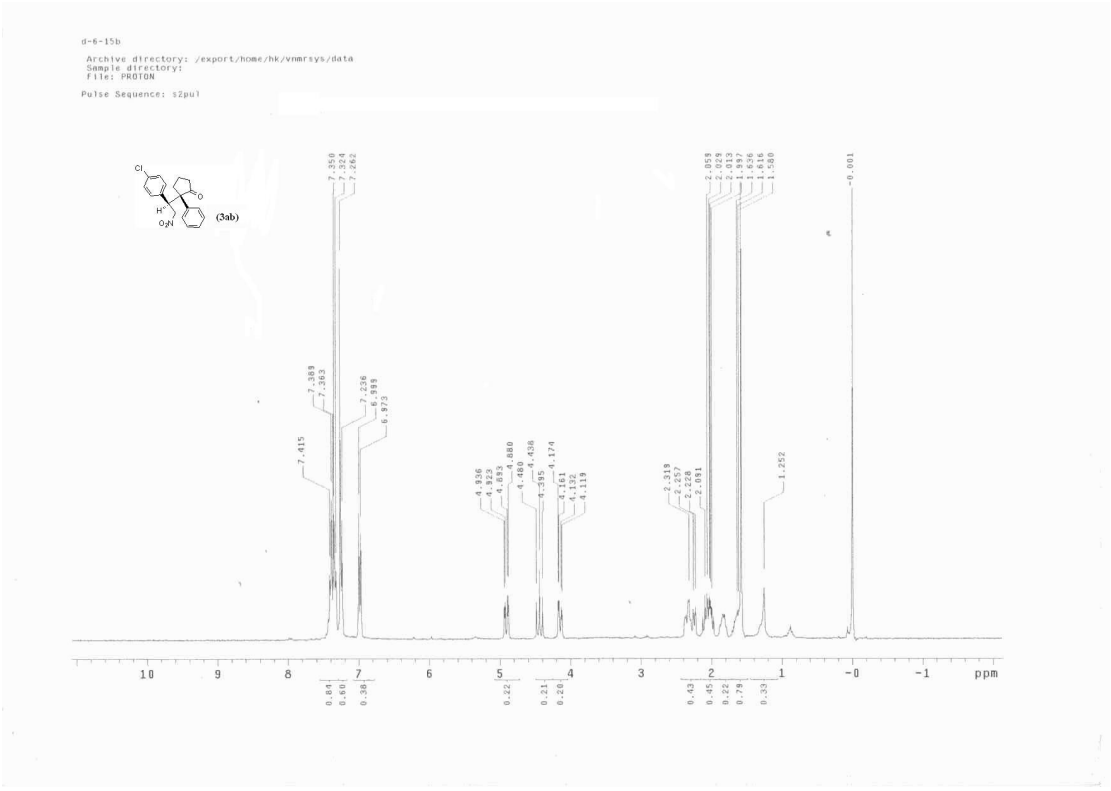
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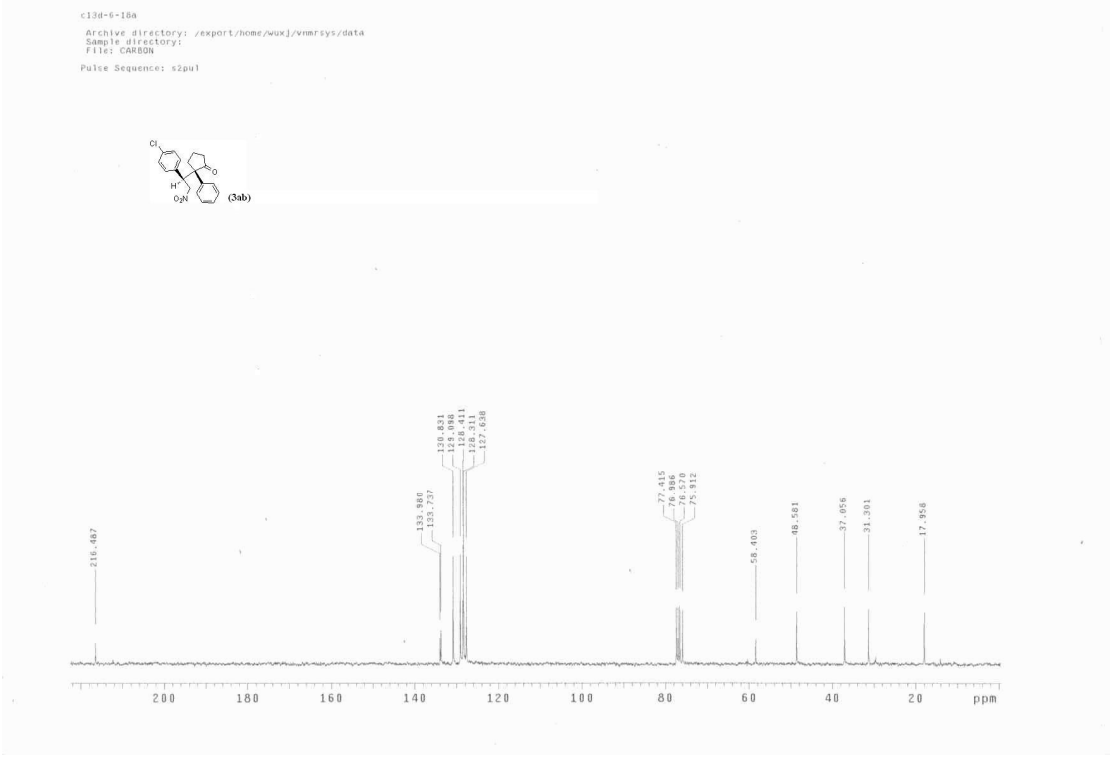
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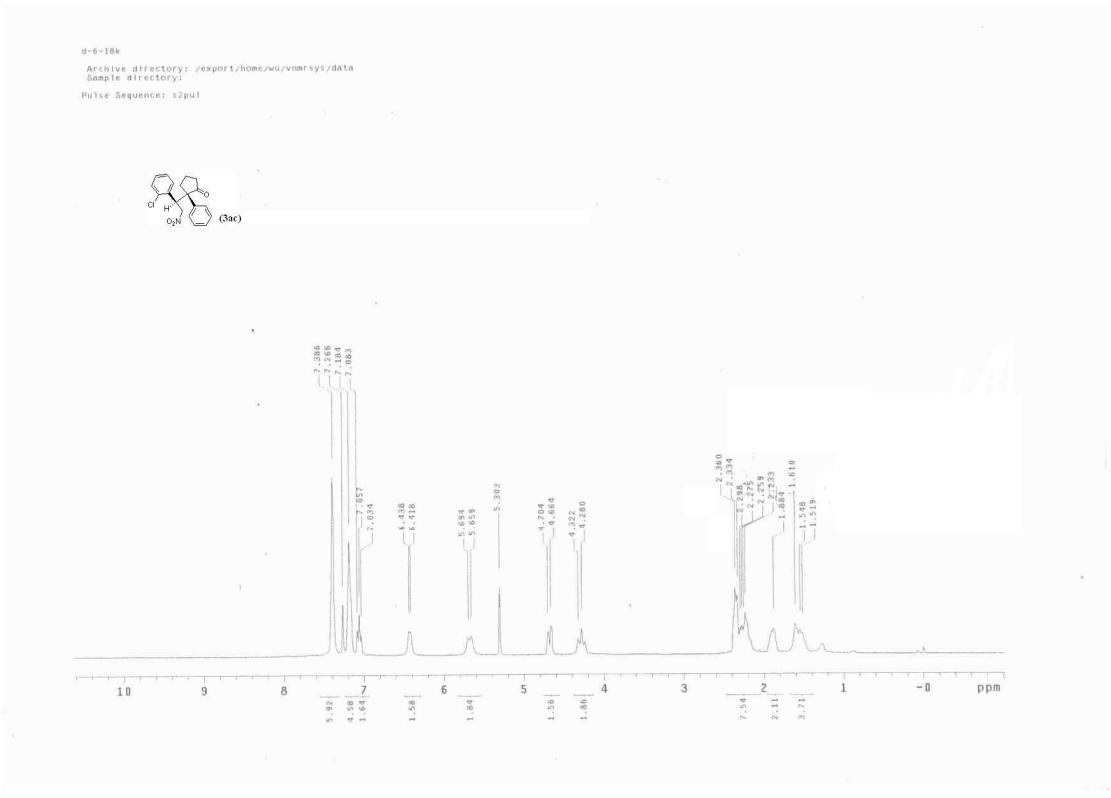
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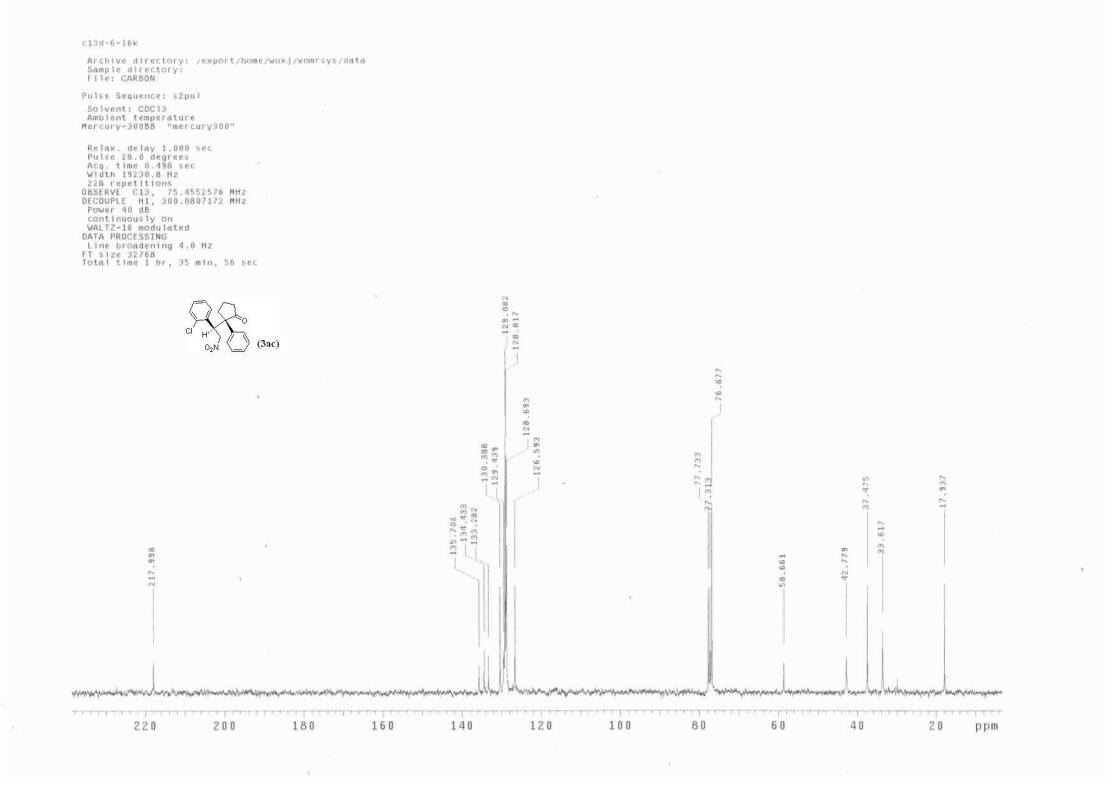
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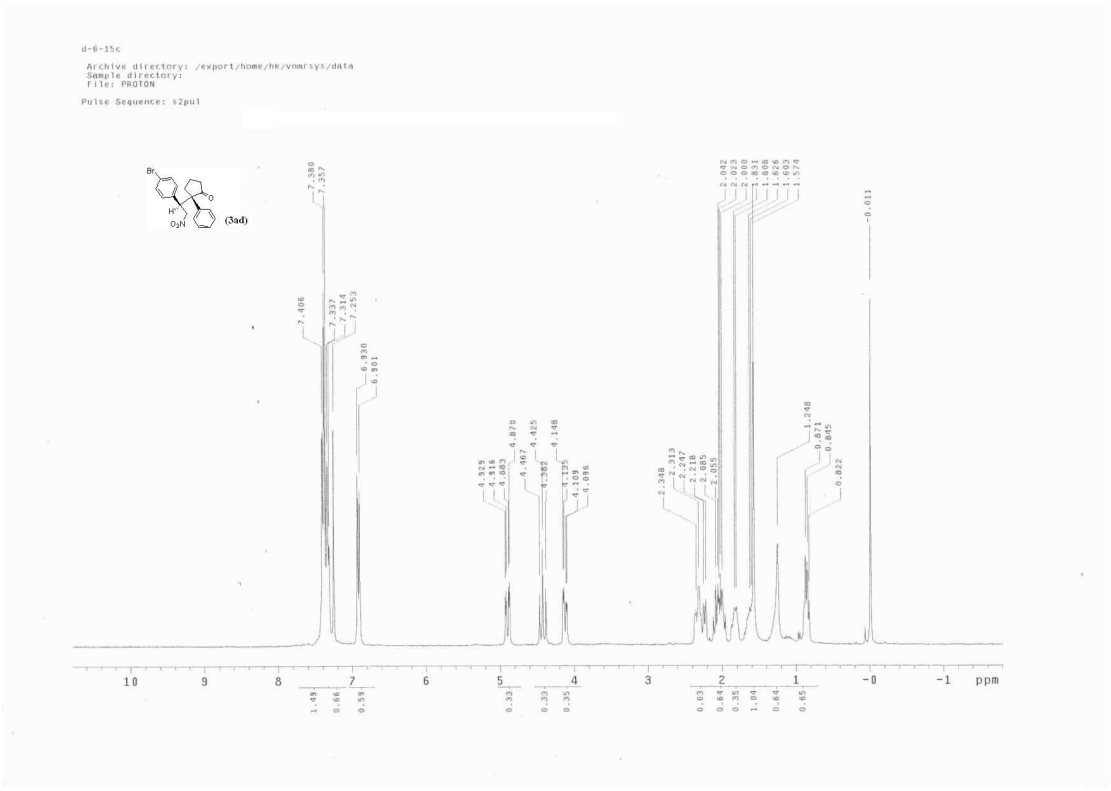
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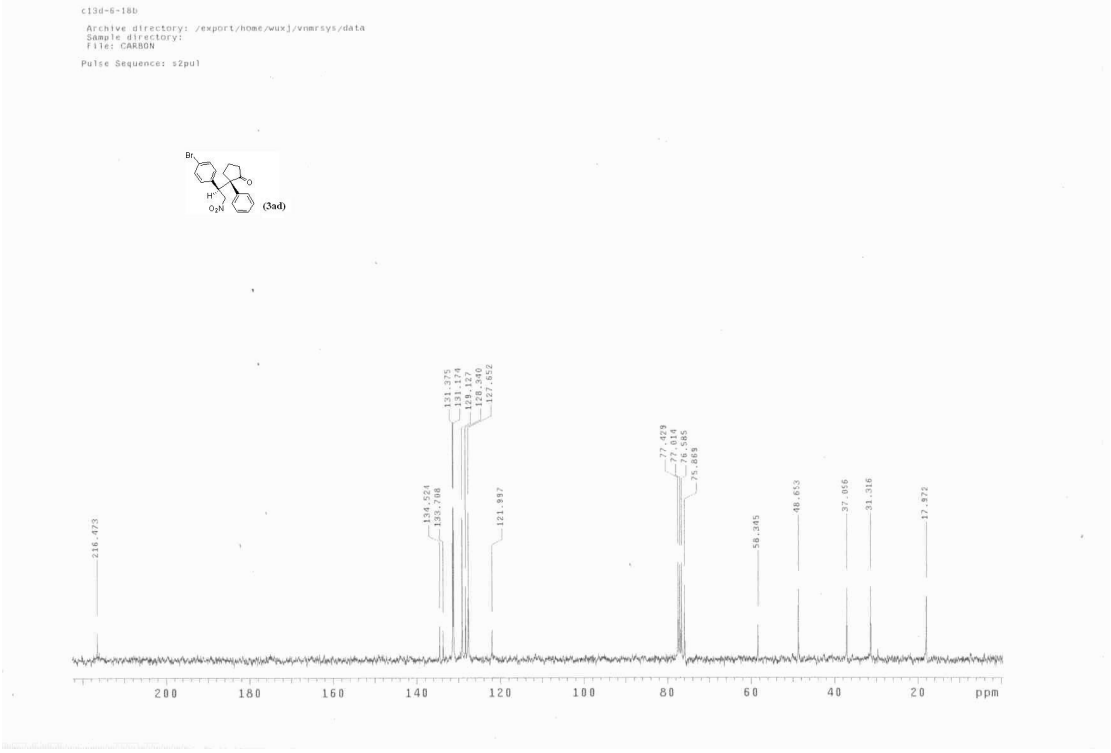
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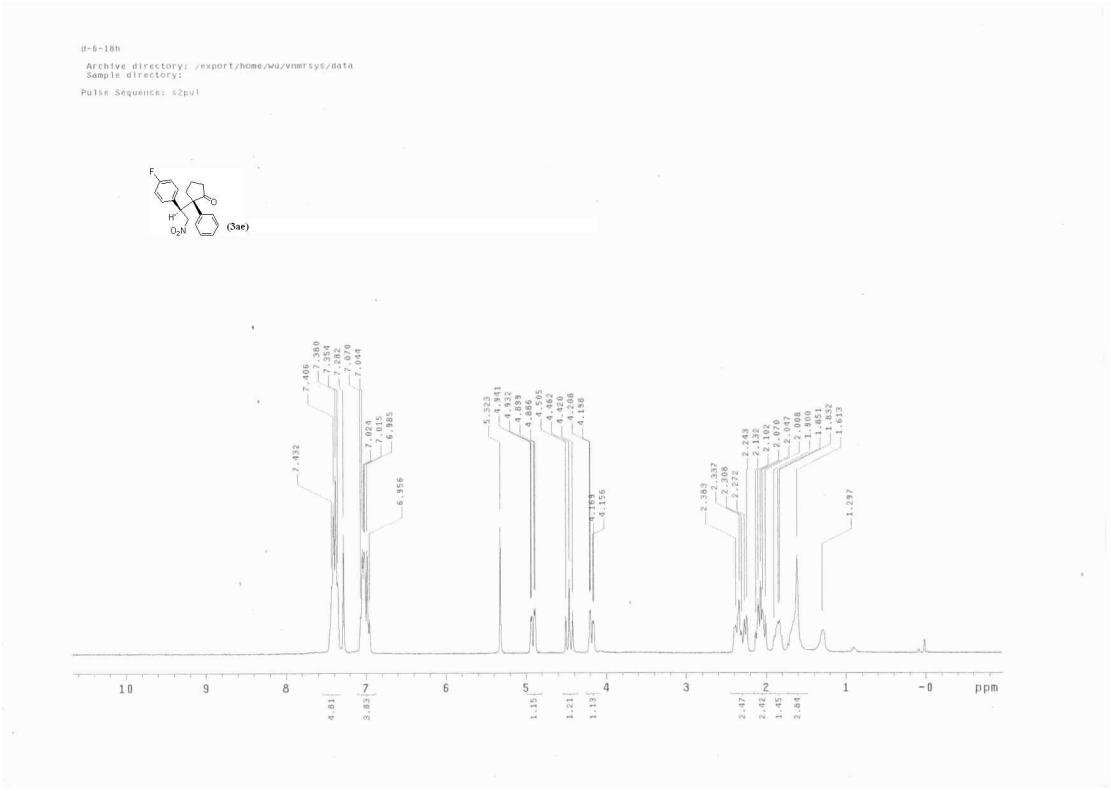
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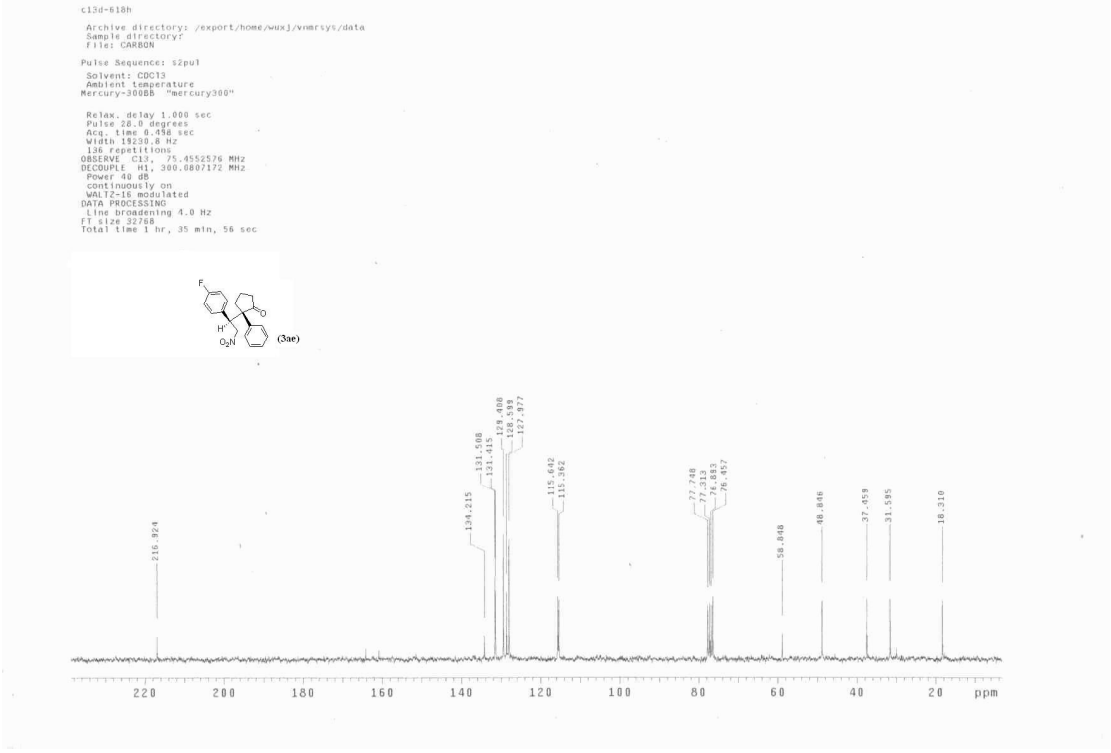
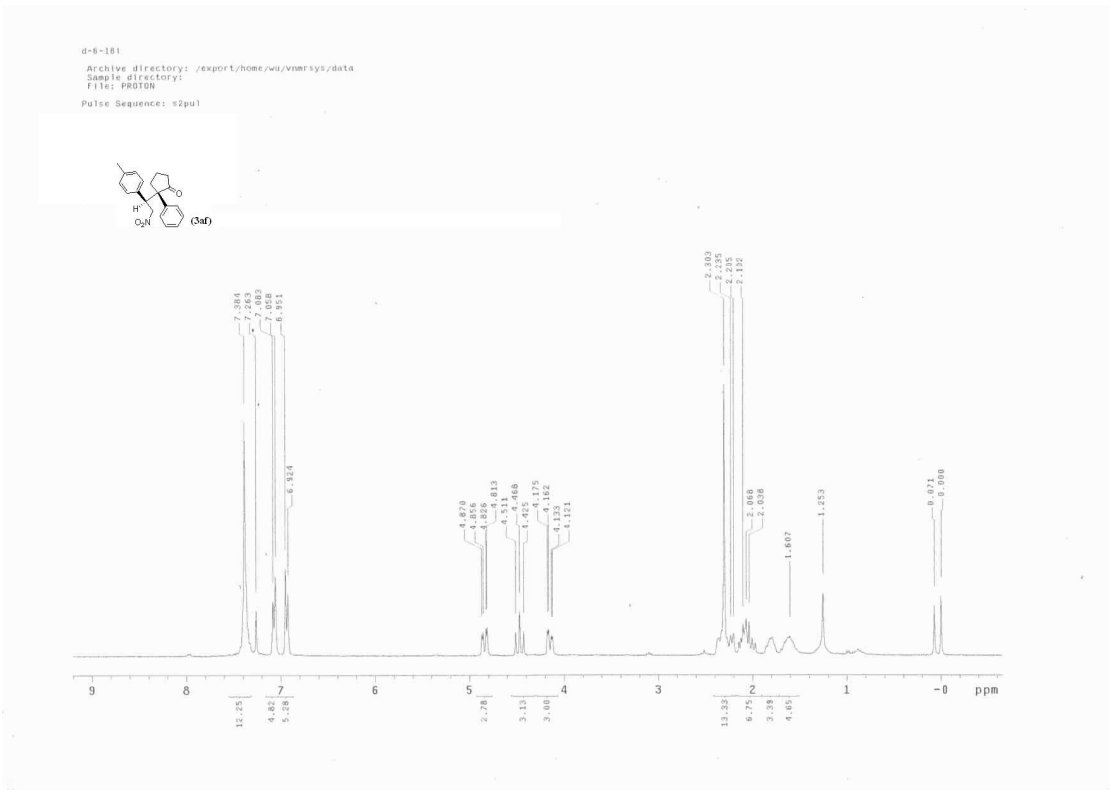
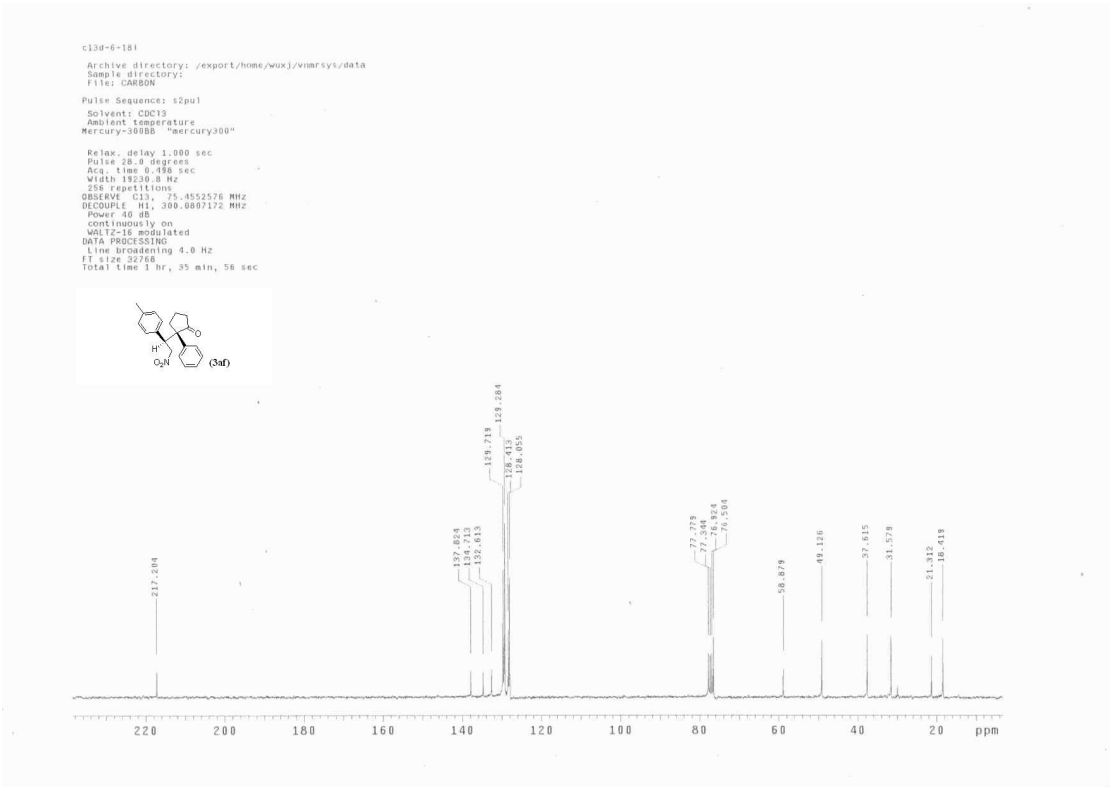
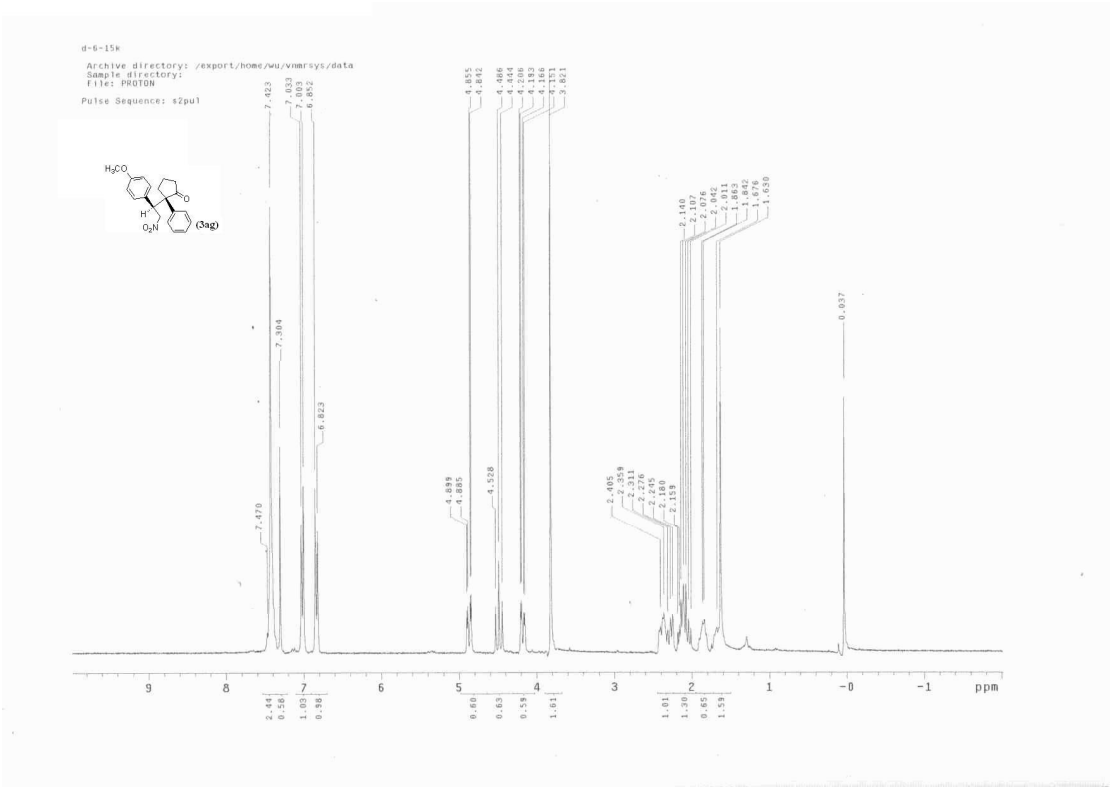
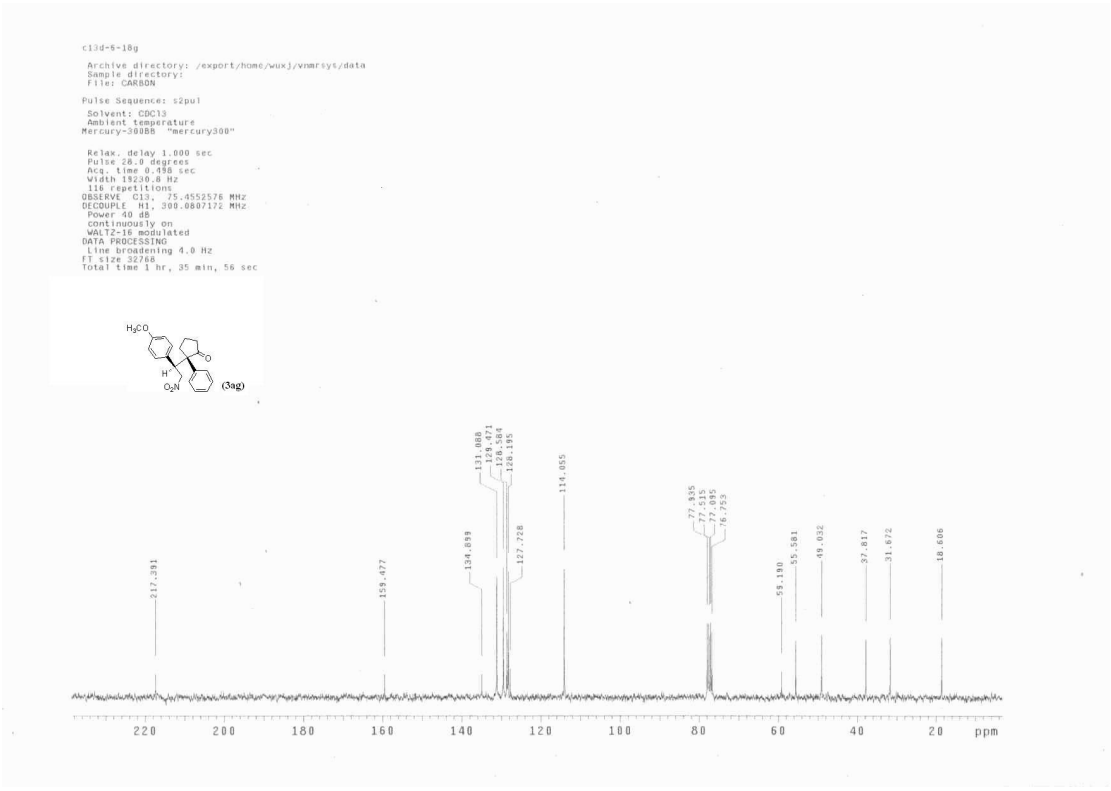
1.45
2.84

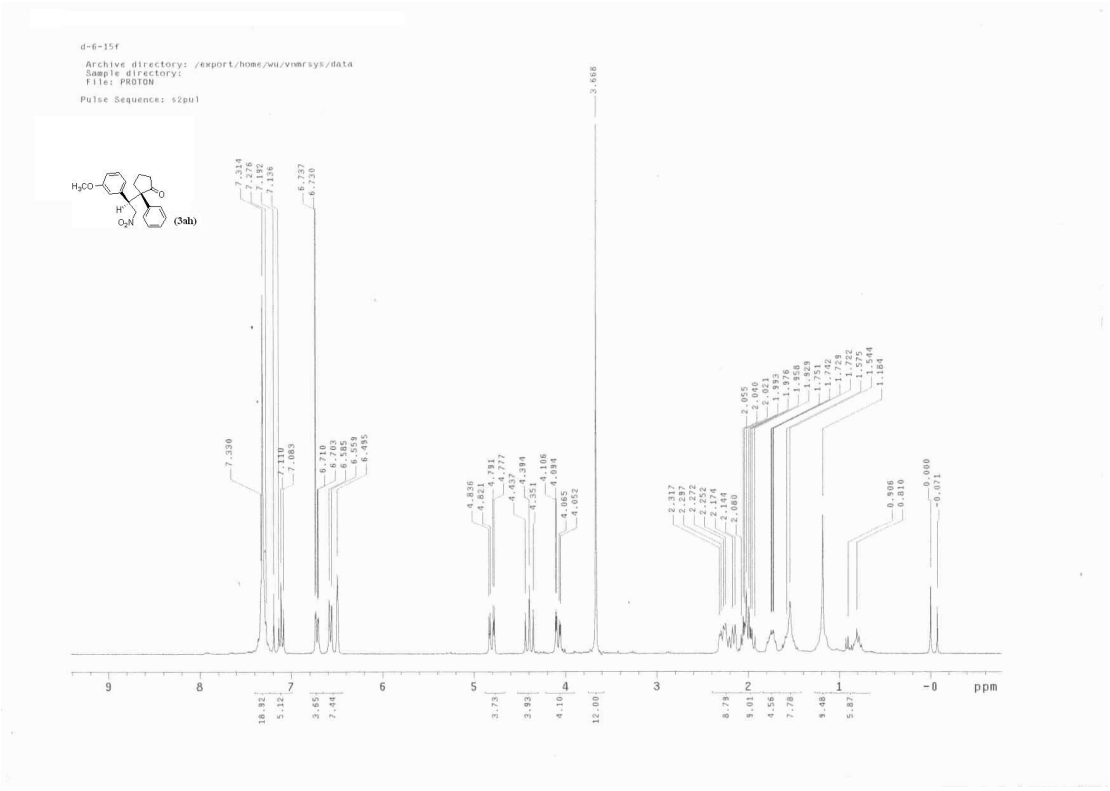

c13d-618h
Archive directory: /export/home/wuxj/vnmrsys/data
Sample directory:
File: CARBON
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
Mercury-300BB "mercury300"
Relax. delay 1.000 sec
Pulse 20.0 degrees
Acq. time 0.458 sec
Width 19230.8 Hz
136 repetitions
OBSERVE C13, 75.4552576 MHz
DECOUPLE H1, 300.0807172 MHz
Power 40 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 4.0 Hz
FT size 32768
Total time 1 hr, 35 min, 56 sec
(3ae)
216.924
134.215
131.508
131.415
129.400
128.599
127.977
115.642
115.362
77.748
77.313
76.878
76.457
58.848
40.846
37.459
31.595
18.310
220
200
180
160
140
120
100
80
60
40
20
ppm

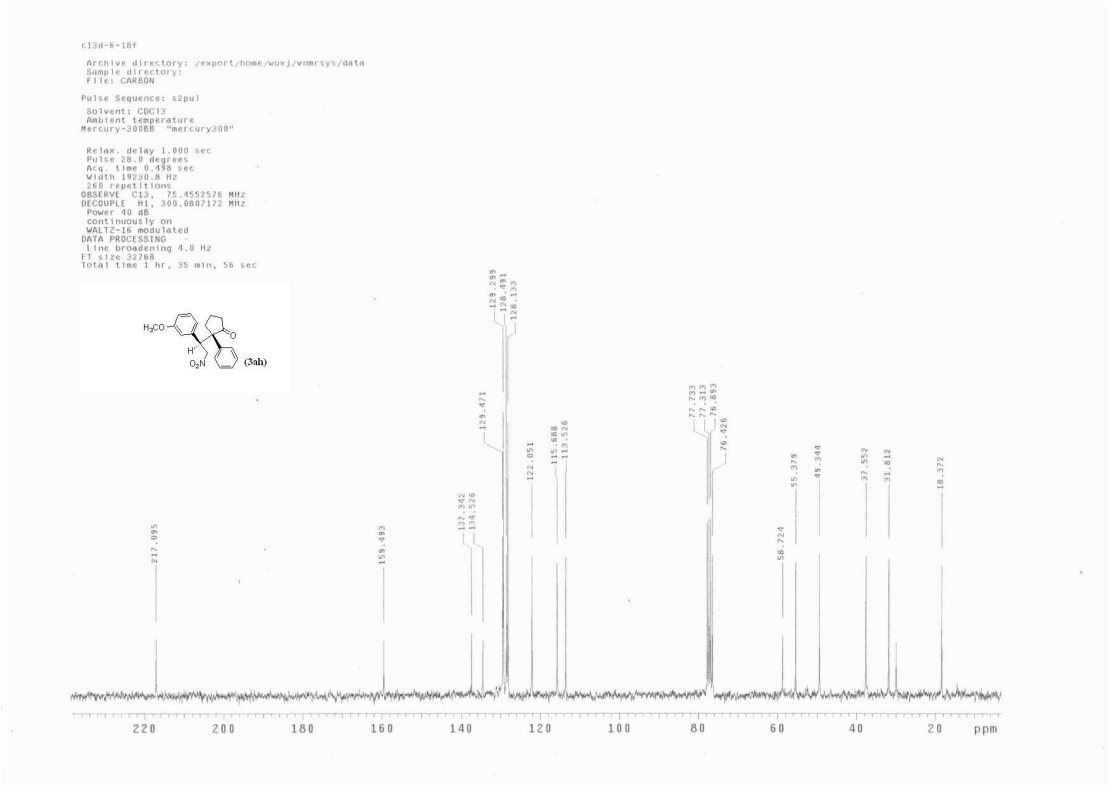

d-6-181
Archive directory: /export/home/wu/vnmrsys/data
Sample directory:
File: PROTON
Pulse Sequence: s2pul
(3af)
7.384
7.263
7.083
7.058
6.951
6.924
4.870
4.856
4.826
4.613
4.611
4.468
4.425
4.175
4.162
4.138
4.121
2.303
2.295
2.285
2.275
2.068
2.036
1.607
1.253
0.071
0.000
ppm

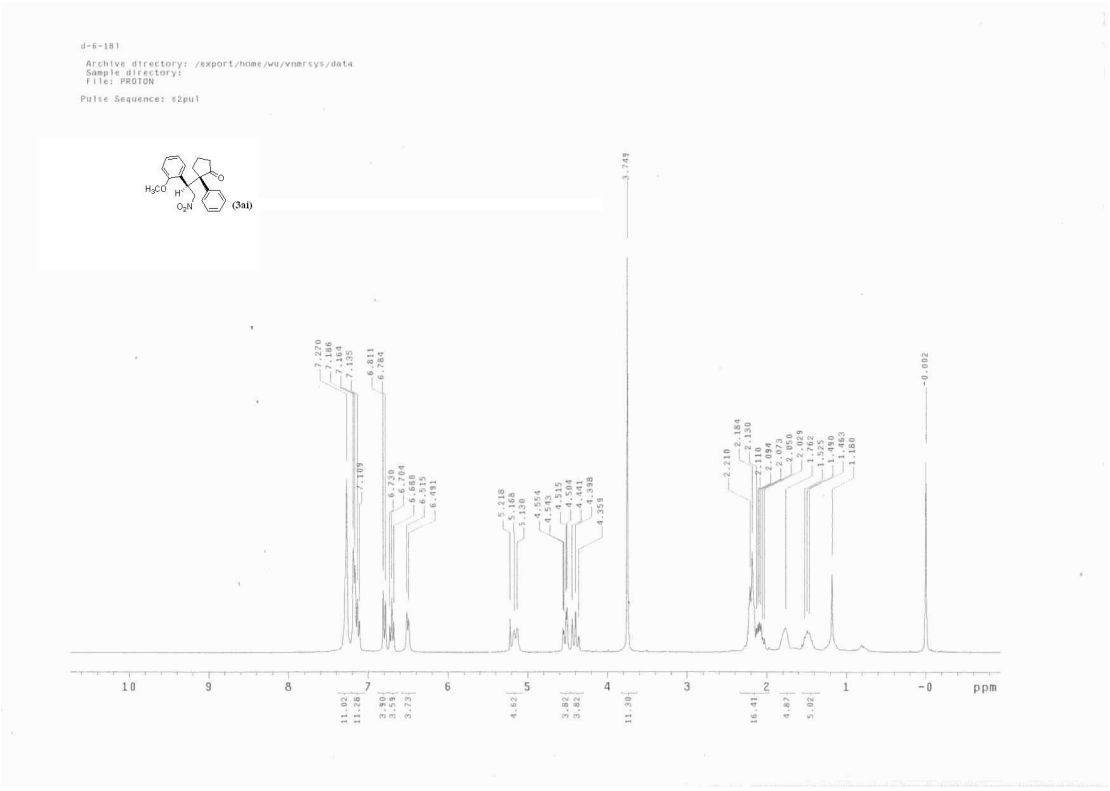

c13d-6-181
Archive directory: /export/home/wuxj/vnmrsys/data
Sample directory:
File: CARBON
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
Mercury-300BB "mercury300"
Relax. delay 1.000 sec
Pulse 20.0 degrees
Acq. time 0.498 sec
Width 19230.8 Hz
256 repetitions
OBSERVE C13, 75.4552576 MHz
DECOUPLE H1, 300.0807172 MHz
Power 40 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 4.0 Hz
FT size 32768
Total time 1 hr, 35 min, 56 sec
(3af)
217.804
137.824
134.713
132.613
129.719
129.284
128.413
128.055
77.779
77.344
77.024
76.504
58.879
49.126
37.615
31.579
21.312
18.418
ppm

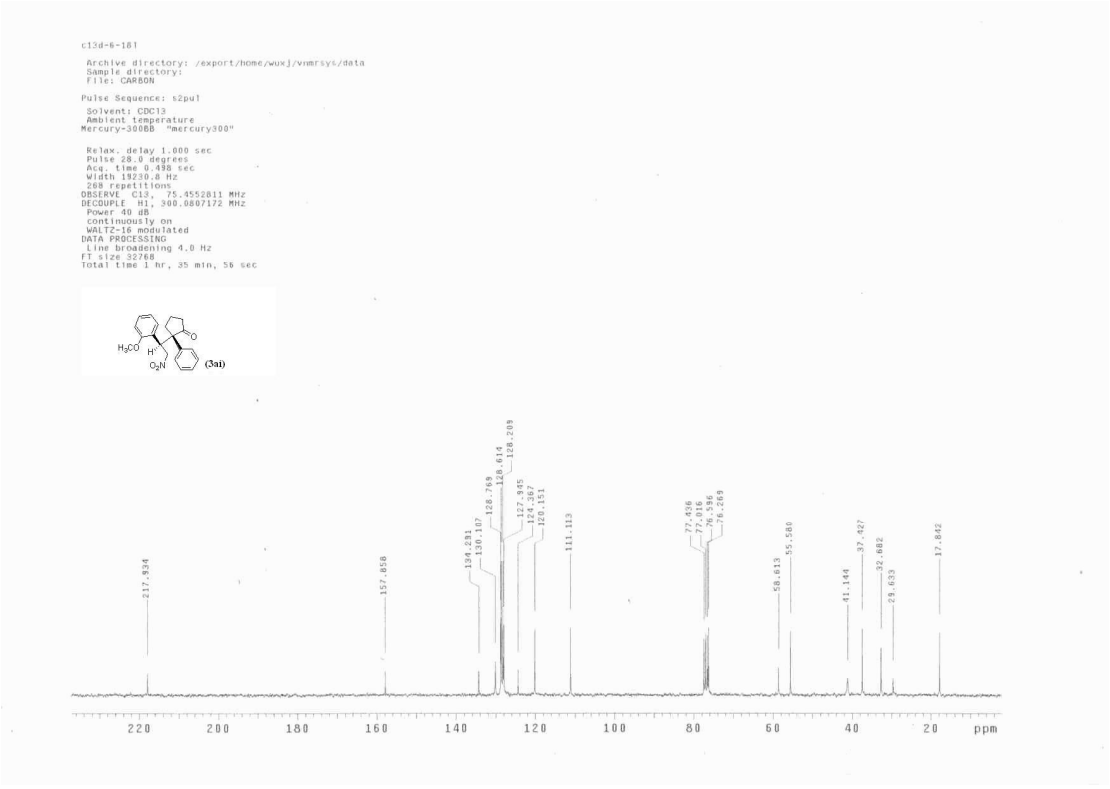

d-6-15k
Archive directory: /export/home/wu/vnmrsys/data
Sample directory:
File: PROTON
Pulse Sequence: s2pul
(3ag)
ppm

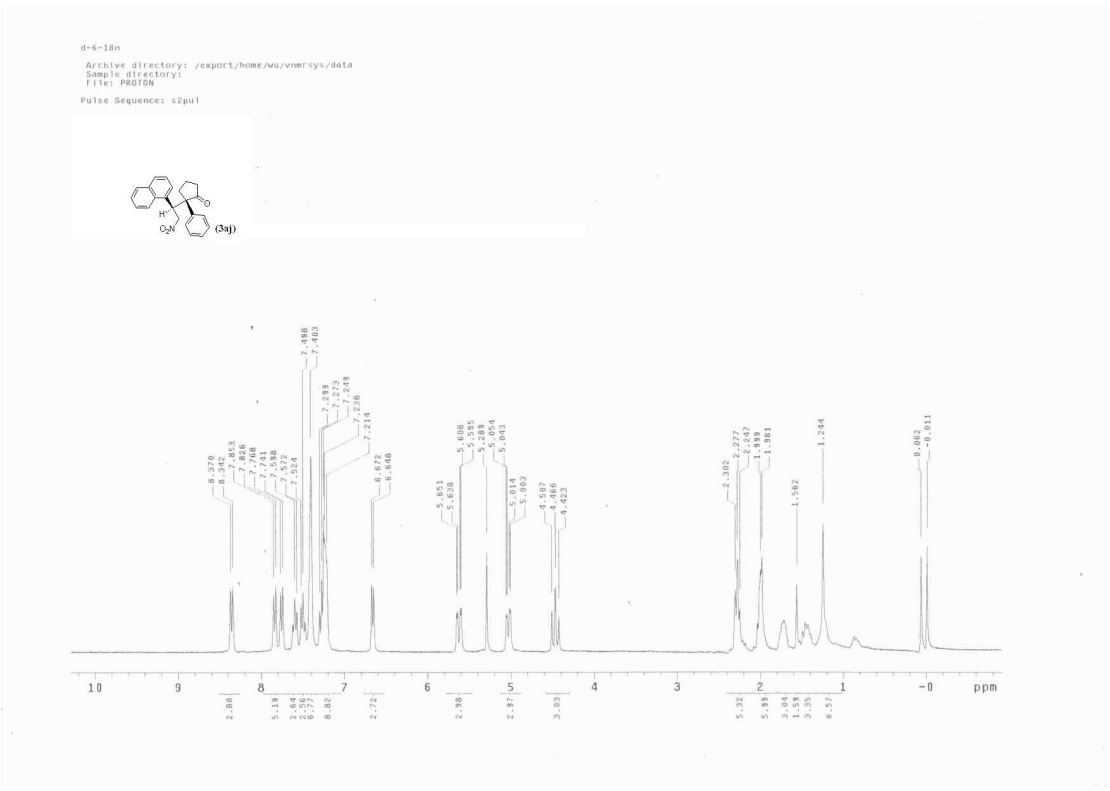

c13d-6-18g
Archive directory: /export/home/wuxj/vnmrsys/data
Sample directory:
File: CARBON
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
Mercury-300BB "mercury300"
Relax. delay 1.000 sec
Pulse 26.0 degrees
Acq. time 0.498 sec
Width 19230.8 Hz
116 repetitions
OBSERVE C13, 75.4552576 MHz
DECOUPLE H1, 300.0807172 MHz
Power 40 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 4.0 Hz
FT size 32768
Total time 1 hr, 35 min, 56 sec
(3ag)
ppm

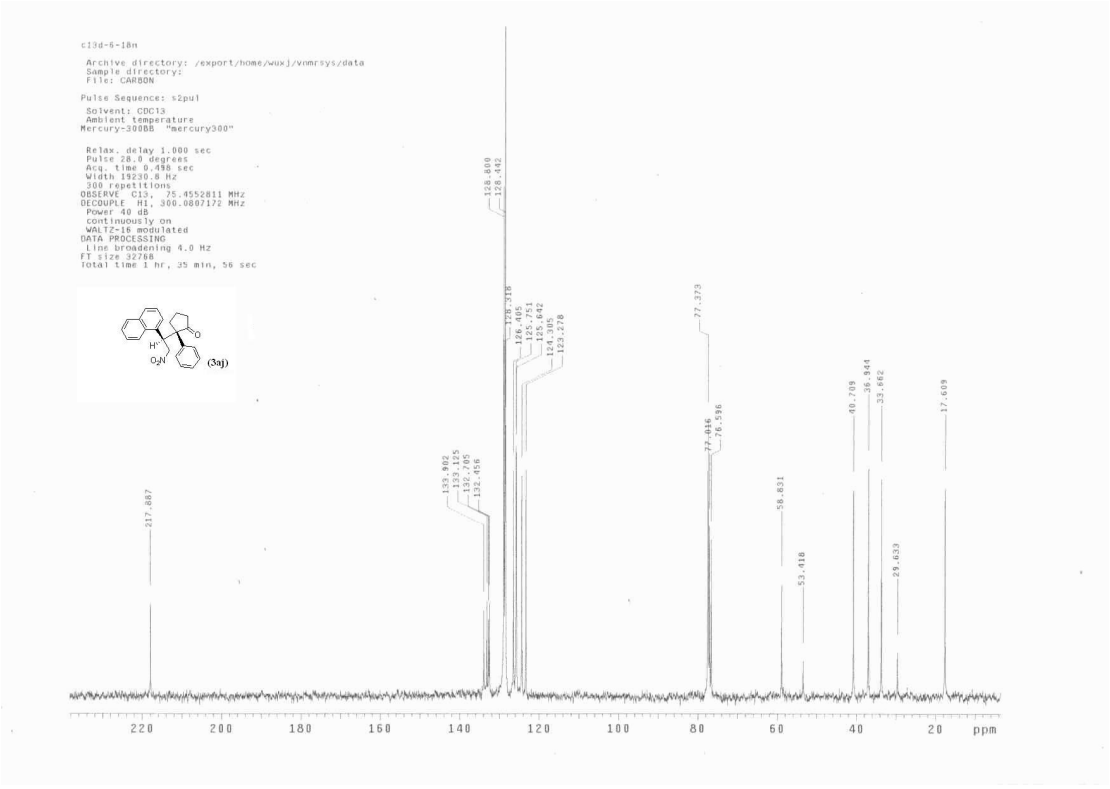

d-6-15f
Archive directory: /export/home/wu/vnmrsys/data
Sample directory:
File: PROTON
Pulse Sequence: s2pul
(3ah)
ppm

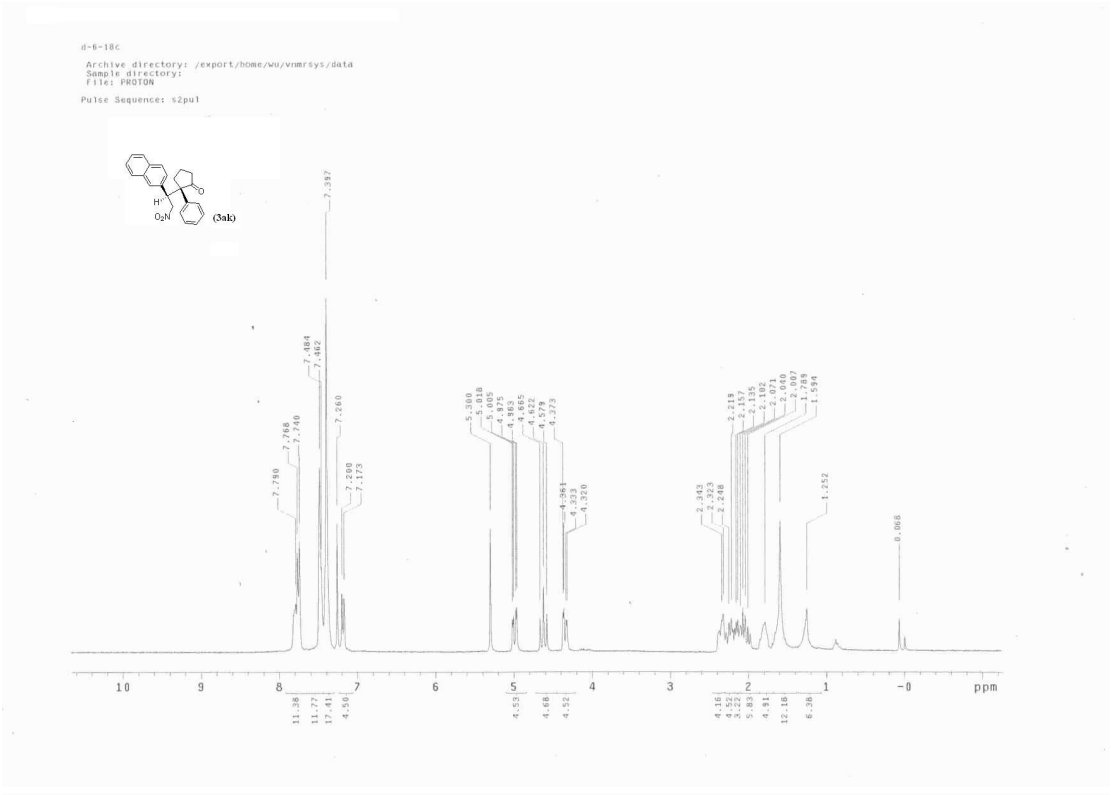

c13d-6-18f
Archive directory: /export/home/wuxj/vnmrsys/data
Sample directory:
File: CARBON
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
Mercury-300BB "mercury300"
Relax. delay 1.000 sec
Pulse 28.0 degrees
Acq. time 0.498 sec
Width 19230.8 Hz
260 repetitions
OBSERVE C13, 75.4552576 MHz
DECOUPLE H1, 300.0807172 MHz
Power 40 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 4.0 Hz
FT size 32768
Total time 1 hr, 35 min, 56 sec
(3ah)
ppm

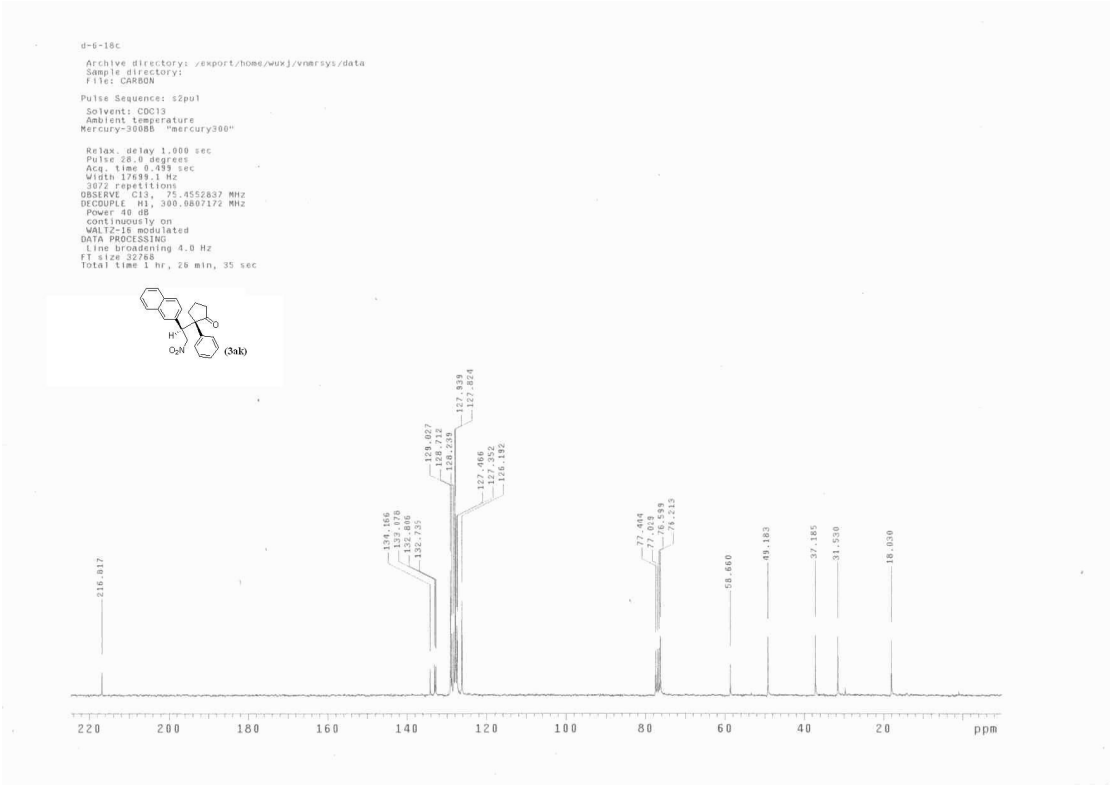

d-6-181
Archive directory: /export/home/wu/vnmrsys/data
Sample directory:
File: PROTON
Pulse Sequence: s2pul
(3ai)
ppm

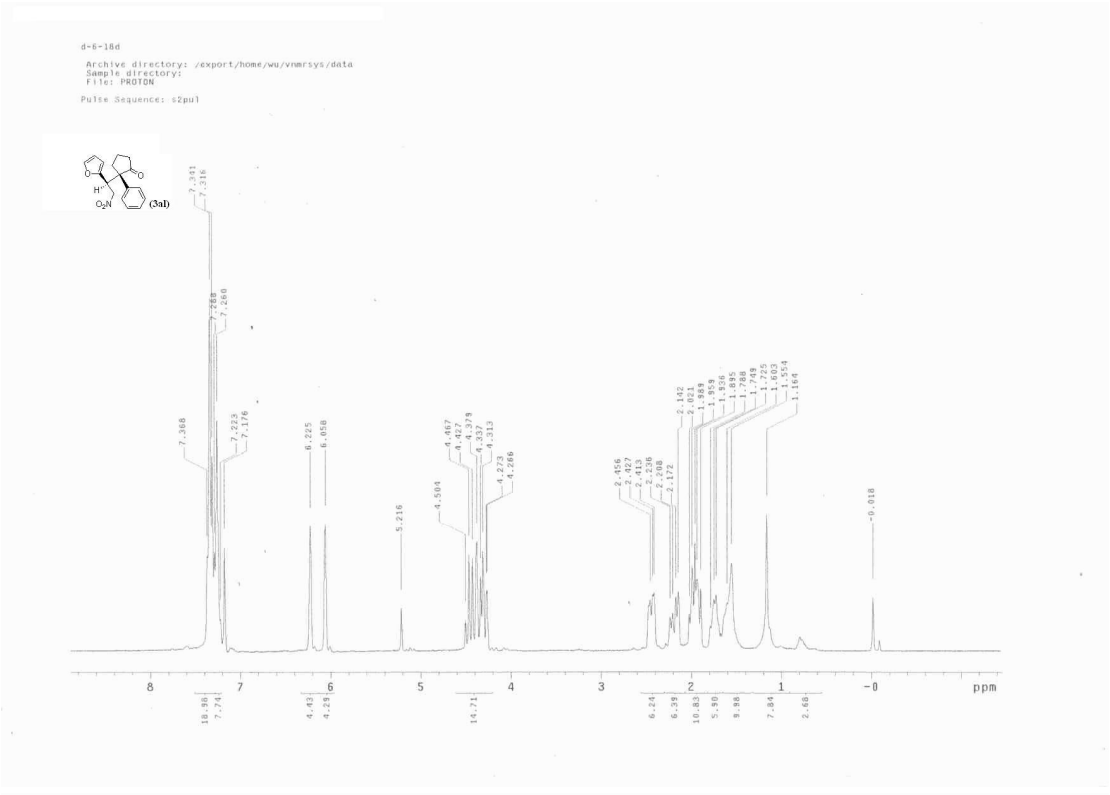

c13d-6-181
Archive directory: /export/home/wuxj/vnmrsys/data
Sample directory:
File: CARBON
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
Mercury-300BB "mercury300"
Relax. delay 1.000 sec
Pulse 28.0 degrees
Acq. time 0.498 sec
Width 19230.8 Hz
268 repetitions
OBSERVE C13, 75.4552811 MHz
DECOUPLE H1, 300.0807172 MHz
Power 40 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 4.0 Hz
FT size 32768
Total time 1 hr, 35 min, 56 sec
(3ai)
ppm

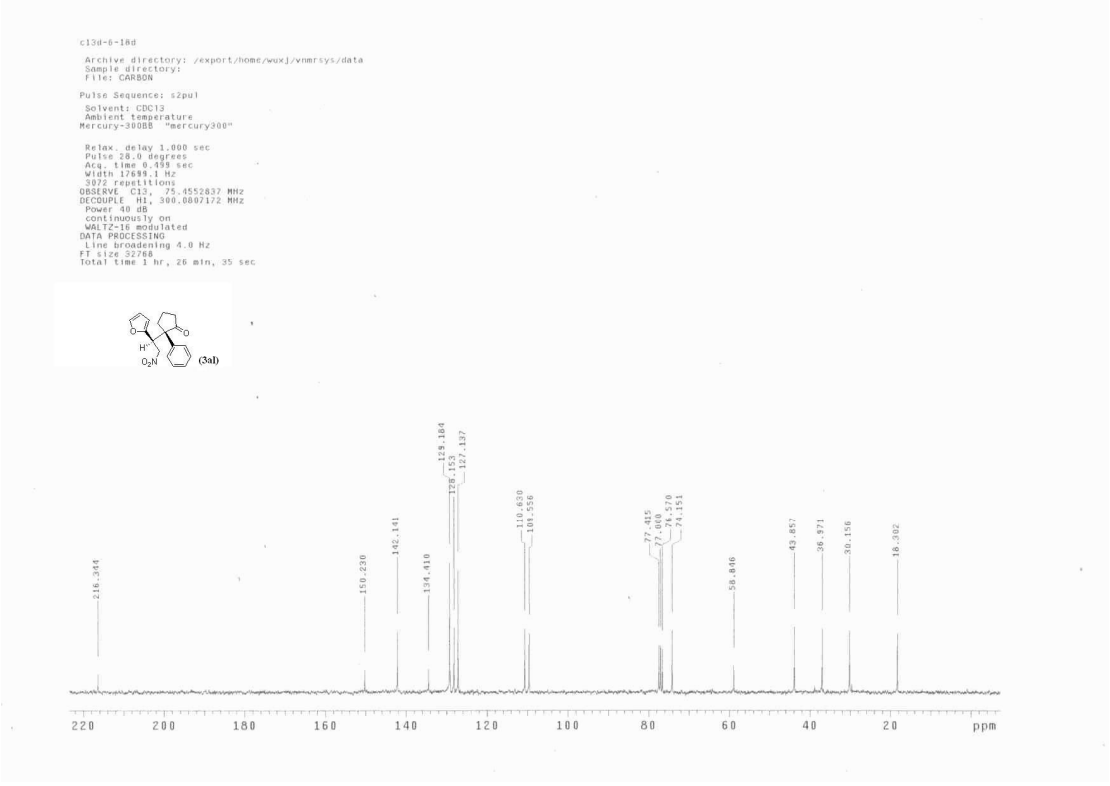

d-6-18n
Archive directory: /export/home/wu/vnmrsys/data
Sample directory:
File: PROTON
Pulse Sequence: s2pul
(3aj)
8.370
8.362
7.853
7.826
7.768
7.744
7.588
7.572
7.524
7.488
7.403
7.299
7.273
7.249
7.236
7.214
6.672
6.648
5.651
5.638
5.608
5.595
5.289
5.054
5.043
5.014
5.003
4.507
4.466
4.423
2.302
2.277
2.247
1.998
1.981
1.562
1.244
0.062
0.011
ppm

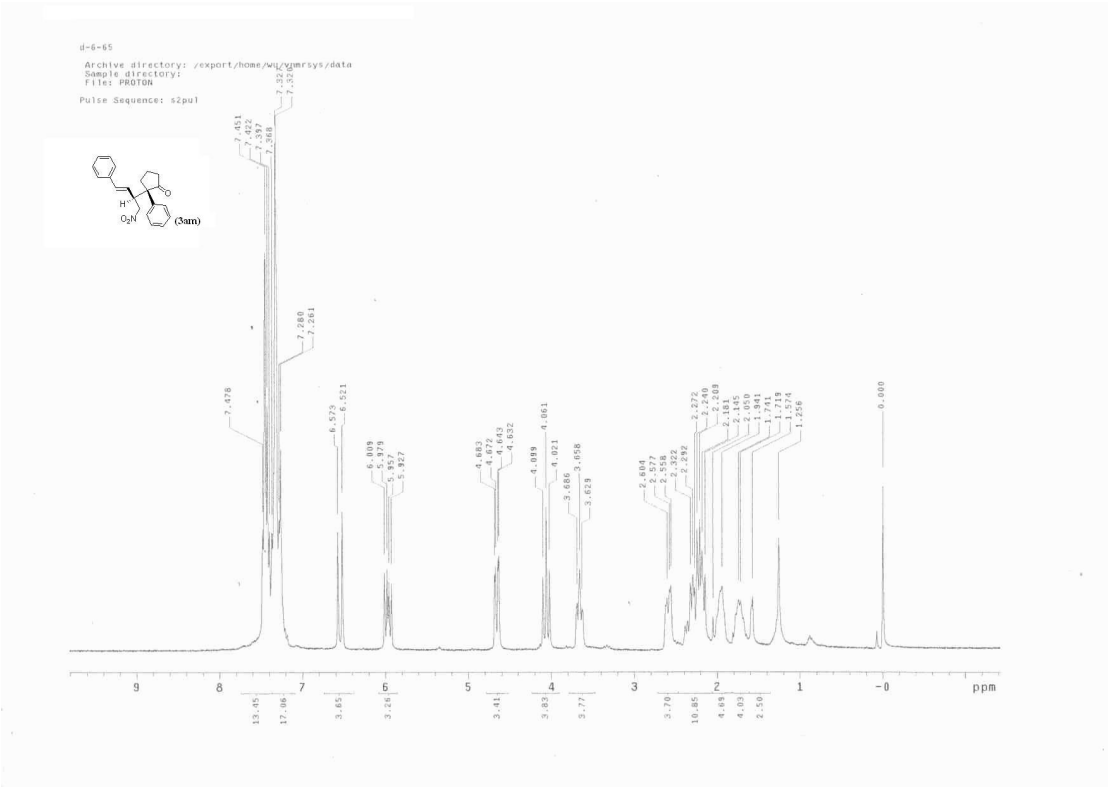

c13d-6-18n
Archive directory: /export/home/wuxj/vnmrsys/data
Sample directory:
File: CARBON
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
Mercury-300BB "mercury300"
Relax. delay 1.000 sec
Pulse 28.0 degrees
Acq. time 0.498 sec
Width 19230.8 Hz
300 repetitions
OBSERVE C13, 75.4552811 MHz
DECOUPLE H1, 300.0807172 MHz
Power 40 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 4.0 Hz
FT size 32768
Total time 1 hr, 35 min, 56 sec
(3aj)
217.867
133.902
133.125
132.705
132.456
128.800
128.442
128.318
126.405
125.751
125.642
124.305
123.278
77.373
77.036
76.596
58.831
53.418
40.709
36.844
33.662
29.633
17.609
ppm

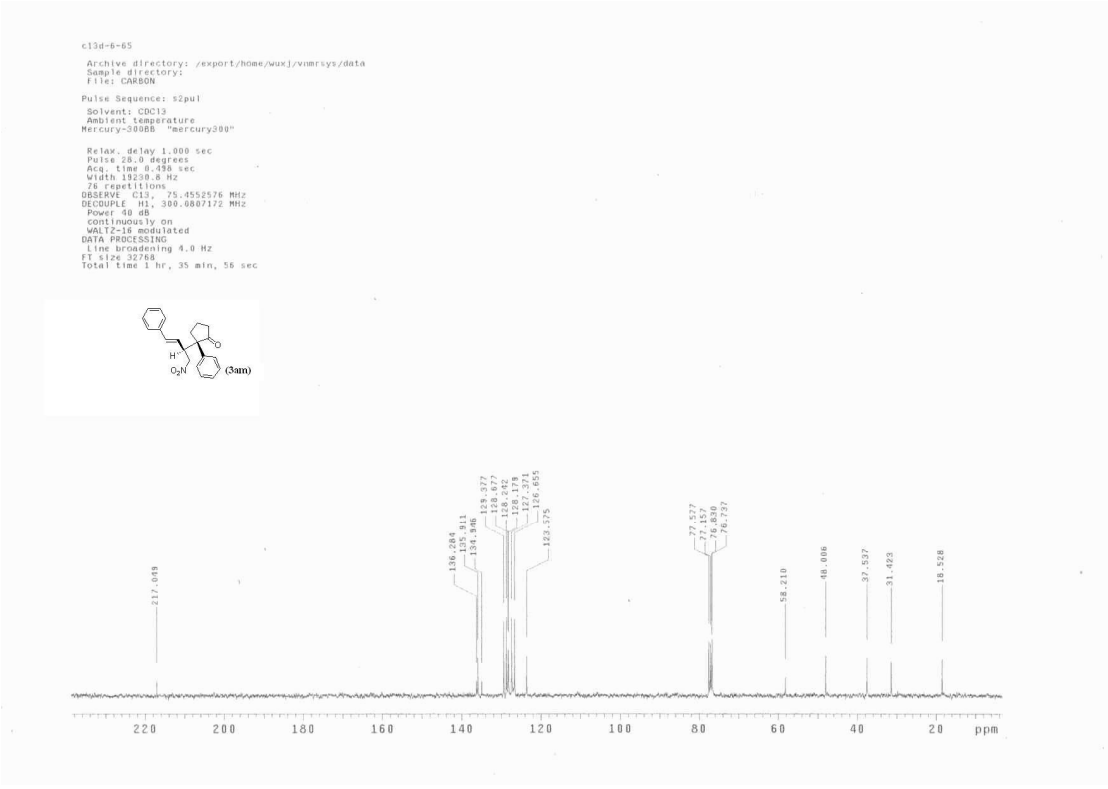

d-6-18c
Archive directory: /export/home/wu/vnmrsys/data
Sample directory:
File: PROTON
Pulse Sequence: s2pul
(3ak)
ppm

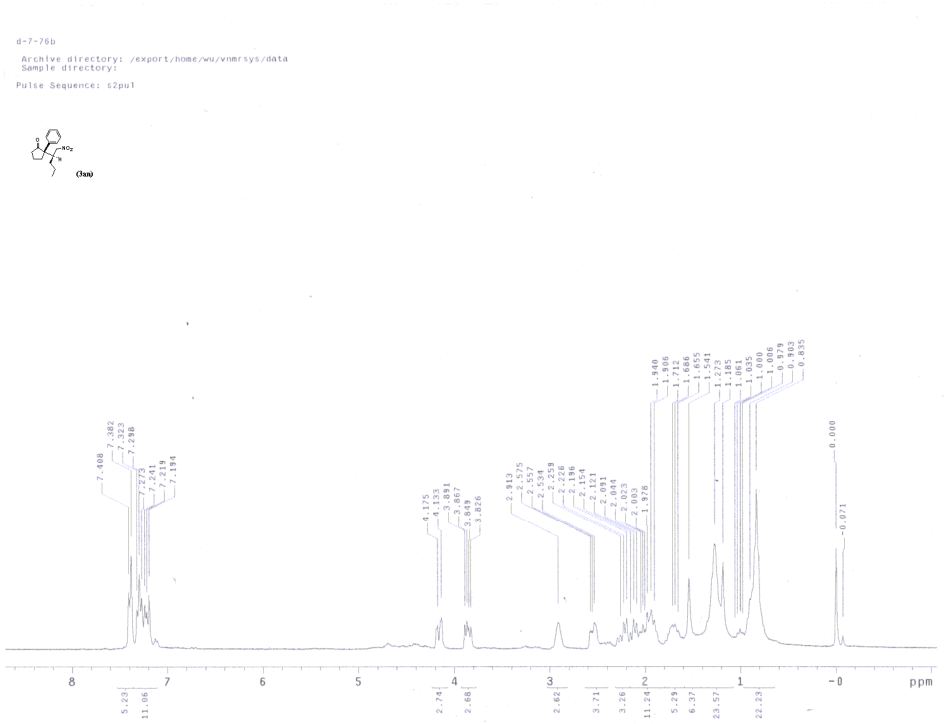

d-6-18c
Archive directory: /export/home/wuxj/vnmrsys/data
Sample directory:
File: CARBON
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
Mercury-300BB "mercury300"
Relax. delay 1.000 sec
Pulse 28.0 degrees
Acq. time 0.499 sec
Width 17699.1 Hz
3072 repetitions
OBSERVE C13, 75.4552837 MHz
DECOUPLE H1, 300.0807172 MHz
Power 40 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 4.0 Hz
FT size 32768
Total time 1 hr, 26 min, 35 sec
(3ak)
ppm

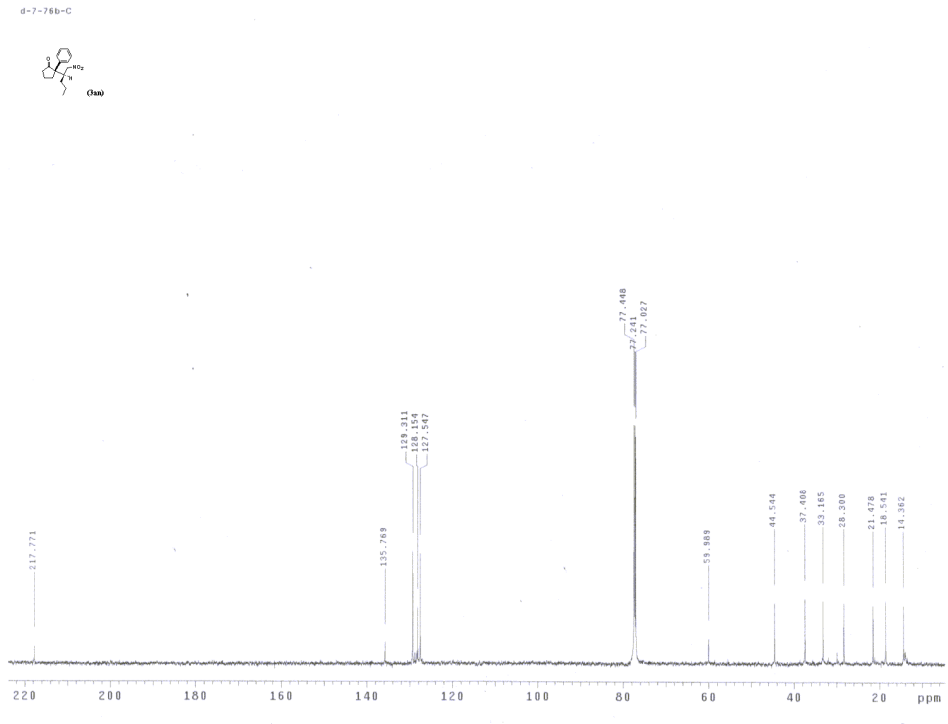

d-6-18d
Archive directory: /export/home/wu/vnmrsys/data
Sample directory:
File: PROTON
Pulse Sequence: s2pul
(3al)
ppm

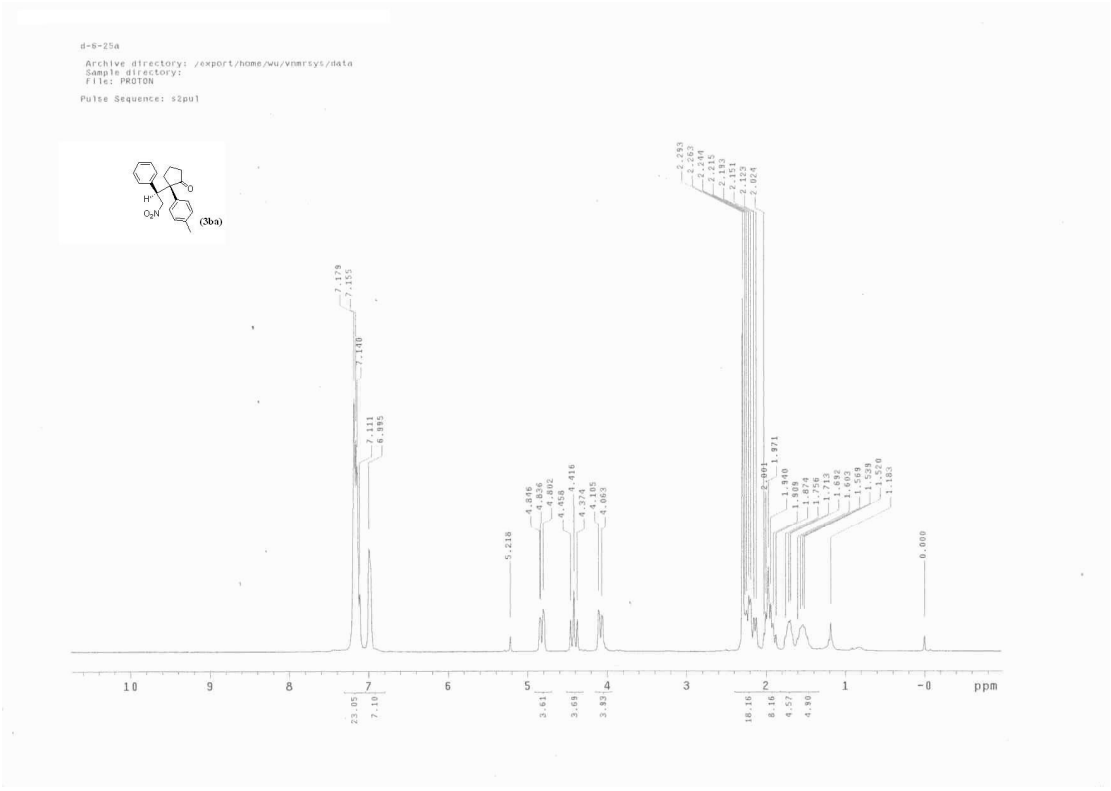

c13d-6-18d
Archive directory: /export/home/wuxj/vnmrsys/data
Sample directory:
File: CARBON
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
Mercury-300BB "mercury300"
Relax. delay 1.000 sec
Pulse 28.0 degrees
Acq. time 0.499 sec
Width 17699.1 Hz
3072 repetitions
OBSERVE C13, 75.4552837 MHz
DECOUPLE H1, 300.0807172 MHz
Power 40 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 4.0 Hz
FT size 32768
Total time 1 hr, 26 min, 35 sec
(3al)
ppm

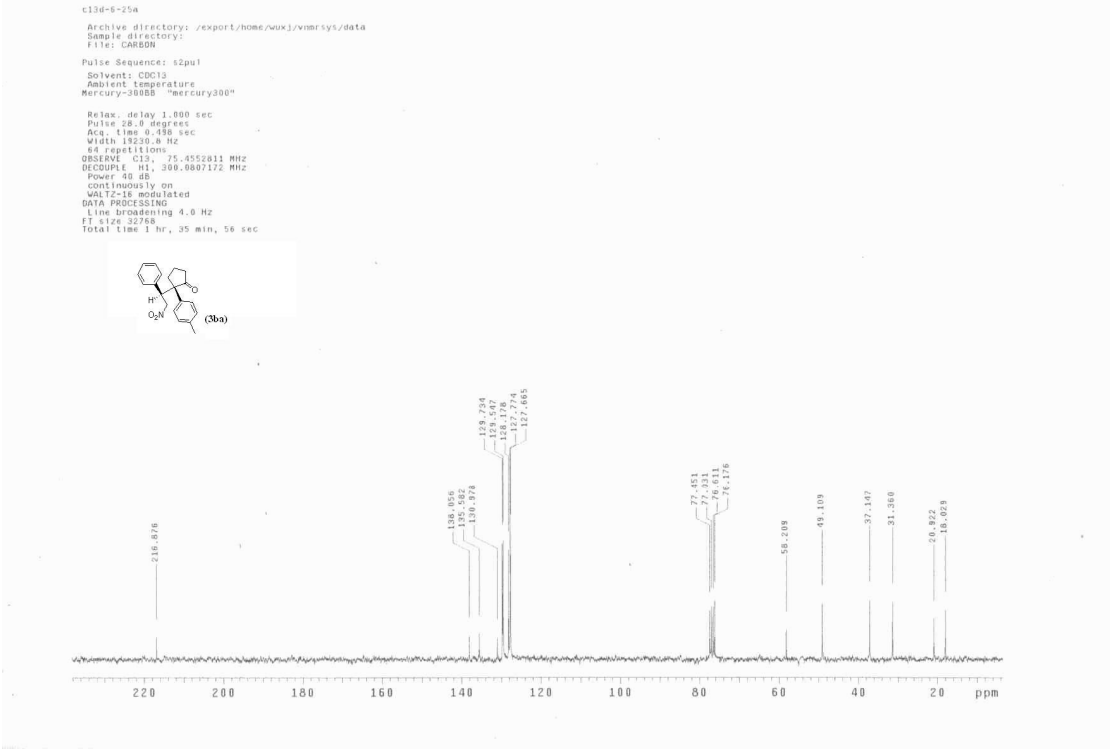

d-6-65
Archive directory: /export/home/wu/vnmrsys/data
Sample directory:
File: PROTON
Pulse Sequence: s2pul
(3am)
ppm

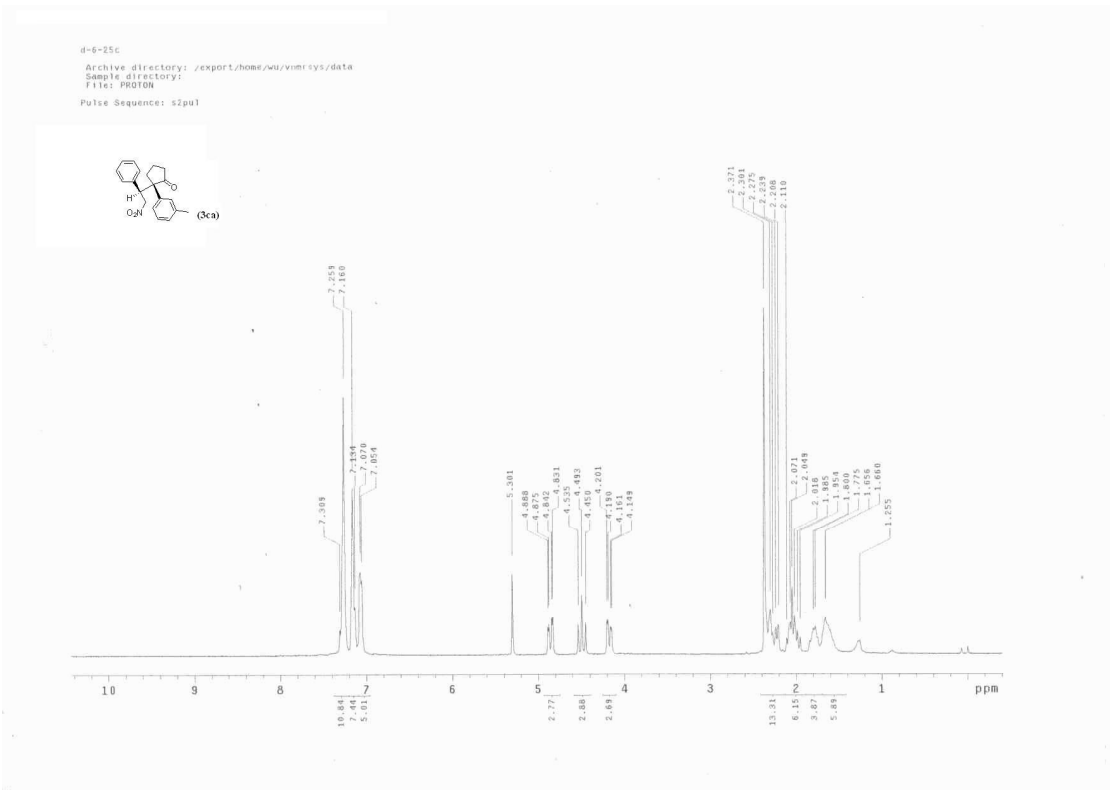

c13d-6-65
Archive directory: /export/home/wuxj/vnmrsys/data
Sample directory:
File: CARBON
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
Mercury-300BB "mercury300"
Relax. delay 1.000 sec
Pulse 28.0 degrees
Acq. time 0.498 sec
Width 19230.8 Hz
76 repetitions
OBSERVE C13, 75.4552576 MHz
DECOUPLE H1, 300.0807172 MHz
Power 40 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 4.0 Hz
FT size 32768
Total time 1 hr, 35 min, 56 sec
(3am)
ppm

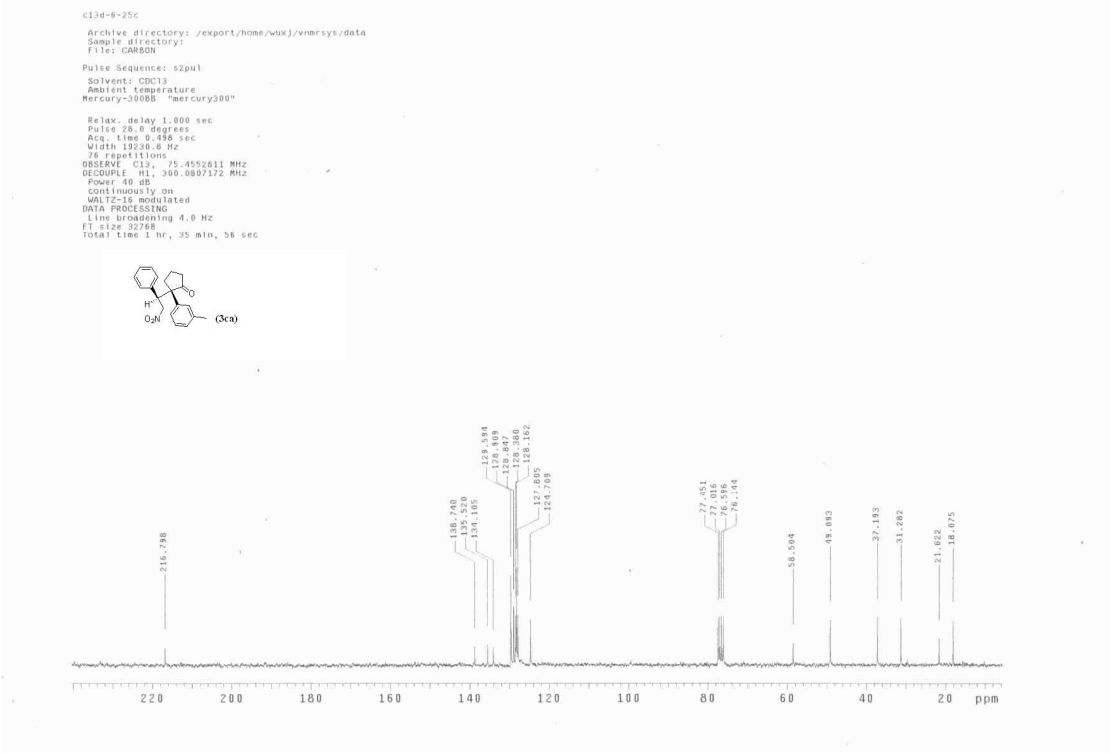

d-7-76b
Archive directory: /export/home/wu/vnmrsys/data
Sample directory:
Pulse Sequence: s2pul
(3an)
ppm

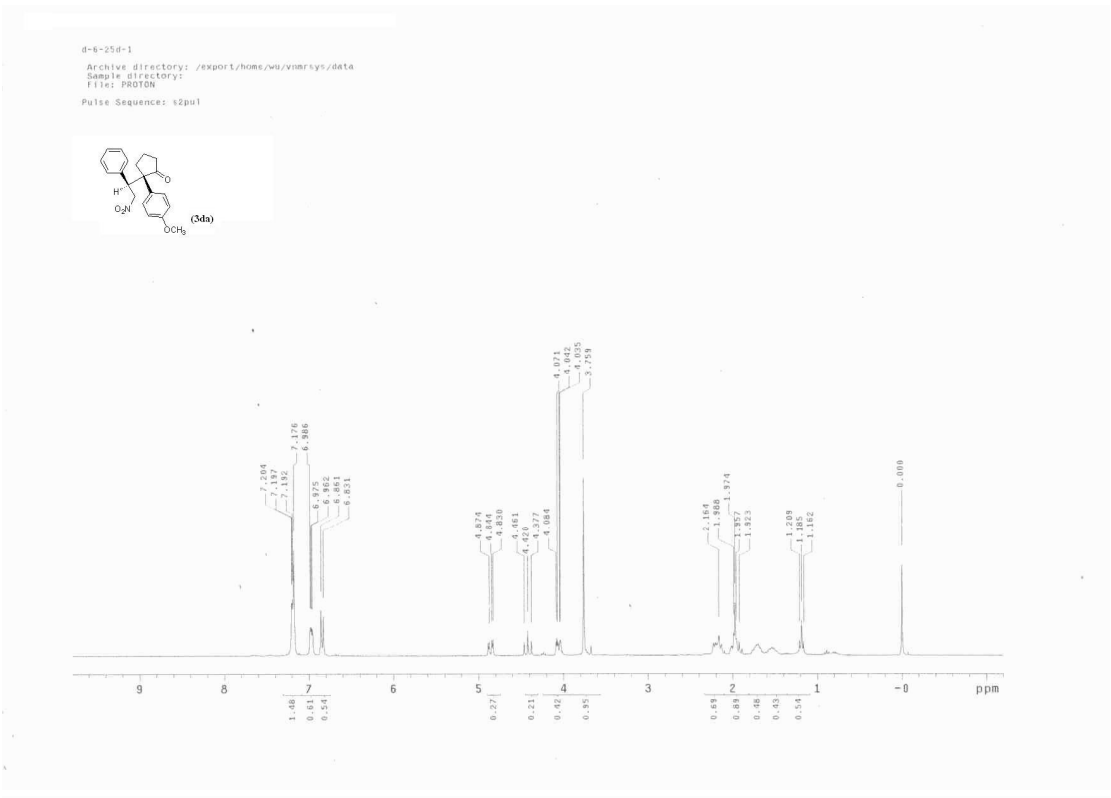

d-7-76b-C
(3an)
ppm

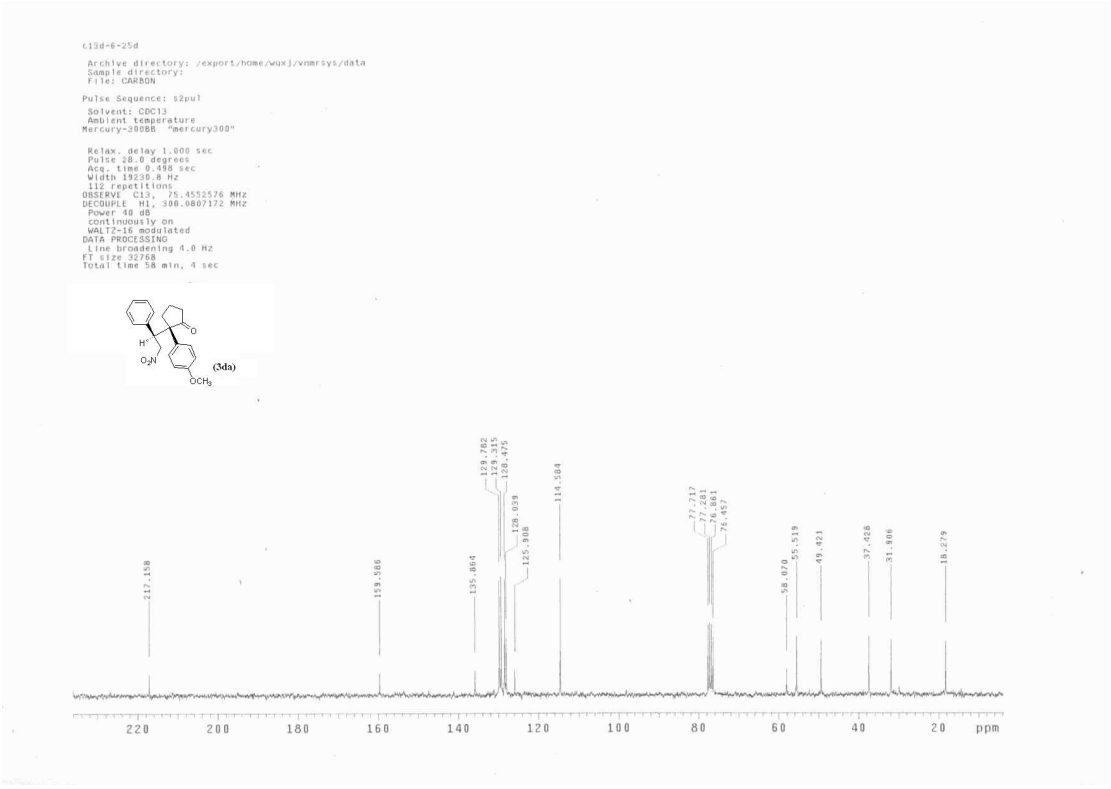

d-6-25a
Archive directory: /export/home/wu/vnmrsys/data
Sample directory:
File: PROTON
Pulse Sequence: s2pul
(3ba)
ppm

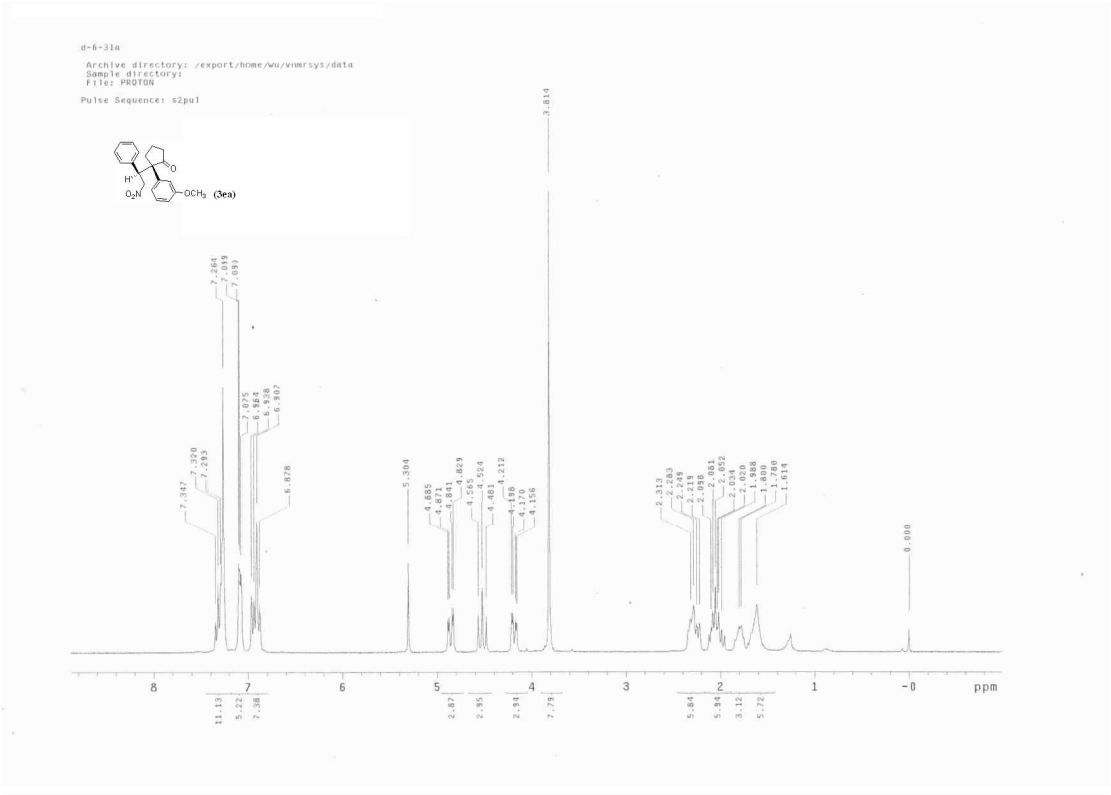

c13d-6-25a
Archive directory: /export/home/wuxj/vnmrsys/data
Sample directory:
File: CARBON
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
Mercury-300BB "mercury300"
Relax. delay 1.000 sec
Pulse 28.0 degrees
Acq. time 0.498 sec
Width 19230.8 Hz
64 repetitions
OBSERVE C13, 75.4552811 MHz
DECOUPLE H1, 300.0807172 MHz
Power 40 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 4.0 Hz
FT size 32768
Total time 1 hr, 35 min, 56 sec
(3ba)
ppm

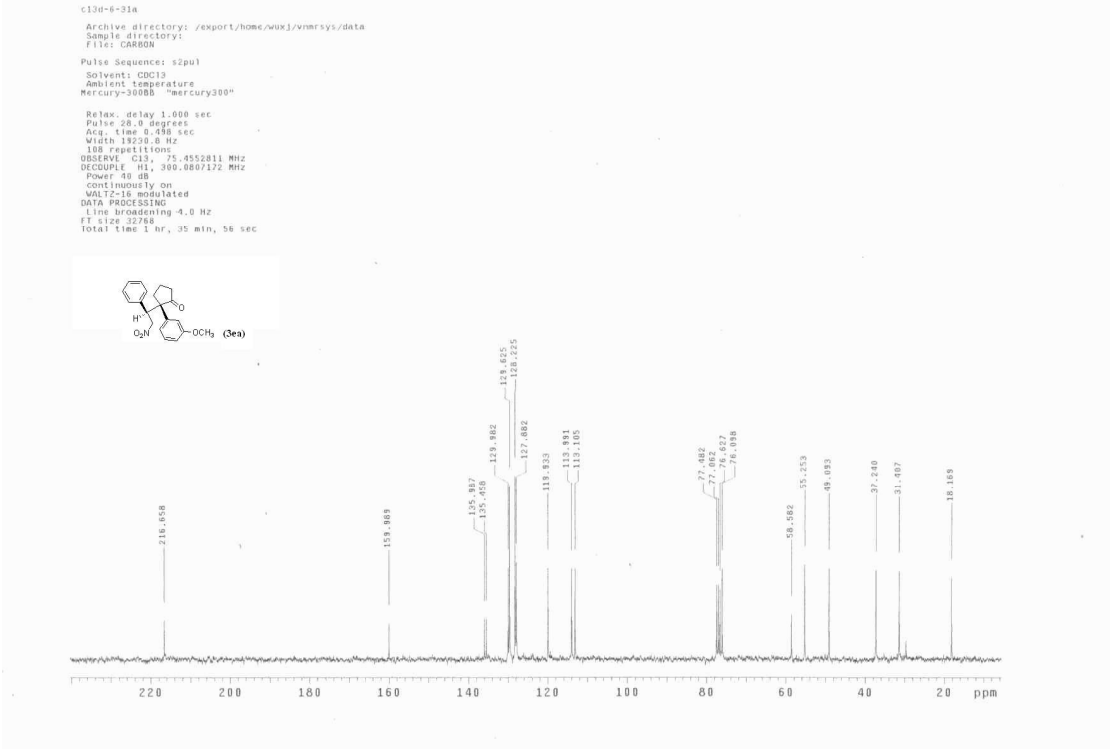

d-6-25c
Archive directory: /export/home/wu/vnmrsys/data
Sample directory:
File: PROTON
Pulse Sequence: s2pul
(3ca)
ppm

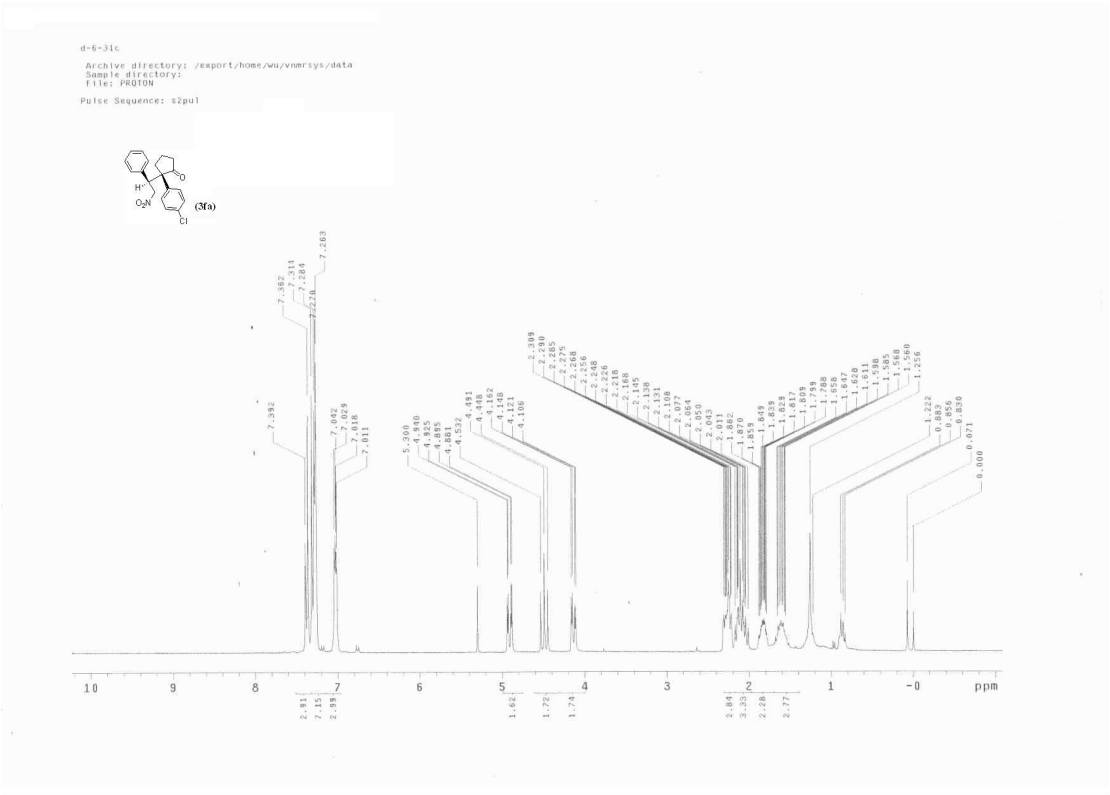

c13d-6-25c
Archive directory: /export/home/wuxj/vnmrsys/data
Sample directory:
File: CARBON
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
Mercury-300BB "mercury300"
Relax. delay 1.000 sec
Pulse 28.0 degrees
Acq. time 0.498 sec
Width 19230.8 Hz
76 repetitions
OBSERVE C13, 75.4552811 MHz
DECOUPLE H1, 300.0887172 MHz
Power 40 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 4.0 Hz
FT size 32768
Total time 1 hr, 35 min, 56 sec
(3ca)
ppm

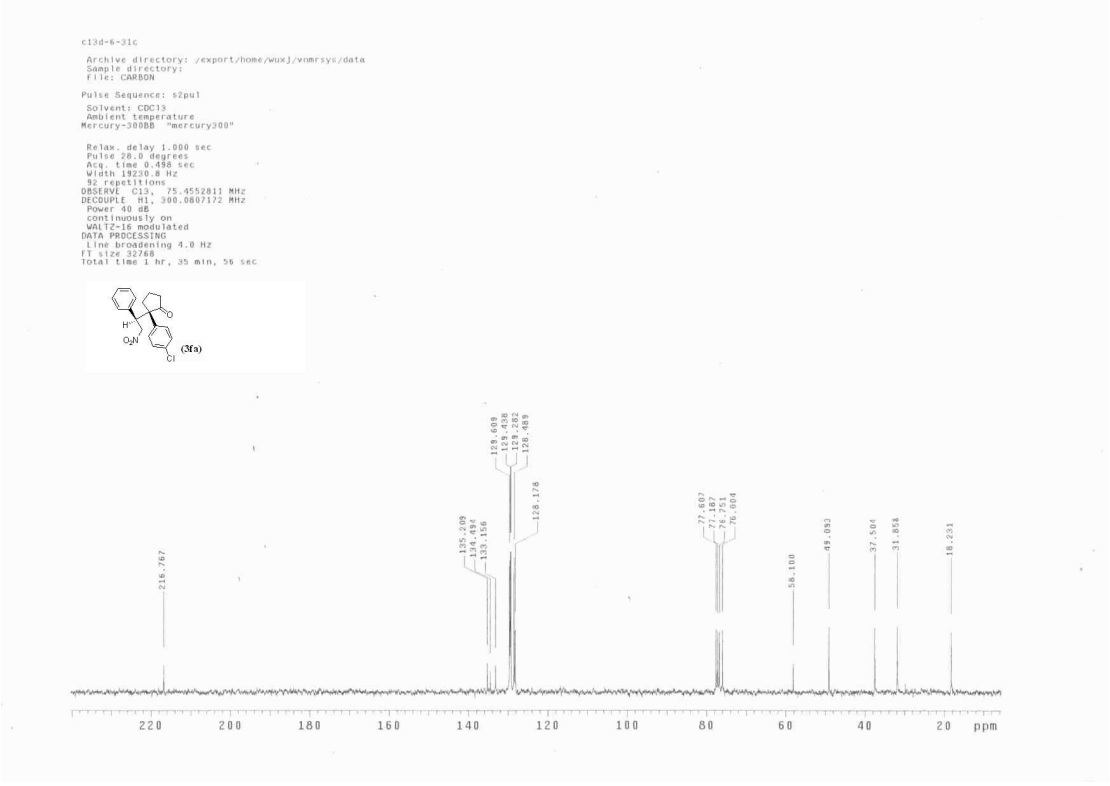

d-6-25d-1
Archive directory: /export/home/wu/vnmrsys/data
Sample directory:
File: PROTON
Pulse Sequence: s2pul
(3da)
ppm

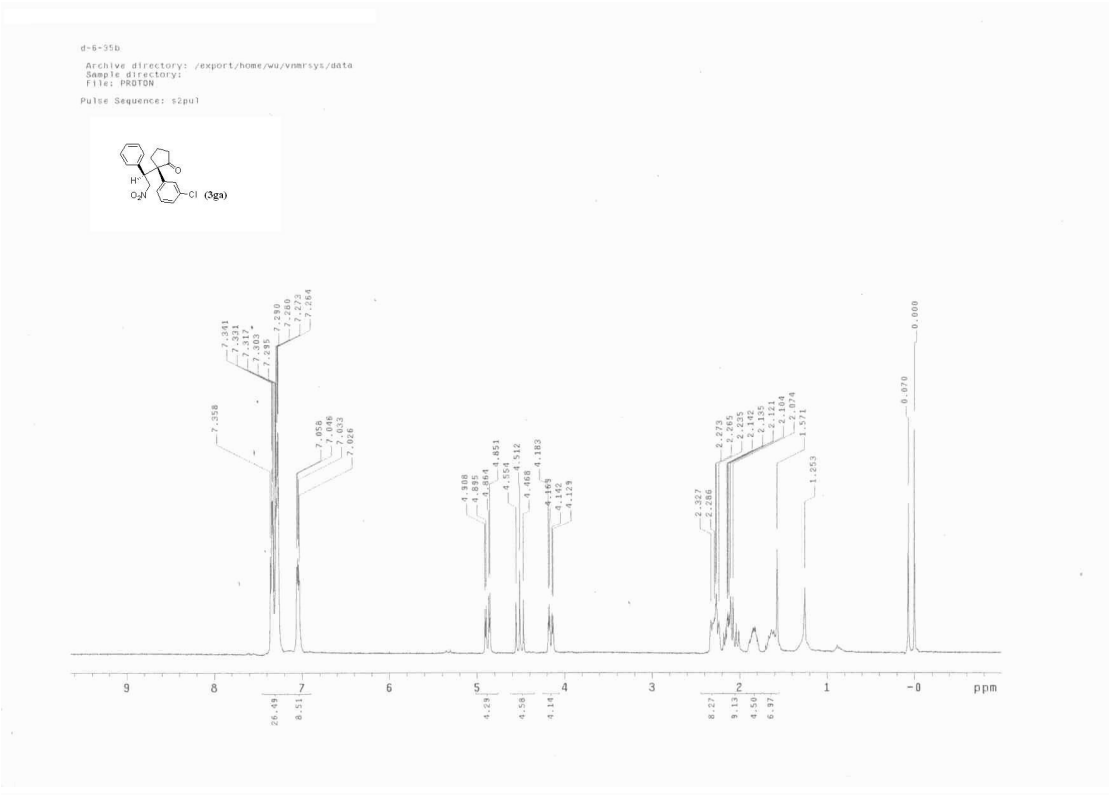

c13d-6-25d
Archive directory: /export/home/wuxj/vnmrsys/data
Sample directory:
File: CARBON
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
Mercury-300BB "mercury300"
Relax. delay 1.000 sec
Pulse 28.0 degrees
Acq. time 0.498 sec
Width 19230.8 Hz
112 repetitions
OBSERVE C13, 75.4552576 MHz
DECOUPLE H1, 300.0807172 MHz
Power 40 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 4.0 Hz
FT size 32768
Total time 58 min, 4 sec
(3da)
ppm

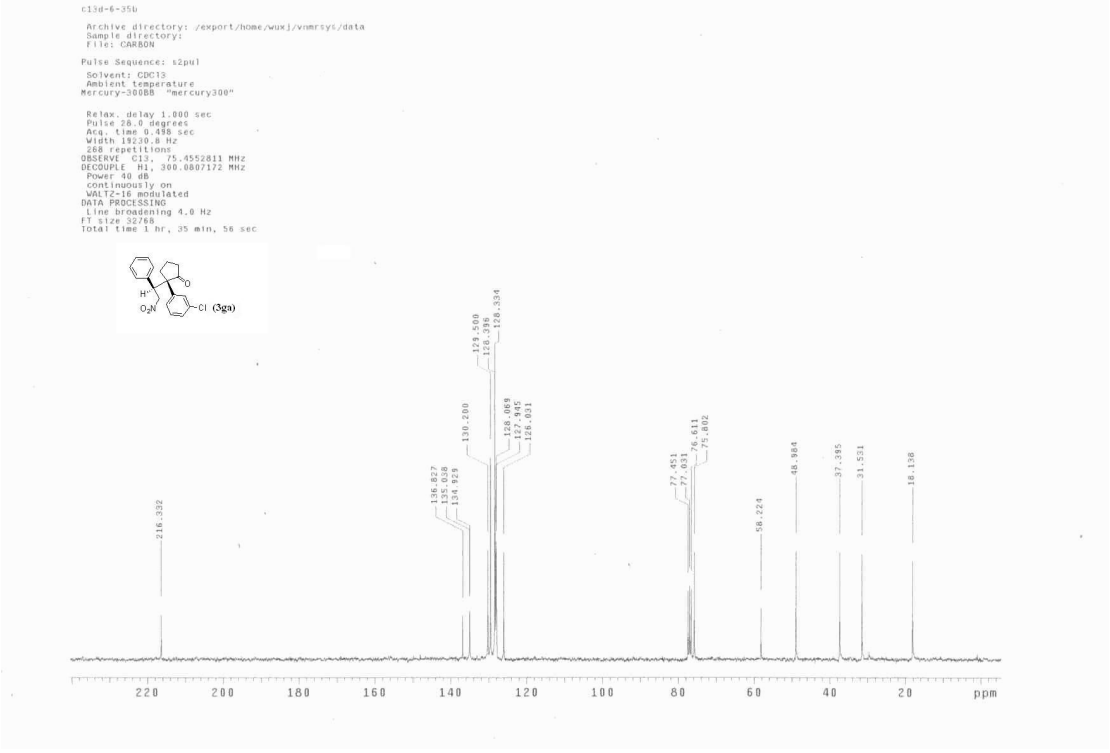

d-6-31a
Archive directory: /export/home/wu/vnmrsys/data
Sample directory:
File: PROTON
Pulse Sequence: s2pul
(3ea)
ppm

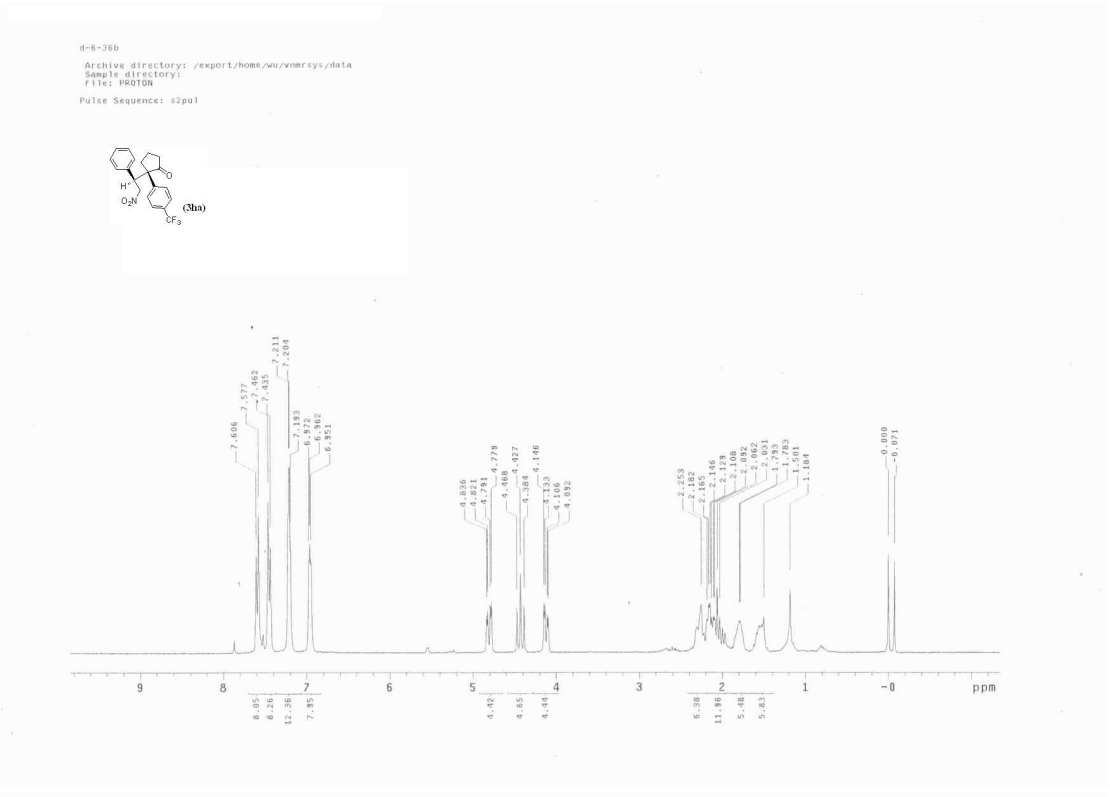

c13d-6-31a
Archive directory: /export/home/wuxj/vnmrsys/data
Sample directory:
File: CARBON
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
Mercury-300BB "mercury300"
Relax. delay 1.000 sec
Pulse 20.0 degrees
Acq. time 0.498 sec
Width 19230.8 Hz
108 repetitions
OBSERVE C13, 75.4552811 MHz
DECOUPLE H1, 300.0807172 MHz
Power 40 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 4.0 Hz
FT size 32768
Total time 1 hr, 35 min, 56 sec
(3ea)
ppm

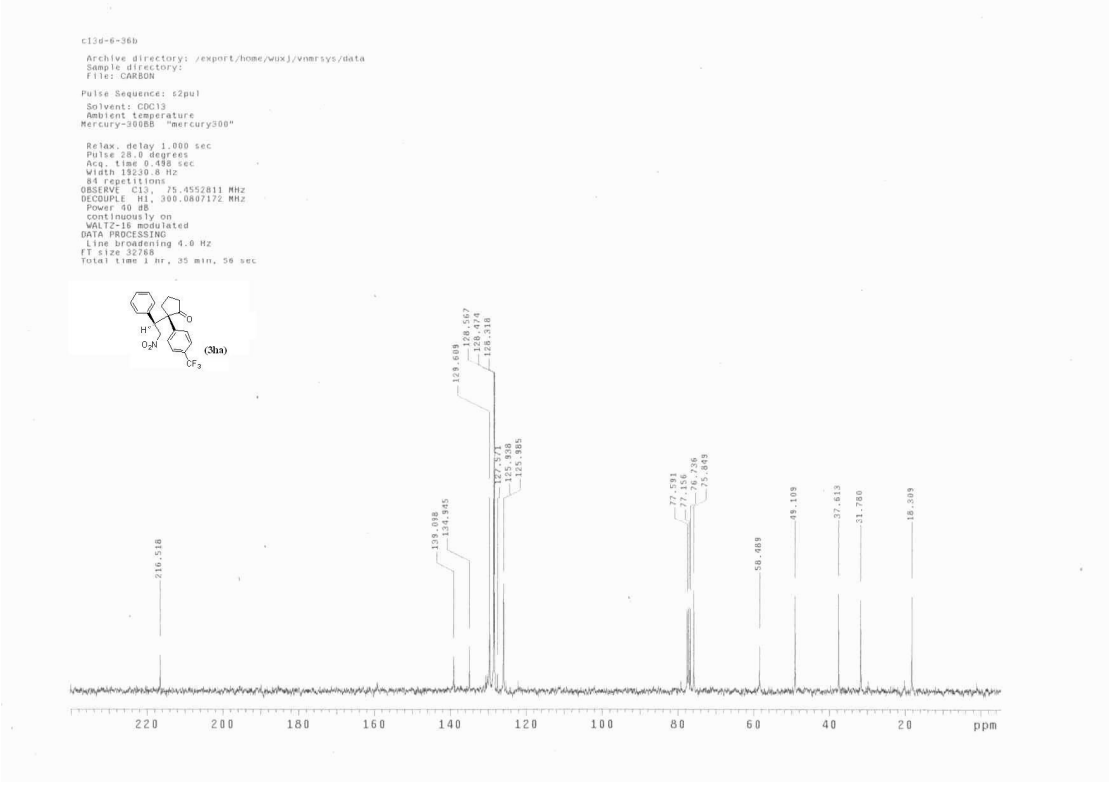

d-6-31c
Archive directory: /export/home/wu/vnmrsys/data
Sample directory:
File: PROTON
Pulse Sequence: s2pul
(3fa)
ppm

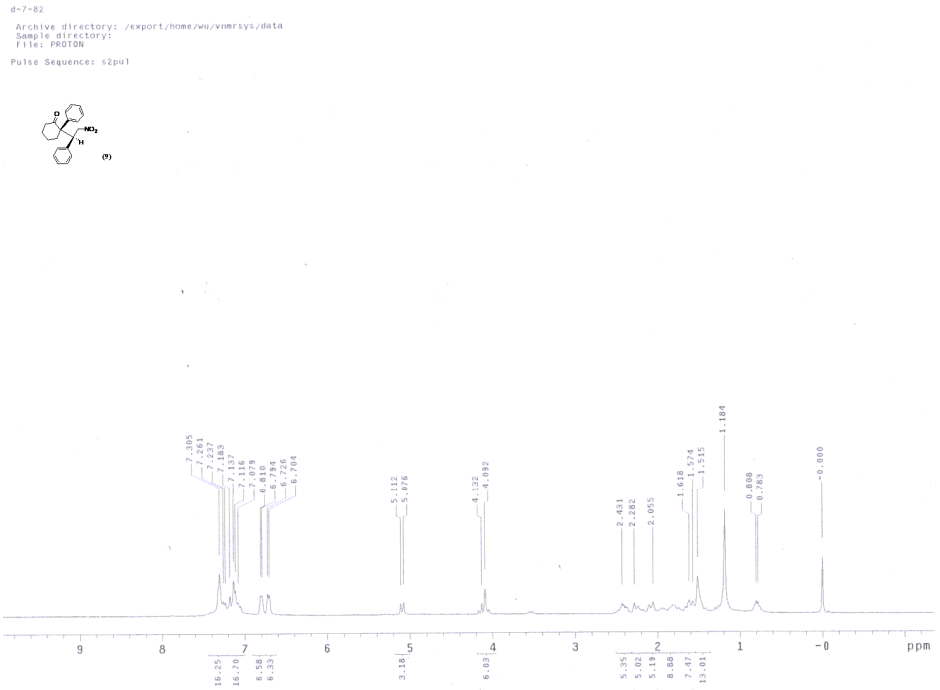

c13d-6-31c
Archive directory: /export/home/wuxj/vnmrsys/data
Sample directory:
File: CARBON
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
Mercury-300BB "mercury300"
Relax. delay 1.000 sec
Pulse 28.0 degrees
Acq. time 0.498 sec
Width 19230.8 Hz
92 repetitions
OBSERVE C13, 75.4552811 MHz
DECOUPLE H1, 300.0807172 MHz
Power 40 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 4.0 Hz
FT size 32768
Total time 1 hr, 35 min, 56 sec
(3fa)
ppm

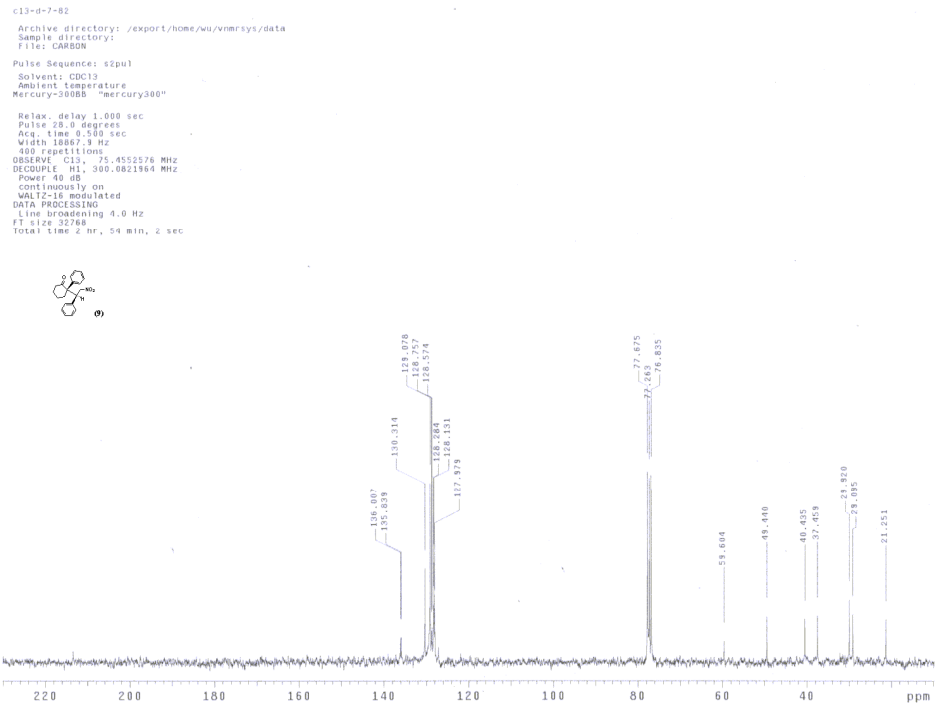

d-6-35b
Archive directory: /export/home/wu/vnmrsys/data
Sample directory:
File: PROTON
Pulse Sequence: s2pul
(3ga)
ppm

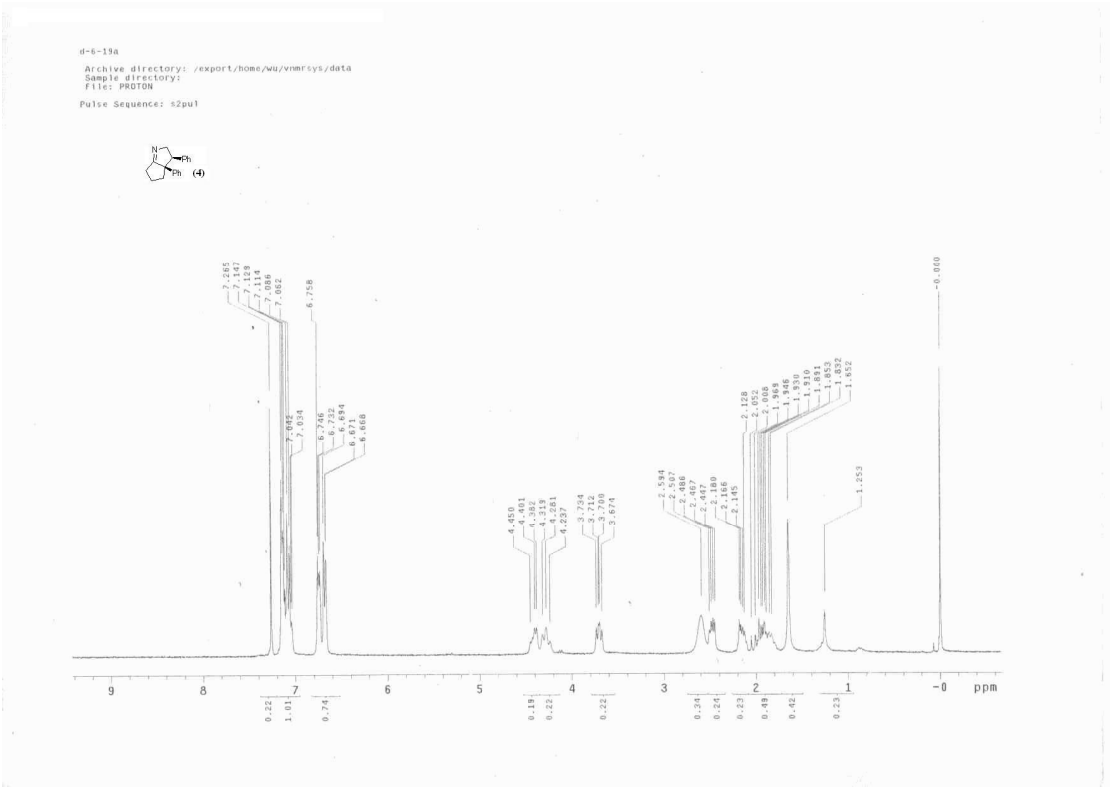

c13d-6-35b
Archive directory: /export/home/wuxj/vnmrsys/data
Sample directory:
File: CARBON
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
Mercury-300BB "mercury300"
Relax. delay 1.000 sec
Pulse 28.0 degrees
Acq. time 0.498 sec
Width 19230.8 Hz
268 repetitions
OBSERVE C13, 75.4552811 MHz
DECOUPLE H1, 300.0807172 MHz
Power 40 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 4.0 Hz
FT size 32768
Total time 1 hr, 35 min, 56 sec
(3ga)
ppm

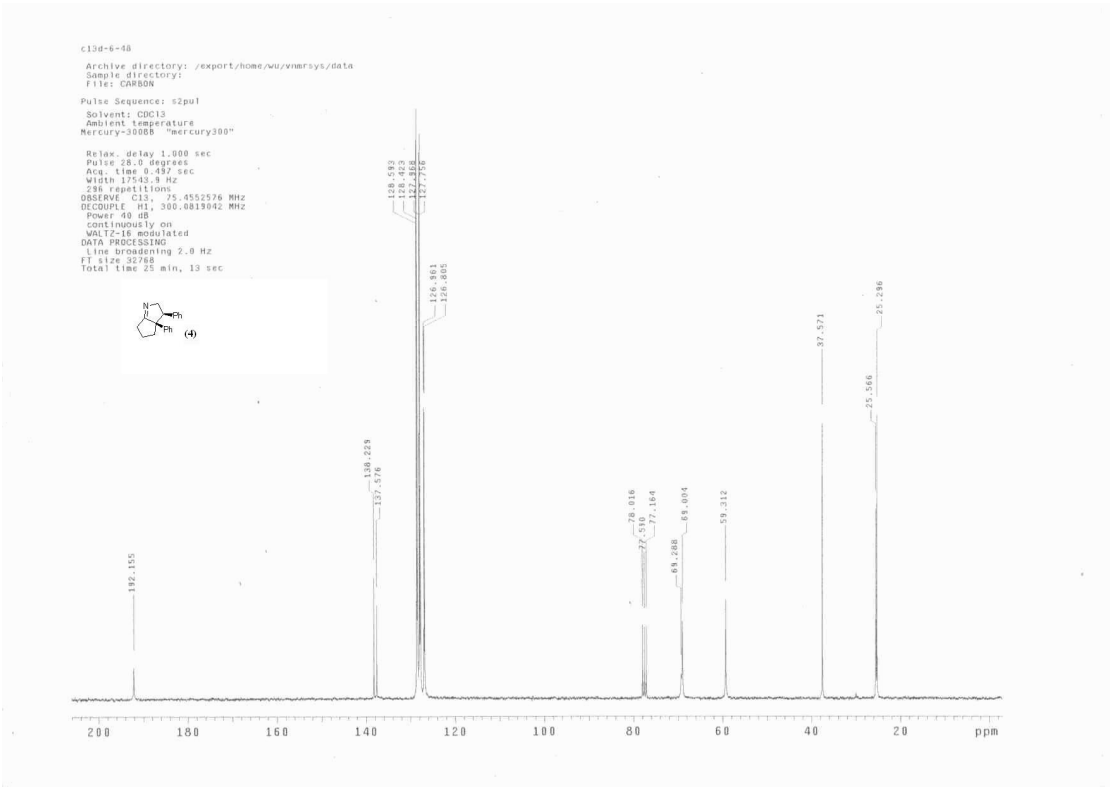

d-6-36b
Archive directory: /export/home/wu/vnmrsys/data
Sample directory:
File: PROTON
Pulse Sequence: s2pul
(3ha)
ppm

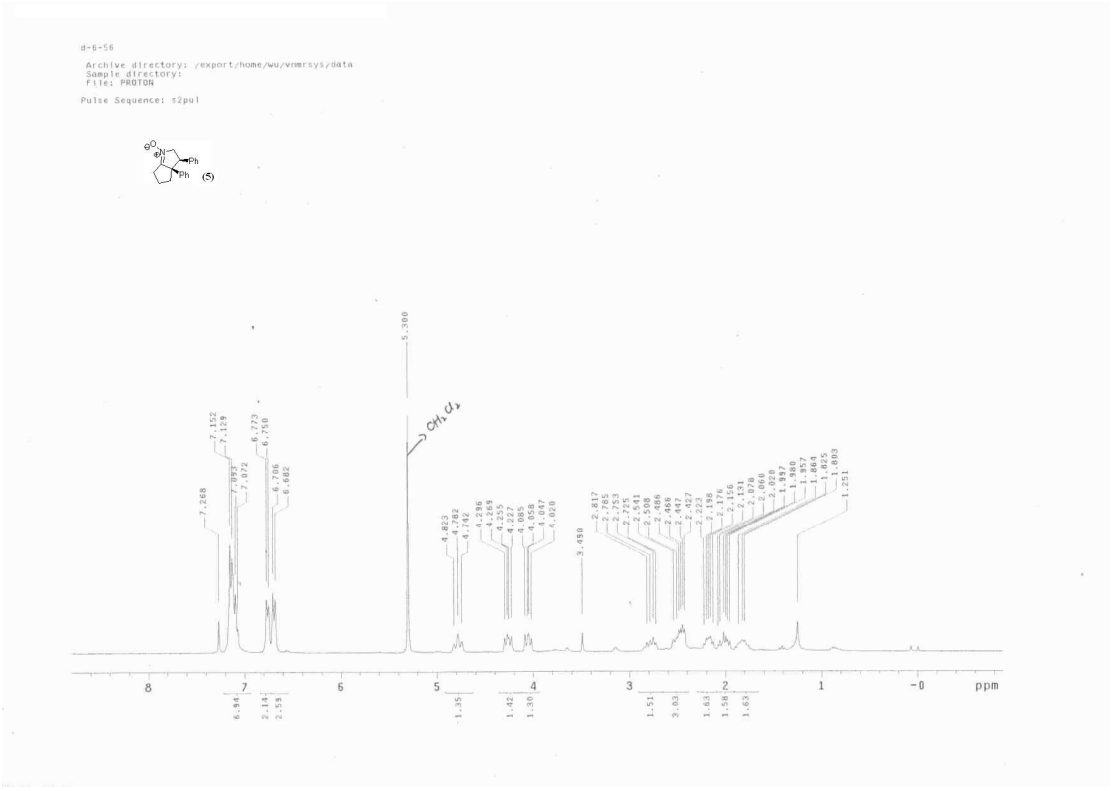

c13d-6-36b
Archive directory: /export/home/wuxj/vnmrsys/data
Sample directory:
File: CARBON
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
Mercury-300BB "mercury300"
Relax. delay 1.000 sec
Pulse 28.0 degrees
Acq. time 0.498 sec
Width 19230.8 Hz
84 repetitions
OBSERVE C13, 75.4552811 MHz
DECOUPLE H1, 300.0807172 MHz
Power 40 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 4.0 Hz
FT size 32768
Total time 1 hr, 35 min, 56 sec
(3ha)
ppm

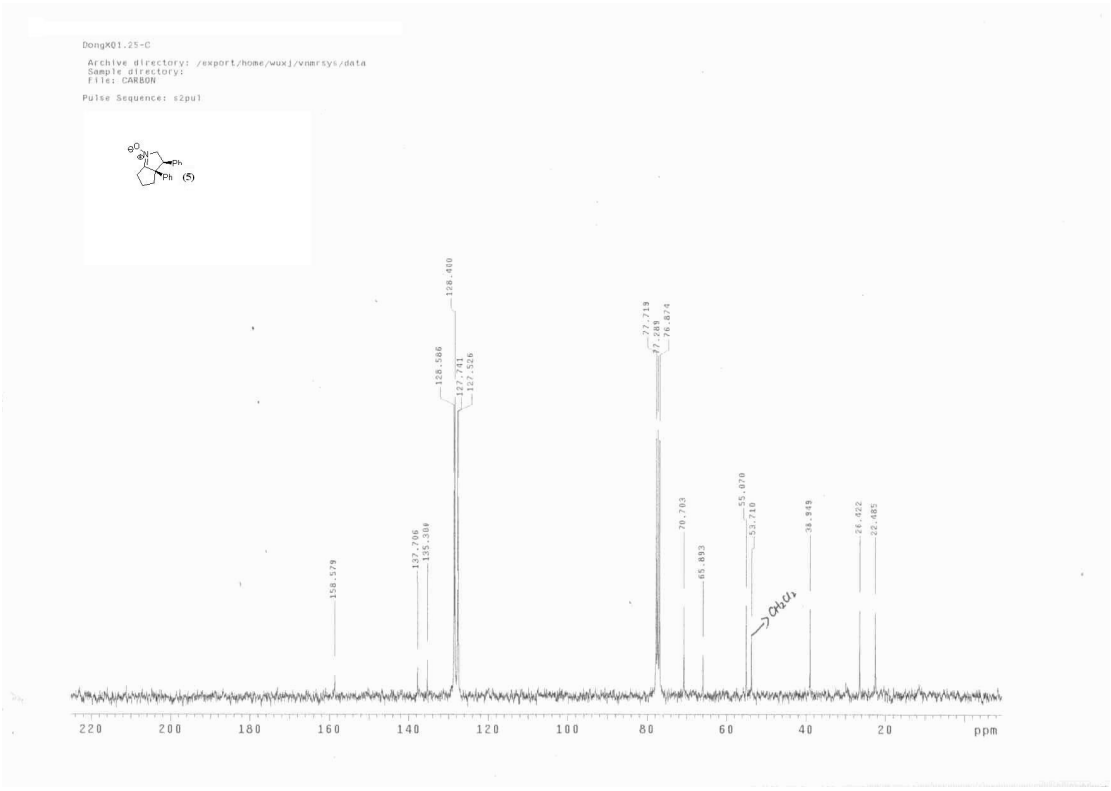

d-7-82
Archive directory: /export/home/wu/vnmrsys/data
Sample directory:
File: PROTON
Pulse Sequence: s2pul
7.305
7.261
7.237
7.188
7.137
7.116
7.079
6.810
6.794
6.726
6.704
5.112
5.076
4.132
4.092
2.431
2.282
2.055
1.618
1.574
1.515
1.184
0.808
0.763
0.000
ppm

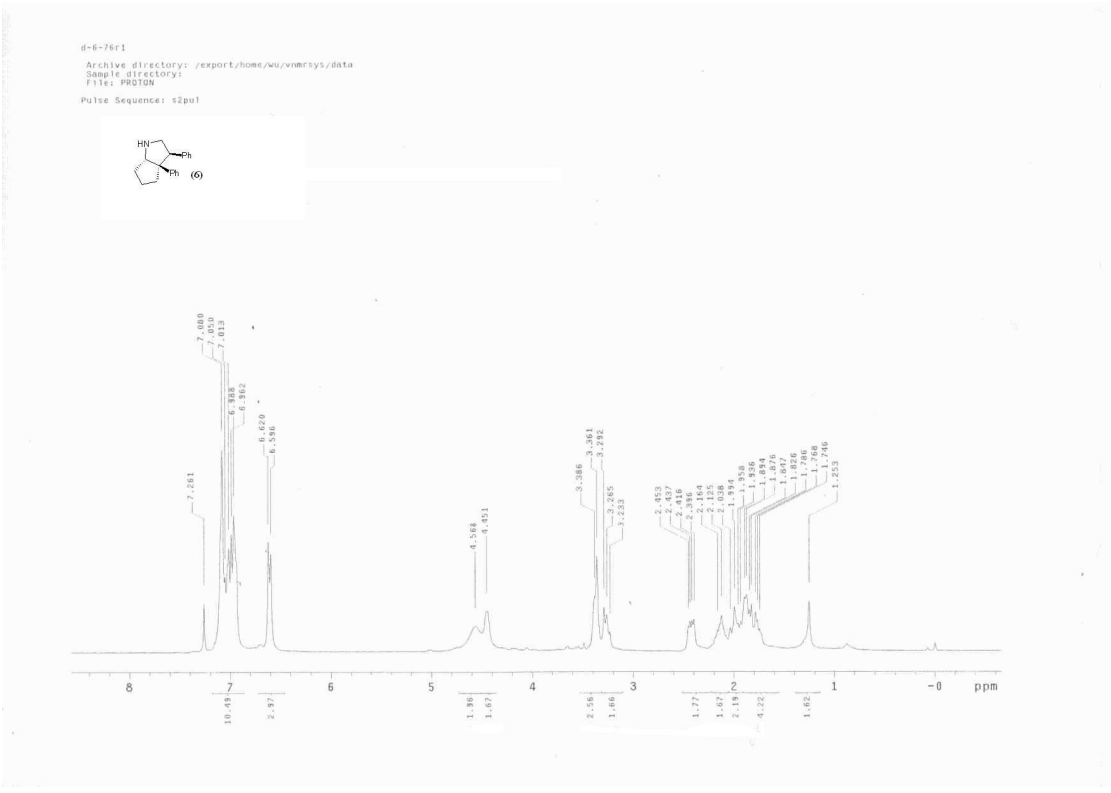

c13-d-7-82
Archive directory: /export/home/wu/vnmrsys/data
Sample directory:
File: CARBON
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
Mercury-300BB "mercury300"
Relax. delay 1.000 sec
Pulse 28.0 degrees
Acq. time 0.500 sec
Width 18867.9 Hz
400 repetitions
OBSERVE C13, 75.4552576 MHz
DECOUPLE H1, 300.0821964 MHz
Power 40 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 4.0 Hz
FT size 32768
Total time 2 hr, 54 min, 2 sec
136.007
135.839
130.314
129.078
128.757
128.574
128.244
126.131
127.979
77.675
77.263
76.835
59.604
49.440
40.435
37.459
31.920
29.005
21.251
ppm

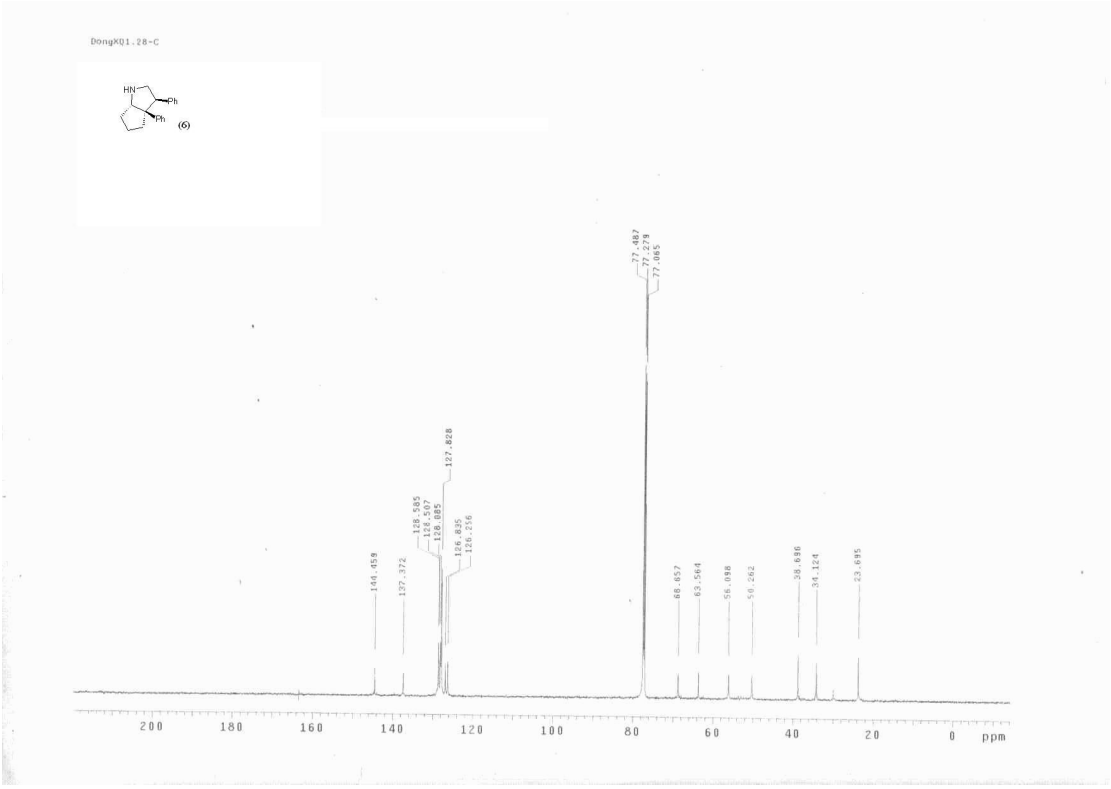

d-6-19a
Archive directory: /export/home/wu/vnmrsys/data
Sample directory:
File: PROTON
Pulse Sequence: s2pul
(4)
ppm

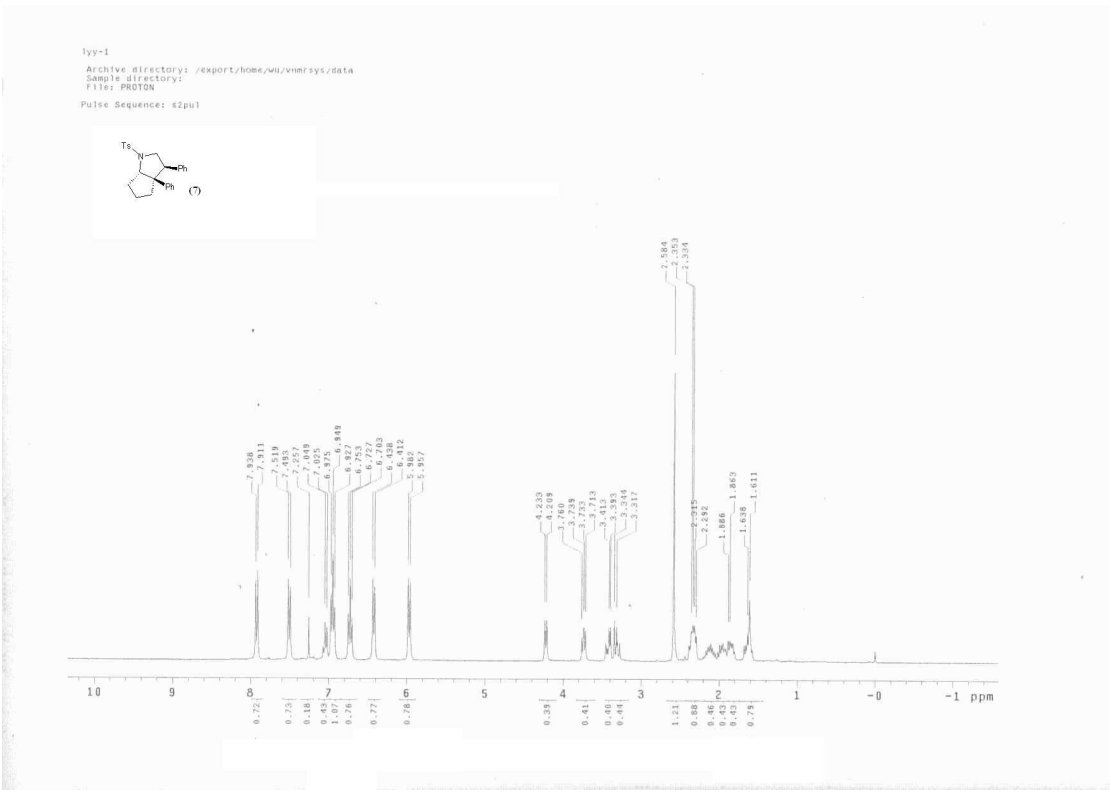

c13d-6-48
Archive directory: /export/home/wu/vnmrsys/data
Sample directory:
File: CARBON
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
Mercury-300BB "mercury300"
Relax. delay 1.000 sec
Pulse 28.0 degrees
Acq. time 0.497 sec
Width 17543.9 Hz
296 repetitions
OBSERVE C13, 75.4552576 MHz
DECOUPLE H1, 300.0813042 MHz
Power 40 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 2.0 Hz
FT size 32768
Total time 25 min, 13 sec
(4)
ppm

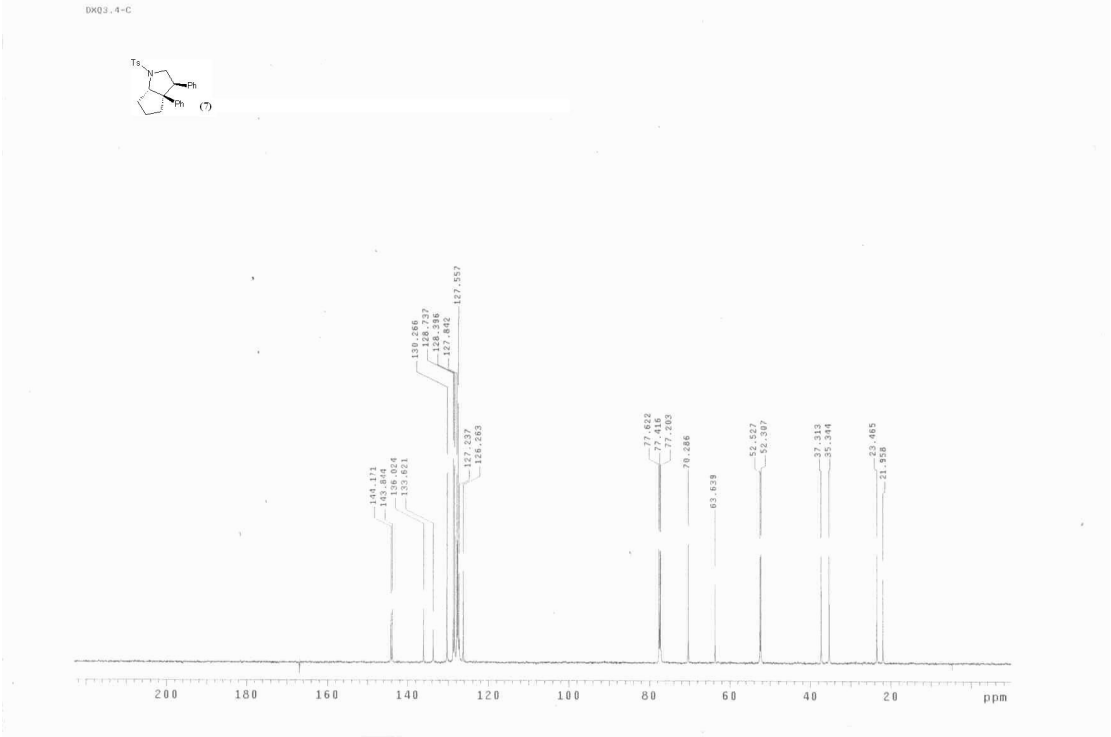

d-6-56
Archive directory: /export/home/wu/vnmrsys/data
Sample directory:
File: PROTON
Pulse Sequence: s2pul
(5)
CH2Cl2
ppm


DongX01.25-C
Archive directory: /export/home/wuxj/vnmrsys/data
Sample directory:
File: CARBON
Pulse Sequence: s2pul
(5)
CH2Cl2
ppm


d-6-76r1
Archive directory: /export/home/wu/vnmrsys/data
Sample directory:
File: PROTON
Pulse Sequence: s2pul
HN
Ph
Ph
(6)
7.261
7.080
7.060
7.013
6.988
6.962
6.620
6.596
4.568
4.451
3.386
3.361
3.282
3.265
3.233
2.453
2.437
2.416
2.396
2.164
2.125
2.038
1.994
1.958
1.936
1.894
1.876
1.857
1.826
1.786
1.768
1.746
1.253
8
7
6
5
4
3
2
1
-0
ppm
10.49
2.97
1.36
1.67
2.56
1.66
1.77
1.67
2.19
4.22
1.62

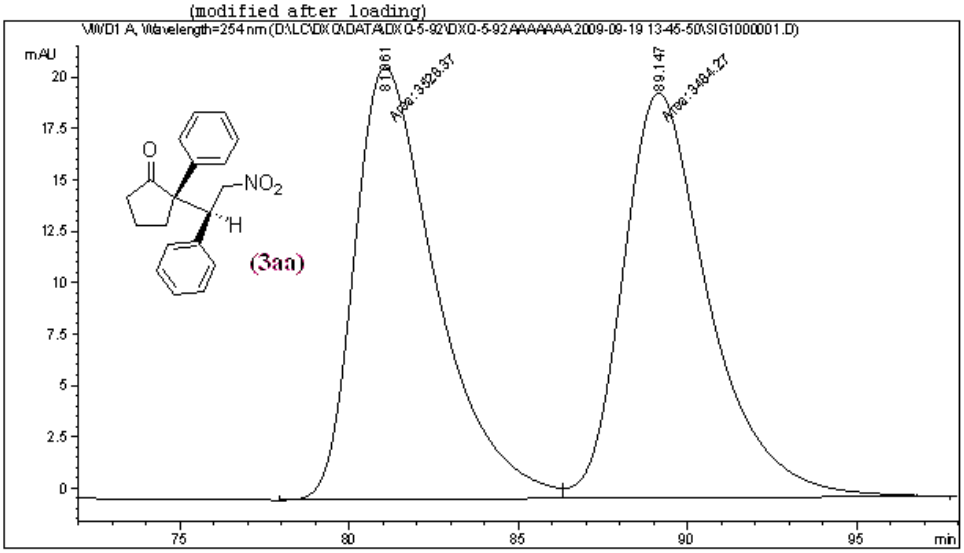

DongXQ1.28-C
HN
Ph
Ph
(6)
144.459
137.372
128.585
128.507
128.085
127.828
126.835
126.256
77.487
77.279
77.065
68.657
63.564
56.088
50.262
38.686
34.124
23.695
200
180
160
140
120
100
80
60
40
20
0
ppm


lyy-1
Archive directory: /export/home/wu/vnmrsys/data
Sample directory:
File: PROTON
Pulse Sequence: s2pul
Ts
Ph
Ph
(7)
7.938
7.911
7.519
7.493
7.253
7.048
7.025
6.975
6.848
6.827
6.753
6.722
6.703
6.688
6.412
5.982
5.957
4.233
4.209
3.760
3.735
3.713
3.415
3.393
3.344
3.317
2.584
2.353
2.304
2.015
2.292
1.886
1.863
1.638
1.611
10
9
8
7
6
5
4
3
2
1
-0
-1 ppm


DXQ3.4-C
Ts
Ph
Ph
(7)
144.171
143.804
136.024
135.621
130.266
128.737
128.396
127.892
127.557
127.237
126.263
77.622
77.416
77.203
70.286
63.639
52.527
52.307
37.313
35.344
23.465
21.958
200
180
160
140
120
100
80
60
40
20
ppm

Data File D:\LC\DXQ\DATA\DXQ-5-92\DXQ-5-92AAAAAAA 2009-09-19 13-45-50\SIG1000001.D
Sample Name: dxq-5-92a

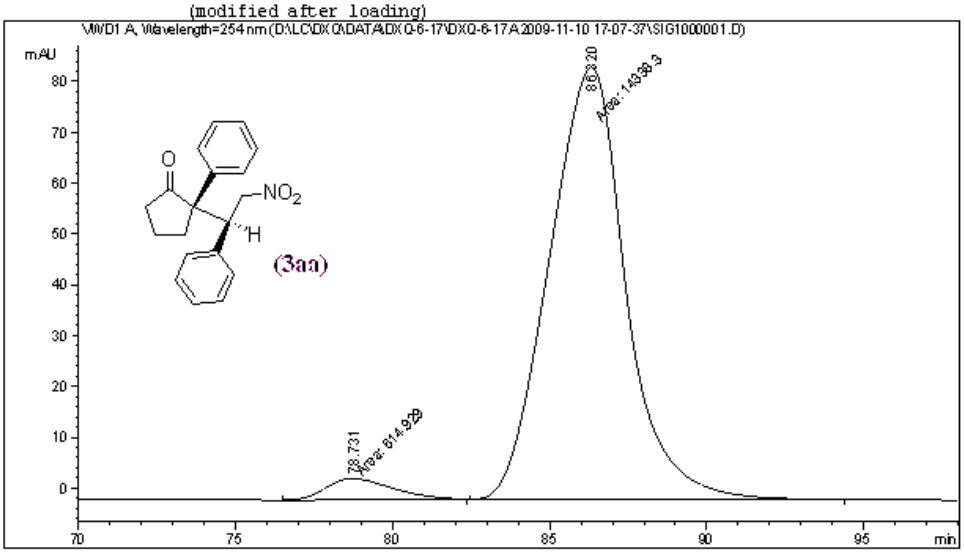
```
    ====================================================================
    Acq. Operator   : dxq                          Seq. Line :   1
    Acq. Instrument : Instrument 1                  Location : Vial 7
    Injection Date  : 9/19/2009 1:47:41 PM               Inj :   1
                                                   Inj Volume : 5 µl
    Acq. Method     : D:\LC\DXQ\data\DXQ-5-92\DXQ-5-92AAAAAAA 2009-09-19 13-45-50\ADH-1-99-
                      025ML-254NM.M
    Last changed    : 9/19/2009 1:44:30 PM by dxq
    Analysis Method : D:\LC\DXQ\DATA\DXQ-5-92\DXQ-5-92AAAAAAA 2009-09-19 13-45-50\SIG1000001.D\
                      DA.M (ADH-1-99-025ML-254NM.M)
    Last changed    : 3/6/2010 3:50:59 PM by zzh
                      (modified after loading)
```




```
    ====================================================================
                            Area Percent Report
    ====================================================================

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


    Signal 1: VWD1 A, Wavelength=254 nm

    Peak RetTime Type  Width     Area      Height     Area
      #   [min]        [min]  mAU   *s   [mAU   ]      %
    ----|-------|----|-------|----------|----------|-------|
       1  81.061 MF    2.7951 3528.36670   21.03867  50.3144
       2  89.147 FM    2.9441 3484.27441   19.72471  49.6856

    Totals :                  7012.64111   40.76339

    ====================================================================
```

Data File D:\LC\DXQ\DATA\DXQ-6-17\DXQ-6-17A 2009-11-10 17-07-37\SIG1000001.D
Sample Name: dxq-6-17a

Acq. Operator	: dxq	Seq. Line	: 1
Acq. Instrument	: Instrument 1	Location	: Vial 1
Injection Date	: 11/10/2009 5:08:52 PM	Inj	: 1
		Inj Volume	: 5 µl
Acq. Method	: D:\LC\DXQ\data\DXQ-6-17\DXQ-6-17A 2009-11-10 17-07-37\ADH-1-99-025ML-254NM-110MIN.M		
Last changed	: 9/26/2009 4:00:20 PM by ltl		
Analysis Method	: D:\LC\DXQ\DATA\DXQ-6-17\DXQ-6-17A 2009-11-10 17-07-37\SIG1000001.D\DA.M (ADH-1-99-025ML-254NM-110MIN.M)		
Last changed	: 3/6/2010 3:54:28 PM by zzh (modified after loading)		



Area Percent Report

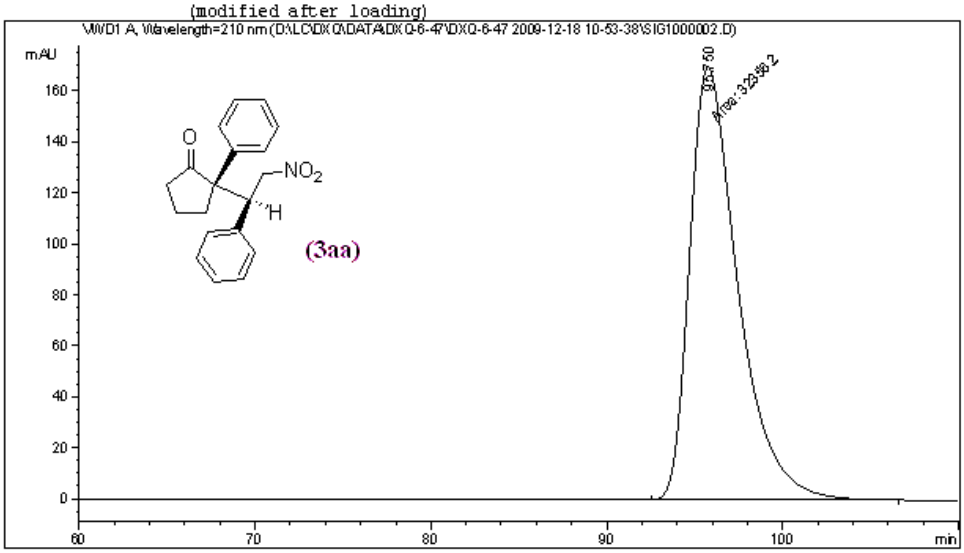
Sorted By : Signal
Multiplier : 1.0000
Dilution : 1.0000
Use Multiplier & Dilution Factor with ISTDs

Signal 1: VWD1 A, Wavelength=254 nm

Peak #	RetTime [min]	Type	Width [min]	Area mAU *s	Height [mAU]	Area %
1	78.731	MM	2.4528	614.92926	4.17848	4.1124
2	86.320	MM	2.8078	1.43383e4	85.11008	95.8876
Totals :				1.49532e4	89.28857	

Data File D:\LC\DXQ\DATA\DXQ-6-47\DXQ-6-47 2009-12-18 10-53-38\SIG1000002.D
Sample Name: dxq-6-47b

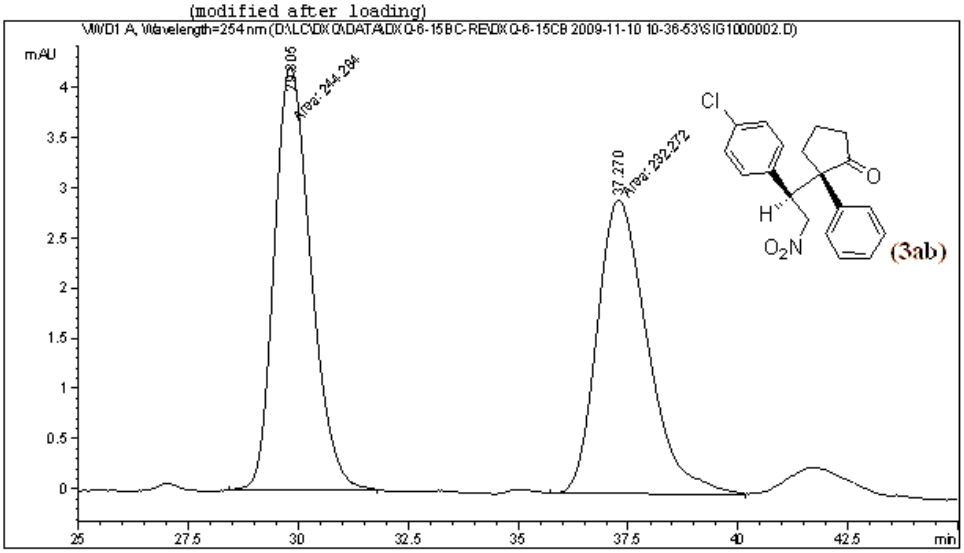
```
    ====================================================================
    Acq. Operator   : dxq                          Seq. Line :   2
    Acq. Instrument : Instrument 1                  Location : Vial 2
    Injection Date  : 12/18/2009 12:46:28 PM             Inj :   1
                                                  Inj Volume : 5 µl
    Acq. Method     : D:\LC\DXQ\data\DXQ-6-47\DXQ-6-47 2009-12-18 10-53-38\ADH-1-99-025ML-210NM-
                      110MIN.M
    Last changed    : 11/11/2009 8:50:09 AM by dxq
    Analysis Method : D:\LC\DXQ\DATA\DXQ-6-47\DXQ-6-47 2009-12-18 10-53-38\SIG1000002.D\DA.M (
                      ADH-1-99-025ML-210NM-110MIN.M)
    Last changed    : 3/15/2010 2:49:16 PM by LTL
                      (modified after loading)
```




```
    ====================================================================
                            Area Percent Report
    ====================================================================

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


    Signal 1: VWD1 A, Wavelength=210 nm

    Peak RetTime Type  Width     Area      Height     Area
      #   [min]        [min]  mAU   *s   [mAU   ]      %
    ----|-------|----|-------|----------|----------|--------|
       1  95.750 MM    3.1765 3.23562e4   169.76732 100.0000

    Totals :                  3.23562e4   169.76732


    ====================================================================
                            *** End of Report ***

Instrument 1 3/15/2010 2:49:20 PM LTL                          Page 1 of 1
```

Data File D:\LC\DXQ\DATA\DXQ-6-15BC-RE\DXQ-6-15CB 2009-11-10 10-36-53\SIG1000002.D
Sample Name: dxq-6-15b

```
    ======================================================================
    Acq. Operator   : dxq                          Seq. Line :   2
    Acq. Instrument : Instrument 1                  Location : Vial 12
    Injection Date  : 11/10/2009 11:29:35 AM             Inj :   1
                                                   Inj Volume : 5 µl
    Acq. Method     : D:\LC\DXQ\data\DXQ-6-15BC-RE\DXQ-6-15CB 2009-11-10 10-36-53\ASH-90-10-
                      05ML-254NM-50MIN.M
    Last changed    : 11/10/2009 10:34:37 AM by dxq
    Analysis Method : D:\LC\DXQ\DATA\DXQ-6-15BC-RE\DXQ-6-15CB 2009-11-10 10-36-53\SIG1000002.D\
                      DA.M (ASH-90-10-05ML-254NM-50MIN.M)
    Last changed    : 3/6/2010 3:59:16 PM by zzh
                      (modified after loading)
```



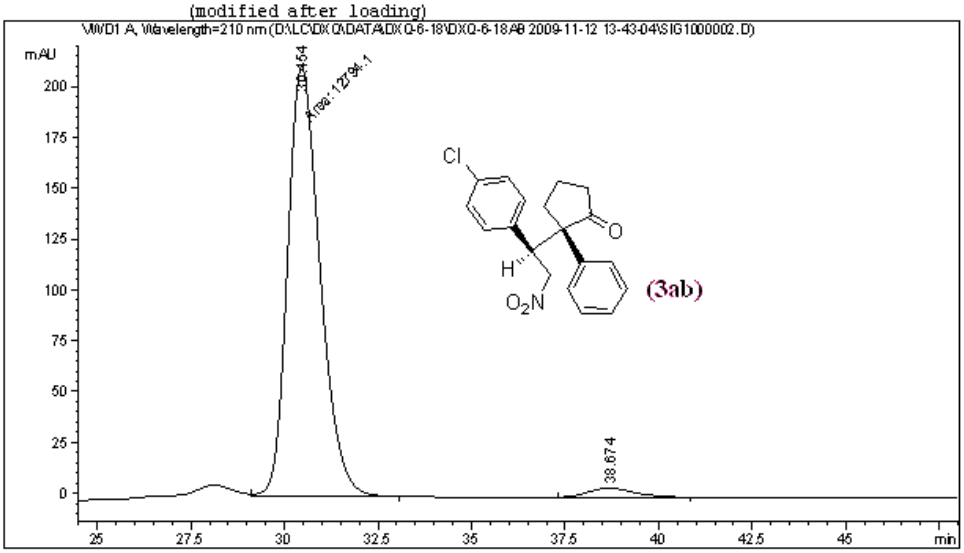

```
    ======================================================================
                            Area Percent Report
    ======================================================================

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


    Signal 1: VWD1 A, Wavelength=254 nm

    Peak RetTime Type  Width     Area      Height     Area
      #   [min]        [min]  mAU   *s   [mAU   ]      %
    ----|-------|----|-------|----------|----------|--------|
       1  29.805 MM    0.9667  244.28448    4.21160  51.2604
       2  37.270 MM    1.3228  232.27158    2.92648  48.7396

    Totals :                   476.55606    7.13807

    ======================================================================
```

Instrument 1 3/6/2010 3:59:20 PM zzh

Page 1 of 1

Data File D:\LC\DXQ\DATA\DXQ-6-18\DXQ-6-18AB 2009-11-12 13-43-04\SIG1000002.D
Sample Name: dxq-6-18a

```
=====================================================================
Acq. Operator   : dxq                          Seq. Line :   2
Acq. Instrument : Instrument 1                  Location : Vial 7
Injection Date  : 11/12/2009 1:55:45 PM              Inj :   1
                                               Inj Volume : 5 µl
Acq. Method     : D:\LC\DXQ\data\DXQ-6-18\DXQ-6-18AB 2009-11-12 13-43-04\ASH-90-10-05ML-
                  210NM-50MIN.M
Last changed    : 11/11/2009 11:13:52 AM by dxq
Analysis Method : D:\LC\DXQ\DATA\DXQ-6-18\DXQ-6-18AB 2009-11-12 13-43-04\SIG1000002.D\DA.M (
                  ASH-90-10-05ML-210NM-50MIN.M)
Last changed    : 3/6/2010 4:02:33 PM by zzh
                  (modified after loading)
```



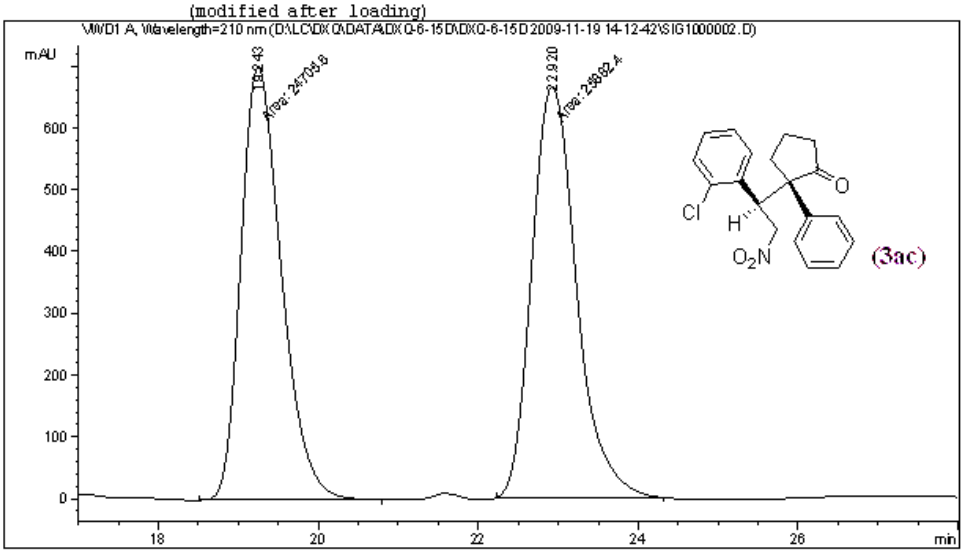

```
=====================================================================
                        Area Percent Report
=====================================================================

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


Signal 1: VWD1 A, Wavelength=210 nm

Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]  mAU   *s   [mAU   ]      %
----|-------|----|-------|----------|----------|--------|
   1  30.454 MM    1.0090 1.2794le4   211.33441  97.0425
   2  38.674 BB    1.0303  389.91882    4.55700   2.9575

Totals :                  1.31840e4   215.89141

=====================================================================
```

Instrument 1 3/6/2010 4:02:37 PM zzh

Page 1 of 1

Data File D:\LC\DXQ\DATA\DXQ-6-15D\DXQ-6-15D 2009-11-19 14-12-42\SIG1000002.D
Sample Name: dxq-6-15d

```
    ======================================================================
    Acq. Operator   : dxq                          Seq. Line :   2
    Acq. Instrument : Instrument 1                  Location : Vial 7
    Injection Date  : 11/19/2009 2:25:05 PM              Inj :   1
                                                   Inj Volume : 5 µl
    Acq. Method     : D:\LC\DXQ\data\DXQ-6-15D\DXQ-6-15D 2009-11-19 14-12-42\ASH-90-10-05ML-
                      210NM.M
    Last changed    : 11/15/2009 2:15:18 PM by DXQ
    Analysis Method : D:\LC\DXQ\DATA\DXQ-6-15D\DXQ-6-15D 2009-11-19 14-12-42\SIG1000002.D\DA.M (
                      ASH-90-10-05ML-210NM.M)
    Last changed    : 3/6/2010 4:07:39 PM by zzh
                      (modified after loading)
```




```
    ======================================================================
                              Area Percent Report
    ======================================================================

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


    Signal 1: VWD1 A, Wavelength=210 nm

    Peak RetTime Type  Width     Area      Height     Area
      #   [min]        [min]  mAU   *s   [mAU   ]      %
    ----|-------|----|-------|----------|----------|--------|
       1  19.243 MM    0.5878 2.47056e4   700.55530  48.8369
       2  22.920 MM    0.6489 2.58824e4   664.75305  51.1631

    Totals :                  5.05879e4  1365.30835

    ======================================================================
```

Instrument 1 3/6/2010 4:07:44 PM zzh

Data File D:\LC\DXQ\DATA\DXQ-6-18\DXQ-6-18K 2009-11-19 15-58-30\SIG1000001.D
Sample Name: dxq-6-18k

```
    ====================================================================
    Acq. Operator   : dxq                          Seq. Line :   1
    Acq. Instrument : Instrument 1                  Location : Vial 9
    Injection Date  : 11/19/2009 3:59:58 PM              Inj :   1
                                                  Inj Volume : 5 µl
    Acq. Method     : D:\LC\DXQ\data\DXQ-6-18\DXQ-6-18K 2009-11-19 15-58-30\ASH-90-10-05ML-
                      210NM-50MIN.M
    Last changed    : 11/11/2009 11:13:52 AM by dxq
    Analysis Method : D:\LC\DXQ\DATA\DXQ-6-18\DXQ-6-18K 2009-11-19 15-58-30\SIG1000001.D\DA.M (
                      ASH-90-10-05ML-210NM-50MIN.M)
    Last changed    : 3/6/2010 4:10:00 PM by zzh
                      (modified after loading)
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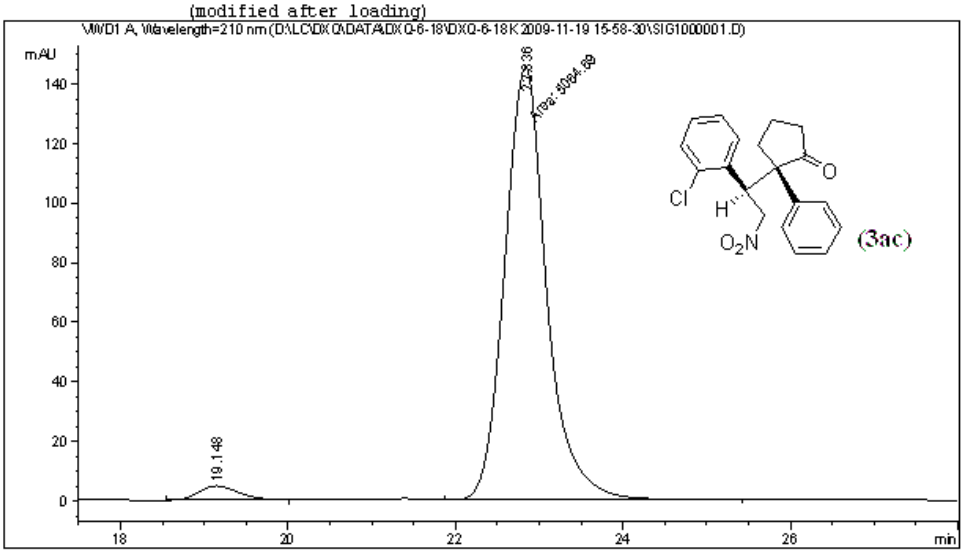



```
    ====================================================================
                            Area Percent Report
    ====================================================================

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


    Signal 1: VWD1 A, Wavelength=210 nm

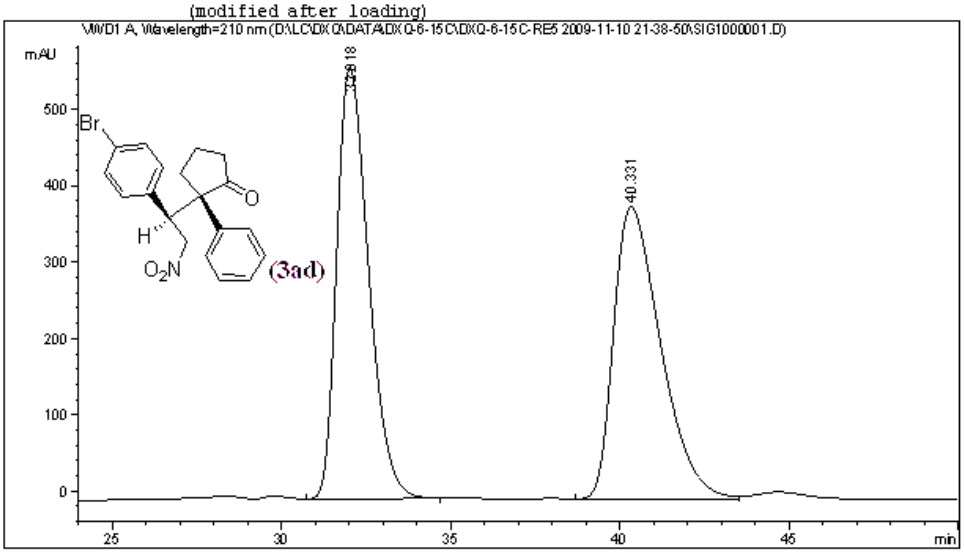
    Peak RetTime Type  Width     Area      Height     Area
      #   [min]        [min]  mAU   *s   [mAU   ]      %
    ----|-------|----|-------|----------|----------|--------|
       1  19.148 BB    0.5045  158.11832    4.77254   3.0275
       2  22.836 MM    0.5808 5064.68701  145.32640  96.9725

    Totals :                  5222.80533  150.09895


    ====================================================================
```

Data File D:\LC\DXQ\DATA\DXQ-6-15C\DXQ-6-15C-RE5 2009-11-10 21-38-50\SIG1000001.D
Sample Name: dxq-6-15c1

Acq. Operator	: dxq	Seq. Line	: 1
Acq. Instrument	: Instrument 1	Location	: Vial 11
Injection Date	: 11/10/2009 9:40:08 PM	Inj	: 1
		Inj Volume	: 5 µl
Acq. Method	: D:\LC\DXQ\data\DXQ-6-15BC\DXQ-6-15C-RE5 2009-11-10 21-38-50\ASH-90-10-05ML-210NM-50MIN.M		
Last changed	: 11/10/2009 9:33:03 PM by dxq		
Analysis Method	: D:\LC\DXQ\DATA\DXQ-6-15C\DXQ-6-15C-RE5 2009-11-10 21-38-50\SIG1000001.D\DA.M (ASH-90-10-05ML-210NM-50MIN.M)		
Last changed	: 3/6/2010 4:11:49 PM by zzh (modified after loading)		



Area Percent Report

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

Signal 1: VWD1 A, Wavelength=210 nm

Peak #	RetTime [min]	Type	Width [min]	Area mAU *s	Height [mAU]	Area %
1	32.018	VB	1.0106	3.68375e4	565.33667	49.9106
2	40.331	VB	1.4896	3.69694e4	382.70236	50.0894
Totals :				7.38069e4	948.03903	

Data File D:\LC\DXQ\DATA\DXQ-6-18\DXQ-6-18AB 2009-11-12 13-43-04\SIG1000003.D
Sample Name: dxq-6-18b

```
    ======================================================================
    Acq. Operator   : dxq                          Seq. Line :   3
    Acq. Instrument : Instrument 1                  Location : Vial 8
    Injection Date  : 11/12/2009 2:47:09 PM              Inj :   1
                                                   Inj Volume : 5 µl
    Acq. Method     : D:\LC\DXQ\data\DXQ-6-18\DXQ-6-18AB 2009-11-12 13-43-04\ASH-90-10-05ML-
                      210NM-50MIN.M
    Last changed    : 11/11/2009 11:13:52 AM by dxq
    Analysis Method : D:\LC\DXQ\DATA\DXQ-6-18\DXQ-6-18AB 2009-11-12 13-43-04\SIG1000003.D\DA.M (
                      ASH-90-10-05ML-210NM-50MIN.M)
    Last changed    : 3/6/2010 4:44:45 PM by zzh
                      (modified after loading)
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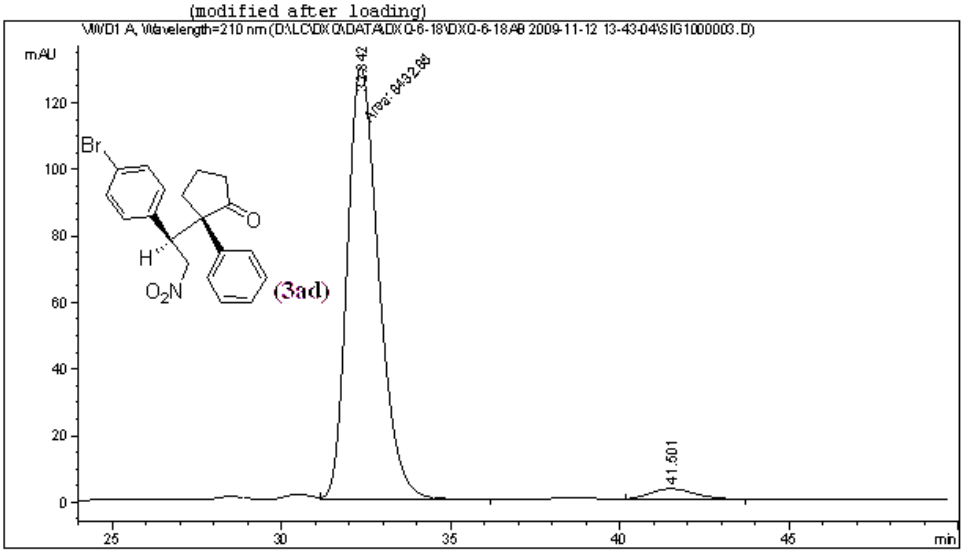
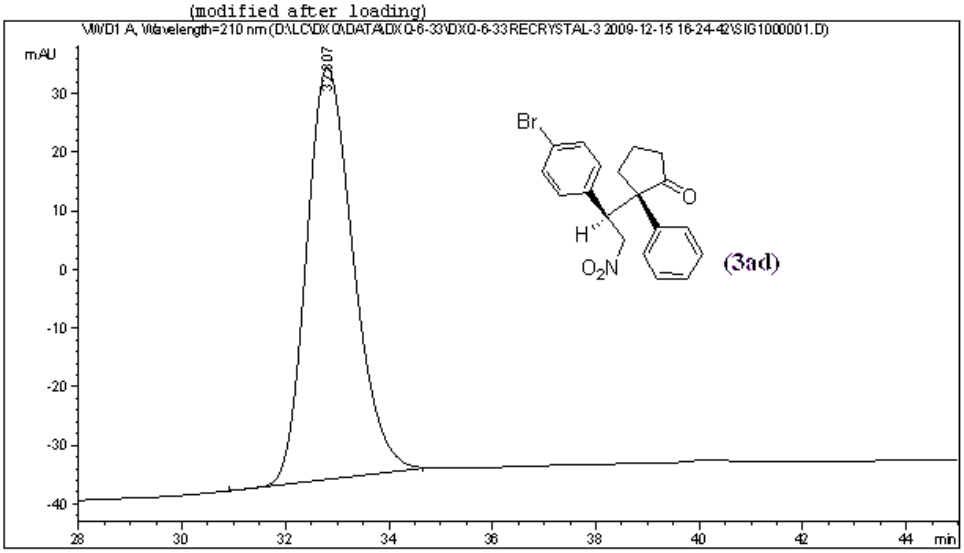



```
    ======================================================================
                           Area Percent Report
    ======================================================================

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


    Signal 1: VWD1 A, Wavelength=210 nm

    Peak RetTime Type  Width     Area      Height     Area
      #   [min]        [min]  mAU   *s    [mAU   ]      %
    ----|-------|----|-------|----------|----------|--------|
       1  32.342 MM    1.0800 8432.84863  130.13411  96.7804
       2  41.501 BB    1.0836  280.53632    3.09942   3.2196

    Totals :                  8713.38495  133.23353

    ======================================================================
```

Data File D:\LC\DXQ\DATA\DXQ-6-33\DXQ-6-33RECRYSTAL-3 2009-12-15 16-24-42\SIG1000001.D
Sample Name: dxq-6-33recrytal-3

Acq. Operator	: dxq
Seq. Line	: 1
Acq. Instrument	: Instrument 1
Location	: Vial 2
Injection Date	: 12/15/2009 4:25:54 PM
Inj	: 1
Inj Volume	: 5 µl
Acq. Method	: D:\LC\DXQ\data\DXQ-6-33\DXQ-6-33RECRYSTAL-3 2009-12-15 16-24-42\ASH-90-10-05ML-210NM-50MIN.M
Last changed	: 11/11/2009 11:13:52 AM by dxq
Analysis Method	: D:\LC\DXQ\DATA\DXQ-6-33\DXQ-6-33RECRYSTAL-3 2009-12-15 16-24-42\SIG1000001.D\DA.M (ASH-90-10-05ML-210NM-50MIN.M)
Last changed	: 3/15/2010 2:46:57 PM by LTL (modified after loading)



Area Percent Report

Sorted By	: Signal
Multiplier	: 1.0000
Dilution	: 1.0000

Use Multiplier & Dilution Factor with ISTDs

Signal 1: VWD1 A, Wavelength=210 nm

Peak #	RetTime [min]	Type	Width [min]	Area mAU *s	Height [mAU]	Area %
1	32.807	BB	0.9593	4417.75391	70.39272	100.0000
Totals :				4417.75391	70.39272	

*** End of Report ***

Instrument 1 3/15/2010 2:47:02 PM LTL

Page 1 of 1

Data File D:\LC\DXQ\DATA\DXQ-6-15N\DXQ-6-15N 2009-11-18 09-36-59\SIG1000002.D
Sample Name: DXQ-6-15N

```
    ====================================================================
    Acq. Operator   : DXQ                          Seq. Line :   2
    Acq. Instrument : Instrument 1                  Location : Vial 2
    Injection Date  : 11/18/2009 9:49:25 AM              Inj :   1
                                                   Inj Volume : 5 µl
    Acq. Method     : D:\LC\LTL\data\DXQ-6-15N\DXQ-6-15N 2009-11-18 09-36-59\ASH-90-10-05ML-
                      210NM.M
    Last changed    : 11/15/2009 2:15:18 PM by DXQ
    Analysis Method : D:\LC\DXQ\DATA\DXQ-6-15N\DXQ-6-15N 2009-11-18 09-36-59\SIG1000002.D\DA.M (
                      ASH-90-10-05ML-210NM.M)
    Last changed    : 3/6/2010 4:47:34 PM by zzh
                      (modified after loading)
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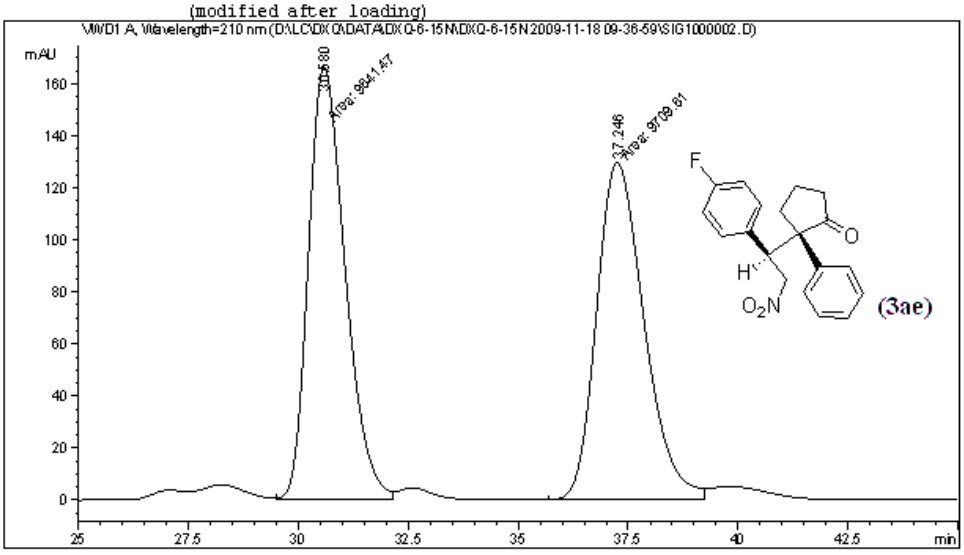


```
    ====================================================================
                          Area Percent Report
    ====================================================================

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


    Signal 1: VWD1 A, Wavelength=210 nm

    Peak RetTime Type  Width     Area      Height     Area
      #   [min]        [min]  mAU   *s    [mAU   ]      %
    ----|-------|----|-------|----------|----------|--------|
       1  30.580 MM    0.9676 9641.46777  166.07919  49.8239
       2  37.246 MM    1.2506 9709.61133  129.39854  50.1761

    Totals :                  1.9351le4   295.47774


    ====================================================================
```

Instrument 1 3/6/2010 4:47:39 PM zzh

Data File D:\LC\DXQ\DATA\DXQ-6-18\DXQ-6-18HIJ 2009-11-18 12-06-25\SIG1000002.D
Sample Name: DXQ-6-18H

```
    ====================================================================
    Acq. Operator   : DXQ                          Seq. Line :   2
    Acq. Instrument : Instrument 1                  Location : Vial 31
    Injection Date  : 11/18/2009 12:18:44 PM             Inj :   1
                                                   Inj Volume : 5 µl
    Acq. Method     : D:\LC\DXQ\data\DXQ-6-18\DXQ-6-18HIJ 2009-11-18 12-06-25\ASH-90-10-05ML-
                      210NM-50MIN.M
    Last changed    : 11/11/2009 11:13:52 AM by dxq
    Analysis Method : D:\LC\DXQ\DATA\DXQ-6-18\DXQ-6-18HIJ 2009-11-18 12-06-25\SIG1000002.D\DA.M
                      (ASH-90-10-05ML-210NM-50MIN.M)
    Last changed    : 3/6/2010 4:53:38 PM by zzh
                      (modified after loading)
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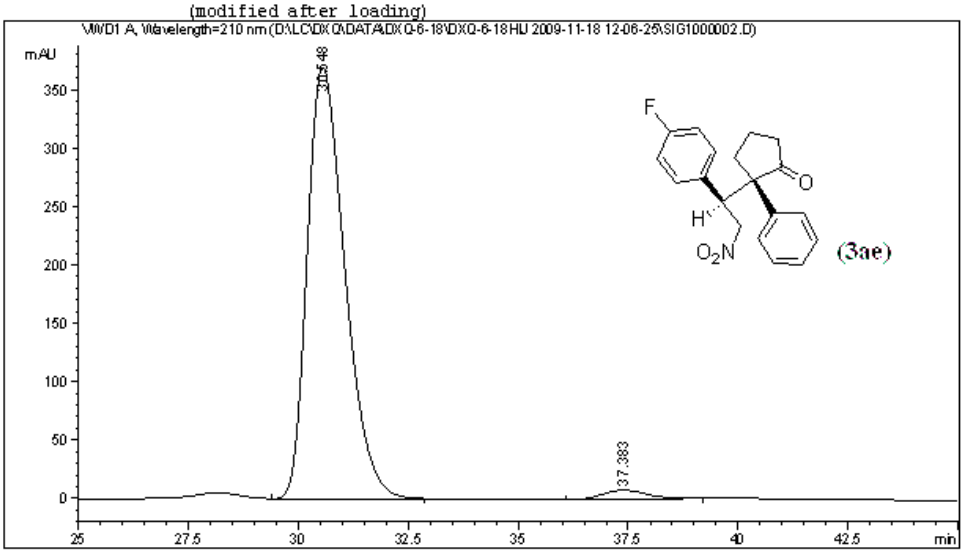


```
    ====================================================================
                          Area Percent Report
    ====================================================================

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


    Signal 1: VWD1 A, Wavelength=210 nm

    Peak RetTime Type  Width     Area      Height     Area
      #   [min]        [min]  mAU   *s   [mAU   ]      %
    ----|-------|----|-------|----------|----------|--------|
       1  30.548 VB    0.9186 2.19731e4   369.95352  97.6917
       2  37.383 BB    1.0012  519.18176    7.60991   2.3083

    Totals :                  2.24923e4   377.56343

    ====================================================================
```

Instrument 1 3/6/2010 4:53:42 PM zzh

Data File D:\LC\DXQ\DATA\DXQ-6-80\DXQ-6-80 2010-01-25 21-48-20\SIG1000002.D
Sample Name: d-6-15a

```
    ======================================================================
    Acq. Operator   : dxq                          Seq. Line :   2
    Acq. Instrument : Instrument 1                  Location : Vial 31
    Injection Date  : 1/25/2010 10:00:43 PM              Inj :   1
                                                  Inj Volume : 5 µl
    Acq. Method     : D:\LC\DXQ\data\DXQ-6-80\DXQ-6-80 2010-01-25 21-48-20\ASH-90-10-05ML-210NM-
                      50MIN.M
    Last changed    : 11/11/2009 11:13:52 AM by dxq
    Analysis Method : D:\LC\DXQ\DATA\DXQ-6-80\DXQ-6-80 2010-01-25 21-48-20\SIG1000002.D\DA.M (
                      ASH-90-10-05ML-210NM-50MIN.M)
    Last changed    : 3/6/2010 5:03:26 PM by zzh
                      (modified after loading)
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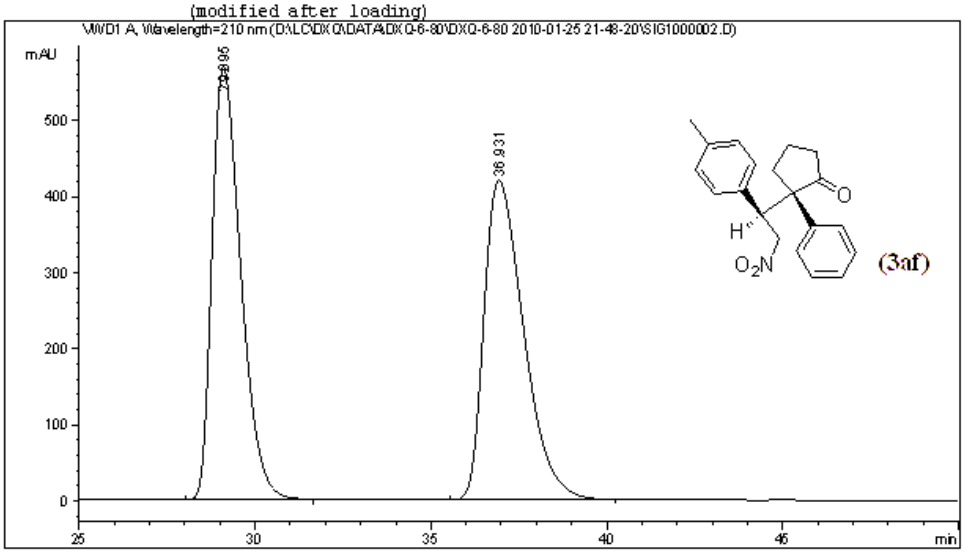



```
    ======================================================================
                           Area Percent Report
    ======================================================================

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


    Signal 1: VWD1 A, Wavelength=210 nm

    Peak RetTime Type  Width     Area      Height     Area
      #   [min]        [min]  mAU   *s    [mAU   ]      %
    ----|-------|----|-------|----------|----------|--------|
       1  29.095 BB    0.8820 3.23742e4   569.20331  49.6043
       2  36.931 VB    1.2223 3.28907e4   420.01007  50.3957

    Totals :                  6.52648e4   989.21338

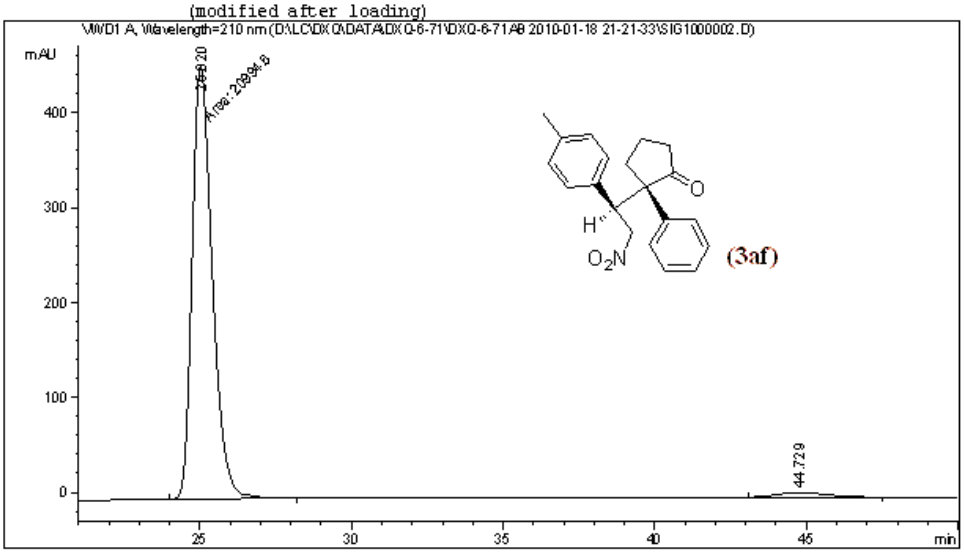

    ======================================================================
```

Instrument 1 3/6/2010 5:03:30 PM zzh

Page 1 of 1

Data File D:\LC\DXQ\DATA\DXQ-6-71\DXQ-6-71AB 2010-01-18 21-21-33\SIG1000002.D
Sample Name: DXQ-6-71A

Acq. Operator	: DXQ	Seq. Line	: 2
Acq. Instrument	: Instrument 1	Location	: Vial 11
Injection Date	: 1/18/2010 9:33:59 PM	Inj	: 1
		Inj Volume	: 5 µl
Acq. Method	: D:\LC\DXQ\data\DXQ-6-71\DXQ-6-71AB 2010-01-18 21-21-33\ASH-90-10-05ML-210NM-50MIN.M		
Last changed	: 11/11/2009 11:13:52 AM by dxq		
Analysis Method	: D:\LC\DXQ\DATA\DXQ-6-71\DXQ-6-71AB 2010-01-18 21-21-33\SIG1000002.D\DA.M (ASH-90-10-05ML-210NM-50MIN.M)		
Last changed	: 3/6/2010 5:05:27 PM by zzh (modified after loading)		



Area Percent Report

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

Signal 1: VWD1 A, Wavelength=210 nm

Peak #	RetTime [min]	Type	Width [min]	Area mAU *s	Height [mAU]	Area %
1	25.020	MM	0.7694	2.09946e4	454.77435	97.4337
2	44.729	BB	1.3407	552.97852	4.87536	2.5663
Totals :				2.15475e4	459.64971	

Instrument 1 3/6/2010 5:05:31 PM zzh

Data File D:\LC\DXQ\DATA\DXQ-6-15\DXQ-6-15K4 2009-11-16 14-20-01\SIG1000001.D
Sample Name: dxq-6-15k

```
    ======================================================================
    Acq. Operator   : DXQ                          Seq. Line :   1
    Acq. Instrument : Instrument 1                  Location : Vial 35
    Injection Date  : 11/16/2009 2:21:40 PM              Inj :   1
                                                   Inj Volume : 5 µl
    Acq. Method     : D:\LC\DXQ\data\DXQ-6-15\DXQ-6-15K4 2009-11-16 14-20-01\ADH-10-90-05ML-
                      210NM-60MIN.M
    Last changed    : 11/15/2009 11:35:05 AM by DXQ
    Analysis Method : D:\LC\DXQ\DATA\DXQ-6-15\DXQ-6-15K4 2009-11-16 14-20-01\SIG1000001.D\DA.M (
                      ADH-10-90-05ML-210NM-60MIN.M)
    Last changed    : 3/6/2010 5:10:46 PM by zzh
                      (modified after loading)
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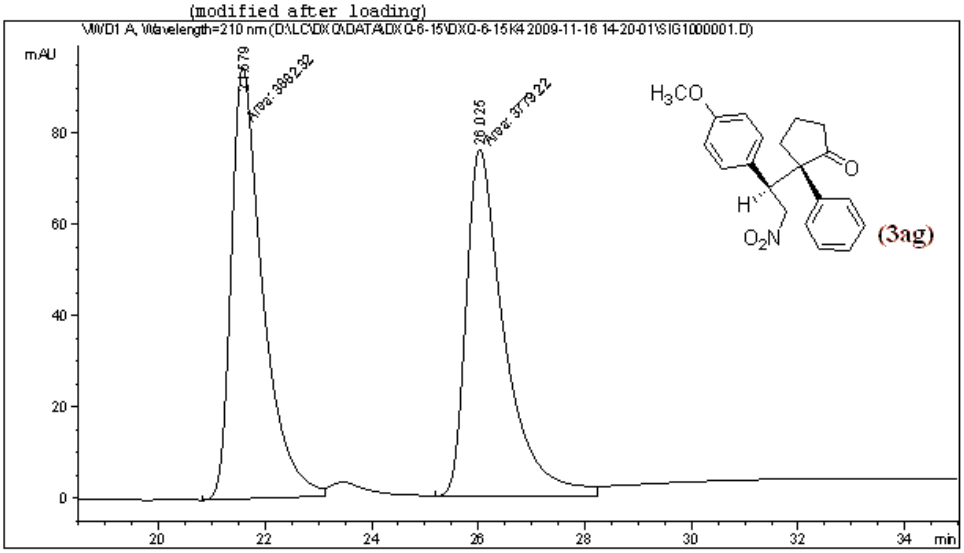


```
    ======================================================================
                              Area Percent Report
    ======================================================================

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


    Signal 1: VWD1 A, Wavelength=210 nm

    Peak RetTime Type  Width     Area      Height     Area
      #   [min]        [min]  mAU   *s   [mAU   ]      %
    ----|-------|----|-------|----------|----------|--------|
       1  21.579 MM    0.6827 3882.32080   94.77477  50.6729
       2  26.025 MM    0.8277 3779.21606   76.10059  49.3271

    Totals :                  7661.53687  170.87536

    ======================================================================
```

Instrument 1 3/6/2010 5:10:52 PM zzh

Data File D:\LC\DXQ\DATA\DXQ-6-7778\DXQ-6-7778 2010-01-21 21-27-37\SIG1000007.D
Sample Name: DXQ-6-78E

```
    ======================================================================
    Acq. Operator   : DXQ                          Seq. Line :   7
    Acq. Instrument : Instrument 1                  Location : Vial 14
    Injection Date  : 1/22/2010 1:06:52 AM               Inj :   1
                                                  Inj Volume : 5 µl
    Acq. Method     : D:\LC\DXQ\data\DXQ-6-7778\DXQ-6-7778 2010-01-21 21-27-37\ADH-10-90-05ML-
                      210NM-40MIN.M
    Last changed    : 1/21/2010 9:15:29 PM by thl
    Analysis Method : D:\LC\DXQ\DATA\DXQ-6-7778\DXQ-6-7778 2010-01-21 21-27-37\SIG1000007.D\DA.M
                      (ADH-10-90-05ML-210NM-40MIN.M)
    Last changed    : 3/6/2010 5:13:40 PM by zzh
                      (modified after loading)
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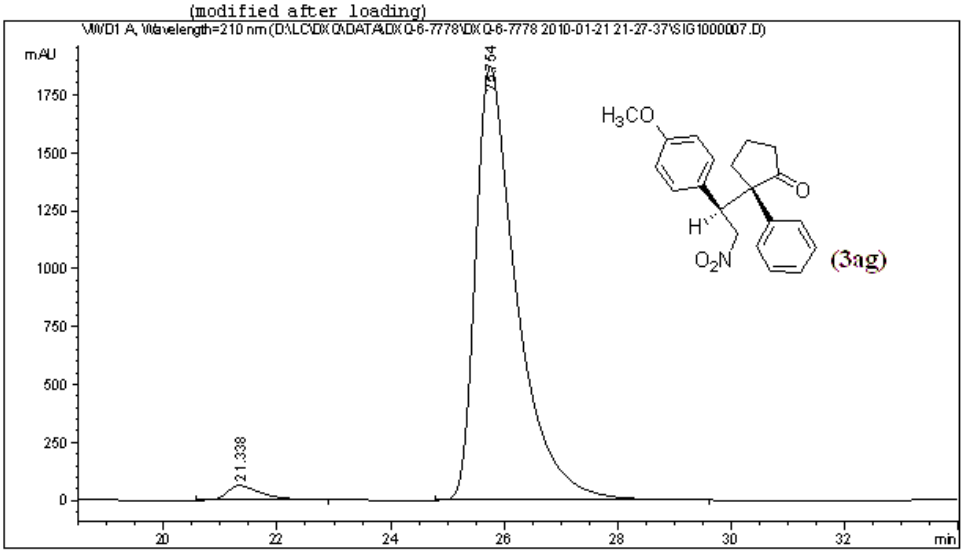



```
    ======================================================================
                            Area Percent Report
    ======================================================================

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


    Signal 1: VWD1 A, Wavelength=210 nm

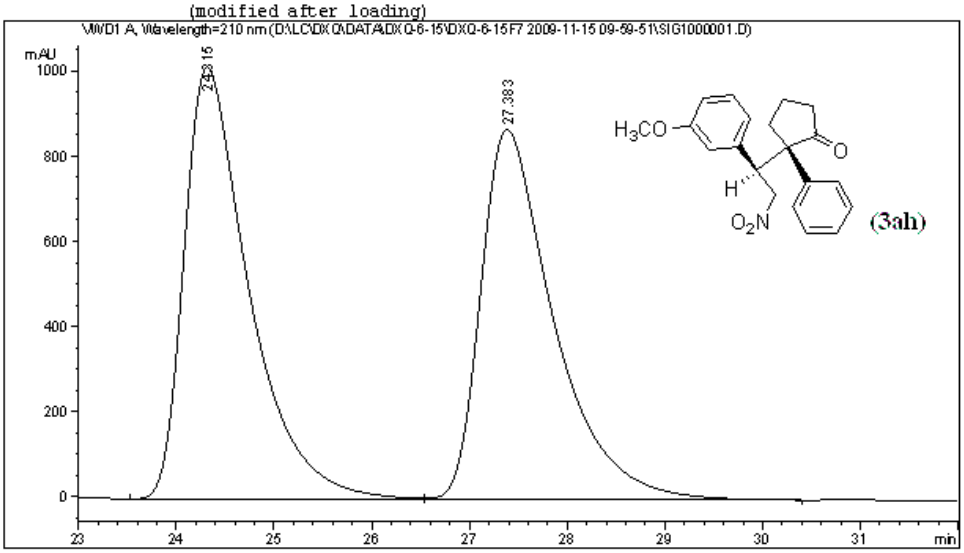
    Peak RetTime Type  Width     Area      Height     Area
      #   [min]        [min]  mAU   *s   [mAU   ]      %
    ----|-------|----|-------|----------|----------|--------|
       1  21.338 BB    0.6066 2622.45264   63.17298   2.7070
       2  25.754 BB    0.7384 9.42550e4  1868.80908  97.2930

    Totals :                  9.68775e4  1931.98206


    ======================================================================
```

Data File D:\LC\DXQ\DATA\DXQ-6-15\DXQ-6-15F7 2009-11-15 09-59-51\SIG1000001.D
Sample Name: DXQ-6-15F

Acq. Operator	: DXQ
Seq. Line	: 1
Acq. Instrument	: Instrument 1
Location	: Vial 6
Injection Date	: 11/15/2009 10:01:26 AM
Inj	: 1
Inj Volume	: 5 µl
Acq. Method	: D:\LC\DXQ\data\DXQ-6-15\DXQ-6-15F7 2009-11-15 09-59-51\ADH-10-90-04ML-210NM.M
Last changed	: 11/15/2009 9:59:12 AM by DXQ
Analysis Method	: D:\LC\DXQ\DATA\DXQ-6-15\DXQ-6-15F7 2009-11-15 09-59-51\SIG1000001.D\DA.M (ADH-10-90-04ML-210NM.M)
Last changed	: 3/6/2010 5:17:33 PM by zzh (modified after loading)



Area Percent Report

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

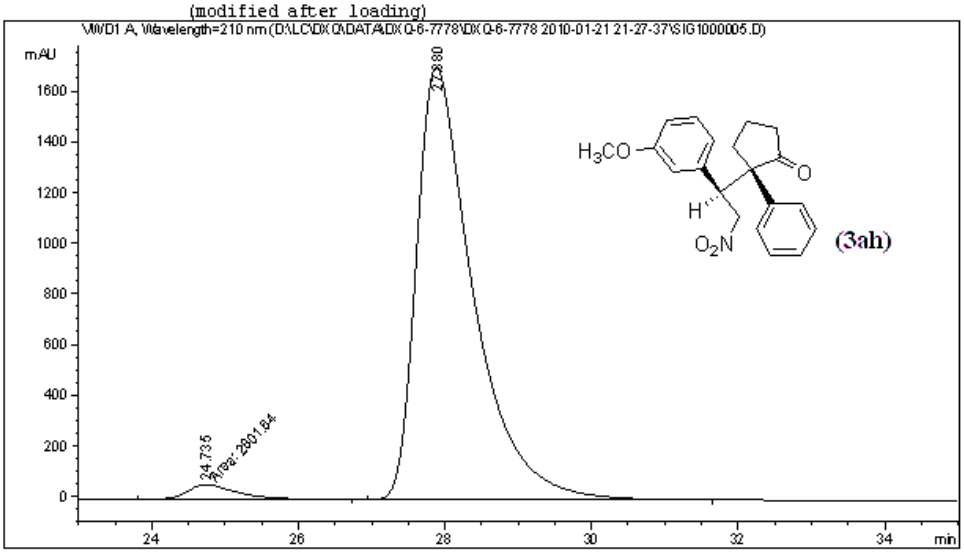
Signal 1: VWD1 A, Wavelength=210 nm

Peak #	RetTime [min]	Type	Width [min]	Area mAU *s	Height [mAU]	Area %
1	24.315	VV	0.6873	4.68318e4	1014.13165	50.9336
2	27.383	VB	0.7657	4.51149e4	869.20813	49.0664
Totals :				9.19466e4	1883.33978	

Instrument 1 3/6/2010 5:17:37 PM zzh

Data File D:\LC\DXQ\DATA\DXQ-6-7778\DXQ-6-7778 2010-01-21 21-27-37\SIG1000005.D
Sample Name: DXQ-6-77C

Acq. Operator	: DXQ	Seq. Line	: 5
Acq. Instrument	: Instrument 1	Location	: Vial 13
Injection Date	: 1/22/2010 12:14:10 AM	Inj	: 1
		Inj Volume	: 5 µl
Acq. Method	: D:\LC\DXQ\data\DXQ-6-7778\DXQ-6-7778 2010-01-21 21-27-37\ADH-10-90-04ML-210NM-40MIN.M		
Last changed	: 1/21/2010 9:13:59 PM by thl		
Analysis Method	: D:\LC\DXQ\DATA\DXQ-6-7778\DXQ-6-7778 2010-01-21 21-27-37\SIG1000005.D\DA.M (ADH-10-90-04ML-210NM-40MIN.M)		
Last changed	: 3/6/2010 5:22:37 PM by zzh (modified after loading)		



Area Percent Report

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

Signal 1: VWD1 A, Wavelength=210 nm

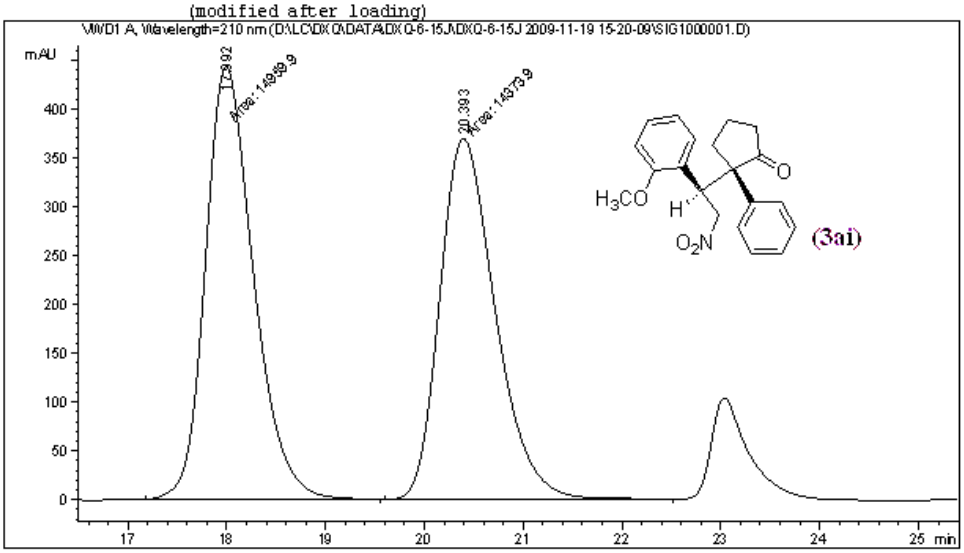
Peak #	RetTime [min]	Type	Width [min]	Area mAU *s	Height [mAU]	Area %
1	24.735	MM	0.7972	2801.63550	58.57557	2.9607
2	27.880	BB	0.7948	9.18254e4	1707.70508	97.0393
Totals :				9.46270e4	1766.28065	

Instrument 1 3/6/2010 5:22:45 PM zzh

Page 1 of 1

Data File D:\LC\DXQ\DATA\DXQ-6-15J\DXQ-6-15J 2009-11-19 15-20-09\SIG1000001.D
Sample Name: dxq-6-15j

```
    ====================================================================
    Acq. Operator   : dxq                          Seq. Line :   1
    Acq. Instrument : Instrument 1                  Location : Vial 8
    Injection Date  : 11/19/2009 3:21:29 PM              Inj :   1
                                                   Inj Volume : 5 µl
    Acq. Method     : D:\LC\DXQ\data\DXQ-6-15J\DXQ-6-15J 2009-11-19 15-20-09\ASH-90-10-05ML-
                      210NM.M
    Last changed    : 11/15/2009 2:15:18 PM by DXQ
    Analysis Method : D:\LC\DXQ\DATA\DXQ-6-15J\DXQ-6-15J 2009-11-19 15-20-09\SIG1000001.D\DA.M (
                      ASH-90-10-05ML-210NM.M)
    Last changed    : 3/6/2010 5:28:33 PM by zzh
                      (modified after loading)
```




```
    ====================================================================
                            Area Percent Report
    ====================================================================

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


    Signal 1: VWD1 A, Wavelength=210 nm

    Peak RetTime Type  Width     Area      Height     Area
      #   [min]        [min]  mAU   *s   [mAU   ]      %
    ----|-------|----|-------|----------|----------|--------|
       1  17.992 MM    0.5639 1.49599e4   442.17953  50.9989
       2  20.393 MM    0.6492 1.43739e4   369.00079  49.0011

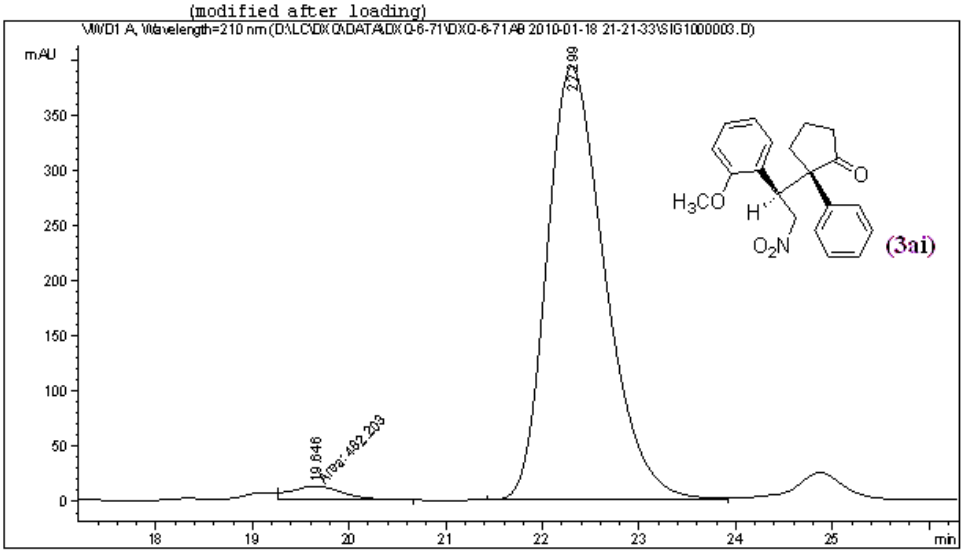
    Totals :                  2.93338e4   811.18033

    ====================================================================
```

Instrument 1 3/6/2010 5:28:37 PM zzh

Data File D:\LC\DXQ\DATA\DXQ-6-71\DXQ-6-71AB 2010-01-18 21-21-33\SIG1000003.D
Sample Name: DXQ-6-71B

Acq. Operator	: DXQ	Seq. Line	: 3
Acq. Instrument	: Instrument 1	Location	: Vial 12
Injection Date	: 1/18/2010 10:25:47 PM	Inj	: 1
		Inj Volume	: 5 µl
Acq. Method	: D:\LC\DXQ\data\DXQ-6-71\DXQ-6-71AB 2010-01-18 21-21-33\ASH-90-10-05ML-210NM-50MIN.M		
Last changed	: 11/11/2009 11:13:52 AM by dxq		
Analysis Method	: D:\LC\DXQ\DATA\DXQ-6-71\DXQ-6-71AB 2010-01-18 21-21-33\SIG1000003.D\DA.M (ASH-90-10-05ML-210NM-50MIN.M)		
Last changed	: 3/6/2010 5:34:51 PM by zzh (modified after loading)		



Area Percent Report

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

Signal 1: VWD1 A, Wavelength=210 nm

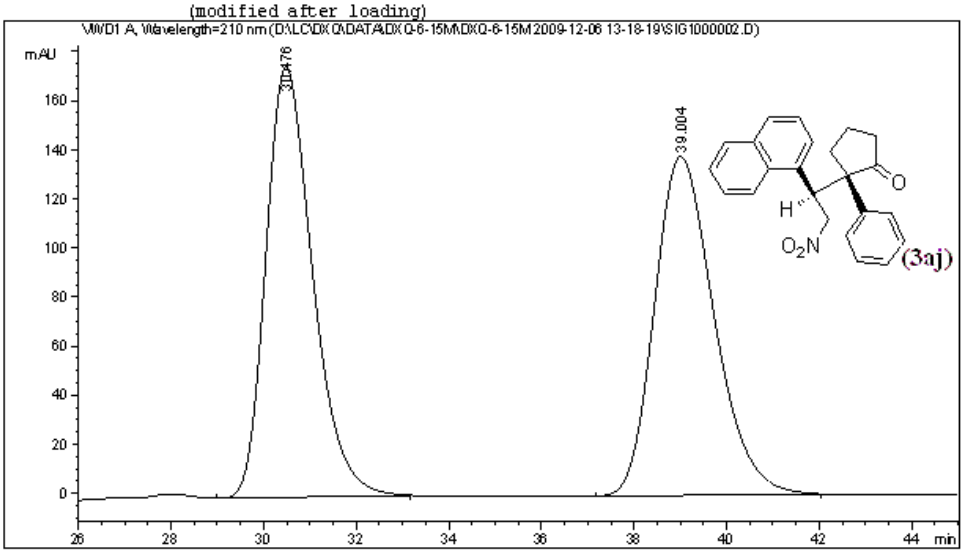
Peak #	RetTime [min]	Type	Width [min]	Area mAU *s	Height [mAU]	Area %
1	19.646	FM	0.6332	482.20276	12.69133	2.8557
2	22.299	BB	0.6468	1.64035e4	392.07166	97.1443
Totals :				1.68857e4	404.76298	

Instrument 1 3/6/2010 5:34:56 PM zzh

Page 1 of 1

Data File D:\LC\DXQ\DATA\DXQ-6-15M\DXQ-6-15M 2009-12-06 13-18-19\SIG1000002.D
Sample Name: dxq-6-15m

Acq. Operator	: dxq
Seq. Line	: 2
Acq. Instrument	: Instrument 1
Location	: Vial 71
Injection Date	: 12/6/2009 1:30:37 PM
Inj	: 1
Inj Volume	: 5 µl
Acq. Method	: D:\LC\DXQ\data\DXQ-6-15M\DXQ-6-15M 2009-12-06 13-18-19\ASH-90-10-05ML-210NM.M
Last changed	: 11/15/2009 2:15:18 PM by DXQ
Analysis Method	: D:\LC\DXQ\DATA\DXQ-6-15M\DXQ-6-15M 2009-12-06 13-18-19\SIG1000002.D\DA.M (ASH-90-10-05ML-210NM.M)
Last changed	: 3/6/2010 6:57:46 PM by zzh (modified after loading)



Area Percent Report

Sorted By	: Signal
Multiplier	: 1.0000
Dilution	: 1.0000

Use Multiplier & Dilution Factor with ISTDs

Signal 1: VWD1 A, Wavelength=210 nm

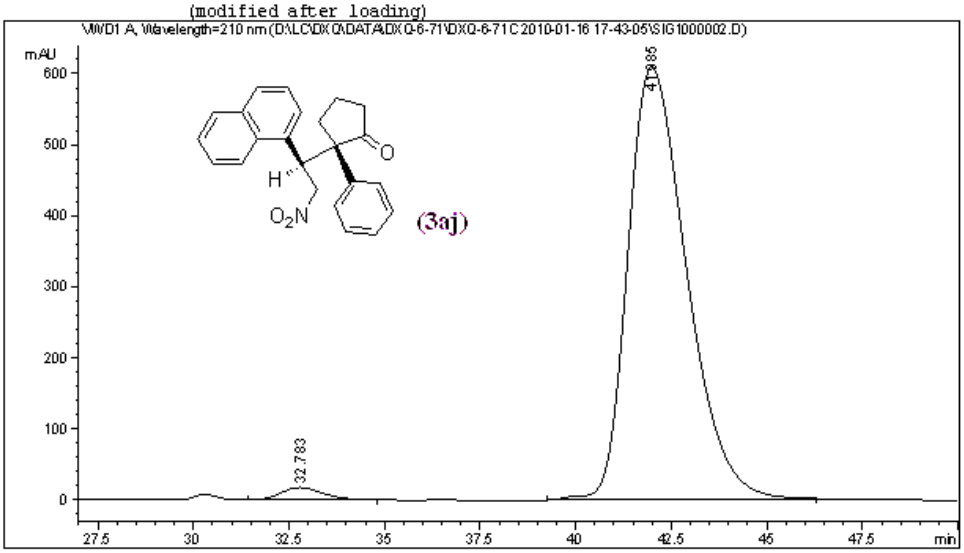
Peak #	RetTime [min]	Type	Width [min]	Area mAU *s	Height [mAU]	Area %
1	30.476	VB	1.0757	1.22863e4	175.23343	49.8351
2	39.004	BB	1.3842	1.23676e4	138.08015	50.1649
Totals :				2.46539e4	313.31358	

Instrument 1 3/6/2010 6:57:50 PM zzh

Page 1 of 1

Data File D:\LC\DXQ\DATA\DXQ-6-71\DXQ-6-71C 2010-01-16 17-43-05\SIG1000002.D
Sample Name: DXQ-6-71C

```
    ====================================================================
    Acq. Operator   : LTL                          Seq. Line :   2
    Acq. Instrument : Instrument 1                  Location : Vial 84
    Injection Date  : 1/16/2010 5:55:34 PM               Inj :   1
                                                   Inj Volume : 5 µl
    Acq. Method     : D:\LC\DXQ\data\DXQ-6-71\DXQ-6-71C 2010-01-16 17-43-05\ASH-90-10-05ML-
                      210NM-50MIN.M
    Last changed    : 11/11/2009 11:13:52 AM by dxq
    Analysis Method : D:\LC\DXQ\DATA\DXQ-6-71\DXQ-6-71C 2010-01-16 17-43-05\SIG1000002.D\DA.M (
                      ASH-90-10-05ML-210NM-50MIN.M)
    Last changed    : 3/6/2010 7:01:45 PM by zzh
                      (modified after loading)
```




```
    ====================================================================
                            Area Percent Report
    ====================================================================

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


    Signal 1: VWD1 A, Wavelength=210 nm

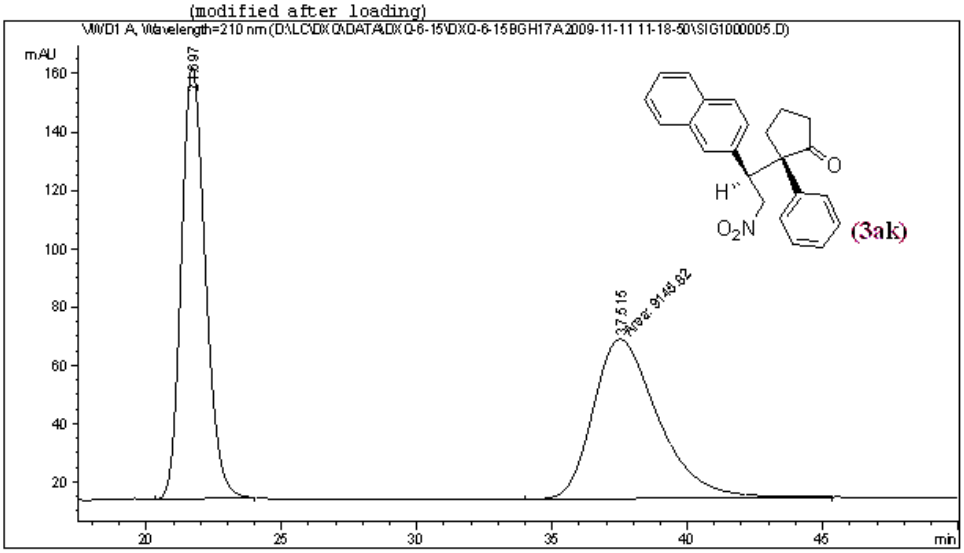
    Peak RetTime Type  Width     Area      Height     Area
      #   [min]        [min]  mAU   *s   [mAU   ]      %
    ----|-------|----|-------|----------|----------|--------|
       1  32.783 BB    1.1383 1318.21973   17.37873   1.9884
       2  41.985 BB    1.6466 6.49763e4   609.70685  98.0116

    Totals :                  6.62945e4   627.08558


    ====================================================================
```

Data File D:\LC\DXQ\DATA\DXQ-6-15\DXQ-6-15BGH17A 2009-11-11 11-18-50\SIG1000005.D
Sample Name: dxq-6-15g

```
    ====================================================================
    Acq. Operator   : dxq                          Seq. Line :   5
    Acq. Instrument : Instrument 1                  Location : Vial 3
    Injection Date  : 11/11/2009 2:25:23 PM              Inj :   1
                                                  Inj Volume : 5 µl
    Acq. Method     : D:\LC\DXQ\data\DXQ-6-15\DXQ-6-15BGH17A 2009-11-11 11-18-50\ASH-90-10-1ML-
                      210NM-50MIN.M
    Last changed    : 11/11/2009 11:14:21 AM by dxq
    Analysis Method : D:\LC\DXQ\DATA\DXQ-6-15\DXQ-6-15BGH17A 2009-11-11 11-18-50\SIG1000005.D\
                      DA.M (ASH-90-10-1ML-210NM-50MIN.M)
    Last changed    : 3/6/2010 7:03:48 PM by zzh
                      (modified after loading)
```




```
    ====================================================================
                          Area Percent Report
    ====================================================================

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


    Signal 1: VWD1 A, Wavelength=210 nm

    Peak RetTime Type  Width     Area      Height     Area
      #   [min]        [min]  mAU   *s    [mAU   ]      %
    ----|-------|----|-------|----------|----------|--------|
       1  21.697 BB    0.9518 9123.03613  148.05623  49.9377
       2  37.515 MM    2.7870 9145.81738   54.69331  50.0623

    Totals :                  1.82689e4   202.74953


    ====================================================================
```

Instrument 1 3/6/2010 7:03:52 PM zzh

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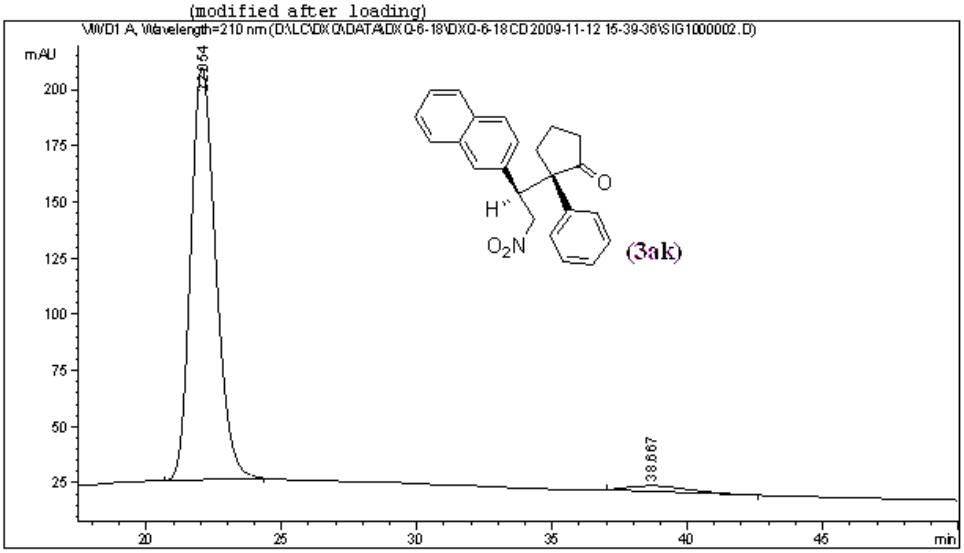
Data File D:\LC\DXQ\DATA\DXQ-6-18\DXQ-6-18CD 2009-11-12 15-39-36\SIG1000002.D
Sample Name: dxq-6-18c

```
    ======================================================================
    Acq. Operator   : dxq                          Seq. Line :   2
    Acq. Instrument : Instrument 1                  Location : Vial 9
    Injection Date  : 11/12/2009 3:52:02 PM              Inj :   1
                                                  Inj Volume : 5 µl
    Acq. Method     : D:\LC\DXQ\data\DXQ-6-18\DXQ-6-18CD 2009-11-12 15-39-36\ASH-90-10-1ML-
                      210NM-50MIN.M
    Last changed    : 11/11/2009 11:14:21 AM by dxq
    Analysis Method : D:\LC\DXQ\DATA\DXQ-6-18\DXQ-6-18CD 2009-11-12 15-39-36\SIG1000002.D\DA.M (
                      ASH-90-10-1ML-210NM-50MIN.M)
    Last changed    : 3/6/2010 7:05:53 PM by zzh
                      (modified after loading)
```




```
    ======================================================================
                              Area Percent Report
    ======================================================================

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


    Signal 1: VWD1 A, Wavelength=210 nm

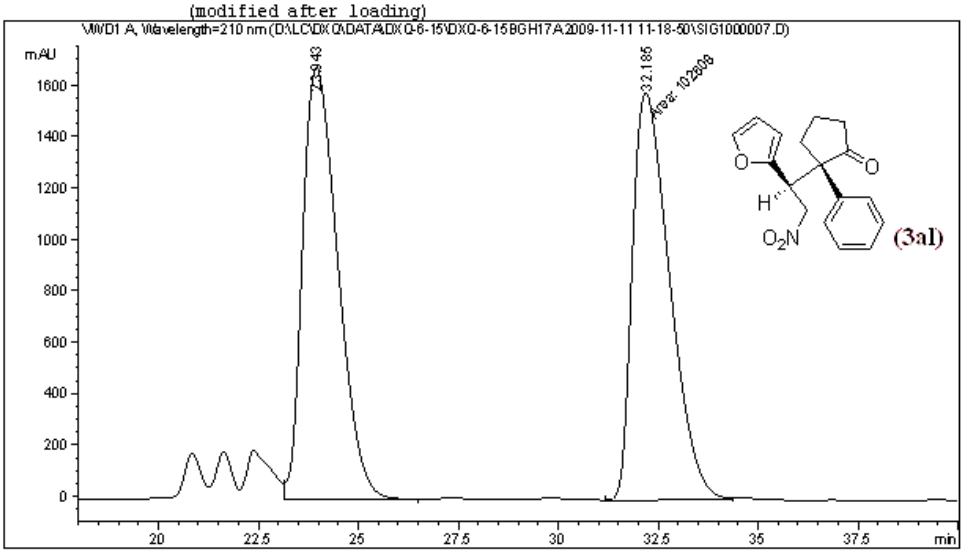
    Peak RetTime Type  Width     Area      Height     Area
      #   [min]        [min]  mAU   *s   [mAU   ]      %
    ----|-------|----|-------|----------|----------|--------|
       1  22.054 BB    0.9864 1.17684e4   183.65190  96.8807
       2  38.667 BB    1.7575  378.91110    2.52999   3.1193

    Totals :                  1.21473e4   186.18189

    ======================================================================
```

Data File D:\LC\DXQ\DATA\DXQ-6-15\DXQ-6-15BGH17A 2009-11-11 11-18-50\SIG1000007.D
Sample Name: dxq-6-15h

```
    ==================================================================
    Acq. Operator   : dxq                          Seq. Line :   7
    Acq. Instrument : Instrument 1                  Location : Vial 4
    Injection Date  : 11/11/2009 3:27:59 PM              Inj :   1
                                                   Inj Volume : 5 µl
    Acq. Method     : D:\LC\DXQ\data\DXQ-6-15\DXQ-6-15BGH17A 2009-11-11 11-18-50\ASH-90-10-05ML-
                      210NM-50MIN.M
    Last changed    : 11/11/2009 11:13:52 AM by dxq
    Analysis Method : D:\LC\DXQ\DATA\DXQ-6-15\DXQ-6-15BGH17A 2009-11-11 11-18-50\SIG1000007.D\
                      DA.M (ASH-90-10-05ML-210NM-50MIN.M)
    Last changed    : 3/6/2010 7:14:52 PM by zzh
                      (modified after loading)
```




```
    ==================================================================
                          Area Percent Report
    ==================================================================

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


    Signal 1: VWD1 A, Wavelength=210 nm

    Peak RetTime Type  Width     Area      Height     Area
      #   [min]        [min]  mAU   *s    [mAU   ]      %
    ----|-------|----|-------|----------|----------|--------|
       1  23.943 VB    0.9687 1.03432e5  1683.41956  50.2001
       2  32.185 MM    1.0788 1.02608e5  1585.25940  49.7999

    Totals :                  2.06040e5  3268.67896

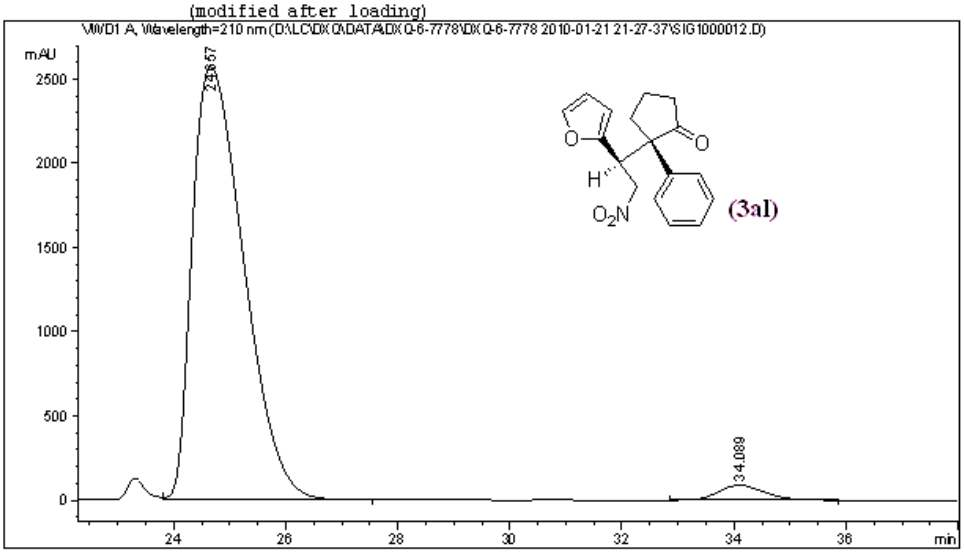

    ==================================================================
```

Instrument 1 3/6/2010 7:14:57 PM zzh

Page 1 of 1

Data File D:\LC\DXQ\DATA\DXQ-6-7778\DXQ-6-7778 2010-01-21 21-27-37\SIG1000012.D
Sample Name: DXQ-6-78G

```
    ====================================================================
    Acq. Operator   : DXQ                          Seq. Line :  12
    Acq. Instrument : Instrument 1                  Location : Vial 18
    Injection Date  : 1/22/2010 4:34:13 AM               Inj :  1
                                                   Inj Volume : 5 µl
    Acq. Method     : D:\LC\DXQ\data\DXQ-6-7778\DXQ-6-7778 2010-01-21 21-27-37\ASH-90-10-05ML-
                      210NM-50MIN.M
    Last changed    : 11/11/2009 11:13:52 AM by dxq
    Analysis Method : D:\LC\DXQ\DATA\DXQ-6-7778\DXQ-6-7778 2010-01-21 21-27-37\SIG1000012.D\DA.M
                      (ASH-90-10-05ML-210NM-50MIN.M)
    Last changed    : 3/6/2010 7:56:18 PM by zzh
                      (modified after loading)
```




```
    ====================================================================
                          Area Percent Report
    ====================================================================

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


    Signal 1: VWD1 A, Wavelength=210 nm

    Peak RetTime Type  Width     Area      Height     Area
      #   [min]        [min]  mAU   *s   [mAU   ]      %
    ----|-------|----|-------|----------|----------|--------|
       1  24.657 VB    1.0048 1.64304e5  2570.87036  96.9882
       2  34.089 BB    0.9008 5102.16748   88.16350   3.0118

    Totals :                  1.69406e5  2659.03386

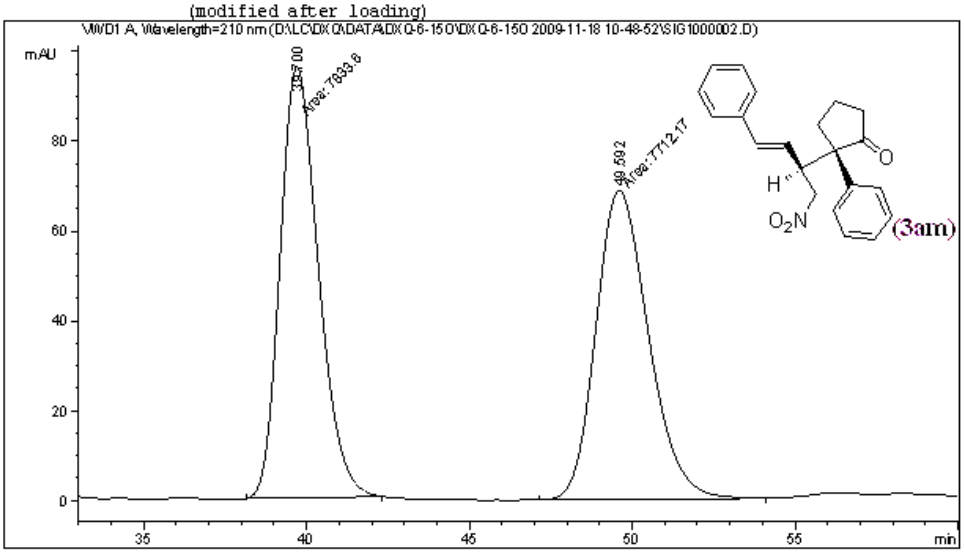

    ====================================================================
```

Instrument 1 3/6/2010 7:56:22 PM zzh

Page 1 of 1

Data File D:\LC\DXQ\DATA\DXQ-6-150\DXQ-6-150 2009-11-18 10-48-52\SIG1000002.D
Sample Name: DXQ-6-150

Acq. Operator	: DXQ	Seq. Line	: 2
Acq. Instrument	: Instrument 1	Location	: Vial 3
Injection Date	: 11/18/2009 11:01:11 AM	Inj	: 1
		Inj Volume	: 5 µl
Acq. Method	: D:\LC\DXQ\data\DXQ-6-150\DXQ-6-150 2009-11-18 10-48-52\ASH-90-10-05ML-210NM.M		
Last changed	: 11/15/2009 2:15:18 PM by DXQ		
Analysis Method	: D:\LC\DXQ\DATA\DXQ-6-150\DXQ-6-150 2009-11-18 10-48-52\SIG1000002.D\DA.M (ASH-90-10-05ML-210NM.M)		
Last changed	: 3/6/2010 7:19:52 PM by zzh (modified after loading)		



Area Percent Report

Sorted By	:	Signal
Multiplier	:	1.0000
Dilution	:	1.0000

Use Multiplier & Dilution Factor with ISTDs

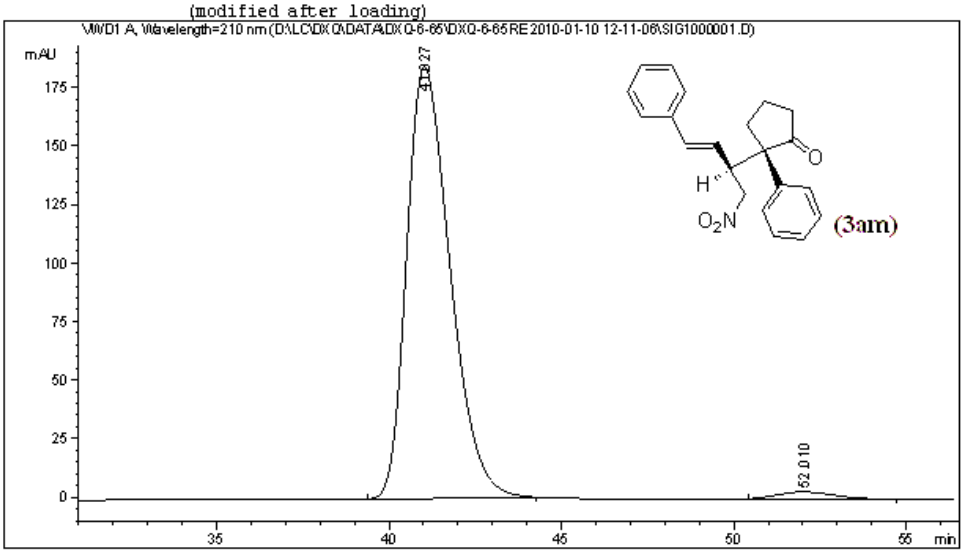
Signal 1: VWD1 A, Wavelength=210 nm

Peak #	RetTime [min]	Type	Width [min]	Area mAU *s	Height [mAU]	Area %
1	39.700	MM	1.3613	7833.60449	95.91184	50.3906
2	49.592	MM	1.8669	7712.17090	68.85076	49.6094
Totals :				1.55458e4	164.76260	

Instrument 1 3/6/2010 7:19:57 PM zzh

Data File D:\LC\DXQ\DATA\DXQ-6-65\DXQ-6-65RE 2010-01-10 12-11-06\SIG1000001.D
Sample Name: dxq-6-65

```
    ====================================================================
    Acq. Operator   : dxq                          Seq. Line :   1
    Acq. Instrument : Instrument 1                  Location : Vial 23
    Injection Date  : 1/10/2010 12:12:24 PM              Inj :   1
                                                   Inj Volume : 5 µl
    Acq. Method     : D:\LC\DXQ\data\DXQ-6-65\DXQ-6-65RE 2010-01-10 12-11-06\ASH-90-10-05ML-
                      210NM-70MIN.M
    Last changed    : 1/10/2010 10:46:15 AM by LTL
    Analysis Method : D:\LC\DXQ\DATA\DXQ-6-65\DXQ-6-65RE 2010-01-10 12-11-06\SIG1000001.D\DA.M (
                      ASH-90-10-05ML-210NM-70MIN.M)
    Last changed    : 3/6/2010 7:21:57 PM by zzh
                      (modified after loading)
```




```
    ====================================================================
                            Area Percent Report
    ====================================================================

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


    Signal 1: VWD1 A, Wavelength=210 nm

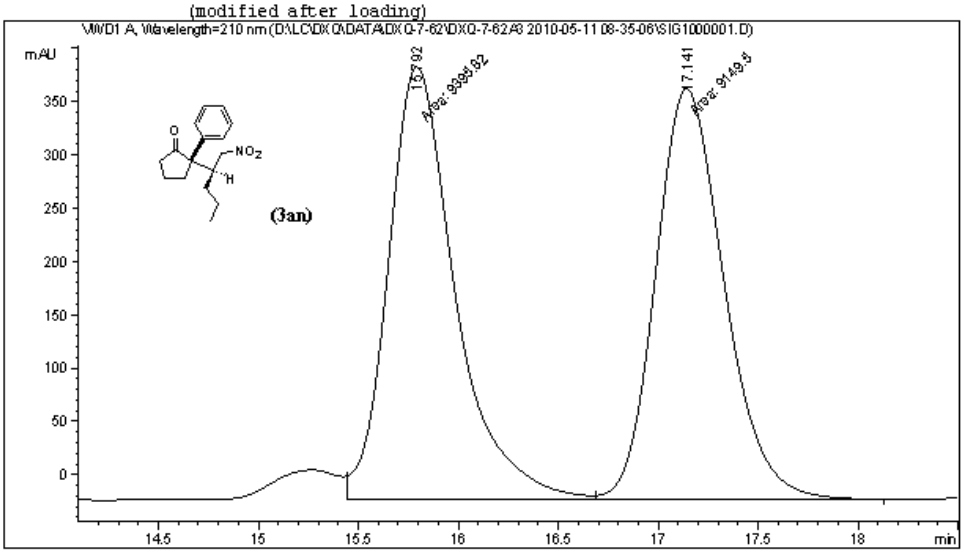
    Peak RetTime Type  Width     Area      Height     Area
      #   [min]        [min]  mAU   *s   [mAU   ]      %
    ----|-------|----|-------|----------|----------|--------|
       1  41.027 BB    1.3642 1.61375e4   184.20241  97.9419
       2  52.010 BB    1.3189  339.11221    3.01970   2.0581

    Totals :                  1.64766e4   187.22211


    ====================================================================
```

Data File D:\LC\DXQ\DATA\DXQ-7-62\DXQ-7-62A3 2010-05-11 08-35-06\SIG1000001.D
Sample Name: DXQ-7-62

```
    ====================================================================
    Acq. Operator   : DXQ                          Seq. Line :   1
    Acq. Instrument : Instrument 1                  Location : Vial 32
    Injection Date  : 5/11/2010 8:36:31 AM               Inj :   1
                                                   Inj Volume : 5 µl
    Acq. Method     : D:\LC\DXQ\data\DXQ-7-62\DXQ-7-62A3 2010-05-11 08-35-06\ODH-95-5-05ML-
                      210NM.M
    Last changed    : 5/11/2010 8:34:41 AM by DXQ
    Analysis Method : D:\LC\DXQ\DATA\DXQ-7-62\DXQ-7-62A3 2010-05-11 08-35-06\SIG1000001.D\DA.M (
                      ODH-95-5-05ML-210NM.M)
    Last changed    : 5/14/2010 7:44:34 PM by LTL
                      (modified after loading)
```




```
    ====================================================================
                            Area Percent Report
    ====================================================================

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


    Signal 1: VWD1 A, Wavelength=210 nm

    Peak RetTime Type  Width     Area      Height     Area
      #   [min]        [min]  mAU   *s   [mAU   ]      %
    ----|-------|----|-------|----------|----------|--------|
       1  15.792 MF    0.3862 9395.82129  405.44400  50.6641
       2  17.141 FM    0.3959 9149.50391  385.14966  49.3359

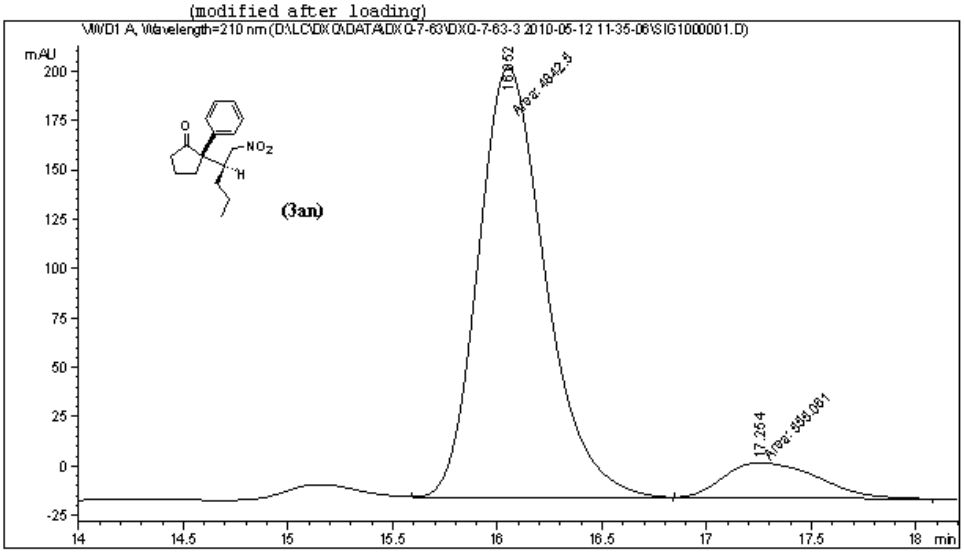
    Totals :                  1.85453e4   790.59366

    ====================================================================
```

Instrument 1 5/14/2010 7:44:38 PM LTL

Data File D:\LC\DXQ\DATA\DXQ-7-63\DXQ-7-63-3 2010-05-12 11-35-06\SIG1000001.D
Sample Name: dxq-7-63

```
    ====================================================================
    Acq. Operator   : dxq                          Seq. Line :   1
    Acq. Instrument : Instrument 1                  Location : Vial 35
    Injection Date  : 5/12/2010 11:36:25 AM              Inj :   1
                                                   Inj Volume : 5 µl
    Acq. Method     : D:\LC\DXQ\data\DXQ-7-63\DXQ-7-63-3 2010-05-12 11-35-06\ODH-95-5-05ML-
                      210NM-30MIN.M
    Last changed    : 5/11/2010 9:31:10 AM by DXQ
    Analysis Method : D:\LC\DXQ\DATA\DXQ-7-63\DXQ-7-63-3 2010-05-12 11-35-06\SIG1000001.D\DA.M (
                      ODH-95-5-05ML-210NM-30MIN.M)
    Last changed    : 5/14/2010 7:47:56 PM by LTL
                      (modified after loading)
```




```
    ====================================================================
                            Area Percent Report
    ====================================================================

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


    Signal 1: VWD1 A, Wavelength=210 nm

    Peak RetTime Type  Width     Area      Height     Area
      #   [min]        [min]  mAU   *s   [mAU   ]      %
    ----|-------|----|-------|----------|----------|--------|
       1  16.052 MM    0.3705 4842.49805  217.81058  89.7165
       2  17.254 MM    0.5203  555.06055   17.77898  10.2835

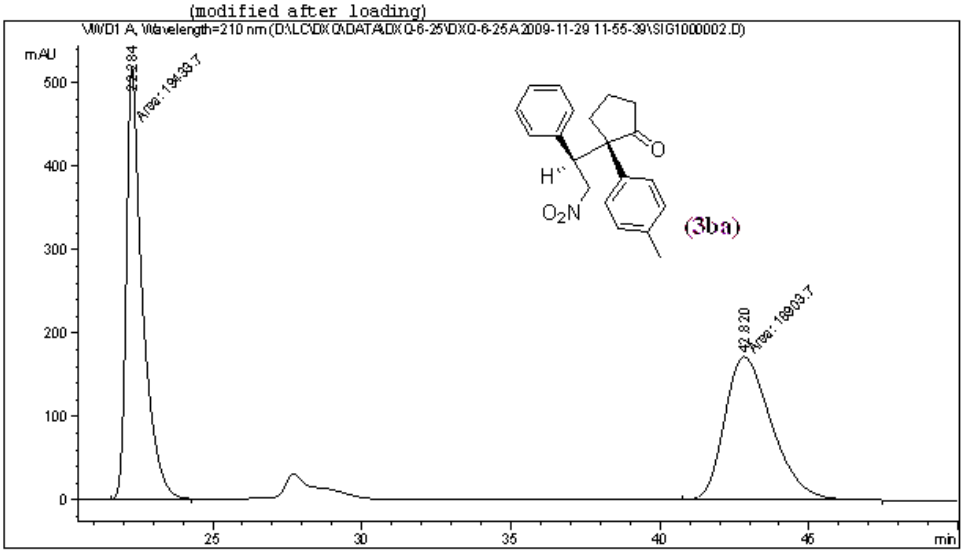
    Totals :                  5397.55859  235.58955

    ====================================================================
```

Instrument 1 5/14/2010 7:48:01 PM LTL

Data File D:\LC\DXQ\DATA\DXQ-6-25\DXQ-6-25A 2009-11-29 11-55-39\SIG1000002.D
Sample Name: dxq-6-25a-rac2

```
    ====================================================================
    Acq. Operator   : dxq                          Seq. Line :   2
    Acq. Instrument : Instrument 1                  Location : Vial 21
    Injection Date  : 11/29/2009 12:08:00 PM             Inj :   1
                                                   Inj Volume : 5 µl
    Acq. Method     : D:\LC\DXQ\data\DXQ-6-25\DXQ-6-25A 2009-11-29 11-55-39\ASH-90-10-05ML-
                      210NM-70MIN.M
    Last changed    : 11/15/2009 3:52:39 PM by DXQ
    Analysis Method : D:\LC\DXQ\DATA\DXQ-6-25\DXQ-6-25A 2009-11-29 11-55-39\SIG1000002.D\DA.M (
                      ASH-90-10-05ML-210NM-70MIN.M)
    Last changed    : 3/6/2010 7:25:06 PM by zzh
                      (modified after loading)
```




```
    ====================================================================
                           Area Percent Report
    ====================================================================

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


    Signal 1: VWD1 A, Wavelength=210 nm

    Peak RetTime Type  Width     Area      Height     Area
      #   [min]        [min]  mAU   *s   [mAU   ]      %
    ----|-------|----|-------|----------|----------|-------|
       1  22.284 MM    0.6257 1.94337e4   517.69098  50.6913
       2  42.820 MM    1.8363 1.89037e4   171.57135  49.3087

    Totals :                  3.83373e4   689.26233


    ====================================================================
```

Instrument 1 3/6/2010 7:25:15 PM zzh

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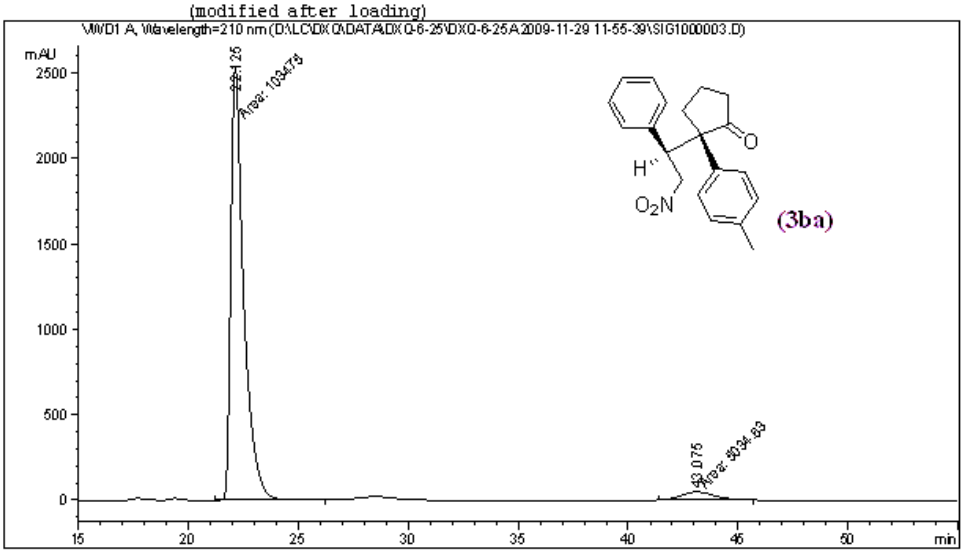
Data File D:\LC\DXQ\DATA\DXQ-6-25\DXQ-6-25A 2009-11-29 11-55-39\SIG1000003.D
Sample Name: dxq-6-25a

```
    ==================================================================
    Acq. Operator   : dxq                          Seq. Line :   3
    Acq. Instrument : Instrument 1                  Location : Vial 22
    Injection Date  : 11/29/2009 1:19:28 PM              Inj :   1
                                                   Inj Volume : 5 µl
    Acq. Method     : D:\LC\DXQ\data\DXQ-6-25\DXQ-6-25A 2009-11-29 11-55-39\ASH-90-10-05ML-
                      210NM-70MIN.M
    Last changed    : 11/15/2009 3:52:39 PM by DXQ
    Analysis Method : D:\LC\DXQ\DATA\DXQ-6-25\DXQ-6-25A 2009-11-29 11-55-39\SIG1000003.D\DA.M (
                      ASH-90-10-05ML-210NM-70MIN.M)
    Last changed    : 3/6/2010 7:27:34 PM by zzh
                      (modified after loading)
```




```
    ==================================================================
                          Area Percent Report
    ==================================================================

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


    Signal 1: VWD1 A, Wavelength=210 nm

    Peak RetTime Type  Width     Area      Height     Area
      #   [min]        [min]  mAU   *s   [mAU   ]      %
    ----|-------|----|-------|----------|----------|--------|
       1  22.125 MM    0.6800 1.03475e5  2536.19287  95.3602
       2  43.075 MM    1.7690 5034.62695   47.43297   4.6398

    Totals :                  1.08510e5  2583.62584


    ==================================================================
```

Instrument 1 3/6/2010 7:27:38 PM zzh

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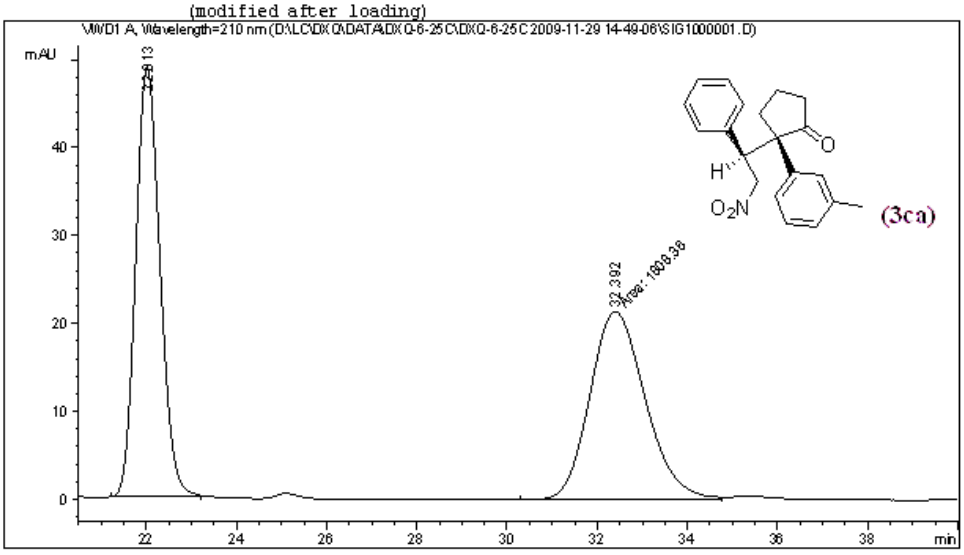
Data File D:\LC\DXQ\DATA\DXQ-6-25C\DXQ-6-25C 2009-11-29 14-49-06\SIG1000001.D
Sample Name: dxq-6-25c-racl

```
    ====================================================================
    Acq. Operator   : dxq                          Seq. Line :   1
    Acq. Instrument : Instrument 1                  Location : Vial 24
    Injection Date  : 11/29/2009 2:50:45 PM              Inj :   1
                                                   Inj Volume : 5 µl
    Acq. Method     : D:\LC\DXQ\data\DXQ-6-25C\DXQ-6-25C 2009-11-29 14-49-06\ASH-90-10-05ML-
                      210NM.M
    Last changed    : 11/15/2009 2:15:18 PM by DXQ
    Analysis Method : D:\LC\DXQ\DATA\DXQ-6-25C\DXQ-6-25C 2009-11-29 14-49-06\SIG1000001.D\DA.M (
                      ASH-90-10-05ML-210NM.M)
    Last changed    : 3/6/2010 7:29:41 PM by zzh
                      (modified after loading)
```




```
    ====================================================================
                            Area Percent Report
    ====================================================================

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


    Signal 1: VWD1 A, Wavelength=210 nm

    Peak RetTime Type  Width     Area      Height     Area
      #   [min]        [min]  mAU   *s    [mAU   ]      %
    ----|-------|----|-------|----------|----------|-------|
       1  22.013 BB    0.5790 1821.51965   48.87113  50.1813
       2  32.392 MM    1.4115 1808.36011   21.35329  49.8187

    Totals :                  3629.87976   70.22442


    ====================================================================
```

Instrument 1 3/6/2010 7:29:45 PM zzh

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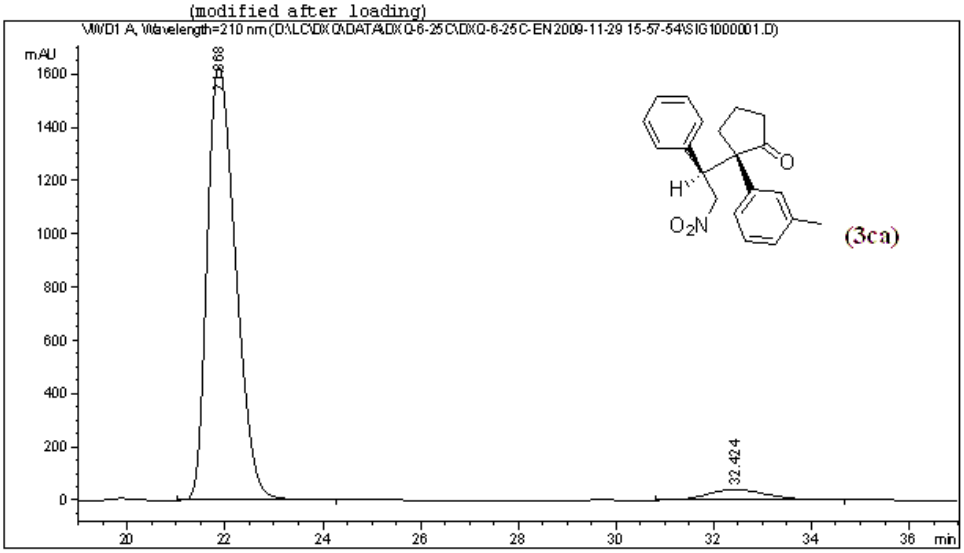
Data File D:\LC\DXQ\DATA\DXQ-6-25C\DXQ-6-25C-EN 2009-11-29 15-57-54\SIG1000001.D
Sample Name: dxq-6-25c

```
    ====================================================================
    Acq. Operator   : dxq                          Seq. Line :   1
    Acq. Instrument : Instrument 1                  Location : Vial 25
    Injection Date  : 11/29/2009 3:59:21 PM              Inj :   1
                                                   Inj Volume : 5 µl
    Acq. Method     : D:\LC\DXQ\data\DXQ-6-25C\DXQ-6-25C-EN 2009-11-29 15-57-54\ASH-90-10-05ML-
                      210NM-40MIN.M
    Last changed    : 11/29/2009 11:50:33 AM by dxq
    Analysis Method : D:\LC\DXQ\DATA\DXQ-6-25C\DXQ-6-25C-EN 2009-11-29 15-57-54\SIG1000001.D\DA.
                      M (ASH-90-10-05ML-210NM-40MIN.M)
    Last changed    : 3/6/2010 7:31:48 PM by zzh
                      (modified after loading)
```




```
    ====================================================================
                            Area Percent Report
    ====================================================================

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


    Signal 1: VWD1 A, Wavelength=210 nm

    Peak RetTime Type  Width     Area      Height     Area
      #   [min]        [min]  mAU   *s   [mAU   ]      %
    ----|-------|----|-------|----------|----------|--------|
       1  21.868 BB    0.6355 6.58475e4  1626.14417  95.1326
       2  32.424 BB    1.2714 3369.03711   40.03880   4.8674

    Totals :                  6.92166e4  1666.18296


    ====================================================================
```

Instrument 1 3/6/2010 7:31:51 PM zzh

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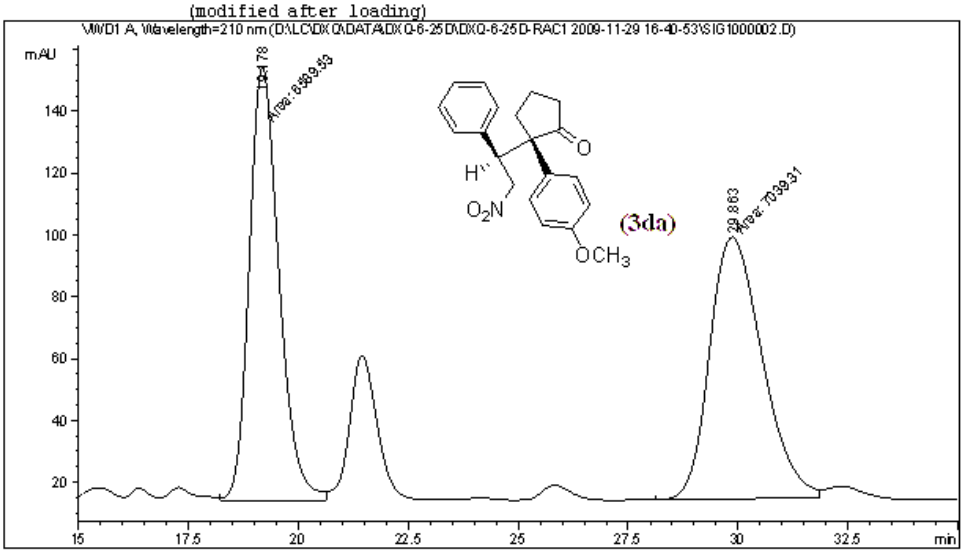
Data File D:\LC\DXQ\DATA\DXQ-6-25D\DXQ-6-25D-RAC1 2009-11-29 16-40-53\SIG1000002.D
Sample Name: dxq-6-25d-rac1

```
    ====================================================================
    Acq. Operator   : dxq                          Seq. Line :   2
    Acq. Instrument : Instrument 1                  Location : Vial 26
    Injection Date  : 11/29/2009 4:53:17 PM              Inj :   1
                                                   Inj Volume : 5 µl
    Acq. Method     : D:\LC\DXQ\data\DXQ-6-25D\DXQ-6-25D-RAC1 2009-11-29 16-40-53\ASH-90-10-1ML-
                      210NM.M
    Last changed    : 11/29/2009 4:38:24 PM by dxq
    Analysis Method : D:\LC\DXQ\DATA\DXQ-6-25D\DXQ-6-25D-RAC1 2009-11-29 16-40-53\SIG1000002.D\
                      DA.M (ASH-90-10-1ML-210NM.M)
    Last changed    : 3/6/2010 7:35:42 PM by zzh
                      (modified after loading)
```




```
    ====================================================================
                            Area Percent Report
    ====================================================================

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


    Signal 1: VWD1 A, Wavelength=210 nm

    Peak RetTime Type  Width     Area      Height     Area
      #   [min]        [min]  mAU   *s   [mAU   ]      %
    ----|-------|----|-------|----------|----------|--------|
       1  19.178 MM    0.7835 6589.53223  140.16492  48.3499
       2  29.863 MM    1.3856 7039.31055   84.67238  51.6501

    Totals :                  1.36288e4   224.83730

    ====================================================================
```

Instrument 1 3/6/2010 7:35:51 PM zzh

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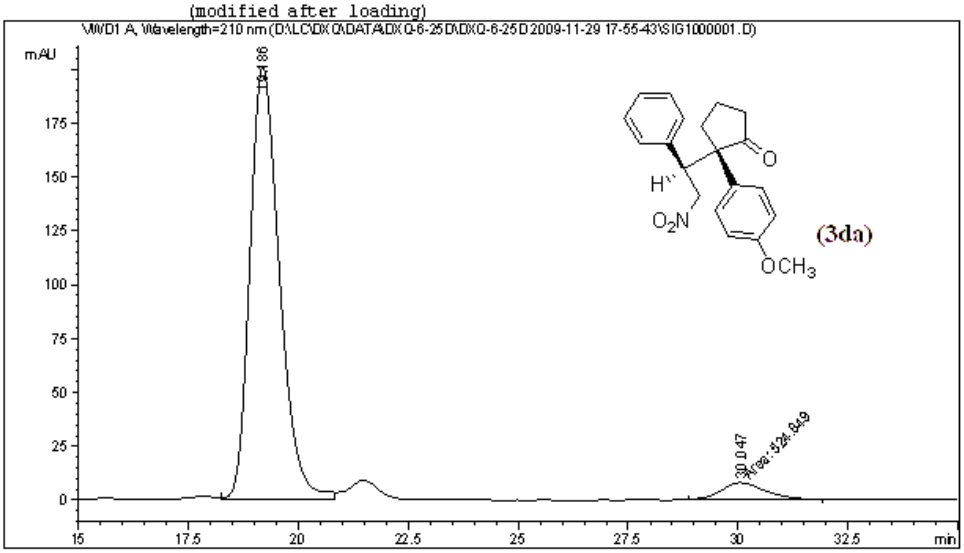
Data File D:\LC\DXQ\DATA\DXQ-6-25D\DXQ-6-25D 2009-11-29 17-55-43\SIG1000001.D
Sample Name: dxq-6-25d

```
    ====================================================================
    Acq. Operator   : dxq                          Seq. Line :   1
    Acq. Instrument : Instrument 1                  Location : Vial 27
    Injection Date  : 11/29/2009 5:57:01 PM              Inj :   1
                                                  Inj Volume : 5 µl
    Acq. Method     : D:\LC\DXQ\data\DXQ-6-25D\DXQ-6-25D 2009-11-29 17-55-43\ASH-90-10-1ML-
                      210NM-35MIN.M
    Last changed    : 11/29/2009 5:54:20 PM by dxq
    Analysis Method : D:\LC\DXQ\DATA\DXQ-6-25D\DXQ-6-25D 2009-11-29 17-55-43\SIG1000001.D\DA.M (
                      ASH-90-10-1ML-210NM-35MIN.M)
    Last changed    : 3/6/2010 7:38:00 PM by zzh
                      (modified after loading)
```




```
    ====================================================================
                           Area Percent Report
    ====================================================================

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


    Signal 1: VWD1 A, Wavelength=210 nm

    Peak RetTime Type  Width     Area      Height     Area
      #   [min]        [min]  mAU   *s    [mAU   ]      %
    ----|-------|----|-------|----------|----------|--------|
       1  19.186 VB    0.7235 9392.25391  200.72893  94.7076
       2  30.047 MM    1.1372  524.84930    7.69234   5.2924

    Totals :                  9917.10321  208.42126


    ====================================================================
```

Instrument 1 3/6/2010 7:38:03 PM zzh

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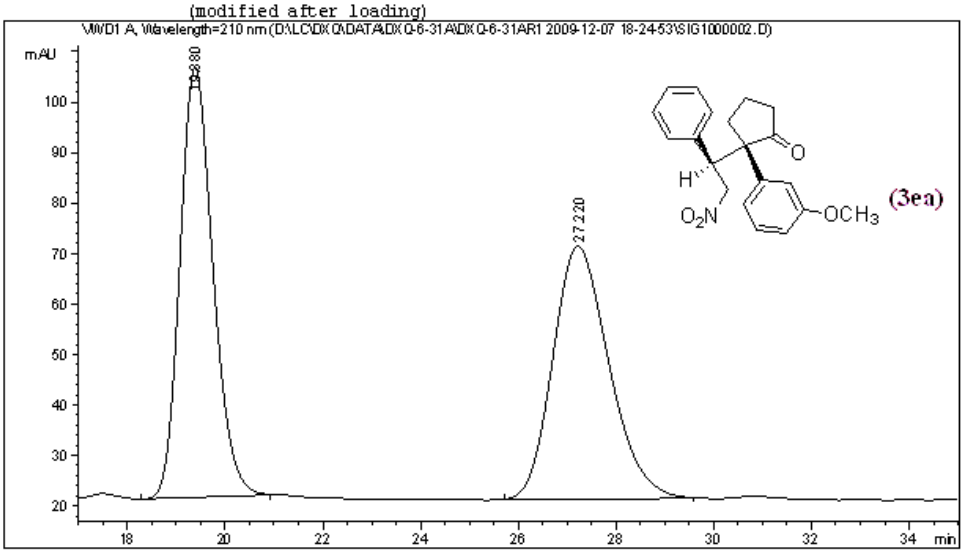
Data File D:\LC\DXQ\DATA\DXQ-6-31A\DXQ-6-31AR1 2009-12-07 18-24-53\SIG1000002.D
Sample Name: dxq-6-31ar

```
    ====================================================================
    Acq. Operator   : dxq                          Seq. Line :   2
    Acq. Instrument : Instrument 1                  Location : Vial 73
    Injection Date  : 12/7/2009 6:37:11 PM               Inj :   1
                                                   Inj Volume : 5 µl
    Acq. Method     : D:\LC\DXQ\data\DXQ-6-31A\DXQ-6-31AR1 2009-12-07 18-24-53\ASH-90-10-1ML-
                      210NM.M
    Last changed    : 11/29/2009 4:38:24 PM by dxq
    Analysis Method : D:\LC\DXQ\DATA\DXQ-6-31A\DXQ-6-31AR1 2009-12-07 18-24-53\SIG1000002.D\DA.M
                      (ASH-90-10-1ML-210NM.M)
    Last changed    : 3/6/2010 7:39:32 PM by zzh
                      (modified after loading)
```




```
    ====================================================================
                          Area Percent Report
    ====================================================================

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


    Signal 1: VWD1 A, Wavelength=210 nm

    Peak RetTime Type  Width     Area      Height     Area
      #   [min]        [min]  mAU   *s   [mAU   ]      %
    ----|-------|----|-------|----------|----------|--------|
       1  19.380 VB    0.7237 3990.17676   85.25044  50.0049
       2  27.220 BB    1.2213 3989.38892   50.04213  49.9951

    Totals :                  7979.56567  135.29257


    ====================================================================
```

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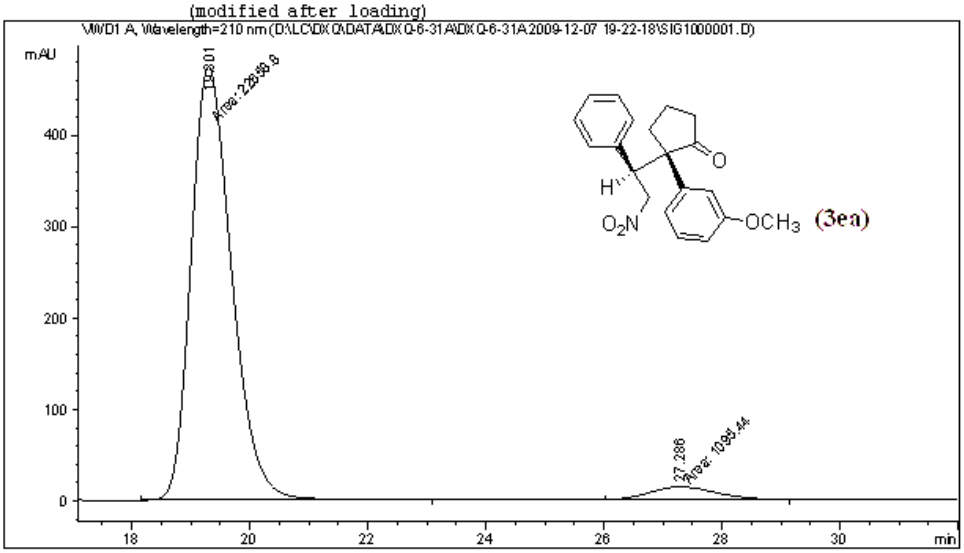
Data File D:\LC\DXQ\DATA\DXQ-6-31A\DXQ-6-31A 2009-12-07 19-22-18\SIG1000001.D
Sample Name: dxq-6-31a

```
    ====================================================================
    Acq. Operator   : dxq                          Seq. Line :   1
    Acq. Instrument : Instrument 1                  Location : Vial 74
    Injection Date  : 12/7/2009 7:23:52 PM               Inj :   1
                                                  Inj Volume : 5 µl
    Acq. Method     : D:\LC\DXQ\data\DXQ-6-31A\DXQ-6-31A 2009-12-07 19-22-18\ASH-90-10-1ML-
                      210NM-35MIN.M
    Last changed    : 11/29/2009 5:54:20 PM by dxq
    Analysis Method : D:\LC\DXQ\DATA\DXQ-6-31A\DXQ-6-31A 2009-12-07 19-22-18\SIG1000001.D\DA.M (
                      ASH-90-10-1ML-210NM-35MIN.M)
    Last changed    : 3/6/2010 7:41:41 PM by zzh
                      (modified after loading)
```




```
    ====================================================================
                            Area Percent Report
    ====================================================================

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


    Signal 1: VWD1 A, Wavelength=210 nm

    Peak RetTime Type  Width     Area      Height     Area
      #   [min]        [min]  mAU   *s    [mAU   ]      %
    ----|-------|----|-------|----------|----------|--------|
       1  19.301 MM    0.7967 2.26588e4   474.03827  95.3884
       2  27.286 MM    1.2893 1095.44360   14.16098   4.6116

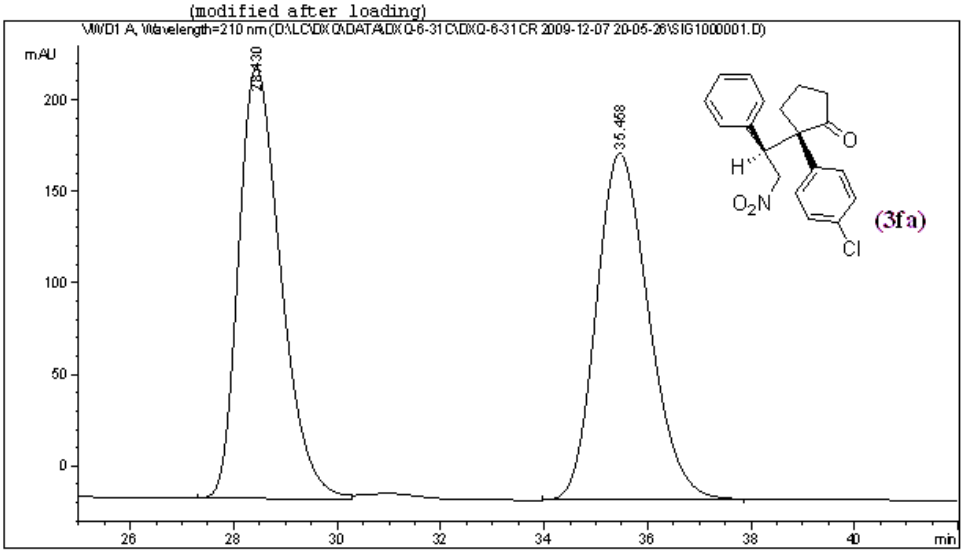
    Totals :                  2.37542e4   488.19925


    ====================================================================
```

Instrument 1 3/6/2010 7:42:23 PM zzh

Data File D:\LC\DXQ\DATA\DXQ-6-31C\DXQ-6-31CR 2009-12-07 20-05-26\SIG1000001.D
Sample Name: dxq-6-31cr1

Acq. Operator	: dxq
Seq. Line	: 1
Acq. Instrument	: Instrument 1
Location	: Vial 75
Injection Date	: 12/7/2009 8:06:48 PM
Inj	: 1
Inj Volume	: 5 µl
Acq. Method	: D:\LC\DXQ\data\DXQ-6-31C\DXQ-6-31CR 2009-12-07 20-05-26\ASH-90-10-05ML-210NM.M
Last changed	: 11/15/2009 2:15:18 PM by DXQ
Analysis Method	: D:\LC\DXQ\DATA\DXQ-6-31C\DXQ-6-31CR 2009-12-07 20-05-26\SIG1000001.D\DA.M (ASH-90-10-05ML-210NM.M)
Last changed	: 3/6/2010 7:44:15 PM by zzh (modified after loading)



Area Percent Report

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

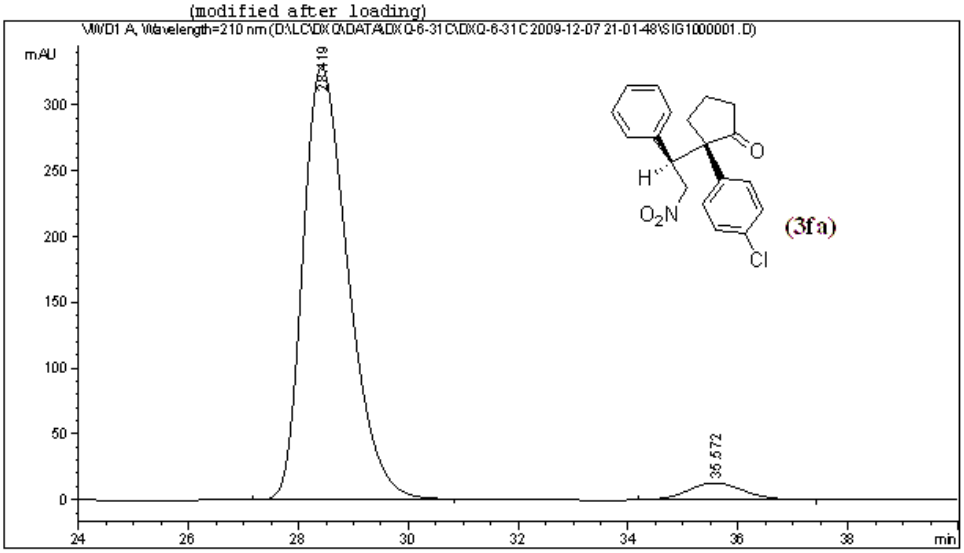
Signal 1: VWD1 A, Wavelength=210 nm

Peak #	RetTime [min]	Type	Width [min]	Area mAU *s	Height [mAU]	Area %
1	28.430	BB	0.8726	1.33339e4	235.69209	50.0522
2	35.458	BB	1.0926	1.3306le4	189.23239	49.9478
Totals :				2.66400e4	424.92448	

Instrument 1 3/6/2010 7:44:18 PM zzh

Data File D:\LC\DXQ\DATA\DXQ-6-31C\DXQ-6-31C 2009-12-07 21-01-48\SIG1000001.D
Sample Name: dxq-6-31c

```
    ====================================================================
    Acq. Operator   : dxq                          Seq. Line :   1
    Acq. Instrument : Instrument 1                  Location : Vial 76
    Injection Date  : 12/7/2009 9:03:10 PM               Inj :   1
                                                  Inj Volume : 5 µl
    Acq. Method     : D:\LC\DXQ\data\DXQ-6-31C\DXQ-6-31C 2009-12-07 21-01-48\ASH-90-10-05ML-
                      210NM-50MIN.M
    Last changed    : 11/11/2009 11:13:52 AM by dxq
    Analysis Method : D:\LC\DXQ\DATA\DXQ-6-31C\DXQ-6-31C 2009-12-07 21-01-48\SIG1000001.D\DA.M (
                      ASH-90-10-05ML-210NM-50MIN.M)
    Last changed    : 3/6/2010 7:57:58 PM by zzh
                      (modified after loading)
```




```
    ====================================================================
                            Area Percent Report
    ====================================================================

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


    Signal 1: VWD1 A, Wavelength=210 nm

    Peak RetTime Type  Width     Area      Height     Area
      #   [min]        [min]  mAU   *s    [mAU   ]      %
    ----|-------|----|-------|----------|----------|--------|
       1  28.419 BB    0.8867 1.87961e4   328.82306  95.3019
       2  35.572 BB    1.0358  926.59918   13.06263   4.6981

    Totals :                  1.97227e4   341.88569


    ====================================================================
```

Instrument 1 3/6/2010 7:58:02 PM zzh

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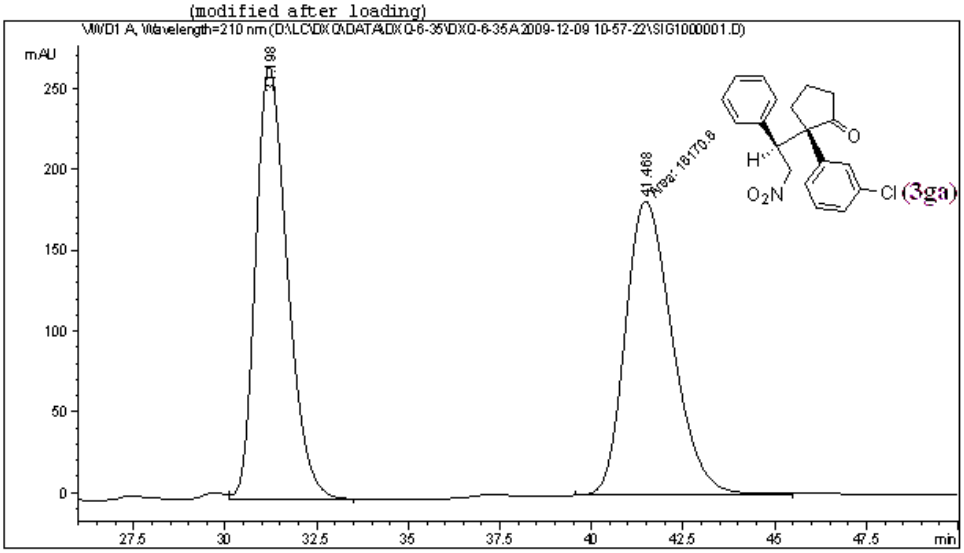
Data File D:\LC\DXQ\DATA\DXQ-6-35\DXQ-6-35A 2009-12-09 10-57-22\SIG1000001.D
Sample Name: dxq-6-35a

```
    ======================================================================
    Acq. Operator   : DXQ                          Seq. Line :   1
    Acq. Instrument : Instrument 1                  Location : Vial 82
    Injection Date  : 12/9/2009 10:58:50 AM              Inj :   1
                                                   Inj Volume : 5 µl
    Acq. Method     : D:\LC\DXQ\data\DXQ-6-35\DXQ-6-35A 2009-12-09 10-57-22\ASH-90-10-05ML-
                      210NM.M
    Last changed    : 11/15/2009 2:15:18 PM by DXQ
    Analysis Method : D:\LC\DXQ\DATA\DXQ-6-35\DXQ-6-35A 2009-12-09 10-57-22\SIG1000001.D\DA.M (
                      ASH-90-10-05ML-210NM.M)
    Last changed    : 3/6/2010 7:47:47 PM by zzh
                      (modified after loading)
```




```
    ======================================================================
                          Area Percent Report
    ======================================================================

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


    Signal 1: VWD1 A, Wavelength=210 nm

    Peak RetTime Type  Width     Area      Height     Area
      #   [min]        [min]  mAU   *s   [mAU   ]      %
    ----|-------|----|-------|----------|----------|--------|
       1  31.198 VB    0.9382 1.61367e4   267.48599  49.9475
       2  41.468 MM    1.4827 1.61706e4   181.77330  50.0525

    Totals :                  3.23073e4   449.25929

    ======================================================================
```

Instrument 1 3/6/2010 7:47:54 PM zzh

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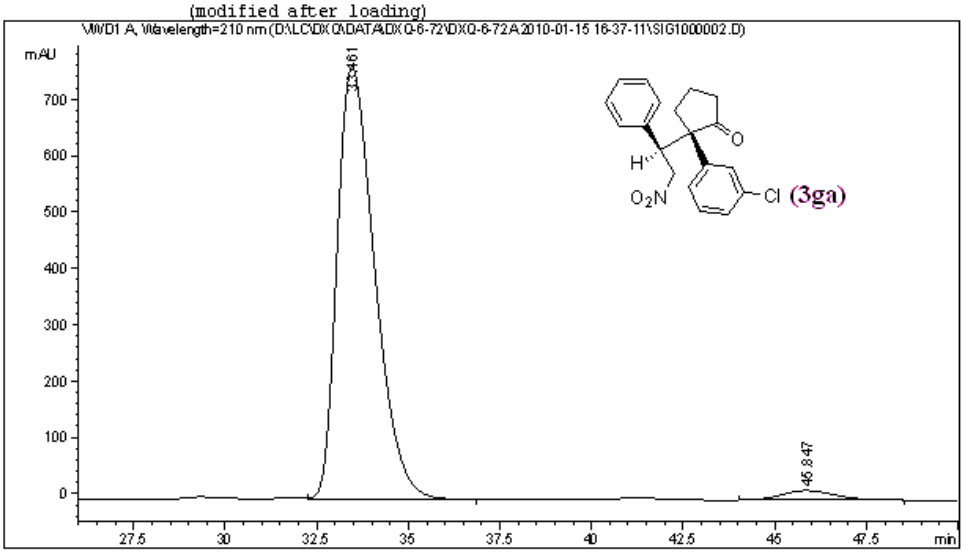
Data File D:\LC\DXQ\DATA\DXQ-6-72\DXQ-6-72A 2010-01-15 16-37-11\SIG1000002.D
Sample Name: DXQ-6-72A

```
    ======================================================================
    Acq. Operator   : DXQ                          Seq. Line :   2
    Acq. Instrument : Instrument 1                  Location : Vial 72
    Injection Date  : 1/15/2010 4:49:27 PM               Inj :   1
                                                   Inj Volume : 5 µl
    Acq. Method     : D:\LC\DXQ\data\DXQ-6-72\DXQ-6-72A 2010-01-15 16-37-11\ASH-90-10-05ML-
                      210NM-50MIN.M
    Last changed    : 11/11/2009 11:13:52 AM by dxq
    Analysis Method : D:\LC\DXQ\DATA\DXQ-6-72\DXQ-6-72A 2010-01-15 16-37-11\SIG1000002.D\DA.M (
                      ASH-90-10-05ML-210NM-50MIN.M)
    Last changed    : 3/6/2010 7:51:02 PM by zzh
                      (modified after loading)
```




```
    ======================================================================
                            Area Percent Report
    ======================================================================

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


    Signal 1: VWD1 A, Wavelength=210 nm

    Peak RetTime Type  Width     Area      Height     Area
      #   [min]        [min]  mAU   *s   [mAU   ]      %
    ----|-------|----|-------|----------|----------|--------|
       1  33.461 VB    1.1071 5.48297e4   767.60748  96.9490
       2  45.847 BB    1.4725 1725.47266   16.97881   3.0510

    Totals :                  5.65552e4   784.58629

    ======================================================================
```

Instrument 1 3/6/2010 7:51:06 PM zzh

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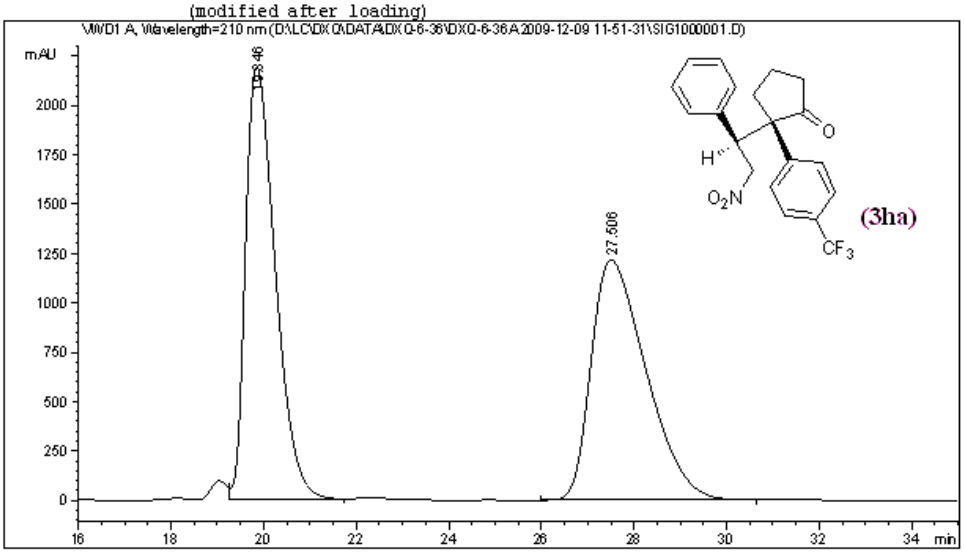
Data File D:\LC\DXQ\DATA\DXQ-6-36\DXQ-6-36A 2009-12-09 11-51-31\SIG1000001.D
Sample Name: dxq-6-36a

Acq. Operator	: DXQ	Seq. Line	: 1
Acq. Instrument	: Instrument 1	Location	: Vial 83
Injection Date	: 12/9/2009 11:53:28 AM	Inj	: 1
		Inj Volume	: 5 µl
Acq. Method	: D:\LC\DXQ\data\DXQ-6-36\DXQ-6-36A 2009-12-09 11-51-31\ASH-90-10-05ML-210NM.M		
Last changed	: 11/15/2009 2:15:18 PM by DXQ		
Analysis Method	: D:\LC\DXQ\DATA\DXQ-6-36\DXQ-6-36A 2009-12-09 11-51-31\SIG1000001.D\DA.M (ASH-90-10-05ML-210NM.M)		
Last changed	: 3/6/2010 7:49:11 PM by zzh (modified after loading)		



Area Percent Report

Sorted By	:	Signal
Multiplier	:	1.0000
Dilution	:	1.0000

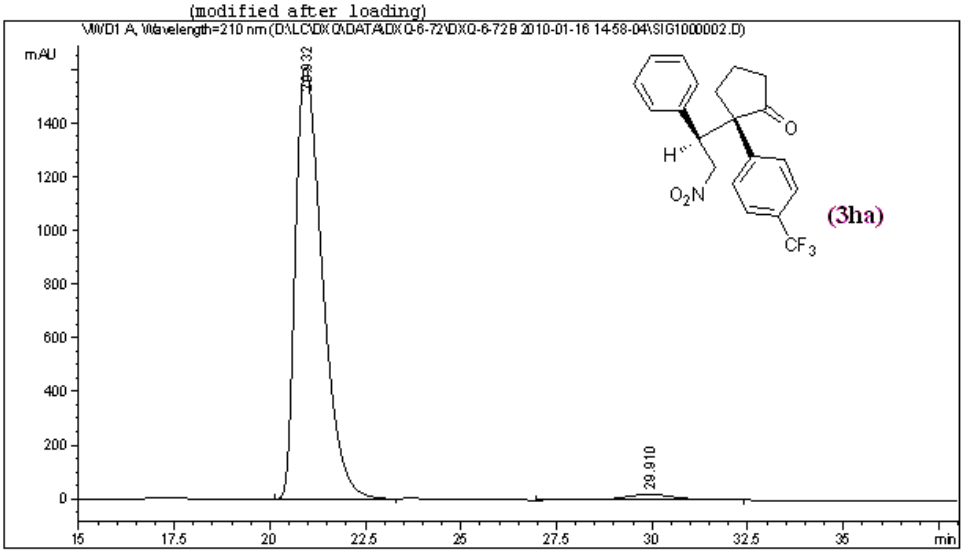
Use Multiplier & Dilution Factor with ISTDs

Signal 1: VWD1 A, Wavelength=210 nm

Peak #	RetTime [min]	Type	Width [min]	Area mAU *s	Height [mAU]	Area %
1	19.846	VV	0.6983	9.84764e4	2194.20679	49.5658
2	27.506	BB	1.2701	1.00202e5	1215.94470	50.4342
Totals :				1.98678e5	3410.15149	

Data File D:\LC\DXQ\DATA\DXQ-6-72\DXQ-6-72B 2010-01-16 14-58-04\SIG1000002.D
Sample Name: DXQ-6-72B

```
    ======================================================================
    Acq. Operator   : LTL                          Seq. Line :   2
    Acq. Instrument : Instrument 1                  Location : Vial 81
    Injection Date  : 1/16/2010 3:10:20 PM               Inj :   1
                                                  Inj Volume : 5 µl
    Acq. Method     : D:\LC\DXQ\data\DXQ-6-72\DXQ-6-72B 2010-01-16 14-58-04\ASH-90-10-05ML-
                      210NM-40MIN.M
    Last changed    : 11/29/2009 11:50:33 AM by dxq
    Analysis Method : D:\LC\DXQ\DATA\DXQ-6-72\DXQ-6-72B 2010-01-16 14-58-04\SIG1000002.D\DA.M (
                      ASH-90-10-05ML-210NM-40MIN.M)
    Last changed    : 3/6/2010 7:52:50 PM by zzh
                      (modified after loading)
```




```
    ======================================================================
                            Area Percent Report
    ======================================================================

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


    Signal 1: VWD1 A, Wavelength=210 nm

    Peak RetTime Type  Width     Area      Height     Area
      #   [min]        [min]  mAU   *s    [mAU   ]      %
    ----|-------|----|-------|----------|----------|--------|
       1  20.932 VV    0.7599 7.90319e4  1611.80432  97.5501
       2  29.910 VB    1.4052 1984.81213   20.86796   2.4499

    Totals :                  8.10167e4  1632.67228

    ======================================================================
```

Instrument 1 3/6/2010 7:52:57 PM zzh

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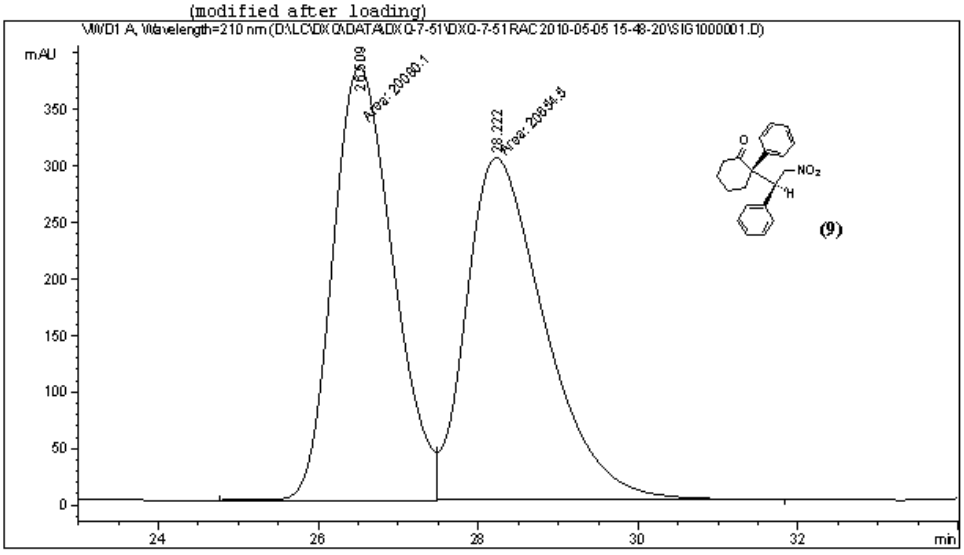
Data File D:\LC\DXQ\DATA\DXQ-7-51\DXQ-7-51RAC 2010-05-05 15-48-20\SIG1000001.D
Sample Name: DXQ-7-51RAC

```
==================================================================
Acq. Operator   : DXQ                          Seq. Line :   1
Acq. Instrument : Instrument 1                  Location : Vial 2
Injection Date  : 5/5/2010 3:49:52 PM                Inj :   1
                                               Inj Volume : 5 µl
Acq. Method     : D:\LC\DXQ\data\DXQ-7-51\DXQ-7-51RAC 2010-05-05 15-48-20\ASH-90-10-05ML-
                  210NM.M
Last changed    : 5/5/2010 3:03:39 PM by THL
Analysis Method : D:\LC\DXQ\DATA\DXQ-7-51\DXQ-7-51RAC 2010-05-05 15-48-20\SIG1000001.D\DA.M
                  (ASH-90-10-05ML-210NM.M)
Last changed    : 5/14/2010 7:38:11 PM by LTL
                  (modified after loading)
```




```
==================================================================
                        Area Percent Report
==================================================================

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


Signal 1: VWD1 A, Wavelength=210 nm

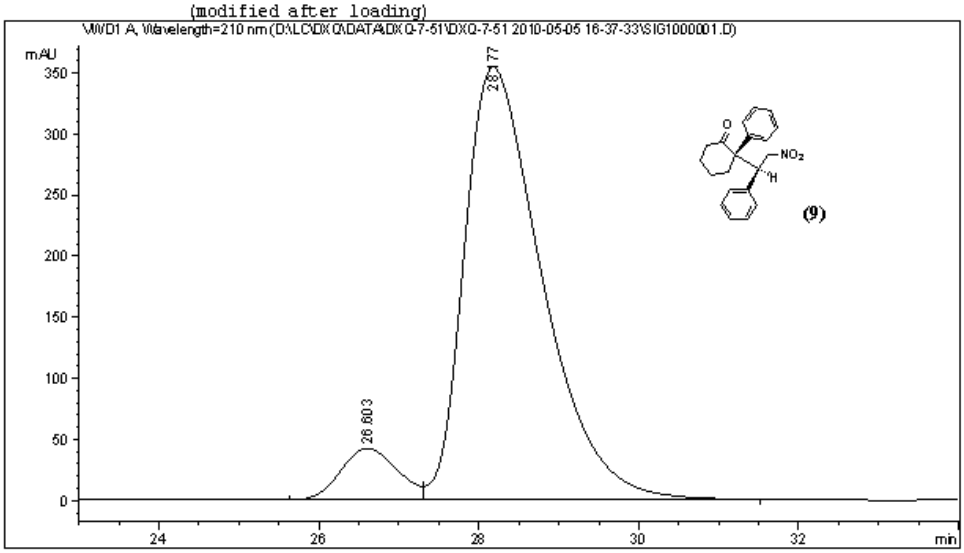
Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]  mAU   *s   [mAU   ]      %
----|-------|----|-------|----------|----------|--------|
   1  26.509 MF    0.8741 2.00801e4   382.89331  49.2949
   2  28.222 FM    1.1378 2.06545e4   302.54550  50.7051

Totals :                  4.07346e4   685.43881

==================================================================
```

Data File D:\LC\DXQ\DATA\DXQ-7-51\DXQ-7-51 2010-05-05 16-37-33\SIG1000001.D
Sample Name: DXQ-7-51

Acq. Operator	: DXQ	Seq. Line	: 1
Acq. Instrument	: Instrument 1	Location	: Vial 3
Injection Date	: 5/5/2010 4:38:50 PM	Inj	: 1
		Inj Volume	: 5 µl
Acq. Method	: D:\LC\DXQ\data\DXQ-7-51\DXQ-7-51 2010-05-05 16-37-33\ASH-90-10-05ML-210NM.M		
Last changed	: 5/5/2010 3:03:39 PM by THL		
Analysis Method	: D:\LC\DXQ\DATA\DXQ-7-51\DXQ-7-51 2010-05-05 16-37-33\SIG1000001.D\DA.M (ASH-90-10-05ML-210NM.M)		
Last changed	: 5/14/2010 7:39:40 PM by LTL (modified after loading)		



Area Percent Report

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

Signal 1: VWD1 A, Wavelength=210 nm

Peak #	RetTime [min]	Type	Width [min]	Area mAU *s	Height [mAU]	Area %
1	26.603	BV	0.7662	2069.03418	42.15705	7.9625
2	28.177	VB	1.0348	2.39158e4	354.40268	92.0375
Totals :				2.59849e4	396.55973	

Data File D:\LC\DXQ\DATA\DXQ-6-4850\DXQ-6-4850 2009-12-21 21-11-17\SIG1000001.D
Sample Name: dxq-6-48

```
    ====================================================================
    Acq. Operator   : dxq                          Seq. Line :   1
    Acq. Instrument : Instrument 1                  Location : Vial 11
    Injection Date  : 12/21/2009 9:12:33 PM              Inj :   1
                                                  Inj Volume : 5 µl
    Acq. Method     : D:\LC\DXQ\data\DXQ-6-4850\DXQ-6-4850 2009-12-21 21-11-17\ADH-10-90-025ML-
                      210NM-35MIN.M
    Last changed    : 12/21/2009 9:07:55 PM by dxq
    Analysis Method : D:\LC\DXQ\DATA\DXQ-6-4850\DXQ-6-4850 2009-12-21 21-11-17\SIG1000001.D\DA.M
                      (ADH-10-90-025ML-210NM-35MIN.M)
    Last changed    : 3/9/2010 10:06:55 AM by dxq
                      (modified after loading)
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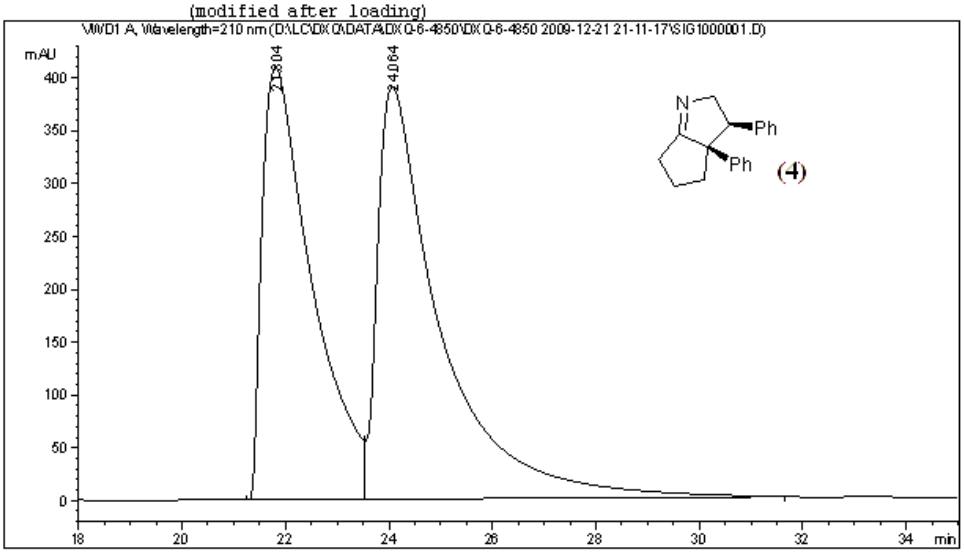


```
    ====================================================================
                          Area Percent Report
    ====================================================================

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


    Signal 1: VWD1 A, Wavelength=210 nm

    Peak RetTime Type  Width     Area      Height     Area
      #   [min]        [min]  mAU   *s   [mAU   ]      %
    ----|-------|----|-------|----------|----------|--------|
       1  21.804 VV    0.9829 2.73415e4   409.45319  45.3491
       2  24.064 VB    1.1937 3.29496e4   390.05441  54.6509

    Totals :                  6.02911e4   799.50760


    ====================================================================
```

Instrument 1 3/9/2010 10:06:59 AM dxq

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Data File D:\LC\DXQ\DATA\DXQ-6-4850\DXQ-6-4850 2009-12-21 21-11-17\SIG1000002.D
Sample Name: dxq-6-50

```
    ====================================================================
    Acq. Operator   : dxq                          Seq. Line :   2
    Acq. Instrument : Instrument 1                  Location : Vial 12
    Injection Date  : 12/21/2009 9:49:16 PM              Inj :   1
                                                   Inj Volume : 5 µl
    Acq. Method     : D:\LC\DXQ\data\DXQ-6-4850\DXQ-6-4850 2009-12-21 21-11-17\ADH-10-90-025ML-
                      210NM-35MIN.M
    Last changed    : 12/21/2009 9:07:55 PM by dxq
    Analysis Method : D:\LC\DXQ\DATA\DXQ-6-4850\DXQ-6-4850 2009-12-21 21-11-17\SIG1000002.D\DA.M
                      (ADH-10-90-025ML-210NM-35MIN.M)
    Last changed    : 3/9/2010 10:09:08 AM by dxq
                      (modified after loading)
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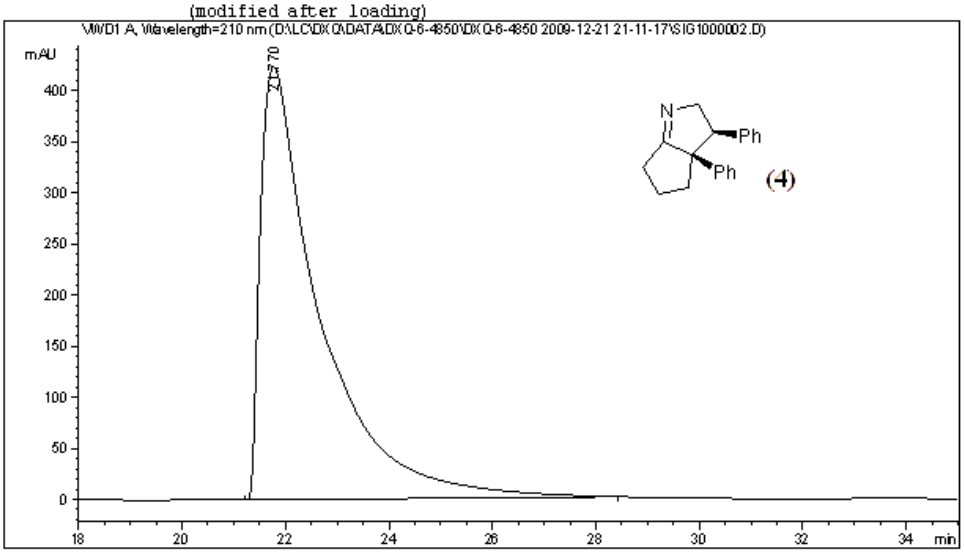



```
    ====================================================================
                            Area Percent Report
    ====================================================================

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


    Signal 1: VWD1 A, Wavelength=210 nm

    Peak RetTime Type  Width     Area      Height     Area
      #   [min]        [min]  mAU   *s   [mAU   ]      %
    ----|-------|----|-------|----------|----------|--------|
       1  21.770 VB    1.1262 3.37857e4   423.32864 100.0000

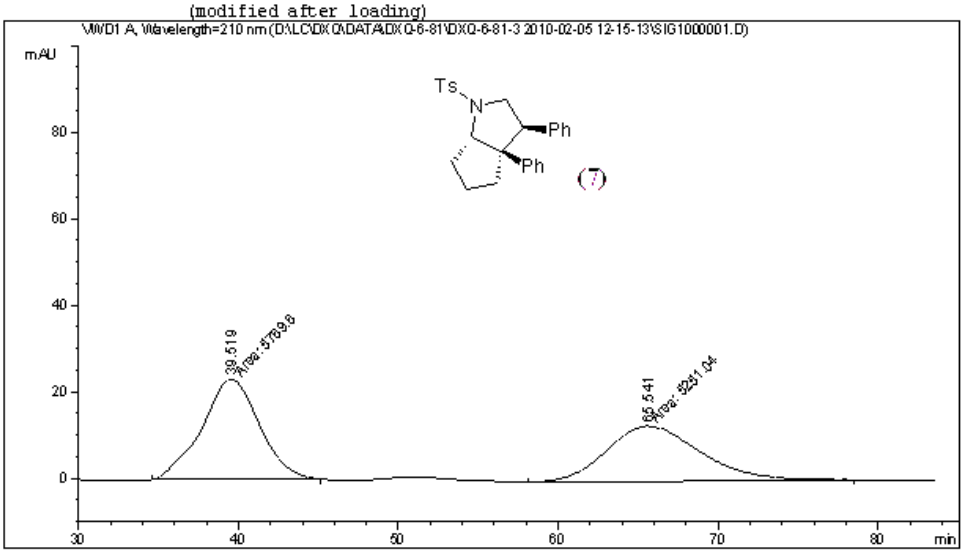
    Totals :                  3.37857e4   423.32864


    ====================================================================
                            *** End of Report ***

Instrument 1 3/9/2010 10:09:12 AM dxq                          Page 1 of 1
```

Data File D:\LC\DXQ\DATA\DXQ-6-81\DXQ-6-81-3 2010-02-05 12-15-13\SIG1000001.D
Sample Name: dxq-6-81

```
    ====================================================================
    Acq. Operator   : dxq                          Seq. Line :   1
    Acq. Instrument : Instrument 1                  Location : Vial 11
    Injection Date  : 2/5/2010 12:16:28 PM               Inj :   1
                                                   Inj Volume : 5 µl
    Acq. Method     : D:\LC\DXQ\data\DXQ-6-81\DXQ-6-81-3 2010-02-05 12-15-13\ASH-90-10-1ML-
                      210NM.M
    Last changed    : 11/29/2009 4:38:24 PM by dxq
    Analysis Method : D:\LC\DXQ\DATA\DXQ-6-81\DXQ-6-81-3 2010-02-05 12-15-13\SIG1000001.D\DA.M (
                      ASH-90-10-1ML-210NM.M)
    Last changed    : 3/10/2010 4:11:17 PM by LTL
                      (modified after loading)
```




```
    ====================================================================
                            Area Percent Report
    ====================================================================

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


    Signal 1: VWD1 A, Wavelength=210 nm

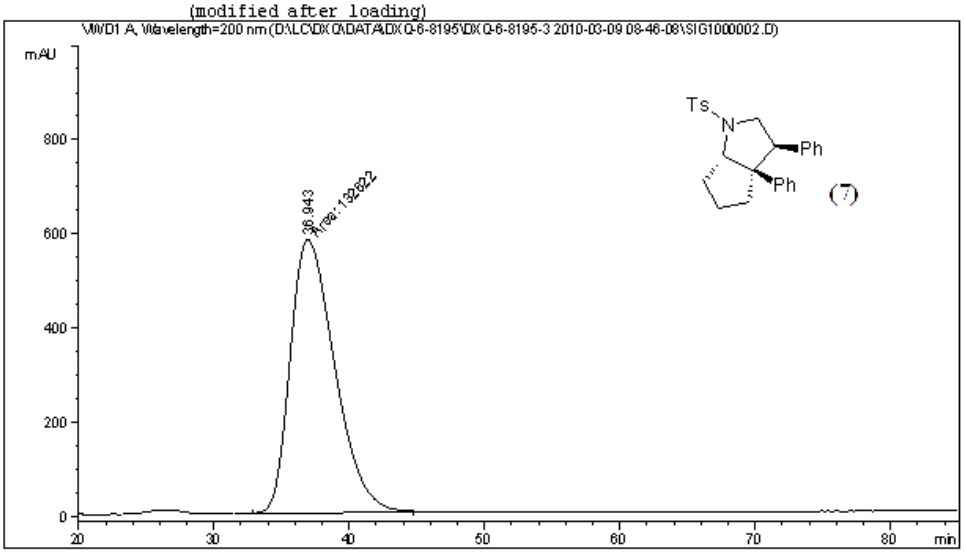
    Peak RetTime Type  Width     Area      Height     Area
      #   [min]        [min]  mAU   *s   [mAU   ]      %
    ----|-------|----|-------|----------|----------|--------|
       1  39.519 MM    4.1685 5769.60156   23.06831  52.3527
       2  65.541 MM    6.8342 5251.03955   12.80587  47.6473

    Totals :                  1.10206e4    35.87418


    ====================================================================
```

Data File D:\LC\DXQ\DATA\DXQ-6-8195\DXQ-6-8195-3 2010-03-09 08-46-08\SIG1000002.D
Sample Name: d-6-95

```
    ======================================================================
    Acq. Operator   : dxq                          Seq. Line :   2
    Acq. Instrument : Instrument 1                  Location : Vial 2
    Injection Date  : 3/9/2010 10:13:52 AM               Inj :   1
                                                   Inj Volume : 5 µl
    Acq. Method     : D:\LC\DXQ\data\DXQ-6-8195\DXQ-6-8195-3 2010-03-09 08-46-08\ASH-90-10-1ML-
                      200NM-85MIN.M
    Last changed    : 3/8/2010 4:10:38 PM by zzh
    Analysis Method : D:\LC\DXQ\DATA\DXQ-6-8195\DXQ-6-8195-3 2010-03-09 08-46-08\SIG1000002.D\
                      DA.M (ASH-90-10-1ML-200NM-85MIN.M)
    Last changed    : 3/10/2010 4:19:12 PM by LTL
                      (modified after loading)
```




```
    ======================================================================
                            Area Percent Report
    ======================================================================

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


    Signal 1: VWD1 A, Wavelength=200 nm

    Peak RetTime Type  Width     Area      Height     Area
      #   [min]        [min]  mAU   *s   [mAU   ]      %
    ----|-------|----|-------|----------|----------|--------|
       1  36.943 MM    3.8068 1.32622e5   580.62769 100.0000

    Totals :                  1.32622e5   580.62769


    ======================================================================
                            *** End of Report ***

Instrument 1 3/10/2010 4:19:16 PM LTL                          Page 1 of 1
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