Highly-Substituted Enantioenriched Cyclopentane Derivatives by Palladium-Catalyzed [3+2] Trimethylenemethane Cycloadditions with Disubstituted Nitroalkenes

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Supporting Information

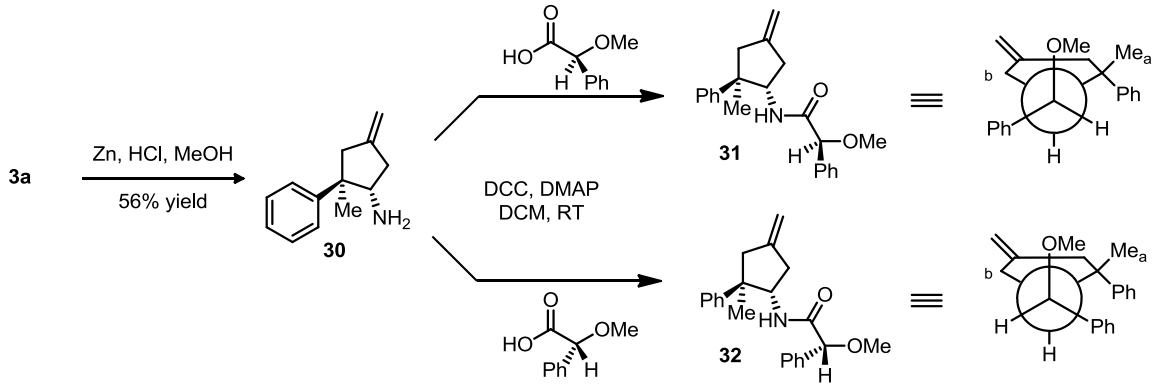
A. General Methods

All TMM reactions were carried out under an argon atmosphere. Solvents were dried by passing through an Alumina column. All compounds were purchased from commercial sources and used directly unless listed. *m*-chloroperoxybenzoic acid was purified by the known method.[i] Diaza(1,3)bicyclo[5.4.0]undene (DBU) and 3-buten-2-one were purified by distillation prior to use. Solutions of potassium *tert*-butoxide were prepared by combining equimolar amounts of distilled *tert*-butanol and potassium hydride (from a 30-35% mineral oil dispersion thrice rinsed with hexanes and dried under vacuum) in THF; after stirring 30 minutes the suspension was allowed to settle and the supernatant was used directly. The following compounds were prepared according to known literature procedures: $Pd(dba)_2$,[ii] 3-acetoxy-2-trimethylsilylmethyl-1-propene **1a**,[iii] 1-cyano-2-((trimethylsilyl)methyl)-allyl acetate **1b**,[iv] **L1**,[v] and **L2**-**L3**.[vi]

Flash chromatography was performed with 0.040-0.063 μm Silica Gel. ^1H and ^{13}C NMR spectroscopy was performed on a Mercury NMR at 400 (^1H) or 100 (^{13}C) MHz and Unity NMR at 500 (^1H) or 125 (^{13}C) MHz. Chemical shifts are reported in ppm relative to tetramethylsilane or residual protiated solvent. All ^{13}C NMR spectra were proton decoupled. Infrared spectroscopic data was recorded on sodium chloride plates as thin films on a Perkin-Elmer Paragon 500 FT-IR spectrometer. Chiral HPLC analysis was performed on a Thermo Separation Products Spectra Series P-100 and on an Agilent Technologies 1200 Series using Chiralcel® columns. Optical rotations were measured on a Jasco DIP-1000 digital polarimeter using 5 cm glass cells with a Na 589 nm filter.

B. Absolute Stereochemistry

The absolute sense of chirality for *trans*-**3a** was determined preparing mandelamides **31** and **32** from cyclopentylamine **30**.[vii] The downfield shift of Me_a (δ 1.38) in **31** compared to Me_a (δ 1.29) in **32**, and the reverse for H_b (δ 2.83, 2.20 for **31** vs δ 2.90, 2.30 for **32**) is consistent with the absolute stereochemistry as depicted.



Scheme S1. Preparation of diastereomeric mandelamides **31** and **32**.

The absolute sense of chirality for **21** was determined by x-ray crystal analysis.

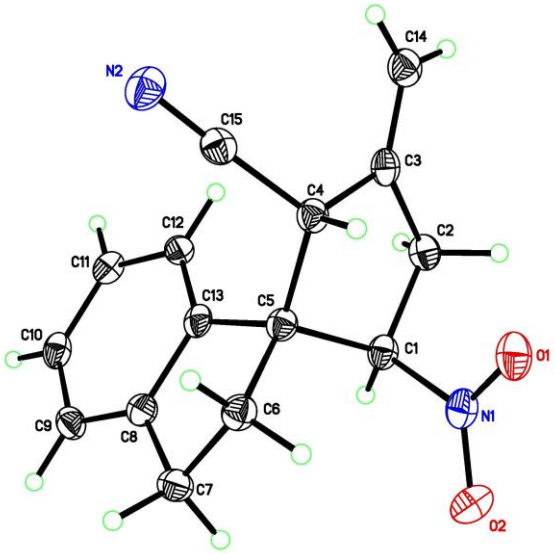


Figure S1. X-ray crystal structure of cyano donor adduct **21**.

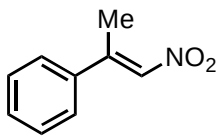
C. General Procedures

General procedure A for the synthesis of nitrolkenes: nitroalkenes were prepared according to the procedure of Stephens:[viii] a mixture of 1,1-disubstituted olefin (10 mmol), sodium nitrite (100 mmol), ceric ammonium nitrate (10 mmol) and acetic acid (120 mmol) in chloroform (100 mL) was sonicated for up to 6 hours in a sealed flask connected to a bubbler. Upon completion, the mixture was filtered and the cake was washed with diethyl ether (100 mL). The solution was quenched with sat. $NaHCO_3$ (75 mL), the aqueous layer was back-extracted with diethyl ether (75 mL), and the combined organic layers were washed with water (50 mL), brine (50 mL), dried over $MgSO_4$, concentrated and purified by chromatography.

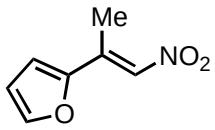
General procedure B for cycloadditions with donor 1a: To an argon-purged vial of substrate (0.076 mmol), ligand **L3** (0.0075 mmol) and $Pd(dba)_2$ (2.2 mg, 0.0038 mmol) was added either dioxane (0.5 mL) or toluene (0.15 mL) and the solution stirred for 2 minutes before 2-((trimethylsilyl)methyl)allyl acetate (25 µL, 0.12 mmol) was added. After stirring for 4 hours at 50 ºC, the solution was concentrated and purified by flash chromatography.

General procedure C for cycloadditions with cyano donor 1b: To an argon-purged vial of substrate (0.076 mmol), ligand **L3** (0.0075 mmol) and $Pd(dba)_2$ (2.2 mg, 0.0038 mmol) was added toluene (0.5 mL) and the solution moved to a cold room at 4ºC. After 3 minutes, 1-cyano-2-((trimethylsilyl)methyl)allyl acetate (30 µL, 0.12 mmol) was added and the reaction was allowed to stir for 1 hour at 4ºC, after which it was immediately purified by flash chromatography.

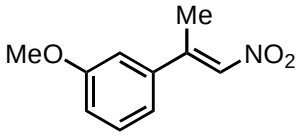
D. Nitroalkenes



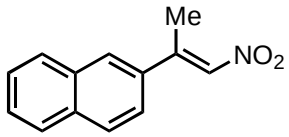
(*E*)-(1-Nitroprop-1-en-2-yl)benzene (2): The reaction was performed according to general procedure A with 5 mL (40 mmol) of α-methylstyrene and purified by chromatography (2% diethyl ether in pet ether) to give the product as a pale yellow oil (2.75 g, 44% yield). Spectral properties matched known characterization.[viii] ^{1}H NMR (500 MHz, $CDCl_3$): δ 7.48-7.43 (m, 5H), 7.33-7.31 (m, 1H), 2.65 (d, *J* = 1.6 Hz, 3H).



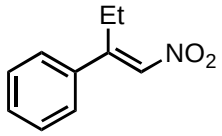
(*E*)-2-(1-Nitroprop-1-en-2-yl)furan: Adapted from the procedure of List and coworkers.[ix] A solution of 2-acetylfuran (6.7 g, 60 mmol), nitromethane (13 mL, 240 mmol) and *n*-butylamine (2.4 mL, 24 mmol) in toluene (43 mL) was heated at reflux using a Dean-Stark apparatus. After 17 hours, the solution was cooled, diluted with ethyl acetate (100 mL) and quenched with 1 M $NaHSO_4$ (50 mL). The organic layer was dried over $MgSO_4$, concentrated and submitted to chromatography (1% diethyl ether in pet ether), then crystallized from diethyl ether/heptane (16 mL, 6:10) at -10 ºC to provide the pure product as pale yellow crystals (1.4 g, 15% yield). Spectral properties matched known characterization.[ix] ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.66 (br d, *J* = 1.2 Hz, 1H), 7.52 (d, *J* = 1.4 Hz, 1H), 6.88 (d, *J* = 3.9 Hz, 1H), 6.53 (dm, *J* = 3.9 Hz, 1H), 2.53 (d, *J* = 1.3 Hz, 3H).



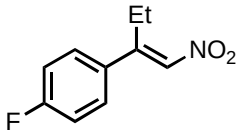
(*E*)-1-Methoxy-3-(1-nitroprop-1-en-2-yl)benzene: The reaction was performed according to general procedure A with 2.58 g (20 mmol) of 3-methoxy-α-methylstyrene and purified by chromatography (5% diethyl ether in pet ether) to give the product as a pale yellow solid (1.71 g, 47% yield). Spectral properties matched known characterization.[viii] ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.37-7.30 (m, 2H), 7.05-6.94 (m, 3H), 3.85 (s, 3H), 2.63 (s, 3H).



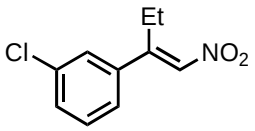
(*E*)-2-(1-Nitroprop-1-en-2-yl)naphthalene: The reaction was performed according to general procedure A with 3.2 g (19.3 mmol) of 2-(2-naphthyl)-1-propene and purified by flash chromatography (1% ether in pet ether) to give the product as a pale yellow solid (1.99 g, 48% yield). Spectral properties matched known characterization.[ix] ^{1}H NMR (500 MHz, $CDCl_3$): δ 7.94-7.84 (m, 4H), 7.57-7.53 (m, 2H), 7.50 (dd, *J* = 2.0, 9.0 Hz, 1H), 7.44 (quartet, J = 1.3 Hz, 1H), 2.73 (d, *J* = 1.3 Hz, 3H).



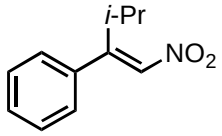
(*E*)-(1-Nitrobut-1-en-2-yl)benzene: The reaction was performed according to general procedure A with 3.2 g (24.4 mmol) of α-ethylstyrene and purified by flash chromatography (2% diethyl ether in pet ether) to give the product as a pale yellow oil (1.74 g, 40% yield). Spectral properties matched known characterization.[ix] ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.47-7.42 (m, 5H), 7.19 (s, 1H), 3.09 (q, *J* = 7.6 Hz, 2H), 1.16 (t, *J* = 7.6 Hz, 3H).



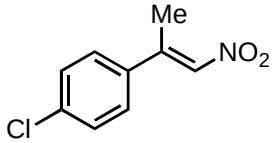
(*E*)-1-Fluoro-4-(1-nitrobut-1-en-2-yl)benzene: The reaction was performed according to general procedure A with 4.4 g (29.0 mmol) of 4-fluoro-α-methylstyrene and purified by flash chromatography (2% diethyl ether in pet ether) to give the product as a pale yellow oil (1.66 g, 29% yield). ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.44-7.38 (m, 2H), 7.15-7.08 (m, 3H), 3.04 (q, *J* = 7.5 Hz, 2H), 1.13 (t, *J* = 7.5 Hz, 3H). ^{13}C NMR (100 MHz, $CDCl_3$): δ 165.3 (d, *J* = 250 Hz), 154.9, 136.0, 133.3 (d, *J* = 3.7 Hz), 129.5 (d, *J* = 8.2 Hz), 116.5 (d, *J* = 21.6 Hz), 25.2, 13.1. IR (thin film): 3104, 2978, 2878, 1602, 1510, 1643, 1340 cm^{-1}.



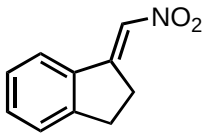
(*E*)-1-Chloro-3-(1-nitrobut-1-en-2-yl)benzene: The reaction was performed according to general procedure A with 4.1 g (24.4 mmol) of 4-chloro-α-methylstyrene and purified by flash chromatography (3% diethyl ether in pet ether) to give the product as a pale yellow oil (1.77 g, 34% yield). ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.43-7.24 (m, 4H), 7.13 (s, 1H), 3.02 (q, *J* = 7.6 Hz, 2H), 1.13 (t, *J* = 7.6 Hz, 3H). ^{13}C NMR (100 MHz, $CDCl_3$): δ 154.3, 139.2, 136.6, 135.4, 130.7, 130.5, 127.6, 125.6, 25.1, 13.0. IR (thin film): 3102, 2977, 2938, 1620, 1564, 1519, 1462, 1342 cm^{-1}.



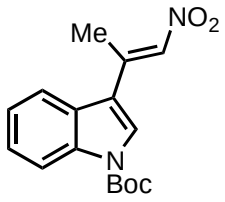
(*E*)-(3-Methyl-1-nitrobut-1-en-2-yl)benzene: The reaction was performed according to general procedure A with 2.1 g (14.6 mmol) of α-isopropylstyrene and purified by flash chromatography (pet ether) to give the product as a pale yellow oil (600 mg, 21% yield). Spectral properties matched known characterization.[x] ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.42-7.37 (m, 3H), 7.24-7.20 (m, 2H), 6.89 (s, 1H), 3.96 (septet, *J* = 6.9 Hz, 1H), 1.13 (d, *J* = 6.9 Hz, 6H).



(*E*)-1-Chloro-4-(1-nitroprop-1-en-2-yl)benzene: The reaction was performed according to general procedure A with 3.0 g (20 mmol) of 4-chloro-α-methylstyrene and purified by chromatography (1% diethyl ether in pet ether) to give the product as a pale yellow oil (2.17 g, 56% yield). Spectral properties matched known characterization.[viii] ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.44-7.38 (m, 4H), 7.29-7.28 (m, 1H), 2.62 (s, 3H).

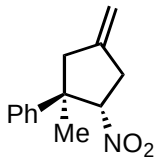


(*E*)-1-(Nitromethylene)-2,3-dihydro-1*H*-indene: The reaction was performed according to general procedure A with 2.2 g (16.9 mmol) of 1-methylene-2,3-dihydro-1*H*-indene and purified by flash chromatography (10% ethyl acetate in hexanes) to give the product as a pale yellow solid (380 mg, 13% yield), mp 80-81 ºC. ^{1}H NMR (500 MHz, $CDCl_3$): δ 7.70 (t, *J* = 2.2 Hz, 1H), 7.59 (d, *J* = 7.9 Hz, 1H), 7.48 (t, *J* = 7.3 Hz, 1H), 7.43 (d, *J* = 7.9 Hz, 1H), 7.32 (t, *J* = 7.3 Hz, 1H), 3.53-3.49 (m, 2H), 3.16 (t, *J* = 5.6 Hz, 2H). ^{13}C NMR (125 MHz, $CDCl_3$): δ 159.2, 151.4, 136.9, 133.2, 130.0, 127.7, 126.3, 122.6, 32.2, 31.0. IR (thin film): 3091, 2919, 1619, 1597, 1499, 1329 cm^{-1}.



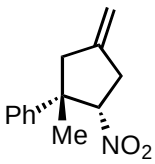
(E)-tert-butyl 3-(1-nitroprop-1-en-2-yl)-1H-indole-1-carboxylate: The reaction was performed according to general procedure A with 0.8 g (3.11 mmol) of *tert*-butyl 3-(prop-1-en-2-yl)-1*H*-indole-1-carboxylate and purified by flash chromatography (5% ethyl acetate in hexanes) to give the product as a bright yellow solid (183 mg, 19% yield), mp 105 ºC. ^{1}H NMR (500 MHz, $CDCl_3$): δ 8.21 (d, *J* = 8.3 Hz, 1 H), 7.93 (s, 1 H), 7.75 (d, *J* = 8.0 Hz, 1 H), 7.66 (m, 1 H), 7.42-7.33 (comp, 2 H), 2.72 (d, *J* = 1.2 Hz, 3 H), 1.71 (s, 9 H). ^{13}C NMR (125 MHz, $CDCl_3$): δ 148.9, 143.6, 136.1, 135.4, 127.8, 126.6, 123.8, 120.2, 118.9, 115.8, 85.2, 28.1, 18.5. IR (thin film): 2878, 1716, 1587, 1317, 1133 cm^{-1}.

E. TMM Cycloadducts

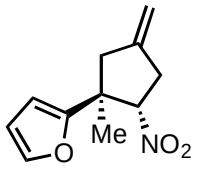


((1*R*,2*S*)-1-Methyl-4-methylene-2-nitrocyclopentyl)benzene (3a): A mixture of

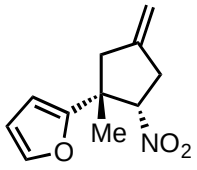
$Pd(dba)_2$ (4.4 mg, 0.0076 mmol) and ligand **L3** (9.6 mg, 0.015 mmol) was purged with argon for 15 minutes. Dioxane (1.0 mL) was added followed by nitroalkene (24.8 mg, 21.5 µL, 0.15 mmol) and the solution was stirred for 2 minutes before 2-((trimethylsilyl)methyl)allyl acetate (50 µL, 0.24 mmol) was added. The solution was immersed in a 50 ºC oil bath and stirred for 4 hours. It was then cooled, concentrated and purified by flash chromatography (4% to 7% diethyl ether in pet ether). The diastereomers thus separated could be independently characterized, but in this case they were recombined to give a clear, colorless oil (24.7 mg, 75% yield, dr 2:1 as determined by ^{1}H NMR, 92% ee and 43% ee, respectively). ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.44-7.23 (m, 5H), 5.14 (quintet, *J* = 2.3 Hz, 1H), 5.10 (dd, *J* = 3.2, 7.0 Hz, 1H), 5.03 (quintet, *J* = 2.3 Hz, 1H), 3.07-3.00 (m, 2H), 2.82-2.76 (m, 1H), 2.72-2.63 (m, 1H), 1.42 (s, 3H). ^{13}C NMR (100 MHz, $CDCl_3$): δ 146.3, 144.5, 129.1, 127.5, 126.4, 108.5, 95.6, 51.1, 44.9, 36.1, 23.9. IR (thin film): 3061, 2974, 1664, 1548, 1497, 1448, 1366, 1309 cm^{-1}. $[\alpha]_{24}^{D}$ = +55.6 (c 0.76, $CHCl_3$). Chiral HPLC: Chiralpak IA, 0.8 mL/min, 0.33% *i*-PrOH in heptane, λ = 220 nm, $t_{R,\ minor}$ = 11.0 min, $t_{R,\ major}$ = 12.1 min. HRMS: calcd for ($M+Na^+$) $C_{13}H_{15}NO_2Na$ 240.1001; found 240.1005. R_f 0.45 (10% diethyl ether in pet ether).



((1*S*,2*S*)-1-Methyl-4-methylene-2-nitrocyclopentyl)benzene (3b): ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.33-7.20 (m, 5H), 5.21-5.18 (m, 1H), 5.13 (dd, *J* = 3.6, 5.9 Hz, 1H), 5.10-5.08 (m, 1H), 3.55 (d, *J* = 15.8 Hz, 1H), 3.05-3.01 (m, 2H), 2.56 (d, *J* = 15.8 Hz, 1H), 1.37 (s, 3H). ^{13}C NMR (100 MHz, $CDCl_3$): δ 146.1, 142.8, 128.9, 127.5, 126.3, 109.7, 95.2, 52.4, 43.0, 37.2, 29.5. IR (thin film): 3062, 2969, 2929, 1665, 1549, 1497, 1447, 1369, 1307 cm^{-1}. $[\alpha]_{24}^{D}$ = +48.9 (c 0.31, $CHCl_3$). Chiral HPLC: Chiralpak IB, 0.8 mL/min, 1% *i*-PrOH in heptane, λ = 220 nm, $t_{R,\ major}$ = 9.9 min, $t_{R,\ minor}$ = 11.5 min. R_f 0.26 (10% diethyl ether in pet ether).

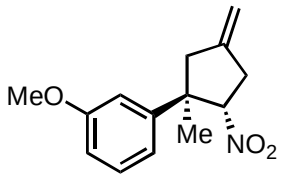


2-((1*S*,2*S*)-1-Methyl-4-methylene-2-nitrocyclopentyl)furan (5a): The reaction was performed with 11.6 mg (0.076 mmol) of nitroalkene according to general procedure B in dioxane at 50 ºC and purified by flash chromatography (5% diethyl ether in pet ether) to give the diastereomeric products as clear, colorless oils (9.3 mg and 2.9 mg, 77% combined yield, 80% ee and 81% ee, respectively). ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.38 (dd, *J* = 0.9, 1.9 Hz, 1H), 6.31 (dd, *J* = 1.9, 3.3 Hz, 1H), 6.16 (dd, *J* = 0.9, 3.3 Hz, 1H), 5.21 (dd, *J* = 5.3, 7.7 Hz, 1H), 5.07 (quintet, *J* = 2.1 Hz, 1H), 5.02 (quintet, *J* = 2.1 Hz, 1H), 3.13 (ddq, *J* = 2.1, 2.1, 2.1, 5.3, 18.1 Hz, 1H), 2.94 (dq, *J* = 2.1, 16.4 Hz, 1H), 2.85 (ddq, *J* = 2.1, 2.1, 2.1, 7.7, 18.1 Hz, 1H), 2.67 (dq, *J* = 2.1, 16.4 Hz, 1H), 1.35 (s, 3H). ^{13}C NMR (100 MHz, $CDCl_3$): δ 156.8, 145.1, 142.4, 110.6, 109.1, 106.4, 92.1, 48.1, 44.5, 35.6, 20.1. IR (thin film): 3122, 3080, 2982, 2940, 1664, 1554, 1505, 1434, 1367, 1316 cm^{-1}. $[\alpha]_{23}^{D}$ = +60.5 (c 0.70, $CHCl_3$). Chiral HPLC: Chiralpak IB, 0.8 mL/min, 1% *i*-PrOH in heptane, λ = 220 nm, $t_{R,\ major}$ = 9.2 min, $t_{R,\ minor}$ = 10.0 min. HRMS: calcd for ($M+H^+$) $C_{11}H_{14}NO_3$ 208.0973; found 208.0963. R_f 0.43 (10% diethyl ether in pet ether).

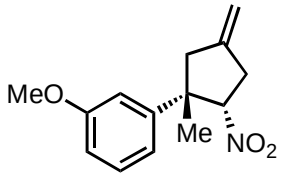


2-((1*R*,2*S*)-1-Methyl-4-methylene-2-nitrocyclopentyl)furan (5b): ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.32 (dd, *J* = 0.8, 1.9, Hz, 1H), 6.27 (dd, *J* = 1.9, 3.3 Hz, 1H), 6.14 (dd, *J* = 0.8, 3.3 Hz, 1H), 5.11 (quintet, *J* = 2.1 Hz, 1H), 5.06 (quintet, *J* = 2.1 Hz, 1H), 4.87 (dd, *J* = 4.1, 7.2 Hz, 1H), 3.28-3.21 (m, 1H), 3.18-3.10 (m, 1H), 2.88 (ddq, *J* = 2.1, 2.1, 2.1, 7.2, 18.2 Hz, 1H), 2.43 (ddq, *J* = 0.5, 1.7, 1.7, 1.7, 16.1 Hz, 1H), 1.48 (d, *J* = 0.5 Hz, 3H). ^{13}C NMR (100 MHz, $CDCl_3$): δ 155.0, 145.0, 142.8, 110.5, 109.6, 106.9, 94.2, 48.8, 43.4, 36.2, 25.1. IR (thin film): 3121, 3079, 2978, 2934, 1664, 1554, 1504, 1434, 1370, 1312 cm^{-1}. $[\alpha]_{25}^{D}$ = +74.8 (c 0.39, $CHCl_3$). Chiral HPLC: Chiralpak IA, 0.8 mL/min, 1% *i*-

PrOH in heptane, λ = 220 nm, $t_{R, major}$ = 9.1 min, $t_{R, minor}$ = 14.4 min. R_f 0.30 (10% diethyl ether in pet ether).

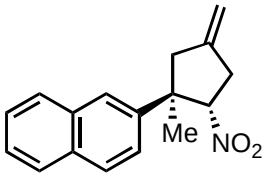


1-Methoxy-3-((1*R*,2*S*)-1-methyl-4-methylene-2-nitrocyclopentyl)benzene (6a): The reaction was performed with 14.7 mg (0.076 mmol) of nitroalkene according to general procedure B in dioxane at 50 ºC, but 3.3 mg $Pd(dba)_2$ and 7.2 mg ligand **L3** were used. The reaction was concentrated and purified by successive flash chromatography (50% dichloromethane in pet ether, then 7% to 10% diethyl ether in pet ether) to give the diastereomeric products as clear, colorless oils (8.0 mg and 3.7 mg, 62% combined yield, 96% ee and 41% ee, respectively). ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.28 (t, *J* = 8.2 Hz, 1H), 7.00 (ddd, *J* = 0.9, 1.8, 7.8 Hz, 1H), 6.96 (t, *J* = 2.4 Hz, 1H), 6.80 (ddd, *J* = 0.8, 2.4, 8.2 Hz, 1H), 5.13 (quintet, *J* = 2.3 Hz, 1H), 5.10 (dd, *J* = 3.4, 7.2 Hz, 1H), 5.02 (quintet, *J* = 2.3 Hz, 1H), 3.81 (s, 3H), 3.07-2.98 (m, 2H), 2.81-2.62 (m, 2H), 1.41 (s, 3H). ^{13}C NMR (100 MHz, $CDCl_3$): δ 160.1, 146.3, 146.3, 130.1, 118.8, 113.1, 112.0, 108.5, 95.5, 55.6, 51.1, 45.0, 36.1, 23.9. IR (thin film): 3078, 2967, 2837, 1663, 1602, 1550, 1491, 1367 cm^{-1}. $[\alpha]_{26}^{D}$ = +61.6 (c 0.76, $CHCl_3$). Chiral HPLC: Chiralpak IB, 0.8 mL/min, 1% *i*-PrOH in heptane, λ = 220 nm, $t_{R, minor}$ = 12.2 min, $t_{R, major}$ = 12.9 min. HRMS: calcd for the amine ($M+H^+$) $C_{14}H_{20}NO$ 218.1545; found 218.1544. R_f 0.33 (10% diethyl ether in pet ether).

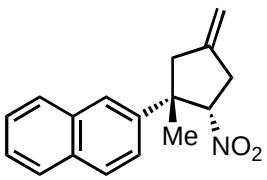


1-Methoxy-3-((1*S*,2*S*)-1-methyl-4-methylene-2-nitrocyclopentyl)benzene (6b): ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.23 (t, *J* = 8.0 Hz, 1H), 6.86 (ddd, *J* = 0.9, 1.7, 7.7 Hz, 1H), 6.81 (t, *J* = 2.1 Hz, 1H), 6.77 (ddd, *J* = 0.9, 2.5, 8.2 Hz, 1H), 5.20-5.17 (m, 1H), 5.11 (dd, *J* = 3.4, 6.1 Hz, 1H), 5.09-5.06 (m, 1H), 3.79 (s, 3H), 3.52 (d, *J* = 15.5 Hz, 1H), 3.05-3.00

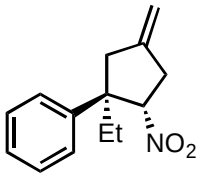
(m, 2H), 2.55 (d, *J* = 15.5 Hz, 1H), 1.35 (d, *J* = 0.8 Hz, 3H). ^{13}C NMR (100 MHz, $CDCl_3$): δ 159.9, 146.1, 144.5, 129.9, 118.6, 112.9, 112.2, 109.7, 95.1, 55.5, 52.4, 43.2, 37.3, 29.6. IR (thin film): 3078, 2967, 2836, 1662, 1604, 1550, 1489, 1432, 1370 cm^{-1}. $[\alpha]_{26}^{D}$ = +71.4 (c 0.31, $CHCl_3$). Chiral HPLC: Chiralpak AD-H, 0.8 mL/min, 5% *i*-PrOH in heptane, λ = 220 nm, $t_{R, minor}$ = 9.2 min, $t_{R, major}$ = 11.8 min. R_f 0.23 (10% diethyl ether in pet ether).



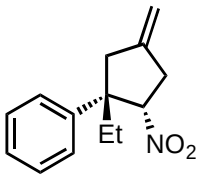
2-((1*R*,2*S*)-1-Methyl-4-methylene-2-nitrocyclopentyl)naphthalene (7a): The reaction was performed with 16.2 mg (0.076 mmol) of nitroalkene according to general procedure B in dioxane at 50 ºC and purified by successive flash chromatography (25% dichloromethane in pet ether, then 5% diethyl ether in pet ether) to give the diastereomeric products as clear, colorless oils (10.0 mg and 4.3 mg, 70% combined yield, 96% ee and 78% ee, respectively). ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.87-7.78 (m, 4H), 7.56-7.46 (m, 3H), 5.22 (dd, *J* = 3.7, 7.4 Hz, 1H), 5.20-5.17 (m, 1H), 5.06-5.03 (m, 1H), 3.17 (d, *J* = 16.9 Hz, 1H), 3.10-3.03 (m, 1H), 2.86 (dd, *J* = 2.5, 16.9 Hz, 1H), 2.69 (ddq, *J* = 2.1, 2.1, 2.1, 7.1, 18.1 Hz, 1H), 1.50 (s, 3H). ^{13}C NMR (100 MHz, $CDCl_3$): δ 146.2, 141.6, 133.5, 132.6, 129.0, 128.5, 127.8, 126.8, 126.6, 125.2, 124.6, 108.6, 95.3, 51.2, 45.0, 36.1, 23.8. IR (thin film): 3058, 2973, 1663, 1599, 1547, 1506, 1456, 1366 cm^{-1}. $[\alpha]_{26}^{D}$ = +93.1 (c 1.0, $CHCl_3$). Chiral HPLC: Chiralpak OJ-H, 0.8 mL/min, 1% *i*-PrOH in heptane, λ = 254 nm, $t_{R, major}$ = 37.2 min, $t_{R, minor}$ = 49.7 min. HRMS: calcd for the amine ($M+H^+$) $C_{17}H_{20}N$ 238.1595; found 238.1602. R_f 0.44 (10% diethyl ether in pet ether).



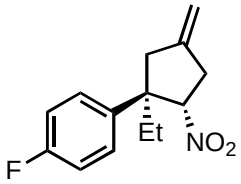
2-((1*S*,2*S*)-1-Methyl-4-methylene-2-nitrocyclopentyl)naphthalene (7b): ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.80-7.76 (m, 3H), 7.67 (d, *J* = 1.9 Hz, 1H), 7.46-7.41 (m, 3H), 5.23-5.20 (m, 2H), 5.11-5.09 (m, 1H), 3.67 (d, *J* = 14.9 Hz, 1H), 3.06-3.01 (m, 2H), 2.65 (d, *J* = 14.9 Hz, 1H), 1.40 (s, 3H). ^{13}C NMR (100 MHz, $CDCl_3$): δ 146.1, 140.2, 133.5, 132.7, 128.5, 128.3, 127.8, 126.5, 126.3, 125.1, 124.6, 109.8, 95.1, 52.6, 43.1, 37.2, 29.4. IR (thin film): 3057, 2969, 2927, 1663, 1600, 1549, 1506, 1370 cm^{-1}. $[\alpha]_{26}^{D}$ = +105.7 (c 0.43, $CHCl_3$). Chiral HPLC: Chiralpak AD-H, 0.8 mL/min, 1% *i*-PrOH in heptane, λ = nm, $t_{R, major}$ = 17.0 min, $t_{R, minor}$ = 19.2 min. R_f 0.28 (10% diethyl ether in pet ether).



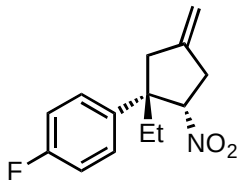
((1*R*,2*S*)-1-Ethyl-4-methylene-2-nitrocyclopentyl)benzene (8a): The reaction was performed with 13.5 mg (12.0 µL, 0.076 mmol) of nitroalkene according to general procedure B in toluene and purified by flash chromatography (4% to 8% diethyl ether in pet ether) to give the diastereomeric products as clear, colorless oils (13.1 mg and 2.1 mg, 87% combined yield, 90% ee and 67% ee, respectively). ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.39-7.24 (m, 5H), 5.18 (bs, 1H), 5.08 (d, *J* = 6.6 Hz, 1H), 5.00 (bs, 1H), 3.15 (d, *J* = 17.0 Hz, 1H), 2.88 (d, *J* = 18.3 Hz, 1H), 2.77 (dd, *J* = 2.8, 17.0 Hz, 1H), 2.55-2.46 (m, 1H), 2.13 (dq, *J* = 7.6, 14.9 Hz, 1H), 1.44 (dq, *J* = 7.6, 14.9 Hz, 1H), 0.60 (t, *J* = 7.6 Hz, 3H). ^{13}C NMR (100 MHz, $CDCl_3$): δ 147.0, 141.8, 129.1, 127.5, 127.2, 108.1, 96.9, 56.0, 40.2, 36.3, 30.5, 9.7. IR (thin film): 2970, 1650, 1548, 1449, 1365 cm^{-1}. $[\alpha]_{23}^{D}$ = +6.1 (c 1.07, $CHCl_3$). Chiral HPLC: Chiralpak IB, 0.8 mL/min, 1% *i*-PrOH in heptane, λ = 220 nm, $t_{R, minor}$ = 6.5 min, $t_{R, major}$ = 7.0 min. HRMS: calcd for ($M+H^+$) $C_{14}H_{18}NO_2$ 232.1337; found 232.1324. R_f 0.51 (10% diethyl ether in pet ether).



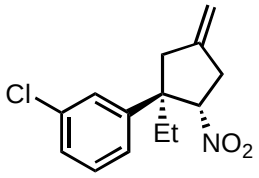
((1*S*,2*S*)-1-Ethyl-4-methylene-2-nitrocyclopentyl)benzene (8b): ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.32-7.19 (m, 5H), 5.19-5.13 (m, 2H), 5.06-5.04 (m, 1H), 3.42-3.35 (m, 1H), 3.02-2.98 (m, 2H), 2.70 (d, *J* = 16.2 Hz, 1H), 1.80-1.70 (m, 1H), 1.61-1.52 (m, 1H), 0.61 (t, *J* = 7.4 Hz, 3H). ^{13}C NMR (100 MHz, $CDCl_3$): δ 146.1, 140.3, 128.6, 127.4, 127.2, 109.5, 95.2, 56.7, 39.3, 37.1, 32.8, 8.8. IR (thin film): 2970, 2930, 1650, 1550, 1498, 1447, 1368 cm^{-1}. $[\alpha]_{25}^{D}$ = +81.3 (c 0.23, $CHCl_3$). Chiral HPLC: Chiralpak AD-H, 0.8 mL/min, 1% *i*-PrOH in heptane, λ = 220 nm, $t_{R,\ major}$ = 9.2 min, $t_{R,\ minor}$ = 10.3 min. R_f 0.36 (10% diethyl ether in pet ether).



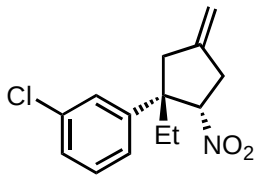
1-((1*R*,2*S*)-1-Ethyl-4-methylene-2-nitrocyclopentyl)-4-fluorobenzene (9a): The reaction was performed with 14.8 mg (12.5 μL, 0.076 mmol) of nitroalkene according to general procedure B in toluene and purified by flash chromatography (5% to 7% diethyl ether in pet ether) to give the diastereomeric products as clear, colorless oils (15.4 mg and 2.3 mg, 93% combined yield, 95% ee and 52% ee, respectively). ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.35 (dd, *J* = 5.2, 9.0 Hz, 2H), 7.05 (dd, *J* = 8.5, 9.0 Hz, 2H), 5.11 (bs, 1H), 5.03-5.00 (m, 2H), 3.10 (d, *J* = 16.5 Hz, 1H), 2.89 (d, *J* = 18.4 Hz, 1H), 2.82-2.75 (m, 1H), 2.55-2.46 (m, 1H), 2.09 (dq, *J* = 7.5, 13.5 Hz, 1H), 1.44 (dq, *J* = 7.5, 13.5 Hz, 1H), 0.60 (t, *J* = 7.5 Hz, 3H). ^{13}C NMR (100 MHz, $CDCl_3$): δ 161.9 (d, *J* = 246 Hz), 146.7, 137.5 (d, *J* = 3.1 Hz), 129.0 (d, *J* = 8.4 Hz), 116.0 (d, *J* = 21.2 Hz), 108.4, 96.8, 55.5, 40.4, 36.2, 30.5, 9.6. IR (thin film): 2971, 2936, 1663, 1603, 1549, 1511, 1367 cm^{-1}. $[\alpha]_{25}^{D}$ = +24.8 (c 1.54, $CHCl_3$). Chiral HPLC: Chiralpak OJ-H, 0.8 mL/min, 0.2% *i*-PrOH in heptane, λ = 220 nm, $t_{R,\ minor}$ = 19.0 min, $t_{R,\ major}$ = 21.7 min. HRMS: calcd for ($M+Na^+$) $C_{14}H_{16}FNO_2Na$ 272.1063; found 272.1058. R_f 0.47 (10% diethyl ether in pet ether).



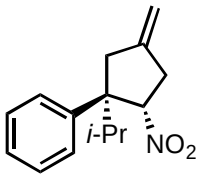
1-((1*S*,2*S*)-1-Ethyl-4-methylene-2-nitrocyclopentyl)-4-fluorobenzene (9b): ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.18 (dd, *J* = 5.2, 8.6 Hz, 2H), 7.00 (t, *J* = 8.6 Hz, 2H), 5.18 (bs, 1H), 5.11 (t, *J* = 4.8 Hz, 1H), 5.06 (bs, 1H), 3.34 (d, *J* = 15.6 Hz, 1H), 3.02-2.99 (m, 2H), 2.69 (d, *J* = 15.6 Hz, 1H), 1.78-1.68 (m, 1H), 1.62-1.52 (m, 1H), 0.61 (t, *J* = 7.4 Hz, 3H). ^{13}C NMR (100 MHz, $CDCl_3$): δ 161.9 (d, *J* = 246 Hz), 145.7, 135.9 (d, *J* = 3.7 Hz), 128.8 (d, *J* = 7.4 Hz), 115.4 (d, *J* = 20.9 Hz), 109.6, 95.1, 56.1, 39.4, 36.9, 32.6, 8.6. IR (thin film): 2971, 2931, 1658, 1606, 1551, 1513, 1369 cm^{-1}. $[\alpha]_{25}^{D}$ = +54.4 (c 0.29, $CHCl_3$). Chiral HPLC: Chiralpak IC, 0.8 mL/min, 1% *i*-PrOH in heptane, λ = 220 nm, $t_{R, major}$ = 12.2 min, $t_{R, minor}$ = 13.4 min. R_f 0.29 (10% diethyl ether in pet ether).



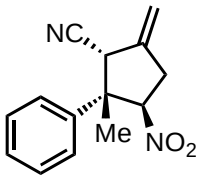
1-Chloro-3-((1*R*,2*S*)-1-ethyl-4-methylene-2-nitrocyclopentyl)benzene (10a): The reaction was performed with 16.1 mg (13 μL, 0.076 mmol) of nitroalkene according to general procedure B in toluene and purified by flash chromatography (4% to 8% diethyl ether in pet ether) to give the diastereomeric products as clear, colorless oils (17.4 mg and 2.5 mg, 99% combined yield, 93% ee and 58% ee, respectively). ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.35-7.24 (m, 4H), 5.20 (bs, 1H), 5.05-5.01 (m, 2H), 3.10 (d, *J* = 17.8 Hz, 1H), 2.91 (d, *J* = 17.8 Hz, 1H), 2.82-2.75 (m, 1H), 2.56-2.47 (m, 1H), 2.09 (dq, *J* = 7.5, 13.6 Hz, 1H), 1.45 (dq, *J* = 7.5, 13.6, 1H), 0.61 (t, *J* = 7.5 Hz, 3H). ^{13}C NMR (100 MHz, $CDCl_3$): δ 146.3, 144.2, 135.2, 130.4, 127.8, 127.6, 125.5, 108.6, 96.5, 55.9, 40.2, 36.2, 30.4, 9.7. IR (thin film): 2970, 1660, 1594, 1549, 1444, 1366 cm^{-1}. $[\alpha]_{25}^{D}$ = +27.6 (c 1.74, $CHCl_3$). Chiral HPLC: Chiralpak OD-H, 0.8 mL/min, 1% *i*-PrOH in heptane, λ = 220 nm, $t_{R, minor}$ = 8.2 min, $t_{R, major}$ = 9.2 min. HRMS: calcd for ($M+H^+$) $C_{14}H_{17}ClNO_2$ 266.0948; found 266.0934. R_f 0.47 (10% diethyl ether in pet ether).



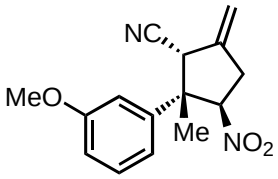
1-Chloro-3-((1*S*,2*S*)-1-ethyl-4-methylene-2-nitrocyclopentyl)benzene (10b): ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.27-7.09 (m, 4H), 5.19 (bs, 1H), 5.12 (dd, *J* = 3.7, 5.9 Hz, 1H), 5.06 (bs, 1H), 3.35 (dd, *J* = 1.8, 15.7 Hz, 1H), 3.03-2.99 (m, 2H), 2.69 (d, *J* = 15.7 Hz, 1H), 1.76-1.66 (m, 1H), 1.57 (dq, *J* = 7.5, 14.5 Hz, 1H), 0.62 (t, *J* = 7.5 Hz, 3H). ^{13}C NMR (100 MHz, $CDCl_3$): δ 145.5, 142.6, 134.6, 129.9, 127.7, 127.6, 125.4, 109.9, 95.0, 56.5, 39.2, 37.1, 32.7, 8.7. IR (thin film): 2971, 2932, 1655, 1597, 1550, 1421, 1368 cm^{-1}. $[\alpha]_{25}^{D}$ = +57.8 (c 0.31, $CHCl_3$). Chiral HPLC: Chiralpak IB, 0.8 mL/min, 1% *i*-PrOH in heptane, λ = 220 nm, $t_{R, major}$ = 8.4 min, $t_{R, minor}$ = 10.2 min. R_f 0.29 (10% diethyl ether in pet ether).



((1*R*,2*S*)-1-Isopropyl-4-methylene-2-nitrocyclopentyl)benzene (11a): The reaction was performed with 14.5 mg (13.5 µL, 0.076 mmol) of nitroalkene according to general procedure B in toluene and purified by flash chromatography (33% dichloromethane in pet ether) to give the product as a clear, colorless oil (16.6 mg, 89% yield, 81% ee). The product was contaminated by approximately 6% of the nitroalkene, which could not be removed by chromatography. ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.40-7.27 (m, 5H), 5.65 (d, *J* = 6.5 Hz, 1H), 5.08-5.05 (m, 1H), 4.87-4.84 (m, 1H), 3.31-3.24 (m, 1H), 2.96-2.89 (m, 1H), 2.84-2.77 (m, 1H), 2.41 (ddq, *J* = 2.7, 2.7, 2.7, 6.5, 18.5 Hz, 1H), 1.80 (septet, *J* = 6.8 Hz, 1H), 0.91 (d, *J* = 6.8 Hz, 3H), 0.69 (d, *J* = 6.8 Hz, 3H). ^{13}C NMR (100 MHz, $CDCl_3$): δ 146.0, 136.8, 128.5, 128.4, 127.6, 108.3, 94.1, 60.1, 41.4, 37.0, 34.5, 19.7, 19.3. IR (thin film): 3060, 2969, 1667, 1599, 1554, 1447, 1367, 1308 cm^{-1}. $[\alpha]_{24}^{D}$ = +58.9 (c 1.05, $CHCl_3$). Chiral HPLC: Chiralpak IC, 0.8 mL/min, 0.2% *i*-PrOH in heptane, λ = 220 nm, $t_{R, major}$ = 19.1. min, $t_{R, minor}$ = 21.8 min. HRMS: calcd for the amine ($M+H^+$) $C_{15}H_{22}N$ 216.1752; found 216.1752. R_f 0.49 (40% dichloromethane in pet ether).

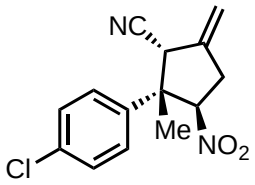


(1*S*,2*R*,3*S*)-2-Methyl-5-methylene-3-nitro-2-phenylcyclopentanecarbonitrile (12): The reaction was performed with 12.7 mg (0.078 mmol) of nitroalkene according to general procedure C and purified by flash chromatography (13% ethyl acetate in hexanes) to give the product as a white solid (18.3 mg, 97% yield, 92% ee), mp 67-69 ºC. ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.50-7.31 (m, 5H), 5.60 (q, *J* = 2.5 Hz, 1H), 5.36 (q, *J* = 2.5 Hz, 1H), 5.05 (dd, *J* = 1.5, 7.2 Hz, 1H), 4.19-4.16 (m, 1H), 3.15-3.07 (dm, *J* = 19.0 Hz, 1H), 2.90 (ddq, *J* = 2.5, 2.5, 2.5, 7.2, 19.0 Hz, 1H), 1.67 (s, 3H). ^{13}C NMR (100 MHz, $CDCl_3$): δ 142.1, 139.7, 129.4, 128.7, 126.7, 117.6, 111.7, 94.4, 53.6, 45.7, 35.2, 22.8. IR (thin film): 2986, 2244, 1665, 1551, 1499, 1446, 1366, 1302 cm^{-1}. $[\alpha]_{23}^{D}$ = -0.6 (c 0.89, $CHCl_3$). Chiral HPLC: Chiralpak AD-H, 0.8 mL/min, 1% *i*-PrOH in heptane, λ = 220 nm, $t_{R,\ minor}$ = 21.2 min, $t_{R,\ major}$ = 27.0 min. HRMS: calcd for the amine ($M+Na^+$) $C_{14}H_{16}N_2Na$ 235.1211; found 235.1217. R_f 0.18 (15% ethyl acetate in hexanes).

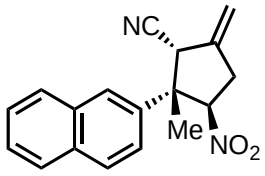


(1*S*,2*R*,3*S*)-2-(3-Methoxyphenyl)-2-methyl-5-methylene-3-nitrocyclopentanecarbonitrile (13): The reaction was performed with 14.7 mg (0.076 mmol) of nitroalkene according to general procedure C and purified by flash chromatography (20% ethyl acetate in hexanes) to give the product as a clear, colorless oil (20.6 mg, 100% yield, 90% ee) that solidified on standing, mp 102-104 ºC. ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.31 (t, *J* = 8.1 Hz, 1H), 7.05 (ddd, *J* = 0.8, 2.2, 7.8 Hz, 1H), 7.03 (t, *J* = 2.2 Hz, 1H), 6.86 (ddd, *J* = 0.8, 2.4, 8.1 Hz, 1H), 5.60 (q, *J* = 2.6 Hz, 1H), 5.36-5.34 (m, 1H), 5.05 (dd, *J* = 1.7, 7.2 Hz, 1H), 4.18-4.15 (m, 1H), 3.81 (s, 3H), 3.14-3.07 (m, 1H), 2.92 (ddq, *J* = 2.6, 2.6, 2.6, 7.2, 19.0 Hz, 1H), 1.65 (s, 3H). ^{13}C NMR (100 MHz, $CDCl_3$): δ 160.2, 142.2, 141.2, 130.4, 118.9, 117.6, 113.4, 113.4, 111.7, 94.4,

55.6, 53.6, 45.8, 35.2, 22.8. IR (thin film): 2939, 2244, 1665, 1603, 1552, 1494, 1463, 1432, 1366, 1295, 1243 cm^{-1}. $[\alpha]_{24}^{D}$ = +5.5 (c 0.93, $CHCl_3$). Chiral HPLC: Chiralpak AD-H, 0.8 mL/min, 1% *i*-PrOH in heptane, λ = 220 nm, $t_{R, minor}$ = 30.7 min, $t_{R, major}$ = 35.6 min. HRMS: calcd for ($M+H^+$) $C_{15}H_{17}N_2O_3$ 273.1239; found 273.1234. R_f 0.40 (30% ethyl acetate in hexanes).

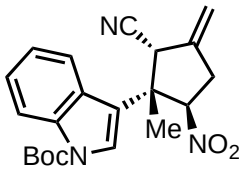


(1*R*, 2*S*, 3*R*)-2-(4-Chlorophenyl)-2-methyl-5-methylene-3-nitrocyclopentanecarbonitrile (14): The reaction was performed with 15.0 mg (0.076 mmol) of nitroalkene according to general procedure C and purified by flash chromatography (10% ethyl acetate in hexanes) to give the product as a light yellow oil (18.0 mg, 87% yield, >20:1 dr): ^{1}H NMR (500 MHz, $CDCl_3$): δ 7.43-7.40 (m, 2 H), 7.38-7.35 (m, 2 H), 5.60 (q, *J* = 2.4 Hz, 1 H), 5.37 (dtd, *J* = 3.1, 2.1, 0.9 Hz, 1 H), 5.00 (dd, *J* = 7.2, 1.7 Hz, 1 H), 4.16 (s, 1 H), 3.14 (ddt, *J* = 19.0, 3.5, 1.9 Hz, 1 H), 2.89 (ddq, *J* = 19.0, 7.2, 2.4 Hz, 1 H), 1.65 (s, 3 H); ^{13}C NMR (125 MHz, $CDCl_3$): δ 141.3, 137.8, 134.5, 129.2, 127.8, 117.0, 111.9, 93.7, 52.9, 45.5, 34.8, 22.4; IR (neat): 2918, 2245, 1553 cm^{-1}. $[\alpha]_{23}^{D}$ = -7.90 (c 1.0, $CHCl_3$). Chiral HPLC: 99% ee, Chiralpak AD-H, 0.8 mL/min, 1% *i*-PrOH in heptane, λ = 220 nm, $t_{R, minor}$ = 30.1 min, $t_{R, major}$ = 39.4 min. HRMS: calcd for the amine (M + H) $C_{14}H_{16}ClN_2$ 247.0924; found 247.0997. R_f 0.41 (20% ethyl acetate in hexanes).

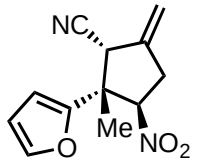


(1*R*, 2*S*, 3*R*)-2-Methyl-5-methylene-2-(naphthalen-2-yl)-3-nitrocyclopentanecarbonitrile (15): The reaction was performed with 16.0 mg (0.076 mmol) of nitroalkene according to general procedure C and purified by flash chromatography (10% ethyl acetate in hexanes) to give the product as a white solid (20.5

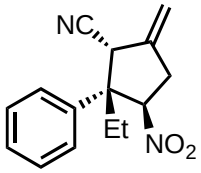
mg, 92% yield, >20:1 dr): mp = 158 °C; ^{1}H NMR (500 MHz, $CDCl_3$): δ 7.97 (d, *J* = 2.0 Hz, 1 H), 7.89 (d, *J* = 8.8 Hz, 1 H), 7.86-7.82 (m, 2 H), 7.57-7.51 (m, 3 H), 5.68 (q, *J* = 2.2 Hz, 1 H), 5.40 (q, *J* = 2.2 Hz, 1 H), 5.16 (dd, *J* = 7.2, 1.5 Hz, 1 H), 4.27 (br s, 1 H), 3.14 (d, *J* = 19.0 Hz, 1 H), 2.92 (ddq, *J* = 19.0, 7.2, 2.4 Hz, 1 H), 1.76 (s, 3 H); ^{13}C NMR (125 MHz, $CDCl_3$): δ 141.7, 136.4, 132.9, 132.5, 129.1, 128.4, 127.4, 127.0, 126.8, 125.8, 123.7, 117.3, 111.5, 93.9, 53.4, 45.4, 34.8, 22.6; IR (neat): 3056, 2245, 1552, 1363, 755 cm^{-1}. $[\alpha]_{23}^{D}$ = 32.33 (c 1.0, $CHCl_3$). Chiral HPLC: 99% ee, Chiralpak AD-H, 0.8 mL/min, 1% *i*-PrOH in heptane, λ = 254 nm, $t_{R,\ minor}$ = 36.2 min, $t_{R,\ major}$ = 43.2 min. Mass: Anal calcd for $C_{18}H_{16}N_2O_2$: C, 73.95; H, 5.52; N, 9.58; found: C, 74.22; H, 5.59; N, 9.41. R_f 0.26 (10% ethyl acetate in hexanes).



***tert*-Butyl-2((1*R*, 2*R*, 5*R*)-2-cyano-1-methyl-3-methylene-5-nitrocyclopentyl)-1*H*-indole-1-carboxylate (16):** The reaction was performed with 23.0 mg (0.076 mmol) of nitroalkene according to general procedure C and purified by flash chromatography (5% ethyl acetate in hexanes) to give the product as an oil (29.0 mg, >99% yield, >20:1 dr): ^{1}H NMR (500 MHz, $CDCl_3$): δ 8.21 (d, *J* = 7.6 Hz, 1 H), 7.88 (s, 1 H), 7.75 (d, *J* = 7.9 Hz, 1 H), 7.38 (t, *J* = 7.6 Hz, 1 H), 7.34-7.31 (m, 1 H), 5.68 (d, *J* = 2.5 Hz, 1 H), 5.43 (d, *J* = 6.3 Hz, 1 H), 5.34 (d, *J* = 2.4 Hz, 1 H), 4.22 (s, 1 H), 3.11 (d, *J* = 18.8 Hz, 1 H), 2.80 (ddq, *J* = 18.9, 7.2, 2.2 Hz,1 H), 1.75 (s, 3 H), 1.66 (s, 9 H); ^{13}C NMR (125 MHz, $CDCl_3$): δ 150.1, 149.0, 140.6, 135.9, 127.2, 125.0, 124.1, 123.0, 119.8, 117.1, 115.9, 111.5, 90.8, 84.4, 50.2, 45.5, 34.5, 28.1, 21.1; IR (neat): 2979, 1738, 1553 cm^{-1}. $[\alpha]_{23}^{D}$ = 49.04 (c 2.0, $CHCl_3$). Chiral HPLC: 95% ee, Chiralpak AD-H, 0.8 mL/min, 1% *i*-PrOH in heptane, λ = 254 nm, $t_{R,\ minor}$ = 11.6 min, $t_{R,\ major}$ = 16.2 min. HRMS: calcd for the amine (M + H) $C_{21}H_{26}N_3O_2$ 352.2025; found 352.2012. R_f 0.20 (10% ethyl acetate in hexanes).

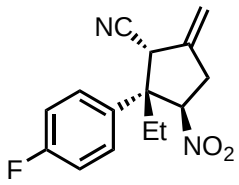


(1*R*, 2*R*, 3*R*)-2-(Furan-2-yl)-2-methyl-5-methylene-3-nitrocyclopentanecarbonitrile (17): The reaction was performed with 12.0 mg (0.076 mmol) of nitroalkene according to general procedure C and purified by flash chromatography (10% ethyl acetate in hexanes) to give the product as an oil (17.1 mg, 97% yield, >20:1 dr): ^{1}H NMR (500 MHz, $CDCl_3$): δ 7.39 (dd, *J* = 1.9, 0.8 Hz, 1 H), 6.42 (dd, *J* = 3.4, 0.8 Hz, 1 H), 6.36 (dd, *J* = 3.4, 1.9 Hz, 1 H), 5.50 (q, *J* = 2.5 Hz, 1 H), 5.31 (dtd, *J* = 3.1, 2.1, 0.9 Hz, 1 H), 5.14 (dd, *J* = 6.1, 3.0 Hz, 1 H), 4.12-4.10 (m, 1 H), 3.16-3.13 (m, 2 H), 1.60 (s, 3 H); ^{13}C NMR (125 MHz, $CDCl_3$): δ 151.7, 143.0, 141.1, 116.7, 111.8, 110.7, 108.4, 91.6, 50.9, 45.2, 35.7, 19.4; IR (neat): 3125, 2909, 2247, 1554, 1367 cm^{-1}. $[\alpha]_{23}^{D}$ = -4.49 (c 1.0, $CHCl_3$). Chiral HPLC: >99% ee, Chiralpak IC, 0.8 mL/min, 1% *i*-PrOH in heptane, λ = 254 nm, $t_{R,\ minor}$ = 27.0 min, $t_{R,\ major}$ = 32.8 min. HRMS: calcd for amine (M + H) $C_{12}H_{15}N_2O$ 203.1184; found 203.1178. R_f 0.37 (20% ethyl acetate in hexanes).

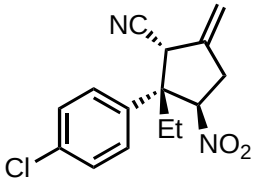


(1*R*, 2*S*, 3*R*)-2-Ethyl-5-methylene-3-nitro-2-phenylcyclopentanecarbonitrile (18): The reaction was performed with 13.0 mg (0.076 mmol) of nitroalkene according to general procedure C and purified by flash chromatography (10% ethyl acetate in hexanes) to give the product as an oil (18.4 mg, 94% yield, >20:1 dr): ^{1}H NMR (500 MHz, $CDCl_3$): δ 7.49-7.46 (m, 2 H), 7.43-7.39 (m, 2 H), 7.35-7.32 (m, 1 H), 5.61 (q, *J* = 2.2 Hz, 1 H), 5.32 (q, *J* = 2.2 Hz, 1 H), 5.04 (d, *J* = 6.8 Hz, 1 H), 4.23 (s, 1 H), 3.00 (dt, *J* = 19.0, 1.0 Hz, 1 H), 2.83 (ddq, *J* = 19.0, 7.0, 2.5 Hz, 1 H), 2.35 (dq, *J* = 14.7, 7.4 Hz, 1 H), 1.76 (dq, *J* = 14.5, 7.3 Hz, 1 H), 0.94 (t, *J* = 7.5 Hz, 3 H); ^{13}C NMR (125 MHz, $CDCl_3$): δ 142.1, 136.8, 129.2, 128.2, 127.1, 118.6, 111.2, 94.9, 57.5, 42.9, 34.8, 29.1, 9.1; IR (neat): 2979, 2243, 1551, 1365 cm^{-1}. $[\alpha]_{23}^{D}$ = -4.49 (c 1.0, $CHCl_3$). Chiral HPLC: 98% ee, Chiralpak AD-H, 0.8 mL/min, 1% *i*-PrOH in heptane, λ = 220 nm, $t_{R,\ minor}$ = 16.9 min, $t_{R,}$

$_{major}$ = 31.6 min. Mass: Anal calcd for $C_{15}H_{16}N_2O_2$: C, 70.29; H, 6.29; N, 10.93; found: C, 70.11; H, 6.35; N, 11.02. R_f 0.50 (20% ethyl acetate in hexanes).

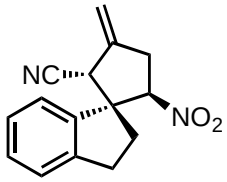


(1*R*, 2*S*, 3*R*)-2-Ethyl-2-(4-fluorophenyl)-5-methylene-3-nitrocyclopentanecarbonitrile (19): The reaction was performed with 15.0 mg (0.078 mmol) of nitroalkene according to general procedure C and purified by flash chromatography (10% ethyl acetate in hexanes) to give the product as a light yellow oil (21.0 mg, >99% yield, >20:1 dr): ^{1}H NMR (500 MHz, $CDCl_3$): δ 7.47-7.43 (m, 2 H), 7.13-7.09 (m, 2 H), 5.61 (d, *J* = 2.5 Hz, 1 H), 5.34 (d, *J* = 2.5 Hz, 1 H), 4.99 (d, *J* = 6.7 Hz, 1 H), 4.22 (s, 1 H), 3.02 (d, *J* = 19.0 Hz, 1 H), 2.82 (ddq, *J* = 19.0, 7.0, 2.4 Hz, 1 H), 2.30 (dq, *J* = 14.7, 7.4 Hz, 1 H), 1.77 (dq, *J* = 14.6, 7.3 Hz, 1 H), 0.93 (t, *J* = 7.5 Hz, 3 H); ^{13}C NMR (125 MHz, $CDCl_3$): δ 161.1, 141.8, 132.6, 129.1, 129.0, 118.4, 116.3, 116.1, 111.6, 94.7, 57.0, 43.1, 34.7, 29.1, 9.1; IR (neat): 2980, 2244, 1552, 1514 cm^{-1}. $[\alpha]_{23}^{D}$ = -6.42 (c 1.0, $CHCl_3$). Chiral HPLC: 98% ee, Chiralpak AD-H, 0.8 mL/min, 1% *i*-PrOH in heptane, λ = 254 nm, $t_{R, minor}$ = 24.6 min, $t_{R, major}$ = 31.4 min. HRMS: calcd for amine (M + H) $C_{15}H_{18}FN_2$ 245.1454: found 245.1455. R_f 0.23 (10% ethyl acetate in hexanes).



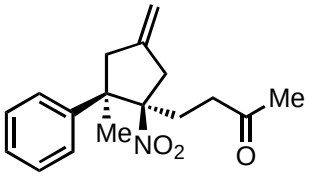
(1*R*, 2*S*, 3*R*)-2-(4-Chlorophenyl)-2-ethyl-5-methylene-3-nitrocyclopentanecarbonitrile (20): The reaction was performed with 16.0 mg (0.076 mmol) of nitroalkene according to general procedure C and purified by flash chromatography (10% ethyl acetate in hexanes) to give the product as an oil (21.6 mg, 98% yield, >20:1 dr): ^{1}H NMR (500 MHz, $CDCl_3$): δ 7.42-7.32 (m, 4 H), 5.62 (d, *J* = 2.2 Hz, 1 H), 5.35 (d, *J* = 2.2 Hz, 1 H), 5.02 (d, *J* = 6.8 Hz, 1 H), 4.23 (s, 1 H), 3.03 (d, *J* =

19.1 Hz, 1 H), 2.83 (ddq, *J* = 19.1, 7.1, 2.4 Hz, 1 H), 2.30 (dq, *J* = 14.8, 7.4 Hz, 1 H), 1.77 (dq, *J* = 14.6, 7.3 Hz, 1 H), 0.95 (t, *J* = 7.5 Hz, 3 H); ^{13}C NMR (125 MHz, $CDCl_3$): δ 141.5, 139.0, 135.3, 130.4, 128.6, 127.3, 125.5, 118.2, 111.7, 94.4, 57.2, 42.9, 34.7, 29.0, 9.1; IR (neat): 2979, 2244, 1553 cm^{-1}. $[\alpha]_{23}^{D}$ = 1.19 (c 1.0, $CHCl_3$). Chiral HPLC: 98% ee, Chiralpak AD-H, 0.8 mL/min, 1% *i*-PrOH in heptane, λ = 254 nm, $t_{R, minor}$ = 19.5 min, $t_{R, major}$ = 25.4 min. HRMS: calcd for amine (M + H) $C_{15}H_{18}ClN_2$ 261.1159: found 261.1154. R_f 0.19 (10% ethyl acetate in hexanes).

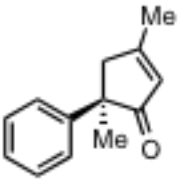


(1*R*,2*S*,5*S*)-3-Methylene-5-nitro-2',3'-dihydrospiro[cyclopentane-1,1'-indene]-2-carbonitrile (21): The reaction was performed with 13.3 mg (0.076 mmol) of nitroalkene according to general procedure C and purified by flash chromatography (15% ethyl acetate in hexanes) to give the product as a white solid (19.1 mg, 99% yield, 92% ee), mp 122-124 ºC. ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.33-7.28 (m, 2H), 7.24-7.19 (m, 1H), 7.12 (d, *J* = 7.5 Hz, 1H), 5.60 (q, *J* = 2.5 Hz, 1H), 5.46-5.44 (m, 1H), 4.94 (t, *J* = 4.6 Hz, 1H), 4.32-4.29 (m, 1H), 3.20 (dq, *J* = 2.2, 2.2, 2.2, 4.6 Hz, 2H), 3.09-3.04 (m, 2H), 2.30 (dt, *J* = 8.3, 8.3, 13.5 Hz, 1H), 2.14 (ddd, *J* = 5.2, 6.8, 13.5 Hz, 1H). ^{13}C NMR (100 MHz, $CDCl_3$): δ 143.9, 141.6, 141.5, 129.6, 127.6, 125.9, 123.5, 117.7, 113.2, 92.1, 62.2, 44.1, 35.8, 34.2, 30.6. IR (thin film): 2926, 2854, 2245, 1665, 1550, 1476, 1458, 1366, 1300 cm^{-1}. $[\alpha]_{23}^{D}$ = -21.4 (c 0.88, $CHCl_3$). Chiral HPLC: Chiralpak AD-H, 0.8 mL/min, 1% *i*-PrOH in heptane, λ = 220 nm, $t_{R, minor}$ = 23.6 min, $t_{R, major}$ = 26.6 min. HRMS: calcd for the amine ($M+Na^+$) $C_{15}H_{16}N_2Na$ 247.1211; found 247.1221. R_f 0.23 (15% ethyl acetate in hexanes).

F. Cycloadduct Derviatives

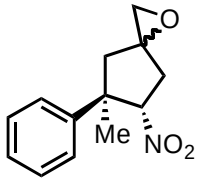


4-((1*R*,2*R*)-2-Methyl-4-methylene-1-nitro-2-phenylcyclopentyl)butan-2-one (4): To a solution of nitrocyclopentane **3a** and **3b** (18.5 mg as a 3:1 mixture, 0.085 mmol) in acetonitrile (1.5 mL) was added DBU (14 µL, 0.094 mmol) and 3-buten-2-one (7.6 µL, 0.094 mmol). The pale yellow solution was stirred at room temperature for 45 minutes before a second charge of 3-buten-2-one (2 µL, 0.025 mmol) was added. After another 45 minutes, the reaction was concentrated and filtered through a plug of silica gel (15% ethyl acetate in hexanes) to give the product as a clear, colorless oil (21.3 mg, 87% yield, dr 7.5:1, 57% ee). The major diastereomer was purified by flash chromatography (10% ethyl acetate in hexanes) for characterization purposes. ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.36-7.24 (m, 5H), 5.13 (quintet, *J* = 2.2 Hz, 1H), 5.07 (quintet, *J* = 2.2 Hz, 1H), 3.44 (d, *J* = 16.7 Hz, 1H), 3.37 (d, *J* = 17.8 Hz, 1H), 2.71-2.64 (m, 1H), 2.60 (dq, *J* = 1.8, 17.8 Hz, 1H), 2.46 (dq, *J* = 1.8, 16.7 Hz, 1H), 2.37-2.31 (m, 2H), 2.19-2.11 (m, 1H), 2.14 (s, 3H), 1.57 (s, 3H). ^{13}C NMR (100 MHz, $CDCl_3$): δ 206.9, 144.9, 141.4, 128.7, 127.9, 126.8, 109.1, 102.8, 53.3, 44.8, 40.0, 39.2, 30.4, 27.2, 24.3. IR (thin film): 3061, 2930, 1717, 1663, 1537, 1499, 1436, 1354 cm^{-1}. $[\alpha]_{24}^{D}$ = -26.86 (c 1.44, $CHCl_3$). Chiral HPLC: Chiralpak IC, 0.8 mL/min, 10% *i*-PrOH in heptane, λ = 220 nm, $t_{R,\ minor}$ = 12.7 min, $t_{R,\ major}$ = 29.5 min. R_f 0.50 (30% ethyl acetate in hexanes).

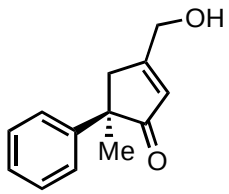


(R)-3-methyl-5-(naphthalen-2-yl)cyclopent-2-enone (27): To a solution of nitrocyclopentane **3a** (20.3 mg, 0.09 mmol) in THF (1.0 mL) at -78 ºC was added KO*t*-Bu (102 µL, 1 M in THF, 0.10 mmol) and the yellow solution was stirred for 20 minutes at -78 ºC. The dry ice-acetone bath was then removed, dimethyldioxirane (1.1 mL, approx. 1 M in acetone, 0.11 mmol) was added, and the mixture was immediately placed in a bath at approximately -20 ºC. The mixture was stirred for 10 minutes and then added

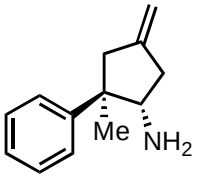
to a well-stirred solution of 0.25 M, pH 7.0 phosphate buffer (5 mL) and extracted with diethyl ether (3 x 3 mL). The combined organic layers were washed with brine, dried over $MgSO_4$, concentrated and purified by flash chromatography (15% ethyl acetate in hexanes) to yield the product as a clear, colorless oil (11.3 mg, 65% yield). ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.33-7.18 (m, 5H), 6.00 (s, 1H), 2.98 (d, *J* = 19.2 Hz, 1H), 2.75 (d, *J* = 19.2 Hz, 1H), 2.18 (s, 3H), 1.53 (s, 3H). ^{13}C NMR (100 MHz, $CDCl_3$): δ 212.1, 176.8, 144.3, 129.0, 128.9, 126.8, 126.2, 52.5, 52.2, 24.6, 19.7. IR (thin film): 3059, 2964, 2924, 1700, 1625, 1496, 1432 cm^{-1}. $[\alpha]_{24}^{D}$ = -7.4 (c 0.96, $CHCl_3$). Chiral HPLC: Chiralpak AD-H, 0.8 mL/min, 2% *i*-PrOH in heptane, λ = 220 nm, t_R, major = 15.6 min, t_R, minor = 17.1 min. HRMS: calcd for ($M+H^+$) $C_{13}H_{15}O$ 187.1123; found 187.1114. R_f 0.42 (30% ethyl acetate in hexanes).



(5*R*,6*S*)-5-Methyl-6-nitro-5-phenyl-1-oxaspiro[2.4]heptane (28): To a solution of nitrocyclopentane **3a** (15.3 mg, 0.071 mmol) in chloroform (0.5 mL) was added *m*-chloroperoxybenzoic acid (18.2 mg, 0.11 mmol). The solution was stirred for 15 hours at room temperature, during which a white solid precipitated. The suspension was diluted with diethyl ether (3 mL), and washed with sat. $NaHSO_3$ (2 mL), sat. $NaHCO_3$ (2 x 2 mL), H_2O (2 mL) and brine (2 mL). The organic layer was dried over $MgSO_4$, concentrated and filtered through a plug of SiO_2 (30% ethyl acetate in hexanes) to yield the product as a clear, colorless oil (15.9 mg, 97% yield, 2.5:1 dr, as determined by ^{1}H NMR). ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.58-7.55 (m, 1.4H), 7.45-7.37 (m, 2.6H), 7.32-7.28 (m, 1H), 5.21-5.16 (m, 1H), 3.06 (d, *J* = 4.7 Hz, 0.7H), 3.04 (d, *J* = 4.7 Hz, 0.7H), 2.98 (d, *J* = 4.7 Hz, 0.3H), 2.88 (d, *J* = 4.7 Hz, 0.3H), 2.62-2.41 (m, 3H), 2.32 (d, *J* = 14.7 Hz, 0.3H), 2.17 (dd, *J* = 6.9, 16.0 Hz, 0.7H), 1.53 (s, 0.9H), 1.46 (s, 2.1H).

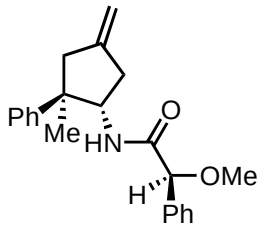


(*R*)-3-(Hydroxymethyl)-5-methyl-5-phenylcyclopent-2-enone (29): To a solution of epoxide **28** (16.1 mg, 0.07 mmol) in THF (1 mL) at -78 ºC was added KO*t*-Bu (100 µL, 1 M in THF, 0.1 mmol) and the yellow solution was stirred for 20 minutes at -78 ºC. Dimethyldioxirane (1.5 mL, approx. 0.1 M in acetone, 0.15 mmol) was added and the suspension was warmed to 0 ºC. After 15 minutes, the reaction was warmed to room temperature and stirred another 15 minutes, then added to a solution of 0.25 M, pH 7.0 phosphate buffer (5 mL) and extracted with diethyl ether (3 x 5 mL). The combined organic layers were washed with brine, dried over $MgSO_4$, concentrated and purified by flash chromatography (2% methanol in dichloromethane) to yield the product as a clear, colorless oil (10.2 mg, 73% yield). ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.32-7.19 (m, 5H), 6.24 (quintet, *J* = 1.7 Hz, 1H), 4.49 (s, 2H), 2.97 (d, *J* = 18.5 Hz, 1H), 2.74 (d, *J* = 18.5 Hz, 1H), 2.57 (bs, 1H), 1.54 (s, 3H). ^{13}C NMR (100 MHz, $CDCl_3$): δ 211.6, 179.3, 143.9, 128.9, 127.0, 126.4, 126.2, 63.1, 51.9, 47.1, 24.6. IR (thin film): 3416, 3060, 2926, 1695, 1622, 1496, 1445 cm^{-1}. $[\alpha]_{23}^{D}$ = -3.4 (c 0.97, $CHCl_3$). Chiral HPLC: Chiralpak AD-H, 0.8 mL/min, 10% *i*-PrOH in heptane, λ = 220 nm, $t_{R, major}$ = 8.1 min, $t_{R, minor}$ = 8.8 min. HRMS: calcd for ($M+Na^+$) $C_{13}H_{14}O_2Na$ 225.0891; found 225.0897. R_f 0.27 (10% methanol in dichloromethane).

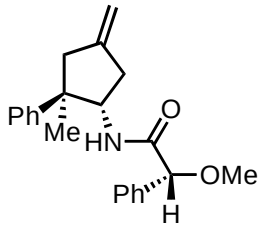


(1*S*,2*R*)-2-Methyl-4-methylene-2-phenylcyclopentanamine (30): A suspension of nitrocyclopentane **3a** (13.6 mg, 0.063 mmol) in concentrated HCl (126 µL, 1.52 mmol) and methanol (0.65 mL) was placed in an ambient water bath. Zinc dust (167.4 mg, 1.30 mmol) was carefully added with vigorous stirring over 1 minute, and the mixture was stirred for 10 minutes. The reaction was then quenched with sat. $NaHCO_3$ (10 mL) and extracted with ethyl acetate (3 x 5 mL). The combined extracts were dried over $MgSO_4$,

concentrated and purified by flash chromatography (dichloromethane to 10% methanol in dichloromethane) to afford the product as a clear, colorless oil (6.6 mg, 56% yield). ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.43-7.19 (m, 5H), 4.95 (s, 1H), 4.92 (s, 1H), 3.53 (t, *J* = 8.9 Hz, 1H), 2.85-2.73 (m, 2H), 2.50 (dd, *J* = 1.5, 16.0 Hz, 1H), 2.26-2.17 (m, 1H), 1.69 (br s, 2H), 1.27 (s, 3H).



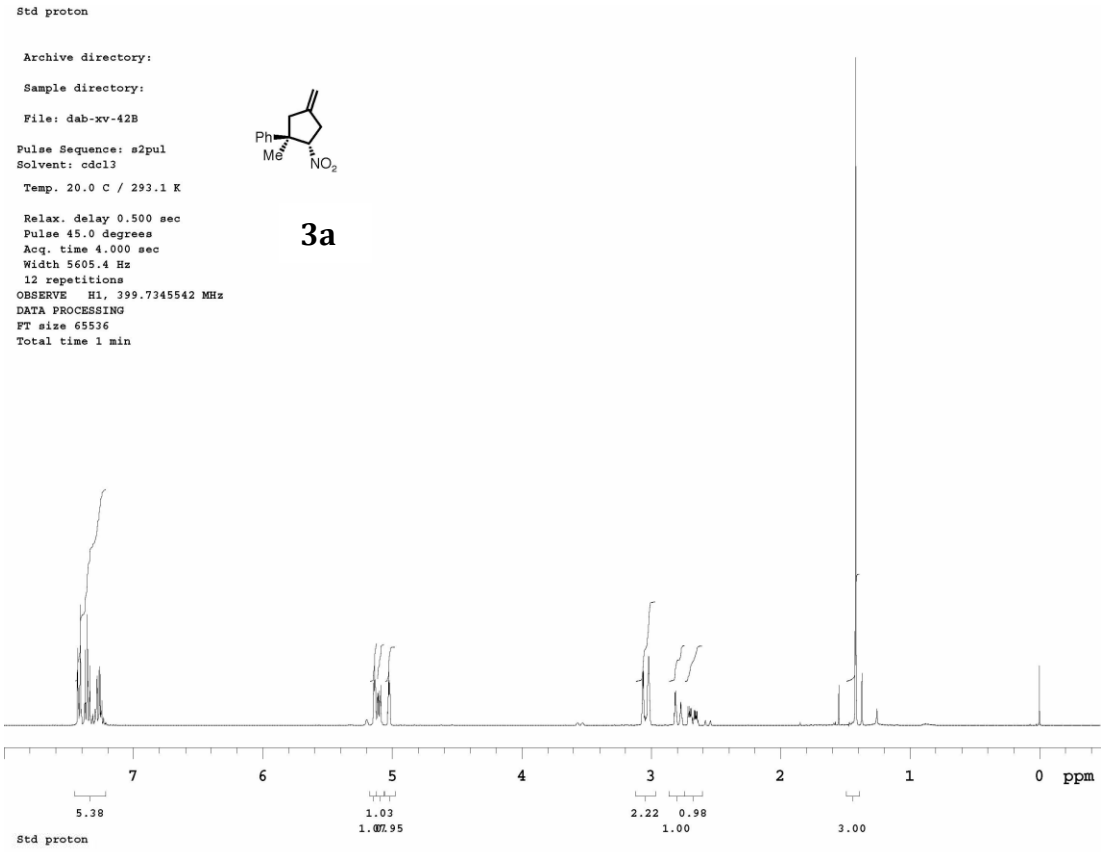
(*S*)-2-Methoxy-*N*-((1*S*,2*R*)-2-methyl-4-methylene-2-phenylcyclopentyl)-2-phenylacetamide (31): To a solution of aminocyclopentane **30** (3.4 mg, 0.018 mmol) in dichloromethane (300 µL) was added (*S*)-*O*-methylmandelic acid (3.3 mg, 0.020 mmol) followed by *N,N'*-dicyclohexylcarbodiimide (4.5 mg, 0.022 mmol). The mixture was stirred under nitrogen for 1 hour, filtered, washed with dichloromethane and concentrated. Purified by flash chromatography (15% ethyl acetate in hexanes) to yield the product as a clear, colorless oil (5.0 mg, 83% yield). ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.43-7.21 (m, 9H observed), 6.90 (d, *J* = 8.9 Hz, 1H), 4.99 (s, 1H), 4.91 (s, 1H), 4.75 (q, *J* = 8.1 Hz, 1H), 4.61 (s, 1H), 3.33 (s, 3H), 2.83 (dd, *J* = 7.9 Hz, 1H), 2.79 (d, *J* = 17.4 Hz, 1H), 2.56 (d, *J* = 7.4 Hz, 1H), 2.20 (dd, *J* = 7.9 Hz, 1H), 1.38 (s, 3H). R_f 0.41 (30% ethyl acetate in hexanes).



(*S*)-2-Methoxy-*N*-((1*S*,2*R*)-2-methyl-4-methylene-2-phenylcyclopentyl)-2-phenylacetamide (32): To a solution of aminocyclopentane **30** (3.2 mg, 0.017 mmol) in dichloromethane (300 µL) was added (*R*)-*O*-methylmandelic acid (3.1 mg, 0.019 mmol) followed by *N,N'*-dicyclohexylcarbodiimide (4.3 mg, 0.021 mmol). The mixture was

stirred under nitrogen for 1 hour, filtered, washed with dichloromethane and concentrated. Purified by flash chromatography (15 to 20% ethyl acetate in hexanes) to yield the product as a clear, colorless oil (4.7 mg, 78% yield). ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.33-7.12 (m, 8H observed), 6.90 (bd, *J* = 8.8 Hz, 1H), 5.02 (s, 1H), 4.95 (s, 1H), 4.69 (q, *J* = 8.7 Hz, 1H), 4.61 (s, 1H), 3.35 (s, 3H), 2.90 (dd, *J* = 8.3, 16.7 Hz, 1H), 2.80 (d, *J* = 16.7 Hz, 1H), 2.55 (dq, *J* = 1.6, 16.1 Hz, 1H), 2.30 (ddq, *J* = 2.3, 7.7, 17.1 Hz, 1H), 1.29 (s, 3H). R_f 0.33 (30% ethyl acetate in hexanes).

F. Spectra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Width 5605.4 Hz
12 repetitions
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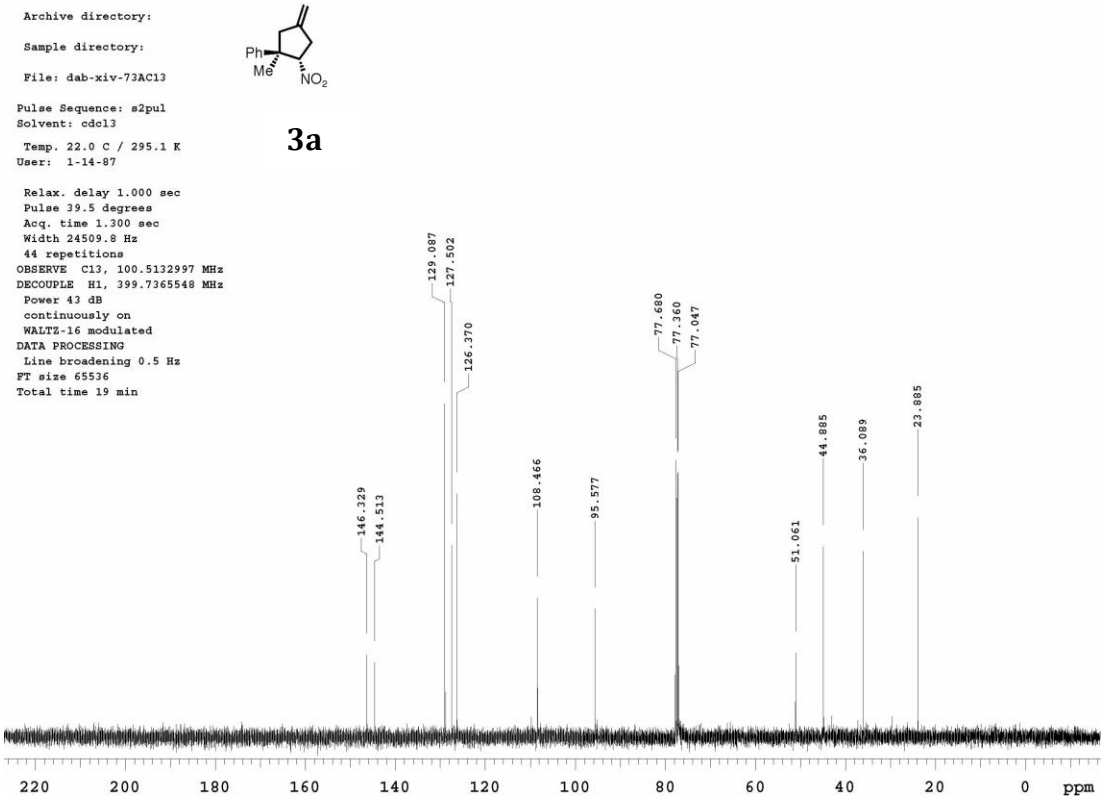
1.00
3.00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Pulse 39.5 degrees
Acq. time 1.300 sec
Width 24509.8 Hz
44 repetitions
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DECOUPLE H1, 399.7365548 MHz
Power 43 dB
continuously on
WALTZ-16 modulated
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126.370
108.466
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77.360
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80
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40
20
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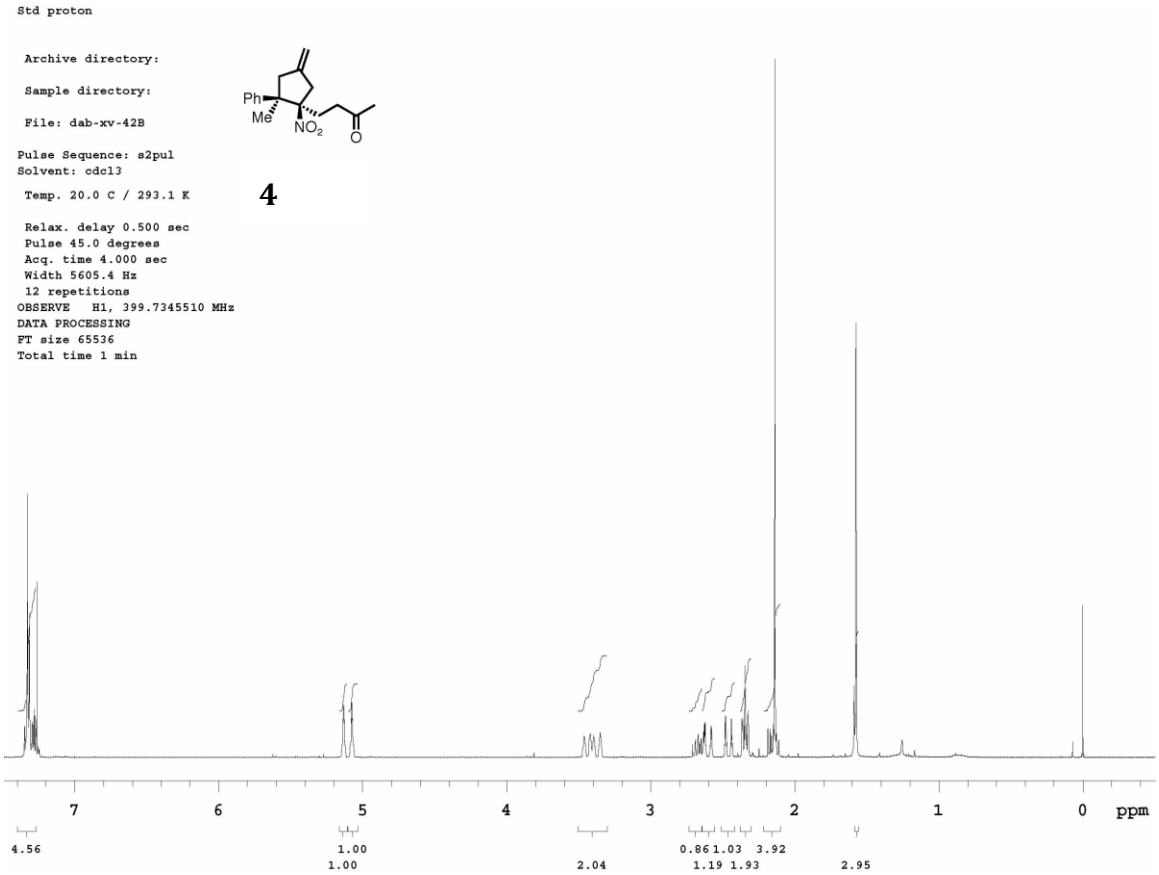
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3
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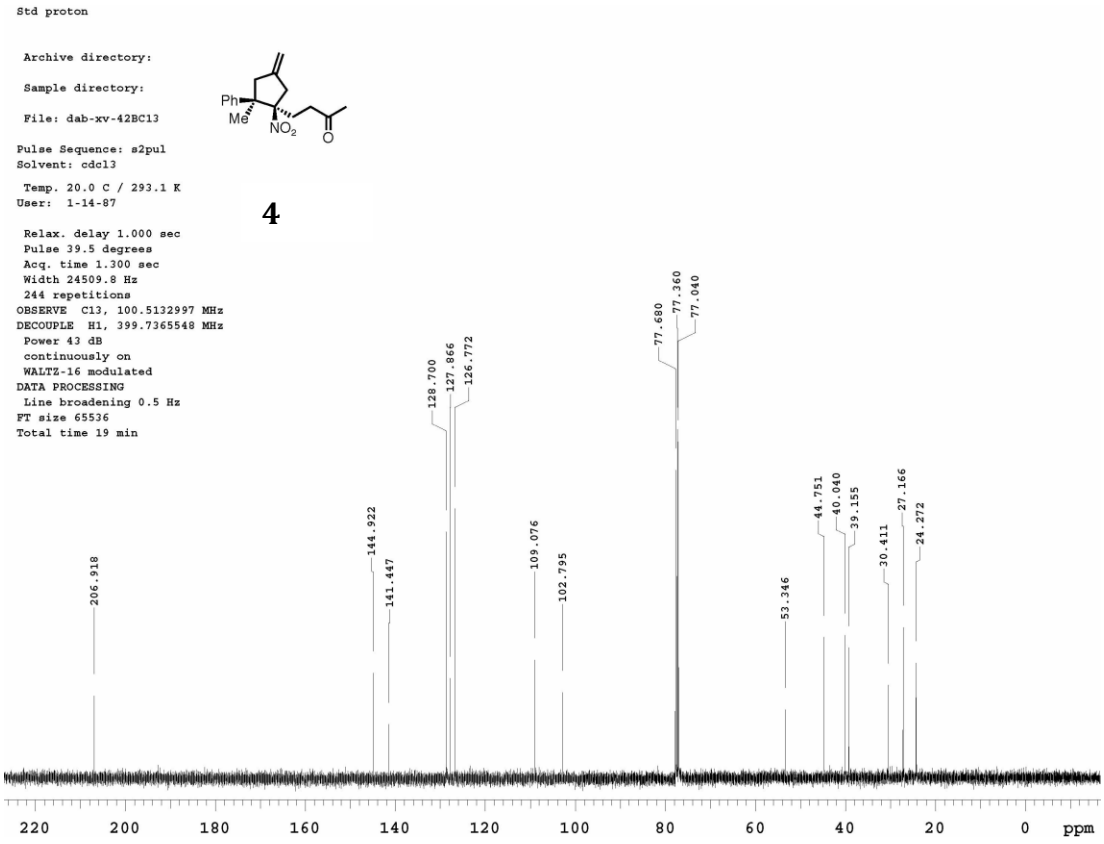
2.95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WALTZ-16 modulated
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O
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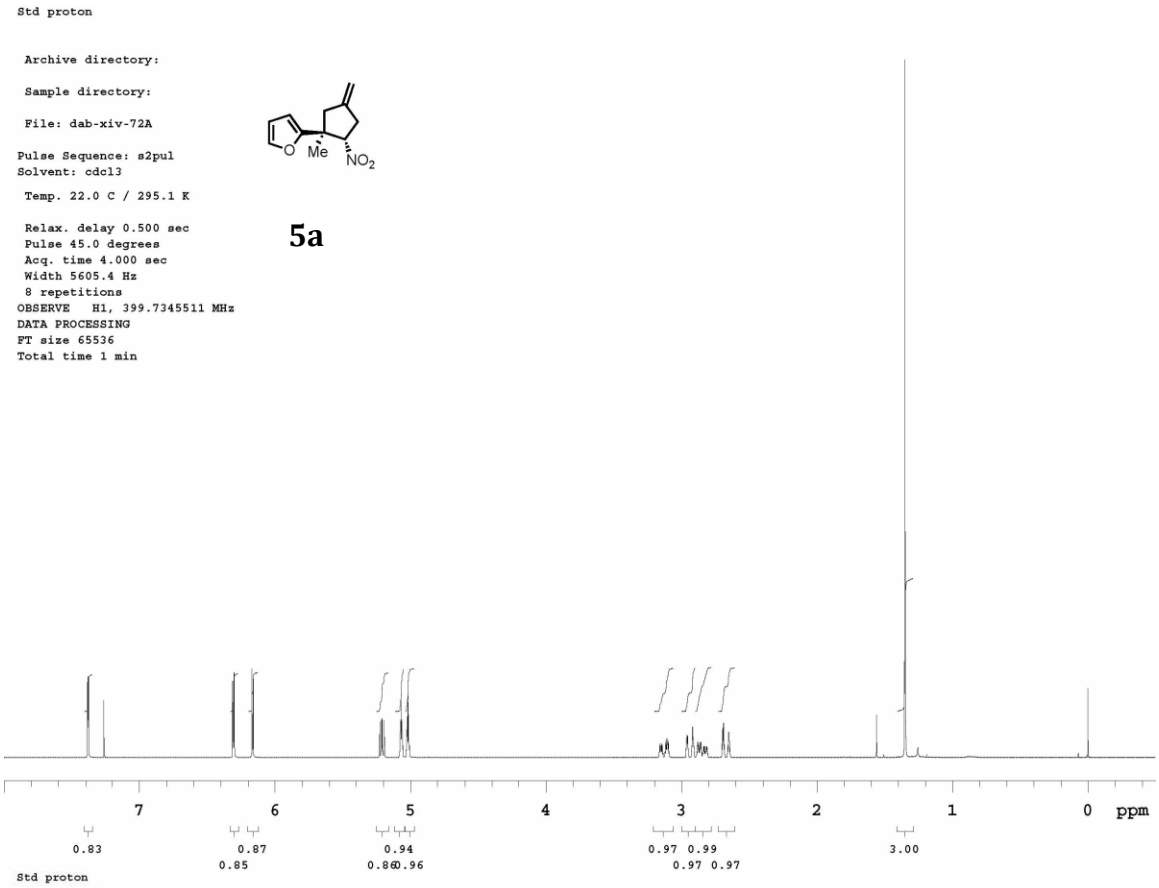
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DATA PROCESSING
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Total time 1 min
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NO2
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7
6
5
4
3
2
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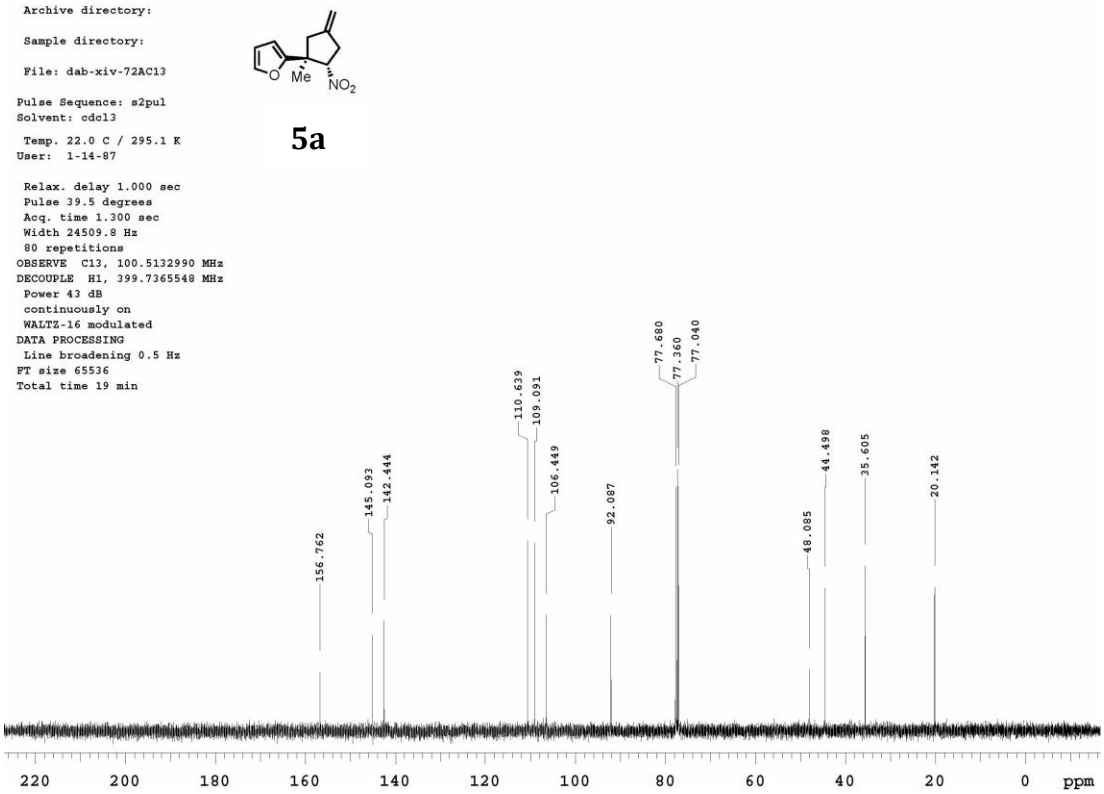
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Width 24509.8 Hz
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DECOUPLE H1, 399.7365548 MHz
Power 43 dB
continuously on
WALTZ-16 modulated
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NO2
5a
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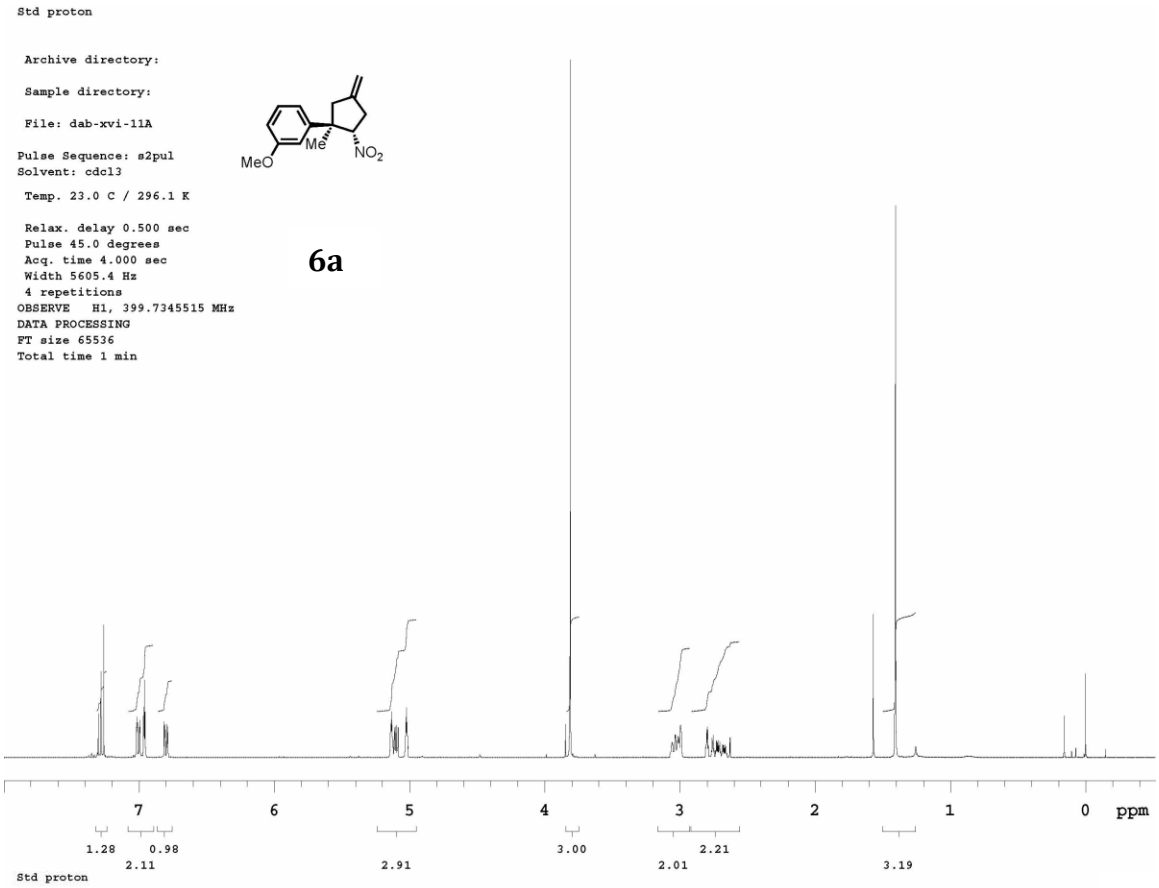
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NO2
6a
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6
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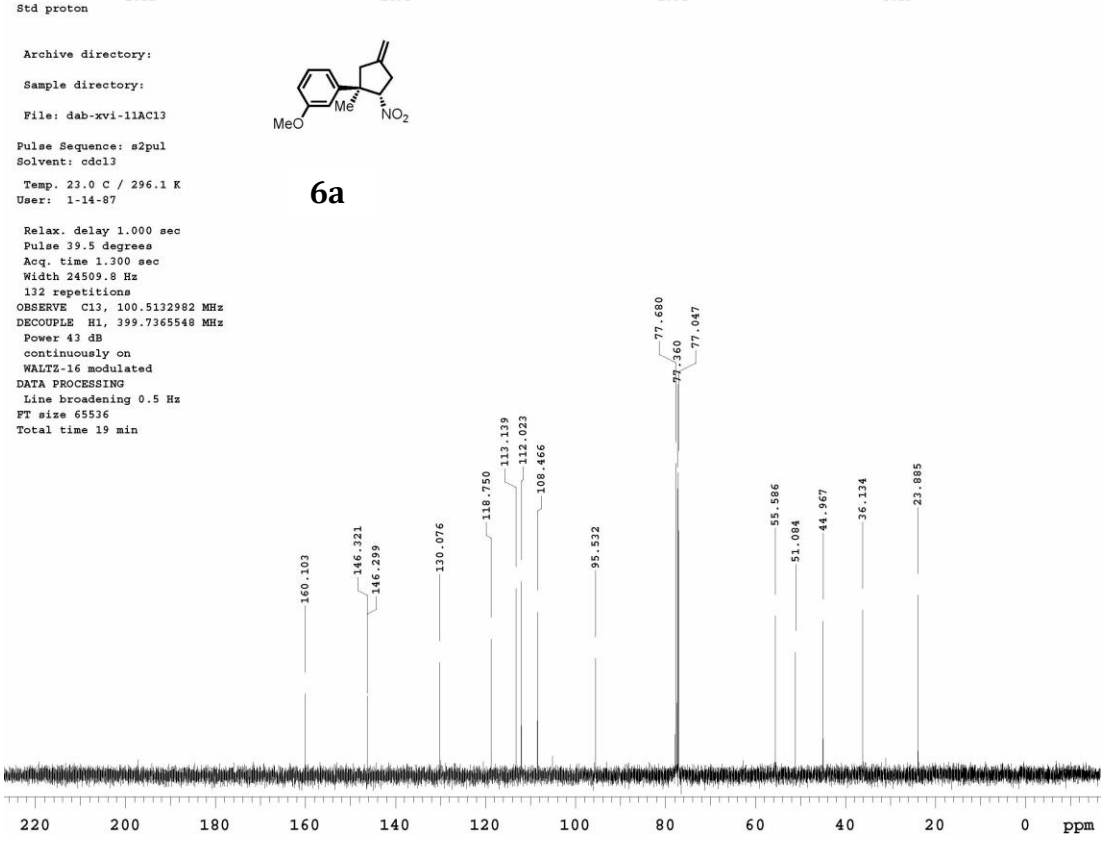
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Acq. time 1.300 sec
Width 24509.8 Hz
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DECOUPLE H1, 399.7365548 MHz
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NO2
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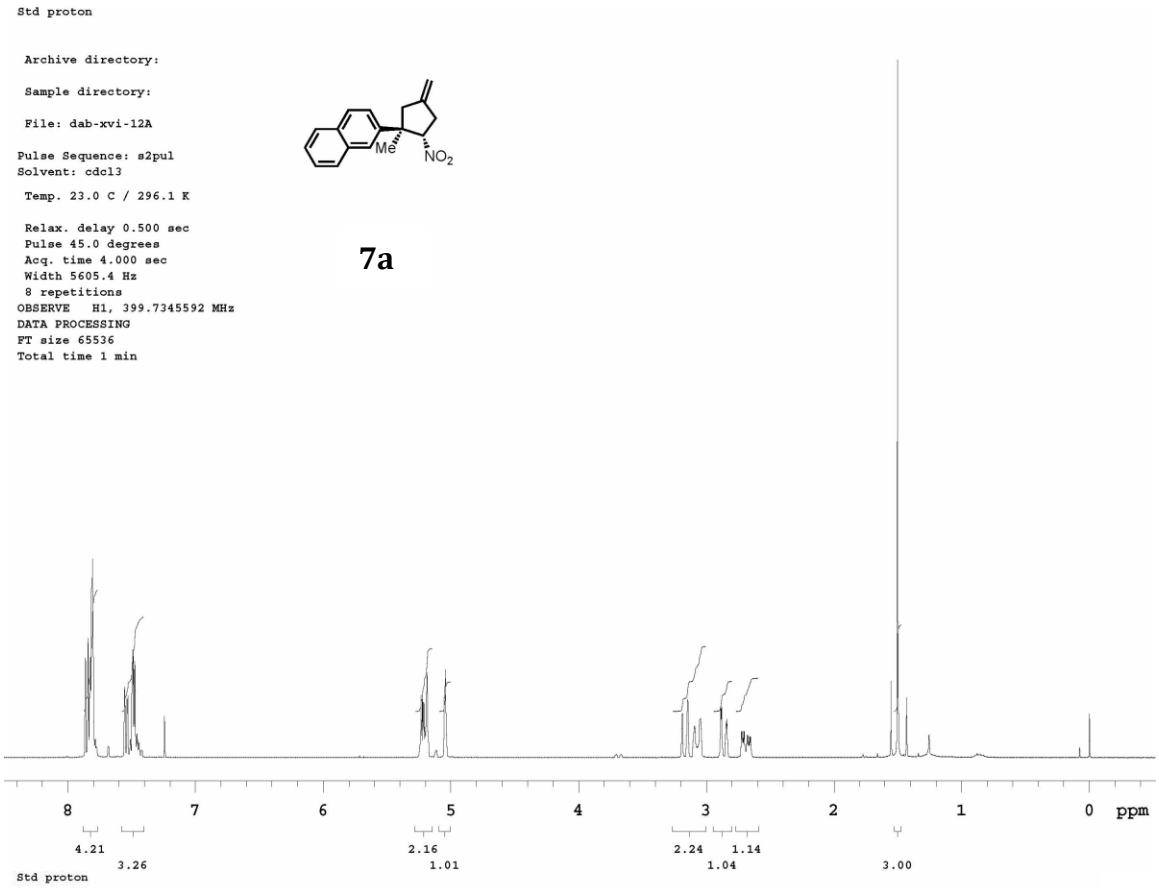
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8 repetitions
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DATA PROCESSING
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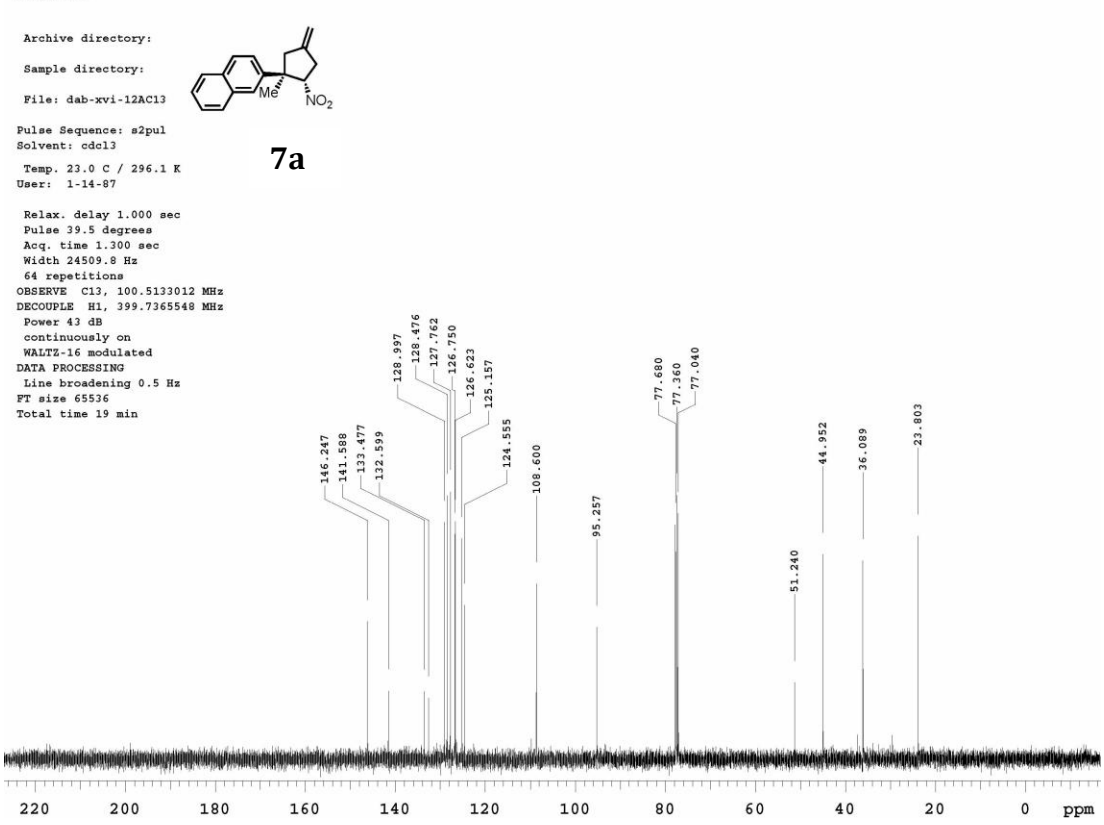
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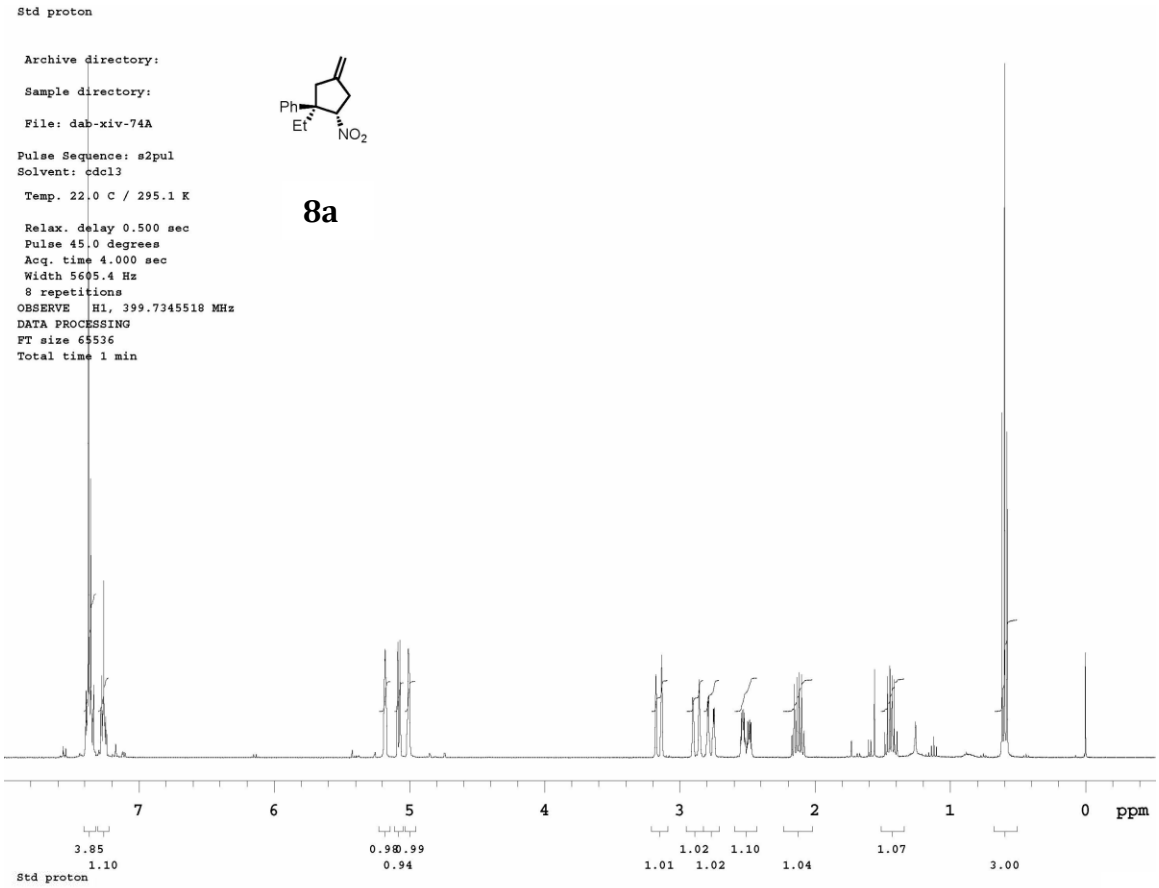
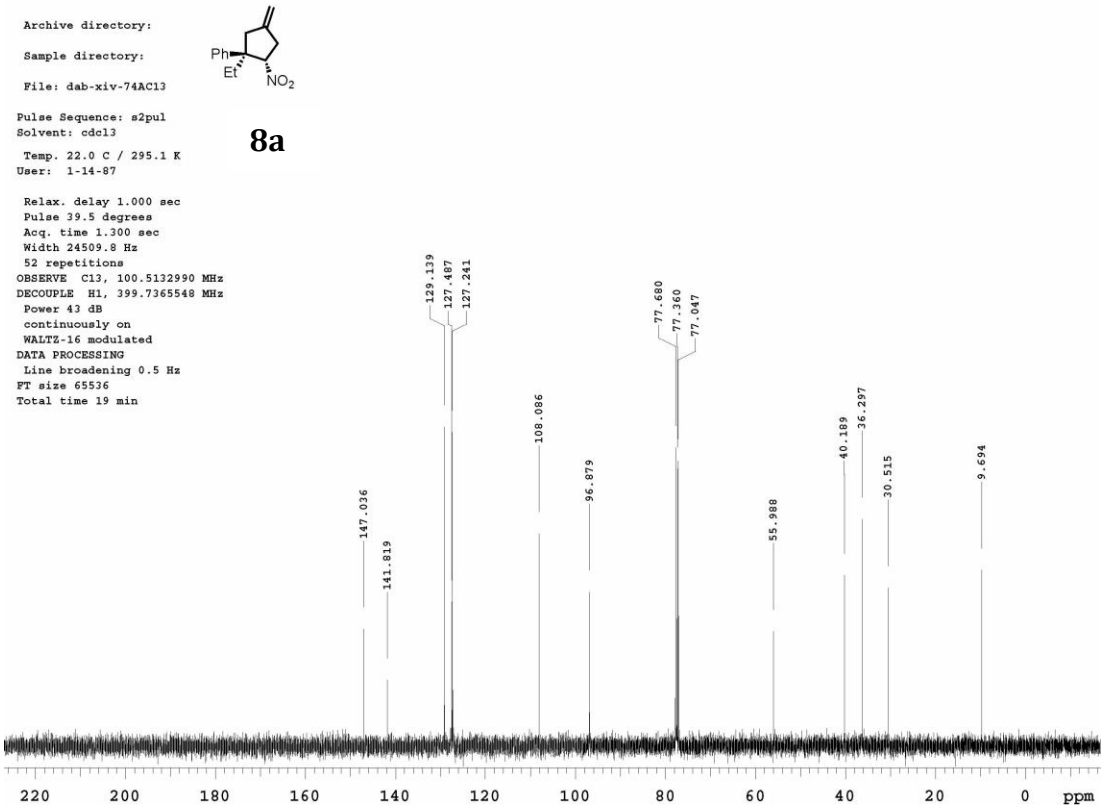
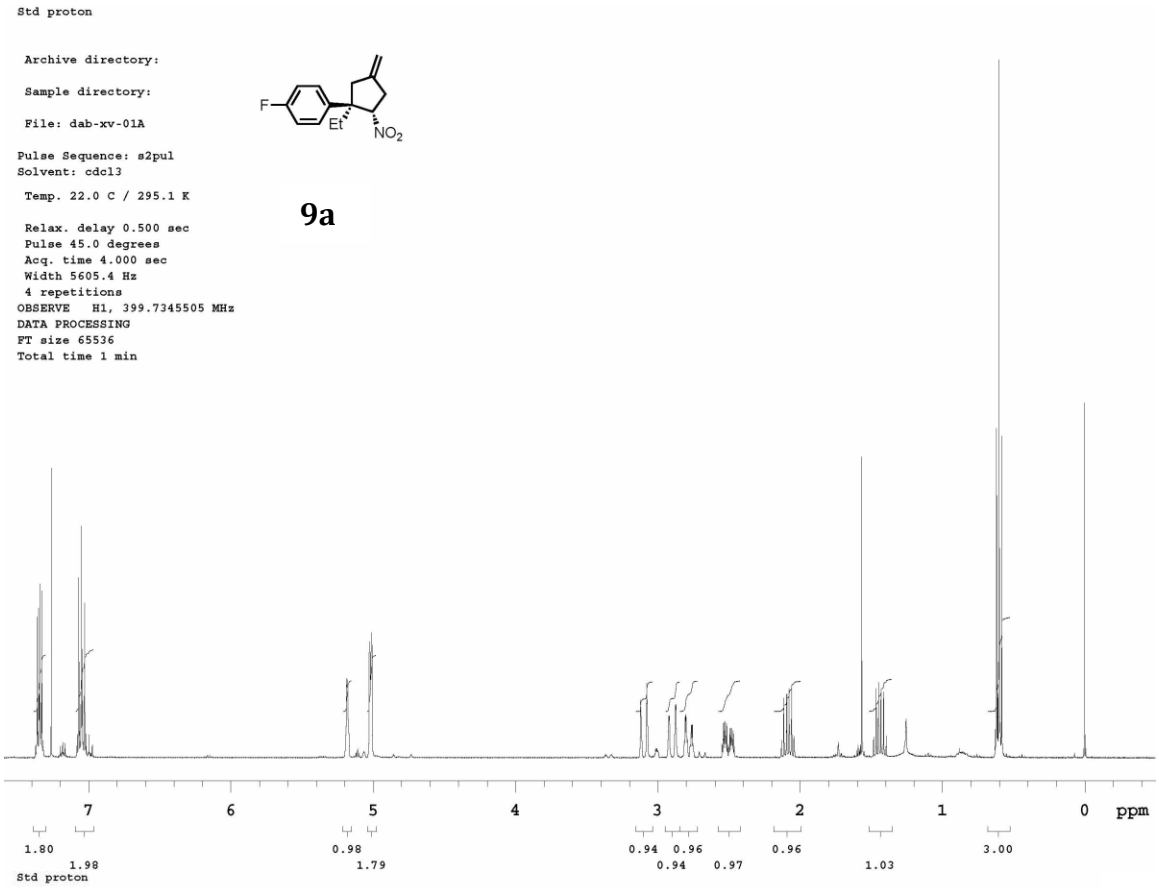
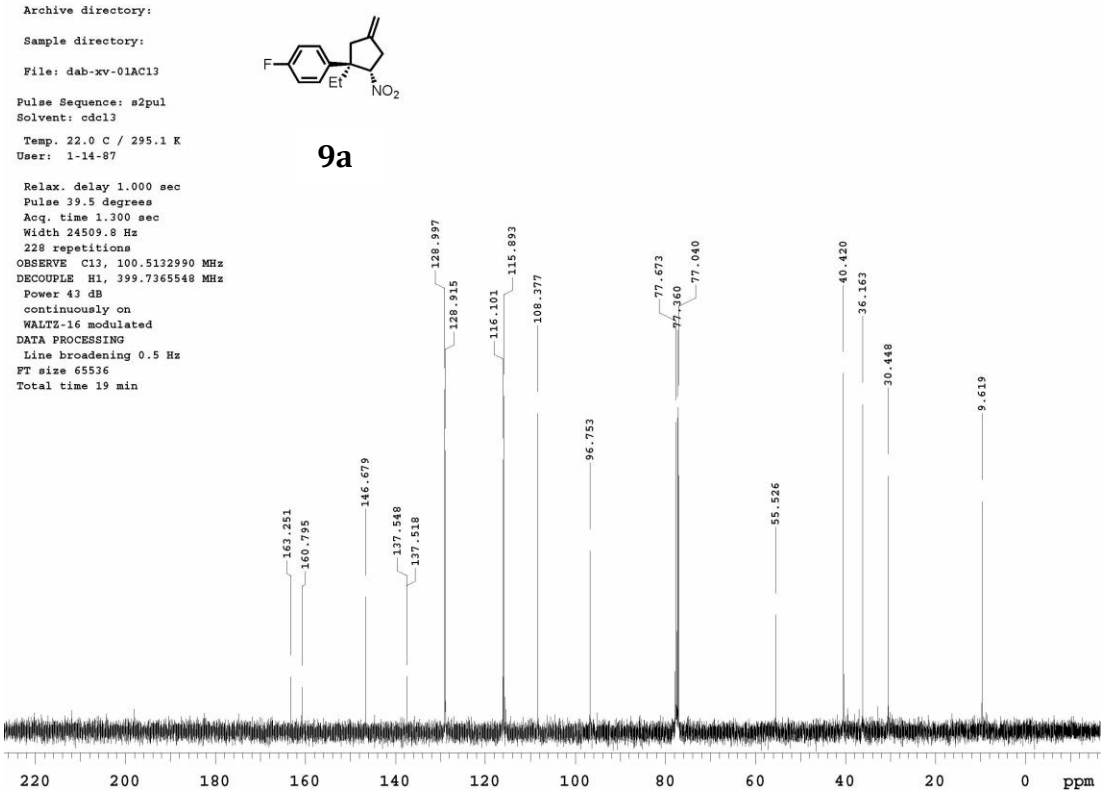
Pulse 39.5 degrees
Acq. time 1.300 sec
Width 24509.8 Hz
64 repetitions
OBSERVE C13, 100.5133012 MHz
DECOUPLE H1, 399.7365548 MHz
Power 43 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 0.5 Hz
FT size 65536
Total time 19 min
Me
NO2
7a
146.247
141.588
133.477
132.599
128.997
128.476
127.762
126.750
126.623
125.157
124.555
108.600
95.257
77.680
77.360
77.040
51.240
44.952
36.089
23.803
220
200
180
160
140
120
100
80
60
40
20
0
ppm


Std proton
Archive directory:
Sample directory:
File: dab-xiv-74A
Pulse Sequence: s2pul
Solvent: cdcl3
Temp. 22.0 C / 295.1 K
Relax. delay 0.500 sec
Pulse 45.0 degrees
Acq. time 4.000 sec
Width 5605.4 Hz
8 repetitions
OBSERVE H1, 399.7345518 MHz
DATA PROCESSING
FT size 65536
Total time 1 min
Ph
Et
NO2
8a
7
6
5
4
3
2
1
0
ppm
3.85
1.10
0.98
0.99
0.94
1.01
1.02
1.02
1.10
1.04
1.07
3.00


Std proton
Archive directory:
Sample directory:
File: dab-xiv-74AC13
Pulse Sequence: s2pul
Solvent: cdcl3
Temp. 22.0 C / 295.1 K
User: 1-14-87
Relax. delay 1.000 sec
Pulse 39.5 degrees
Acq. time 1.300 sec
Width 24509.8 Hz
52 repetitions
OBSERVE C13, 100.5132990 MHz
DECOUPLE H1, 399.7365548 MHz
Power 43 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 0.5 Hz
FT size 65536
Total time 19 min
Ph
Et
NO2
8a
147.036
141.819
129.139
127.487
127.241
108.086
96.879
77.680
77.360
77.047
55.988
40.189
36.297
30.515
9.694
220
200
180
160
140
120
100
80
60
40
20
0
ppm


Std proton
Archive directory:
Sample directory:
File: dab-xv-01A
Pulse Sequence: s2pul
Solvent: cdcl3
Temp. 22.0 C / 295.1 K
Relax. delay 0.500 sec
Pulse 45.0 degrees
Acq. time 4.000 sec
Width 5605.4 Hz
4 repetitions
OBSERVE H1, 399.7345505 MHz
DATA PROCESSING
FT size 65536
Total time 1 min
F
Et
NO2
9a
7
6
5
4
3
2
1
0
ppm
1.80
1.98
0.98
1.79
0.94
0.96
0.94
0.97
0.96
1.03
3.00

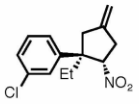

Std proton
Archive directory:
Sample directory:
File: dab-xv-01AC13
Pulse Sequence: s2pul
Solvent: cdcl3
Temp. 22.0 C / 295.1 K
User: 1-14-87
Relax. delay 1.000 sec
Pulse 39.5 degrees
Acq. time 1.300 sec
Width 24509.8 Hz
228 repetitions
OBSERVE C13, 100.5132990 MHz
DECOUPLE H1, 399.7365548 MHz
Power 43 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 0.5 Hz
FT size 65536
Total time 19 min
F
Et
NO2
9a
163.251
160.795
146.679
137.548
137.518
128.997
128.915
116.101
115.893
108.377
96.753
77.673
77.360
77.040
55.526
40.420
36.163
30.448
9.619
220
200
180
160
140
120
100
80
60
40
20
0
ppm

Std proton

Archive directory:

Sample directory:

File: dab-xiv-88A



10a

Pulse Sequence: s2pul
Solvent: cdcl3

Temp. 22.0 C / 295.1 K

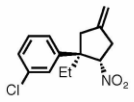
Relax. delay 0.500 sec
Pulse 45.0 degrees
Acq. time 4.000 sec
Width 5605.4 Hz
8 repetitions
OBSERVE H1, 399.7345505 MHz
DATA PROCESSING
FT size 65536
Total time 1 min

Std proton

Archive directory:

Sample directory:

File: dab-xiv-88AC13

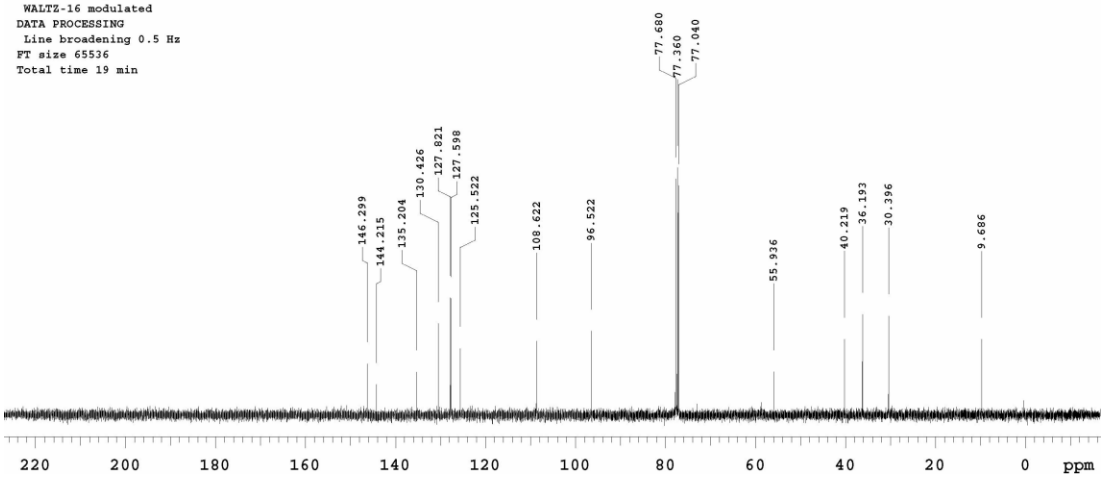


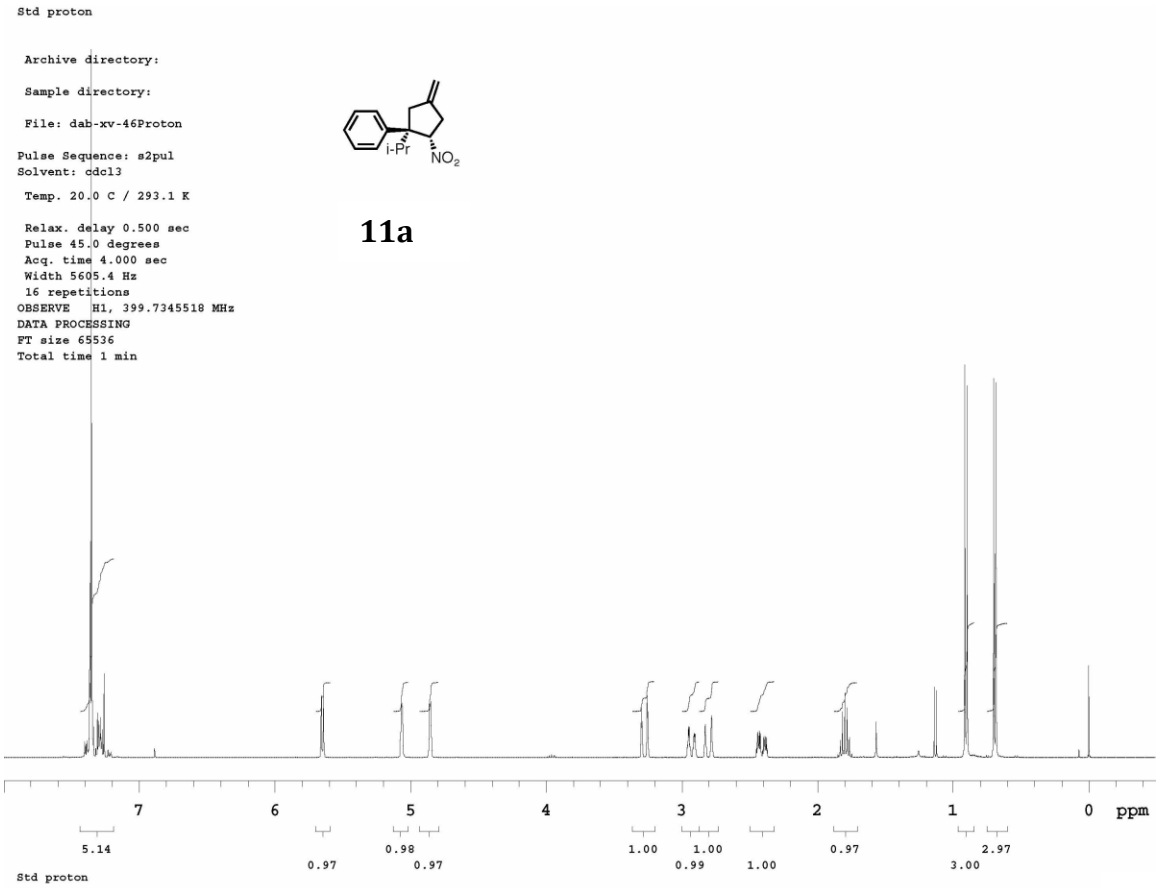
10a

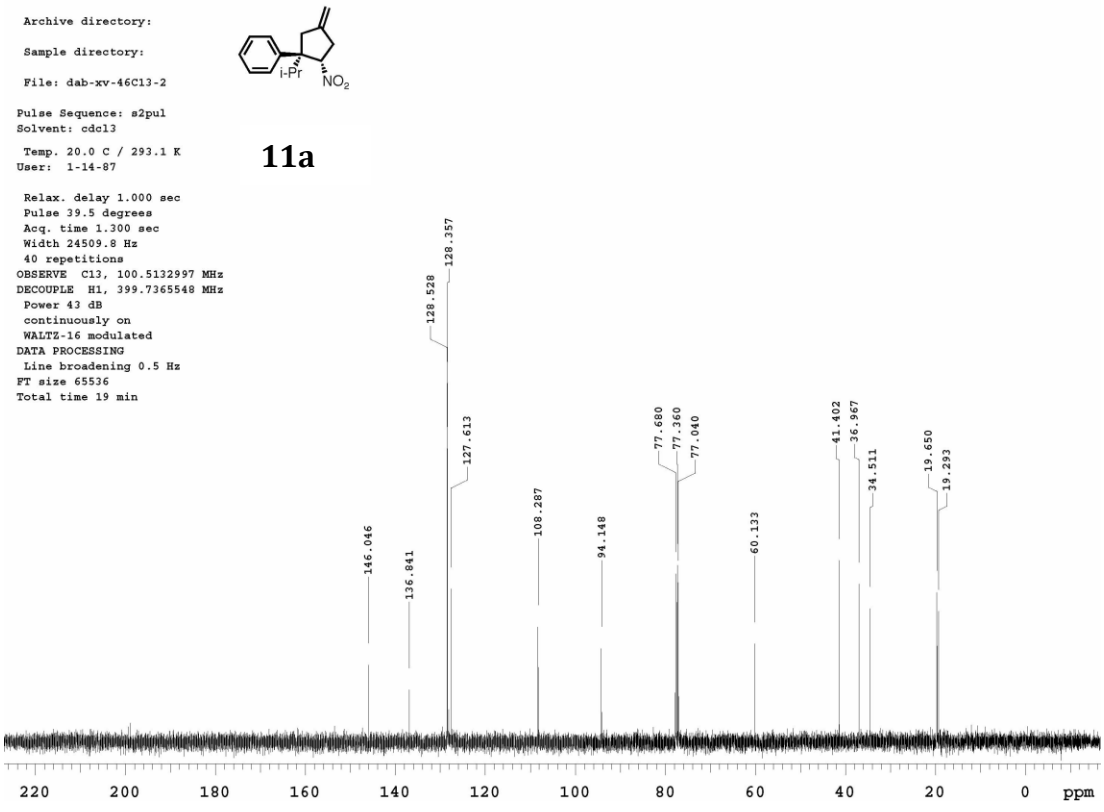
Pulse Sequence: s2pul
Solvent: cdcl3

Temp. 22.0 C / 295.1 K
User: 1-14-87

Relax. delay 1.000 sec
Pulse 39.5 degrees
Acq. time 1.300 sec
Width 24509.8 Hz
164 repetitions
OBSERVE C13, 100.5132982 MHz
DECOUPLE H1, 399.7365548 MHz
Power 43 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 0.5 Hz
FT size 65536
Total time 19 min




Std proton
Archive directory:
Sample directory:
File: dab-xv-46Proton
Pulse Sequence: s2pul
Solvent: cdcl3
Temp. 20.0 C / 293.1 K
Relax. delay 0.500 sec
Pulse 45.0 degrees
Acq. time 4.000 sec
Width 5605.4 Hz
16 repetitions
OBSERVE H1, 399.7345518 MHz
DATA PROCESSING
FT size 65536
Total time 1 min
i-Pr
NO2
11a
7
6
5
4
3
2
1
0
ppm
5.14
0.97
0.98
0.97
1.00
1.00
0.99
1.00
0.97
2.97
3.00


Std proton
Archive directory:
Sample directory:
File: dab-xv-46C13-2
Pulse Sequence: s2pul
Solvent: cdcl3
Temp. 20.0 C / 293.1 K
User: 1-14-87
Relax. delay 1.000 sec
Pulse 39.5 degrees
Acq. time 1.300 sec
Width 24509.8 Hz
40 repetitions
OBSERVE C13, 100.5132997 MHz
DECOUPLE H1, 399.7365548 MHz
Power 43 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 0.5 Hz
FT size 65536
Total time 19 min
i-Pr
NO2
11a
146.046
136.041
128.528
128.357
127.613
108.287
94.148
77.680
77.360
77.040
60.133
41.402
36.967
34.511
19.650
19.293
220
200
180
160
140
120
100
80
60
40
20
0
ppm

STANDARD 1H OBSERVE

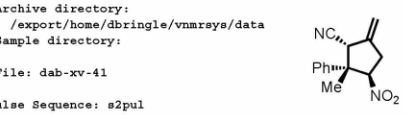
Archive directory:
/export/home/dbringle/vnmrsys/data
Sample directory:

File: dab-xv-41

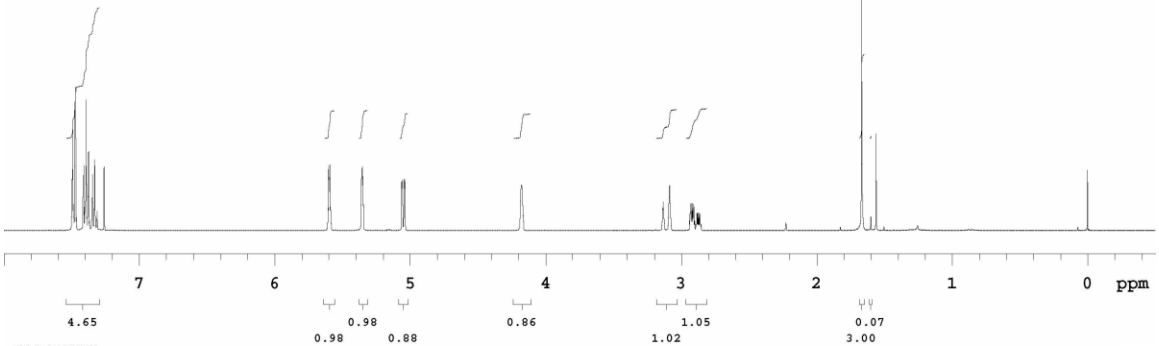
Pulse Sequence: s2pul
Solvent: CDCl3

Temp. 23.0 C / 296.1 K

Relax. delay 0.500 sec
Pulse 52.1 degrees
Acq. time 4.002 sec
Width 4997.5 Hz
16 repetitions
OBSERVE H1, 400.1115371 MHz
DATA PROCESSING
FT size 65536
Total time 1 min



12

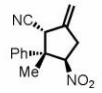


13C OBSERVE

Archive directory:
/export/home/dbringle/vnmrsys/data
Sample directory:

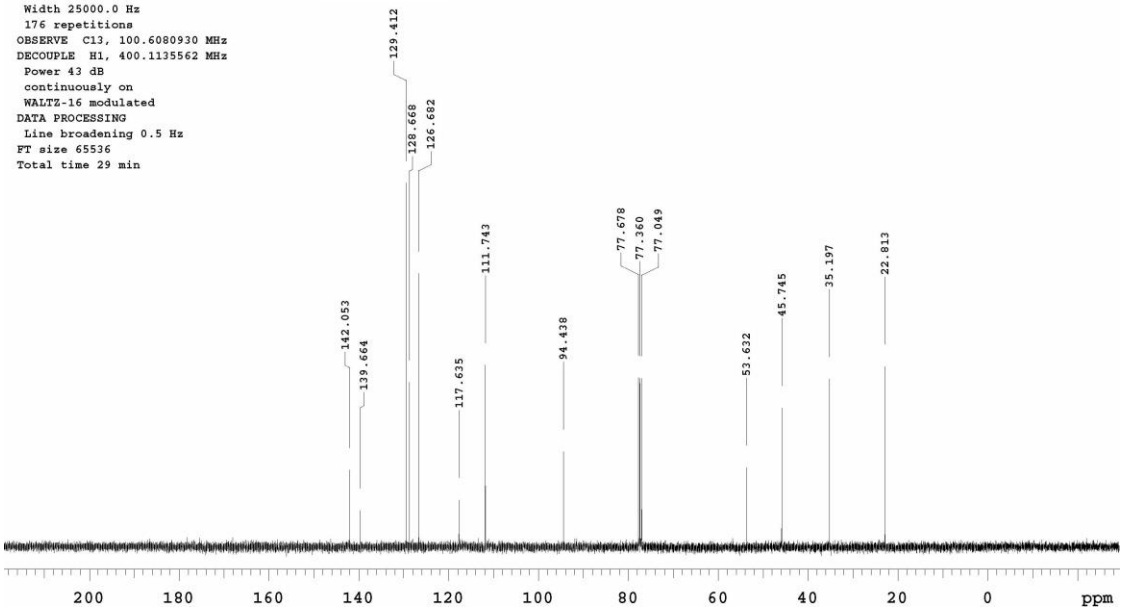
File: dab-xv-41AC13

Pulse Sequence: s2pul
Solvent: CDCl3



12

Relax. delay 0.500 sec
Pulse 38.1 degrees
Acq. time 1.199 sec
Width 25000.0 Hz
176 repetitions
OBSERVE C13, 100.6080930 MHz
DECOUPLE H1, 400.1135562 MHz
Power 43 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 0.5 Hz
FT size 65536
Total time 29 min



STANDARD 1H OBSERVE

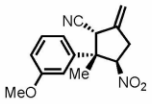
Archive directory:
/export/home/dbringle/vnmrsys/data
Sample directory:

File: dab-xv-32

Pulse Sequence: s2pul
Solvent: CDCl3

Temp. 23.0 C / 296.1 K

Relax. delay 0.500 sec
Pulse 52.1 degrees
Acq. time 4.002 sec
Width 4997.5 Hz
16 repetitions
OBSERVE H1, 400.1115367 MHz
DATA PROCESSING
FT size 65536
Total time 1 min



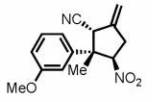
13

13C OBSERVE

Archive directory:
/export/home/dbringle/vnmrsys/data
Sample directory:

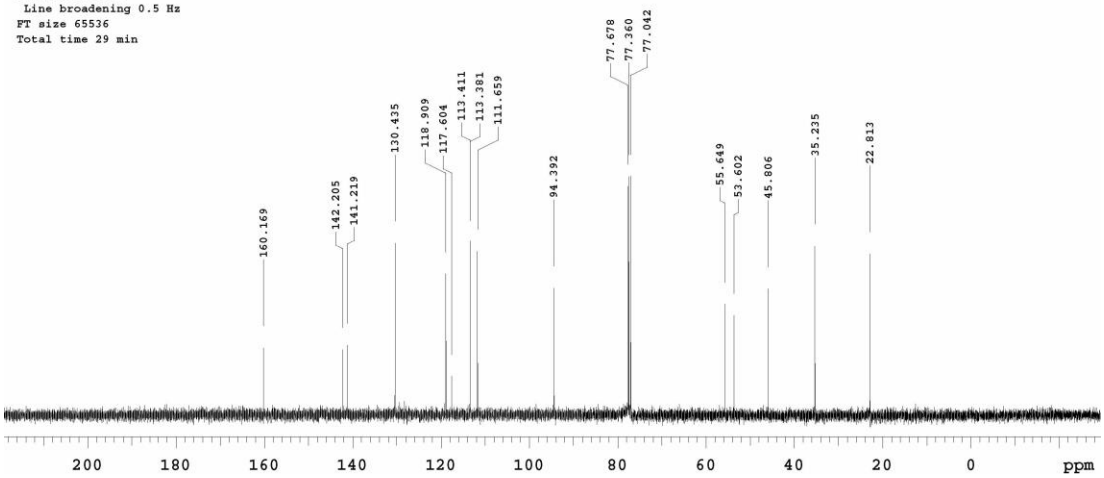
File: dab-xv-32BC13

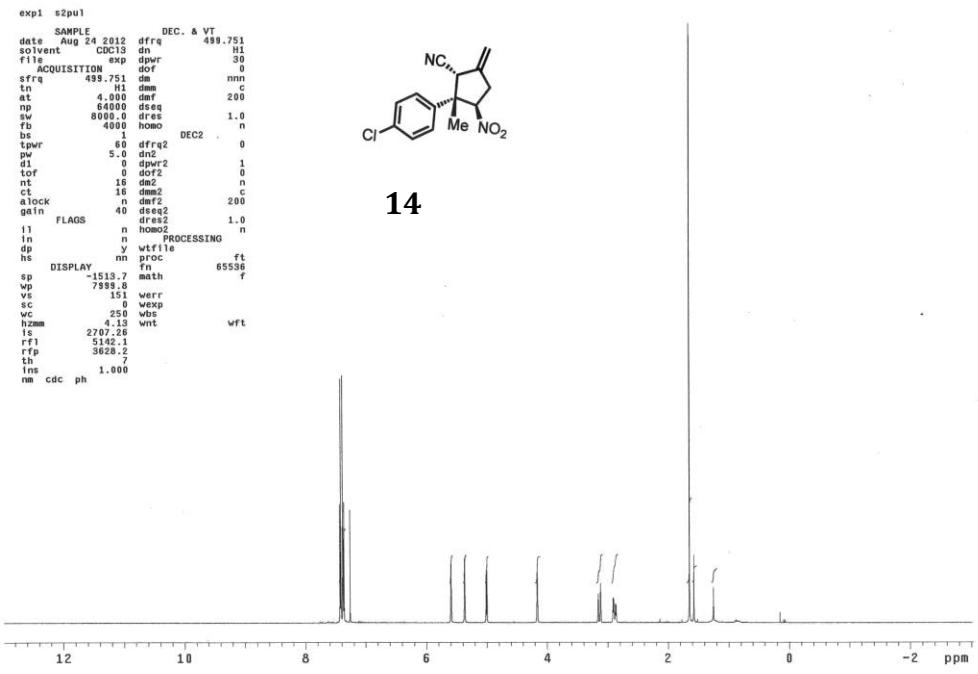
Pulse Sequence: s2pul
Solvent: CDCl3

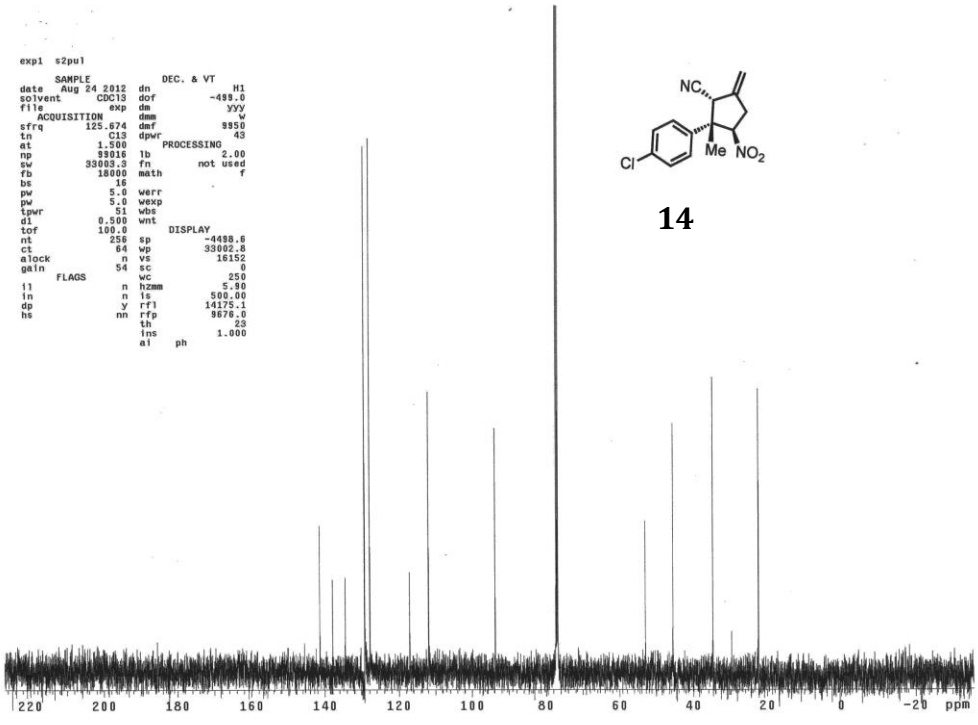


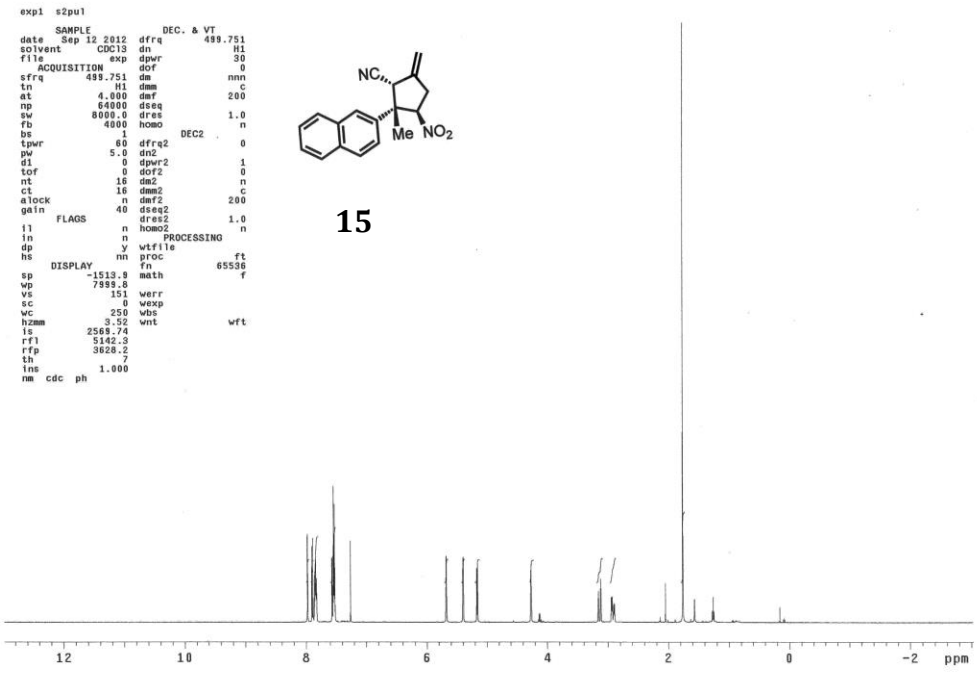
13

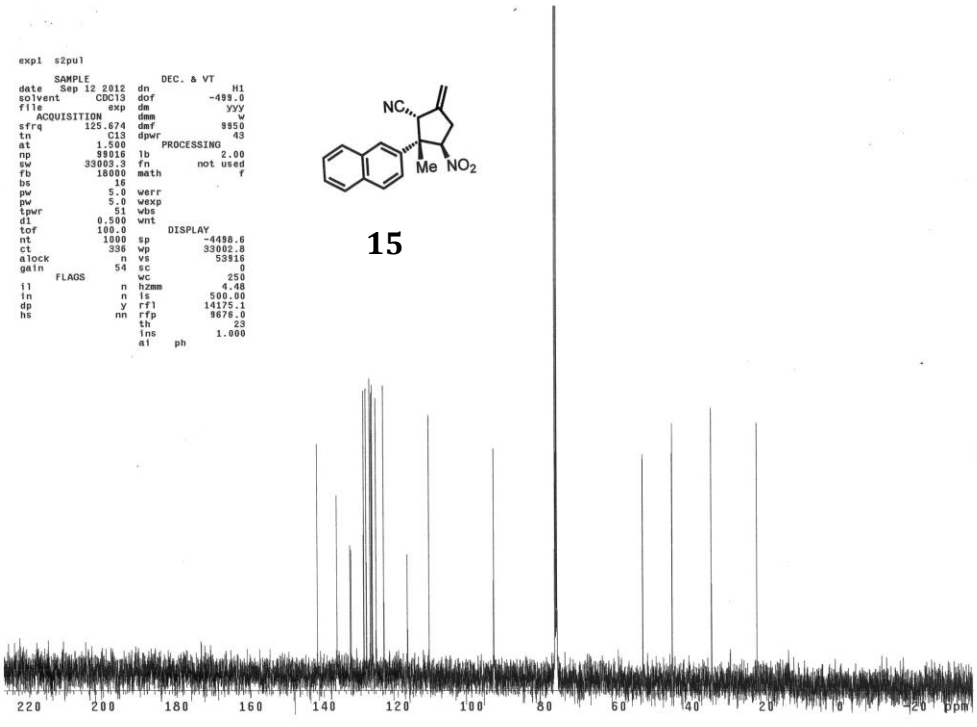
Relax. delay 0.500 sec
Pulse 38.1 degrees
Acq. time 1.199 sec
Width 25000.0 Hz
192 repetitions
OBSERVE C13, 100.6080930 MHz
DECOUPLE H1, 400.1135562 MHz
Power 43 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 0.5 Hz
FT size 65536
Total time 29 min

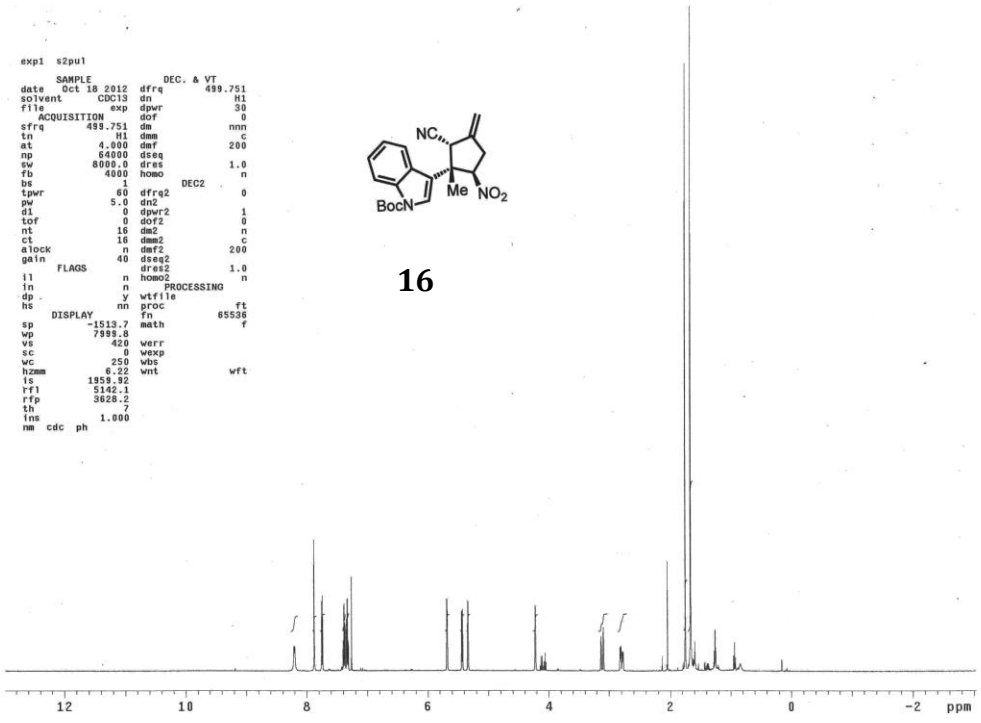


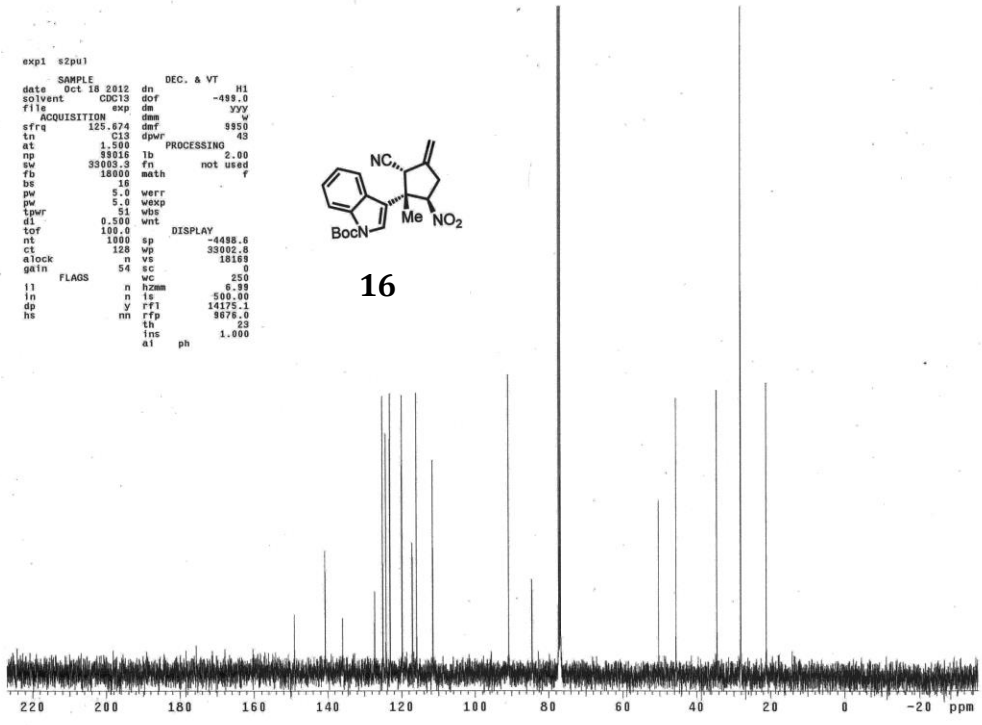

exp1 s2pul
SAMPLE
date Aug 24 2012
solvent CDCl3
file exp
ACQUISITION
sfrq 499.751
tn H1
at 4.000
np 64000
sw 8000.0
fb 4000
bs 1
tpwr 60
pw 5.0
d1 0
tof 0
nt 16
ct 16
alock n
gain 40
FLAGS
il n
in n
dp y
hs nn
DISPLAY
sp -1513.7
wp 7999.8
vs 151
sc 0
wc 250
hzmm 4.13
is 2707.26
rfl 5142.1
rfp 3628.2
th 7
ins 1.000
nm cdc ph
DEC. & VT
dfrq 499.751
dn H1
dpwr 30
dof 0
dm nnn
dmm c
dmf 200
dseq
dres 1.0
homo n
DEC2
dfrq2 0
dn2
dpwr2 1
dof2 0
dm2 n
dmm2 c
dmf2 200
dseq2
dres2 1.0
homo2 n
PROCESSING
wtfile
proc ft
fn 65536
math f
werr
wexp
wbs
wnt wft
NC
Me
NO2
Cl
14
12
10
8
6
4
2
0
-2
ppm

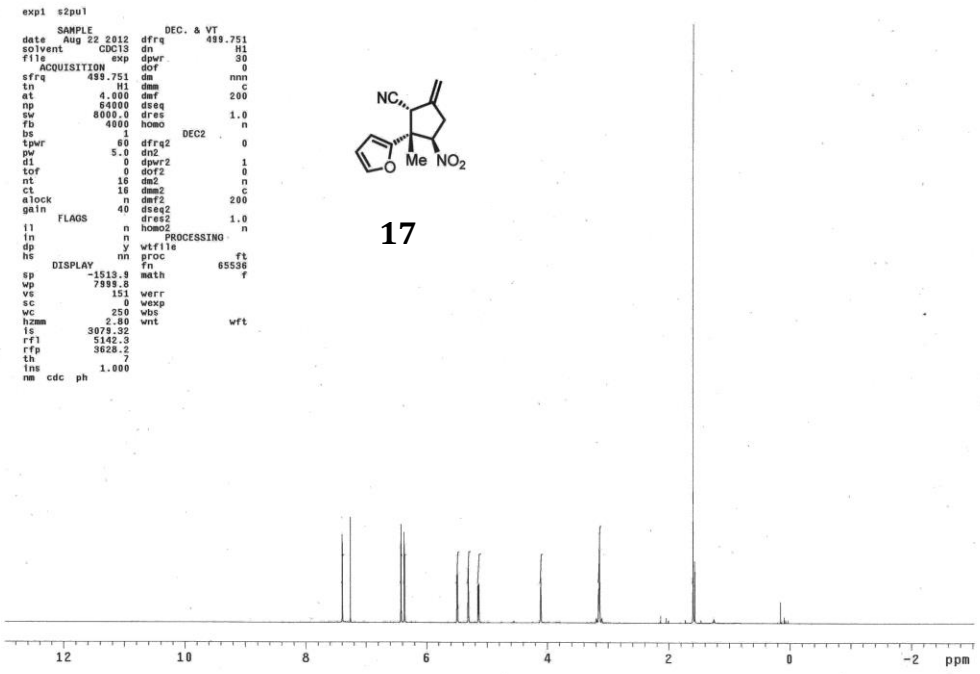

exp1 s2pul
SAMPLE
date Aug 24 2012
solvent CDCl3
file exp
ACQUISITION
sfrq 125.674
tn C13
at 1.500
np 99016
sw 33003.3
fb 18000
bs 16
pw 5.0
pw 5.0
tpwr 51
d1 0.500
tof 100.0
nt 256
ct 64
alock n
gain 54
FLAGS
il n
in n
dp y
hs nn
DEC. & VT
dn H1
dof -499.6
dm yyy
dmm w
dmf 9950
dpwr 43
PROCESSING
lb 2.00
fn not used
math f
werr
wexp
wbs
wnt
DISPLAY
sp -4498.6
wp 33002.8
vs 16152
sc 0
wc 250
hzmm 5.90
is 500.00
rfl 14175.1
rfp 9676.0
th 23
ins 1.000
ai ph
NC
Me
NO2
Cl
14
220
200
180
160
140
120
100
80
60
40
20
0
-20
ppm

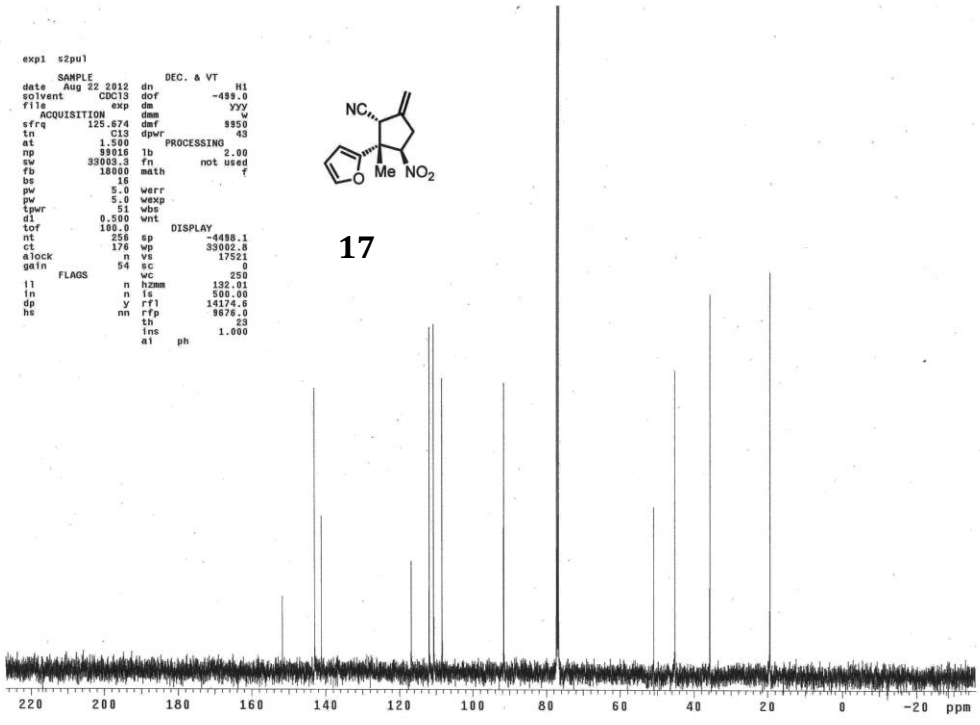

exp1 s2pul
SAMPLE
date Sep 12 2012
solvent CDCl3
file exp
ACQUISITION
sfrq 499.751
tn H1
at 4.000
np 64000
sw 8000.0
fb 4000
bs 1
tpwr 60
pw 5.0
d1 0
tof 0
nt 16
ct 16
alock n
gain 40
FLAGS
il n
in n
dp y
hs nn
DISPLAY
sp -1513.9
wp 7999.8
vs 151
sc 0
wc 250
hzmm 3.52
is 2569.74
rfl 5142.3
rfp 3628.2
th 7
ins 1.000
nm cdc ph
DEC. & VT
dfrq 499.751
dn H1
dpwr 30
dof 0
dm nnn
dmm c
dmf 200
dseq
dres 1.0
homo n
DEC2
dfrq2 0
dn2
dpwr2 1
dof2 0
dm2 n
dmm2 c
dmf2 200
dseq2
dres2 1.0
homo2 n
PROCESSING
wtfile
proc ft
fn 65536
math f
werr
wexp
wbs
wnt wft
NC
Me
NO2
15
12
10
8
6
4
2
0
-2
ppm

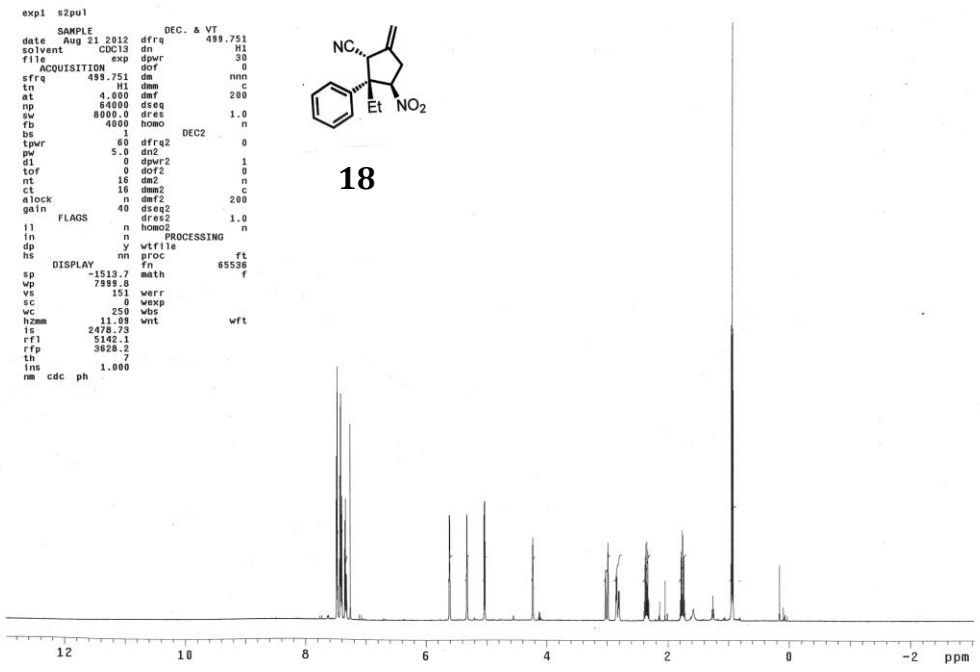

exp1 s2pul
SAMPLE
date Sep 12 2012
solvent CDCl3
file exp
ACQUISITION
sfrq 125.674
tn C13
at 1.500
np 98016
sw 33003.3
fb 18000
bs 16
pw 5.0
pw 5.0
tpwr 51
d1 0.500
tof 100.0
nt 1000
ct 336
alock n
gain 54
FLAGS
il n
in n
dp y
hs nn
DEC. & VT
dn H1
dof -499.0
dm yyy
dmm w
dmf 9850
dpwr 43
PROCESSING
lb 2.00
fn not used
math f
werr
wexp
wbs
wnt
DISPLAY
sp -4498.6
wp 33002.8
vs 53816
sc 0
wc 250
hzmm 4.48
is 500.00
rfl 14175.1
rfp 9676.0
th 23
ins 1.000
ai ph
NC
Me
NO2
15
220
200
180
160
140
120
100
80
60
40
20
0
-20
ppm

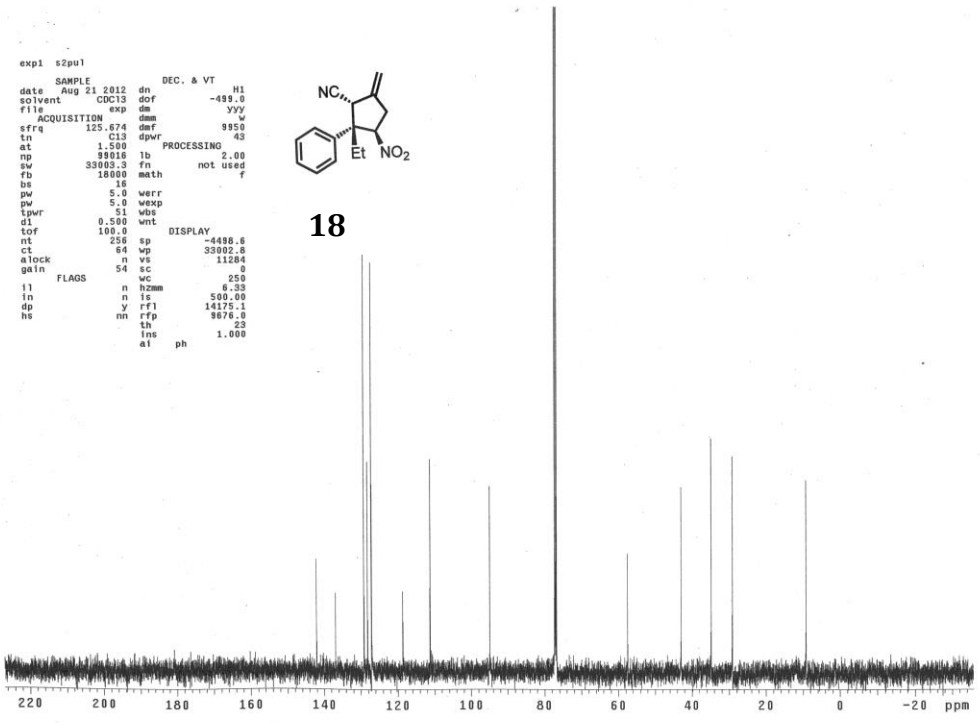

exp1 s2pul
SAMPLE
date Oct 18 2012
solvent CDCl3
file exp
ACQUISITION
sfrq 499.751
tn H1
at 4.000
np 64000
sw 8000.0
fb 4000
bs 1
tpwr 60
pw 5.0
d1 0
tof 0
nt 16
ct 16
alock n
gain 40
FLAGS
il n
in n
dp y
hs nn
DISPLAY
sp -1513.7
wp 7999.8
vs 420
sc 0
wc 250
hzmm 6.22
is 1959.92
rfl 5142.1
rfp 3628.2
th 7
ins 1.000
nm cdc ph
DEC. & VT
dfrq 499.751
dn H1
dpwr 30
dof 0
dm nnn
dmm c
dmf 200
dseq
dres 1.0
homo n
DEC2
dfrq2 0
dn2
dpwr2 1
dof2 0
dm2 n
dmm2 c
dmf2 200
dseq2
dres2 1.0
homo2 n
PROCESSING
wtfile
proc ft
fn 65536
math f
werr
wexp
wbs
wnt wft
NC
Me
NO2
BocN
16
12
10
8
6
4
2
0
-2
ppm

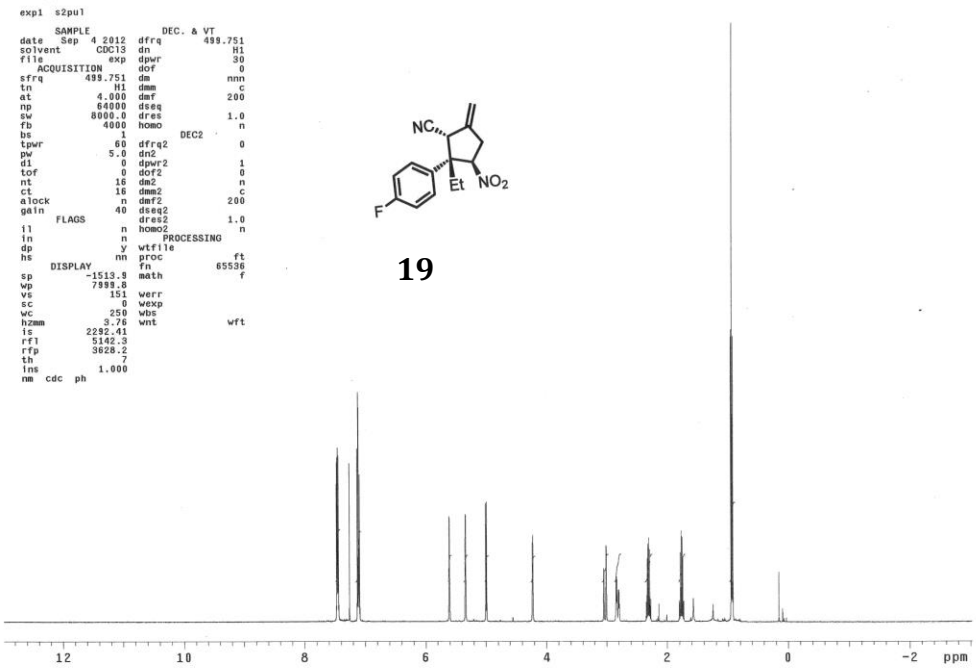

exp1 s2pul
SAMPLE
date Oct 18 2012
solvent CDCl3
file exp
ACQUISITION
sfrq 125.674
tn C13
at 1.500
np 99016
sw 33003.3
fb 18000
bs 16
pw 5.0
pw 5.0
tpwr 51
d1 0.500
tof 100.0
nt 1000
ct 128
alock n
gain 54
FLAGS
il n
in n
dp y
hs nn
DEC. & VT
dn H1
dof -499.0
dm yyy
dmm w
dmf 9950
dpwr 43
PROCESSING
lb 2.00
fn not used
math f
werr
wexp
wbs
wnt
DISPLAY
sp -4498.6
wp 33002.8
vs 18168
sc 0
wc 250
hzmm 6.99
is 500.00
rfl 14175.1
rfp 9676.0
th 23
ins 1.000
ai ph
NC
Me
NO2
BocN
16
220
200
180
160
140
120
100
80
60
40
20
0
-20
ppm

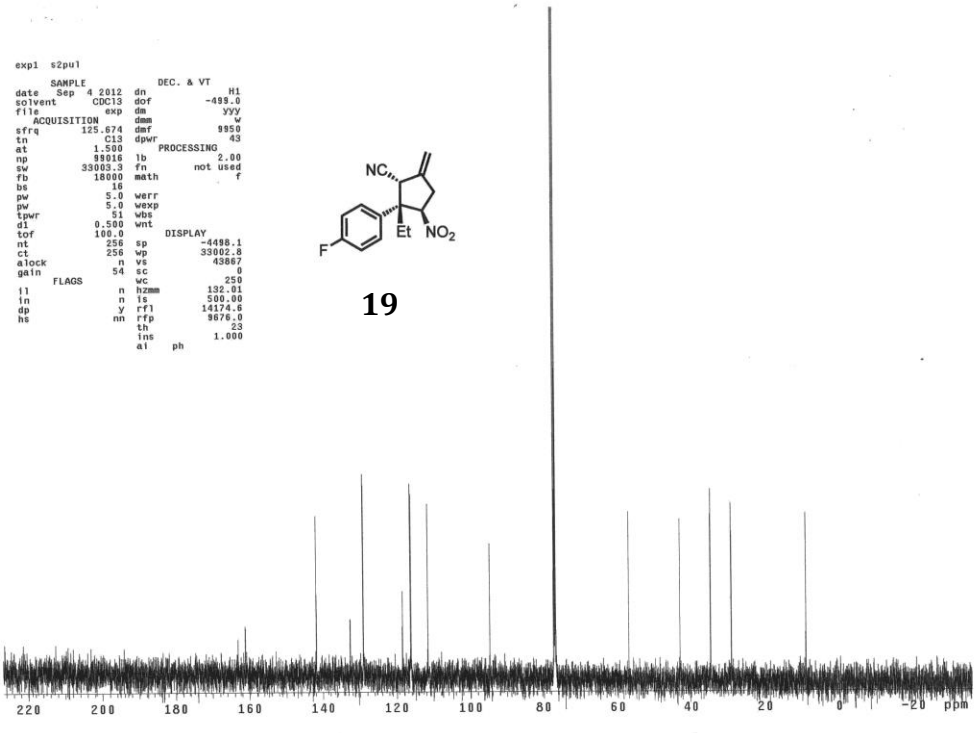

exp1 s2pul
SAMPLE
date Aug 22 2012
solvent CDCl3
file exp
ACQUISITION
sfrq 499.751
tn H1
at 4.000
np 64000
sw 8000.0
fb 4000
bs 1
tpwr 60
pw 5.0
d1 0
tof 0
nt 16
ct 16
alock n
gain 40
FLAGS
il n
in n
dp y
hs nn
DISPLAY
sp -1513.9
wp 7999.8
vs 151
sc 0
wc 250
hzmm 2.80
is 3079.32
rfl 5142.3
rfp 3628.2
th 7
ins 1.000
nm cdc ph
DEC. & VT
dfrq 499.751
dn H1
dpwr 30
dof 0
dm nnn
dmm c
dmf 200
dseq
dres 1.0
homo n
DEC2
dfrq2 0
dn2
dpwr2 1
dof2 0
dm2 n
dmm2 c
dmf2 200
dseq2
dres2 1.0
homo2 n
PROCESSING
wtfile
proc ft
fn 65536
math f
werr
wexp
wbs
wnt wft
NC
Me
NO2
O
17
12
10
8
6
4
2
0
-2
ppm

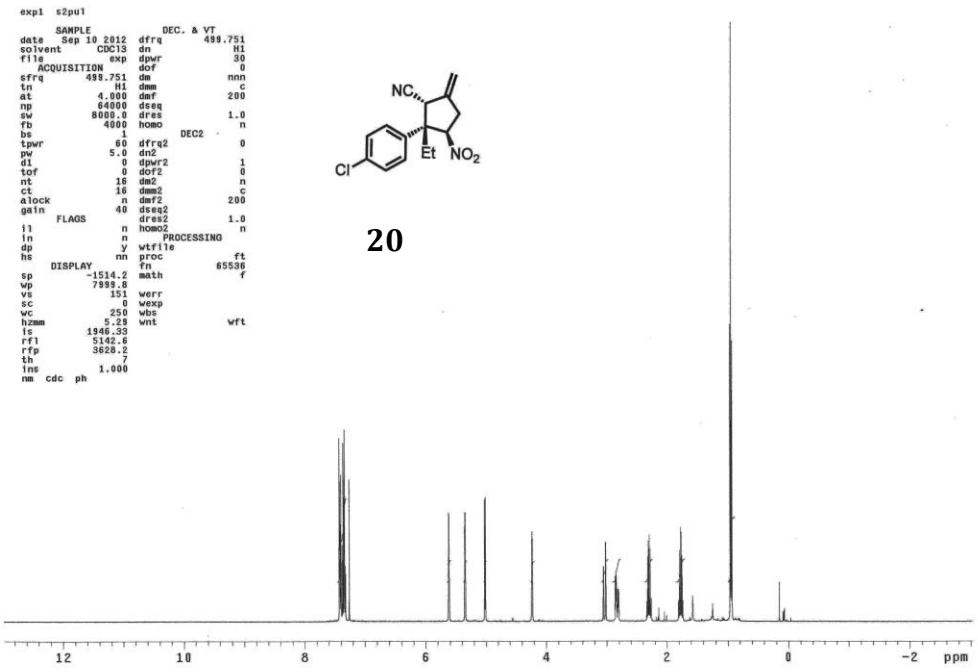

exp1 s2pul
SAMPLE
date Aug 22 2012
solvent CDCl3
file exp
ACQUISITION
sfrq 125.674
tn C13
at 1.500
np 99016
sw 33003.3
fb 18000
bs 16
pw 5.0
pw 5.0
tpwr 51
d1 0.500
tof 100.0
nt 256
ct 176
alock n
gain 54
FLAGS
il n
in n
dp y
hs nn
DEC. & VT
dn H1
dof -499.0
dm yyy
dmm w
dmf 9950
dpwr 43
PROCESSING
lb 2.00
fn not used
math f
werr
wexp
wbs
wnt
DISPLAY
sp -4498.1
wp 33002.8
vs 17521
sc 0
wc 250
hzmm 132.01
is 500.00
rfl 14174.6
rfp 9676.0
th 23
ins 1.000
ai ph
NC
Me
NO2
O
17
220
200
180
160
140
120
100
80
60
40
20
0
-20
ppm

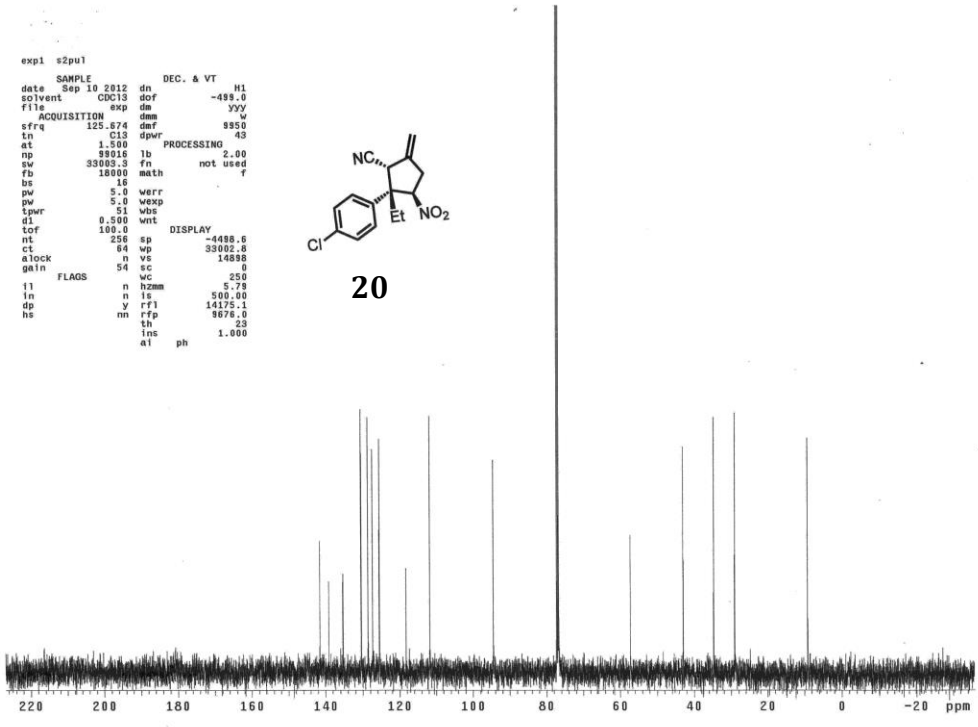

exp1 s2pul
SAMPLE
date Aug 21 2012
solvent CDCl3
file exp
ACQUISITION
sfrq 499.751
tn H1
at 4.000
np 64000
sw 8000.0
fb 4000
bs 1
tpwr 60
pw 5.0
d1 0
tof 0
nt 16
ct 16
alock n
gain 40
FLAGS
il n
in n
dp y
hs nn
DISPLAY
sp -1513.7
wp 7999.8
vs 151
sc 0
wc 250
hzmm 11.08
is 2478.73
rfl 5142.1
rfp 3628.2
th 7
ins 1.000
nm cdc ph
DEC. & VT
dfrq 499.751
dn H1
dpwr 30
dof 0
dm nnn
dmm c
dmf 200
dseq
dres 1.0
homo n
DEC2
dfrq2 0
dn2
dpwr2 1
dof2 0
dm2 n
dmm2 c
dmf2 200
dseq2
dres2 1.0
homo2 n
PROCESSING
wtfile
proc ft
fn 65536
math f
werr
wexp
wbs
wnt wft
NC
Et
NO2
18
12
10
8
6
4
2
0
-2
ppm


exp1 s2pul
SAMPLE
date Aug 21 2012
solvent CDCl3
file exp
ACQUISITION
sfrq 125.674
tn C13
at 1.500
np 98016
sw 33003.3
fb 18000
bs 16
pw 5.0
pw 5.0
tpwr 51
d1 0.500
tof 100.0
nt 256
ct 64
alock n
gain 54
FLAGS
il n
in n
dp y
hs nn
DEC. & VT
dn H1
dof -499.0
dm yyy
dmm w
dmf 9950
dpwr 43
PROCESSING
lb 2.00
fn not used
math f
werr
wexp
wbs
wnt
DISPLAY
sp -4498.6
wp 33002.8
vs 11284
sc 0
wc 250
hzmm 6.33
is 500.00
rfl 14175.1
rfp 9676.0
th 23
ins 1.000
ai ph
NC
Et
NO2
18
220
200
180
160
140
120
100
80
60
40
20
0
-20
ppm


exp1 s2pul
SAMPLE
date Sep 4 2012
solvent CDCl3
file exp
ACQUISITION
sfrq 499.751
tn H1
at 4.000
np 64000
sw 8000.0
fb 4000
bs 1
tpwr 60
pw 5.0
dl 0
tof 0
nt 16
ct 16
alock n
gain 40
FLAGS
il n
in n
dp y
hs nn
DISPLAY
sp -1513.9
wp 7999.8
vs 151
sc 0
wc 250
hzmm 3.76
is 2282.41
rfl 5142.3
rfp 3628.2
th 7
ins 1.000
nm cdc ph
DEC. & VT
dfrq 499.751
dn H1
dpwr 30
dof 0
dm nnn
dmm c
dmf 200
dseq
dres 1.0
homo n
DEC2
dfrq2 0
dn2
dpwr2 1
dof2 0
dm2 n
dmm2 c
dmf2 200
dseq2
dres2 1.0
homo2 n
PROCESSING
wtfile
proc ft
fn 65536
math f
werr
wexp
wbs
wnt wft
NC
Et
NO2
F
19
12
10
8
6
4
2
0
-2
ppm


exp1 s2pul
SAMPLE
date Sep 4 2012
solvent CDCl3
file exp
ACQUISITION
sfrq 125.674
tn C13
at 1.500
np 98016
sw 33003.3
fb 18000
bs 16
pw 5.0
pw 5.0
tpwr 51
dl 0.500
tof 100.0
nt 256
ct 256
alock n
gain 54
FLAGS
il n
in n
dp y
hs nn
dn H1
dof -499.0
dm yyy
dmm w
dmf 9950
dpwr 43
PROCESSING
lb 2.00
fn not used
math f
werr
wexp
wbs
wnt
DISPLAY
sp -4498.1
wp 33002.8
vs 43867
sc 0
wc 250
hzmm 132.01
is 500.00
rfl 14174.6
rfp 9676.0
th 23
ins 1.000
al ph
NC
Et
NO2
F
19
220
200
180
160
140
120
100
80
60
40
20
0
-20
ppm

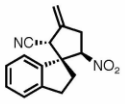

exp1 s2pul
SAMPLE
date Sep 10 2012
solvent CDCl3
file exp
ACQUISITION
sfrq 499.751
tn H1
at 4.000
np 64000
sw 8000.0
fb 4000
bs 1
tpwr 60
pw 5.0
d1 0
tof 0
nt 16
ct 16
alock n
gain 40
FLAGS
il n
in n
dp y
hs nn
DISPLAY
sp -1514.2
wp 7999.8
vs 151
sc 0
wc 250
hzmm 5.28
is 1946.33
rf1 5142.6
rfp 3628.2
th 7
ins 1.000
nm cdc ph
DEC. & VT
dfrq 499.751
dn H1
dpwr 30
dof 0
dm nnn
dmm c
dmf 200
dseq
dres 1.0
homo n
DEC2
dfrq2 0
dn2
dpwr2 1
dof2 0
dm2 n
dmm2 c
dmf2 200
dseq2
dres2 1.0
homo2 n
PROCESSING
wtfile
proc ft
fn 65536
math f
werr
wexp
wbs
wnt wft
NC
Et
NO2
Cl
20
12
10
8
6
4
2
0
-2
ppm


exp1 s2pul
SAMPLE
date Sep 10 2012
solvent CDCl3
file exp
ACQUISITION
sfrq 125.674
tn C13
at 1.500
np 99016
sw 33003.3
fb 18000
bs 16
pw 5.0
pw 5.0
tpwr 51
d1 0.500
tof 100.0
nt 256
ct 64
alock n
gain 54
FLAGS
il n
in n
dp y
hs nn
DEC. & VT
dn H1
dof -499.0
dm yyy
dmm w
dmf 9950
dpwr 43
PROCESSING
lb 2.00
fn not used
math f
werr
wexp
wbs
wnt
DISPLAY
sp -4488.6
wp 33002.8
vs 14898
sc 0
wc 250
hzmm 5.78
is 500.00
rf1 14175.1
rfp 9676.0
th 23
ins 1.000
ai ph
NC
Et
NO2
Cl
20
220
200
180
160
140
120
100
80
60
40
20
0
-20
ppm

STANDARD 1H OBSERVE

Archive directory:
/export/home/dbringle/vnmrsys/data
Sample directory:

File: dab-xv-31

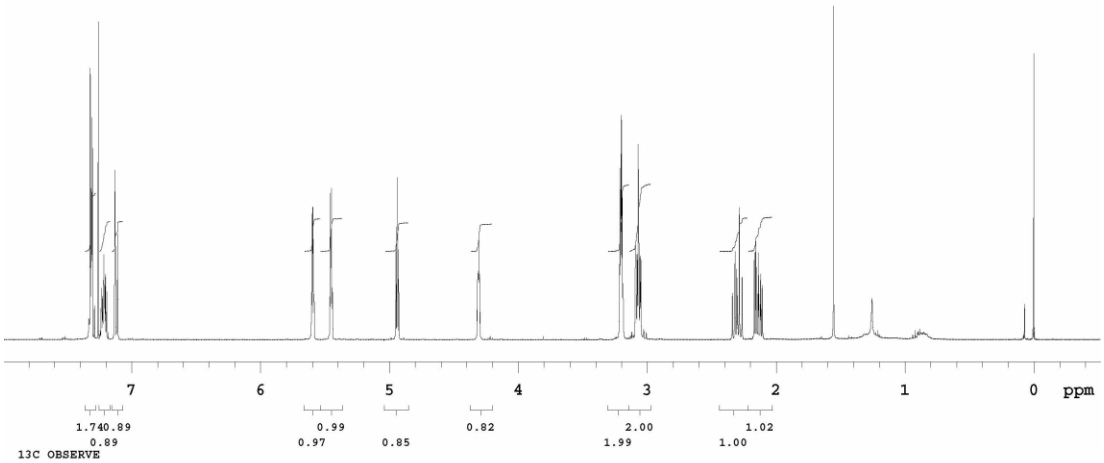


21

Pulse Sequence: s2pul
Solvent: CDCl3

Temp. 23.0 C / 296.1 K

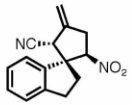
Relax. delay 0.500 sec
Pulse 52.1 degrees
Acq. time 4.002 sec
Width 4997.5 Hz
16 repetitions
OBSERVE H1, 400.1115373 MHz
DATA PROCESSING
FT size 65536
Total time 1 min



13C OBSERVE

Archive directory:
/export/home/dbringle/vnmrsys/data
Sample directory:

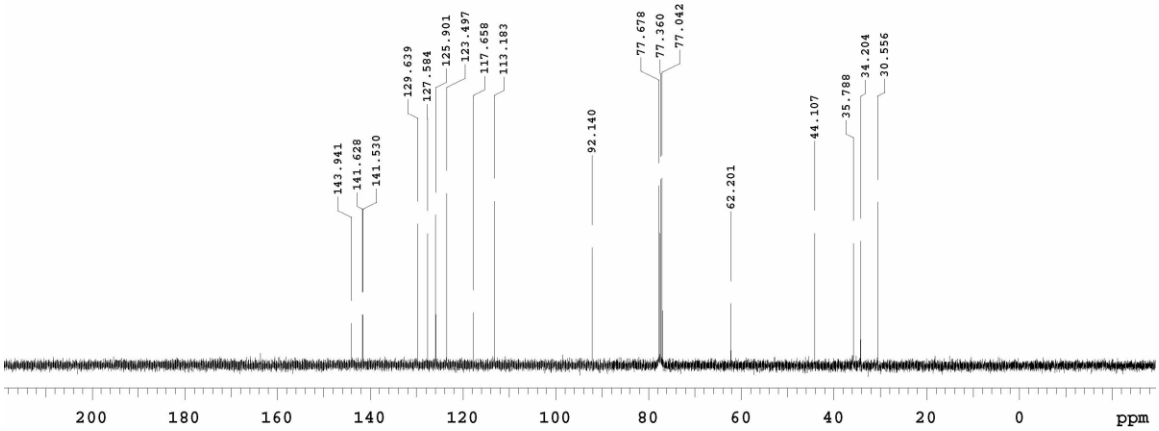
File: dab-xv-31BC13

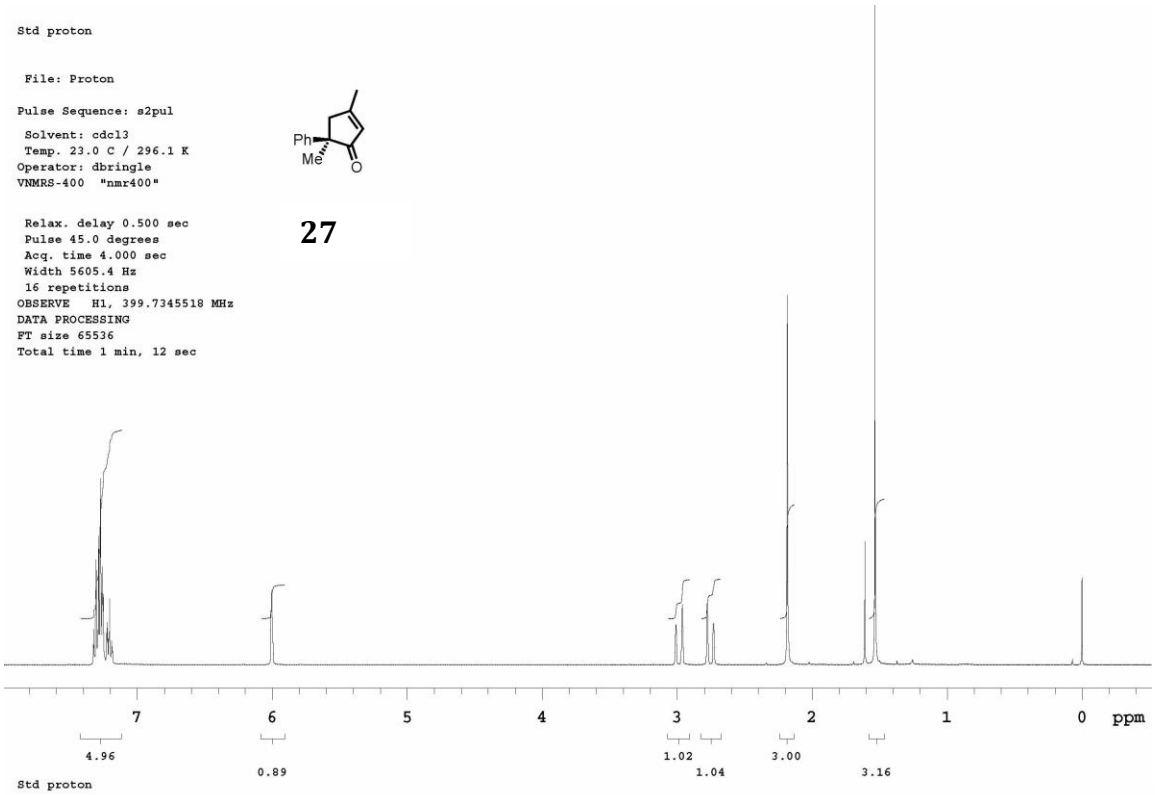


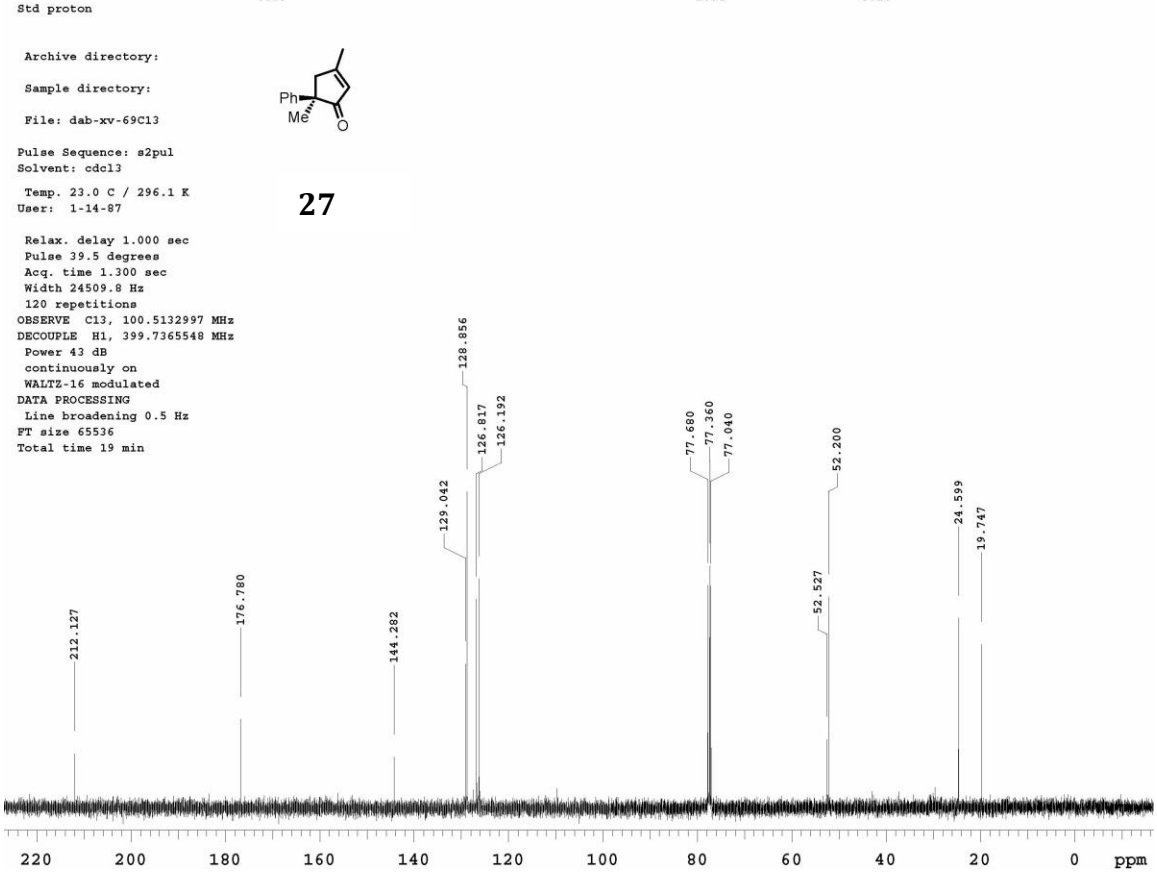
21

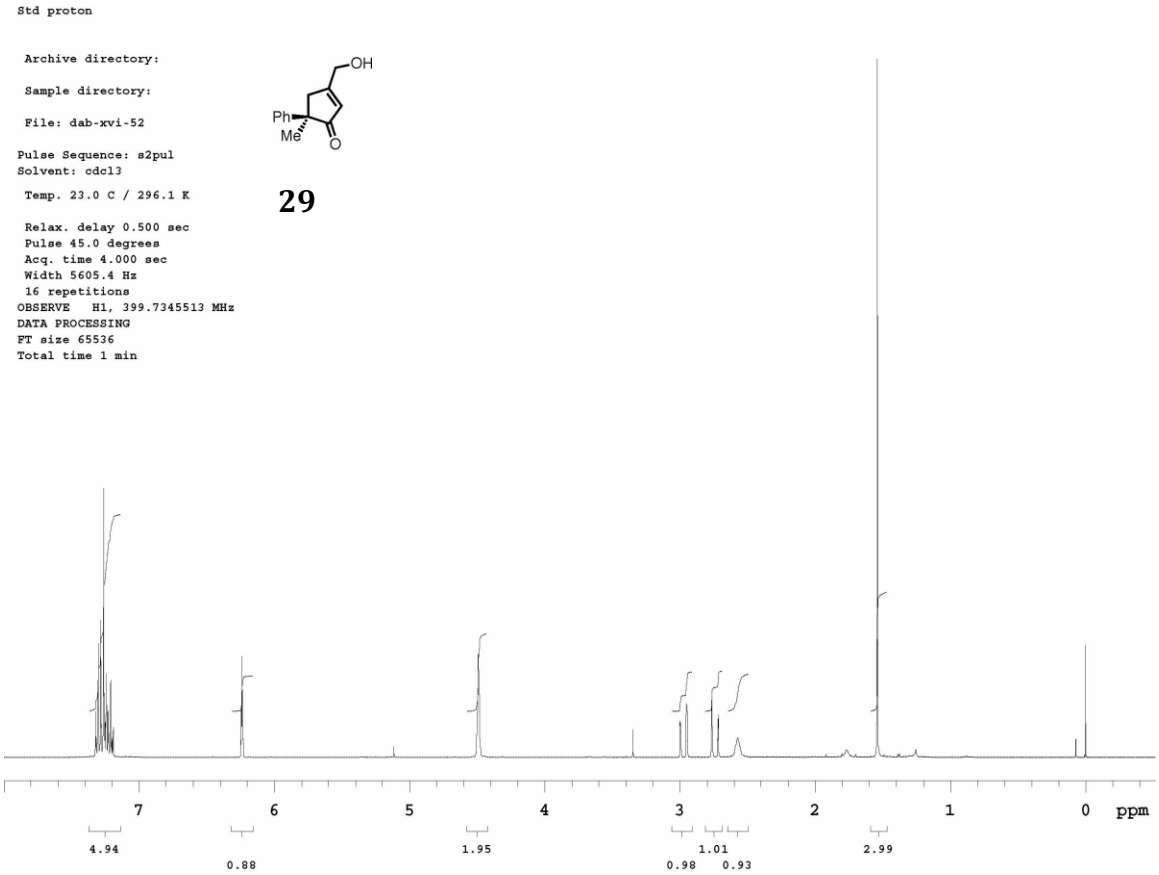
Pulse Sequence: s2pul
Solvent: CDCl3

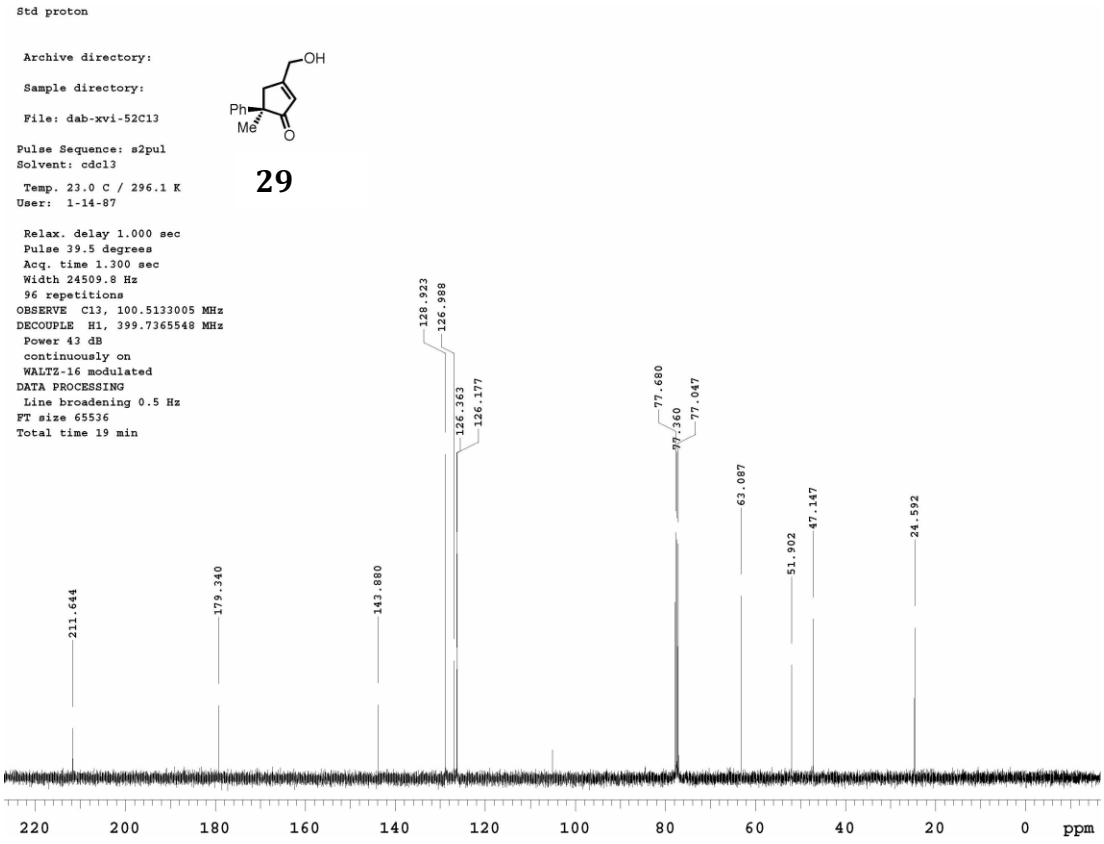
Relax. delay 0.500 sec
Pulse 38.1 degrees
Acq. time 1.199 sec
Width 25000.0 Hz
160 repetitions
OBSERVE C13, 100.6080930 MHz
DECOUPLE H1, 400.1135562 MHz
Power 43 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 0.5 Hz
FT size 65536
Total time 29 min




Std proton
File: Proton
Pulse Sequence: s2pul
Solvent: cdcl3
Temp. 23.0 C / 296.1 K
Operator: dbringle
VNMRS-400 "nmr400"
Relax. delay 0.500 sec
Pulse 45.0 degrees
Acq. time 4.000 sec
Width 5605.4 Hz
16 repetitions
OBSERVE H1, 399.7345518 MHz
DATA PROCESSING
FT size 65536
Total time 1 min, 12 sec
Ph
Me
O
27
7
6
5
4
3
2
1
0
ppm
4.96
0.89
1.02
1.04
3.00
3.16


Std proton
Archive directory:
Sample directory:
File: dab-xv-69C13
Pulse Sequence: s2pul
Solvent: cdcl3
Temp. 23.0 C / 296.1 K
User: 1-14-87
Ph
Me
O
27
Relax. delay 1.000 sec
Pulse 39.5 degrees
Acq. time 1.300 sec
Width 24509.8 Hz
120 repetitions
OBSERVE C13, 100.5132997 MHz
DECOUPLE H1, 399.7365548 MHz
Power 43 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 0.5 Hz
FT size 65536
Total time 19 min
212.127
176.780
144.282
129.042
128.856
126.817
126.192
77.680
77.360
77.040
52.527
52.200
24.599
19.747
220
200
180
160
140
120
100
80
60
40
20
0
ppm


Std proton
Archive directory:
Sample directory:
File: dab-xvi-52
Pulse Sequence: s2pul
Solvent: cdcl3
Temp. 23.0 C / 296.1 K
Relax. delay 0.500 sec
Pulse 45.0 degrees
Acq. time 4.000 sec
Width 5605.4 Hz
16 repetitions
OBSERVE H1, 399.7345513 MHz
DATA PROCESSING
FT size 65536
Total time 1 min
OH
Ph
Me
O
29
7
6
5
4
3
2
1
0
ppm
4.94
0.88
1.95
0.98
1.01
0.93
2.99


Std proton
Archive directory:
Sample directory:
File: dab-xvi-52C13
Pulse Sequence: s2pul
Solvent: cdcl3
Temp. 23.0 C / 296.1 K
User: 1-14-87
Relax. delay 1.000 sec
Pulse 39.5 degrees
Acq. time 1.300 sec
Width 24509.8 Hz
96 repetitions
OBSERVE C13, 100.5133005 MHz
DECOUPLE H1, 399.7365548 MHz
Power 43 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 0.5 Hz
FT size 65536
Total time 19 min
OH
Ph
Me
O
29
128.923
126.988
126.363
126.177
77.680
77.360
77.047
63.087
51.902
47.147
24.592
211.644
179.340
143.880
220
200
180
160
140
120
100
80
60
40
20
0
ppm

G. References

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