Supporting Information

Trisubstituted 2-Trifluoromethyl Pyrrolidines via Catalytic Asymmetric Michael Addition/Reductive Cyclization

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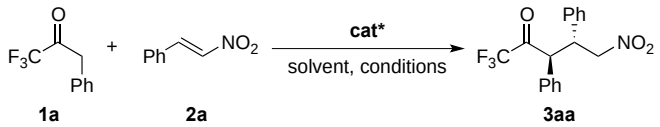
Table of Contents **Page**

General Information

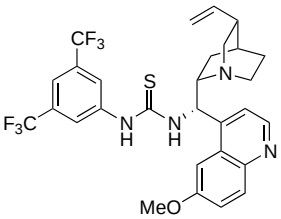
Methods: Infrared (IR) spectra were obtained using a Jasco 260 Plus Fourier transform infrared spectrometer. Proton and carbon magnetic resonance spectra (^{1}H NMR, ^{13}C NMR, and ^{19}F NMR) were recorded on a Bruker model DRX 400 or 600 (^{1}H NMR at 400 MHz or 600 MHz, ^{13}C NMR at 100 MHz or 150 MHz, and ^{19}F NMR at 376 or 564 MHz) spectrometer with solvent resonance as the internal standard (^{1}H NMR: $CDCl_3$ at 7.26 ppm and ^{13}C NMR: $CDCl_3$ at 77.0 ppm). ^{1}H NMR data are reported as follows: chemical shift, multiplicity (app = apparent, s = singlet, br s = broad singlet, d = doublet, dd = doublet of doublet, t = triplet, q = quartet, qt = quintet, sept = septuplet, oct = octuplet, m = multiplet), coupling constants (Hz), and integration. HPLC analysis was performed on an Agilent Technologies 1200 System equipped with Chiralpak IA, IB, and IC columns (φ 4.6 mm x 250 mm, constant flow at 1.00 mL/min). Supercritical fluid chromatography (SFC) was performed on a Berger SFC system equipped with Chiralpak AD, AS, OD, and WO columns (φ 4.6 mm x 250 mm). Samples were eluted with SFC grade CO_2 at the indicated percentage of MeOH with an oven temperature of 40 °C. Optical rotations were measured using a 2 mL cell with a 1 dm path length on a Jasco DIP 1000 digital polarimeter. Mass spectra were obtained using a Thermo Scientific LTQ FT Ultra instrument with electrospray ionization; samples were prepared in MeOH. Analytical thin layer chromatography (TLC) was performed on Sorbtech 0.25 mm silica gel 60 plates. Visualization was accomplished with UV light and/or aqueous ceric ammonium molybdate solution followed by heating. Purification of the reaction products was carried out by using Siliaflash-P60 silica gel (40-63μm) purchased from Silicycle. All reactions were carried out with magnetic stirring. Yield refers to isolated yield of analytically pure material unless otherwise noted. Yields and diastereomeric ratios (dr) are reported for a specific experiment and as a result may differ slightly from those found in the tables, which are averages of at least two experiments.

Materials: 1,1,1-Trifluoromethylketones **1**,[1] nitroolefins **2**,[2] and catalyst **QD-TU**[3] were prepared according to known literature procedures. Triethylamine (Et_3N) was freshly distilled from calcium hydride prior to use. Dichloromethane (CH_2Cl_2), diethyl ether (Et_2O), tetrahydrofuran (THF), and toluene were dried by passage through a column of neutral alumina under nitrogen prior to use. All other reagents were purchased from commercial sources and were used as received unless otherwise noted.

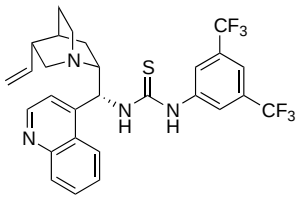
Detailed Optimization Studies[a]



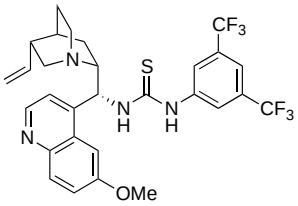
1a **2a** **3aa**



QN-TU

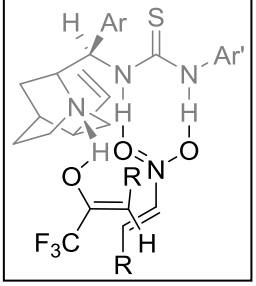


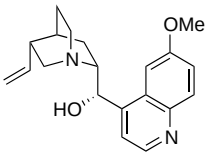
CN-TU



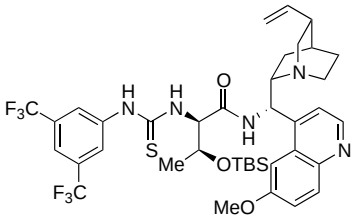
QD-TU

Plausible transition state:

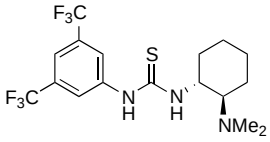




QD



Tri-TU

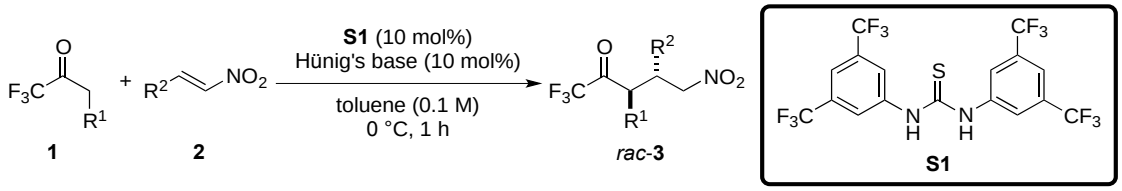


TK-TU

entry	cat* (%)	solvent ([M])	T (°C)	t (h)	conv. (%)[b]	dr[b]	er[c]
1	**QN-TU** (10)	toluene (0.2)	0	1	>95	>20:1	10:90
2	**QN-TU** (10)	CH_2Cl_2 (0.2)	0	1	>95	>20:1	15.5:84.5
3	**QN-TU** (10)	Et_2O (0.2)	0	1	>95	>20:1	14.5:85.5
4	**QN-TU** (10)	EtOAc (0.2)	0	1	>95	>20:1	15:85
5	**QN-TU** (10)	MeCN (0.2)	0	1	>95	10:1	24:76
6	**CN-TU** (10)	toluene (0.2)	0	1	>95	>20:1	90.5:9.5
7	**QD-TU** (10)	toluene (0.2)	0	1	>95	>20:1	92.5:7.5
8	**Tri-TU** (10)	toluene (0.2)	0	1	>95	>20:1	15:85
9	**TK-TU** (10)	toluene (0.2)	0	1	>95	>20:1	90:10
10	**QD** (10)	toluene (0.2)	0	1	>95	>20:1	77:23
11	**QD-TU** (10)	toluene (0.2)[d]	0	1	>95	>20:1	92:8
12[e]	**QD-TU** (10)	toluene (0.2)	0	1	>95	>20:1	93.5:6.5
13[e]	**QD-TU** (10)	toluene (0.2)	-30	4	94	>20:1	95:5
14[e]	**QD-TU** (10)	toluene (0.1)	0	1	>95	>20:1	94:6
15[e]	**QD-TU** (5)	toluene (0.1)	0	1	>95	>20:1	94:6
16[e]	**QD-TU** (2.5)	toluene (0.1)	0	1	>95	>20:1	94.5:5.5
17[e]	**QD-TU** (1)	toluene (0.1)	0	3	>95	>20:1	94.5:5.5
18[e]	**QD-TU** (0.5)	toluene (0.1)	0	24	65	>20:1	94.5:5.5
19[e,f]	**QD-TU** (2.5)	toluene (0.1)	0	1	>95	>20:1	95:5

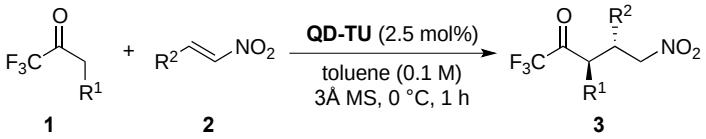
[a] Reactions were performed with **1a** (0.20 mmol) and **2a** (0.24 mmol). [b] The conversion and diastereomeric ratio were determined by ^{19}F NMR spectroscopic analysis of the crude product. [c] The enantiomeric ratio was determined by HPLC or SFC analysis on a chiral stationary phase. [d] Employing reagent grade toluene. [e] Reactions were performed with 3Å MS (50 mg). [f] Reaction was performed with **1a** (0.21 mmol) and **2a** (0.20 mmol).

General Procedure A for the Preparation of Racemic Michael Adducts

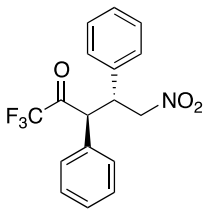


A 20-mL scintillation vial containing a magnetic stir bar was charged with 1,1,1-trifluoromethyl ketone **1** (0.21 mmol, 1.05 equiv), nitroolefin **2** (0.20 mmol, 1.00 equiv), and thiourea **S1** (5.0 mg, 0.01 mmol, 0.05 equiv) in toluene (2.0 mL, 0.1 M). The resulting solution was cooled to 0 °C in an ice bath. Hünig's base (2.0 µL, 0.01 mmol, 0.05 equiv) was added and the reaction was capped. The reaction was allowed to stir at 0 °C until adjudged complete by TLC, generally 1 h. The reaction was quenched by addition of 2 M HCl (2 mL). The biphasic solution was diluted with H_2O (5 mL) and EtOAc (10 mL). The layers were separated and the organic layer was washed with brine (5 mL), dried over Na_2SO_4, filtered, and concentrated *in vacuo*. The diastereomeric ratio was determined by ^{19}F NMR spectroscopic analysis of the crude residue. The crude residue was purified by column chromatography on silica gel to afford Michael adduct *rac*-**3** as a mixture of diastereomers.

General Procedure B for the Asymmetric Michael Addition

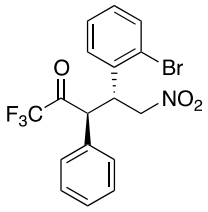


A flame-dried 20-mL scintillation vial containing a magnetic stir bar and activated 3Å molecular sieves (powder form, 50 mg) was charged with 1,1,1-trifluoromethyl ketone **1** (0.210 mmol, 1.050 equiv) and nitroolefin **2** (0.200 mmol, 1.000 equiv) in toluene (2.0 mL, 0.1 M). The resulting turbid solution was cooled to 0 °C in an ice bath. **QD-TU** (3.0 mg, 0.005 mmol, 0.025 equiv) was added and the reaction was capped. The reaction was allowed to stir at 0 °C until adjudged complete by TLC, generally 1 h. The reaction was quenched by addition of 2 M HCl (2 mL). The biphasic solution was diluted with H_2O (5 mL) and EtOAc (10 mL). The layers were separated and the organic layer was washed with brine (5 mL), dried over Na_2SO_4, filtered, and concentrated *in vacuo*. The diastereomeric ratio was determined by ^{19}F NMR spectroscopic analysis of the crude residue. The crude residue was purified by column chromatography on silica gel to afford Michael adduct **3**.

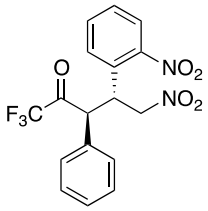


(3*S*,4*R*)-1,1,1-trifluoro-5-nitro-3,4-diphenylpentan-2-one (3aa): The title compound was prepared according to General Procedure B using 1,1,1-trifluoromethyl ketone **1a** (39.5 mg, 0.210 mmol) and nitroolefin **2a** (29.8 mg, 0.200 mmol) affording **3aa** (66.2 mg, 0.196 mmol, 98% yield, >20:1 dr) as a white solid (mp: 121-122 ºC). Analytical data for **3aa**: **^{1}H NMR** (600 MHz, $CDCl_3$): δ 7.47-7.40 (m, 5H), 7.37-7.34 (m, 2H), 7.32-7.29 (m, 3H), 4.64 (d, J = 11.6 Hz, 1H), 4.46 (dd, J = 12.7, 9.6 Hz, 1H), 4.35-4.31 (m, 1H), 4.27 (dd, J = 12.6, 4.2 Hz, 1H); **^{13}C NMR** (150 MHz, $CDCl_3$): δ 188.4 (q, J_{C-F} = 35.1 Hz), 136.3, 131.6, 130.0, 129.6, 129.2, 128.8, 128.6, 127.8, 115.3 (q, J_{C-F} = 291.0 Hz), 78.1, 55.7, 46.0; **^{19}F NMR** (564 MHz, $CDCl_3$): δ -

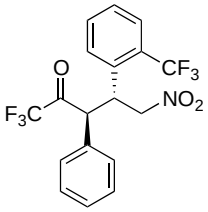
77.9; **IR** (thin film): 1757, 1557, 1382, 1204, 1147, 699 cm^{-1}; **TLC** (4:1 hexanes:ethyl acetate): R_f = 0.48; **HRMS** (ESI): Calcd. for $C_{18}H_{18}F_3NaNO_4$ ($[M+MeOH+Na]^+$): 392.1086, Found: 392.1093; **HPLC**: Chiralpak IC, H:IPA = 99:1, flow rate = 1.0 mL/min, λ = 230 nm, $t_{R\ (minor)}$ 8.4 min, $t_{R\ (major)}$ 9.7 min, 95:5 er; $[\alpha]_D$ +192 (*c* = 0.9, $CHCl_3$).



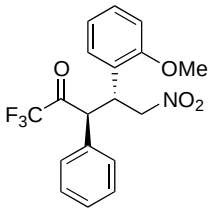
(3*S*,4*R*)-4-(2-bromophenyl)-1,1,1-trifluoro-5-nitro-3-phenylpentan-2-one (3ab): The title compound was prepared according to General Procedure B using 1,1,1-trifluoromethyl ketone **1a** (39.5 mg, 0.210 mmol) and nitroolefin **2b** (45.6 mg, 0.200 mmol) affording **3ab** (79.2 mg, 0.190 mmol, 95% yield, >20:1 dr) as a white solid (mp: 121-122 ºC). Analytical data for **3ab**: **^{1}H NMR** (600 MHz, $CDCl_3$): δ 7.64 (d, *J* = 6.4 Hz, 1H), 7.47-7.39 (m, 5H), 7.30 (t, *J* = 7.4 Hz, 1H), 7.21-7.17 (m, 1H), 7.18 (t, *J* = 7.6 Hz, 1H), 5.08 (br s, 1H), 4.71 (br s, 1H), 4.57 (br s, 1H), 4.30 (br s, 1H); **^{13}C NMR** (150 MHz, $CDCl_3$): δ 188.8 (q, J_{C-F} = 35.0 Hz), 135.4, 133.9, 131.3, 129.9, 129.6, 129.2, 128.2, 127.0, 115.3 (q, J_{C-F} = 291.0 Hz), 75.7, 54.1, 44.3; **^{19}F NMR** (376 MHz, $CDCl_3$): δ -77.1; **IR** (thin film): 1759, 1556, 1376, 1207, 1152, 1026, 756 cm^{-1}; **TLC** (4:1 hexanes:ethyl acetate): R_f = 0.49; **HRMS** (ESI): Calcd. for $C_{18}H_{17}BrF_3NaNO_4$ ($[M+MeOH+Na]^+$): 470.0191, Found: 470.0195; **HPLC**: Chiralpak IC, H:IPA = 99:1, flow rate = 1.0 mL/min, λ = 230 nm, $t_{R\ (minor)}$ 5.9 min, $t_{R\ (major)}$ 6.4 min, 96.5:3.5 er; $[\alpha]_D$ +150 (*c* = 1.1, $CHCl_3$).



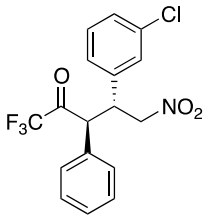
(3*S*,4*R*)-1,1,1-trifluoro-5-nitro-4-(2-nitrophenyl)-3-phenylpentan-2-one (3ac): The title compound was prepared according to General Procedure B using 1,1,1-trifluoromethyl ketone **1a** (39.5 mg, 0.210 mmol) and nitroolefin **2c** (38.8 mg, 0.200 mmol) affording **3ac** (71.9 mg, 0.188 mmol, 94% yield, >20:1 dr) as a white solid (mp: 116-117 ºC). Analytical data for **3ac**: **^{1}H NMR** (600 MHz, $CDCl_3$): δ 8.01 (d, *J* = 7.8 Hz, 1H), 7.61 (t, *J* = 7.6 Hz, 1H), 7.52-7.36 (m, 7H), 5.17 (d, *J* = 9.5 Hz, 1H), 4.81 (br s, 1H), 4.71-4.69 (m, 1H), 4.41 (br s, 1H); **^{13}C NMR** (150 MHz, $CDCl_3$): δ 188.9 (q, J_{C-F} = 35.1 Hz), 150.1, 133.7, 131.5, 130.7, 130.0, 129.8, 129.5, 129.3, 127.5, 125.8, 115.2 (q, J_{C-F} = 290.9 Hz), 76.1, 54.4, 40.2; **^{19}F NMR** (376 MHz, $CDCl_3$): δ -77.0; **IR** (thin film): 1757, 1558, 1530, 1353, 1209, 1154, 1033, 700 cm^{-1}; **TLC** (4:1 hexanes:ethyl acetate): R_f = 0.30; **HRMS** (ESI): Calcd. for $C_{17}H_{13}F_3NaN_2O_5$ ($[M+Na]^+$): 405.0675, Found: 405.0680; **HPLC**: Chiralpak IC, H:IPA = 95:5, flow rate = 1.0 mL/min, λ = 230 nm, $t_{R\ (minor)}$ 6.6 min, $t_{R\ (major)}$ 7.3 min, 97.5:2.5 er; $[\alpha]_D$ +262 (*c* = 0.9, $CHCl_3$).



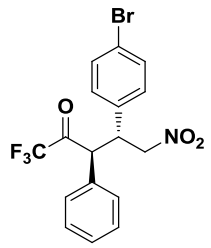
(3*S*,4*R*)-1,1,1-trifluoro-5-nitro-3-phenyl-4-(2-(trifluoromethyl)phenyl)pentan-2-one (3ad): The title compound was prepared according to General Procedure B using 1,1,1-trifluoromethyl ketone **1a** (39.5 mg, 0.210 mmol) and nitroolefin **2d** (43.4 mg, 0.200 mmol) affording **3ad** (77.9 mg, 0.192 mmol, 96% yield, >20:1 dr) as a white solid (mp: 67-68 ºC). Analytical data for **3ad**: **^{1}H NMR** (600 MHz, $CDCl_3$): δ 7.77 (d, *J* = 7.8 Hz, 1H), 7.53 (t, *J* = 7.6 Hz, 1H), 7.48-7.40 (m, 6H), 7.29 (d, *J* = 7.8 Hz, 1H), 5.23 (d, *J* = 10.3 Hz, 1H), 4.61 (dd, *J* = 12.6, 4.6 Hz, 1H), 4.50-4.47 (m, 1H), 4.34 (dd, *J* = 12.6, 4.4 Hz, 1H); **^{13}C NMR** (150 MHz, $CDCl_3$): δ 188.7 (q, J_{C-F} = 34.8 Hz), 135.2, 132.5, 131.2, 129.9, 129.7, 129.5, 129.0, 128.6, 127.4 (q, J_{C-F} = 6.0 Hz), 126.9, 124.2 (q, J_{C-F} = 272.4 Hz), 115.3 (q, J_{C-F} = 291.5 Hz), 76.7, 54.1, 41.5; **^{19}F NMR** (376 MHz, $CDCl_3$): δ -58.7, -77.2; **IR** (thin film): 1761, 1558, 1457, 1376, 1312, 1210, 1158, 1121, 1037 cm^{-1}; **TLC** (4:1 hexanes:ethyl acetate): R_f = 0.48; **HRMS** (ESI): Calcd. for $C_{19}H_{17}F_6NaNO_4$ ($[M+MeOH+Na]^+$): 460.0960, Found: 460.0964; $[\alpha]_D$ +180 (*c* = 1.0, $CHCl_3$).



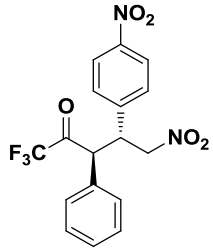
(3*S*,4*R*)-1,1,1-trifluoro-4-(2-methoxyphenyl)-5-nitro-3-phenylpentan-2-one (3ae): The title compound was prepared according to General Procedure B using 1,1,1-trifluoromethyl ketone **1a** (39.5 mg, 0.210 mmol) and nitroolefin **2e** (35.8 mg, 0.200 mmol) affording **3ae** (72.3 mg, 0.197 mmol, 98% yield, >20:1 dr) as a white solid (mp: 80-81 ºC). Analytical data for **3ae**: **^{1}H NMR** (600 MHz, $CDCl_3$): δ 7.45-7.39 (m, 5H), 7.28 (dt, *J* = 8.0, 1.4 Hz, 1H), 7.21 (d, *J* = 7.8 Hz, 1H), 6.93-6.91 (m, 2H), 5.08 (d, *J* = 11.5 Hz, 1H), 4.73 (dd, *J* = 12.6, 9.2 Hz, 1H), 4.44-4.40 (m, 1H), 4.24 (dd, *J* = 12.6, 4.3 Hz, 1H), 3.93 (s, 3H); **^{13}C NMR** (150 MHz, $CDCl_3$): δ 189.0 (q, $J_{C–F}$ = 34.8 Hz), 157.1, 132.2, 131.3, 129.8, 129.3, 129.0, 123.5, 121.1, 115.4 (q, $J_{C–F}$ = 291.5 Hz), 111.2, 75.9, 55.3, 53.0, 44.1 (Rotomer A), 43.0 (Rotomer B); **^{19}F NMR** (564 MHz, $CDCl_3$): δ -78.1; **IR** (thin film): 1759, 1635, 1557, 1495, 1378, 1248, 1207, 1152, 1027 cm^{-1}; **TLC** (4:1 hexanes:ethyl acetate): R_f = 0.45; **HRMS** (ESI): Calcd. for $C_{18}H_{16}F_3NaNO_4$ ($[M+Na]^+$): 390.0929, Found: 390.0934; **HPLC**: Chiralpak IC, H:IPA = 99:1, flow rate = 1.0 mL/min, λ = 230 nm, $t_{R\ (minor)}$ 7.2 min, $t_{R\ (major)}$ 7.8 min, 94:6 er; **[α]$_D$** +190 (*c* = 0.8, $CHCl_3$).



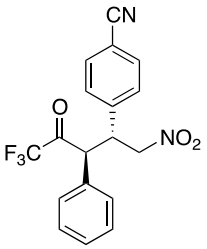
(3*S*,4*R*)-4-(3-chlorophenyl)-1,1,1-trifluoro-5-nitro-3-phenylpentan-2-one (3af): The title compound was prepared according to General Procedure B using 1,1,1-trifluoromethyl ketone **1a** (39.5 mg, 0.210 mmol) and nitroolefin **2f** (36.7 mg, 0.200 mmol) affording **3af** (72.8 mg, 0.196 mmol, 98% yield, 19:1 dr) as a white solid (mp: 129-131 ºC). Analytical data for **3af**: **^{1}H NMR** (600 MHz, $CDCl_3$): δ 7.48-7.42 (m, 3H), 7.38-7.37 (m, 2H), 7.32 (s, 1H), 7.30-7.29 (m, 2H), 7.22-7.19 (m, 1H), 4.60 (d, *J* = 11.3 Hz, 1H), 4.43 (dd, *J* = 12.5, 9.3 Hz, 1H), 4.32-4.24 (m, 2H); **^{13}C NMR** (150 MHz, $CDCl_3$): δ 188.2 (q, $J_{C–F}$ = 35.1 Hz), 138.4, 135.1, 131.1, 130.5, 130.1, 129.7, 128.9, 128.8, 128.1, 126.0, 115.2 (q, $J_{C–F}$ = 290.9 Hz), 77.7, 55.5, 45.6; **^{19}F NMR** (376 MHz, $CDCl_3$): δ -77.8; **IR** (thin film): 1759, 1557, 1377, 1209, 1153, 656 cm^{-1}; **TLC** (4:1 hexanes:ethyl acetate): R_f = 0.46; **HRMS** (ESI): Calcd. for $C_{18}H_{17}ClF_3NaNO_4$ ($[M+MeOH+Na]^+$): 426.0696, Found: 426.0701; **HPLC**: Chiralpak IC, H:IPA = 99:1, flow rate = 1.0 mL/min, λ = 230 nm, $t_{R\ (minor)}$ 7.6 min, $t_{R\ (major)}$ 8.7 min, 93.5:6.5 er; **[α]$_D$** +199 (*c* = 0.8, $CHCl_3$).



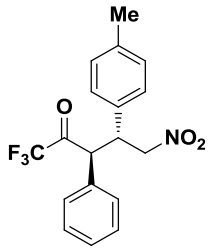
(3*S*,4*R*)-4-(4-bromophenyl)-1,1,1-trifluoro-5-nitro-3-phenylpentan-2-one (3ag): The title compound was prepared according to General Procedure B using 1,1,1-trifluoromethyl ketone **1a** (39.5 mg, 0.210 mmol) and nitroolefin **2g** (45.6 mg, 0.200 mmol) affording **3ag** (82.4 mg, 0.198 mmol, 99% yield, 11:1 dr) as a white solid (mp: 114-115 ºC). Analytical data for **3ag**: **^{1}H NMR** (400 MHz, $CDCl_3$): δ 7.53 (d, *J* = 8.5 Hz, 2H), 7.49-7.38 (m, 5H), 7.23 (d, *J* = 8.5 Hz, 2H), 4.62 (d, *J* = 11.2 Hz, 1H), 4.44 (dd, *J* = 12.4, 9.3 Hz, 1H), 4.34-4.25 (m, 2H); **^{13}C NMR** (150 MHz, $CDCl_3$): δ 188.6 (q, $J_{C–F}$ = 35.1 Hz), 135.4, 132.4, 131.2, 130.1, 129.8, 129.6, 128.8, 122.7, 118.2 (q, $J_{C–F}$ = 291.0 Hz), 77.8, 55.6, 45.5; **^{19}F NMR** (376 MHz, $CDCl_3$): δ -77.7; **IR** (thin film): 2255, 1761, 1560, 1383, 1159, 903 cm^{-1}; **TLC** (10:1 hexanes:ethyl acetate): R_f = 0.15; **HRMS** (ESI): Calcd. for $C_{18}H_{16}BrF_3NO_3$ ($[M+H+MeOH-H_2O]^+$): 430.0266, Found: 430.0272; **SFC**: Chiralpak AS, 1.5% MeOH, flow rate = 1.5 mL/min, λ = 210 nm, $t_{R\ (minor)}$ 8.2 min, $t_{R\ (major)}$ 12.7 min, 95.6:4.4 er; **[α]$_D$** +184.5 (*c* = 1.0, $CHCl_3$).



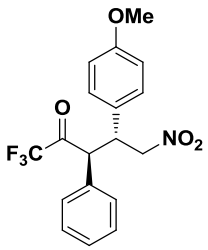
(3*S*,4*R*)-1,1,1-trifluoro-5-nitro-4-(4-nitrophenyl)-3-phenylpentan-2-one (3ah): The title compound was prepared according to General Procedure B using 1,1,1-trifluoromethyl ketone **1a** (39.5 mg, 0.210 mmol) and nitroolefin **2h** (38.8 mg, 0.200 mmol) affording **3ah** (75.7 mg, 0.198 mmol, 99% yield, 7:1 dr) as a white solid (mp: 118-119 ºC). Analytical data for **3ah**: **^{1}H NMR** (600 MHz, $CDCl_3$): δ 7.52 (d, *J* = 8.5 Hz, 2H), 7.49-7.45 (m, 5H), 7.22 (d, *J* = 8.5 Hz, 2H), 4.61 (d, *J* = 11.3 Hz, 1H), 4.46 (dd, *J* = 12.4, 9.4 Hz, 1H), 4.33-4.26 (m, 2H); **^{13}C NMR** (150 MHz, $CDCl_3$): δ 188.6 (q, J_{C-F} = 35.0 Hz), 135.4, 132.4, 131.2, 130.1, 129.8, 129.6, 128.8, 122.7, 118.2 (q, J_{C-F} = 291.0 Hz), 77.8, 55.6, 45.5; **^{19}F NMR** (564 MHz, $CDCl_3$): δ -77.7; **IR** (thin film): 1761, 1560, 1352, 1159 cm^{-1}; **TLC** (3:1 hexanes:ethyl acetate): R_f = 0.4; **HRMS** (ESI): Calcd. for $C_{18}H_{16}F_3N_2O_5$ ($[M+H+MeOH-H_2O]^+$): 397.1011, Found: 397.1016; Chiralpak AS, 1.5% MeOH, flow rate = 1.5 mL/min, λ = 210 nm, $t_{R\ (minor)}$ 14.7 min, $t_{R\ (major)}$ 20.6 min, 95.7:4.3 er; **[α]$_D$** +130.4 (*c* = 1.0, $CHCl_3$).



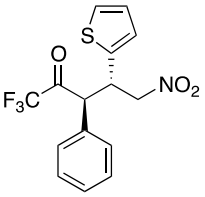
4-((2*R*,3*S*)-5,5,5-trifluoro-1-nitro-4-oxo-3-phenylpentan-2-yl)benzonitrile (3ai): The title compound was prepared according to General Procedure B using 1,1,1-trifluoromethyl ketone **1a** (39.5 mg, 0.210 mmol) and nitroolefin **2i** (34.8 mg, 0.200 mmol) affording **3ai** (69.6 mg, 0.192 mmol, 96% yield, >20:1 dr) as a white solid (mp: 129-131 ºC). Analytical data for **3ai**: **^{1}H NMR** (600 MHz, $CDCl_3$): δ 7.67 (d, *J* = 8.2 Hz, 2H), 7.49-7.43 (m, 5H), 7.36 (d, *J* = 6.8 Hz, 2H), 4.61 (d, *J* = 11.5 Hz, 1H), 4.46 (dd, *J* = 13.0, 9.8 Hz, 1H), 4.39-4.35 (m, 1H), 4.29 (dd, *J* = 13.0, 10.0 Hz, 1H); **^{13}C NMR** (150 MHz, $CDCl_3$): δ 188.0 (q, J_{C-F} = 35.1 Hz), 141.8, 132.9, 130.6, 130.2, 130.0, 128.8, 128.7, 118.1, 115.2 (q, J_{C-F} = 290.9 Hz), 112.7, 77.4, 55.4, 45.8; **^{19}F NMR** (376 MHz, $CDCl_3$): δ -77.6; **IR** (thin film): 2231, 1759, 1647, 1557, 1378, 1211, 1153 cm^{-1}; **TLC** (4:1 hexanes:ethyl acetate): R_f = 0.25; **HRMS** (ESI): Calcd. for $C_{19}H_{17}F_3NaN_2O_4$ ($[M+MeOH+Na]^+$): 417.1038, Found: 417.1043; **HPLC**: Chiralpak IC, H:IPA = 95:5, flow rate = 1.0 mL/min, λ = 230 nm, $t_{R\ (minor)}$ 21.3 min, $t_{R\ (major)}$ 22.8 min, 96:4 er; **[α]$_D$** +245 (*c* = 0.7, $CHCl_3$).



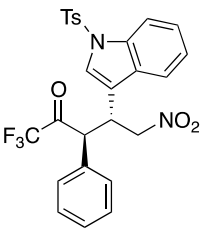
(3*S*,4*R*)-1,1,1-trifluoro-5-nitro-3-phenyl-4-(p-tolyl)pentan-2-one (3aj): The title compound was prepared according to General Procedure B using 1,1,1-trifluoromethyl ketone **1a** (39.5 mg, 0.210 mmol) and nitroolefin **2j** (32.6 mg, 0.200 mmol) affording **3aj** (67.5 mg, 0.192 mmol, 96% yield, 8:1 dr) as a white solid (mp: 111-112 ºC). Analytical data for **3aj**: **^{1}H NMR** (600 MHz, $CDCl_3$): δ 7.49-7.42 (m, 5H), 7.23 (d, *J* = 7.9 Hz, 2H), 7.18 (d, *J* = 7.9 Hz, 2H), 4.67 (d, *J* = 11.5 Hz, 1H), 4.48 (dd, *J* = 12.1, 9.7 Hz, 1H), 4.34-4.26 (m, 2H), 2.34 (s, 3H); **^{13}C NMR** (150 MHz, $CDCl_3$): δ 188.9 (q, J_{C-F} = 35.2 Hz), 135.4, 133.2, 131.8, 130.0, 129.9, 129.5, 128.9, 127.7, 118.3 (q, J_{C-F} = 291.0 Hz), 78.3, 55.8, 45.8, 21.1; **^{19}F NMR** (564 MHz, $CDCl_3$): δ -77.9; **IR** (thin film): 1761, 1560, 1383, 1159, 841 cm^{-1}; **TLC** (10:1 hexanes:ethyl acetate): R_f = 0.18; **HRMS** (ESI): Calcd. for $C_{19}H_{19}F_3NO_3$ ($[M+H+MeOH-H_2O]^+$): 366.1317, Found: 366.1320; Chiralpak AS, 1.5% MeOH, flow rate = 1.5 mL/min, λ = 210 nm, $t_{R\ (minor)}$ 5.2 min, $t_{R\ (major)}$ 7.1 min, 93.1:6.9 er; **[α]$_D$** +183.4 (*c* = 1.0, $CHCl_3$).



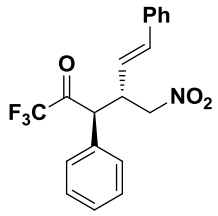
(3*S*,4*R*)-1,1,1-trifluoro-4-(4-methoxyphenyl)-5-nitro-3-phenylpentan-2-one (3ak): The title compound was prepared according to General Procedure B using 1,1,1-trifluoromethyl ketone **1a** (39.5 mg, 0.210 mmol) and nitroolefin **2k** (35.8 mg, 0.200 mmol) affording **3ak** (66.9 mg, 0.182 mmol, 91% yield, 6:1 dr) as a white solid (mp: 124-125 ºC). Analytical data for **3ak**: **^{1}H NMR** (600 MHz, $CDCl_3$): δ 7.48-7.41 (m, 5H), 7.25 (d, J = 8.7 Hz, 2H), 6.90 (d, J = 8.7 Hz, 2H), 4.62 (d, J = 11.4 Hz, 1H), 4.46 (dd, J = 12.3, 9.5 Hz, 1H), 4.32-4.24 (m, 2H), 3.80 (s, 3H); **^{13}C NMR** (150 MHz, $CDCl_3$): δ 188.9 (q, J_{C-F} = 36.0 Hz), 159.5, 131.8, 130.0, 129.9, 129.5, 129.0, 128.8, 128.0, 116.3 (q, J_{C-F} = 291.0 Hz), 114.6, 78.3, 55.9, 55.2, 45.8; **^{19}F NMR** (564 MHz, $CDCl_3$): δ -77.9; **IR** (thin film): 1761, 1560, 1259, 1159 cm^{-1}; **TLC** (3:1 hexanes:ethyl acetate): R_f = 0.5; **HRMS** (ESI): Calcd. for $C_{19}H_{19}F_3NO_4$ ([M+H+MeOH-H_2O]$^+$): 382.1266, Found: 382.1270; Chiralpak AS, 1.5% MeOH, flow rate = 1.5 mL/min, λ = 210 nm, $t_{R\ (minor)}$ 7.0 min, $t_{R\ (major)}$ 9.8 min, 86.2:13.8 er; **$[\alpha]_D$** +170.1 (c = 1.0, $CHCl_3$).



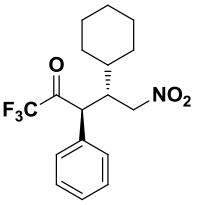
(3*S*,4*S*)-1,1,1-trifluoro-5-nitro-3-phenyl-4-(thiophen-2-yl)pentan-2-one (3al): The title compound was prepared according to General Procedure B using 1,1,1-trifluoromethyl ketone **1a** (39.5 mg, 0.210 mmol) and nitroolefin **2l** (31.0 mg, 0.200 mmol) affording **3al** (66.4 mg, 0.193 mmol, 97% yield, 15:1 dr) as a white solid (mp: 67-69 ºC). Analytical data for **3al**: **^{1}H NMR** (600 MHz, $CDCl_3$): δ 7.47-7.41 (m, 3H), 7.37-7.36 (m, 2H), 7.26 (d, J = 4.8 Hz, 1H), 7.01 (d, J = 2.9 Hz, 1H), 6.95 (dd, J = 5.0, 3.5 Hz, 1H), 4.70 (d, J = 11.5 Hz, 1H), 4.66-4.62 (m, 1H), 4.39 (dd, J = 13.1, 8.6 Hz, 1H), 4.30 (dd, J = 13.1, 4.2 Hz, 1H); **^{13}C NMR** (150 MHz, $CDCl_3$): δ 188.3 (q, J_{C-F} = 35.3 Hz), 138.7, 131.3, 130.1, 129.7, 128.8, 127.3, 127.1, 125.6, 115.3 (q, J_{C-F} = 291.0 Hz), 78.4, 56.7, 41.2; **^{19}F NMR** (376 MHz, $CDCl_3$): δ -77.4; **IR** (thin film): 1759, 1557, 1378, 1209, 1154, 1033, 699 cm^{-1}; **TLC** (4:1 hexanes:ethyl acetate): R_f = 0.50; **HRMS** (ESI): Calcd. for $C_{16}H_{16}F_3NaNO_4S$ ([M+MeOH+Na]$^+$): 398.0650, Found: 398.0654; **HPLC**: Chiralpak IC, H:IPA = 99:1, flow rate = 1.0 mL/min, λ = 230 nm, $t_{R\ (minor)}$ 7.9 min, $t_{R\ (major)}$ 8.7 min, 91.5:8.5 er; **$[\alpha]_D$** +189 (c = 1.1, $CHCl_3$).



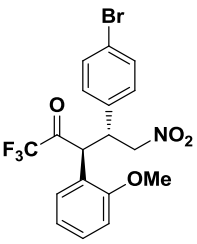
(3*S*,4*R*)-1,1,1-trifluoro-5-nitro-3-phenyl-4-(1-tosyl-1*H*-indol-3-yl)pentan-2-one (3am): The title compound was prepared according to General Procedure B using 1,1,1-trifluoromethyl ketone **1a** (39.5 mg, 0.210 mmol) and nitroolefin **2m** (68.5 mg, 0.200 mmol) affording **3am** (97.9 mg, 0.185 mmol, 92% yield, >20:1 dr) as a white foam (mp: 72-74 ºC). Analytical data for **3am**: **^{1}H NMR** (600 MHz, $CDCl_3$): δ 7.96 (d, J = 8.2 Hz, 1H), 7.67 (d, J = 8.5 Hz, 2H), 7.62-7.60 (m, 2H), 7.47-7.41 (m, 3H), 7.38-7.32 (m, 3H), 7.31 (t, J = 7.1 Hz, 1H), 7.20 (d, J = 8.2 Hz, 2H), 4.88 (d, J = 11.3 Hz, 1H), 4.55-4.51 (m, 1H), 4.45-4.37 (m, 2H), 2.32 (s, 3H); **^{13}C NMR** (150 MHz, $CDCl_3$): δ 188.6 (q, J_{C-F} = 35.1 Hz), 145.3, 134.9, 134.5, 131.3, 130.0, 129.9, 129.7, 128.9, 128.8, 126.6, 125.5, 124.7, 123.8, 118.9, 118.4, 115.3 (q, J_{C-F} = 290.9 Hz), 113.9, 76.5, 55.0, 37.0, 21.5; **^{19}F NMR** (376 MHz, $CDCl_3$): δ -77.4; **IR** (thin film): 1759, 1646, 1556, 1448, 1372, 1212, 1174, 749 cm^{-1}; **TLC** (4:1 hexanes:ethyl acetate): R_f = 0.31; **HRMS** (ESI): Calcd. for $C_{26}H_{22}F_3N_2O_5S$ ([M+H]$^+$): 531.1202, Found: 531.1209; **HPLC**: Chiralpak IC, H:IPA = 85:15, flow rate = 1.0 mL/min, λ = 280 nm, $t_{R\ (major)}$ 13.7 min, $t_{R\ (minor)}$ 16.9 min, 88:12 er; **$[\alpha]_D$** +114 (c = 0.9, $CHCl_3$).



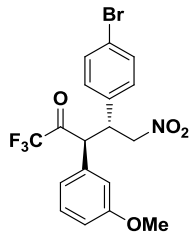
(3*S*,4*S*,*E*)-1,1,1-trifluoro-4-(nitromethyl)-3,6-diphenylhex-5-en-2-one (3an): The title compound was prepared according to General Procedure B using 1,1,1-trifluoromethyl ketone **1a** (39.5 mg, 0.210 mmol) and nitroolefin **2n** (35.0 mg, 0.200 mmol) affording **3an** (33.6 mg, 0.0925 mmol, 46% yield with 50% conversion, >20:1 dr) as a white solid (mp: 123-124 ºC). Analytical data for **3an**: **^{1}H NMR** (600 MHz, $CDCl_3$): δ 7.47-7.29 (m, 10H), 6.67 (d, *J* = 15.6 Hz, 1H), 6.05 (dd, *J* = 15.6, 9.1 Hz, 1H), 4.46 (d, *J* = 10.8 Hz, 1H), 4.25-4.24 (m, 2H), 3.90-2.85 (m, 1H); **^{13}C NMR** (150 MHz, $CDCl_3$): δ 189.3 (q, $J_{C–F}$ = 36.0 Hz), 136.8, 135.7, 131.4, 130.0, 129.5, 128.9, 128.7, 128.4, 126.7, 123.3, 118.4 (q, $J_{C–F}$ = 291.0 Hz), 54.6, 44.1; **^{19}F NMR** (376 MHz, $CDCl_3$): δ -78.0; **IR** (thin film): 1761, 1560, 1383, 1159, 965 cm^{-1}; **TLC** (5:1 hexanes:ethyl acetate): R_f = 0.31; **HRMS** (ESI): Calcd. for $C_{20}H_{19}F_3NO_3$ ($[M+H+MeOH-H_2O]^+$): 378.1317, Found: 378.1321; Chiralpak AS, 1.5% MeOH, flow rate = 1.5 mL/min, λ = 210 nm, $t_{R\ (minor)}$ 6.3 min, $t_{R\ (major)}$ 7.8 min, 90.4:9.6 er; **[α]$_D$** +82.2 (*c* = 1.0, $CHCl_3$).



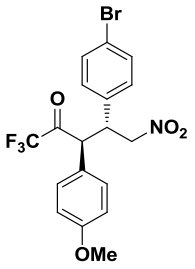
(3*S*,4*S*)-4-cyclohexyl-1,1,1-trifluoro-5-nitro-3-phenylpentan-2-one (3ao): The title compound was prepared according to General Procedure B using 1,1,1-trifluoromethyl ketone **1a** (39.5 mg, 0.210 mmol) and nitroolefin **2o** (31.0 mg, 0.200 mmol) affording **3ao** (28.7 mg, 0.0836 mmol, 42% yield with 43% conversion, >20:1 dr) as a white solid (mp: 100-101 ºC). Analytical data for **3ao**: **^{1}H NMR** (600 MHz, $CDCl_3$): δ 7.40-7.37 (m, 3H), 7.28-7.26 (m, 2H), 4.39 (d, *J* = 11.4 Hz, 1H), 4.19 (dd, *J* = 14.4, 6.0 Hz, 1H), 4.10 (dd, *J* = 14.4, 4.8 Hz, 1H), 3.31-3.27 (m, 1H), 1.85-1.79 (m, 2H), 1.75-1.70 (m, 3H), 1.50-1.45 (m, 1H), 1.32-1.10 (m, 4H), 1.02 (dq, *J* = 12.2, 3.6 Hz, 1H); **^{13}C NMR** (150 MHz, $CDCl_3$): δ 190.3 (q, $J_{C–F}$ = 34.5 Hz), 131.8, 129.7, 129.5, 129.3, 118.7 (q, $J_{C–F}$ = 291.0 Hz), 74.1, 53.1, 44.1, 39.8, 31.2, 28.1, 26.5, 26.4, 26.1; **^{19}F NMR** (376 MHz, $CDCl_3$): δ -77.1; **IR** (thin film): 2035, 2865, 1761, 1560, 1143 cm^{-1}; **TLC** (5:1 hexanes:ethyl acetate): R_f = 0.49; **HRMS** (ESI): Calcd. for $C_{18}H_{23}F_3NO_3$ ($[M+H+MeOH-H_2O]^+$): 358.1630, Found: 358.1634; Chiralpak OD, 1.0% MeOH, flow rate = 1.5 mL/min, λ = 210 nm, $t_{R\ (minor)}$ 6.3 min, $t_{R\ (major)}$ 7.0 min, 94.1:5.9 er; **[α]$_D$** +171.0 (*c* = 1.0, $CHCl_3$).



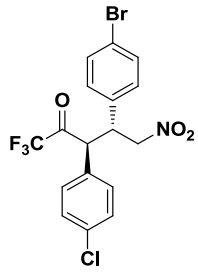
(3*S*,4*R*)-4-(4-bromophenyl)-1,1,1-trifluoro-3-(2-methoxyphenyl)-5-nitropentan-2-one (3bg): The title compound was prepared according to General Procedure B using 1,1,1-trifluoromethyl ketone **1b** (45.8 mg, 0.210 mmol) and nitroolefin **2g** (45.6 mg, 0.200 mmol) affording **3bg** (86.5 mg, 0.194 mmol, 97% yield, >20:1 dr) as a white solid (mp: 115-116 ºC). Analytical data for **3bg**: **^{1}H NMR** (600 MHz, $CDCl_3$): δ 7.51 (d, *J* = 8.4 Hz, 2H), 7.43 (dt, *J* = 8.4, 1.6 Hz, 1H), 7.25 (d, *J* = 8.4 Hz, 2H), 7.20 (dd, *J* = 7.7, 1.5 Hz, 1H), 7.06 (dt, *J* = 7.5, 0.6 Hz, 1H), 7.02 (d, *J* = 8.3 Hz, 1H), 5.09 (d, *J* = 11.0 Hz, 1H), 4.54 (dd, *J* = 12.8, 11.1 Hz, 1H), 4.33-4.28 (m, 1H), 4.26 (dd, *J* = 12.9, 4.4 Hz, 1H), 3.96 (s, 3H); **^{13}C NMR** (150 MHz, $CDCl_3$): δ 188.7 (q, $J_{C–F}$ = 34.5 Hz), 157.2, 136.2, 132.3, 131.0, 129.8, 128.6, 122.4, 121.9, 119.1, 118.1 (q, $J_{C–F}$ = 291.1 Hz), 111.5, 77.7, 56.0, 47.9, 44.9; **^{19}F NMR** (564 MHz, $CDCl_3$): δ -77.8; **IR** (thin film): 1761, 1560, 1491, 1158, 1012 cm^{-1}; **TLC** (5:1 hexanes:ethyl acetate): R_f = 0.31; **HRMS** (ESI): Calcd. for $C_{18}H_{15}BrF_3NNaO_4$ ($[M+Na]^+$): 468.0034, Found: 468.0037; Chiralpak AS, 5.0% MeOH, flow rate = 1.5 mL/min, λ = 210 nm, $t_{R\ (minor)}$ 4.4 min, $t_{R\ (major)}$ 5.1 min, 95.4:4.6 er; **[α]$_D$** +242.2 (*c* = 1.0, $CHCl_3$).



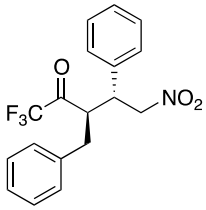
(3*S*,4*R*)-4-(4-bromophenyl)-1,1,1-trifluoro-3-(3-methoxyphenyl)-5-nitropentan-2-one (3cg): The title compound was prepared according to General Procedure B using 1,1,1-trifluoromethyl ketone **1c** (45.8 mg, 0.210 mmol) and nitroolefin **2g** (45.6 mg, 0.200 mmol) affording **3cg** (85.7 mg, 0.192 mmol, 96% yield, >20:1 dr) as a white solid (mp: 124-125 ºC). Analytical data for **3cg**: **^{1}H NMR** (600 MHz, $CDCl_3$): δ 7.51 (d, *J* = 8.4 Hz, 2H), 7.40 (t, *J* = 8.0 Hz, 1H), 7.22 (d, *J* = 8.4 Hz, 2H), 6.98-6.95 (m, 2H), 6.90 (s, 1H), 4.56 (d, *J* = 11.4 Hz, 1H), 4.48 (dd, *J* = 14.1, 11.2 Hz, 1H), 4.31-4.27 (m, 2H), 3.86 (s, 3H); **^{13}C NMR** (150 MHz, $CDCl_3$): δ 188.5 (q, $J_{C–F}$ = 36.0 Hz), 160.6, 135.3, 132.5, 132.4, 131.2, 129.6, 122.7, 118.2 (q, $J_{C–F}$ = 291.0Hz), 114.8, 77.9, 55.5, 55.4, 45.4; **^{19}F NMR** (564 MHz, $CDCl_3$): δ -77.7; **IR** (thin film): 1761, 1599, 1560, 1491, 1267, 1150 cm^{-1}; **TLC** (5:1 hexanes:ethyl acetate): R_f = 0.31; **HRMS** (ESI): Calcd. for $C_{18}H_{15}BrF_3NNaO_4$ ($[M+Na]^+$): 468.0034, Found: 468.0028; Chiralpak AS, 5.0% MeOH, flow rate = 1.5 mL/min, λ = 210 nm, $t_{R\ (minor)}$ 4.7 min, $t_{R\ (major)}$ 6.0 min, 88.1:11.9 er; **[α]$_D$** +135.8 (*c* = 1.0, $CHCl_3$).



(3*S*,4*R*)-4-(4-bromophenyl)-1,1,1-trifluoro-3-(4-methoxyphenyl)-5-nitropentan-2-one (3dg): The title compound was prepared according to General Procedure B using 1,1,1-trifluoromethyl ketone **1d** (45.8 mg, 0.210 mmol) and nitroolefin **2g** (45.6 mg, 0.200 mmol) affording **3dg** (86.7 mg, 0.195 mmol, 97% yield, >20:1 dr) as a white solid (mp: 151-152 ºC). Analytical data for **3dg**: **^{1}H NMR** (600 MHz, $CDCl_3$): δ 7.51 (d, *J* = 8.4 Hz, 2H), 7.30 (d, *J* = 8.4 Hz, 2H), 7.21 (d, *J* = 8.4 Hz, 2H), 6.99 (d, *J* = 8.4 Hz, 2H), 4.55 (d, *J* = 11.4 Hz, 1H), 4.45 (dd, *J* = 12.6, 9.6 Hz, 1H), 4.32-4.24 (m, 2H), 3.85 (s, 3H); **^{13}C NMR** (150 MHz, $CDCl_3$): δ 188.6 (q, $J_{C–F}$ = 35.0 Hz), 160.6, 135.6, 132.4, 130.1, 129.5, 122.7, 122.6, 118.2 (q, $J_{C–F}$ = 291.0Hz), 115.5, 77.9, 55.4, 54.9, 45.5; **^{19}F NMR** (564 MHz, $CDCl_3$): δ -77.6; **IR** (thin film): 1761, 1614, 1565, 1514, 1267, 1159 cm^{-1}; **TLC** (5:1 hexanes:ethyl acetate): R_f = 0.29; **HRMS** (ESI): Calcd. for $C_{18}H_{15}BrF_3NNaO_4$ ($[M+Na]^+$): 468.0034, Found: 468.0027; Chiralpak AS, 5.0% MeOH, flow rate = 1.5 mL/min, λ = 210 nm, $t_{R\ (minor)}$ 5.5 min, $t_{R\ (major)}$ 7.2 min, 87.0:13.0 er; **[α]$_D$** +215.5 (*c* = 1.0, $CHCl_3$).

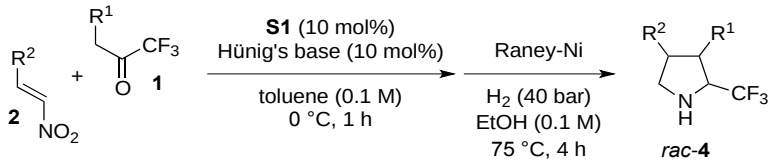


(3*S*,4*R*)-4-(4-bromophenyl)-3-(4-chlorophenyl)-1,1,1-trifluoro-5-nitropentan-2-one (3eg): The title compound was prepared according to General Procedure B using 1,1,1-trifluoromethyl ketone **1e** (46.7 mg, 0.210 mmol) and nitroolefin **2g** (45.6 mg, 0.200 mmol) affording **3eg** (85.8 mg, 0.190 mmol, 95% yield, 17:1 dr) as a white solid (mp: 132-133 ºC). Analytical data for **3eg**: **^{1}H NMR** (600 MHz, $CDCl_3$): δ 7.52 (d, *J* = 8.4 Hz, 2H), 7.48 (d, *J* = 8.5 Hz, 2H), 7.36 (d, *J* = 8.5 Hz, 2H), 7.20 (d, *J* = 8.4 Hz, 2H), 4.63 (d, *J* = 11.3 Hz, 1H), 4.45 (dd, *J* = 12.7, 9.2 Hz, 1H), 4.31-4.23 (m, 2H); **^{13}C NMR** (150 MHz, $CDCl_3$): δ 188.5 (q, $J_{C–F}$ = 36.0 Hz), 136.1, 135.0, 132.5, 130.4, 129.8, 129.6, 129.5, 122.5, 118.1 (q, $J_{C–F}$ = 291.0Hz), 77.6, 55.7, 45.5; **^{19}F NMR** (564 MHz, $CDCl_3$): δ -77.8; **IR** (thin film): 1761, 1560, 1491, 1375, 1159, 1097, 1012 cm^{-1}; **TLC** (5:1 hexanes:ethyl acetate): R_f = 0.36; **MS** (ESI): Calcd. for $C_{17}H_{16}BrClF_3KN_2O_3$ ($[M+K+NH4]^+$): 505.96, Found: 505.97; (**HRMS** (ESI): Calcd. for $C_{17}H_{16}BrClF_3KN_2O_3$ ($[M+K+NH4]^+$): 505.9622, Found: 505.9685.)Chiralpak AS, 5.0% MeOH, flow rate = 1.5 mL/min, λ = 210 nm, $t_{R\ (minor)}$ 5.6 min, $t_{R\ (major)}$ 7.6 min, 93.9:6.1 er; **[α]$_D$** +94.2 (*c* = 1.0, $CHCl_3$).



(3*R*,4*R*)-3-benzyl-1,1,1-trifluoro-5-nitro-4-phenylpentan-2-one (3fa): The title compound was prepared according to General Procedure B using 1,1,1-trifluoromethyl ketone **1f** (42.5 mg, 0.210 mmol) and nitroolefin **2a** (29.8 mg, 0.200 mmol) affording **3fa** (51.3 mg, 0.146 mmol, 73% yield, >20:1 dr) as a white solid (mp: 94-95 ºC). Analytical data for **3fa**: **^{1}H NMR** (600 MHz, $CDCl_3$): δ 7.38-7.27 (m, 6H), 7.23 (d, *J* = 7.3 Hz, 2H), 7.09 (d, *J* = 7.3 Hz, 2H), 4.83 (d, *J* = 7.4 Hz, 1H), 4.03-3.99 (m, 1H), 3.84-3.80 (m, 1H), 3.09-3.03 (m, 2H); **^{13}C NMR** (150 MHz, $CDCl_3$): δ 193.0 (q, J_{C-F} = 35.3 Hz), 135.9, 129.2, 128.9, 128.8, 128.6, 127.9, 127.4, 114.6 (q, J_{C-F} = 290.4 Hz), 76.5, 51.9, 45.2, 35.4 (one carbon missing due to overlap); **^{19}F NMR** (376 MHz, $CDCl_3$): δ -79.3; **IR** (thin film): 1748, 1550, 1456, 1387, 1281, 1204, 1038 cm^{-1}; **TLC** (4:1 hexanes:ethyl acetate): R_f = 0.47; **HRMS** (ESI): Calcd. for $C_{18}H_{16}F_3NaNO_3$ ($[M+Na]^+$): 374.0980, Found: 374.0985; **HPLC**: Chiralpak IC, H:IPA = 99:1, flow rate = 1.0 mL/min, λ = 230 nm, $t_{R\ (minor)}$ 14.2 min, $t_{R\ (major)}$ 18.5 min, 97:3 er; **$[\alpha]_D$** +5 (*c* = 1.1, $CHCl_3$).

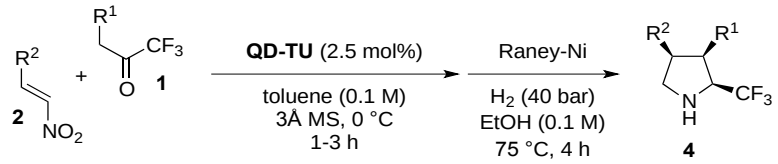
General Procedure C for the One-Pot Racemic Michael Addition/Reductive Cyclization Protocol



***NOTE**: Caution should be used when conducting hydrogenations at elevated pressures and temperatures and necessary experimental precautions should be taken.*

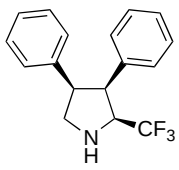
A 20-mL scintillation vial containing a magnetic stir bar was charged with 1,1,1-trifluoromethyl ketone **1** (0.21 mmol, 1.05 equiv), nitroolefin **2** (0.20 mmol, 1.00 equiv), and thiourea **S1** (5.0 mg, 0.01 mmol, 0.05 equiv) in toluene (2.0 mL, 0.1 M). The resulting solution was cooled to 0 °C in an ice bath. Hünig's base (2.0 µL, 0.01 mmol, 0.05 equiv) was added and the reaction was capped. The reaction was allowed to stir at 0 °C until adjudged complete by TLC, generally 1 h. The reaction was removed from the ice bath and diluted with EtOH (2 mL). Raney-Ni (W.R. Grace and Co. Raney®2800, slurry, in H_2O, 200 µL) was added under a N_2 atmosphere. The scintillation vial was carefully placed inside a 600 mL high-pressure vessel (Parr Instrument Co., Model 4605). The vessel was sealed and purged with a H_2 atmosphere (3x) before slowly pressuring the vessel to 40 bar behind a blast shield. The sealed, pressurized vessel was then carefully placed in a pre-heated oil bath (75 °C). The reaction was allowed to stir for 4 h at 75 °C behind a blast shield. After 4 h, the vessel was removed from the oil bath and placed in an ice bath to cool the vessel to room temperature. After the pressure vessel reached room temperature, it was slowly vented to atmospheric pressure. The pressure vessel was opened and the scintillation vial was removed. The turbid solution was filtered through a 2 cm pad of Celite® to remove the Raney-Ni washing with MeOH (3 x 5 mL). The obtained solution was concentrated *in vacuo*. The diastereomeric ratio was determined by ^{19}F NMR spectroscopic analysis of the crude residue. The crude residue was purified by column chromatography on silica gel to afford pyrrolidine *rac*-**4**.

General Procedure D for the Asymmetric Michael Addition/Reductive Cyclization Protocol

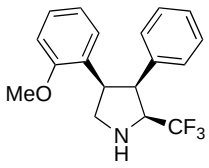


***NOTE**: Caution should be used when conducting hydrogenations at elevated pressures and temperatures and necessary experimental precautions should be taken.*

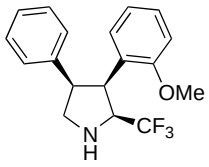
A flame-dried 20-mL scintillation vial containing a magnetic stir bar and activated 3Å molecular sieves (powder form, 50 mg) was charged with 1,1,1-trifluoromethyl ketone **1** (0.210 mmol, 1.050 equiv) and nitroolefin **2** (0.200 mmol, 1.000 equiv) in toluene (2.0 mL, 0.1 M). The resulting turbid solution was cooled to 0 °C in an ice bath. **QD-TU** (3.0 mg, 0.005 mmol, 0.025 equiv) was added and the reaction was capped. The reaction was allowed to stir at 0 °C until adjudged complete by TLC, generally 1 h. The reaction was quenched by addition of 2 M HCl (2 mL). The biphasic solution was diluted with H_2O (5 mL) and EtOAc (10 mL). The layers were separated and the organic layer was washed with brine (5 mL), dried over Na_2SO_4, filtered, and concentrated *in vacuo*. The resulting crude residue was taken up in EtOH (2 mL) and added to a 20-mL scintillation vial containing a magnetic stir bar and Raney-Ni (W.R. Grace and Co. Raney®2800, slurry, in H_2O, 200 µL) in EtOH (2 mL) under a N_2 atmosphere. The scintillation vial was carefully placed inside a 600 mL high-pressure vessel (Parr Instrument Co., Model 4605). The vessel was sealed and purged with a H_2 atmosphere (3x) before slowly pressuring the vessel to 40 bar behind a blast shield. The sealed, pressurized vessel was then carefully placed in a pre-heated oil bath (75 °C). The reaction was allowed to stir for 4 h at 75 °C behind a blast shield. After 4 h, the vessel was removed from the oil bath and placed in an ice bath to cool the vessel to room temperature. After the pressure vessel reached room temperature, it was slowly vented to atmospheric pressure. The pressure vessel was opened and the scintillation vial was removed. The turbid solution was filtered through a 2 cm pad of Celite® to remove the Raney-Ni washing with MeOH (3 x 5 mL). The obtained solution was concentrated *in vacuo*. The diastereomeric ratio was determined by ^{19}F NMR spectroscopic analysis of the crude residue. The crude residue was purified by column chromatography on silica gel to afford pyrrolidine **4**.



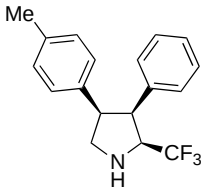
(2*S*,3*S*,4*R*)-3,4-diphenyl-2-(trifluoromethyl)pyrrolidine (4a): The title compound was prepared according to General Procedure D using 1,1,1-trifluoromethyl ketone **1a** (39.5 mg, 0.210 mmol) and nitroolefin **2a** (29.8 mg, 0.200 mmol) affording **4a** (47.2 mg, 0.162 mmol, 81% yield, >20:1 dr) as a colorless oil. Analytical data for **4a**: **^{1}H NMR** (600 MHz, $CDCl_3$): δ 7.15-7.06 (m, 8H), 6.91 (d, *J* = 7.3 Hz, 1H), 4.30 (dq, *J* = 7.8, 1.8 Hz, 1H), 3.93-3.88 (m, 1H), 3.78-3.69 (m, 3H), 2.29 (br s, 1H); **^{13}C NMR** (150 MHz, $CDCl_3$): δ 137.6, 135.6, 130.5, 128.1, 127.8, 127.6, 126.7, 126.3, 125.5 (q, J_{C-F} = 277.8 Hz), 64.4 (q, J_{C-F} = 29.1 Hz), 50.9, 49.7, 48.3 (one carbon missing due to overlap); **^{19}F NMR** (376 MHz, $CDCl_3$): δ -69.4; **IR** (thin film): 3471, 1604, 1295, 1149, 1122, 698 cm^{-1}; **TLC** (4:1 hexanes:ethyl acetate): R_f = 0.30; **HRMS** (ESI): Calcd. for $C_{17}H_{17}F_3N$ ($[M+H]^+$): 292.1314, Found: 292.1315; **HPLC**: Chiralpak IC, H:IPA = 97:3, flow rate = 1.0 mL/min, λ = 230 nm, $t_{R\ (minor)}$ 5.5 min, $t_{R\ (major)}$ 6.2 min, 94.5:5.5 er; **$[\alpha]_D$** +44 (*c* = 1.3, $CHCl_3$).



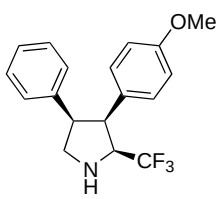
(2*S*,3*S*,4*R*)-4-(2-methoxyphenyl)-3-phenyl-2-(trifluoromethyl)pyrrolidine (4b): The title compound was prepared according to General Procedure D using 1,1,1-trifluoromethyl ketone **1a** (39.5 mg, 0.210 mmol) and nitroolefin **2e** (35.8 mg, 0.200 mmol) affording **4b** (52.8 mg, 0.164 mmol, 82% yield, >20:1 dr) as a pale yellow oil. Analytical data for **4b**: **^{1}H NMR** (600 MHz, $CDCl_3$): δ 7.11-7.02 (m, 6H), 6.77 (d, *J* = 7.4 Hz, 1H), 6.71 (d, *J* = 8.2 Hz, 1H), 6.63 (t, *J* = 7.5 Hz, 1H), 4.32 (dq, *J* = 8.2, 1.9 Hz, 1H), 4.09-4.01 (m, 2H), 3.83 (s, 3H), 3.83-3.80 (m, 1H), 3.56 (t, *J* = 9.2 Hz, 1H), 2.22 (br s, 1H); **^{13}C NMR** (150 MHz, $CDCl_3$): δ 157.1, 136.4, 130.0, 127.4, 127.3, 127.2, 126.4, 125.9, 125.7 (q, $J_{C–F}$ = 277.7 Hz), 119.7, 109.6, 64.3 (q, $J_{C–F}$ = 29.3 Hz), 55.1, 48.7, 47.1, 44.1; **^{19}F NMR** (376 MHz, $CDCl_3$): δ -69.3; **IR** (thin film): 3432, 1541, 1495, 1294, 1247, 1115, 1030 cm^{-1}; **TLC** (4:1 hexanes:ethyl acetate): R_f = 0.29; **HRMS** (ESI): Calcd. for $C_{18}H_{19}F_3NO$ ($[M+H]^+$): 322.1419, Found: 322.1421; **HPLC**: Chiralpak IC, H:IPA = 97:3, flow rate = 1.0 mL/min, λ = 230 nm, $t_{R\ (minor)}$ 5.6 min, $t_{R\ (major)}$ 6.6 min, 93.5:6.5 er; **[α]$_D$** +58 (*c* = 0.7, $CHCl_3$).



(2*S*,3*S*,4*R*)-3-(2-methoxyphenyl)-4-phenyl-2-(trifluoromethyl)pyrrolidine (4c): The title compound was prepared according to General Procedure D using 1,1,1-trifluoromethyl ketone **1b** (45.8 mg, 0.210 mmol) and nitroolefin **2a** (29.8 mg, 0.200 mmol) affording **4c** (46.9 mg, 0.146 mmol, 73% yield, >20:1 dr) as a white solid (mp: 87-88 ºC). Analytical data for **4c**: **^{1}H NMR** (600 MHz, $CDCl_3$): δ 7.43 (d, *J* = 6.5 Hz, 1H), 7.10-7.03 (m, 4H), 6.90 (d, *J* = 7.1 Hz, 1H), 6.82 (t, *J* = 7.4 Hz, 1H), 6.63 (d, *J* = 8.2 Hz, 1H), 4.59 (br s, 1H), 4.33-4.28 (m, 1H), 3.90-3.86 (m, 1H), 3.69-3.63 (m, 2H), 3.51 (s, 3H), 2.26 (br s, 1H); **^{13}C NMR** (150 MHz, $CDCl_3$): δ 157.5, 137.9, 131.2, 128.0, 127.7, 127.5, 126.1, 125.6 (q, $J_{C–F}$ = 277.8 Hz), 124.3, 119.8, 110.4, 64.0 (q, $J_{C–F}$ = 29.6 Hz), 55.4, 49.6, 48.3, 40.1; **^{19}F NMR** (376 MHz, $CDCl_3$): δ -69.3; **IR** (thin film): 3431, 1494, 1294, 1245, 1151, 1123, 1029 cm^{-1}; **TLC** (4:1 hexanes:ethyl acetate): R_f = 0.32; **HRMS** (ESI): Calcd. for $C_{18}H_{19}F_3NO$ ($[M+H]^+$): 322.1419, Found: 322.1421; **HPLC**: Chiralpak IC, H:IPA = 97:3, flow rate = 1.0 mL/min, λ = 230 nm, $t_{R\ (minor)}$ 5.5 min, $t_{R\ (major)}$ 6.2 min, 93.5:6.5 er; **[α]$_D$** -21 (*c* = 1.2, $CHCl_3$).



(2*S*,3*S*,4*R*)-3-phenyl-4-(*p*-tolyl)-2-(trifluoromethyl)pyrrolidine (4d): The title compound was prepared according to General Procedure D using 1,1,1-trifluoromethyl ketone **1a** (39.5 mg, 0.210 mmol) and nitroolefin **2j** (32.6 mg, 0.200 mmol) affording **4d** (47.5 mg, 0.156 mmol, 78% yield, >20:1 dr) as a colorless oil. Analytical data for **4d**: **^{1}H NMR** (600 MHz, $CDCl_3$): δ 7.16-7.13 (m, 5H), 6.89 (d, *J* = 7.7 Hz, 2H), 6.78 (d, *J* = 7.7 Hz, 2H), 4.31-4.26 (m, 1H), 3.88-3.84 (m, 1H), 3.74 (t, *J* = 6.4 Hz, 1H), 3.70-3.68 (m, 2H), 2.26 (br s, 1H), 2.20 (s, 3H); **^{13}C NMR** (150 MHz, $CDCl_3$): δ 135.8, 135.7, 134.4, 130.5, 128.6, 128.0, 127.6, 126.4, 125.5 (q, $J_{C–F}$ = 277.8 Hz), 64.4 (q, $J_{C–F}$ = 29.1 Hz), 50.8, 49.5, 48.6, 20.9; **^{19}F NMR** (376 MHz, $CDCl_3$): δ -69.6; **IR** (thin film): 3431, 1518, 1295, 1150, 1121, 817 cm^{-1}; **TLC** (4:1 hexanes:ethyl acetate): R_f = 0.30; **HRMS** (ESI): Calcd. for $C_{18}H_{19}F_3N$ ($[M+H]^+$): 306.1470, Found: 306.1472; **HPLC**: Chiralpak IC, H:IPA = 97:3, flow rate = 1.0 mL/min, λ = 230 nm, $t_{R\ (minor)}$ 5.2 min, $t_{R\ (major)}$ 6.0 min, 94:6 er; **[α]$_D$** +28 (*c* = 0.9, $CHCl_3$).



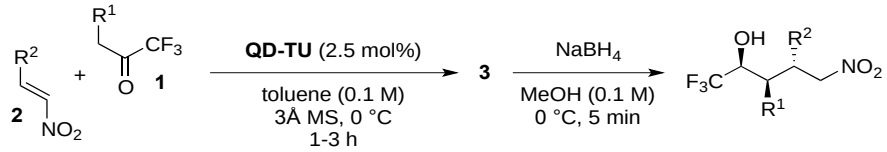
(2*S*,3*S*,4*R*)-3-(4-methoxyphenyl)-4-phenyl-2-(trifluoromethyl)pyrrolidine (4e): The title compound was prepared according to General Procedure D using 1,1,1-trifluoromethyl ketone **1d** (45.8 mg, 0.210 mmol) and nitroolefin **2a** (29.8 mg, 0.200 mmol) affording **4e** (56.0 mg, 0.174 mmol, 87% yield, >20:1 dr) as a pale yellow oil. Analytical data for **4e**: **^{1}H NMR** (600 MHz, $CDCl_3$): δ 7.11-7.05 (m,

5H), 6.91 (d, J = 7.4 Hz, 2H), 6.68 (d, J = 8.6 Hz, 2H), 4.28-4.23 (m, 1H), 3.88-3.83 (m, 1H), 3.73-3.68 (m, 3H), 3.72 (s, 3H), 2.21 (br s, 1H); **^{13}C NMR** (150 MHz, $CDCl_3$): δ 158.2, 137.8, 131.5, 128.1, 127.9, 127.6, 126.3, 125.5 (q, J_{C-F} = 277.8 Hz), 112.9, 64.0 (q, J_{C-F} = 29.6 Hz), 54.9, 50.1, 49.7, 48.3; **^{19}F NMR** (376 MHz, $CDCl_3$): δ -69.4; **IR** (thin film): 3367, 1514, 1296, 1247, 1149, 1119, 1037, 698 cm^{-1}; **TLC** (4:1 hexanes:ethyl acetate): R_f = 0.23; **HRMS** (ESI): Calcd. for $C_{18}H_{19}F_3NO$ ($[M+H]^+$): 322.1419, Found: 322.1421; **HPLC**: Chiralpak IC, H:IPA = 97:3, flow rate = 1.0 mL/min, λ = 230 nm, $t_{R\ (minor)}$ 7.0 min, $t_{R\ (major)}$ 8.2 min, 94:6 er; **$[\alpha]_D$** +35 (c = 0.9, $CHCl_3$).

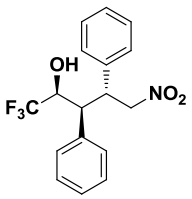
Recrystallization of 3ai

A 1-dram vial was charged with **3ai** (40.0 mg, 0.11 mmol; 96:4 er) in 19:1 hexanes:ethyl acetate (2 mL). The solution was gently warmed until it became homogenous. The vial was then capped and allowed to slowly cool down to -10 °C. After sitting in the freezer overnight, the crystals were collected via vacuum filtration (rinsing with hexanes) to afford **3ai** (35.7 mg, 0.10 mmol, 89% yield, >20:1 dr); **HPLC**: Chiralpak IC, H:IPA = 95:5, flow rate = 1.0 mL/min, λ = 230 nm, $t_{R\ (minor)}$ 19.5 min, $t_{R\ (major)}$ 20.7 min, 99.5:0.5 er.

General Procedure E for the Diastereoselective Reduction of Michael Adduct 3

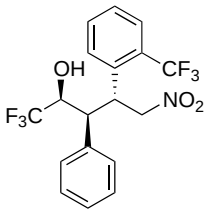


The Michael addition was conducted as described in General Procedures A and B with a modification to the workup. Upon completion of the Michael addition as adjudged by TLC, the reaction was diluted with MeOH (2 mL). $NaBH_4$ (~10 mg) was added to the reaction at 0 °C. After stirring for 5 min at 0 °C, the reaction was carefully quenched with sat. aq. NH_4Cl (2 mL). The biphasic solution was diluted with H_2O (3 mL) and CH_2Cl_2 (5 mL). The layers were separated and the aqueous layer was extracted with CH_2Cl_2 (2 x 5 mL). The combined organic extracts were washed with brine (5 mL), dried over Na_2SO_4, filtered, and concentrated *in vacuo*. The diastereomeric ratio was determined by ^{19}F NMR of the crude residue. The crude residue was purified by column chromatography on silica gel to afford the carbinol.

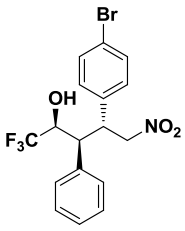


(2*S*,3*S*,4*R*)-1,1,1-trifluoro-5-nitro-3,4-diphenylpentan-2-ol (5aa): The title compound was prepared according to General Procedure E using 1,1,1-trifluoromethyl ketone **1a** (39.5 mg, 0.210 mmol) and nitroolefin **2a** (29.8 mg, 0.200 mmol) affording **5aa** (63.7 mg, 0.188 mmol, 94% yield, >20:1 dr) as colorless crystals (mp: 179-180 ºC). Analytical data for **5aa**: **^{1}H NMR** (600 MHz, $CDCl_3$): δ 7.47-7.37 (m, 10H), 4.50 (dd, J = 12.6, 11.4 Hz, 1H), 4.26-4.18 (m, 2H), 3.81-3.84 (m, 1H), 3.29 (dd, J = 12.0, 1.8 Hz, 1H), 2.25 (t, J = 6.6 Hz, 1H); **^{13}C NMR** (150 MHz, $CDCl_3$): δ 137.1, 134.9, 129.6, 129.3, 128.9, 128.6, 128.0, 127.3 (q, J_{C-F} = 282.0 Hz), 79.5, 70.2 (q, J_{C-F} = 30.0 Hz), 48.1, 46.0; **^{19}F NMR** (376 MHz, $CDCl_3$): δ -75.4; **IR** (thin film): 3699, 3028, 1560, 1166, 1140 cm^-

[1]; **TLC** (5:1 hexanes:ethyl acetate): R_f = 0.24; **HRMS** (ESI): Calcd. for $C_{17}H_{16}F_3NaNO_3$ ($[M+Na]^+$): 362.0980, Found: 362.0980; **$[\alpha]_D$** +21.0 (*c* = 1.0, $CHCl_3$).

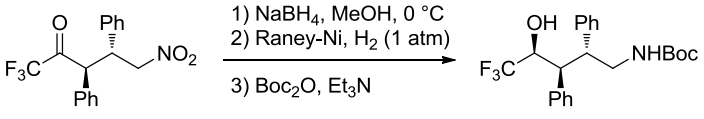


(2*S*,3*S*,4*R*)-1,1,1-trifluoro-5-nitro-3-phenyl-4-(2-(trifluoromethyl)phenyl)pentan-2-ol (5ad): The title compound was prepared according to General Procedure E using 1,1,1-trifluoromethyl ketone **1a** (39.5 mg, 0.210 mmol) and nitroolefin **2d** (43.4 mg, 0.200 mmol) affording **5ad** (70.9 mg, 0.174 mmol, 87% yield, >20:1 dr) as colorless crystals (mp: 151-153 ºC). Analytical data for **5ad**: **^{1}H NMR** (600 MHz, $CDCl_3$): δ 7.80 (d, *J* = 7.9 Hz, 1H), 7.65 (t, *J* = 7.6 Hz, 1H), 7.52-7.40 (m, 7H), 4.48-4.43 (m, 2H), 4.40-4.37 (m, 1H), 3.89-3.85 (m, 1H), 3.74 (d, *J* = 10.9 Hz, 1H), 2.26 (d, *J* = 8.0 Hz, 1H); **^{13}C NMR** (150 MHz, $CDCl_3$): δ 135.9, 134.0, 133.0, 129.9 (q, J_{C-F} = 29.4 Hz), 129.0, 128.7, 128.6, 127.8, 127.3 (q, J_{C-F} = 5.7 Hz), 124.3 (q, J_{C-F} = 281.6 Hz), 124.1 (q, J_{C-F} = 272.4 Hz), 78.7, 70.0 (q, J_{C-F} = 30.0 Hz), 47.5, 41.4, (one carbon missing due to overlap); **^{19}F NMR** (376 MHz, $CDCl_3$): δ -57.7, -75.8; **TLC** (4:1 hexanes:ethyl acetate): R_f = 0.37; **HRMS** (ESI): Calcd. for $C_{18}H_{15}F_6NaNO_3$ ($[M+Na]^+$): 430.0854, Found: 430.0860; **HPLC**: Chiralpak IA, H:IPA = 98:2, flow rate = 1.0 mL/min, λ = 230 nm, $t_{R\ (minor)}$ 11.3 min, $t_{R\ (major)}$ 15.5 min, 96.5:3.5 er; **$[\alpha]_D$** +27 (*c* = 0.6, $CHCl_3$).



(2*S*,3*S*,4*R*)-4-(4-bromophenyl)-1,1,1-trifluoro-5-nitro-3-phenylpentan-2-ol (5ag): The title compound was prepared according to General Procedure E using 1,1,1-trifluoromethyl ketone **1a** (39.5 mg, 0.210 mmol) and nitroolefin **2g** (45.6 mg, 0.200 mmol) affording **5ag** (75.4 mg, 0.180 mmol, 90% yield, >20:1 dr) as colorless crystals (mp: 179-180 ºC). Analytical data for **5ag**: **^{1}H NMR** (600 MHz, $CDCl_3$): δ 7.59 (d, *J* = 8.4 Hz, 2H), 7.45-7.39 (m, 5H), 7.29 (d, *J* = 8.1 Hz, 2H), 4.45 (dd, *J* = 13.2, 11.4 Hz, 1H), 4.25-4.17 (m, 2H), 3.83-3.78 (m, 1H), 3.24 (dd, *J* = 12.0, 2.4 Hz, 1H), 2.25 (d, *J* = 7.8 Hz, 1H); **^{13}C NMR** (150 MHz, $CDCl_3$): δ 136.2, 134.5, 132.8, 129.7, 129.4, 128.8, 127.1 (q, J_{C-F} = 281.6 Hz), 122.7, 79.2, 70.2 (q, J_{C-F} = 30.0 Hz), 48.0, 45.5; **^{19}F NMR** (376 MHz, $CDCl_3$): δ -75.4; **IR** (thin film): 3599, 3337, 1560, 1491, 1383, 1282, 1135 cm^{-1}; **TLC** (10:1 hexanes:ethyl acetate): R_f = 0.15; **HRMS** (ESI): Calcd. for $C_{17}H_{15}BrF_3NNaO_3$ ($[M+Na]^+$): 440.0085, Found: 440.0086; **$[\alpha]_D$** +13.7 (*c* = 1.0, $CHCl_3$).

Procedure for the Synthesis of Amino Alcohol 6

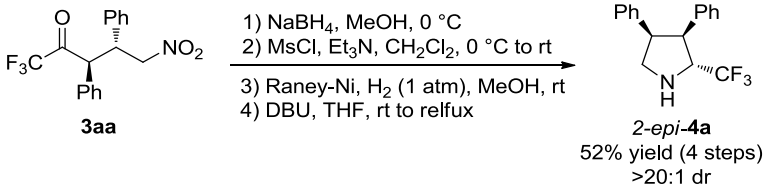


3aa → **6**, 80% yield, >20:1 dr

The Michael addition was conducted as described in General Procedure B with a modification to the workup. Upon completion of the Michael addition as adjudged by TLC, the reaction was diluted with MeOH (2 mL). $NaBH_4$ (10 mg, 0.264 mmol) was added to the reaction at 0 °C. After stirring for 5 min at 0 °C, the mixture was filtered through a pad of celite to remove 3Å molecular sieves. The residue was washed with MeOH (0.5 mL*2). Raney-Ni (W.R. Grace and Co. Raney®2800, slurry, in H_2O, 200 µL) was added to the filtrate. The mixture was put under hydrogen balloon for 1 hour at rt.

Triethylamine (0.139 mL, 1.0 mmol) was added, followed by the addition of Boc anhydride (87.3 mg, 0.4 mmol). The mixture was stirred overnight. The reaction was quenched with sat. aq. NH_4Cl (2 mL). The biphasic solution was diluted with H_2O (3 mL) and EtOAc (5 mL). The layers were separated and the aqueous layer was extracted with EtOAc (2 x 5 mL). The combined organic extracts were washed with brine (5 mL), dried over Na_2SO_4, filtered, and concentrated *in vacuo*. The crude residue was purified by column chromatography on silica gel to afford the amino alcohol **6** (65.9 mg, 0.161 mmol, 80% yield) as a semi-solid. Analytical data for **6**: **^{1}H NMR** (600 MHz, $CDCl_3$): δ 7.44-7.35 (m, 10H), 4.08 (br s, 1H), 3.80 (br s, 1H), 3.54 (br s, 1H), 3.28 (br s, 1H), 3.22 (dd, J = 11.7, 1.2 Hz, 1H), 2.96 (br s, 1H), 2.41 (d, J = 8.8 Hz, 1H), 1.32 (s, 9H); **^{13}C NMR** (150 MHz, $CDCl_3$): δ 155.5, 140.3, 135.9, 129.4, 128.9, 128.3, 128.0, 127.7, 127.5 (q, J_{C-F} = 282.0 Hz), 79.1, 70.6 (q, J_{C-F} = 29.5 Hz), 49.1, 46.9, 44.4, 28.3; **^{19}F NMR** (564 MHz, $CDCl_3$): δ -75.3; **IR** (thin film): 3453, 2981, 1707, 1498, 1367, 1274, 1166, 1135 cm^{-1}; **TLC** (5:1 hexanes:ethyl acetate): R_f = 0.17; **HRMS** (ESI): Calcd. for $C_{22}H_{26}F_3NNaO_3$ ($[M+Na]^+$): 432.1762, Found: 432.1793; **$[\alpha]_D$** +41.8 (c = 1.0, $CHCl_3$).

Procedure for the Synthesis of (2*R*,3*S*,4*R*)-3,4-diphenyl-2-(trifluoromethyl)pyrrolidine (2-*epi*-4a)



The Michael addition was conducted as described in General Procedure B with a modification to the workup. Upon completion of the Michael addition as adjudged by TLC, the reaction was diluted with MeOH (2 mL). $NaBH_4$ (10 mg, 0.264 mmol) was added to the reaction at 0 °C. After stirring for 5 min at 0 °C, the reaction was quenched with sat. aq. NH_4Cl (2 mL). The biphasic solution was diluted with H_2O (3 mL) and EtOAc (5 mL). The layers were separated and the aqueous layer was extracted with EtOAc (2 x 5 mL). The combined organic extracts were washed with brine (5 mL), dried over Na_2SO_4, filtered, and concentrated *in vacuo*.

The crude residue was dissolved in CH_2Cl_2 (2 mL) and the flask was put in an ice bath. Triethylamine (0.056 mL, 0.4 mmol) was added, followed by MsCl (0.023 mL, 0.3 mmol). The mixture was brought to rt and stirred overnight. The reaction was quenched with sat. aq. NaCl (2 mL). The biphasic mixture was diluted with H_2O (3 mL) and CH_2Cl_2 (5 mL). The layers were separated and the aqueous layer was extracted with CH_2Cl_2 (2 x 5 mL). The combined organic extracts were washed with brine (5 mL), dried over Na_2SO_4, filtered, and concentrated *in vacuo*.

The crude residue was dissolved in MeOH (2 mL). Raney-Ni (W.R. Grace and Co. Raney®2800, slurry, in H_2O, 200 µL) was added to the filtrate. The mixture was put under a hydrogen balloon for 1 h at rt. The reaction was quenched with sat. aq. NaCl (2 mL). The biphasic solution was diluted with H_2O (3 mL) and EtOAc (5 mL). The layers were separated and the aqueous layer was extracted with EtOAc (2 x 5 mL). The combined organic extracts were washed with brine (5 mL), dried over Na_2SO_4, filtered, and concentrated *in vacuo*.

The crude residue was dissolved in anhydrous THF (4 mL). DBU (0.089 mL, 0.6 mmol) was added. The mixture was stirred at rt for 8 h, then brought to reflux overnight. The reaction was quenched

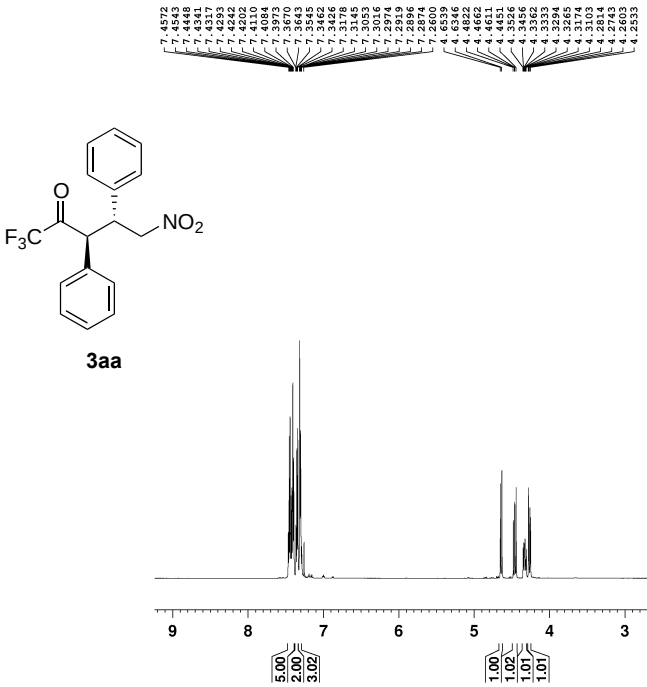
with sat. aq. NaCl (2 mL). The biphasic solution was diluted with H_2O (3 mL) and EtOAc (5 mL). The layers were separated and the aqueous layer was extracted with EtOAc (2 x 5 mL). The combined organic extracts were washed with brine (5 mL), dried over Na_2SO_4, filtered, and concentrated *in vacuo*. The crude residue was purified by column chromatography on silica gel to afford 2-*epi*-**4a** (30.5 mg, 0.105 mmol, 52% yield) as a white solid (mp: 75-76 ºC). Analytical data for 2-*epi*-**4a: ^{1}H NMR** (600 MHz, $CDCl_3$): δ 7.15-7.14 (m, 3H), 7.10-7.09 (m, 3H), 6.88-6.86 (m, 2H), 6.83-6.81 (m, 2H), 4.16-4.13 (m, 1H), 3.84 (q, J = 7.7 Hz, 1H), 3.74 (dd, J = 7.7, 3.7 Hz, 1H), 3.63, (t, J = 9.4 Hz, 1H), 3.55 (dd, J = 9.4, 6.7 Hz, 1H), 2.28 (br s, 1H); **^{13}C NMR** (150 MHz, $CDCl_3$): δ 139.0, 138.0, 129.3 (q, J_{C-F} = 278.1 Hz), 128.4, 128.3, 128.0, 127.9, 126.7, 126.5, 64.7 (q, J_{C-F} = 28.5 Hz), 50.3, 49.9, 49.4; **^{19}F NMR** (376 MHz, $CDCl_3$): δ -76.7; **IR** (thin film): 3391, 2935, 1290, 1150, 1125 cm^{-1}; **TLC** (4:1 hexanes:ethyl acetate): R_f = 0.30; **HRMS** (ESI): Calcd. for $C_{17}H_{17}F_3N$ ($[M+H]^+$): 292.1313, Found: 292.1353; **$[\alpha]_D$** +44.4 (*c* = 1.0, $CHCl_3$).

References

[1] Creary, X. *J. Org. Chem*. **1987**, *52*, 5026–5030.

[2] Simpson, A. J.; Lam, H. W. *Org. Lett*. **2013**, *15*, 2586–2589.

[3] Asano, K.; Matsubara, S. *Org. Lett*. **2012**, *14*, 1620–1623.



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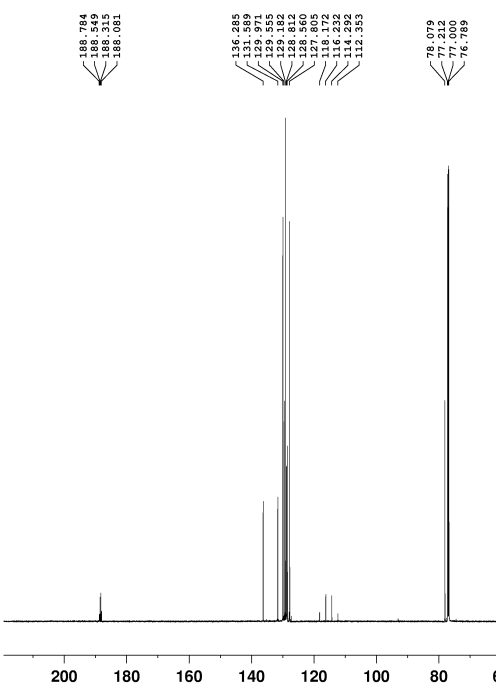

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PC                1.40
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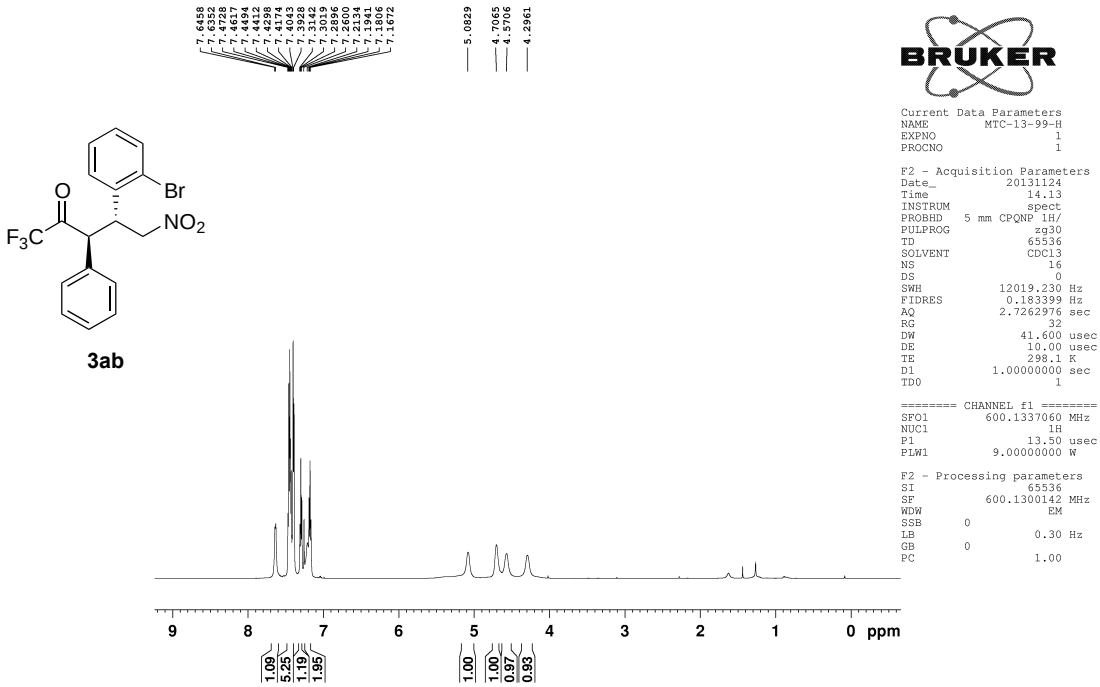

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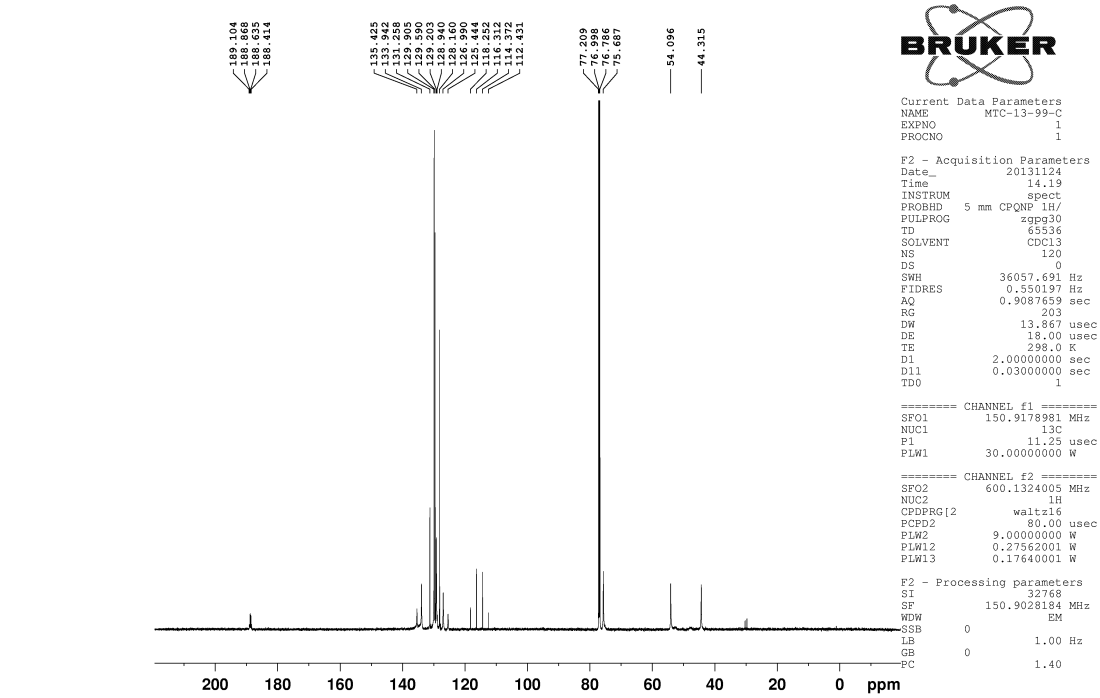
```
Current Data Parameters
NAME         MTC-13-99-H
EXPNO                  1
PROCNO                 1

F2 - Acquisition Parameters
Date_           20131124
Time               14.13
INSTRUM            spect
PROBHD   5 mm CPQNP 1H/
PULPROG            zg30
TD                65536
SOLVENT           CDCl3
NS                   16
DS                    0
SWH           12019.230 Hz
FIDRES            0.183399 Hz
AQ            2.7262976 sec
RG                   32
DW               41.600 usec
DE               10.00 usec
TE                298.1 K
D1            1.00000000 sec
TD0                    1

======== CHANNEL f1 ========
SFO1         600.1337060 MHz
NUC1                   1H
P1                 13.50 usec
PLW1          9.00000000 W

F2 - Processing parameters
SI                 65536
SF           600.1300142 MHz
WDW                   EM
SSB       0
LB                 0.30 Hz
GB        0
PC                 1.00
```



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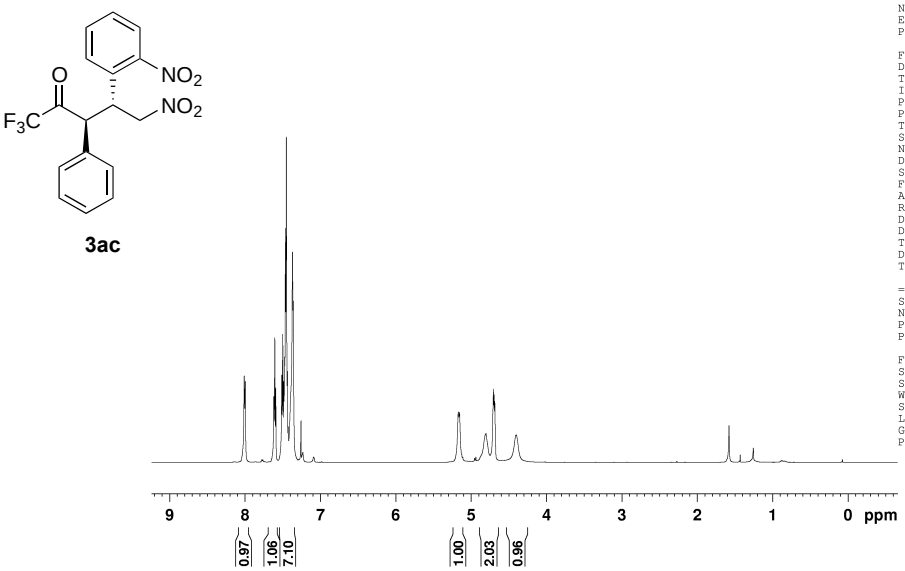
```
Current Data Parameters
NAME         MTC-13-99-C
EXPNO                  1
PROCNO                 1

F2 - Acquisition Parameters
Date_           20131124
Time               14.19
INSTRUM            spect
PROBHD   5 mm CPQNP 1H/
PULPROG          zgpg30
TD                65536
SOLVENT           CDCl3
NS                  120
DS                    0
SWH           36057.691 Hz
FIDRES            0.550197 Hz
AQ            0.9087659 sec
RG                  203
DW               13.867 usec
DE                18.00 usec
TE                298.0 K
D1            2.00000000 sec
D11           0.03000000 sec
TD0                    1

======== CHANNEL f1 ========
SFO1         150.9178981 MHz
NUC1                   13C
P1                 11.25 usec
PLW1         30.00000000 W

======== CHANNEL f2 ========
SFO2         600.1324005 MHz
NUC2                   1H
CPDPRG[2          waltz16
PCPD2              80.00 usec
PLW2          9.00000000 W
PLW12         0.27562001 W
PLW13         0.17640001 W

F2 - Processing parameters
SI                 32768
SF           150.9028184 MHz
WDW                   EM
SSB       0
LB                 1.00 Hz
GB        0
PC                 1.40
```



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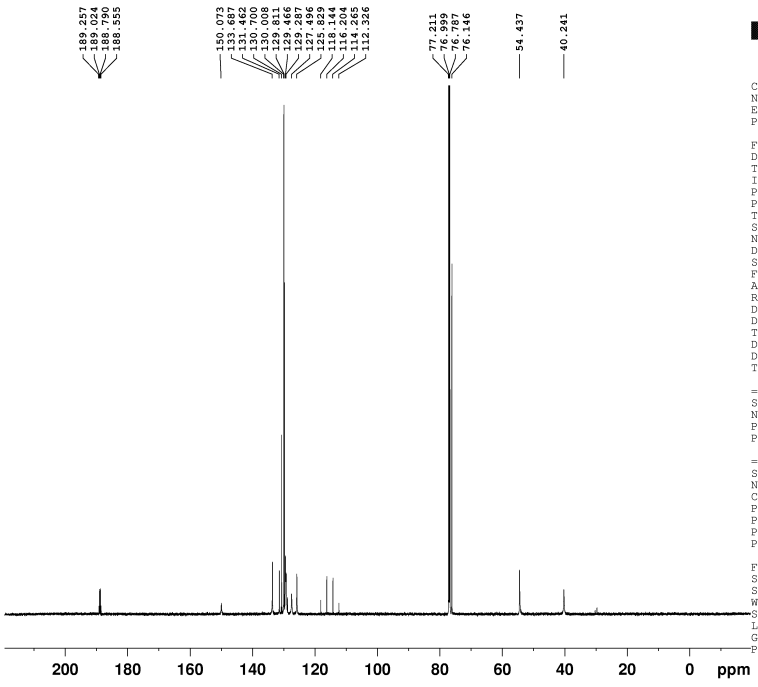
```
Current Data Parameters
NAME         MTC-13-87-H
EXPNO                  1
PROCNO                 1

F2 - Acquisition Parameters
Date_           20131118
Time                9.26
INSTRUM            spect
PROBHD   5 mm CPQNP 1H/
PULPROG             zg30
TD                 65536
SOLVENT            CDCl3
NS                    16
DS                     0
SWH           12019.230 Hz
FIDRES            0.183399 Hz
AQ            2.7262976 sec
RG                    32
DW               41.600 usec
DE               10.00 usec
TE                298.0 K
D1            1.00000000 sec
TD0                    1

======== CHANNEL f1 ========
SFO1         600.1337060 MHz
NUC1                   1H
P1                  13.50 usec
PLW1          9.00000000 W

F2 - Processing parameters
SI                 65536
SF           600.1300131 MHz
WDW                   EM
SSB       0
LB                 0.30 Hz
GB        0
PC                 1.00
```



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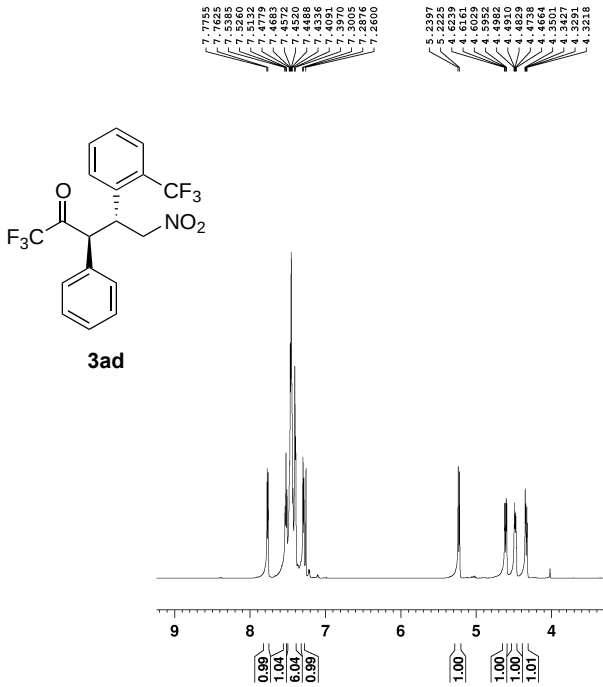
```
Current Data Parameters
NAME         MTC-13-87-C
EXPNO                  1
PROCNO                 1

F2 - Acquisition Parameters
Date_           20131118
Time                9.30
INSTRUM            spect
PROBHD   5 mm CPQNP 1H/
PULPROG          zgpg30
TD                 65536
SOLVENT            CDCl3
NS                   120
DS                     0
SWH           36057.691 Hz
FIDRES            0.550197 Hz
AQ            0.9087659 sec
RG                   203
DW               13.867 usec
DE                18.00 usec
TE                298.0 K
D1            2.00000000 sec
D11           0.03000000 sec
TD0                    1

======== CHANNEL f1 ========
SFO1         150.9178981 MHz
NUC1                   13C
P1                  11.25 usec
PLW1         30.00000000 W

======== CHANNEL f2 ========
SFO2         600.1324005 MHz
NUC2                   1H
CPDPRG[2          waltz16
PCPD2                80.00 usec
PLW2          9.00000000 W
PLW12         0.27562001 W
PLW13         0.17640001 W

F2 - Processing parameters
SI                 32768
SF           150.9028173 MHz
WDW                   EM
SSB       0
LB                 1.00 Hz
GB        0
PC                 1.40
```

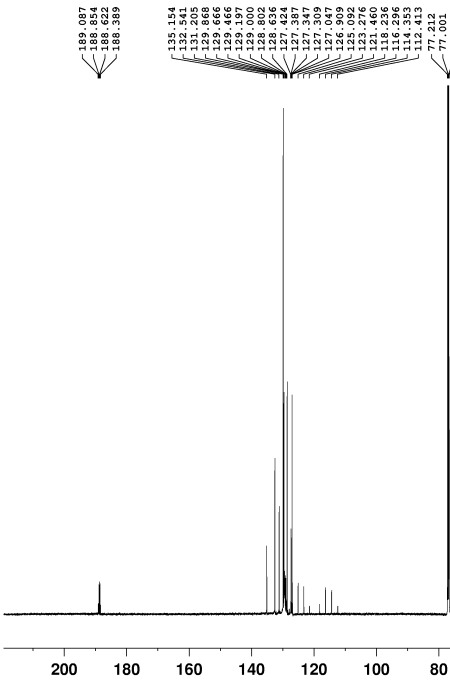


```
Current Data Parameters
NAME         MTC-13-95-H
EXPNO                  1
PROCNO                 1

F2 - Acquisition Parameters
Date_           20131121
Time               17.11
INSTRUM            spect
PROBHD   5 mm CPQNP 1H/
PULPROG            zg30
TD                65536
SOLVENT           CDCl3
NS                   16
DS                    0
SWH           12019.230 Hz
FIDRES            0.183399 Hz
AQ            2.7262976 sec
RG                   32
DW               41.600 usec
DE               10.00 usec
TE                298.0 K
D1           1.00000000 sec
TD0                    1

======== CHANNEL f1 ========
SFO1         600.1337060 MHz
NUC1                   1H
P1                 13.50 usec
PLW1          9.00000000 W

F2 - Processing parameters
SI                 65536
SF           600.1300122 MHz
WDW                   EM
SSB       0
LB                 0.30 Hz
GB        0
PC                 1.00
```



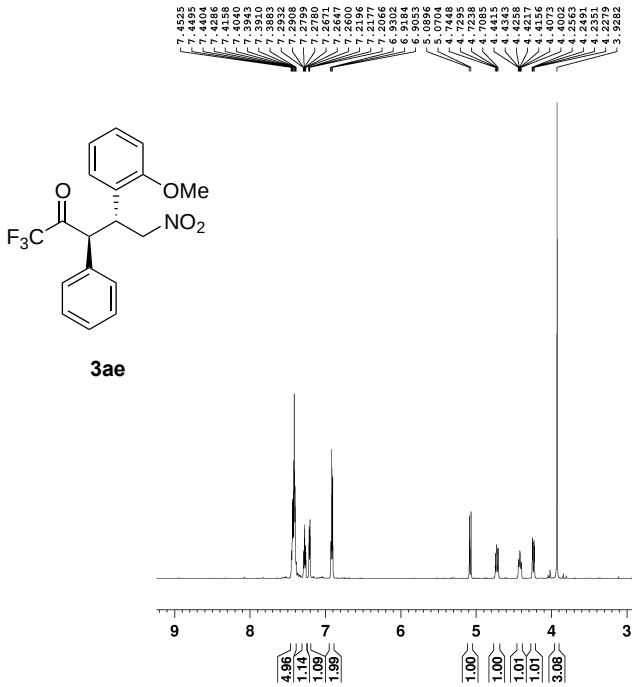
```
Current Data Parameters
NAME         MTC-13-95-C
EXPNO                  1
PROCNO                 1

F2 - Acquisition Parameters
Date_           20131121
Time               17.12
INSTRUM            spect
PROBHD   5 mm CPQNP 1H/
PULPROG          zgpg30
TD                65536
SOLVENT           CDCl3
NS                  160
DS                    0
SWH           36057.691 Hz
FIDRES            0.550197 Hz
AQ            0.9087659 sec
RG                  203
DW               13.867 usec
DE                18.00 usec
TE                298.0 K
D1           2.00000000 sec
D11          0.03000000 sec
TD0                    1

======== CHANNEL f1 ========
SFO1         150.9178981 MHz
NUC1                   13C
P1                 11.25 usec
PLW1         30.00000000 W

======== CHANNEL f2 ========
SFO2         600.1324005 MHz
NUC2                   1H
CPDPRG[2          waltz16
PCPD2                80.00 usec
PLW2          9.00000000 W
PLW12         0.27562001 W
PLW13         0.17640001 W

F2 - Processing parameters
SI                 32768
SF           150.9028140 MHz
WDW                   EM
SSB       0
LB                 1.00 Hz
GB        0
PC                 1.40
```

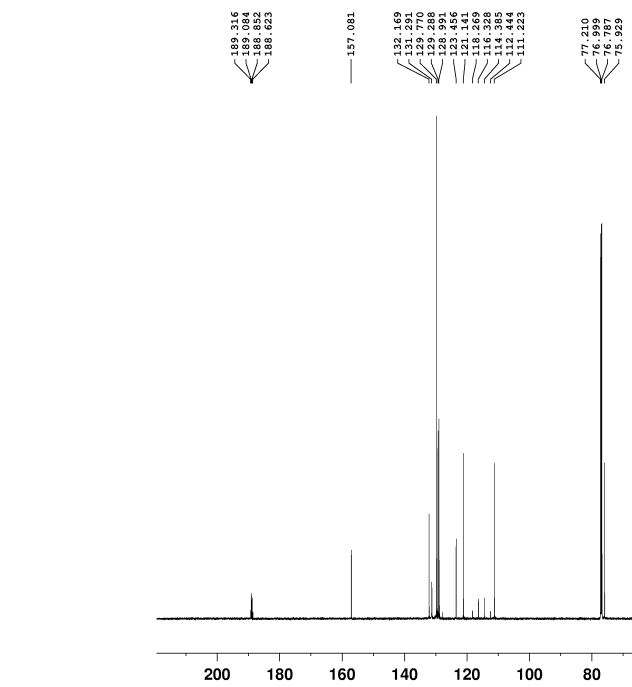


Current Data Parameters
NAME MTC-13-38-H
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20131016
Time 16.43
INSTRUM spect
PROBHD 5 mm CPQNP 1H/
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 0
SWH 12019.230 Hz
FIDRES 0.183399 Hz
AQ 2.7262976 sec
RG 32
DW 41.600 usec
DE 10.00 usec
TE 298.0 K
D1 1.00000000 sec
TD0 1

======== CHANNEL f1 ========
SFO1 600.1337060 MHz
NUC1 1H
P1 13.50 usec
PLW1 9.00000000 W

F2 - Processing parameters
SI 65536
SF 600.1300136 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00



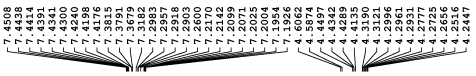
Current Data Parameters
NAME MTC-13-38-C
EXPNO 1
PROCNO 1

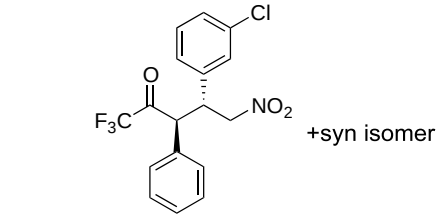
F2 - Acquisition Parameters
Date_ 20131016
Time 16.39
INSTRUM spect
PROBHD 5 mm CPQNP 1H/
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 50
DS 0
SWH 36057.691 Hz
FIDRES 0.550197 Hz
AQ 0.9087659 sec
RG 203
DW 13.867 usec
DE 18.00 usec
TE 298.0 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1

======== CHANNEL f1 ========
SFO1 150.9178981 MHz
NUC1 13C
P1 11.25 usec
PLW1 30.00000000 W

======== CHANNEL f2 ========
SFO2 600.1324005 MHz
NUC2 1H
CPDPRG[2 waltz16
PCPD2 80.00 usec
PLW2 9.00000000 W
PLW12 0.27562001 W
PLW13 0.17640001 W

F2 - Processing parameters
SI 32768
SF 150.9028189 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40





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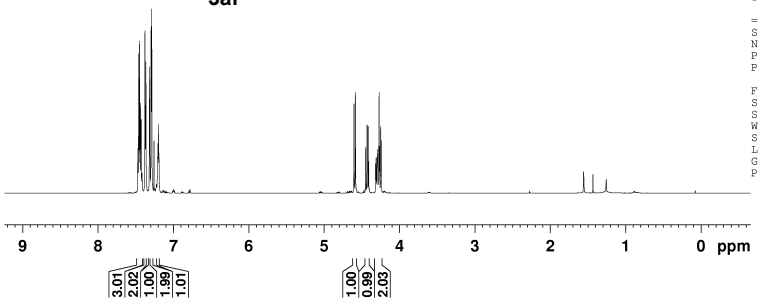

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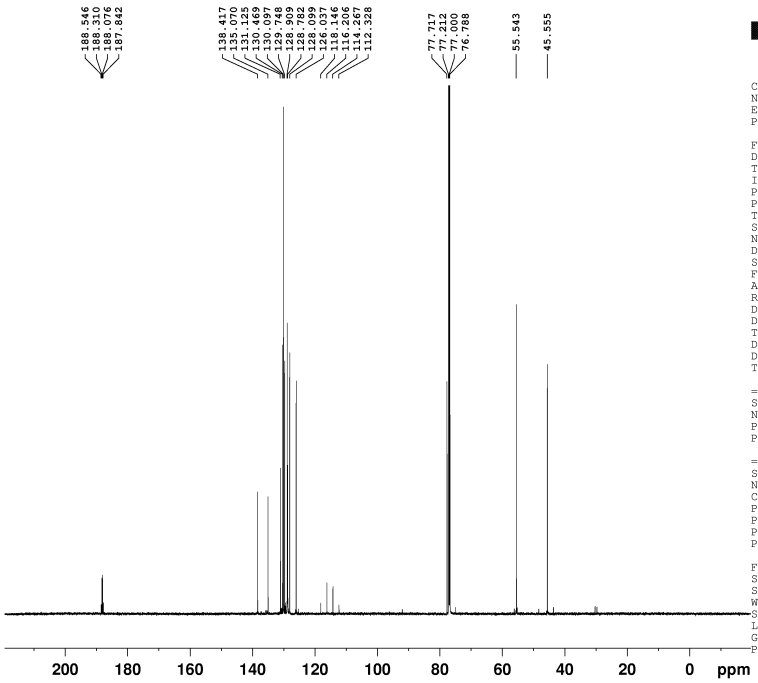
```
Current Data Parameters
NAME         MTC-13-85-H
EXPNO                  1
PROCNO                 1

F2 - Acquisition Parameters
Date_           20131116
Time                6.55
INSTRUM            spect
PROBHD   5 mm CPQNP 1H/
PULPROG             zg30
TD                 65536
SOLVENT            CDCl3
NS                    16
DS                     0
SWH            12019.230 Hz
FIDRES             0.183399 Hz
AQ             2.7262976 sec
RG                    57
DW                41.600 usec
DE                10.00 usec
TE                298.0 K
D1             1.00000000 sec
TD0                    1

======== CHANNEL f1 ========
SFO1          600.1337060 MHz
NUC1                  1H
P1                 13.50 usec
PLW1           9.00000000 W

F2 - Processing parameters
SI                 65536
SF            600.1300139 MHz
WDW                   EM
SSB       0
LB                 0.30 Hz
GB        0
PC                 1.00
```



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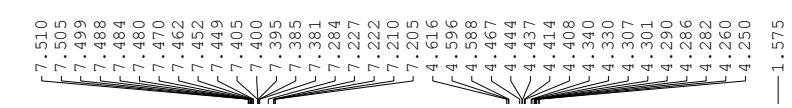
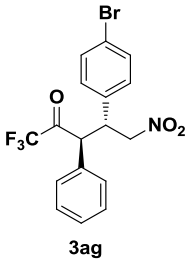
```
Current Data Parameters
NAME         MTC-13-85-C
EXPNO                  1
PROCNO                 1

F2 - Acquisition Parameters
Date_           20131116
Time                6.58
INSTRUM            spect
PROBHD   5 mm CPQNP 1H/
PULPROG          zgpg30
TD                 65536
SOLVENT            CDCl3
NS                    80
DS                     0
SWH            36057.691 Hz
FIDRES             0.550197 Hz
AQ             0.9087659 sec
RG                   203
DW                13.867 usec
DE                18.00 usec
TE                298.0 K
D1             2.00000000 sec
D11            0.03000000 sec
TD0                    1

======== CHANNEL f1 ========
SFO1          150.9178981 MHz
NUC1                  13C
P1                 11.25 usec
PLW1          30.00000000 W

======== CHANNEL f2 ========
SFO2          600.1324005 MHz
NUC2                  1H
CPDPRG[2          waltz16
PCPD2              80.00 usec
PLW2           9.00000000 W
PLW12          0.27562001 W
PLW13          0.17640001 W

F2 - Processing parameters
SI                 32768
SF            150.9028156 MHz
WDW                   EM
SSB       0
LB                 1.00 Hz
GB        0
PC                 1.40
```





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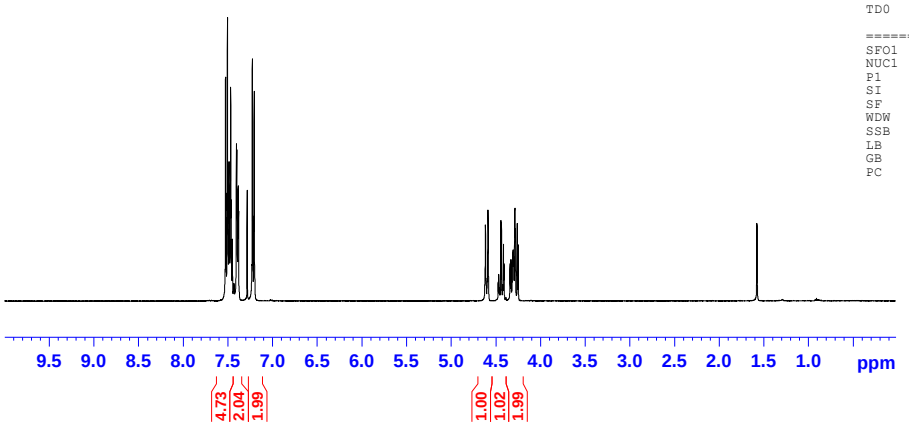
BRUKER

```
NAME          QX1250C-crystall
EXPNO                        1
PROCNO                       1
Date_             20131219
Time                     11.52
INSTRUM                  spect
PROBHD    5 mm PABBO BB-
PULPROG                  zg30
TD                      65536
SOLVENT                 CDCl3
NS                           9
DS                           2
SWH                 8012.820 Hz
FIDRES              0.122266 Hz
AQ                  4.0894966 sec
RG                     134.63
DW                     62.400 usec
DE                      6.50 usec
TE                     295.7 K
D1                  1.00000000 sec
TD0                          1

======== CHANNEL f1 ========
SFO1           399.9824700 MHz
NUC1                      1H
P1                     12.38 usec
SI                     65536
SF              399.9800000 MHz
WDW                       EM
SSB                        0
LB                      0.30 Hz
GB                         0
PC                      1.00
```



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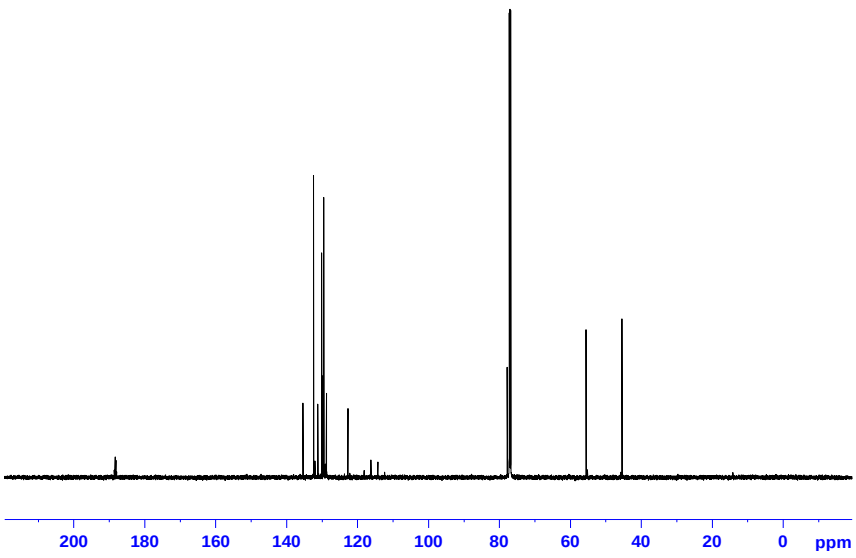
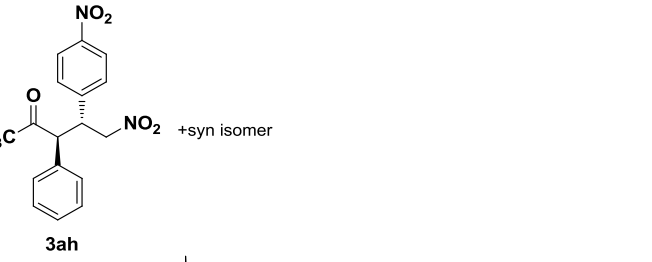
```
NAME              QX1250C
EXPNO                   3
PROCNO                  1
Date_            20131007
Time                20.07
INSTRUM             spect
PROBHD    5 mm CPQCI 1H/
PULPROG          zgpg30
TD                  65536
SOLVENT             CDCl3
NS                     70
DS                      4
SWH             36057.691 Hz
FIDRES          0.550197 Hz
AQ              0.9088159 sec
RG                    203
DW                 13.867 usec
DE                 18.00 usec
TE                 298.0 K
D1              2.00000000 sec
D11             0.03000000 sec
TD0                     1

======== CHANNEL f1 ========
SFO1          150.9178981 MHz
NUC1                    13C
P1                 11.00 usec
SI                  32768
SF            150.9028090 MHz
WDW                     EM
SSB                      0
LB                    1.00 Hz
GB                       0
PC                    1.40
```





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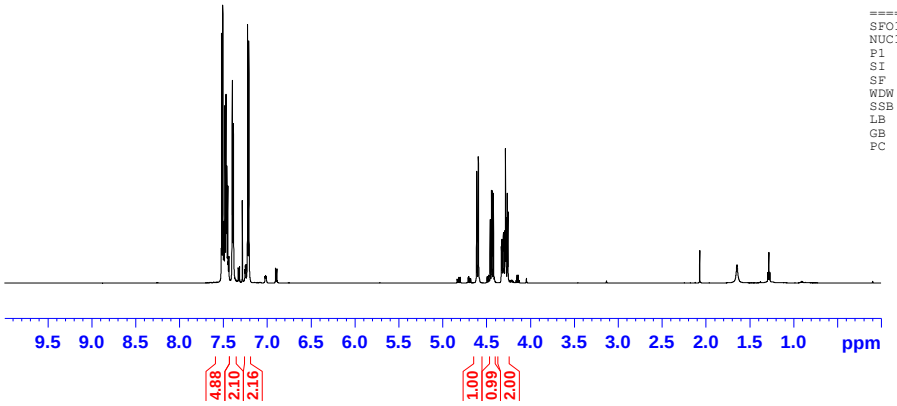
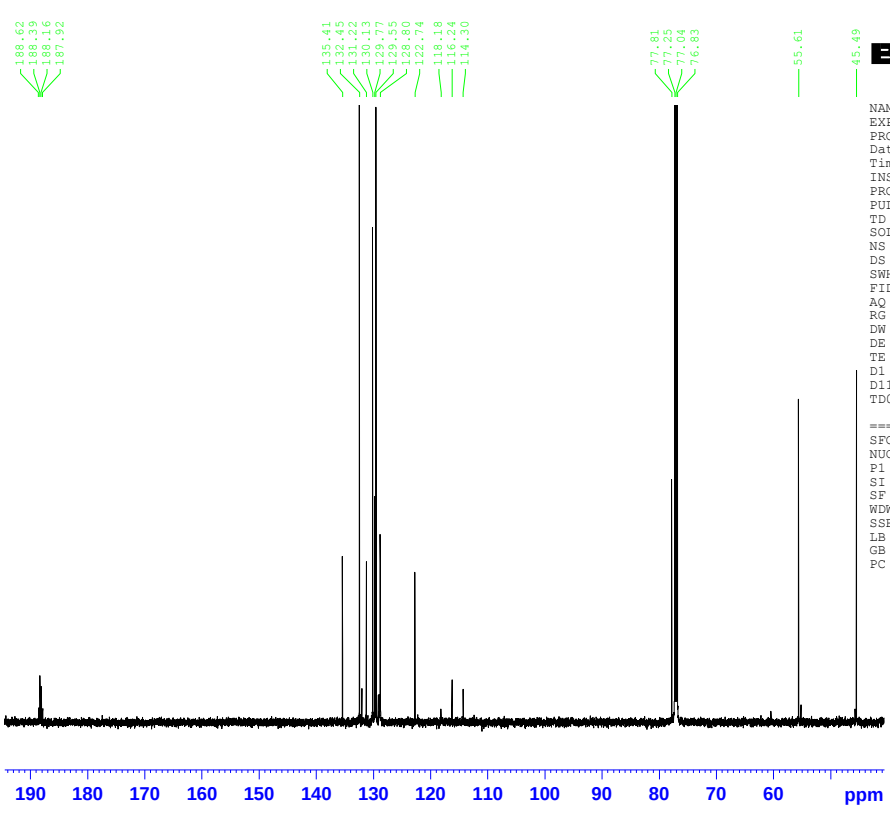
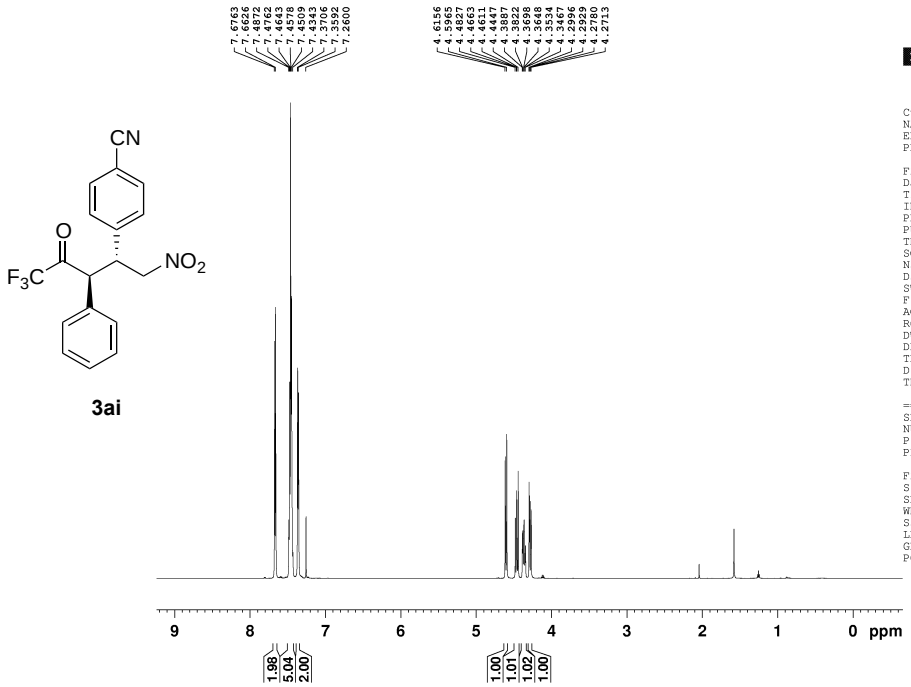
```
NAME                 QX1250D
EXPNO                      1
PROCNO                     1
Date_               20131007
Time                  20.18
INSTRUM               spect
PROBHD     5 mm CPQCI 1H/
PULPROG                zg30
TD                    65536
SOLVENT               CDCl3
NS                       12
DS                        2
SWH               12019.230 Hz
FIDRES             0.183399 Hz
AQ                 2.7263477 sec
RG                       32
DW                   41.600 usec
DE                   10.00 usec
TE                   298.0 K
D1                1.00000000 sec
TD0                       1

======== CHANNEL f1 ========
SFO1            600.1337060 MHz
NUC1                    1H
P1                   11.75 usec
SI                    65536
SF              600.1300000 MHz
WDW                     EM
SSB                      0
LB                    0.30 Hz
GB                       0
PC                    1.00
```



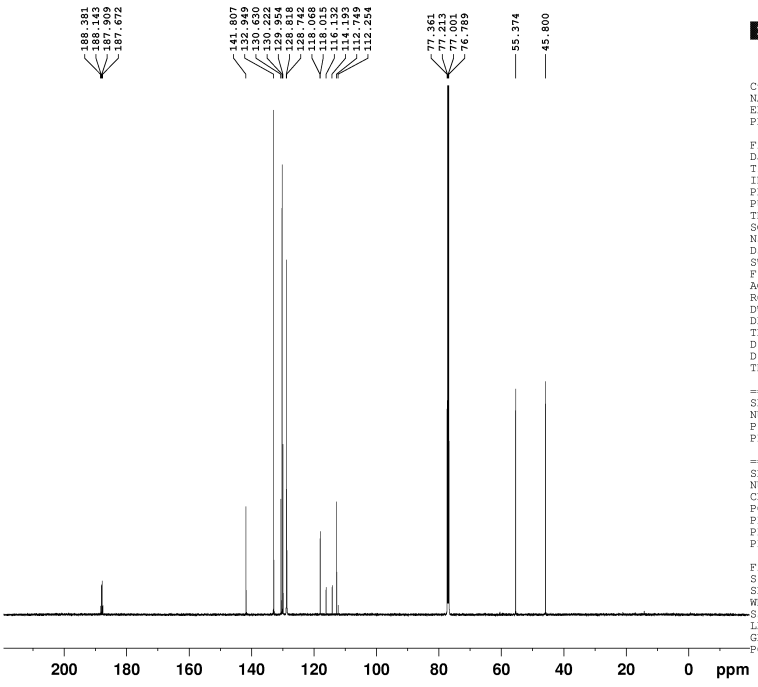


```
NAME                 QX1250D
EXPNO                      3
PROCNO                     1
Date_               20131007
Time                  20.13
INSTRUM               spect
PROBHD     5 mm CPQCI 1H/
PULPROG             zgpg30
TD                    65536
SOLVENT               CDCl3
NS                       76
DS                        4
SWH               36057.691 Hz
FIDRES             0.550197 Hz
AQ                 0.9088159 sec
RG                      203
DW                   13.867 usec
DE                   18.00 usec
TE                   298.0 K
D1                2.00000000 sec
D11               0.03000000 sec
TD0                       1

======== CHANNEL f1 ========
SFO1            150.9178981 MHz
NUC1                    13C
P1                   11.00 usec
SI                    32768
SF              150.9028090 MHz
WDW                     EM
SSB                      0
LB                    1.00 Hz
GB                       0
PC                    1.40
```



BRUKER

```
Current Data Parameters
NAME        MTC-13-57-H2
EXPNO                  1
PROCNO                 1

F2 - Acquisition Parameters
Date_           20131116
Time                7.10
INSTRUM            spect
PROBHD   5 mm CPQNP 1H/
PULPROG             zg30
TD                 65536
SOLVENT            CDCl3
NS                    16
DS                     0
SWH            12019.230 Hz
FIDRES             0.183399 Hz
AQ             2.7262976 sec
RG                    57
DW                41.600 usec
DE                10.00 usec
TE                298.0 K
D1             1.00000000 sec
TD0                    1

======== CHANNEL f1 ========
SFO1          600.1337060 MHz
NUC1                  1H
P1                 13.50 usec
PLW1           9.00000000 W

F2 - Processing parameters
SI                 65536
SF            600.1300141 MHz
WDW                   EM
SSB       0
LB                 0.30 Hz
GB        0
PC                 1.00
```



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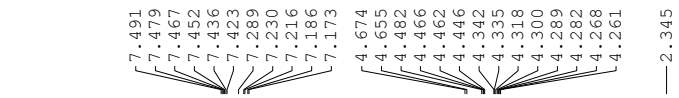
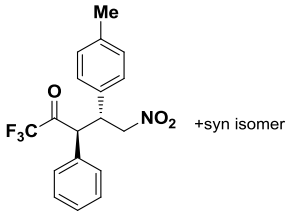
```
Current Data Parameters
NAME        MTC-13-57-C2
EXPNO                  1
PROCNO                 1

F2 - Acquisition Parameters
Date_           20131116
Time                7.12
INSTRUM            spect
PROBHD   5 mm CPQNP 1H/
PULPROG           zgpg30
TD                 65536
SOLVENT            CDCl3
NS                   100
DS                     0
SWH            36057.691 Hz
FIDRES             0.550197 Hz
AQ             0.9087659 sec
RG                   203
DW                13.867 usec
DE                18.00 usec
TE                298.0 K
D1             2.00000000 sec
D11            0.03000000 sec
TD0                    1

======== CHANNEL f1 ========
SFO1          150.9178981 MHz
NUC1                  13C
P1                 11.25 usec
PLW1          30.00000000 W

======== CHANNEL f2 ========
SFO2          600.1324005 MHz
NUC2                  1H
CPDPRG[2          waltz16
PCPD2              80.00 usec
PLW2           9.00000000 W
PLW12          0.27562001 W
PLW13          0.17640001 W

F2 - Processing parameters
SI                 32768
SF            150.9028168 MHz
WDW                   EM
SSB       0
LB                 1.00 Hz
GB        0
PC                 1.40
```





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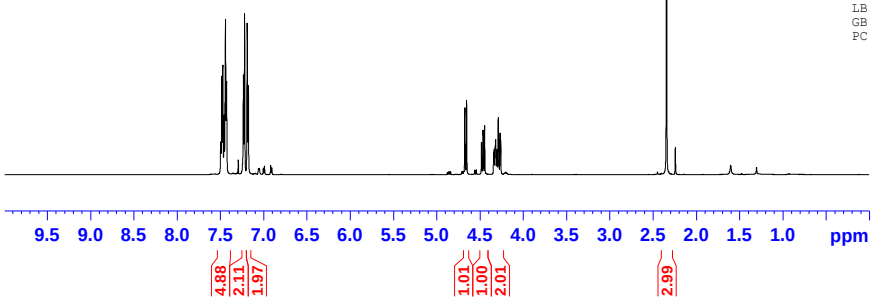
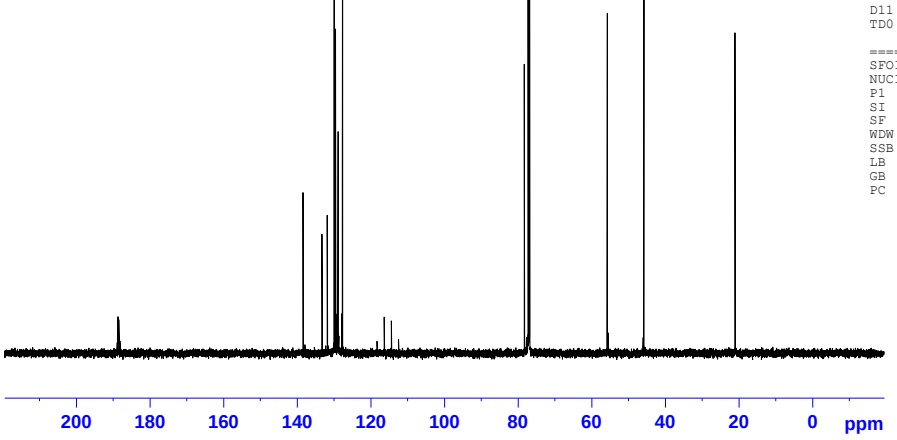
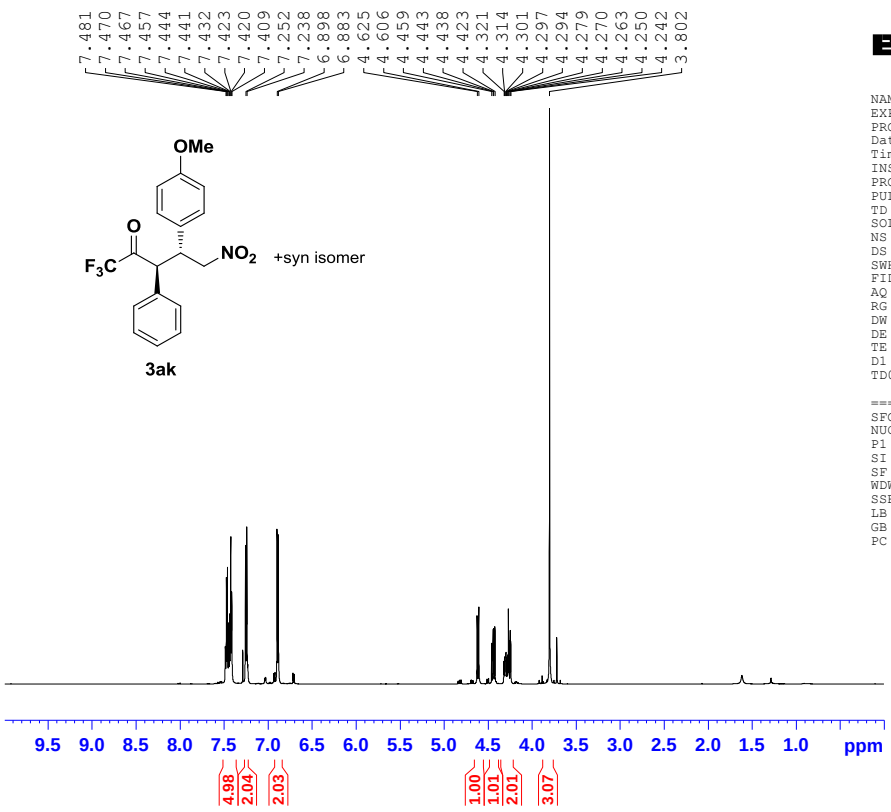
```
NAME                  QX1250A
EXPNO                       1
PROCNO                      1
Date_                20131007
Time                    19.06
INSTRUM                 spect
PROBHD   5 mm CPQCI 1H/
PULPROG                 zg30
TD                      65536
SOLVENT                 CDCl3
NS                         16
DS                          2
SWH                12019.230 Hz
FIDRES              0.183399 Hz
AQ                  2.7263477 sec
RG                       11.3
DW                     41.600 usec
DE                     10.00 usec
TE                     295.8 K
D1                 1.00000000 sec
TD0                         1

======== CHANNEL f1 ========
SFO1            600.1337060 MHz
NUC1                      1H
P1                     11.75 usec
SI                      65536
SF              600.1300000 MHz
WDW                        EM
SSB                         0
LB                       0.30 Hz
GB                          0
PC                       1.00
```



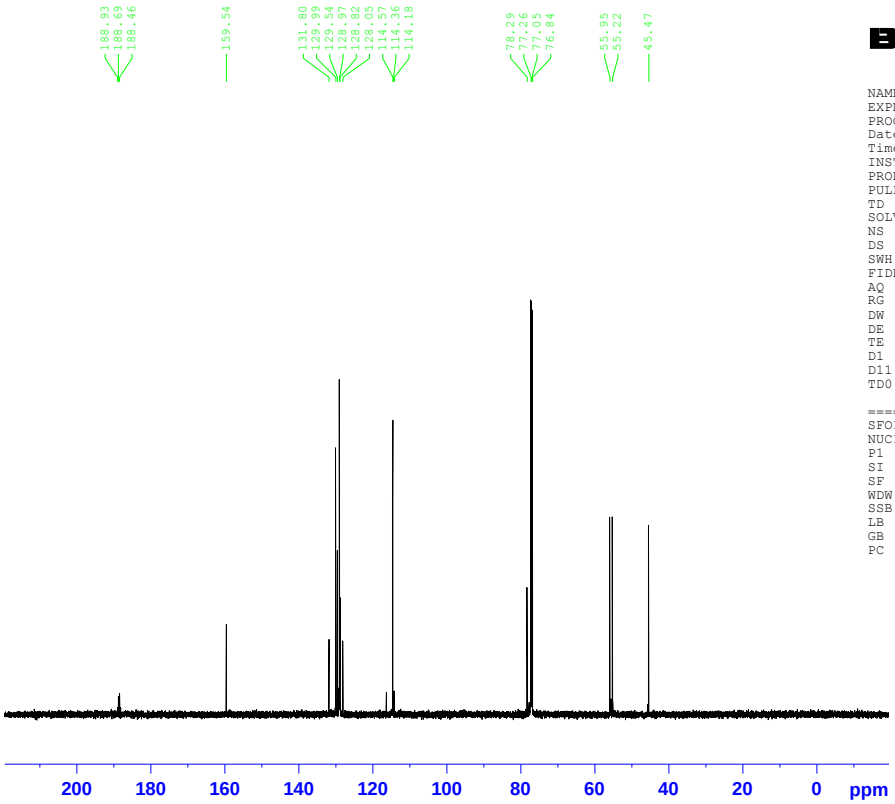
```
NAME                  QX1250A
EXPNO                       8
PROCNO                      1
Date_                20131007
Time                    19.49
INSTRUM                 spect
PROBHD   5 mm CPQCI 1H/
PULPROG              zgpg30
TD                      65536
SOLVENT                 CDCl3
NS                         31
DS                          4
SWH                36057.691 Hz
FIDRES              0.550197 Hz
AQ                  0.9088159 sec
RG                        203
DW                     13.867 usec
DE                     18.00 usec
TE                     298.0 K
D1                 2.00000000 sec
D11                0.03000000 sec
TD0                         1

======== CHANNEL f1 ========
SFO1            150.9178981 MHz
NUC1                      13C
P1                     11.00 usec
SI                      32768
SF              150.9028090 MHz
WDW                        EM
SSB                         0
LB                       1.00 Hz
GB                          0
PC                       1.40
```





BRUKER

```
NAME                QX1250B
EXPNO                     2
PROCNO                    1
Date_              20131007
Time                  19.57
INSTRUM               spect
PROBHD    5 mm CPQCI 1H/
PULPROG                zg30
TD                    65536
SOLVENT               CDCl3
NS                       16
DS                        2
SWH               12019.230 Hz
FIDRES                0.183399 Hz
AQ                2.7263477 sec
RG                       32
DW                   41.600 usec
DE                   10.00 usec
TE                   298.0 K
D1                1.00000000 sec
TD0                       1

======== CHANNEL f1 ========
SFO1            600.1337060 MHz
NUC1                      1H
P1                   11.75 usec
SI                    65536
SF              600.1300000 MHz
WDW                      EM
SSB                       0
LB                    0.30 Hz
GB                        0
PC                    1.00
```



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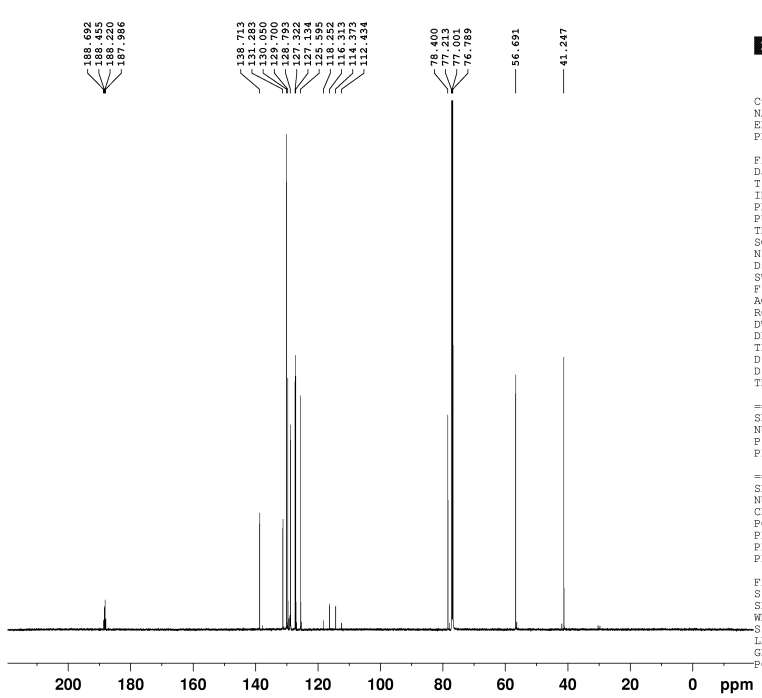
```
NAME                QX1250B
EXPNO                     3
PROCNO                    1
Date_              20131007
Time                  19.54
INSTRUM               spect
PROBHD    5 mm CPQCI 1H/
PULPROG              zgpg30
TD                    65536
SOLVENT               CDCl3
NS                       27
DS                        4
SWH               36057.691 Hz
FIDRES                0.550197 Hz
AQ                0.9088159 sec
RG                      203
DW                   13.867 usec
DE                   18.00 usec
TE                   298.0 K
D1                2.00000000 sec
D11               0.03000000 sec
TD0                       1

======== CHANNEL f1 ========
SFO1            150.9178981 MHz
NUC1                      13C
P1                   11.00 usec
SI                    32768
SF              150.9028090 MHz
WDW                      EM
SSB                       0
LB                    1.00 Hz
GB                        0
PC                    1.40
```

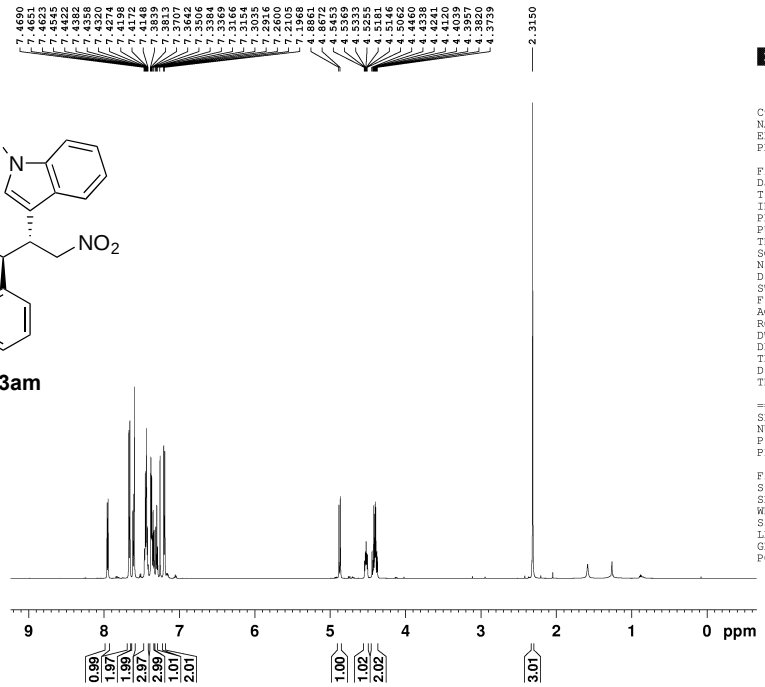
```
Current Data Parameters
NAME         MTC-13-86-C
EXPNO                  1
PROCNO                 1

F2 - Acquisition Parameters
Date_           20131116
Time                7.05
INSTRUM            spect
PROBHD   5 mm CPQNP 1H/
PULPROG          zgpg30
TD                65536
SOLVENT           CDCl3
NS                   99
DS                    0
SWH           36057.691 Hz
FIDRES         0.550197 Hz
AQ            0.9087659 sec
RG                  203
DW               13.867 usec
DE               18.00 usec
TE               298.0 K
D1            2.00000000 sec
D11           0.03000000 sec
TD0                    1

======== CHANNEL f1 ========
SFO1         150.9178981 MHz
NUC1                 13C
P1                 11.25 usec
PLW1         30.00000000 W

======== CHANNEL f2 ========
SFO2         600.1324005 MHz
NUC2                 1H
CPDPRG[2          waltz16
PCPD2              80.00 usec
PLW2          9.00000000 W
PLW12         0.27562001 W
PLW13         0.17640001 W

F2 - Processing parameters
SI                 32768
SF           150.9028151 MHz
WDW                   EM
SSB       0
LB                 1.00 Hz
GB        0
PC                 1.40
```

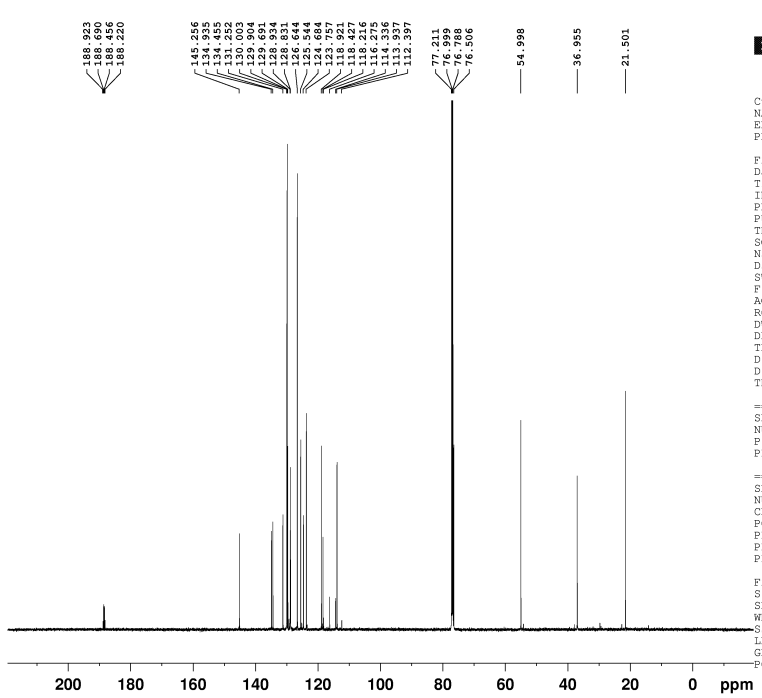


```
Current Data Parameters
NAME         MTC-13-89-H
EXPNO                  1
PROCNO                 1

F2 - Acquisition Parameters
Date_           20131118
Time               14.50
INSTRUM            spect
PROBHD   5 mm CPQNP 1H/
PULPROG            zg30
TD                65536
SOLVENT           CDCl3
NS                   16
DS                    0
SWH           12019.230 Hz
FIDRES         0.183399 Hz
AQ            2.7262976 sec
RG                   32
DW               41.600 usec
DE               10.00 usec
TE               298.0 K
D1            1.00000000 sec
TD0                    1

======== CHANNEL f1 ========
SFO1         600.1337060 MHz
NUC1                 1H
P1                 13.50 usec
PLW1          9.00000000 W

F2 - Processing parameters
SI                 65536
SF           600.1300140 MHz
WDW                   EM
SSB       0
LB                 0.30 Hz
GB        0
PC                 1.00
```



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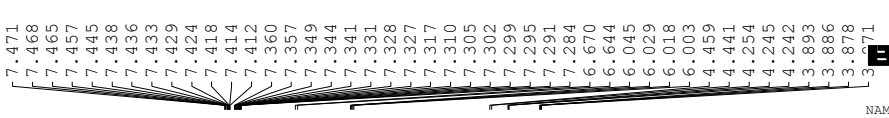
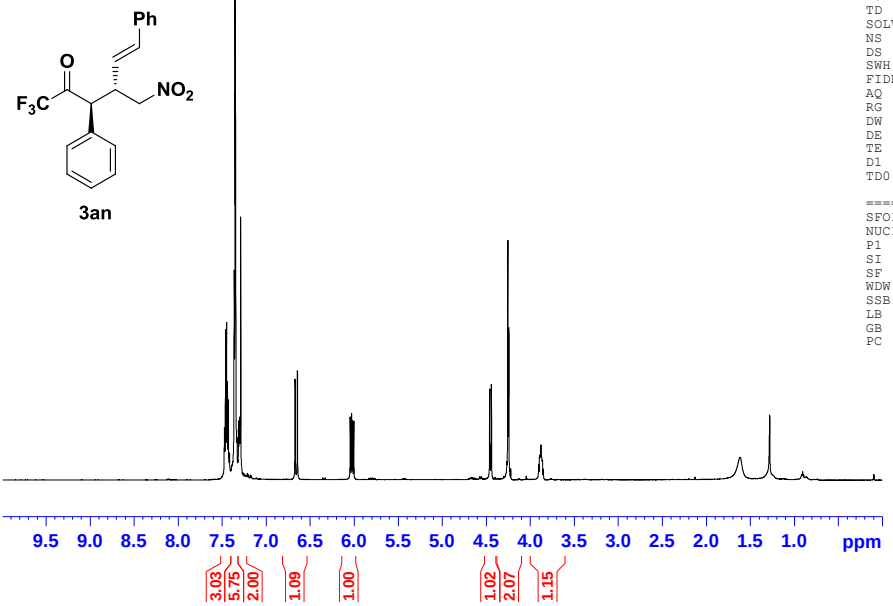
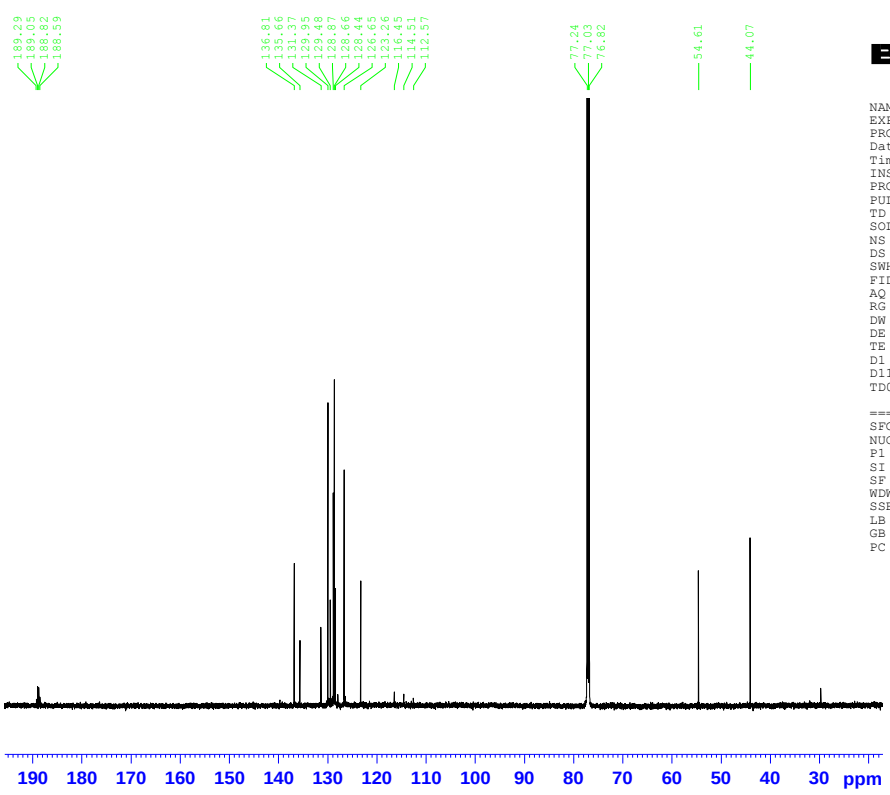
```
Current Data Parameters
NAME         MTC-13-89-C
EXPNO                  1
PROCNO                 1

F2 - Acquisition Parameters
Date_           20131118
Time               14.53
INSTRUM            spect
PROBHD   5 mm CPQNP 1H/
PULPROG          zgpg30
TD                65536
SOLVENT           CDCl3
NS                  150
DS                    0
SWH           36057.691 Hz
FIDRES         0.550197 Hz
AQ            0.9087659 sec
RG                  203
DW             13.867 usec
DE                18.00 usec
TE                298.0 K
D1           2.00000000 sec
D11          0.03000000 sec
TD0                   1

======== CHANNEL f1 ========
SFO1        150.9178981 MHz
NUC1               13C
P1                11.25 usec
PLW1        30.00000000 W

======== CHANNEL f2 ========
SFO2        600.1324005 MHz
NUC2               1H
CPDPRG[2         waltz16
PCPD2             80.00 usec
PLW2         9.00000000 W
PLW12        0.27562001 W
PLW13        0.17640001 W

F2 - Processing parameters
SI                 32768
SF          150.9028173 MHz
WDW                   EM
SSB       0
LB                 1.00 Hz
GB        0
PC                 1.40
```



BRUKER

```
NAME              QX1268A1
EXPNO                    4
PROCNO                   1
Date_             20131021
Time                11.30
INSTRUM             spect
PROBHD    5 mm CPQNP 1H/
PULPROG               zg30
TD                   65536
SOLVENT              CDCl3
NS                      12
DS                       2
SWH              12019.230 Hz
FIDRES            0.183399 Hz
AQ                2.7263477 sec
RG                      57
DW                  41.600 usec
DE                  10.00 usec
TE                  298.0 K
D1               1.00000000 sec
TD0                      1

======== CHANNEL f1 ========
SFO1            600.1337060 MHz
NUC1                    1H
P1                   13.50 usec
SI                   65536
SF              600.1300000 MHz
WDW                     EM
SSB                      0
LB                    0.30 Hz
GB                       0
PC                    1.00
```





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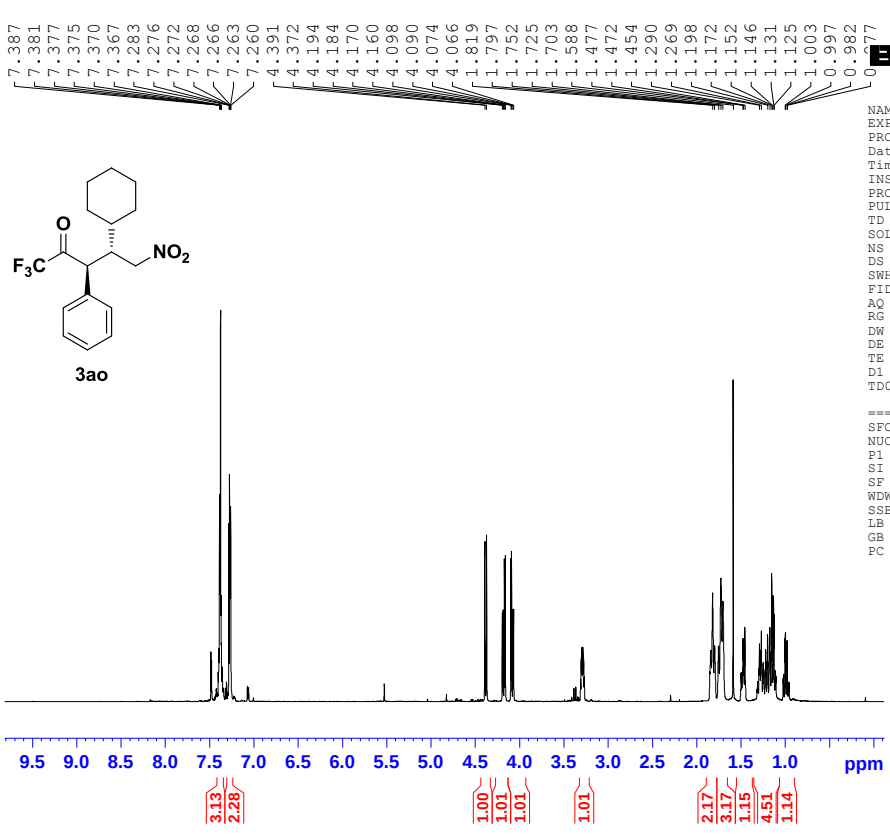
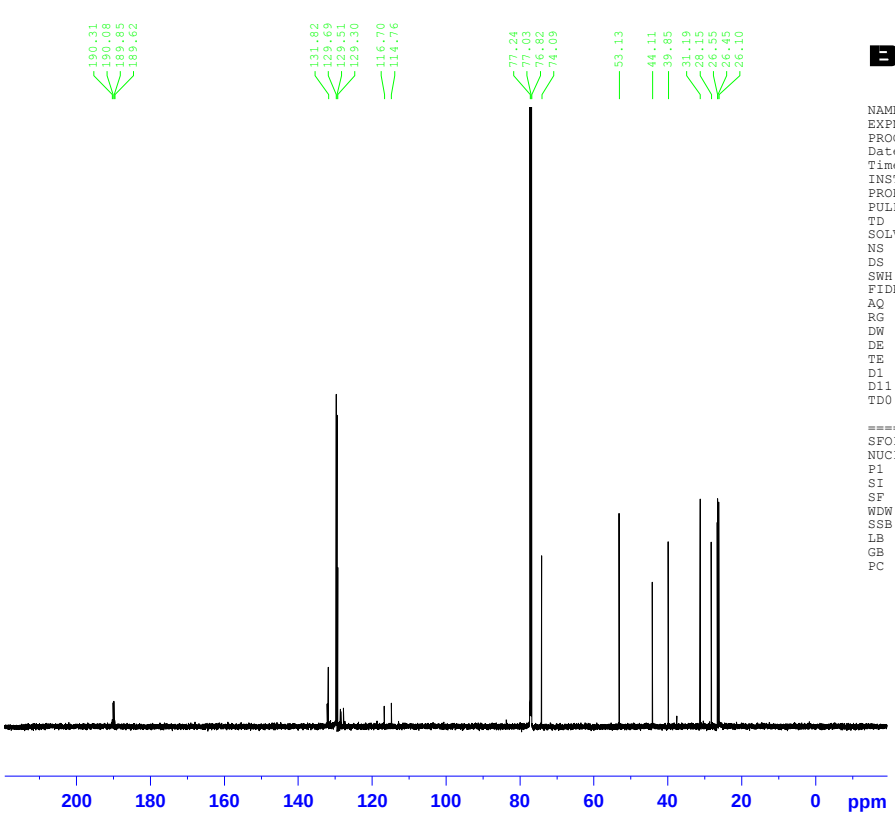
```
NAME                QX1268A1
EXPNO                      3
PROCNO                     1
Date_               20131021
Time                   11.34
INSTRUM                spect
PROBHD    5 mm CPQNP 1H/
PULPROG                zgpg30
TD                      65536
SOLVENT                 CDCl3
NS                        126
DS                          4
SWH                36057.691 Hz
FIDRES              0.550197 Hz
AQ                  0.9088159 sec
RG                        203
DW                     13.867 usec
DE                     18.00 usec
TE                     298.0 K
D1                 2.00000000 sec
D11                0.03000000 sec
TD0                        1

======== CHANNEL f1 ========
SFO1             150.9178981 MHz
NUC1                     13C
P1                     11.25 usec
SI                      32768
SF               150.9028090 MHz
WDW                       EM
SSB                        0
LB                      1.00 Hz
GB                         0
PC                      1.40
```

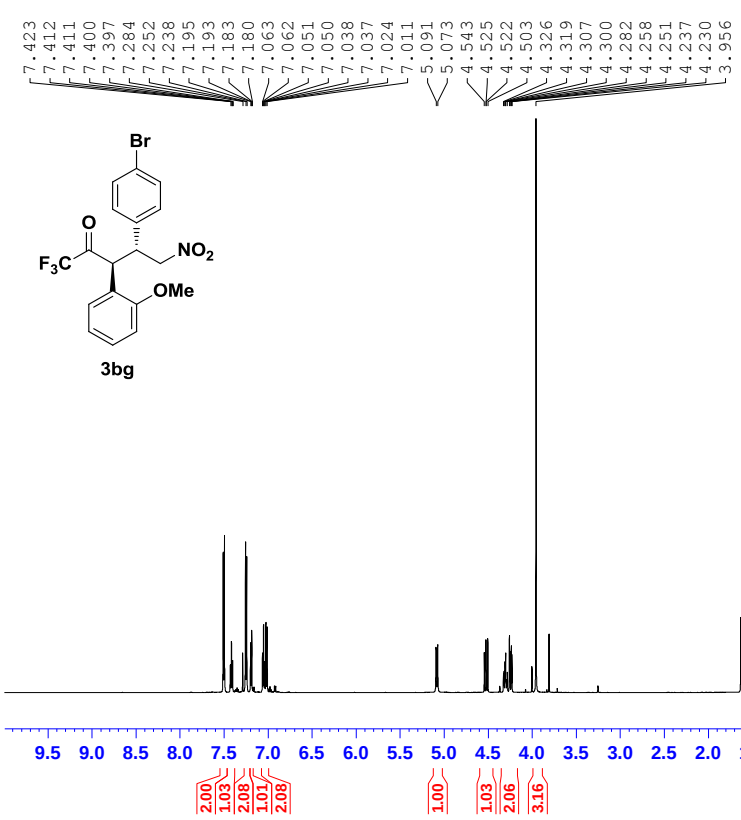


BRUKER

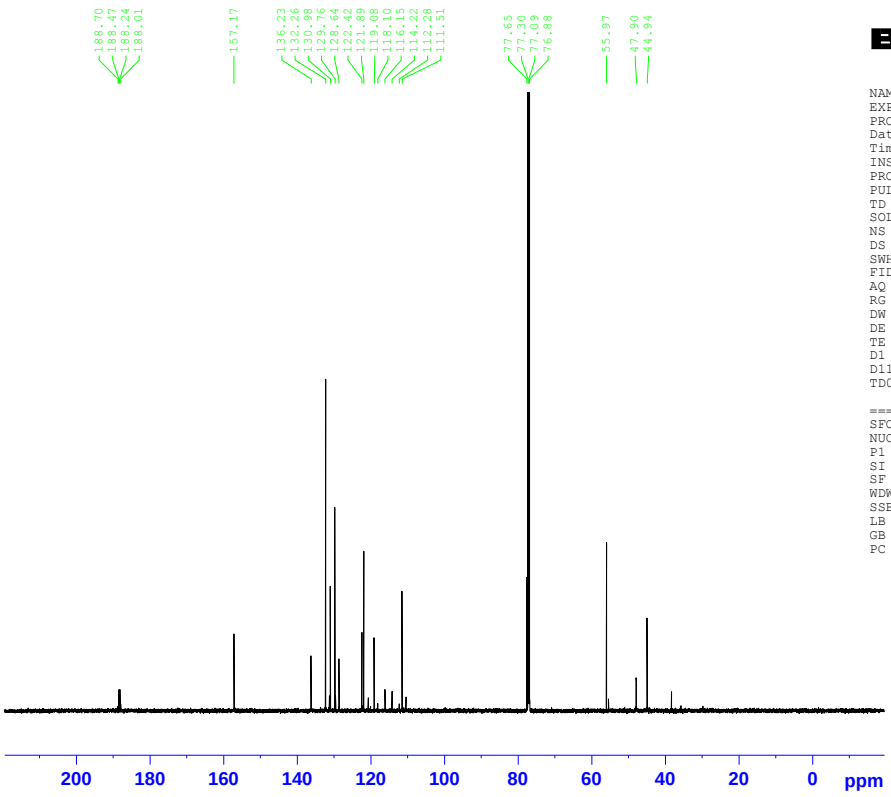
```
NAME                QX1267C1
EXPNO                      1
PROCNO                     1
Date_               20131018
Time                   15.18
INSTRUM                spect
PROBHD    5 mm CPQNP 1H/
PULPROG                zg30
TD                      65536
SOLVENT                 CDCl3
NS                         13
DS                          2
SWH                12019.230 Hz
FIDRES              0.183399 Hz
AQ                  2.7263477 sec
RG                         32
DW                     41.600 usec
DE                     10.00 usec
TE                     298.0 K
D1                 1.00000000 sec
TD0                        1

======== CHANNEL f1 ========
SFO1             600.1337060 MHz
NUC1                     1H
P1                     13.50 usec
SI                      65536
SF               600.1300000 MHz
WDW                       EM
SSB                        0
LB                      0.30 Hz
GB                         0
PC                      1.00
```



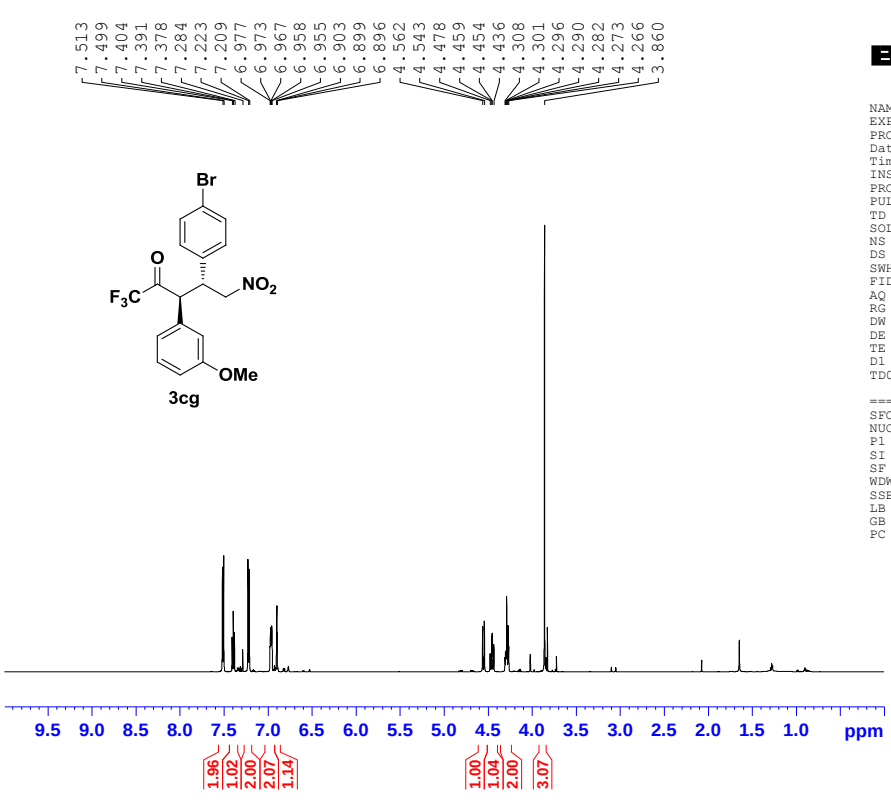

```
NAME                QX1267C1
EXPNO                      2
PROCNO                     1
Date_               20131018
Time                   15.21
INSTRUM                spect
PROBHD    5 mm CPQNP 1H/
PULPROG                zgpg30
TD                     65536
SOLVENT                CDCl3
NS                        44
DS                         4
SWH                36057.691 Hz
FIDRES                 0.550197 Hz
AQ                     0.9088159 sec
RG                       203
DW                    13.867 usec
DE                    18.00 usec
TE                    298.0 K
D1                 2.00000000 sec
D11                0.03000000 sec
TD0                        1

======== CHANNEL f1 ========
SFO1             150.9178981 MHz
NUC1                     13C
P1                    11.25 usec
SI                     32768
SF               150.9028090 MHz
WDW                       EM
SSB                        0
LB                     1.00 Hz
GB                         0
PC                     1.40
```



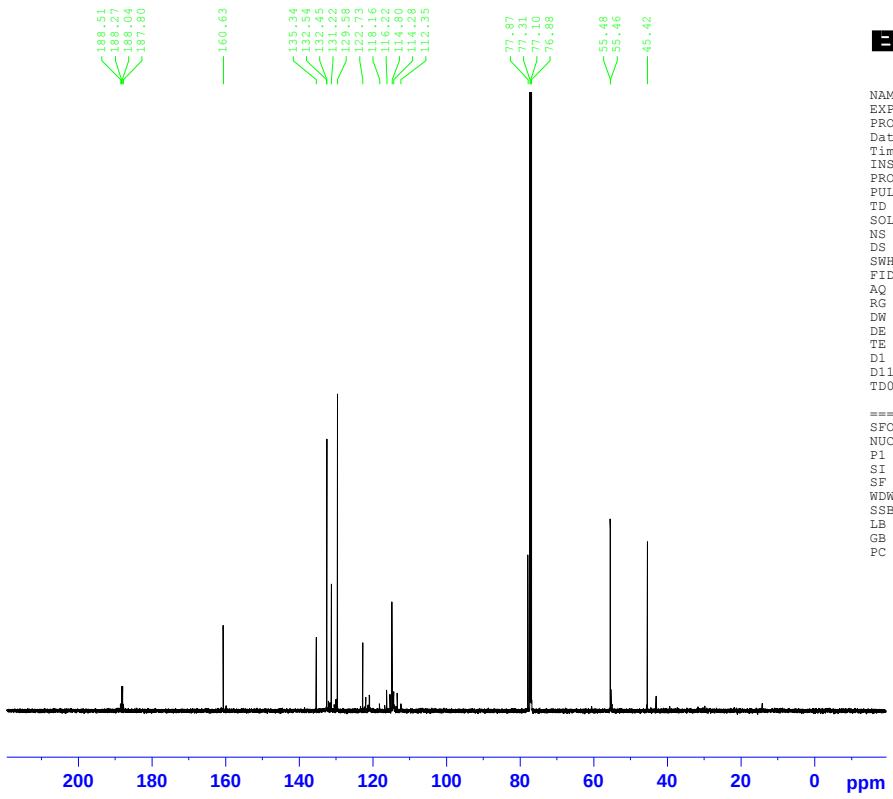

```
NAME                QX1282A1
EXPNO                      1
PROCNO                     1
Date_               20131107
Time                   10.24
INSTRUM                spect
PROBHD    5 mm CPQNP 1H/
PULPROG                zg30
TD                     65536
SOLVENT                CDCl3
NS                        16
DS                         2
SWH                12019.230 Hz
FIDRES                 0.183399 Hz
AQ                     2.7263477 sec
RG                        32
DW                    41.600 usec
DE                    10.00 usec
TE                    283.2 K
D1                 1.00000000 sec
TD0                        1

======== CHANNEL f1 ========
SFO1             600.1337060 MHz
NUC1                     1H
P1                    13.50 usec
SI                     65536
SF               600.1300000 MHz
WDW                       EM
SSB                        0
LB                     0.30 Hz
GB                         0
PC                     1.00
```



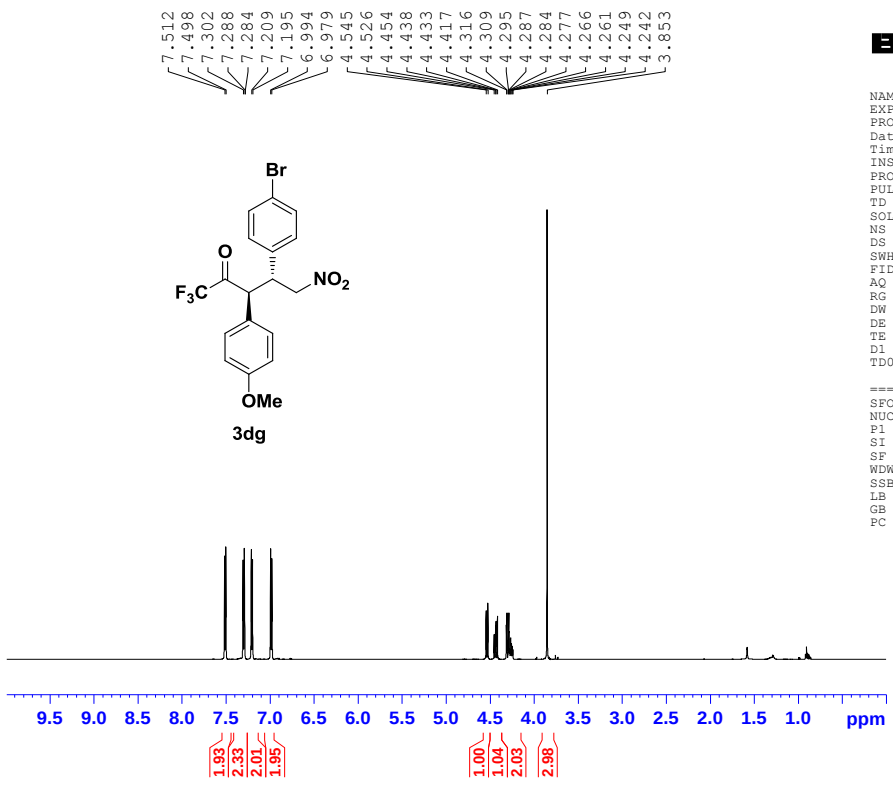

```
NAME                QX1282A1
EXPNO                      2
PROCNO                     1
Date_               20131107
Time                   10.27
INSTRUM                spect
PROBHD    5 mm CPQNP 1H/
PULPROG                zgpg30
TD                     65536
SOLVENT                CDCl3
NS                        41
DS                         4
SWH                36057.691 Hz
FIDRES                 0.550197 Hz
AQ                     0.9088159 sec
RG                       203
DW                      13.867 usec
DE                      18.00 usec
TE                      283.2 K
D1                   2.00000000 sec
D11                  0.03000000 sec
TD0                        1

======== CHANNEL f1 ========
SFO1               150.9178981 MHz
NUC1                       13C
P1                      11.25 usec
SI                         32768
SF                 150.9028090 MHz
WDW                        EM
SSB                        0
LB                      1.00 Hz
GB                         0
PC                      1.40
```



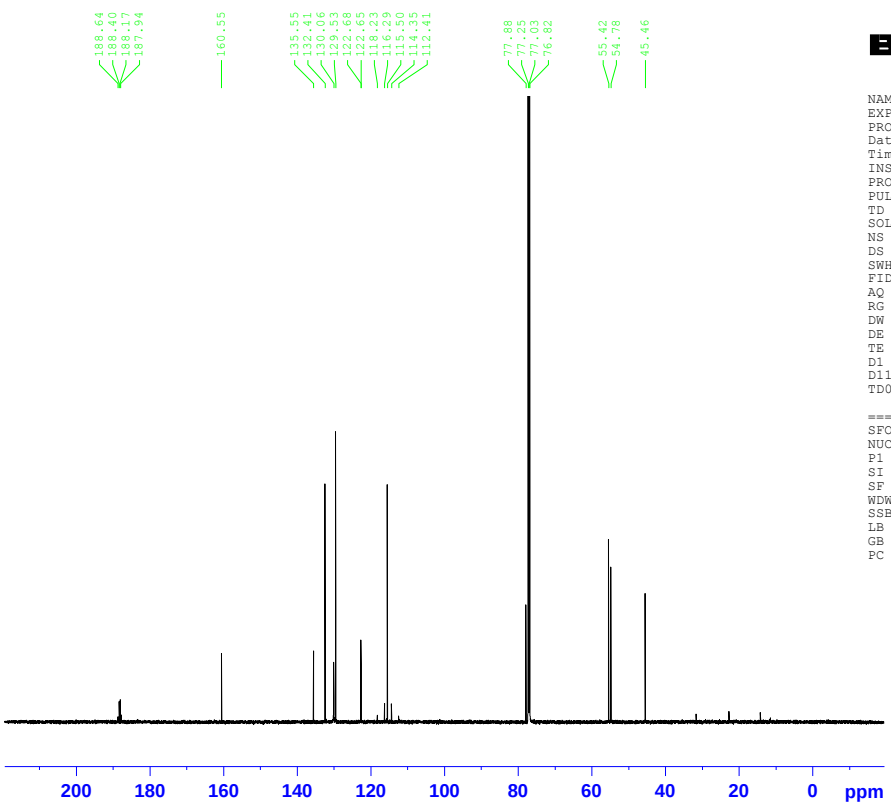

```
NAME                QX1282B1
EXPNO                      1
PROCNO                     1
Date_               20131107
Time                   10.35
INSTRUM                spect
PROBHD    5 mm CPQNP 1H/
PULPROG                zg30
TD                     65536
SOLVENT                CDCl3
NS                        16
DS                         2
SWH                12019.230 Hz
FIDRES                 0.183399 Hz
AQ                     2.7263477 sec
RG                        32
DW                     41.600 usec
DE                      10.00 usec
TE                      283.5 K
D1                   1.00000000 sec
TD0                        1

======== CHANNEL f1 ========
SFO1               600.1337060 MHz
NUC1                       1H
P1                      13.50 usec
SI                         65536
SF                 600.1300000 MHz
WDW                        EM
SSB                        0
LB                      0.30 Hz
GB                         0
PC                      1.00
```



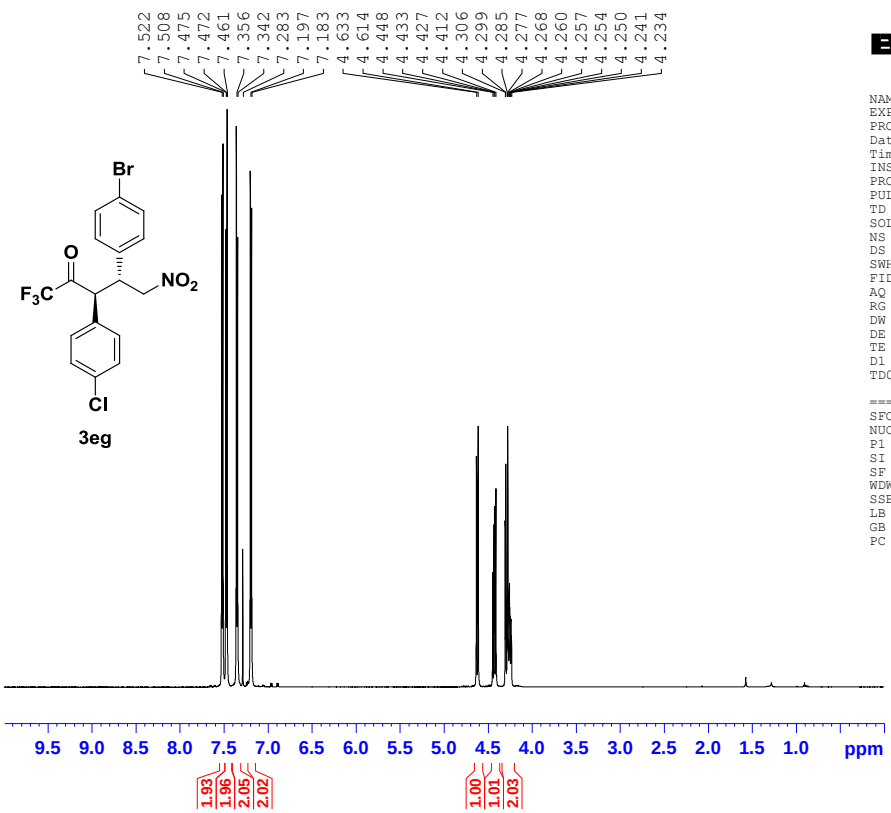

```
NAME                QX1282B1
EXPNO                      2
PROCNO                     1
Date_               20131107
Time                   10.32
INSTRUM                spect
PROBHD    5 mm CPQNP 1H/
PULPROG                zgpg30
TD                     65536
SOLVENT                CDCl3
NS                        41
DS                         4
SWH                36057.691 Hz
FIDRES              0.550197 Hz
AQ                  0.9088159 sec
RG                       203
DW                    13.867 usec
DE                    18.00 usec
TE                    283.4 K
D1                 2.00000000 sec
D11                0.03000000 sec
TD0                        1

======== CHANNEL f1 ========
SFO1              150.9178981 MHz
NUC1                      13C
P1                      11.25 usec
SI                      32768
SF                150.9028090 MHz
WDW                       EM
SSB                        0
LB                      1.00 Hz
GB                         0
PC                      1.40
```



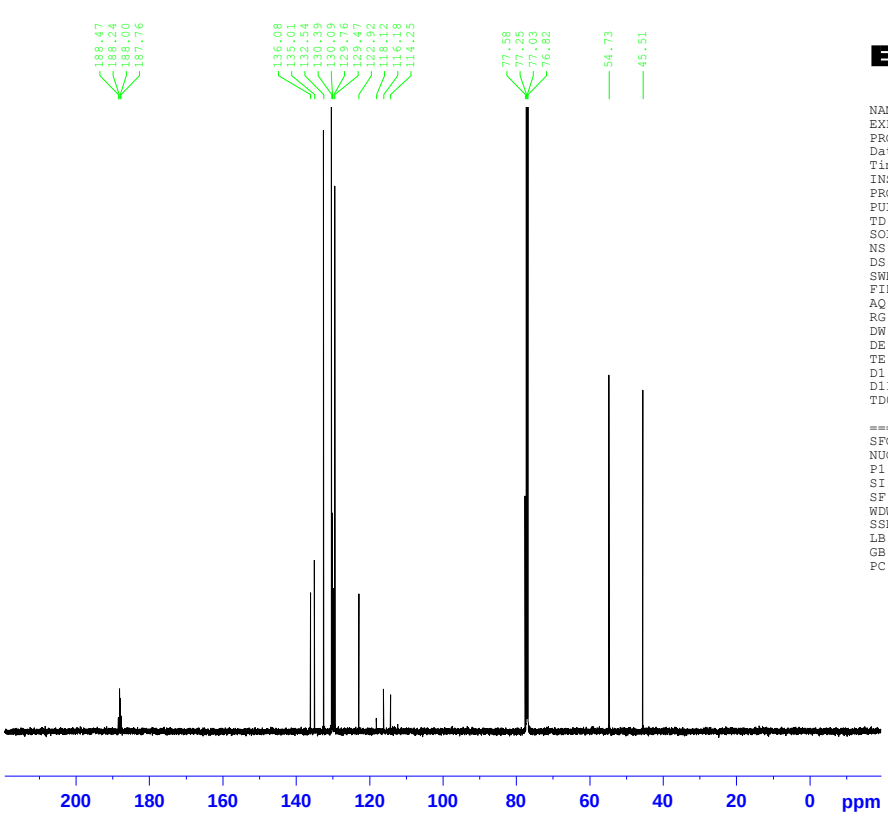

```
NAME              QX1282C1-2
EXPNO                      1
PROCNO                     1
Date_               20131112
Time                   17.53
INSTRUM                spect
PROBHD    5 mm CPQNP 1H/
PULPROG                zg30
TD                     65536
SOLVENT                CDCl3
NS                        14
DS                         2
SWH                12019.230 Hz
FIDRES              0.183399 Hz
AQ                  2.7263477 sec
RG                      50.8
DW                   41.600 usec
DE                    10.00 usec
TE                    298.0 K
D1                 1.00000000 sec
TD0                        1

======== CHANNEL f1 ========
SFO1              600.1337060 MHz
NUC1                       1H
P1                      13.50 usec
SI                      65536
SF                600.1300000 MHz
WDW                       EM
SSB                        0
LB                      0.30 Hz
GB                         0
PC                      1.00
```




```
NAME                QX1282C1-2
EXPNO                        2
PROCNO                       1
Date_                 20131112
Time                     17.56
INSTRUM                  spect
PROBHD    5 mm CPQNP 1H/
PULPROG                zgpg30
TD                        65536
SOLVENT                  CDCl3
NS                           79
DS                            4
SWH                 36057.691 Hz
FIDRES                0.550197 Hz
AQ                    0.9088159 sec
RG                          203
DW                       13.867 usec
DE                       18.00 usec
TE                       298.0 K
D1                    2.00000000 sec
D11                   0.03000000 sec
TD0                          1

======== CHANNEL f1 ========
SFO1                150.9178981 MHz
NUC1                        13C
P1                       11.25 usec
SI                        32768
SF                  150.9028090 MHz
WDW                         EM
SSB                          0
LB                        1.00 Hz
GB                           0
PC                        1.40
```




```
NAME          QX1283A-cryst
EXPNO                     1
PROCNO                    1
Date_              20140108
Time                  13.15
INSTRUM               spect
PROBHD    5 mm CPQNP 1H/
PULPROG                zg30
TD                     65536
SOLVENT               CDCl3
NS                        6
DS                        2
SWH              12019.230 Hz
FIDRES            0.183399 Hz
AQ                2.7263477 sec
RG                       57
DW                   41.600 usec
DE                   10.00 usec
TE                   298.0 K
D1                1.00000000 sec
TD0                       1

======== CHANNEL f1 ========
SFO1             600.1337060 MHz
NUC1                     1H
P1                   13.50 usec
SI                    65536
SF               600.1300000 MHz
WDW                      EM
SSB                       0
LB                     0.30 Hz
GB                        0
PC                     1.00
```



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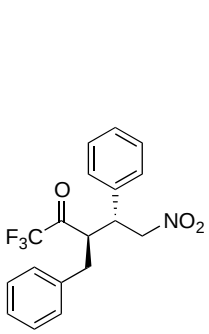
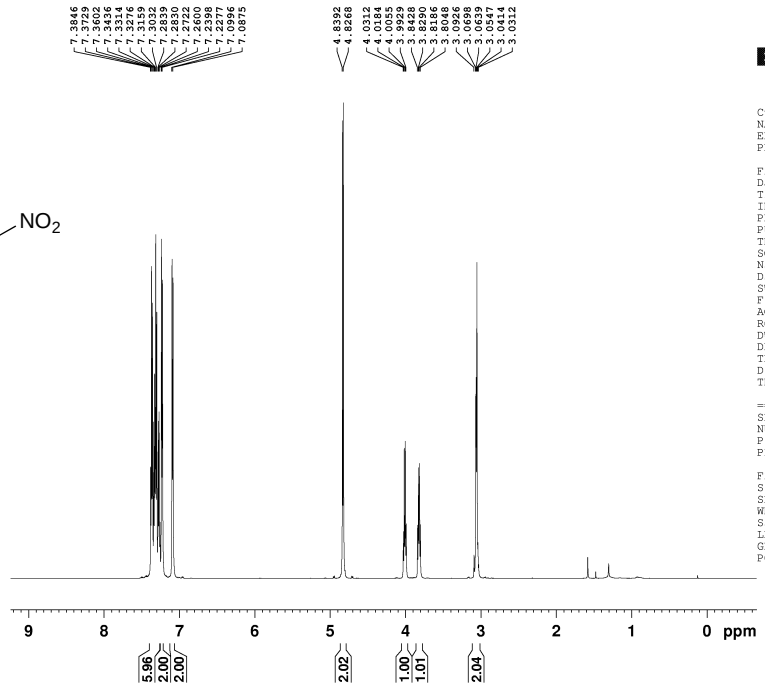
NAME	QX1283A-cryst
EXPNO	2
PROCNO	1
Date_	20140108
Time	13.12
INSTRUM	spect
PROBHD	5 mm CPQNP 1H/
PULPROG	zgpg30
TD	65536
SOLVENT	CDCl3
NS	30
DS	4
SWH	36057.691 Hz
FIDRES	0.550197 Hz
AQ	0.9088159 sec
RG	203
DW	13.867 usec
DE	18.00 usec
TE	298.0 K
D1	2.00000000 sec
D11	0.03000000 sec
TD0	1
======== CHANNEL f1 ========	
SFO1	150.9178981 MHz
NUC1	13C
P1	11.25 usec
SI	32768
SF	150.9028090 MHz
WDW	EM
SSB	0
LB	1.00 Hz
GB	0
PC	1.40

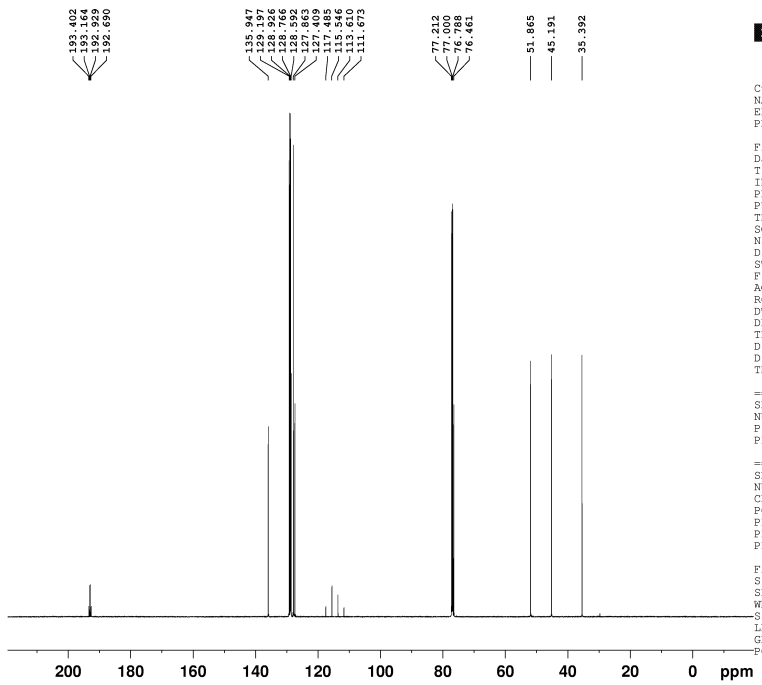


3fa



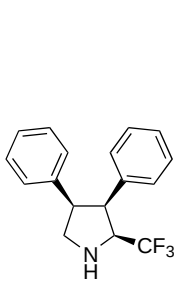
BRUKER

```
Current Data Parameters
NAME          MTC-13-74-H
EXPNO                   1
PROCNO                  1

F2 - Acquisition Parameters
Date_              20131116
Time                   6.42
INSTRUM               spect
PROBHD   5 mm CPQNP 1H/
PULPROG               zg30
TD                   65536
SOLVENT              CDCl3
NS                      16
DS                       0
SWH              12019.230 Hz
FIDRES           0.183399 Hz
AQ               2.7262976 sec
RG                    25.4
DW                  41.600 usec
DE                  10.00 usec
TE                  298.0 K
D1              1.00000000 sec
TD0                     1

======== CHANNEL f1 ========
SFO1           600.1337060 MHz
NUC1                   1H
P1                  13.50 usec
PLW1            9.00000000 W

F2 - Processing parameters
SI                   65536
SF             600.1300008 MHz
WDW                     EM
SSB          0
LB                   0.30 Hz
GB           0
PC                   1.00
```

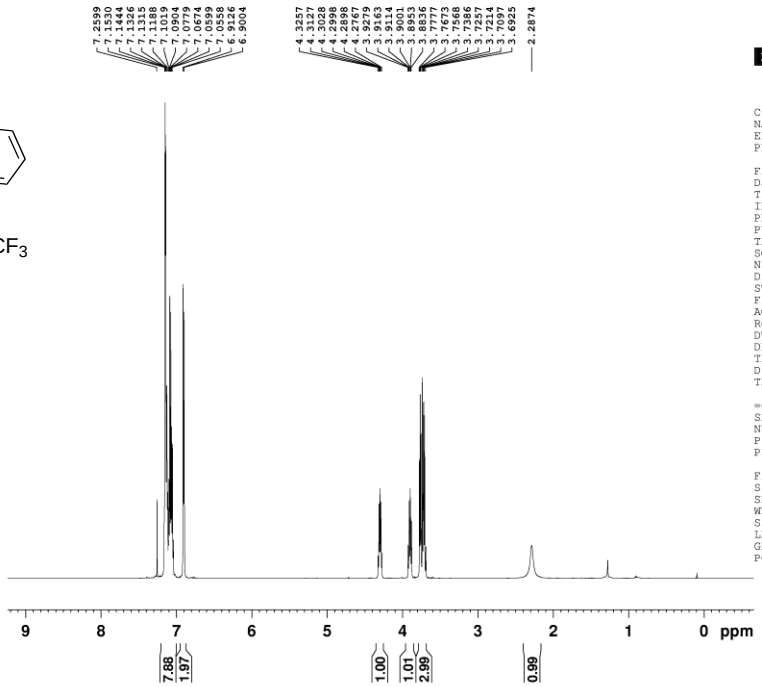


BRUKER

```
Current Data Parameters
NAME          MTC-13-74-C
EXPNO                   1
PROCNO                  1

F2 - Acquisition Parameters
Date_              20131116
Time                   6.47
INSTRUM               spect
PROBHD   5 mm CPQNP 1H/
PULPROG           zgpg30
TD                   65536
SOLVENT              CDCl3
NS                      70
DS                       0
SWH              36057.691 Hz
FIDRES           0.550197 Hz
AQ               0.9087659 sec
RG                     203
DW                  13.867 usec
DE                  18.00 usec
TE                  298.0 K
D1              2.00000000 sec
D11             0.03000000 sec
TD0                     1

======== CHANNEL f1 ========
SFO1           150.9178981 MHz
NUC1                   13C
P1                  11.25 usec
PLW1           30.00000000 W

======== CHANNEL f2 ========
SFO2           600.1324005 MHz
NUC2                   1H
CPDPRG[2           waltz16
PCPD2                80.00 usec
PLW2            9.00000000 W
PLW12           0.27562001 W
PLW13           0.17640001 W

F2 - Processing parameters
SI                   32768
SF             150.9028217 MHz
WDW                     EM
SSB          0
LB                   1.00 Hz
GB           0
PC                   1.40
```



4a



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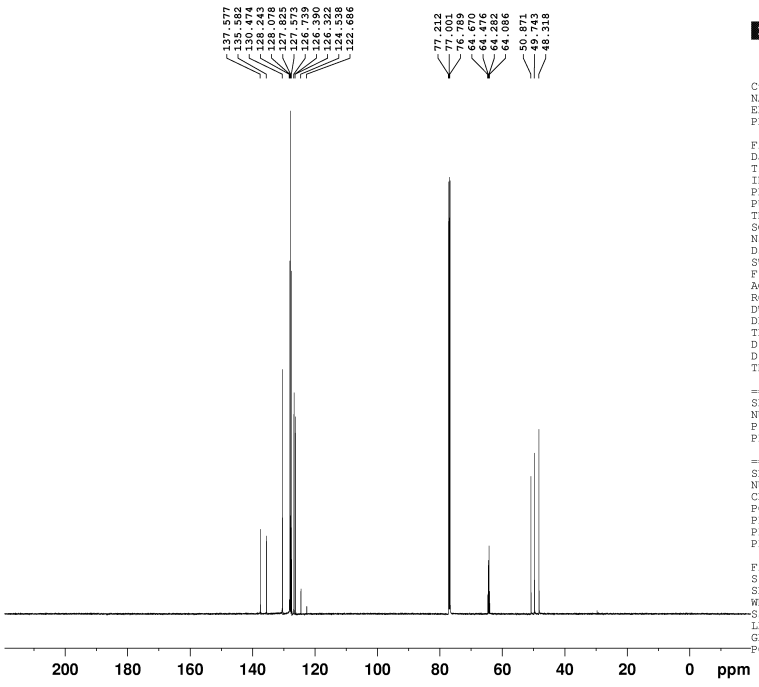
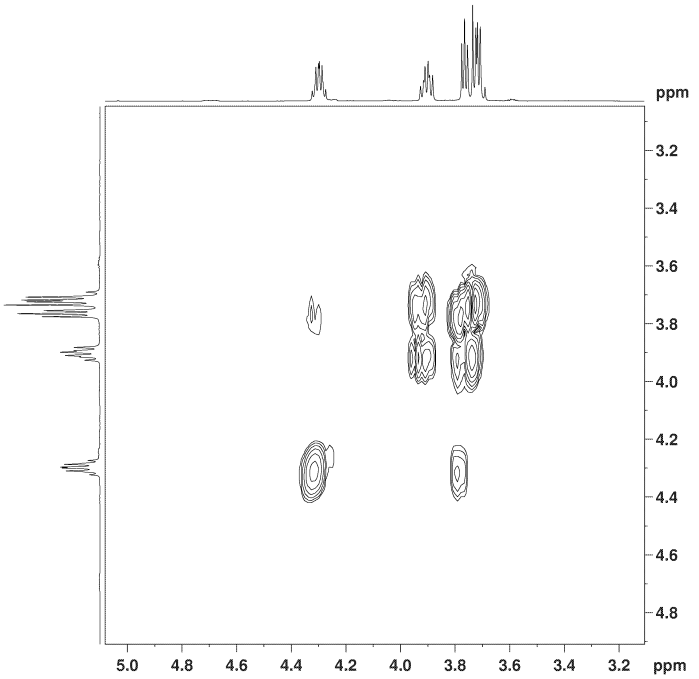
```
Current Data Parameters
NAME         MTC-13-79-H
EXPNO                  1
PROCNO                 1

F2 - Acquisition Parameters
Date_           20131112
Time               16.43
INSTRUM            spect
PROBHD   5 mm CPQNP 1H/
PULPROG            zg30
TD                65536
SOLVENT           CDCl3
NS                   16
DS                    0
SWH           12019.230 Hz
FIDRES            0.183399 Hz
AQ            2.7262976 sec
RG                   32
DW               41.600 usec
DE               10.00 usec
TE                298.0 K
D1            1.00000000 sec
TD0                    1

======== CHANNEL f1 ========
SFO1         600.1337060 MHz
NUC1                   1H
P1                 13.50 usec
PLW1          9.00000000 W

F2 - Processing parameters
SI                 65536
SF           600.1300153 MHz
WDW                   EM
SSB       0
LB                 0.30 Hz
GB        0
PC                 1.00
```

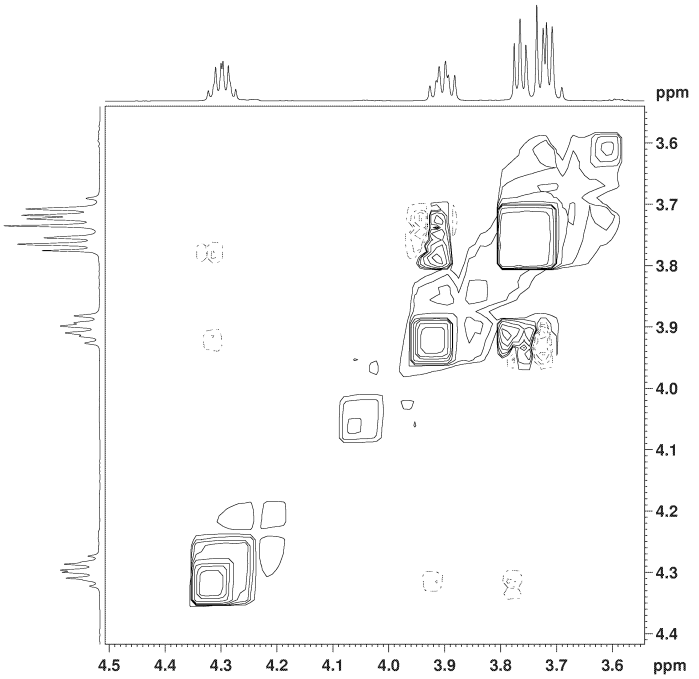


BRUKER

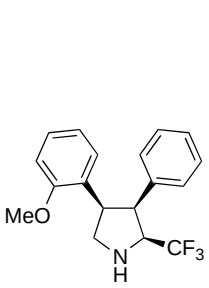
```
Current Data Parameters
NAME         MTC-13-79-C
EXPNO                  1
PROCNO                 1

F2 - Acquisition Parameters
Date_           20131112
Time               16.47
INSTRUM            spect
PROBHD   5 mm CPQNP 1H/
PULPROG          zgpg30
TD                65536
SOLVENT           CDCl3
NS                   80
DS                    0
SWH           36057.691 Hz
FIDRES            0.550197 Hz
AQ            0.9087659 sec
RG                  203
DW               13.867 usec
DE                18.00 usec
TE                298.0 K
D1            2.00000000 sec
D11           0.03000000 sec
TD0                    1

======== CHANNEL f1 ========
SFO1         150.9178981 MHz
NUC1                   13C
P1                 11.25 usec
PLW1         30.00000000 W

======== CHANNEL f2 ========
SFO2         600.1324005 MHz
NUC2                   1H
CPDPRG[2          waltz16
PCPD2              80.00 usec
PLW2          9.00000000 W
PLW12         0.27562001 W
PLW13         0.17640001 W

F2 - Processing parameters
SI                 32768
SF           150.9028195 MHz
WDW                   EM
SSB       0
LB                 1.00 Hz
GB        0
PC                 1.40
```



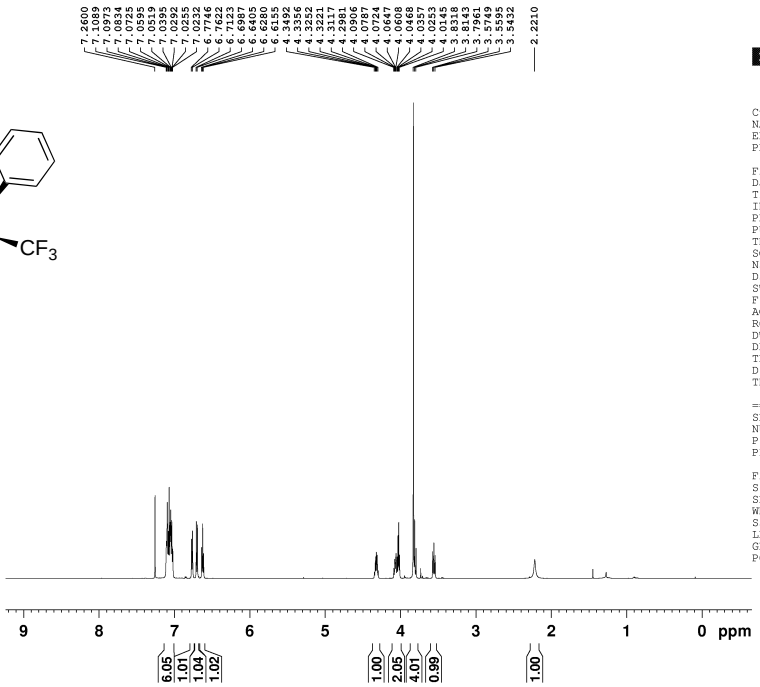

```
Current Data Parameters
NAME      MTC-13-72-COSY
EXPNO                  1
PROCNO                 1

F2 - Acquisition Parameters
Date_           20131102
Time                6.06
INSTRUM            spect
PROBHD   5 mm CPQNP 1H/
PULPROG      cosygpppqf
TD                  2048
SOLVENT             CDCl3
NS                     1
DS                     0
SWH            8012.820 Hz
FIDRES         3.912510 Hz
AQ             0.1277952 sec
RG                   161
DW               62.400 usec
DE               10.00 usec
TE                298.2 K
D0            0.00000300 sec
D1            2.00000000 sec
D11           0.03000000 sec
D12           0.00002000 sec
D13           0.00000400 sec
D16           0.00020000 sec
IN0           0.00012480 sec

======== CHANNEL f1 ========
SFO1          600.1336081 MHz
NUC1                  1H
P0                  13.50 usec
P1                  13.50 usec
P17               2500.00 usec
PLW1          9.00000000 W
PLW10         2.42639995 W

====== GRADIENT CHANNEL =====
GPNAM(1)       SMSQ10.100
GPZ1                10.00 %
P16               1000.00 usec

F1 - Acquisition parameters
TD                    128
SFO1           600.1336 MHz
FIDRES         62.600159 Hz
SW                13.352 ppm
FnMODE                QF

F2 - Processing parameters
SI                   1024
SF            600.1299999 MHz
WDW                 QSINE
SSB                    0
LB                 0 Hz
GB                     0
PC                  1.40

F1 - Processing parameters
SI                   1024
MC2                    QF
SF            600.1300008 MHz
WDW                 QSINE
SSB                    0
LB                 0 Hz
GB                     0
```



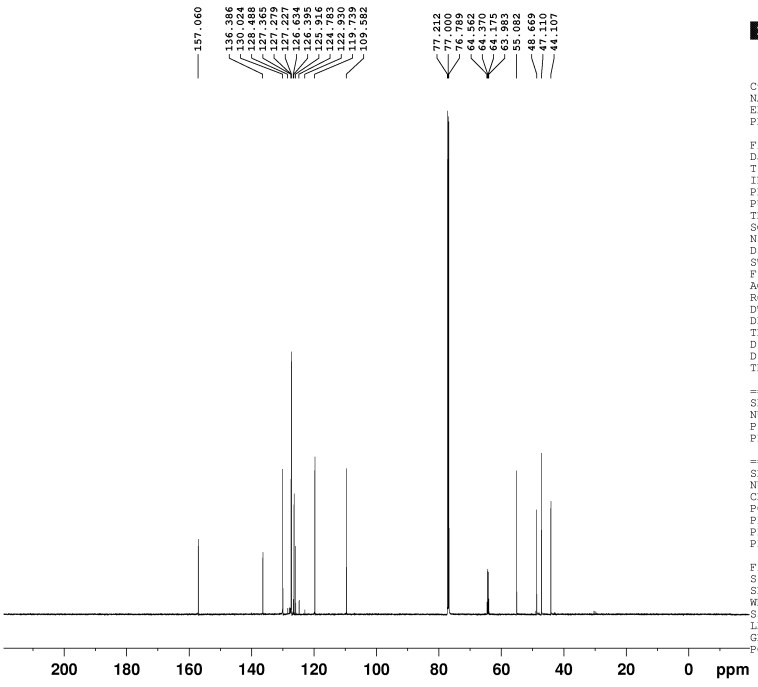

```
Current Data Parameters
NAME      MTC-13-72-NOESY
EXPNO                  1
PROCNO                 1

F2 - Acquisition Parameters
Date_           20131102
Time                6.11
INSTRUM            spect
PROBHD   5 mm CPQNP 1H/
PULPROG      noesygpphpp
TD                  2048
SOLVENT             CDCl3
NS                     4
DS                     0
SWH            6009.615 Hz
FIDRES         2.934382 Hz
AQ             0.1703936 sec
RG                    64
DW               83.200 usec
DE               10.00 usec
TE                298.1 K
D0            0.00006601 sec
D1            2.00000000 sec
D8            0.30000001 sec
D11           0.03000000 sec
D12           0.00002000 sec
D16           0.00020000 sec
IN0           0.00016640 sec

======== CHANNEL f1 ========
SFO1          600.1327625 MHz
NUC1                  1H
P1                  13.50 usec
P2                  27.00 usec
P17               2500.00 usec
PLW1          9.00000000 W
PLW10         2.42639995 W

====== GRADIENT CHANNEL =====
GPNAM(1)       SMSQ10.100
GPZ1                40.00 %
P16               1000.00 usec

F1 - Acquisition parameters
TD                    128
SFO1           600.1328 MHz
FIDRES         48.076923 Hz
SW                10.014 ppm
FnMODE       States-TPPI

F2 - Processing parameters
SI                   1024
SF            600.1299999 MHz
WDW                 QSINE
SSB                    2
LB                 0 Hz
GB                     0
PC                  1.00

F1 - Processing parameters
SI                   1024
MC2          States-TPPI
SF            600.1299999 MHz
WDW                 QSINE
SSB                    2
LB                 0 Hz
GB                     0
```



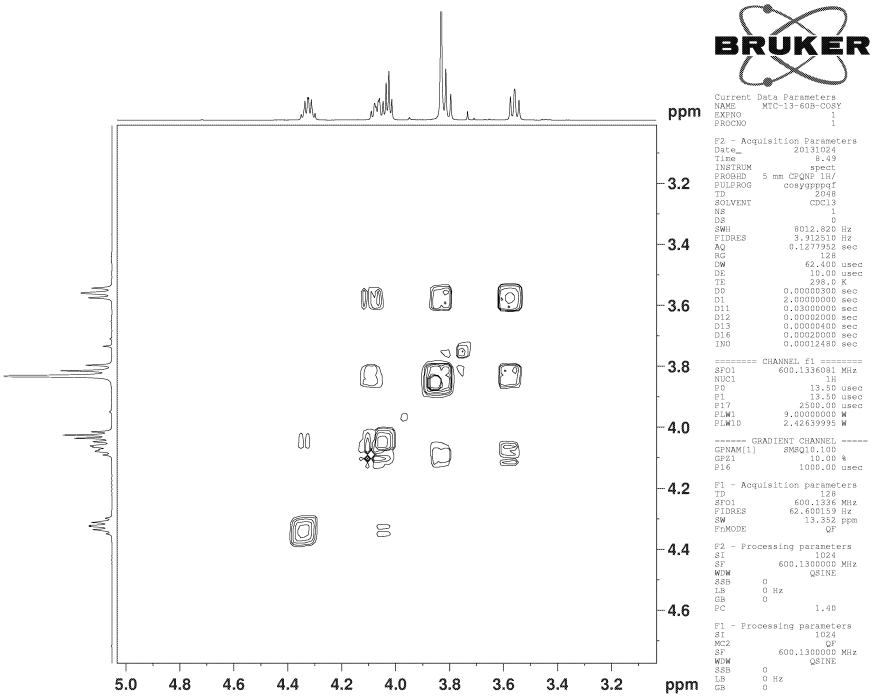
4b



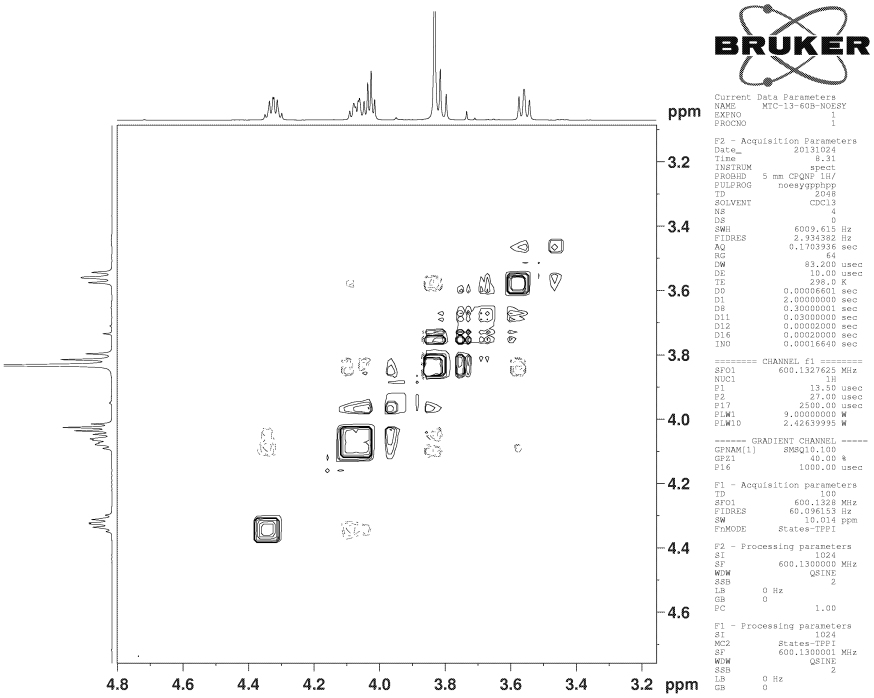

```
Current Data Parameters
NAME        MTC-13-60B-H
EXPNO                  1
PROCNO                 1

F2 - Acquisition Parameters
Date_           20131023
Time               17.01
INSTRUM            spect
PROBHD   5 mm CPQNP 1H/
PULPROG            zg30
TD                65536
SOLVENT           CDCl3
NS                   16
DS                    0
SWH           12019.230 Hz
FIDRES            0.183399 Hz
AQ            2.7262976 sec
RG                  181
DW               41.600 usec
DE               10.00 usec
TE               298.0 K
D1            1.00000000 sec
TD0                    1

======== CHANNEL f1 ========
SFO1         600.1337060 MHz
NUC1                   1H
P1                 13.50 usec
PLW1          9.00000000 W

F2 - Processing parameters
SI                 65536
SF           600.1300141 MHz
WDW                   EM
SSB       0
LB                 0.30 Hz
GB        0
PC                 1.00
```



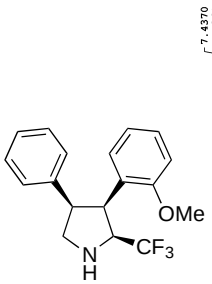

```
Current Data Parameters
NAME        MTC-13-60B-C
EXPNO                  1
PROCNO                 1

F2 - Acquisition Parameters
Date_           20131023
Time               17.05
INSTRUM            spect
PROBHD   5 mm CPQNP 1H/
PULPROG          zgpg30
TD                65536
SOLVENT           CDCl3
NS                  149
DS                    0
SWH           36057.691 Hz
FIDRES            0.550197 Hz
AQ            0.9087659 sec
RG                  203
DW               13.867 usec
DE                18.00 usec
TE               298.0 K
D1            2.00000000 sec
D11           0.03000000 sec
TD0                    1

======== CHANNEL f1 ========
SFO1         150.9178981 MHz
NUC1                   13C
P1                 11.25 usec
PLW1         30.00000000 W

======== CHANNEL f2 ========
SFO2         600.1324005 MHz
NUC2                   1H
CPDPRG[2          waltz16
PCPD2              80.00 usec
PLW2          9.00000000 W
PLW12         0.27562001 W
PLW13         0.17640001 W

F2 - Processing parameters
SI                 32768
SF           150.9028184 MHz
WDW                   EM
SSB       0
LB                 1.00 Hz
GB        0
PC                 1.40
```



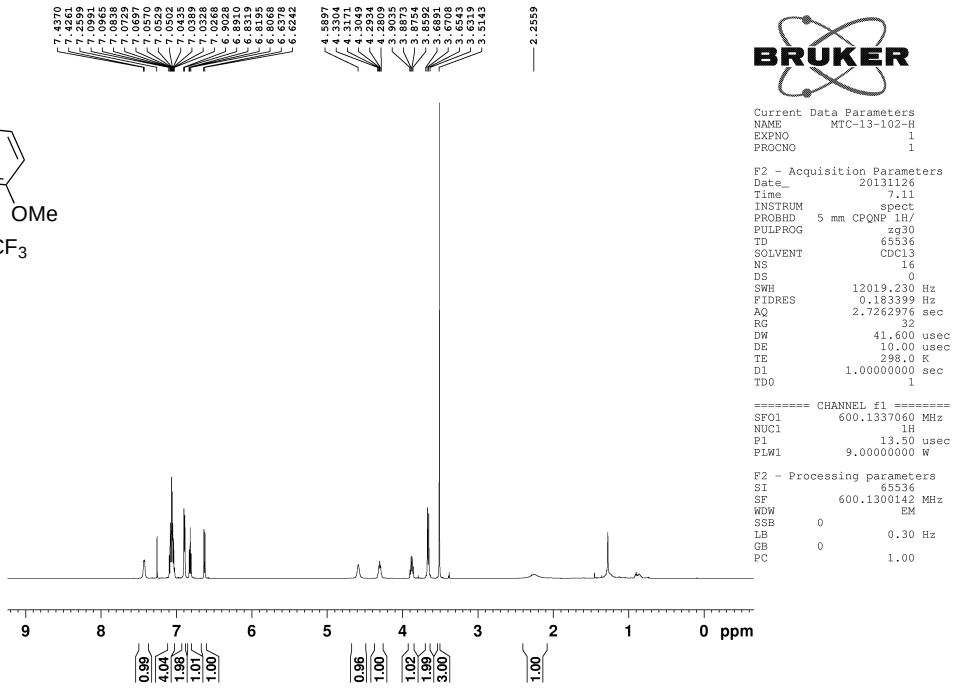

```
Current Data Parameters
NAME      MTC-13-60S-COSY
EXPNO                   1
PROCNO                  1

F2 - Acquisition Parameters
Date_           20131024
Time                8.49
INSTRUM            spect
PROBHD   5 mm CPQNP 1H/
PULPROG      cosygpppqf
TD                  2048
SOLVENT           CDCl3
NS                     1
DS                     0
SWH            8012.820 Hz
FIDRES         3.912510 Hz
AQ             0.1277952 sec
RG                   128
DW              62.400 usec
DE              10.00 usec
TE                298.0 K
D0          0.00000300 sec
D1          2.00000000 sec
D11         0.03000000 sec
D12         0.00002000 sec
D13         0.00000400 sec
D16         0.00020000 sec
IN0         0.00012480 sec

======== CHANNEL f1 ========
SFO1          600.1336081 MHz
NUC1                  1H
P0                  13.50 usec
P1                  13.50 usec
P17               2500.00 usec
PLW1         9.00000000 W
PLW10        2.42639995 W

====== GRADIENT CHANNEL =====
GPNAM(1)       SMSQ10.100
GPZ1                10.00 %
P16               1000.00 usec

F1 - Acquisition parameters
TD                   128
SFO1           600.1336 MHz
FIDRES         62.600159 Hz
SW                13.352 ppm
FnMODE                QF

F2 - Processing parameters
SI                   1024
SF            600.1300000 MHz
WDW                  QSINE
SSB                  0
LB                0 Hz
GB                   0
PC                  1.40

F1 - Processing parameters
SI                   1024
MC2                    QF
SF            600.1300000 MHz
WDW                  QSINE
SSB                  0
LB                0 Hz
GB                   0
```




```
Current Data Parameters
NAME      MTC-13-60S-NOESY
EXPNO                   1
PROCNO                  1

F2 - Acquisition Parameters
Date_           20131024
Time                8.31
INSTRUM            spect
PROBHD   5 mm CPQNP 1H/
PULPROG     noesygpphpp
TD                  2048
SOLVENT           CDCl3
NS                     4
DS                     0
SWH            6009.615 Hz
FIDRES         2.934382 Hz
AQ             0.1703936 sec
RG                    64
DW              83.200 usec
DE              10.00 usec
TE                298.0 K
D0          0.00006601 sec
D1          2.00000000 sec
D8          0.30000001 sec
D11         0.03000000 sec
D12         0.00002000 sec
D16         0.00020000 sec
IN0         0.00016640 sec

======== CHANNEL f1 ========
SFO1          600.1327625 MHz
NUC1                  1H
P1                  13.50 usec
P2                  27.00 usec
P17               2500.00 usec
PLW1         9.00000000 W
PLW10        2.42639995 W

====== GRADIENT CHANNEL =====
GPNAM(1)       SMSQ10.100
GPZ1                40.00 %
P16               1000.00 usec

F1 - Acquisition parameters
TD                   100
SFO1           600.1328 MHz
FIDRES         60.096153 Hz
SW                10.014 ppm
FnMODE       States-TPPI

F2 - Processing parameters
SI                   1024
SF            600.1300000 MHz
WDW                  QSINE
SSB                     2
LB                0 Hz
GB                   0
PC                  1.00

F1 - Processing parameters
SI                   1024
MC2          States-TPPI
SF            600.1300001 MHz
WDW                  QSINE
SSB                     2
LB                0 Hz
GB                   0
```



4c



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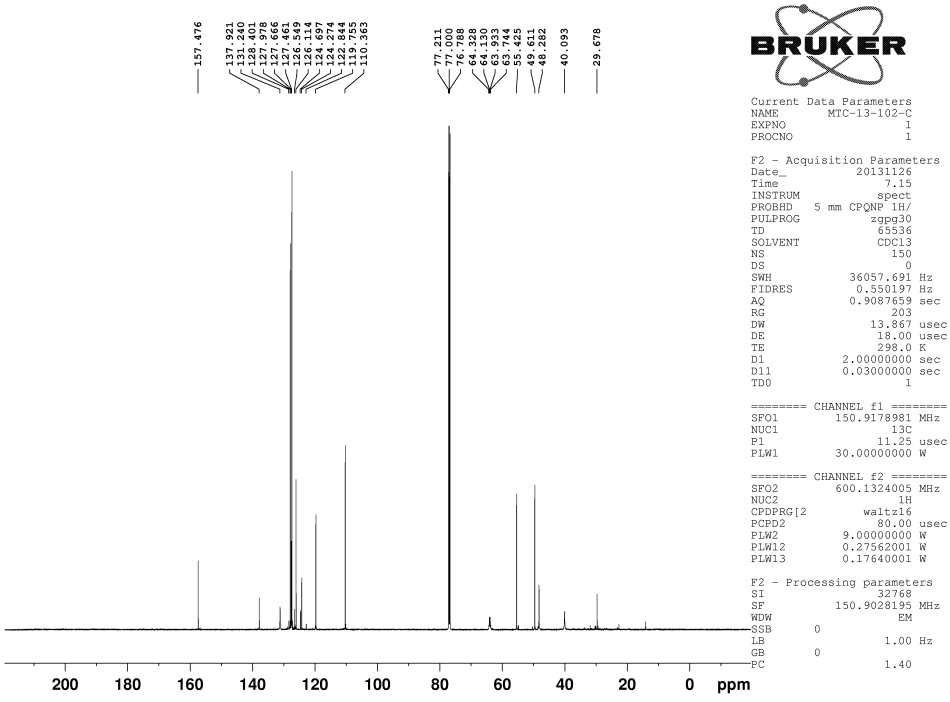
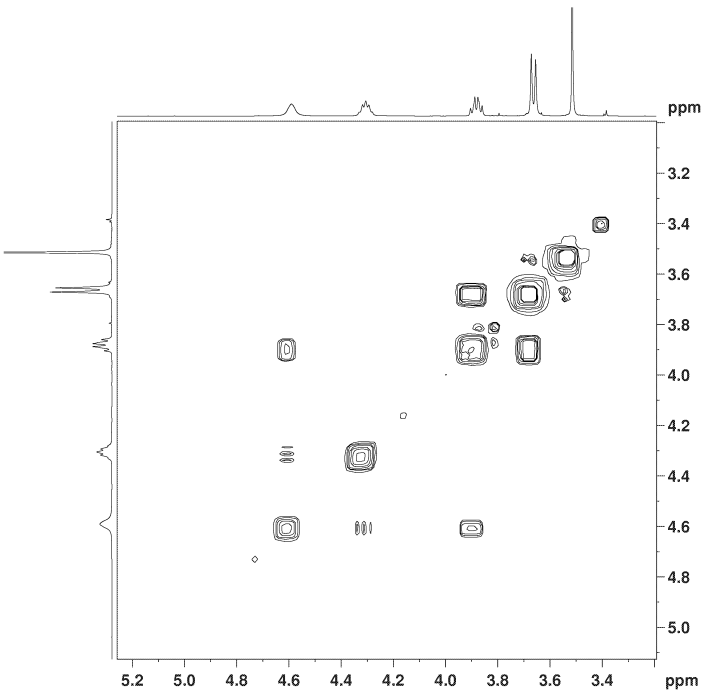
```
Current Data Parameters
NAME        MTC-13-102-H
EXPNO                  1
PROCNO                 1

F2 - Acquisition Parameters
Date_           20131126
Time                7.11
INSTRUM            spect
PROBHD   5 mm CPQNP 1H/
PULPROG             zg30
TD                 65536
SOLVENT            CDCl3
NS                    16
DS                     0
SWH            12019.230 Hz
FIDRES             0.183399 Hz
AQ             2.7262976 sec
RG                    32
DW                41.600 usec
DE                10.00 usec
TE                298.0 K
D1             1.00000000 sec
TD0                    1

======== CHANNEL f1 ========
SFO1          600.1337060 MHz
NUC1                  1H
P1                 13.50 usec
PLW1           9.00000000 W

F2 - Processing parameters
SI                 65536
SF            600.1300142 MHz
WDW                   EM
SSB       0
LB                 0.30 Hz
GB        0
PC                 1.00
```

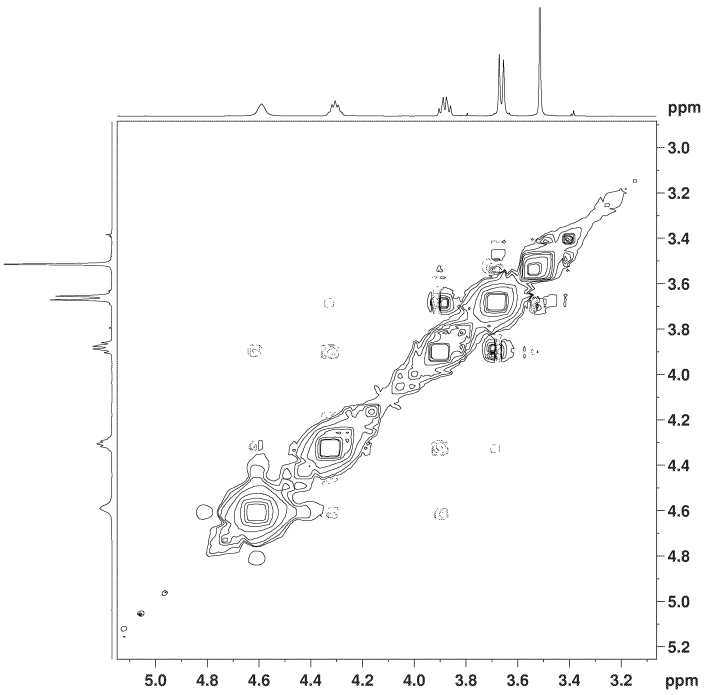


BRUKER

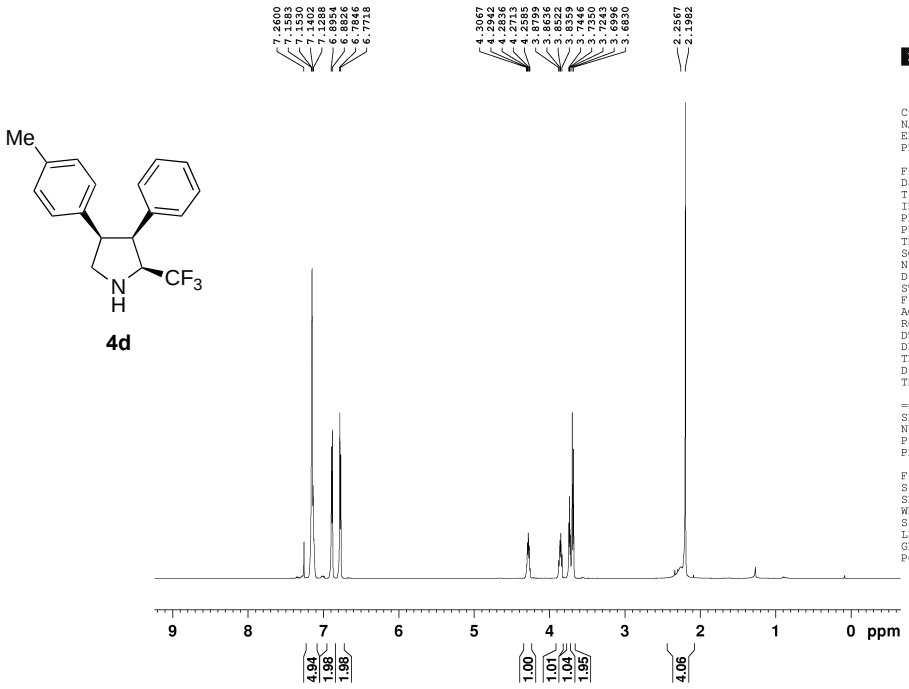
```
Current Data Parameters
NAME        MTC-13-102-C
EXPNO                  1
PROCNO                 1

F2 - Acquisition Parameters
Date_           20131126
Time                7.15
INSTRUM            spect
PROBHD   5 mm CPQNP 1H/
PULPROG          zgpg30
TD                 65536
SOLVENT            CDCl3
NS                   150
DS                     0
SWH            36057.691 Hz
FIDRES             0.550197 Hz
AQ             0.9087659 sec
RG                   203
DW                13.867 usec
DE                18.00 usec
TE                298.0 K
D1             2.00000000 sec
D11            0.03000000 sec
TD0                    1

======== CHANNEL f1 ========
SFO1          150.9178981 MHz
NUC1                  13C
P1                 11.25 usec
PLW1          30.00000000 W

======== CHANNEL f2 ========
SFO2          600.1324005 MHz
NUC2                  1H
CPDPRG[2          waltz16
PCPD2              80.00 usec
PLW2           9.00000000 W
PLW12          0.27562001 W
PLW13          0.17640001 W

F2 - Processing parameters
SI                 32768
SF            150.9028195 MHz
WDW                   EM
SSB       0
LB                 1.00 Hz
GB        0
PC                 1.40
```




```
Current Data Parameters
NAME      MTC-13-102-COSY
EXPNO                   1
PROCNO                  1

F2 - Acquisition Parameters
Date_           20131126
Time                7.20
INSTRUM             spect
PROBHD   5 mm CPQNP 1H/
PULPROG      cosygpppqf
TD                  2048
SOLVENT           CDCl3
NS                     1
DS                     0
SWH            8012.820 Hz
FIDRES         3.912510 Hz
AQ             0.1277952 sec
RG                    64
DW               62.400 usec
DE               10.00 usec
TE                298.0 K
D0             0.00000300 sec
D1             2.00000000 sec
D11            0.03000000 sec
D12            0.00002000 sec
D13            0.00000400 sec
D16            0.00020000 sec
IN0            0.00012480 sec

======== CHANNEL f1 ========
SFO1          600.1336081 MHz
NUC1                  1H
P0                  13.50 usec
P1                  13.50 usec
P17               2500.00 usec
PLW1         9.00000000 W
PLW10        2.42639995 W

====== GRADIENT CHANNEL =====
GPNAM(1)       SMSQ10.100
GPZ1                10.00 %
P16               1000.00 usec

F1 - Acquisition parameters
TD                    128
SFO1           600.1336 MHz
FIDRES         62.600159 Hz
SW                13.352 ppm
FnMODE                QF

F2 - Processing parameters
SI                   1024
SF            600.1300000 MHz
WDW                 QSINE
SSB          0
LB           0 Hz
GB           0
PC                   1.40

F1 - Processing parameters
SI                   1024
MC2                    QF
SF            600.1300000 MHz
WDW                 QSINE
SSB          0
LB           0 Hz
GB           0
```




```
Current Data Parameters
NAME      MTC-13-102-NOESY
EXPNO                   1
PROCNO                  1

F2 - Acquisition Parameters
Date_           20131126
Time                7.26
INSTRUM             spect
PROBHD   5 mm CPQNP 1H/
PULPROG      noesygpphpp
TD                  2048
SOLVENT           CDCl3
NS                     4
DS                     0
SWH            6009.615 Hz
FIDRES         2.934382 Hz
AQ             0.1703936 sec
RG                    64
DW               83.200 usec
DE               10.00 usec
TE                298.0 K
D0             0.00006601 sec
D1             2.00000000 sec
D8             0.30000001 sec
D11            0.03000000 sec
D12            0.00002000 sec
D16            0.00020000 sec
IN0            0.00016640 sec

======== CHANNEL f1 ========
SFO1          600.1327625 MHz
NUC1                  1H
P1                  13.50 usec
P2                  27.00 usec
P17               2500.00 usec
PLW1         9.00000000 W
PLW10        2.42639995 W

====== GRADIENT CHANNEL =====
GPNAM(1)       SMSQ10.100
GPZ1                40.00 %
P16               1000.00 usec

F1 - Acquisition parameters
TD                    161
SFO1           600.1328 MHz
FIDRES         37.326805 Hz
SW                10.014 ppm
FnMODE       States-TPPI

F2 - Processing parameters
SI                   1024
SF            600.1300000 MHz
WDW                 QSINE
SSB                    2
LB           0 Hz
GB           0
PC                   1.00

F1 - Processing parameters
SI                   1024
MC2          States-TPPI
SF            600.1300000 MHz
WDW                 QSINE
SSB                    2
LB           0 Hz
GB           0
```



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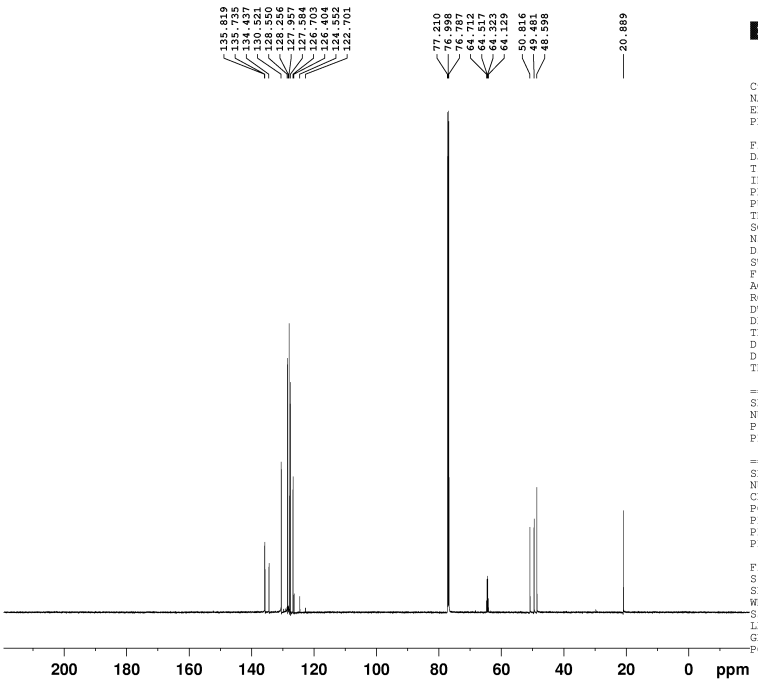
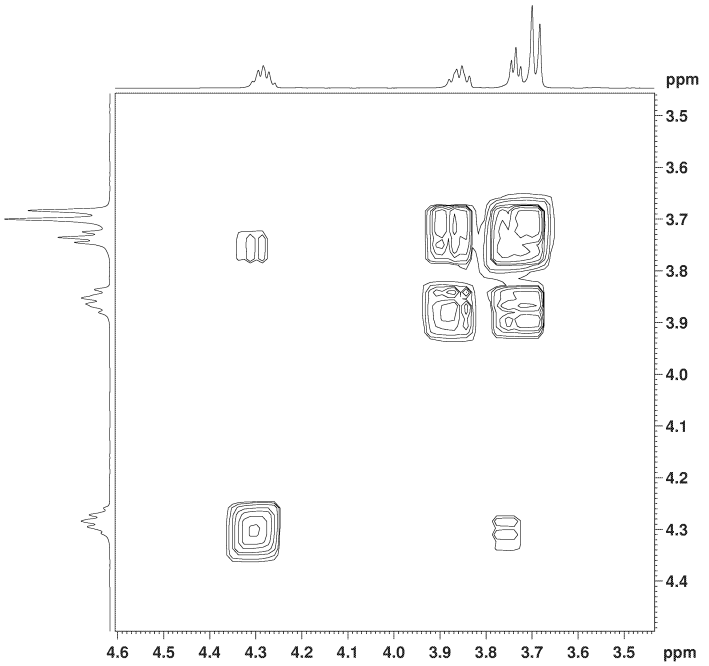
```
Current Data Parameters
NAME         MTC-13-80-H
EXPNO                  1
PROCNO                 1

F2 - Acquisition Parameters
Date_           20131112
Time               16.50
INSTRUM            spect
PROBHD   5 mm CPQNP 1H/
PULPROG            zg30
TD                65536
SOLVENT           CDCl3
NS                   16
DS                    0
SWH           12019.230 Hz
FIDRES            0.183399 Hz
AQ            2.7262976 sec
RG                   64
DW               41.600 usec
DE               10.00 usec
TE                298.0 K
D1            1.00000000 sec
TD0                   1

======== CHANNEL f1 ========
SFO1         600.1337060 MHz
NUC1                 1H
P1                13.50 usec
PLW1          9.00000000 W

F2 - Processing parameters
SI                65536
SF           600.1300144 MHz
WDW                  EM
SSB       0
LB                0.30 Hz
GB        0
PC                1.00
```

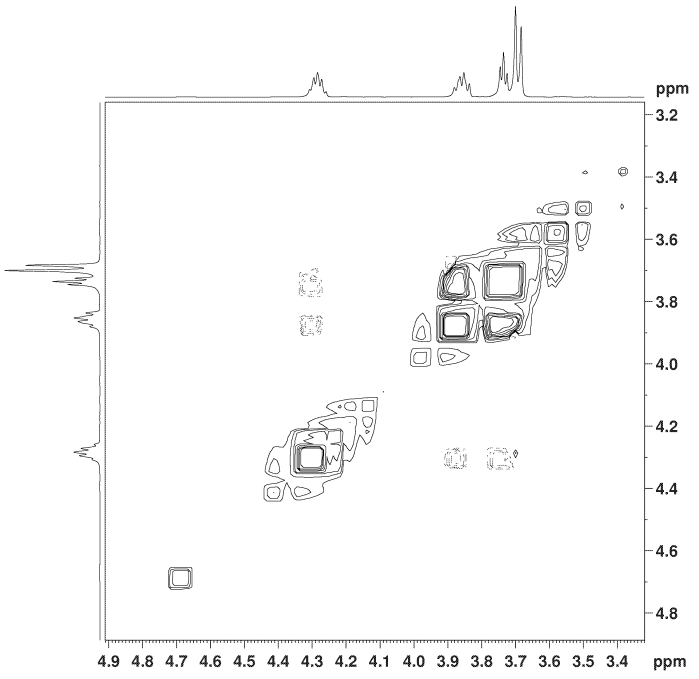


BRUKER

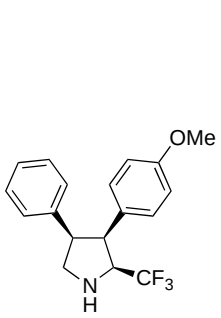
```
Current Data Parameters
NAME         MTC-13-80-C
EXPNO                  1
PROCNO                 1

F2 - Acquisition Parameters
Date_           20131112
Time               16.54
INSTRUM            spect
PROBHD   5 mm CPQNP 1H/
PULPROG          zgpg30
TD                65536
SOLVENT           CDCl3
NS                  100
DS                    0
SWH           36057.691 Hz
FIDRES            0.550197 Hz
AQ            0.9087659 sec
RG                  203
DW               13.867 usec
DE                18.00 usec
TE                298.0 K
D1            2.00000000 sec
D11           0.03000000 sec
TD0                   1

======== CHANNEL f1 ========
SFO1         150.9178981 MHz
NUC1                 13C
P1               11.25 usec
PLW1         30.00000000 W

======== CHANNEL f2 ========
SFO2         600.1324005 MHz
NUC2                 1H
CPDPRG[2         waltz16
PCPD2             80.00 usec
PLW2          9.00000000 W
PLW12         0.27562001 W
PLW13         0.17640001 W

F2 - Processing parameters
SI                32768
SF           150.9028173 MHz
WDW                  EM
SSB       0
LB                1.00 Hz
GB        0
PC                1.40
```



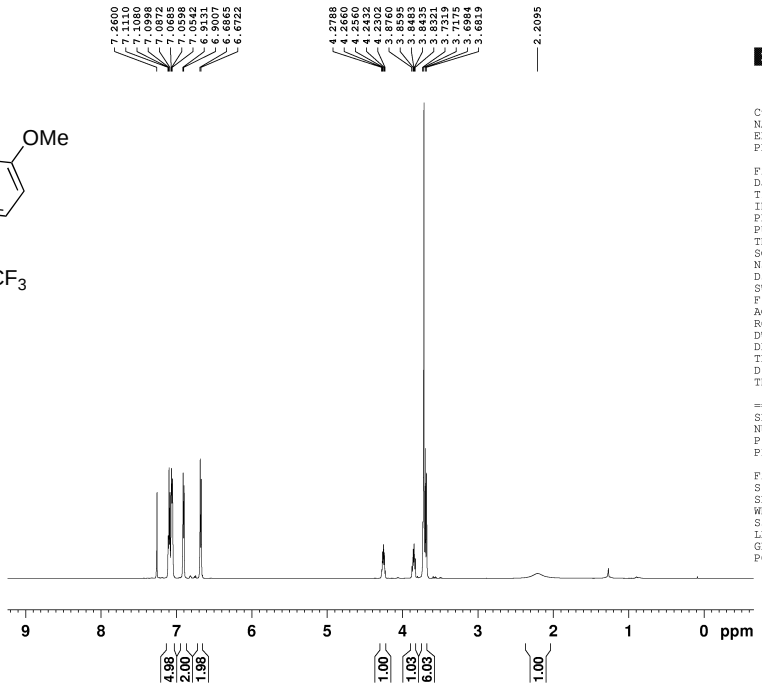

```
Current Data Parameters
NAME      MTC-13-80-COSY
EXPNO                  1
PROCNO                 1

F2 - Acquisition Parameters
Date_           20131112
Time               16.56
INSTRUM             spect
PROBHD   5 mm CPQNP 1H/
PULPROG      cosygpppqf
TD                 2048
SOLVENT            CDCl3
NS                    1
DS                    0
SWH           8012.820 Hz
FIDRES        3.912510 Hz
AQ            0.1277952 sec
RG                   64
DW              62.400 usec
DE              10.00 usec
TE                298.0 K
D0          0.00000300 sec
D1          2.00000000 sec
D11         0.03000000 sec
D12         0.00002000 sec
D13         0.00000400 sec
D16         0.00020000 sec
IN0         0.00012480 sec

======== CHANNEL f1 ========
SFO1         600.1336081 MHz
NUC1                   1H
P0                  13.50 usec
P1                  13.50 usec
P17               2500.00 usec
PLW1         9.00000000 W
PLW10        2.42639995 W

====== GRADIENT CHANNEL =====
GPNAM(1)       SMSQ10.100
GPZ1                10.00 %
P16               1000.00 usec

F1 - Acquisition parameters
TD                    102
SFO1           600.1336 MHz
FIDRES        78.557060 Hz
SW                13.352 ppm
FnMODE                  QF

F2 - Processing parameters
SI                    1024
SF           600.1300000 MHz
WDW                 QSINE
SSB                   0
LB                0 Hz
GB                    0
PC                 1.40

F1 - Processing parameters
SI                    1024
MC2                     QF
SF           600.1300000 MHz
WDW                 QSINE
SSB                   0
LB                0 Hz
GB                    0
```




```
Current Data Parameters
NAME      MTC-13-80-NOESY
EXPNO                  1
PROCNO                 1

F2 - Acquisition Parameters
Date_           20131113
Time                9.03
INSTRUM             spect
PROBHD   5 mm CPQNP 1H/
PULPROG      noesygpphpp
TD                 2048
SOLVENT            CDCl3
NS                    4
DS                    0
SWH           6009.615 Hz
FIDRES        2.934382 Hz
AQ            0.1703936 sec
RG                   64
DW              83.200 usec
DE              10.00 usec
TE                298.0 K
D0          0.00006601 sec
D1          2.00000000 sec
D8          0.30000001 sec
D11         0.03000000 sec
D12         0.00002000 sec
D16         0.00020000 sec
IN0         0.00016640 sec

======== CHANNEL f1 ========
SFO1         600.1327625 MHz
NUC1                   1H
P1                  13.50 usec
P2                  27.00 usec
P17               2500.00 usec
PLW1         9.00000000 W
PLW10        2.42639995 W

====== GRADIENT CHANNEL =====
GPNAM(1)       SMSQ10.100
GPZ1                40.00 %
P16               1000.00 usec

F1 - Acquisition parameters
TD                     98
SFO1           600.1328 MHz
FIDRES        61.322605 Hz
SW                10.014 ppm
FnMODE       States-TPPI

F2 - Processing parameters
SI                    1024
SF           600.1300000 MHz
WDW                 QSINE
SSB                   2
LB                0 Hz
GB                    0
PC                 1.00

F1 - Processing parameters
SI                    1024
MC2          States-TPPI
SF           600.1300000 MHz
WDW                 QSINE
SSB                   2
LB                0 Hz
GB                    0
```

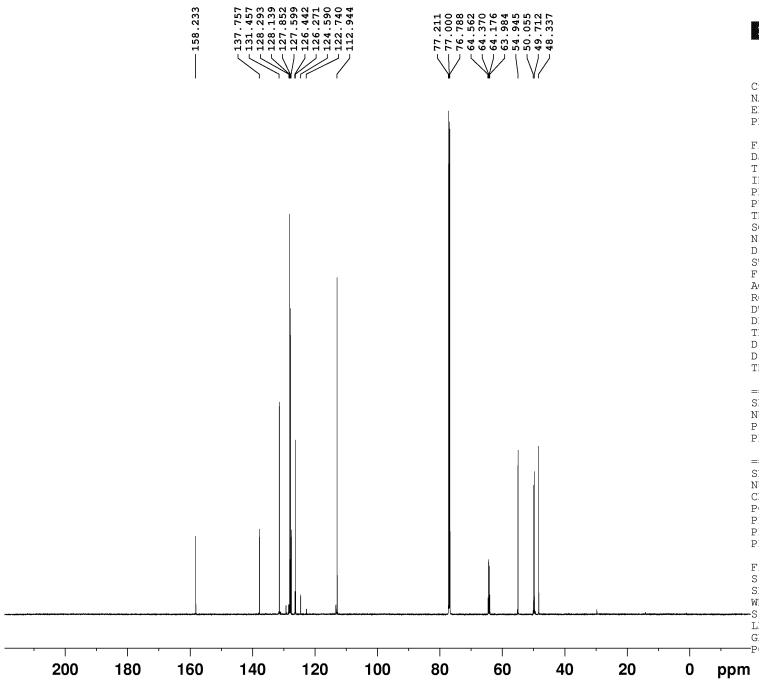


4e



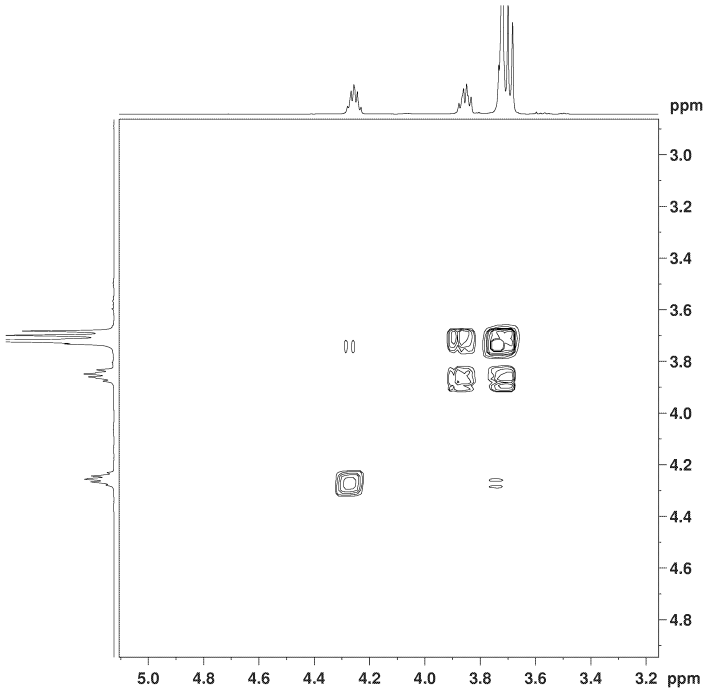
BRUKER

```
Current Data Parameters
NAME         MTC-13-93-H
EXPNO                  1
PROCNO                 1

F2 - Acquisition Parameters
Date_           20131121
Time                9.12
INSTRUM            spect
PROBHD   5 mm CPQNP 1H/
PULPROG             zg30
TD                 65536
SOLVENT            CDCl3
NS                    16
DS                     0
SWH            12019.230 Hz
FIDRES             0.183399 Hz
AQ             2.7262976 sec
RG                    32
DW                41.600 usec
DE                10.00 usec
TE                298.0 K
D1             1.00000000 sec
TD0                    1

======== CHANNEL f1 ========
SFO1          600.1337060 MHz
NUC1                  1H
P1                 13.50 usec
PLW1           9.00000000 W

F2 - Processing parameters
SI                 65536
SF            600.1300134 MHz
WDW                   EM
SSB       0
LB                 0.30 Hz
GB        0
PC                 1.00
```

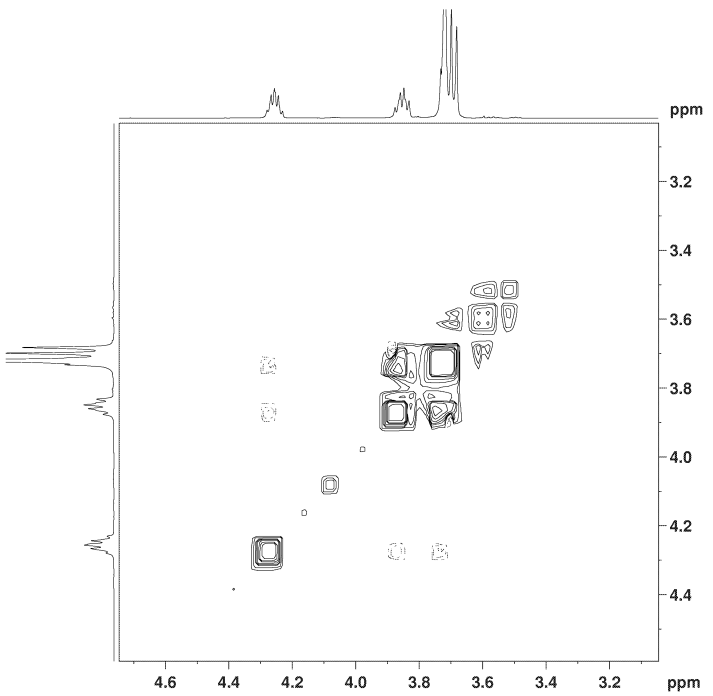


BRUKER

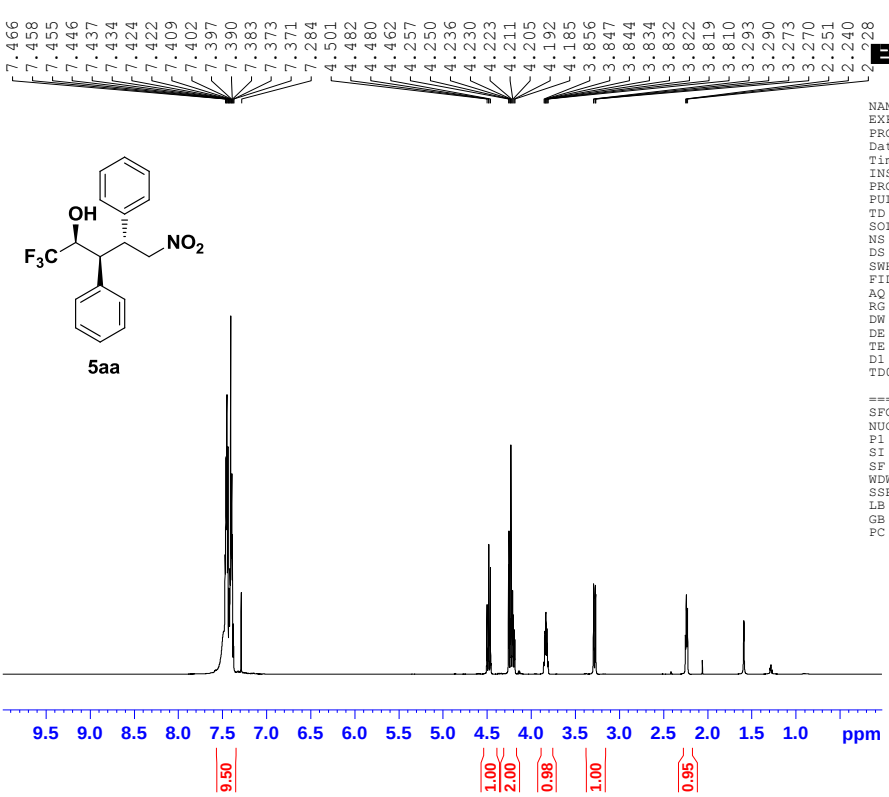
```
Current Data Parameters
NAME         MTC-13-93-C
EXPNO                  1
PROCNO                 1

F2 - Acquisition Parameters
Date_           20131121
Time                9.15
INSTRUM            spect
PROBHD   5 mm CPQNP 1H/
PULPROG          zgpg30
TD                 65536
SOLVENT            CDCl3
NS                   120
DS                     0
SWH            36057.691 Hz
FIDRES             0.550197 Hz
AQ             0.9087659 sec
RG                   203
DW                13.867 usec
DE                18.00 usec
TE                298.0 K
D1             2.00000000 sec
D11            0.03000000 sec
TD0                    1

======== CHANNEL f1 ========
SFO1          150.9178981 MHz
NUC1                  13C
P1                 11.25 usec
PLW1          30.00000000 W

======== CHANNEL f2 ========
SFO2          600.1324005 MHz
NUC2                  1H
CPDPRG[2          waltz16
PCPD2              80.00 usec
PLW2           9.00000000 W
PLW12          0.27562001 W
PLW13          0.17640001 W

F2 - Processing parameters
SI                 32768
SF            150.9028184 MHz
WDW                   EM
SSB       0
LB                 1.00 Hz
GB        0
PC                 1.40
```



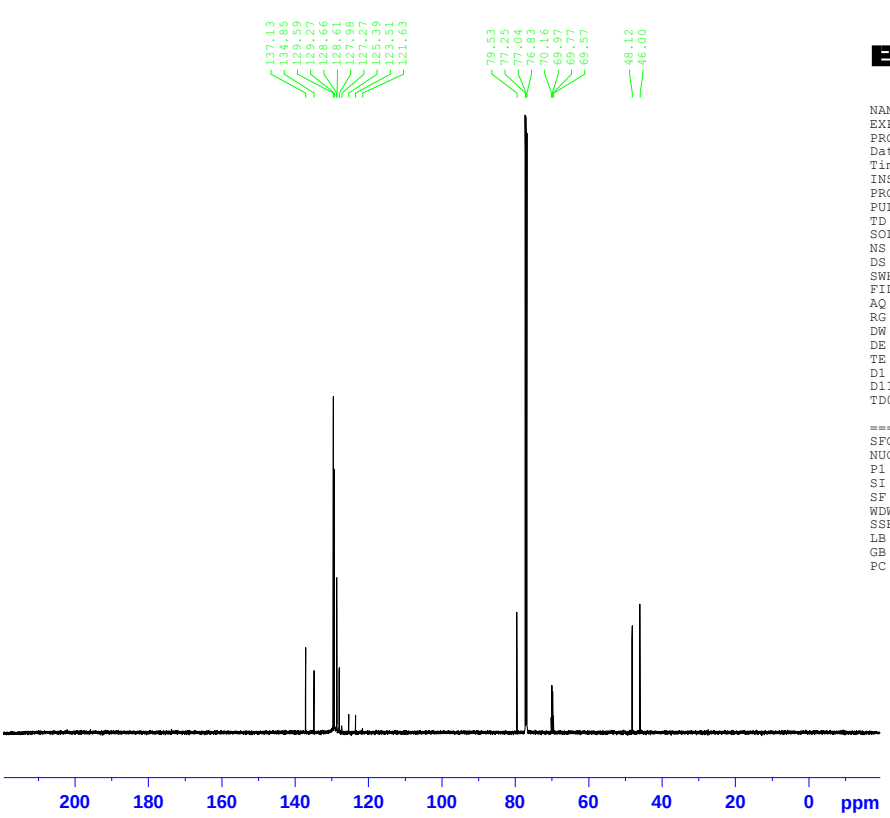

```
Current Data Parameters
NAME      MTC-13-93-COSY
EXPNO                  1
PROCNO                 1

F2 - Acquisition Parameters
Date_           20131121
Time               9.19
INSTRUM           spect
PROBHD   5 mm CPQNP 1H/
PULPROG      cosygpppqf
TD                 2048
SOLVENT            CDCl3
NS                    1
DS                    0
SWH            8012.820 Hz
FIDRES         3.912510 Hz
AQ             0.1277952 sec
RG                   64
DW               62.400 usec
DE               10.00 usec
TE                298.0 K
D0            0.00000300 sec
D1            2.00000000 sec
D11           0.03000000 sec
D12           0.00002000 sec
D13           0.00000400 sec
D16           0.00020000 sec
IN0           0.00012480 sec

======== CHANNEL f1 ========
SFO1          600.1336081 MHz
NUC1                  1H
P0                  13.50 usec
P1                  13.50 usec
P17               2500.00 usec
PLW1         9.00000000 W
PLW10        2.42639995 W

====== GRADIENT CHANNEL =====
GPNAM(1)       SMSQ10.100
GPZ1                10.00 %
P16               1000.00 usec

F1 - Acquisition parameters
TD                   128
SFO1           600.1336 MHz
FIDRES         62.600159 Hz
SW                13.352 ppm
FnMODE                QF

F2 - Processing parameters
SI                   1024
SF            600.1300000 MHz
WDW                  QSINE
SSB                   0
LB                 0 Hz
GB                    0
PC                  1.40

F1 - Processing parameters
SI                   1024
MC2                    QF
SF            600.1300000 MHz
WDW                  QSINE
SSB                   0
LB                 0 Hz
GB                    0
```



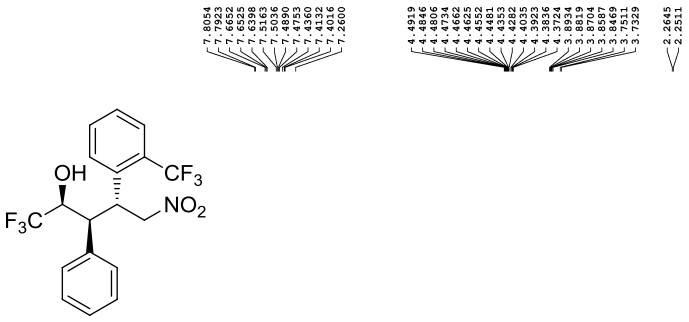

```
Current Data Parameters
NAME      MTC-13-93-NOESY
EXPNO                  1
PROCNO                 1

F2 - Acquisition Parameters
Date_           20131121
Time               17.21
INSTRUM           spect
PROBHD   5 mm CPQNP 1H/
PULPROG      noesygpphpp
TD                 2048
SOLVENT            CDCl3
NS                    4
DS                    0
SWH            6009.615 Hz
FIDRES         2.934382 Hz
AQ             0.1703936 sec
RG                   64
DW               83.200 usec
DE               10.00 usec
TE                298.0 K
D0            0.00006601 sec
D1            2.00000000 sec
D8            0.30000001 sec
D11           0.03000000 sec
D12           0.00002000 sec
D16           0.00020000 sec
IN0           0.00016640 sec

======== CHANNEL f1 ========
SFO1          600.1327625 MHz
NUC1                  1H
P1                  13.50 usec
P2                  27.00 usec
P17               2500.00 usec
PLW1         9.00000000 W
PLW10        2.42639995 W

====== GRADIENT CHANNEL =====
GPNAM(1)       SMSQ10.100
GPZ1                40.00 %
P16               1000.00 usec

F1 - Acquisition parameters
TD                   106
SFO1           600.1328 MHz
FIDRES         56.694485 Hz
SW                10.014 ppm
FnMODE       States-TPPI

F2 - Processing parameters
SI                   1024
SF            600.1300000 MHz
WDW                  QSINE
SSB                   2
LB                 0 Hz
GB                    0
PC                  1.00

F1 - Processing parameters
SI                   1024
MC2          States-TPPI
SF            600.1300000 MHz
WDW                  QSINE
SSB                   2
LB                 0 Hz
GB                    0
```



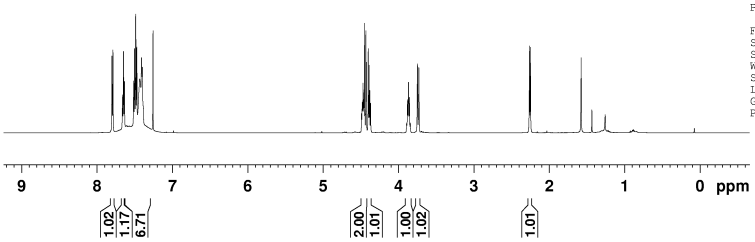

```
NAME              QX12991-2
EXPNO                     1
PROCNO                    1
Date_              20131230
Time                 13.21
INSTRUM              spect
PROBHD    5 mm CPQNP 1H/
PULPROG               zg30
TD                   65536
SOLVENT              CDCl3
NS                      13
DS                       2
SWH              12019.230 Hz
FIDRES            0.183399 Hz
AQ                2.7263477 sec
RG                      32
DW                  41.600 usec
DE                  10.00 usec
TE                  298.0 K
D1              1.00000000 sec
TD0                       1

======== CHANNEL f1 ========
SFO1            600.1337060 MHz
NUC1                    1H
P1                   13.50 usec
SI                   65536
SF              600.1300000 MHz
WDW                     EM
SSB                      0
LB                    0.30 Hz
GB                       0
PC                    1.00
```



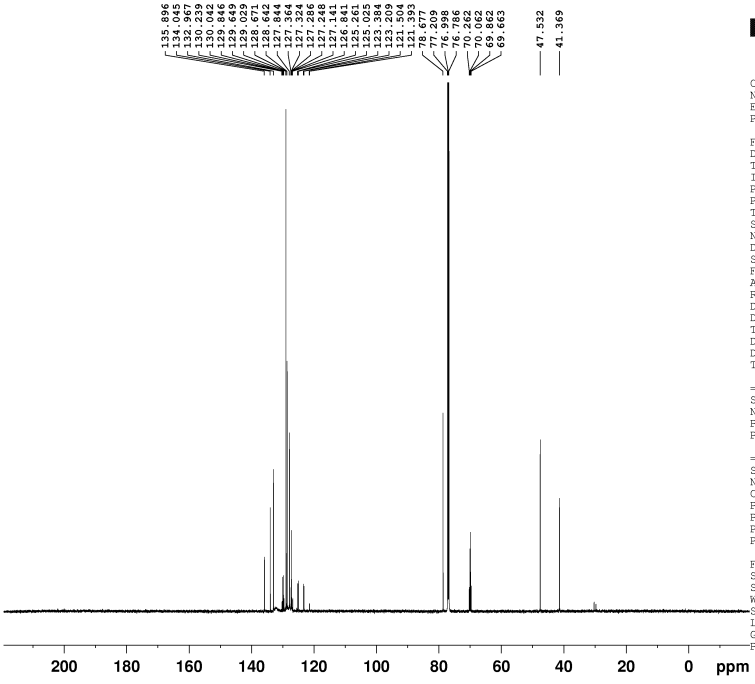

```
NAME              QX12991-2
EXPNO                     2
PROCNO                    1
Date_              20131230
Time                 13.25
INSTRUM              spect
PROBHD    5 mm CPQNP 1H/
PULPROG             zgpg30
TD                   65536
SOLVENT              CDCl3
NS                      46
DS                       4
SWH              36057.691 Hz
FIDRES            0.550197 Hz
AQ                0.9088159 sec
RG                     203
DW                  13.867 usec
DE                  18.00 usec
TE                  298.0 K
D1              2.00000000 sec
D11             0.03000000 sec
TD0                       1

======== CHANNEL f1 ========
SFO1            150.9178981 MHz
NUC1                    13C
P1                   11.25 usec
SI                   32768
SF              150.9028090 MHz
WDW                     EM
SSB                      0
LB                    1.00 Hz
GB                       0
PC                    1.40
```



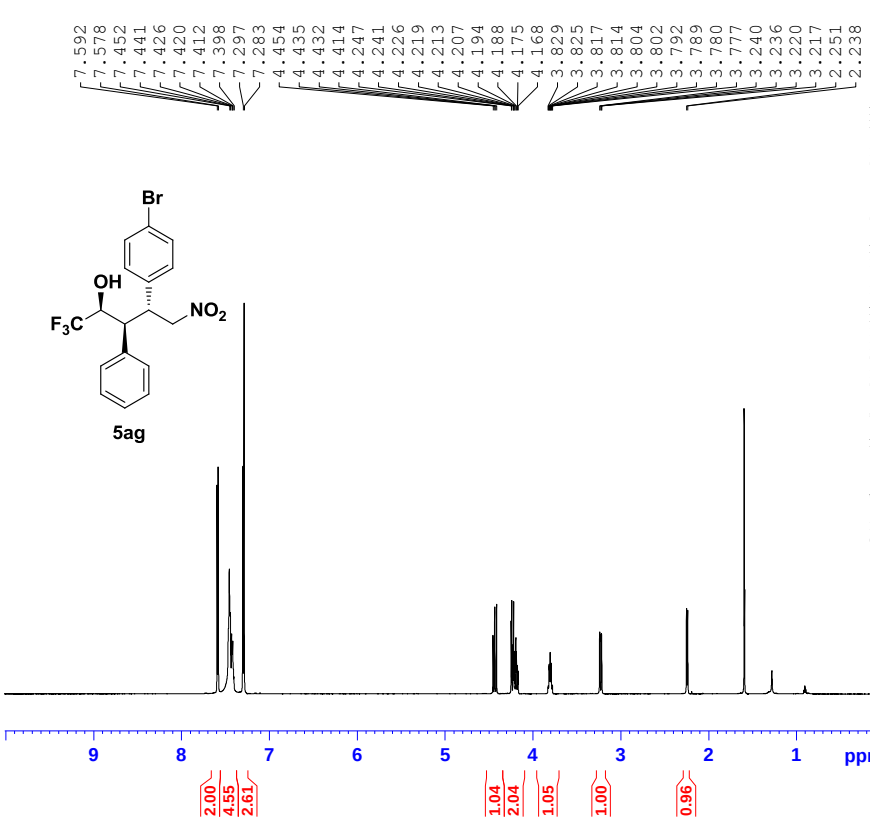
5ad




```
Current Data Parameters
NAME          MTC-13-96-H
EXPNO                   1
PROCNO                  1

F2 - Acquisition Parameters
Date_            20131122
Time                11.13
INSTRUM             spect
PROBHD   5 mm CPQNP 1H/
PULPROG             zg30
TD                 65536
SOLVENT            CDCl3
NS                    16
DS                     0
SWH            12019.230 Hz
FIDRES             0.183399 Hz
AQ             2.7262976 sec
RG                    57
DW                41.600 usec
DE                10.00 usec
TE                298.0 K
D1             1.00000000 sec
TD0                    1

======== CHANNEL f1 ========
SFO1          600.1337060 MHz
NUC1                  1H
P1                 13.50 usec
PLW1           9.00000000 W

F2 - Processing parameters
SI                 65536
SF            600.1300138 MHz
WDW                   EM
SSB       0
LB                 0.30 Hz
GB        0
PC                 1.00
```

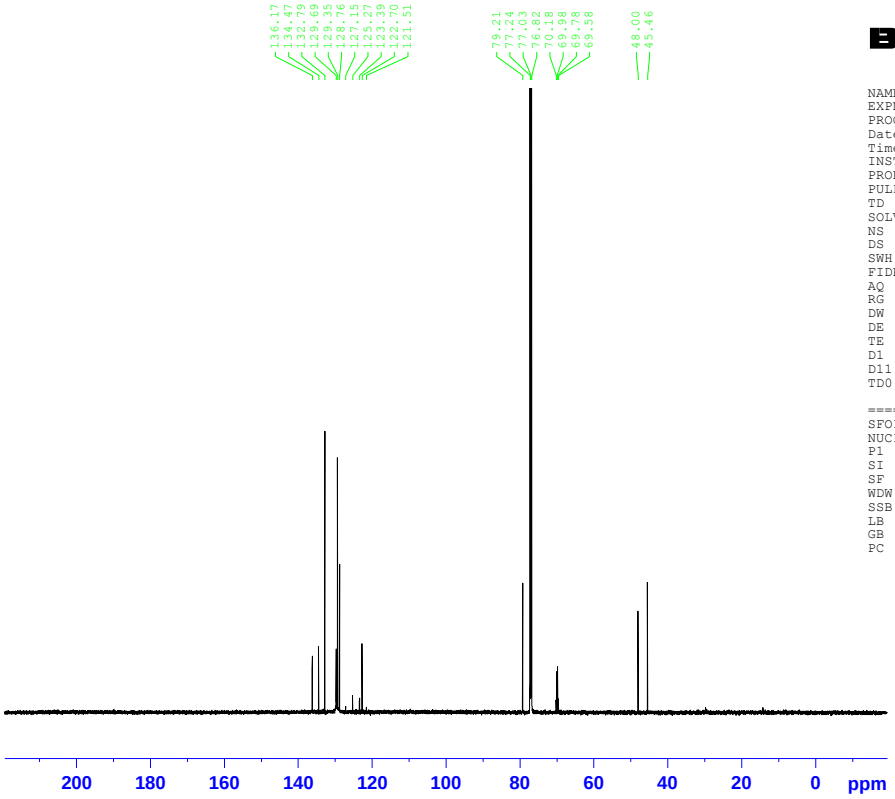



```
Current Data Parameters
NAME          MTC-13-96-C
EXPNO                   1
PROCNO                  1

F2 - Acquisition Parameters
Date_            20131122
Time                11.16
INSTRUM             spect
PROBHD   5 mm CPQNP 1H/
PULPROG          zgpg30
TD                 65536
SOLVENT            CDCl3
NS                   100
DS                     0
SWH            36057.691 Hz
FIDRES             0.550197 Hz
AQ             0.9087659 sec
RG                   203
DW                13.867 usec
DE                18.00 usec
TE                298.0 K
D1             2.00000000 sec
D11            0.03000000 sec
TD0                    1

======== CHANNEL f1 ========
SFO1          150.9178981 MHz
NUC1                  13C
P1                 11.25 usec
PLW1          30.00000000 W

======== CHANNEL f2 ========
SFO2          600.1324005 MHz
NUC2                  1H
CPDPRG[2          waltz16
PCPD2              80.00 usec
PLW2           9.00000000 W
PLW12          0.27562001 W
PLW13          0.17640001 W

F2 - Processing parameters
SI                 32768
SF            150.9028151 MHz
WDW                   EM
SSB       0
LB                 1.00 Hz
GB        0
PC                 1.40
```



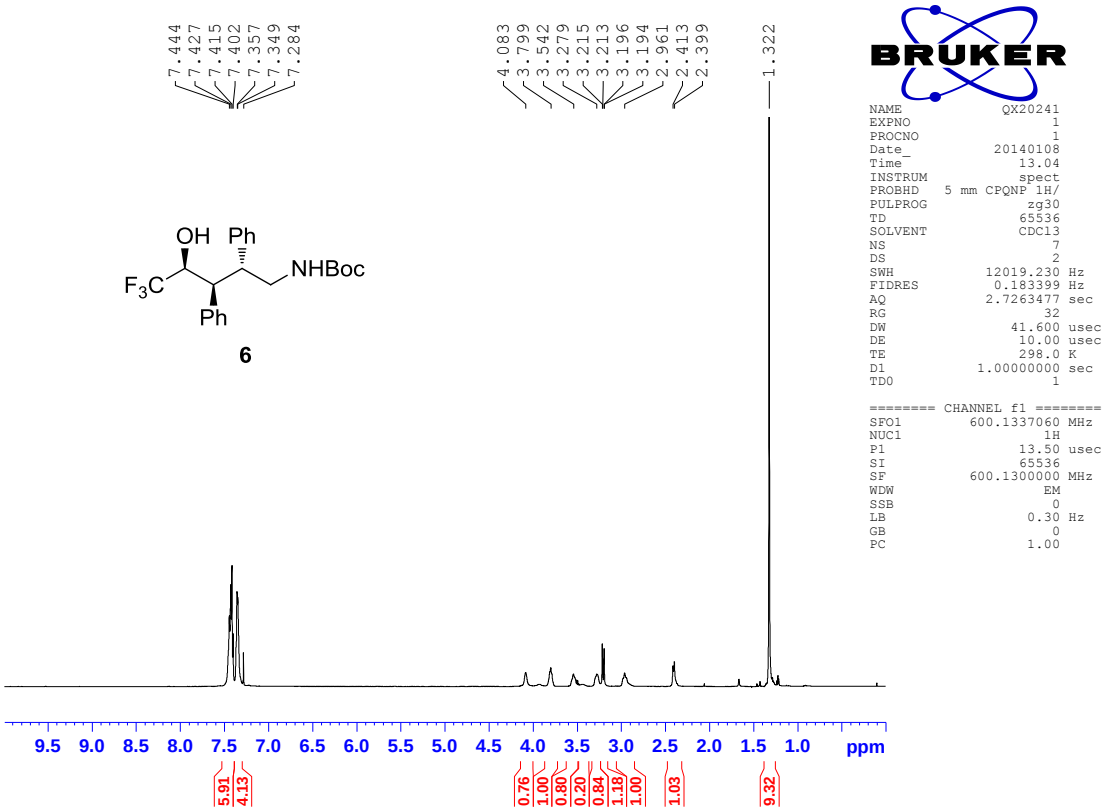
BRUKER

NAME	QX12761
EXPNO	1
PROCNO	1
Date_	20131104
Time	18.32
INSTRUM	spect
PROBHD	5 mm CPQNP 1H/
PULPROG	zg30
TD	65536
SOLVENT	CDCl3
NS	16
DS	2
SWH	12019.230 Hz
FIDRES	0.183399 Hz
AQ	2.7263477 sec
RG	64
DW	41.600 usec
DE	10.00 usec
TE	298.0 K
D1	1.00000000 sec
TD0	1
======== CHANNEL f1 ========	
SFO1	600.1337060 MHz
NUC1	1H
P1	13.50 usec
SI	65536
SF	600.1300000 MHz
WDW	EM
SSB	0
LB	0.30 Hz
GB	0
PC	1.00



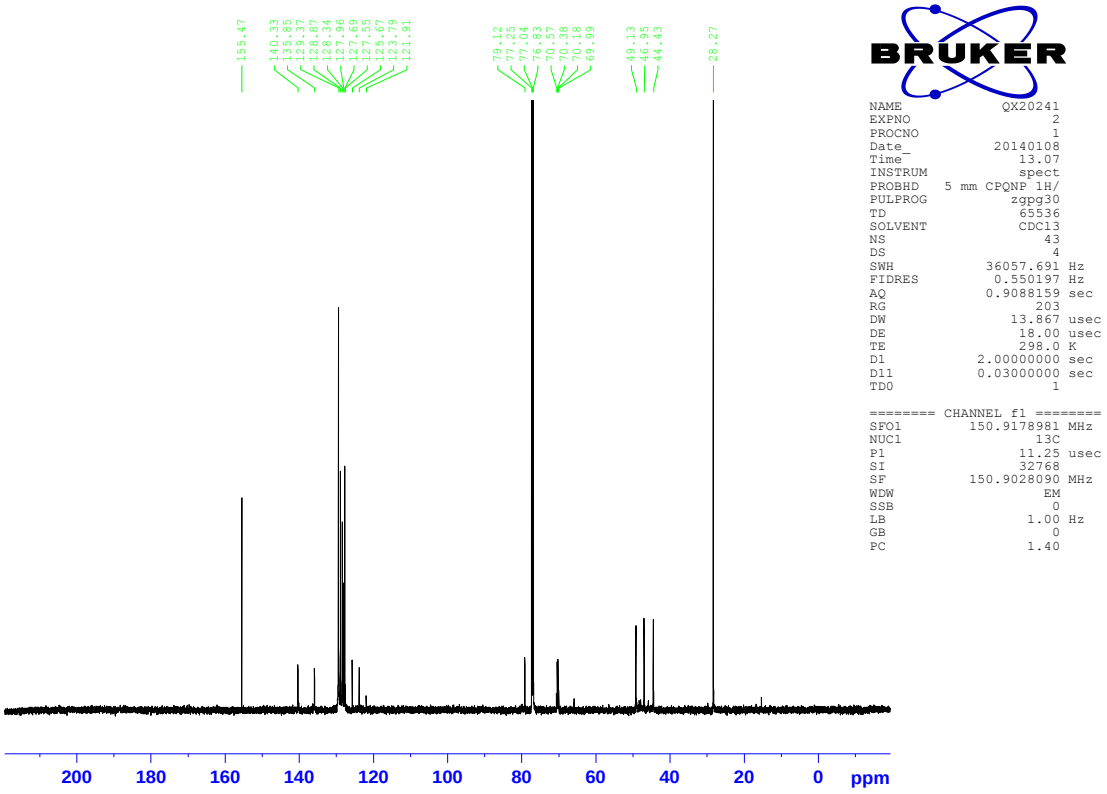
BRUKER

NAME	QX12761
EXPNO	2
PROCNO	1
Date_	20131104
Time	17.22
INSTRUM	spect
PROBHD	5 mm CPQNP 1H/
PULPROG	zgpg30
TD	65536
SOLVENT	CDCl3
NS	162
DS	4
SWH	36057.691 Hz
FIDRES	0.550197 Hz
AQ	0.9088159 sec
RG	203
DW	13.867 usec
DE	18.00 usec
TE	298.0 K
D1	2.00000000 sec
D11	0.03000000 sec
TD0	1
======== CHANNEL f1 ========	
SFO1	150.9178981 MHz
NUC1	13C
P1	11.25 usec
SI	32768
SF	150.9028090 MHz
WDW	EM
SSB	0
LB	1.00 Hz
GB	0
PC	1.40



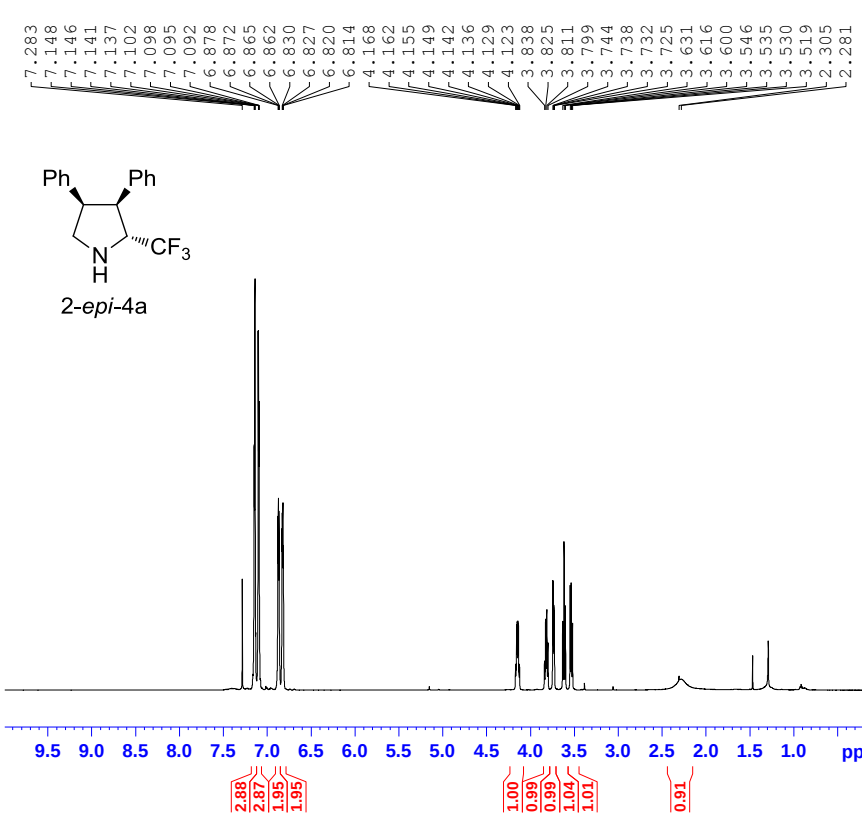
BRUKER

```
NAME                QX20241
EXPNO                     1
PROCNO                    1
Date_              20140108
Time                 13.04
INSTRUM              spect
PROBHD    5 mm CPQNP 1H/
PULPROG               zg30
TD                   65536
SOLVENT              CDCl3
NS                        7
DS                        2
SWH              12019.230 Hz
FIDRES           0.183399 Hz
AQ               2.7263477 sec
RG                       32
DW                  41.600 usec
DE                  10.00 usec
TE                  298.0 K
D1              1.00000000 sec
TD0                       1

======== CHANNEL f1 ========
SFO1          600.1337060 MHz
NUC1                    1H
P1                   13.50 usec
SI                   65536
SF            600.1300000 MHz
WDW                     EM
SSB                      0
LB                    0.30 Hz
GB                       0
PC                    1.00
```



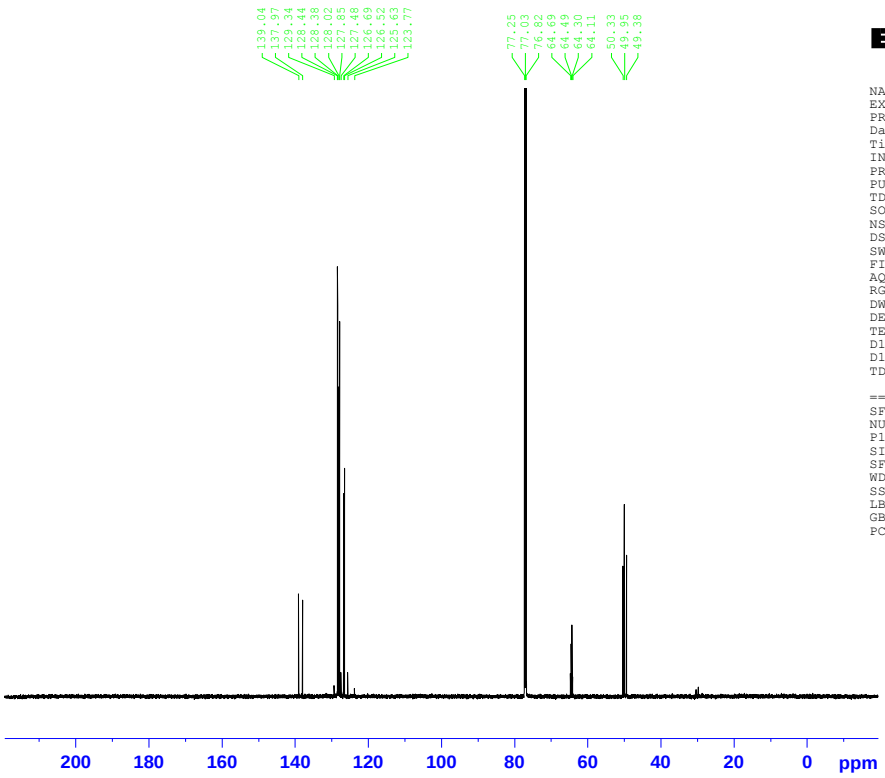
BRUKER

```
NAME                QX20241
EXPNO                     2
PROCNO                    1
Date_              20140108
Time                 13.07
INSTRUM              spect
PROBHD    5 mm CPQNP 1H/
PULPROG           zgpg30
TD                   65536
SOLVENT              CDCl3
NS                       43
DS                        4
SWH              36057.691 Hz
FIDRES           0.550197 Hz
AQ               0.9088159 sec
RG                      203
DW                  13.867 usec
DE                  18.00 usec
TE                  298.0 K
D1              2.00000000 sec
D11             0.03000000 sec
TD0                       1

======== CHANNEL f1 ========
SFO1          150.9178981 MHz
NUC1                    13C
P1                   11.25 usec
SI                   32768
SF            150.9028090 MHz
WDW                     EM
SSB                      0
LB                    1.00 Hz
GB                       0
PC                    1.40
```



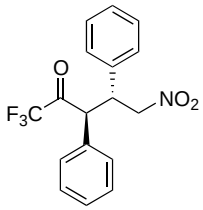
BRUKER

```
NAME          QX20192-HC1-2
EXPNO                     1
PROCNO                    1
Date_              20131218
Time                  17.26
INSTRUM               spect
PROBHD    5 mm CPQNP 1H/
PULPROG                zg30
TD                    65536
SOLVENT               CDCl3
NS                       12
DS                        2
SWH              12019.230 Hz
FIDRES               0.183399 Hz
AQ                2.7263477 sec
RG                       32
DW                  41.600 usec
DE                  10.00 usec
TE                  298.2 K
D1               1.00000000 sec
TD0                       1

======== CHANNEL f1 ========
SFO1            600.1337060 MHz
NUC1                    1H
P1                   13.50 usec
SI                    65536
SF              600.1300000 MHz
WDW                     EM
SSB                      0
LB                    0.30 Hz
GB                       0
PC                    1.00
```

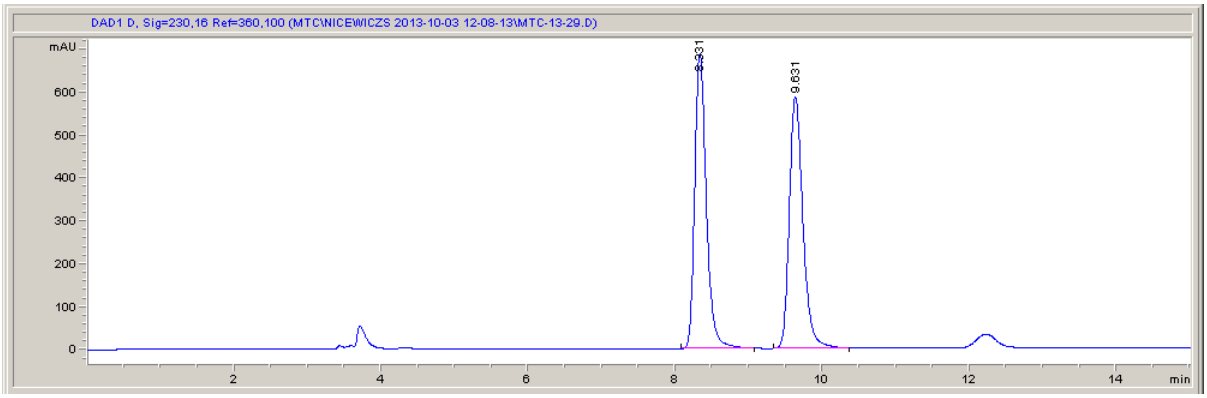


BRUKER

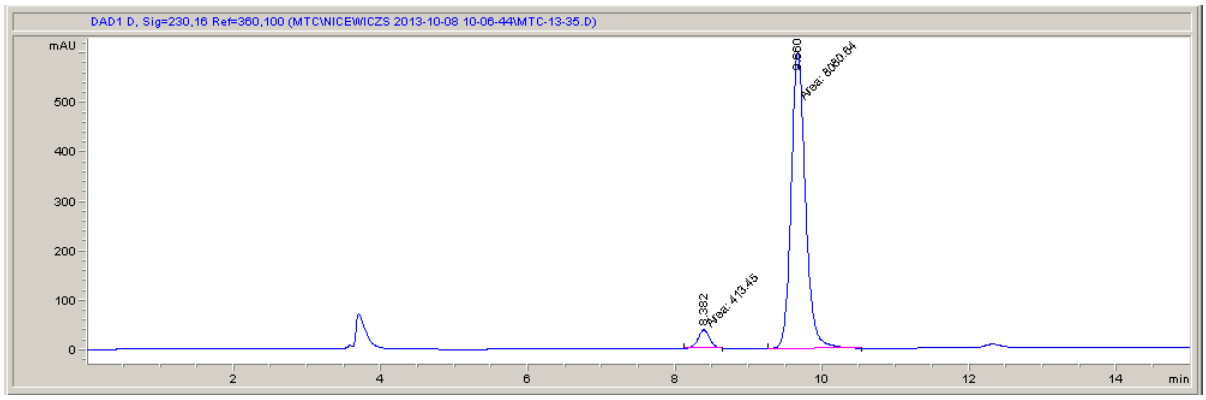
```
NAME          QX20192-HC1-2
EXPNO                     2
PROCNO                    1
Date_              20131218
Time                  17.18
INSTRUM               spect
PROBHD    5 mm CPQNP 1H/
PULPROG             zgpg30
TD                    65536
SOLVENT               CDCl3
NS                       59
DS                        4
SWH              36057.691 Hz
FIDRES               0.550197 Hz
AQ                0.9088159 sec
RG                      203
DW                  13.867 usec
DE                  18.00 usec
TE                  298.1 K
D1               2.00000000 sec
D11              0.03000000 sec
TD0                       1

======== CHANNEL f1 ========
SFO1            150.9178981 MHz
NUC1                    13C
P1                   11.25 usec
SI                    32768
SF              150.9028090 MHz
WDW                     EM
SSB                      0
LB                    1.00 Hz
GB                       0
PC                    1.40
```



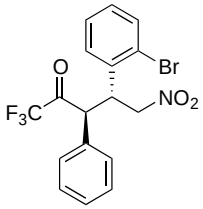
3aa



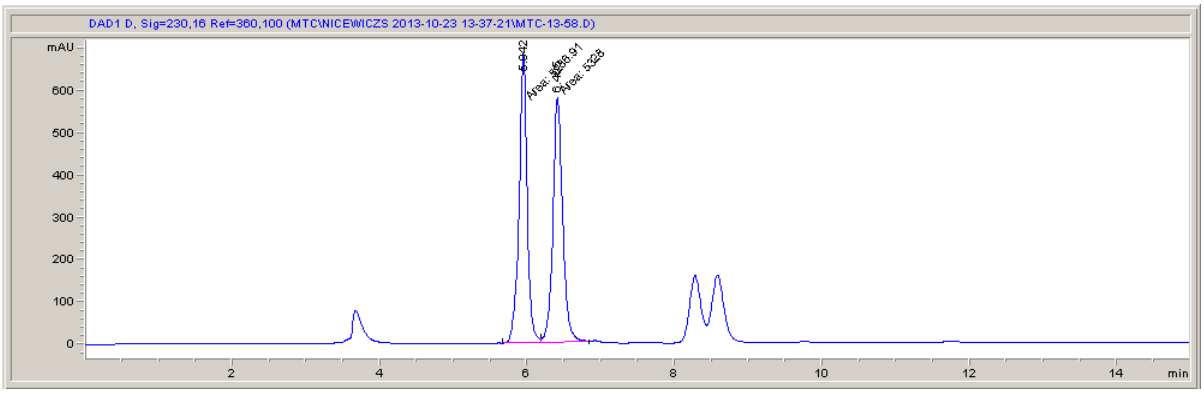
#	Time	Area	Height	Width	Area%	Symmetry
1	8.331	7511.4	685.6	0.1658	50.014	0.72
2	9.631	7507.2	585	0.1957	49.986	0.737



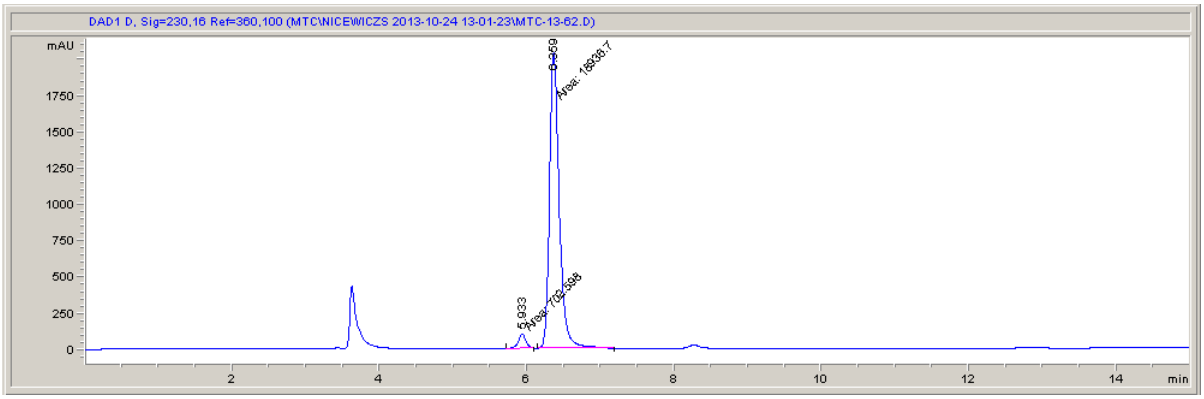
#	Time	Area	Height	Width	Area%	Symmetry
1	8.382	413.4	37.4	0.1841	4.868	0.977
2	9.66	8080.6	597.7	0.2253	95.132	0.822



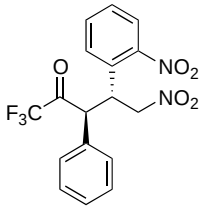
3ab



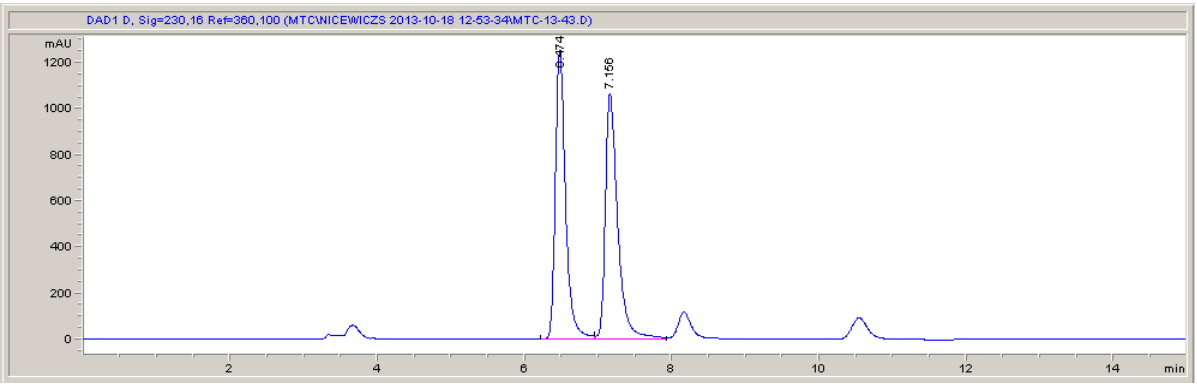
#	Time	Area	Height	Width	Area%	Symmetry
1	5.942	5286.9	681.8	0.1292	49.806	1.087
2	6.405	5328	580.4	0.153	50.194	0.85



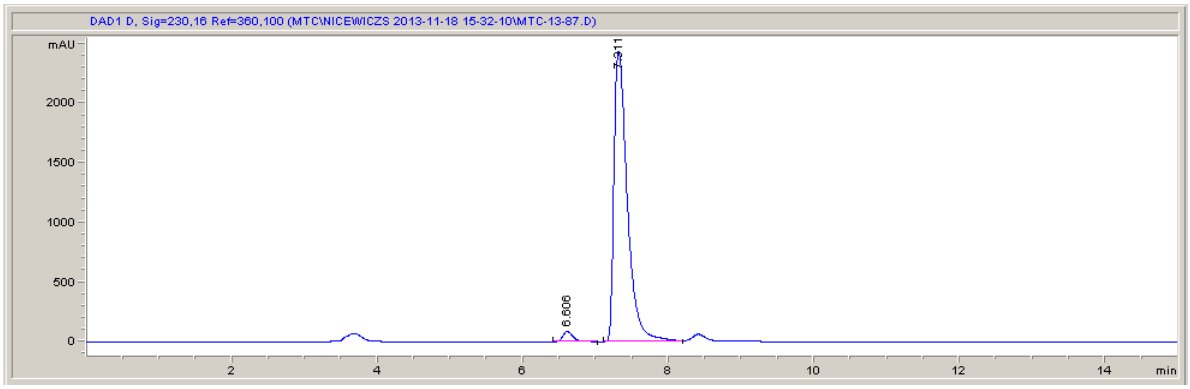
#	Time	Area	Height	Width	Area%	Symmetry
1	5.933	702.6	98.7	0.1186	3.578	1.074
2	6.359	18936.7	2034.3	0.1551	96.422	0.683



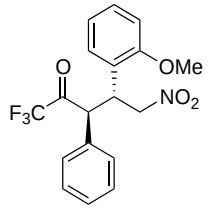
3ac



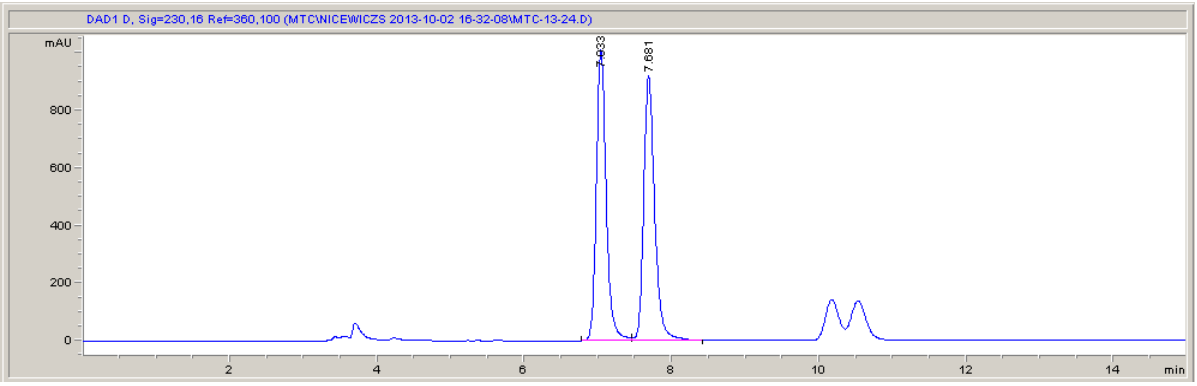
#	Time	Area	Height	Width	Area%	Symmetry
1	6.474	12078.8	1252.3	0.1445	49.256	0.693
2	7.156	12443.6	1066	0.1762	50.744	0.553



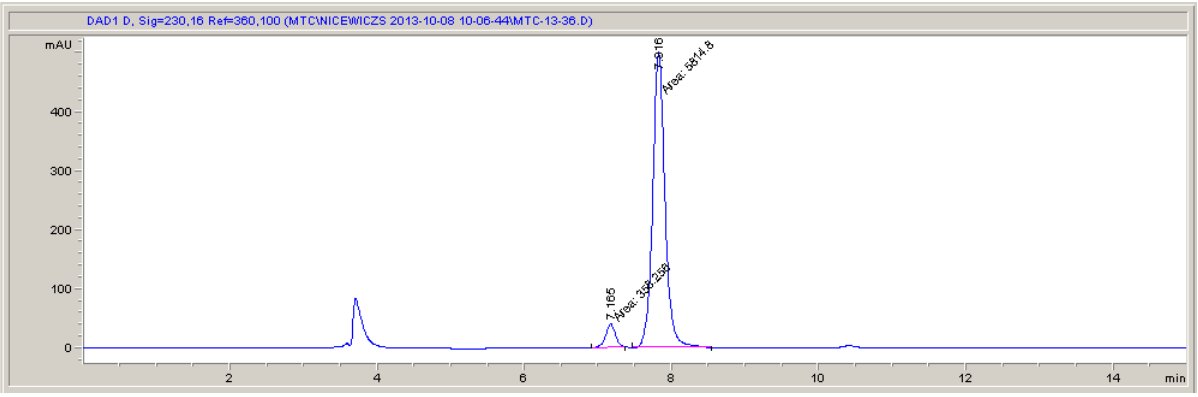
#	Time	Area	Height	Width	Area%	Symmetry
1	6.606	823.8	86	0.1438	2.597	0.712
2	7.311	30895.5	2430.6	0.1923	97.403	0.469



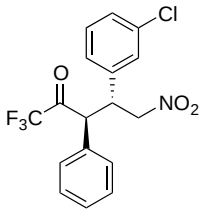
3ae



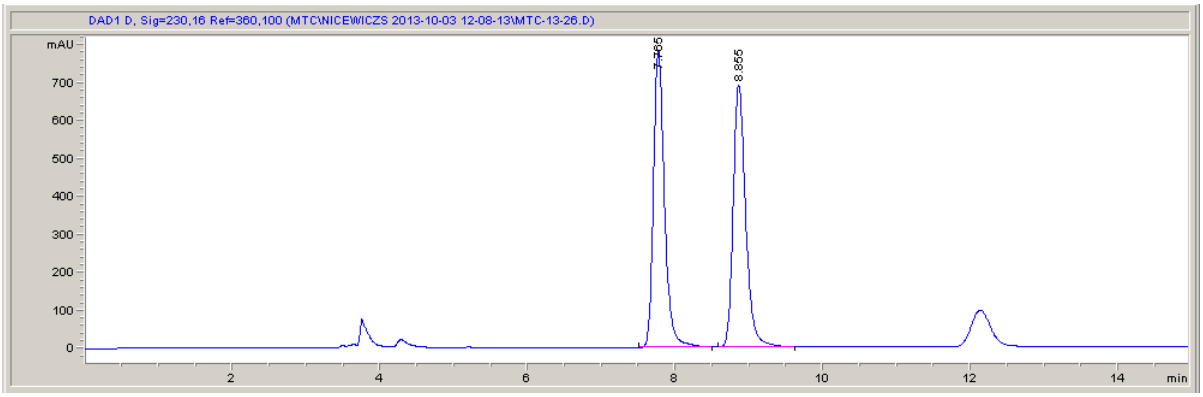
#	Time	Area	Height	Width	Area%	Symmetry
1	7.033	9380.6	1010.2	0.1403	49.681	0.79
2	7.681	9501	919.6	0.1585	50.319	0.726



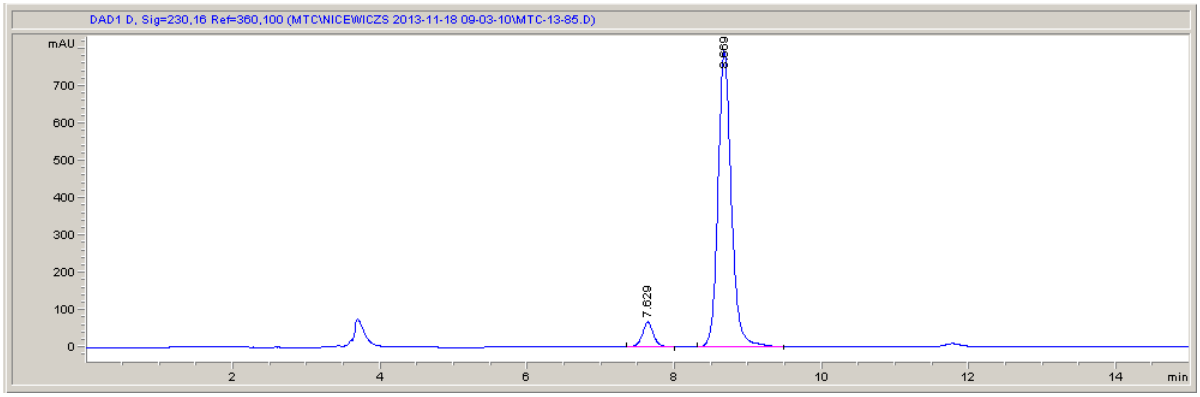
#	Time	Area	Height	Width	Area%	Symmetry
1	7.165	358.3	39.3	0.1518	5.804	1.069
2	7.816	5814.8	500.7	0.1935	94.196	0.877



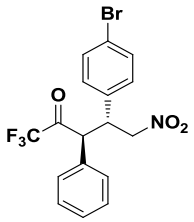
3af



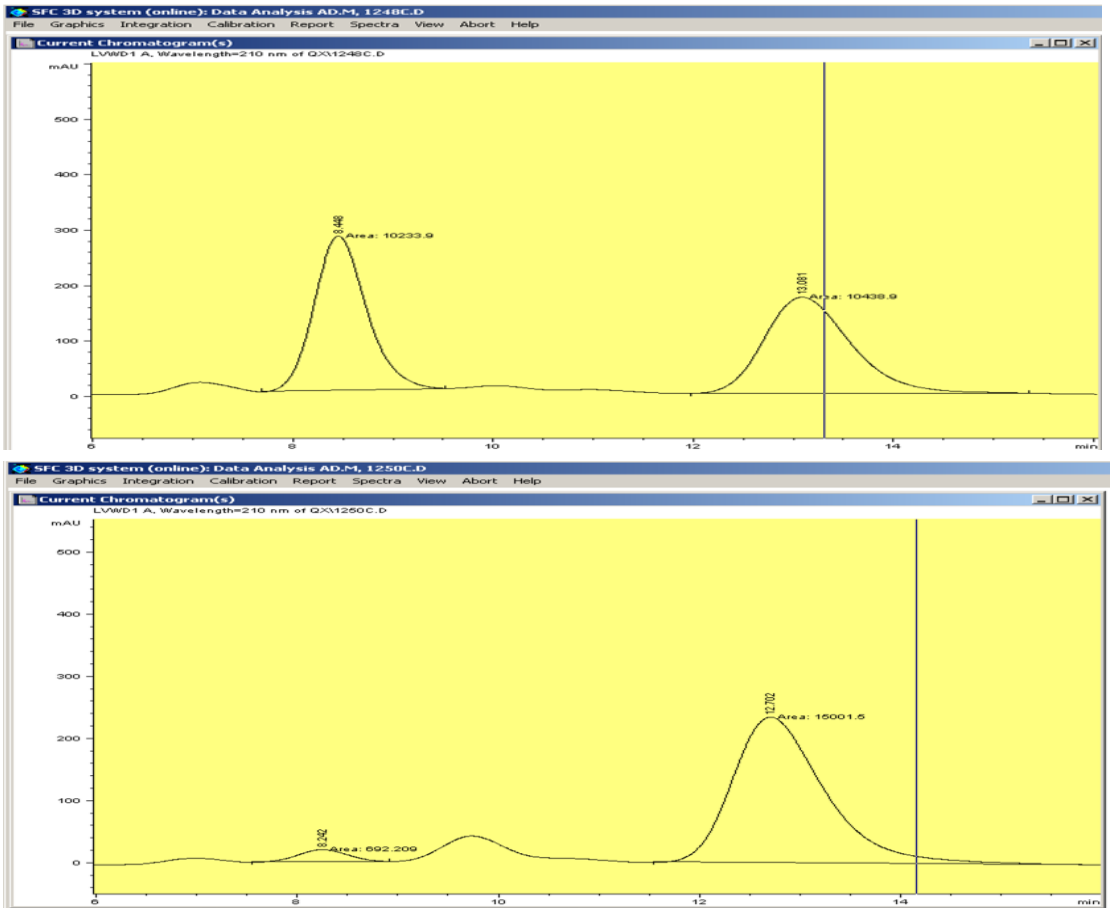
#	Time	Area	Height	Width	Area%	Symmetry
1	7.765	8140.4	779.3	0.1579	50.008	0.718
2	8.855	8137.7	689.8	0.1796	49.992	0.728



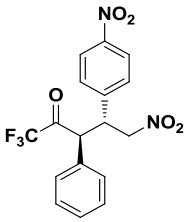
#	Time	Area	Height	Width	Area%	Symmetry
1	7.629	704.3	67.3	0.158	6.576	0.922
2	8.669	10004.7	792.1	0.1914	93.424	0.807



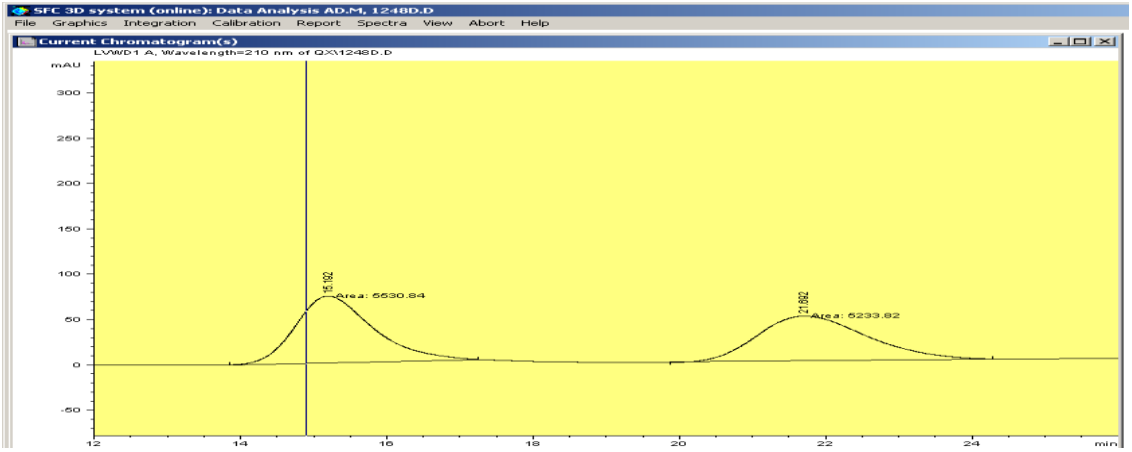
3ag

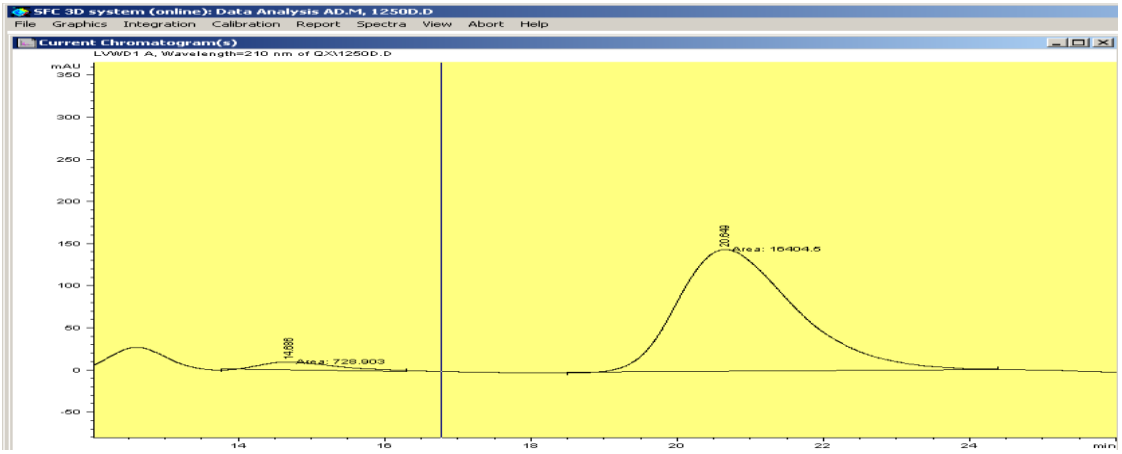


Peak #	RT [min]	Type	Width [min]	Area [mAU*sec]	Height [mAU]	Area %
1	8.242	MM	0.583	692.20898	19.78505	4.4107
2	12.702	MM	1.067	15001.52539	234.33128	95.5893
Totals :				15693.73437	254.11633	

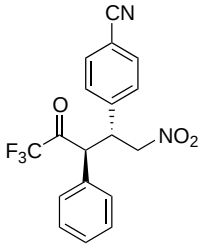


3ah

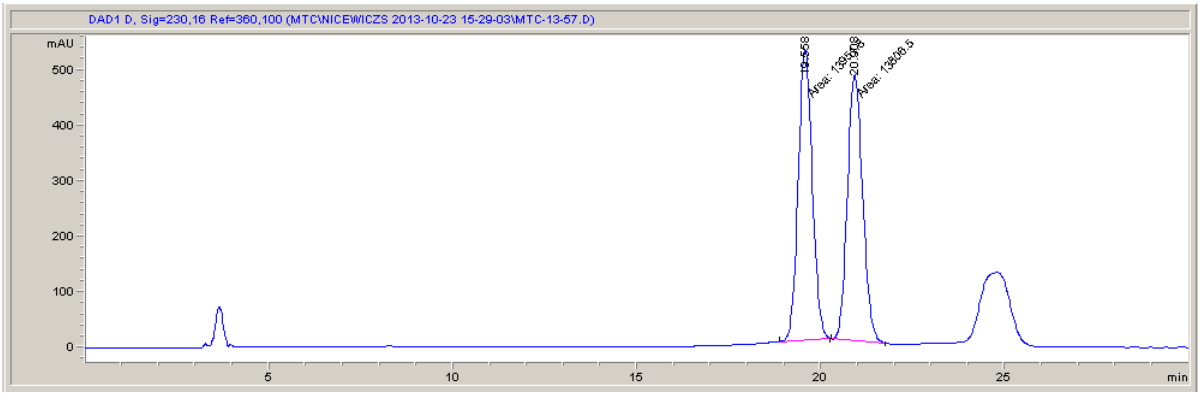




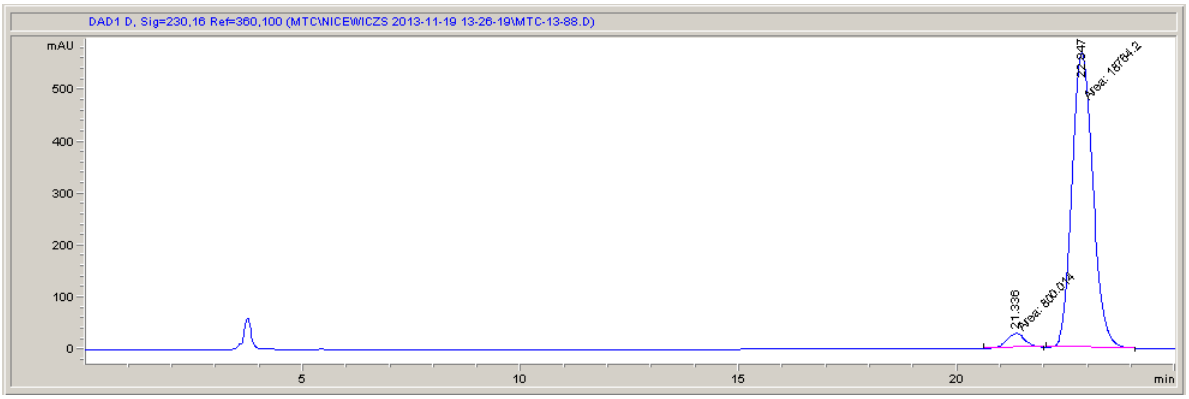
Peak #	RT [min]	Type	Width [min]	Area [mAU*sec]	Height [mAU]	Area %
1	14.686	MM	1.255	728.90271	9.68124	4.2543
2	20.649	MM	1.885	16404.48242	145.04874	95.7457
Totals :				17133.38477	154.72998	



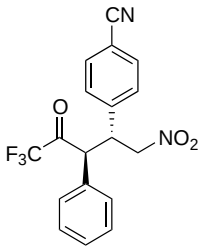
3ai



#	Time	Area	Height	Width	Area%	Symmetry
1	19.558	13951.8	521.3	0.446	50.262	0.929
2	20.908	13806.5	479.3	0.4801	49.738	0.875

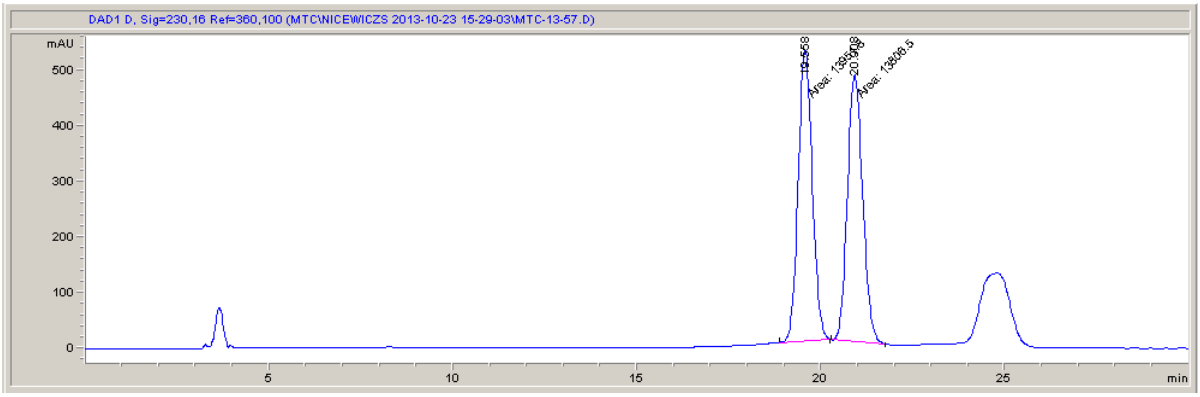


#	Time	Area	Height	Width	Area%	Symmetry
1	21.336	800	27.4	0.4861	4.089	0.954
2	22.847	18764.2	567	0.5516	95.911	0.822

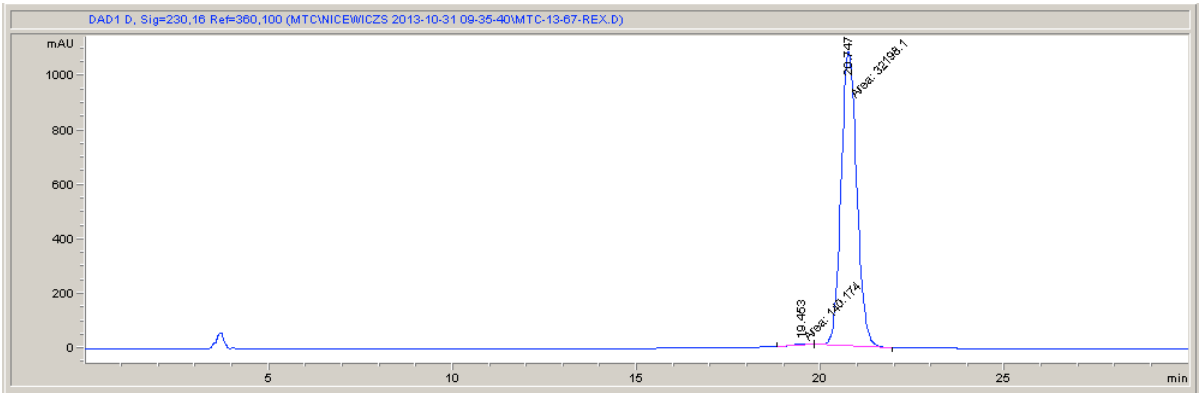


3ai

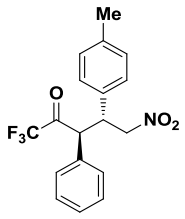
(recrystallized)



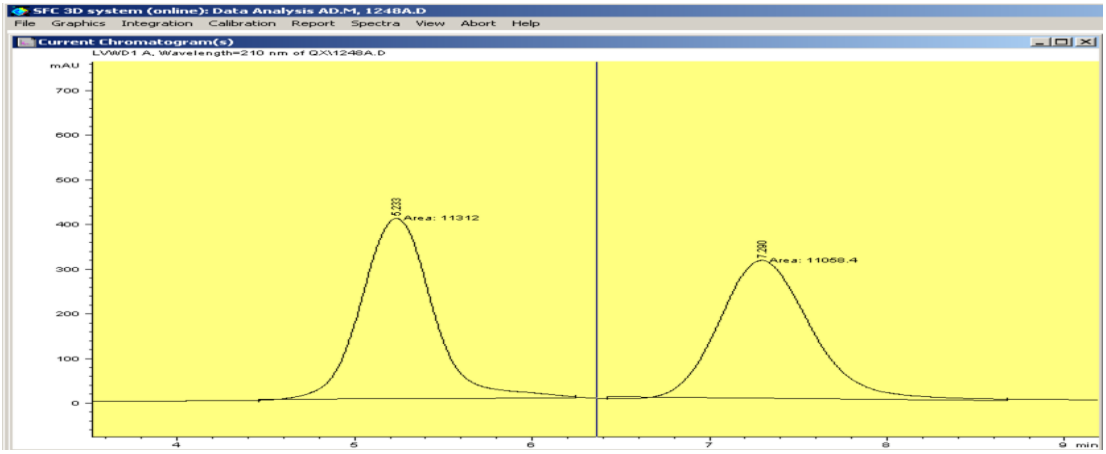
#	Time	Area	Height	Width	Area%	Symmetry
1	19.558	13951.8	521.3	0.446	50.262	0.929
2	20.908	13806.5	479.3	0.4801	49.738	0.875

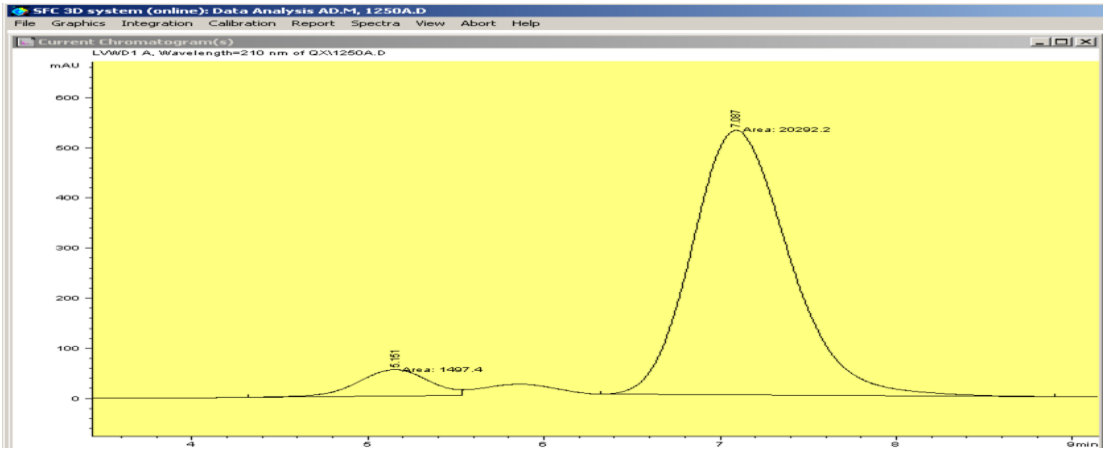


#	Time	Area	Height	Width	Area%	Symmetry
1	19.453	140.2	5.8	0.4017	0.433	1.388
2	20.747	32198.1	1082	0.496	99.567	0.902

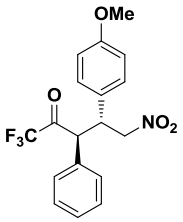


3aj

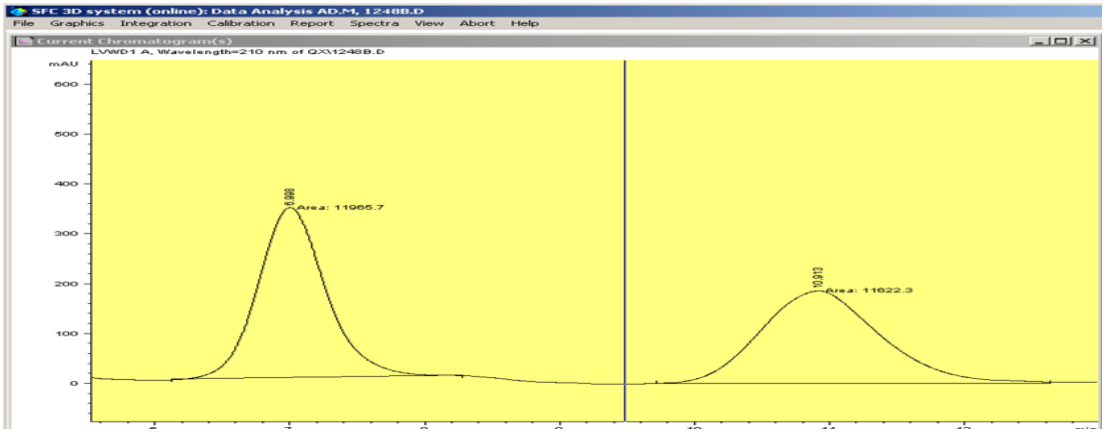


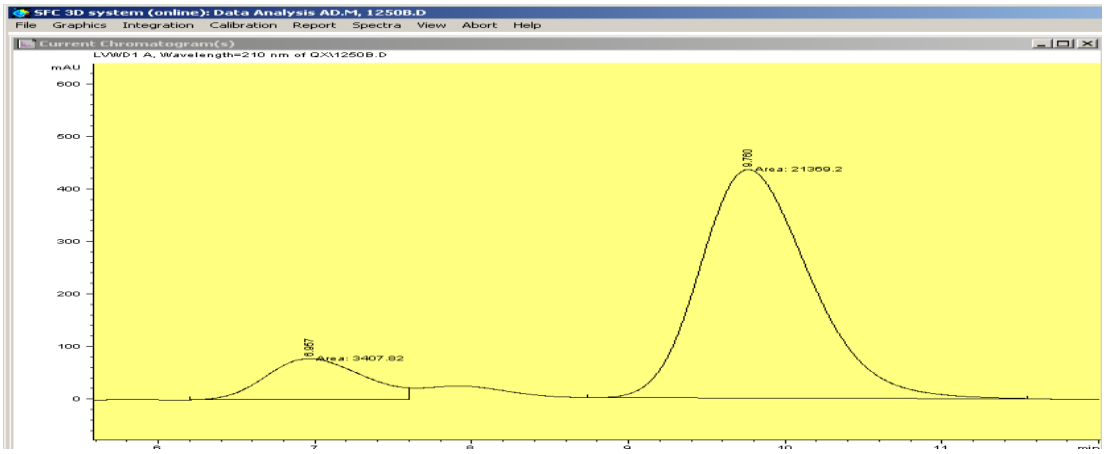


Peak #	RT [min]	Type	Width [min]	Area [mAU*sec]	Height [mAU]	Area %
1	5.151	MF	0.474	1497.39624	52.67319	6.8721
2	7.087	MM	0.640	20292.20898	528.35779	93.1279
Totals :				21789.60547	581.03101	

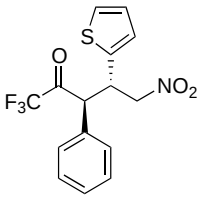


3ak

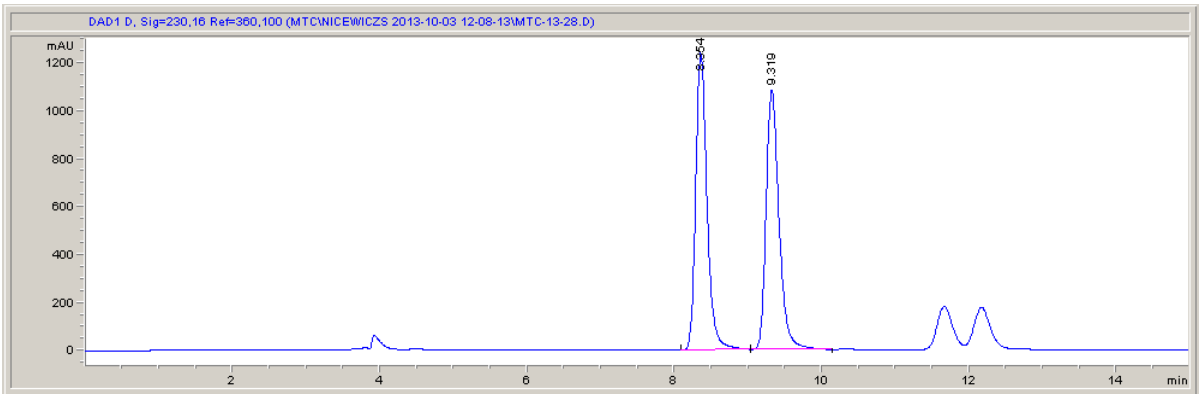




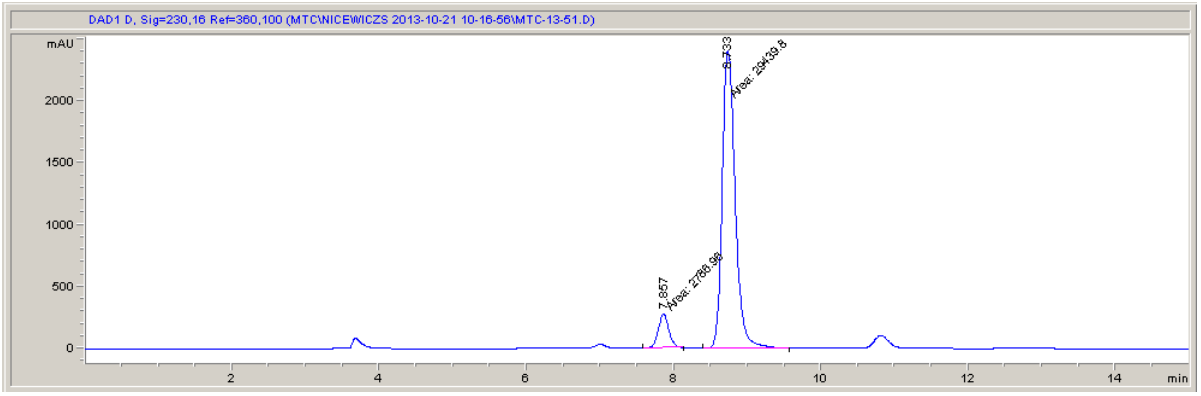
Peak #	RT [min]	Type	Width [min]	Area [mAU*sec]	Height [mAU]	Area %
1	6.957	MF	0.726	3407.81860	78.22980	13.7539
2	9.760	MM	0.818	21369.20313	435.28253	86.2461
Totals :				24777.02148	513.51233	



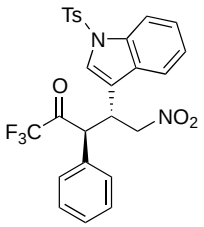
3al



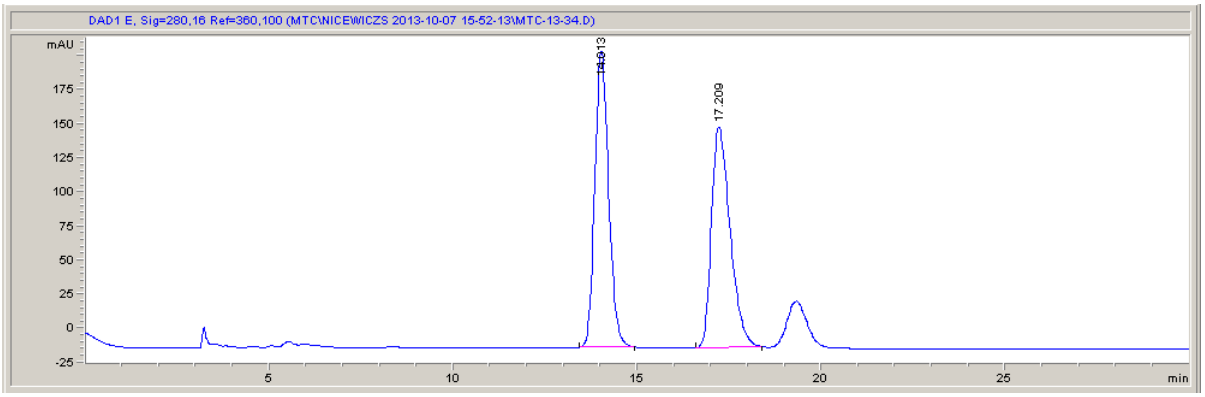
#	Time	Area	Height	Width	Area%	Symmetry
1	8.354	13310.5	1239	0.1633	50.029	0.729
2	9.319	13295.3	1084.8	0.187	49.971	0.71



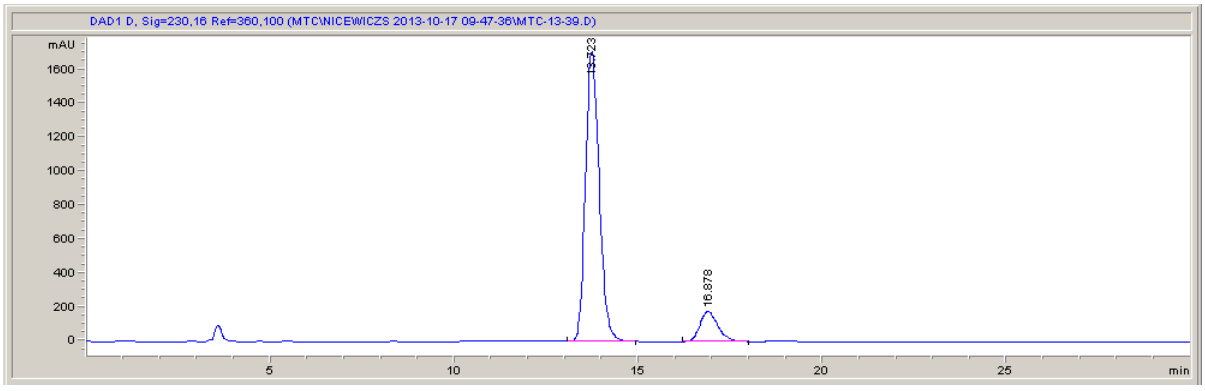
#	Time	Area	Height	Width	Area%	Symmetry
1	7.857	2787	276.5	0.168	8.648	0.936
2	8.733	29439.8	2401.4	0.2043	91.352	0.746



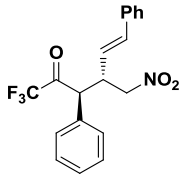
3am



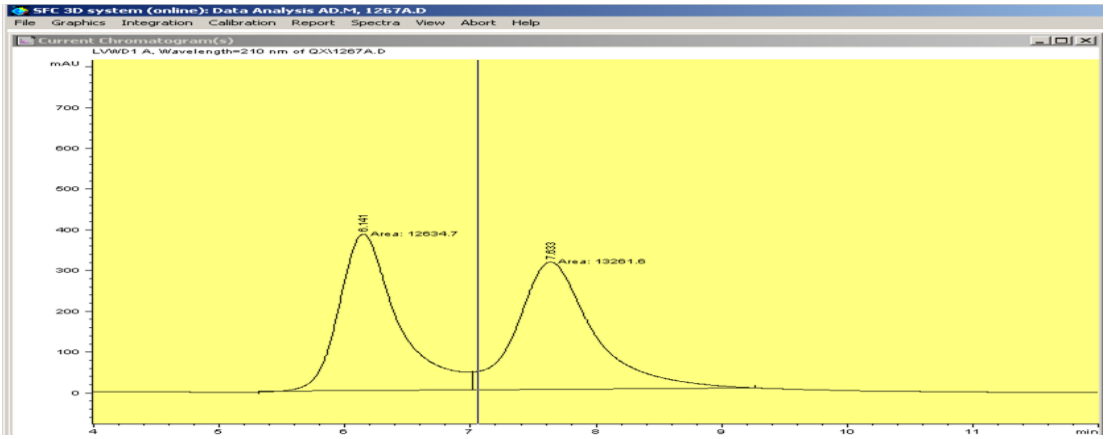
#	Time	Area	Height	Width	Area%	Symmetry
1	14.013	5716.6	216.9	0.4075	50.070	0.824
2	17.209	5700.6	162	0.5404	49.930	0.62

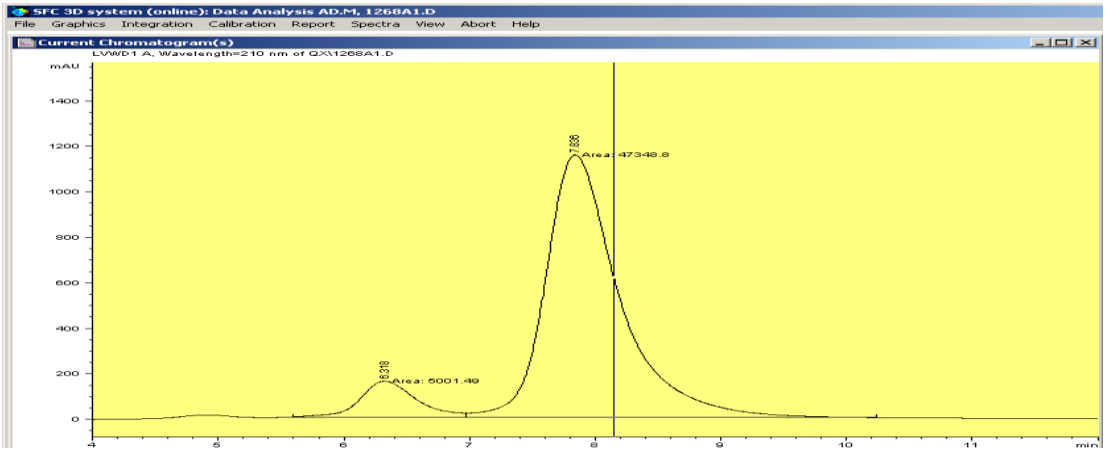


#	Time	Area	Height	Width	Area%	Symmetry
1	13.723	44304.6	1704.7	0.4032	88.056	0.794
2	16.878	6009.4	176.6	0.5229	11.944	0.742

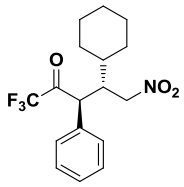


3an

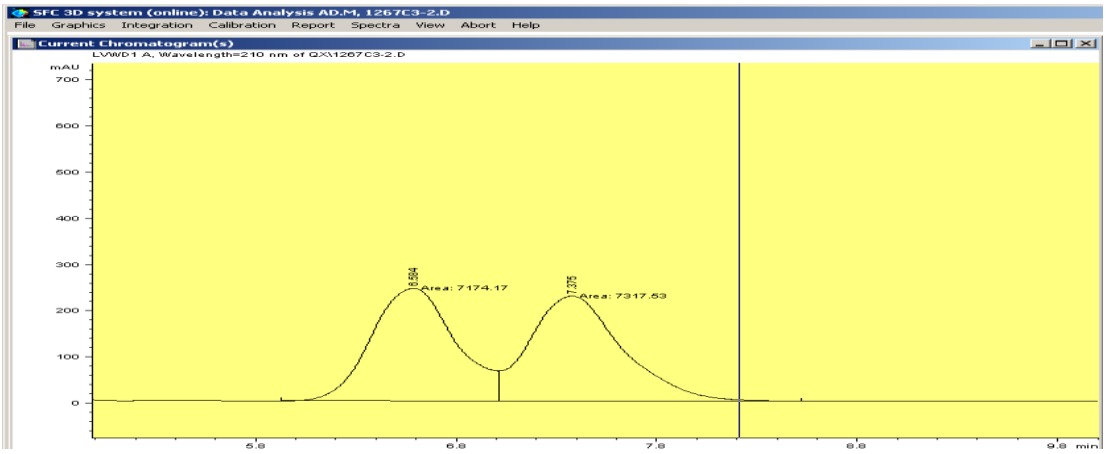


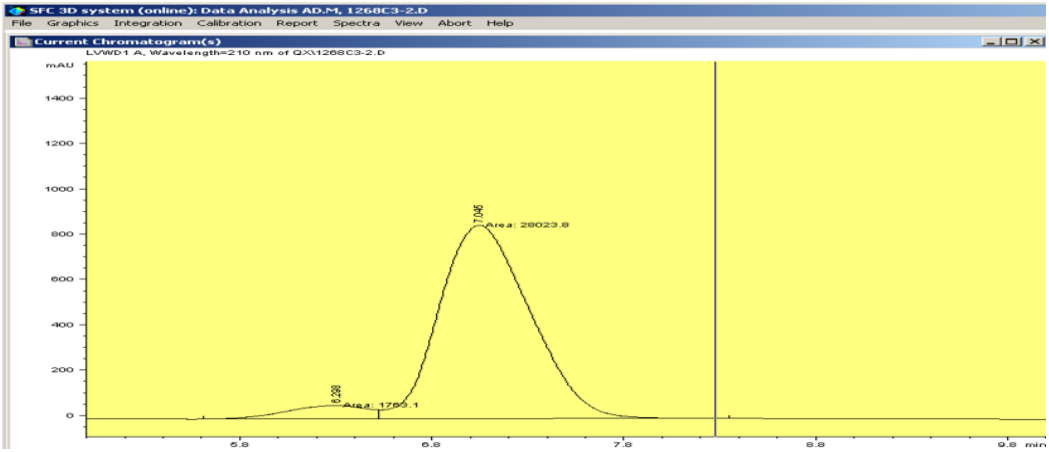


Peak #	RT [min]	Type	Width [min]	Area [mAU*sec]	Height [mAU]	Area %
1	6.318	MF	0.522	5001.49365	159.79164	9.5539
2	7.836	FM	0.683	47348.82031	1154.99268	90.4461
Totals :				52350.31250	1314.78430	

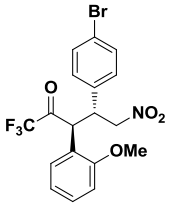


3ao

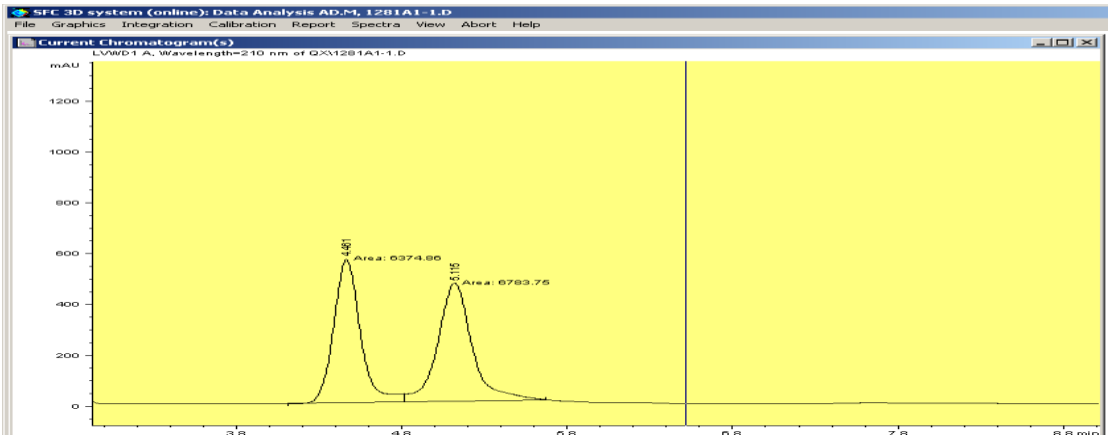


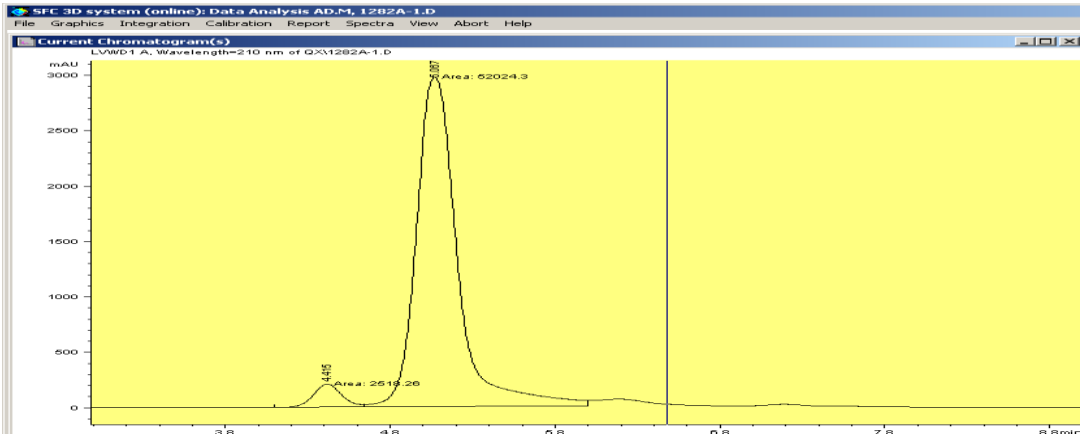


Peak #	RT [min]	Type	Width [min]	Area [mAU*sec]	Height [mAU]	Area %
1	6.298	MF	0.496	1763.09607	59.25628	5.9190
2	7.045	FM	0.547	28023.78125	854.41028	94.0810
Totals :				29786.87695	913.66656	

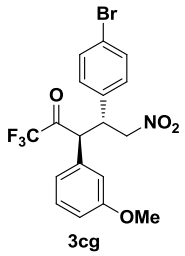


3bg

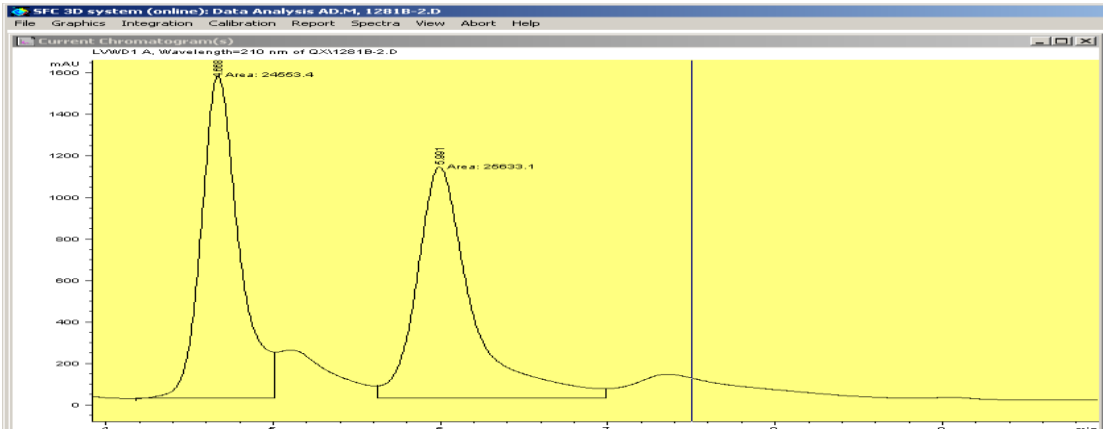


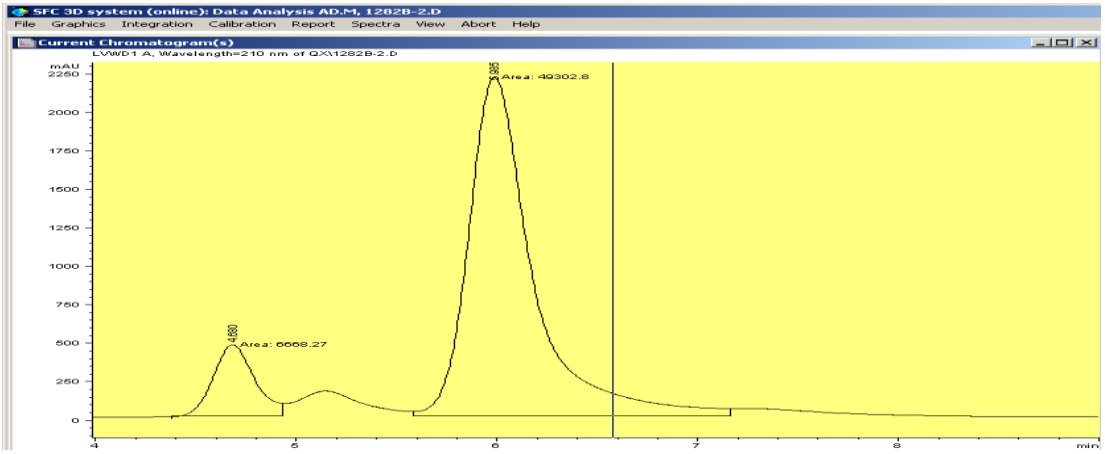


Peak #	RT [min]	Type	Width [min]	Area [mAU*sec]	Height [mAU]	Area %
1	4.415	MF	0.199	2518.26465	211.09296	4.6171
2	5.067	MF	0.291	52024.33203	2980.33887	95.3829
Totals :				54542.59766	3191.43188	

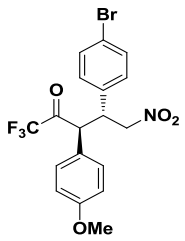


3cg

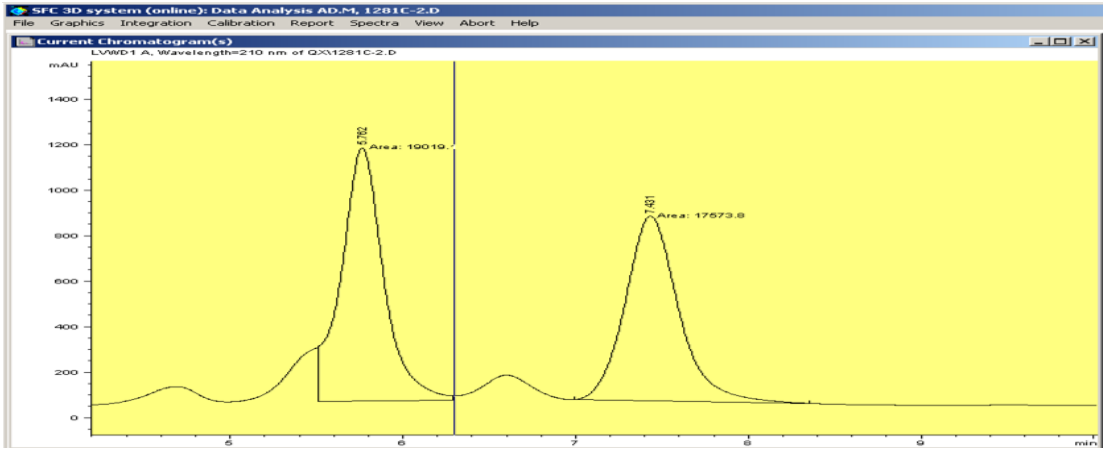


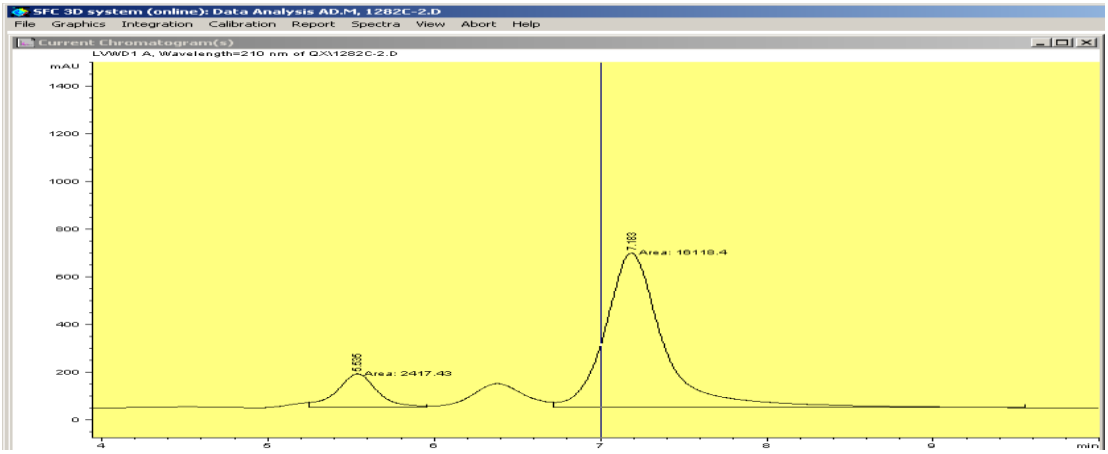


Peak #	RT [min]	Type	Width [min]	Area [mAU*sec]	Height [mAU]	Area %
1	4.680	MF	0.239	6668.26953	465.58774	11.9138
2	5.985	MF	0.373	49302.81250	2202.64038	88.0862
Totals :				55971.08203	2668.22803	

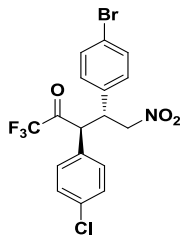


3dg

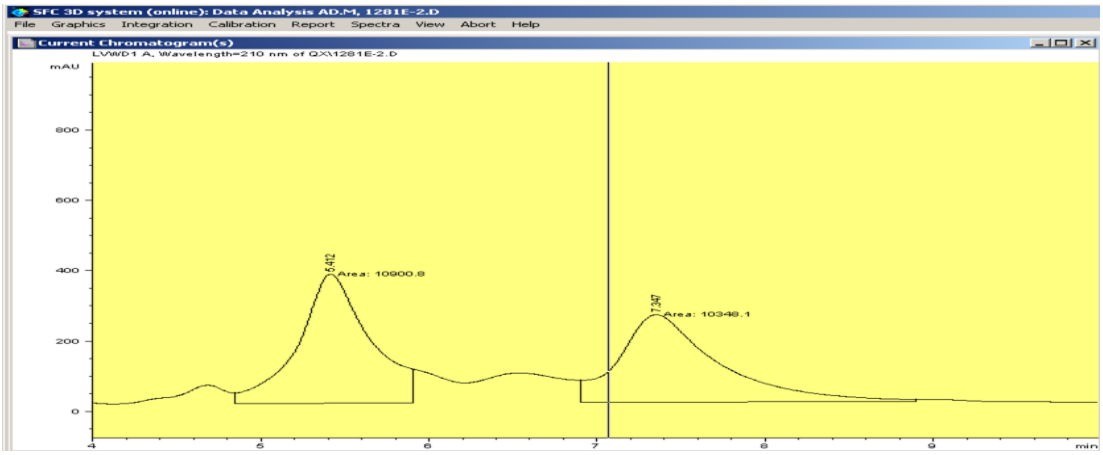


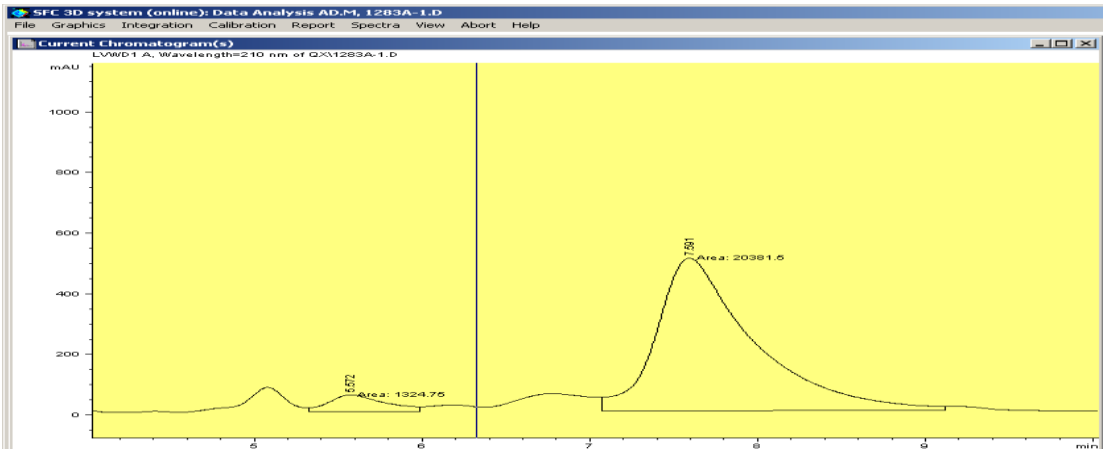


Peak #	RT [min]	Type	Width [min]	Area [mAU*sec]	Height [mAU]	Area %
1	5.535	FM	0.283	2417.42944	142.58124	13.0419
2	7.183	FM	0.413	16118.42773	650.80109	86.9581
Totals :				18535.85742	793.38232	

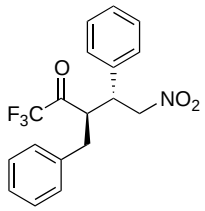


3eg

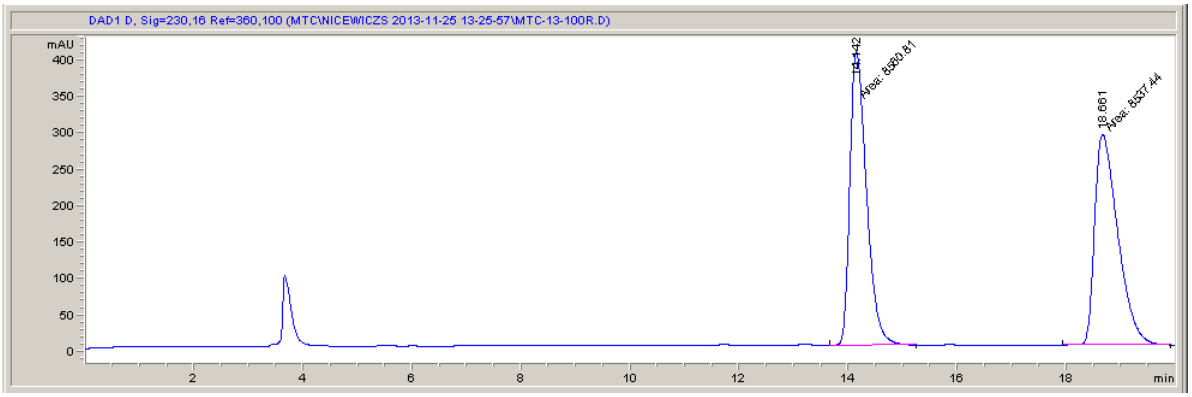




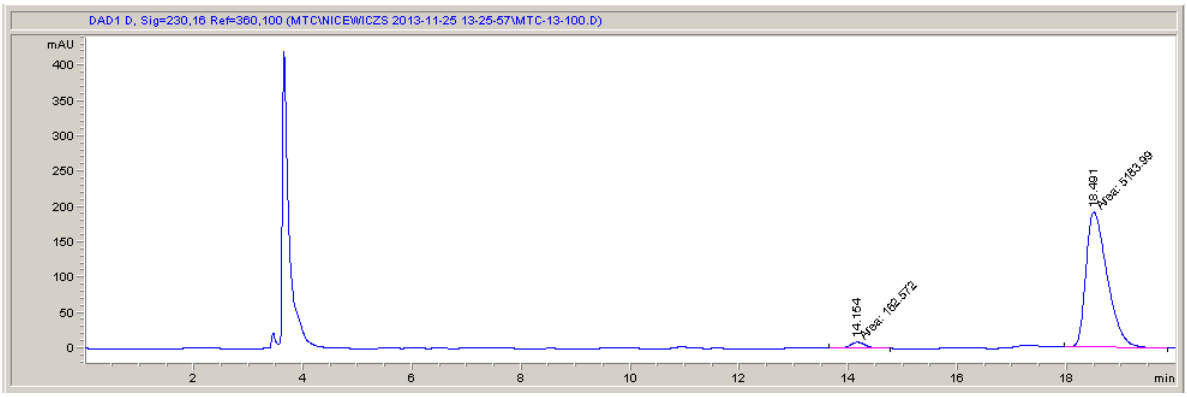
Peak #	RT [min]	Type	Width [min]	Area [mAU*sec]	Height [mAU]	Area %
1	5.572	MF	0.390	1324.74707	56.59434	6.1031
2	7.591	MF	0.673	20381.54297	504.41257	93.8969
Totals :				21706.28906	561.00690	



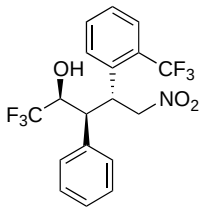
3fa



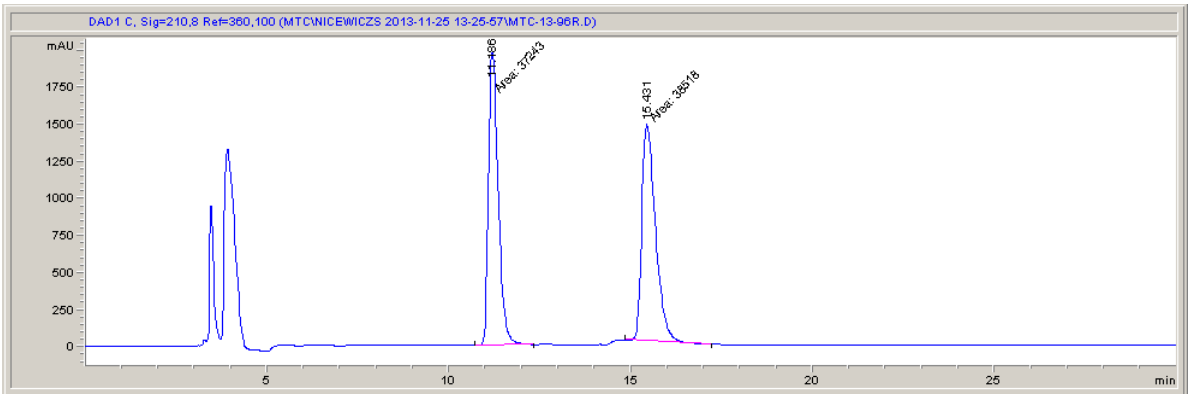
#	Time	Area	Height	Width	Area%	Symmetry
1	14.142	8560.8	403	0.3541	50.068	0.616
2	18.661	8537.4	289.1	0.4922	49.932	0.507



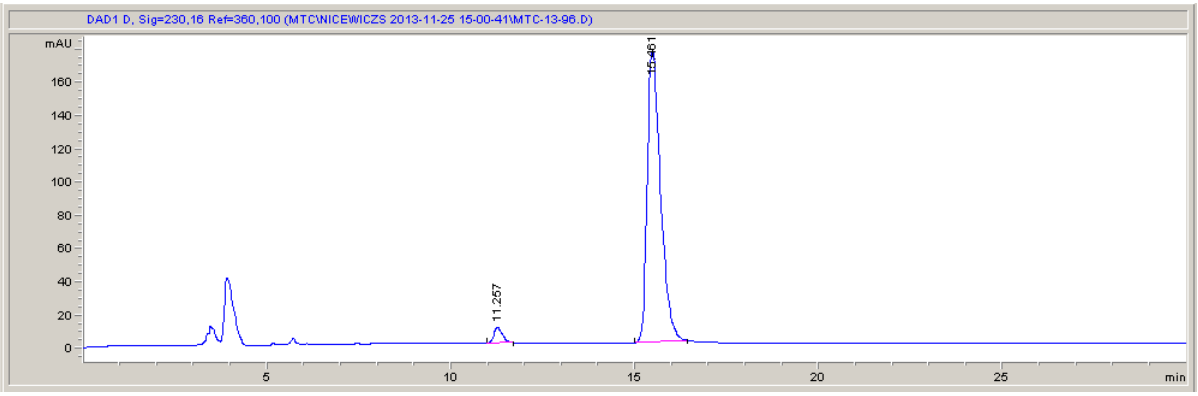
#	Time	Area	Height	Width	Area%	Symmetry
1	14.154	162.6	8.7	0.3132	3.041	0.843
2	18.491	5184	191.1	0.4522	96.959	0.576



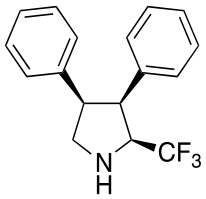
S2

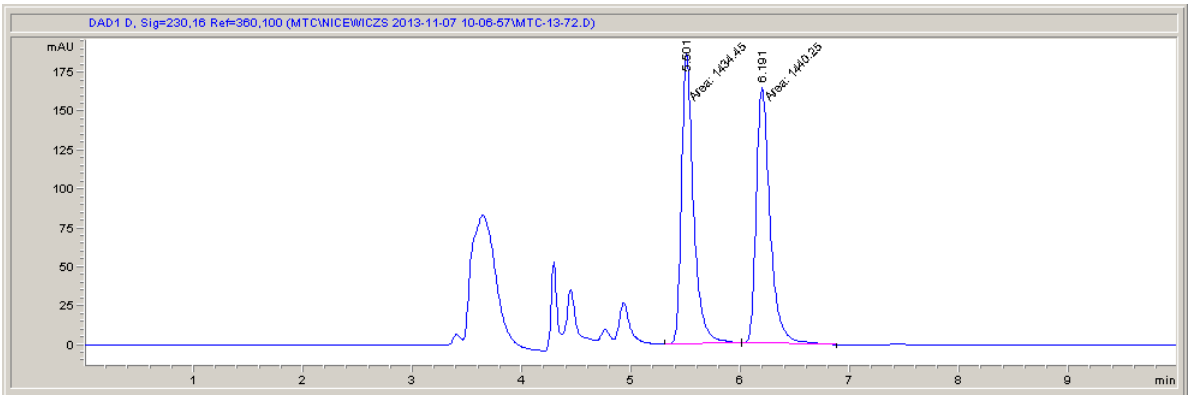


#	Time	Area	Height	Width	Area%	Symmetry
1	11.186	37243	1970	0.3151	49.159	0.637
2	15.431	38518	1463.3	0.4387	50.841	0.561

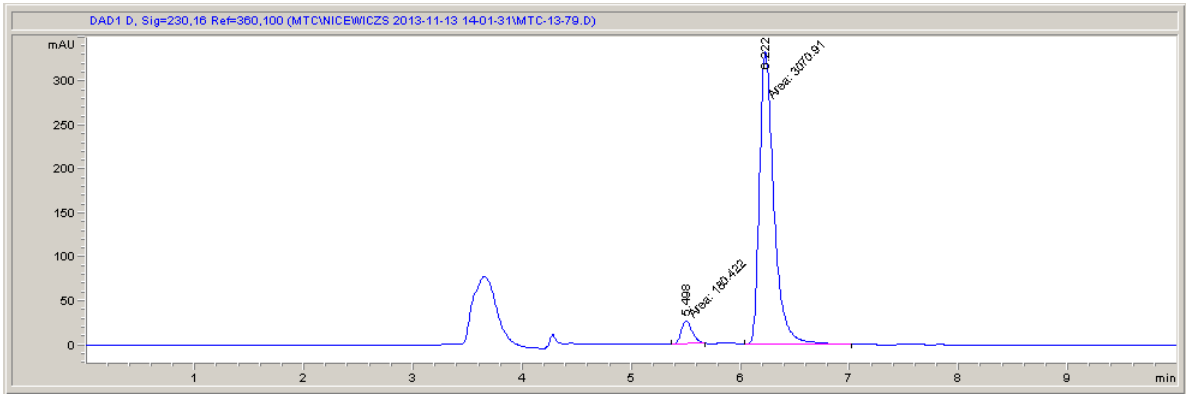


#	Time	Area	Height	Width	Area%	Symmetry
1	11.257	155.8	9.4	0.2539	3.394	0.751
2	15.461	4434.2	175.2	0.3853	96.606	0.576

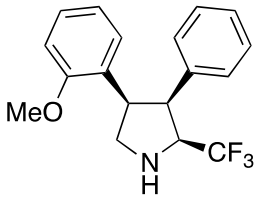


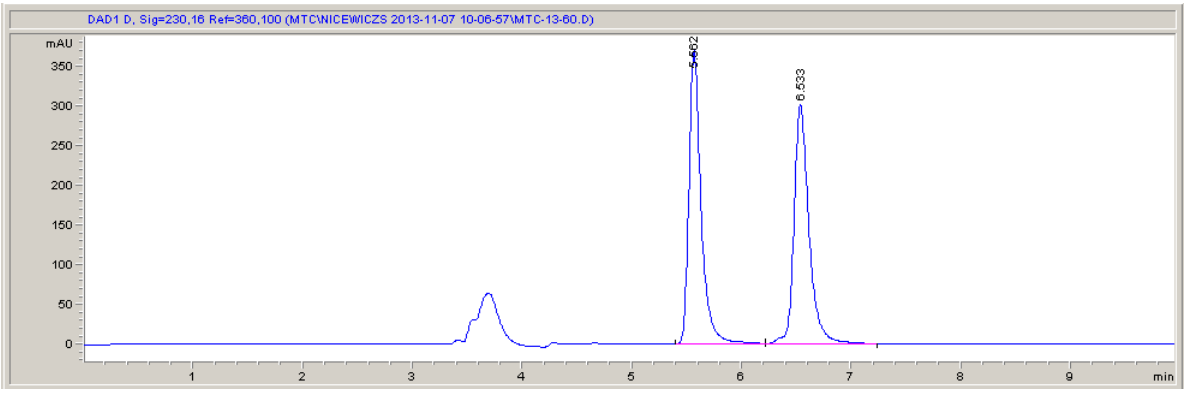


#	Time	Area	Height	Width	Area%	Symmetry
1	5.501	1434.5	186.6	0.1281	49.899	0.672
2	6.191	1440.2	164.9	0.1456	50.101	0.667

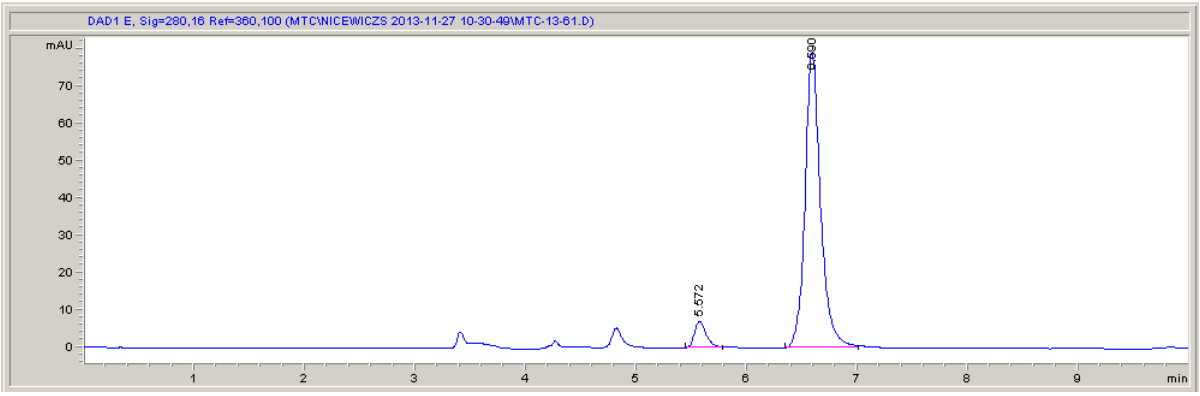


#	Time	Area	Height	Width	Area%	Symmetry
1	5.498	180.4	26.5	0.1134	5.549	0.783
2	6.222	3070.9	333	0.1537	94.451	0.633

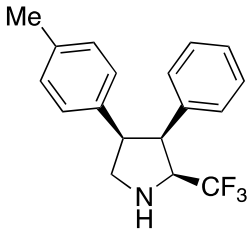


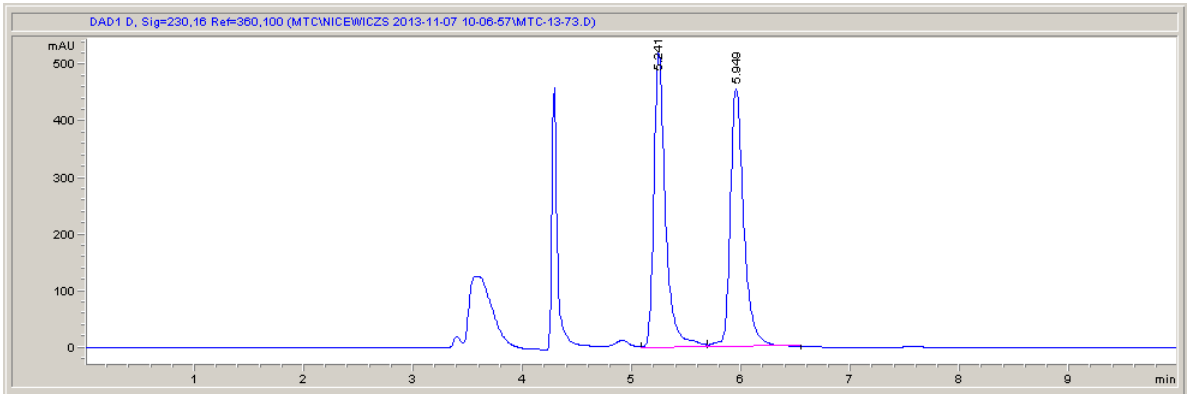


#	Time	Area	Height	Width	Area%	Symmetry
1	5.562	2868.5	370.1	0.1144	49.972	0.636
2	6.533	2871.7	301.2	0.1412	50.028	0.7

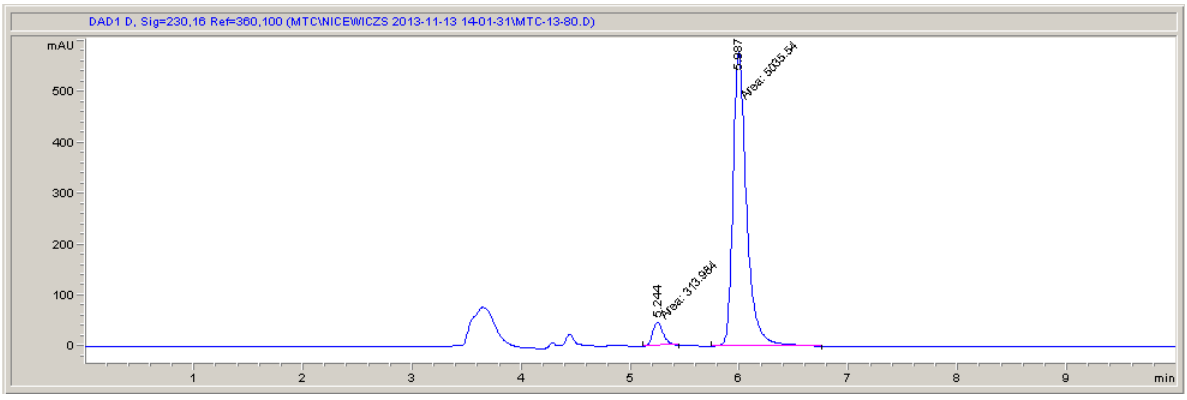


#	Time	Area	Height	Width	Area%	Symmetry
1	5.572	51.8	7.1	0.1107	6.288	0.708
2	6.59	772.7	79.1	0.1459	93.712	0.777

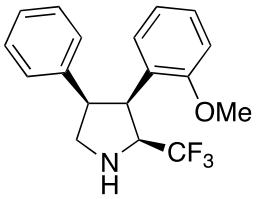


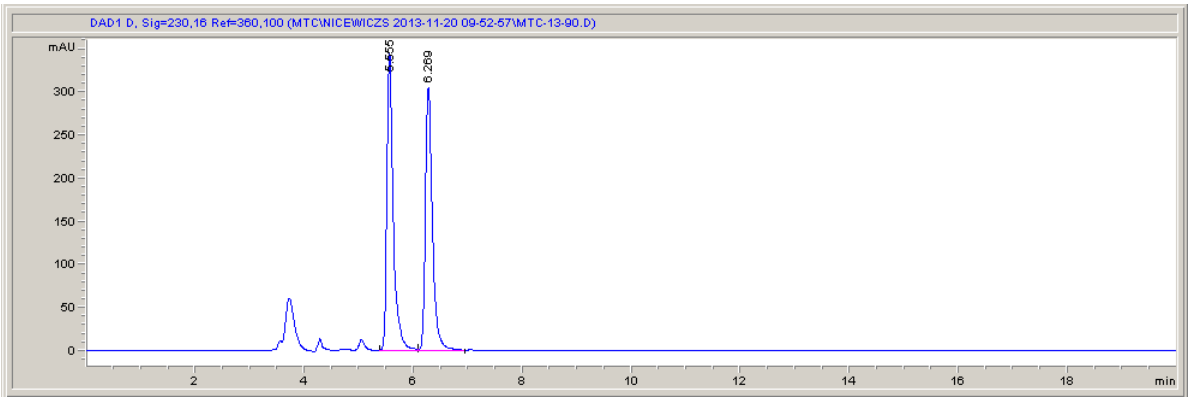


#	Time	Area	Height	Width	Area%	Symmetry
1	5.241	3929.8	521.5	0.1119	50.008	0.616
2	5.949	3928.5	457.3	0.1282	49.992	0.687

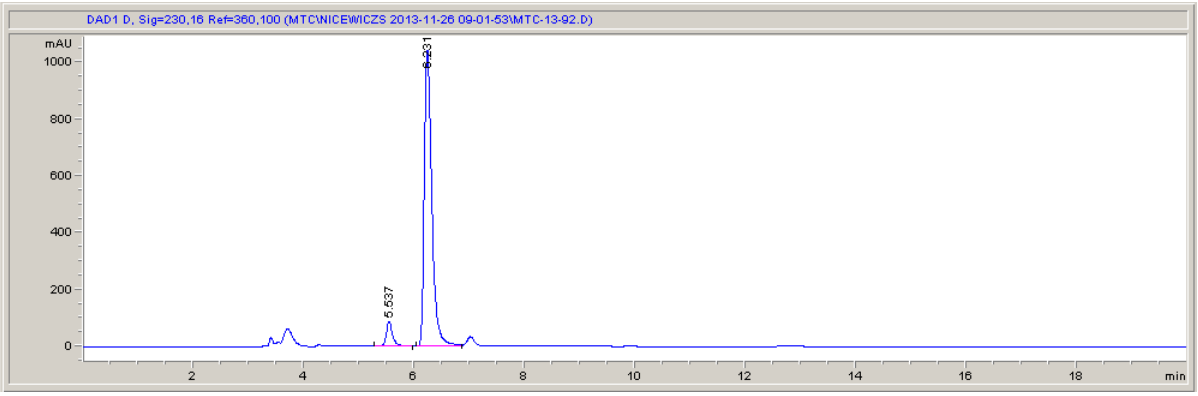


#	Time	Area	Height	Width	Area%	Symmetry
1	5.244	314	47.1	0.1111	5.869	0.765
2	5.987	5035.5	577	0.1454	94.131	0.669

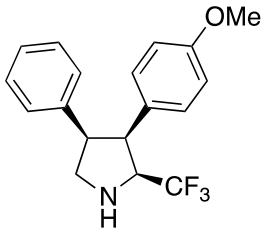


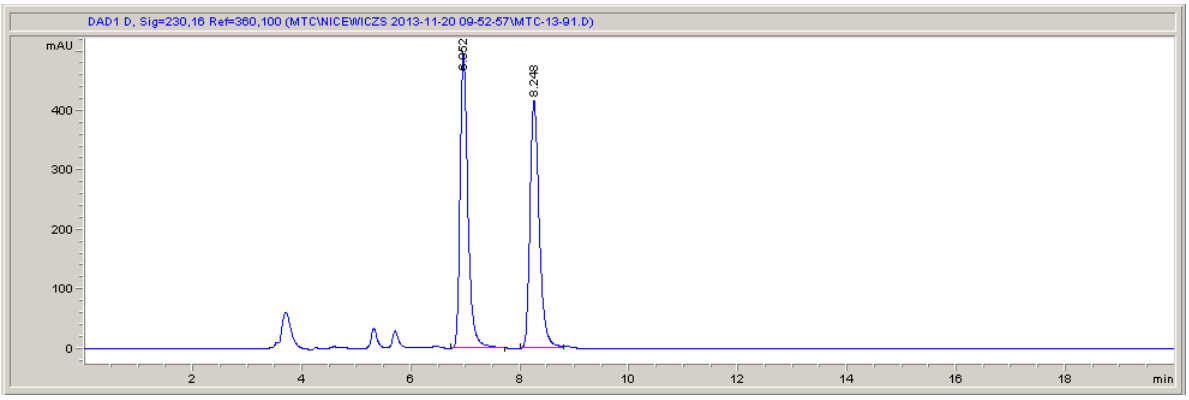


#	Time	Area	Height	Width	Area%	Symmetry
1	5.555	2837.2	344.1	0.1222	50.880	0.589
2	6.269	2739.1	305.4	0.1346	49.120	0.645

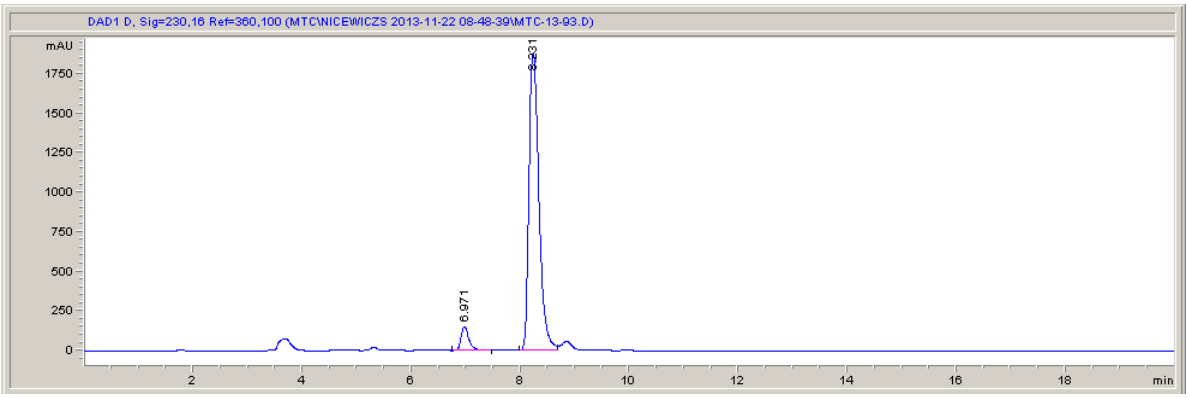


#	Time	Area	Height	Width	Area%	Symmetry
1	5.537	689.1	85.8	0.1197	6.495	0.71
2	6.231	9919.6	1040.9	0.1452	93.505	0.608





#	Time	Area	Height	Width	Area%	Symmetry
1	6.952	4986.7	497	0.1511	50.496	0.673
2	8.248	4888.7	415.4	0.1793	49.504	0.704



#	Time	Area	Height	Width	Area%	Symmetry
1	6.971	1521.1	151	0.1515	5.982	0.666
2	8.231	23905.7	1879.2	0.1964	94.018	0.65