Electronic supplementary information

First one-pot stereoselective synthesis of *cis*-2,3-dihydro-4-perfluoroalkyl -1*H*-1,5-benzodiazepines *via* a catalyst-free three-component reaction

Jiechao Xu,[a] Jiamei Wei,[a] Linglin Bian,[a] Jiaping Zhang,[a] Jie Chen,[a] Hongmei Deng,[a] Xiaoyu Wu*[a] Hui Zhang*[a] and Weiguo Cao*[a,b,c]

[a] *Department of Chemistry, Shanghai University, 99 Shangda Rd, Shanghai 200444, People's Republic of China. E-mail: wgcao@staff.shu.edu.cn, yehao7171@shu.edu.cn, wuxy@shu.edu.cn; Fax: (+86)-21-66134856*

[b] *State Key Laboratory of Organometallic Chemistry, Shanghai Institute of Organic Chemistry, Chinese Academy of Sciences, Shanghai 200032, People's Republic of China*

[c] *Key Laboratory of Organofluorine Chemistry, Shanghai Institute of Organic Chemistry, Chinese Academy of Sciences, Shanghai 200032, People's Republic of China*

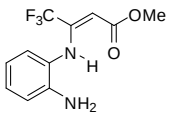
Experimental Section

General information

All reagents and solvents were purchased from commercial sources and used without further purification, except methyl 2-perfluoroalkynoates **2** were prepared according to reported literature. Melting points were uncorrected. ^{1}H, ^{19}F and ^{13}C NMR spectra were recorded on 500MHz spectrometer. All chemical shifts are reported in parts per million downfield (positive) of the standard: C_6F_6 for ^{19}F, TMS for ^{1}H and ^{13}C NMR spectra. IR spectras were obtained on a FT-IR spectrometer. Elemental analysis was performed on an elemental analysis instrument. MS (ESI) was run on a mass spectrometer and MS (EI) on a mass spectrometer. X-ray analysis was performed on an X-ray spectrometer.

*Synthesis of product **3a**.*

A mixtrure of *o*-phenylenediamine **1** (0.5 mmol), methyl 2-perfluoroalkynoates **2a** (0.5 mmol) in anhydrous ethanol (5 mL) was stirred at room temperature for about 30 min. The completion of the reaction was monitored by TLC. The solvent was removed under vaccum. The residue was purified by silica gel column chromatography eluted with petroleum ether:ethyl acetate (*v*:*v* = 20:1) to give product **3a**.

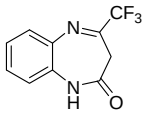


3a

(*Z*)-Methyl 3-(2-aminophenylamino)-4,4,4-trifluorobut-2-enoate **3a**: Yellow solid. Yield: 88%. M.p.: 120.1~120.2℃. Anal. Calcd. for $C_{11}H_{11}F_3N_2O_2$: C, 50.77; H, 4.26; N, 10.77. Found C, 50.98; H, 4.47; N, 10.80. IR (KBr, cm^{-1}): *v* 1669 (C=O), 2952 (CH), 3030 (Ar-H), 3386 (NH_2), 3473 (NH). ^{1}H NMR ($CDCl_3$, ppm): δ 3.76 (s, 3H, OCH_3), 3.86 (s, 2H, NH), 5.39 (s, 1H, CH), 6.69-7.12 (m, 4H, ArH), 9.25 (s, 1H, NH). ^{13}C NMR ($CDCl_3$, ppm): δ 170.2, 149.0 (q, CCF_3, $^{2}J_{C\text{-}F}$ = 31.0 Hz), 144.1, 129.6, 129.5, 129.0, 123.8, 120.2 (q, CF_3, $^{1}J_{C\text{-}F}$ = 276.0 Hz), 118.2, 115.8, 88.5 (q, $C{=}CCF_3$, $^{3}J_{C\text{-}F}$= 6.2 Hz), 51.5. ^{19}F NMR ($CDCl_3$, ppm): δ -65.5 (s, CF_3). ESI-MS (m/z): 260 (M^+).

*Synthesis of product **4a**.*

A mixture of *o*-phenylenediamine **1** (0.5 mmol), methyl 2-perfluoroalkynoates **2a** (0.5 mmol) in anhydrous ethanol (5 mL) was stirred at refluxing temperature for about 24h. The completion of the reaction was monitored by TLC. When the reaction finished, the solvent was removed by vaccum. The residue was purified by silica gel column chromatography eluted with petroleum ether:ethyl acetate (*v*:*v* = 10:1) to afford product **4a**.



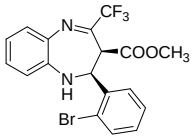
4a

(*E*)-4-(Trifluoromethyl)-1*H*-benzo[*b*][1,4]diazepin-2(*3H*)-one **4a**:

Yellow solid. Yield: 73%. M.p.: 188.9~190.4℃. ^{1}H NMR ($CDCl_3$, ppm): δ 3.36 (s, 2H, CH_2), 7.15-7.51 (m, 4H, ArH), 9.37 (s, 1H, NH).

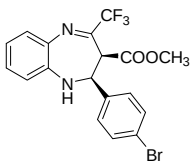
*Synthesis of products **6**.*

General method: A mixture of *o*-phenylenediamine **1** (0.5 mmol), aldehydes **5** (0.5 mmol) and methyl 2-perfluoroalkynoates **2** (0.5 mmol) in anhydrous ethanol (5 mL) was stirred at refluxing temperature for about 20~24h. The completion of the reaction was monitored by TLC. When the reaction finished, the solvent was removed by vaccum. And the residue was purified by silica gel column chromatography eluted with petroleum ether:ethyl acetate (v:v = 10:1) to get product **6**. Futher purification could be carried out by recrystallization from petroleum ether:ethyl acetate (*v*:*v* = 2:1).



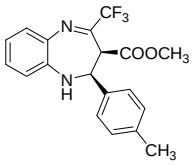
6a

cis-Methyl 2-(2-bromophenyl)-4-(trifluoromethyl)-2,3-dihydro-1*H*-benzo[*b*][1,4]diazepine-3-carboxylate **6a:** Yellow solid. Yield: 57%. M.p.: 150.9~153.5 ℃. Anal. Calcd. for $C_{18}H_{14}BrF_3N_2O_2$: C, 50.61; H, 3.30; N, 6.56. Found: C, 50.68; H, 3.24; N, 6.73. IR (KBr, cm^{-1}): *v* 1724 (C=O); 2953 (C-H); 3046 (Ar-H); 3427 (N-H). ^{1}H NMR ($CDCl_3$, ppm): δ 3.56 (s, 3H, OCH_3), 4.46 (d, *J* = 4.5 Hz, 1H, CH), 4.62 (d, *J* = 6.0 Hz, 1H, NH), 4.83 (dd, J_1 = 6.0 Hz, J_2 = 4.5 Hz, 1H, CH), 6.75-7.67 (m, 8H, ArH). ^{13}C NMR ($CDCl_3$, ppm): δ 166.7, 145.4 (q, CCF_3, $^2J_{C\text{-}F}$ = 34 Hz) , 141.8, 138.1, 136.8, 133.8, 131.1, 130.3, 129.3, 128.0, 127.3, 120.5 (q, CF_3, $^1J_{C\text{-}F}$ = 276 Hz), 119.9, 118.4, 117.6, 56.1, 55.3, 52.6. ^{19}F NMR ($CDCl_3$, ppm): δ -71.39 (s, CF_3). ESI-MS (m/z): 426 (M^+).



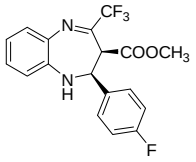
6b

cis-Methyl 2-(4-bromophenyl)-4-(trifluoromethyl)-2,3-dihydro-1*H*-benzo[*b*][1,4]diazepine-3-carboxylate **6b:** Yellow solid. Yield: 73%. M.p.: 135.9~138.3℃. Anal. Calcd. for $C_{18}H_{14}BrF_3N_2O_2$: C, 50.61; H, 3.30; N, 6.56. Found: C, 50.90; H, 3.43; N, 6.81. IR (KBr, cm^{-1}): *v* 1750 (C=O); 2957 (C-H); 3051 (Ar-H); 3329 (N-H). ^{1}H NMR ($CDCl_3$, ppm): δ 3.58 (s, 3H, OCH_3), 4.44 (d, J = 4.5 Hz, 1H, CH), 4.94 (d, J = 6.0 Hz, 1H, NH), 5.44 (dd, J_1 = 6.0 Hz, J_2 = 4.5 Hz, 1H, CH), 6.76-7.44 (m, 8H, ArH). ^{13}C NMR ($CDCl_3$, ppm): δ 167.6, 147.0 (q, CF_3, $^2J_{C\text{-}F}$ = 34 Hz), 140.8, 139.8, 134.7, 131.9, 130.7, 130.3, 127.4, 121.9, 120.0 (q, CF_3, $^1J_{C\text{-}F}$ = 276 Hz), 119.4, 118.3, 77.1, 60.6, 54.2, 53.1. ^{19}F NMR ($CDCl_3$, ppm): δ -71.69 (s, CF_3). ESI-MS (m/z): 426 (M^+).



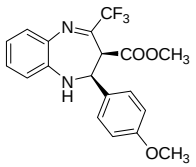
6c

cis-Methyl 2-*p*-tolyl-4-(trifluoromethyl)-2,3-dihydro-1*H*-benzo[*b*][1,4]diazepine-3-carboxylate **6c:** Yellow solid. Yield: 90%. M.p.: 148.6~148.9℃. Anal. Calcd. for $C_{19}H_{17}F_3N_2O_2$: C, 62.98; H, 4.73; N, 7.73. Found: C, 62.81; H, 4.56; N, 7.82. IR (KBr, cm^{-1}): *v* 1745 (C=O); 2957 (C-H); 3080 (Ar-H); 3329 (N-H). ^{1}H NMR ($CDCl_3$, ppm): δ 2.27 (s, 3H, CH_3), 3.58 (s, 3H, OCH_3), 4.47 (d, J = 4.5 Hz, 1H, CH), 4.94 (d, J = 6.0 Hz, 1H, NH), 5.39 (dd, J_1 = 6.0 Hz, J_2 = 4.5 Hz, 1H, CH), 6.73-7.44 (m, 8H, ArH). ^{13}C NMR ($CDCl_3$, ppm): δ 168.2, 147.0 (q, CF_3, $^2J_{C\text{-}F}$ = 34 Hz), 141.6, 138.0, 137.8, 135.0, 130.8, 130.4, 129.6, 125.7, 122.5, 120.7, 120.1 (q, CF_3, $^1J_{C\text{-}F}$ = 267.5 Hz), 119.0, 118.4, 60.6, 54.9, 53.1, 21.2. ^{19}F NMR ($CDCl_3$, ppm): δ -71.54 (s, CF_3). ESI-MS (m/z): 362 (M^+).



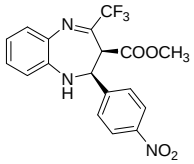
6d

cis-Methyl 2-(4-fluorophenyl)-4-(trifluoromethyl)-2,3-dihydro-1*H*-benzo[*b*][1,4]diazepine-3-carboxylate **6d:** Yellow solid. Yield: 54%. M.p.: 95.1~96.1℃. Anal. Calcd. for $C_{18}H_{14}F_4N_2O_2$: C, 59.02; H, 3.85; N, 7.65. Found: C, 59.09; H, 3.56; N, 7.82. IR (KBr, cm^{-1}): *v* = 1745 (C=O); 2959 (C-H); 3080 (Ar-H); 3335 (N-H). ^{1}H NMR ($CDCl_3$, ppm): δ= 3.55 (s, 3H, OCH_3), 4.44 (d, J =4.5 Hz, 1H, CH), 5.09 (d, J =6.5 Hz, 1H, NH), 5.44 (dd, J_1 = 6.5 Hz, J_2 = 4.5 Hz, 1H, CH), 6.73-7.43 (m, 8H, ArH). ^{13}C-NMR ($CDCl_3$, ppm): δ 167.7, 163.2, 161.2, 146.8 (q, CF_3, $^2J_{C\text{-}F}$ =34 Hz), 141.1, 136.8 (d, Ar-F, $^2J_{C\text{-}F}$ =2.5 Hz), 134.7, 130.7, 130.2, 127.4 (d, Ar-F, $^1J_{C\text{-}F}$ =7.5 Hz), 119.9 (q, CF_3, $^1J_{C\text{-}F}$ =276 Hz), 119.2, 118.3, 115.7, 115.6, 60.4, 54.5, 53.0. ^{19}F NMR ($CDCl_3$, ppm): δ -71.67 (s, CF_3), -114.22 (m, ArF). ESI-MS (m/z): 366 (M^+).



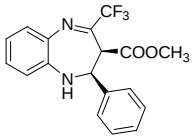
6e

cis-Methyl 2-(4-methoxyphenyl)-4-(trifluoromethyl)-2,3-dihydro-1*H*-benzo[*b*][1,4]diazepine-3-carboxylate **6e:** Yellow solid. Yield: 80%. M.p.: 110.7~111.2℃. Anal. Calcd. for $C_{19}H_{17}F_3N_2O_3$: C, 60.32; H, 4.53; N, 7.40. Found: C, 57.91; H, 4.43; N, 7.17. IR (KBr, cm^{-1}): *v* 1736 (C=O); 2956 (C-H); 3004 (Ar-H); 3398 (N-H). ^{1}H NMR ($CDCl_3$, ppm): δ 3.58 (s, 3H, OCH_3), 3.73 (s, 3H, OCH_3), 4.44 (d, J = 4.5 Hz, 1H, CH), 4.95 (d, J = 4.0 Hz, 1H, NH), 5.38 (dd, J_1 = 4.0 Hz, J_2 = 4.5 Hz, 1H, CH), 6.74-7.44 (m, 8H, ArH). ^{13}C NMR ($CDCl_3$, ppm): δ 168.0, 159.1, 146.8 (q, CF_3, $^2J_{C\text{-}F}$ = 34 Hz), 141.6, 134.9, 133.0, 132.1, 130.7, 130.1, 126.9, 120.1 (q, CF_3, $^1J_{C\text{-}F}$ = 276 Hz), 118.9, 118.3, 114.1, 114.5, 59.9, 55.2, 54.9, 53.0. ^{19}F NMR ($CDCl_3$, ppm): δ -71.39 (s, CF_3). ESI-MS (m/z): 378 (M^+).



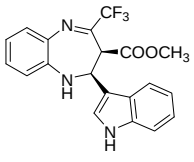
6f

cis-Methyl 2-(4-nitrophenyl)-4-(trifluoromethyl)-2,3-dihydro-1*H*-benzo[*b*][1,4]diazepine-3-carboxylate **6f:** Yellow solid. Yield: 50%. M.p.: 176.5~180.0℃. Anal. Calcd. for $C_{18}H_{14}F_3N_3O_4$: C, 54.97; H, 3.59; N, 10.68. Found: C, 54.99; H: 3.35; N: 10.89. IR (KBr, cm^{-1}): *v* 1743 (C=O); 2971 (C-H); 3079 (Ar-H); 3375 (N-H). ^{1}H NMR ($CDCl_3$, ppm): δ 3.61 (s, 3H, OCH_3), 4.36 (d, J = 4.0 Hz, 1H, CH), 4.65 (d, J = 6.0 Hz, 1H, NH), 4.70 (dd, J_1 = 6.0 Hz, J_2 = 4.0 Hz, 1H, CH), 6.78-8.31 (m, 8H, ArH). ^{13}C NMR ($CDCl_3$, ppm): δ 166.4, 148.2, 146.4, 145.5 (q, CF_3, $^2J_{C\text{-}F}$ = 34 Hz), 140.9, 136.4, 131.3, 129.6, 128.2, 124.4, 120.4, 120.3 (q, CF_3, $^1J_{C\text{-}F}$ = 276 Hz), 120.1, 118.6, 113.4, 58.1, 56.6, 52.9. ^{19}F NMR ($CDCl_3$, ppm): δ -71.57 (s, CF_3). ESI-MS (m/z): 393 (M^+).



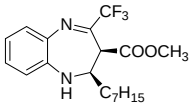
6g

cis-Methyl 2-phenyl-4-(trifluoromethyl)-2,3-dihydro-1*H*-benzo[*b*][1,4]diazepine-3-carboxylate **6g:** Yellow solid. Yield: 72%. M.p.: 113.8~114.2℃. Anal. Calcd. for $C_{18}H_{15}F_3N_2O_2$: C, 62.07; H, 4.34; N, 8.04. Found: C, 61.80; H, 4.12; N, 8.02. IR (KBr, cm^{-1}): *v* 1742 (C=O); 2957 (C-H); 3075 (Ar-H); 3309 (N-H). ^{1}H NMR ($CDCl_3$, ppm): δ 3.59 (s, 3H, OCH_3), 4.47 (d, J = 4.0 Hz, 1H, CH), 4.92 (d, J = 6.0 Hz, 1H, NH), 5.47 (dd, J_1 = 6.0 Hz, J_2 = 4.0 Hz, 1H, CH), 6.77-7.45 (m, 9H, ArH). ^{13}C NMR ($CDCl_3$, ppm): δ 167.9, 146.8 (q, CF_3, $^2J_{C\text{-}F}$ = 34 Hz), 141.4, 140.9, 138.4, 134.8, 130.7, 130.2, 128.8, 127.9, 125.6, 119.9 (q, CF_3, $^1J_{C\text{-}F}$ = 276 Hz), 119.7, 119.0, 118.3, 60.8, 54.7, 53.0. ^{19}F NMR ($CDCl_3$, ppm): δ -71.74 (s, CF_3). ESI-MS (m/z): 348 (M^+).



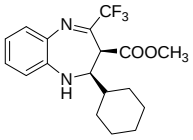
6h

cis-Methyl 2-(1*H*-indol-3-yl)-4-(trifluoromethyl)-2,3-dihydro-1*H*-benzo[*b*][1,4]diazepine-3-carboxylate **6h:** Yellow solid. Yield: 56%. M.p.: 149.4~152.0℃. Anal. Calcd. for $C_{20}H_{16}F_3N_3O_2$: C, 62.01; H, 4.16; N, 10.85. Found: C, 61.74; H, 3.94; N, 10.87. IR (KBr, cm^{-1}): *v* 1747 (C=O); 2956 (C-H); 3063 (Ar-H); 3388 (N-H). ^{1}H NMR ($CDCl_3$, ppm): δ 3.67 (s, 3H, OCH_3), 4.60 (d, J = 4.0 Hz, 1H, CH), 4.99 (d, J = 6.0 Hz, 1H, NH), 5.66 (dd, J_1 = 6.0 Hz, J_2 = 4.0 Hz, 1H, CH), 6.70-7.97 (m, 10H, ArH). ^{13}C NMR ($CDCl_3$, ppm): δ 168.6, , 147.2 (q, CF_3, $^2J_{C\text{-}F}$ = 34 Hz), 141.9, 136.6, 136.1, 135.4, 132.2, 131.1, 130.8, 129.1, 124.9, 124.7, 120.4 (q, CF_3, $^1J_{C\text{-}F}$ = 273 Hz), 120.2, 118.6, 115.9, 111.7, 54.9, 53.2, 52.9. ^{19}F NMR ($CDCl_3$, ppm): δ -71.34 (s, CF_3). ESI-MS (m/z): 387 (M^+).



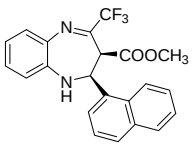
6i

cis-Methyl 2-*n*-heptyl-4-(trifluoromethyl)-2,3-dihydro-1*H*-benzo[*b*][1,4]diazepine-3-carboxylate **6i:** Yellow oil. Yield: 56%. Anal. Calcd. for $C_{19}H_{25}F_3N_2O_2$: C, 61.61; H, 6.80; N, 7.56. Found: C, 61.43; H, 6.67; N, 7.53. IR (KBr, cm^{-1}): *v* 1737 (C=O); 2928 (C-H); 3065 (Ar-H); 3398 (N-H). ^{1}H NMR ($CDCl_3$, ppm): δ 0.84 (t, J = 7.0, 3H, CH_3), 1.19~1.28 (m, 12H, C_6H_{12}), 3.62 (s, 3H, OCH_3), 3.99 (m, 1H, CH), 4.17 (d, J = 4.0 Hz, 1H, NH), 4.89 (d, J = 5.5 Hz, 1H, CH), 6.64-7.56 (m, 4H, ArH). ^{13}C NMR ($CDCl_3$, ppm): δ 168.9, 144.5 (q, CF_3, $^2J_{C\text{-}F}$ = 34 Hz), 141.0, 136.3, 131.0, 129.5, 120.5 (q, CF_3, $^1J_{C\text{-}F}$ = 276 Hz), 118.7, 118.4, 117.4, 54.9, 53.0, 52.8, 32.7, 31.6, 29.0, 26.0, 22.6, 14.0. ^{19}F NMR ($CDCl_3$, ppm): δ -71.26 (s, CF_3). ESI-MS (m/z): 370 (M^+).



6j

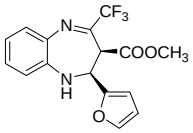
cis-Methyl 2-cyclohexyl-4-(trifluoromethyl)-2,3-dihydro-1*H*-benzo[*b*][1,4]diazepine-3-carboxylate **6j**: Yellow solid. Yield: 68%. M.p.: 126.6~127.2℃. Anal. Calcd. for $C_{18}H_{21}F_3N_2O_2$: C, 61.01; H, 5.97; N, 7.91. Found: C, 61.30; H, 6.01; N, 8.01. IR (KBr, cm^{-1}): *v* 1735 (C=O); 2960 (C-H); 3060 (Ar-H); 3403 (N-H). ^{1}H NMR ($CDCl_3$, ppm): δ 0.88-1.76 (m, 11H, C_6H_{11}), 2.10 (dd, J_1 = 4.0, J_2 = 3.5, 1H, CH), 3.62 (s, 3H, CH_3), 4.39 (d, J = 4.0 Hz, 1H, CH), 4.90 (d, J = 3.5 Hz, 1H, NH), 6.64-7.54 (m, 4H, ArH). ^{13}C NMR ($CDCl_3$, ppm): δ 169.6, 144.8 (q, CF_3, $^2J_{C\text{-}F}$ = 34 Hz), 141.5, 136.7, 131.4, 129.9, 120.7 (q, CF3, $^1J_{C\text{-}F}$ = 276 Hz), 118.9, 116.2, 58.2, 53.0, 52.3, 39.1, 29.9, 29.4, 26.2, 25.8, 25.7. ^{19}F NMR ($CDCl_3$, ppm): δ -70.65 (s, CF_3). ESI-MS (m/z): 354 (M^+).



6k

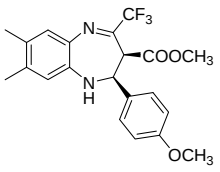
cis-Methyl 2-(naphthalen-1-yl)-4-(trifluoromethyl)-2,3-dihydro-1*H*-benzo[*b*][1,4]diazepine-3-carboxylate **6k:** Yellow solid. Yield: 85%. M.p.: 204.3~206.4℃. Anal. Calcd. for $C_{22}H_{17}F_3N_2O_2$:

C, 66.33; H, 4.30; N, 7.03. Found: C, 66.07; H, 4.04; N, 7.15. IR (KBr, cm^{-1}): *v* 1739 (C=O); 2960 (C-H); 3059 (Ar-H); 3405 (N-H). ^{1}H NMR ($CDCl_3$, ppm): δ 3.57 (s, 3H, OCH_3), 4.51 (d, J = 3.5 Hz, 1H, CH), 4.76 (d, J = 6.0 Hz, 1H, NH), 6.39 (dd, J_1 = 6.0 Hz, J_2 = 3.5 Hz, 1H, CH), 6.81-7.80 (m, 11H, ArH). ^{13}C NMR ($CDCl_3$, ppm): δ 167.8, 147.1 (q, CF_3, $^2J_{C\text{-}F}$ = 34 Hz), 143.7, 138.8, 134.9, 134.8, 131.1, 130.4, 130.1, 130.0, 129.4, 127.8, 126.7, 126.1, 124.6, 122.5, 120.9 (q, CF_3, $^1J_{C\text{-}F}$ = 275 Hz), 119.3, 118.5, 60.3, 53.5, 20.8. ^{19}F NMR ($CDCl_3$, ppm): δ -71.39 (s, CF_3). ESI-MS (m/z): 398 (M^+).



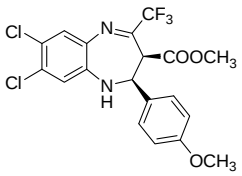
6l

cis-Methyl 2-(furan-2-yl)-4-(trifluoromethyl)-2,3-dihydro-1*H*-benzo[*b*][1,4]diazepine-3-carboxylate **6l:** Yellow solid. Yield: 81%. M.p.: 88.1~89.7℃. Anal. Calcd. for $C_{16}H_{13}F_3N_2O_3$: C, 56.81; H, 3.87; N, 8.28. Found: C, 56.70; H, 3.69; N, 8.37. IR (KBr, cm^{-1}): *v* 1730 (C=O); 2959 (C-H); 3034 (Ar-H); 3412 (N-H). ^{1}H NMR ($CDCl_3$, ppm): δ 3.65 (s, 3H, OCH_3), 4.66 (d, J = 4.0 Hz, 1H, CH), 5.04 (d, J = 5.5 Hz, 1H, NH), 5.33 (dd, J_1 = 5.5 Hz, J_2 = 4.0 Hz, 1H, CH), 5.92 (d, J_2 = 1.5 Hz, 1H, Furan-H), 6.15 (dd, J_1 = 4 Hz, J_2 = 1.5 Hz, 1H, Furan-H), 6.72-7.47 (m, 4H, ArH), 7.46 (d, J = 4 Hz, 1H, Furan-H). ^{13}C NMR ($CDCl_3$, ppm): δ 168.1, 151.9, 145.7 (q, CF_3, $^2J_{C\text{-}F}$ = 34 Hz), 142.3, 141.1, 135.9, 131.1, 128.8, 120.4 (q, CF_3, $^1J_{C\text{-}F}$ = 275 Hz), 119.7, 118.5, 113.1, 110.6, 107.4, 53.4, 52.6. ^{19}F NMR ($CDCl_3$, ppm): δ -71.40 (s, CF_3). ESI-MS (m/z): 338 (M^+).



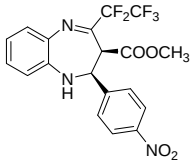
6m

methyl 2-(4-methoxyphenyl)-7,8-dimethyl-4-(trifluoromethyl)-2,3-dihydro-1*H*-benzo[*b*][1,4] diazepine-3-carboxylate **6m**: Yellow solid. Yield: 62%. M.p.: 153.8~156.3℃. Anal. Calcd. for $C_{21}H_{21}F_3N_2O_3$: C, 62.06; H, 5.21; N, 6.89. Found: C, 62.23; H, 5.33; N, 6.90. IR (KBr, cm^{-1}): *v* 1747 (C=O); 2953 (C-H); 3038 (Ar-H); 3393 (N-H). ^{1}H NMR ($CDCl_3$, ppm): δ 2.14 (s, 3H, CH_3), 2.18 (s, 3H, CH_3), 3.59 (s, 3H, OCH_3), 3.70 (s, 3H, OCH_3), 4.46 (d, J = 4.5 Hz, 1H, CH), 4.87 (d, J = 5.5 Hz, 1H, NH), 5.28 (dd, J_1 = 4.5 Hz, J_2 = 5.5 Hz, 1H, CH), 6.51-7.21 (m, 6H, ArH). ^{13}C NMR ($CDCl_3$, ppm): δ 168.1, 159.0, 145.2 (q, CF_3, $^2J_{C\text{-}F}$ = 34 Hz), 140.2, 139.3, 135.4, 133.1, 128.0, 127.2, 126.8, 120.2 (q, CF_3, $^1J_{C\text{-}F}$ = 276 Hz), 118.9, 114.0, 59.0, 55.2, 54.8, 52.9, 19.6, 18.3. ^{19}F NMR ($CDCl_3$, ppm): δ -71.24 (s, CF_3). ESI-MS (m/z): 406 (M^+).



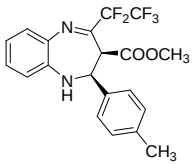
6n

methyl 7,8-dichloro-2-(4-methoxyphenyl)-4-(trifluoromethyl)-2,3-dihydro-1*H*-benzo[*b*][1,4] diazepine-3-carboxylate **6n**: Yellow oil. Yield: 60%. Anal. Calcd. for $C_{19}H_{15}C_{l2}F_3N_2O_3$: C, 51.03; H, 3.38; N, 6.26. Found: C, 51.22; H, 3.40; N, 6.28. IR (KBr, cm^{-1}): *v* 1738 (C=O); 2956 (C-H); 3064 (Ar-H); 3392 (N-H). ^{1}H NMR ($CDCl_3$, ppm): δ 3.63 (s, 3H, OCH_3), 3.72 (s, 3H, OCH_3), 4.52 (d, J = 5.0 Hz, 1H, CH), 5,31 (dd, J_1 = 5.0 Hz, J_2 = 5.5 Hz, 1H, CH), 5.41 (br-s, 1H, NH), 6.76-7.53 (m, 6H, ArH). ^{13}C NMR ($CDCl_3$, ppm): δ 167.6, 159.4, 148.2 (q, CF_3, $^2J_{C\text{-}F}$ = 35 Hz), 141.2, 136.1, 134.2, 132.2, 130.6, 129.3, 126.7, 121.4, 119.8 (q, CF_3, $^1J_{C\text{-}F}$ = 276 Hz), 118.9, 114.4, 60.6, 58.9, 55.4, 53.3, 21.2, 14.3. ^{19}F NMR ($CDCl_3$, ppm): δ -71.79 (s, CF_3). ESI-MS (m/z): 446 (M^+).



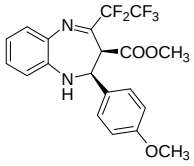
6o

cis-Methyl 2-(4-nitrophenyl)-4-(pentafluoroethyl)-2,3-dihydro-1*H*-benzo[*b*][1,4]diazepine-3-carboxylate **6o:** Yellow solid. Yield: 31%. M.p.: 150.4~151.1℃. Anal. Calcd. for $C_{19}H_{14}F_5N_3O_4$: C, 51.48; H, 3.18; N, 9.48. Found: C, 51.24; H, 3.34; N, 9.57. IR (KBr, cm^{-1}): *v* 1741 (C=O); 2956 (C-H); 3080 (Ar-H); 3421 (N-H). ^{1}H NMR ($CDCl_3$, ppm): δ 3.59 (s, 3H, OCH_3), 4.53 (d, J = 4.5 Hz, 1H, CH), 5.03 (d, J = 6.5 Hz, 1H, NH), 5.66 (dd, J_1 = 6.5 Hz, J_2 = 4.5 Hz, 1H, CH), 6.82-8.14 (m, 8H, ArH). ^{13}C NMR ($CDCl_3$, ppm): δ 167.4, 148.2, 147.5, 147.2, 146.9, 140.4, 134.9, 131.2, 130.6, 126.9, 124.1, 120.0, 119.7, 118.3, 118.2 (qt, CF_3, $^1J_{C\text{-}F}$ = 270 Hz, $^2J_{C\text{-}F}$ = 36 Hz), 109.4 (m, CF_2), 61.3, 53.9, 53.3. ^{19}F NMR ($CDCl_3$, ppm): δ -80.77 (s, CF_3), -114.19 (m, CF_2). ESI-MS (m/z): 443 (M^+).



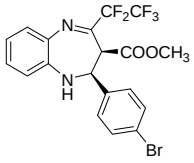
6p

cis-Methyl 4-(pentafluoroethyl)-2-*p*-tolyl-2,3-dihydro-1*H*-benzo[*b*][1,4]diazepine-3-carboxylate **6p:** Yellow solid. Yield: 61%. M.p.: 109.6~112.2℃. Anal. Calcd. for $C_{20}H_{17}F_5N_2O_2$: C, 58.25; H, 4.16; N, 6.79. Found: C, 58.34; H, 4.34; N, 6.81. IR (KBr, cm^{-1}): *v* 1720 (C=O); 2955 (C-H); 3037 (Ar-H); 3381 (N-H). ^{1}H NMR ($CDCl_3$, ppm): δ 2.27 (s, 3H, CH_3), 3.57 (s, 3H, OCH_3), 4.51 (d, J = 4.5 Hz, 1H, CH), 4.95 (d, J = 6.0 Hz, 1H, NH), 5.41 (dd, J_1 = 6.0 Hz, J_2 = 4.5 Hz, 1H, CH), 6.72-7.42 (m, 8H, ArH). ^{13}C NMR ($CDCl_3$, ppm): δ 168.1, 147.5, 147.2, 141.5, 138.1, 137.7, 135.2, 130.8, 130.5, 129.4, 125.6, 122.5, 119.6, 118.9, 118.1, 118.8 (qt, CF_3, $^1J_{C\text{-}F}$ = 285 Hz, $^2J_{C\text{-}F}$ = 36 Hz), 110.4 (m, CF_2), 60.5, 54.7, 52.9. ^{19}F NMR ($CDCl_3$, ppm): δ -80.78 (s, CF_3), -114.14 (m, CF_2). ESI-MS (m/z): 412 (M^+).



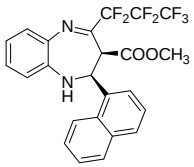
6q

cis-Methyl 2-(4-methoxyphenyl)-4-(pentafluoroethyl)-2,3-dihydro-1*H*-benzo[*b*][1,4]diazepine-3-carboxylate **6q:** Yellow solid. Yield: 51%. M.p.: 100.3~103.3℃. Anal. Calcd. for $C_{20}H_{17}F_5N_2O_3$: C, 56.08; H, 4.00; N, 6.54. Found: C, 56.26; H, 4.22; N, 6.58. IR (KBr, cm^{-1}): *v* 1728 (C=O); 2957 (C-H); 3076 (Ar-H); 3335 (N-H). ^{1}H NMR ($CDCl_3$, ppm): δ 3.58 (s, 3H, OCH_3), 3.74 (s, 3H, OCH_3), 4.47 (d, J = 5.0 Hz, 1H, CH), 4.96 (d, J = 6.0 Hz, 1H, NH), 5.40 (dd, J_1 = 6.0 Hz, J_2 = 5.0 Hz, 1H, CH), 6.73-7.43 (m, 8H, ArH). ^{13}C NMR ($CDCl_3$, ppm): δ 168.1, 159.2, 147.6, 147.4, 141.4, 135.1, 133.3, 130.8, 130.5, 126.9, 125.6, 124.1, 118.9, 118.8 (qt, CF_3, $^1J_{C\text{-}F}$ = 259 Hz, $^2J_{C\text{-}F}$ = 36 Hz), 118.2, 110.4 (m, CF_2), 60.4, 55.3, 54.8, 52.9. ^{19}F NMR ($CDCl_3$, ppm): δ -80.79 (s, CF_3), -114.16 (m, CF_2). ESI-MS (m/z): 428 (M^+).



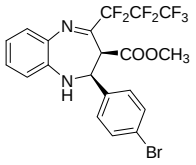
6r

cis-Methyl 2-(4-bromophenyl)-4-(pentafluoroethyl)-2,3-dihydro-1*H*-benzo[*b*][1,4]diazepine-3-carboxylate **6r:** Yellow solid. Yield: 71%. M.p.: 113.0~114.2℃. Anal. Calcd. for $C_{19}H_{14}BrF_5N_2O_2$: C, 47.82; H, 2.96; N, 5.87. Found: C, 47.96; H, 3.02; N, 5.96. IR (KBr, cm^{-1}): *v* 1724 (C=O); 2953 (C-H); 3096 (Ar-H); 3340 (N-H). ^{1}H NMR ($CDCl_3$, ppm): δ 3.55 (s, 3H, OCH_3), 4.49 (d, J = 4.5 Hz, 1H, CH), 5.07 (d, J = 6.5 Hz, 1H, NH), 5.42 (dd, J_1 = 6.5 Hz, J_2 = 4.5 Hz, 1H, CH), 6.73-7.41 (m, 8H, ArH). ^{13}C NMR ($CDCl_3$, ppm): δ 167.8, 147.4, 147.2, 140.5, 135.0, 131.9, 130.7, 128.7, 128.4, 127.5, 127,2, 126.1, 120.1, 118.7 (qt, CF_3, $^1J_{C\text{-}F}$ = 285 Hz, $^2J_{C\text{-}F}$ = 36 Hz), 118.2, 110.4 (m, CF_2), 60.6, 54.2, 53.1. ^{19}F NMR ($CDCl_3$, ppm): δ -80.73 (s, CF_3), -114.10 (m, CF_2). ESI-MS (m/z): 476 (M^+).



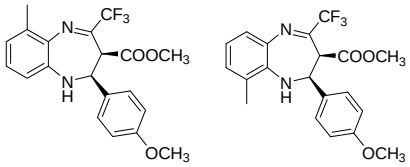
6s

cis-Methyl 2-(naphthalen-1-yl)-4-(*n*-heptafluoropropyl)-2,3-dihydro-1*H*-benzo[*b*][1,4]diazepine-3-carboxylate **6s:** Yellow solid. Yield: 74%. M.p.: 129.5~130.0℃. Anal. Calcd. for $C_{24}H_{17}F_7N_2O_2$: C, 57.84; H, 3.44; N, 5.62. Found: C, 57.99; H, 3.67; N, 5.76. IR (KBr, cm^{-1}): *v* 1728 (C=O); 2956 (C-H); 3063 (Ar-H); 3381 (N-H). ^{1}H-NMR ($CDCl_3$, ppm): δ 3.58 (s, 3H, OCH_3), 4.75 (d, J = 4.5 Hz, 1H, CH), 4.99 (d, J = 6.0 Hz, 1H, NH), 6.35 (dd, J_1 = 6.0 Hz, J_2 = 4.5 Hz, 1H, CH), 6.76-8.04 (m, 11H, ArH). ^{13}C NMR ($CDCl_3$, ppm): δ 168.0, 149.0, 141.4, 137.3, 136.9, 134.6, 133.8, 131.2, 130.3, 129.6, 129.1, 127.3, 126.0, 125.5, 124.2, 123.4, 121.6, 120.2 (qt, CF_3, $^1J_{C\text{-}F}$ = 286 Hz, $^2J_{C\text{-}F}$ = 35 Hz), 119.6, 118.5, 110.4 (m, CF_2), 108.7 (m, CF_2), 61.3, 52.9. ^{19}F NMR ($CDCl_3$, ppm): δ -79.65 (t, J = 9.4 Hz, CF_3), -112.95 (m, CF_2), -125.08 (m, CF_2). ESI-MS (m/z): 498 (M^+).



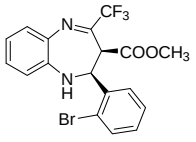
6t

cis-Methyl 2-(4-bromophenyl)-4-(*n*-heptafluoropropyl)-2,3-dihydro-1*H*-benzo[*b*][1,4]diazepine-3-carboxylate **6t:** Yellow oil. Yield: 70%. Anal. Calcd. for $C_{20}H_{14}BrF_7N_2O_2$: C, 45.56; H, 2.68; N, 5.31. Found: C, 45.87; H, 2.88; N, 5.45. IR (KBr, cm^{-1}): *v* 1753 (C=O); 2960 (C-H); 3097 (Ar-H); 3345 (N-H). ^{1}H NMR ($CDCl_3$, ppm): δ 3.50 (s, 3H, OCH_3), 4.53 (d, J = 4.5 Hz, 1H, CH), 5.28 (d, J = 6.5 Hz, 1H, NH), 5.39 (dd, J_1 = 6.5 Hz, J_2 = 4.5 Hz, 1H, CH), 6.71-7.41 (m, 8H, ArH). ^{13}C NMR ($CDCl_3$, ppm): δ 167.7, 147.5, 147.2, 141.2, 140.3, 134.9, 131.6, 131.0, 130.5, 128.7, 127.5, 118.8, 117.9, 115.5 (qt, CF_3, $^1J_{C\text{-}F}$ = 303 Hz, $^2J_{C\text{-}F}$ = 36 Hz), 113.6, 110.6 (m, CF_2), 109.5 (m, CF_2), 60.5, 54.7, 52.9. ^{19}F NMR ($CDCl_3$, ppm): δ -79.67 (t, J = 9.4 Hz, CF_3), -111.78 (m, CF_2), -124.64 (m, CF_2). ESI-MS (m/z): 526 (M^+).

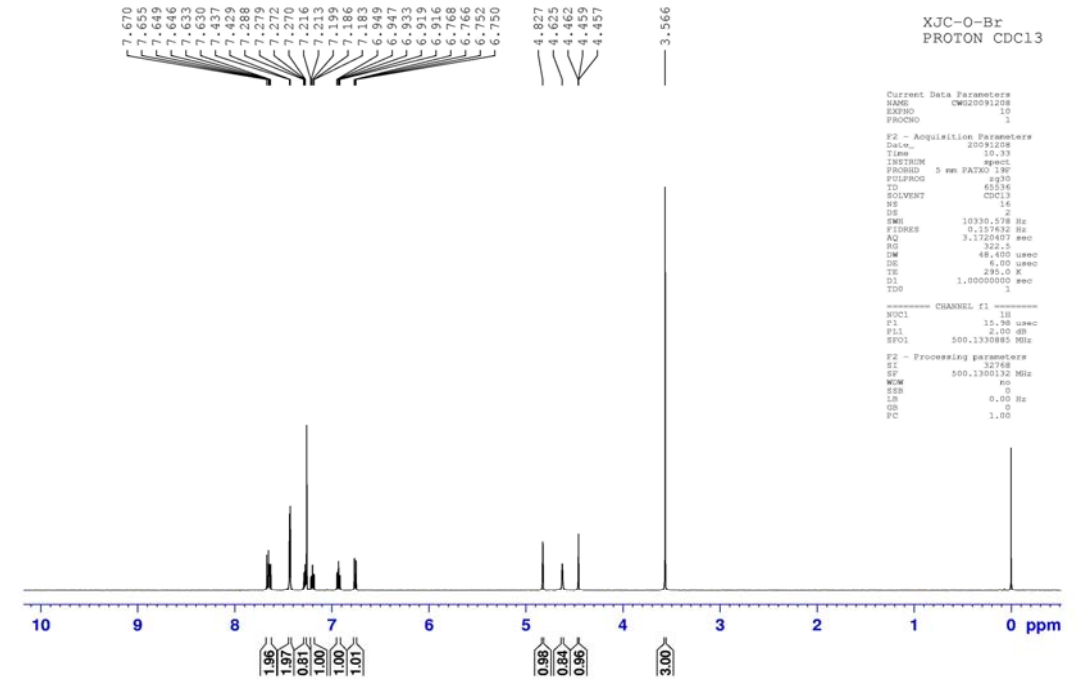


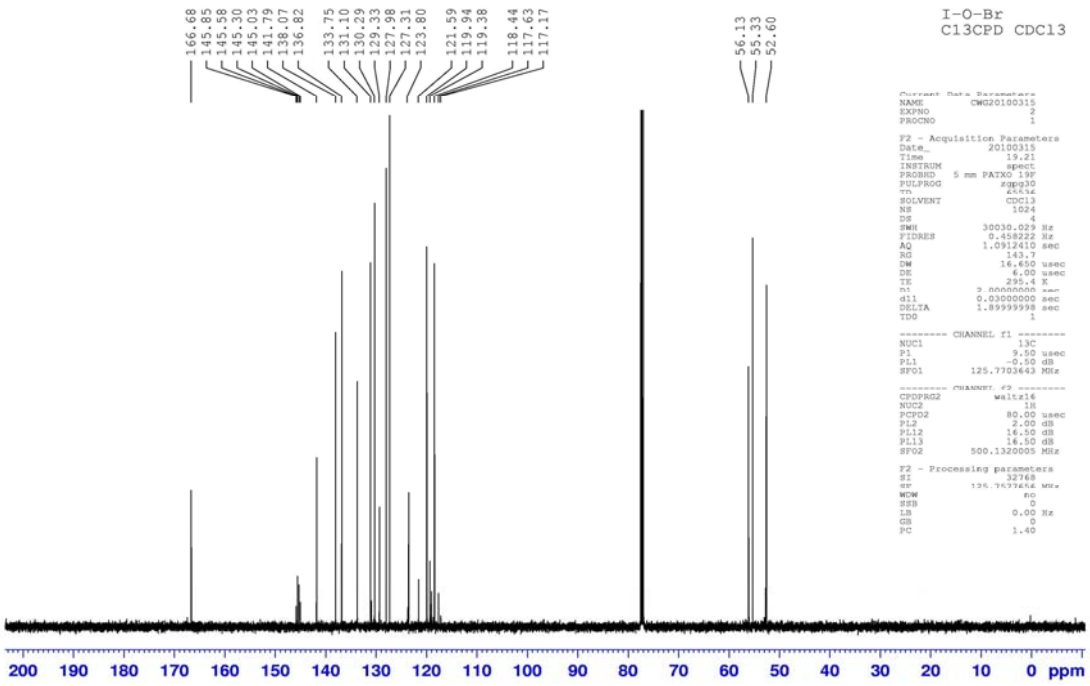
6u **6u'**

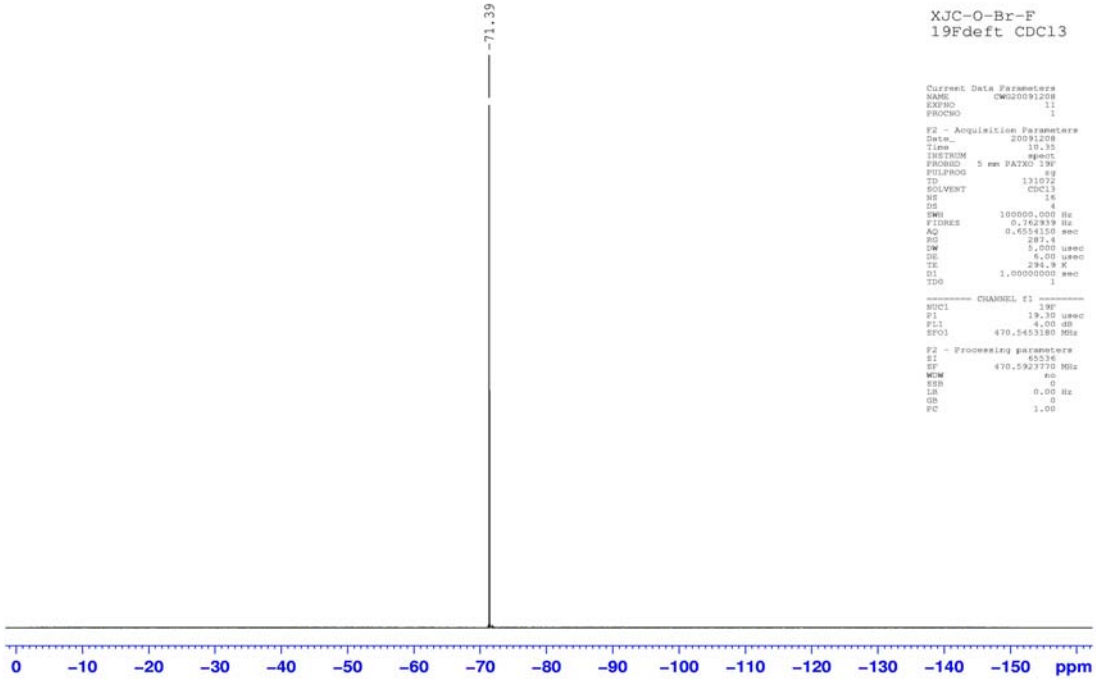
Methyl 2-(4-methoxyphenyl)-6-methyl-4-(trifluoromethyl)-2,3-dihydro-1*H*-benzo[*b*][1,4] diazepine-3-carboxylate **6u** and Methyl 2-(4-methoxyphenyl)-9-methyl-4-(trifluoromethyl)-2,3-dihydro-1*H*-benzo[*b*][1,4]diazepine-3-carboxylate **6u'**: Yellow oil. Yield: 64%. Anal. Calcd. for $C_{20}H_{19}F_3N_2O_3$: C, 61.22; H, 4.88; N, 7.14. IR (KBr, cm^{-1}): *v* 1739 (C=O); 2956 (C-H); 3003 (Ar-H); 3399 (N-H). **6u** ^{1}H NMR ($CDCl_3$, ppm): δ 2.38 (s, 3H, CH_3), 3.53 (s, 3H, OCH_3), 3.77 (s, 3H, OCH_3), 4.17 (d, J = 4.5 Hz, 1H, CH), 4.36 (br-s, 1H, NH), 5.46 (dd, J_1 = 5 Hz, J_2 = 4.5 Hz, 1H, CH), 6.60-7.35 (m, 7H, ArH). **6u'** ^{1}H NMR ($CDCl_3$, ppm): δ 2.26 (s, 1H, CH_3), 3.58 (s, 1H, OCH_3), 3.74 (s, 1H, OCH_3), 4.46 (d, J = 4.5 Hz, 0.3H, CH), 4.74 (d, J = 5.5 Hz, 0.3H, NH), 5.50 (dd, J_1 = 4.5 Hz, J_2 = 5.5 Hz, 0.3H, CH), 6.60-7.35 (m, 2.1H, ArH). ^{13}C NMR ($CDCl_3$, ppm): δ 168.3, 168.2, 159.4, 159.3, 148.4 (q, CF_3, $^2J_{C\text{-}F}$ = 35 Hz), 147.0 (q, CF_3, $^2J_{C\text{-}F}$ = 34 Hz), 140.7, 140.3, 139.7, 133.5, 133.0, 132.9, 132.2, 131.8, 131.5, 131.1, 129.8, 127.9, 127.4, 126.9, 124.6, 122.4, 120.1 (q, CF_3, $^1J_{C\text{-}F}$ = 276 Hz), 117.6, 114.5, 114.3, 114.2, 60.9, 60.6, 55.4, 55.3, 54.7, 54.2, 53.0, 52.9, 21.2, 19.2, 18.4, 14.4. ^{19}F NMR ($CDCl_3$, ppm): δ -71.25 (s, CF_3), -71.37 (s, CF_3). ESI-MS (m/z): 392 (M^+).

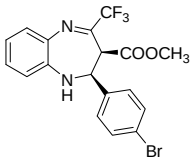


6a

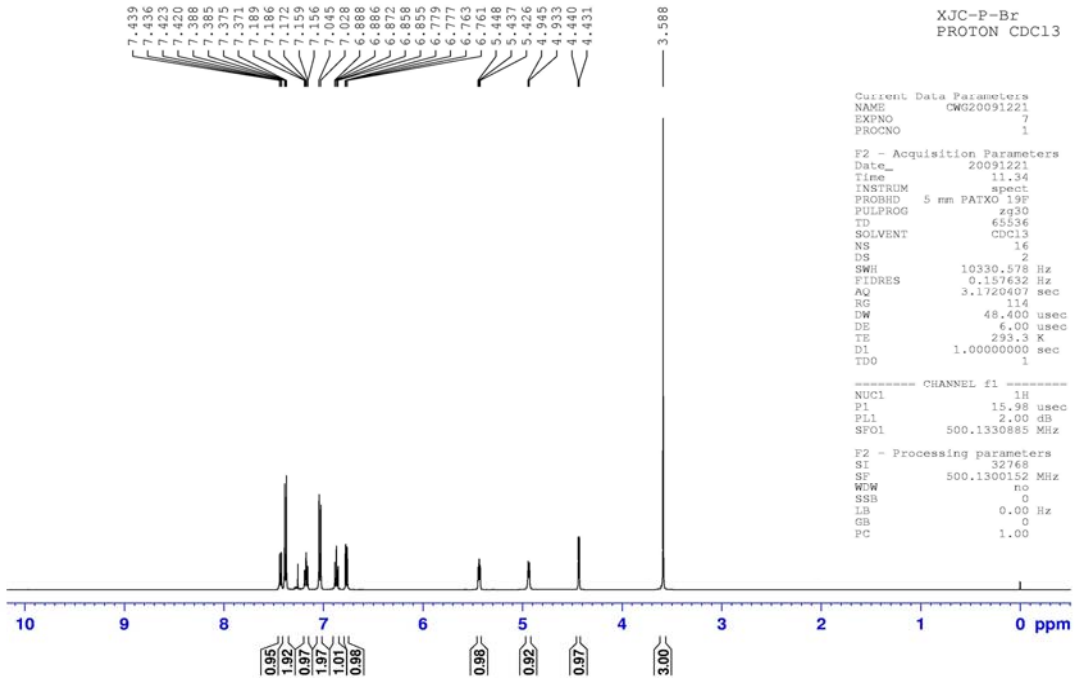

7.670
7.655
7.649
7.646
7.633
7.630
7.437
7.429
7.288
7.279
7.272
7.270
7.216
7.213
7.199
7.186
7.183
6.949
6.947
6.933
6.919
6.916
6.768
6.766
6.752
6.750
4.827
4.625
4.462
4.459
4.457
3.566
XJC-O-Br
PROTON CDCl3
10
9
8
7
6
5
4
3
2
1
0 ppm
1.96
1.97
0.81
1.00
1.00
1.01
0.98
0.84
0.96
3.00

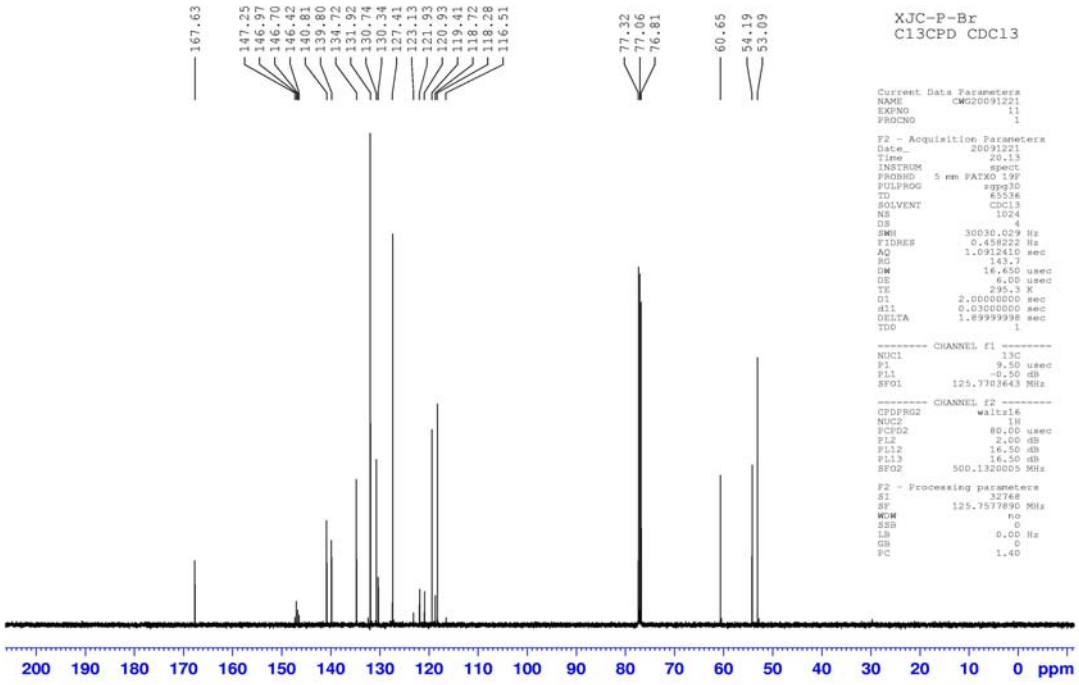

166.68
145.85
145.58
145.30
145.03
141.79
138.07
136.82
133.75
131.10
130.29
129.33
127.98
127.31
123.80
121.59
119.94
119.38
118.44
117.63
117.17
56.13
55.33
52.60
I-O-Br
C13CPD CDCl3
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0 ppm

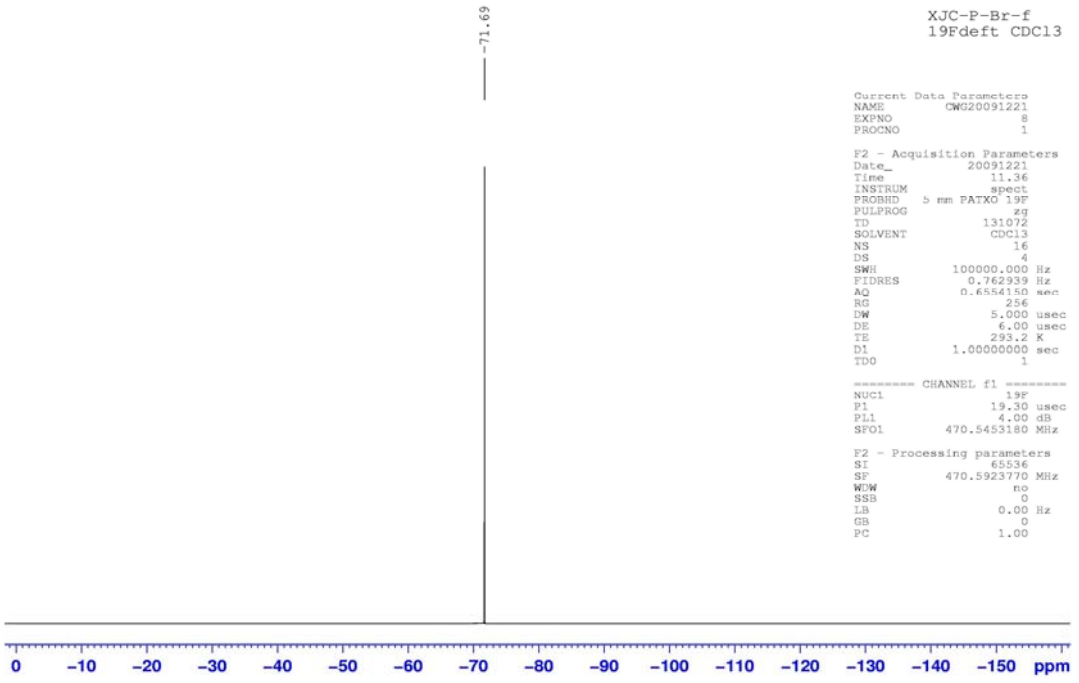

-71.39
XJC-O-Br-F
19Fdeft CDCl3
0 -10 -20 -30 -40 -50 -60 -70 -80 -90 -100 -110 -120 -130 -140 -150 ppm

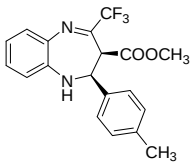

CF3
N
COOCH3
N
H
Br

6b

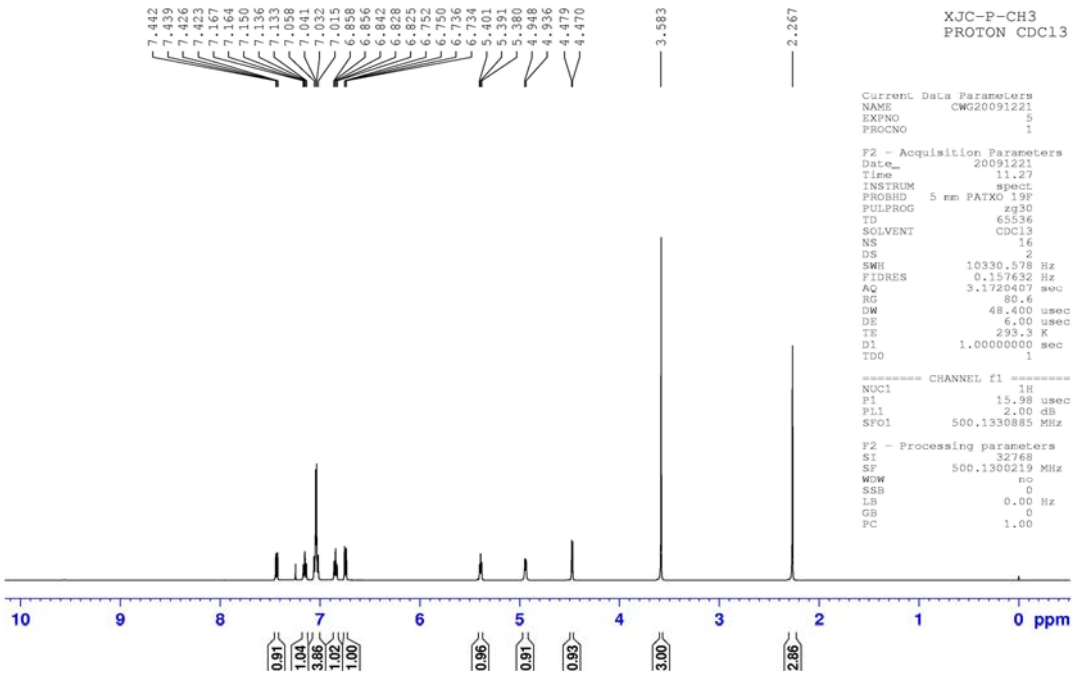

7.439
7.436
7.423
7.420
7.388
7.385
7.375
7.371
7.189
7.186
7.172
7.159
7.156
7.045
7.028
6.888
6.886
6.872
6.858
6.855
6.779
6.777
6.763
6.761
5.448
5.437
5.426
4.945
4.933
4.440
4.431
3.588
XJC-P-Br
PROTON CDCl3
10 9 8 7 6 5 4 3 2 1 0 ppm
0.95
1.92
0.97
1.97
1.01
0.98
0.98
0.92
0.97
3.00

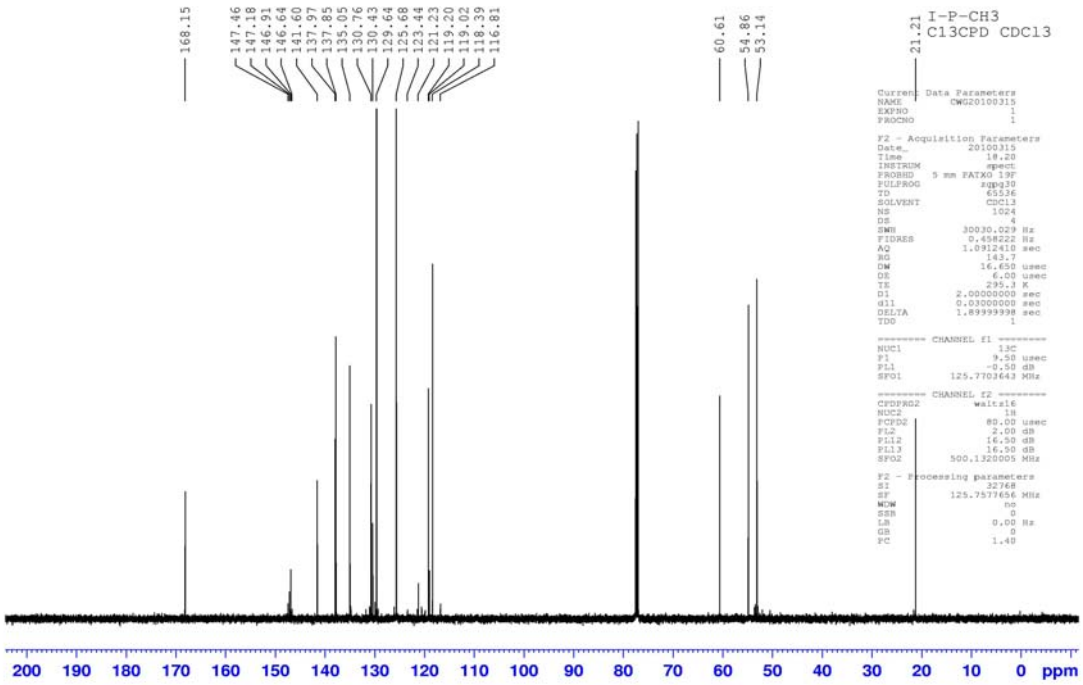

167.63
147.25
146.97
146.70
146.42
140.81
139.80
134.72
131.92
130.74
130.34
127.41
123.13
121.93
120.93
119.41
118.72
118.28
116.51
77.32
77.06
76.81
60.65
54.19
53.09
XJC-P-Br
C13CPD CDC13
200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

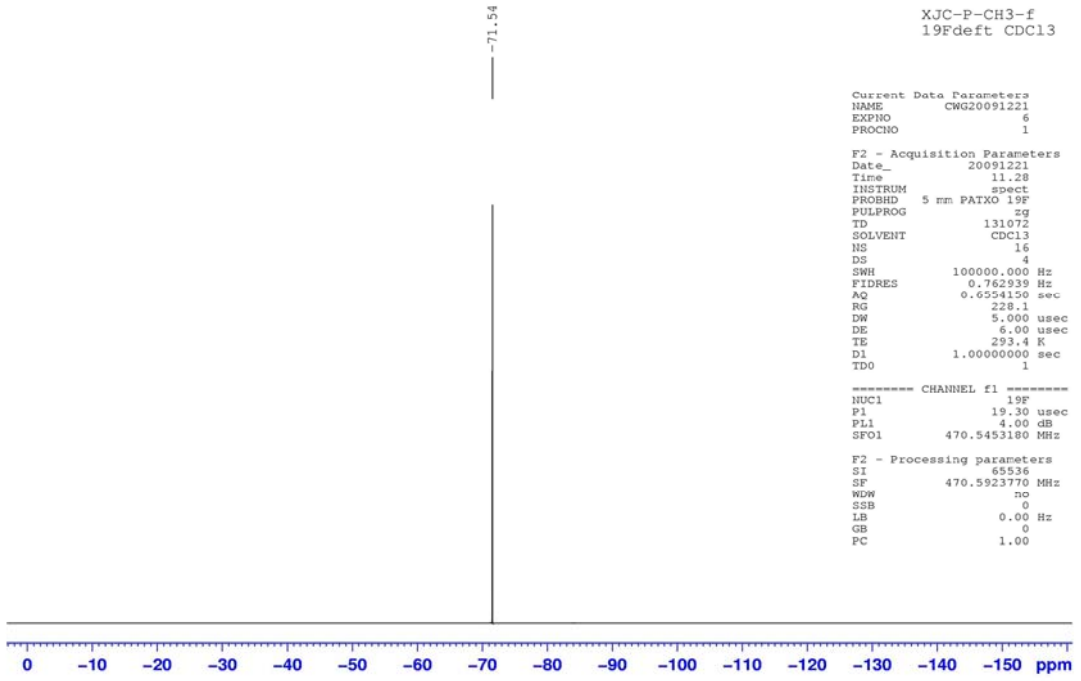

-71.69
XJC-P-Br-f
19Fdeft CDC13
0 −10 −20 −30 −40 −50 −60 −70 −80 −90 −100 −110 −120 −130 −140 −150 ppm

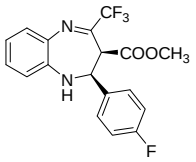

CF3
N
COOCH3
N
H
CH3

6c

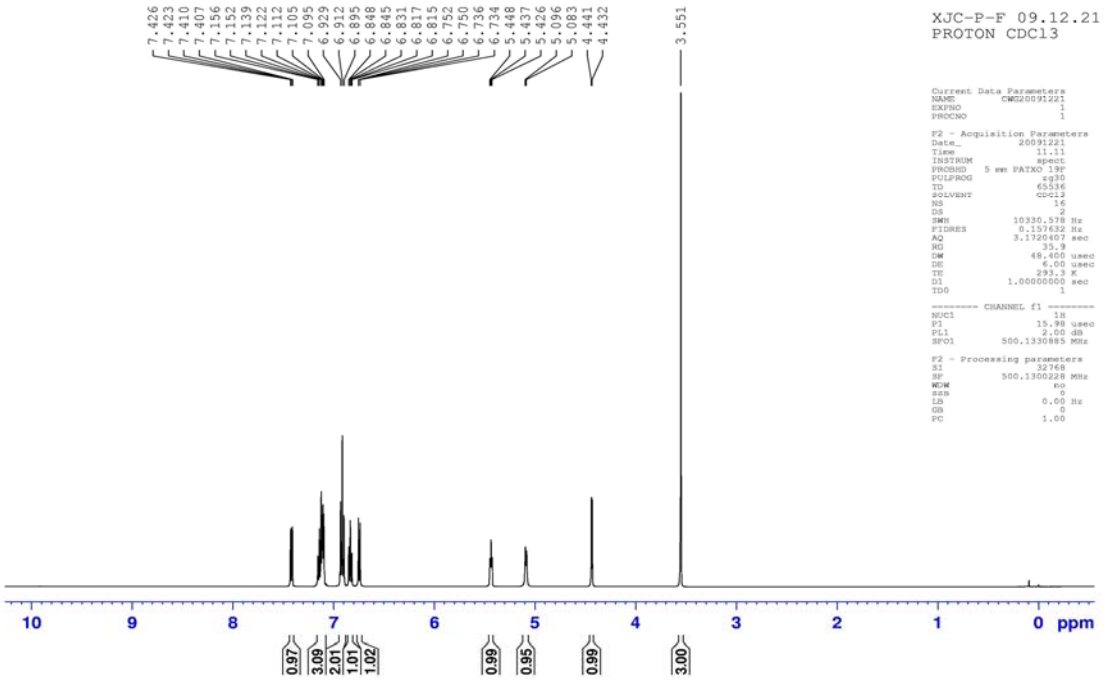

7.442
7.439
7.426
7.423
7.167
7.164
7.150
7.136
7.133
7.058
7.041
7.032
7.015
6.858
6.856
6.842
6.828
6.825
6.752
6.750
6.736
6.734
5.401
5.391
5.380
4.948
4.936
4.479
4.470
3.583
2.267
XJC-P-CH3
PROTON CDC13
Current Data Parameters
NAME CWG20091221
EXPNO 5
PROCNO 1
F2 - Acquisition Parameters
Date_ 20091221
Time 11.27
INSTRUM spect
PROBHD 5 mm PATXO 19F
PULPROG zg30
TD 65536
SOLVENT CDC13
NS 16
DS 2
SWH 10330.578 Hz
FIDRES 0.157632 Hz
AQ 3.1720407 sec
RG 80.6
DW 48.400 usec
DE 6.00 usec
TE 295.3 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 15.98 usec
PL1 2.00 dB
SFO1 500.1330885 MHz
F2 - Processing parameters
SI 32768
SF 500.1300219 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
PC 1.00
10 9 8 7 6 5 4 3 2 1 0 ppm
0.91 1.04 3.86 1.02 1.00 0.96 0.91 0.93 3.00 2.86

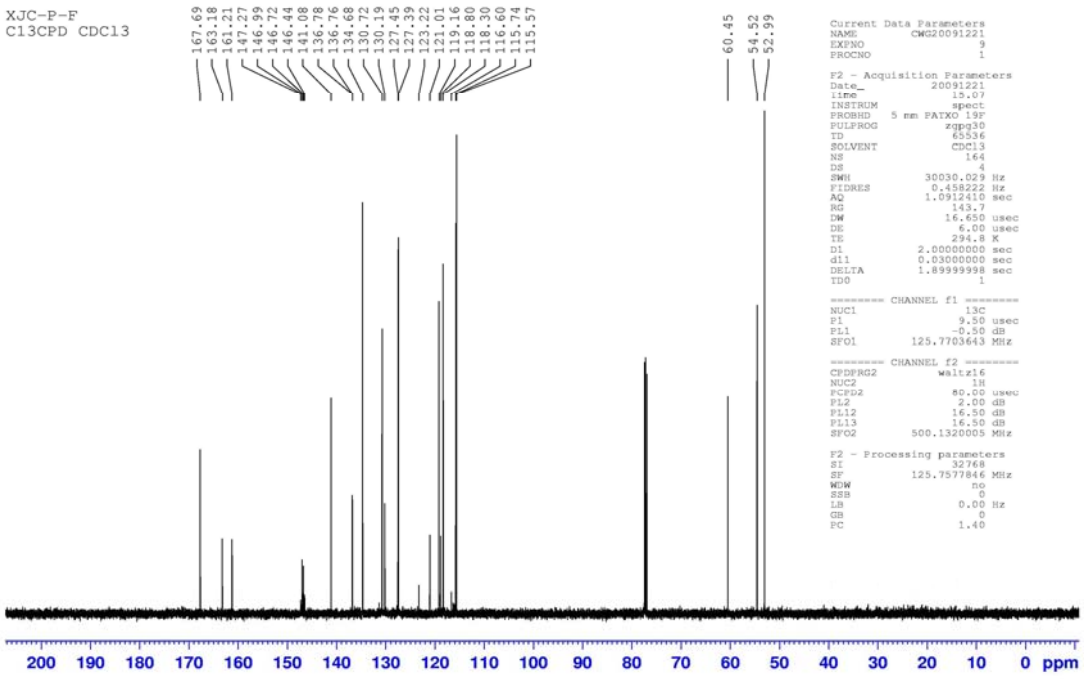

168.15
147.46
147.18
146.91
146.64
141.60
137.97
137.85
135.05
130.76
130.43
129.64
125.68
123.44
121.23
119.20
119.02
118.39
116.81
60.61
54.86
53.14
21.21
I-P-CH3
C13CPD CDC13
Current Data Parameters
NAME CWG20100315
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20100315
Time 18.20
INSTRUM spect
PROBHD 5 mm PATXO 19F
PULPROG zgpg30
TD 65536
SOLVENT CDC13
NS 1024
DS 4
SWH 30030.029 Hz
FIDRES 0.458222 Hz
AQ 1.0912410 sec
RG 143.7
DW 16.650 usec
DE 6.00 usec
TE 295.3 K
D1 2.00000000 sec
d11 0.03000000 sec
DELTA 1.89999998 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 9.50 usec
PL1 -0.50 dB
SFO1 125.7703643 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 2.00 dB
PL12 16.50 dB
PL13 16.50 dB
SFO2 500.1320005 MHz
F2 - Processing parameters
SI 32768
SF 125.7577656 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
PC 1.40
200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

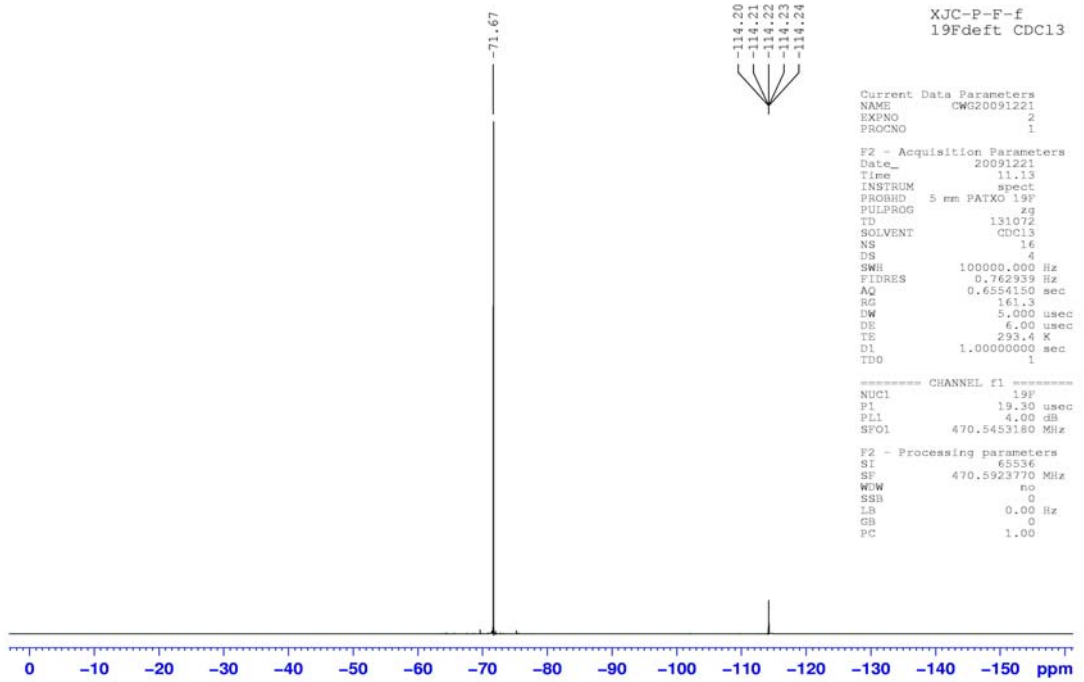

-71.54
XJC-P-CH3-f
19Fdeft CDCl3
Current Data Parameters
NAME CWG20091221
EXPNO 6
PROCNO 1
F2 - Acquisition Parameters
Date_ 20091221
Time 11.28
INSTRUM spect
PROBHD 5 mm PATXO 19F
PULPROG zg
TD 131072
SOLVENT CDCl3
NS 16
DS 4
SWH 100000.000 Hz
FIDRES 0.762939 Hz
AQ 0.6554150 sec
RG 228.1
DW 5.000 usec
DE 6.00 usec
TE 293.4 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 19F
P1 19.30 usec
PL1 4.00 dB
SFO1 470.5453180 MHz
F2 - Processing parameters
SI 65536
SF 470.5923770 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
PC 1.00
0 -10 -20 -30 -40 -50 -60 -70 -80 -90 -100 -110 -120 -130 -140 -150 ppm

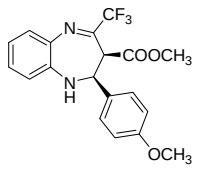

CF3
N
COOCH3
N
H
F

6d

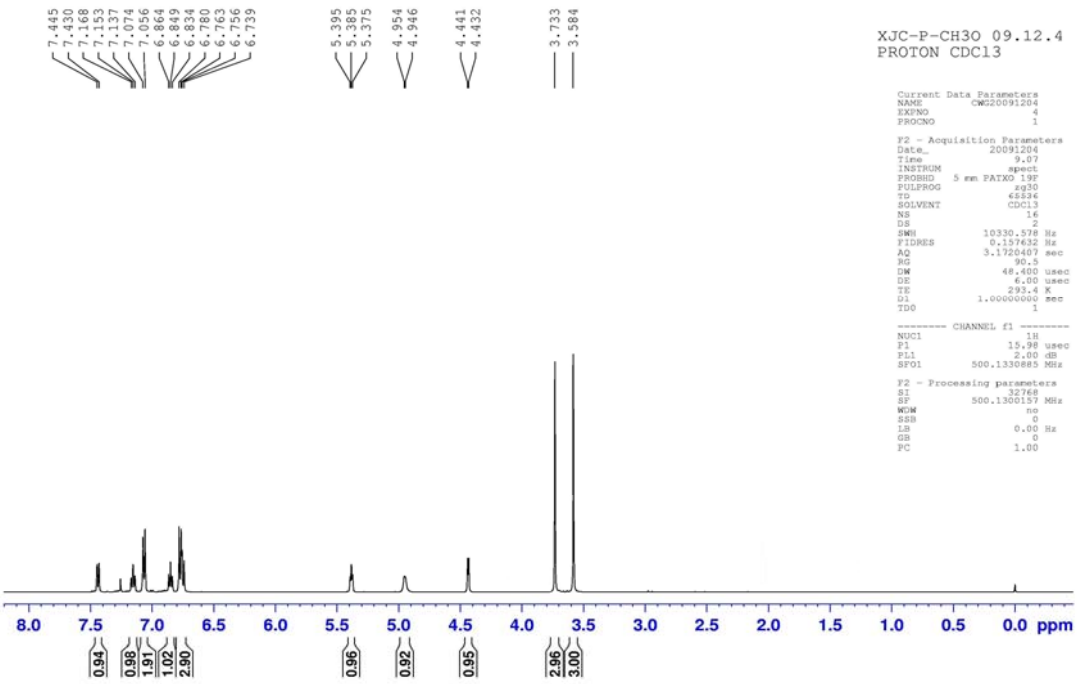

7.426
7.423
7.410
7.407
7.156
7.152
7.139
7.122
7.112
7.105
7.095
6.929
6.912
6.895
6.848
6.845
6.831
6.817
6.815
6.752
6.750
6.736
6.734
5.448
5.437
5.426
5.096
5.083
4.441
4.432
3.551
XJC-P-F 09.12.21
PROTON CDCl3
Current Data Parameters
NAME CWG20091221
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20091221
Time 11.11
INSTRUM spect
PROBHD 5 mm PATXO 19F
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 10330.578 Hz
FIDRES 0.157632 Hz
AQ 3.1720407 sec
RG 35.9
DW 48.400 usec
DE 6.00 usec
TE 293.3 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 15.98 usec
PL1 2.00 dB
SFO1 500.1330885 MHz
F2 - Processing parameters
SI 32768
SF 500.1300228 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
PC 1.00
10 9 8 7 6 5 4 3 2 1 0 ppm
0.97
3.09
2.01
1.01
1.02
0.99
0.95
0.99
3.00

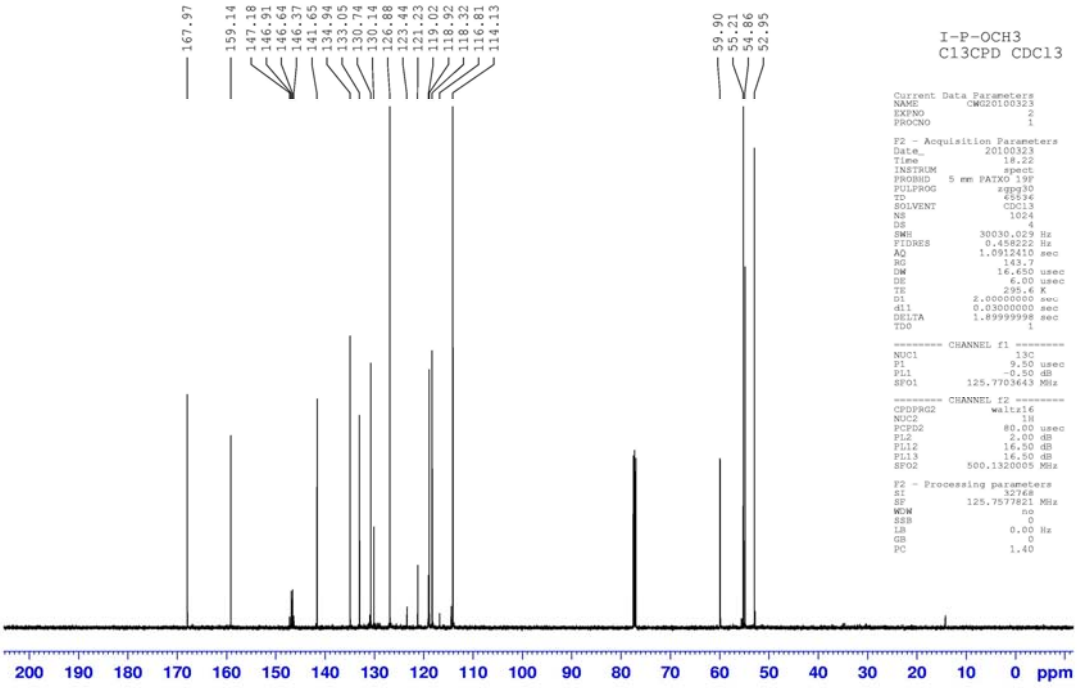

XJC-P-F
C13CPD CDCl3
167.69
163.18
161.21
147.27
146.99
146.72
146.44
141.08
136.78
136.76
134.68
130.72
130.19
127.45
127.39
123.22
121.01
119.16
118.80
118.30
116.60
115.74
115.57
60.45
54.52
52.99
Current Data Parameters
NAME CWG20091221
EXPNO 9
PROCNO 1
F2 - Acquisition Parameters
Date_ 20091221
Time 15.07
INSTRUM spect
PROBHD 5 mm PATXO 19F
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 164
DS 4
SWH 30030.029 Hz
FIDRES 0.458222 Hz
AQ 1.0912410 sec
RG 143.7
DW 16.650 usec
DE 6.00 usec
TE 294.8 K
D1 2.00000000 sec
d11 0.03000000 sec
DELTA 1.89999998 sec
TD0 1
CHANNEL f1
NUC1 13C
P1 9.50 usec
PL1 -0.50 dB
SFO1 125.7703643 MHz
CHANNEL f2
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 2.00 dB
PL12 16.50 dB
PL13 16.50 dB
SFO2 500.1320005 MHz
F2 - Processing parameters
SI 32768
SF 125.7577846 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
PC 1.40
200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

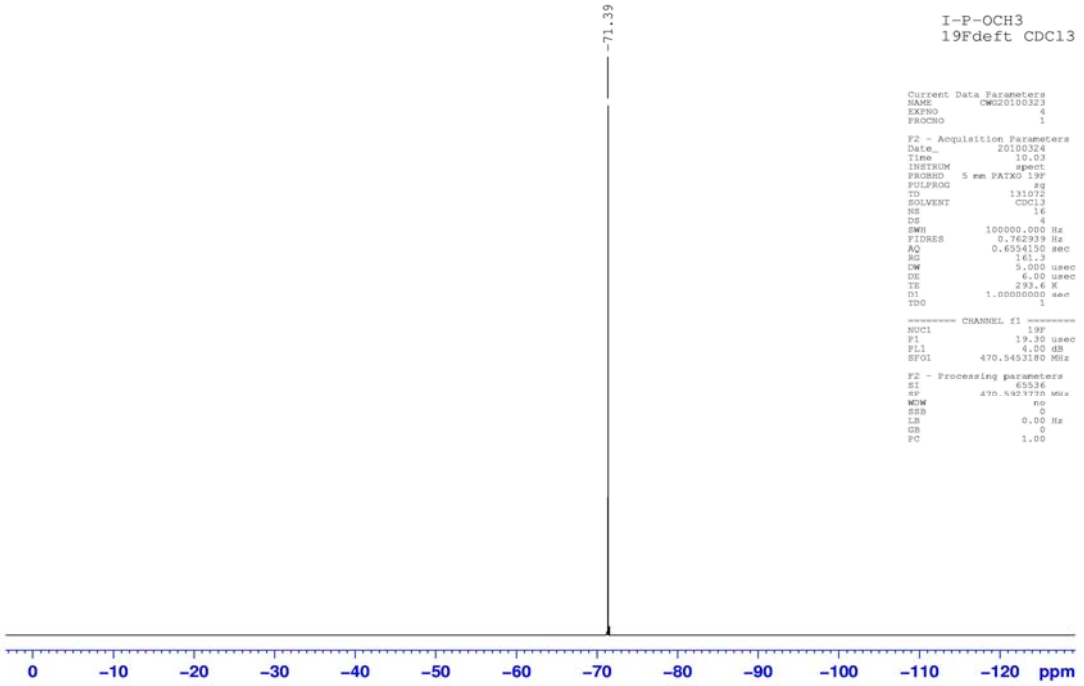

-71.67
-114.20
-114.21
-114.22
-114.23
-114.24
XJC-P-F-f
19Fdeft CDCl3
Current Data Parameters
NAME CWG20091221
EXPNO 2
PROCNO 1
F2 - Acquisition Parameters
Date_ 20091221
Time 11.13
INSTRUM spect
PROBHD 5 mm PATXO 19F
PULPROG zg
TD 131072
SOLVENT CDCl3
NS 16
DS 4
SWH 100000.000 Hz
FIDRES 0.762939 Hz
AQ 0.6554150 sec
RG 161.3
DW 5.000 usec
DE 6.00 usec
TE 293.4 K
D1 1.00000000 sec
TD0 1
CHANNEL f1
NUC1 19F
P1 19.30 usec
PL1 4.00 dB
SFO1 470.5453180 MHz
F2 - Processing parameters
SI 65536
SF 470.5923770 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
PC 1.00
0 -10 -20 -30 -40 -50 -60 -70 -80 -90 -100 -110 -120 -130 -140 -150 ppm

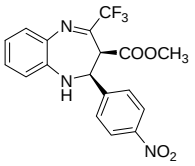

CF3
N
COOCH3
N
H
OCH3

6e

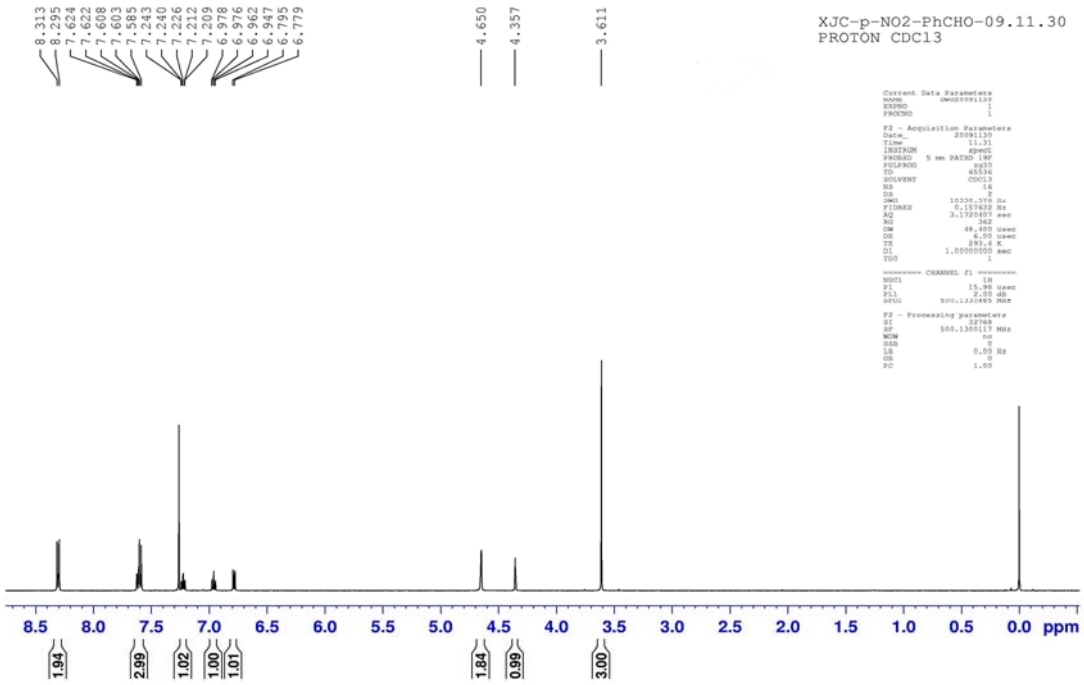

7.445
7.430
7.168
7.153
7.137
7.074
7.056
6.864
6.849
6.834
6.780
6.763
6.756
6.739
5.395
5.385
5.375
4.954
4.946
4.441
4.432
3.733
3.584
XJC-P-CH3O 09.12.4
PROTON CDC13
8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 ppm
0.94
0.98
1.91
1.02
2.90
0.96
0.92
0.95
2.96
3.00

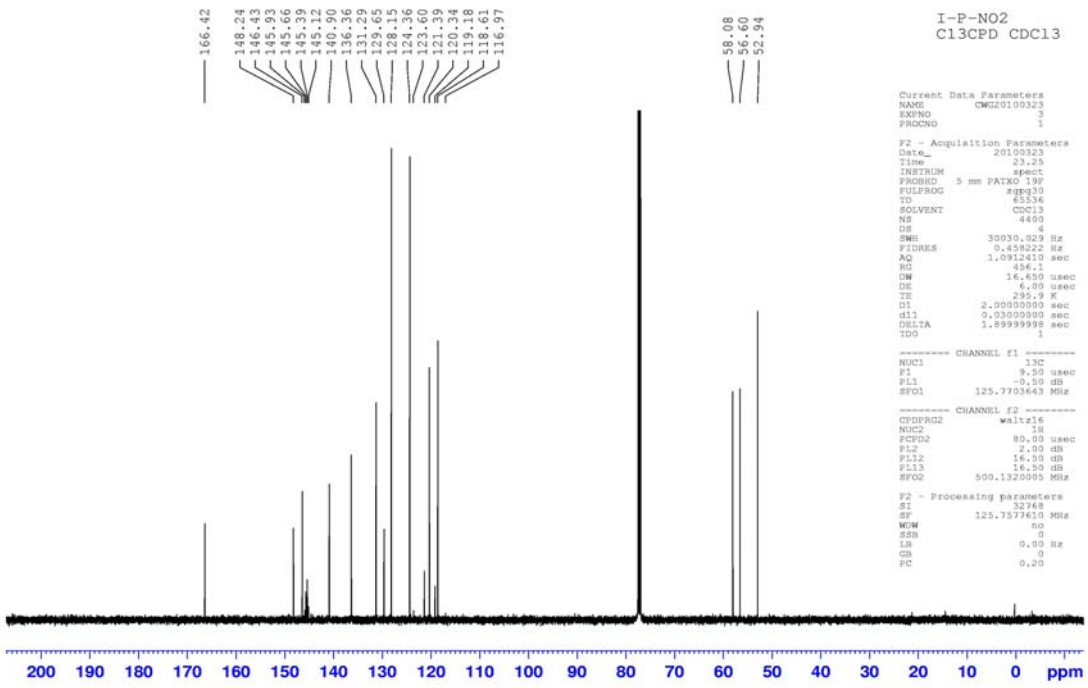

167.97
159.14
147.18
146.91
146.64
146.37
141.65
134.94
133.05
130.74
130.14
126.88
123.44
121.23
119.02
118.92
118.32
116.81
114.13
59.90
55.21
54.86
52.95
I-P-OCH3
C13CPD CDC13
200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

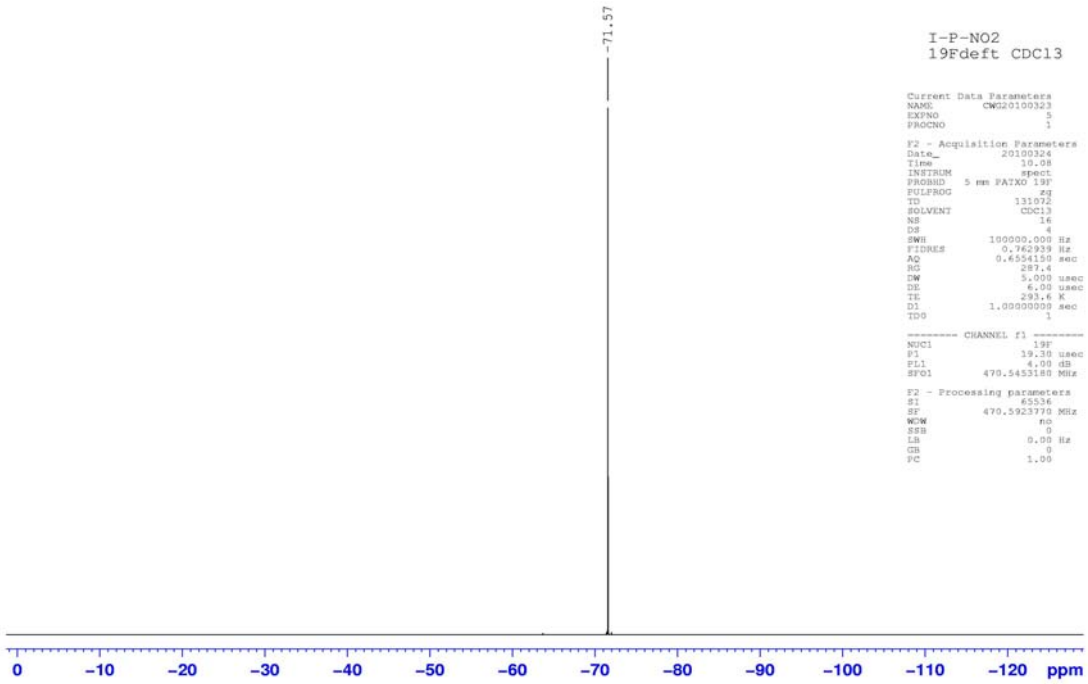

I-P-OCH3
19Fdeft CDCl3
-71.39
0
-10
-20
-30
-40
-50
-60
-70
-80
-90
-100
-110
-120
ppm

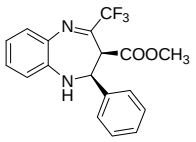

CF3
N
COOCH3
N
H
NO2

6f

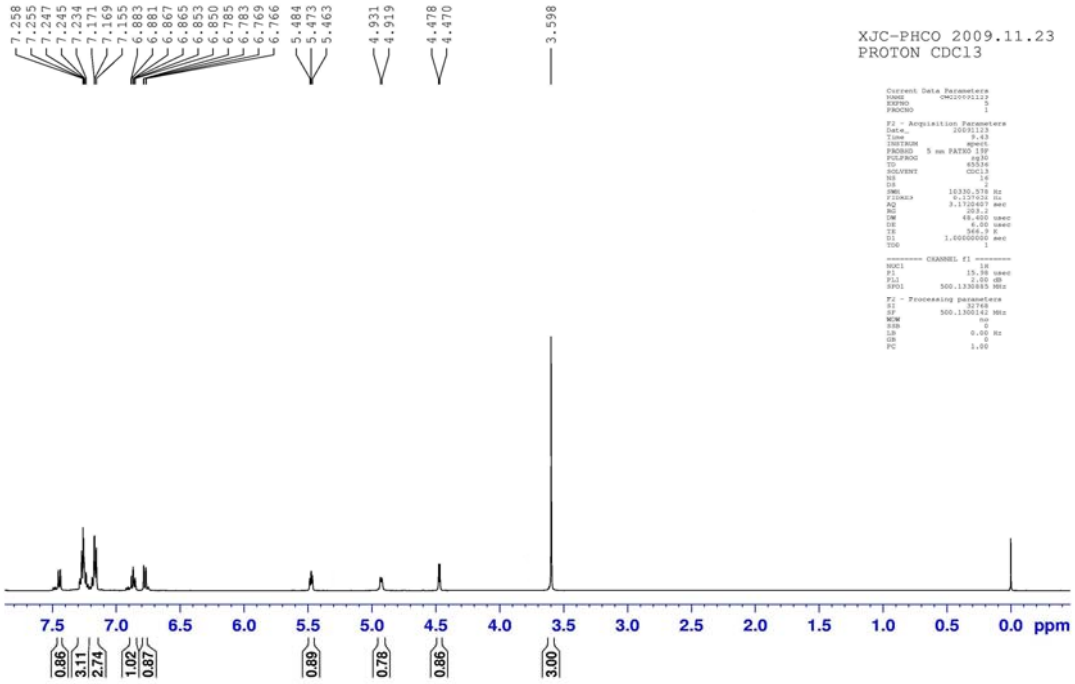

8.313
8.295
7.624
7.622
7.608
7.603
7.585
7.243
7.240
7.226
7.212
7.209
6.978
6.976
6.962
6.947
6.795
6.779
4.650
4.357
3.611
XJC-p-NO2-PhCHO-09.11.30
PROTON CDCl3
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
ppm
1.94
2.99
1.02
1.00
1.01
1.84
0.99
3.00

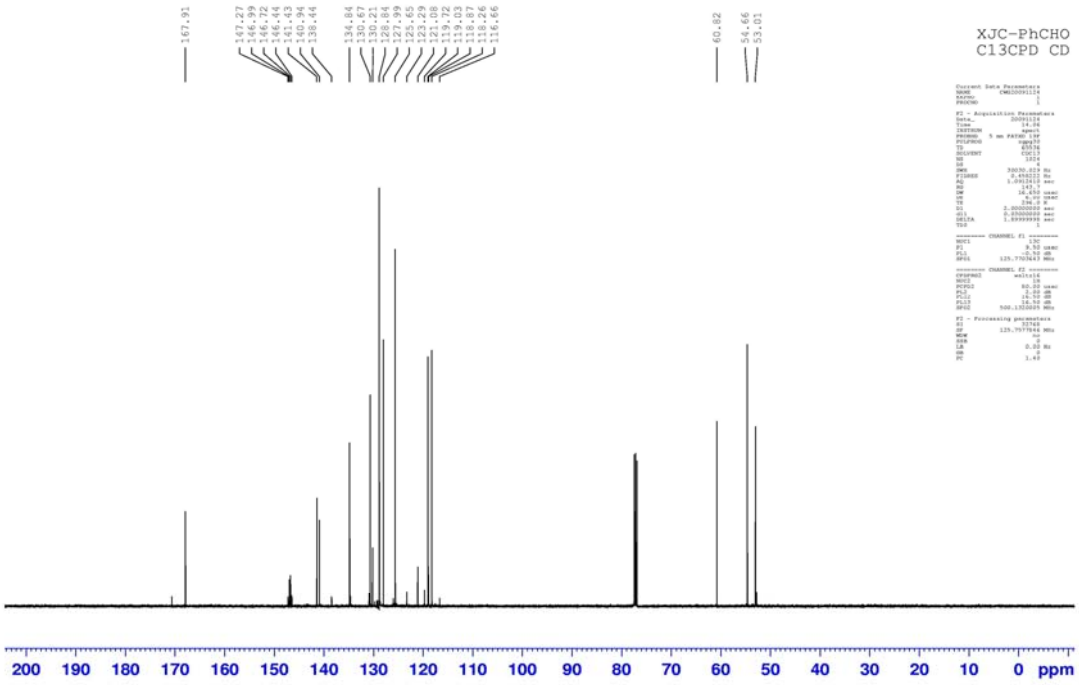

166.42
148.24
146.43
145.93
145.66
145.39
145.12
140.90
136.36
131.29
129.65
128.15
124.36
123.60
121.39
120.34
119.18
118.61
116.97
58.08
56.60
52.94
I-P-NO2
C13CPD CDC13
200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

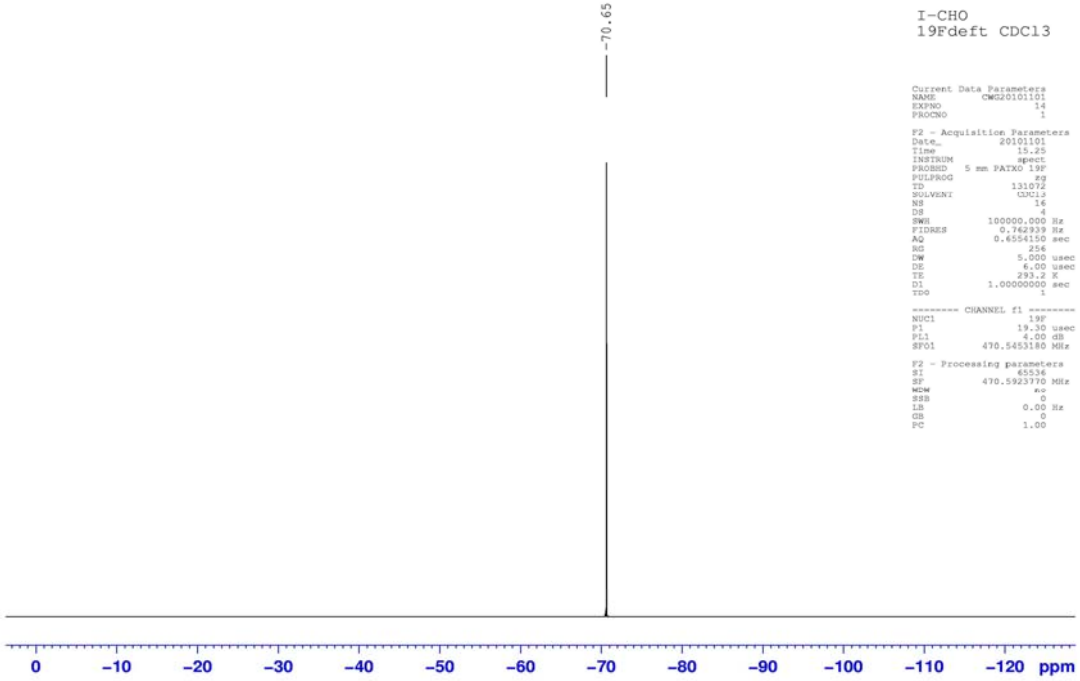

-71.57
I-P-NO2
19Fdeft CDC13
0 -10 -20 -30 -40 -50 -60 -70 -80 -90 -100 -110 -120 ppm

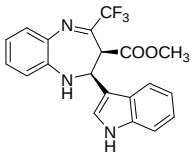

CF3
N
COOCH3
N
H

6g

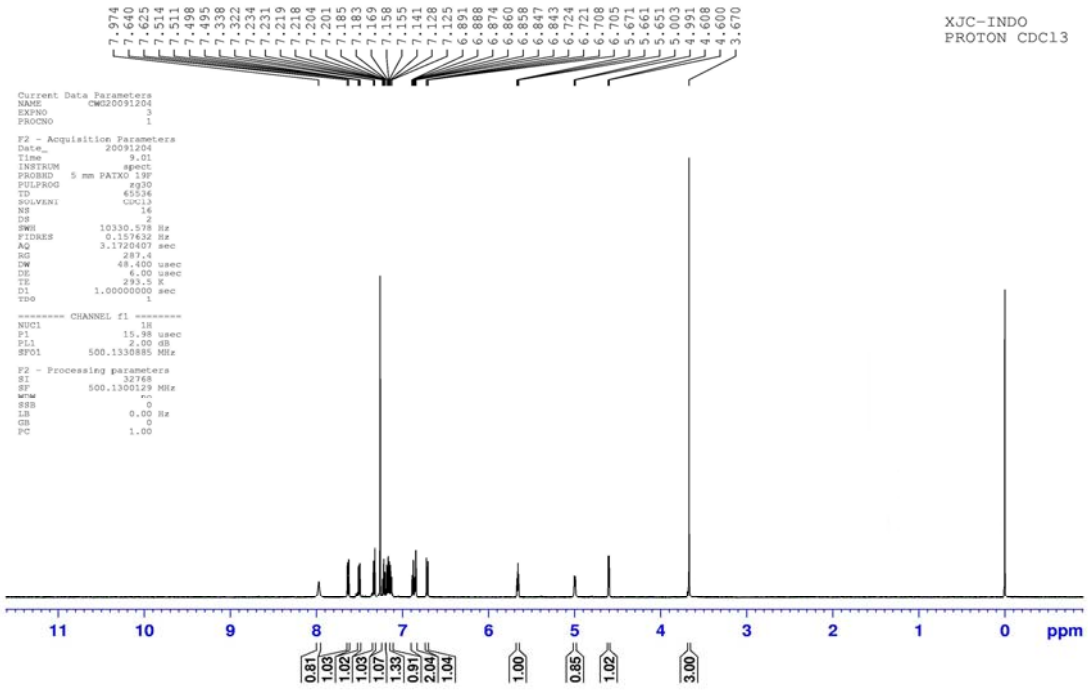

7.258
7.255
7.247
7.245
7.234
7.171
7.169
7.155
6.883
6.881
6.867
6.865
6.853
6.850
6.785
6.783
6.769
6.766
5.484
5.473
5.463
4.931
4.919
4.478
4.470
3.598
XJC-PHCO 2009.11.23
PROTON CDCl3
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0 ppm
0.86
3.11
2.74
1.02
0.87
0.89
0.78
0.86
3.00

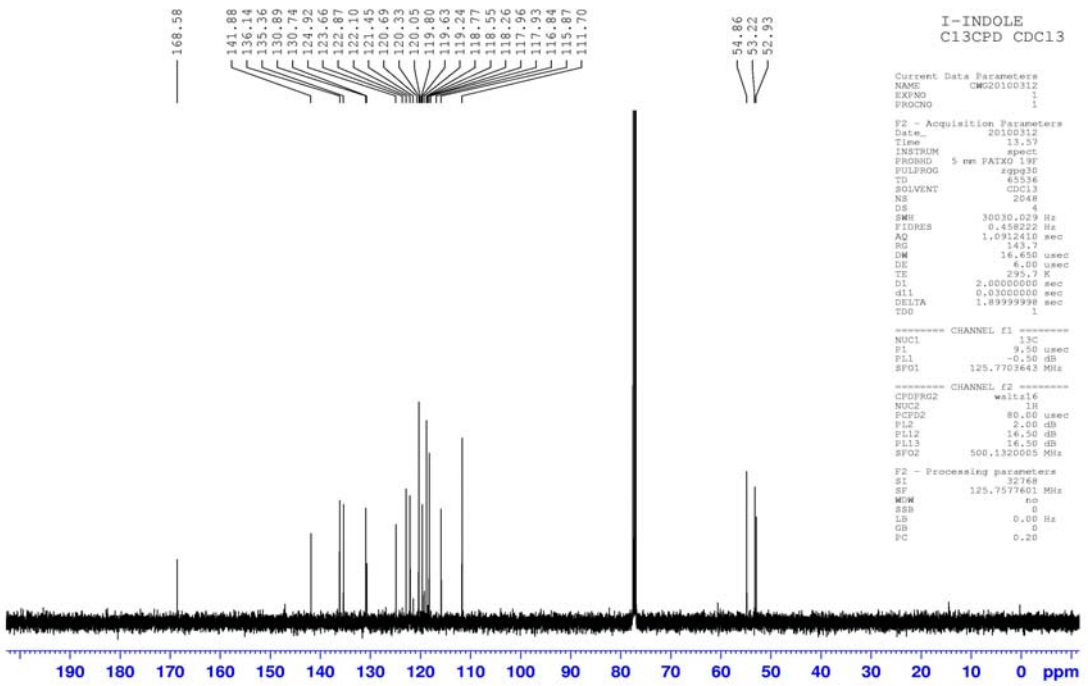

167.91
147.27
146.99
146.72
146.44
141.33
140.94
138.44
134.84
130.67
130.21
128.84
127.99
125.65
123.29
121.08
119.72
119.03
118.87
118.26
116.66
60.82
54.66
53.01
XJC-PhCHO
C13CPD CD
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0 ppm

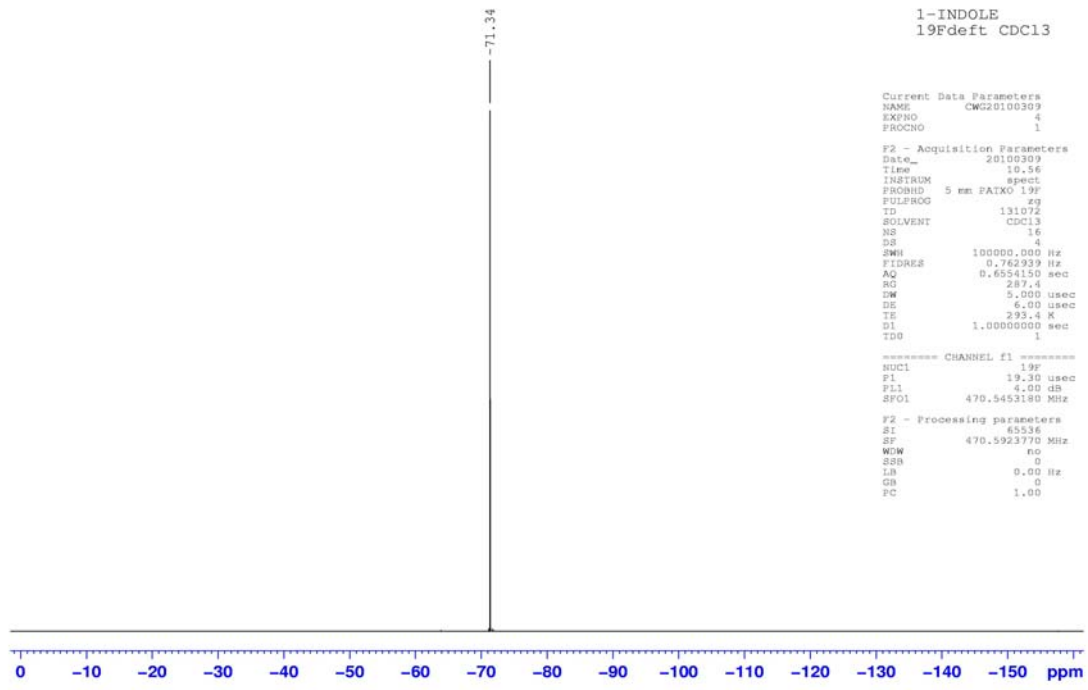

I-CHO
19Fdeft CDC13
-70.65
0
-10
-20
-30
-40
-50
-60
-70
-80
-90
-100
-110
-120
ppm

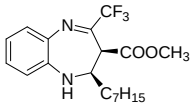

CF3
N
COOCH3
N
H
N
H

6h

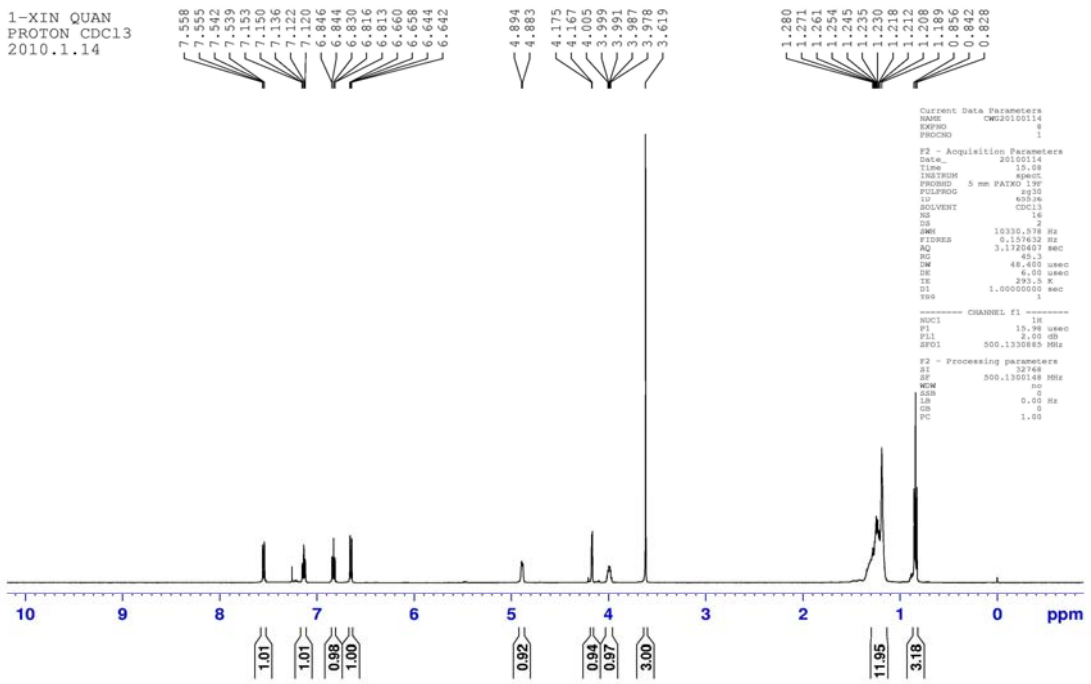

XJC-INDO
PROTON CDC13
11
10
9
8
7
6
5
4
3
2
1
0
ppm

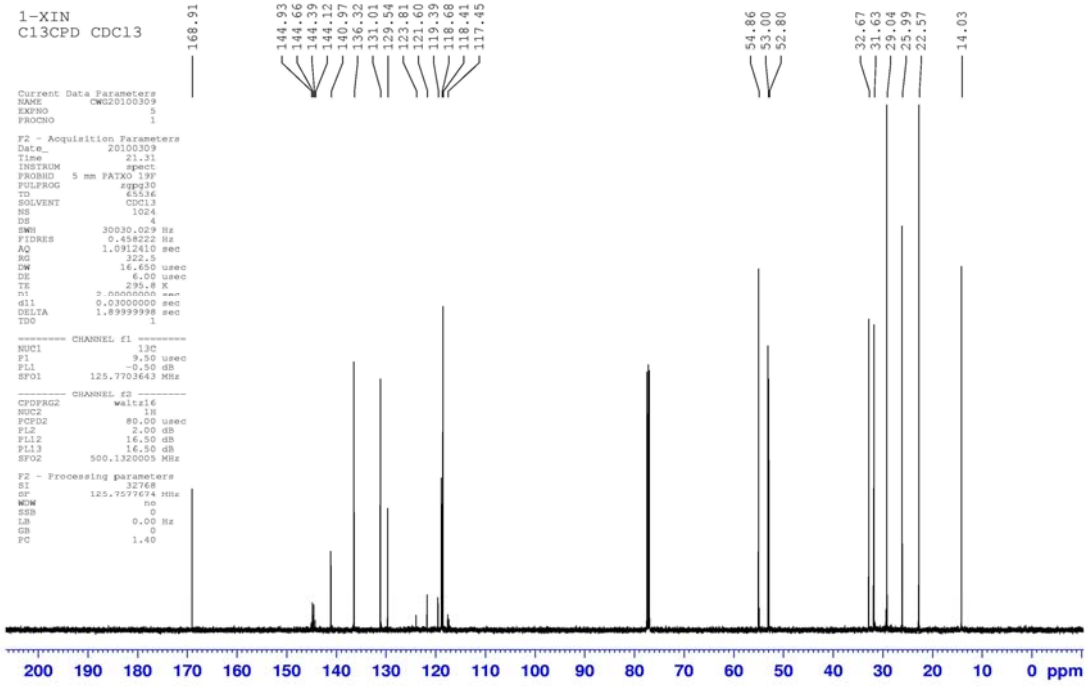

168.58
141.88
136.14
135.36
130.89
130.74
124.92
123.66
122.87
122.10
121.45
120.69
120.33
120.05
119.80
119.63
119.24
118.77
118.55
118.26
117.96
117.93
116.84
115.87
111.70
54.86
53.22
52.93
I-INDOLE
C13CPD CDC13
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

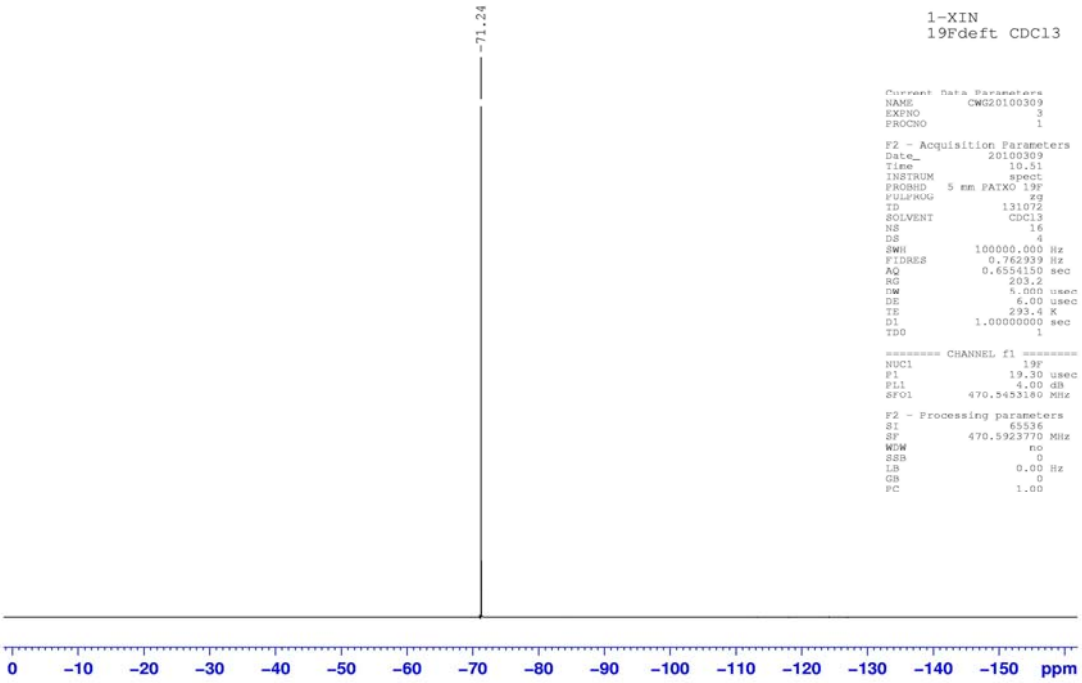

-71.34
1-INDOLE
19Fdeft CDCl3
0 -10 -20 -30 -40 -50 -60 -70 -80 -90 -100 -110 -120 -130 -140 -150 ppm

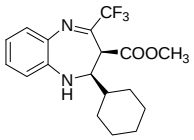

CF3
N
COOCH3
N
H
C7H15

6i

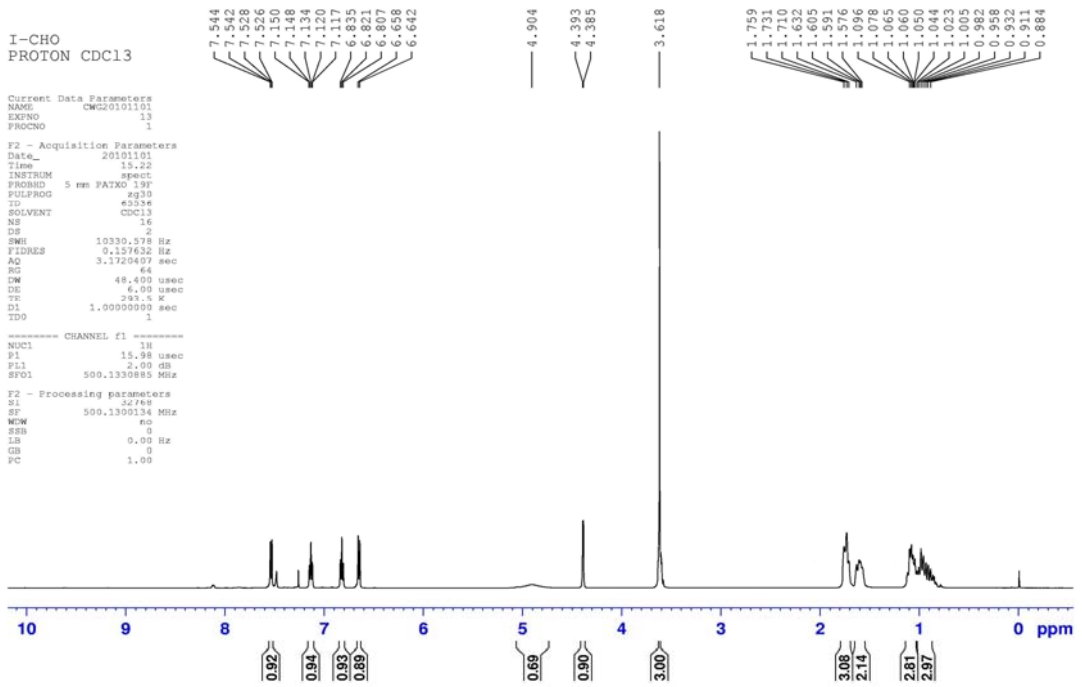

1-XIN QUAN
PROTON CDCl3
2010.1.14
7.558
7.555
7.542
7.539
7.153
7.150
7.136
7.122
7.120
6.846
6.844
6.830
6.816
6.813
6.660
6.658
6.644
6.642
4.894
4.883
4.175
4.167
4.005
3.999
3.991
3.987
3.978
3.619
1.280
1.271
1.261
1.254
1.245
1.235
1.230
1.218
1.212
1.208
1.189
0.856
0.842
0.828
10
9
8
7
6
5
4
3
2
1
0
ppm
1.01
1.01
0.98
1.00
0.92
0.94
0.97
3.00
11.95
3.18

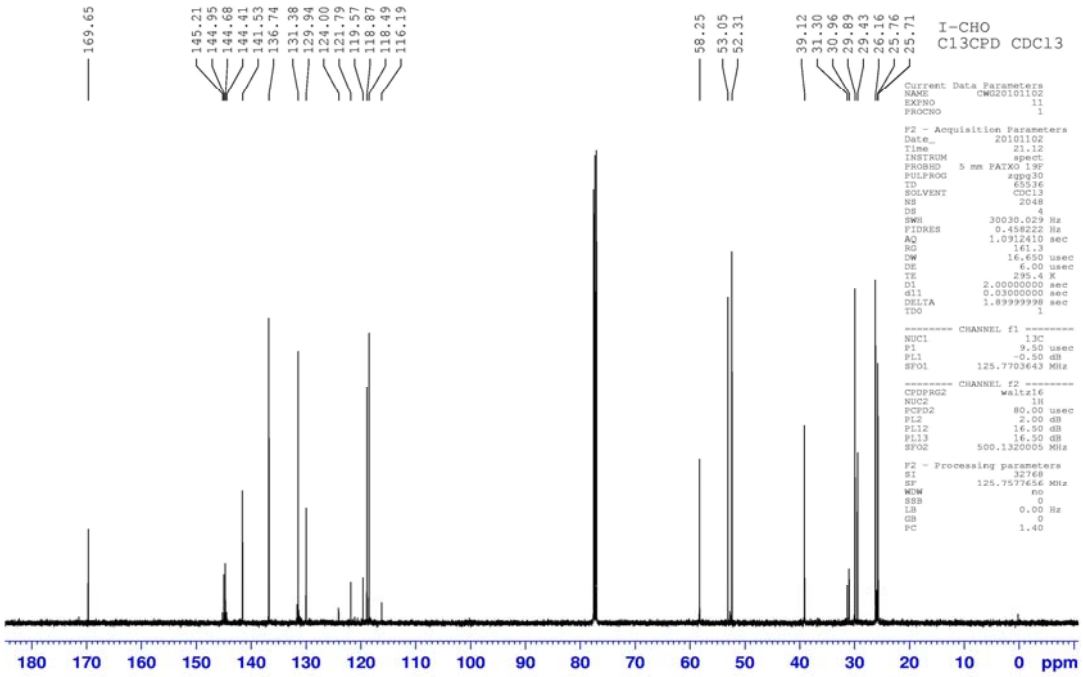

1-XIN
C13CPD CDCl3
168.91
144.93
144.66
144.39
144.12
140.97
136.32
131.01
129.54
123.81
121.60
119.39
118.68
118.41
117.45
54.86
53.00
52.80
32.67
31.63
29.04
25.99
22.57
14.03
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0 ppm

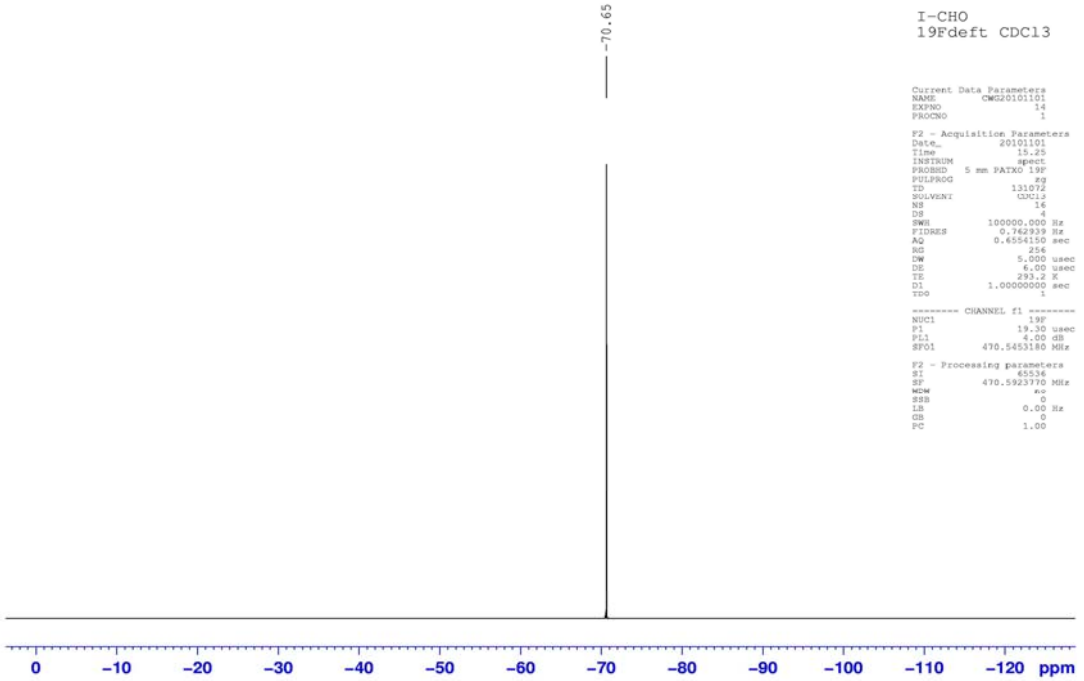

-71.24
1-XIN
19Fdeft CDCl3
Current Data Parameters
NAME CWG20100309
EXPNO 3
PROCNO 1
F2 - Acquisition Parameters
Date_ 20100309
Time 10.51
INSTRUM spect
PROBHD 5 mm PATXO 19F
PULPROG zg
TD 131072
SOLVENT CDCl3
NS 16
DS 4
SWH 100000.000 Hz
FIDRES 0.762939 Hz
AQ 0.6554150 sec
RG 203.2
DW 5.000 usec
DE 6.00 usec
TE 293.4 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 19F
P1 19.30 usec
PL1 4.00 dB
SFO1 470.5453180 MHz
F2 - Processing parameters
SI 65536
SF 470.5923770 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
PC 1.00
0 -10 -20 -30 -40 -50 -60 -70 -80 -90 -100 -110 -120 -130 -140 -150 ppm

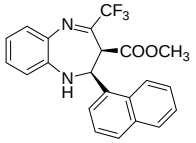

CF3
N
COOCH3
N
H

6j

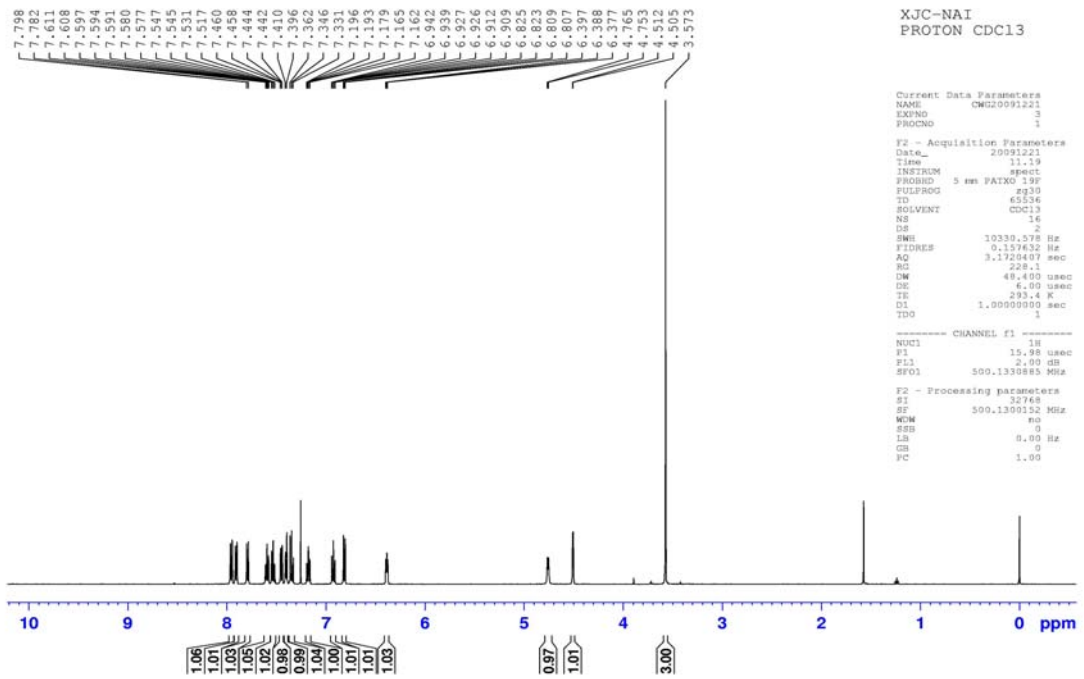

I-CHO
PROTON CDCl3
7.544
7.542
7.528
7.526
7.150
7.148
7.134
7.120
7.117
6.835
6.821
6.807
6.658
6.642
4.904
4.393
4.385
3.618
1.759
1.731
1.710
1.632
1.605
1.591
1.576
1.096
1.078
1.065
1.060
1.050
1.044
1.023
1.005
0.982
0.958
0.932
0.911
0.884
Current Data Parameters
NAME CWG20101101
EXPNO 13
PROCNO 1
F2 - Acquisition Parameters
Date_ 20101101
Time 15.22
INSTRUM spect
PROBHD 5 mm PATXO 19F
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 10330.578 Hz
FIDRES 0.157632 Hz
AQ 3.1720407 sec
RG 64
DW 48.400 usec
DE 6.00 usec
TE 293.5 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 15.98 usec
PL1 2.00 dB
SFO1 500.1330885 MHz
F2 - Processing parameters
SI 32768
SF 500.1300134 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
PC 1.00
10 9 8 7 6 5 4 3 2 1 0 ppm
0.92
0.94
0.93
0.89
0.69
0.90
3.00
3.08
2.14
2.81
2.97

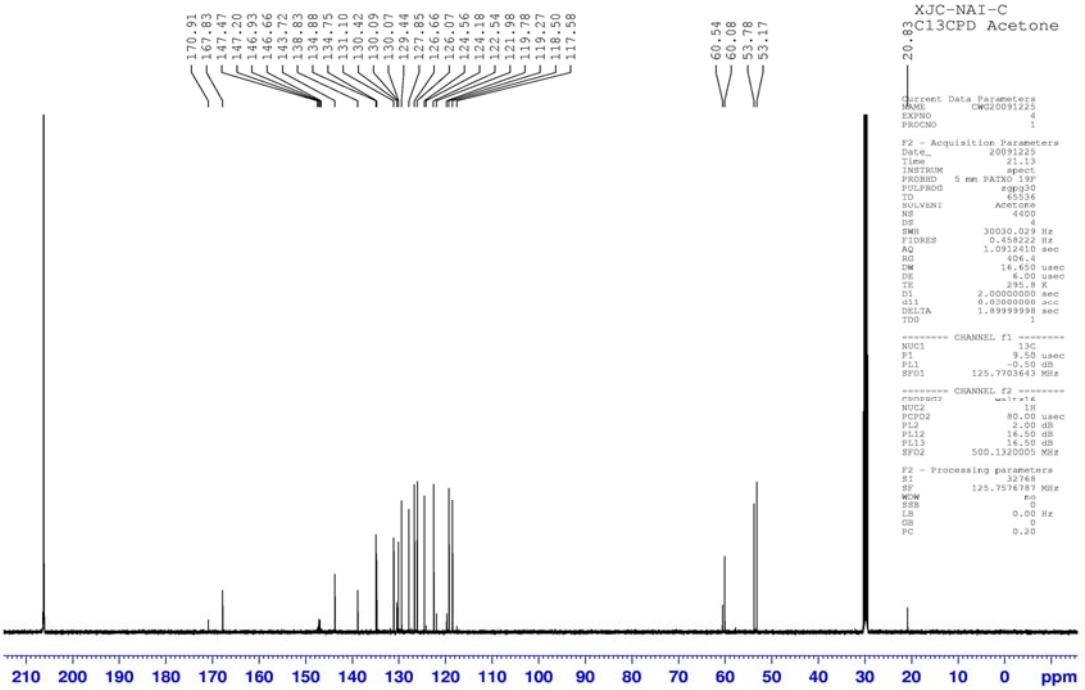

169.65
145.21
144.95
144.68
144.41
141.53
136.74
131.38
129.94
124.00
121.79
119.57
118.87
118.49
116.19
58.25
53.05
52.31
39.12
31.30
30.96
29.89
29.43
26.16
25.76
25.71
I-CHO
C13CPD CDC13
180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

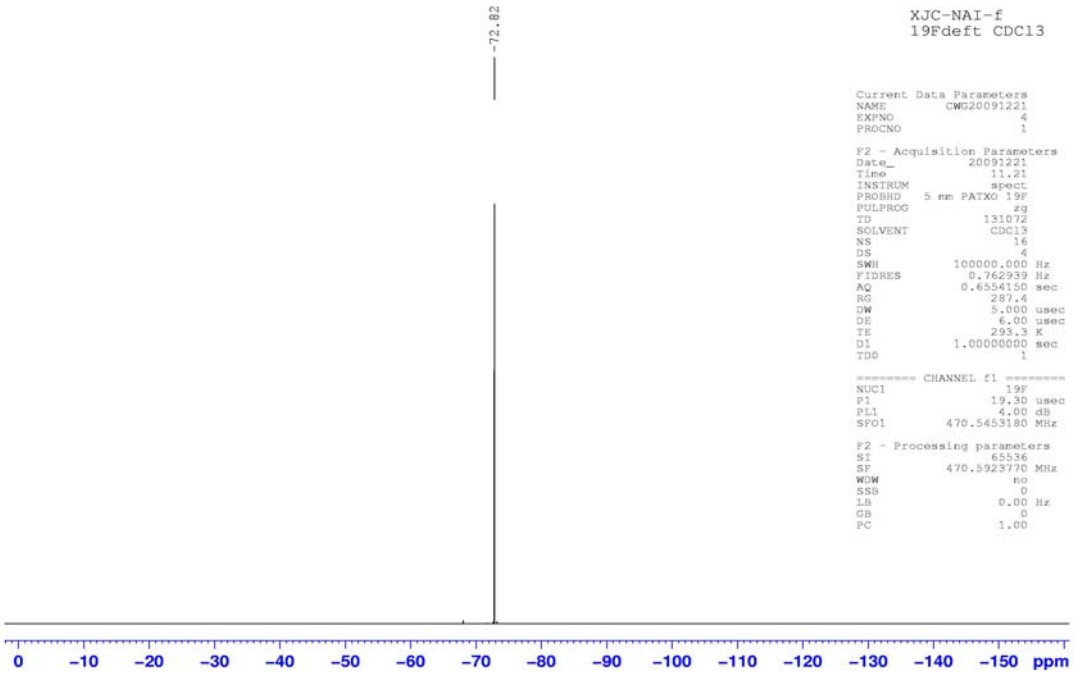

-70.65
I-CHO
19Fdeft CDC13
0 -10 -20 -30 -40 -50 -60 -70 -80 -90 -100 -110 -120 ppm

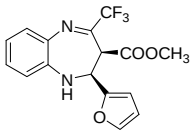

CF3
N
COOCH3
N
H

6k

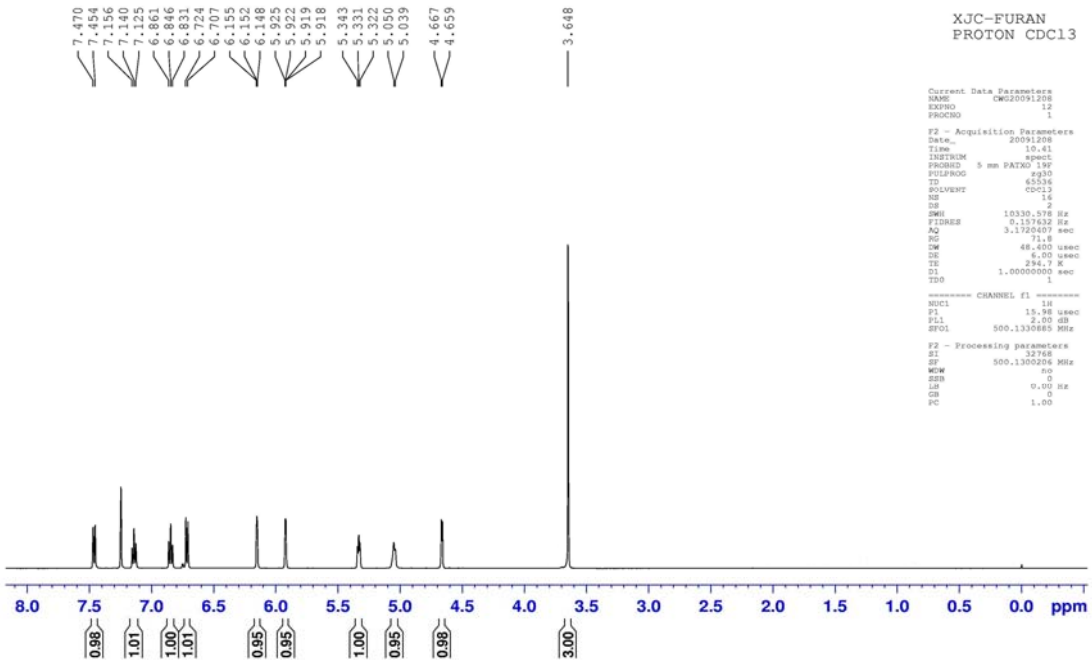

XJC-NAI
PROTON CDCl3
7.798
7.782
7.611
7.608
7.597
7.594
7.591
7.580
7.577
7.547
7.545
7.531
7.517
7.460
7.458
7.444
7.442
7.410
7.396
7.362
7.346
7.331
7.196
7.193
7.179
7.165
7.162
6.942
6.939
6.927
6.926
6.912
6.909
6.825
6.823
6.809
6.807
6.397
6.388
6.377
4.765
4.753
4.512
4.505
3.573
1.06
1.01
1.03
1.05
1.02
0.98
0.99
1.04
1.00
1.01
1.01
1.03
0.97
1.01
3.00
10
9
8
7
6
5
4
3
2
1
0
ppm

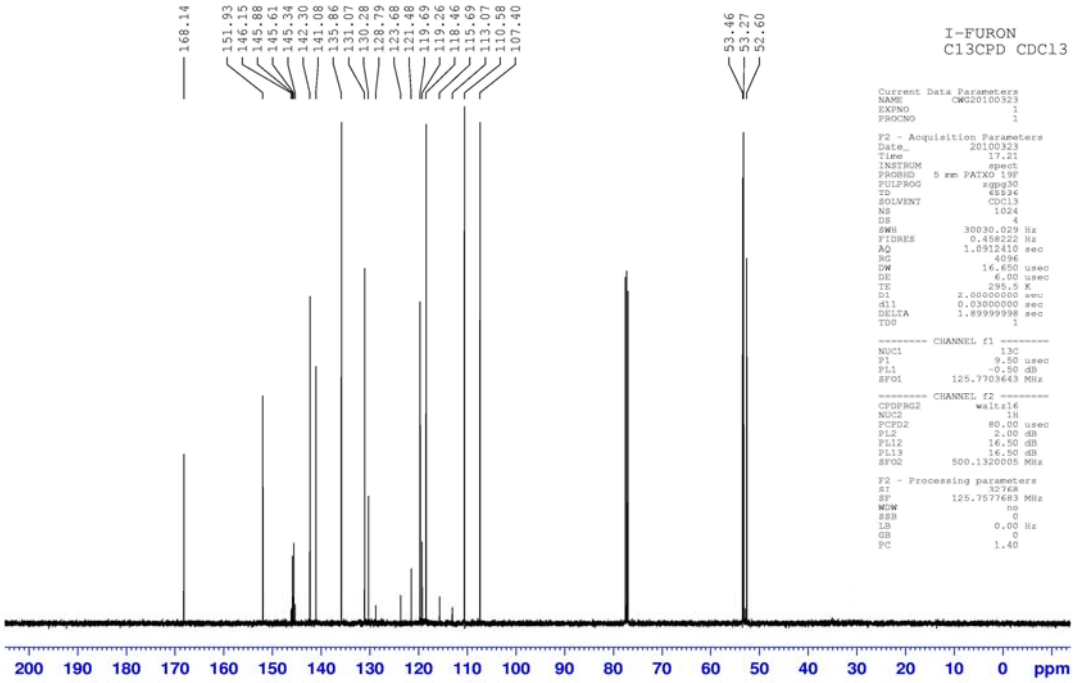

XJC-NAI-C
C13CPD Acetone
170.91
167.83
147.47
147.20
146.93
146.66
143.72
138.83
134.88
134.75
131.10
130.42
130.09
130.07
129.44
127.85
126.66
126.07
124.56
124.18
122.54
121.98
119.78
119.27
118.50
117.58
60.54
60.08
53.78
53.17
20.83
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
ppm

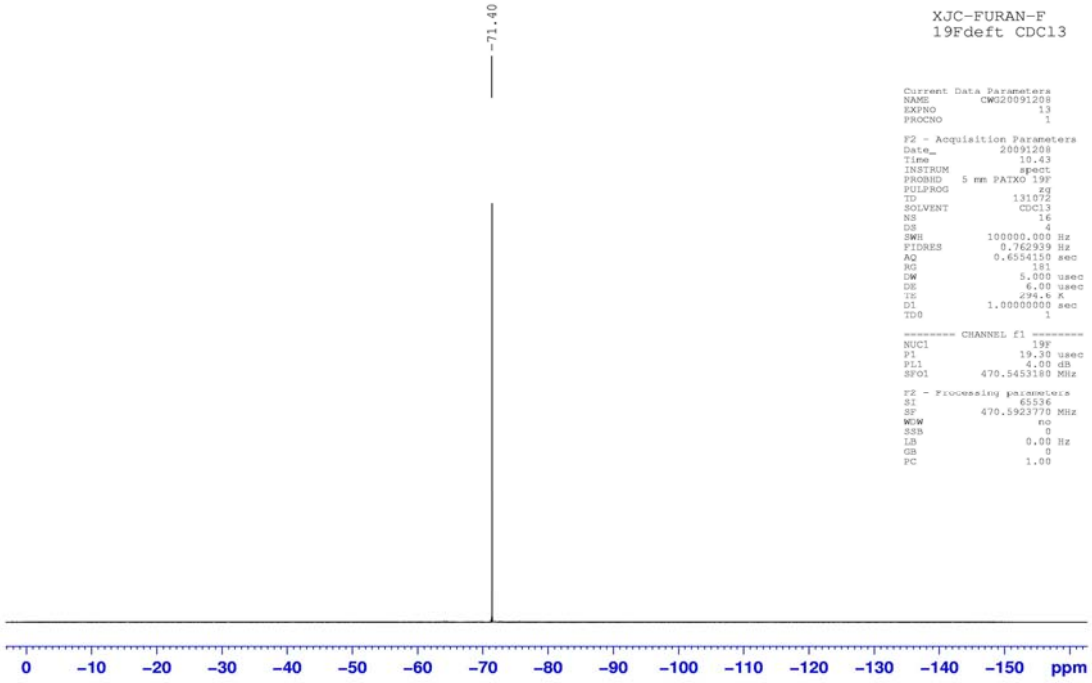

-72.82
XJC-NAI-f
19Fdeft CDC13
Current Data Parameters
NAME CWG20091221
EXPNO 4
PROCNO 1
F2 - Acquisition Parameters
Date_ 20091221
Time 11.21
INSTRUM spect
PROBHD 5 mm PATXO 19F
PULPROG zg
TD 131072
SOLVENT CDC13
NS 16
DS 4
SWH 100000.000 Hz
FIDRES 0.762939 Hz
AQ 0.6554150 sec
RG 287.4
DW 5.000 usec
DE 6.00 usec
TE 293.3 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 19F
P1 19.30 usec
PL1 4.00 dB
SFO1 470.5453180 MHz
F2 - Processing parameters
SI 65536
SF 470.5923770 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
PC 1.00
0 -10 -20 -30 -40 -50 -60 -70 -80 -90 -100 -110 -120 -130 -140 -150 ppm

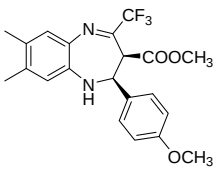

CF3
N
COOCH3
N
H
O

6l

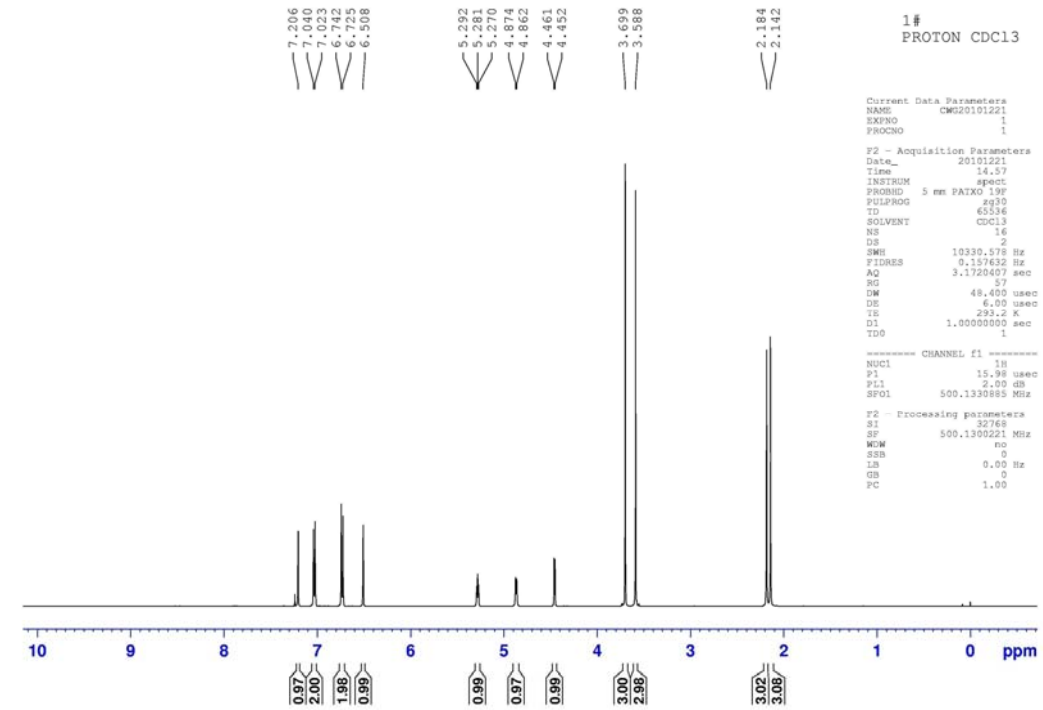

7.470
7.454
7.156
7.140
7.125
6.861
6.846
6.831
6.724
6.707
6.155
6.152
6.148
5.925
5.922
5.919
5.918
5.343
5.331
5.322
5.050
5.039
4.667
4.659
3.648
XJC-FURAN
PROTON CDC13
Current Data Parameters
NAME CWG20091208
EXPNO 12
PROCNO 1
F2 - Acquisition Parameters
Date_ 20091208
Time 10.41
INSTRUM spect
PROBHD 5 mm PATXO 19F
PULPROG zg30
TD 65536
SOLVENT CDC13
NS 16
DS 2
SWH 10330.578 Hz
FIDRES 0.157632 Hz
AQ 3.1720407 sec
RG 71.8
DW 48.400 usec
DE 6.00 usec
TE 294.7 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 15.98 usec
PL1 2.00 dB
SFO1 500.1330885 MHz
F2 - Processing parameters
SI 32768
SF 500.1300206 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
PC 1.00
8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 ppm
0.98 1.01 1.00 1.01 0.95 0.95 1.00 0.95 0.98 3.00

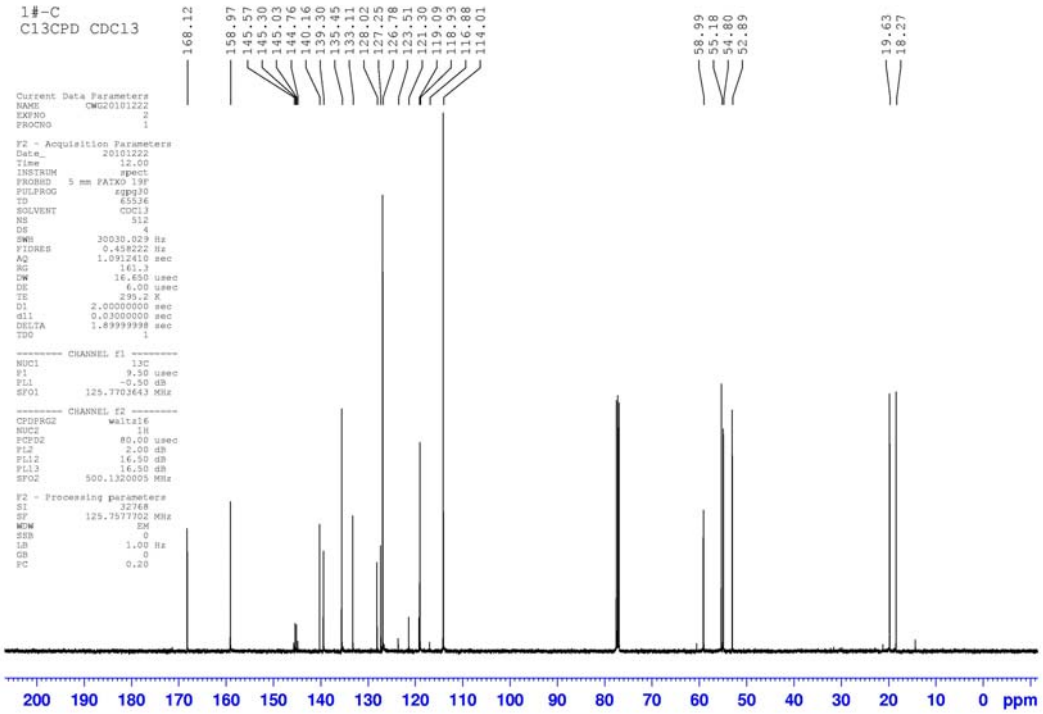

168.14
151.93
146.15
145.88
145.61
145.34
142.30
141.08
135.86
131.07
130.28
128.79
123.68
121.48
119.69
119.26
118.46
115.69
113.07
110.58
107.40
53.46
53.27
52.60
I-FURON
C13CPD CDC13
200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

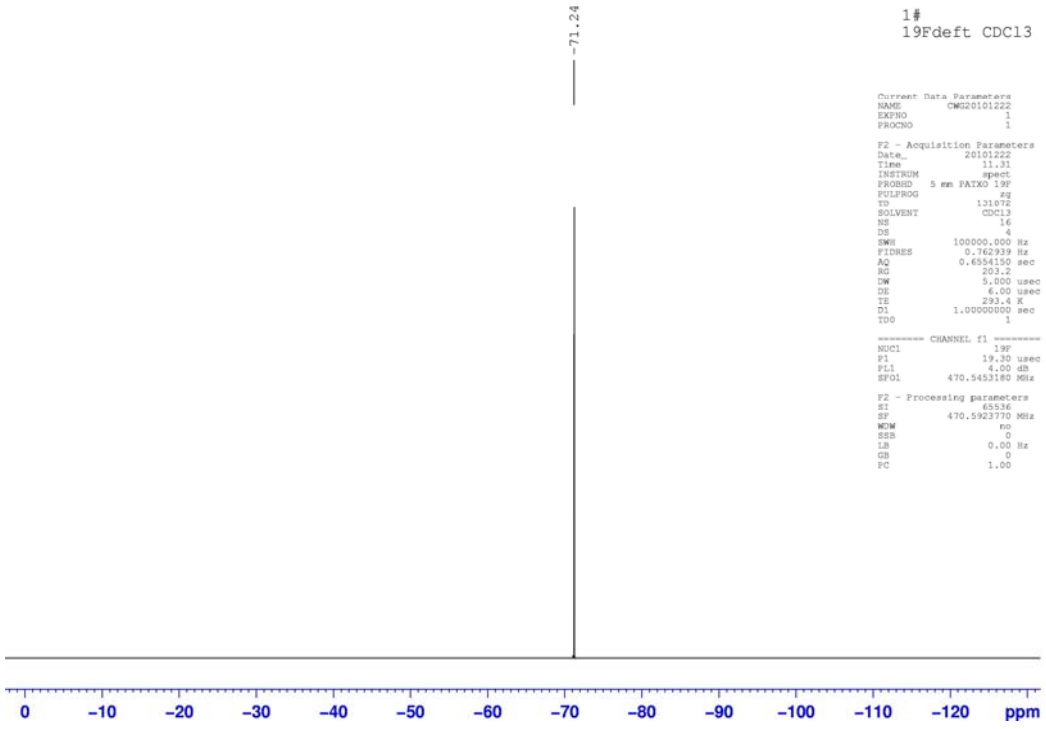

-71.40
XJC-FURAN-F
19Fdeft CDC13
0 -10 -20 -30 -40 -50 -60 -70 -80 -90 -100 -110 -120 -130 -140 -150 ppm

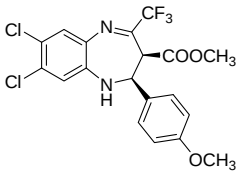

CF3
N
COOCH3
N
H
OCH3

6m

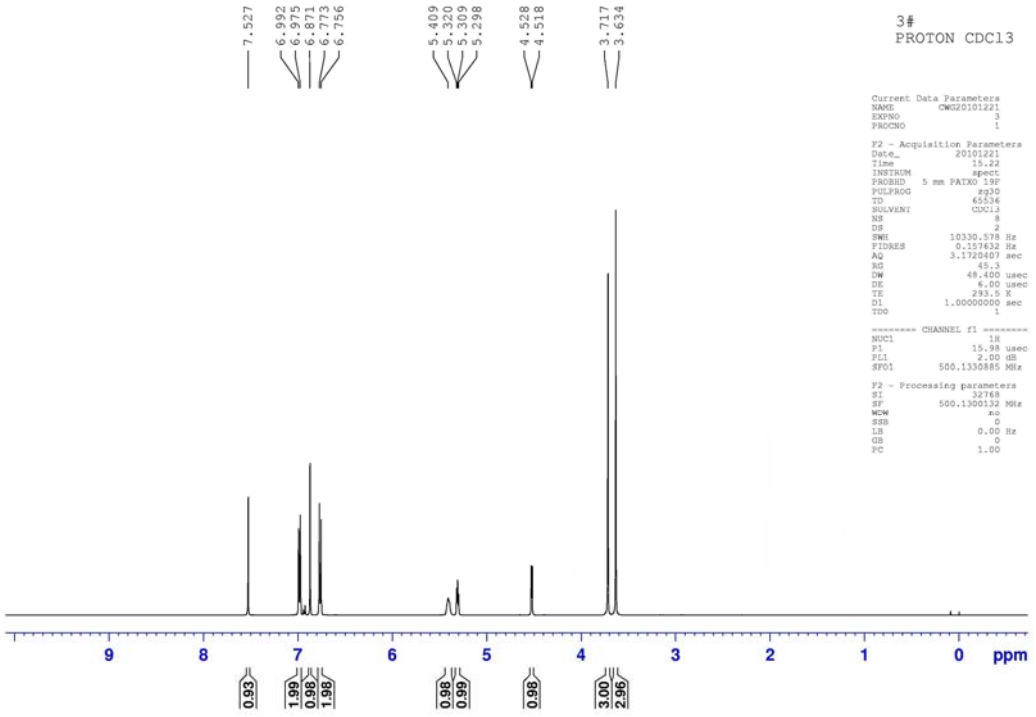

7.206
7.040
7.023
6.742
6.725
6.508
5.292
5.281
5.270
4.874
4.862
4.461
4.452
3.699
3.588
2.184
2.142
1#
PROTON CDC13
Current Data Parameters
NAME CWG20101221
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20101221
Time 14.57
INSTRUM spect
PROBHD 5 mm PATXO 19F
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 10330.578 Hz
FIDRES 0.157632 Hz
AQ 3.1720407 sec
RG 57
DW 48.400 usec
DE 6.00 usec
TE 293.2 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 15.98 usec
PL1 2.00 dB
SFO1 500.1330885 MHz
F2 - Processing parameters
SI 32768
SF 500.1300221 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
PC 1.00
10 9 8 7 6 5 4 3 2 1 0 ppm
0.97 2.00 1.98 0.99 0.99 0.97 0.99 3.00 2.98 3.02 3.06

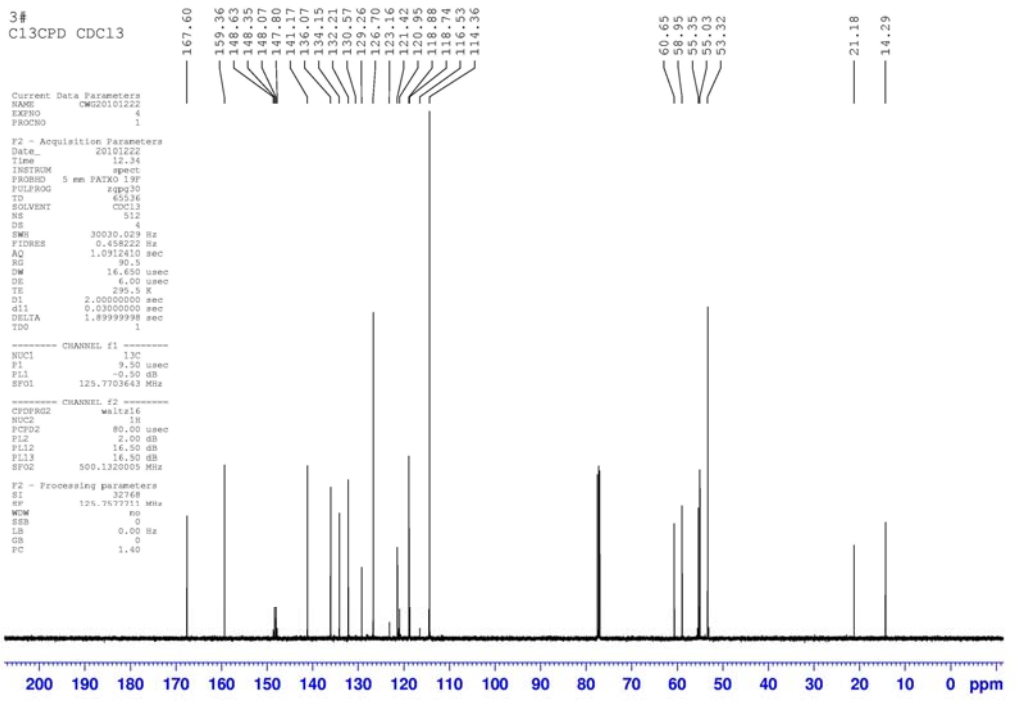

1#-C
C13CPD CDC13
168.12
158.97
145.57
145.30
145.03
144.76
140.16
139.30
135.45
133.11
128.02
127.25
126.78
123.51
121.30
119.09
118.93
116.88
114.01
58.99
55.18
54.80
52.89
19.63
18.27
Current Data Parameters
NAME CWG20101222
EXPNO 2
PROCNO 1
F2 - Acquisition Parameters
Date_ 20101222
Time 12.00
INSTRUM spect
PROBHD 5 mm PATXO 19F
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 512
DS 4
SWH 30030.029 Hz
FIDRES 0.458222 Hz
AQ 1.0912410 sec
RG 161.3
DW 16.650 usec
DE 6.00 usec
TE 295.2 K
D1 2.00000000 sec
d11 0.03000000 sec
DELTA 1.89999998 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 9.50 usec
PL1 -0.50 dB
SFO1 125.7703643 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 2.00 dB
PL12 16.50 dB
PL13 16.50 dB
SFO2 500.1320005 MHz
F2 - Processing parameters
SI 32768
SF 125.7577702 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 0.20
200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

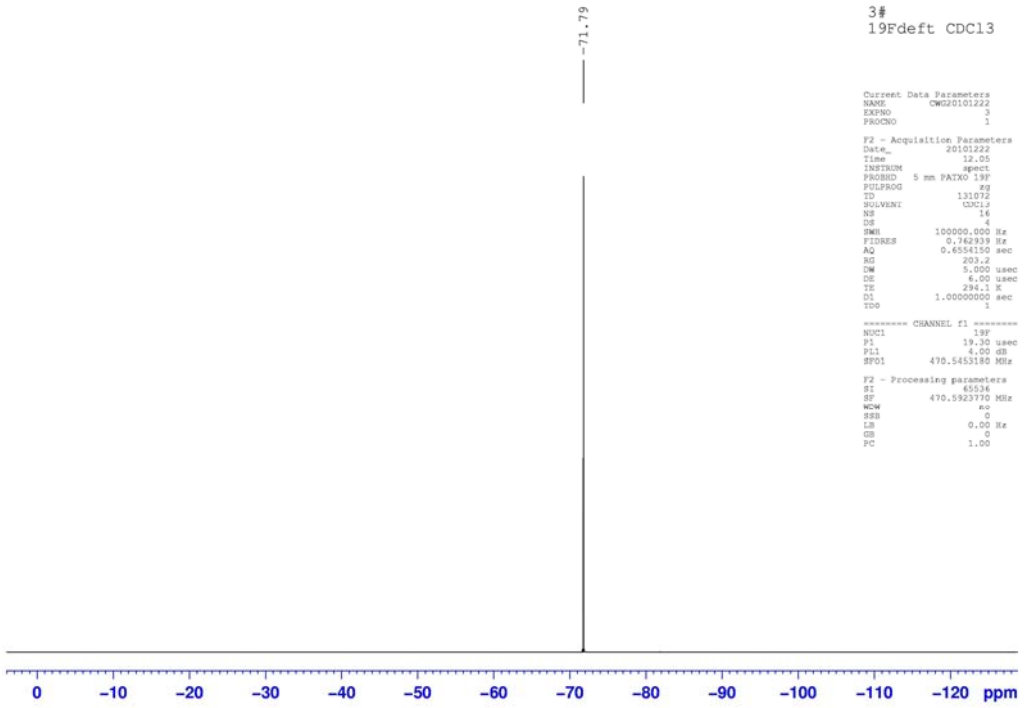

-71.24
1#
19Fdeft CDCl3
Current Data Parameters
NAME CWG20101222
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20101222
Time 11.31
INSTRUM spect
PROBHD 5 mm PATXO 19F
PULPROG zg
TD 131072
SOLVENT CDCl3
NS 16
DS 4
SWH 100000.000 Hz
FIDRES 0.762939 Hz
AQ 0.6554150 sec
RG 203.2
DW 5.000 usec
DE 6.00 usec
TE 293.4 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 19F
P1 19.30 usec
PL1 4.00 dB
SFO1 470.5453180 MHz
F2 - Processing parameters
SI 65536
SF 470.5923770 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
PC 1.00
0
-10
-20
-30
-40
-50
-60
-70
-80
-90
-100
-110
-120
ppm

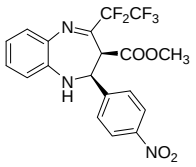

Cl
Cl
N
CF3
COOCH3
N
H
OCH3

6n

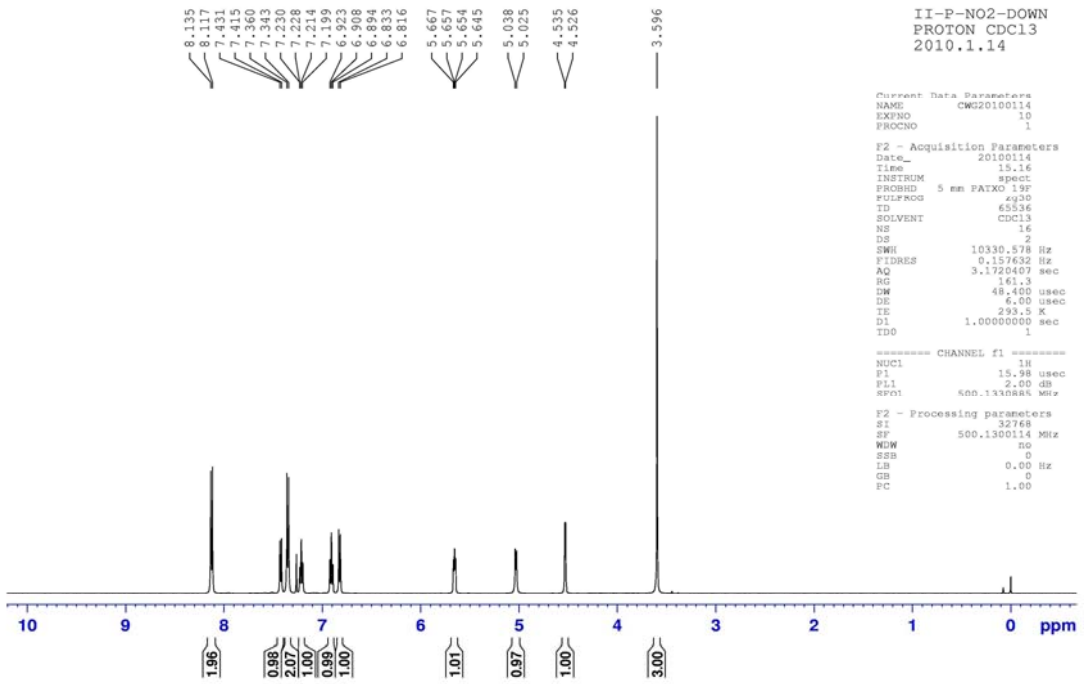

7.527
6.992
6.975
6.871
6.773
6.756
5.409
5.320
5.309
5.298
4.528
4.518
3.717
3.634
3#
PROTON CDCl3
Current Data Parameters
NAME CWG20101221
EXPNO 3
PROCNO 1
F2 - Acquisition Parameters
Date_ 20101221
Time 15.22
INSTRUM spect
PROBHD 5 mm PATXO 19F
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 8
DS 2
SWH 10330.578 Hz
FIDRES 0.157632 Hz
AQ 3.1720407 sec
RG 45.3
DW 48.400 usec
DE 6.00 usec
TE 293.5 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 15.98 usec
PL1 2.00 dB
SFO1 500.1330885 MHz
F2 - Processing parameters
SI 32768
SF 500.1300132 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
PC 1.00
9
8
7
6
5
4
3
2
1
0
ppm
0.93
1.99
0.98
1.98
0.98
0.99
0.98
3.00
2.96

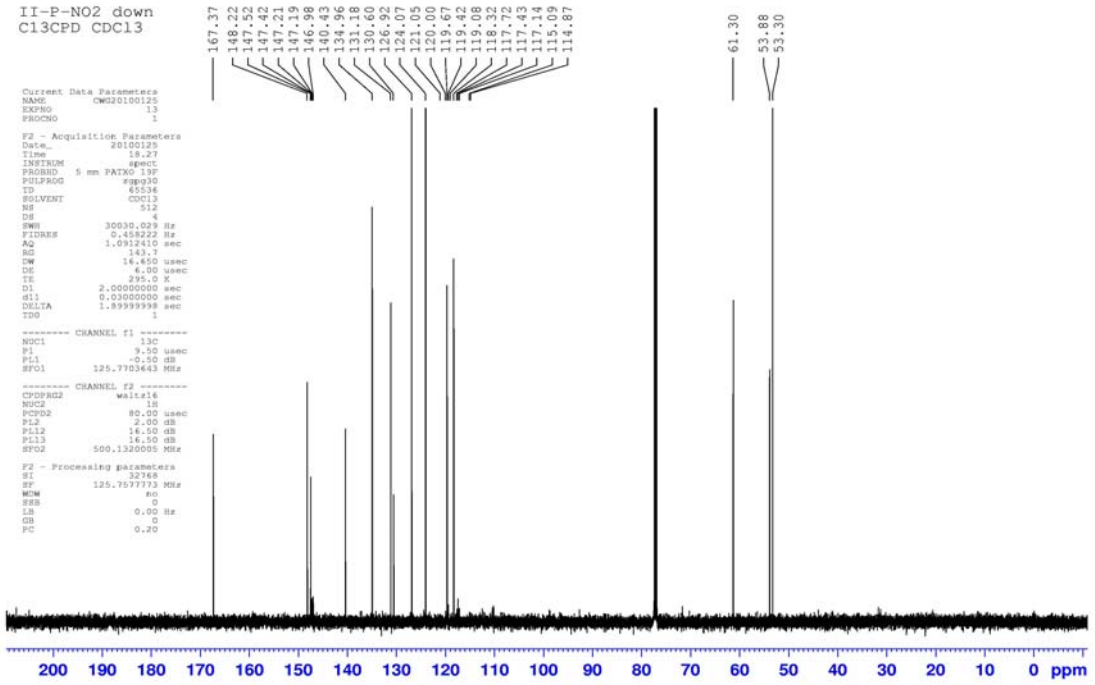

3#
C13CPD CDC13
167.60
159.36
148.63
148.35
148.07
147.80
141.17
136.07
134.15
132.21
130.57
129.26
126.70
123.16
121.42
120.95
118.88
118.74
116.53
114.36
60.65
58.95
55.35
55.03
53.32
21.18
14.29
200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

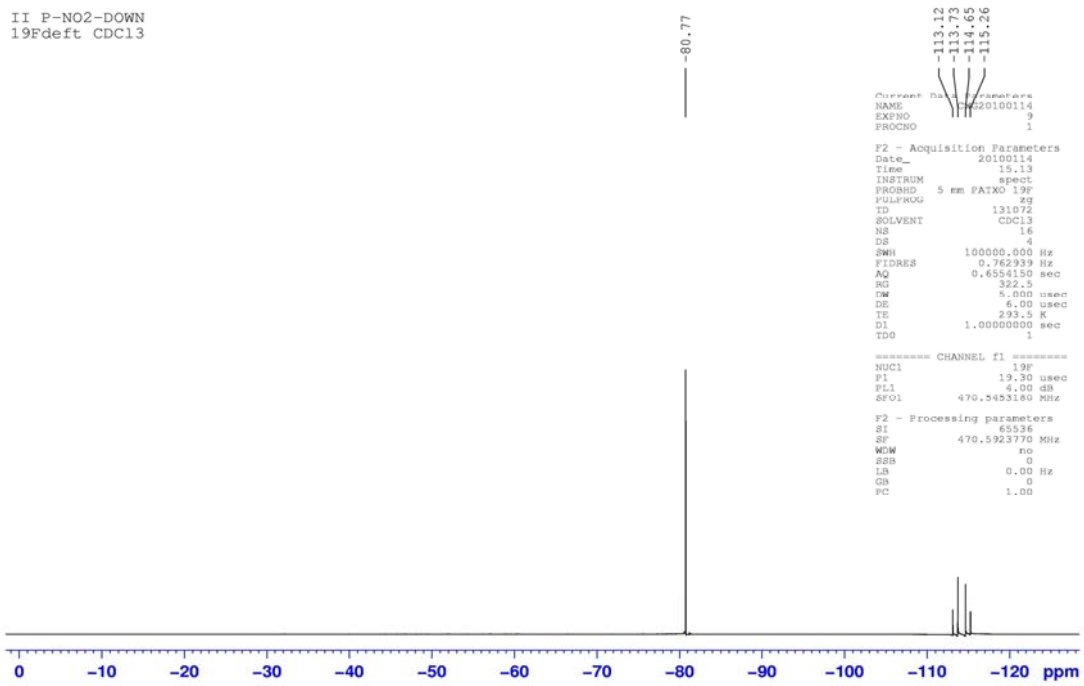

3#
19Fdeft CDC13
-71.79
0 -10 -20 -30 -40 -50 -60 -70 -80 -90 -100 -110 -120 ppm

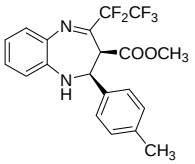

CF2CF3
N
COOCH3
N
H
NO2

6o

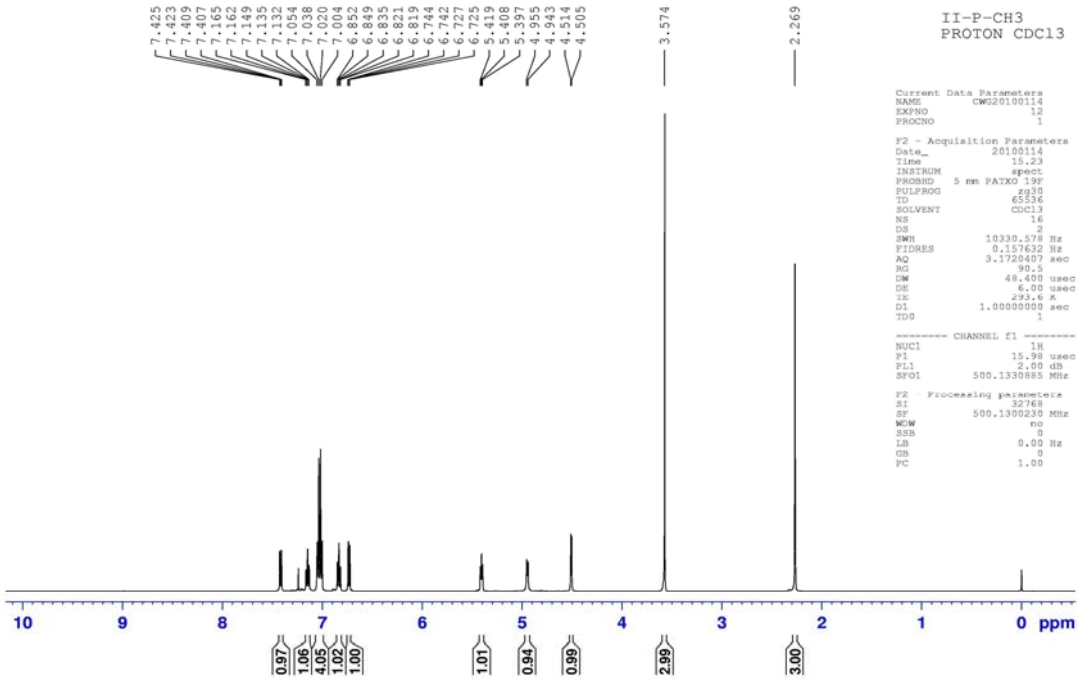

8.135
8.117
7.431
7.415
7.360
7.343
7.230
7.228
7.214
7.199
6.923
6.908
6.894
6.833
6.816
5.667
5.657
5.654
5.645
5.038
5.025
4.535
4.526
3.596
II-P-NO2-DOWN
PROTON CDC13
2010.1.14
10
9
8
7
6
5
4
3
2
1
0
ppm
1.96
0.98
2.07
1.00
0.99
1.00
1.01
0.97
1.00
3.00

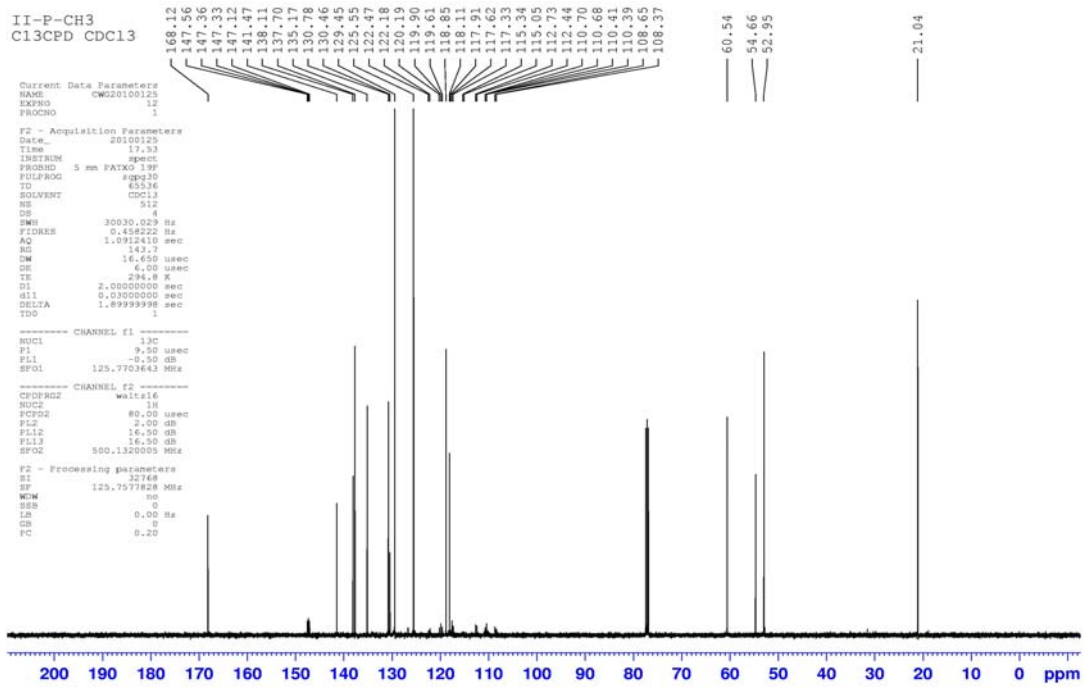

II-P-NO2 down
C13CPD CDC13
167.37
148.22
147.52
147.42
147.21
147.19
146.98
140.43
134.96
131.18
130.60
126.92
124.07
121.05
120.00
119.67
119.42
119.08
118.32
117.72
117.43
117.14
115.09
114.87
61.30
53.88
53.30
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
ppm

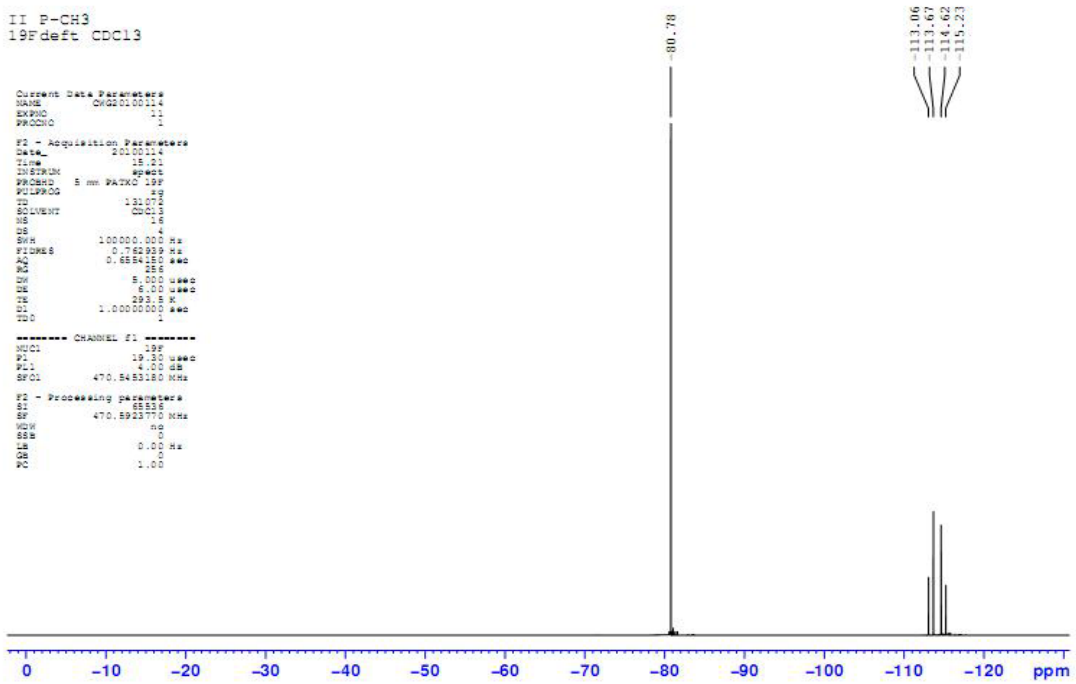

II P-NO2-DOWN
19Fdeft CDC13
-80.77
-113.12
-113.73
-114.65
-115.26
0
-10
-20
-30
-40
-50
-60
-70
-80
-90
-100
-110
-120
ppm

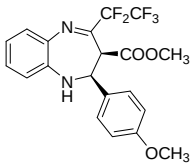

CF2CF3
COOCH3
N
H
CH3

6p

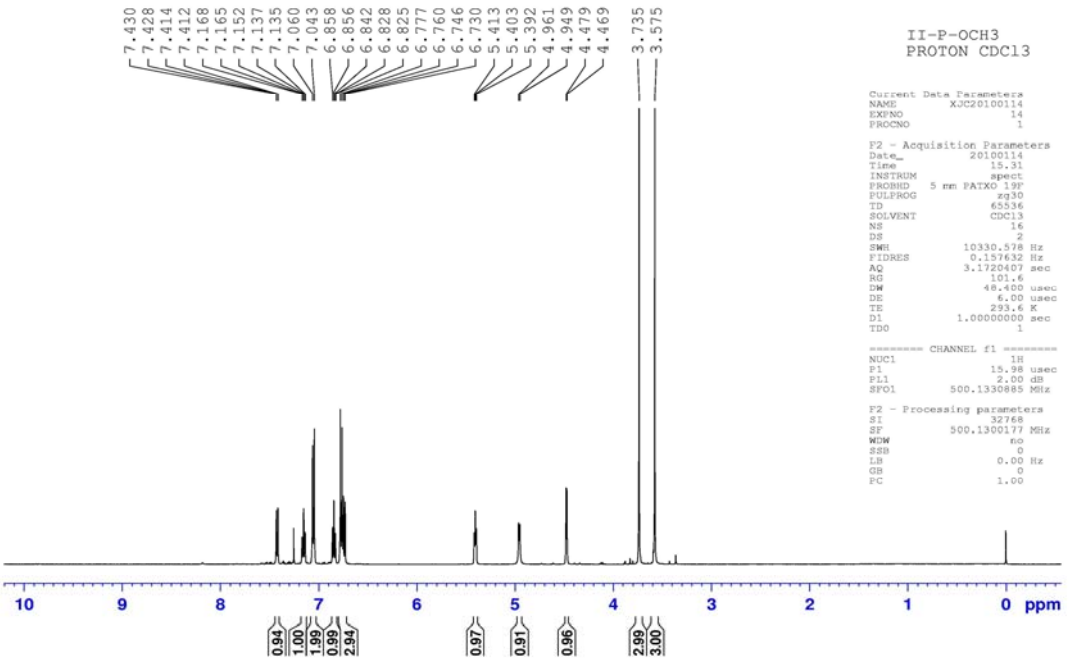

II-P-CH3
PROTON CDC13
7.425
7.423
7.409
7.407
7.165
7.162
7.149
7.135
7.132
7.054
7.038
7.020
7.004
6.852
6.849
6.835
6.821
6.819
6.744
6.742
6.727
6.725
5.419
5.408
5.397
4.955
4.943
4.514
4.505
3.574
2.269
10
9
8
7
6
5
4
3
2
1
0 ppm
0.97
1.06
4.05
1.02
1.00
1.01
0.94
0.99
2.99
3.00

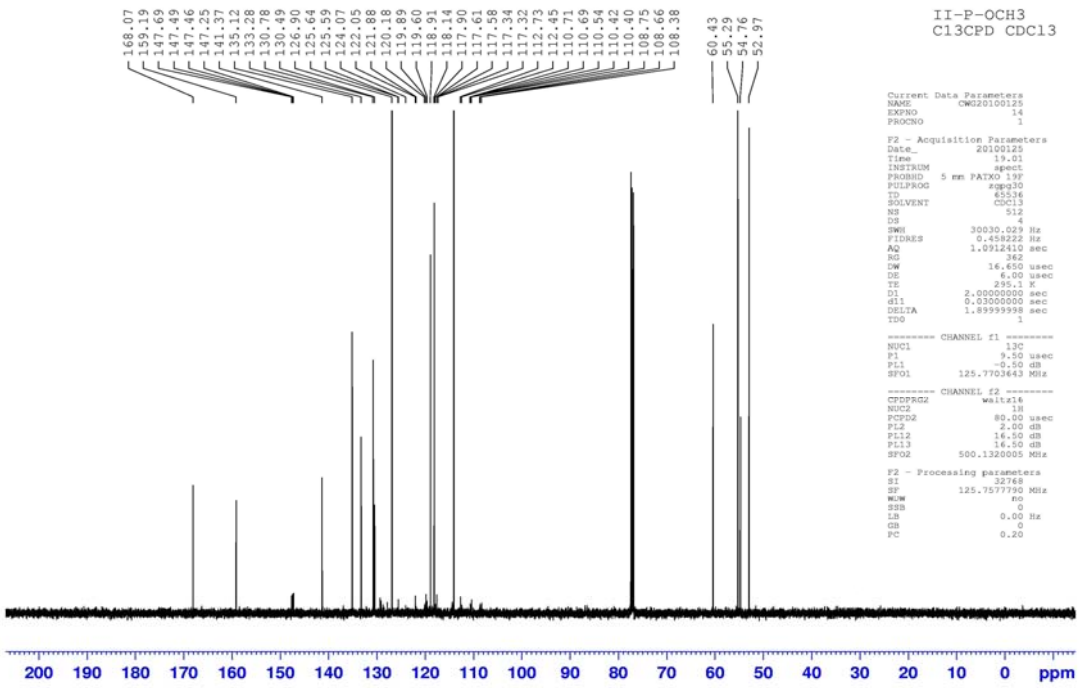

II-P-CH3
C13CPD CDC13
168.12
147.56
147.36
147.33
147.12
141.47
138.11
137.70
135.17
130.78
130.46
129.45
125.55
122.47
122.18
120.19
119.90
119.61
118.85
118.11
117.91
117.62
117.33
115.34
115.05
112.73
112.44
110.70
110.68
110.41
110.39
108.65
108.37
60.54
54.66
52.95
21.04
200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

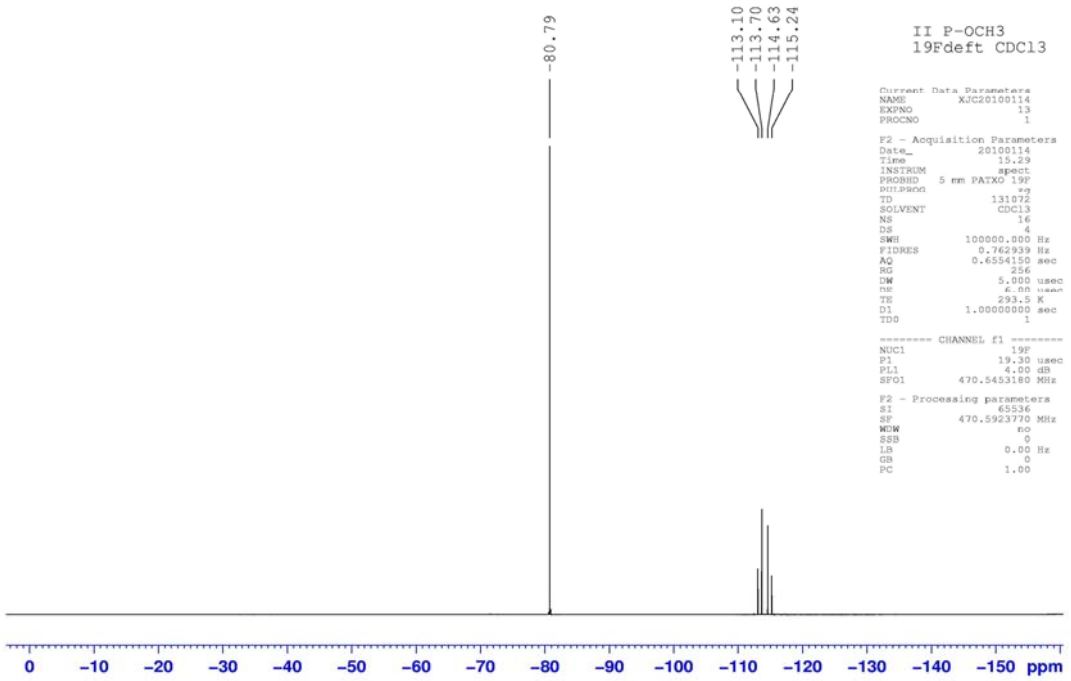

II P-CH3
19Fdeft CDC13
-80.78
-113.06
-113.67
-114.62
-115.23
0 -10 -20 -30 -40 -50 -60 -70 -80 -90 -100 -110 -120 ppm

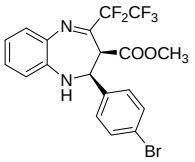

CF2CF3
N
COOCH3
N
H
OCH3

6q

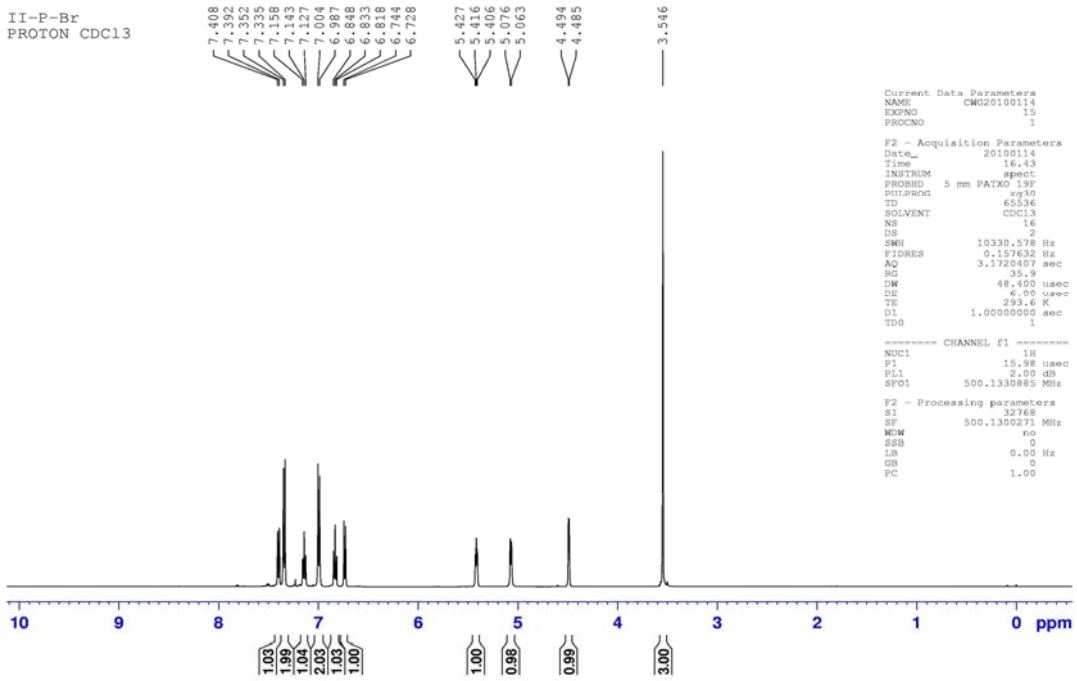

7.430
7.428
7.414
7.412
7.168
7.165
7.152
7.137
7.135
7.060
7.043
6.858
6.856
6.842
6.828
6.825
6.777
6.760
6.746
6.730
5.413
5.403
5.392
4.961
4.949
4.479
4.469
3.735
3.575
II-P-OCH3
PROTON CDCl3
Current Data Parameters
NAME XJC20100114
EXPNO 14
PROCNO 1
F2 - Acquisition Parameters
Date_ 20100114
Time 15.31
INSTRUM spect
PROBHD 5 mm PATXO 19F
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 10330.578 Hz
FIDRES 0.157632 Hz
AQ 3.1720407 sec
RG 101.6
DW 48.400 usec
DE 6.00 usec
TE 293.6 K
D1 1.00000000 sec
TD0 1
CHANNEL f1
NUC1 1H
P1 15.98 usec
PL1 2.00 dB
SFO1 500.1330885 MHz
F2 - Processing parameters
SI 32768
SF 500.1300177 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
PC 1.00
10 9 8 7 6 5 4 3 2 1 0 ppm
0.94 1.00 1.99 0.99 2.94 0.97 0.91 0.96 2.99 3.00

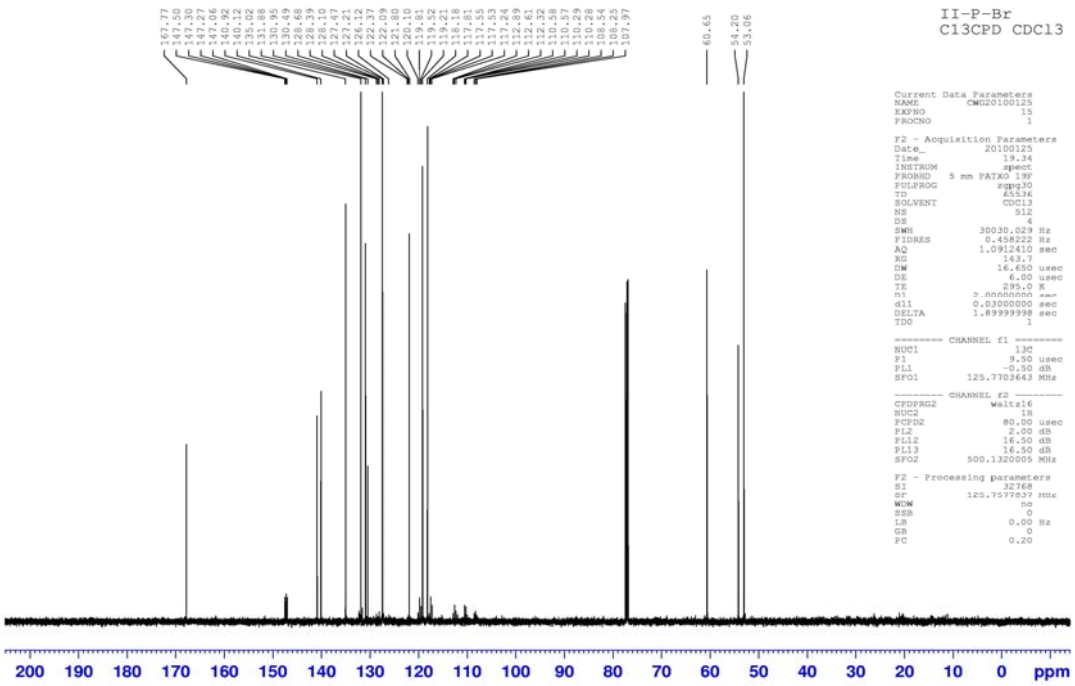

168.07
159.19
147.69
147.49
147.46
147.25
141.37
135.12
133.28
130.78
130.49
126.90
125.64
125.59
124.07
122.05
121.88
120.18
119.89
119.60
118.91
118.14
117.90
117.61
117.58
117.34
117.32
112.73
112.45
110.71
110.69
110.54
110.42
110.40
108.75
108.66
108.38
60.43
55.29
54.76
52.97
II-P-OCH3
C13CPD CDCl3
Current Data Parameters
NAME CWG20100125
EXPNO 14
PROCNO 1
F2 - Acquisition Parameters
Date_ 20100125
Time 19.01
INSTRUM spect
PROBHD 5 mm PATXO 19F
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 512
DS 4
SWH 30030.029 Hz
FIDRES 0.458222 Hz
AQ 1.0912410 sec
RG 362
DW 16.650 usec
DE 6.00 usec
TE 295.1 K
D1 2.00000000 sec
d11 0.03000000 sec
DELTA 1.89999998 sec
TD0 1
CHANNEL f1
NUC1 13C
P1 9.50 usec
PL1 -0.50 dB
SFO1 125.7703643 MHz
CHANNEL f2
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 2.00 dB
PL12 16.50 dB
PL13 16.50 dB
SFO2 500.1320005 MHz
F2 - Processing parameters
SI 32768
SF 125.7577790 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
PC 0.20
200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

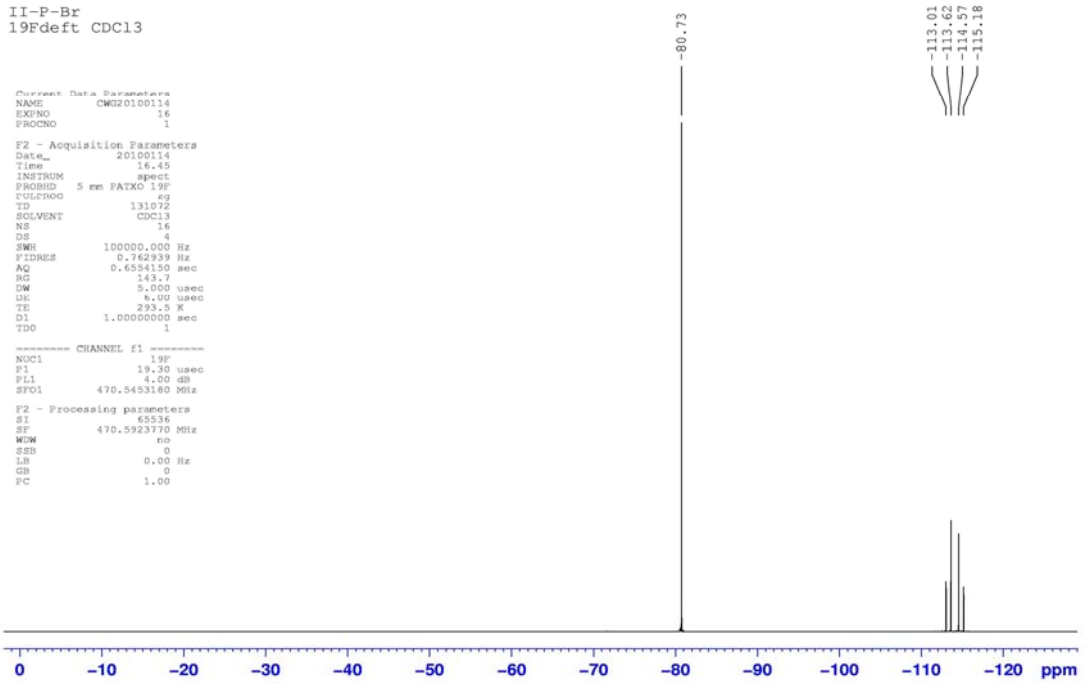

II P-OCH3
19Fdeft CDCl3
-80.79
-113.10
-113.70
-114.63
-115.24
0 -10 -20 -30 -40 -50 -60 -70 -80 -90 -100 -110 -120 -130 -140 -150 ppm

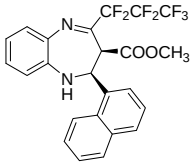

CF2CF3
COOCH3
N
H
Br

6r

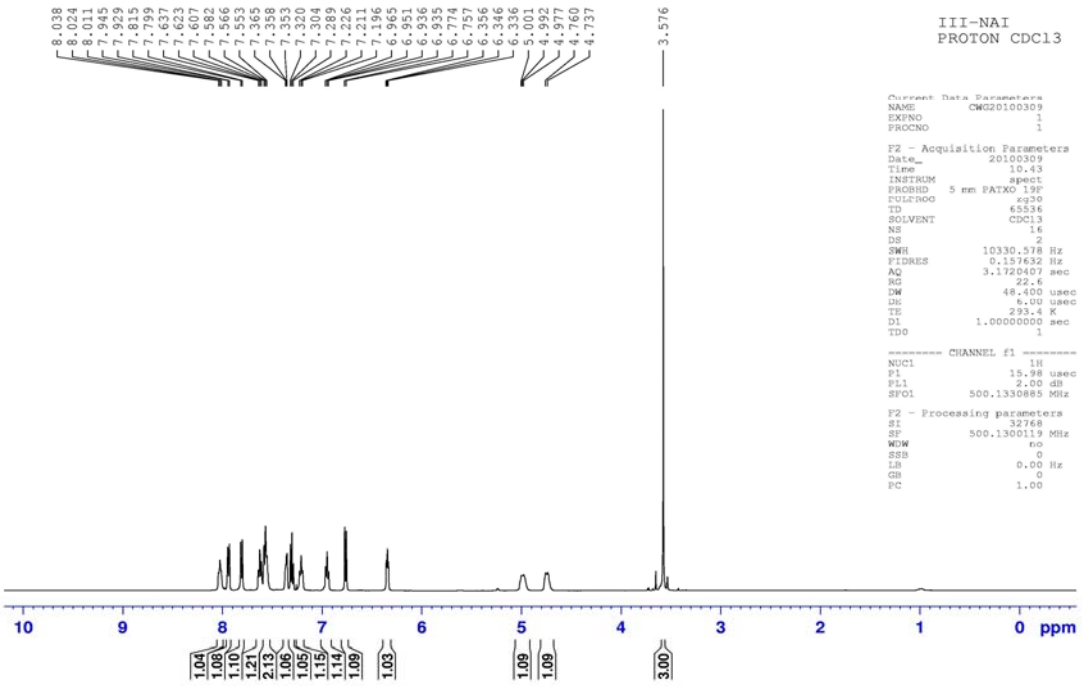

II-P-Br
PROTON CDCl3
7.408
7.392
7.352
7.335
7.158
7.143
7.127
7.004
6.987
6.848
6.833
6.818
6.744
6.728
5.427
5.416
5.406
5.076
5.063
4.494
4.485
3.546
10 9 8 7 6 5 4 3 2 1 0 ppm
1.03
1.99
1.04
2.03
1.03
1.00
1.00
0.98
0.99
3.00

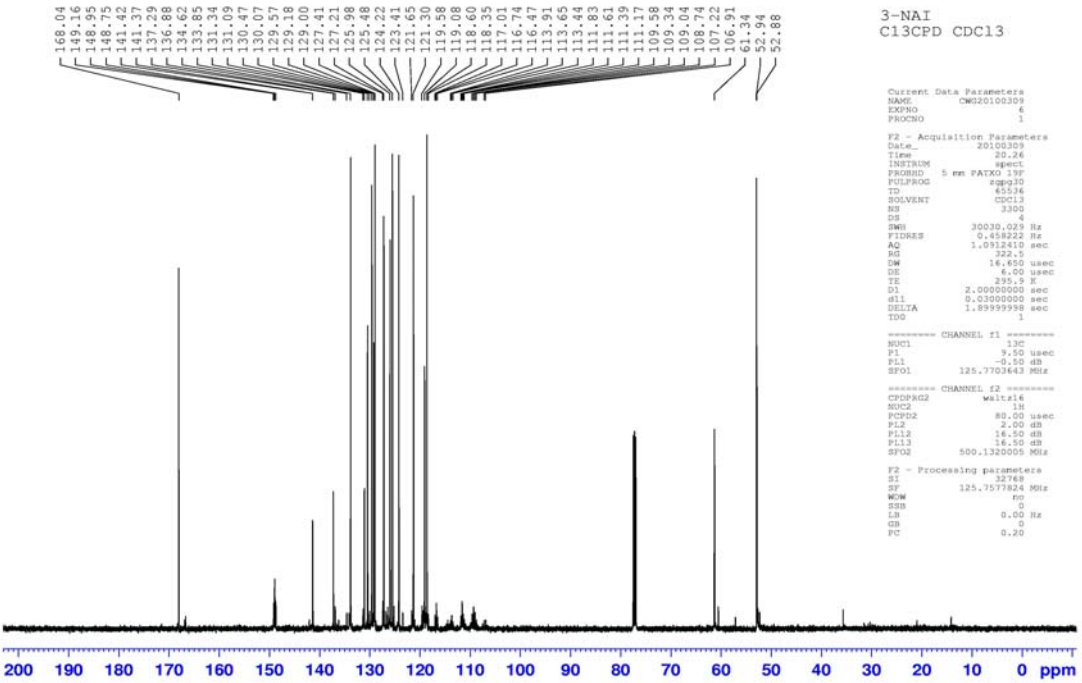

II-P-Br
C13CPD CDC13
167.77
167.50
147.30
147.27
147.06
140.92
140.12
135.02
131.88
130.95
130.49
128.68
128.39
128.10
127.47
127.21
125.12
122.37
122.09
121.80
120.10
119.81
119.52
119.21
118.18
117.81
117.55
117.53
117.24
112.89
112.61
112.32
110.58
110.57
110.29
110.28
108.54
108.25
107.97
60.65
54.20
53.06
200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

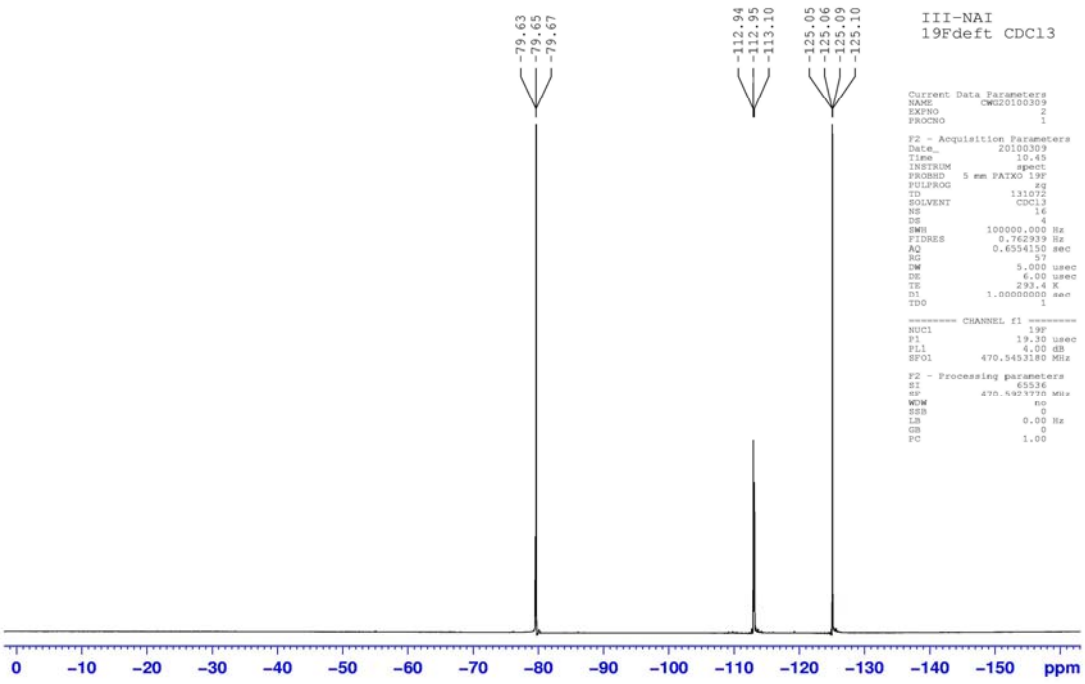

II-P-Br
19Fdeft CDC13
-80.73
-113.01
-113.62
-114.57
-115.18
0 -10 -20 -30 -40 -50 -60 -70 -80 -90 -100 -110 -120 ppm

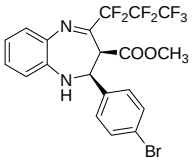

CF2CF2CF3
COOCH3
N
H

6s

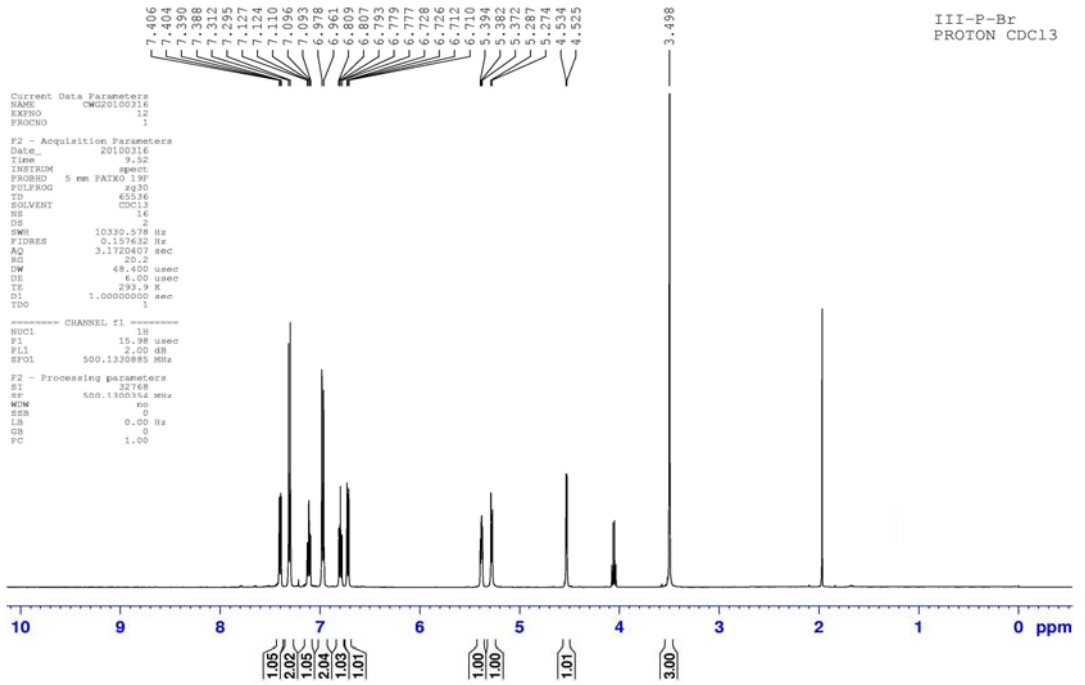

8.038
8.024
8.011
7.945
7.929
7.815
7.799
7.637
7.623
7.607
7.582
7.566
7.553
7.365
7.358
7.353
7.320
7.304
7.289
7.226
7.211
7.196
6.965
6.951
6.936
6.935
6.774
6.757
6.356
6.346
6.336
5.001
4.992
4.977
4.760
4.737
3.576
III-NAI
PROTON CDC13
Current Data Parameters
NAME CWG20100309
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20100309
Time 10.43
INSTRUM spect
PROBHD 5 mm PATXO 19F
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 10330.578 Hz
FIDRES 0.157632 Hz
AQ 3.1720407 sec
RG 22.6
DW 48.400 usec
DE 6.00 usec
TE 293.4 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 15.98 usec
PL1 2.00 dB
SFO1 500.1330885 MHz
F2 - Processing parameters
SI 32768
SF 500.1300119 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
PC 1.00
10 9 8 7 6 5 4 3 2 1 0 ppm
1.04 1.08 1.10 1.21 2.13 1.06 1.05 1.15 1.14 1.09 1.03 1.09 1.09 3.00

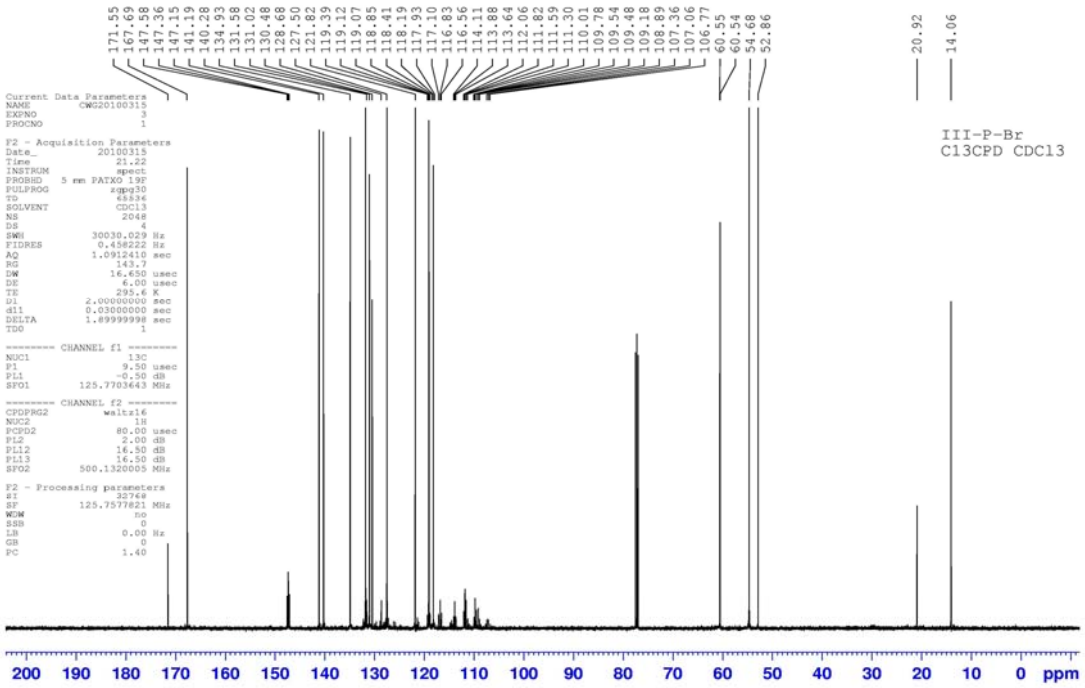

168.04
149.16
148.95
148.75
141.42
141.37
137.29
136.88
134.62
133.85
131.34
131.09
130.47
130.07
129.57
129.18
129.00
127.41
127.21
125.98
125.48
124.22
123.41
121.65
121.30
119.58
119.08
118.60
118.35
117.01
116.74
116.47
113.91
113.65
113.44
111.83
111.61
111.39
111.17
109.58
109.34
109.04
108.74
107.22
106.91
61.34
52.94
52.88
3-NAI
C13CPD CDC13
Current Data Parameters
NAME CWG20100309
EXPNO 6
PROCNO 1
F2 - Acquisition Parameters
Date_ 20100309
Time 20.26
INSTRUM spect
PROBHD 5 mm PATXO 19F
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 3300
DS 4
SWH 30030.029 Hz
FIDRES 0.458222 Hz
AQ 1.0912410 sec
RG 322.5
DW 16.650 usec
DE 6.00 usec
TE 295.9 K
D1 2.00000000 sec
d11 0.03000000 sec
DELTA 1.89999998 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 9.50 usec
PL1 -0.50 dB
SFO1 125.7703643 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 2.00 dB
PL12 16.50 dB
PL13 16.50 dB
SFO2 500.1320005 MHz
F2 - Processing parameters
SI 32768
SF 125.7577824 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
PC 0.20
200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

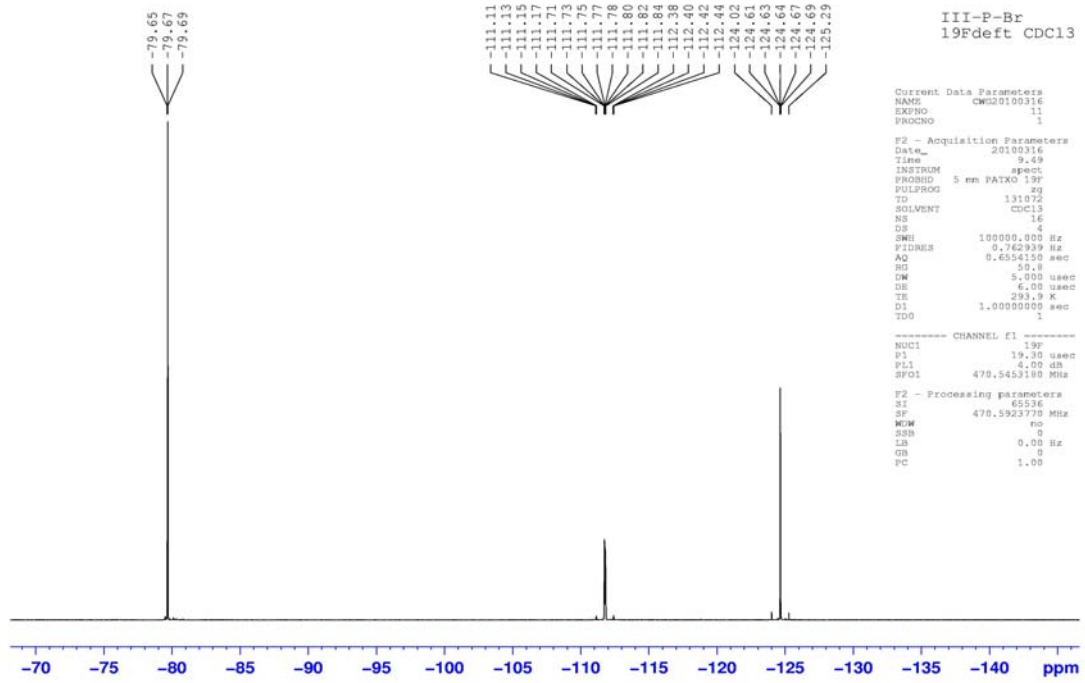

III-NAI
19Fdeft CDCl3
-79.63
-79.65
-79.67
-112.94
-112.95
-113.10
-125.05
-125.06
-125.09
-125.10
0
-10
-20
-30
-40
-50
-60
-70
-80
-90
-100
-110
-120
-130
-140
-150
ppm

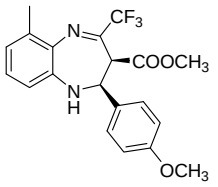

CF2CF2CF3
COOCH3
N
H
Br

6t

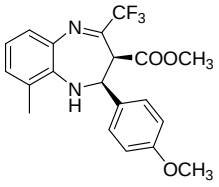

III-P-Br
PROTON CDCl3
7.406
7.404
7.390
7.388
7.312
7.295
7.127
7.124
7.110
7.096
7.093
6.978
6.961
6.809
6.807
6.793
6.779
6.777
6.728
6.726
6.712
6.710
5.394
5.392
5.372
5.287
5.274
4.534
4.525
3.498
10
9
8
7
6
5
4
3
2
1
0 ppm
1.05
2.02
1.05
2.04
1.03
1.01
1.00
1.00
1.01
3.00


III-P-Br
C13CPD CDC13
200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

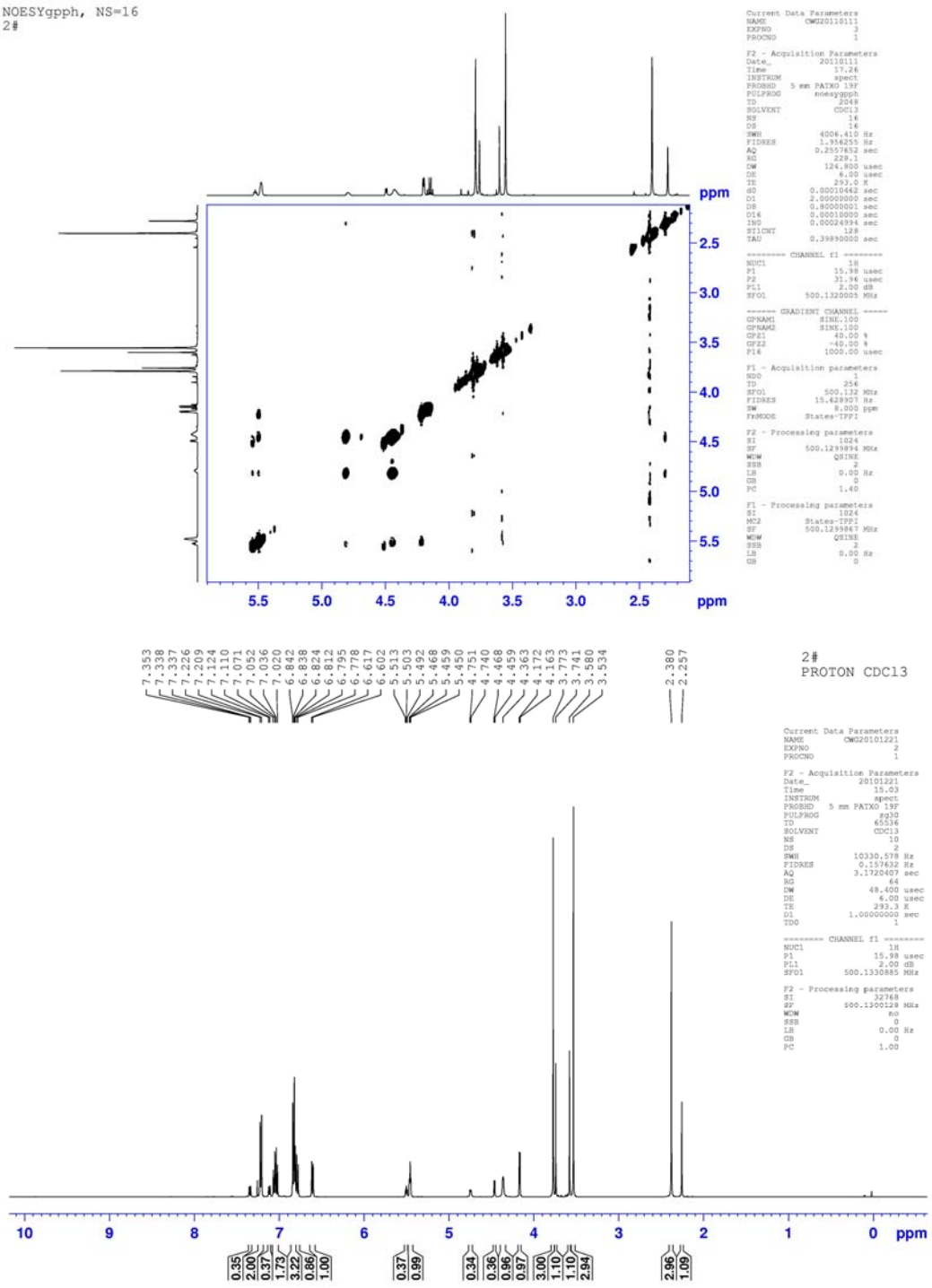

III-P-Br
19Fdeft CDC13
-70 -75 -80 -85 -90 -95 -100 -105 -110 -115 -120 -125 -130 -135 -140 ppm

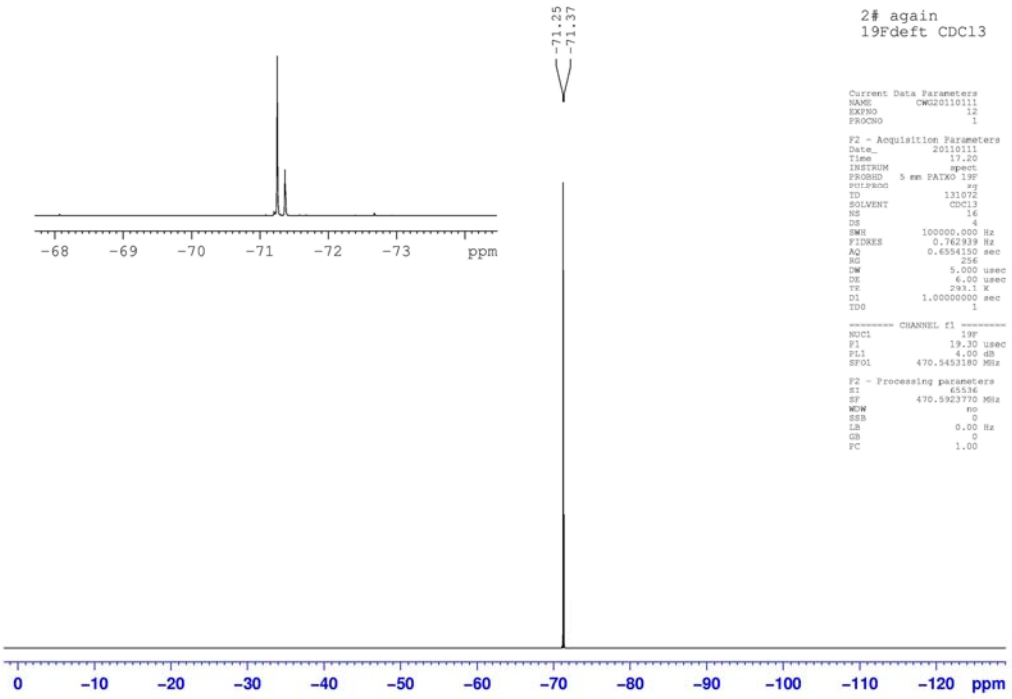

CF3
N
COOCH3
N
H
OCH3

6u


CF3
N
COOCH3
N
H
OCH3

6u'


NOESYgpph, NS=16
2#
ppm
2#
PROTON CDC13
7.353
7.338
7.337
7.226
7.209
7.124
7.110
7.071
7.052
7.036
7.020
6.842
6.838
6.824
6.812
6.795
6.778
6.617
6.602
5.513
5.503
5.492
5.468
5.459
5.450
4.751
4.740
4.468
4.459
4.363
4.172
4.163
3.773
3.741
3.580
3.534
2.380
2.257
0.35
2.00
0.37
1.73
3.22
0.86
1.00
0.37
0.99
0.34
0.36
0.96
0.97
3.00
1.10
1.10
2.94
2.96
1.09


-71.25
-71.37
2# again
19Fdeft CDCl3
Current Data Parameters
NAME CWG20110111
EXPNO 12
PROCNO 1
F2 - Acquisition Parameters
Date_ 20110111
Time 17.20
INSTRUM spect
PROBHD 5 mm PATXO 19F
PULPROG zg
TD 131072
SOLVENT CDCl3
NS 16
DS 4
SWH 100000.000 Hz
FIDRES 0.762939 Hz
AQ 0.6554150 sec
RG 256
DW 5.000 usec
DE 6.00 usec
TE 293.1 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 19F
P1 19.30 usec
PL1 4.00 dB
SFO1 470.5453180 MHz
F2 - Processing parameters
SI 65536
SF 470.5923770 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
PC 1.00
-68 -69 -70 -71 -72 -73 ppm
0 -10 -20 -30 -40 -50 -60 -70 -80 -90 -100 -110 -120 ppm