Intramolecular Nitration-Aminocarbonylation of Unactivated Olefins: Metal-Free Synthesis of γ-Lactams

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SUPPORTING INFORMATION

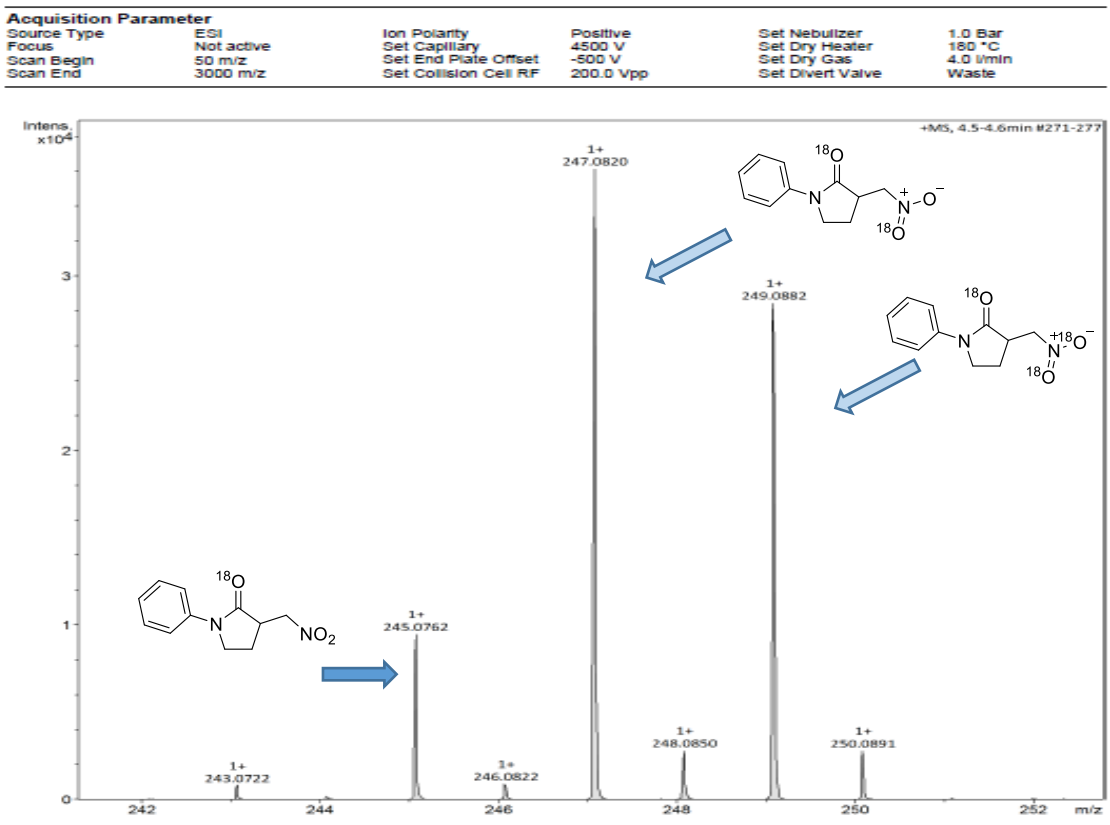
Contents

1. General experimental details

All reactions were maintained under a nitrogen atmosphere unless otherwise stated. Commercially available reagents were used without further purification. *tert*-Butyl nitrite was obtained from Energy Chemical (purity: ~90%). Infrared (FT-IR) spectra were recorded on a BRUKER VERTEX 70, ν_{max} in cm^{-1}. ^{1}H-NMR spectra were recorded on a BRUKER AVANCE III HD (400 MHz) spectrometer. Chemical shifts are reported in ppm from tetramethylsilane with the solvent resonance as internal standard ($CDCl_3$: δ 7.26). Data are reported as follows: chemical shift, multiplicity (s = singlet, d = doublet, t = triplet, q = quadruplet, br = broad, m = multiplet), coupling constants (Hz) and integration. ^{13}C-NMR spectra were recorded on a BRUKER AVANCE III HD (100 MHz) spectrometer with complete proton decoupling. Chemical shifts are reported in ppm from tetramethylsilane with the solvent resonance as the internal standard ($CDCl_3$: δ 77.16). ^{19}F-NMR spectra were recorded on a BRUKER AVANCE III HD (376 MHz) spectrometer. Mass spectra were measured with an Agilent Technologies 6120 Quadrupole LC/MS. High resolution mass spectrometry (HRMS) were measured with a GCT Premier^{TM} and BRUKER micrOTF-Q III. Melting points were measured using INESA WRR and values are uncorrected.

2. Mechanistic investigation

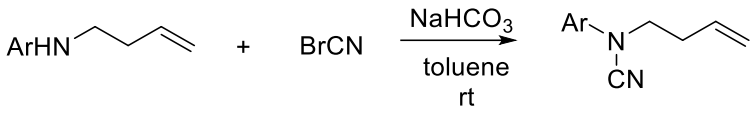
High-resolution mass spectrum for the γ-lactam products of ^{18}O-labelled experiment



HRMS spectrum of ^{18}O labelled product (**3a**)

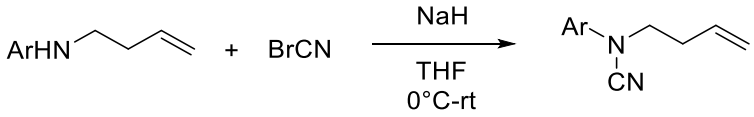
3. General procedure for synthesis of cyanamides

Procedure A (for synthesis of **1a-1d, 1n-1q, 1s-1w)**



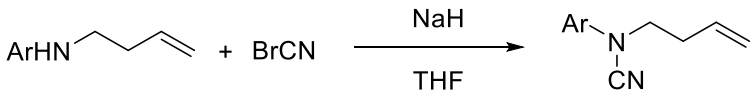
Cyanogen bromide (10.0 mmol, 1.06 g, 2.0 equiv.) was dissolved in 20 mL toluene under N_2. Aniline (5.0 mmol, 1.0 equiv.) and $NaHCO_3$ (18.0 mmol, 1.51 g, 3.6 equiv.) were added to the mixture and stirred for 12-24 h at room temperature. The mixture was quenched with water and extracted with EtOAc (3×40 mL). The organic layers were combined, washed with H_2O and brine, dried by $MgSO_4$, and concentrated under reduced pressure to give a residue, which was purified by flash column chromatography on silica gel (eluent: ethyl acetate/ petroleum ether (v/v, 1:30)).

Procedure B (for synthesis of **1h-1k)**



A 25 mL three-necked flask was charged with NaH (320 mg, 60% wt, 8.0 mmol), which was then evacuated and backfilled with N_2 for three times. THF (8 mL) was added and the solution was cooled to 0℃. Then a THF solution (4 mL) of benzamide (760 mg, 4.0 mmol) was added over 10 min to the solution. Then cyanogen bromide (850 mg, 8.0 mmol) was added into the reaction at 0℃ and the reaction was kept at RT for 24-72 h. The solution was filtered, and the resulting filtrate was concentrated and then purified through flash column chromatography on silica gel (eluent: ethyl acetate/ petroleum ether (v/v, 1:30)).

Procedure C (for synthesis of **1e-1g, 1l-1m, 1r)**

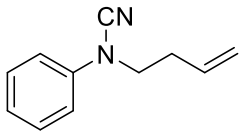


A 25 mL three-necked flask was charged with NaH (320 mg, 60% wt, 8.0 mmol), which was then evacuated and backfilled with N_2 for three times. THF (8 mL) was added and the solution was cooled to 0 ℃. Then a THF solution (4 mL) of benzamide (760 mg, 4.0 mmol) was added over 10 min to the solution and the reaction was kept at 70 ℃ for 2 h. Then cyanogen bromide (850 mg, 8.0 mmol) was added into the reaction solution at 0 ℃ and the reaction was kept at RT for 24-72 h. The solution was filtered, and the resulting filtrate was concentrated and then purified through flash column chromatography on silica gel (eluent: ethyl acetate/ petroleum ether (v/v, 1:30)).

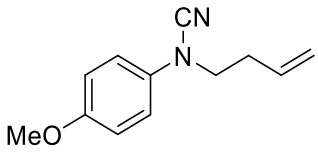
4. General procedure for nitration of olefins

Cyanamide **1** (0.2 mmol, 1.0 equiv.) was loaded in a flame-dried Schlenk tube. CH_3CN (2.0 mL) followed by *t*BuONO (0.60 mmol, 3.0 equiv.) was added to the mixture via syringe and the mixture was then stirred at 100℃ for 12 h. The mixture was concentrated, and purified by flash column chromatography on silica gel (eluent: ethyl acetate/ petroleum ether) to give the product **3**.

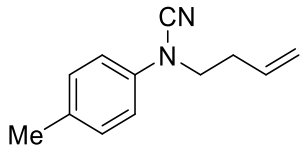
5. Characterization of cyanamides



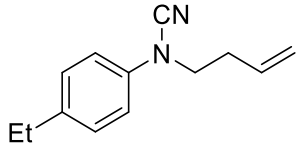
1a: yellow oil. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.40-7.34 (m, 2H), 7.14-7.07 (m, 3H), 5.90-5.78 (m, 1H), 5.22-5.13 (m, 2H), 3.65 (t, *J* = 7.2 Hz, 2H), 2.61-2.53 (m, 2H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 139.3, 132.6, 129.2, 123.2, 118.1, 115.5, 113.0, 48.3, 31.2; FT-IR: ν (cm^{-1}) 3055, 2922, 2854, 2216, 1678, 1614, 1572, 1509, 1483, 1455, 1439, 1406, 1367, 1311, 1278. HRMS [ESI] calcd for $C_{11}H_{12}N_2Na$ $[M+Na]^+$ 195.0893, found 195.0902.



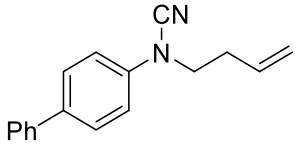
1b: dark oil. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.08-7.02 (m, 2H), 6.92-6.85 (m, 2H), 5.87-5.74 (m, 1H), 5.20-5.10 (m, 2H), 3.79-3.75 (m, 3H), 3.60-3.54 (m, 2H), 2.56-2.47 (m, 2H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 156.3, 133.2, 133.1, 118.4, 118.4, 114.9, 114.4, 55.6, 49.8, 31.7; FT-IR: ν (cm^{-1}) 3078, 3003, 2935, 2836, 2213, 1641, 1589, 1508, 1463, 1441, 1354, 1291, 1246, 1212. HRMS [ESI] calcd for $C_{12}H_{14}N_2ONa$ $[M+Na]^+$ 225.0998, found 225.0991.



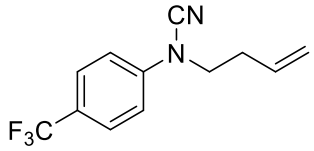
1c: yellow oil. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.19-7.12 (m, 2H), 7.03-6.97 (m, 2H), 5.88-5.76 (m, 1H), 5.21-5.11 (m, 2H), 2.61 (t, *J* = 7.2 Hz, 2H), 2.58-2.50 (m, 2H), 2.31 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 136.9, 132.9, 132.7, 129.7, 118.0, 115.7, 113.4, 48.5, 31.2, 20.1; FT-IR: ν (cm^{-1}) 3079, 3032, 2980, 2214, 1642, 1614, 1584, 1458, 1418, 1353, 1312, 1296, 1266, 1213. HRMS [ESI] calcd for $C_{12}H_{14}N_2Na$ $[M+Na]^+$ 209.1049, found 209.1039.



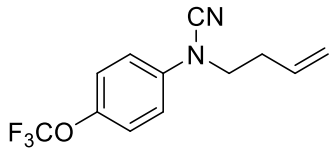
1d: yellow oil. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.16-7.07 (m, 2H), 7.02-6.94 (m, 2H), 5.84-5.69 (m, 1H), 5.16-5.04 (m, 2H), 3.59-3.48 (m, 2H), 2.61-2.50 (m, 2H), 2.50-2.42 (m, 2H), 1.20-1.11 (m, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 139.5, 137.5, 133.2, 128.9, 118.3, 116.0, 113.7, 48.7, 31.6, 27.9, 15.6; FT-IR: ν (cm^{-1}) 3080, 2965, 2932, 2215, 1642, 1612, 1582, 1540, 1512, 1458, 1437, 1353, 1266, 1214. HRMS [ESI] calcd for $C_{13}H_{16}N_2Na$ $[M+Na]^+$ 223.1206, found 223.1191.



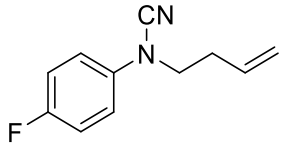
1e: white solid, m.p. 85-86 ℃. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.64-7.59 (m, 2H), 7.59-7.54 (m, 2H), 7.48-7.41 (m, 2H), 7.38-7.32 (m, 1H), 7.22-7.17 (m, 2H), 5.93-5.80 (m, 1H), 5.26-5.16 (m, 2H), 3.70 (t, J = 7.2 Hz, 2H), 2.66-2.59 (m, 2H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 139.4, 138.6, 136.2, 132.5, 128.4, 127.8, 126.9, 126.3, 118.2, 115.9, 113.0, 48.4, 31.2; FT-IR: ν (cm^{-1}) 3083, 3033, 2923, 2209, 1642, 1609, 1521, 1484, 1469, 1452, 1418, 1397, 1331, 1312, 1267. HRMS [ESI] calcd for $C_{17}H_{16}N_2Na$ $[M+Na]^+$ 271.1206, found 271.1202.



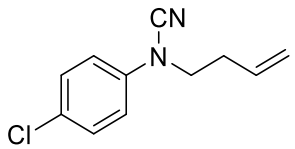
1f: yellow oil. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.61 (d, J = 8.4 Hz, 2H), 7.19 (d, J = 8.4 Hz, 2H), 5.89-5.76 (m, 1H), 5.24-5.14 (m, 2H), 3.69 (t, J = 7.2 Hz, 2H), 2.62-2.54 (m, 2H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 142.8, 132.6, 127.0 (q, $J_{C\text{-}F}$ = 3.8 Hz), 125.6 (q, $J_{C\text{-}F}$ = 39.9 Hz), 123.9 (q, $J_{C\text{-}F}$ = 269.9 Hz), 119.1, 115.6, 112.3, 48.8, 31.5; ^{19}F NMR (376 MHz, $CDCl_3$) δ -62.0 (s). FT-IR: ν (cm^{-1}) 3077, 2956, 2922, 2857, 2218, 1687, 1605, 1491, 1456, 1408, 1364, 1316, 1277, 1217. HRMS [ESI] calcd for $C_{12}H_{11}F_3N_2Na$ $[M+Na]^+$ 263.0767, found 263.0760.



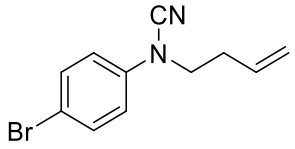
1g: yellow oil. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.15 (d, J = 8.4 Hz, 2H), 7.11-7.02 (m, 2H), 5.84-5.70 (m, 1H), 5.18-5.03 (m, 2H), 3.63-3.53 (m, 2H), 2.55-2.45 (m, 2H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 144.8, 138.6, 132.7, 122.4, 120.4 (d, $J_{C\text{-}F}$ = 255.4 Hz), 118.5 (d, $J_{C\text{-}F}$ = 1.2 Hz), 117.0, 112.8, 48.9, 31.4; ^{19}F NMR (376 MHz, $CDCl_3$) δ -58.4 (s). FT-IR: ν (cm^{-1}) 3057, 2956, 2924, 2854, 2216, 1739, 1611, 1506, 1458, 1408, 1367, 1324, 1222. HRMS [ESI] calcd for $C_{12}H_{11}F_3N_2ONa$ $[M+Na]^+$ 279.0716, found 279.0701.



1h: yellow oil. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.09-6.98 (m, 4H), 5.85-5.72 (m, 1H), 5.18-5.08 (m, 2H), 3.58 (t, J = 7.2 Hz, 2H), 2.55-2.46 (m, 2H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 158.6 (d, $J_{C\text{-}F}$ = 242.1 Hz), 135.6 (d, $J_{C\text{-}F}$ = 2.6 Hz), 132.5, 118.2, 117.5 (d, $J_{C\text{-}F}$ = 8.0 Hz), 116.0 (d, $J_{C\text{-}F}$ = 23.0 Hz), 113.1, 48.9, 31.1; ^{19}F NMR (376 MHz, $CDCl_3$) δ -119.3 (s). FT-IR: ν (cm^{-1}) 3080, 2981, 2217, 1643, 1558, 1506, 1472, 1437, 1355, 1318, 1297, 1278, 1230, 1210. HRMS [ESI] calcd for $C_{11}H_{12}FN_2$ $[M+H]^+$ 191.0979, found 191.0973.

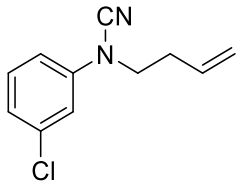


1i: yellow oil. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.32-7.27 (m, 2H), 7.05-6.99 (m, 2H), 5.86-5.73 (m, 1H), 5.20-5.10 (m, 2H), 3.60 (t, J = 7.2 Hz, 2H), 2.57-2.48 (m, 2H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 138.5, 132.8, 129.7, 128.9, 118.8, 117.3, 113.0, 49.0, 31.6; FT-IR: ν (cm^{-1}) 3079, 2931, 2218, 1642, 1596, 1492, 1437, 1419, 1353, 1319, 1293, 1269, 1217. HRMS [ESI] calcd for $C_{11}H_{11}ClN_2Na$ $[M+Na]^+$ 229.0503, found 229.0509.

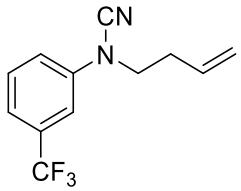


1j: yellow oil. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.47-7.40 (m, 2H), 7.00-6.93 (m, 2H), 5.86-5.73 (m, 1H), 5.20-5.11 (m, 2H), 3.60 (t, J = 7.2 Hz, 2H), 2.58-2.49 (m, 2H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 139.1, 132.8, 132.6, 118.8, 117.6, 116.3, 112.9, 48.9, 31.5; FT-IR: ν (cm^{-1}) 3076,

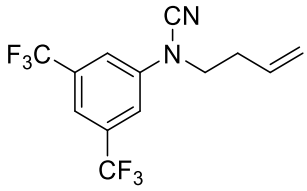
3006, 2948, 2213, 1643, 1591, 1489, 1469, 1435, 1324, 1292, 1263, 1217. HRMS [ESI] calcd for $C_{11}H_{12}BrN_2$ $[M+H]^+$ 251.0178, found 251.0176.



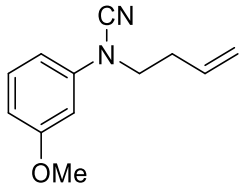
1k: yellow oil. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.27-7.20 (m, 1H), 7.08-7.03 (m, 1H), 7.03-6.98 (m, 1H), 6.95 (dd, J = 8.4, 2.0 Hz, 1H), 5.85-5.70 (m, 1H), 5.20-5.06 (m, 2H), 3.58 (t, J = 7.2 Hz, 2H), 2.55-2.46 (m, 2H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 140.7, 135.0, 132.3, 130.2, 123.2, 118.3, 115.5, 113.6, 112.1, 48.2, 31.0; FT-IR: ν (cm^{-1}) 3079, 2980, 2923, 2219, 1694, 1580, 1480, 1420, 1398, 1377, 1350, 1313, 1288, 1263, 1219. HRMS [ESI] calcd for $C_{11}H_{11}ClN_2Na$ $[M+Na]^+$ 229.0503, found 229.0497.



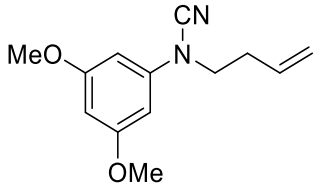
1l: yellow oil. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.52-7.45 (m, 1H), 7.36-7.28 (m, 3H), 5.90-5.75 (m, 1H), 5.22-5.13 (m, 2H), 2.68 (t, J = 7.2 Hz, 2H), 2.62-2.53 (m, 2H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 139.6, 131.7, 131.2 (q, $J_{C\text{-}F}$ = 32.5 Hz), 129.4, 122.6 (q, $J_{C\text{-}F}$ = 271.0 Hz), 119.3 (q, $J_{C\text{-}F}$ = 3.7 Hz), 118.1 (q, $J_{C\text{-}F}$ = 0.9 Hz), 117.9, 111.5 (q, $J_{C\text{-}F}$ = 3.8 Hz), 111.5, 47.8, 30.5;^{19}F NMR (376 MHz, $CDCl_3$) δ -62.9 (s).FT-IR: ν (cm^{-1}) 3081, 2982, 2931, 2222, 1771, 1616, 1596, 1496, 1457, 1323, 1217. HRMS [ESI] calcd for $C_{12}H_{11}F_3N_2Na$ $[M+Na]^+$ 263.0767, found 263.0760.



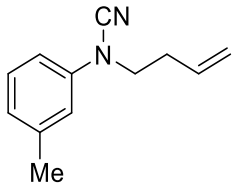
1m: yellow oil. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.60 (s, 1H), 7.52 (s, 2H), 5.90-5.76 (m, 1H), 5.28-5.15 (m, 2H), 3.74 (t, J = 7.2 Hz, 2H), 2.66-2.58 (m, 2H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 141.3, 132.9 (q, $J_{C\text{-}F}$ = 33.6 Hz), 131.7, 121.6 (q, $J_{C\text{-}F}$ = 271.4 Hz), 118.9, 116.7-116.5 (m), 115.3-115.0 (m), 110.9, 48.5, 30.9;^{19}F NMR (376 MHz, $CDCl_3$) δ -63.2 (s). FT-IR: ν (cm^{-1}) 3063, 2922, 2854, 2220, 1683, 1596, 1574, 1506, 1479, 1395, 1367, 1336, 1311, 1277, 1211. HRMS [ESI] calcd for $C_{13}H_{10}F_6N_2Na$ $[M+Na]^+$ 331.0640, found 331.0630.



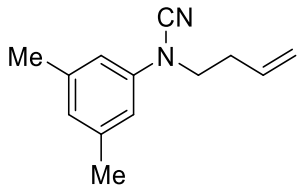
1n: yellow oil. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.14-7.04 (m, 1H), 6.58-6.42 (m, 3H), 5.74-5.62 (m, 1H), 5.07-4.95 (m, 2H), 3.61 (s, 3H), 3.43 ((t, J = 7.2 Hz, 2H), 2.42-2.32 (m, 2H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 160.2, 140.6, 132.8, 129.9, 117.7, 112.6, 108.0, 107.4, 101.8, 54.7, 48.0, 31.0,; FT-IR: ν (cm^{-1}) 3079, 2941, 2838, 2216, 1603, 1589, 1491, 1456, 1438, 1351, 1334 1293, 1256, 1230, 1201. HRMS [ESI] calcd for $C_{12}H_{15}N_2O$ $[M+H]^+$ 203.1179, found 203.1180.



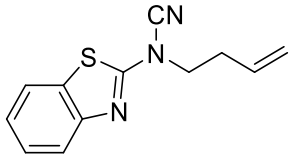
1o: dark oil. ^{1}H NMR (400 MHz, $CDCl_3$) δ 6.26-6.07 (m, 3H), 5.88-5.68 (m, 1H), 5.22-5.02 (m, 2H), 3.83-3.64 (m, 6H), 3.62-3.45 (m, 2H), 2.59-2.41 (m, 2H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 161.7, 141.7, 133.1, 118.6, 113.3, 95.2, 94.8, 55.5, 48.8, 31.6; FT-IR: ν (cm^{-1}) 3084, 3002, 2909, 2213, 1598, 1559, 1541, 1521, 1507, 1459, 1441, 1403, 1381, 1331, 1301, 1211. HRMS [ESI] calcd for $C_{13}H_{16}N_2O_2Na$ $[M+Na]^+$ 255.1104, found 255.1104.



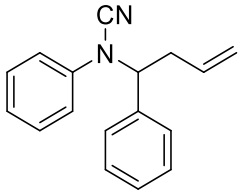
1p: yellow oil. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.27-7.19 (m, 1H), 6.94 (s, 1H), 6.92-6.85 (m, 2H), 5.89-5.76 (m, 1H), 5.21-5.11 (m, 2H), 3.65-3.57 (m, 2H), 2.58-2.50 (m, 2H), 2.34 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 139.8, 139.8, 133.2, 129.5, 124.5, 118.5, 116.8, 113.7, 113.0, 48.7, 31.7, 21.6; FT-IR: ν (cm^{-1}) 3079, 2979, 2925, 2214, 1642, 1607, 1589, 1493, 1462, 1438, 1350, 1319, 1276, 1238. HRMS [ESI] calcd for $C_{12}H_{14}N_2Na$ $[M+Na]^+$ 209.1049, found 209.1049.



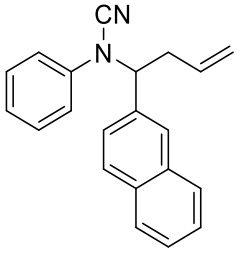
1q: yellow oil. ^{1}H NMR (400 MHz, $CDCl_3$) δ 6.59 (s, 2H), 6.53 (s, 1H), 5.75-5.63 (m, 1H), 5.13-4.96 (m, 2H), 3.39 (t, *J* = 7.2 Hz, 2H), 2.40-2.31 (m, 2H), 2.13 (s, 6H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 139.8, 139.3, 133.4, 125.1, 118.0, 113.5, 113.3, 48.3, 31.6, 21.2; FT-IR: ν (cm^{-1}) 3084, 3002, 2909, 2213, 1598, 1559, 1541, 1521, 1507, 1459, 1441, 1403, 1381, 1331, 1301, 1211. HRMS [ESI] calcd for $C_{13}H_{16}N_2Na$ $[M+Na]^+$ 223.1206, found 223.1199.



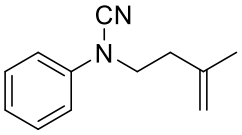
1r: yellow solid, m.p. 44-45 °C. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.77 (d, *J* = 8.0 Hz, 1H), 7.73 (d, *J* = 7.6 Hz, 1H), 7.46-7.40 (m, 1H), 7.34-7.27 (m, 1H), 5.92-5.78 (m, 1H), 5.31-5.15 (m, 2H), 4.03 (t, *J* = 7.2 Hz, 2H), 2.72-2.62 (m, 2H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 161.4, 151.1, 132.6, 132.3, 126.7, 124.4, 121.6, 121.4, 119.1, 111.2, 50.1, 31.8; FT-IR: ν (cm^{-1}) 3058, 2925, 2856, 2226, 1644, 1594, 1524, 1473, 1457, 1441, 1381, 1311, 1267, 1222, 1204. HRMS [ESI] calcd for $C_{12}H_{11}N_3SNa$ $[M+Na]^+$ 252.0566, found 252.0560.



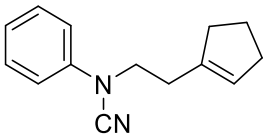
1s: yellow oil. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.47-7.35 (m, 4H), 7.35-7.26 (m, 3H), 7.15 (d, *J* = 8.0 Hz, 1H), 7.06 (t, *J* = 7.6 Hz, 1H), 5.92-5.77 (m, 1H), 5.29 (d, *J* = 16.8 Hz, 1H), 5.19 (d, *J* = 10.4 Hz, 1H), 4.72 (dd, *J* = 9.2, 6.0 Hz, 1H), 3.13-3.00 (m, 1H), 2.87-2.74 (m, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 140.0, 138.7, 132.5, 129.1, 128.6, 127.9, 125.8, 123.5, 118.6, 116.7, 112.1, 62.1, 39.3; FT-IR: ν (cm^{-1}) 3077, 3028, 2932, 2214, 1644, 1596, 1491, 1453, 1434, 1420, 1374, 1346, 1305, 1276, 1258, 1227. HRMS [ESI] calcd for $C_{17}H_{16}N_2Na$ $[M+Na]^+$ 271.1206, found 271.1200.



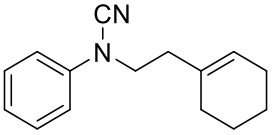
1t: white solid, m.p. 63-64 °C. ^{1}H NMR (400 MHz, $CDCl_3$) δ7.94-7.82 (m, 4H), 7.59 (dd, *J* = 8.4, 1.6 Hz, 1H), 7.56-7.48 (m, 2H), 7.34-7.27 (m, 2H), 7.26-7.20 (m, 2H), 5.98-5.85 (m, 1H), 5.40-5.31 (m, 1H), 5.26-5.20 (m, 1H), 4.92 (dd, *J* = 9.2, 6.0 Hz, 1H), 3.24-3.12 (m, 1H), 2.99-2.89 (m, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 140.3, 136.2, 132.8, 132.7, 132.6, 129.2, 128.8, 127.6, 127.3, 126.2, 126.1, 125.1, 123.7, 123.5, 118.8, 116.9, 112.3, 62.3, 39.4; FT-IR: ν (cm^{-1}) 3076, 3055, 2966, 2216, 1643, 1596, 1558, 1541, 1513, 1496, 1474, 1457, 1346, 1304, 1231. HRMS [ESI] calcd for $C_{21}H_{18}N_2Na$ $[M+Na]^+$ 321.1362, found 321.1365.



1u: yellow oil. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.29-7.19 (m, 2H), 7.12-6.90 (m, 3H), 4.79-4.75 (m, 1H), 4.69-4.65 (m, 1H), 3.53 (t, *J* = 7.2 Hz, 2H), 2.34 (t, *J* = 7.2 Hz, 2H), 1.67 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 140.7, 139.8, 129.6, 123.4, 115.8, 113.2, 47.4, 35.0, 22.4; FT-IR: ν (cm^{-1}) 3076, 2971, 2937, 2216, 1716, 1698, 1650, 1598, 1541, 1497, 1458, 1357, 1317, 1275, 1234, 1210. HRMS [ESI] calcd for $C_{12}H_{14}N_2Na$ $[M+Na]^+$ 209.1049, found 209.1049.

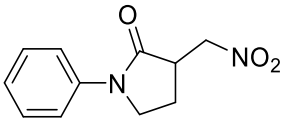


1v: yellow oil. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.34-7.26 (m, 2H), 7.09-7.00 (m, 3H), 5.44-5.38 (m, 1H), 3.63 (t, *J* = 7.2 Hz, 2H), 2.51 (t, *J* = 7.2 Hz, 2H), 2.31-2.19 (m, 4H), 1.89-1.77 (m, 2H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 139.8, 139.3, 129.7, 126.9, 123.5, 115.9, 113.4, 47.7, 35.0, 32.6, 28.9, 23.3; FT-IR: ν (cm^{-1}) 2959, 2892, 2842, 1597, 1497, 1470, 1459, 1442, 1405, 1359, 1316, 1298, 1258, 1222. HRMS [ESI] calcd for $C_{14}H_{16}N_2Na$ $[M+Na]^+$ 235.1206, found 235.1199.

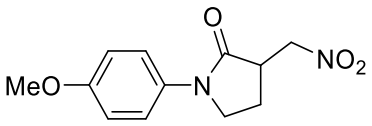


1w: dark oil. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.38-7.26 (m, 2H), 7.12-6.99 (m, 3H), 5.50-5.40 (m, 1H), 3.68-3.54 (m, 2H), 2.44-2.30 (m, 2H), 2.01-1.88 (m, 4H), 1.64-1.46 (m, 4H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 139.4, 132.3, 129.1, 124.5, 122.9, 115.4, 113.0, 47.5, 35.1, 27.7, 24.8, 22.3, 21.6; FT-IR: ν (cm^{-1}) 2926, 2857, 2834, 1597, 1497, 1458, 1436, 1367, 1355, 1321, 1311, 1277, 1266, 1210. HRMS [ESI] calcd for $C_{15}H_{19}N_2$ $[M+H]^+$ 227.1543, found 227.1541.

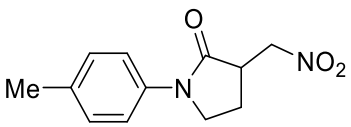
6. Characterization of products



3a: 37.1 mg, yield 84%. Yellow solid, 97-98 ℃. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.63-7.52 (m, 2H), 7.43-7.30 (m, 2H), 7.22-7.13 (m, 1H), 4.92-4.84 (m, 1H), 4.62-4.52 (m, 1H), 3.93-3.77 (m, 2H), 3.45-3.31 (m, 1H), 2.56-2.43 (m, 1H), 2.09-1.93 (m, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 170.7, 138.8, 129.0, 125.2, 120.0, 75.8, 46.5, 41.9, 23.3. FT-IR: ν (cm^{-1}) 3055, 2982, 1671, 1652, 1558, 1533, 1480, 1445, 1266, 1213. HRMS [ESI] calcd for $C_{11}H_{12}N_2O_3Na$ $[M+Na]^+$ 243.0740, found 243.0747.

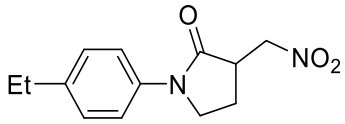


3b: 32.6 mg, yield 65%. Yellow solid, m.p. 108-109 ℃. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.51-7.45 (m, 2H), 6.95-6.87 (m, 2H), 4.97-4.88 (m, 1H), 4.63-4.54 (m, 1H), 3.93-3.76 (m, 2H), 3.80 (s, 3H), 3.46-3.34 (m, 1H), 2.59-2.47 (m, 1H), 2.11-1.97 (m, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 170.3, 157.1, 131.9, 121.9, 114.2, 76.1, 55.5, 47.0, 41.8, 23.5. FT-IR: ν (cm^{-1}) 2955, 2919, 2851, 1670, 1596, 1549, 1510, 1456, 1434, 1401, 1377, 1332, 1287, 1253. HRMS [ESI] calcd for $C_{12}H_{14}N_2O_4Na$ $[M+Na]^+$ 273.0846, found 273.0856.

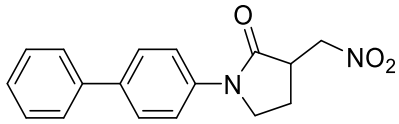


3c: 37.0 mg, yield 79%. Yellow solid, m.p. 63-64 ℃. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.50-7.42 (m, 2H), 7.22-7.14 (m, 2H), 4.96-4.88 (m, 1H), 4.63-4.53 (m, 1H), 3.94-3.78 (m, 2H), 3.47-3.35 (m, 1H), 2.58-2.47 (m, 1H), 2.34 (s, 3H), 2.10-1.97 (m, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 170.4, 136.2, 135.0, 129.5, 120.1, 76.0, 46.7, 41.9, 23.5, 20.9. FT-IR: ν (cm^{-1})

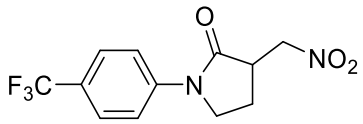
2960, 2888, 1683, 1536, 1508, 1482, 1453, 1419, 1392, 1381, 1310, 1248, 1223, 1200. HRMS [ESI] calcd for $C_{12}H_{14}N_2O_3Na$ [M+Na]$^+$ 257.0897, found 257.0900.



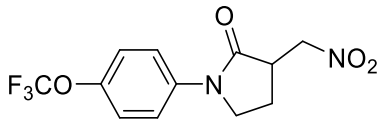
3d: 26.8 mg, yield 54%. Yellow solid, m.p. 64-65 ℃. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.52-7.47 (m, 2H), 7.24-7.19 (m, 2H), 4.97-4.90 (m, 1H), 3.94-3.80 (m, 2H), 3.47-3.37 (m, 1H), 2.64 (q, *J* = 7.6 Hz, 2H), 2.59-2.49 (m, 1H), 2.11-1.98 (m, 1H), 1.23 (t, *J* = 7.6 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 170.4, 141.5, 136.4, 128.4, 120.2, 76.1, 46.7, 41.9, 28.3, 23.5, 15.6. FT-IR: ν (cm^{-1}) 2952, 2822, 1679, 1610, 1577, 1519, 1489, 1453, 1439, 1403, 1377, 1321, 1267. HRMS [ESI] calcd for $C_{13}H_{16}N_2O_3Na$ [M+Na]$^+$ 271.1053, found 271.1057.



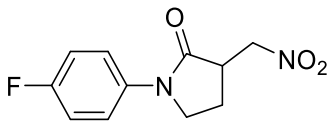
3e: 26.1 mg, yield 44%. Yellow solid, m.p. 108-109 ℃. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.71-7.66 (m, 2H), 7.64-7.61 (m, 2H), 7.60-7.53 (m, 2H), 7.47-7.41 (m, 2H), 7.37-7.32 (m, 1H), 4.98-4.92 (m, 1H), 4.66-4.58 (m, 1H), 4.00-3.87 (m, 2H), 3.51-3.40 (m, 1H), 2.63-2.52 (m, 1H), 2.15-2.01 (m, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 170.7, 140.2, 138.1, 138.0, 128.9, 127.6, 127.4, 126.9, 120.3, 75.9, 46.6, 42.0, 23.4. FT-IR: ν (cm^{-1}) 3077, 2952, 2854, 1683, 1605, 1577, 1506, 1483, 1453, 1436, 1406, 1367, 1322, 1222. HRMS [ESI] calcd for $C_{17}H_{16}N_2O_3Na$ [M+Na]$^+$ 319.1053., found 319.1049.



3f: 31.7 mg, yield 55%. Yellow solid, m.p. 101-102 ℃. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.80-7.73 (m, 2H), 7.68-7.61 (m, 2H), 4.96-4.87 (m, 1H), 4.69-4.59 (m, 1H), 3.99-3.86 (m, 2H), 3.50-3.39 (m, 1H), 2.64-2.53 (m, 1H), 2.16-2.02 (m, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 171.2, 141.7 (q, $J_{C\text{-}F}$ = 0.8 Hz), 126.8 (q, $J_{C\text{-}F}$ = 32.6 Hz), 126.2 (q, $J_{C\text{-}F}$ = 3.8 Hz), 124.0 (q, $J_{C\text{-}F}$ = 270.0 Hz), 119.4, 75.8, 46.8, 41.7, 23.3; ^{19}FNMR (376MHz, $CDCl_3$) δ -62.3 (s). FT-IR: ν (cm^{-1}) 3077, 2922, 2854, 1677, 1596, 1577, 1509, 1483, 1456, 14376, 1405, 1378, 1309, 1277. HRMS [ESI] calcd for $C_{12}H_{11}F_3N_2O_3Na$ [M+Na]$^+$ 311.0614, found 311.0615.

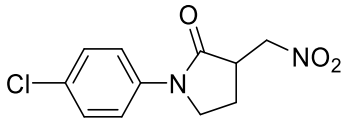


3g: 53.5 mg, yield 88%. Yellow solid, m.p. 75-76 ℃. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.68-7.62 (m, 2H), 7.29-7.20 (m, 2H), 4.95-4.86 (m, 1H), 4.67-4.58 (m, 1H), 3.95-3.81 (m, 2H), 3.47-3.36 (m, 1H), 2.63-2.49 (m, 1H), 2.14-2.00 (m, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 170.8, 145.9 (q, $J_{C\text{-}F}$ = 2.0 Hz), 137.4, 121.7, 121.1, 120.7 (q, $J_{C\text{-}F}$ = 255.6 Hz), 75.6, 46.5, 41.8, 23.2; ^{19}FNMR (376MHz, $CDCl_3$) δ -58.1 (s). FT-IR: ν (cm^{-1}) 2953, 2922, 2872, 1687, 1605, 1592, 1549, 1503, 1455, 1463, 1421, 1362, 1309, 1277. HRMS [ESI] calcd for $C_{12}H_{11}F_3N_2O_4Na$ [M+Na]$^+$ 327.0563, found 327.0553.

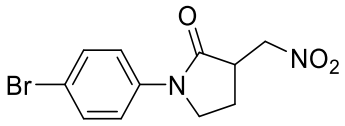


3h: 31.4 mg, yield 66%. Yellow solid, m.p. 76-77 ℃. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.61-7.53 (m, 2H), 7.13-7.04 (m, 2H), 4.97-4.88 (m, 1H), 4.66-4.57 (m, 1H), 3.95-3.79 (m, 2H), 3.48-3.36 (m, 1H), 2.61-2.50 (m, 1H), 2.14-2.00 (m, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 170.6, 159.9 (d, $J_{C\text{-}F}$ = 243.3 Hz), 134.9 (d, $J_{C\text{-}F}$ = 2.3 Hz), 121.9 (d, $J_{C\text{-}F}$ = 8.0 Hz), 115.8 (d, $J_{C\text{-}F}$ = 22.4 Hz), 75.8, 46.8, 41.7, 23.3 (d, $J_{C\text{-}F}$ = 2.4 Hz); ^{19}FNMR (376MHz, $CDCl_3$) δ -116.6 (s). FT-IR:

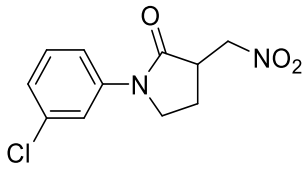
ν (cm^{-1}) 3077, 2922, 2854, 1683, 1605, 1572, 1508, 1483, 1455, 1436, 1406, 1357, 1309, 1277. HRMS [ESI] calcd for $C_{11}H_{11}FN_2O_3Na$ $[M+Na]^+$ 261.0646, found 261.0647.



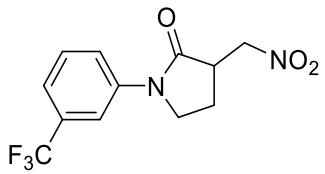
3i: 30.0 mg, yield 59%. Yellow solid, m.p. 115-116 ℃. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.62-7.52 (m, 2H), 7.40-7.30 (m, 2H), 4.96-4.84 (m, 1H), 4.68-4.54 (m, 1H), 3.94-3.76 (m, 2H), 3.48-3.34 (m, 1H), 2.61-2.47 (m, 1H), 2.12-1.97 (m, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 170.7, 137.4, 130.3, 129.0, 121.1, 75.7, 46.4, 41.8, 23.2. FT-IR: ν (cm^{-1}) 2949, 2918, 2850, 1682, 1596, 1549, 1494, 1459, 1434, 1422, 1394, 1376, 1317, 1291, 1278, 1255, 1225. HRMS [ESI] calcd for $C_{11}H_{11}ClN_2O_3Na$ $[M+Na]^+$ 277.0350, found 277.0354.



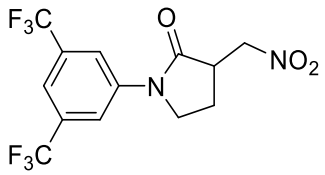
3j: 32.7 mg, yield 55%. Yellow solid, m.p. 129-130 ℃. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.55-7.45 (m, 4H), 4.95-4.86 (m, 1H), 4.65-4.56 (m, 1H), 3.94-3.77 (m, 2H), 3.46-3.34 (m, 1H), 2.60-2.50 (m, 1H), 2.12-1.98 (m, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 170.7, 137.8, 132.0, 121.3, 118.1, 75.7, 46.3, 41.9, 23.2. FT-IR: ν (cm^{-1}) 2917, 2850, 1683, 1588, 1549, 1516, 1507, 1491, 1457, 1435, 1419, 1393, 1375, 1318, 1290, 1225. HRMS [ESI] calcd for $C_{11}H_{11}BrN_2O_3Na$ $[M+Na]^+$ 320.9845, found 320.9849.



3k: 27.4 mg, yield 54%. Yellow solid, m.p. 70-71 ℃. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.69-7.66 (m, 1H), 7.55-7.49 (m, 1H), 7.35-7.28 (m, 1H), 7.19-7.14 (m, 1H), 4.95-4.87 (m, 1H), 4.66-4.57 (m, 1H), 3.94-3.81 (m, 2H), 3.48-3.37 (m, 1H), 2.61-2.50 (m, 1H), 2.13-2.00 (m, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 170.8, 139.9, 134.8, 130.0, 125.2, 119.9, 117.7, 75.7, 46.4, 41.9, 23.2. FT-IR: ν (cm^{-1}) 3116, 3074, 2958, 2919, 1692, 1594, 1574, 1543, 1484, 1455, 1438, 1406, 1380, 1338, 1288, 1233. HRMS [ESI] calcd for $C_{11}H_{11}ClN_2O_3Na$ $[M+Na]^+$ 277.0350, found 277.0352.

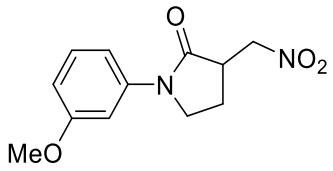


3l: 38.0 mg, yield 66%. Yellow soil. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.92-7.82 (m, 2H), 7.55-7.47 (m, 1H), 7.47-7.41 (m, 1H), 4.97-4.87 (m, 1H), 4.69-4.58 (m, 1H), 4.04-3.83 (m, 2H), 3.50-3.37 (m, 1H), 2.65-2.52 (m, 1H), 2.16-2.01 (m, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 171.1, 139.3, 131.4 (q, J_{C-F} = 2.2 Hz), 129.6, 123.8 (q, J_{C-F} = 270.8 Hz), 122.8, 121.7 (q, J_{C-F} = 3.7 Hz), 116.4 (q, J_{C-F} = 3.9 Hz), 75.5, 46.4, 41.9, 23.1; ^{19}FNMR (376MHz, $CDCl_3$) δ -62.7 (s). FT-IR: ν (cm^{-1}) 2977, 2922, 2854, 1687, 1654, 1572, 1509, 1453, 1377, 1329, 1311, 1277. HRMS [ESI] calcd for $C_{12}H_{11}F_3N_2O_3Na$ $[M+Na]^+$ 311.0614, found 311.0618.

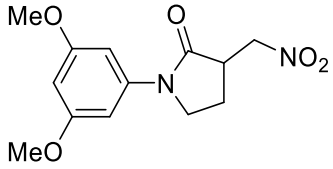


3m: 39.2 mg, yield 55%. Yellow solid, m.p. 68-69 ℃. ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.15 (s, 2H), 7.69 (s, 1H), 4.96-4.84 (m, 1H), 4.74-4.62 (m, 1H), 4.04-3.90 (m, 2H), 3.54-3.39 (m, 1H), 2.71-2.56 (m, 1H), 2.24-2.03 (m, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 171.5, 140.1, 132.4 (q, J_{C-F} = 33.5 Hz), 122.3 (q, J_{C-F} = 271.1 Hz), 119.1 (q, J_{C-F} = 3.2 Hz), 118.4-118.0 (m), 75.1, 46.2, 41.7, 22.8; ^{19}FNMR (376MHz, $CDCl_3$) δ -63.0 (s). FT-IR: ν

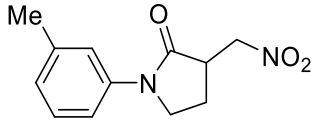
(cm^{-1}) 2973, 2927, 2884, 1697, 1684, 1653, 1576, 1455, 1379, 1329, 1276. HRMS [ESI] calcd for $C_{13}H_{10}F_6N_2O_3Na$ $[M+Na]^+$ 379.0488, found 379.0484.



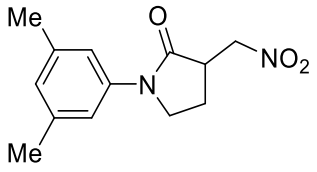
3n: 37.0 mg, yield 74%. Yellow solid, m.p. 65-66 ℃. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.35-7.31 (m, 1H), 7.31-7.25 (m, 1H), 7.14-7.06 (m, 1H), 6.77-6.71 (m, 1H), 4.97-4.87 (m, 1H), 4.64-4.54 (m, 1H), 3.95-3.83 (m, 2H), 3.82 (s, 3H), 3.49-3.36 (m, 1H), 2.59-2.48 (m, 1H), 2.33 (s, 6H), 2.10-1.97 (m, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 170.7, 160.4, 140.0, 130.0, 112.0, 110.8, 106.1, 75.9, 55.4, 46.7, 42.1, 23.4. FT-IR: ν (cm^{-1}) 2974, 2893, 2846, 1682, 1602, 1580, 1555, 1489, 1454, 1334, 1300, 1258, 1248, 1213. HRMS [ESI] calcd for $C_{12}H_{14}N_2O_4Na$ $[M+Na]^+$ 273.0846, found 273.0843.



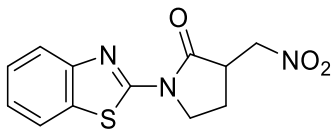
3o: 31.4 mg, yield 56%. Yellow solid, m.p. 111-112 ℃. ^{1}H NMR (400 MHz, $CDCl_3$) δ 6.84 (d, *J* = 2.4 Hz, 2H), 6.32-6.29 (m, 1H), 4.95-4.88 (m, 1H), 4.62-4.54 (m, 1H), 3.88-3.80 (m, 2H), 3.79 (s, 6H), 3.47-3.37 (m, 1H), 2.57-2.47 (m, 1H), 2.09-1.96 (m, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 170.8, 161.0, 140.5, 98.5, 97.2, 75.8, 55.5, 46.7, 42.2, 23.3. FT-IR: ν (cm^{-1}) 2948, 2920, 2848, 1686, 1605, 1582, 1548, 1497, 1457, 1436, 1381, 1320, 1298, 1270, 1222. HRMS [ESI] calcd for $C_{13}H_{16}N_2O_5Na$ $[M+Na]^+$ 303.0951, found 303.0949.



3p: 28.1 mg, yield 60%. Yellow solid, m.p. 69-70 ℃. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.42 (m, 1H), 7.35-7.29 (m, 1H), 7.25-7.21 (m, 1H), 6.98 (d *J* = 7.2 Hz, 1H), 4.94-4.86 (m, 1H), 4.61-4.50 (m, 1H), 3.93-3.74 (m, 2H), 3.45-3.33 (m, 1H), 2.56-2.44 (m, 1H), 2.35 (s, 3H), 2.08-1.95 (m, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 170.6, 139.0, 138.7, 128.8, 126.1, 120.9, 117.2, 76.0, 47.0, 42.0, 23.5, 21.6. FT-IR: ν (cm^{-1}) 2922, 2851, 1698, 1683, 1674, 1653, 1558, 1541, 1518, 1496, 1490, 1396, 1376, 1339, 1314, 1287, 1267. HRMS [ESI] calcd for $C_{12}H_{14}N_2O_3Na$ $[M+Na]^+$ 257.0897, found 257.0899.

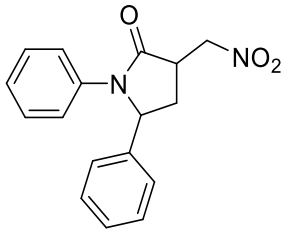


3q: 33.7 mg, yield 68%. Yellow solid, m.p. 77-78 ℃. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.20 (s, 2H), 6.84 (s, 1H), 4.97-4.89 (m, 1H), 4.64-4.53 (m, 1H), 3.95-3.78 (m, 2H), 3.47-3.35 (m, 1H), 2.58-2.47 (m, 1H), 2.33 (s, 6H), 2.10-1.95 (m, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 170.5, 138.7, 138.6, 127.1, 118.1, 76.0, 46.8, 42.0, 23.6, 21.5. FT-IR: ν (cm^{-1}) 3054, 2922, 2852, 1679, 1605, 1578, 1509, 1495, 1397, 1376, 1311, 1277, 1205. HRMS [ESI] calcd for $C_{13}H_{16}N_2O_3Na$ $[M+Na]^+$ 271.1053, found 271.1059.



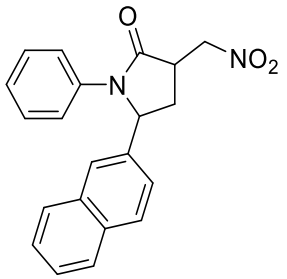
3r: 43.2 mg, yield 78%. Yellow solid, m.p. 169-170 ℃. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.91-7.79 (m, 2H), 7.51-7.42 (m, 1H), 7.39-7.29 (m, 1H), 5.00-4.87 (m, 1H), 4.74-4.63 (m, 1H), 4.52-4.41 (m, 1H), 4.21-4.09 (m, 1H), 3.63-3.43 (m, 1H), 2.74-2.59 (m, 1H), 2.25-2.09 (m, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 171.1, 156.5, 148.5, 132.3, 126.4, 124.4, 121.7, 121.5, 74.8, 46.2, 41.5, 23.4. FT-IR: ν (cm^{-1}) 2927, 2850, 1698, 1626, 1576, 1543, 1515, 1488, 1443,

1419, 1376, 1316, 1284, 1266, 1241. HRMS [ESI] calcd for $C_{12}H_{11}N_3O_3SNa$ $[M+Na]^+$ 300.0413, found 300.0410.



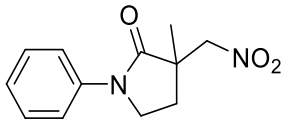
3s-a: 31.3 mg, yield 53%. Yellow solid, m.p. 125-126 °C. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.54-7.47 (m, 2H), 7.39-7.33 (m, 2H), 7.33-7.26 (m, 3H), 7.24-7.20 (m, 2H), 7.15-7.08 (m, 1H), 5.29 (dd, *J* = 8.4, 1.2 Hz, 1H), 4.97-4.89 (m, 1H), 4.68-4.59 (m, 1H), 3.69-3.50 (m, 1H), 2.61-2.50 (m, 1H), 2.44-2.35 (m, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 171.4, 140.1, 138.0, 129.3, 129.0, 128.2, 125.5, 125.4, 121.4, 75.6, 61.7, 39.8, 33.9. FT-IR: ν (cm^{-1}) 2921, 2851, 1698, 1674, 1653, 1541, 1518, 1490, 1457, 1395, 1377, 1362, 1314, 1287, 1259, 1233. HRMS [ESI] calcd for $C_{17}H_{16}N_2O_3Na$ $[M+Na]^+$ 319.1053, found 319.1060.

3s-b: 16.0 mg, yield 27%. Yellow solid, m.p. 145-146 °C. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.33-7.27 (m, 2H), 7.27-7.22 (m, 3H), 7.22-7.16 (m, 4H), 7.11-7.03 (m, 1H), 5.31-5.22 (m, 1H), 5.02-4.92 (m, 1H), 4.79-4.68 (m, 1H), 3.51-3.40 (m, 1H), 3.00-2.89 (m, 1H), 1.99-1.87 (m, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 171.4, 139.8, 137.0, 129.0, 128.7, 128.2, 126.6, 125.8, 123.3, 75.7, 61.9, 41.4, 34.9. FT-IR: ν (cm^{-1}) 3030, 3006, 2954, 2851, 1700, 1597, 1548, 1493, 1456, 1428, 1418, 1383, 1358, 1321, 1285, 1254, 1210. HRMS [ESI] calcd for $C_{17}H_{16}N_2O_3Na$ $[M+Na]^+$ 319.1053, found 319.1056.



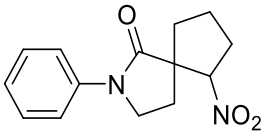
3t-a: 44.2 mg, yield 64%. Yellow solid, m.p. 143-144 °C. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.93-7.88 (m, 1H), 7.88-7.82 (m, 1H), 7.81-7.75 (m, 1H), 7.66 (s, 1H), 7.59-7.53 (m, 2H), 7.53-7.47 (m, 2H), 7.38 (dd, *J* = 8.4, 1.2 Hz, 1H), 7.31-7.25 (m, 2H), 7.17-7.08 (m, 1H), 5.45 (d, *J* = 8.0 Hz, 1H), 5.00-4.90 (m, 1H), 4.74-4.64 (m, 1H), 3.66-3.54 (m, 1H), 2.70-2.56 (m, 1H), 2.54-2.42 (m, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 171.5, 138.0., 137.5, 133.3, 133.1, 129.6, 129.0, 127.9, 127.8, 126.8, 126.5, 125.5, 124.4, 123.3, 121.4, 75.6, 61.9, 39.9, 33.7. FT-IR: ν (cm^{-1}) 3052, 2959, 2851, 1697, 1598, 1458, 1431, 1399, 1377, 1351, 1310, 1289, 1261, 1233. HRMS [ESI] calcd for $C_{21}H_{18}N_2NaO_3$ $[M+Na]^+$ 369.1210, found 369.1213.

3t-b: 13.8 mg, yield 20%. Yellow solid, m.p. 150-151 °C. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.80-7.72 (m, 3H), 7.70 (s, 1H), 7.50-7.41 (m, 2H), 7.37-7.28 (m, 3H), 7.24-7.16 (m, 2H), 7.06-6.99 (m, 1H), 5.47-5.40 (m, 1H), 5.03-4.95 (m, 1H), 4.82-4.73 (m, 1H), 3.55-3.44 (m, 1H), 3.04-2.93 (m, 1H), 2.08-1.96 (m, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 171.5, 137.1., 137.0, 133.1, 133.1, 129.3, 128.7, 127.8, 127.8, 126.6, 126.4, 125.8, 123.5, 123.4, 75.6, 62.1, 41.3, 34.7. FT-IR: ν (cm^{-1}) 3052, 2959, 2851, 1697, 1598, 1458, 1431, 1399, 1377, 1351, 1310, 1289, 1261, 1233. HRMS [ESI] calcd for $C_{21}H_{18}N_2NaO_3$ $[M+Na]^+$ 369.1210, found 369.1211.

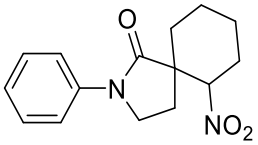


3u: 36.5 mg, yield 78%. Yellow solid, m.p. 70-71 °C. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.66-7.59 (m, 2H), 7.43-7.36 (m, 2H), 7.22-7.16 (m, 1H), 4.74-4.67 (m, 1H), 4.60-4.53 (m, 1H), 3.95-3.78 (m, 2H), 2.46-2.35 (m, 1H), 2.17-2.09 (m, 1H), 1.35 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 173.8, 139.0, 129.0, 125.2, 120.1, 80.7, 45.7, 45.1, 29.0, 20.9. FT-IR: ν (cm^{-1}) 3053, 2954,

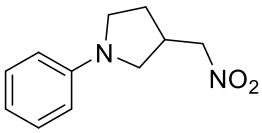
2852, 1687, 1605, 1574, 1543, 1483, 1455, 1436, 1405, 1376, 1311, 1277. HRMS [ESI] calcd for $C_{12}H_{15}N_2O_3$ $[M+H]^+$ 235.1077, found 235.1070.



3v: 34.8 mg, yield 67%. Yellow oil. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.65-7.59 (m, 2H), 7.42-7.35 (m, 2H), 7.21-7.15 (m, 1H), 5.25 (t, *J* = 6.8 Hz, 1H), 3.89-3.75 (m, 2H), 2.49-2.40 (m, 2H), 2.20-2.10 (m, 2H), 2.08-1.95 (m, 4H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 174.4, 139.0, 129.0, 125.1, 120.0, 91.0, 56.8, 45.7, 35.8, 30.1, 27.8, 22.6. FT-IR: ν (cm^{-1}) 2926, 2854, 1697, 1624, 1578, 1544, 1516, 1468, 1431, 1411, 1367, 1309, 1277, 1253, 1222. HRMS [ESI] calcd for $C_{14}H_{16}N_2O_3Na$ $[M+Na]^+$ 283.1053, found 283.1063.

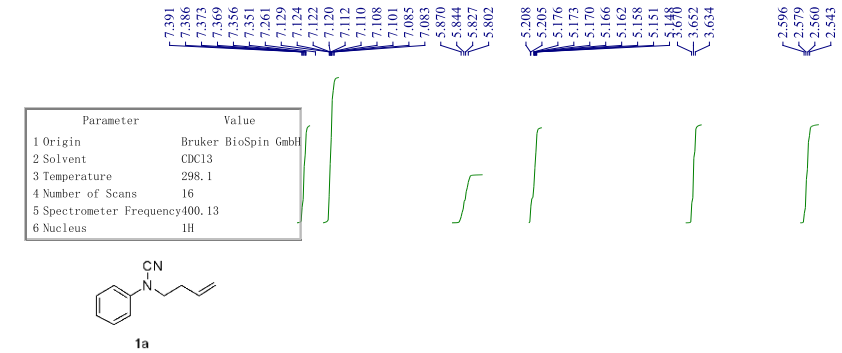


3w: 31.2 mg, yield 57%. Yellow solid, m.p. 57-58 °C. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.64-7.59 (m, 2H), 7.41-7.34 (m, 2H), 7.20-7.14 (m, 1H), 4.83 (dd, *J* = 12.4, 4.0 Hz, 1H), 3.89-3.80 (m, 1H), 3.80-3.72 (m, 1H), 2.53-2.43 (m, 1H), 2.24-2.14 (m, 1H), 2.14-2.05 (m, 1H), 2.00-1.90 (m, 1H), 1.85-1.71 (m, 4H), 1.55-1.44 (m, 1H), 1.41-1.31 (m, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 174.6, 139.2, 128.9, 125.1, 120.5, 86.5, 50.5, 45.3, 34.1, 26.8, 24.0, 23.4, 20.8. FT-IR: ν (cm^{-1}) 2926, 2852, 1699, 1625, 1577, 1545, 1506, 1498, 1441, 1409, 1377, 1319, 1287, 1263, 1214. HRMS [ESI] calcd for $C_{15}H_{18}N_2O_3Na$ $[M+Na]^+$ 297.1210, found 297.1215.



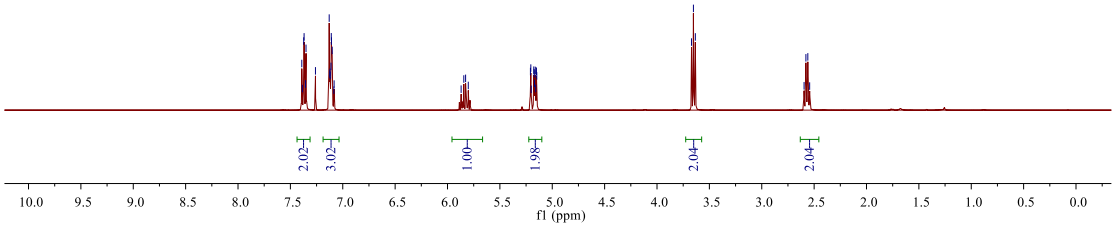
4: 30.9 mg, yield 75%. brown oil. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.27-7.21 (m, 2H), 6.75-6.69 (m, 1H), 6.59-6.54 (m, 2H), 4.47 (d, *J* = 7.2 Hz, 2H), 3.59-3.50 (m, 1H), 3.48-3.40 (m, 1H), 3.39-3.31 (m, 1H), 3.19-3.06 (m, 2H), 2.33-2.22 (m, 1H), 1.90-1.78 (m, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 147.3, 129.3, 116.6, 112.0, 78.1, 51.0, 46.7, 37.0, 28.9. FT-IR: ν (cm^{-1}) 3060, 3031, 2961, 2848, 1733, 1698, 1635, 1598, 1550, 1484, 1430, 1372, 1264. HRMS [ESI] calcd for $C_{11}H_{14}N_2O_2Na$ $[M+Na]^+$ 229.0947, found 229.0948.

7. ^{1}H, ^{13}C, and ^{19}F NMR spectra

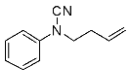


Parameter	Value
1 Origin	Bruker BioSpin GmbH
2 Solvent	CDCl3
3 Temperature	298.1
4 Number of Scans	16
5 Spectrometer Frequency	400.13
6 Nucleus	1H

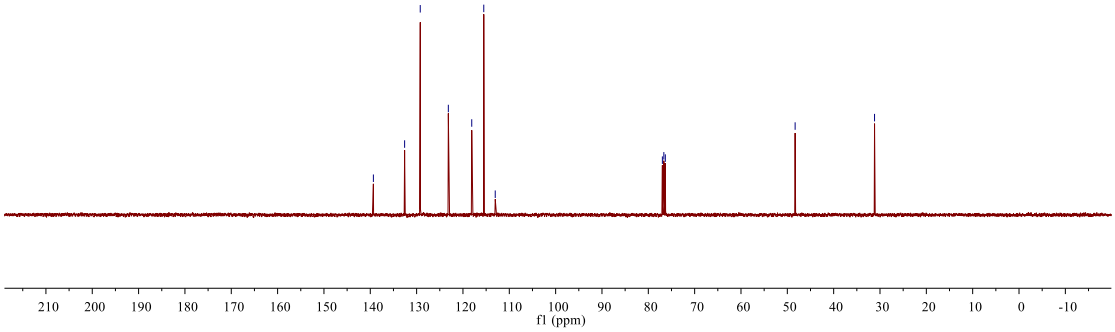
1a

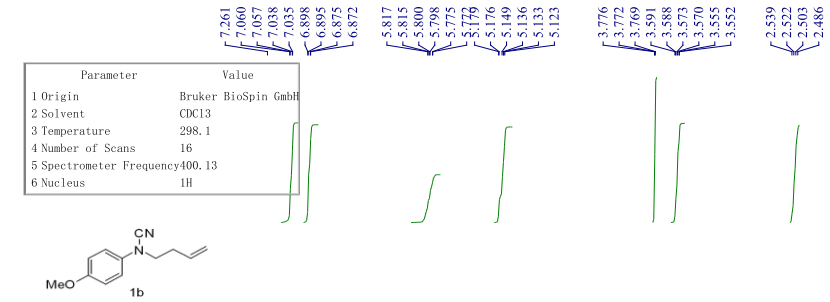


Parameter	Value
1 Origin	Bruker BioSpin GmbH
2 Solvent	CDCl3
3 Temperature	298.1
4 Number of Scans	20
5 Spectrometer Frequency	100.61
6 Nucleus	13C



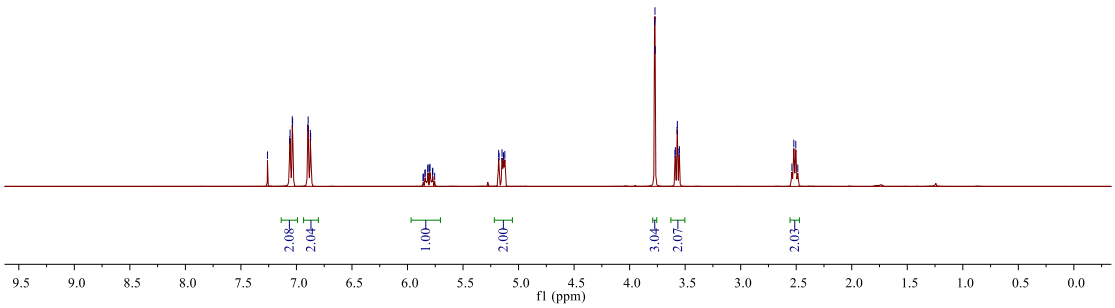
1a



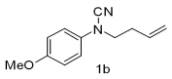


Parameter	Value
1 Origin	Bruker BioSpin GmbH
2 Solvent	CDCl3
3 Temperature	298.1
4 Number of Scans	16
5 Spectrometer Frequency	400.13
6 Nucleus	1H

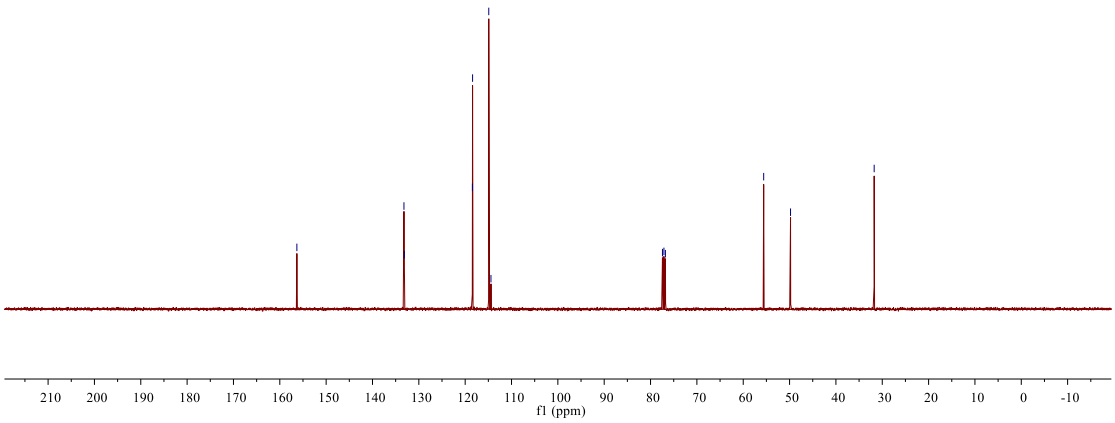
1b

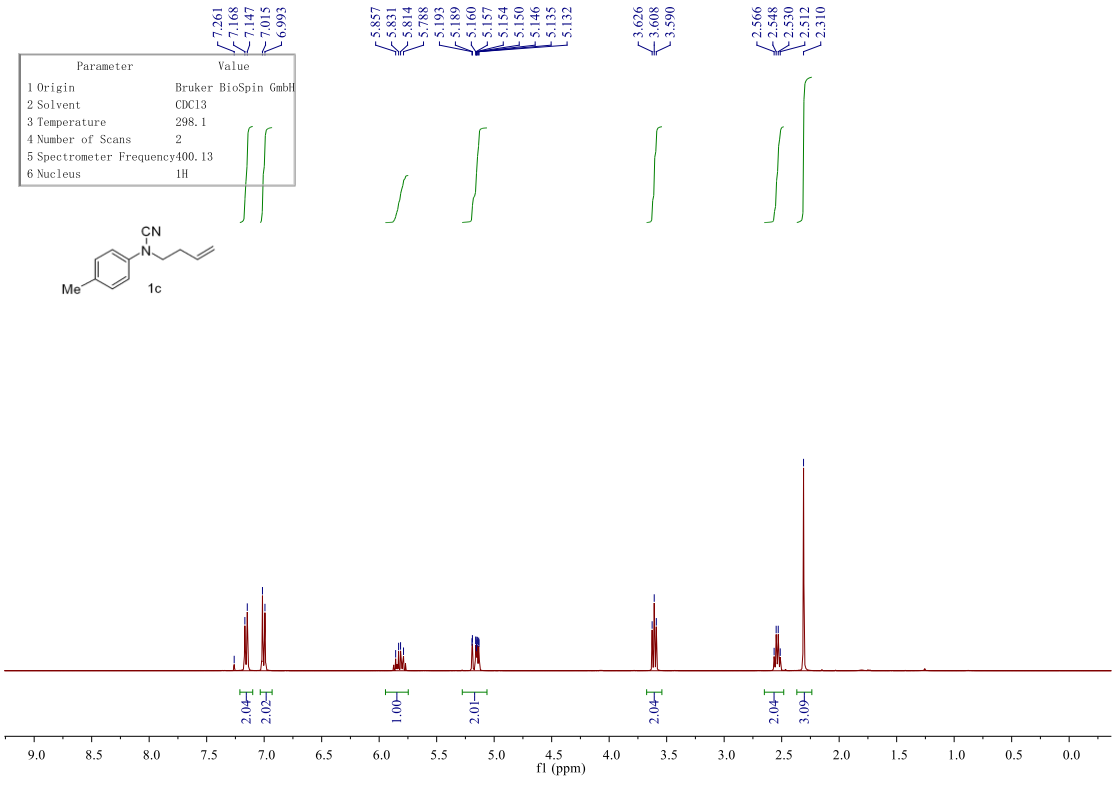


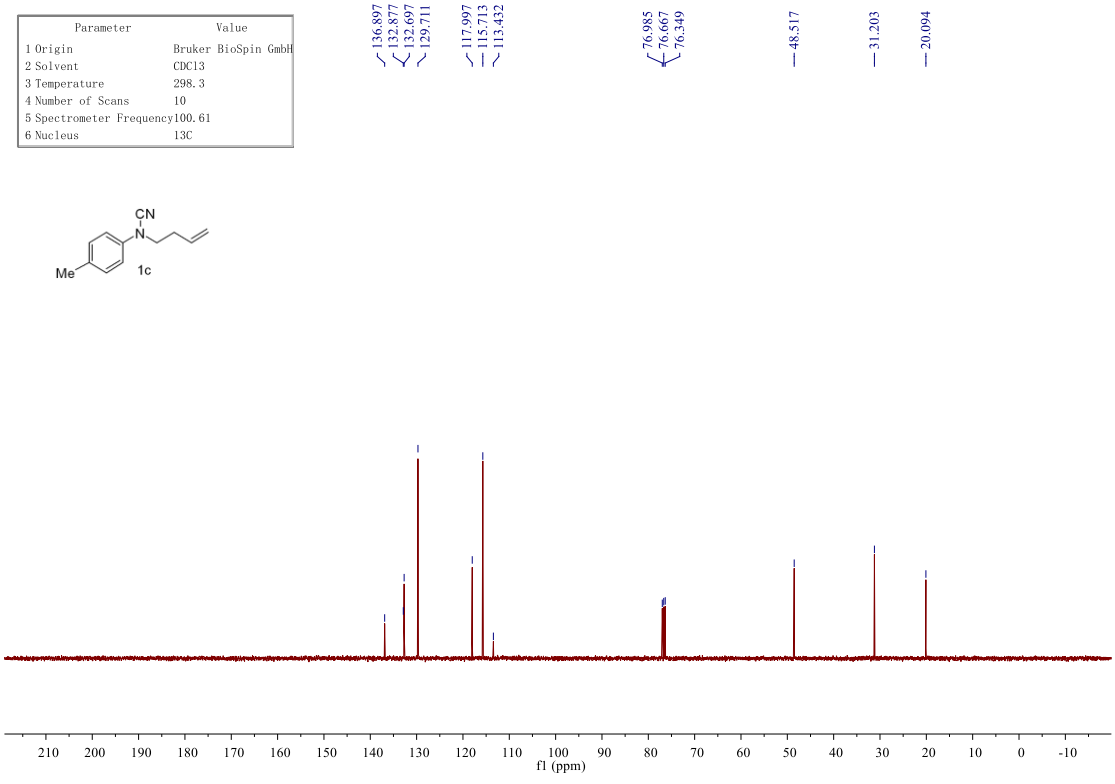
Parameter	Value
1 Origin	Bruker BioSpin GmbH
2 Solvent	CDCl3
3 Temperature	298.2
4 Number of Scans	100
5 Spectrometer Frequency	100.62
6 Nucleus	13C

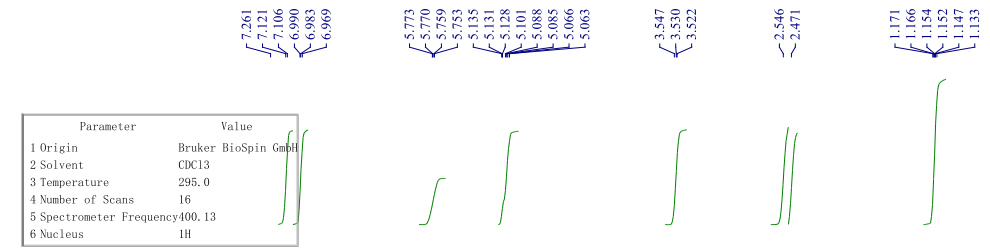


1b

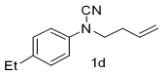



7.261
7.168
7.147
7.015
6.993
5.857
5.831
5.814
5.788
5.193
5.189
5.160
5.157
5.154
5.150
5.146
5.135
5.132
3.626
3.608
3.590
2.566
2.548
2.530
2.512
2.310
Parameter Value
1 Origin Bruker BioSpin GmbH
2 Solvent CDCl3
3 Temperature 298.1
4 Number of Scans 2
5 Spectrometer Frequency 400.13
6 Nucleus 1H
CN
N
Me
1c
2.04
2.02
1.00
2.01
2.04
2.04
3.09
9.0 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
f1 (ppm)

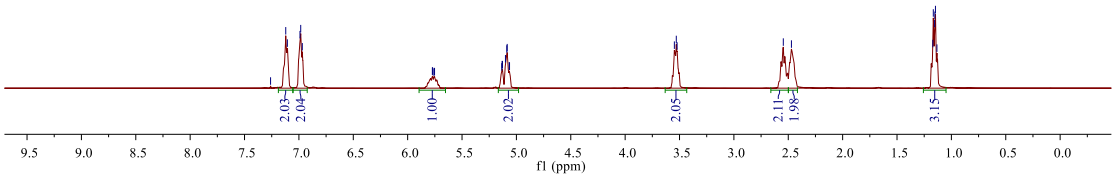

Parameter Value
1 Origin Bruker BioSpin GmbH
2 Solvent CDCl3
3 Temperature 298.3
4 Number of Scans 10
5 Spectrometer Frequency 100.61
6 Nucleus 13C
136.897
132.877
132.697
129.711
117.997
115.713
113.432
76.985
76.667
76.349
48.517
31.203
20.094
CN
N
Me
1c
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)



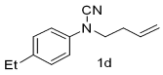
Parameter	Value
1 Origin	Bruker BioSpin GmbH
2 Solvent	CDCl3
3 Temperature	295.0
4 Number of Scans	16
5 Spectrometer Frequency	400.13
6 Nucleus	1H



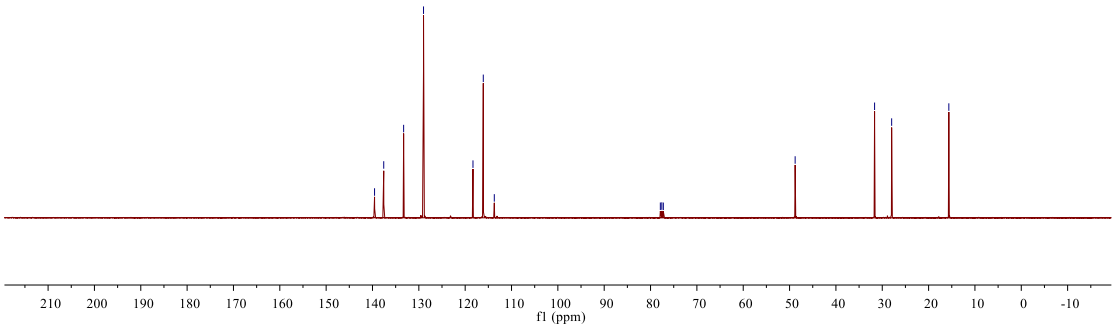
1d

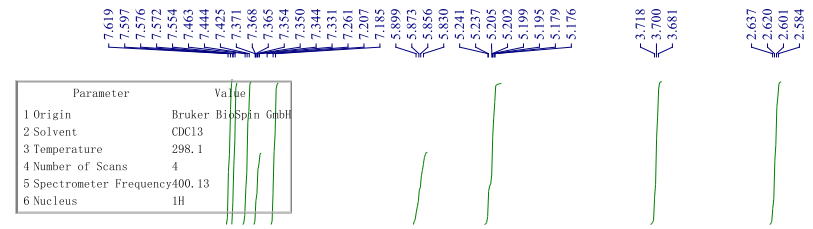


Parameter	Value
1 Origin	Bruker BioSpin GmbH
2 Solvent	CDCl3
3 Temperature	296.0
4 Number of Scans	80
5 Spectrometer Frequency	100.62
6 Nucleus	13C

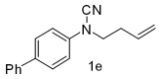


1d

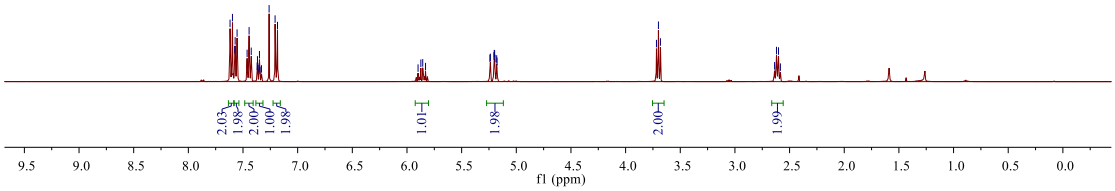




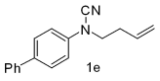
Parameter	Value
1 Origin	Bruker BioSpin GmbH
2 Solvent	CDCl3
3 Temperature	298.1
4 Number of Scans	4
5 Spectrometer Frequency	400.13
6 Nucleus	1H



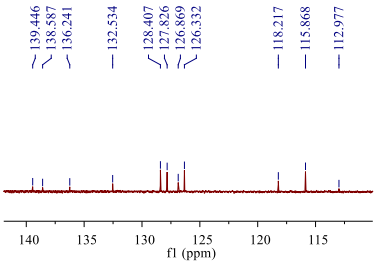
1e

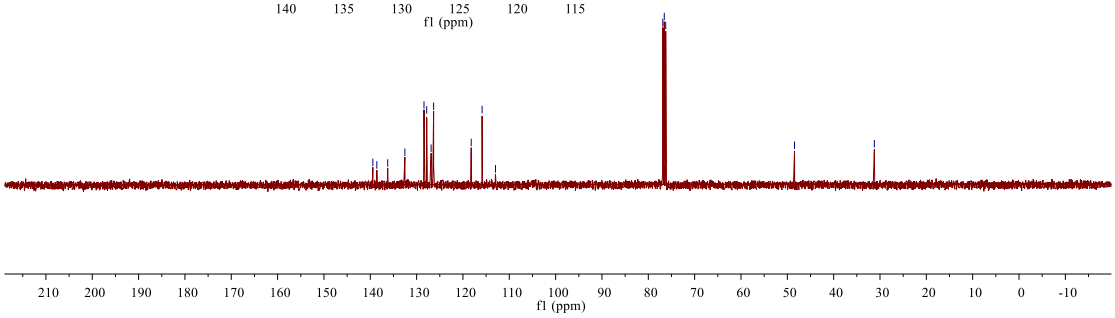


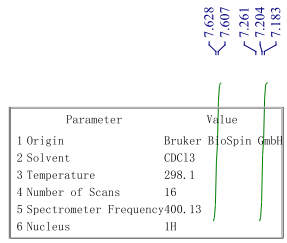
Parameter	Value
1 Origin	Bruker BioSpin GmbH
2 Solvent	CDCl3
3 Temperature	298.2
4 Number of Scans	20
5 Spectrometer Frequency	100.61
6 Nucleus	13C



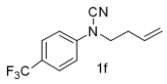
1e



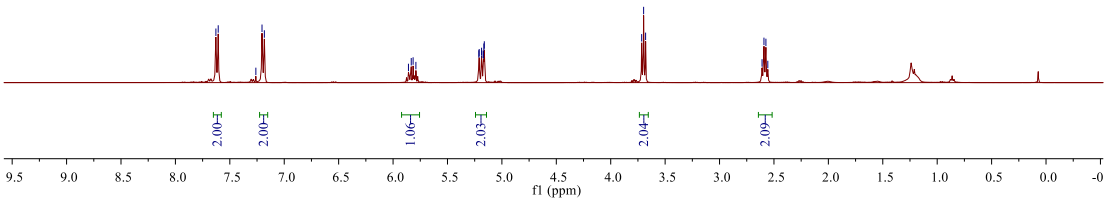




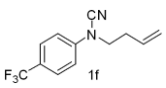
Parameter	Value
1 Origin	Bruker BioSpin GmbH
2 Solvent	CDCl3
3 Temperature	298.1
4 Number of Scans	16
5 Spectrometer Frequency	400.13
6 Nucleus	1H



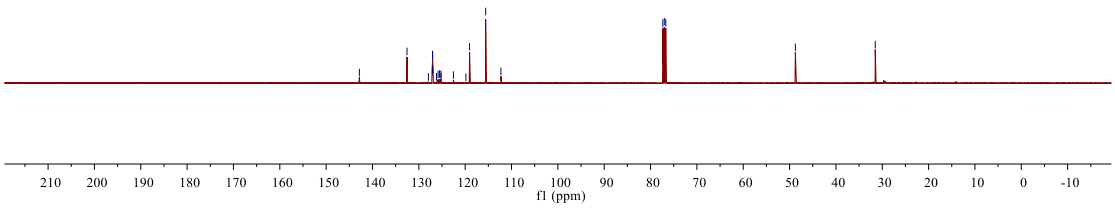
1f



Parameter	Value
1 Origin	Bruker BioSpin GmbH
2 Solvent	CDCl3
3 Temperature	298.1
4 Number of Scans	1024
5 Spectrometer Frequency	100.62
6 Nucleus	13C

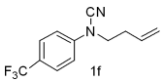


1f

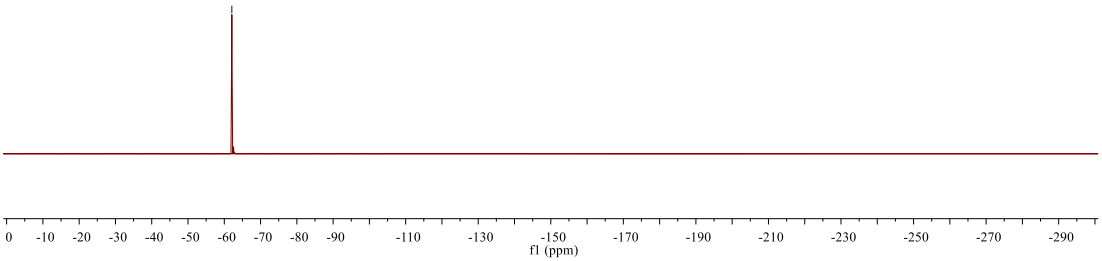


-62.08

Parameter	Value
1 Origin	Bruker BioSpin GmbH
2 Solvent	CDCl3
3 Temperature	298.2
4 Number of Scans	16
5 Spectrometer Frequency	376.44
6 Nucleus	19F

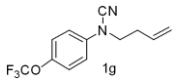


1f

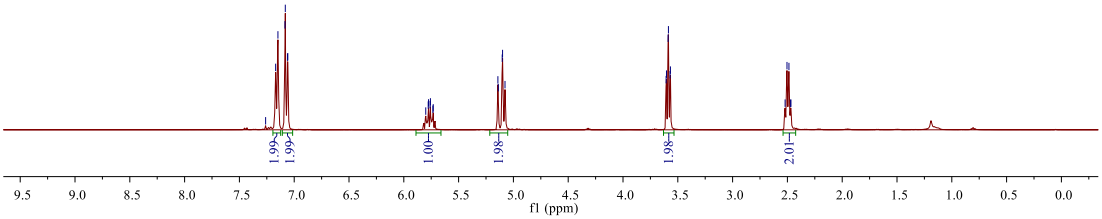


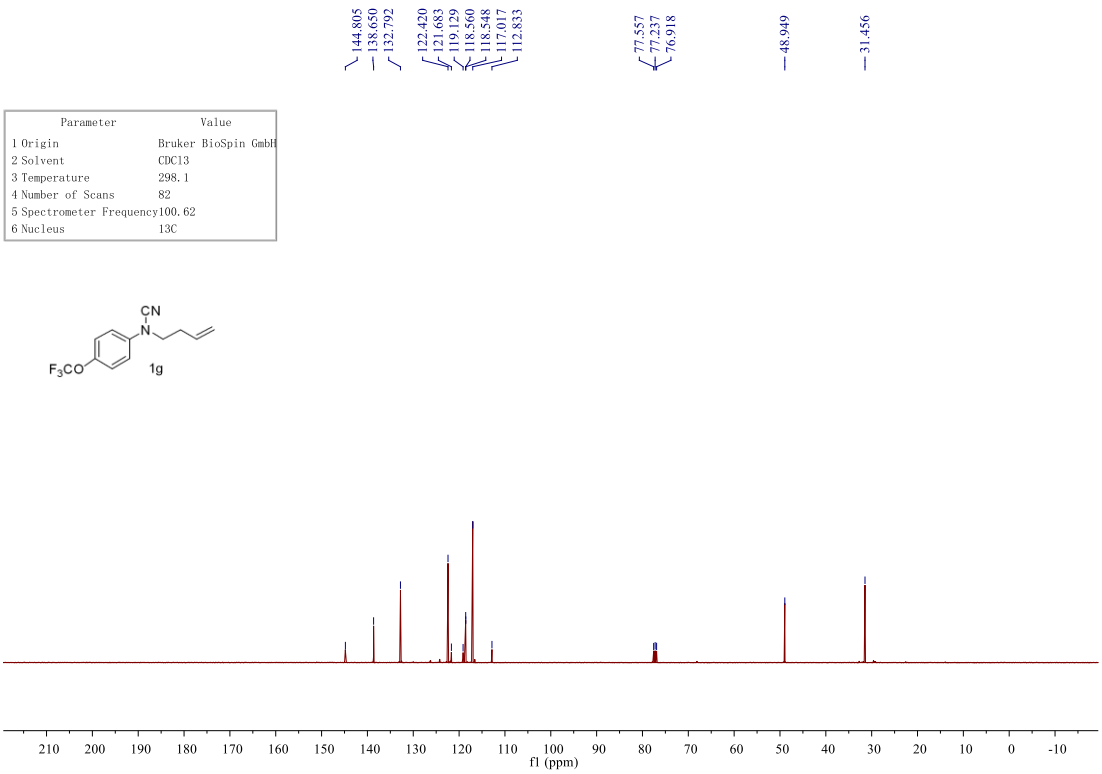
7.261 7.169 7.148 7.084 7.081 7.061 7.058 5.800 5.777 5.774 5.760 5.757 5.735 5.732 5.141 5.138 5.102 5.099 5.077 3.607 3.603 3.588 3.586 3.571 3.567 2.521 2.504 2.486 2.471 2.468

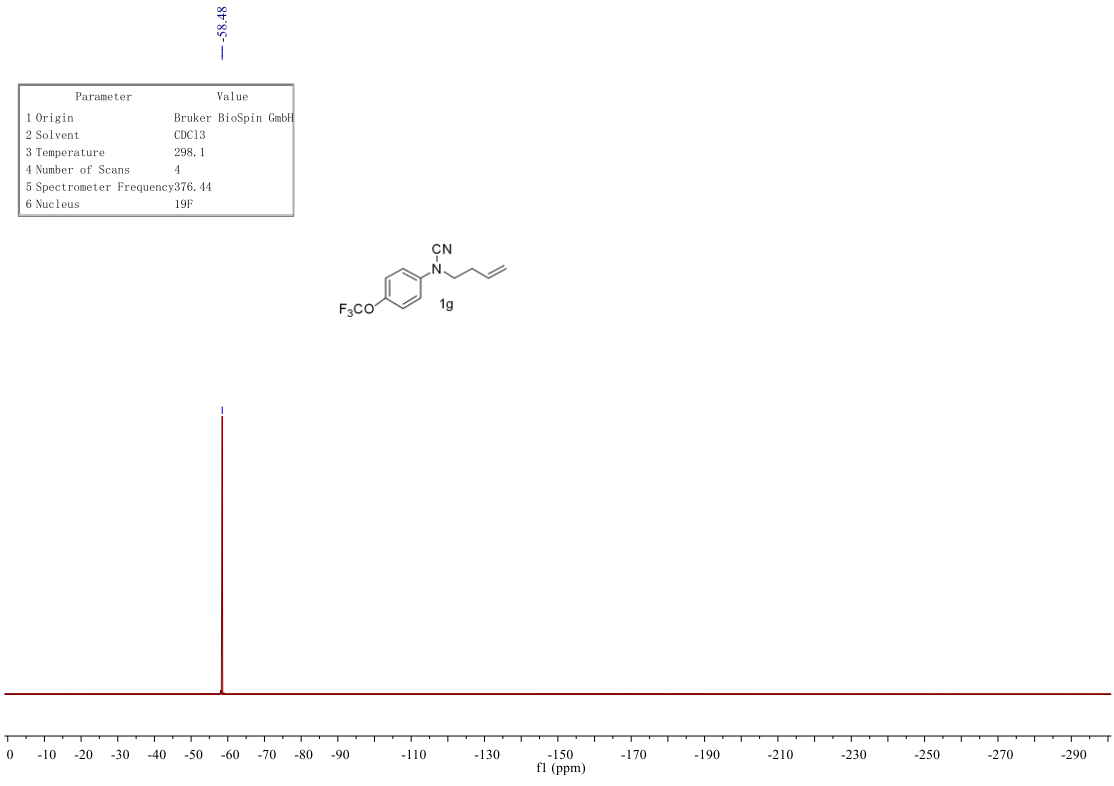
Parameter	Value
1 Origin	Bruker BioSpin GmbH
2 Solvent	CDCl3
3 Temperature	298.1
4 Number of Scans	16
5 Spectrometer Frequency	400.13
6 Nucleus	1H

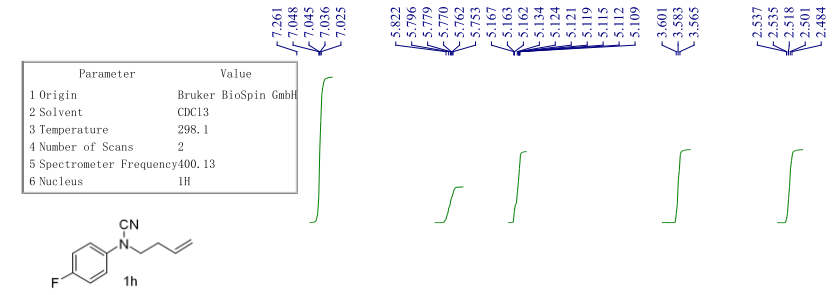


1g



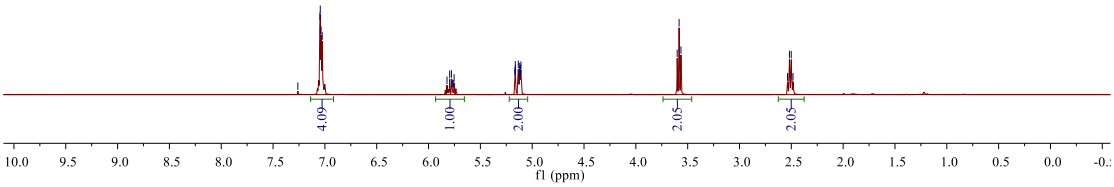

144.805
138.650
132.792
122.420
121.683
119.129
118.560
118.548
117.017
112.833
77.557
77.237
76.918
48.949
31.456
Parameter Value
1 Origin Bruker BioSpin GmbH
2 Solvent CDCl3
3 Temperature 298.1
4 Number of Scans 82
5 Spectrometer Frequency 100.62
6 Nucleus 13C
CN
N
F3CO
1g
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)


-58.48
Parameter Value
1 Origin Bruker BioSpin GmbH
2 Solvent CDCl3
3 Temperature 298.1
4 Number of Scans 4
5 Spectrometer Frequency 376.44
6 Nucleus 19F
CN
N
F3CO
1g
0 -10 -20 -30 -40 -50 -60 -70 -80 -90 -110 -130 -150 -170 -190 -210 -230 -250 -270 -290
f1 (ppm)

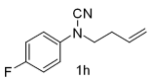


Parameter	Value
1 Origin	Bruker BioSpin GmbH
2 Solvent	CDCl3
3 Temperature	298.1
4 Number of Scans	2
5 Spectrometer Frequency	400.13
6 Nucleus	1H

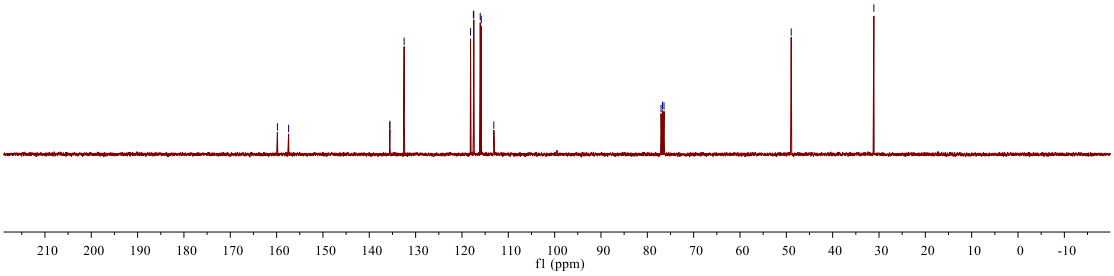
1h



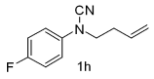
Parameter	Value
1 Origin	Bruker BioSpin GmbH
2 Solvent	CDCl3
3 Temperature	298.3
4 Number of Scans	11
5 Spectrometer Frequency	100.61
6 Nucleus	13C



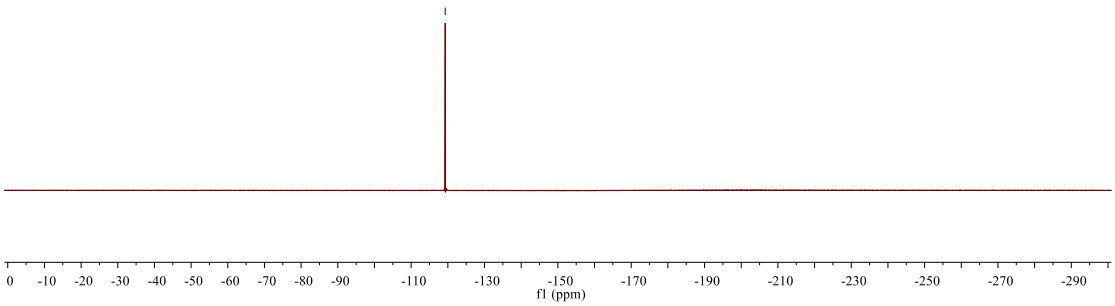
1h



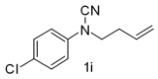
Parameter	Value
1 Origin	Bruker BioSpin GmbH
2 Solvent	CDCl3
3 Temperature	298.2
4 Number of Scans	2
5 Spectrometer Frequency	376.44
6 Nucleus	19F



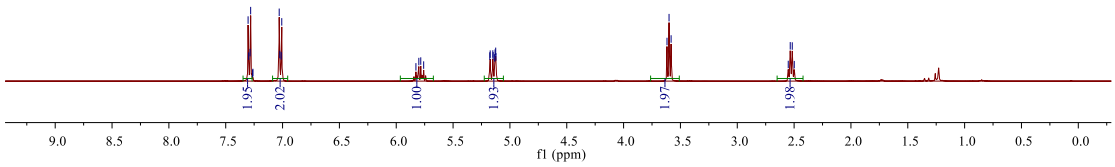
1h



Parameter	Value
1 Origin	Bruker BioSpin GmbH
2 Solvent	CDCl3
3 Temperature	298.2
4 Number of Scans	16
5 Spectrometer Frequency	400.13
6 Nucleus	1H

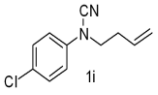


1i

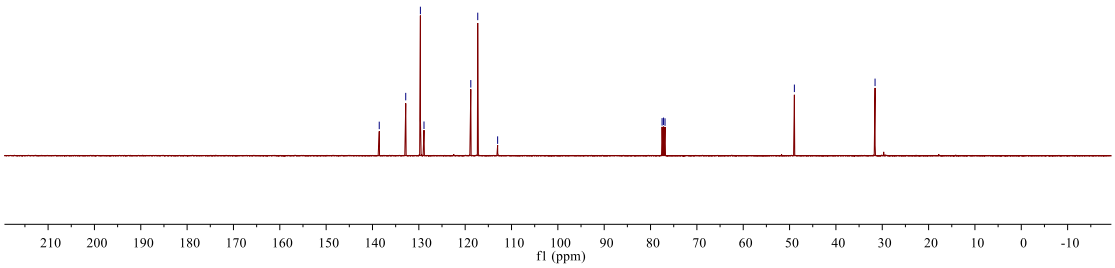


Parameter	Value
1 Origin	Bruker BioSpin GmbH
2 Solvent	CDCl3
3 Temperature	298.2
4 Number of Scans	80
5 Spectrometer Frequency	100.62
6 Nucleus	13C

138.542, 132.826, 129.664, 128.884, 118.786, 117.287, 112.995, 77.510, 77.192, 76.874, 48.964, 31.552

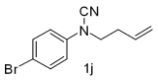


1i

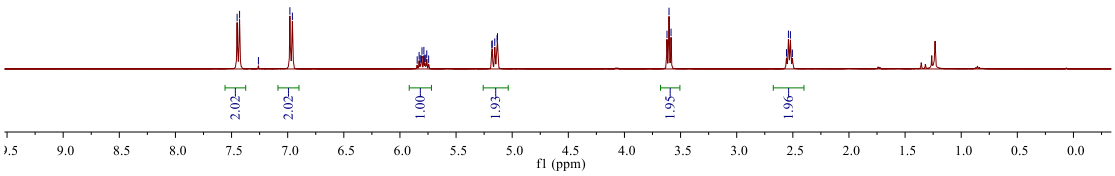


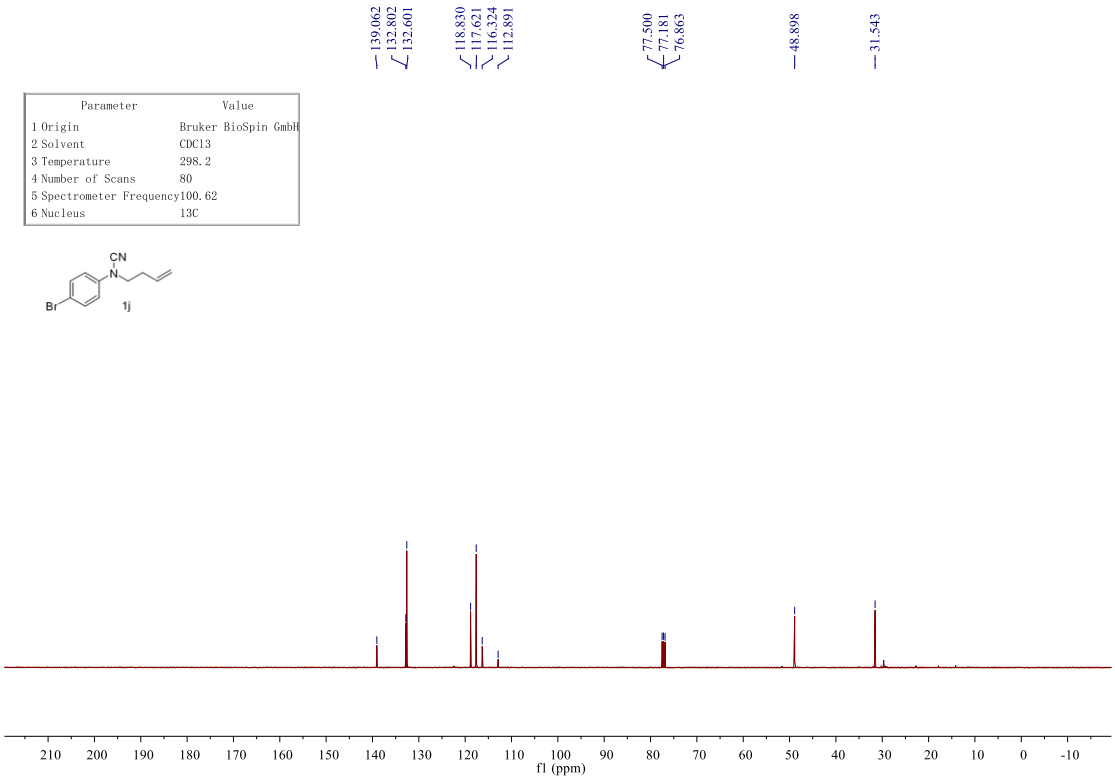
7.451, 7.429, 7.261, 6.980, 6.958, 5.829, 5.821, 5.812, 5.804, 5.787, 5.778, 5.770, 5.761, 5.744, 5.181, 5.178, 5.155, 5.135, 5.131, 3.621, 3.603, 3.585, 2.557, 2.539, 2.521, 2.504

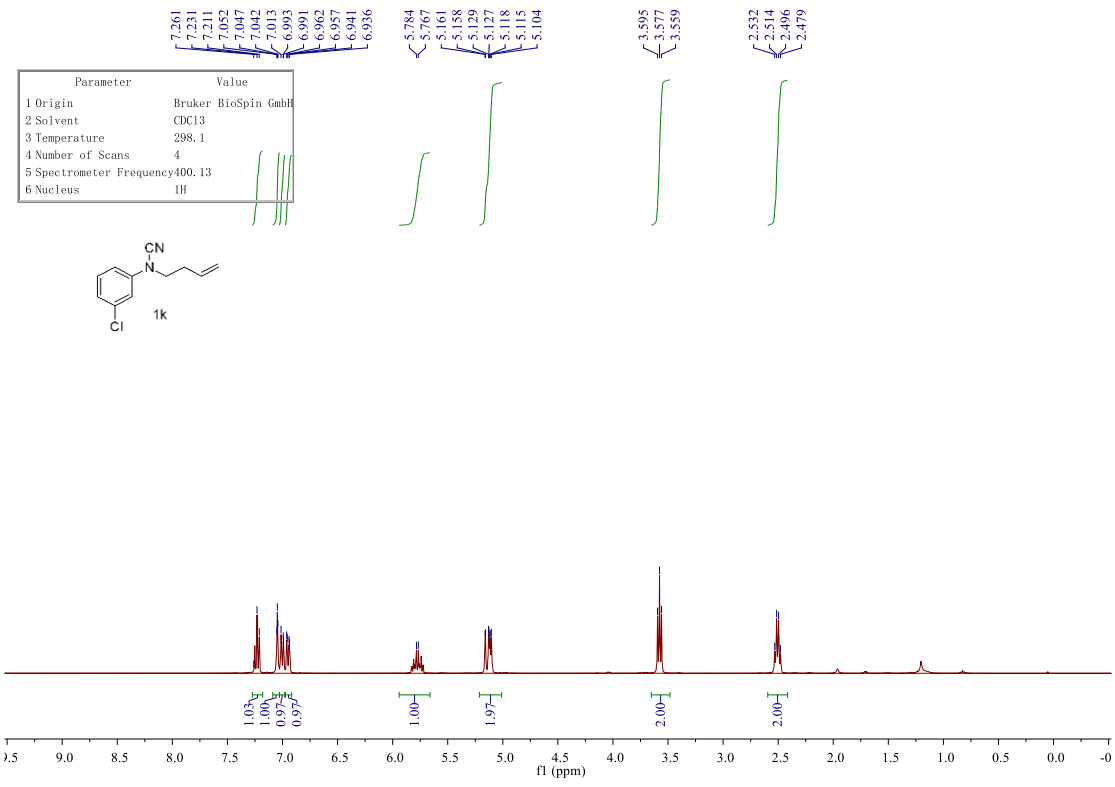
Parameter	Value
1 Origin	Bruker BioSpin GmbH
2 Solvent	CDCl3
3 Temperature	298.2
4 Number of Scans	16
5 Spectrometer Frequency	400.13
6 Nucleus	1H



1j

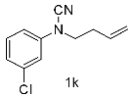



139.062
132.802
132.601
118.830
117.621
116.324
112.891
77.500
77.181
76.863
48.898
31.543
Parameter Value
1 Origin Bruker BioSpin GmbH
2 Solvent CDCl3
3 Temperature 298.2
4 Number of Scans 80
5 Spectrometer Frequency 100.62
6 Nucleus 13C
CN
N
Br
1j
f1 (ppm)

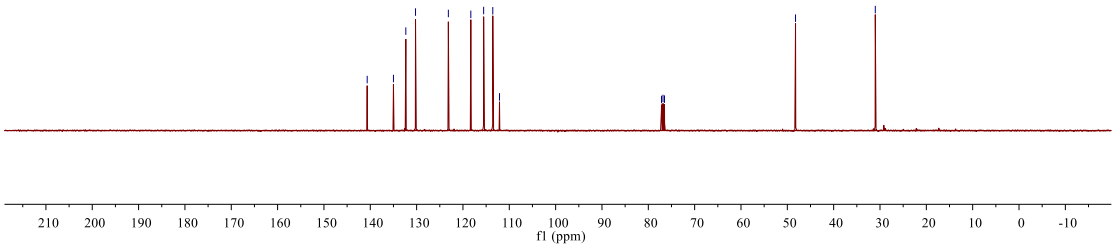

7.261
7.231
7.211
7.052
7.047
7.042
7.013
6.993
6.991
6.962
6.957
6.941
6.936
5.784
5.767
5.161
5.158
5.129
5.127
5.118
5.115
5.104
3.595
3.577
3.559
2.532
2.514
2.496
2.479
Parameter Value
1 Origin Bruker BioSpin GmbH
2 Solvent CDCl3
3 Temperature 298.1
4 Number of Scans 4
5 Spectrometer Frequency 400.13
6 Nucleus 1H
CN
N
Cl
1k
1.03
1.00
0.95
0.97
1.00
1.97
2.00
2.00
f1 (ppm)

Parameter	Value
1 Origin	Bruker BioSpin GmbH
2 Solvent	CDCl3
3 Temperature	298.1
4 Number of Scans	40
5 Spectrometer Frequency	100.61
6 Nucleus	13C

140.670
134.999
132.331
130.236
123.152
118.304
115.533
113.562
112.111
77.147
76.828
76.509
48.229
31.018

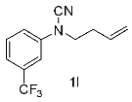


1k

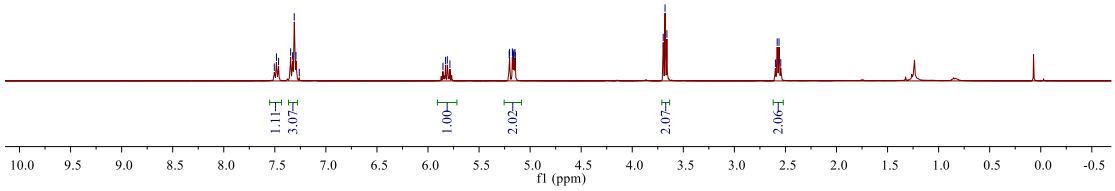


7.505
7.484
7.463
7.345
7.326
7.310
7.293
7.261
5.854
5.828
5.811
5.785
5.205
5.201
5.174
5.171
5.162
5.158
5.148
5.145
3.697
3.679
3.661
2.598
2.581
2.563
2.546

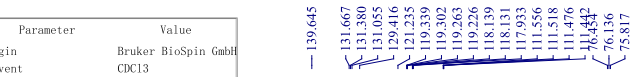
Parameter	Value
1 Origin	Bruker BioSpin GmbH
2 Solvent	CDCl3
3 Temperature	298.2
4 Number of Scans	2
5 Spectrometer Frequency	400.13
6 Nucleus	1H

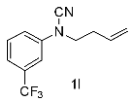


1l

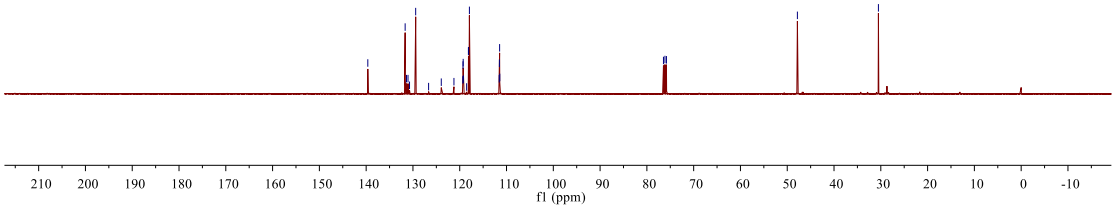


Parameter	Value
1 Origin	Bruker BioSpin GmbH
2 Solvent	CDCl3
3 Temperature	298.2
4 Number of Scans	109
5 Spectrometer Frequency	100.62
6 Nucleus	13C

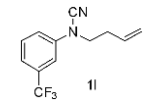




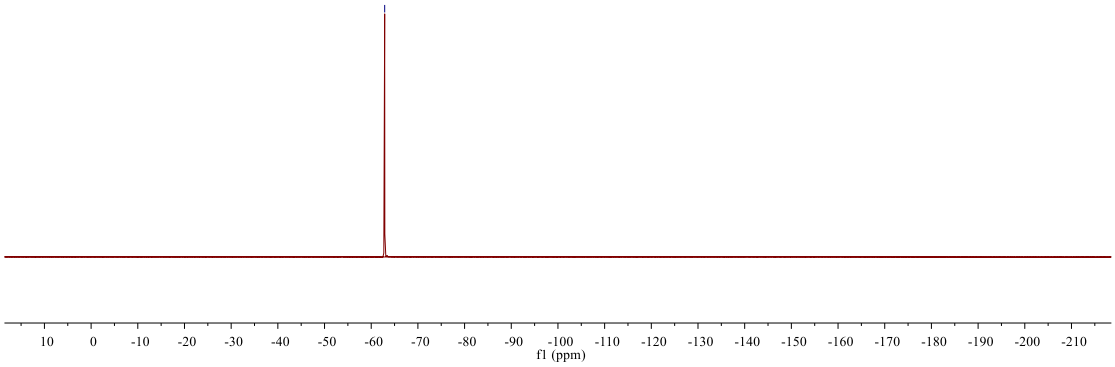
1l

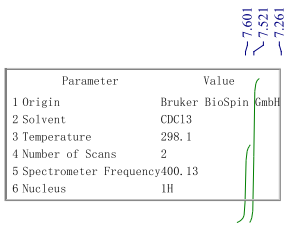


Parameter	Value
1 Origin	Bruker BioSpin GmbH
2 Solvent	CDCl3
3 Temperature	298.2
4 Number of Scans	2
5 Spectrometer Frequency	376.50
6 Nucleus	19F

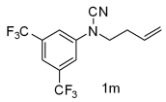


1l

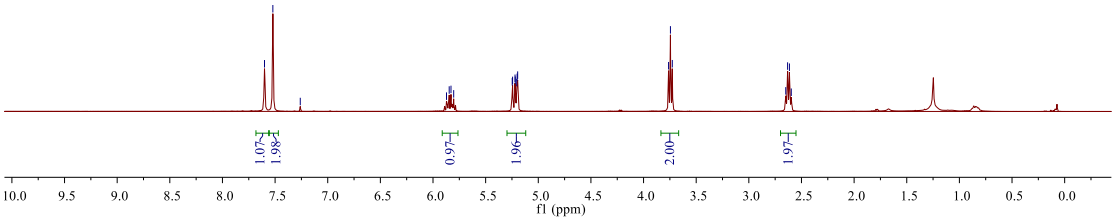




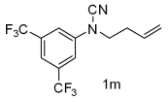
Parameter	Value
1 Origin	Bruker BioSpin GmbH
2 Solvent	CDCl3
3 Temperature	298.1
4 Number of Scans	2
5 Spectrometer Frequency	400.13
6 Nucleus	1H



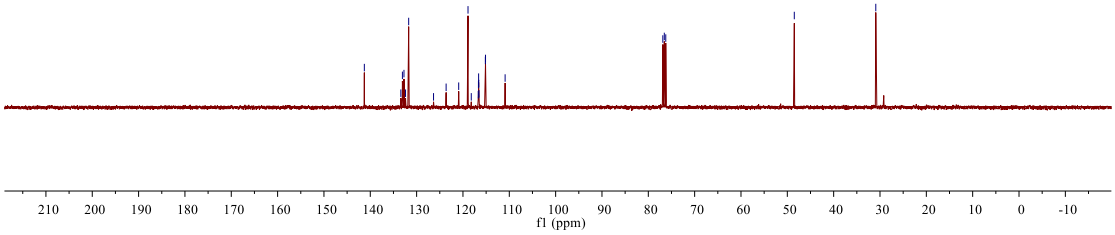
1m

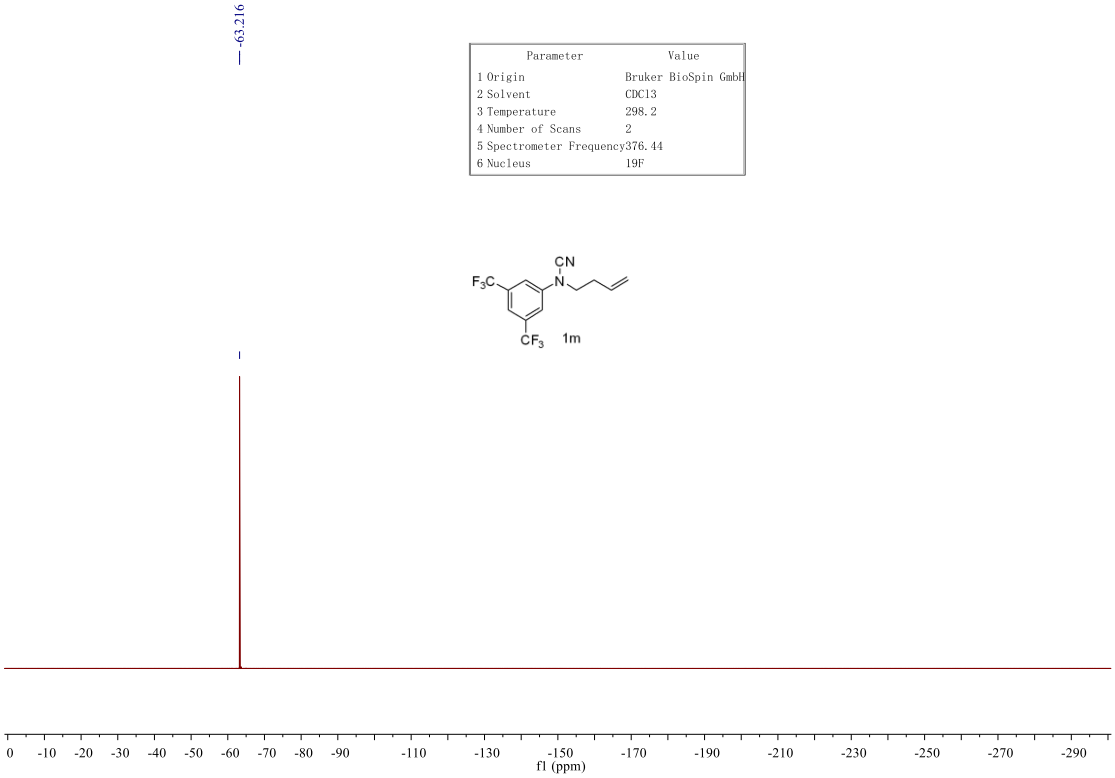


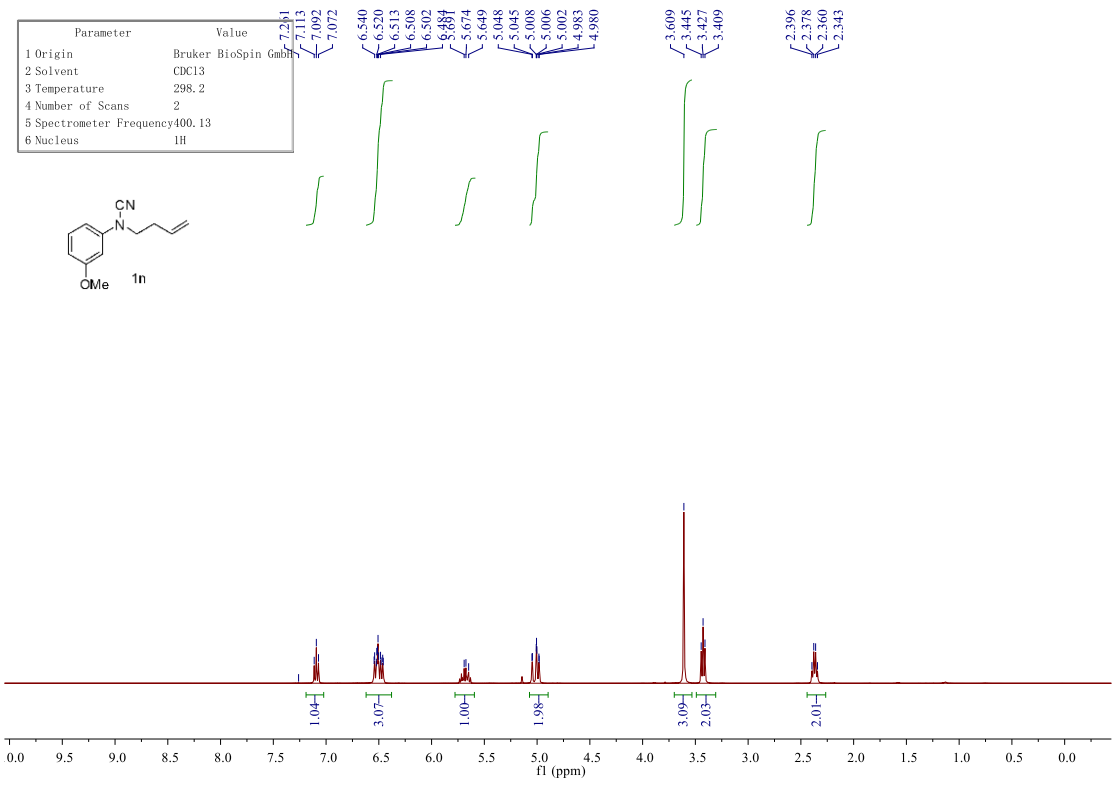
Parameter	Value
1 Origin	Bruker BioSpin GmbH
2 Solvent	CDCl3
3 Temperature	298.1
4 Number of Scans	28
5 Spectrometer Frequency	100.61
6 Nucleus	13C



1m

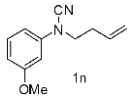



-63.216
Parameter Value
1 Origin Bruker BioSpin GmbH
2 Solvent CDCl3
3 Temperature 298.2
4 Number of Scans 2
5 Spectrometer Frequency 376.44
6 Nucleus 19F
CN
N
F3C
CF3
1m
0 -10 -20 -30 -40 -50 -60 -70 -80 -90 -110 -130 -150 -170 -190 -210 -230 -250 -270 -290
f1 (ppm)

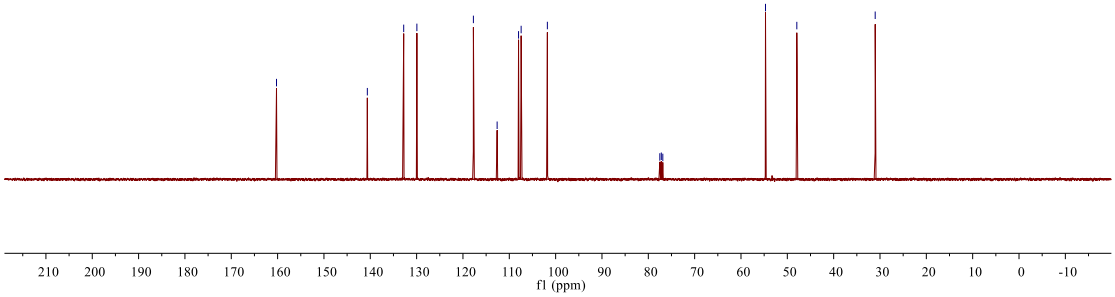

Parameter Value
1 Origin Bruker BioSpin GmbH
2 Solvent CDCl3
3 Temperature 298.2
4 Number of Scans 2
5 Spectrometer Frequency 400.13
6 Nucleus 1H
7.261 7.113 7.092 7.072 6.540 6.520 6.513 6.508 6.502 5.674 5.649 5.048 5.045 5.008 5.006 5.002 4.983 4.980 3.609 3.445 3.427 3.409 2.396 2.378 2.360 2.343
CN
N
OMe
1n
1.04 3.07 1.00 1.98 3.09 2.03 2.01
0.0 9.5 9.0 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
f1 (ppm)

Parameter	Value
1 Origin	Bruker BioSpin GmbH
2 Solvent	CDCl3
3 Temperature	298.3
4 Number of Scans	4
5 Spectrometer Frequency	100.61
6 Nucleus	13C

160.238 140.637 132.789 129.945 117.748 112.625 108.002 107.445 101.784 77.514 77.193 76.871 54.710 47.950 31.037

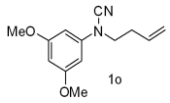


1n



7.261 6.244 6.239 6.233 6.200 6.176 6.171 6.165 5.804 5.779 5.774 5.190 5.151 5.148 5.131 5.086 3.770 3.763 3.757 3.748 3.741 3.735 3.727 3.719 3.713 3.699 3.686 3.570 2.548 2.530 2.514 2.485

Parameter	Value
1 Origin	Bruker BioSpin GmbH
2 Solvent	CDCl3
3 Temperature	298.2
4 Number of Scans	2
5 Spectrometer Frequency	400.13
6 Nucleus	1H



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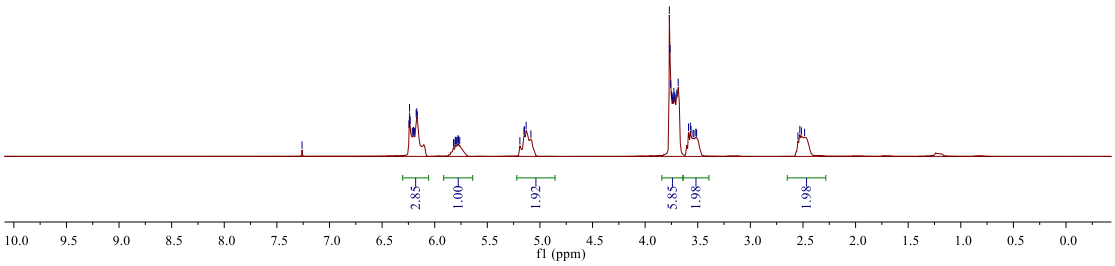
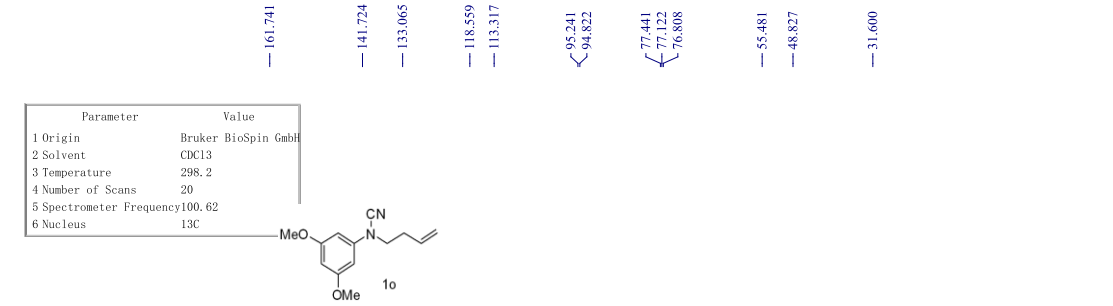
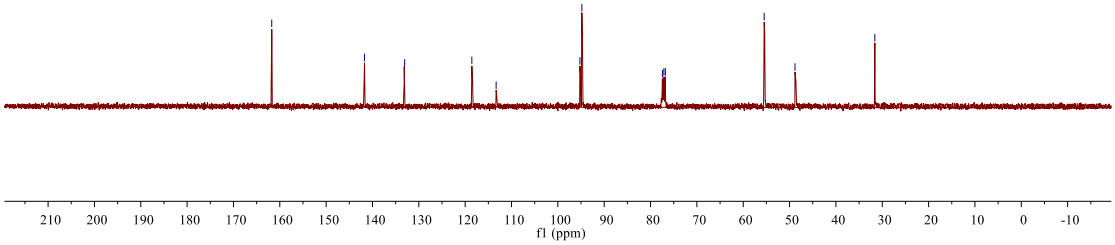


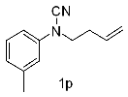


Parameter	Value
1 Origin	Bruker BioSpin GmbH
2 Solvent	CDCl3
3 Temperature	298.2
4 Number of Scans	20
5 Spectrometer Frequency	100.62
6 Nucleus	13C

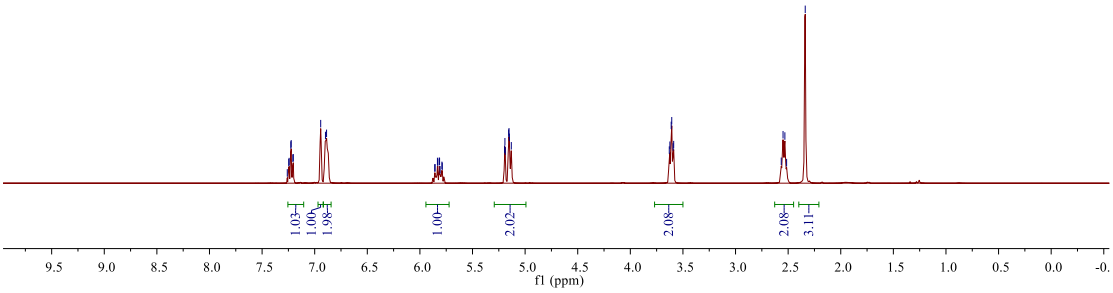
1o



Parameter	Value
1 Origin	Bruker BioSpin GmbH
2 Solvent	CDCl3
3 Temperature	298.1
4 Number of Scans	16
5 Spectrometer Frequency	400.15
6 Nucleus	1H

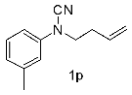


1p



Parameter	Value
1 Origin	Bruker BioSpin GmbH
2 Solvent	CDCl3
3 Temperature	298.1
4 Number of Scans	80
5 Spectrometer Frequency	100.62
6 Nucleus	13C

139.842
139.791
133.192
129.486
124.495
118.493
116.786
113.651
112.965
77.589
77.275
76.956
48.737
31.692
21.553



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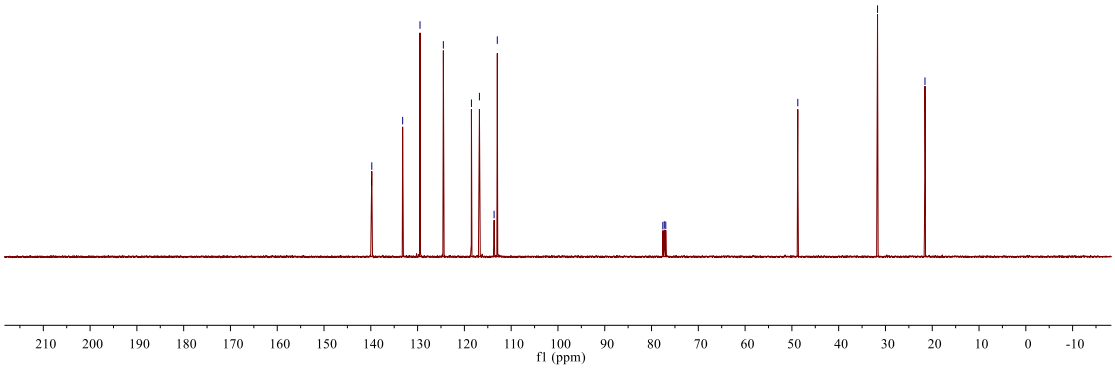
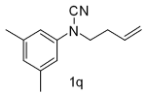
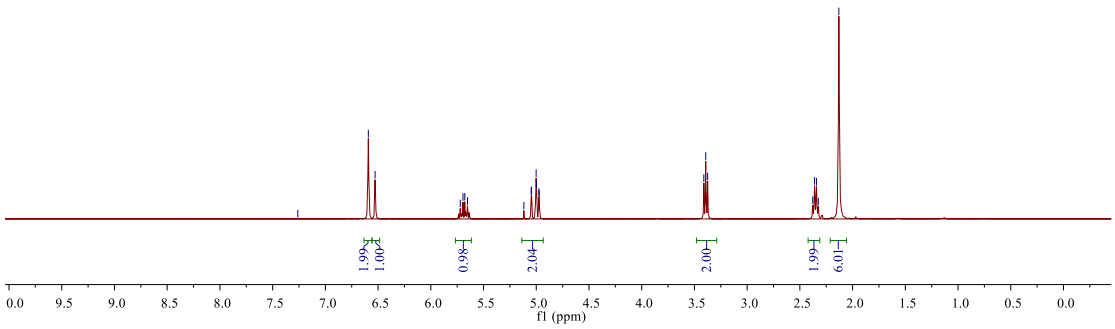


7.261
6.592
6.528
5.721
5.695
5.678
5.652
5.118
5.048
5.044
5.001
4.998
4.975
4.972
3.411
3.393
3.375
2.378
2.361
2.343
2.325
2.130

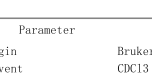
Parameter	Value
1 Origin	Bruker BioSpin GmbH
2 Solvent	CDCl3
3 Temperature	298.1
4 Number of Scans	2
5 Spectrometer Frequency	400.13
6 Nucleus	1H

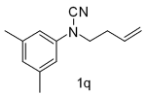


1q

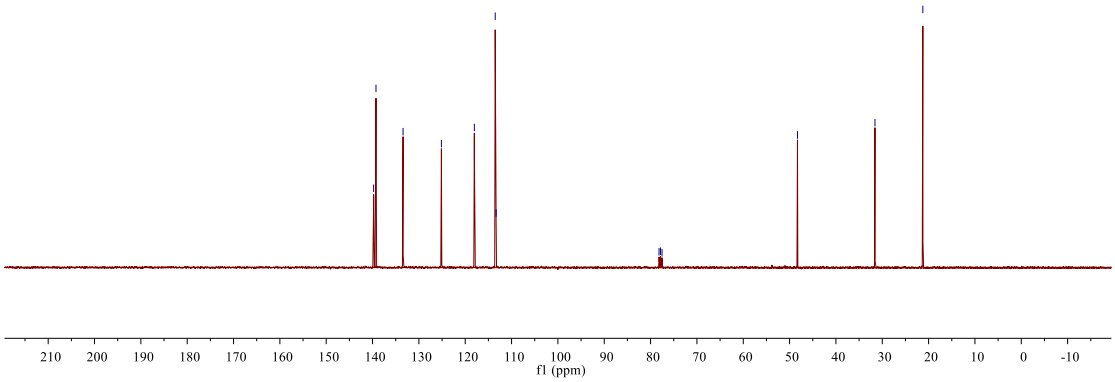


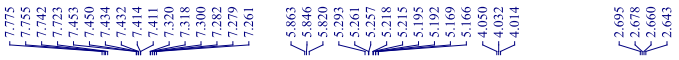
Parameter	Value
1 Origin	Bruker BioSpin GmbH
2 Solvent	CDCl3
3 Temperature	298.3
4 Number of Scans	4
5 Spectrometer Frequency	100.61
6 Nucleus	13C



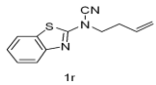


1q

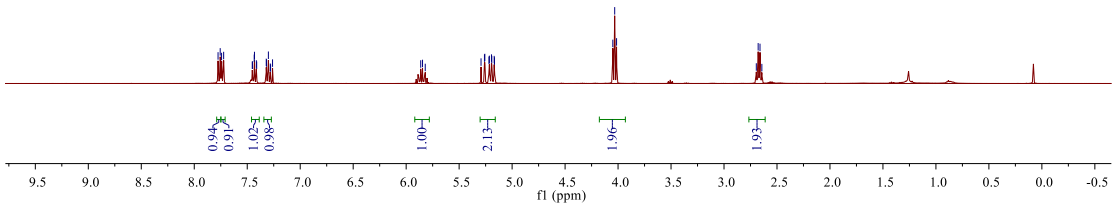


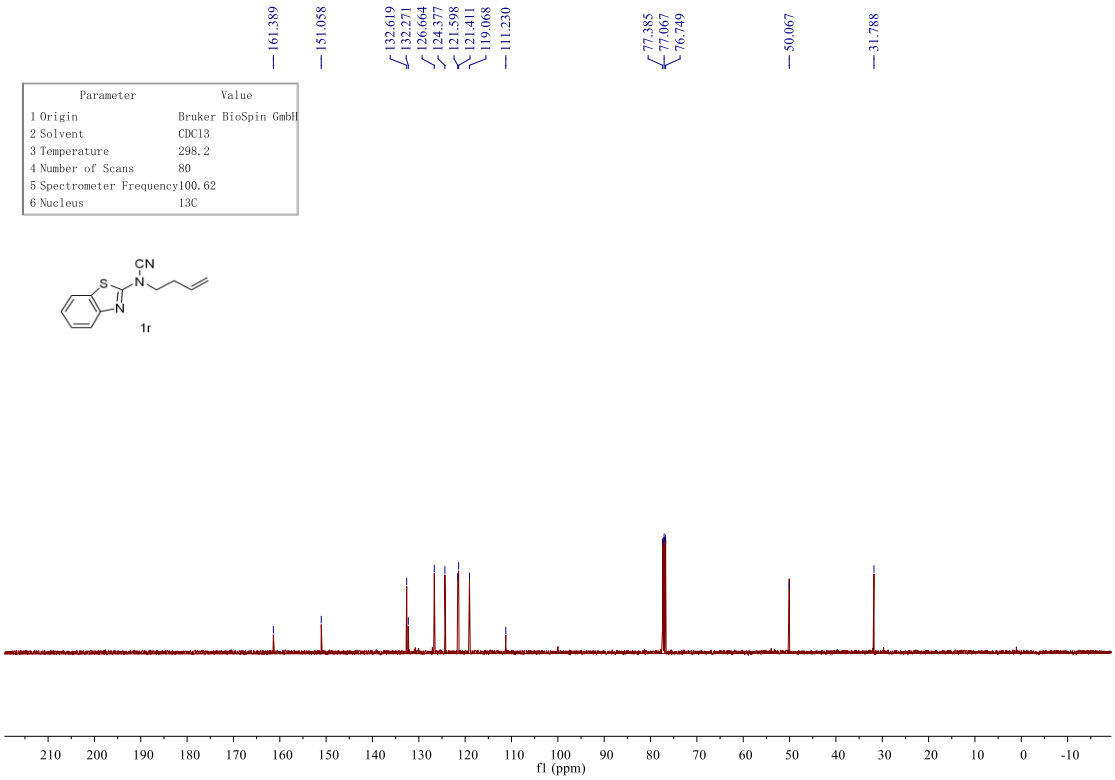


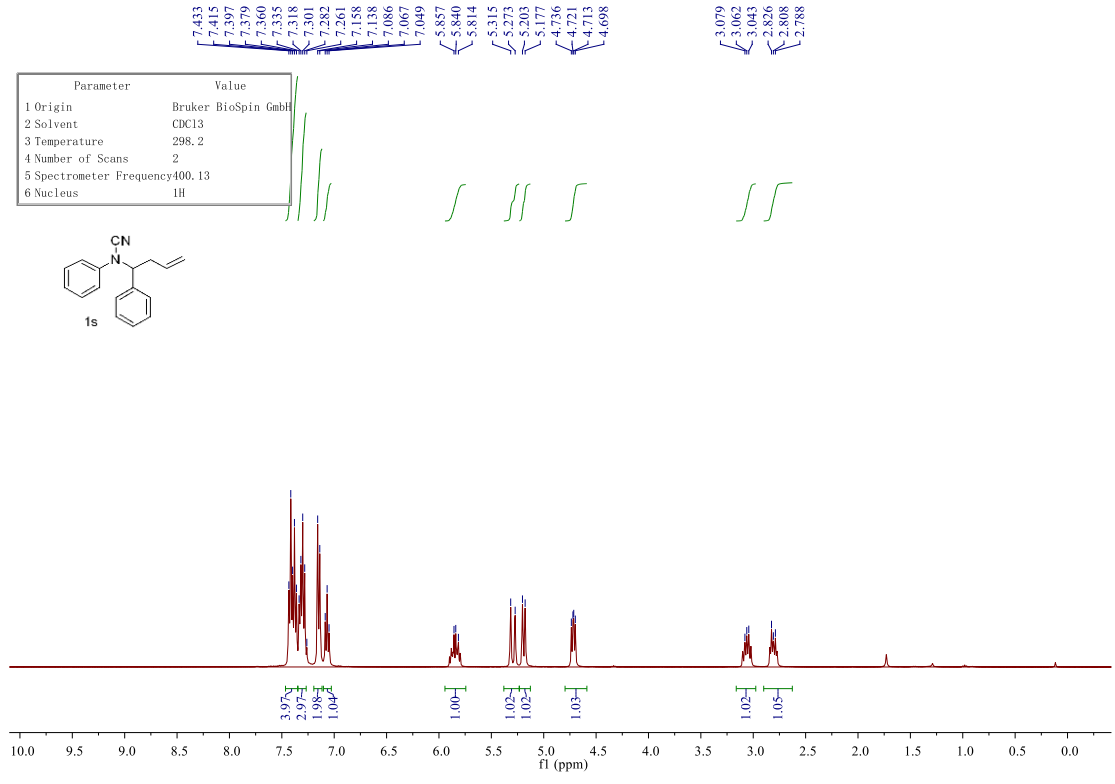
Parameter	Value
1 Origin	Bruker BioSpin GmbH
2 Solvent	CDCl3
3 Temperature	298.1
4 Number of Scans	4
5 Spectrometer Frequency	400.13
6 Nucleus	1H



1r




161.389
151.058
132.619
132.271
126.664
124.377
121.598
121.411
119.068
111.230
77.385
77.067
76.749
50.067
31.788
Parameter Value
1 Origin Bruker BioSpin GmbH
2 Solvent CDCl3
3 Temperature 298.2
4 Number of Scans 80
5 Spectrometer Frequency 100.62
6 Nucleus 13C
CN
S
N
N
1r
f1 (ppm)


7.433
7.415
7.397
7.379
7.360
7.335
7.318
7.301
7.282
7.261
7.158
7.138
7.086
7.067
7.049
5.857
5.840
5.814
5.315
5.273
5.203
5.177
4.736
4.721
4.713
4.698
3.079
3.062
3.043
2.826
2.808
2.788
Parameter Value
1 Origin Bruker BioSpin GmbH
2 Solvent CDCl3
3 Temperature 298.2
4 Number of Scans 2
5 Spectrometer Frequency 400.13
6 Nucleus 1H
CN
N
1s
3.97
2.97
1.98
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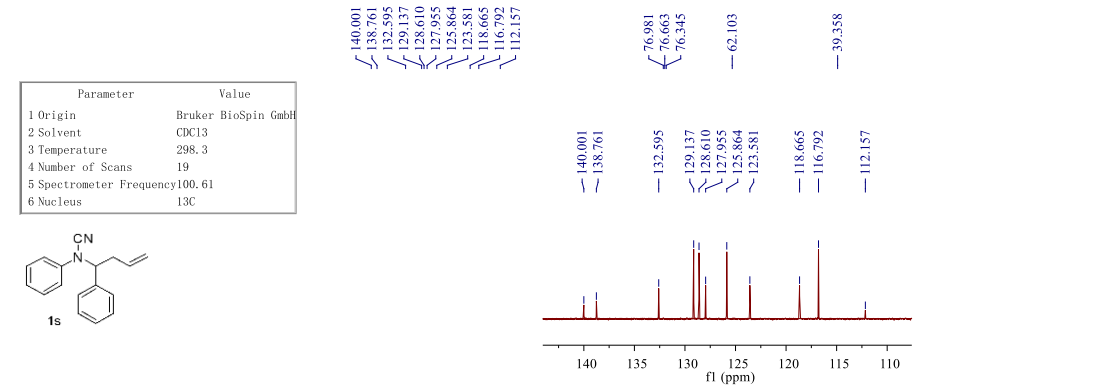
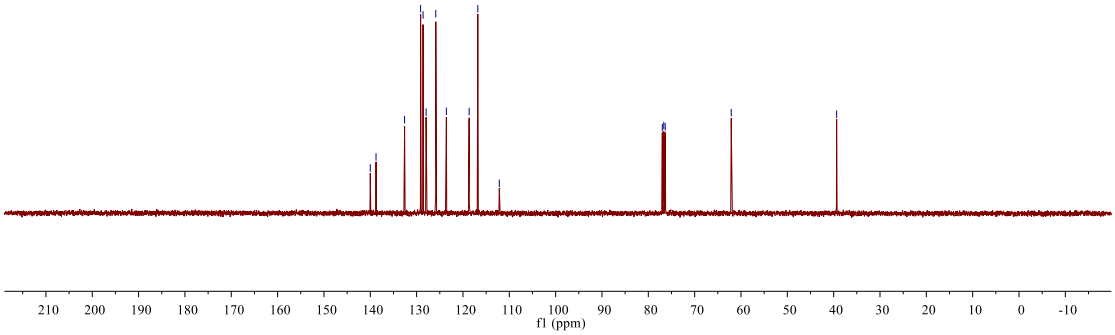
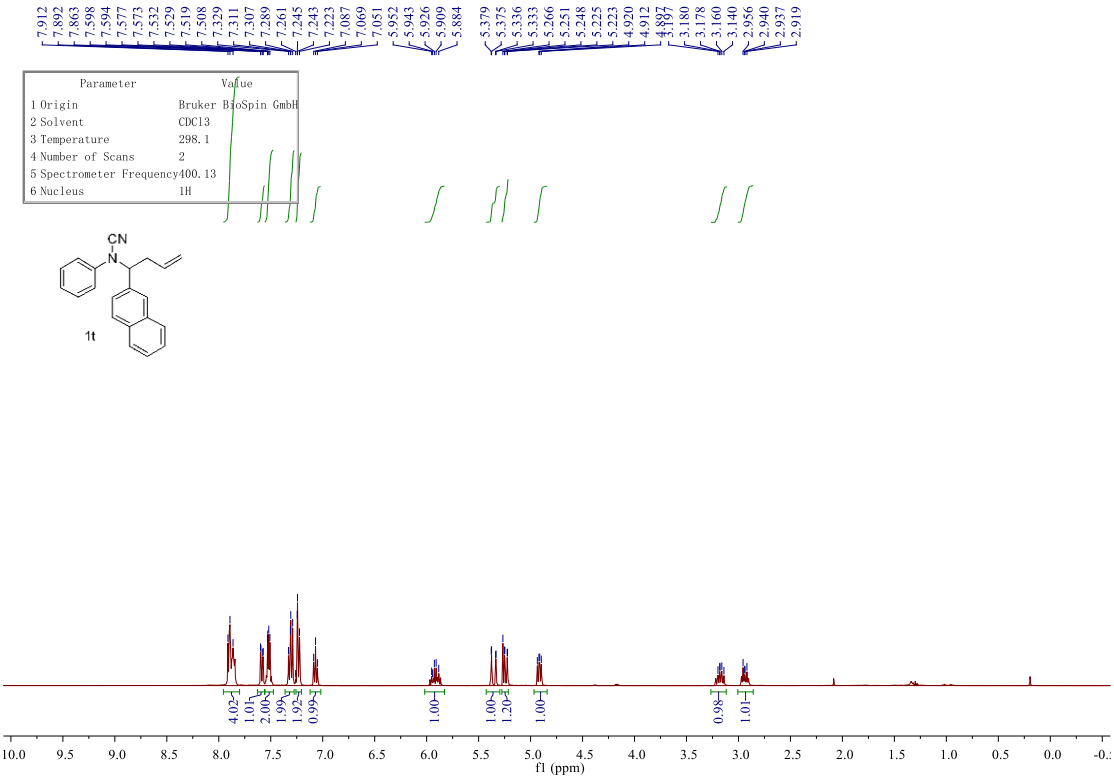
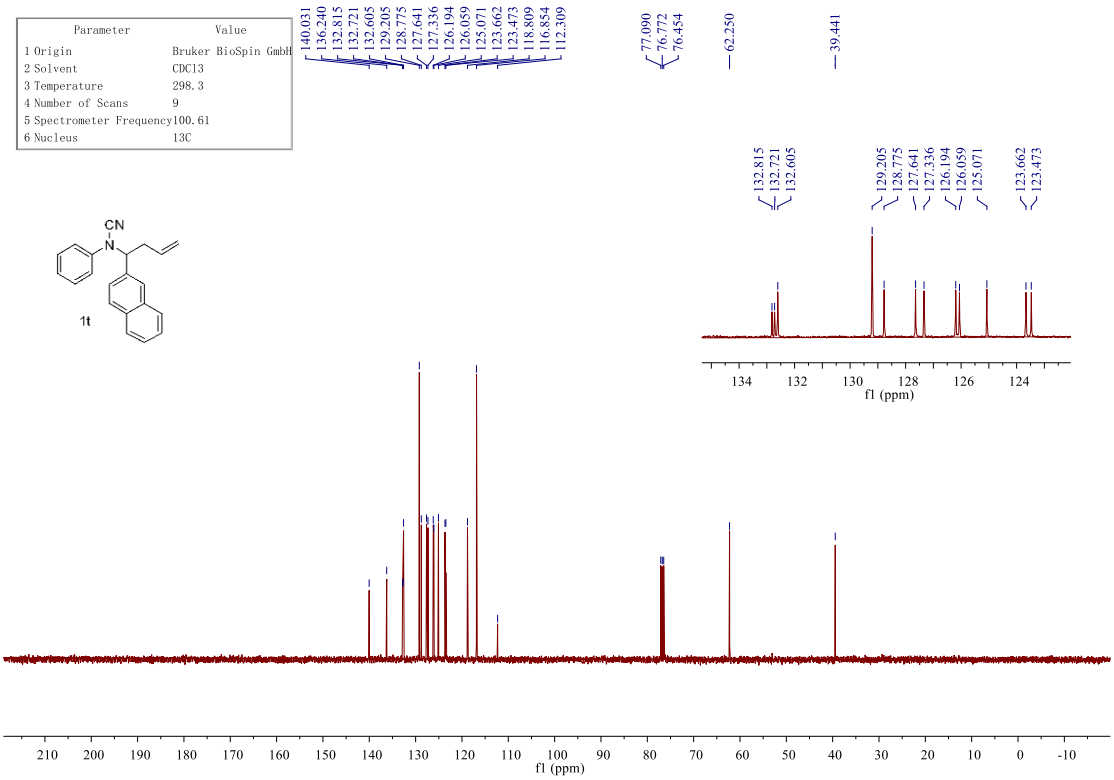
1.00
1.02
1.02
1.03
1.02
1.05
f1 (ppm)


140.001
138.761
132.595
129.137
128.610
127.955
125.864
123.581
118.665
116.792
112.157
76.981
76.663
76.345
62.103
39.358
Parameter Value
1 Origin Bruker BioSpin GmbH
2 Solvent CDCl3
3 Temperature 298.3
4 Number of Scans 19
5 Spectrometer Frequency 100.61
6 Nucleus 13C
CN
N
1s
f1 (ppm)

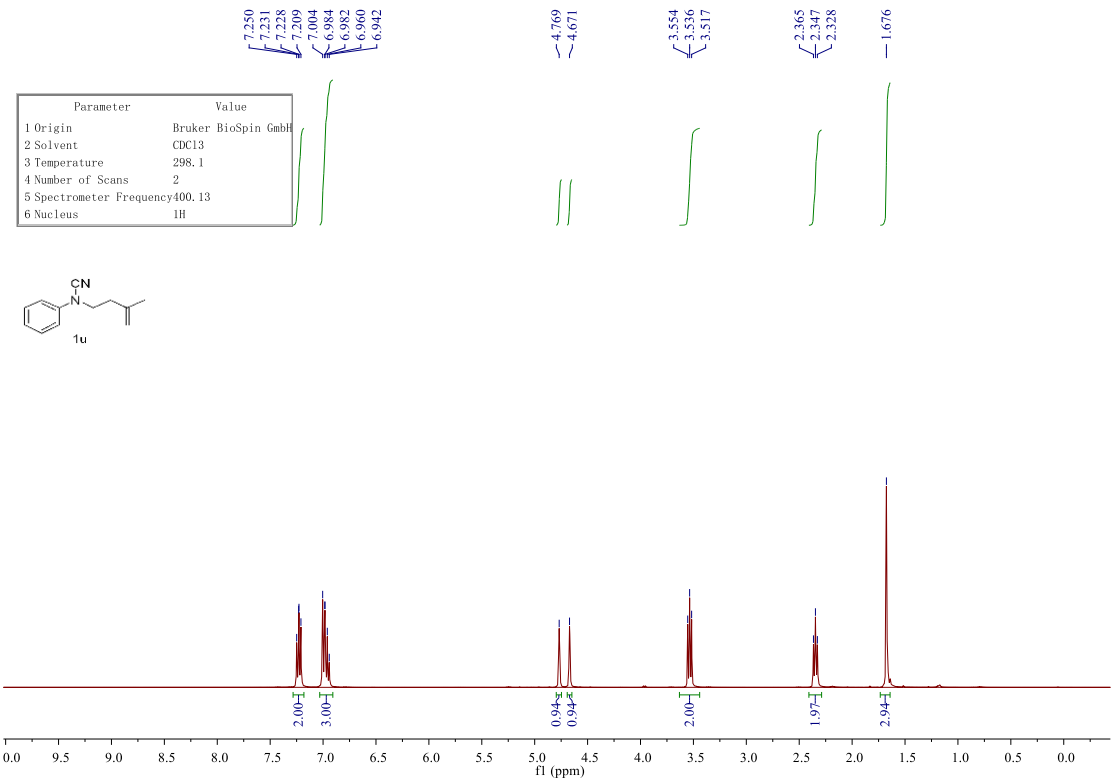

f1 (ppm)

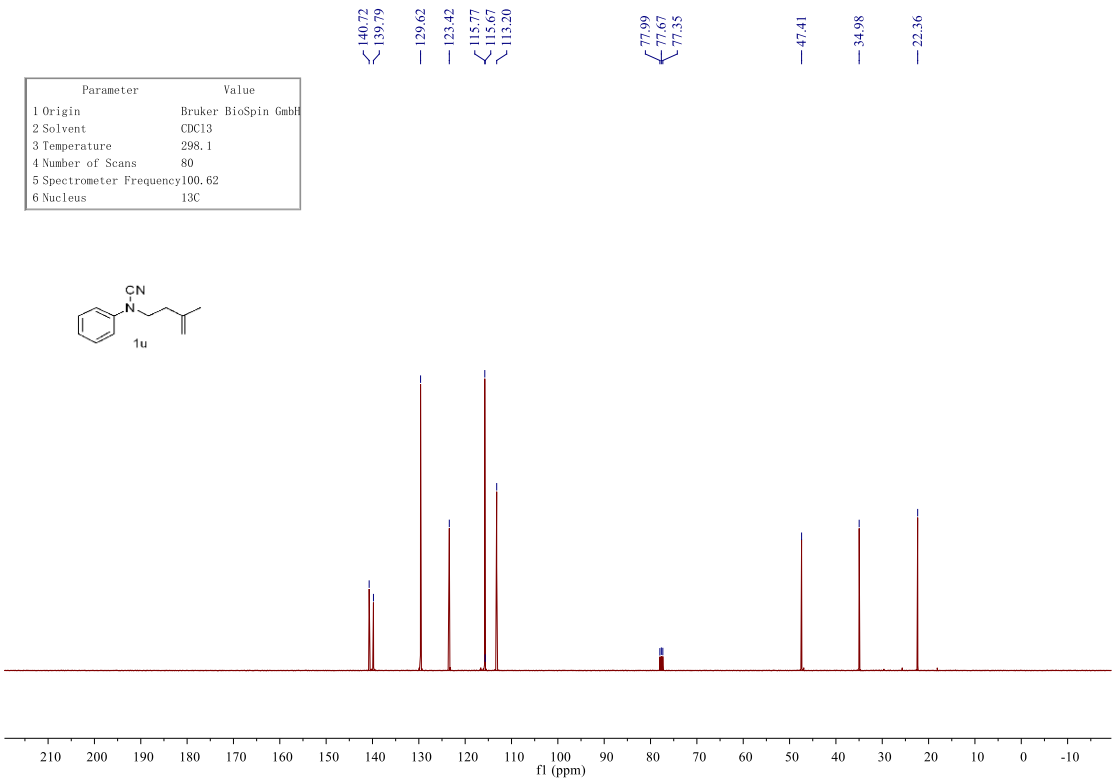

7.912
7.892
7.863
7.598
7.594
7.577
7.573
7.532
7.529
7.519
7.508
7.329
7.311
7.307
7.289
7.261
7.245
7.243
7.223
7.087
7.069
7.051
5.952
5.943
5.926
5.909
5.884
5.379
5.375
5.336
5.333
5.266
5.251
5.248
5.225
5.223
4.920
4.912
3.180
3.178
3.160
3.140
2.956
2.940
2.937
2.919
Parameter Value
1 Origin Bruker BioSpin GmbH
2 Solvent CDCl3
3 Temperature 298.1
4 Number of Scans 2
5 Spectrometer Frequency 400.13
6 Nucleus 1H
CN
N
1t
4.02
1.01
2.00
1.98
1.92
0.99
1.00
1.00
1.20
1.00
0.98
1.01
f1 (ppm)

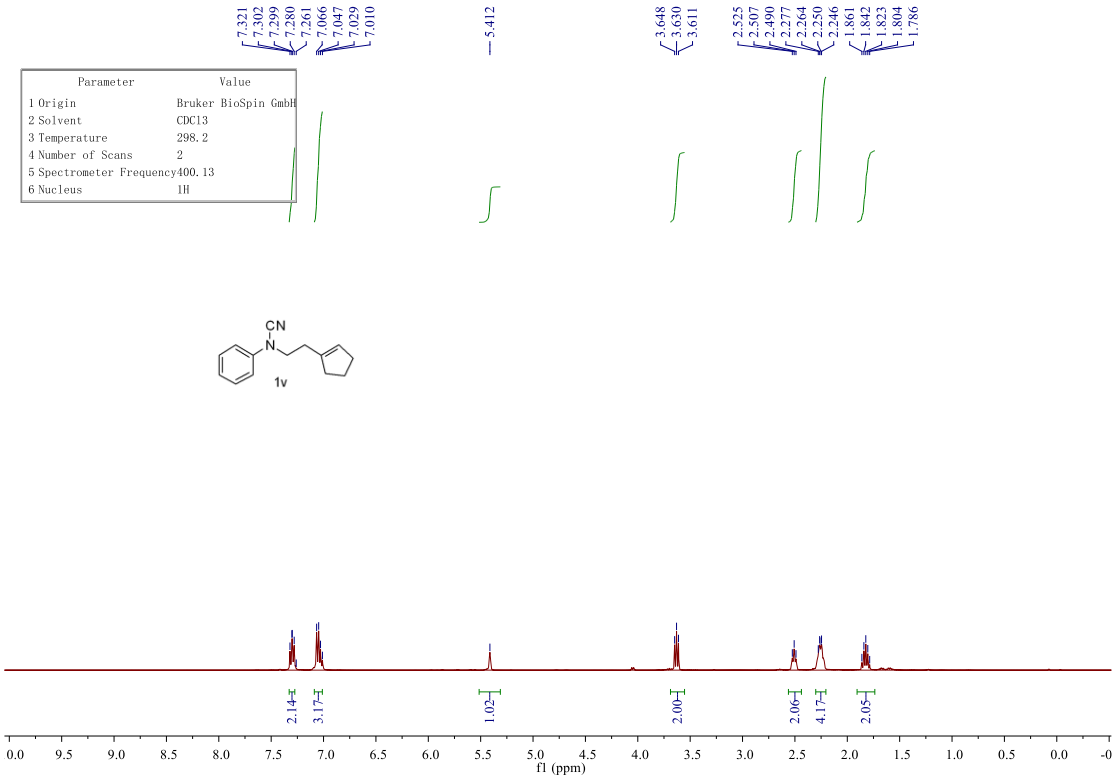
Parameter	Value
1 Origin	Bruker BioSpin GmbH
2 Solvent	CDCl3
3 Temperature	298.3
4 Number of Scans	9
5 Spectrometer Frequency	100.61
6 Nucleus	13C



Parameter	Value
1 Origin	Bruker BioSpin GmbH
2 Solvent	CDCl3
3 Temperature	298.1
4 Number of Scans	2
5 Spectrometer Frequency	400.13
6 Nucleus	1H

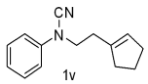



140.72
139.79
129.62
123.42
115.77
115.67
113.20
77.99
77.67
77.35
47.41
34.98
22.36
Parameter Value
1 Origin Bruker BioSpin GmbH
2 Solvent CDCl3
3 Temperature 298.1
4 Number of Scans 80
5 Spectrometer Frequency 100.62
6 Nucleus 13C
CN
N
1u
f1 (ppm)

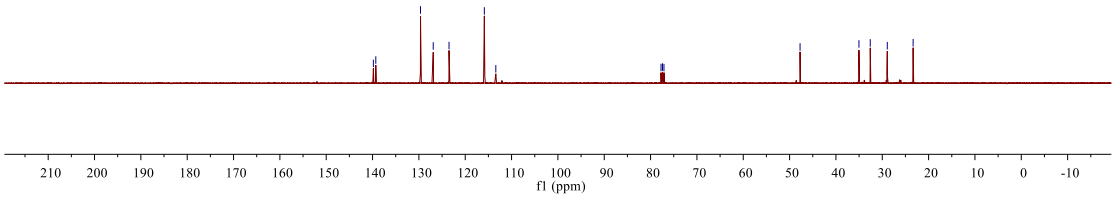

7.321
7.302
7.299
7.280
7.261
7.066
7.047
7.029
7.010
5.412
3.648
3.630
3.611
2.525
2.507
2.490
2.277
2.264
2.250
2.246
1.861
1.842
1.823
1.804
1.786
Parameter Value
1 Origin Bruker BioSpin GmbH
2 Solvent CDCl3
3 Temperature 298.2
4 Number of Scans 2
5 Spectrometer Frequency 400.13
6 Nucleus 1H
CN
N
1v
2.14
3.17
1.02
2.00
2.06
4.17
2.05
f1 (ppm)

Parameter	Value
1 Origin	Bruker BioSpin GmbH
2 Solvent	CDCl3
3 Temperature	298.2
4 Number of Scans	10
5 Spectrometer Frequency	100.61
6 Nucleus	13C

139.823
139.301
129.653
126.904
123.498
115.874
113.398
77.750
77.431
77.111
47.742
35.034
32.594
28.916
23.341

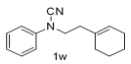


1v

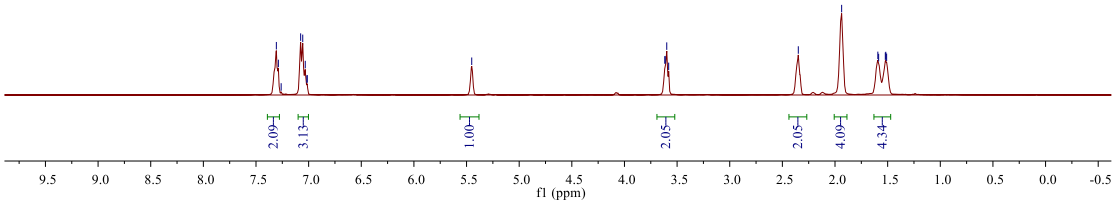


7.307
7.287
7.261
7.076
7.056
7.032
7.016
7.014
5.451
3.616
3.614
3.598
3.580
2.349
1.937
1.593
1.586
1.522
1.518
1.510

Parameter	Value
1 Origin	Bruker BioSpin GmbH
2 Solvent	CDCl3
3 Temperature	298.1
4 Number of Scans	2
5 Spectrometer Frequency	400.13
6 Nucleus	1H

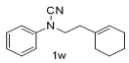


1w

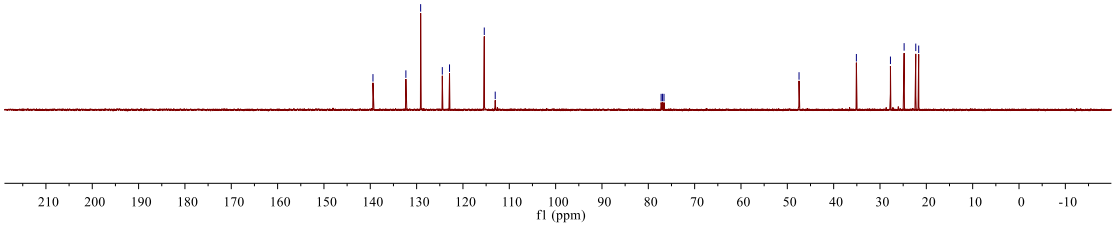


Parameter	Value
1 Origin	Bruker BioSpin GmbH
2 Solvent	CDCl3
3 Temperature	298.4
4 Number of Scans	5
5 Spectrometer Frequency	100.61
6 Nucleus	13C

139.421
132.319
129.130
124.455
122.900
115.400
113.039
77.235
76.930
76.613
47.473
35.097
27.745
24.793
22.281
21.648

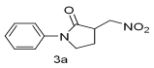


1w



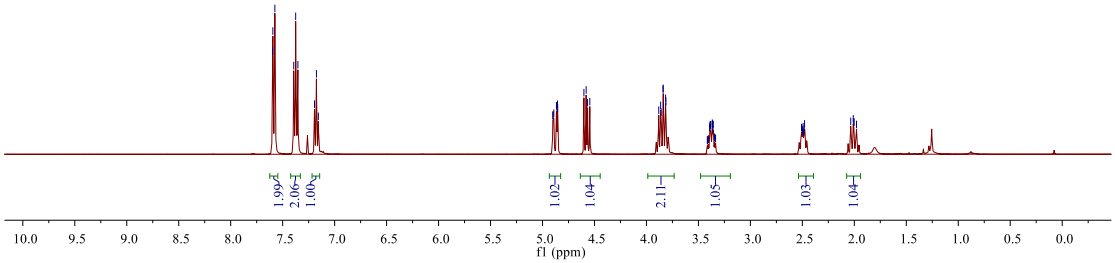
7.597
7.595
7.575
7.394
7.375
7.354
7.194
7.175
7.157
4.901
4.898
4.891
4.866
4.863
4.856
4.854
4.602
4.580
4.567
4.545
3.841
3.839
3.816
3.383
3.367
3.364
3.357
2.508
2.493
2.484
2.479
2.035
2.011
2.005
2.003
1.979

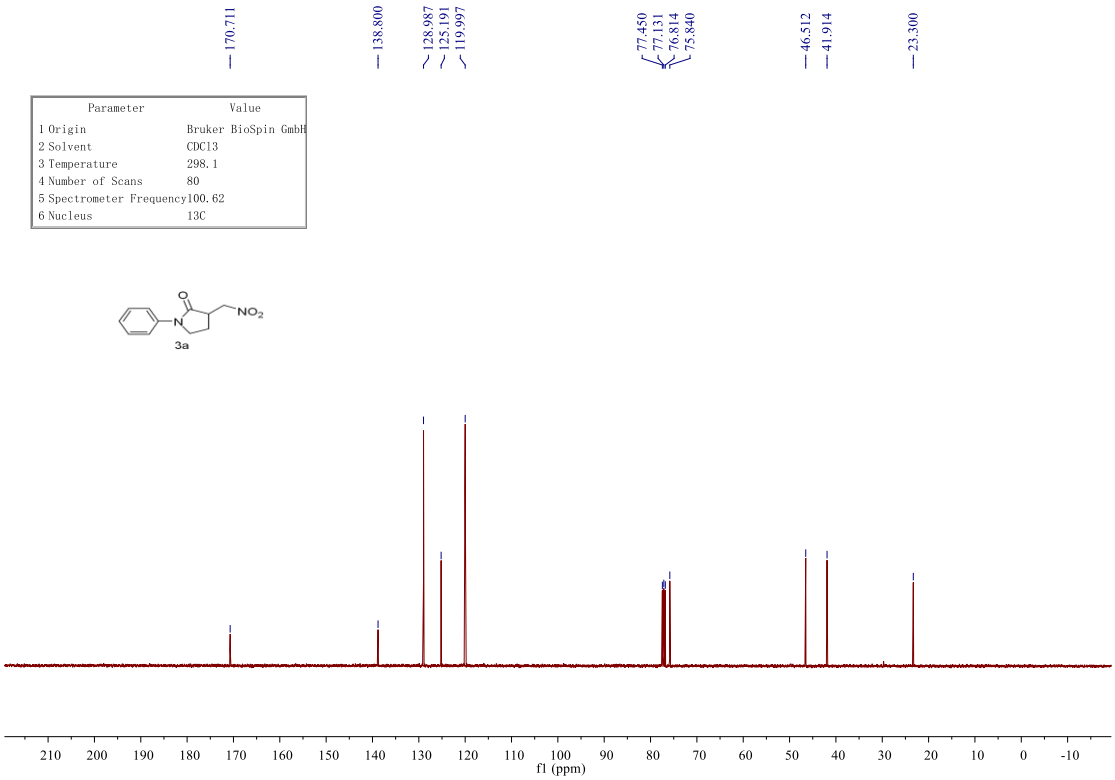
Parameter	Value
1 Origin	Bruker BioSpin GmbH
2 Solvent	CDCl3
3 Temperature	298.2
4 Number of Scans	2
5 Spectrometer Frequency	400.13
6 Nucleus	1H

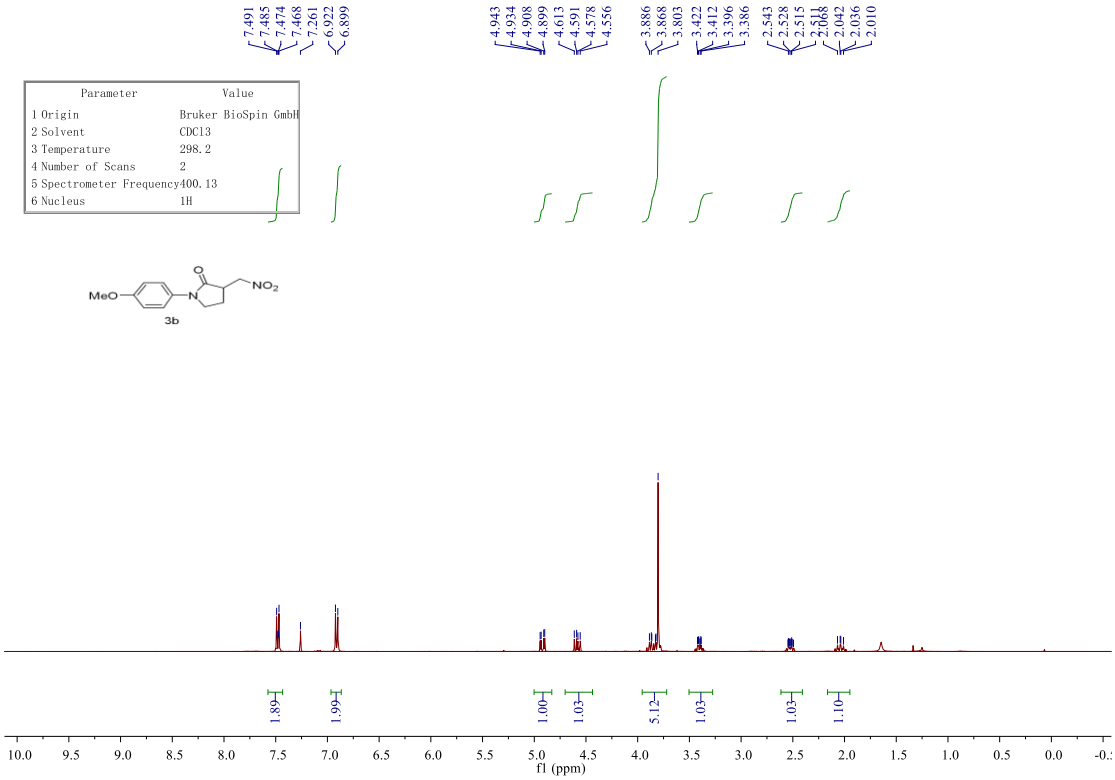


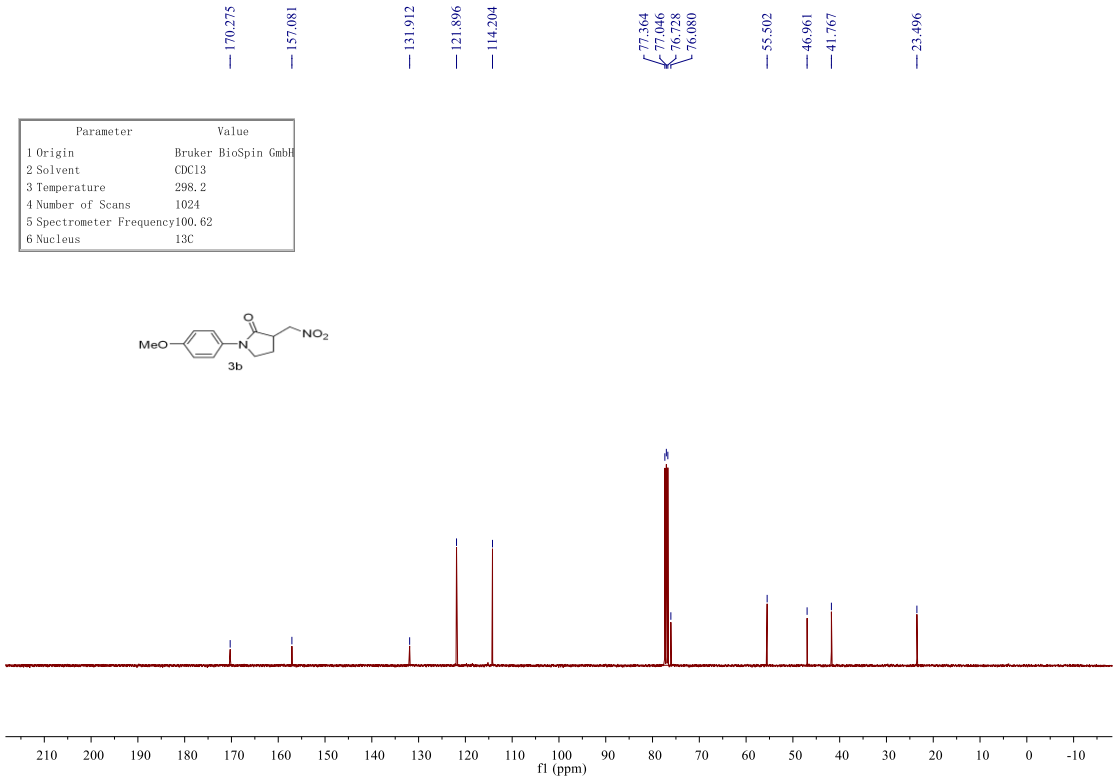
3a

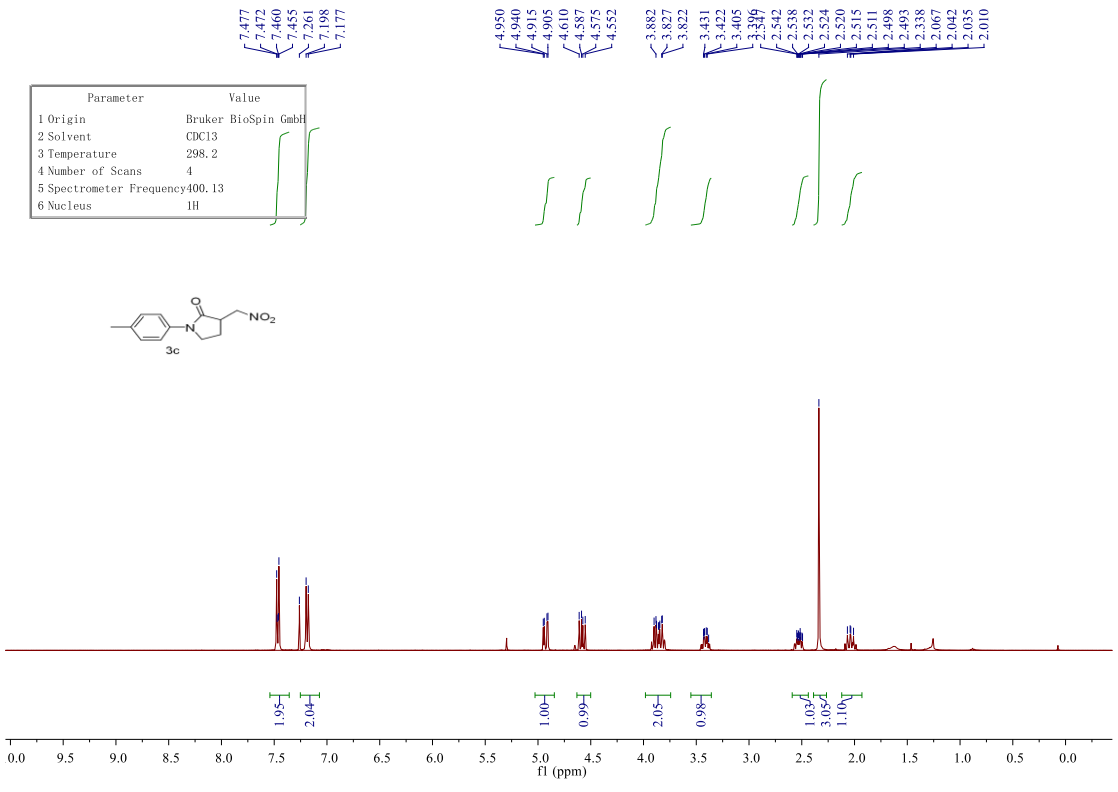
1.99
2.06
1.00
1.02
1.04
2.11
1.05
1.03
1.04

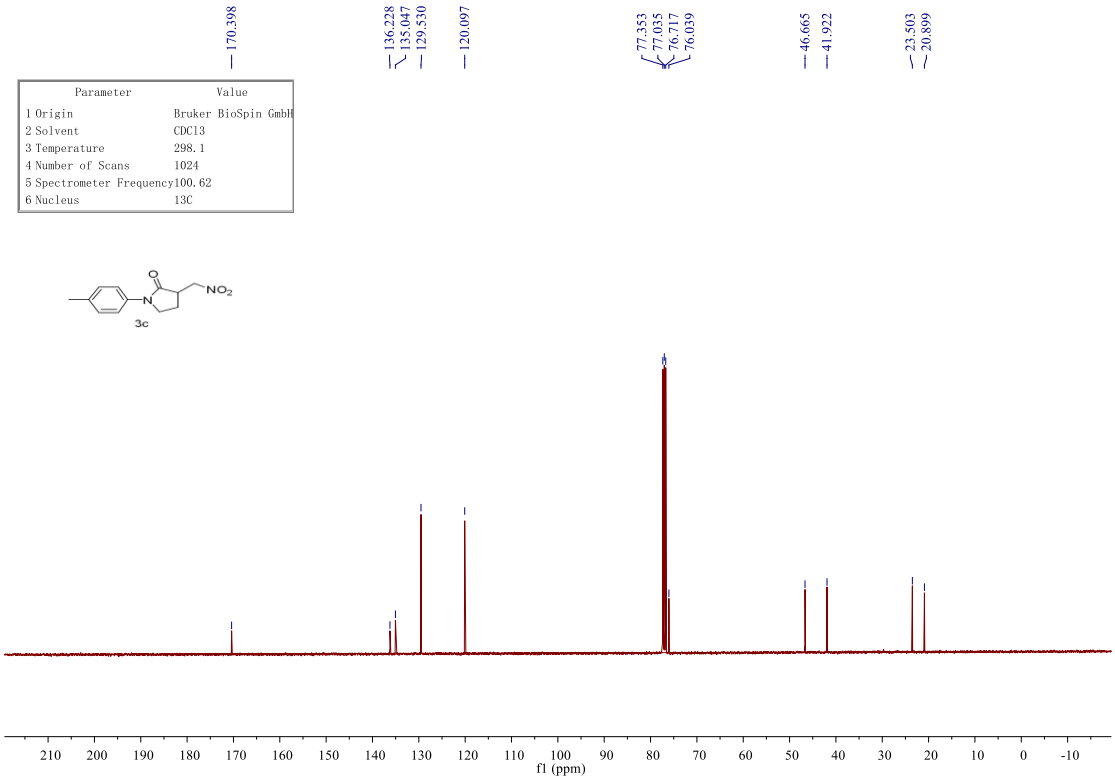


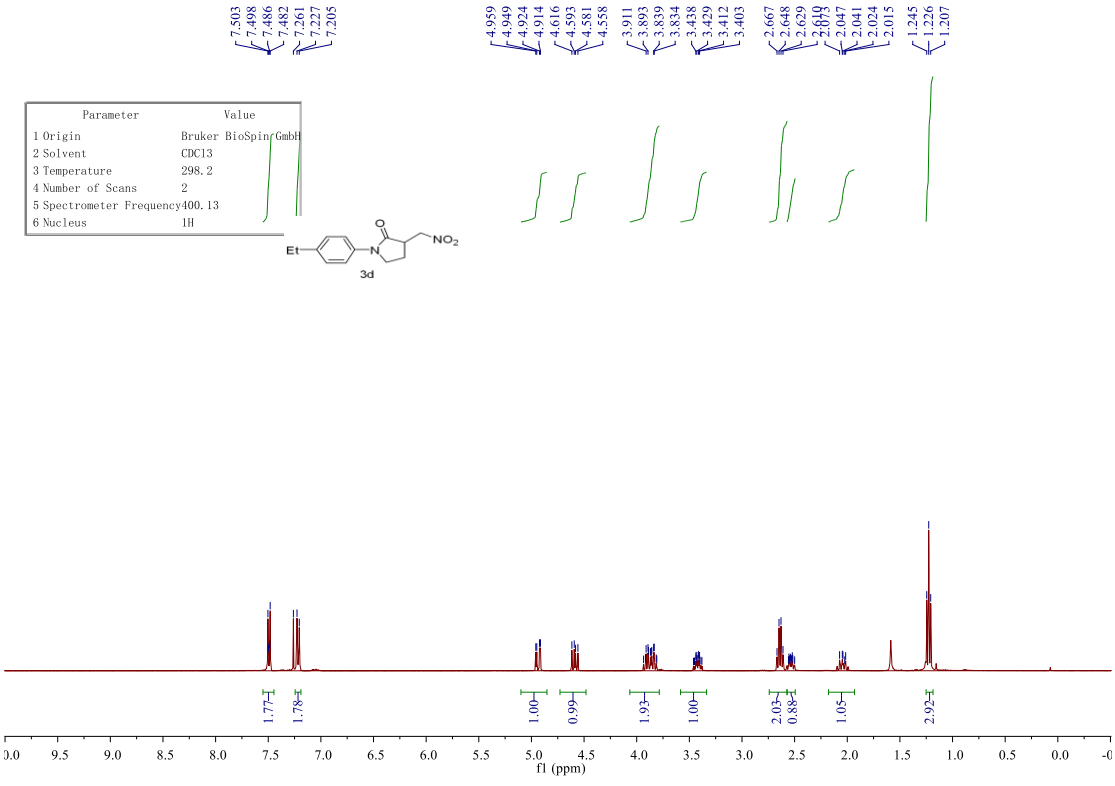

170.711
138.800
128.987
125.191
119.997
77.450
77.131
76.814
75.840
46.512
41.914
23.300
Parameter Value
1 Origin Bruker BioSpin GmbH
2 Solvent CDCl3
3 Temperature 298.1
4 Number of Scans 80
5 Spectrometer Frequency 100.62
6 Nucleus 13C
NO2
3a
f1 (ppm)

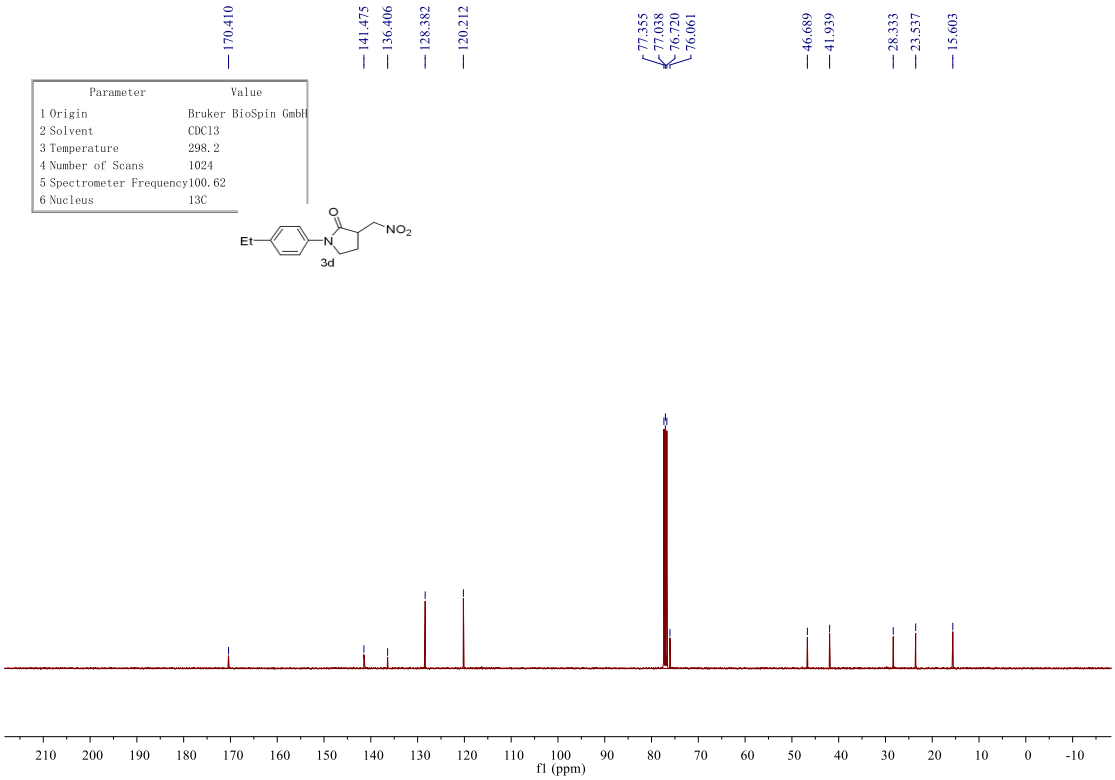

7.491
7.485
7.474
7.468
7.261
6.922
6.899
4.943
4.934
4.908
4.899
4.613
4.591
4.578
4.556
3.886
3.868
3.803
3.422
3.412
3.396
3.386
2.543
2.528
2.515
2.068
2.042
2.036
2.010
Parameter Value
1 Origin Bruker BioSpin GmbH
2 Solvent CDCl3
3 Temperature 298.2
4 Number of Scans 2
5 Spectrometer Frequency 400.13
6 Nucleus 1H
MeO
NO2
3b
1.89
1.99
1.00
1.03
5.12
1.03
1.03
1.10
f1 (ppm)

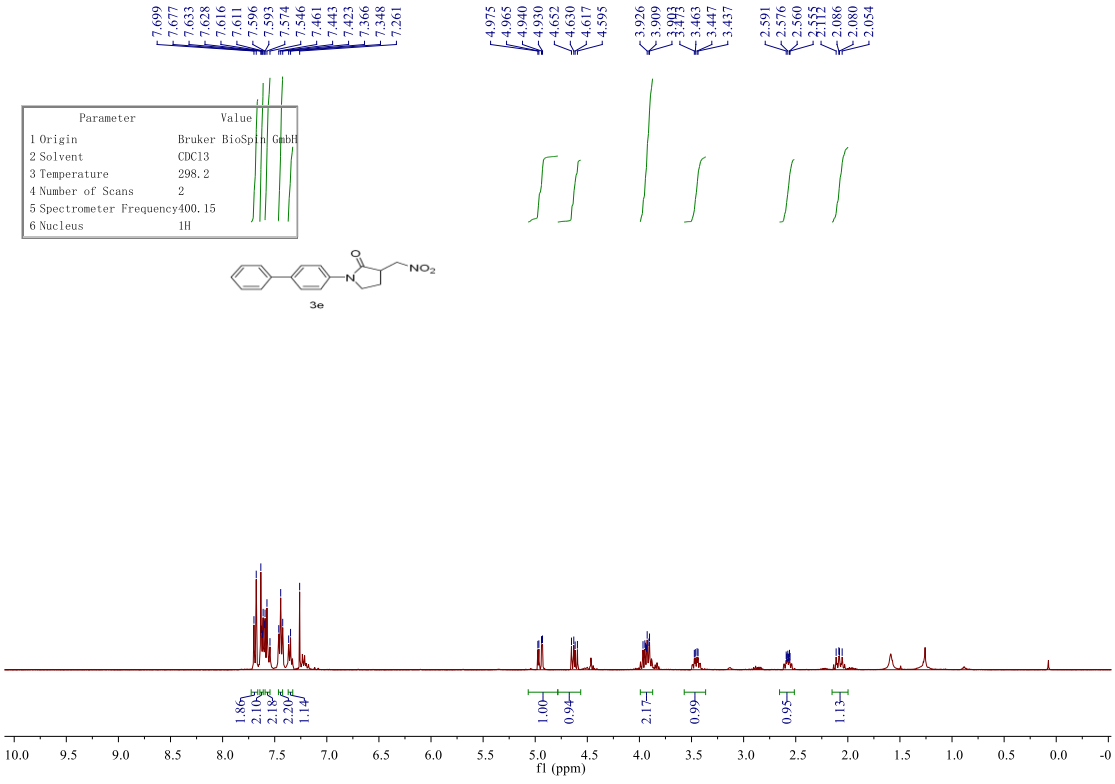

170.275
157.081
131.912
121.896
114.204
77.364
77.046
76.728
76.080
55.502
46.961
41.767
23.496
Parameter Value
1 Origin Bruker BioSpin GmbH
2 Solvent CDCl3
3 Temperature 298.2
4 Number of Scans 1024
5 Spectrometer Frequency 100.62
6 Nucleus 13C
MeO
NO2
3b
f1 (ppm)

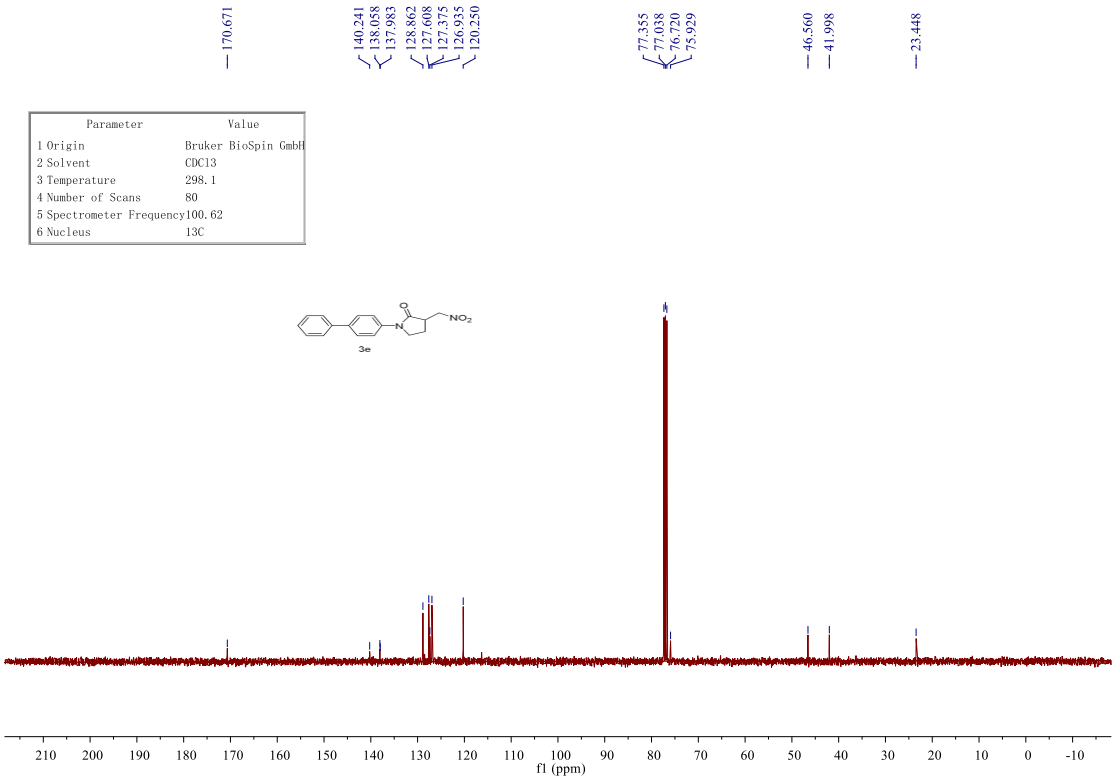

7.477
7.472
7.460
7.455
7.261
7.198
7.177
4.950
4.940
4.915
4.905
4.610
4.587
4.575
4.552
3.882
3.827
3.822
3.431
3.422
3.405
2.542
2.538
2.532
2.524
2.520
2.515
2.511
2.498
2.493
2.338
2.067
2.042
2.035
2.010
Parameter Value
1 Origin Bruker BioSpin GmbH
2 Solvent CDCl3
3 Temperature 298.2
4 Number of Scans 4
5 Spectrometer Frequency 400.13
6 Nucleus 1H
NO2
3c
1.95
2.04
1.00
0.99
2.05
0.98
1.03
3.05
1.10
f1 (ppm)

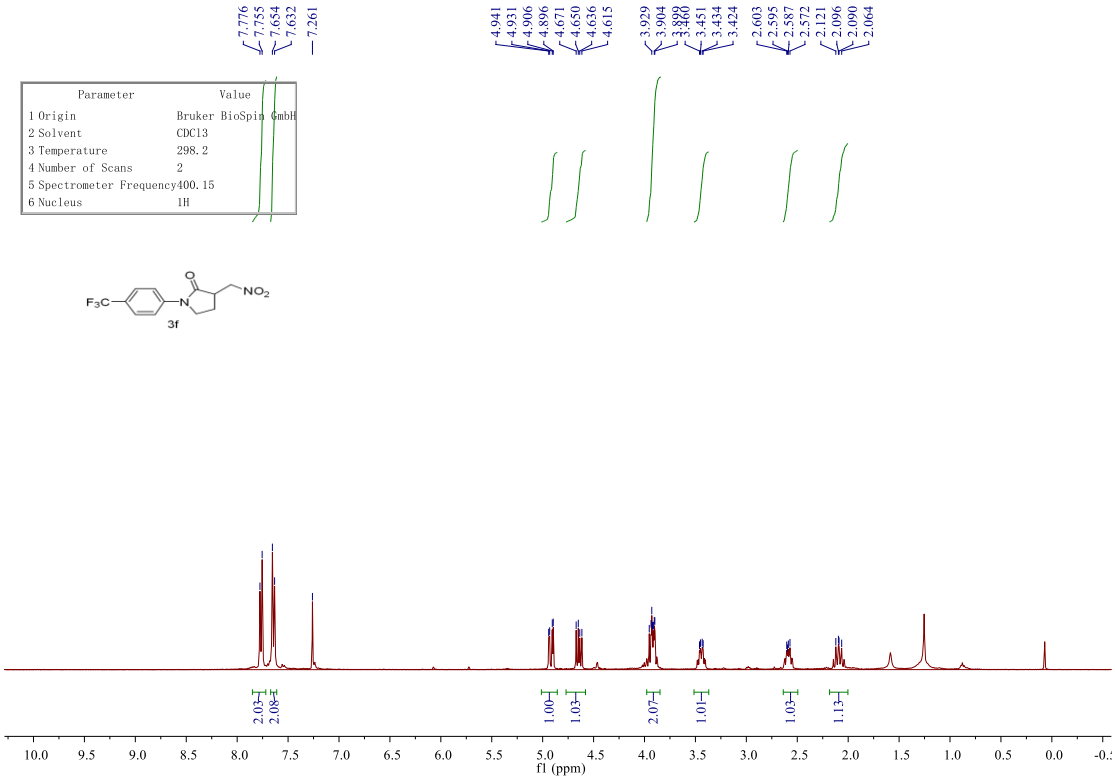

170.398
136.228
135.047
129.530
120.097
77.353
77.035
76.717
76.039
46.665
41.922
23.503
20.899
Parameter Value
1 Origin Bruker BioSpin GmbH
2 Solvent CDCl3
3 Temperature 298.1
4 Number of Scans 1024
5 Spectrometer Frequency 100.62
6 Nucleus 13C
NO2
3c
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)

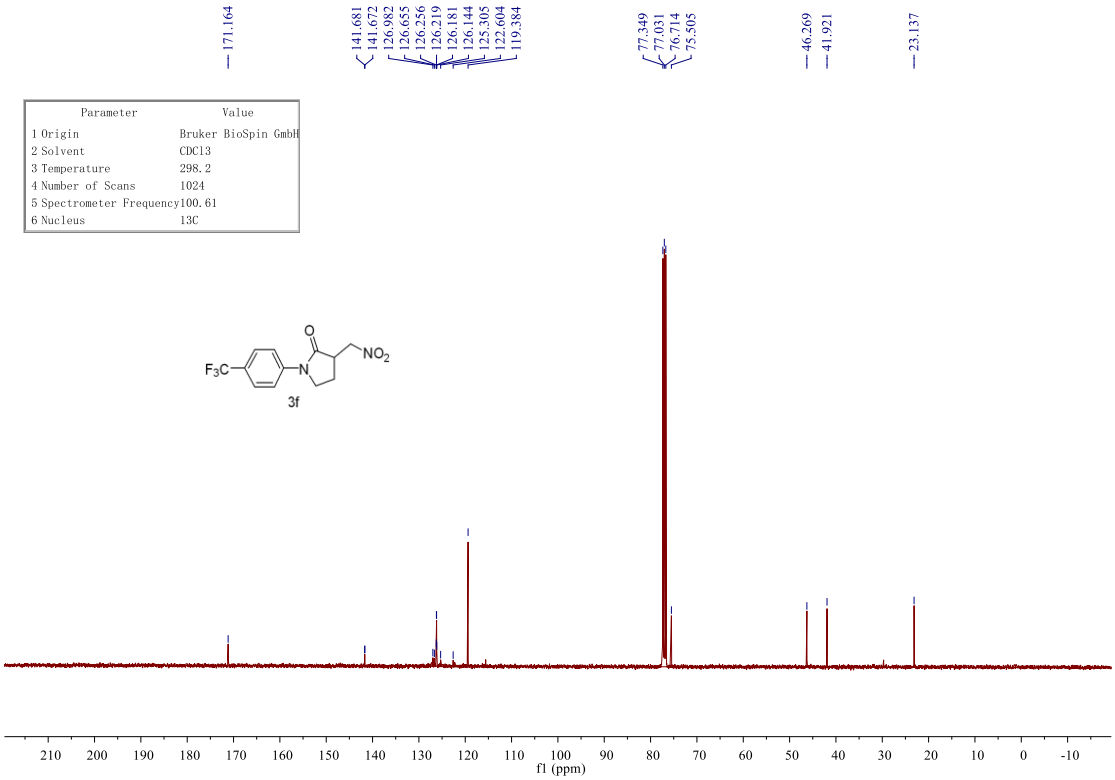

7.503
7.498
7.486
7.482
7.261
7.227
7.205
4.959
4.949
4.924
4.914
4.616
4.593
4.581
4.558
3.911
3.893
3.839
3.834
3.438
3.429
3.412
3.403
2.667
2.648
2.629
2.610
2.047
2.041
2.024
2.015
1.245
1.226
1.207
Parameter Value
1 Origin Bruker BioSpin GmbH
2 Solvent CDCl3
3 Temperature 298.2
4 Number of Scans 2
5 Spectrometer Frequency 400.13
6 Nucleus 1H
Et
NO2
3d
1.77
1.78
1.00
0.99
1.93
1.00
2.03
0.88
1.05
2.92
0.0 9.5 9.0 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
f1 (ppm)

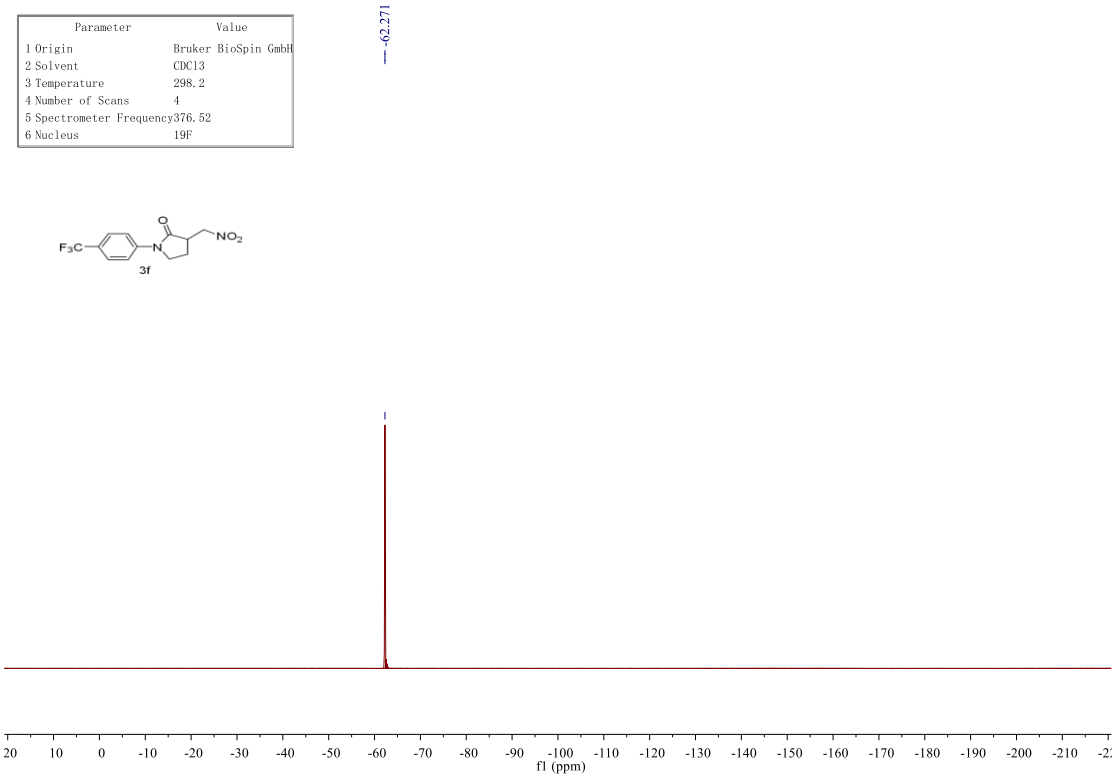

170.410
141.475
136.406
128.382
120.212
77.355
77.038
76.720
76.061
46.689
41.939
28.333
23.537
15.603
Parameter Value
1 Origin Bruker BioSpin GmbH
2 Solvent CDCl3
3 Temperature 298.2
4 Number of Scans 1024
5 Spectrometer Frequency 100.62
6 Nucleus 13C
Et
NO2
3d
f1 (ppm)

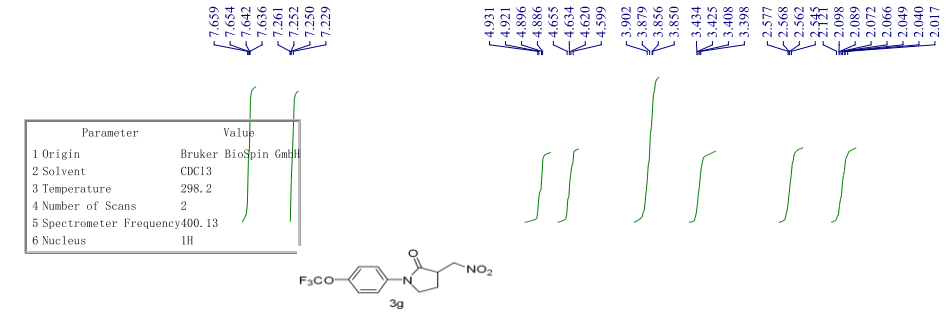

7.699
7.677
7.633
7.628
7.616
7.611
7.596
7.593
7.574
7.546
7.461
7.443
7.423
7.366
7.348
7.261
4.975
4.965
4.940
4.930
4.652
4.630
4.617
4.595
3.926
3.909
3.903
3.473
3.463
3.447
3.437
2.591
2.576
2.560
2.555
2.112
2.086
2.080
2.054
Parameter Value
1 Origin Bruker BioSpin GmbH
2 Solvent CDCl3
3 Temperature 298.2
4 Number of Scans 2
5 Spectrometer Frequency 400.15
6 Nucleus 1H
NO2
3e
1.86
2.10
2.18
2.20
1.14
1.00
0.94
2.17
0.99
0.95
1.13
f1 (ppm)


170.671
140.241
138.058
137.983
128.862
127.608
127.375
126.935
120.250
77.355
77.038
76.720
75.929
46.560
41.998
23.448
Parameter Value
1 Origin Bruker BioSpin GmbH
2 Solvent CDCl3
3 Temperature 298.1
4 Number of Scans 80
5 Spectrometer Frequency 100.62
6 Nucleus 13C
NO2
3e
f1 (ppm)


7.776
7.755
7.654
7.632
7.261
4.941
4.931
4.906
4.896
4.671
4.650
4.636
4.615
3.929
3.904
3.451
3.434
3.424
2.603
2.595
2.587
2.572
2.121
2.096
2.090
2.064
Parameter Value
1 Origin Bruker BioSpin GmbH
2 Solvent CDCl3
3 Temperature 298.2
4 Number of Scans 2
5 Spectrometer Frequency 400.15
6 Nucleus 1H
F3C
NO2
3f
2.03
2.08
1.00
1.03
2.07
1.01
1.03
1.13
f1 (ppm)

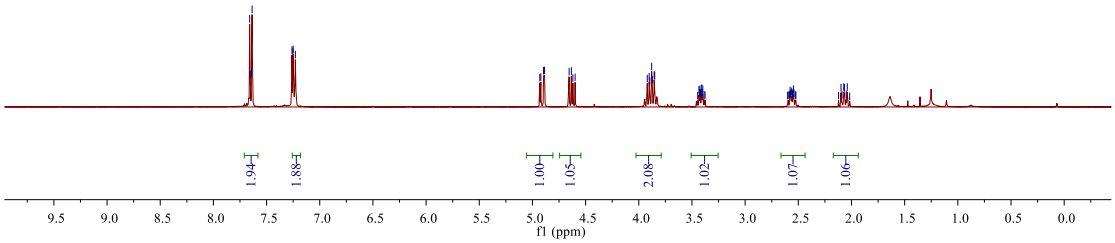

171.164
141.681
141.672
126.982
126.655
126.256
126.219
126.181
126.144
125.305
122.604
119.384
77.349
77.031
76.714
75.505
46.269
41.921
23.137
Parameter Value
1 Origin Bruker BioSpin GmbH
2 Solvent CDCl3
3 Temperature 298.2
4 Number of Scans 1024
5 Spectrometer Frequency 100.61
6 Nucleus 13C
F3C
N
O
NO2
3f
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)


-62.271
Parameter Value
1 Origin Bruker BioSpin GmbH
2 Solvent CDCl3
3 Temperature 298.2
4 Number of Scans 4
5 Spectrometer Frequency 376.52
6 Nucleus 19F
F3C
N
O
NO2
3f
20 10 0 -10 -20 -30 -40 -50 -60 -70 -80 -90 -100 -110 -120 -130 -140 -150 -160 -170 -180 -190 -200 -210
f1 (ppm)

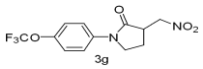


Parameter	Value
1 Origin	Bruker BioSpin GmbH
2 Solvent	CDCl3
3 Temperature	298.2
4 Number of Scans	2
5 Spectrometer Frequency	400.13
6 Nucleus	1H

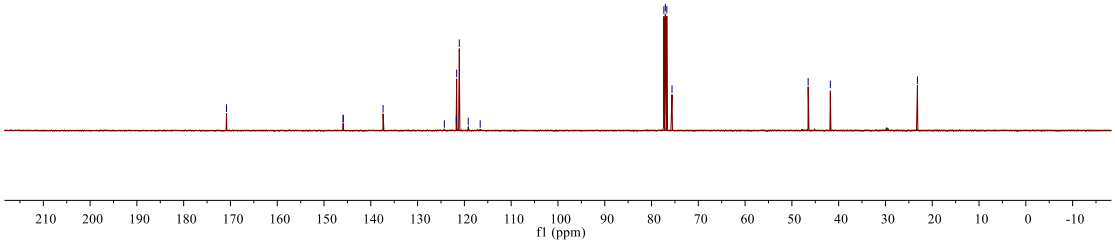
3g



Parameter	Value
1 Origin	Bruker BioSpin GmbH
2 Solvent	CDCl3
3 Temperature	298.2
4 Number of Scans	1024
5 Spectrometer Frequency	100.62
6 Nucleus	13C

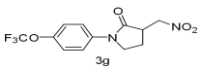


3g

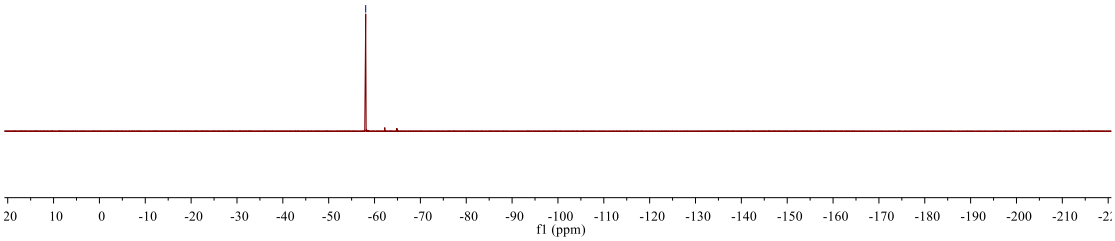


Parameter	Value
1 Origin	Bruker BioSpin GmbH
2 Solvent	CDCl3
3 Temperature	298.2
4 Number of Scans	4
5 Spectrometer Frequency	376.52
6 Nucleus	19F

-58.074

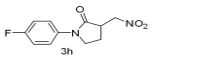


3g

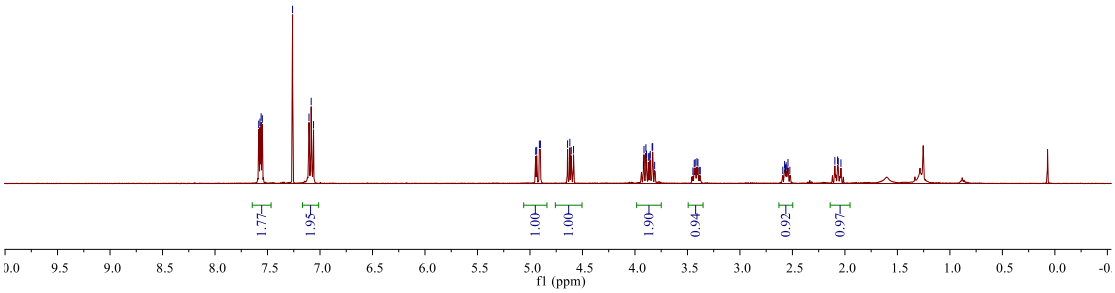


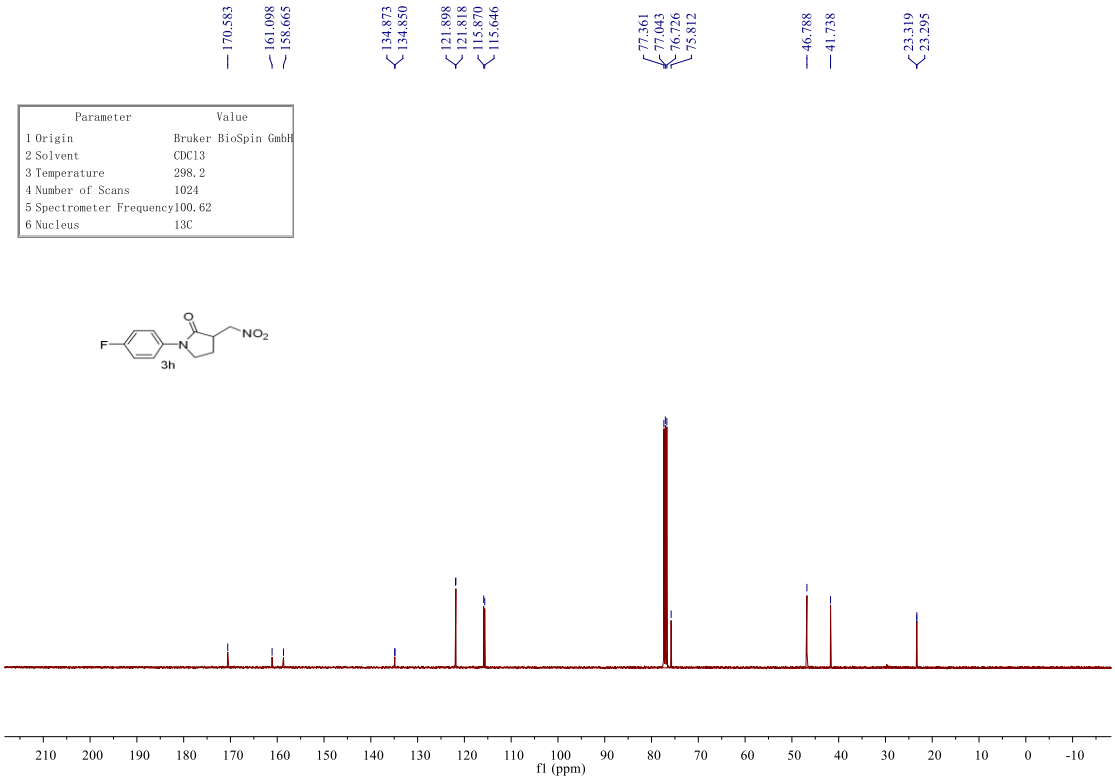
7.584 7.572 7.561 7.549 7.261 7.105 7.084 7.061 4.945 4.936 4.910 4.901 4.642 4.620 4.607 4.585 3.914 3.896 3.836 3.831 3.435 3.426 3.409 3.400 2.574 2.565 2.559 2.542 2.096 2.070 2.064 2.038

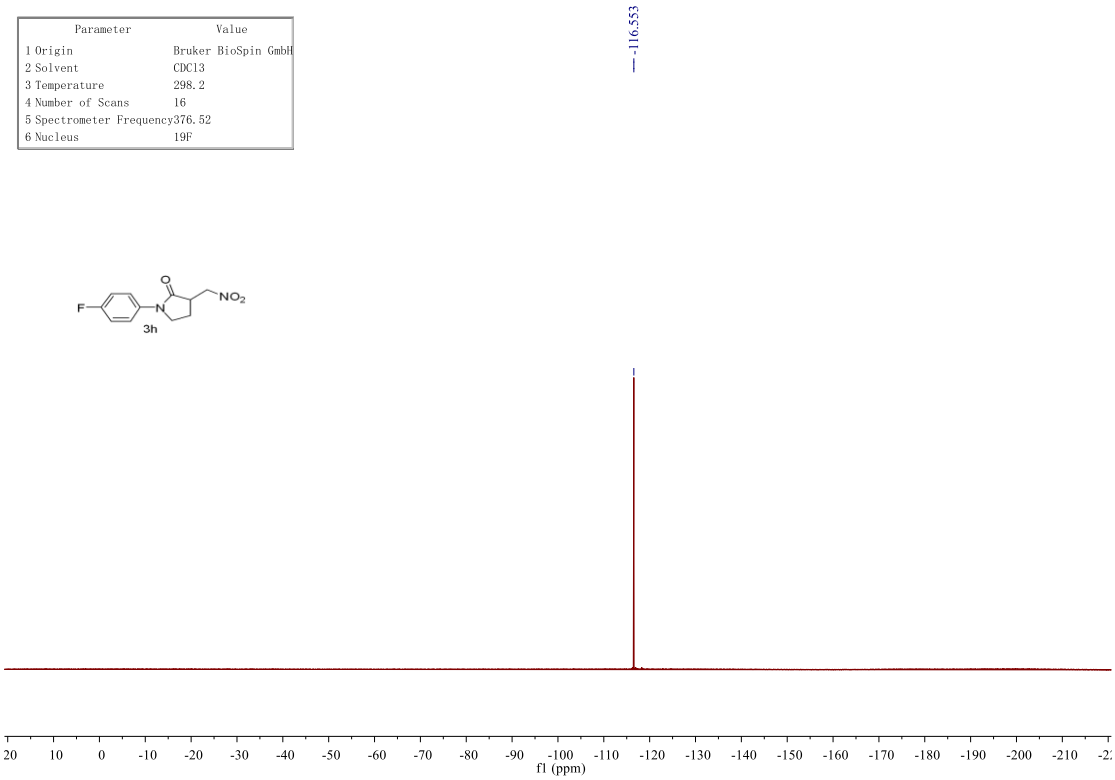
Parameter	Value
1 Origin	Bruker BioSpin GmbH
2 Solvent	CDCl3
3 Temperature	298.1
4 Number of Scans	2
5 Spectrometer Frequency	400.13
6 Nucleus	1H

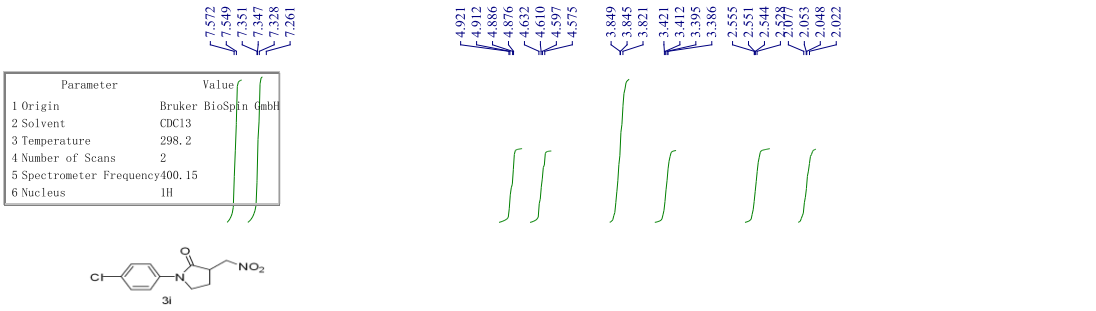


3h



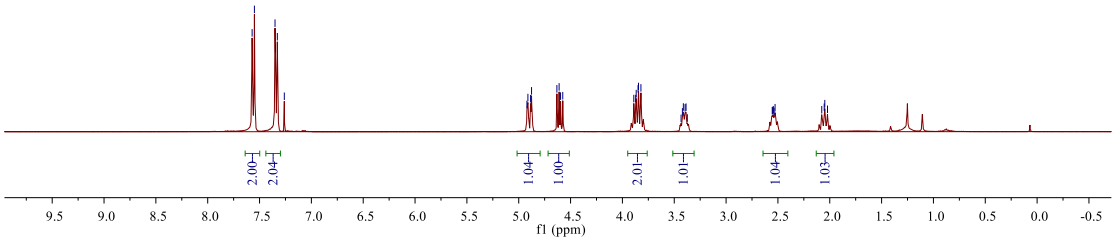

170.583
161.098
158.665
134.873
134.850
121.898
121.818
115.870
115.646
77.361
77.043
76.726
75.812
46.788
41.738
23.319
23.295
Parameter Value
1 Origin Bruker BioSpin GmbH
2 Solvent CDCl3
3 Temperature 298.2
4 Number of Scans 1024
5 Spectrometer Frequency 100.62
6 Nucleus 13C
F
N
O
NO2
3h
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)


-116.553
Parameter Value
1 Origin Bruker BioSpin GmbH
2 Solvent CDCl3
3 Temperature 298.2
4 Number of Scans 16
5 Spectrometer Frequency 376.52
6 Nucleus 19F
F
N
O
NO2
3h
20 10 0 -10 -20 -30 -40 -50 -60 -70 -80 -90 -100 -110 -120 -130 -140 -150 -160 -170 -180 -190 -200 -210
f1 (ppm)

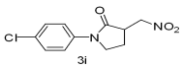


Parameter	Value
1 Origin	Bruker BioSpin GmbH
2 Solvent	CDCl3
3 Temperature	298.2
4 Number of Scans	2
5 Spectrometer Frequency	400.15
6 Nucleus	1H

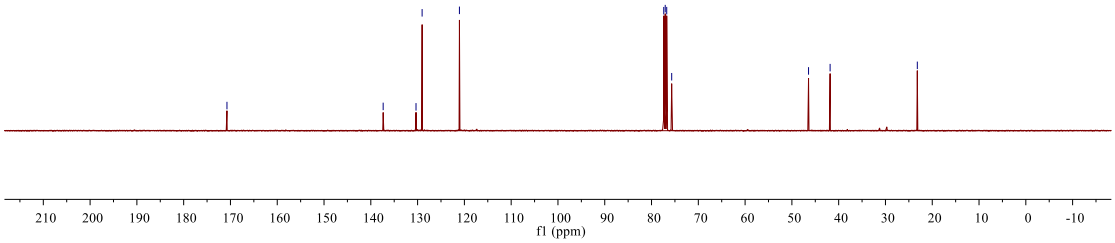
3i

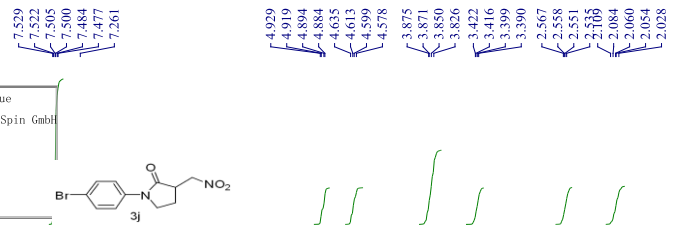


Parameter	Value
1 Origin	Bruker BioSpin GmbH
2 Solvent	CDCl3
3 Temperature	298.2
4 Number of Scans	1024
5 Spectrometer Frequency	100.62
6 Nucleus	13C



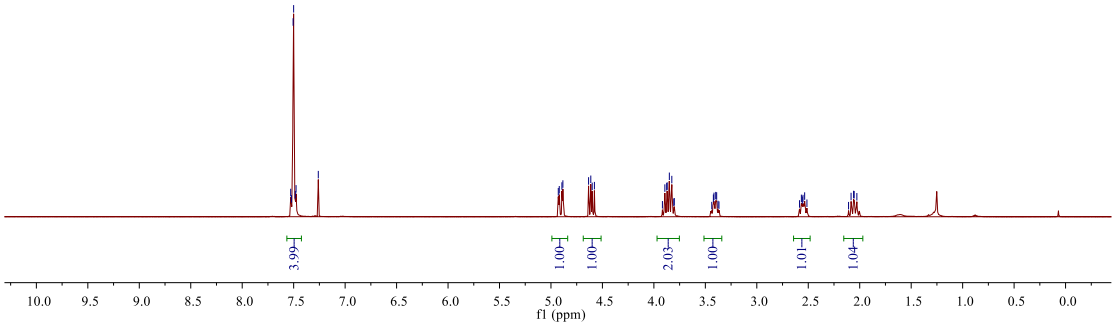
3i



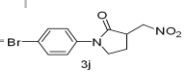


Parameter	Value
1 Origin	Bruker BioSpin GmbH
2 Solvent	CDCl3
3 Temperature	298.1
4 Number of Scans	2
5 Spectrometer Frequency	400.15
6 Nucleus	1H

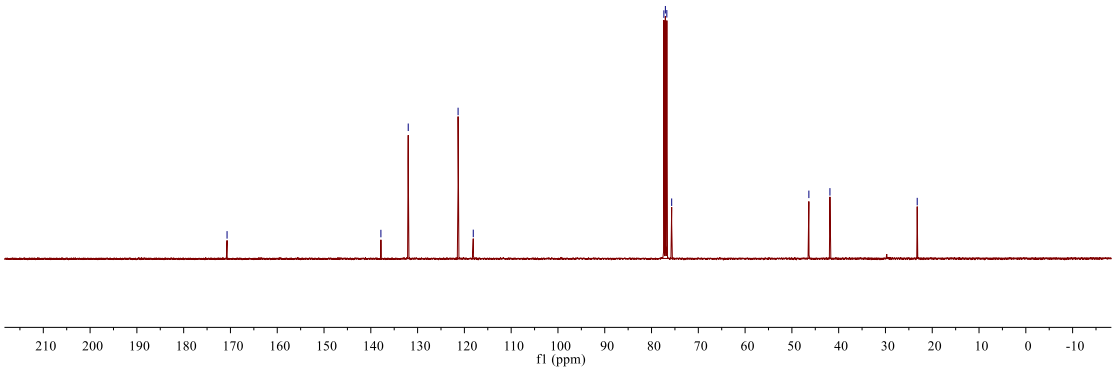
3j

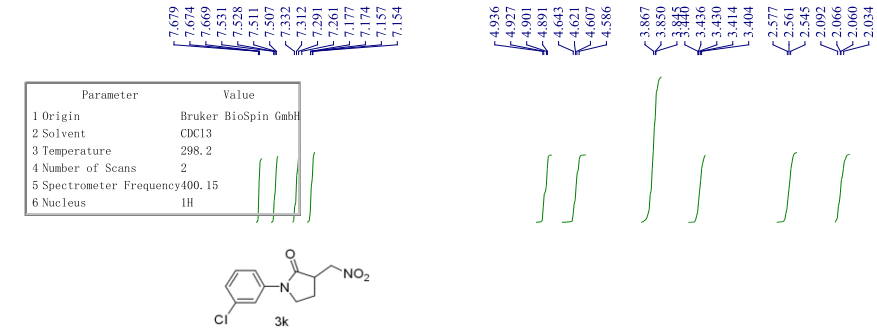


Parameter	Value
1 Origin	Bruker BioSpin GmbH
2 Solvent	CDCl3
3 Temperature	298.1
4 Number of Scans	1024
5 Spectrometer Frequency	100.62
6 Nucleus	13C



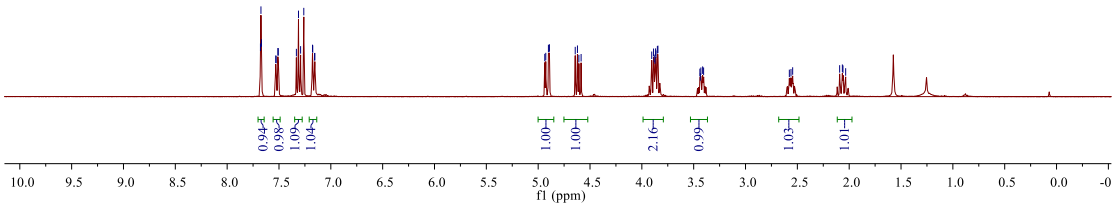
3j



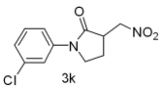


Parameter	Value
1 Origin	Bruker BioSpin GmbH
2 Solvent	CDCl3
3 Temperature	298.2
4 Number of Scans	2
5 Spectrometer Frequency	400.15
6 Nucleus	1H

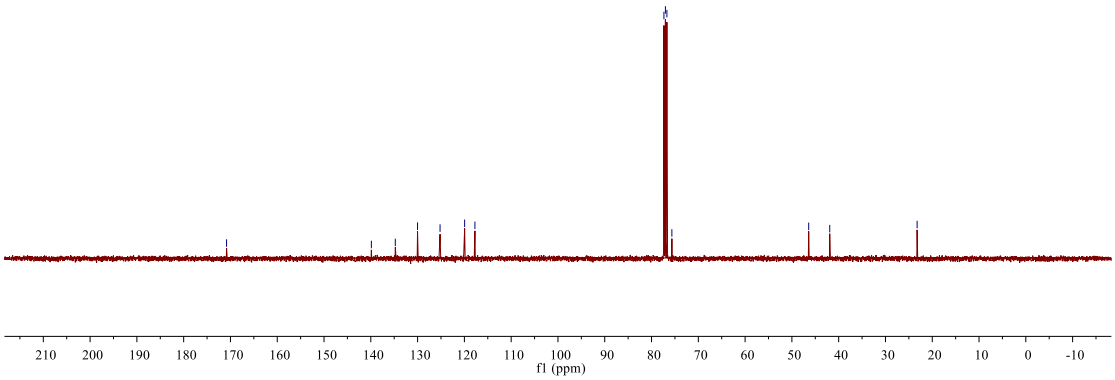
3k

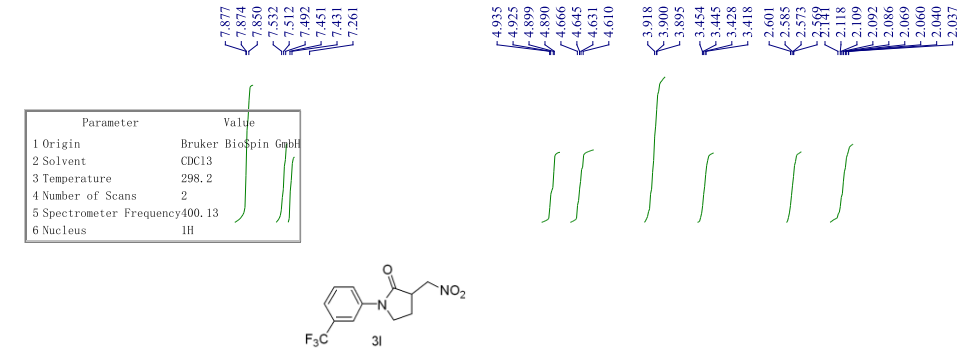


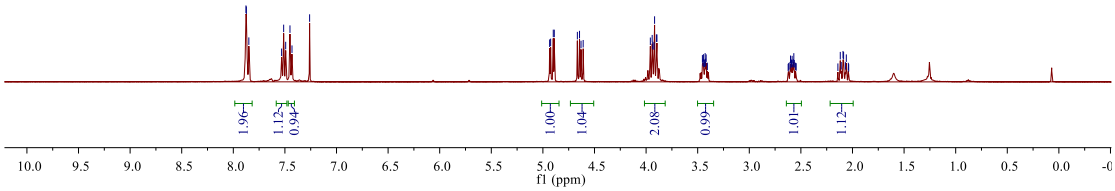
Parameter	Value
1 Origin	Bruker BioSpin GmbH
2 Solvent	CDCl3
3 Temperature	298.1
4 Number of Scans	80
5 Spectrometer Frequency	100.62
6 Nucleus	13C

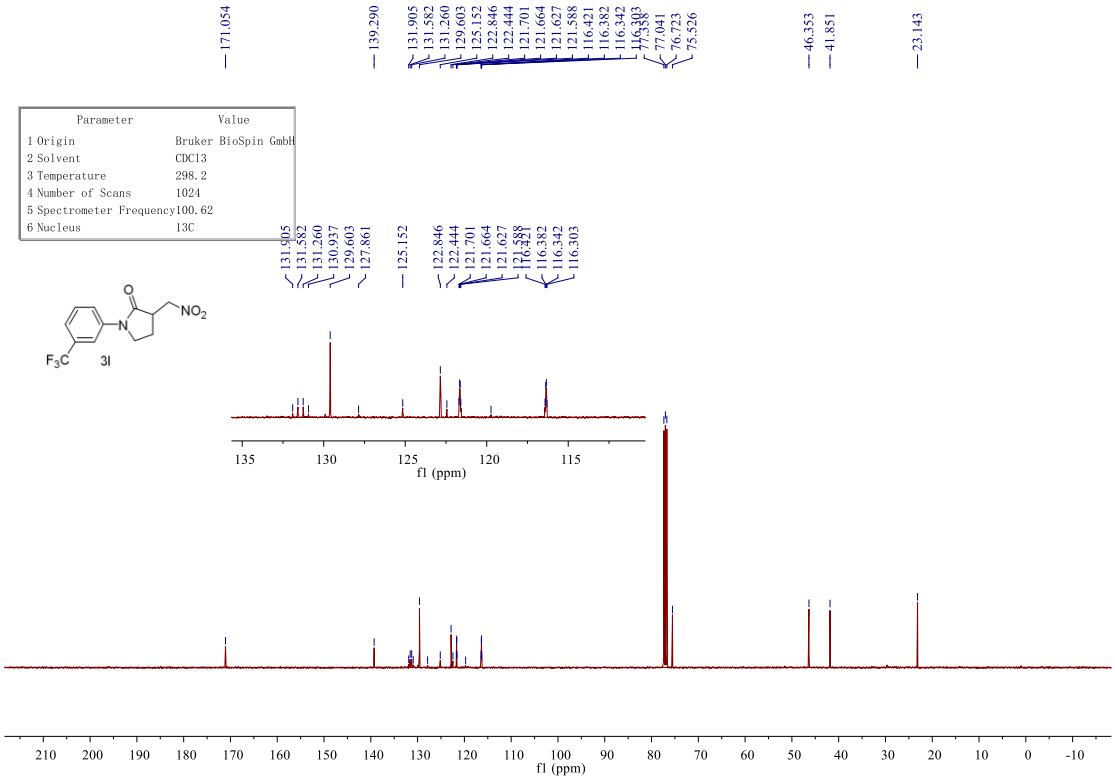


3k



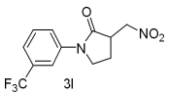

7.877
7.874
7.850
7.532
7.512
7.492
7.451
7.431
7.261
4.935
4.925
4.899
4.890
4.666
4.645
4.631
4.610
3.918
3.900
3.895
3.454
3.445
3.428
3.418
2.601
2.585
2.573
2.118
2.109
2.092
2.086
2.069
2.060
2.040
2.037
Parameter Value
1 Origin Bruker BioSpin GmbH
2 Solvent CDCl3
3 Temperature 298.2
4 Number of Scans 2
5 Spectrometer Frequency 400.13
6 Nucleus 1H
O
N
NO2
F3C
3l


1.96
1.12
0.94
1.00
1.04
2.08
0.99
1.01
1.12
f1 (ppm)

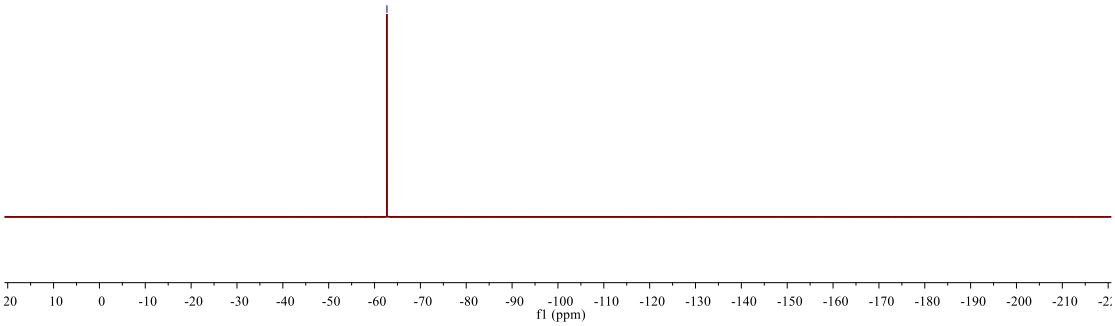

171.054
139.290
131.905
131.582
131.260
130.937
129.603
127.861
125.152
122.846
122.444
121.701
121.664
121.627
121.588
116.421
116.382
116.342
116.303
77.041
76.723
75.526
46.353
41.851
23.143
Parameter Value
1 Origin Bruker BioSpin GmbH
2 Solvent CDCl3
3 Temperature 298.2
4 Number of Scans 1024
5 Spectrometer Frequency 100.62
6 Nucleus 13C
O
N
NO2
F3C
3l
135
130
125
120
115
f1 (ppm)
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
-10
f1 (ppm)

Parameter	Value
1 Origin	Bruker BioSpin GmbH
2 Solvent	CDCl3
3 Temperature	298.2
4 Number of Scans	4
5 Spectrometer Frequency	376.52
6 Nucleus	19F

-62.705



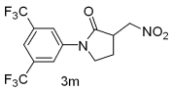
3l



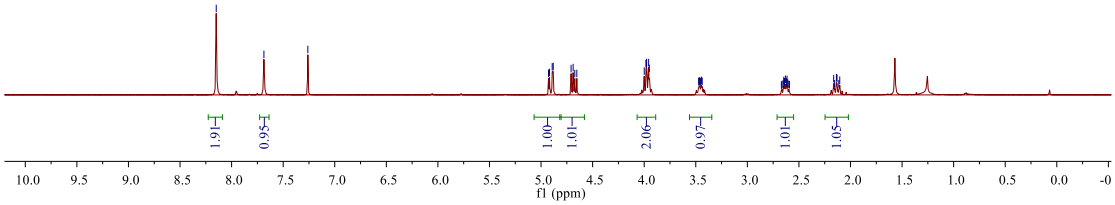
8.149 7.687 7.261

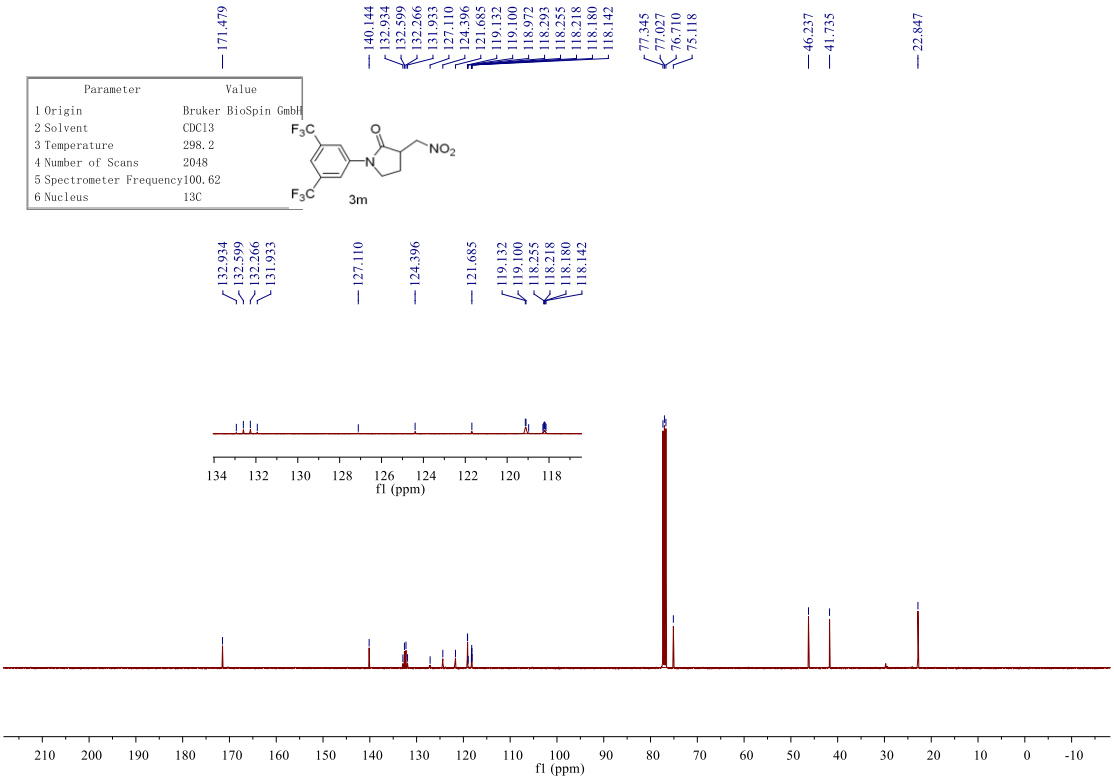
4.928 4.918 4.892 4.882 4.709 4.689 4.673 4.653 3.982 3.976 3.957 3.468 3.462 3.445 3.438 2.647 2.631 2.621 2.615 2.163 2.154 2.136 2.131 2.113 2.104

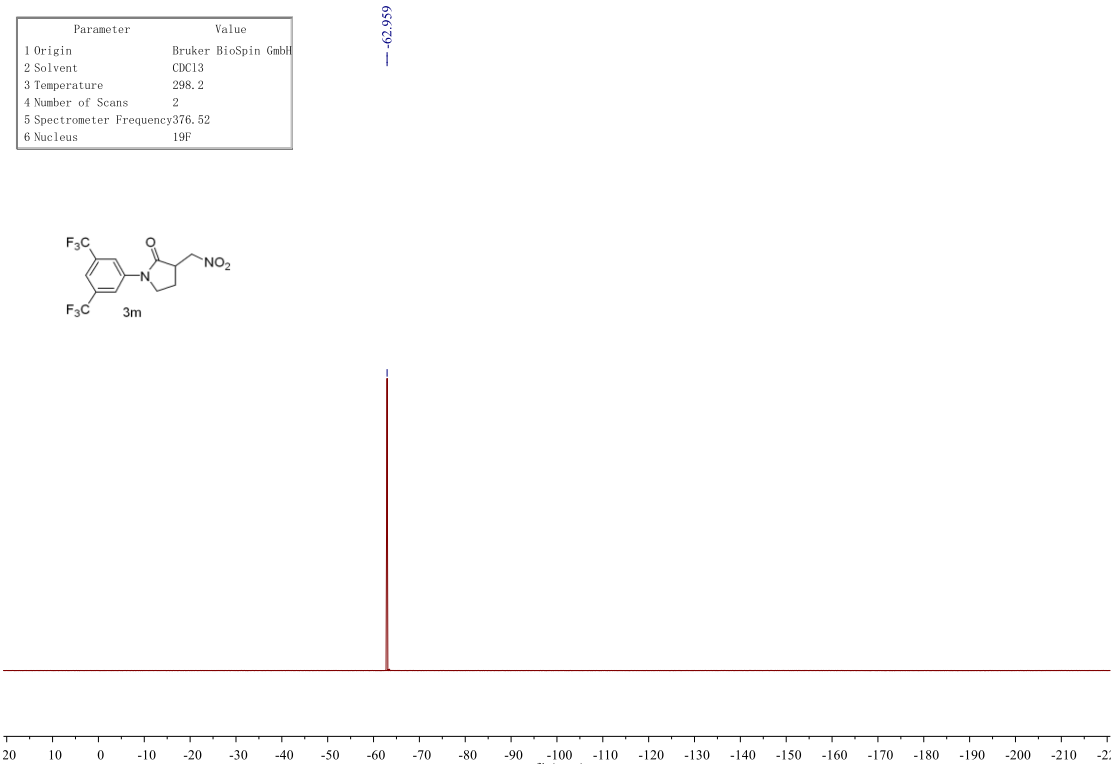
Parameter	Value
1 Origin	Bruker BioSpin GmbH
2 Solvent	CDCl3
3 Temperature	298.1
4 Number of Scans	2
5 Spectrometer Frequency	400.15
6 Nucleus	1H

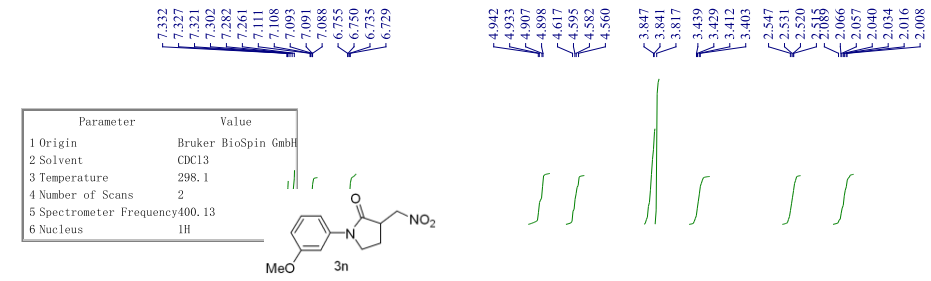


3m



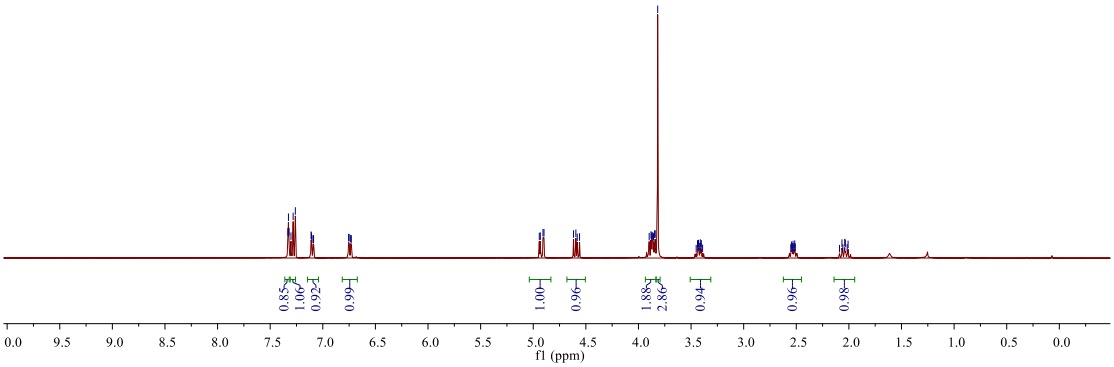

171.479
140.144
132.934
132.599
132.266
131.933
127.110
124.396
121.685
119.132
119.100
118.972
118.293
118.255
118.218
118.180
118.142
77.345
77.027
76.710
75.118
46.237
41.735
22.847
Parameter Value
1 Origin Bruker BioSpin GmbH
2 Solvent CDCl3
3 Temperature 298.2
4 Number of Scans 2048
5 Spectrometer Frequency 100.62
6 Nucleus 13C
3m
f1 (ppm)


-62.959
Parameter Value
1 Origin Bruker BioSpin GmbH
2 Solvent CDCl3
3 Temperature 298.2
4 Number of Scans 2
5 Spectrometer Frequency 376.52
6 Nucleus 19F
3m
f1 (ppm)

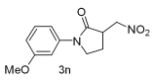


Parameter	Value
1 Origin	Bruker BioSpin GmbH
2 Solvent	CDCl3
3 Temperature	298.1
4 Number of Scans	2
5 Spectrometer Frequency	400.13
6 Nucleus	1H

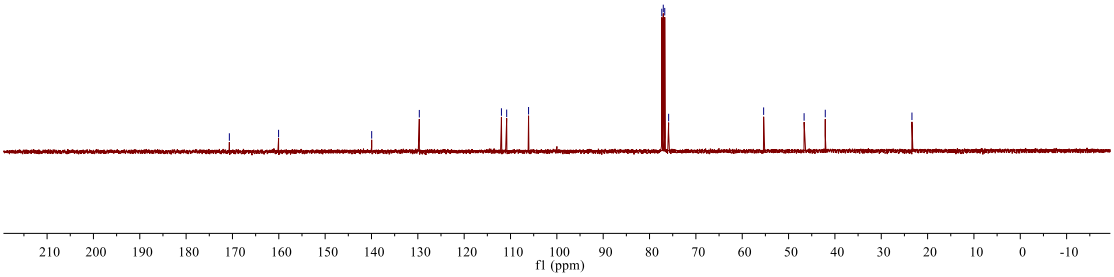
3n

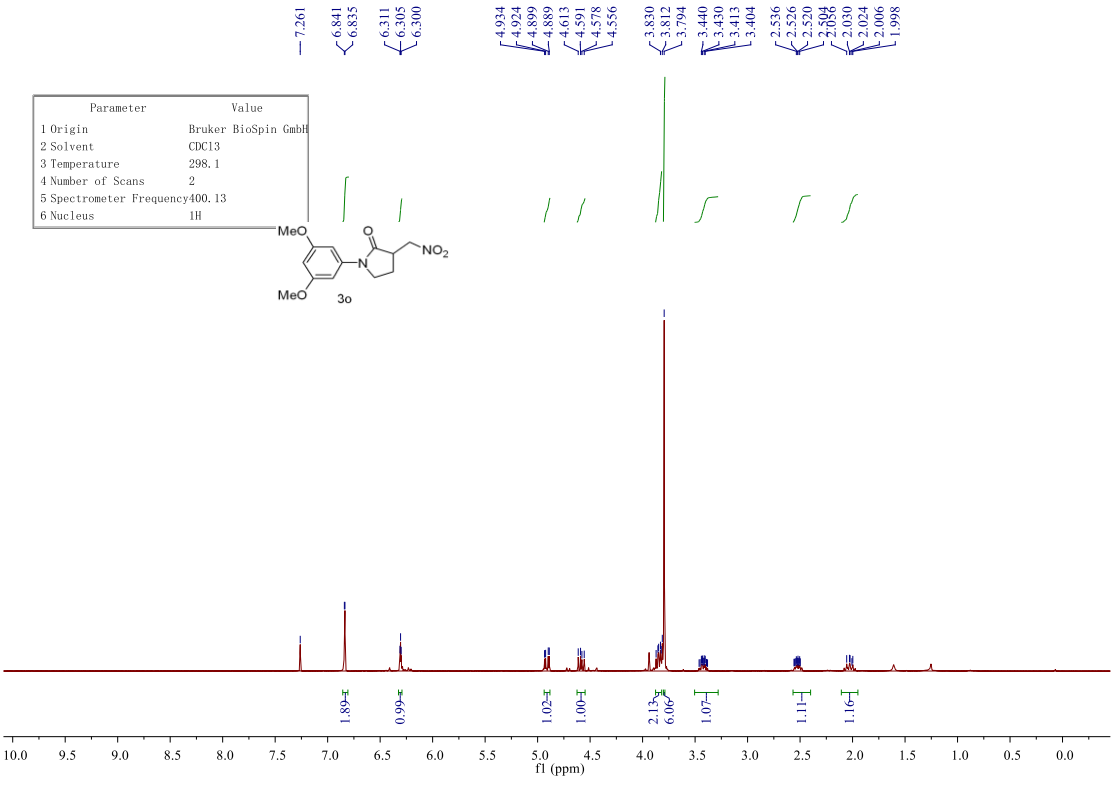


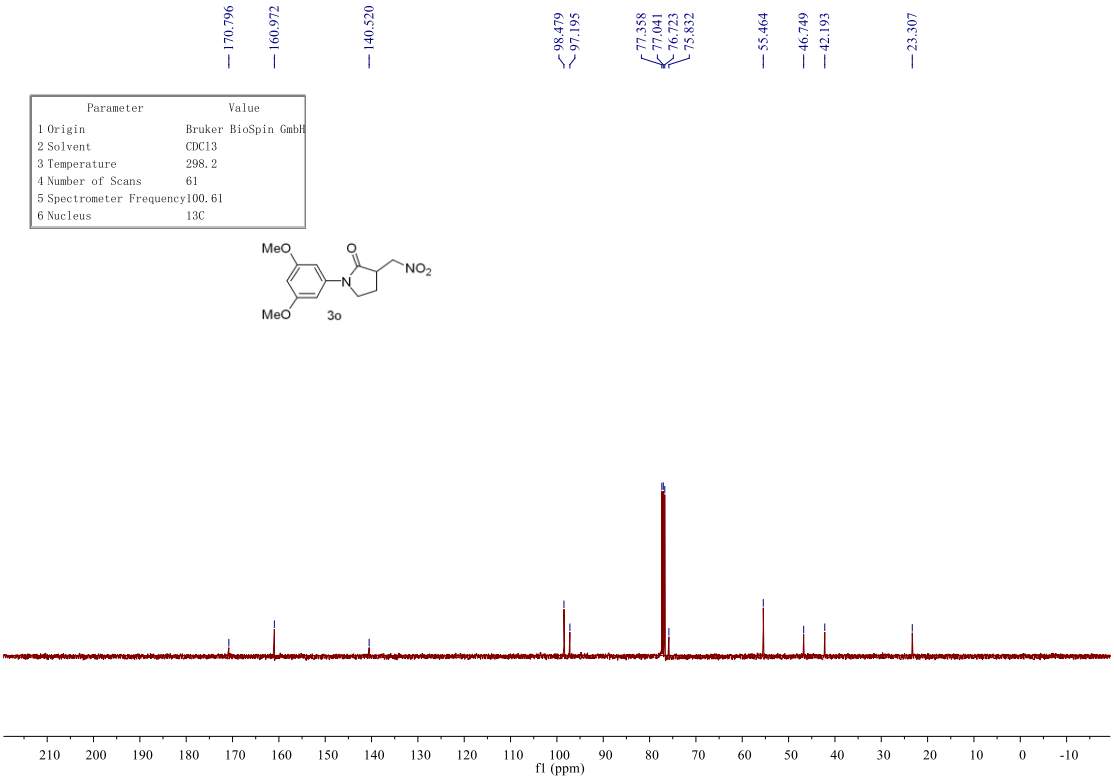
Parameter	Value
1 Origin	Bruker BioSpin GmbH
2 Solvent	CDCl3
3 Temperature	298.1
4 Number of Scans	82
5 Spectrometer Frequency	100.62
6 Nucleus	13C

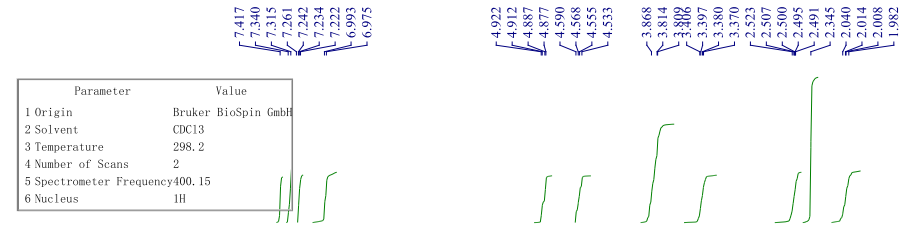


3n

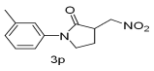


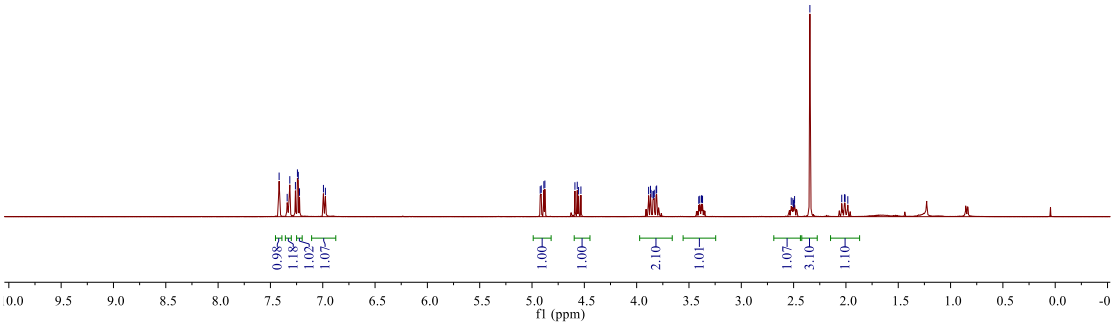

7.261
6.841
6.835
6.311
6.305
6.300
4.934
4.924
4.899
4.889
4.613
4.591
4.578
4.556
3.830
3.812
3.794
3.440
3.430
3.413
3.404
2.536
2.526
2.520
2.504
2.056
2.030
2.024
2.006
1.998
Parameter Value
1 Origin Bruker BioSpin GmbH
2 Solvent CDCl3
3 Temperature 298.1
4 Number of Scans 2
5 Spectrometer Frequency 400.13
6 Nucleus 1H
MeO
MeO
O
N
NO2
3o
1.89
0.99
1.02
1.00
2.13
6.06
1.07
1.11
1.16
f1 (ppm)


170.796
160.972
140.520
98.479
97.195
77.358
77.041
76.723
75.832
55.464
46.749
42.193
23.307
Parameter Value
1 Origin Bruker BioSpin GmbH
2 Solvent CDCl3
3 Temperature 298.2
4 Number of Scans 61
5 Spectrometer Frequency 100.61
6 Nucleus 13C
MeO
MeO
O
N
NO2
3o
f1 (ppm)

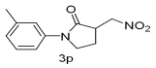


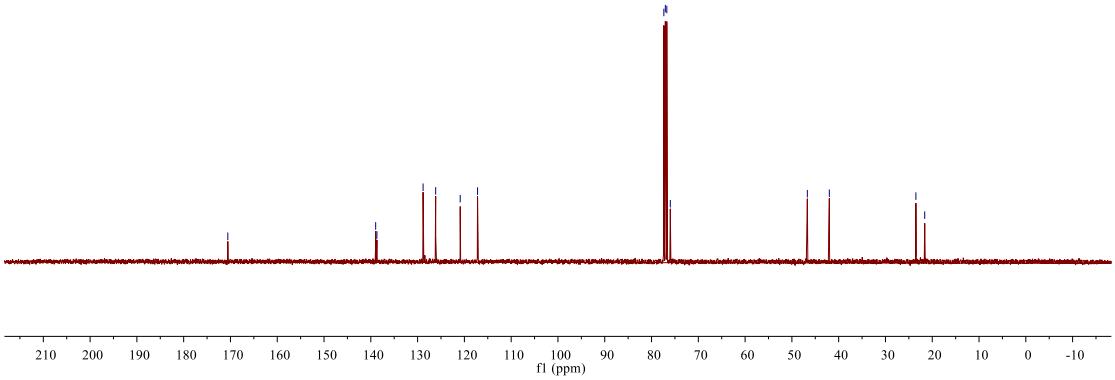
Parameter	Value
1 Origin	Bruker BioSpin GmbH
2 Solvent	CDCl3
3 Temperature	298.2
4 Number of Scans	2
5 Spectrometer Frequency	400.15
6 Nucleus	1H

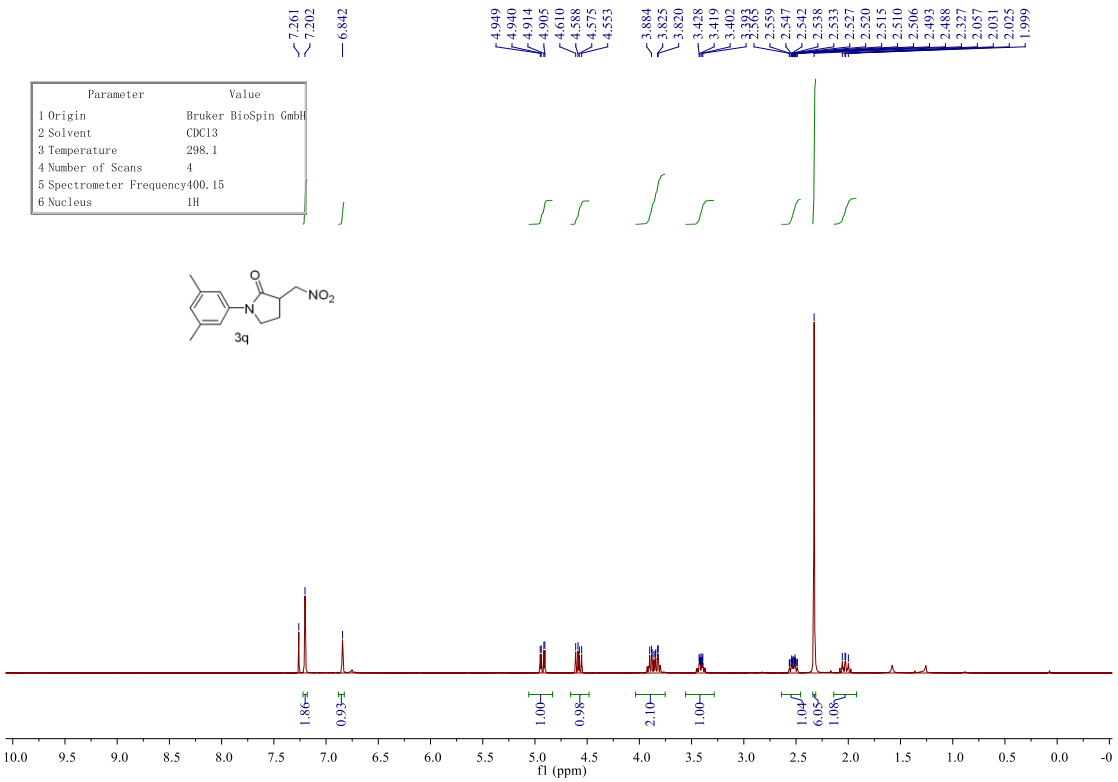


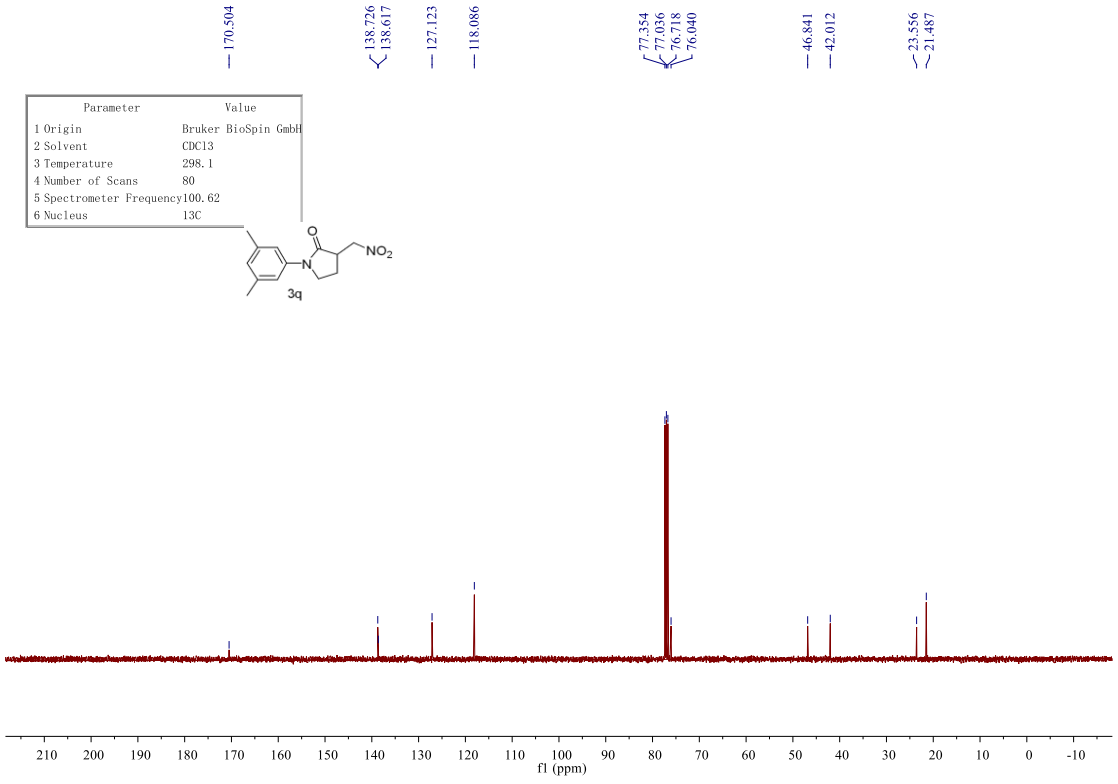


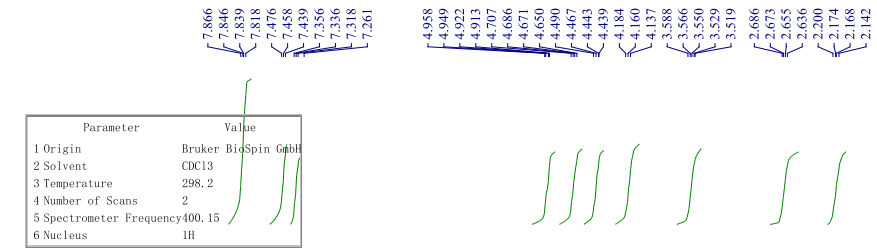
Parameter	Value
1 Origin	Bruker BioSpin GmbH
2 Solvent	CDCl3
3 Temperature	298.2
4 Number of Scans	135
5 Spectrometer Frequency	100.62
6 Nucleus	13C



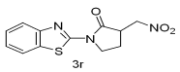



7.261
7.202
6.842
4.949
4.940
4.914
4.905
4.610
4.588
4.575
4.553
3.884
3.825
3.820
3.428
3.419
3.402
2.559
2.547
2.542
2.538
2.533
2.527
2.520
2.515
2.510
2.506
2.493
2.488
2.327
2.057
2.031
2.025
1.999
Parameter Value
1 Origin Bruker BioSpin GmbH
2 Solvent CDCl3
3 Temperature 298.1
4 Number of Scans 4
5 Spectrometer Frequency 400.15
6 Nucleus 1H
NO2
3q
1.86
0.93
1.00
0.98
2.10
1.00
1.04
6.05
1.08
f1 (ppm)


170.504
138.726
138.617
127.123
118.086
77.354
77.036
76.718
76.040
46.841
42.012
23.556
21.487
Parameter Value
1 Origin Bruker BioSpin GmbH
2 Solvent CDCl3
3 Temperature 298.1
4 Number of Scans 80
5 Spectrometer Frequency 100.62
6 Nucleus 13C
NO2
3q
f1 (ppm)



Parameter	Value
1 Origin	Bruker BioSpin GmbH
2 Solvent	CDCl3
3 Temperature	298.2
4 Number of Scans	2
5 Spectrometer Frequency	400.15
6 Nucleus	1H



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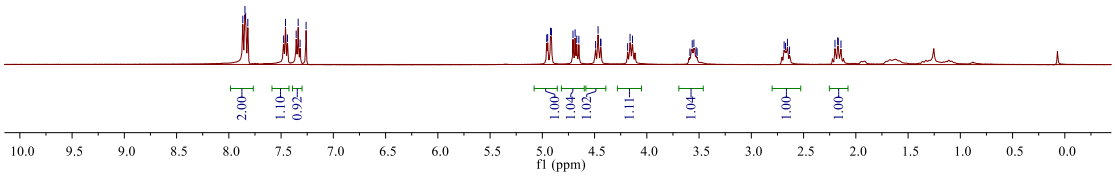
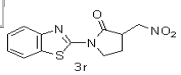


Parameter	Value
1 Origin	Bruker BioSpin GmbH
2 Solvent	CDCl3
3 Temperature	298.2
4 Number of Scans	80
5 Spectrometer Frequency	100.62
6 Nucleus	13C



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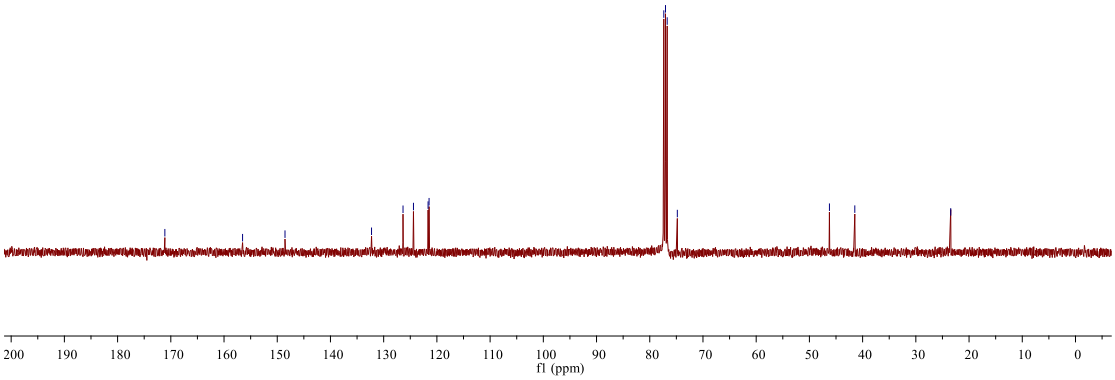
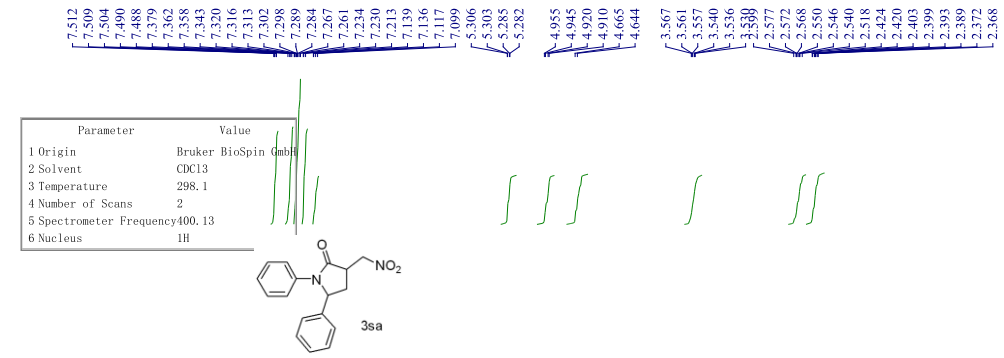
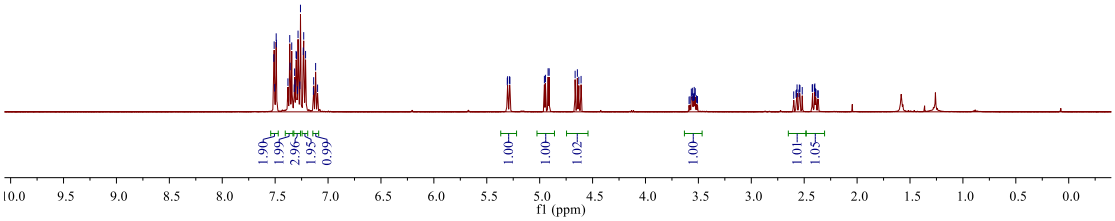


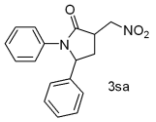


Parameter	Value
1 Origin	Bruker BioSpin GmbH
2 Solvent	CDCl3
3 Temperature	298.1
4 Number of Scans	2
5 Spectrometer Frequency	400.13
6 Nucleus	1H

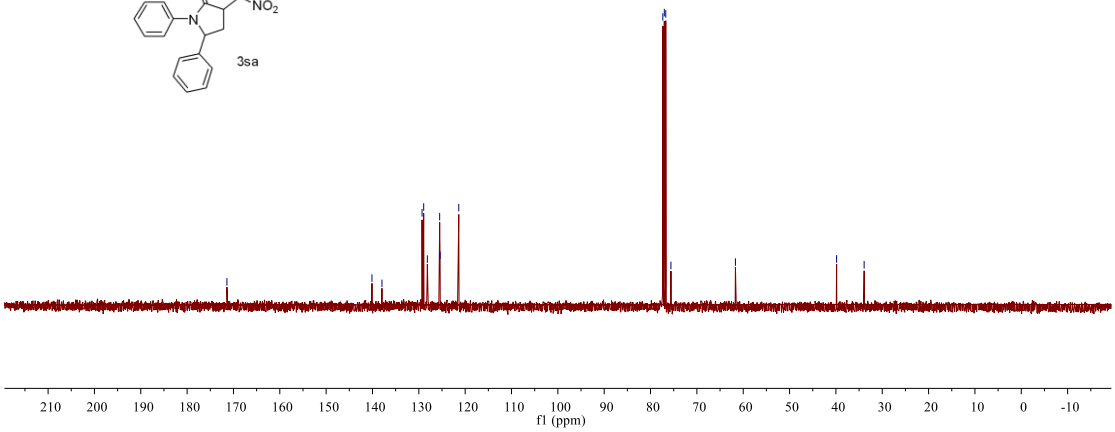
3sa

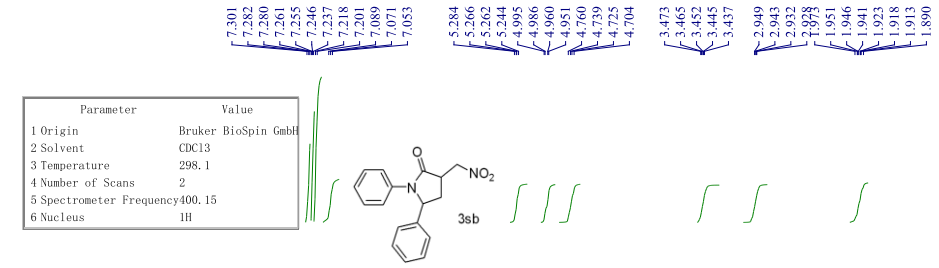


Parameter	Value
1 Origin	Bruker BioSpin GmbH
2 Solvent	CDCl3
3 Temperature	298.1
4 Number of Scans	50
5 Spectrometer Frequency	100.62
6 Nucleus	13C



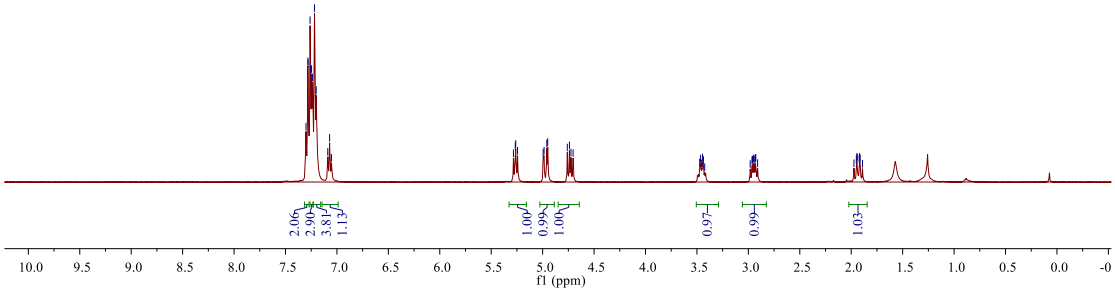
3sa



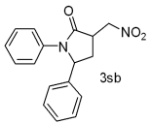


Parameter	Value
1 Origin	Bruker BioSpin GmbH
2 Solvent	CDCl3
3 Temperature	298.1
4 Number of Scans	2
5 Spectrometer Frequency	400.15
6 Nucleus	1H

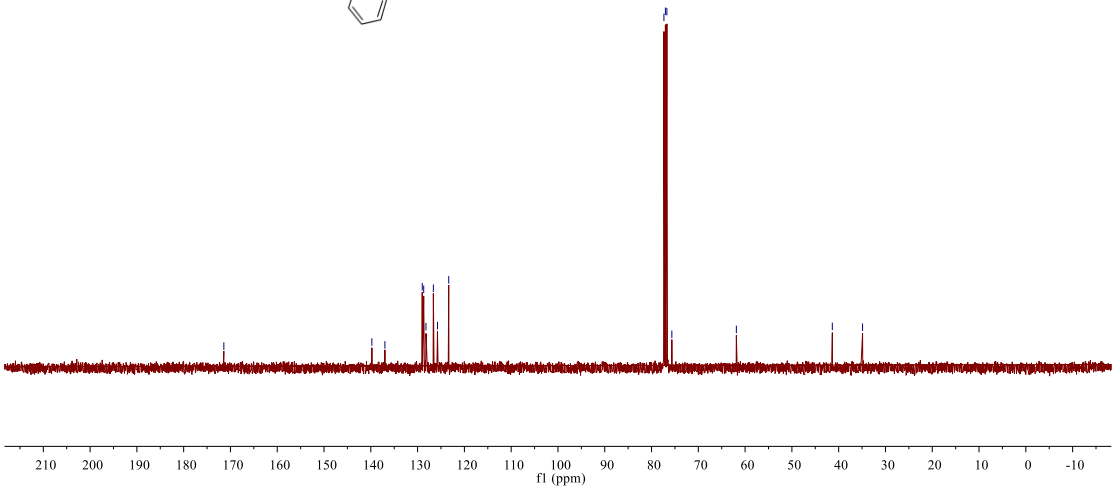
3sb

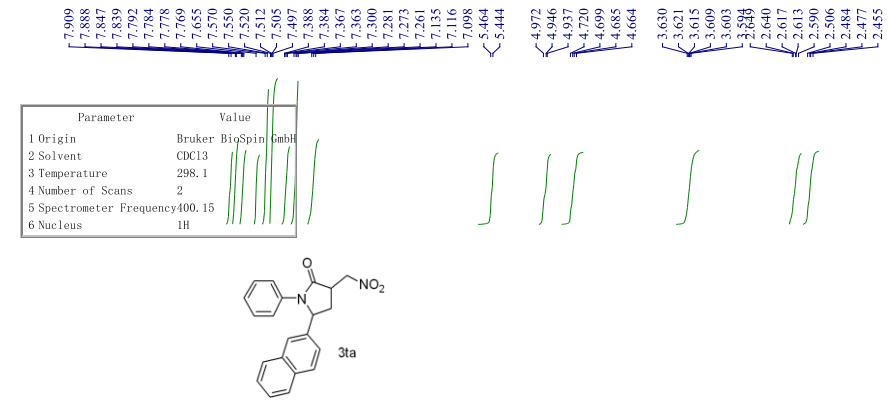


Parameter	Value
1 Origin	Bruker BioSpin GmbH
2 Solvent	CDCl3
3 Temperature	298.1
4 Number of Scans	82
5 Spectrometer Frequency	100.62
6 Nucleus	13C



3sb

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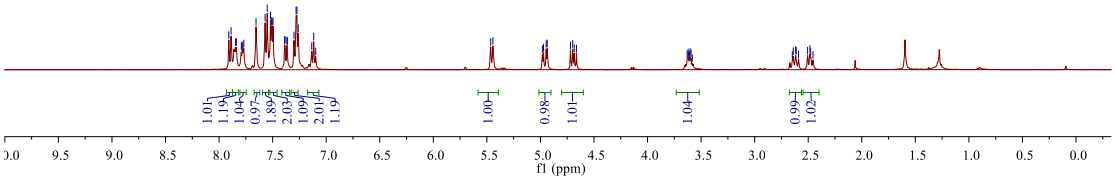
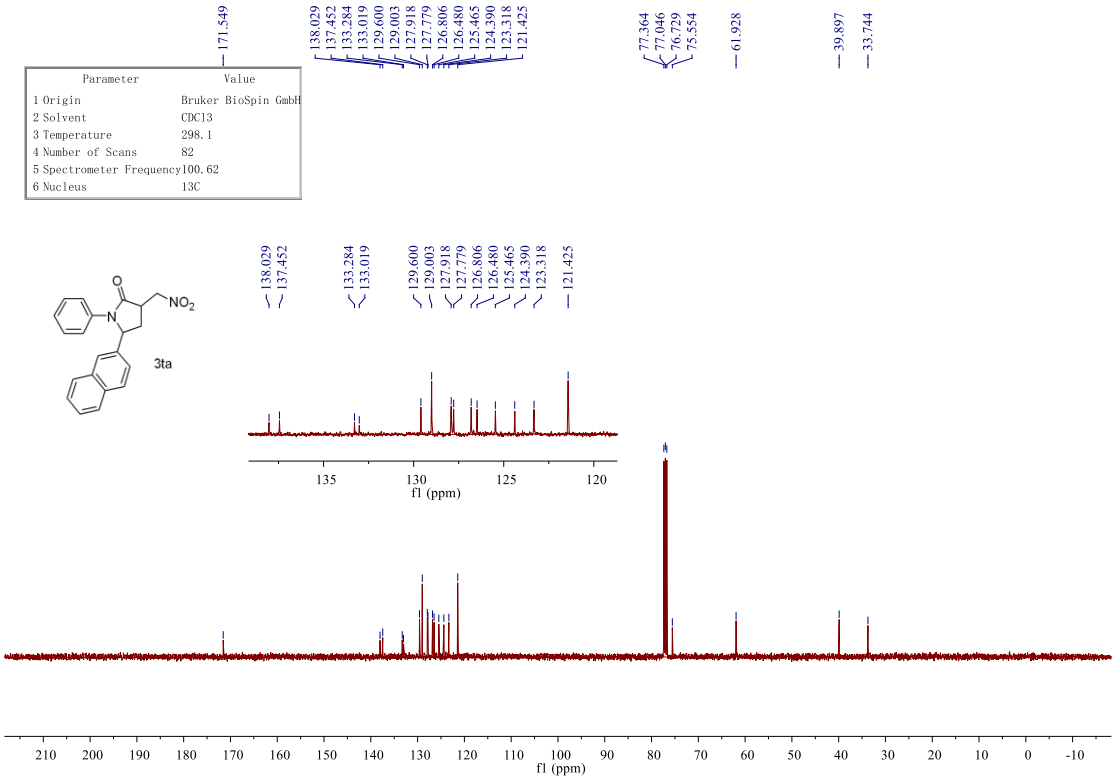
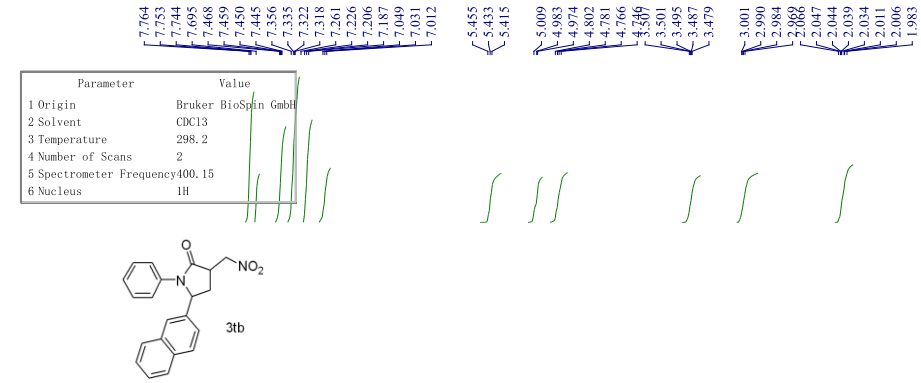
7.888
7.847
7.839
7.792
7.784
7.778
7.769
7.655
7.570
7.550
7.520
7.512
7.505
7.497
7.388
7.384
7.367
7.363
7.300
7.281
7.273
7.261
7.135
7.116
7.098
5.464
5.444
4.972
4.946
4.937
4.720
4.699
4.685
4.664
3.630
3.621
3.615
3.609
3.603
3.594
2.640
2.617
2.613
2.590
2.506
2.484
2.477
2.455
Parameter Value
1 Origin Bruker BioSpin GmbH
2 Solvent CDCl3
3 Temperature 298.1
4 Number of Scans 2
5 Spectrometer Frequency 400.15
6 Nucleus 1H
O
N
NO2
3ta


1.01
1.19
1.04
0.97
1.89
2.03
1.09
2.01
1.19
1.00
0.98
1.01
1.04
0.99
1.02
f1 (ppm)


171.549
138.029
137.452
133.284
133.019
129.600
129.003
127.918
127.779
126.806
126.480
125.465
124.390
123.318
121.425
77.364
77.046
76.729
75.554
61.928
39.897
33.744
Parameter Value
1 Origin Bruker BioSpin GmbH
2 Solvent CDCl3
3 Temperature 298.1
4 Number of Scans 82
5 Spectrometer Frequency 100.62
6 Nucleus 13C
O
N
NO2
3ta
f1 (ppm)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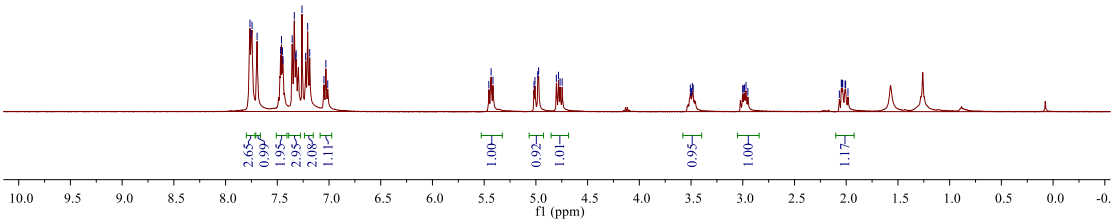
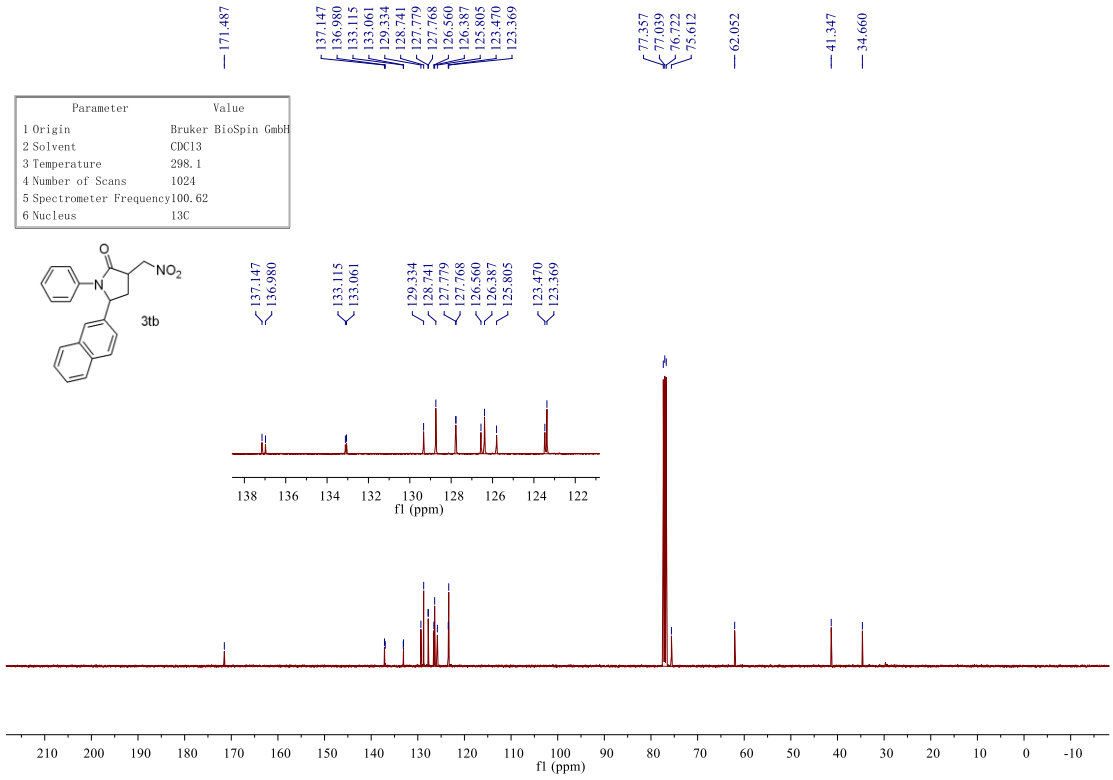
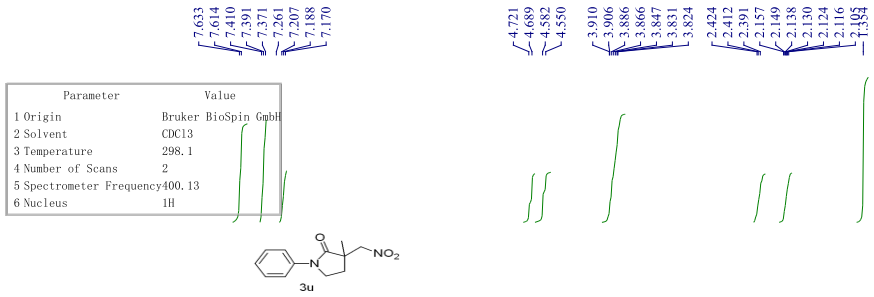
7.753
7.744
7.695
7.468
7.459
7.450
7.445
7.356
7.335
7.322
7.318
7.261
7.226
7.206
7.187
7.049
7.031
7.012
5.455
5.433
5.415
5.009
4.983
4.974
4.802
4.781
4.766
4.746
3.507
3.501
3.495
3.487
3.479
3.001
2.990
2.984
2.969
2.966
2.047
2.044
2.039
2.034
2.011
2.006
1.983
Parameter Value
1 Origin Bruker BioSpin GmbH
2 Solvent CDCl3
3 Temperature 298.2
4 Number of Scans 2
5 Spectrometer Frequency 400.15
6 Nucleus 1H
NO2
3tb


2.65
0.99
1.95
2.95
2.08
1.11
1.00
0.92
1.01
0.95
1.00
1.17
f1 (ppm)


171.487
137.147
136.980
133.115
133.061
129.334
128.741
127.779
127.768
126.560
126.387
125.805
123.470
123.369
77.357
77.039
76.722
75.612
62.052
41.347
34.660
Parameter Value
1 Origin Bruker BioSpin GmbH
2 Solvent CDCl3
3 Temperature 298.1
4 Number of Scans 1024
5 Spectrometer Frequency 100.62
6 Nucleus 13C
NO2
3tb
f1 (ppm)
f1 (ppm)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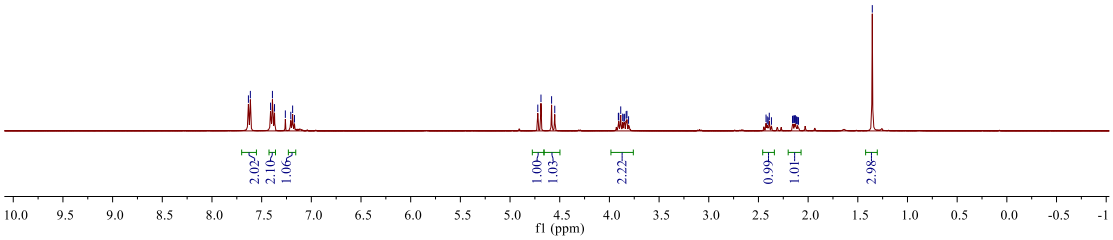
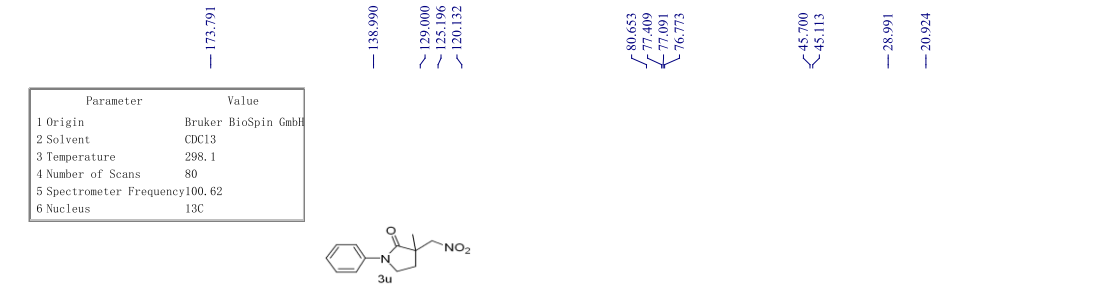
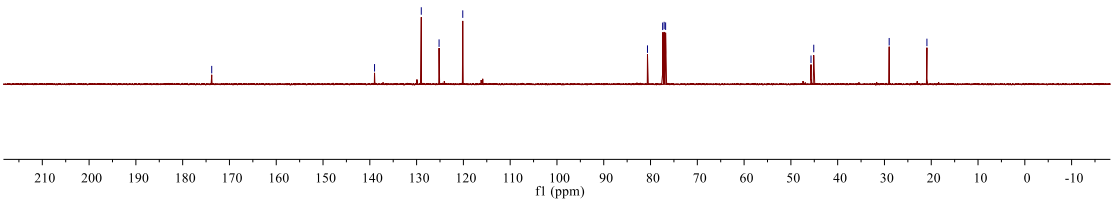
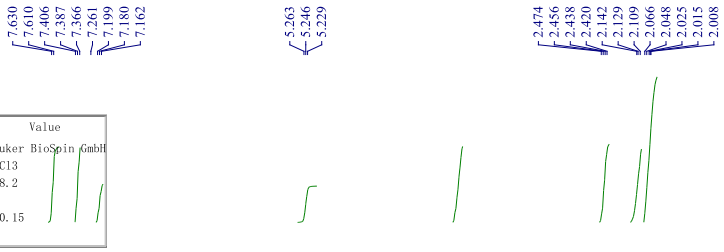
7.614
7.410
7.391
7.371
7.261
7.207
7.188
7.170
4.721
4.689
4.582
4.550
3.910
3.906
3.886
3.866
3.847
3.831
3.824
2.424
2.412
2.391
2.157
2.149
2.138
2.130
2.124
2.116
2.105
1.354
Parameter Value
1 Origin Bruker BioSpin GmbH
2 Solvent CDCl3
3 Temperature 298.1
4 Number of Scans 2
5 Spectrometer Frequency 400.13
6 Nucleus 1H
O
N
NO2
3u

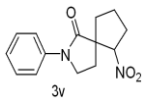

2.02
2.10
1.06
1.00
1.03
2.22
0.99
1.01
2.98
10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5 -1
f1 (ppm)


173.791
138.990
129.000
125.196
120.132
80.653
77.409
77.091
76.773
45.700
45.113
28.991
20.924
Parameter Value
1 Origin Bruker BioSpin GmbH
2 Solvent CDCl3
3 Temperature 298.1
4 Number of Scans 80
5 Spectrometer Frequency 100.62
6 Nucleus 13C
O
N
NO2
3u

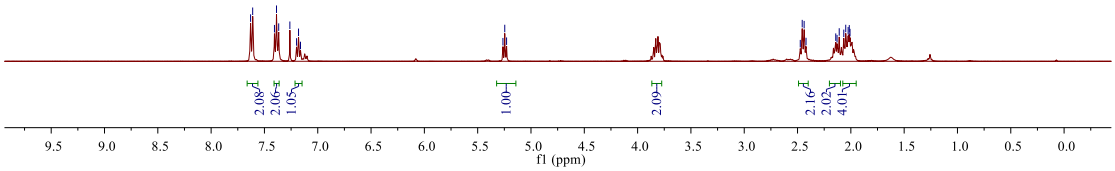

210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)



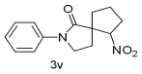
Parameter	Value
1 Origin	Bruker BioSpin GmbH
2 Solvent	CDCl3
3 Temperature	298.2
4 Number of Scans	2
5 Spectrometer Frequency	400.15
6 Nucleus	1H



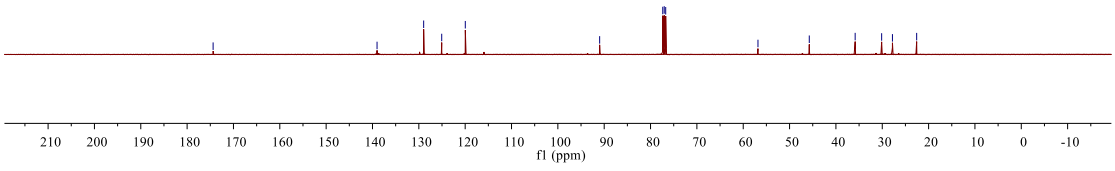
3v

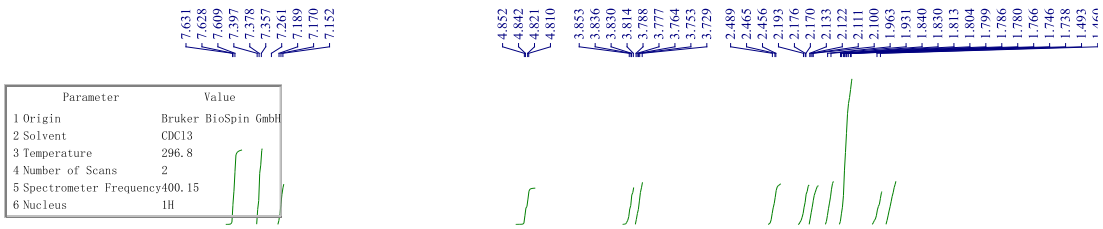


Parameter	Value
1 Origin	Bruker BioSpin GmbH
2 Solvent	CDCl3
3 Temperature	298.2
4 Number of Scans	1024
5 Spectrometer Frequency	100.62
6 Nucleus	13C

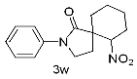


3v

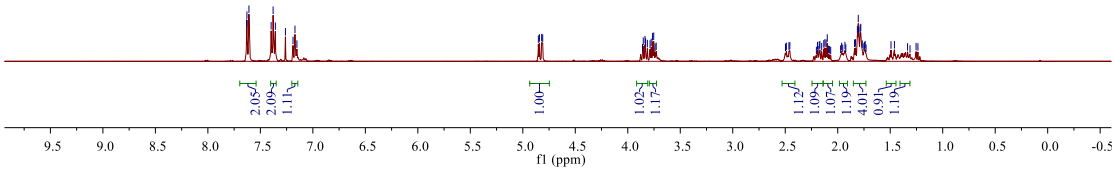




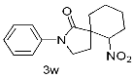
Parameter	Value
1 Origin	Bruker BioSpin GmbH
2 Solvent	CDCl3
3 Temperature	296.8
4 Number of Scans	2
5 Spectrometer Frequency	400.15
6 Nucleus	1H



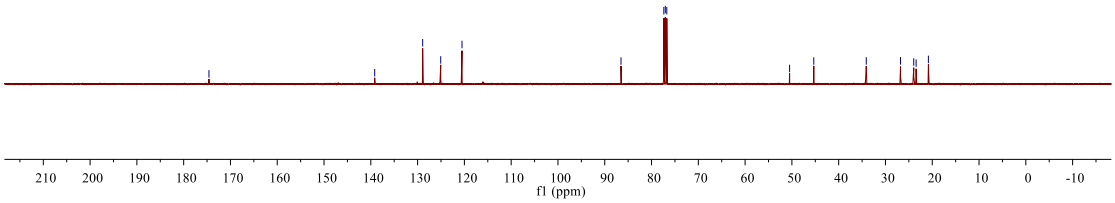
3w

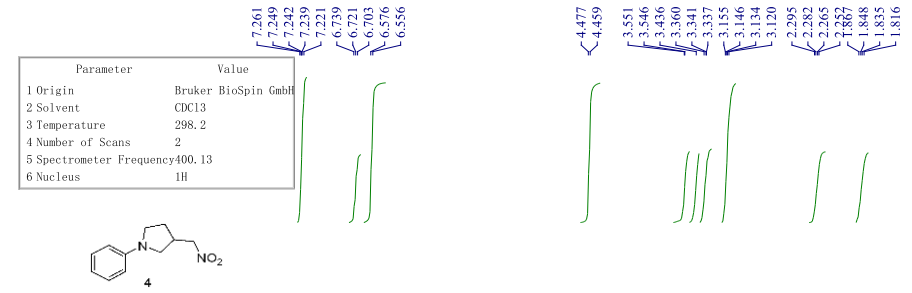


Parameter	Value
1 Origin	Bruker BioSpin GmbH
2 Solvent	CDCl3
3 Temperature	297.5
4 Number of Scans	150
5 Spectrometer Frequency	100.62
6 Nucleus	13C



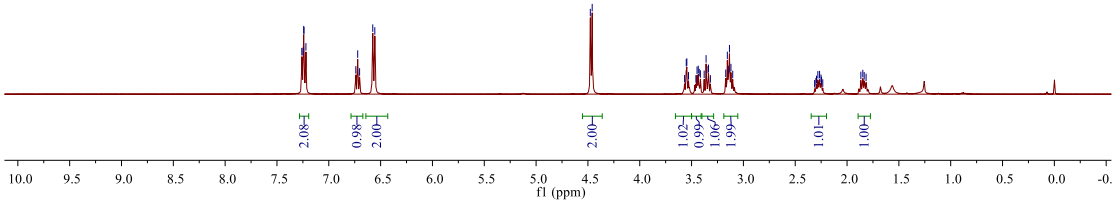
3w



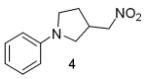


Parameter	Value
1 Origin	Bruker BioSpin GmbH
2 Solvent	CDCl3
3 Temperature	298.2
4 Number of Scans	2
5 Spectrometer Frequency	400.13
6 Nucleus	1H

4



Parameter	Value
1 Origin	Bruker BioSpin GmbH
2 Solvent	CDCl3
3 Temperature	298.2
4 Number of Scans	80
5 Spectrometer Frequency	100.62
6 Nucleus	13C



4