Supporting Information

Atom- and step-economic synthesis of multiply substituted butenolides from keto Acids and terminal alkynes promoted by combined acids

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1. General experimental details

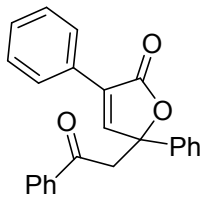
All reactions were maintained under air unless otherwise stated. Commercially available reagents were used without further purification. Infrared (FT-IR) spectra were recorded on a BRUKER VERTEX 70, ν_{max} in cm^{-1}. ^{1}H-NMR spectra were recorded on a BRUKER AVANCE III HD (400 MHz) spectrometer. 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 Agelent 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.

The starting materials keto acids were prepared according to the reported procedures.[1]

2. General procedure for the synthesis of butenolides

Keto acid (0.3 mmol, 1.0 equiv.) and *p*-toluenesulfonic acid monohydrate (0.6 mmol, 2.0 equiv.) were added to a test tube. Then chlorobenzene (2 mL), alkyne (0.9 mmol, 3.0 equiv.), and boron trifluoride etherate (0.06 mmol, 0.2 equiv.) were added to the test tube in order. The reaction mixture was stirred at 70 ^{o}C under an air atmosphere until the starting material had been consumed as determined by TLC. The reaction was cooled down, and the solvent was removed under vacuum. The residue was subjected to flash column chromatography on silica gel (eluent: ethyl acetate/ petroleum ether) to afford the desired butenolide.

3. Characterization of products



3a: yellow oil. ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.35 (s, 1H), 7.91-7.86 (m, 4H), 7.59-7.53 (m, 3H), 7.46-7.30 (m, 8H), 4.13 (d, *J* = 16.4 Hz, 1H), 3.69 (d, *J* = 16.4 Hz, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 195.1, 169.8, 149.9, 138.3, 136.1, 133.3, 130.1, 129.0, 128.9, 128.4, 128.3, 128.2, 128.1, 127.8, 126.8, 125.4, 85.7, 48.3. FT-IR: ν (cm^{-1}) 3059, 2918, 2850, 1735, 1683, 1596. HRMS [ESI] calcd for $C_{24}H_{19}O_3$ $[M+H]^+$ 355.1334, found 355.1328. ESI $[M+Na]^+$ 377.0.

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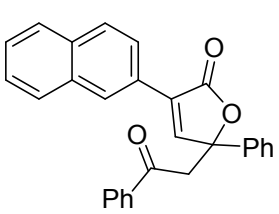
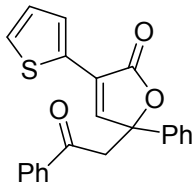
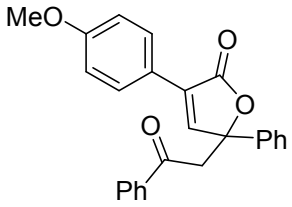
[1] a) X. Beebe, A. M. Nilius, P. J. Merta, N. B. Soni, M. H. Bui, R. Wagner and B. A. Beutel, *Bioorg. Med. Chem. Lett.* **2003**, *13*, 3133-3136; b) J. Zhuang, C. Wang, F. Xie and W. Zhang, *Tetrahedron* **2009**, *65*, 9797-9800; c) E. Johannes, R. Horbert, J. Schlosser, D. Schmidt and C. Peifer, *Tetrahedron Lett.* **2013**, *54*, 4067-4072.



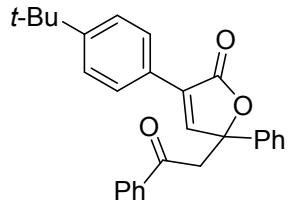
3b: yellow solid, m.p. 138-140 °C. ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.60 (s, 1H), 8.47 (s, 1H), 7.93-7.87 (m, 3H), 7.86-7.81 (m, 3H), 7.60-7.54 (m, 3H), 7.53-7.48 (m, 2H), 7.47-7.37 (m, 4H), 7.36-7.31 (m, 1H), 4.16 (d, J = 16.4 Hz, 1H), 3.72 (d, J = 16.4 Hz, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 195.2, 169.9, 149.9, 138.3, 136.1, 133.3, 133.1, 132.7, 129.9, 128.4, 128.3, 128.2, 127.9, 127.8, 127.2, 126.7, 126.5, 126.1, 126.0 , 125.4, 123.8, 85.8, 48.3. FT-IR: ν (cm^{-1}) 3057, 2927, 2853, 1750, 1683, 1595. HRMS [ESI] calcd for $C_{28}H_{21}O_3$ $[M+H]^+$ 407.1647, found 407.1647. ESI $[M+H]^+$ 407.1.



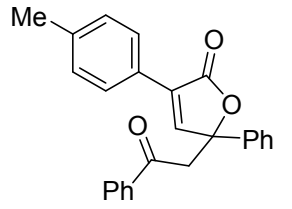
3c: brown solid, m.p. 124-126 °C. ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.18 (s, 1H), 7.89-7.85 (m, 2H), 7.78 (dd, J = 3.6, 0.8 Hz, 1H), 7.60-7.50 (m, 3H), 7.46-7.41 (m, 2H), 7.40-7.29 (m, 4H), 7.08 (dd, J = 5.2, 3.6 Hz, 1H), 4.16 (d, J = 16.8 Hz, 1H), 3.66 (d, J = 16.8 Hz, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 195.0, 169.0, 145.8, 138.1, 136.0, 133.3, 130.8, 128.3, 128.3, 128.2, 127.8, 127.4, 127.3, 127.1, 125.4, 125.2, 86.5, 48.3. FT-IR: ν (cm^{-1}) 3029, 2958, 2853, 1757, 1633, 1579. HRMS [ESI] calcd for $C_{22}H_{16}O_3SNa$ $[M+Na]^+$ 383.0718, found 383.0705. ESI $[M+H]^+$ 361.1.



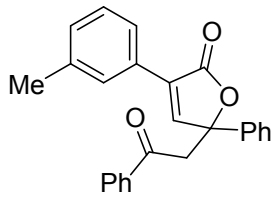
3d: yellow solid, m.p. 124-126 °C. ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.23 (s, 1H), 7.91-7.82 (m, 4H), 7.58-7.51 (m, 3H), 7.46-7.40 (m, 2H), 7.39-7.34 (m, 2H), 7.33-7.28 (m, 1H), 6.95-6.90 (m, 2H), 4.12 (d, J = 16.4 Hz, 1H), 3.81 (s, 3H), 3.67 (d, J = 16.4 Hz, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 195.6, 170.6, 160.5, 148.1, 138.9, 136.6, 133.7, 129.9, 128.8, 128.8, 128.7, 128.6, 128.3, 125.9, 121.9, 114.1, 86.1, 55.4, 48.8. FT-IR: ν (cm^{-1}) 3076, 2922, 2850, 1754, 1681, 1573. HRMS [ESI] calcd for $C_{25}H_{20}O_4Na$ $[M+Na]^+$ 407.1259, found 407.1253. ESI $[M+H]^+$ 407.1.



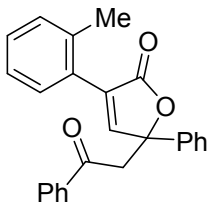
3e: yellow solid, m.p. 163-165 °C. ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.32 (s, 1H), 7.91-7.87 (m, 2H), 7.87-7.82 (m, 2H), 7.59-7.53 (m, 3H), 7.47-7.42 (m, 4H), 7.40-7.29 (m, 3H), 4.14 (d, J = 16.4 Hz, 1H), 3.70 (d, J = 16.4 Hz, 1H), 1.34 (s, 9H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 195.6, 170.5, 152.7, 149.6, 138.9, 136.6, 133.7, 130.4, 128.8, 128.7, 128.6, 128.3, 127.0, 126.5, 125.9, 125.6, 86.2, 48.7, 34.8, 31.2. FT-IR: ν (cm^{-1}) 3058, 2962, 2868, 1753, 1681, 1577. HRMS [ESI] calcd for $C_{28}H_{27}O_3$ $[M+H]^+$ 411.1960, found 411.1950. ESI $[M+H]^+$ 411.1.



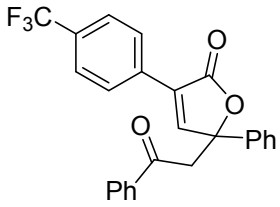
3f: yellow solid, m.p. 113-115 °C. ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.31 (s, 1H), 7.91-7.86 (m, 2H), 7.80 (d, J = 8.0 Hz, 2H), 7.58-7.53 (m, 3H), 7.47-7.41 (m, 2H), 7.40-7.35 (m, 2H), 7.35-7.29 (m, 1H), 7.22 (d, J = 8.0 Hz, 2H), 4.14 (d, J = 16.4 Hz, 1H), 3.68 (d, J = 16.4 Hz, 1H), 2.38 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 195.2, 170.0, 148.9, 139.1, 138.4, 136.1, 133.3, 130.0, 128.9, 128.3, 128.3, 128.1, 127.8, 126.7, 126.0, 125.4, 85.7, 48.3, 21.0. FT-IR: ν (cm^{-1}) 3062, 2922, 2856, 1757, 1682, 1578. HRMS [ESI] calcd for $C_{25}H_{21}O_3$ $[M+Na]^+$ 369.1491, found 369.1486; ESI $[M+Na]^+$ 391.7.



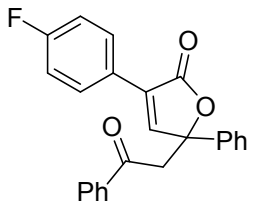
3g: yellow oil. ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.34 (s, 1H), 7.92-7.86 (m, 2H), 7.72-7.68 (m, 2H), 7.59-7.53 (m, 3H), 7.47-7.41 (m, 2H), 7.41-7.36 (m, 2H), 7.35-7.28 (m, 2H), 7.21 (d, J = 7.6 Hz, 1H), 4.14 (d, J = 16.4 Hz, 1H), 3.69 (d, J = 16.4 Hz, 1H), 2.39 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 195.2, 169.9, 149.8, 138.3, 137.9, 136.0, 133.3, 130.2, 129.8, 128.8, 128.4, 128.3, 128.2, 128.1, 127.8, 127.4, 125.4, 123.9, 85.7, 48.3, 21.0. FT-IR: ν (cm^{-1}) 3029, 2978, 2857, 1752, 1684, 1579. HRMS [ESI] calcd for $C_{25}H_{20}O_3Na$ $[M+Na]^+$ 391.1310, found 391.1312. ESI $[M+Na]^+$ 391.1.



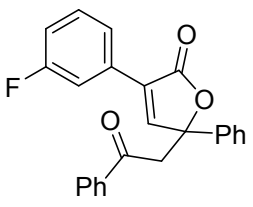
3h: yellow solid, m.p. 106-108 °C. ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.09 (s, 1H), 7.93-7.89 (m, 2H), 7.60-7.53 (m, 3H), 7.48-7.31 (m, 6H), 7.30-7.19 (m, 3H), 4.04 (d, J = 16.0 Hz, 1H), 3.78 (d, J = 16.0 Hz, 1H), 2.33 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 195.1, 170.2, 153.4, 138.5, 136.3, 136.1, 133.3, 131.7, 130.1, 129.2, 128.7, 128.6, 128.4, 128.3, 128.2, 128.0, 125.4, 125.2, 86.3, 48.3, 20.0. FT-IR: ν (cm^{-1}) 3060, 2955, 2838, 1751, 1682, 1511. HRMS [ESI] calcd for $C_{25}H_{21}O_3$ $[M+H]^+$ 369.1491, found 369.1500. ESI $[M+Na]^+$ 391.1.



3i: yellow solid, m.p. 109-111 °C. ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.47 (s, 1H), 8.01 (d, J = 8.0 Hz, 2H), 7.91-7.86 (m, 2H), 7.66 (d, J = 8.4 Hz, 2H), 7.61-7.52 (m, 3H), 7.48-7.42 (m, 2H), 7.42-7.37 (m, 2H), 7.37-7.31 (m, 1H), 4.14 (d, J = 16.8 Hz, 1H), 3.73 (d, J = 16.8 Hz, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 195.5, 169.8, 152.3, 138.3, 136.4, 133.9, 132.7 (q, J_{C-F} = 0.9 Hz), 131.2 (q, J_{C-F} = 32.6 Hz), 129.4, 128.9, 128.8, 128.8, 128.3, 127.6, 125.7, 125.6 (q, J_{C-F} = 3.7 Hz), 123.9 (q, J_{C-F} = 270.6 Hz), 86.5, 48.7; ^{19}F NMR (376 MHz, $CDCl_3$) δ -62.8 (s). FT-IR: ν (cm^{-1}) 3055, 2919, 2850, 1754, 1680, 1578. HRMS [ESI] calcd for $C_{25}H_{18}O_3F_3$ $[M+H]^+$ 423.1208, found 423.1219. ESI $[M+Na]^+$ 445.0.

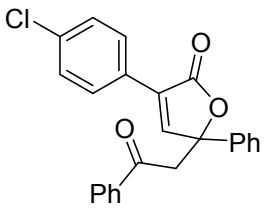


3j: yellow oil. ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.32 (s, 1H), 7.93-7.85 (m, 4H), 7.60-7.52 (m, 3H), 7.47-7.41 (m, 2H), 7.41-7.35 (m, 2H), 7.35-7.30 (m, 1H), 7.13-7.05 (m, 2H), 4.13 (d, J = 16.4 Hz, 1H), 3.70 (d, J = 16.4 Hz, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 195.6, 170.2, 163.3 (d, J_{C-F} = 249.9 Hz), 149.9, 138.6, 136.5, 133.8, 129.5, 129.2 (d, J_{C-F} = 8.3 Hz), 128.9, 128.8, 128.7, 128.3, 125.8, 125.5 (d, J_{C-F} = 3.2 Hz), 115.7 (d, J_{C-F} = 21.7 Hz), 86.2, 48.7; ^{19}F NMR (376 MHz, $CDCl_3$) δ -110.8--111.0 (m). FT-IR: ν (cm^{-1}) 3038, 2923, 2854, 1751, 1685, 1579. HRMS [ESI] calcd for $C_{24}H_{17}O_3FNa$ $[M+Na]^+$ 395.1059, found 395.1057. ESI $[M+Na]^+$ 395.0.

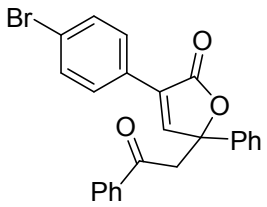


3k: yellow solid, m.p. 100-102 °C. ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.38 (s, 1H), 7.90-7.85 (m, 2H), 7.68-7.62 (m, 2H), 7.59-7.51 (m, 3H), 7.46-7.30 (m, 6H), 7.11-7.04 (m, 1H), 4.13 (d, J = 16.4 Hz, 1H), 3.70 (d, J = 16.4 Hz, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 195.1, 169.4, 162.3 (d, J_{C-F} = 244.6 Hz), 150.9, 138.0, 136.0, 133.4, 130.8 (d, J_{C-F} = 8.4 Hz), 129.8 (d, J_{C-F} = 8.2 Hz), 129.0 (d, J_{C-F} = 2.7 Hz), 128.4, 128.3, 128.3, 127.8, 125.3, 122.5 (d, J_{C-F} = 3.0 Hz), 115.9 (d, J_{C-F} = 21.0 Hz), 113.8 (d, J_{C-F} = 23.0 Hz), 85.8, 48.2;

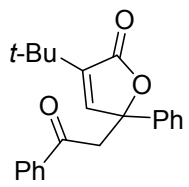
^{19}F NMR (376 MHz, $CDCl_3$) δ -112.1--112.2 (m). FT-IR: ν (cm^{-1}) 3068, 2926, 2853, 1737, 1687, 1580. HRMS [ESI] calcd for $C_{24}H_{17}O_3FNa$ $[M+Na]^+$ 395.1059, found 395.1067. ESI $[M+Na]^+$ 395.0.



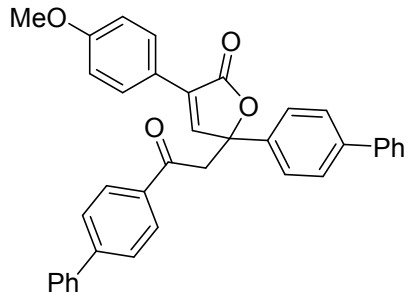
3l: yellow solid, m.p. 100-102 °C. ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.36 (s, 1H), 7.90-7.81 (m, 4H), 7.59-7.51 (m, 3H), 7.47-7.30 (m, 7H), 4.12 (d, *J* = 16.4 Hz, 1H), 3.69 (d, *J* = 16.4 Hz, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 195.0, 169.5, 150.1, 138.0, 135.9, 135.0, 133.3, 128.9, 128.4, 128.3, 128.2, 128.1, 127.8, 127.3, 125.2, 85.8, 48.2. FT-IR: ν (cm^{-1}) 3067, 2957, 2850, 1757, 1671, 1579. HRMS [ESI] calcd for $C_{24}H_{17}O_3ClNa$ $[M+Na]^+$ 411.0764, found 411.0766. ESI $[M+Na]^+$ 411.1.



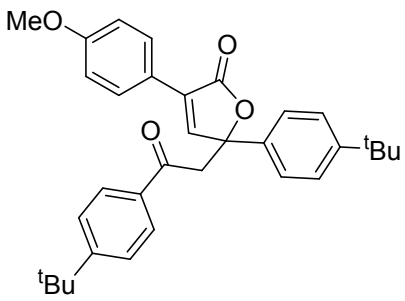
3m: yellow solid, m.p. 106-108 °C. ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.37 (s, 1H), 7.89-7.85 (m, 2H), 7.79-7.75 (m, 2H), 7.60-7.50 (m, 5H), 7.47-7.41 (m, 2H), 7.41-7.29 (m, 3H), 4.12 (d, *J* = 16.8 Hz, 1H), 3.69 (d, *J* = 16.8 Hz, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 195.5, 170.0, 150.7, 138.5, 136.4, 133.8, 131.9, 129.5, 128.9, 128.8, 128.8, 128.7, 128.3, 128.2, 125.8, 123.9, 86.3, 48.7. FT-IR: ν (cm^{-1}) 3063, 2922, 2853, 1752, 1685, 1595. HRMS [ESI] calcd for $C_{24}H_{17}O_3BrNa$ $[M+Na]^+$ 455.0259, found 455.0254. ESI $[M+H]^+$ 455.0.



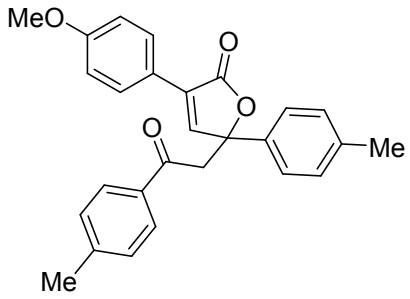
3n: white solid, m.p. 110-112 °C. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.89-7.85 (m, 2H), 7.67 (s, 1H), 7.59-7.53 (m, 1H), 7.49-7.41 (m, 4H), 7.39-7.33 (m, 2H), 7.33-7.28 (m, 1H), 3.95 (d, *J* = 16.0 Hz, 1H), 3.60 (d, *J* = 16.0 Hz, 1H), 1.23 (s, 9H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 195.7, 170.5, 148.6, 141.7, 139.2, 136.6, 133.7, 128.7, 128.4, 128.3, 125.6, 85.4, 48.8, 31.6, 28.1. FT-IR: ν (cm^{-1}) 3058, 2962, 2850, 1742, 1638, 1578. HRMS [ESI] calcd for $C_{22}H_{23}O_3$ $[M+H]^+$ 335.1647, found 335.1655. ESI $[M+Na]^+$ 357.1.



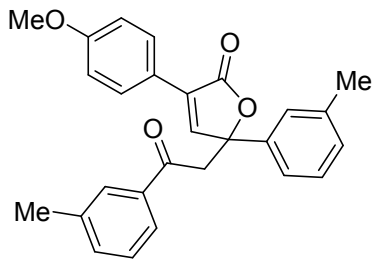
3o: yellow solid, m.p. 83-85 °C. ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.31 (s, 1H), 7.99 (d, *J* = 8.4 Hz, 2H), 7.92 (d, *J* = 8.8 Hz, 2H), 7.70-7.59 (m, 8H), 7.57 (d, *J* = 7.6 Hz, 2H), 7.50-7.40 (m, 5H), 7.39-7.34 (m, 1H), 6.96 (d, *J* = 8.8 Hz, 2H), 4.22 (d, *J* = 16.4 Hz, 1H), 3.84 (s, 3H), 3.74 (d, *J* = 16.4 Hz, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 195.3, 170.6, 160.6, 148.0, 146.4, 141.5, 140.2, 139.6, 137.9, 135.2, 130.0, 129.0, 129.0, 128.9, 128.7, 128.5, 127.7, 127.5, 127.4, 127.3, 127.1, 126.4, 122.0, 114.1, 86.1, 55.4, 48.9. FT-IR: ν (cm^{-1}) 3051, 2955, 2835, 1750, 1678, 1560. HRMS [ESI] calcd for $C_{37}H_{28}O_4Na$ $[M+Na]^+$ 559.1885, found 559.1878. ESI $[M+Na]^+$ 559.2.



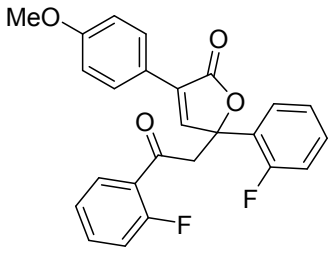
3p: yellow solid, m.p. 81-83 °C. ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.26 (s, 1H), 7.89-7.81 (m, 4H), 7.50-7.43 (m, 4H), 7.41-7.35 (m, 2H), 6.96-6.90 (m, 2H), 4.13 (d, *J* = 16.4 Hz, 1H), 3.82 (s, 3H), 3.63 (d, *J* = 16.4 Hz, 1H), 1.33 (s, 9H), 1.29 (s, 9H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 195.4, 170.7, 160.5, 157.5, 151.5, 148.4, 136.0, 134.1, 129.7, 128.7, 128.3, 125.7, 125.7, 125.6, 122.1, 114.0, 86.2, 55.3, 48.8, 35.2, 34.6, 31.2, 31.0. FT-IR: ν (cm^{-1}) 3051, 2960, 2867, 1754, 1681, 1572. HRMS [ESI] calcd for $C_{33}H_{36}O_4Na$ $[M+Na]^+$ 519.2511, found 519.2506. ESI $[M+Na]^+$ 519.2.



3q: yellow solid, m.p. 44-46 °C. ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.22 (s, 1H), 7.88-7.83 (m, 2H), 7.79 (d, *J* = 8.0 Hz, 2H), 7.42 (d, *J* = 8.0 Hz, 2H), 7.23 (d, *J* = 8.0 Hz, 2H), 7.17 (d, *J* = 8.0 Hz, 2H), 6.95-6.90 (m, 2H), 4.10 (d, *J* = 16.4 Hz, 1H), 3.82 (s, 3H), 3.62 (d, *J* = 16.4 Hz, 1H), 2.39 (s, 3H), 2.32 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 194.8, 170.2, 160.0, 147.9, 144.2, 137.9, 135.5, 133.7, 129.3, 128.9, 128.9, 128.2, 128.0, 125.3, 121.6, 113.6, 85.7, 54.8, 48.2, 21.2, 20.6. FT-IR: ν (cm^{-1}) 3032, 2954, 2837, 1751, 1681, 1572. HRMS [ESI] calcd for $C_{27}H_{24}O_4Na$ $[M+Na]^+$ 435.1572, found 435.1574. ESI $[M+Na]^+$ 435.1.

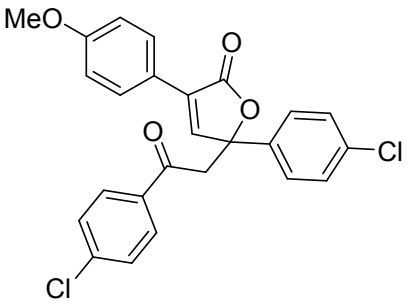


3r: yellow oil. ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.17 (s, 1H), 7.86-7.80 (m, 2H), 7.67-7.62 (m, 2H), 7.35-7.27 (m, 4H), 7.24-7.19 (m, 1H), 7.09 (d, *J* = 7.6 Hz, 1H), 6.92-6.86 (m, 2H), 4.05 (d, *J* = 16.4 Hz, 1H), 3.78 (s, 3H), 3.63 (d, *J* = 16.4 Hz, 1H), 2.33 (s, 3H), 2.31 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 195.4, 170.2, 160.0, 147.8, 138.5, 138.1, 136.2, 134.0, 129.3, 128.8, 128.3, 128.2, 128.2, 128.1, 126.0, 125.1, 122.4, 121.5, 113.6, 85.7, 54.9, 48.3, 21.1, 20.9. FT-IR: ν (cm^{-1}) 3042, 2920, 2837, 1750, 1630, 1585. HRMS [ESI] calcd for $C_{27}H_{25}O_4$ $[M+H]^+$ 413.1753, found 413.1760. ESI $[M+Na]^+$ 435.1.

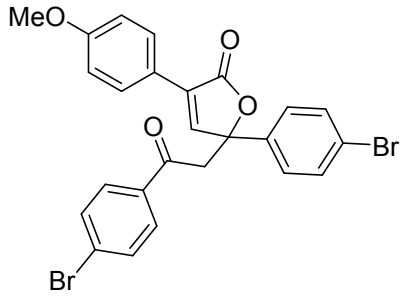


3s: yellow solid, m.p. 75-77 °C. ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.01 (d, *J* = 3.2 Hz, 1H), 7.85-7.79 (m, 2H), 7.70 (ddd, *J* = 7.6, 7.6, 1.6 Hz, 1H), 7.62 (ddd, *J* = 7.6, 7.6, 1.6 Hz, 1H), 7.53-7.46 (m, 1H), 7.35-7.29 (m, 1H), 7.20-7.14 (m, 2H), 7.13-7.05 (m, 2H), 6.94-6.87 (m, 2H), 3.96 (dd, *J* = 16.8, 2.0 Hz, 1H), 3.89 (dd, *J* = 16.8, 2.0 Hz, 1H), 3.82 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 192.7 (d, $J_{C\text{-}F}$ = 3.7 Hz), 170.0, 161.3 (d, $J_{C\text{-}F}$ = 252.8 Hz), 160.1, 158.6 (d, $J_{C\text{-}F}$ = 244.3 Hz), 146.1 (d, $J_{C\text{-}F}$ = 5.1 Hz), 134.6 (d, $J_{C\text{-}F}$ = 9.2 Hz), 130.1 (d, $J_{C\text{-}F}$ = 2.2 Hz), 129.9 (d, $J_{C\text{-}F}$ = 8.5 Hz), 129.4, 128.2, 126.9 (d, $J_{C\text{-}F}$ = 3.6 Hz), 125.8 (d, $J_{C\text{-}F}$ = 11.9 Hz), 125.2 (d, $J_{C\text{-}F}$ = 12.5 Hz), 124.4 (d, $J_{C\text{-}F}$ = 3.2 Hz), 124.1 (d, $J_{C\text{-}F}$ = 3.3 Hz), 121.2, 116.2 (d, $J_{C\text{-}F}$ = 23.6 Hz), 115.6 (d, $J_{C\text{-}F}$ = 22.1 Hz), 113.6, 83.4, 54.8, 49.9 (dd, $J_{C\text{-}F}$ = 8.1, 3.3 Hz); ^{19}F NMR (376 MHz, $CDCl_3$) δ -108.9--109.1 (m), -113.7--113.9 (m). FT-IR: ν (cm^{-1}) 3041, 2934, 2838,

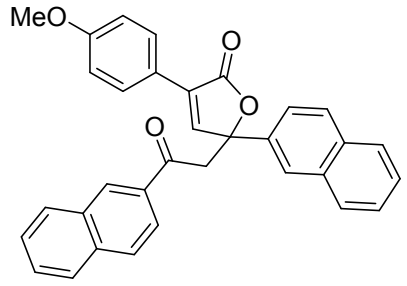
1758, 1688, 1574. HRMS [ESI] calcd for $C_{25}H_{19}O_4F_2$ $[M+H]^+$ 421.1251, found 421.1259. ESI $[M+Na]^+$ 443.0.



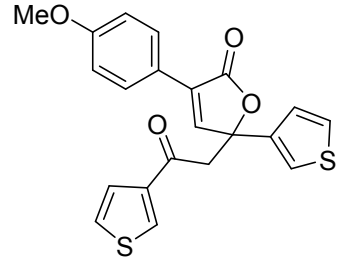
3t: yellow solid, m.p. 175-177 °C. ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.15 (s, 1H), 7.84 (d, *J* = 8.8 Hz, 2H), 7.80 (d, *J* = 8.8 Hz, 2H), 7.46 (d, *J* = 8.8 Hz, 2H), 7.41 (d, *J* = 8.8 Hz, 2H), 7.33 (d, *J* = 8.8 Hz, 2H), 6.92 (d, *J* = 8.8 Hz, 2H), 4.04 (d, *J* = 16.8 Hz, 1H), 3.83 (s, 3H), 3.62 (d, *J* = 16.8 Hz, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 193.8, 169.7, 160.2, 146.6, 140.0, 136.8, 134.2, 134.1, 129.8, 129.2, 128.7, 128.5, 128.2, 126.9, 121.1, 113.6, 84.9, 54.9, 48.4. FT-IR: ν (cm^{-1}) 3060, 2961, 2831, 1748, 1643, 1573. HRMS [ESI] calcd for $C_{25}H_{19}O_4Cl_2$ $[M+H]^+$ 453.0660, found 453.0688. ESI $[M+H]^+$ 453.1.



3u: yellow solid, m.p. 201-203 °C. ^{1}H NMR (400 MHz, DMSO) δ 8.35 (s, 1H), 7.86 (d, *J* = 8.0 Hz, 4H), 7.71 (d, *J* = 8.0 Hz, 2H), 7.60 (d, *J* = 8.0 Hz, 2H), 7.53 (d, *J* = 8.0 Hz, 2H), 7.00 (d, *J* = 8.0 Hz, 2H), 4.16 (d, *J* = 17.2 Hz, 1H), 4.05 (d, *J* = 17.2 Hz, 1H), 3.78 (s, 3H); ^{13}C NMR (100 MHz, DMSO) δ 194.8, 170.4, 160.0, 149.5, 138.7, 135.5, 131.7, 131.5, 130.2, 128.4, 128.2, 127.8, 127.7, 121.6, 121.5, 114.0, 85.4, 55.2, 46.0. FT-IR: ν (cm^{-1}) 3068, 2957, 2850, 1748, 1635, 1585. HRMS [ESI] calcd for $C_{25}H_{18}O_4Br_2Na$ $[M+Na]^+$ 562.9470, found 562.9466. ESI $[M+Na]^+$ 563.0.



3v: yellow oil. ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.41 (s, 1H), 8.38 (s, 1H), 8.06 (d, *J* = 1.2 Hz, 1H), 7.95-7.89 (m, 4H), 7.87-7.77 (m, 5H), 7.69 (dd, *J* = 8.8, 1.6 Hz, 1H), 7.62-7.57 (m, 1H), 7.55-7.51 (m, 1H), 7.51-7.46 (m, 2H), 6.97-6.91 (m, 2H), 4.35 (d, *J* = 16.4 Hz, 1H), 3.90 (d, *J* = 16.4 Hz, 1H), 3.82 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 195.0, 170.2, 160.1, 147.6, 135.8, 135.3, 133.4, 132.6, 132.5, 131.9, 130.0, 129.6, 129.3, 128.4, 128.3, 128.2, 128.2, 127.9, 127.3, 127.2, 126.5, 126.3, 126.2, 124.7, 123.1, 122.9, 121.5, 113.6, 85.9, 54.9, 48.4. FT-IR: ν (cm^{-1}) 3057, 2957, 2837, 1751, 1626, 1573. HRMS [ESI] calcd for $C_{33}H_{24}O_4Na$ $[M+Na]^+$ 507.1572, found 507.1570. ESI $[M+Na]^+$ 507.1.

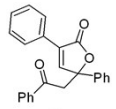


3w: yellow solid, m.p. 123-125 °C. ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.14 (s, 1H), 8.03 (dd, *J* = 2.8, 1.2 Hz, 1H), 7.84 (d, *J* = 8.8 Hz, 2H), 7.47 (dd, *J* = 5.2, 1.2 Hz, 1H), 7.35 (dd, *J* = 2.8, 1.2 Hz, 1H), 7.32-7.28 (m, 2H), 7.17 (dd, *J* = 5.2, 1.2 Hz, 1H), 6.92 (d, *J* = 8.8 Hz, 2H), 4.02 (d, *J* = 16.0 Hz, 1H), 3.82 (s, 3H), 3.51 (d, *J* = 16.0 Hz, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 189.2, 170.1, 160.1, 147.1, 141.4, 138.8, 132.9, 129.2, 128.2, 126.4, 126.3, 126.2,

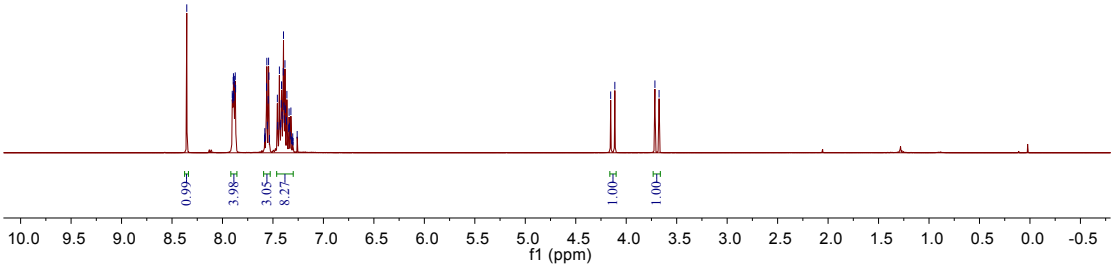
125.1, 122.1, 121.3, 113.6, 83.6, 54.9, 49.4. FT-IR: ν (cm^{-1}) 3058, 2960, 2851, 1748, 1664, 1578. HRMS [ESI] calcd for $C_{21}H_{16}O_4S_2Na$ $[M+Na]^+$ 419.0388, found 419.0387. ESI $[M+Na]^+$ 419.0.

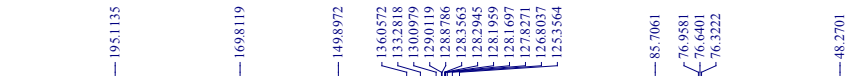
4. ^{1}H, ^{13}C, and ^{19}F NMR spectra

Parameter	Value
1 Origin	Bruker BioSpin GmbH
2 Solvent	CDCl3
3 Temperature	302.7
4 Number of Scans	6
5 Spectrometer Frequency	400.13
6 Nucleus	1H

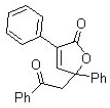


3a

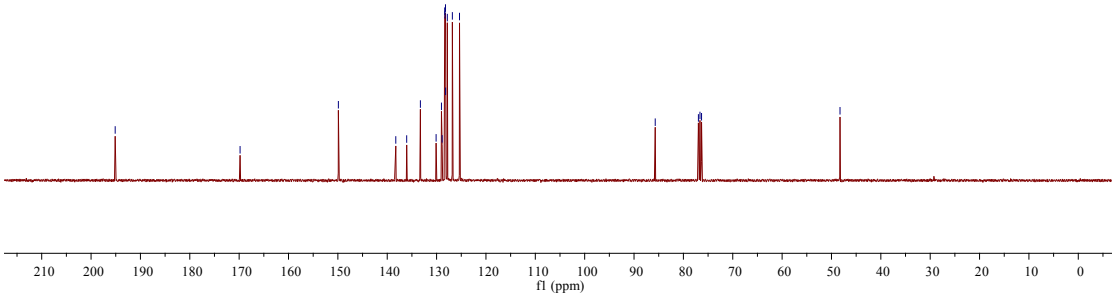




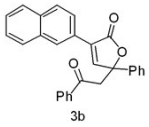
Parameter	Value
1 Origin	Bruker BioSpin GmbH
2 Solvent	CDCl3
3 Temperature	297.4
4 Number of Scans	66
5 Spectrometer Frequency	100.61
6 Nucleus	13C



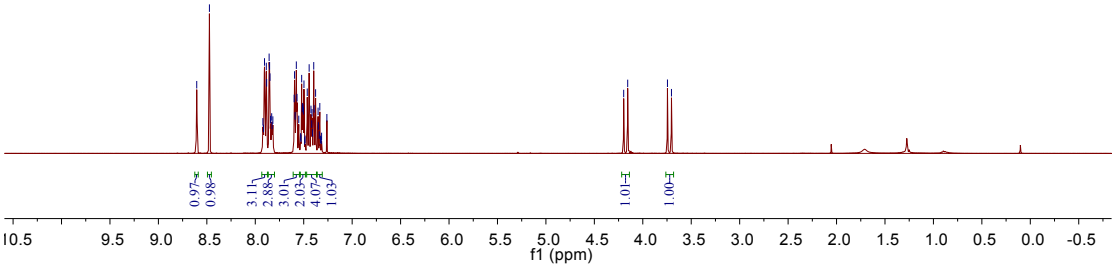
3a

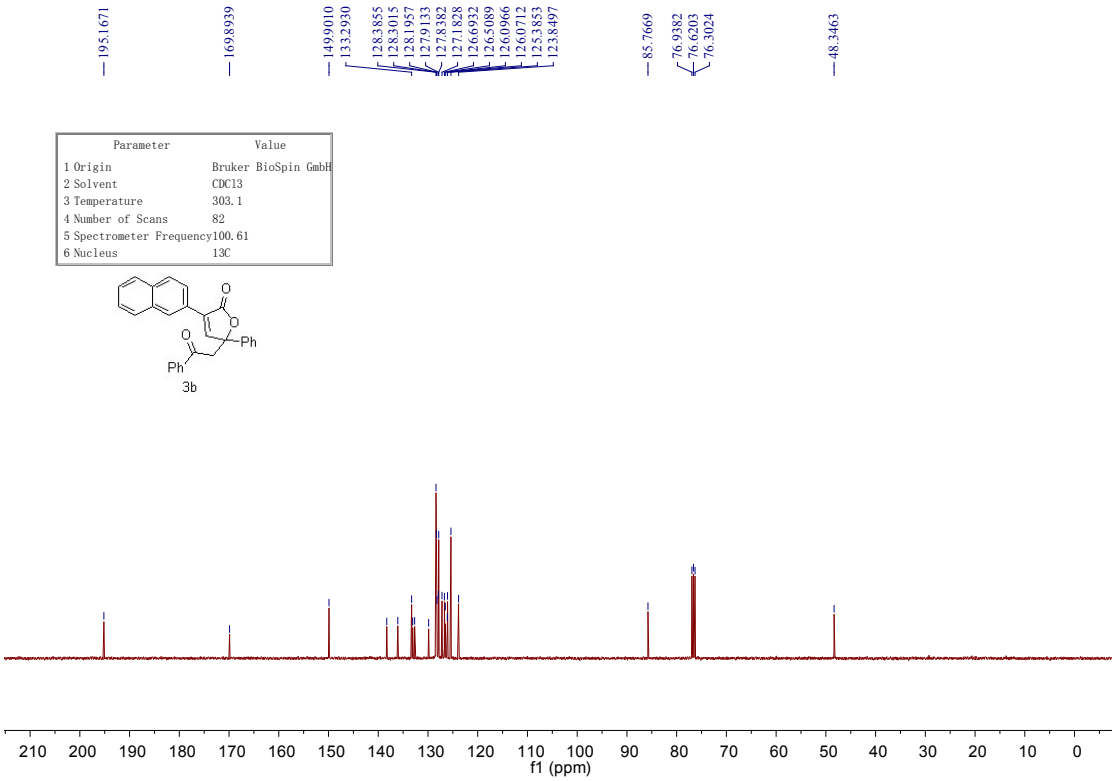


Parameter	Value
1 Origin	Bruker BioSpin GmbH
2 Solvent	CDCl3
3 Temperature	301.8
4 Number of Scans	4
5 Spectrometer Frequency	400.13
6 Nucleus	1H



3b

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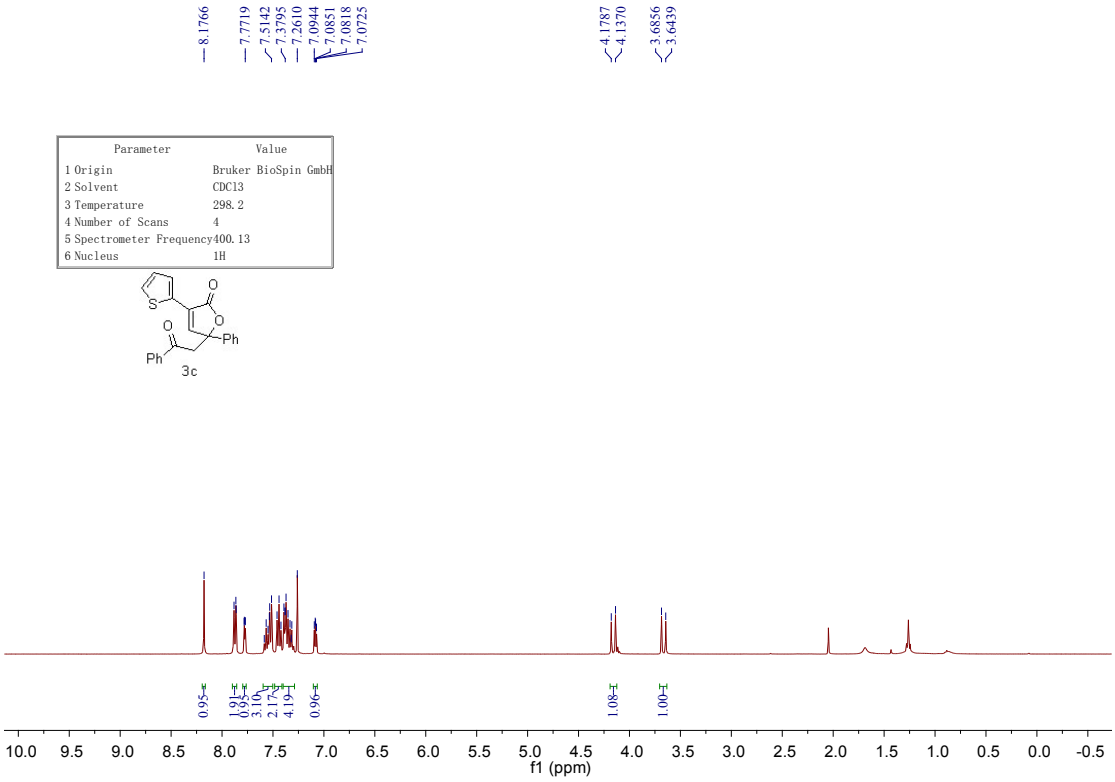
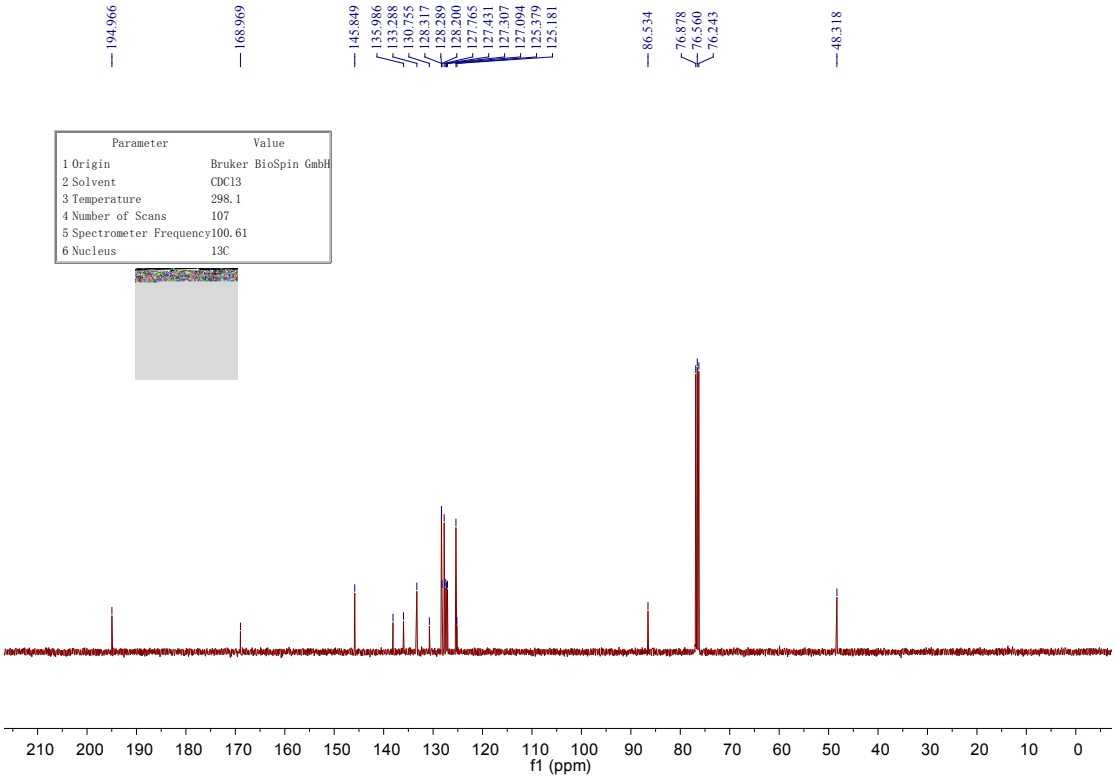
169.8939
149.9010
133.2930
128.3855
128.3015
128.1957
127.9133
127.8382
127.1828
126.6932
126.5089
126.0966
126.0712
125.3853
123.8497
85.7669
76.9382
76.6203
76.3024
48.3463
Parameter Value
1 Origin Bruker BioSpin GmbH
2 Solvent CDCl3
3 Temperature 303.1
4 Number of Scans 82
5 Spectrometer Frequency 100.61
6 Nucleus 13C
O
O
Ph
O
Ph
3b
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)


8.1766
7.7719
7.5142
7.3795
7.2610
7.0944
7.0851
7.0818
7.0725
4.1787
4.1370
3.6856
3.6439
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
S
O
O
O
Ph
Ph
3c
0.95
1.91
0.95
3.10
2.17
4.19
0.96
1.08
1.00
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
f1 (ppm)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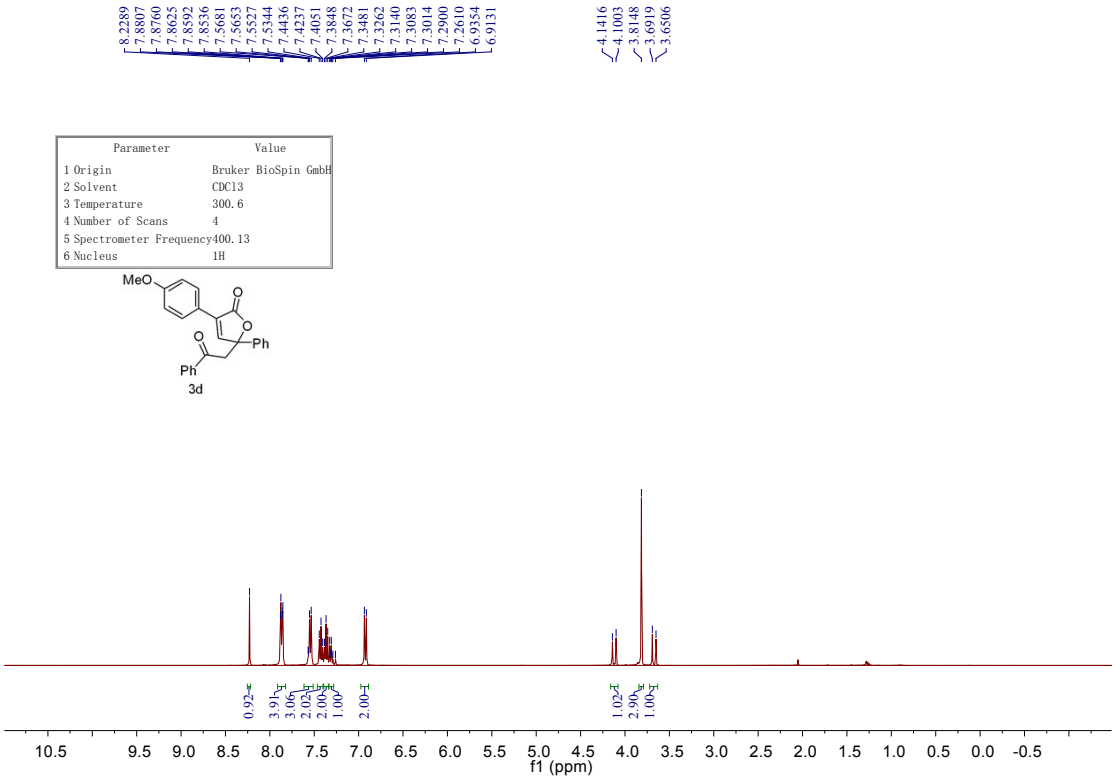
145.849
135.986
133.288
130.755
128.317
128.289
128.200
127.765
127.431
127.307
127.094
125.379
125.181
86.534
76.878
76.560
76.243
48.318
Parameter Value
1 Origin Bruker BioSpin GmbH
2 Solvent CDCl3
3 Temperature 298.1
4 Number of Scans 107
5 Spectrometer Frequency 100.61
6 Nucleus 13C
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)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7.8625
7.8592
7.8536
7.5681
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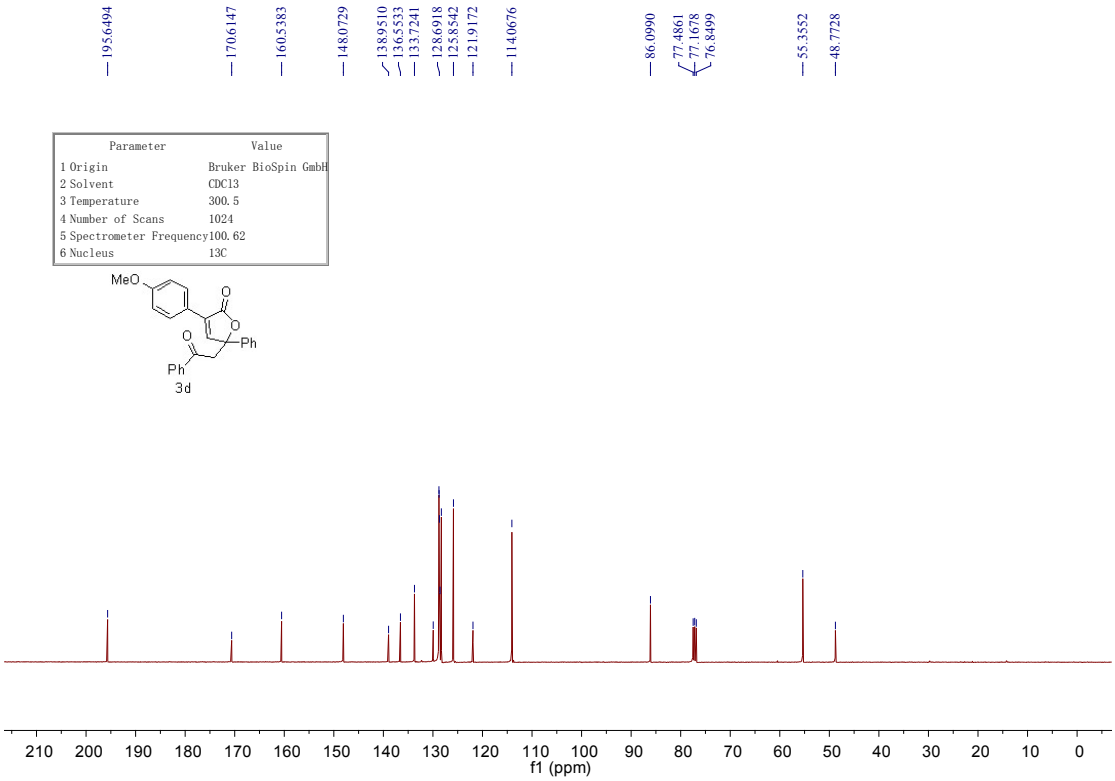
7.2610
6.9354
6.9131
4.1416
4.1003
3.8148
3.6919
3.6506
Parameter Value
1 Origin Bruker BioSpin GmbH
2 Solvent CDCl3
3 Temperature 300.6
4 Number of Scans 4
5 Spectrometer Frequency 400.13
6 Nucleus 1H
MeO
O
O
Ph
O
Ph
3d
0.92
3.91
3.06
2.02
2.00
1.00
2.00
1.02
2.90
1.00
10.5 9.5 9.0 8.5 8.0 7.5 7.0 6.5 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
f1 (ppm)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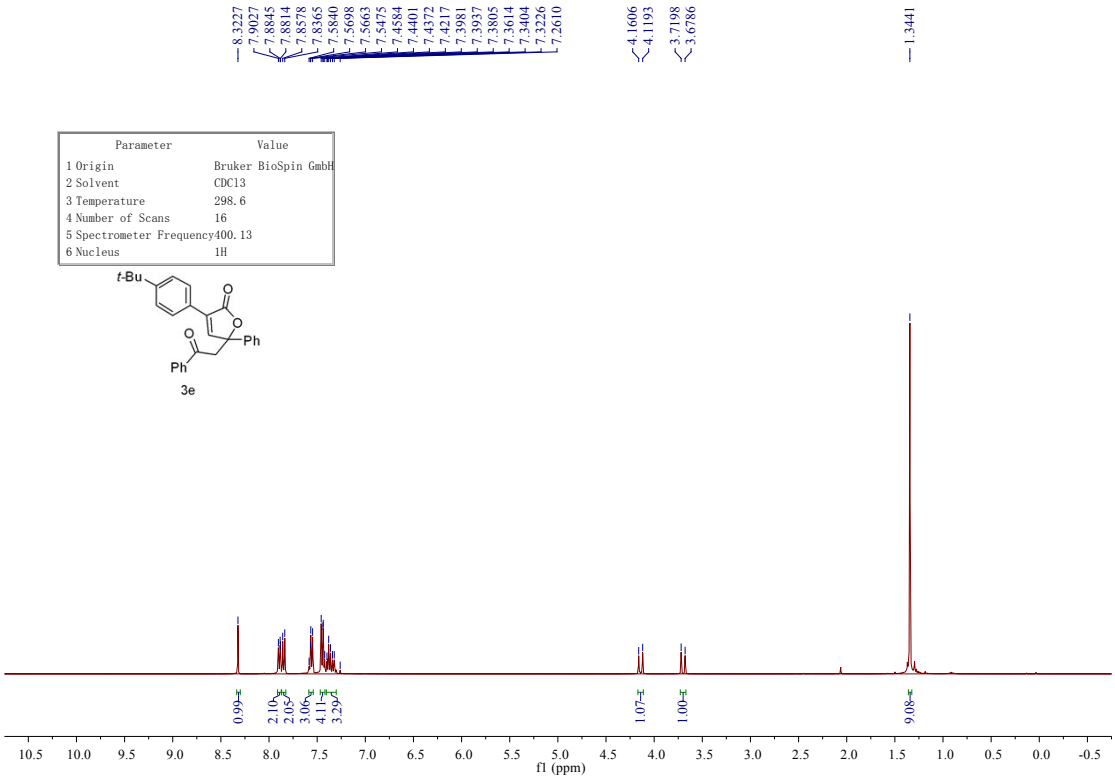
148.0729
138.9510
136.5533
133.7241
128.6918
125.8542
121.9172
114.0676
86.0990
77.4861
77.1678
76.8499
55.3552
48.7728
Parameter Value
1 Origin Bruker BioSpin GmbH
2 Solvent CDCl3
3 Temperature 300.5
4 Number of Scans 1024
5 Spectrometer Frequency 100.62
6 Nucleus 13C
MeO
O
O
O
Ph
Ph
3d
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)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7.8365
7.5840
7.5698
7.5663
7.5475
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7.4401
7.4372
7.4217
7.3981
7.3937
7.3805
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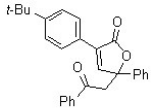
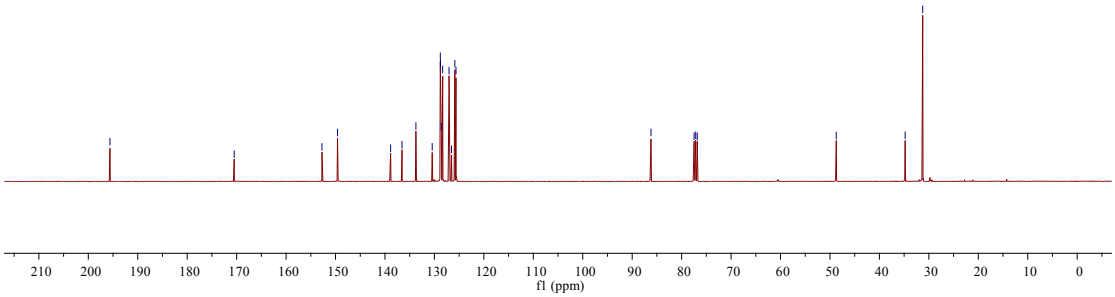
7.3404
7.3226
7.2610
4.1606
4.1193
3.7198
3.6786
1.3441
Parameter Value
1 Origin Bruker BioSpin GmbH
2 Solvent CDCl3
3 Temperature 298.6
4 Number of Scans 16
5 Spectrometer Frequency 400.13
6 Nucleus 1H
t-Bu
O
O
O
Ph
Ph
3e
0.99
2.10
2.05
3.06
4.11
3.29
1.07
1.00
9.08
10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 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
f1 (ppm)

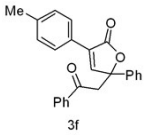
Parameter	Value
1 Origin	Bruker BioSpin GmbH
2 Solvent	CDCl3
3 Temperature	299.2
4 Number of Scans	1024
5 Spectrometer Frequency	100.62
6 Nucleus	13C



3e



Parameter	Value
1 Origin	Bruker BioSpin GmbH
2 Solvent	CDCl3
3 Temperature	300.2
4 Number of Scans	4
5 Spectrometer Frequency	400.13
6 Nucleus	1H



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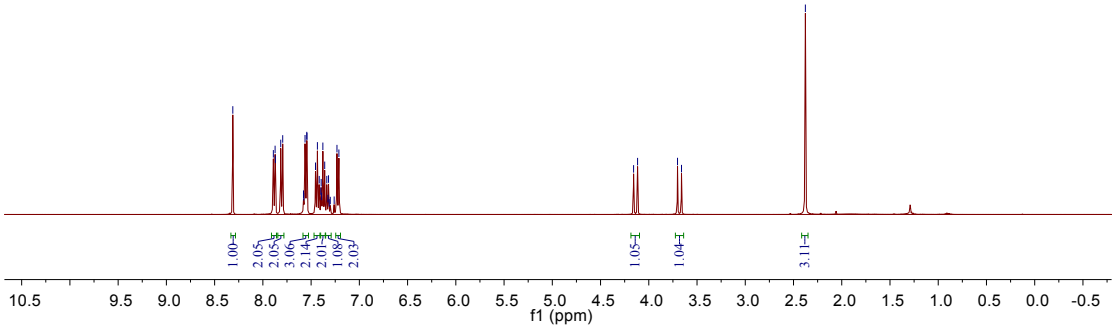
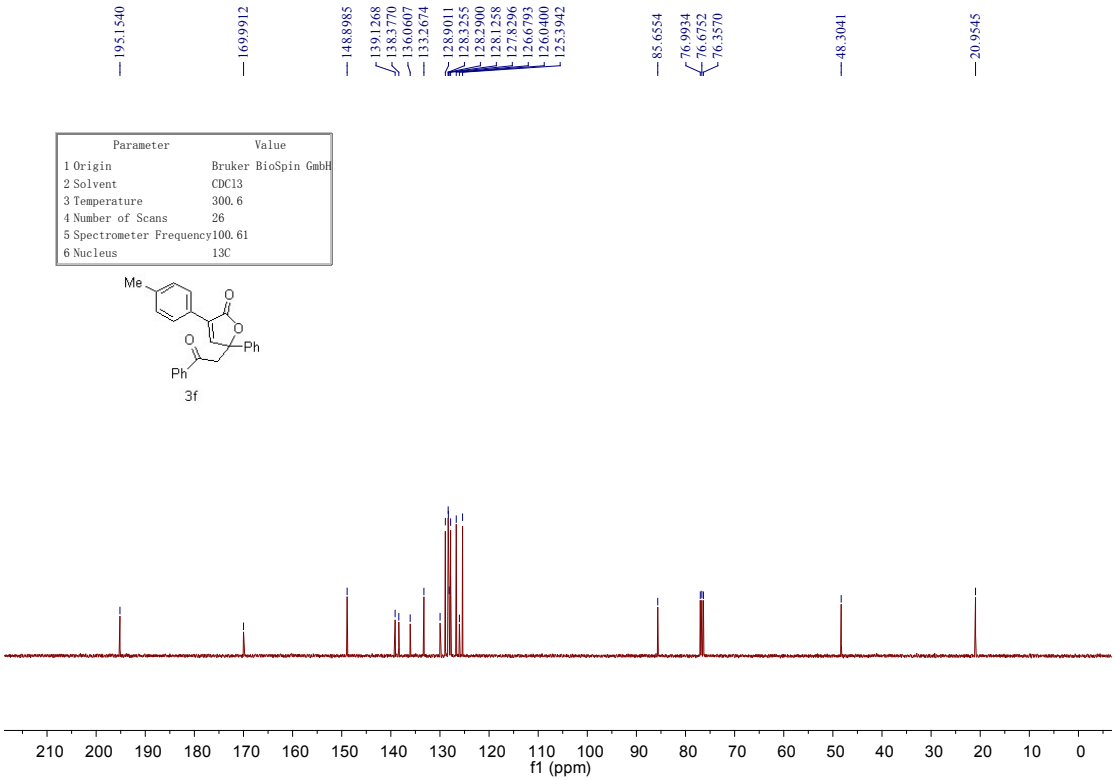
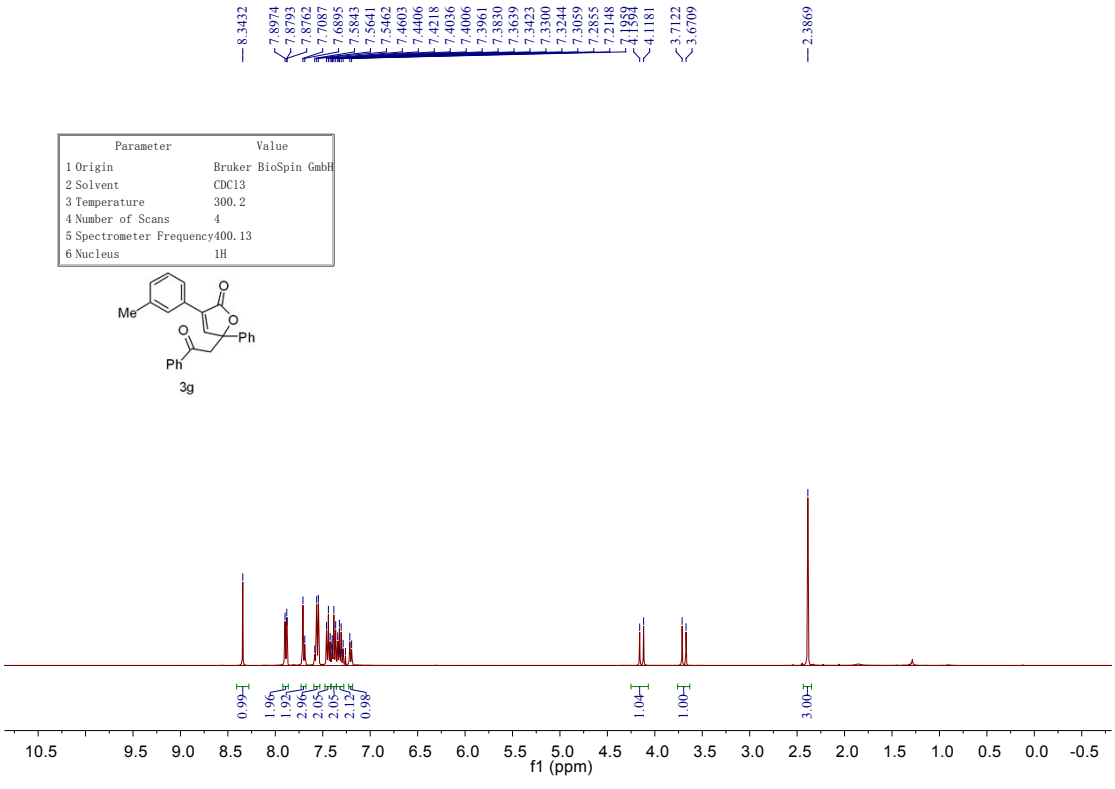
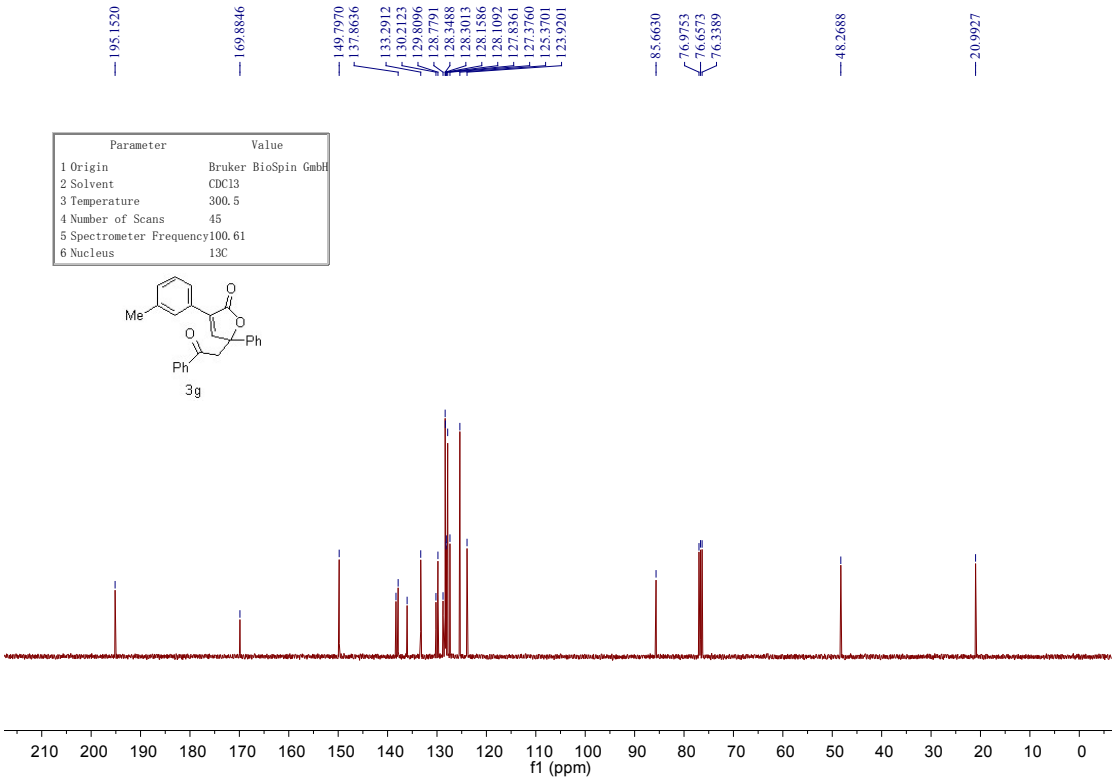
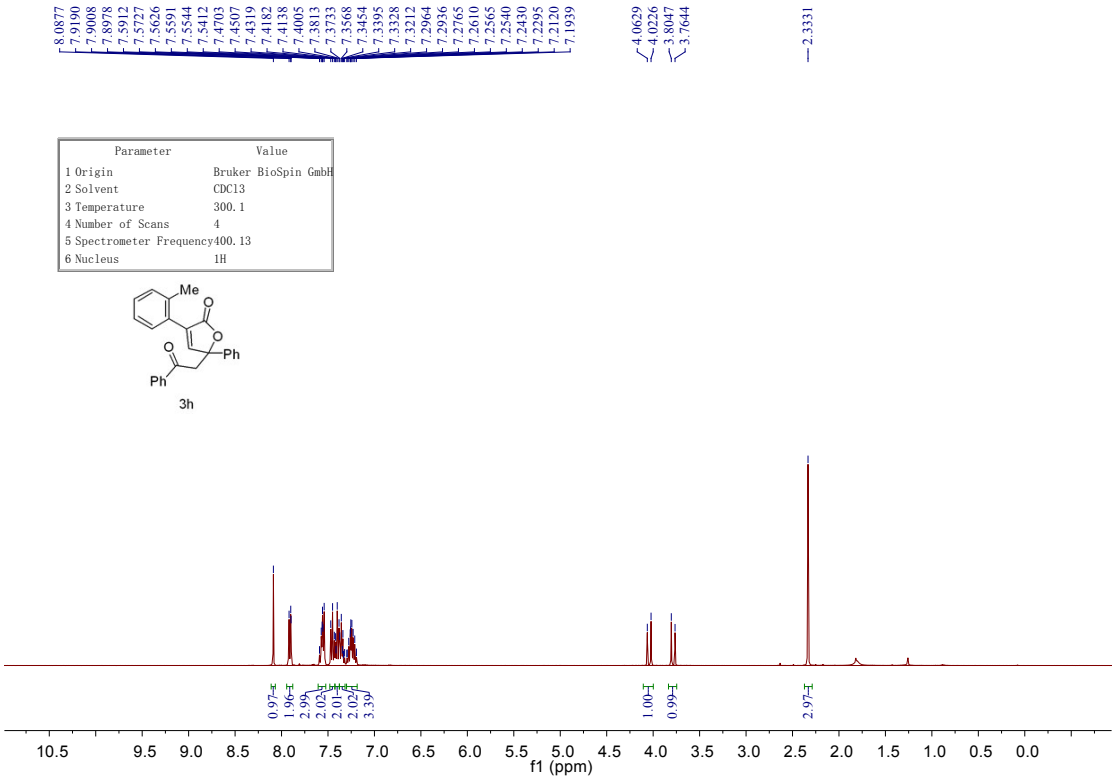
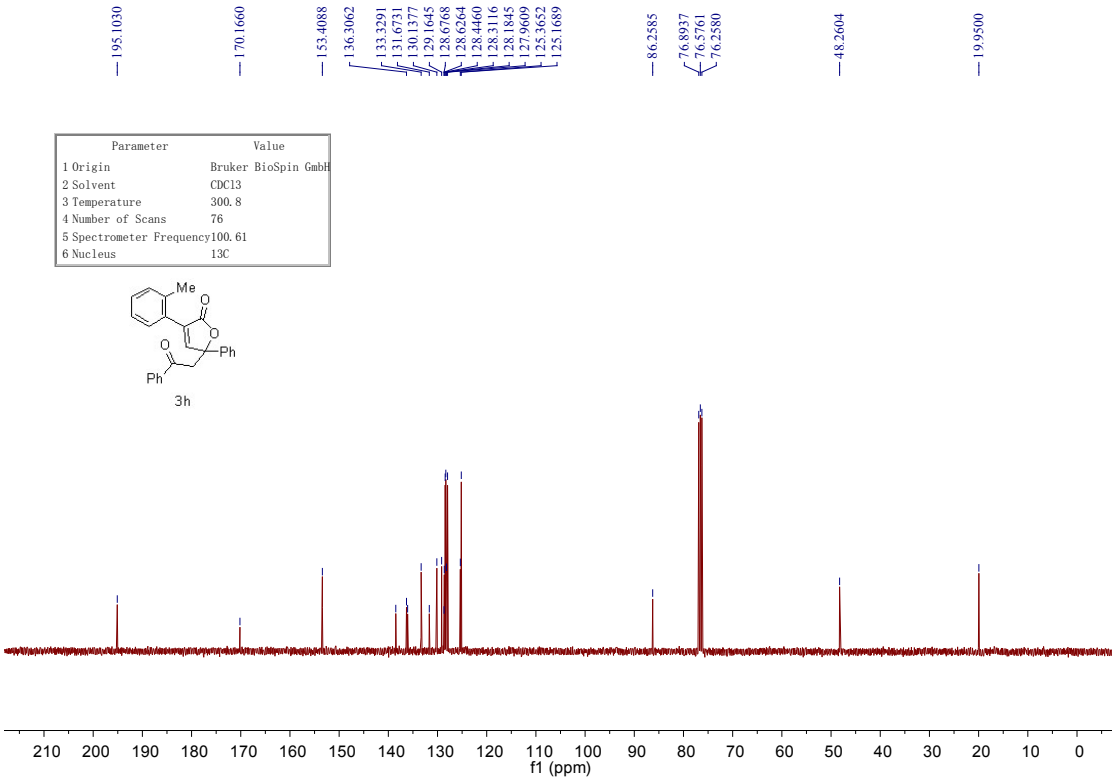


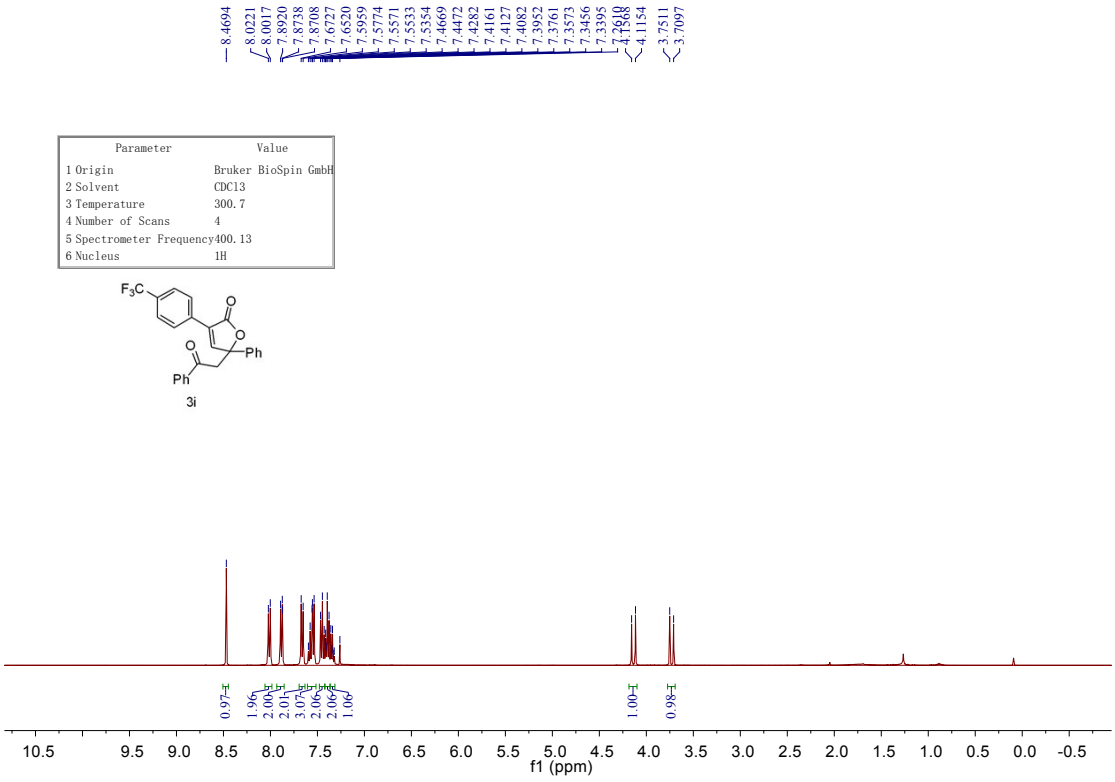

195.1540
169.9912
148.8985
139.1268
138.3770
136.0607
133.2674
128.9011
128.3255
128.2900
128.1258
127.8296
126.6793
126.0400
125.3942
85.6554
76.9934
76.6752
76.3570
48.3041
20.9545
Parameter Value
1 Origin Bruker BioSpin GmbH
2 Solvent CDCl3
3 Temperature 300.6
4 Number of Scans 26
5 Spectrometer Frequency 100.61
6 Nucleus 13C
Me
O
O
Ph
O
Ph
3f
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)

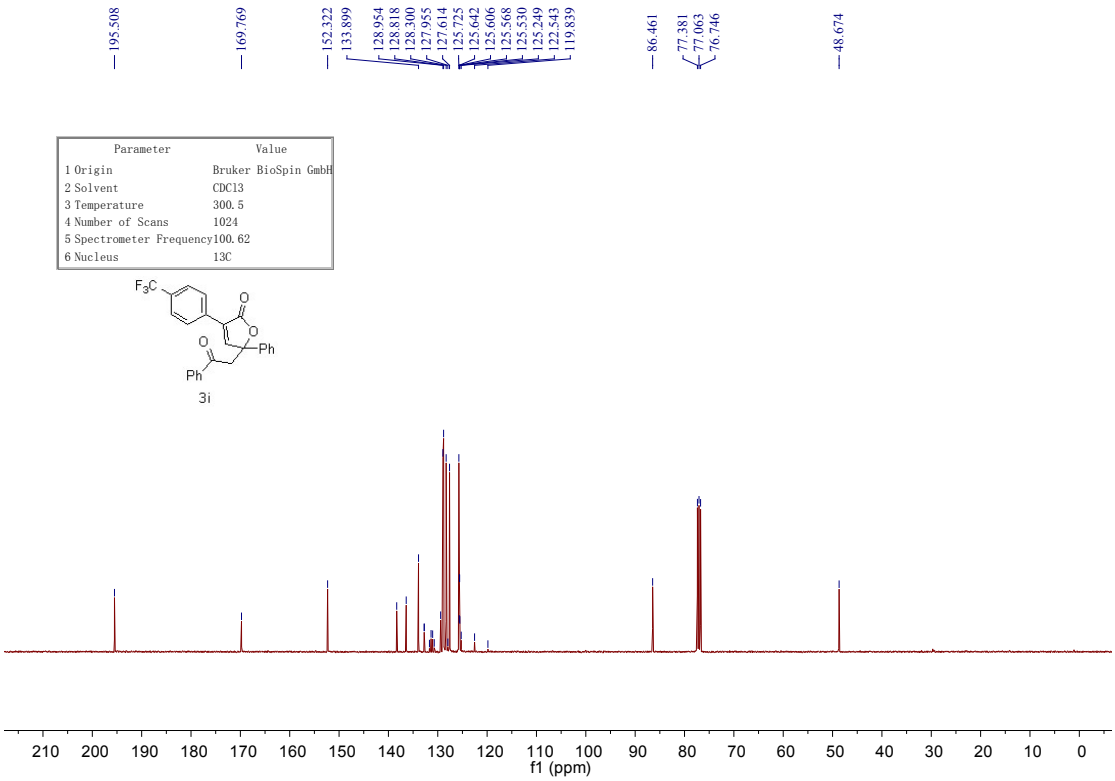

8.3432
7.8974
7.8793
7.8762
7.7087
7.6895
7.5843
7.5641
7.5462
7.4603
7.4406
7.4218
7.4036
7.4006
7.3961
7.3830
7.3639
7.3423
7.3300
7.3244
7.3059
7.2855
7.2148
4.1594
4.1181
3.7122
3.6709
2.3869
Parameter Value
1 Origin Bruker BioSpin GmbH
2 Solvent CDCl3
3 Temperature 300.2
4 Number of Scans 4
5 Spectrometer Frequency 400.13
6 Nucleus 1H
Me
O
O
Ph
O
Ph
3g
0.99
1.96
1.92
2.96
2.05
2.05
2.12
0.98
1.04
1.00
3.00
10.5 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
f1 (ppm)

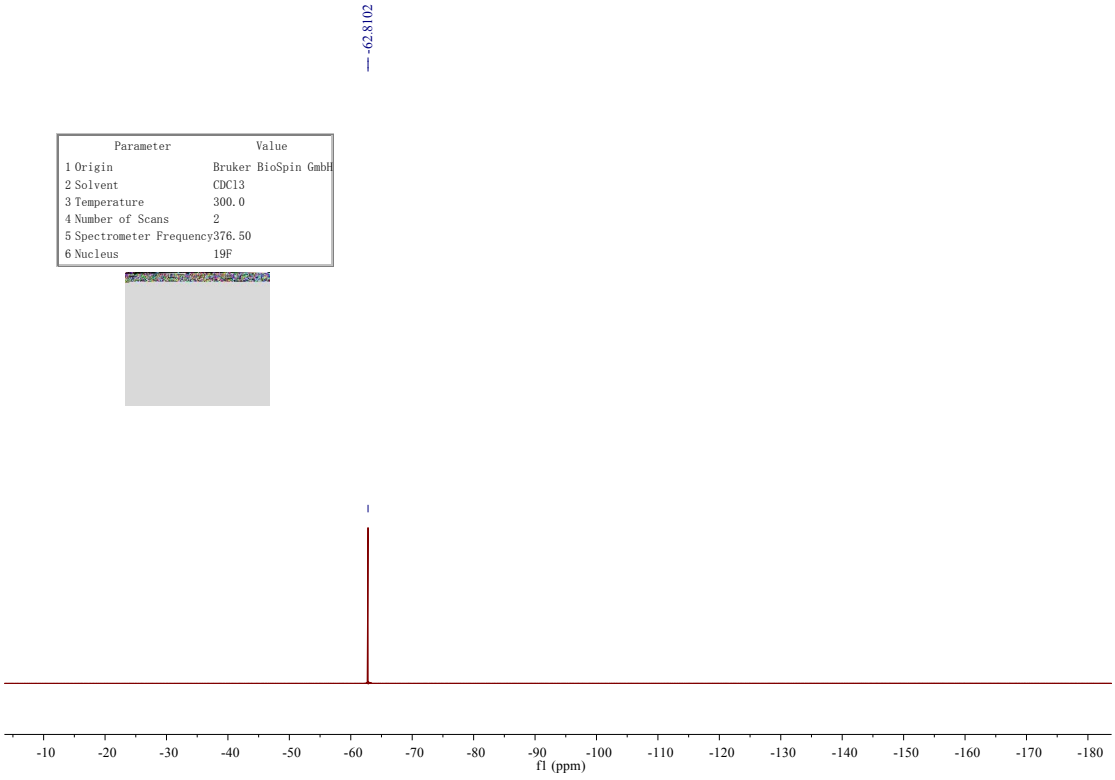

195.1520
169.8846
149.7970
137.8636
133.2912
130.2123
129.8096
128.7791
128.3488
128.3013
128.1586
128.1092
127.8361
127.3760
125.3701
123.9201
85.6630
76.9753
76.6573
76.3389
48.2688
20.9927
Parameter Value
1 Origin Bruker BioSpin GmbH
2 Solvent CDCl3
3 Temperature 300.5
4 Number of Scans 45
5 Spectrometer Frequency 100.61
6 Nucleus 13C
Me
O
O
Ph
O
Ph
3g
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)


8.0877
7.9190
7.9008
7.8978
7.5912
7.5727
7.5626
7.5591
7.5544
7.5412
7.4703
7.4507
7.4319
7.4182
7.4138
7.4005
7.3813
7.3733
7.3568
7.3454
7.3395
7.3328
7.3212
7.2964
7.2936
7.2765
7.2610
7.2565
7.2540
7.2430
7.2295
7.2120
7.1939
4.0629
4.0226
3.8047
3.7644
2.3331
Parameter Value
1 Origin Bruker BioSpin GmbH
2 Solvent CDCl3
3 Temperature 300.1
4 Number of Scans 4
5 Spectrometer Frequency 400.13
6 Nucleus 1H
Me
O
O
Ph
O
Ph
3h
0.97
1.96
2.99
2.03
2.01
2.02
3.39
1.00
0.99
2.97
10.5 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)

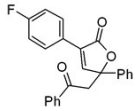

195.1030
170.1660
153.4088
136.3062
133.3291
131.6731
130.1377
129.1645
128.6768
128.6264
128.4460
128.3116
128.1845
127.9609
125.3652
125.1689
86.2585
76.8937
76.5761
76.2580
48.2604
19.9500
Parameter Value
1 Origin Bruker BioSpin GmbH
2 Solvent CDCl3
3 Temperature 300.8
4 Number of Scans 76
5 Spectrometer Frequency 100.61
6 Nucleus 13C
Me
O
O
Ph
O
Ph
3h
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)


8.4694
8.0221
8.0017
7.8920
7.8738
7.8708
7.6727
7.6520
7.5959
7.5774
7.5571
7.5533
7.5354
7.4669
7.4472
7.4282
7.4161
7.4127
7.4082
7.3952
7.3761
7.3573
7.3456
7.3395
7.2610
7.1568
4.1154
3.7511
3.7097
Parameter Value
1 Origin Bruker BioSpin GmbH
2 Solvent CDCl3
3 Temperature 300.7
4 Number of Scans 4
5 Spectrometer Frequency 400.13
6 Nucleus 1H
F3C
O
O
Ph
O
Ph
3i
0.97
1.96
2.00
2.01
3.07
2.06
2.06
1.06
1.00
0.98
10.5 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
f1 (ppm)

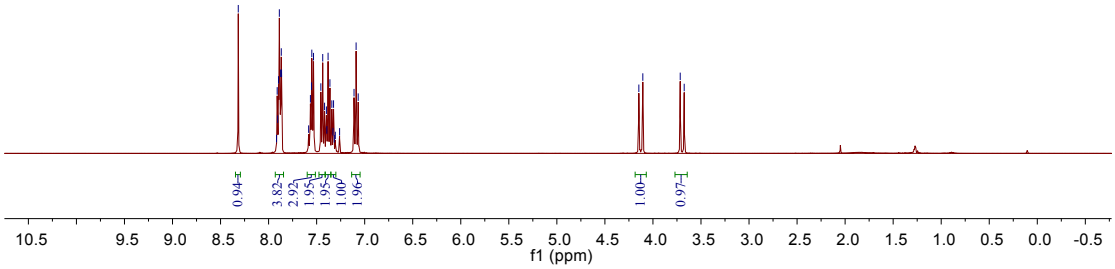

195.508
169.769
152.322
133.899
128.954
128.818
128.300
127.955
127.614
125.725
125.642
125.606
125.568
125.530
125.249
122.543
119.839
86.461
77.381
77.063
76.746
48.674
Parameter Value
1 Origin Bruker BioSpin GmbH
2 Solvent CDCl3
3 Temperature 300.5
4 Number of Scans 1024
5 Spectrometer Frequency 100.62
6 Nucleus 13C
F3C
O
O
Ph
O
Ph
3i
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)

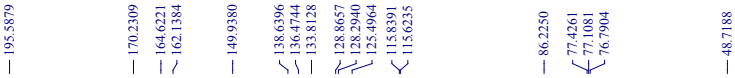

-62.8102
Parameter Value
1 Origin Bruker BioSpin GmbH
2 Solvent CDCl3
3 Temperature 300.0
4 Number of Scans 2
5 Spectrometer Frequency 376.50
6 Nucleus 19F
-10 -20 -30 -40 -50 -60 -70 -80 -90 -100 -110 -120 -130 -140 -150 -160 -170 -180
f1 (ppm)

Parameter	Value
1 Origin	Bruker BioSpin GmbH
2 Solvent	CDCl3
3 Temperature	300.6
4 Number of Scans	4
5 Spectrometer Frequency	400.13
6 Nucleus	1H

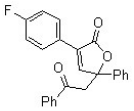


3j

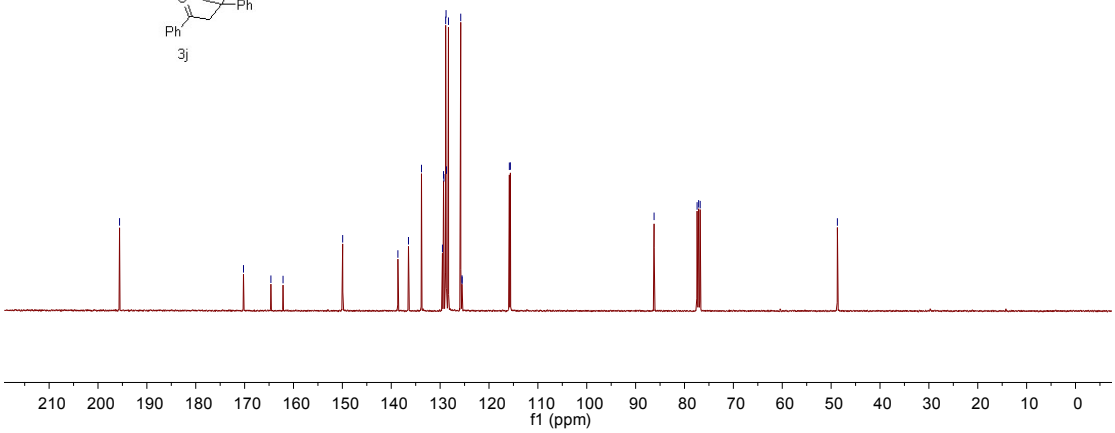




Parameter	Value
1 Origin	Bruker BioSpin GmbH
2 Solvent	CDCl3
3 Temperature	300.5
4 Number of Scans	1024
5 Spectrometer Frequency	100.62
6 Nucleus	13C

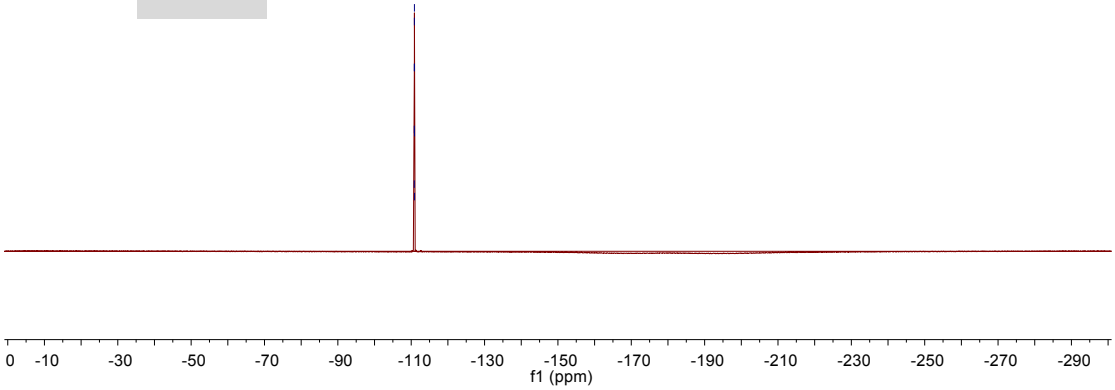


3j



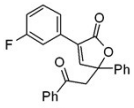
-110.8313
-110.8452
-110.8531
-110.8668
-110.8752
-110.8889
-110.8972
-110.9113

Parameter	Value
1 Origin	Bruker BioSpin GmbH
2 Solvent	CDCl3
3 Temperature	299.9
4 Number of Scans	16
5 Spectrometer Frequency	376.44
6 Nucleus	19F

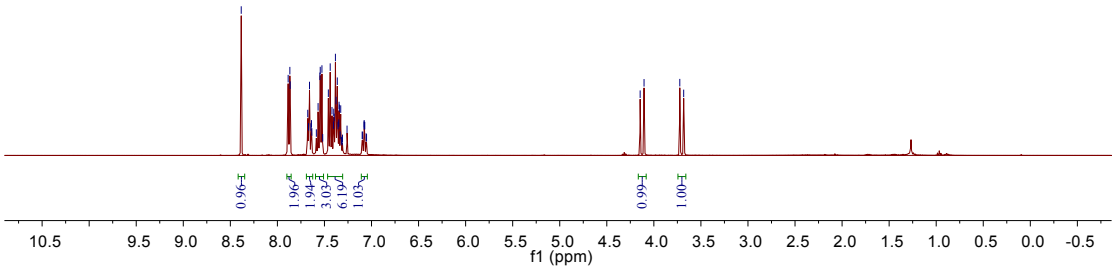


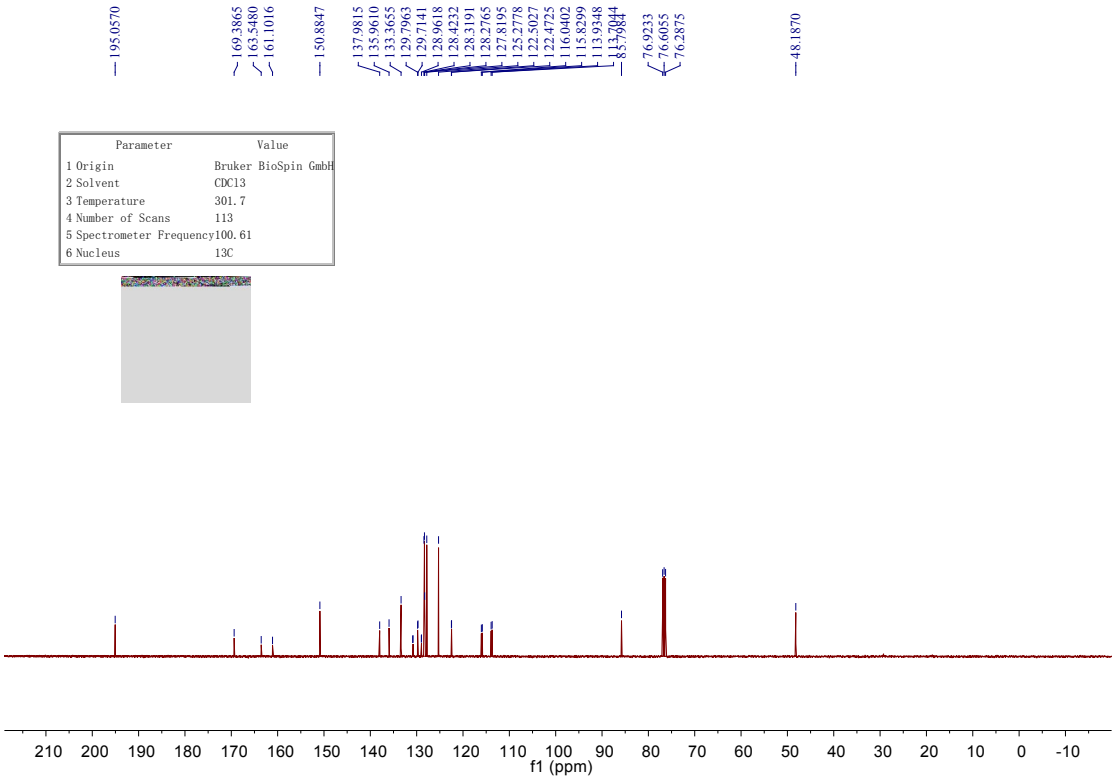
8.3843
7.8857
7.8676
7.8644
7.6775
7.6588
7.6379
7.5676
7.5486
7.5448
7.5268
7.4587
7.4389
7.4201
7.4009
7.3967
7.3834
7.3768
7.3635
7.3479
7.3447
7.3415
7.3271
7.0781
4.1436
4.1042
3.7244
3.6830

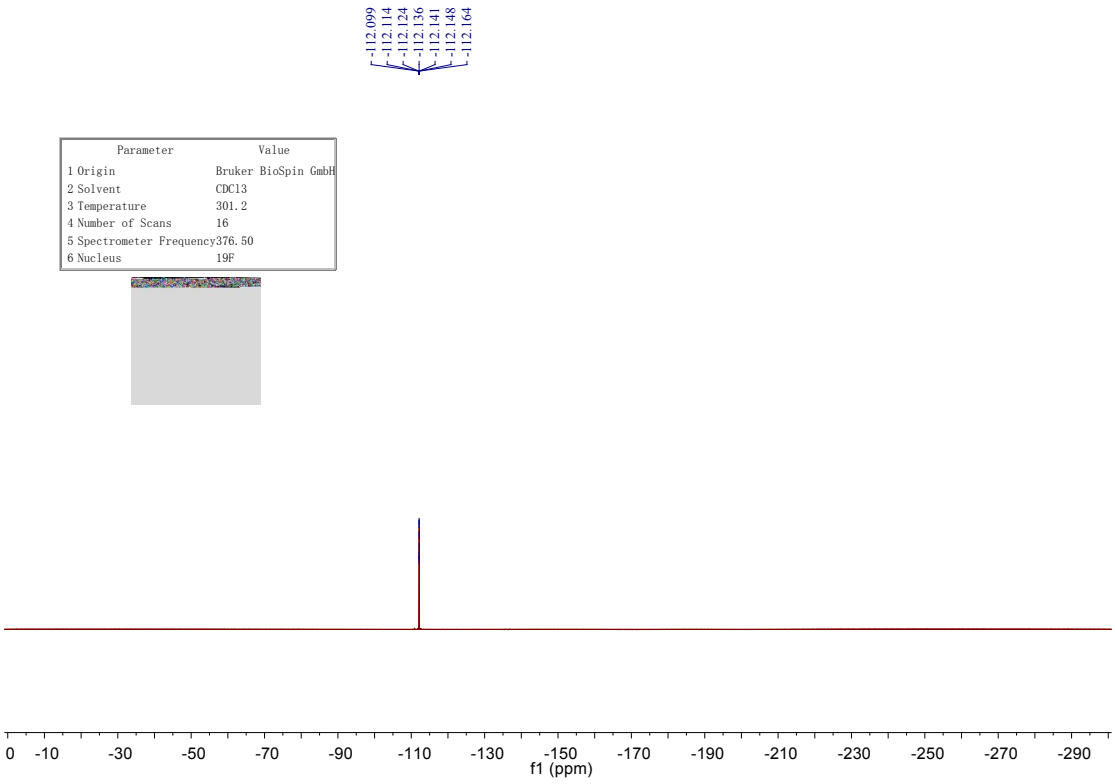
Parameter	Value
1 Origin	Bruker BioSpin GmbH
2 Solvent	CDCl3
3 Temperature	301.3
4 Number of Scans	8
5 Spectrometer Frequency	400.13
6 Nucleus	1H



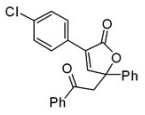
3k



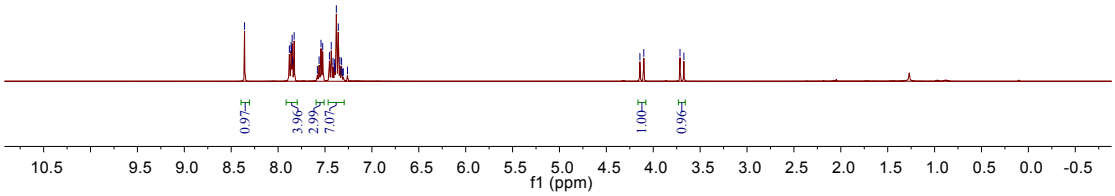

195.0570
169.3865
163.5480
161.1016
150.8847
137.9815
135.9610
133.3655
129.7963
129.7141
128.9618
128.4232
128.3191
128.2765
127.8195
125.2778
122.5027
122.4725
116.0402
115.8299
113.9348
113.7044
85.7984
76.9233
76.6055
76.2875
48.1870
Parameter Value
1 Origin Bruker BioSpin GmbH
2 Solvent CDCl3
3 Temperature 301.7
4 Number of Scans 113
5 Spectrometer Frequency 100.61
6 Nucleus 13C
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)


-112.099
-112.114
-112.124
-112.136
-112.141
-112.148
-112.164
Parameter Value
1 Origin Bruker BioSpin GmbH
2 Solvent CDCl3
3 Temperature 301.2
4 Number of Scans 16
5 Spectrometer Frequency 376.50
6 Nucleus 19F
0 -10 -30 -50 -70 -90 -110 -130 -150 -170 -190 -210 -230 -250 -270 -290
f1 (ppm)

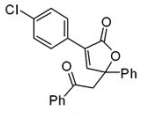
Parameter	Value
1 Origin	Bruker BioSpin GmbH
2 Solvent	CDCl3
3 Temperature	301.1
4 Number of Scans	4
5 Spectrometer Frequency	400.13
6 Nucleus	1H



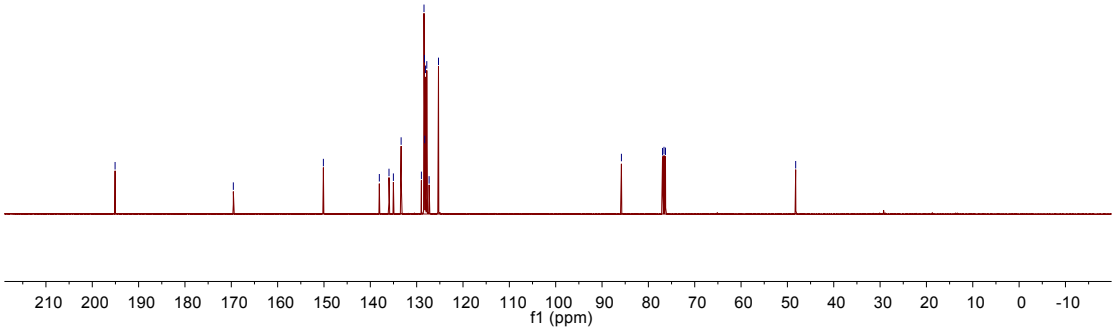
3l



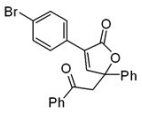
Parameter	Value
1 Origin	Bruker BioSpin GmbH
2 Solvent	CDCl3
3 Temperature	301.2
4 Number of Scans	1024
5 Spectrometer Frequency	100.61
6 Nucleus	13C



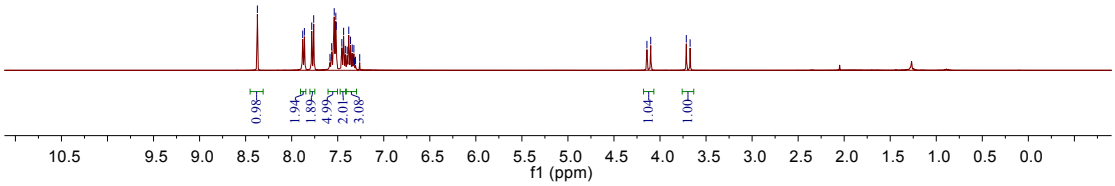
3l



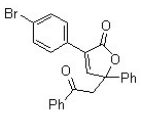
Parameter	Value
1 Origin	Bruker BioSpin GmbH
2 Solvent	CDCl3
3 Temperature	300.6
4 Number of Scans	4
5 Spectrometer Frequency	400.13
6 Nucleus	1H



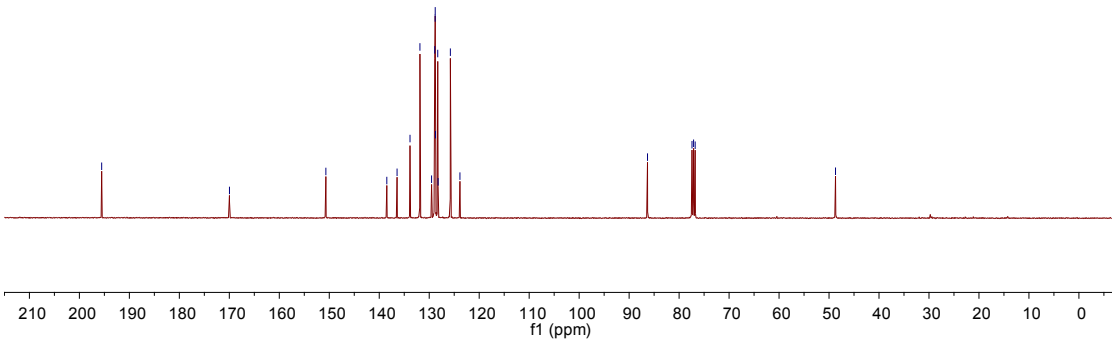
3m



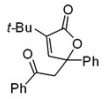
Parameter	Value
1 Origin	Bruker BioSpin GmbH
2 Solvent	CDCl3
3 Temperature	300.5
4 Number of Scans	1024
5 Spectrometer Frequency	100.62
6 Nucleus	13C



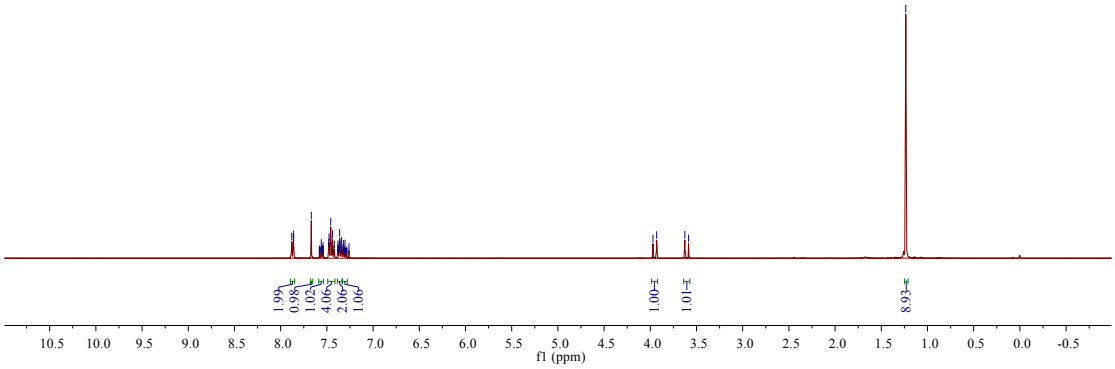
3m



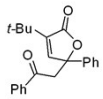
Parameter	Value
1 Origin	Bruker BioSpin GmbH
2 Solvent	CDCl3
3 Temperature	300.7
4 Number of Scans	4
5 Spectrometer Frequency	400.13
6 Nucleus	1H



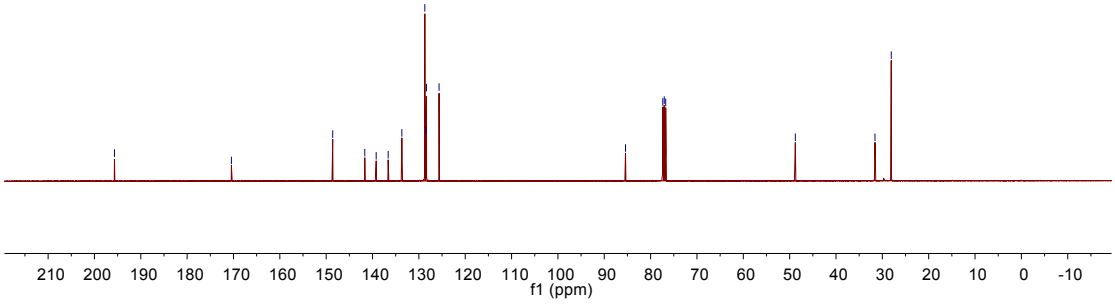
3n



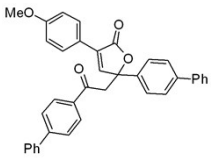
Parameter	Value
1 Origin	Bruker BioSpin GmbH
2 Solvent	CDCl3
3 Temperature	299.1
4 Number of Scans	1024
5 Spectrometer Frequency	100.62
6 Nucleus	13C



3n



Parameter	Value
1 Origin	Bruker BioSpin GmbH
2 Solvent	CDCl3
3 Temperature	300.7
4 Number of Scans	4
5 Spectrometer Frequency	400.13
6 Nucleus	1H



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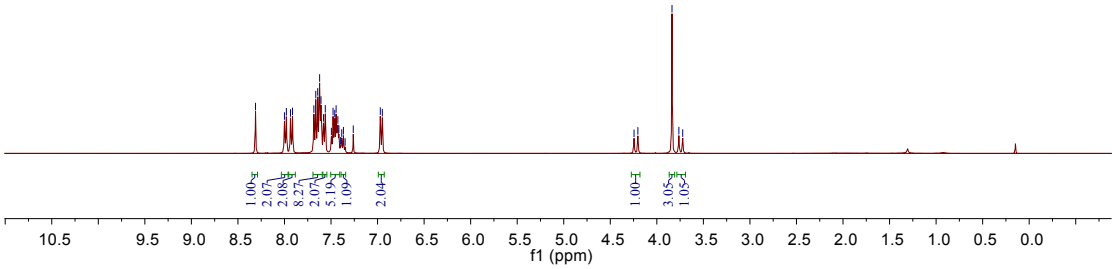
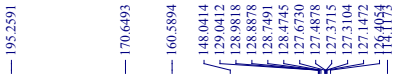
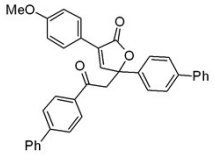




Parameter	Value
1 Origin	Bruker BioSpin GmbH
2 Solvent	CDCl3
3 Temperature	300.4
4 Number of Scans	1024
5 Spectrometer Frequency	100.62
6 Nucleus	13C



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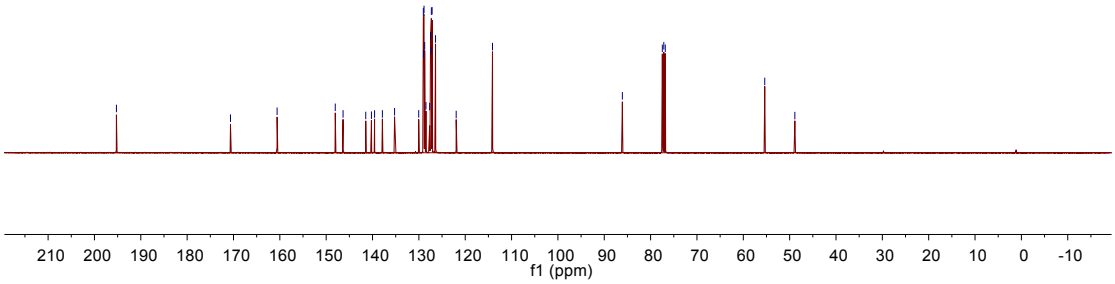
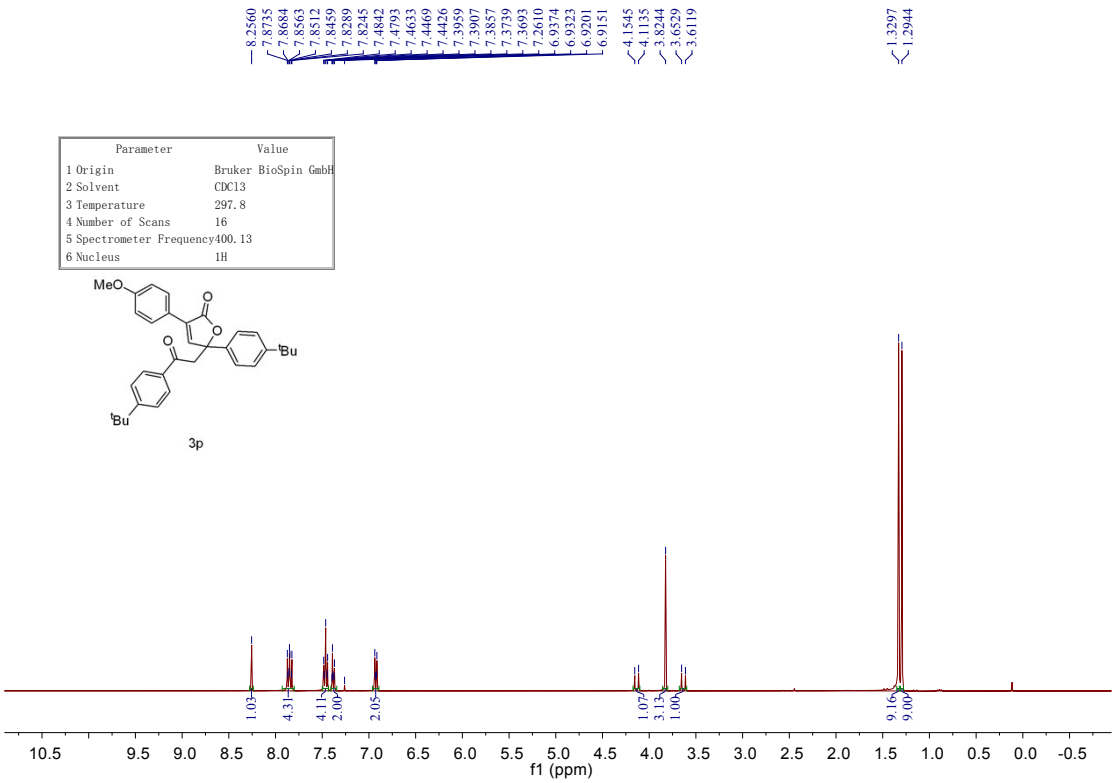
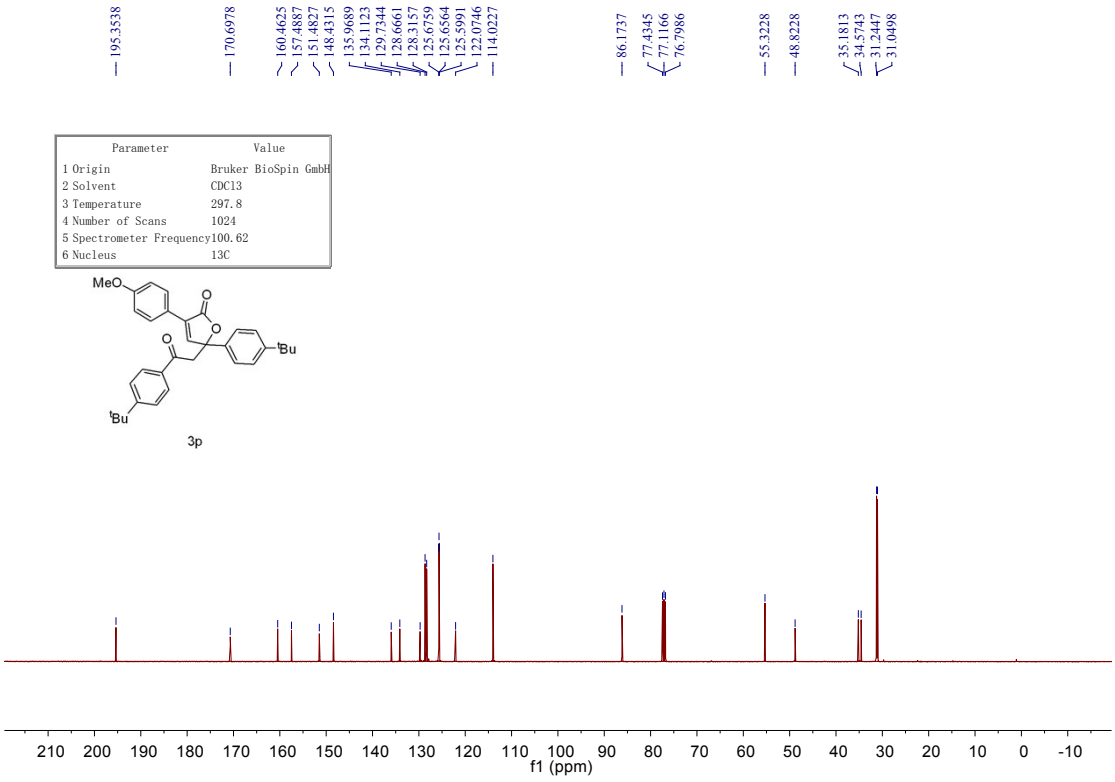
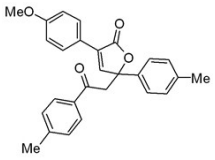



8.2560
7.8735
7.8684
7.8563
7.8512
7.8459
7.8289
7.8245
7.4842
7.4793
7.4633
7.4469
7.4426
7.3959
7.3907
7.3857
7.3739
7.3693
7.2610
6.9374
6.9323
6.9201
6.9151
4.1545
4.1135
3.8244
3.6529
3.6119
1.3297
1.2944
Parameter Value
1 Origin Bruker BioSpin GmbH
2 Solvent CDCl3
3 Temperature 297.8
4 Number of Scans 16
5 Spectrometer Frequency 400.13
6 Nucleus 1H
MeO
tBu
tBu
3p
1.03
4.31
4.11
2.00
2.05
1.07
3.13
1.00
9.16
9.00
f1 (ppm)

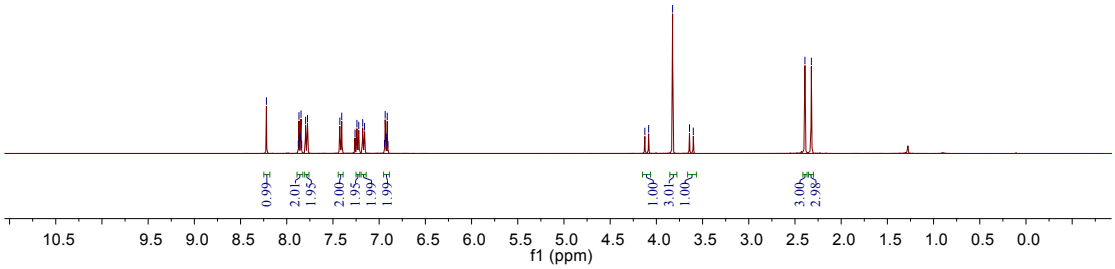

195.3538
170.6978
160.4625
157.4887
151.4827
148.4315
135.9689
134.1123
129.7344
128.6661
128.3157
125.6759
125.6564
125.5991
122.0746
114.0227
86.1737
77.4345
77.1166
76.7986
55.3228
48.8228
35.1813
34.5743
31.2447
31.0498
Parameter Value
1 Origin Bruker BioSpin GmbH
2 Solvent CDCl3
3 Temperature 297.8
4 Number of Scans 1024
5 Spectrometer Frequency 100.62
6 Nucleus 13C
MeO
tBu
tBu
3p
f1 (ppm)

8.2201
7.8751
7.8680
7.8629
7.8508
7.8457
7.8385
7.7963
7.7757
7.4258
7.4051
7.2610
7.2409
7.2208
7.1781
7.1581
6.9420
6.9349
6.9298
6.9177
6.9126
6.9053
4.1247
4.0837
3.8245
3.6415
3.6004
2.3924
2.3227

Parameter	Value
1 Origin	Bruker BioSpin GmbH
2 Solvent	CDCl3
3 Temperature	304.5
4 Number of Scans	4
5 Spectrometer Frequency	400.13
6 Nucleus	1H

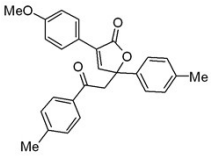


3q

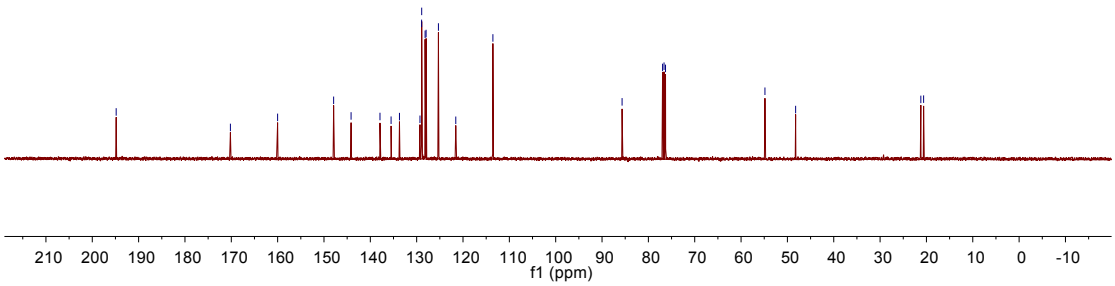


194.8422
170.1935
160.0067
147.9178
144.1543
137.9168
135.5209
133.7118
129.2864
128.9337
128.9155
128.1809
127.9659
125.3051
121.5599
113.5504
85.6745
76.9375
76.6196
76.3016
54.8469
48.2250
21.2080
20.6071

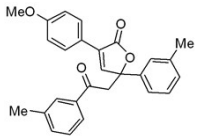
Parameter	Value
1 Origin	Bruker BioSpin GmbH
2 Solvent	CDCl3
3 Temperature	304.8
4 Number of Scans	61
5 Spectrometer Frequency	100.61
6 Nucleus	13C



3q



Parameter	Value
1 Origin	Bruker BioSpin GmbH
2 Solvent	CDCl3
3 Temperature	302.0
4 Number of Scans	4
5 Spectrometer Frequency	400.13
6 Nucleus	1H



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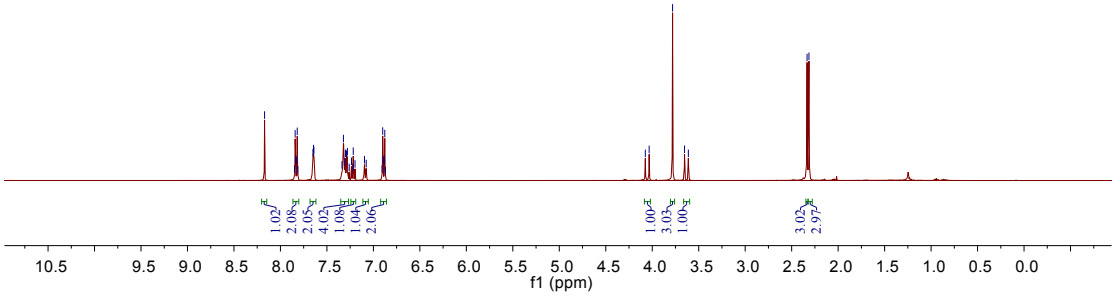
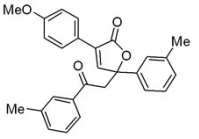
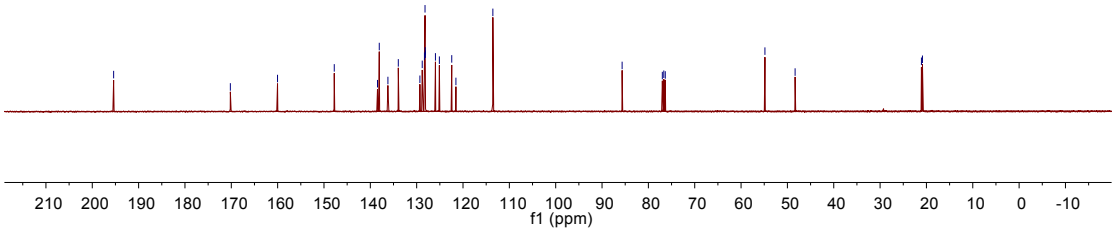
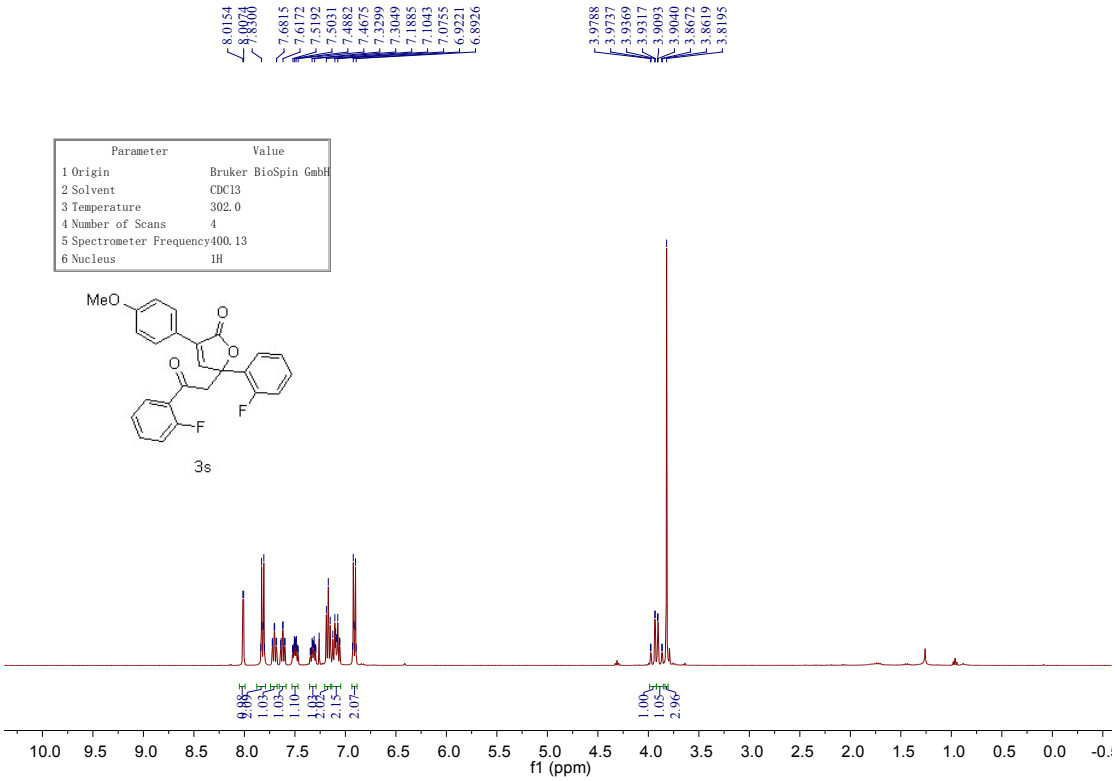


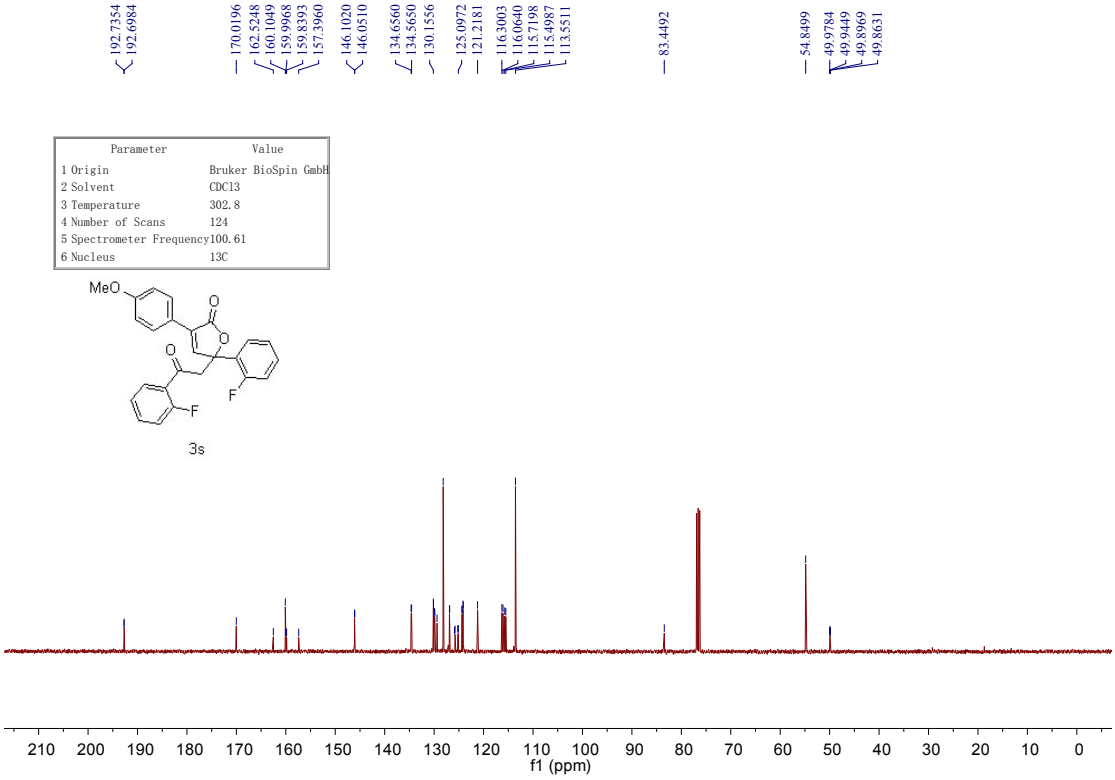
Parameter	Value
1 Origin	Bruker BioSpin GmbH
2 Solvent	CDCl3
3 Temperature	302.2
4 Number of Scans	112
5 Spectrometer Frequency	100.61
6 Nucleus	13C



3r

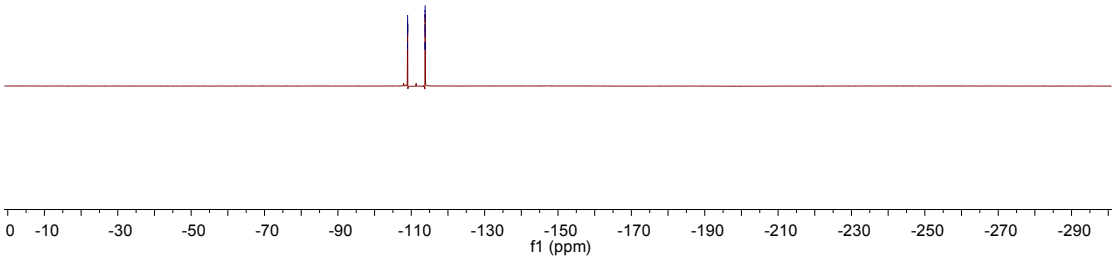



8.0154
8.0074
7.8500
7.6815
7.6172
7.5192
7.5031
7.4882
7.4675
7.3299
7.3049
7.1885
7.1043
7.0755
6.9221
6.8926
3.9788
3.9737
3.9369
3.9317
3.9093
3.9040
3.8672
3.8619
3.8195
Parameter Value
1 Origin Bruker BioSpin GmbH
2 Solvent CDCl3
3 Temperature 302.0
4 Number of Scans 4
5 Spectrometer Frequency 400.13
6 Nucleus 1H
MeO
O
O
O
F
F
3s
1.00
1.05
2.96
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
f1 (ppm)


192.7354
192.6984
170.0196
162.5248
160.1049
159.9968
159.8393
157.3960
146.1020
146.0510
134.6560
134.5650
130.1556
125.0972
121.2181
116.3003
116.0640
115.7198
115.4987
113.5511
83.4492
54.8499
49.9784
49.9449
49.8969
49.8631
Parameter Value
1 Origin Bruker BioSpin GmbH
2 Solvent CDCl3
3 Temperature 302.8
4 Number of Scans 124
5 Spectrometer Frequency 100.61
6 Nucleus 13C
MeO
O
O
O
F
F
3s
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)

-108.979
-108.993
-108.998
-109.008
-109.018
-109.022
-113.742
-113.753
-113.762
-113.773
-113.783
-113.793
-113.804

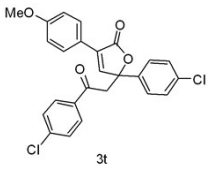
Parameter	Value
1 Origin	Bruker BioSpin GmbH
2 Solvent	CDCl3
3 Temperature	302.5
4 Number of Scans	16
5 Spectrometer Frequency	376.50
6 Nucleus	19F



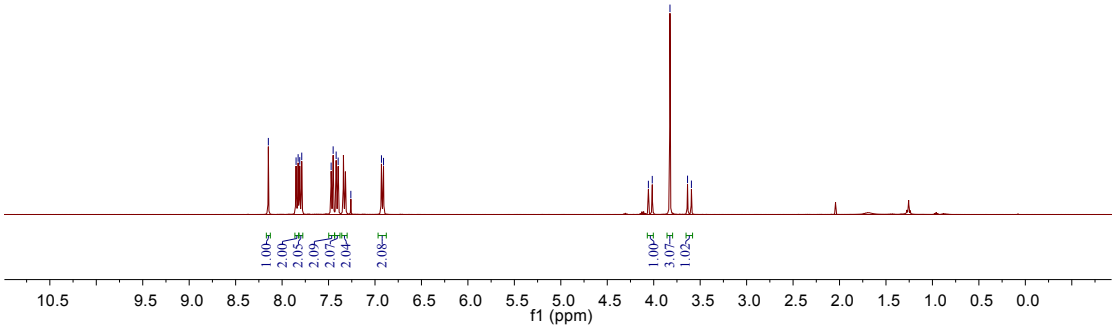
8.1480
7.8496
7.8274
7.8100
7.7885
7.4721
7.4505
7.4181
7.3966
7.2600
6.9305
6.9083

4.0573
4.0156
3.8250
3.6359
3.5943

Parameter	Value
1 Origin	Bruker BioSpin GmbH
2 Solvent	CDCl3
3 Temperature	301.2
4 Number of Scans	4
5 Spectrometer Frequency	400.13
6 Nucleus	1H

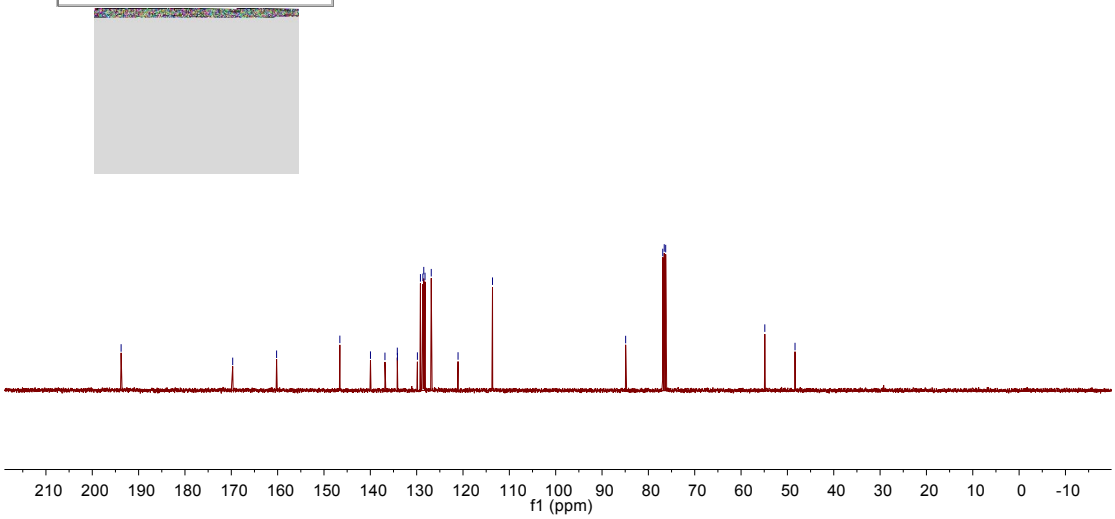


3t



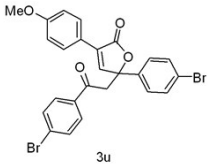
193.7770
169.6978
160.2262
146.5757
139.9679
136.8443
134.1762
134.1488
129.8410
129.1879
128.6610
128.4676
128.1997
126.8534
121.0709
113.6340
84.9043
76.8978
76.5801
76.2624
54.8757
48.3553

Parameter	Value
1 Origin	Bruker BioSpin GmbH
2 Solvent	CDCl3
3 Temperature	302.2
4 Number of Scans	80
5 Spectrometer Frequency	100.61
6 Nucleus	13C

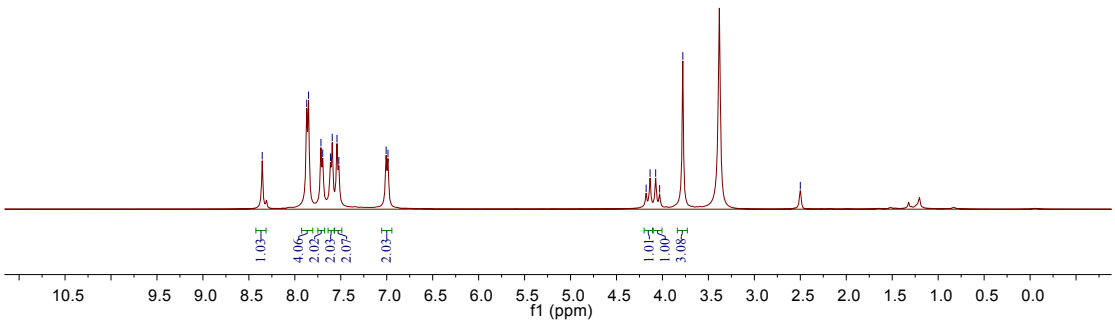


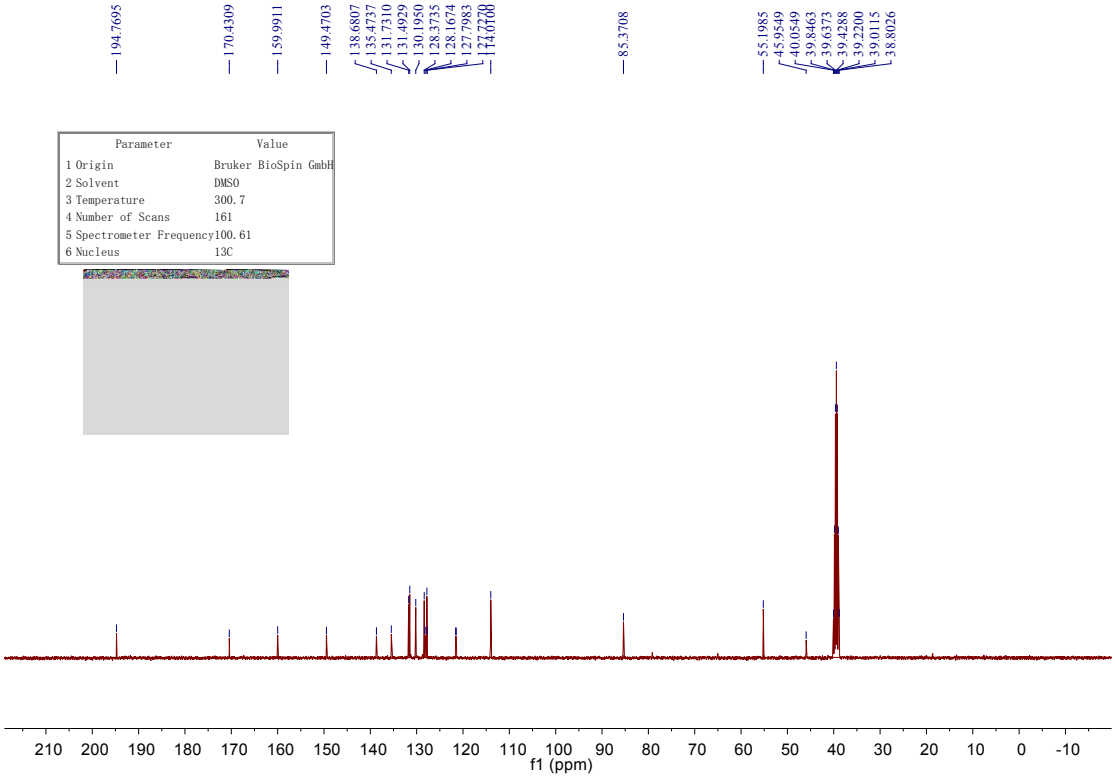
8.3548
7.8705
7.8507
7.7158
7.6962
7.5930
7.5412
7.5208
6.9865
4.1775
4.1348
4.0739
4.0309
3.7785
2.5000

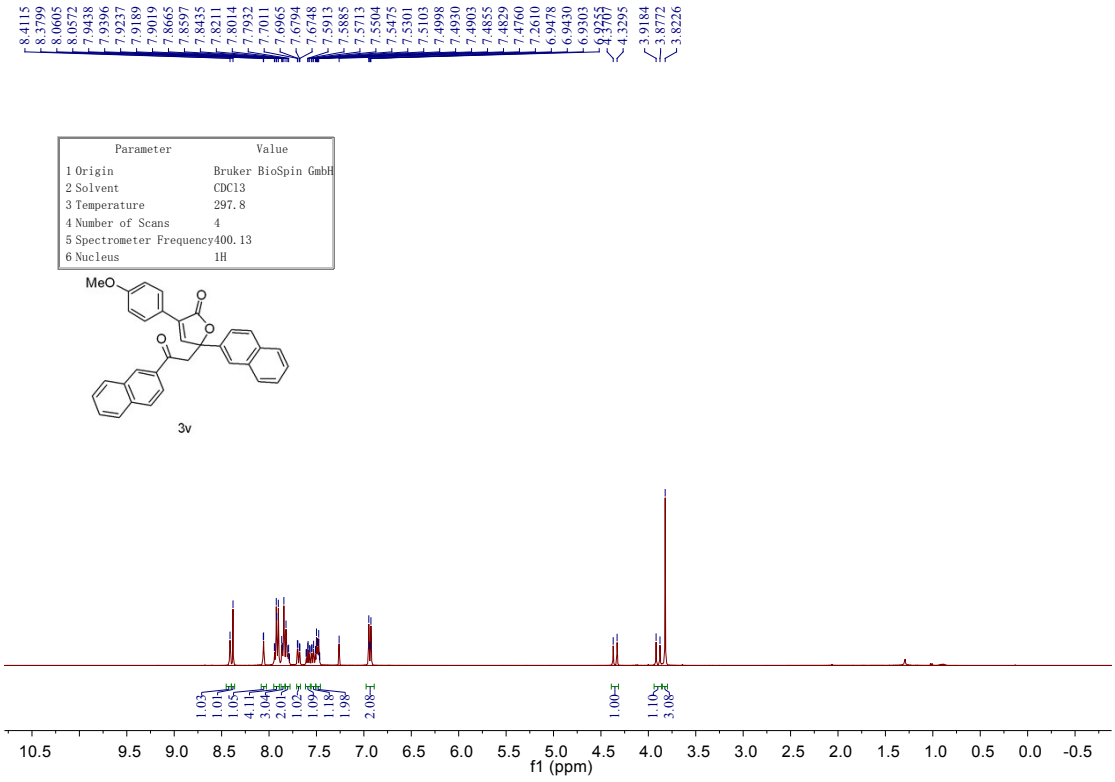
Parameter	Value
1 Origin	Bruker BioSpin GmbH
2 Solvent	DMSO
3 Temperature	301.6
4 Number of Scans	16
5 Spectrometer Frequency	400.13
6 Nucleus	1H



3u

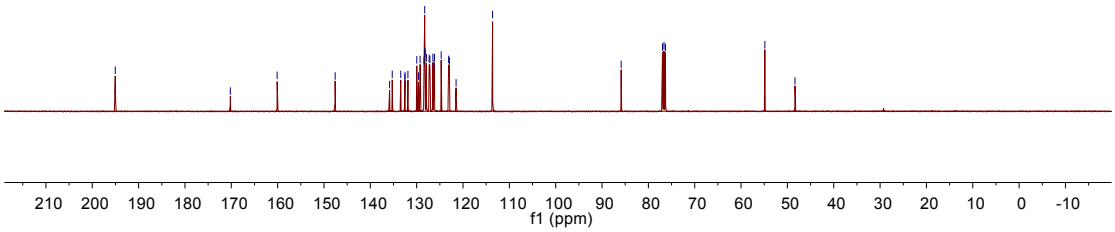



194.7695
170.4309
159.9911
149.4703
138.6807
135.4737
131.7310
131.4929
130.1950
128.3735
128.1674
127.7983
85.3708
55.1985
45.9549
40.0549
39.8463
39.6373
39.4288
39.2200
39.0115
38.8026
Parameter Value
1 Origin Bruker BioSpin GmbH
2 Solvent DMSO
3 Temperature 300.7
4 Number of Scans 161
5 Spectrometer Frequency 100.61
6 Nucleus 13C
f1 (ppm)


8.4115
8.3799
8.0605
8.0572
7.9438
7.9396
7.9237
7.9189
7.9019
7.8665
7.8597
7.8435
7.8211
7.8014
7.7932
7.7011
7.6965
7.6794
7.6748
7.5913
7.5885
7.5713
7.5504
7.5475
7.5301
7.5103
7.4998
7.4930
7.4903
7.4855
7.4829
7.4760
7.2610
6.9478
6.9430
6.9303
4.3707
4.3295
3.9184
3.8772
3.8226
Parameter Value
1 Origin Bruker BioSpin GmbH
2 Solvent CDCl3
3 Temperature 297.8
4 Number of Scans 4
5 Spectrometer Frequency 400.13
6 Nucleus 1H
MeO
3v
1.03
1.01
1.05
4.11
3.04
2.01
1.02
1.09
1.18
1.98
2.08
1.00
1.10
3.08
f1 (ppm)

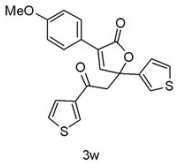
195.0371
170.2092
160.1076
147.5910
128.2679
128.2231
128.1866
127.8881
127.3041
126.5079
126.1598
85.8727
76.9524
76.6345
76.3167
54.8672
48.3595

Parameter	Value
1 Origin	Bruker BioSpin GmbH
2 Solvent	CDCl3
3 Temperature	297.8
4 Number of Scans	1024
5 Spectrometer Frequency	100.61
6 Nucleus	13C

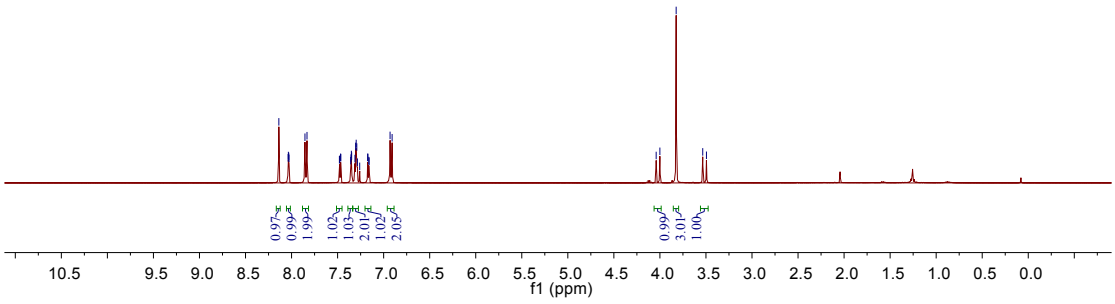


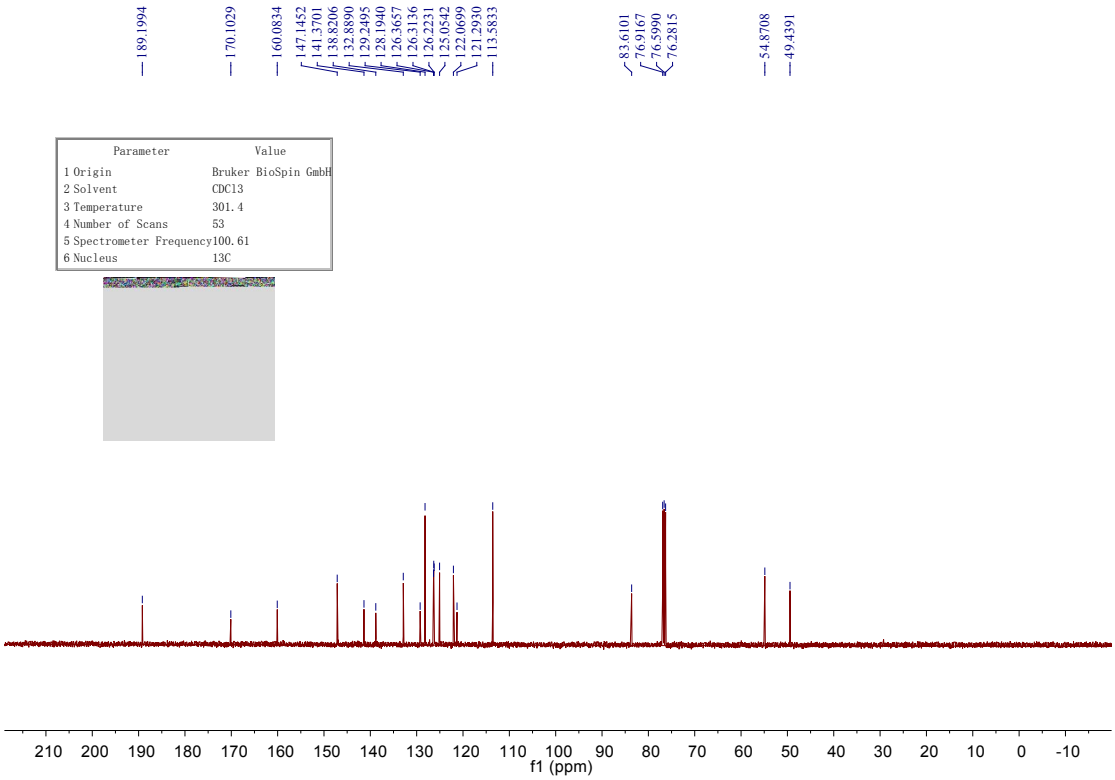
8.1380
8.0373
8.0347
8.0305
8.0278
7.8545
7.8324
7.4805
7.4780
7.4678
7.4653
7.3578
7.3547
7.3506
7.3475
7.3115
7.3043
7.2982
7.2916
7.2848
7.1738
7.1708
7.1611
7.1581
6.9303
6.8081
4.0406
4.0003
3.8235
3.5349
3.4946

Parameter	Value
1 Origin	Bruker BioSpin GmbH
2 Solvent	CDCl3
3 Temperature	301.1
4 Number of Scans	2
5 Spectrometer Frequency	400.13
6 Nucleus	1H



3w




189.1994
170.1029
160.0834
147.1452
141.3701
138.8206
132.8890
129.2495
128.1940
126.3657
126.3136
126.2231
125.0542
122.0699
121.2930
113.5833
83.6101
76.9167
76.5990
76.2815
54.8708
49.4391
Parameter Value
1 Origin Bruker BioSpin GmbH
2 Solvent CDCl3
3 Temperature 301.4
4 Number of Scans 53
5 Spectrometer Frequency 100.61
6 Nucleus 13C
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)