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Supporting Information

Synthesis of Highly Substituted γ-Hydroxybutenolides through the Annulation of Keto Acids with Alkynes and Subsequent Hydroxyl Transposition

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1. General experimental details

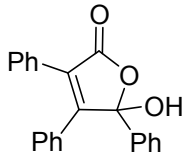
All reactions were maintained under air unless otherwise stated. Commercially available reagents were used without further purification. Infrared (FT-IR) spectra were recorded on a BRUKER VERTEX 70, ν_{max} in cm^{-1}. ^{1}H-NMR spectra were recorded on a BRUKER AVANCE III HD (400 MHz) spectrometer.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

Keto acids and alkynes were prepared according to the reported procedures.[1,2]

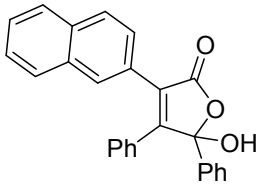
2. General procedure for the synthesis of γ-hydroxybutenolides

Keto acid (0.3 mmol, 1 equiv) and alkyne (0.6 mmol, 2 equiv) were loaded in a test tube. Then fluorobenzene (2 mL) and boron trifluoride etherate (0.09mmol, 0.3 equiv) were sequentially added to the test tube. The resulting reaction mixture was stirred at 70 °C under air until the starting materials had been consumed as determined by TLC. At the end of the reaction, the reaction mixture was cooled to room temperature. After removal of the solvent, the residue was subjected to column chromatography on silica gel (eluent: ethyl acetate/ petroleum ether) to afford the desired product.

3. Characterization of new compounds



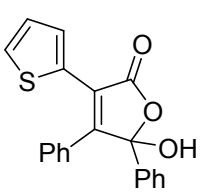
3a: white solid, m.p. 166-168 °C. ^{1}H NMR (400 MHz, DMSO) δ 8.69 (s, 1H), 7.56-7.51 (m, 2H), 7.45-7.32 (m, 8H), 7.26-7.18 (m, 5H); ^{13}C NMR (100 MHz, DMSO) δ 170.2, 158.6, 137.5, 130.5, 129.8, 129.5, 129.3, 128.9, 128.8, 128.6, 128.5, 128.4, 128.3, 126.9, 125.8, 105.7. FT-IR: ν (cm^{-1}) 3059, 2949, 2893, 2850, 1733, 1642, 1576. HRMS [ESI] calcd for $C_{22}H_{17}O_3$ $[M+H]^+$ 329.1178, found 329.1181; ESI $[M+H]^+$ 329.1.



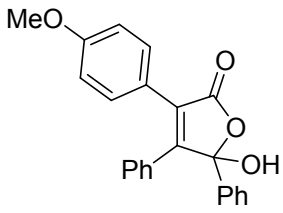
3b: white solid, m.p. 187-188 °C. ^{1}H NMR (400 MHz, DMSO) δ 8.79 (br, 1H), 8.16 (s, 1H), 7.95-7.86 (m, 3H), 7.61-7.57 (m, 2H), 7.57-7.51 (m, 2H), 7.41-7.30 (m, 4H), 7.29-7.22 (m, 3H), 7.22-7.16 (m, 2H); ^{13}C NMR (100 MHz, DMSO) δ 170.3, 158.7, 137.6, 132.7, 132.6, 130.5, 129.7, 129.1, 129.0, 128.8, 128.4, 128.3, 128.2, 127.9, 127.6, 127.4, 127.0, 126.8, 126.6, 125.9, 105.8. FT-IR: ν (cm^{-1}) 3052, 2984, 2923, 2894, 1741, 1653, 1598. HRMS [ESI] calcd for $C_{26}H_{19}O_3$ $[M+H]^+$ 379.1334, found 379.1332; ESI $[M+H]^+$ 379.1.

(1) (a) Breed, J.; Sills, M. R. *Tetrahedron Lett.* **2003**, *44*, 3131-3134. (b) Magnier, E.; Tordeux, M.; Wakselman, C. *Synlett* **2003**, 1374-1376. (c) Yang, L. M.; Huang, L. F.; Luh, T. Y. *Org. Lett.* **2004**, *6*, 1461-1463. (d) Diesendruck, C. E.; Zhu, L.; Moore, J. S. *Chem. Commun.* **2014**, *50*, 13235-13238.

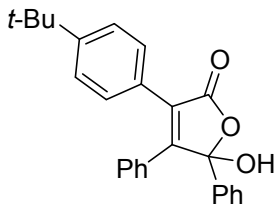
(2) (a) Schmidt, B.; Berger, R.; Kelling, A.; Schilde, U. *Chem. Eur. J.* **2011**, *17*, 7032-7040. (b) Zhang, J.; Ugrinov, A.; Zhao, P. *Angew. Chem. Int. Ed.* **2014**, *53*, 8437-8440. (c) Yang, L. M.; Huang, L. F.; Luh, T. Y. *Org. Lett.* **2004**, *6*, 1461-1463. (d) Diesendruck, C. E.; Zhu, L.; Moore, J. S. *Chem. Commun.* **2014**, *50*, 13235-13238.



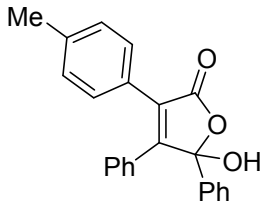
3c: brown oil. ^{1}H NMR (400 MHz, DMSO) δ 8.55 (s, 1H), 7.60 (dd, *J* = 5.2, 1.2 Hz, 1H), 7.45 (dd, *J* = 3.6, 1.2 Hz, 1H), 7.39-7.32 (m, 8H), 7.15-7.09 (m, 2H), 7.05 (dd, *J* = 5.2, 3.6 Hz, 1H); ^{13}C NMR (100 MHz, DMSO) δ 169.4, 156.6, 136.7, 130.9, 130.3, 129.5, 129.0, 128.8, 128.6, 128.3, 128.1, 127.2, 125.9, 120.8, 106.1. FT-IR: ν (cm^{-1}) 3061, 2925, 2849, 1754, 1643, 1490. HRMS [ESI] calcd for $C_{20}H_{15}O_3S$ $[M+H]^+$ 335.0742, found 335.0735; ESI $[M+H]^+$ 335.1.



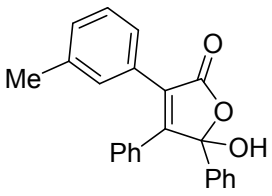
3d: white solid, m.p. 170-171 °C. ^{1}H NMR (400 MHz, DMSO) δ 8.60 (s, 1H), 7.51-7.47 (m, 2H), 7.39-7.31 (m, 5H), 7.29-7.16 (m, 5H), 6.95 (d, *J* = 8.8 Hz, 2H), 3.75 (s, 3H); ^{13}C NMR (100 MHz, DMSO) δ 170.4, 159.6, 157.3, 137.6, 130.9, 130.7, 129.4, 128.9, 128.6, 128.4, 128.3, 126.3, 125.8, 121.7, 113.9, 105.4, 55.1. FT-IR: ν (cm^{-1}) 3063, 3033, 2964, 2934, 2840, 1731, 1656, 1569. HRMS [ESI] calcd for $C_{23}H_{19}O_4$ $[M+H]^+$ 359.1283, found 359.1291; ESI $[M+H]^+$ 359.1.



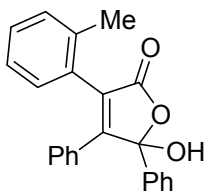
3e: white solid, m.p. 86-88 °C. ^{1}H NMR (400 MHz, DMSO) δ 8.62 (s, 1H), 7.49-7.45 (m, 2H), 7.42-7.38 (m, 2H), 7.38-7.32 (m, 5H), 7.27-7.15 (m, 5H), 1.27 (s, 9H); ^{13}C NMR (100 MHz, DMSO) δ 170.3, 158.3, 151.4, 137.5, 130.8, 129.4, 129.0, 128.9, 128.5, 128.4, 128.3, 126.8, 126.6, 125.8, 125.2, 105.6, 34.4, 31.0. FT-IR: ν (cm^{-1}) 3060, 2962, 2904, 2868, 1736, 1608, 1509. HRMS [ESI] calcd for $C_{26}H_{25}O_3$ $[M+H]^+$ 385.1804, found 385.1812; ESI $[M+H]^+$ 385.1.



3f: white solid, m.p. 155-157 °C. ^{1}H NMR (400 MHz, DMSO) δ 8.64 (s, 1H), 7.50 (d, *J* = 6.8 Hz, 2H), 7.39-7.29 (m, 5H), 7.26-7.17 (m, 7H), 2.30 (s, 3H); ^{13}C NMR (100 MHz, DMSO) δ 170.3, 158.1, 138.4, 137.6, 130.7, 129.4, 129.2, 129.1, 128.9, 128.6, 128.4, 128.3, 126.8, 126.7, 125.8, 105.6, 20.9. FT-IR: ν (cm^{-1}) 3060, 3032, 2922, 2853, 1754, 1645, 1574. HRMS [ESI] calcd for $C_{23}H_{19}O_3$ $[M+H]^+$ 343.1334, found 343.1343; ESI $[M+H]^+$ 343.1.

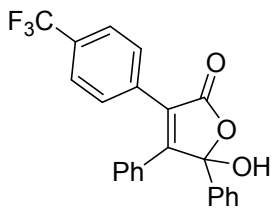


3g: yellow oil. ^{1}H NMR (400 MHz, DMSO) δ 8.68 (br, 1H), 7.54-7.50 (m, 2H), 7.39-7.33 (m, 3H), 7.30-7.14 (m, 9H), 2.27 (s, 3H); ^{13}C NMR (100 MHz, DMSO) δ 174.9, 163.2, 142.4, 142.3, 135.3, 134.6, 134.5, 134.3, 134.2, 133.7, 133.4, 133.2, 133.1, 133.0, 131.8, 131.2, 130.6, 110.4, 25.7. FT-IR: ν (cm^{-1}) 3059, 2959, 2922, 2852, 1753, 1645, 1584. HRMS [ESI] calcd for $C_{23}H_{19}O_3$ $[M+H]^+$ 343.1334, found 343.1327; ESI $[M+H]^+$ 343.1.

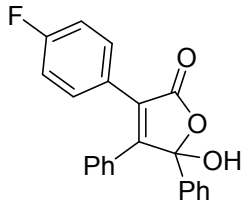


3h: white solid, m.p. 120-122 °C. ^{1}H NMR (400 MHz, DMSO, 80 °C) δ 8.53 (s, 1H), 7.58 (d, *J* = 7.2 Hz, 2H), 7.43-7.30 (m, 5H), 7.28-7.13 (m, 7H), 2.21 (s, 3H); ^{13}C NMR (100 MHz, DMSO, 80 °C) δ 169.8, 158.9, 138.2, 136.7, 130.5, 130.4, 130.2, 129.7, 128.8, 128.8, 128.5, 128.4, 128.2,

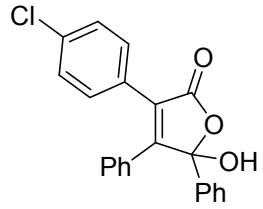
128.1, 126.0, 125.7, 106.0, 19.3. FT-IR: ν (cm^{-1}) 3063, 3026, 2924, 2855, 1731, 1639, 1573. HRMS [ESI] calcd for $C_{23}H_{19}O_3$ $[M+H]^+$ 343.1334, found 343.1339; ESI $[M+H]^+$ 343.1.



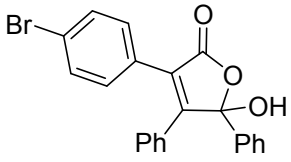
3i: white solid, m.p. 52-54 °C. ^{1}H NMR (400 MHz, DMSO) δ 8.79 (br, 1H), 7.78 (d, J = 8.4 Hz, 2H), 7.64 (d, J = 8.4 Hz, 2H), 7.55 (d, J = 6.8 Hz, 2H), 7.39-7.32 (m, 3H), 7.28-7.18 (m, 5H); ^{13}C NMR (100 MHz, DMSO) δ 169.7, 160.2, 137.1, 134.2 (q, $J_{C\text{-}F}$ = 1.2 Hz), 130.3, 130.0, 129.9, 129.1 (q, $J_{C\text{-}F}$ = 31.8 Hz), 129.0, 128.6, 128.5, 128.4, 125.9, 125.8, 125.4 (q, $J_{C\text{-}F}$ = 3.6 Hz), 124.0 (q, $J_{C\text{-}F}$ = 270.0 Hz), 105.9; ^{19}F NMR (376 MHz, DMSO) δ -61.3. FT-IR: ν (cm^{-1}) 3063, 3036, 2985, 2935, 2849, 1735, 1646, 1575. HRMS [ESI] calcd for $C_{23}H_{16}F_3O_3$ $[M+H]^+$ 397.1052, found 397.1045; ESI $[M+H]^+$ 397.1.



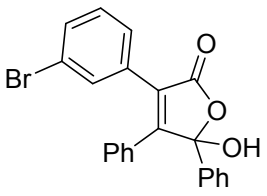
3j: white solid, m.p. 85-86 °C. ^{1}H NMR (400 MHz, DMSO) δ 8.64 (s, 1H), 7.50-7.47 (m, 2H), 7.46-7.41 (m, 2H), 7.39-7.32 (m, 3H), 7.30-7.21 (m, 5H), 7.18-7.14 (m, 2H); ^{13}C NMR (100 MHz, DMSO) δ 170.1, 162.2 (d, $J_{C\text{-}F}$ = 245.2 Hz), 158.7, 137.3, 131.6 (d, $J_{C\text{-}F}$ = 8.4 Hz), 130.4, 129.6, 128.9, 128.6, 128.4, 128.4, 126.1 (d, $J_{C\text{-}F}$ = 3.2 Hz), 125.9, 125.9, 115.6 (d, $J_{C\text{-}F}$ = 21.6 Hz), 105.7; ^{19}F NMR (376 MHz, DMSO) δ -112.0. FT-IR: ν (cm^{-1}) 3063, 2985, 2934, 2848, 1754, 1647, 1599. HRMS [ESI] calcd for $C_{22}H_{16}FO_3$ $[M+H]^+$ 347.1083, found 347.1086; ESI $[M+H]^+$ 347.1.



3k: white solid, m.p. 82-84 °C. ^{1}H NMR (400 MHz, DMSO) δ 8.67 (s, 1H), 7.51-7.46 (m, 4H), 7.42-7.32 (m, 5H), 7.29-7.22 (m, 3H), 7.18-7.15 (m, 2H); ^{13}C NMR (100 MHz, DMSO) δ 169.9, 159.1, 137.2, 133.7, 131.2, 130.3, 129.7, 129.0, 128.7, 128.6, 128.6, 128.4, 128.4, 125.9, 125.8, 105.8. FT-IR: ν (cm^{-1}) 3061, 3036, 2928, 2828, 1752, 1645, 1592. HRMS [ESI] calcd for $C_{22}H_{16}ClO_3$ $[M+H]^+$ 363.0788, found 363.0798; ESI $[M+H]^+$ 363.1.

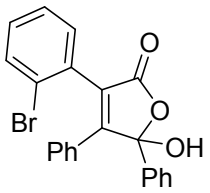


3l: white solid, m.p. 47-49 °C. ^{1}H NMR (400 MHz, DMSO) δ 8.71 (s, 1H), 7.61 (d, J = 8.4 Hz, 2H), 7.52 (d, J = 6.4 Hz, 2H), 7.39-7.31 (m, 5H), 7.28-7.18 (m, 5H); ^{13}C NMR (100 MHz, DMSO) δ 169.8, 159.1, 137.2, 131.6, 131.5, 130.3, 129.7, 129.1, 129.0, 128.6, 128.4, 128.4, 125.9, 125.9, 122.4, 105.8. FT-IR: ν (cm^{-1}) 3061, 2982, 2936, 2871, 1755, 1646, 1587. HRMS [ESI] calcd for $C_{22}H_{16}BrO_3$ $[M+H]^+$ 407.0283, found 407.0278; ESI $[M+H]^+$ 407.1.

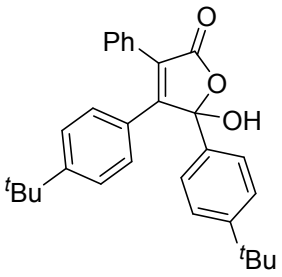


3m: white solid, m.p. 112-113 °C. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.57 (dd, J = 1.6, 1.6 Hz, 1H), 7.46-7.40 (m, 3H), 7.32-7.24 (m, 5H), 7.21-7.07 (m, 5H), 4.14 (br, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 170.4, 160.0, 136.0, 131.8, 131.6, 130.8, 129.6, 129.5, 129.4, 129.0, 128.4, 128.1, 128.1, 127.7, 125.5, 125.3, 121.9, 105.5. FT-IR: ν (cm^{-1}) 3061, 3035, 2929, 2830,

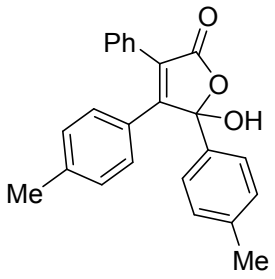
1753, 1645, 1592. HRMS [ESI] calcd for $C_{22}H_{16}BrO_3$ $[M+H]^+$ 407.0283, found 407.0281; ESI $[M+H]^+$ 407.0.



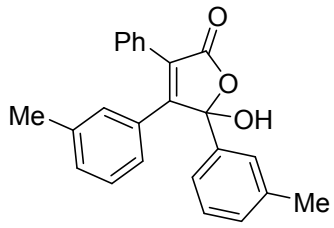
3n: white solid, m.p. 217-219 °C. ^{1}H NMR (400 MHz, DMSO) δ 8.85 (s, 1H), 7.84 (d, J = 7.2 Hz, 1H), 7.61 (d, J = 7.2 Hz, 2H), 7.49-7.31 (m, 5H), 7.30-7.15 (m, 6H); ^{13}C NMR (100 MHz, DMSO) δ 169.7, 156.8, 136.3, 134.5, 133.8, 130.4, 129.3, 129.3, 129.2, 129.1, 128.7, 128.6, 128.5, 127.9, 127.5, 104.9. FT-IR: ν (cm^{-1}) 3091, 2951, 2880, 2842, 1729, 1638, 1585. HRMS [ESI] calcd for $C_{22}H_{16}BrO_3$ $[M+H]^+$ 407.0283, found 407.0288; ESI [M+H]+ 407.0.



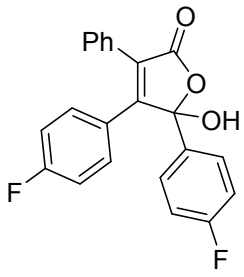
3o: white solid, m.p. 203-204 °C. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.46 (d, J = 8.4 Hz, 2H), 7.43-7.40 (m, 2H), 7.37 (d, J = 8.4 Hz, 2H), 7.34-7.28 (m, 3H), 7.19-7.13 (m, 4H), 4.16 (br, 1H), 1.30 (s, 9H), 1.23 (s, 9H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 170.5, 158.0, 152.8, 151.9, 133.8, 129.4, 129.1, 128.6, 128.3, 128.0, 126.8, 126.6, 125.2, 125.1, 124.8, 105.2, 34.3, 34.2, 30.8, 30.6. FT-IR: ν (cm^{-1}) 3061, 2965, 2906, 2869, 1736, 1633, 1574. HRMS [ESI] calcd $C_{30}H_{32}O_3Na$ for $[M+Na]^+$ 463.2249, found 463.2242; ESI $[M+H]^+$ 441.2.



3p: white solid, m.p. 170-171 °C. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.40-7.34 (m, 4H), 7.30-7.23 (m, 3H), 7.12-7.07 (m, 4H), 6.95 (d, J = 8.0 Hz, 2H), 4.87 (s, 1H), 2.30 (s, 3H), 2.24 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 170.9, 158.7, 139.5, 138.7, 133.6, 129.2, 129.1, 128.7, 128.6, 128.6, 128.3, 128.0, 127.0, 126.2, 125.5, 105.4, 20.9, 20.7. FT-IR: ν (cm^{-1}) 3058, 2943, 2894, 2850, 1733, 1623, 1504. HRMS [ESI] calcd for $C_{24}H_{21}O_3$ $[M+H]^+$ 357.1491, found 357.1483; ESI $[M+H]^+$ 357.1.

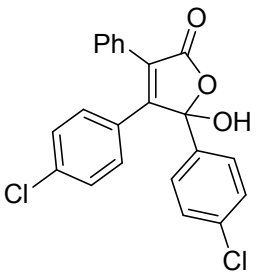


3q: white solid, m.p. 147-149 °C. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.41-7.37 (m, 2H), 7.30-7.22 (m, 5H), 7.19 (dd, J = 7.6, 7.6 Hz, 1H), 7.13 (d, J = 7.6 Hz, 1H), 7.06-7.02 (m, 2H), 6.97 (s, 1H), 6.93-6.90 (m, 1H), 4.69 (br, 1H), 2.30 (s, 3H), 2.15 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 170.8, 159.0, 137.8, 137.5, 136.3, 130.0, 129.9, 129.6, 129.1, 129.0, 128.9, 128.4, 127.9, 127.7, 126.5, 126.1, 125.7, 122.8, 105.3, 21.0, 20.9. FT-IR: ν (cm^{-1}) 3026, 2955, 2922, 2853, 1740, 1652, 1582. HRMS [ESI] calcd for $C_{24}H_{21}O_3$ $[M+H]^+$ 357.1491, found 357.1480; ESI $[M+H]^+$ 357.1.

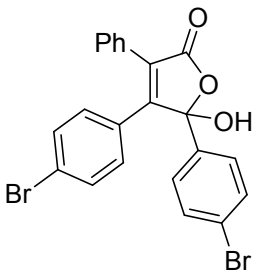


3r: white solid, m.p. 43-44 °C. ^{1}H NMR (400 MHz, DMSO) δ 8.78 (s, 1H), 7.58-7.53 (m, 2H), 7.43-7.37 (m, 5H), 7.27-7.23 (m, 2H), 7.20-7.15 (m, 2H), 7.11-7.06 (m, 2H); ^{13}C NMR (100 MHz, DMSO) δ 169.9, 163.6 (d, J_{C-F} = 18.6 Hz), 161.1 (d, J_{C-F} = 15.9 Hz), 157.2, 133.7 (d, J_{C-F} = 2.9 Hz), 131.0 (d, J_{C-F} = 8.6 Hz), 129.6, 129.3, 128.9, 128.5, 128.3 (d,

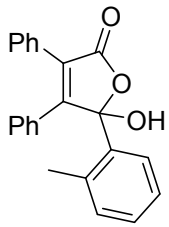
$J_{C\text{-}F}$ = 8.6 Hz), 127.1, 126.8 (d, $J_{C\text{-}F}$ = 3.3 Hz), 115.6 (d, $J_{C\text{-}F}$ = 21.8 Hz), 115.3 (d, $J_{C\text{-}F}$ = 21.7 Hz), 105.1; ^{19}F NMR (376 MHz, DMSO) δ -110.4, -112.8. FT-IR: ν (cm^{-1}) 3076, 2985, 2939, 2864, 1735, 1649, 1509. HRMS [ESI] calcd for $C_{22}H_{15}F_2O_3$ $[M+H]^+$ 365.0989, found 365.0980; ESI $[M+H]^+$ 365.1.



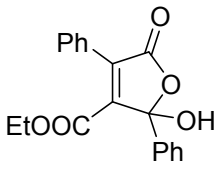
3s: white solid, m.p. 79-80 °C. ^{1}H NMR (400 MHz, DMSO) δ 8.83 (s, 1H), 7.52-7.49 (m, 2H), 7.44-7.38 (m, 7H), 7.36-7.32 (m, 2H), 7.20-7.17 (m, 2H); ^{13}C NMR (100 MHz, DMSO) δ 169.7, 156.8, 136.2, 134.5, 133.8, 130.4, 129.3, 129.3, 129.2, 129.1, 128.7, 128.6, 127.9, 127.5, 104.9. FT-IR: ν (cm^{-1}) 3091, 2924, 2854, 1739, 1644, 1591. HRMS [ESI] calcd for $C_{22}H_{14}Cl_2O_3Na$ $[M+Na]^+$ 419.0218, found 419.0212; ESI $[M+H]^+$ 397.0.



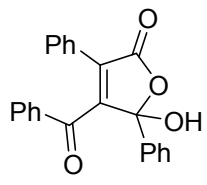
3t: white solid, m.p. 107-108 °C. ^{1}H NMR (400 MHz, DMSO) δ 8.84 (br, 1H), 7.57-7.54 (m, 2H), 7.48-7.43 (m, 4H), 7.42-7.38 (m, 5H), 7.15-7.12 (m, 2H); ^{13}C NMR (100 MHz, DMSO) δ 169.7, 156.9, 136.7, 131.6, 131.5, 130.6, 129.5, 129.3, 129.3, 129.1, 128.6, 128.2, 127.5, 123.4, 122.5, 105.0. FT-IR: ν (cm^{-1}) 3058, 2982, 2937, 2828, 1756, 1648, 1586. HRMS [ESI] calcd for $C_{22}H_{14}Br_2O_3Na$ $[M+Na]^+$ 506.9207, found 506.9203; ESI $[M+H]^+$ 484.9.



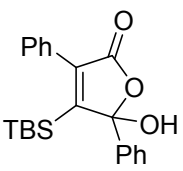
3u: white solid, m.p. 224-226 °C. ^{1}H NMR (400 MHz, DMSO) δ 8.55 (s, 1H), 7.72 (dd, J = 7.6, 1.6 Hz, 1H), 7.42-7.34 (m, 5H), 7.27-7.13 (m, 8H), 2.29 (s, 3H); ^{13}C NMR (100 MHz, DMSO) δ 170.5, 157.9, 135.5, 134.7, 132.0, 130.5, 129.8, 129.6, 129.2, 129.1, 128.9, 128.6, 128.4, 128.0, 127.6, 125.9, 105.7, 20.0. FT-IR: ν (cm^{-1}) 3051, 2968, 2927, 2850, 1752, 1648, 1575. HRMS [ESI] calcd for $C_{23}H_{18}O_3Na$ $[M+Na]^+$ 365.1154, found 365.1147; ESI $[M+H]^+$ 343.1.



3v: yellow oil. ^{1}H NMR (400 MHz, DMSO) δ 8.67 (br, 1H), 7.57-7.47 (m, 7H), 7.45-7.40 (m, 3H), 4.08-3.99 (m, 2H), 0.93 (t, J = 7.2 Hz, 3H); ^{13}C NMR (100 MHz, DMSO) δ 168.9, 161.4, 148.4, 136.7, 133.5, 130.0, 129.2, 129.1, 128.3, 128.2, 128.1, 125.9, 104.8, 61.3, 13.4. FT-IR: ν (cm^{-1}) 3061, 2983, 2928, 2852, 1766, 1666, 1576. HRMS [ESI] calcd for $C_{19}H_{16}O_5Na$ $[M+Na]^+$ 347.0895, found 347.0904; ESI $[M+H]^+$ 325.1.

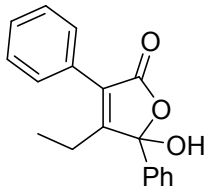


3w: white solid, m.p. 150-152 °C. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.59-7.56 (m, 2H), 7.53-7.47 (m, 4H), 7.40 (dd, J = 7.6, 7.6 Hz, 1H), 7.32-7.27 (m, 3H), 7.24-7.16 (m, 5H), 5.41 (br, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 192.2, 169.1, 154.7, 135.5, 134.2, 134.0, 129.7, 129.3, 129.0, 129.0, 128.5, 128.2, 128.2, 128.1, 127.1, 125.4, 104.1. FT-IR: ν (cm^{-1}) 3059, 3037, 2925, 2853, 1752, 1647, 1581. HRMS [ESI] calcd for $C_{23}H_{16}O_4Na$ $[M+Na]^+$ 379.0946, found 379.0934; ESI $[M+H]^+$ 357.1.

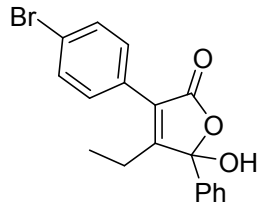


3x: white solid, m.p. 182-184 °C. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.58-7.54 (m,

2H), 7.44-7.34 (m, 6H), 7.33-7.29 (m, 2H), 4.68 (br, 1H), 0.58 (s, 9H), -0.04 (s, 3H), -0.33 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 172.0, 164.5, 143.4, 137.0, 131.5, 129.4, 128.7, 128.4, 128.0, 127.5, 125.7, 109.1, 26.7, 17.5, -3.1, -4.4. FT-IR: ν (cm^{-1}) 3056, 2955, 2931, 2857, 1732, 1691, 1591. HRMS [ESI] calcd for $C_{22}H_{27}O_3Si$ $[M+H]^+$ 367.1729, found 367.1721; ESI $[M+H]^+$ 367.1.

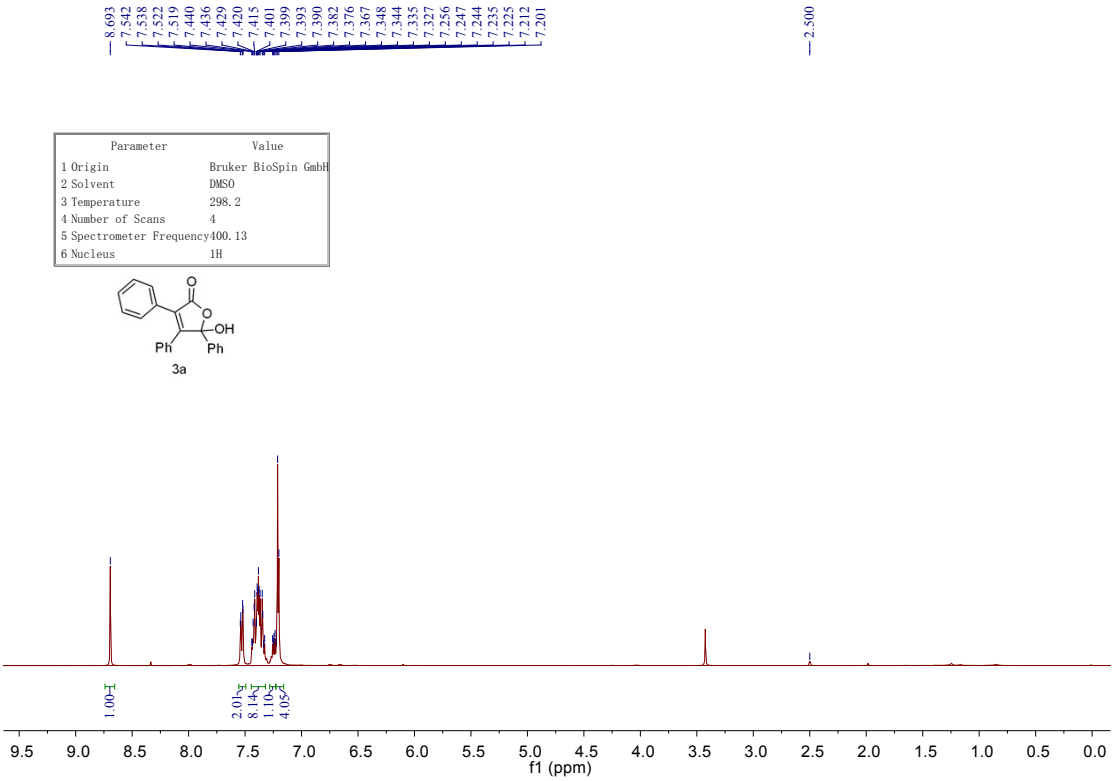


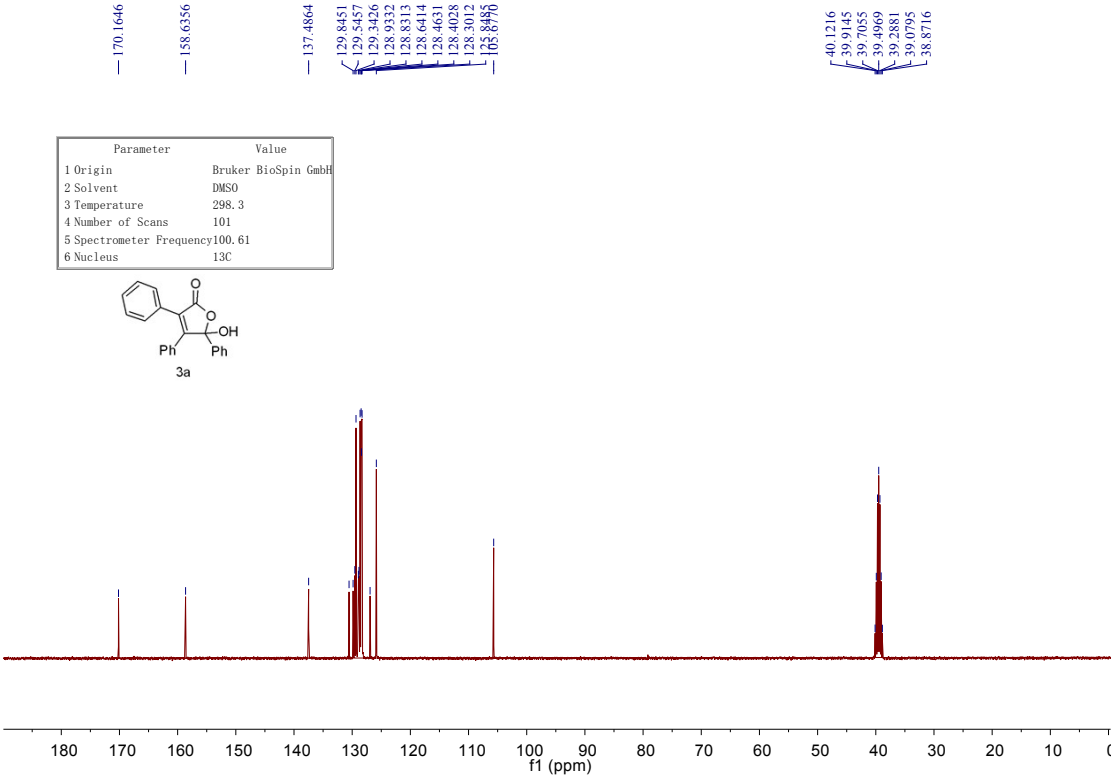
3y: white solid, m.p. 120-121 °C. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.55-7.51 (m, 2H), 7.46-7.36 (m, 8H), 4.83 (br, 1H), 2.51-2.41 (m, 1H), 2.34-2.24 (m, 1H), 0.90 (t, *J* = 7.6 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 171.7, 165.0, 136.4, 129.0, 128.9, 128.5, 128.3, 128.2, 128.0, 126.6, 125.4, 105.6, 19.1, 11.9. FT-IR: ν (cm-1) 3075, 2972, 2931, 2874, 1736, 1655, 1494. HRMS [ESI] calcd for $C_{18}H_{16}O_3Na$ $[M+Na]^+$ 303.0997, found 303.0998; ESI $[M+H]^+$ 281.0.



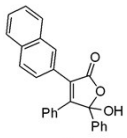
3z: white solid, m.p. 113-114 °C. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.54-7.48 (m, 4H), 7.43-7.38 (m, 3H), 7.31 (d, *J* = 8.4 Hz, 2H), 4.72 (s, 1H), 2.50-2.38 (m, 1H), 2.33-2.22 (m, 1H), 0.91 (t, *J* = 7.6 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 170.8, 165.1, 136.1, 131.3, 130.0, 129.1, 128.3, 127.8, 125.6, 125.3, 122.8, 105.4, 19.1, 11.8. FT-IR: ν (cm^{-1}) 3065, 2982, 2951, 2864, 1706, 1655, 1474. HRMS [ESI] calcd for $C_{18}H_{15}BrO_3Na$ $[M+Na]^+$ 381.0102, found 381.0101; ESI $[M+H]^+$ 359.0.

4. 1D (1H, 13C, 19F) and 2D (HMBC, NOE) NMR spectra

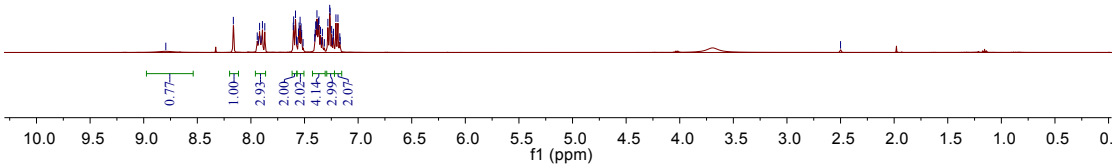

8.693
7.542
7.538
7.522
7.519
7.440
7.436
7.429
7.420
7.415
7.401
7.399
7.393
7.390
7.382
7.376
7.367
7.348
7.344
7.335
7.327
7.256
7.247
7.244
7.235
7.225
7.212
7.201
2.500
Parameter Value
1 Origin Bruker BioSpin GmbH
2 Solvent DMSO
3 Temperature 298.2
4 Number of Scans 4
5 Spectrometer Frequency 400.13
6 Nucleus 1H
O
O
OH
Ph
Ph
3a
1.00
2.01
8.14
1.10
4.05
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)


170.1646
158.6356
137.4864
129.8451
129.5457
129.3426
128.9332
128.8313
128.6414
128.4631
128.4028
128.3012
125.8485
105.6770
40.1216
39.9145
39.7055
39.4969
39.2881
39.0795
38.8716
Parameter Value
1 Origin Bruker BioSpin GmbH
2 Solvent DMSO
3 Temperature 298.3
4 Number of Scans 101
5 Spectrometer Frequency 100.61
6 Nucleus 13C
O
O
OH
Ph
Ph
3a
180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10
f1 (ppm)

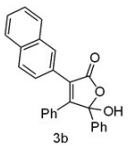
Parameter	Value
1 Origin	Bruker BioSpin GmbH
2 Solvent	DMSO
3 Temperature	298.2
4 Number of Scans	4
5 Spectrometer Frequency	400.13
6 Nucleus	1H



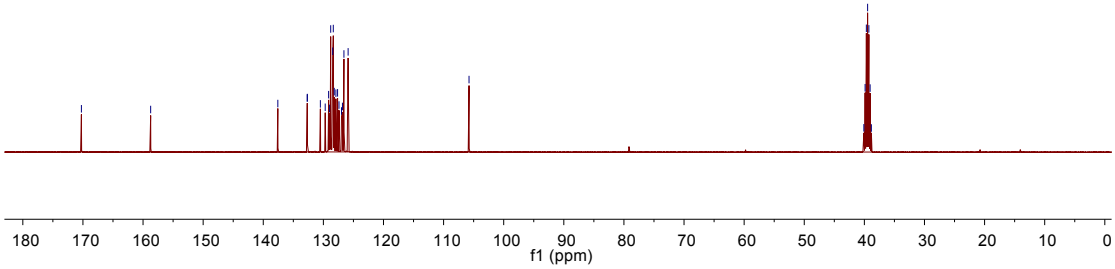
3b



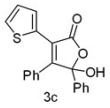
Parameter	Value
1 Origin	Bruker BioSpin GmbH
2 Solvent	DMSO
3 Temperature	298.2
4 Number of Scans	1024
5 Spectrometer Frequency	100.61
6 Nucleus	13C



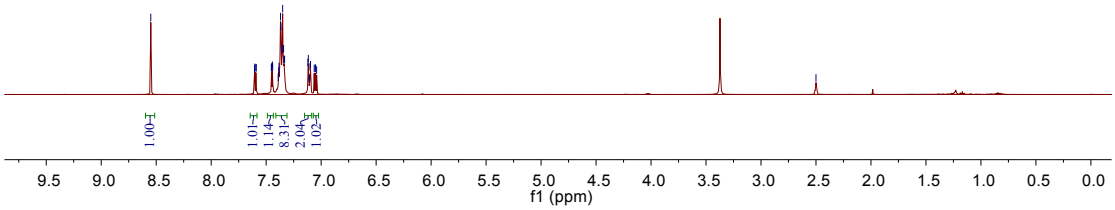
3b



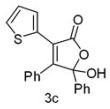
Parameter	Value
1 Origin	Bruker BioSpin GmbH
2 Solvent	DMSO
3 Temperature	298.2
4 Number of Scans	4
5 Spectrometer Frequency	400.13
6 Nucleus	1H



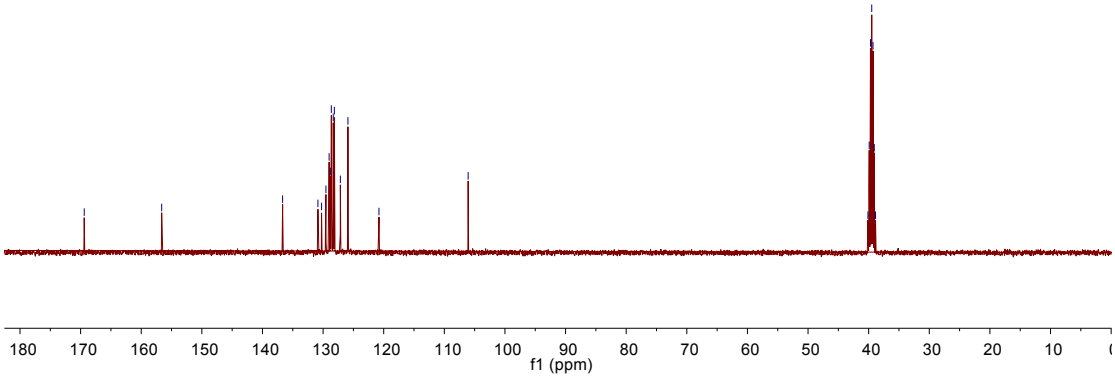
3c

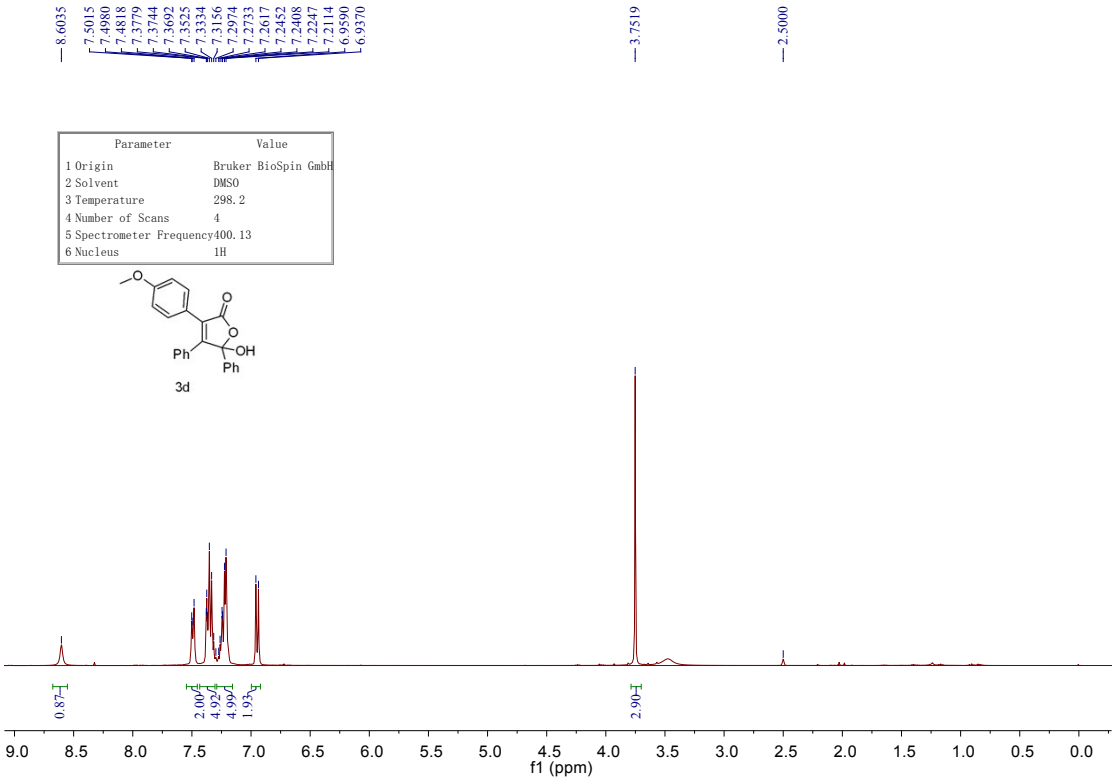


Parameter	Value
1 Origin	Bruker BioSpin GmbH
2 Solvent	DMSO
3 Temperature	298.2
4 Number of Scans	57
5 Spectrometer Frequency	100.61
6 Nucleus	13C



3c

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7.4980
7.4818
7.3779
7.3744
7.3692
7.3525
7.3334
7.3156
7.2974
7.2733
7.2617
7.2452
7.2408
7.2247
7.2114
6.9590
6.9370
3.7519
2.5000
Parameter Value
1 Origin Bruker BioSpin GmbH
2 Solvent DMSO
3 Temperature 298.2
4 Number of Scans 4
5 Spectrometer Frequency 400.13
6 Nucleus 1H
Ph
OH
Ph
3d
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2.00
4.92
4.99
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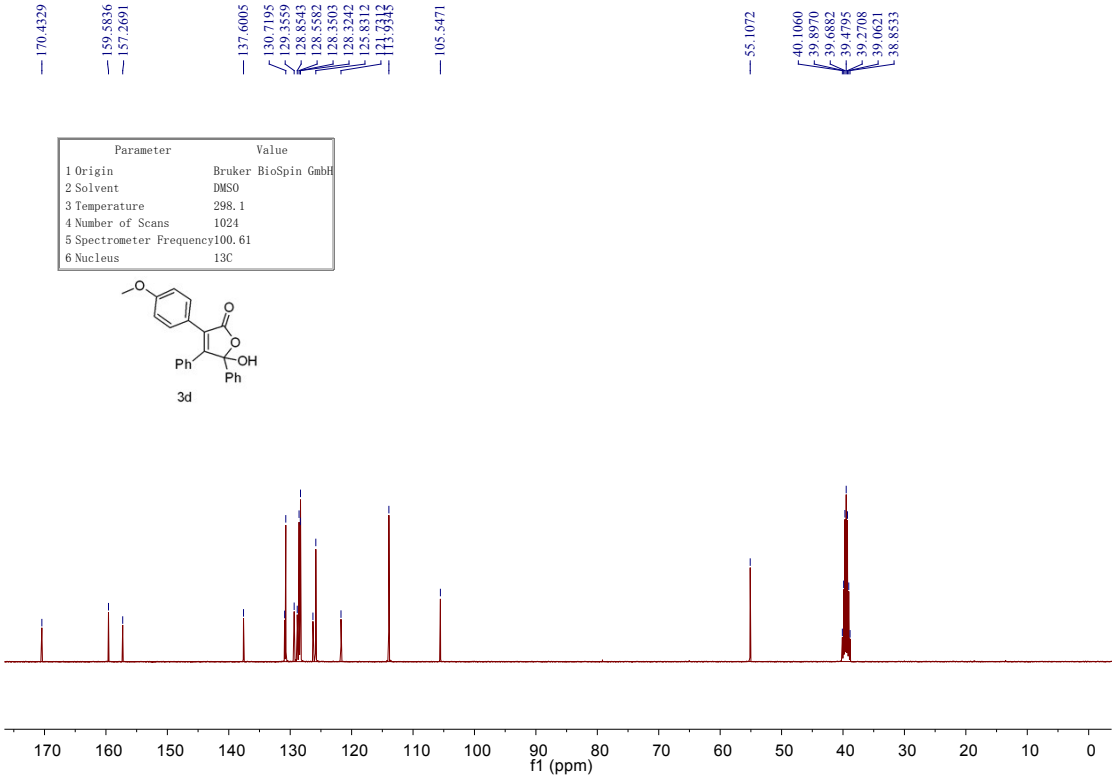
2.90
f1 (ppm)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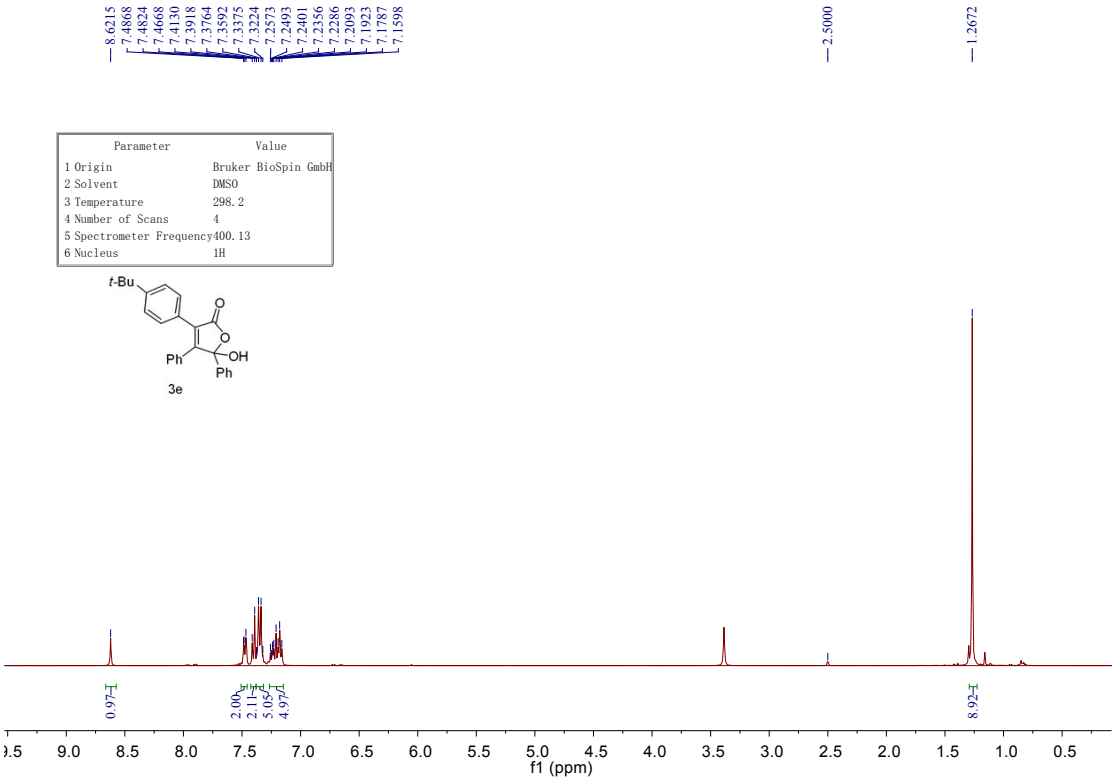
157.2691
137.6005
130.7195
129.3559
128.8543
128.5582
128.3503
128.3242
125.8312
121.7311
113.9345
105.5471
55.1072
40.1060
39.8970
39.6682
39.4795
39.2708
39.0621
38.8533
Parameter Value
1 Origin Bruker BioSpin GmbH
2 Solvent DMSO
3 Temperature 298.1
4 Number of Scans 1024
5 Spectrometer Frequency 100.61
6 Nucleus 13C
Ph
OH
Ph
3d
f1 (ppm)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7.4824
7.4668
7.4130
7.3918
7.3764
7.3592
7.3375
7.3224
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7.2493
7.2401
7.2356
7.2286
7.2093
7.1923
7.1787
7.1598
2.5000
1.2672
Parameter Value
1 Origin Bruker BioSpin GmbH
2 Solvent DMSO
3 Temperature 298.2
4 Number of Scans 4
5 Spectrometer Frequency 400.13
6 Nucleus 1H
t-Bu
Ph
OH
Ph
3e
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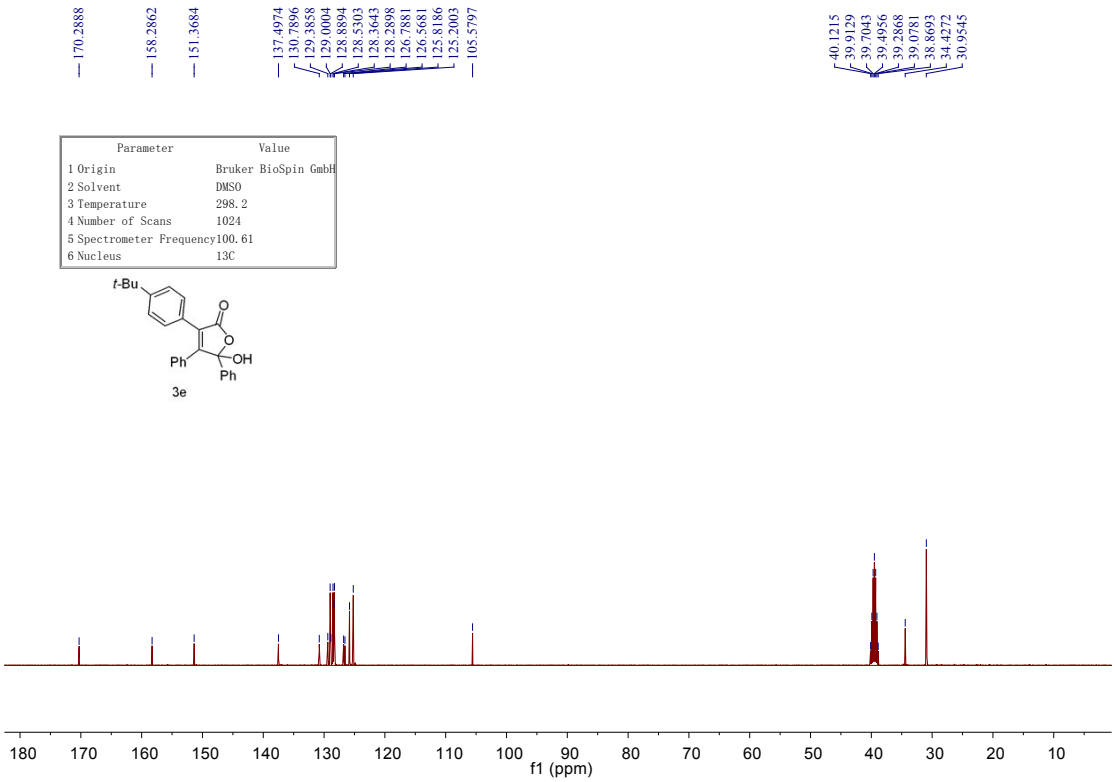
2.00
2.11
5.05
4.97
8.92
f1 (ppm)


170.2888
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151.3684
137.4974
130.7896
129.3858
129.0004
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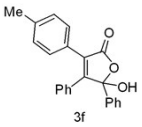
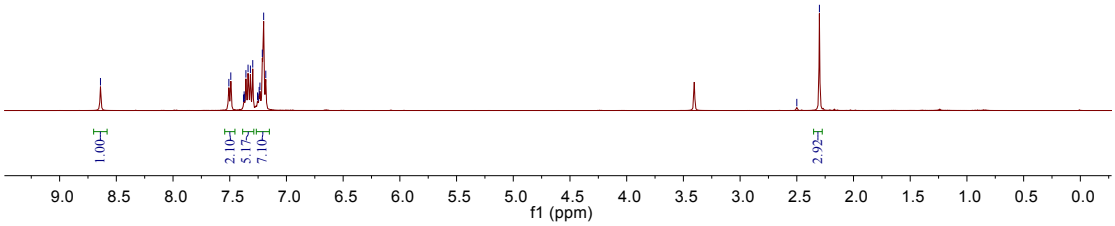
128.5303
128.3643
128.2898
126.7881
126.5681
125.8186
125.2003
105.5797
40.1215
39.9129
39.7043
39.4956
39.2868
39.0781
38.8693
34.4272
30.9545
Parameter Value
1 Origin Bruker BioSpin GmbH
2 Solvent DMSO
3 Temperature 298.2
4 Number of Scans 1024
5 Spectrometer Frequency 100.61
6 Nucleus 13C
t-Bu
Ph
OH
Ph
3e
f1 (ppm)

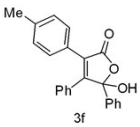
Parameter	Value
1 Origin	Bruker BioSpin GmbH
2 Solvent	DMSO
3 Temperature	298.2
4 Number of Scans	16
5 Spectrometer Frequency	400.13
6 Nucleus	1H



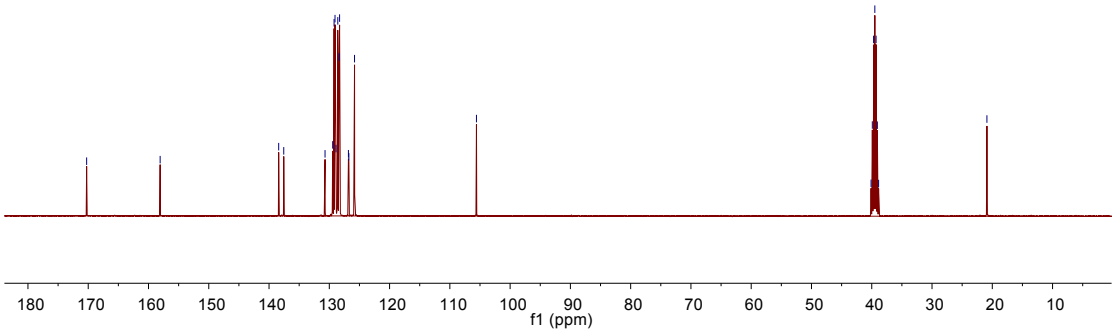
3f



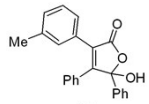
Parameter	Value
1 Origin	Bruker BioSpin GmbH
2 Solvent	DMSO
3 Temperature	298.1
4 Number of Scans	1024
5 Spectrometer Frequency	100.61
6 Nucleus	13C



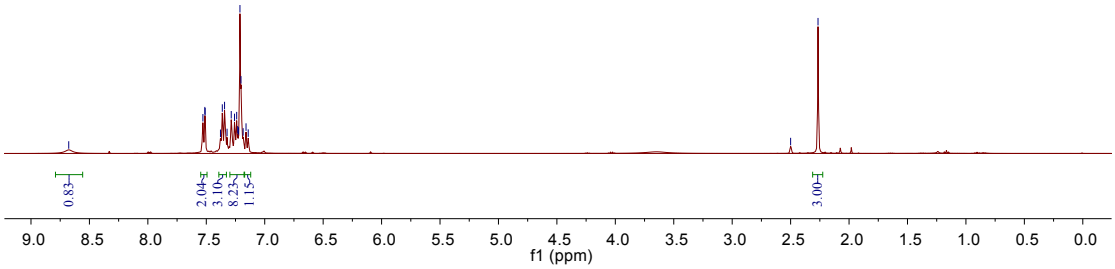
3f



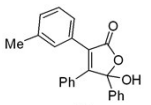
Parameter	Value
1 Origin	Bruker BioSpin GmbH
2 Solvent	DMSO
3 Temperature	298.1
4 Number of Scans	4
5 Spectrometer Frequency	400.13
6 Nucleus	1H



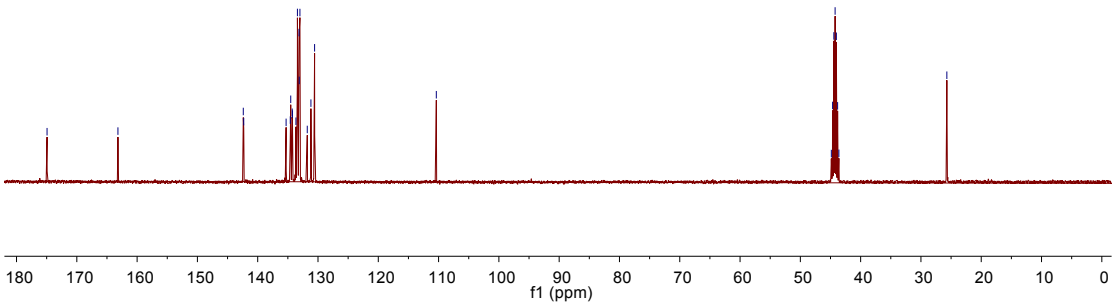
3g

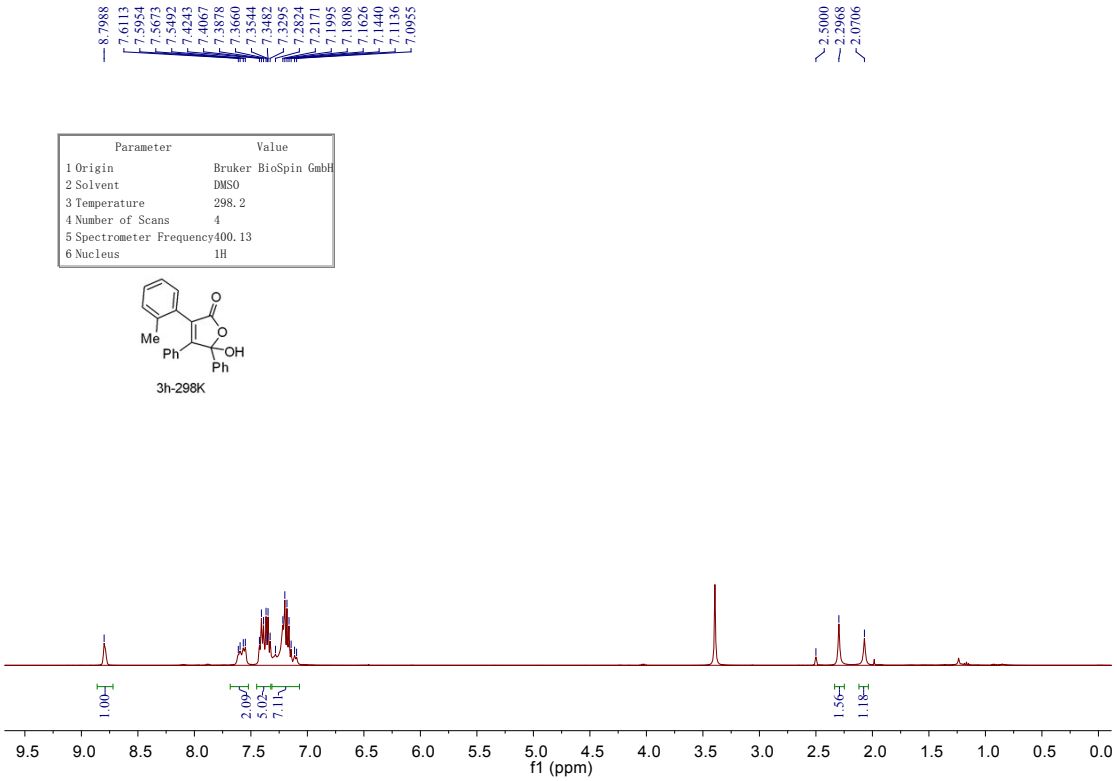


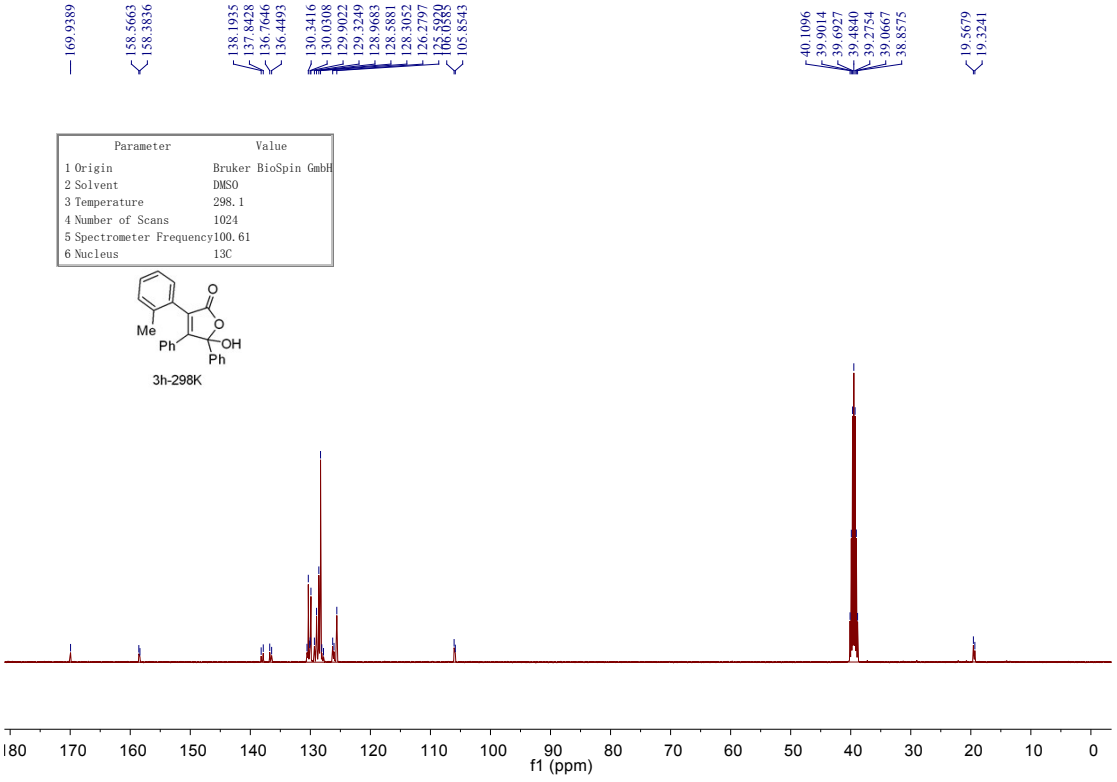
Parameter	Value
1 Origin	Bruker BioSpin GmbH
2 Solvent	DMSO
3 Temperature	298.1
4 Number of Scans	79
5 Spectrometer Frequency	100.61
6 Nucleus	13C



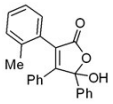
3g



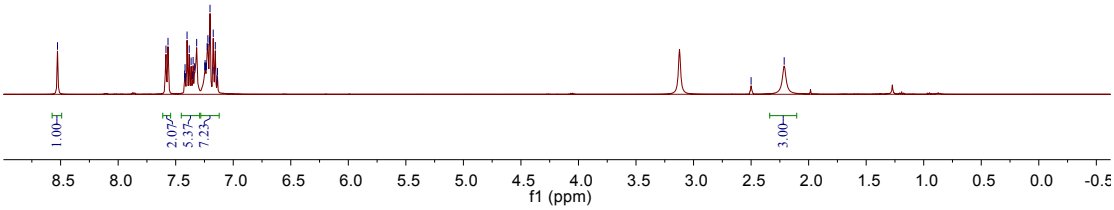

8.7988
7.6113
7.5954
7.5673
7.5492
7.4243
7.4067
7.3878
7.3660
7.3544
7.3482
7.3295
7.2824
7.2171
7.1995
7.1808
7.1626
7.1440
7.1136
7.0955
2.5000
2.2968
2.0706
Parameter Value
1 Origin Bruker BioSpin GmbH
2 Solvent DMSO
3 Temperature 298.2
4 Number of Scans 4
5 Spectrometer Frequency 400.13
6 Nucleus 1H
Me
Ph
O
O
OH
Ph
3h-298K
1.00
2.09
5.02
7.11
1.56
1.18
f1 (ppm)


169.9389
158.5663
158.3836
138.1935
137.8428
136.7646
136.4493
130.3416
130.0308
129.9022
129.3249
128.9683
128.5881
128.3052
126.2797
105.8543
40.1096
39.9014
39.6927
39.4840
39.2754
39.0667
38.8575
19.5679
19.3241
Parameter Value
1 Origin Bruker BioSpin GmbH
2 Solvent DMSO
3 Temperature 298.1
4 Number of Scans 1024
5 Spectrometer Frequency 100.61
6 Nucleus 13C
Me
Ph
O
O
OH
Ph
3h-298K
f1 (ppm)

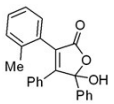
Parameter	Value
1 Origin	Bruker BioSpin GmbH
2 Solvent	DMSO
3 Temperature	353.3
4 Number of Scans	8
5 Spectrometer Frequency	400.13
6 Nucleus	1H



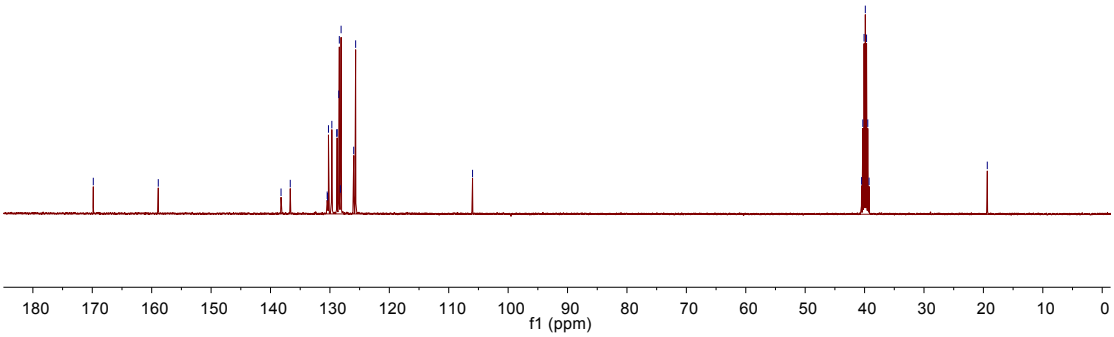
3h-353K

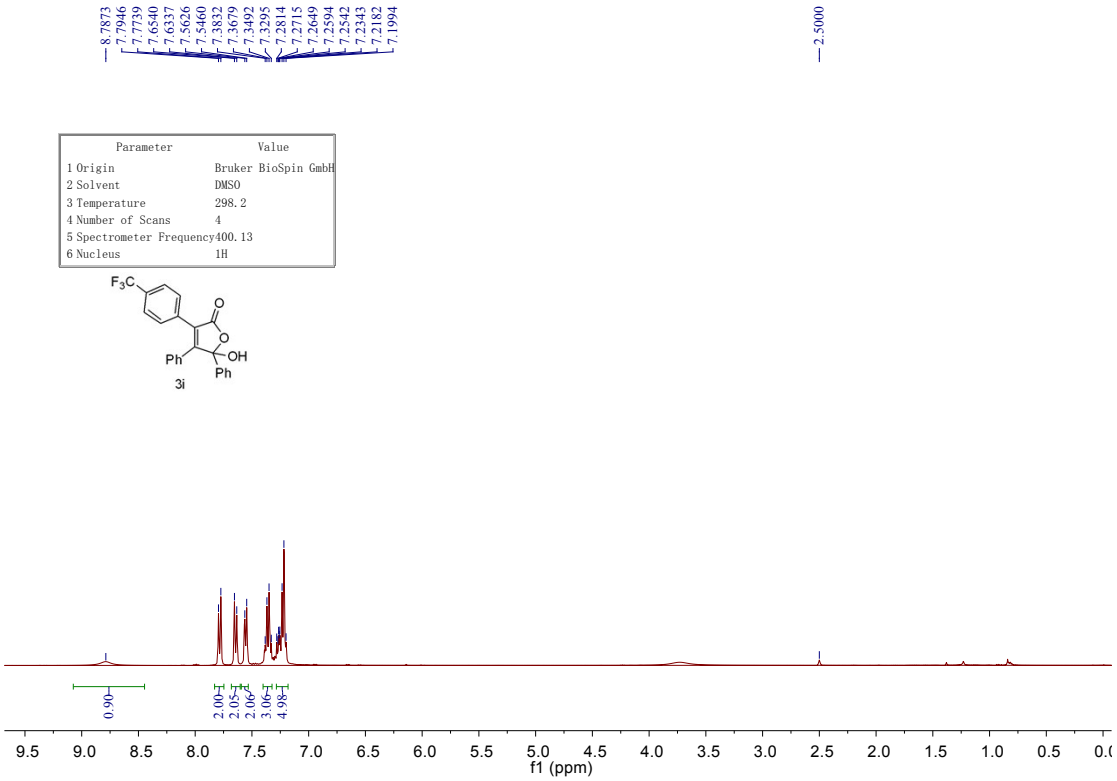


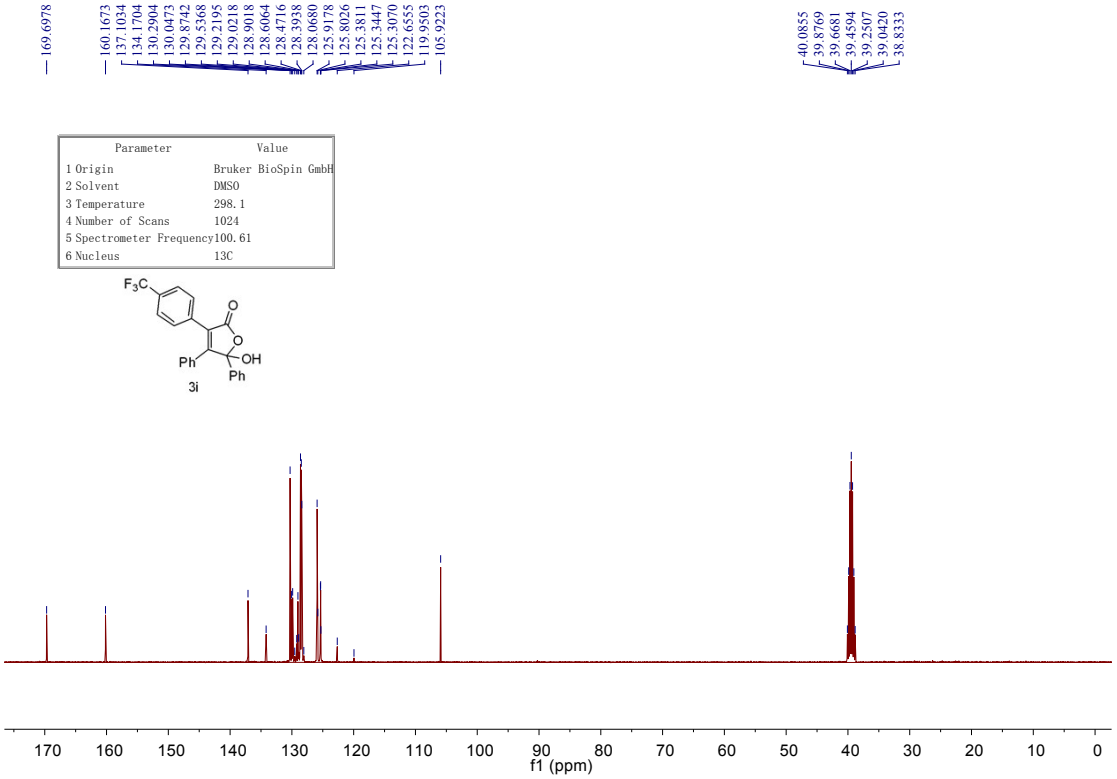
Parameter	Value
1 Origin	Bruker BioSpin GmbH
2 Solvent	DMSO
3 Temperature	353.3
4 Number of Scans	599
5 Spectrometer Frequency	100.61
6 Nucleus	13C

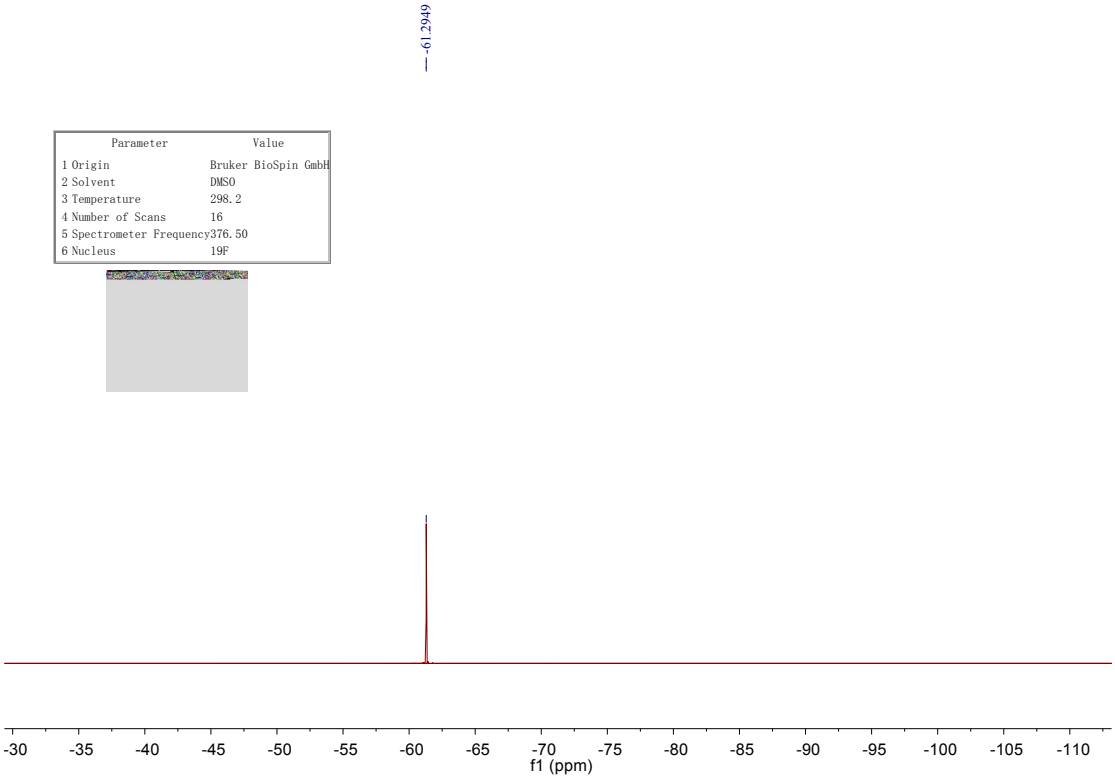


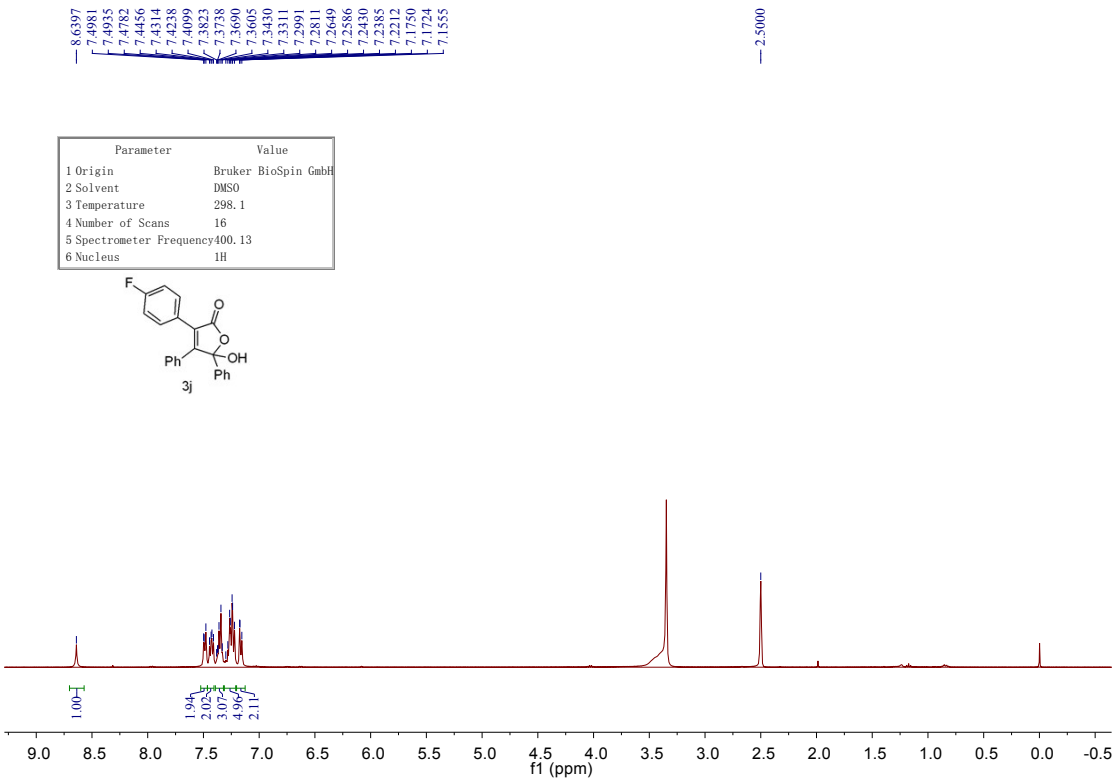
3h-353K

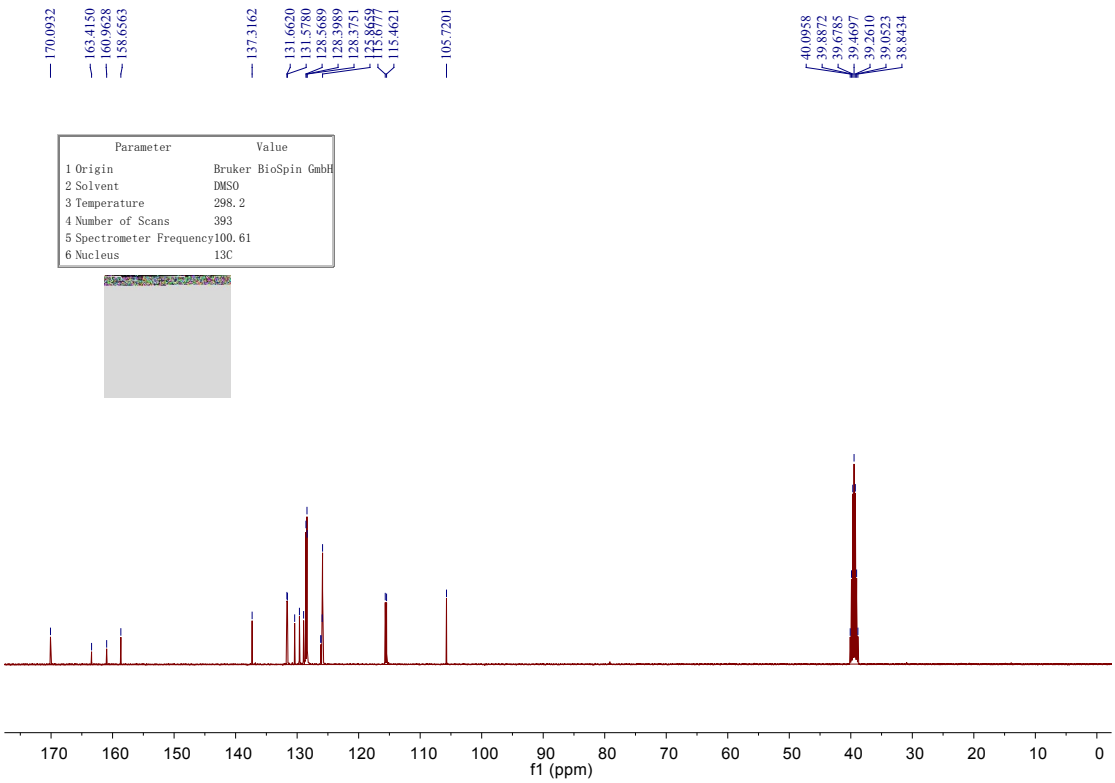


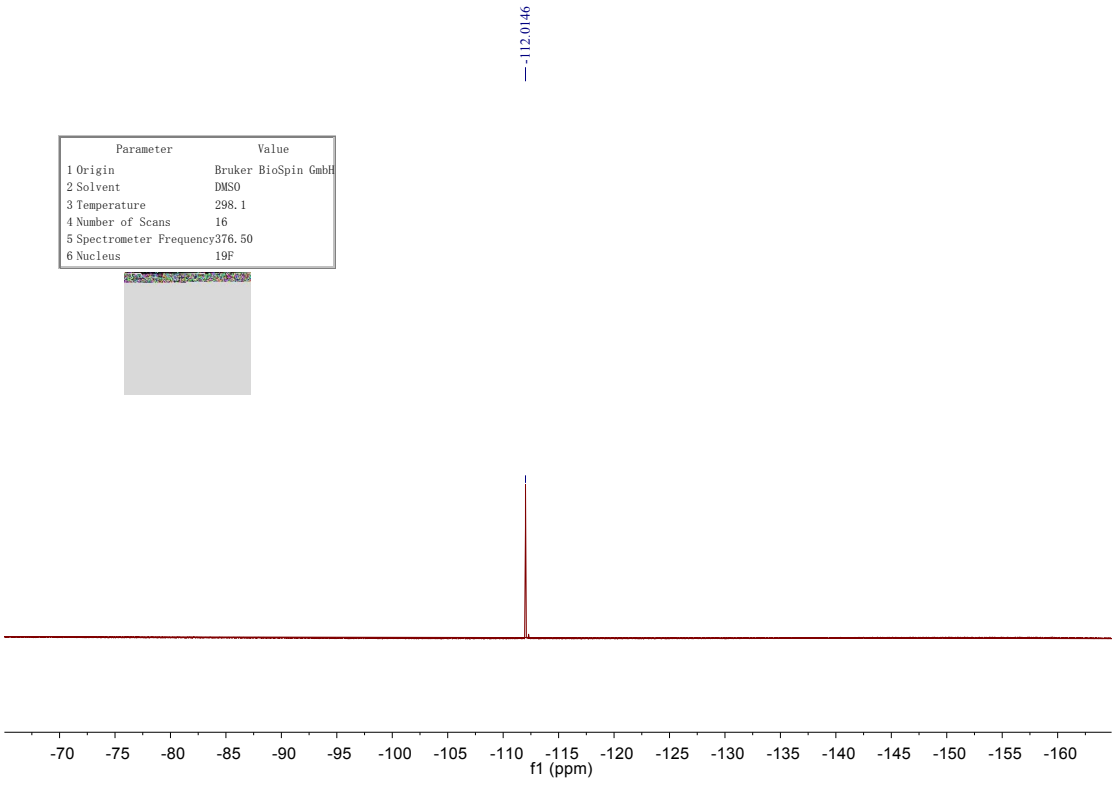

8.7873
7.7946
7.7739
7.6540
7.6337
7.5626
7.5460
7.3832
7.3679
7.3492
7.3295
7.2814
7.2715
7.2649
7.2594
7.2542
7.2343
7.2182
7.1994
2.5000
Parameter Value
1 Origin Bruker BioSpin GmbH
2 Solvent DMSO
3 Temperature 298.2
4 Number of Scans 4
5 Spectrometer Frequency 400.13
6 Nucleus 1H
F3C
O
O
Ph
OH
Ph
3i
0.90
2.00
2.05
2.06
3.06
4.98
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)


169.6978
160.1673
137.1034
134.1704
130.2904
130.0473
129.8742
129.5368
129.2195
129.0218
128.9018
128.6064
128.4716
128.3938
128.0680
125.9178
125.8026
125.3811
125.3447
125.3070
122.6555
119.9503
105.9223
40.0855
39.8769
39.6681
39.4594
39.2507
39.0420
38.8333
Parameter Value
1 Origin Bruker BioSpin GmbH
2 Solvent DMSO
3 Temperature 298.1
4 Number of Scans 1024
5 Spectrometer Frequency 100.61
6 Nucleus 13C
F3C
O
O
Ph
OH
Ph
3i
170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)

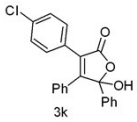

-61.2949
Parameter Value
1 Origin Bruker BioSpin GmbH
2 Solvent DMSO
3 Temperature 298.2
4 Number of Scans 16
5 Spectrometer Frequency 376.50
6 Nucleus 19F
-30 -35 -40 -45 -50 -55 -60 -65 -70 -75 -80 -85 -90 -95 -100 -105 -110
f1 (ppm)


8.6397
7.4981
7.4935
7.4782
7.4456
7.4314
7.4238
7.4099
7.3823
7.3738
7.3690
7.3605
7.3430
7.3311
7.2991
7.2811
7.2649
7.2586
7.2430
7.2385
7.2212
7.1750
7.1724
7.1555
2.5000
Parameter Value
1 Origin Bruker BioSpin GmbH
2 Solvent DMSO
3 Temperature 298.1
4 Number of Scans 16
5 Spectrometer Frequency 400.13
6 Nucleus 1H
F
O
O
Ph
OH
Ph
3j
1.00
1.94
2.02
3.07
4.96
2.11
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)

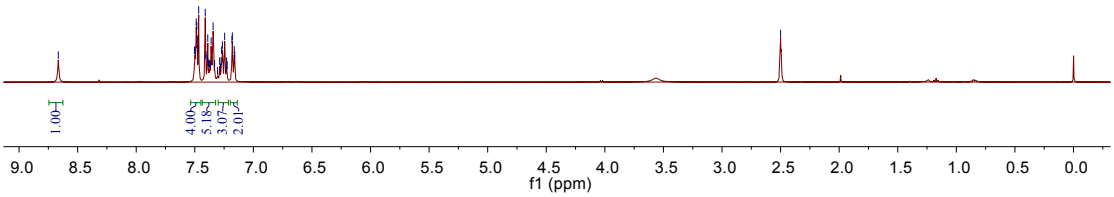

170.0932
163.4150
160.9628
158.6563
137.3162
131.6620
131.5780
128.5689
128.3989
128.3751
115.4621
105.7201
40.0958
39.8872
39.6785
39.4697
39.2610
39.0523
38.8434
Parameter Value
1 Origin Bruker BioSpin GmbH
2 Solvent DMSO
3 Temperature 298.2
4 Number of Scans 393
5 Spectrometer Frequency 100.61
6 Nucleus 13C
170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)


-112.0146
Parameter Value
1 Origin Bruker BioSpin GmbH
2 Solvent DMSO
3 Temperature 298.1
4 Number of Scans 16
5 Spectrometer Frequency 376.50
6 Nucleus 19F
-70 -75 -80 -85 -90 -95 -100 -105 -110 -115 -120 -125 -130 -135 -140 -145 -150 -155 -160
f1 (ppm)

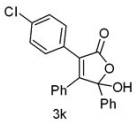
Parameter	Value
1 Origin	Bruker BioSpin GmbH
2 Solvent	DMSO
3 Temperature	298.1
4 Number of Scans	16
5 Spectrometer Frequency	400.13
6 Nucleus	1H



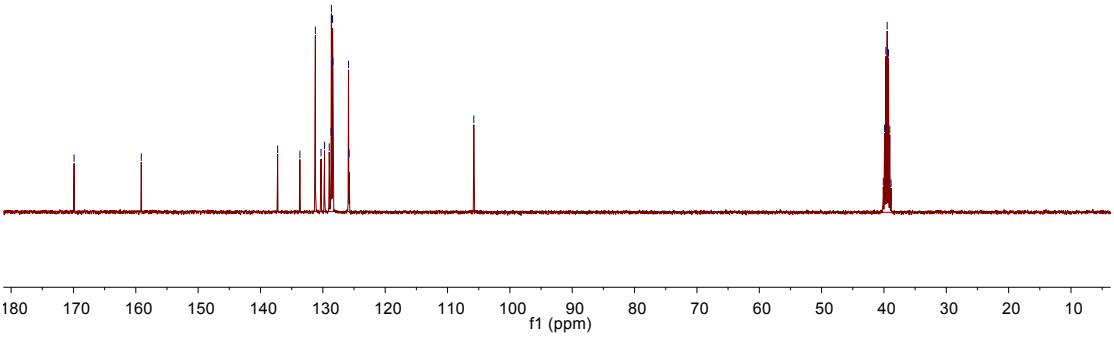
3k



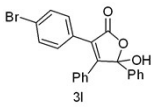
Parameter	Value
1 Origin	Bruker BioSpin GmbH
2 Solvent	DMSO
3 Temperature	298.1
4 Number of Scans	62
5 Spectrometer Frequency	100.61
6 Nucleus	13C



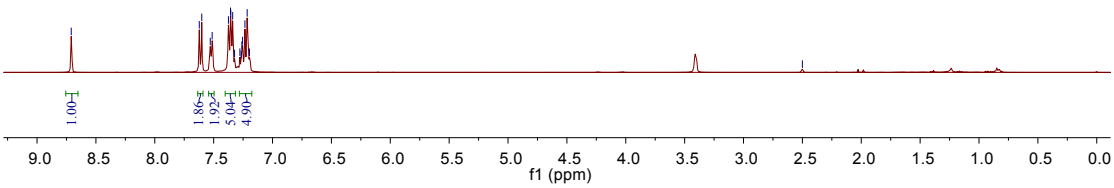
3k



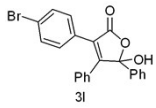
Parameter	Value
1 Origin	Bruker BioSpin GmbH
2 Solvent	DMSO
3 Temperature	298.2
4 Number of Scans	16
5 Spectrometer Frequency	400.13
6 Nucleus	1H



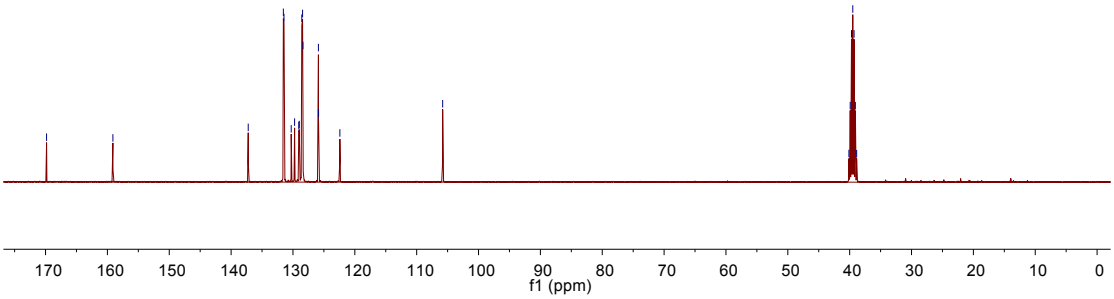
3l



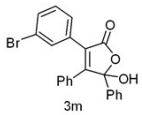
Parameter	Value
1 Origin	Bruker BioSpin GmbH
2 Solvent	DMSO
3 Temperature	298.1
4 Number of Scans	1024
5 Spectrometer Frequency	100.61
6 Nucleus	13C



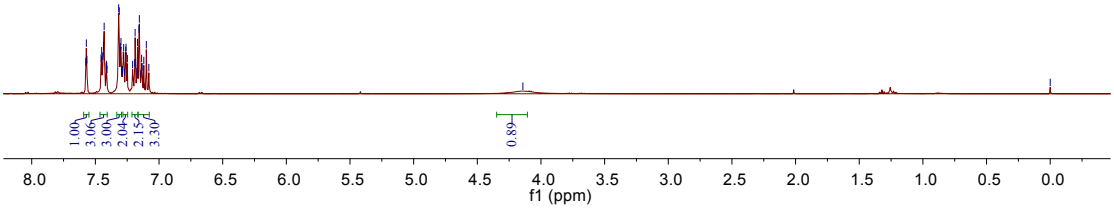
3l



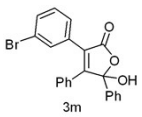
Parameter	Value
1 Origin	Bruker BioSpin GmbH
2 Solvent	CDCl3
3 Temperature	298.2
4 Number of Scans	4
5 Spectrometer Frequency	400.13
6 Nucleus	1H



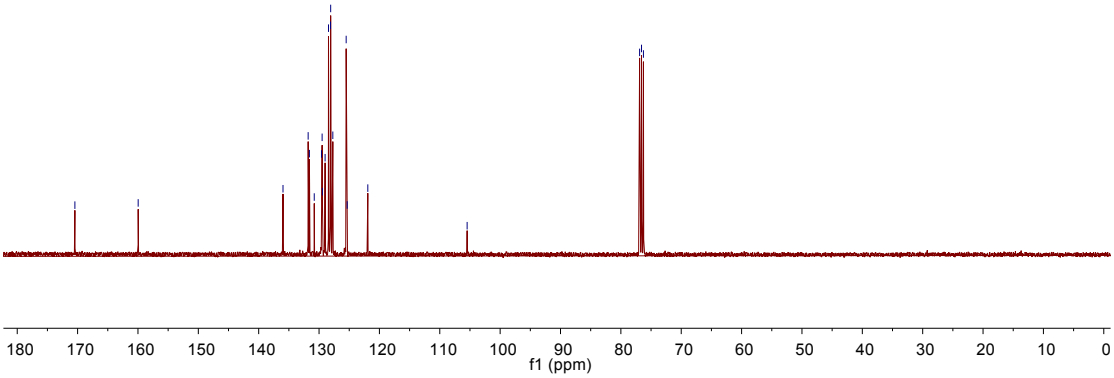
3m

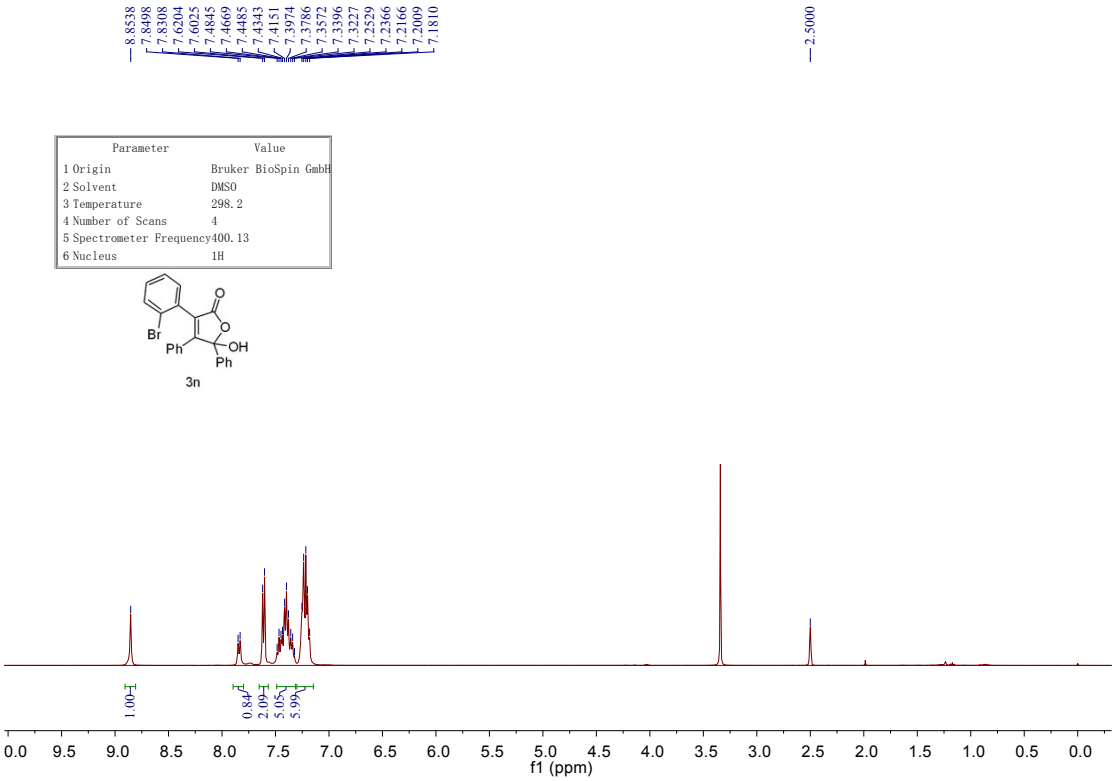


Parameter	Value
1 Origin	Bruker BioSpin GmbH
2 Solvent	CDCl3
3 Temperature	298.2
4 Number of Scans	173
5 Spectrometer Frequency	100.61
6 Nucleus	13C



3m

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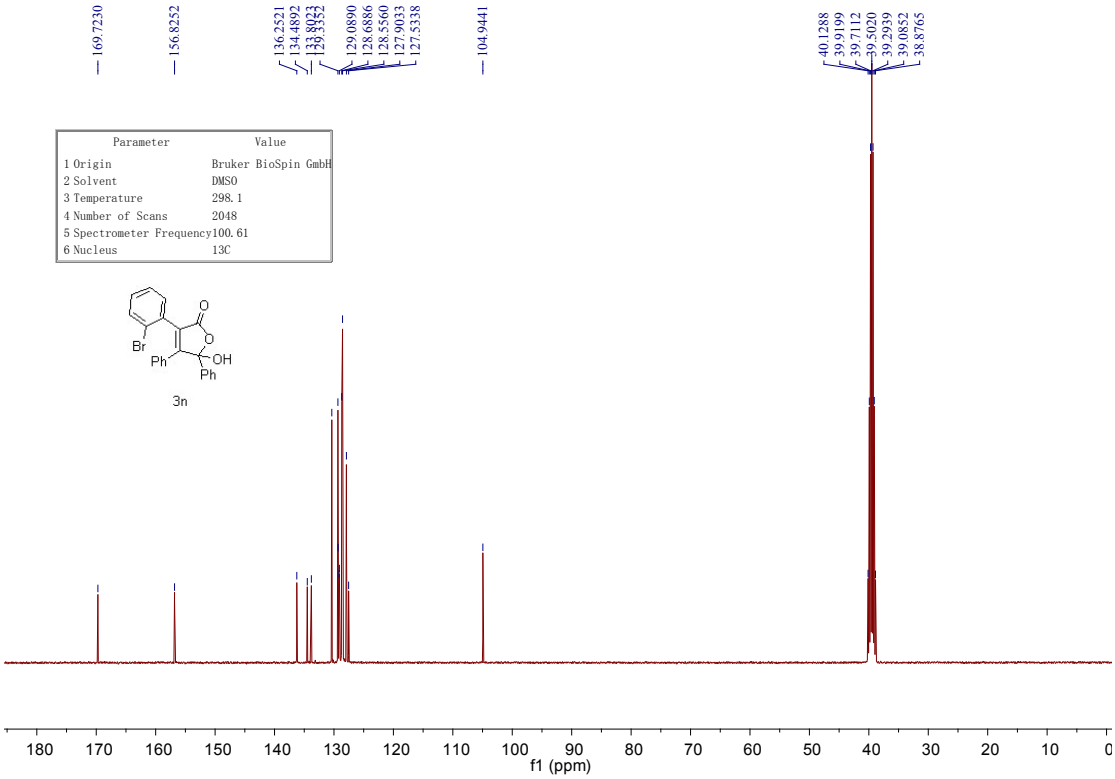
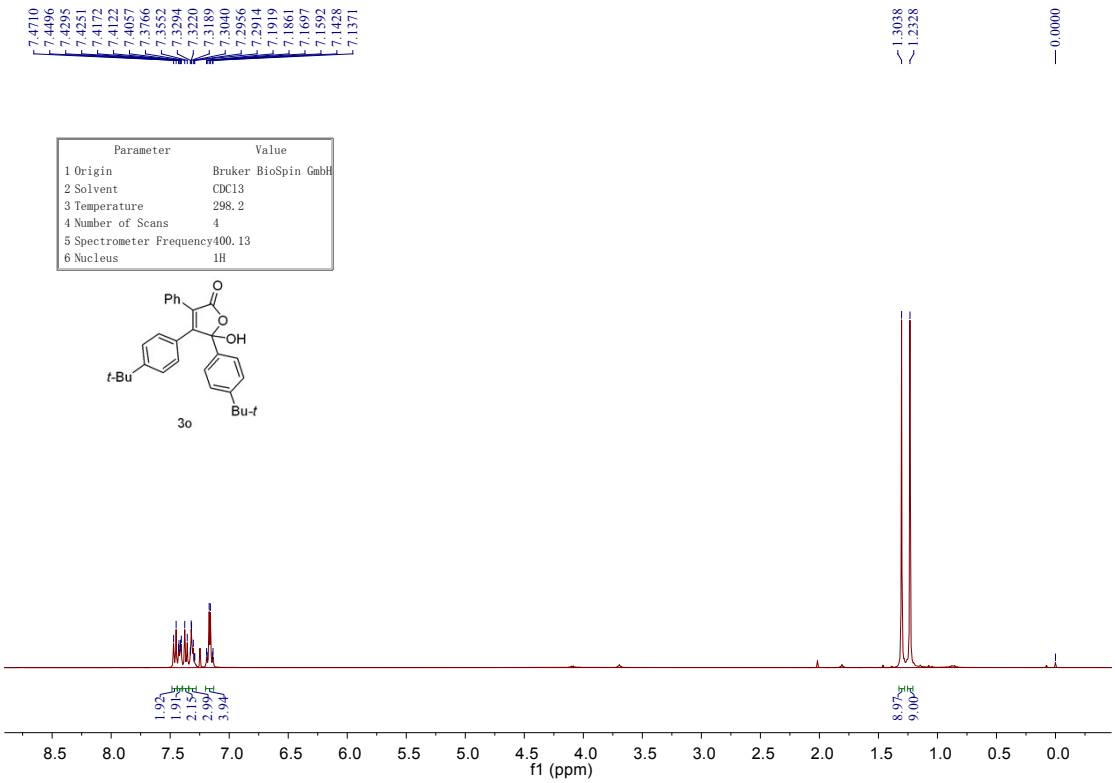
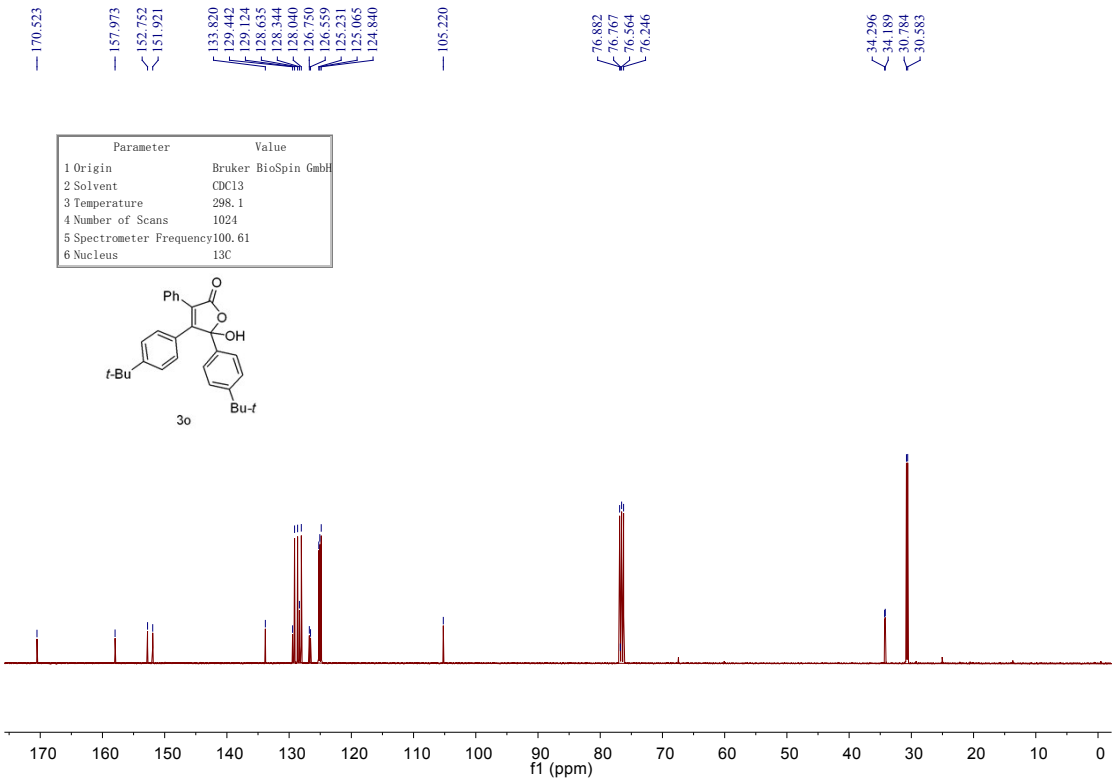
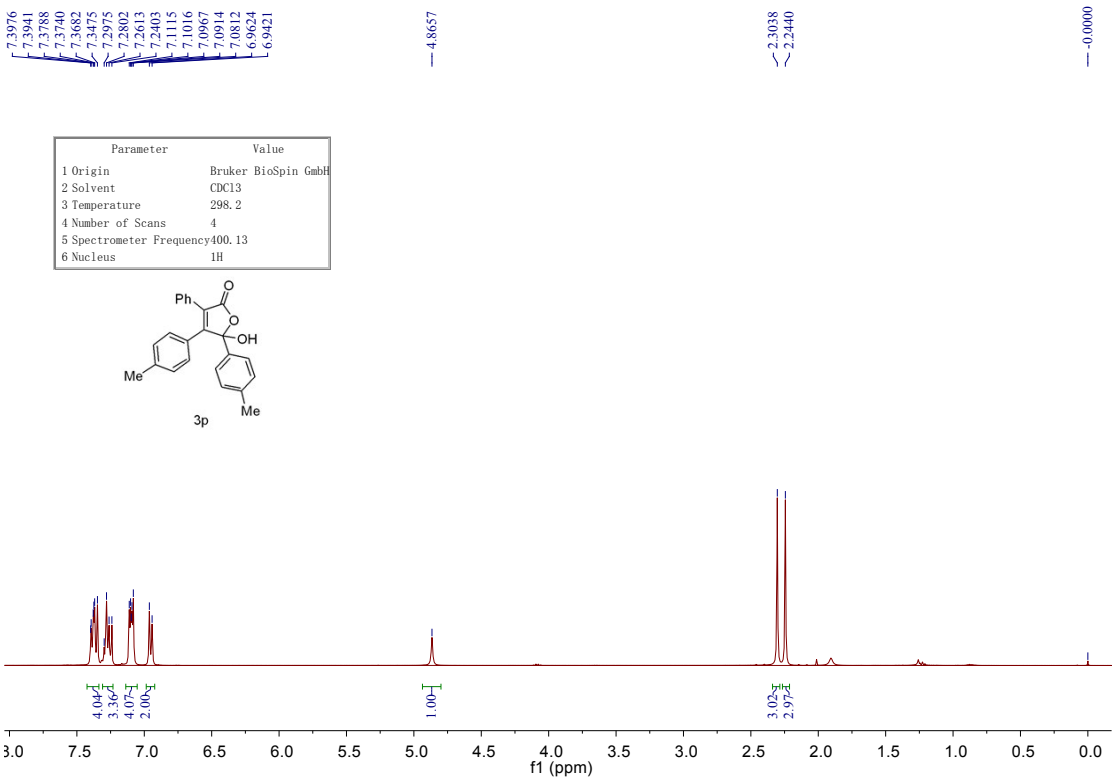
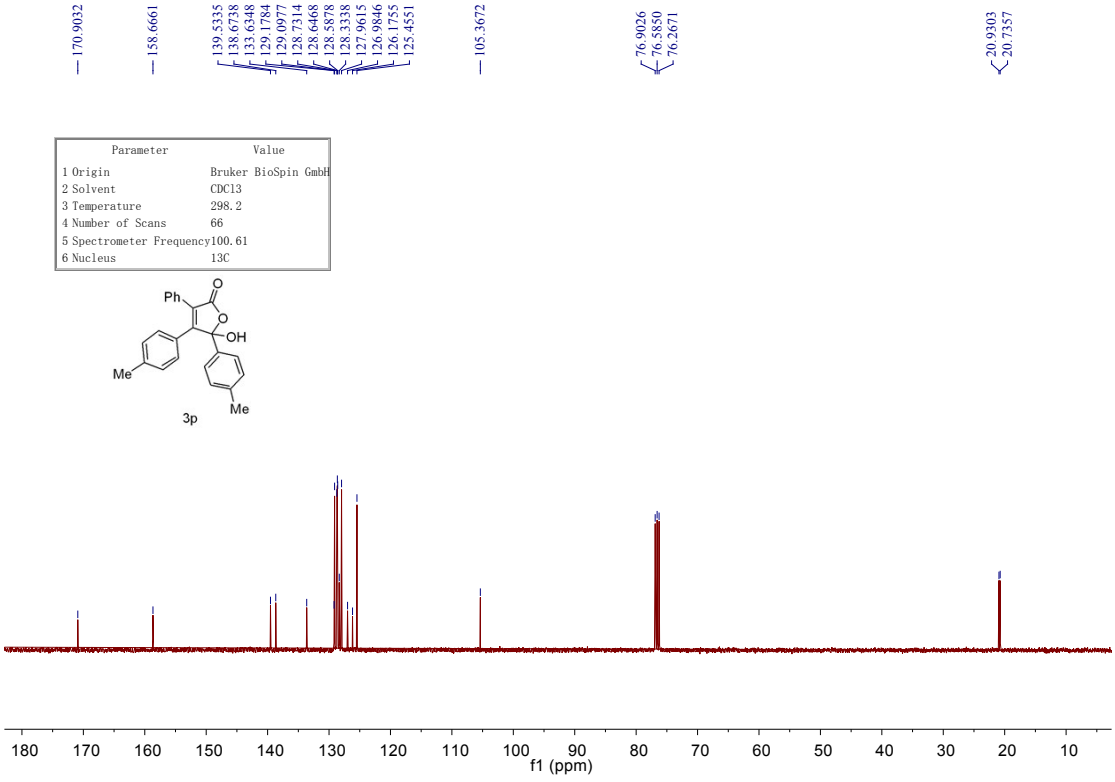
7.8498
7.8308
7.6204
7.6025
7.4845
7.4669
7.4485
7.4343
7.4151
7.3974
7.3786
7.3572
7.3396
7.3227
7.2529
7.2366
7.2166
7.2009
7.1810
2.5000
Parameter Value
1 Origin Bruker BioSpin GmbH
2 Solvent DMSO
3 Temperature 298.2
4 Number of Scans 4
5 Spectrometer Frequency 400.13
6 Nucleus 1H
Br
Ph
O
OH
Ph
3n
1.00
0.84
2.09
5.05
5.99
f1 (ppm)


169.7230
156.8252
136.2521
134.4892
131.9353
129.0890
128.6886
128.5560
127.9033
127.5338
104.9441
40.1288
39.9199
39.7112
39.5020
39.2939
39.0852
38.8765
Parameter Value
1 Origin Bruker BioSpin GmbH
2 Solvent DMSO
3 Temperature 298.1
4 Number of Scans 2048
5 Spectrometer Frequency 100.61
6 Nucleus 13C
Br
Ph
O
OH
Ph
3n
f1 (ppm)


7.4710
7.4496
7.4295
7.4251
7.4172
7.4122
7.4057
7.3766
7.3552
7.3294
7.3220
7.3189
7.3040
7.2956
7.2914
7.1919
7.1861
7.1697
7.1592
7.1428
7.1371
1.3038
1.2328
0.0000
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
Ph
O
O
OH
t-Bu
Bu-t
3o
1.92
1.91
2.15
2.99
3.94
8.97
9.00
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)


170.523
157.973
152.752
151.921
133.820
129.442
129.124
128.635
128.344
128.040
126.750
126.559
125.231
125.065
124.840
105.220
76.882
76.767
76.564
76.246
34.296
34.189
30.784
30.583
Parameter Value
1 Origin Bruker BioSpin GmbH
2 Solvent CDCl3
3 Temperature 298.1
4 Number of Scans 1024
5 Spectrometer Frequency 100.61
6 Nucleus 13C
Ph
O
O
OH
t-Bu
Bu-t
3o
170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)


7.3976
7.3941
7.3788
7.3740
7.3682
7.3475
7.2975
7.2802
7.2613
7.2403
7.1115
7.1016
7.0967
7.0914
7.0812
6.9624
6.9421
4.8657
2.3038
2.2440
-0.0000
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
Ph
O
O
OH
Me
Me
3p
4.04
3.36
4.07
2.00
1.00
3.02
2.97
f1 (ppm)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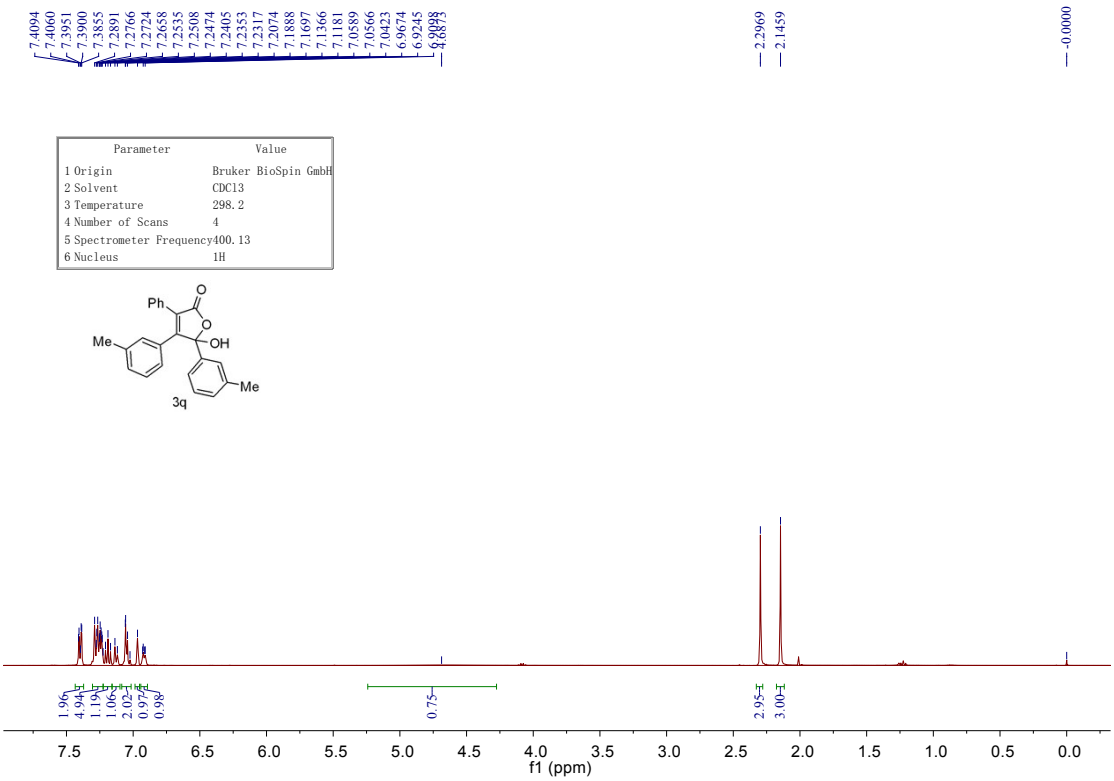
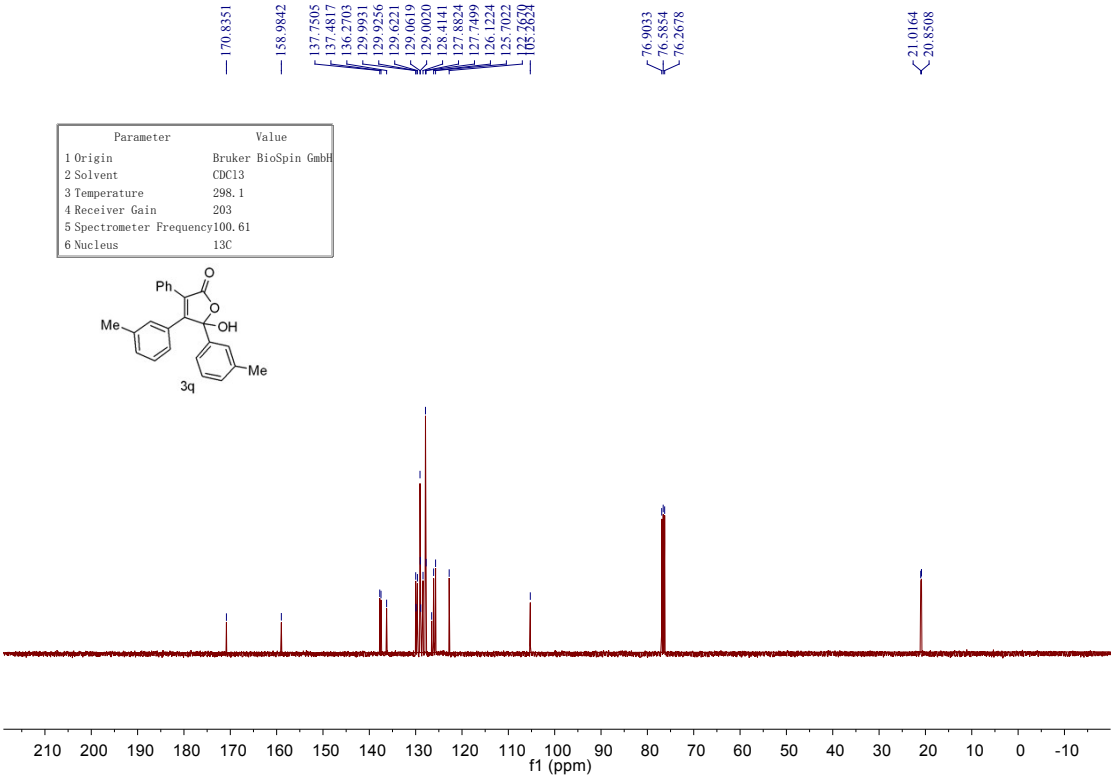
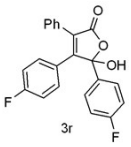
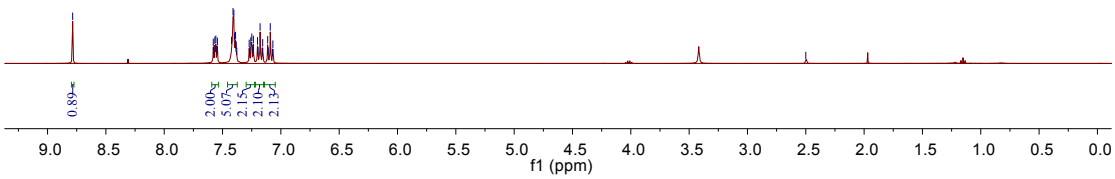
139.5335
138.6738
133.6348
129.1784
129.0977
128.7314
128.6468
128.5878
128.3338
127.9615
126.9846
126.1755
125.4551
105.3672
76.9026
76.5850
76.2671
20.9303
20.7357
Parameter Value
1 Origin Bruker BioSpin GmbH
2 Solvent CDCl3
3 Temperature 298.2
4 Number of Scans 66
5 Spectrometer Frequency 100.61
6 Nucleus 13C
Ph
O
O
OH
Me
Me
3p
f1 (ppm)


7.4094
7.4060
7.3951
7.3900
7.3855
7.2891
7.2766
7.2724
7.2658
7.2535
7.2508
7.2474
7.2405
7.2353
7.2317
7.2074
7.1888
7.1697
7.1366
7.1181
7.0589
7.0566
7.0423
6.9674
6.9245
6.9098
6.8873
2.2969
2.1459
-0.0000
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
Ph
O
O
OH
Me
Me
3q
1.96
4.94
1.19
1.06
2.02
0.97
0.98
0.75
2.95
3.00
f1 (ppm)

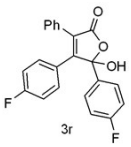

170.8351
158.9842
137.7505
137.4817
136.2703
129.9931
129.9256
129.6221
129.0619
129.0020
128.4141
127.8824
127.7499
126.1224
125.7022
105.2624
76.9033
76.5854
76.2678
21.0164
20.8508
Parameter Value
1 Origin Bruker BioSpin GmbH
2 Solvent CDCl3
3 Temperature 298.1
4 Receiver Gain 203
5 Spectrometer Frequency 100.61
6 Nucleus 13C
Ph
O
O
OH
Me
Me
3q
f1 (ppm)

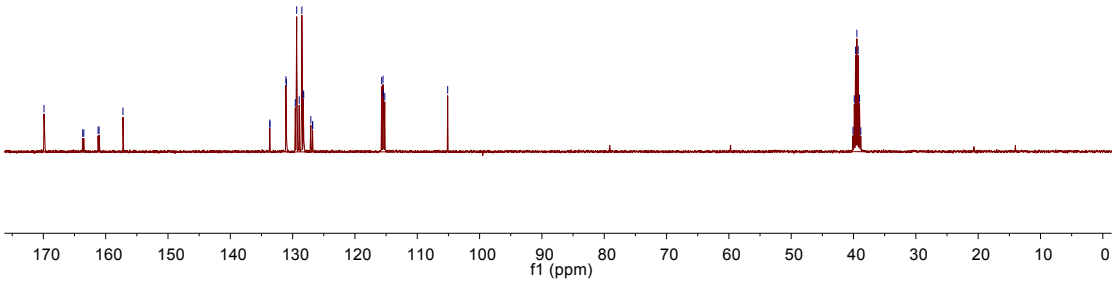
Parameter	Value
1 Origin	Bruker BioSpin GmbH
2 Solvent	DMSO
3 Temperature	298.2
4 Number of Scans	4
5 Spectrometer Frequency	400.13
6 Nucleus	1H

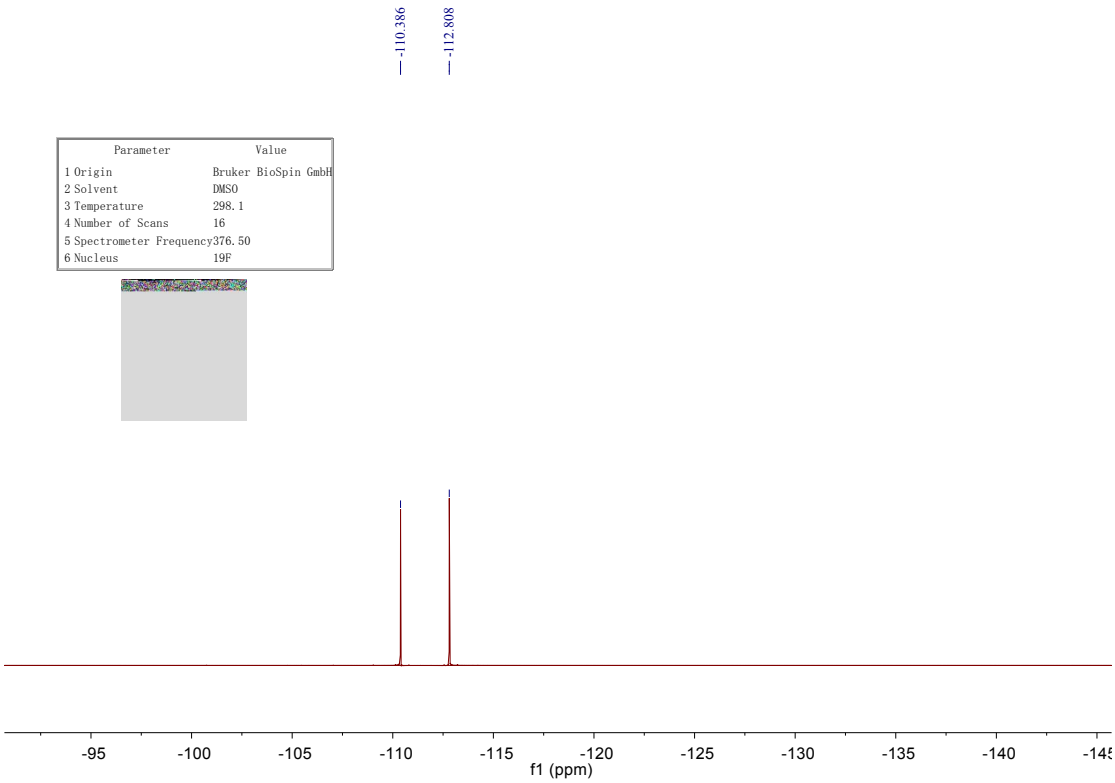


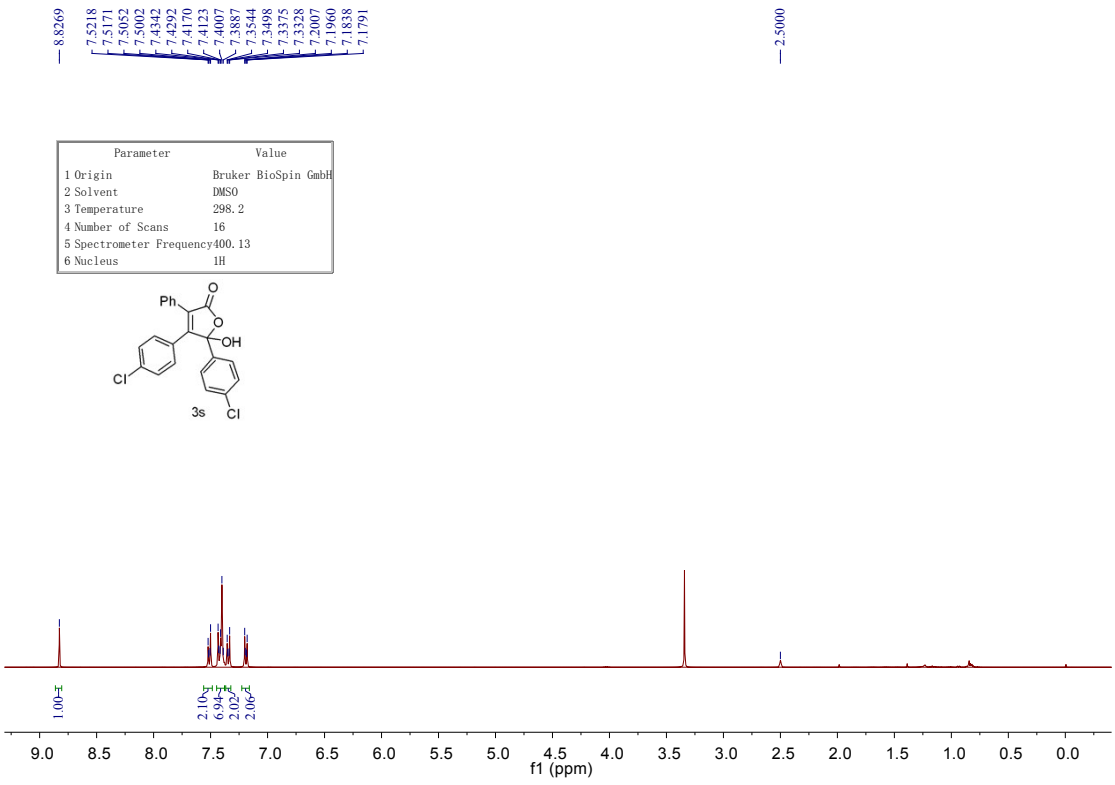


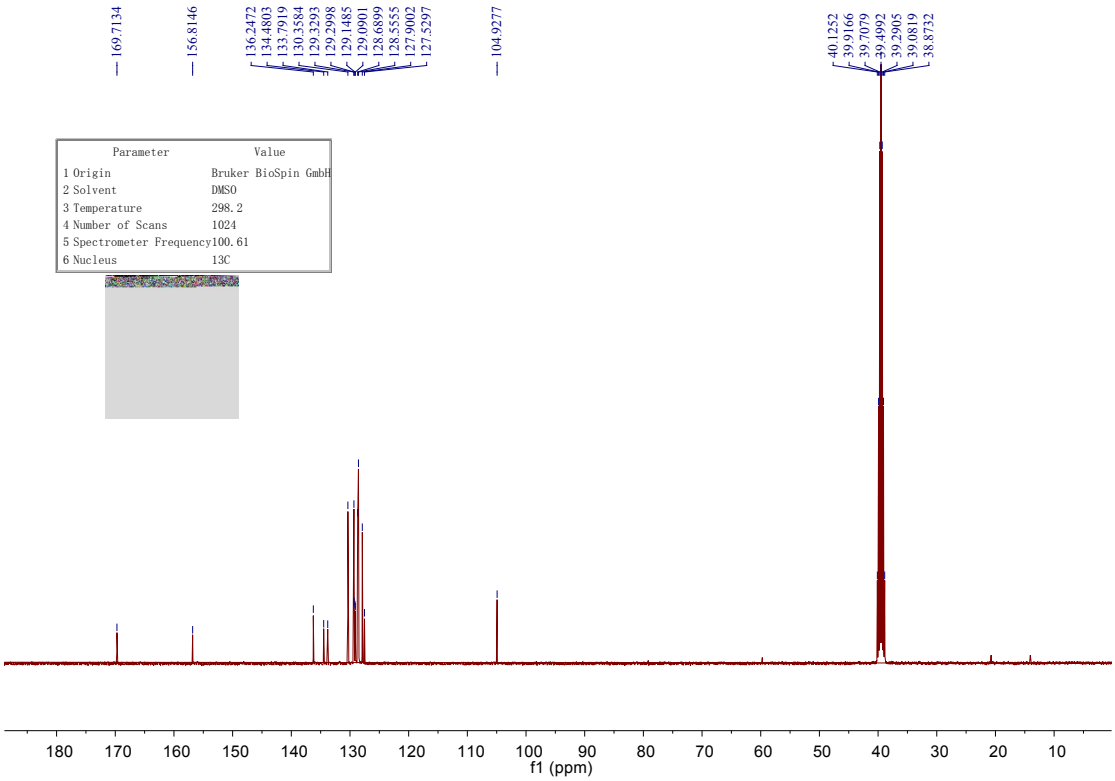
Parameter	Value
1 Origin	Bruker BioSpin GmbH
2 Solvent	DMSO
3 Temperature	298.2
4 Number of Scans	59
5 Spectrometer Frequency	100.61
6 Nucleus	13C

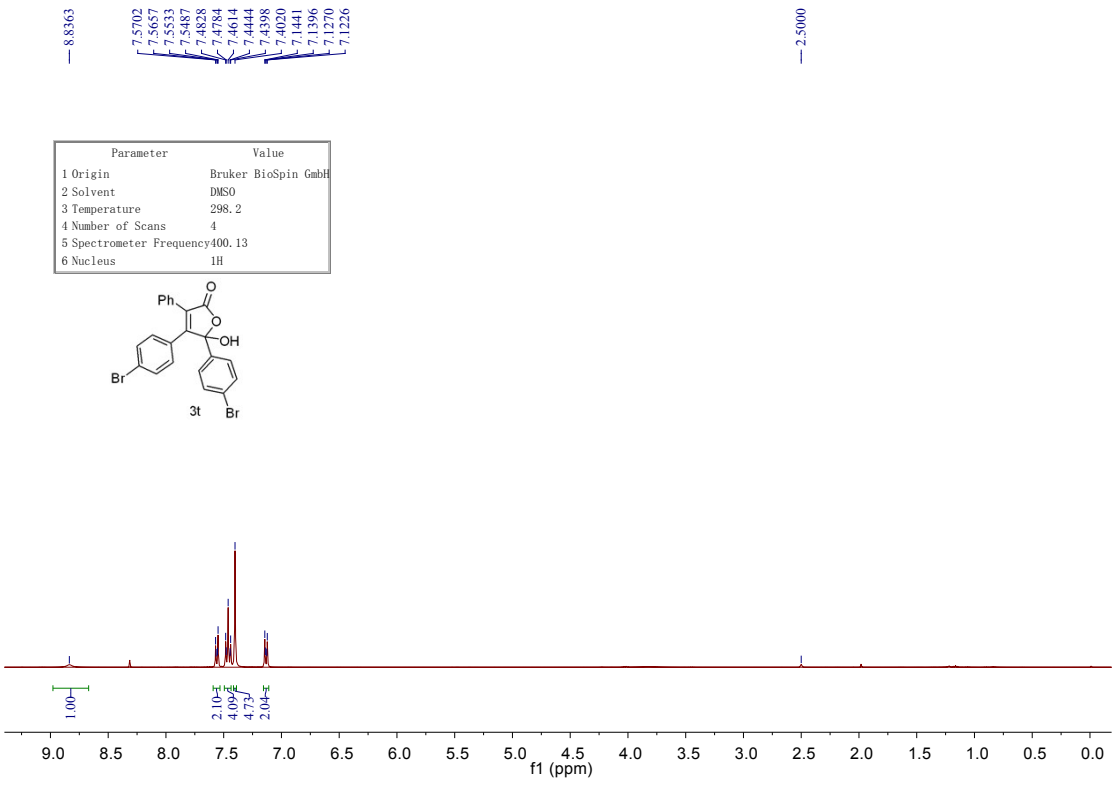


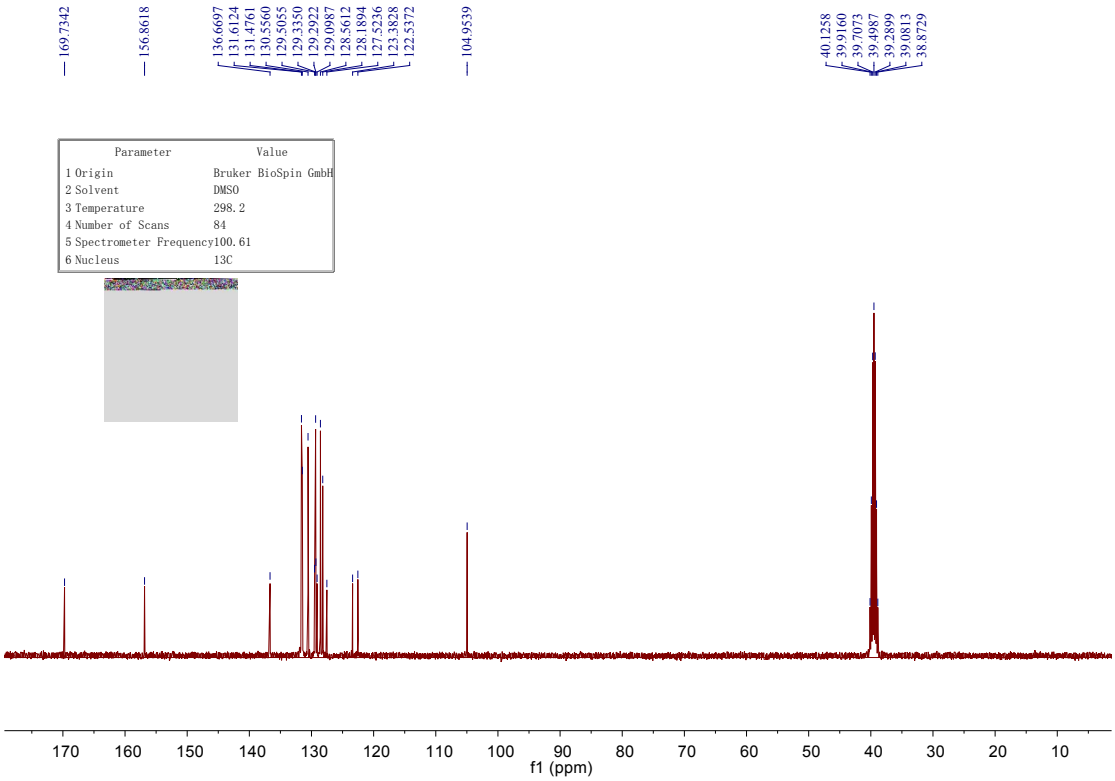


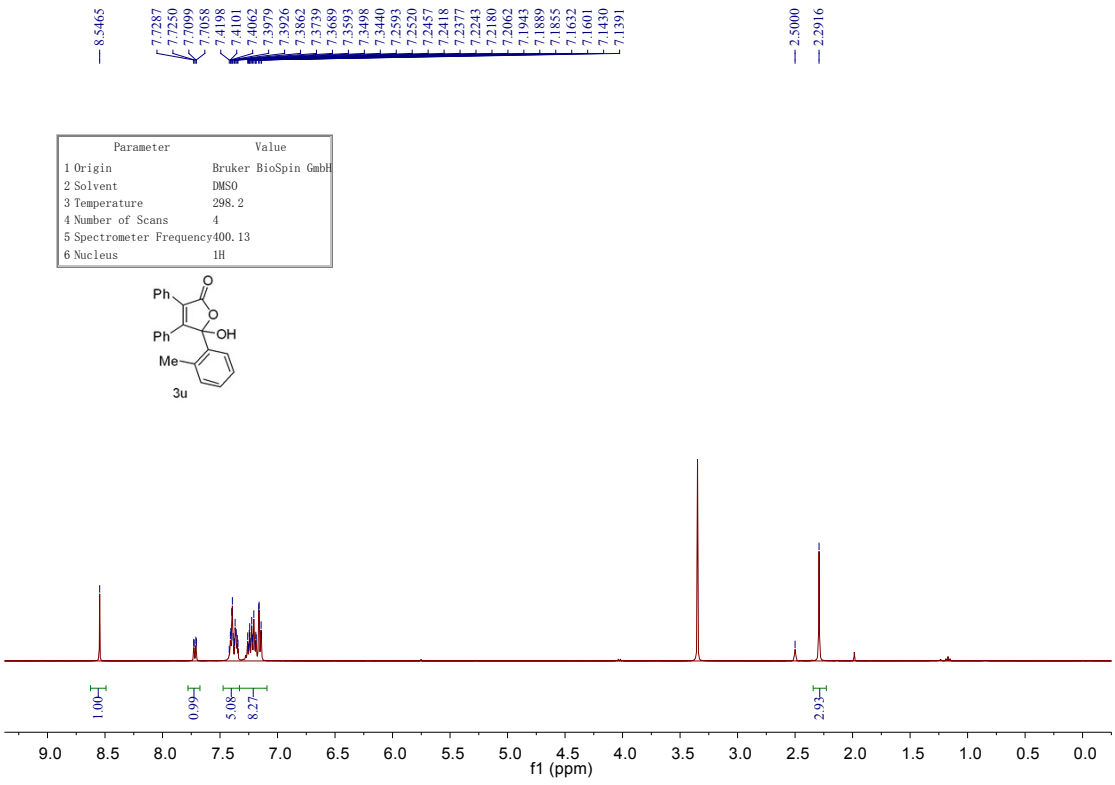

-110.386
-112.808
Parameter Value
1 Origin Bruker BioSpin GmbH
2 Solvent DMSO
3 Temperature 298.1
4 Number of Scans 16
5 Spectrometer Frequency 376.50
6 Nucleus 19F
-95 -100 -105 -110 -115 -120 -125 -130 -135 -140 -145
f1 (ppm)

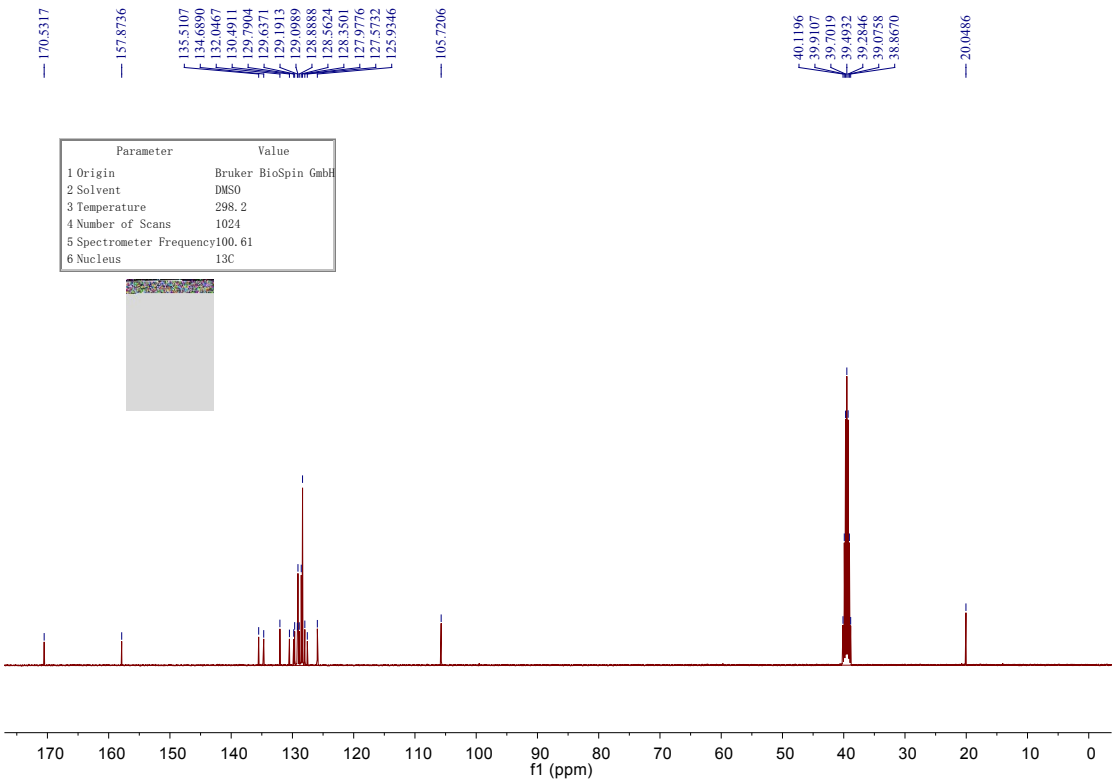

8.8269
7.5218
7.5171
7.5052
7.5002
7.4342
7.4292
7.4170
7.4123
7.4007
7.3887
7.3544
7.3498
7.3375
7.3328
7.2007
7.1960
7.1838
7.1791
2.5000
Parameter Value
1 Origin Bruker BioSpin GmbH
2 Solvent DMSO
3 Temperature 298.2
4 Number of Scans 16
5 Spectrometer Frequency 400.13
6 Nucleus 1H
Ph
O
O
OH
Cl
3s
Cl
1.00
2.10
6.94
2.02
2.06
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)

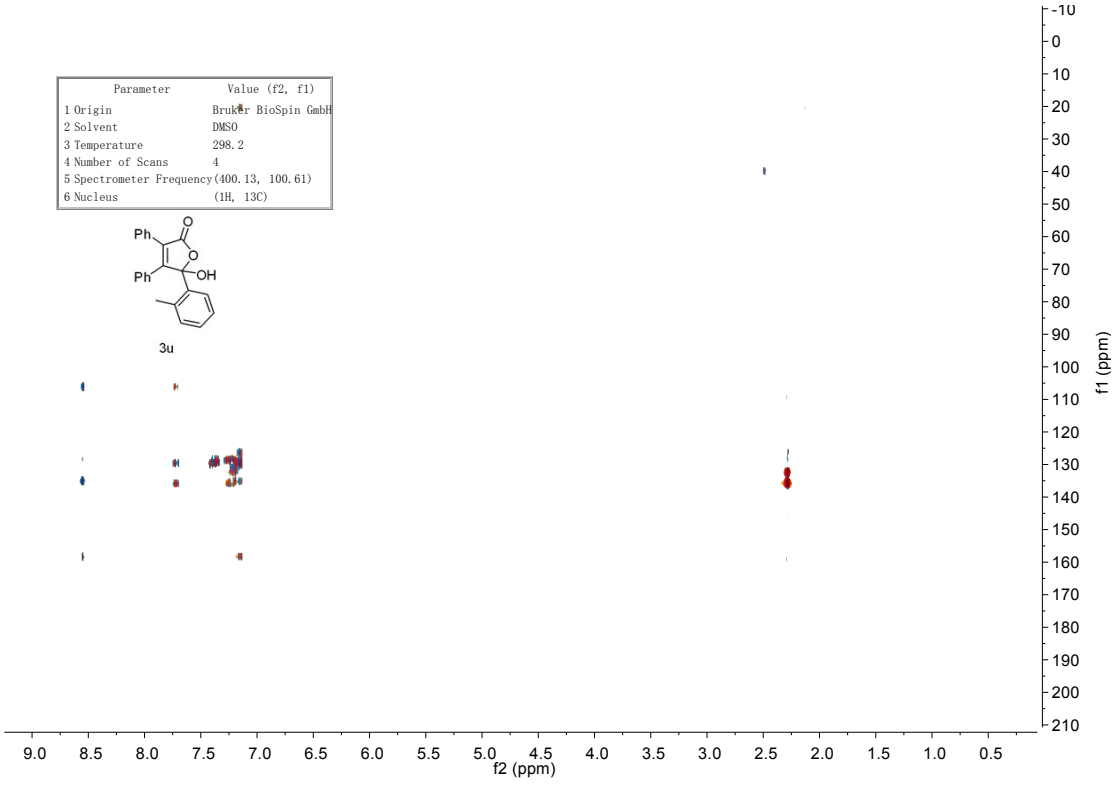

169.7134
156.8146
136.2472
134.4803
133.7919
130.3584
129.3293
129.2998
129.1485
129.0901
128.6899
128.5555
127.9002
127.5297
104.9277
40.1252
39.9166
39.7079
39.4992
39.2905
39.0819
38.8732
Parameter Value
1 Origin Bruker BioSpin GmbH
2 Solvent DMSO
3 Temperature 298.2
4 Number of Scans 1024
5 Spectrometer Frequency 100.61
6 Nucleus 13C
f1 (ppm)

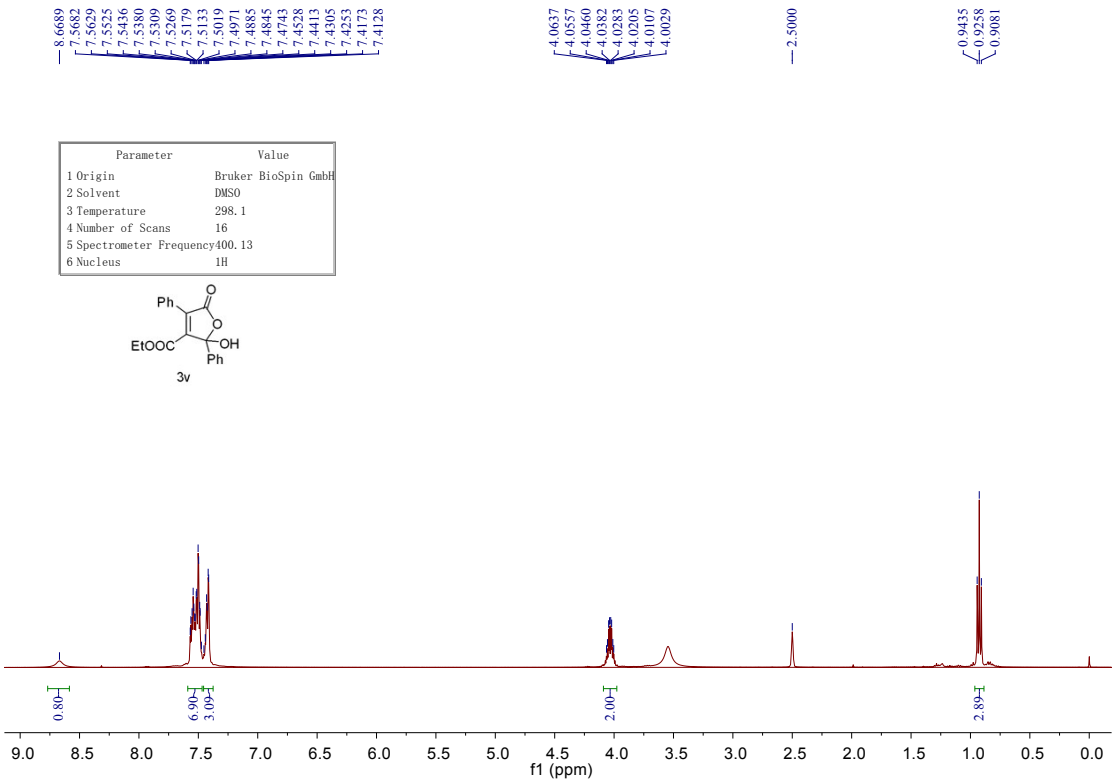

8.8363
7.5702
7.5657
7.5533
7.5487
7.4828
7.4784
7.4614
7.4444
7.4398
7.4020
7.1441
7.1396
7.1270
7.1226
2.5000
Parameter Value
1 Origin Bruker BioSpin GmbH
2 Solvent DMSO
3 Temperature 298.2
4 Number of Scans 4
5 Spectrometer Frequency 400.13
6 Nucleus 1H
Ph
O
O
OH
Br
3t
Br
1.00
2.10
4.09
4.73
2.04
f1 (ppm)

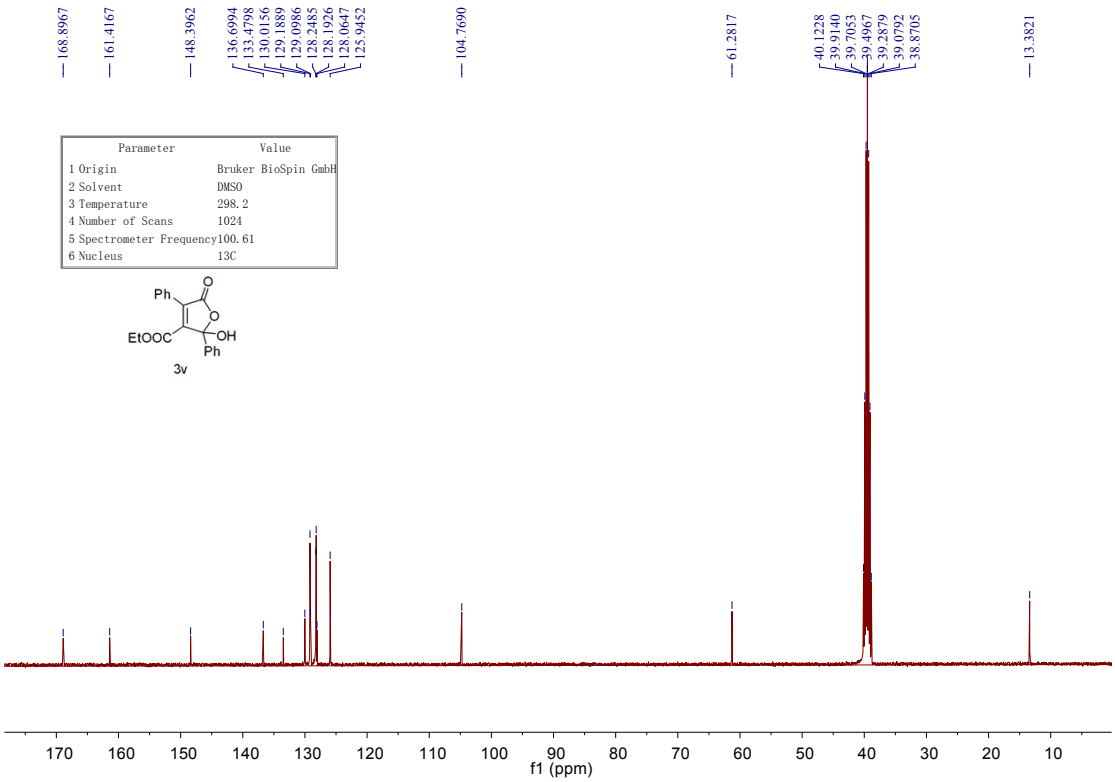

169.7342
156.8618
136.6697
131.6124
131.4761
130.5560
129.5055
129.3350
129.2922
129.0987
128.5612
128.1894
127.5236
123.3828
122.5372
104.9539
40.1258
39.9160
39.7073
39.4987
39.2899
39.0813
38.8729
Parameter Value
1 Origin Bruker BioSpin GmbH
2 Solvent DMSO
3 Temperature 298.2
4 Number of Scans 84
5 Spectrometer Frequency 100.61
6 Nucleus 13C
170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10
f1 (ppm)

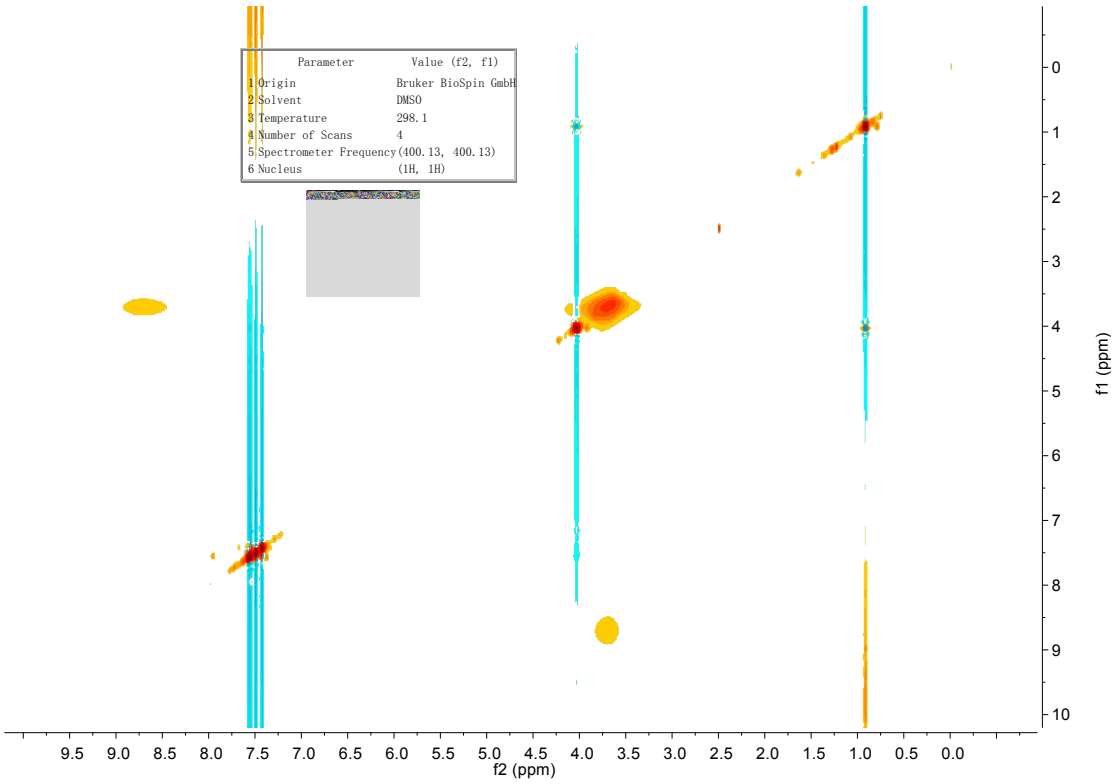

8.5465
7.7287
7.7250
7.7099
7.7058
7.4198
7.4101
7.4062
7.3979
7.3926
7.3862
7.3739
7.3689
7.3593
7.3498
7.3440
7.2593
7.2520
7.2457
7.2418
7.2377
7.2243
7.2180
7.2062
7.1943
7.1889
7.1855
7.1632
7.1601
7.1430
7.1391
2.5000
2.2916
Parameter Value
1 Origin Bruker BioSpin GmbH
2 Solvent DMSO
3 Temperature 298.2
4 Number of Scans 4
5 Spectrometer Frequency 400.13
6 Nucleus 1H
Ph
Ph
OH
Me
3u
1.00
0.99
5.08
8.27
2.93
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)

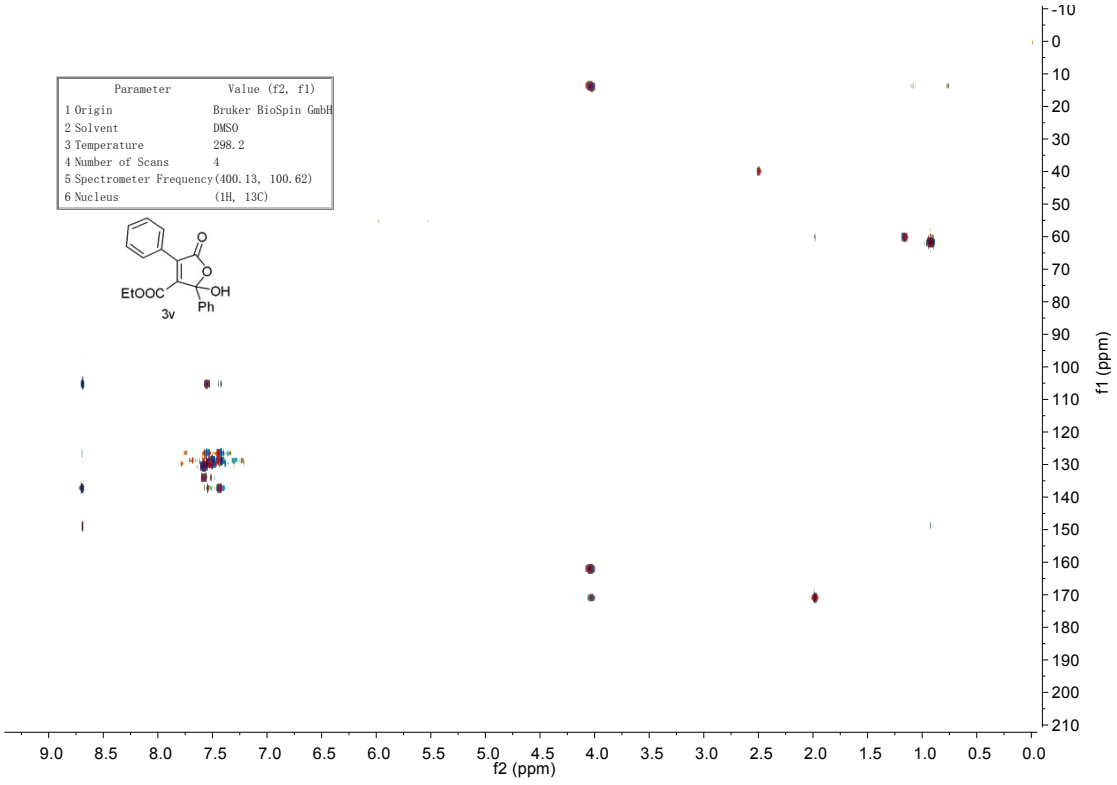

170.5317
157.8736
135.5107
134.6890
132.0467
130.4911
129.7904
129.6371
129.1913
129.0989
128.8888
128.5624
128.3501
127.9776
127.5732
125.9346
105.7206
40.1196
39.9107
39.7019
39.4932
39.2846
39.0758
38.8670
20.0486
Parameter Value
1 Origin Bruker BioSpin GmbH
2 Solvent DMSO
3 Temperature 298.2
4 Number of Scans 1024
5 Spectrometer Frequency 100.61
6 Nucleus 13C
f1 (ppm)


Parameter Value (f2, f1)
1 Origin Bruker BioSpin GmbH
2 Solvent DMSO
3 Temperature 298.2
4 Number of Scans 4
5 Spectrometer Frequency (400.13, 100.61)
6 Nucleus (1H, 13C)
Ph
Ph
OH
3u
f2 (ppm)
f1 (ppm)

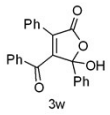

8.689
7.5682
7.5629
7.5525
7.5436
7.5380
7.5309
7.5269
7.5179
7.5133
7.5019
7.4971
7.4885
7.4845
7.4743
7.4528
7.4413
7.4305
7.4253
7.4173
7.4128
4.0637
4.0557
4.0460
4.0382
4.0283
4.0205
4.0107
4.0029
2.5000
0.9435
0.9258
0.9081
Parameter Value
1 Origin Bruker BioSpin GmbH
2 Solvent DMSO
3 Temperature 298.1
4 Number of Scans 16
5 Spectrometer Frequency 400.13
6 Nucleus 1H
Ph
O
O
EtOOC
OH
Ph
3v
0.80
6.90
3.09
2.00
2.89
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)


168.8967
161.4167
148.3962
136.6994
133.4798
130.0156
129.1889
129.0986
128.2485
128.1926
128.0647
125.9452
104.7690
61.2817
40.1228
39.9140
39.7053
39.4967
39.2879
39.0792
38.8705
13.3821
Parameter Value
1 Origin Bruker BioSpin GmbH
2 Solvent DMSO
3 Temperature 298.2
4 Number of Scans 1024
5 Spectrometer Frequency 100.61
6 Nucleus 13C
Ph
O
O
EtOOC
OH
Ph
3v
170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10
f1 (ppm)


Parameter Value (f2, f1)
1 Origin Bruker BioSpin GmbH
2 Solvent DMSO
3 Temperature 298.1
4 Number of Scans 4
5 Spectrometer Frequency (400.13, 400.13)
6 Nucleus (1H, 1H)
f2 (ppm)
f1 (ppm)


Parameter Value (f2, f1)
1 Origin Bruker BioSpin GmbH
2 Solvent DMSO
3 Temperature 298.2
4 Number of Scans 4
5 Spectrometer Frequency (400.13, 100.62)
6 Nucleus (1H, 13C)
EtOOC
OH
Ph
3v
f2 (ppm)
f1 (ppm)

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



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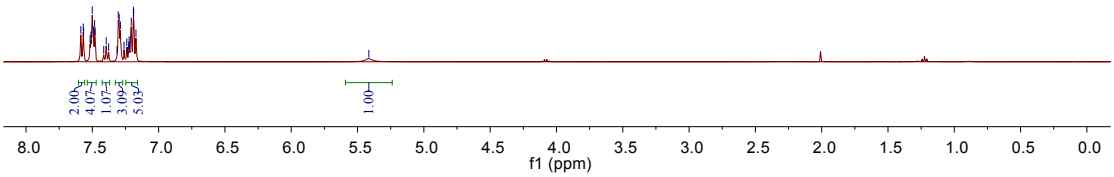
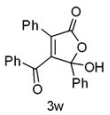
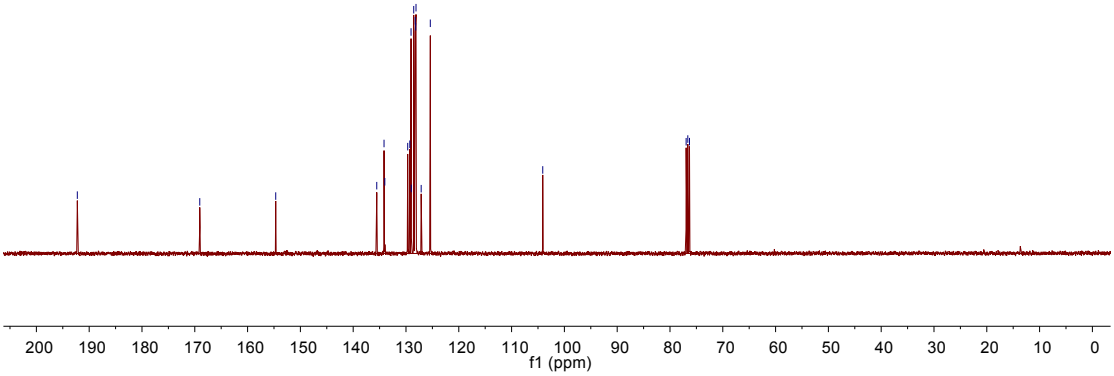
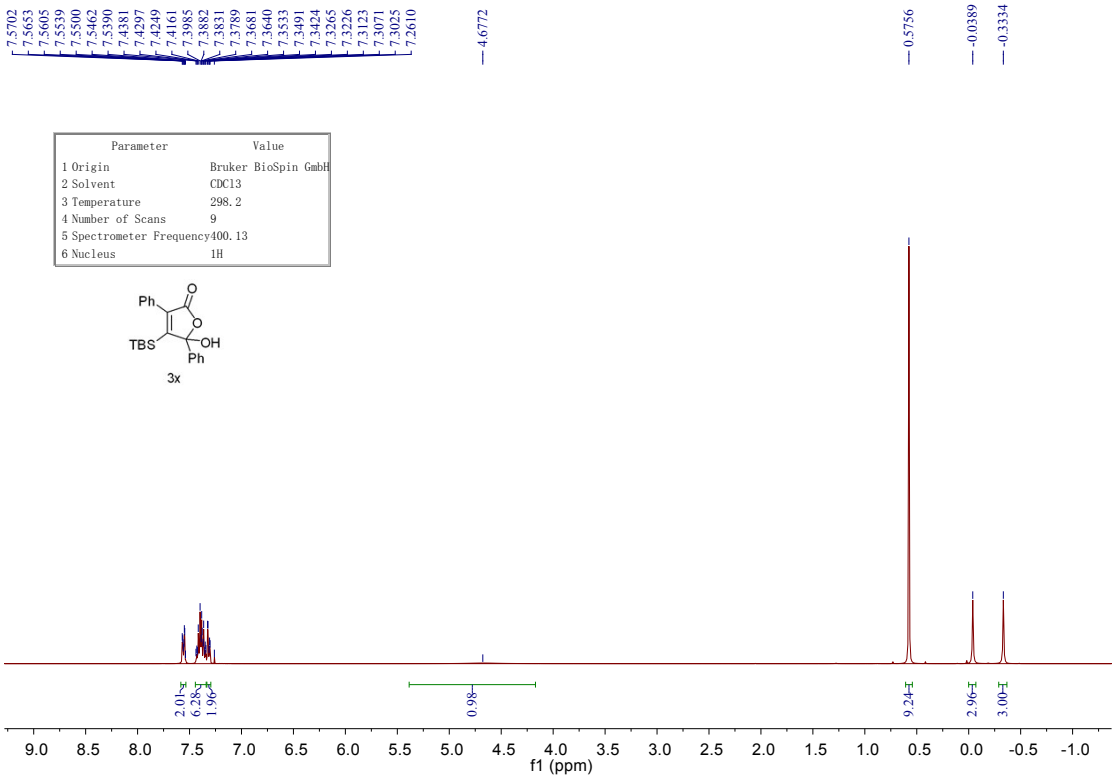


Parameter	Value
1 Origin	Bruker BioSpin GmbH
2 Solvent	CDCl3
3 Temperature	298.1
4 Number of Scans	57
5 Spectrometer Frequency	100.61
6 Nucleus	13C



3w

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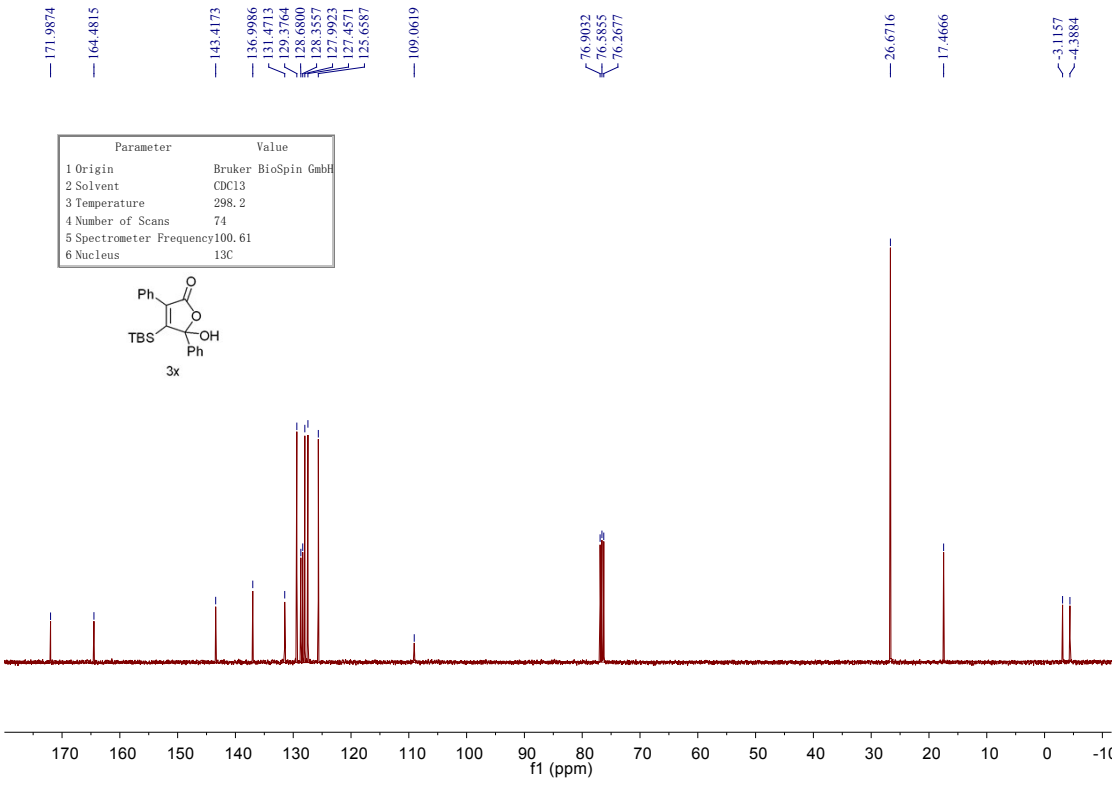
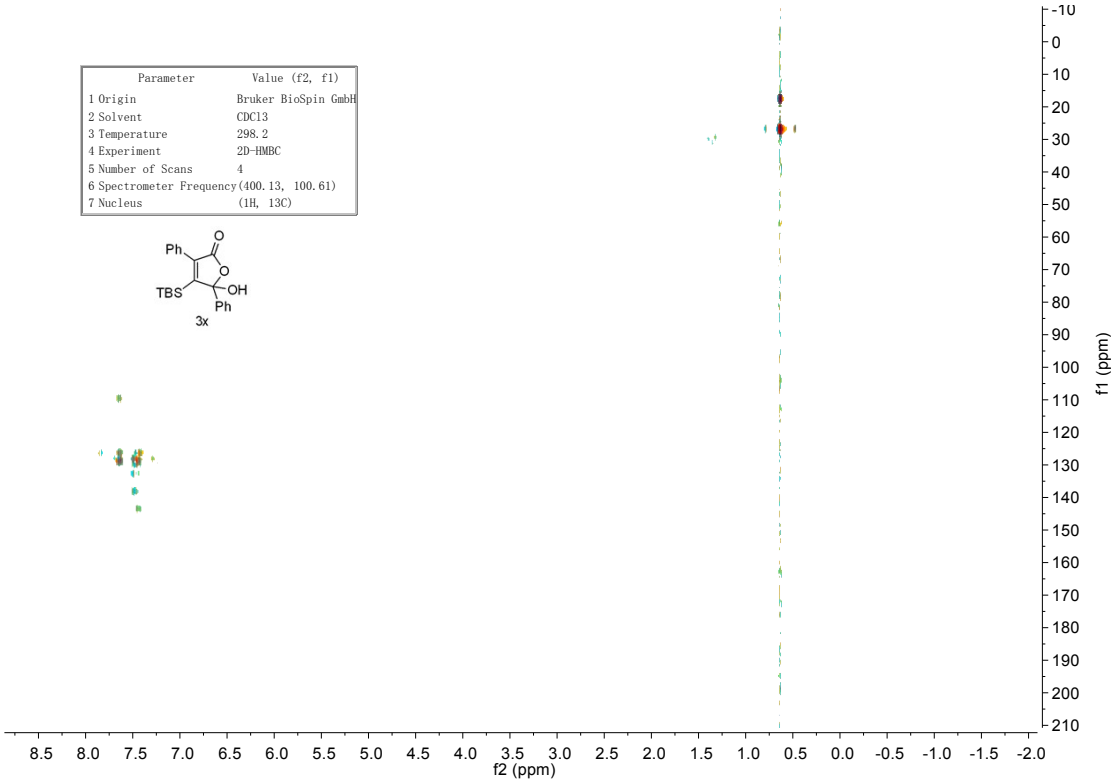
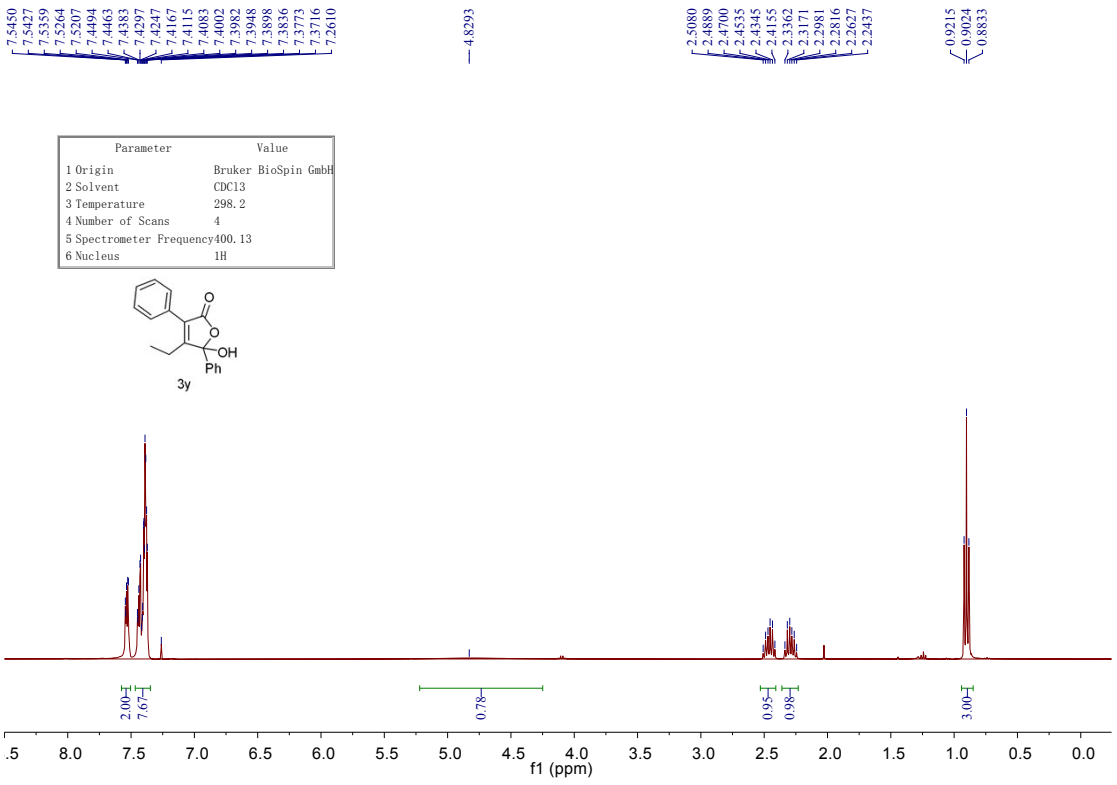
7.5605
7.5539
7.5500
7.5462
7.5390
7.4381
7.4297
7.4249
7.4161
7.3985
7.3882
7.3831
7.3789
7.3681
7.3640
7.3533
7.3491
7.3424
7.3265
7.3226
7.3123
7.3071
7.3025
7.2610
4.6772
0.5756
-0.0389
-0.3334
Parameter Value
1 Origin Bruker BioSpin GmbH
2 Solvent CDCl3
3 Temperature 298.2
4 Number of Scans 9
5 Spectrometer Frequency 400.13
6 Nucleus 1H
Ph
O
O
TBS
OH
Ph
3x
2.01
6.28
1.96
0.98
9.24
2.96
3.00
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 -1.0
f1 (ppm)


171.9874
164.4815
143.4173
136.9986
131.4713
129.3764
128.6800
128.3557
127.9923
127.4571
125.6587
109.0619
76.9032
76.5855
76.2677
26.6716
17.4666
-3.1157
-4.3884
Parameter Value
1 Origin Bruker BioSpin GmbH
2 Solvent CDCl3
3 Temperature 298.2
4 Number of Scans 74
5 Spectrometer Frequency 100.61
6 Nucleus 13C
Ph
O
O
TBS
OH
Ph
3x
170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)


Parameter Value (f2, f1)
1 Origin Bruker BioSpin GmbH
2 Solvent CDCl3
3 Temperature 298.2
4 Experiment 2D-HMBC
5 Number of Scans 4
6 Spectrometer Frequency (400.13, 100.61)
7 Nucleus (1H, 13C)
Ph
O
O
TBS
OH
Ph
3x
f2 (ppm)
f1 (ppm)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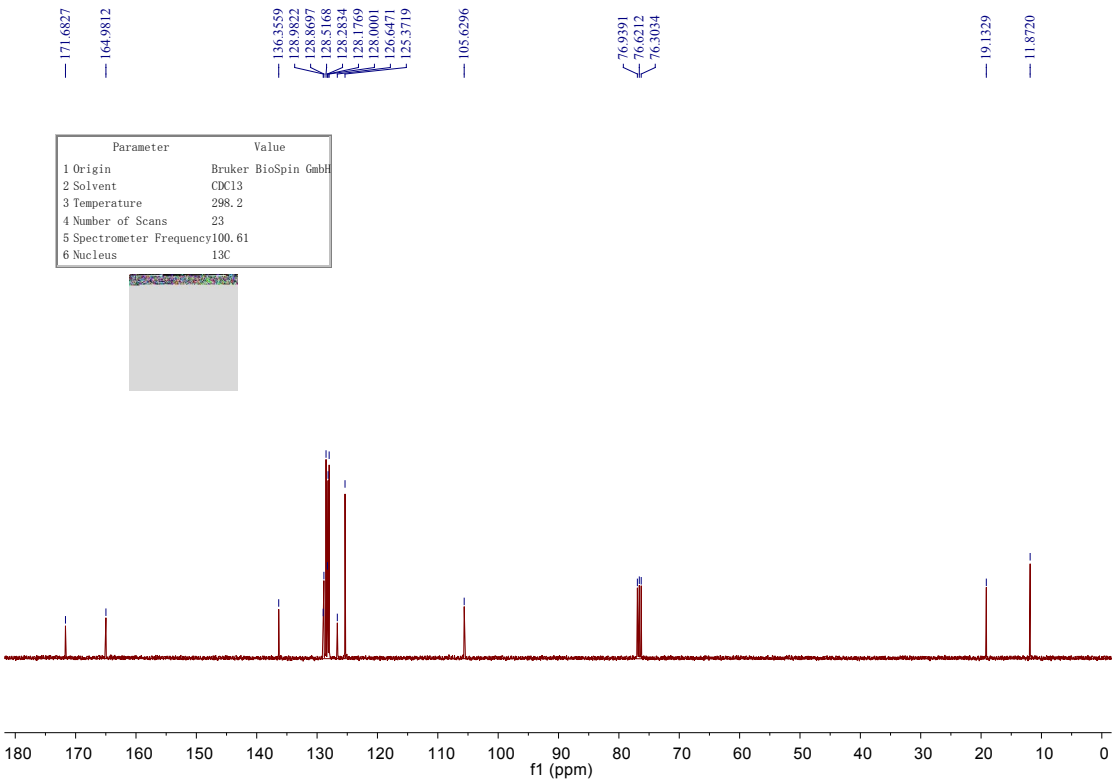
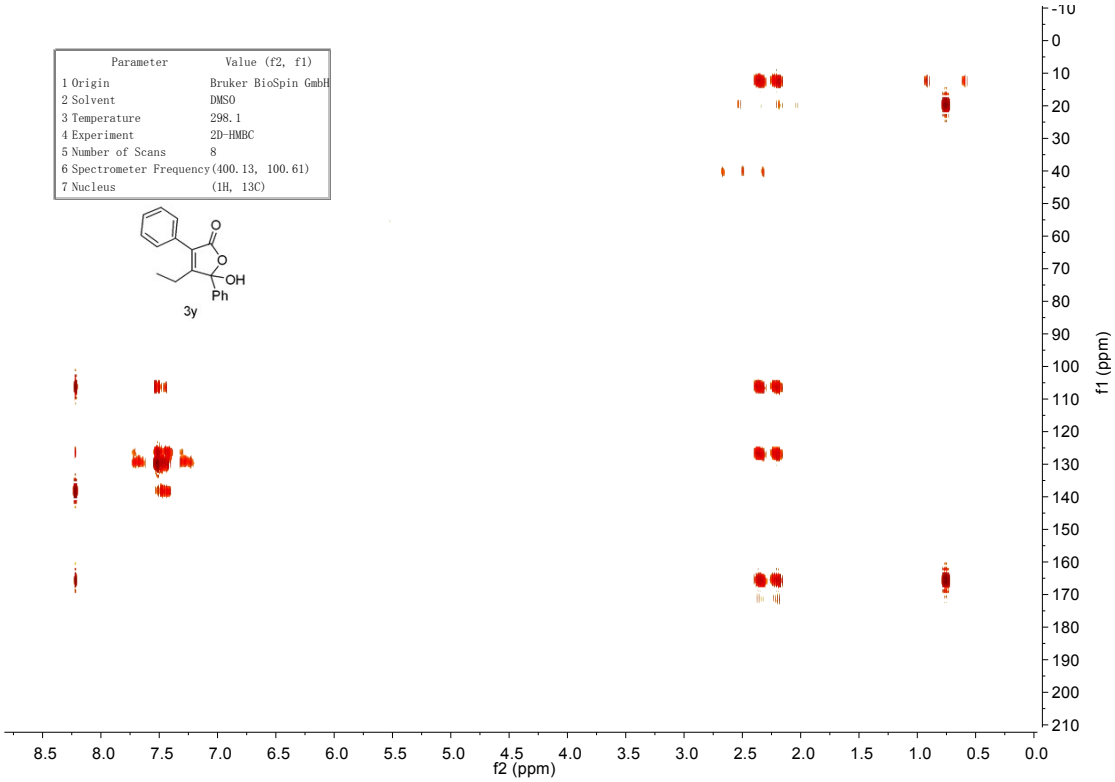
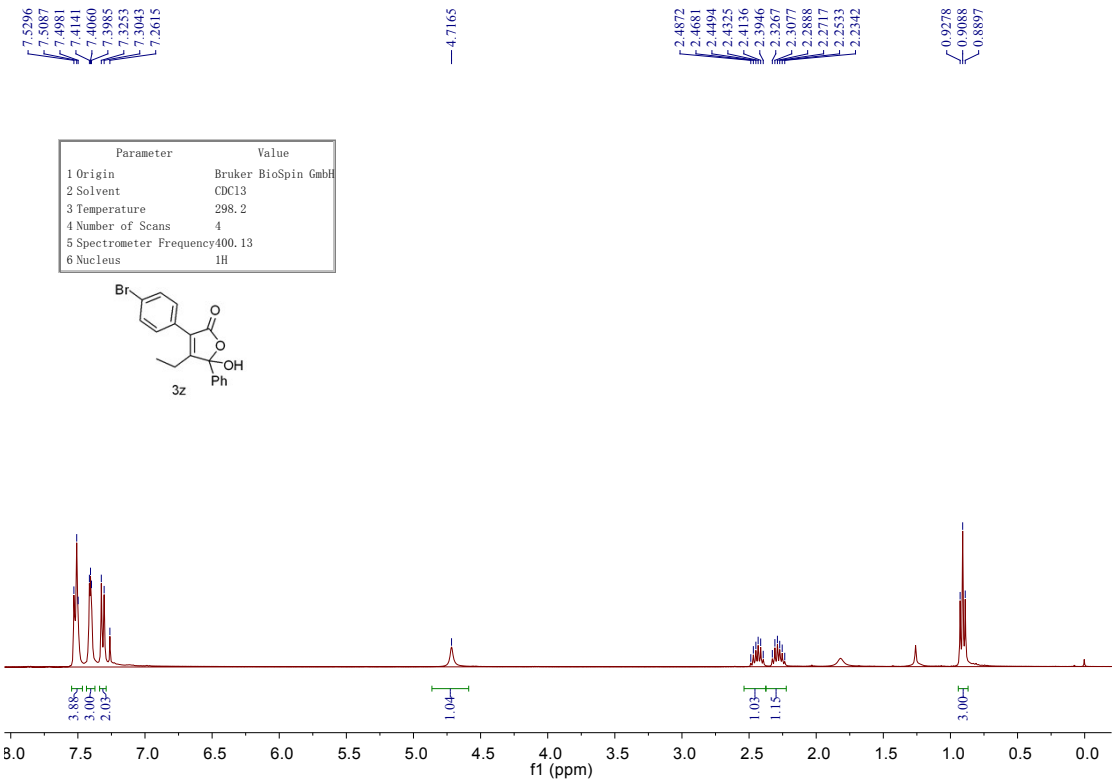
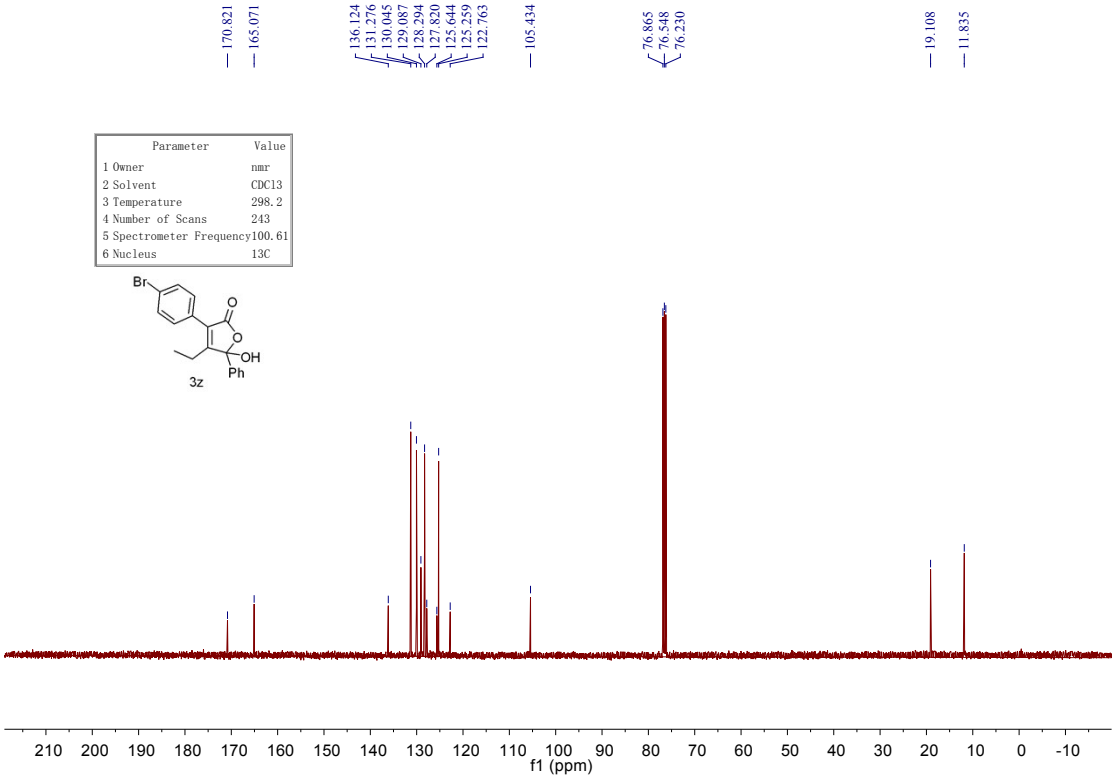
7.5359
7.5264
7.5207
7.4494
7.4463
7.4383
7.4297
7.4247
7.4167
7.4115
7.4083
7.4002
7.3982
7.3948
7.3898
7.3836
7.3773
7.3716
7.2610
4.8293
2.5080
2.4889
2.4700
2.4535
2.4345
2.4155
2.3362
2.3171
2.2981
2.2816
2.2627
2.2437
0.9215
0.9024
0.8833
Parameter Value
1 Origin Bruker BioSpin GmbH
2 Solvent CDCl3
3 Temperature 298.2
4 Number of Scans 4
5 Spectrometer Frequency 400.13
6 Nucleus 1H
O
O
OH
Ph
3y
2.00
7.67
0.78
0.95
0.98
3.00
f1 (ppm)


171.6827
164.9812
136.3559
128.9822
128.8697
128.5168
128.2834
128.1769
128.0001
126.6471
125.3719
105.6296
76.9391
76.6212
76.3034
19.1329
11.8720
Parameter Value
1 Origin Bruker BioSpin GmbH
2 Solvent CDCl3
3 Temperature 298.2
4 Number of Scans 23
5 Spectrometer Frequency 100.61
6 Nucleus 13C
f1 (ppm)


Parameter Value (f2, f1)
1 Origin Bruker BioSpin GmbH
2 Solvent DMSO
3 Temperature 298.1
4 Experiment 2D-HMBC
5 Number of Scans 8
6 Spectrometer Frequency (400.13, 100.61)
7 Nucleus (1H, 13C)
O
O
OH
Ph
3y
f2 (ppm)
f1 (ppm)


7.5296
7.5087
7.4981
7.4141
7.4060
7.3985
7.3253
7.3043
7.2615
4.7165
2.4872
2.4681
2.4494
2.4325
2.4136
2.3946
2.3267
2.3077
2.2888
2.2717
2.2533
2.2342
0.9278
0.9088
0.8897
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
Br
O
O
OH
Ph
3z
3.88
3.00
2.03
1.04
1.03
1.15
3.00
f1 (ppm)


170.821
165.071
136.124
131.276
130.045
129.087
128.294
127.820
125.644
125.259
122.763
105.434
76.865
76.548
76.230
19.108
11.835
Parameter Value
1 Owner nmr
2 Solvent CDCl3
3 Temperature 298.2
4 Number of Scans 243
5 Spectrometer Frequency 100.61
6 Nucleus 13C
Br
O
O
OH
Ph
3z
f1 (ppm)