Supplementary Information for

Aryne reaction with trifluoromethyl ketones in three modes: C–C bond cleavage, [2+2] cycloaddition and *O*-arylation

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Contents

General remarks.

All manipulations of oxygen- and moisture-sensitive materials were conducted with a standard Schlenk technique under a purified argon atmosphere. Nuclear magnetic resonance spectra were taken on a Varian 400-MR (^{1}H, 400 MHz; ^{13}C, 100 MHz) spectrometer or a Varian System 500 (^{1}H, 500 MHz; ^{13}C, 125 MHz) spectrometer using residual chloroform (^{1}H, δ = 7.26) or $CDCl_3$ (^{13}C, δ = 77.0) as an internal standard. ^{1}H NMR data are reported as follows: chemical shift, multiplicity (s = singlet, d = doublet, t = triplet, q = quartet, m = multiplet, br = broad), coupling constants (Hz), integration. Infrared spectra (IR) were recorded on a Perkin Elmer spectrum one spectrometer. High-resolution mass spectra (ESI or APCI/FTMS; Negative Mode) were obtained with Thermo Fisher Scientific LTQ Orbitrap XL spectrometer. Melting points were measured with Yanaco Micro Melting Point apparatus and uncorrected. Preparative recycling gel permeation chromatography was performed with GL Science PU 614 equipped with Shodex GPC H-2001L and -2002L columns (chloroform or toluene as an eluent). Column chromatography was carried out using Merck Kieselgel 60. Unless otherwise noted, commercially available reagents were used without purification. 18-Crown-6 was recrystallized from distilled MeCN. KF (spray-dried) was vacuum dried at 100 °C for 12 h. THF was distilled from sodium/benzophenone ketyl. MeCN was distilled from phosphorus pentoxide.

Aryne precursors.

2-(Trimethylsilyl)phenyl triflate (**1a**),[1] 4,5-dimethyl-2-(trimethylsilyl)phenyl triflate (**1b**),[2] 3-(trimethylsilyl)-2-naphthyl triflate (**1c**)[3] and 3-methoxy-2-(trimethylsilyl)phenyl triflate (**1d**)[4] were prepared according to literature procedures.

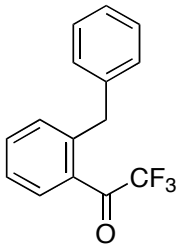
Trifluoromethyl ketones.

All trifluoromethyl ketones were synthesized according to literature procedures.[5]

Reaction of arynes with trifluoromethyl ketones: a general procedure.

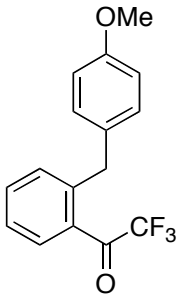
A Schlenk tube equipped with a magnetic stirring bar was charged with KF (0.30 mmol) and 18-crown-6 (0.30 mmol). The tube was evacuated at room temperature for 1 h with stirring before addition of THF (5 mL), a trifluoromethyl ketone (0.23 mmol), and an aryne precursor (0.15 mmol) under an argon atmosphere. The resulting mixture was stirred at the temperature for the period as specified in Schemes 2, 3 and 5. The mixture was diluted with ethyl acetate and the organic solution was washed twice with brine. The organic layer was dried over $MgSO_4$ and the solvent was removed in vacuo. Silica gel-column chromatography (hexane/ethyl acetate as an eluent) or preparative recycling gel permeation chromatography (chloroform or toluene as an eluent) gave the corresponding product.

1-(2-Benzylphenyl)-2,2,2-trifluoroethanone (3aa)



Isolated in 60% yield as a colorless oil: ^{1}H NMR ($CDCl_3$) δ 4.31 (s, 2 H, C*H*$_2$), 7.13 (d, *J* = 7.3 Hz, 2 H, Ar*H*), 7.21 (t, *J* = 7.3 Hz, 1 H, Ar*H*), 7.29 (t, *J* = 7.3 Hz, 2 H, Ar*H*), 7.33 (d, *J* = 7.6 Hz, 1 H, Ar*H*), 7.40 (td, *J* = 7.7, 1.0 Hz, 1 H, Ar*H*), 7.57 (td, *J* = 7.5, 1.2 Hz, 1 H, Ar*H*), 7.89 (dt, *J* = 7.8, 1.5 Hz, 1 H, Ar*H*); ^{13}C NMR ($CDCl_3$) δ 39.4, 116.3 (q, J_{C-F} = 292.6 Hz), 126.3, 126.5, 128.5, 129.0, 129.6, 130.2 (q, J_{C-F} = 3.8 Hz), 132.5, 134.0, 139.7, 144.3, 182.7 (q, J_{C-F} = 34.0 Hz); IR (neat) 611, 663, 696, 730, 750, 926, 937, 1139, 1181, 1449, 1495, 1572, 1599, 1716 cm^{-1}; HRMS Calcd for $C_{15}H_{10}F_3O$: [M-H]$^-$, 263.06892. Found: *m*/*z* 263.06854.

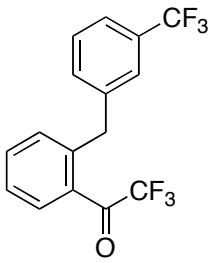
2,2,2-Trifluoro-1-[2-(4-methoxybenzyl)phenyl]ethanone (3ab)



Isolated in 55% yield as an orange oil: ^{1}H NMR ($CDCl_3$) δ 3.79 (s, 3 H, OC*H*$_3$), 4.23 (s, 2 H, C*H*$_2$), 6.82 (d, *J* = 8.8 Hz, 2 H, Ar*H*), 7.05 (d, *J* = 8.8 Hz, 2 H, Ar*H*), .7.32 (d. *J* = 7.5

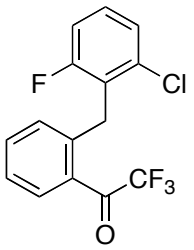
Hz, 1 H, Ar*H*), 7.38 (td, *J* = 7.9, 1.3 Hz, 1 H, Ar*H*), 7.56 (td, *J*, = 7.6, 1.2 Hz, 1 H, Ar*H*), 7.87 (dt, *J* = 7.8, 1.7 Hz, 1 H, Ar*H*); ^{13}C NMR ($CDCl_3$) δ 38.5, 55.2, 113.9, 116.3 (q, $J_{C–F}$ = 293.1 Hz), 126.3, 129.5, 130.0, 130.2 (q, $J_{C–F}$ = 3.7 Hz), 131.8, 132.2, 133.9, 144.8, 158.1, 182.7 (q, $J_{C–F}$ = 34.0 Hz); IR (neat) 663, 732, 742, 769, 802, 835, 908, 935, 1034, 1139, 1178, 1245, 1510, 1716 cm^{-1}; HRMS Calcd for $C_{16}H_{12}F_3O_2$: [M-H]$^-$, 293.07949. Found: *m/z* 293.07901.

2,2,2-Trifluoro-1-{2-[3-(trifluoromethyl)benzyl]phenyl}ethanone (3ac)



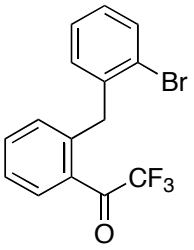
Isolated in 63% yield as a yellow oil: ^{1}H NMR ($CDCl_3$) δ 4.35 (s, 2 H, C*H*$_2$), 7.30-7.35 (m, 2 H, Ar*H*), 7.36-7.42 (m, 2 H, Ar*H*), 7.43-7.49 (m, 2 H, Ar*H*), 7.61 (td, *J* = 7.6, 1.2 Hz, 1 H, Ar*H*), 7.93 (dt, *J* = 7.8, 1.7 Hz, 1 H, Ar*H*); ^{13}C NMR ($CDCl_3$) δ 39.3, 116.3 (q, $J_{C–F}$ = 292.6 Hz), 123.2 (q, $J_{C–F}$ = 3.7 Hz), 124.1 (q, $J_{C–F}$ = 272.1 Hz), 125.5 (q, $J_{C–F}$ = 3.7 Hz), 127.0, 128.9, 129.5, 130.6 (q, $J_{C–F}$ = 3.7 Hz), 130.8 (q, $J_{C–F}$ = 32.1 Hz), 132.3, 132.6, 134.3, 140.7, 143.1, 182.6 (q, $J_{C–F}$ = 34.4 Hz); IR (neat) 660, 701, 735, 792, 916, 929, 941, 1073, 1094, 1120, 1144, 1182, 1326, 1449, 1717 cm^{-1}; HRMS Calcd for $C_{16}H_9F_6O$: [M-H]$^-$, 331.05631. Found: *m/z* 331.05569.

1-[2-(2-Chloro-6-fluorobenzyl)phenyl]-2,2,2-trifluoroethanone (3ad)



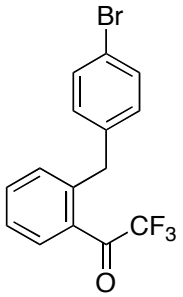
Isolated in 42% yield as a pale yellow solid: m.p. 54–55 °C; ^{1}H NMR ($CDCl_3$) δ 4.49 (d, $J_{H–F}$ = 1.5 Hz, 2 H, C*H*$_2$), 6.98-7.06 (m, 2 H, Ar*H*), 7.20-7.26 (m, 2 H, Ar*H*), 7.37 (t, *J* = 7.6 Hz, 1 H, Ar*H*), 7.48 (td, *J* = 7.8, 1.2 Hz, 1 H, Ar*H*), 7.93 (dt, *J* = 7.8, 1.7 Hz, 1 H, Ar*H*); ^{13}C NMR ($CDCl_3$) δ 29.8 (d, $J_{C–F}$ = 3.3 Hz), 114.1 (d, $J_{C–F}$ = 22.8 Hz), 116.4 (q, $J_{C–F}$ = 292.6 Hz), 125.5 (d, $J_{C–F}$ = 3.7 Hz), 125.6, 126.3, 128.8 (d, $J_{C–F}$ = 9.8 Hz), 129.3, 129.5, 130.2 (q, $J_{C–F}$ = 3.7 Hz), 134.2, 135.9 (d, $J_{C–F}$ = 5.6 Hz), 142.0, 161.7 (d, $J_{C–F}$ = 248.4 Hz), 182.8 (q, $J_{C–F}$ = 34.5 Hz); IR (neat) 665, 719, 730, 774, 936, 1144, 1179, 1246, 1453, 1572, 1709 cm^{-1}; HRMS Calcd for $C_{15}H_9ClF_4O$: M$^-$, 316.02835. Found: *m/z* 316.02771.

1-[2-(2-Bromobenzyl)phenyl]-2,2,2-trifluoroethanone (3ae)



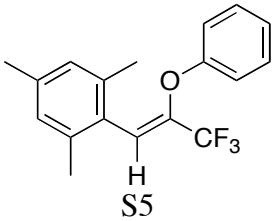
Isolated in 65% yield as a pale yellow oil: ^{1}H NMR ($CDCl_3$) δ 4.40 (s, 2 H, C*H*$_2$), 6.97 (dd, *J* = 7.6, 1.5 Hz, 1 H, Ar*H*), 7.10-7.16 (m, 2 H, Ar*H*), 7.23 (td, *J* = 7.4, 1.2 Hz, 1 H, Ar*H*), 7.42 (td, *J* = 8.1, 1.2 Hz, 1 H, Ar*H*), 7.55 (td, *J* = 7.9, 1.2 Hz, 1 H, Ar*H*), 7.60 (dd, *J* = 8.1, 1.2 Hz, 1 H, Ar*H*), 7.96 (dt, *J* = 7.8, 1.7 Hz, 1 H, Ar*H*); ^{13}C NMR ($CDCl_3$) δ 39.9, 116.4 (q, J_{C-F} = 293.1 Hz), 125.2, 126.6, 127.6, 128.2, 129.5, 130.4 (q, J_{C-F} = 3.7 Hz), 130.7, 131.7, 133.0, 134.2, 139.2, 143.0, 182.4 (q, J_{C-F} = 34.4 Hz); IR (neat) 662, 717, 735, 913, 936, 1026, 1139, 1181, 1440, 1467, 1571, 1601, 1715 cm^{-1}; HRMS Calcd for $C_{15}H_9BrF_3O$: [M-H]$^-$, 340.97944. Found: *m/z* 340.97922.

1-[2-(4-Bromobenzyl)phenyl]-2,2,2-trifluoroethanone (3af)



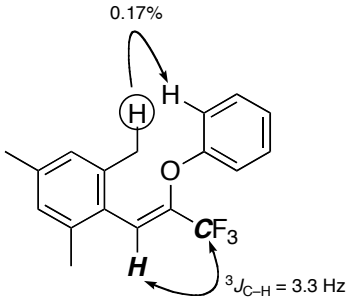
Isolated in 58% yield as an orange oil: ^{1}H NMR ($CDCl_3$) δ 4.25 (s, 2 H, C*H*$_2$), 7.00 (d, *J* = 8.3 Hz, 2 H, Ar*H*), 7.32 (d, *J* = 7.8 Hz, 1 H, Ar*H*), 7.39 (d, *J* = 8.3 Hz, 2 H, Ar*H*), 7.42 (t, *J* = 8.1 Hz, 1 H, Ar*H*), 7.59 (td, *J* = 7.6, 1.3 Hz, 1 H, Ar*H*), 7.91 (dt, *J* = 8.1, 1.4 Hz, 1 H, Ar*H*); ^{13}C NMR ($CDCl_3$) δ 39.0, 116.3 (q, J_{C-F} = 292.6 Hz), 120.2, 126.8, 129.4, 130.6 (q, J_{C-F} = 3.8 Hz), 130.7, 131.5, 132.5, 134.2, 138.8, 143.6, 182.5 (q, J_{C-F} = 34.4 Hz); IR (neat) 478, 664, 736, 750, 790, 911, 937, 1011, 1071, 1141, 1181, 1487, 1716 cm^{-1}; HRMS Calcd for $C_{15}H_9BrF_3O$: [M-H]$^-$, 340.97944. Found: *m/z* 340.97919.

(*Z*)-1,3,5-Trimethyl-2-(3,3,3-trifluoro-2-phenoxyprop-1-enyl)benzene (3ag)

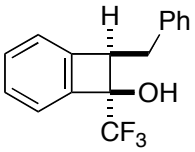


Isolated in 55% yield as a yellow oil: ^{1}H NMR ($CDCl_3$) δ 2.16 (s, 3 H, C*H*$_3$), 2.22 (s, 6 H, C*H*$_3$), 6.70 (s, 2 H, Ar*H*), 6.79 (d, *J* = 7.9 Hz, 2 H, Ar*H*), 6.85 (s, 1 H, C=C*H*), 6.88 (d, *J* = 7.3 Hz, 1 H, Ar*H*), 7.05-7.11 (m, 2 H, Ar*H*) ; ^{13}C NMR ($CDCl_3$) δ 20.0, 20.9, 115.9, 118.9 (q, J_{C-F} = 3.7 Hz), 120.6 (q, J_{C-F} = 274.5 Hz), 122.9, 126.5, 128.1, 129.0, 136.0, 137.8, 140.2 (q, J_{C-F} = 33.9 Hz), 156.2; IR (neat) 493, 688, 749, 850, 1061, 1130, 1177, 1205, 1291, 1352, 1490, 1592 cm^{-1}; HRMS Calcd for $C_{18}H_{16}F_3O$: [M-H]$^-$, 305.11587. Found: *m*/*z* 305.11523.

The stereochemistry of **3ag** was determined by NOE experiment and $^{3}J_{C-H}$ coupling constant[6] as shown below.

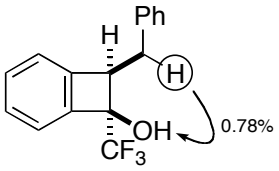


***c*-2-Benzyl-1-(trifluoromethyl)-1,2-dihydrocyclobutabenzen-*r*-1-ol (3ah)**

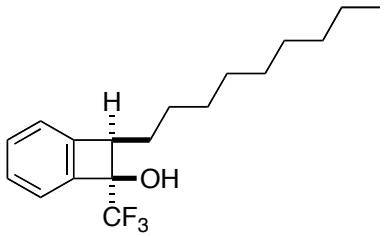


Isolated in 47% yield as a colorless oil: ^{1}H NMR ($CDCl_3$) δ 2.75 (brs, 1 H, O*H*), 2.97 (dd, *J* = 13.5, 9.5 Hz, 1 H, PhC*H*H), 3.18 (dd, *J* = 13.5, 7.0 Hz, 1 H, PhCH*H*), 4.14 (dd, *J* = 9.5, 7.0 Hz, 1 H, PhCH$_2$C*H*), 6.88-6.92 (m, 1 H, Ar*H*), 7.26-7.39 (m, 8 H, Ar*H*); ^{13}C NMR ($CDCl_3$) δ 35.5, 51.4, 79.8 (q, J_{C-F} = 32.5 Hz), 122.3, 123.4, 124.7 (q, J_{C-F} = 280.4 Hz), 126.6, 128.6, 128.7, 129.0, 131.1, 138.7, 140.1, 146.8; IR (neat) 497, 696, 706, 752, 929, 1072, 1116, 1146, 1307, 1455 cm^{-1}; HRMS Calcd for $C_{16}H_{12}F_3O$: [M-H]$^-$, 277.08457. Found: *m*/*z* 277.08411.

The stereochemistry of **3ah** was determined by NOE experiment as shown below.

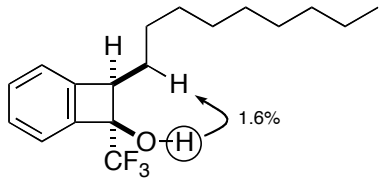


***c*-2-Nonyl-1-(trifluoromethyl)-1,2-dihydrocyclobutabenzen-*r*-1-ol (3ai)**

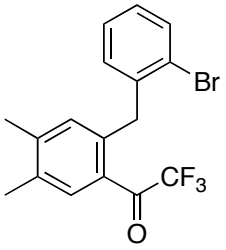


Isolated in 35% yield as a a colorless oil: ^{1}H NMR ($CDCl_3$) δ 0.89 (t, *J* = 7.0 Hz, 3 H, C*H*$_3$), 1.22-1.80 (m, 16 H, -(C*H*$_2$)$_8$-), 2.67 (brs, 1 H, O*H*), 3.79 (t, *J* = 7.5 Hz, 1 H, C*H*), 7.21 (dd, *J* = 8.0, 1.0 Hz, 1 H, Ar*H*), 7.27-7.33 (m, 2 H, Ar*H*), 7.39 (td, *J* = 7.0, 1.5 Hz, 1 H, Ar*H*); ^{13}C NMR ($CDCl_3$) δ 14.1, 22.7, 27.4, 29.3, 29.51, 29.54, 29.6, 31.9, 50.6, 79.4 (q, J_{C-F} = 31.9 Hz), 122.2, 123.0, 124.8 (q, J_{C-F} = 280.4 Hz), 128.3, 131.1, 140.4, 147.7; IR (neat) 742, 757, 1079, 1155, 1285, 1308, 1459, 2855, 2924 cm^{-1}; HRMS Calcd for $C_{18}H_{24}F_3O$: $[M-H]^-$, 313.17847. Found: *m*/*z* 313.17780.

The stereochemistry of **3ai** was determined by NOE experiment as shown below.

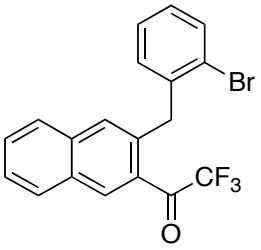


1-[2-(2-Bromobenzyl)-4,5-dimethylphenyl]-2,2,2-trifluoroethanone (3be)



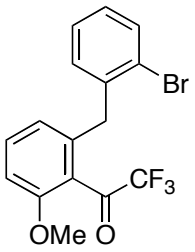
Isolated in 57% yield as a colorless oil: ^{1}H NMR ($CDCl_3$) δ 2.27 (s, 3 H, C*H*$_3$), 2.33 (s, 3 H, C*H*$_3$), 4.35 (s, 2 H, C*H*$_2$), 6.89 (dd, *J* = 7.6, 1.8 Hz, 1 H, Ar*H*), 6.92 (s, 1 H, Ar*H*), 7.10 (td, *J* = 7.9, 1.7 Hz, 1 H, Ar*H*), 7.19 (td, *J* = 7.6, 1.2 Hz, 1 H, Ar*H*), 7.59 (dd, *J* = 8.1, 1.2 Hz, 1 H, Ar*H*), 7.72 (s, 1 H, Ar*H*); ^{13}C NMR ($CDCl_3$) δ 19.4, 20.2, 39.7, 116.5 (q, J_{C-F} = 291.5 Hz), 125.1, 127.0, 127.4, 127.9, 130.3, 131.8 (q, J_{C-F} = 3.8 Hz), 132.8, 133.3, 135.1, 139.7, 140.7, 144.4, 181.7 (q, J_{C-F} = 33.8 Hz); IR (neat) 732, 741, 897, 911, 1024, 1074, 1138, 1191, 1439, 1708 cm^{-1}; HRMS Calcd for $C_{17}H_{13}BrF_3O$: $[M-H]^-$, 369.01074. Found: *m*/*z* 369.01044.

1-[3-(2-Bromobenzyl)naphthalen-2-yl]-2,2,2-trifluoroethanone (3ce)



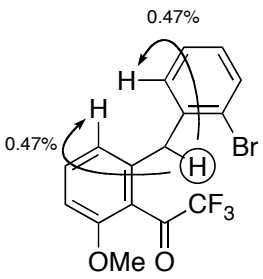
Isolated in 33% yield as a colorless solid: m.p. 78–81 °C; ^{1}H NMR ($CDCl_3$) δ 4.53 (s, 2 H, C*H*$_2$), 6.97 (d, *J* = 7.5 Hz, 1 H, Ar*H*), 7.13 (td, *J* = 7.5, 1.5 Hz, 1 H, Ar*H*), 7.22 (td, *J* = 7.5, 1.0 Hz, 1 H, Ar*H*), 7.51 (s, 1 H, Ar*H*), 7.54-7.68 (m, 3 H, Ar*H*), 7.74 (d, *J* = 8.0 Hz, 1 H, Ar*H*), 7.98 (d, *J* = 8.0 Hz, 1 H, Ar*H*), 8.53 (s, 1 H, Ar*H*) ; ^{13}C NMR ($CDCl_3$) δ 40.1, 116.5 (q, J_{C-F} = 291.0 Hz), 125.2, 127.1, 127.5, 127.6, 128.1, 129.4, 130.3, 130.58, 130.64, 130.7, 132.9, 133.6 (q, J_{C-F} = 3.6 Hz), 135.5, 136.9, 139.6, 182.1 (q, J_{C-F} = 33.8 Hz); IR (neat) 470, 729, 742, 750, 1022, 1095, 1145, 1178, 1465, 1696 cm^{-1}; HRMS Calcd for $C_{19}H_{11}BrF_3O$: $[M-H]^-$, 390.99509. Found: *m*/*z* 390.99478.

1-[2-(2-Bromobenzyl)-6-methoxyphenyl]-2,2,2-trifluoroethanone (3de)



Isolated in 53% yield as a colorless oil: ^{1}H NMR ($CDCl_3$) δ 3.88 (s, 3 H, OC*H*$_3$), 4.03 (s, 2 H, C*H*$_2$), 6.69 (d, *J* = 8.0 Hz, 1 H, Ar*H*), 6.87 (d, *J* = 8.5 Hz, 1 H, Ar*H*), 7.03 (dd, *J* = 8.0, 1.5 Hz, 1 H, Ar*H*), 7.10 (td, *J* = 8.0, 1.5 Hz, 1 H, Ar*H*), 7.23 (td, *J* = 7.5, 1.0 Hz, 1 H, Ar*H*), 7.37 (t, *J* = 8.0 Hz, 1 H, Ar*H*), 7.56 (dd, *J* = 8.0, 1.0 Hz, 1 H, Ar*H*); ^{13}C NMR ($CDCl_3$) δ 38.3, 55.9, 109.2, 115.3 (q, J_{C-F} = 290.0 Hz), 122.6, 123.4, 124.8, 127.5, 128.3, 131.2, 132.85, 132.87, 138.8, 140.2, 158.5, 188.6 (q, J_{C-F} = 37.4 Hz); IR (neat) 651, 739, 750, 768, 921, 941, 1025, 1068, 1144, 1174, 1203, 1270, 1437, 1471, 1579, 1725 cm^{-1}; HRMS Calcd for $C_{16}H_{11}BrF_3O_2$: $[M-H]^-$, 370.99000. Found: *m*/*z* 370.98962.

The structure of **3de** was determined by NOE experiment as shown below.



References

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5 (*a*) X. Creary, *J. Org. Chem.*, 1987, **52**, 5026; (*b*) J. T. Reeves, J. J. Song, Z. Tan, H. Lee, N. K. Yee and C. H. Senanayake, *J. Org. Chem.*, 2008, **73**, 9476.

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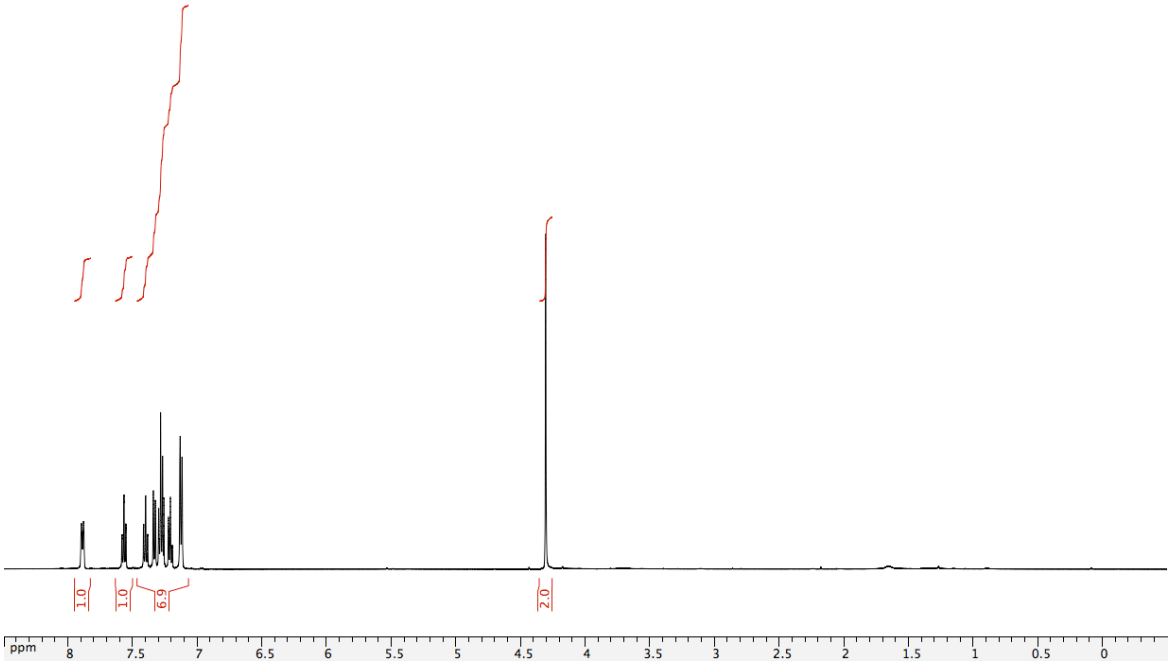
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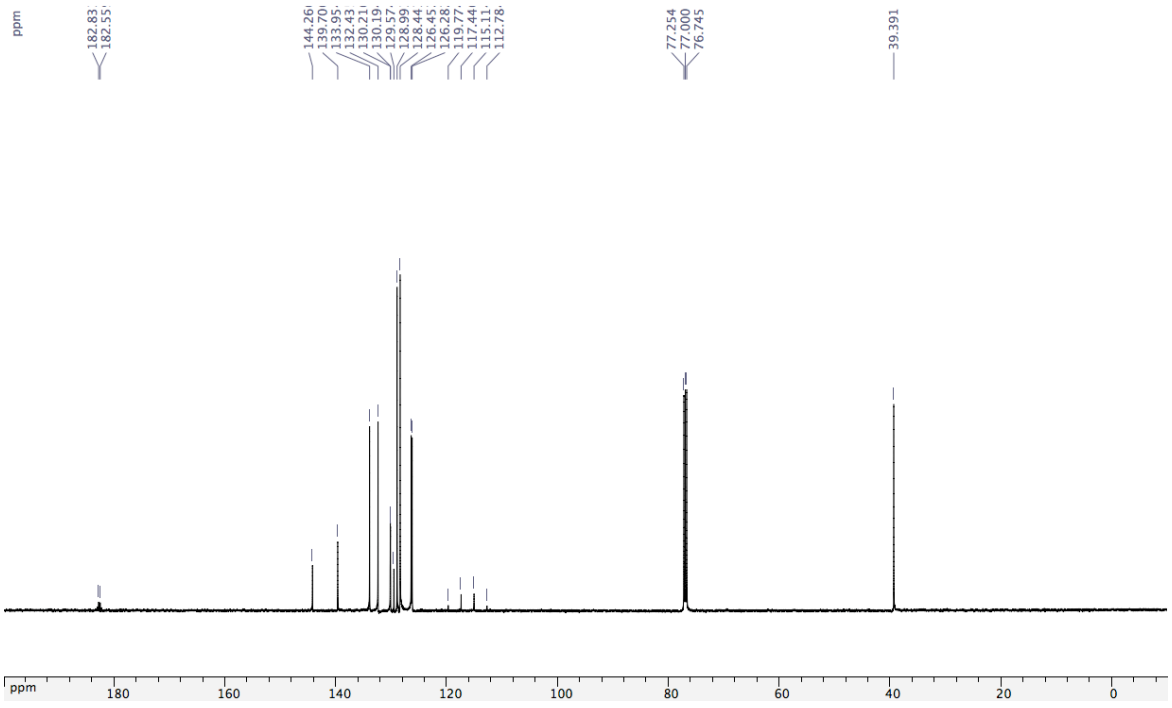
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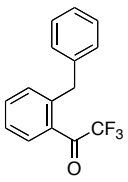
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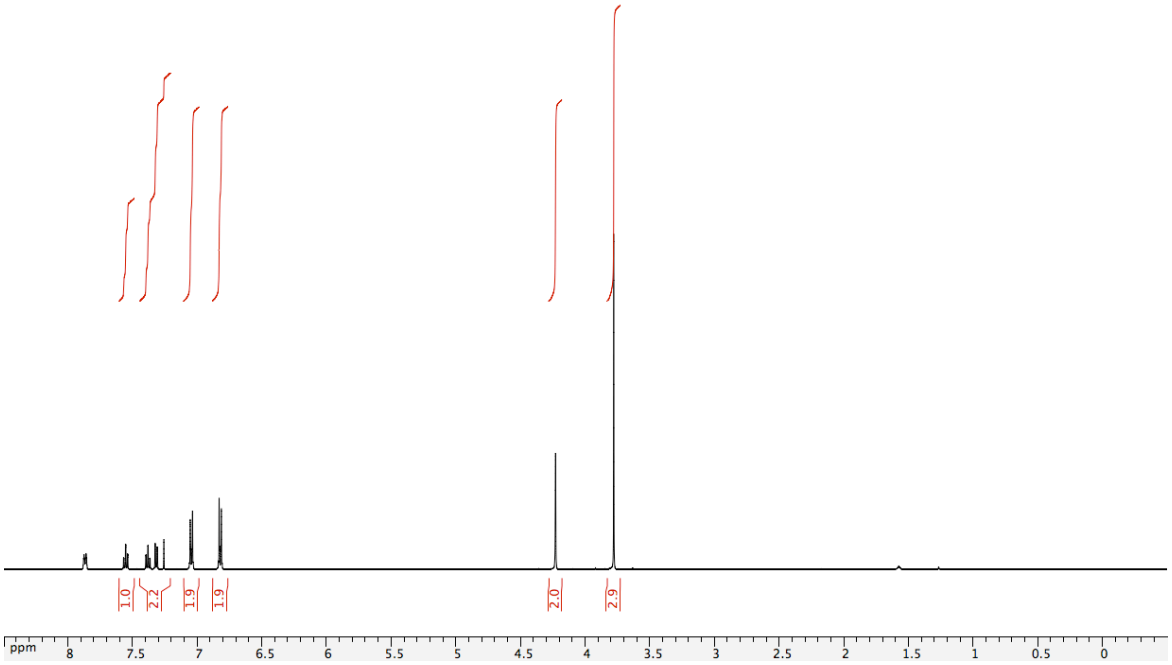
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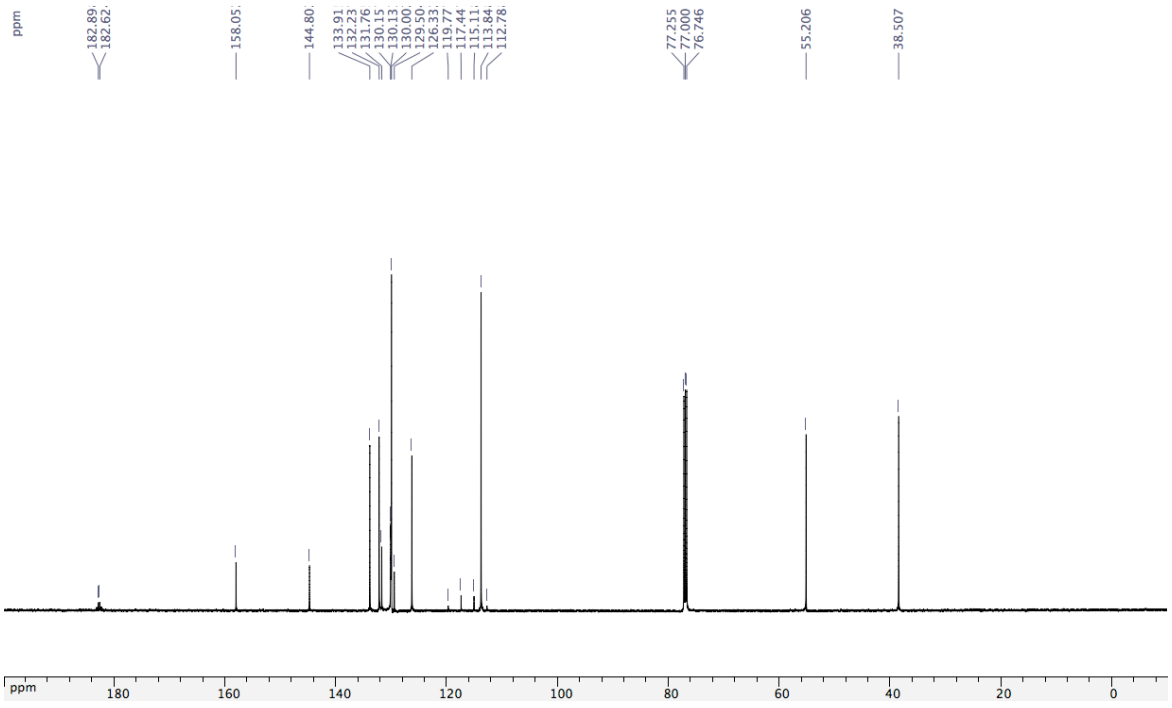
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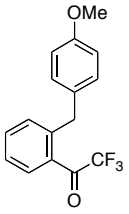
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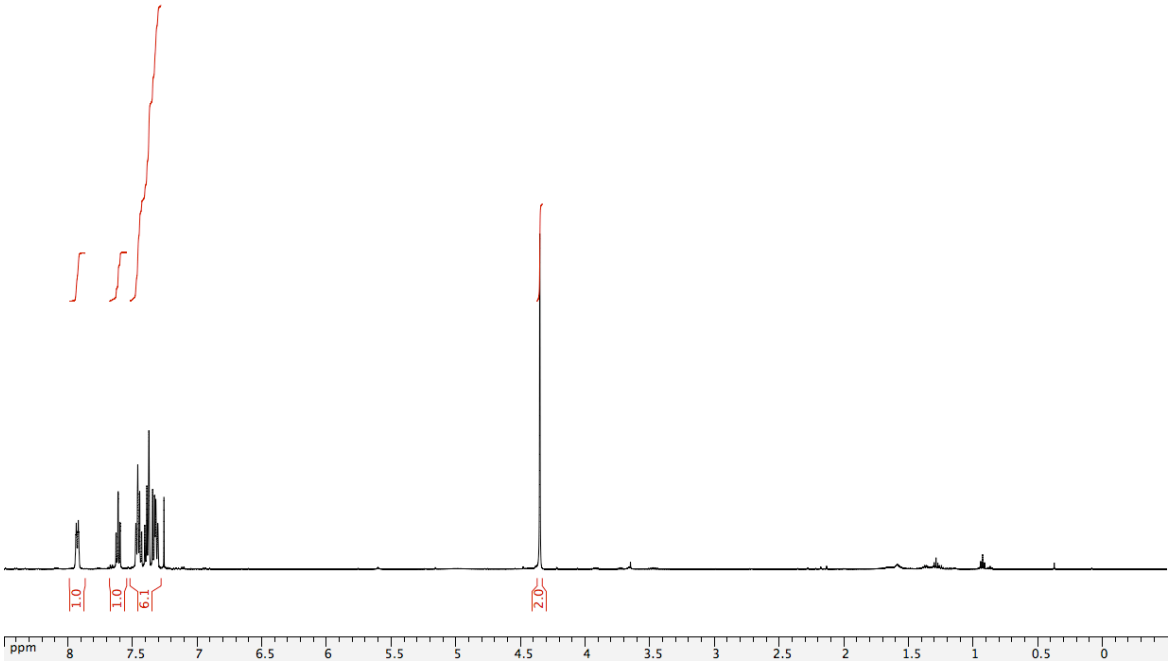
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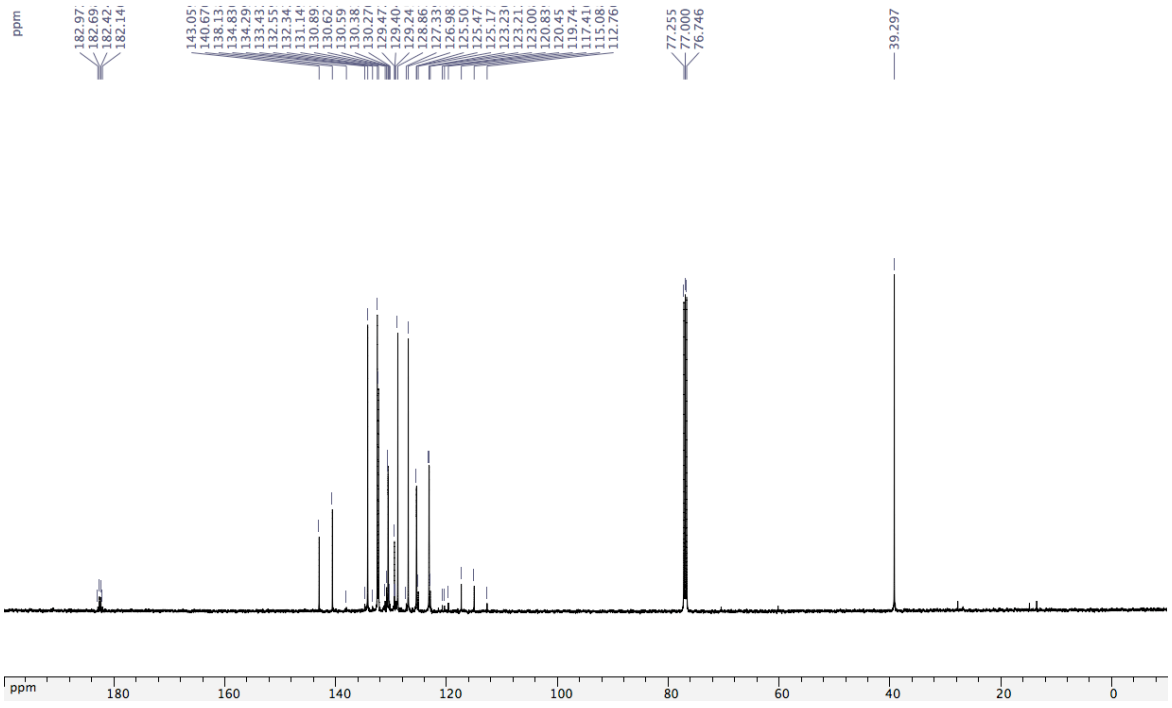
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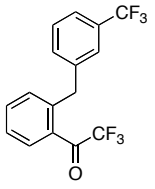
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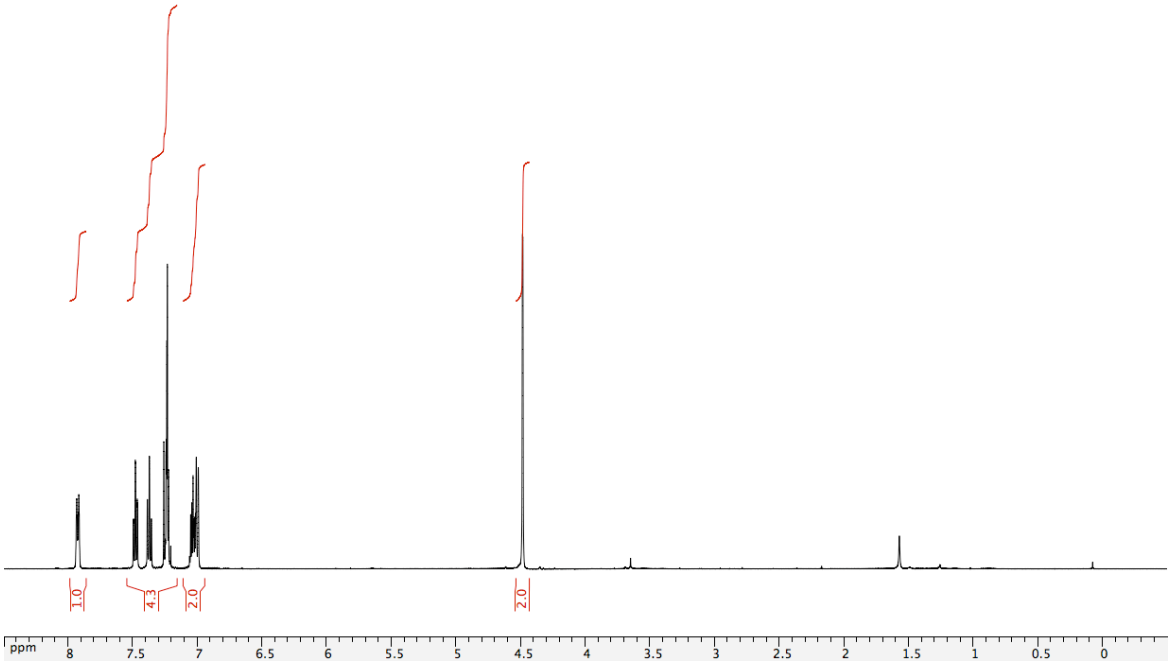
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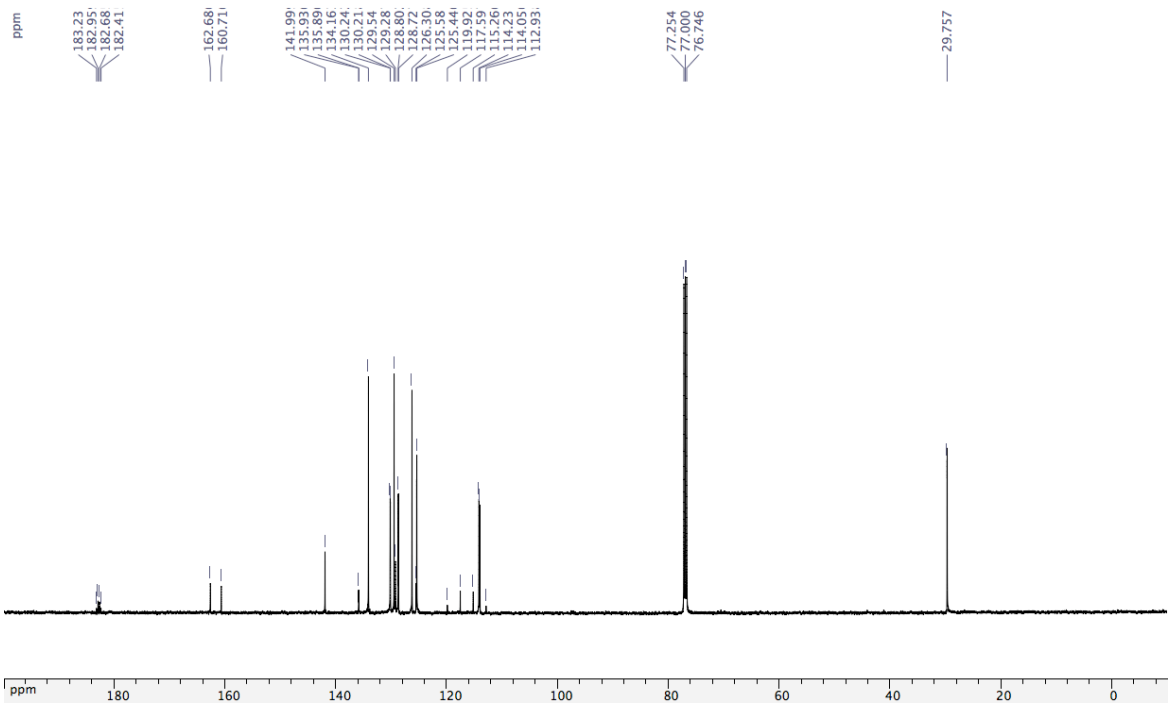
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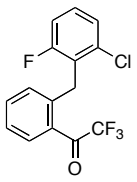
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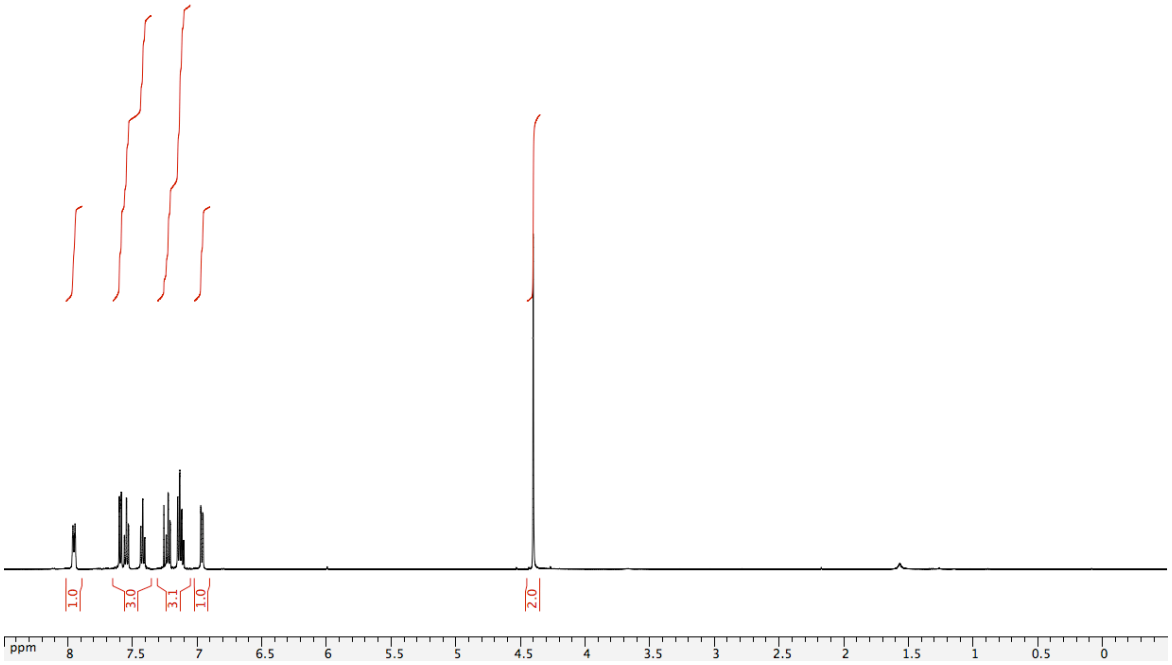
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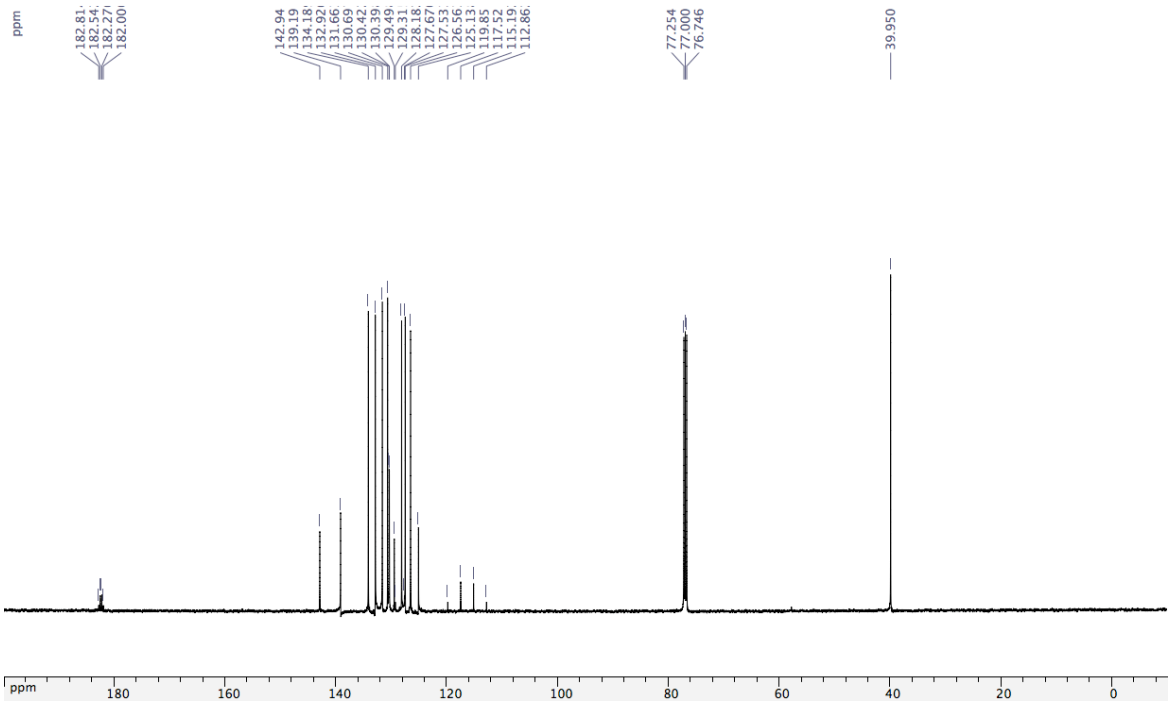
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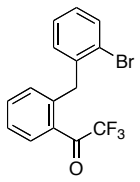
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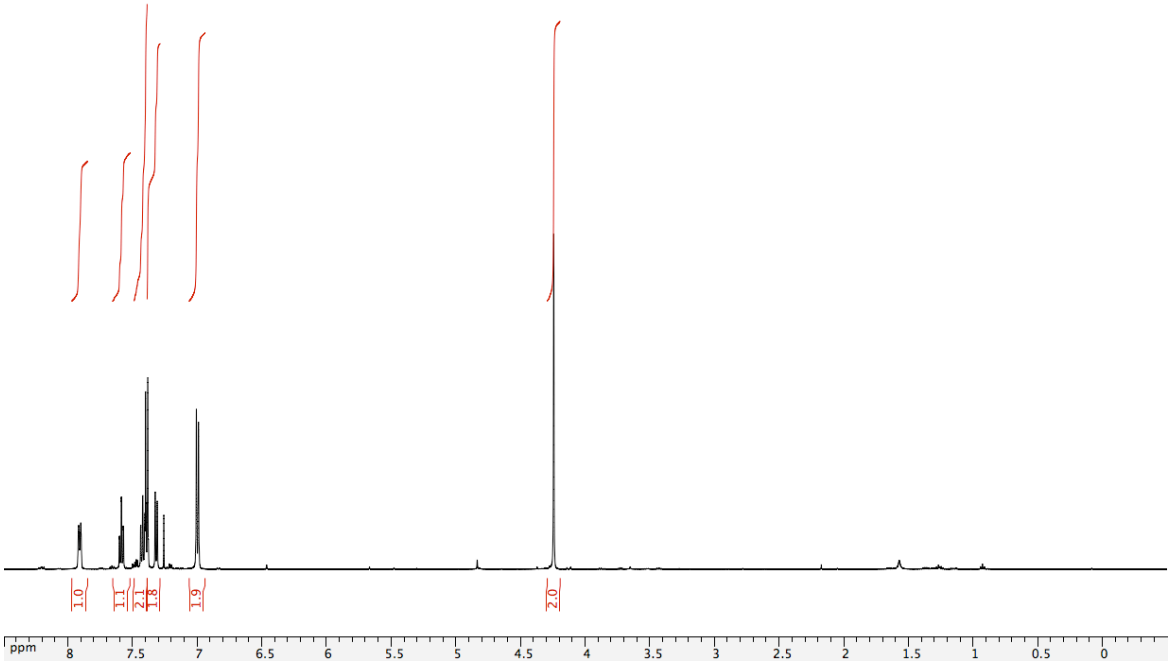
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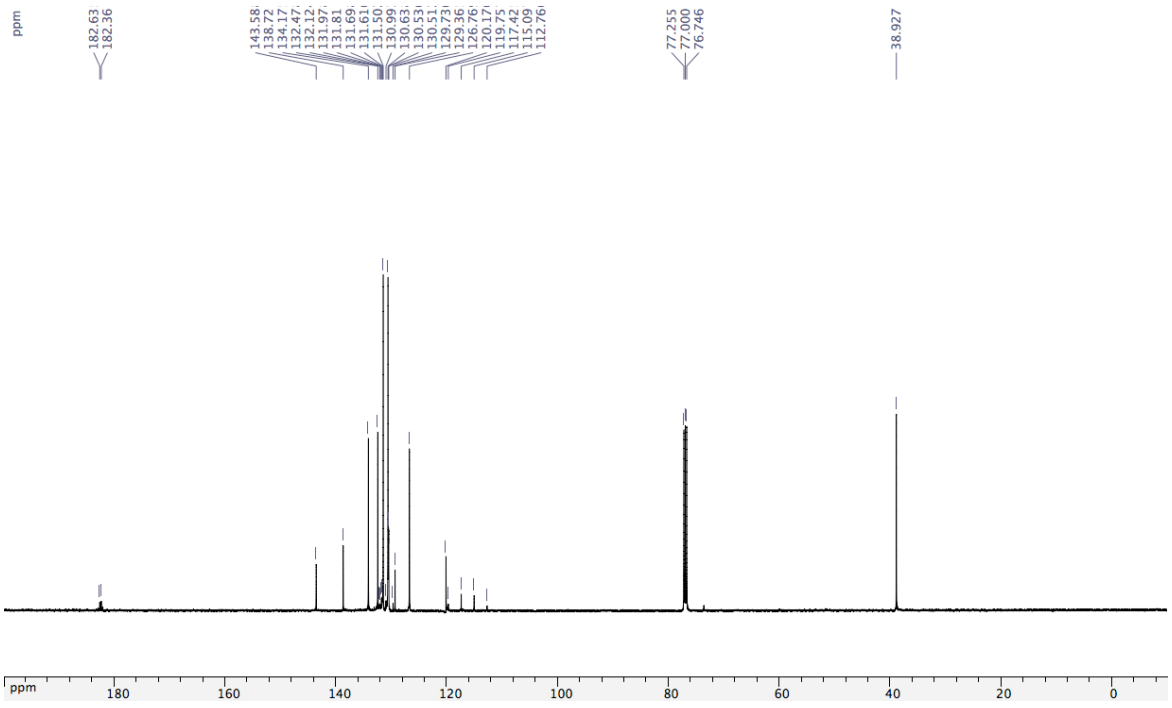
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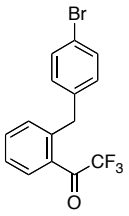
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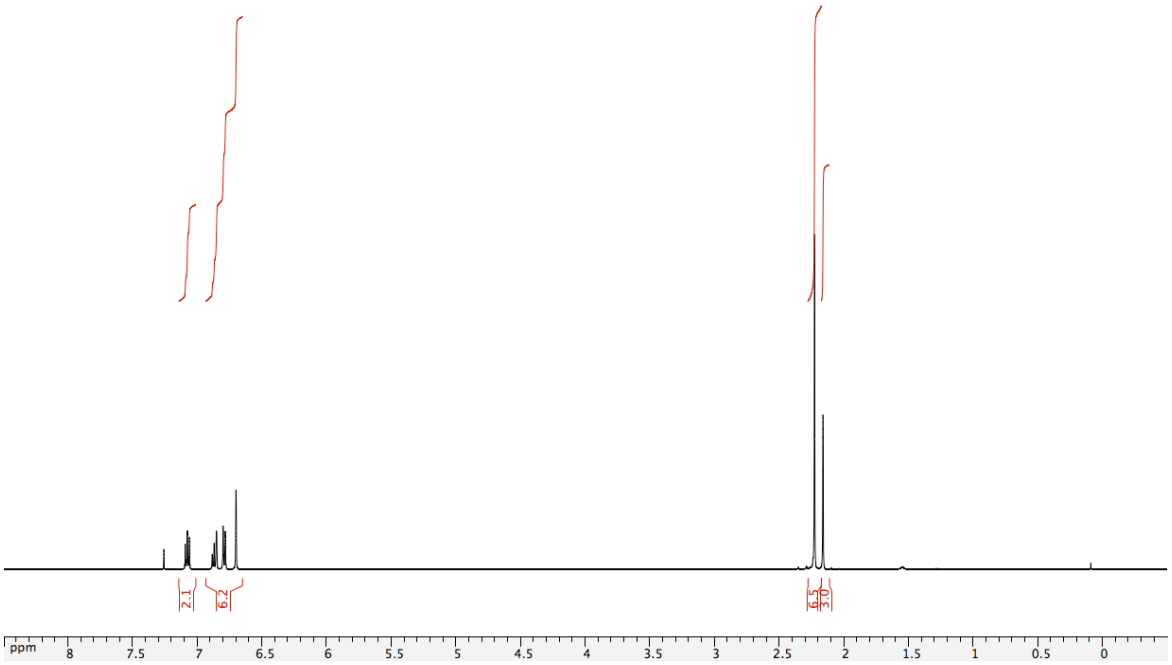
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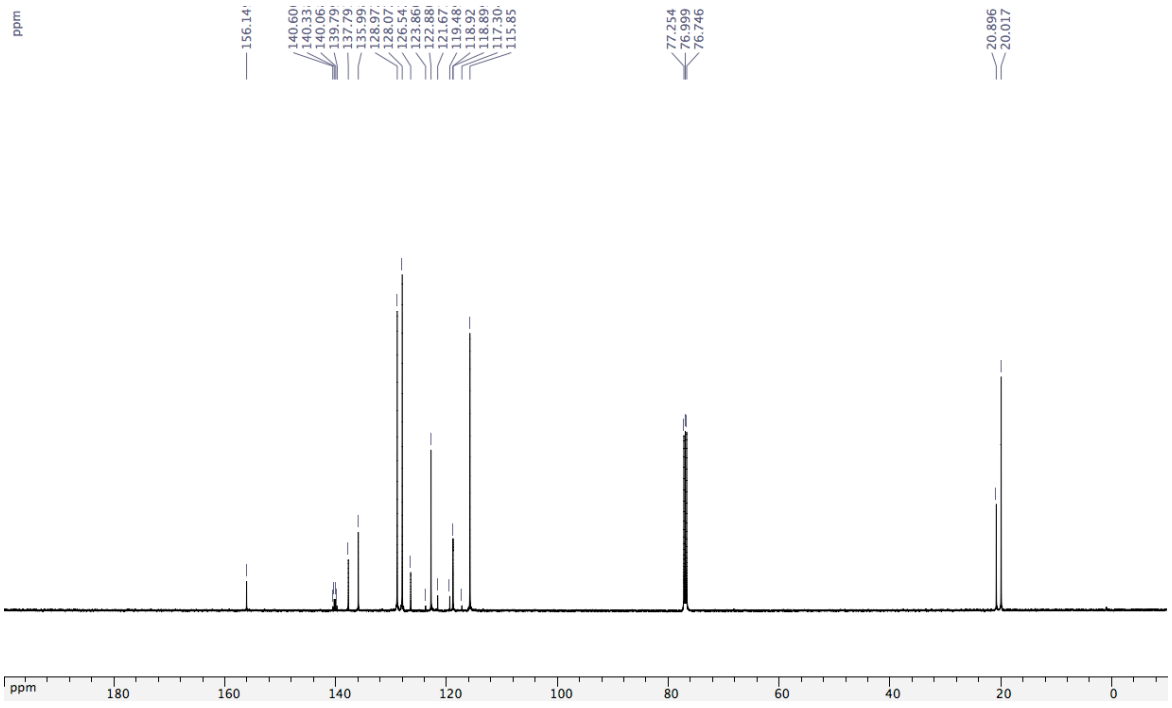
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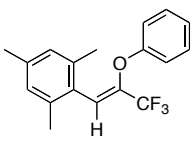
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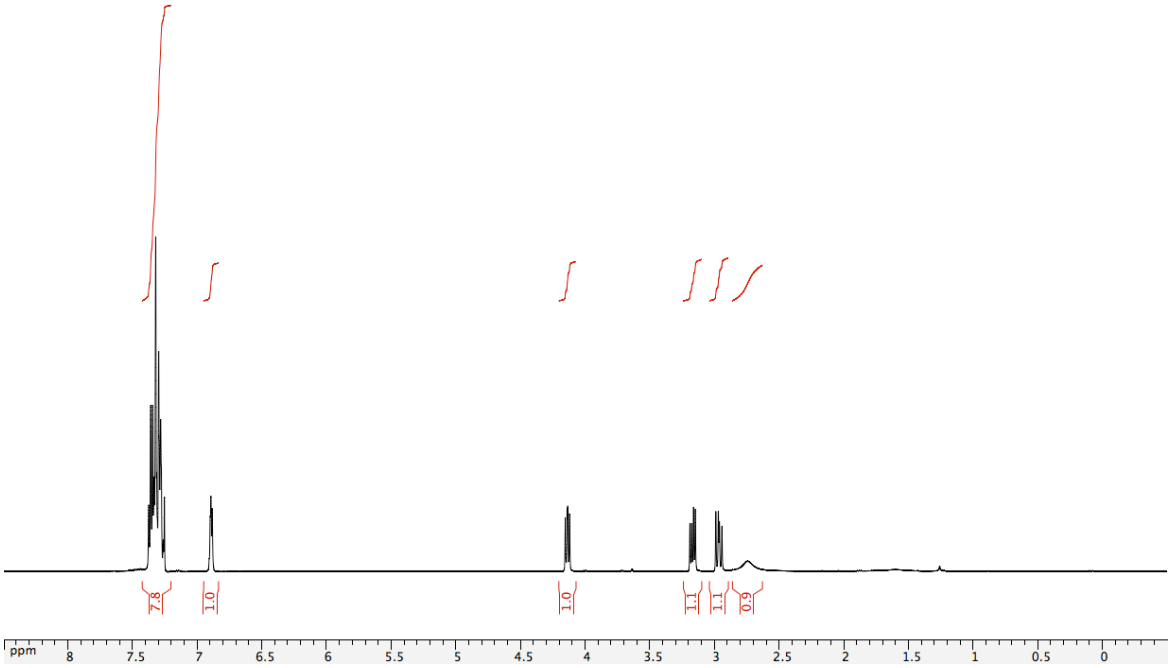
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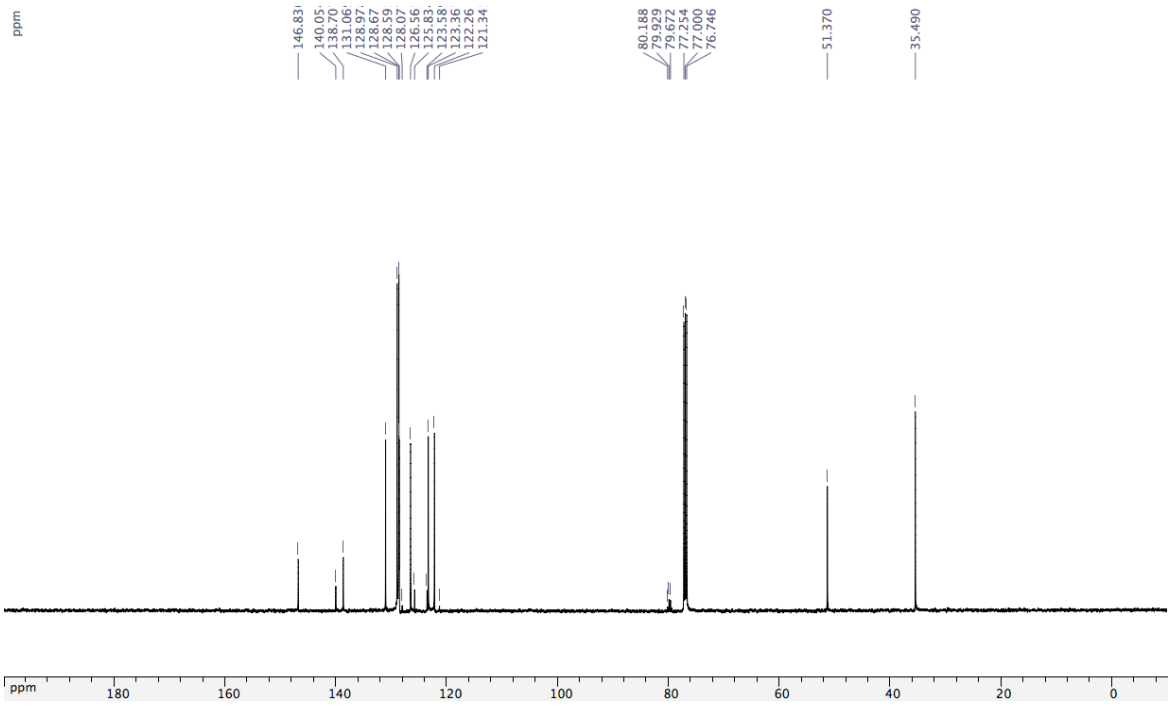
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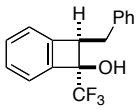
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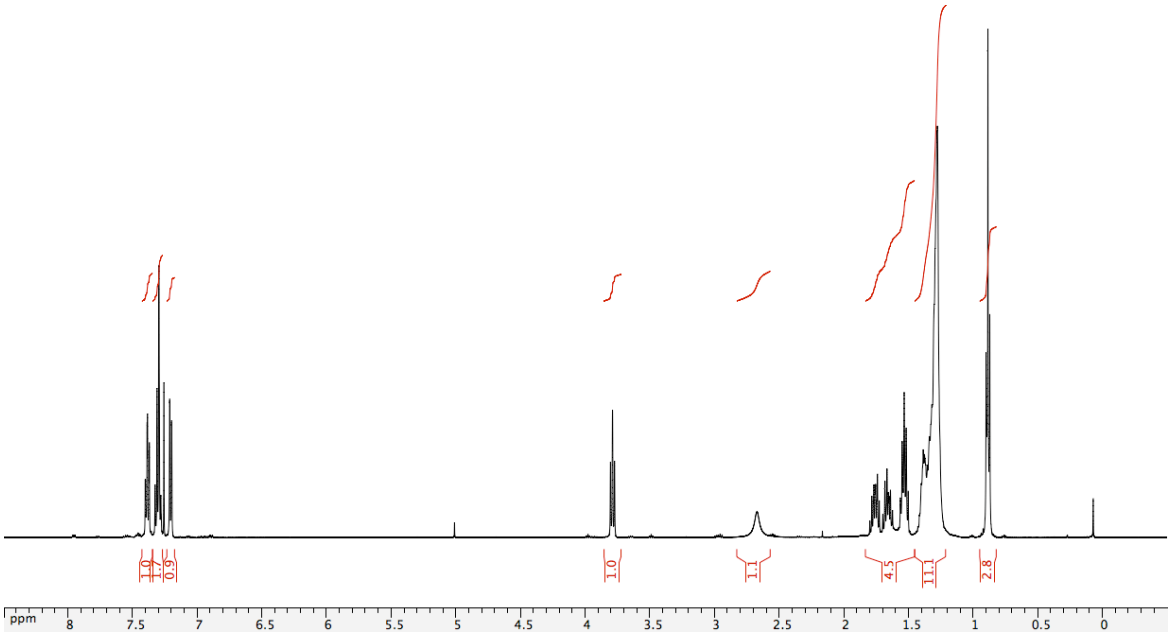
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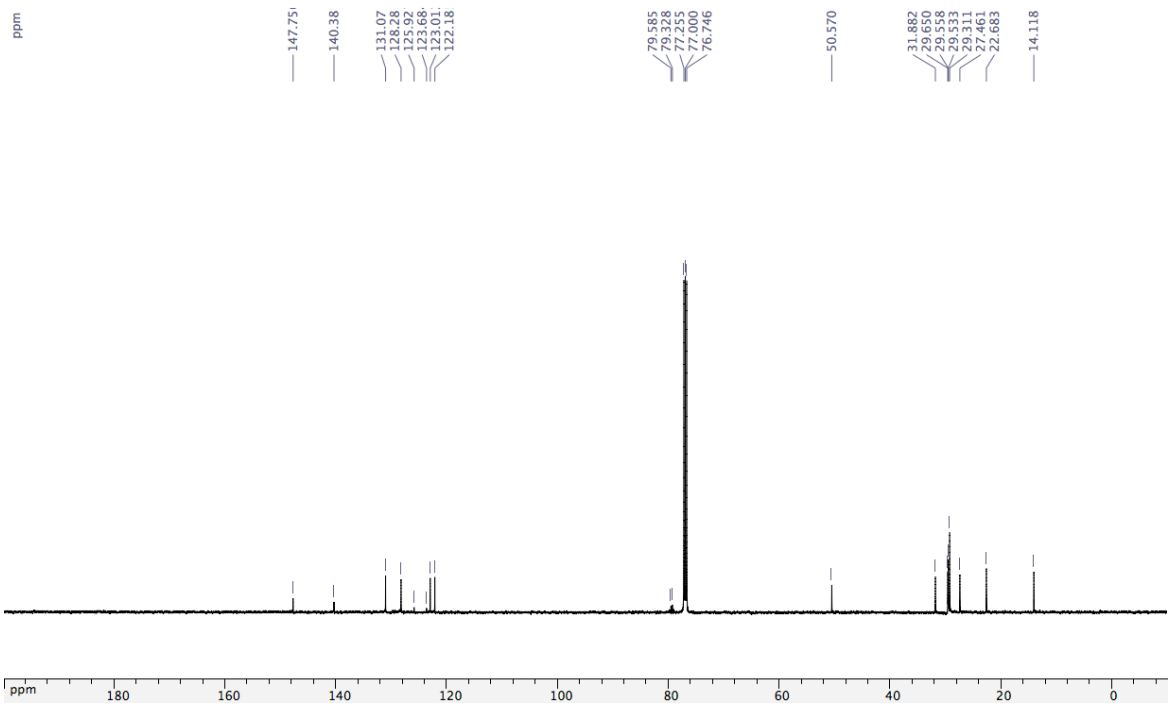
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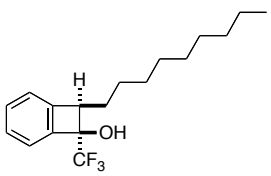
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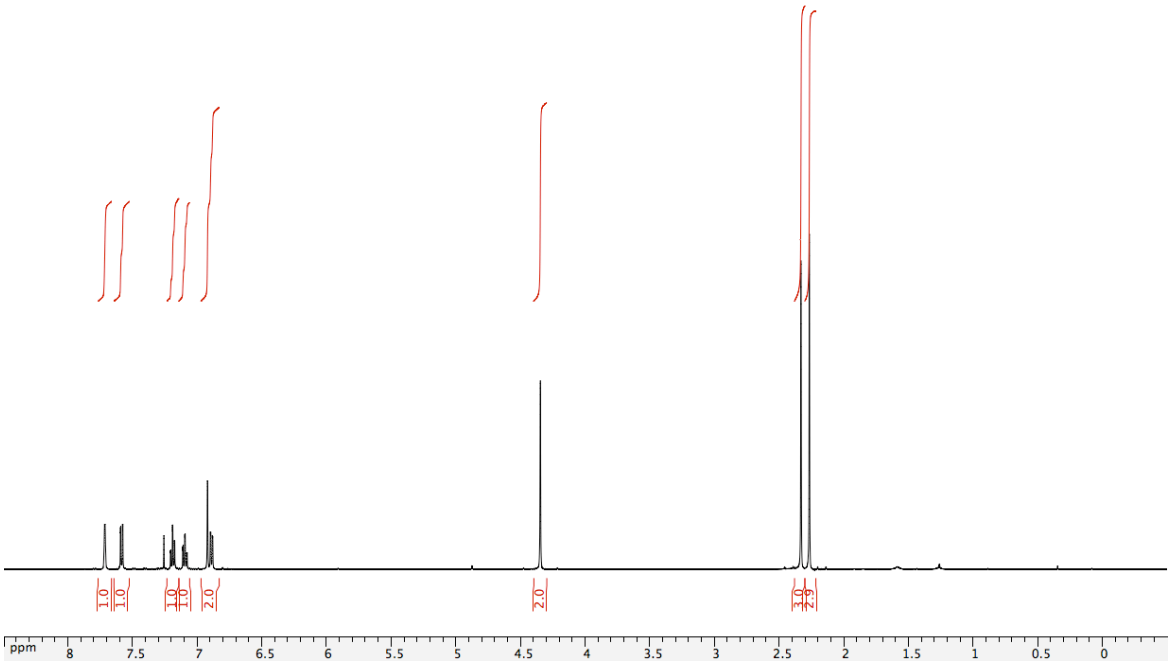
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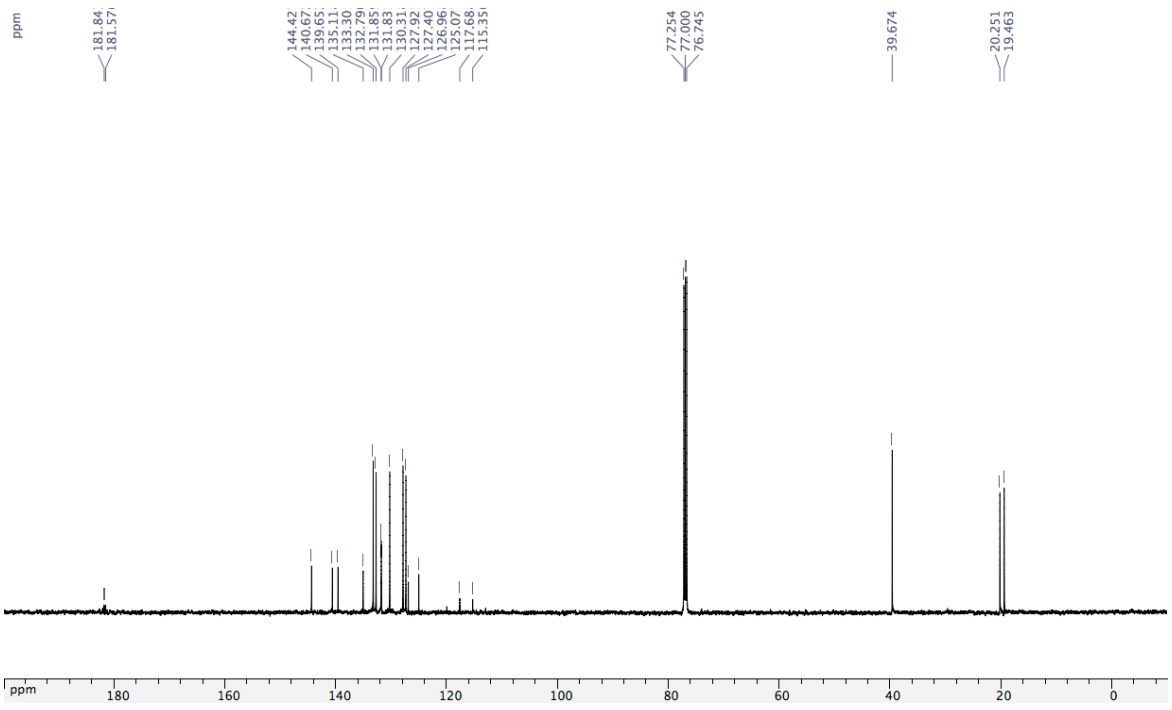
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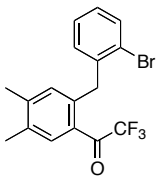
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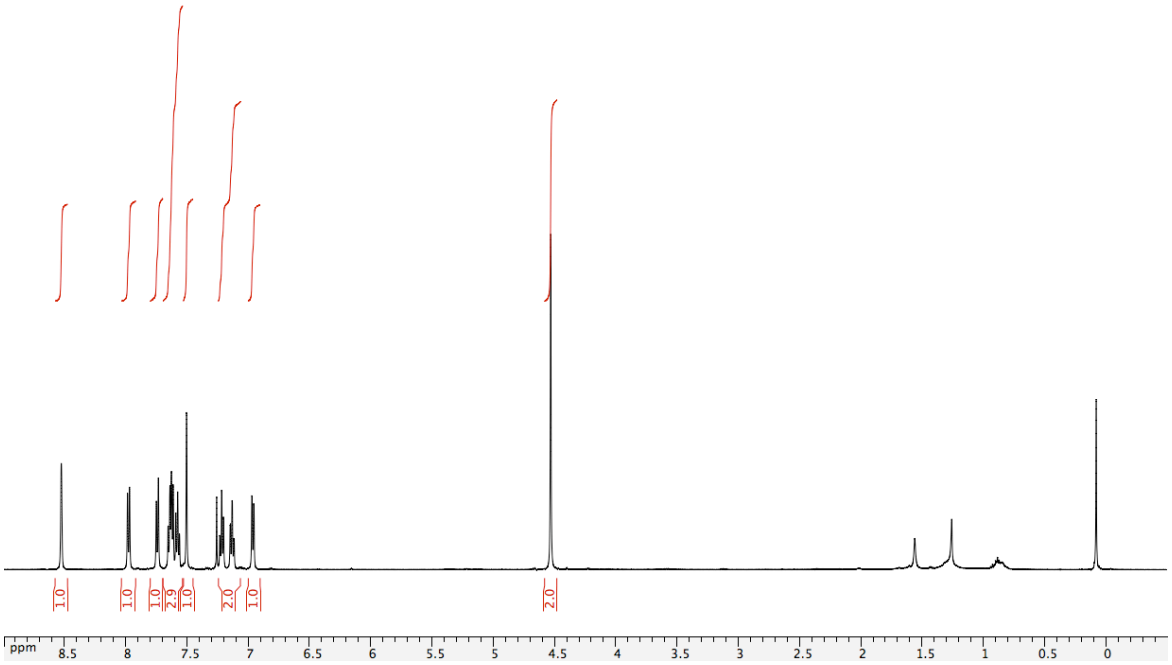
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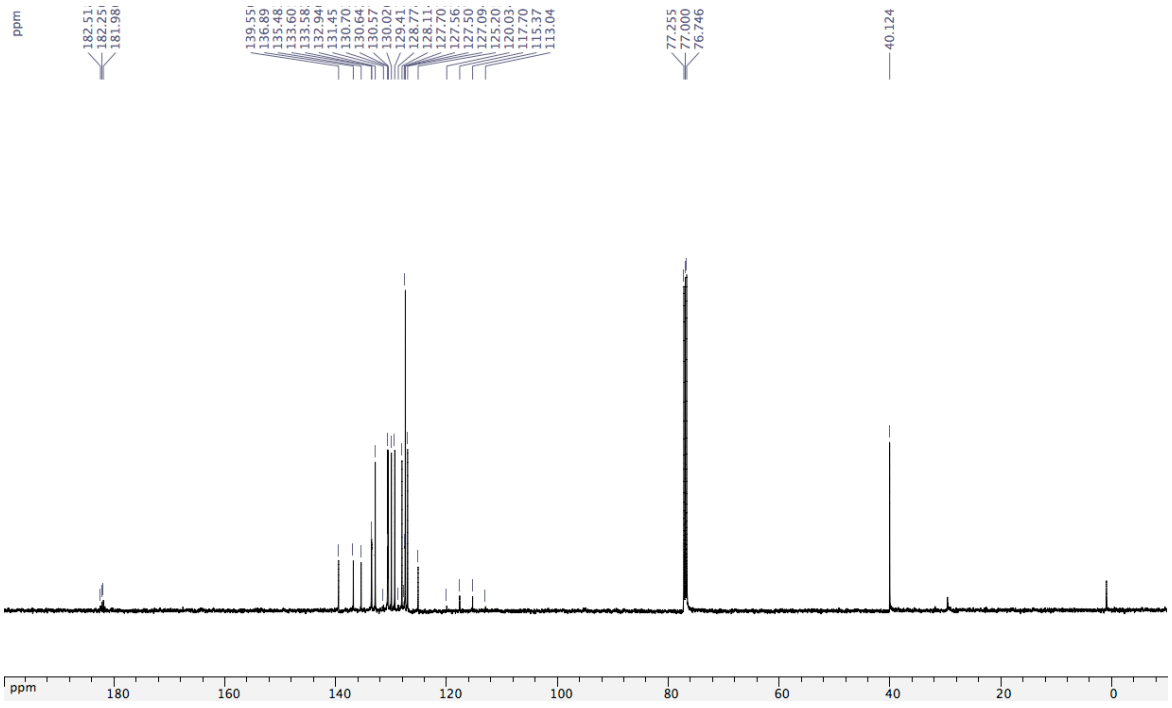
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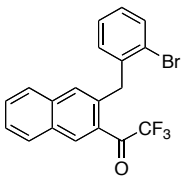
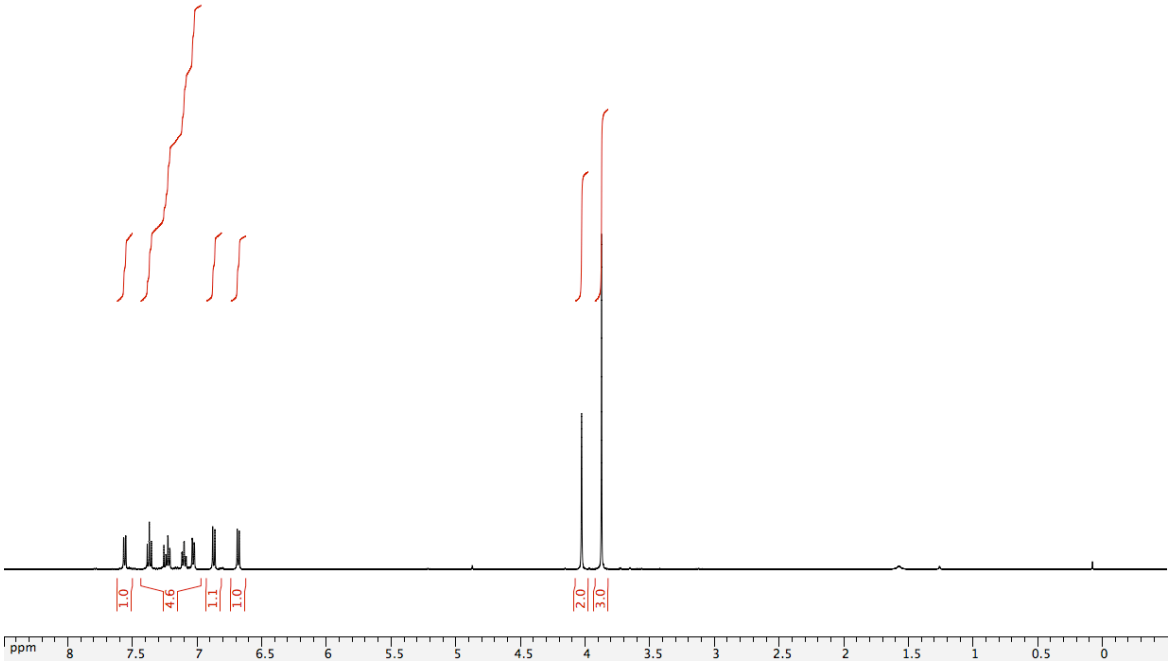

Br
CF3
O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1.1
1.0
2.0
3.0
ppm
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7.5
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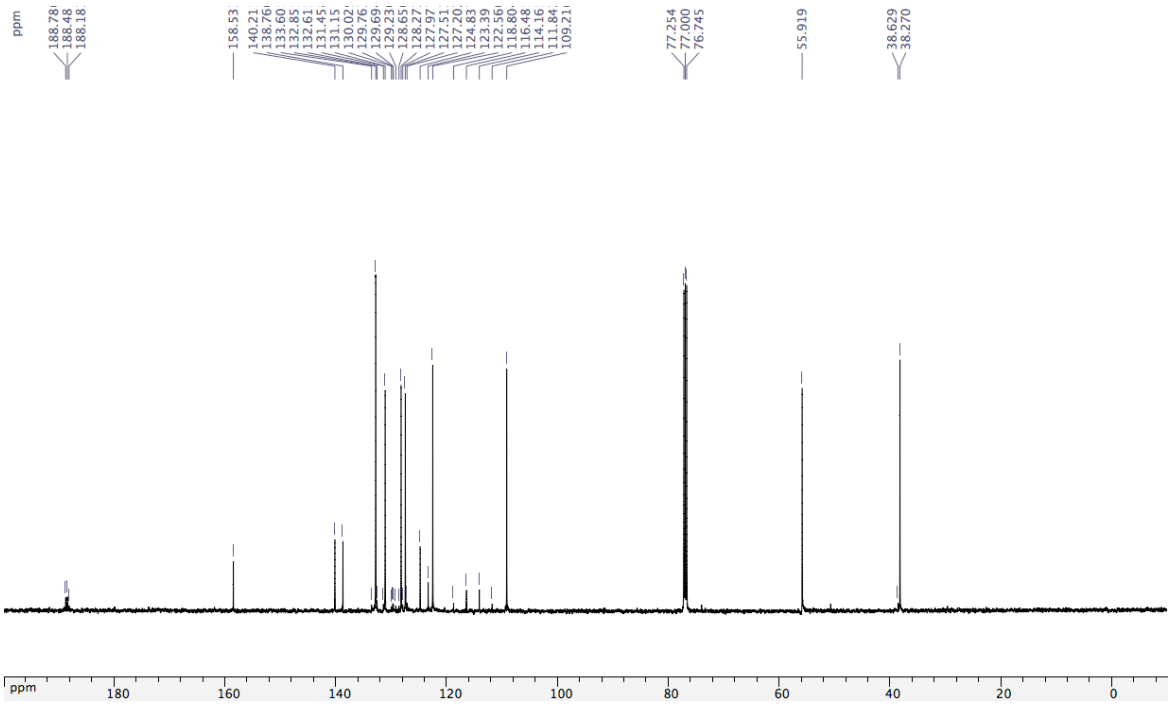
6
5.5
5
4.5
4
3.5
3
2.5
2
1.5
1
0.5
0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158.53
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131.15
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129.76
129.69
129.23
128.65
128.27
127.97
127.51
127.20
124.83
123.39
122.56
118.80
116.48
114.16
111.84
109.21
77.254
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55.919
38.629
38.270
ppm
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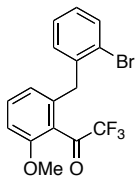
160
140
120
100
80
60
40
20
0


Br
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OMe O