Electronic Supplementary Material (ESI) for Organic Chemistry Frontiers.

Cationic Pd(II)-Catalyzed *N*-Tosyl-Aniline Tethered Alkynyl Ketones Initiated by Hydropalladation of Alkynes: A Facile Way to 1,2-Dihydro or 1,2,3,4-Tetrahydroquinoline Derivatives

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Supporting Information

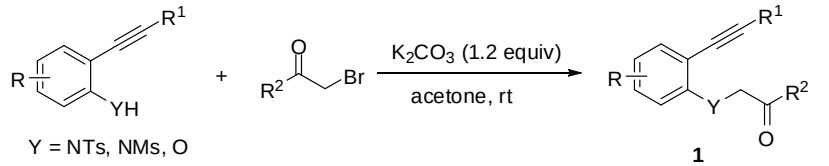
Contents

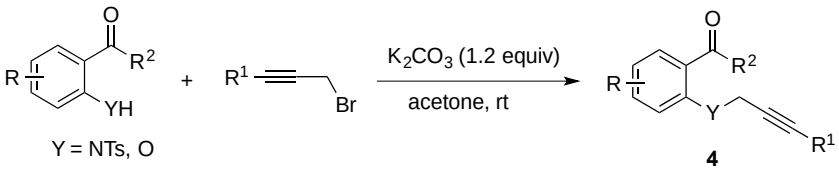
I. General

NMR spectra were recorded on a Varian Mercury V x 400 spectrometer. Infrared spectra were obtained on a Bio-Rad FTS-185 instrument. Mass spectra were provided on Agilent 6224 instruments. [Pd(*rac*-binap)$(H_2O)_2$]$(OTf)_2$,[1] [Pd(*rac*-binap)$(H_2O)_2$]$(BF_4)_2$,[1] [Pd(dppp)$(H_2O)_2$]$(BF_4)_2$[2] and [Pd(*S*,*S*-bdpp)$(H_2O)_2$]$(BF_4)_2$[3] were synthesized according to the literature procedures. Palladium acetate was purchased from ACROS. Palladium trifluoroacetate were purchased from Alfa Aesar. All melting points were uncorrected. All solvents were dried and distilled before use according to the standard procedure.

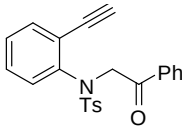
II. Synthesis of substrates

General procedure for the synthesis of substrates 1 and 4.





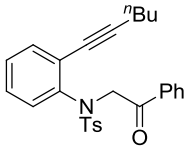
Substrates **1** and **4** were synthesized according to the literature with appropriate modification.[4]



1a

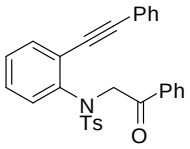
m.p. 136.7-137.8 ^{o}C. ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.91 (d, J=7.6 Hz, 2H), 7.68-7.52 (m, 4H), 7.48-7.38 (m, 3H), 7.33 (t, J=7.2 Hz, 1H), 7.29-7.20 (m, 3H), 5.26 (s, 2H), 3.02 (s, 1H), 2.42 (s, 3H). ^{13}C NMR (100 MHz, $CDCl_3$): δ 193.5, 143.4, 140.8, 136.9, 134.8, 133.8, 133.5, 133.0, 129.4, 129.3, 128.6, 128.2, 127.9, 127.8, 122.1, 82.2, 79.8, 56.5, 21.5. IR (KBr): 3277, 2964, 2914, 1698, 1596, 1485, 1445, 1339, 1222, 1164, 1120, 1097, 980, 888, 875, 752, 669, 580, 557, 536 cm^{-1}. MS (*m/z*, ESI):

390 $[M+H]^+$. HRMS-ESI calcd for $C_{23}H_{20}NO_3S$: 390.1158. Found: 390.1151.



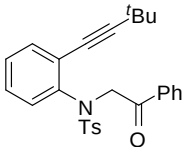
1b

m.p. 124.5-125.3 °C. ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.91-7.86 (m, 2H), 7.67 (dd, J=8.0, 1.2 Hz, 1H), 7.63-7.59 (m, 2H), 7.58-7.52 (m, 1H), 7.43 (t, J=7.6 Hz, 2H), 7.33-7.16 (m, 5H), 5.24 (s, 2H), 2.41 (s, 3H), 2.19 (t, J=6.8 Hz, 2H), 1.52-1.37 (m, 4H), 0.96 (t, J=7.2 Hz, 3H). ^{13}C NMR (100 MHz, $CDCl_3$): δ 193.5, 143.0, 139.8, 137.3, 134.9, 133.4, 133.2, 133.1, 129.2, 128.6, 128.1, 128.0, 127.8, 127.6, 123.4, 95.9, 76.9, 56.1, 30.5, 22.0, 21.4, 19.1, 13.6. IR (KBr): 2955, 2928, 2859, 2225, 1704, 1596, 1486, 1449, 1339, 1216, 1163, 1124, 1091, 980, 815, 757, 731, 666, 579, 546 cm^{-1}. MS (*m/z*, ESI): 446 $[M+H]^+$. HRMS-ESI calcd for $C_{27}H_{27}NO_3S$: 445.1712. Found: 445.1720.



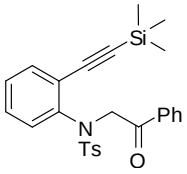
1c

m.p. 108.5-109.4 °C. ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.89 (d, J=7.6 Hz, 2H), 7.75 (d, J=7.6 Hz, 1H), 7.62 (d, J=8.0 Hz, 2H), 7.53 (t, J=7.4 Hz, 1H), 7.49-7.22 (m, 10H), 7.10 (d, J=8.0 Hz, 2H), 5.31 (s, 2H), 2.23 (s, 3H). ^{13}C NMR (100 MHz, $CDCl_3$): δ 193.5, 143.3, 140.0, 137.0, 134.8, 133.5, 133.4, 133.1, 131.3, 129.3, 128.9, 128.6, 128.2, 127.9, 127.5, 122.7, 122.5, 94.4, 85.7, 56.1, 21.2. IR (KBr): 3055, 2925, 2218, 1702, 1596, 1495, 1448, 1343, 1218, 1162, 1126, 980, 838, 758, 751, 598 cm^{-1}. MS (*m/z*, ESI): 466 $[M+H]^+$. HRMS-ESI calcd for $C_{29}H_{23}NO_3S$: 465.1399. Found: 465.1415.



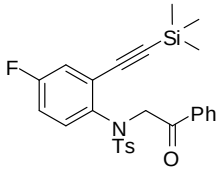
1d

m.p. 116.4-117.2 °C. ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.90-7.85 (m, 2H), 7.71 (dd, J=8.0, 1.2 Hz, 1H), 7.63-7.53 (m, 3H), 7.44 (t, J=8.0 Hz, 2H), 7.32 (dd, J=7.4, 1.8 Hz, 1H), 7.29-7.17 (m, 4H), 5.30 (s, 2H), 2.40 (s, 3H), 1,23 (s, 9H). ^{13}C NMR (100 MHz, $CDCl_3$): δ 193.5, 143.1, 139.8, 137.5, 134.9, 133.4, 133.2, 132.9, 129.3, 128.6, 128.1, 127.9, 127.7, 127.6, 123.2, 103.9, 75.8, 55.9, 30.6, 27.9, 21.4. IR (KBr): 2966, 2918, 2868, 2234, 1703, 1596, 1488, 1474, 1448, 1350, 1224, 1160, 1120, 1105, 1091, 1010, 980, 868, 754, 725, 581, 547 cm^{-1}. MS (*m/z*, ESI): 446 $[M+H]^+$. HRMS-ESI calcd for $C_{27}H_{27}NO_3S$: 445.1712. Found: 445.1707.



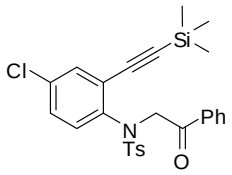
1e

m.p. 90.0-91.1 °C. ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.91-7.85 (m, 2H), 7.78 (dd, J=8.0, 0.8 Hz, 1H), 7.61-7.54 (m, 3H), 7.45 (t, J=7.6 Hz, 2H), 7.38 (dd, J=7.6, 1.4 Hz, 1H), 7.33 (td, J=8.0, 2.0 Hz, 1H), 7.26-7.20 (m, 3H), 5.31 (s, 2H), 2.41 (s, 3H), 0.20 (s, 9H). ^{13}C NMR (100 MHz, $CDCl_3$): δ 193.5, 143.2, 140.5, 137.2, 134.9, 133.6, 133.5, 133.3, 129.3, 129.2, 128.7, 127.9, 127.8, 127.6, 122.2, 101.3, 100.1, 56.0, 21.5, -0.2. IR (KBr): 2958, 2925, 2158, 1705, 1597, 1481, 1448, 1352, 1249, 1223, 1163, 1120, 1092, 981, 859, 752, 721, 578, 543 cm^{-1}. MS (*m/z*, ESI): 462 $[M+H]^+$. HRMS-ESI calcd for $C_{26}H_{28}NO_3SSi$: 462.1554. Found: 462.1547.



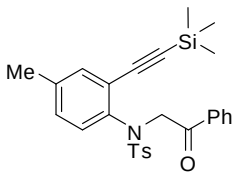
1f

m.p. 106.0-106.6 ^{o}C. ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.88 (d, J=8.0 Hz, 2H), 7.84-7.74 (m, 1H), 7.65-7.52 (m, 3H), 7.50-7.40 (m, 2H), 7.23 (d, J=7.6 Hz, 2H), 7.10-6.98 (m, 2H), 5.29 (s, 2H), 2.41 (s, 3H), 0.21 (s, 9H). ^{13}C NMR (100 MHz, $CDCl_3$): δ 193.2, 161.1 (d, J=247.8 Hz), 143.2, 136.9, 136.4 (d, J=1.9 Hz), 135.2 (d, J=9.1 Hz), 134.6, 133.5, 129.3, 128.6, 127.6, 127.4, 124.0 (d, J=9.9 Hz), 119.6 (d, J=23.9 Hz), 116.3 (d, J=22.4 Hz), 101.3, 99.8, 55.8, 21.4, -0.4. ^{19}F NMR (376 MHz, $CDCl_3$): δ -112.5 (m, 1F). IR (KBr): 2959, 2925, 2156, 1705, 1598, 1579, 1488, 1449, 1410, 1354, 1250, 1224, 1205, 1163, 1119, 1093, 981, 960, 849, 752, 711, 668, 568, 547 cm^{-1}. MS (*m/z*, ESI): 497 $[M+NH_4]^+$. HRMS-ESI calcd for $C_{26}H_{26}FNO_3SSi$: 479.1387. Found: 479.1394.



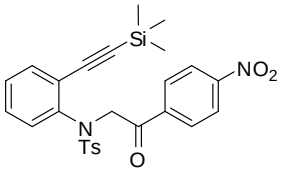
1g

m.p. 99.8-100.6 ^{o}C. ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.86 (d, J=7.6 Hz, 2H), 7.75 (d, J=8.8 Hz, 1H), 7.70-7.50 (m, 3H), 7.49-7.38 (m, 2H), 7.38-7.16 (m, 4H), 5.29 (s, 2H), 2.40 (s, 3H), 0.19 (s, 9H). ^{13}C NMR (100 MHz, $CDCl_3$): δ 193.0, 143.3, 138.8, 136.8, 134.6, 134.5, 133.6, 133.5, 132.8, 129.3, 129.1, 128.6, 127.6, 127.3, 123.6, 101.6, 99.6, 55.6, 21.4, -0.5. IR (KBr): 3065, 2959, 2925, 2160, 1705, 1597, 1481, 1449, 1393, 1354, 1249, 1226, 1164, 1109, 1091, 981, 903, 866, 846, 753, 724, 688, 666, 564, 547 cm^{-1}. MS (*m/z*, ESI): 513 $[M+NH_4]^+$. HRMS-ESI calcd for $C_{26}H_{26}ClNO_3SSi$: 495.1091. Found: 495.1098.



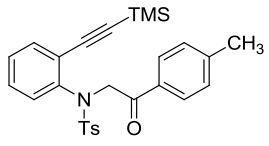
1h

m.p. 72.4-73.4 °C. ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.85 (d, J=7.6 Hz, 2H), 7.67 (d, J=8.0 Hz, 1H), 7.58 (d, J=8.4 Hz, 2H), 7.50 (t, J=7.4 Hz, 1H), 7.41 (t, J=7.6 Hz, 2H), 7.22-7.15 (m, 3H), 7.11 (d, J=8.4 Hz, 1H), 5.29 (s, 2H), 2.36 (s, 3H), 2.23 (s, 3H), 0.19 (s, 9H). ^{13}C NMR (100 MHz, $CDCl_3$): δ 193.1, 142.8, 137.7, 137.5, 137.0, 134.6, 133.6, 133.2, 132.7, 129.7, 129.1, 128.3, 127.4, 127.2, 121.5, 101.2, 99.3, 55.7, 21.2, 20.4, -0.5. IR (KBr): 2959, 2924, 2154, 1705, 1598, 1490, 1449, 1351, 1249, 1216, 1163, 1105, 1092, 911, 869, 846, 751, 733, 689, 668, 589, 547 cm^{-1}. MS (*m/z*, ESI): 476 $[M+H]^+$. HRMS-ESI calcd for $C_{27}H_{29}NO_3SSi$: 475.1637. Found: 475.1647.



1i

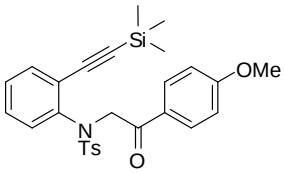
m.p. 142.6-144.0 °C. ^{1}H NMR (400 MHz, $CDCl_3$): δ 8.28 (d, J=8.0 Hz, 2H), 8.02 (d, J=7.6 Hz, 2H), 7.69 (d, J=7.2 Hz, 1H), 7.53 (d, J=7.2 Hz, 2H), 7.42-7.16 (m, 5H), 5.26 (s, 2H), 2.41 (s, 3H), 0.17 (s, 9H). ^{13}C NMR (100 MHz, $CDCl_3$): δ 192.6, 150.3, 139.9, 139.5, 136.4, 133.7, 132.9, 129.4, 129.2, 128.9,128.1, 127.5, 123.8, 122.0, 101.0, 100.4, 56.4, 21.5, -0.3. IR (KBr): 2964, 2936, 2156, 1708, 1600, 1521, 1482, 1445, 1344, 1334, 1319, 1249, 1217, 1205, 1160, 1123, 1102, 1093, 986, 854, 814, 761, 747, 689, 658, 580, 544 cm^{-1}. MS (*m/z*, ESI): 507 $[M+H]^+$. HRMS-ESI calcd for $C_{26}H_{26}N_2O_5SSi$: 506.1332. Found: 506.1351.



1j

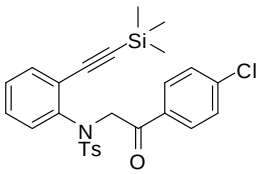
Colorless crystal, m. p. 109-110 °C. ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.80-7.77 (m, 3H), 7.58 (d, *J* = 8.0 Hz, 2H), 7.37 (dd, *J* = 7.6, 1.2 Hz, 1H), 7.33 (td, *J* = 7.6, 2.0 Hz, 1H), 7.25-7.20 (m, 5H), 5.29 (s, 2H), 2.40 (s, 3H), 2.39 (s, 3H), 0.20 (s, 9H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 193.0, 144.3, 143.1, 140.5, 137.2, 133.5, 133.3, 132.4,

129.3, 129.1, 127.9, 127.8, 127.6, 122.2, 101.3, 100.0, 55.8, 21.6, 21.5, -0.3; IR (neat): ν 3064, 3032, 2956, 2924, 2855, 2360, 2158, 1701, 1607, 1481 cm^{-1}; HRMS (ESI) calcd for $C_{27}H_{33}N_2O_3SSi$ $[M+NH_4]^+$: 493.1976, Found: 493.1977.



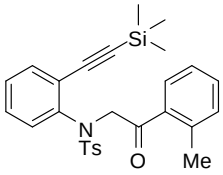
1k

m.p. 111.5-112.4 ^oC. ^1H NMR (400 MHz, $CDCl_3$): δ 7.87 (d, J=6.8 Hz, 2H), 7.77 (d, J=6.8 Hz, 1H), 7.58 (d, J=6.4 Hz, 2H), 7.42-7.18 (m, 5H), 6.91 (d, J=6.8 Hz, 2H), 5.26 (s, 2H), 3.85 (s, 3H), 2.40 (s, 3H), 0.20 (s, 9H). ^{13}C NMR (100 MHz, $CDCl_3$): δ 191.8, 163.7, 143.1, 140.5, 137.3, 133.4, 133.2, 130.0, 129.3, 129.1, 127.9, 127.6, 122.3, 113.8, 101.3, 100.0, 55.6, 55.4, 21.5, -0.3. IR (KBr): 2969, 2920, 2153, 1686, 1603, 1575, 1509, 1484, 1447, 1421, 1340, 1249, 1233, 1177, 1155, 1125, 1098, 1037, 984, 835, 757, 720, 654, 578, 548, 533 cm^{-1}. MS (*m/z*, ESI): 492 $[M+H]^+$. HRMS-ESI calcd for $C_{27}H_{29}NO_4SSi$: 491.1587. Found: 491.1598.



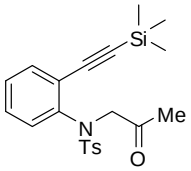
1l

m.p. 161.1-162.1 ^oC. ^1H NMR (400 MHz, $CDCl_3$): δ 7.82 (d, J=8.8 Hz, 2H), 7.74 (dd, J=8.4, 1.2 Hz, 1H), 7.53 (d, J=8.4 Hz, 2H), 7.42 (d, J=8.4 Hz, 2H), 7.40-7.30 (m, 2H), 7.28-7.20 (m, 3H), 5.24 (s, 2H), 2.41 (s, 3H), 0.19 (s, 9H). ^{13}C NMR (100 MHz, $CDCl_3$): δ 192.5, 143.3, 140.2, 139.9, 136.9, 133.6, 133.3, 133.1, 129.4, 129.3, 129.2, 129.0, 128.0, 127.6, 122.2, 101.2, 100.2, 55.9, 21.5, -0.2. IR (KBr): 2964, 2923, 2160, 1701, 1589, 1482, 1448, 1399, 1340, 1249, 1220, 1155, 1125, 1099, 1088, 983, 889, 873, 859, 846, 816, 581, 547, 533 cm^{-1}. MS (*m/z*, ESI): 496 $[M+H]^+$. HRMS-ESI calcd for $C_{26}H_{26}ClNO_3SSi$: 495.1091. Found: 495.1114.



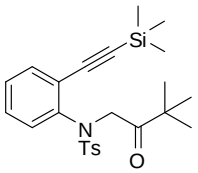
1m

m.p. 69.7-71.0 ^{o}C. ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.66 (dd, J=8.0, 0.8 Hz, 1H), 7.58-7.50 (m, 3H), 7.40-7.28 (m, 3H), 7.26-7.18 (m, 5H), 5.11 (s, 2H), 2.39 (s, 3H), 2.38 (s, 3H), 0.15 (s, 9H). ^{13}C NMR (100 MHz, $CDCl_3$): δ 197.5, 143.1, 140.3, 138.4, 136.9, 135.5, 133.5, 133.1, 131.8, 131.1, 129.2, 128.9, 127.8, 127.7, 127.4, 125.3, 121.8, 101.0, 100.0, 57.7, 21.3, 20.6, -0.4. IR (KBr): 2964, 2934, 2150, 1700, 1599, 1570, 1480, 1445, 1348, 1334, 1250, 1219, 1162, 1116, 1099, 857, 758, 583, 544 cm^{-1}. MS (*m/z*, ESI): 476 $[M+H]^+$. HRMS-ESI calcd for $C_{27}H_{29}NO_3SSi$: 475.1637. Found: 475.1642.



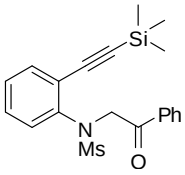
1n

m.p. 56.4-57.2 ^{o}C. ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.60 (dd, J=8.2, 1.0 Hz, 1H), 7.50 (d, J=8.4 Hz, 2H), 7.39 (dd, J=7.6, 1.6 Hz, 1H), 7.33 (td, J=7.6, 1.6 Hz, 1H), 7.28-7.18 (m, 3H), 4.43 (s, 2H), 2.40 (s, 3H), 2.27 (s, 3H), 0.13 (s, 9H). ^{13}C NMR (100 MHz, $CDCl_3$): δ 204.0, 143.4, 140.6, 136.2, 134.0, 132.1, 129.4, 129.2, 128.0, 127.7, 122.3, 101.0, 100.3, 59.7, 27.0, 21.5, -0.3. IR (KBr): 2961, 2926, 2154, 1740, 1597, 1482, 1446, 1413, 1342, 1250, 1160, 934, 862, 842, 726, 657, 586, 546, 535 cm^{-1}. MS (*m/z*, ESI): 400 $[M+H]^+$. HRMS-ESI calcd for $C_{21}H_{25}NO_3SSi$: 399.1324. Found: 399.1343.



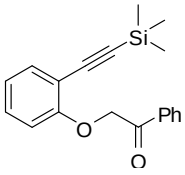
1o

^{1}H NMR (400 MHz, $CDCl_3$): δ 7.76 (dd, J=8.0, 0.8 Hz, 1H), 7.53 (d, J=6.8 Hz, 2H), 7.37-7.29 (m, 2H), 7.24-7.17 (m, 3H), 4.83 (s, 2H), 2.39 (s, 3H), 1.12 (s, 9H), 0.16 (s, 9H). ^{13}C NMR (100 MHz, $CDCl_3$): δ 208.9, 143.0, 140.5, 137.4, 133.4, 133.3, 129.3, 129.1, 127.8, 127.4, 121.9, 54.0, 43.1, 26.1, 21.5, -0.3. IR (neat): 2964, 2158, 1727, 1598, 1480, 1445, 1351, 1249, 1122, 1100, 1059, 1002, 857, 812, 761, 715, 658, 588, 544 cm^{-1}. MS (*m/z*, ESI): 442 $[M+H]^+$. HRMS-ESI calcd for $C_{24}H_{31}NO_3SSi$: 441.1794. Found: 441.1794.



1p

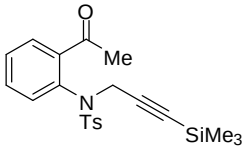
m.p. 99.2-99.7 ^{o}C. ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.88 (d, J=7.6 Hz, 2H), 7.81 (d, J=8.0 Hz, 1H), 7.58 (t, J=7.2 Hz, 1H), 7.54-7.42 (m, 3H), 7.37 (t, J=7.6 Hz, 1H), 7.29 (t, J=7.6 Hz, 1H), 5.26 (s, 2H), 3.14 (s, 3H), 0.33 (s, 9H). ^{13}C NMR (100 MHz, $CDCl_3$): δ 193.5, 140.8, 134.6, 133.6, 133.4, 132.8, 129.6, 128.7, 128.5, 127.7, 122.7, 101.6, 100.9, 56.6, 41.0, -0.2. IR (KBr): 2956, 2932, 2898, 2157, 1712, 1596, 1479, 1449, 1367, 1327, 1249, 1222, 1157, 1126, 1100, 847, 753, 522 cm^{-1}. MS (*m/z*, ESI): 386 $[M+H]^+$. HRMS-ESI calcd for $C_{20}H_{23}NO_3SSi$: 385.1168. Found: 385.1173.



1q

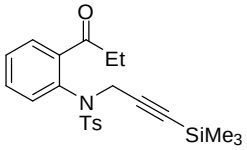
m.p. 74.2-75.2 ^{o}C. ^{1}H NMR (400 MHz, $CDCl_3$): δ 8.04 (d, J=7.6 Hz, 2H), 7.60 (t,

J=7.2 Hz, 1H), 7.48-7.40 (m, 3H), 7.23 (t, J=7.6 Hz, 1H), 6.93 (t, J=7.6 Hz, 1H), 6.83 (d, J=8.4 Hz, 1H), 5.31 (s, 2H), 0.21 (s, 9H). ^{13}C NMR (100 MHz, $CDCl_3$): δ 194.5, 158.8, 134.5, 134.1, 133.7, 129.8, 128.6, 128.4, 121.5, 113.2, 100.8, 99.0, 72.0, -0.1. IR (KBr): 3067, 2958, 2898, 2157, 1705, 1597, 1576, 1488, 1449, 1281, 1249, 1221, 1202, 1119, 968, 866, 842, 751, 689 cm^{-1}. MS (*m/z*, ESI): 309 $[M+H]^+$. HRMS-ESI calcd for $C_{19}H_{20}O_2Si$: 308.1233. Found: 308.1241.



4a

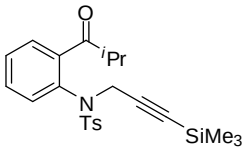
m.p. 77.1-78.5 ^{o}C. ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.64 (d, J=7.6 Hz, 1H), 7.55 (d, J=8.0 Hz, 2H), 7.41 (t, J=7.4 Hz, 1H), 7.32 (t, J=7.4 Hz, 1H), 7.25 (d, J=8.0 Hz, 2H), 6.93 (d, J=7.6 Hz, 1H), 4.54 (s, br, 2H), 2.66 (s, 3H), 2.43 (s, 3H), 0.01 (s, 9H). ^{13}C NMR (100 MHz, $CDCl_3$): δ 200.7, 143.7, 141.7, 136.2, 135.6, 130.9, 129.5, 129.3, 128.8, 128.7, 128.1, 99.5, 91.3, 42.3, 30.1, 21.5, -0.5. IR (KBr): 3068, 2960, 2929, 2184, 1680, 1595, 1486, 1448, 1347, 1278, 1248, 1161, 1090, 1067, 856, 662 cm^{-1}. MS (*m/z*, ESI): 422 $[M+Na]^+$. HRMS-ESI calcd for $C_{21}H_{25}NO_3SSi$: 399.1324. Found: 399.1316.



4b

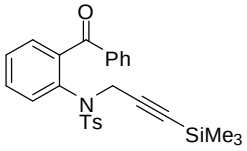
^{1}H NMR (400 MHz, $CDCl_3$): δ 7.58-7.51 (m, 3H), 7.41-7.35 (m, 1H), 7.32-7.19 (m, 3H), 6.94 (d, J=7.6 Hz, 1H), 5.10-4.00 (br, 2H), 2.98 (s, br, 2H), 2.40 (s, 3H), 1.16 (t, J=7.2 Hz, 3H), 0.00 (s, 9H). ^{13}C NMR (100 MHz, $CDCl_3$): δ 204.1, 143.6, 142.1, 135.9, 135.8, 130.4, 129.9, 129.2, 128.7, 128.2, 128.0, 99.8, 91.1, 42.5, 35.3, 21.4, 7.9, -0.6. IR (neat): 2960, 2178, 1698, 1596, 1485, 1445, 1409, 1251, 1163, 1093, 846, 663, 579 cm^{-1}. MS (*m/z*, ESI): 414 $[M+H]^+$. HRMS-ESI calcd for $C_{22}H_{27}NO_3SSi$:

413.1481. Found: 413.1468.



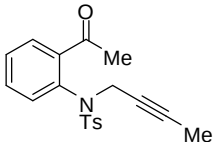
4c

m.p. 104.8-105.1 ^{o}C. ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.60 (d, J=8.0 Hz, 2H), 7.55 (dd, J=8.0, 1.6 Hz, 1H), 7.41 (td, J=7.6, 1.2 Hz, 1H), 7.33-7.24 (m, 3H), 6.92 (dd, J=8.0, 1.2 Hz, 1H), 4.68 (br, 1H), 4.41 (br, 1H), 3.60-3.48 (m, 1H), 2.43 (s, 3H), 1.33-1.10 (br, 6H), 0.0 (s, 9H). ^{13}C NMR (100 MHz, $CDCl_3$): δ 208.1, 143.7, 141.6, 136.2, 135.7, 130.3, 129.3, 128.8, 128.6, 128.3, 99.4, 91.3, 42.4, 39.2, 21.5, -0.5. IR (KBr): 2959, 2930, 2871, 2178, 1683, 1594, 1452, 1351, 1163, 1067, 981, 813, 660, 580 cm^{-1}. MS (*m/z*, ESI): 428 $[M+H]^+$. HRMS-ESI calcd for $C_{23}H_{29}NO_3SSi$: 427.1637. Found: 427.1624.



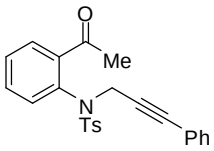
4d

m.p. 111.5-112.3 ^{o}C. ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.77 (dd, J=8.0, 1.6 Hz, 2H), 7.60-7.53 (m, 3H), 7.46-7.37 (m, 5H), 7.32-7.25 (m, 1H), 7.24 (d, J=8.0 Hz, 2H), 4.58 (br, 2H), 2.35 (s, 3H), 0.00 (s, 9H). ^{13}C NMR (100 MHz, $CDCl_3$): δ 195.5, 143.3, 140.9, 137.1, 137.0, 136.6, 133.0, 131.3, 129.5, 129.1, 128.1, 128.0, 100.4, 90.9, 42.8, 21.4, -0.5. IR (KBr): 2960, 2937, 2175, 1656, 1597, 1350, 1291, 1166, 855, 841, 702, 668, 641, 557 cm^{-1}. MS (*m/z*, ESI): 462 $[M+H]^+$. HRMS-ESI calcd for $C_{26}H_{27}NO_3SSi$: 461.1481. Found: 461.1476.



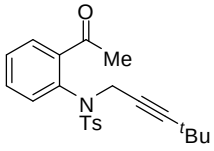
4e

m.p. 112.2-113.2 °C. ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.66 (d, J=8.0 Hz, 1H), 7.52 (d, J=8.0 Hz, 2H), 7.41 (7, J=7.6 Hz, 1H), 7.34 (t, J=7.6 Hz, 1H), 7.30-7.20 (m, 2H), 6.92 (d, J=7.6 Hz, 1H), 4.47 (s, br, 2H), 2.67 (s, 3H), 2.43 (s, 3H), 1.68-1.62 (m, 3H). ^{13}C NMR (100 MHz, $CDCl_3$): δ 100.8, 143.7, 141.6, 136.7, 135.3, 131.1, 129.1, 129.0, 128.9, 128.6, 128.0, 82.1, 72.9, 42.0, 30.1, 21.5, 3.3. IR (KBr): 2964, 2938, 1698, 1596, 1486, 1423, 1345, 1285, 1257, 1159, 1090, 1064, 854, 657, 575, 551 cm^{-1}. MS (*m/z*, ESI): 342 $[M+H]^+$. HRMS-ESI calcd for $C_{19}H_{19}NO_3S$: 341.1086. Found: 341.1071.



4f

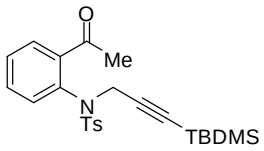
m.p. 96.3-97.2 °C. ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.66 (dd, J=7.6, 1.6 Hz, 1H), 7.58 (d, J=8.0 Hz, 2H), 7.43 (t, J=7.6 Hz, 1H), 7.39-7.14 (m, 8H), 7.03 (d, J=8.0 Hz, 1H), 4.77 (s, br, 2H), 2.69 (s, 3H), 2.39 (s, 3H). ^{13}C NMR (100 MHz, $CDCl_3$): δ 200.8, 143.8, 141.7, 136.4, 135.6, 131.3, 131.1, 129.3, 128.8, 128.7, 128.2, 128.1, 122.1, 85.8, 83.2, 42.3, 30.1, 21.5. IR (KBr): 3055, 1683, 1595, 1489, 1445, 1351, 1288, 1277, 1242, 1163, 1090, 1055, 888, 866, 757, 690, 659, 557, 539 cm^{-1}. MS (*m/z*, ESI): 404 $[M+H]^+$. HRMS-ESI calcd for $C_{24}H_{21}NO_3S$: 403.1242. Found: 403.1228.



4g

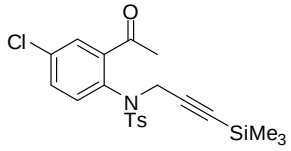
m.p. 66.3-67.5 °C. ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.66 (dd, J=8.0, 1.6 Hz, 1H), 7.54 (d, J=7.6 Hz, 2H), 7.40 (td, J=7.6, 1.2 Hz, 1H), 7.32 (td, J=7.6, 1.6 Hz, 1H), 7.25 (d, J=8.0 Hz, 2H), 6.89 (d, J=8.0 Hz, 1H), 4.49 (s, br, 2H), 2.69 (s, 3H), 2.43 (s, 3H), 1.00 (s, 9H). ^{13}C NMR (100 MHz, $CDCl_3$): δ 200.8, 143.7, 141.7, 136.4, 135.4, 130.8, 129.3, 129.1, 128.8, 128.5, 128.1, 94.8, 72.3, 41.7, 30.3, 30.1, 27.1, 21.4. IR (KBr):

2969, 2929, 2867, 2239, 1693, 1596, 1485, 1446, 1351, 1282, 1265, 1165, 1093, 1037, 864, 833, 740, 672, 575, 547 cm^{-1}. MS (*m/z*, ESI): 384 $[M+H]^+$. HRMS-ESI calcd for $C_{22}H_{25}NO_3S$: 383.1555. Found: 383.1552.



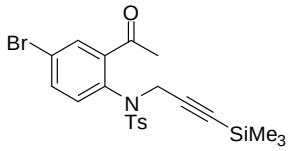
4h

m.p. 87.7-88.5 ^oC. ^1H NMR (400 MHz, $CDCl_3$): δ 7.66 (dd, J=7.6, 1.2 Hz, 1H), 7.52 (d, J=8.4 Hz, 2H), 7.40 (td, J=7.6, 1.2 Hz, 1H), 7.30 (td, J=7.6, 1.2 Hz, 1H), 7.24 (d, J=8.0 Hz, 2H), 6.91 (d, J=8.0 Hz, 1H), 4.57 (s, br, 2H), 2.66 (s, 3H), 2.41 (s, 3H), 0.74 (s, 9H), -0.06 (s, 6H). ^{13}C NMR (100 MHz, $CDCl_3$): δ 200.7, 143.7, 141.7, 136.1, 135.3, 130.9, 129.4, 129.3, 128.9, 128.7, 128.0, 99.8, 89.7, 42.1, 30.0, 25.7, 21.5, 16.2, -5.0. IR (KBr): 2955, 2925, 2883, 2855, 2184, 1683, 1595, 1445, 1348, 1287, 1242, 1163, 1091, 1065, 824, 779, 684, 578 cm^{-1}. MS (*m/z*, ESI): 442 $[M+H]^+$. HRMS-ESI calcd for $C_{24}H_{31}NO_3SSi$: 441.1794. Found: 441.1790.



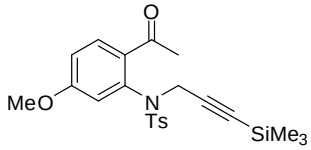
4i

m.p. 98.8-101.0 ^oC. ^1H NMR (400 MHz, $CDCl_3$): δ 7.62 (d, J=2.4 Hz, 1H), 7.55 (d, J=8.8 Hz, 2H), 7.32-7.25 (m, 3H), 6.86 (d, J=8.8, 1H), 4.52 (s, br, 2H), 2.65 (s, 3H), 2.44 (s, 3H), 0.03 (s, 9H). ^{13}C NMR (100 MHz, $CDCl_3$): δ 199.2, 144.0, 143.0, 135.2, 134.8, 134.7, 130.8, 130.7, 129.4, 128.8, 128.1, 99.1, 91.7, 42.2, 29.9, 21.5, -0.6. IR (KBr): 2958, 2921, 2185, 1686, 1355, 1164, 853, 667, 600 cm^{-1}. MS (*m/z*, ESI): 434 $[M+H]^+$. HRMS-ESI calcd for $C_{21}H_{24}ClNO_3SSi$: 433.0935. Found: 433.0933.



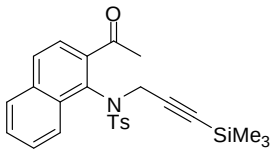
4j

m.p. 105.1-105.8 ^{o}C. ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.74 (s, 1H), 7.53 (d, J=8.0 Hz, 2H), 7.42 (d, J=6.8 Hz, 1H), 7.24 (d, J=7.6 Hz, 2H), 6.77 (d, J=8.4, 1H), 4.50 (s, br, 2H), 2.63 (s, 3H), 2.41 (s, 3H), 0.00 (s, 9H). ^{13}C NMR (100 MHz, $CDCl_3$): δ 199.0, 144.0, 143.3, 135.3, 135.2, 133.8, 131.8, 130.9, 129.4, 128.1, 122.9, 99.1, 91.8, 42.2, 29.9, 21.5, -0.6. IR (KBr): 3072, 2961, 2184, 1690, 1359, 1346, 1164, 1061, 860, 843, 665, 598, 583, 541 cm^{-1}. MS (*m/z*, ESI): 478 $[M+H]^+$. HRMS-ESI calcd for $C_{21}H_{24}BrNO_3SSi$: 477.0430. Found: 477.0426.



4k

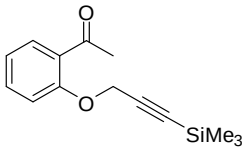
m.p. 108.5-109.5 ^{o}C. ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.65 (d, J=8.8 Hz, 1H), 7.54 (d, J=8.0 Hz, 2H), 7.21 (d, J=8.0 Hz, 2H), 6.86 (dd, J=8.4, 2.4 Hz, 1H), 6.44 (d, J=2.0, 1H), 4.54 (s, br, 2H), 3.61 (s, 3H), 2.56 (s, 3H), 2.37 (s, 3H), -0.03 (s, 9H). ^{13}C NMR (100 MHz, $CDCl_3$): δ 198.8, 161.6, 143.7, 138.4, 135.8, 133.3, 131.1, 129.2, 128.2, 115.3, 114.1, 99.5, 91.2, 55.3, 42.3, 29.8, 21.5, -0.5. IR (KBr): 2964, 2189, 1673, 1600, 1359, 1252, 1163, 1067, 839, 665, 584, 544 cm^{-1}. MS (*m/z*, ESI): 430 $[M+H]^+$. HRMS-ESI calcd for $C_{22}H_{27}NO_4SSi$: 429.1430. Found: 429.1426.



4l

m.p. 98.6-99.6 ^{o}C. ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.86 (d, J=8.4 Hz, 1H), 7.78 (t, J=7.4 Hz, 2H), 7.59 (d, J=8.4 Hz, 1H), 7.52 (d, J=8.4 Hz, 2H), 7.43 (t, J=7.6 Hz, 1H), 7.22-7.10 (m, 3H), 5.06 (d, J=18.4 Hz, 1H), 4.37 (d, J=18.4, 1H), 2.55 (s, 3H), 2.36 (s, 3H), -0.06 (s, 9H). ^{13}C NMR (100 MHz, $CDCl_3$): δ 201.3, 143.3, 139.0, 137.5, 135.4,

133.9, 132.9, 129.2, 127.7, 127.6, 127.5, 126.8, 126.5, 124.1, 100.7, 90.9, 44.5, 29.9, 21.4, -0.6. IR (KBr): 2959, 2179, 1697, 1375, 1162, 1093, 846, 819, 782, 544 cm^{-1}. MS (*m/z*, ESI): 450 $[M+H]^+$. HRMS-ESI calcd for $C_{25}H_{27}NO_3SSi$: 449.1481. Found: 449.1477.

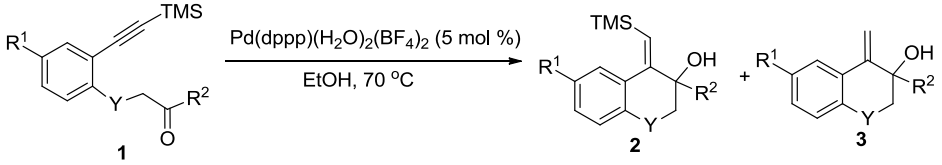


4m

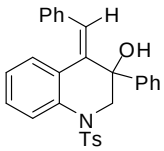
^{1}H NMR (400 MHz, $CDCl_3$): δ 7.74 (dd, J=7.6, 1.6 Hz, 1H), 7.46 (ddd, J=8.4, 7.2, 2.0 Hz, 1H), 7.11-7.01 (m, 2H), 4.79 (s, 2H), 2.65 (s, 3H), 0.16 (s, 9H). ^{13}C NMR (100 MHz, $CDCl_3$): δ 199.8, 157.0, 133.4, 130.4, 129.0, 121.4, 113.4, 99.3, 93.5, 57.2, 31.9, -0.4. IR (neat): 2960, 2900, 2179, 1678, 1579, 1483, 1452, 1417, 1358, 1294, 1251, 1216, 1126, 1031, 984, 845, 759. MS (*m/z*, EI): 245 (M^+-1), 231, 216, 203, 193, 175, 156, 151, 132, 115, 111, 96, 83 (100), 59, 43. HRMS Calcd for $C_{14}H_{17}O_2Si$ (M^+-1): 245.0998. Found: 245.1002.

III. General procedure for the synthesis of hydroquinolines.

General procedure for the synthesis of tetrahydroquinolines 2 and 3

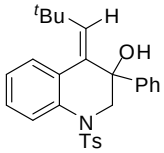


To a Schlenk tube were added $[Pd(dppp)(H_2O)_2](BF_4)_2$ (7.4 mg, 5 mol %), substrate **1** (0.2 mmol) and EtOH (2.0 mL) successively under nitrogen atmosphere. The reaction mixture was stirred at 70 ^{o}C until the consumption of the substrate as monitored by TLC. Then the mixture was purified by flash column chromatography (ethyl acetate: petroleum ether = 1:5) to obtain the products **2** and **3**.



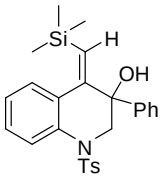
2c

^{1}H NMR (400 MHz, $CDCl_3$): δ 7.72 (d, J=8.0 Hz, 2H), 7.71 (d, J=7.6 Hz, 1H), 7.44 (d, J=7.2 Hz, 2H), 7.32-7.03 (m, 10H), 6.91-6.81 (m, 3H), 6.68 (s, 1H), 4.15 (d, J=13.6 Hz, 1H), 3.94 (d, J=13.6 Hz, 1H), 2.66 (s, 1H), 2.30 (s, 3H). ^{13}C NMR (100 MHz, $CDCl_3$): δ 144.0, 143.1, 139.9, 138.0, 136.4, 135.9, 129.9, 129.6, 128.9, 128.6, 128.3, 128.2, 128.0, 127.7, 127.5, 127.2, 125.8, 125.1, 124.6, 79.5, 59.2, 21.4. IR (neat): 3494, 3059, 3027, 2924, 1598, 1493, 1479, 1447, 1351, 1167, 1089, 925, 758, 736, 698, 670, 571 cm^{-1}. MS (70 eV, EI) *m/z* (%): 467 [M^+], 312, 206, 178, 167, 130, 105 (100), 91, 84, 77, 65. HRMS calcd for $C_{29}H_{25}NO_3S$: 467.1555. Found: 467.1553.



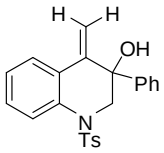
2d

m.p. 176.1-177.3 ^{o}C. ^{1}H NMR (400 MHz, DMSO): δ 7.62 (d, J=8.4 Hz, 1H), 7.49 (d, J=8.0 Hz, 2H), 7.30 (d, J=7.6 Hz, 2H), 7.25-7.00 (m, 6H), 6.93-6.78 (m, 2H), 6.04 (s, 1H), 5.77 (s, 1H), 4.36 (d, J=11.6 Hz, 1H), 3.59 (d, J=12.0 Hz, 1H), 2.31 (s, 3H), 0.74 (s, 9H). ^{13}C NMR (100 MHz, DMSO): δ 143.9, 143.2, 135.9, 134.3, 134.0, 133.7, 131.1, 130.2, 129.8, 127.7, 127.6, 127.1, 126.8, 126.0, 123.7, 123.1, 75.9, 56.7, 32.5, 30.6, 20.8. IR (KBr): 3517, 2958, 2600, 1598, 1492, 1478, 1469, 1447, 1344, 1170, 1089, 764, 694, 668, 658, 573 cm^{-1}. MS (*m/z*, ESI): 470 [M+Na]$^+$. HRMS-ESI calcd for $C_{27}H_{29}NO_3S$: 447.1868. Found: 447.1862.



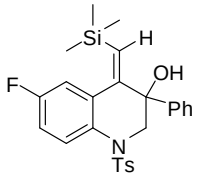
2e

m.p. 176.7-177.7 °C. ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.77 (dd, J=8.4, 1.2 Hz, 1H), 7.60 (d, J=8.4 Hz, 2H), 7.32-7.12 (m, 9H), 7.09 (td, J=7.6, 1.2 Hz, 1H), 5.78 (s, 1H), 4.10 (d, J=13.6 Hz, 1H), 3.78 (d, J=14.0 Hz, 1H), 2.32 (s, 3H), 2.31 (s, 1H), -0.12 (s, 9H). ^{13}C NMR (100 MHz, $CDCl_3$): δ 155.7, 143.7, 143.5, 137.9, 136.4, 132.2, 129.5, 129.2, 128.7, 128.6, 128.2, 127.5, 127.4, 125.7, 125.1, 123.9, 80.3, 59.4, 21.4, 0.1. IR (KBr): 3516, 2948, 2886, 1614, 1598, 1478, 1349, 1249, 1168, 1087, 920, 868, 835, 753, 703, 669, 558 cm^{-1}. MS (*m/z*, ESI): 481 $[M+NH_4]^+$. HRMS-ESI calcd for $C_{26}H_{33}N_2O_3SSi$: 481.1976. Found: 481.1969.



3e

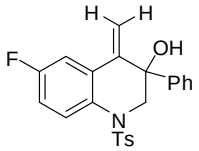
^{1}H NMR (400 MHz, $CDCl_3$): δ 7.73-7.64 (m, 4H), 7.54 (dd, J=8.0, 1.2 Hz, 2H), 7.41-7.30 (m, 3H), 7.28-7.19 (m, 3H), 7.18-7.12 (m, 1H), 5.81 (s, 1H), 5.20 (s, 1H), 4.22 (d, J=13.2 Hz, 1H), 3.98 (d, J=13.6 Hz, 1H), 2.97 (s, br, 1H), 2.38 (s, 3H). ^{13}C NMR (100 MHz, $CDCl_3$): δ 146.4, 143.6, 142.8, 136.6, 135.9, 129.3, 128.4, 127.9, 127.5, 127.2, 126.8, 126.2, 125.1, 124.5, 121.1, 113.5, 75.6, 56.9, 21.3. IR (neat): 3497, 3063, 3030, 2924, 2253, 1700, 1598, 1483, 1448, 1343, 1161, 1090, 911, 730, 565 cm^{-1}. MS (*m/z*, ESI): 409 $[M+NH_4]^+$. HRMS-ESI calcd for $C_{23}H_{25}N_2O_3S$: 409.1580. Found: 409.1572.



2f

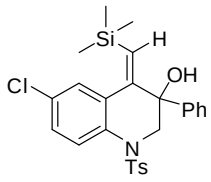
m.p. 159.5-160.2 °C. ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.74-7.66 (m, 1H), 7.62 (d, J=8.0 Hz, 2H), 7.37-7.15 (m, 7H), 7.04-6.92 (m, 2H), 5.90 (s, 1H), 4.11 (d, J=14.0 Hz, 1H), 3.83 (d, J=14.0 Hz, 1H), 2.41 (s, 1H), 2.37 (s, 3H), -0.05 (s, 9H). ^{13}C NMR (100 MHz, $CDCl_3$): δ 159.8 (d, J=244.5 Hz), 154.6, 143.9, 143.2, 135.9, 134.2 (d, J=8.0

Hz), 133.7 (d, J=2.3 Hz), 130.0, 129.6, 128.3, 127.6, 127.5, 126.0 (d, J=8.4 Hz), 125.5, 115.8 (d, J=22.4 Hz), 115.0 (d, J=23.1 Hz), 80.1, 59.3, 21.4, 0.0. ^{19}F NMR (376 MHz, $CDCl_3$): δ -116.4 (m, 1F). IR (KBr): 3526, 2949, 2886, 1620, 1597, 1585, 1482, 1447, 1437, 1350, 1249, 1169, 885, 865, 832, 705, 556, 546 cm^{-1}. MS (*m/z*, ESI): 499 $[M+NH_4]^+$. HRMS-ESI calcd for $C_{26}H_{28}FNO_3SSi$: 481.1543. Found: 481.1553.



3f

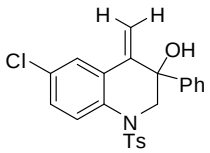
^{1}H NMR (400 MHz, $CDCl_3$): δ 7.66-7.58 (m, 3H), 7.48-7.43 (m, 2H), 7.37-7.27 (m, 4H), 7.23 (d, J=8.4 Hz, 2H), 7.00-6.93 (m, 1H), 5.74 (s, 1H), 5.18 (s, 1H), 4.20 (d, J=14.4 Hz, 1H), 3.78 (d, J=14.0 Hz, 1H), 2.59 (s, 1H), 2.39 (s, 3H). ^{13}C NMR (100 MHz, $CDCl_3$): δ 159.9 (d, J=242.8 Hz), 146.4, 143.9, 142.9, 136.6, 132.3 (d, J=2.9 Hz), 129.7 (d, J=7.8 Hz), 129.6, 128.2, 127.8, 127.5, 126.2, 124.1 (d, J=8.2 Hz), 115.8, 115.6 (d, J=4.4 Hz), 111.4 (d, J=23.4 Hz), 76.1, 57.6, 21.5. ^{19}F NMR (376 MHz, $CDCl_3$): δ -117.0 (m, 1F). IR (neat): 3497, 3061, 2923, 2853, 1597, 1583, 1486, 1447, 1432, 1341, 1288, 1186, 1162, 1089, 1063, 916, 869, 813, 704, 558 cm^{-1}. MS (*m/z*, ESI): 427 $[M+NH_4]^+$. HRMS-ESI calcd for $C_{23}H_{20}FNO_3S$: 409.1148. Found: 409.1157.



2g

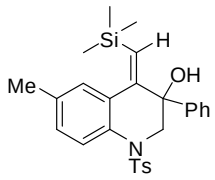
m.p. 150.7-151.5 ^{o}C. ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.68 (d, J=8.4 Hz, 1H), 7.62 (d, J=7.6 Hz, 2H), 7.40-7.14 (m, 9H), 5.89 (s, 1H), 4.09 (d, J=13.6 Hz, 1H), 3.87 (d, J=13.6 Hz, 1H), 2.37 (s, 4H), -0.04 (s, 9H). ^{13}C NMR (100 MHz, $CDCl_3$): δ 154.3, 144.0, 142.9, 136.1, 135.8, 133.3, 130.4, 129.9, 129.6, 128.8, 128.3, 127.7, 127.4, 125.7, 124.8, 79.7, 59.0, 21.4, 0.0. IR (KBr): 3512, 2956, 2924, 2895, 1722, 1623,

1597, 1494, 1473, 1447, 1414, 1358, 1328, 1249, 1242, 1165, 1088, 1017, 866, 836, 696, 541 cm^{-1}. MS (*m/z*, ESI): 515 $[M+NH_4]^+$. HRMS-ESI calcd for $C_{26}H_{28}ClNO_3SSi$: 497.1248. Found: 497.1257. The structure of **2g** was confirmed by X-ray crystallography. Deposit number of this compound from Crystallographic Data Centre is: **2g** (CCDC 1038047). Copies of the data can be obtained free of charge on application to CCDC, 12, Union Road, Cambridge CB21EZ, UK; fax: +44 1223 336 033; e-mail: deposit@ccdc.cam.ac.uk.



3g

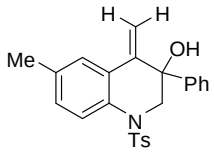
^{1}H NMR (400 MHz, $CDCl_3$): δ 7.64-7.54 (m, 4H), 7.46 (dd, J=8.2, 1.4 Hz, 2H), 7.37-7.27 (m, 3H), 7.24-7.15 (m, 3H), 5.76 (s, 1H), 5.19 (s, 1H), 4.17 (d, J=14.4 Hz, 1H), 3.87 (d, J=14.4 Hz, 1H), 2.53 (s, 1H), 2.38 (s, 3H). ^{13}C NMR (100 MHz, $CDCl_3$): δ 145.9, 144.0, 142.6, 136.4, 134.7, 130.3, 129.6, 128.7, 128.5, 128.2, 127.9, 127.5, 126.3, 125.0, 123.0, 114.9, 75.7, 57.1, 21.5. IR (neat): 3501, 3061, 2918, 2849, 1597, 1476, 1447, 1415, 1340, 1161, 1089, 1060, 971, 929, 813, 769, 737, 702, 672, 570, 542 cm^{-1}. MS (*m/z*, ESI): 443 $[M+NH_4]^+$. HRMS-ESI calcd for $C_{23}H_{20}ClNO_3S$: 425.0852. Found: 425.0867



2h

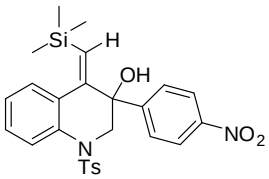
m.p. 156.9-157.1 ^{o}C. ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.65-7.59 (m, 3H), 7.35 (d, J=11.2 Hz, 2H), 7.25-7.20 (m, 5H), 7.18-7.13 (m, 2H), 5.76 (s, 1H), 4.17 (d, J=13.6 Hz, 1H), 3.73 (d, J=14.0 Hz, 1H), 2.36 (s, 3H), 2.33 (s, 3H), 2.29 (s, 1H), -0.07 (s, 9H). ^{13}C NMR (100 MHz, $CDCl_3$): δ 156.2, 143.8, 143.5, 136.6, 135.4, 134.8, 132.1, 129.9, 129.5, 129.2, 128.8, 128.1, 127.5, 127.4, 125.6, 123.9, 80.5, 59.8, 21.4, 20.7,

0.2. IR (KBr): 3511, 2956, 1719, 1616, 1599, 1483, 1448, 1376, 1344, 1246, 1170, 1089, 1036, 935, 863, 841, 694, 559 cm^{-1}. MS (*m/z*, ESI): 495 $[M+NH_4]^+$. HRMS-ESI calcd for $C_{27}H_{31}NO_3SSi$: 477.1794. Found: 477.1805.



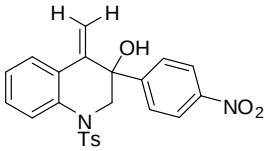
3h

^1H NMR (400 MHz, $CDCl_3$): δ 7.63 (d, J=8.4 Hz, 2H), 7.51 (d, J=8.4 Hz, 1H), 7.49-7.43 (m, 3H), 7.35-7.24 (m, 3H), 7.20 (d, J=8.0 Hz, 2H), 7.05 (dd, J=8.2, 1.4 Hz, 1H), 5.75 (s, 1H), 5.11 (s, 1H), 4.17 (d, J=13.6 Hz, 1H), 3.79 (d, J=13.6 Hz, 1H), 2.58 (s, 1H), 2.37 (s, 3H), 2.33 (s, 3H). ^{13}C NMR (100 MHz, $CDCl_3$): δ 147.1, 143.6, 143.2, 137.0, 134.5, 133.8, 129.6, 129.5, 128.0, 127.6, 127.4, 126.2, 125.5, 121.8, 113.8, 76.3, 57.7, 21.4, 20.8. IR (neat): 3499, 3059, 3029, 2922, 2856, 1699, 1597, 1492, 1448, 1340, 1161, 1089, 1062, 933, 912, 813, 770, 736, 704, 669, 658, 577, 557 cm^{-1}. MS (*m/z*, ESI): 423 $[M+NH_4]^+$. HRMS-ESI calcd for $C_{24}H_{23}NO_3S$: 405.1399. Found: 405.1411



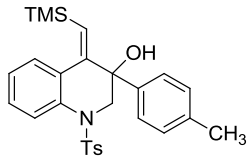
2i

^1H NMR (400 MHz, $CDCl_3$): δ 8.08 (dt, J=8.8, 2.4 Hz, 2H), 7.71-7.64 (m, 3H), 7.52 (dt, J=8.8, 2.4 Hz, 2H), 7.38-7.28 (m, 2H), 7.26-7.16 (m, 3H), 5.81 (s, 1H), 4.18 (d, J=14.0 Hz, 1H), 3.73 (d, J=14.4 Hz, 1H), 2.73 (s, 1H), 2.38 (s, 3H), -0.06 (s, 9H). ^{13}C NMR (100 MHz, $CDCl_3$): δ 155.0, 150.9, 147.0, 144.0, 138.0, 136.3, 132.0, 130.5, 129.7, 129.6, 128.7, 127.4, 126.6, 125.7, 124.3, 123.3, 80.5, 59.6, 21.4, 0.0. IR (neat): 3493, 3067, 2954, 2896, 1598, 1521, 1477, 1455, 1347, 1249, 1165, 1089, 962, 919, 857, 736, 703, 659, 571 cm^{-1}. MS (*m/z*, ESI): 509 $[M+H]^+$. HRMS-ESI calcd for $C_{26}H_{28}N_2O_5SSi$: 508.1488. Found: 508.1501.



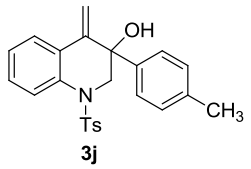
3i

^{1}H NMR (400 MHz, $CDCl_3$): δ 8.16 (d, J=8.4 Hz, 2H), 7.73-7.64 (m, 5H), 7.61 (d, J=8.0 Hz, 1H), 7.32-7.23 (m, 3H), 7.19 (t, J=8.0 Hz, 1H), 5.83 (s, 1H), 5.08 (s, 1H), 4.20 (d, J=14.0 Hz, 1H), 3.75 (d, J=14.0 Hz, 1H), 2.91 (s, 1H), 2.40 (s, 3H). ^{13}C NMR (100 MHz, $CDCl_3$): δ 150.4, 147.3, 146.3, 144.1, 136.8, 136.3, 129.7, 129.1, 127.4, 127.3, 127.1, 125.4, 125.2, 123.2, 122.2, 115.1, 76.3, 57.5, 21.5. IR (neat): 3486, 3070, 2923, 2852, 1598, 1519, 1483, 1456, 1346, 1161, 1088, 855, 569 cm^{-1}. MS (*m/z*, ESI): 437 $[M+H]^+$. HRMS-ESI calcd for $C_{23}H_{20}N_2O_5S$: 436.1093. Found: 436.1088.



2j

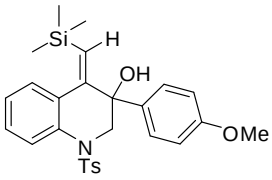
Colorless oil, yield: 59%; ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.96 (dd, *J* = 8.4, 1.2 Hz, 1H), 7.85 (d, *J* = 8.4 Hz, 2H), 7.52-7.48 (m, 2H), 7.45 (d, *J* = 8.4 Hz, 2H), 7.40 (d, *J* = 8.0 Hz, 2H), 7.34 (td, *J* = 7.6, 1.2 Hz, 1H), 7.26 (d, *J* = 8.0 Hz, 2H), 6.06 (s, 1H), 4.32 (d, *J* = 13.6 Hz, 1H), 4.09 (d, *J* = 13.6 Hz, 1H), 2.60 (br s, 1H), 2.58 (s, 3H), 2.48 (s, 3H), 0.15 (s, 9H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 155.6, 143.6, 140.4, 137.7, 137.2, 136.2, 132.0, 129.5, 129.1, 128.8, 128.7, 127.8, 127.4, 125.6, 125.0, 123.7, 79.9, 59.2, 21.4, 20.9, 0.2; IR (neat): ν 3502, 3374, 3029, 2955, 2924, 2897, 1599, 1512 cm^{-1}; HRMS (ESI) calcd for $C_{27}H_{35}N_2O_3SSi$ $[M+NH_4]^+$: 495.2132, Found: 495.2130.



3j

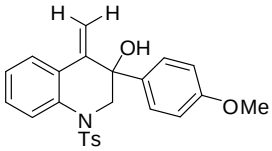
White solid, m. p. 158-159 ^{o}C. ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.71 (t, *J* = 8.4 Hz, 4H), 7.43 (d, *J* = 8.0 Hz, 2H), 7.32-7.25 (m, 3H), 7.20 (d, *J* = 8.4 Hz, 3H), 5.83 (s, 1H), 5.23 (s, 1H), 4.23 (d, *J* = 13.6 Hz, 1H), 3.95 (d, *J* = 13.6 Hz, 1H), 2.66 (br d, *J* = 3.2 Hz, 1H), 2.44 (s, 3H), 2.41 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 146.9, 143.7,

140.0, 137.4, 136.9, 136.2, 129.5, 128.8, 128.6, 127.4, 127.2, 126.3, 125.3, 124.7, 121.5, 113.7, 75.9, 57.3, 21.5, 21.0; IR (neat): ν 3537, 3464, 3029, 2921, 2871, 1594 1516 cm^{-1}; HRMS (ESI) calcd for $C_{24}H_{27}N_2O_3S$ $[M+NH_4]^+$: 423.1737, Found: 423.1738.



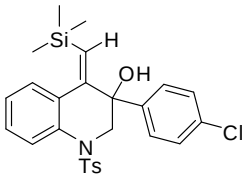
2k

^{1}H NMR (400 MHz, $CDCl_3$): δ 7.95 (d, J=7.6 Hz, 1H), 7.85 (d, J=8.4 Hz, 2H), 7.54-7.46 (m, 4H), 7.41 (d, J=8.4 Hz, 2H), 7.33 (t, J=8.0 Hz, 1H), 6.98 (d, J=7.6 Hz, 2H), 6.06 (s, 1H), 4.29 (d, J=13.6 Hz, 1H), 4.11 (d, J=13.6 Hz, 1H), 3.93 (s, 3H), 2.58 (s, 3H), 2.54 (s, br, 1H), 0.15 (s, 9H). ^{13}C NMR (100 MHz, $CDCl_3$): δ 158.8, 155.6, 143.7, 137.6, 136.2, 135.4, 131.9, 129.5, 129.2, 128.8, 127.6, 127.4, 127.0, 124.9, 123.6, 113.5, 79.5, 59.0, 55.1, 21.4, 0.2. IR (neat): 3500, 3066, 2954, 2917, 2849, 1599, 1510, 1477, 1454, 1351, 1306, 1248, 1165, 1089, 1032, 959, 914, 832, 759, 732, 719, 663, 564 cm^{-1}. MS (*m/z*, ESI): 516 $[M+Na]^+$. HRMS-ESI calcd for $C_{27}H_{31}NO_4SSi$: 493.1743. Found: 493.1751.



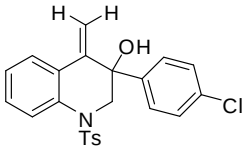
3k

^{1}H NMR (400 MHz, $CDCl_3$): δ 7.68-7.59 (m, 4H), 7.39 (d, J=8.8 Hz, 2H), 7.25-7.17 (m, 3H), 7.12 (t, J=7.6 Hz, 1H), 6.85 (d, J=8.8 Hz, 2H), 5.77 (s, 1H), 5.19 (s, 1H), 4.14 (d, J=13.6 Hz, 1H), 3.89 (d, J=13.2 Hz, 1H), 2.80 (s, 1H), 2.51 (s, 3H), 2.37 (s, 3H). ^{13}C NMR (100 MHz, $CDCl_3$): δ 159.0, 147.0, 143.7, 136.9, 136.2, 135.0, 129.5, 128.6, 127.6, 127.4, 127.1, 125.3, 124.7, 121.4, 113.5, 113.4, 75.6, 57.3, 55.2, 21.5. IR (neat): 3483, 2921, 2850, 1598, 1510, 1482, 1458, 1340, 1305, 1250, 1158, 1076, 1032, 972, 911, 878, 833, 812, 733, 660, 563 cm^{-1}. MS (*m/z*, ESI): 444 $[M+Na]^+$. HRMS-ESI calcd for $C_{24}H_{23}NO_4S$: 421.1348. Found: 421.1352.



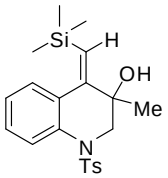
2l

^{1}H NMR (400 MHz, $CDCl_3$): δ 7.69 (d, J=8.4 Hz, 1H), 7.62 (d, J=8.0 Hz, 2H), 7.33-7.23 (m, 4H), 7.22-7.16 (m, 4H), 7.13 (td, J=7.6, 0.8 Hz, 1H), 5.78 (s, 1H), 4.10 (d, J=14.0 Hz, 1H), 3.73 (d, J=14.0 Hz, 1H), 2.38 (s, 1H), 2.35 (s, 3H), -0.08 (s, 9H). ^{13}C NMR (100 MHz, $CDCl_3$): δ 155.4, 143.8, 142.1, 137.9, 136.4, 133.3, 132.0, 129.6, 129.4, 129.1, 128.7, 128.3, 127.4, 127.2, 125.3, 123.9, 80.0, 59.4, 21.4, 0.1. IR (neat): 3494, 3066, 2953, 2896, 1598, 1490, 1478, 1455, 1401, 1351, 1248, 1165, 1090, 1013, 961, 919, 864, 833, 738, 709, 660, 572, 542 cm^{-1}. MS (*m/z*, ESI): 520 $[M+Na]^+$. HRMS-ESI calcd for $C_{26}H_{28}ClNO_3SSi$: 497.1248. Found: 497.1252.



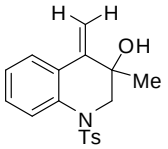
3l

^{1}H NMR (400 MHz, $CDCl_3$): δ 7.68-7.60 (m, 4H), 7.42 (dt, J=8.8, 2.4 Hz, 2H), 7.32-7.22 (m, 5H), 7.15 (td, J=7.6, 1.6 Hz, 1H), 5.79 (s, 1H), 5.13 (s, 1H), 4.16 (d, J=14.0 Hz, 1H), 3.79 (d, J=13.6 Hz, 1H), 2.62 (s, 1H), 2.39 (s, 3H). ^{13}C NMR (100 MHz, $CDCl_3$): δ 146.7, 143.9, 141.6, 136.9, 136.3, 133.6, 129.6, 128.9, 128.3, 127.8, 127.3, 127.2, 125.2, 125.0, 121.8, 114.2, 75.9, 57.4, 21.5. IR (neat): 3480, 3066, 2924, 2854, 1686, 1596, 1489, 1452, 1401, 1350, 1163, 1091, 814, 737, 663, 569, 544 cm^{-1}. MS (70 eV, EI) *m/z* (%): 425 [M^+], 302, 282, 270, 250, 240, 217, 204, 176, 155, 139 (100), 130, 111, 91, 77, 65, 51, 43. HRMS calcd for $C_{23}H_{20}NO_3SCl$: 425.0852. Found: 425.0856.



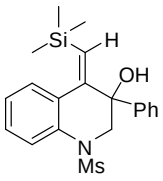
2n

^{1}H NMR (400 MHz, $CDCl_3$): δ 7.74 (d, J=8.4 Hz, 1H), 7.60 (d, J=8.4 Hz, 2H), 7.31 (td, J=8.4, 2.0 Hz, 1H), 7.24 (dd, J=8.0, 1.6 Hz, 1H), 7.18 (d, J=8.0 Hz, 2H), 7.13 (td, J=7.6, 1.2 Hz, 1H), 5.77 (s, 1H), 3.95 (d, J=13.2 Hz, 1H), 3.50 (d, J=14.0 Hz, 1H), 2.36 (s, 3H), 1.87 (s, br, 1H), 1.29 (s, 3H), -0.07 (s, 9H). ^{13}C NMR (100 MHz, $CDCl_3$): δ 155.3, 143.7, 137.2, 136.4, 131.4, 129.6, 129.2, 128.9, 127.3, 124.8, 124.4, 123.8, 75.9, 59.0, 27.1, 21.4, 0.2. IR (neat): 3515, 2956, 2926, 1599, 1484, 1455, 1347, 1251, 1162, 1089, 843, 753, 660, 574, 545 cm^{-1}. MS (70 eV, EI) *m/z* (%): 401 [M^+], 386, 329, 246 (100), 231, 218, 174, 156, 130, 115, 103, 91, 73, 65, 43. HRMS calcd for $C_{21}H_{27}NO_3SSi$: 401.1481. Found: 401.1478.



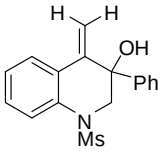
3n

^{1}H NMR (400 MHz, $CDCl_3$): δ 7.73 (d, J=8.0 Hz, 2H), 7.58 (d, J=8.4 Hz, 1H), 7.50 (dd, J=8.0, 1.2 Hz, 1H), 7.26 (d, J=8.0 Hz, 2H), 7.21 (td, J=8.0, 1.2 Hz, 1H), 7.09 (td, J=7.6, 0.8 Hz, 1H), 5.53 (s, 1H), 5.42 (s, 1H), 3.89 (d, J=13.2 Hz, 1H), 3.73 (d, J=12.8 Hz, 1H), 2.39 (s, 3H), 2.14 (s, br, 1H), 1.45 (s, 3H). ^{13}C NMR (100 MHz, $CDCl_3$): δ 146.9, 143.9, 137.0, 135.9, 129.7, 128.6, 127.1, 126.8, 125.6, 124.6, 121.3, 109.2, 71.1, 57.1, 26.3, 21.5. IR (neat): 3500, 3066, 2977, 2924, 1694, 1598, 1483, 1454, 1346, 1162, 1089, 916, 814, 746, 660, 573, 545 cm^{-1}. MS (*m/z*, ESI): 352 $[M+Na]^+$. HRMS-ESI calcd for $C_{18}H_{19}NO_3S$: 329.1086. Found: 329.1086.



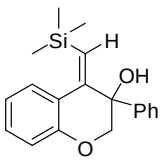
2p

^{1}H NMR (400 MHz, $CDCl_3$): δ 7.69 (dd, J=8.0, 1.2 Hz, 1H), 7.54 (dd, J=8.0, 1.6 Hz, 1H), 7.44-7.39 (m, 2H), 7.38-1.19 (m, 5H), 5.96 (s, 1H), 4.23 (d, J=14.4 Hz, 1H), 3.72 (d, J=14.4 Hz, 1H), 2.95 (s, 3H), 2.73 (s, 1H), 0.08 (s, 9H). ^{13}C NMR (100 MHz, $CDCl_3$): δ 156.1, 143.7, 137.8, 131.6, 130.2, 129.6, 128.8, 128.3, 127.8, 125.9, 125.4, 123.8, 80.7, 59.0, 38.8, 29.6, 0.4. IR (neat): 3500, 2953, 1599, 1476, 1456, 1339, 1249, 1155, 1068, 973, 857, 775, 700, 541, 510 cm^{-1}. MS (*m/z*, ESI): 410 $[M+Na]^+$. HRMS-ESI calcd for $C_{20}H_{25}NO_3SSi$: 387.1324. Found: 387.1327.



3p

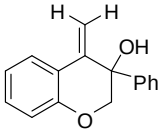
^{1}H NMR (400 MHz, $CDCl_3$): δ 7.87 (dd, J=8.4, 1.2 Hz, 1H), 7.79 (dd, J=8.0, 1.6 Hz, 1H), 7.50-7.45 (m, 2H), 7.38-7.27 (m, 4H), 7.24-7.18 (m, 1H), 5.94 (s, 1H), 5.18 (s, 1H), 4.20 (d, J=14.4 Hz, 1H), 3.85 (d, J=14.4 Hz, 1H), 2.85 (s, 3H), 2.45 (s, 1H). ^{13}C NMR (100 MHz, $CDCl_3$): δ 146.5, 142.7, 136.0, 129.3, 128.2, 128.0, 126.6, 126.5, 125.3, 125.2, 122.2, 113.6, 76.1, 56.5, 38.6. IR (neat): 3512, 3031, 2933, 1617, 1597, 1481, 1340, 1320, 1147, 1069, 978, 910, 774, 765, 542, 512 cm^{-1}. MS (*m/z*, ESI): 338 $[M+Na]^+$. HRMS-ESI calcd for $C_{17}H_{17}NO_3S$: 315.0929. Found: 315.0944.



2q

^{1}H NMR (400 MHz, $CDCl_3$): δ 7.51-7.44 (m, 3H), 7.34-7.23 (m, 3H), 7.21-7.14 (m, 1H), 6.92-6.86 (m, 1H), 6.80 (dd, J=8.0, 0.8 Hz, 1H), 5.89 (s, 1H), 4.53 (d, J=11.2

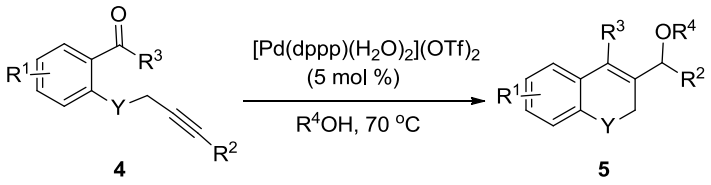
Hz, 1H), 4.17 (d, J=11.2 Hz, 1H), 2.28 (s, 1H), 0.19 (s, 9H). ^{13}C NMR (100 MHz, $CDCl_3$): δ 154.3, 152.2, 141.1, 130.0, 128.9, 128.3, 127.9, 126.7, 123.4, 122.3, 120.2, 116.5, 73.9, 73.7, 0.4. IR (neat): 3454, 3061, 3033, 2953, 2895, 1604, 1481, 1448, 1249, 1048, 875, 753, 698 cm^{-1}. MS (70 eV, EI) *m/z* (%): 310 [M^+], 295, 277, 261, 219, 191, 175 (100), 163, 151, 135, 115, 105, 91, 73, 51. HRMS calcd for $C_{20}H_{22}OS$: 310.1391. Found: 310.1392.



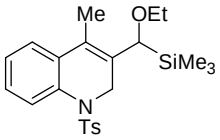
3q

^{1}H NMR (400 MHz, $CDCl_3$): δ 7.67 (dd, J=8.0, 1.6 Hz, 1H), 7.55-7.50 (m, 2H), 7.39-7.28 (m, 3H), 7.24-7.19 (m, 1H), 7.01-6.95 (m, 1H), 6.92 (dd, J=8.0, 1.2 Hz, 1H), 5.80 (s, 1H), 5.10 (s, 1H), 4.32 (d, J=11.2 Hz, 1H), 4.10 (d, J=11.6 Hz, 1H), 2.43 (s, 1H). ^{13}C NMR (100 MHz, $CDCl_3$): δ 153.5, 143.9, 141.0, 129.6, 128.2, 127.8, 126.5, 124.7, 121.5, 120.7, 117.6, 109.2, 73.8, 72.8. IR (neat): 3444, 3061, 3033, 2923, 2870, 1605, 1575, 1482, 1459, 1447, 1310, 1249, 1223, 1040, 907, 835, 753, 699 cm^{-1}. MS (70 eV, EI) *m/z* (%): 238 [M^+], 219, 178, 133, 105 (100), 91, 77, 51. HRMS calcd for $C_{16}H_{14}O_2$: 238.0994. Found: 238.0996.

General procedure for the synthesis of dihydroquinolines 5:

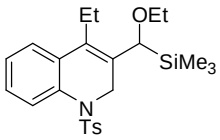


To a Schlenk tube were added $[Pd(dppp)(H_2O)_2](OTf)_2$ (8.6 mg, 5 mol %), alkyne-ketone substrate **4** (0.2 mmol) and alcohol (2.0 mL) successively under nitrogen atmosphere. The reaction mixture was stirred at 70 ^{o}C until the consumption of the substrate as monitored by TLC. Then the mixture was purified by flash column chromatography (ethyl acetate: petroleum ether = 1:5) to obtain the products **5**.



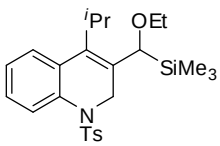
5a

^{1}H NMR (400 MHz, $CDCl_3$): δ 7.65-7.59 (m, 1H), 7.47-7.41 (m, 2H), 7.24-7.15 (m, 3H), 7.11 (d, J=8.4 Hz, 2H), 4.78-4.70 (m, 1H), 4.03-3.94 (m, 2H), 3.44-3.34 (m, 1H), 3.21-3.11 (m, 1H), 2.34 (s, 3H), 1.78 (s, 3H), 1.20 (t, J=7.0 Hz, 3H), 0.02 (s, 9H). ^{13}C NMR (100 MHz, $CDCl_3$): δ 143.0, 137.2, 135.2, 134.8, 132.4, 128.9, 126.9, 126.7, 125.6, 123.9, 123.5, 123.2, 73.6, 67.3, 46.1, 21.3, 15.2, 13.4, -2.7. IR (neat): 2969, 2925, 2866, 1657, 1598, 1484, 1452, 1347, 1248, 1163, 1092, 1061, 841, 574 cm^{-1}. MS (*m/z*, ESI): 452 $[M+Na]^+$. HRMS-ESI calcd for $C_{23}H_{31}NO_3SSi$: 429.1794. Found: 429.1785.



5b

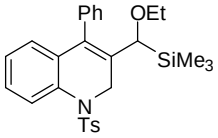
^{1}H NMR (400 MHz, $CDCl_3$): δ 7.73-7.68 (m, 1H), 7.39 (d, J=8.0 Hz, 2H), 7.25-7.15 (m, 3H), 7.07 (d, J=8.4 Hz, 2H), 4.76 (d, J=13.2 Hz, 1H), 3.96 (d, J=13.2 Hz, 1H), 3.93 (s, 1H), 3.42-3.32 (m, 1H), 3.20-3.10 (m, 1H), 2.31 (s, 3H), 2.35-2.22 (m, 2H), 1.20 (t, J=7.0 Hz, 3H), 0.67 (t, J=7.4 Hz, 3H), 0.01 (s, 9H). ^{13}C NMR (100 MHz, $CDCl_3$): δ 143.0, 137.2, 135.3, 134.7, 130.8, 129.5, 128.9, 126.9, 126.7, 125.8, 125.0, 123.5, 73.5, 67.3, 46.0, 21.3, 19.4, 15.3, 13.0, -2.6. IR (neat): 2969, 2928, 2873, 1598, 1485, 1453, 1348, 1247, 1164, 1095, 1069, 841, 767, 668, 576 cm^{-1}. MS (*m/z*, ESI): 466 $[M+Na]^+$. HRMS-ESI calcd for $C_{24}H_{33}NO_3SSi$: 443.1950. Found: 443.1947.



5c

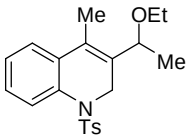
^{1}H NMR (400 MHz, $CDCl_3$): δ 7.70 (dd, J=8.0, 1.6 Hz, 1H), 7.45-7.37 (m, 3H),

7.23-7.06 (m, 4H), 4.47 (d, J=17.2 Hz, 1H), 4.12 (d, J=17.6 Hz, 1H), 4.01 (s, 1H), 3.40-3.30 (m, 1H), 3.20-3.10 (m, 1H), 2.96-2.84 (m, 1H), 2.32 (s, 3H), 1.18 (t, J=7.0 Hz, 3H), 1.11 (d, J=7.6 Hz, 3H), 1.03 (d, J=7.2 Hz, 3H), 0.05 (s, 9H). ^{13}C NMR (100 MHz, $CDCl_3$): δ 143.0, 137.4, 136.5, 135.7, 134.0, 130.9, 129.0, 127.0, 126.5, 125.2, 125.1, 125.0, 72.8, 67.0, 46.6, 27.7, 21.3, 21.1, 15.3, -2.4. IR (neat): 2969, 2930, 2874, 1598, 1482, 1452, 1348, 1248, 1163, 1094, 1068, 841, 764, 680, 576 cm^{-1}. MS (*m/z*, ESI): 480 $[M+Na]^+$. HRMS-ESI calcd for $C_{25}H_{35}NO_3SSi$: 457.2107. Found: 457.2103.



5d

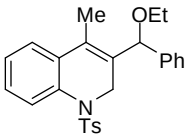
^{1}H NMR (400 MHz, $CDCl_3$): δ 7.73 (dd, J=8.0, 1.2 Hz, 1H), 7.47 (d, J=8,4 Hz, 2H), 7.40-7.26 (m, 3H), 7.24-7.14 (m, 3H), 7.01 (td, J=8.0, 1.6 Hz, 1H), 7.00-6.85 (br, 1H), 6.55 (dd, J=8.0, 1.6 Hz, 1H), 6.50-6.30 (br, 1H), 4.92 (d, J=17.2 Hz, 1H), 4.22 (d, J=17.6 Hz, 1H), 3.48 (s, 1H), 3.35-3.25 (m, 1H), 2.96-2.86 (m, 1H), 2.41 (s, 3H), 1.07 (t, J=7.0 Hz, 3H), 0.00 (s, 9H). ^{13}C NMR (100 MHz, $CDCl_3$): δ 143.2, 137.1, 136.6, 136.2, 134.3, 131.9, 131.7, 129.1, 127.3, 127.2, 127.1, 126.3, 125.7, 124.7, 72.8, 66.6, 46.4, 21.4, 15.1, -2.3. IR (neat): 3061, 2970, 2926, 2865, 1596, 1481, 1453, 1349, 1248, 1165, 1091, 1069, 841, 767, 705, 670, 576 cm^{-1}. MS (*m/z*, ESI): 514 $[M+Na]^+$. HRMS-ESI calcd for $C_{28}H_{33}NO_3SSi$: 491.1950. Found: 491.1946.



5e

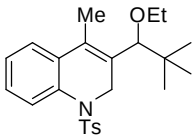
^{1}H NMR (400 MHz, $CDCl_3$): δ 7.71 (dd, J=8.0, 1.2 Hz, 1H), 7.32-7.18 (m, 5H), 7.06 (d, J=8.0 Hz, 2H), 4.52 (d, J=17.6 Hz, 1H), 4.24 (q, J=6.8 Hz, 1H), 4.13 (dd, J=17.6, 1.6 Hz, 1H), 3.32-3.16 (m, 2H), 2.32 (s, 3H), 1.69 (s, 3H), 1.16 (t, J=6.8 Hz,

3H), 1.13 (d, J=6.8 Hz, 3H). ^{13}C NMR (100 MHz, $CDCl_3$): δ 143.0, 136.4, 134.9, 134.1, 132.3, 128.7, 127.3, 127.2, 126.2, 125.9, 125.6, 123.4, 73.0, 63.9, 44.1, 21.4, 19.3, 15.3, 12.4. IR (neat): 2974, 2927, 2889, 1598, 1484, 1453, 1346, 1164, 1090, 813, 763, 671, 577, 544 cm^{-1}. MS (*m/z*, ESI): 394 $[M+Na]^+$. HRMS-ESI calcd for $C_{21}H_{25}NO_3S$: 371.1555. Found: 371.1540.



5f

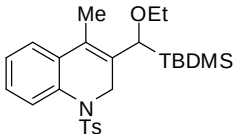
^{1}H NMR (400 MHz, $CDCl_3$): δ 7.68 (dd, J=8.0, 1.6 Hz, 1H), 7.40-7.12 (m, 8H), 7.01 (d, J=8.4 Hz, 2H), 6.91 (d, J=8.4 Hz, 2H), 5.19 (s, 1H), 4.63 (dd, J=17.6, 1.6 Hz, 1H), 4.13 (dd, J=17.6, 1.6 Hz, 1H), 3.52-3.34 (m, 2H), 2.30 (s, 3H), 1.81 (s, 3H), 1.29 (t, J=7.0 Hz, 3H). ^{13}C NMR (100 MHz, $CDCl_3$): δ 142.6, 139.9, 135.8, 134.6, 132.7, 132.4, 128.5, 128.4, 127.6, 127.4, 127.3, 126.3, 126.2, 126.1, 125.6, 123.7, 78.3, 64.2, 44.3, 21.4, 15.3, 13.2. IR (neat): 2971, 2871, 1598, 1487, 1450, 1338, 1162, 1097, 1055, 768, 669, 566 cm^{-1}. MS (*m/z*, ESI): 456 $[M+Na]^+$. HRMS-ESI calcd for $C_{26}H_{27}NO_3S$: 433.1712. Found: 433.1700.



5g

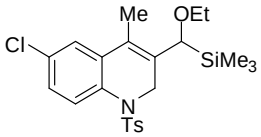
^{1}H NMR (400 MHz, $CDCl_3$): δ 7.58 (dd, J=7.6, 1.2 Hz, 1H), 7.52 (d, J=8,4 Hz, 2H), 7.26 (dd, J=7.6, 1.6 Hz, 1H), 7.23-7.12 (m, 4H), 4.70 (d, J=15.6 Hz, 1H), 4.08 (d, J=16.4 Hz, 1H), 3.86 (s, 1H), 3.50-3.30 (m, 1H), 3.26-3.14 (m, 1H), 2.35 (s, 3H), 1.91 (s, 3H), 1.20 (t, J=6.8 Hz, 3H), 0.89 (s, 9H). ^{13}C NMR (100 MHz, $CDCl_3$): δ 143.1, 137.6, 135.6, 131.8, 129.1, 128.7, 127.2, 126.8, 125.1, 124.1, 122.6, 65.3, 37.2, 26.9, 21.4, 15.1, 14.4. IR (neat): 2955, 2871, 1598, 1485, 1453, 1347, 1162, 1095, 909, 733, 678, 577, 543 cm^{-1}. MS (*m/z*, ESI): 436 $[M+Na]^+$. HRMS-ESI calcd for $C_{24}H_{31}NO_3S$:

413.2025. Found: 413.2024.



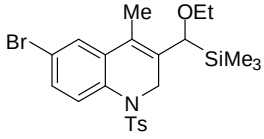
5h

^{1}H NMR (400 MHz, $CDCl_3$): δ 7.61-7.56 (m, 1H), 7.48 (d, J=8.8 Hz, 2H), 7.24-7.10 (m, 5H), 4.72 (d, J=17.2 Hz, 1H), 4.20 (s, 1H), 3.99 (d, J=16.8 Hz, 1H), 3.38-3.26 (m, 1H), 3.18-3.06 (m, 1H), 2.35 (s, 3H), 1.83 (s, 3H), 1.18 (t, J=7.0 Hz, 3H), 0.93 (s, 9H), 0.07 (s, 3H), -0.25 (s, 3H). ^{13}C NMR (100 MHz, $CDCl_3$): δ 143.1, 137.5, 135.0, 132.2, 129.1, 126.8, 126.7, 125.4, 123.6, 123.5, 123.4, 67.0, 26.7, 21.4, 17.1, 15.4, 13.5, -7.0, -7.1. IR (neat): 2954, 2927, 2856, 1598, 1484, 1453, 1348, 1247, 1163, 1093, 1062, 835, 824, 811, 765, 669, 574, 545 cm^{-1}. MS (*m/z*, ESI): 494 $[M+Na]^+$. HRMS-ESI calcd for $C_{26}H_{37}NO_3SSi$: 471.2263. Found: 471.2267.



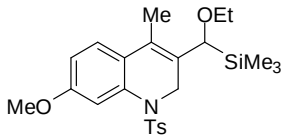
5i

^{1}H NMR (400 MHz, $CDCl_3$): δ 7.55 (d, J=8.4 Hz, 1H), 7.43 (d, J=8.4 Hz, 2H), 7.17-7.10 (m, 4H), 4.72 (d, J=17.2 Hz, 1H), 4.00-3.92 (m, 1H), 3.94 (s, 1H), 3.40-3.30 (m, 1H), 3.19-3.10 (m, 1H), 2.35 (s, 3H), 1.74 (s, 3H), 1.19 (t, J=7.0 Hz, 3H), 0.01 (s, 9H). ^{13}C NMR (100 MHz, $CDCl_3$): δ 143.8, 137.0, 136.9, 134.0, 133.3, 131.3, 129.1, 126.9, 126.4, 125.4, 123.6, 122.2, 73.8, 67.5, 46.1, 21.4, 15.3, 13.4, -2.7. IR (neat): 2963, 1595, 1479, 1348, 1247, 1162, 1090, 838, 811, 666 cm^{-1}. MS (*m/z*, ESI): 486 $[M+Na]^+$. HRMS-ESI calcd for $C_{23}H_{30}ClNO_3SSi$: 463.1404. Found: 463.1408.



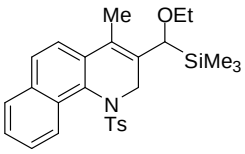
5j

^{1}H NMR (400 MHz, $CDCl_3$): δ 7.63 (d, J=8.4 Hz, 1H), 7.58 (d, J=8.4 Hz, 2H), 7.46-7.40 (m, 2H), 7.28 (d, J=8,4 Hz, 2H), 4.87 (d, J=17.2 Hz, 1H), 4.11 (d, J=17.2 Hz, 1H), 4.09 (s, 1H), 3.54-3.44 (m, 1H), 3.34-3.24 (m, 1H), 2.49 (s, 3H), 1.89 (s, 3H), 1.33 (t, J=7.0 Hz, 3H), 0.15 (s, 9H). ^{13}C NMR (100 MHz, $CDCl_3$): δ 143.3, 136.9, 136.8, 134.2, 133.8, 129.3, 129.1, 126.9, 126.5, 125.6, 122.1, 119.1, 73.8, 67.5, 46.0, 21.4, 15.2, 13.4, -2.7. IR (neat): 2968, 1596, 1478, 1347, 1247, 1162, 1089, 1074, 1050, 838, 812, 730, 665 cm^{-1}. MS (*m/z*, ESI): 530 $[M+Na]^+$. HRMS-ESI calcd for $C_{23}H_{30}BrNO_3SSi$: 507.0899. Found: 507.0905.



5k

^{1}H NMR (400 MHz, $CDCl_3$): δ 7.47 (d, J=8.4 Hz, 2H), 7.22 (d, J=2.8 Hz, 1H), 7.12 (d, J=8.0 Hz, 2H), 7.02 (d, J=8.8 Hz, 1H), 6.72 (dd, J=8,4, 2.4 Hz, 1H), 4.69 (d, J=17.2 Hz, 1H), 3.98 (d, J=17.2 Hz, 1H), 3.94 (s, 1H), 3.81 (s, 3H), 3.42-3.32 (m, 1H), 3.18-3.08 (m, 1H), 2.34 (s, 3H), 1.76 (s, 3H), 1.17 (t, J=7.0 Hz, 3H), 0.00 (s, 9H). ^{13}C NMR (100 MHz, $CDCl_3$): δ 158.3, 143.3, 137.2, 136.1, 131.1, 129.7, 129.0, 127.8, 127.0, 125.4, 124.4, 123.2, 111.5, 109.2, 73.5, 67.1, 55.4, 46.2, 21.4, 15.3, 13.5, -2.7. IR (neat): 2961, 2928, 2898, 1606, 1497, 1447, 1347, 1298, 1247, 1212, 1159, 1056, 1002, 913, 837, 729, 678 cm^{-1}. MS (*m/z*, ESI): 414 $[M-OEt]^+$. HRMS-ESI calcd for $C_{22}H_{28}NO_3SSi$: 414.1554. Found: 414.1542.

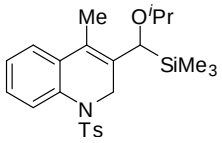


5l

m.p. 132.4-133.4 ^{o}C. ^{1}H NMR (400 MHz, $CDCl_3$): δ 8.57 (d, J=8.4 Hz, 1H), 7.79 (d, J=8.4 Hz, 1H), 7.76 (d, J=8.4 Hz, 1H), 7.55 (t, J=7.6 Hz, 1H), 7.46 (t, J=7.6 Hz, 1H), 7.23 (d, J=8.4 Hz, 1H), 7.18 (d, J=8.4 Hz, 2H), 7.01 (d, J=8.4 Hz, 2H), 5.20 (d,

J=18.4 Hz, 1H), 3.89 (s, 1H), 3.78 (d, J=18.0 Hz, 1H), 3.74-3.62 (m, 1H), 3.47-3.37 (m, 1H), 2.33 (s, 3H), 1.52 (s, 3H), 1.35 (t, J=6.8 Hz, 3H), -0.06 (s, 9H). ^{13}C NMR (100 MHz, $CDCl_3$): δ 148.2, 137.7, 135.7, 133.0, 132.7, 130.8., 130.0, 128.1, 127.9, 127.6, 127.1, 123.6, 126.0, 125.9, 122.8, 121.3, 73.5, 68.2, 48.1, 21.4, 15.8, 13.5, -2.5. IR (neat): 2954, 2924, 2897, 1594, 1446, 1382, 1343, 1247, 1161, 1114, 1085, 1051, 1020, 966, 838, 817, 781, 753, 703, 673, 630, 618 cm^{-1}. MS (*m/z*, ESI): 480 $[M+H]^+$. HRMS-ESI calcd for $C_{27}H_{34}NO_3SSi$: 480.2023. Found: 480.2011.

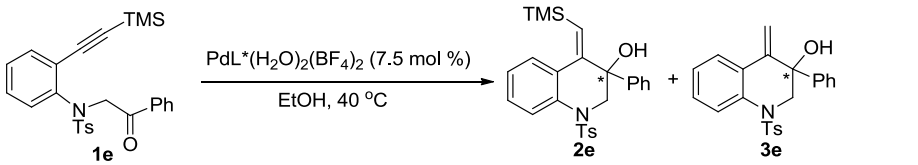
The structure of **5l** was confirmed by X-ray crystallography. Deposit number of this compound from Crystallographic Data Centre is: **5l** (CCDC 1021338). Copies of the data can be obtained free of charge on application to CCDC, 12, Union Road, Cambridge CB21EZ, UK; fax: +44 1223 336 033; e-mail: deposit@ccdc.cam.ac.uk.



5n

^{1}H NMR (400 MHz, $CDCl_3$): δ 7.64-7.59 (m, 1H), 7.45 (d, J=8,4 Hz, 2H), 7.22-7.14 (m, 3H), 7.12 (d, J=8,4 Hz, 2H), 4.70 (d, J=17.2 Hz, 1H), 4.18 (s, 1H), 4.07 (dt, J=17.2, 1.6 Hz, 1H), 3.37-3.26 (m, 1H), 2.34 (s, 3H), 1.81 (s, 3H), 1.21 (d, J=6.4 Hz, 3H), 1.06 (d, J=6.0 Hz, 3H), 0.01 (s, 9H). ^{13}C NMR (100 MHz, $CDCl_3$): δ 143.0, 137.2, 136.0, 134.8, 132.3, 129.0, 126.8, 126.6, 125.4, 123.5, 123.4, 123.0, 70.7, 69.8, 46.3, 23.3, 21.3, 20.5, 13.5, -2.7. IR (neat): 2965, 1598, 1484, 1452, 1345, 1246, 1160, 1091, 1022, 838, 731, 662 cm^{-1}. MS (*m/z*, ESI): 466 $[M+Na]^+$. HRMS-ESI calcd for $C_{24}H_{33}NO_3SSi$: 443.1950. Found: 443.1954.

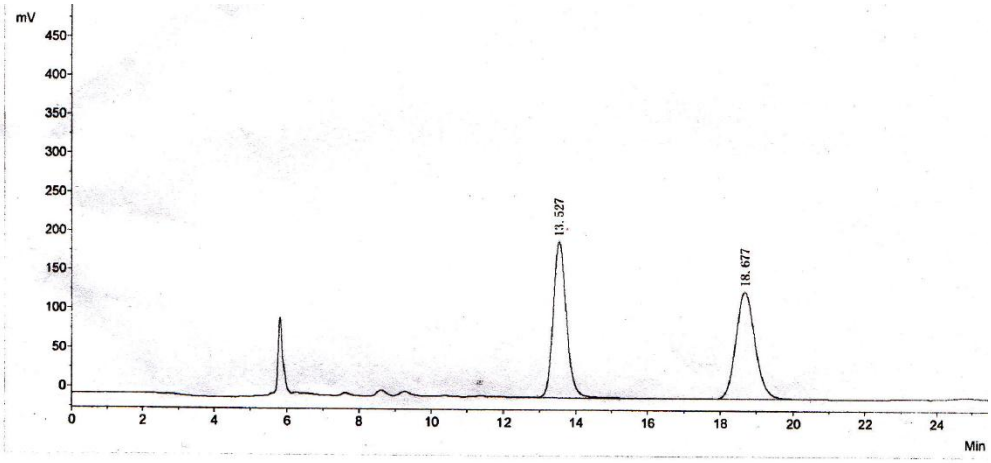
IV. Asymmetric reductive cyclization of 1e.



L*=(*S,S*)-bdpp yield: **2e** (76%), **3e** (19%); ee (**2e**): 56%
L*=(*R*)-binap yield: **2e** (20%), **3e** (19%); ee (**2e**): 82%

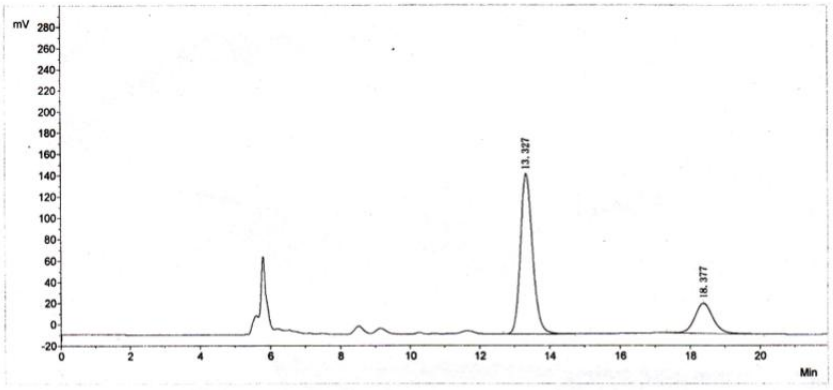
To a Schlenk tube were added catalyst (5 mol %), substrate **1e** (0.1 mmol) and EtOH (1.0 mL) successively under nitrogen atmosphere. The reaction mixture was stirred at 40 °C until the consumption of the starting material as monitored by TLC. Then the mixture was purified by flash column chromatography (ethyl acetate: petroleum ether = 1:5) to obtain the product **2e** and **3e**. The ee value of **2e** was determined by chiral HPLC using a Phenomenex Lux-cellulose-2 column with hexane : isopropanol = 70 : 30, flow = 0.5 mL/min; λ = 214 nm.

Racemic (**2e**):



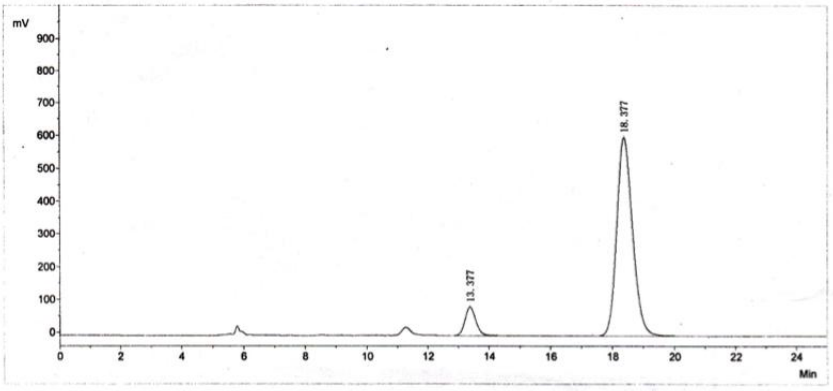
No.	PeakNo	R.Time	PeakHeight	PeakArea	PerCent
1	1	13.527	198331.1	5039622.1	50.1203
2	2	18.677	136821.0	5015436.1	49.8797
Total			335152.1	10055058.2	100.0000

[Pd(*s*,*s*-bdpp)(H_2O)](BF_4)$_2$ as the catalyst. yield: **2e** (76%), **3e** (19%); ee (**2e**): 56%



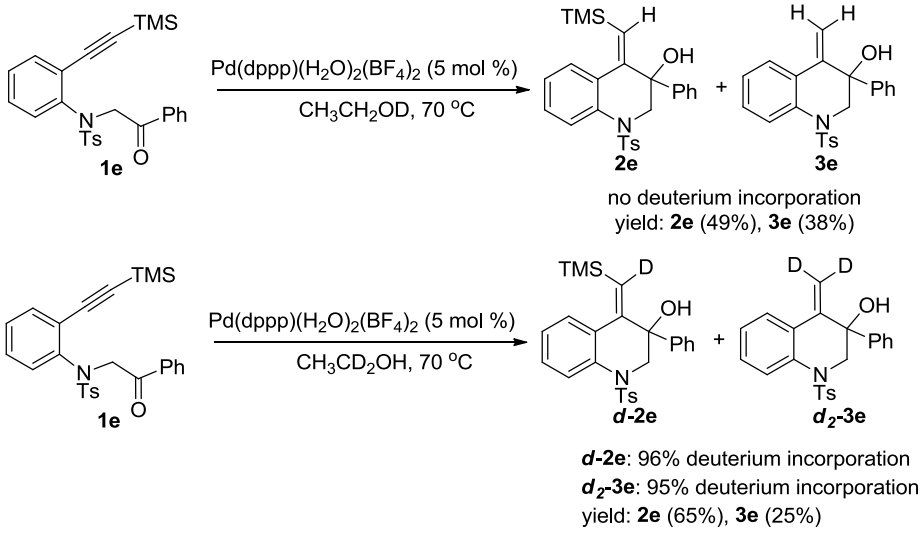
No.	PeakNo	R.Time	PeakHeight	PeakArea	PerCent
1	1	13.327	149744.8	3618436.8	78.3825
2	2	18.377	27899.6	997948.3	21.6175
Total			177644.4	4616385.1	100.0000

[Pd(*R*-binap)(H_2O)](BF_4)$_2$ as the catalyst. yield: **2e** (20%), **3e** (19%); ee (**2e**): 82%

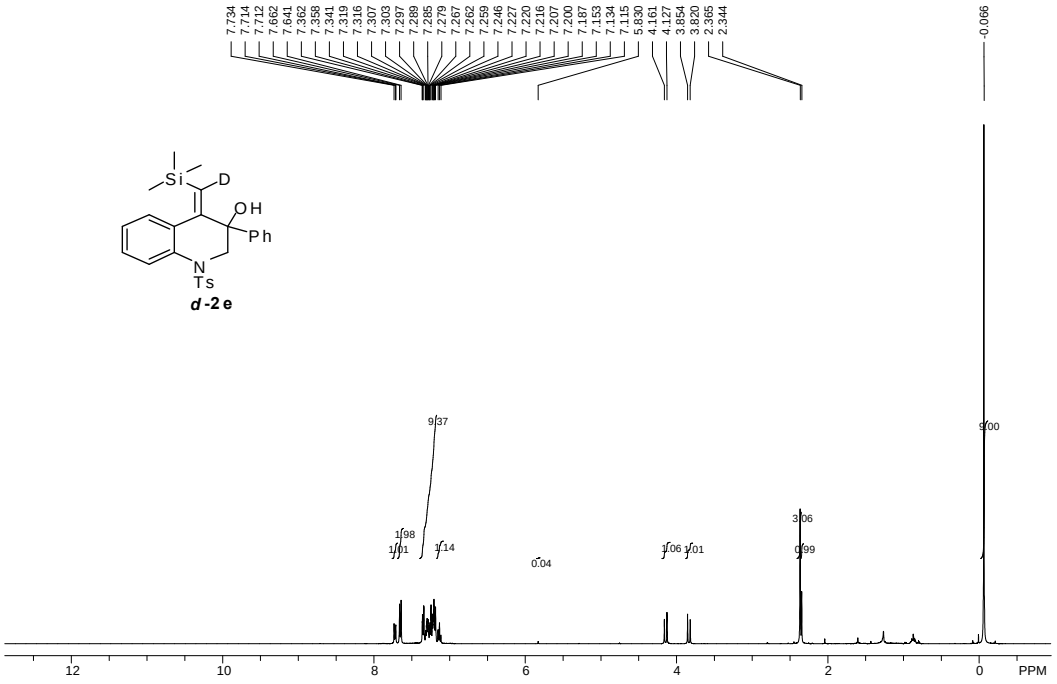


No.	PeakNo	R.Time	PeakHeight	PeakArea	PerCent
1	1	13.377	86572.3	2113305.8	8.9666
2	2	18.377	603943.5	21455358.2	91.0334
Total			690515.8	23568664.0	100.0000

V. Deuterium labeling studies



To a Schlenk tube were added $[Pd(dppp)(H_2O)_2](BF_4)_2$ (3.7 mg, 5 mol %), substrate **1e** (46.1 mg, 0.1 mmol) and *d*-ethanol (0.5 mL) successively under nitrogen atmosphere. The reaction mixture was stirred at 70 ^oC until the consumption of the substrate as monitored by TLC. Then the mixture was purified by flash column chromatography (ethyl acetate: petroleum ether = 1:5) to obtain the products **2e** and **3e**.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7.712
7.662
7.641
7.362
7.358
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7.319
7.316
7.307
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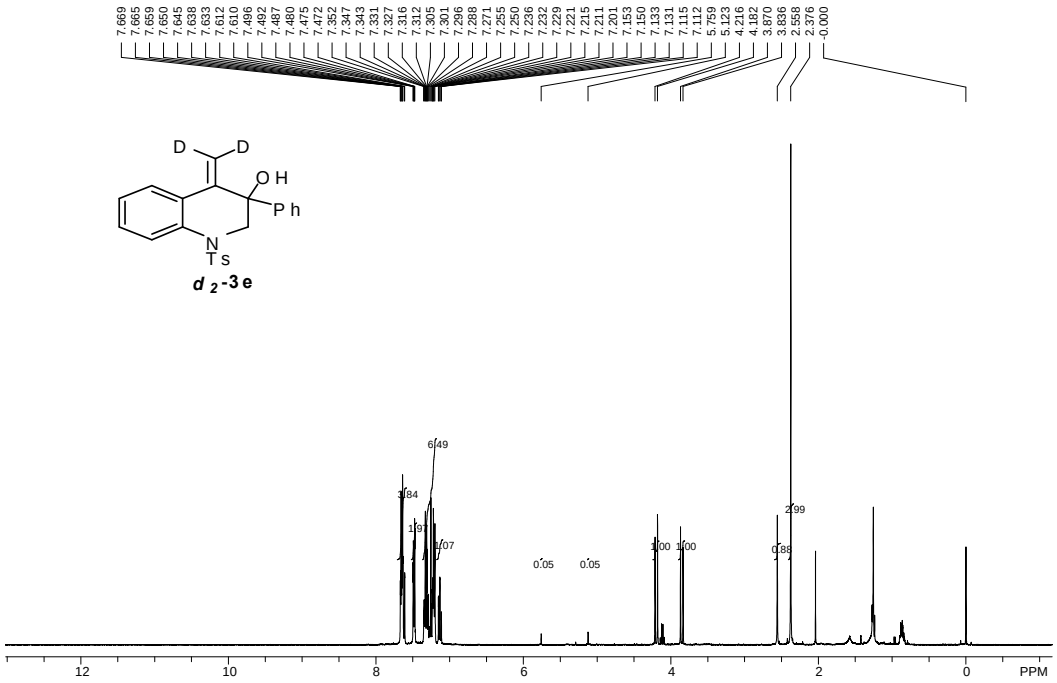
7.297
7.288
7.285
7.279
7.267
7.262
7.259
7.246
7.227
7.220
7.216
7.207
7.200
7.187
7.153
7.134
7.115
5.830
4.161
4.127
3.854
3.820
2.365
2.344
-0.066
Si
D
OH
Ph
N
Ts
d-2e
9.37
1.98
1.01
1.14
0.04
1.06
1.01
3.06
0.99
9.00
12
10
8
6
4
2
0
PPM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7.659
7.650
7.645
7.638
7.633
7.612
7.610
7.496
7.492
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7.475
7.472
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7.347
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7.131
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1.91
1.07
0.05
0.05
1.00
1.00
2.99
0.88
12
10
8
6
4
2
0
PPM

VI. References

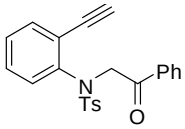
1 A. Fujii, E. Hagiwara, M. Sodeoka, *J. Am. Chem. Soc.* **1999**, *121*, 5450.

2 X. Yu, X. Lu, *Org. Lett.* **2009**, *11*, 4366.

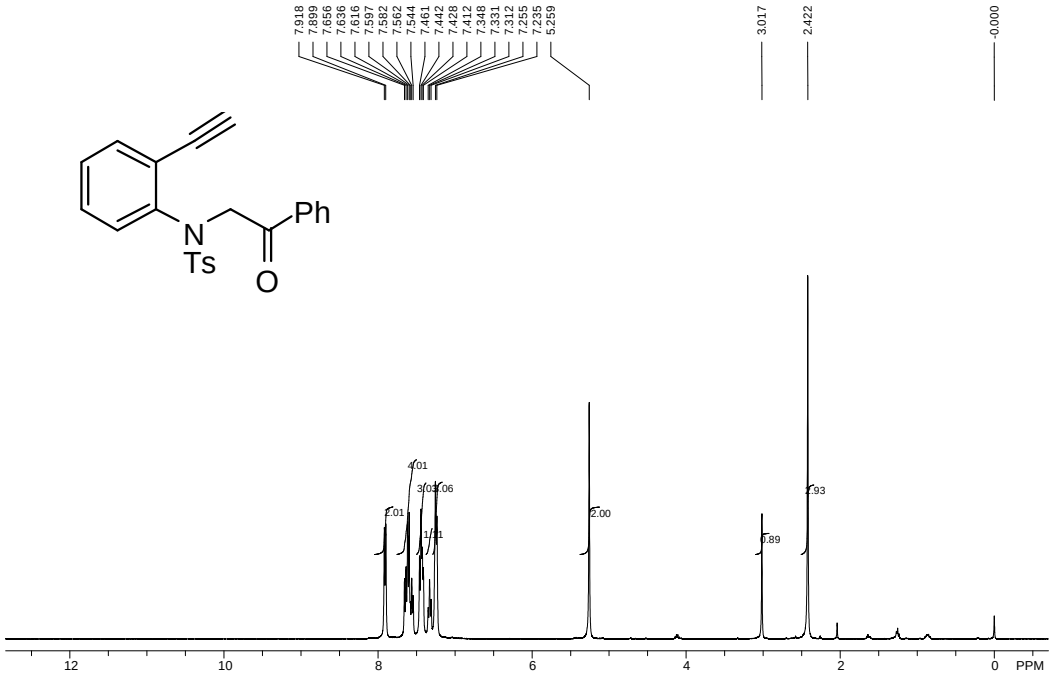
3 X. Han, X. Lu, *Org. Lett.* **2010**, *12*, 108.

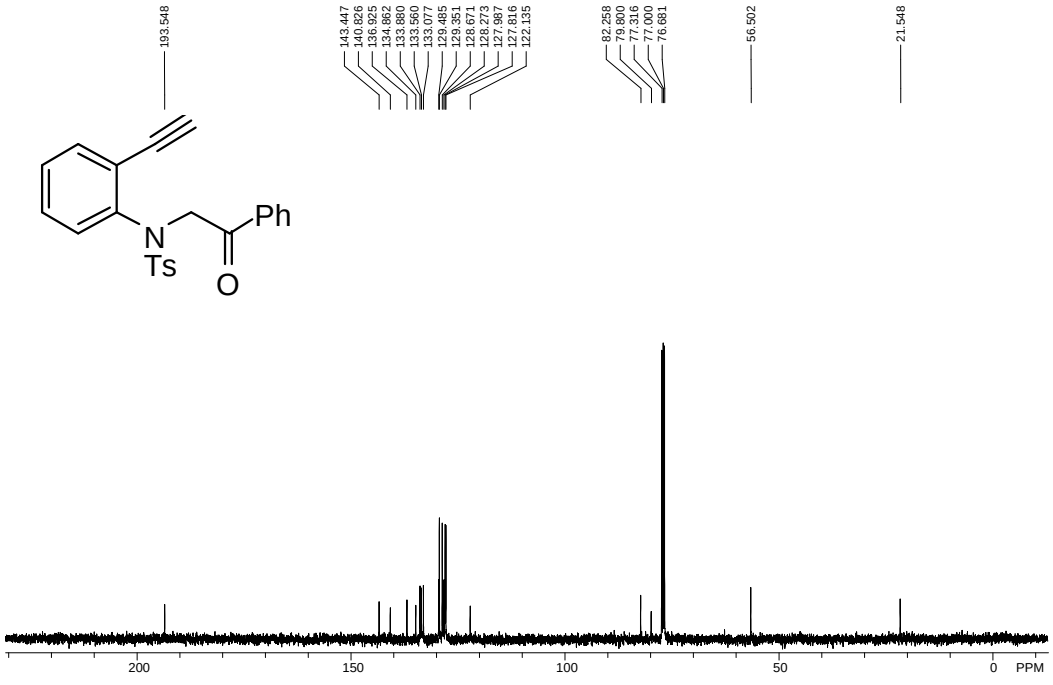
4 K. Shen, X. Han, X. Lu, *Org. Lett.* **2013**, *15*, 1732.

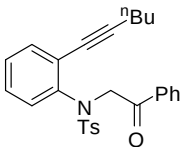
VII. Copies of ^{1}H, ^{13}C NMR spectra of the substrates and products



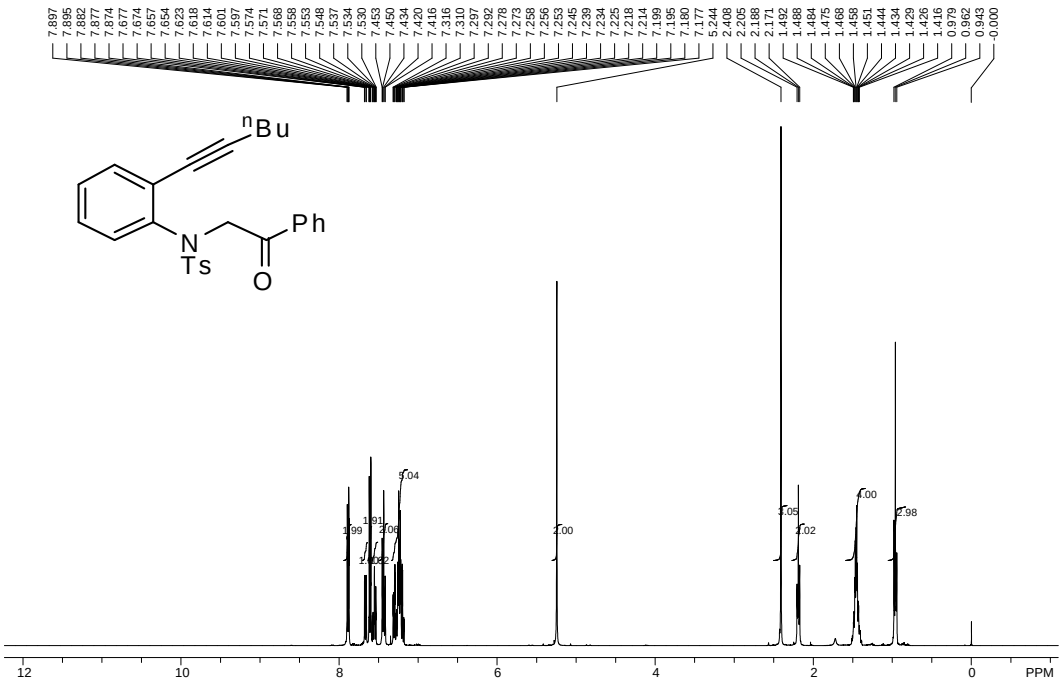
1a






nBu
Ph
N
Ts
O

1b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7.895
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7.623
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7.597
7.574
7.571
7.568
7.558
7.553
7.548
7.537
7.534
7.530
7.453
7.450
7.434
7.420
7.416
7.316
7.310
7.297
7.292
7.278
7.273
7.258
7.256
7.253
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1.488
1.484
1.475
1.468
1.458
1.451
1.444
1.434
1.429
1.426
1.416
0.979
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Ph
N
Ts
O
5.04
1.99
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2.02
4.00
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12
10
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4
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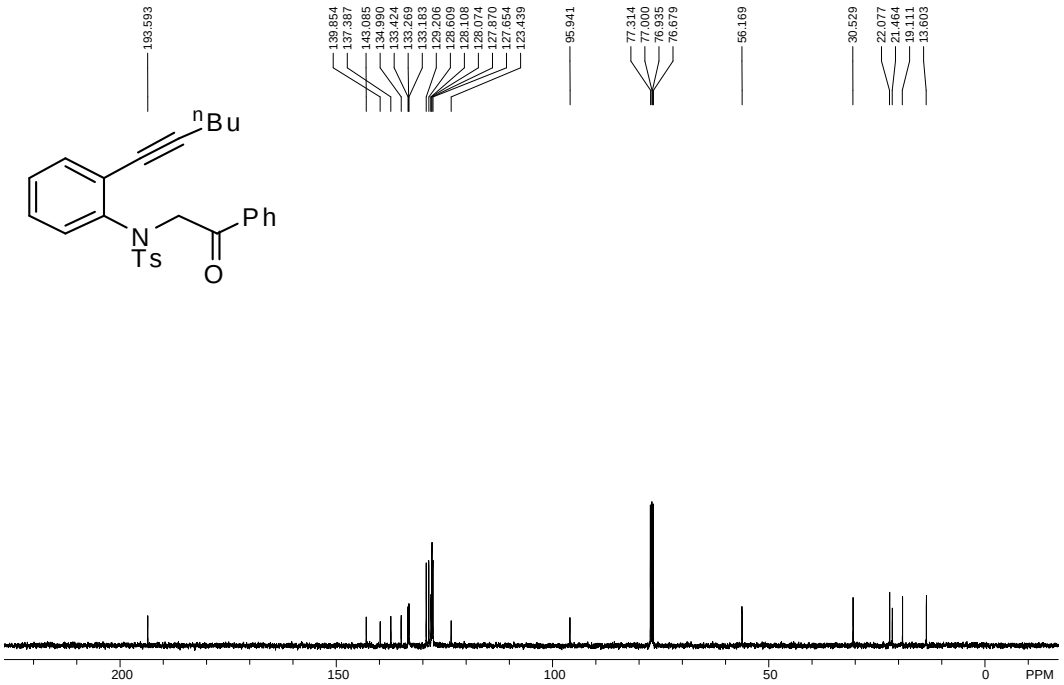
0
PPM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133.269
133.183
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128.609
128.108
128.074
127.870
127.654
123.439
95.941
77.314
77.000
76.935
76.679
56.169
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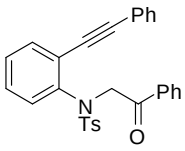
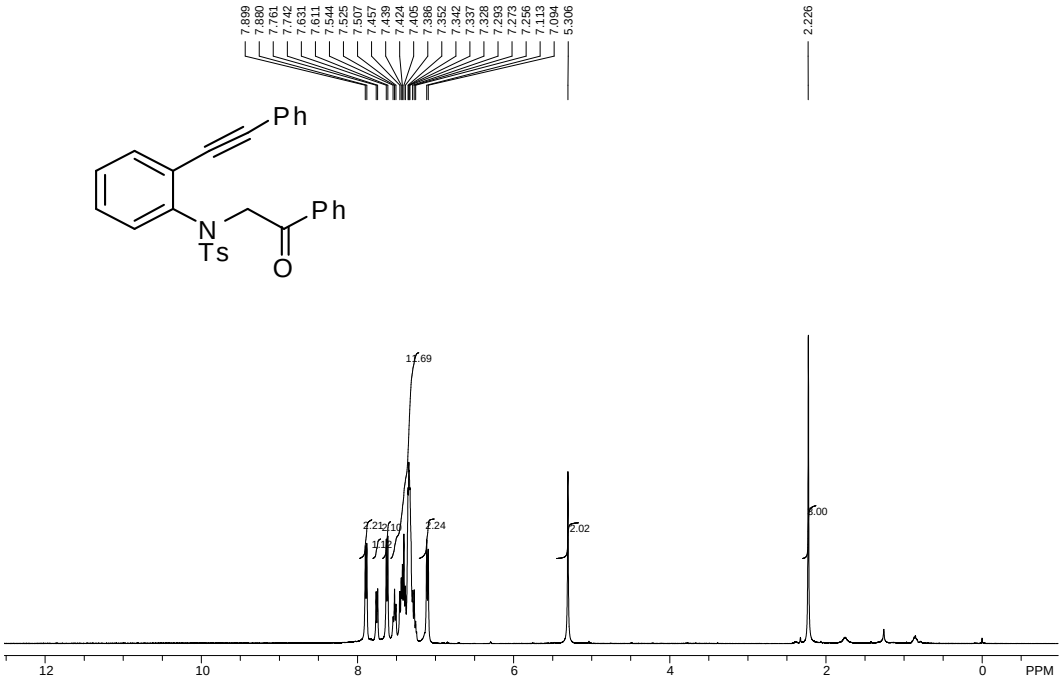
22.077
21.464
19.111
13.603
nBu
Ph
N
Ts
O
200
150
100
50
0
PPM


Ph
N
Ts
Ph
O

1c


7.899
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7.611
7.544
7.525
7.507
7.457
7.439
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7.405
7.386
7.352
7.342
7.337
7.328
7.293
7.273
7.256
7.113
7.094
5.306
2.226
Ph
N
Ts
Ph
O
11.69
2.21
2.10
1.13
2.24
2.02
3.00
12
10
8
6
4
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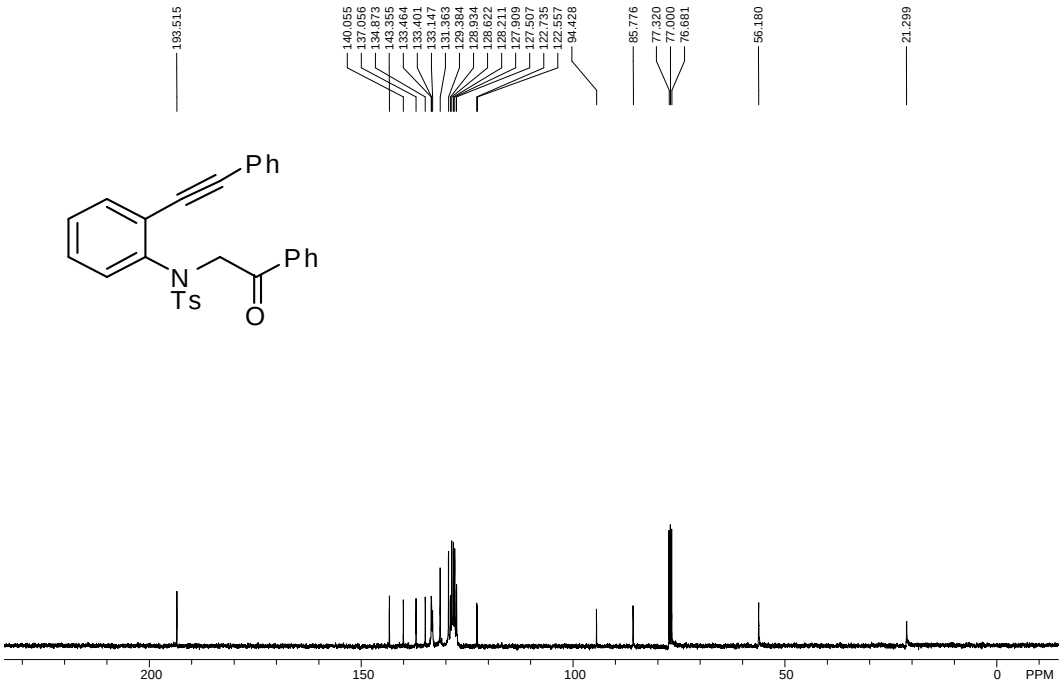
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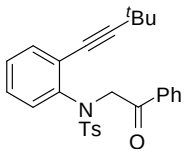
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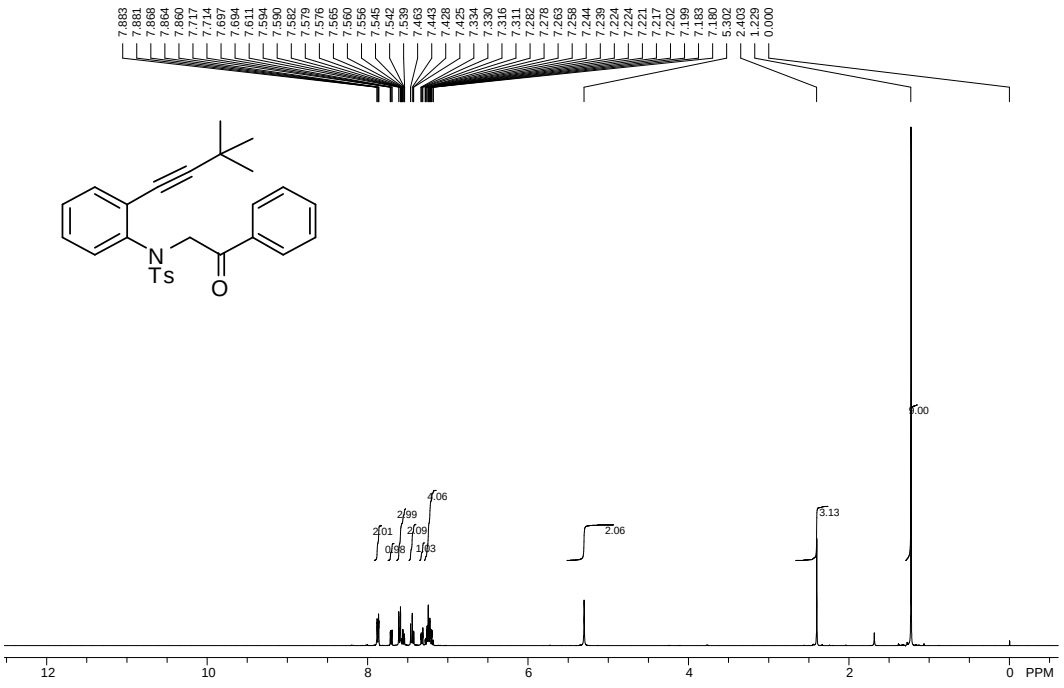
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7.202
7.199
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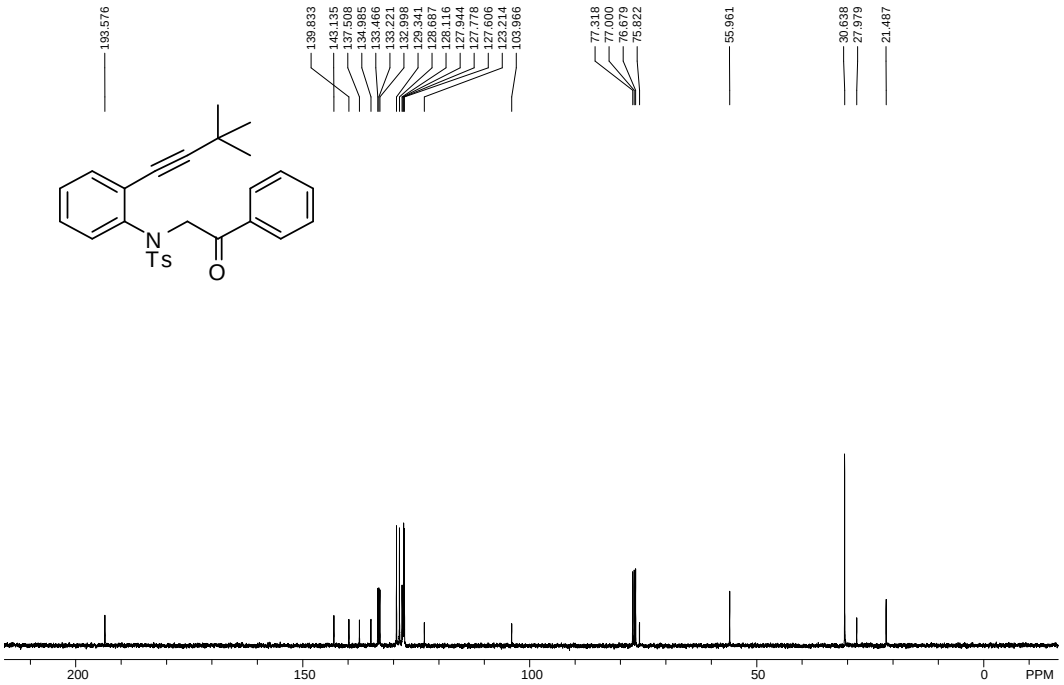
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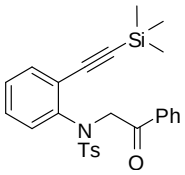
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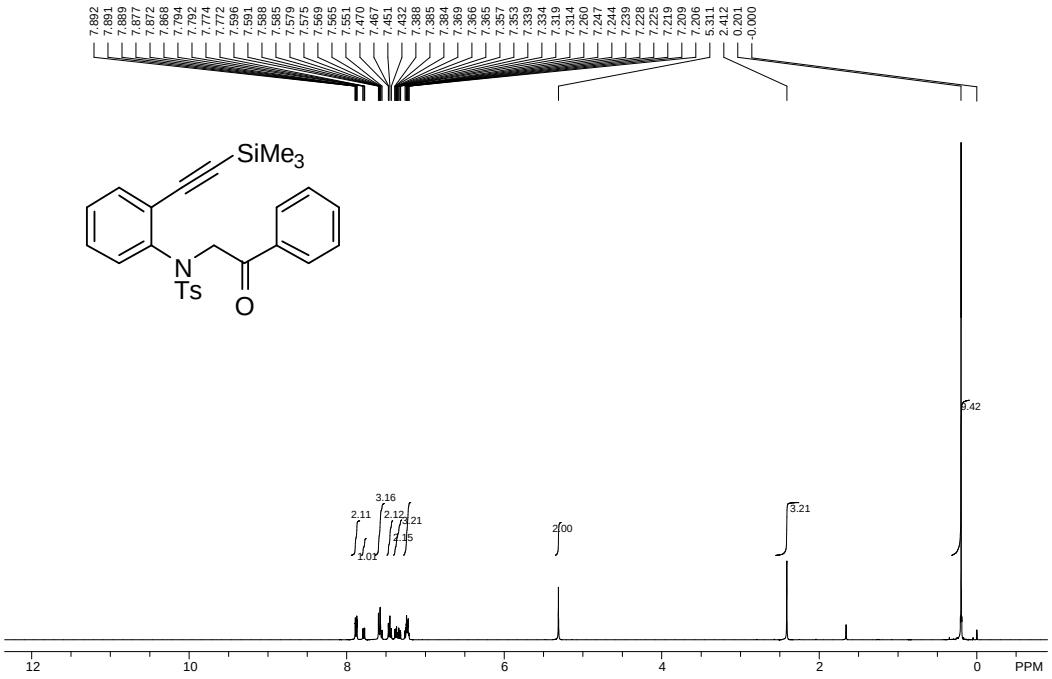
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Ts
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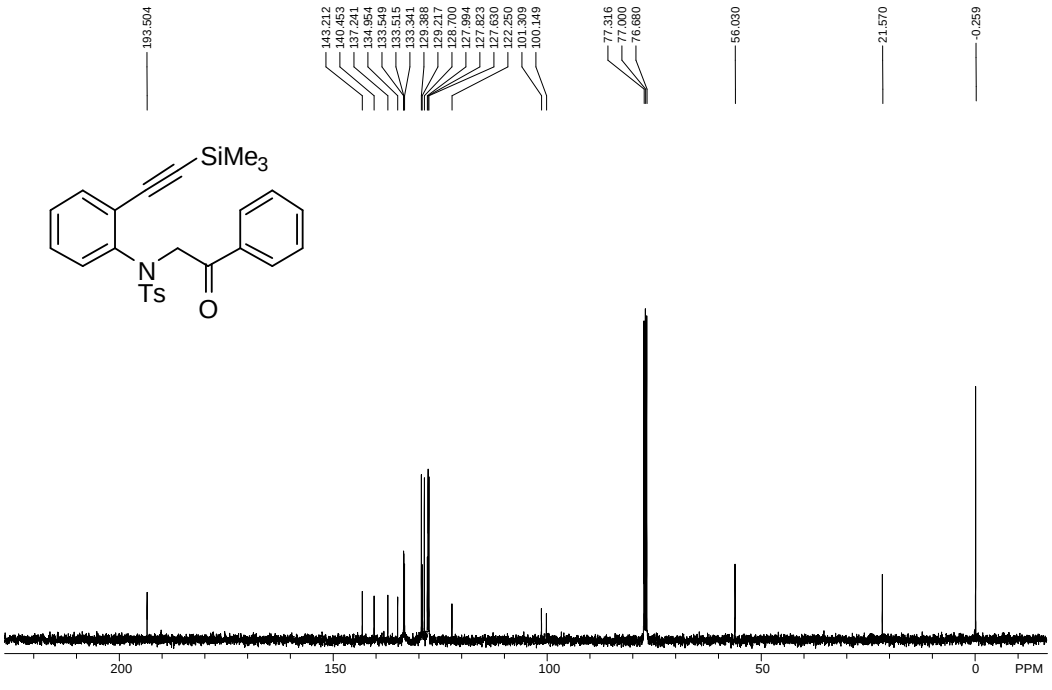
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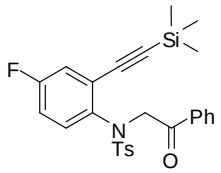
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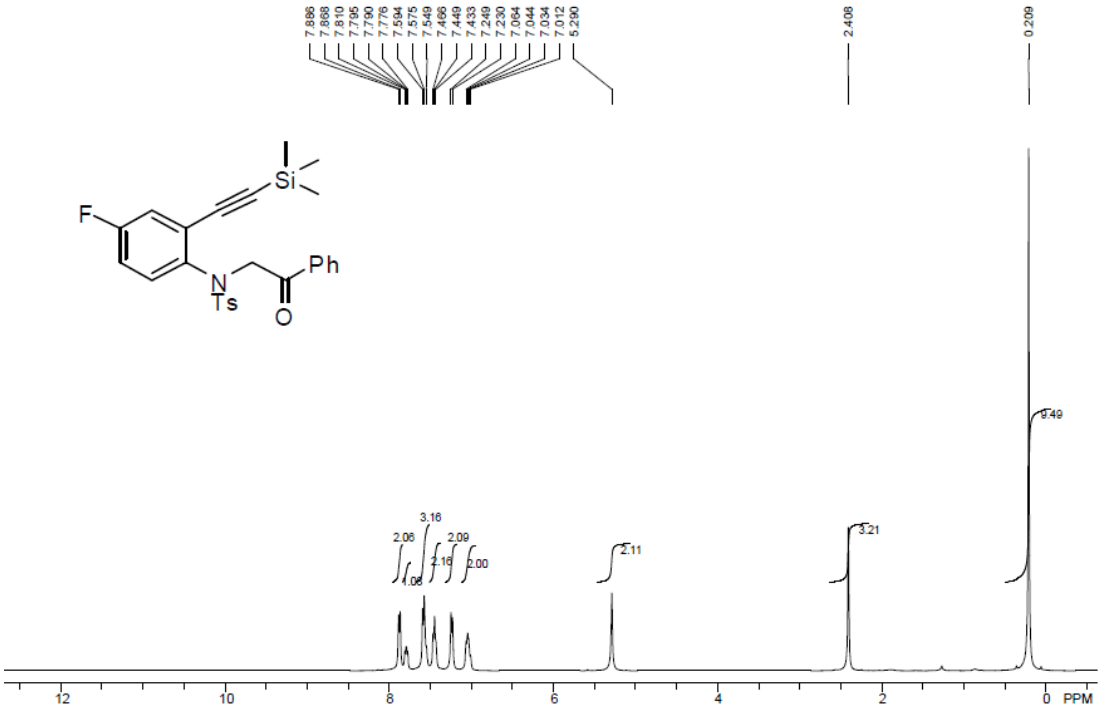
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7.230
7.064
7.044
7.034
7.012
5.290
2.408
0.209
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F
Ph
N
Ts
O
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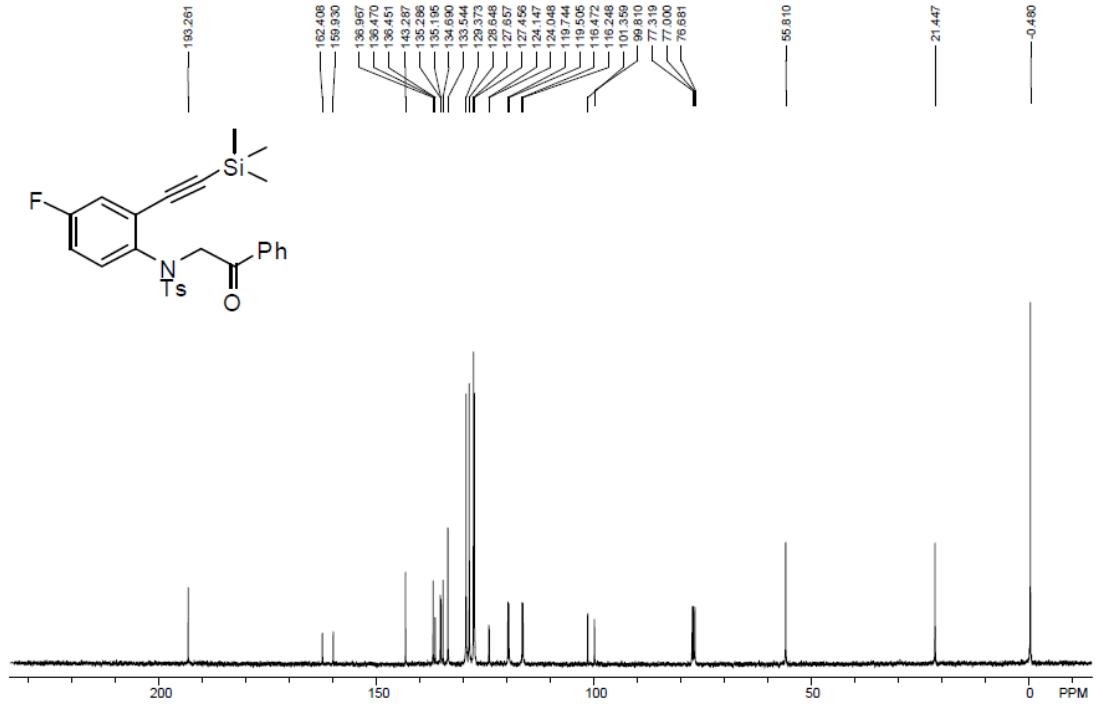
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127.456
124.147
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119.505
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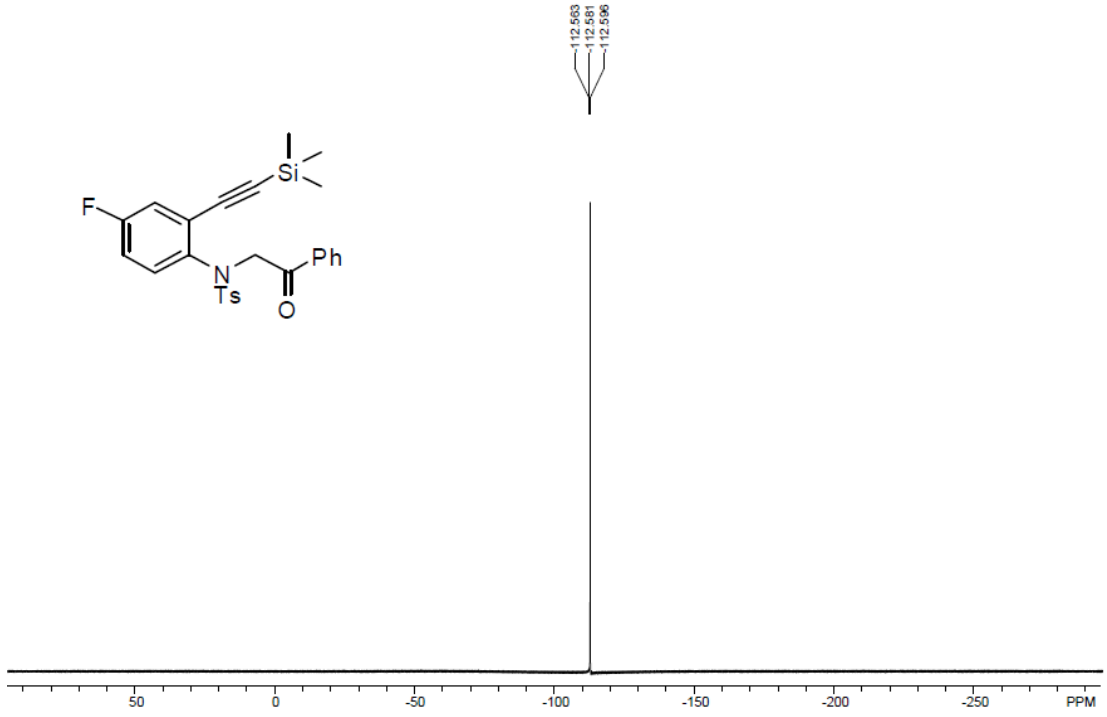
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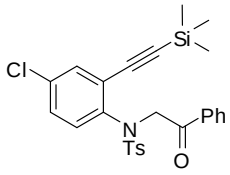
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Ph
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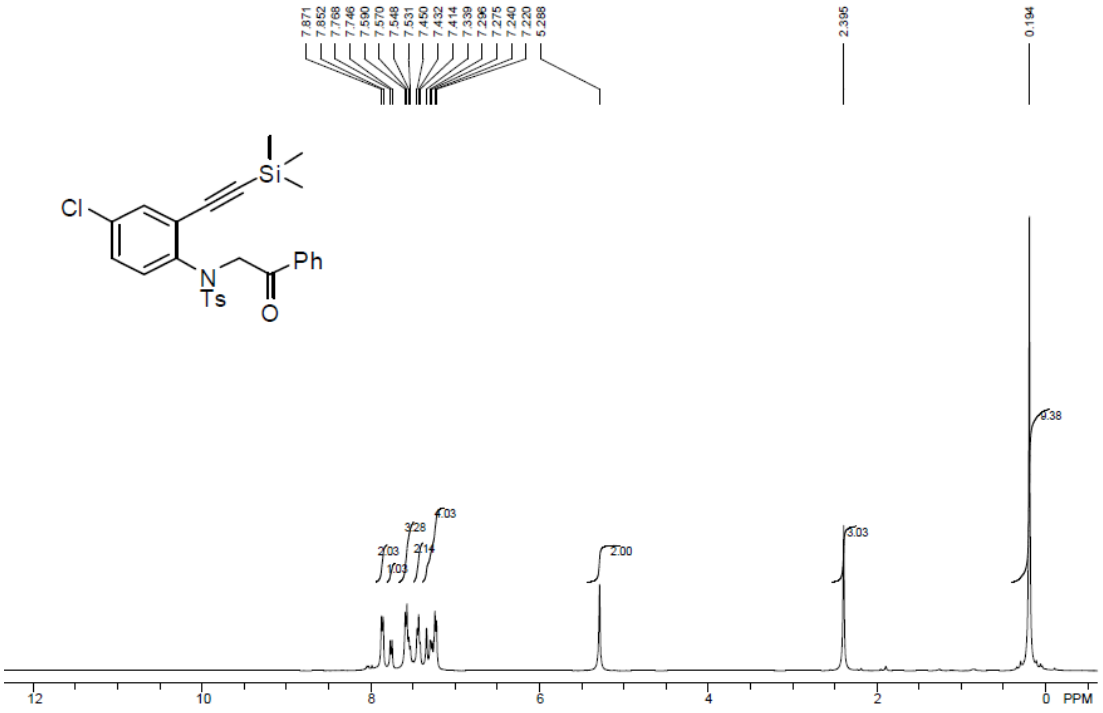
Ts
O

1g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7.240
7.220
5.288
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Cl
Ph
N
Ts
O
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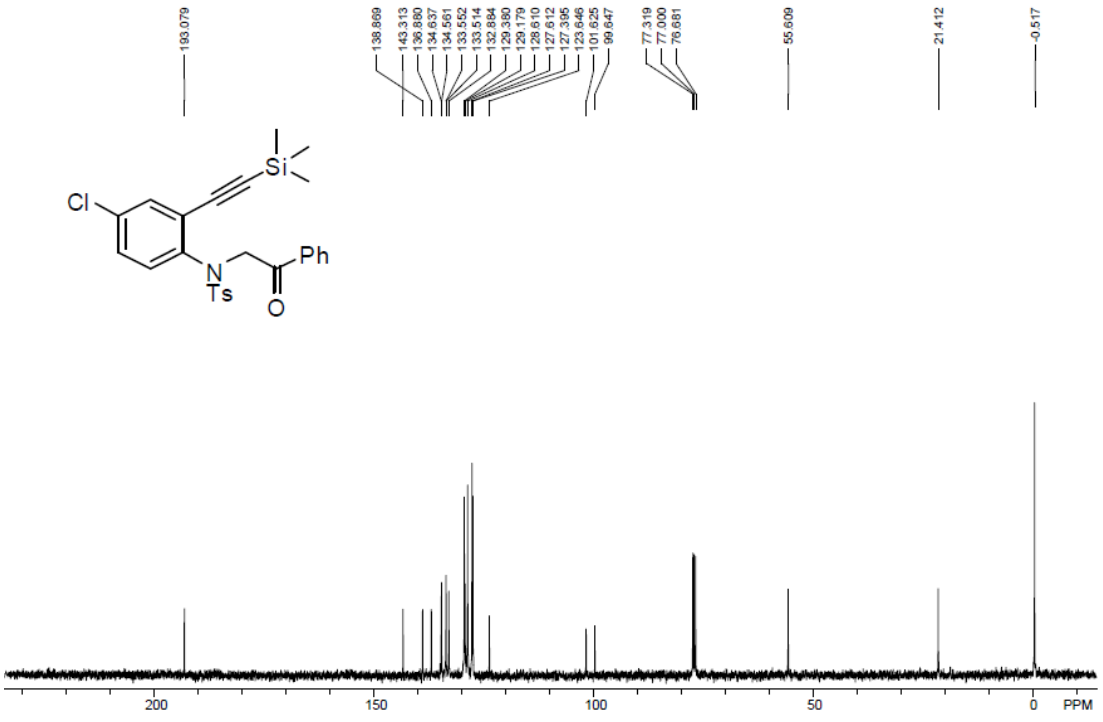
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127.612
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Ts
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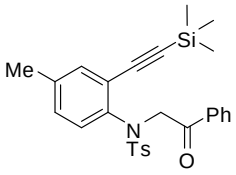
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Ph
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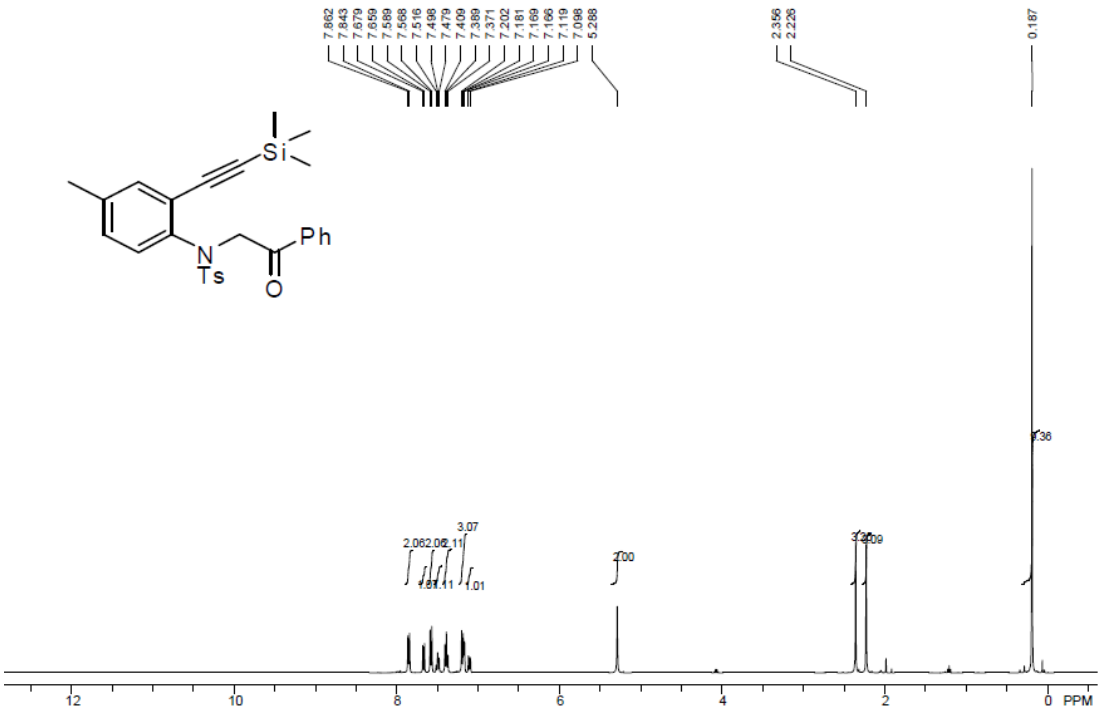
Ts
O

1h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7.166
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7.098
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2.356
2.226
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Ts
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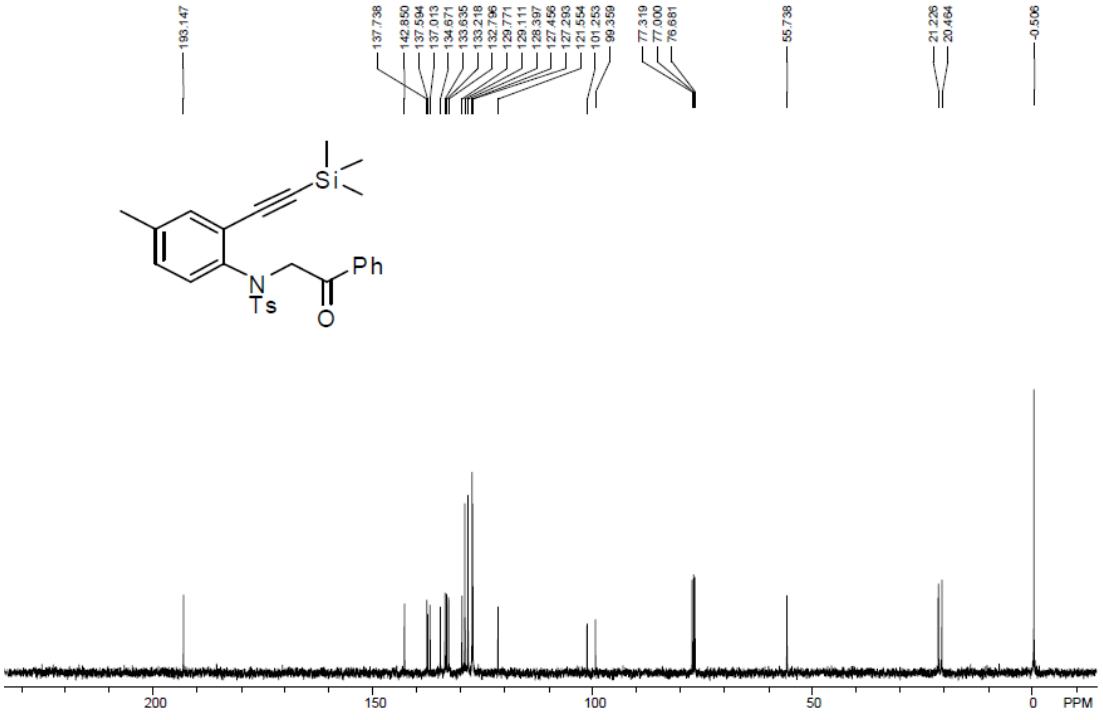
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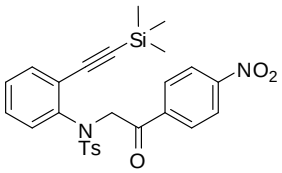
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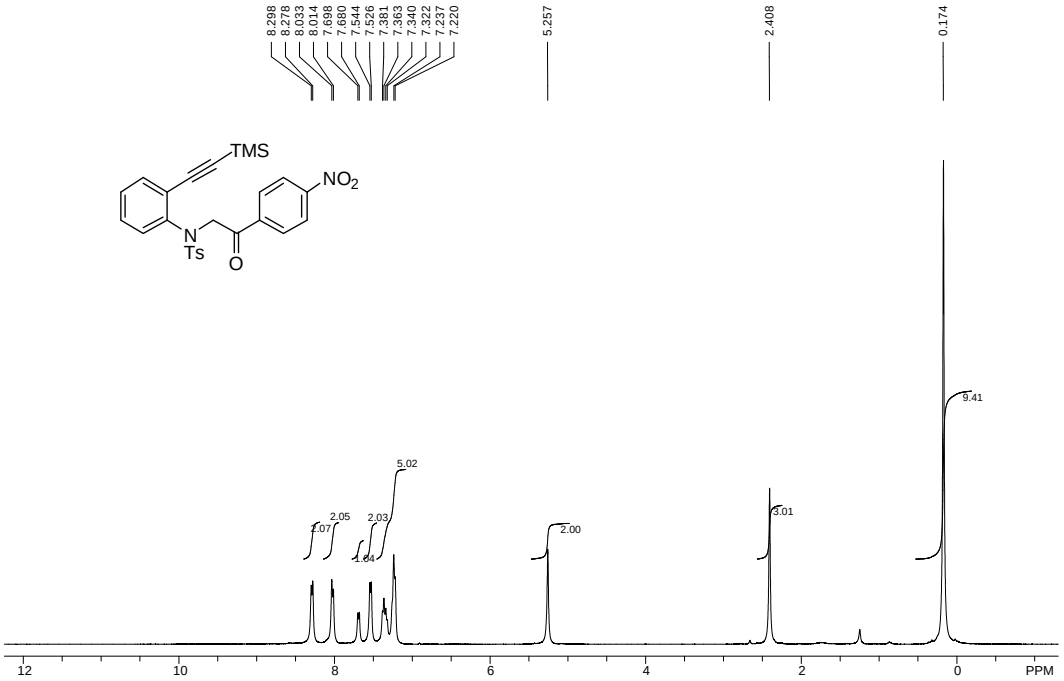
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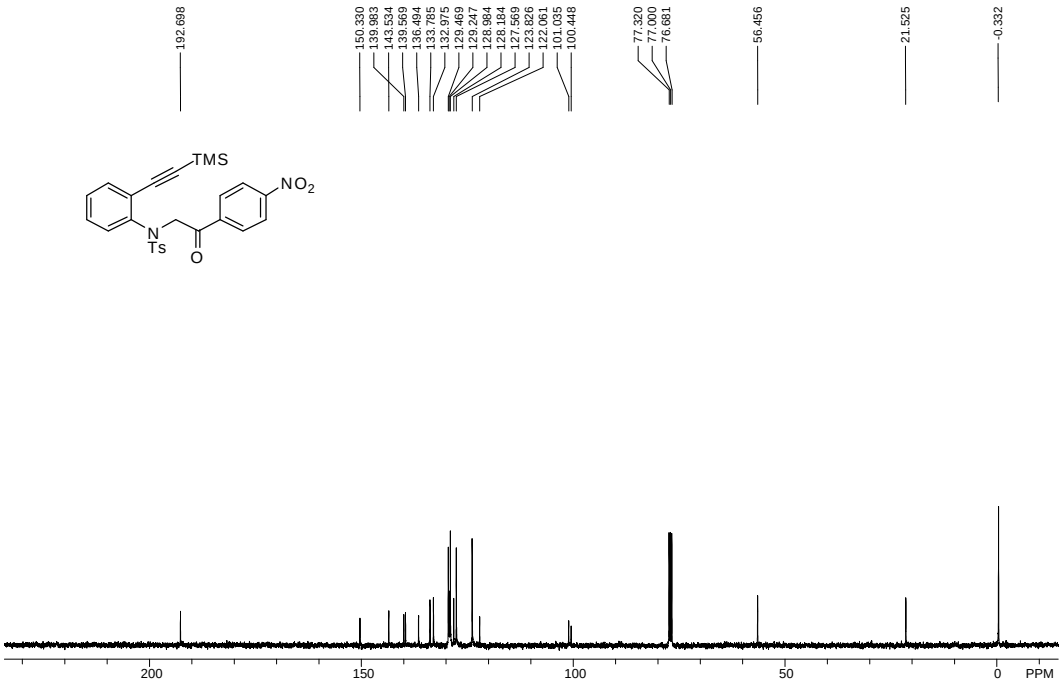
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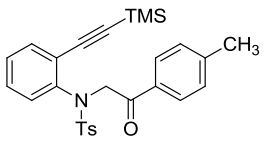
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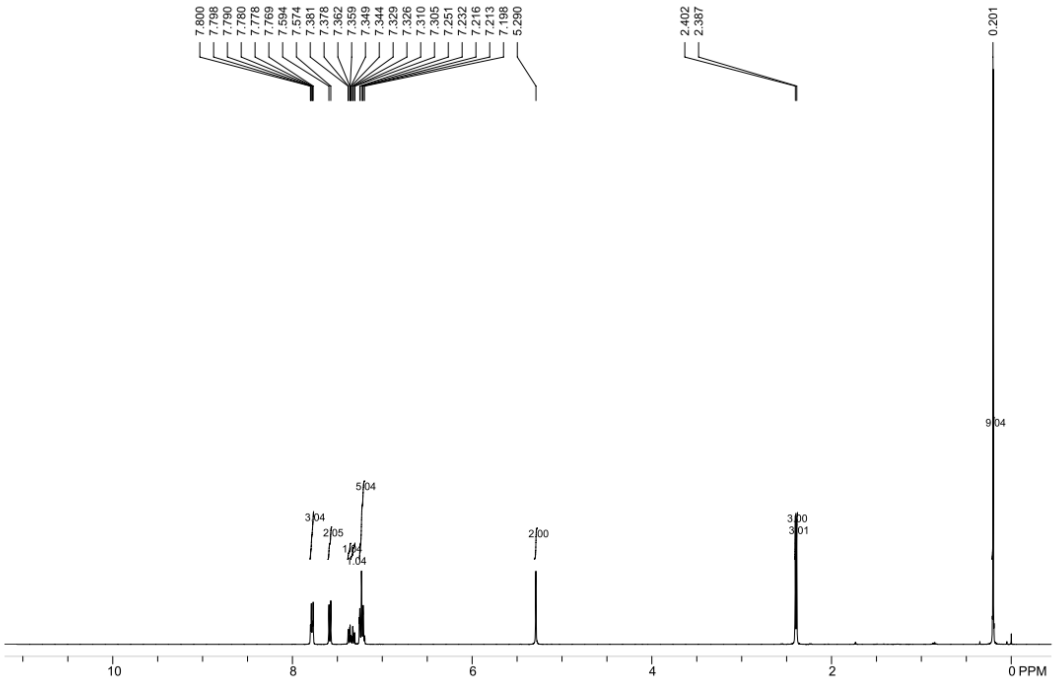
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7.216
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3.01
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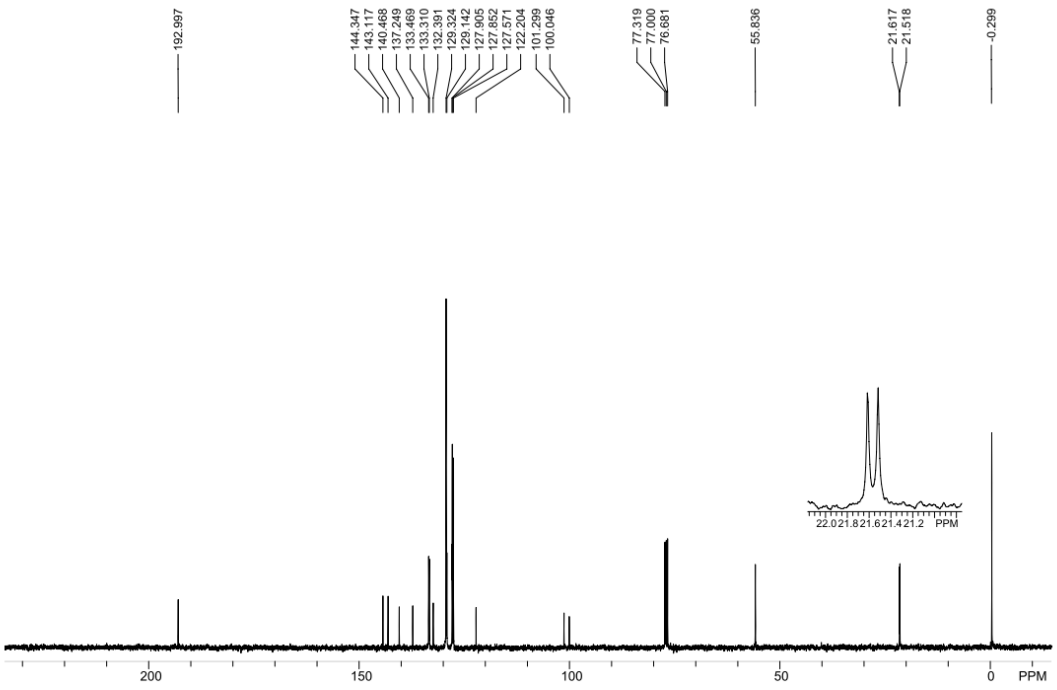
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100
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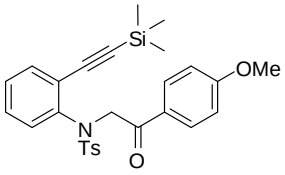
0 PPM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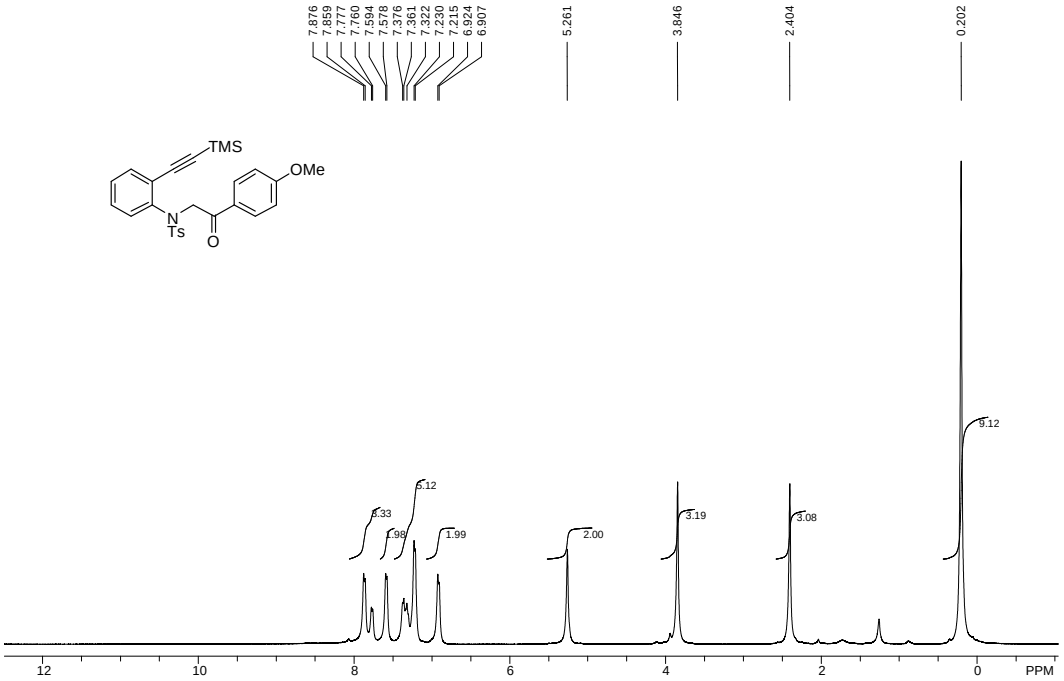
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OMe
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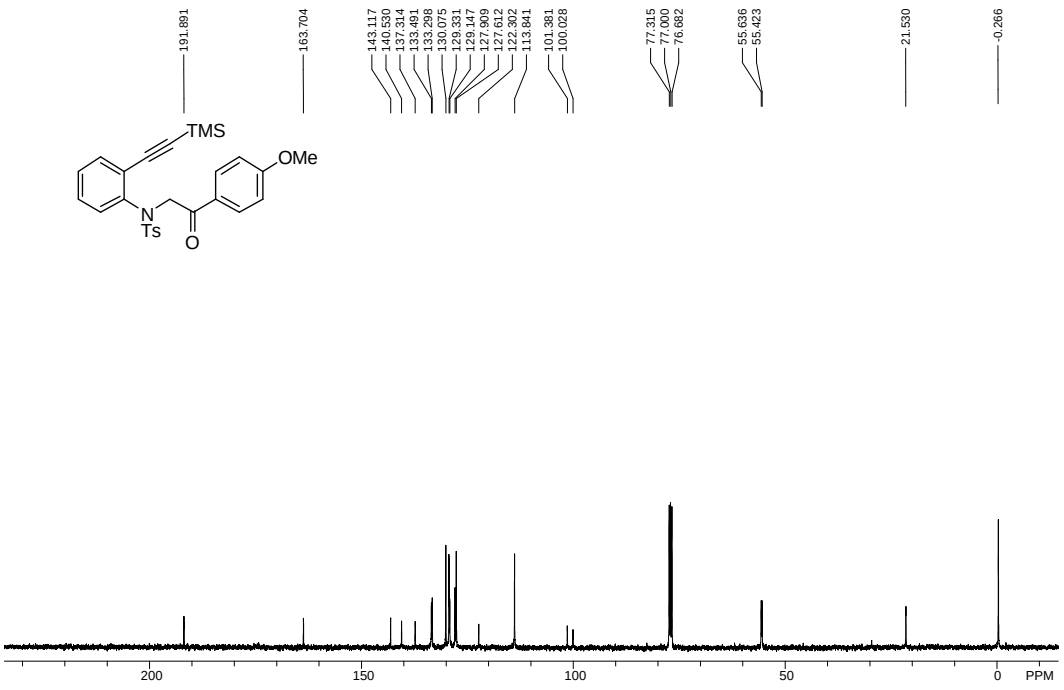
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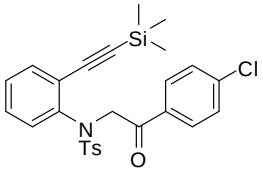
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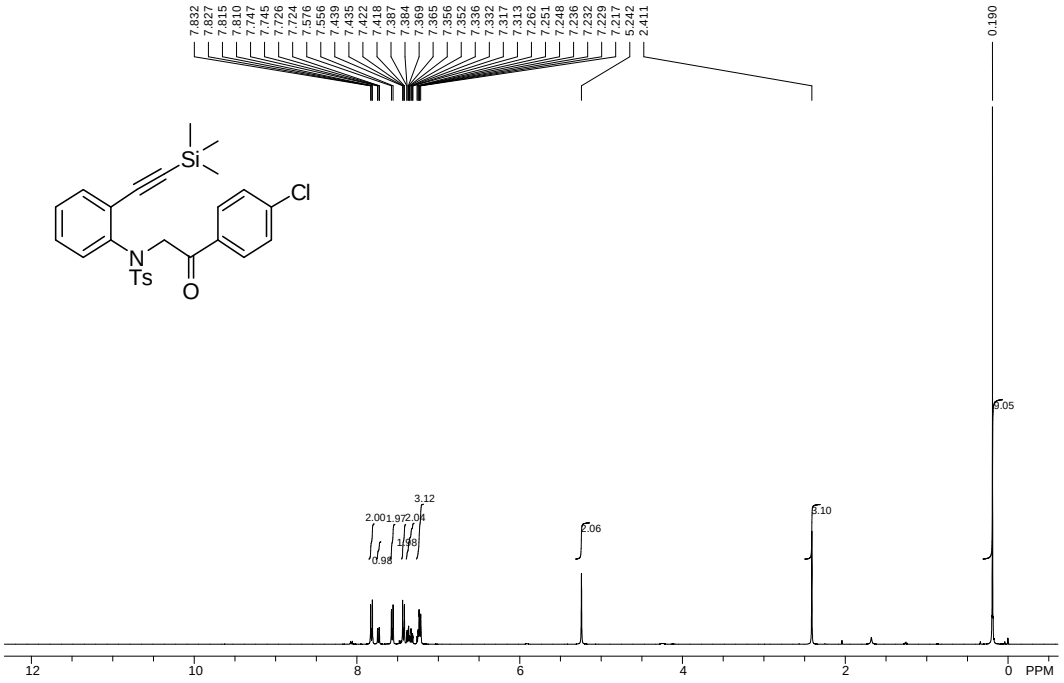
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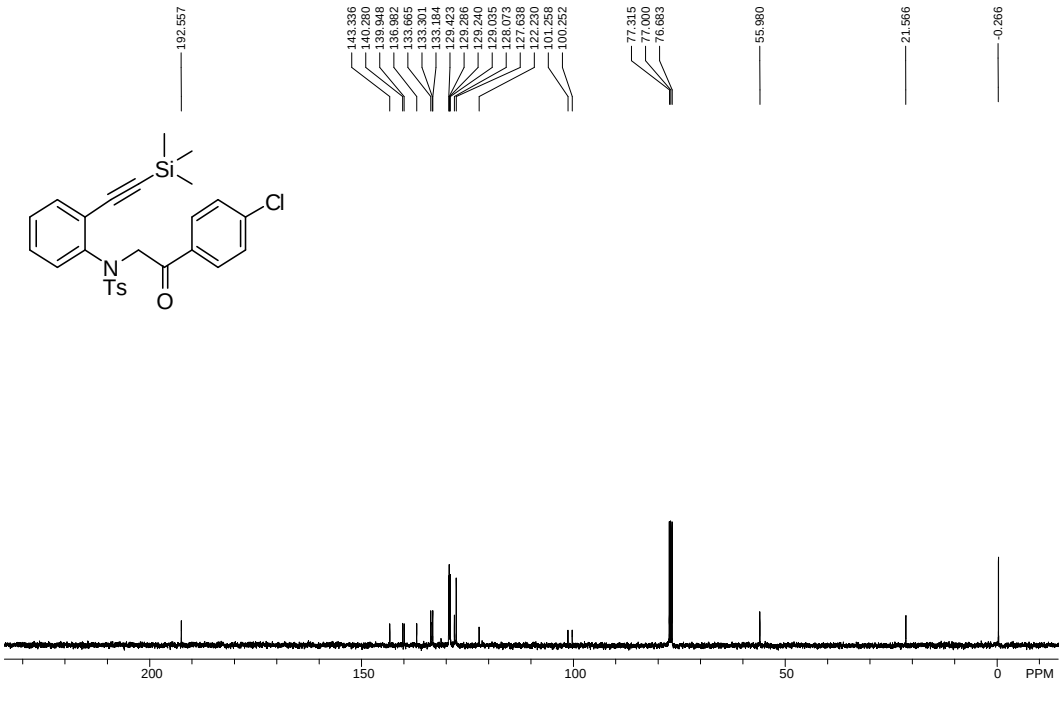
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127.638
122.230
101.258
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Cl
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Ts
O
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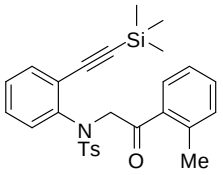
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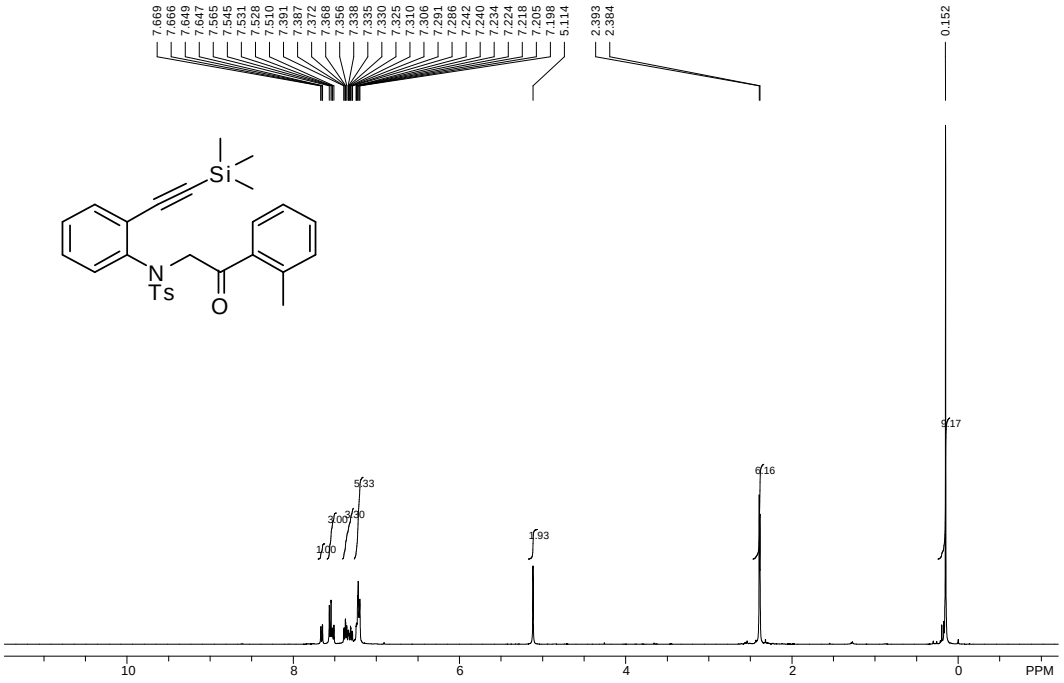
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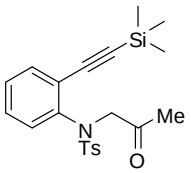
Si
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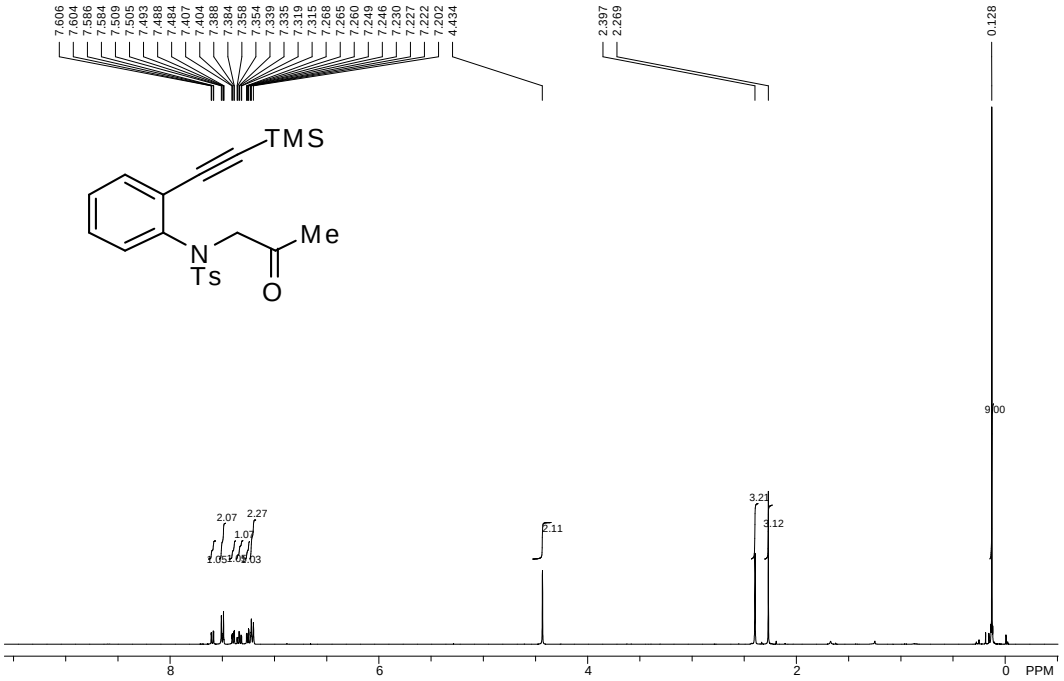
Ts
Me
O

1n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7.404
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7.364
7.358
7.354
7.339
7.335
7.319
7.315
7.268
7.265
7.260
7.249
7.246
7.230
7.227
7.222
7.202
4.434
2.397
2.269
0.128
TMS
N
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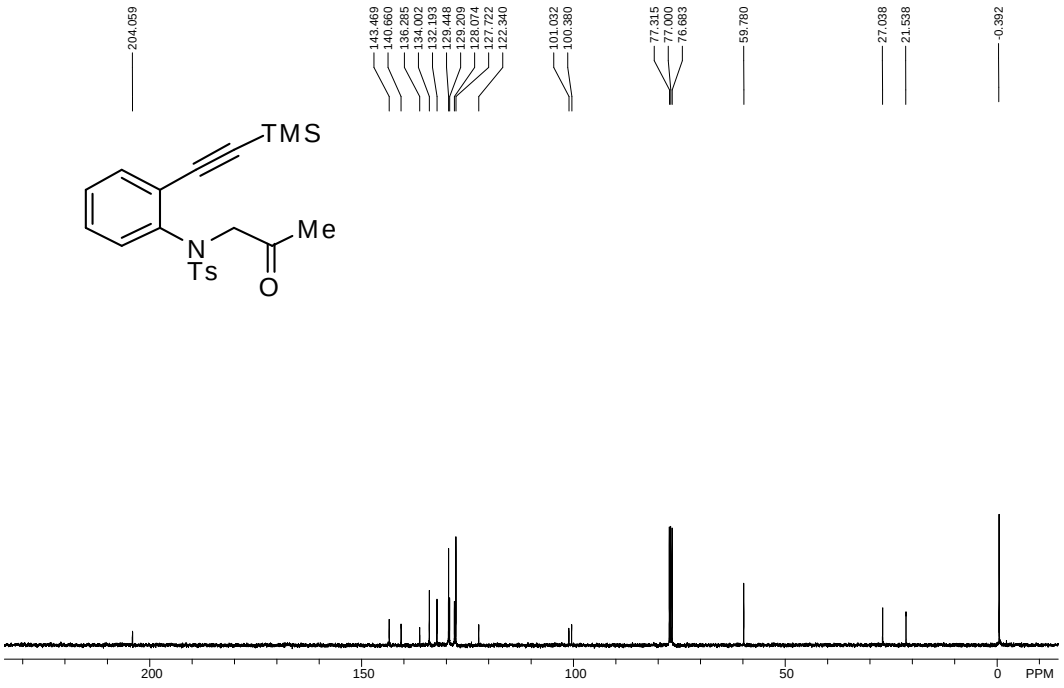
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129.209
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127.722
122.340
101.032
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77.000
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Ts
Me
O
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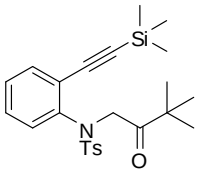
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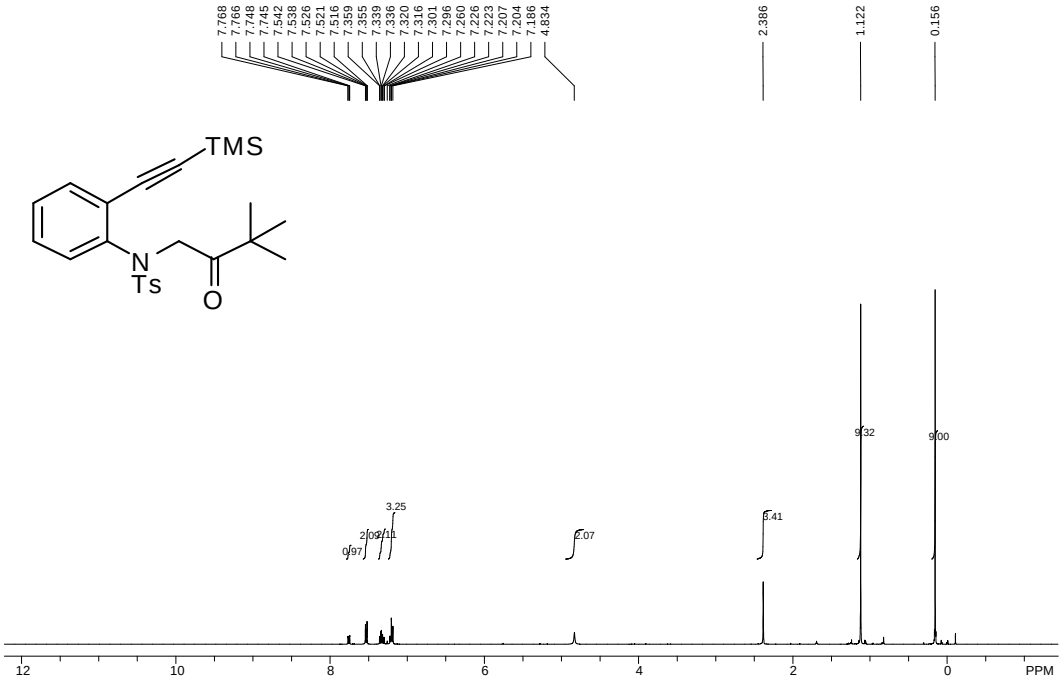
Ts
O

1o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7.521
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7.359
7.355
7.339
7.336
7.320
7.316
7.301
7.296
7.260
7.226
7.223
7.207
7.204
7.186
4.834
2.386
1.122
0.156
TMS
N
Ts
O
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10
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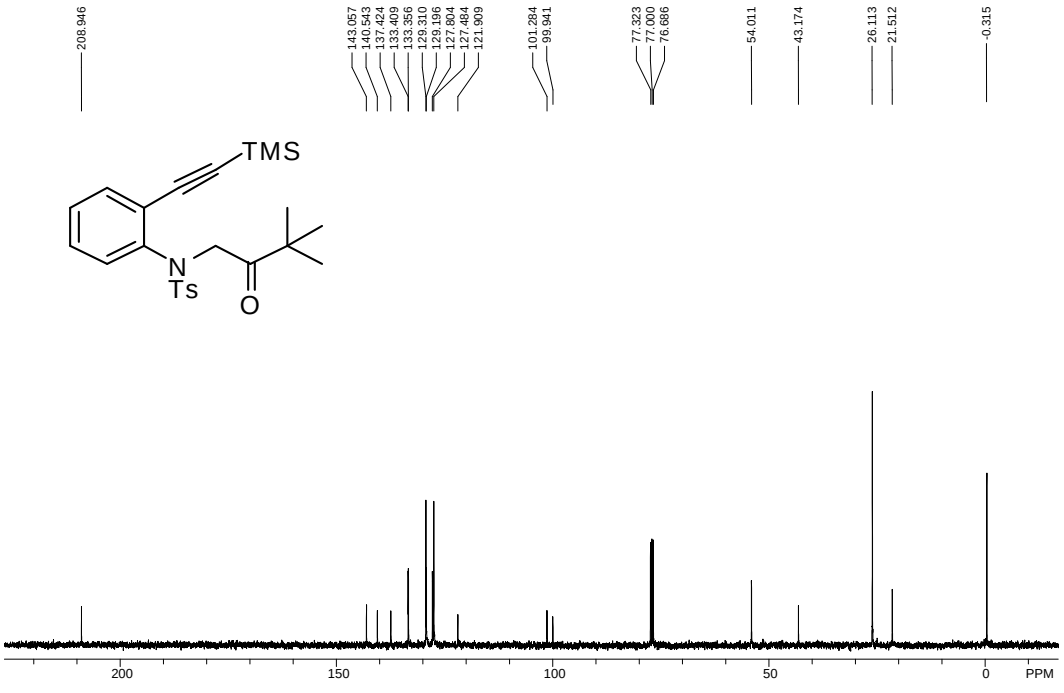
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129.196
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127.484
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101.284
99.941
77.323
77.000
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26.113
21.512
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TMS
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Ts
O
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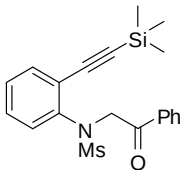
PPM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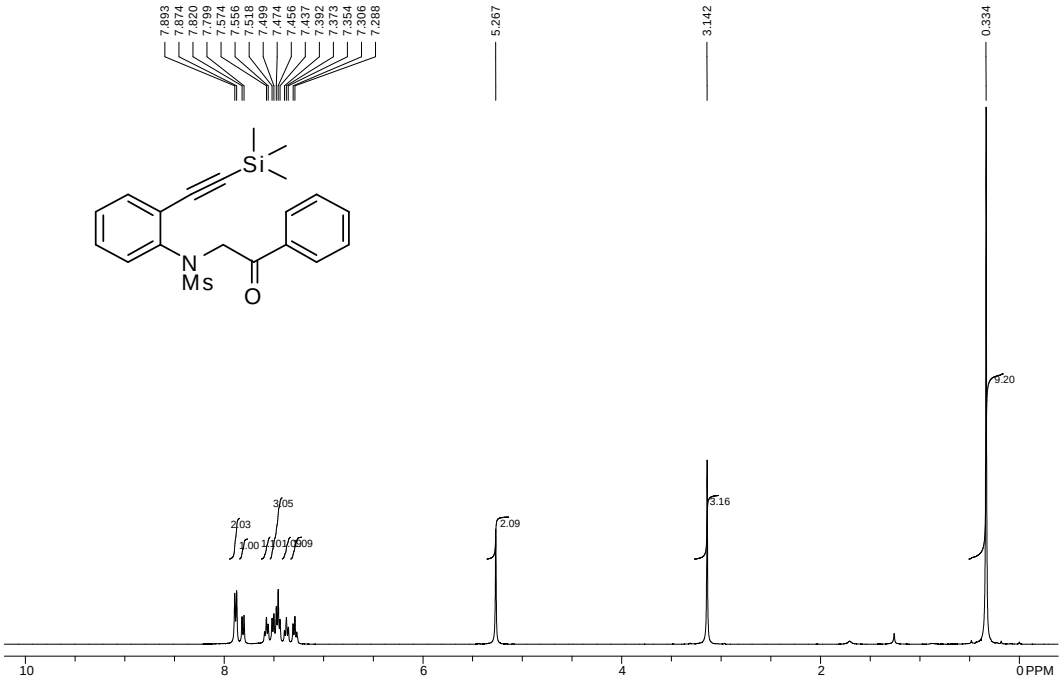
Ph
N
Ms
O

1p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7.499
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7.456
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7.373
7.354
7.306
7.288
5.267
3.142
0.334
Si
N
Ms
O
9.20
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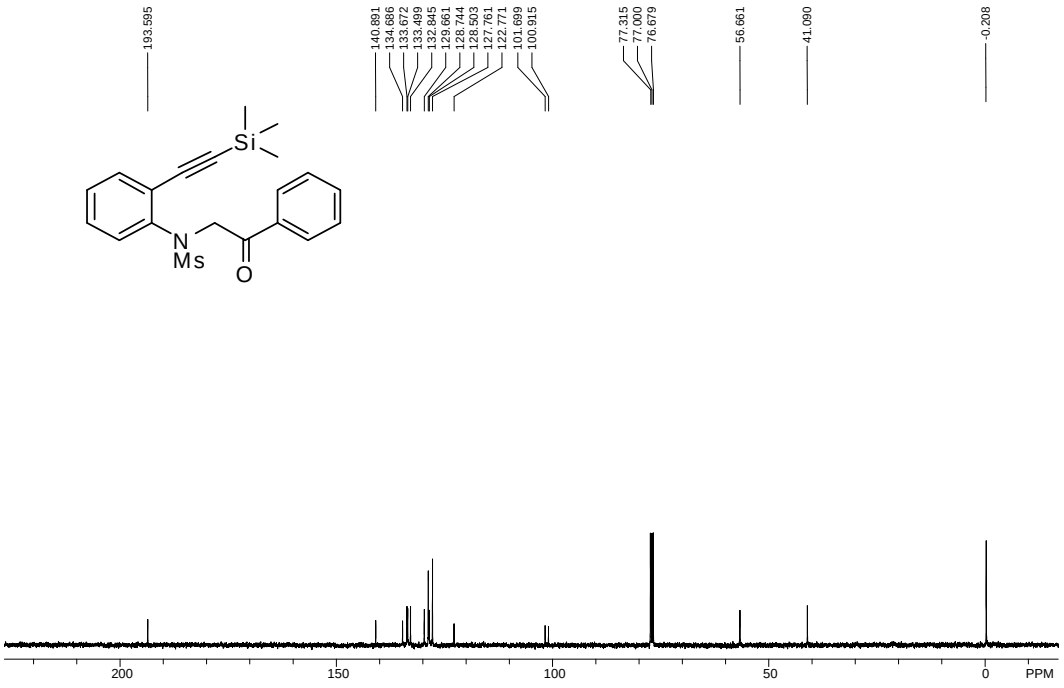
2.09
2.03
1.00
1.10
1.01
1.09
10
8
6
4
2
0 PPM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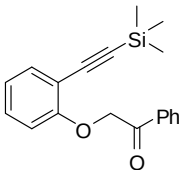
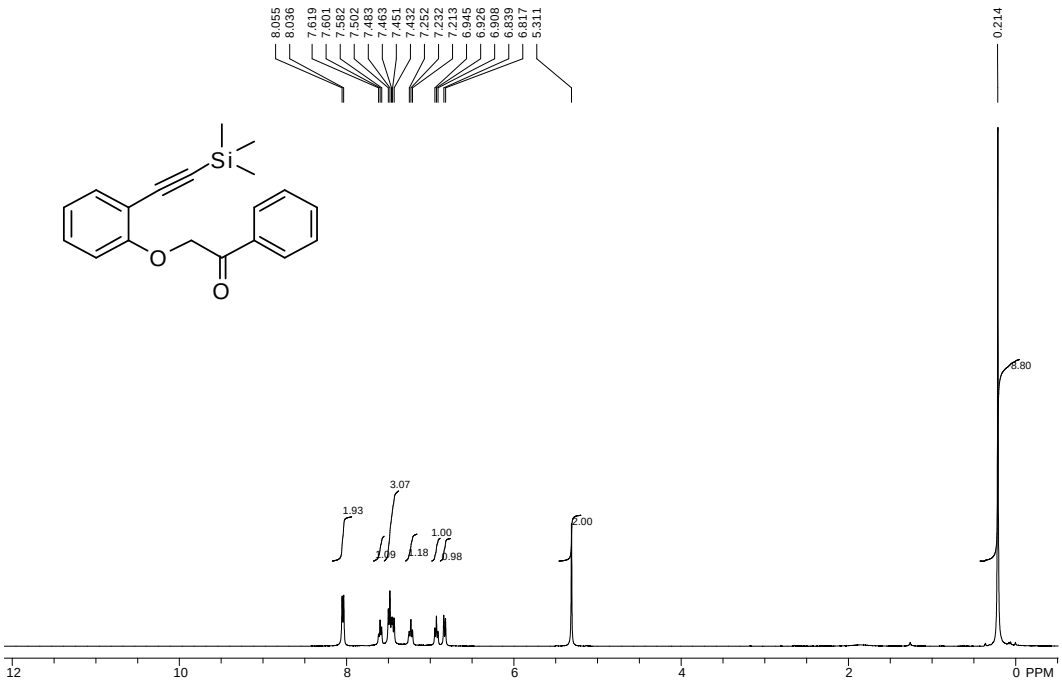
134.686
133.672
133.499
132.845
129.661
128.744
128.503
127.761
122.771
101.699
100.915
77.315
77.000
76.679
56.661
41.090
-0.208
Si
N
Ms
O
200
150
100
50
0
PPM


Si
Ph
O
O

1q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8.036
7.619
7.601
7.582
7.502
7.483
7.463
7.451
7.432
7.252
7.232
7.213
6.945
6.926
6.908
6.839
6.817
5.311
0.214
Si
O
O
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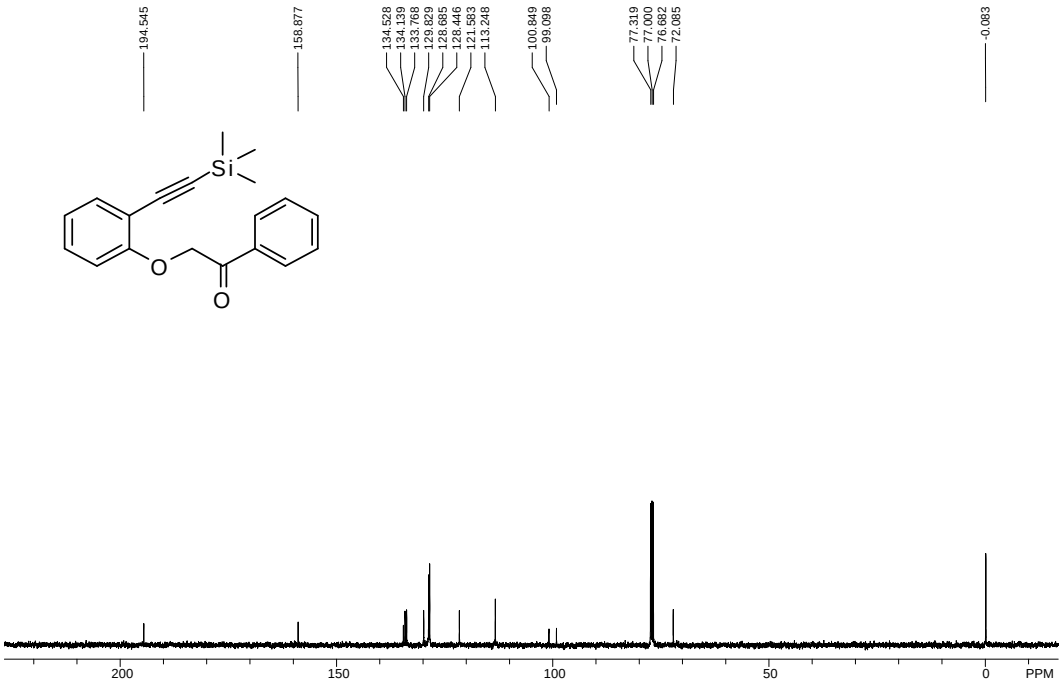
1.93
1.09
1.18
1.00
0.98
2.00
12
10
8
6
4
2
0 PPM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133.768
129.829
128.685
128.446
121.583
113.248
100.849
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77.000
76.682
72.085
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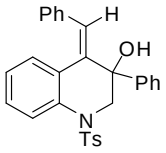
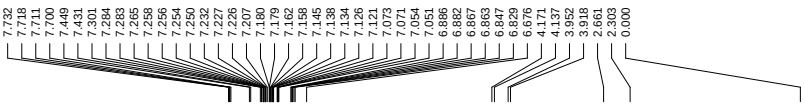
Si
O
O
200
150
100
50
0
PPM


Ph
H
OH
Ph
N
Ts

2c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7.284
7.283
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7.258
7.256
7.254
7.250
7.232
7.227
7.226
7.207
7.180
7.179
7.162
7.158
7.145
7.138
7.134
7.126
7.121
7.073
7.071
7.054
7.051
6.886
6.882
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6.829
6.676
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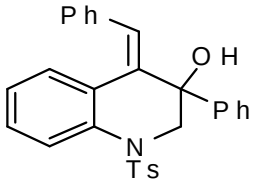
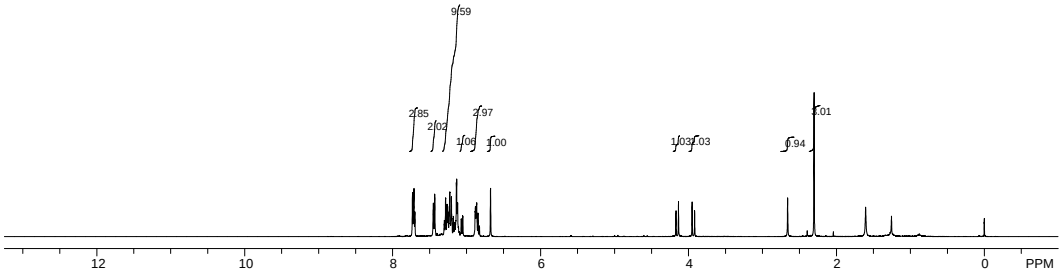
2.303
0.000


Ph
OH
Ph
N
Ts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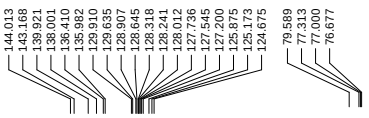
1.06
1.00
1.03
1.03
0.94
3.01
12
10
8
6
4
2
0
PPM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129.635
128.907
128.645
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128.241
128.012
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125.875
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77.000
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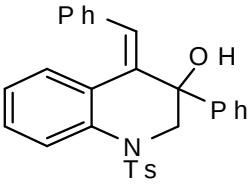
59.239
21.475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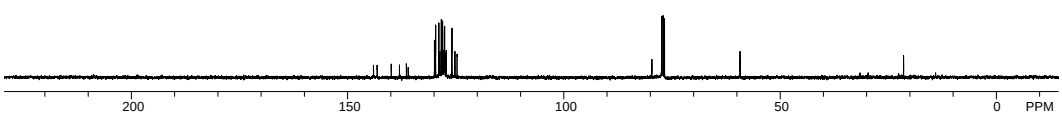
OH
Ph
N
Ts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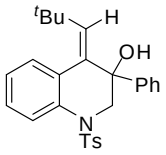
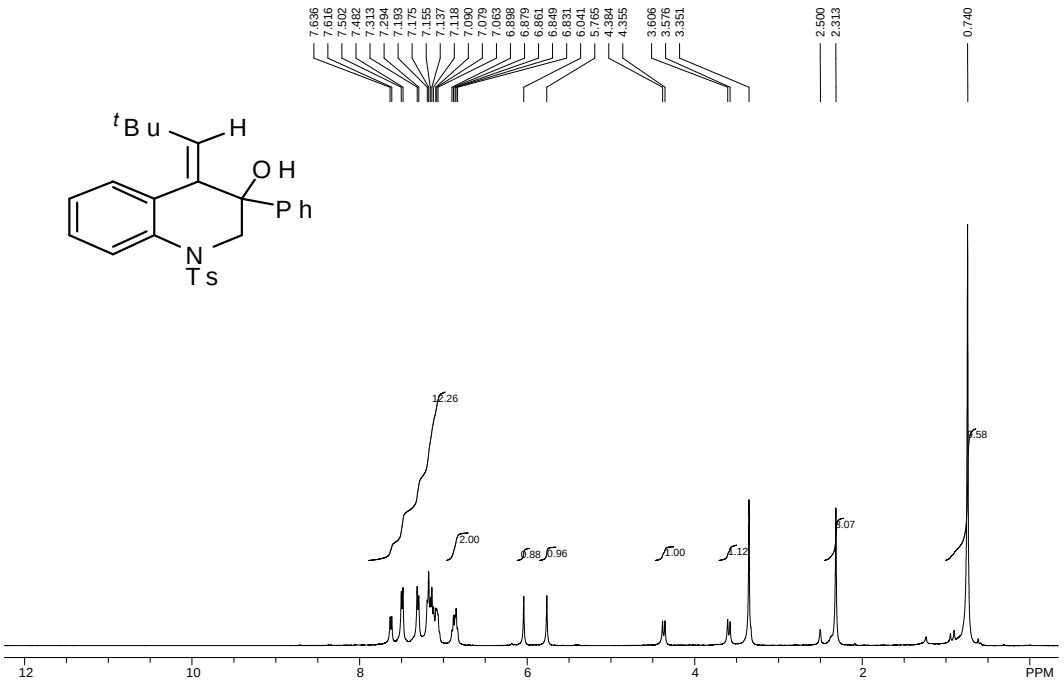
150
100
50
0
PPM


tBu
H
OH
Ph
N
Ts

2d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7.313
7.294
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7.137
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7.090
7.079
7.063
6.898
6.879
6.861
6.849
6.831
6.041
5.765
4.384
4.355
3.606
3.576
3.351
2.500
2.313
0.740
tBu
H
OH
Ph
N
Ts
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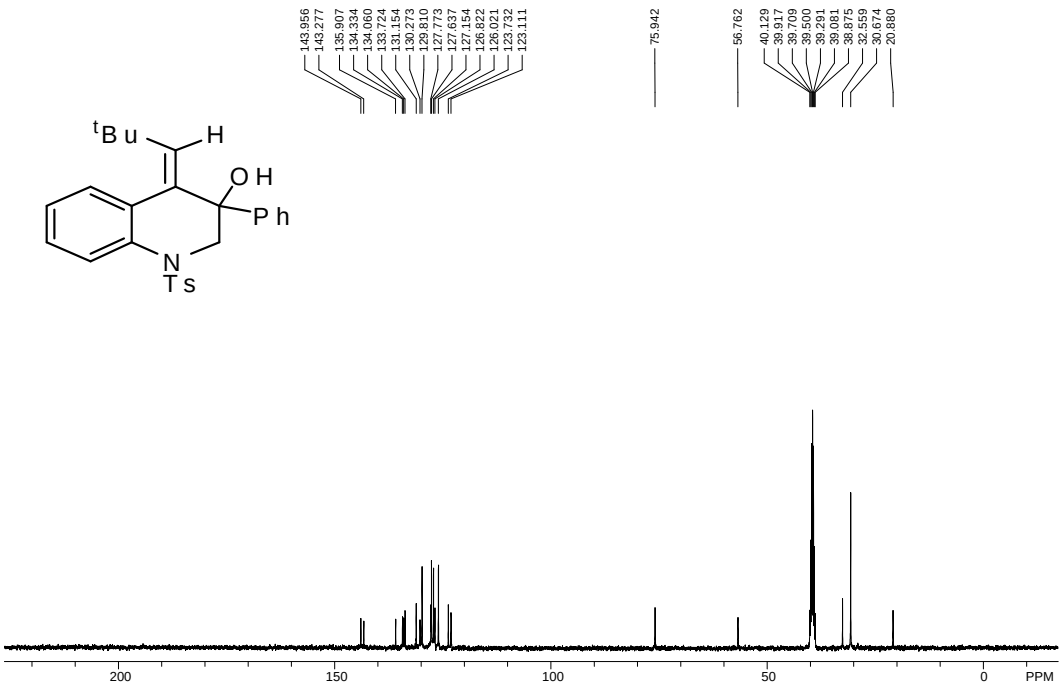
4
2
PPM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130.273
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129.773
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126.822
126.021
123.732
123.111
75.942
56.762
40.129
39.917
39.709
39.500
39.291
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30.674
20.880
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OH
Ph
N
Ts
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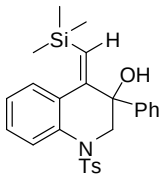
150
100
50
0
PPM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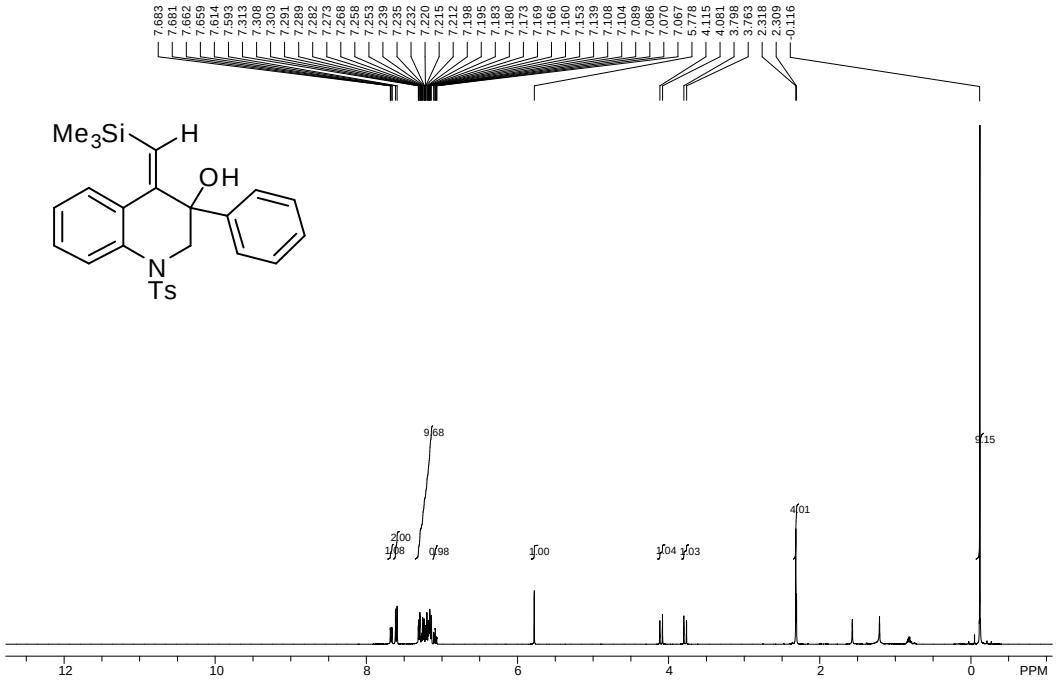
OH
Ph
N
Ts

2e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7.308
7.303
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7.289
7.282
7.273
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7.258
7.253
7.239
7.235
7.232
7.220
7.215
7.212
7.198
7.195
7.183
7.180
7.173
7.169
7.166
7.160
7.153
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7.108
7.104
7.089
7.086
7.070
7.067
5.778
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4.081
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3.763
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2.309
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H
OH
N
Ts
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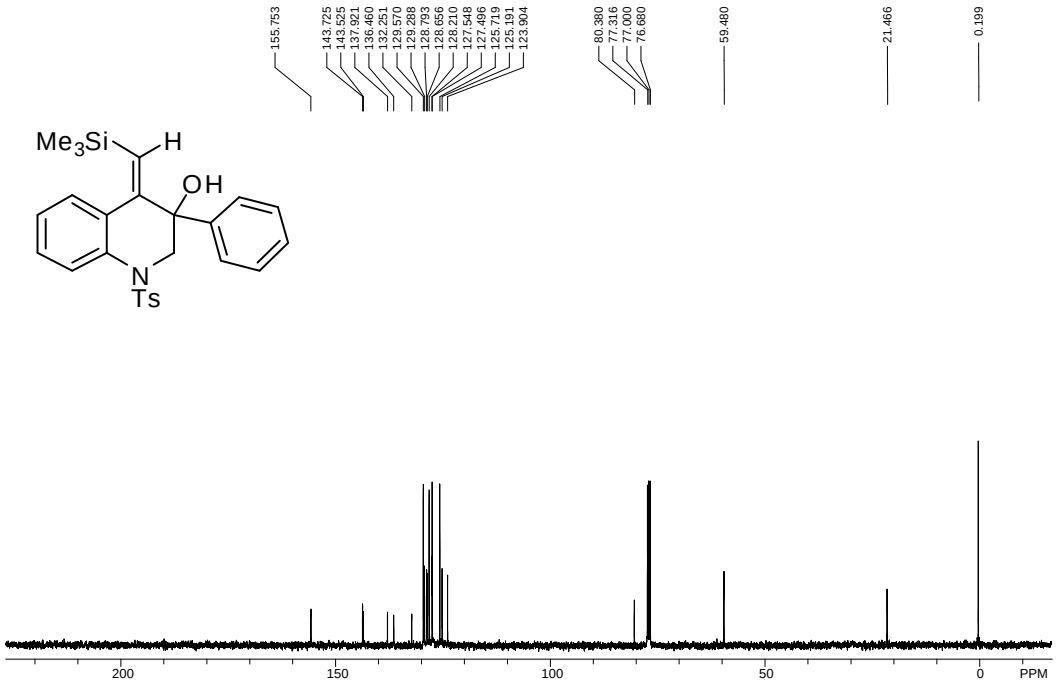
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129.288
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128.656
128.210
127.548
127.496
125.719
125.191
123.904
80.380
77.316
77.000
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21.466
0.199
Me3Si
H
OH
N
Ts
200
150
100
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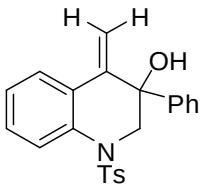
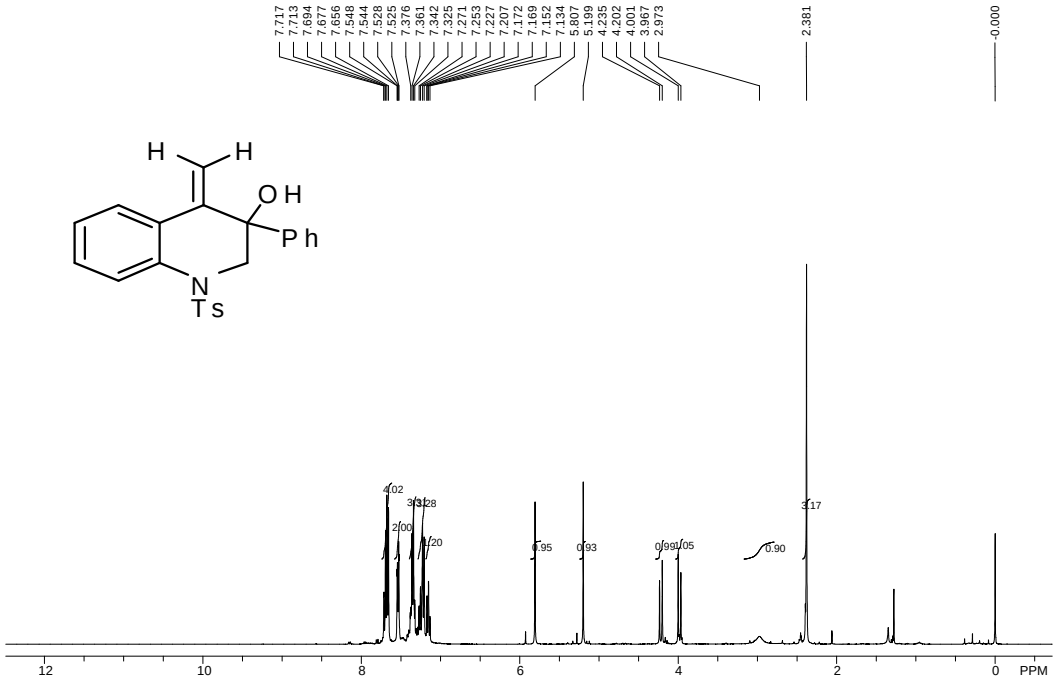
0
PPM


H
H
OH
Ph
N
Ts

3e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7.544
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7.376
7.361
7.342
7.325
7.271
7.253
7.227
7.207
7.172
7.169
7.152
7.134
5.807
5.199
4.235
4.202
4.001
3.967
2.973
2.381
-0.000
H
H
OH
Ph
N
Ts
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2.00
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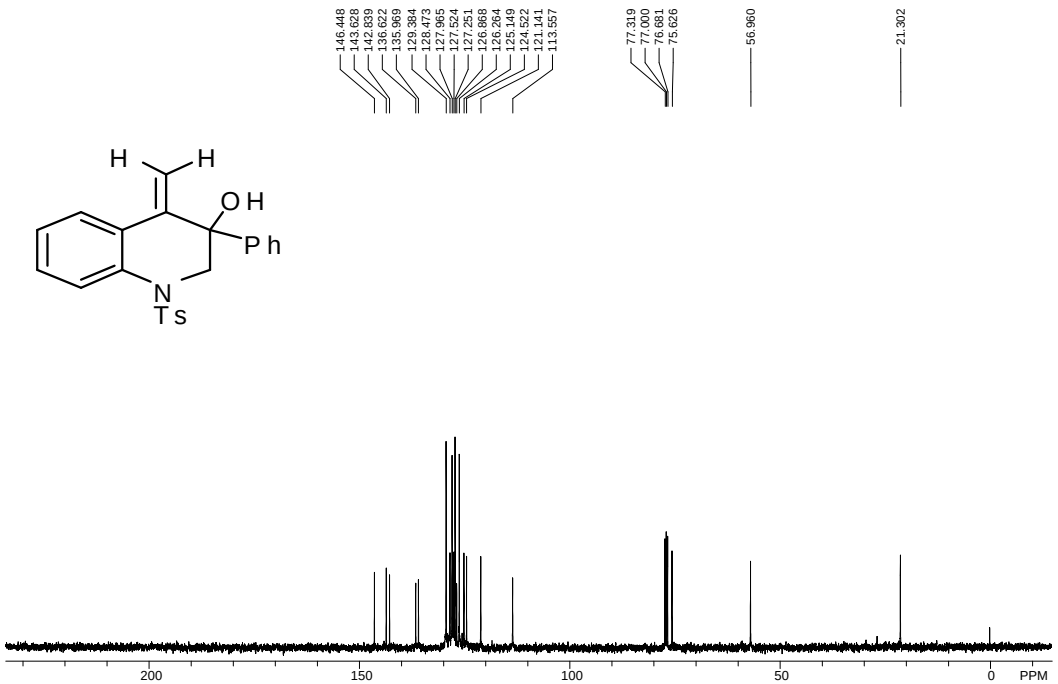
PPM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125.149
124.522
121.141
113.557
77.319
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OH
Ph
N
Ts
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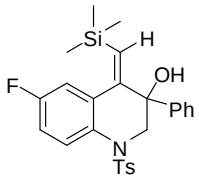
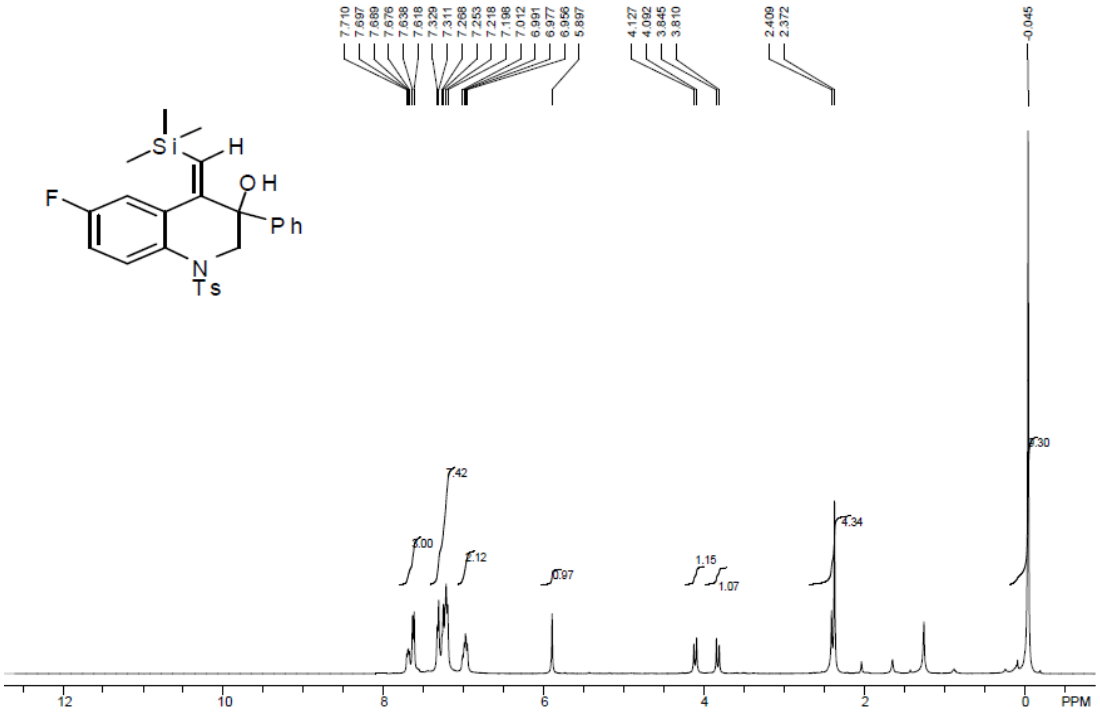
PPM


Si
H
OH
F
Ph
N
Ts

2f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7.253
7.218
7.198
7.012
6.991
6.977
6.956
5.897
4.127
4.092
3.845
3.810
2.409
2.372
0.045
Si
H
OH
F
Ph
N
Ts
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10
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4
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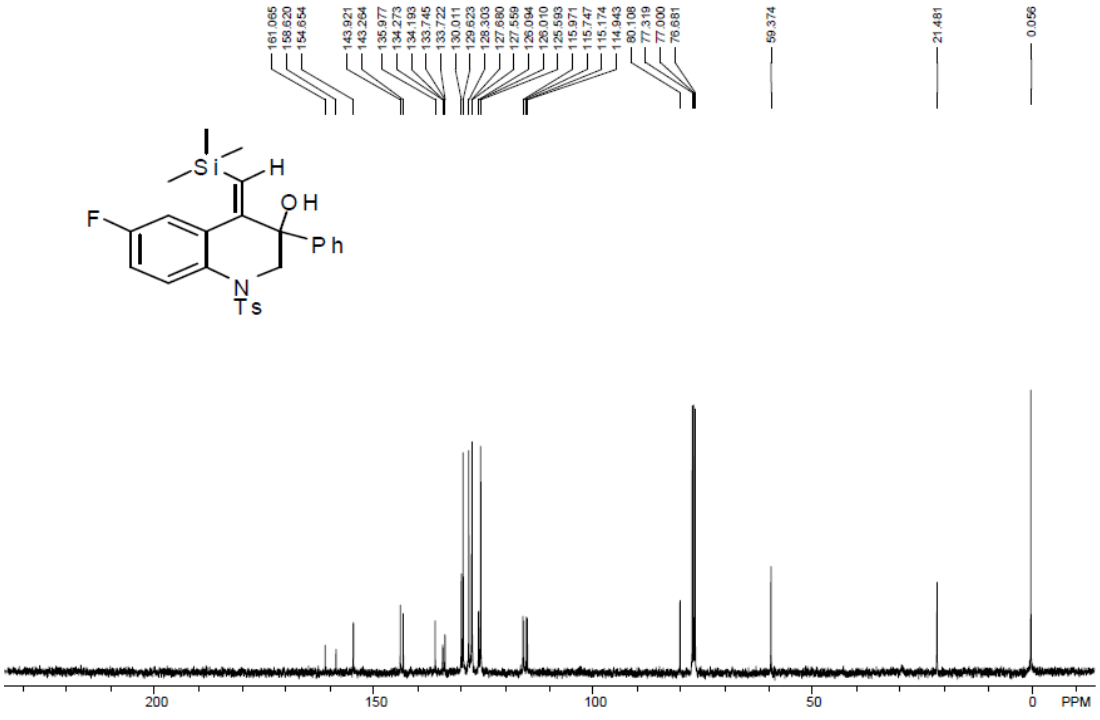
PPM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128.303
127.680
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126.010
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115.971
115.747
115.174
114.943
80.108
77.319
77.000
76.681
59.374
21.481
0.056
Si
H
OH
F
Ph
N
Ts
200
150
100
50
0
PPM


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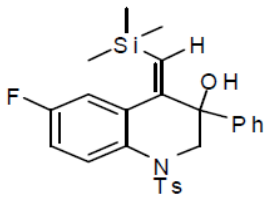
-116.460
-116.472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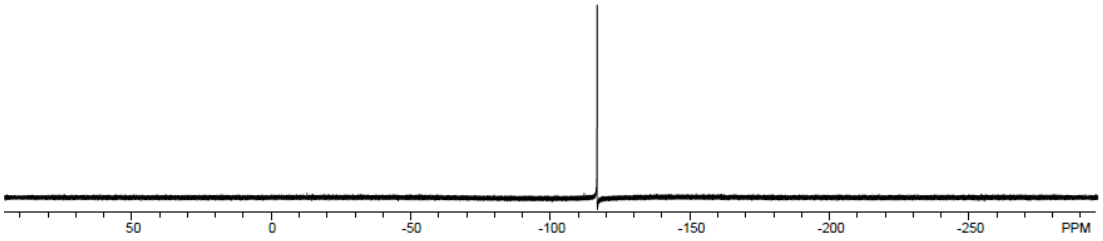
OH
F
Ph
N
Ts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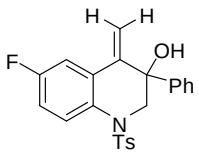
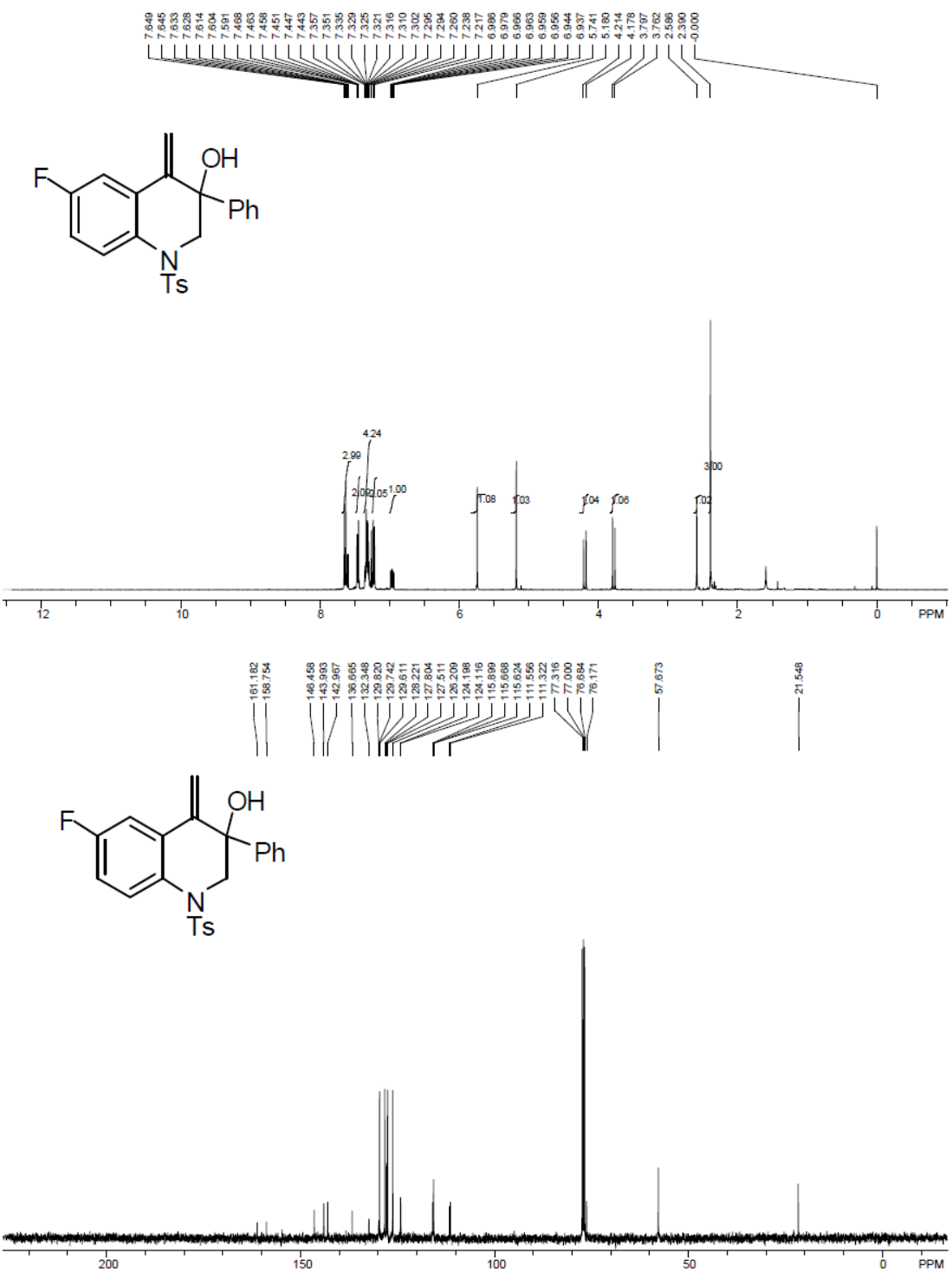

H
H
OH
F
Ph
N
Ts

3f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7.321
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7.310
7.302
7.295
7.294
7.260
7.238
7.217
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6.979
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6.963
6.959
6.956
6.944
6.937
5.741
5.180
4.214
4.178
3.797
3.762
2.586
2.390
-0.000
OH
F
Ph
N
Ts
4.24
2.99
2.09
2.05
1.00
1.08
1.03
1.04
1.06
3.00
1.02
12
10
8
6
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PPM
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146.458
143.993
142.967
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132.348
129.820
129.742
129.611
128.221
127.804
127.511
126.209
124.196
124.116
115.899
115.668
115.624
111.556
111.322
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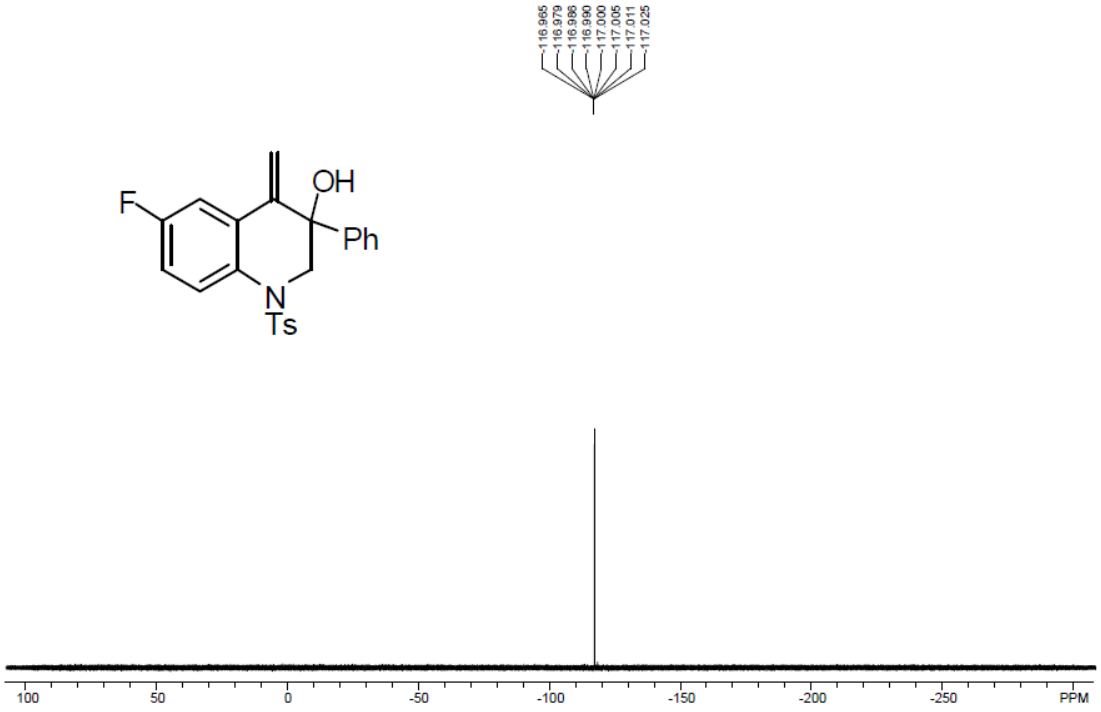
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Ph
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Ts
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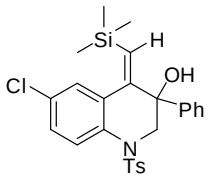
-150
-200
-250
PPM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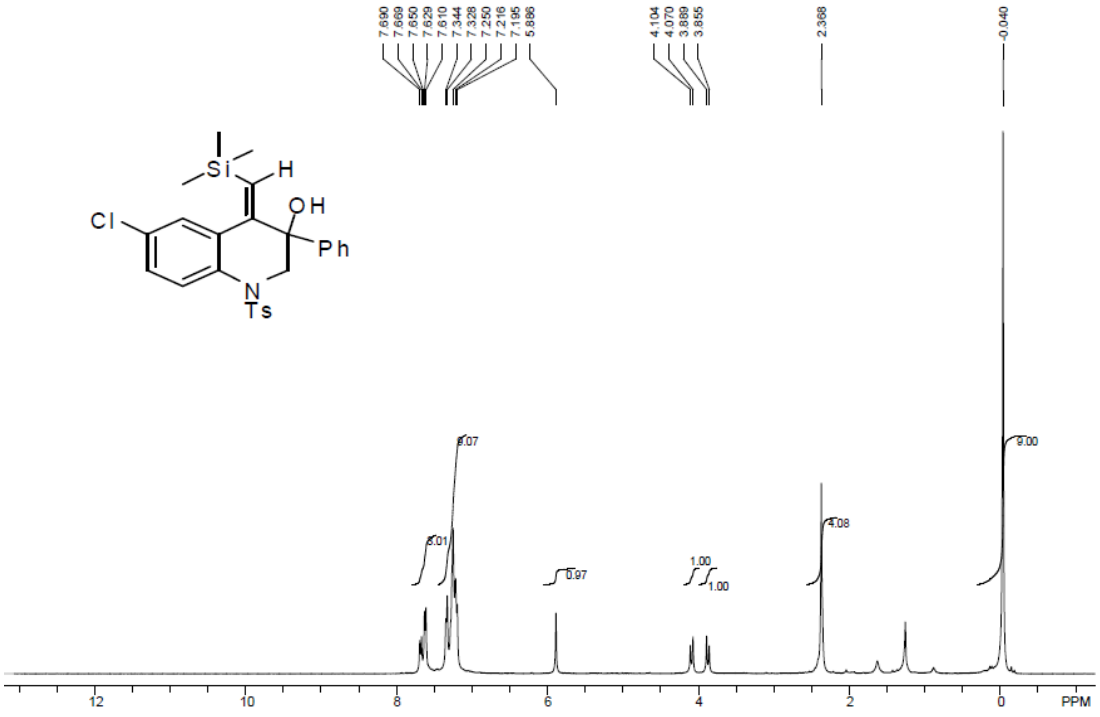
OH
Cl
Ph
N
Ts

2g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7.195
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4.070
3.889
3.855
2.368
-0.040
Si
H
OH
Cl
Ph
N
Ts
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0.97
1.00
1.00
4.08
9.00
12
10
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4
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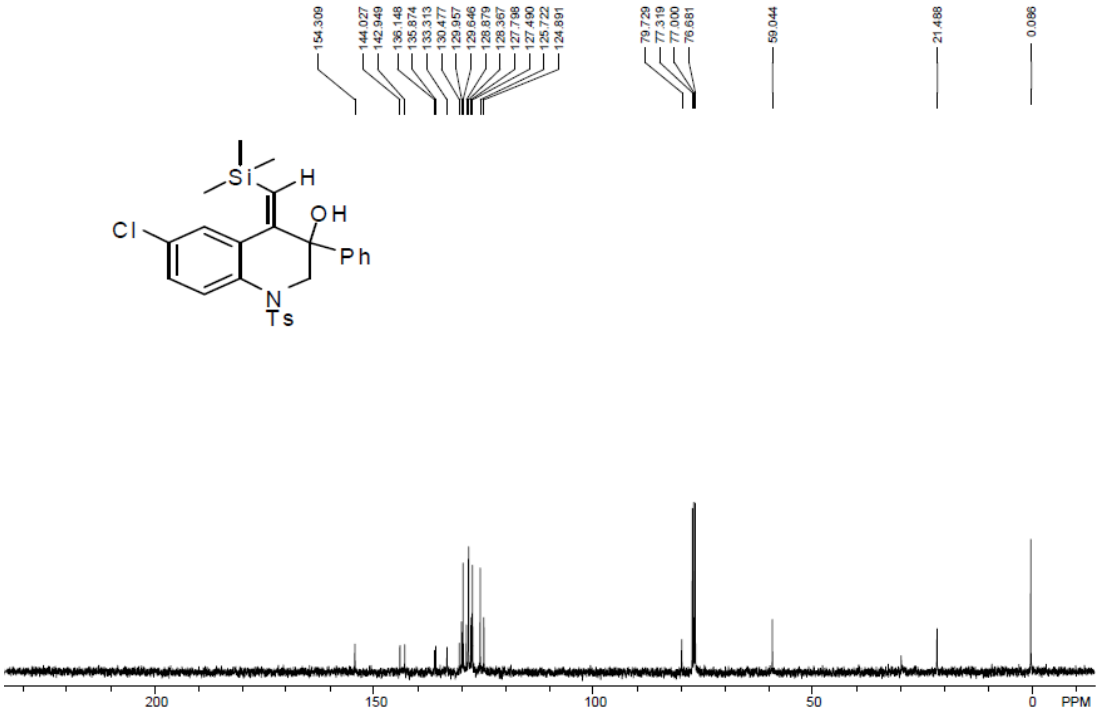
PPM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125.722
124.891
79.729
77.319
77.000
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H
OH
Cl
Ph
N
Ts
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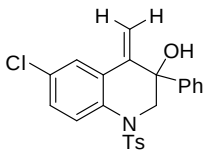
PPM


H
H
OH
Cl
Ph
N
Ts

3g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7.444
7.361
7.355
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7.320
7.314
7.302
7.256
7.222
7.202
7.194
7.188
7.172
7.165
5.763
5.192
4.185
4.151
3.887
3.853
2.527
2.380
0.000
OH
Cl
Ph
N
Ts
4.00
3.30
2.99
2.14
2.02
2.00
2.04
2.07
5.24
2.04
12
10
8
6
4
2
0
PPM
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144.075
142.617
136.497
134.765
130.336
129.637
129.771
128.589
128.284
127.916
127.507
126.317
125.075
123.030
114.969
77.316
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Cl
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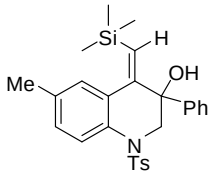
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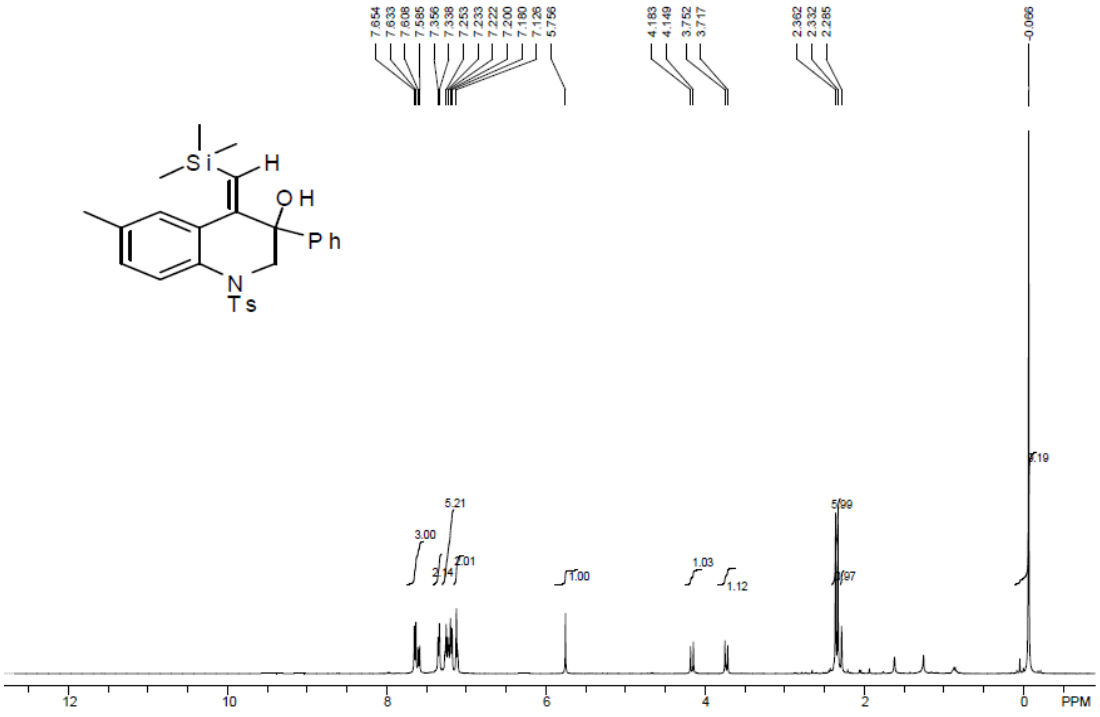
OH
Me
Ph
N
Ts

2h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7.233
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7.126
5.756
4.183
4.149
3.752
3.717
2.362
2.332
2.285
-0.066
Si
H
OH
Ph
N
Ts
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3.00
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1.00
1.03
1.12
5.99
2.19
12
10
8
6
4
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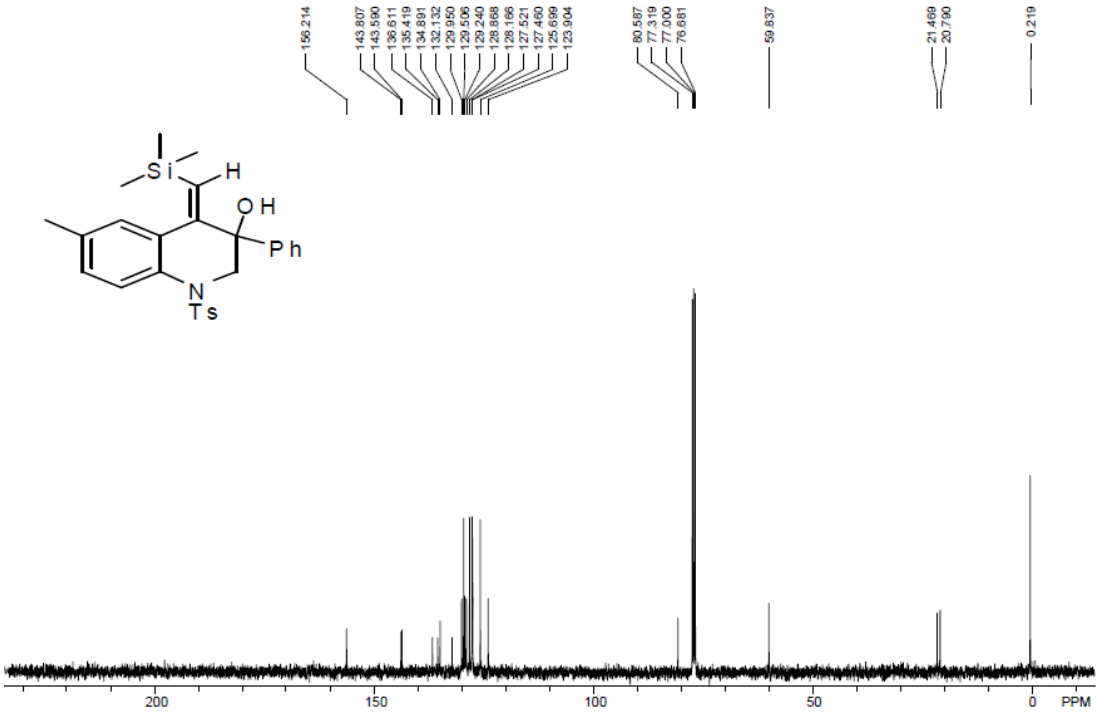
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129.950
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129.240
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127.521
127.460
125.699
123.904
80.587
77.319
77.000
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20.790
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Si
H
OH
Ph
N
Ts
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50
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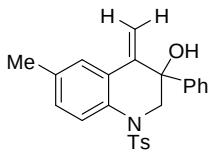
PPM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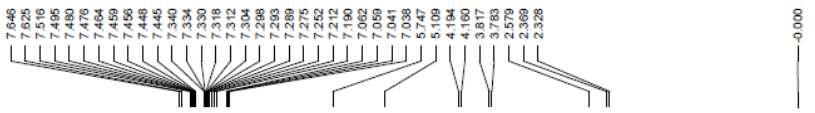
H
OH
Me
Ph
N
Ts

3h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7.334
7.330
7.318
7.312
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7.298
7.293
7.289
7.275
7.252
7.212
7.190
7.062
7.059
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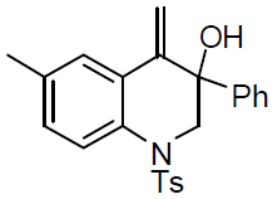
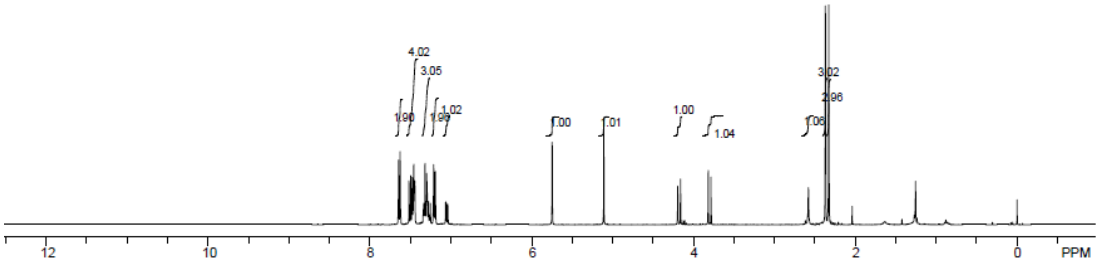

OH
Ph
N
Ts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1.00
1.04
3.02
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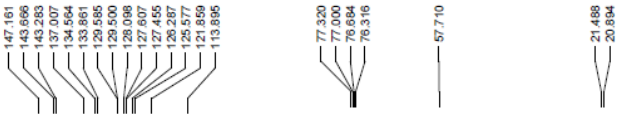
1.08
12
10
8
6
4
2
0
PPM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127.607
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126.287
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77.320
77.000
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76.316
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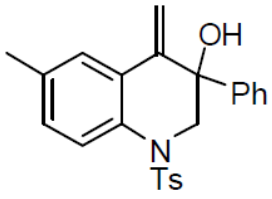
20.894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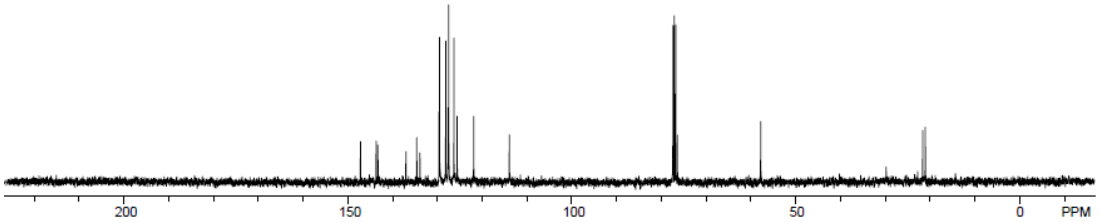
Ph
N
Ts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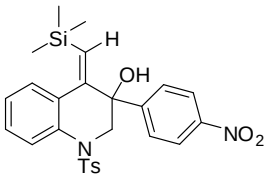
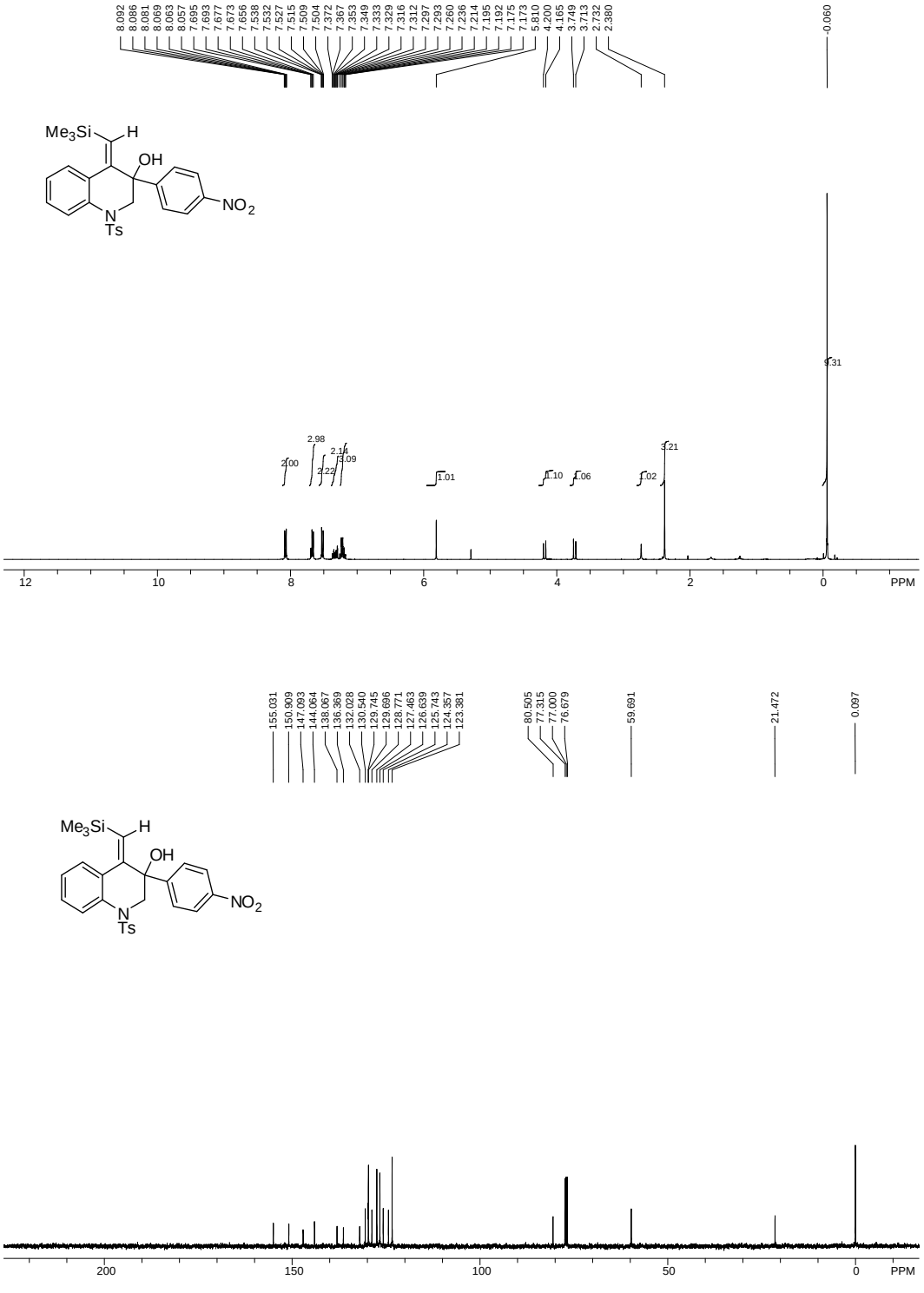
0
PPM


Si
H
OH
NO2
N
Ts

2i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7.527
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7.509
7.504
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7.353
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7.312
7.297
7.293
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7.236
7.214
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7.192
7.175
7.173
5.810
4.200
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3.713
2.732
2.380
-0.060
Me3Si
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OH
NO2
N
Ts
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2.14
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2.22
1.01
1.10
1.06
3.21
1.02
12
10
8
6
4
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126.639
125.743
124.357
123.381
80.505
77.315
77.000
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OH
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N
Ts
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100
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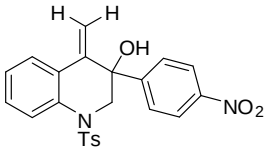
PPM


H
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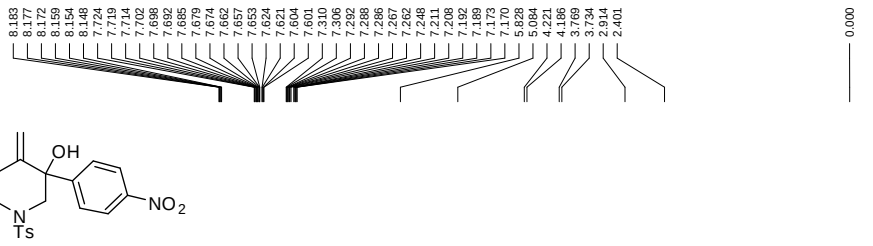
OH
NO2
N
Ts

3i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7.657
7.653
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7.621
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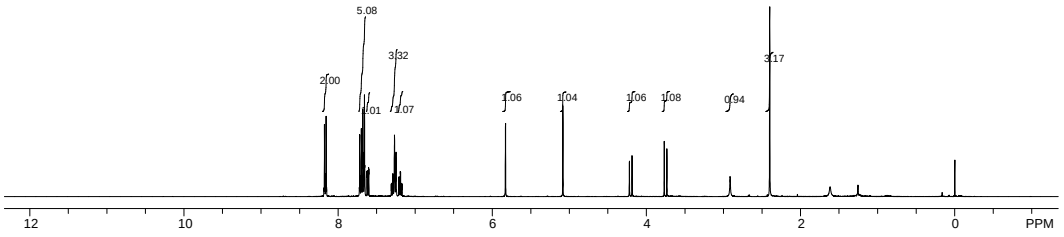
NO2
N
Ts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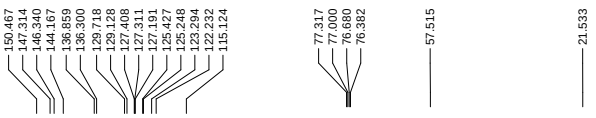
PPM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127.408
127.311
127.191
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125.248
123.294
122.232
115.124
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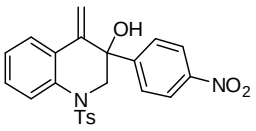
76.382
57.515
21.533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NO2
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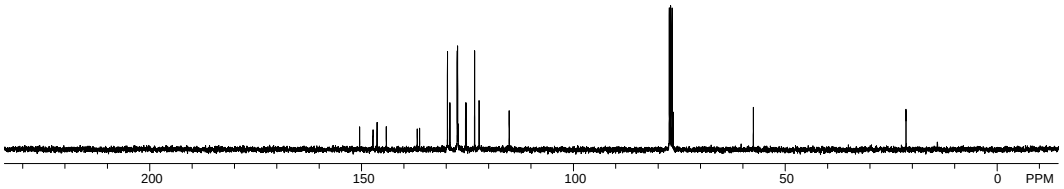
Ts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100
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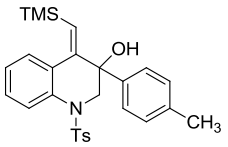
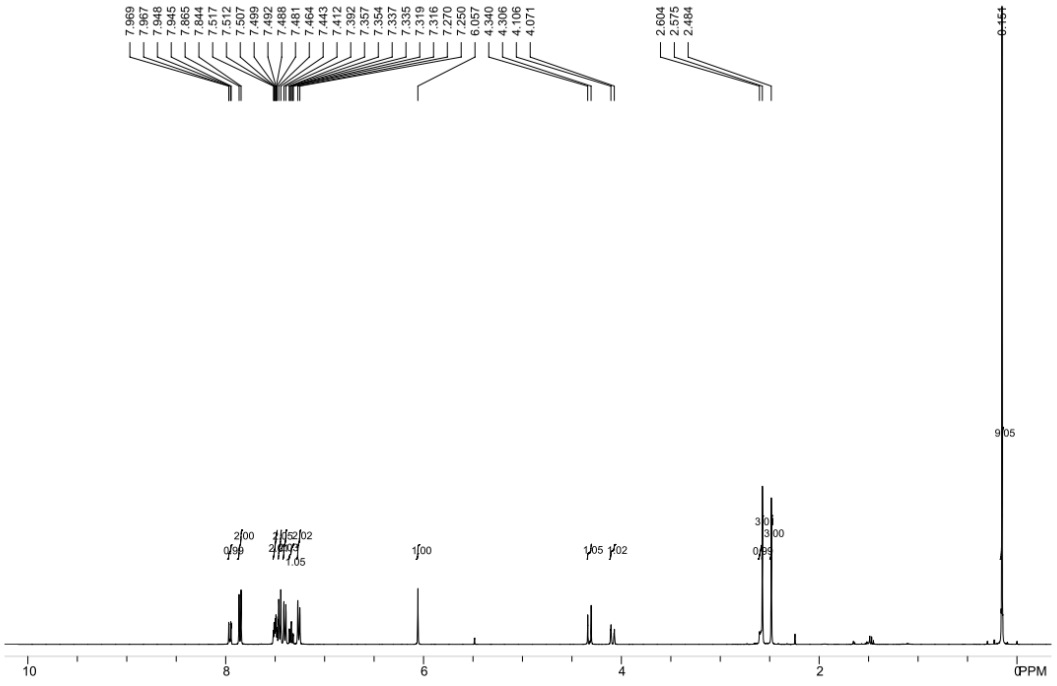
PPM


TMS
OH
N
Ts
CH3

2j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7.412
7.392
7.357
7.354
7.337
7.335
7.319
7.316
7.270
7.250
6.057
4.340
4.306
4.106
4.071
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1.05
1.00
1.05
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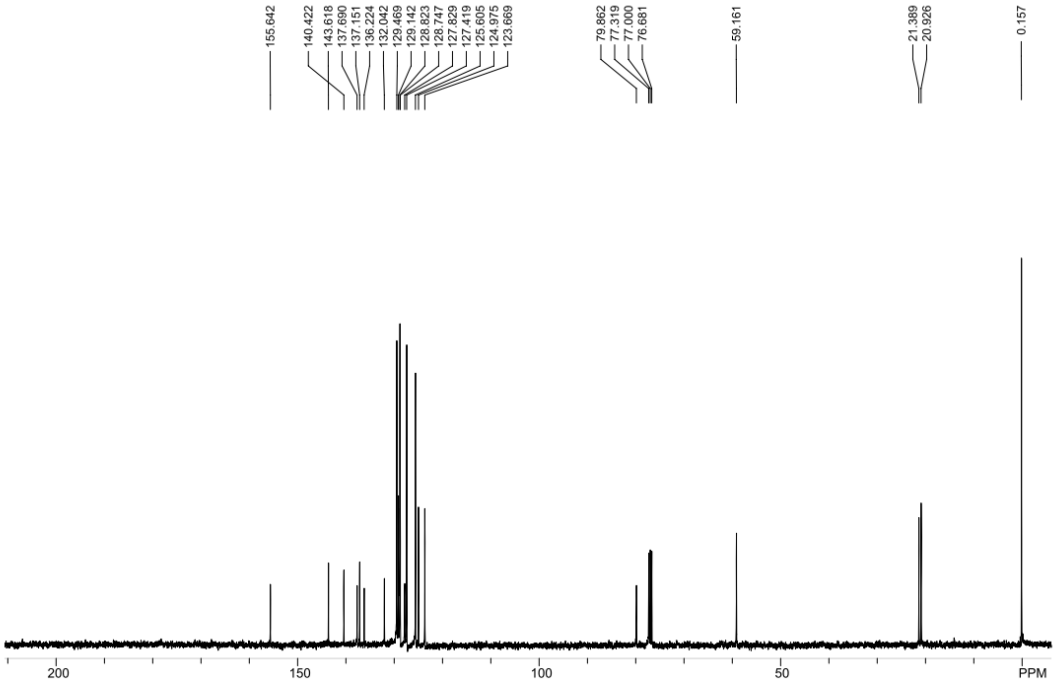
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124.975
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77.000
76.681
59.161
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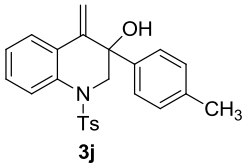
20.926
0.157
200
150
100
50
PPM


OH
N
Ts
CH3

3j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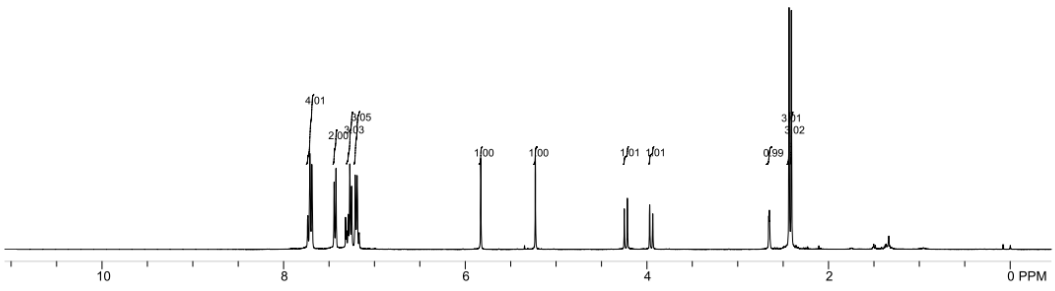
2.410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3.02
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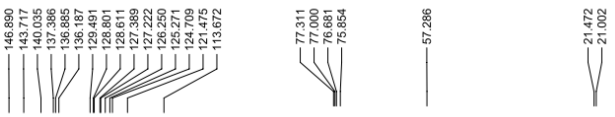
0 PPM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127.222
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75.854
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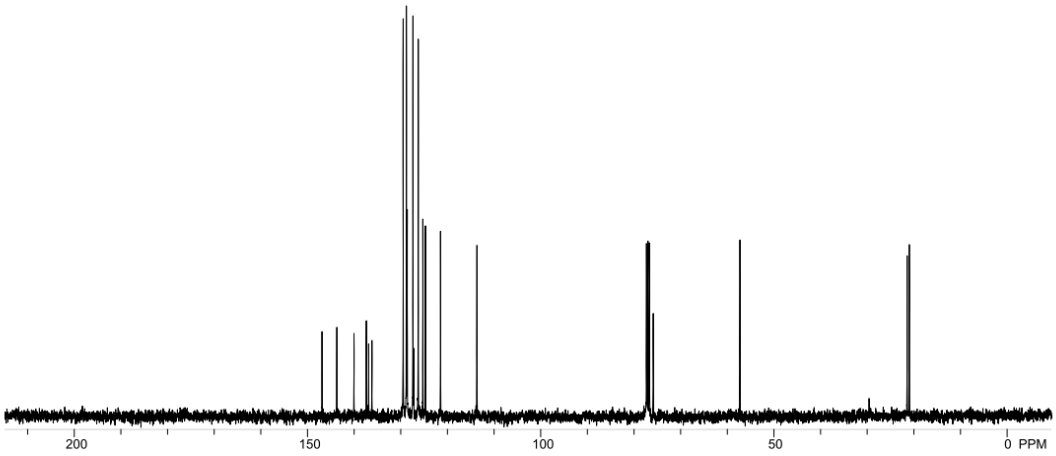
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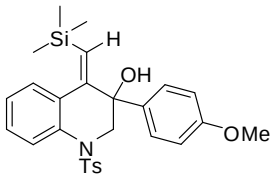
0 PPM


Si
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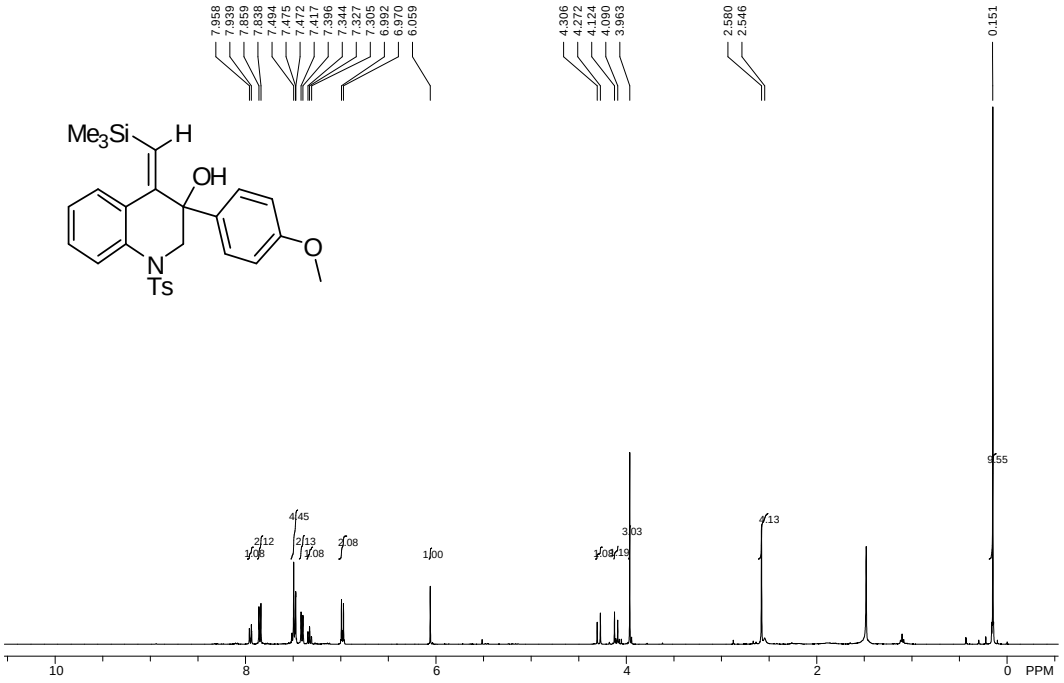
OH
OMe
N
Ts

2k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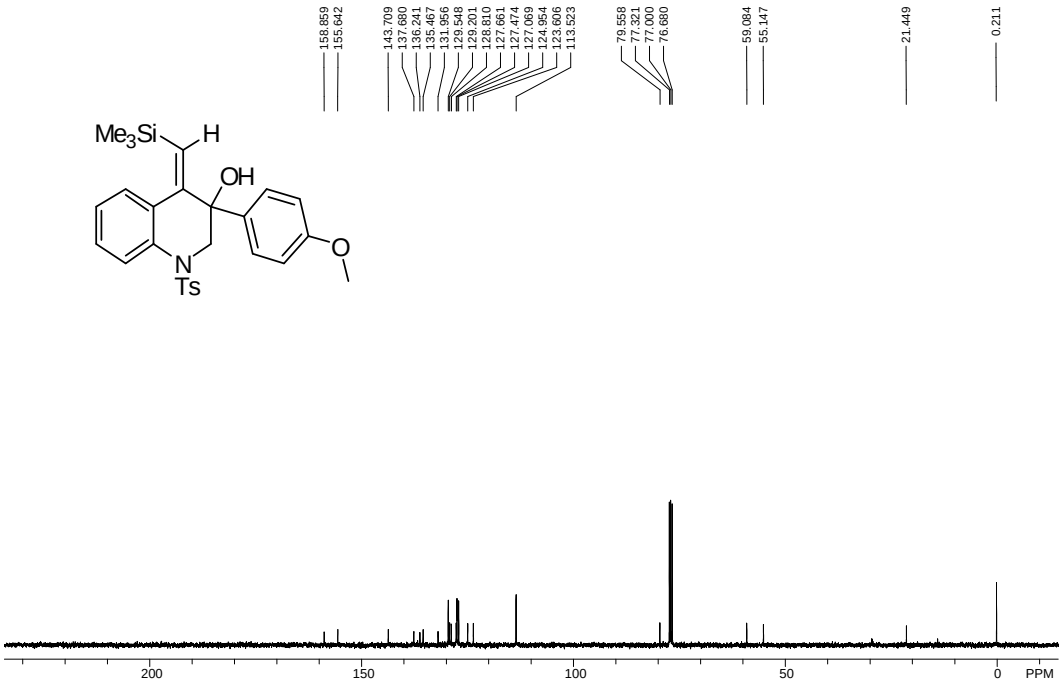
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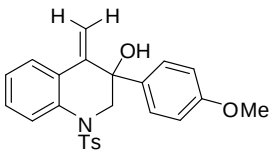
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Ts

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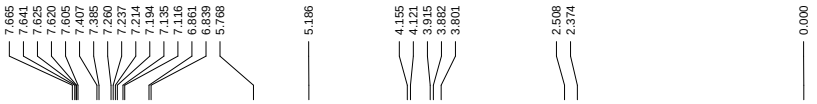
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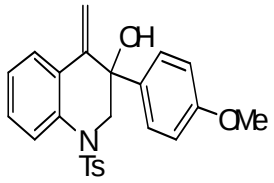
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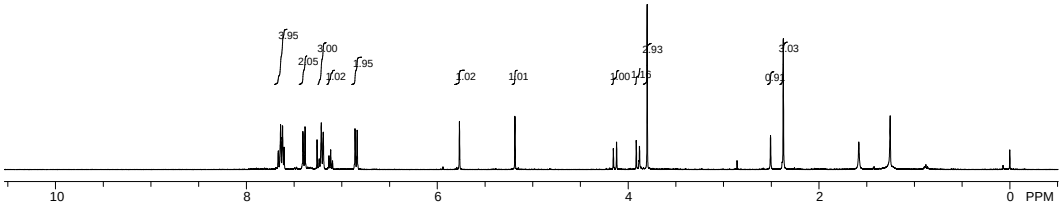
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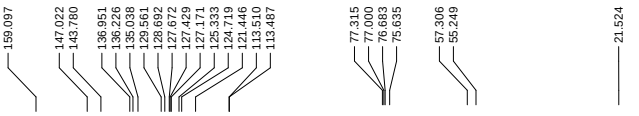
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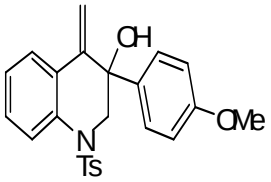
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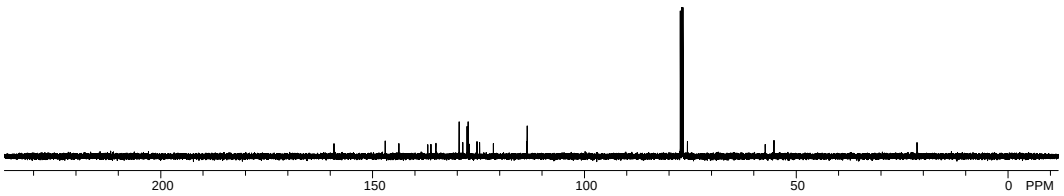
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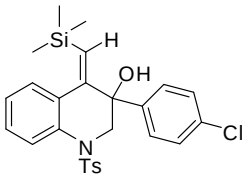
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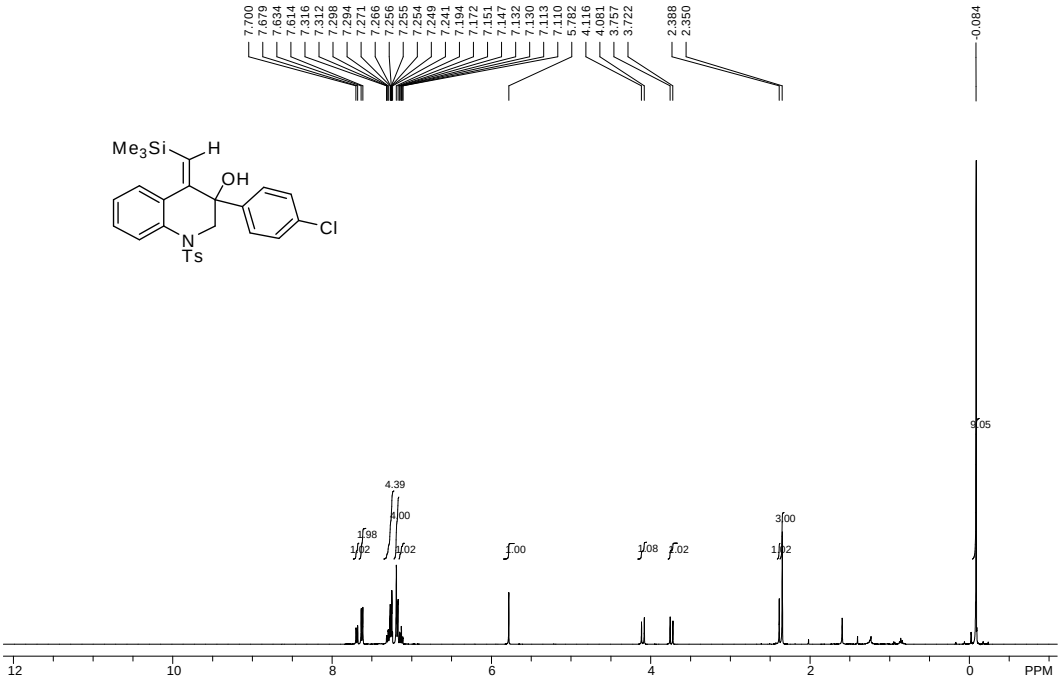
2l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OH
Cl
N
Ts
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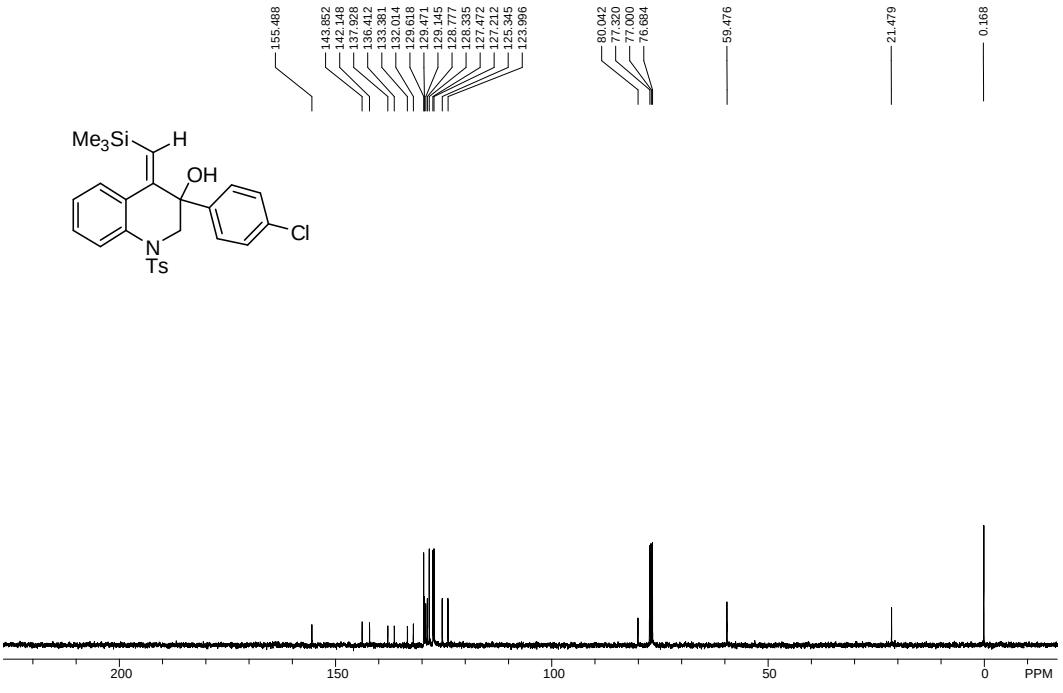
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129.145
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127.472
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125.345
123.996
80.042
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77.000
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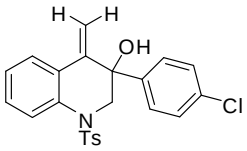
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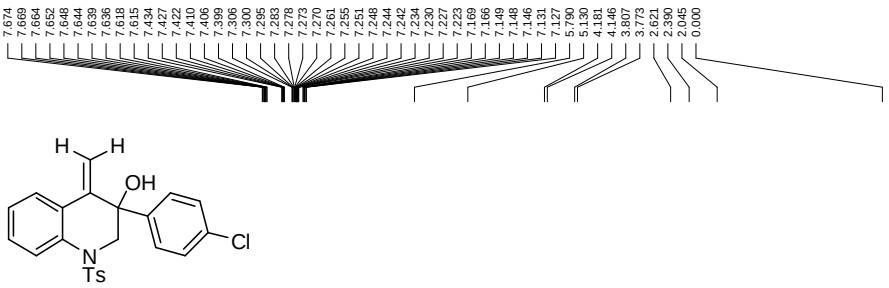
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7.300
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7.273
7.270
7.261
7.255
7.251
7.248
7.244
7.242
7.234
7.230
7.227
7.223
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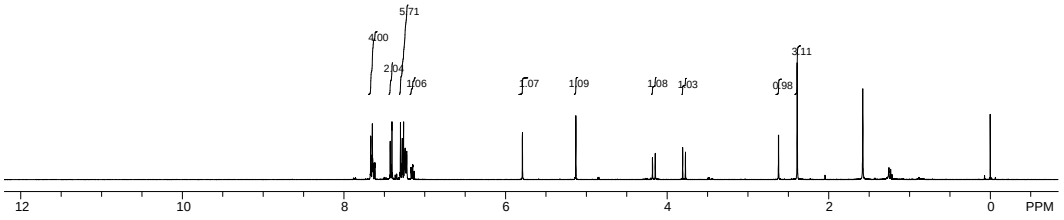
H
OH
Cl
N
Ts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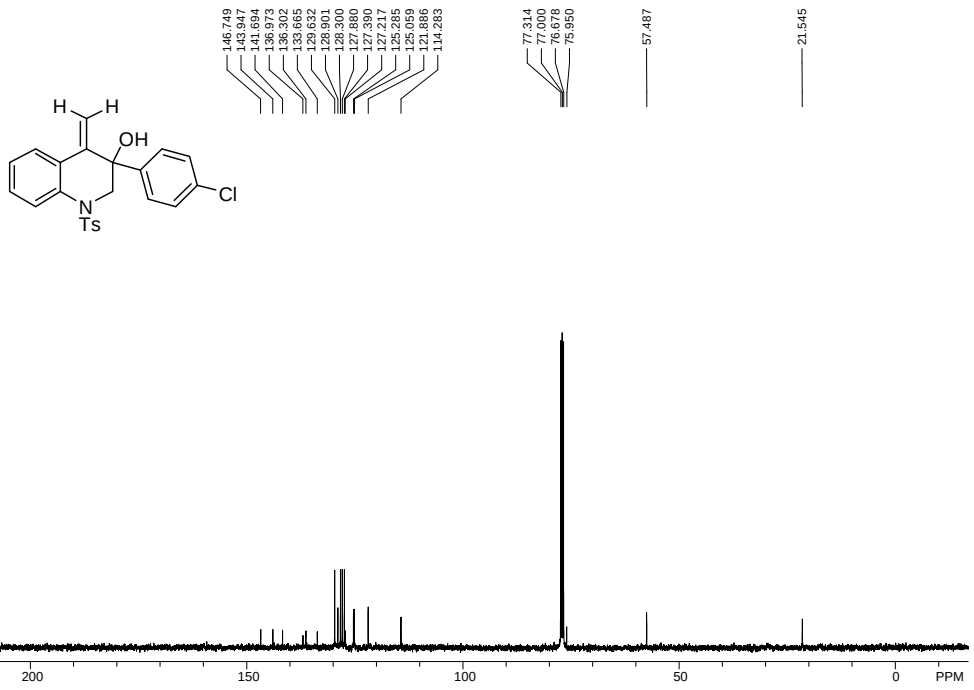
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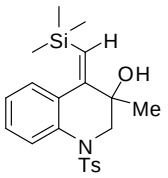
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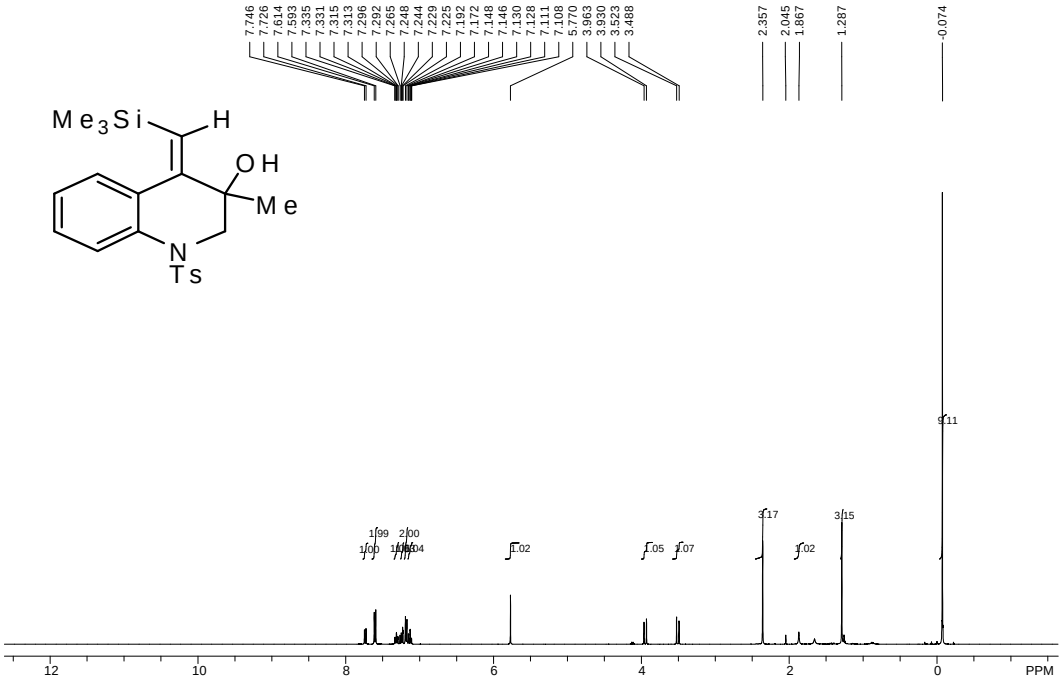
Me
N
Ts

2n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7.244
7.229
7.225
7.192
7.172
7.148
7.146
7.130
7.128
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3.930
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Me3Si
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OH
Me
N
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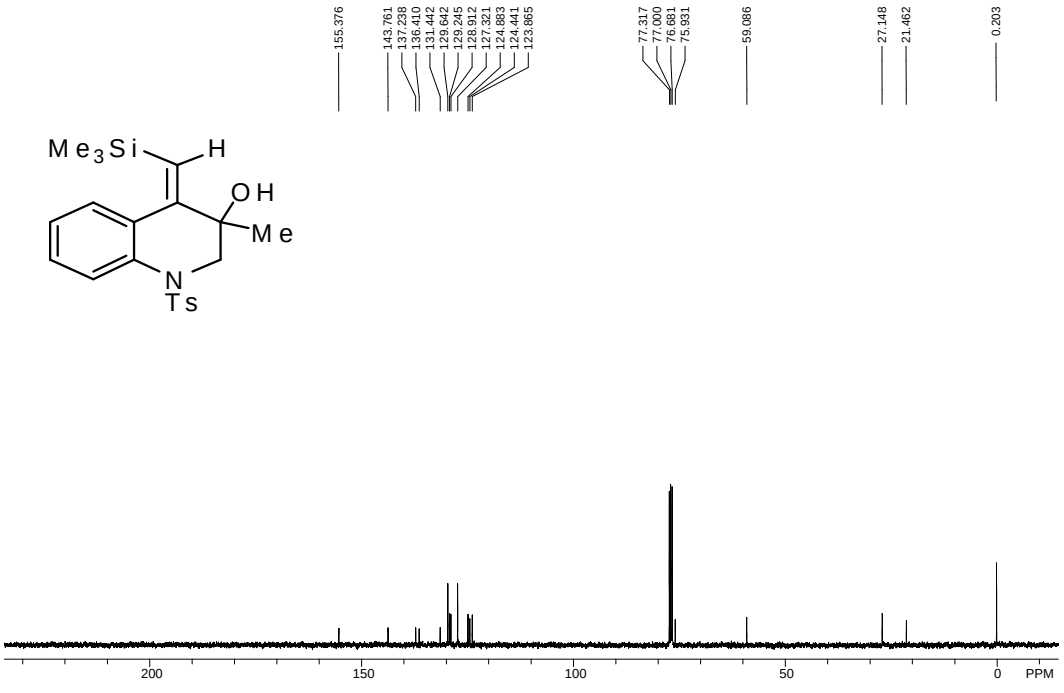
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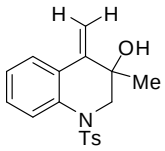
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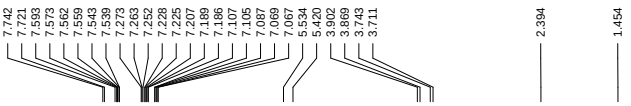
Me
N
Ts

3n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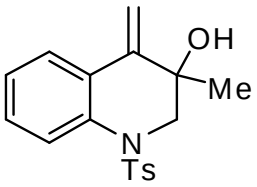
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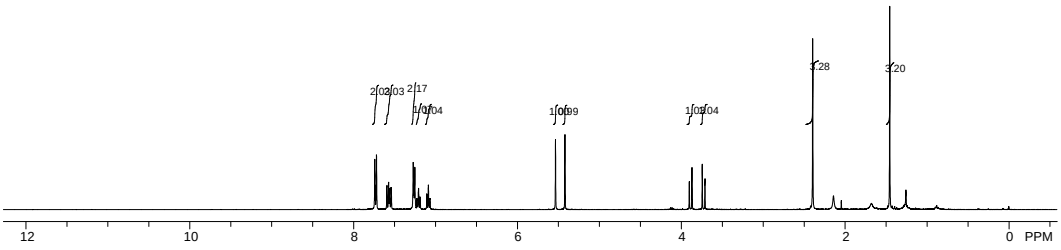
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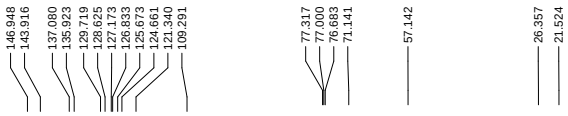
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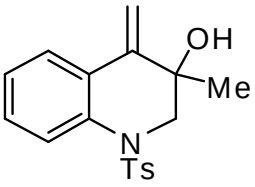
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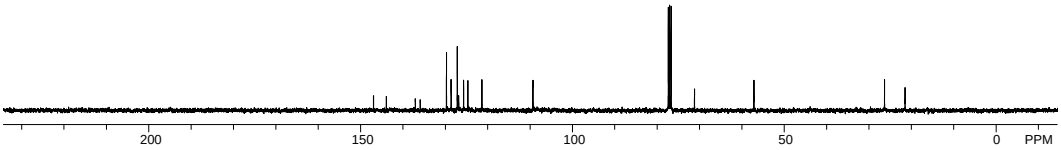
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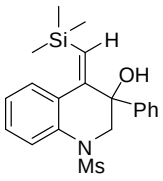
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OH
Ph
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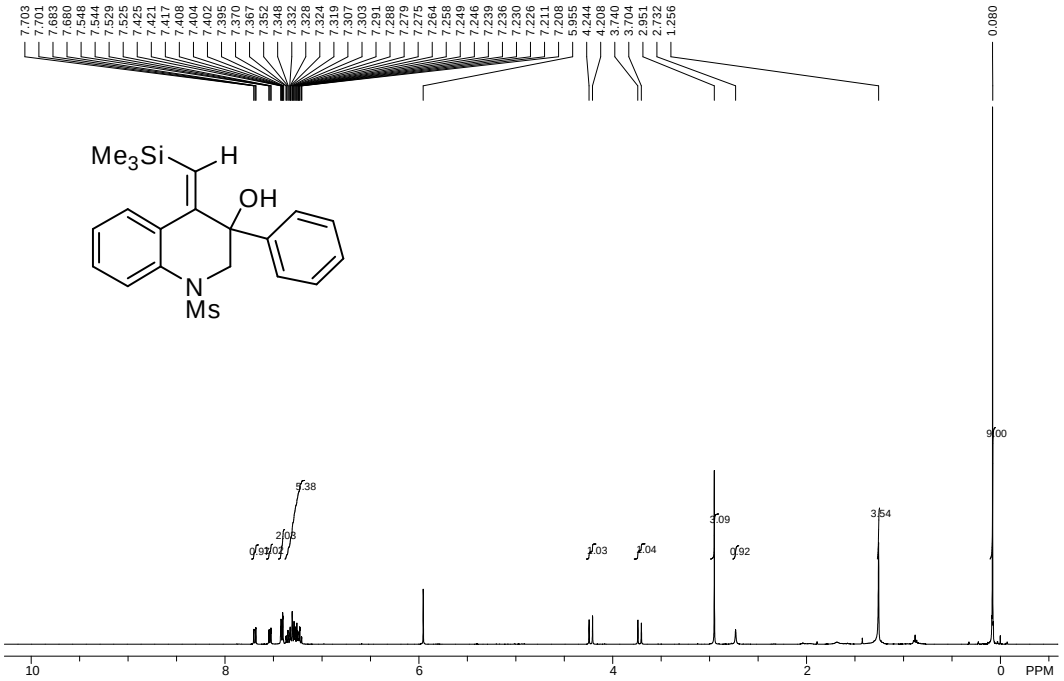
Ms

2p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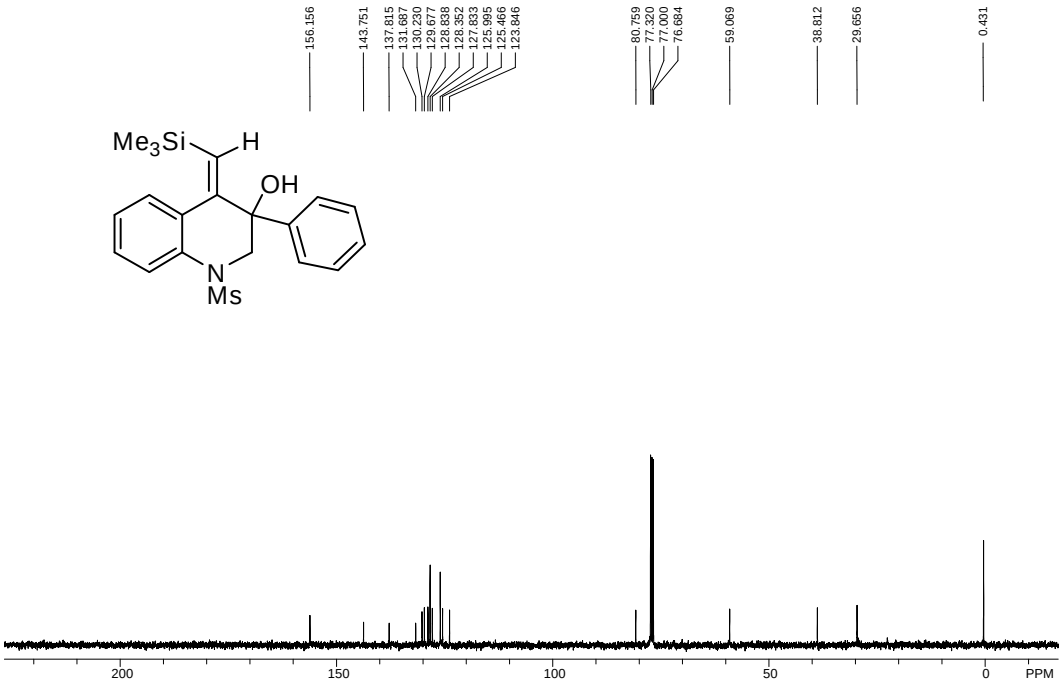
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77.000
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29.656
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Me3Si
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OH
N
Ms
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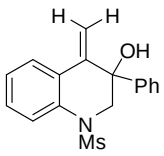
150
100
50
0
PPM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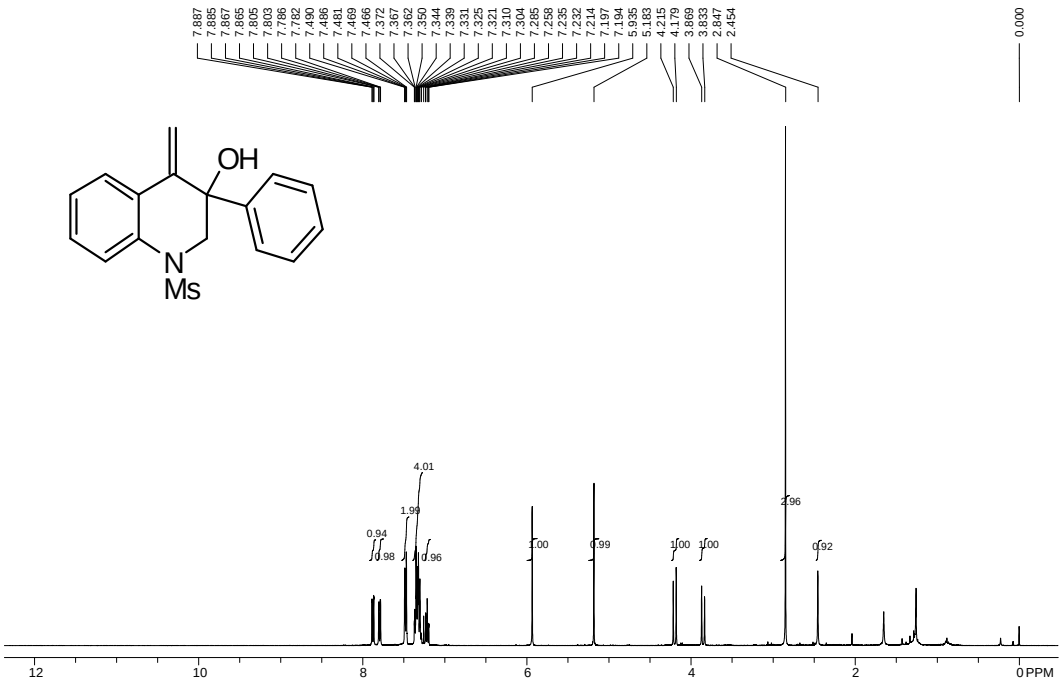
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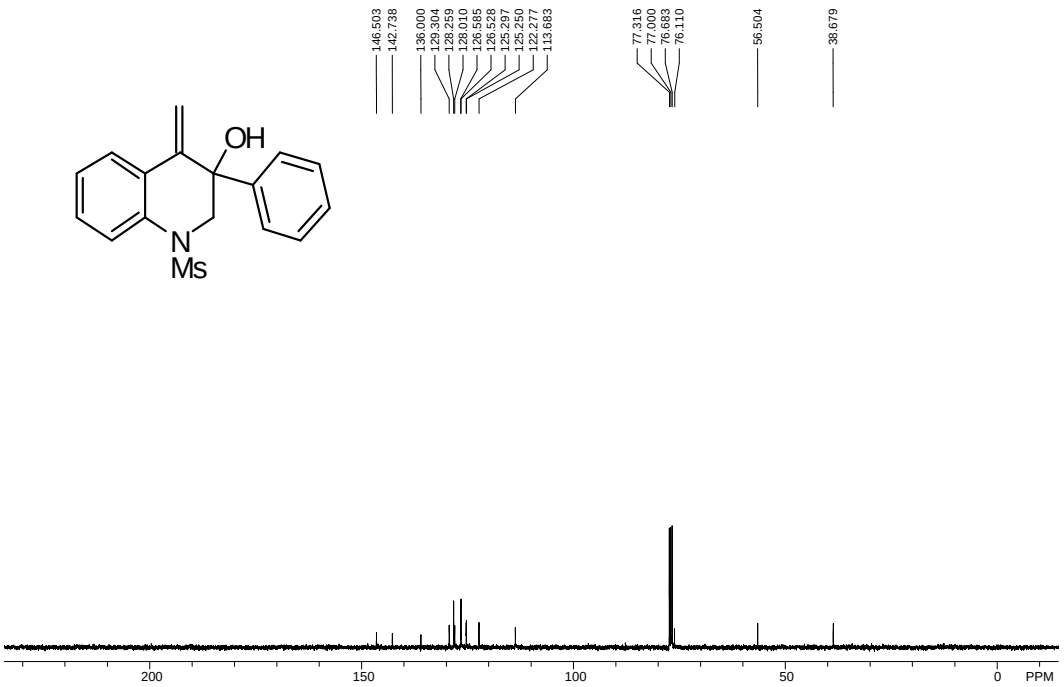
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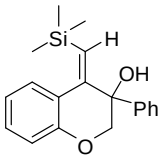
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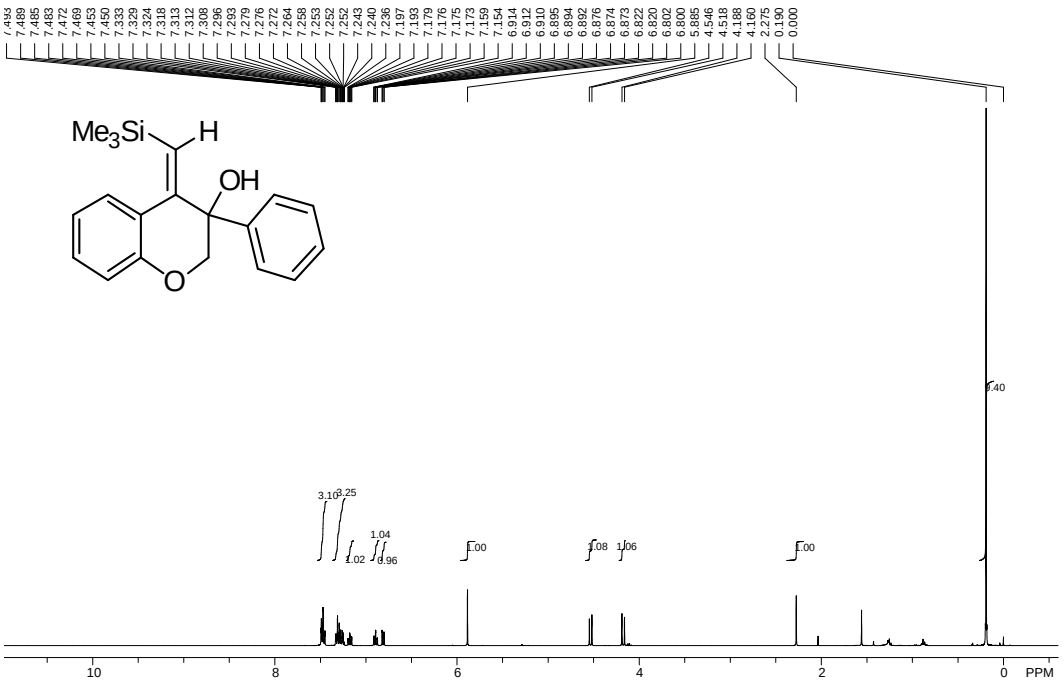
Ph
O

2q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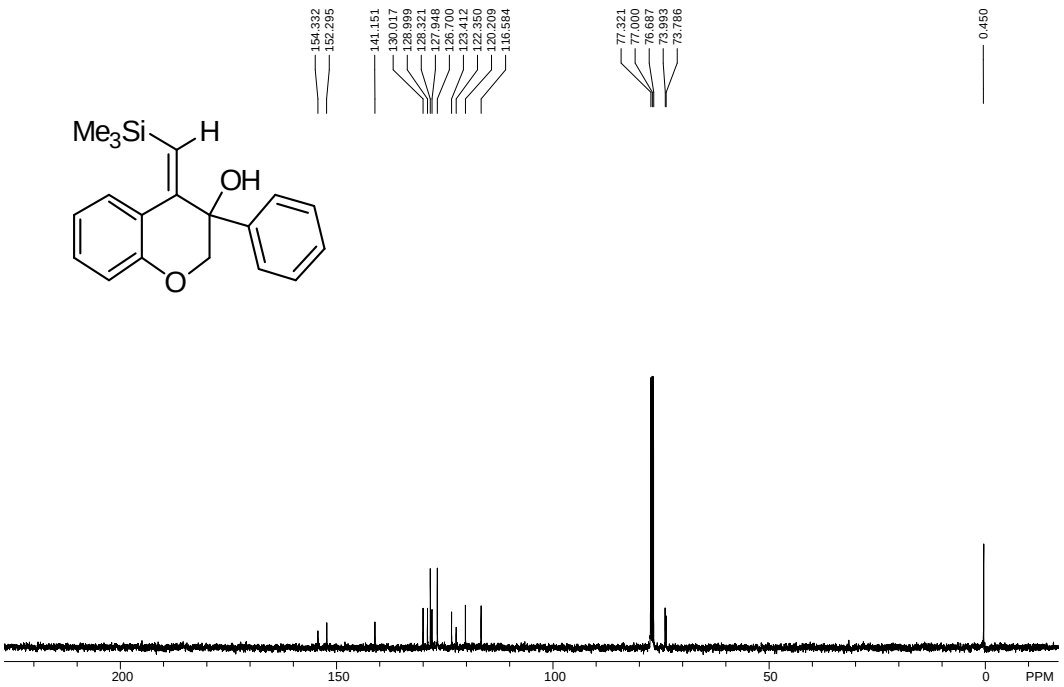
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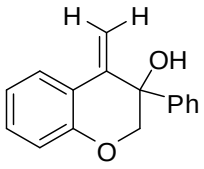
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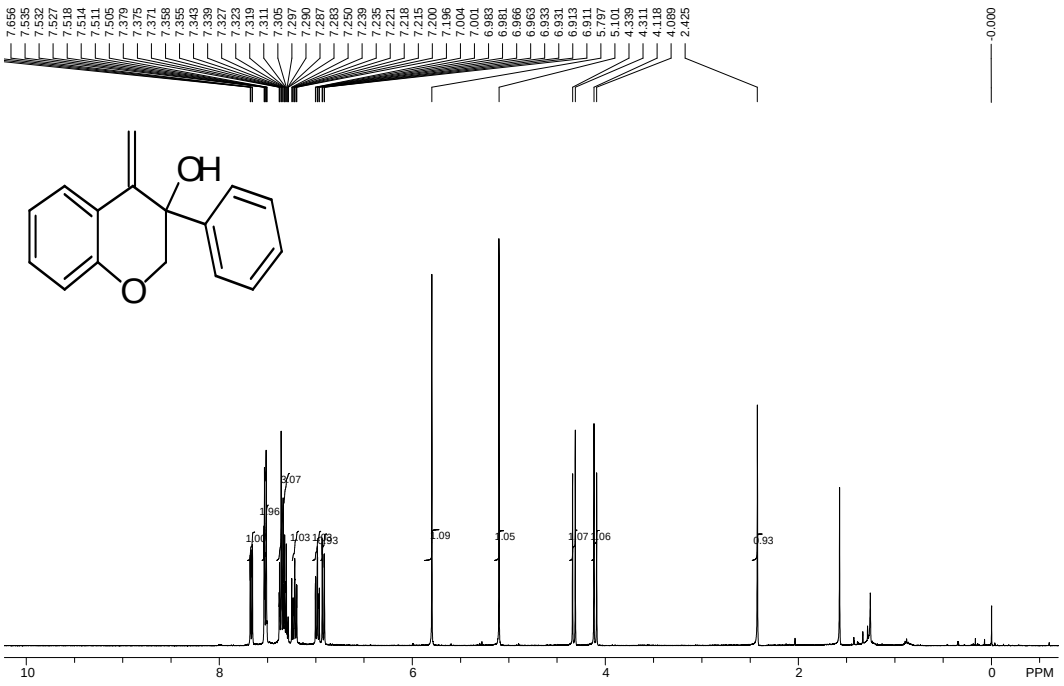
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7.235
7.221
7.218
7.215
7.200
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7.001
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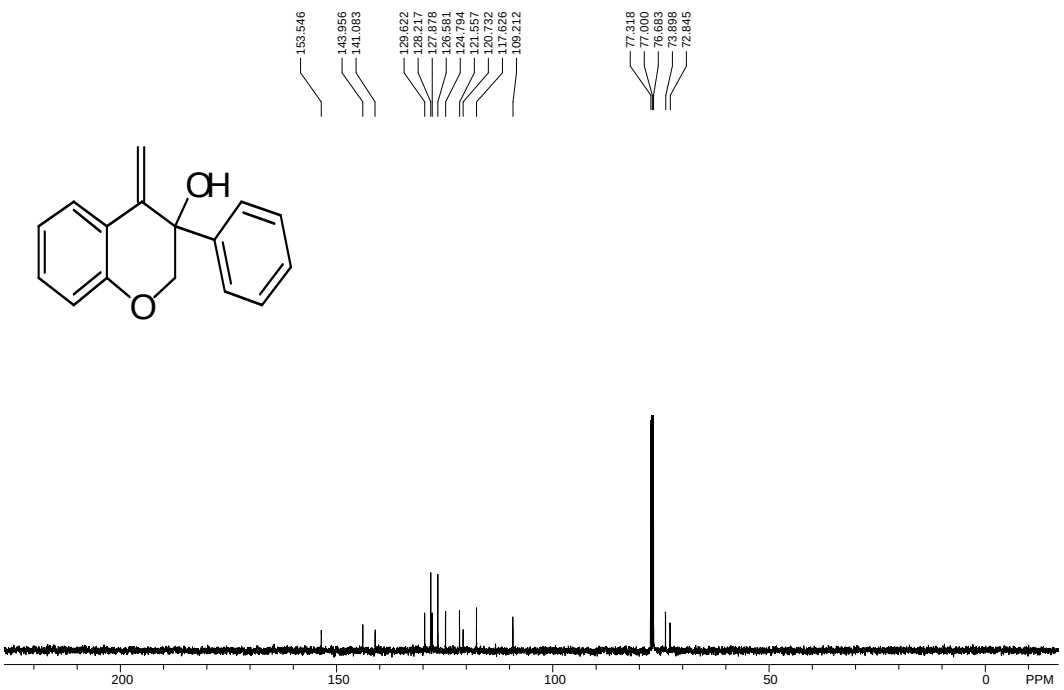
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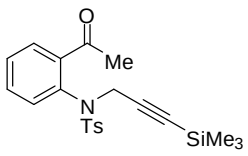
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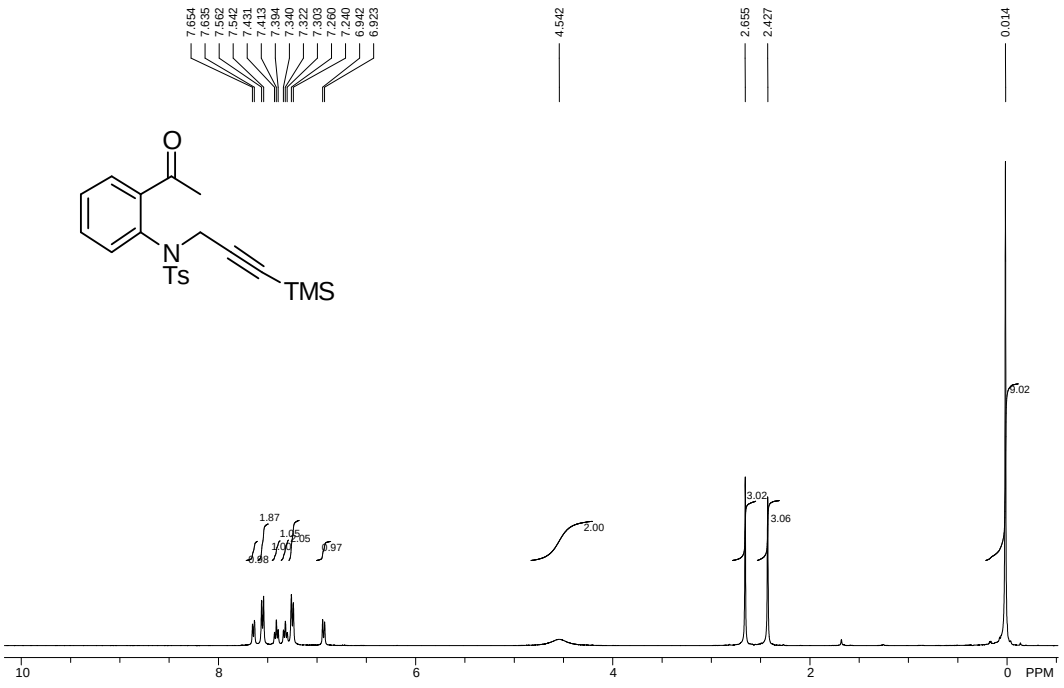
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6.923
4.542
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O
N
Ts
TMS
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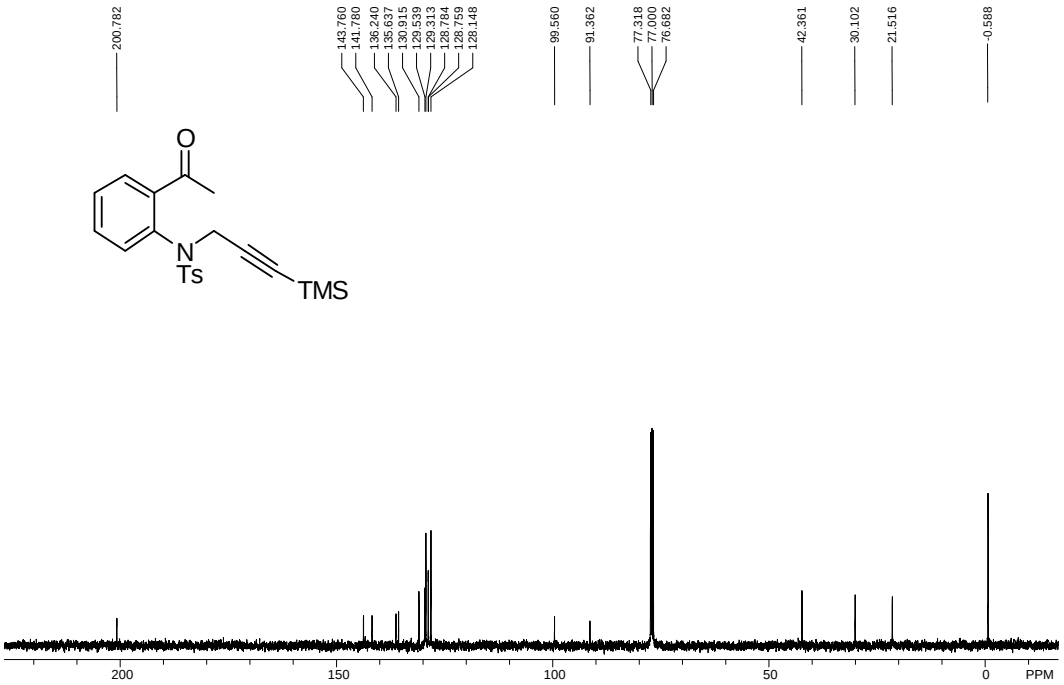
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TMS
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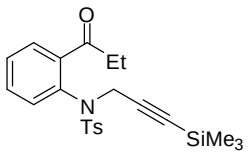
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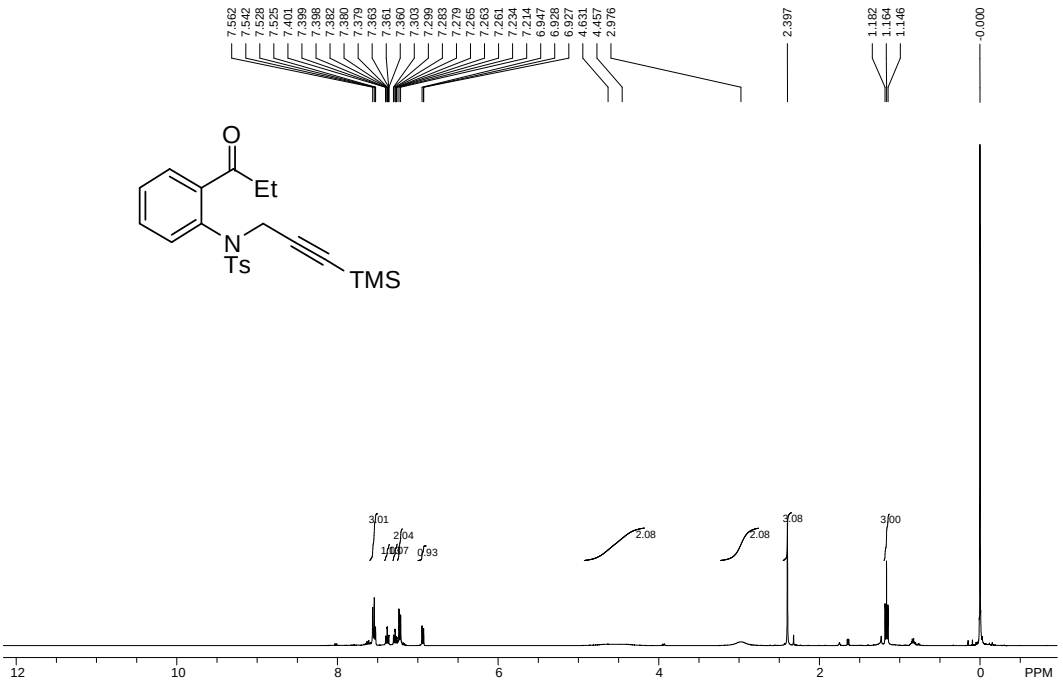
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7.283
7.279
7.265
7.263
7.261
7.234
7.214
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6.928
6.927
4.631
4.457
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2.397
1.182
1.164
1.146
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O
Et
N
Ts
TMS
3.01
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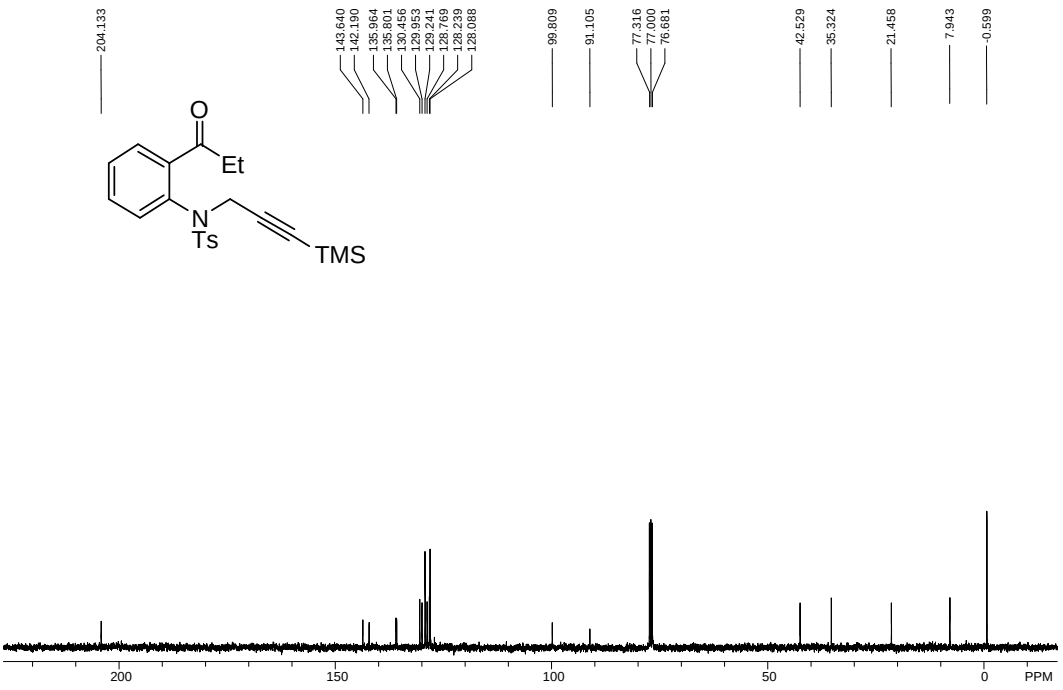
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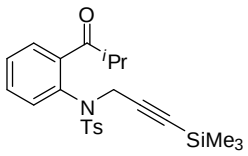
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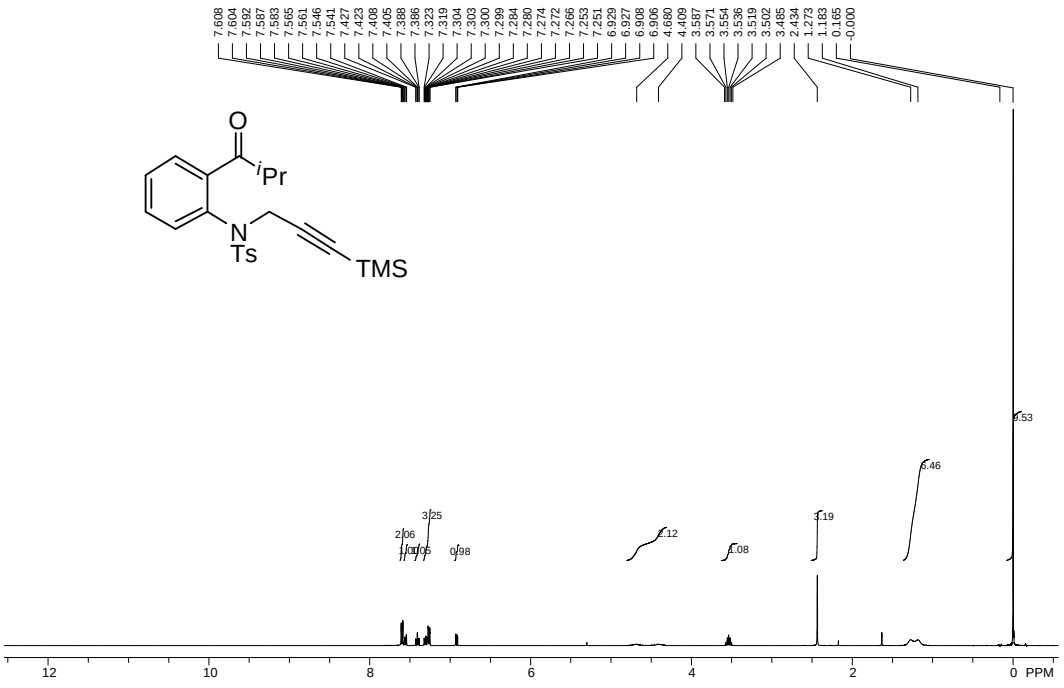
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7.405
7.388
7.386
7.323
7.319
7.304
7.303
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7.280
7.274
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6.929
6.927
6.908
6.906
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4.409
3.587
3.571
3.554
3.536
3.519
3.502
3.485
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iPr
N
Ts
TMS
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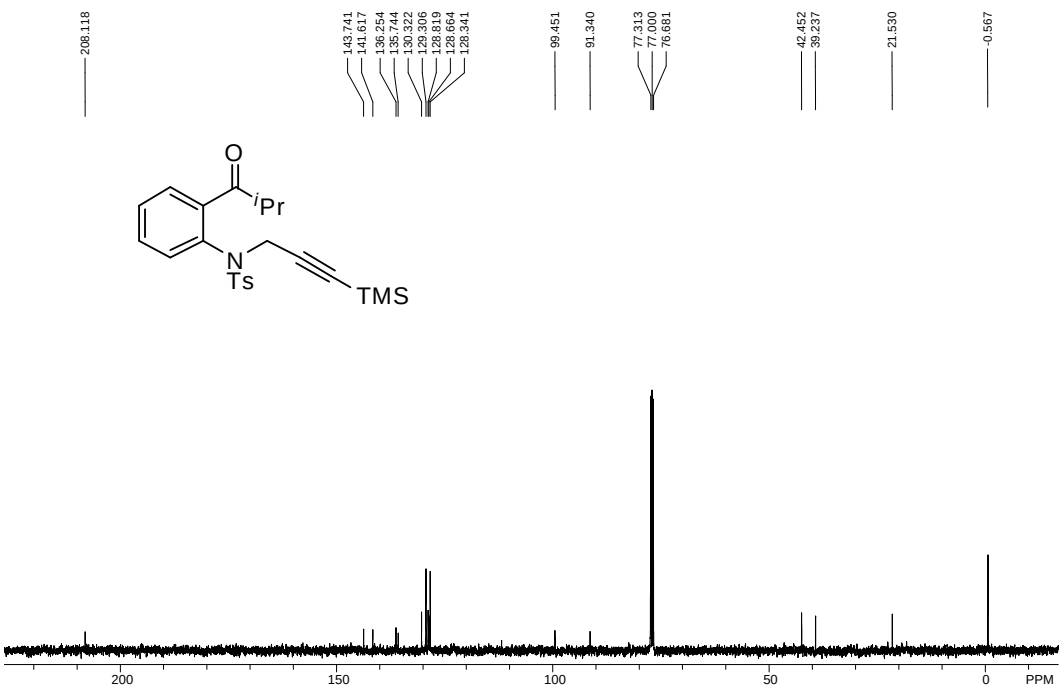
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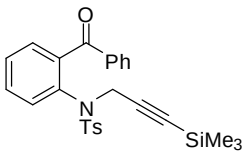
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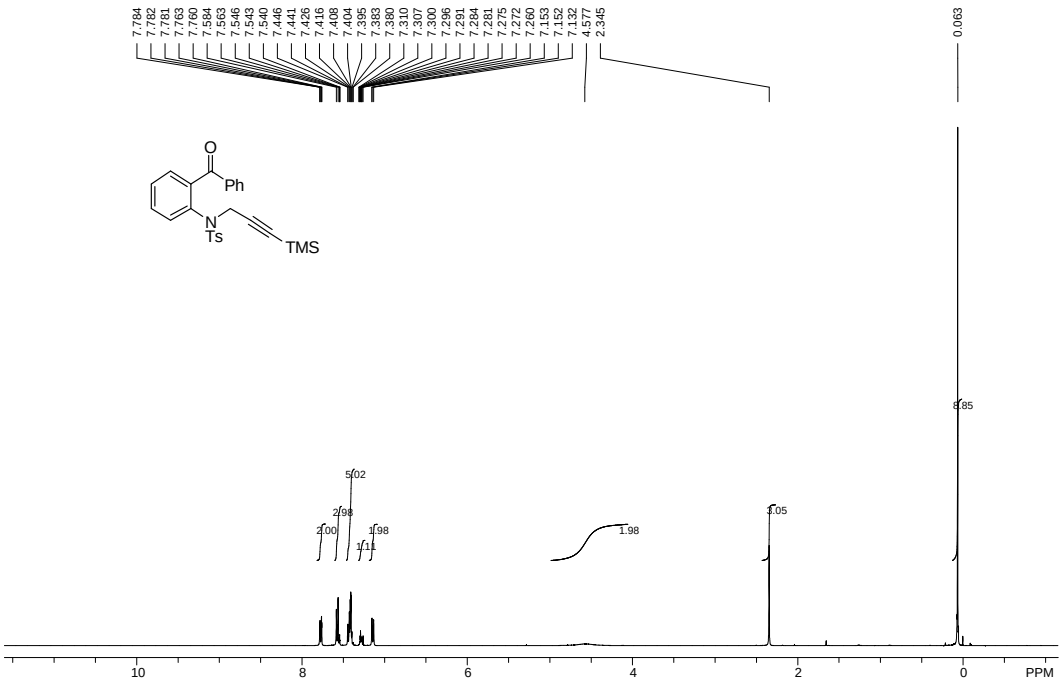
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7.383
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7.307
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7.281
7.275
7.272
7.260
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7.152
7.132
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Ph
N
Ts
TMS
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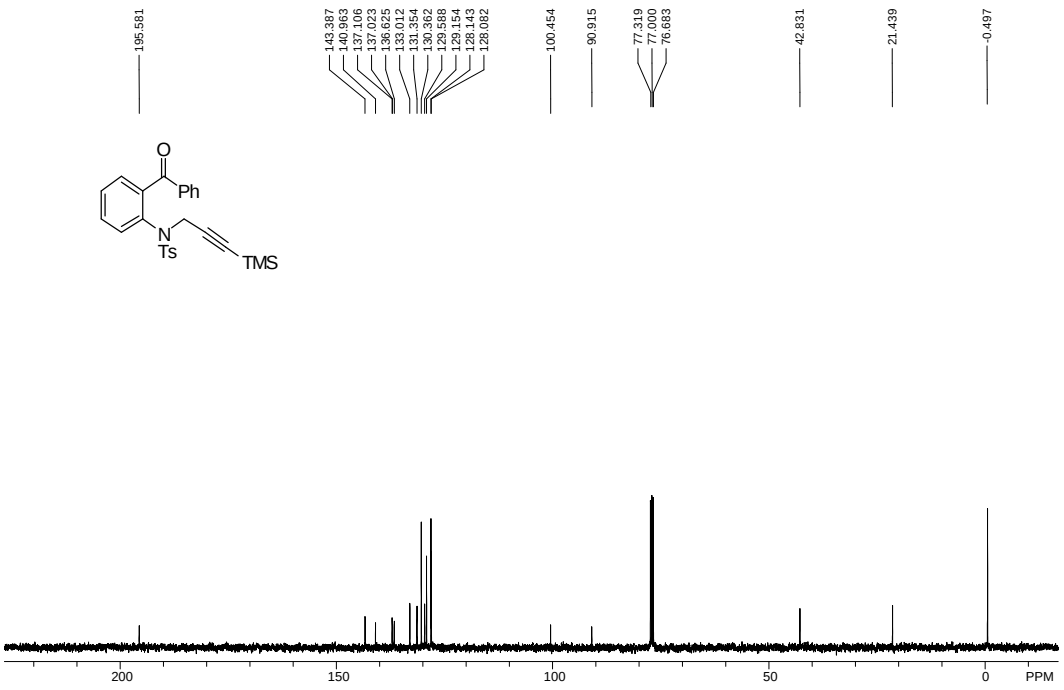
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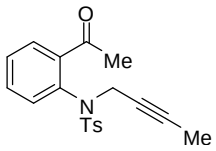
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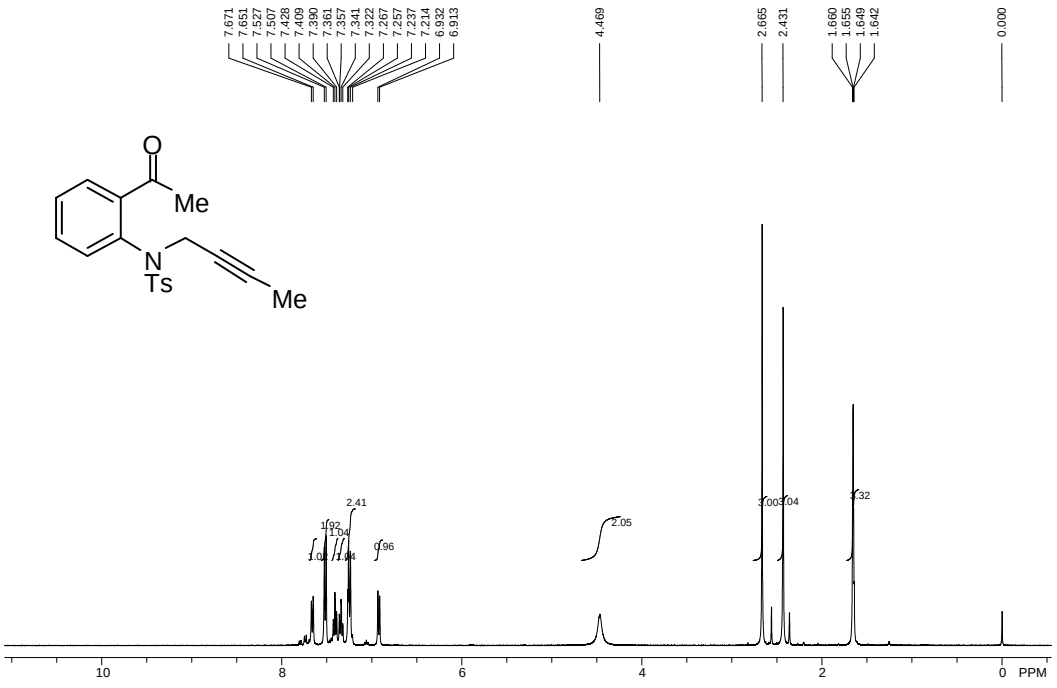
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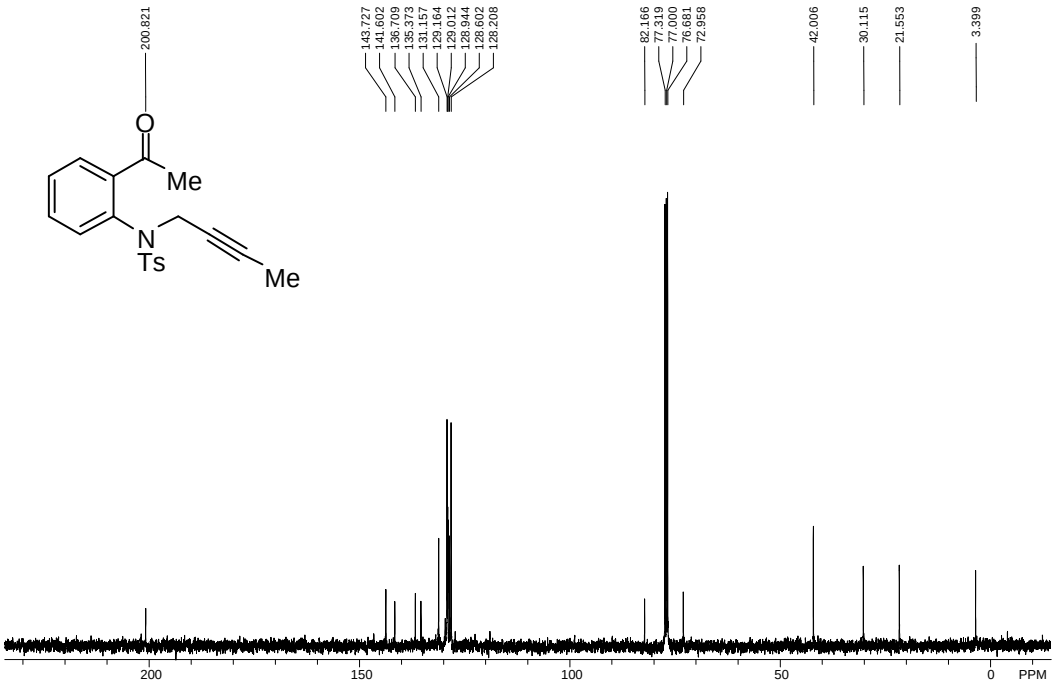
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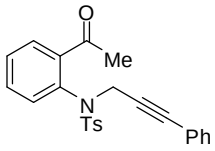
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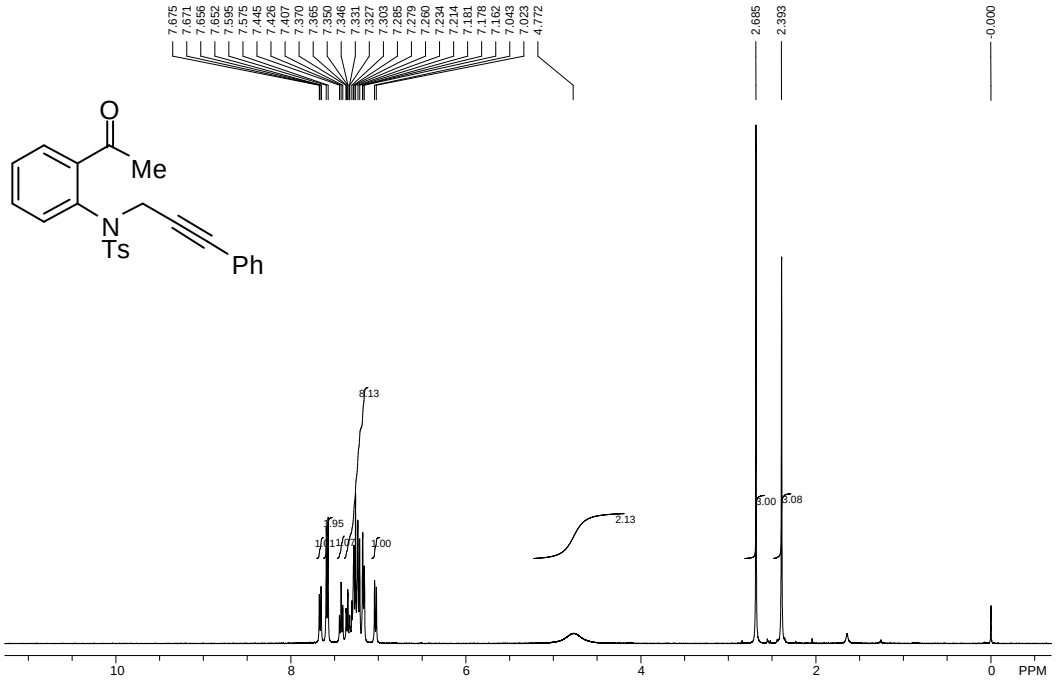
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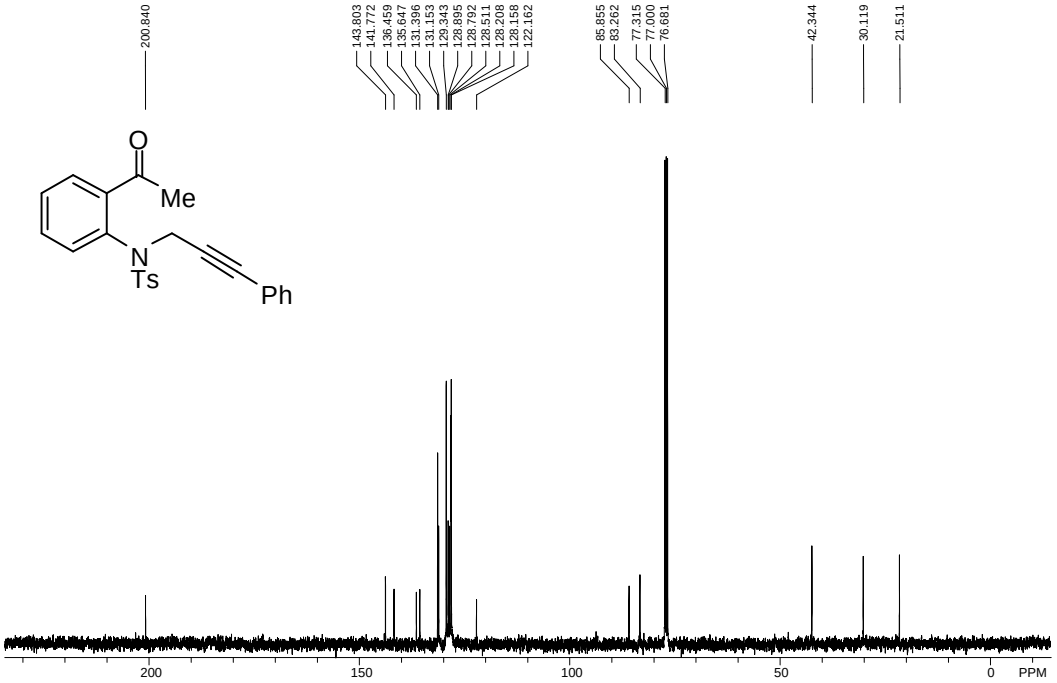
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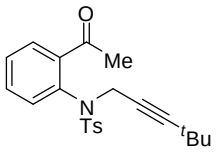
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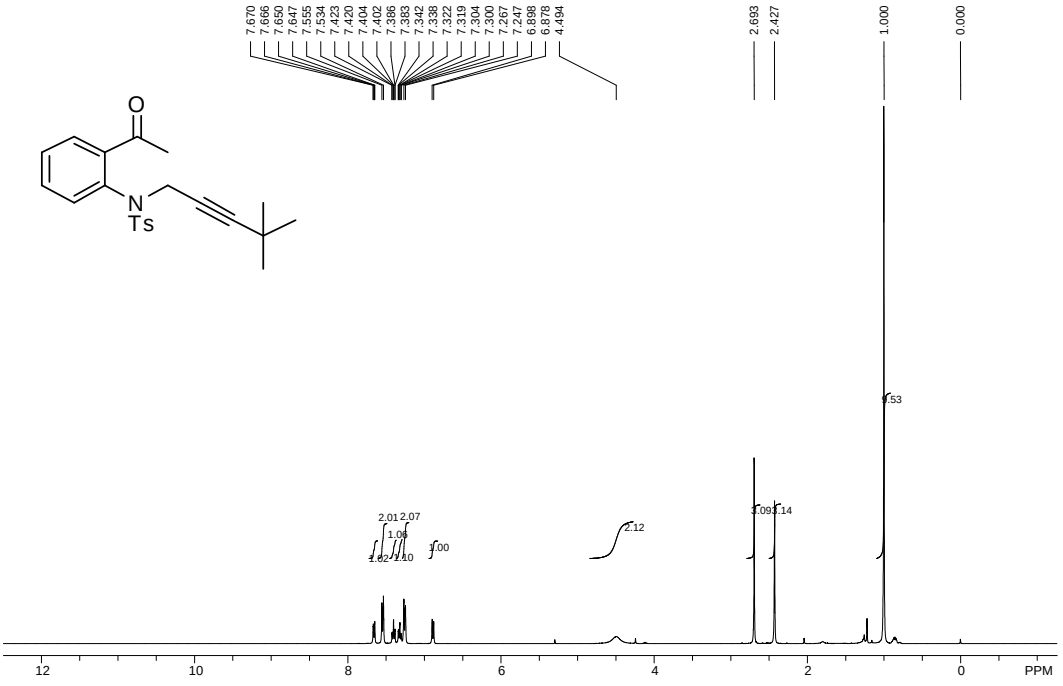
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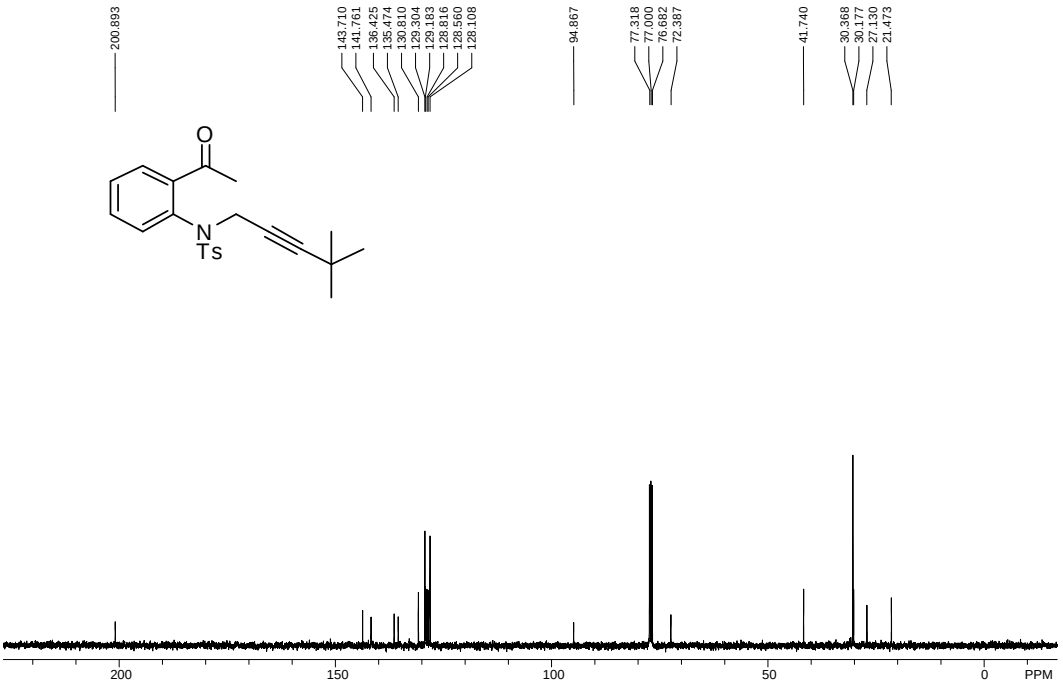
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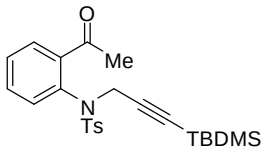
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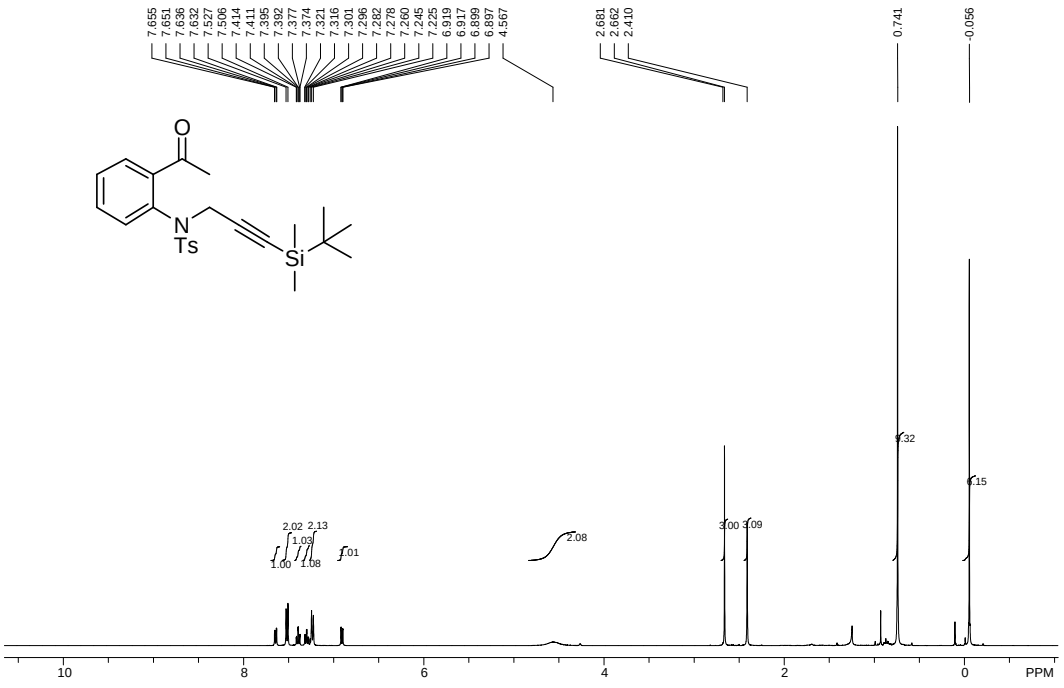
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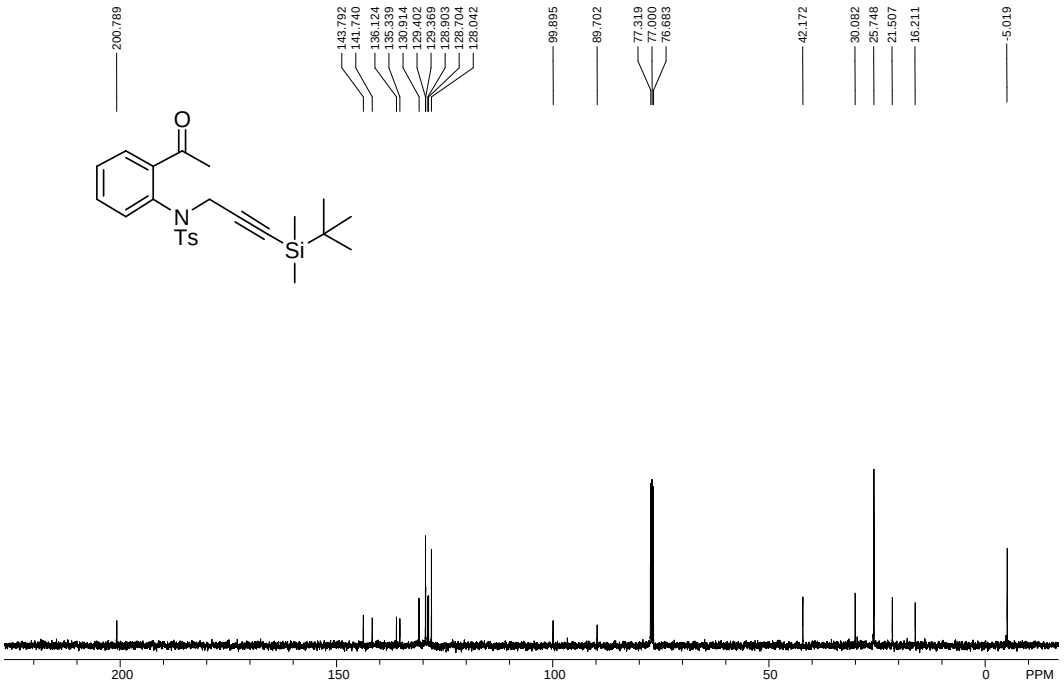
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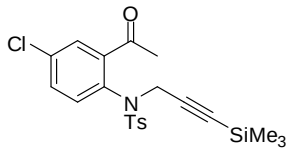
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SiMe3

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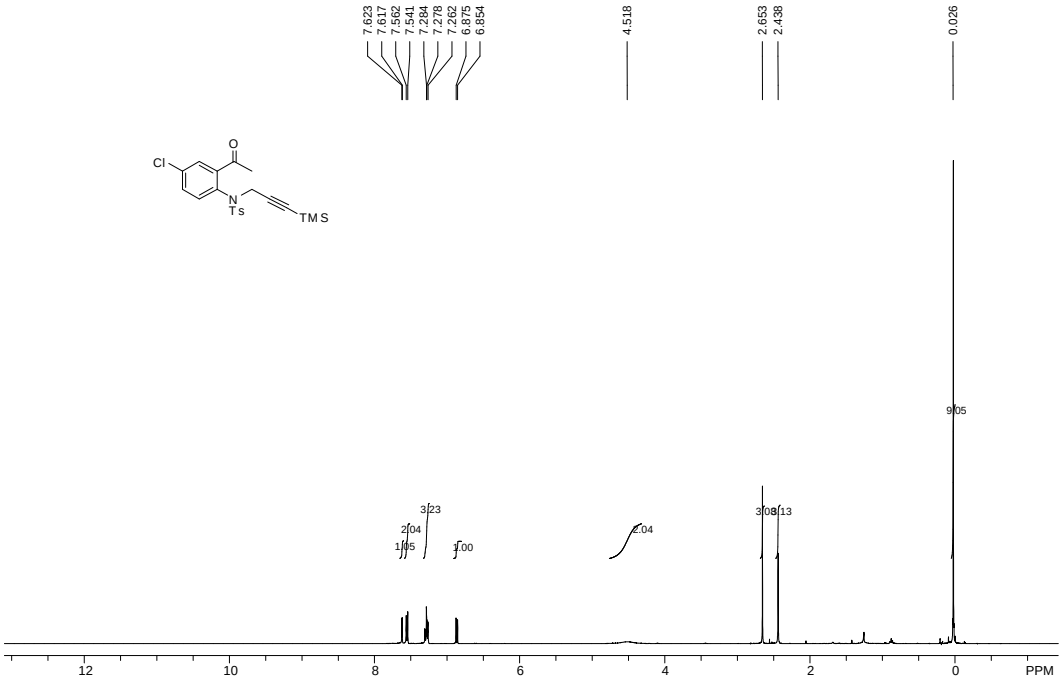
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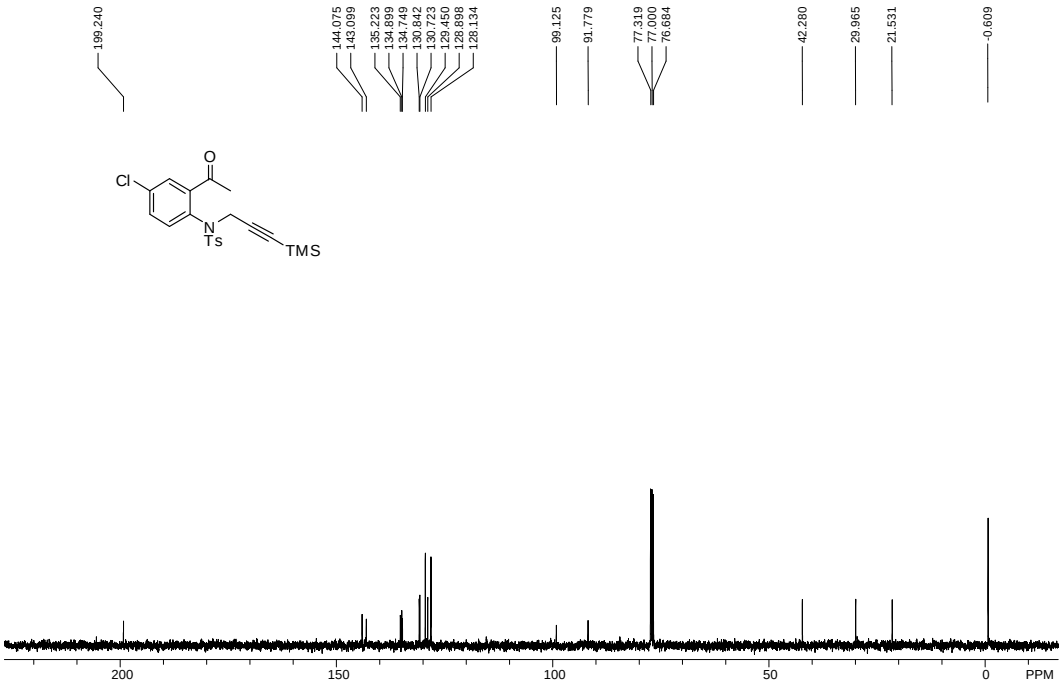
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TMS
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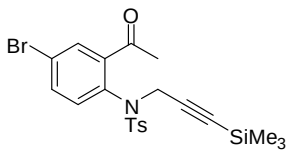
O
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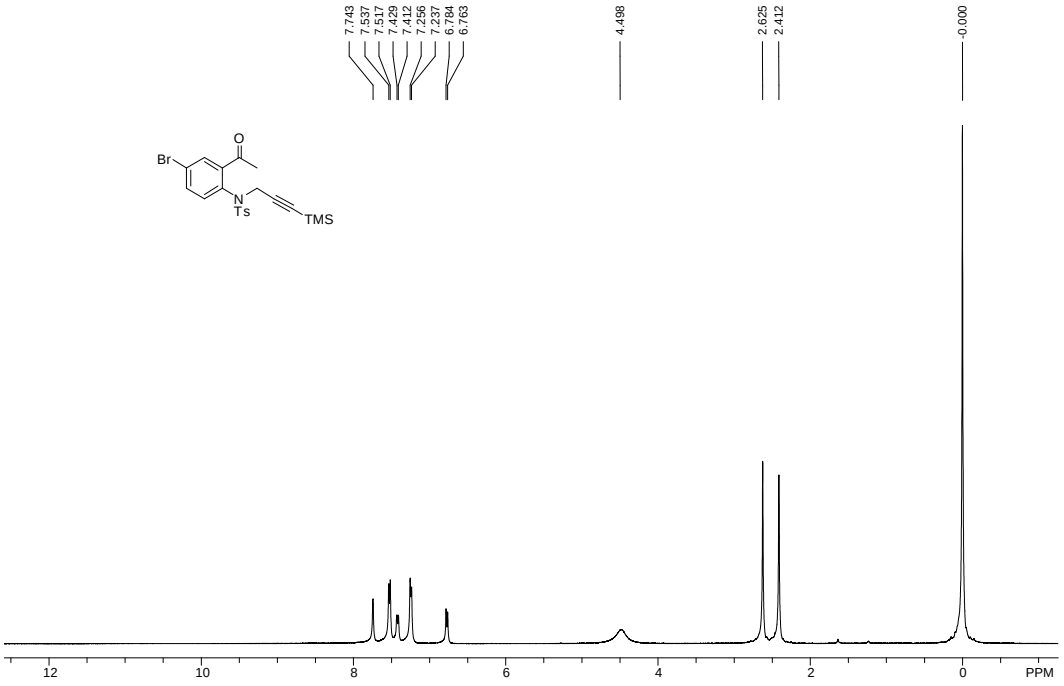
4j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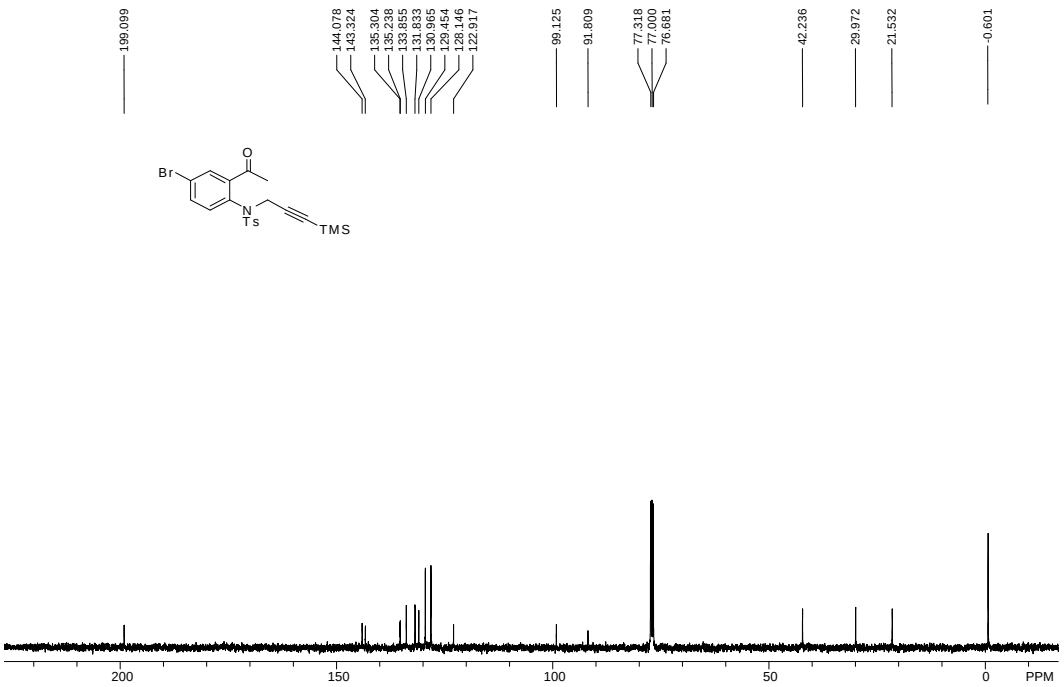
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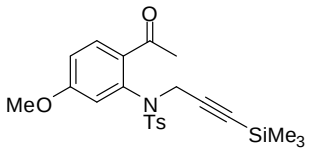
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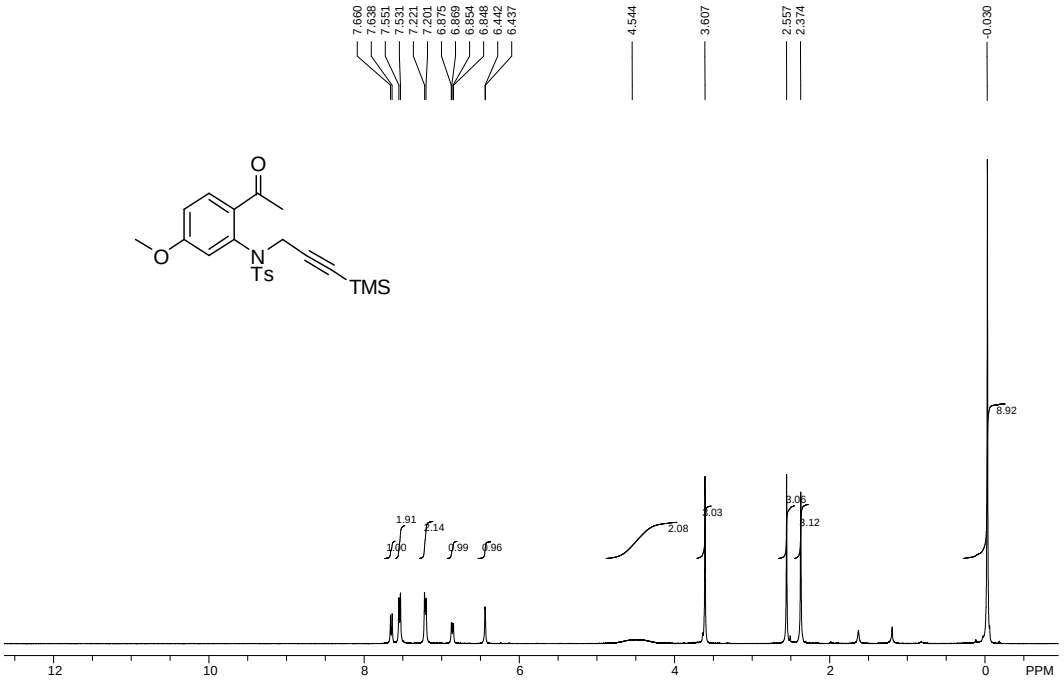
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O
N
Ts
TMS
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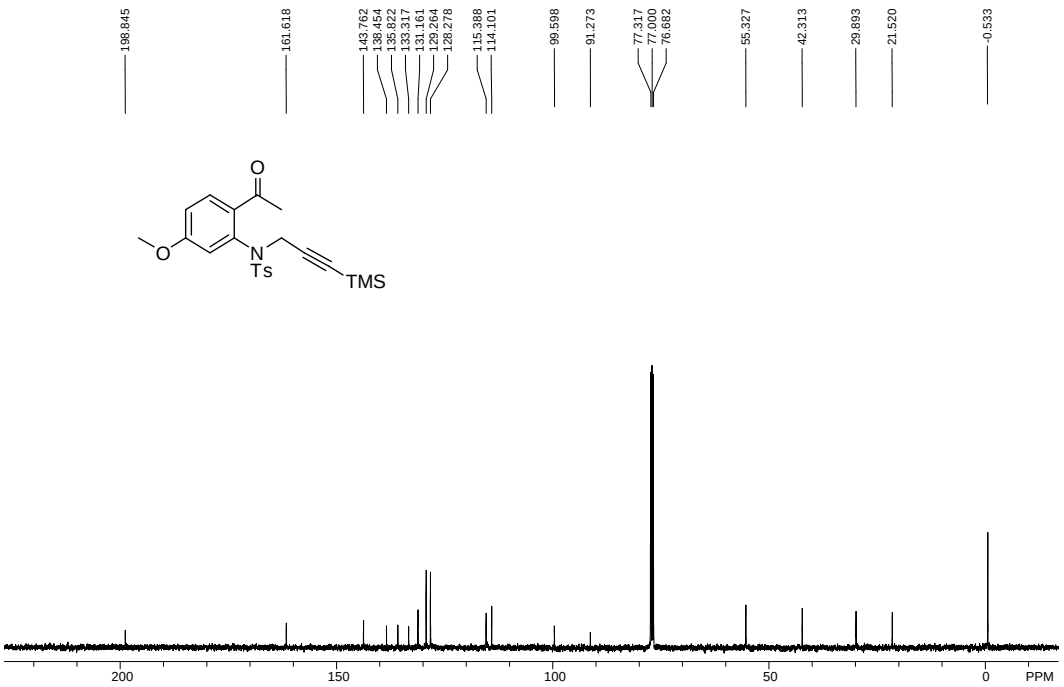
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TMS
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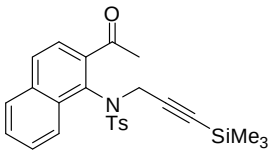
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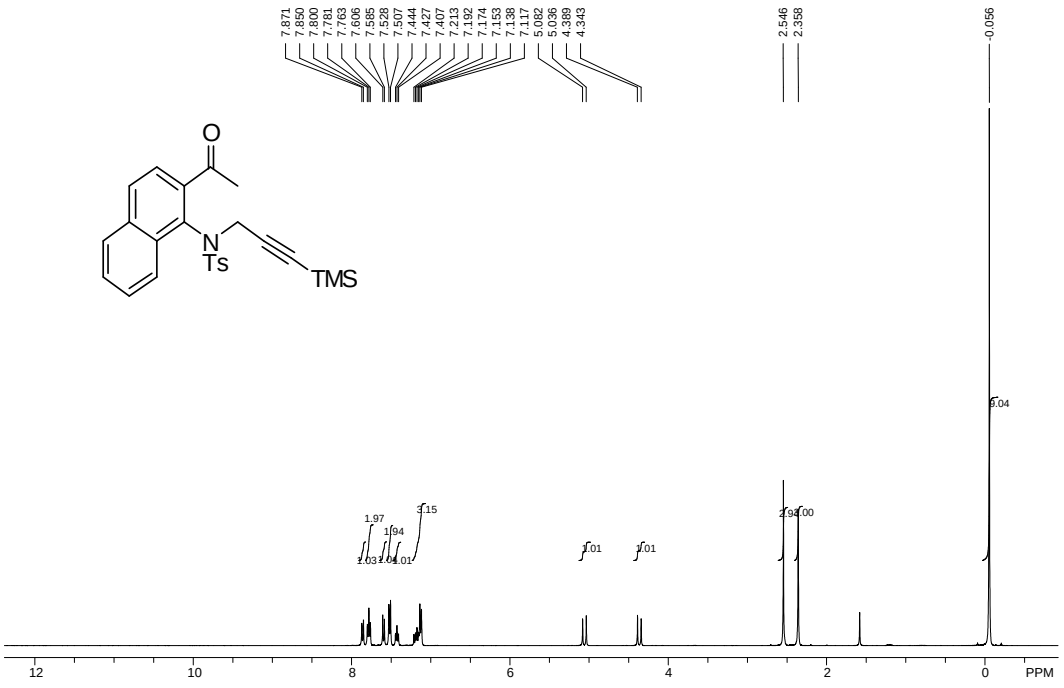
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N
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TMS
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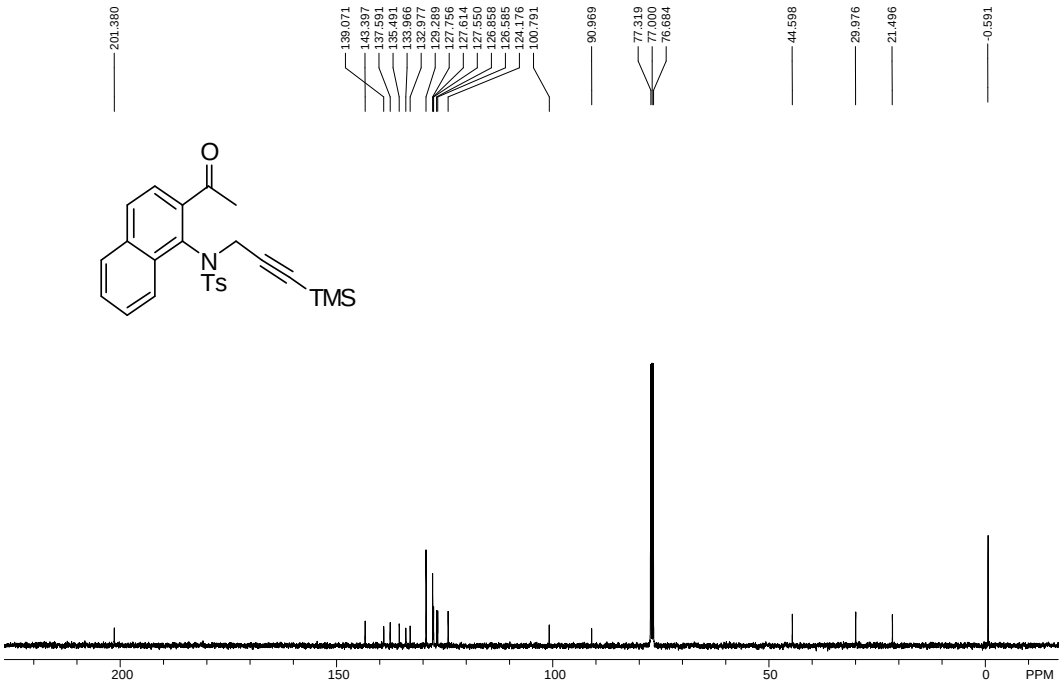
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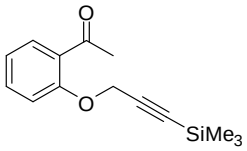
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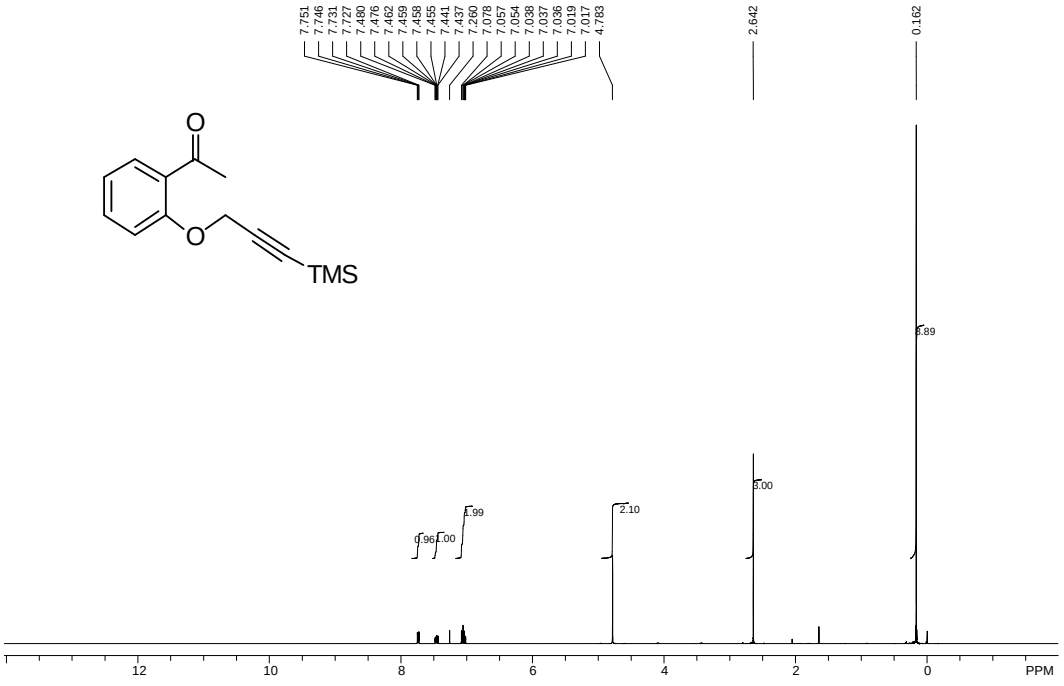
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7.260
7.078
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7.038
7.037
7.036
7.019
7.017
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O
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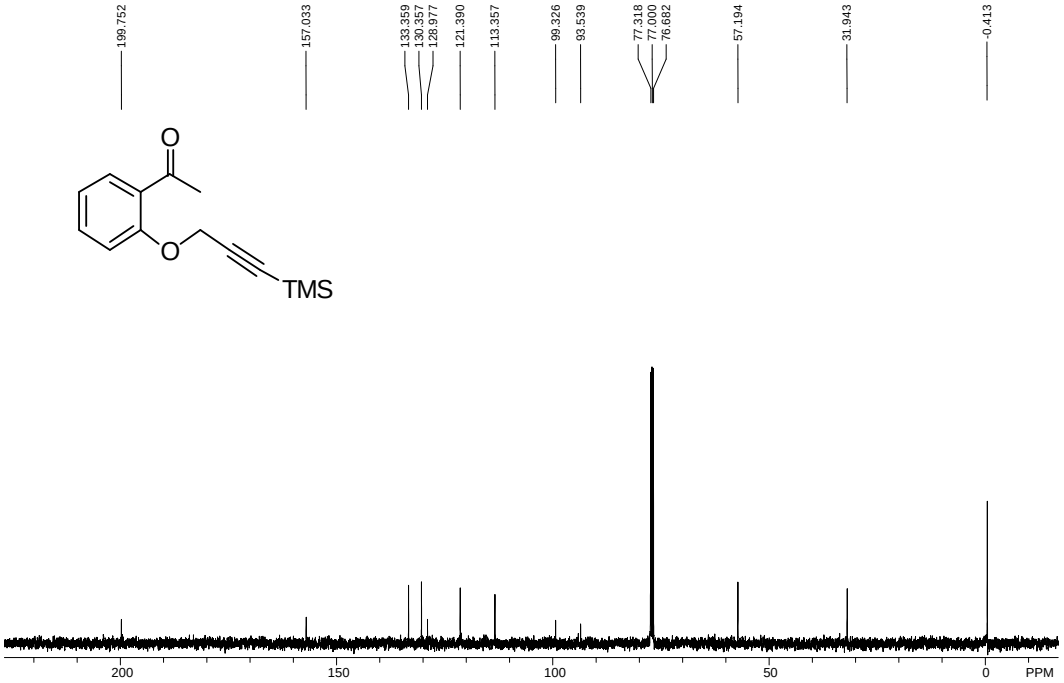
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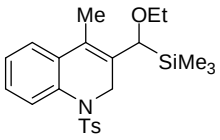
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SiMe3
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Ts

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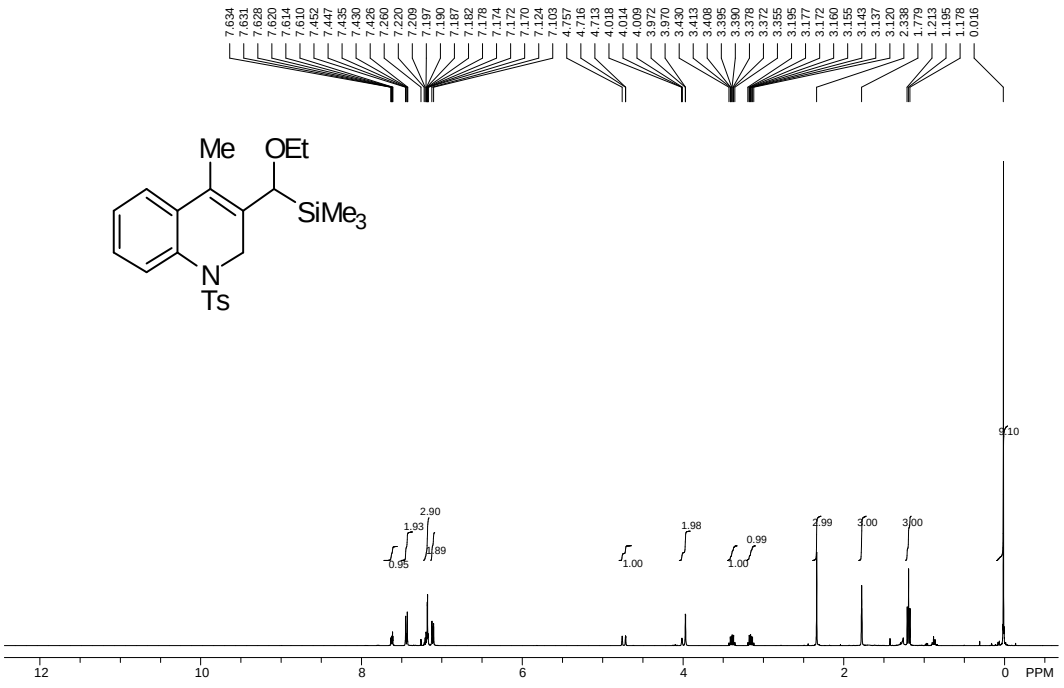
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7.197
7.190
7.187
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7.178
7.174
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7.170
7.124
7.103
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4.713
4.018
4.014
4.009
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Me
OEt
SiMe3
N
Ts
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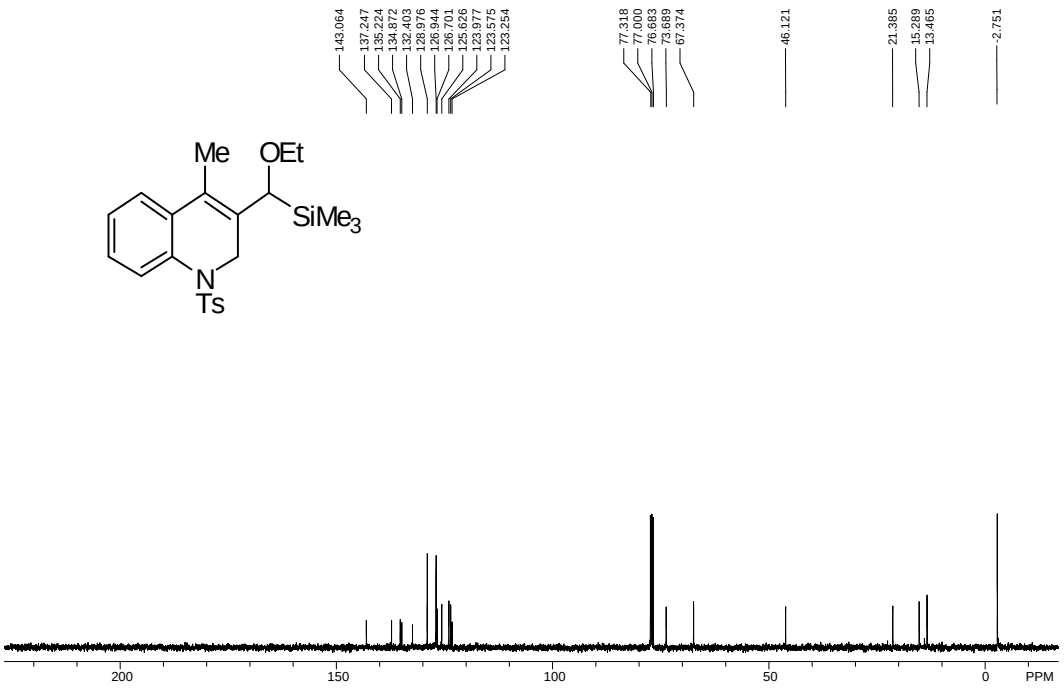
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SiMe3
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Ts
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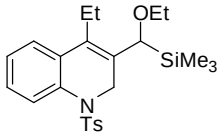
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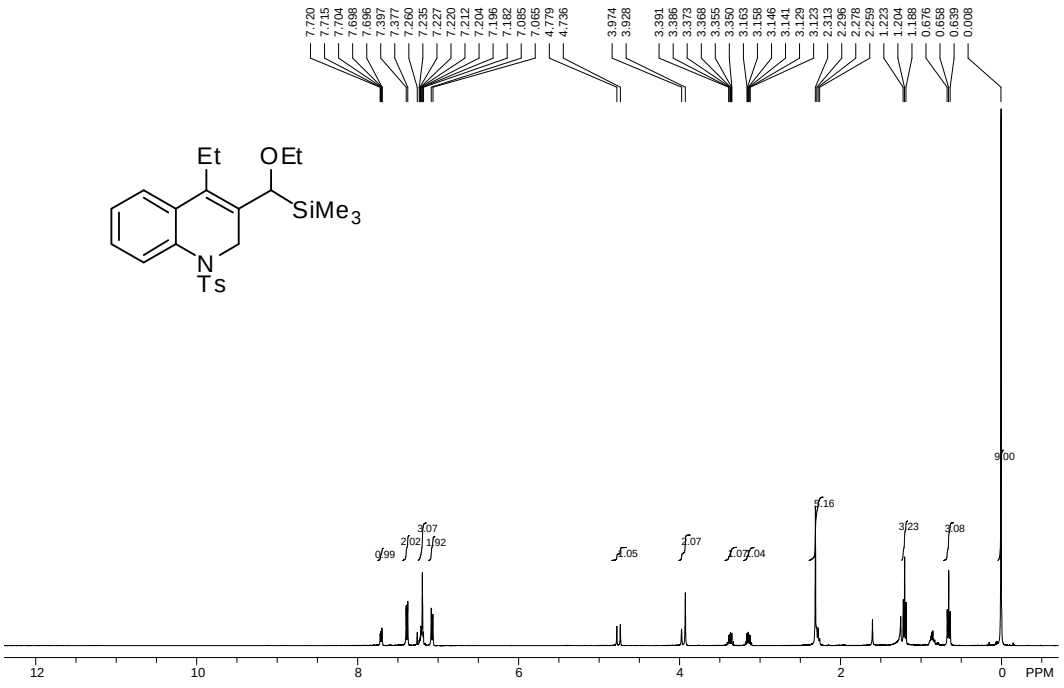
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3.146
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SiMe3
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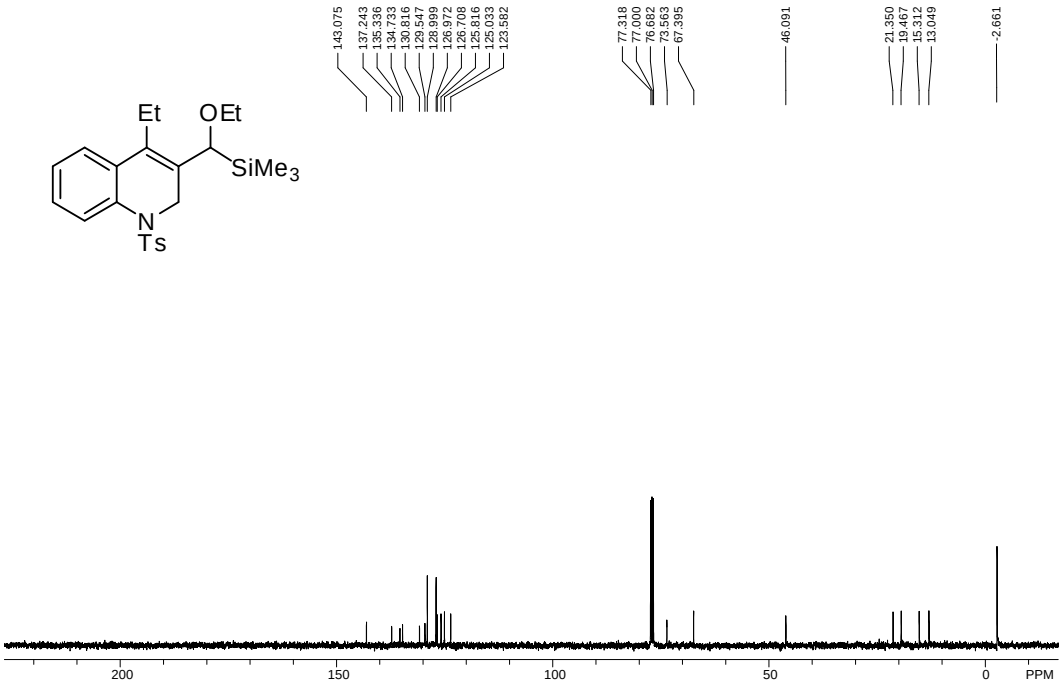
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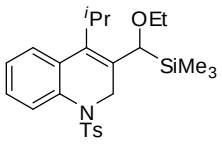
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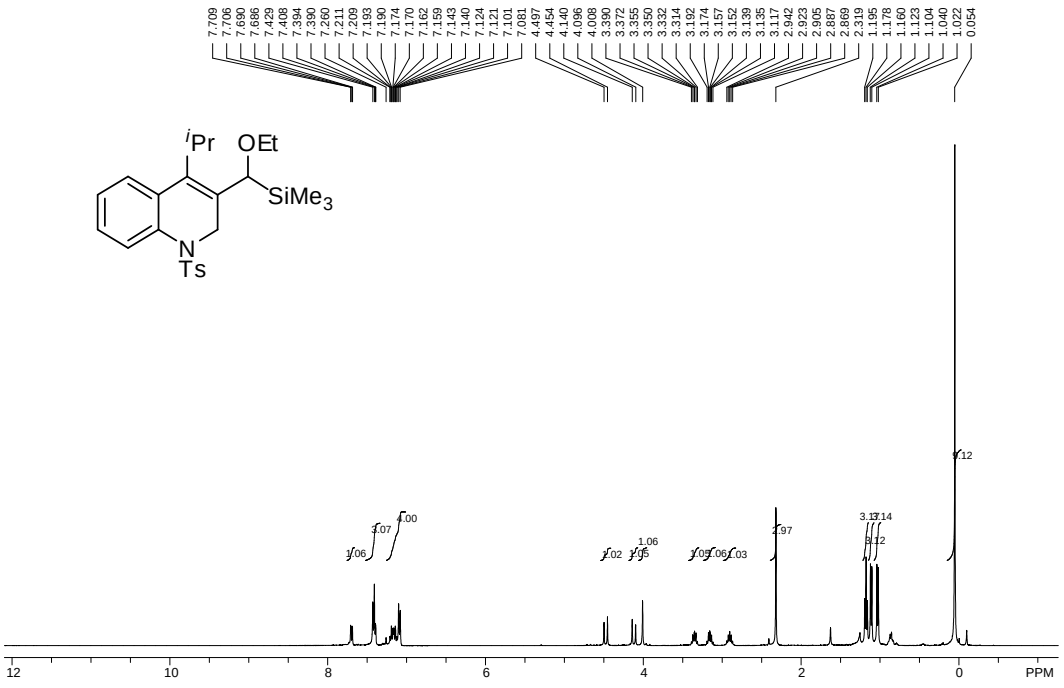
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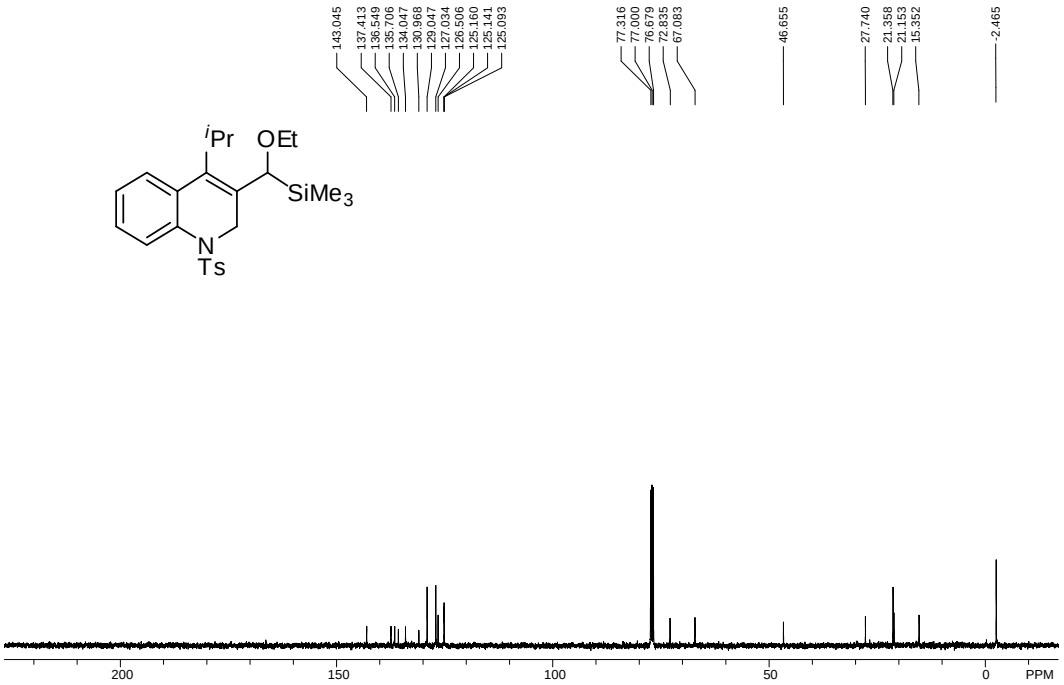
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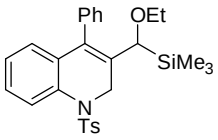
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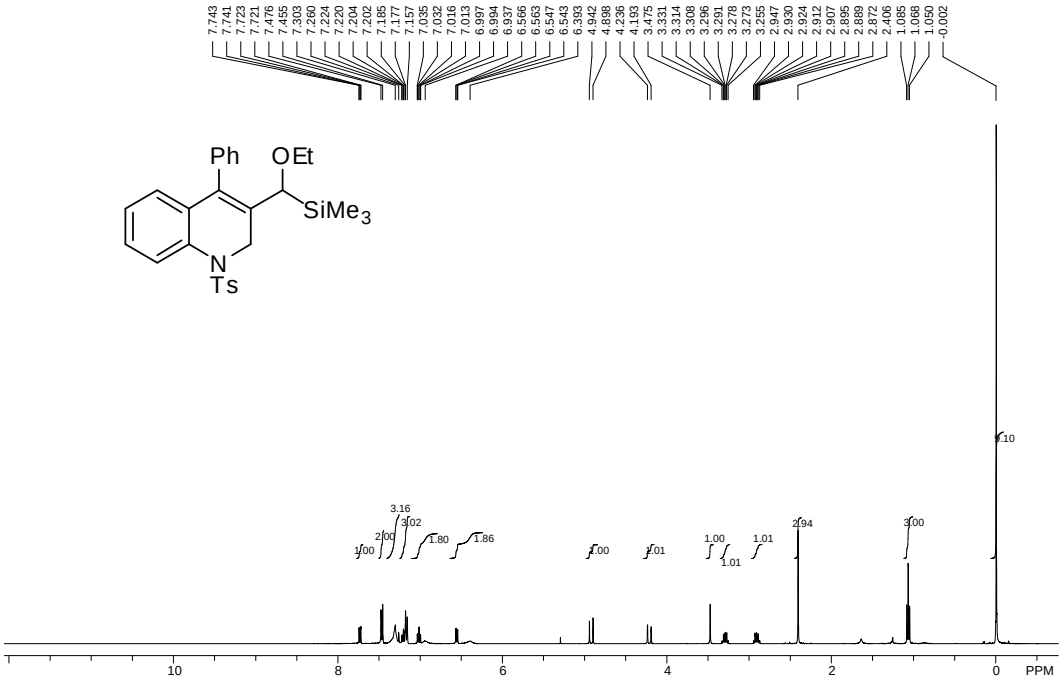
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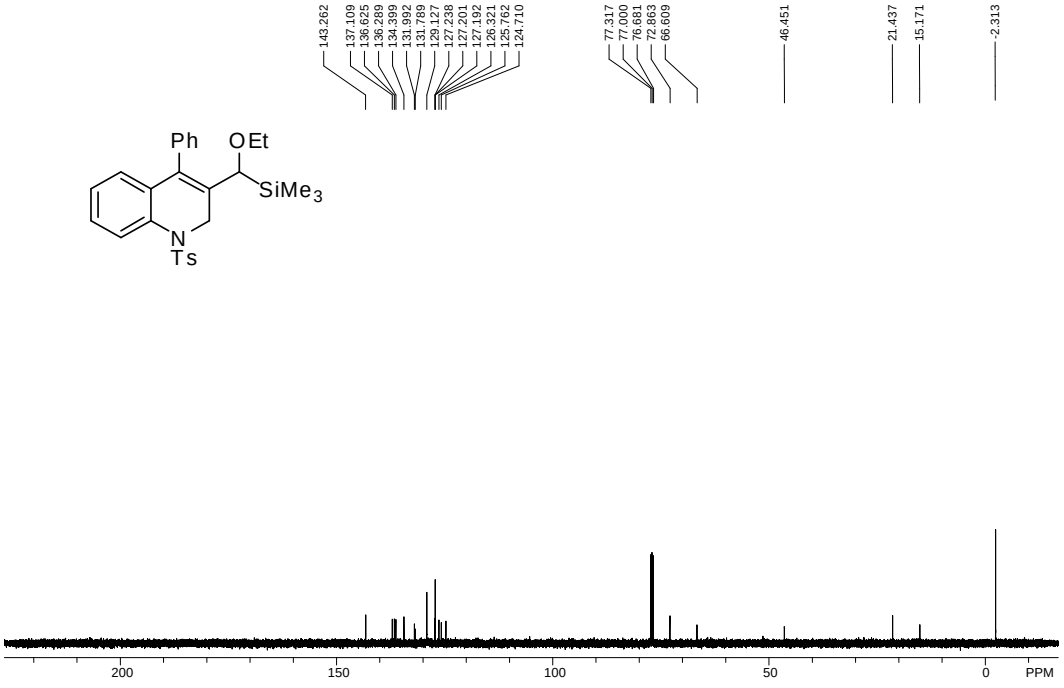
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Ts
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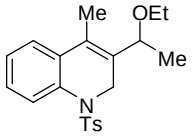
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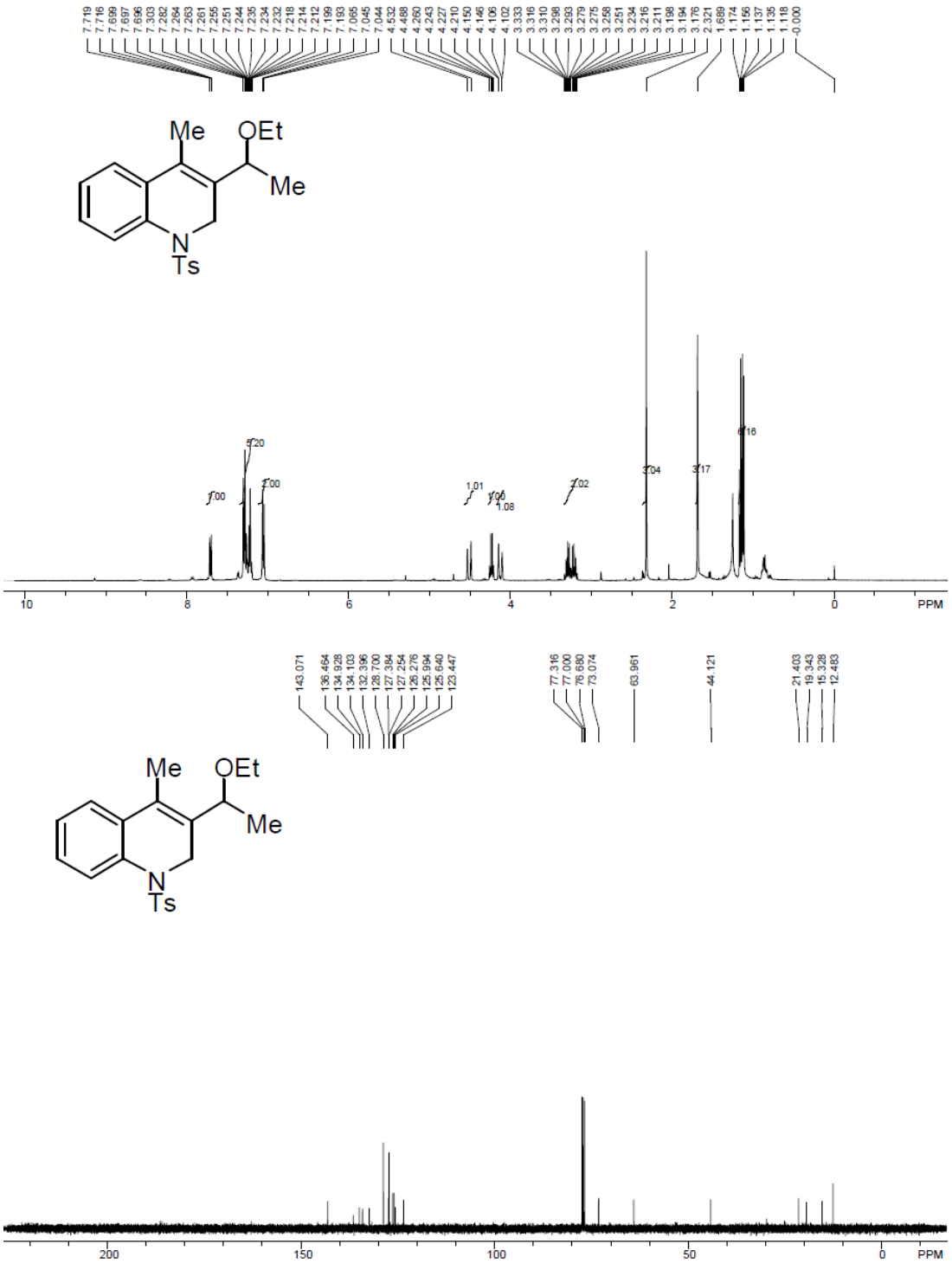
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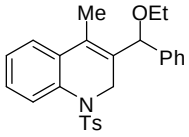

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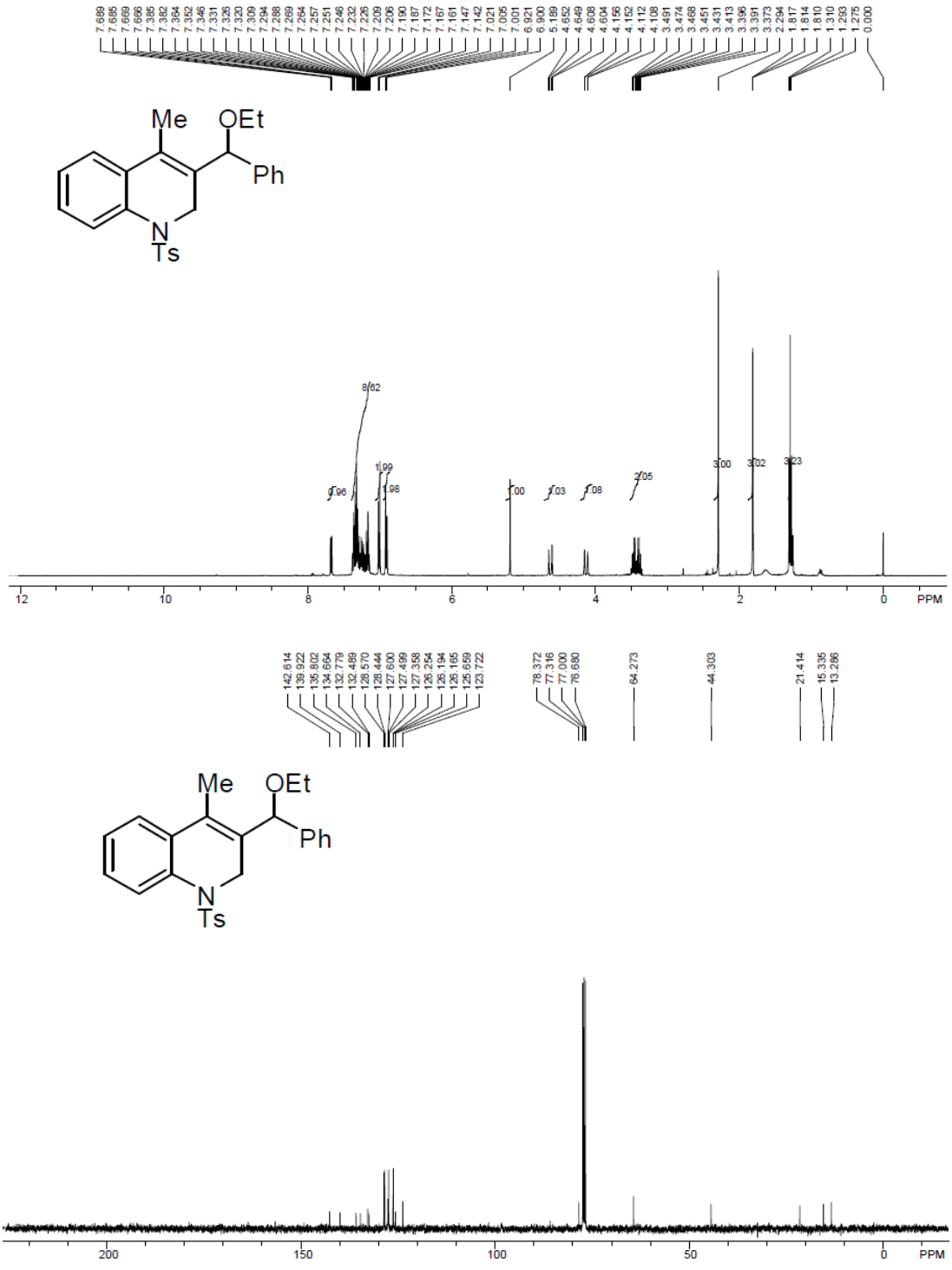

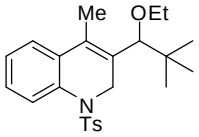
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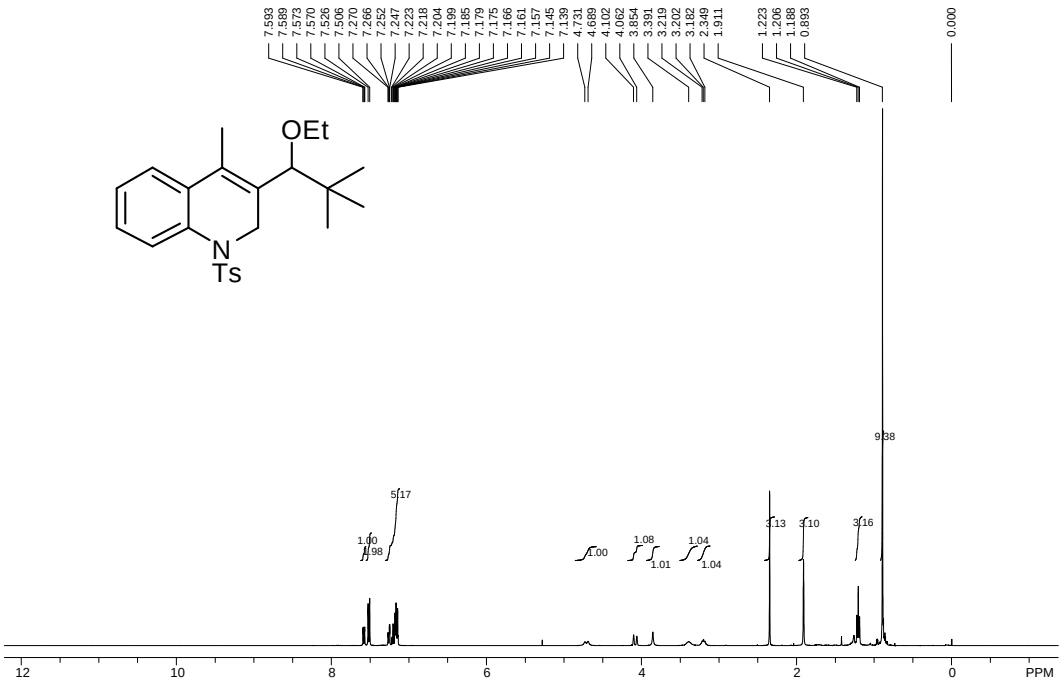
Ts

5g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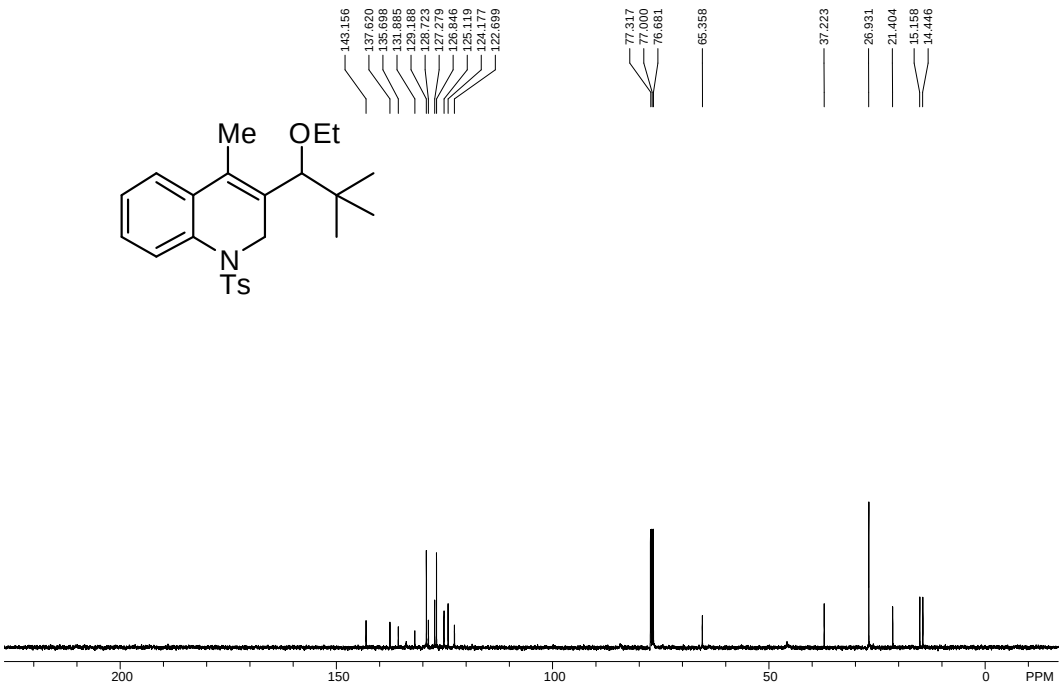
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76.681
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N
Ts
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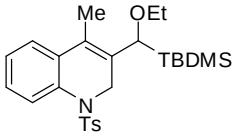
PPM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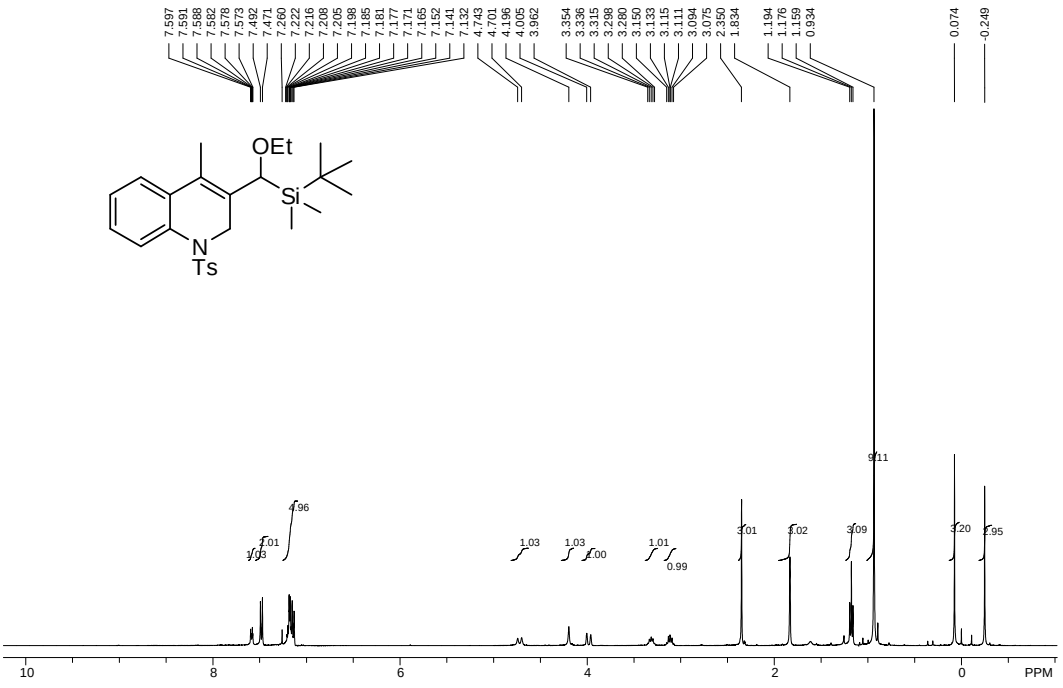
TBDMS
N
Ts

5h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7.205
7.198
7.185
7.181
7.177
7.171
7.165
7.152
7.141
7.132
4.743
4.701
4.196
4.005
3.962
3.354
3.336
3.315
3.298
3.280
3.150
3.133
3.115
3.111
3.094
3.075
2.350
1.834
1.194
1.176
1.159
0.934
0.074
-0.249
OEt
Si
N
Ts
4.96
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1.03
1.03
1.00
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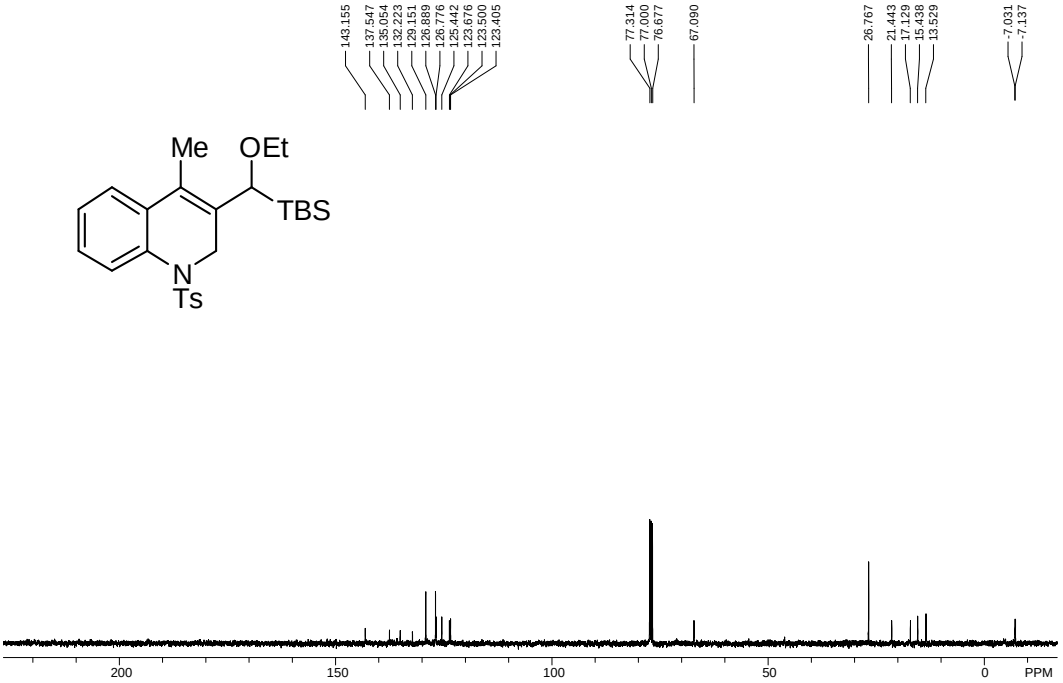
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123.500
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67.090
26.767
21.443
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TBS
N
Ts
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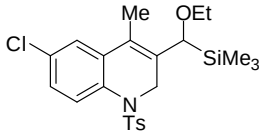
150
100
50
0
PPM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Cl
SiMe3
N
Ts

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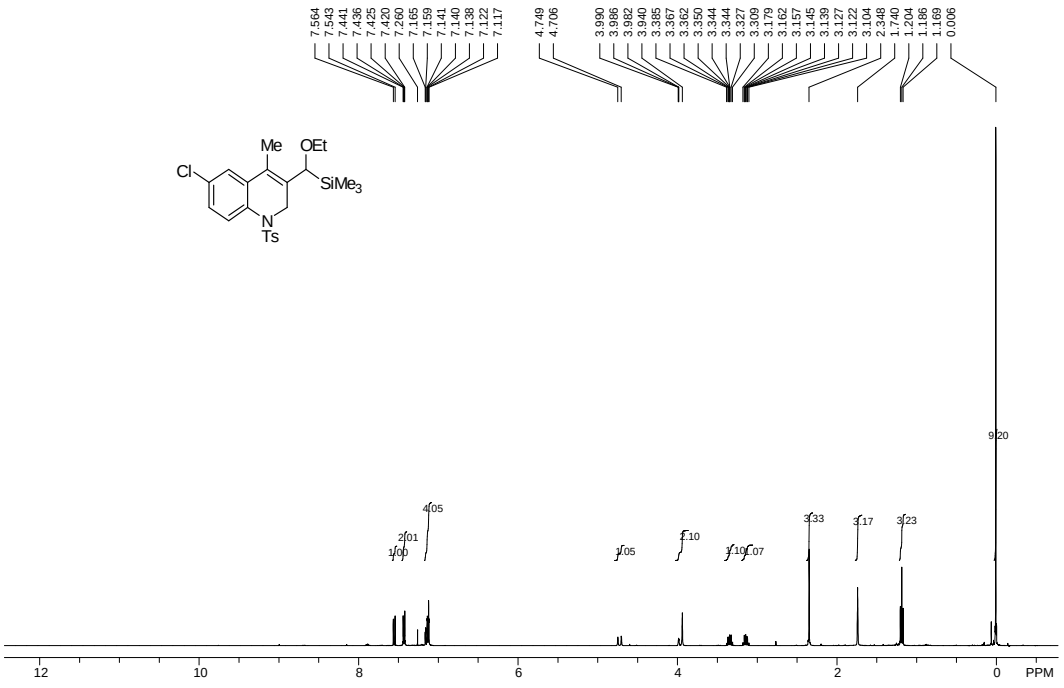
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4.706
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3.982
3.940
3.385
3.367
3.362
3.350
3.344
3.344
3.327
3.309
3.179
3.162
3.157
3.145
3.139
3.127
3.122
3.104
2.348
1.740
1.204
1.186
1.169
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OEt
Cl
SiMe3
N
Ts
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4.05
2.01
1.00
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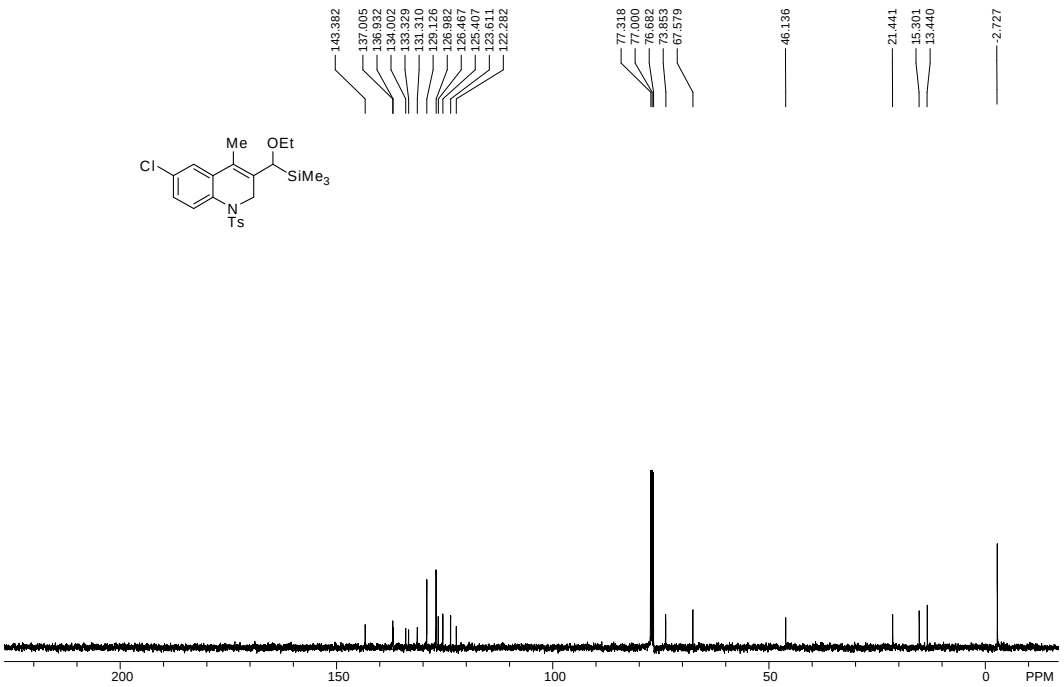
1.07
3.33
3.17
3.23
12
10
8
6
4
2
0
PPM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67.579
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Cl
SiMe3
N
Ts
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100
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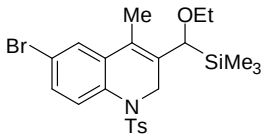
PPM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Br
SiMe3
N
Ts

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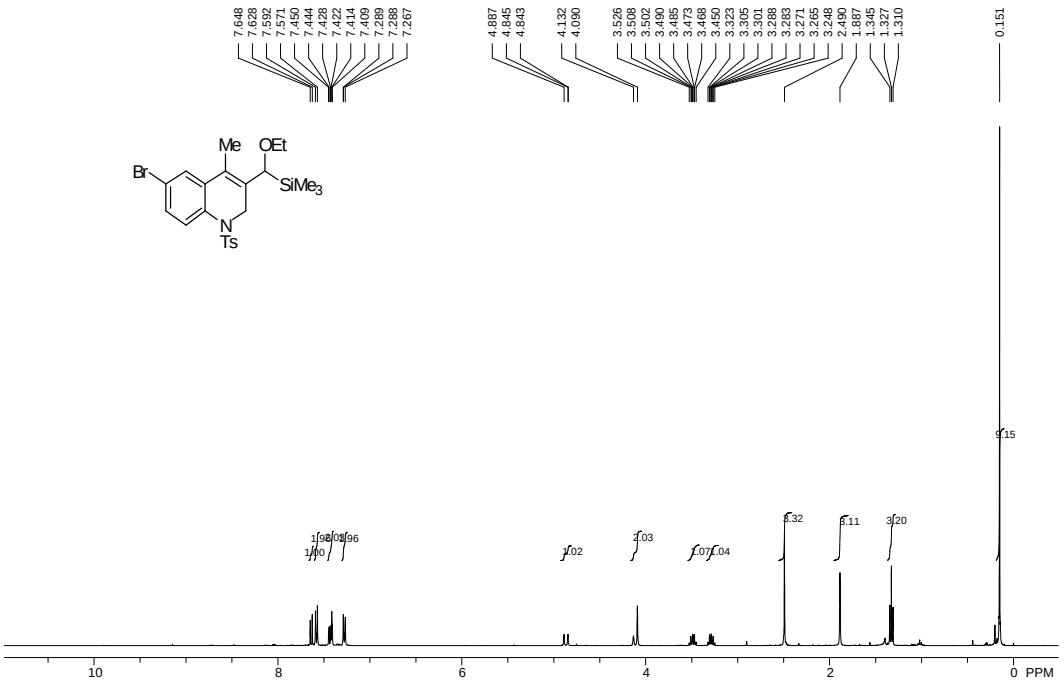
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7.422
7.414
7.409
7.289
7.288
7.267
4.887
4.845
4.843
4.132
4.090
3.526
3.508
3.502
3.490
3.485
3.473
3.468
3.450
3.323
3.305
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3.288
3.283
3.271
3.265
3.248
2.490
1.887
1.345
1.327
1.310
0.151
Me
OEt
Br
SiMe3
N
Ts
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2.03
1.02
1.07
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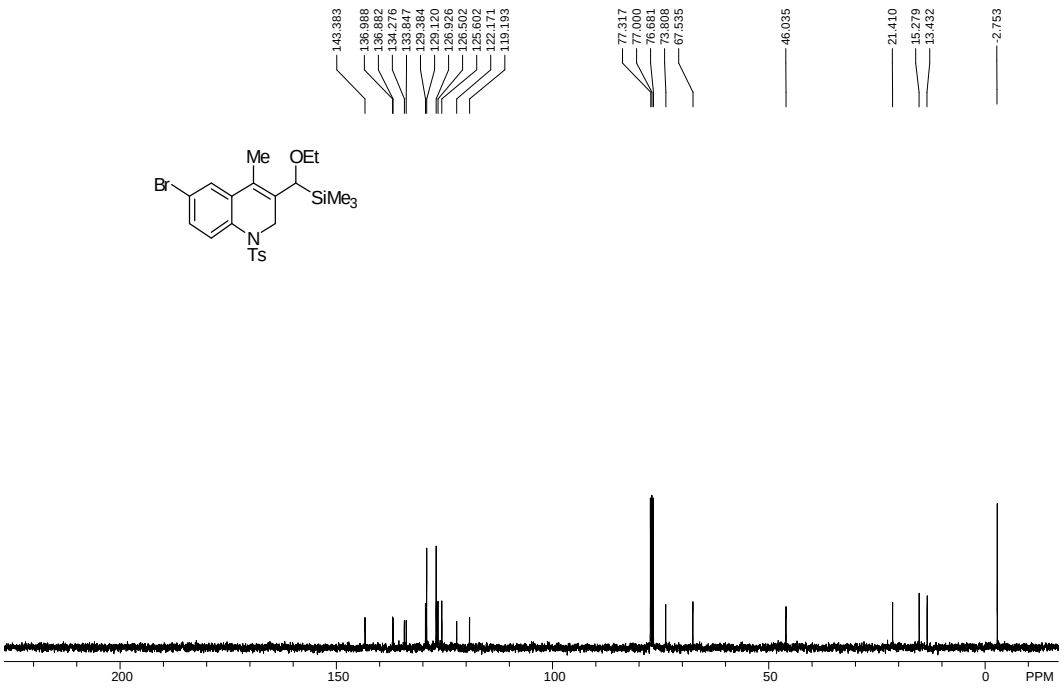
PPM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77.000
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46.035
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N
Ts
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100
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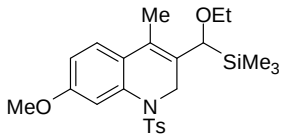
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Ts

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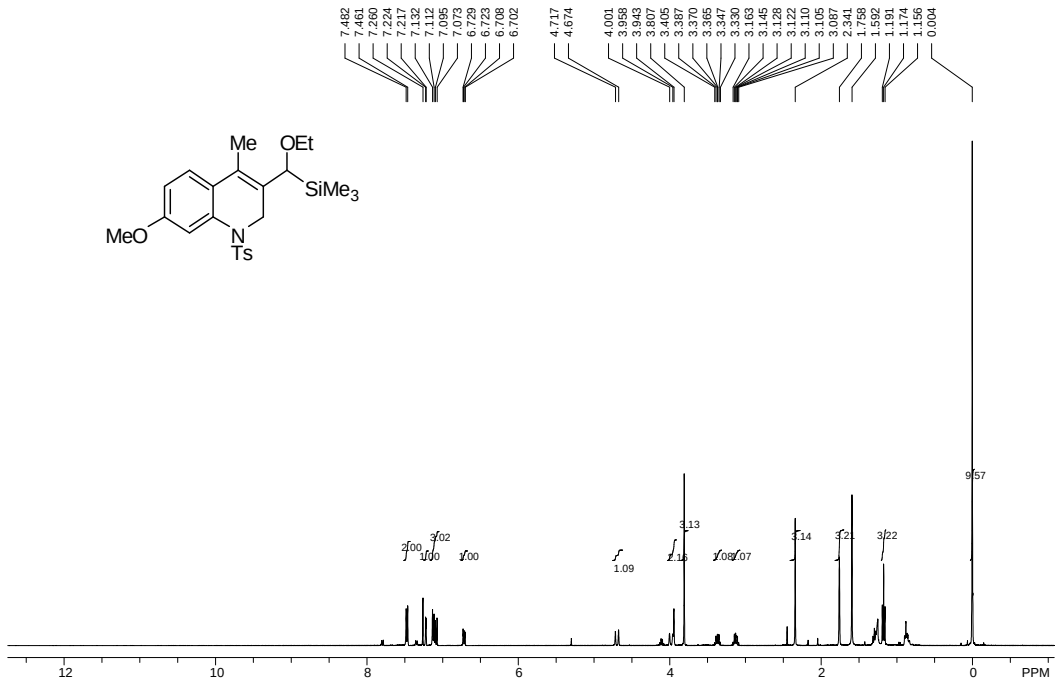
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6.702
4.717
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4.001
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3.943
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3.405
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3.365
3.347
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3.128
3.122
3.110
3.105
3.087
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1.191
1.174
1.156
0.004
Me
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MeO
N
Ts
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3.02
1.00
1.00
1.09
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10
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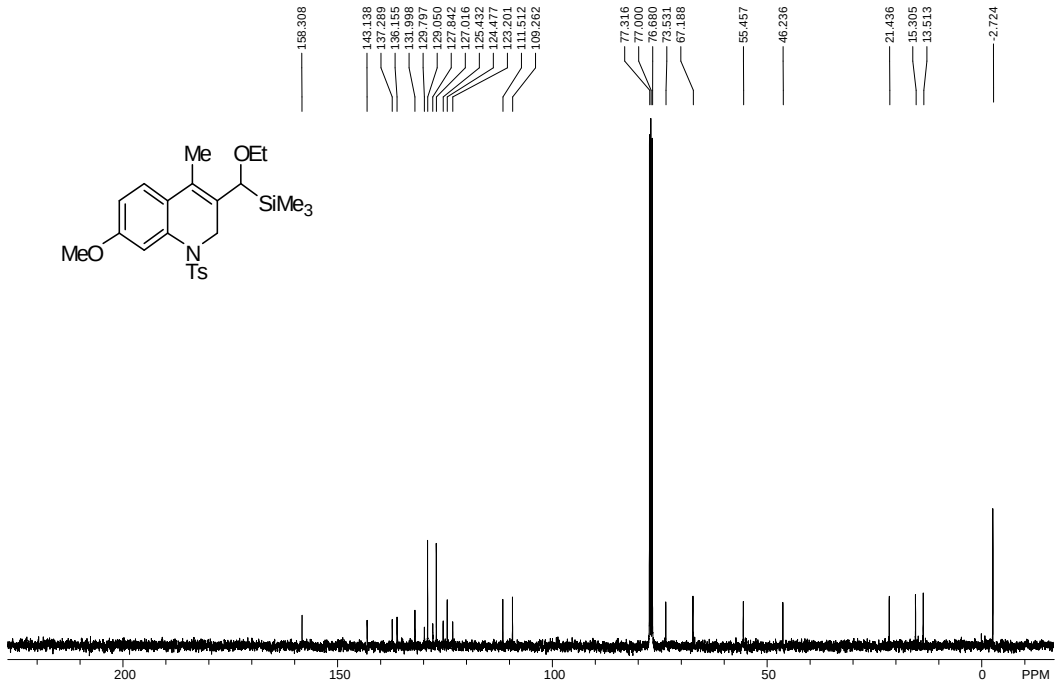
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124.477
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77.316
77.000
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MeO
N
Ts
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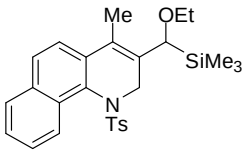
0
PPM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N
Ts

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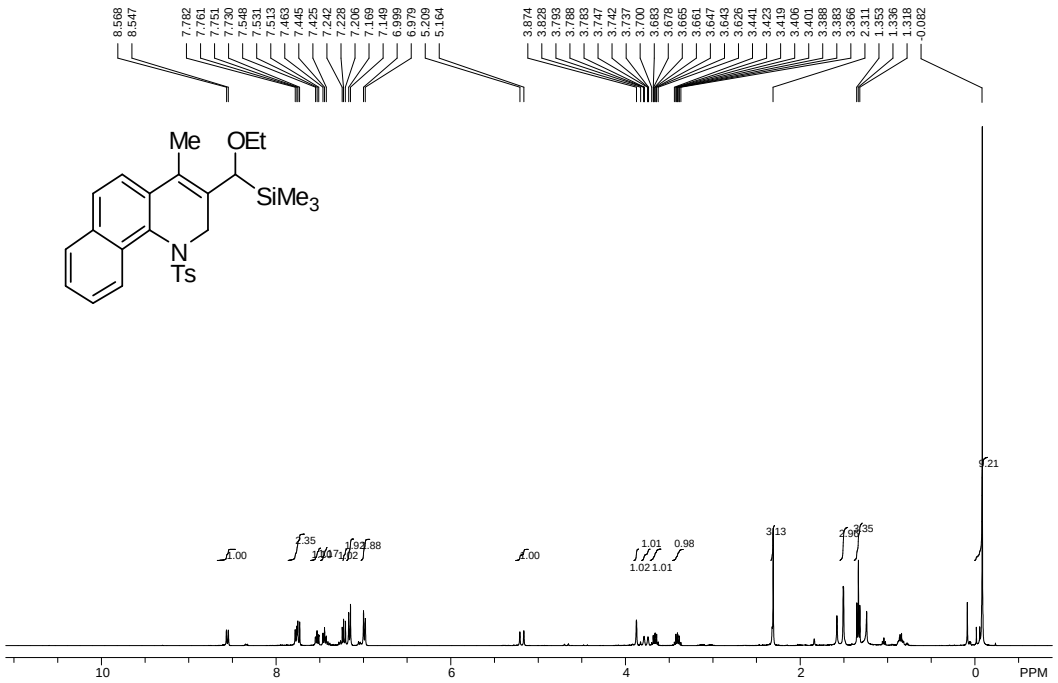
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7.242
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SiMe3
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Ts
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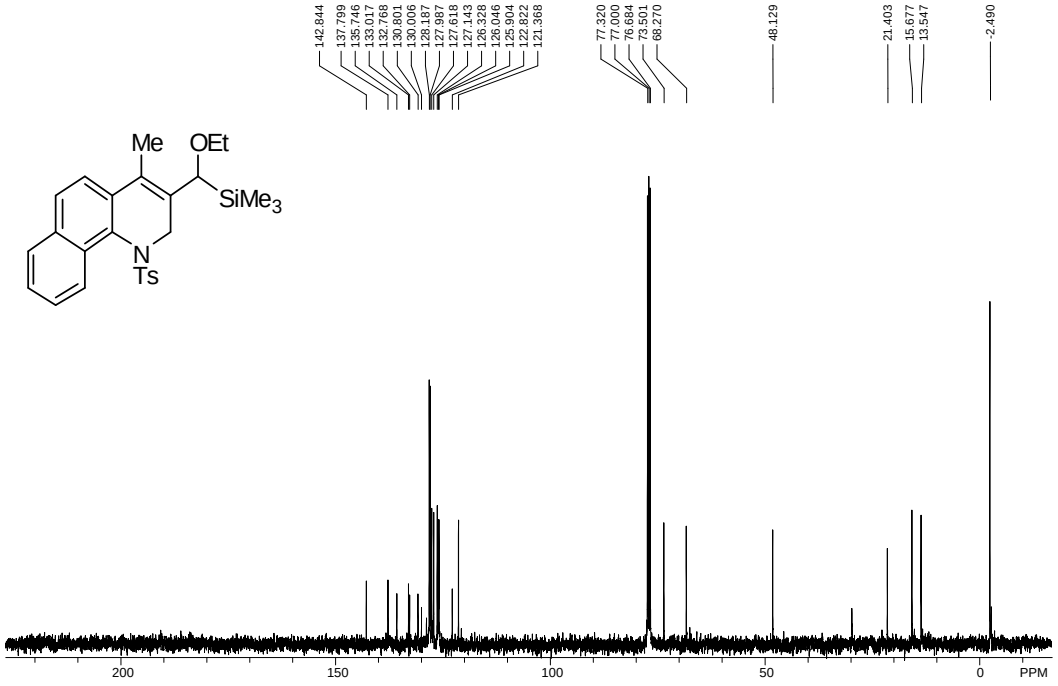
PPM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N
Ts
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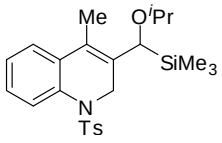
PPM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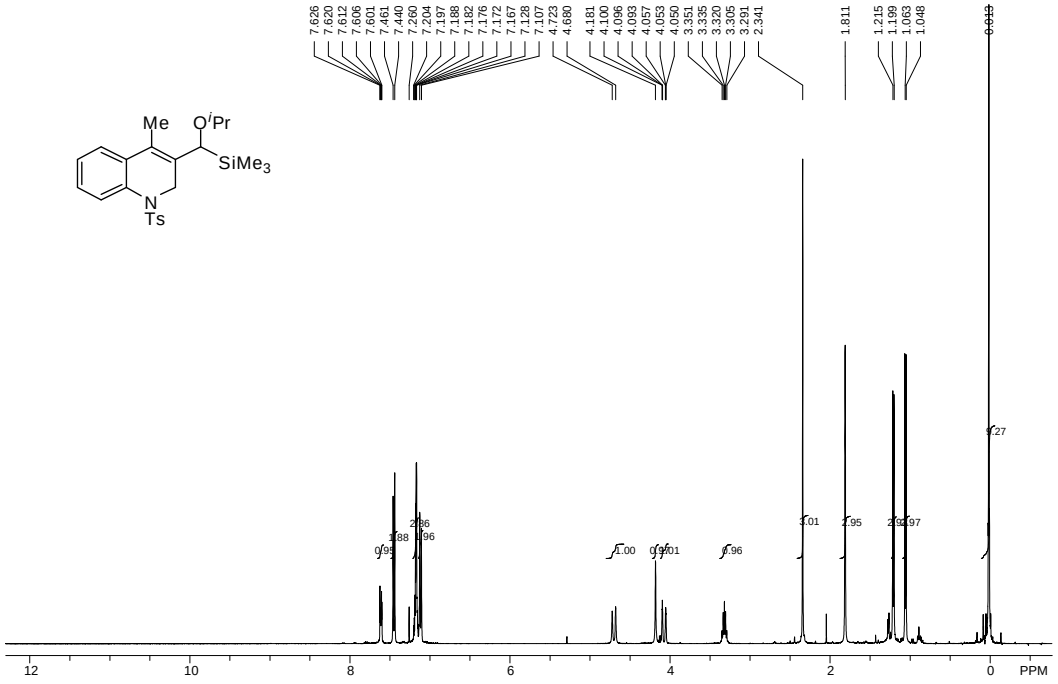
SiMe$_3$
N
Ts

5n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1.215
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1.063
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0.013
Me
O^{i}Pr
SiMe$_3$
N
Ts
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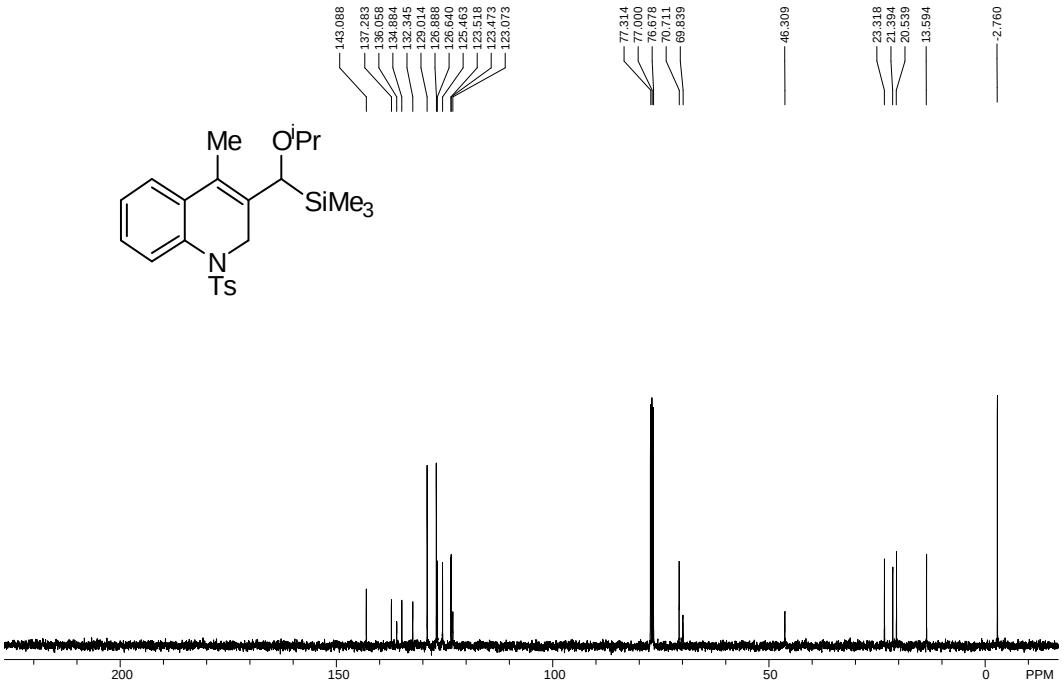
PPM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76.678
70.711
69.839
46.309
23.318
21.394
20.539
13.594
-2.760
Me
O^{i}Pr
SiMe$_3$
N
Ts
200
150
100
50
0
PPM