Supporting Information

Palladium-Catalyzed Carbonylative Coupling of Benzyl Chlorides with Terminal Alkynes to 1,4-Diaryl-3-butyn-2-ones and related Furanones

Xiao-Feng Wu,[a] Helfried Neumann,[a] and Matthias Beller[a]*

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General comments: All reactions were carried out under argon atmosphere. Toluene, triethylamine and THF were distilled from sodium ketyl or CaH_2 and stored in ©Aldrich Sure/Stor flasks under argon. Benzyl chlorides and alkynes were purchased from Aldrich or ABCR and used as received. Column chromatography was performed using Merck Silicagel 60 (0.043-0.06 mm). NMR data were recorded on a Bruker ARX 300 and Bruker ARX 400 spectrometers. ^{13}C and ^{1}H NMR spectra were referenced to signals of deutero solvents and residual protiated solvents, respectively. Gas chromatography analysis was performed on an Agilent HP-5890 instrument with a FID detector and HP-5 capillary column (polydimethylsiloxane with 5% phenyl groups, 30 m, 0.32 mm i.d., 0.25 μm film thickness) using argon as carrier gas. Gas chromatography-mass analysis was carried out on an Agilent HP-5890 instrument with an Agilent HP-5973 Mass Selective Detector (EI) and HP-5 capillary column (polydimethylsiloxane with 5% phenyl groups, 30 m, 0.25 mm i.d., 0.25 μm film thickness) using helium carrier gas.

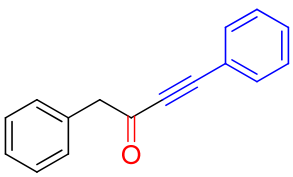
General procedure for the carbonylative Sonogashira coupling of benzyl chloride with phenyl acetylene: $PdCl_2(PPh_3)_2$ (2 mol%) was transferred into a vial (4 mL reaction volume) equipped with a septum, a small cannula and a stirring bar. After the vial was purged with argon, benzyl chloride (1 mmol), phenyl acetylene (1.2 mmol), toluene (2 mL), NEt_3 (2 mmol) and $P(OPh)_3$ (8 mol%) were injected into the vial by syringe. Then, the vial was placed in an alloy plate, which was transferred into a 300 mL autoclave of the 4560 series from Parr Instruments® under argon atmosphere. After flushing the autoclave three times with CO, a pressure of 10 bar was adjusted and the reaction was performed

for 20 hours at 100 °C. After the reaction, the autoclave was cooled down to room temperature and the pressure was released carefully. To the reaction mixture 6 mL water was added and the solution was extracted 3-5 times with 2-3 ml of ethyl acetate. The organic layer was separated and evaporated. After adsorption on silica gel, the crude product was purified by column chromatography using n-heptane and n-heptane/AcOEt (40:1) as eluent. The product was obtained in 176 mg (80% yields) as yellow oil.

General procedure for the carbonylative Sonogashira coupling of benzyl chloride with benzyl acetylene $PdCl_2(PPh_3)_2$ (2 mol%) was transferred into a vial (4 mL reaction volume) equipped with a septum, a small cannula and a stirring bar. After the vial was purged with argon, benzyl chloride (1 mmol), benzyl acetylene (1.2 mmol), toluene (2 mL), NEt_3 (2 mmol) and $P(OPh)_3$ (8 mol%) were injected into the vial by syringe. Then, the vial was placed in an alloy plate, which was transferred into a 300 mL autoclave of the 4560 series from Parr Instruments® under argon atmosphere. After flushing the autoclave three times with CO, a pressure of 10 bar was adjusted and the reaction was performed for 20 hours at 100 °C. After the reaction, the autoclave was cooled down to room temperature and the pressure was released carefully. To the reaction mixture 6 mL water was added and the solution was extracted 3-5 times with 2-3 ml of ethyl acetate. The organic layer was separated and evaporated. After adsorption on silica gel, the crude product was purified by column chromatography using n-heptane and n-heptane/AcOEt (40:1) as eluent. The product was obtained in 178 mg (68% yields) as white solid.

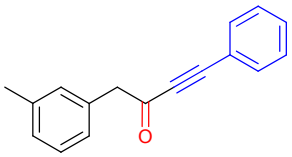
Analytic data:

1,4-Diphenylbut-3-yn-2-one



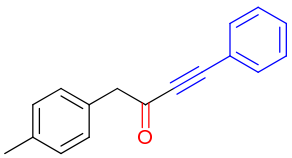
^{1}H NMR (300 MHz, $CDCl_3$): δ 7.16-7.37 (m, 10H), 3.81 (s, 2H).
^{13}C NMR (75 MHz, $CDCl_3$): δ 185.1, 132.9, 130.7, 129.8, 128.6, 128.5, 127.3, 121.3, 119.7, 92.8, 87.6, 52.1.
GC-MS (EI, 70eV): m/z(%) = 220 (M^+, 10), 192 (20), 129 (100), 101 (10), 91 (10), 75 (10).
IR (ATR, cm^{-1}): 3061 (vw), 3029 (vw), 2200 (s), 1752 (w), 1662 (vs), 1595 (m), 1489 (s), 1453 (m), 1443 (m), 1281 (w), 1186 (w), 1160 (w), 1092 (m), 1072 (vs), 1027 (w), 999 (w), 921 (w), 956 (vs), 687 (vs), 592 (m), 538 (vw), 509 (w), 472 (w).

4-Phenyl-1-*m*-tolylbut-3-yn-2-one



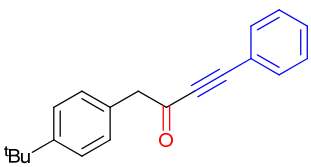
^{1}H NMR (300 MHz, $CDCl_3$): δ 7.19-7.57 (m, 9H), 3.98 (s, 2H), 2.45 (s, 3H).
^{13}C NMR (75 MHz, $CDCl_3$): δ 185.4, 138.4, 133.2, 130.9, 130.7, 129.4, 128.7, 128.2, 127.0, 121.6, 119.9, 92.8, 87.9, 52.2, 21.5.
GC-MS (EI, 70eV): m/z(%) = = 234 (M^+, 10), 219 (10), 206 (15), 191 (10), 129 (100), 105 (10), 75 (10).
IR (ATR, cm^{-1}): 3024 (vw), 2919 (vw), 2201 (s), 1753 (m), 1663 (vs), 1607 (w), 1591 (w), 1488 (s), 1443 (w), 1280 (w), 1192 (s), 1162 (w), 1119 (s), 1099 (m), 1073 (vs), 1025 (vw), 998 (w), 930 (w), 755 (vs), 712 (w), 687 (vs), 596 (m), 945 (w), 514 (w), 435 (m).

4-Phenyl-1-*p*-tolylbut-3-yn-2-one



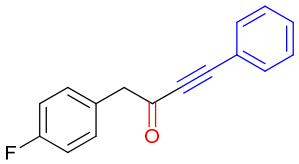
^{1}H NMR (300 MHz, $CDCl_3$): δ 7.13-7.36 (m, 7H), 7.06 (m, 2H), 3.78 (s, 2H), 2.22 (s, 3H).
^{13}C NMR (75 MHz, $CDCl_3$): δ 185.5, 137.0, 133.2, 130.9, 129.8, 129.5, 128.7, 121.6, 120.0, 92.7, 87.9, 51.9, 21.2.
GC-MS (EI, 70eV): m/z(%) = 234 (M^+, 10), 219 (5), 206 (25), 191 (10), 129 (100), 105 (15), 75 (10).
IR (ATR, cm^{-1}): 3024 (w), 2921 (w), 2201 (s), 1754 (m), 1663 (vs), 1594 (w), 1514 (m), 1489 (m), 1443 (w), 1329 (vw), 1280 (w), 1233 (vw), 1186 (m), 1161 (w), 1118 (s), 1071 (vs), 1022 (w), 998 (vw), 922 (w), 847 (vw), 806 (m), 756 (vs), 718 (w), 687 (vs), 589 (w), 535 (w), 498 (m).

1-(4-*tert*-Butylphenyl)-4-phenylbut-3-yn-2-one



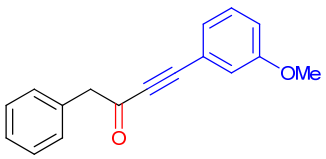
^{1}H NMR (300 MHz, $CDCl_3$): δ 7.30-7.53 (m, 9H), 3.98 (s, 2H), 1.41 (s, 9H).
^{13}C NMR (75 MHz, $CDCl_3$): δ 185.6, 150.3, 133.2, 129.6, 129.0, 128.6, 125.7, 121.6, 120.0, 93.0, 87.9, 51.7, 34.6, 31.4.
GC-MS (EI, 70eV): m/z(%) = 276 (M^+, 5), 261 (5), 233 (10), 219 (20), 192 (15), 147 (10), 129 (100), 117 (15).
IR (ATR, cm^{-1}): 3058 (vw), 2961 (m), 2904 (w), 2868 (w), 2201 (s), 1756 (w), 1665 (vs), 1596 (w), 1573 (vw), 1514 (w), 1489 (m), 1461 (vw), 1444 (w), 1414 (vw), 1364 (w), 1268 (m), 1190 (m), 1108 (m), 1071 (s), 1021 (w), 999 (vw), 922 (w), 836 (w), 812 (m), 757 (vs), 687 (vs), 629 (vw), 604 (vw), 556 (s), 535 (m), 422 (w).

1-(4-Fluorophenyl)-4-phenylbut-3-yn-2-one



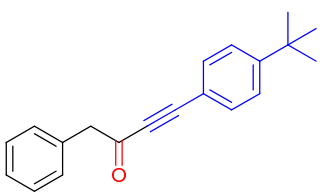
^{1}H NMR (300 MHz, $CDCl_3$): δ 7.46-7.55 (m, 3H), 7.39-7.44 (m, 2H), 7.29-7.36 (m, 2H), 7.07-7.15 (m, 2H), 3.96 (s, 2H).
^{13}C NMR (75 MHz, $CDCl_3$): δ 185.0, 162.5 (d, J = 245.5 Hz), 133.1, 131.5 (d, J = 7.5 Hz), 130.9, 128.7, 126.0, 121.5, 115.6 (d, J = 21.7 Hz), 93.2, 87.4, 51.4.
GC-MS (EI, 70eV): m/z(%) = 238 (M^+, 10), 207 (10), 129 (100), 109 (10).
IR (ATR, cm^{-1}): 3066 (w), 2200 (s), 1755 (w), 1662 (vs), 1600 (m), 1508 (vs), 1489 (m), 1443 (w), 1418 (vw), 1331 (vw), 1281 (w), 1221 (vs), 1189 (w), 1157 (m), 1103 (m), 1072 (vs), 1016 (vw), 998 (vw), 923 (vw), 857 (vw), 823 (m), 785 (s), 756 (vs), 719 (w), 687 (vs), 589 (w), 536 (m), 511 (s), 418 (m).

4-(3-Methoxyphenyl)-1-phenylbut-3-yn-2-one



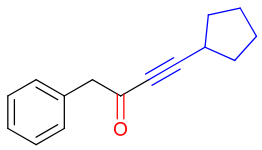
^{1}H NMR (300 MHz, $CDCl_3$): δ 7.27-7.47 (m, 6H), 7.09-7.13 (m, 1H), 6.99-7.07 (m, 2H), 3.99 (s, 2H), 3.84 (s, 3H).
^{13}C NMR (75 MHz, $CDCl_3$): δ 185.3, 159.4, 133.3, 129.9, 129.7, 128.8, 127.4, 125.6, 120.8, 117.7, 117.6, 92.9, 87.4, 55.4, 52.2.
GC-MS (EI, 70eV): m/z(%) = 250 (M^+, 15), 222 (10), 207 (10), 159 (100), 144 (10), 116 (10).
IR (ATR, cm^{-1}): 3029 (w), 2939 (w), 2836 (w), 2190 (vs), 1769 (w), 1661 (vs), 1595 (s), 1574 (s), 1488 (s), 1454 (m), 1421 (m), 1323 (m), 1287 (s), 1220 (vs), 1164 (s), 1091 (s), 1069 (s), 1035 (vs), 993 (w), 942 (vw), 856 (w), 783 (vs), 746 (s), 704 (vs), 682 (vs), 595 (s), 563 (w), 515 (m), 474 (m).

4-(4-*tert*-Butylphenyl)-1-phenylbut-3-yn-2-one



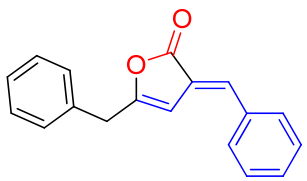
^{1}H NMR (300 MHz, $CDCl_3$): δ 7.21-7.30 (m, 9H), 3.83 (s, 2H), 1.21 (s, 9H).
^{13}C NMR (75 MHz, $CDCl_3$): δ 185.5, 154.7, 133.2, 130.0, 128.8, 127.4, 125.8, 121.5, 116.8, 93.7, 87.7, 52.2, 34.9, 31.1.
GC-MS (EI, 70eV): m/z(%) = 276 (M^+, 5), 220 (10), 185 (100), 170 (15), 155 (15), 91 (5).
IR (ATR, cm^{-1}): 3031 (vw), 2962 (m), 2904 (w), 2868 (w), 2199 (vs), 1755 (w), 1662 (vs), 1602 (m), 1494 (m), 1455 (w), 1406 (vw), 1364 (w), 1289 (w), 1267 (m), 1197 (w), 1161 (vw), 1117 (m), 1097 (m), 1067 (s), 1017 (w), 924 (vw), 835 (vs), 720 (m), 697 (vs), 614 (m), 565 (s), 515 (m), 496 (w).

4-Cyclopentyl-1-phenylbut-3-yn-2-one



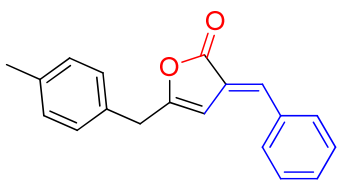
^{1}H NMR (300 MHz, $CDCl_3$): δ 7.29-7.48 (m, 5H), 3.87 (s, 2H), 2.72-2.85 (m, 1H), 1.89-2.03 (m, 2H), 1.58-1.79 (m, 6H).
^{13}C NMR (75 MHz, $CDCl_3$): δ 185.8, 133.5, 129.9, 128.7, 127.3, 100.9, 80.5, 52.3, 33.1, 31.9, 25.2.
GC-MS (EI, 70eV): m/z(%) = 212 (M^+, 5), 121 (100).
IR (ATR, cm^{-1}): 3030 (vw), 2959 (w), 2871 (w), 2204 (s), 1753 (s), 1666 (vs), 1593 (w), 1492 (m), 1453 (w), 1337 (vw), 1234 (s), 1192 (s), 1161 (m), 1120 (vs), 1073 (w), 1030 (w), 1003 (w), 928 (w), 894 (w), 816 (vw), 743 (m), 693 (vs), 594 (m), 496 (s), 474 (w).

(*E*)-5-Benzyl-3-benzylidenefuran-2(3*H*)-one



^{1}H NMR (300 MHz, $CDCl_3$): δ 7.61-7.67 (m, 2H), 7.14-7.31 (m, 8H), 6.85 (t, 1H, *J* = 1.6 Hz), 5.77 (s, 1H), 3.62 (bs, 2H).
^{13}C NMR (75 MHz, $CDCl_3$): δ 170.4, 147.4, 139.6, 137.1, 133.1, 132.5, 130.4, 128.9, 128.82, 128.78, 128.71, 126.9, 112.6, 31.6.
GC-MS (EI, 70eV): m/z(%) = 262 (M^+, 100), 217 (40), 202 (10), 115 (25), 90 (25).
IR (ATR, cm^{-1}): 3103 (w), 3026 (w), 1757 (vs), 1743 (vs), 1647 (m), 1602 (m), 1495 (m), 1450 (s), 1422 (w), 1357 (w), 1317 (vw), 1274 (w), 1234 (w), 1194 (vw), 1145 (m), 1075 (w), 1016 (vs), 999 (s), 935 (s), 918 (s), 893 (s), 862 (w), 849 (w), 823 (vw), 755 (s), 727 (w), 692 (vs), 636 (s), 615 (m), 592 (w), 524 (vw), 512 (m), 474 (m).

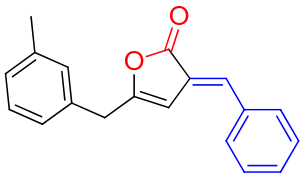
(*E*)-3-Benzylidene-5-(4-methylbenzyl)furan-2(*3H*)-one



^{1}H NMR (300 MHz, $CDCl_3$): δ 7.52 (d, 2H, *J* = 8.5 Hz), 7.11-7.28 (m, 5H), 7.02-7.09 (m, 2H), 6.78-6.83 (m, 1H), 5.72 (s, 1H), 3.60 (s, 2H), 2.24 (s, 3H).
^{13}C NMR (75 MHz, $CDCl_3$): δ 170.6, 146.9, 139.8, 139.3, 137.4, 131.9, 130.5, 129.6, 129.5, 129.0, 128.9, 127.0, 112.9, 31.7, 21.5.

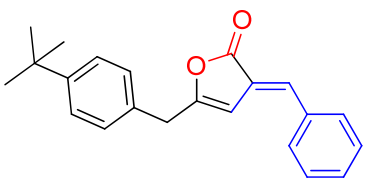
GC-MS (EI, 70eV): m/z(%) = 276 (M^+, 100), 231 (10), 215 (10), 132 (15), 115 (15), 91 (10), 78 (10).
IR (ATR, cm^{-1}): 3027 (w), 2929 (w), 1750 (vs), 1650 (m), 1596 (s), 1510 (vw), 1492 (m), 1453 (w), 1432 (w), 1351 (vw), 1318 (vw), 1291 (w), 1262 (m), 1182 (m), 1072 (w), 1037 (vs), 954 (vw), 935 (m), 824 (m), 894 (w), 858 (vw), 843 (s), 774 (s), 762 (w), 716 (vs), 646 (s), 637 (s), 577 (w), 559 (m), 514 (s), 463 (m).

(*E*)-3-Benzylidene-5-(3-methylbenzyl)furan-2(*3H*)-one



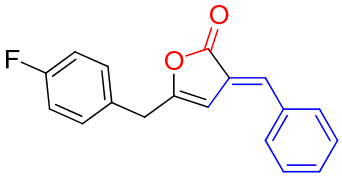
^{1}H NMR (300 MHz, $CDCl_3$): δ 7.40-7.48 (m, 2H), 7.09-7.30 (m, 6H), 6.97-7.05 (m, 1H), 6.81-6.84 (m, 1H), 5.72 (s, 1H), 3.61 (s, 2H), 2.26 (s, 3H).
^{13}C NMR (75 MHz, $CDCl_3$): δ 170.6, 147.4, 139.8, 138.5, 137.3, 133.1, 132.3, 131.0, 129.9, 129.0, 128.9, 128.7, 127.8, 127.0, 113.0, 31.7, 21.5.
GC-MS (EI, 70eV): m/z(%) = 276 (M^+, 100), 231 (15), 215 (15), 132 (10), 115 (15), 103 (10), 78 (10).
IR (ATR, cm^{-1}): 3031 (w), 1841 (vw), 1759 (vs), 1647 (m), 1606 (m), 1495 (m), 1453 (m), 1424 (w), 1345 (w), 1276 (m), 1214 (m), 1198 (w), 1098 (vw), 1075 (vw), 1019 (s), 947 (w), 919 (w), 896 (s), 874 (s), 846 (vw), 794 (s), 747 (s), 716 (vw), 693 (vs), 639 (m), 531 (w), 519 (w), 467 (w), 441 (m).

(*E*)-3-Benzylidene-5-(4-tert-butylbenzyl)furan-2(*3H*)-one



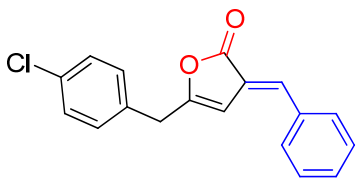
^{1}H NMR (300 MHz, $CDCl_3$): δ 7.55 (d, 2H, J = 8.3 Hz), 7.18-7.27 (m, 5H), 7.12-7.16 (m, 2H), 6.79 (t, 1H, J = 1.4 Hz), 5.71 (s, 1H), 3.56 (s, 2H), 1.18 (s, 9H).
^{13}C NMR (75 MHz, $CDCl_3$): δ 170.6, 152.4, 147.2, 139.8, 137.4, 132.1, 130.4, 129.0, 128.95, 128.90, 127.0, 125.9, 112.8, 34.9, 31.7, 31.3.
GC-MS (EI, 70eV): m/z(%) = 318 (M^+, 40), 303 (100), 131 (10), 115 (10), 91 (10).
IR (ATR, cm^{-1}): 3029 (vw), 2961 (m), 2903 (w), 2867 (w), 1841 (vw), 1762 (vs), 1697 (m), 1648 (w), 1602 (m), 1495 (m), 1454 (w), 1414 (vw), 1363 (w), 1268 (m), 1219 (w), 1108 (w), 1025 (s), 936 (m), 889 (m), 853 (s), 749 (s), 698 (vs), 558 (vs).

(*E*)-3-Benzylidene-5-(4-fluorobenzyl)furan-2(*3H*)-one



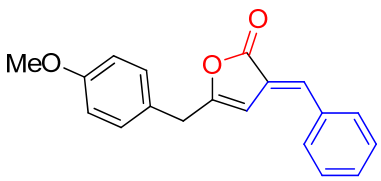
^{1}H NMR (300 MHz, $CDCl_3$): δ 7.71-7.83 (m, 2H), 7.27-7.46 (m, 5H), 7.05-7.17 (m, 2H), 6.97-7.03 (m, 1H), 5.88 (s, 1H), 3.77 (s, 2H).
^{13}C NMR (75 MHz, $CDCl_3$): δ 170.4, 162.8 (d, *J* = 252.8 Hz), 147.1, 139.6, 137.2, 132.5, 132.3 (d, *J* = 8.4 Hz), 129.5 (d, *J* = 3.06 Hz), 128.97, 128.93, 127.1, 115.9 (d, *J* = 21.4 Hz), 111.4, 31.7.
GC-MS (EI, 70eV): m/z(%) = 280 (M^+, 100), 262 (15), 235 (30), 220 (10), 185 (10), 136 (10), 108 (20).
IR (ATR, cm^{-1}): 3108 (w), 3059 (w), 2905 (w), 1742 (vs), 1655 (w), 1595 (s), 1504 (vs), 1493 (s), 1453 (w), 1482 (vw), 1416 (vw), 1354 (vw), 1307 (vw), 1292 (w), 1262 (w), 1227 (vs), 1193 (w), 1159 (s), 1099 (vw), 1075 (vw), 1036 (vs), 999 (m), 934 (m), 893 (m), 860 (s), 852 (s), 810 (s), 810 (s), 769 (m), 746 (vs), 716 (s), 698 (s), 668 (s), 639 (w), 579 (vw), 559 (s), 522 (vs), 459 (m), 410 (w).

(*E*)-3-Benzylidene-5-(4-chlorobenzyl)furan-2(*3H*)-one



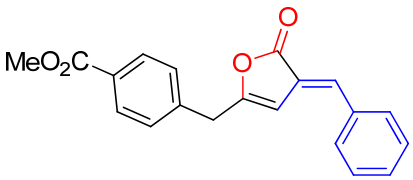
^{1}H NMR (300 MHz, $CDCl_3$): δ 7.57 (d, 2H, *J* = 8.4 Hz), 7.15-7.31 (m, 7H), 6.85 (t, 1H, *J* = 1.4 Hz), 5.72 (s, 1H), 3.64 (s, 2H).
^{13}C NMR (75 MHz, $CDCl_3$): δ 170.2, 147.7, 139.5, 137.1, 134.8, 132.9, 131.7, 131.6, 129.1, 128.96, 128.93, 127.1, 111.2, 31.8.
GC-MS (EI, 70eV): m/z(%) = 296 (M^+, 100), 243 (25), 215 (35), 202 (10), 152 (10), 115 (35), 89 (40).
IR (ATR, cm^{-1}): 3030 (w), 2929 (w), 1761 (vs), 1649 (w), 1599 (w), 1583 (w), 1490 (s), 1453 (m), 1433 (w), 1408 (w), 1336 (vw), 1296 (vw), 1280 (vw), 1258 (vw), 1179 (w), 1146 (vw), 1088 (m), 1028 (s), 1011 (m), 936 (w), 925 (w), 893 (w), 855 (m), 835 (vw), 812 (s), 774 (vw), 748 (vs), 719 (vw), 695 (vs), 648 (m), 638 (m), 576 (vw), 543 (m), 517 (s), 443 (w).

(*E*)-3-Benzylidene-5-(4-methoxybenzyl)furan-2(*3H*)-one



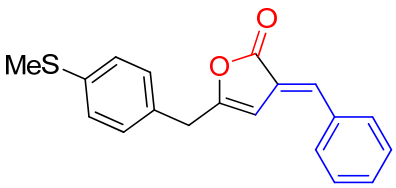
^{1}H NMR (300 MHz, $CDCl_3$): δ 7.58 (d, 2H, *J* = 8.7 Hz), 7.12-7.23 (m, 5H), 6.82 (t, 1H, *J* = 1.4 Hz), 6.77 (d, 2H, *J* = 8.9 Hz), 5.71 (s, 1H), 3.71 (s, 3H), 3.59 (s, 2H).
^{13}C NMR (75 MHz, $CDCl_3$): δ 170.7, 160.2, 139.8, 137.5, 134.9, 132.3, 130.5, 128.96, 128.92, 128.86, 127.0, 114.5, 112.8, 55.4, 31.7.
GC-MS (EI, 70eV): m/z(%) = 292 (M^+, 100), 148 (10), 133 (10), 91 (10).
IR (ATR, cm^{-1}): 3025 (w), 2967 (w), 2837 (w), 1733 (vs), 1592 (s), 1510 (s), 1492 (m), 1452 (w), 1438 (vw), 1426 (vw), 1363 (vw), 1299 (m), 1250 (vs), 1174 (s), 1074 (vw), 1053 (m), 1025 (vs), 966 (m), 934 (s), 896 (m), 868 (m), 849 (vw), 823 (s), 749 (s), 699 (s), 639 (m), 627 (m), 607 (w9, 588 (w), 557 (vw), 525 (s), 482 (w), 463 (w), 399 (vw).

(*E*)-Methyl 4-(4-benzylidene-5-oxo-4,5-dihydrofuran-2-yl)methylbenzoate



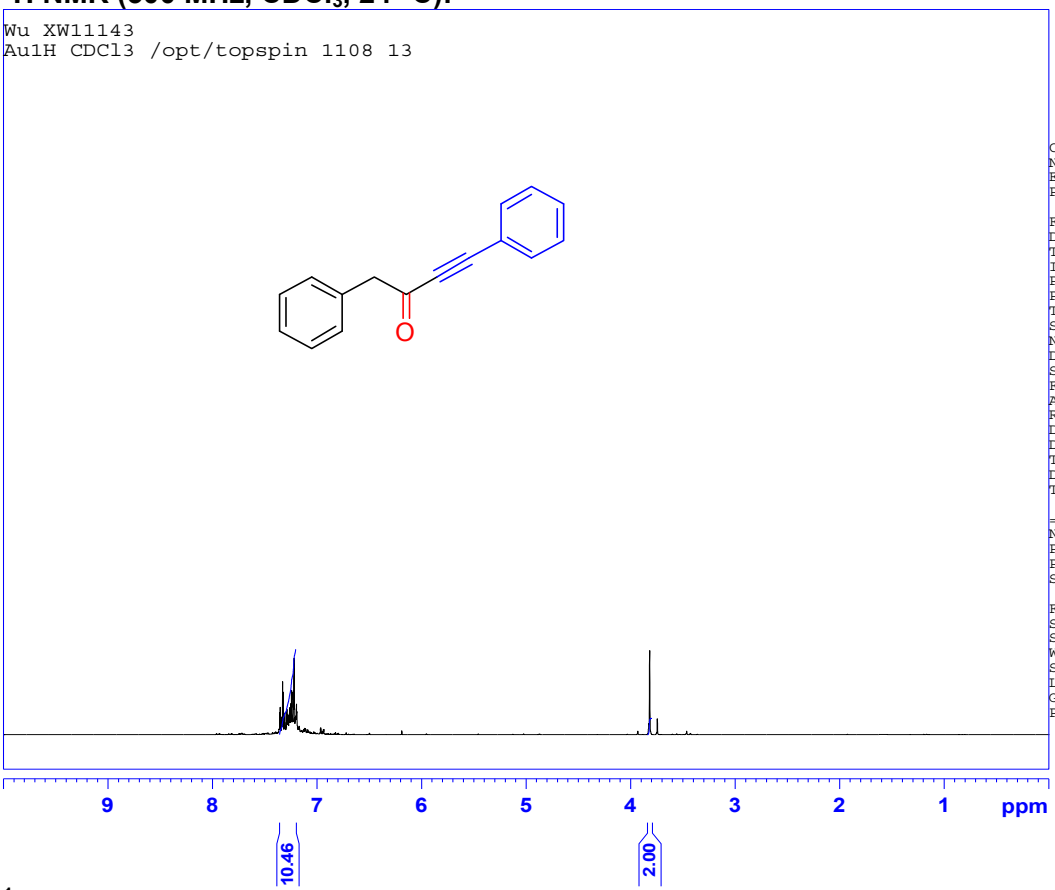
^{1}H NMR (300 MHz, $CDCl_3$): δ 7.56 (d, 2H, *J* = 8.6 Hz), 7.13-7.26 (m, 5H), 6.86 (t, 1H, *J* = 1.4 Hz), 6.75 (d, 2H, *J* = 8.9 Hz), 5.72 (s, 1H), 3.81 (s, 3H), 3.59 (s, 2H).
^{13}C NMR (75 MHz, $CDCl_3$): δ 170.2, 168.5, 147.7, 139.5, 137.1, 134.8, 132.9, 131.7, 131.6, 129.1, 128.96, 128.93, 127.1, 111.2, 53.5, 31.8.
GC-MS (EI, 70eV): m/z(%) = 320 (M^+, 15), 278 (100), 207 (10), 133 (10), 115 (10).

(*E*)-3-Benzylidene-5-(4-(methylthio)benzyl)furan-2(*3H*)-one



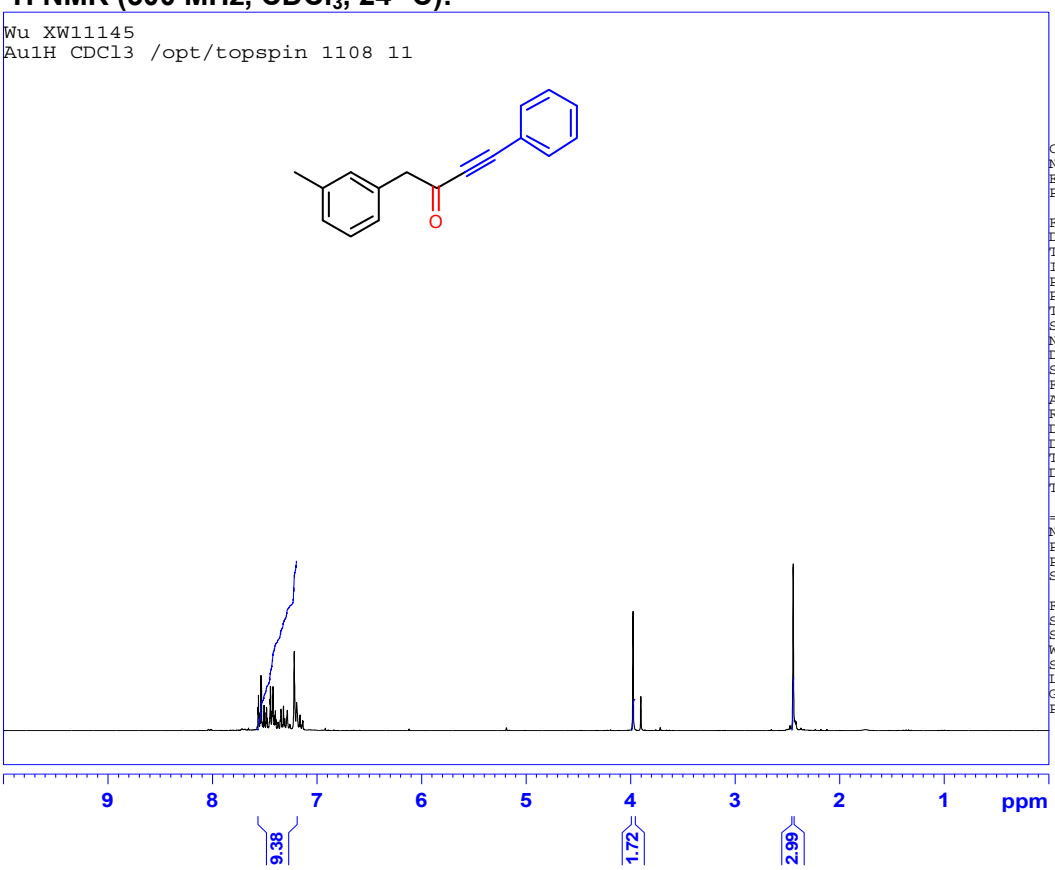
^{1}H NMR (300 MHz, $CDCl_3$): δ 7.57 (d, 2H, *J* = 8.7 Hz), 7.15-7.26 (m, 5H), 6.83 (t, 1H, *J* = 1.4 Hz), 6.78 (d, 2H, *J* = 8.9 Hz), 5.72 (s, 1H), 3.70 (s, 3H), 3.58 (s, 2H).
^{13}C NMR (75 MHz, $CDCl_3$): δ 170.7, 139.8, 137.5, 134.9, 133.5, 132.3, 130.5, 129.0, 128.9, 128.8, 127.0, 114.5, 112.8, 31.7, 15.6.
GC-MS (EI, 70eV): m/z(%) = 308 (M^+, 100), 281 (10), 207 (20), 164 (20), 135 (10), 121 (10).

^{1}H NMR (300 MHz, $CDCl_3$, 24 °C):



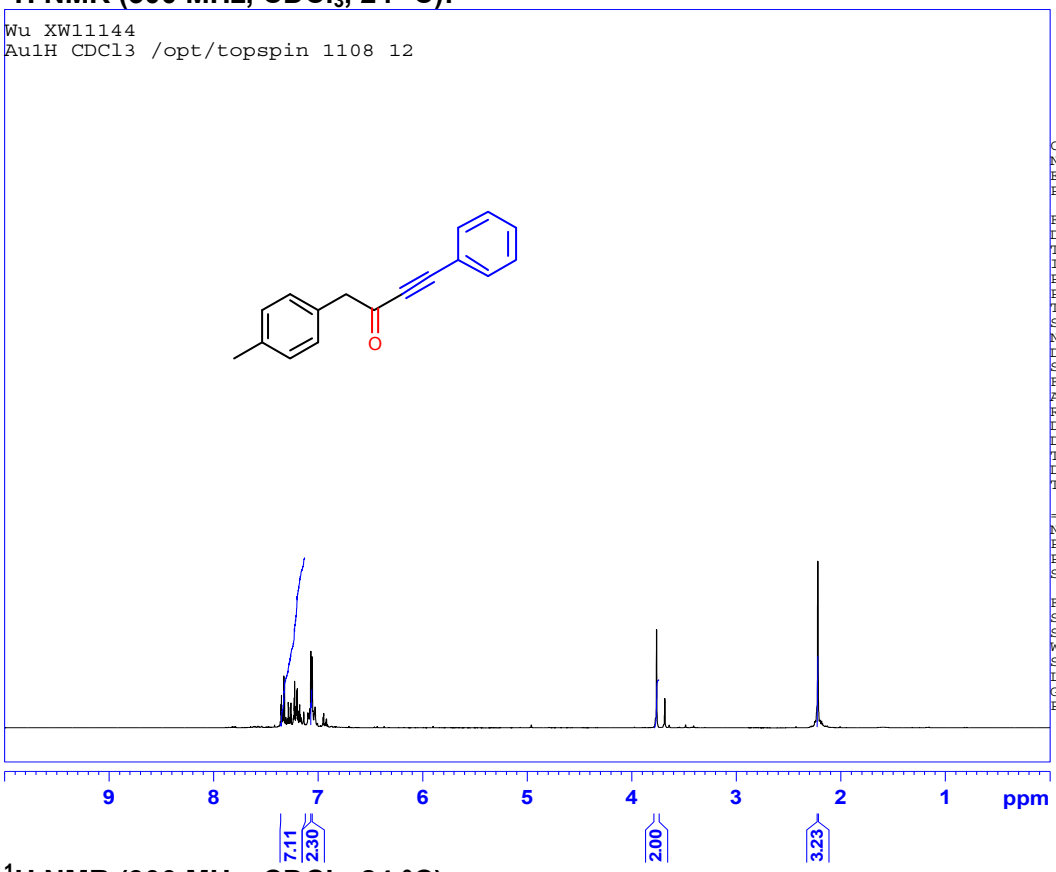

```
Current Data Parameters
NAME              xw11143
EXPNO                  10
PROCNO                  1

F2 - Acquisition Parameters
Date_            20110826
Time                13.47
INSTRUM           AV300
PROBHD   5 mm PABBO BB-
PULPROG              zg30
TD                  32768
SOLVENT             CDCl3
NS                     32
DS                      4
SWH              6172.839 Hz
FIDRES           0.188380 Hz
AQ               2.6542580 sec
RG                   80.6
DW                 81.000 usec
DE                  6.00 usec
TE                 297.1 K
D1            1.00000000 sec
TD0                     1

======== CHANNEL f1 ========
NUC1                   1H
P1                  11.00 usec
PL1                  0.00 dB
SFO1         300.1318534 MHz

F2 - Processing parameters
SI                  32768
SF           300.1300576 MHz
WDW                    EM
SSB                     0
LB                   0.10 Hz
GB                      0
PC                   1.00
```

^{1}H NMR (300 MHz, $CDCl_3$, 24 °C):



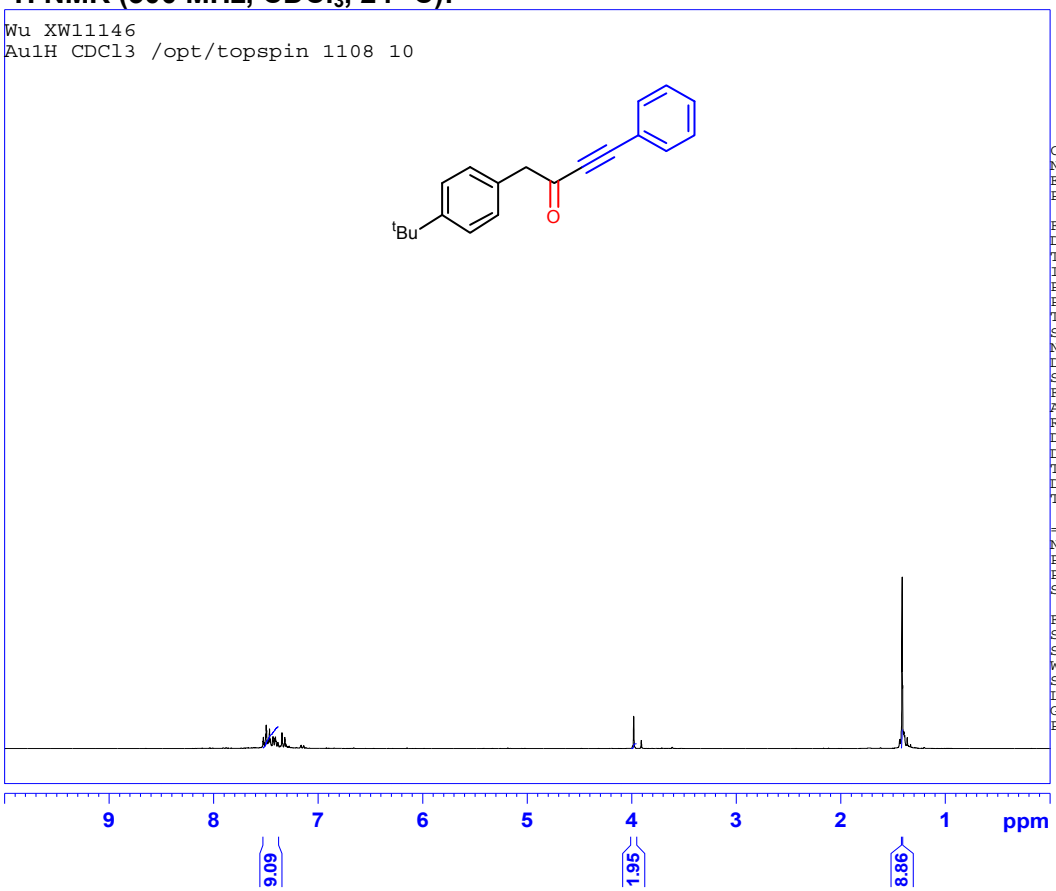

```
Current Data Parameters
NAME              xw11145
EXPNO                  10
PROCNO                  1

F2 - Acquisition Parameters
Date_            20110826
Time                12.44
INSTRUM           AV300
PROBHD   5 mm PABBO BB-
PULPROG              zg30
TD                  32768
SOLVENT             CDCl3
NS                     32
DS                      4
SWH              6172.839 Hz
FIDRES           0.188380 Hz
AQ               2.6542580 sec
RG                   71.8
DW                 81.000 usec
DE                  6.00 usec
TE                 297.0 K
D1            1.00000000 sec
TD0                     1

======== CHANNEL f1 ========
NUC1                   1H
P1                  11.00 usec
PL1                  0.00 dB
SFO1         300.1318534 MHz

F2 - Processing parameters
SI                  32768
SF           300.1300000 MHz
WDW                    EM
SSB                     0
LB                   0.10 Hz
GB                      0
PC                   1.00
```

^{1}H NMR (300 MHz, $CDCl_3$, 24 °C):



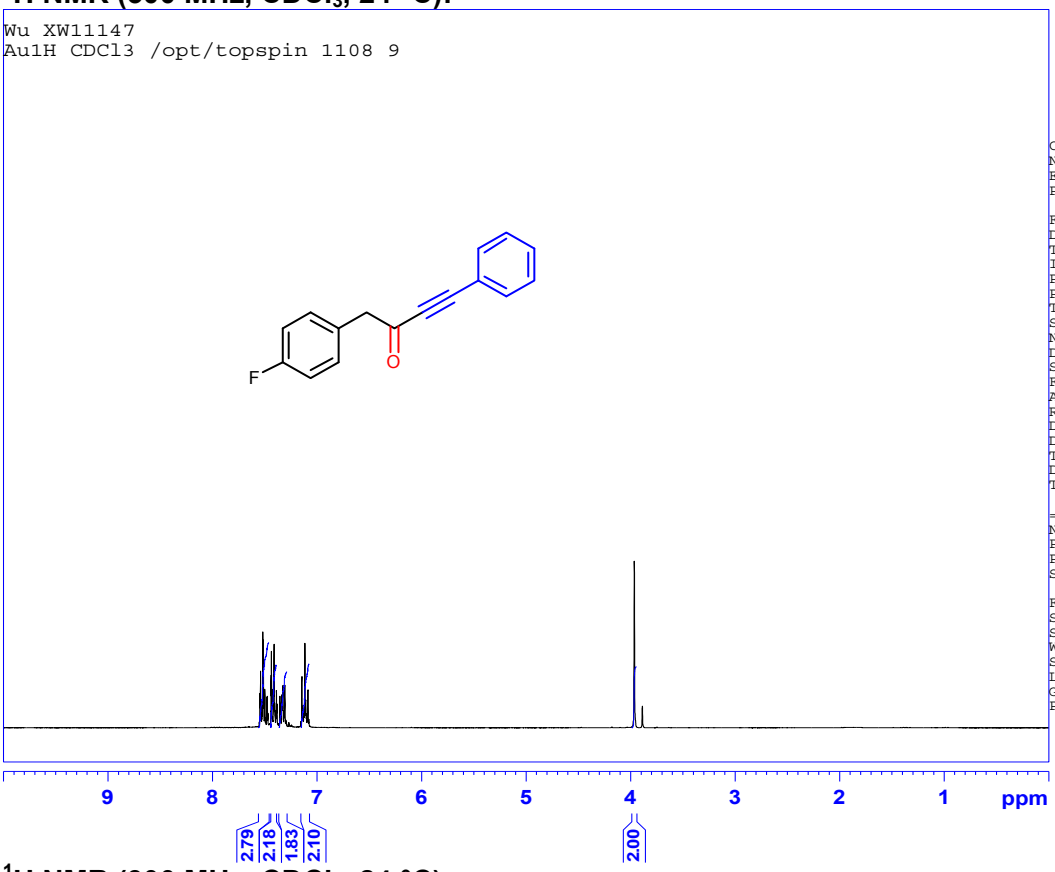

```
Current Data Parameters
NAME                 xw11144
EXPNO                     10
PROCNO                     1

F2 - Acquisition Parameters
Date_              20110826
Time                  13.16
INSTRUM              AV300
PROBHD    5 mm PABBO BB-
PULPROG                zg30
TD                    32768
SOLVENT               CDCl3
NS                       32
DS                        4
SWH                6172.839 Hz
FIDRES             0.188380 Hz
AQ                 2.6542580 sec
RG                      50.8
DW                   81.000 usec
DE                    6.00 usec
TE                   297.0 K
D1                 1.00000000 sec
TD0                        1

======== CHANNEL f1 ========
NUC1                      1H
P1                     11.00 usec
PL1                     0.00 dB
SFO1           300.1318534 MHz

F2 - Processing parameters
SI                        32768
SF             300.1300676 MHz
WDW                        EM
SSB                         0
LB                      0.10 Hz
GB                          0
PC                      1.00
```

^{1}H NMR (300 MHz, $CDCl_3$, 24 °C):



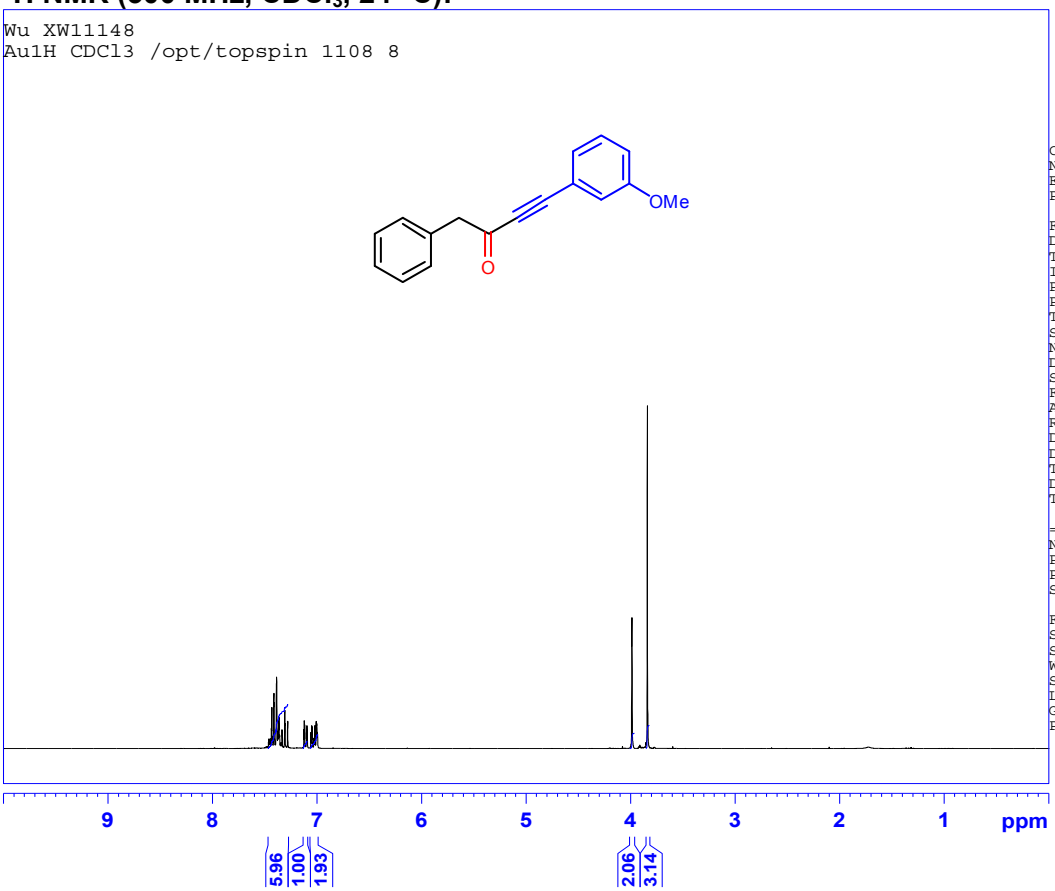

```
Current Data Parameters
NAME                 xw11146
EXPNO                     10
PROCNO                     1

F2 - Acquisition Parameters
Date_              20110826
Time                  12.14
INSTRUM              AV300
PROBHD    5 mm PABBO BB-
PULPROG                zg30
TD                    32768
SOLVENT               CDCl3
NS                       32
DS                        4
SWH                6172.839 Hz
FIDRES             0.188380 Hz
AQ                 2.6542580 sec
RG                      71.8
DW                   81.000 usec
DE                    6.00 usec
TE                   296.9 K
D1                 1.00000000 sec
TD0                        1

======== CHANNEL f1 ========
NUC1                      1H
P1                     11.00 usec
PL1                     0.00 dB
SFO1           300.1318534 MHz

F2 - Processing parameters
SI                        32768
SF             300.1300000 MHz
WDW                        EM
SSB                         0
LB                      0.10 Hz
GB                          0
PC                      1.00
```

^{1}H NMR (300 MHz, $CDCl_3$, 24 °C):



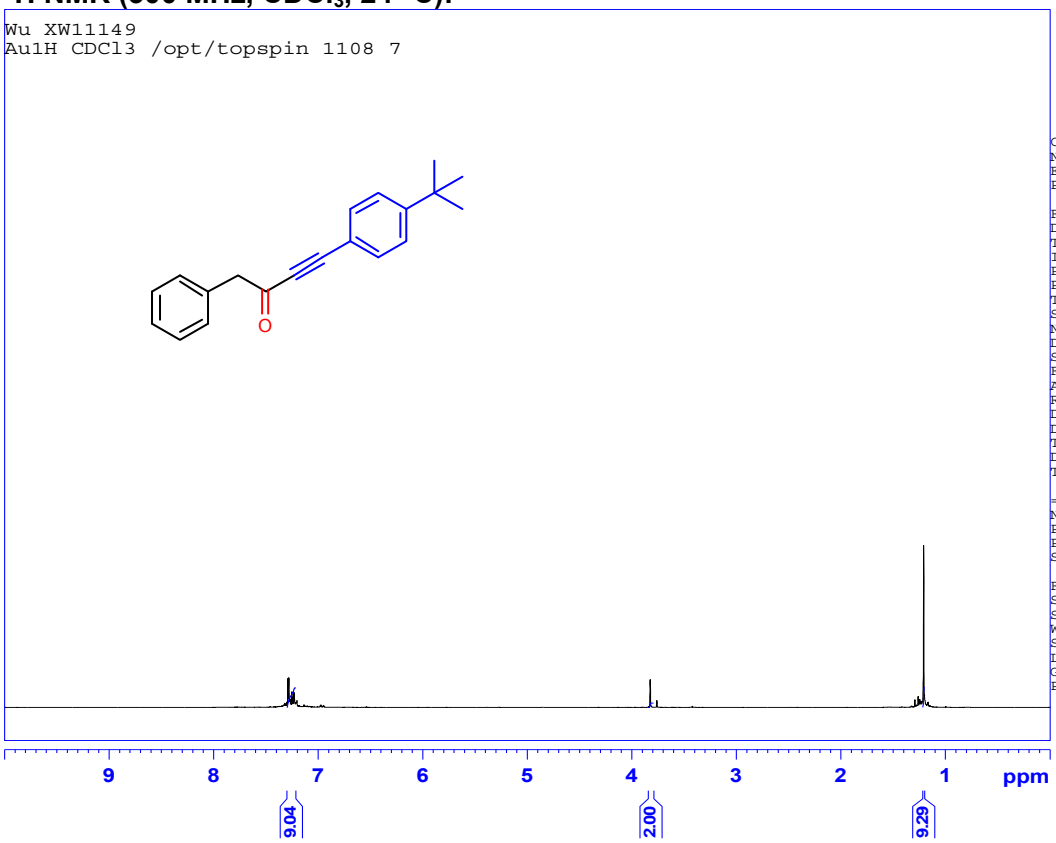

```
Current Data Parameters
NAME                  xw11147
EXPNO                      10
PROCNO                      1

F2 - Acquisition Parameters
Date_              20110826
Time                  11.42
INSTRUM                 AV300
PROBHD    5 mm PABBO BB-
PULPROG                 zg30
TD                     32768
SOLVENT                CDCl3
NS                        32
DS                         4
SWH                6172.839 Hz
FIDRES             0.188380 Hz
AQ                 2.6542580 sec
RG                     101.6
DW                    81.000 usec
DE                     6.00 usec
TE                    296.8 K
D1              1.00000000 sec
TD0                        1

======== CHANNEL f1 ========
NUC1                      1H
P1                     11.00 usec
PL1                     0.00 dB
SFO1           300.1318534 MHz

F2 - Processing parameters
SI                     32768
SF             300.1300000 MHz
WDW                       EM
SSB                        0
LB                      0.10 Hz
GB                         0
PC                      1.00
```

^{1}H NMR (300 MHz, $CDCl_3$, 24 °C):



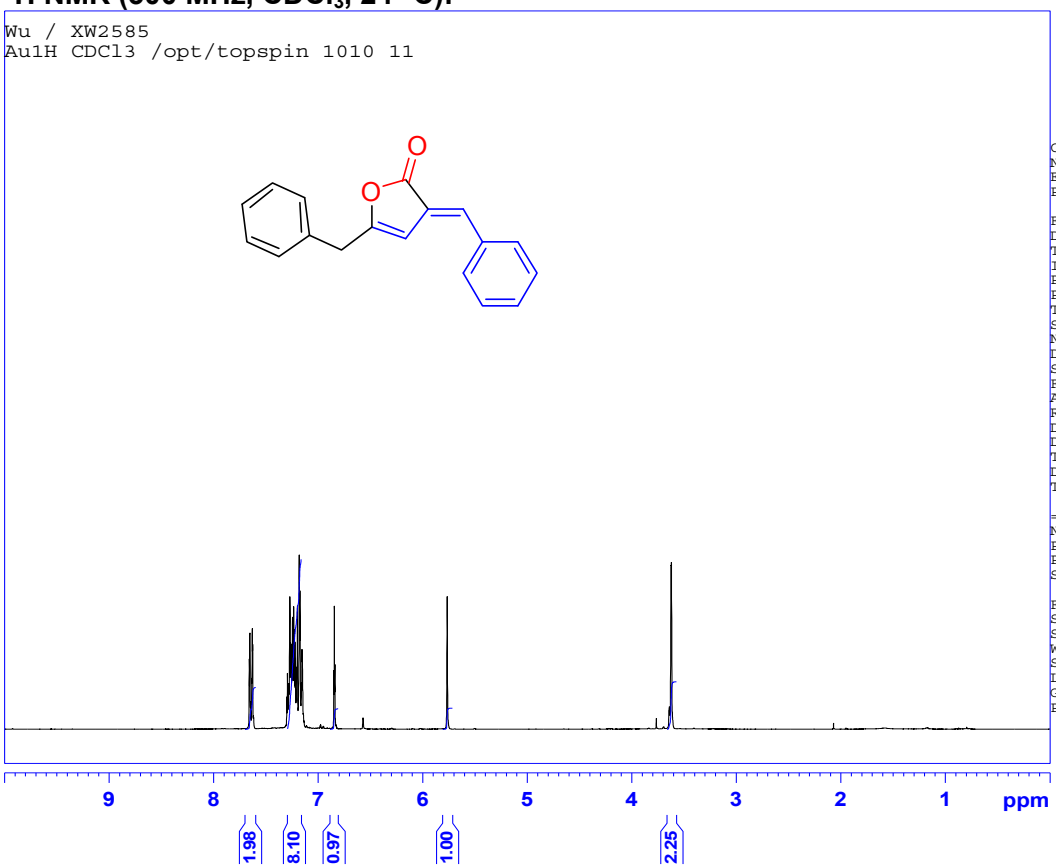

```
Current Data Parameters
NAME                  xw11148
EXPNO                      10
PROCNO                      1

F2 - Acquisition Parameters
Date_              20110826
Time                  10.51
INSTRUM                 AV300
PROBHD    5 mm PABBO BB-
PULPROG                 zg30
TD                     32768
SOLVENT                CDCl3
NS                        32
DS                         4
SWH                6172.839 Hz
FIDRES             0.188380 Hz
AQ                 2.6542580 sec
RG                       128
DW                    81.000 usec
DE                     6.00 usec
TE                    296.6 K
D1              1.00000000 sec
TD0                        1

======== CHANNEL f1 ========
NUC1                      1H
P1                     11.00 usec
PL1                     0.00 dB
SFO1           300.1318534 MHz

F2 - Processing parameters
SI                     32768
SF             300.1300000 MHz
WDW                       EM
SSB                        0
LB                      0.10 Hz
GB                         0
PC                      1.00
```

^{1}H NMR (300 MHz, $CDCl_3$, 24 °C):



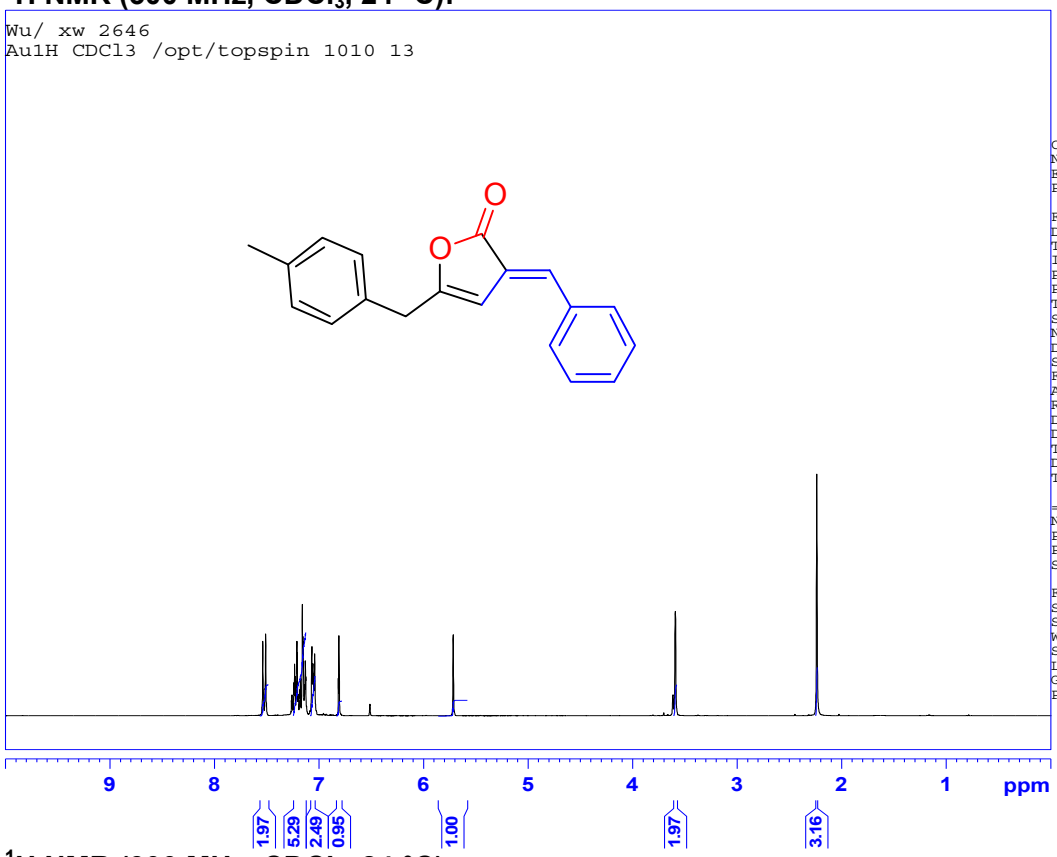

```
Current Data Parameters
NAME                  xw11149
EXPNO                      10
PROCNO                      1

F2 - Acquisition Parameters
Date_              20110826
Time                  10.20
INSTRUM               AV300
PROBHD    5 mm PABBO BB-
PULPROG                zg30
TD                    32768
SOLVENT               CDCl3
NS                       32
DS                        4
SWH                6172.839 Hz
FIDRES             0.188380 Hz
AQ                 2.6542580 sec
RG                      71.8
DW                    81.000 usec
DE                     6.00 usec
TE                    296.5 K
D1                 1.00000000 sec
TD0                         1

======== CHANNEL f1 ========
NUC1                       1H
P1                      11.00 usec
PL1                      0.00 dB
SFO1            300.1318534 MHz

F2 - Processing parameters
SI                        32768
SF              300.1300520 MHz
WDW                          EM
SSB                           0
LB                         0.10 Hz
GB                            0
PC                         1.00
```

^{1}H NMR (300 MHz, $CDCl_3$, 24 °C):




```
Current Data Parameters
NAME                  xw2585
EXPNO                      10
PROCNO                      1

F2 - Acquisition Parameters
Date_              20101019
Time                  16.27
INSTRUM               AV300
PROBHD    5 mm PABBO BB-
PULPROG                zg30
TD                    32768
SOLVENT               CDCl3
NS                       32
DS                        4
SWH                6172.839 Hz
FIDRES             0.188380 Hz
AQ                 2.6542580 sec
RG                     114
DW                    81.000 usec
DE                     6.00 usec
TE                    295.2 K
D1                 1.00000000 sec
TD0                         1

======== CHANNEL f1 ========
NUC1                       1H
P1                      11.00 usec
PL1                      0.00 dB
SFO1            300.1318534 MHz

F2 - Processing parameters
SI                        32768
SF              300.1300476 MHz
WDW                          EM
SSB                           0
LB                         0.10 Hz
GB                            0
PC                         1.00
```

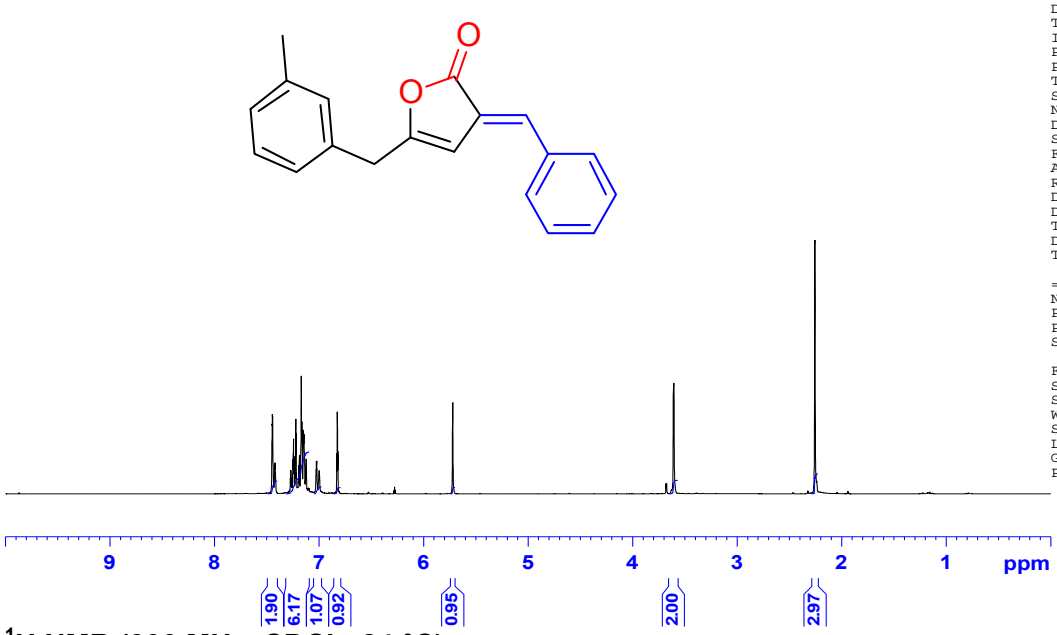
^{1}H NMR (300 MHz, $CDCl_3$, 24 °C):




```
Current Data Parameters
NAME                 xw2646
EXPNO                    10
PROCNO                    1

F2 - Acquisition Parameters
Date_              20101022
Time                 16.43
INSTRUM              AV300
PROBHD    5 mm PABBO BB-
PULPROG                zg30
TD                    32768
SOLVENT               CDCl3
NS                       32
DS                        4
SWH               6172.839 Hz
FIDRES            0.188380 Hz
AQ                2.6542580 sec
RG                     80.6
DW                  81.000 usec
DE                   6.00 usec
TE                   295.2 K
D1              1.00000000 sec
TD0                       1

======== CHANNEL f1 ========
NUC1                     1H
P1                   11.00 usec
PL1                   0.00 dB
SFO1          300.1318534 MHz

F2 - Processing parameters
SI                    32768
SF            300.1300553 MHz
WDW                      EM
SSB                       0
LB                    0.10 Hz
GB                        0
PC                    1.00
```

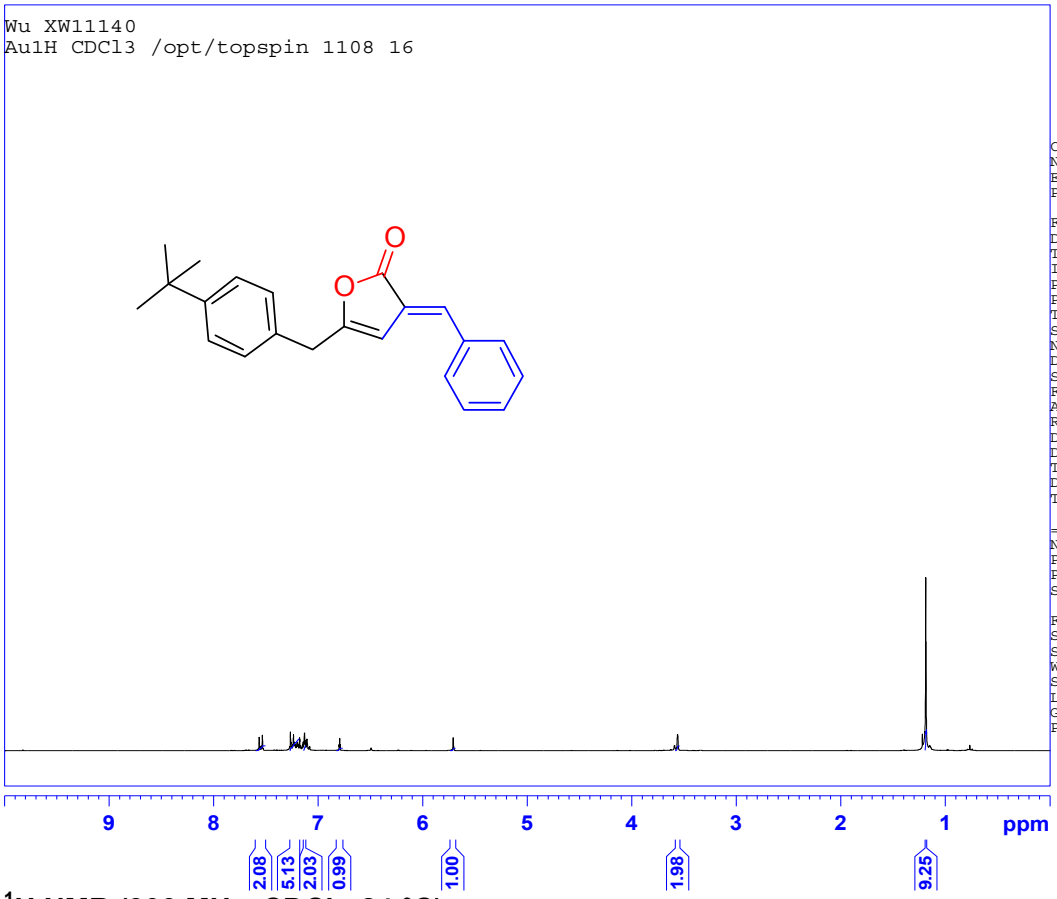
^{1}H NMR (300 MHz, $CDCl_3$, 24 °C):

Wu/ xw 2647
Au1H CDCl3 /opt/topspin 1010 14




```
Current Data Parameters
NAME                 xw2647
EXPNO                    10
PROCNO                    1

F2 - Acquisition Parameters
Date_              20101022
Time                 17.15
INSTRUM              AV300
PROBHD    5 mm PABBO BB-
PULPROG                zg30
TD                    32768
SOLVENT               CDCl3
NS                       32
DS                        4
SWH               6172.839 Hz
FIDRES            0.188380 Hz
AQ                2.6542580 sec
RG                     90.5
DW                  81.000 usec
DE                   6.00 usec
TE                   295.2 K
D1              1.00000000 sec
TD0                       1

======== CHANNEL f1 ========
NUC1                     1H
P1                   11.00 usec
PL1                   0.00 dB
SFO1          300.1318534 MHz

F2 - Processing parameters
SI                    32768
SF            300.1300518 MHz
WDW                      EM
SSB                       0
LB                    0.10 Hz
GB                        0
PC                    1.00
```


^{1}H NMR (300 MHz, $CDCl_3$, 24 °C):



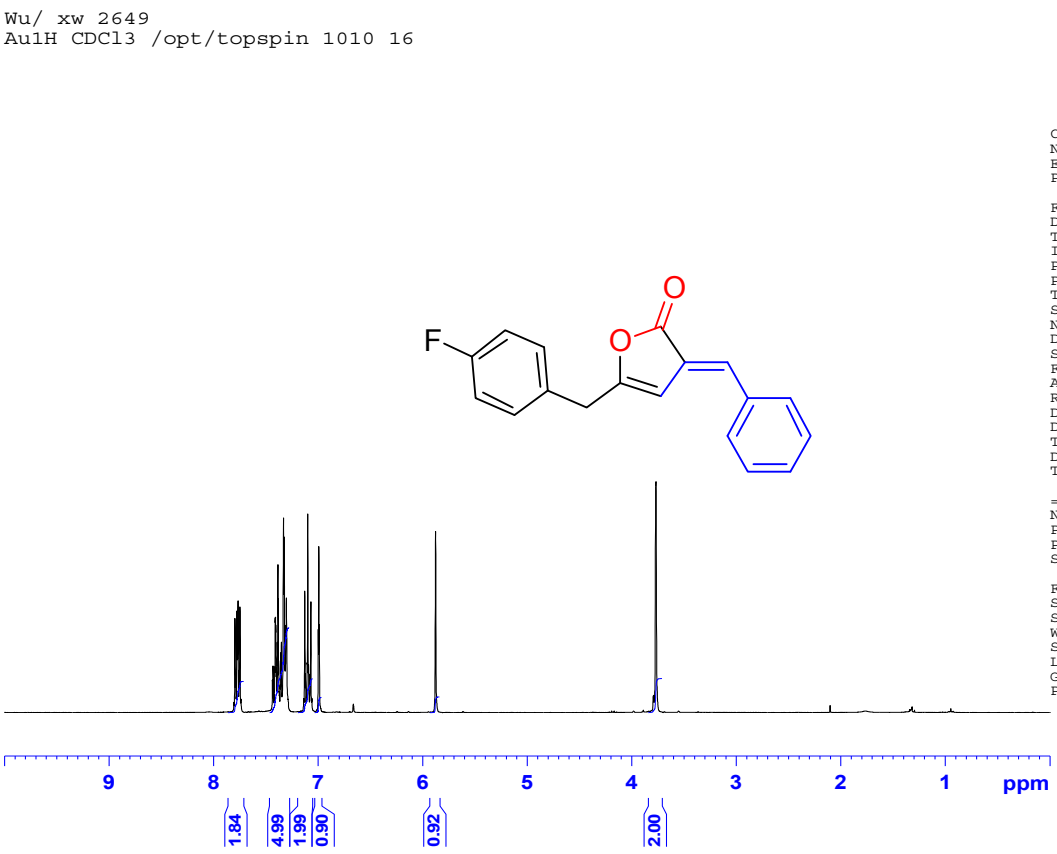
Current Data Parameters
NAME xw11140
EXPNO 10
PROCNO 1

F2 - Acquisition Parameters
Date_ 20110826
Time 15.21
INSTRUM AV300
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 32
DS 4
SWH 6172.839 Hz
FIDRES 0.188380 Hz
AQ 2.6542580 sec
RG 35.9
DW 81.000 usec
DE 6.00 usec
TE 297.1 K
D1 1.00000000 sec
TD0 1

======== CHANNEL f1 ========
NUC1 1H
P1 11.00 usec
PL1 0.00 dB
SFO1 300.1318534 MHz

F2 - Processing parameters
SI 32768
SF 300.1300689 MHz
WDW EM
SSB 0
LB 0.10 Hz
GB 0
PC 1.00

^{1}H NMR (300 MHz, $CDCl_3$, 24 °C):



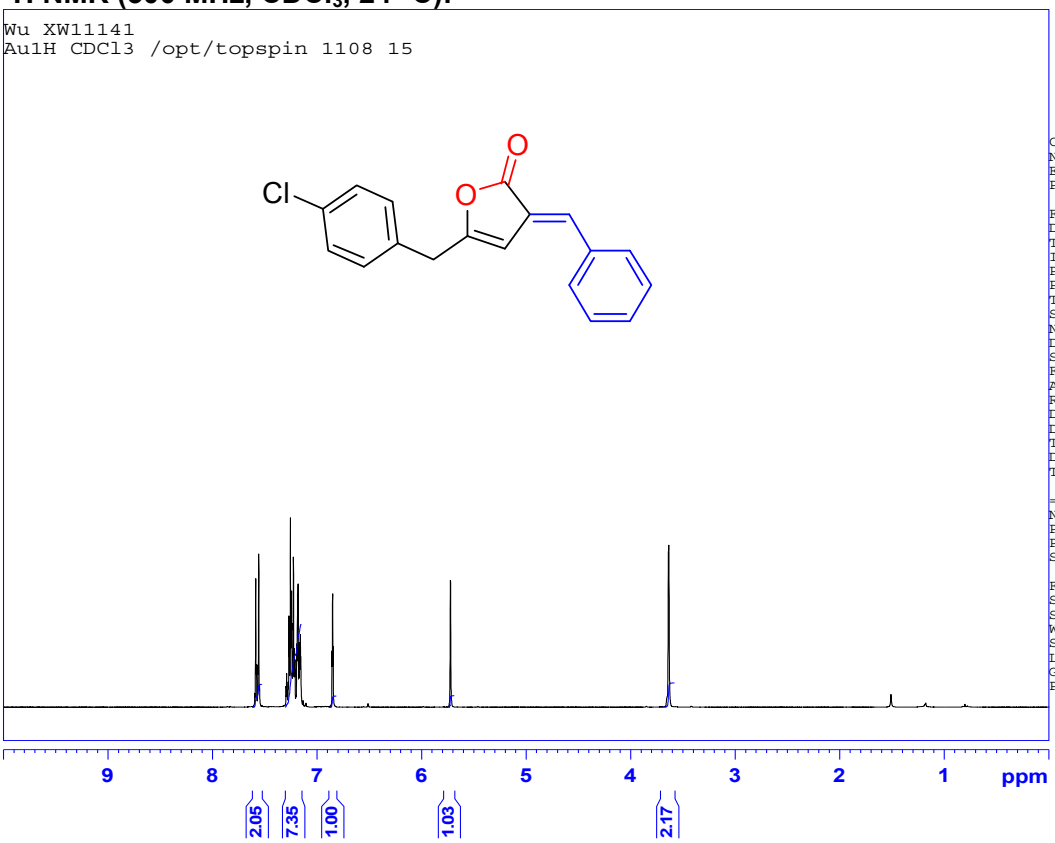
Current Data Parameters
NAME xw2649
EXPNO 10
PROCNO 1

F2 - Acquisition Parameters
Date_ 20101022
Time 18.15
INSTRUM AV300
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 32
DS 4
SWH 6172.839 Hz
FIDRES 0.188380 Hz
AQ 2.6542580 sec
RG 101.6
DW 81.000 usec
DE 6.00 usec
TE 295.1 K
D1 1.00000000 sec
TD0 1

======== CHANNEL f1 ========
NUC1 1H
P1 11.00 usec
PL1 0.00 dB
SFO1 300.1318534 MHz

F2 - Processing parameters
SI 32768
SF 300.1300000 MHz
WDW EM
SSB 0
LB 0.10 Hz
GB 0
PC 1.00

^{1}H NMR (300 MHz, $CDCl_3$, 24 °C):




```
Current Data Parameters
NAME                  xw11141
EXPNO                      10
PROCNO                      1

F2 - Acquisition Parameters
Date_              20110826
Time                  14.49
INSTRUM               AV300
PROBHD    5 mm PABBO BB-
PULPROG                zg30
TD                    32768
SOLVENT               CDCl3
NS                       32
DS                        4
SWH                6172.839 Hz
FIDRES             0.188380 Hz
AQ                 2.6542580 sec
RG                       181
DW                  81.000 usec
DE                   6.00 usec
TE                  297.1 K
D1              1.00000000 sec
TD0                       1

======== CHANNEL f1 ========
NUC1                     1H
P1                   11.00 usec
PL1                   0.00 dB
SFO1          300.1318534 MHz

F2 - Processing parameters
SI                    32768
SF             300.1300416 MHz
WDW                      EM
SSB                       0
LB                    0.10 Hz
GB                        0
PC                    1.00
```