Electronic Supplementary Material (ESI) for Organic Chemistry Frontiers.

Enantiodivergent Reduction of α-Keto Amides Catalyzed by High Valent, Chiral Oxido-Vanadium (V) Complexes

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Table of contents

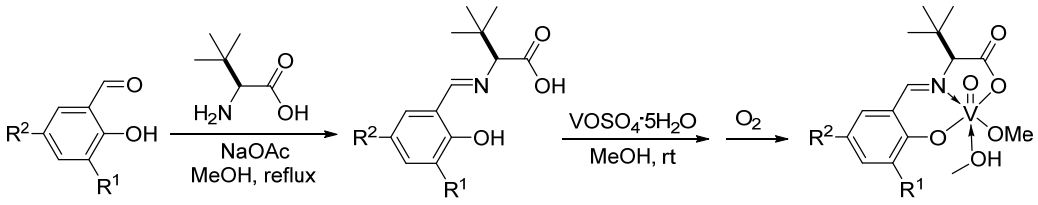
1. General materials and methods:

^{1}H NMR and ^{13}C NMR spectra were recorded on 400 MHz ^{1}H (100 MHz ^{13}C) spectrometers in deutero chloroform with chloroform or deutero methanol with methanol as an internal reference unless otherwise stated. Chemical shifts are reported in ppm (δ). Coupling constants, *J*, are reported in Hz. The abbreviations s, d, t, pent, quint, sext, dd, ddd, dt, and m stand for the resonance multiplicities singlet, doublet, triplet, pentet, quintet, sextet, doublet of doublets, doublet of doublet of doublets, doublet of triplets, and multiplet, respectively. Mass spectra were recorded with an ionization voltage of 70 or 20 eV unless otherwise stated. Elemental analyses were obtained by the Department of Chemistry, National Tsing Hua University, Taiwan or Department of Photonics, National Chiao Tung University, Taiwan. Fast atom bombardment (FAB) and electrospray ionization (ESI) mass spectra were recorded with data reported in the form m/e (intensity relative to base peak). Analytical TLC was performed on silica gel plates. Visualization was accomplished with UV light (254 nm) or with KMnO4 staining agents. Column (flash) chromatography was performed using 40-60 μm silica gel. Analytical high pressure liquid chromatography (HPLC) was performed with a built-in photometric detector (λ = 220 nm or 254 nm) using a Diacel AS-H, AD-H (0.46 cm × 25cm). Solvents for HPLC analyses were of spectroscopic grade and filtered before use. All enantiomeric excess determinations for optically enriched products were correlated with the corresponding racemic samples by HPLC analyses on chiral columns. Solvents for extraction and chromatography were reagent grade. Optical rotations are reported as follows: $[\alpha]^{T}_{D}$ (c = g/100mL, solvent). Toluene, Tetrahydrofuran (THF) and 1,2-Dimethoxyethane (DME) were dried over sodium benzophenone-ketyl intermediate under N_2 atmosphere and distilled before use. Dichloromethane (DCM) were dried over CaH_2. Chlorobenzene was distilled from anhydrous K_2CO_3. 4,4,5,5-Tetramethyl-1,3,2-dioxaborolane (Pinacolborane) and Catecholborane are brought from Acros Organics and are used as such without any further purification. Benzyl isocyanide was purchased from ACROS and used as received. $SiCl_4$ was purchased from Sigma Aldrich and used as received. All reaction products were isolated as chromatographically pure materials. All the other chemicals are bought from Sigma Aldrich or Acros Organics and are used as such without any purification. Catalysts were synthesized according our previously reported procedure.[1] Compound **3h-j**, **3n** are new compounds which were prepared according to method-b.[3]

List of Abbreviations:

HPLC - high performance liquid chromatography, FT-IR – Fourier transform infrared, HRMS (ESI^+) – High resolution mass spectra (electrospray ionization), ee - enantiomeric excess, THF-Tetrahydrofuran, TLC - Thin layer chromatography, DME - 1,2 Dimethoxyethane, DCM-Dichloromethane, TBME - *tert*-Butyl methyl ether, HB(pin) - Pinacolborane, HB(Cat) - Catecholborane, Ar – Argon,

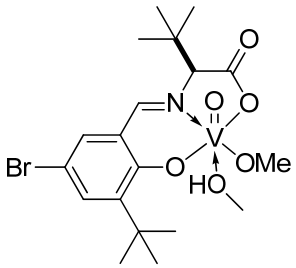
2. General Synthetic procedure and characterization data of oxo-vanadium(V) complexes 1a-1e.[1]



1a : R^1 = *t*-Bu R^2 = Br
1b : R^1 = *t*-Bu R^2 = NO_2
1c : R^1 = *t*-Bu R^2 = *t*-Bu
1d : R^1 = (2,5-Me)C_6H_4 R^2 = Br
1e : R^1 = (2,5-Me)C_6H_4 R^2 = NO_2

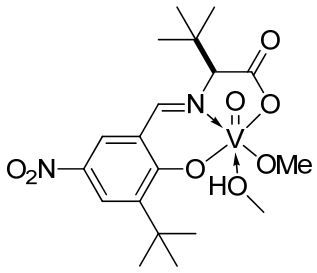
In a 50-mL, two-necked, round -bottomed flask was placed L-tert-leucine (5 mmol) and NaOAc-5·H_2O (1.17 g, 10 mmol) in degassed methanol (10 mL). After having been stirred at 60 °C for 10 min to affect their complete dissolution, the reaction mixture was treated dropwise with a solution of respective 2-hydroxy-benzaldehyde derivatives (5 mmol) in degassed methanol (12.5 mL). The reaction mixture became homogeneous by heating at 80 °C for 15 min and then gradually cooled to ambient temperature for 2 h. To the resultant Schiff base was added a solution of vanadyl (IV) sulfate trihydrate (1.08 g, 5 mmol) in degassed methanol (20 mL). Dark green complex started crashing out in 15 min. The reaction mixture was stirred for 2h and then concentrated to half of the original solvent volume. The crude vanadyl(IV) complex collected by filtration was washed sequentially with water (5 × 25 mL) and cold ether (5 × 25 mL) and then dried *in vacuo* to furnish pure vanadyl(IV) catalyst. The corresponding analytically pure oxo-vanadium (V) complexes were obtained by re-crystallization from oxygen-saturated MeOH and were used for asymmetric

reduction experiments.



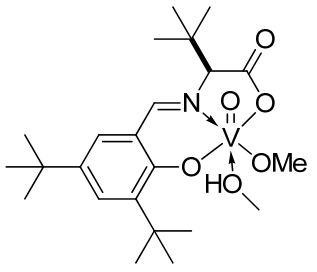
CH_3OH-M-OCH_3

Data for complex 1a: ^{1}H NMR (CD_3OD, 400 MHz) δ 8.54 (bs, 1H), 7.66 (d, *J* = 2.5 Hz, 1H), 7.63 (d, *J* = 2.5 Hz, 1H), 4.15 (s, 1H), 3.35 (s, OCH_3), 1.45 (s, 9H), 1.20 (s, 9H); **^{51}V NMR (CD_3OD, 105 MHz) δ** –567.3; **^{13}C NMR (CD_3OD, 100 MHz) δ** 167.7, 142.3, 137.0, 136.3, 135.1, 134.7, 123.8, 111.9, 84.7, 49.8, 38.3, 36.3, 29.9, 28.0; **IR (KBr)** 2965 (s), 2913 (m), 2869 (m), 1663 (s), 1615 (s, C=N), 1578 (m), 1548 (m, COO), 1480 (w), 1429 (m), 1368 (m), 1320 (m), 1297 (s), 1181 (m), 1055 (w), 1031 (w), 993 (m, V=O) ; M.W. (**M**: $C_{17}H_{22}BrNO_4V$) 434; **MS (ESI)** 450 (**M**OH-1^{+}, 100); $[\alpha]_D^{34}$ 306.53 (*c* 0.1, CH_3OH); **TLC R_f** 0.20 (MeOH/CH_2Cl_2, 1/9); Anal. Calcd. For [(H_2O)**M**OH]: C, 43.42; H, 5.36; N, 2.98. Found: C, 43.54; H, 5.13; N, 3.41.



CH_3OH-M-OCH_3

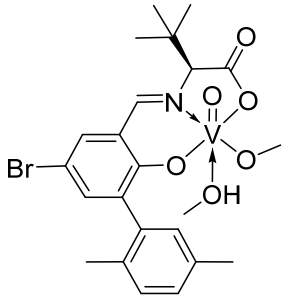
Data for complex 1b: ^{1}H NMR (CD_3OD, 400 MHz) δ 8.71 (bs, 1H), 8.54 (d, *J* = 2.6 Hz, 1H), 8.39 (d, *J* = 2.5 Hz, 1H), 4.24 (s, 1H), 3.31 (s, OCH_3),1.49 (s, 9H), 1.22 (s, 9H); ^{51}V NMR (CD_3OD, 105 MHz) δ –549.8, –568.8; **^{13}C NMR (CD_3OD, 100 MHz) δ** 168.0, 140.5, 139.1, 130.1, 130.0, 128.4, 127.6, 121.5, 84.9, 38.3, 36.5, 29.7, 28.0; **IR (KBr)** 2965 (w), 2916 (w), 2879 (w), 1627 (m, C=N), 1598 (m, COO), 1509 (w), 1326 (m), 1326 (w), 1225 (w), 1187 (w), 1113 (w), 1034 (w), 990 (w), 927 (w, V=O); M.W. (**M**: $C_{17}H_{22}N_2O_6V$) 401.3; **MS (ESI)** 850 (**M**$_2$O+H_2O^{+}, 90), 419 (**M**OH+H^{+}, 9), 417 (**M**OH-1^{+}, 100); $[\alpha]_D^{34}$ 83.93 (*c* 0.1, CH_3OH); **TLC R_f** 0.30 (MeOH/CH_2Cl_2, 1/4); Anal. Calcd. For [(H_2O)**M**OH]: C, 46.80; H, 5.78; N, 6.42. Found: C, 45.57; H, 5.83; N, 6.15.



CH_3OH-M-OCH_3

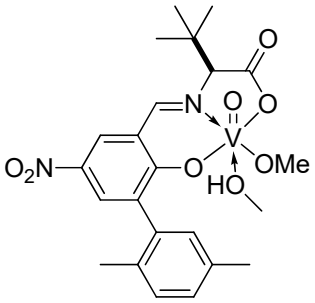
Data for 1c : ^{1}H NMR (CD_3OD, 400 MHz) δ 8.60 (bs, 1H), 7.68 (d, *J* = 2.3 Hz, 1H), 7.48 (d, *J* = 2.3 Hz, 1H), 4.14 (s, 1H), 3.35 (s, OCH_3), 1.47 (s, 9H), 1.35 (s, 9H), 1.20 (s, 9H); **^{51}V NMR (CD_3OD, 105 MHz) δ** –563.2; **^{13}C NMR (CD_3OD, 100 MHz) δ** 180.1, 168.9, 161.7, 143.5, 138.6, 132.4, 129.4, 121.9, 84.7, 38.3, 36.3, 35.3, 31.8, 30.3, 28.1; **IR (KBr)** 2958 (m), 2913 (w), 2868 (w), 1699 (m), 1612 (s, C=N), 1559

(w, COO), 1474 (w), 1459 (m), 1436 (w), 1418 (w), 1395 (w), 1363 (w), 1275 (w), 1260 (w), 1210 (w), 1183 (w), 1000 (w, V=O); M.W. (**M**: $C_{21}H_{31}NO_4V$) 412.4; MS (ESI) 881 (**M**$_2$O+H_2O+Na^+, 6), 460 ([H_2O**M**OCH_3] $^+$, 77), 444 ([**M**OCH_3+H]$^+$, 13), 413 (**M**+H^+, 100); **[α]$_D^{34}$** 262.87 (*c* 0.1, CH_3OH); **TLC R$_f$** 0.37 (MeOH/CH_2Cl_2, 1/8); Anal. Calcd. For [(H_2O)**M**OH]: C, 56.37; H, 7.66; N, 3.13. Found: C, 55.72; H, 7.32; N, 2.71.



CH_3OH-M-OCH_3

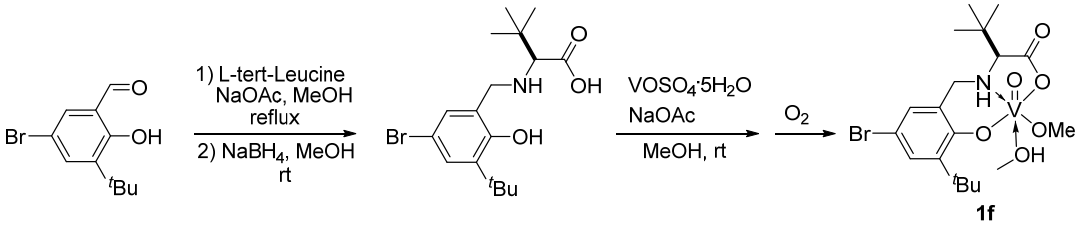
Data for 1d: ^{1}H NMR (CD_3OD, 400 MHz). δ 8.61 (s, 1H), 7.79 (s, 1H), 7.52 (s, 1H), 7.14 (d, *J* = 7.6 Hz, 1H), 7.07 (d, *J* = 7.6 Hz, 1H), 6.97 (s, 1H), 4.17 (s, 1H), 3.33 (s, OCH_3), 2.30 (s, 3H), 2.22 (s, 3H), 1.19 (s, 9H); **^{51}V NMR (CD_3OD, 105 MHz) δ** –559.3; **^{13}C NMR (CD_3OD, 100 MHz) δ** 167.3, 140.4, 137.6, 136.1, 135.9, 135.0, 131.8, 130.7, 129.6, 123.4, 111.4, 84.6, 38.3, 28.0, 21.0, 19.8; **IR (KBr)** 3446 (br, w, NH), 2961 (m), 1687 (m), 1616 (s), 1553 (s), 1432 (s), 1314 (s), 1002 (m, V=O); **[α]$_D^{25}$** 236.43 (*c* 0.1, CH_3OH); **TLC R$_f$** 0.28 (^{i}PrOH/Hexanes, 1/5); **HRMS (ESI)** $[M+H]^+$ Calcd for $C_{22}H_{26}BrNO_5V$: 514.0434, found: 514.0434.



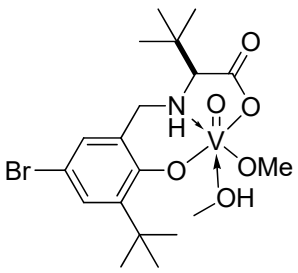
CH_3OH-M-OCH_3

Data for 1e: ^{1}H NMR (CD_3OD, 400 MHz) δ 8.79 (bs, 1H), 8.67 (d, *J* = 2.9 Hz, 1H), 8.28 (d, *J* = 2.8 Hz, 1H), 7.21 (d, *J* = 7.8 Hz, 1H), 7.15 (dd, *J* = 1.3,7.8 Hz, 1H), 7.05 (s, 1H), 3.35 (s, OCH_3), 4.25 (s, 1H), 3.35 (s, OCH_3), 2.36 (s, 3H, CH_3), 2.25 (s, 3H, CH_3), 1.23 (s, 9H); **^{51}V NMR (105 MHz, CD_3OD) δ –**559.6; **^{13}C NMR (100 MHz, CD_3OD) δ** 178.8, 167.8, 166.2, 140.6, 137.2, 136.3, 135.1, 134.3, 132.1, 131.8, 130.8, 130.1, 121.2, 85.0, 38.4, 28.1, 21.0, 19.8; **IR (KBr)** 3005(w), 2975 (w), 1628 (m, C=N), 1599 (m), 1509 (m), 1456 (w), 1434 (w), 1326 (s), 1276 (s), 1260 (m), 1100 (m), 999 (w, V=O) ; **[α]$_D^{34}$** 150.73 (*c* 0.1, CH_3OH); **TLC R$_f$** 0.25 (MeOH/CH_2Cl_2, 1/9). M.W. (**M**:$C_{21}H_{22}N_2O_6V$,449.4); **MS(ESI)** calcd. For $C_{22}H_{25}N_2O_7V$+1: 481.1101(**M**+**OCH_3+1**); found.481.1173 (**M**+**OCH_3**+**1**, 100);

3. Synthetic procedure and characterization data of oxo-vanadium(V) complex 1f.



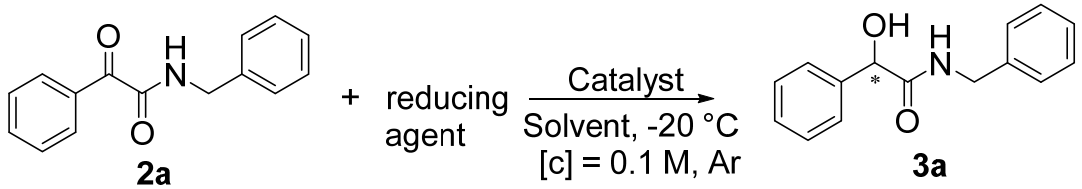
In a 50-mL, two-necked, round -bottomed flask was placed L-tert-leucine (5 mmol) and NaOAc-5·H_2O (643.5 mg, 5.5 mmol) in degassed methanol (10 mL). After having been stirred at 60 °C for 10 min to affect their complete dissolution, the reaction mixture was treated dropwise with a solution of 5-bromo-3-(*tert*-butyl)-2-hydroxybenzaldehyde (5 mmol) in degassed MeOH (12.5 mL). The reaction mixture refluxed at 80 °C for 20 hours and then gradually cooled to ambient temperature. To this mixture sodiumborohydride (5 mmol) was added. After 10 mins, under ice bath 1 N aqueous solution of HCl was added to make the pH = 2, white solid was precipitated, washed with distilled water and dried to give compound 5. A solution of vanadyl (IV) sulfate trihydrate (1.08 g, 5 mmol) and NaOAc-5·H_2O (643.5 mg, 5.5 mmol) in degassed methanol (5 mL) was added to compound 5. The reaction mixture was stirred for 4h and then concentrated to half of the original solvent volume. The crude vanadyl(IV) complex collected by filtration was washed sequentially with water (5 × 25 mL) and cold ether (5 × 25 mL) and then dried *in vacuo* to furnish pure vanadyl(IV) catalyst. The corresponding analytically pure oxo-vanadium (V) complexes were obtained by re-crystallization from oxygen-saturated MeOH to obtain dark brown crystals of **1f** in 47% yield.



CH_3OH-M-OCH_3

Data for complex **1f** : ^{1}H NMR (400 MHz, CD_3OD) δ 7.40 (d, *J* = 2.2 Hz, 1H), 7.26 (d, *J* = 2.2 Hz,1H), 4.57 (bs, 1H), 3.95 (d, *J* =11.8 Hz, 1H); 3.73 (d, *J* =11.9 Hz, 1H), 3.35 (s, OCH_3), 3.28 (s, 1H), 1.48 (s, 9H), 1.21 (s, 9H); ^{13}C NMR (100 MHz, CD_3OD) δ 164.8, 140.4, 130.9, 130.4, 128.4, 126.5, 113.3, 76.0, 53.6, 49.8, 37.2, 36.2, 30.6, 27.2; ^{51}V NMR (105 MHz, CD_3OD) δ −512.0, −525.1; IR 3449(br, w, NH), 2958 (m), 2872 (m), 1654(s), 1463 (m), 1434 (s), 1409 (m), 1340 (m), 1267(s), 1247(s),1053(s), 970(m, V=O); $[\alpha]_D^{34}$ 562.00 (*c* 0.1, CH_3OH); TLC R_f 0.1 ($MeOH/CH_2Cl_2$, 1/10).

4. General reaction procedure for the asymmetric reduction of *N*-benzyl-α-keto-amides.

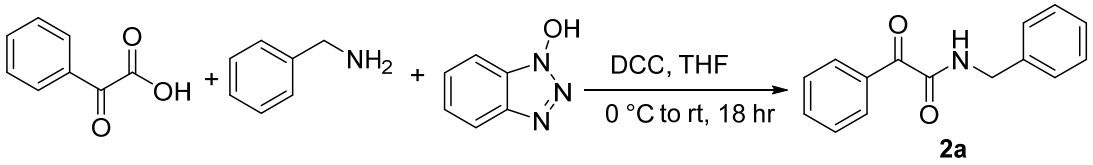


In a dried Schlenk tube was placed *N*-benzyl *α*-keto-amide (47.9 mg, 0.2 mmol) and vanadyl catalyst (9.4 mg, 0.02 mmol, 10 mol%) in anhydrous toluene (1.75 mL) under argon atmosphere. After having been stirred at ambient temperature for 30 min, the Schlenk tube was cooled to -20 ºC for 30 min. A solution of HBpin or HBCat (64 μL, 0.6 mmol, 3 equiv) in anhydrous toluene (186 μL) was cooled to -20 ºC and then added to the reaction mixture thru a gas-tight microsyringe. After having been complete as checked by TLC, the reaction was quenched by deionized water (2.5 mL) at -20 ºC (10 min) and then gradually warmed to ambient temperature for 30 min. The reaction mixture was extracted with EtOAc (10 mL × 3). The combined organic extracts were dried ($MgSO_4$, 1-1.2 g), filtered, and evaporated. The crude residue was purified by column chromatography (eluent: ethyl acetate in hexane) on silica gel to provide *N*-benzyl *α*-hydroxy-amide (46.9 mg, 98% yield).

5. General synthetic procedure for starting materials:

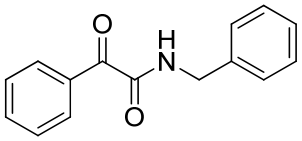
5.1 Method (a)[2]

N-Benzyl-2-oxo-2-phenylacetamide (2a) [1]



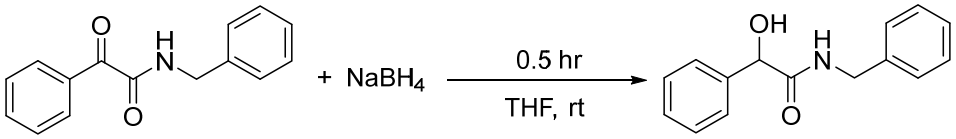
2a

In a 250 mL, two-necked, round bottomed flask was placed phenylglyoxylic (1.5 g, 10.0 mmol, 1.0 equiv), benzylamine (1.1 mL, 10.0 mmol, 1.0 equiv) and *N*,*N*-dicyclohexylcarbodiimide (DCC, 2.1 g, 10.0 mmol, 1.0 equiv) in 100 mL anhydrous THF. To this reaction mixture was added hydroxybenzotriazole (HOBt, 1.4 g, 10.0 mmol, 1.0 equiv) and stirred at room temperature for 18 hours. The resulting DCU was filtered off with suction filter and the organic filtrate was dried over anhydrous $MgSO_4$ (3.0 g), and then evaporated to dryness. The crude product was recrystallized from hot diethyl ether to give **2a** (1.93 g, 80% yield).



Data for **2a** : ^{1}H NMR ($CDCl_3$, 400 MHz) δ 8.37 (d, J = 7.6 Hz, 2H), 7.63 (t, J = 7.6 Hz, 1H), 7.49 (t, J = 8.0 Hz, 2H), 7.39-7.32 (m, 5H), 4.58 (d, J = 6.0 Hz, 2H); ^{13}C NMR ($CDCl_3$, 100 MHz) δ 187.5, 161.6, 137.0, 134.2, 133.1, 131.0, 128.6, 127.7, 127.6, 43.2; TLC R_f 0.22 (EtOAc/Hexane, 1/5); HRMS (ESI) Calcd for $C_{15}H_{13}NO_2$: 239.0946, found : 239.0945.

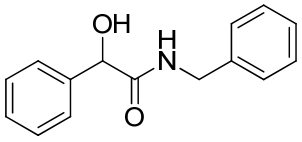
N-Benzyl-2-hydroxy-2-phenylacetamide (3a) [1]



2a **3a**

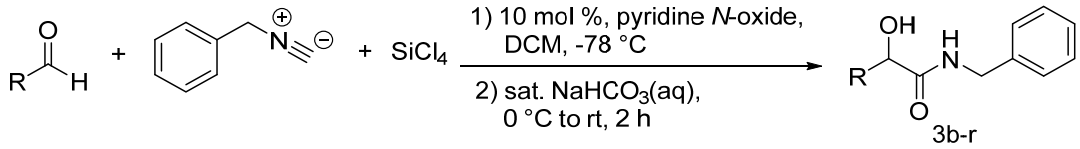
In a 25 mL, two-necked, round bottomed flask was placed compound **2a** (224 mg, 0.94 mmol, 1.0 eq.) in anhydrous THF (9.0 mL). Sodium borohydride (36 mg, 0.94 mmol) was then added and the reaction mixture was stirred at ambient temperature for 30 minutes. The reaction mixture was quenched with deionized water (8 mL) and then extracted with ethyl acetate (8 mL × 3). The

combined extracts were dried over anhydrous $MgSO_4$, filtered, evaporated. The crude product was purified by column chromatography (ethyl acetate/hexane) on silica gel.



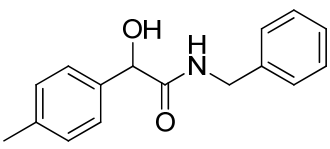
Data for 3a : **^{1}H NMR ($CDCl_3$, 400 MHz) δ** 7.39-7.27 (m, 8H), 7.18-7.16 (m, 2H), 6.50 (bs, 1H, NH), 5.05 (d, *J* = 3.2 Hz, 1H), 4.48-4.37 (m, 2H), 3.64 (d, *J* = 3.2 Hz, 1H, OH); **^{13}C NMR ($CDCl_3$, 100 MHz) δ** 172.3, 139.4, 137.4, 128.5, 128.3, 127.4, 126.6, 73.9, 43.1; **TLC R_f** 0.28 (EtOAc/hecane, 1/2); **HPLC conditions for 3a** : t_R 17.04 min (*R*-isomer), 27.90 min (*S*-isomer) (Chiralpak AD-H, hexane/*i*-PrOH, 92/8, 1.0 mL/min, λ = 254 nm); **HRMS (ESI)** Calcd for $C_{15}H_{15}NO_2$: 241.1103, found : 241.1103.

5.2 Method (b) (3b-r):[3]



In a 100 mL, three-necked. round bottomed flask was placed pyridine *N*-oxide (38 mg, 0.4 mmol, 0.1 equiv) in 12 ml of anhydrous CH_2Cl_2. To this reaction mixture, respective aldehyde (4.0 mmol, 1.0 equiv) was added. Benzyl isocyanide (585 µl, 4.8 mmol, 1.2 equiv) in 10 mL of CH_2Cl_2 was added dropwise through addition funnel at −78 °C, and then silicon tetrachloride ($SiCl_4$, 504 µl, 4.4 mmol, 1.1 equiv) was slowly added to the reaction. After the reaction was completed, a sat. aqueous solution of $NaHCO_3$ (30 mL) was added under ice bath and stirred for two hours. A white precipitate of SiO_2 was formed, and the mixture was filtered through Celite to remove white solid. The resulting filtrate was extracted with CH_2Cl_2 (30 mL × 3), dried ($MgSO_4$), and evaporated to give a crude product. The crude product was purified by column chromatography on silica gel with EtOAc/hexanes as eluents. The purified product was recrystallized from hot benzene (20 mL) to give *N*-benzyl-α-hydroxy-amide derivatives in 48–92% yields.

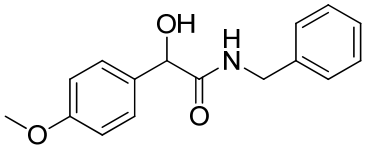
N-Benzyl-2-hydroxy-2-*p*-tolyl-acetamide (3b) [1]



Data for 3b : **^{1}H NMR ($CDCl_3$, 400 MHz) δ** 7.33-7.28 (m, 5H), 7.20-7.17 (m, 4H), 6.35 (bs, 1H, NH), 5.06 (d, *J* = 3.3 Hz, 1H), 4.52-4.39 (m, 2H), 3.43 (d, *J* = 3.4 Hz, 1H), 2.35 (s, 1H**); ^{13}C NMR**

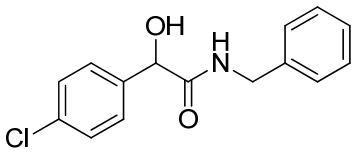
($CDCl_3$, 100 MHz) δ 172.6, 138.0, 137.6, 136.4, 129.2, 128.5, 127.4, 127.3, 126.5, 73.8, 43.0, 21.0; **TLC R_f** 0.2 (EtOAc/hexane, 1/4); **HPLC conditions for 3b** : t_R 16.35 min (*R*-isomer), 28.10 min (*S*-isomer) (Chiralpak AD-H, hexane/*i*-PrOH, 92/8, 1.0 mL/min, λ = 254 nm); **HRMS (ESI)** Calcd for $C_{16}H_{17}NO_2$: 255.1059, found : 255.1258.

N-Benzyl-2-hydroxy-2-(4'-methoxyphenyl)-acetamide (3c) [1]



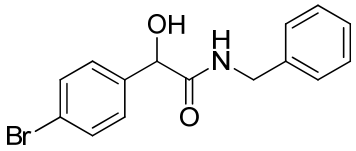
Data for 3c : ^{1}H NMR ($CDCl_3$, 400 MHz) δ 7.32-7.26 (m, 5H), 7.19 (d, *J* = 6.8 Hz, 2H), 6.88 (d, *J* = 8.6 Hz, 2H), 6.47 (bs, 1H, NH), 5.01 (d, *J* = 2.8 Hz, 1H), 4.49-4.37 (m, 2H), 4.38 (s, 1H), 3.54 (d, *J* = 3.2 Hz, 1H, OH); **^{13}C NMR ($CDCl_3$, 100 MHz) δ** 172.3, 159.8, 137.7, 131.5, 128.6, 128.1, 127.5, 114.2 73.7, 55.2, 43.4; **TLC R_f** 0.075 (EtOAc/hexane, 2/5); **HPLC conditions for 3c** : t_R 12.21 min (*R*-isomer), 21.55 min (*S*-isomer) (Chiralpak AD-H, hexane/*i*-PrOH, 85/15, 1.0 mL/min, λ = 254 nm); **HRMS (ESI)** Calcd for $C_{16}H_{17}NO_3$: 271.1208, found : 271.1211.

N-Benzyl-2-hydroxy-2-(4'-chlorophenyl)-acetamide (3d) [1]



Data for 3d : ^{1}H NMR ($CDCl_3$, 400 MHz) δ 8.36 (d, *J* = 8.8 Hz, 2H), 7.46 (d, *J* = 8.5 Hz, 2H), 7.42 (bs, 1H, NH), 7.39-7.29 (m, 5H), 4.56 (d, *J* = 6.0 Hz, 2H); **^{13}C NMR ($CDCl_3$, 100 MHz) δ** 172.2, 137.9, 137.3, 133.9, 128.5, 128.4, 127.8, 127.4, 1327.3, 73.1, 43.0; **TLC R_f** 0.175 (EtOAc/hexane, 1/2); **HPLC conditions for 3d** : t_R 14.30 min (*R*-isomer), 29.31 min (*S*-isomer) (Chiralpak AD-H, hexane/*i*-PrOH, 92/8, 1.0 mL/min, λ = 254 nm); **HRMS (ESI)** Calcd for $C_{15}H_{14}ClNO_2$: 275.0713, found : 275.0716.

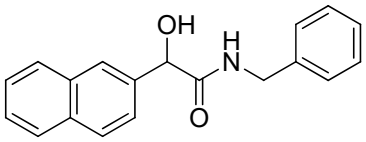
N-Benzyl-2-hydroxy-2-(4'-bromophenyl)-acetamide (3e) [1]



Data for 3e : ^{1}H NMR ($CDCl_3$, 400 MHz) δ 7.49 (d, *J* = 8.4 Hz, 2H), 7.36-7.26 (m, 5H), 7.18 (d, *J* = 7.2 Hz, 2H), 6.54 (bs, 1H, NH), 5.03 (d, *J* = 3.6 Hz, 1H), 4.47-4.37 (m, 2H), 3.59 (d, *J* = 3.6 Hz, 1H, OH); **^{13}C NMR ($CDCl_3$, 100 MHz) δ** 171.6, 138.3, 137.4, 131.7, 128.7, 128.3, 127.6, 127.5, 122.5, 73.4, 43.3; **TLC R_f** 0.07 (EtOAc/hexane, 1/4); **HPLC conditions for 3e** : t_R 16.71 min (*R*-isomer), 36.24 min (*S*-isomer) (Chiralpak AD-H, hexane/*i*-PrOH, 92/8, 1.0 mL/min, λ = 254 nm); **HRMS (ESI)** Calcd for $C_{15}H_{14}BrNO_2$: 319.0208, found :

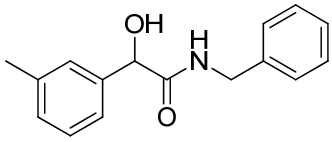
319.0207.

N-Benzyl-2-hydroxy-2-naphthanlen-2-yl-acetamide (3f) [1]



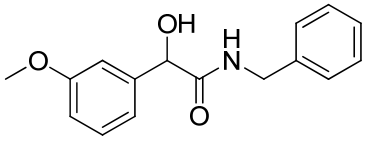
Data for 3f : ^{1}H NMR (DMSO-d6, 400 MHz) δ 8.63 (t, *J* = 6.0 Hz, 1H), 7.92-7.86 (m, 4H), 7.58 (d, *J* = 4.2 Hz, 1H), 7.63-7.47 (m, 2H), 7.27-7.18 (m, 5H), 6.38 (d, *J* = 3.6 Hz, 1H, NH), 5.14 (d, *J* = 3.5 Hz, 1H, OH), 4.34-4.24 (m, 2H); **^{13}C NMR (DMSO-d6, 100 MHz) δ** 172.2, 139.6, 138.9, 132.6, 132.5, 128,2, 127.5, 127.5, 126.7, 126.2, 125.9, 125.3, 124.8 73.7, 41.8; **TLC R_f** 0.05 (EtOAc/hexane, 1/3); **HPLC conditions for 3f** : t_R 10.39 min (*R*-isomer), 18.26 min (*S*-isomer) (Chiralpak AD-H, hexane/*i*-PrOH, 80/20, 1.0 mL/min, λ = 254 nm); **HRMS (ESI)** Calcd for $C_{19}H_{17}NO_2$: 291.1259, found : 291.1258.

N-Benzyl-2-hydroxy-2-*m*-tolyl-acetamide (3g)[4]



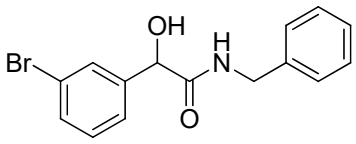
Data for 3g : ^{1}H NMR ($CDCl_3$, 400 MHz) δ 7.33-7.23 (m, 4H), 7.20-7.13 (m, 5H), 6.45 (bs, 1H, NH), 5.02 (d, *J* = 3.2 Hz, 1H), 4.50-4.38 (m, 2H), 3.59 (d, *J* = 3.4 Hz, 1H), 2.34 (s, 1H); **^{13}C NMR ($CDCl_3$, 100 MHz) δ** 172.2, 139.3, 138.5, 137.7, 129.3, 128.6, 127.5, 127.4, 123.8, 74.1, 43.3, 21.3; TLC R_f 0.15 (EtOAc/hexane, 1/2); **HPLC conditions for 3g** : t_R 13.50 min (*R*-isomer), 22.21 min (*S*-isomer) (Chiralpak AD-H, hexane/*i*-PrOH, 92/8, 1.0 mL/min, λ = 254 nm); **HRMS (ESI)** Calcd for $C_{16}H_{18}NO_2$: 256.1337 ($[M+H]^+$), found : 256.1333 ($[M+H]^+$).

N-Benzyl-2-hydroxy-2-(3'-methoxyphenyl)-acetamide (3h)



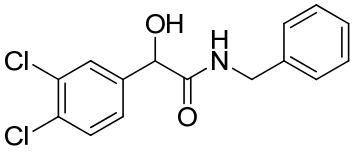
Data for 3h : ^{1}H NMR ($CDCl_3$, 400 MHz) δ 7.32-7.24 (m, 4H), 7.18 (d, *J* = 1.7 Hz, 2H), 7.16 (s, 1H), 6.97 (d, *J* = 7.6 Hz, 1H), 6.93 (t, *J* = 2.2 Hz, 1H), 6.88-6.85 (m, 1H), 6.51 (bs, 1H, NH), 5.01 (d, *J* = 2.8 Hz, 1H), 4.48-4.36 (m, 2H), 3.77 (s, 3H), 3.75 (d, *J* = 3.2 Hz, 1H, OH); **^{13}C NMR ($CDCl_3$, 100 MHz) δ** 172.1, 159.8, 140.9, 137.6, 129.7, 128.6, 127.4, 119.0, 114.3, 111.9, 73.9, 55.1, 43.3; **TLC R_f** 0.18 (EtOAc/hexane, 1/2); **HPLC conditions for 3h** : t_R 12.06 min (*R*-isomer), 16.55 min (*S*-isomer) (Chiralpak AD-H, hexane/*i*-PrOH, 85/15, 1.0 mL/min, λ = 254 nm); **HRMS (ESI)** Calcd for $C_{16}H_{18}NO_3$: 272.1286 ($[M+H]^+$), found : 272.1288 ($[M+H]^+$).

N-Benzyl-2-hydroxy-2-(3'-bromophenyl)-acetamide (3i)



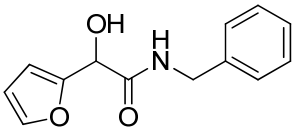
Data for 3i : ^{1}H NMR ($CDCl_3$, 400 MHz) δ 7.58 (s, 1H), 7.47 (td, *J* = 8.0 Hz, 0.8 Hz, 1H), 7.36-7.18 (m, 7H), 6.49 (bs, 1H, NH), 5.05 (d, *J* = 3.6 Hz, 1H), 4.49-4.40 (m, 2H), 3.51 (d, *J* = 3.7 Hz, 1H); **^{13}C NMR ($CDCl_3$, 100 MHz) δ** 171.4, 141.5, 137.4, 131.6, 130.2, 129.6, 128.6, 127.6, 127.5, 125.3, 122.7, 73.4, 43.4; **TLC R_f** 0.08 (EtOAc/hexane, 1/3); **HPLC conditions for 3i** : t_R 14.02 min (*R*-isomer), 21.79 min (*S*-isomer) (Chiralpak AD-H, hexane/*i*-PrOH, 92/8, 1.0 mL/min, λ = 254 nm); **HRMS (ESI)** Calcd for $C_{15}H_{15}BrNO_2$: 320.0286 ($[M+H]^+$), found : 320.0282 ($[M+H]^+$).

N-Benzyl-2-hydroxy-2-(3',4'-dichlorophenyl)-acetamide (3j)



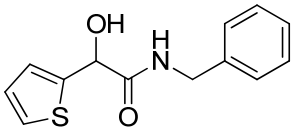
Data for 3j : ^{1}H NMR ($CDCl_3$, 400 MHz) δ 7.53 (d, *J* = 2.0 Hz, 1H), 7.43 (d, *J* = 8.2 Hz, 1H), 7.35-7.19 (m, 6H), 6.62 (bs, 1H, NH), 5.04 (d, *J* = 3.8 Hz, 1H), 4.47-4.36 (m, 2H), 3.57 (d, *J* = 4.0 Hz, 1H, OH); **^{13}C NMR ($CDCl_3$,100 MHz) δ** 171.1, 139.4, 137.3, 132.8, 132.5, 130.5, 128.7, 128.5, 127.7, 127.5, 125.9, 72.8, 43.4; **TLC R_f** 0.08 (hexane/EtOAc, 3/1); **HPLC conditions for 3j** : t_R 12.59 min (*R*-isomer), 24.86 min (*S*-isomer) (Chiralpak AD-H, hexane/*i*-PrOH, 92/8, 1.0 mL/min, λ = 254 nm); **HRMS (ESI)** Calcd for $C_{15}H_{14}Cl_2NO_2$: 310.0396 ($[M+H]^+$), found : 310.0401 ($[M+H]^+$).

N-Benzyl-2-hydroxy-2-furan-2-yl -acetamide (3k) [1]



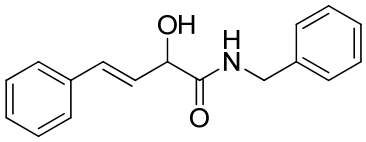
Data for 3k : ^{1}H NMR ($CDCl_3$, 400 MHz) δ 7.38 (d, *J* = 1.6 Hz, 1H), 7.34-7.22 (m, 5H), 6.70 (bs, 1H, NH), 6.38 (d, *J* = 3.2 Hz, 1H), 6.35 (dd, *J* = 1.8 Hz, 3.2 Hz, 1H), 5.14 (s, 1H), 4.53-4.43 (m, 2H), 3.84 (s, 1H); **^{13}C NMR ($CDCl_3$, 100MHz) δ** 169.9, 151.5, 142.9, 137.4, 128.7, 127.6, 127.5, 110.6, 108.7, 67.7, 43.5; **TLC R_f** 0.1 (EtOAc/hexane, 1/2); **HPLC conditions for 3k** : t_R 22.06 min (*R*-isomer), 28.14 min (*S*-isomer) (Chiralpak AD-H, hexane/*i*-PrOH, 92/8, 1.0 mL/min, λ = 254 nm); **HRMS (ESI)** Calcd for $C_{13}H_{14}NO_3$: 232.0973 ($[M+H]^+$), found : 232.0973 ($[M+H]^+$).

N-Benzyl-2-hydroxy-2-thiophen-2-yl -acetamide (3l) [1]



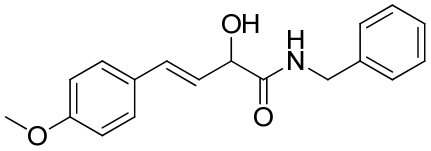
Data for 3l : ^{1}H NMR ($CDCl_3$, 400 MHz) δ 7.34-7.21 (m, 6H), 7.12 (d, *J* = 3.6 Hz, 1H), 6.98 (dd, *J* = 3.5 Hz, 5.0 Hz, 1H), 6.53 (bs, 1H, NH), 5.37 (d, *J* = 4.0 Hz, 1H), 4.53-4.43 (m, 2H), 3.66 (d, *J* = 4.4 Hz, 1H, OH); **^{13}C NMR ($CDCl_3$, 100 MHz) δ** 171.1, 142.3, 137.4, 128.6, 127.5, 126.8, 126.0, 126.0, 70.1, 43.5; TLC R_f 0.075 (EtOAc/hexane, 1/3); **HPLC conditions for 3l** : t_R 18.51 min (*R*-isomer), 27.42 min (*S*-isomer) (Chiralpak AD-H, hexane/*i*-PrOH, 92/8, 1.0 mL/min, λ = 254 nm); **HRMS (ESI)** Calcd for $C_{13}H_{13}NO_2S$: 247.0667, found : 247.0667.

2-Hydroxy-4-phenyl-but-3-enoic acid methylamide (3m) [1]



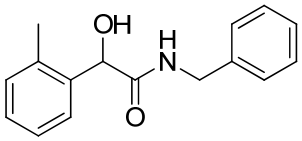
Data for 3m : ^{1}H NMR ($CDCl_3$, 400 MHz) δ 7.40-7.26 (m, 10H), 6.75 (d, *J* = 15.7 Hz, 1H), 6.54 (bs, 1H, NH), 6.31 (dd, *J* = 7.0 Hz, 15.8 Hz, 1H), 4.77 (d, *J* = 6.8 Hz, 1H), 4.55-4.44 (m, 2H), 3.19 (bs, 1H, NH); **^{13}C NMR ($CDCl_3$, 100 MHz) δ** 171.6, 137.6, 135.7, 133.5, 128.7, 128.6, 128.2, 127.7, 127.6, 1267, 126.6, 72.9, 43.6; TLC R_f 0.2 (EtOAc/hexane, 2/5); **HPLC conditions for 3m** : t_R 18.40 min (*R*-isomer), 26.94 min (*S*-isomer) (Chiralpak AD-H, hexane/*i*-PrOH, 92/8, 1.0 mL/min, λ = 254 nm); **HRMS (ESI)** Calcd for $C_{17}H_{17}NO_2$: 267.1259, found : 267.1260.

2-Hydroxy-4-(4-methoxy-phenyl)-but-3-enoic acid methylamide (3n)



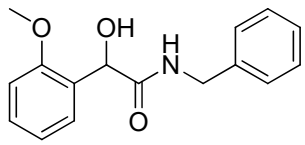
Data for 3n : ^{1}H NMR ($CDCl_3$, 400 MHz) δ 7.36-7.27 (m, 7H), 6.85 (d, *J* = 8.7 Hz, 1H), 6.69 (d, *J* = 15.8 Hz, 1H), 6.54 (bs, 1H, NH), 6.15 (dd, *J* = 7.3 Hz, 15.8 Hz, 1H), 4.73 (dd, *J* = 3.5 Hz, 7.2 Hz, 1H), 4.54-4.43 (m, 2H), 3.81 (s, 1H), 3.21 (d, *J* = 3.7 Hz, 1H); **^{13}C NMR ($CDCl_3$, 100 MHz) δ** 171.9, 159.6, 137.6, 133.3, 128.7, 128.5, 127.9, 127.7, 127.6, 124.3, 114.0, 73.1, 55.2, 43.5 ; **TLC R_f** 0.2 (EtOAc/hexane, 1/1); **HPLC conditions for 3n** : t_R 18.32 min (*R*-isomer), 23.28 min (*S*-isomer) (Chiralpak AD-H, hexane/*i*-PrOH, 88/12, 1.0 mL/min, λ = 254 nm); **HRMS (ESI)** Calcd for $C_{18}H_{20}NO_3$: 298.1443 ($[M+H]^+$), found : 298.1440 ($[M+H]^+$).

N-Benzyl-2-hydroxy-2-*o*-tolyl-acetamide (3o) [1]



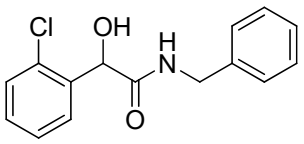
Data for 3o : ^{1}H NMR (CDCl$_3$, 400 MHz) δ 7.34-7.27 (m, 4H), 7.24-7.17 (m, 5H), 6.37 (bs, 1H, NH), 5.27 (d, *J* = 3.0 Hz, 1H), 4.53-4.41 (m, 2H), 3.40 (d, *J* = 3.0 Hz, 1H), 2.40 (s, 3H); **^{13}C NMR (CDCl$_3$, 100 MHz) δ** 172.4, 137.7, 137.2, 136.8, 131.1, 128.6, 127.7, 127.6, 127.5, 126.3, 72.2, 43.4, 19.2; **TLC R$_f$** 0.16 (EtOAc/hexane, 1/3); **HPLC conditions for 3o** : t_R 16.11 min (*R*-isomer), 23.57 min (*S*-isomer) (Chiralpak AD-H, hexane/*i*-PrOH, 92/8, 1.0 mL/min, λ = 254 nm); **HRMS (ESI)** Calcd for $C_{16}H_1NO_2$: 256.1337 ($[M+H]^+$), found : 256.1336 ($[M+H]^+$).

N-Benzyl-2-hydroxy-2-(2'-methoxyphenyl)-acetamide (3p) [1]



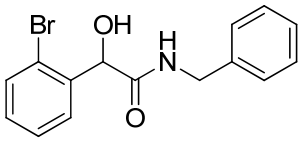
Data for 3p : ^{1}H NMR (CDCl$_3$, 400 MHz) δ 7.48 (dd, *J* = 1.6 Hz, 7.4 Hz, 1H), 7.32-7.25 (m, 4H), 7.14 (d, *J* = 7.1 Hz, 2H), 7.03 (td, *J* = 1.6 Hz, 7.5 Hz, 1H), 6.91 (d, *J* = 8.2 Hz, 1H), 6.90 (bs, 1H, NH), 5.44 (d, *J* = 5.2 Hz, 1H), 4.53-4.37 (m, 2H), 4.16 (d, *J* = 5.2 Hz, 1H, OH), 3.81 (s, 3H); **^{13}C NMR (CDCl$_3$, 100 MHz) δ**; 172.7, 155.7, 137.8, 129.2, 128.6, 128.2, 127.4, 127.2, 121.6, 110.8, 68.2, 55.6, 43.6; **TLC R$_f$** 0.25 (EtOAc/hexane, 1/2); **HPLC conditions for 3p** : t_R 19.98 min (*R*-isomer), 25.78 min (*S*-isomer) (Chiralpak AD-H, hexane/*i*-PrOH, 88/12, 1.0 mL/min, λ = 254 nm); **HRMS (ESI)** Calcd for $C_{16}H_{17}NO_3$: 271.1208, found : 271.1209.

N-Benzyl-2-hydroxy-2-(2'-chlorophenyl)-acetamide (3q) [1]



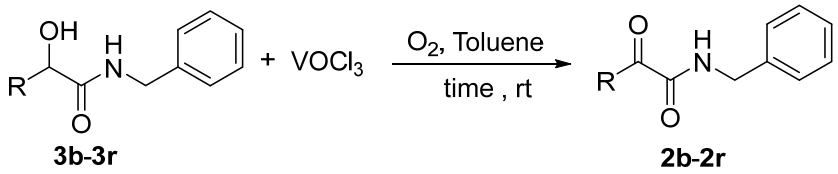
Data for 3q : ^{1}H NMR (CDCl$_3$, 400 MHz) δ 7.48 (dd, *J* = 1.7 Hz, 7.4 Hz, 1H), 7.38 (dd, *J* = 1.2 Hz, 7.3 Hz, 1H), 7.33-7.26 (m, 5H), 7.18-7.16 (m, 2H), 6.47 (bs, 1H, NH), 5.56 (d, *J* = 3.6 Hz, H), 4.56-4.40 (m , 2H), 4.03 (d, *J* = 3.8 Hz, 1H); **^{13}C NMR (CDCl$_3$, 100 MHz) δ** 171.5, 137.4, 137.2, 132.6, 129.7, 128.6, 127.5, 127.5, 127.4, 70.2, 43.7; **TLC R$_f$** 0.15 (EtOAc/hexane, 1/3); **HPLC conditions for 3q** : t_R 21.18 min (*R*-isomer), 27.33 min (*S*-isomer) (Chiralpak AD-H, hexane/*i*-PrOH, 92/8, 1.0 mL/min, λ = 254 nm); **HRMS (ESI)** Calcd for $C_{15}H_{15}ClNO_2$: 276.0791 ($[M+H]^+$), found : 276.0789 ($[M+H]^+$).

N-Benzyl-2-hydroxy-2-(2'-bromophenyl)-acetamide (3r) [4]



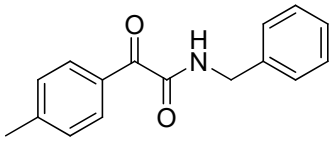
Data for 3r : ^{1}H NMR ($CDCl_3$, 400 MHz) δ 7.55 (dd, *J* = 0.8 Hz, 8.0 Hz, 1H), 7.45 (dd, *J* = 1.7 Hz, 7.7 Hz, 1H), 7.36-7.26 (m, 4H), 7.21-7.16 (m, 3H), 6.56 (bs, 1H, NH), 5.54 (d, *J* = 4.4 Hz, 1H), 4.54-4.39 (m, 2H), 4.13 (d, *J* = 4.4 Hz, 1H, OH); **^{13}C NMR ($CDCl_3$, 100 MHz) δ** 171.4, 138.9, 137.3, 132.9, 130.0, 128.7, 128.2, 127.6, 127.3, 122.8, 72.3, 43.7; TLC R_f 0.15 (EtOAc/hexane, 1/3); **HPLC conditions for 3r** : t_R 24.57 min (*R*-isomer), 28.22 min (*S*-isomer) (Chiralpak AD-H, hexane/*i*-PrOH, 92/8, 1.0 mL/min, λ = 254 nm); **HRMS (ESI)** Calcd for $C_{15}H_{14}BrNO_2$: 319.0208, found : 319.0207.

N-Benzyl 2-Oxo-Benzeneacetamide Derivatives (2b-r)



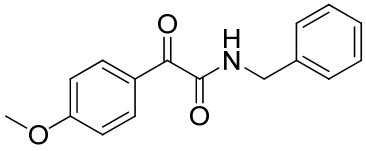
In a 100 ml double-neck round bottomed flask was placed respective *N*-benzyl, α-hydroxy-amide derivative (3.0 mmol, 1.0 eq.) in oxygen saturated toluene (30 mL). $VOCl_3$ (Vanadium(V) oxychloride, 28 l, 0.3 mmol, 0.1 eq.) was added slowly to the reaction mixture and stirred at room temperature. After the completion of reaction, solvent was evaporated under reduced pressure and the crude mixture directly loaded on column chromatography. Ethyl acetate/hexane, 1/10 mixture was used as eluents to get the product and recrystallized from hot diethyl ether to get *N*-benzyl-α- keto-amide derivatives.

N-Benzyl-2-oxo-2-*p*-tolyl-acetamide (2b) [1]



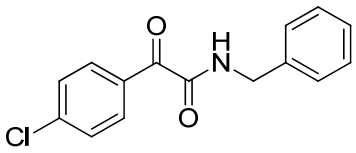
Data for 2b : ^{1}H NMR ($CDCl_3$, 400 MHz) δ 8.29 (d, *J* = 8.0 Hz, 2H), 7.40 (bs, 1H, NH), 7.38-7.27 (m, 7H), 4.56 (d, *J* = 6.0 Hz, 2H), 2.43 (s, 1H); **^{13}C NMR ($CDCl_3$, 100 MHz) δ** 187.0, 161.8, 145.6, 137.1, 131.3, 130.7, 129.1, 128.7, 127.7, 127.6, 43.3, 21.8; **TLC R_f** 0.2 (EtOAc/hexane, 1/10; **HRMS (ESI)** Calcd for $C_{16}H_{15}NO_2$: 253.1103, found : 253.1102.

N-Benzyl-2-oxo-2-(4'-methoxyphenyl)acetamide (2c) [1]



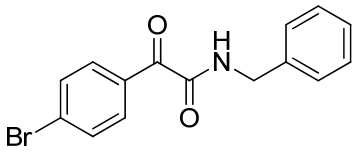
Data for 2c : ^{1}H NMR ($CDCl_3$, 400 MHz) δ 8.43 (d, *J* = 9.2 Hz, 2H), 7.38 (bs, 1H, NH), 7.37-7.29 (m, 5H), 6.94 (d, *J* = 9.2 Hz, 2H), 4.56 (d, *J* = 6.4 Hz, 2H), 3.89 (s, 1H); **^{13}C NMR ($CDCl_3$**, 100 MHz) δ 185.4, 164.6, 162.0, 137.2, 133.9, 128.7, 127.8, 127.7, 126.3, 113.8, 55.5, 43.3; **TLC R_f 0.36** (EtOAc/hexane, 3/1); **HRMS (ESI)** Calcd for $C_{16}H_{15}NO_3$: 269.1052, found : 269.1050 ($[M+H]^+$).

N-Benzyl-2-oxo-2-(4'-chlorophenyl)-acetamide (2d) [1]



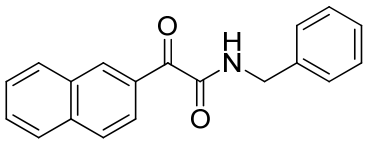
Data for 2d : ^{1}H NMR (CDCl$_3$, 400 MHz) δ 8.36 (d, *J* = 8.5 Hz, 2H), 7.46 (d, *J* = 8.5 Hz, 2H), 7.42 (bs, 1H, NH), 4.56 (d, *J* = 6.0 Hz, 2H); **^{13}C NMR (CDCl$_3$, 100 MHz) δ** 186.0, 161.1, 141.1, 136.9, 132.6, 131.5, 128.7, 127.7, 43.4; **TLC R_f** 0.525 (EtOAc/hexane, 1/3); **HRMS (ESI)** Calcd for $C_{15}H_{12}ClNO_2$: 273.0557, found : 273.0556($[M+H]^+$).

N-Benzyl-2-oxo-2-(4'-bromophenyl)-acetamide (2e) [1]



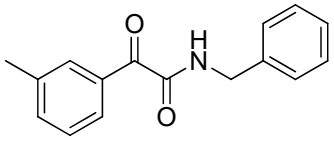
Data for 2e : ^{1}H NMR (CDCl$_3$, 400 MHz) δ 8.28 (d, *J* = 8.8 Hz, 2H), 7.63 (d, *J* = 8.4 Hz, 2H), 7.41 (bs, 1H, NH), 7.41-7.30 (m, 5H), 4.56 (d, *J* = 6.0 Hz, 2H); **^{13}C NMR (CDCl$_3$, 100 MHz) δ** 186.3, 161.0, 136.9, 132.7, 132.0, 131.8, 130.1, 128.8, 127.8, 43.5; **TLC R_f 0.15** (EtOAc/hexane, 1/10); **HRMS (ESI)** Calcd for $C_{15}H_{12}BrNO_2$: 317.0051 found : 317.0051.

N-Benzyl-2-oxo-2-naphthanlen-2-yl-acetamide (2f) [1]



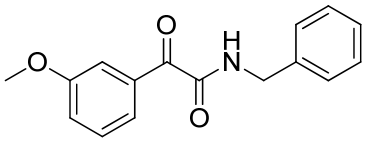
Data for 2f : **^{1}H NMR (CDCl$_3$, 400 MHz) δ** 9.25 (s, 1H), 8.21 (dd, *J* = 1.6 Hz, 8.6 Hz, 1H), 8.02 (d, *J* = 8.4 Hz, 1H), 7.90 (d, *J* = 8.8 Hz, 1H), 7.87 (d, *J* = 8.0 Hz, 1H), 7.64 (ddd, *J* = 1.2 Hz, 6.0 Hz, 7.0 Hz, 1H), 7.56 (ddd, *J* = 1.2 Hz, 6.0 Hz, 7.0 Hz, 1H), 7.55 (bs, 1H, NH), 7.40-7.30 (m, 5H), 4.62 (d, *J* = 6.0 Hz, 2H); **^{13}C NMR (CDCl$_3$, 100 MHz) δ** 187.0, 161.7, 137.1, 136.1, 135.0, 132.3, 130.5, 130.3, 129.3, 128.8, 128.3, 127.8, 127.8, 127.7, 126.8, 125.2, 43.5; **TLC R_f** 0.44 (EtOAc/hexane, 1/3); **HRMS (ESI)** Calcd for $C_{19}H_{15}NO_2$: 289.1103, found : 289.1103.

N-Benzyl-2-oxo-2-*m*-tolyl-acetamide (2g)[4]



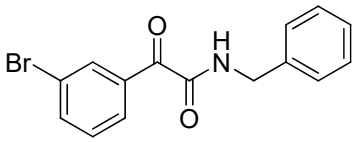
Data for 2g : ^{1}H NMR (CDCl$_3$, 400 MHz) δ 8.18 (s, 1H), 8.16 (s, 1H), 7.44 (d, *J* = 7.6 Hz, 1H), 7.39-7.28 (m, 7H), 4.57 (d, *J* = 6.0 Hz, 2H), 2.42 (s, 1H); **^{13}C NMR (CDCl$_3$, 100 MHz) δ** 187.7, 161.6, 138.3, 137.1, 135.2, 133.3, 131.5, 128.8, 128.4, 128.4, 127.8, 127.8, 43.4, 21.3; **TLC R_f** 0.2 (EtOAc/hexane, 10/1); **HRMS (ESI)** Calcd for $C_{16}H_{16}NO_2$: 254.1181 ($[M+H]^+$), found : 254.1178 ($[M+H]^+$).

N-Benzyl-2-oxo-2-(3'-methoxyphenyl)-acetamide (2h)



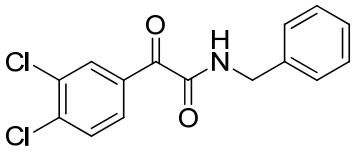
Data for 2h : ^{1}H NMR (CDCl$_3$, 400 MHz) δ 8.02 (dt, *J* = 7.6 Hz, 1.2 Hz, 1H), 7.86 (dd, *J* = 1.5 Hz, 2.6 Hz, 1H), 7.42-.7.31 (m, 7H), 7.18 (ddd, *J* = 8.2 Hz, 2.7 Hz, 0.9 Hz, 1H), 4.57 (d, *J* = 6.0 Hz, 2H), 3.86 (s, 3H); **^{13}C NMR (CDCl$_3$, 100 MHz) δ** 187.2, 161.5, 159.5, 137.0, 134.4, 129.5, 128.8, 127.8, 124.1, 121.5, 114.7, 55.4, 43.4; **TLC R$_f$** 0.22 (EtOAc/hexane, 1/6); **HRMS (ESI)** Calcd for $C_{16}H_{16}NO_3$: 270.1130 ($[M+H]^+$), found : 270.1133 ($[M+H]^+$).

N-Benzyl-2-oxo-2-(3'-bromophenyl)-acetamide (2i)[6]



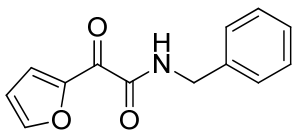
Data for 2i : **^{1}H NMR (CDCl$_3$, 400 MHz) δ** 8.51 (s, 1H), 8.34 (dd, *J* = 0.8 Hz, 7.6 Hz, 1H), 7.75 (td, *J* = 1.0 Hz, 7.8 Hz, 1H), 7.39-7.27 (m, 7H, NH), 4.57 (d, *J* = 6.0 Hz, 2H); **^{13}C NMR (CDCl$_3$, 100 MHz) δ** 186.0, 160.8, 137.2, 136.8, 134.9, 133.9, 130.0, 129.8, 128.8, 127.9, 122.6, 43.5; **TLC R$_f$** 0.5 (EtOAc/hexane, 1/3); **HRMS (ESI)** Calcd for $C_{15}H_{13}BrNO_2$: 318.0127 ($[M+H]^+$), found : 318.0127 ($[M+H]^+$).

N-Benzyl-2-oxo-2-(3',4'-dichlorophenyl)-acetamide (2j)



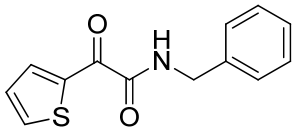
Data for 2j : ^{1}H NMR (CDCl$_3$, 400 MHz) δ 8.54 (d, *J* = 1.9 Hz, 1H), 8.27 (dd, *J* = 2.0 Hz, 8.4 Hz, 1H), 7.56 (d, *J* = 8.4 Hz, 1H), 7.43 (bs, 1H, NH), 7.39-7.29 (m, 5H), 4.56 (d, *J* = 6.0 Hz, 2H); **^{13}C NMR (CDCl$_3$, 100 MHz) δ** 184.9, 160.6, 139.3, 136.7, 133.1, 133.0, 132.7, 130.6, 130.3, 128.8, 127.9, 127.8, 43.5; **TLC R$_f$** 0.35 (EtOAc/hexane, 1/6); **HRMS (ESI)** Calcd for $C_{15}H_{12}Cl_2NO_2$: 308.0245 ($[M+H]^+$), found : 308.0241 ($[M+H]^+$).

N-Benzyl-2-oxo-2-furan-2-yl -acetamide (2k)[1]



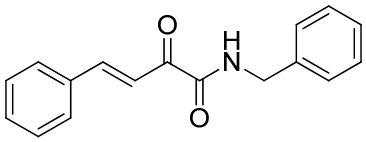
Data for 2k : **^{1}H NMR (CDCl$_3$, 400 MHz) δ** 8.22 (d, *J* = 3.5 Hz, 1H), 7.76 (d, *J* = 3.5 Hz, 1H), 7.60 (bs, 1H, NH), 7.37-7.30 (m ,5H), 6.63 (dd, *J* = 1.6 Hz, 3.7 Hz, 1H), 4.54 (d, *J* = 6.0 Hz, 2H); **^{13}C NMR (CDCl$_3$, 100 MHz) δ** 173.4, 159.9, 149.4, 136.8, 128.8, 127.8, 126.9, 113.1, 43.3; TLC R$_f$ 0.45 (EtOAc/hexane, 1/2); HRMS (ESI) Calcd for $C_{13}H_{11}NO_3$: 229.0739, found : 229.0739.

N-Benzyl-2-oxo-2-thiophen-2-yl -acetamide (2l) [1]



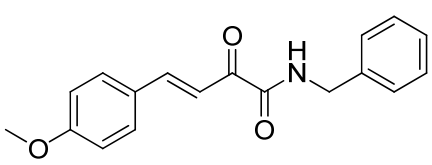
Data for 15 : ^{1}H NMR (CDCl$_3$, 400 MHz) δ 8.42 (dd, *J* = 1.0 Hz, 3.5 Hz, 1H), 7.84 (dd, *J* = 1.1 Hz, 4.8 Hz, 1H), 7.62 (bs, 1H, NH), 7.38-7.28 (m, 5H), 7.19 (dd, *J* = 4.7 Hz, 4.0 Hz, 1H), 4.56 (d, *J* = 6.0 Hz, 2H); **^{13}C NMR (CDCl$_3$, 100 MHz) δ** 178.1, 160.5, 138.6, 138.0, 136.9, 136.7, 128.7, 128.1, 127.8, 127.7, 43.4 ; **TLC R$_f$** 0.44 (EtOAc/hexane, 1/3); **HRMS (ESI)** Calcd for $C_{13}H_{11}NO_2S$: 245.0510, found : 245.0509.

2-Oxo-4-phenyl-but-3-enoic acid methylamide (2m) [1]



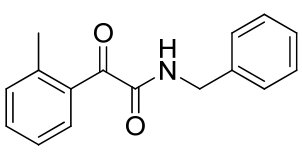
Data for 2m : ^{1}H NMR (CDCl$_3$, 400 MHz) δ 7.96 (d, *J* = 16.4 Hz, 1H), 7.81 (d, *J* = 16.0 Hz, 1H), 7.68 (dd, *J* = 1.6 Hz, 7.4 Hz, 2H), 7.49 (bs, 1H, NH), 7.47-7.28 (m, 9H), 4.54 (d, *J* = 6.0 Hz, 2H); **^{13}C NMR (CDCl$_3$, 100 MHz) δ** 185.3, 161.1, 148.1, 137.0, 134.3, 131.4, 129.1, 129.0, 128.8, 127.8, 127.8, 118.6, 43.5; **TLC R$_f$** 0.23 (EtOAc/hexane, 1/10); **HRMS (ESI)** Calcd for $C_{17}H_{15}NO_2$: 265.1103, found : 265.1103.

2-oxo-4-(4-methoxy-phenyl)-but-3-enoic acid methylamide (2n)



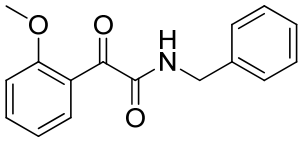
Data for 2n : ^{1}H NMR (CDCl$_3$, 400 MHz) δ 7.93 (d, *J* = 16.0 Hz, 1H), 7.68 (d, *J* = 16.0 Hz, 1H), 7.64 (dd, *J* = 7.1 Hz, 1.9 Hz, 2H), 7.51 (bs, 1H, NH), 7.37-7.30 (m, 5H), 6.93 (dd, *J* = 6.9 Hz, 1.9 Hz, 2H), 4.54 (d, *J* = 6.1 Hz, 2H), 3.86 (s, 3H); **^{13}C NMR (CDCl$_3$, 100 MHz) δ** 185.0, 162.5, 161.4, 148.0, 137.1, 131.1, 128.8, 127.8, 127.7, 127.2, 116.5, 114.5, 55.4, 43.5; **TLC R$_f$** 0.15 (EtOAc/hexane, 1/6); **HRMS (ESI)** Calcd for $C_{18}H_{18}NO_3$: 296.1286, found : 296.1286 ($[M+H]^+$).

N-Benzyl-2-oxo-2-*o*-tolyl-acetamide (2o) [1]



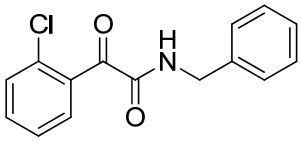
Data for 2o : ^{1}H NMR (CDCl$_3$, 400 MHz) δ 7.97 (d, *J* = 7.8 Hz, 1H), 7.45 (td, *J* = 1.3 Hz, 8.7 Hz, 1H), 7.39-7.27 (m, 8H), 4.57 (d, *J* = 6.0 Hz, 2H), 2.50 (s, 3H); **^{13}C NMR (CDCl$_3$, 100 MHz) δ** 191.0, 161.8, 140.1, 137.1, 132.7, 132.6, 132.0, 131.6, 128.8, 127.8, 127.7, 125.3, 43.5, 20.8; **TLC R$_f$** 0.45 (EtOAc/hexane, 1/3); **HRMS (ESI)** Calcd for $C_{16}H_{15}NO_2$: 253.1103, found : 253.1102 ($[M+H]^+$).

N-Benzyl-2-oxo-2-(2'-methoxyphenyl)-acetamide (2p) [1]



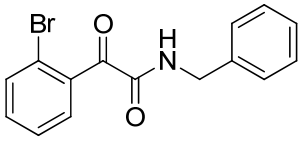
Data for 2p : ^{1}H NMR ($CDCl_3$, 400 MHz) δ 7.68 (dd, *J* = 2.0 Hz, 7.6 Hz, 2H), 7.41 (ddd, *J* = 1.6 Hz, 7.4 Hz, 8.5 Hz, 1H), 7.38-7.28 (m, 5H), 7.03 (td, *J* = 0.8 Hz, 7.6 Hz, 1H), 6.97 (d, *J* = 8.0 Hz, 2H), 4.56 (d, *J* = 6.0 Hz, 2H), 3.79 (s, 3H); **^{13}C NMR ($CDCl_3$, 100 MHz) δ** 191.4, 163.0, 159.5, 137.4, 134.6, 131.0, 128.7, 127.8, 127.6, 124.6, 120.7, 111.9, 56.0, 43.3; **TLC R_f** 0.35 (EtOAc/hexane, 1/2); **HRMS (ESI)** Calcd for $C_{16}H_{15}NO_3$: 269.1052, found : 269.1050.

N-Benzyl-2-oxo-2-(2'-chlorophenyl)-acetamide (2q) [1]



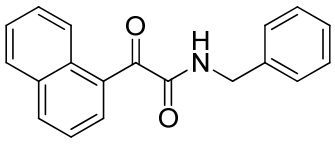
Data for 2q : ^{1}H NMR ($CDCl_3$, 400 MHz) δ 7.71-7.69 (m, 1H), 7.50-7.44 (m, 3H), 7.39-7.32 (m, 6H, NH), 4.58 (d, *J* = 6.0 Hz, 2H); **^{13}C NMR ($CDCl_3$, 100 MHz) δ** 189.9, 160.5, 136.8, 133.9, 133.0, 131.2, 130.3, 128.7, 127.8, 127.8, 126.5, 43.6; **TLC R_f** 0.4 (EtOAc/hexane, 1/3); **HRMS (ESI)** Calcd for $C_{15}H_{12}ClNO_2$: 273.0557, found : 273.0557.

N-Benzyl-2-oxo-2-(2'-bromophenyl)-acetamide (2r) [4]



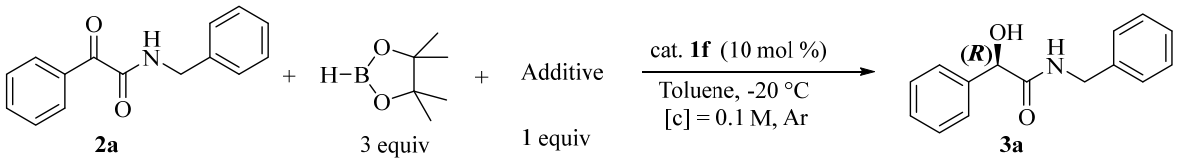
Data for 2r : ^{1}H NMR ($CDCl_3$, 400 MHz) δ 7.66-7.63 (m, 2H), 7.44-7.30 (m, 8H), 4.57 (d, *J* = 6.4 Hz, 2H); **^{13}C NMR ($CDCl_3$, 100 MHz) δ** 190.5, 160.1, 136.8, 135.9, 133.45, 132.9, 131.1, 127.9, 127.8, 127.0, 120.8, 43.7; **TLC R_f** 0.4 (EtOAc/hexane, 1/3); **HRMS (ESI)** Calcd for $C_{15}H_{12}BrNO_2$: 317.0051, found : 317.0052.

N-Benzyl-2-oxo-2-naphthanlen-1-yl -acetamide (2s) [1]



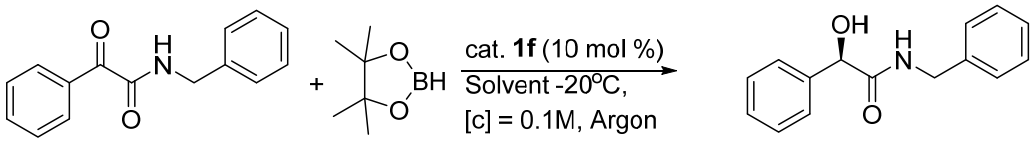
Data for 2s : **^{1}H NMR ($CDCl_3$, 400 MHz) δ** 8.58 (d, *J* = 8.4 Hz, 1H), 8.36 (dd, *J* = 1.2 Hz, 7.2 Hz, 1H), 8.08 (d, *J* = 8.4 Hz, 1H), 7.91 (dd, *J* = 0.8 Hz, 8.8 Hz, 1H), 7.64-7.53 (m, 3H), 7.52 (bs, 1H, NH), 7.44-7.31 (m, 5H), 4.63 (d, *J* = 6 Hz, 2H); **^{13}C NMR ($CDCl_3$, 100 MHz) δ** 190.1, 161.9, 137.1, 134.6, 133.8, 133.2, 131.1, 129.5, 128.8, 128.6, 128.4, 127.9, 127.8, 126.5, 124.2, 43.7; **TLC R_f** 0.36 (EtOAc/hexane, 1/4); **HRMS (ESI)** Calcd for $C_{19}H_{15}NO_2$: 289.1103, found : 289.1101.

6. Table S1: Effect of additives on the asymmetric reduction.



Entry	Reducing Agent	Additive	Time (h)	Yield (%)	Ee (%)
1	PinB–H	—	96	65	88 (*R*)
2	PinB–H	MeOH	96	40	99 (*R*)
3	PinB–H	CCl_3CH_2OH	96	47	91 (*R*)
4	PinB–H	CF_3CH_2OH	96	22	96 (*R*)
5	CatB–H	—	14	99	64 (*S*)
6	CatB–H	MeOH	14	92	47 (*S*)
7	CatB–H	CCl_3CH_2OH	14	86	58 (*S*)
8	CatB–H	CF_3CH_2OH	14	84	62 (*S*)

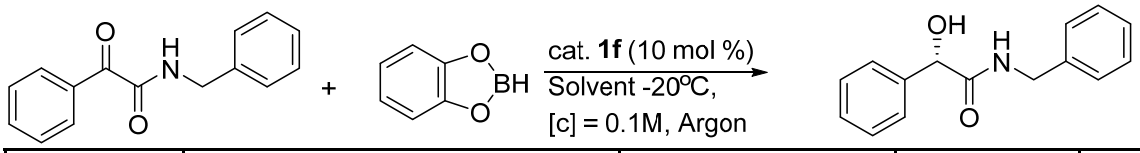
Table S2: Solvent Screening with HBPin:



entry	Solvent	Time(h)	Yield(%)	ee%
1 (final)	toluene	24+24+48	65	88 (*R*)
2	MeOH	24+24+48	35	52 (*R*)
3	*i*-PrOH	24+24+48	43	56 (*R*)
4	1,4-dioxane	24+24+48	21	36 (*R*)

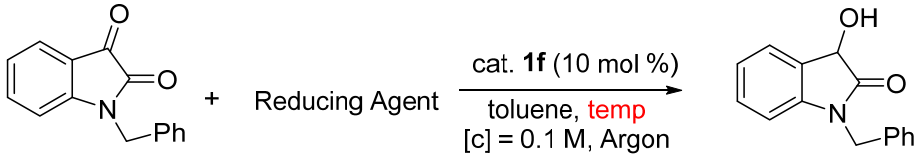
5	CH_3CN	24+24+48	28	38 (*R*)
6	DMSO (r.t.)	24+24+48	52	12 (*R*)
7	DMF	24+24+48	19	24 (*R*)

Table S3: Solvent Screening with HBCat:



entry	Solvent	Time(h)	Yield(%)	ee%
1 (final)	toluene	12	99	64 (*S*)
2	CH_2Cl_2	12	97	2 (*R*)
3	PhCl	14	95	57 (*S*)
4	THF	12	98	35 (*S*)
5	*t*-BuOMe	15	87	4 (*R*)

Table S4



mw: 237.26

mw: 239.27

reducing agent	temperature, °C	Yield, %	ee, %

HBPin	−20/-40	99/0	0/---
HBCat	−20/-40	92/28	2/8 (*S*)

7. X-ray data of Catalyst 1f:

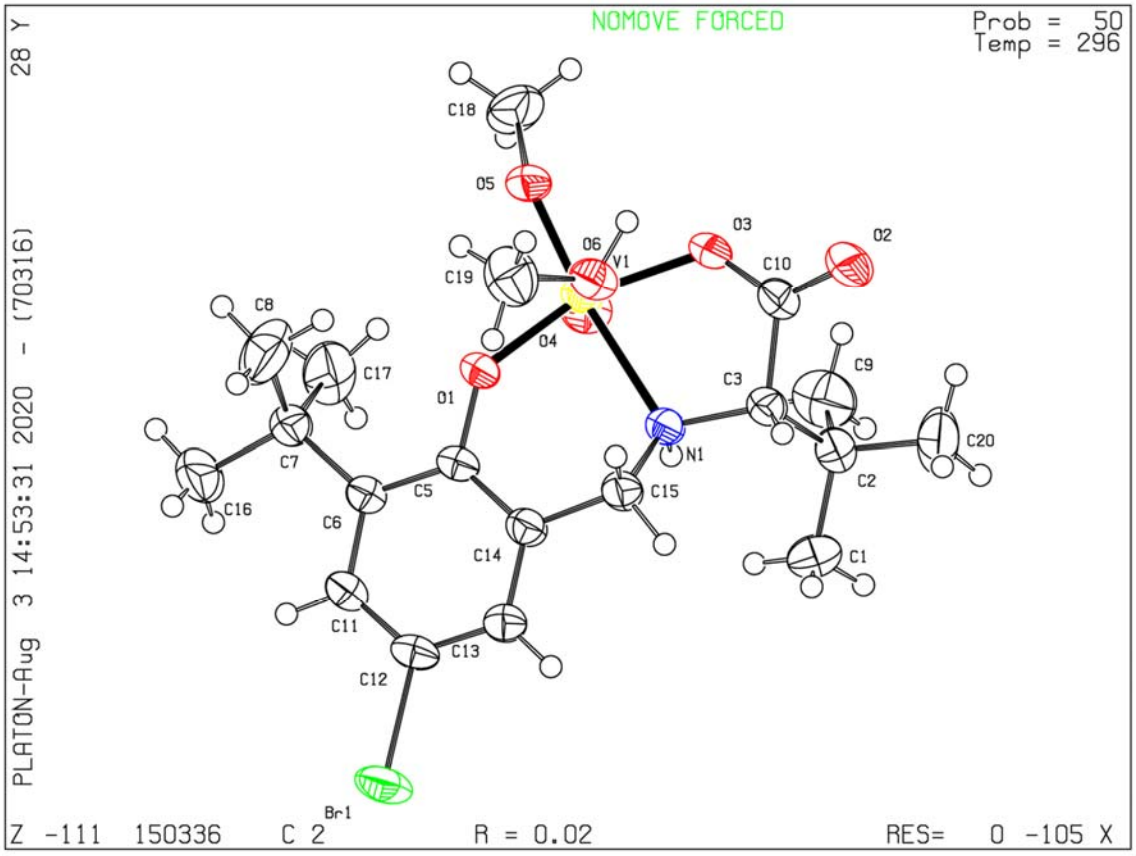


Figure S1. X-ray ORTEP drawing of catalyst **1f** (thermal ellipsoid is scaled to the 50% probability level).

Table 1. Crystal data and structure refinement for 150336.

Identification code	150336
Empirical formula	C19 H31 Br N O6 V
Formula weight	500.30
Temperature	296(2) K
Wavelength	0.71073 Å

Crystal system	Monoclinic	
Space group	C 2	
Unit cell dimensions	a = 27.3247(8) Å	α= 90°.
	b = 7.3295(2) Å	β= 102.472(2)°.
	c = 11.7690(3) Å	γ = 90°.
Volume	2301.43(11) $Å^3$	
Z	4	
Density (calculated)	1.444 Mg/m^3	
Absorption coefficient	2.199 mm^{-1}	
F(000)	1032	
Crystal size	0.15 x 0.15 x 0.12 mm^3	
Theta range for data collection	1.526 to 26.394°.	
Index ranges	-34<=h<=34, -9<=k<=9, -14<=l<=14	
Reflections collected	10084	
Independent reflections	4260 [R(int) = 0.0237]	
Completeness to theta = 25.242°	99.9 %	
Absorption correction	Semi-empirical from equivalents	
Max. and min. transmission	0.9485 and 0.742	
Refinement method	Full-matrix least-squares on F^2	
Data / restraints / parameters	4260 / 1 / 262	
Goodness-of-fit on F^2	0.558	
Final R indices [I>2sigma(I)]	R1 = 0.0249, wR2 = 0.0711	
R indices (all data)	R1 = 0.0275, wR2 = 0.0762	
Absolute structure parameter	0.013(10)	
Extinction coefficient	n/a	
Largest diff. peak and hole	0.561 and -0.339 e.$Å^{-3}$	

Datablock: 150336

Bond precision: C-C = 0.0058 A Wavelength=0.71073
Cell: a=27.3247(8) b=7.3295(2) c=11.7690(3)
alpha=90 beta=102.472(2) gamma=90

Temperature: 296 K

	Calculated	Reported
Volume	2301.43(11)	2301.43(11)
Space group	C 2	C 2
Hall group	C 2y	C 2y
Moiety formula	C19 H31 Br N O6 V	?
Sum formula	C19 H31 Br N O6 V	C19 H31 Br N O6 V
Mr	500.29	500.30
Dx,g cm-3	1.444	1.444
Z	4	4
Mu (mm-1)	2.199	2.199
F000	1032.0	1032.0
F000'	1032.50	
h,k,lmax	34,9,14	34,9,14
Nref	4727[2556]	4260
Tmin,Tmax	0.726,0.768	0.742,0.948
Tmin'	0.712	

Correction method= # Reported T Limits: Tmin=0.742 Tmax=0.948 AbsCorr = MULTI-SCAN

Data completeness= 1.67/0.90 Theta(max)= 26.394

R(reflections)= 0.0249(3926) wR2(reflections)= 0.0762(4260)

S = 0.558 Npar= 262

The following ALERTS were generated. Each ALERT has the format
test-name_ALERT_alert-type_alert-level.
Click on the hyperlinks for more details of the test.

Alert level B

GOODF01_ALERT_2_B The least squares goodness of fit parameter lies outside the range 0.60 <> 4.00
Goodness of fit given = 0.558

Alert level C

PLAT242_ALERT_2_C Low 'MainMol' Ueq as Compared to Neighbors of C2 Check
PLAT242_ALERT_2_C Low 'MainMol' Ueq as Compared to Neighbors of C7 Check
PLAT420_ALERT_2_C D-H Without Acceptor N1 --H1 . Please Check
PLAT911_ALERT_3_C Missing FCF Refl Between Thmin & STh/L= 0.600 2 Report
PLAT915_ALERT_3_C No Flack x Check Done: Low Friedel Pair Coverage 79 %

PLAT915_ALERT_3_C No Flack x Check Done: Low Friedel Pair Coverage 79
%
PLAT918_ALERT_3_C Reflection(s) with I(obs) much Smaller I(calc) . 1
Check
PLAT939_ALERT_3_C Large Value of Not (SHELXL) Weight Optimized S . 15.00
Check

Alert level G

PLAT007_ALERT_5_G Number of Unrefined Donor-H Atoms 2
Report
PLAT128_ALERT_4_G Alternate Setting for Input Space Group C2 I2
Note
PLAT169_ALERT_4_G The CIF-Embedded .res File Contains AFIX 1 Recds 1
Report
PLAT791_ALERT_4_G Model has Chirality at N1 (Sohnke SpGr) R
Verify
PLAT791_ALERT_4_G Model has Chirality at C3 (Sohnke SpGr) S
Verify
PLAT794_ALERT_5_G Tentative Bond Valency for V1 (V) . 5.18
Info
PLAT883_ALERT_1_G No Info/Value for _atom_sites_solution_primary . Please
Do !
PLAT912_ALERT_4_G Missing # of FCF Reflections Above STh/L= 0.600 8
Note
PLAT913_ALERT_3_G Missing # of Very Strong Reflections in FCF 1
Note
PLAT941_ALERT_3_G Average HKL Measurement Multiplicity 4.0
Low
PLAT965_ALERT_2_G The SHELXL WEIGHT Optimisation has not Converged Please
Check
PLAT978_ALERT_2_G Number C-C Bonds with Positive Residual Density. 4
Info

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0 **ALERT level A** = Most likely a serious problem - resolve or explain
1 **ALERT level B** = A potentially serious problem, consider carefully
8 **ALERT level C** = Check. Ensure it is not caused by an omission or oversight
12 **ALERT level G** = General information/check it is not something unexpected

1 ALERT type 1 CIF construction/syntax error, inconsistent or missing data
6 ALERT type 2 Indicator that the structure model may be wrong or deficient
7 ALERT type 3 Indicator that the structure quality may be low
5 ALERT type 4 Improvement, methodology, query or suggestion
2 ALERT type 5 Informative message, check

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8. References

1. S. S. Weng, M. W. Shen, J. Q. Kao, Y. S. Munot, C. T. Chen, Chiral N-Salicylidene Vanadyl Carboxylate-Catalyzed Enantioselective Aerobic Oxidation of α-Hydroxy Esters and Amides, *Proc. Natl. Acad. Sci. U S A* **2006**, *103*, 3522-3527.
2. S. E.; Denmark, Y. Fan, The First Catalytic, Asymmetric α-Additions of Isocyanides. Lewis-Base-Catalyzed, Enantioselective Passerini-Type Reactions., *J. Am. Chem. Soc.* **2003**, *125*, 7825-

7827.
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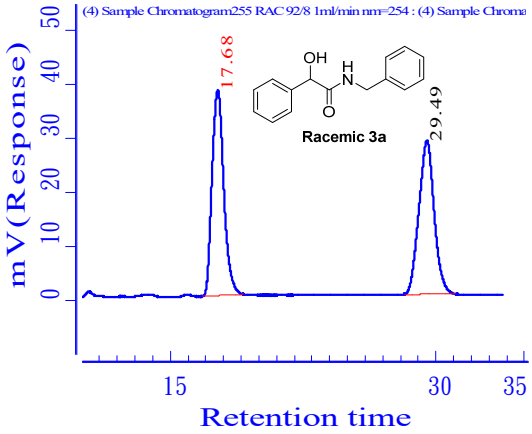
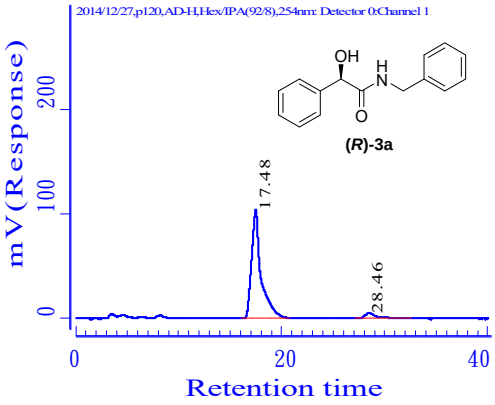
5. Y.-K. Zhang and B. Wang, European, Synthesis of α-Ketoamides from β-Ketonitriles and Primary Amines: A Catalyst-Free Oxidative Decyanation–Amidation Reaction, *J. Org. Chem.*, 2019, **2019**, 5732–5735.
6. Q. W. Tan, P. Chovatia and M. C. Willis, Copper-Catalysed Synthesis of Alkylidene 2-Pyrrolinone Derivatives from the Combination of α-Keto Amides and Alkynes, *Org. Biomol. Chem.*, 2018, **16**, 7797–7800.

9. HPLC chromatograms:

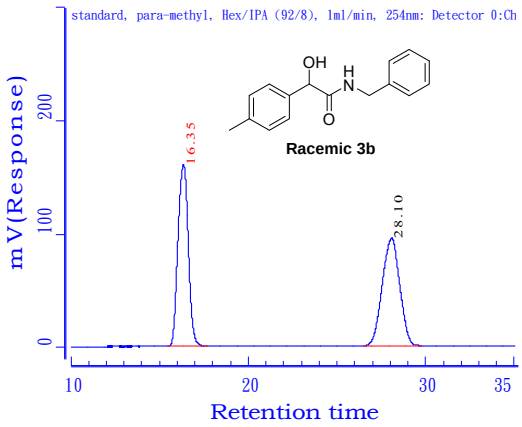


#	Peak name	ix/min (mim)	Area	Area %	Assay %
0		17.682	1761.21	50.72	0.00
1		29.485	1711.11	49.28	0.00

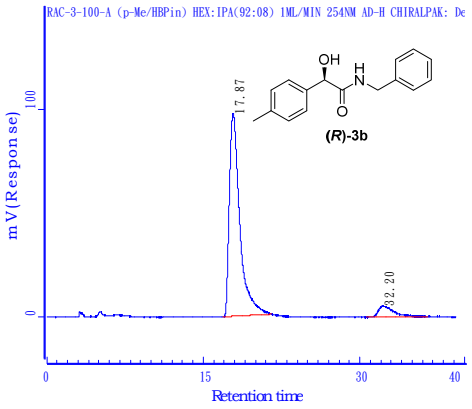


#	Peak name	x/min (mim)	Area	Area %	Assay %
0		17.484	6668.31	94.05	0.00
1		28.462	421.77	5.95	0.00

HPLC conditions for **3a** : t_R 17.68 min (major ***R***-iosmer), 29.49 min (***S***-isomer) (Chiralpak AD-H, hexane/*i*-PrOH, 92/8, 1.0 ml/min, λ = 254 nm) for 88% ee (***R***)

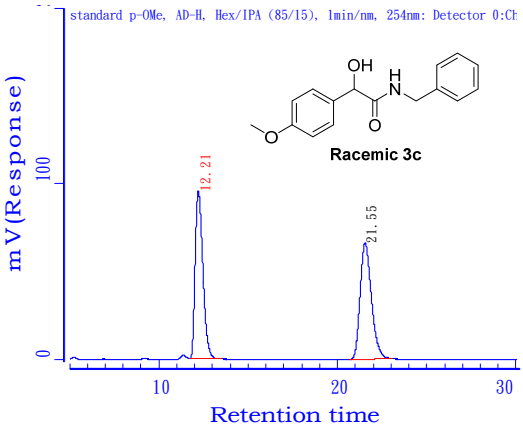


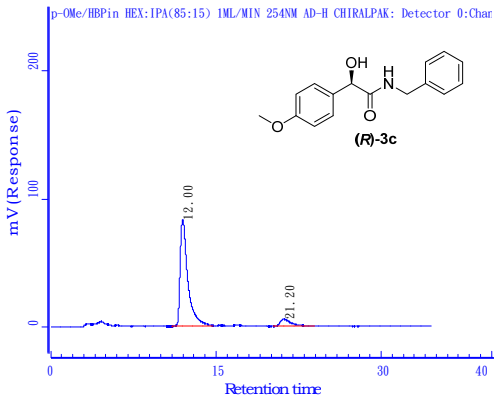
#	Peak name	max/min (mim)	Area	Area %	Assay %
0		16.354	6491.10	49.87	0.00
1		28.096	6524.62	50.13	0.00



#	Peak name	ax/min (mim)	Area	Area %	Assay %
0		17.866	6814.76	92.32	0.00
1		32.201	566.64	7.68	0.00

HPLC conditions for **3b** : t_R 16.35 min (major ***R***-iosmer), 28.10 min (***S***-isomer) (Chiralpak AD-H, hexane/*i*-PrOH, 92/8, 1.0 ml/min, λ = 254 nm) for 85% ee (***R***).

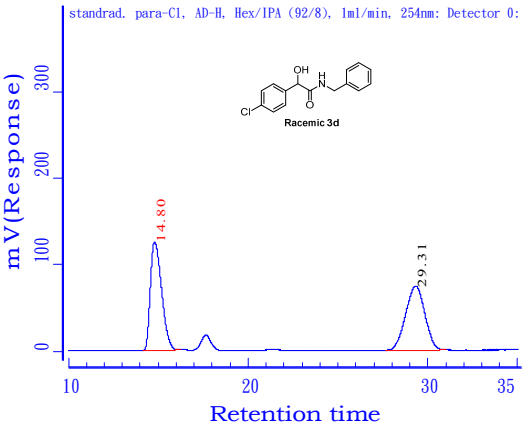


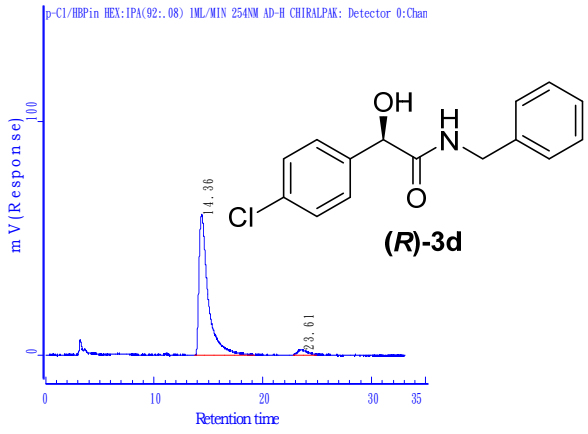


#	Peak name	max/min (mim)	Area	Area %	Assay %
0		12.212	2869.52	49.68	0.00
1		21.554	2906.32	50.32	0.00

#	Peak name	ax/min (mim)	Area	Area %	Assay %
0		11.995	4028.13	91.04	0.00
1		21.201	396.56	8.96	0.00

HPLC conditions for **3c** : t_R 12.21 min (major ***R***-iosmer), 21.55 min (***S***-isomer) (Chiralpak AD-H, hexane/*i*-PrOH, 85/15, 1.0 ml/min, λ = 254 nm) for 82% ee (***R***).

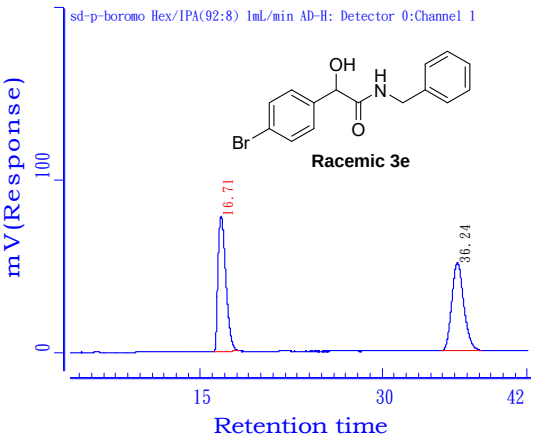


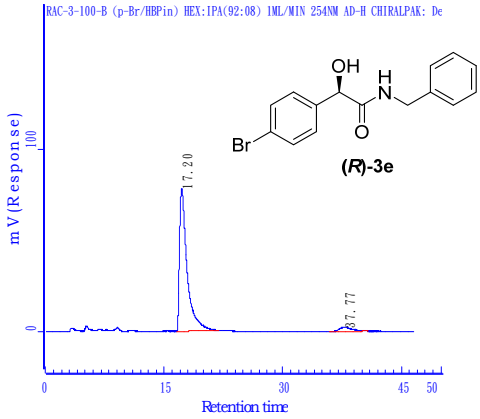


#	Peak name	max/min (mim)	Area	Area %	Assay %
0		14.800	5462.06	49.93	0.00
1		29.312	5478.30	50.07	0.00

#	Peak name	ax/min (mim)	Area	Area %	Assay %
0		14.358	3538.19	96.27	0.00
1		23.606	137.24	3.73	0.00

HPLC conditions for **3d** : t_R 14.30 min (major ***R***-iosmer), 29.31 min (***S***-isomer) (Chiralpak AD-H, hexane/*i*-PrOH, 92/8, 1.0 ml/min, λ = 254 nm) for 93% ee (***R***).

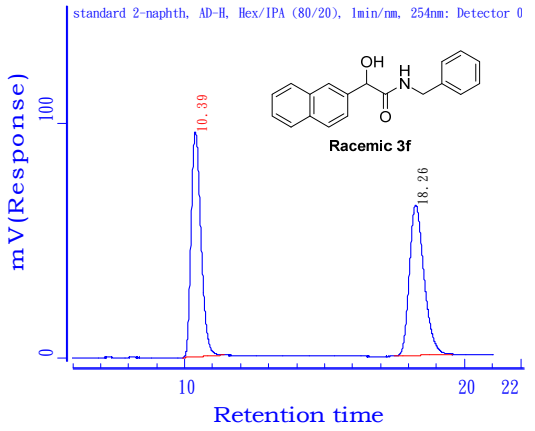


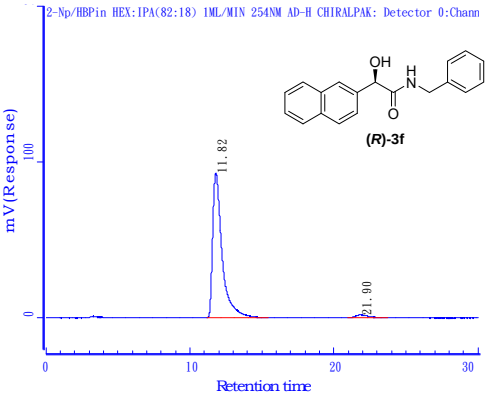


#	Peak name	ax/min (mim)	Area	Area %	Assay %
0		16.712	3511.85	49.93	0.00
1		36.236	3522.01	50.07	0.00

#	Peak name	ax/min (mim)	Area	Area %	Assay %
0		17.197	5247.14	95.67	0.00
1		37.770	237.55	4.33	0.00

HPLC conditions for **3e** : t_R 16.71 min (major ***R***-iosmer), 36.24 min (***S***-isomer) (Chiralpak AD-H, hexane/*i*-PrOH, 92/8, 1.0 ml/min, λ = 254 nm) for 91% ee (***R***).

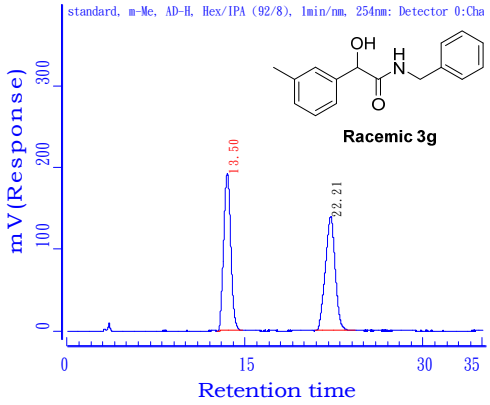




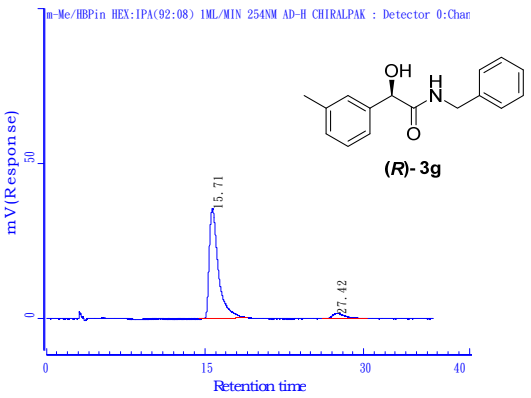
#	Peak name	max/min (mim)	Area	Area %	Assay %
0		10.388	2339.20	50.02	0.00
1		18.259	2337.73	49.98	0.00

#	Peak name	ax/min (mim)	Area	Area %	Assay %
0		11.817	4249.61	97.15	0.00
1		21.898	124.78	2.85	0.00

HPLC conditions for **3f** : t_R 10.39 min (major ***R***-iosmer), 18.26 min (***S***-isomer) (Chiralpak AD-H, hexane/*i*-PrOH, 80/20, 1.0 ml/min, λ = 254 nm) for 94% ee(***R***).

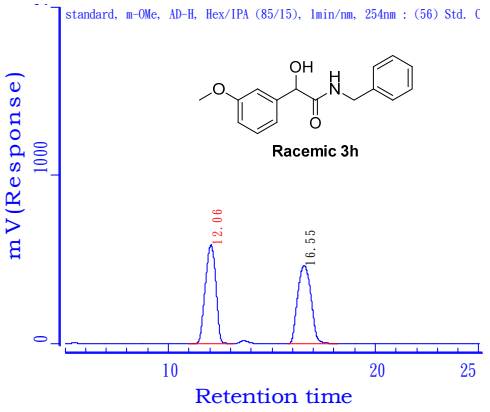


#	Peak name	max/min (mim)	Area	Area %	Assay %
0		13.501	8072.94	49.88	0.00
1		22.210	8112.04	50.12	0.00

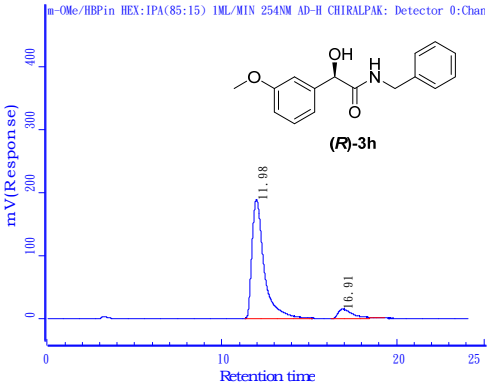


#	Peak name	ax/min (mim)	Area	Area %	Assay %
0		15.715	2137.57	93.23	0.00
1		27.420	155.19	6.77	0.00

HPLC conditions for **3g** : t_R 13.50 min (major ***R***-iosmer), 22.21 min (***S***-isomer) (Chiralpak AD-H, hexane/*i*-PrOH, 92/8, 1.0 ml/min, λ = 254 nm) for 86% ee (***R***).

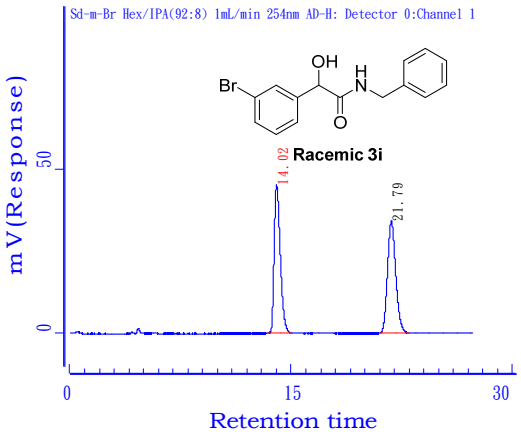


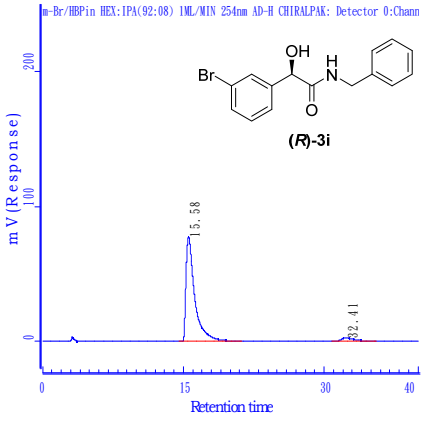
#	Peak name	max/min (mim)	Area	Area %	Assay %
0		12.058	20649.21	49.89	0.00
1		16.553	20737.65	50.11	0.00



#	Peak name	ax/min (mim)	Area	Area %	Assay %
0		11.983	9155.65	91.41	0.00
1		16.911	860.30	8.59	0.00

HPLC conditions for **3h** : t_R 12.06 min (major ***R***-iosmer), 16.55 min (***S***-isomer) (Chiralpak AD-H, hexane/*i*-PrOH, 85/15, 1.0 ml/min, λ = 254 nm) for 83% ee (***R***).

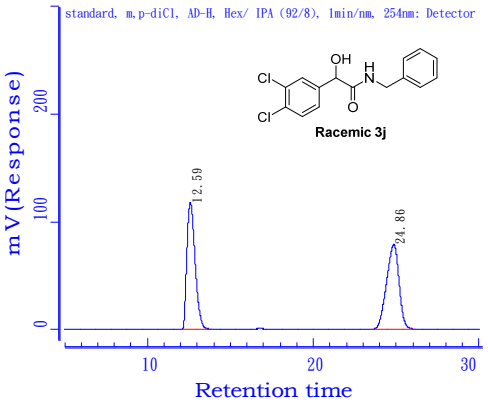


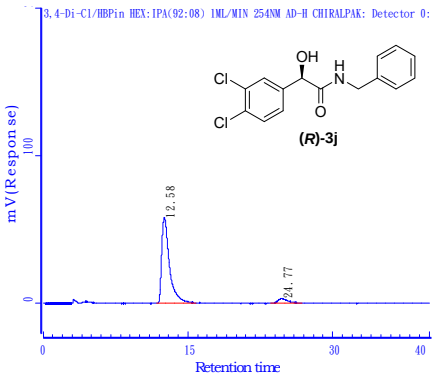


#	Peak name	ax/min (mim)	Area	Area %	Assay %
0		14.022	1308.88	49.52	0.00
1		21.789	1334.19	50.48	0.00

#	Peak name	ax/min (mim)	Area	Area %	Assay %
0		15.577	5073.37	95.49	0.00
1		32.406	239.47	4.51	0.00

HPLC conditions for **3i** : t_R 14.02 min (major ***R***-iosmer), 21.79 min (***S***-isomer) (Chiralpak AD-H, hexane/*i*-PrOH, 92/8, 1.0 ml/min, λ = 254 nm) for 91% ee (***R***).

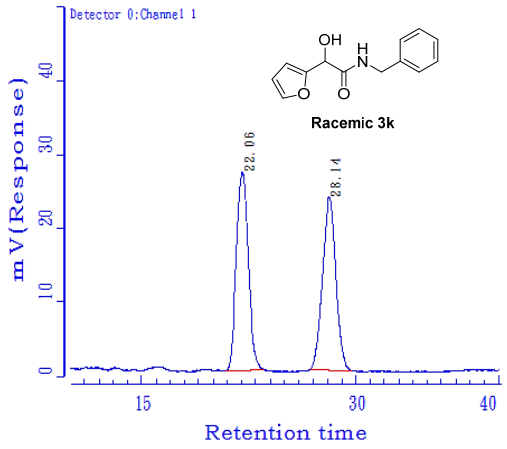




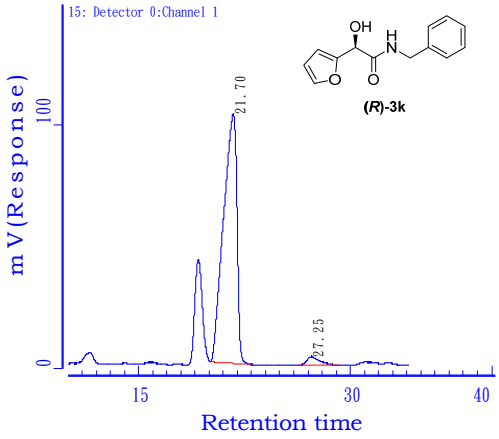
#	Peak name	max/min (mim)	Area	Area %	Assay %
0		12.586	4074.42	49.80	0.00
1		24.858	4106.63	50.20	0.00

#	Peak name	ax/min (mim)	Area	Area %	Assay %
0		12.576	3250.54	93.27	0.00
1		24.768	234.55	6.73	0.00

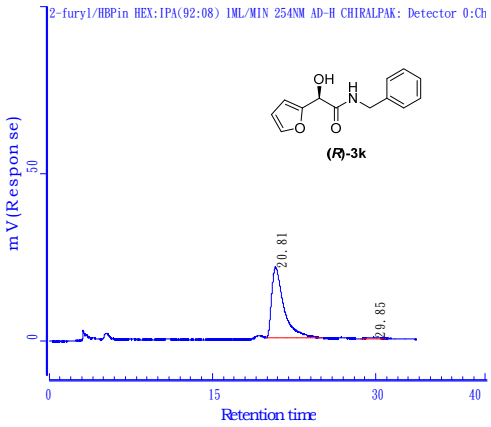
HPLC conditions for **3j** : t_R 12.59 min (major ***R***-iosmer), 24.86 min (***S***-isomer) (Chiralpak AD-H, hexane/*i*-PrOH, 92/8, 1.0 ml/min, λ = 254 nm) for 87% ee.



#	Peak name	x/min (mim)	Area	Area %	Assay %
0		22.065	1560.10	50.33	0.00
1		28.144	1539.55	49.67	0.00

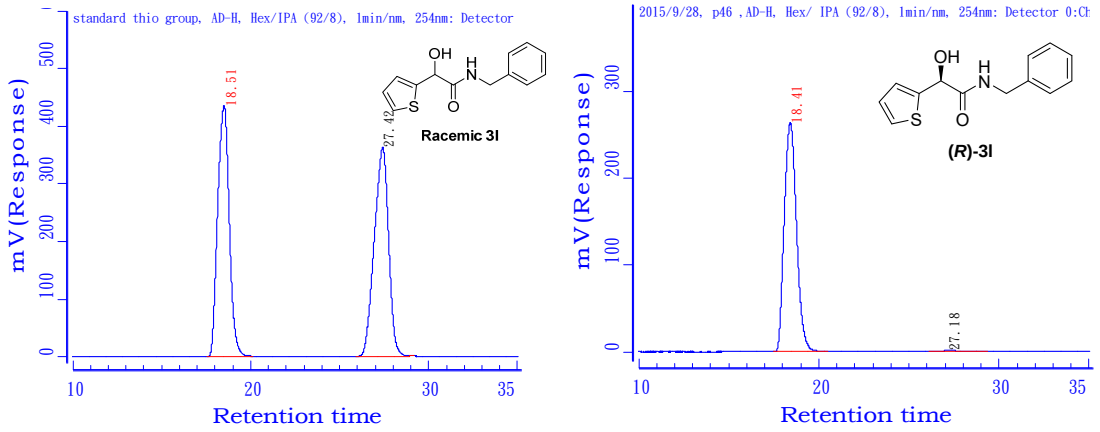


#	Peak name	x/min (mim)	Area	Area %	Assay %
0		21.704	6793.19	96.85	0.00
1		27.245	220.89	3.15	0.00



#	Peak name	ax/min (mim)	Area	Area %	Assay %
0		20.815	1650.71	98.01	0.00
1		29.852	33.54	1.99	0.00

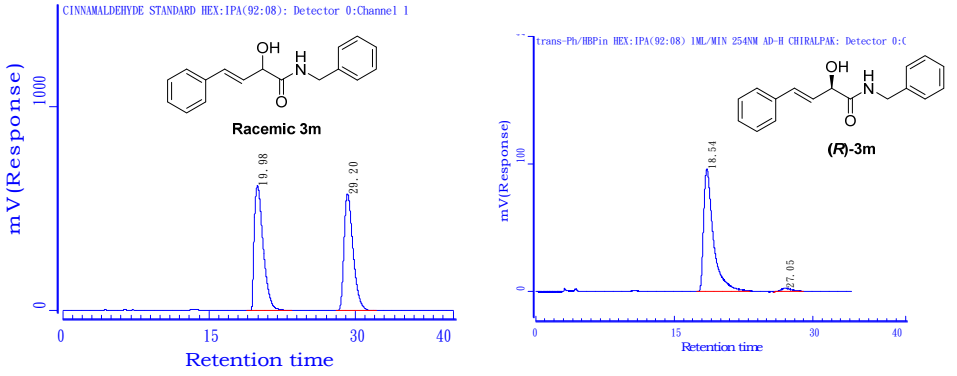
HPLC conditions for **3k** : t_R 22.06 min (major ***R***-iosmer), 28.14 min (***S***-isomer) (Chiralpak AD-H, hexane/*i*-PrOH, 92/8, 1.0 ml/min, λ = 254 nm) for 94% ee (***R***) and 96% ee (*R*).



#	Peak name	max/min (mim)	Area	Area %	Assay %
0		18.508	19638.88	49.29	0.00
1		27.419	20202.77	50.71	0.00

#	Peak name	max/min (mim)	Area	Area %	Assay %
0		18.414	12423.07	99.62	0.00
1		27.179	47.99	0.38	0.00

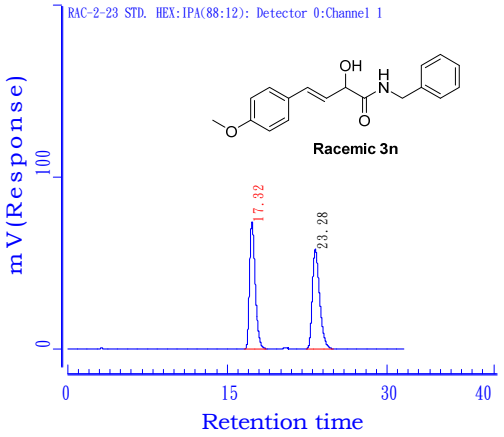
HPLC conditions for **3l** : t_R 18.51 min (major ***R***-iosmer), 27.42 min (***S***-isomer) (Chiralpak AD-H, hexane/*i*-PrOH, 92/8, 1.0 ml/min, λ = 254 nm) for 99% ee (***R***).



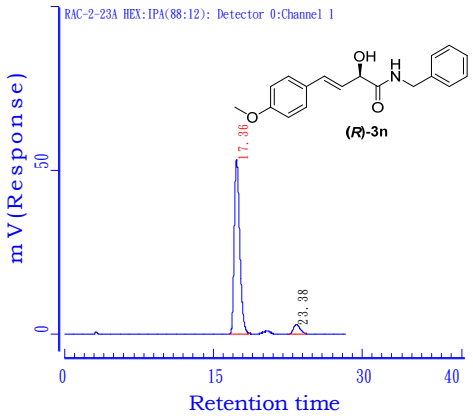
#	Peak name	ax/min (mim)	Area	Area %	Assay %
0		19.984	39344.14	51.41	0.00
1		29.205	37185.57	48.59	0.00

#	Peak name	ax/min (mim)	Area	Area %	Assay %
0		18.539	7085.97	97.03	0.00
1		27.047	216.80	2.97	0.00

HPLC conditions for **3m** : t_R 19.98 min (major ***R***-iosmer), 29.20 min (***S***-isomer) (Chiralpak AD-H, hexane/*i*-PrOH, 92/8, 1.0 ml/min, λ = 254 nm) for 93% ee (***R***).

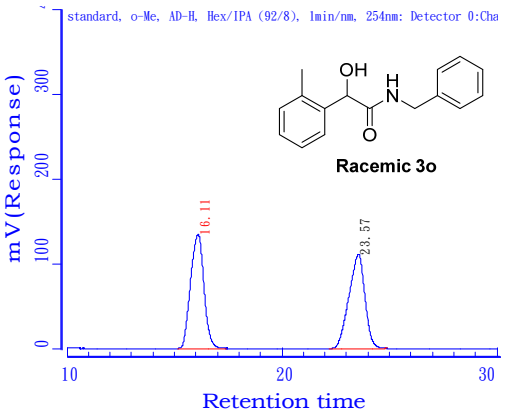


#	Peak name	ax/min (mim)	Area	Area %	Assay %
0		17.321	2758.72	49.86	0.00
1		23.284	2774.73	50.14	0.00

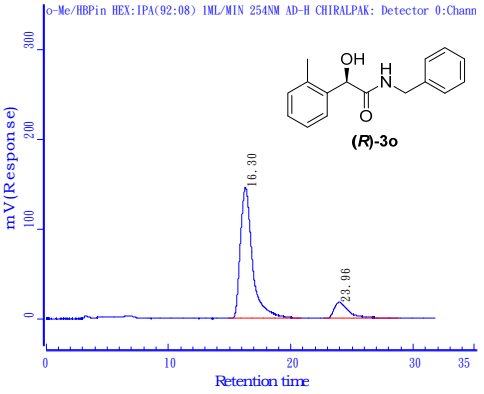


#	Peak name	ax/min (mim)	Area	Area %	Assay %
0		17.364	2061.16	93.52	0.00
1		23.378	142.73	6.48	0.00

HPLC conditions for **3n** : t_R 18.32 min (major ***R***-iosmer), 23.28 min (***S***-isomer) (Chiralpak AD-H, hexane/*i*-PrOH, 88/12, 1.0 ml/min, λ = 254 nm) for 87% ee.

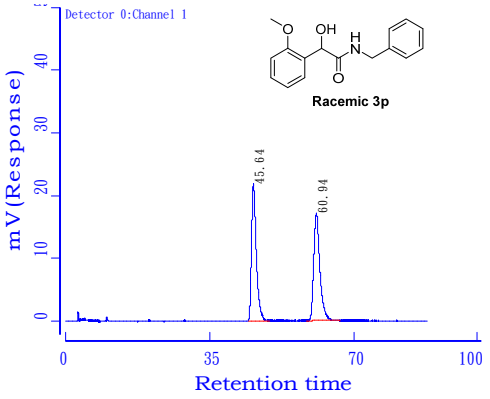


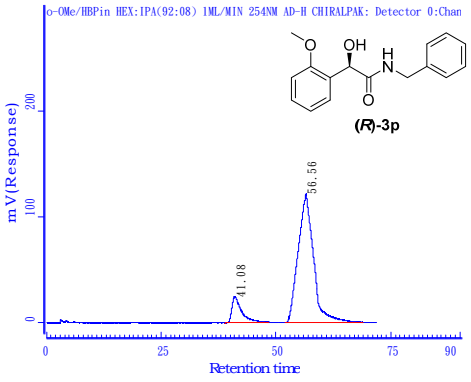
#	Peak name	max/min (mim)	Area	Area %	Assay %
0		16.110	5815.39	50.37	0.00
1		23.570	5731.04	49.63	0.00



#	Peak name	ax/min (mim)	Area	Area %	Assay %
0		16.298	10030.24	86.80	0.00
1		23.964	1525.81	13.20	0.00

HPLC conditions for **3o** : t_R 16.11 min (major ***R***-iosmer), 23.57 min (***S***-isomer) (Chiralpak AD-H, hexane/*i*-PrOH, 92/8, 1.0 ml/min, λ = 254 nm) for 74% ee.

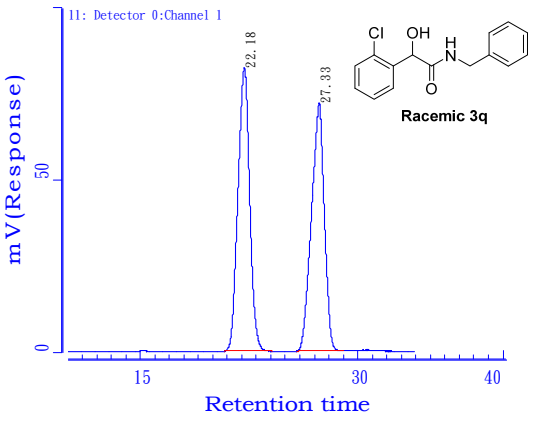


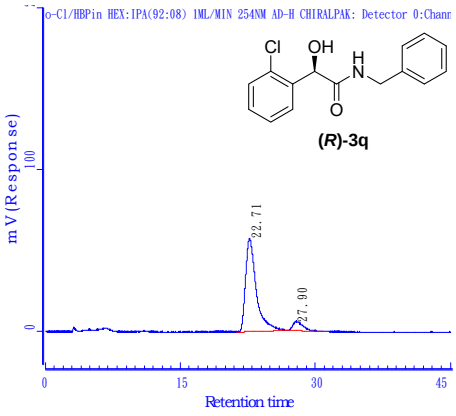


#	Peak name	ax/min (mim)	Area	Area %	Assay %
0		45.639	1758.72	50.01	0.00
1		60.939	1758.36	49.99	0.00

#	Peak name	ax/min (mim)	Area	Area %	Assay %
0		41.079	3594.86	11.30	0.00
1		56.565	28206.65	88.70	0.00

HPLC conditions for **3p** : t_R 16.11 min (***R***-iosmer), 23.57 min (major ***S***-isomer) (Chiralpak AD-H, hexane/*i*-PrOH, 92/8, 1.0 ml/min, λ = 254 nm) for 77% ee.

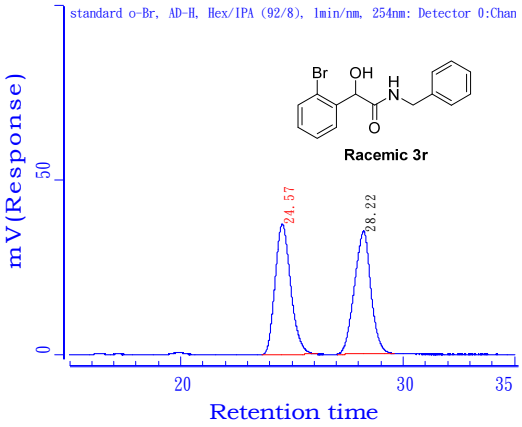


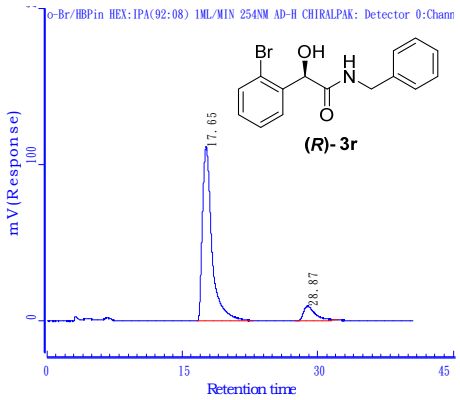


#	Peak name	ax/min (mim)	Area	Area %	Assay %
0		22.177	4579.48	50.87	0.00
1		27.333	4422.92	49.13	0.00

#	Peak name	ax/min (mim)	Area	Area %	Assay %
0		22.714	4752.32	91.30	0.00
1		27.899	452.94	8.70	0.00

HPLC conditions for **3q** : t_R 22.18 min (major ***R***-iosmer), 27.33 min (***S***-isomer) (Chiralpak AD-H, hexane/*i*-PrOH, 92/8, 1.0 ml/min, λ = 254 nm) for 83% ee.

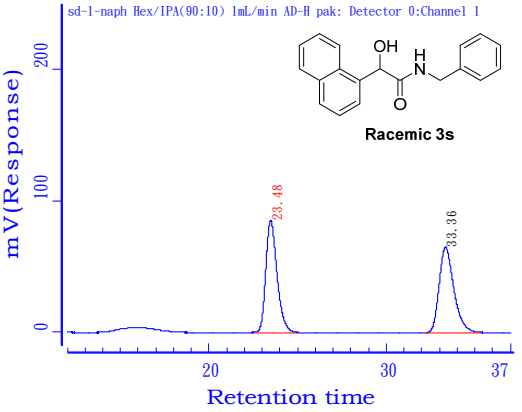


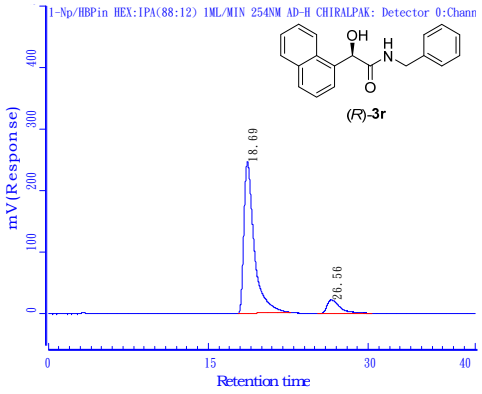


#	Peak name	max/min (mim)	Area	Area %	Assay %
0		24.568	1821.72	50.12	0.00
1		28.224	1813.33	49.88	0.00

#	Peak name	ax/min (mim)	Area	Area %	Assay %
0		17.653	8145.64	90.50	0.00
1		28.871	855.09	9.50	0.00

HPLC conditions for **3r** : t_R 24.57 min (major ***R***-iosmer), 28.22 min (***S***-isomer) (Chiralpak AD-H, hexane/*i*-PrOH, 92/8, 1.0 ml/min, λ = 254 nm) for 81% ee.

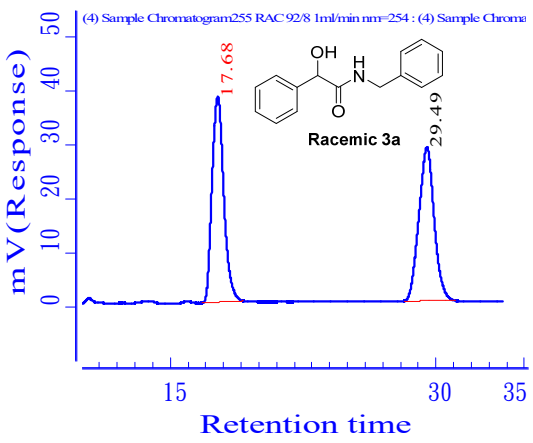


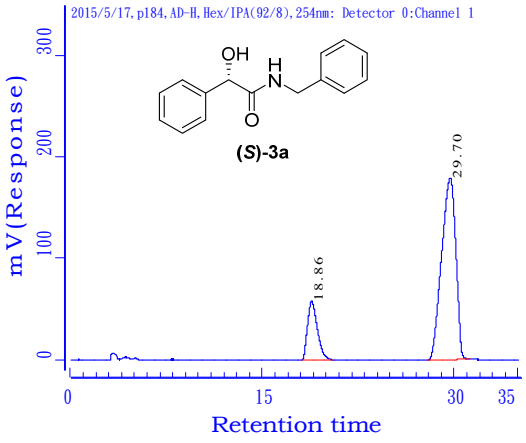


#	Peak name	ax/min (mim)	Area	Area %	Assay %
0		23.483	3753.35	49.97	0.00
1		33.357	3757.27	50.03	0.00

#	Peak name	ax/min (mim)	Area	Area %	Assay %
0		18.686	16666.70	89.57	0.00
1		26.557	1941.26	10.43	0.00

HPLC conditions for **3s** : t_R 23.48 min (major *R*-isomer), 33.36 min (*S*-isomer) (Chiralpak AD-H, hexane/*i*-PrOH, 90/10, 1.0 mL/min, λ = 254 nm) for 79% ee.

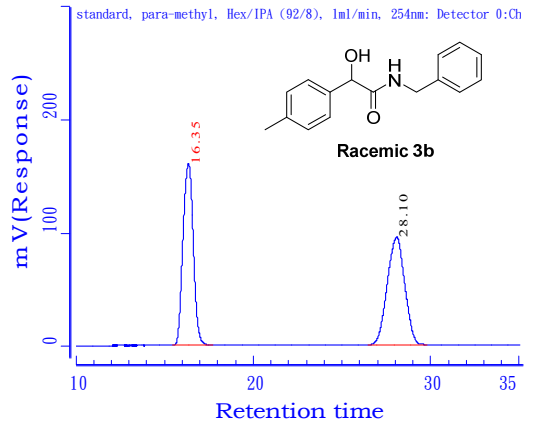


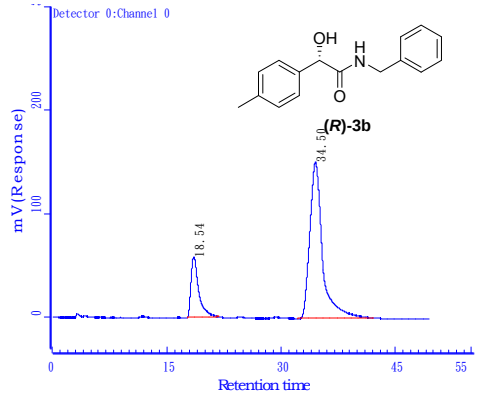


#	Peak name	x/min (mim)	Area	Area %	Assay %
0		17.682	1761.21	50.72	0.00
1		29.485	1711.11	49.28	0.00

#	Peak name	x/min (mim)	Area	Area %	Assay %
0		18.859	3097.18	18.20	0.00
1		29.701	13919.38	81.80	0.00

HPLC conditions for **3a** : t_R 17.68 min (***R***-iosmer), 29.49 min (major ***S***-isomer) (Chiralpak AD-H, hexane/*i*-PrOH, 92/8, 1.0 ml/min, λ = 254 nm) for 64% ee.

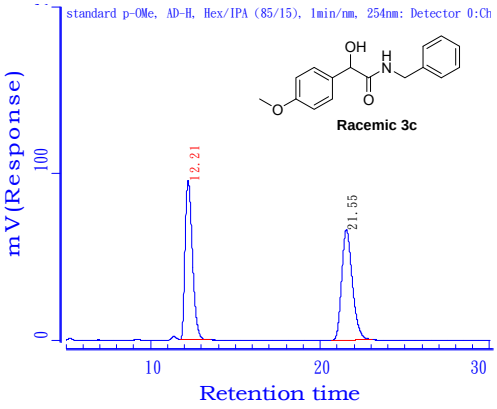




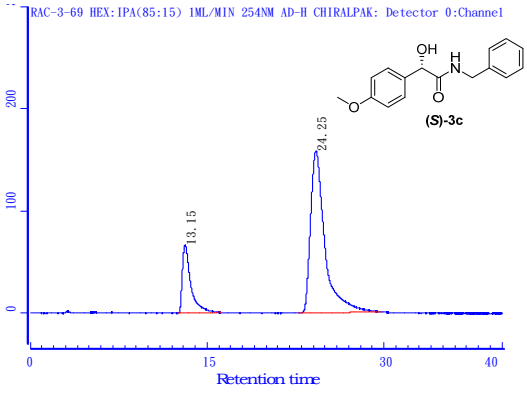
#	Peak name	max/min (mim)	Area	Area %	Assay %
0		16.354	6491.10	49.87	0.00
1		28.096	6524.62	50.13	0.00

#	Peak name	ax/min (mim)	Area	Area %	Assay %
0		18.536	3997.59	18.70	0.00
1		34.504	17377.08	81.30	0.00

HPLC conditions for **3b** : t_R 16.35 min (***R***-iosmer), 28.10 min (major ***S***-isomer) (Chiralpak AD-H, hexane/*i*-PrOH, 92/8, 1.0 ml/min, λ = 254 nm) for 63% ee.

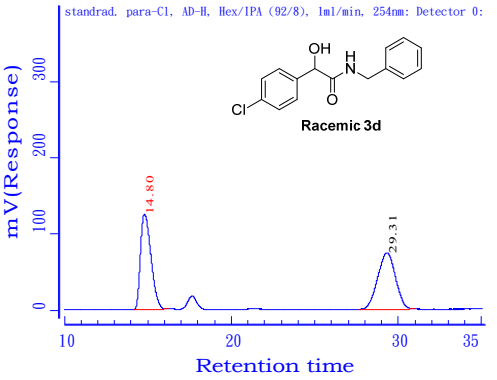


#	Peak name	max/min (mim)	Area	Area %	Assay %
0		12.212	2869.52	49.68	0.00
1		21.554	2906.32	50.32	0.00

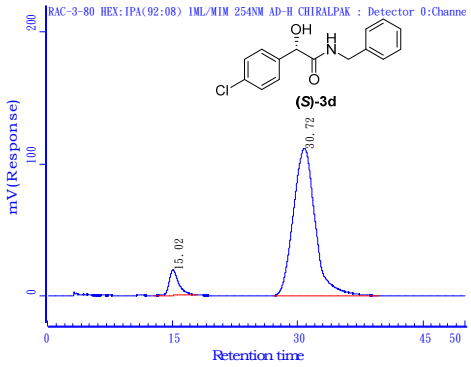


Peak name	ax/min (mim)	Area	Area %	Assay %
	13.148	3302.90	19.78	0.00
	24.253	13398.16	80.22	0.00

HPLC conditions for **3c** : t_R 12.21 min (***R***-iosmer), 21.55 min (major ***S***-isomer) (Chiralpak AD-H, hexane/*i*-PrOH, 85/15, 1.0 ml/min, λ = 254 nm) for 60% ee.

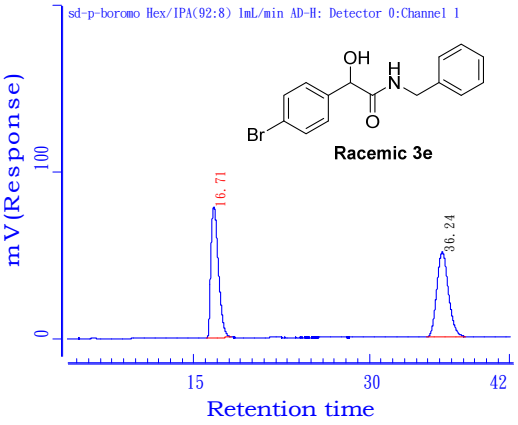


#	Peak name	max/min (mim)	Area	Area %	Assay %
0		14.800	5462.06	49.93	0.00
1		29.312	5478.30	50.07	0.00

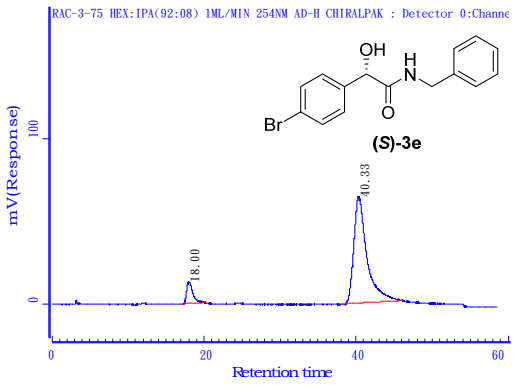


#	Peak name	ax/min (mim)	Area	Area %	Assay %
0		15.023	1547.37	7.05	0.00
1		30.724	20394.67	92.95	0.00

HPLC conditions for **3d** : t_R 14.30 min (***R***-iosmer), 29.31 min (major ***S***-isomer) (Chiralpak AD-H, hexane/*i*-PrOH, 92/8, 1.0 ml/min, λ = 254 nm) for 86% ee.

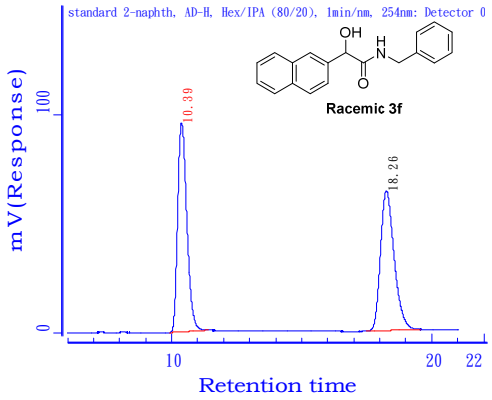


#	Peak name	x/min (mim)	Area	Area %	Assay %
0		16.712	3511.85	49.93	0.00
1		36.236	3522.01	50.07	0.00

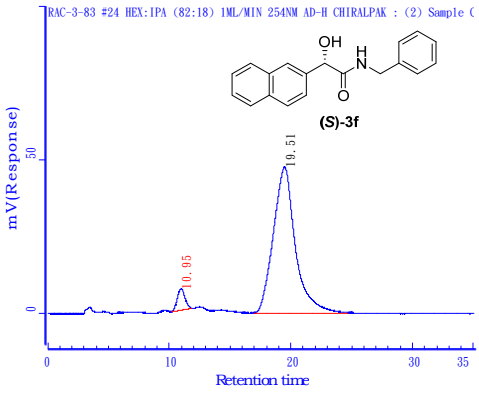


#	Peak name	ax/min (mim)	Area	Area %	Assay %
0		18.005	713.61	8.53	0.00
1		40.333	7648.40	91.47	0.00

HPLC conditions for **3e** : t_R 16.71 min (***R***-iosmer), 36.24 min (major ***S***-isomer) (Chiralpak AD-H, hexane/*i*-PrOH, 92/8, 1.0 ml/min, λ = 254 nm) for 83% ee.

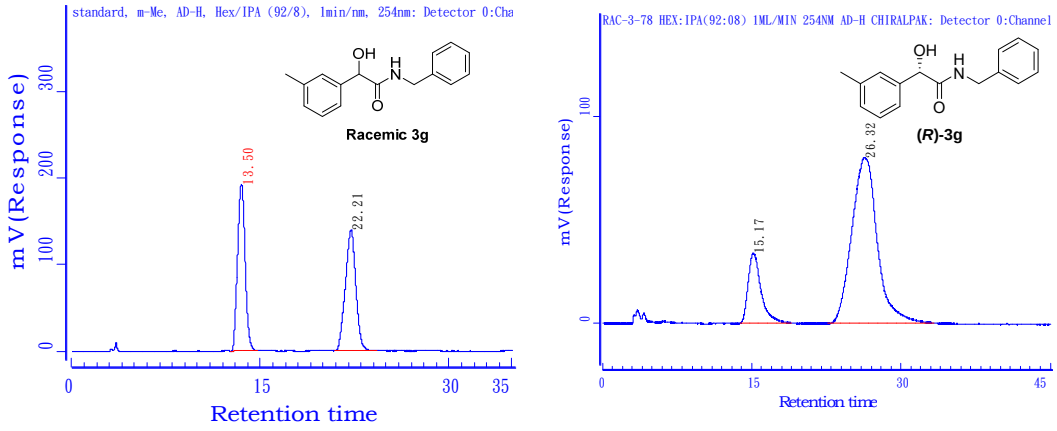


#	Peak name	max/min (mim)	Area	Area %	Assay %
0		10.388	2339.20	50.02	0.00
1		18.259	2337.73	49.98	0.00



#	Peak name	ax/min (mim)	Area	Area %	Assay %
0		10.948	309.20	4.97	0.00
1		19.513	5914.63	95.03	0.00

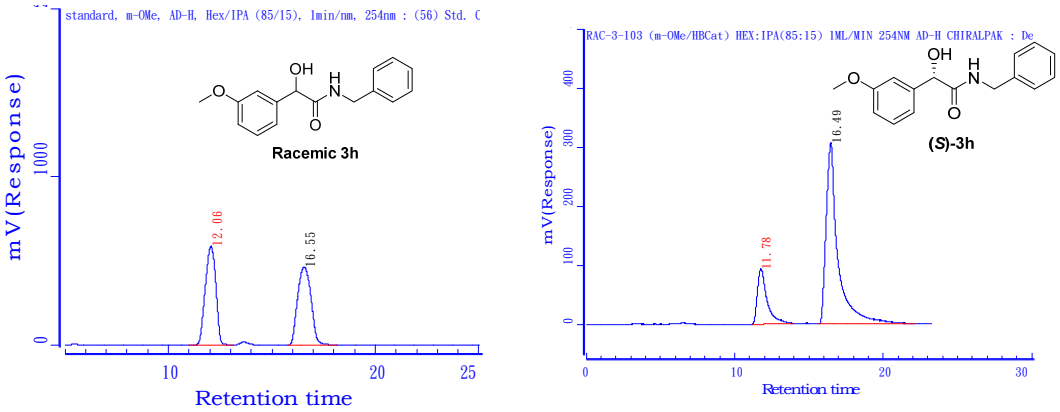
HPLC conditions for **3f** : t_R 10.39 min (***R***-iosmer), 18.26 min (major ***S***-isomer) (Chiralpak AD-H, hexane/*i*-PrOH, 80/20, 1.0 ml/min, λ = 254 nm) for 90% ee.



#	Peak name	max/min (mim)	Area	Area %	Assay %
0		13.501	8072.94	49.88	0.00
1		22.210	8112.04	50.12	0.00

#	Peak name	ax/min (mim)	Area	Area %	Assay %
0		15.174	3087.54	17.68	0.00
1		26.317	14375.21	82.32	0.00

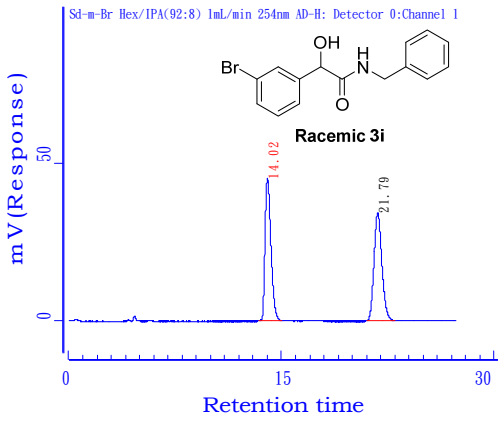
HPLC conditions for **3g** : t_R 13.50 min (***R***-iosmer), 22.21 min (major ***S***-isomer) (Chiralpak AD-H, hexane/*i*-PrOH, 92/8, 1.0 ml/min, λ = 254 nm) for 65% ee.

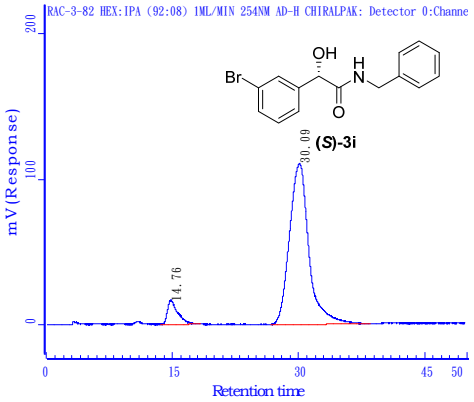


#	Peak name	max/min (mim)	Area	Area %	Assay %
0		12.058	20649.21	49.89	0.00
1		16.553	20737.65	50.11	0.00

#	Peak name	ax/min (mim)	Area	Area %	Assay %
0		11.783	4209.42	19.85	0.00
1		16.487	16999.30	80.15	0.00

HPLC conditions for **3h** : t_R 12.06 min (***R***-iosmer), 16.55 min (major ***S***-isomer) (Chiralpak AD-H, hexane/*i*-PrOH, 85/15, 1.0 ml/min, λ = 254 nm) for 60% ee (***S***).

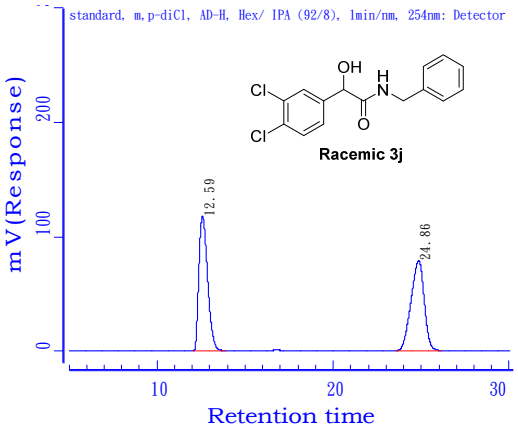


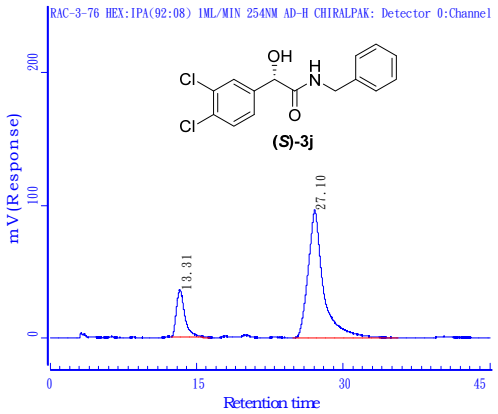


#	Peak name	ax/min (mim)	Area	Area %	Assay %
0		14.022	1308.88	49.52	0.00
1		21.789	1334.19	50.48	0.00

#	Peak name	ax/min (mim)	Area	Area %	Assay %
0		14.759	1422.15	7.40	0.00
1		30.090	17803.85	92.60	0.00

HPLC conditions for **3i** : t_R 14.02 min (***R***-iosmer), 21.79 min (major ***S***-isomer) (Chiralpak AD-H, hexane/*i*-PrOH, 92/8, 1.0 ml/min, λ = 254 nm) for 85% ee (***R***).

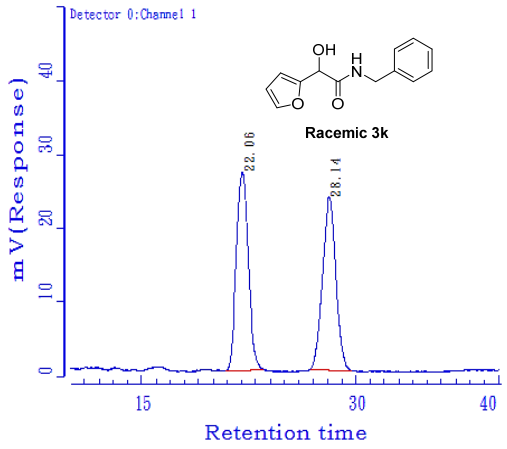




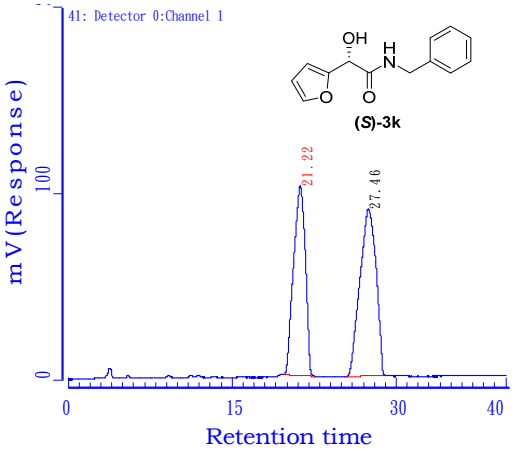
#	Peak name	max/min (mim)	Area	Area %	Assay %
0		12.586	4074.42	49.80	0.00
1		24.858	4106.63	50.20	0.00

#	Peak name	ax/min (mim)	Area	Area %	Assay %
0		13.308	2156.12	17.05	0.00
1		27.101	10488.94	82.95	0.00

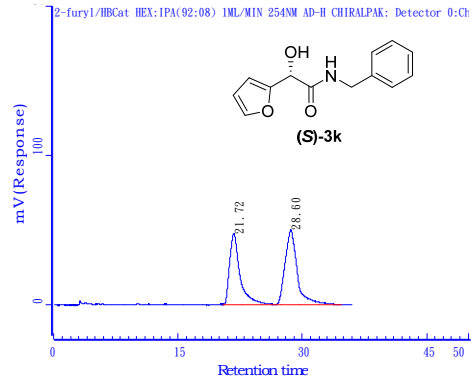
HPLC conditions for **3j** : t_R 12.59 min (***R***-iosmer), 24.86 min (major ***S***-isomer) (Chiralpak AD-H, hexane/*i*-PrOH, 92/8, 1.0 ml/min, λ = 254 nm) for 66% ee.



#	Peak name	x/min (mim)	Area	Area %	Assay %
0		22.065	1560.10	50.33	0.00
1		28.144	1539.55	49.67	0.00

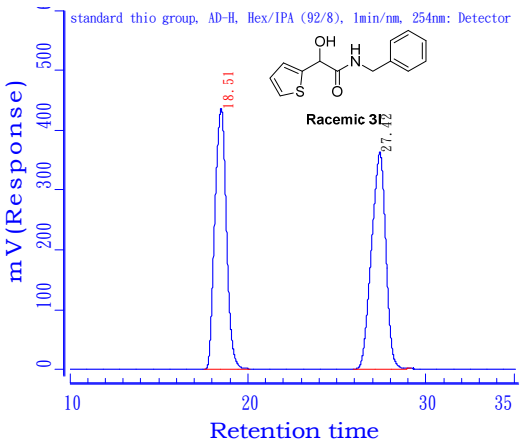


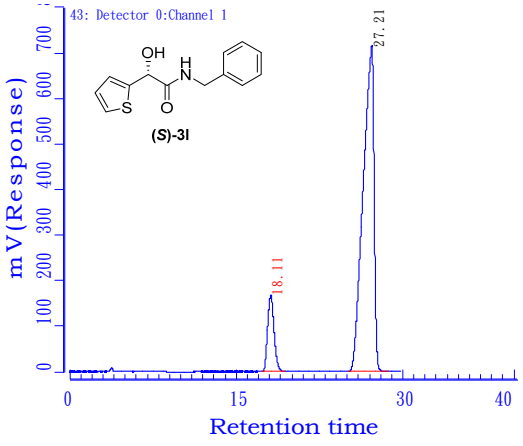
#	Peak name	ax/min (mim)	Area	Area %	Assay %
0		21.221	7651.67	45.28	0.00
1		27.455	9247.44	54.72	0.00



#	Peak name	ax/min (mim)	Area	Area %	Assay %
0		21.723	4076.55	44.75	0.00
1		28.603	5032.30	55.25	0.00

HPLC conditions for **3k** : t_R 22.06 min (***R***-iosmer), 28.14 min (major ***S***-isomer) (Chiralpak AD-H, hexane/*i*-PrOH, 92/8, 1.0 ml/min, λ = 254 nm) for 10% ee (*S*) and 11%ee (*S*).

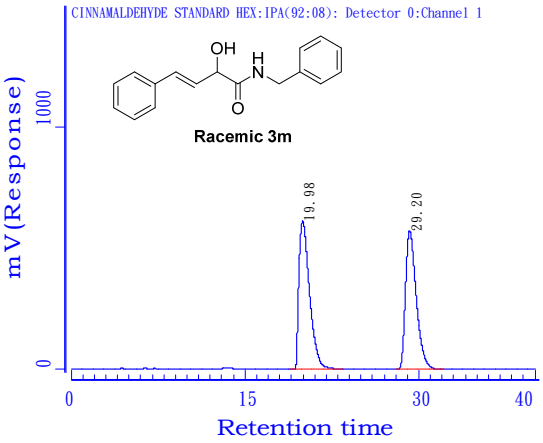


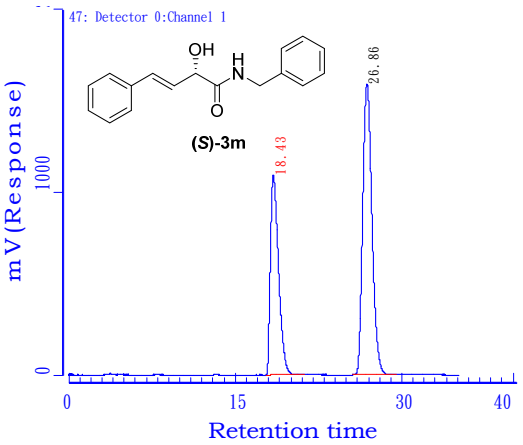


#	Peak name	max/min (mim)	Area	Area %	Assay %
0		18.508	19638.88	49.29	0.00
1		27.419	20202.77	50.71	0.00

#	Peak name	x/min (mim)	Area	Area %	Assay %
0		18.114	6808.47	12.29	0.00
1		27.208	48576.18	87.71	0.00

HPLC conditions for **3n** : t_R 18.51 min (***R***-iosmer), 27.42 min (major ***S***-isomer) (Chiralpak AD-H, hexane/*i*-PrOH, 92/8, 1.0 ml/min, λ = 254 nm) for 75% ee.

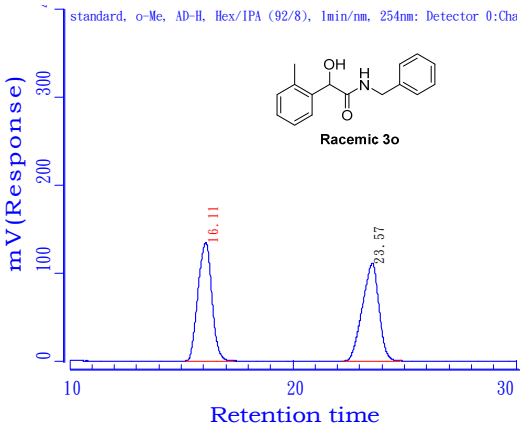




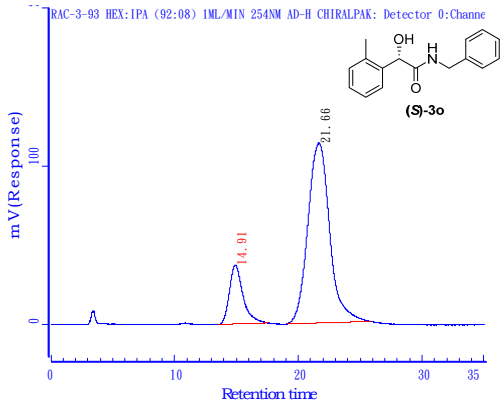
#	Peak name	x/min (mim)	Area	Area %	Assay %
0		19.984	39344.14	51.41	0.00
1		29.205	37185.57	48.59	0.00

#	Peak name	x/min (mim)	Area	Area %	Assay %
0		18.433	55928.74	37.74	0.00
1		26.856	92266.18	62.26	0.00

HPLC conditions for **3m** : t_R 19.98 min (***R***-iosmer), 29.20 min (major ***S***-isomer) (Chiralpak AD-H, hexane/*i*-PrOH, 92/8, 1.0 ml/min, λ = 254 nm) for 25% ee.

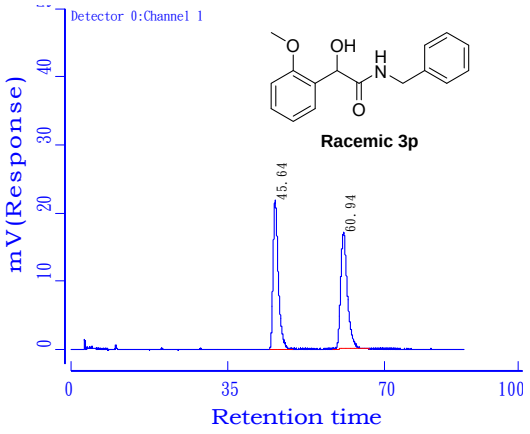


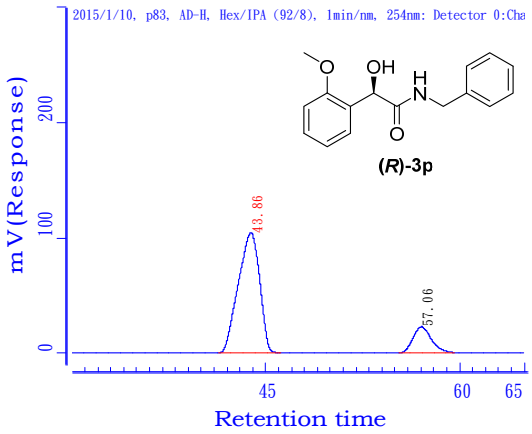
#	Peak name	max/min (mim)	Area	Area %	Assay %
0		16.110	5815.39	50.37	0.00
1		23.570	5731.04	49.63	0.00



#	Peak name	ax/min (mim)	Area	Area %	Assay %
0		14.906	2833.79	16.94	0.00
1		21.664	13892.88	83.06	0.00

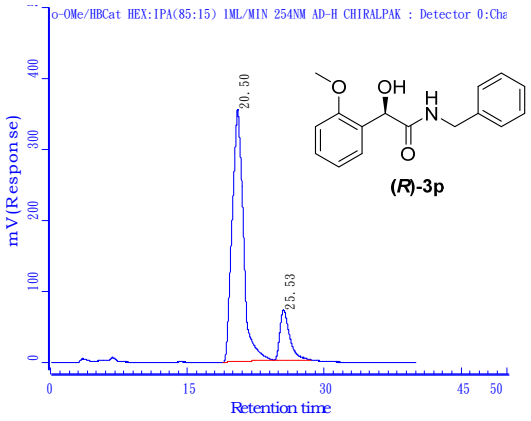
HPLC conditions for **3o** : t_R 16.11 min (***R***-iosmer), 23.57 min (major ***S***-isomer) (Chiralpak AD-H, hexane/*i*-PrOH, 92/8, 1.0 ml/min, λ = 254 nm) for 66% ee.





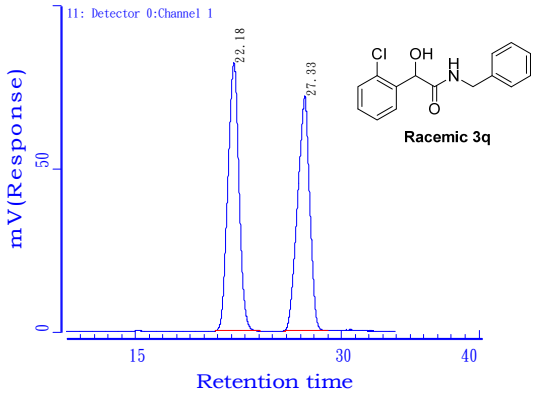
#	Peak name	ax/min (mim)	Area	Area %	Assay %
0		45.639	1758.72	50.01	0.00
1		60.939	1758.36	49.99	0.00

#	Peak name	max/min (mim)	Area	Area %	Assay %
0		43.862	12017.23	84.35	0.00
1		57.057	2229.99	15.65	0.00

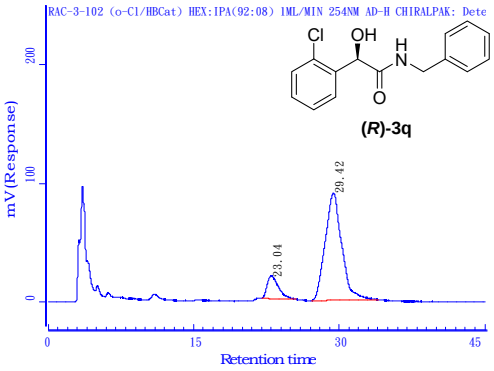


#	Peak name	ax/min (mim)	Area	Area %	Assay %
0		20.503	30523.28	85.43	0.00
1		25.534	5206.17	14.57	0.00

HPLC conditions for **3p** : t_R 43.86 min (major ***R***-iosmer), 57.06 min (***S***-isomer) (Chiralpak AD-H, hexane/*i*-PrOH, 92/8, 1.0 ml/min, λ = 254 nm) for 69% ee (*R*)and 71%ee (*R*).

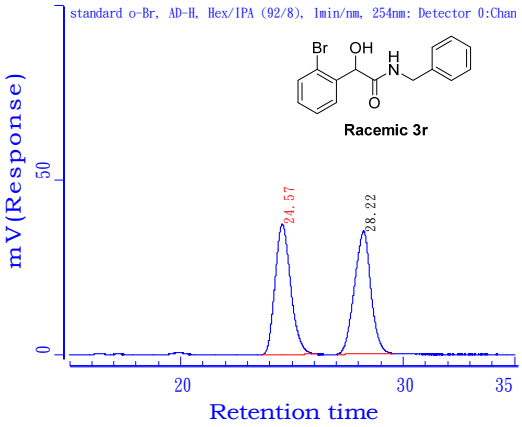


#	Peak name	ax/min (mim)	Area	Area %	Assay %
0		22.177	4579.48	50.87	0.00
1		27.333	4422.92	49.13	0.00

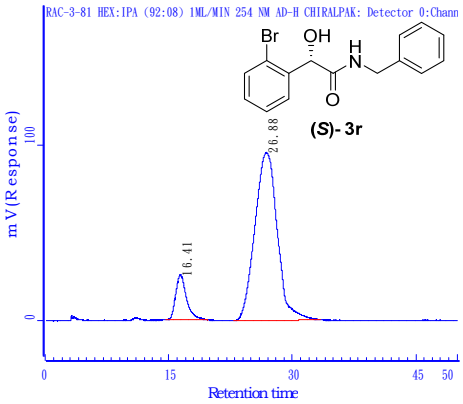


#	Peak name	ax/min (mim)	Area	Area %	Assay %
0		23.044	1568.23	12.76	0.00
1		29.423	10721.67	87.24	0.00

HPLC conditions for **3q** : t_R 22.18 min (***R***-iosmer), 27.33 min (major ***S***-isomer) (Chiralpak AD-H, hexane/*i*-PrOH, 92/8, 1.0 ml/min, λ = 254 nm) for 75% ee.

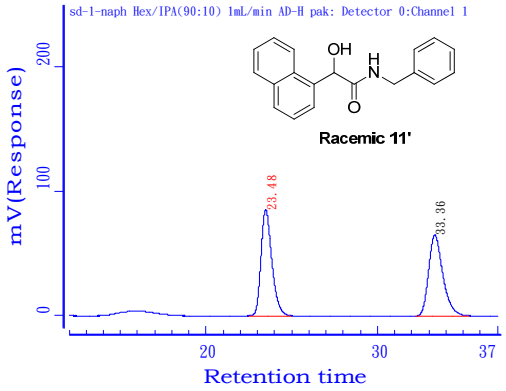


#	Peak name	max/min (mim)	Area	Area %	Assay %
0		24.568	1821.72	50.12	0.00
1		28.224	1813.33	49.88	0.00

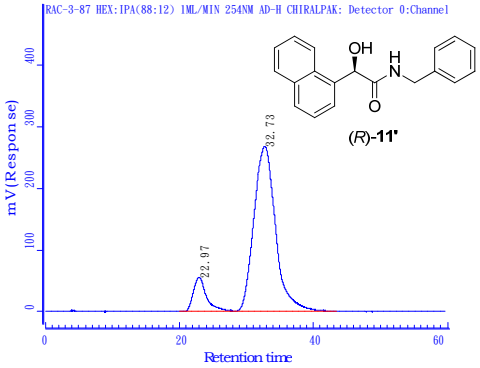


#	Peak name	ax/min (mim)	Area	Area %	Assay %
0		16.407	2336.90	11.42	0.00
1		26.876	18130.53	88.58	0.00

HPLC conditions for **3r** : t_R 24.57 min (***R***-iosmer), 28.22 min (major ***S***-isomer) (Chiralpak AD-H, hexane/*i*-PrOH, 92/8, 1.0 ml/min, λ = 254 nm) for 77% ee.



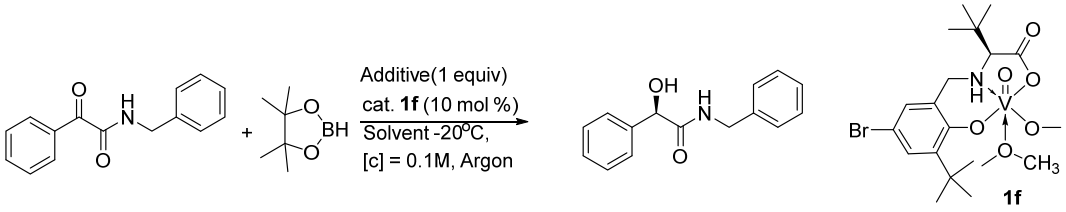
#	Peak name	ax/min (mim)	Area	Area %	Assay %
0		23.483	3753.35	49.97	0.00
1		33.357	3757.27	50.03	0.00



#	Peak name	ax/min (mim)	Area	Area %	Assay %
0		22.971	7474.71	11.09	0.00
1		32.734	59912.84	88.91	0.00

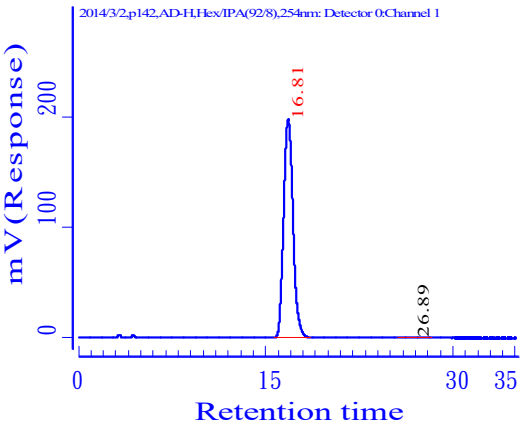
HPLC conditions for **11'** : t_R 23.48 min (*R*-isomer), 33.36 min (major *S*-isomer) (Chiralpak AD-H, hexane/*i*-PrOH, 90/10, 1.0 mL/min, λ = 254 nm) for 78% ee.

Table S1



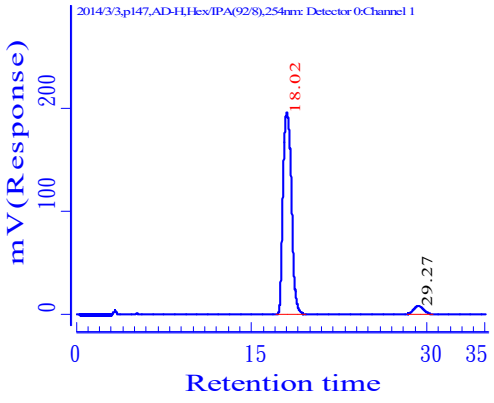
Entry	Reducing Agent	Additive	Time (h)	Yield (%)	Ee (%)
1	PinB–H	—	96	65	88 (*R*)
2	PinB–H	MeOH	96	40	99 (*R*)
3	PinB–H	CCl_3CH_2OH	96	47	91 (*R*)
4	PinB–H	CF_3CH_2OH	96	22	96 (*R*)
5	CatB–H	—	14	99	64 (*S*)
6	CatB–H	MeOH	14	92	47 (*S*)
7	CatB–H	CCl_3CH_2OH	14	86	58 (*S*)
8	CatB–H	CF_3CH_2OH	14	84	62 (*S*)

Entry 2 Table S1



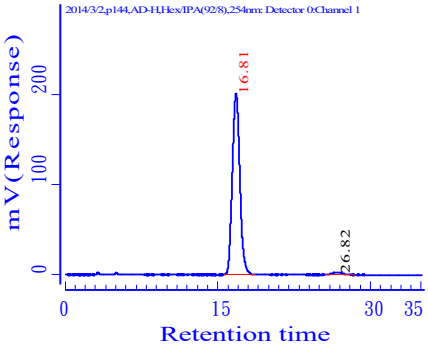
#	Peak name	x/min (mim)	Area	Area %	Assay %
0		16.805	10363.80	99.87	0.00
1		26.893	13.15	0.13	0.00

Entry 3 Table S1



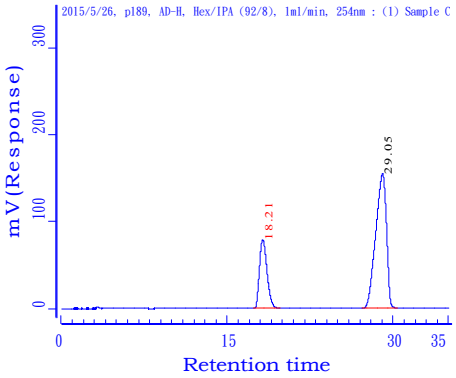
#	Peak name	x/min (mim)	Area	Area %	Assay %
0		18.025	9871.17	95.53	0.00
1		29.275	461.69	4.47	0.00

Entry 4 Table S1



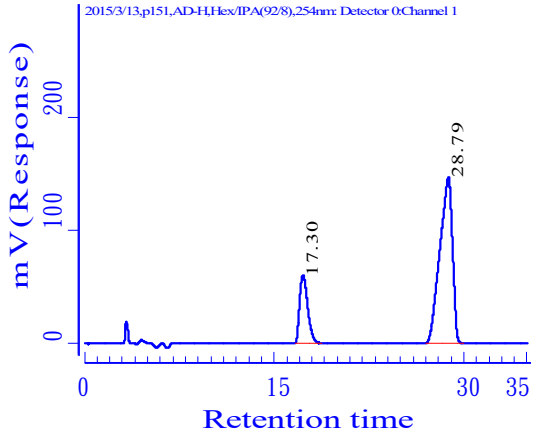
#	Peak name	x/min (mim)	Area	Area %	Assay %
0		16.809	10396.77	98.46	0.00
1		26.821	162.78	1.54	0.00

Entry 6 Table S1



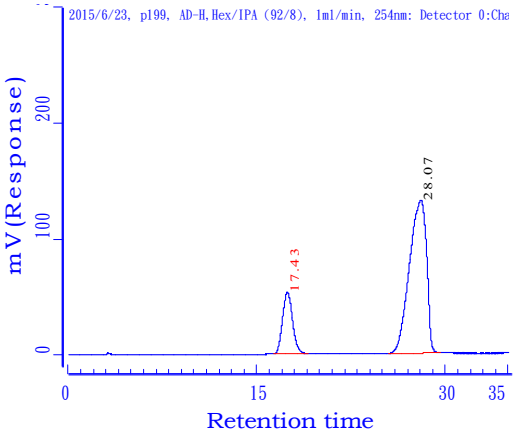
#	Peak name	max/min (mim)	面積	Area %	Assay %
0		18.208	3746.65	26.24	0.00
1		29.045	10529.06	73.76	0.00

Entry 7 Table S1



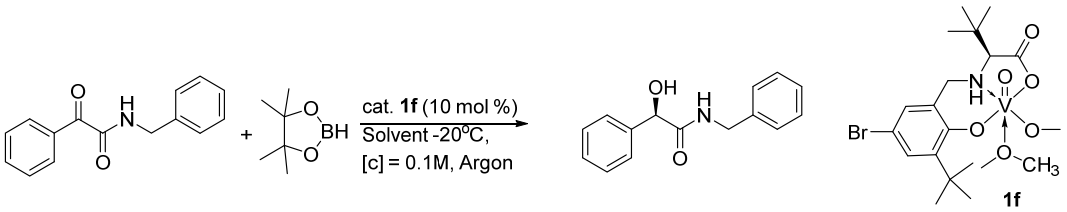
#	Peak name	x/min (mim)	Area	Area %	Assay %
0		17.300	2711.52	21.06	0.00
1		28.789	10163.16	78.94	0.00

Entry 8 Table S1



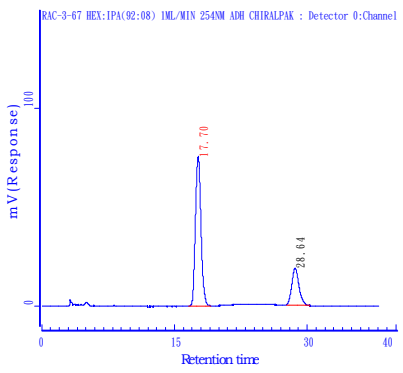
#	Peak name	max/min (mim)	面積	Area %	Assay %
0		17.429	2950.49	19.02	0.00
1		28.065	12558.30	80.98	0.00

Table S2



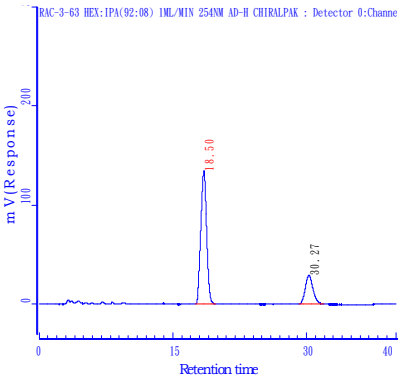
entry	Solvent	Time(h)	Yield(%)	ee%
1 (final)	toluene	24+24+48	65	88 (*R*)
2	MeOH	24+24+48	35	52 (*R*)
3	*i*-PrOH	24+24+48	43	56 (*R*)
4	1,4-dioxane	24+24+48	21	36 (*R*)
5	CH_3CN	24+24+48	28	38 (*R*)
6	DMSO (r.t.)	24+24+48	52	12 (*R*)
7	DMF	24+24+48	19	24 (*R*)

Entry 2 Table S2



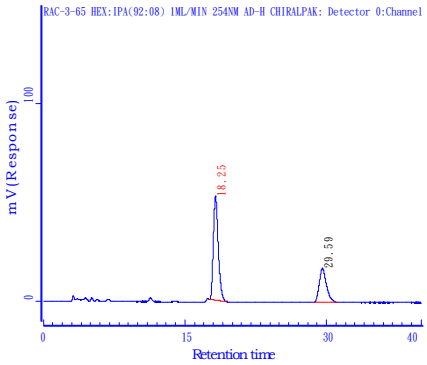
#	Peak name	x/min (mim)	Area	Area %	Assay %
0		17.696	3321.98	75.88	0.00
1		28.641	1055.82	24.12	0.00

Entry 3 Table S2



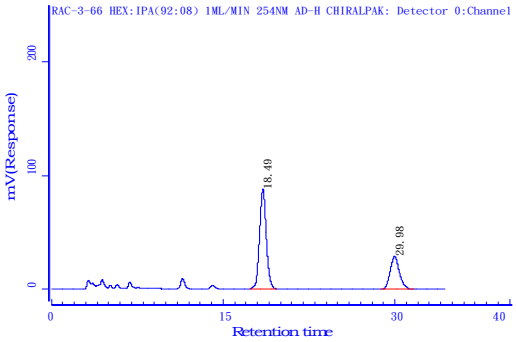
#	Peak name	x/min (mim)	Area	Area %	Assay %
0		18.502	6143.29	78.02	0.00
1		30.272	1730.47	21.98	0.00

Entry 4 Table S2



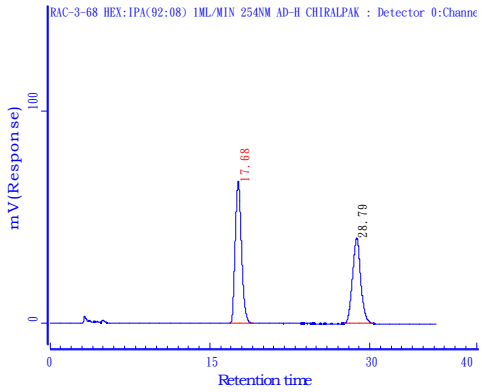
#	Peak name	x/min (mim)	Area	Area %	Assay %
0		18.252	1895.46	67.85	0.00
1		29.587	898.09	32.15	0.00

Entry 5 Table S2



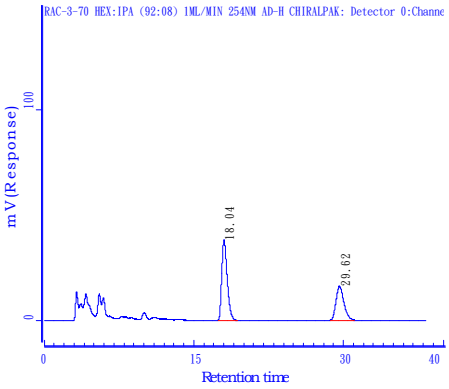
#	Peak name	x/min (mim)	Area	Area %	Assay %
0		18.489	3596.96	68.99	0.00
1		29.983	1616.70	31.01	0.00

Entry 6 Table S2



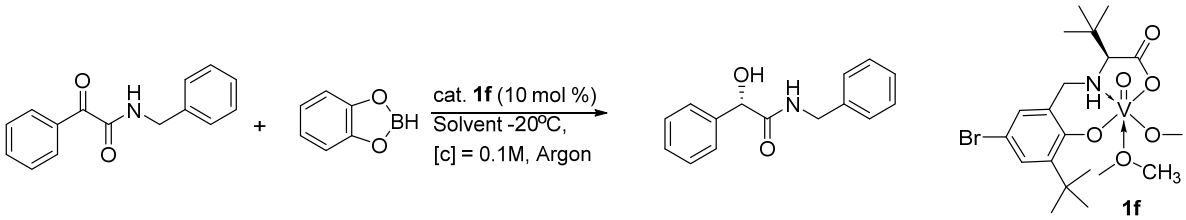
#	Peak name	x/min (mim)	Area	Area %	Assay %
0		17.683	2750.27	55.82	0.00
1		28.792	2176.45	44.18	0.00

Entry 7 Table S2



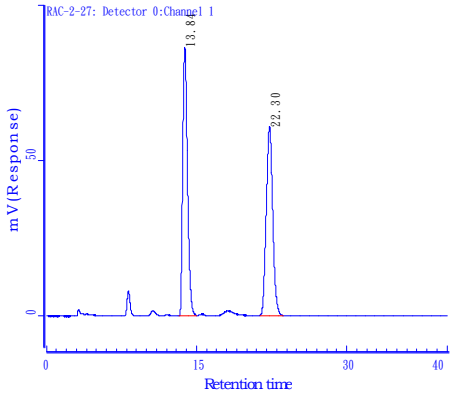
#	Peak name	x/min (mim)	Area	Area %	Assay %
0		18.044	1441.29	62.20	0.00
1		29.617	876.03	37.80	0.00

Table S3



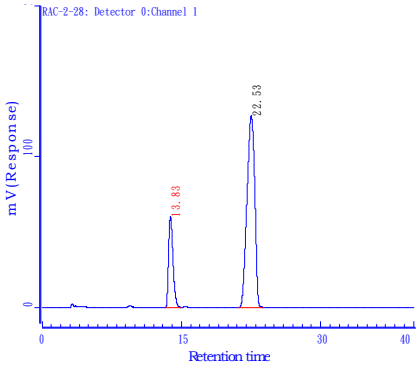
entry	Solvent	Time(h)	Yield(%)	ee%
1 (final)	toluene	12	99	64 (*S*)
2	CH_2Cl_2	12	97	2 (*R*)
3	PhCl	14	95	57 (*S*)
4	THF	12	98	35 (*S*)
5	*t*-BuOMe	15	87	4 (*R*)

Entry 2 Table S3



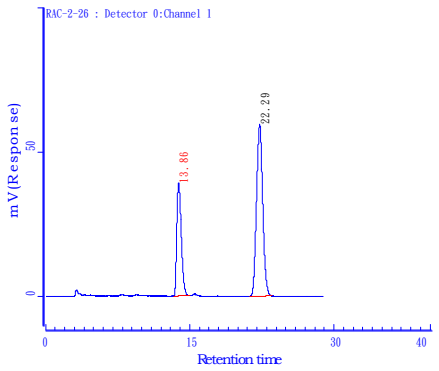
#	Peak name	x/min (mim)	Area	Area %	Assay %
0		13.839	2849.17	51.18	0.00
1		22.305	2717.27	48.82	0.00

Entry 3 Table S3



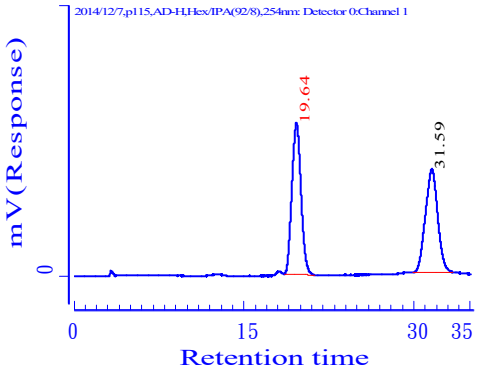
#	Peak name	x/min (mim)	Area	Area %	Assay %
0		13.834	2003.39	21.34	0.00
1		22.534	7384.36	78.66	0.00

Entry 4 Table S3



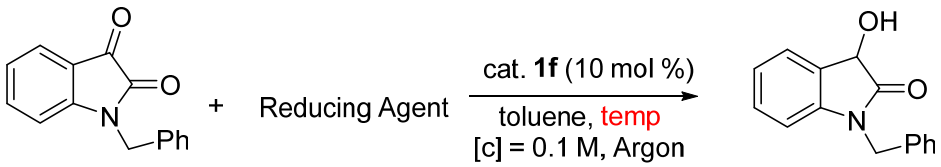
#	Peak name	x/min (mim)	Area	Area %	Assay %
0		13.860	1277.85	32.42	0.00
1		22.289	2663.32	67.58	0.00

Entry 5 Table S3



#	Peak name	x/min (mim)	Area	Area %	Assay %
0		19.642	3133.66	52.16	0.00
1		31.586	2873.65	47.84	0.00

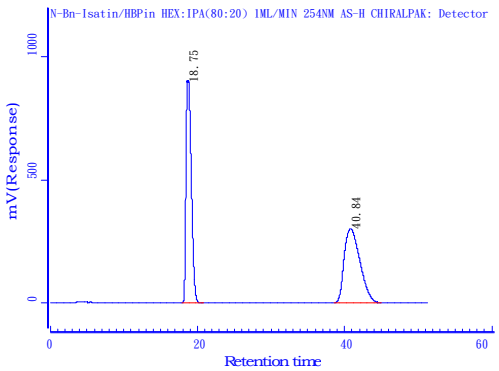
Table S4



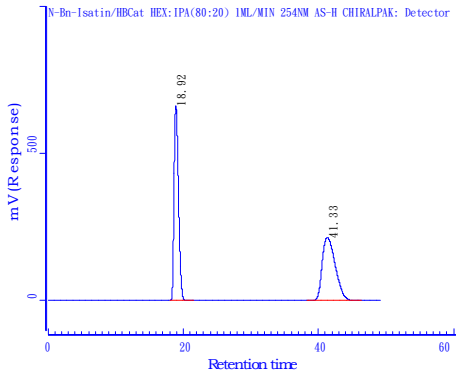
mw: 237.26

mw: 239.27

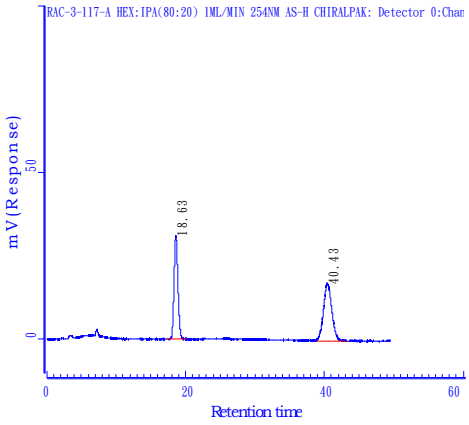
reducing agent	temperature, °C	Yield, %	ee, %
HBPin	−20/-40	99/0	0/---
HBCat	−20/-40	92/28	2/8 (*S*)



#	Peak name	ax/min (mim)	Area	Area %	Assay %
0		18.753	45594.00	50.36	0.00
1		40.842	44942.66	49.64	0.00

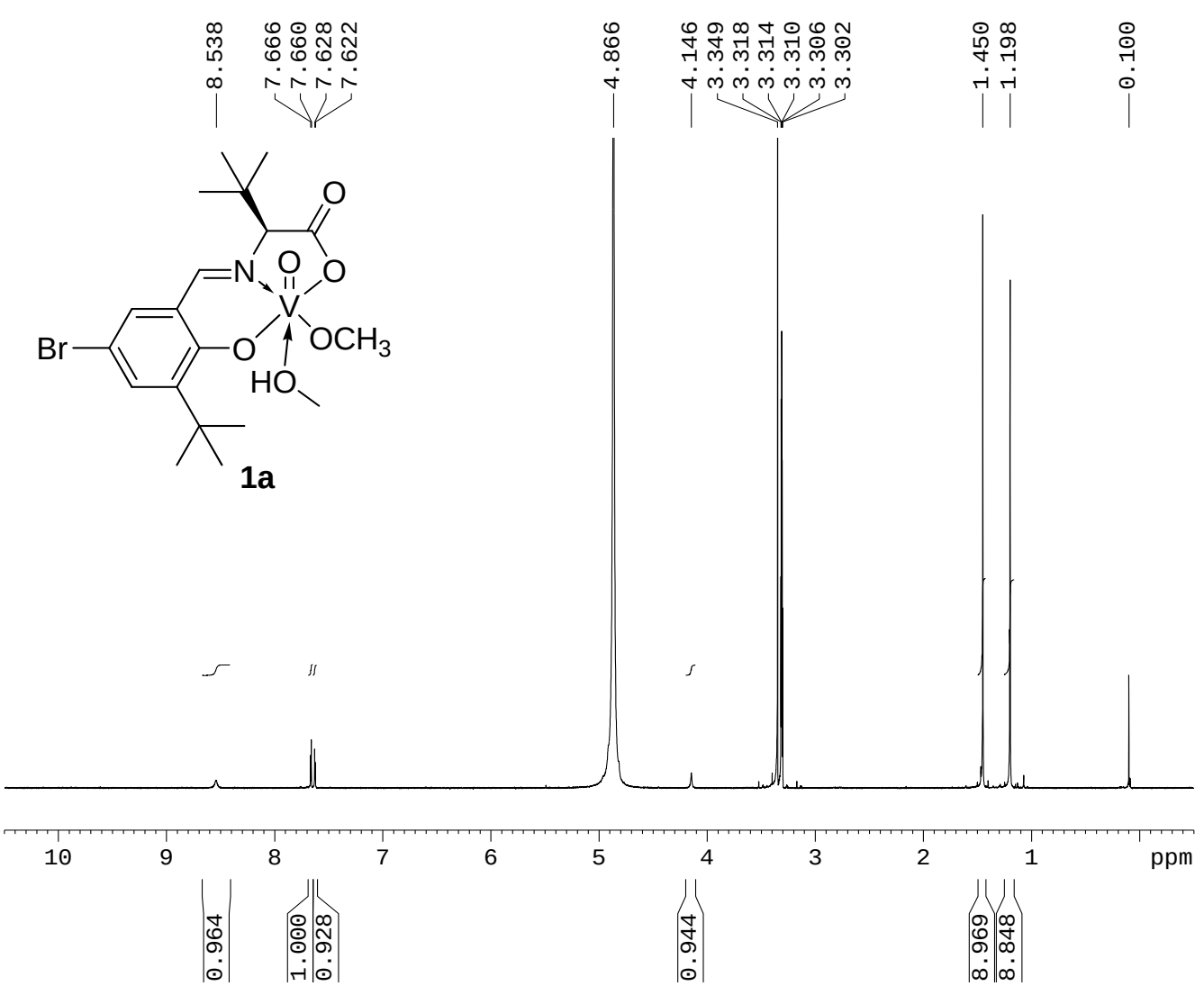


#	Peak name	ax/min (mim)	Area	Area %	Assay %
0		18.917	30101.72	50.96	0.00
1		41.334	28965.59	49.04	0.00



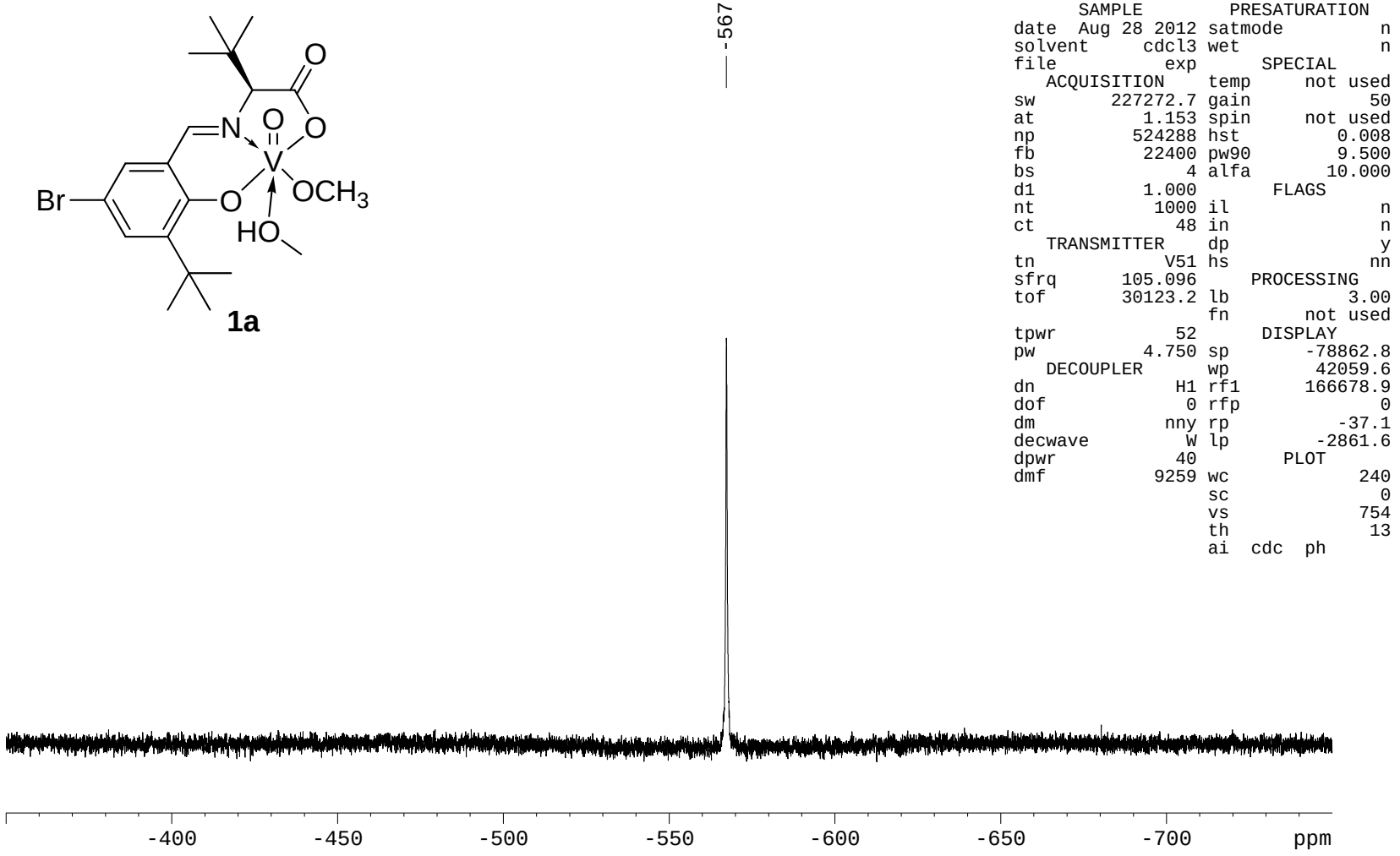
#	Peak name	ax/min (mim)	Area	Area %	Assay %
0		18.625	1223.93	46.08	0.00
1		40.425	1432.16	53.92	0.00

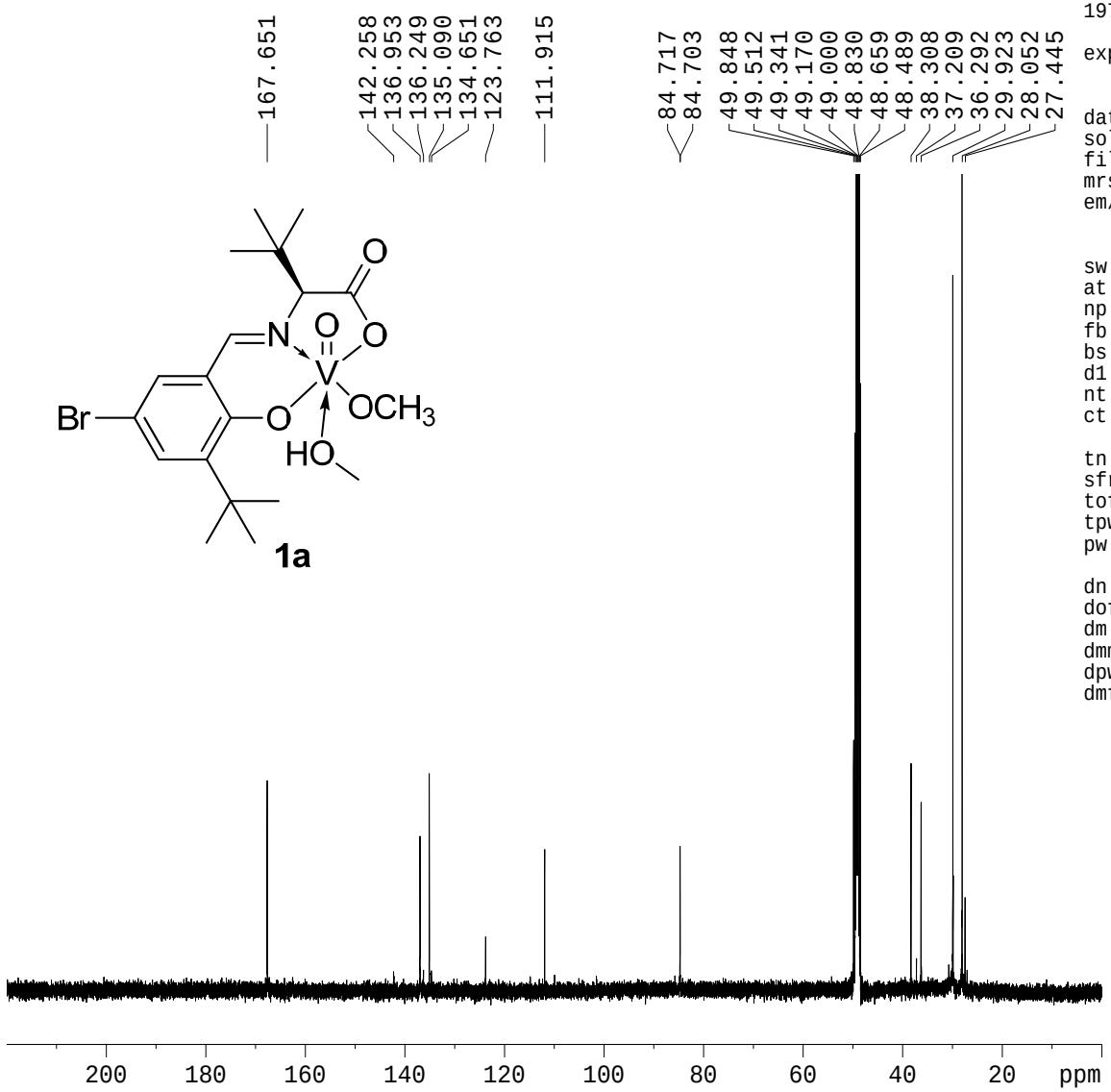
7.^{1}H and ^{13}C NMR spectra



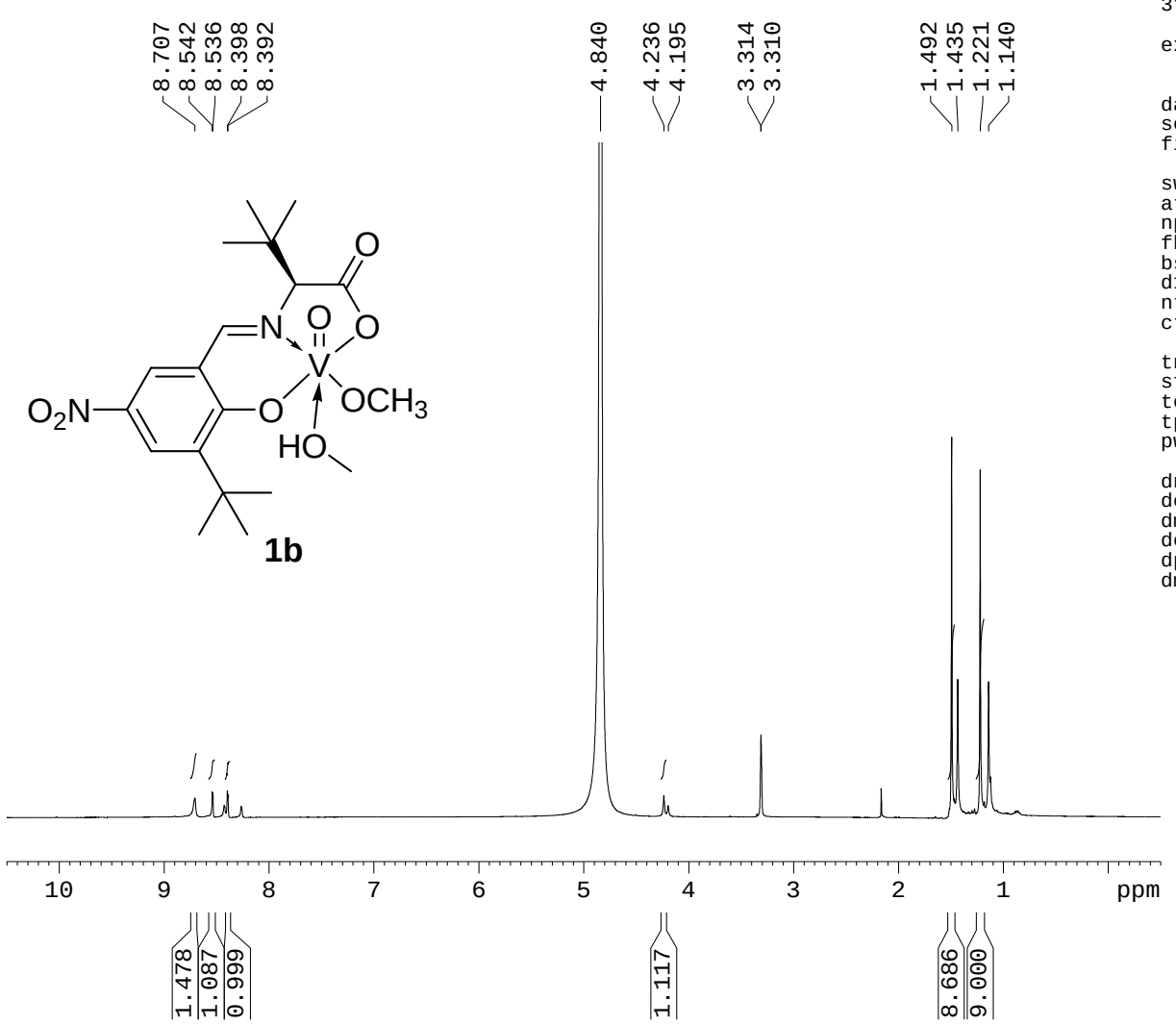
1a

```
NAME                 other
EXPNO                   31
PROCNO                   1
Date_             20120829
Time                 16.01
INSTRUM              spect
PROBHD   5 mm DUL 13C-1
PULPROG               zg30
TD                   32768
SOLVENT               MeOD
NS                       8
DS                       0
SWH               6410.256 Hz
FIDRES            0.195625 Hz
AQ                2.5559540 sec
RG                       4
DW                  78.000 usec
DE                   6.00 usec
TE                   300.0 K
D1              2.00000000 sec
TD0                      1

======== CHANNEL f1 ========
NUC1                    1H
P1                   10.00 usec
PL1                  -2.40 dB
SFO1           400.1528010 MHz
SI                   16384
SF             400.1500067 MHz
WDW                     EM
SSB                      0
LB                    0.00 Hz
GB                       0
PC                    1.00
```

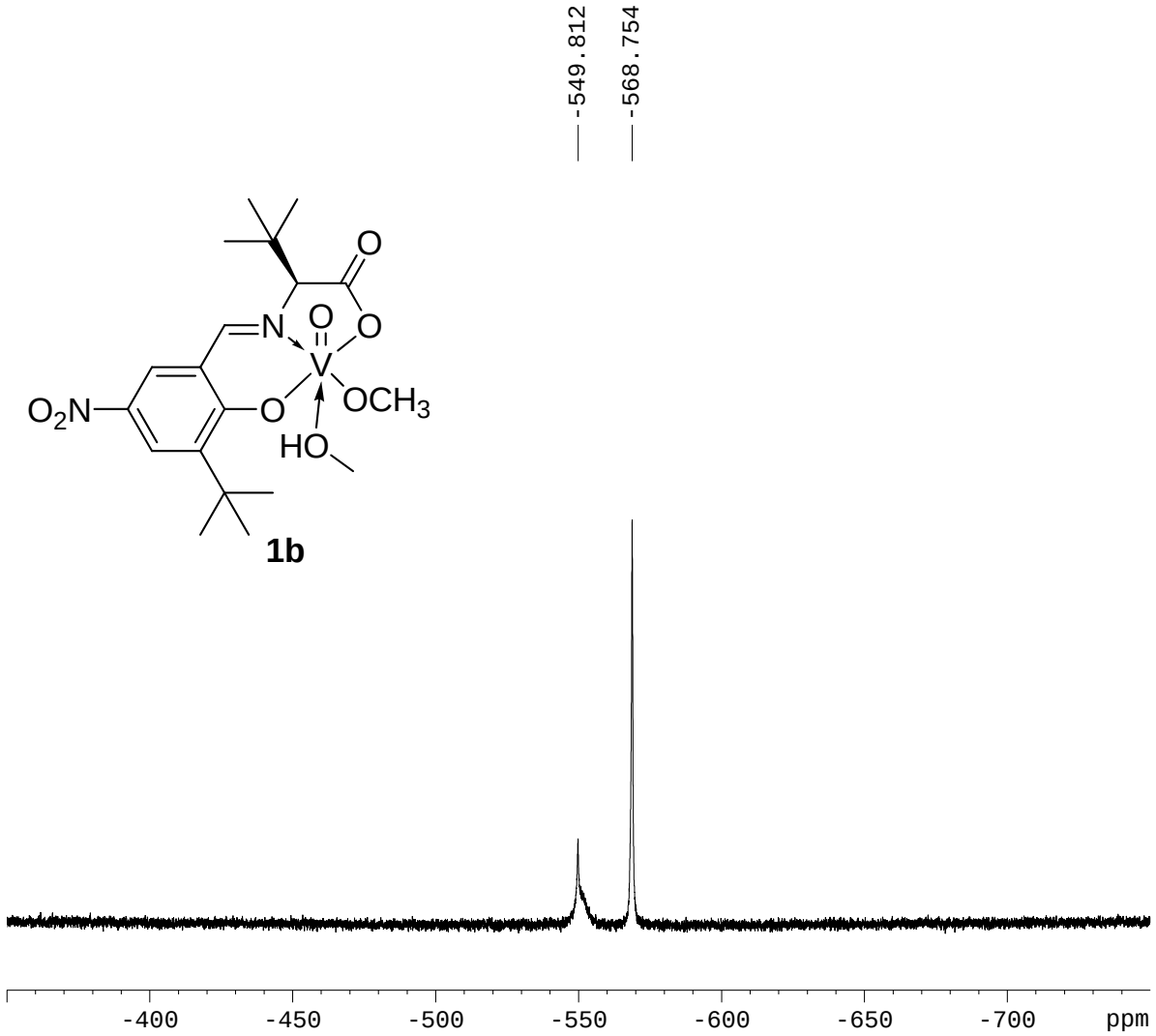

-567.268
3tBu-5Br
exp1 PHOSPHORUS
SAMPLE
date Aug 28 2012
solvent cdcl3
file exp
ACQUISITION
sw 227272.7
at 1.153
np 524288
fb 22400
bs 4
d1 1.000
nt 1000
ct 48
TRANSMITTER
tn V51
sfrq 105.096
tof 30123.2
tpwr 52
pw 4.750
DECOUPLER
dn H1
dof 0
dm nny
decwave W
dpwr 40
dmf 9259
PRESATURATION
satmode n
wet n
SPECIAL
temp not used
gain 50
spin not used
hst 0.008
pw90 9.500
alfa 10.000
FLAGS
il n
in n
dp y
hs nn
PROCESSING
lb 3.00
fn not used
DISPLAY
sp -78862.8
wp 42059.6
rf1 166678.9
rfp 0
rp -37.1
lp -2861.6
PLOT
wc 240
sc 0
vs 754
th 13
ai cdc ph
Br
O
N
O
O
V
O
OCH3
HO
1a
-400
-450
-500
-550
-600
-650
-700
ppm




```
197

exp33 Carbon

      SAMPLE              SPECIAL
date  Jun 16 2014   temp       not used
solvent     cd3od   gain       not used
file /home/oper/vn~ spin       not used
mrsys/data/NTHU_ch~ hst           0.008
em/CHEN/chen140616~ pw90         12.500
         .001C.fid alfa         10.000
   ACQUISITION             FLAGS
sw         36363.6   il                n
at           0.511   in                n
np           37172   dp                y
fb       not used    hs               nn
bs             16       PROCESSING
d1           2.000   lb             3.00
nt           30000   fn         not used
ct           22944         DISPLAY
   TRANSMITTER
tn             C13   sp              -0.9
sfrq       125.692   wp           27647.8
tof         3140.4   rfl           9114.2
tpwr            55   rfp           6158.1
pw           6.250   rp             -26.4
   DECOUPLER         lp            -145.6
dn              H1           PLOT
dof              0   wc              250
dm             yyy   sc                0
dmm              w   vs              443
dpwr            38   th                2
dmf          11299   nm    ph
```



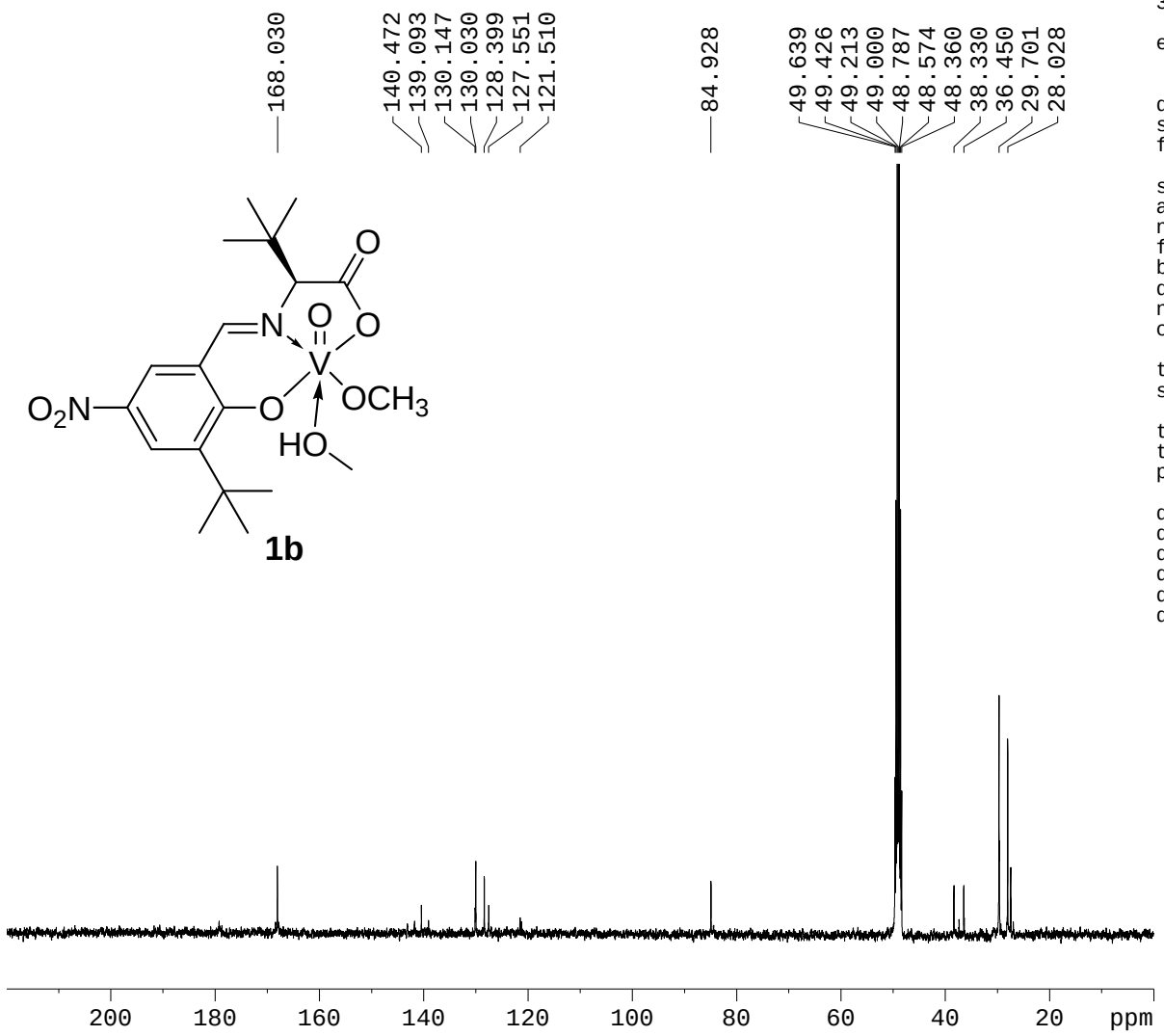

```
3tert5no2H

exp1 PROTON

      SAMPLE          PRESATURATION
date  Jun 24 2014 satmode          n
solvent     cd3od wet              n
file          exp       SPECIAL
   ACQUISITION     temp         25.0
sw          6410.3 gain      not used
at             2.556 spin      not used
np            32768 hst          0.008
fb             4000 pw90        14.000
bs                4 alfa        10.000
d1            1.000        FLAGS
nt             1000 il               n
ct              100 in               n
   TRANSMITTER     dp               y
tn               H1 hs              nn
sfrq       399.766      PROCESSING
tof          399.7 fn        not used
tpwr            61       DISPLAY
pw            7.000 sp          -160.4
   DECOUPLER       wp          3837.4
dn               C13 rf1         2121.4
dof                0 rfp         1323.2
dm               nnn rp          -156.1
decwave   W40_atb lp                0
dpwr              39         PLOT
dmf           29412 wc             215
                    sc              35
                    vs             156
                    th               1
                    ai  cdc  ph
```



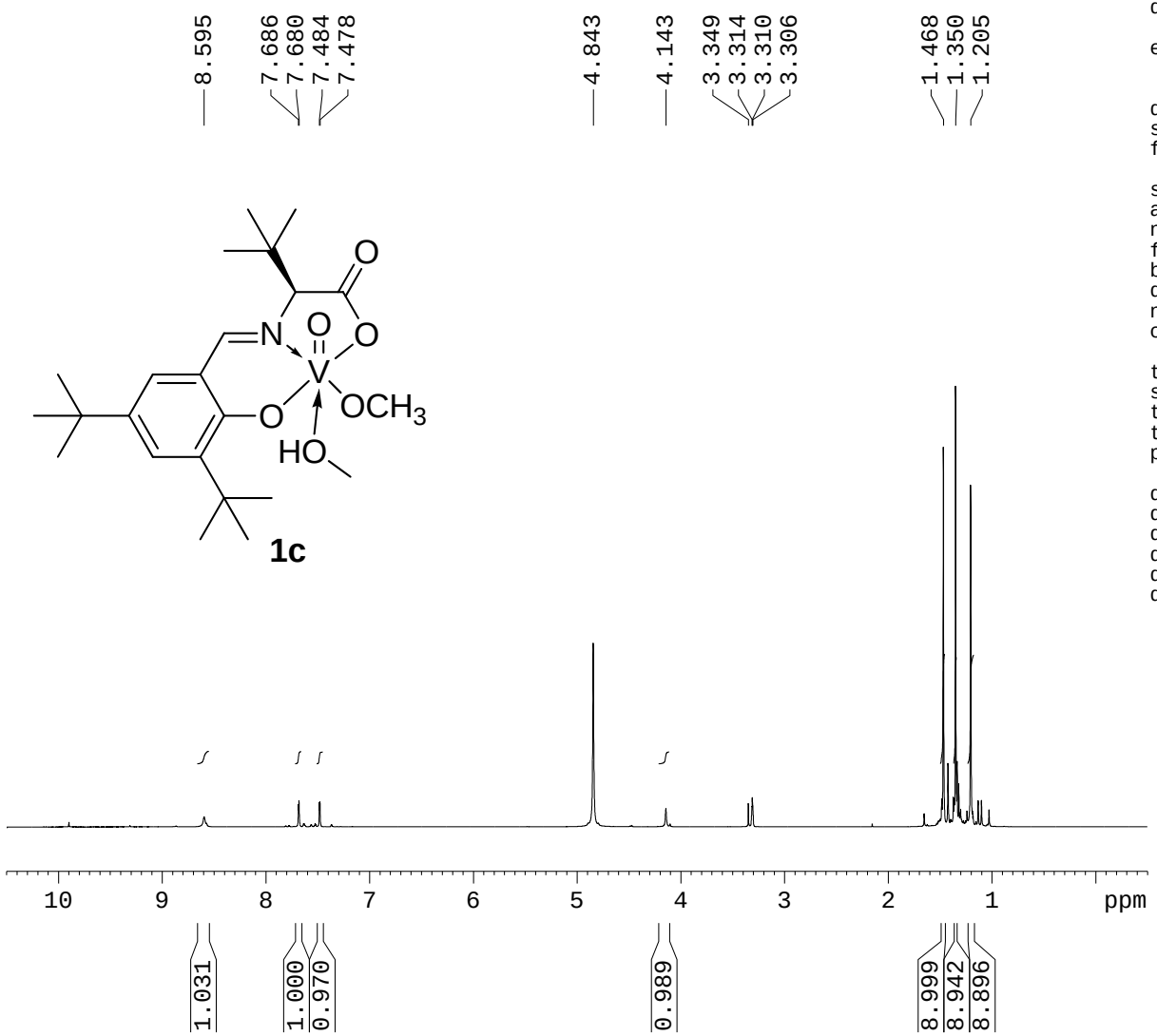
NO2tertV

exp2 PHOSPHORUS

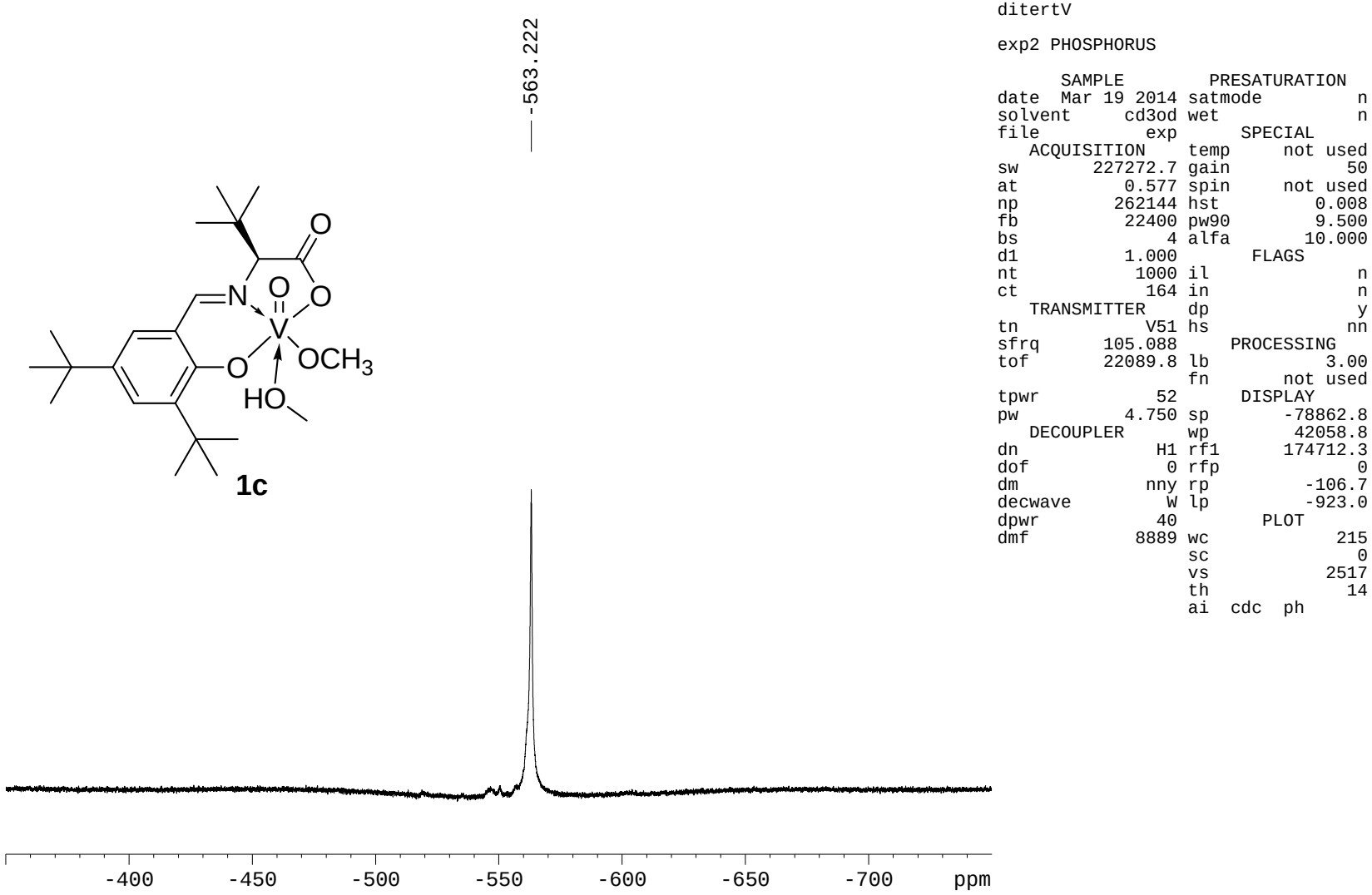
	SAMPLE		PRESATURATION
date	Apr 12 2014	satmode	n
solvent	cd3od	wet	n
file	exp		SPECIAL
	ACQUISITION	temp	not used
sw	227272.7	gain	50
at	0.577	spin	not used
np	262144	hst	0.008
fb	22400	pw90	9.500
bs	4	alfa	10.000
d1	1.000		FLAGS
nt	1000	il	n
ct	164	in	n
	TRANSMITTER	dp	y
tn	V51	hs	nn
sfrq	105.088		PROCESSING
tof	22089.8	lb	3.00
		fn	not used
tpwr	52		DISPLAY
pw	4.750	sp	-78862.8
	DECOUPLER	wp	42058.8
dn	H1	rf1	174712.3
dof	0	rfp	0
dm	nny	rp	-106.7
decwave	W	lp	-923.0
dpwr	40		PLOT
dmf	8889	wc	215
		sc	0
		vs	2517
		th	14
		ai	cdc ph



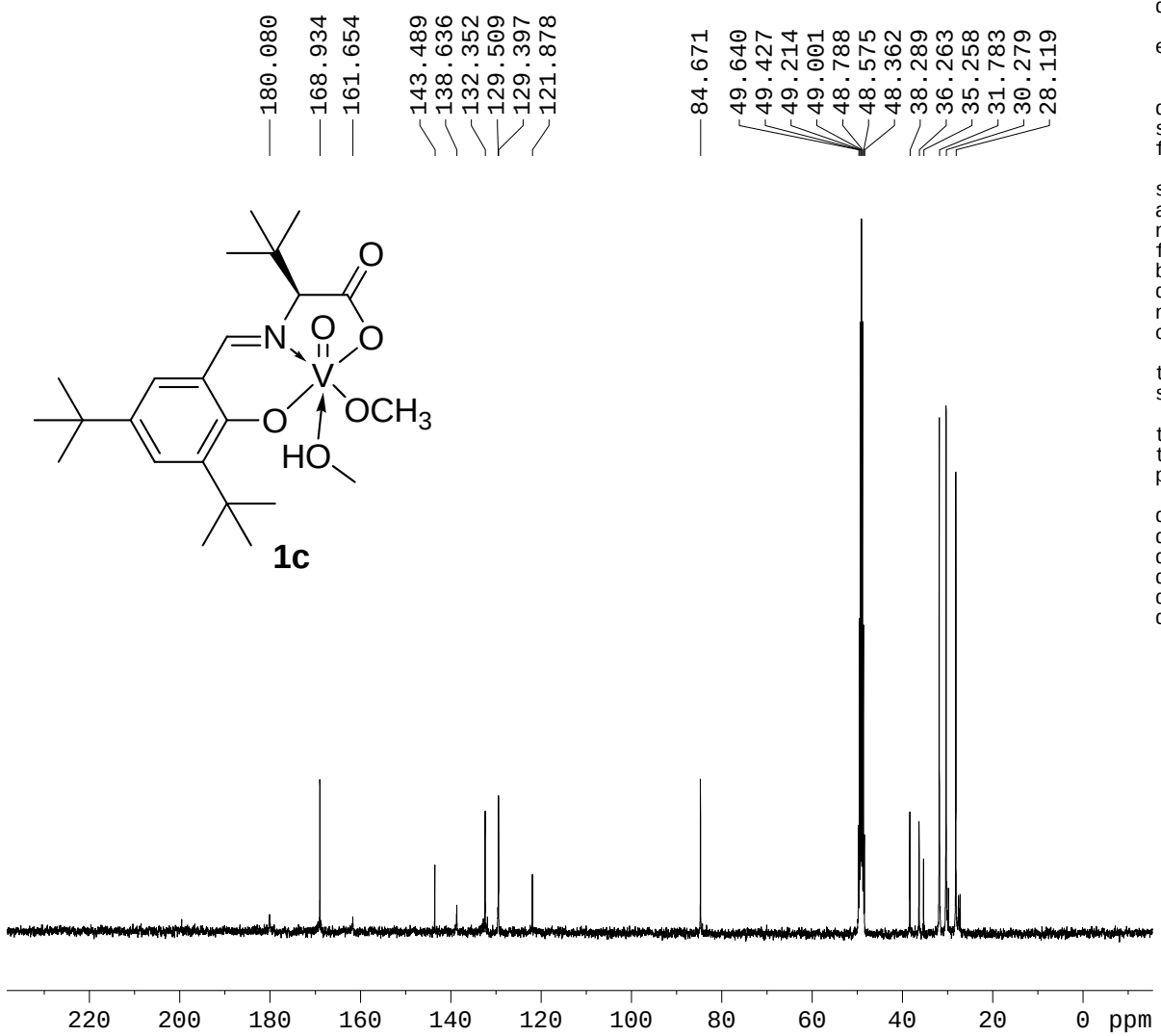

```
3tert5no2C

exp33 CARBON

      SAMPLE          PRESATURATION
date  Jun 24 2014 satmode          n
solvent     cd3od wet              n
file          exp       SPECIAL
   ACQUISITION     temp          25.0
sw         25510.2 gain            30
at           1.285 spin      not used
np           65536 hst          0.008
fb           17000 pw90         9.800
bs               4 alfa        10.000
d1           1.000        FLAGS
nt          100000 il               n
ct             988 in               n
   TRANSMITTER     dp               y
tn              C13 hs              nn
sfrq       100.532      PROCESSING
                    lb            3.00
tof         1530.7 fn        not used
tpwr            60       DISPLAY
pw           4.900 sp            -0.9
   DECOUPLER       wp         22114.3
dn               H1 rf1         6483.4
dof               0 rfp         4925.5
dm              yyy rp           -90.2
decwave           W lp            14.0
dpwr             41         PLOT
dmf          10695 wc             250
                   sc               0
                   vs             405
                   th               5
                   nm  cdc  ph
```



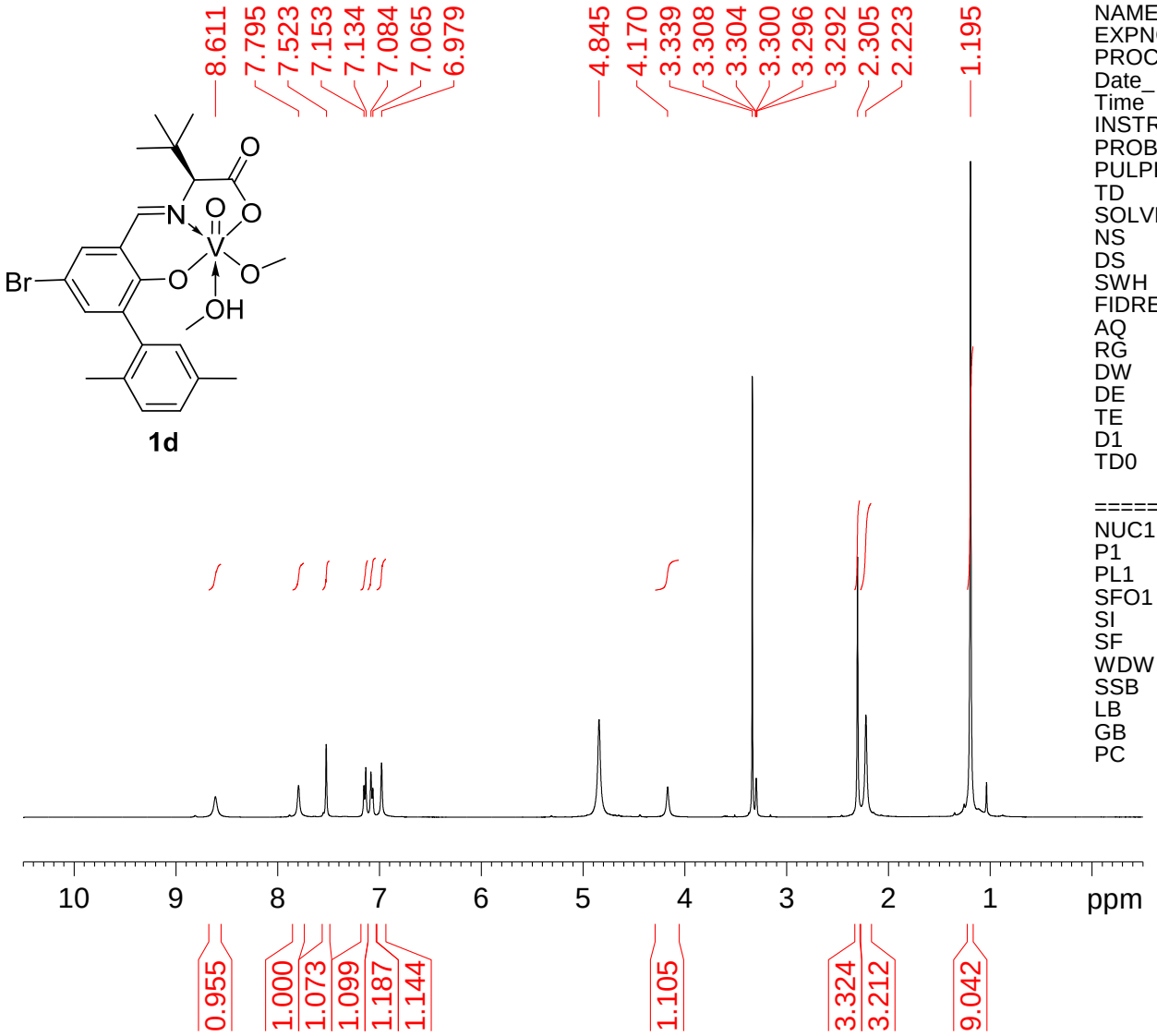

```
ditertH

exp33 PROTON

      SAMPLE          PRESATURATION
date  Jun 25 2014 satmode          n
solvent     cd3od wet              n
file          exp       SPECIAL
   ACQUISITION     temp      not used
sw          6410.3 gain      not used
at           2.556 spin      not used
np           32768 hst          0.008
fb            4000 pw90        14.000
bs               4 alfa        10.000
d1           1.000        FLAGS
nt            1000 il               n
ct             104 in               n
   TRANSMITTER     dp               y
tn              H1 hs              nn
sfrq       399.766     PROCESSING
tof          399.7 fn       not used
tpwr            61       DISPLAY
pw           7.000 sp           -199.9
   DECOUPLER       wp          4197.3
dn             C13 rf1         2121.4
dof              0 rfp         1323.2
dm             nnn rp          -155.5
decwave   W40_atb lp             2.9
dpwr            39         PLOT
dmf          29412 wc             215
                   sc              35
                   vs             945
                   th              15
                   ai  cdc  ph
```




```
ditertV

exp2 PHOSPHORUS

      SAMPLE          PRESATURATION
date  Mar 19 2014 satmode          n
solvent     cd3od wet              n
file          exp       SPECIAL
   ACQUISITION    temp      not used
sw       227272.7 gain            50
at           0.577 spin      not used
np          262144 hst           0.008
fb           22400 pw90          9.500
bs               4 alfa        10.000
d1           1.000         FLAGS
nt            1000 il               n
ct             164 in               n
   TRANSMITTER    dp               y
tn              V51 hs              nn
sfrq       105.088      PROCESSING
tof        22089.8 lb            3.00
                   fn        not used
tpwr            52       DISPLAY
pw           4.750 sp         -78862.8
   DECOUPLER       wp          42058.8
dn               H1 rf1        174712.3
dof               0 rfp              0
dm              nny rp           -106.7
decwave           W lp           -923.0
dpwr             40         PLOT
dmf            8889 wc             215
                    sc               0
                    vs            2517
                    th              14
                    ai  cdc  ph
```

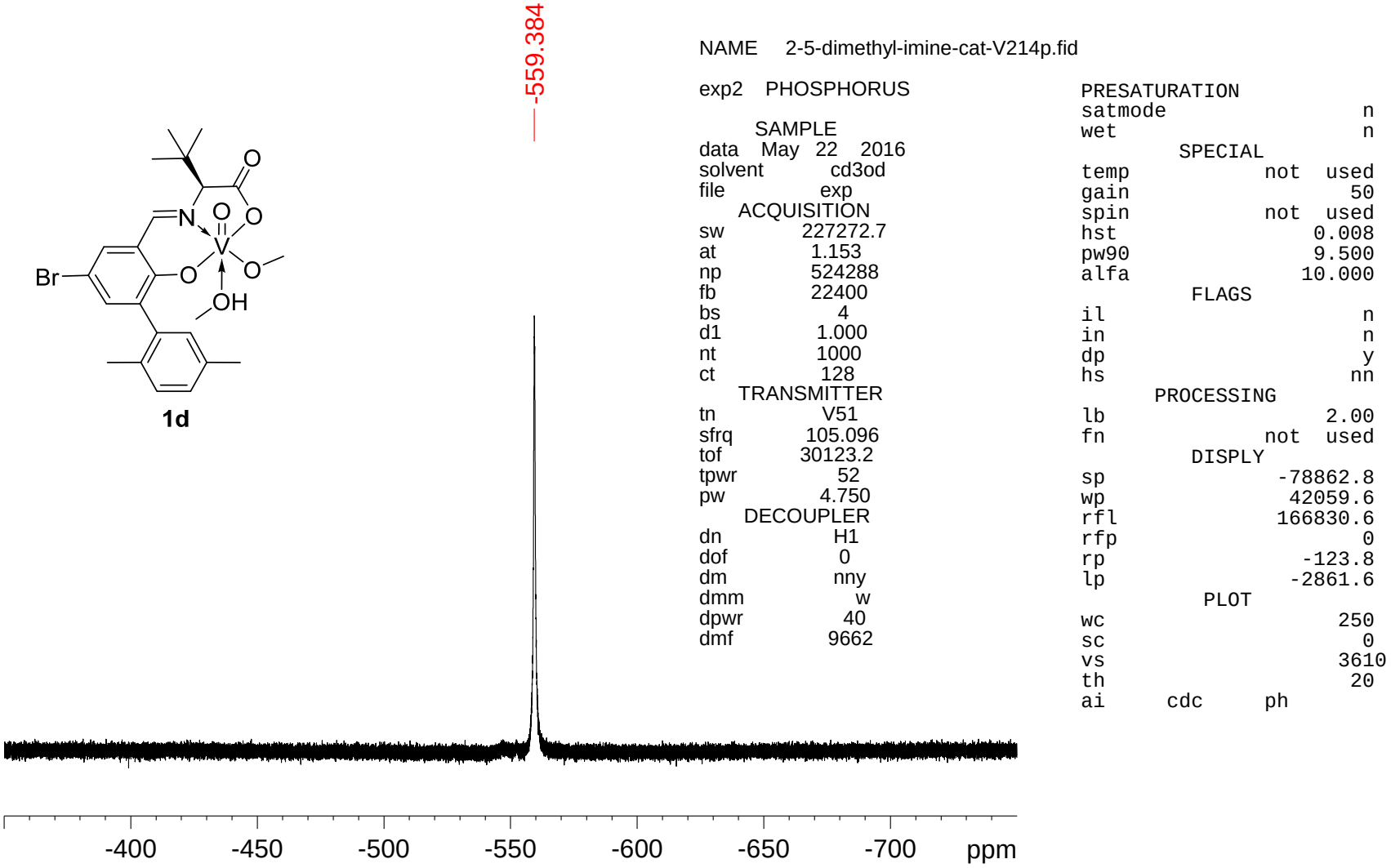



```
ditertC

exp33 CARBON

      SAMPLE          PRESATURATION
date  Jun 25 2014 satmode          n
solvent     cd3od wet              n
file          exp       SPECIAL
   ACQUISITION     temp      not used
sw         25510.2 gain            30
at           1.285 spin      not used
np           65536 hst          0.008
fb           17000 pw90         9.800
bs               4 alfa        10.000
d1           1.000        FLAGS
nt          100000 il               n
ct            1036 in               n
   TRANSMITTER     dp               y
tn              C13 hs              nn
sfrq       100.532      PROCESSING
                    lb            3.00
tof         1530.7 fn        not used
tpwr            60       DISPLAY
pw           4.900 sp            -0.9
   DECOUPLER       wp         22114.3
dn               H1 rf1         6480.3
dof               0 rfp         4925.5
dm              yyy rp           -91.9
decwave           W lp            27.2
dpwr             41         PLOT
dmf          10695 wc             250
                   sc               0
                   vs             333
                   th               7
                   nm  cdc  ph
```



NAME 2-5-dimethyl-Br-imine-cat-H-0308
EXPNO 1
PROCNO 1
Date_ 20160408
Time 13.38
INSTRUM spect
PROBHD 5 mm DUL 13C-1
PULPROG zg30
TD 32768
SOLVENT MeOD
NS 6
DS 0
SWH 6410.256 Hz
FIDRES 0.195625 Hz
AQ 2.5559540 sec
RG 4
DW 78.000 usec
DE 6.00 usec
TE 300.0 K
D1 2.00000000 sec
TD0 1

======== CHANNEL f1 ========
NUC1 1H
P1 10.00 usec
PL1 -2.40 dB
SFO1 400.1528010 MHz
SI 16384
SF 400.1500168 MHz
WDW EM
SSB 0
LB 0.00 Hz
GB 0
PC 1.00

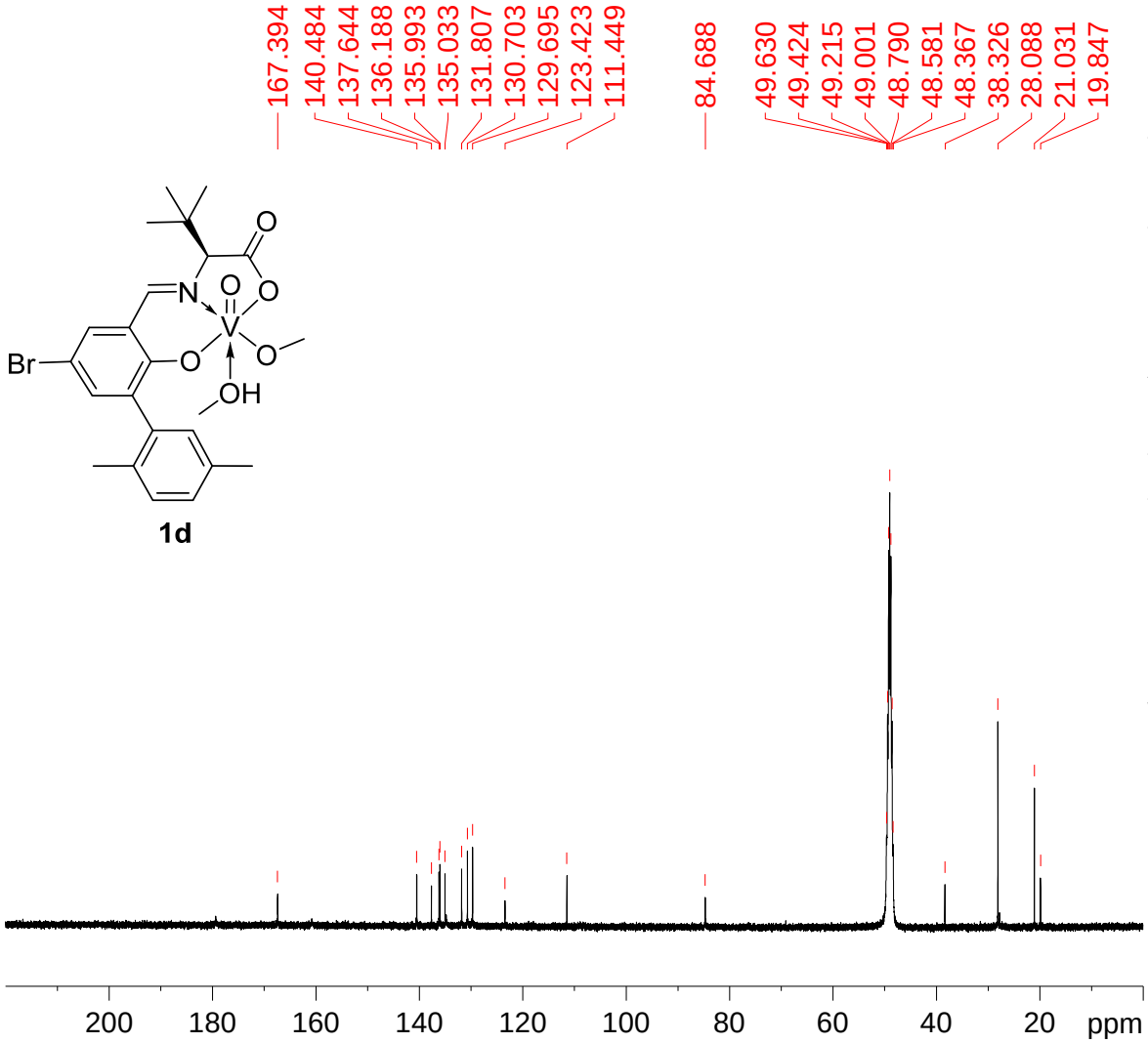


NAME 2-5-dimethyl-imine-cat-V214p.fid

exp2 PHOSPHORUS

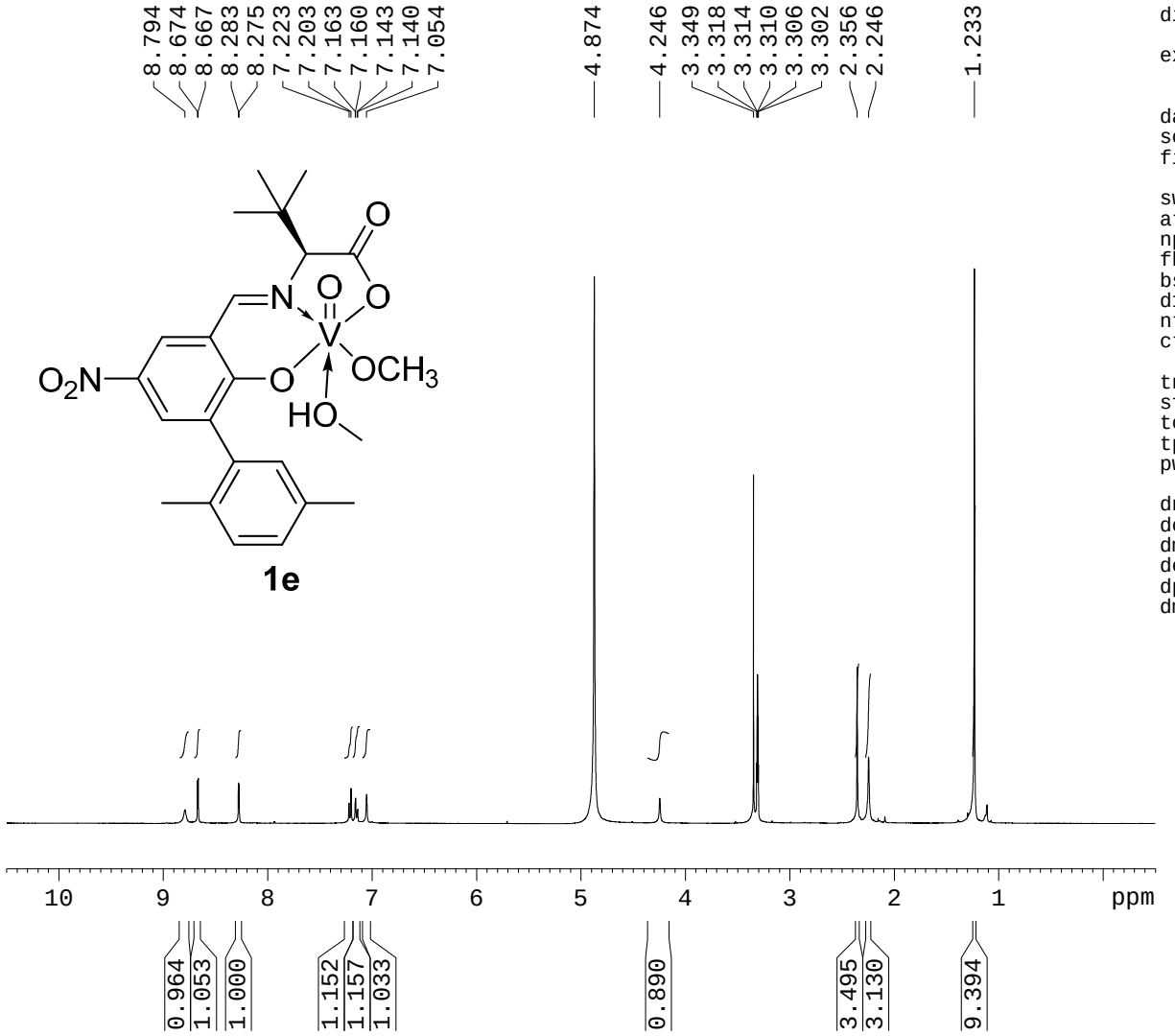
SAMPLE	
data	May 22 2016
solvent	cd3od
file	exp
ACQUISITION	
sw	227272.7
at	1.153
np	524288
fb	22400
bs	4
d1	1.000
nt	1000
ct	128
TRANSMITTER	
tn	V51
sfrq	105.096
tof	30123.2
tpwr	52
pw	4.750
DECOUPLER	
dn	H1
dof	0
dm	nny
dmm	w
dpwr	40
dmf	9662

PRESATURATION	
satmode	n
wet	n
SPECIAL	
temp	not used
gain	50
spin	not used
hst	0.008
pw90	9.500
alfa	10.000
FLAGS	
il	n
in	n
dp	y
hs	nn
PROCESSING	
lb	2.00
fn	not used
DISPLY	
sp	-78862.8
wp	42059.6
rfl	166830.6
rfp	0
rp	-123.8
lp	-2861.6
PLOT	
wc	250
sc	0
vs	3610
th	20
ai cdc	ph

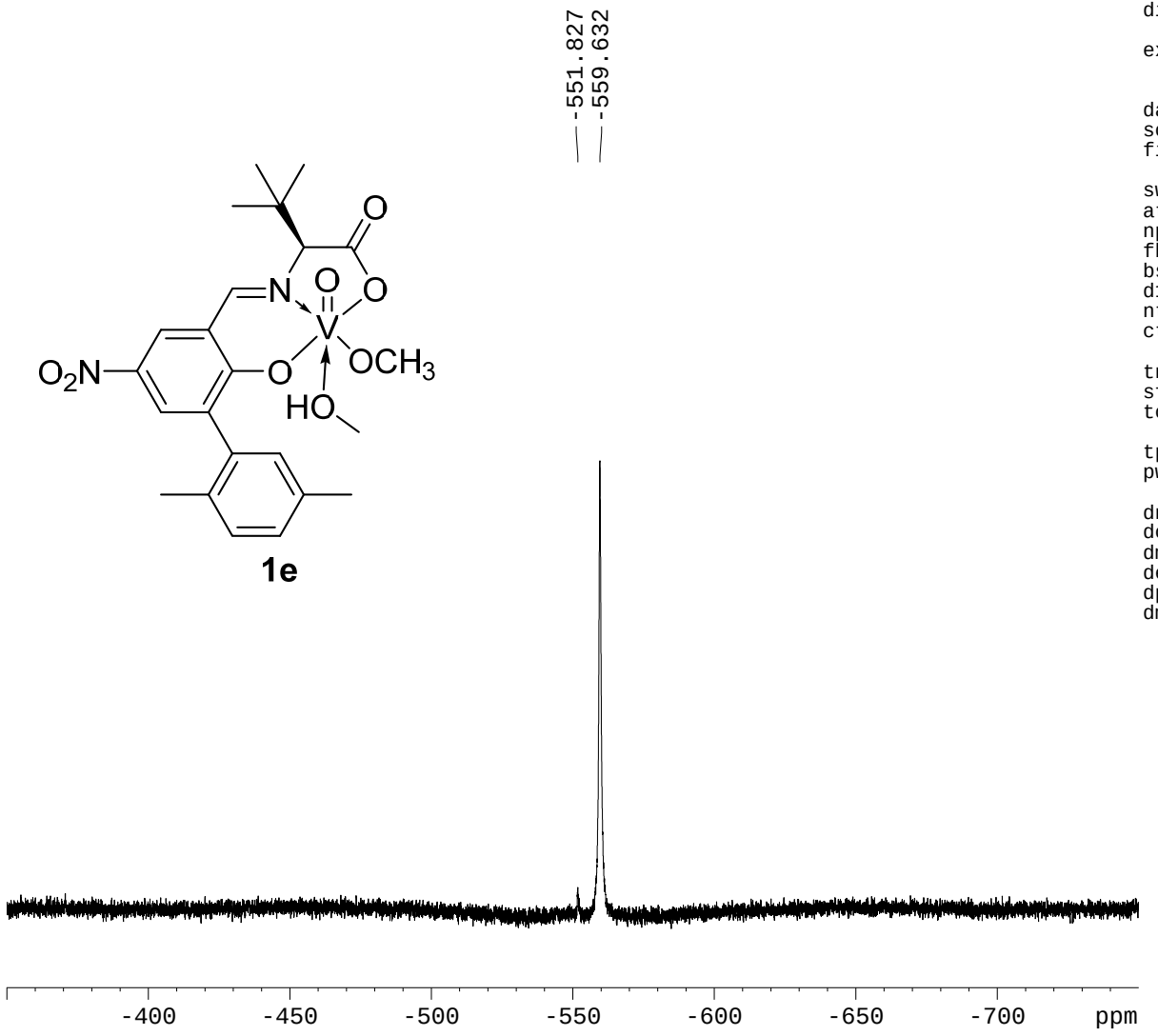


NAME 2-5-dimethyl-imine-cat-13C
EXPNO 3
PROCNO 1
Date_ 20161208
Time 9.59
INSTRUM spect
PROBHD 5 mm DUL 13C-1
PULPROG zg30
TD 32768
SOLVENT MeOD
NS 172
DS 0
SWH 6410.256 Hz
FIDRES 0.195625 Hz
AQ 2.5559540 sec
RG 4
DW 78.000 usec
DE 6.00 usec
TE 300.0 K
D1 2.00000000 sec
TD0 1

======== CHANNEL f1 ========
NUC1 1H
P1 10.00 usec
PL1 -2.40 dB
SFO1 400.1528010 MHz
SI 16384
SF 400.1500168 MHz
WDW EM
SSB 0
LB 0.00 Hz
GB 0
PC 1.00



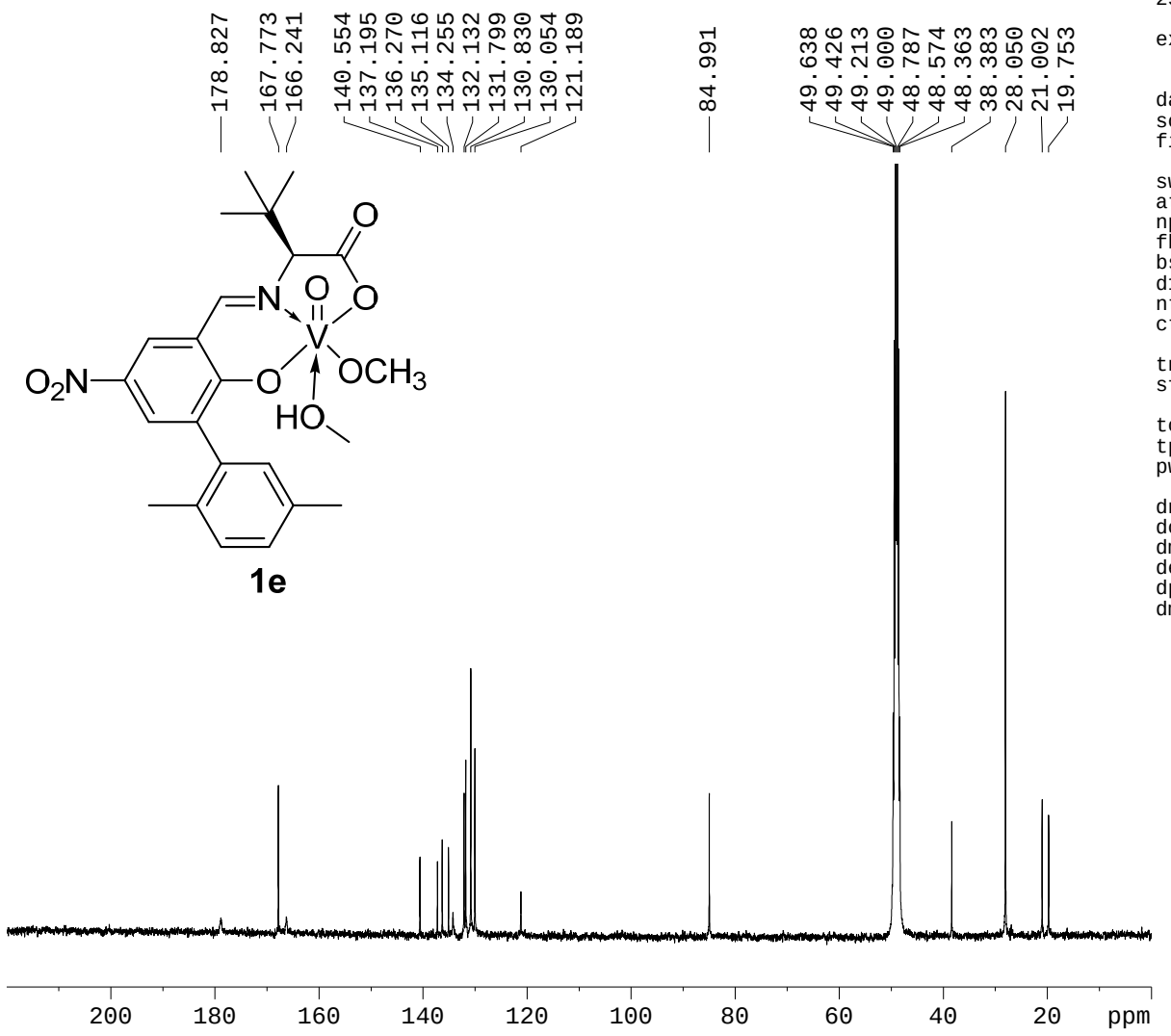

```
dimecatalyst3

exp33 PROTON

      SAMPLE          PRESATURATION
date  Sep 18 2013 satmode          n
solvent     cd3od wet              n
file          exp       SPECIAL
   ACQUISITION     temp      not used
sw          6410.3 gain      not used
at           2.556 spin      not used
np           32768 hst          0.008
fb            4000 pw90        14.000
bs               4 alfa        10.000
d1           1.000        FLAGS
nt            1000 il               n
ct             104 in               n
   TRANSMITTER     dp               y
tn              H1 hs              nn
sfrq       399.766     PROCESSING
tof          399.7 fn       not used
tpwr            61       DISPLAY
pw           7.000 sp          -199.9
   DECOUPLER       wp          4197.3
dn             C13 rf1         2121.4
dof              0 rfp         1323.2
dm             nnn rp          -155.5
decwave   W40_atb lp             2.9
dpwr            39         PLOT
dmf          29412 wc             215
                   sc              35
                   vs             945
                   th              15
                   ai  cdc  ph
```



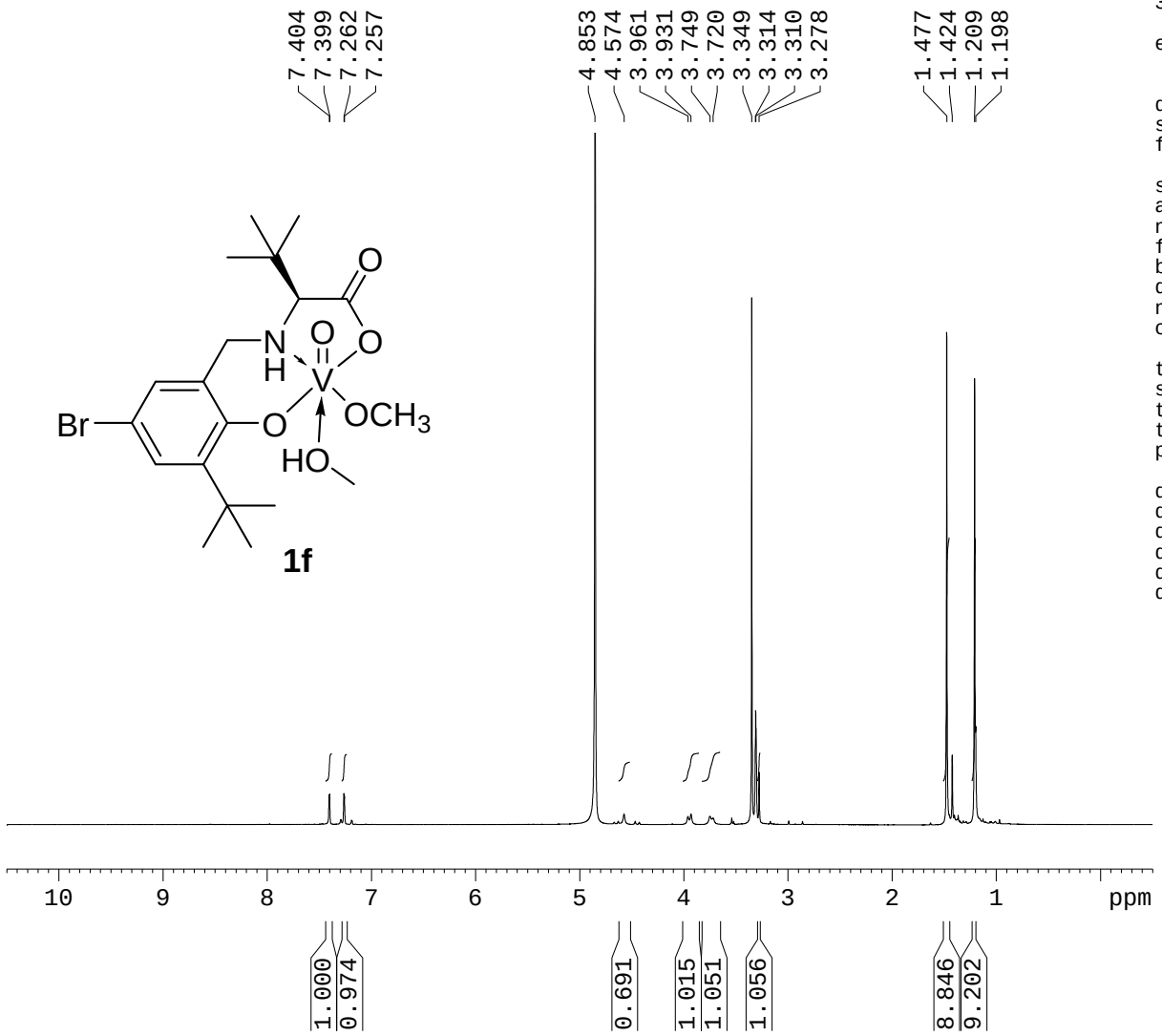
dime catalyst

exp3 PHOSPHORUS

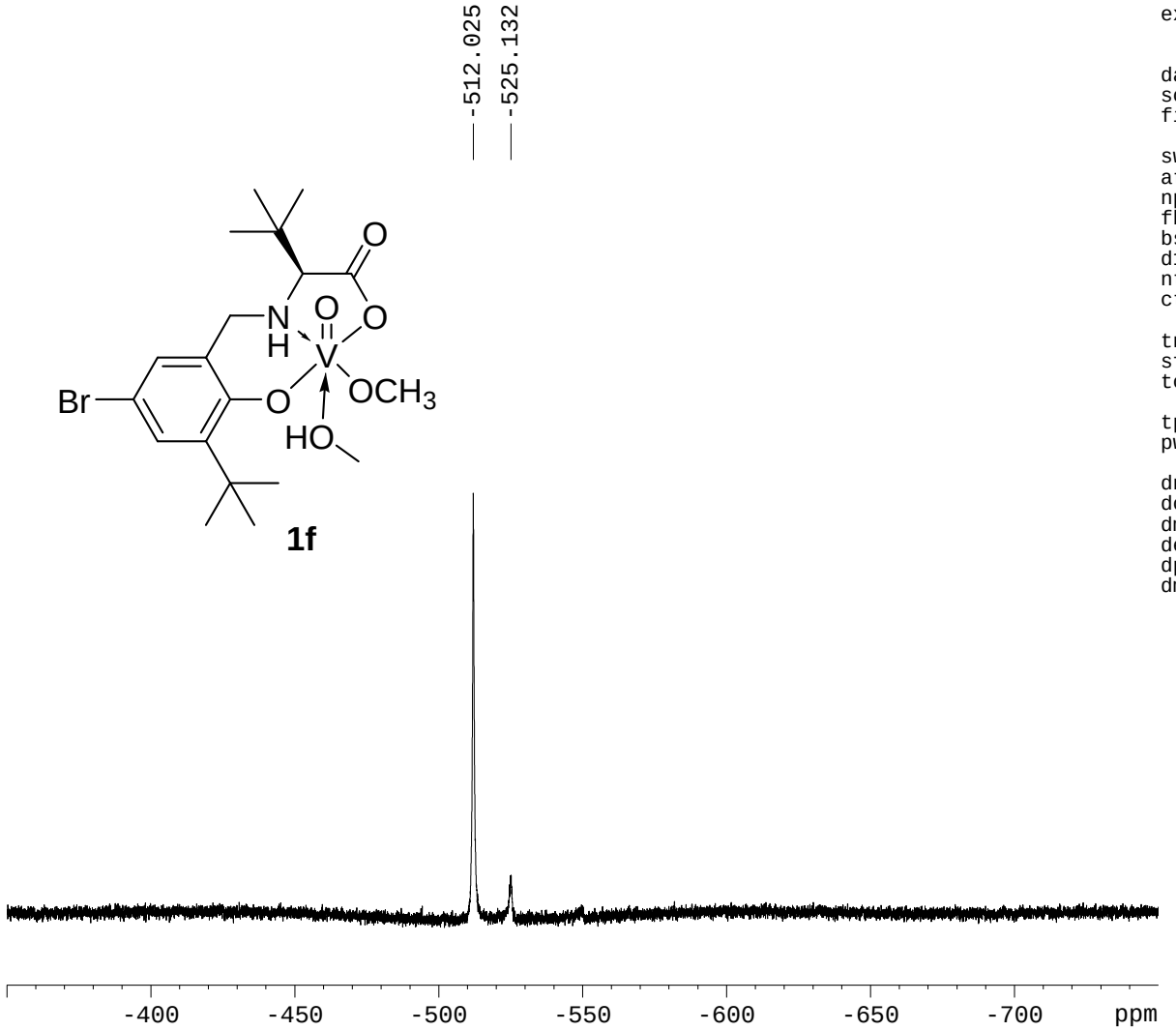
SAMPLE		PRESATURATION	
date	Sep 18 2013	satmode	n
solvent	cd3od	wet	n
file	exp	SPECIAL	
ACQUISITION		temp	not used
sw	227272.7	gain	50
at	1.153	spin	not used
np	524288	hst	0.008
fb	22400	pw90	9.500
bs	4	alfa	10.000
d1	1.000	FLAGS	
nt	1000	il	n
ct	80	in	n
TRANSMITTER		dp	y
tn	V51	hs	nn
sfrq	105.096	PROCESSING	
tof	30123.2	lb	3.00
		fn	not used
tpwr	52	DISPLAY	
pw	4.750	sp	-78862.8
DECOUPLER		wp	42059.6
dn	H1	rf1	166678.9
dof	0	rfp	0
dm	nny	rp	70.9
decwave	W	lp	-2861.6
dpwr	40	PLOT	
dmf	8889	wc	250
		sc	0
		vs	1156
		th	8
		ai cdc	ph



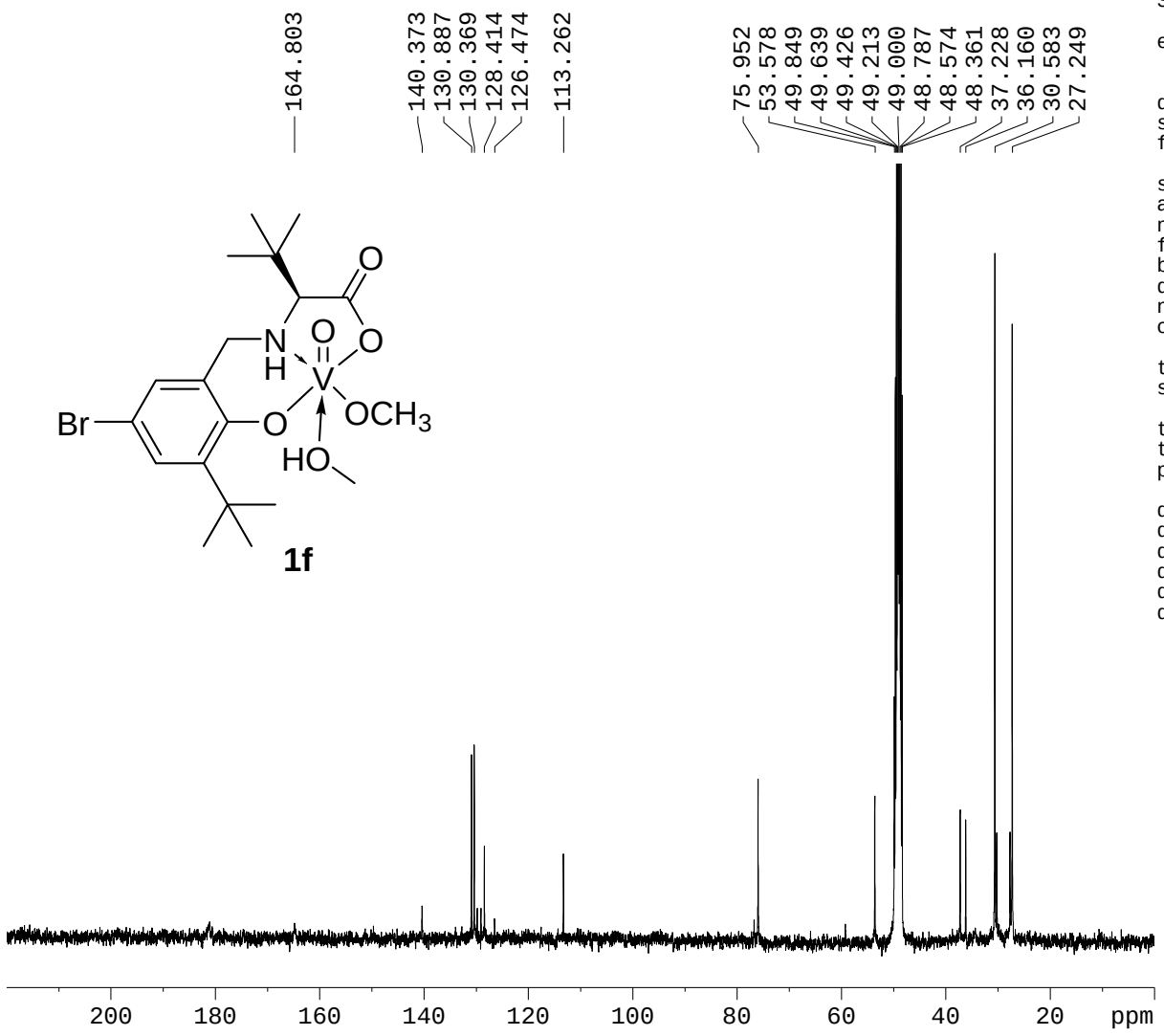

```
25diMe5no2carbasalateC

exp1  CARBON

      SAMPLE          PRESATURATION
date  Jul  5 2014 satmode          n
solvent     cd3od wet              n
file          exp       SPECIAL
   ACQUISITION     temp      not used
sw         25510.2 gain            30
at           1.285 spin      not used
np           65536 hst          0.008
fb           17000 pw90         9.800
bs               4 alfa        10.000
d1           1.000         FLAGS
nt          100000 il               n
ct            4760 in               n
   TRANSMITTER     dp               y
tn             C13 hs              nn
sfrq       100.532      PROCESSING
                    lb            3.00
tof         1530.7 fn        not used
tpwr            60       DISPLAY
pw           4.900 sp            -0.6
   DECOUPLER       wp         22114.3
dn              H1 rf1         1697.8
dof              0 rfp              0
dm             yyy rp           -94.9
decwave          W lp            -4.5
dpwr            41          PLOT
dmf          10695 wc             250
                   sc               0
                   vs             400
                   th               5
                   nm  cdc  ph
```



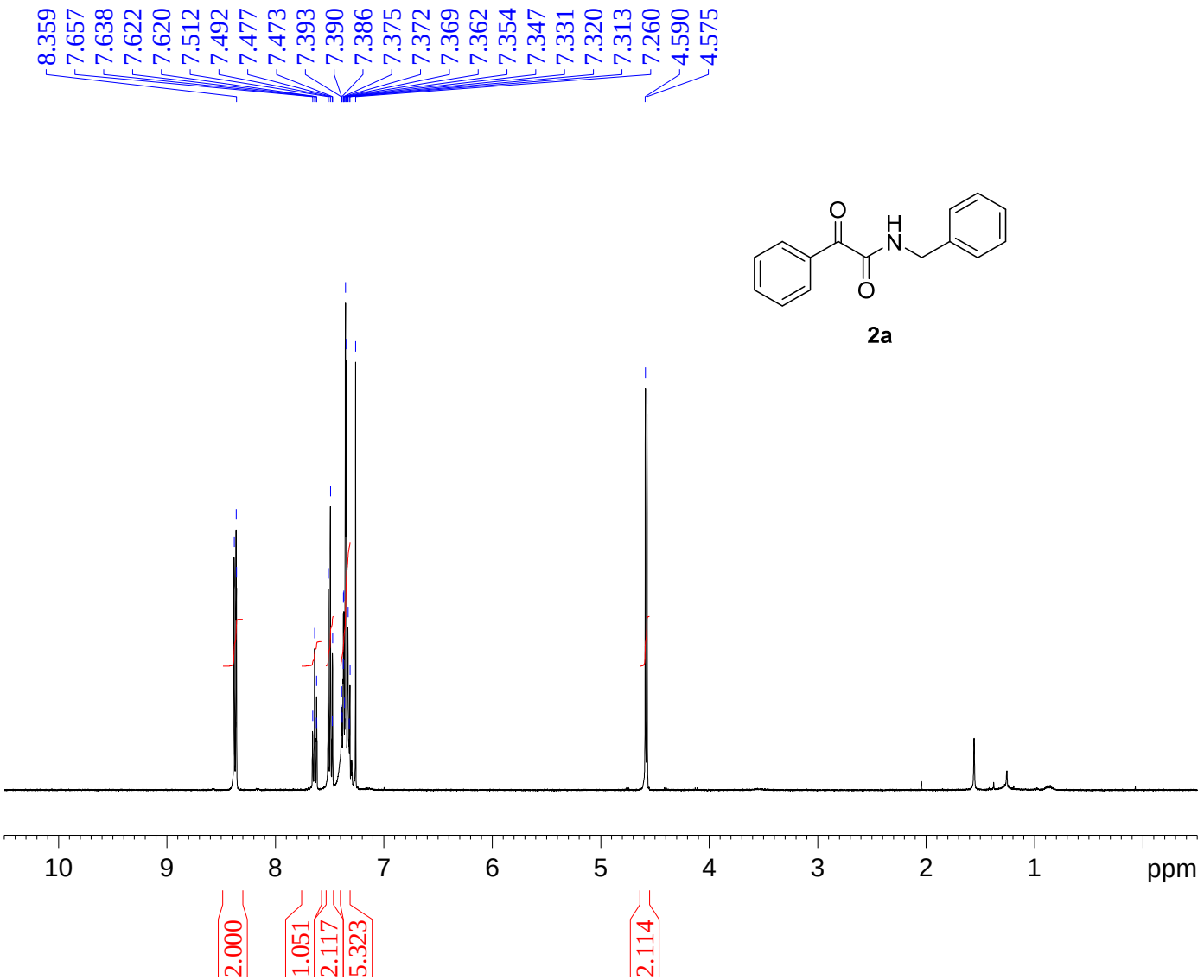

```
3tbu5brreducedH

exp1  PROTON

      SAMPLE          PRESATURATION
date  Jun 26 2014 satmode          n
solvent     cd3od wet              n
file          exp       SPECIAL
   ACQUISITION     temp      not used
sw          6410.3 gain      not used
at           2.556 spin      not used
np           32768 hst          0.008
fb            4000 pw90        14.000
bs               4 alfa        10.000
d1           1.000        FLAGS
nt            1000 il               n
ct              64 in               n
   TRANSMITTER     dp               y
tn               H1 hs              nn
sfrq       399.766     PROCESSING
tof          399.7 fn       not used
tpwr            61       DISPLAY
pw           7.000 sp           -160.4
   DECOUPLER       wp          3837.4
dn              C13 rf1         2121.0
dof               0 rfp         1323.2
dm              nnn rp          -158.1
decwave   W40_atb lp                0
dpwr             39         PLOT
dmf           29412 wc             215
                    sc              35
                    vs             500
                    th               5
                    ai  cdc  ph
```




```
36

exp33 PHOSPHORUS

      SAMPLE        PRESATURATION
date  Mar 30 2014 satmode          n
solvent     cd3od wet              n
file          exp      SPECIAL
   ACQUISITION    temp      not used
sw       227272.7 gain            50
at          1.153 spin      not used
np         524288 hst          0.008
fb          22400 pw90         9.500
bs              4 alfa        10.000
d1          1.000       FLAGS
nt           1000 il               n
ct            192 in               n
   TRANSMITTER    dp               y
tn             V51 hs              nn
sfrq      105.096     PROCESSING
tof       30123.2 lb            3.00
                  fn        not used
tpwr            52      DISPLAY
pw           4.750 sp         -78862.8
   DECOUPLER      wp          42059.6
dn              H1 rf1       166678.9
dof              0 rfp              0
dm             nny rp           125.2
decwave          W lp         -2861.6
dpwr            40        PLOT
dmf           8889 wc             250
                   sc               0
                   vs            1832
                   th               8
                   ai  cdc  ph
```



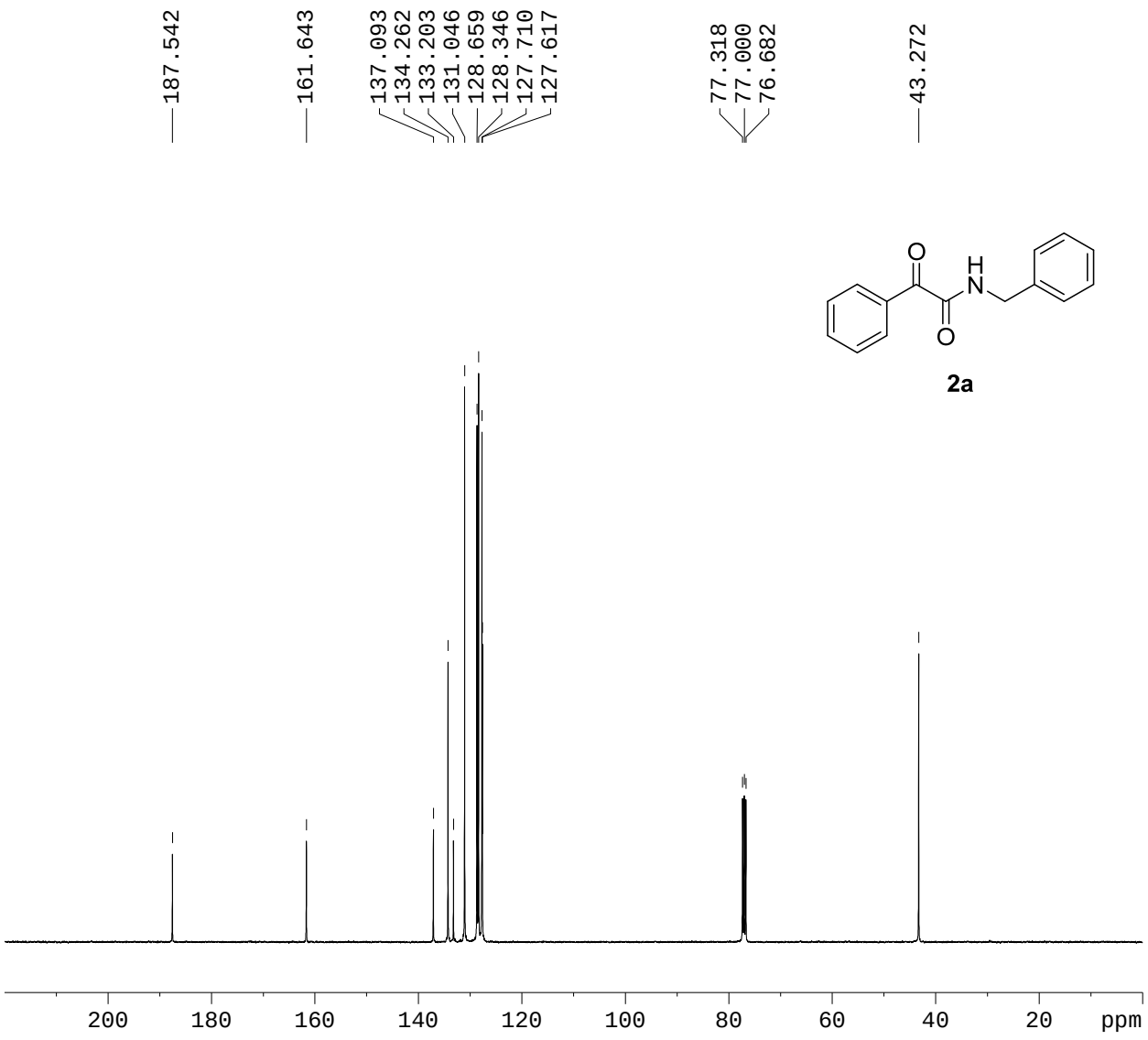

```
3tbu5brreducedC

exp33 CARBON

      SAMPLE          PRESATURATION
date  Jun 26 2014 satmode          n
solvent     cd3od wet              n
file          exp       SPECIAL
   ACQUISITION     temp      not used
sw         25510.2 gain            30
at           1.285 spin      not used
np           65536 hst           0.008
fb           17000 pw90          9.800
bs               4 alfa        10.000
d1           1.000        FLAGS
nt          100000 il               n
ct           15396 in               n
   TRANSMITTER     dp               y
tn              C13 hs              nn
sfrq       100.532      PROCESSING
                    lb            3.00
tof         1530.7 fn        not used
tpwr            60       DISPLAY
pw           4.900 sp            -0.9
   DECOUPLER       wp         22114.3
dn               H1 rf1          6478.8
dof               0 rfp          4925.5
dm              yyy rp           -90.0
decwave           W lp            11.6
dpwr             41         PLOT
dmf          10695 wc             250
                   sc               0
                   vs            2252
                   th              13
                   nm  cdc  ph
```



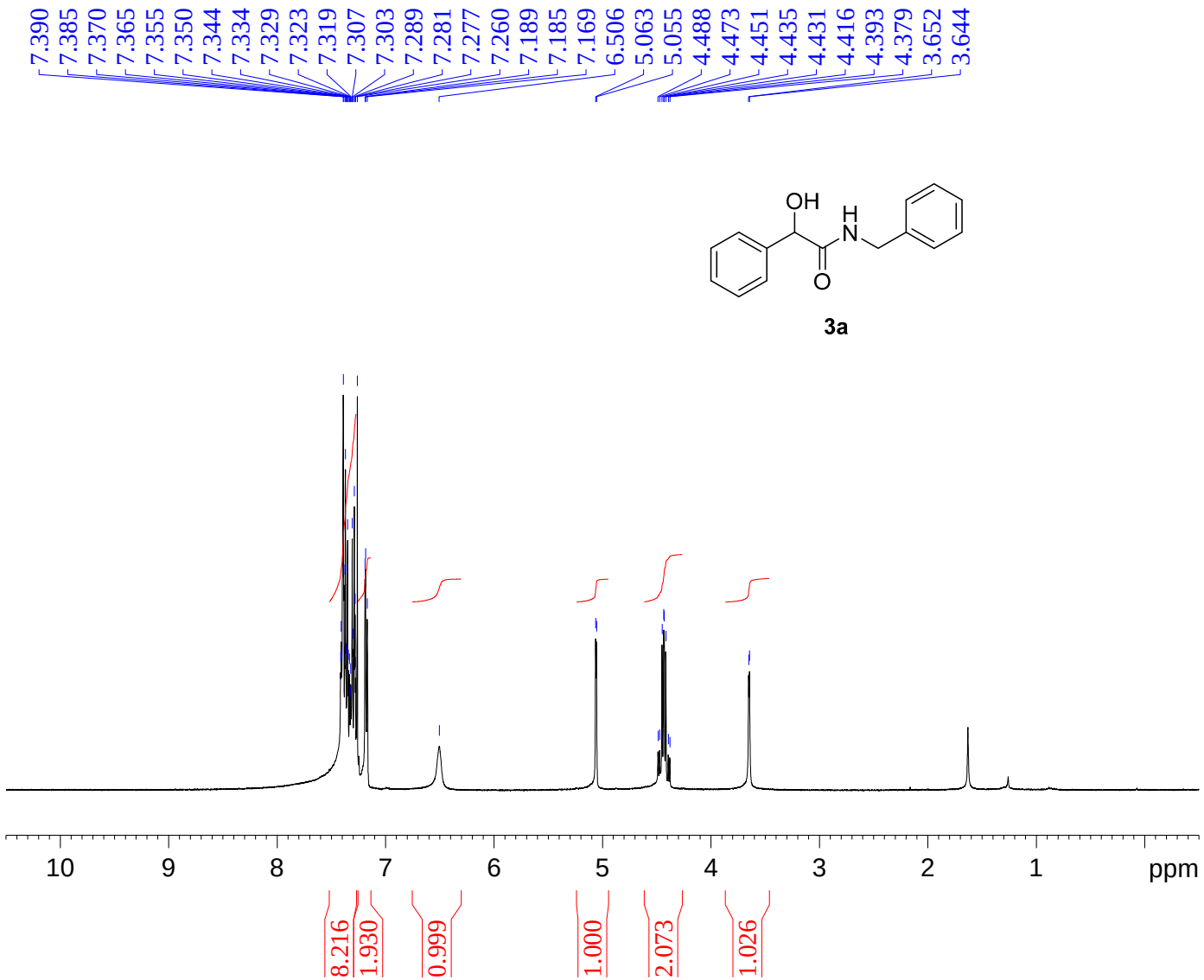
NAME	20141117
EXPNO	1
PROCNO	1
Date_	20141117
Time	20.10
INSTRUM	spect
PROBHD	5 mm DUL 13C-1
PULPROG	zg30
TD	32768
SOLVENT	CDCl3
NS	44
DS	0
SWH	6410.256 Hz
FIDRES	0.195625 Hz
AQ	2.5559540 sec
RG	4
DW	78.000 usec
DE	6.00 usec
TE	300.0 K
D1	2.00000000 sec
TD0	1

======== CHANNEL f1 ========

NUC1	1H
P1	10.00 usec
PL1	-2.40 dB
SFO1	400.1528010 MHz
SI	16384
SF	400.1500092 MHz
WDW	EM
SSB	0
LB	0.00 Hz
GB	0
PC	1.00

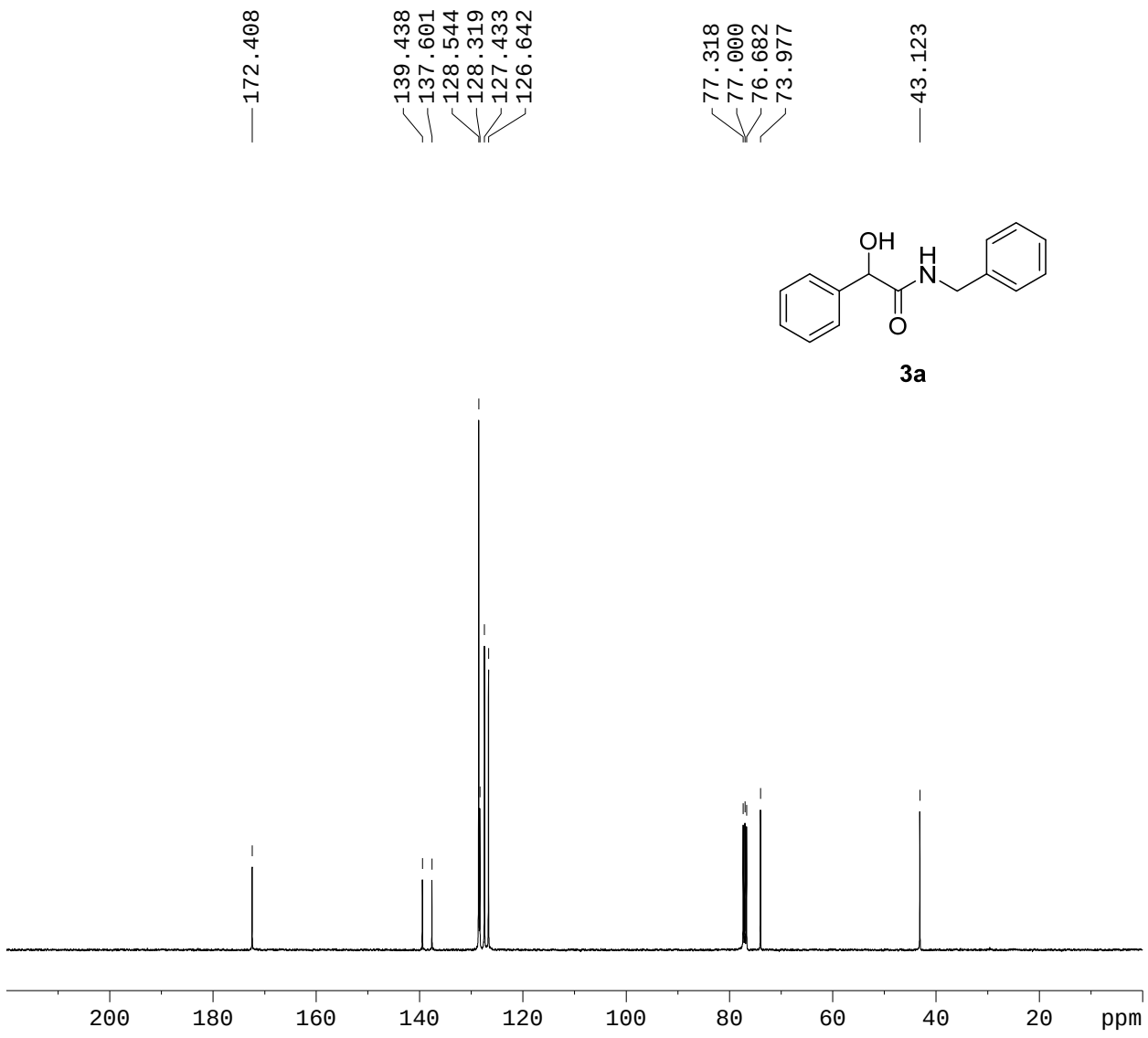


NAME	13C
EXPNO	2
PROCNO	1
Date_	20150110
Time	19.41
INSTRUM	spect
PROBHD	5 mm DUL 13C-1
PULPROG	zgpg30
TD	65536
SOLVENT	CDCl3
NS	1363
DS	0
SWH	22727.273 Hz
FIDRES	0.346791 Hz
AQ	1.4418420 sec
RG	57
DW	22.000 usec
DE	6.00 usec
TE	300.0 K
D1	2.00000000 sec
d11	0.03000000 sec
DELTA	1.89999998 sec
TD0	1
======== CHANNEL f1 ========	
NUC1	13C
P1	9.70 usec
PL1	-0.50 dB
SFO1	100.6288660 MHz
======== CHANNEL f2 ========	
CPDPRG2	waltz16
NUC2	1H
PCPD2	90.00 usec
PL2	-2.40 dB
PL12	15.10 dB
PL13	18.10 dB
SFO2	400.1516010 MHz
SI	32768
SF	100.6178159 MHz
WDW	EM
SSB	0
LB	3.00 Hz
GB	0
PC	1.00



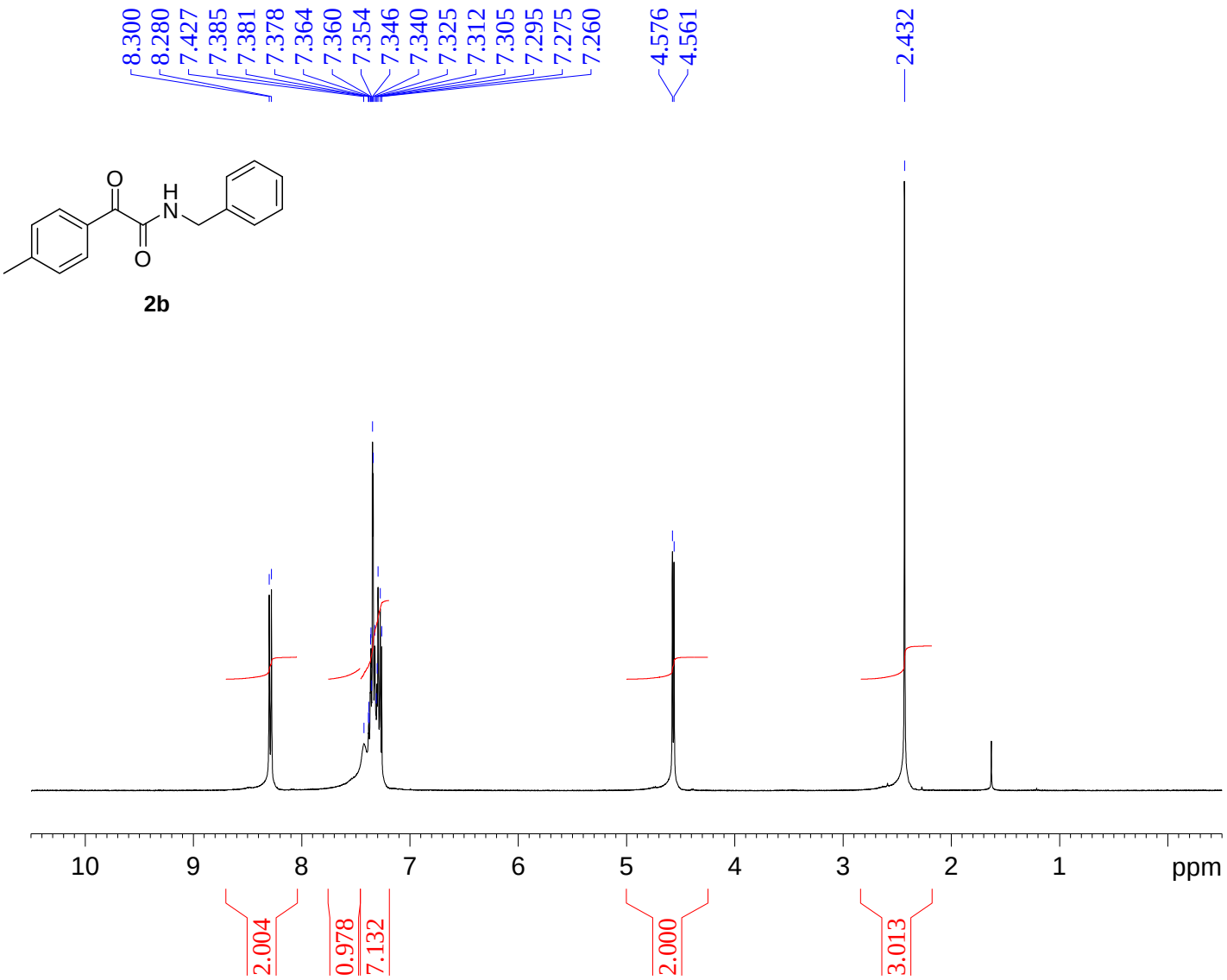
NAME Substrate
EXPNO 7
PROCNO 1
Date_ 20150702
Time 18.27
INSTRUM spect
PROBHD 5 mm DUL 13C-1
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 48
DS 0
SWH 6410.256 Hz
FIDRES 0.195625 Hz
AQ 2.5559540 sec
RG 4
DW 78.000 usec
DE 6.00 usec
TE 300.0 K
D1 2.00000000 sec
TD0 1

======== CHANNEL f1 ========
NUC1 1H
P1 10.00 usec
PL1 -2.40 dB
SFO1 400.1528010 MHz
SI 16384
SF 400.1500088 MHz
WDW EM
SSB 0
LB 0.00 Hz
GB 0
PC 1.00



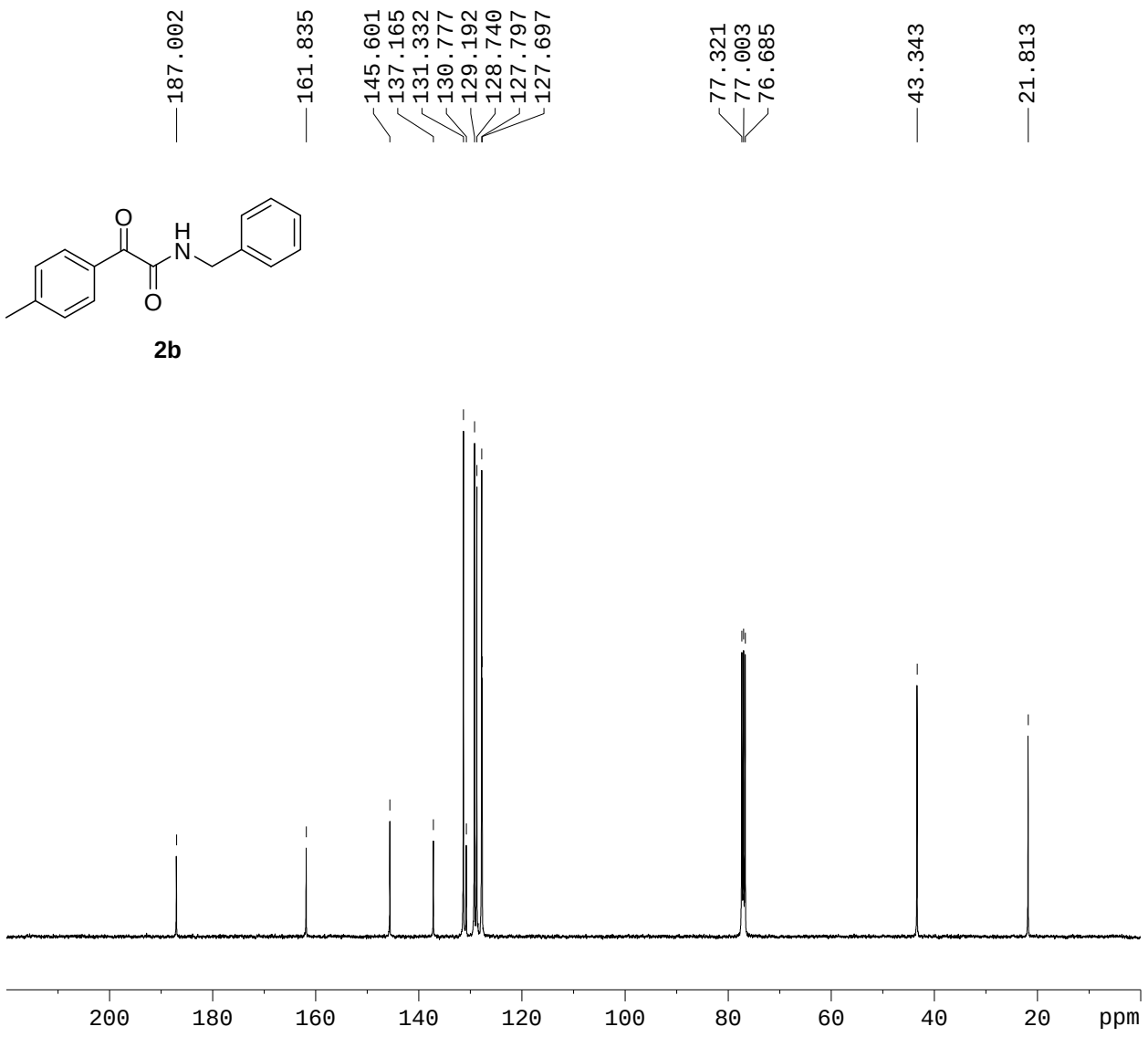
3a

NAME	13C
EXPNO	8
PROCNO	1
Date_	20150426
Time	17.01
INSTRUM	spect
PROBHD	5 mm DUL 13C-1
PULPROG	zgpg30
TD	65536
SOLVENT	CDCl3
NS	500
DS	0
SWH	22727.273 Hz
FIDRES	0.346791 Hz
AQ	1.4418420 sec
RG	57
DW	22.000 usec
DE	6.00 usec
TE	300.0 K
D1	2.00000000 sec
d11	0.03000000 sec
DELTA	1.89999998 sec
TD0	1
======== CHANNEL f1 ========	
NUC1	13C
P1	9.70 usec
PL1	-0.50 dB
SFO1	100.6288660 MHz
======== CHANNEL f2 ========	
CPDPRG2	waltz16
NUC2	1H
PCPD2	90.00 usec
PL2	-2.40 dB
PL12	15.10 dB
PL13	18.10 dB
SFO2	400.1516010 MHz
SI	32768
SF	100.6178128 MHz
WDW	EM
SSB	0
LB	3.00 Hz
GB	0
PC	1.00



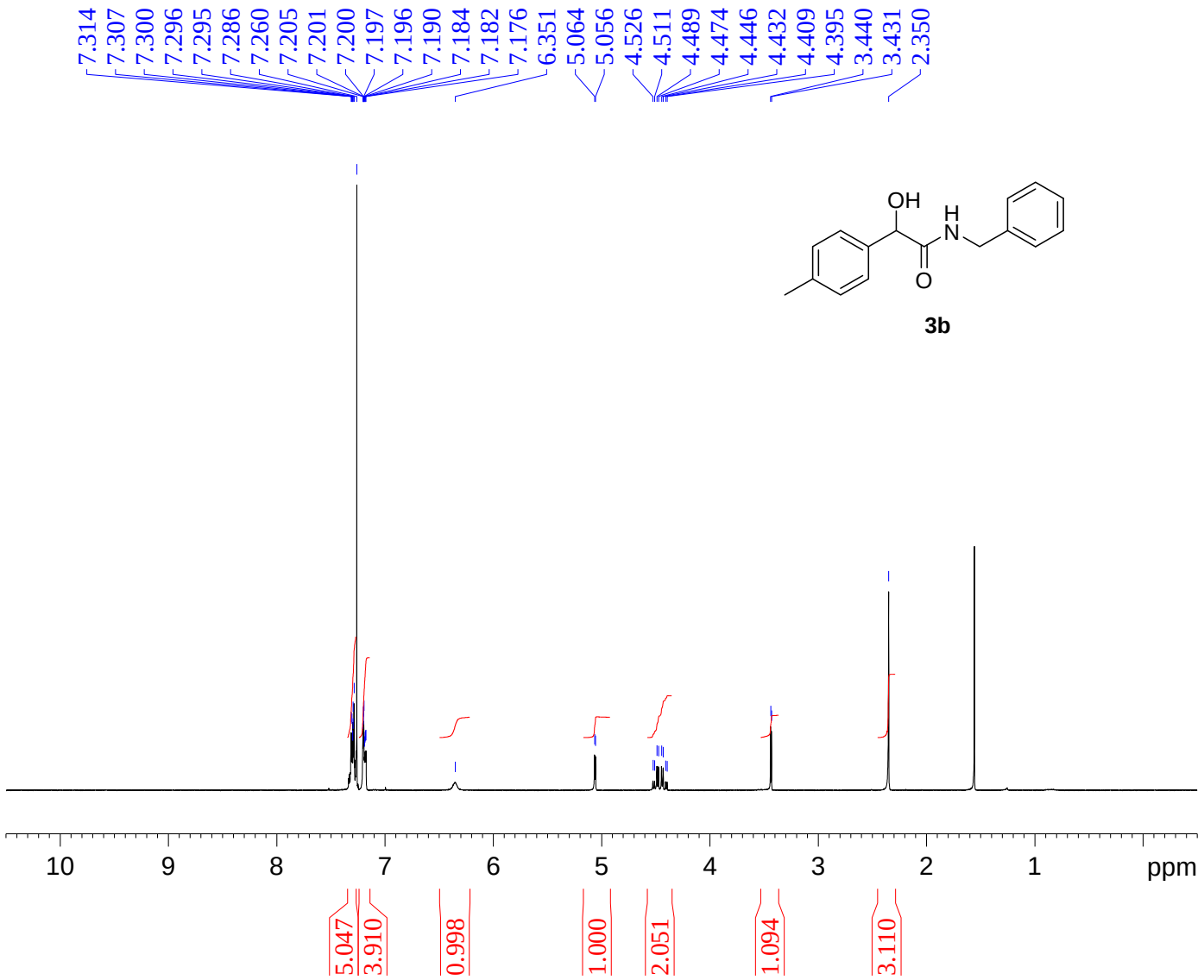
NAME Substrate 1H
EXPNO 1
PROCNO 1
Date_ 20150503
Time 16.45
INSTRUM spect
PROBHD 5 mm DUL 13C-1
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 32
DS 0
SWH 6410.256 Hz
FIDRES 0.195625 Hz
AQ 2.5559540 sec
RG 4
DW 78.000 usec
DE 6.00 usec
TE 300.0 K
D1 2.00000000 sec
TD0 1

======== CHANNEL f1 ========
NUC1 1H
P1 10.00 usec
PL1 -2.40 dB
SFO1 400.1528010 MHz
SI 16384
SF 400.1500092 MHz
WDW EM
SSB 0
LB 0.00 Hz
GB 0
PC 1.00



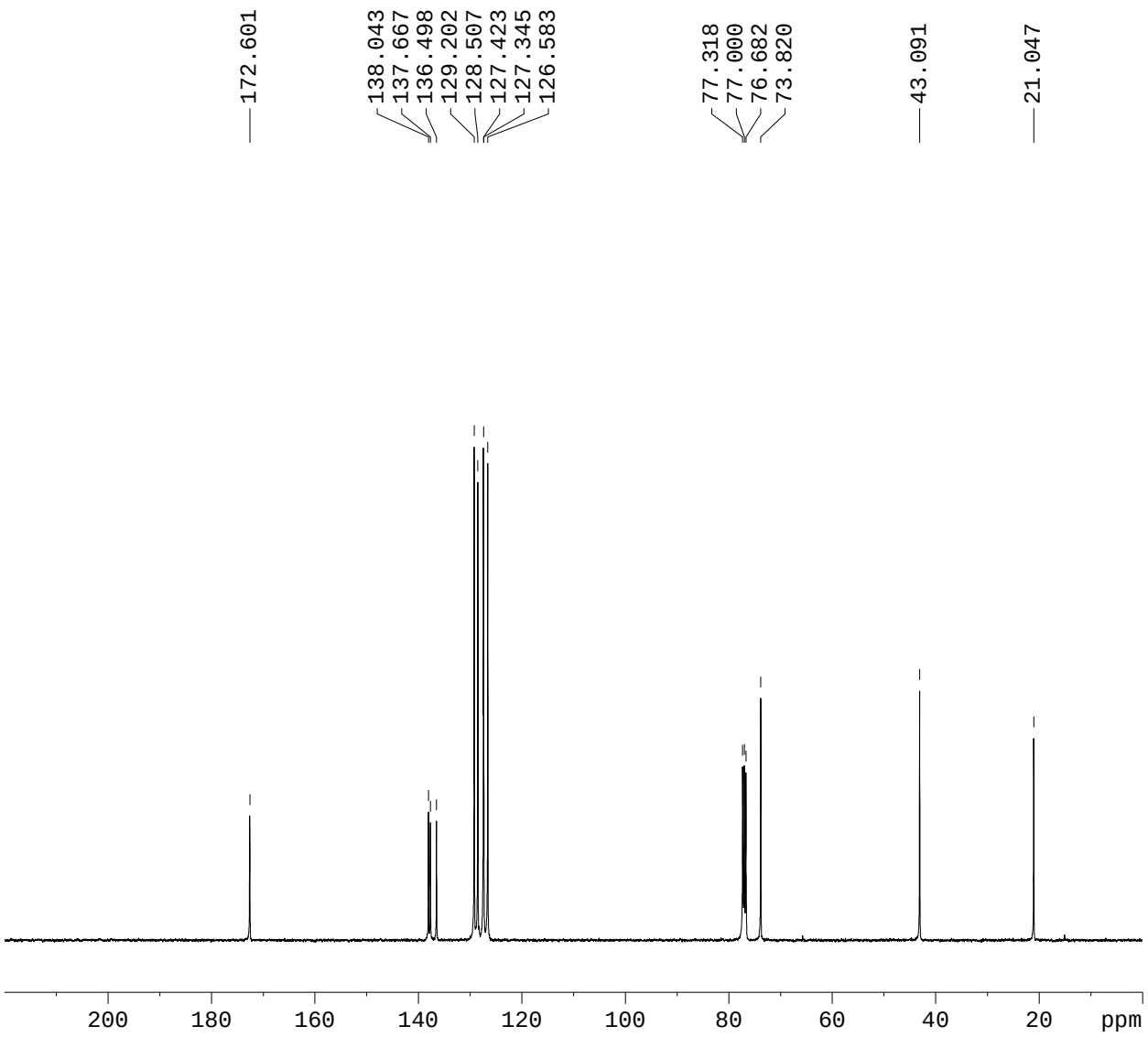
2b

```
NAME        Substrate 13C
EXPNO                   1
PROCNO                  1
Date_            20150503
Time                16.53
INSTRUM             spect
PROBHD   5 mm DUL 13C-1
PULPROG           zgpg30
TD                 65536
SOLVENT            CDCl3
NS                  1512
DS                     0
SWH           22727.273 Hz
FIDRES         0.346791 Hz
AQ            1.4418420 sec
RG                    57
DW                22.000 usec
DE                 6.00 usec
TE                300.0 K
D1           2.00000000 sec
d11          0.03000000 sec
DELTA        1.89999998 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                  13C
P1                   9.70 usec
PL1                 -0.50 dB
SFO1         100.6288660 MHz

======== CHANNEL f2 ========
CPDPRG2           waltz16
NUC2                  1H
PCPD2               90.00 usec
PL2                 -2.40 dB
PL12                15.10 dB
PL13                18.10 dB
SFO2         400.1516010 MHz
SI                 32768
SF           100.6178076 MHz
WDW                   EM
SSB                    0
LB                  3.00 Hz
GB                     0
PC                  1.00
```

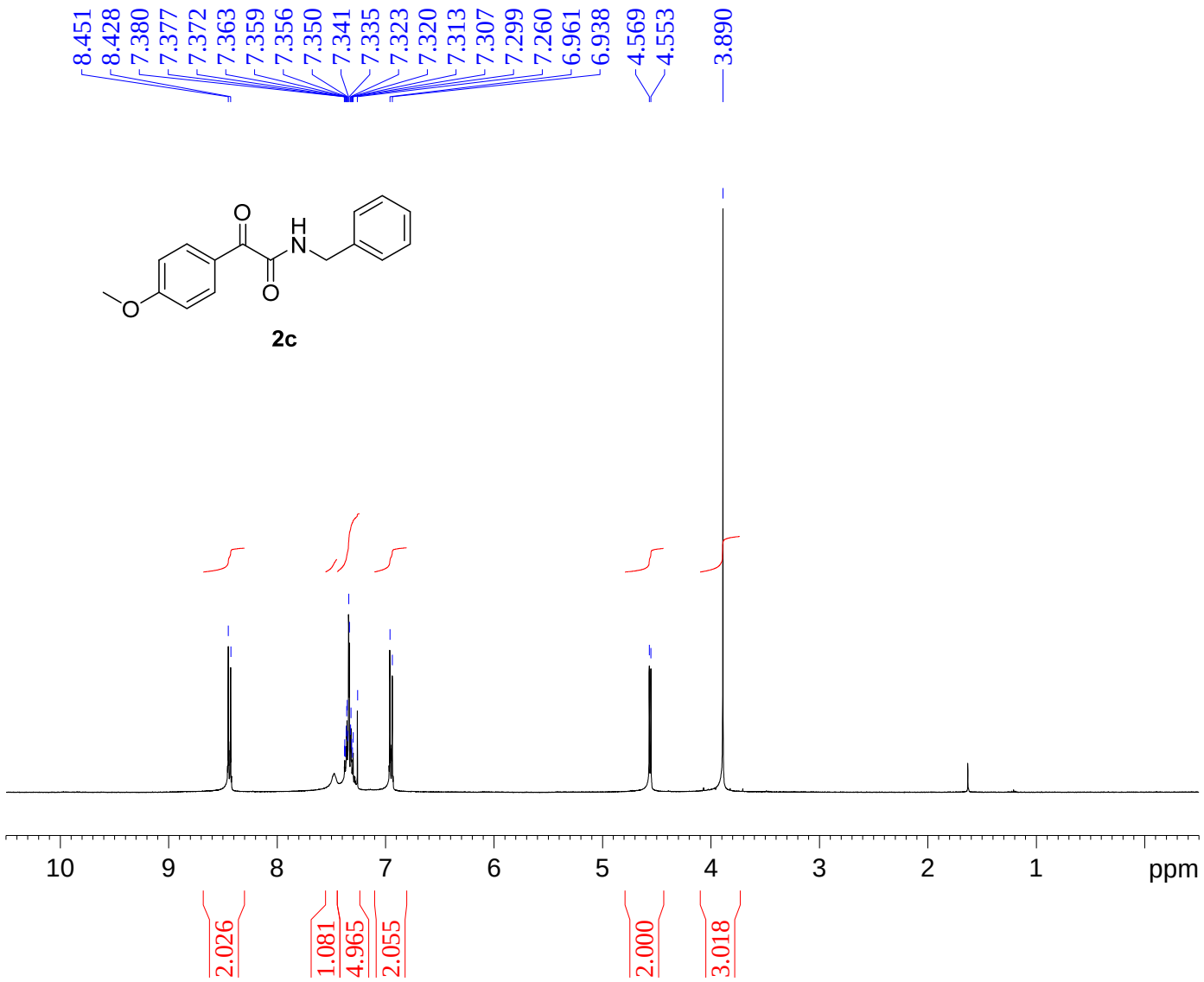


NAME 20151029
EXPNO 1
PROCNO 1
Date_ 20151029
Time 13.48
INSTRUM spect
PROBHD 5 mm DUL 13C-1
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 36
DS 0
SWH 6410.256 Hz
FIDRES 0.195625 Hz
AQ 2.5559540 sec
RG 4
DW 78.000 usec
DE 6.00 usec
TE 300.0 K
D1 2.00000000 sec
TD0 1

======== CHANNEL f1 ========
NUC1 1H
P1 10.00 usec
PL1 -2.40 dB
SFO1 400.1528010 MHz
SI 16384
SF 400.1500088 MHz
WDW EM
SSB 0
LB 0.00 Hz
GB 0
PC 1.00

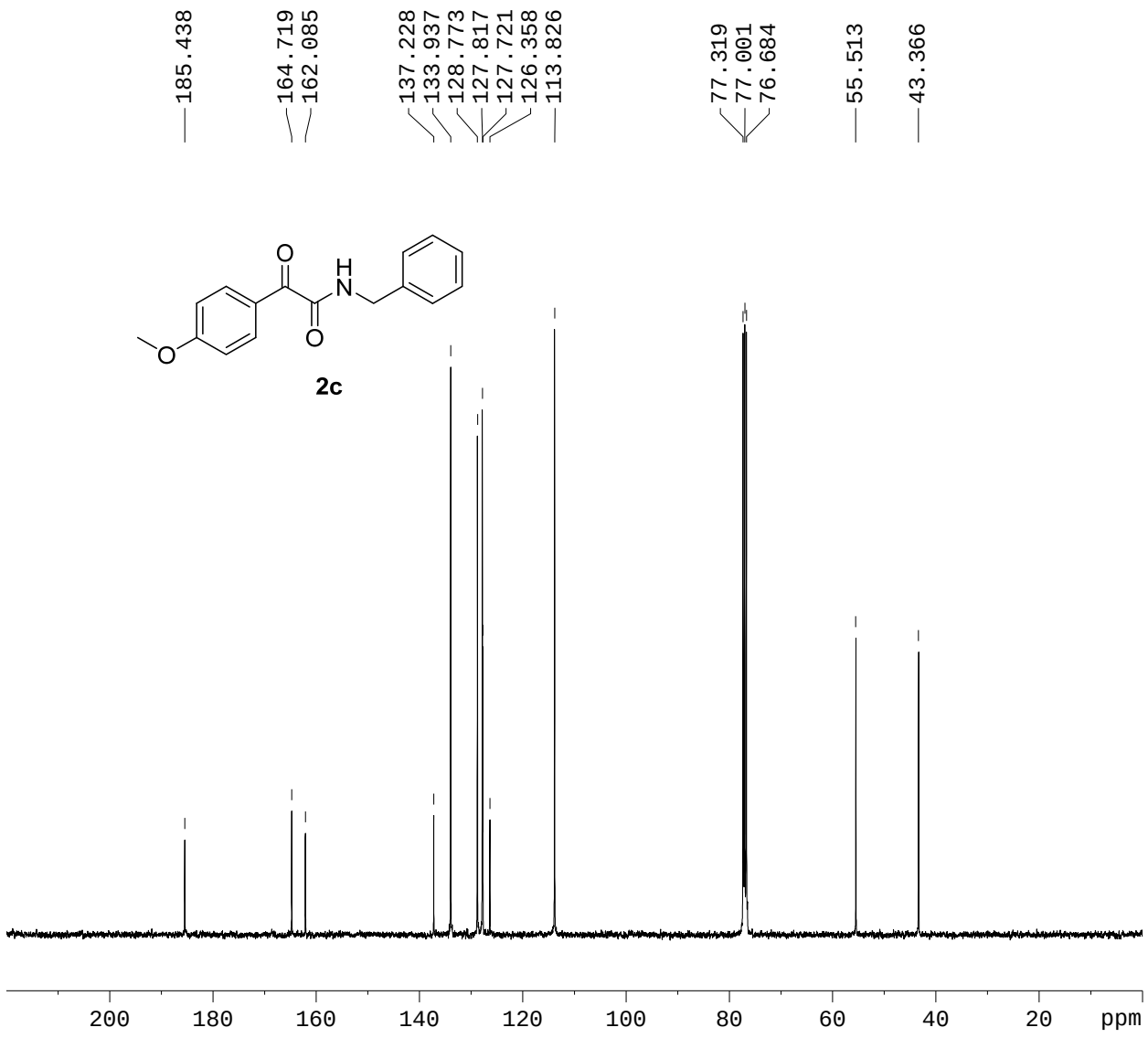


NAME	13C
EXPNO	6
PROCNO	1
Date_	20150321
Time	17.40
INSTRUM	spect
PROBHD	5 mm DUL 13C-1
PULPROG	zgpg30
TD	65536
SOLVENT	CDCl3
NS	1520
DS	0
SWH	22727.273 Hz
FIDRES	0.346791 Hz
AQ	1.4418420 sec
RG	57
DW	22.000 usec
DE	6.00 usec
TE	300.0 K
D1	2.00000000 sec
d11	0.03000000 sec
DELTA	1.89999998 sec
TD0	1
======== CHANNEL f1 ========	
NUC1	13C
P1	9.70 usec
PL1	-0.50 dB
SFO1	100.6288660 MHz
======== CHANNEL f2 ========	
CPDPRG2	waltz16
NUC2	1H
PCPD2	90.00 usec
PL2	-2.40 dB
PL12	15.10 dB
PL13	18.10 dB
SFO2	400.1516010 MHz
SI	32768
SF	100.6178151 MHz
WDW	EM
SSB	0
LB	3.00 Hz
GB	0
PC	1.00

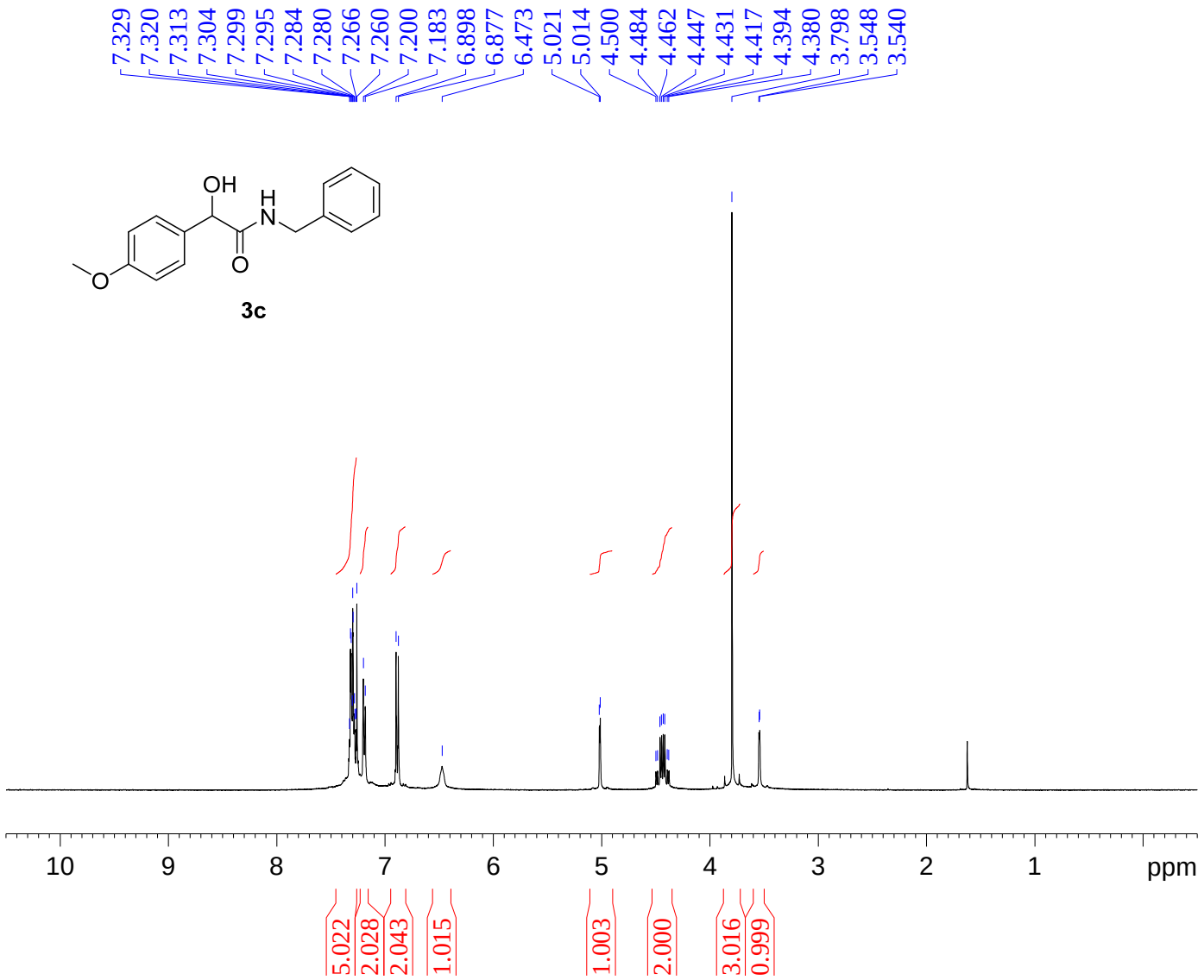


NAME Substrate 1H
EXPNO 11
PROCNO 1
Date_ 20150719
Time 15.26
INSTRUM spect
PROBHD 5 mm DUL 13C-1
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 20
DS 0
SWH 6410.256 Hz
FIDRES 0.195625 Hz
AQ 2.5559540 sec
RG 4
DW 78.000 usec
DE 6.00 usec
TE 300.0 K
D1 2.00000000 sec
TD0 1

======== CHANNEL f1 ========
NUC1 1H
P1 10.00 usec
PL1 -2.40 dB
SFO1 400.1528010 MHz
SI 16384
SF 400.1500088 MHz
WDW EM
SSB 0
LB 0.00 Hz
GB 0
PC 1.00

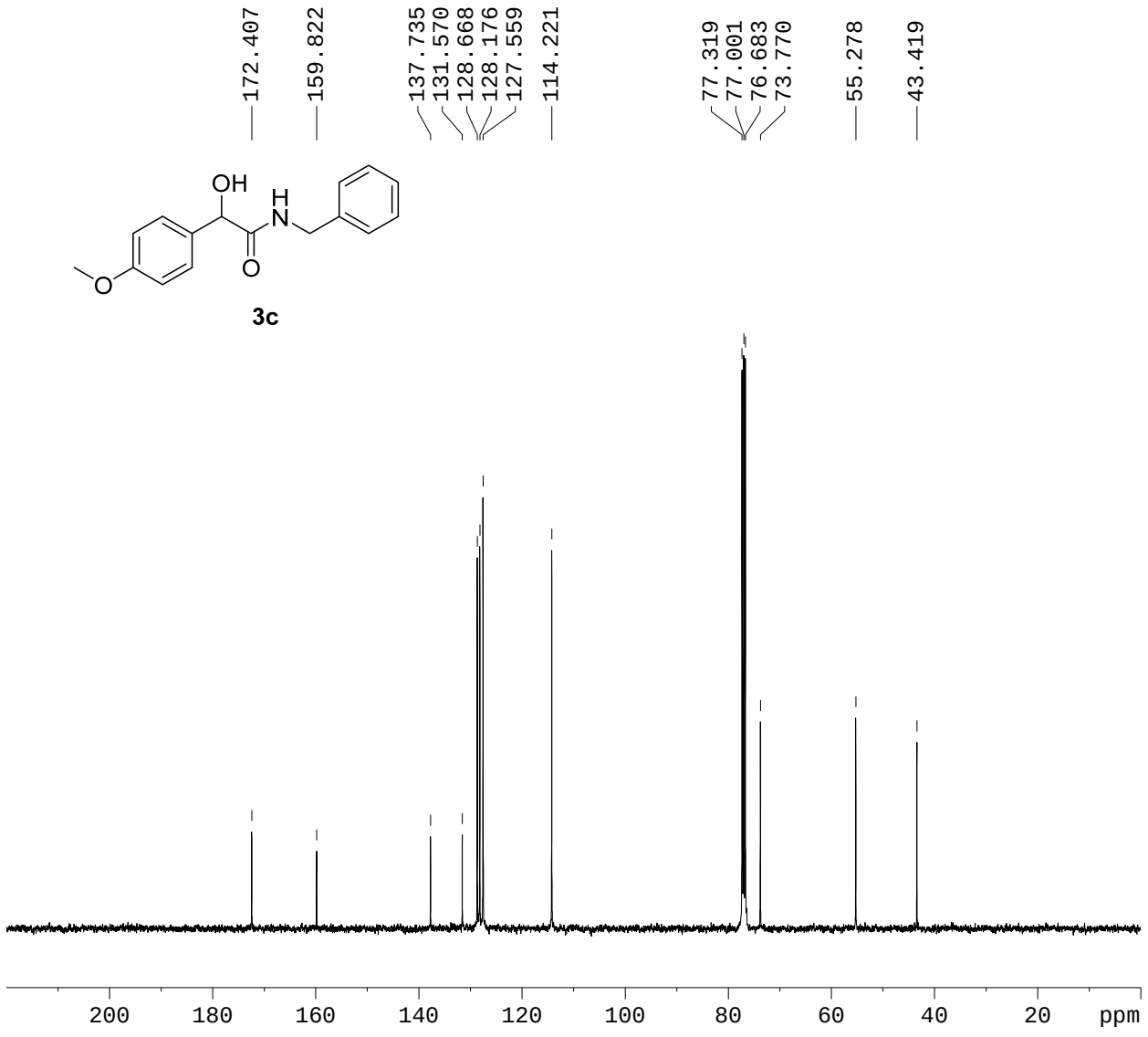



```
NAME       Substrate 13C
EXPNO                 11
PROCNO                 1
Date_           20150719
Time               15.31
INSTRUM            spect
PROBHD   5 mm DUL 13C-1
PULPROG          zgpg30
TD                65536
SOLVENT           CDCl3
NS                 1191
DS                    0
SWH           22727.273 Hz
FIDRES         0.346791 Hz
AQ             1.4418420 sec
RG                   57
DW                22.000 usec
DE                 6.00 usec
TE                300.0 K
D1            2.00000000 sec
d11           0.03000000 sec
DELTA         1.89999998 sec
TD0                   1

======== CHANNEL f1 ========
NUC1                 13C
P1                  9.70 usec
PL1                -0.50 dB
SFO1        100.6288660 MHz

======== CHANNEL f2 ========
CPDPRG2          waltz16
NUC2                 1H
PCPD2              90.00 usec
PL2                -2.40 dB
PL12               15.10 dB
PL13               18.10 dB
SFO2        400.1516010 MHz
SI                 32768
SF          100.6178033 MHz
WDW                  EM
SSB                   0
LB                 3.00 Hz
GB                    0
PC                 1.00
```

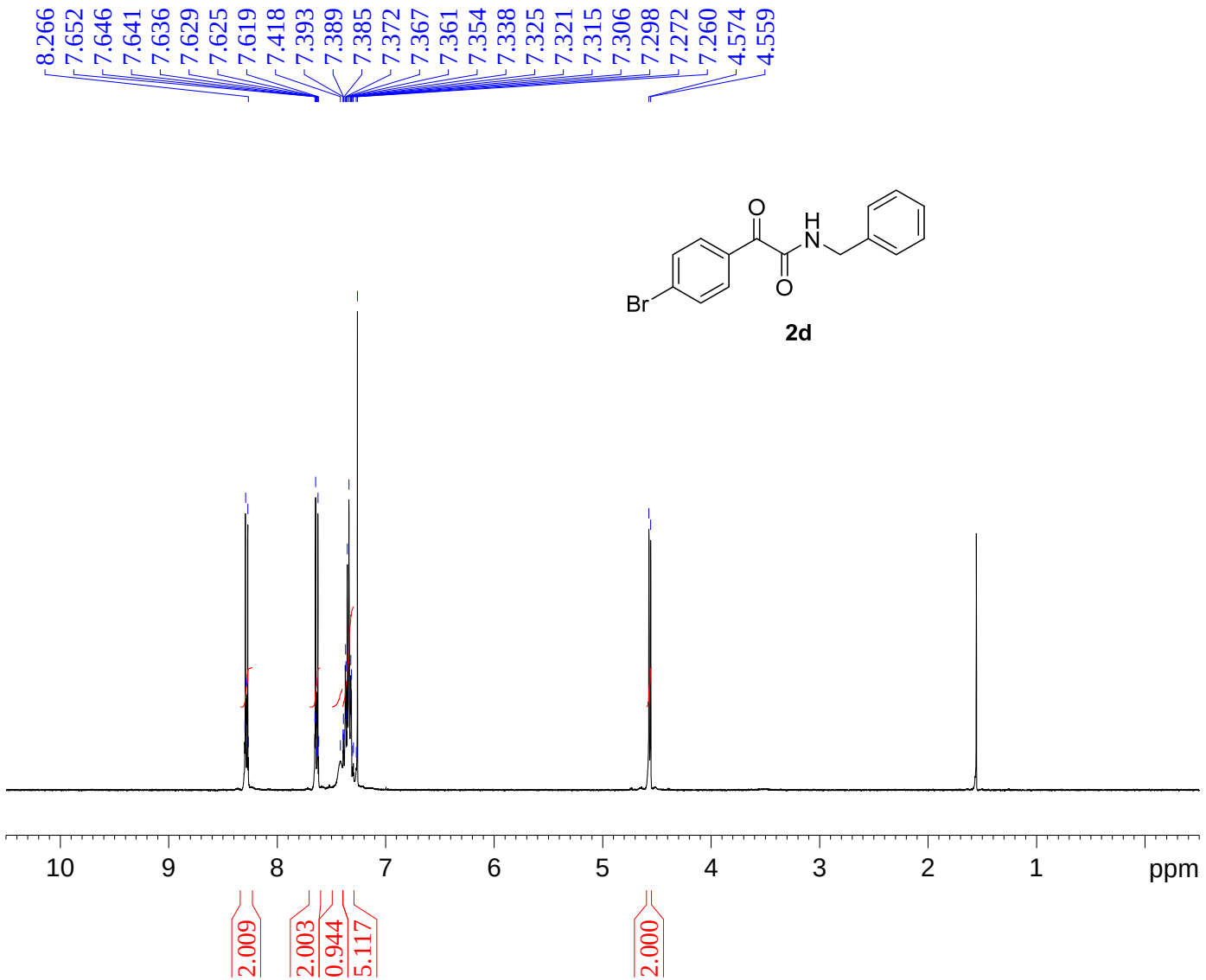


NAME Substrate 1H
EXPNO 12
PROCNO 1
Date_ 20150720
Time 20.11
INSTRUM spect
PROBHD 5 mm DUL 13C-1
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 32
DS 0
SWH 6410.256 Hz
FIDRES 0.195625 Hz
AQ 2.5559540 sec
RG 4
DW 78.000 usec
DE 6.00 usec
TE 300.0 K
D1 2.00000000 sec
TD0 1

======== CHANNEL f1 ========
NUC1 1H
P1 10.00 usec
PL1 -2.40 dB
SFO1 400.1528010 MHz
SI 16384
SF 400.1500092 MHz
WDW EM
SSB 0
LB 0.00 Hz
GB 0
PC 1.00

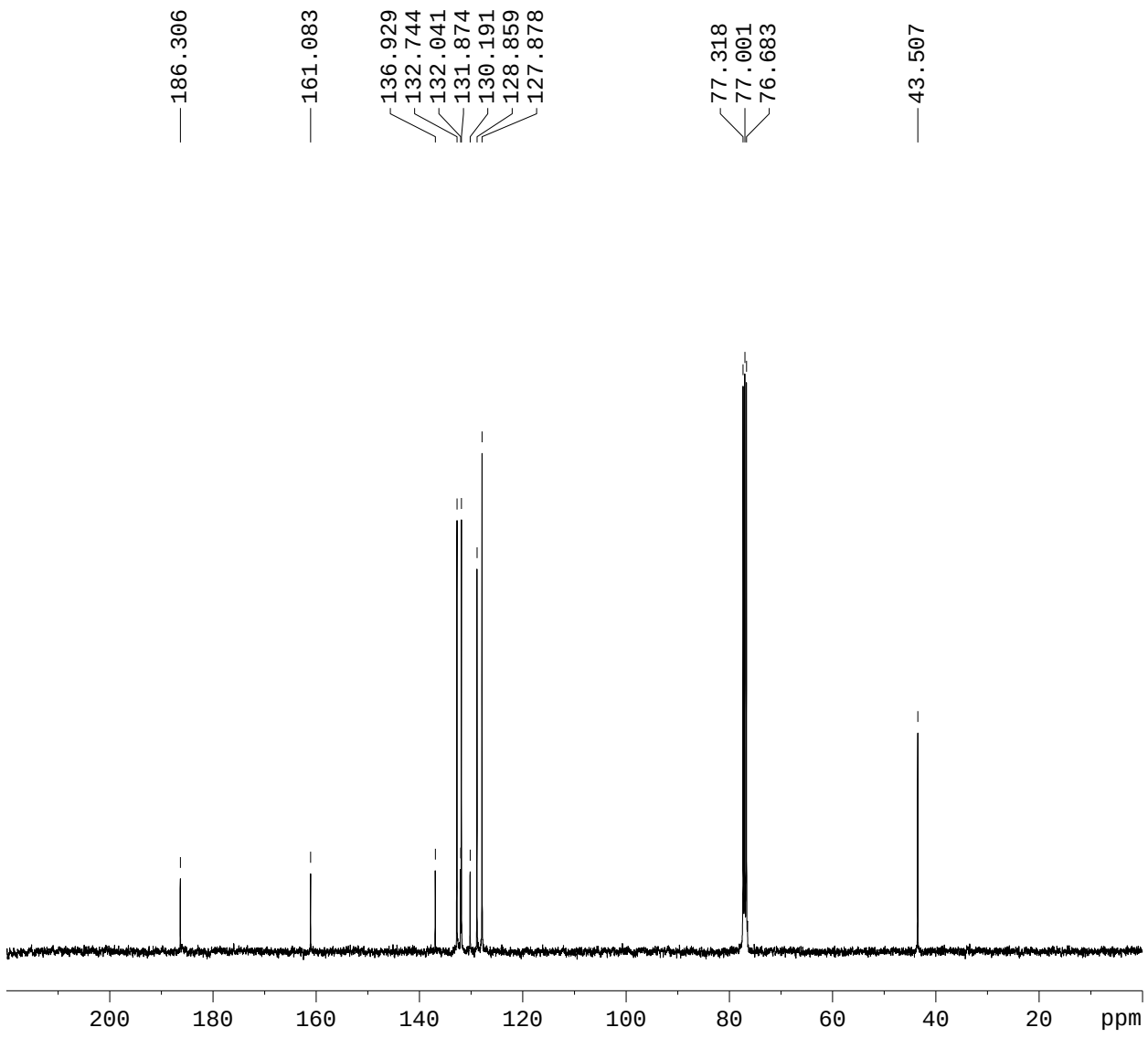



```
NAME       Substrate 13C
EXPNO                 12
PROCNO                 1
Date_           20150720
Time               20.16
INSTRUM            spect
PROBHD   5 mm DUL 13C-1
PULPROG          zgpg30
TD                 65536
SOLVENT            CDCl3
NS                   734
DS                     0
SWH           22727.273 Hz
FIDRES         0.346791 Hz
AQ            1.4418420 sec
RG                    57
DW                22.000 usec
DE                 6.00 usec
TE                300.0 K
D1           2.00000000 sec
d11          0.03000000 sec
DELTA        1.89999998 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                 13C
P1                  9.70 usec
PL1                -0.50 dB
SFO1        100.6288660 MHz

======== CHANNEL f2 ========
CPDPRG2          waltz16
NUC2                 1H
PCPD2              90.00 usec
PL2                -2.40 dB
PL12               15.10 dB
PL13               18.10 dB
SFO2        400.1516010 MHz
SI                 32768
SF          100.6178029 MHz
WDW                  EM
SSB                   0
LB                 3.00 Hz
GB                    0
PC                 1.00
```

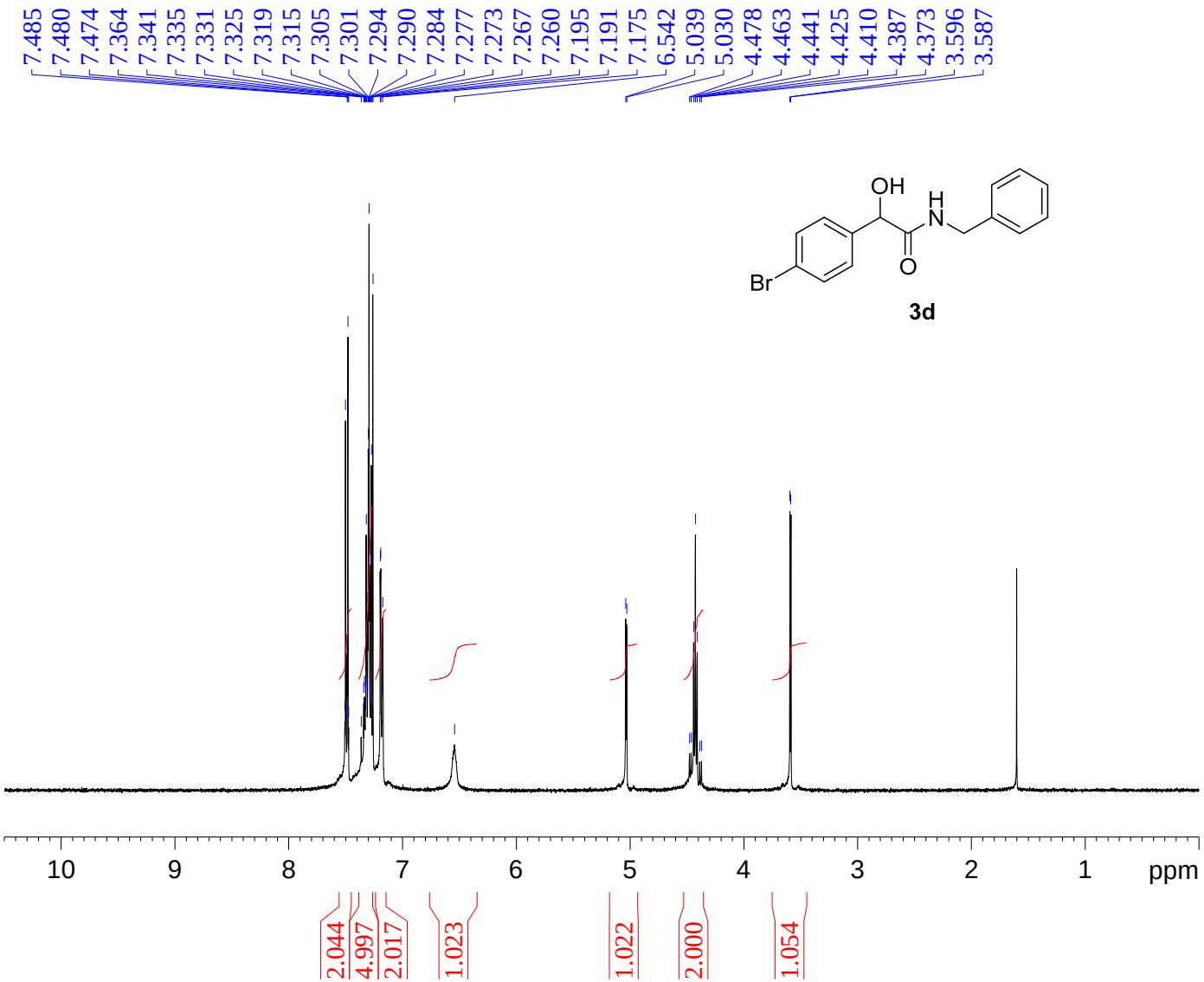


NAME Substrate 1H
EXPNO 14
PROCNO 1
Date_ 20150723
Time 20.25
INSTRUM spect
PROBHD 5 mm DUL 13C-1
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 44
DS 0
SWH 6410.256 Hz
FIDRES 0.195625 Hz
AQ 2.5559540 sec
RG 4
DW 78.000 usec
DE 6.00 usec
TE 300.0 K
D1 2.00000000 sec
TD0 1

======== CHANNEL f1 ========
NUC1 1H
P1 10.00 usec
PL1 -2.40 dB
SFO1 400.1528010 MHz
SI 16384
SF 400.1500088 MHz
WDW EM
SSB 0
LB 0.00 Hz
GB 0
PC 1.00

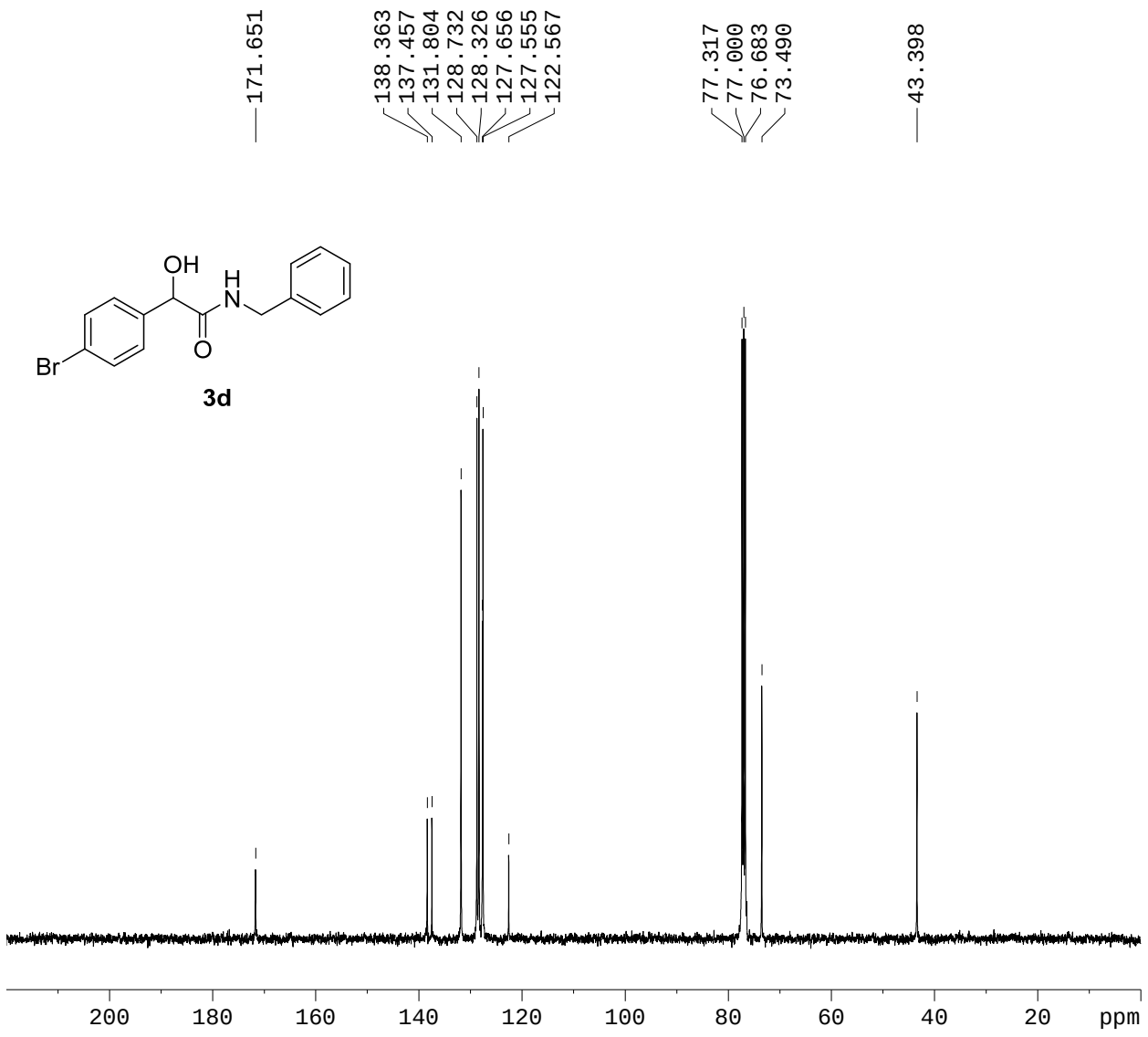


NAME	Substrate 13C	
EXPNO	14	
PROCNO	1	
Date_	20150723	
Time	20.31	
INSTRUM	spect	
PROBHD	5 mm DUL 13C-1	
PULPROG	zgpg30	
TD	65536	
SOLVENT	CDCl3	
NS	496	
DS	0	
SWH	22727.273	Hz
FIDRES	0.346791	Hz
AQ	1.4418420	sec
RG	57	
DW	22.000	usec
DE	6.00	usec
TE	300.0	K
D1	2.00000000	sec
d11	0.03000000	sec
DELTA	1.89999998	sec
TD0	1	
======== CHANNEL f1 ========		
NUC1	13C	
P1	9.70	usec
PL1	-0.50	dB
SFO1	100.6288660	MHz
======== CHANNEL f2 ========		
CPDPRG2	waltz16	
NUC2	1H	
PCPD2	90.00	usec
PL2	-2.40	dB
PL12	15.10	dB
PL13	18.10	dB
SFO2	400.1516010	MHz
SI	32768	
SF	100.6178020	MHz
WDW	EM	
SSB	0	
LB	3.00	Hz
GB	0	
PC	1.00	

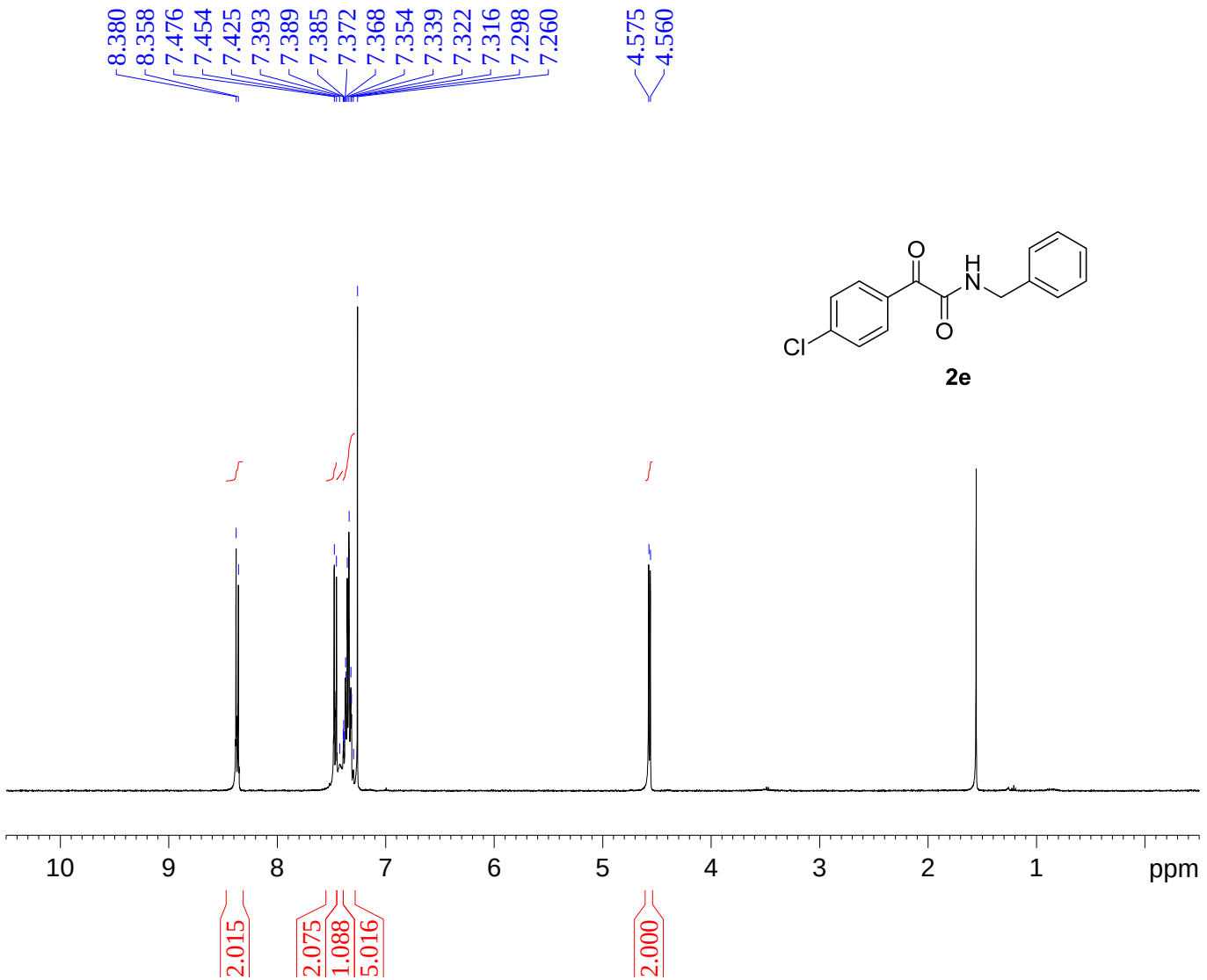


NAME Substrate 1H
EXPNO 13
PROCNO 1
Date_ 20150721
Time 19.52
INSTRUM spect
PROBHD 5 mm DUL 13C-1
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 16
DS 0
SWH 6410.256 Hz
FIDRES 0.195625 Hz
AQ 2.5559540 sec
RG 4
DW 78.000 usec
DE 6.00 usec
TE 300.0 K
D1 2.00000000 sec
TD0 1

======== CHANNEL f1 ========
NUC1 1H
P1 10.00 usec
PL1 -2.40 dB
SFO1 400.1528010 MHz
SI 16384
SF 400.1500088 MHz
WDW EM
SSB 0
LB 0.00 Hz
GB 0
PC 1.00

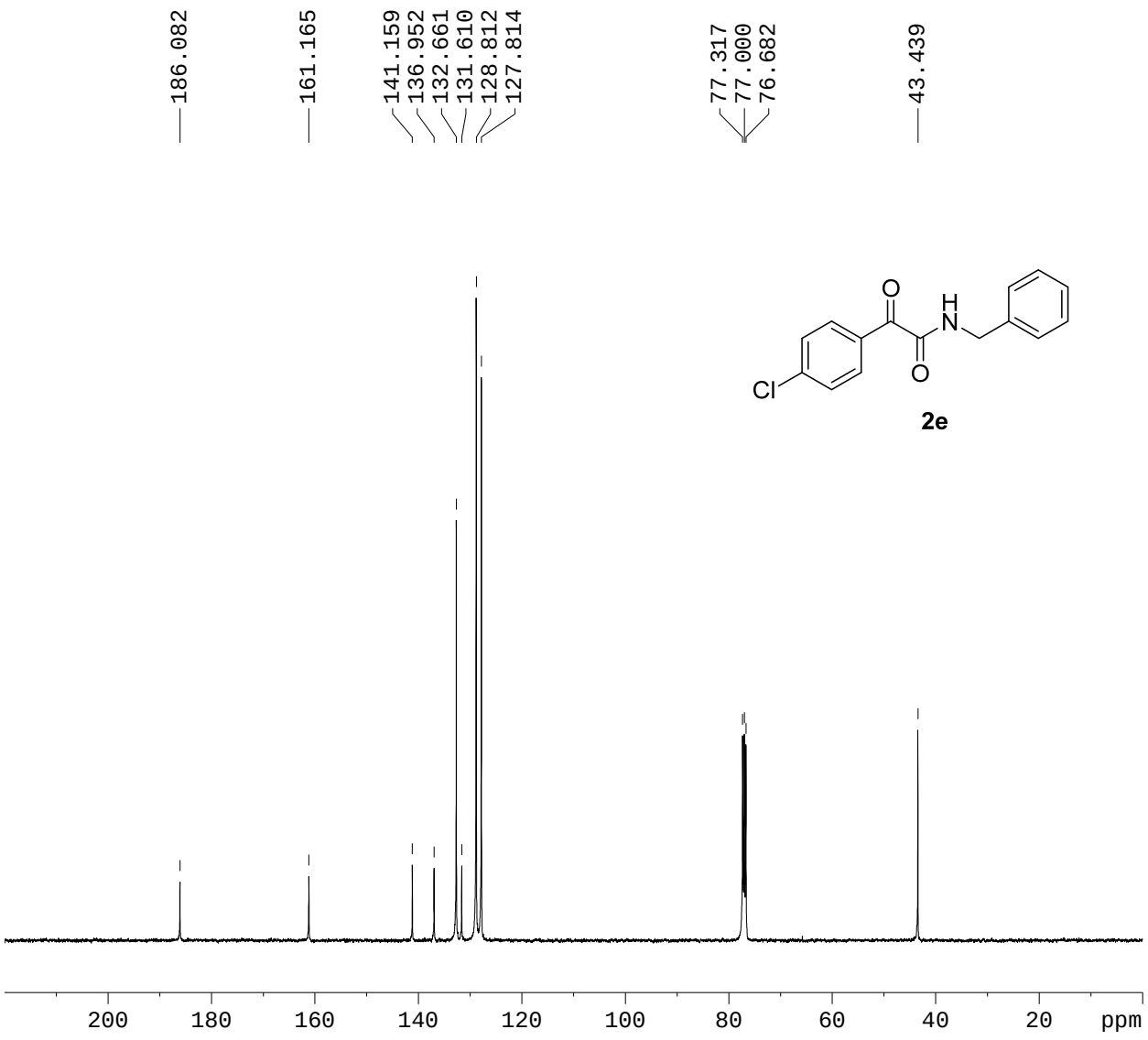



```
NAME       Substrate 13C
EXPNO                 13
PROCNO                 1
Date_           20150721
Time               19.56
INSTRUM            spect
PROBHD   5 mm DUL 13C-1
PULPROG          zgpg30
TD                 65536
SOLVENT            CDCl3
NS                   816
DS                     0
SWH           22727.273 Hz
FIDRES         0.346791 Hz
AQ             1.4418420 sec
RG                    57
DW                22.000 usec
DE                 6.00 usec
TE                300.0 K
D1             2.00000000 sec
d11            0.03000000 sec
DELTA          1.89999998 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                 13C
P1                  9.70 usec
PL1                -0.50 dB
SFO1         100.6288660 MHz

======== CHANNEL f2 ========
CPDPRG2          waltz16
NUC2                 1H
PCPD2              90.00 usec
PL2                -2.40 dB
PL12               15.10 dB
PL13               18.10 dB
SFO2         400.1516010 MHz
SI                 32768
SF           100.6178030 MHz
WDW                  EM
SSB                   0
LB                 3.00 Hz
GB                    0
PC                 1.00
```

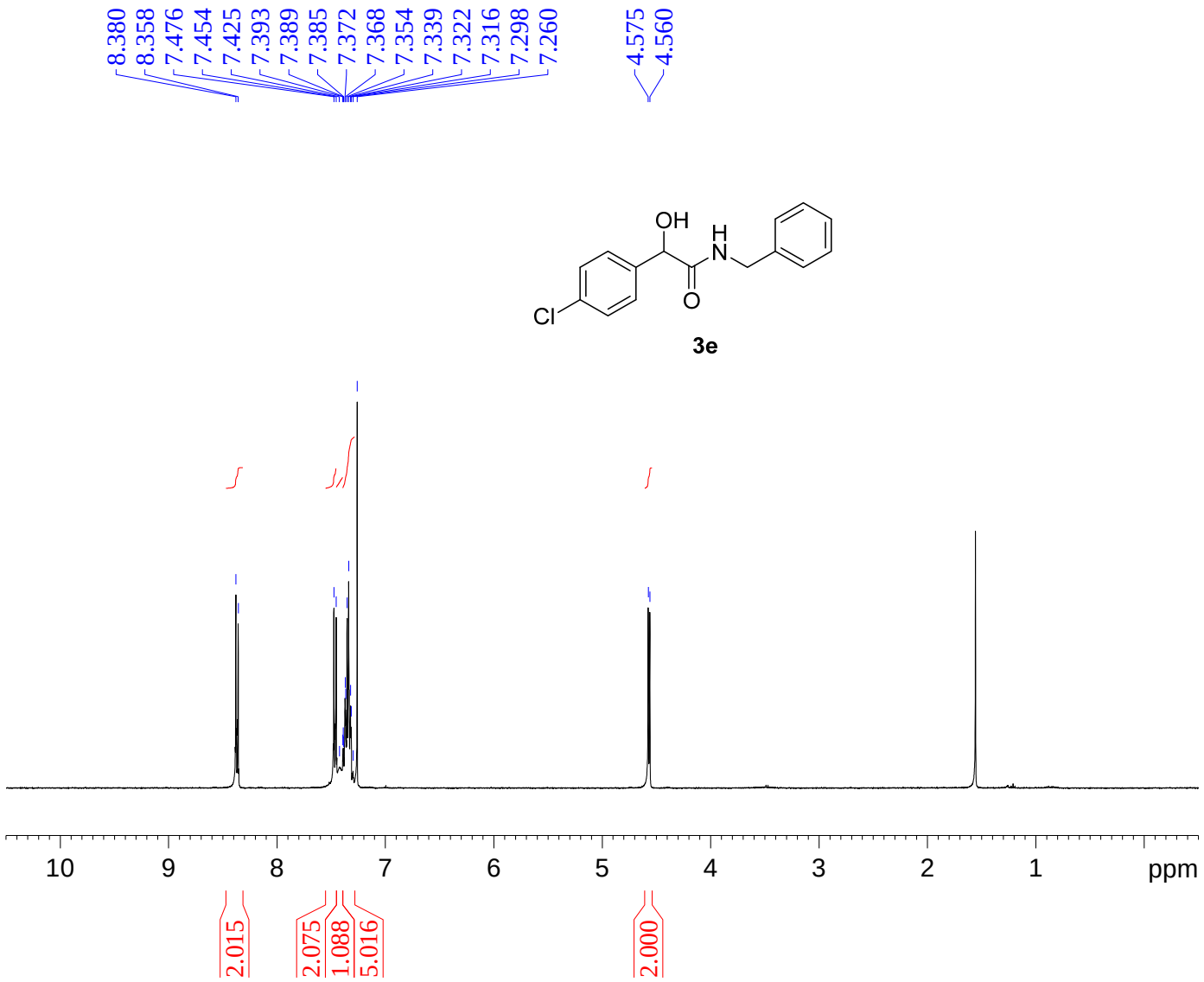


NAME 20151029
EXPNO 2
PROCNO 1
Date_ 20151029
Time 13.54
INSTRUM spect
PROBHD 5 mm DUL 13C-1
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 71
DS 0
SWH 6410.256 Hz
FIDRES 0.195625 Hz
AQ 2.5559540 sec
RG 4
DW 78.000 usec
DE 6.00 usec
TE 300.0 K
D1 2.00000000 sec
TD0 1

======== CHANNEL f1 ========
NUC1 1H
P1 10.00 usec
PL1 -2.40 dB
SFO1 400.1528010 MHz
SI 16384
SF 400.1500088 MHz
WDW EM
SSB 0
LB 0.00 Hz
GB 0
PC 1.00

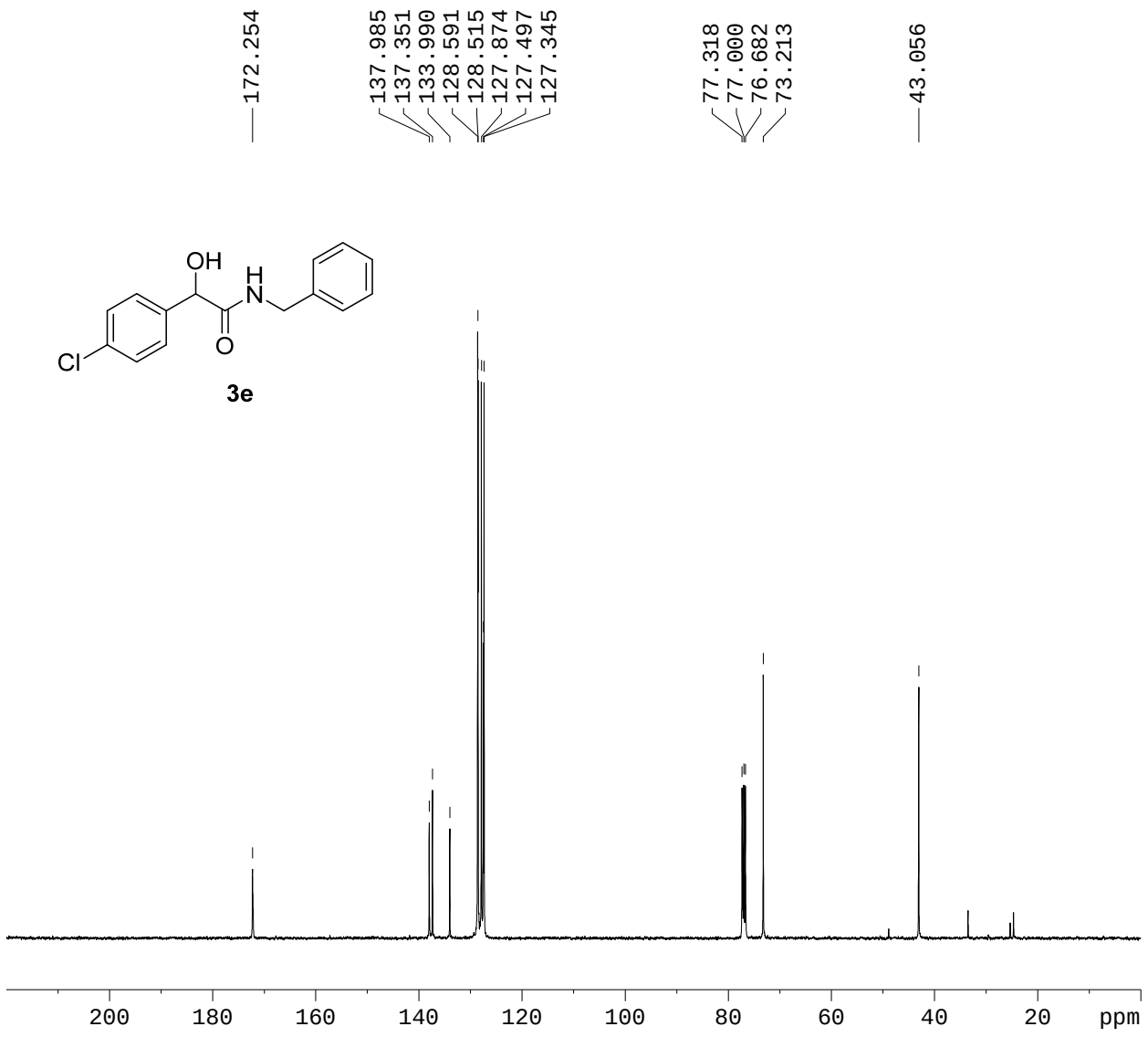


NAME	Substrate
EXPNO	2
PROCNO	1
Date_	20150208
Time	18.15
INSTRUM	spect
PROBHD	5 mm DUL 13C-1
PULPROG	zgpg30
TD	65536
SOLVENT	CDCl3
NS	1637
DS	0
SWH	22727.273 Hz
FIDRES	0.346791 Hz
AQ	1.4418420 sec
RG	57
DW	22.000 usec
DE	6.00 usec
TE	300.0 K
D1	2.00000000 sec
d11	0.03000000 sec
DELTA	1.89999998 sec
TD0	1
======== CHANNEL f1 ========	
NUC1	13C
P1	9.70 usec
PL1	-0.50 dB
SFO1	100.6288660 MHz
======== CHANNEL f2 ========	
CPDPRG2	waltz16
NUC2	1H
PCPD2	90.00 usec
PL2	-2.40 dB
PL12	15.10 dB
PL13	18.10 dB
SFO2	400.1516010 MHz
SI	32768
SF	100.6178062 MHz
WDW	EM
SSB	0
LB	3.00 Hz
GB	0
PC	1.00

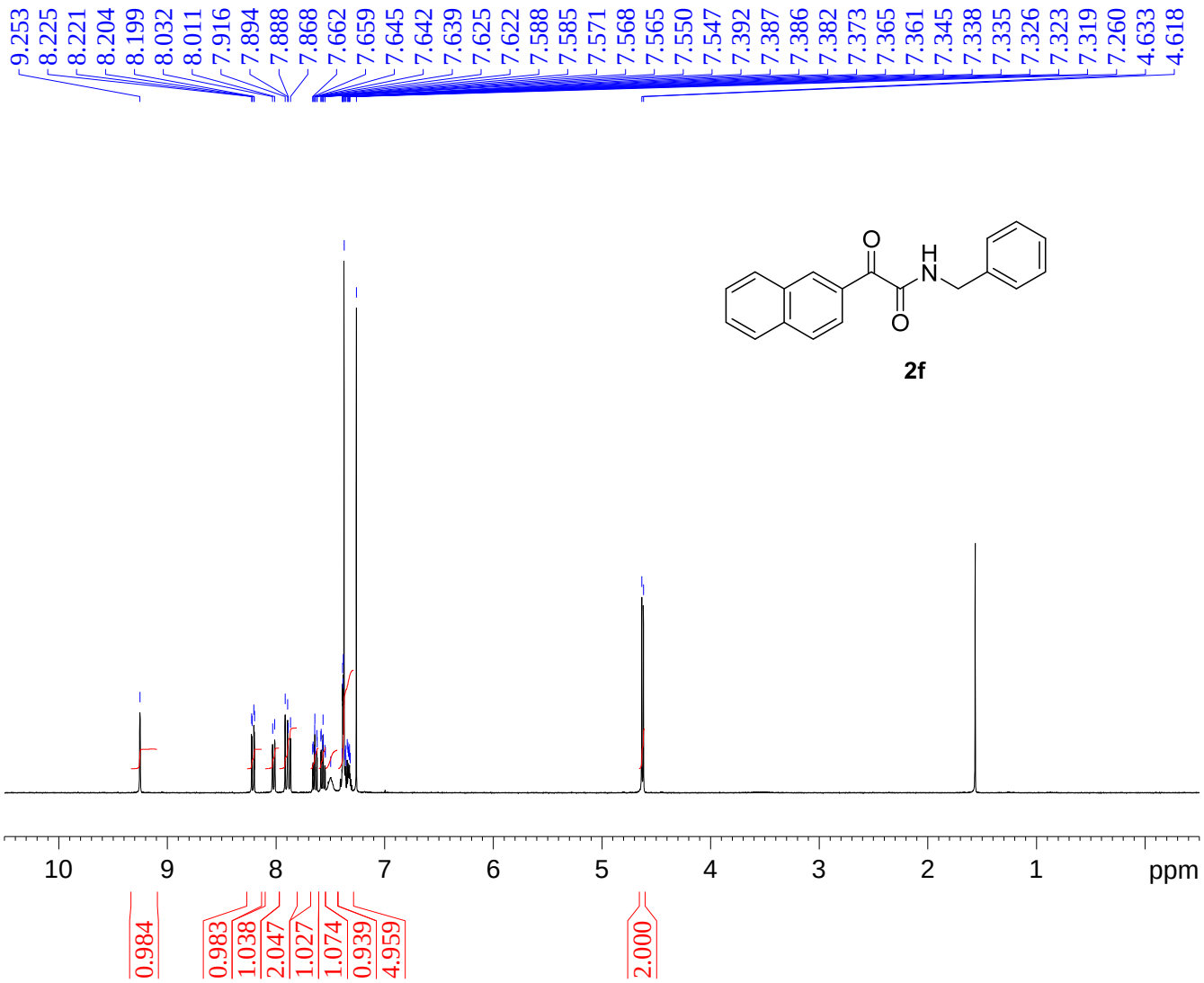


NAME 20151029
EXPNO 2
PROCNO 1
Date_ 20151029
Time 13.54
INSTRUM spect
PROBHD 5 mm DUL 13C-1
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 71
DS 0
SWH 6410.256 Hz
FIDRES 0.195625 Hz
AQ 2.5559540 sec
RG 4
DW 78.000 usec
DE 6.00 usec
TE 300.0 K
D1 2.00000000 sec
TD0 1

======== CHANNEL f1 ========
NUC1 1H
P1 10.00 usec
PL1 -2.40 dB
SFO1 400.1528010 MHz
SI 16384
SF 400.1500088 MHz
WDW EM
SSB 0
LB 0.00 Hz
GB 0
PC 1.00

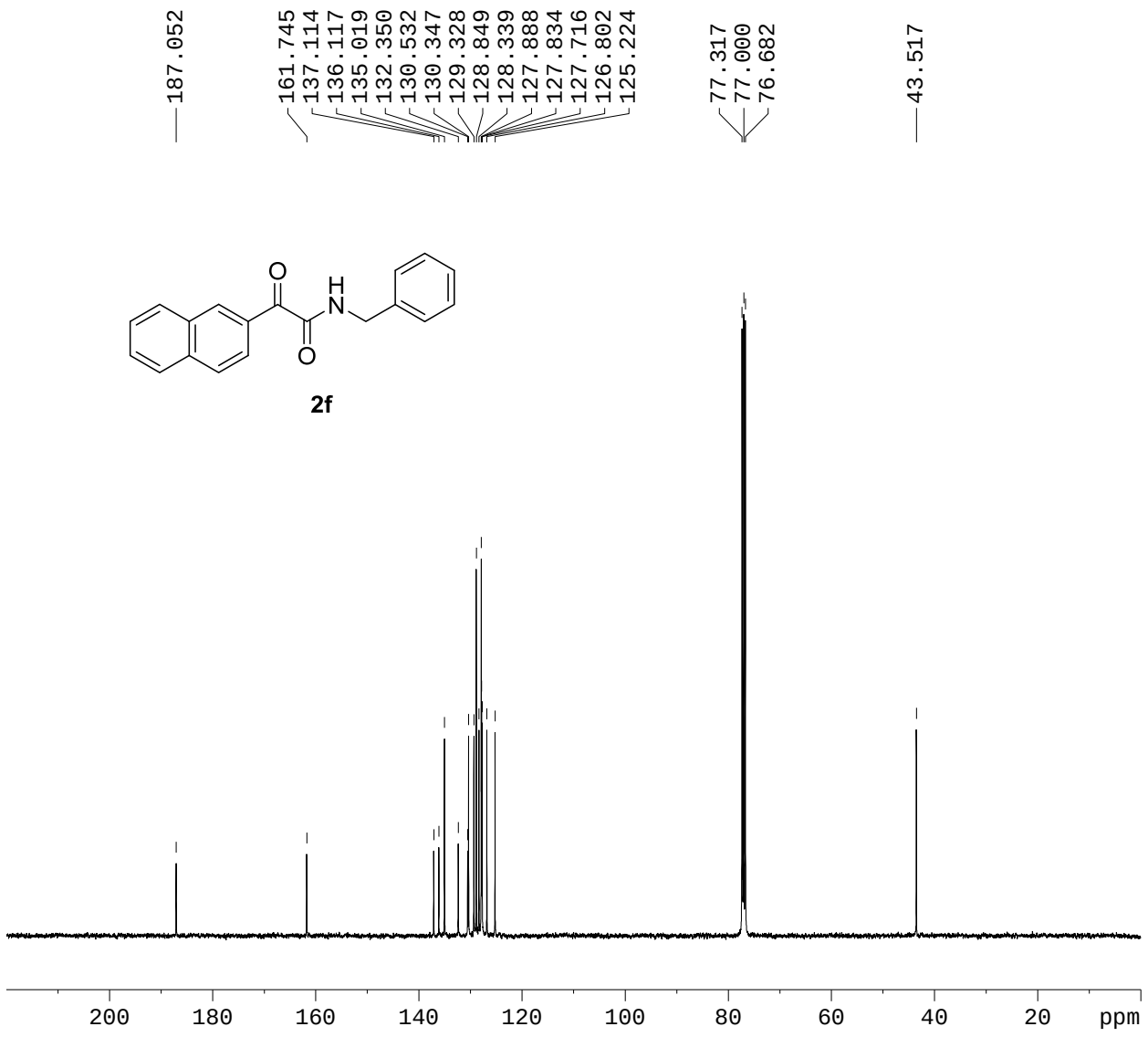


NAME	13C
EXPNO	3
PROCNO	1
Date_	20150125
Time	18.17
INSTRUM	spect
PROBHD	5 mm DUL 13C-1
PULPROG	zgpg30
TD	65536
SOLVENT	CDCl3
NS	1157
DS	0
SWH	22727.273 Hz
FIDRES	0.346791 Hz
AQ	1.4418420 sec
RG	57
DW	22.000 usec
DE	6.00 usec
TE	300.0 K
D1	2.00000000 sec
d11	0.03000000 sec
DELTA	1.89999998 sec
TD0	1
======== CHANNEL f1 ========	
NUC1	13C
P1	9.70 usec
PL1	-0.50 dB
SFO1	100.6288660 MHz
======== CHANNEL f2 ========	
CPDPRG2	waltz16
NUC2	1H
PCPD2	90.00 usec
PL2	-2.40 dB
PL12	15.10 dB
PL13	18.10 dB
SFO2	400.1516010 MHz
SI	32768
SF	100.6178146 MHz
WDW	EM
SSB	0
LB	3.00 Hz
GB	0
PC	1.00



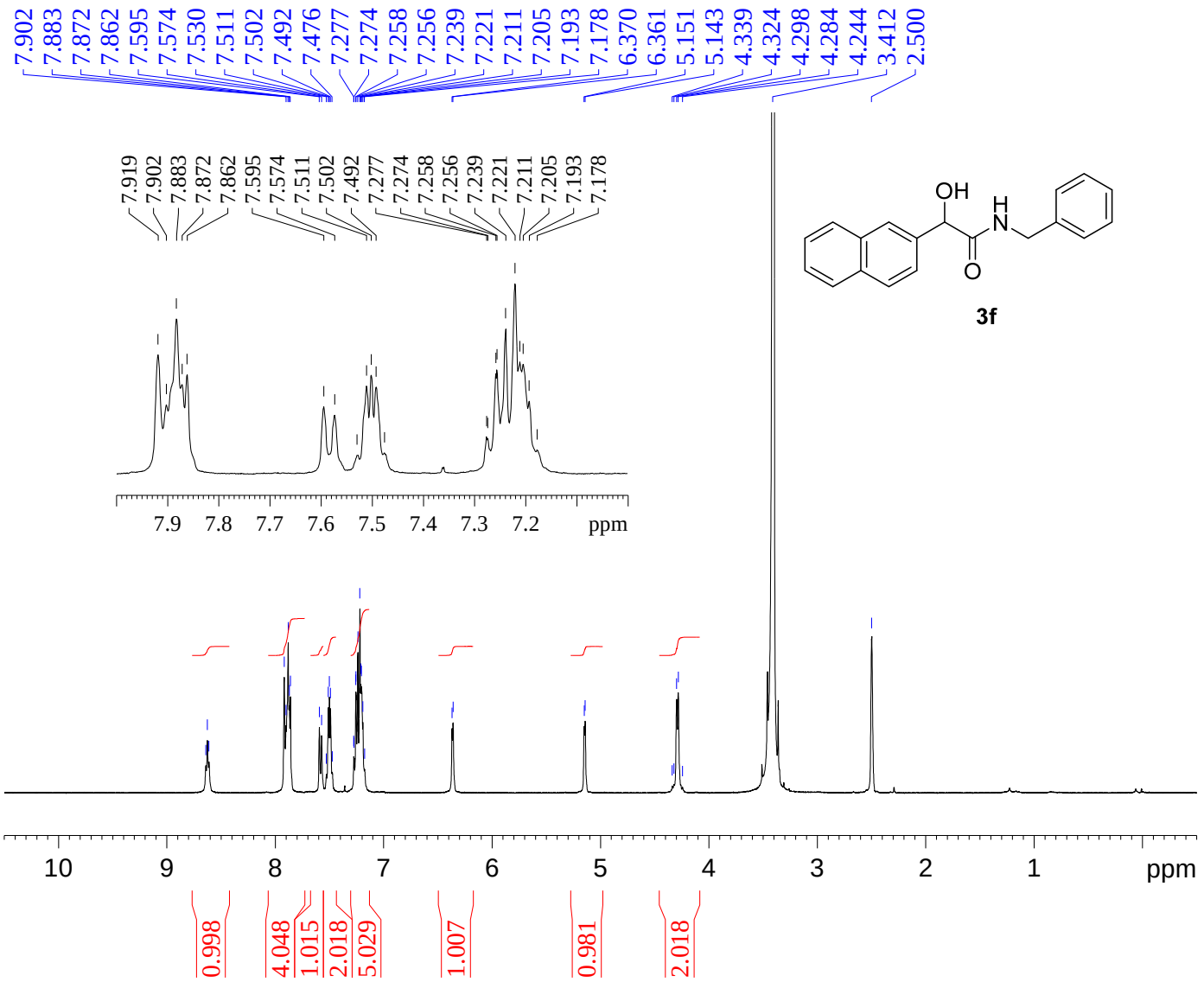
NAME Substrate 1H
EXPNO 4
PROCNO 1
Date_ 20150613
Time 16.55
INSTRUM spect
PROBHD 5 mm DUL 13C-1
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 44
DS 0
SWH 6410.256 Hz
FIDRES 0.195625 Hz
AQ 2.5559540 sec
RG 4
DW 78.000 usec
DE 6.00 usec
TE 300.0 K
D1 2.00000000 sec
TD0 1

======== CHANNEL f1 ========
NUC1 1H
P1 10.00 usec
PL1 -2.40 dB
SFO1 400.1528010 MHz
SI 16384
SF 400.1500088 MHz
WDW EM
SSB 0
LB 0.00 Hz
GB 0
PC 1.00



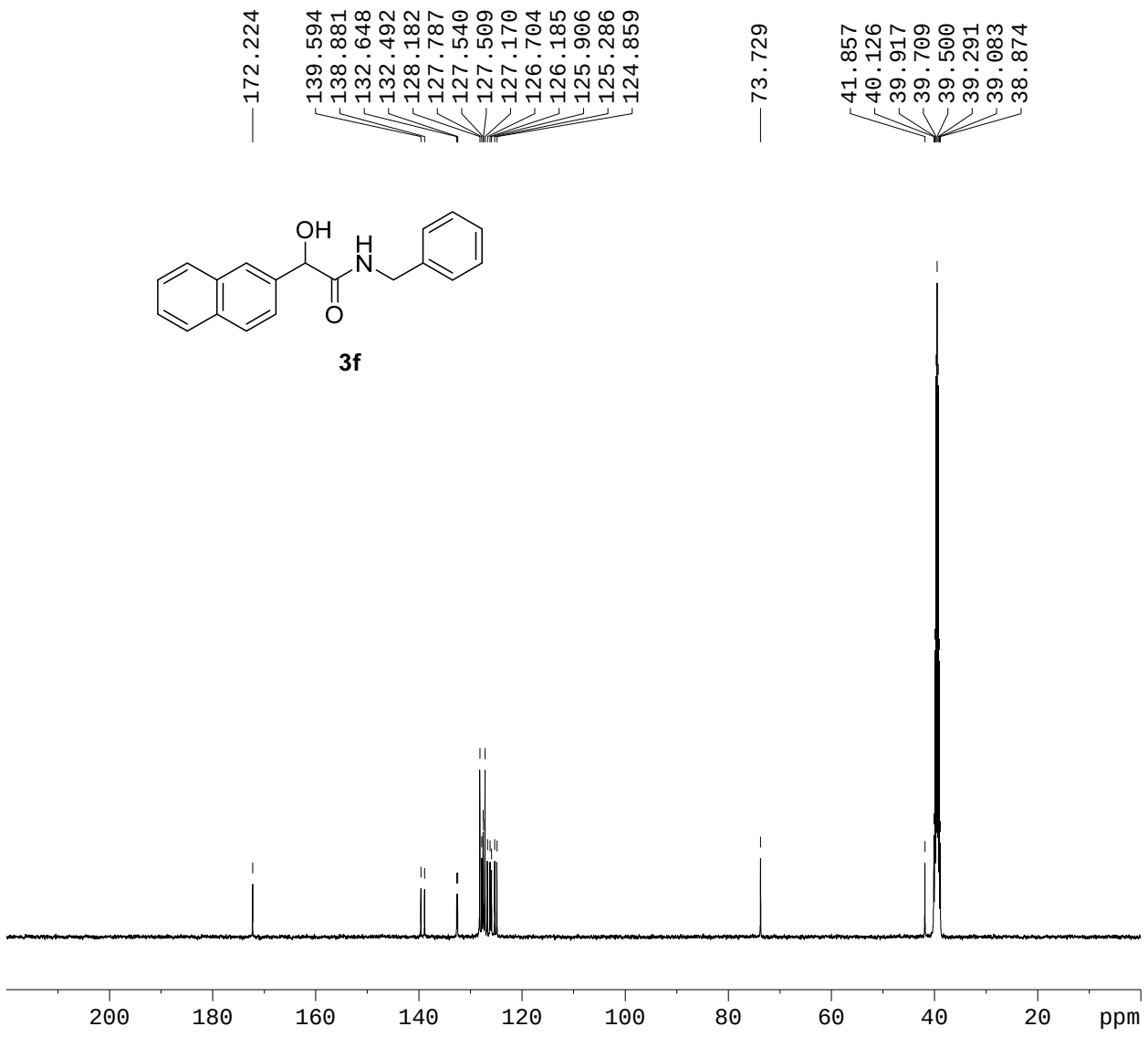
2f

```
NAME       Substrate 13C
EXPNO                 4
PROCNO                1
Date_          20150613
Time              17.03
INSTRUM           spect
PROBHD   5 mm DUL 13C-1
PULPROG          zgpg30
TD                65536
SOLVENT           CDCl3
NS                 1880
DS                    0
SWH           22727.273 Hz
FIDRES         0.346791 Hz
AQ             1.4418420 sec
RG                   57
DW                22.000 usec
DE                 6.00 usec
TE                300.0 K
D1           2.00000000 sec
d11          0.03000000 sec
DELTA        1.89999998 sec
TD0                   1

======== CHANNEL f1 ========
NUC1                 13C
P1                  9.70 usec
PL1                -0.50 dB
SFO1        100.6288660 MHz

======== CHANNEL f2 ========
CPDPRG2          waltz16
NUC2                 1H
PCPD2              90.00 usec
PL2                -2.40 dB
PL12               15.10 dB
PL13               18.10 dB
SFO2        400.1516010 MHz
SI                 32768
SF          100.6178023 MHz
WDW                  EM
SSB                   0
LB                 3.00 Hz
GB                    0
PC                 1.00
```

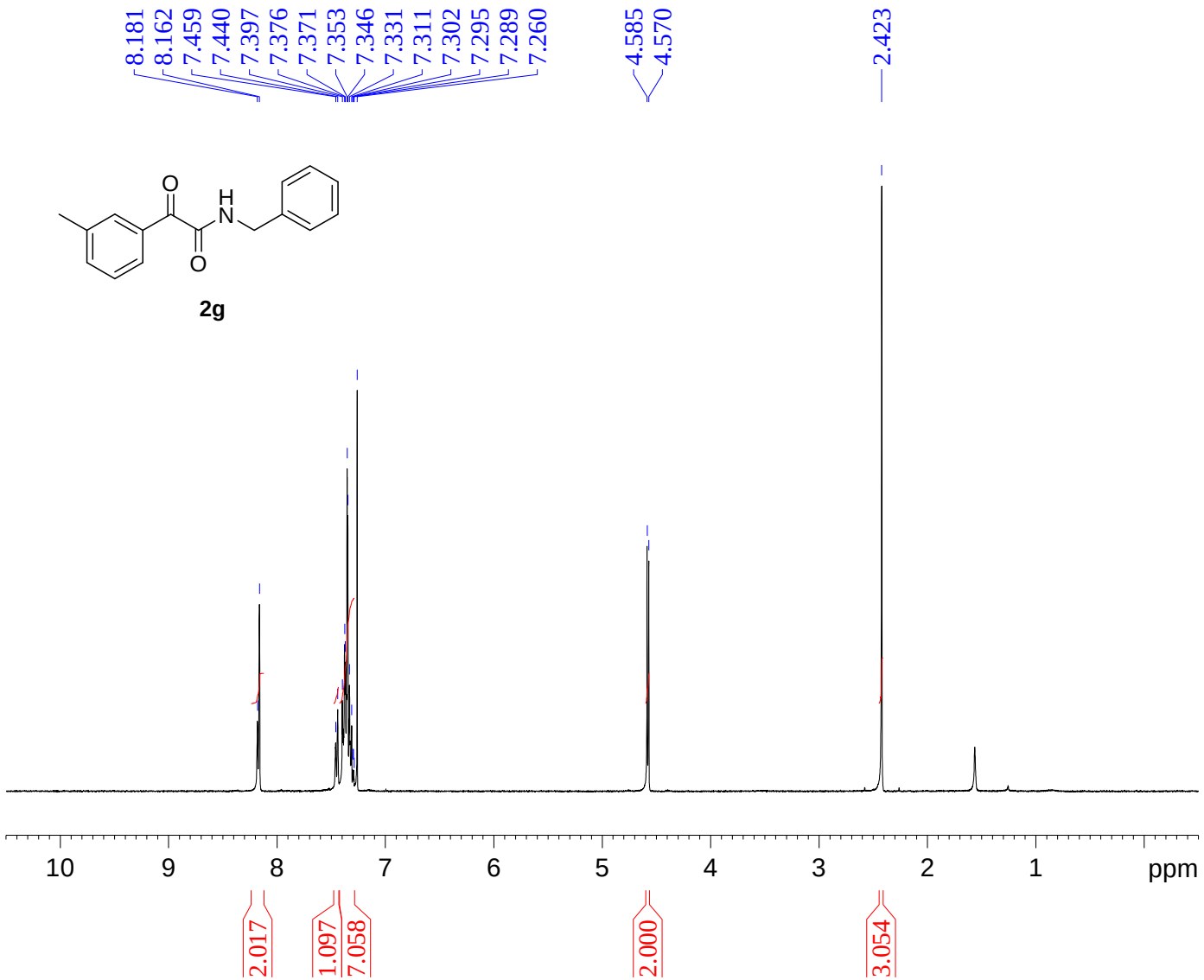


NAME Substrate 1H
EXPNO 5
PROCNO 1
Date_ 20150707
Time 11.12
INSTRUM spect
PROBHD 5 mm DUL 13C-1
PULPROG zg30
TD 32768
SOLVENT DMSO
NS 64
DS 0
SWH 6410.256 Hz
FIDRES 0.195625 Hz
AQ 2.5559540 sec
RG 4
DW 78.000 usec
DE 6.00 usec
TE 300.0 K
D1 2.00000000 sec
TD0 1

======== CHANNEL f1 ========
NUC1 1H
P1 10.00 usec
PL1 -2.40 dB
SFO1 400.1528010 MHz
SI 16384
SF 400.1500033 MHz
WDW EM
SSB 0
LB 0.00 Hz
GB 0
PC 1.00

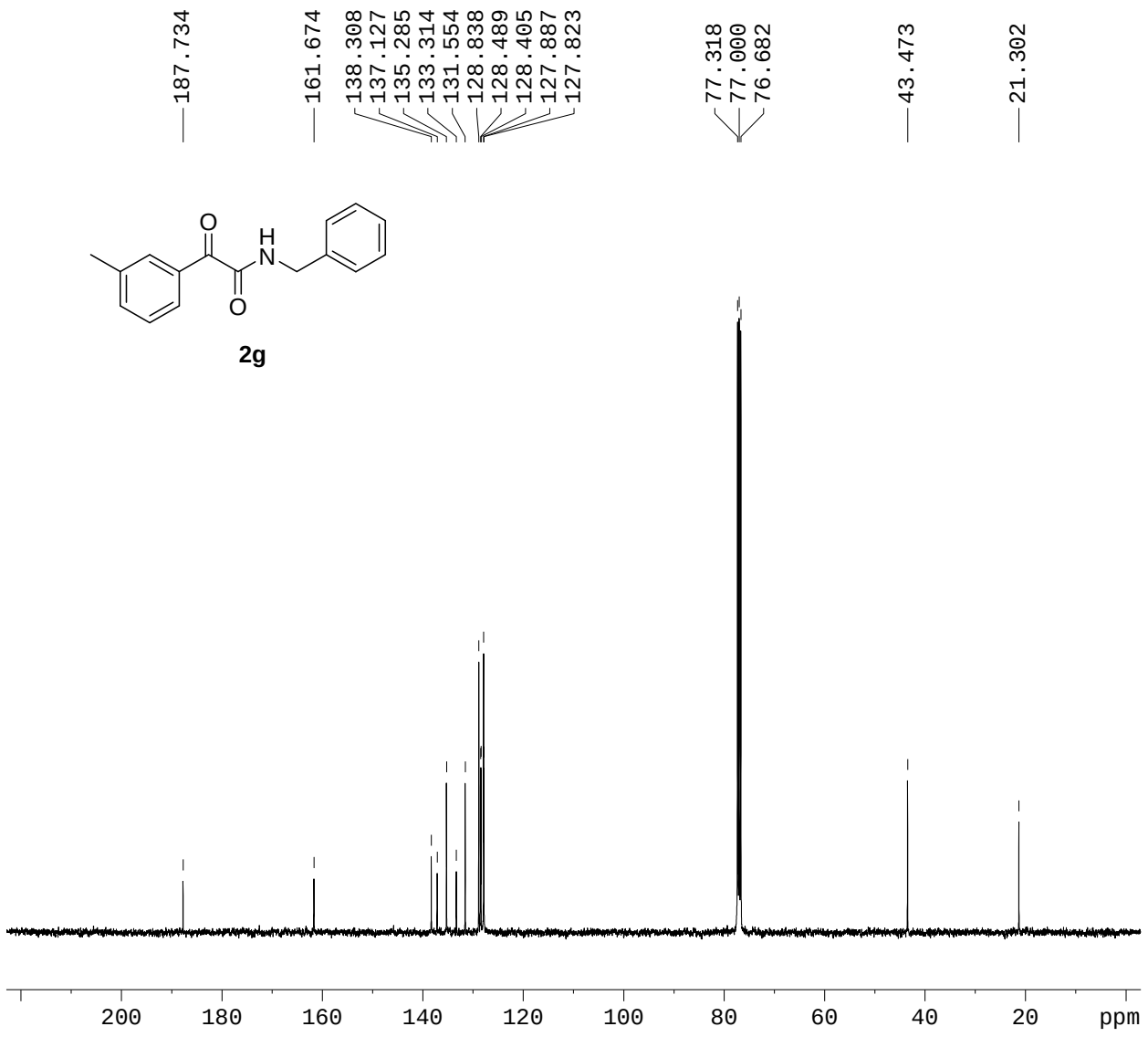


NAME	Substrate 13C	
EXPNO	5	
PROCNO	1	
Date_	20150711	
Time	16.48	
INSTRUM	spect	
PROBHD	5 mm DUL 13C-1	
PULPROG	zgpg30	
TD	65536	
SOLVENT	CDCl3	
NS	773	
DS	0	
SWH	22727.273	Hz
FIDRES	0.346791	Hz
AQ	1.4418420	sec
RG	57	
DW	22.000	usec
DE	6.00	usec
TE	300.0	K
D1	2.00000000	sec
d11	0.03000000	sec
DELTA	1.89999998	sec
TD0	1	
======== CHANNEL f1 ========		
NUC1	13C	
P1	9.70	usec
PL1	-0.50	dB
SFO1	100.6288660	MHz
======== CHANNEL f2 ========		
CPDPRG2	waltz16	
NUC2	1H	
PCPD2	90.00	usec
PL2	-2.40	dB
PL12	15.10	dB
PL13	18.10	dB
SFO2	400.1516010	MHz
SI	32768	
SF	100.6178424	MHz
WDW	EM	
SSB	0	
LB	3.00	Hz
GB	0	
PC	1.00	

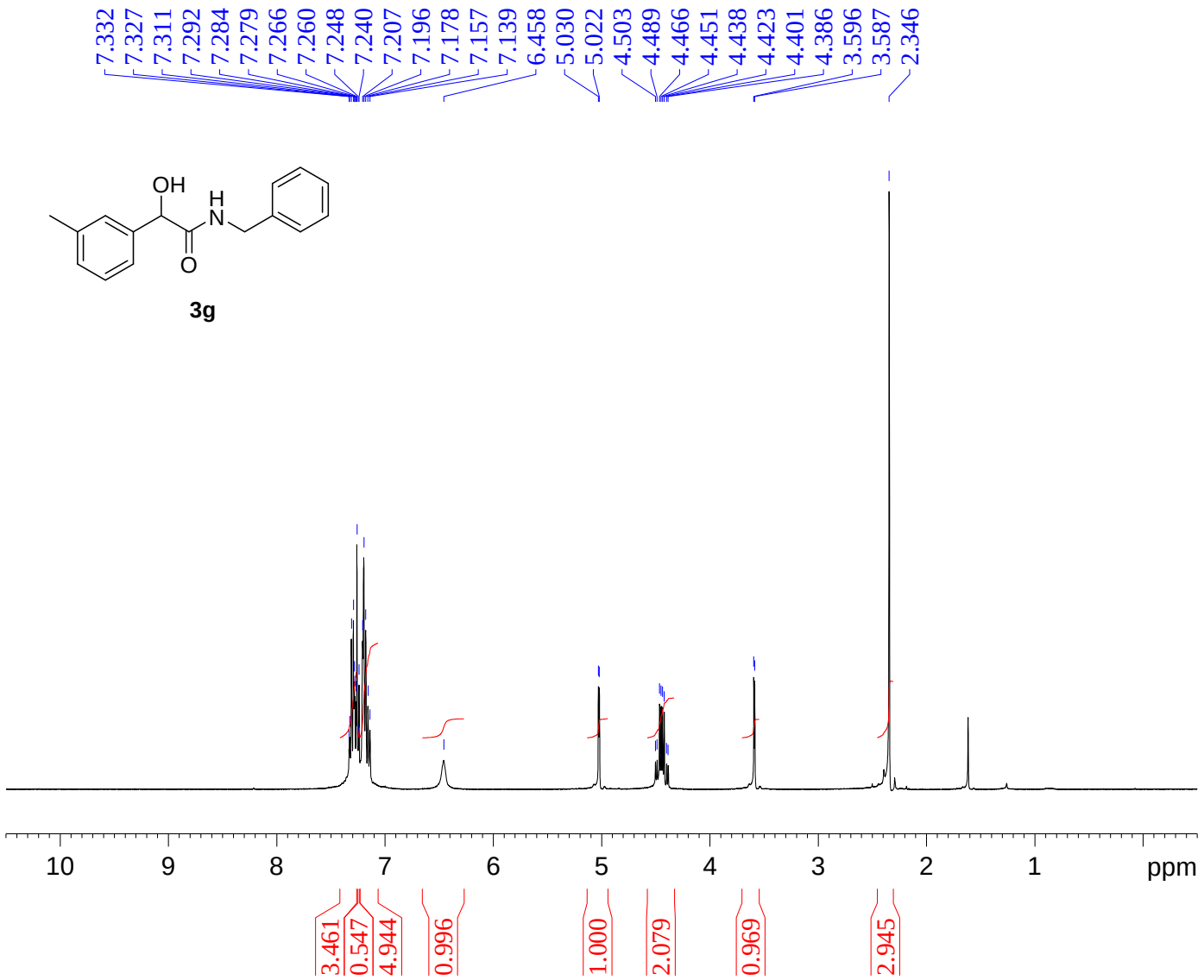


NAME 20151103
EXPNO 2
PROCNO 1
Date_ 20151103
Time 20.08
INSTRUM spect
PROBHD 5 mm DUL 13C-1
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 24
DS 0
SWH 6410.256 Hz
FIDRES 0.195625 Hz
AQ 2.5559540 sec
RG 4
DW 78.000 usec
DE 6.00 usec
TE 300.0 K
D1 2.00000000 sec
TD0 1

======== CHANNEL f1 ========
NUC1 1H
P1 10.00 usec
PL1 -2.40 dB
SFO1 400.1528010 MHz
SI 16384
SF 400.1500089 MHz
WDW EM
SSB 0
LB 0.00 Hz
GB 0
PC 1.00

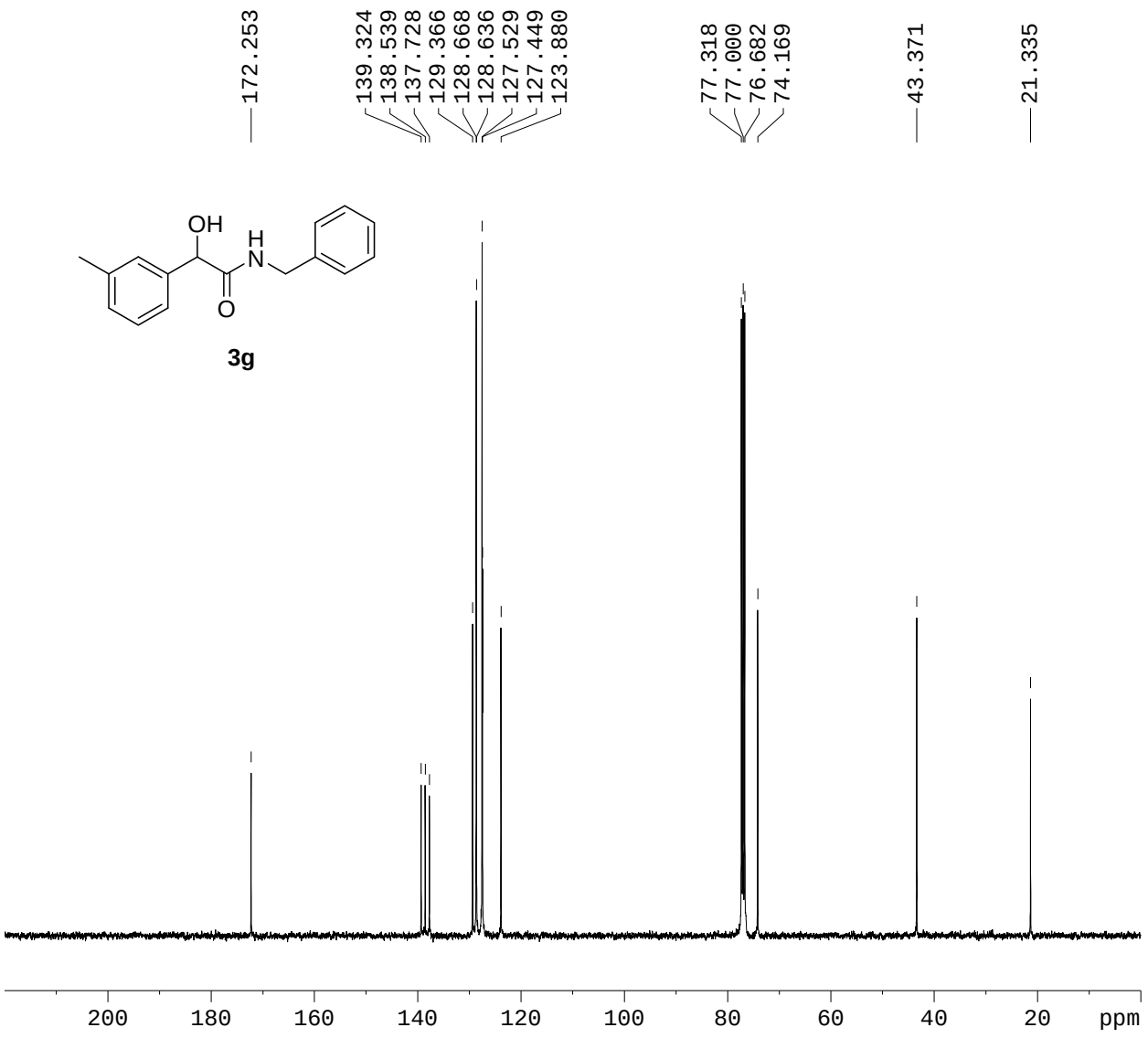



```
NAME            20151103
EXPNO                  3
PROCNO                 1
Date_           20151103
Time               20.14
INSTRUM             spect
PROBHD   5 mm DUL 13C-1
PULPROG           zgpg30
TD                 65536
SOLVENT             CDCl3
NS                    761
DS                      0
SWH           22727.273 Hz
FIDRES         0.346791 Hz
AQ             1.4418420 sec
RG                     57
DW                22.000 usec
DE                 6.00 usec
TE                300.0 K
D1           2.00000000 sec
d11          0.03000000 sec
DELTA        1.89999998 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                  13C
P1                  9.70 usec
PL1                -0.50 dB
SFO1        100.6288660 MHz

======== CHANNEL f2 ========
CPDPRG2           waltz16
NUC2                  1H
PCPD2              90.00 usec
PL2                -2.40 dB
PL12               15.10 dB
PL13               18.10 dB
SFO2        400.1516010 MHz
SI                 32768
SF          100.6178003 MHz
WDW                   EM
SSB                    0
LB                 3.00 Hz
GB                     0
PC                  1.00
```

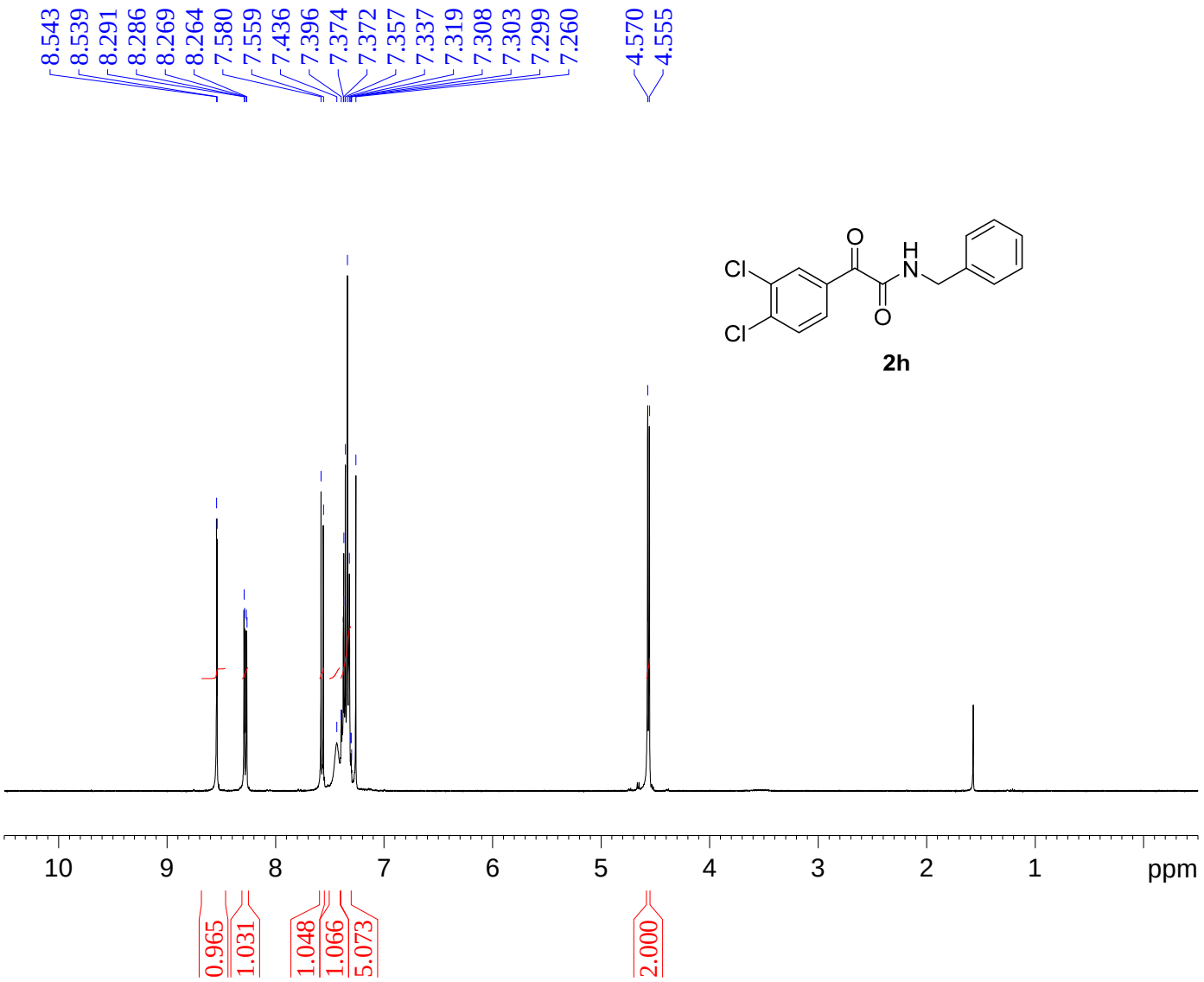


NAME 20151017
EXPNO 1
PROCNO 1
Date_ 20151017
Time 17.29
INSTRUM spect
PROBHD 5 mm DUL 13C-1
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 40
DS 0
SWH 6410.256 Hz
FIDRES 0.195625 Hz
AQ 2.5559540 sec
RG 4
DW 78.000 usec
DE 6.00 usec
TE 300.0 K
D1 2.00000000 sec
TD0 1

======== CHANNEL f1 ========
NUC1 1H
P1 10.00 usec
PL1 -2.40 dB
SFO1 400.1528010 MHz
SI 16384
SF 400.1500092 MHz
WDW EM
SSB 0
LB 0.00 Hz
GB 0
PC 1.00

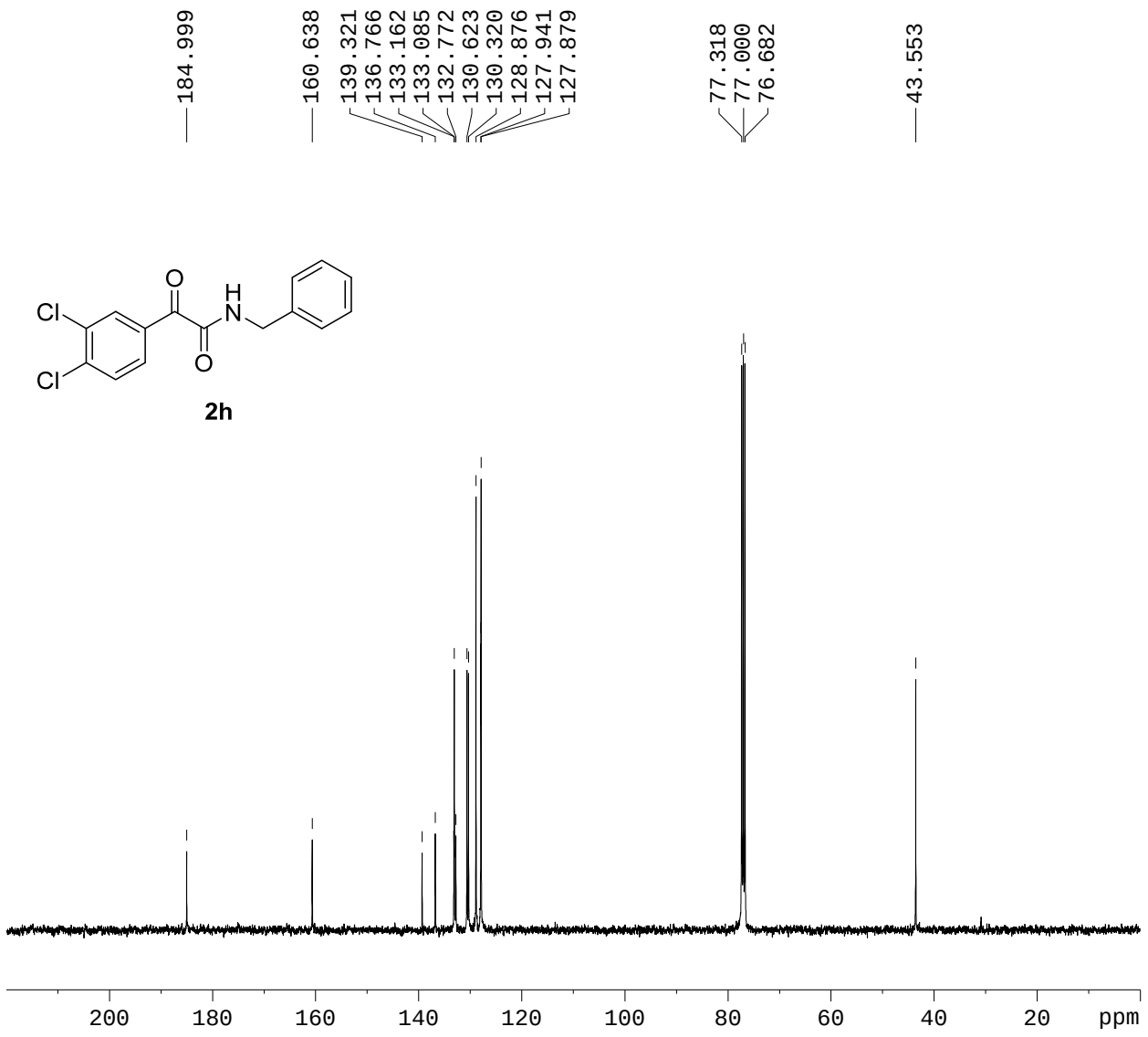



```
NAME       Substrate 13C
EXPNO                 23
PROCNO                 1
Date_           20151018
Time               16.11
INSTRUM            spect
PROBHD   5 mm DUL 13C-1
PULPROG          zgpg30
TD                65536
SOLVENT           CDCl3
NS                 1116
DS                    0
SWH           22727.273 Hz
FIDRES         0.346791 Hz
AQ            1.4418420 sec
RG                   57
DW                22.000 usec
DE                 6.00 usec
TE                300.0 K
D1           2.00000000 sec
d11          0.03000000 sec
DELTA        1.89999998 sec
TD0                   1

======== CHANNEL f1 ========
NUC1                 13C
P1                  9.70 usec
PL1                -0.50 dB
SFO1        100.6288660 MHz

======== CHANNEL f2 ========
CPDPRG2          waltz16
NUC2                 1H
PCPD2              90.00 usec
PL2                -2.40 dB
PL12               15.10 dB
PL13               18.10 dB
SFO2        400.1516010 MHz
SI                32768
SF          100.6178044 MHz
WDW                  EM
SSB                   0
LB                 3.00 Hz
GB                    0
PC                 1.00
```

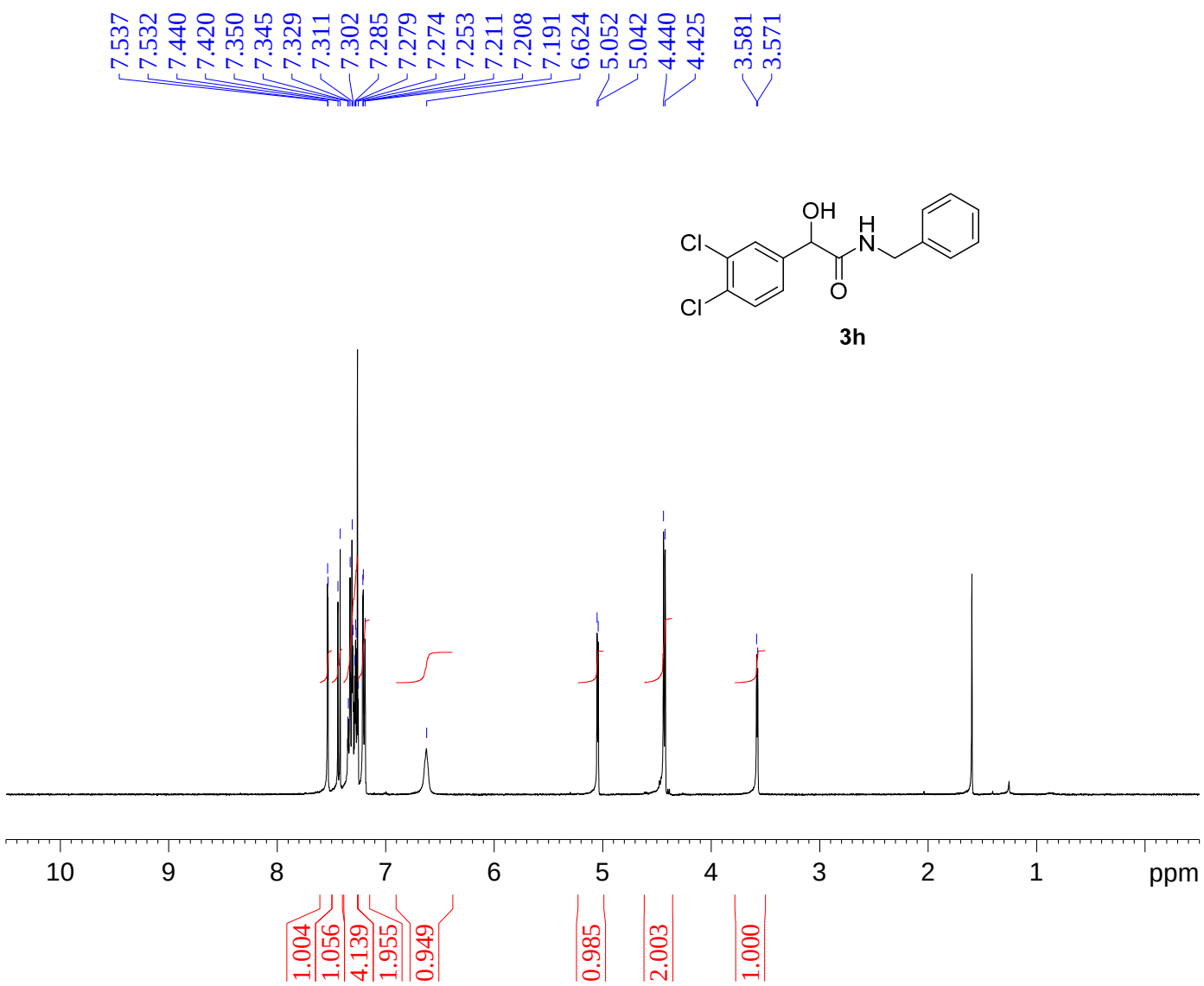


NAME 20151207
EXPNO 1
PROCNO 1
Date_ 20151207
Time 9.50
INSTRUM spect
PROBHD 5 mm DUL 13C-1
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 32
DS 0
SWH 6410.256 Hz
FIDRES 0.195625 Hz
AQ 2.5559540 sec
RG 4
DW 78.000 usec
DE 6.00 usec
TE 300.0 K
D1 2.00000000 sec
TD0 1

======== CHANNEL f1 ========
NUC1 1H
P1 10.00 usec
PL1 -2.40 dB
SFO1 400.1528010 MHz
SI 16384
SF 400.1500089 MHz
WDW EM
SSB 0
LB 0.00 Hz
GB 0
PC 1.00

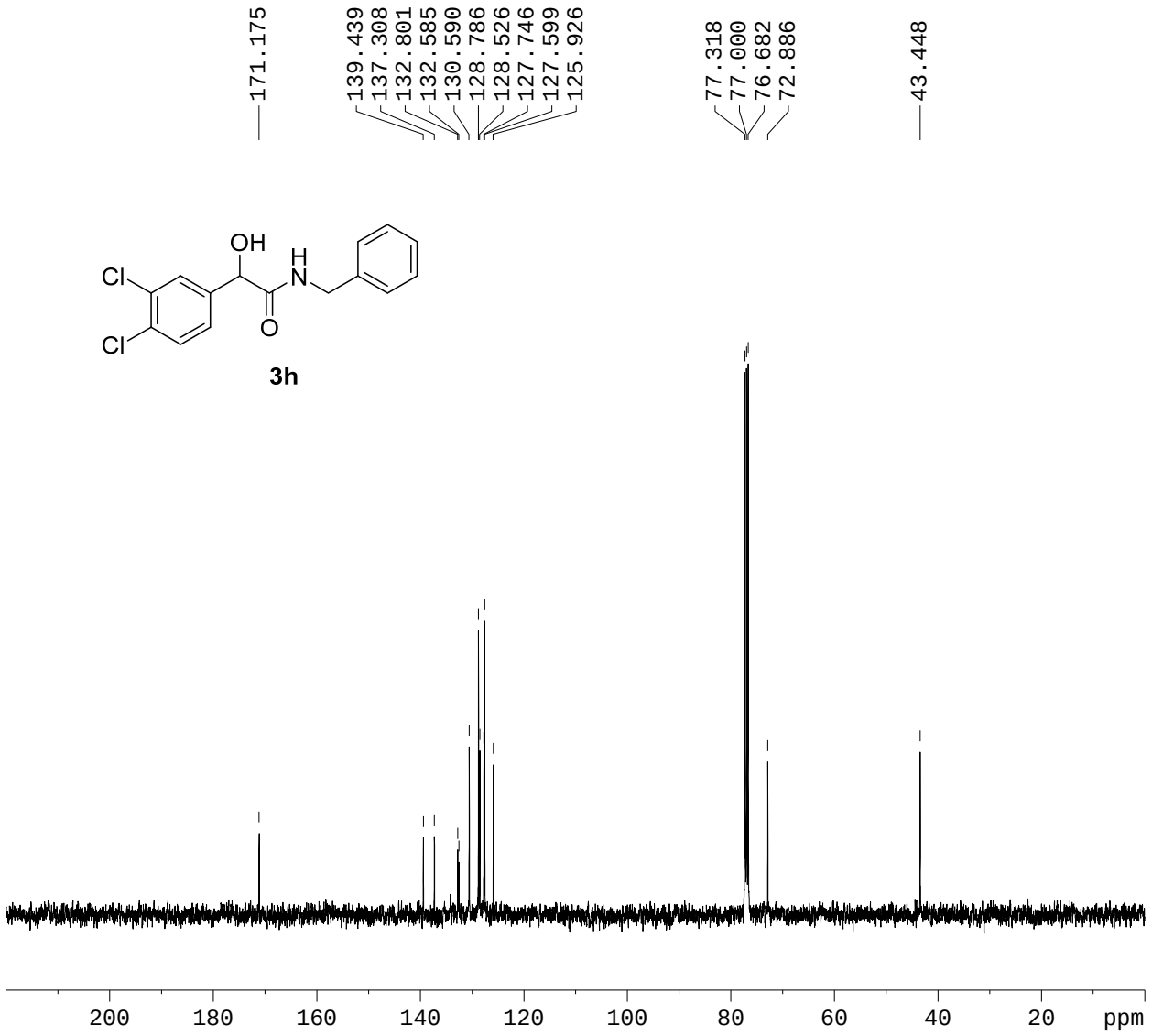


NAME	20151208
EXPNO	2
PROCNO	1
Date_	20151208
Time	21.22
INSTRUM	spect
PROBHD	5 mm DUL 13C-1
PULPROG	zgpg30
TD	65536
SOLVENT	CDCl3
NS	666
DS	0
SWH	22727.273 Hz
FIDRES	0.346791 Hz
AQ	1.4418420 sec
RG	57
DW	22.000 usec
DE	6.00 usec
TE	300.0 K
D1	2.00000000 sec
d11	0.03000000 sec
DELTA	1.89999998 sec
TD0	1
======== CHANNEL f1 ========	
NUC1	13C
P1	9.70 usec
PL1	-0.50 dB
SFO1	100.6288660 MHz
======== CHANNEL f2 ========	
CPDPRG2	waltz16
NUC2	1H
PCPD2	90.00 usec
PL2	-2.40 dB
PL12	15.10 dB
PL13	18.10 dB
SFO2	400.1516010 MHz
SI	32768
SF	100.6178028 MHz
WDW	EM
SSB	0
LB	3.00 Hz
GB	0
PC	1.00

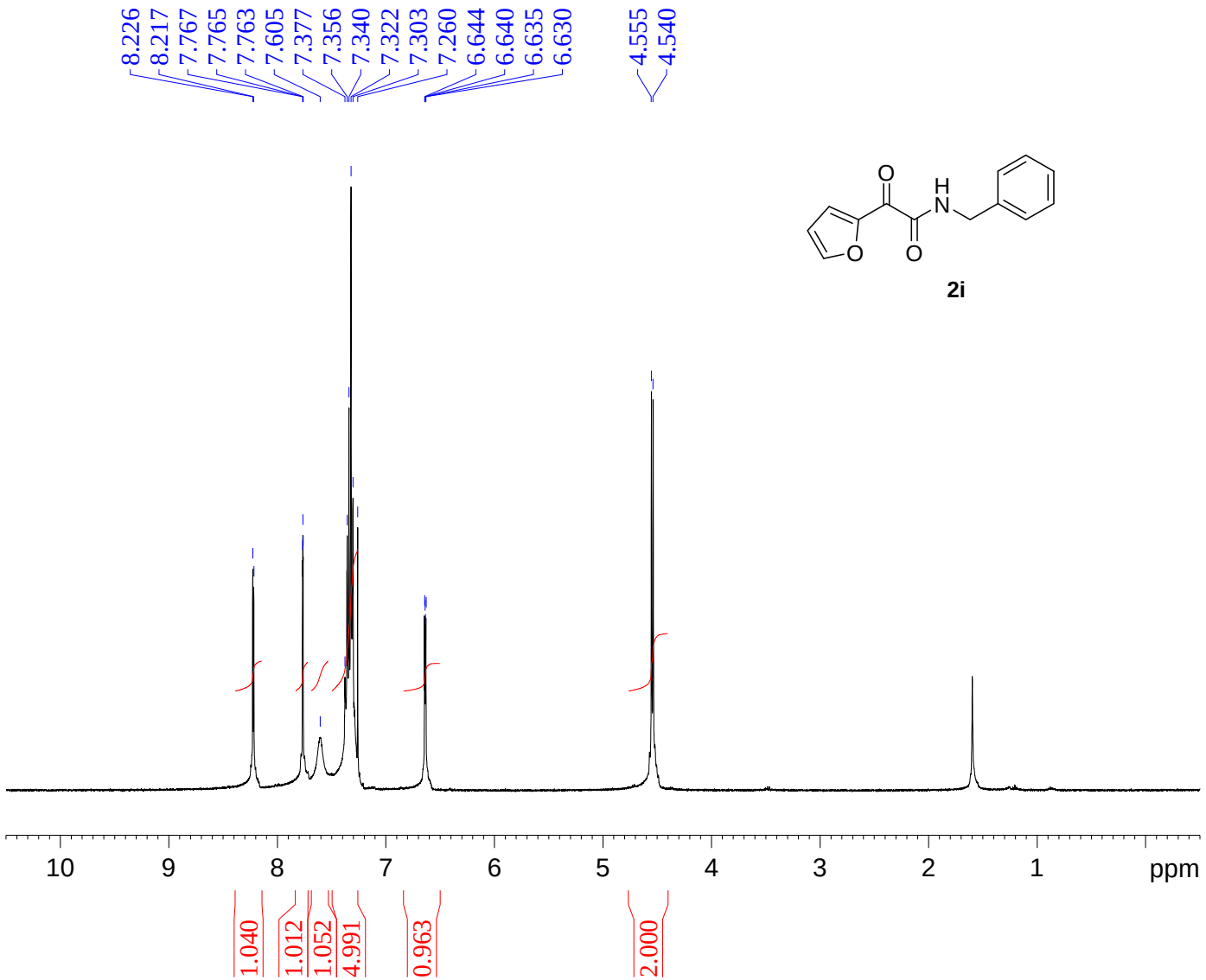


NAME 20151209
EXPNO 1
PROCNO 1
Date_ 20151209
Time 20.04
INSTRUM spect
PROBHD 5 mm DUL 13C-1
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 64
DS 0
SWH 6410.256 Hz
FIDRES 0.195625 Hz
AQ 2.5559540 sec
RG 4
DW 78.000 usec
DE 6.00 usec
TE 300.0 K
D1 2.00000000 sec
TD0 1

======== CHANNEL f1 ========
NUC1 1H
P1 10.00 usec
PL1 -2.40 dB
SFO1 400.1528010 MHz
SI 16384
SF 400.1500088 MHz
WDW EM
SSB 0
LB 0.00 Hz
GB 0
PC 1.00



NAME	20151209	
EXPNO	2	
PROCNO	1	
Date_	20151209	
Time	20.13	
INSTRUM	spect	
PROBHD	5 mm DUL 13C-1	
PULPROG	zgpg30	
TD	65536	
SOLVENT	CDCl3	
NS	100	
DS	0	
SWH	22727.273	Hz
FIDRES	0.346791	Hz
AQ	1.4418420	sec
RG	57	
DW	22.000	usec
DE	6.00	usec
TE	300.0	K
D1	2.00000000	sec
d11	0.03000000	sec
DELTA	1.89999998	sec
TD0	1	
======== CHANNEL f1 ========		
NUC1	13C	
P1	9.70	usec
PL1	-0.50	dB
SFO1	100.6288660	MHz
======== CHANNEL f2 ========		
CPDPRG2	waltz16	
NUC2	1H	
PCPD2	90.00	usec
PL2	-2.40	dB
PL12	15.10	dB
PL13	18.10	dB
SFO2	400.1516010	MHz
SI	32768	
SF	100.6178021	MHz
WDW	EM	
SSB	0	
LB	3.00	Hz
GB	0	
PC	1.00	

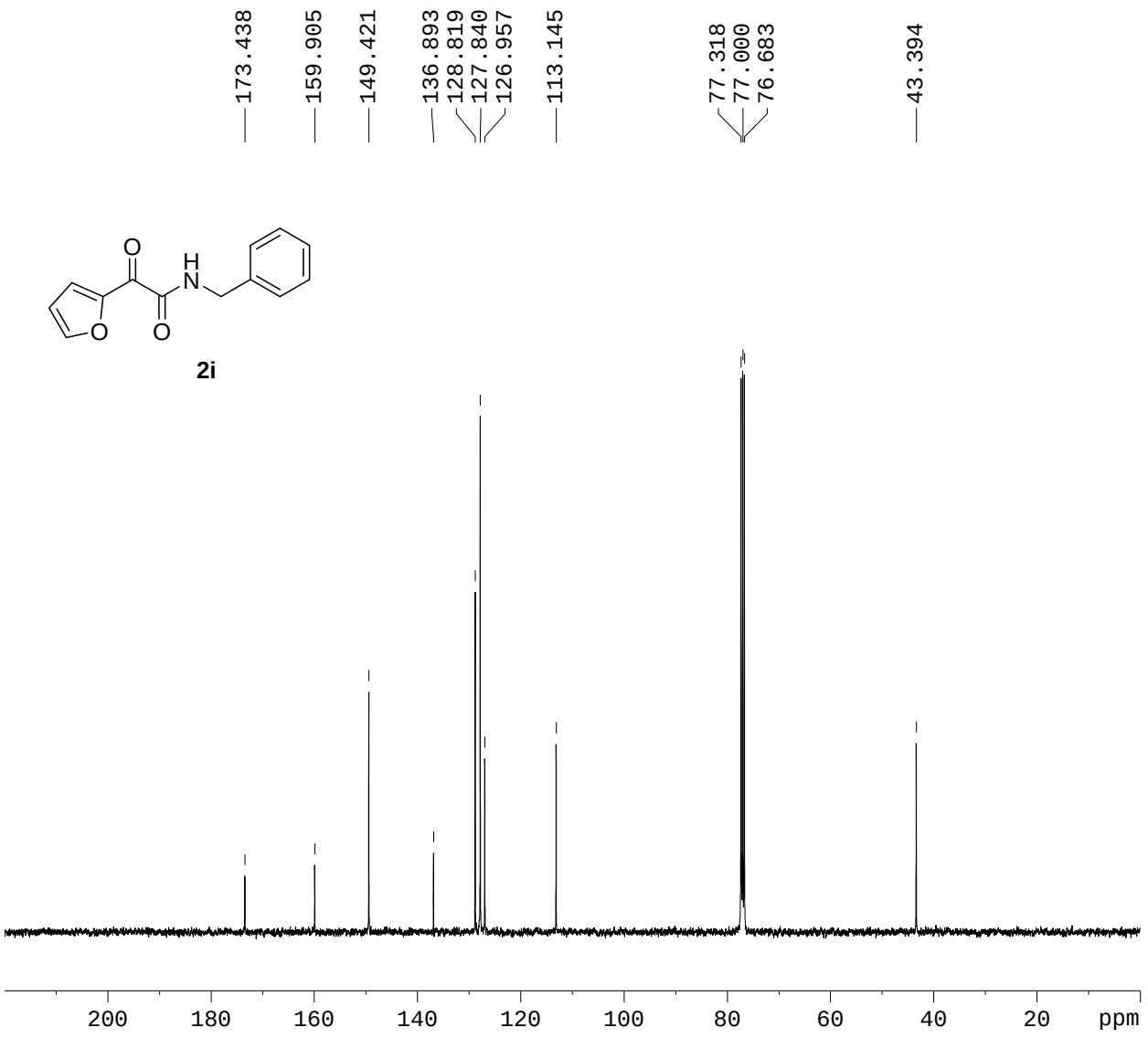


2i

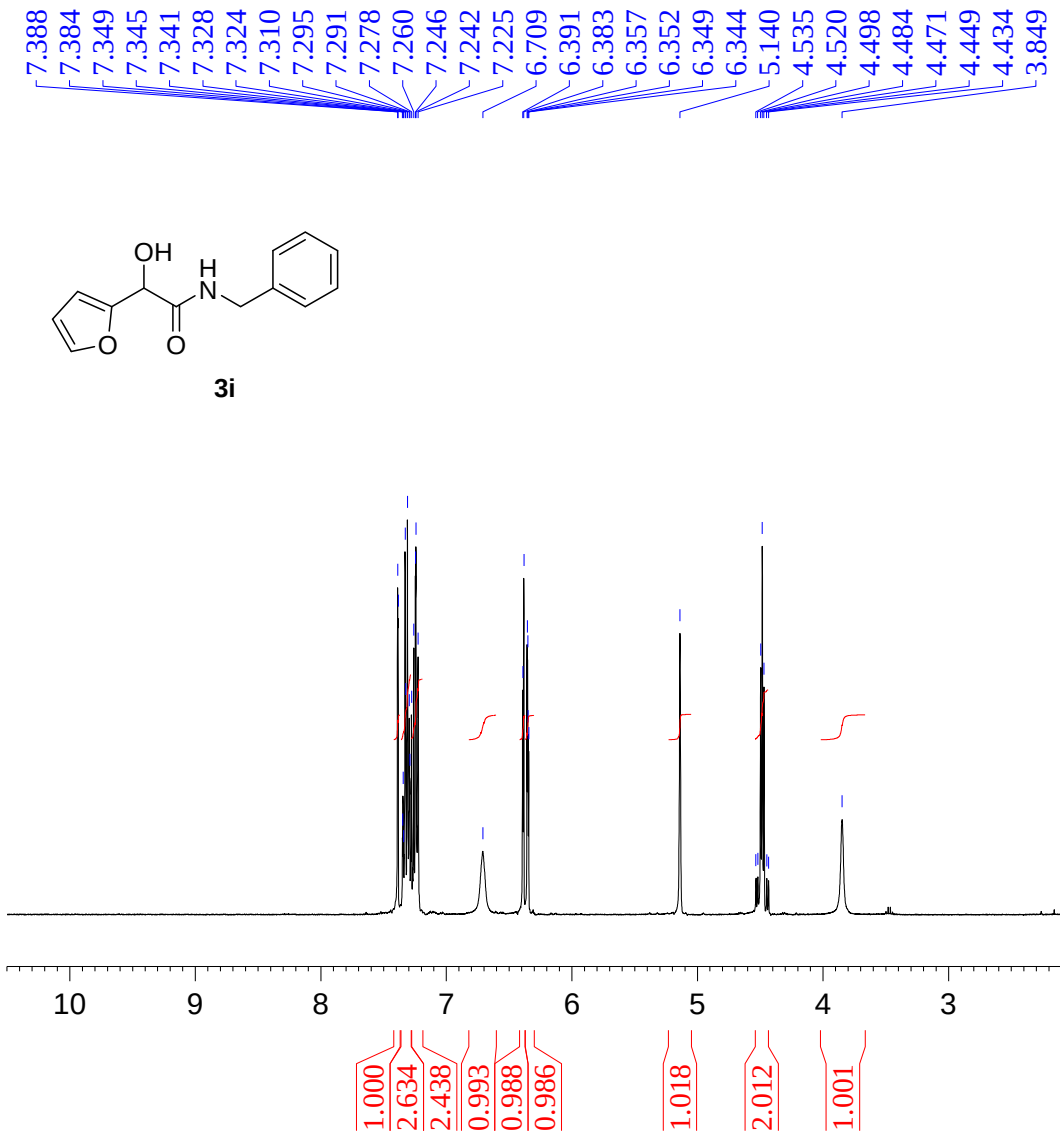
NAME	20151116
EXPNO	3
PROCNO	1
Date_	20151117
Time	20.12
INSTRUM	spect
PROBHD	5 mm DUL 13C-1
PULPROG	zg30
TD	32768
SOLVENT	CDCl3
NS	32
DS	0
SWH	6410.256 Hz
FIDRES	0.195625 Hz
AQ	2.5559540 sec
RG	4
DW	78.000 usec
DE	6.00 usec
TE	300.0 K
D1	2.00000000 sec
TD0	1

======== CHANNEL f1 ========

NUC1	1H
P1	10.00 usec
PL1	-2.40 dB
SFO1	400.1528010 MHz
SI	16384
SF	400.1500092 MHz
WDW	EM
SSB	0
LB	0.00 Hz
GB	0
PC	1.00

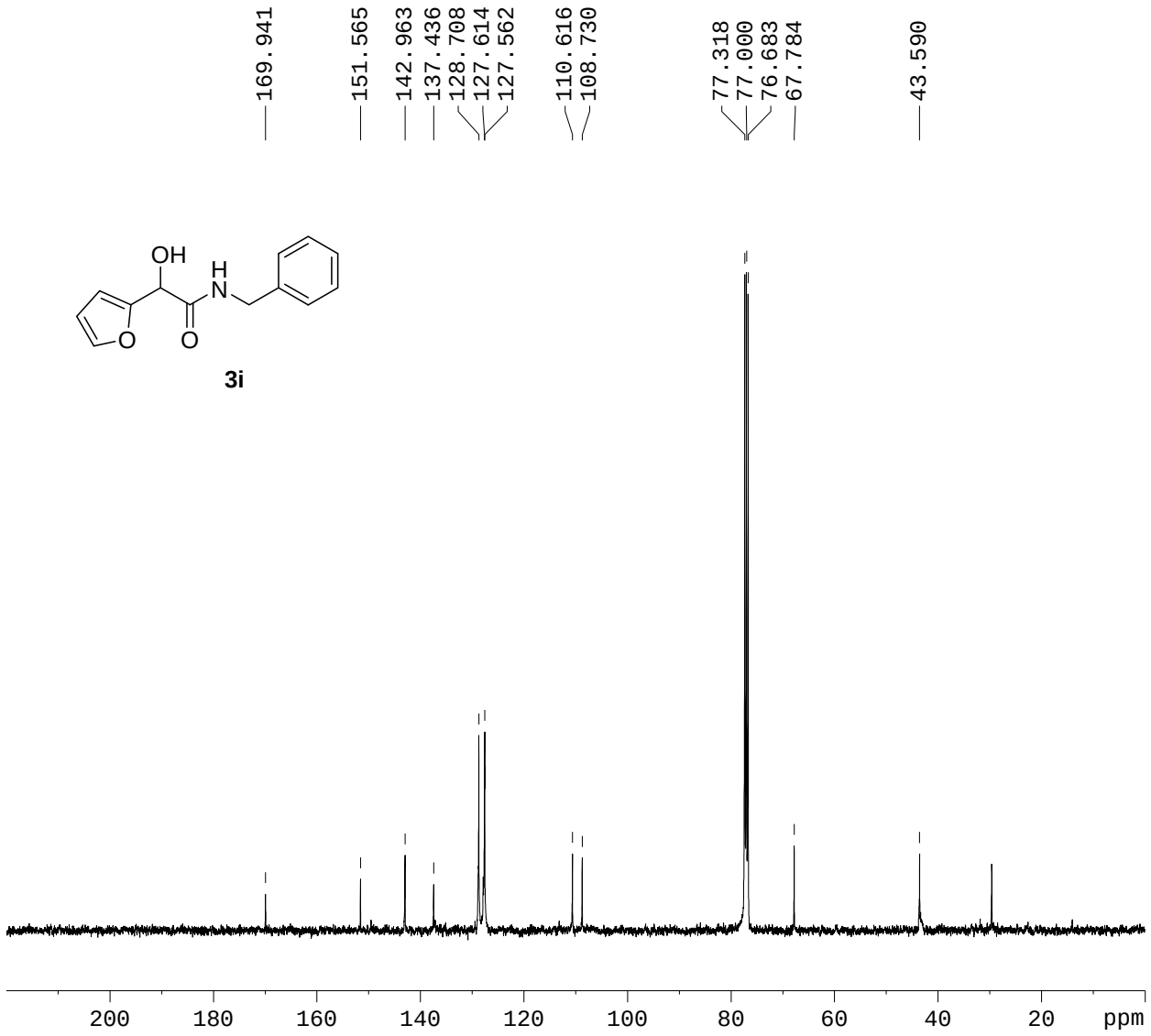


NAME	20151116
EXPNO	4
PROCNO	1
Date_	20151117
Time	20.18
INSTRUM	spect
PROBHD	5 mm DUL 13C-1
PULPROG	zgpg30
TD	65536
SOLVENT	CDCl3
NS	656
DS	0
SWH	22727.273 Hz
FIDRES	0.346791 Hz
AQ	1.4418420 sec
RG	57
DW	22.000 usec
DE	6.00 usec
TE	300.0 K
D1	2.00000000 sec
d11	0.03000000 sec
DELTA	1.89999998 sec
TD0	1
======== CHANNEL f1 ========	
NUC1	13C
P1	9.70 usec
PL1	-0.50 dB
SFO1	100.6288660 MHz
======== CHANNEL f2 ========	
CPDPRG2	waltz16
NUC2	1H
PCPD2	90.00 usec
PL2	-2.40 dB
PL12	15.10 dB
PL13	18.10 dB
SFO2	400.1516010 MHz
SI	32768
SF	100.6178023 MHz
WDW	EM
SSB	0
LB	3.00 Hz
GB	0
PC	1.00



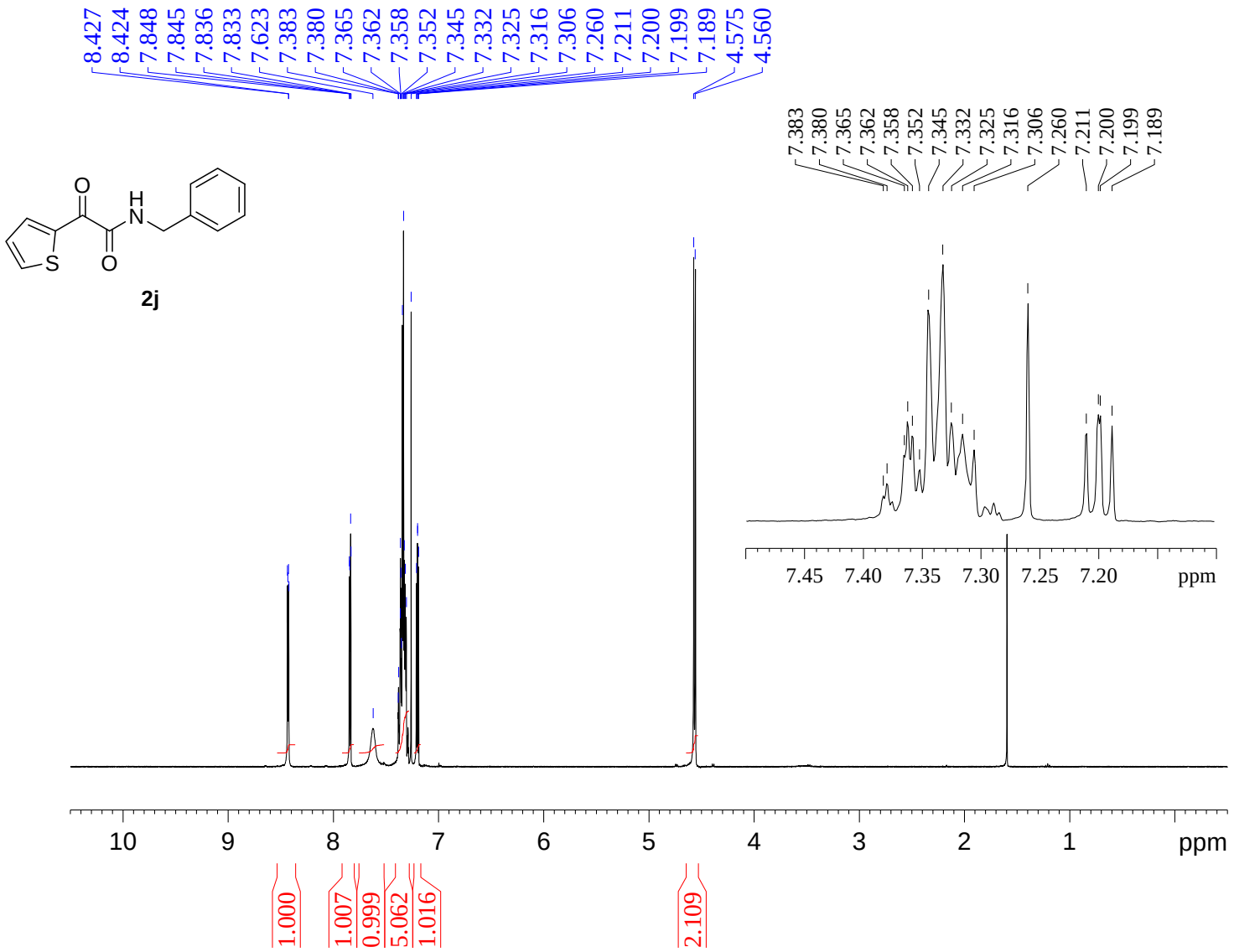
NAME 20160216
EXPNO 3
PROCNO 1
Date_ 20150608
Time 18.28
INSTRUM spect
PROBHD 5 mm DUL 13C-1
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 31
DS 0
SWH 6410.256 Hz
FIDRES 0.195625 Hz
AQ 2.5559540 sec
RG 4
DW 78.000 usec
DE 6.00 usec
TE 300.0 K
D1 2.00000000 sec
TD0 1

======== CHANNEL f1 ========
NUC1 1H
P1 10.00 usec
PL1 -2.40 dB
SFO1 400.1528010 MHz
SI 16384
SF 400.1500088 MHz
WDW EM
SSB 0
LB 0.00 Hz
GB 0
PC 1.00



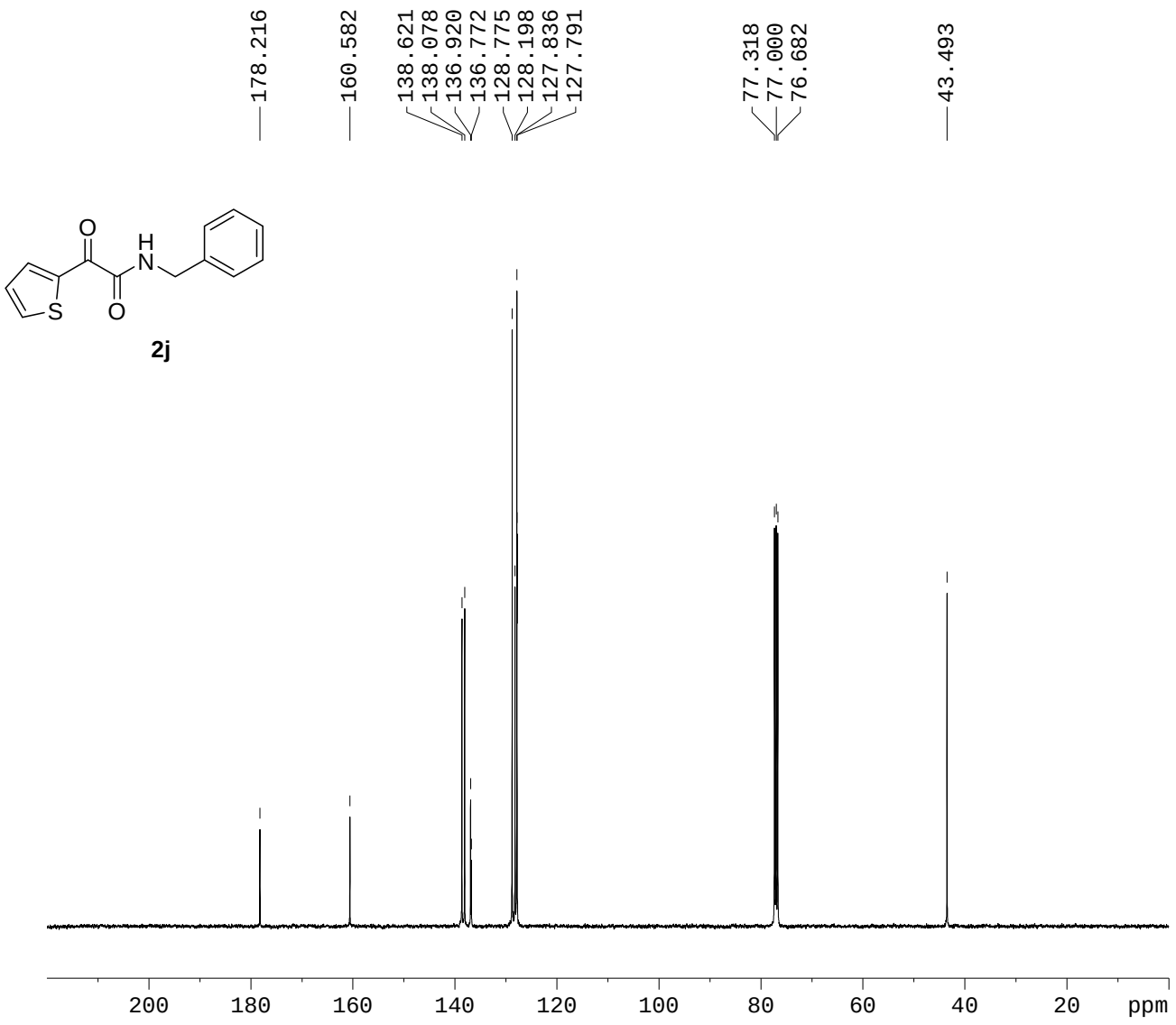
3i

```
NAME            20160216
EXPNO                  1
PROCNO                 1
Date_           20160216
Time               18.59
INSTRUM            spect
PROBHD   5 mm DUL 13C-1
PULPROG          zgpg30
TD                 65536
SOLVENT            CDCl3
NS                   960
DS                     0
SWH          22727.273 Hz
FIDRES        0.346791 Hz
AQ           1.4418420 sec
RG                    57
DW               22.000 usec
DE                6.00 usec
TE               300.0 K
D1           2.00000000 sec
d11          0.03000000 sec
DELTA        1.89999998 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                 13C
P1                 9.70 usec
PL1               -0.50 dB
SFO1        100.6288660 MHz

======== CHANNEL f2 ========
CPDPRG2          waltz16
NUC2                 1H
PCPD2             90.00 usec
PL2               -2.40 dB
PL12              15.10 dB
PL13              18.10 dB
SFO2        400.1516010 MHz
SI                32768
SF          100.6178033 MHz
WDW                  EM
SSB                   0
LB                 3.00 Hz
GB                    0
PC                 1.00
```



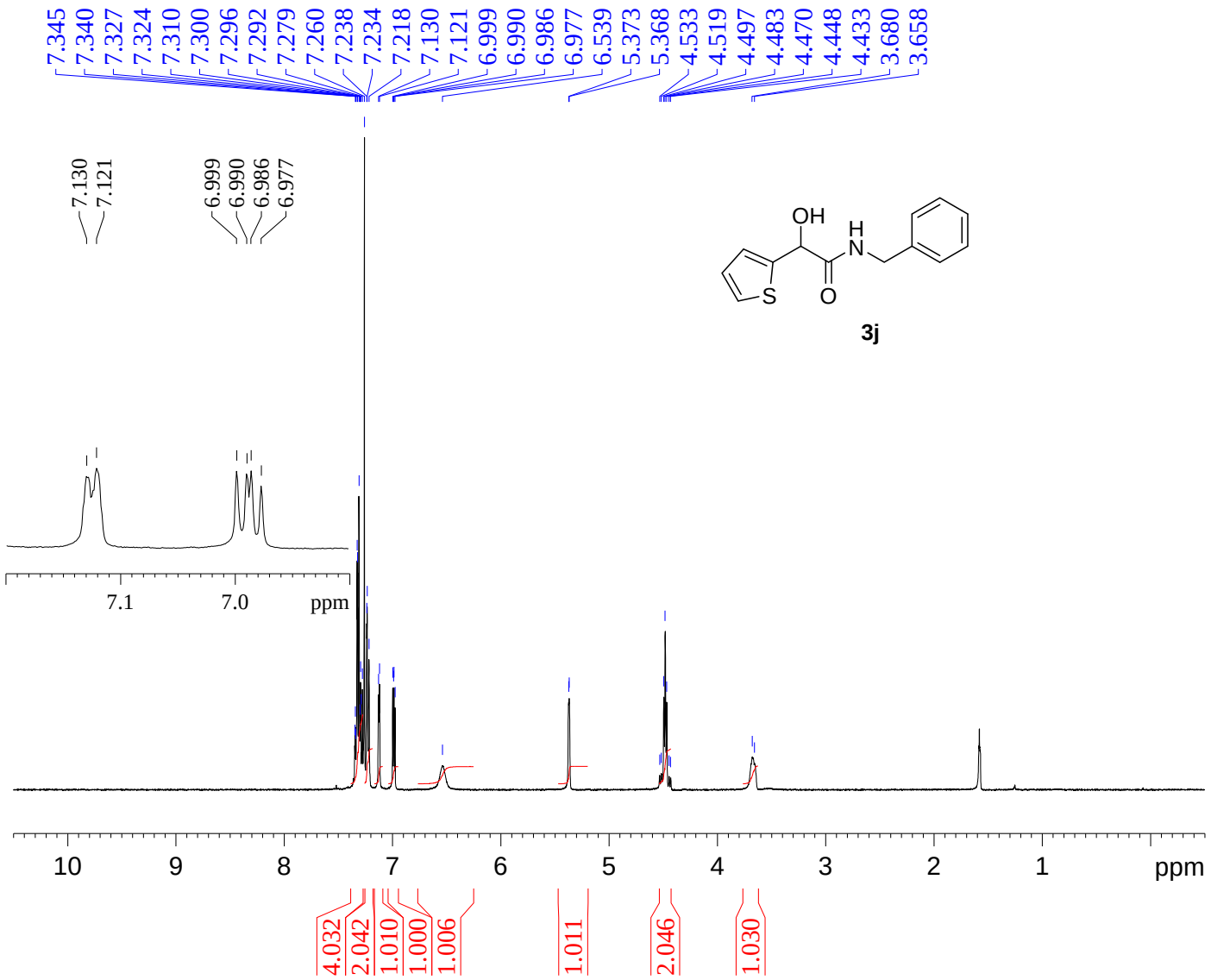
NAME Substrate 1H
EXPNO 2
PROCNO 1
Date_ 20150530
Time 18.10
INSTRUM spect
PROBHD 5 mm DUL 13C-1
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 32
DS 0
SWH 6410.256 Hz
FIDRES 0.195625 Hz
AQ 2.5559540 sec
RG 4
DW 78.000 usec
DE 6.00 usec
TE 300.0 K
D1 2.00000000 sec
TD0 1

======== CHANNEL f1 ========
NUC1 1H
P1 10.00 usec
PL1 -2.40 dB
SFO1 400.1528010 MHz
SI 16384
SF 400.1500088 MHz
WDW EM
SSB 0
LB 0.00 Hz
GB 0
PC 1.00



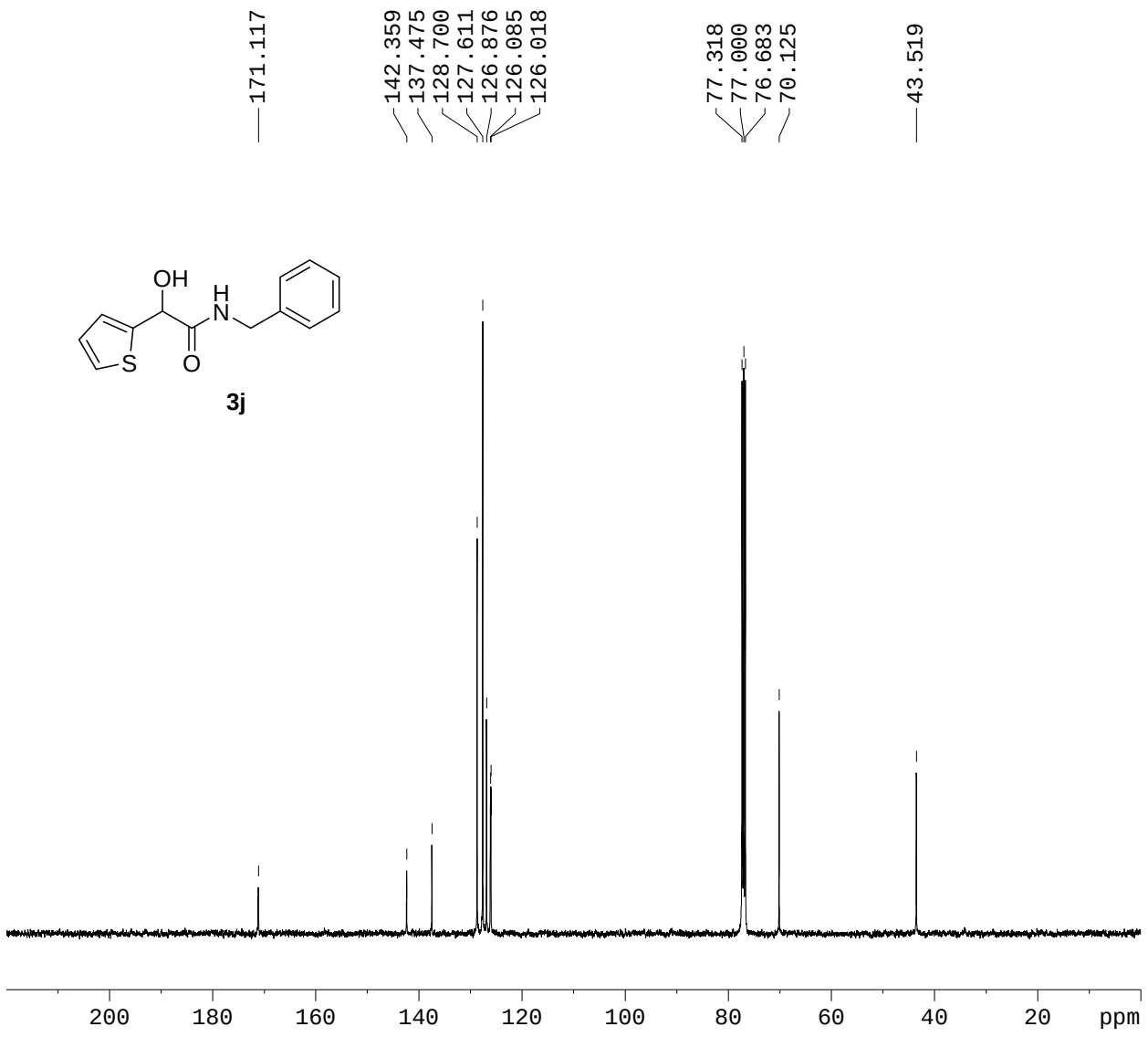
2j

NAME	Substrate 13C	
EXPNO	2	
PROCNO	1	
Date_	20150530	
Time	18.16	
INSTRUM	spect	
PROBHD	5 mm DUL 13C-1	
PULPROG	zgpg30	
TD	65536	
SOLVENT	CDCl3	
NS	1467	
DS	0	
SWH	22727.273	Hz
FIDRES	0.346791	Hz
AQ	1.4418420	sec
RG	57	
DW	22.000	usec
DE	6.00	usec
TE	300.0	K
D1	2.00000000	sec
d11	0.03000000	sec
DELTA	1.89999998	sec
TD0	1	
======== CHANNEL f1 ========		
NUC1	13C	
P1	9.70	usec
PL1	-0.50	dB
SFO1	100.6288660	MHz
======== CHANNEL f2 ========		
CPDPRG2	waltz16	
NUC2	1H	
PCPD2	90.00	usec
PL2	-2.40	dB
PL12	15.10	dB
PL13	18.10	dB
SFO2	400.1516010	MHz
SI	32768	
SF	100.6178066	MHz
WDW	EM	
SSB	0	
LB	3.00	Hz
GB	0	
PC	1.00	

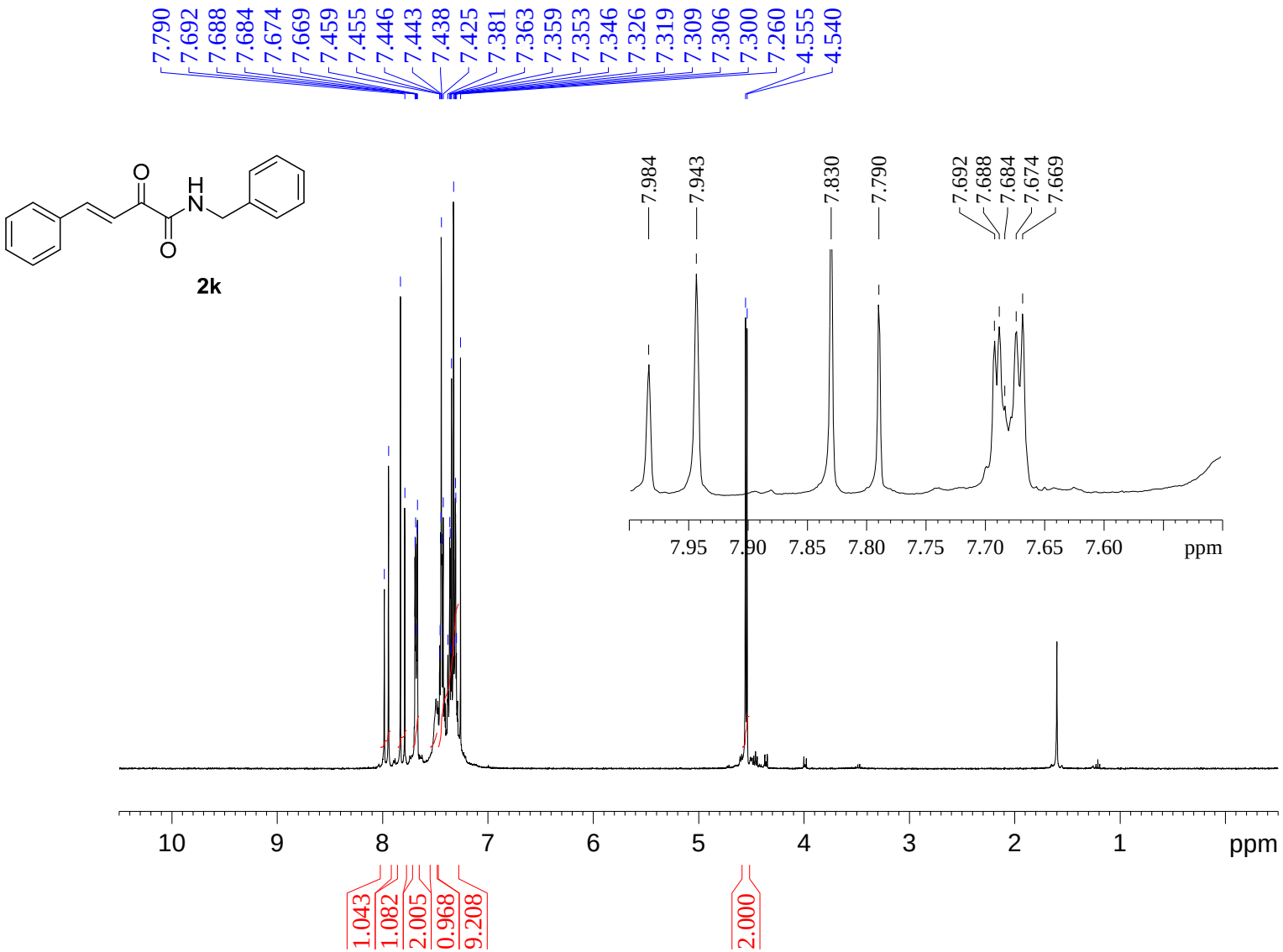


NAME Substrate 1H
EXPNO 3
PROCNO 1
Date_ 20150607
Time 16.32
INSTRUM spect
PROBHD 5 mm DUL 13C-1
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 48
DS 0
SWH 6410.256 Hz
FIDRES 0.195625 Hz
AQ 2.5559540 sec
RG 4
DW 78.000 usec
DE 6.00 usec
TE 300.0 K
D1 2.00000000 sec
TD0 1

======== CHANNEL f1 ========
NUC1 1H
P1 10.00 usec
PL1 -2.40 dB
SFO1 400.1528010 MHz
SI 16384
SF 400.1500088 MHz
WDW EM
SSB 0
LB 0.00 Hz
GB 0
PC 1.00

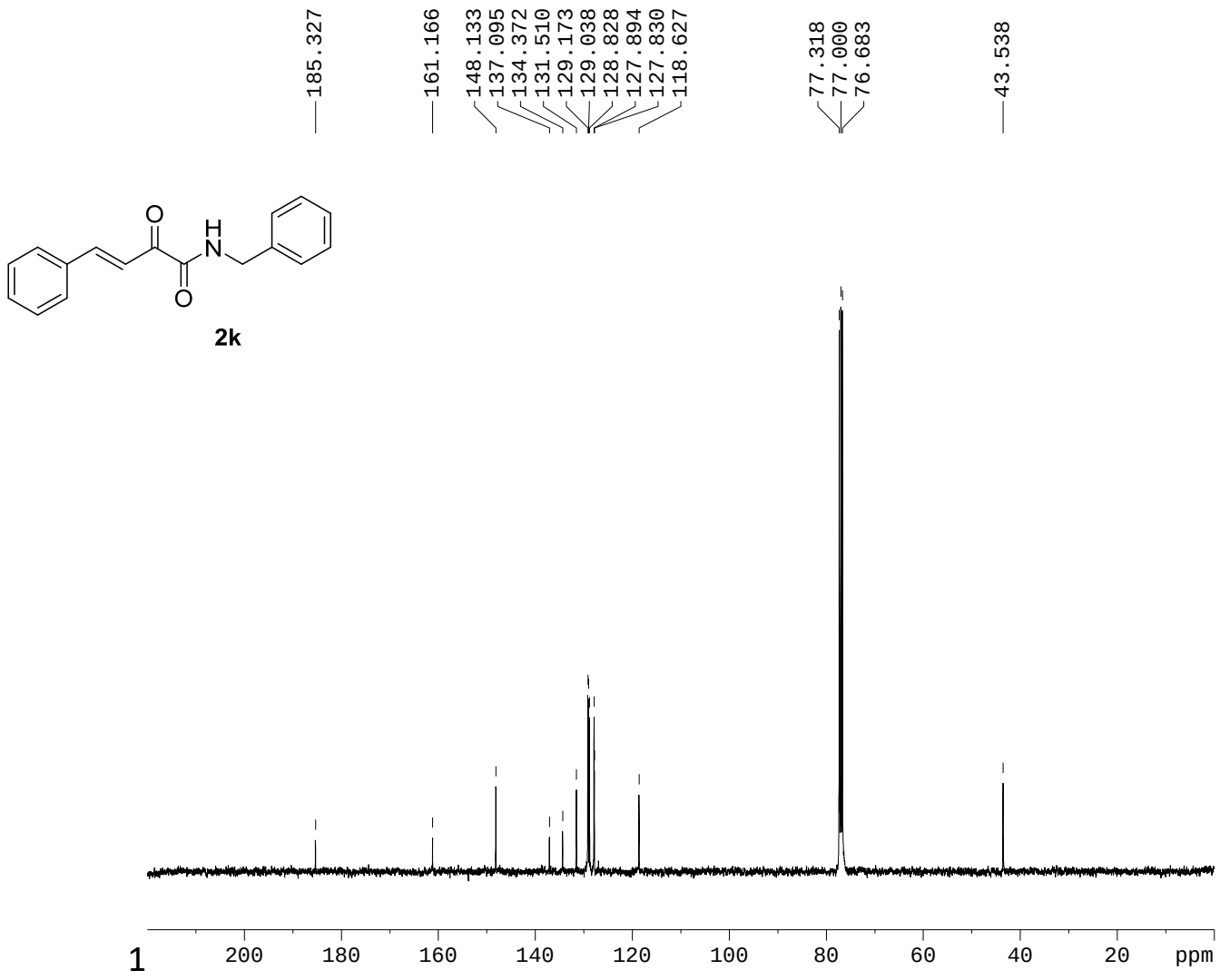



```
NAME       Substrate 13C
EXPNO                  3
PROCNO                 1
Date_           20150607
Time               16.40
INSTRUM             spect
PROBHD   5 mm DUL 13C-1
PULPROG          zgpg30
TD                 65536
SOLVENT            CDCl3
NS                  1284
DS                     0
SWH           22727.273 Hz
FIDRES         0.346791 Hz
AQ             1.4418420 sec
RG                    57
DW                22.000 usec
DE                 6.00 usec
TE                300.0 K
D1            2.00000000 sec
d11           0.03000000 sec
DELTA         1.89999998 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                 13C
P1                  9.70 usec
PL1                -0.50 dB
SFO1        100.6288660 MHz

======== CHANNEL f2 ========
CPDPRG2          waltz16
NUC2                 1H
PCPD2              90.00 usec
PL2                -2.40 dB
PL12               15.10 dB
PL13               18.10 dB
SFO2        400.1516010 MHz
SI                 32768
SF          100.6178029 MHz
WDW                  EM
SSB                   0
LB                 3.00 Hz
GB                    0
PC                 1.00
```

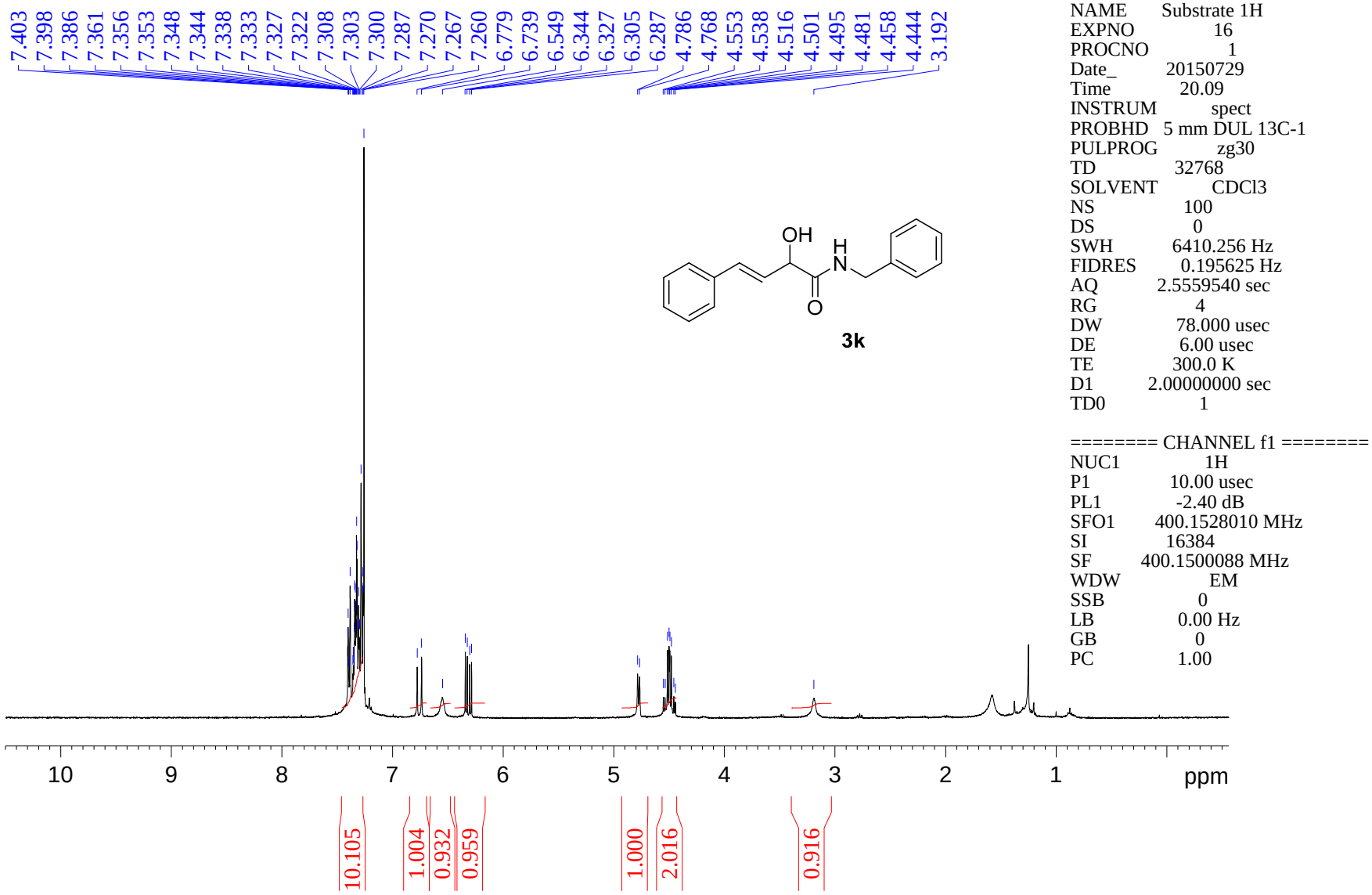


NAME Substrate 1H
EXPNO 15
PROCNO 1
Date_ 20150727
Time 20.20
INSTRUM spect
PROBHD 5 mm DUL 13C-1
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 49
DS 0
SWH 6410.256 Hz
FIDRES 0.195625 Hz
AQ 2.5559540 sec
RG 4
DW 78.000 usec
DE 6.00 usec
TE 300.0 K
D1 2.00000000 sec
TD0 1

======== CHANNEL f1 ========
NUC1 1H
P1 10.00 usec
PL1 -2.40 dB
SFO1 400.1528010 MHz
SI 16384
SF 400.1500088 MHz
WDW EM
SSB 0
LB 0.00 Hz
GB 0
PC 1.00

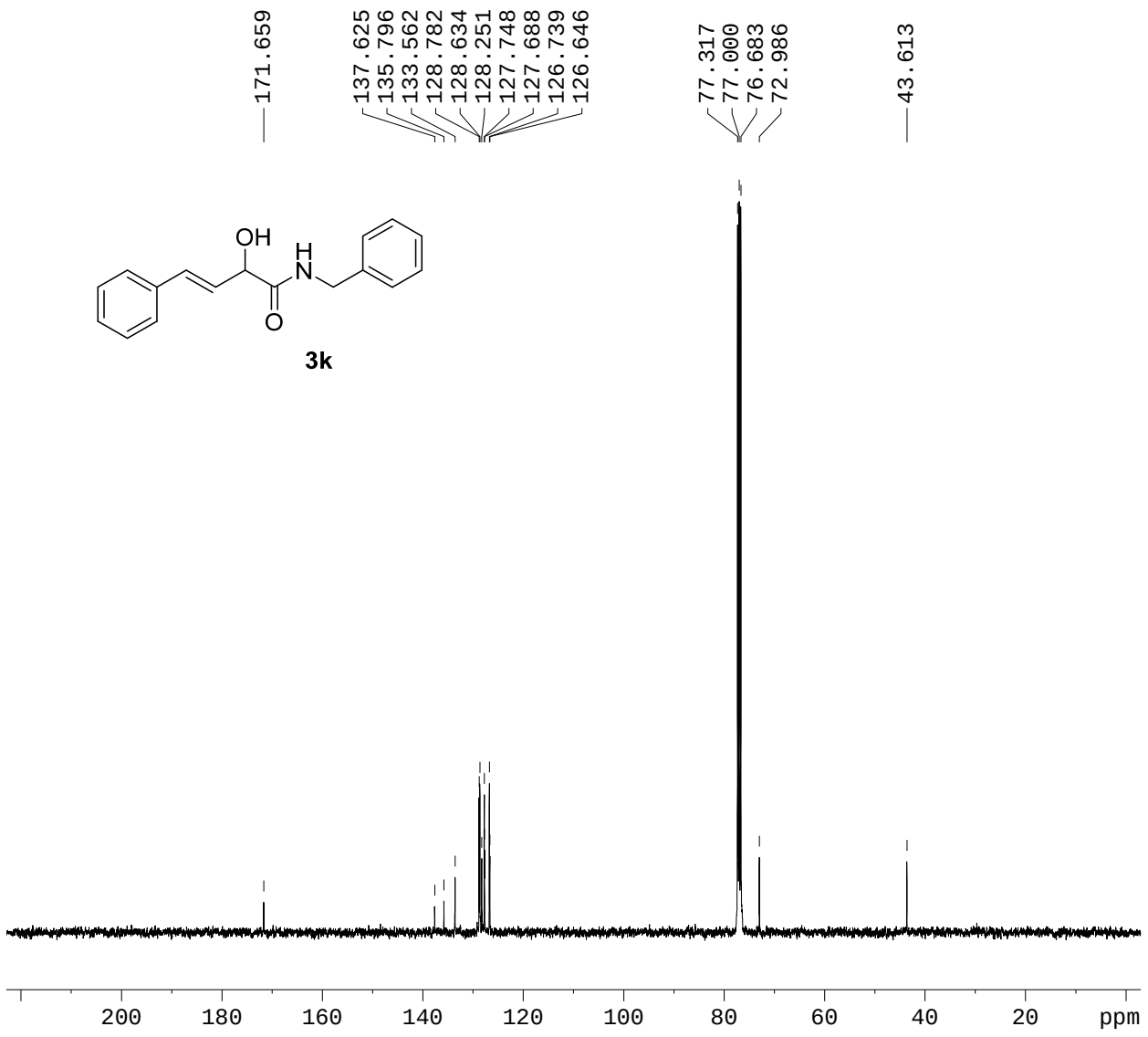



```
NAME       Substrate 13C
EXPNO                15
PROCNO                1
Date_          20150727
Time              20.26
INSTRUM            spect
PROBHD   5 mm DUL 13C-1
PULPROG          zgpg30
TD                65536
SOLVENT           CDCl3
NS                  661
DS                    0
SWH           22727.273 Hz
FIDRES         0.346791 Hz
AQ            1.4418420 sec
RG                   57
DW                22.000 usec
DE                 6.00 usec
TE                300.0 K
D1           2.00000000 sec
d11          0.03000000 sec
DELTA        1.89999998 sec
TD0                   1

======== CHANNEL f1 ========
NUC1                 13C
P1                  9.70 usec
PL1                -0.50 dB
SFO1        100.6288660 MHz

======== CHANNEL f2 ========
CPDPRG2          waltz16
NUC2                 1H
PCPD2              90.00 usec
PL2                -2.40 dB
PL12               15.10 dB
PL13               18.10 dB
SFO2        400.1516010 MHz
SI                 32768
SF          100.6178002 MHz
WDW                  EM
SSB                   0
LB                 3.00 Hz
GB                    0
PC                 1.00
```

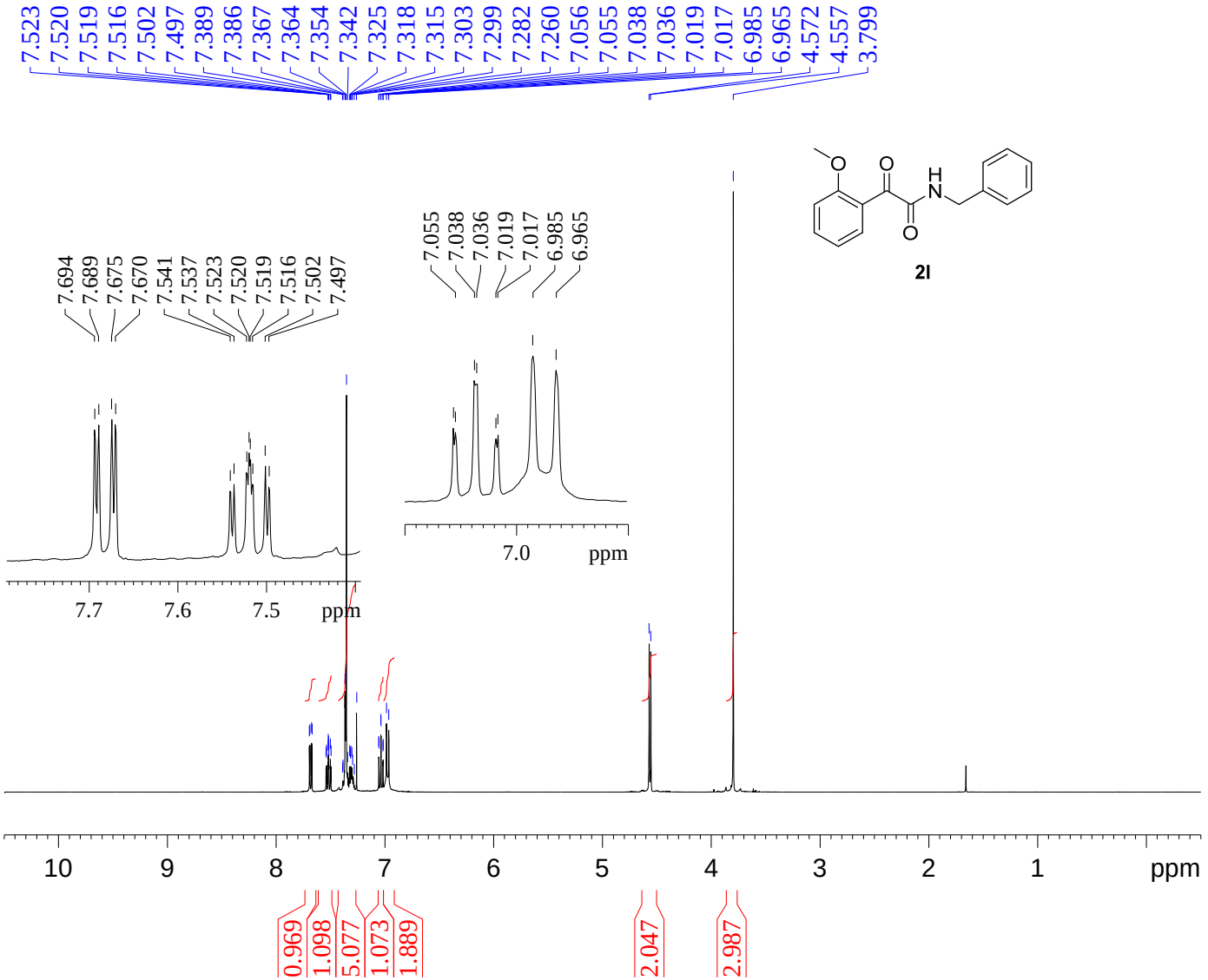


NAME Substrate 1H
EXPNO 16
PROCNO 1
Date_ 20150729
Time 20.09
INSTRUM spect
PROBHD 5 mm DUL 13C-1
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 100
DS 0
SWH 6410.256 Hz
FIDRES 0.195625 Hz
AQ 2.5559540 sec
RG 4
DW 78.000 usec
DE 6.00 usec
TE 300.0 K
D1 2.00000000 sec
TD0 1

======== CHANNEL f1 ========
NUC1 1H
P1 10.00 usec
PL1 -2.40 dB
SFO1 400.1528010 MHz
SI 16384
SF 400.1500088 MHz
WDW EM
SSB 0
LB 0.00 Hz
GB 0
PC 1.00

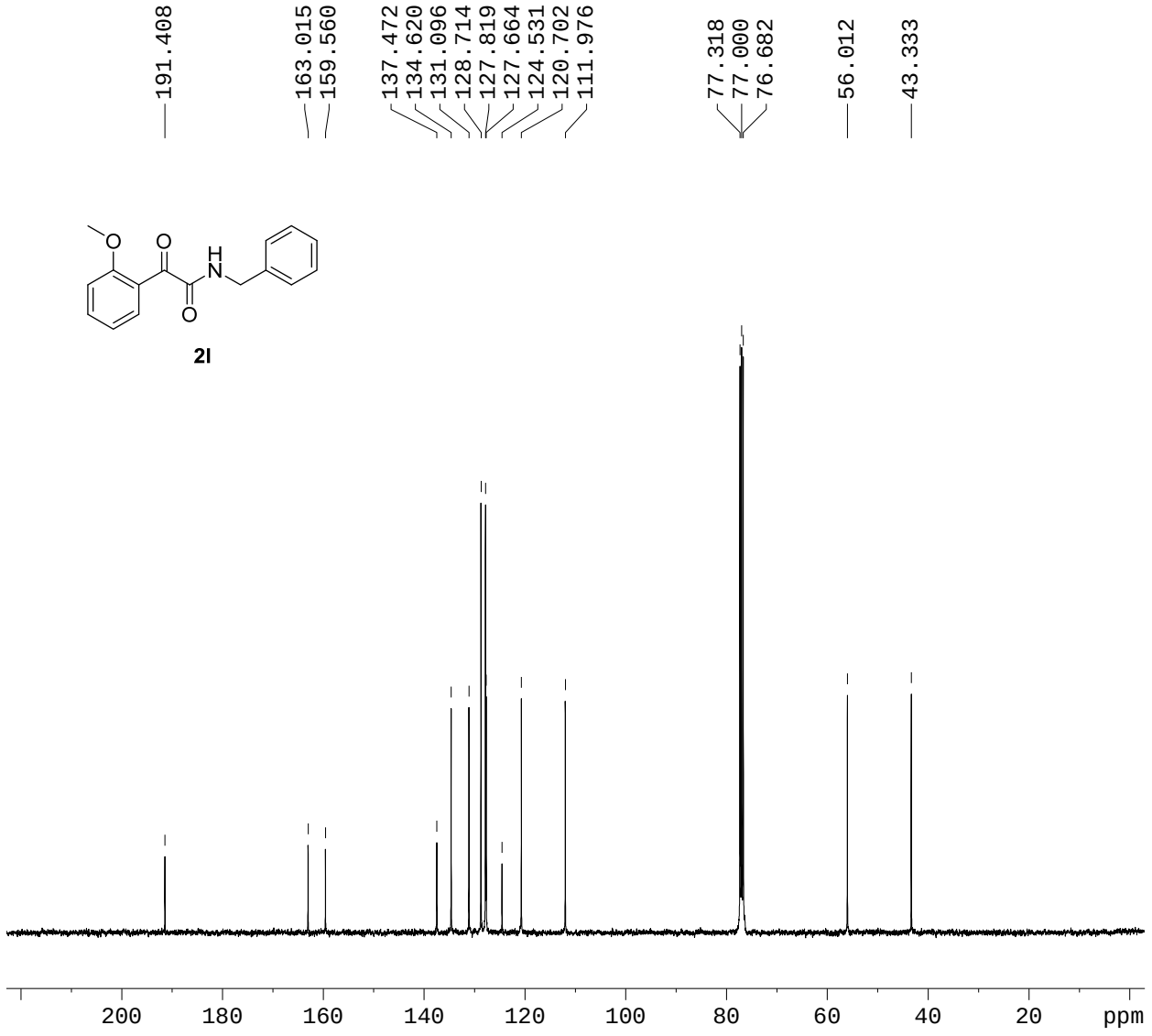



```
NAME       Substrate 13C
EXPNO                16
PROCNO                1
Date_          20150729
Time              20.20
INSTRUM           spect
PROBHD   5 mm DUL 13C-1
PULPROG          zgpg30
TD                65536
SOLVENT           CDCl3
NS                  723
DS                    0
SWH           22727.273 Hz
FIDRES         0.346791 Hz
AQ            1.4418420 sec
RG                   57
DW                22.000 usec
DE                 6.00 usec
TE                300.0 K
D1           2.00000000 sec
d11          0.03000000 sec
DELTA        1.89999998 sec
TD0                   1

======== CHANNEL f1 ========
NUC1                 13C
P1                  9.70 usec
PL1                -0.50 dB
SFO1        100.6288660 MHz

======== CHANNEL f2 ========
CPDPRG2          waltz16
NUC2                 1H
PCPD2              90.00 usec
PL2                -2.40 dB
PL12               15.10 dB
PL13               18.10 dB
SFO2        400.1516010 MHz
SI                32768
SF          100.6178001 MHz
WDW                  EM
SSB                   0
LB                 3.00 Hz
GB                    0
PC                 1.00
```

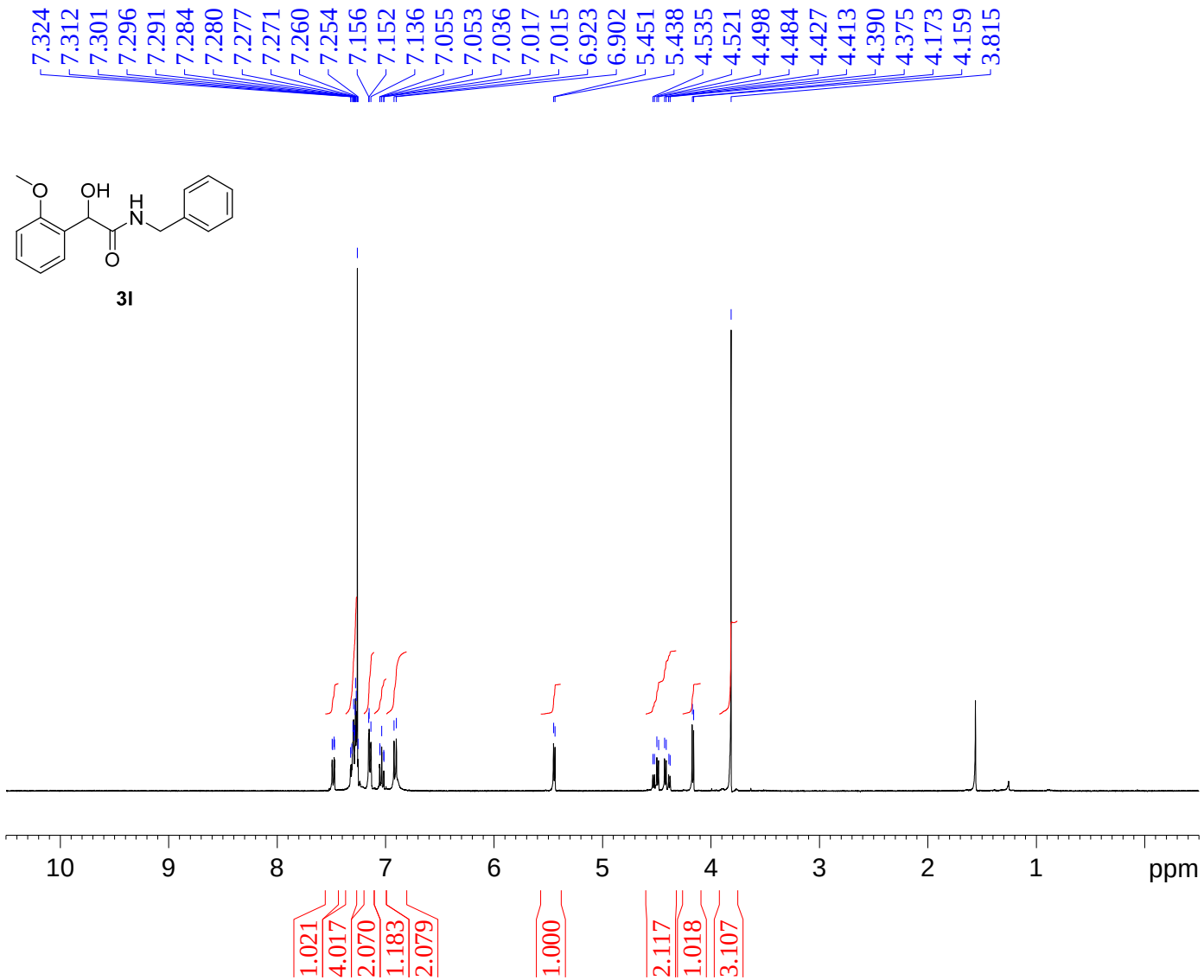


NAME Substrate 1H
EXPNO 10
PROCNO 1
Date_ 20150719
Time 14.05
INSTRUM spect
PROBHD 5 mm DUL 13C-1
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 32
DS 0
SWH 6410.256 Hz
FIDRES 0.195625 Hz
AQ 2.5559540 sec
RG 4
DW 78.000 usec
DE 6.00 usec
TE 300.0 K
D1 2.00000000 sec
TD0 1

======== CHANNEL f1 ========
NUC1 1H
P1 10.00 usec
PL1 -2.40 dB
SFO1 400.1528010 MHz
SI 16384
SF 400.1500088 MHz
WDW EM
SSB 0
LB 0.00 Hz
GB 0
PC 1.00

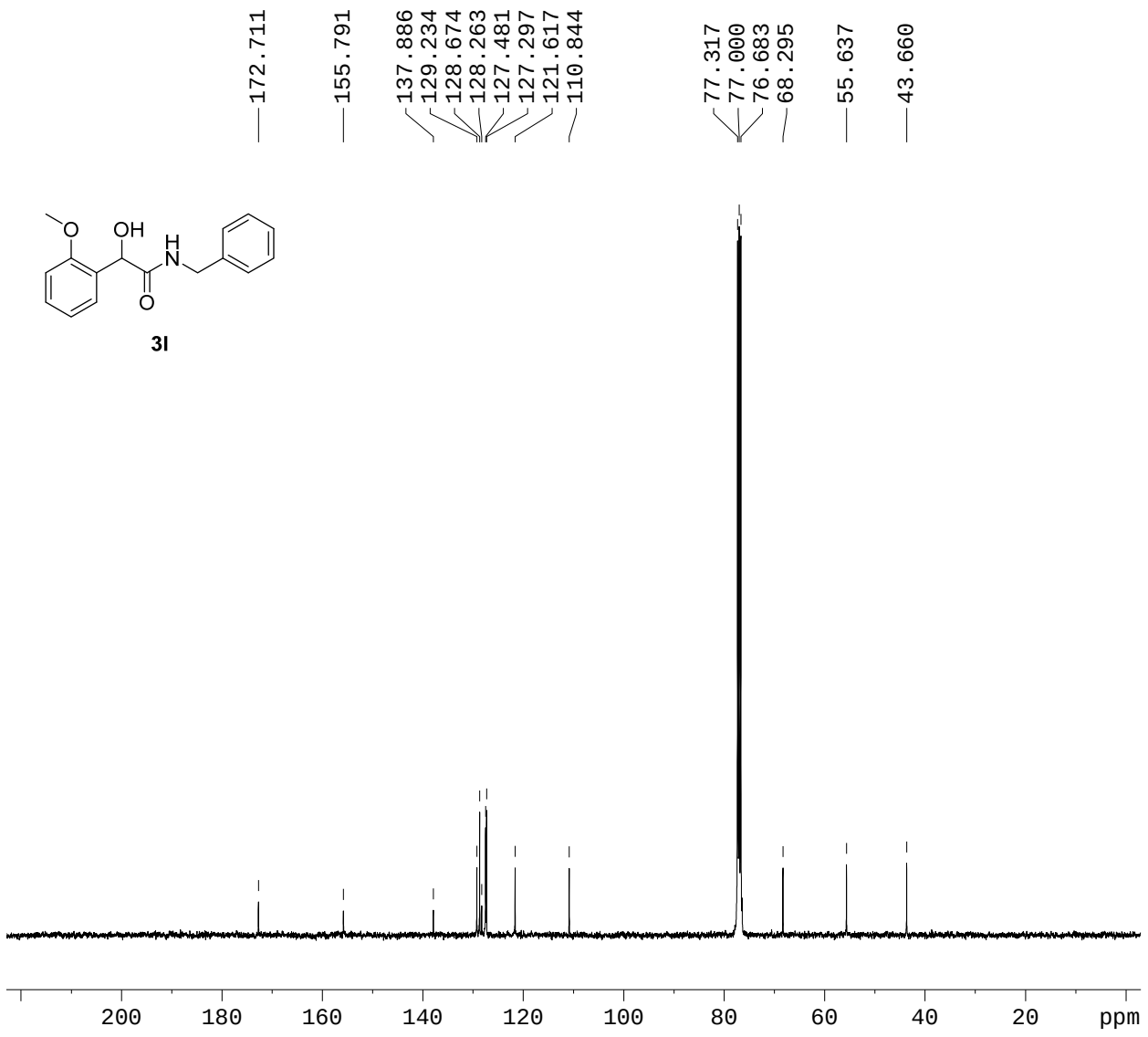


NAME	Substrate 13C	
EXPNO	10	
PROCNO	1	
Date_	20150719	
Time	14.09	
INSTRUM	spect	
PROBHD	5 mm DUL 13C-1	
PULPROG	zgpg30	
TD	65536	
SOLVENT	CDCl3	
NS	1239	
DS	0	
SWH	22727.273	Hz
FIDRES	0.346791	Hz
AQ	1.4418420	sec
RG	57	
DW	22.000	usec
DE	6.00	usec
TE	300.0	K
D1	2.00000000	sec
d11	0.03000000	sec
DELTA	1.89999998	sec
TD0	1	
======== CHANNEL f1 ========		
NUC1	13C	
P1	9.70	usec
PL1	-0.50	dB
SFO1	100.6288660	MHz
======== CHANNEL f2 ========		
CPDPRG2	waltz16	
NUC2	1H	
PCPD2	90.00	usec
PL2	-2.40	dB
PL12	15.10	dB
PL13	18.10	dB
SFO2	400.1516010	MHz
SI	32768	
SF	100.6178033	MHz
WDW	EM	
SSB	0	
LB	3.00	Hz
GB	0	
PC	1.00	

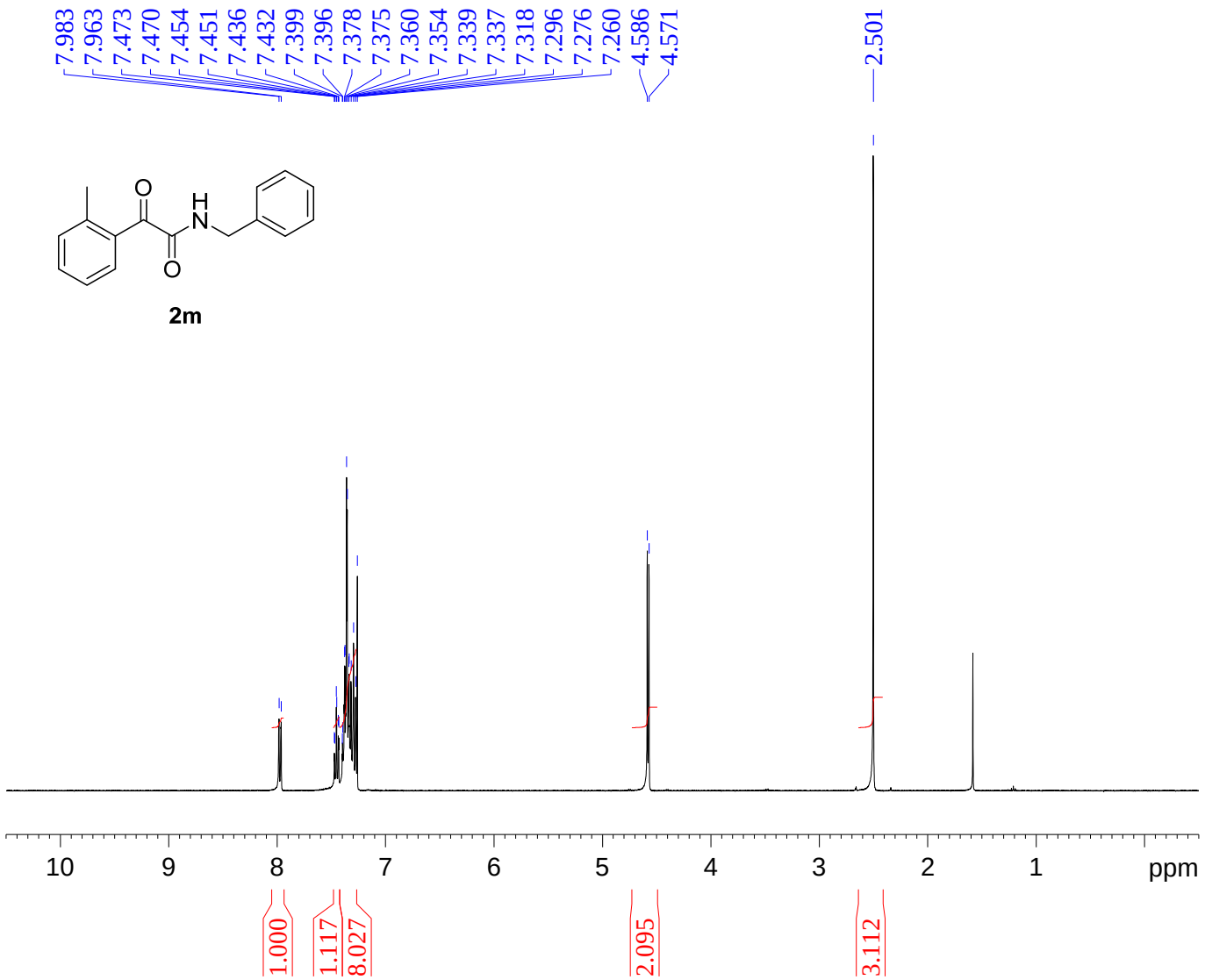


NAME Substrate 1H
EXPNO 9
PROCNO 1
Date_ 20150718
Time 12.28
INSTRUM spect
PROBHD 5 mm DUL 13C-1
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 40
DS 0
SWH 6410.256 Hz
FIDRES 0.195625 Hz
AQ 2.5559540 sec
RG 4
DW 78.000 usec
DE 6.00 usec
TE 300.0 K
D1 2.00000000 sec
TD0 1

======== CHANNEL f1 ========
NUC1 1H
P1 10.00 usec
PL1 -2.40 dB
SFO1 400.1528010 MHz
SI 16384
SF 400.1500088 MHz
WDW EM
SSB 0
LB 0.00 Hz
GB 0
PC 1.00

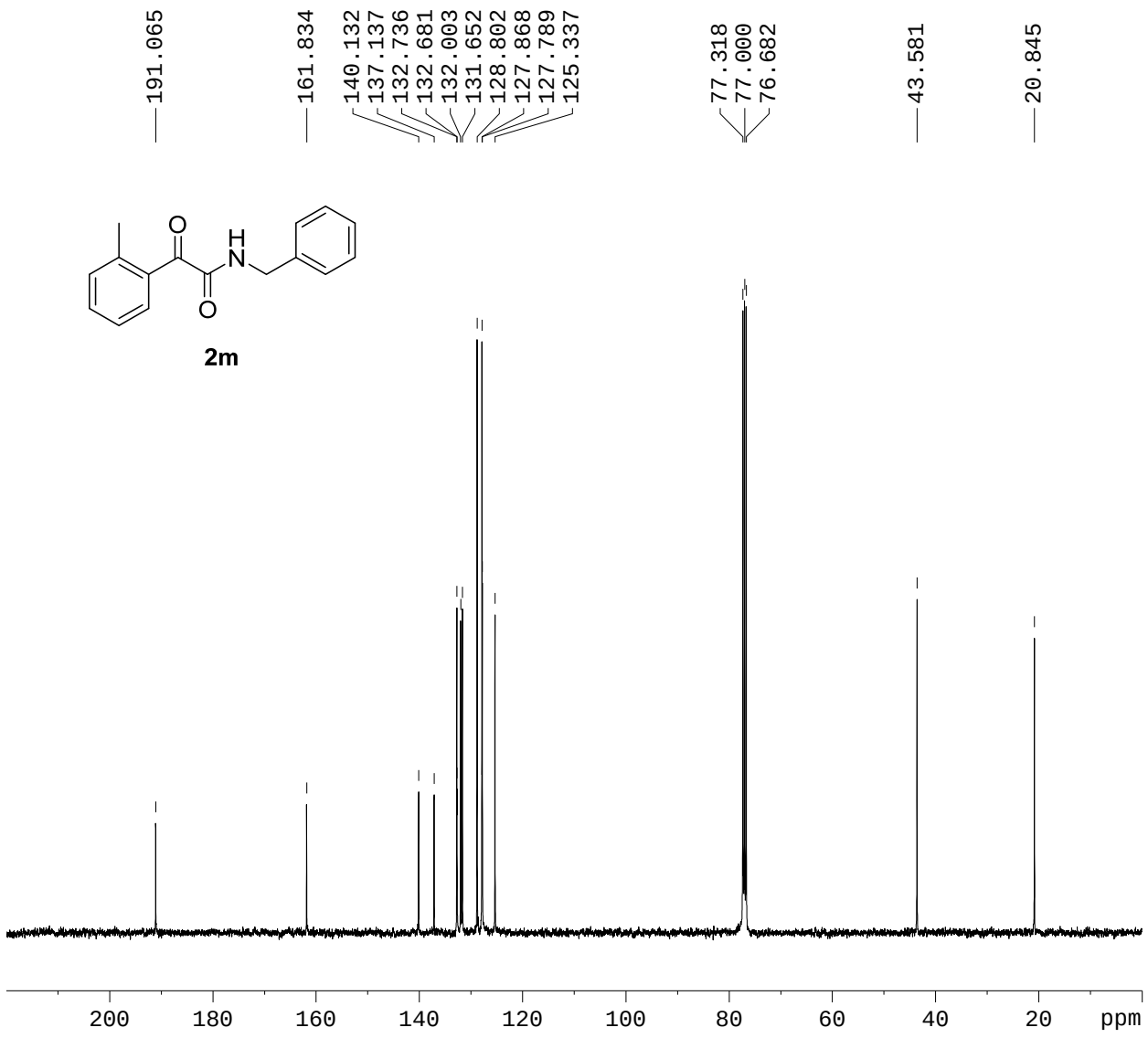



```
NAME       Substrate 13C
EXPNO                 9
PROCNO                1
Date_          20150718
Time              12.36
INSTRUM           spect
PROBHD   5 mm DUL 13C-1
PULPROG         zgpg30
TD                65536
SOLVENT           CDCl3
NS                 2359
DS                    0
SWH           22727.273 Hz
FIDRES         0.346791 Hz
AQ            1.4418420 sec
RG                   57
DW               22.000 usec
DE                6.00 usec
TE               300.0 K
D1            2.00000000 sec
d11           0.03000000 sec
DELTA         1.89999998 sec
TD0                   1

======== CHANNEL f1 ========
NUC1                 13C
P1                9.70 usec
PL1              -0.50 dB
SFO1        100.6288660 MHz

======== CHANNEL f2 ========
CPDPRG2          waltz16
NUC2                 1H
PCPD2             90.00 usec
PL2               -2.40 dB
PL12              15.10 dB
PL13              18.10 dB
SFO2        400.1516010 MHz
SI                32768
SF          100.6177996 MHz
WDW                  EM
SSB                   0
LB                3.00 Hz
GB                    0
PC                 1.00
```

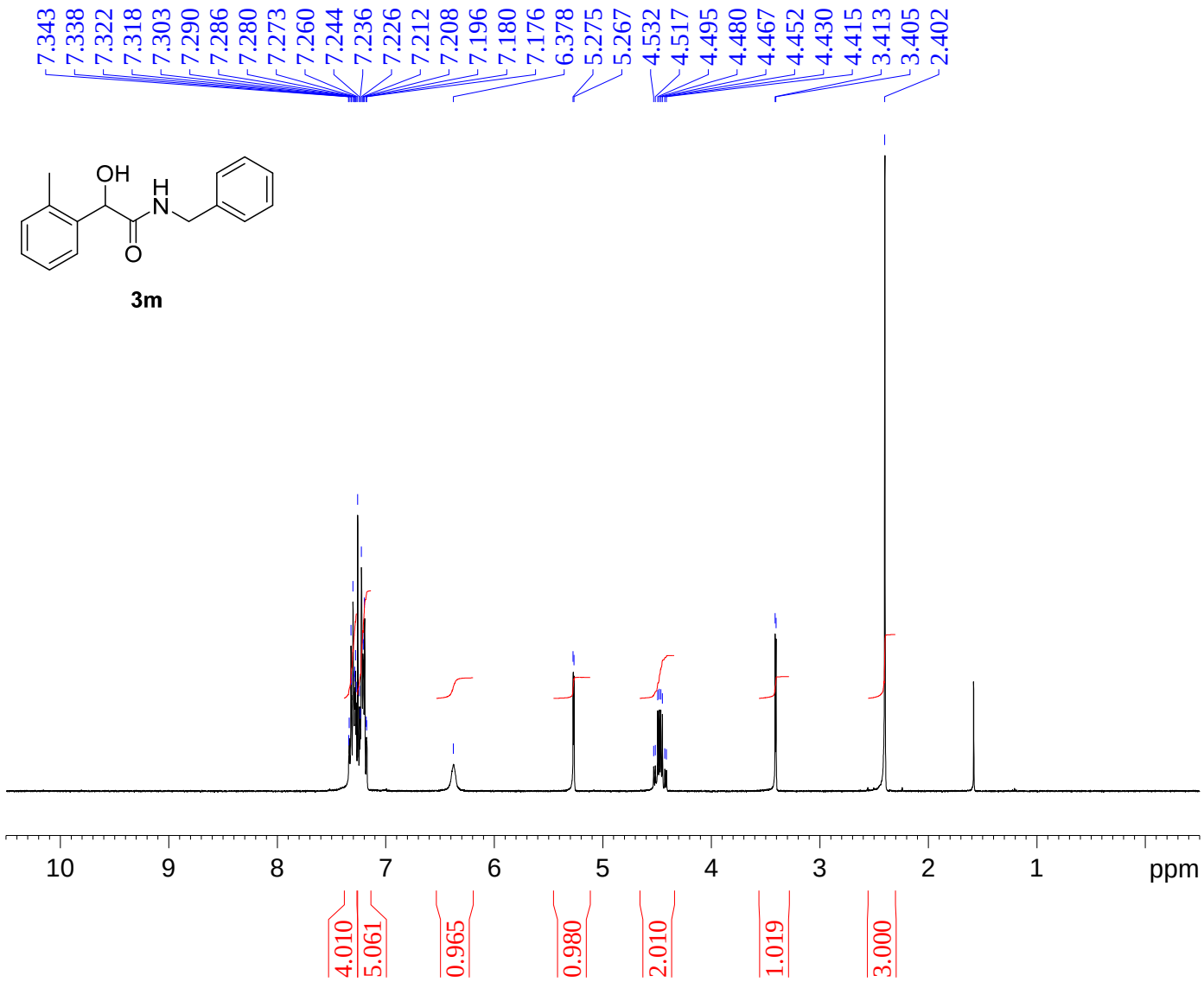


NAME 20151115
EXPNO 1
PROCNO 1
Date_ 20151115
Time 20.05
INSTRUM spect
PROBHD 5 mm DUL 13C-1
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 16
DS 0
SWH 6410.256 Hz
FIDRES 0.195625 Hz
AQ 2.5559540 sec
RG 4
DW 78.000 usec
DE 6.00 usec
TE 300.0 K
D1 2.00000000 sec
TD0 1

======== CHANNEL f1 ========
NUC1 1H
P1 10.00 usec
PL1 -2.40 dB
SFO1 400.1528010 MHz
SI 16384
SF 400.1500089 MHz
WDW EM
SSB 0
LB 0.00 Hz
GB 0
PC 1.00

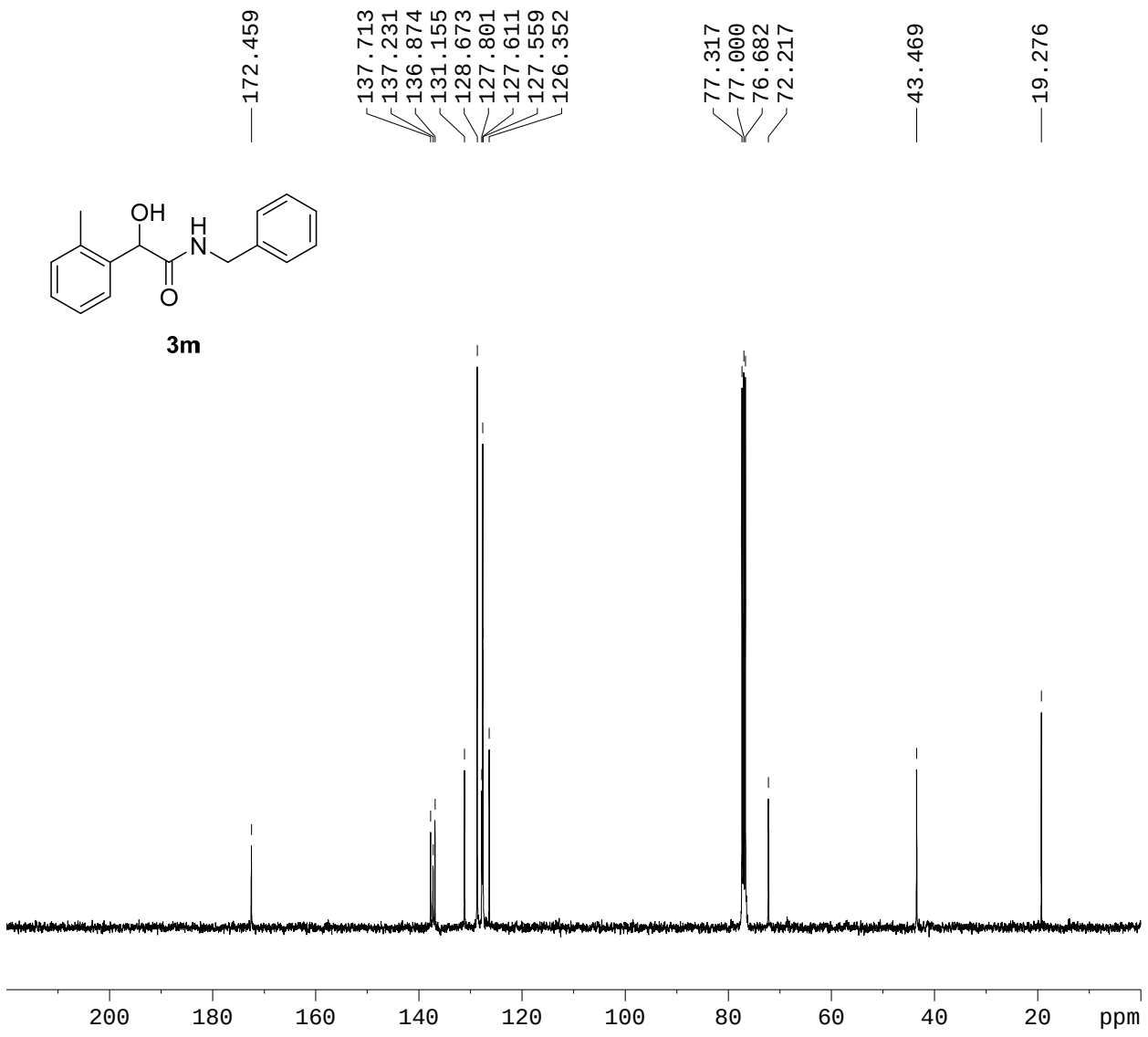


NAME	20151115	
EXPNO	2	
PROCNO	1	
Date_	20151115	
Time	20.12	
INSTRUM	spect	
PROBHD	5 mm DUL 13C-1	
PULPROG	zgpg30	
TD	65536	
SOLVENT	CDCl3	
NS	756	
DS	0	
SWH	22727.273	Hz
FIDRES	0.346791	Hz
AQ	1.4418420	sec
RG	57	
DW	22.000	usec
DE	6.00	usec
TE	300.0	K
D1	2.00000000	sec
d11	0.03000000	sec
DELTA	1.89999998	sec
TD0	1	
======== CHANNEL f1 ========		
NUC1	13C	
P1	9.70	usec
PL1	-0.50	dB
SFO1	100.6288660	MHz
======== CHANNEL f2 ========		
CPDPRG2	waltz16	
NUC2	1H	
PCPD2	90.00	usec
PL2	-2.40	dB
PL12	15.10	dB
PL13	18.10	dB
SFO2	400.1516010	MHz
SI	32768	
SF	100.6178050	MHz
WDW	EM	
SSB	0	
LB	3.00	Hz
GB	0	
PC	1.00	

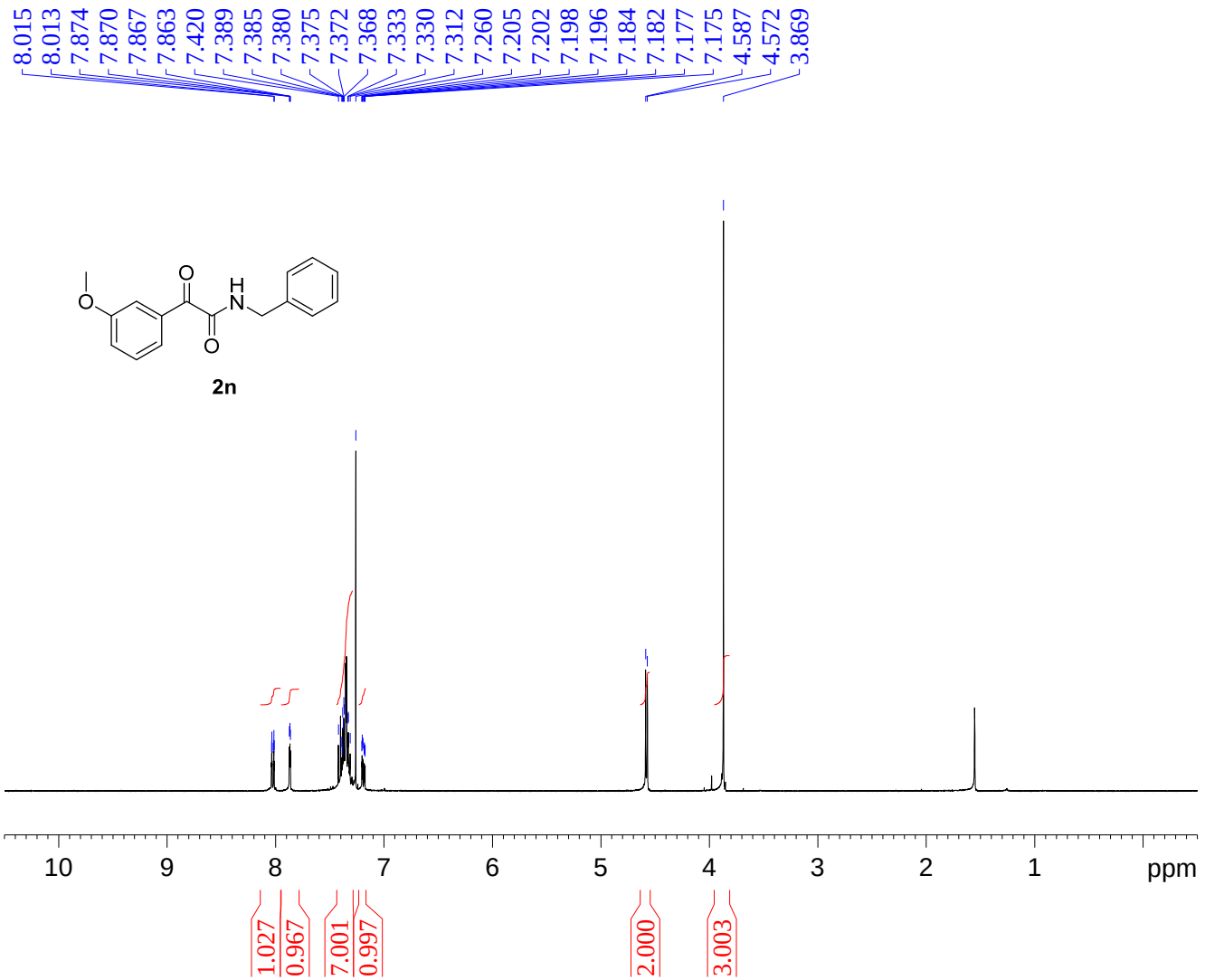


NAME 20160114
EXPNO 2
PROCNO 1
Date_ 20160114
Time 20.27
INSTRUM spect
PROBHD 5 mm DUL 13C-1
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 13
DS 0
SWH 6410.256 Hz
FIDRES 0.195625 Hz
AQ 2.5559540 sec
RG 4
DW 78.000 usec
DE 6.00 usec
TE 300.0 K
D1 2.00000000 sec
TD0 1

======== CHANNEL f1 ========
NUC1 1H
P1 10.00 usec
PL1 -2.40 dB
SFO1 400.1528010 MHz
SI 16384
SF 400.1500091 MHz
WDW EM
SSB 0
LB 0.00 Hz
GB 0
PC 1.00

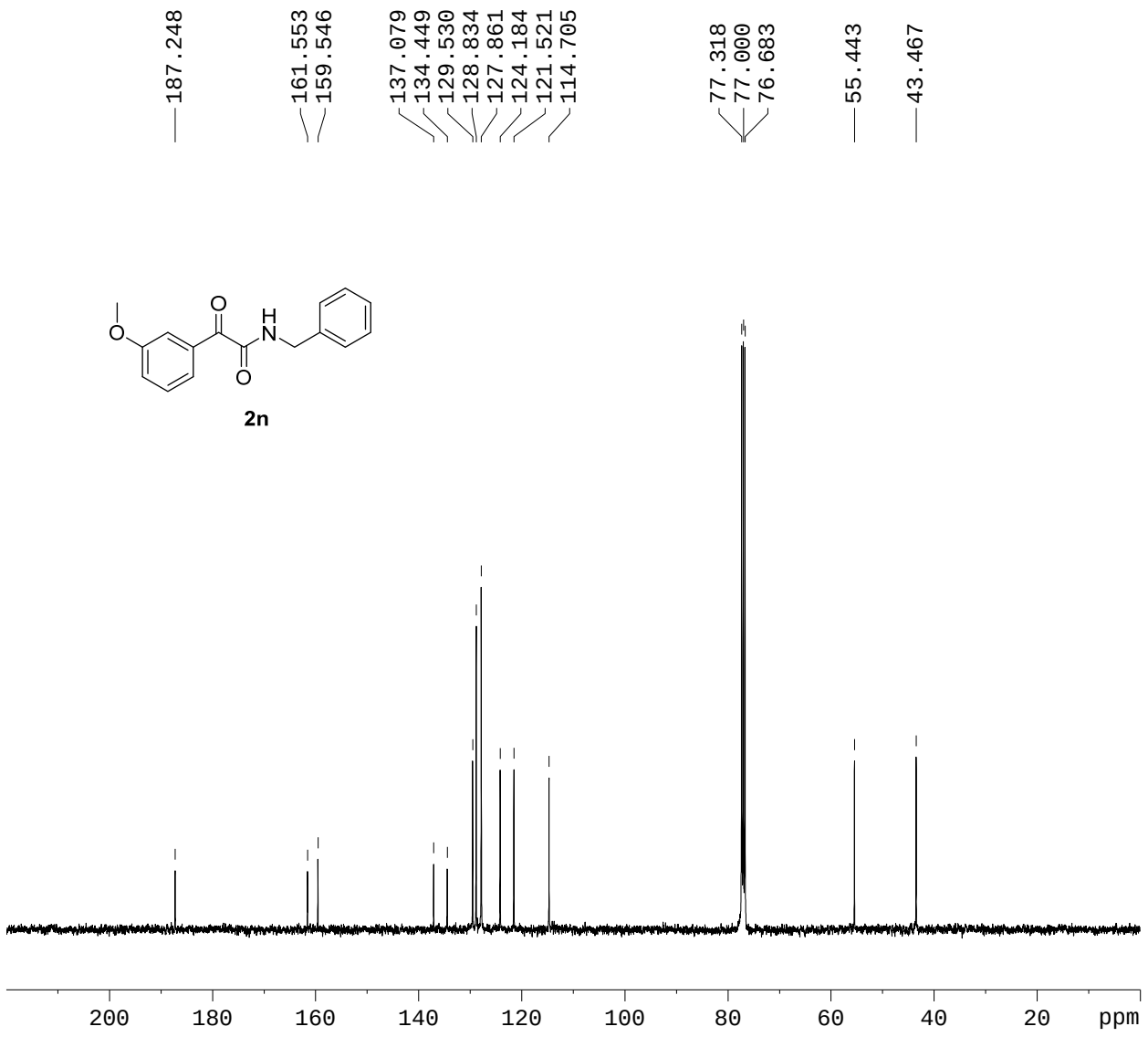


NAME	Substrate 13C
EXPNO	22
PROCNO	1
Date_	20150814
Time	20.30
INSTRUM	spect
PROBHD	5 mm DUL 13C-1
PULPROG	zgpg30
TD	65536
SOLVENT	CDCl3
NS	460
DS	0
SWH	22727.273 Hz
FIDRES	0.346791 Hz
AQ	1.4418420 sec
RG	57
DW	22.000 usec
DE	6.00 usec
TE	300.0 K
D1	2.00000000 sec
d11	0.03000000 sec
DELTA	1.89999998 sec
TD0	1
======== CHANNEL f1 ========	
NUC1	13C
P1	9.70 usec
PL1	-0.50 dB
SFO1	100.6288660 MHz
======== CHANNEL f2 ========	
CPDPRG2	waltz16
NUC2	1H
PCPD2	90.00 usec
PL2	-2.40 dB
PL12	15.10 dB
PL13	18.10 dB
SFO2	400.1516010 MHz
SI	32768
SF	100.6178037 MHz
WDW	EM
SSB	0
LB	3.00 Hz
GB	0
PC	1.00

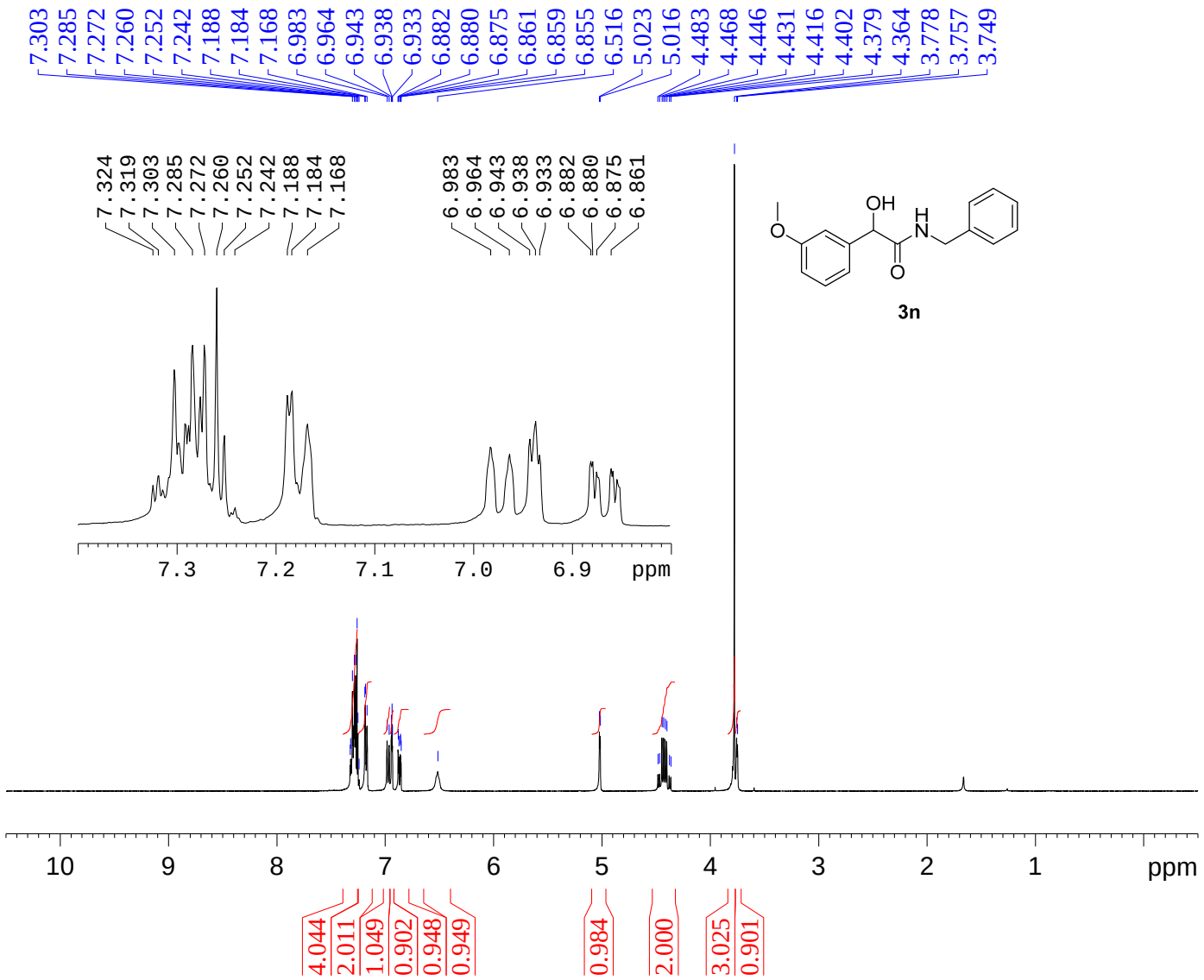


NAME 20151125
EXPNO 1
PROCNO 1
Date_ 20151125
Time 19.08
INSTRUM spect
PROBHD 5 mm DUL 13C-1
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 30
DS 0
SWH 6410.256 Hz
FIDRES 0.195625 Hz
AQ 2.5559540 sec
RG 4
DW 78.000 usec
DE 6.00 usec
TE 300.0 K
D1 2.00000000 sec
TD0 1

======== CHANNEL f1 ========
NUC1 1H
P1 10.00 usec
PL1 -2.40 dB
SFO1 400.1528010 MHz
SI 16384
SF 400.1500089 MHz
WDW EM
SSB 0
LB 0.00 Hz
GB 0
PC 1.00

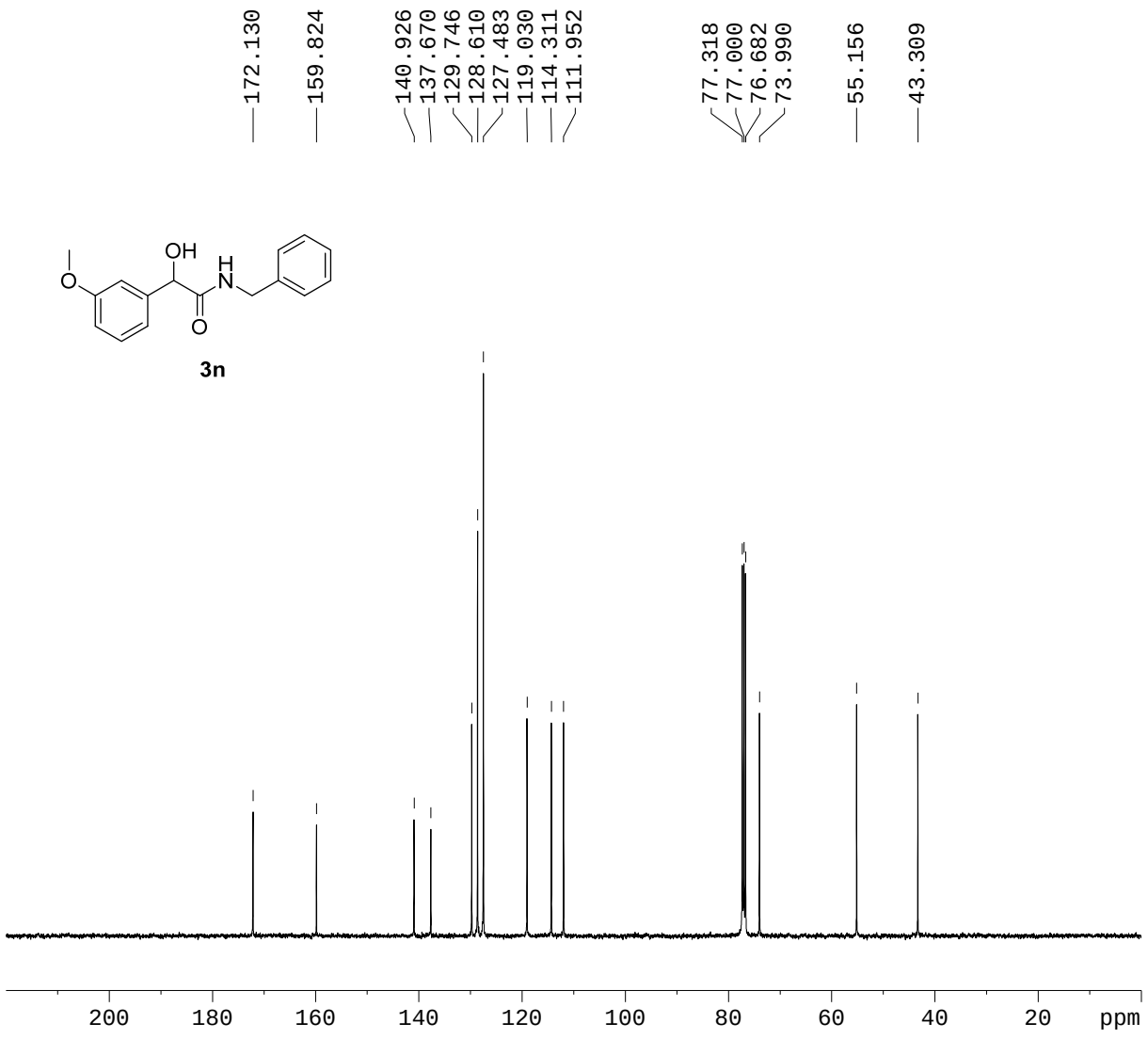


NAME	20151125
EXPNO	2
PROCNO	1
Date_	20151125
Time	19.38
INSTRUM	spect
PROBHD	5 mm DUL 13C-1
PULPROG	zgpg30
TD	65536
SOLVENT	CDCl3
NS	492
DS	0
SWH	22727.273 Hz
FIDRES	0.346791 Hz
AQ	1.4418420 sec
RG	57
DW	22.000 usec
DE	6.00 usec
TE	300.0 K
D1	2.00000000 sec
d11	0.03000000 sec
DELTA	1.89999998 sec
TD0	1
======== CHANNEL f1 ========	
NUC1	13C
P1	9.70 usec
PL1	-0.50 dB
SFO1	100.6288660 MHz
======== CHANNEL f2 ========	
CPDPRG2	waltz16
NUC2	1H
PCPD2	90.00 usec
PL2	-2.40 dB
PL12	15.10 dB
PL13	18.10 dB
SFO2	400.1516010 MHz
SI	32768
SF	100.6178017 MHz
WDW	EM
SSB	0
LB	3.00 Hz
GB	0
PC	1.00

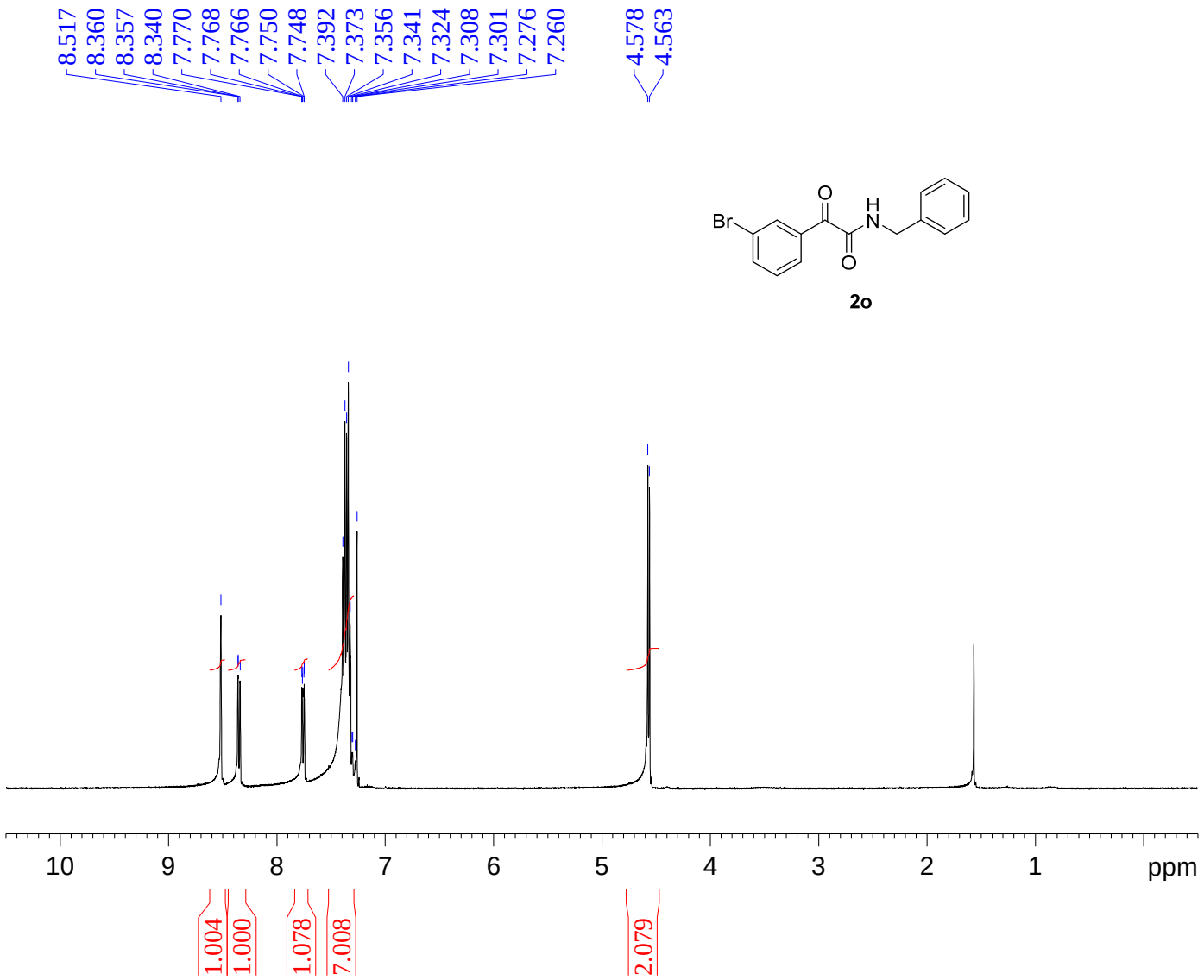


NAME 20151116
EXPNO 1
PROCNO 1
Date_ 20151116
Time 20.08
INSTRUM spect
PROBHD 5 mm DUL 13C-1
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 16
DS 0
SWH 6410.256 Hz
FIDRES 0.195625 Hz
AQ 2.5559540 sec
RG 4
DW 78.000 usec
DE 6.00 usec
TE 300.0 K
D1 2.00000000 sec
TD0 1

======== CHANNEL f1 ========
NUC1 1H
P1 10.00 usec
PL1 -2.40 dB
SFO1 400.1528010 MHz
SI 16384
SF 400.1500089 MHz
WDW EM
SSB 0
LB 0.00 Hz
GB 0
PC 1.00

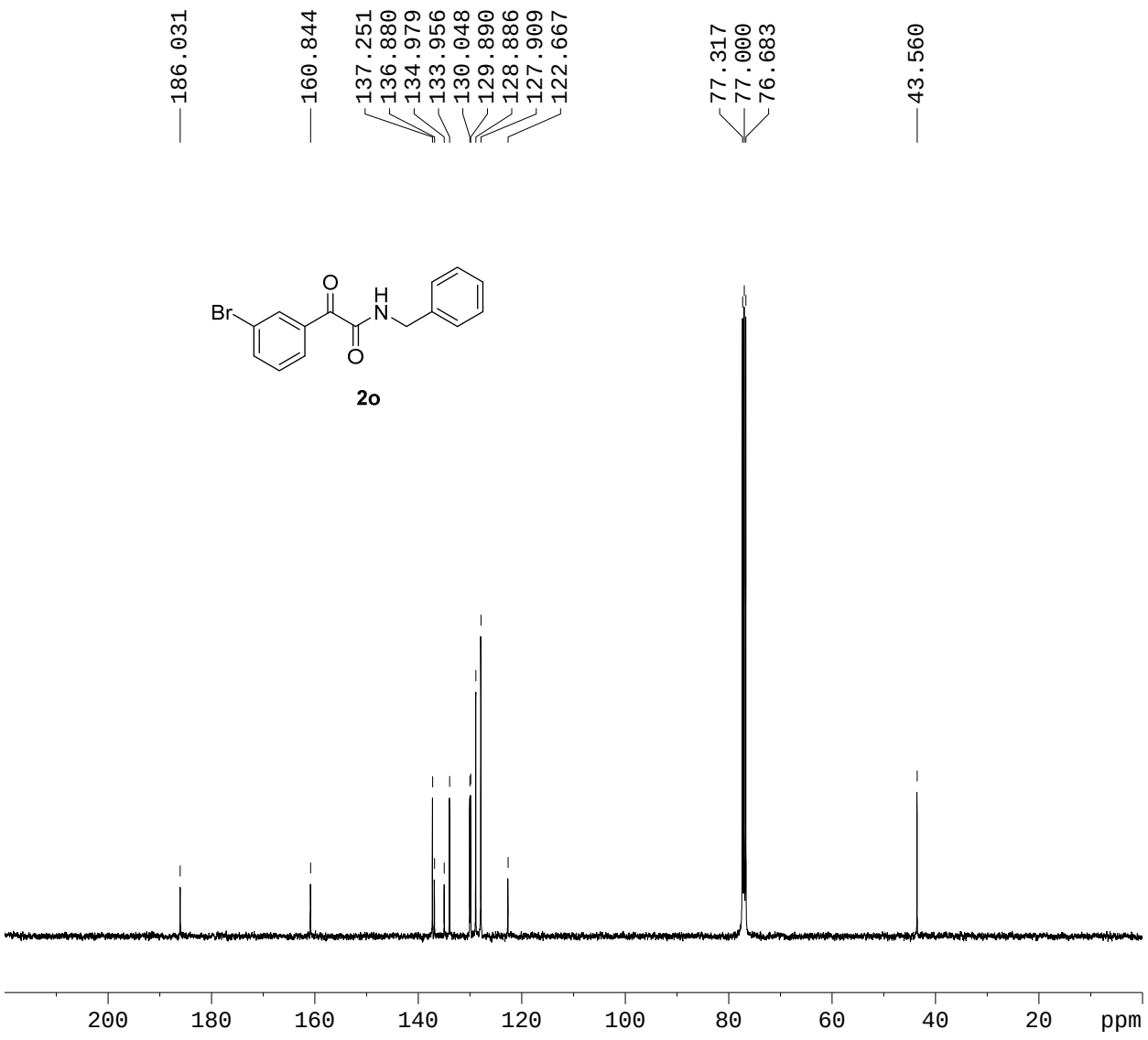


NAME	20151116
EXPNO	2
PROCNO	1
Date_	20151116
Time	20.15
INSTRUM	spect
PROBHD	5 mm DUL 13C-1
PULPROG	zgpg30
TD	65536
SOLVENT	CDCl3
NS	748
DS	0
SWH	22727.273 Hz
FIDRES	0.346791 Hz
AQ	1.4418420 sec
RG	57
DW	22.000 usec
DE	6.00 usec
TE	300.0 K
D1	2.00000000 sec
d11	0.03000000 sec
DELTA	1.89999998 sec
TD0	1
======== CHANNEL f1 ========	
NUC1	13C
P1	9.70 usec
PL1	-0.50 dB
SFO1	100.6288660 MHz
======== CHANNEL f2 ========	
CPDPRG2	waltz16
NUC2	1H
PCPD2	90.00 usec
PL2	-2.40 dB
PL12	15.10 dB
PL13	18.10 dB
SFO2	400.1516010 MHz
SI	32768
SF	100.6178066 MHz
WDW	EM
SSB	0
LB	3.00 Hz
GB	0
PC	1.00

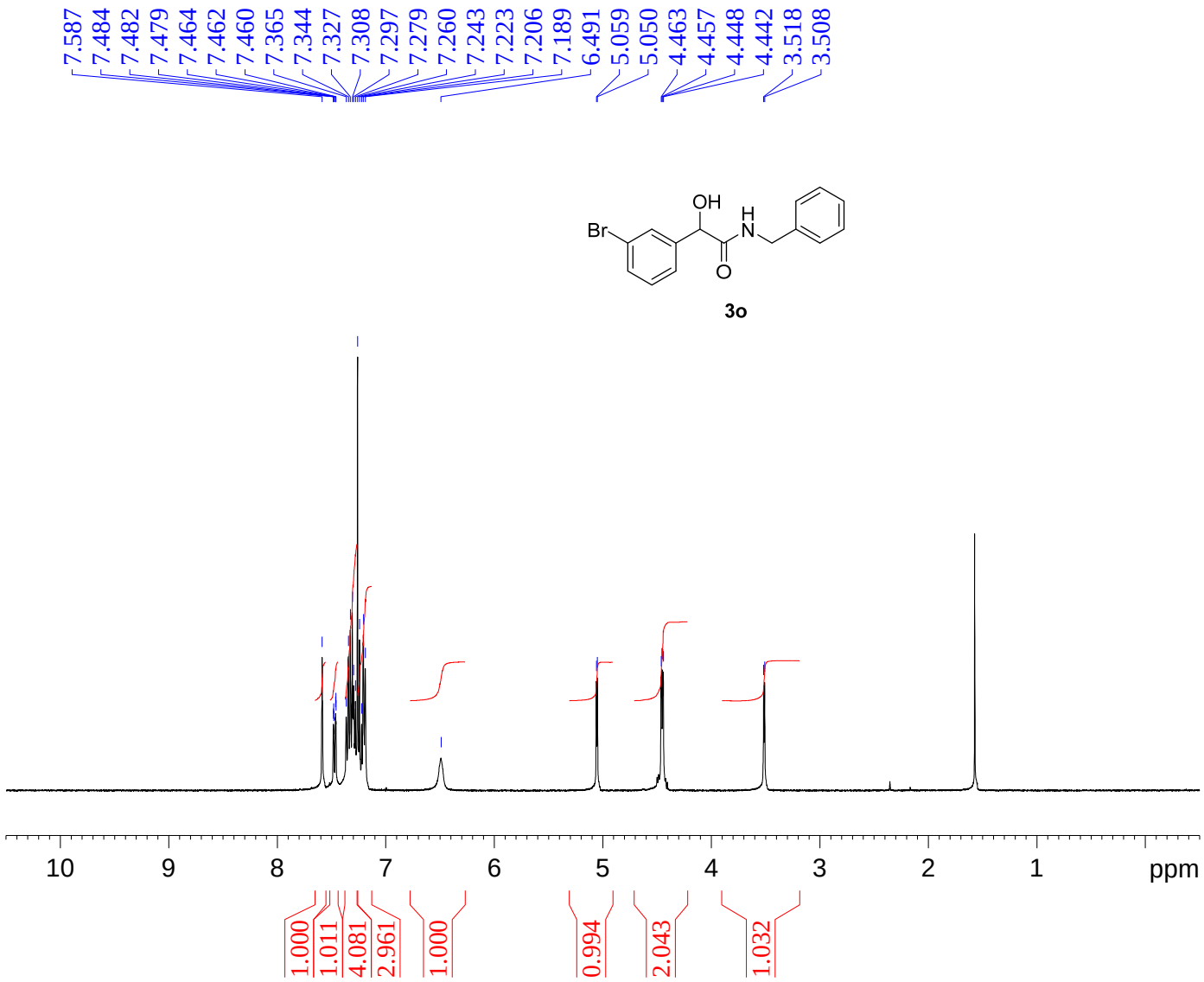


NAME 20151130
EXPNO 4
PROCNO 1
Date_ 20151107
Time 19.23
INSTRUM spect
PROBHD 5 mm DUL 13C-1
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 73
DS 0
SWH 6410.256 Hz
FIDRES 0.195625 Hz
AQ 2.5559540 sec
RG 4
DW 78.000 usec
DE 6.00 usec
TE 300.0 K
D1 2.00000000 sec
TD0 1

======== CHANNEL f1 ========
NUC1 1H
P1 10.00 usec
PL1 -2.40 dB
SFO1 400.1528010 MHz
SI 16384
SF 400.1500088 MHz
WDW EM
SSB 0
LB 0.00 Hz
GB 0
PC 1.00

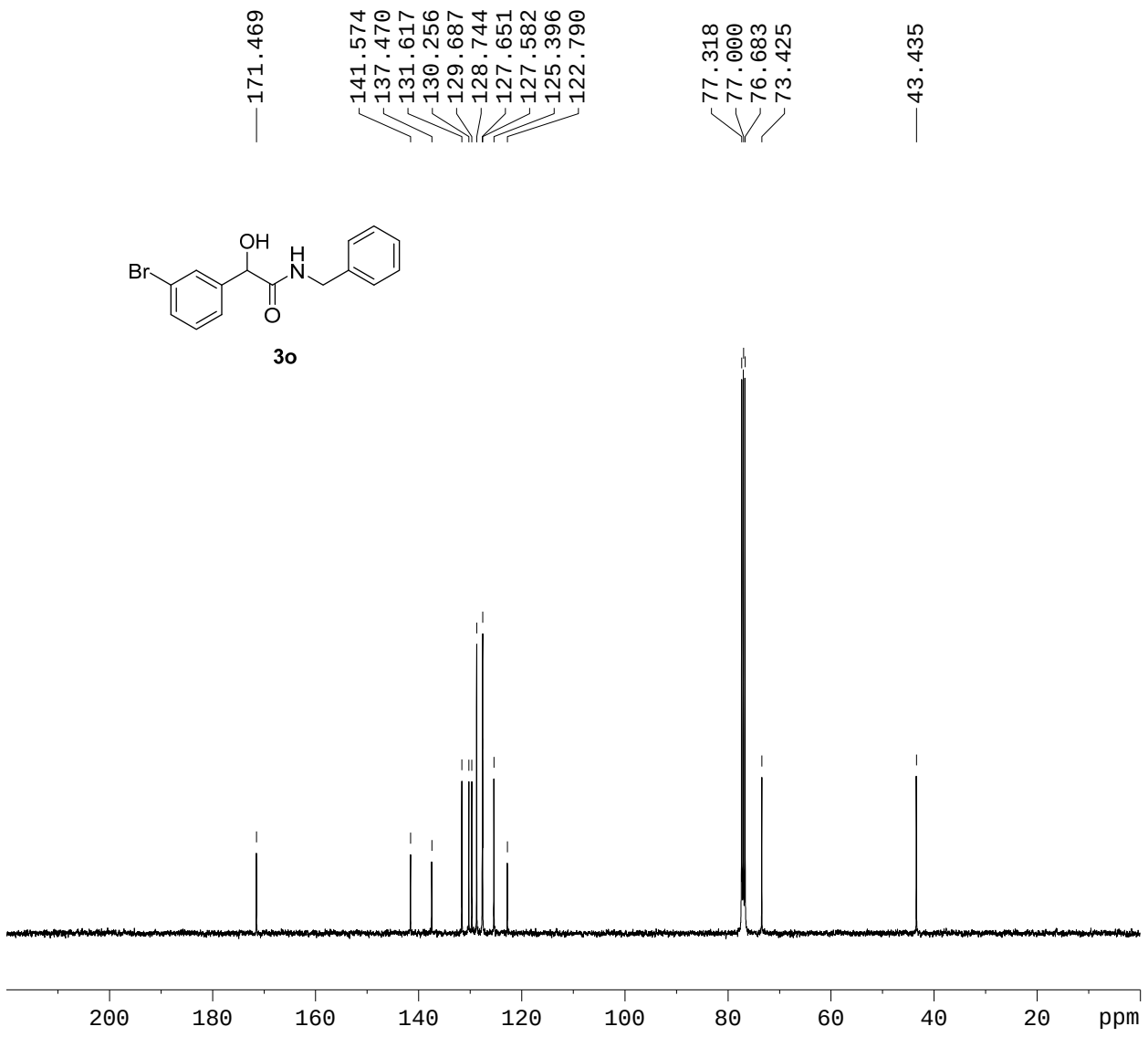


NAME	20151113	
EXPNO	1	
PROCNO	1	
Date_	20151113	
Time	20.02	
INSTRUM	spect	
PROBHD	5 mm DUL 13C-1	
PULPROG	zgpg30	
TD	65536	
SOLVENT	CDCl3	
NS	993	
DS	0	
SWH	22727.273	Hz
FIDRES	0.346791	Hz
AQ	1.4418420	sec
RG	57	
DW	22.000	usec
DE	6.00	usec
TE	300.0	K
D1	2.00000000	sec
d11	0.03000000	sec
DELTA	1.89999998	sec
TD0	1	
======== CHANNEL f1 ========		
NUC1	13C	
P1	9.70	usec
PL1	-0.50	dB
SFO1	100.6288660	MHz
======== CHANNEL f2 ========		
CPDPRG2	waltz16	
NUC2	1H	
PCPD2	90.00	usec
PL2	-2.40	dB
PL12	15.10	dB
PL13	18.10	dB
SFO2	400.1516010	MHz
SI	32768	
SF	100.6178009	MHz
WDW	EM	
SSB	0	
LB	3.00	Hz
GB	0	
PC	1.00	

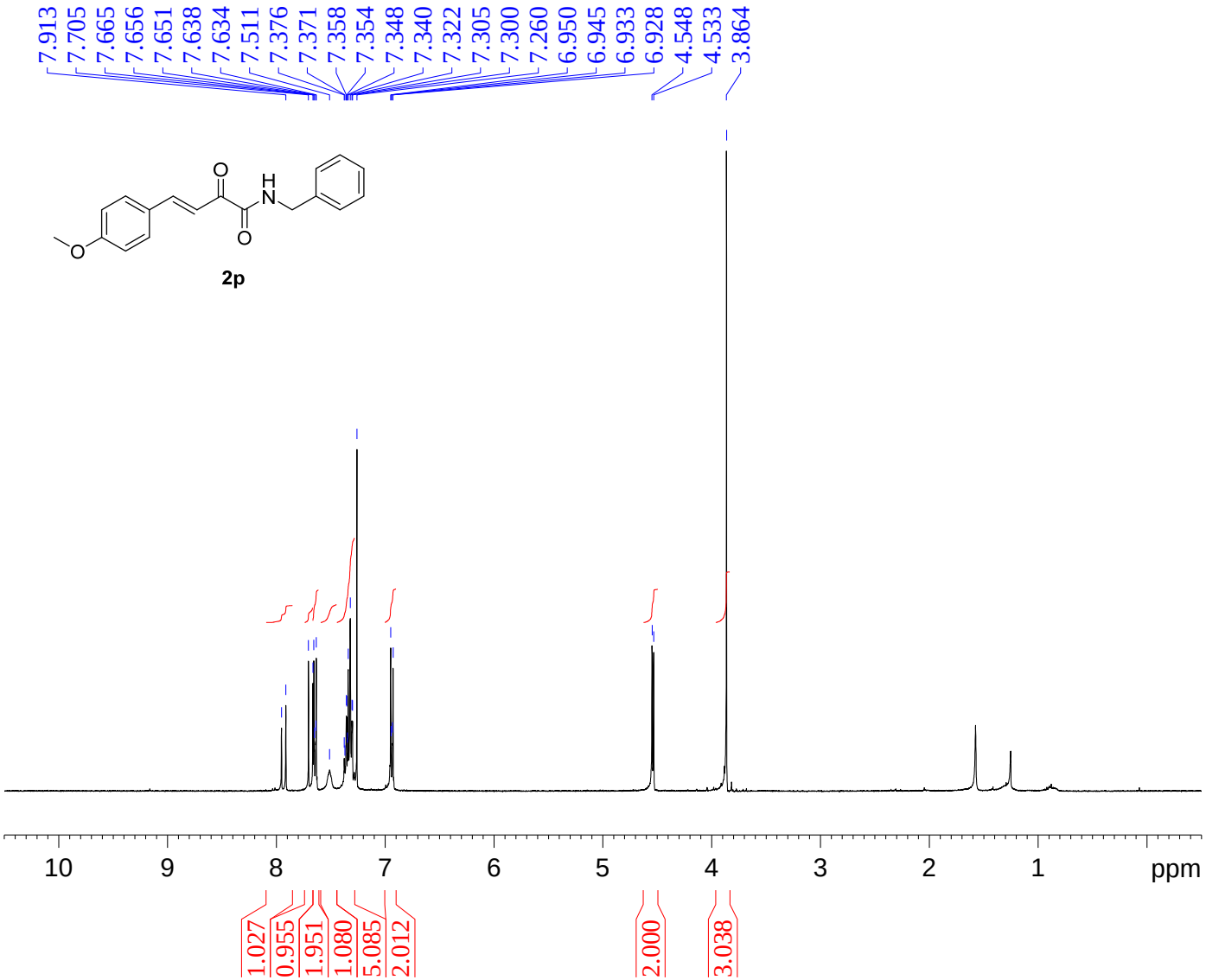


NAME 20151208
EXPNO 1
PROCNO 1
Date_ 20151208
Time 21.13
INSTRUM spect
PROBHD 5 mm DUL 13C-1
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 64
DS 0
SWH 6410.256 Hz
FIDRES 0.195625 Hz
AQ 2.5559540 sec
RG 4
DW 78.000 usec
DE 6.00 usec
TE 300.0 K
D1 2.00000000 sec
TD0 1

======== CHANNEL f1 ========
NUC1 1H
P1 10.00 usec
PL1 -2.40 dB
SFO1 400.1528010 MHz
SI 16384
SF 400.1500093 MHz
WDW EM
SSB 0
LB 0.00 Hz
GB 0
PC 1.00

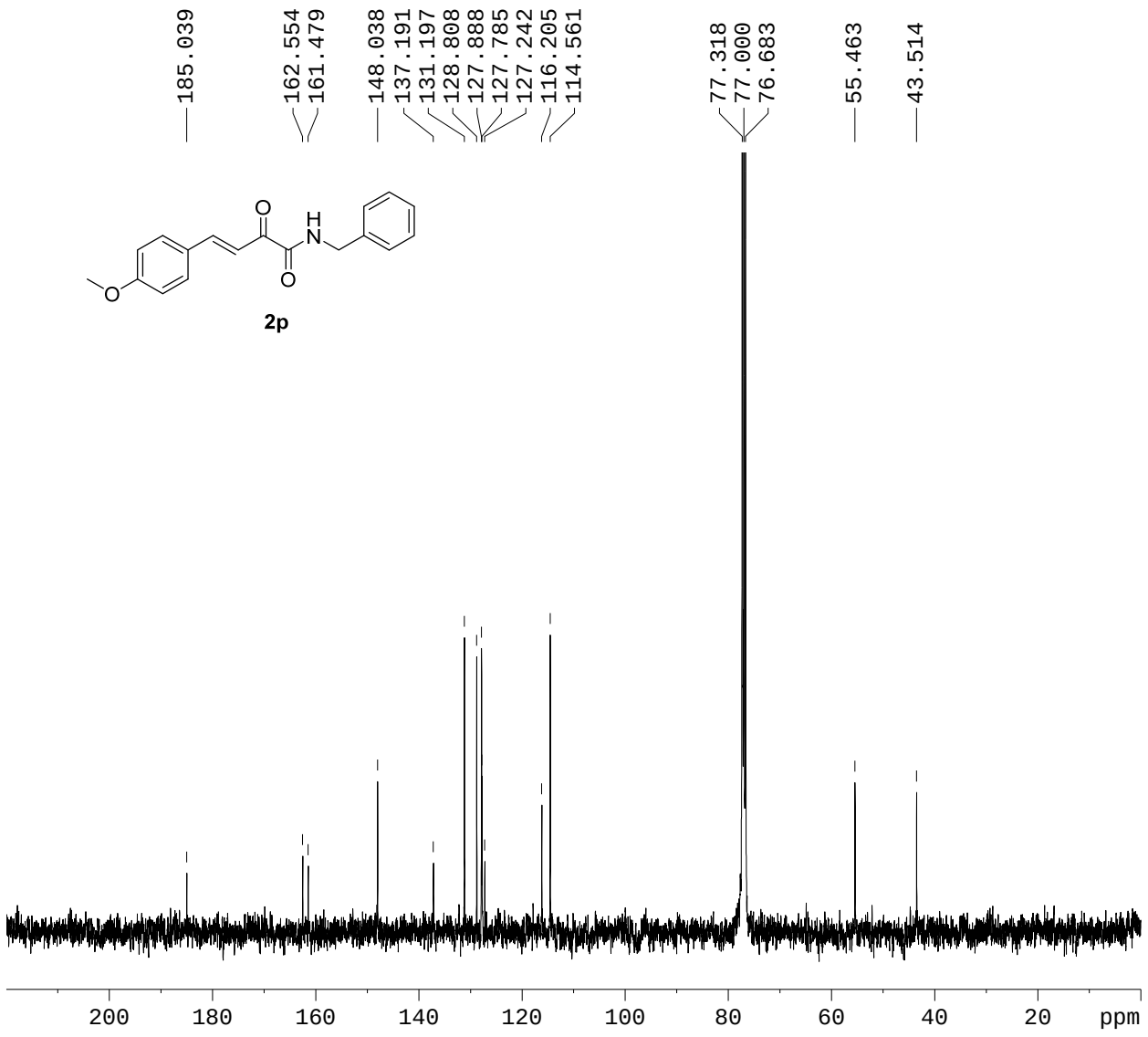


NAME	20151114	
EXPNO	1	
PROCNO	1	
Date_	20151114	
Time	20.09	
INSTRUM	spect	
PROBHD	5 mm DUL 13C-1	
PULPROG	zgpg30	
TD	65536	
SOLVENT	CDCl3	
NS	752	
DS	0	
SWH	22727.273	Hz
FIDRES	0.346791	Hz
AQ	1.4418420	sec
RG	57	
DW	22.000	usec
DE	6.00	usec
TE	300.0	K
D1	2.00000000	sec
d11	0.03000000	sec
DELTA	1.89999998	sec
TD0	1	
======== CHANNEL f1 ========		
NUC1	13C	
P1	9.70	usec
PL1	-0.50	dB
SFO1	100.6288660	MHz
======== CHANNEL f2 ========		
CPDPRG2	waltz16	
NUC2	1H	
PCPD2	90.00	usec
PL2	-2.40	dB
PL12	15.10	dB
PL13	18.10	dB
SFO2	400.1516010	MHz
SI	32768	
SF	100.6178022	MHz
WDW	EM	
SSB	0	
LB	3.00	Hz
GB	0	
PC	1.00	

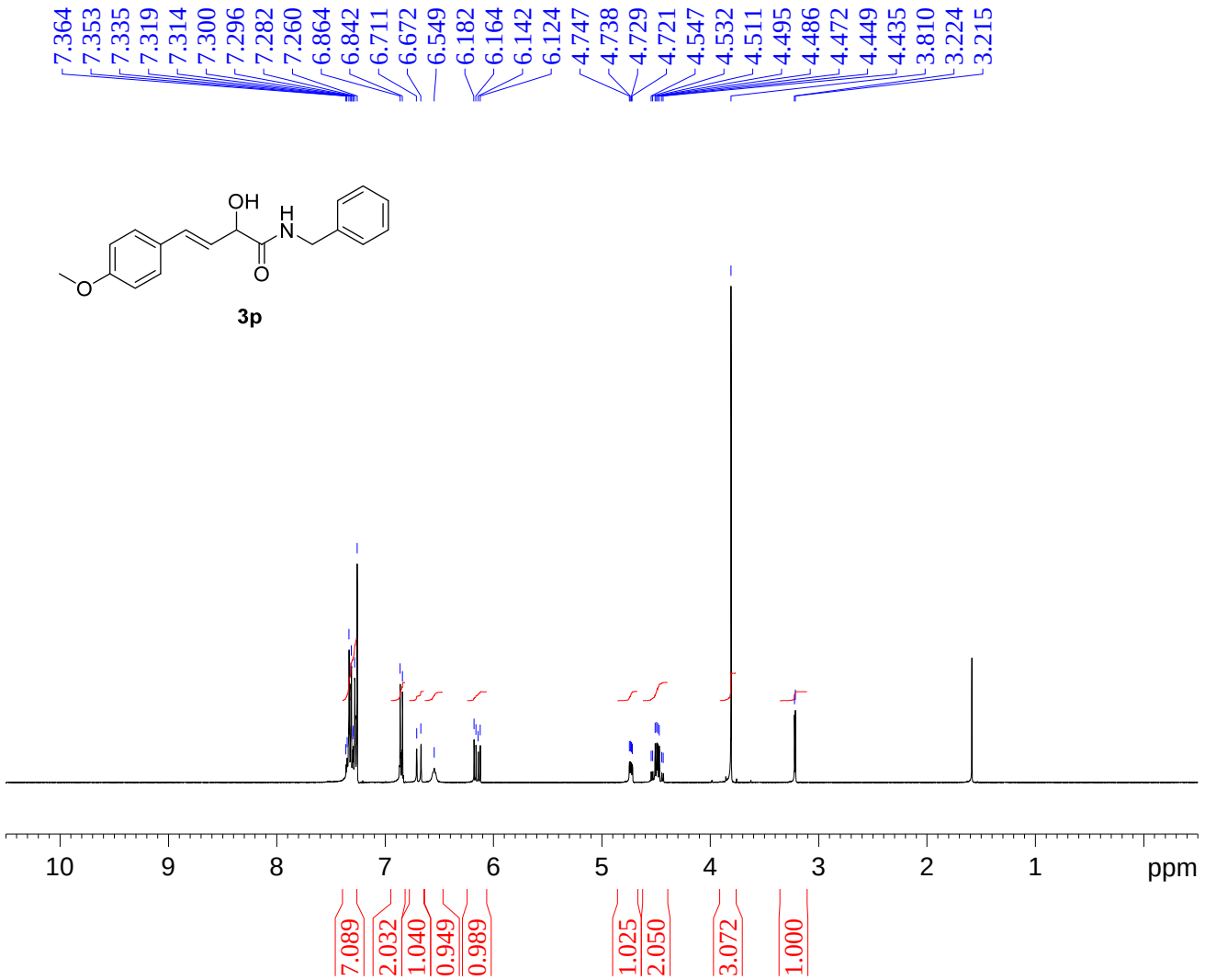


NAME 20151213
EXPNO 1
PROCNO 1
Date_ 20151213
Time 19.00
INSTRUM spect
PROBHD 5 mm DUL 13C-1
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 45
DS 0
SWH 6410.256 Hz
FIDRES 0.195625 Hz
AQ 2.5559540 sec
RG 4
DW 78.000 usec
DE 6.00 usec
TE 300.0 K
D1 2.00000000 sec
TD0 1

======== CHANNEL f1 ========
NUC1 1H
P1 10.00 usec
PL1 -2.40 dB
SFO1 400.1528010 MHz
SI 16384
SF 400.1500088 MHz
WDW EM
SSB 0
LB 0.00 Hz
GB 0
PC 1.00

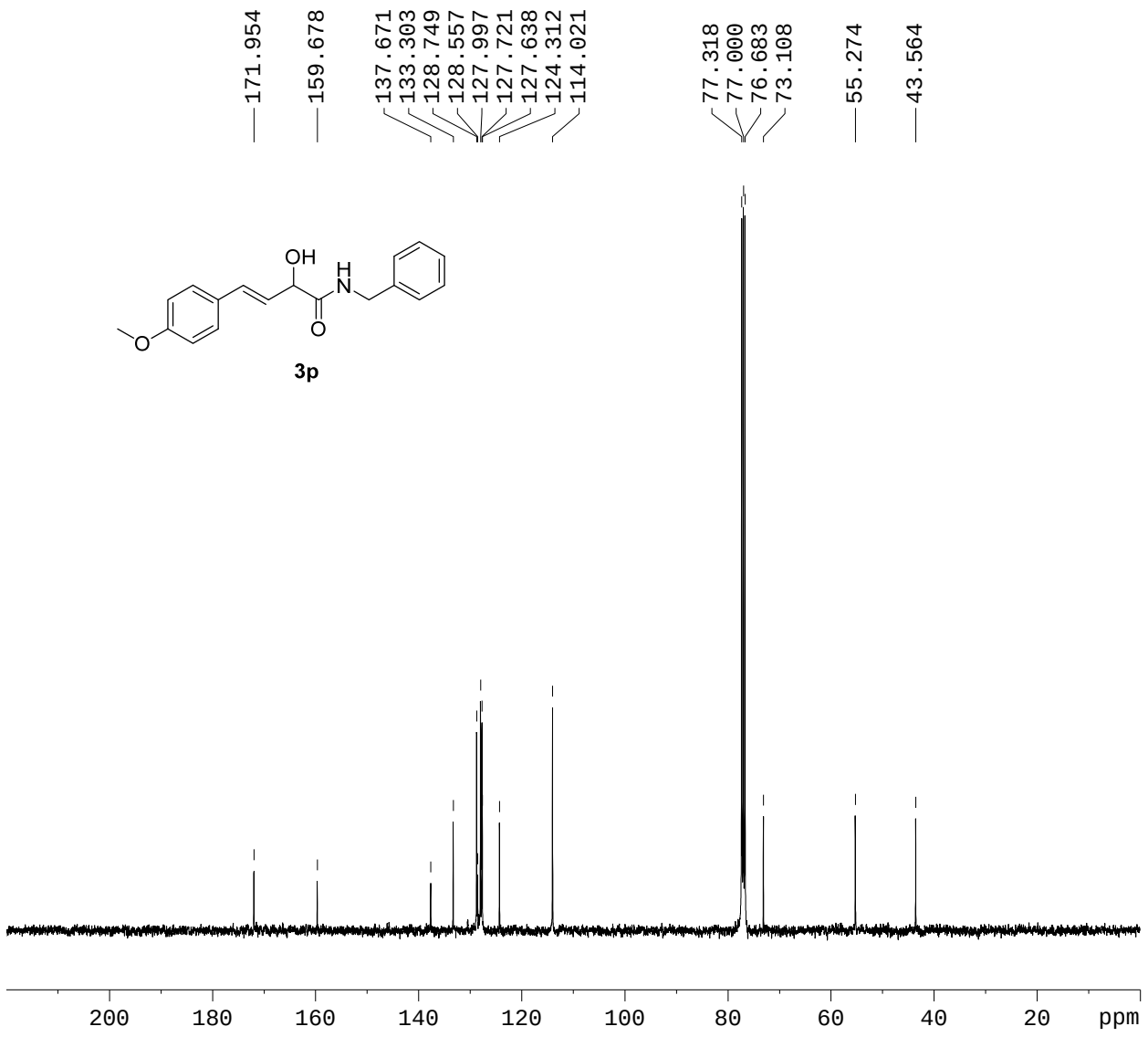


NAME	20151213	
EXPNO	2	
PROCNO	1	
Date_	20151213	
Time	19.05	
INSTRUM	spect	
PROBHD	5 mm DUL 13C-1	
PULPROG	zgpg30	
TD	65536	
SOLVENT	CDCl3	
NS	786	
DS	0	
SWH	22727.273	Hz
FIDRES	0.346791	Hz
AQ	1.4418420	sec
RG	57	
DW	22.000	usec
DE	6.00	usec
TE	300.0	K
D1	2.00000000	sec
d11	0.03000000	sec
DELTA	1.89999998	sec
TD0	1	
======== CHANNEL f1 ========		
NUC1	13C	
P1	9.70	usec
PL1	-0.50	dB
SFO1	100.6288660	MHz
======== CHANNEL f2 ========		
CPDPRG2	waltz16	
NUC2	1H	
PCPD2	90.00	usec
PL2	-2.40	dB
PL12	15.10	dB
PL13	18.10	dB
SFO2	400.1516010	MHz
SI	32768	
SF	100.6177996	MHz
WDW	EM	
SSB	0	
LB	3.00	Hz
GB	0	
PC	1.00	

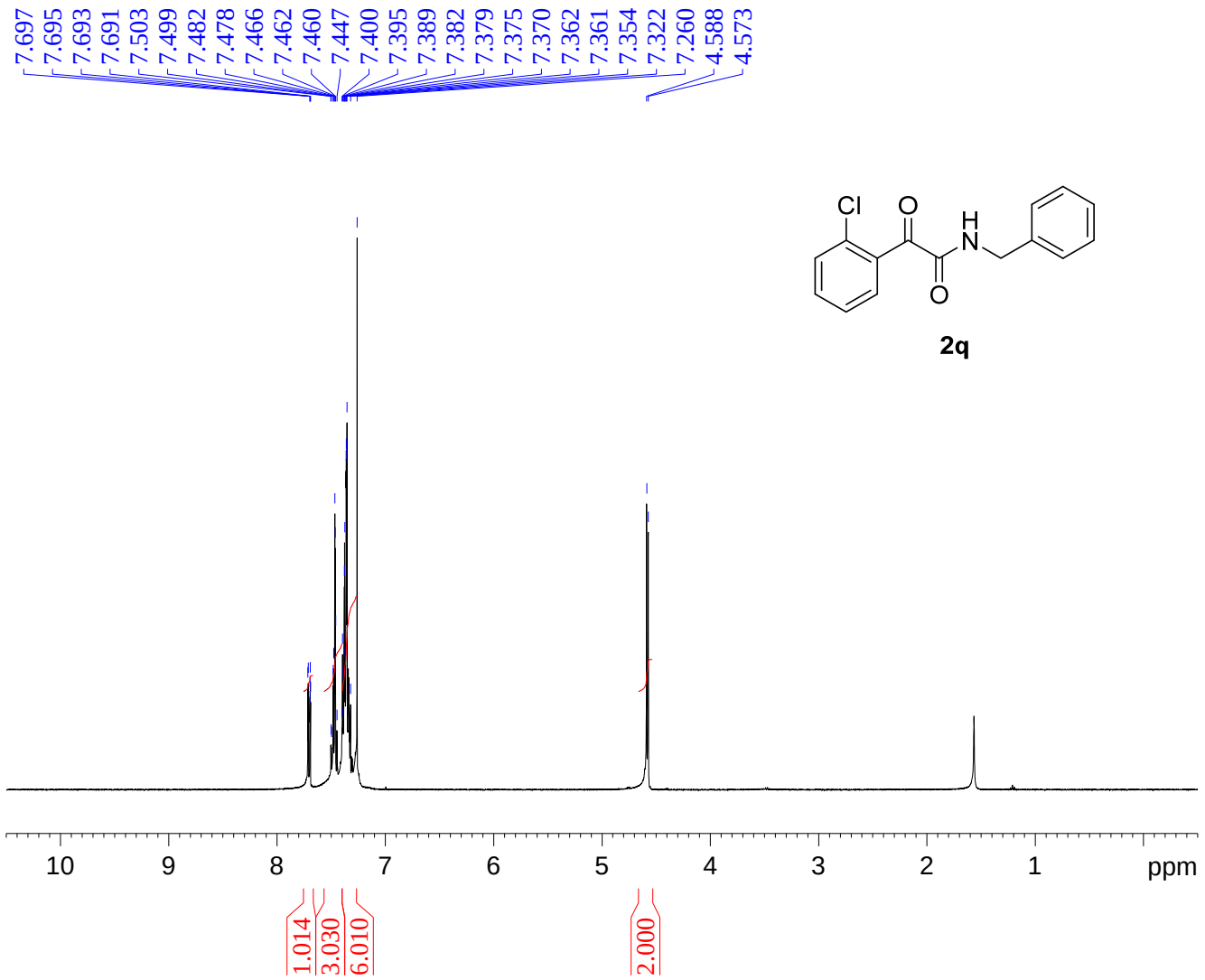


NAME 20151210
EXPNO 1
PROCNO 1
Date_ 20151210
Time 15.11
INSTRUM spect
PROBHD 5 mm DUL 13C-1
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 38
DS 0
SWH 6410.256 Hz
FIDRES 0.195625 Hz
AQ 2.5559540 sec
RG 4
DW 78.000 usec
DE 6.00 usec
TE 300.0 K
D1 2.00000000 sec
TD0 1

======== CHANNEL f1 ========
NUC1 1H
P1 10.00 usec
PL1 -2.40 dB
SFO1 400.1528010 MHz
SI 16384
SF 400.1500091 MHz
WDW EM
SSB 0
LB 0.00 Hz
GB 0
PC 1.00



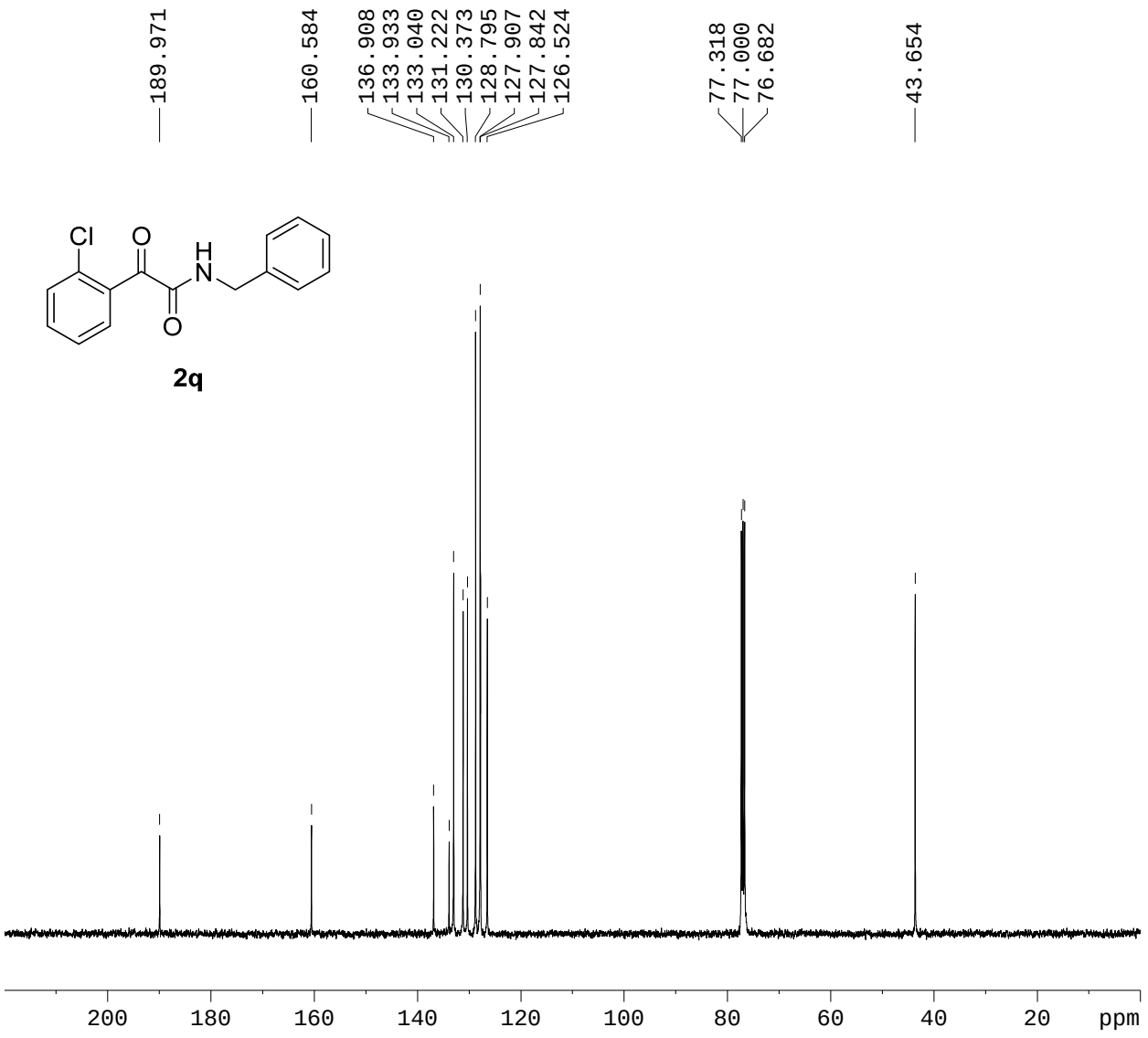
NAME	20151210
EXPNO	2
PROCNO	1
Date_	20151210
Time	21.23
INSTRUM	spect
PROBHD	5 mm DUL 13C-1
PULPROG	zgpg30
TD	65536
SOLVENT	CDCl3
NS	712
DS	0
SWH	22727.273 Hz
FIDRES	0.346791 Hz
AQ	1.4418420 sec
RG	57
DW	22.000 usec
DE	6.00 usec
TE	300.0 K
D1	2.00000000 sec
d11	0.03000000 sec
DELTA	1.89999998 sec
TD0	1
======== CHANNEL f1 ========	
NUC1	13C
P1	9.70 usec
PL1	-0.50 dB
SFO1	100.6288660 MHz
======== CHANNEL f2 ========	
CPDPRG2	waltz16
NUC2	1H
PCPD2	90.00 usec
PL2	-2.40 dB
PL12	15.10 dB
PL13	18.10 dB
SFO2	400.1516010 MHz
SI	32768
SF	100.6178015 MHz
WDW	EM
SSB	0
LB	3.00 Hz
GB	0
PC	1.00



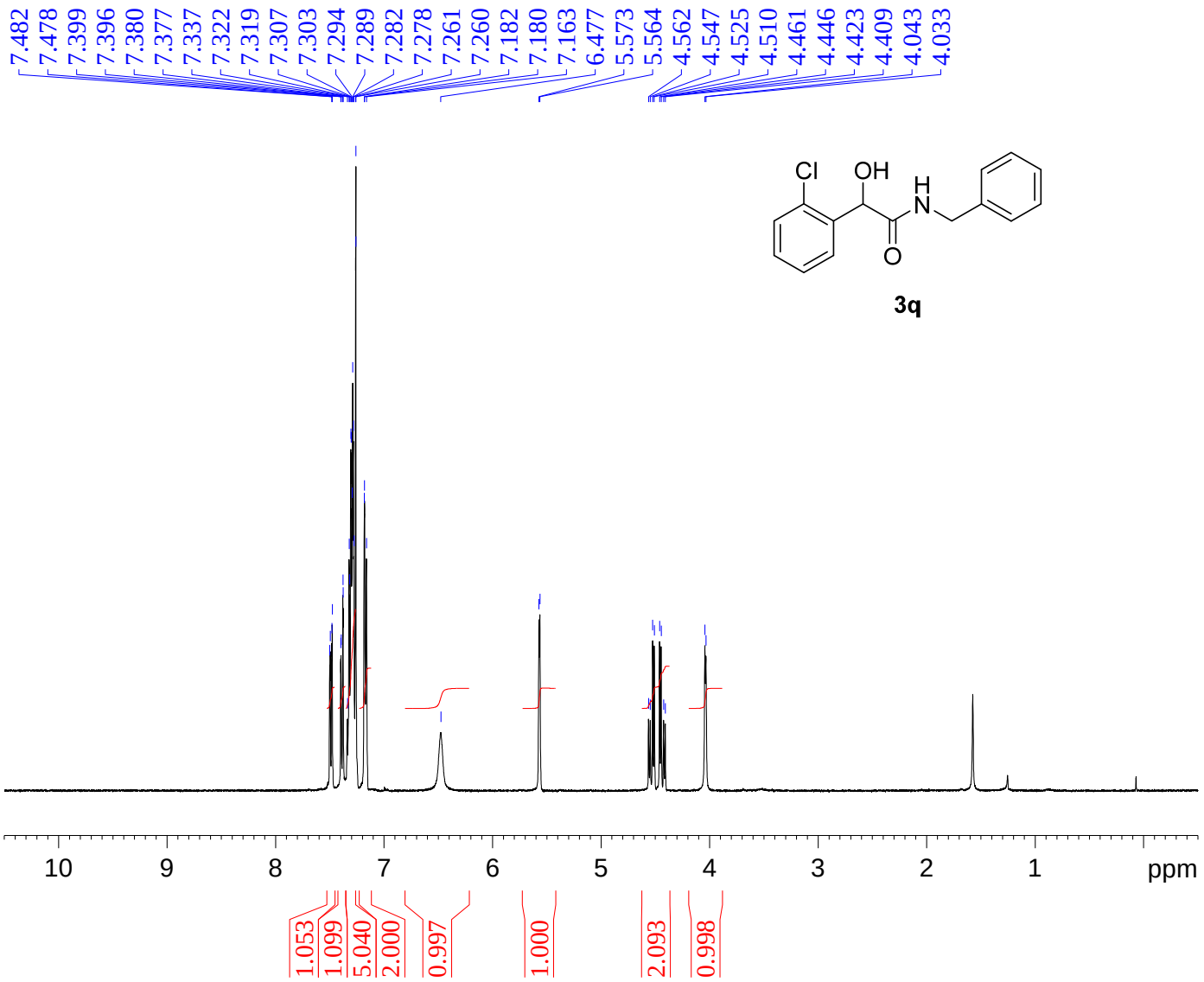
NAME	20151118
EXPNO	1
PROCNO	1
Date_	20151118
Time	19.54
INSTRUM	spect
PROBHD	5 mm DUL 13C-1
PULPROG	zg30
TD	32768
SOLVENT	CDCl3
NS	64
DS	0
SWH	6410.256 Hz
FIDRES	0.195625 Hz
AQ	2.5559540 sec
RG	4
DW	78.000 usec
DE	6.00 usec
TE	300.0 K
D1	2.00000000 sec
TD0	1

======== CHANNEL f1 ========

NUC1	1H
P1	10.00 usec
PL1	-2.40 dB
SFO1	400.1528010 MHz
SI	16384
SF	400.1500088 MHz
WDW	EM
SSB	0
LB	0.00 Hz
GB	0
PC	1.00

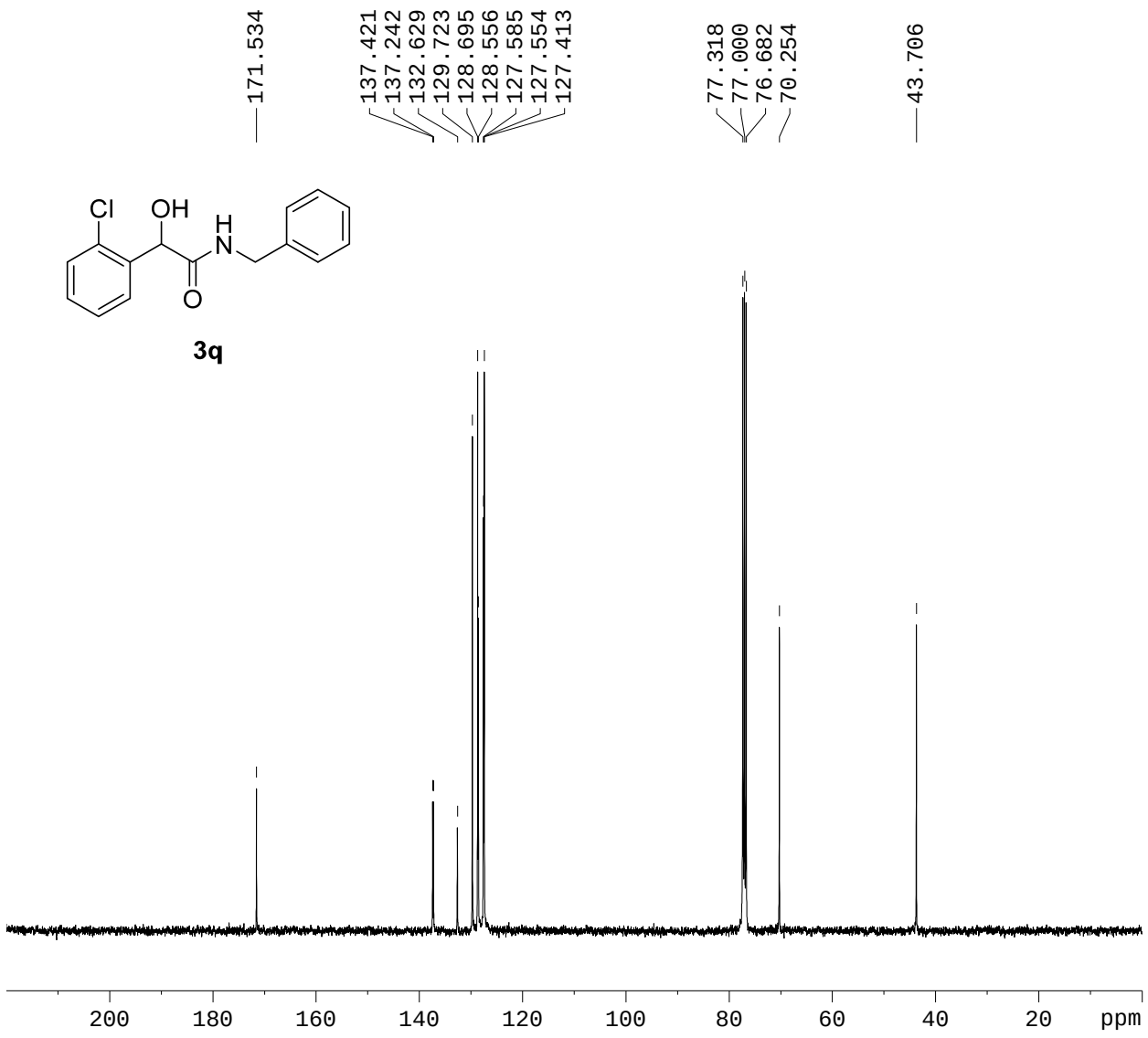


NAME	Substrate 13C	
EXPNO	20	
PROCNO	1	
Date_	20150812	
Time	20.25	
INSTRUM	spect	
PROBHD	5 mm DUL 13C-1	
PULPROG	zgpg30	
TD	65536	
SOLVENT	CDCl3	
NS	572	
DS	0	
SWH	22727.273	Hz
FIDRES	0.346791	Hz
AQ	1.4418420	sec
RG	57	
DW	22.000	usec
DE	6.00	usec
TE	300.0	K
D1	2.00000000	sec
d11	0.03000000	sec
DELTA	1.89999998	sec
TD0	1	
======== CHANNEL f1 ========		
NUC1	13C	
P1	9.70	usec
PL1	-0.50	dB
SFO1	100.6288660	MHz
======== CHANNEL f2 ========		
CPDPRG2	waltz16	
NUC2	1H	
PCPD2	90.00	usec
PL2	-2.40	dB
PL12	15.10	dB
PL13	18.10	dB
SFO2	400.1516010	MHz
SI	32768	
SF	100.6178062	MHz
WDW	EM	
SSB	0	
LB	3.00	Hz
GB	0	
PC	1.00	

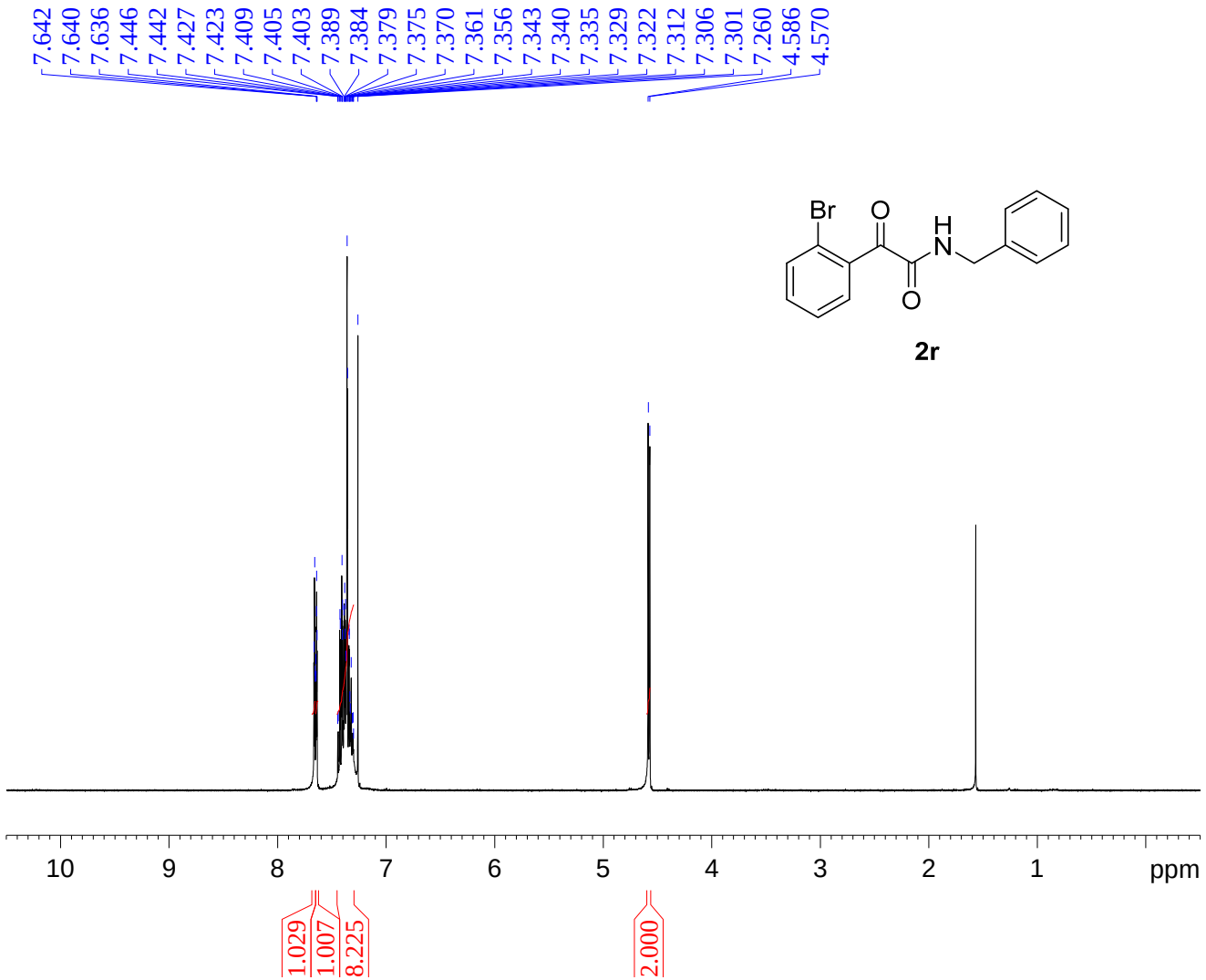


NAME 20160120
EXPNO 1
PROCNO 1
Date_ 20160120
Time 19.55
INSTRUM spect
PROBHD 5 mm DUL 13C-1
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 48
DS 0
SWH 6410.256 Hz
FIDRES 0.195625 Hz
AQ 2.5559540 sec
RG 4
DW 78.000 usec
DE 6.00 usec
TE 300.0 K
D1 2.00000000 sec
TD0 1

======== CHANNEL f1 ========
NUC1 1H
P1 10.00 usec
PL1 -2.40 dB
SFO1 400.1528010 MHz
SI 16384
SF 400.1500089 MHz
WDW EM
SSB 0
LB 0.00 Hz
GB 0
PC 1.00

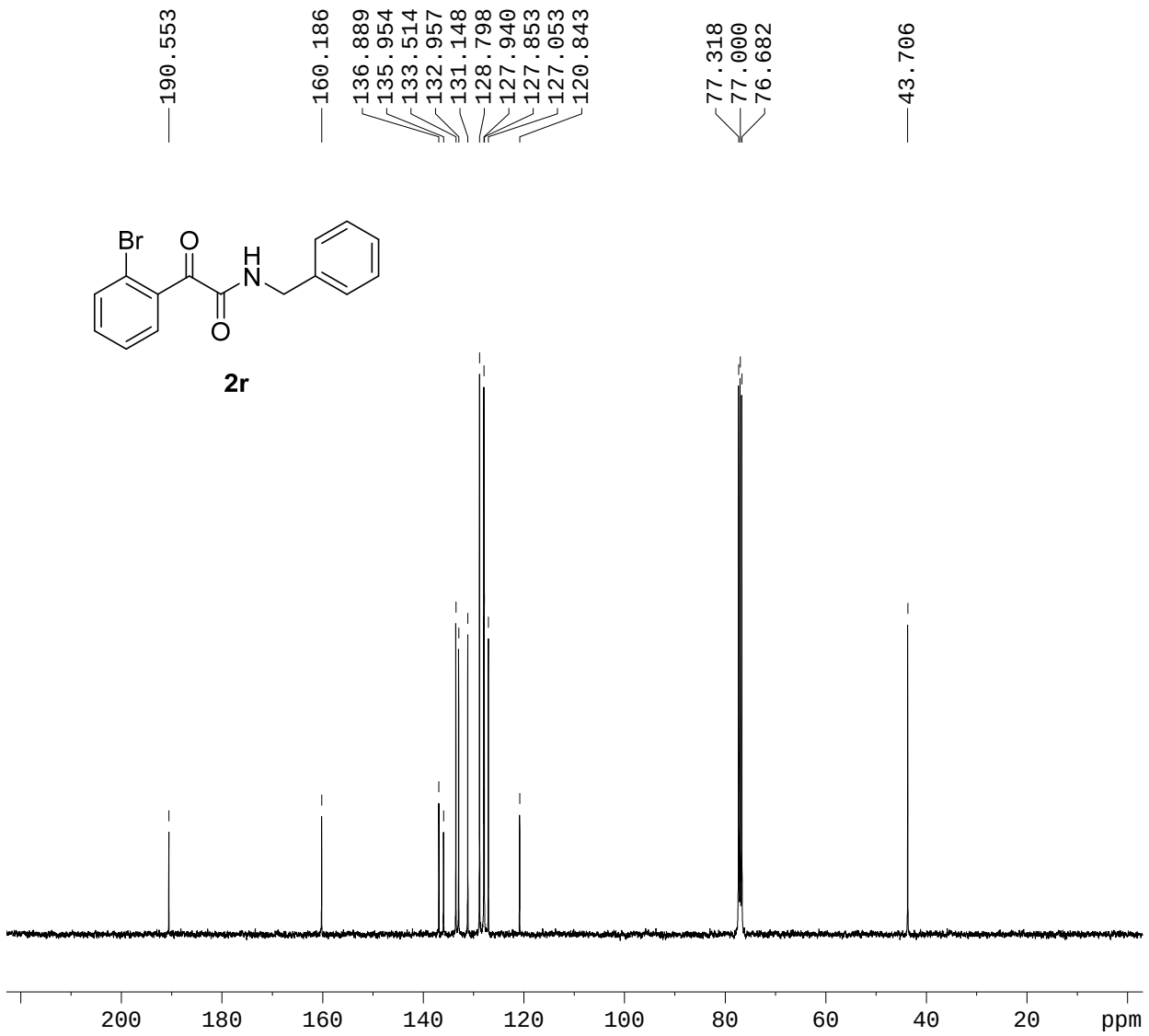



```
NAME            20160120
EXPNO                  2
PROCNO                 1
Date_           20160120
Time                20.02
INSTRUM             spect
PROBHD   5 mm DUL 13C-1
PULPROG           zgpg30
TD                 65536
SOLVENT            CDCl3
NS                   900
DS                     0
SWH            22727.273 Hz
FIDRES          0.346791 Hz
AQ             1.4418420 sec
RG                    57
DW                22.000 usec
DE                 6.00 usec
TE                300.0 K
D1           2.00000000 sec
d11          0.03000000 sec
DELTA        1.89999998 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                  13C
P1                   9.70 usec
PL1                 -0.50 dB
SFO1         100.6288660 MHz

======== CHANNEL f2 ========
CPDPRG2           waltz16
NUC2                  1H
PCPD2               90.00 usec
PL2                 -2.40 dB
PL12                15.10 dB
PL13                18.10 dB
SFO2         400.1516010 MHz
SI                 32768
SF           100.6178045 MHz
WDW                   EM
SSB                    0
LB                  3.00 Hz
GB                     0
PC                  1.00
```

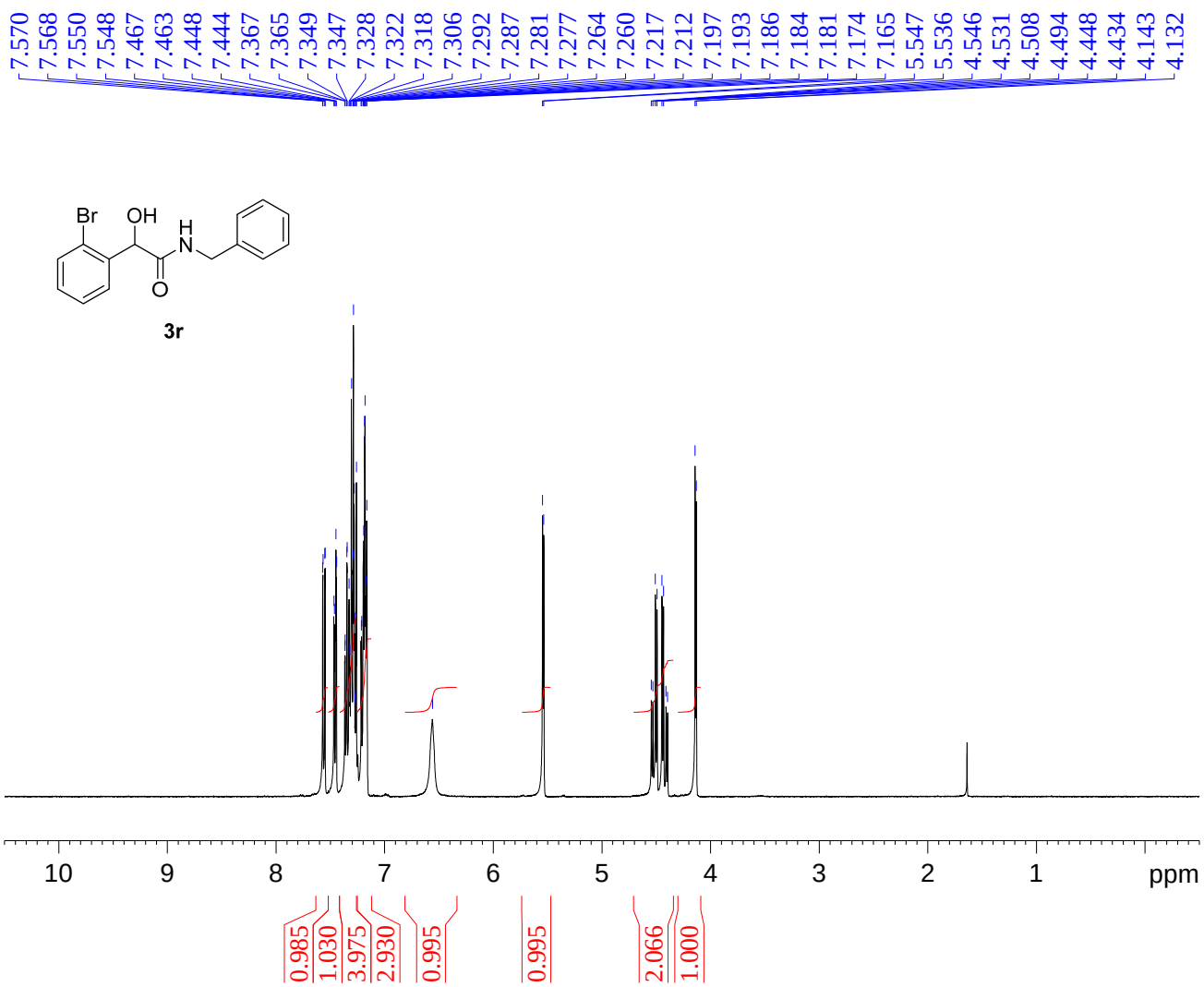


NAME 20151111
EXPNO 1
PROCNO 1
Date_ 20151111
Time 20.12
INSTRUM spect
PROBHD 5 mm DUL 13C-1
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 16
DS 0
SWH 6410.256 Hz
FIDRES 0.195625 Hz
AQ 2.5559540 sec
RG 4
DW 78.000 usec
DE 6.00 usec
TE 300.0 K
D1 2.00000000 sec
TD0 1

======== CHANNEL f1 ========
NUC1 1H
P1 10.00 usec
PL1 -2.40 dB
SFO1 400.1528010 MHz
SI 16384
SF 400.1500089 MHz
WDW EM
SSB 0
LB 0.00 Hz
GB 0
PC 1.00



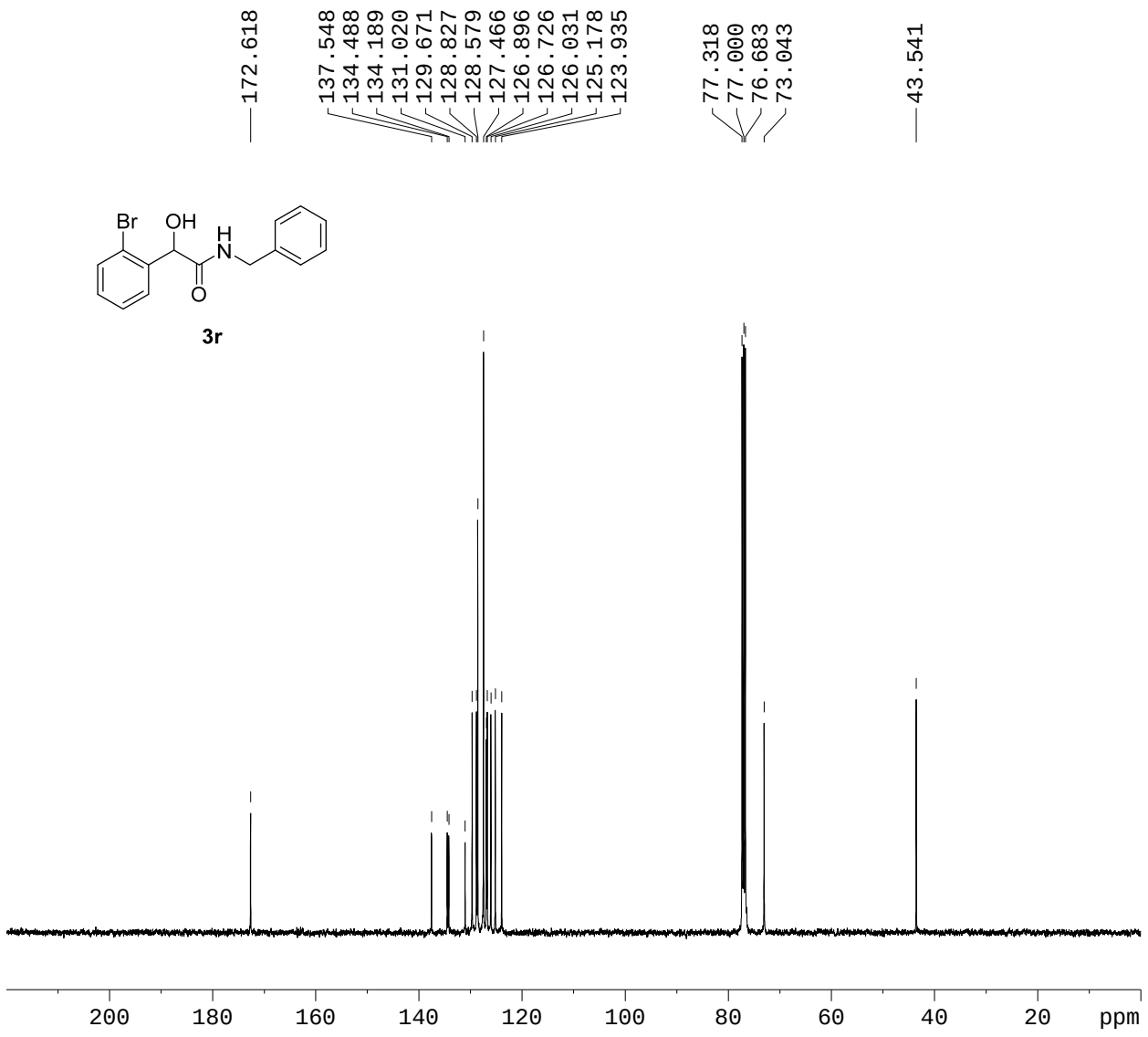
NAME	20151111
EXPNO	2
PROCNO	1
Date_	20151111
Time	20.17
INSTRUM	spect
PROBHD	5 mm DUL 13C-1
PULPROG	zgpg30
TD	65536
SOLVENT	CDCl3
NS	716
DS	0
SWH	22727.273 Hz
FIDRES	0.346791 Hz
AQ	1.4418420 sec
RG	57
DW	22.000 usec
DE	6.00 usec
TE	300.0 K
D1	2.00000000 sec
d11	0.03000000 sec
DELTA	1.89999998 sec
TD0	1
======== CHANNEL f1 ========	
NUC1	13C
P1	9.70 usec
PL1	-0.50 dB
SFO1	100.6288660 MHz
======== CHANNEL f2 ========	
CPDPRG2	waltz16
NUC2	1H
PCPD2	90.00 usec
PL2	-2.40 dB
PL12	15.10 dB
PL13	18.10 dB
SFO2	400.1516010 MHz
SI	32768
SF	100.6178060 MHz
WDW	EM
SSB	0
LB	3.00 Hz
GB	0
PC	1.00



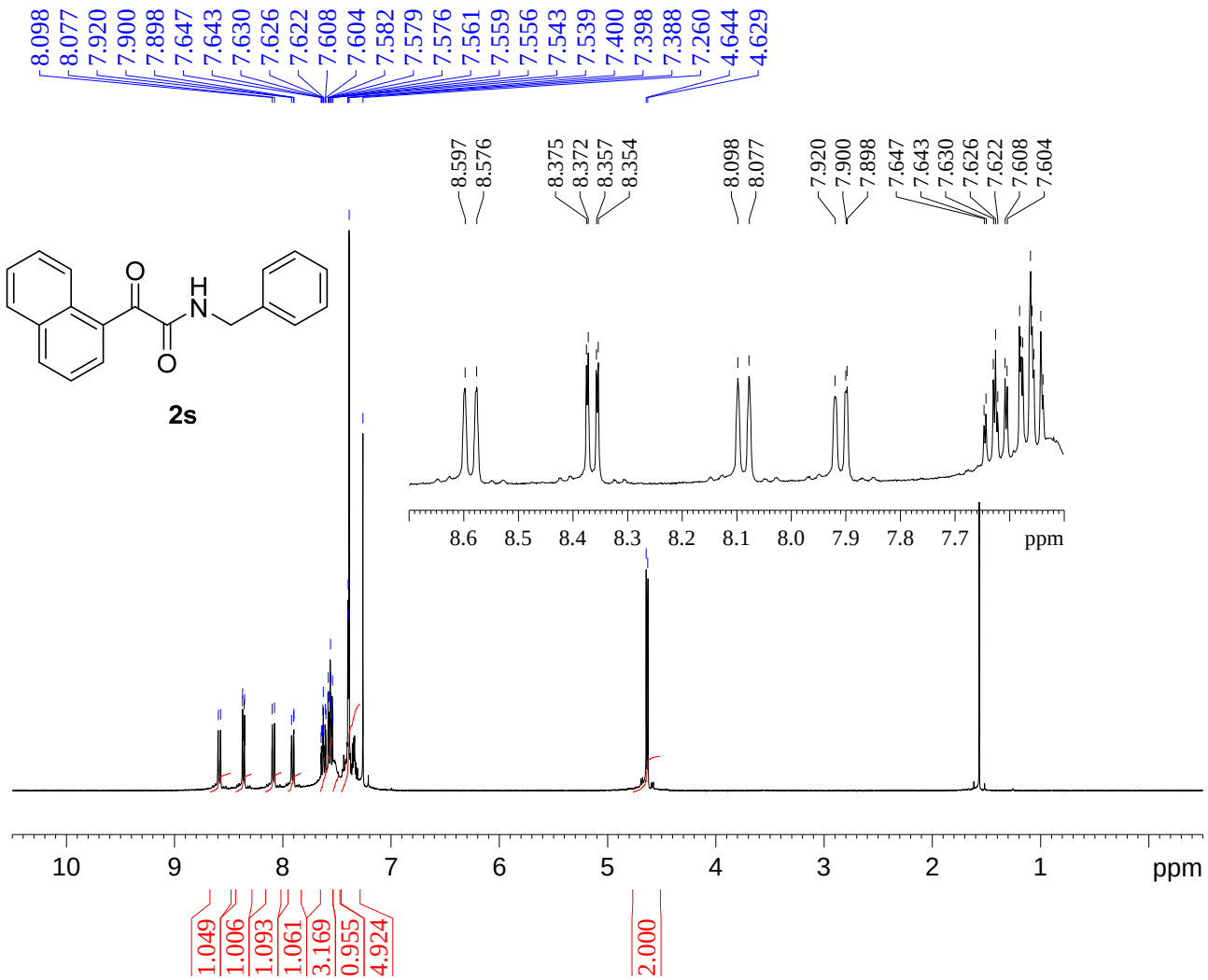
NAME Substrate
EXPNO 5
PROCNO 1
Date_ 20150426
Time 17.39
INSTRUM spect
PROBHD 5 mm DUL 13C-1
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 44
DS 0
SWH 6410.256 Hz
FIDRES 0.195625 Hz
AQ 2.5559540 sec
RG 4
DW 78.000 usec
DE 6.00 usec
TE 300.0 K
D1 2.00000000 sec
TD0 1

======== CHANNEL f1 ========
NUC1 1H
P1 10.00 usec
PL1 -2.40 dB
SFO1 400.1528010 MHz
SI 16384
SF 400.1500092 MHz
WDW EM
SSB 0
LB 0.00 Hz
GB 0
PC 1.00

.

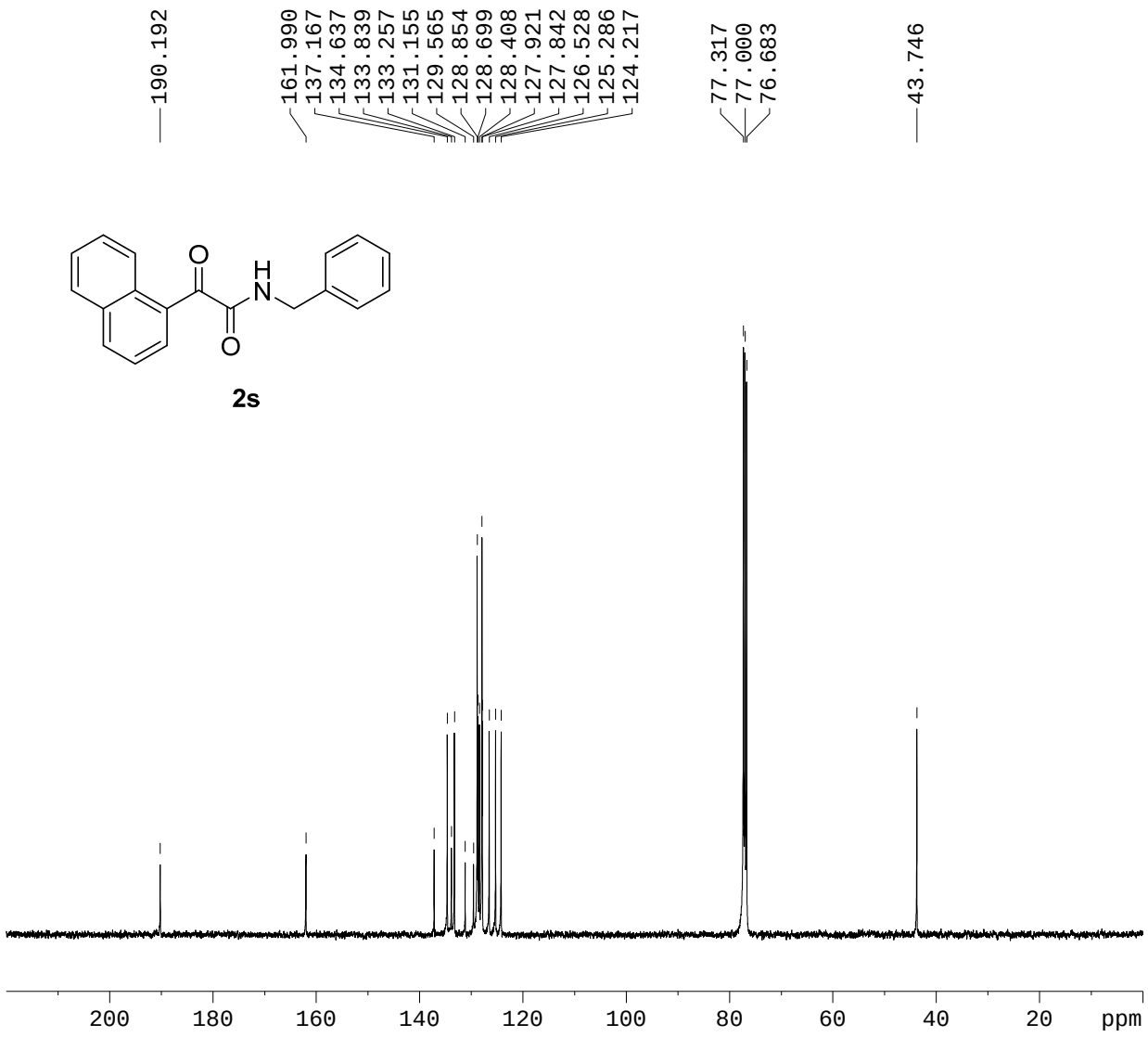


NAME	Substrate 13C	
EXPNO	7	
PROCNO	1	
Date_	20150712	
Time	15.35	
INSTRUM	spect	
PROBHD	5 mm DUL 13C-1	
PULPROG	zgpg30	
TD	65536	
SOLVENT	CDCl3	
NS	1029	
DS	0	
SWH	22727.273	Hz
FIDRES	0.346791	Hz
AQ	1.4418420	sec
RG	57	
DW	22.000	usec
DE	6.00	usec
TE	300.0	K
D1	2.00000000	sec
d11	0.03000000	sec
DELTA	1.89999998	sec
TD0	1	
======== CHANNEL f1 ========		
NUC1	13C	
P1	9.70	usec
PL1	-0.50	dB
SFO1	100.6288660	MHz
======== CHANNEL f2 ========		
CPDPRG2	waltz16	
NUC2	1H	
PCPD2	90.00	usec
PL2	-2.40	dB
PL12	15.10	dB
PL13	18.10	dB
SFO2	400.1516010	MHz
SI	32768	
SF	100.6178041	MHz
WDW	EM	
SSB	0	
LB	3.00	Hz
GB	0	
PC	1.00	

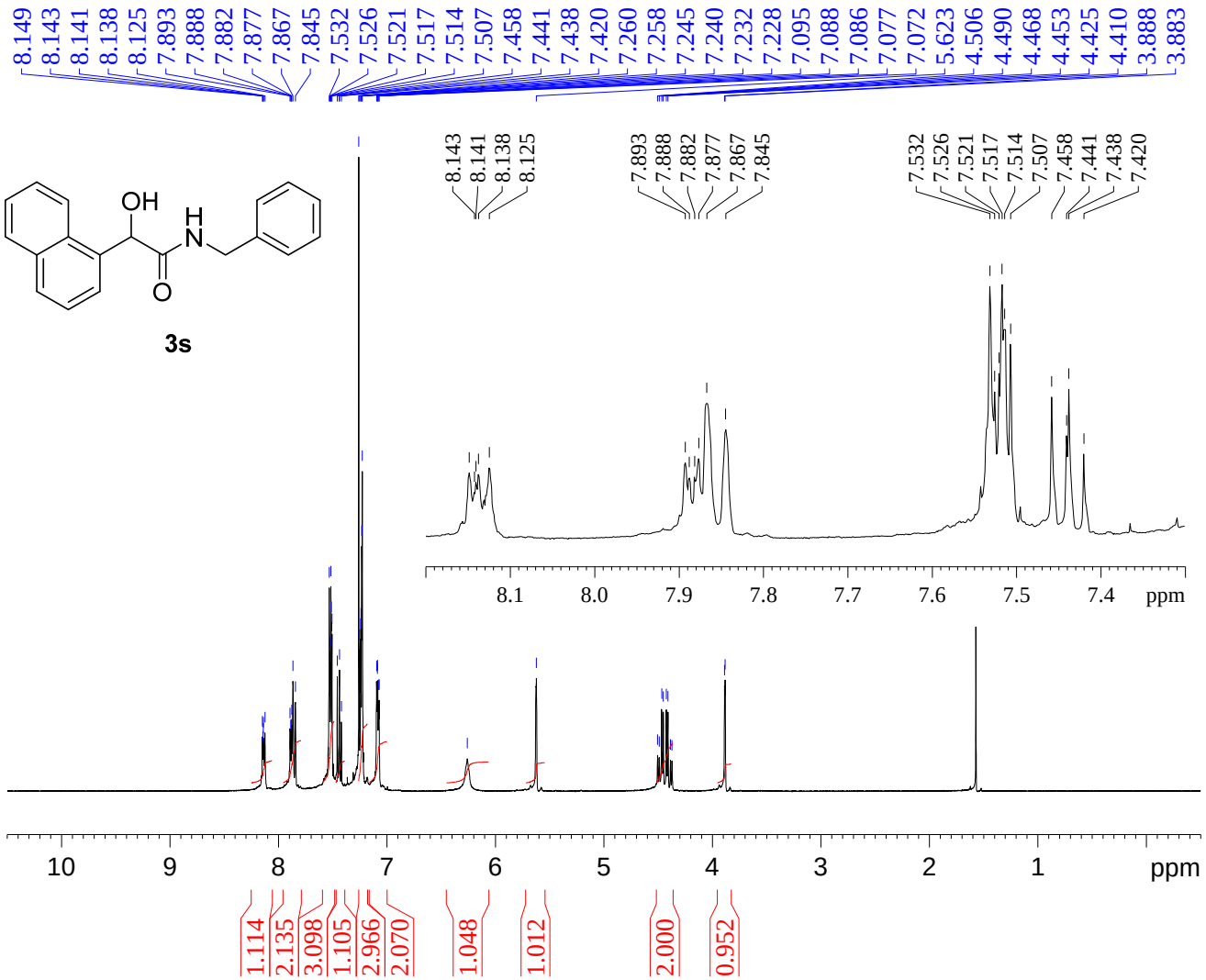


NAME Substrate 1H
EXPNO 6
PROCNO 1
Date_ 20150712
Time 15.19
INSTRUM spect
PROBHD 5 mm DUL 13C-1
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 52
DS 0
SWH 6410.256 Hz
FIDRES 0.195625 Hz
AQ 2.5559540 sec
RG 4
DW 78.000 usec
DE 6.00 usec
TE 300.0 K
D1 2.00000000 sec
TD0 1

======== CHANNEL f1 ========
NUC1 1H
P1 10.00 usec
PL1 -2.40 dB
SFO1 400.1528010 MHz
SI 16384
SF 400.1500088 MHz
WDW EM
SSB 0
LB 0.00 Hz
GB 0
PC 1.00

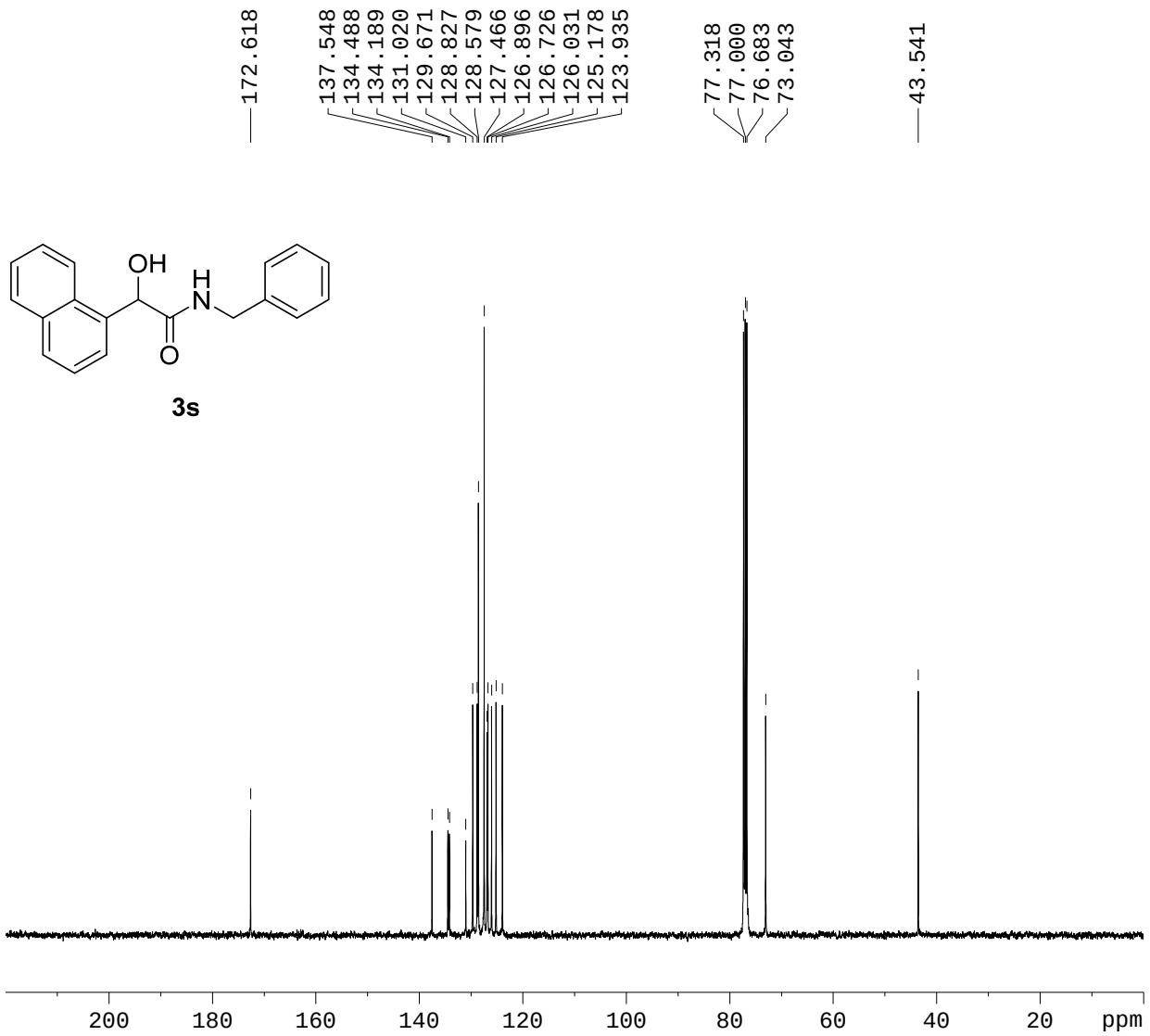


NAME	Substrate 13C
EXPNO	6
PROCNO	1
Date_	20150711
Time	17.40
INSTRUM	spect
PROBHD	5 mm DUL 13C-1
PULPROG	zgpg30
TD	65536
SOLVENT	CDCl3
NS	1739
DS	0
SWH	22727.273 Hz
FIDRES	0.346791 Hz
AQ	1.4418420 sec
RG	57
DW	22.000 usec
DE	6.00 usec
TE	300.0 K
D1	2.00000000 sec
d11	0.03000000 sec
DELTA	1.89999998 sec
TD0	1
======== CHANNEL f1 ========	
NUC1	13C
P1	9.70 usec
PL1	-0.50 dB
SFO1	100.6288660 MHz
======== CHANNEL f2 ========	
CPDPRG2	waltz16
NUC2	1H
PCPD2	90.00 usec
PL2	-2.40 dB
PL12	15.10 dB
PL13	18.10 dB
SFO2	400.1516010 MHz
SI	32768
SF	100.6178034 MHz
WDW	EM
SSB	0
LB	3.00 Hz
GB	0
PC	1.00



NAME Substrate 1H
EXPNO 7
PROCNO 1
Date_ 20150712
Time 15.27
INSTRUM spect
PROBHD 5 mm DUL 13C-1
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 65
DS 0
SWH 6410.256 Hz
FIDRES 0.195625 Hz
AQ 2.5559540 sec
RG 4
DW 78.000 usec
DE 6.00 usec
TE 300.0 K
D1 2.00000000 sec
TD0 1

======== CHANNEL f1 ========
NUC1 1H
P1 10.00 usec
PL1 -2.40 dB
SFO1 400.1528010 MHz
SI 16384
SF 400.1500092 MHz
WDW EM
SSB 0
LB 0.00 Hz
GB 0
PC 1.00



NAME	Substrate 13C
EXPNO	7
PROCNO	1
Date_	20150712
Time	15.35
INSTRUM	spect
PROBHD	5 mm DUL 13C-1
PULPROG	zgpg30
TD	65536
SOLVENT	CDCl3
NS	1029
DS	0
SWH	22727.273 Hz
FIDRES	0.346791 Hz
AQ	1.4418420 sec
RG	57
DW	22.000 usec
DE	6.00 usec
TE	300.0 K
D1	2.00000000 sec
d11	0.03000000 sec
DELTA	1.89999998 sec
TD0	1
======== CHANNEL f1 ========	
NUC1	13C
P1	9.70 usec
PL1	-0.50 dB
SFO1	100.6288660 MHz
======== CHANNEL f2 ========	
CPDPRG2	waltz16
NUC2	1H
PCPD2	90.00 usec
PL2	-2.40 dB
PL12	15.10 dB
PL13	18.10 dB
SFO2	400.1516010 MHz
SI	32768
SF	100.6178041 MHz
WDW	EM
SSB	0
LB	3.00 Hz
GB	0
PC	1.00