

Supporting Information

Retention or Inversion in Stereospecific Nickel-Catalyzed Cross-Coupling of Benzylic Carbamates with Arylboronic Esters: Control of Absolute Stereochemistry with an Achiral Catalyst

Michael R. Harris,[†] Luke E. Hanna,[†] Margaret A. Greene,[†] Curtis E. Moore,[‡] Elizabeth R. Jarvo[†]

[†]*Department of Chemistry, 1102 Natural Sciences II, University of California, Irvine, CA 92697*

[‡]*Department of Chemistry and Biochemistry, University of California, San Diego, 9500 Gilman Drive, MC 0358, La Jolla, California 92093-0358*

I.	General procedures	S1
II.	Catalyst controlled retention or inversion	S3
	A. Demonstration of stereochemical course of cross-coupling reaction	S3
	B. Mechanistic model	S8
III.	Synthesis and characterization of substrates	S9
	A. Racemic diarylmethyl alcohols	S9
	B. Enantioenriched diarylmethyl alcohols	S9
	C. Suzuki cross-coupling of enantioenriched alcohols	S11
	D. Preparation of protected carbinols	S12
IV.	General procedure for cross-coupling reactions	S15
	A. With retention of configuration using PCy ₃ ligand	S15
	B. With inversion of configuration using SIMes ligand	S16
V.	Characterization data for products	S16
VI.	Tables of results using alternative ligands and bases	S29
VII.	References and notes for supporting information	S30
VIII.	Crystallographic Data	S31
IX.	¹ H and ¹³ C NMR spectra	S44
X.	SFC Traces	S100

I. General Procedures

All reactions were carried out under an atmosphere of N₂, or Ar when noted. All glassware was oven- or flame-dried prior to use. Tetrahydrofuran (THF), diethyl ether (Et₂O), dichloromethane (CH₂Cl₂), and toluene (PhMe) were degassed with Ar and then passed through two 4 x 36 inch columns of anhydrous neutral A-2 alumina (8 x 14 mesh; LaRoche Chemicals; activated under a flow of argon at 350 °C for 12 h) to remove H₂O. All other solvents utilized were purchased “anhydrous” commercially, or purified as described. ¹H NMR spectra were recorded on Bruker DRX-400 (400 MHz ¹H, 100 MHz ¹³C, 376.5 MHz ¹⁹F), GN-500 (500 MHz ¹H, 125.7 MHz ¹³C), or CRYO-500 (500 MHz ¹H, 125.7 MHz ¹³C) spectrometers. Proton chemical shifts are reported in ppm (δ) relative to internal tetramethylsilane (TMS, δ 0.00). Data are reported as follows: chemical shift (multiplicity [singlet (s), broad singlet (br s), doublet (d), doublet of doublets (dd), triplet (t), doublet of triplets (dt), doublet of doublet of triplets (ddt), triplet of

triplets (tt), quartet (q), multiplet (m)], coupling constants [Hz], integration). Carbon chemical shifts are reported in ppm (δ) relative to TMS with the respective solvent resonance as the internal standard (CDCl_3 , δ 77.16 ppm). Unless otherwise indicated, NMR data were collected at 25 °C. Infrared (IR) spectra were obtained on a Perkin-Elmer Spectrum 1000 FT-IR Systems and are reported in terms of frequency of absorption (cm^{-1}). Analytical thin-layer chromatography (TLC) was performed using Silica Gel 60 F₂₅₄ precoated plates (0.25 mm thickness). Visualization was accomplished by irradiation with a UV lamp and/or staining with KMnO_4 , ceric ammonium molybdate (CAM), or *p*-anisaldehyde (PAA) solutions. Flash chromatography was performed using Silica Gel 60 (170-400 mesh) from Fisher Scientific. Melting points (m.p.) were obtained using a Mel-Temp melting point apparatus and are uncorrected. Optical rotations were measured on a Rudolph Research Analytical Autopol IV Automatic Polarimeter. SFC determinations of enantiopurity were performed on a Berger Analytical instrument using a Daicel™ Chiralpak® column (OD-H, OJ-H, or AD-H; 100 bar, 50 °C, 215 nm). High resolution mass spectrometry was performed by the University of California, Irvine Mass Spectrometry Center.

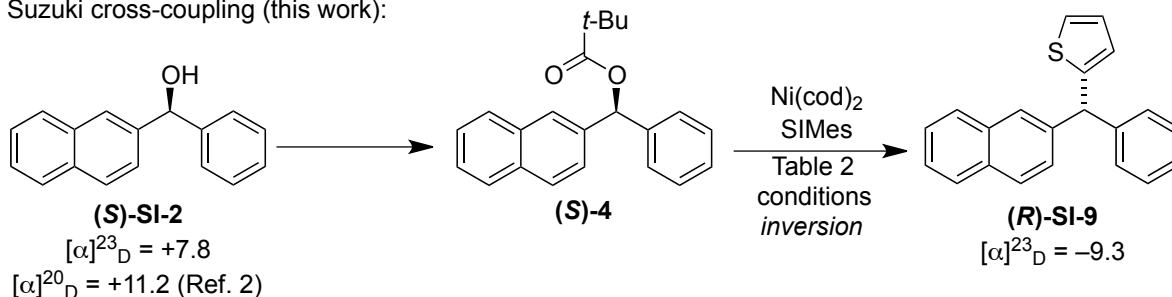
Boronic esters were prepared from the corresponding boronic acids and 2,2-dimethylpropane-1,3-diol.¹ Boronic acids were generously donated from Frontier, stored at 4 °C, and used as received. 1,8-bis(1,5-cyclooctadiene)nickel was purchased from Strem, stored in a glovebox freezer (-20 °C) under an atmosphere of N_2 , and used as received. Tricyclohexylphosphine (PCy_3), (1,3-Bis(2,6-diisopropylphenyl)-4,5-dihydroimidazoliumtetrafluoroborate (SIMes), and tris(di-benzylideneacetone)dipalladium ($\text{Pd}_2(\text{dba})_3$) were purchased from Strem, stored in a glovebox, and used as received. All other reagents were purchased commercially and used as received.

II. Catalyst controlled retention or inversion

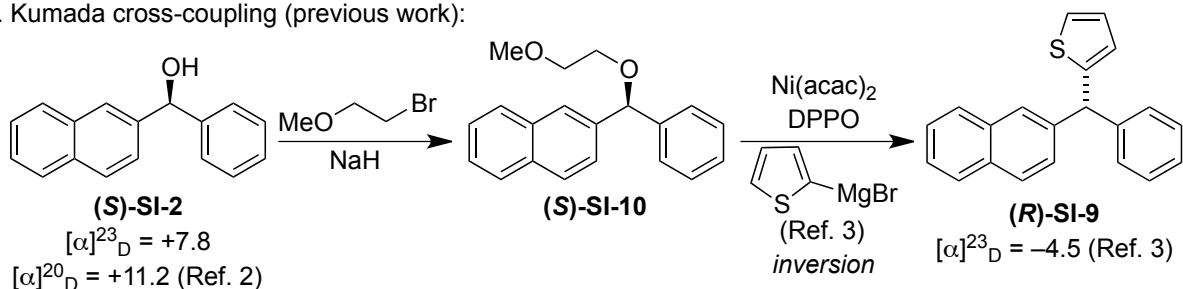
A. Demonstration of stereochemical course of cross-coupling reaction

Scheme S11. Optical rotation compared to literature data from crystal structure.

a. Suzuki cross-coupling (this work):

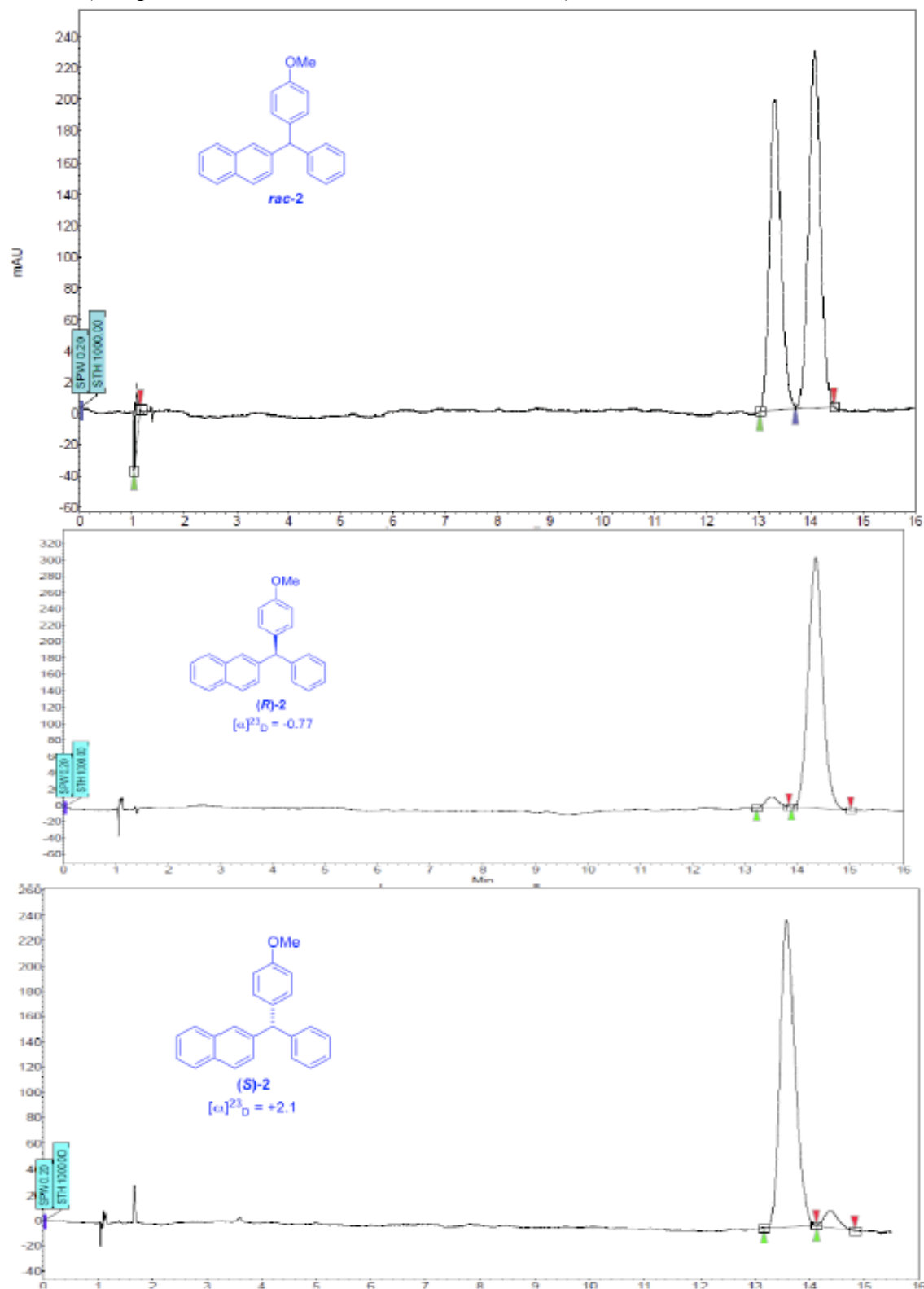


b. Kumada cross-coupling (previous work):

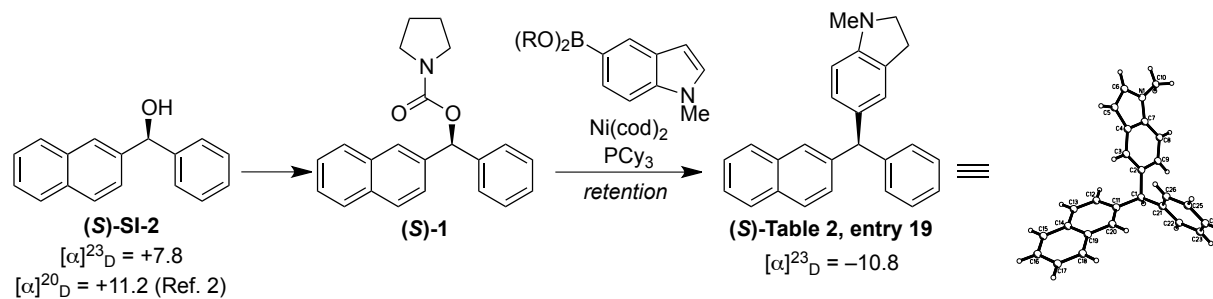


Enantioenriched alcohol **(S)-SI-2** was prepared by asymmetric arylation using catalytic methyl (*S*)-(1-tritylaziridin-2-yl)diphenylmethanol (vide infra) and was assigned as the (*S*)-enantiomer by comparison of the optical rotation to the literature value.² Conversion to compound **(S)-4**, followed by stereospecific cross-coupling produced **(R)-SI-9**, which was assigned as the (*R*)-enantiomer by comparison of the optical rotation to the literature value.³ This product corresponds to net inversion at the benzylic carbon during the cross-coupling reaction. In our previous work, we demonstrated that cross-coupling of Grignard reagents also results in inversion at the benzylic carbon³ and assigned the absolute configuration of **(R)-SI-9** based on X-ray crystallographic analysis. The SFC traces of the products formed from the two different methods are shown below.

SFC traces (complete SFC data can be found in section X):



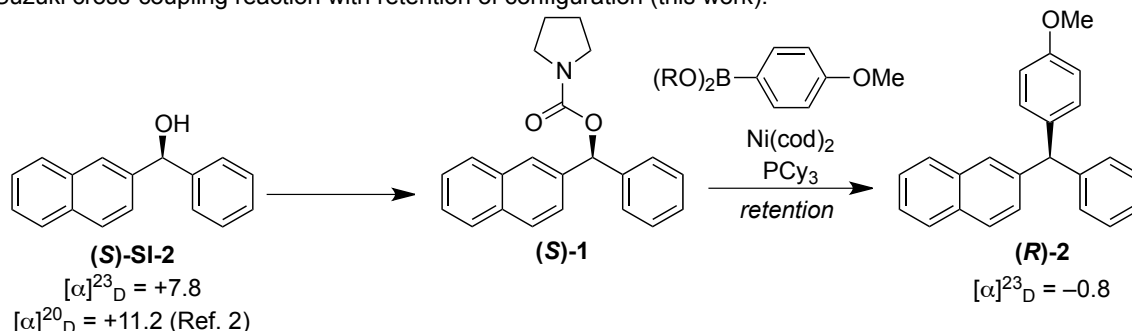
Scheme SI2. Synthesis of (*S*)-**Table 2, entry 19** as confirmed by X-ray crystallography.



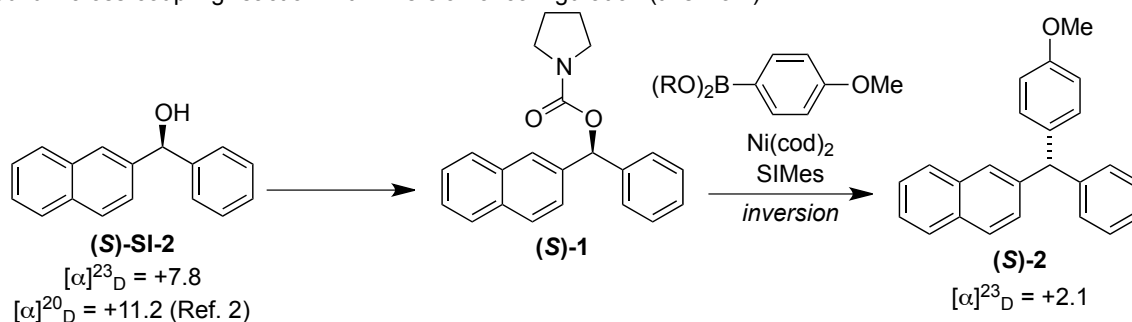
Enantioenriched alcohol (*S*)-**SI-2** was prepared by asymmetric arylation using catalytic methyl (*S*)-(1-tritylaziridin-2-yl)diphenylmethanol (vide infra) and was assigned as the (*S*)-enantiomer by comparison of the optical rotation to the literature value.² Conversion to compound (*S*)-**1**, followed by Suzuki cross-coupling using a nickel catalyst and phosphine ligand (Scheme SI2), produced product (*S*)-**Table 2, entry 19**, which was assigned as the (*S*)-enantiomer by X-ray crystallography. See section VIII for crystallographic data.

Scheme SI3. Selective synthesis of each enantiomer of product using different catalysts.

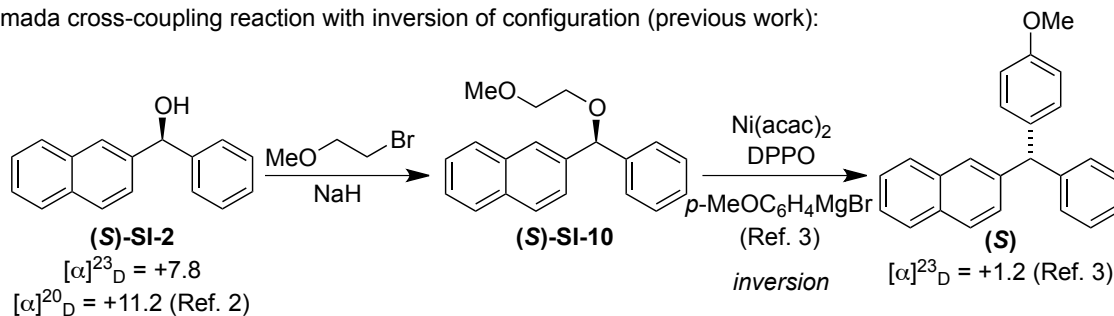
a. Suzuki cross-coupling reaction with retention of configuration (this work):



b. Suzuki cross-coupling reaction with inversion of configuration (this work):

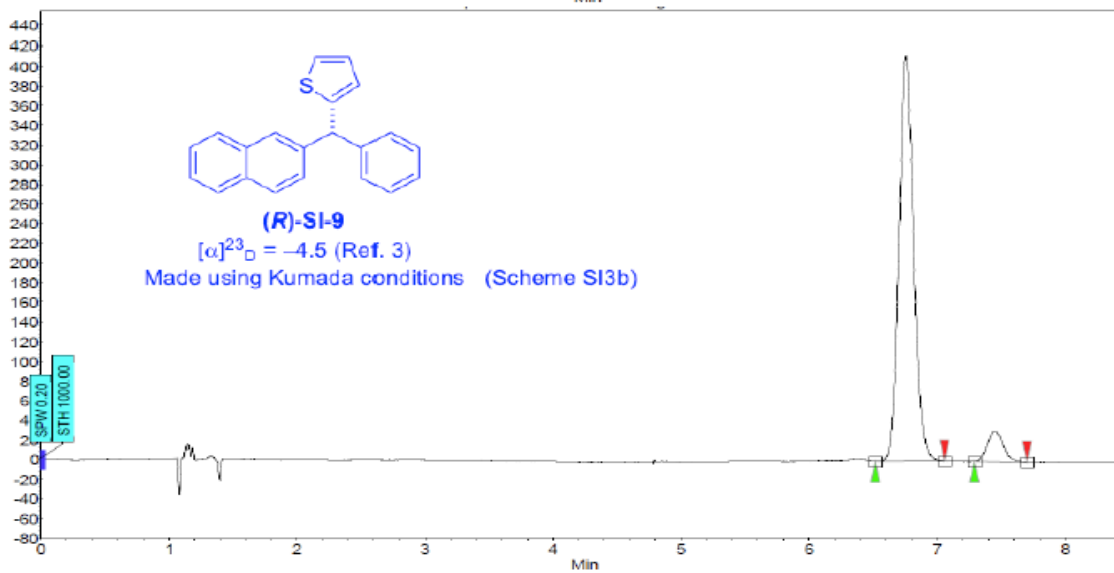
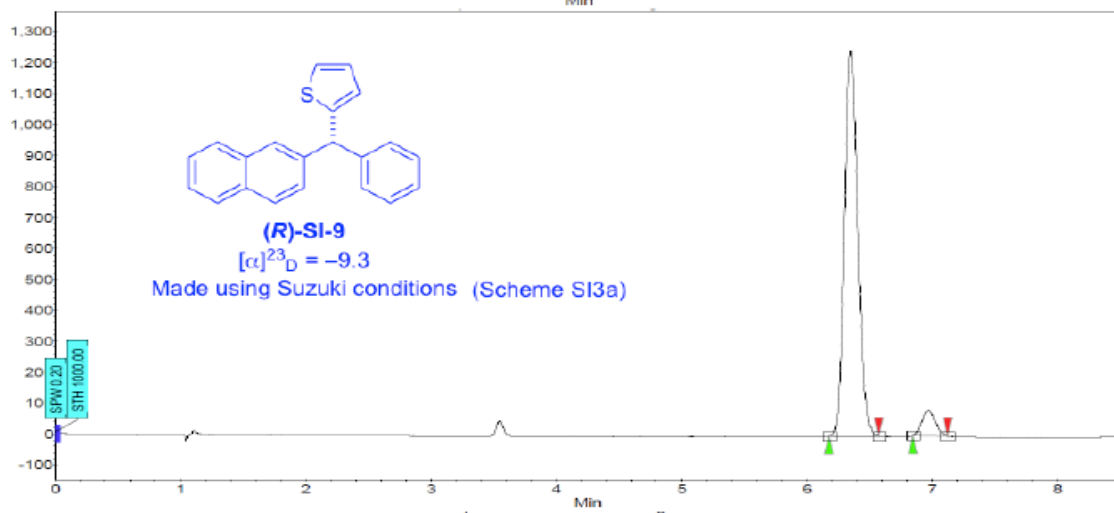
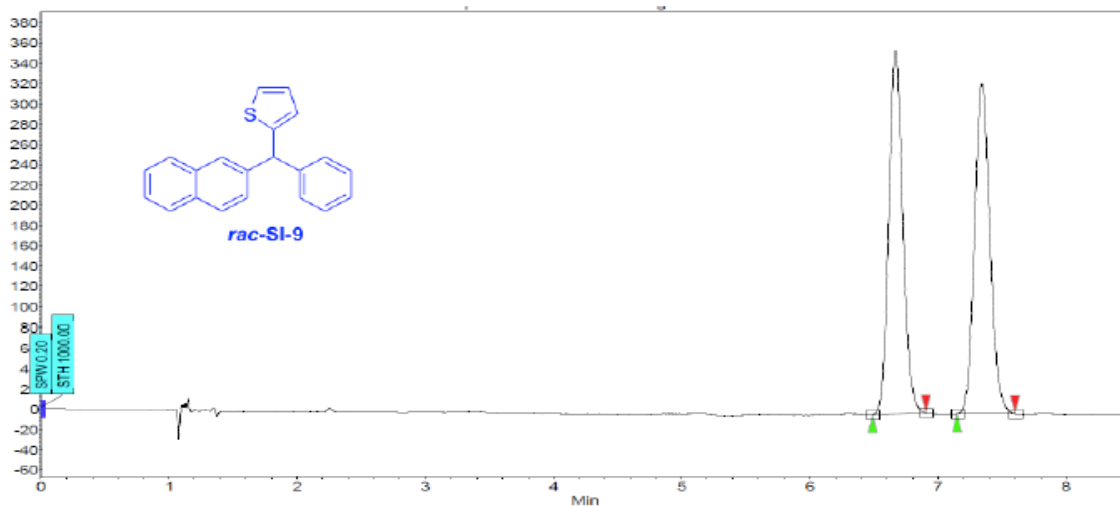


c. Kumada cross-coupling reaction with inversion of configuration (previous work):



Enantioenriched alcohol (**S**)-**SI-2** was prepared by asymmetric arylation using catalytic (*S*)-(1-tritylaziridin-2-yl)diphenylmethanol (vide infra) and was assigned as the (*S*)-enantiomer by comparison of the optical rotation to the literature value.² Conversion to compound (**S**)-**1**, followed by Suzuki cross-coupling using a nickel catalyst and phosphine ligand (Scheme SI3a), produced product (**R**)-**2**, which was assigned as the (*R*)-enantiomer by comparison of the optical rotation to the literature value.³ This product corresponds to retention at the benzylic carbon during the cross-coupling reaction. Suzuki cross-coupling of (**S**)-**1** using a nickel catalyst and NHC ligand (Scheme SI3b), produced product (**S**)-**2**, which was assigned as the (*S*)-enantiomer by comparison of the optical rotation to the literature value.³ This product corresponds to net inversion at the benzylic carbon during the cross-coupling reaction. In our previous work, we demonstrated that cross-coupling of Grignard reagents also results in inversion at the benzylic carbon.³ Comparison of these data shows that stereospecific Suzuki cross-coupling reactions can selectively form products with retention or inversion of configuration, depending on the nature of the ligand. The SFC traces of the products formed from the two different methods are shown below.

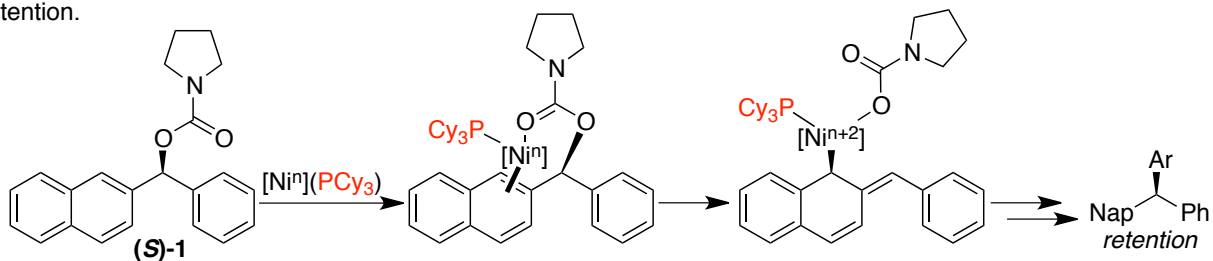
SFC traces (complete SFC data can be found in section X):



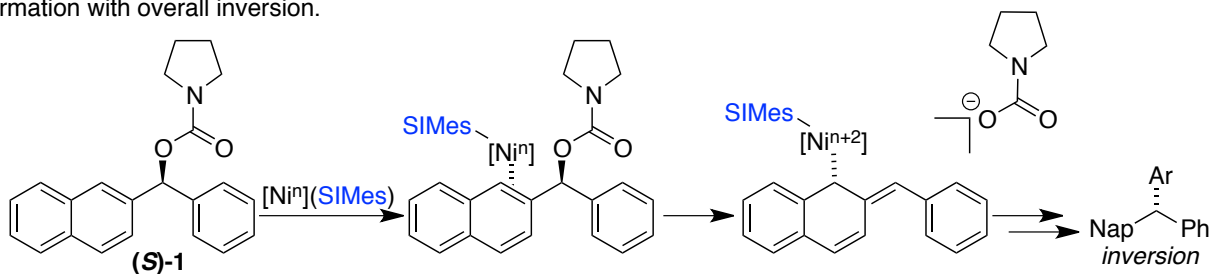
B. Mechanistic model

Scheme S14. Proposed mechanistic model for stereodivergent pathways when employing PCy₃ and SIMes ligand.

a) With PCy₃ ligand: carbamate ligation directs syn oxidative addition, resulting in product formation with overall retention.

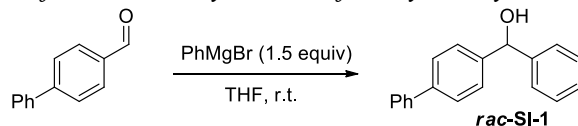


b) With SIMes ligand: oxidative addition occurs with inversion, as is more commonly observed, resulting in product formation with overall inversion.

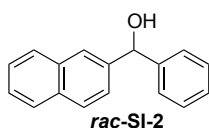


III. Synthesis and characterization of substrates

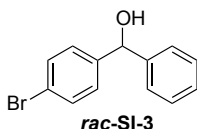
A. Representative procedure for racemic synthesis of diarylmethyl alcohols.



Rac-SI-1. In a flame-dried round-bottom flask, to a solution of biphenyl-4-carboxaldehyde (1.04 g, 5.68 mmol, 1.00 equiv) in THF (10 mL) was added phenylmagnesium bromide (0.71 M in THF, 12 mL, 8.5 mmol, 1.5 equiv). After stirring at room temperature for 4 h, saturated ammonium chloride (10 mL) was added and the reaction was extracted with EtOAc (3 x 5 mL). The combined organic layers were washed with brine (3 x 5 mL), dried over MgSO₄, and concentrated in vacuo to afford **rac-SI-1** as a white solid (1.2 g, 4.7 mmol, 55%). Analytical data is consistent with the values listed for (**S**)-**SI-1** (vide infra).

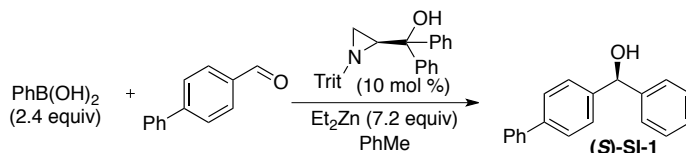


Rac-SI-2. Using the representative procedure A outlined above, the following amounts of reagents were used: 2-naphthaldehyde (6.24 g, 40.0 mmol, 1.00 equiv), phenylmagnesium bromide (58 mL, 0.83 M in THF, 48 mmol, 1.2 equiv), and THF (25 mL). The reaction mixture was purified by silica gel flash column chromatography (5–20% EtOAc/hexanes) to afford the product as a white solid (6.74 g, 28.7 mmol, 72%). Analytical data is consistent with the values listed for (**S**)-**SI-2** (vide infra).



Rac-SI-3. Using the representative procedure A outlined above, the following amounts of reagents were used: 4-bromobenzaldehyde (1.85 g, 10.0 mmol, 1.00 equiv), phenylmagnesium bromide (7.0 mL, 1.7 M in THF, 12 mmol, 1.2 equiv), and THF (10 mL). The crude reaction mixture was purified by flash chromatography (5–20% EtOAc/hexanes) to afford the product as a white solid (1.92 g, 7.29 mmol, 73%). Analytical data is consistent with the values listed below for (**S**)-**SI-3**.

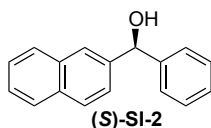
B. Representative procedure for enantioselective synthesis of diarylmethyl alcohols by asymmetric arylation.



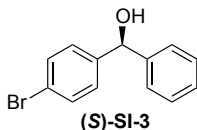
Enantioenriched alcohols were prepared according to a modified procedure of Braga and co-workers.⁴

(S)-SI-1. To a solution of phenylboronic acid (0.732 g, 6.00 mmol, 2.40 equiv) in toluene (10 mL) was added diethylzinc (18 mL, 18 mmol, 1.0 M in toluene, 7.2 equiv), and the solution was

allowed to stir at 60 °C for 12 h. Upon cooling to room temperature, (*S*)-(1-tritylaziridin-2-yl)diphenylmethanol (0.084 g, 0.06 mmol, 0.01 equiv) was added as a solution in toluene (5 mL) and the reaction mixture was allowed to stir for 10 minutes before the addition of a solution of biphenyl-4-carboxaldehyde (0.456 g, 2.50 mmol, 1.00 equiv) in toluene (5 mL). After stirring 12 h at room temperature, 1 N hydrochloric acid (10 mL) was added and the product was extracted with EtOAc (3 x 10 mL). The combined organics were washed with brine (10 mL), dried over MgSO₄, and concentrated in vacuo. The product was purified by flash column chromatography (0–1% EtOAc/benzene) and then recrystallized from hexanes and EtOAc to upgrade the ee (0.488 g, 1.85 mmol, 75% yield, 96% ee). **TLC** *R_f* = 0.2 (benzene); **m.p.** = 90–92 °C; **¹H NMR** (400 MHz, CDCl₃) δ 7.55 (m, 4H), 7.40 (m, 6H), 7.32 (m, 4H), 5.81 (s, 1H), 2.32 (d, *J* = 2.8, 1H); **¹³C NMR** δ (100 MHz, CDCl₃) δ 143.8, 142.9, 140.9, 140.6, 128.9, 128.7, 127.8, 127.4, 127.38, 127.2, 127.1, 126.7, 76.1; **IR** (neat) 3361, 3029, 1408, 1006, 763 cm⁻¹; **HRMS** (TOF MS ES+) *m/z* calcd for C₁₉H₁₆O (M + Na)⁺ 283.1099, found 283.1110; **[α]²³_D** +4.72 (*c* 1.10, CHCl₃); **SFC** analysis (AD-H, 15% IPA, 3 mL/min) indicated 96% ee: *t_R* (major) = 18.9 minutes, *t_R* (minor) = 20.5 minutes.



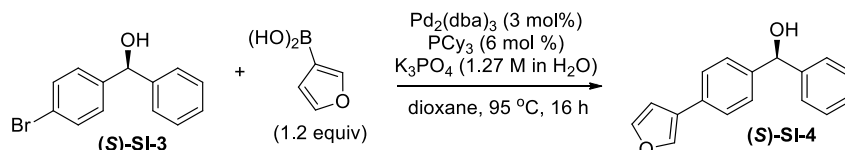
(S)-SI-2. Using the representative procedure B outlined above, the following amounts of reagents were used: phenylboronic acid (0.732 g, 6.00 mmol, 2.4 equiv), diethylzinc (18 mL, 18 mmol, 1.0 M in toluene), (*S*)-diphenyl(1-tritylaziridin-2-yl)methanol (116 mg, 0.250 mmol, 0.100 equiv), and 2-naphthaldehyde (0.390 g, 2.50 mmol, 1.00 equiv). The product was purified by flash chromatography (10–20% EtOAc/hexanes) to afford the product as a white solid (0.608 g, 2.59 mmol, 93%, 89% ee). The product was then recrystallized from hexanes to upgrade the ee (99% ee). Analytical data is consistent with literature values.² **¹H NMR** (400 MHz, CDCl₃) δ 7.91 (s, 1H), 7.82 (dt, *J* = 9.2, 2.6 Hz, 2H), 7.80 (d, *J* = 8.2 Hz, 1H), 7.49–7.40 (m, 5H), 7.35 (t, *J* = 7.0 Hz, 2H), 7.29 (dt, *J* = 7.4, 1.5 Hz, 1H), 6.02 (d, *J* = 3.5 Hz, 1H), 2.29 (d, *J* = 3.5 Hz, 1H); **[α]²³_D** +7.8 (*c* 0.92, CHCl₃), literature **[α]²⁰_D** +11.2 (*c* 0.83, CHCl₃); **SFC** analysis (OD-H, 20% 2-propanol, 3 mL/min) indicated >99% ee: *t_R* (major) = 6.4 min, *t_R* (minor) = 7.3 min.



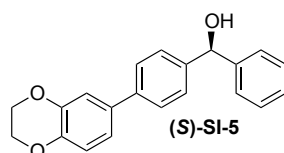
(S)-SI-3. Using the representative procedure B outlined above, the following amounts of reagents were used: phenylboronic acid (0.732 g, 6.00 mmol, 2.4 equiv), diethylzinc (18 mL, 18 mmol, 1.0 M in toluene), (*S*)-(1-tritylaziridin-2-yl)diphenylmethanol (116 mg, 0.250 mmol, 0.100 equiv), and 4-bromobenzaldehyde (0.463 g, 2.50 mmol, 1.00 equiv). The product was purified by flash chromatography (10–20% EtOAc/hexane) to afford the product as a white solid (0.608 g, 2.31 mmol, 93%, 92% ee). The product was then recrystallized from hexanes to yield higher enantiopurity (96% ee). Analytical data is consistent with literature values.⁵ **¹H NMR** (500 MHz, CDCl₃) δ 7.44 (d, *J* = 8.4 Hz, 2H), 7.36–7.30 (m, 4H), 7.29–7.25 (m, 1H), 7.23 (d, *J* = 8.4 Hz, 2H), 5.76 (d, *J* = 3.3 Hz, 1H), 2.34 (d, *J* = 3.3 Hz, 1H); **¹³C NMR** (125 MHz, CDCl₃) δ 143.5, 142.8, 131.7, 128.8, 128.3, 128.0, 126.6, 121.5, 75.8; **[α]²³_D** +17.5 (*c* 1.65, CHCl₃); **SFC**

analysis (AD-H, 10% IPA, 2.5 mL/min) indicated 96% ee: t_R (major) = 10.4 minutes, t_R (minor) = 9.8 minutes.

C. Representative procedure for the Suzuki cross-coupling of aryl bromide (**S**)-**SI-3** with aryl boronic acids.

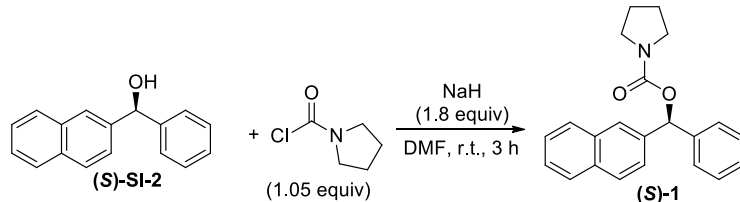


(S)-**SI-4**. The product was prepared according to a modified procedure by Fu and co-workers.⁶ Tris(dibenzylideneacetone)dipalladium (55 mg, 0.060 mmol, 0.030 equiv) and tricyclohexylphosphine (39 mg, 0.14 mmol, 0.070 equiv) were weighed out into a flame dried two neck, round bottom flask inside a glovebox. The flask was fitted with septa, removed from the glovebox, and 3-furanboronic acid (0.262 g, 2.20 mmol, 1.10 equiv), **(S)**-**SI-3** (0.526 g, 2.00 mmol, 1.00 equiv), aqueous potassium phosphate (2.7 mL, 3.4 mmol, 1.3 M in H₂O, 1.7 equiv) and dioxane (6 mL) were added. The reaction flask was fitted with a reflux condenser and heated to 95 °C for 16 h. After cooling, the solvent was removed under reduced pressure. The resultant residue was purified by flash column chromatography (10–20% EtOAc/hexane) to afford **(S)**-**SI-4** as a yellow solid (0.437 g, 1.75 mmol, 87%, 97% ee). **TLC** R_f = 0.2 (4:1 hexane/EtOAc); **m.p.** = 97–99 °C; **¹H NMR** (500 MHz, CDCl₃) δ 7.70 (s, 1H), 7.45 (s, 2H), 7.43 (s, 1H), 7.37 (t, J = 8.2 Hz, 3H), 7.33 (t, J = 7.3 Hz, 3H), 7.27 (d, J = 7.4 Hz, 1H), 6.67 (s, 1H), 5.82 (s, 1H), 2.32 (s, 1H); **¹³C NMR** (125 MHz, CDCl₃) δ 143.9, 143.8, 142.7, 138.6, 131.8, 128.7, 127.8, 127.1, 126.7, 126.2, 126.1, 108.9, 76.1; **IR** (neat) 3279, 1160, 1012, 780, 699 cm⁻¹; **HRMS** (TOF MS ES+) m/z calcd for C₁₇H₁₄O₂ (M + Na)⁺ 273.0891, found 273.0883; **[α]_D²⁹** = -37.3 (c 1.00, CHCl₃); **SFC** analysis (OD-H, 13% IPA, 2.5 mL/min) indicated 97% ee: t_R (major) = 12.9 minutes, t_R (minor) = 14.7 minutes.

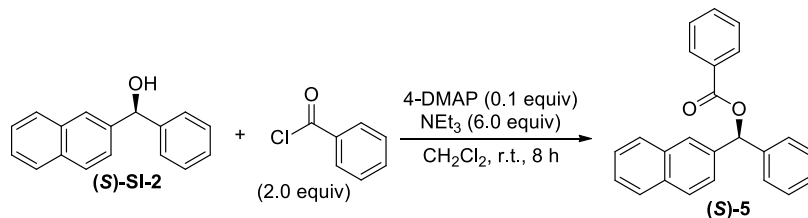


(S)-**SI-5**. Using representative procedure C outlined above, the following amounts of reagents were used: tris(dibenzylideneacetone)dipalladium (28 mg, 0.030 mmol, 0.030 equiv), tricyclohexylphosphine (20 mg, 0.07 mmol, 0.070 equiv), 1,4-benzodioxane-6-boronic acid (0.198 g, 1.10 mmol, 1.10 equiv), **(S)**-**SI-3** (0.263 g, 1.00 mmol, 1.00 equiv), aqueous potassium phosphate (1.4 mL, 1.7 mmol, 1.3 M in H₂O, 1.7 equiv) and dioxane (3 mL). The product was purified by flash column chromatography (10–30% EtOAc/hexane) to afford **(S)**-**SI-5** as a brown solid (0.296 g, 0.929 mmol, 93%, 96% ee). **TLC** R_f = 0.2 (4:1 hexane/EtOAc); **TLC** R_f = 0.3 (30% EtOAc/hexanes); **m.p.** = 108–110 °C; **¹H NMR** (500 MHz, CDCl₃) δ 7.48 (d, J = 7.7 Hz, 2H), 7.40 (q, J = 7.8 Hz, 4H), 7.34 (t, J = 7.2 Hz, 2H), 7.27 (d, J = 7.3 Hz, 1H), 7.08 (d, J = 1.9 Hz, 1H), 7.04 (dd, J = 8.5, 2.1 Hz, 1H), 6.90 (d, J = 8.2 Hz, 1H), 5.85 (s, 1H), 4.36 (s, 4H), 2.35 (s, 1H); **¹³C NMR** (125 MHz, CDCl₃) δ 143.9, 143.8, 143.3, 142.5, 140.0, 134.5, 128.7, 127.7, 127.1, 127.0, 126.6, 120.2, 117.7, 115.9, 76.2, 64.6, 64.5; **IR** (neat) 3550, 1494, 1304, 1284, 1070 cm⁻¹; **HRMS** (TOF MS ES+) m/z calcd for C₂₁H₁₈O₃ (M + Na)⁺ 341.1154, found 341.1147; **[α]_D²⁹** = +3.1 (c 1.04, CHCl₃); **SFC** analysis (AD-H, 14% IPA, 2.5 mL/min) indicated 96%: t_R (major) = 6.9 minutes, t_R (minor) = 8.8 minutes.

D. Preparation of protected carbinols.

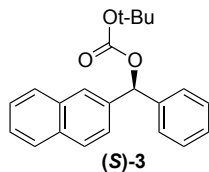


(S)-1. The product was prepared according to a modified procedure by Zhang and co-workers.⁷ To a suspension of NaH (0.153 g, 6.37 mmol, 1.80 equiv) in DMF (3 mL) was added a solution of **(S)-SI-2** (0.823 g, 3.54 mmol, 1.00 equiv) in DMF (2 mL) at 0 °C. The mixture was stirred for 1 h before addition of neat 1-pyrrolidinecarbonyl chloride (0.41 mL, 3.7 mmol, 1.1 equiv) at room temperature. After stirring for 3 h, the reaction was quenched with saturated aqueous ammonium chloride (6 mL), and the product was extracted with methylene chloride (3 x 10 mL). The combined organics were washed with brine (5 mL), dried over Na₂SO₄, and concentrated in vacuo. The product was purified by flash column chromatography (20% EtOAc/hexane) to afford **(S)-1** as a white solid (0.963 g, 2.91 mmol, 83%, 94% ee): **TLC** *R_f* = 0.2 (20% EtOAc/hexanes); **m.p.** = 151–153 °C; **¹H NMR** (500 MHz, CDCl₃) δ 7.84 (s, 1H), 7.82–7.77 (m, 3H), 7.47–7.44 (m, 3H), 7.41 (d, *J* = 7.3 Hz, 2H), 7.33 (t, *J* = 7.3 Hz, 2H), 7.27 (d, *J* = 7.4 Hz, 1H), 7.00 (s, 1H), 3.55 (t, *J* = 6.7 Hz, 2H), 3.40 (t, *J* = 6.7 Hz, 2H), 1.90 (dt, *J* = 13.3, 6.7 Hz, 2H), 1.84 (dt, *J* = 13.3, 6.7 Hz, 2H); **¹³C NMR** (125 MHz, CDCl₃) δ 154.2, 141.4 138.7, 133.2, 133.0, 128.5, 128.4 128.3, 127.8, 127.7, 127.2, 126.3, 126.2, 126.1, 125.2, 77.4, 46.4, 46.0, 25.9, 25.0; **IR** (neat) 1690, 1412, 1102, 828, 765 cm⁻¹; **HRMS** (TOF MS ES+) *m/z* calcd for C₂₂H₂₁NO₂ (M + Na)⁺ 354.1470, found 354.1463; **[α]_D²⁰** +45.9 (*c* 1.15, CHCl₃); **SFC** analysis (OD-H, 18% IPA, 2.5 mL/min) indicated 93% ee: *t_R* (major) = 7.1 minutes, *t_R* (minor) = 6.6 minutes.

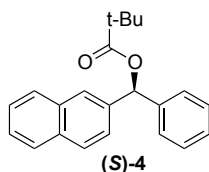


(S)-5. The product was prepared according to a modified procedure by Hassner and co-workers.⁸ To a 25 mL round bottom flask was added alcohol **(S)-SI-2** (0.175 g, 0.750 mmol, 1.00 equiv), and 4-(dimethylamino)pyridine (9.0 mg, 0.075 mmol, 0.10 equiv). The flask was evacuated and backfilled with nitrogen before addition of methylene chloride (6 mL), triethylamine (0.48 mL, 4.5 mmol, 6.0 equiv), and benzoyl chloride (0.18 mL, 1.5 mmol, 2.0 equiv). After stirring for 8 h, the reaction was quenched with 1 M HCl (6 mL), and the product was extracted with methylene chloride (3 x 10 mL). The combined organics were washed with brine (10 mL), dried over MgSO₄, and concentrated in vacuo. The product was purified by flash column chromatography (5–10% EtOAc/hexane) to afford **(S)-5** as a white solid (0.177 g, 0.523 mmol, 70%, 89% ee): **TLC** *R_f* = 0.4 (10% EtOAc/hexanes); **m.p.** = 91–93 °C; **¹H NMR** (500 MHz, CDCl₃) δ 8.26 (d, *J* = 7.9 Hz, 2H), 8.00 (s, 1H), 7.91–7.90 (m, 3H), 7.66 (t, *J* = 7.2 Hz, 1H), 7.61–7.53 (m, 6H), 7.45 (t, *J* = 7.1 Hz, 2H), 7.39 (d, *J* = 7.2 Hz, 2H); **¹³C NMR** (125 MHz, CDCl₃) δ 165.7, 140.3, 137.7, 133.3, 133.2, 133.1, 130.3, 130.0, 128.7, 128.6, 128.3, 128.2, 127.8, 127.4, 126.44, 126.40, 126.3, 125.1, 77.7; **IR** (neat) 1712, 1259, 1108, 732, 700 cm⁻¹;

HRMS (TOF MS ES+) m/z calcd for $C_{24}H_{18}O_2$ ($M + Na$)⁺ 361.1205, found 361.1201; $[\alpha]_D^{29}$ +10.0 (c 0.99, $CHCl_3$); **SFC analysis** (OD-H, 10.0% IPA, 2.5 mL/min) indicated 89% ee: t_R (major) = 6.5 minutes, t_R (minor) = 6.3 minutes.

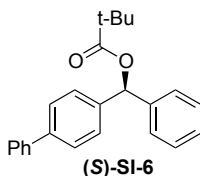


(S)-3. The product was prepared according to a modified procedure by Hassner and co-workers.⁸ To a 25 mL round bottom flask was added alcohol **(S)-SI-2** (0.234 g, 1.00 mmol, 1.00 equiv), and 4-(dimethylamino)pyridine (12 mg, 0.010 mmol, 0.10 equiv). The flask was evacuated and backfilled with nitrogen before addition of methylene chloride (8 mL), triethylamine (0.10 mL, 1.2 mmol, 1.2 equiv), and di-*tert*-butyl dicarbonate (0.228 g, 1.05 mmol, 1.05 equiv). After stirring for 8 h, the reaction was quenched with 1 M HCl (6 mL), and the product was extracted with methylene chloride (3 x 10 mL). The combined organics were washed with brine (10 mL), dried over $MgSO_4$, and concentrated in vacuo. The product was purified by flash column chromatography (5–10% EtOAc/hexane) to afford **(S)-3** as a white solid (0.284 g, 0.849 mmol, 85%, 88% ee): **TLC** R_f = 0.4 (9:1 hexane/EtOAc); **m.p.** = 90–92 °C; **¹H NMR** (500 MHz, $CDCl_3$) δ 7.87 (s, 1H), 7.83 (d, J = 7.0 Hz, 1H), 7.79 (d, J = 8.2 Hz, 2H), 7.48–7.45 (m, 2H), 7.43 (s, 1H), 7.41 (d, J = 8.3 Hz, 2H), 7.33 (t, J = 7.3 Hz, 2H), 7.27 (d, J = 7.7 Hz, 1H), 1.47 (s, 9H); **¹³C NMR** (125 MHz, $CDCl_3$) δ 153.1, 140.1, 137.6, 133.2, 133.1, 128.7, 128.5, 128.3, 128.1, 127.8, 127.2, 126.4, 126.3, 126.0, 125.0, 82.7, 80.0, 27.9; **IR** (neat) 1742, 1270, 1251, 1150, 1081 cm^{-1} ; **HRMS** (TOF MS ES+) m/z calcd for $C_{22}H_{22}O_3$ ($M + Na$)⁺ 357.1467, found 357.1467; $[\alpha]_D^{29}$ -19.3 (c 0.90, $CHCl_3$); **SFC analysis** (AD-H, 5% IPA, 3.0 mL/min) indicated 88% ee: t_R (major) = 5.6 minutes, t_R (minor) = 6.1 minutes.

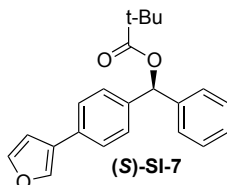


(S)-4. The product was prepared according to a modified procedure by Hassner and co-workers.⁸ To a 25 mL round bottom flask was added alcohol **(S)-SI-2** (0.281 g, 1.20 mmol, 1.20 equiv), and 4-(dimethylamino)pyridine (15 mg, 0.012 mmol, 0.10 equiv). The flask was evacuated and backfilled with nitrogen before addition of methylene chloride (8 mL), triethylamine (0.19 mL, 2.6 mmol, 2.2 equiv), and trimethylacetyl chloride (0.160 mL, 1.26 mmol, 1.05 equiv). After stirring for 8 h, the reaction was quenched with 1M HCl (6 mL), and the product was extracted with methylene chloride (3 x 10 mL). The combined organics were washed with brine (10 mL), dried over $MgSO_4$, and concentrated in vacuo. The product was purified by flash column chromatography (5–10% EtOAc/hexane) to afford **(S)-4** as a white solid (0.334 g, 1.05 mmol, 88%, 82% ee): **TLC** R_f = 0.5 (10% EtOAc/hexanes); **m.p.** = 80–83 °C; **¹H NMR** (500 MHz, $CDCl_3$) δ 7.82 (s, 2H), 7.79 (d, J = 8.8 Hz, 2H), 7.48–7.45 (m, 2H), 7.42 (d, J = 8.3 Hz, 1H), 7.38 (d, J = 7.6 Hz, 2H), 7.33 (t, J = 7.1 Hz, 2H), 7.28 (d, J = 7.6 Hz, 1H), 1.27 (s, 9H); **¹³C NMR** (125 MHz, $CDCl_3$) δ 177.4, 140.6, 138.0, 133.2, 133.0, 128.6, 128.5, 128.3, 127.9, 127.8, 127.1, 126.4, 126.3, 126.1, 125.0, 76.8, 39.1, 27.3; **IR** (neat) 1721, 1276, 1148, 1123, 823 cm^{-1} ; **HRMS** (TOF MS ES+) m/z calcd for $C_{22}H_{22}O_2$ ($M + Na$)⁺ 341.1518, found 341.1526; $[\alpha]_D^{29}$ -

37.4 (*c* 1.18, CHCl₃); **SFC analysis** (AD-H, 5.0% IPA, 3.0 mL/min) indicated 82% ee: *t_R* (major) = 6.8 minutes, *t_R* (minor) = 7.1 minutes.

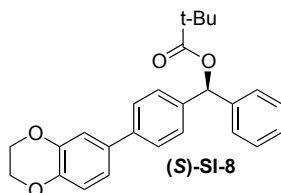


(S)-SI-6. The product was prepared according to a modified procedure by Zhang and co-workers.⁷ NaH (500 mg, 20.8 mmol, 4.00 equiv) was suspended in 40 mL of dry DMF and cooled to 0 °C. To this solution, alcohol **(S)-SI-1** (1.28 g, 4.92 mmol, 1.00 equiv) in dry DMF (10 mL) was added dropwise. The mixture was allowed to stir at 0 °C for 30 minutes after which pivaloyl chloride (4.3 mL, 35 mmol, 7.0 equiv) was added dropwise. The reaction was stirred at 0 °C for 1.5 hours then warmed to room temperature and stirred for 22 hours. The reaction was quenched by consecutive addition of water (5 x 2 mL) and stirring for 3 minutes. The reaction was diluted with more water (10 mL) and the organics were extracted with EtOAc (3 x 30 mL). The combined organic layers were dried over MgSO₄ and the solvent was removed under reduced pressure. The crude was purified by flash chromatography (0–1% Et₂O/petroleum ether) yielding **(S)-SI-1** as a white solid (1.56 g, 4.53 mmol, 92%). **TLC** *R_f* = 0.4 (10% Et₂O:petroleum ether); **m.p.** = 109–110 °C; **¹H NMR** (400 MHz, CDCl₃) δ 7.54 (dd, *J* = 6.1, 1.9 Hz, 4H), 7.35 (m, 10H), 6.87 (s, 1H), 1.27 (s, 9H); **¹³C NMR** (100 MHz, CDCl₃) δ 177.4, 140.8, 140.6, 139.8, 128.9, 128.7, 127.9, 127.8, 127.5, 127.40, 127.38, 127.2, 127.0, 76.5, 39.1, 27.3; **IR** (neat) 3029, 2974, 1722, 1275, 1138 cm⁻¹; **HRMS** (TOF MS ES+) *m/z* calcd for C₂₄H₂₄O₂ (M + Na)⁺ 367.1674, found 367.1681; **[α]_D²³** -23.6 (*c* 1.09, CHCl₃); **SFC analysis** (AD-H, 10% IPA, 3 mL/min) indicated 96% ee: *t_R* (minor) = 4.0 minutes, *t_R* (major) = 6.4 minutes.



(S)-SI-7. The product was prepared according to a modified procedure by Zhang and co-workers.⁷ To a suspension of NaH (72 mg, 3.0 mmol, 2.0 equiv) in DMF (3 mL) was added a solution of **(S)-SI-4** (0.374 g, 1.50 mmol, 1.00 equiv) in DMF (2 mL) at 0 °C. The mixture was stirred for 1 h before addition of neat trimethylacetyl chloride (0.200 mL, 1.60 mmol, 1.05 equiv) at room temperature. After stirring for 3 h, the reaction was quenched with saturated aqueous ammonium chloride (6 mL), and the product was extracted with methylene chloride (3 x 10 mL). The combined organics were washed with brine (5 mL), dried over Na₂SO₄, and concentrated in vacuo. The product was purified by flash column chromatography (30% Et₂O/hexane) to afford **(S)-SI-7** as a pale yellow solid (0.427 g, 1.28 mmol, 85%, 93% ee): **TLC** *R_f* = 0.2 (4:1 hexane/Et₂O); **m.p.** = 105–108 °C; **¹H NMR** (500 MHz, CDCl₃) δ 7.70 (s, 1H), 7.45 (s, 2H), 7.43 (s, 1H), 7.35 (s, 4H), 7.33 (s, 2H), 7.29–7.26 (m, 1H), 1.26 (s, 9H); **¹³C NMR** (125 MHz, CDCl₃) δ 177.4, 143.8, 140.6, 139.5, 138.7, 132.1, 128.6, 127.9, 127.6, 127.0, 126.2, 126.1, 108.9, 76.5, 39.0, 27.3; **IR** (neat) 1724, 1159, 1138, 757, 699 cm⁻¹; **HRMS** (TOF MS ES+) *m/z* calcd for C₂₂H₂₂O₃ (M + Na)⁺ 357.1467, found 357.1475; **[α]_D²⁹** -26.0 (*c* 1.25, CHCl₃); **SFC**

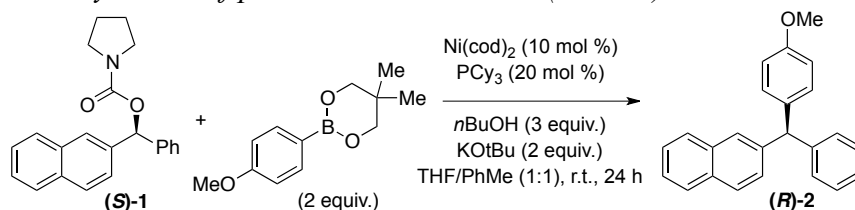
analysis (OJ-H, 8% IPA, 2.0 mL/min) indicated 93% ee: t_R (major) = 3.6 minutes, t_R (minor) = 4.2 minutes.



(S)-SI-8. The product was prepared according to a modified procedure by Zhang and co-workers.⁷ To a suspension of NaH (35 mg, 1.4 mmol, 1.8 equiv) in DMF (3 mL) was added a solution of **(S)-SI-5** (0.254 g, 0.800 mmol, 1.00 equiv) in DMF (2 mL) at 0 °C. The mixture was stirred for 1 h before addition of neat trimethylacetyl chloride (0.103 mL, 0.840 mmol, 1.05 equiv) at room temperature. After stirring for 3 h, the reaction was quenched with saturated aqueous ammonium chloride (6 mL), and the product was extracted with methylene chloride (3 x 10 mL). The combined organics were washed with brine (5 mL), dried over Na₂SO₄, and concentrated in vacuo. The product was purified by flash column chromatography (30% Et₂O/hexanes) to afford **(S)-SI-8** as a tan solid (0.232 g, 0.576 mmol, 73%, 94% ee): **TLC** R_f = 0.1 (20% Et₂O/hexanes); **¹H NMR** (500 MHz, CDCl₃) δ 7.48 (d, J = 8.2 Hz, 2H), 7.37–7.32 (m, 6H), 7.28 (d, J = 7.2 Hz, 1H), 7.08 (s, 1H), 7.04 (d, J = 8.4 Hz, 1H), 6.90 (d, J = 8.6 Hz, 1H), 6.84 (s, 1H), 4.27 (s, 4H), 1.27 (s, 9H); **¹³C NMR** (125 MHz, CDCl₃) δ 177.4, 143.8, 143.4, 140.7, 140.2, 139.3, 134.4, 128.7, 127.9, 127.4, 127.0, 126.9, 120.2, 117.7, 115.9, 76.5, 64.6, 64.5, 39.0, 27.3; **IR** (neat) 1723, 1494, 1309, 1147, 1068 cm⁻¹; **HRMS** (TOF MS ES+) m/z calcd for C₂₆H₂₆O₄ (M + Na)⁺ 425.1729, found 425.1715; **[α]_D²⁹** -20.3 (c 0.96, CHCl₃); **SFC** analysis (OD-H, 30% MeOH, 2.5 mL/min) indicated 94% ee: t_R (major) = 6.9 minutes, t_R (minor) = 8.8 minutes.

IV. Procedures for Cross-Coupling Reactions

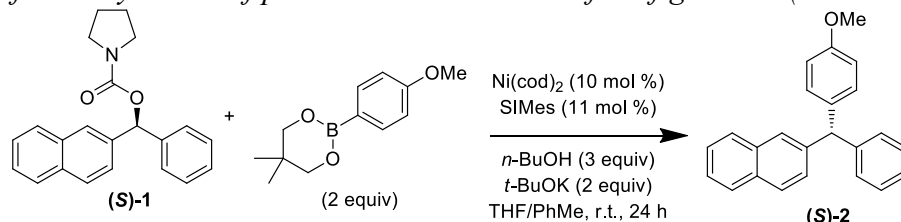
A. Procedure for the synthesis of products with retention (Table 2).



(R)-2. To a flame dried vial in a glovebox was added 1,8-bis(1,5-cyclooctadiene)nickel (5.5 mg, 0.020 mmol, 0.10 equiv), tricyclohexylphosphine (11 mg, 0.040 mmol, 0.20 equiv), potassium *tert*-butoxide (45 mg, 0.40 mmol, 2.0 equiv), 1-butanol (54 μ L, 0.60 mmol, 3.0 equiv), 5,5-dimethyl-2-(4-methoxyphenyl)-1,3,2-dioxaborinane (88 mg, 0.40 mmol, 2.0 equiv), **(S)-1** (66 mg, 0.20 mmol, 1.0 equiv), tetrahydrofuran (1 mL) and toluene (1 mL). The reaction was stirred for 24 hours before removing the vial from the glovebox, opening to atmosphere, and running through a silica gel plug (1:1 Et₂O:hexane). The combined organics were concentrated in vacuo, internal standard (PhTMS, 0.20 mmol) was added and ¹H NMR yield was collected. The product was purified by flash chromatography (1–3% Et₂O/pentane) to afford **(R)-2** as a colorless oil. First run: (56.0 mg, 0.173 mmol, 86%, 93% ee). Second run: (56.4 mg, 0.174 mmol, 87%, 93% ee). Analytical data is consistent with literature values:³ **¹H NMR** (500 MHz, CDCl₃) δ 7.81–7.76 (m, 1H), 7.74 (d, J = 8.6 Hz, 1H), 7.72–7.67 (m, 1H), 7.46 (s, 1H), 7.42 (dt, J = 9.5, 3.2 Hz,

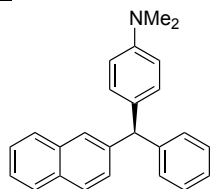
2H), 7.29 (t, $J = 7.2$ Hz, 3H), 7.24–7.19 (m, 1H), 7.15 (d, $J = 7.2$ Hz, 2H), 7.07 (d, $J = 8.4$ Hz, 2H), 6.83 (d, $J = 8.8$ Hz, 2H), 5.65 (s, 1H), 3.78 (s, 3H); ^{13}C NMR (125 MHz, CDCl_3) δ 158.2, 144.1, 142.0, 136.0, 133.5, 132.2, 130.6, 129.6, 128.4, 128.2, 128.0, 127.9, 127.8, 127.6, 126.4, 126.1, 125.7, 113.8, 56.2, 55.3; $[\alpha]_D^{23}$ -0.77 (c 2.70, CHCl_3); SFC analysis (AD-H, 15% IPA, 2.5 mL/min) indicated 93% ee: t_R (major) = 13.9 minutes, t_R (minor) = 13.2 minutes.

B. Procedure for the synthesis of products with inversion of configuration (Tables 2 and 3).



(S)-2. To a flame dried vial in a glovebox was added 1,8-bis(1,5-cyclooctadiene)nickel (5.5 mg, 0.020 mmol, 0.10 equiv), 1,3-Bis(2,4,6-trimethylphenyl)-4,5-dihydroimidazolium tetrafluoroborate (8.27 mg, 0.0210 mmol, 0.105 equiv), potassium *tert*-butoxide (45 mg, 0.40 mmol, 2.0 equiv), 1-butanol (54 μL , 0.60 mmol, 3.0 equiv), 5,5-dimethyl-2-(4-methoxyphenyl)-1,3,2-dioxaborinane (88 mg, 0.40 mmol, 2.0 equiv), **(S)-1** (66 mg, 0.20 mmol, 1.0 equiv), tetrahydrofuran (1 mL) and toluene (1 mL). The reaction was stirred for 24 hours before removing the vial from the glovebox, opening to atmosphere, and running through a silica gel plug (1:1 Et_2O :hexane). The combined organics were concentrated in vacuo, internal standard (PhTMS, 0.20 mmol) was added and ^1H NMR yield was collected. The product was purified by flash chromatography (1–3% Et_2O /pentane) to afford **(S)-2** as a colorless oil. First run: (53.2 mg, 0.164 mmol, 82%, 93% ee). Second run: (56.0 mg, 0.173 mmol, 86%, 93% ee). Analytical data is consistent with the values listed above for **(R)-2**. $[\alpha]_D^{23}$ $+2.1$ (c 2.70, CHCl_3); SFC analysis (AD-H, 15% IPA, 2.5 mL/min) indicated 90% ee: t_R (major) = 13.2 minutes, t_R (minor) = 13.9 minutes.

V. Characterization Data for Products

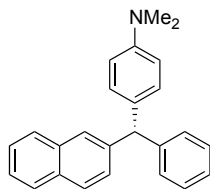


(R)

Table 2, entry 3

Table 2, entry 3. Using representative procedure A outlined above, the following amounts of reagents were used: 1,8-bis(1,5-cyclooctadiene)nickel (5.5 mg, 0.020 mmol, 0.10 equiv), tricyclohexylphosphine (11 mg, 0.040 mmol, 0.20 equiv), potassium *tert*-butoxide (45 mg, 0.40 mmol, 2.0 equiv), 1-butanol (54 μL , 0.60 mmol, 3.0 equiv), 5,5-dimethyl-2-(4-dimethylaminophenyl)-1,3,2-dioxaborinane (83 mg, 0.40 mmol, 2.0 equiv), **(S)-1** (66 mg, 0.20 mmol, 1.0 equiv), tetrahydrofuran (1 mL) and toluene (1 mL). The product was purified by flash chromatography (1–3% Et_2O /pentane) to afford the product as a colorless oil. First run: (49.8 mg, 0.148 mmol, 80%, 90% ee). Second run: (52.6 mg, 0.167 mmol, 84%, 90% ee). Analytical data is consistent with literature values: ^1H NMR (500 MHz, CDCl_3) δ 7.80–7.75 (m, 1H), 7.73 (d, $J = 8.7$ Hz, 1H), 7.71–7.67 (m, 1H), 7.48 (s, 1H), 7.40 (dt, $J = 9.4, 3.3$ Hz, 2H), 7.31 (d, $J =$

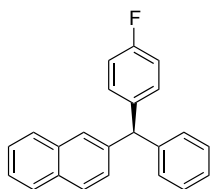
8.4 Hz, 1H), 7.27 (t, $J = 7.6$ Hz, 2H), 7.19 (t, $J = 7.5$ Hz, 1H), 7.16 (d, $J = 7.5$ Hz, 2H), 7.01 (d, $J = 8.8$ Hz, 2H), 6.67 (d, $J = 8.8$ Hz, 2H), 5.61 (s, 1H), 2.90 (s, 6H); ^{13}C NMR (125 MHz, CDCl_3) δ 149.2, 144.6, 142.5, 133.6, 132.2, 131.8, 130.3, 129.6, 128.4 (2C), 128.0, 127.8, 127.7, 127.6, 126.3, 126.0, 125.6, 112.6, 56.2, 40.8; IR (neat) 3054, 3023, 2879, 1612, 1350 cm^{-1} ; $[\alpha]_D^{23} -9.43$ (c 2.28, CHCl_3); SFC analysis (AD-H, 20% MeOH, 3 mL/min) indicated 92% ee: t_R (major) = 4.2 min, t_R (minor) = 4.8 min.



(S)

Table 2, entry 4

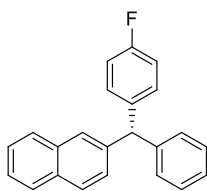
(S)-Table 2, entry 4. Using representative procedure B outlined above, the following amounts of reagents were used: 1,8-bis(1,5-cyclooctadiene)nickel (5.5 mg, 0.020 mmol, 0.10 equiv), 1,3-Bis(2,4,6-trimethylphenyl)-4,5-dihydroimidazolium tetrafluoroborate (8.27 mg, 0.0210 mmol, 0.105 equiv), potassium *tert*-butoxide (45 mg, 0.40 mmol, 2.0 equiv), 1-butanol (54 μL , 0.60 mmol, 3.0 equiv), 5,5-dimethyl-2-(4-dimethylaminophenyl)-1,3,2-dioxaborinane (83 mg, 0.40 mmol, 2.0 equiv), **(S)-SI-16** (66 mg, 0.20 mmol, 1.0 equiv), tetrahydrofuran (1 mL) and toluene (1 mL). The product was purified by flash chromatography (1–3% Et_2O /pentane) to afford the product as a colorless oil. First run: (42.1 mg, 0.125 mmol, 62%, 92% ee). Second run: (53.6 mg, 79%, 0.159 mmol, 92% ee). Analytical data is consistent with the values listed above for **(R)-Table 2, entry 3**. $[\alpha]_D^{23} +8.0$ (c 1.00, CHCl_3); SFC analysis (AD-H, 20% MeOH, 3 mL/min) indicated 92% ee: t_R (major) = 3.9 min, t_R (minor) = 4.6 min.



(R)

Table 2, entry 5

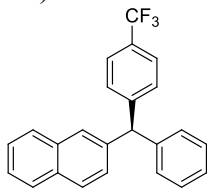
(R)-Table 2, entry 5. Using representative procedure A outlined above, the following amounts of reagents were used: 1,8-bis(1,5-cyclooctadiene)nickel (5.5 mg, 0.020 mmol, 0.10 equiv), tricyclohexylphosphine (11 mg, 0.040 mmol, 0.2 equiv), potassium *tert*-butoxide (45 mg, 0.40 mmol, 2.0 equiv), 1-butanol (54 μL , 0.60 mmol, 3.0 equiv), 5,5-dimethyl-2-(4-fluorophenyl)-1,3,2-dioxaborinane (83 mg, 0.40 mmol, 2.0 equiv), **(S)-1** (66 mg, 0.20 mmol, 1.0 equiv), tetrahydrofuran (1 mL) and toluene (1 mL). The product was purified by flash chromatography (1–3% Et_2O /pentane) to afford the product as a colorless oil. First run: (49.8 mg, 0.159 mmol, 80%, 90% ee). Second run: (52.6 mg, 0.168 mmol, 84%, 90% ee). Analytical data is consistent with literature values: ^1H NMR (500 MHz, CDCl_3) δ 7.81–7.77 (m, 1H), 7.75 (d, $J = 8.6$ Hz, 1H), 7.71–7.67 (m, 1H), 7.45–7.39 (m, 3H), 7.31–7.21 (m, 4H), 7.15–7.07 (m, 4H), 6.97 (t, $J = 8.8$ Hz, 2H), 5.70 (s, 1H); ^{13}C NMR (125 MHz, CDCl_3) δ 161.6 (d, $J = 245$ Hz), 143.6, 141.4, 139.5 (d, $J = 3$ Hz), 133.5, 132.3, 131.1 (d, $J = 8$ Hz), 129.6, 128.6, 128.1, 128.05, 127.99, 127.8, 127.7, 126.7, 126.2, 125.9, 115.3 (d, $J = 21$ Hz), 56.3; $[\alpha]_D^{23} +4.5$ (c 4.47, CHCl_3); SFC analysis (OJ-H, 12% IPA, 2.5 mL/min) indicated 90% ee: t_R (major) = 9.4 minutes, t_R (minor) = 8.7 minutes.



(S)

Table 2, entry 6

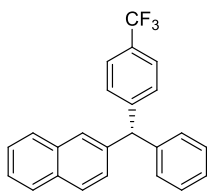
(R)-Table 2, entry 6. Using representative procedure B outlined above, the following amounts of reagents were used: 1,8-bis(1,5-cyclooctadiene)nickel (5.5 mg, 0.020 mmol, 0.10 equiv), 1,3-Bis(2,4,6-trimethylphenyl)-4,5-dihydroimidazolium tetrafluoroborate (8.27 mg, 0.0210 mmol, 0.105 equiv), potassium *tert*-butoxide (45 mg, 0.40 mmol, 2.0 equiv), 1-butanol (54 μ L, 0.60 mmol, 3.0 equiv), 5,5-dimethyl-2-(4-fluorophenyl)-1,3,2-dioxaborinane (83 mg, 0.40 mmol, 2.0 equiv), **(S)-1** (66 mg, 0.20 mmol, 1.0 equiv), tetrahydrofuran (1 mL) and toluene (1 mL). The product was purified by flash chromatography (1–3% Et₂O/pentane) to afford the product as a colorless oil. First run: (49.8 mg, 0.159 mmol, 80%, 88% ee). Second run: (50.0 mg, 0.168 mmol, 84%, 88% ee). Analytical data is consistent with literature values: ¹H NMR (500 MHz, CDCl₃) δ 7.81–7.77 (m, 1H), 7.75 (d, *J* = 8.6 Hz, 1H), 7.71–7.67 (m, 1H), 7.45–7.39 (m, 3H), 7.31–7.21 (m, 4H), 7.15–7.07 (m, 4H), 6.97 (t, *J* = 8.8 Hz, 2H), 5.70 (s, 1H); ¹³C NMR (125 MHz, CDCl₃) δ 161.6 (d, *J* = 245 Hz), 143.6, 141.4, 139.5 (d, *J* = 3 Hz), 133.5, 132.3, 131.1 (d, *J* = 8 Hz), 129.6, 128.6, 128.1, 128.05, 127.99, 127.8, 127.7, 126.7, 126.2, 125.9, 115.3 (d, *J* = 21 Hz), 56.3; [α]_D²³ –3.6 (*c* 4.10, CHCl₃); SFC analysis (OJ-H, 12% IPA, 2.5 mL/min) indicated 88% ee: *t*_R (major) = 9.7 minutes, *t*_R (minor) = 10.6 minutes.



(R)

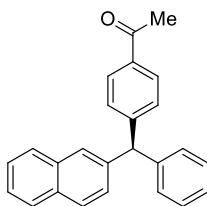
Table 2, entry 7

(S)-Table 2, entry 7. Using representative procedure A outlined above, the following amounts of reagents were used: 1,8-bis(1,5-cyclooctadiene)nickel (5.5 mg, 0.020 mmol, 0.10 equiv), tricyclohexylphosphine (11 mg, 0.040 mmol, 0.2 equiv), potassium *tert*-butoxide (45 mg, 0.40 mmol, 2.0 equiv), 1-butanol (54 μ L, 0.60 mmol, 3.00 equiv), 5,5-dimethyl-2-(4-trifluoromethylphenyl)-1,3,2-dioxaborinane (103 mg, 0.400 mmol, 2.00 equiv), **(S)-1** (66 mg, 0.20 mmol, 1.0 equiv), tetrahydrofuran (1 mL) and toluene (1 mL). The product was purified by flash column chromatography (1% Et₂O/pentane) to afford the product as a colorless oil. First run: (64.4 mg, 0.178 mmol, 89%, 57% ee). Second run: (62.1 mg, 0.172 mmol, 86%, 57% ee). TLC *R*_f = 0.4 (pentane); ¹H NMR (500 MHz, CDCl₃) δ 7.85–7.79 (m, 1H), 7.77 (d, *J* = 8.6 Hz, 1H), 7.74–7.68 (m, 1H), 7.55 (d, *J* = 8.1 Hz, 2H), 7.49–7.41 (m, 3H), 7.32 (t, *J* = 7.4 Hz, 2H), 7.29–7.22 (m, 4H), 7.14 (d, *J* = 7.6 Hz, 2H), 5.75 (s, 1H); ¹³C NMR (125 MHz, CDCl₃) δ 148.0, 142.9, 140.6, 133.5, 132.4, 130.0, 129.6, 128.9 (q, *J* = 32.4 Hz), 128.7, 128.3, 128.02, 128.00, 127.9, 127.7, 126.9, 126.4, 126.1, 125.5 (q, *J* = 3.7 Hz), 124.4 (q, *J* = 271.9 Hz), 56.9; IR (neat) 3057, 1600, 1323, 1119 cm⁻¹; HRMS (TOF MS CI+) *m/z* calcd for C₁₅H₁₆O (M)⁺ 362.1282, found 362.1273; [α]_D²³ +4.84 (*c* 0.915, CHCl₃); SFC analysis (AD-H, 5% IPA, 2.5 mL/min) indicated 57% ee: *t*_R (major) = 7.7 minutes, *t*_R (minor) = 7.0 minutes.



(**S**)
Table 2, entry 8

(S)-Table 2, entry 8. Using representative procedure B outlined above, the following amounts of reagents were used: 1,8-bis(1,5-cyclooctadiene)nickel (5.5 mg, 0.020 mmol, 0.10 equiv), 1,3-bis(2,4,6-trimethylphenyl)-4,5-dihydroimidazolium tetrafluoroborate (8.27 mg, 0.0210 mmol, 0.105 equiv), potassium *tert*-butoxide (45 mg, 0.40 mmol, 2.0 equiv), 1-butanol (54 μ L, 0.60 mmol, 3.00 equiv), 5,5-dimethyl-2-(4-trifluoromethylphenyl)-1,3,2-dioxaborinane (103 mg, 0.400 mmol, 2.00 equiv), (**S**)-**1** (66 mg, 0.20 mmol, 1.0 equiv), tetrahydrofuran (1 mL) and toluene (1 mL). The product was purified by flash column chromatography (1% Et₂O/pentane) to afford the product as a colorless oil. First run: (52.8 mg, 0.146 mmol, 73%, 91% ee). Second run: (48.6 mg, 0.134 mmol, 67%, 90% ee). **TLC** R_f = 0.4 (pentane); **¹H NMR** (500 MHz, CDCl₃) δ 7.85–7.79 (m, 1H), 7.77 (d, J = 8.6 Hz, 1H), 7.74–7.68 (m, 1H), 7.55 (d, J = 8.1 Hz, 2H), 7.49–7.41 (m, 3H), 7.32 (t, J = 7.4 Hz, 2H), 7.29–7.22 (m, 4H), 7.14 (d, J = 7.6 Hz, 2H), 5.75 (s, 1H); **¹³C NMR** (125 MHz, CDCl₃) δ 148.0, 142.9, 140.6, 133.5, 132.4, 130.0, 129.6, 128.9 (q, J = 32.4 Hz), 128.7, 128.3, 128.02, 128.00, 127.9, 127.7, 126.9, 126.4, 126.1, 125.5 (q, J = 3.7 Hz), 124.4 (q, J = 271.9 Hz), 56.9; **IR** (neat) 3057, 1600, 1323, 1119 cm⁻¹; **HRMS** (TOF MS CI+) m/z calcd for C₁₅H₁₆O (M)⁺ 362.1282, found 362.1273; [α]_D²³ -16.5 (c 1.00, CHCl₃); **SFC** analysis (AD-H, 5% IPA, 2.5 mL/min) indicated 89% ee: t_R (major) = 6.6 minutes, t_R (minor) = 7.3 minutes.



(**R**)
Table 2, entry 9

(R)-Table 2, entry 9. Using representative procedure A outlined above, the following amounts of reagents were used: 1,8-bis(1,5-cyclooctadiene)nickel (5.5 mg, 0.020 mmol, 0.10 equiv), tricyclohexylphosphine (11 mg, 0.040 mmol, 0.20 equiv), potassium *tert*-butoxide (45 mg, 0.40 mmol, 2.0 equiv), 1-butanol (54 μ L, 0.60 mmol, 3.0 equiv), 5,5-dimethyl-2-(4-acetylphenyl)-1,3,2-dioxaborinane (93 mg, 0.40 mmol, 2.0 equiv), (**S**)-**1** (66 mg, 0.20 mmol, 1.0 equiv), tetrahydrofuran (1 mL) and toluene (1 mL). The product was purified by flash column chromatography (10% EtOAc/hexanes) to afford the product as an amorphous white solid. First run: (50.8 mg, 0.151 mmol, 76%, 89% ee). Second run: (51.0 mg, 0.152 mmol, 76%, 89% ee). **TLC** R_f = 0.4 (20% EtOAc/hexanes); **¹H NMR** (500 MHz, CDCl₃) δ 7.80 (d, J = 8.4 Hz, 2H), 7.73–7.69 (m, 1H), 7.67 (d, J = 8.6 Hz, 1H), 7.63–7.57 (m, 1H), 7.40–7.30 (m, 3H), 7.21 (q, J = 7.7 Hz, 2H), 7.19–7.10 (m, 4H), 7.05 (d, J = 7.5 Hz, 2H), 5.65 (s, 1H), 2.47 (s, 3H); **¹³C NMR** (125 MHz, CDCl₃) δ 197.9, 149.4, 142.9, 140.7, 135.6, 133.5, 132.3, 129.9, 129.6, 128.7, 128.6, 128.3, 127.97, 127.95, 127.9, 127.7, 126.9, 126.3, 126.0, 57.0, 26.7; **IR** (neat) 3055, 2923, 1679, 1600, 1506 cm⁻¹; **HRMS** (TOF MS CI+) m/z calcd for C₂₅H₂₀O (M)⁺ 336.1514, found

3316.1514; $[\alpha]_D^{23}$ -17.2 (c 2.3, CHCl_3); SFC analysis (OD-H, 20% IPA, 3.0 mL/min) indicated 89% ee: t_R (major) = 6.3 minutes, t_R (minor) = 5.9 minutes.

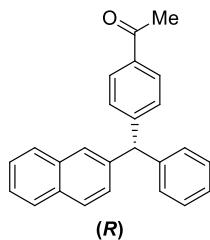


Table 2, entry 10

(R)-Table 2, entry 10. Using representative procedure B outlined above, the following amounts of reagents were used: 1,8-bis(1,5-cyclooctadiene)nickel (5.5 mg, 0.020 mmol, 0.10 equiv), 1,3-Bis(2,4,6-trimethylphenyl)-4,5-dihydroimidazolium tetrafluoroborate (8.67 mg, 0.0220 mmol, 0.11 equiv), potassium *tert*-butoxide (45 mg, 0.40 mmol, 2.0 equiv), 1-butanol (54 μL , 0.60 mmol, 3.0 equiv), 5,5-dimethyl-2-(4-acetylphenyl)-1,3,2-dioxaborinane (93 mg, 0.40 mmol, 2.0 equiv), **(S)-1** (66 mg, 0.20 mmol, 1.0 equiv), tetrahydrofuran (1 mL) and toluene (1 mL). The product was purified by flash column chromatography (10% EtOAc/hexanes) to afford the product as an amorphous white solid. First run: (66.5 mg, 0.198 mmol, 99%, 97% ee). Second run: (66.0 mg, 0.196 mmol, 98%, 97% ee). TLC R_f = 0.4 (20% EtOAc/hexanes); $^1\text{H NMR}$ (500 MHz, CDCl_3) δ 7.80 (d, J = 8.4 Hz, 2H), 7.73–7.69 (m, 1H), 7.67 (d, J = 8.6 Hz, 1H), 7.63–7.57 (m, 1H), 7.40–7.30 (m, 3H), 7.21 (q, J = 7.7 Hz, 2H), 7.19–7.10 (m, 4H), 7.05 (d, J = 7.5 Hz, 2H), 5.65 (s, 1H), 2.47 (s, 3H); $^{13}\text{C NMR}$ (125 MHz, CDCl_3) δ 197.9, 149.4, 142.9, 140.7, 135.6, 133.5, 132.3, 129.9, 129.6, 128.7, 128.6, 128.3, 127.97, 127.95, 127.9, 127.7, 126.9, 126.3, 126.0, 57.0, 26.7; IR (neat) 3055, 2923, 1679, 1600, 1506 cm^{-1} ; HRMS (TOF MS CI^+) m/z calcd for $\text{C}_{25}\text{H}_{20}\text{O}$ (M^+) 336.1514, found 336.1514; $[\alpha]_D^{29}$ +5.05 (c 1.01, CHCl_3); SFC analysis (OD-H, 20% IPA, 3.0 mL/min) indicated 97% ee: t_R (major) = 5.9 minutes, t_R (minor) = 6.5 minutes.

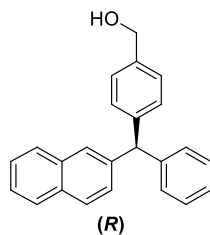
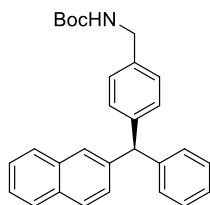


Table 2, entry 11

(R)-Table 2, entry 11. Using representative procedure A outlined above, the following amounts of reagents were used: 1,8-bis(1,5-cyclooctadiene)nickel (5.5 mg, 0.020 mmol, 0.10 equiv), tricyclohexylphosphine (11 mg, 0.040 mmol, 0.20 equiv), potassium *tert*-butoxide (45 mg, 0.40 mmol, 2.0 equiv), 1-butanol (54 μL , 0.60 mmol, 3.0 equiv), 5,5-dimethyl-2-(4-hydroxymethylphenyl)-1,3,2-dioxaborinane (88 mg, 0.40 mmol, 2.0 equiv), **(S)-1** (66 mg, 0.20 mmol, 1.0 equiv), tetrahydrofuran (1 mL) and toluene (1 mL). The product was purified by flash column chromatography (10% EtOAc/hexane) to afford the product as an oil. First run: (51.0 mg, 0.157 mmol, 79%, 82% ee). Second run: (50.0 mg, 0.154 mmol, 77%, 81% ee). TLC R_f = 0.2 (20% EtOAc/hexanes); $^1\text{H NMR}$ (500 MHz, CDCl_3) δ 7.82–7.76 (m, 1H), 7.74 (d, J = 8.7 Hz, 1H), 7.71–7.66 (m, 1H), 7.46 (s, 1H), 7.41 (dt, J = 9.5, 4.4 Hz, 2H), 7.34–7.24 (m, 5H),

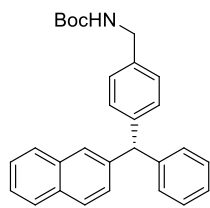
7.24–7.19 (m, 1H), 7.15 (d, $J = 8.4$ Hz, 4H), 5.69 (s, 1H), 4.64 (d, $J = 4$ Hz, 2H), 1.77 (t, $J = 4.5$ Hz, 1H); ^{13}C NMR (125 MHz, CDCl_3) δ 143.7, 143.3, 141.5, 139.1, 133.5, 132.3, 129.9, 129.6, 128.5, 128.1, 128.02, 127.96, 127.9, 127.7, 127.3, 126.6, 126.1, 125.8, 65.2, 56.8; IR (neat) 3330 (br), 2953, 1600, 1506 cm^{-1} ; HRMS (TOF MS CI+) m/z calcd for $\text{C}_{24}\text{H}_{18}\text{O}$ ($\text{M} - 2\text{H}$) $^+$ 322.1358, found 322.1364; $[\alpha]_D^{23}$ -18.3 (c 1.66, CHCl_3); SFC analysis (AD-H, 30% MeOH, 2.5 mL/min) indicated 89% ee: t_R (major) = 4.3 minutes, t_R (minor) = 6.1 minutes.



(R)

Table 2, entry 13

(R)-Table 2, entry 12. Using representative procedure A outlined above, the following amounts of reagents were used: 1,8-bis(1,5-cyclooctadiene)nickel (5.5 mg, 0.020 mmol, 0.10 equiv), tricyclohexylphosphine (11 mg, 0.040 mmol, 0.2 equiv), potassium *tert*-butoxide (45 mg, 0.40 mmol, 2.0 equiv), 1-butanol (54 μL , 0.60 mmol, 3.0 equiv), 5,5-dimethyl-2-(4-{(tert-butoxycarbonyl)amino}methyl}phenyl)-1,3,2-dioxaborinane (128 mg, 0.400 mmol, 2.00 equiv), **(S)-1** (66 mg, 0.20 mmol, 1.0 equiv), tetrahydrofuran (1 mL) and toluene (1 mL). The product was purified by flash column chromatography (15–25% Et_2O /hexanes) to afford the product as a white solid. First run: (71.0 mg, 0.168 mmol, 84%, 92% ee). Second run: (70.5 mg, 0.166 mmol, 83%, 89% ee). TLC $R_f = 0.3$ (20% EtOAc /hexanes); **m.p.** = 57 $^\circ\text{C}$; ^1H NMR (500 MHz, CDCl_3) δ 7.81–7.76 (m, 1H), 7.74 (d, $J = 8.6$ Hz, 1H), 7.71–7.66 (m, 1H), 7.45 (s, 1H), 7.41 (dt, $J = 9.5, 1.0$ Hz, 2H), 7.28 (t, $J = 7.4$ Hz, 3H), 7.22 (d, $J = 7.6$ Hz, 1H), 7.19 (d, $J = 8.0$ Hz, 2H), 7.14 (d, $J = 7.5$ Hz, 2H), 7.11 (d, $J = 8.0$ Hz, 2H), 5.67 (s, 1H), 4.83 (br s, 1H), 4.29 (d, $J = 5.1$ Hz, 2H), 1.45 (s, 9H); ^{13}C NMR (125 MHz, CDCl_3) δ 156.0, 143.7, 142.9, 141.5, 137.1, 133.5, 132.2, 129.9, 129.6, 128.5, 128.1, 128.01, 127.95, 127.8, 127.64, 127.59, 126.5, 126.1, 125.8, 79.5, 56.7, 44.4, 28.5; IR (neat) 3346, 2876, 1698, 1600, 1365 cm^{-1} ; HRMS (TOF MS ES+) m/z calcd for $\text{C}_{29}\text{H}_{29}\text{O}_2\text{N}$ ($\text{M} + \text{Na}$) $^+$ 446.2096, found 446.2078; $[\alpha]_D^{23}$ -14.3 (c 4.4, CHCl_3); SFC analysis (AS-H, 20% MeOH, 2.5 mL/min) indicated 92% ee: t_R (major) = 4.3 minutes, t_R (minor) = 4.7 minutes.

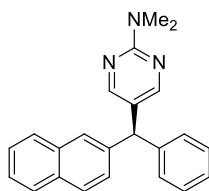


(S)

Table 2, entry 14

(R)-Table 2, entry 13. Using representative procedure B outlined above, the following amounts of reagents were used: 1,8-bis(1,5-cyclooctadiene)nickel (5.5 mg, 0.020 mmol, 0.10 equiv), 1,3-Bis(2,4,6-trimethylphenyl)-4,5-dihydroimidazolium tetrafluoroborate (8.27 mg, 0.0210 mmol, 0.105 equiv), potassium *tert*-butoxide (45 mg, 0.40 mmol, 2.0 equiv), 1-butanol (54 μL , 0.60 mmol, 3.0 equiv), 5,5-dimethyl-2-(4-{(tert-butoxycarbonyl)amino}methyl}phenyl)-1,3,2-dioxaborinane (128 mg, 0.400 mmol, 2.00 equiv), **(S)-1** (66 mg, 0.20 mmol, 1.0 equiv),

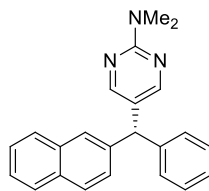
tetrahydrofuran (1 mL) and toluene (1 mL). The product was purified by flash column chromatography (15–25% Et₂O/hexanes) to afford the product as a white solid. First run: (84.0 mg, 0.198 mmol, 99%, 96% ee). Second run: (75.4 mg, 0.178 mmol, 89%, 94% ee). **TLC** R_f = 0.3 (20% EtOAc/hexanes); **m.p.** = 57 °C; **¹H NMR** (500 MHz, CDCl₃) δ 7.81–7.76 (m, 1H), 7.74 (d, J = 8.6 Hz, 1H), 7.71–7.66 (m, 1H), 7.45 (s, 1H), 7.41 (dt, J = 9.5, 1.0 Hz, 2H), 7.28 (t, J = 7.4 Hz, 3H), 7.22 (d, J = 7.6 Hz, 1H), 7.19 (d, J = 8.0 Hz, 2H), 7.14 (d, J = 7.5 Hz, 2H), 7.11 (d, J = 8.0 Hz, 2H), 5.67 (s, 1H), 4.83 (br s, 1H), 4.29 (d, J = 5.1 Hz, 2H), 1.45 (s, 9H); **¹³C NMR** (125 MHz, CDCl₃) δ 156.0, 143.7, 142.9, 141.5, 137.1, 133.5, 132.2, 129.9, 129.6, 128.5, 128.1, 128.01, 127.95, 127.8, 127.64, 127.59, 126.5, 126.1, 125.8, 79.5, 56.7, 44.4, 28.5; **IR** (neat) 3346, 2876, 1698, 1600, 1365 cm⁻¹; **HRMS** (TOF MS ES+) m/z calcd for C₂₉H₂₉O₂N (M + Na)⁺ 446.2096, found 446.2078; **[α]_D²⁵** +22.1 (c 1.01, CHCl₃); **SFC** analysis (AS-H, 20% MeOH, 2.5 mL/min) indicated 96% ee: t_R (major) = 4.5 minutes, t_R (minor) = 4.3 minutes.



(S)

Table 2, entry 15

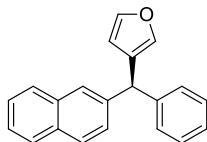
(S)-Table 2, entry 14. Using representative procedure A outlined above, the following amounts of reagents were used: 1,8-bis(1,5-cyclooctadiene)nickel (5.5 mg, 0.020 mmol, 0.10 equiv), tricyclohexylphosphine (11 mg, 0.040 mmol, 0.20 equiv), potassium *tert*-butoxide (45 mg, 0.40 mmol, 2.0 equiv), 1-butanol (54 μL, 0.60 mmol, 3.0 equiv), 5,5-dimethyl-2-((dimethylamino)-5-pyrimidinylphenyl)-1,3,2-dioxaborinane (94 mg, 0.40 mmol, 2.0 equiv), **(S)-1** (66 mg, 0.20 mmol, 1.0 equiv), tetrahydrofuran (1 mL) and toluene (1 mL). The product was purified by flash column chromatography (5% EtOAc/benzene) to afford the product as a white solid. First run: (58.2 mg, 0.171 mmol, 86%, 89% ee). Second run: (58.6 mg, 0.173 mmol, 86%, 89% ee). **TLC** R_f = 0.5 (5% EtOAc/benzene); **m.p.** = 45–47 °C; **¹H NMR** (500 MHz, CDCl₃) δ 8.04 (s, 2H), 7.73–7.67 (m, 1H), 7.66 (d, J = 8.6 Hz, 1H), 7.64–7.59 (m, 1H), 7.40 (s, 1H), 7.33 (dt, J = 9.5, 3.5 Hz, 2H), 7.23–7.16 (m, 3H), 7.15–7.10 (m, 1H), 7.06 (d, J = 7.6 Hz, 2H), 2.39 (s, 1H), 3.08 (s, 6H); **¹³C NMR** (125 MHz, CDCl₃) δ 161.3, 158.5, 142.9, 140.7, 133.5, 132.3, 129.8, 128.7, 128.3, 127.9, 127.67, 127.65, 127.6, 126.8, 126.3, 125.9, 123.6, 51.7, 37.2; **IR** (neat) 3054, 3023, 2861, 1599, 1531 cm⁻¹; **HRMS** (TOF MS ES+) m/z calcd for C₂₃H₂₁N₃ (M + H)⁺ 340.1814, found 340.1819; **[α]_D²³** +15.7 (c 2.51, CHCl₃); **SFC** analysis (AD-H, 30% MeOH, 2.5 mL/min) indicated 89% ee: t_R (major) = 4.6 minutes, t_R (minor) = 6.4 minutes.



(R)

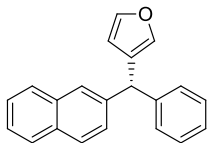
Table 2, entry 16

(S)-Table 2, entry 15. Using representative procedure B outlined above, the following amounts of reagents were used: 1,8-bis(1,5-cyclooctadiene)nickel (5.5 mg, 0.020 mmol, 0.10 equiv), 1,3-Bis(2,4,6-trimethylphenyl)-4,5-dihydroimidazolium tetrafluoroborate (8.27 mg, 0.0210 mmol, 0.105 equiv), potassium *tert*-butoxide (45 mg, 0.40 mmol, 2.0 equiv), 1-butanol (54 μ L, 0.60 mmol, 3.0 equiv), 5,5-dimethyl-2-((dimethylamino)-5-pyrimidinylphenyl)-1,3,2-dioxaborinane (94 mg, 0.40 mmol, 2.0 equiv), **(S)-1** (66 mg, 0.20 mmol, 1.0 equiv), tetrahydrofuran (1 mL) and toluene (1 mL). The product was purified by flash column chromatography (5% EtOAc/benzene) to afford the product as a white solid (50.8 mg, 0.150 mmol, 75%, 92% ee). **TLC** R_f = 0.5 (5% EtOAc/benzene); **m.p.** = 45–47 $^{\circ}$ C; $^1\text{H NMR}$ (500 MHz, CDCl_3) δ 8.04 (s, 2H), 7.73–7.67 (m, 1H), 7.66 (d, J = 8.6 Hz, 1H), 7.64–7.59 (m, 1H), 7.40 (s, 1H), 7.33 (dt, J = 9.5, 3.5 Hz, 2H), 7.23–7.16 (m, 3H), 7.15–7.10 (m, 1H), 7.06 (d, J = 7.6 Hz, 2H), 2.39 (s, 1H), 3.08 (s, 6H); $^{13}\text{C NMR}$ (125 MHz, CDCl_3) δ 161.3, 158.5, 142.9, 140.7, 133.5, 132.3, 129.8, 128.7, 128.3, 127.9, 127.67, 127.65, 127.6, 126.8, 126.3, 125.9, 123.6, 51.7, 37.2; **IR** (neat) 3054, 3023, 2861, 1599, 1531 cm^{-1} ; **HRMS** (TOF MS ES+) m/z calcd for $\text{C}_{23}\text{H}_{21}\text{N}_3$ ($\text{M} + \text{H}$) $^+$ 340.1814, found 340.1819; $[\alpha]_D^{29}$ -13.2 (c 0.675, CHCl_3); **SFC** analysis (AD-H, 30% MeOH, 2.5 mL/min) indicated 92% ee: t_R (major) = 6.1 minutes, t_R (minor) = 4.5 minutes.



(S)
Table 2, entry 17

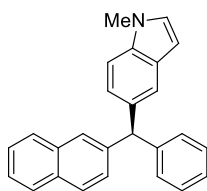
(S)-Table 2, entry 16. Using representative procedure A outlined above, the following amounts of reagents were used: 1,8-bis(1,5-cyclooctadiene)nickel (5.5 mg, 0.020 mmol, 0.10 equiv), tricyclohexylphosphine (11 mg, 0.040 mmol, 0.20 equiv), potassium *tert*-butoxide (45 mg, 0.40 mmol, 2.0 equiv), 1-butanol (54 μ L, 0.60 mmol, 3.0 equiv), 5,5-dimethyl-2-(3-furanyl)-1,3,2-dioxaborinane (72 mg, 0.40 mmol, 2.0 equiv), **(S)-1** (66 mg, 0.20 mmol, 1.0 equiv), tetrahydrofuran (1 mL) and toluene (1 mL). The product was purified by flash column chromatography (0.5–1% Et_2O /pentane) to afford the product as a white solid. First run: (45.8 mg, 0.161 mmol, 80%, 94% ee). Second run: (44.0 mg, 0.155 mmol, 78%, 94% ee). **TLC** R_f = 0.5 (1% Et_2O /pentane); **m.p.** = 65–67 $^{\circ}$ C; $^1\text{H NMR}$ (500 MHz, CDCl_3) δ 7.83–7.69 (m, 3H), 7.60 (s, 1H), 7.47–7.37 (m, 3H), 7.34 (dd, J = 8.6, 1 Hz, 1H), 7.31–7.16 (m, 5H), 6.97 (s, 1H), 6.26 (s, 1H), 5.42 (s, 1H); $^{13}\text{C NMR}$ (125 MHz, CDCl_3) δ 143.4, 143.3, 141.3, 141.1, 133.5, 132.4, 129.0, 128.6, 128.3, 128.1, 128.0, 127.7, 127.6, 127.1, 126.7, 126.2, 125.8, 111.6, 48.3; **IR** (neat) 3145, 3024, 1599, 1492 cm^{-1} ; **HRMS** (TOF MS CI+) m/z calcd for $\text{C}_{21}\text{H}_{16}\text{O}$ (M) $^+$ 284.1201, found 284.1203; $[\alpha]_D^{23}$ $+22.3$ (c 1.67, CHCl_3); **SFC** analysis (AD-H, 5% IPA, 2.5 mL/min) indicated 94% ee: t_R (major) = 12.2 minutes, t_R (minor) = 11.3 minutes.



(R)
Table 2, entry 18

(S)-Table 2, entry 17. Using representative procedure B outlined above, the following amounts of reagents were used: 1,8-bis(1,5-cyclooctadiene)nickel (5.5 mg, 0.020 mmol, 0.10 equiv), 1,3-

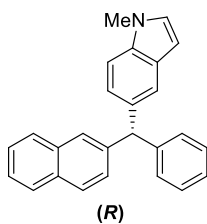
Bis(2,4,6-trimethylphenyl)-4,5-dihydroimidazolium tetrafluoroborate (8.27 mg, 0.0210 mmol, 0.105 equiv), potassium *tert*-butoxide (45 mg, 0.40 mmol, 2.0 equiv), 1-butanol (54 μ L, 0.60 mmol, 3.0 equiv), 5,5-dimethyl-2-(3-furanyl)-1,3,2-dioxaborinane (72 mg, 0.40 mmol, 2.0 equiv), (**S**)-**1** (66 mg, 0.20 mmol, 1.0 equiv), tetrahydrofuran (1 mL) and toluene (1 mL). The product was purified by flash column chromatography (0.5–1% Et₂O/pentane) to afford the product as a white solid. First run: (35.5 mg, 0.125 mmol, 62.5 %, 82% ee). Second run: (38.7 mg, 0.136 mmol, 68%, 84% ee). **TLC** R_f = 0.5 (1% Et₂O/pentane); **m.p.** = 65–67 °C; **¹H NMR** (500 MHz, CDCl₃) δ 7.83–7.69 (m, 3H), 7.60 (s, 1H), 7.47–7.37 (m, 3H), 7.34 (dd, J = 8.6, 1 Hz, 1H), 7.31–7.16 (m, 5H), 6.97 (s, 1H), 6.26 (s, 1H), 5.42 (s, 1H); **¹³C NMR** (125 MHz, CDCl₃) δ 143.4, 143.3, 141.3, 141.1, 133.5, 132.4, 129.0, 128.6, 128.3, 128.1, 128.0, 127.7, 127.6, 127.1, 126.7, 126.2, 125.8, 111.6, 48.3; **IR** (neat) 3145, 3024, 1599, 1492 cm⁻¹; **HRMS** (TOF MS CI⁺) m/z calcd for C₂₁H₁₆O (M)⁺ 284.1201, found 284.1203; $[\alpha]_D^{29}$ –22.0 (c 1.00, CHCl₃); **SFC** analysis (AD-H, 5% IPA, 2.5 mL/min) indicated 84% ee: t_R (major) = 12.2 minutes, t_R (minor) = 13.4 minutes.



(**S**)
Table 2, entry 19

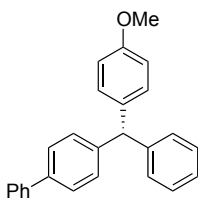
(**S**)-**Table 2, entry 18**. Using representative procedure A outlined above, the following amounts of reagents were used: 1,8-bis(1,5-cyclooctadiene)nickel (5.5 mg, 0.020 mmol, 0.10 equiv), tricyclohexylphosphine (11 mg, 0.040 mmol, 0.20 equiv), potassium *tert*-butoxide (45 mg, 0.40 mmol, 2.0 equiv), 1-butanol (54 μ L, 0.60 mmol, 3.0 equiv), 5-(5,5-dimethyl-1,3,2-dioxaborinan-2-yl)-1-methyl-1*H*-indole (97 mg, 0.40 mmol, 2.0 equiv), (**S**)-**1** (66 mg, 0.20 mmol, 1.0 equiv), tetrahydrofuran (1 mL) and toluene (1 mL). The product was purified by flash column chromatography (5–20% Et₂O/hexane, 0.5% TEA) to afford the product as a white solid. First run: (63.4 mg, 0.182 mmol, 91%, 92% ee). Second run: (61.4 mg, 0.178 mmol, 89%, 93% ee). **TLC** R_f = 0.3 (20% Et₂O/hexane); **m.p.** = 49–52 °C; **¹H NMR** (500 MHz, CDCl₃) δ 7.80–7.75 (m, 1H), 7.73 (d, J = 8.6 Hz, 1H), 7.70–7.64 (m, 1H), 7.50 (s, 1H), 7.40 (dt, J = 9.3, 4.9 Hz, 2H), 7.37–7.31 (m, 2H), 7.27 (t, J = 7.3 Hz, 2H), 7.25–7.16 (m, 4H), 7.07 (dd, J = 8.7, 1.0 Hz, 1H), 6.98 (d, J = 2.9 Hz, 1H), 6.36 (d, J = 2.9 Hz, 1H), 5.83 (s, 1H), 3.72 (s, 3H); **¹³C NMR** (125 MHz, CDCl₃) δ 144.7, 142.6, 135.6, 134.9, 133.5, 132.2, 129.8, 129.2, 128.55, 128.52, 128.4, 128.0, 127.9, 127.8, 127.6, 126.3, 126.0, 125.6, 123.9, 121.7, 109.2, 101.1, 57.1, 33.0; **IR** (neat) 3022, 2884, 1599, 1489 cm⁻¹; **HRMS** (TOF MS ES⁺) m/z calcd for C₂₆H₂₁N (M + Na)⁺ 370.1572, found 370.1576; $[\alpha]_D^{23}$ –10.8 (c 1.00, CHCl₃); **SFC** analysis (AD-H, 20% MeOH, 2.5 mL/min) indicated 93% ee: t_R (major) = 8.1 minutes, t_R (minor) = 9.0 minutes.

Single crystals suitable for X-ray crystallographic analysis were grown by slow diffusion of hexane into a solution of (**S**)-**Table 2, entry 19** in benzene at 4 °C. See Section VIII for crystallographic data.



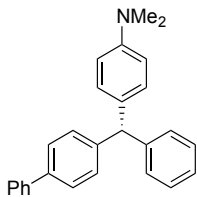
(R)
Table 2, entry 20

(R)-Table 2, entry 19. Using representative procedure B outlined above, the following amounts of reagents were used: 1,8-bis(1,5-cyclooctadiene)nickel (5.5 mg, 0.020 mmol, 0.10 equiv), 1,3-bis(2,4,6-trimethylphenyl)-4,5-dihydroimidazolium tetrafluoroborate (8.3 mg, 0.021 mmol, 0.11 equiv), potassium *tert*-butoxide (45 mg, 0.40 mmol, 2.0 equiv), 1-butanol (54 μ L, 0.60 mmol, 3.0 equiv), 5-(5,5-dimethyl-1,3,2-dioxaborinan-2-yl)-1-methyl-1*H*-indole (97 mg, 0.40 mmol, 2.0 equiv), **(S)-1** (66 mg, 0.20 mmol, 1.0 equiv), tetrahydrofuran (1 mL) and toluene (1 mL). The product was purified by flash column chromatography (5–20% Et₂O/hexanes, 0.5% TEA) to afford the product as a white solid. First run: (41.5 mg, 0.119 mmol, 57%, 92% ee). Second run: (57.0 mg, 0.164 mmol, 82%, 92% ee). Analytical data is consistent with the values listed above for **(S)-Table 2, entry 12**. $[\alpha]_D^{23} +6.0$ (*c* 0.9, CHCl₃); **SFC** analysis (AD-H, 20% MeOH, 2.5 mL/min) indicated 93% ee: *t_R* (major) = 8.9 minutes, *t_R* (minor) = 8.1 minutes.



(R)
Table 3, entry 1

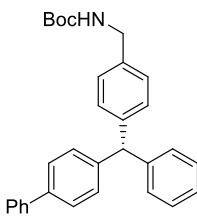
(R)-Table 3, entry 1. Using representative procedure B above, the following amounts of reagents were used: 1,8-bis(1,5-cyclooctadiene)nickel (5.5 mg, 0.020 mmol, 0.10 equiv), 1,3-bis(2,4,6-trimethylphenyl)-4,5-dihydroimidazolium tetrafluoroborate (8.3 mg, 0.0210 mmol, 0.11 equiv), potassium *tert*-butoxide (45 mg, 0.40 mmol, 2.0 equiv), 1-butanol (54 μ L, 0.60 mmol, 3.0 equiv), 5,5-dimethyl-2-(4-methoxyphenyl)-1,3,2-dioxaborinane (88 mg, 0.40 mmol, 2.0 equiv), **(S)-SI-6** (69 mg, 0.20 mmol, 1.0 equiv), tetrahydrofuran (1 mL) and toluene (1 mL). The product was purified by flash column chromatography (1–3% Et₂O/hexanes) to afford the product as a colorless oil. First run: (54.8 mg, 0.156 mmol, 78%, 81% ee). Second run: (55.8 mg, 0.159 mmol, 80%, 81% ee). **TLC** *R_f* = 0.4 (10% Et₂O/Hexanes); **¹H NMR** (500 MHz, CDCl₃) δ 7.56 (d, *J* = 7.2, 2H), 7.51 (d, *J* = 7.1, 2H), 7.42 (t, *J* = 7.1, 2H), 7.29 (m, 3H), 7.19 (m, 5H), 7.06 (d, *J* = 7.8, 2H), 6.83 (d, *J* = 8.2, 2H) 5.53 (s, 1H), 3.78 (s, 3H); **¹³C NMR** (125 MHz, CDCl₃) δ 158.2, 144.3, 143.5, 141.0, 139.2, 136.2, 130.5, 129.9, 129.5, 128.9, 128.5, 128.3, 127.15, 127.14, 126.4, 113.9, 55.9, 55.4; **IR** (neat) 3020, 2996, 1508, 1244, 1030 cm⁻¹; **HRMS** (TOF MS ES⁺) *m/z* calcd for C₂₆H₂₂O (M + Na)⁺ 350.1671, found 367.1679; $[\alpha]_D^{23} +1.2$ (*c* 1.01, CHCl₃), **SFC** analysis (AD-H, 10% MeOH, 2.5 mL/min) indicated 84% ee: *t_R* (minor) = 21.5 minutes, *t_R* (major) = 19.8 minutes.



(R)

Table 3, entry 2

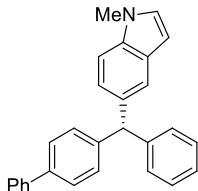
(R)-Table 3, entry 2. Using representative procedure B above, the following amounts and reagents: 1,8-bis(1,5-cyclooctadiene)nickel (8.3 mg, 0.030 mmol, 0.10 equiv), 1,3-bis(2,4,6-trimethylphenyl)-4,5-dihydroimidazolium tetrafluoroborate (12 mg, 0.030 mmol, 0.10 equiv), potassium *tert*-butoxide (64 mg, 0.60 mmol, 2.0 equiv), 4-(5,5-dimethyl-1,3,2-dioxaborinan-2-yl)-*N,N*-dimethylaniline (134 mg, 0.600 mmol, 2.00 equiv), **(S)-SI-6** (103 mg, 0.300 mmol, 1.00 equiv) and 1-butanol (54 μ L, 0.90 mmol, 3.00 equiv). Purified by flash column chromatography (0–10% Et₂O/hexanes) to afford **(R)-8**, as a light yellow oil (55 mg, 0.15 mmol, 75%). **TLC** R_f = 0.3 (10% Et₂O/Hexanes); **¹H NMR** (500 MHz, CDCl₃) δ 7.57 (d, J = 7.2, 2H), 7.49 (d, J = 8.2, 2H), 7.41 (t, J = 7.7, 2H), 7.29 (m, 3H), 7.27 (s, 1H) 7.21 (m, 5H), 7.02 (dd, J = 8.6, 2H) 6.67 (d, J = 8.9, 2H), 6.4 (d, J = 2.8, 1H), 5.5 (s, 1H), 2.9 (s, 3H); **¹³C NMR** (125 MHz, CDCl₃) δ 149.2, 144.7, 143.9, 141.1, 139.0, 131.9, 130.2, 129.9, 129.5, 128.8, 128.4, 127.20, 127.15, 127.1, 126.3, 112.7, 55.7, 40.8; **IR** (neat) 3024, 2841, 2360, 1613, 1485, 1347, 763 cm⁻¹; **HRMS** (TOF MS EI+) m/z calcd for C₂₇H₂₅O (M + Na)⁺ 364.2065, found 364.2061; $[\alpha]_D^{23}$ -2.9 (c 1.07, CHCl₃); **SFC** analysis (AD-H, 16% MeOH, 3.0 mL/min) indicated 79% ee: t_R (minor) = 26.3 minutes, t_R (major) = 11.6 minutes.



(S)

Table 3, entry 3

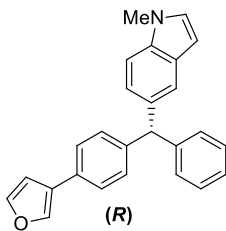
tert-butyl 4-([1,1'-biphenyl]-4-yl(phenyl)methyl)benzylcarbamate (10). Using representative procedure B outlined above, the following amounts of reagents were used: 1,8-bis(1,5-cyclooctadiene)nickel (5.5 mg, 0.020 mmol, 0.10 equiv), 1,3-bis(2,4,6-trimethylphenyl)-4,5-dihydroimidazolium tetrafluoroborate (8.67 mg, 0.022 mmol, 0.11 equiv), potassium *tert*-butoxide (45 mg, 0.4 mmol, 2.0 equiv), *tert*-butyl 4-(5,5-dimethyl-1,3,2-dioxaborinan-2-yl)benzylcarbamate (127.3 mg, 0.200 mmol, 2.00 equiv), **S-SI-6** (68.9 mg, 0.200 mmol, 1.00 equiv) and 1-butanol (54 μ L, 0.60 mmol, 3.0 equiv). Purified by flash column chromatography (0–15 % EtOAc/Hexane) to afford the desired triarylmethane as a clear colorless oil (48.5 mg, 54%); **TLC** R_f = 0.1 (9:1 Hexanes:EtOAc); **¹H NMR** (500 MHz, CDCl₃) δ 7.57 (dd, J = 1.5, J = 8.4, 2H), 7.51 (d, J = 8.3, 2H), 7.39 (t, J = 7.5, 2H), 7.29 (m, 3H), 7.25 (m, 3H), 7.15 (m, 6H), 5.56 (s, 1H), 4.82 (s, 1H), 4.30 (d, J = 5.3, 1H); **¹³C NMR** (125 MHz, CDCl₃) δ 156.0, 143.9, 143.13, 143.05, 140.9, 139.3, 137.1, 129.9, 129.8, 129.5, 128.9, 128.5, 127.6, 127.3, 127.16, 127.14, 126.5, 56.3, 44.3, 28.5; **IR** (neat) 3294, 3028, 1695, 1486, 1316, 1016, 757 cm⁻¹; **HRMS** (TOF MS EI+) m/z calcd for C₃₁H₃₁NO₂ [M+Na]⁺ 472.2253, found 472.2261. $[\alpha]_D^{29}$ -8.2 **SFC** analysis (AS-H, 20% MeOH, 2.5 mL/min) indicated 92% ee: t_R (major) = 6.86 minutes, t_R (minor) = 7.47 minutes.



(R)

Table 3, entry 4

(R)-Table 3, entry 3. Using representative procedure B outlined above, the following amounts of reagents were used: 1,8-bis(1,5-cyclooctadiene)nickel (5.5 mg, 0.020 mmol, 0.10 equiv), 1,3-bis(2,4,6-trimethylphenyl)-4,5-dihydroimidazolium tetrafluoroborate (8.27 mg, 0.0210 mmol, 0.105 equiv), potassium *tert*-butoxide (45 mg, 0.40 mmol, 2.0 equiv), 1-butanol (54 μ L, 0.60 mmol, 3.0 equiv), 5-(5,5-dimethyl-1,3,2-dioxaborinan-2-yl)-1-methyl-1*H*-indole (97 mg, 0.40 mmol, 2.0 equiv), **(S)-SI-6** (69 mg, 0.20 mmol, 1.0 equiv), tetrahydrofuran (1 mL) and toluene (1 mL). The product was purified by flash column chromatography (5–20% Et₂O/hexane, 0.5% TEA) to afford the product as a white solid. First run: (61.0 mg, 0.163 mmol, 82%, 96% ee). Second run: (37.8 mg, 0.101 mmol, 51%, 96% ee). **TLC** R_f = 0.3 (10% EtOAc/hexanes); **m.p.** = 54–55 °C; **¹H NMR** (400 MHz, CDCl₃) δ 7.56 (d, J = 7.4, 2H), 7.49 (d, J = 8.2, 2H), 7.37 (t, J = 7.4, 2H), 7.27 (m, 3H), 7.26 (s, 1H) 7.22 (m, 9H), 7.07 (dd, J = 1.3, 8.6, 1H) 6.87 (d, J = 3.0, 1H), 6.37 (d, J = 2.8, 1H), 5.70 (s, 1H), 3.73 (s, 3H); **¹³C NMR** (100 MHz, CDCl₃) δ 144.9, 144.2, 141.1, 138.9, 135.6, 135.1, 130.1, 129.7, 129.2, 128.8, 128.6, 128.4, 127.17, 127.15, 127.0, 126.2, 123.8, 121.6, 109.2, 101.1, 56.7, 32.9; **IR** (neat) 3025, 2360, 1486, 1449, 1246, 1006, 760 cm^{-1} ; **HRMS** submitted; **$[\alpha]_D^{23}$** –5.2; **SFC** analysis (AD-H, 25% MeOH, 2.5 mL/min) indicated 96% ee: t_R (major) = 11.3 minutes, t_R (minor) = 16.1 minutes.

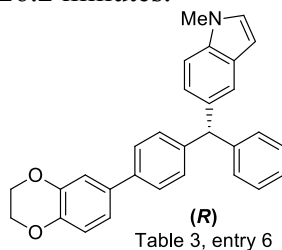


(R)

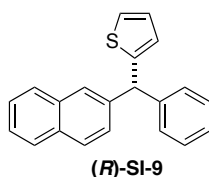
Table 3, entry 5

(R)-Table 3, entry 4. Using representative procedure B outlined above, the following amounts of reagents were used: 1,8-bis(1,5-cyclooctadiene)nickel (5.5 mg, 0.020 mmol, 0.10 equiv), 1,3-Bis(2,4,6-trimethylphenyl)-4,5-dihydroimidazolium tetrafluoroborate (8.27 mg, 0.0210 mmol, 0.105 equiv), potassium *tert*-butoxide (45 mg, 0.40 mmol, 2.0 equiv), 1-butanol (54 μ L, 0.60 mmol, 3.0 equiv), 5-(5,5-dimethyl-1,3,2-dioxaborinan-2-yl)-1-methyl-1*H*-indole (97 mg, 0.40 mmol, 2.0 equiv), **(S)-SI-7** (67 mg, 0.20 mmol, 1.0 equiv), tetrahydrofuran (1 mL) and toluene (1 mL). The product was purified twice by flash column chromatography (6% Et₂O/hexane and then 60% benzene/pentane) to afford the desired triarylmethane as a white solid. First run: (58.5 mg, 0.161 mmol, 80%, 87% ee). Second run: (58.5 mg, 0.161 mmol, 80%, 87% ee). **TLC** R_f = 0.6 (40% pentane/benzene); **m.p.** = 149–151 °C; **¹H NMR** (500 MHz, CDCl₃) δ 7.59 (s, 1H), 7.34 (s, 1H), 7.29 (d, J = 8.3 Hz, 2H), 7.23 (s, 1H), 7.18 (t, J = 7.4 Hz, 2H), 7.15–7.09 (m, 2H), 7.07 (t, J = 7.4 Hz, 4H), 6.95 (d, J = 8.6 Hz, 1H), 6.90 (d, J = 2.8 Hz, 1H), 6.57 (s, 1H), 6.29 (d, J = 2.8 Hz, 1H), 5.58 (s, 1H), 3.63 (s, 3H); **¹³C NMR** (125 MHz, CDCl₃) δ 144.9, 143.9, 141.7, 138.5, 135.6, 135.0, 130.3, 130.1, 129.7, 129.2, 128.5, 128.3, 126.4, 126.2, 125.8, 123.7, 121.5,

109.2, 109.0, 101.1, 56.7, 33.0; **IR** (neat) 3145, 3024, 1599, 1492 cm^{-1} ; $[\alpha]_D^{23}$ -3.1 (c 2.24, CHCl_3); **HRMS** submitted; **SFC** analysis (OJ-H, 30% MeOH, 3.0 mL/min) indicated 87% ee: t_R (major) = 23.6 minutes, t_R (minor) = 26.2 minutes.



(R)-Table 3, entry 5. Using representative procedure B outlined above, the following amounts of reagents were used: 1,8-bis(1,5-cyclooctadiene)nickel (5.5 mg, 0.020 mmol, 0.10 equiv), 1,3-bis(2,4,6-trimethylphenyl)-4,5-dihydroimidazolium tetrafluoroborate (8.27 mg, 0.0210 mmol, 0.105 equiv), potassium *tert*-butoxide (45 mg, 0.40 mmol, 2.0 equiv), 1-butanol (54 μL , 0.60 mmol, 3.0 equiv), 5-(5,5-dimethyl-1,3,2-dioxaborinan-2-yl)-1-methyl-1*H*-indole (97 mg, 0.40 mmol, 2.0 equiv), **(S)-SI-8** (67 mg, 0.20 mmol, 1.0 equiv), tetrahydrofuran (1 mL) and toluene (1 mL). The product was purified twice by flash column chromatography (6% EtOAc/hexanes and then 70% benzene/pentane) to afford the desired triarylmethane as a white solid. First run: (51.4 mg, 0.119 mmol, 60%, 93% ee). Second run: (52.0 mg, 0.121 mmol, 60%, 93% ee). **TLC** R_f = 0.3 (40% pentane/benzene); **m.p.** = 81–84 $^\circ\text{C}$; **$^1\text{H NMR}$** (500 MHz, CDCl_3) δ 7.43 (s, J = 8.0 2H), 7.33 (s, 1H), 7.29 (t, J = 7.4 Hz, 2H), 7.24 (s, 1H), 7.22–7.14 (m, 5H), 7.10 (s, 1H), 7.06 (dt, J = 8.2, 2.0 Hz, 2H), 7.01 (d, J = 2.7 Hz, 1H), 6.90 (d, J = 8.2 Hz, 1H), 6.39 (d, J = 2.5 Hz, 1H), 5.69 (s, 1H), 4.27 (s, 4H), 3.76 (s, 3H); **$^{13}\text{C NMR}$** (125 MHz, CDCl_3) δ 144.9, 143.8, 143.7, 143.1, 138.4, 135.6, 135.1, 134.8, 130.0, 129.7, 129.2, 128.6, 128.4, 126.6, 126.2, 123.8, 121.6, 120.2, 117.6, 115.8, 109.2, 101.1, 64.58, 64.56, 56.7, 33.0; **IR** (neat) 2916, 1586, 1513, 1449 cm^{-1} ; **HRMS** (TOF MS ES+) m/z calcd for $\text{C}_{30}\text{H}_{25}\text{O}_2\text{N}$ ($\text{M} + \text{Na}$) $^+$ 454.1783, found 454.1772; $[\alpha]_D^{23}$ -8.2 (c 2.36, CHCl_3); **SFC** analysis (OD-H, 25% IPA, 2.5 mL/min) indicated 93% ee: t_R (major) = 23.6 minutes, t_R (minor) = 26.2 minutes.



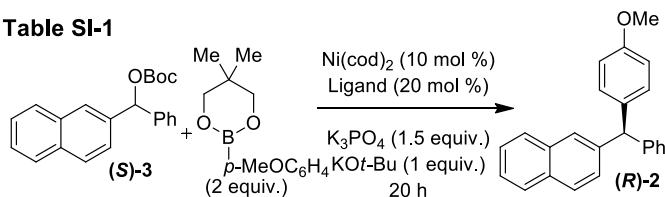
(R)-SI-9. Prepared according to general procedure B using the following amounts and reagents: $\text{Ni}(\text{cod})_2$ (2.8 mg, 0.010 mmol, 0.10 equiv), 1,3-bis(2,4,6-trimethylphenyl)-4,5-dihydroimidazolium tetrafluoroborate (3.9 mg, 0.010 mmol, 0.10 equiv), potassium *tert*-butoxide (22 mg, 0.20 mmol, 2.0 equiv), 5,5-dimethyl-2-(thiophen-2-yl)-1,3,2-dioxaborinane (39.2 mg, 0.200 mmol, 2.00 equiv), **(S)-1** (33.1 mg, 0.100 mmol, 1.00 equiv) and 1-butanol (27 μL , 0.30 mmol, 3.0 equiv). Purified by flash column chromatography (0–5% Et_2O /hexanes) to afford the desired triarylmethane as a yellow solid (11.4 mg, 0.0379 mmol, 38%). Analytical data is consistent with literature values.³ **$^1\text{H NMR}$** (400 MHz, CDCl_3) δ 7.84–7.72 (m, 3H), 7.61 (s, 1H), 7.37–7.41 (m, 2H), 7.37 (dd, J = 1.6, 8.5), 7.34–7.20 (m, 6H), 6.95 (t, J = 4.3), 6.73 (d, J = 3.3), 5.84 (s, 1H); **$^{13}\text{C NMR}$** δ (125 MHz, CDCl_3) δ 147.8, 143.7, 141.4, 133.5, 132.4, 129.1,

128.6, 128.2, 128.1, 127.7, 127.5, 127.4, 126.9, 126.8, 126.7, 126.2, 125.9, 124.8, 52.3 [α]_D²⁵ – 9.3 (*c* 0.57, CHCl₃).

VI. Tables of results using alternative ligands and bases.

Other ligands and bases were tested under reaction conditions similar to Table 1. Representative examples are shown below.

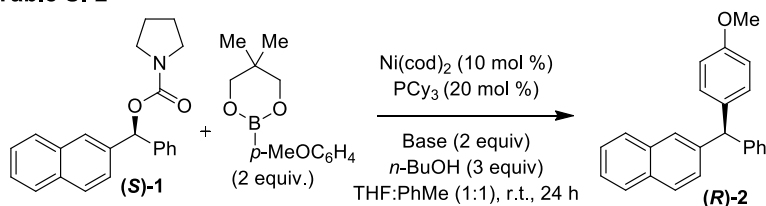
Table SI-1



Entry	ligand	yield ^a
1	DPEphos (Bis[(2-diphenylphosphino)phenyl] ether)	< 5%
2	Cy-DPEphos (Bis[(2-dicyclohexylphosphino)phenyl] ether)	< 5%
3	DPPO (1,8-bis(diphenylphosphino)octane)	13%
4	PPh ₃ (triphenylphosphine)	22%
5	P(<i>t</i> -Bu) ₃ tri- <i>tert</i> -butylphosphine	< 5%
6	XPhos (2-Dicyclohexylphosphino-2',4',6'-triisopropylbiphenyl)	< 5%
7	SPhos (2-Dicyclohexylphosphino-2',6'-dimethoxybiphenyl)	< 5%
8	SIPr-HBF ₄ (1,3-Bis(2,6-diisopropylphenyl)-4,5-dihydroimidazolium tetrafluoroborate)	31%
9	tricyclohexylphosphine	86%
10	PCy ₃ tricyclohexylphosphine (11 mol %)	83%
11	SIMes-HBF ₄ 1,3-bis(2,4,6-trimethylphenyl)-4,5-dihydroimidazolium tetrafluoroborate	84%
12	None	< 5%

^aDetermined by ¹H NMR analysis using an internal standard (PhSiMe₃).

Table SI-2



Entry	Base	yield (%) ^a	SM ee (%) ^b	product ee (%) ^b	es (%)
1	LiOt-Bu	8	98	53	54
2	NaOt-Bu	75	94	93	99
3	KOt-Bu	87	93	92	99

^bIsolated yield after chromatography. ^cDetermined by chiral SFC chromatography.

VII. References and Notes

- ¹ Balma Tivola, P.; Deagostino, A.; Prandi, C.; Venturello, P. *Org. Lett.* **2002**, *4*, 1275.
- ² (a) Yamamoto, Y.; Kurihara, K.; Miyaura, N. *Angew. Chem. Int. Ed.* **2009**, *48*, 4414. (b) Shannon, J.; Bernier, D.; Rawson, D.; Woodward, S. *Chem. Commun.* **2007**, 3945. (c) Tjosaas, F.; Anthonsen, T.; Jacobsen, E. E. *ARKIVOC* **2008**, (6), 8190.
- ³ Taylor, B. L. H.; Harris, M. R.; Jarvo, E. R. *Angew. Chem. Int. Ed.* **2012**, *51*, 7790.
- ⁴ Braga, A. R.; Paixao, M. W.; Westeman, B.; Schneider, P. H.; Wessjohan, L.A. *J. Org. Chem.* **2008**, *73*, 2879.
- ⁵ Wu, X.; Liu, X.; Zhao, G. *Tetrahedron: Asymmetry* **2005**, *16*, 2299.
- ⁶ Kudo, N.; Perseghini, M.; Fu, G. C. *Angew. Chem. Int. Ed.* **2006**, *45*, 1282.
- ⁷ DeKorver, K. A.; Johnson, W. L.; Zhang, Y.; Hsung, R. P.; Dai, H.; Deng, J.; Lohse, A. G.; Zhang, Y. S. *J. Org. Chem.* **2011**, *76*, 5092.
- ⁸ Basel, Y.; Hassner, A. *J. Org. Chem.* **2000**, *65*, 6368.

VIII. Crystallographic Data

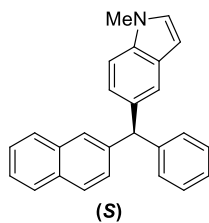


Table 2, entry 19

X-ray Data Collection, Structure Solution and Refinement for (S)-Table 2, entry 19.

A colorless crystal of approximate dimensions 0.22 x 0.28 x 0.33 mm was mounted on a glass fiber and transferred to a Nonius FR-591 rotating-anode system with Bruker APEX detector (Montels Optics). The APEX2¹ program package was used to determine the unit-cell parameters and for data collection (2.0 sec/frame scan time). The raw frame data was processed using SAINT² and SADABS³ to yield the reflection data file. Subsequent calculations were carried out using the SHELXTL⁴ program. The diffraction symmetry was $2/m$ and the systematic absences were consistent with the monoclinic space groups $P2_1$ and $P2_1/m$. It was later determined that space group $P2_1$ was correct.

The structure was solved by direct methods and refined on F^2 by full-matrix least-squares techniques. The analytical scattering factors⁵ for neutral atoms were used throughout the analysis. Hydrogen atoms were located from a difference-Fourier map and refined (x, y, z and U_{iso}).

At convergence, $wR2 = 0.0770$ and $Goof = 1.043$ for 328 variables refined against 3454 data (0.82\AA), $R1 = 0.0297$ for those 3412 data with $I > 2.0\sigma(I)$. The absolute structure was assigned according to the methods of Parsons and Flack⁶⁻⁷.

References.

1. APEX2 Version 2012.4-0, Bruker AXS, Inc.; Madison, WI 2012.
2. SAINT Version 7.68a, Bruker AXS, Inc.; Madison, WI 2009.
3. Sheldrick, G. M. SADABS, Version 2008/1, Bruker AXS, Inc.; Madison, WI 2008.
4. Sheldrick, G. M. SHELXTL, Version 2012/9, 2012.
5. International Tables for X-Ray Crystallography 1992, Vol. C., Dordrecht: Kluwer Academic Publishers.
6. Flack, H. D. Acta. Cryst., A39, 876-881, 1983.
7. Parsons, S., Flack, H. D. Acta. Cryst., A60, s61, 2004.

Definitions:

$$wR2 = [\Sigma[w(F_o^2 - F_c^2)^2] / \Sigma[w(F_o^2)^2]]^{1/2}$$

$$R1 = \Sigma||F_o| - |F_c|| / \Sigma|F_o|$$

Goof = S = $[\Sigma[w(F_o^2 - F_c^2)^2] / (n-p)]^{1/2}$ where n is the number of reflections and p is the total number of parameters refined.

The thermal ellipsoid plot is shown at the 50% probability level.

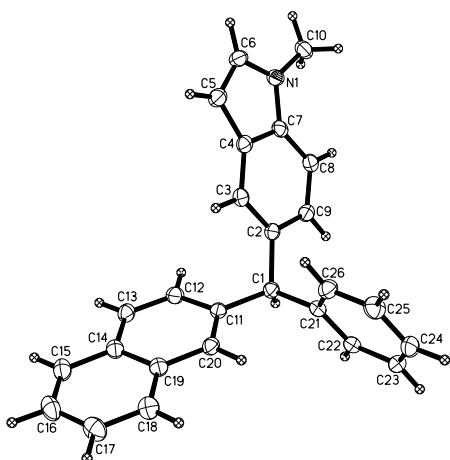


Table 1. Crystal data and structure refinement for erj10.

Identification code	erj10 (Michael Harris)	
Empirical formula	$C_{26} H_{21} N$	
Formula weight	347.44	
Temperature	173(2) K	
Wavelength	1.54178 Å	
Crystal system	Monoclinic	
Space group	$P2_1$	
Unit cell dimensions	$a = 8.6116(2)$ Å	$\alpha = 90^\circ$.
	$b = 11.5924(2)$ Å	$\beta = 100.1452(7)^\circ$.
	$c = 9.6267(2)$ Å	$\gamma = 90^\circ$.
Volume	$946.00(3)$ Å ³	
Z	2	
Density (calculated)	1.220 Mg/m ³	
Absorption coefficient	0.534 mm ⁻¹	
F(000)	368	
Crystal color	colorless	
Crystal size	$0.331 \times 0.280 \times 0.218$ mm ³	
Theta range for data collection	4.666 to 69.774°	
Index ranges	$-9 \leq h \leq 10, -14 \leq k \leq 14, -11 \leq l \leq 11$	
Reflections collected	24745	
Independent reflections	3454 [R(int) = 0.0343]	
Completeness to theta = 67.679°	99.4 %	
Absorption correction	Semi-empirical from equivalents	

Refinement method	Full-matrix least-squares on F ²
Data / restraints / parameters	3454 / 1 / 328
Goodness-of-fit on F ²	1.043
Final R indices [I>2sigma(I) = 3412 data]	R1 = 0.0297, wR2 = 0.0765
R indices (all data, 0.82Å)	R1 = 0.0302, wR2 = 0.0770
Absolute structure parameter	-0.10(16)
Largest diff. peak and hole	0.157 and -0.146 e.Å ⁻³

Table 2. Atomic coordinates ($\times 10^4$) and equivalent isotropic displacement parameters ($\text{\AA}^2 \times 10^3$) for erj10. $U(\text{eq})$ is defined as one third of the trace of the orthogonalized U^{ij} tensor.

	x	y	z	$U(\text{eq})$
N(1)	13910(2)	6748(1)	9901(2)	23(1)
C(1)	9548(2)	10396(1)	8488(2)	19(1)
C(2)	10723(2)	9410(1)	8861(2)	19(1)
C(3)	10709(2)	8428(2)	8045(2)	20(1)
C(4)	11837(2)	7559(2)	8465(2)	20(1)
C(5)	12142(2)	6453(2)	7916(2)	25(1)
C(6)	13398(2)	6000(2)	8817(2)	26(1)
C(7)	12970(2)	7715(2)	9709(2)	21(1)
C(8)	13009(2)	8707(2)	10529(2)	22(1)
C(9)	11880(2)	9540(2)	10092(2)	21(1)
C(10)	15216(2)	6573(2)	11064(2)	28(1)
C(11)	7966(2)	10000(1)	7634(2)	19(1)
C(12)	7165(2)	9073(2)	8182(2)	22(1)
C(13)	5735(2)	8692(2)	7493(2)	24(1)
C(14)	4987(2)	9213(2)	6213(2)	22(1)
C(15)	3499(2)	8845(2)	5466(2)	27(1)
C(16)	2814(2)	9384(2)	4243(2)	31(1)
C(17)	3574(2)	10329(2)	3729(2)	31(1)
C(18)	5014(2)	10701(2)	4422(2)	26(1)
C(19)	5763(2)	10152(2)	5674(2)	21(1)
C(20)	7255(2)	10522(2)	6409(2)	21(1)
C(21)	10306(2)	11392(1)	7807(2)	20(1)
C(22)	10357(2)	12484(2)	8399(2)	21(1)
C(23)	11043(2)	13406(2)	7801(2)	25(1)
C(24)	11687(2)	13242(2)	6596(2)	28(1)
C(25)	11653(2)	12143(2)	5993(2)	29(1)
C(26)	10969(2)	11228(2)	6599(2)	24(1)

Table 3. Bond lengths [Å] and angles [°] for erj10.

N(1)-C(6)	1.369(3)
N(1)-C(7)	1.376(2)
N(1)-C(10)	1.455(2)
C(1)-C(2)	1.527(2)
C(1)-C(21)	1.529(2)
C(1)-C(11)	1.532(2)
C(1)-H(1A)	0.96(2)
C(2)-C(3)	1.382(2)
C(2)-C(9)	1.416(2)
C(3)-C(4)	1.409(2)
C(3)-H(3A)	0.96(2)
C(4)-C(7)	1.417(2)
C(4)-C(5)	1.428(2)
C(5)-C(6)	1.367(3)
C(5)-H(5A)	0.96(3)
C(6)-H(6A)	0.95(3)
C(7)-C(8)	1.392(3)
C(8)-C(9)	1.382(2)
C(8)-H(8A)	0.95(2)
C(9)-H(9A)	0.95(3)
C(10)-H(10A)	0.96(3)
C(10)-H(10B)	0.95(3)
C(10)-H(10C)	0.94(3)
C(11)-C(20)	1.370(2)
C(11)-C(12)	1.427(2)
C(12)-C(13)	1.365(3)
C(12)-H(12A)	1.00(3)
C(13)-C(14)	1.421(3)
C(13)-H(13A)	0.96(3)
C(14)-C(15)	1.420(3)
C(14)-C(19)	1.421(2)
C(15)-C(16)	1.370(3)
C(15)-H(15A)	0.97(3)
C(16)-C(17)	1.410(3)

C(16)-H(16A)	0.99(3)
C(17)-C(18)	1.371(3)
C(17)-H(17A)	0.97(3)
C(18)-C(19)	1.414(3)
C(18)-H(18A)	0.94(3)
C(19)-C(20)	1.420(2)
C(20)-H(20A)	0.97(3)
C(21)-C(22)	1.386(2)
C(21)-C(26)	1.396(3)
C(22)-C(23)	1.394(3)
C(22)-H(22A)	0.95(3)
C(23)-C(24)	1.384(3)
C(23)-H(23A)	0.95(3)
C(24)-C(25)	1.398(3)
C(24)-H(24A)	0.95(3)
C(25)-C(26)	1.390(3)
C(25)-H(25A)	0.94(3)
C(26)-H(26A)	0.94(3)
C(6)-N(1)-C(7)	108.16(15)
C(6)-N(1)-C(10)	126.79(15)
C(7)-N(1)-C(10)	125.05(16)
C(2)-C(1)-C(21)	110.53(14)
C(2)-C(1)-C(11)	113.04(14)
C(21)-C(1)-C(11)	113.75(14)
C(2)-C(1)-H(1A)	105.5(14)
C(21)-C(1)-H(1A)	108.4(14)
C(11)-C(1)-H(1A)	105.1(13)
C(3)-C(2)-C(9)	119.84(15)
C(3)-C(2)-C(1)	122.97(15)
C(9)-C(2)-C(1)	117.18(15)
C(2)-C(3)-C(4)	119.34(15)
C(2)-C(3)-H(3A)	123.2(15)
C(4)-C(3)-H(3A)	117.5(15)
C(3)-C(4)-C(7)	119.20(15)
C(3)-C(4)-C(5)	134.29(16)

C(7)-C(4)-C(5)	106.50(15)
C(6)-C(5)-C(4)	106.67(16)
C(6)-C(5)-H(5A)	126.2(17)
C(4)-C(5)-H(5A)	127.1(17)
C(5)-C(6)-N(1)	110.61(16)
C(5)-C(6)-H(6A)	130.1(16)
N(1)-C(6)-H(6A)	119.3(16)
N(1)-C(7)-C(8)	129.92(16)
N(1)-C(7)-C(4)	108.06(15)
C(8)-C(7)-C(4)	122.02(16)
C(9)-C(8)-C(7)	117.27(15)
C(9)-C(8)-H(8A)	121.0(14)
C(7)-C(8)-H(8A)	121.6(14)
C(8)-C(9)-C(2)	122.32(16)
C(8)-C(9)-H(9A)	119.6(14)
C(2)-C(9)-H(9A)	118.1(14)
N(1)-C(10)-H(10A)	109.4(18)
N(1)-C(10)-H(10B)	110.4(18)
H(10A)-C(10)-H(10B)	107(3)
N(1)-C(10)-H(10C)	111.3(17)
H(10A)-C(10)-H(10C)	110(2)
H(10B)-C(10)-H(10C)	109(2)
C(20)-C(11)-C(12)	118.63(16)
C(20)-C(11)-C(1)	123.31(16)
C(12)-C(11)-C(1)	118.00(15)
C(13)-C(12)-C(11)	121.12(16)
C(13)-C(12)-H(12A)	120.4(15)
C(11)-C(12)-H(12A)	118.5(15)
C(12)-C(13)-C(14)	121.11(16)
C(12)-C(13)-H(13A)	119.9(14)
C(14)-C(13)-H(13A)	118.9(14)
C(15)-C(14)-C(13)	122.84(17)
C(15)-C(14)-C(19)	119.03(17)
C(13)-C(14)-C(19)	118.12(16)
C(16)-C(15)-C(14)	120.77(19)
C(16)-C(15)-H(15A)	120.4(14)

C(14)-C(15)-H(15A)	118.8(14)
C(15)-C(16)-C(17)	119.96(18)
C(15)-C(16)-H(16A)	120.1(17)
C(17)-C(16)-H(16A)	120.0(17)
C(18)-C(17)-C(16)	120.64(19)
C(18)-C(17)-H(17A)	119.3(16)
C(16)-C(17)-H(17A)	120.0(16)
C(17)-C(18)-C(19)	120.72(19)
C(17)-C(18)-H(18A)	118.5(16)
C(19)-C(18)-H(18A)	120.8(16)
C(18)-C(19)-C(20)	121.68(17)
C(18)-C(19)-C(14)	118.84(17)
C(20)-C(19)-C(14)	119.47(16)
C(11)-C(20)-C(19)	121.53(16)
C(11)-C(20)-H(20A)	121.3(14)
C(19)-C(20)-H(20A)	117.1(14)
C(22)-C(21)-C(26)	118.62(16)
C(22)-C(21)-C(1)	119.87(15)
C(26)-C(21)-C(1)	121.51(15)
C(21)-C(22)-C(23)	121.02(16)
C(21)-C(22)-H(22A)	118.7(16)
C(23)-C(22)-H(22A)	120.3(16)
C(24)-C(23)-C(22)	120.19(17)
C(24)-C(23)-H(23A)	120.3(15)
C(22)-C(23)-H(23A)	119.5(15)
C(23)-C(24)-C(25)	119.38(17)
C(23)-C(24)-H(24A)	117.7(17)
C(25)-C(24)-H(24A)	122.8(17)
C(26)-C(25)-C(24)	120.04(17)
C(26)-C(25)-H(25A)	121.9(19)
C(24)-C(25)-H(25A)	118.0(19)
C(25)-C(26)-C(21)	120.75(17)
C(25)-C(26)-H(26A)	117.8(16)
C(21)-C(26)-H(26A)	121.4(16)

Table 4. Anisotropic displacement parameters ($\text{\AA}^2 \times 10^3$) for erj10. The anisotropic displacement factor exponent takes the form: $-2\pi^2 [h^2 a^{*2} U^{11} + \dots + 2 h k a^* b^* U^{12}]$

	U ¹¹	U ²²	U ³³	U ²³	U ¹³	U ¹²
N(1)	19(1)	21(1)	30(1)	4(1)	7(1)	2(1)
C(1)	19(1)	18(1)	20(1)	-1(1)	4(1)	0(1)
C(2)	18(1)	18(1)	22(1)	2(1)	5(1)	-2(1)
C(3)	19(1)	22(1)	21(1)	1(1)	4(1)	-2(1)
C(4)	20(1)	19(1)	21(1)	1(1)	7(1)	-2(1)
C(5)	26(1)	21(1)	28(1)	-2(1)	7(1)	-1(1)
C(6)	26(1)	17(1)	35(1)	0(1)	10(1)	2(1)
C(7)	17(1)	21(1)	25(1)	5(1)	7(1)	-1(1)
C(8)	19(1)	24(1)	23(1)	2(1)	3(1)	-3(1)
C(9)	22(1)	19(1)	22(1)	-1(1)	5(1)	-2(1)
C(10)	19(1)	29(1)	36(1)	6(1)	4(1)	1(1)
C(11)	17(1)	17(1)	23(1)	-2(1)	6(1)	0(1)
C(12)	21(1)	21(1)	25(1)	2(1)	5(1)	1(1)
C(13)	22(1)	21(1)	30(1)	-1(1)	10(1)	-3(1)
C(14)	19(1)	24(1)	26(1)	-7(1)	7(1)	-1(1)
C(15)	22(1)	30(1)	31(1)	-9(1)	8(1)	-4(1)
C(16)	23(1)	41(1)	30(1)	-13(1)	2(1)	-3(1)
C(17)	30(1)	41(1)	21(1)	-5(1)	-1(1)	2(1)
C(18)	28(1)	30(1)	22(1)	-2(1)	5(1)	-2(1)
C(19)	21(1)	22(1)	22(1)	-5(1)	6(1)	1(1)
C(20)	21(1)	18(1)	24(1)	-2(1)	7(1)	-1(1)
C(21)	16(1)	20(1)	23(1)	1(1)	1(1)	1(1)
C(22)	16(1)	22(1)	24(1)	0(1)	3(1)	2(1)
C(23)	22(1)	18(1)	34(1)	1(1)	1(1)	0(1)
C(24)	23(1)	25(1)	35(1)	8(1)	5(1)	-3(1)
C(25)	26(1)	32(1)	29(1)	3(1)	11(1)	-1(1)
C(26)	26(1)	21(1)	28(1)	-2(1)	7(1)	0(1)

Table 5. Hydrogen coordinates ($\times 10^4$) and isotropic displacement parameters ($\text{\AA}^2 \times 10^{-3}$) for erj10.

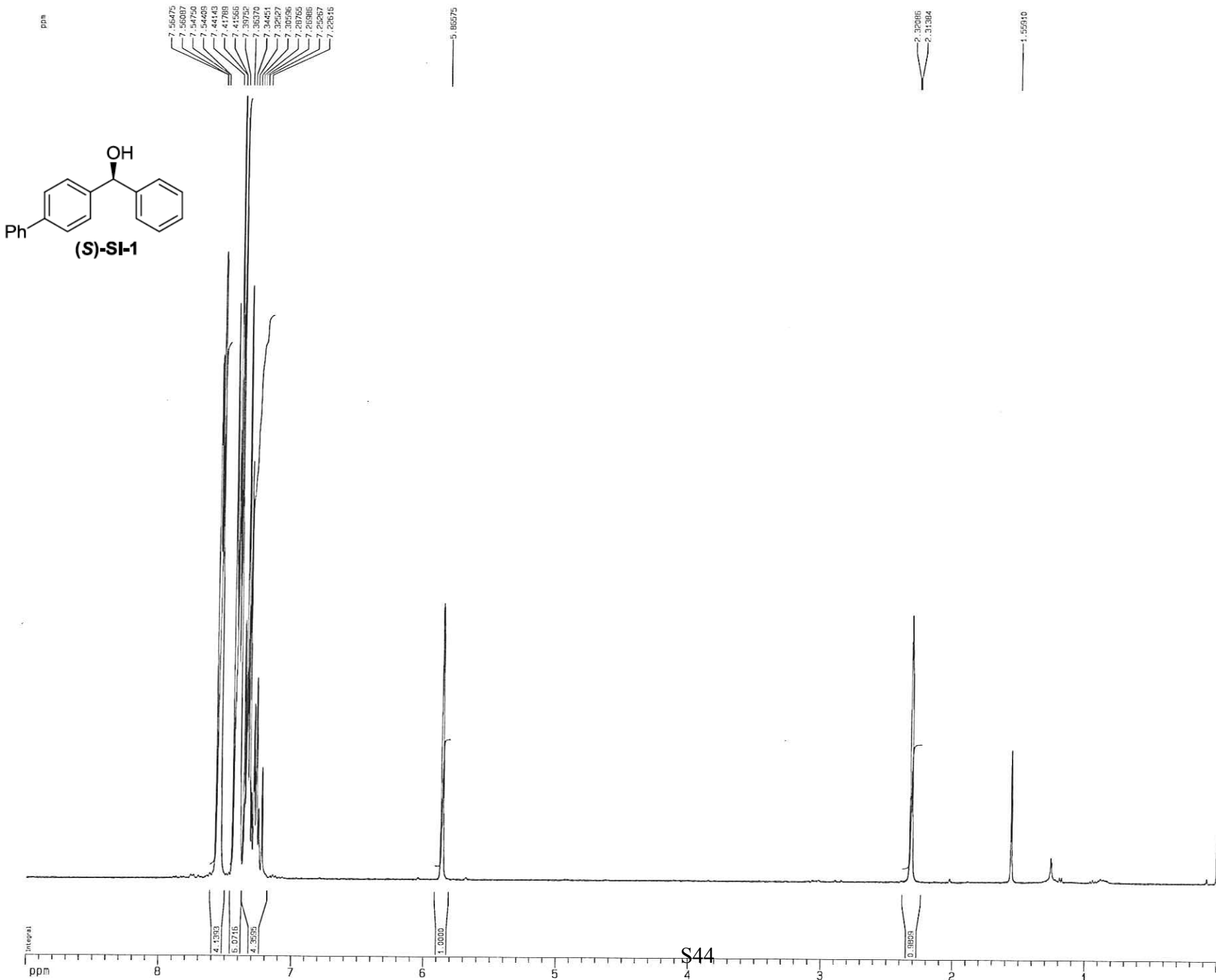
	x	y	z	U(eq)
H(1A)	9310(30)	10660(20)	9370(20)	18(5)
H(3A)	9970(30)	8310(20)	7190(30)	23(5)
H(5A)	11600(30)	6100(20)	7070(30)	35(7)
H(6A)	13920(30)	5280(20)	8790(30)	31(6)
H(8A)	13750(30)	8800(20)	11380(20)	20(5)
H(9A)	11860(30)	10220(20)	10630(20)	22(5)
H(10A)	15640(40)	5810(30)	11000(30)	45(8)
H(10B)	16050(40)	7100(30)	11000(30)	43(7)
H(10C)	14890(30)	6670(20)	11940(30)	33(6)
H(12A)	7670(30)	8710(20)	9100(30)	27(6)
H(13A)	5210(30)	8080(20)	7880(30)	25(6)
H(15A)	2970(30)	8210(20)	5840(20)	24(5)
H(16A)	1800(40)	9100(20)	3710(30)	40(7)
H(17A)	3100(30)	10700(20)	2850(30)	32(6)
H(18A)	5490(30)	11330(20)	4050(30)	31(6)
H(20A)	7740(30)	11170(20)	6020(20)	24(5)
H(22A)	9900(30)	12600(20)	9230(30)	30(6)
H(23A)	11050(30)	14150(20)	8220(30)	27(6)
H(24A)	12190(30)	13880(30)	6240(30)	37(7)
H(25A)	12100(30)	12050(30)	5180(30)	42(7)
H(26A)	10990(30)	10500(20)	6180(30)	31(6)

Table 6. Torsion angles [°] for erj10.

C(21)-C(1)-C(2)-C(3)	-100.94(18)
C(11)-C(1)-C(2)-C(3)	27.9(2)
C(21)-C(1)-C(2)-C(9)	78.04(18)
C(11)-C(1)-C(2)-C(9)	-153.15(15)
C(9)-C(2)-C(3)-C(4)	1.0(2)
C(1)-C(2)-C(3)-C(4)	179.91(15)
C(2)-C(3)-C(4)-C(7)	-0.5(2)
C(2)-C(3)-C(4)-C(5)	178.32(17)
C(3)-C(4)-C(5)-C(6)	-178.75(18)
C(7)-C(4)-C(5)-C(6)	0.14(19)
C(4)-C(5)-C(6)-N(1)	-0.1(2)
C(7)-N(1)-C(6)-C(5)	0.1(2)
C(10)-N(1)-C(6)-C(5)	-179.95(16)
C(6)-N(1)-C(7)-C(8)	179.34(18)
C(10)-N(1)-C(7)-C(8)	-0.6(3)
C(6)-N(1)-C(7)-C(4)	0.03(18)
C(10)-N(1)-C(7)-C(4)	-179.96(15)
C(3)-C(4)-C(7)-N(1)	178.98(14)
C(5)-C(4)-C(7)-N(1)	-0.11(18)
C(3)-C(4)-C(7)-C(8)	-0.4(2)
C(5)-C(4)-C(7)-C(8)	-179.48(15)
N(1)-C(7)-C(8)-C(9)	-178.51(16)
C(4)-C(7)-C(8)-C(9)	0.7(2)
C(7)-C(8)-C(9)-C(2)	-0.2(3)
C(3)-C(2)-C(9)-C(8)	-0.6(2)
C(1)-C(2)-C(9)-C(8)	-179.65(16)
C(2)-C(1)-C(11)-C(20)	-130.10(16)
C(21)-C(1)-C(11)-C(20)	-3.0(2)
C(2)-C(1)-C(11)-C(12)	52.8(2)
C(21)-C(1)-C(11)-C(12)	179.91(14)
C(20)-C(11)-C(12)-C(13)	1.3(2)
C(1)-C(11)-C(12)-C(13)	178.56(16)
C(11)-C(12)-C(13)-C(14)	-0.6(3)
C(12)-C(13)-C(14)-C(15)	-179.86(17)

C(12)-C(13)-C(14)-C(19)	-0.6(2)
C(13)-C(14)-C(15)-C(16)	179.19(17)
C(19)-C(14)-C(15)-C(16)	-0.1(3)
C(14)-C(15)-C(16)-C(17)	-1.2(3)
C(15)-C(16)-C(17)-C(18)	1.6(3)
C(16)-C(17)-C(18)-C(19)	-0.6(3)
C(17)-C(18)-C(19)-C(20)	-179.99(17)
C(17)-C(18)-C(19)-C(14)	-0.7(3)
C(15)-C(14)-C(19)-C(18)	1.0(2)
C(13)-C(14)-C(19)-C(18)	-178.26(16)
C(15)-C(14)-C(19)-C(20)	-179.66(15)
C(13)-C(14)-C(19)-C(20)	1.0(2)
C(12)-C(11)-C(20)-C(19)	-0.8(2)
C(1)-C(11)-C(20)-C(19)	-177.94(15)
C(18)-C(19)-C(20)-C(11)	178.96(16)
C(14)-C(19)-C(20)-C(11)	-0.3(2)
C(2)-C(1)-C(21)-C(22)	-122.88(16)
C(11)-C(1)-C(21)-C(22)	108.69(17)
C(2)-C(1)-C(21)-C(26)	56.5(2)
C(11)-C(1)-C(21)-C(26)	-71.9(2)
C(26)-C(21)-C(22)-C(23)	0.5(2)
C(1)-C(21)-C(22)-C(23)	179.93(15)
C(21)-C(22)-C(23)-C(24)	0.0(3)
C(22)-C(23)-C(24)-C(25)	-0.4(3)
C(23)-C(24)-C(25)-C(26)	0.3(3)
C(24)-C(25)-C(26)-C(21)	0.3(3)
C(22)-C(21)-C(26)-C(25)	-0.7(3)
C(1)-C(21)-C(26)-C(25)	179.94(17)

1H spectrum



Current Data Parameters
 USER Inanna
 NAME LEH-1-77-c1-140-2
 EXPNO 1
 PROCNO 1

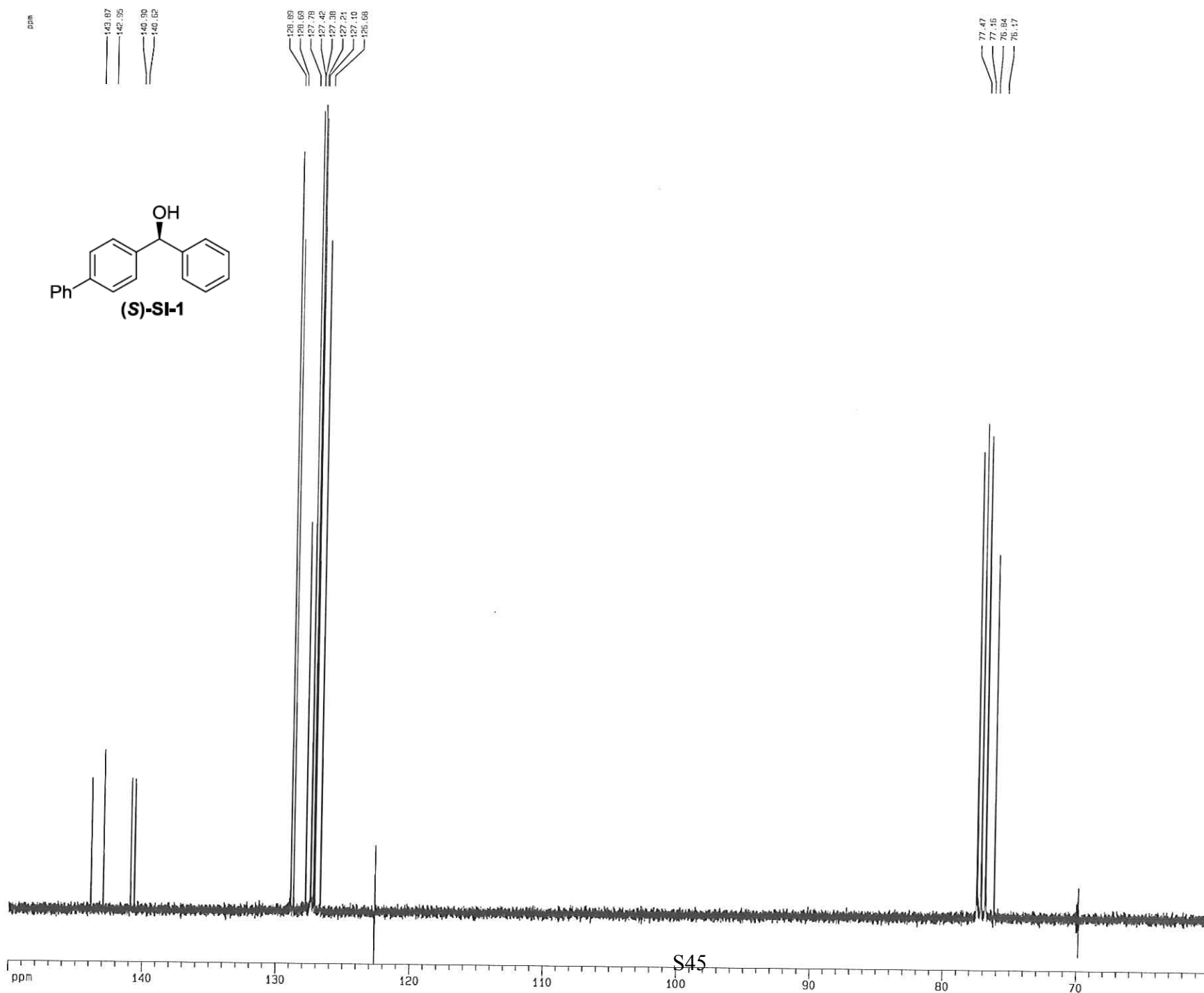
F2 - Acquisition Parameters
 Date_ 20121101
 Time 10.41
 INSTRUM d-x400
 PROBHD 5 mm QNP HFF/P
 PULPROG zg30
 TD 65536
 SOLVENT CDCl3
 NS 8
 DS 2
 SWH 6410.256 Hz
 FIDRES 0.027813 Hz
 AQ 5.1118579 sec
 RG 143.7
 DM 78.000 usec
 DE 4.50 usec
 TE 298.0 K
 D1 0.10000000 sec
 MCREST 0.00000000 sec
 MCWRC 0.01500000 sec

***** CHANNEL f1 *****
 NUC1 1H
 P1 12.00 usec
 PL1 -0.60 dB
 SFO1 400.1328009 MHz

F2 - Processing parameters
 SI 65536
 SF 400.1300345 MHz
 WDW rd
 SSB 0
 LB 0.00 Hz
 GB 0
 PC 2.00

1D NMR plot parameters
 CX 22.60 cm
 CY 15.00 cm
 F1 9.000 ppm
 F1 3501.17 Hz
 F2 0.000 ppm
 F2 0.00 Hz
 PRMCM 0.33474 ppm/cm
 HZCM 157.94508 Hz/cm

¹³C spectrum with ¹H decoupling



Current Data Parameters
 USER Ihanna
 NAME LEH-1-77-C13
 EX-NO 1
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20121101
 Time 11.10
 INSTRUM drx400
 PROBNM 5 mm QNP H/F/P
 PULPROG zgpg30
 TD 65536
 SOLVENT CDCl3
 NS 1024
 DS 4
 SWH 24154.590 Hz
 FIDRES 0.358970 Hz
 AQ 1.3556452 sec
 RG 14595.5
 DM 20.700 usec
 DE 20.39 usec
 TE 299.0 K
 D1 0.10000000 sec
 d11 0.03000000 sec
 MCREST 0.00000000 sec
 MCWRK 0.01500000 sec

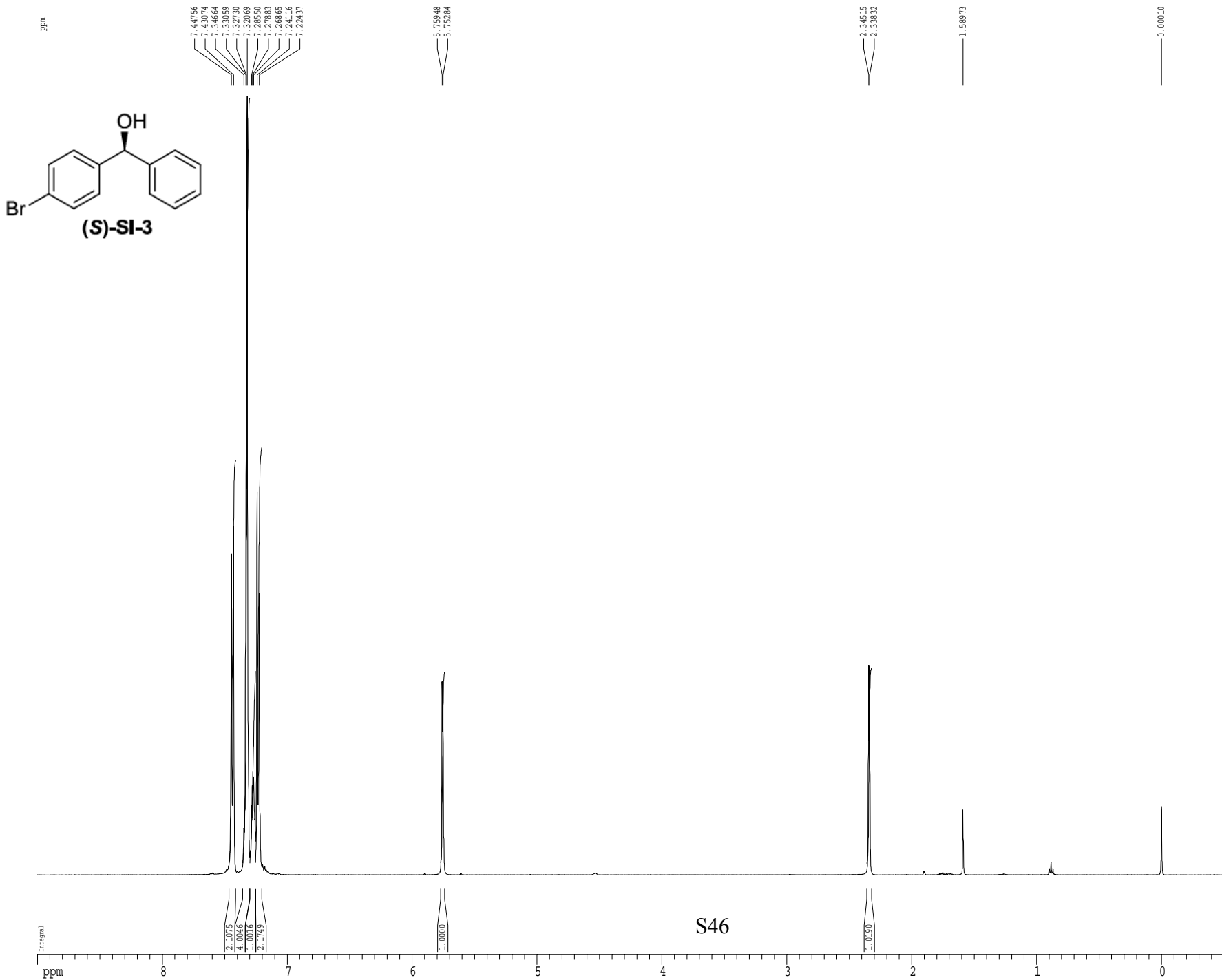
***** CHANNEL f1 *****
 NUC1 ¹³C
 P1 11.00 usec
 PL1 0.00 dB
 SF01 100.6237954 MHz

***** CHANNEL f2 *****
 CPDPRG2 mlev16
 NUC2 ¹H
 PCPD2 80.00 usec
 PL2 0.00 dB
 PL12 16.20 dB
 SF02 400.1328009 MHz

F2 - Processing parameters
 SI 65536
 SF 100.6127632 MHz
 WDW no
 SSB 0
 LB 0.00 Hz
 GB 0
 PC 1.00

1D NMR plot parameters
 CX 22.80 cm
 CY 15.50 cm
 F1P 150.000 ppm
 F1 15091.91 Hz
 F2P 60.000 ppm
 F2 6035.77 Hz
 PPMCM 3.94737 ppm/cm
 HZCM 397.15564 Hz/cm

¹H spectrum



Current Data Parameters
 USER mharr
 NAME MRH-IV-124-1HNMR
 EXPNO 1
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20121117
 Time 18.32
 INSTRUM cryo500
 PROBHD 5 mm CPTCI 1H-
 PULPROG zg30
 TD 81728
 SOLVENT CDCl3
 NS 1
 DS 2
 SWH 8012.820 Hz
 FIDRES 0.098043 Hz
 AQ 5.0998774 sec
 RG 5
 DW 62.400 usec
 DE 6.00 usec
 TE 298.0 K
 DL 0.10000000 sec
 MCREST 0.00000000 sec
 MCWEX 0.01500000 sec

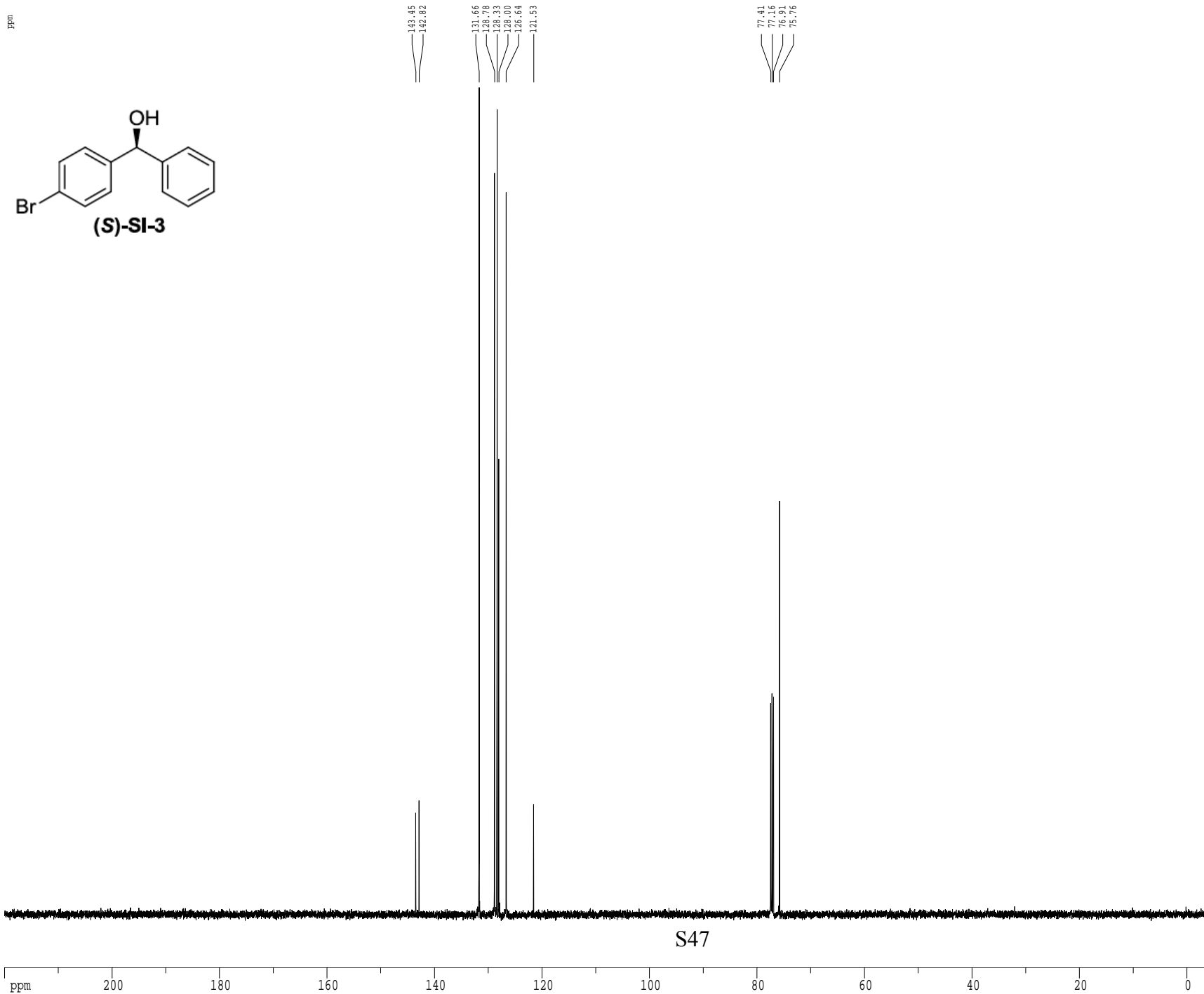
===== CHANNEL f1 =====
 NUC1 1H
 P1 7.50 usec
 PL1 1.60 dB
 SF01 500.2235015 MHz

F2 - Processing parameters
 SI 65536
 SF 500.2200406 MHz
 WDW EM
 SSB 0
 LB 0.30 Hz
 GB 0
 PC 4.00

1D NMR plot parameters
 CX 22.80 cm
 CY 15.00 cm
 F1P 9.000 ppm
 F1 4501.98 Hz
 F2P -0.500 ppm
 F2 -250.11 Hz
 PPMCM 0.41667 ppm/cm
 HZCM 208.42502 Hz/cm

S46

Z-restored spin-echo 13C spectrum with 1H decoupling



Current Data Parameters
 USER mharri
 NAME MRH-IV-124-13CNMR
 EXPNO 1
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20121117
 Time 18.36
 INSTRUM cryo500
 PROBHD 5 mm CPTCI 1H-
 PULPROG SpinEchopg30gp.prd
 TD 65536
 SOLVENT CDCl3T
 NS 126
 DS 16
 SWH 30303.031 Hz
 FIDRES 0.462388 Hz
 AQ 1.0813940 sec
 RG 2580.3
 DW 16.500 usec
 DE 6.00 usec
 TE 298.0 K
 D1 0.25000000 sec
 d11 0.03000000 sec
 D16 0.00020000 sec
 d17 0.00019600 sec
 MCREST 0.00000000 sec
 MCWRK 0.01500000 sec
 P2 31.00 usec

***** CHANNEL f1 *****
 NUC1 13c
 P1 15.50 usec
 P11 500.00 usec
 P12 2000.00 usec
 PL0 120.00 dB
 PL1 -1.00 dB
 SFO1 125.7942548 MHz
 SF1 3.20 dB
 SF2 3.20 dB
 SPNAM1 Crp60,0.5,20.1
 SPNAM2 Crp60comp,4
 SPOFF1 0.00 Hz
 SPOFF2 0.00 Hz

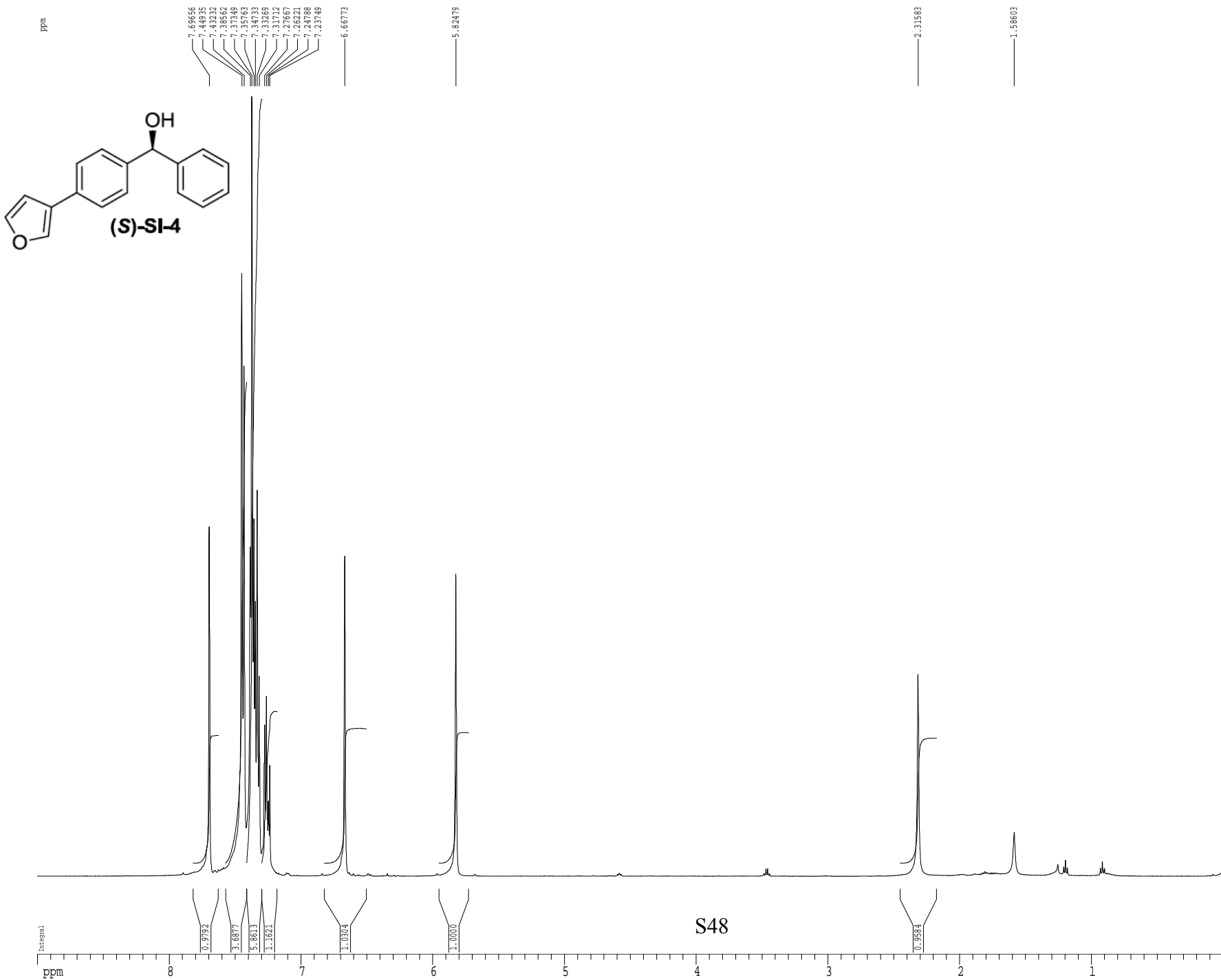
***** CHANNEL f2 *****
 CPDPRG2 waltz16
 NUC2 1H
 PCPD2 100.00 usec
 PL2 1.60 dB
 PL12 24.60 dB
 SFO2 500.2225011 MHz

***** GRADIENT CHANNEL *****
 GENAM1 SINE.100
 GENAM2 SINE.100
 GFX1 0.00 %
 GFX2 0.00 %
 GPY1 0.00 %
 GPY2 0.00 %
 GPZ1 30.00 %
 GPZ2 50.00 %
 p15 500.00 usec
 p16 1000.00 usec

F2 - Processing parameters
 SI 65536
 SF 125.7804131 MHz
 WM HM
 SSB 0
 LB 1.00 Hz
 GB 0
 PC 2.00

1D NMR plot parameters
 CX 22.80 cm
 CY 15.65 cm
 F1P 220.000 ppm
 F1 27671.69 Hz
 F2P -5.000 ppm
 F2 -628.90 Hz
 PFMCM 9.86842 ppm/cm
 HZCM 1241.25415 Hz/cm

¹H spectrum



Current Data Parameters
 USER: greene
 NAME: mrh-4-133
 EXPNO: 1
 PROCNO: 1

F2 - Acquisition Parameters
 Date_: 20121117
 Time: 10.00
 INSTRUM: cryo500
 PROBHD: 5 mm CPTCI 1H-
 PULPROG: zg30
 TD: 81728
 SOLVENT: CDCl3
 NS: 8
 DS: 2
 SWH: 8012.820 Hz
 FIDRES: 0.098043 Hz
 AQ: 5.0999398 sec
 RG: 5.7
 DW: 62.400 usec
 DE: 6.00 usec
 TE: 298.0 K
 DL: 0.10000000 sec
 MCREST: 0.00000000 sec
 MCWRR: 0.01500000 sec

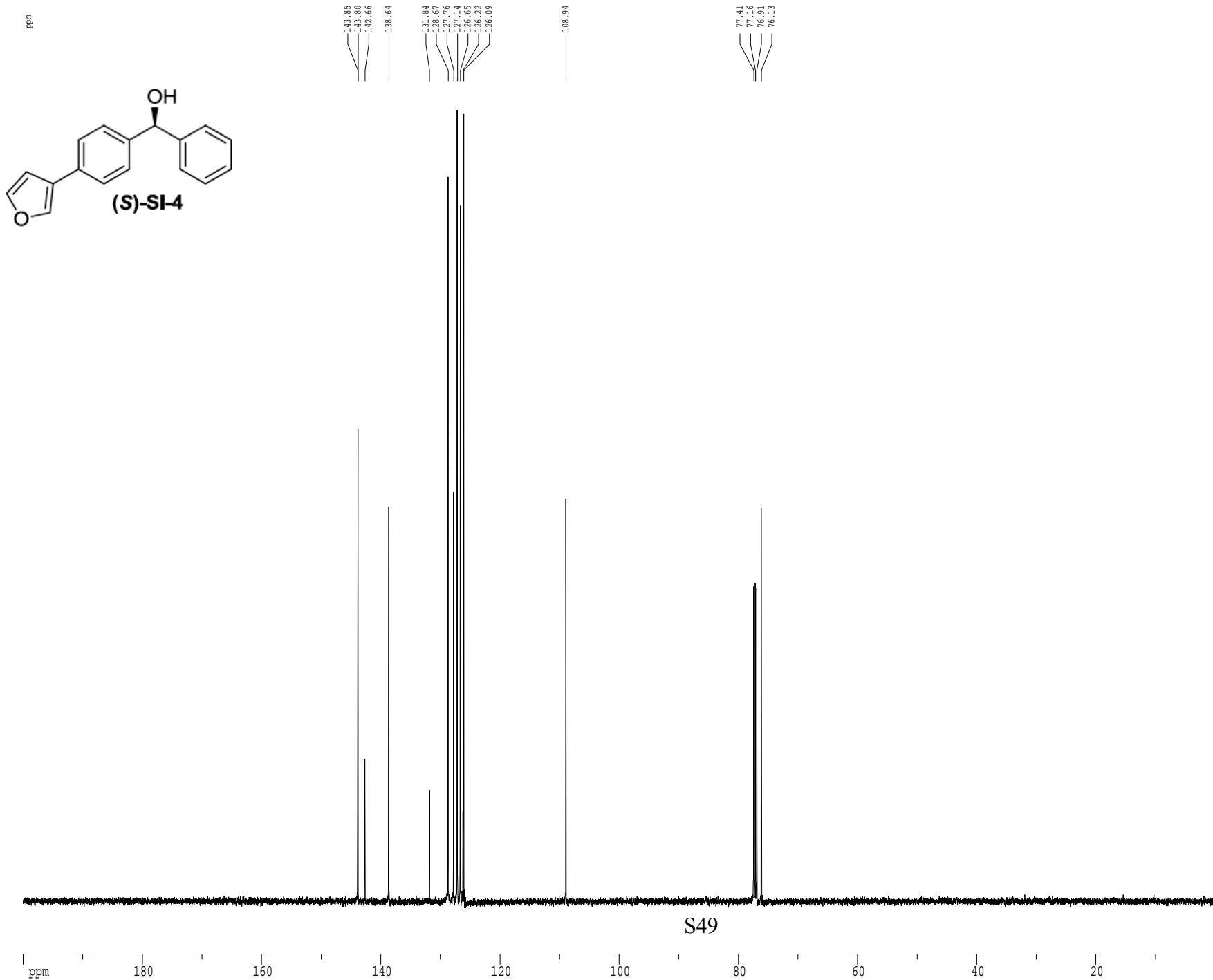
===== CHANNEL f1 =====
 NUC1: 1H
 P1: 7.50 usec
 PL1: 1.60 dB
 SF01: 500.2235015 MHz

F2 - Processing parameters
 SI: 65536
 SF: 500.2200421 MHz
 WDW: EM
 SSB: 0
 LB: 0.30 Hz
 GB: 0
 PC: 4.00

1D NMR plot parameters
 CX: 22.80 cm
 CY: 15.00 cm
 F1P: 9.000 ppm
 F1: 4501.98 Hz
 F2P: 0.000 ppm
 F2: 0.00 Hz
 PPMCM: 0.39474 ppm/cm
 HZCM: 197.45528 Hz/cm

S48

Z-restored spin-echo 13C spectrum with 1H decoupling



```

Current Data Parameters
USER      greene
NAME      mrh-4-133
EXPNO     2
PROCNO    1

F2 - Acquisition Parameters
Date_     20121117
Time      10.03
INSTRUM   cryo500
PROBHD    5 mm CPTCI 1H-
PULPROG   SpinEchopg30gp.prd
TD         65536
SOLVENT   CDCl3
NS         295
DS         16
SWH        30303.031 Hz
FIDRES     0.462388 Hz
AQ         1.0814105 sec
RG         9195.2
DW         16.500 usec
DE         6.00 usec
TE         298.0 K
D1         0.25000000 sec
d11        0.03000000 sec
D16        0.00020000 sec
d17        0.00019600 sec
MCREST     0.00000000 sec
MCMRXX     0.01500000 sec
P2         31.00 usec

===== CHANNEL f1 =====
NUC1       13c
P1         15.50 usec
P11        500.00 usec
P12        2000.00 usec
PL0        120.00 dB
PL1        -1.00 dB
SFO1       125.7942548 MHz
SF1         3.20 dB
SFO2       Crp60,0.5,20.1
SFO3       Crp60comp,4
SFOFF1     0.00 Hz
SFOFF2     0.00 Hz

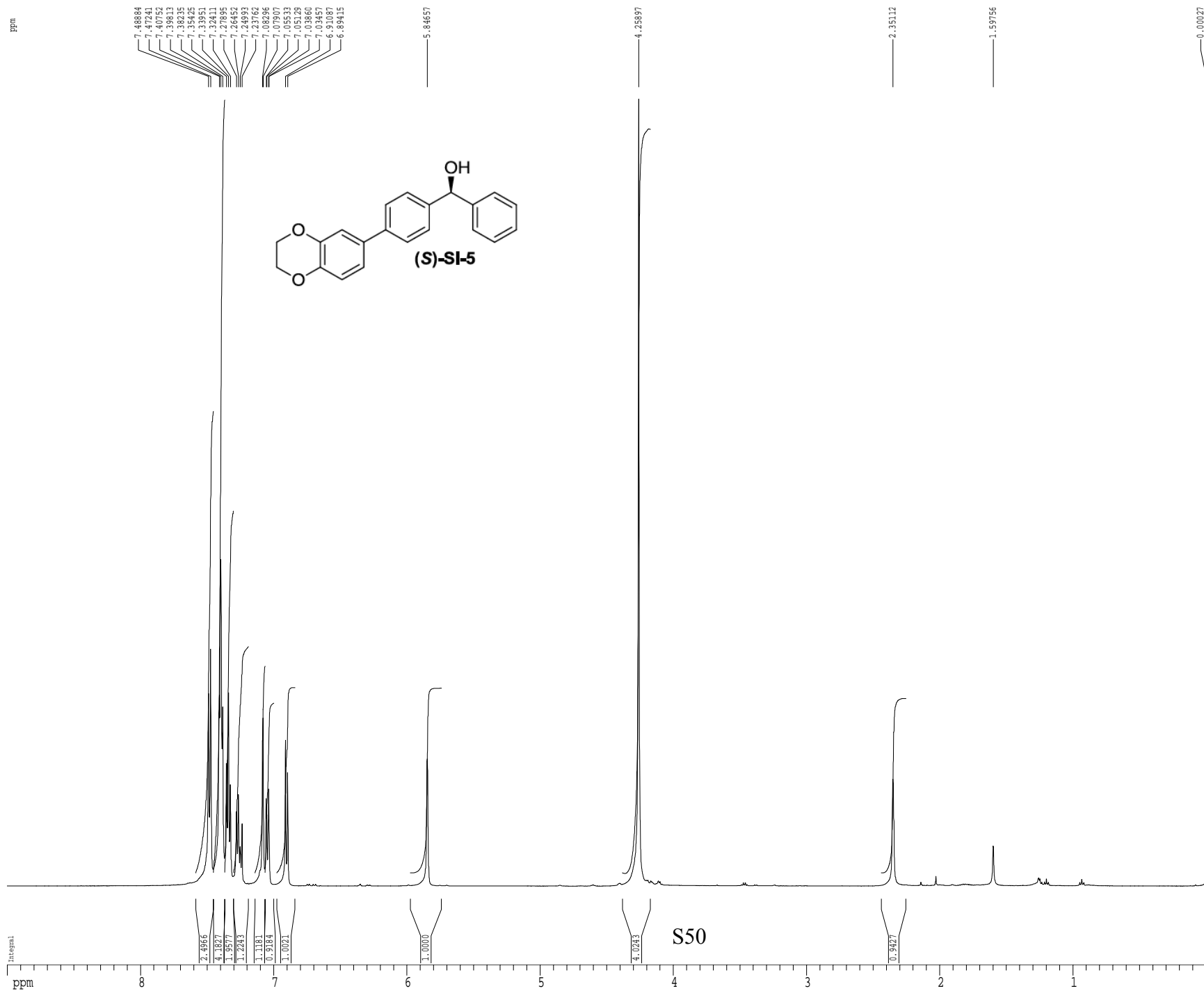
===== CHANNEL f2 =====
CPDPRG2    waltz16
NUC2       1H
PCPD2      100.00 usec
PL2        1.60 dB
PL12       24.60 dB
SFO2       500.2225011 MHz

===== GRADIENT CHANNEL =====
GENAM1     SINE.100
GENAM2     SINE.100
GFX1       0.00 %
GFX2       0.00 %
GPY1       0.00 %
GPY2       0.00 %
GPZ1       30.00 %
GPZ2       50.00 %
p15        500.00 usec
p16        1000.00 usec

F2 - Processing parameters
SI         65536
SF         125.7804127 MHz
WDW        HM
SSB        0
LB         1.00 Hz
GB         0
PC         2.00

1D NMR plot parameters
CX         22.80 cm
CY         15.65 cm
F1P        200.000 ppm
F1         25156.08 Hz
F2P        0.000 ppm
F2         0.00 Hz
PFMCM      8.77193 ppm/cm
HZCM       1103.33691 Hz/cm
    
```

¹H spectrum



Current Data Parameters
 USER: greene
 NAME: mrh-4-138
 EXPNO: 1
 PROCNO: 1

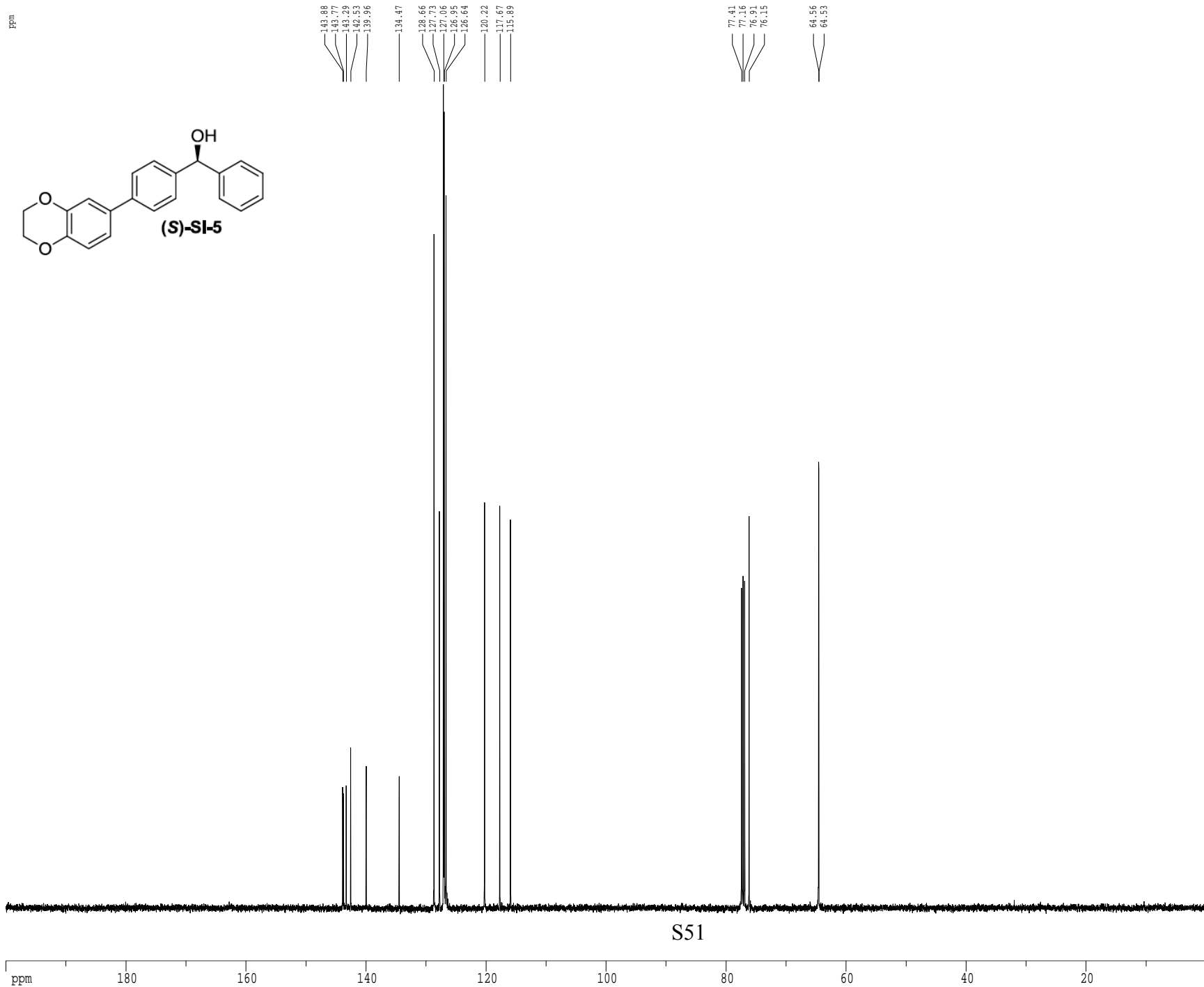
F2 - Acquisition Parameters
 Date_: 20121117
 Time: 9.35
 INSTRUM: cryo500
 PROBHD: 5 mm CPTCI 1H-
 PULPROG: zg30
 TD: 81728
 SOLVENT: CDCl3
 NS: 8
 DS: 2
 SWH: 8012.820 Hz
 FIDRES: 0.098043 Hz
 AQ: 5.0999398 sec
 RG: 6.3
 DW: 62.400 usec
 DE: 6.00 usec
 TE: 298.0 K
 DL: 0.10000000 sec
 MCREST: 0.00000000 sec
 MCWRX: 0.01500000 sec

===== CHANNEL f1 =====
 NUC1: 1H
 P1: 7.50 usec
 PL1: 1.60 dB
 SF01: 500.2235015 MHz

F2 - Processing parameters
 SI: 65536
 SF: 500.2200418 MHz
 WDW: EM
 SSB: 0
 LB: 0.30 Hz
 GB: 0
 PC: 4.00

1D NMR plot parameters
 CX: 22.80 cm
 CY: 15.00 cm
 F1P: 9.000 ppm
 F1: 4501.98 Hz
 F2P: 0.000 ppm
 F2: 0.00 Hz
 PPMCM: 0.39474 ppm/cm
 HZCM: 197.45528 Hz/cm

Z-restored spin-echo 13C spectrum with 1H decoupling



```

Current Data Parameters
USER      greene
NAME      mrh-4-138
EXPNO     2
PROCNO    1

F2 - Acquisition Parameters
Date_     20121117
Time      9.40
INSTRUM   cryo500
PROBHD    5 mm CPTCI 1H-
PULPROG   SpinEchopg30gp.prd
TD         65536
SOLVENT   CDCl3
NS         298
DS         16
SWH        30303.031 Hz
FIDRES     0.462388 Hz
AQ         1.0814105 sec
RG         5792.6
DW         16.500 usec
DE         6.00 usec
TE         298.0 K
d1         0.25000000 sec
d11        0.03000000 sec
d16        0.00020000 sec
d17        0.00019600 sec
MCREST     0.00000000 sec
MCMRXX     0.01500000 sec
P2         31.00 usec

===== CHANNEL f1 =====
NUC1       13c
P1         15.50 usec
P11        500.00 usec
P12        2000.00 usec
PL0        120.00 dB
PL1        -1.00 dB
SFO1       125.7942548 MHz
SF1        3.20 dB
SF2        3.20 dB
SFO1M1     Crp60,0.5,20.1
SFO1M2     Crp60comp,4
SFOFF1     0.00 Hz
SFOFF2     0.00 Hz

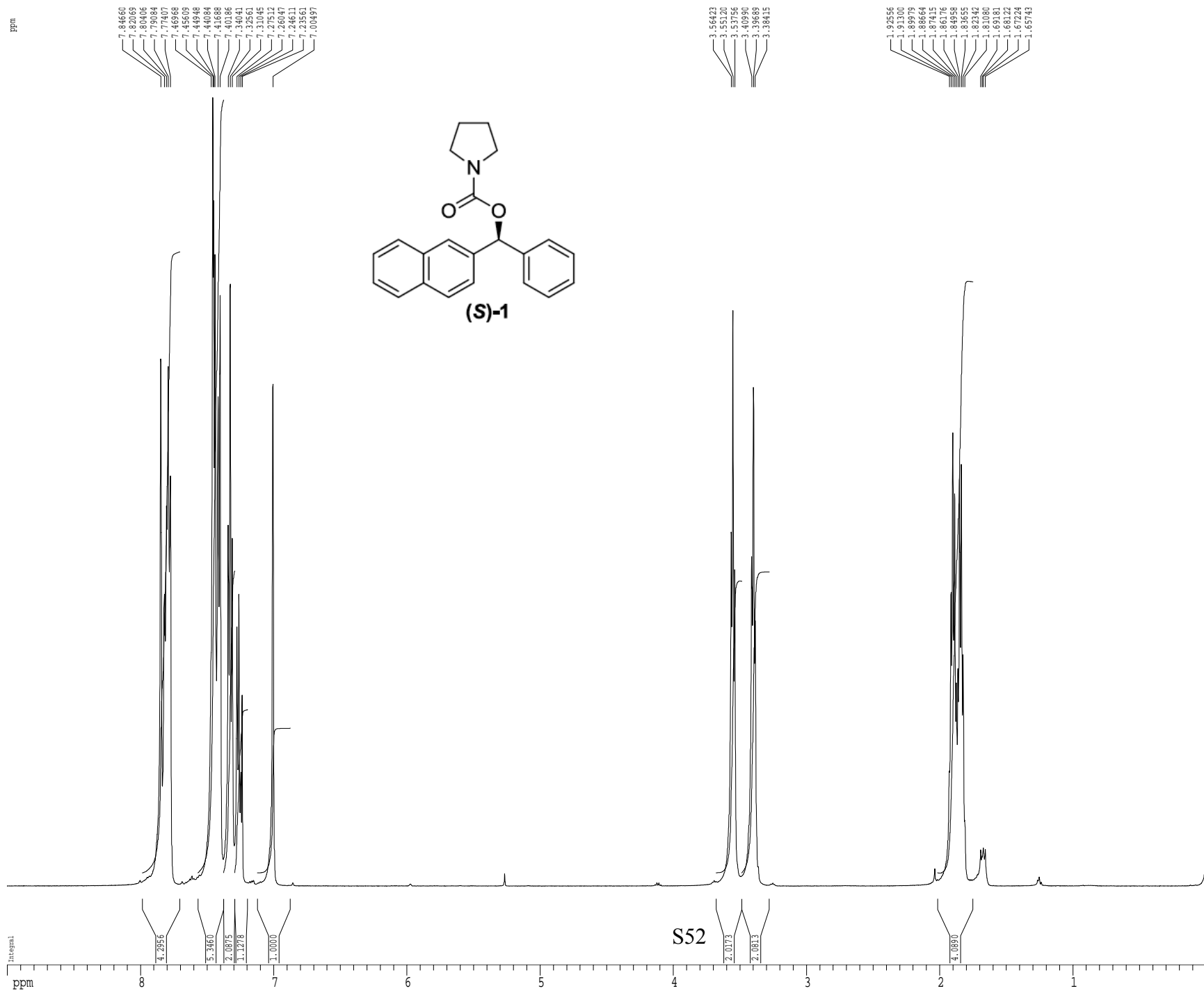
===== CHANNEL f2 =====
CPDPRG2    waltz16
NUC2       1H
PCPD2     100.00 usec
PL2       1.60 dB
PL12      24.60 dB
SFO2      500.2225011 MHz

===== GRADIENT CHANNEL =====
GENAM1     SINE.100
GENAM2     SINE.100
GFX1       0.00 %
GFX2       0.00 %
GPY1       0.00 %
GPY2       0.00 %
GPZ1       30.00 %
GPZ2       50.00 %
p15        500.00 usec
p16        1000.00 usec

F2 - Processing parameters
SI         65536
SF         125.7804131 MHz
WDW        EM
SSB        0
LB         1.00 Hz
GB         0
PC         2.00

1D NMR plot parameters
CX         22.80 cm
CY         15.65 cm
F1P        200.000 ppm
F1         25156.08 Hz
F2P        0.000 ppm
F2         0.00 Hz
PFMCM      8.77193 ppm/cm
HZCM       1103.33691 Hz/cm
    
```

¹H spectrum



Current Data Parameters
 USER: greene
 NAME: mrh-3-259
 EXPNO: 1
 PROCNO: 1

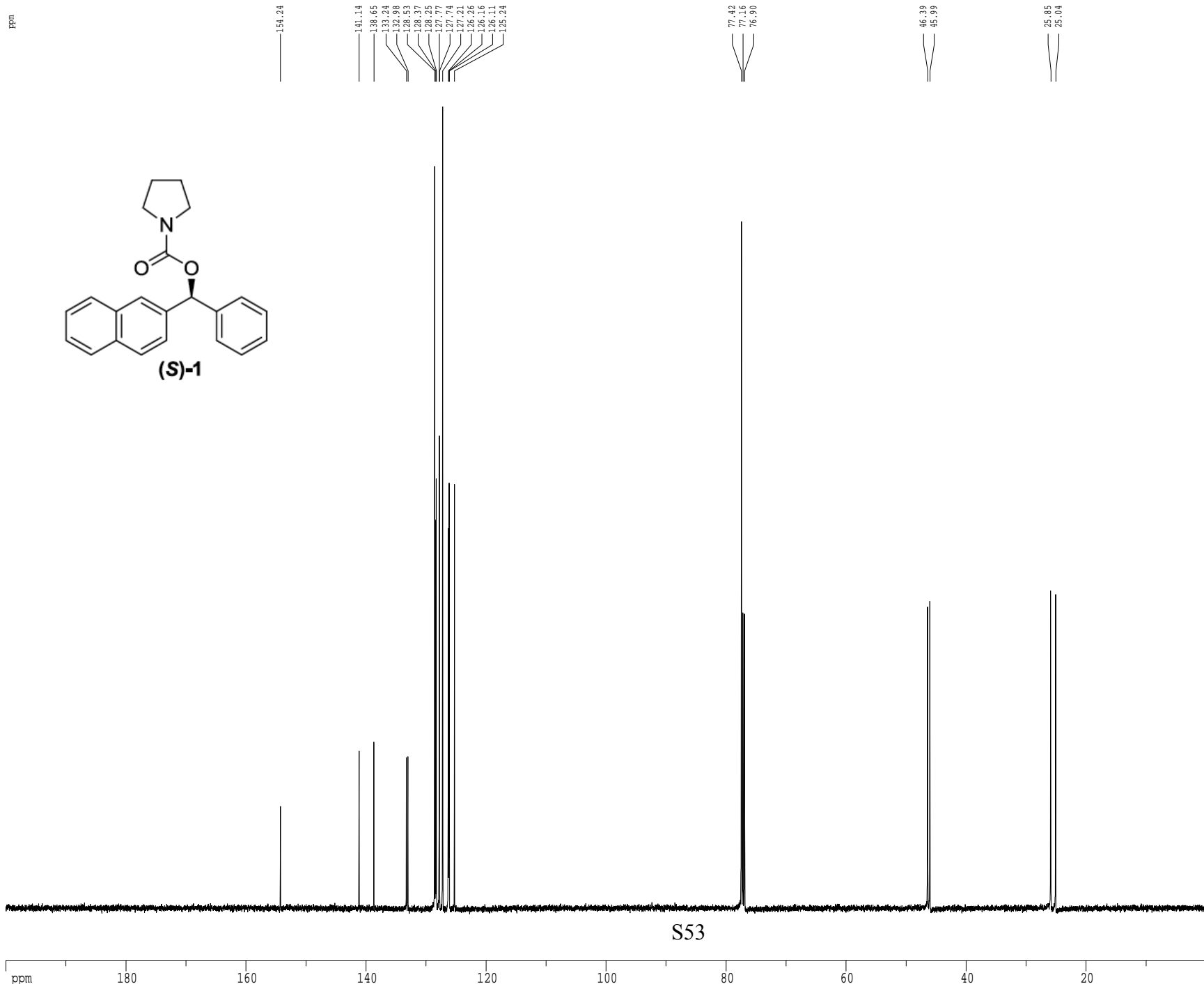
F2 - Acquisition Parameters
 Date_: 20121117
 Time: 9.00
 INSTRUM: cryo500
 PROBHD: 5 mm CPTCI 1H-
 PULPROG: zg30
 TD: 81728
 SOLVENT: CDCl3
 NS: 8
 DS: 2
 SWH: 8012.820 Hz
 FIDRES: 0.098043 Hz
 AQ: 5.0999398 sec
 RG: 5.7
 DW: 62.400 usec
 DE: 6.00 usec
 TE: 298.0 K
 DL: 0.10000000 sec
 MCREST: 0.00000000 sec
 MCWRR: 0.01500000 sec

===== CHANNEL f1 =====
 NUC1: 1H
 P1: 7.50 usec
 PL1: 1.60 dB
 SF01: 500.2235015 MHz

F2 - Processing parameters
 SI: 65536
 SF: 500.2200429 MHz
 WDW: EM
 SSB: 0
 LB: 0.30 Hz
 GB: 0
 PC: 4.00

1D NMR plot parameters
 CX: 22.80 cm
 CY: 15.00 cm
 F1P: 9.000 ppm
 F1: 4501.98 Hz
 F2P: 0.000 ppm
 F2: 0.00 Hz
 PPMCM: 0.39474 ppm/cm
 HZCM: 197.45528 Hz/cm

Z-restored spin-echo 13C spectrum with 1H decoupling



```

Current Data Parameters
USER      greene
NAME      mrh-3-259
EXPNO     2
PROCNO    1

F2 - Acquisition Parameters
Date_     20121117
Time      9.06
INSTRUM   cryo500
PROBHD    5 mm CPTCI 1H-
PULPROG   SpinEchopg30gp.prd
TD         65536
SOLVENT   CDCl3
NS         360
DS         16
SWH        30303.031 Hz
FIDRES     0.462388 Hz
AQ         1.0814105 sec
RG         7298.2
DW         16.500 usec
DE         6.00 usec
TE         298.0 K
D1         0.25000000 sec
d11        0.03000000 sec
D16        0.00020000 sec
d17        0.00019600 sec
MCREST     0.00000000 sec
MCMRXX     0.01500000 sec
P2         31.00 usec

===== CHANNEL f1 =====
NUC1       13c
P1         15.50 usec
P11        500.00 usec
P12        2000.00 usec
PL0        120.00 dB
PL1        -1.00 dB
SFO1       125.7942548 MHz
SF1        3.20 dB
SF2        3.20 dB
SFO1M1     Crp60,0.5,20.1
SFO1M2     Crp60comp,4
SFOFF1     0.00 Hz
SFOFF2     0.00 Hz

===== CHANNEL f2 =====
CPDPRG2    waltz16
NUC2       1H
PCPD2     100.00 usec
PL2       1.60 dB
PL12      24.60 dB
SFO2      500.2225011 MHz

===== GRADIENT CHANNEL =====
GENAM1     SINE.100
GENAM2     SINE.100
GFX1       0.00 %
GFX2       0.00 %
GPY1       0.00 %
GPY2       0.00 %
GPZ1       30.00 %
GPZ2       50.00 %
p15        500.00 usec
p16        1000.00 usec

F2 - Processing parameters
SI         65536
SF         125.7804150 MHz
WDW        EM
SSB        0
LB         1.00 Hz
GB         0
PC         2.00

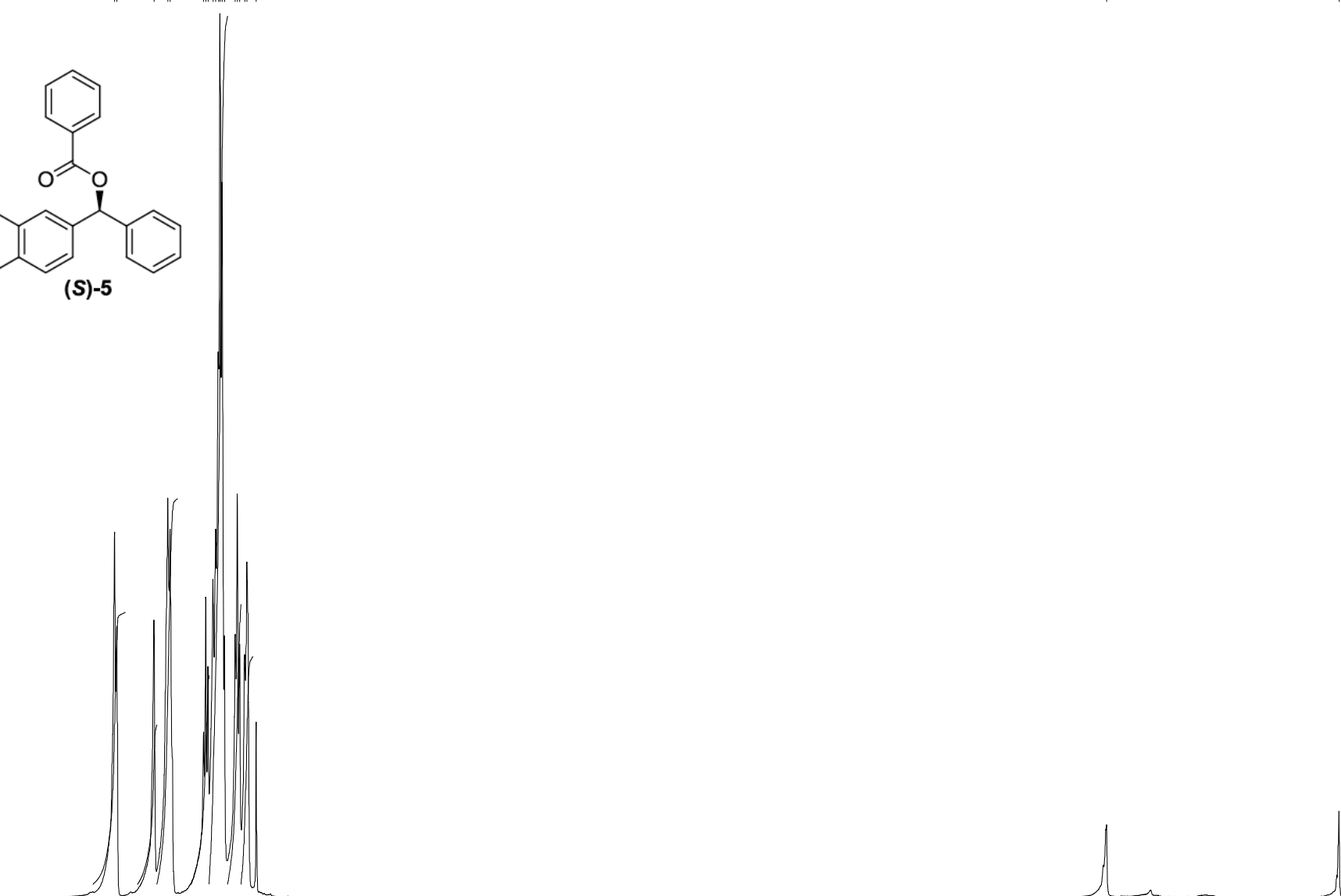
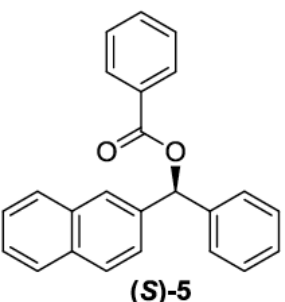
1D NMR plot parameters
CX         22.80 cm
CY         15.65 cm
F1P        200.000 ppm
F1         25156.08 Hz
F2P        0.000 ppm
F2         0.00 Hz
PFMCM      8.77193 ppm/cm
HZCM       1103.33704 Hz/cm
    
```

¹H spectrum

ppm
 8.26811
 8.25253
 8.00423
 7.91254
 7.89662
 7.67324
 7.65863
 7.64402
 7.60962
 7.57259
 7.55336
 7.55026
 7.53958
 7.46228
 7.44810
 7.43301
 7.39818
 7.38370
 7.32170

1.63897

0.08460



```

Current Data Parameters
USER      greene
NAME      mrh-3-146
EXPNO     3
PROCNO    1

F2 - Acquisition Parameters
Date_     20121118
Time      9.17
INSTRUM   cryo500
PROBHD    5 mm CPTCI 1H-
PULPROG   zg30
TD         81728
SOLVENT   CDCl3
NS         8
DS         2
SWH        8012.820 Hz
FIDRES     0.098043 Hz
AQ         5.0998774 sec
RG         16
DW         62.400 usec
DE         6.00 usec
TE         298.0 K
DL         0.10000000 sec
MCREST    0.00000000 sec
MCWRX     0.01500000 sec

===== CHANNEL f1 =====
NUC1       1H
P1         7.50 usec
PL1        1.60 dB
SF01       500.2235015 MHz

F2 - Processing parameters
SI         65536
SF         500.2200000 MHz
WDW        no
SSB        0
LB         0.00 Hz
GB         0
PC         4.00

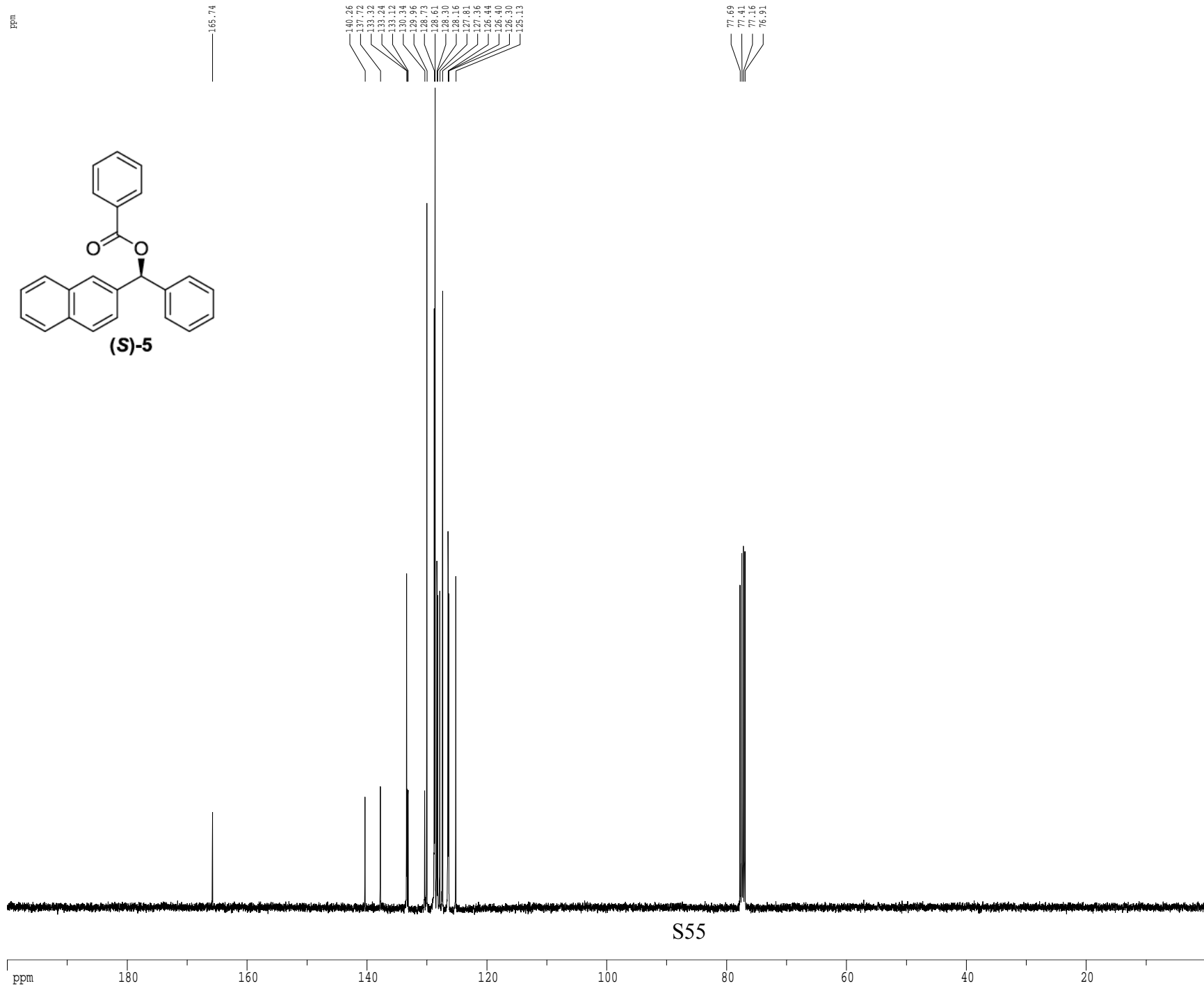
1D NMR plot parameters
CX         22.80 cm
CY         15.00 cm
F1P        9.000 ppm
F1         4501.98 Hz
F2P        0.000 ppm
F2         0.00 Hz
PPMCM      0.39474 ppm/cm
HZCM       197.45528 Hz/cm
    
```

Integral
 2.0000
 1.1723
 2.8354
 1.5354
 6.3856
 3.0572
 1.6730

S54

ppm
 8
 7
 6
 5
 4
 3
 2
 1

Z-restored spin-echo 13C spectrum with 1H decoupling



```

Current Data Parameters
USER      greene
NAME      mrh-3-146
EXPNO     2
PROCNO    1

F2 - Acquisition Parameters
Date_     20121118
Time      9.10
INSTRUM   cryo500
PROBHD    5 mm CPTCI 1H-
PULPROG   SpinEchopg30gp.prd
TD         65536
SOLVENT   CDCl3
NS         546
DS         16
SWH        30303.031 Hz
FIDRES     0.462388 Hz
AQ         1.0814105 sec
RG         11585.2
DW         16.500 usec
DE         6.00 usec
TE         298.0 K
d1         0.25000000 sec
d11        0.03000000 sec
d16        0.00020000 sec
d17        0.00019600 sec
MCREST     0.00000000 sec
MCWRX      0.01500000 sec
P2         31.00 usec

===== CHANNEL f1 =====
NUC1       13c
P1         15.50 usec
P11        500.00 usec
P12        2000.00 usec
PL0        120.00 dB
PL1        -1.00 dB
SFO1       125.7942548 MHz
SF1        3.20 dB
SF2        3.20 dB
SFO1M1     Crp60,0.5,20.1
SFO1M2     Crp60comp,4
SFOFF1     0.00 Hz
SFOFF2     0.00 Hz

===== CHANNEL f2 =====
CPDPRG2    waltz16
NUC2       1H
PCPD2     100.00 usec
PL2       1.60 dB
PL12      24.60 dB
SFO2      500.2225011 MHz

===== GRADIENT CHANNEL =====
GENAM1     SINE.100
GENAM2     SINE.100
GFX1       0.00 %
GFX2       0.00 %
GPY1       0.00 %
GPY2       0.00 %
GPZ1       30.00 %
GPZ2       50.00 %
p15        500.00 usec
p16        1000.00 usec

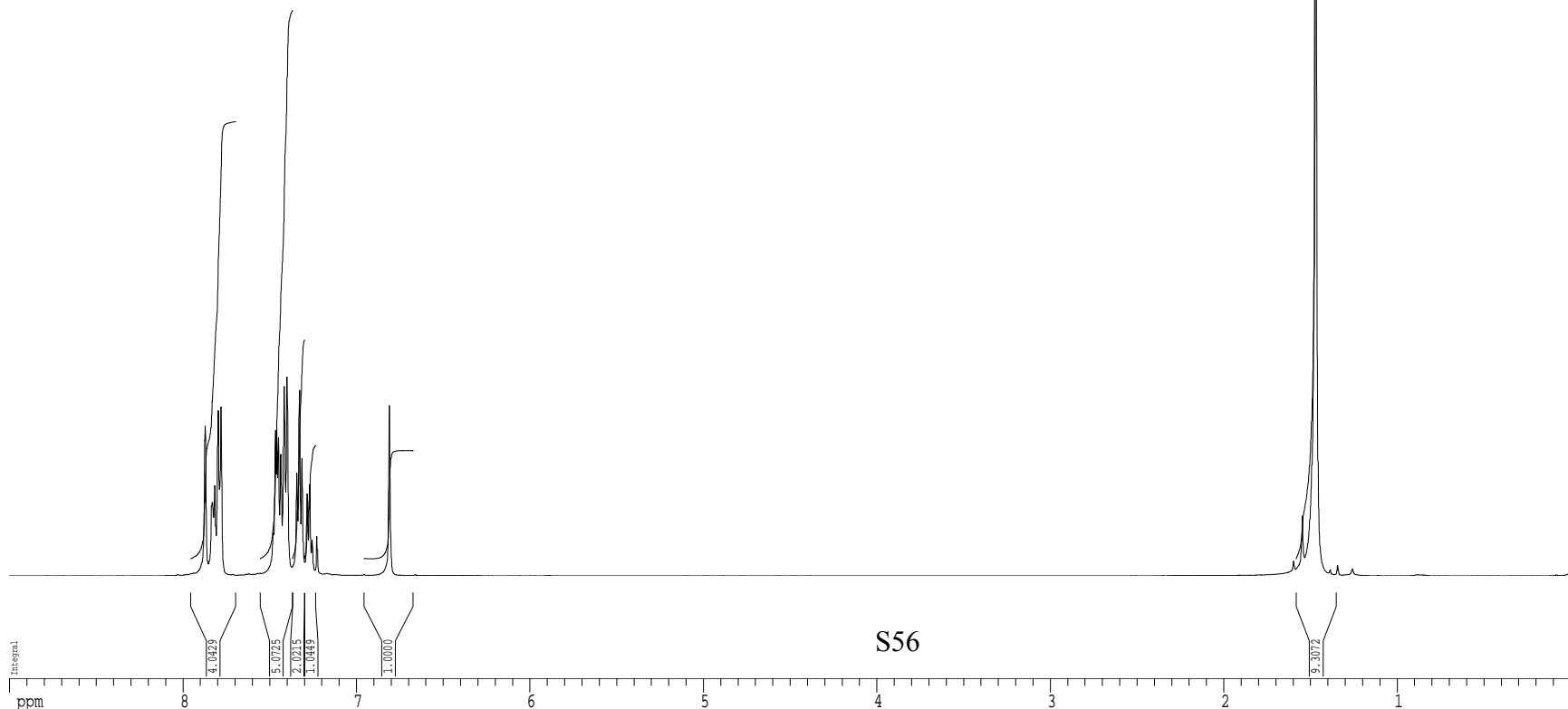
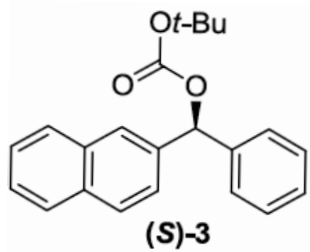
F2 - Processing parameters
SI         65536
SF         125.7804122 MHz
WDW        EM
SSB        0
LB         1.00 Hz
GB         0
PC         2.00

1D NMR plot parameters
CX         22.80 cm
CY         15.65 cm
F1P        200.000 ppm
F1         25156.08 Hz
F2P        0.000 ppm
F2         0.00 Hz
PFMCM      8.77193 ppm/cm
HZCM       1103.33691 Hz/cm
    
```

S55

¹H spectrum

ppm
 7.87142
 7.83300
 7.82903
 7.81585
 7.79642
 7.77943
 7.47801
 7.46501
 7.45730
 7.44952
 7.43439
 7.41517
 7.39866
 7.34299
 7.32827
 7.31300
 7.28335
 7.28877
 7.27544
 7.27154
 6.80985



S56

```

Current Data Parameters
USER      greene
NAME      mrh-3-123a
EXPNO     1
PROCNO    1

F2 - Acquisition Parameters
Date_     20121117
Time      8.35
INSTRUM   cryo500
PROBHD    5 mm CPTCI 1H-
PULPROG   zg30
TD         81728
SOLVENT   CDCl3
NS         8
DS         2
SWH        8012.820 Hz
FIDRES     0.098043 Hz
AQ         5.0999398 sec
RG         10.1
DW         62.400 usec
DE         6.00 usec
TE         298.0 K
DL         0.10000000 sec
MCREST     0.00000000 sec
MCWRX      0.01500000 sec

===== CHANNEL f1 =====
NUC1       1H
P1         7.50 usec
PL1        1.60 dB
SF01       500.2235015 MHz

F2 - Processing parameters
SI         65536
SF         500.2200469 MHz
WDW        EM
SSB        0
LB         0.30 Hz
GB         0
PC         4.00

1D NMR plot parameters
CX         22.80 cm
CY         15.00 cm
F1P        9.000 ppm
F1         4501.98 Hz
F2P        0.000 ppm
F2         0.00 Hz
PPMCM      0.39474 ppm/cm
HZCM       197.45528 Hz/cm
    
```

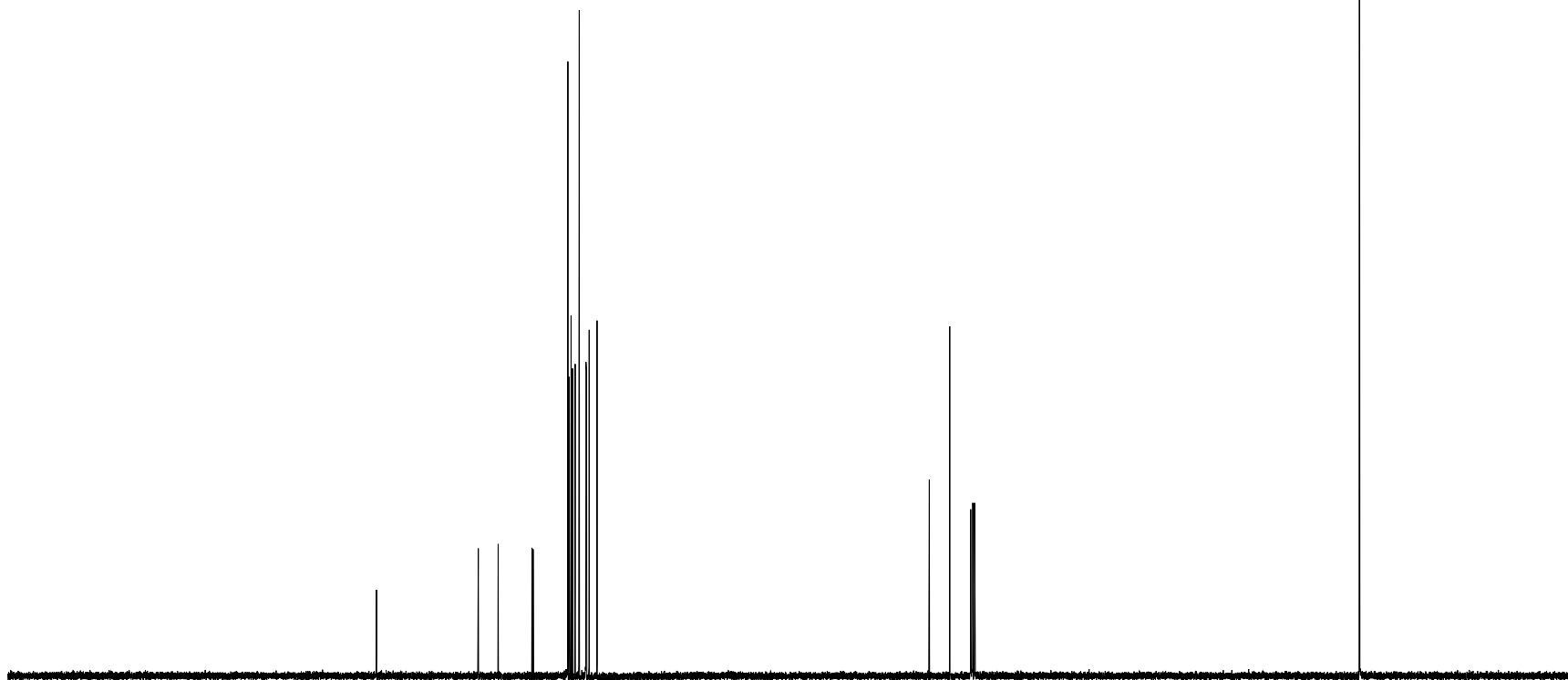
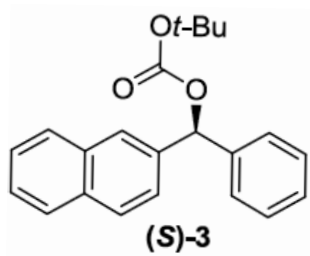

Z-restored spin-echo 13C spectrum with 1H decoupling

ppm

153.06
140.07
137.55
133.24
133.09
128.66
128.53
128.29
128.12
127.78
127.22
126.37
126.32
125.96
124.97

82.69
80.05
77.41
77.06
76.31

27.94



S57

ppm 180 160 140 120 100 80 60 40 20

```

Current Data Parameters
USER      greene
NAME      mch-3-123a
EXPNO     2
PROCNO    1

F2 - Acquisition Parameters
Date_     20121117
Time      8.57
INSTRUM   cryo500
PROBHD    5 mm CPTCI 1H-
PULPROG   SpinEchopg30gp.prd
TD         65536
SOLVENT   CDCl3
NS         296
DS         16
SWH        30303.031 Hz
FIDRES     0.462388 Hz
AQ         1.0814105 sec
RG         4096
DW         16.500 usec
DE         6.00 usec
TE         298.0 K
d1         0.25000000 sec
d11        0.03000000 sec
d16        0.00020000 sec
d17        0.00019600 sec
MCREST     0.00000000 sec
MCMRXX     0.01500000 sec
P2         31.00 usec

===== CHANNEL f1 =====
NUC1       13c
P1         15.50 usec
P11        500.00 usec
P12        2000.00 usec
PL0        120.00 dB
PL1        -1.00 dB
SFO1       125.7942548 MHz
SF1        3.20 dB
SF2        3.20 dB
SFOFF1     Crp60,0.5,20.1
SFOFF2     Crp60comp,4
SFOFF1     0.00 Hz
SFOFF2     0.00 Hz

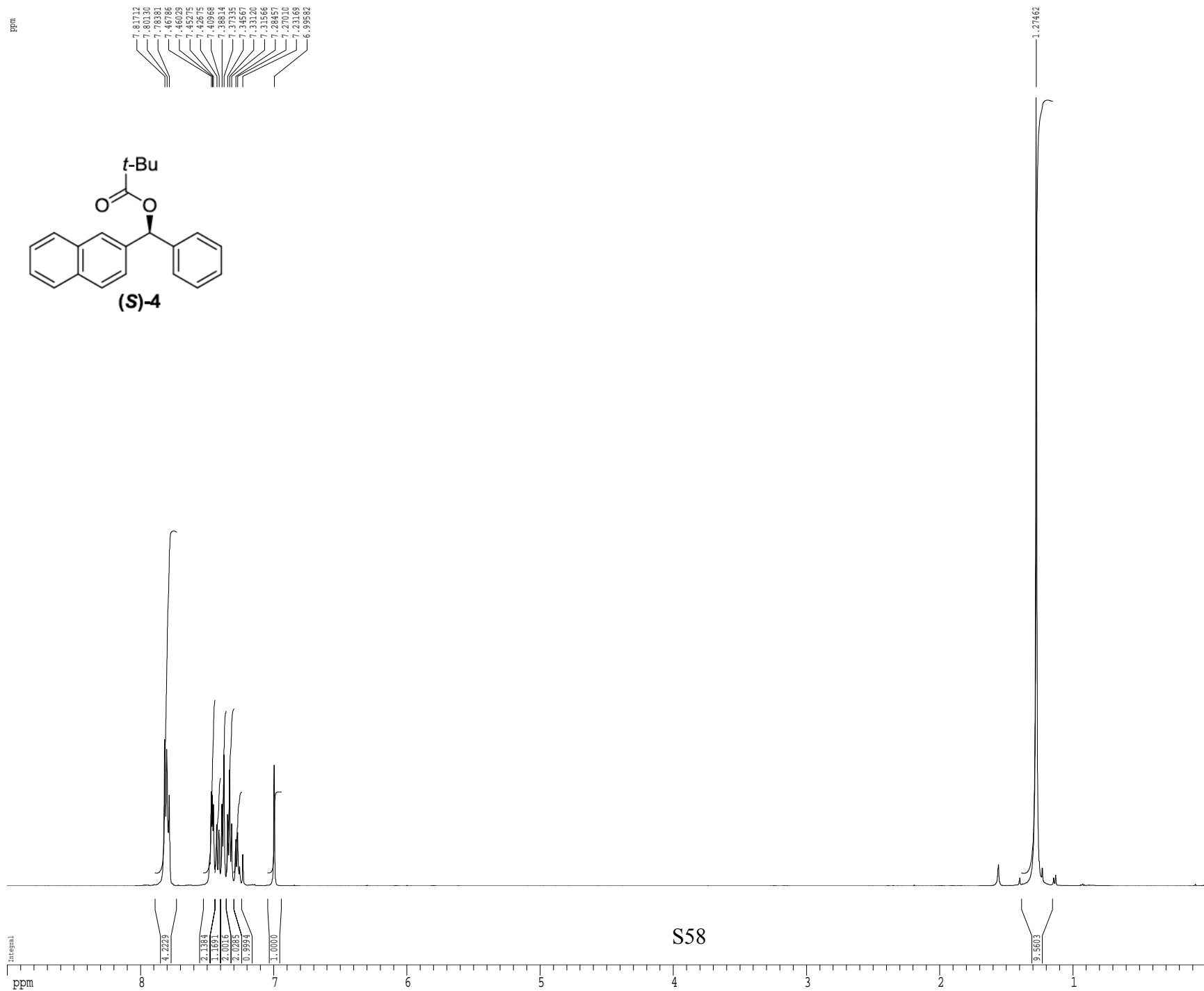
===== CHANNEL f2 =====
CPDPRG2    waltz16
NUC2       1H
PCPD2     100.00 usec
PL2        1.60 dB
PL12       24.60 dB
SFO2       500.2225011 MHz

===== GRADIENT CHANNEL =====
GENAM1     SINE.100
GENAM2     SINE.100
GFX1       0.00 %
GFX2       0.00 %
GPY1       0.00 %
GPY2       0.00 %
GPZ1       30.00 %
GPZ2       50.00 %
p15        500.00 usec
p16        1000.00 usec

F2 - Processing parameters
SI         65536
SF         125.7804140 MHz
WDW        no
SSB        0
LB         0.00 Hz
GB         0
PC         2.00

1D NMR plot parameters
CX         22.80 cm
CY         15.65 cm
F1P        200.000 ppm
F1         25156.08 Hz
F2P        0.000 ppm
F2         0.00 Hz
PFMCM      8.77193 ppm/cm
HZCM       1103.33691 Hz/cm
    
```

¹H spectrum



```

Current Data Parameters
USER      greene
NAME      mrh-3-190
EXPNO    1
PROCNO    1

F2 - Acquisition Parameters
Date_     20121117
Time      9.24
INSTRUM   cryo500
PROBHD    5 mm CPTCI 1H-
PULPROG   zg30
TD         81728
SOLVENT   CDCl3
NS         8
DS         2
SWH        8012.820 Hz
FIDRES     0.098043 Hz
AQ         5.0999398 sec
RG         5.7
DW         62.400 usec
DE         6.00 usec
TE         298.0 K
DL         0.10000000 sec
MCREST    0.00000000 sec
MCWRX     0.01500000 sec

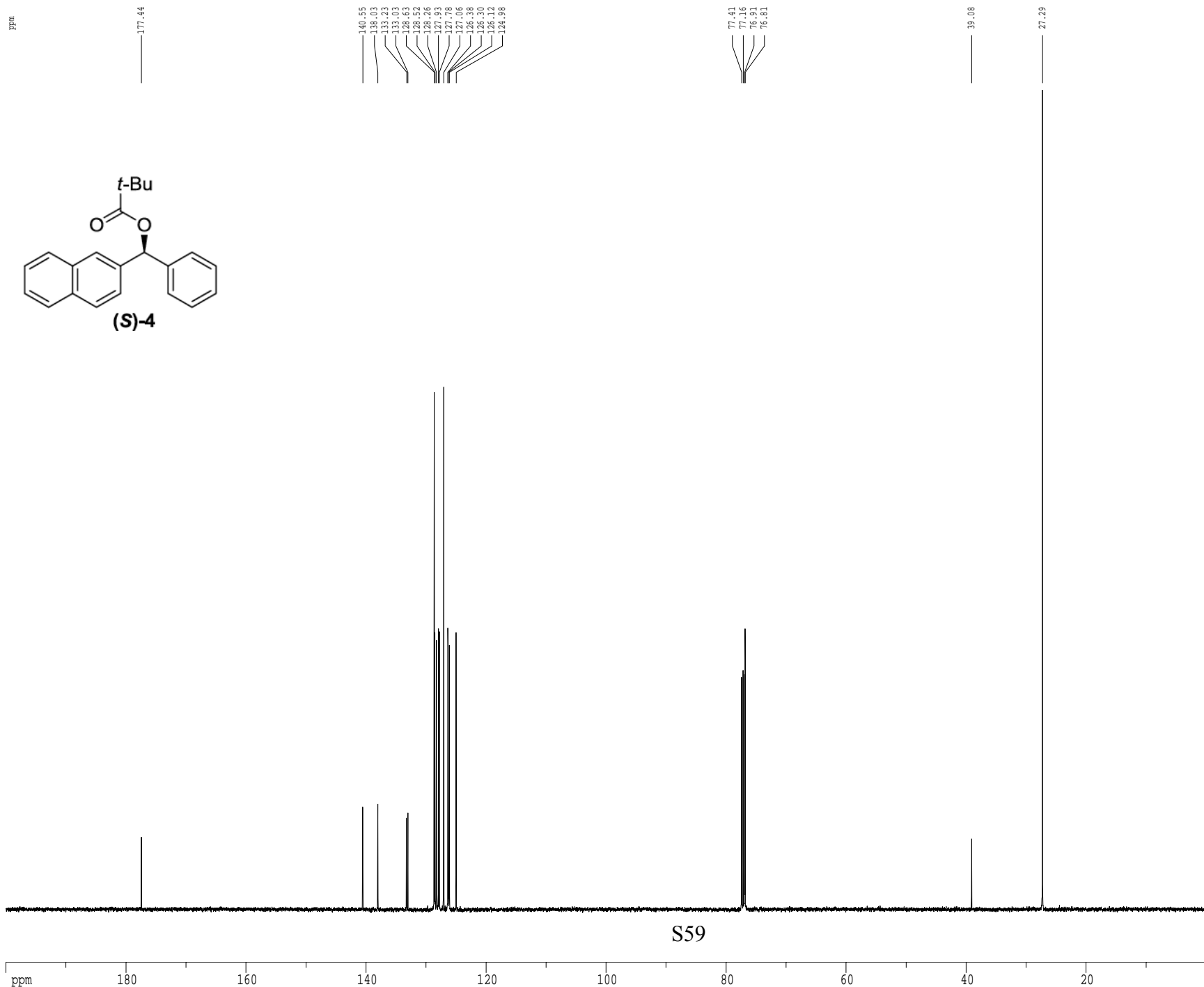
===== CHANNEL f1 =====
NUC1      1H
P1        7.50 usec
PL1       1.60 dB
SF01      500.2235015 MHz

F2 - Processing parameters
SI        65536
SF        500.2200451 MHz
WDW       EM
SSB       0
LB        0.30 Hz
GB        0
PC        4.00

1D NMR plot parameters
CX        22.80 cm
CY        15.00 cm
F1P       9.000 ppm
F1        4501.98 Hz
F2P       0.000 ppm
F2        0.00 Hz
PPMCM     0.39474 ppm/cm
HZCM      197.45528 Hz/cm
    
```

S58

Z-restored spin-echo 13C spectrum with 1H decoupling



```

Current Data Parameters
USER      greene
NAME      mrh-3-190
EXPNO     2
PROCNO    1

F2 - Acquisition Parameters
Date_     20121117
Time      9.31
INSTRUM   cryo500
PROBHD    5 mm CPTCI 1H-
PULPROG   SpinEchopg30gp.prd
TD         65536
SOLVENT   CDCl3
NS         266
DS         16
SWH        30303.031 Hz
FIDRES     0.462388 Hz
AQ         1.0814105 sec
RG         7298.2
DW         16.500 usec
DE         6.00 usec
TE         298.0 K
D1         0.25000000 sec
d11        0.03000000 sec
D16        0.00020000 sec
d17        0.00019600 sec
MCREST     0.00000000 sec
MCMRXX     0.01500000 sec
P2         31.00 usec

===== CHANNEL f1 =====
NUC1       13c
P1         15.50 usec
P11        500.00 usec
P12        2000.00 usec
PL0        120.00 dB
PL1        -1.00 dB
SFO1       125.7942548 MHz
SF1        3.20 dB
SF2        3.20 dB
SFO1M1     Crp60,0.5,20.1
SFO1M2     Crp60comp,4
SFOFF1     0.00 Hz
SFOFF2     0.00 Hz

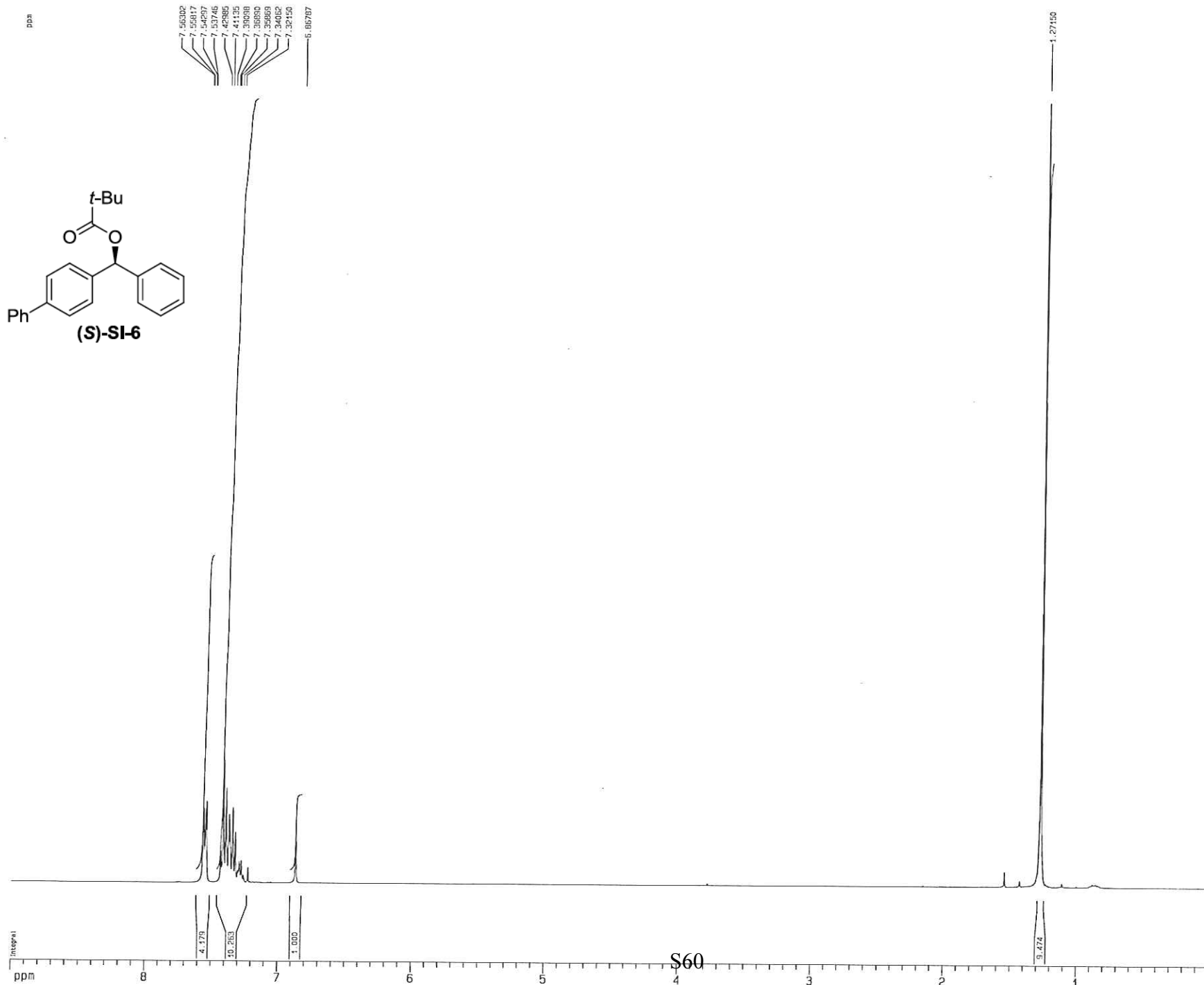
===== CHANNEL f2 =====
CPDPRG2    waltz16
NUC2       1H
PCPD2      100.00 usec
PL2        1.60 dB
PL12       24.60 dB
SFO2       500.2225011 MHz

===== GRADIENT CHANNEL =====
GENAM1     SINE.100
GENAM2     SINE.100
GFX1       0.00 %
GFX2       0.00 %
GPY1       0.00 %
GPY2       0.00 %
GPZ1       30.00 %
GPZ2       50.00 %
p15        500.00 usec
p16        1000.00 usec

F2 - Processing parameters
SI         65536
SF         125.7804127 MHz
WDW        HM
SSB        0
LB         1.00 Hz
GB         0
PC         2.00

1D NMR plot parameters
CX         22.80 cm
CY         15.65 cm
F1P        200.000 ppm
F1         25156.08 Hz
F2P        0.000 ppm
F2         0.00 Hz
PFMCM      8.77193 ppm/cm
HZCM       1103.33691 Hz/cm
    
```

1H spectrum



Current Data Parameters
 USER jhanna
 NAME LEH-1-121-c2-H1
 EXPNO 1
 PROCNO 1

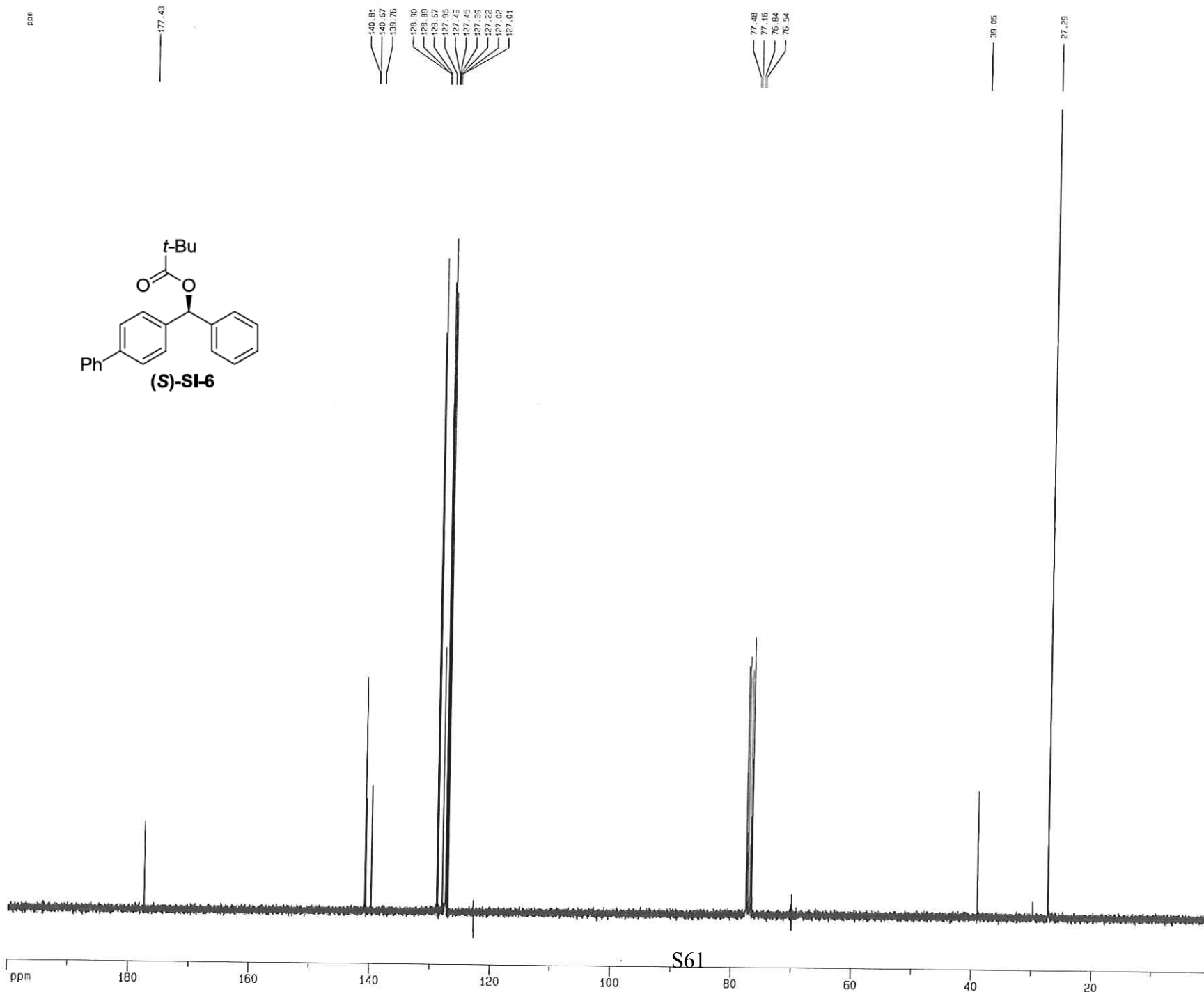
F2 - Acquisition Parameters
 Date_ 20121005
 Time 13.58
 INSTRUM drx400
 PROBHD 5 mm QNP H/F/P
 PULPROG zg30
 TD 65536
 SOLVENT CDCl3
 NS 8
 DS 2
 SFO1 6410.256 Hz
 FIDRES 0.097813 Hz
 AQ 5.1116579 sec
 RG 90.5
 DM 78.000 usec
 DE 4.50 usec
 TE 298.0 K
 D1 0.10000000 sec
 MCREST 0.00000000 sec
 MCWEX 0.01500000 sec

***** CHANNEL f1 *****
 NUC1 1H
 P1 12.00 usec
 PL1 -0.50 dB
 SFO1 400.1328009 MHz

F2 - Processing parameters
 SI 65536
 SF 400.1300353 MHz
 WDW no
 SSB 0
 LB 0.00 Hz
 GB 0
 PC 2.00

ID NMR plot parameters
 CX 22.80 cm
 CY 15.00 cm
 F1P 9.000 cm
 F1 3501.17 Hz
 F2P 0.000 cm
 F2 0.00 Hz
 PPMCM 0.33474 ppm/cm
 HZCM 157.94508 Hz/cm

¹³C spectrum with ¹H decoupling



Current Data Parameters
 USER Ihanna
 NAME LEH-1-121-c2-C13
 EXPNO 1
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20121005
 Time 13.54
 INSTRUM dnx400
 PROBHD 5 mm QNP H/F/P
 PULPROG zgpg30
 TD 65535
 SOLVENT CDCl3
 NS 1024
 DS 4
 SWH 24154.590 Hz
 FIDRES 0.368570 Hz
 AQ 1.3558452 sec
 RG 13004
 DW 20.700 usec
 DE 20.39 usec
 TE 298.0 K
 D1 0.10000000 sec
 d11 0.03000000 sec
 MCREST 0.00000000 sec
 MCWRR 0.01500000 sec

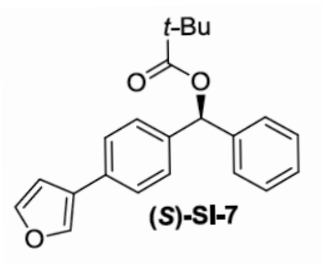
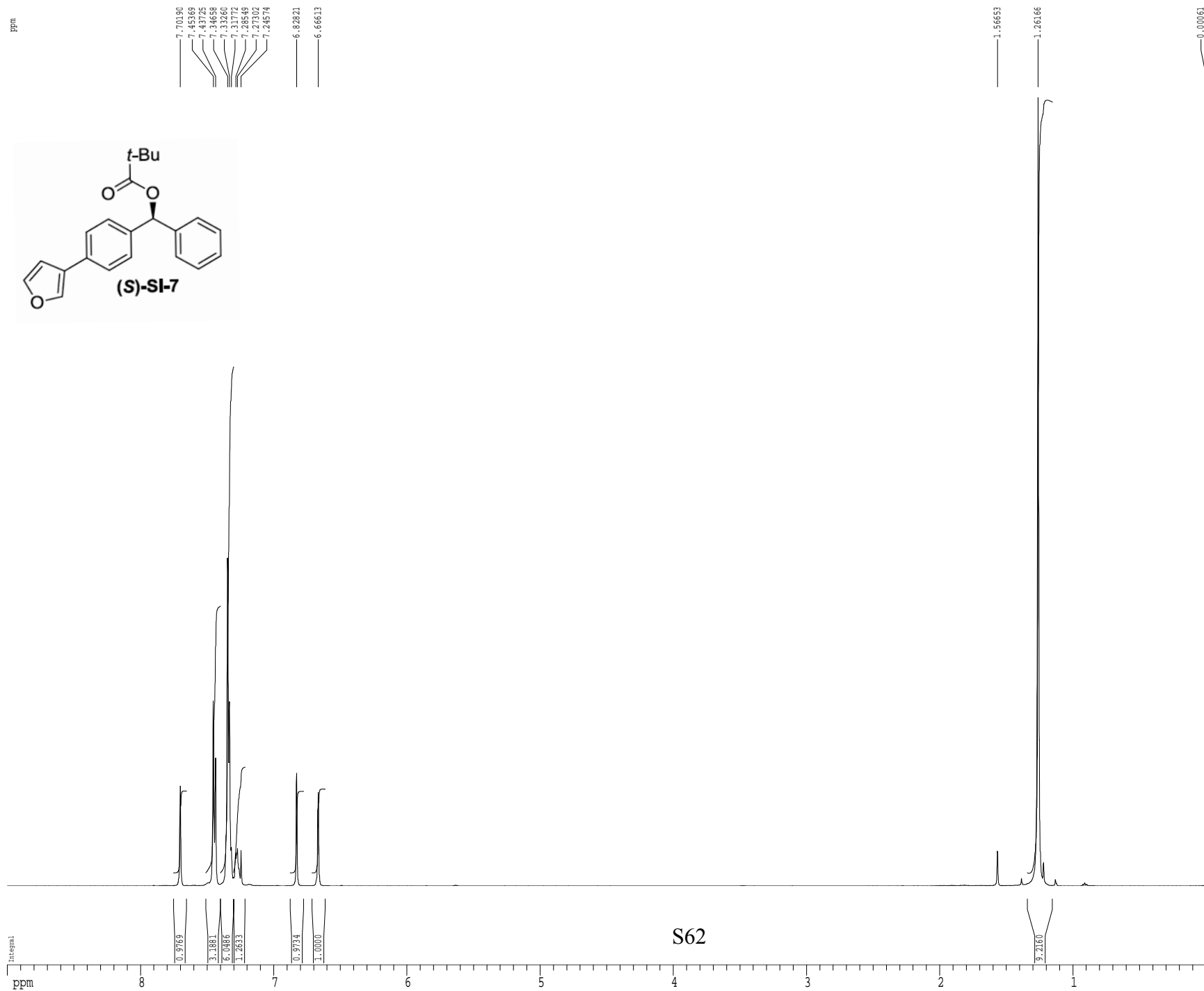
***** CHANNEL f1 *****
 NUC1 ¹³C
 P1 11.00 usec
 PL1 0.00 dB
 SF01 100.6237954 MHz

***** CHANNEL f2 *****
 CPDPRG2 mlev16
 NUC2 ¹H
 PCPD2 80.00 usec
 PL2 0.00 dB
 PL12 16.20 dB
 SF02 400.1328309 MHz

F2 - Processing parameters
 SI 65535
 SF 100.6127624 MHz
 WDW no
 SSB 0
 LB 0.00 Hz
 GB 0
 PC 1.00

1D NMR plot parameters
 CX 22.80 cm
 CY 15.50 cm
 F1P 200.000 ppm
 F1 20122.55 Hz
 F2P 0.000 ppm
 F2 0.00 Hz
 PPMCM 8.77193 ppm/cm
 HZCM 882.55812 Hz/cm

¹H spectrum



```

Current Data Parameters
USER      greene
NAME      mrh-4-134
EXPNO     1
PROCNO    1

F2 - Acquisition Parameters
Date_     20121117
Time      9.46
INSTRUM   cryo500
PROBHD    5 mm CPTCI 1H-
PULPROG   zg30
TD         81728
SOLVENT   CDCl3
NS         8
DS         2
SWH        8012.820 Hz
FIDRES     0.098043 Hz
AQ         5.0999398 sec
RG         6.3
DW         62.400 usec
DE         6.00 usec
TE         298.0 K
DL         0.10000000 sec
MCREST    0.00000000 sec
MCWRX     0.01500000 sec

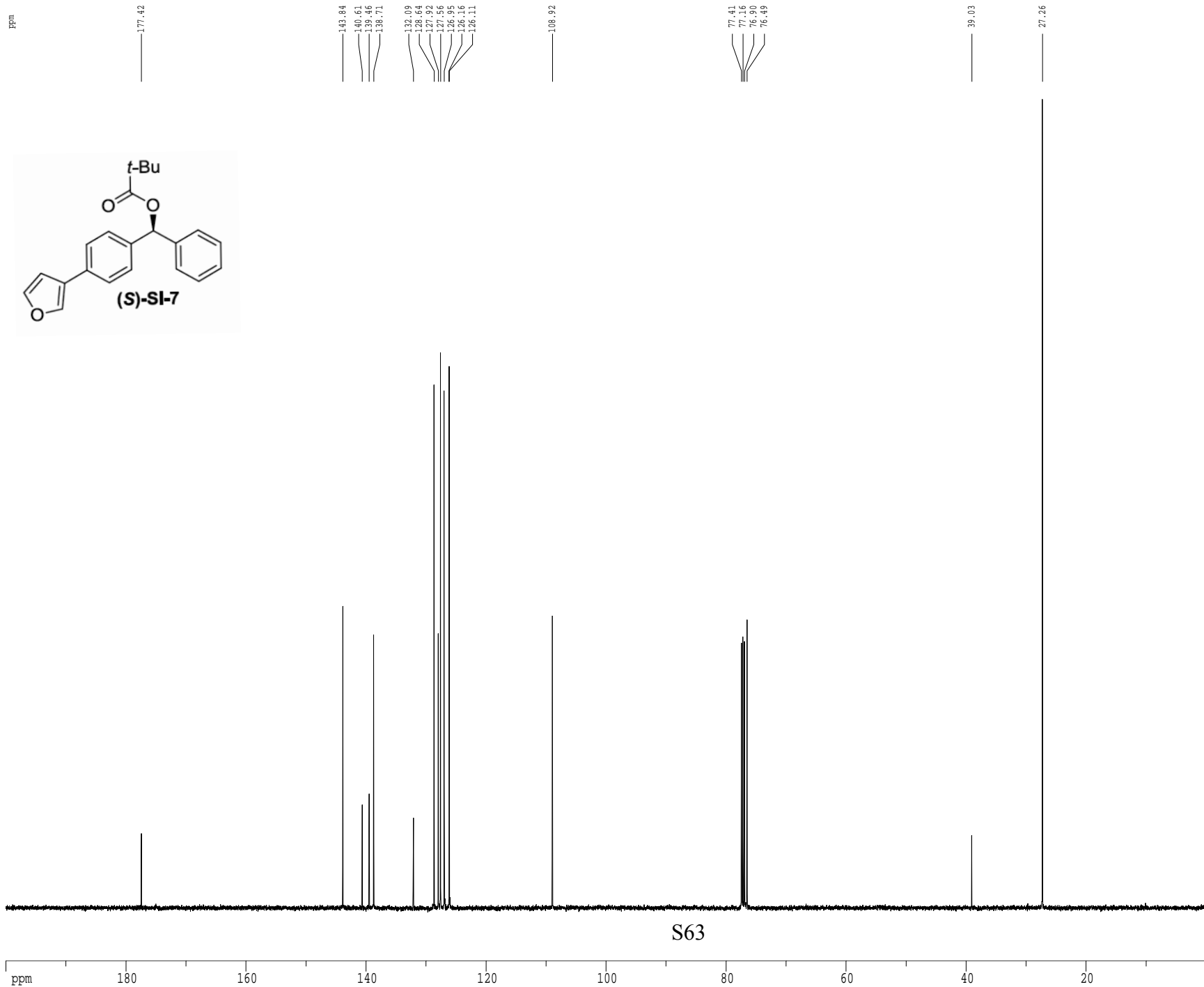
===== CHANNEL f1 =====
NUC1      1H
P1        7.50 usec
PL1       1.60 dB
SF01      500.2235015 MHz

F2 - Processing parameters
SI         65536
SF         500.2200381 MHz
WDW        EM
SSB        0
LB         0.30 Hz
GB         0
PC         4.00

1D NMR plot parameters
CX         22.80 cm
CY         15.00 cm
F1P        9.000 ppm
F1         4501.98 Hz
F2P        0.000 ppm
F2         0.00 Hz
PPMCM      0.39474 ppm/cm
HZCM       197.45528 Hz/cm
    
```

S62

Z-restored spin-echo 13C spectrum with 1H decoupling



```

Current Data Parameters
USER      greene
NAME      mrh-4-134
EXPNO     2
PROCNO    1

F2 - Acquisition Parameters
Date_     20121117
Time      9.54
INSTRUM   cryo500
PROBHD    5 mm CPTCI 1H-
PULPROG   SpinEchopg30gp.prd
TD         65536
SOLVENT   CDCl3
NS         311
DS         16
SWH        30303.031 Hz
FIDRES     0.462388 Hz
AQ         1.0814105 sec
RG         7298.2
DW         16.500 usec
DE         6.00 usec
TE         298.0 K
D1         0.25000000 sec
d11        0.03000000 sec
D16        0.00020000 sec
d17        0.00019600 sec
MCREST     0.00000000 sec
MCONRX     0.01500000 sec
P2         31.00 usec

===== CHANNEL f1 =====
NUC1       13c
P1         15.50 usec
P11        500.00 usec
P12        2000.00 usec
PL0        120.00 dB
PL1        -1.00 dB
SFO1       125.7942548 MHz
SF1        3.20 dB
SF2        3.20 dB
SFO1M1     Crp60,0.5,20.1
SFO1M2     Crp60comp,4
SFOFF1     0.00 Hz
SFOFF2     0.00 Hz

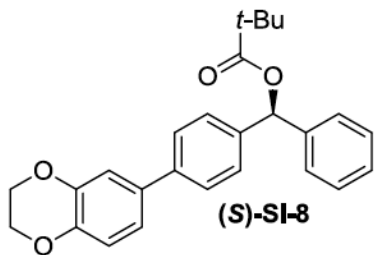
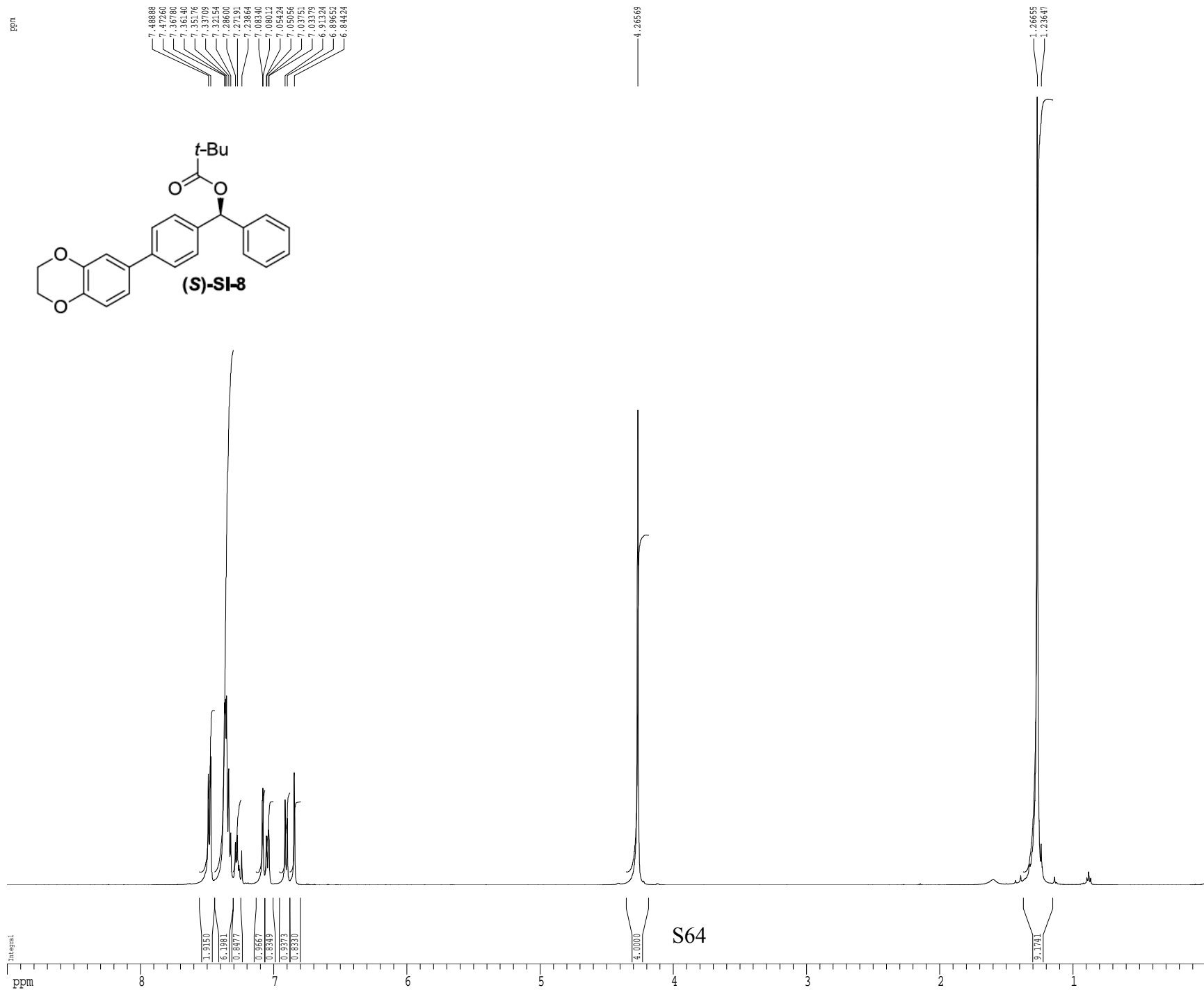
===== CHANNEL f2 =====
CPDPRG2    waltz16
NUC2       1H
PCPD2      100.00 usec
PL2        1.60 dB
PL12       24.60 dB
SFO2       500.2225011 MHz

===== GRADIENT CHANNEL =====
GENAM1     SINE.100
GENAM2     SINE.100
GFX1       0.00 %
GFX2       0.00 %
GPY1       0.00 %
GPY2       0.00 %
GPZ1       30.00 %
GPZ2       50.00 %
p15        500.00 usec
p16        1000.00 usec

F2 - Processing parameters
SI         65536
SF         125.7804108 MHz
WDW        EM
SSB        0
LB         1.00 Hz
GB         0
PC         2.00

ID NMR plot parameters
CX         22.80 cm
CY         15.65 cm
F1P        200.000 ppm
F1         25156.08 Hz
F2P        0.000 ppm
F2         0.00 Hz
PFMCM      8.77193 ppm/cm
HZCM       1103.33691 Hz/cm
    
```

¹H spectrum



Current Data Parameters
 USER greene
 NAME mrh-4-139
 EXPNO 1
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20121117
 Time 9.14
 INSTRUM cryo500
 PROBHD 5 mm CPTCI 1H-
 PULPROG zg30
 TD 81728
 SOLVENT CDCl3
 NS 8
 DS 2
 SWH 8012.820 Hz
 FIDRES 0.098043 Hz
 AQ 5.0999398 sec
 RG 5.7
 DW 62.400 usec
 DE 6.00 usec
 TE 298.0 K
 DL 0.10000000 sec
 MCREST 0.00000000 sec
 MCWEX 0.01500000 sec

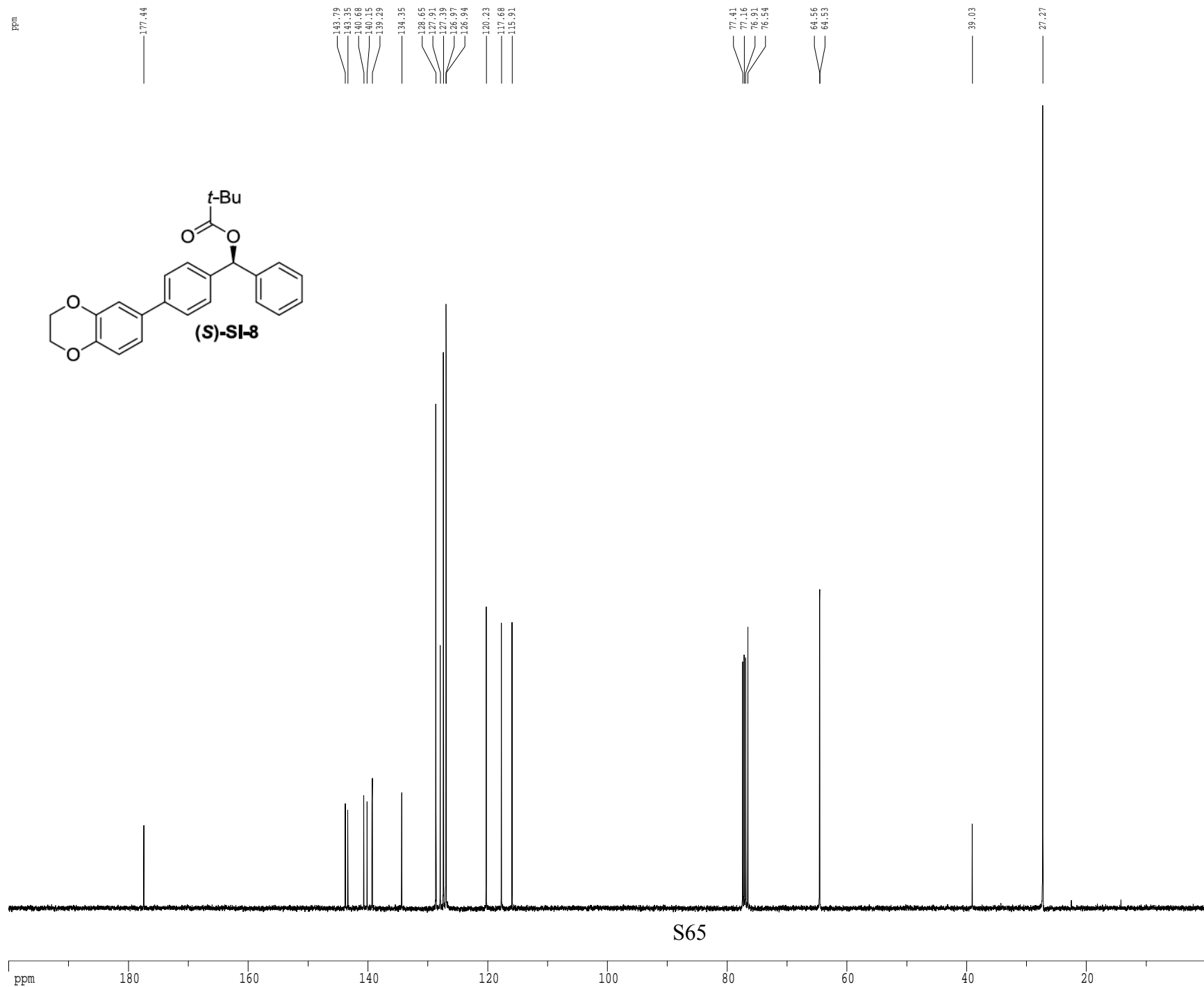
===== CHANNEL f1 =====
 NUC1 1H
 P1 7.50 usec
 PL1 1.60 dB
 SF01 500.2235015 MHz

F2 - Processing parameters
 SI 65536
 SF 500.2200413 MHz
 WDW EM
 SSB 0
 LB 0.30 Hz
 GB 0
 PC 4.00

1D NMR plot parameters
 CX 22.80 cm
 CY 15.00 cm
 F1P 9.000 ppm
 F1 4501.98 Hz
 F2P 0.000 ppm
 F2 0.00 Hz
 PPMCM 0.39474 ppm/cm
 HZCM 197.45528 Hz/cm

S64

Z-restored spin-echo 13C spectrum with 1H decoupling



```

Current Data Parameters
USER      greene
NAME      mrh-4-139
EXPNO     2
PROCNO    1

F2 - Acquisition Parameters
Date_     20121117
Time      9.20
INSTRUM   cryo500
PROBHD    5 mm CPTCI 1H-
PULPROG   SpinEchopg30gp.prd
TD         65536
SOLVENT   CDCl3
NS         253
DS         16
SWH        30303.031 Hz
FIDRES     0.462388 Hz
AQ         1.0814105 sec
RG         4096
DW         16.500 usec
DE         6.00 usec
TE         298.0 K
D1         0.25000000 sec
d11        0.03000000 sec
d16        0.00020000 sec
d17        0.00019600 sec
MCREST     0.00000000 sec
MCMRXX     0.01500000 sec
P2         31.00 usec

===== CHANNEL f1 =====
NUC1       13C
P1         15.50 usec
P11        500.00 usec
P12        2000.00 usec
PL0        120.00 dB
PL1        -1.00 dB
SFO1       125.7942548 MHz
SF1        3.20 dB
SF2        3.20 dB
SFO1M1     Crp60,0.5,20.1
SFO1M2     Crp60comp,4
SFOFF1     0.00 Hz
SFOFF2     0.00 Hz

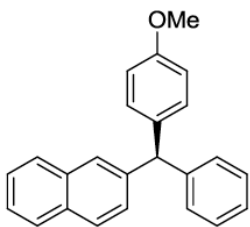
===== CHANNEL f2 =====
CPDPRG2    waltz16
NUC2       1H
PCPD2      100.00 usec
PL2        1.60 dB
PL12       24.60 dB
SFO2       500.2225011 MHz

===== GRADIENT CHANNEL =====
GENAM1     SINE.100
GENAM2     SINE.100
GFX1       0.00 %
GFX2       0.00 %
GPF1       0.00 %
GPF2       0.00 %
GPF3       0.00 %
GPF4       30.00 %
GPF5       50.00 %
p15        500.00 usec
p16        1000.00 usec

F2 - Processing parameters
SI         65536
SF         125.7804127 MHz
WDW        EM
SSB        0
LB         1.00 Hz
GB         0
PC         2.00

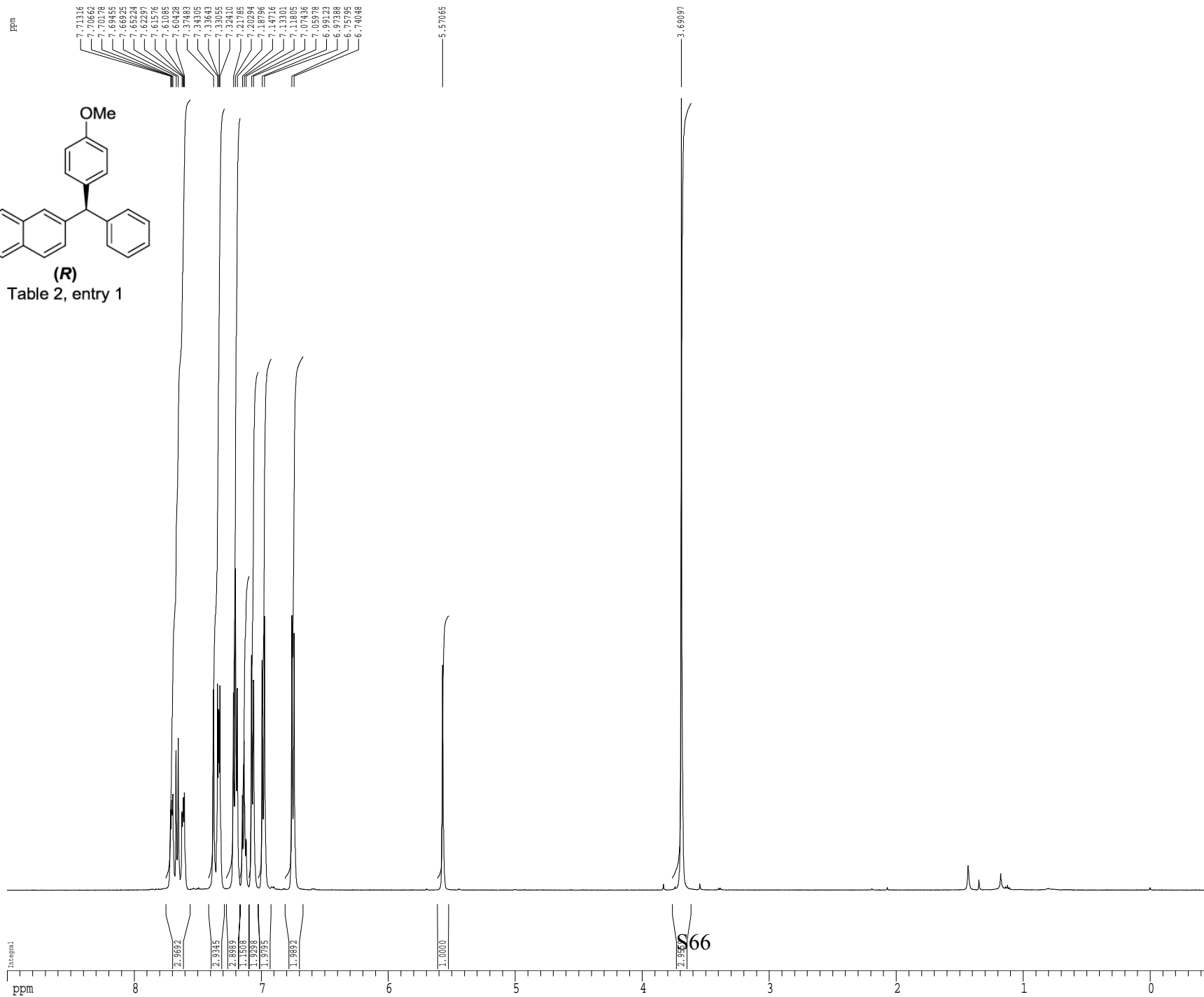
1D NMR plot parameters
CX         22.80 cm
CY         15.65 cm
F1P        200.000 ppm
F1         25156.08 Hz
F2P        0.000 ppm
F2         0.00 Hz
PFMCM      8.77193 ppm/cm
HZCM       1103.33691 Hz/cm
    
```

¹H spectrum



(R)

Table 2, entry 1



Current Data Parameters
 USER mharri
 NAME MRH-III-264-1HNMR
 EXPNO 3
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20120928
 Time 10.49
 INSTRUM cryo500
 PROBHD 5 mm CPTCI 1H-
 PULPROG zg30
 TD 81728
 SOLVENT CDCl3
 DS 2
 NS 1
 SWH 8012.820 Hz
 FIDRES 0.098043 Hz
 AQ 5.0998774 sec
 RG 5
 DW 62.400 usec
 DE 6.00 usec
 TE 298.1 K
 D1 0.10000000 sec
 MCREST 0.00000000 sec
 MCNRK 0.01500000 sec

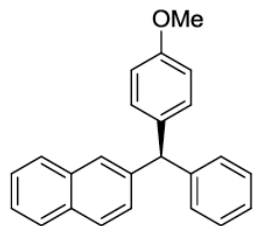
===== CHANNEL f1 =====
 NUC1 1H
 P1 7.50 usec
 PL1 1.60 dB
 SFO1 500.2235015 MHz

F2 - Processing parameters
 SI 65536
 SF 500.2200931 MHz
 WDW EM
 SSB 0
 LB 0.30 Hz
 GB 0
 PC 4.00

1D NMR plot parameters
 CX 22.80 cm
 CY 15.00 cm
 F1 9.000 ppm
 F1 4501.98 Hz
 F2P -0.500 ppm
 F2 -250.11 Hz
 PPMCM 0.41667 ppm/cm
 HZCM 208.42505 Hz/cm

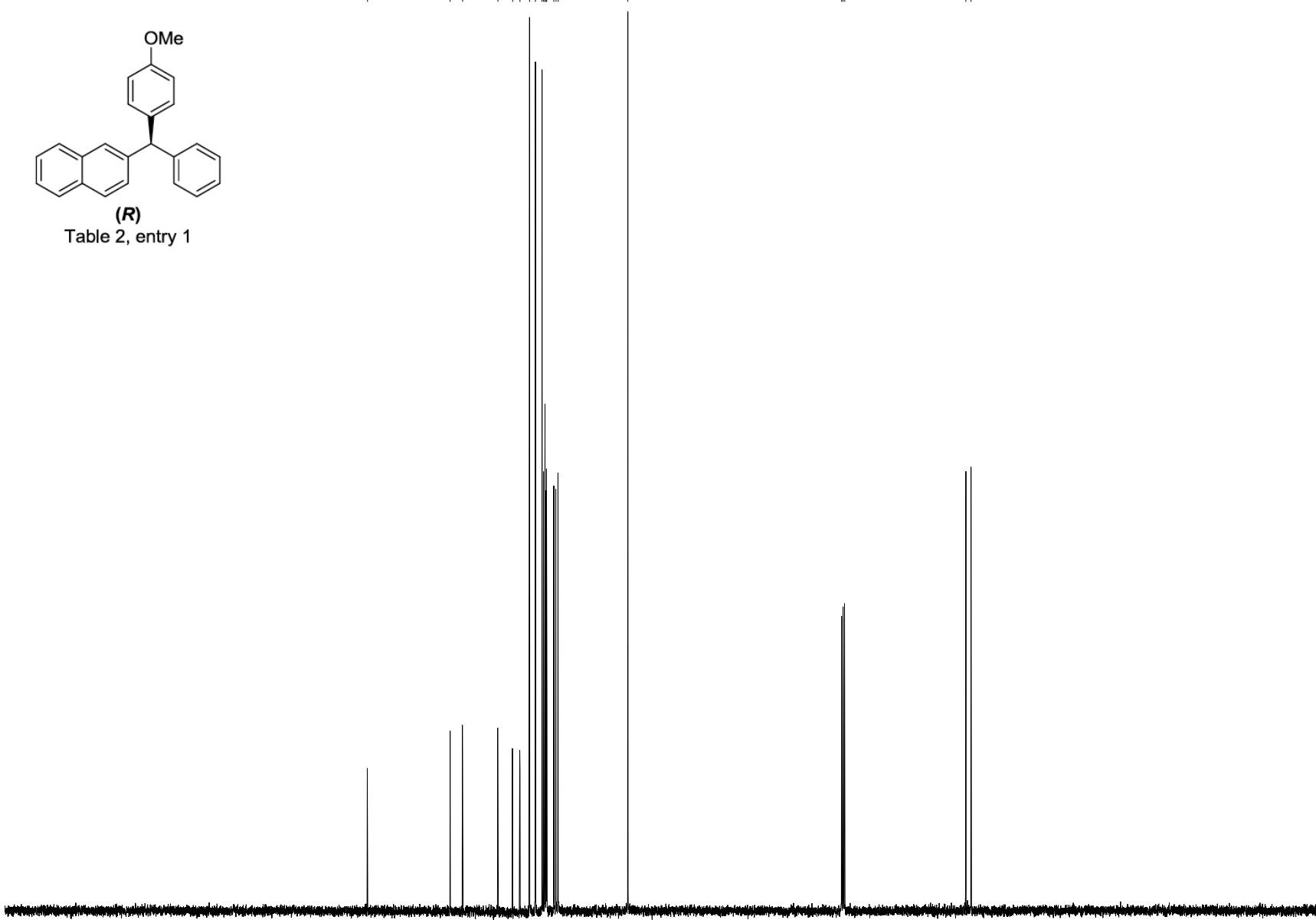
Z-restored spin-echo 13C spectrum with 1H decoupling

ppm



(R)
Table 2, entry 1

159.21
144.16
142.00
135.99
133.51
132.25
130.62
129.63
128.47
128.22
127.99
127.98
127.79
127.67
126.46
126.10
125.72
113.85
77.41
77.16
76.51
56.24
55.36



Current Data Parameters
USER mharri
NAME MRH-III-264-13CNMR
EXPNO 3
PROCNO 1

F2 - Acquisition Parameters
Date_ 20120928
Time 10.52
INSTRUM cryo500
PROBHD 5 mm CPTCI 1H-
PULPROG SpinEchopg30gp.prd
TD 65536
SOLVENT CDCl3T
NS 110
DS 16
SWH 30303.031 Hz
FIDRES 0.462388 Hz
AQ 1.0813940 sec
RG 11585.2
DW 16.500 usec
DE 6.00 usec
TE 298.0 K
D1 0.2500000 sec
d11 0.0300000 sec
D16 0.0002000 sec
d17 0.00019600 sec
MCREST 0.0000000 sec
MCMRK 0.01500000 sec
P2 31.00 usec

***** CHANNEL f1 *****
NUC1 13c
P1 15.50 usec
P11 500.00 usec
P12 2000.00 usec
PL0 120.00 dB
PL1 -1.00 dB
SFO1 125.7942548 MHz
SF1 3.20 dB
SF2 3.20 dB
SFOAM1 Crp60,0.5,20.1
SFOAM2 Crp60comp,4
SFOFF1 0.00 Hz
SFOFF2 0.00 Hz

***** CHANNEL f2 *****
CPDPRG2 waltz16
NUC2 1H
PCPD2 100.00 usec
PL2 1.60 dB
PL12 24.60 dB
SFO2 500.2225011 MHz

***** GRADIENT CHANNEL *****
GENAM1 SINE.100
GENAM2 SINE.100
GFX1 0.00 %
GFX2 0.00 %
GPY1 0.00 %
GPY2 0.00 %
GPZ1 30.00 %
GPZ2 50.00 %
p15 500.00 usec
p16 1000.00 usec

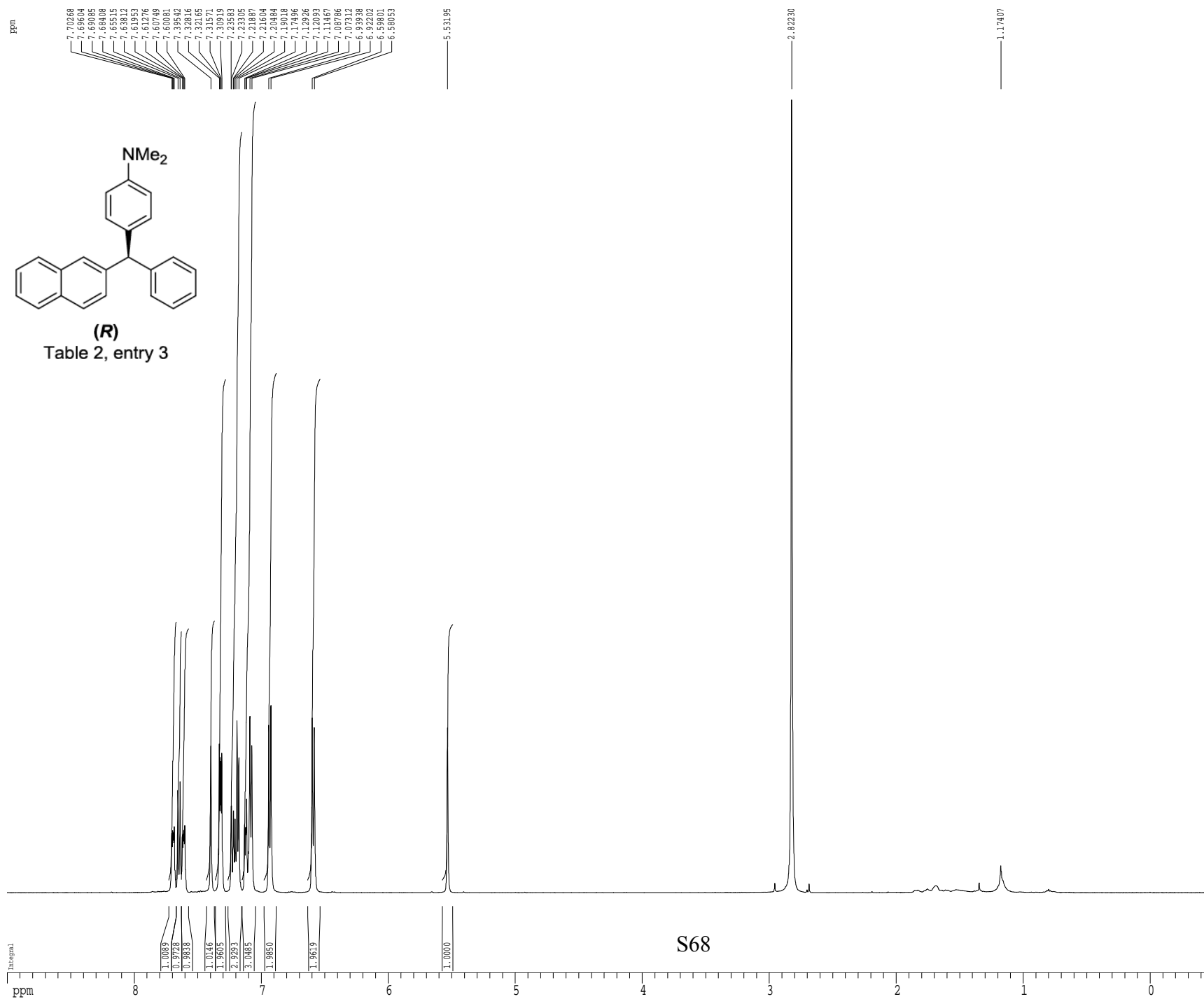
F2 - Processing parameters
SI 65536
SF 125.7804140 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 2.00

1D NMR plot parameters
CX 22.80 cm
CY 15.65 cm
F1P 220.000 ppm
F1 27671.69 Hz
F2P -5.000 ppm
F2 -628.90 Hz
PFMCM 9.86842 ppm/cm
HZCM 1241.25415 Hz/cm

S67

ppm 200 180 160 140 120 100 80 60 40 20 0

¹H spectrum



```

Current Data Parameters
USER          mharri
NAME          MRH-III-269-1HNMR
EXPNO        3
PROCNO       1

F2 - Acquisition Parameters
Date_        20120928
Time         10.55
INSTRUM      cryo500
PROBHD       5 mm CPTCI 1H-
PULPROG      zg30
TD            81728
SOLVENT      CDCl3
NS            1
DS            2
SWH           8012.820 Hz
FIDRES        0.098043 Hz
AQ            5.0998774 sec
RG            5
DW            62.400 usec
DE            6.00 usec
TE            298.0 K
D1            0.10000000 sec
MCREST        0.00000000 sec
MCNRK         0.01500000 sec

===== CHANNEL f1 =====
NUC1          1H
P1            7.50 usec
PL1           1.60 dB
SFO1          500.2235015 MHz

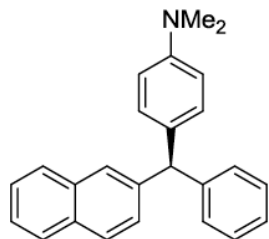
F2 - Processing parameters
SI            65536
SF            500.2201002 MHz
WDW           EM
SSB           0
LB            0.30 Hz
GB            0
PC            4.00

1D NMR plot parameters
CY            22.80 cm
CY            15.00 cm
F1            9.000 ppm
F2            4501.98 Hz
F2P           -0.500 ppm
F2            -250.11 Hz
PPMCM         0.41667 ppm/cm
HZCM          208.42505 Hz/cm
    
```

S68

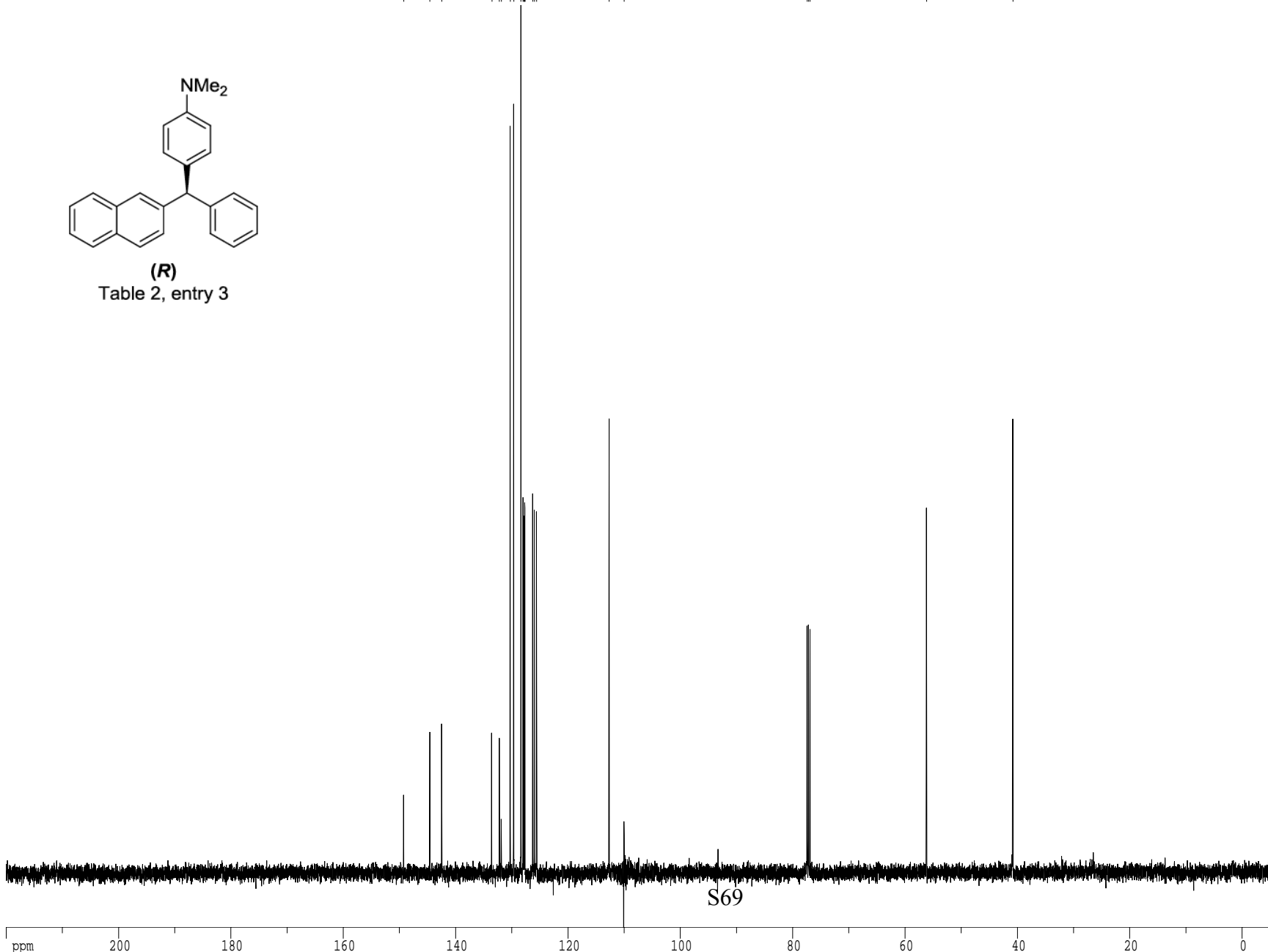
Z-restored spin-echo 13C spectrum with 1H decoupling

ppm



(R)

Table 2, entry 3



Current Data Parameters
 USER mharri
 NAME MRH-III-269-13CNMR
 EXPNO 3
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20120928
 Time 10.57
 INSTRUM cryo500
 PROBHD 5 mm CPTCI 1H-
 PULPROG SpinEchopg30gp.prd
 TD 65536
 SOLVENT CDCl3T
 NS 175
 DS 16
 SWH 30303.031 Hz
 FIDRES 0.462388 Hz
 AQ 1.0813940 sec
 RG 5
 DW 16.500 usec
 DE 6.00 usec
 TE 298.0 K
 DL 0.25000000 sec
 d11 0.03000000 sec
 d16 0.00020000 sec
 d17 0.00019600 sec
 MCREST 0.00000000 sec
 MCWRX 0.01500000 sec
 P2 31.00 usec

===== CHANNEL f1 =====
 NUC1 13c
 P1 15.50 usec
 P11 500.00 usec
 P12 2000.00 usec
 PL0 120.00 dB
 PL1 -1.00 dB
 SFO1 125.7942548 MHz
 SF1 3.20 dB
 SF2 3.20 dB
 SPNAM1 Crp60,0.5,20.1
 SPNAM2 Crp60comp,4
 SPOFF1 0.00 Hz
 SPOFF2 0.00 Hz

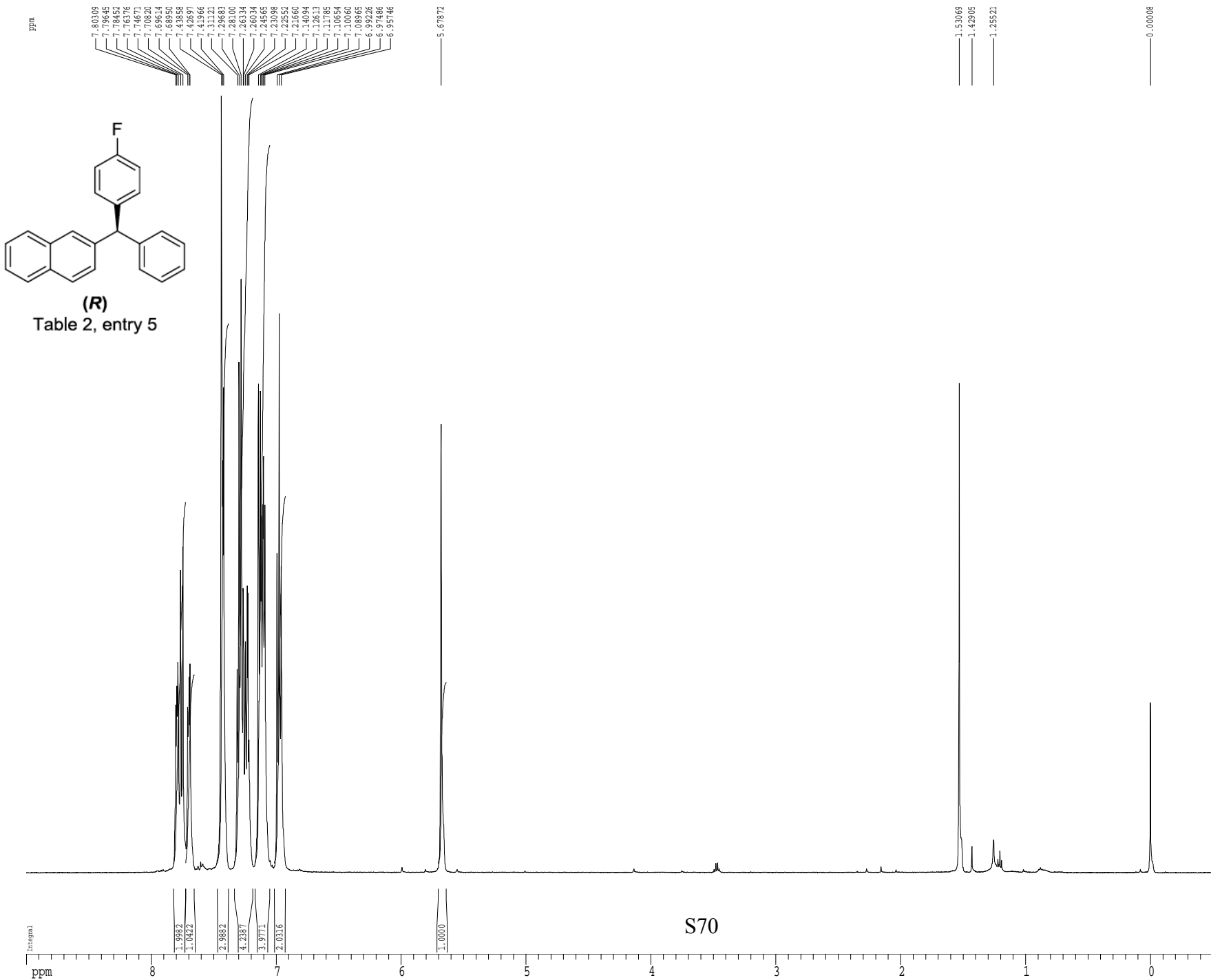
===== CHANNEL f2 =====
 CPDPRG2 waltz16
 NUC2 1H
 PCPD2 100.00 usec
 PL2 1.60 dB
 PL12 24.60 dB
 SFO2 500.2225011 MHz

===== GRADIENT CHANNEL =====
 GENAM1 SINE.100
 GENAM2 SINE.100
 GFX1 0.00 %
 GFX2 0.00 %
 GPY1 0.00 %
 GPY2 0.00 %
 GPZ1 30.00 %
 GPZ2 50.00 %
 p15 500.00 usec
 p16 1000.00 usec

F2 - Processing parameters
 SI 65536
 SF 125.7804168 MHz
 HN
 SSB 0
 LB 1.00 Hz
 GB 0
 PC 2.00

1D NMR plot parameters
 CX 22.80 cm
 CY 15.65 cm
 F1P 220.000 ppm
 F1 27671.69 Hz
 F2P -5.000 ppm
 F2 -628.90 Hz
 PFMCM 9.86842 ppm/cm
 HZCM 1241.25415 Hz/cm

¹H spectrum



Current Data Parameters
USER mharri
NAME MRH-III-275-1HNMR
EXPNO 2
PROCNO 1

F2 - Acquisition Parameters
Date_ 20120925
Time 19.34
INSTRUM cryo500
PROBHD 5 mm CPTCI 1H-
PULPROG zg30
TD 81728
SOLVENT CDCl3T
NS 1
DS 2
SWH 8012.820 Hz
FIDRES 0.098043 Hz
AQ 5.0998774 sec
RG 5
DW 62.400 usec
DE 6.00 usec
TE 298.0 K
D1 0.10000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec

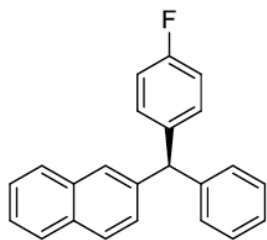
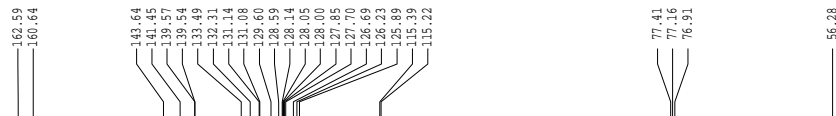
===== CHANNEL f1 =====
NUC1 1H
P1 7.50 usec
PL1 1.60 dB
SFO1 500.2235015 MHz

F2 - Processing parameters
SI 65536
SF 500.2200480 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 4.00

1D NMR plot parameters
CX 22.80 cm
CY 15.00 cm
F1P 9.000 ppm
F1 4501.98 Hz
F2P -0.500 ppm
F2 -250.11 Hz
PPMCM 0.41667 ppm/cm
HZCM 208.42503 Hz/cm

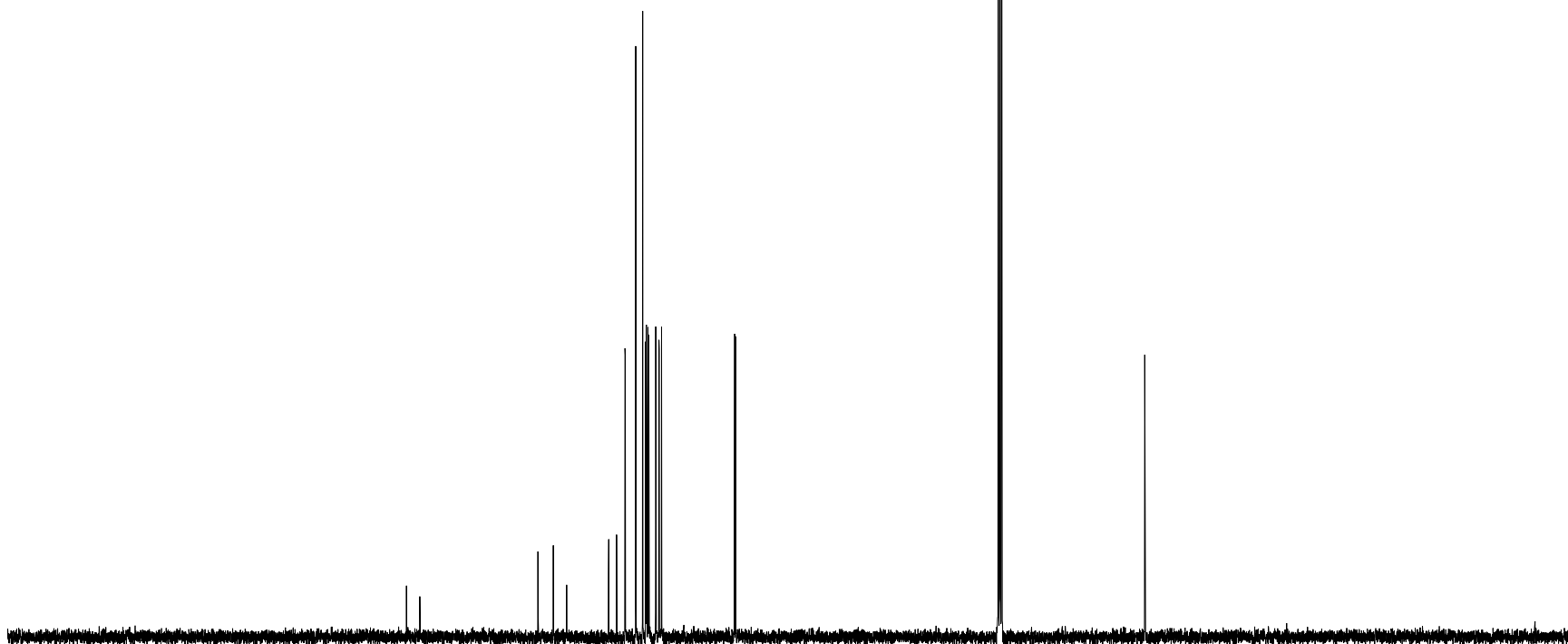
Z-restored spin-echo 13C spectrum with 1H decoupling

ppm



(R)

Table 2, entry 5



```

Current Data Parameters
USER          mharri
NAME          MRH-II-101-A-C13CNMR
EXPNO        2
PROCNO       1

F2 - Acquisition Parameters
Date_        20111130
Time         15.11
INSTRUM      cryo500
PROBHD       5 mm CP131H-
PULPROG      SpinEchoqs30sp.prd
TD           65536
SOLVENT      CDCl3
NS           968
DS           16
SWH          30303.031 Hz
FIDRES       0.462398 Hz
AQ           1.0813940 sec
RG           11585.2
DW           16.500 usec
DE           6.00 usec
TE           298.0 K
d1           0.25000000 sec
d11          0.03000000 sec
d16          0.00020000 sec
d17          0.00019600 sec
MCREST       0.00000000 sec
MCMRCK       0.01500000 sec
P2           31.00 usec

===== CHANNEL f1 =====
NUC1         13C
P1           15.50 usec
P11          500.00 usec
P12          2000.00 usec
PL0          120.00 dB
PL1          -1.00 dB
SP01         125.7942548 MHz
SP1          3.20 dB
SP2          3.20 dB
SPNAM1       Crp60,0.5,20.1
SPNAM2       Crp60comp.4
SPOFF1       0.00 Hz
SPOFF2       0.00 Hz

===== CHANNEL f2 =====
CFDPRG2      waltz16
NUC2         1H
PCPD2        100.00 usec
PL2          1.60 dB
PL12         24.60 dB
SFO2         500.2225011 MHz

===== GRADIENT CHANNEL =====
GPNAM1       SINE.100
GPNAM2       SINE.100
GPX1         0.00 %
GPX2         0.00 %
GPY1         0.00 %
GPY2         0.00 %
GPZ1         30.00 %
GPZ2         50.00 %
p15          500.00 usec
p16          1000.00 usec

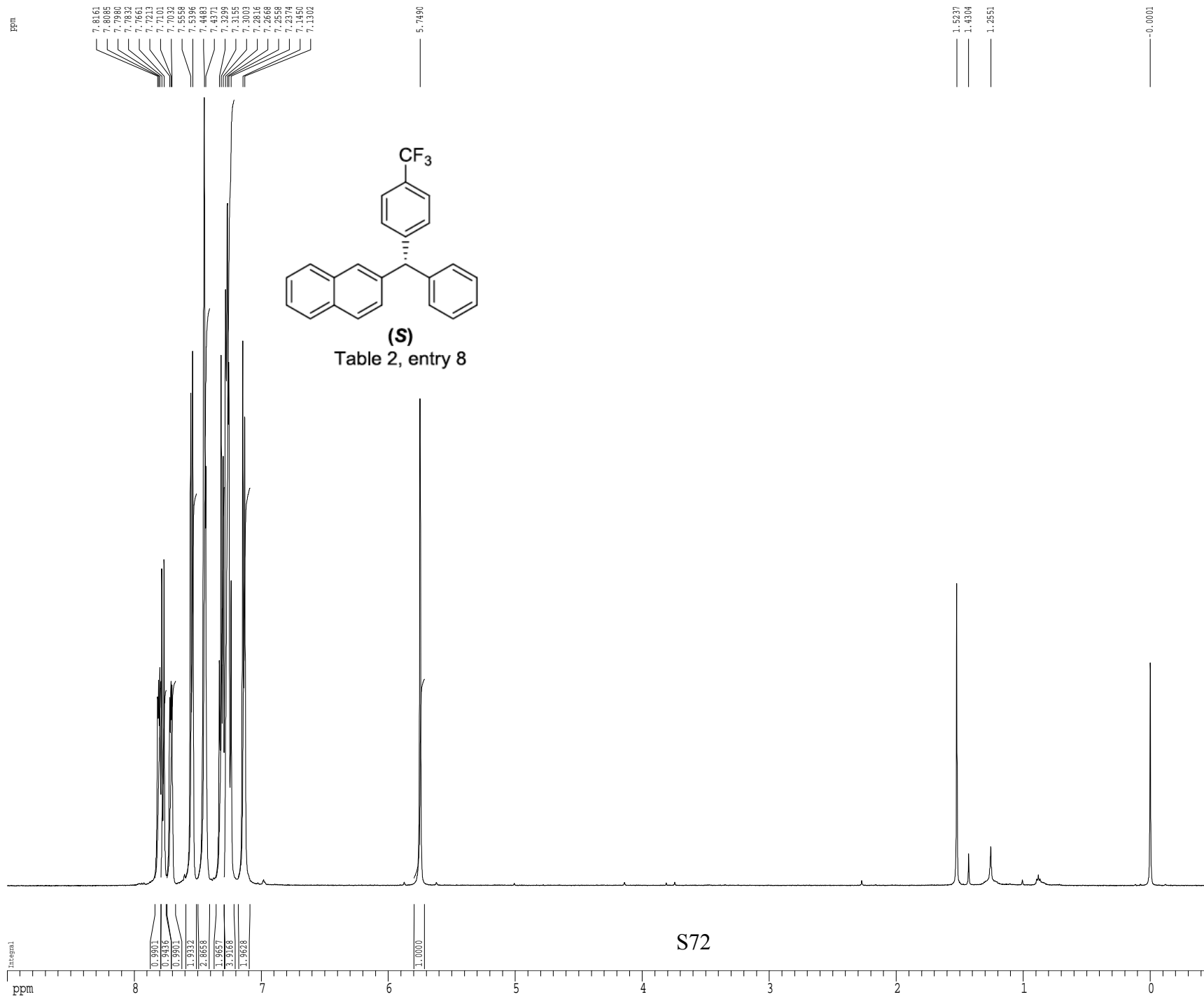
F2 - Processing parameters
SI           65536
SF           125.7804090 MHz
WDW          EM
SSB          0
LB           1.00 Hz
GB           0
PC           2.00

ID NMR plot parameters
CX           22.80 cm
CY           15.65 cm
F1P          220.000 ppm
F1           27671.69 Hz
F2P          -5.000 ppm
F2           -628.90 Hz
PPMCM        9.86842 ppm/cm
HZCM         1241.25415 Hz/cm
    
```

S71

ppm 200 180 160 140 120 100 80 60 40 20 0

¹H spectrum



```

Current Data Parameters
USER          mharri
NAME          MRH-IV-29-1HNMR
EXPNO        2
PROCNO       1

F2 - Acquisition Parameters
Date_        20121019
Time         14.43
INSTRUM      cryo500
PROBHD       5 mm CPTCI 1H-
PULPROG      zg30
TD           81728
SOLVENT      CDCl3
NS           3
DS           2
SWH          8012.820 Hz
FIDRES       0.098043 Hz
AQ           5.0998774 sec
RG           4.5
DW           62.400 usec
DE           6.00 usec
TE           298.0 K
DL           0.10000000 sec
MCREST       0.00000000 sec
MCWRX        0.01500000 sec

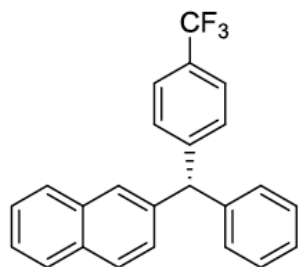
===== CHANNEL f1 =====
NUC1         1H
P1           7.50 usec
PL1          1.60 dB
SF01         500.2235015 MHz

F2 - Processing parameters
SI           65536
SF           500.2200421 MHz
WDW          EM
SSB          0
LB           0.30 Hz
GB           0
PC           4.00

1D NMR plot parameters
CX           22.80 cm
CY           15.00 cm
FLP          9.000 ppm
F1           4501.98 Hz
F2P         -0.500 ppm
F2           -250.11 Hz
PPMCM       0.41667 ppm/cm
HZCM        208.42502 Hz/cm
    
```

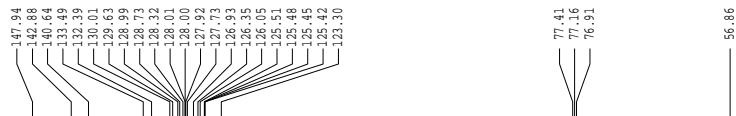

Z-restored spin-echo 13C spectrum with 1H decoupling

ppm



(S)

Table 2, entry 8



Current Data Parameters
 USER mharri
 NAME MRH-IV-29-13CNMR
 EXPNO 2
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20121019
 Time 14.46
 INSTRUM cryo500
 PROBHD 5 mm CPTCI 1H-
 PULPROG SpinEchopg30gp.prd
 TD 65536
 SOLVENT CDCl3T
 NS 317
 DS 16
 SWH 30303.031 Hz
 FIDRES 0.462388 Hz
 AQ 1.081394 sec
 RG 7298.2
 DW 16.500 usec
 DE 6.00 usec
 TE 298.0 K
 D1 0.2500000 sec
 d11 0.0300000 sec
 D16 0.0002000 sec
 d17 0.0001960 sec
 MCREST 0.0000000 sec
 MCMRK 0.0150000 sec
 P2 31.00 usec

***** CHANNEL f1 *****
 NUC1 13c
 P1 15.50 usec
 P11 500.00 usec
 P12 2000.00 usec
 PL0 120.00 dB
 PL1 -1.00 dB
 SFO1 125.7942548 MHz
 SF1 3.20 dB
 SF2 3.20 dB
 SPNAM1 Crp60,0.5,20.1
 SPNAM2 Crp60comp,4
 SPOFF1 0.00 Hz
 SPOFF2 0.00 Hz

***** CHANNEL f2 *****
 CPDPRG2 waltz16
 NUC2 1H
 PCPD2 100.00 usec
 PL2 1.60 dB
 PL12 24.60 dB
 SFO2 500.2225011 MHz

***** GRADIENT CHANNEL *****
 GENAM1 SINE.100
 GENAM2 SINE.100
 GFX1 0.00 %
 GFX2 0.00 %
 GPY1 0.00 %
 GPY2 0.00 %
 GPZ1 30.00 %
 GPZ2 50.00 %
 p15 500.00 usec
 p16 1000.00 usec

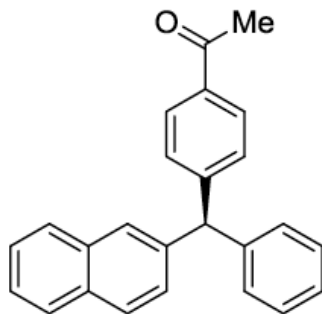
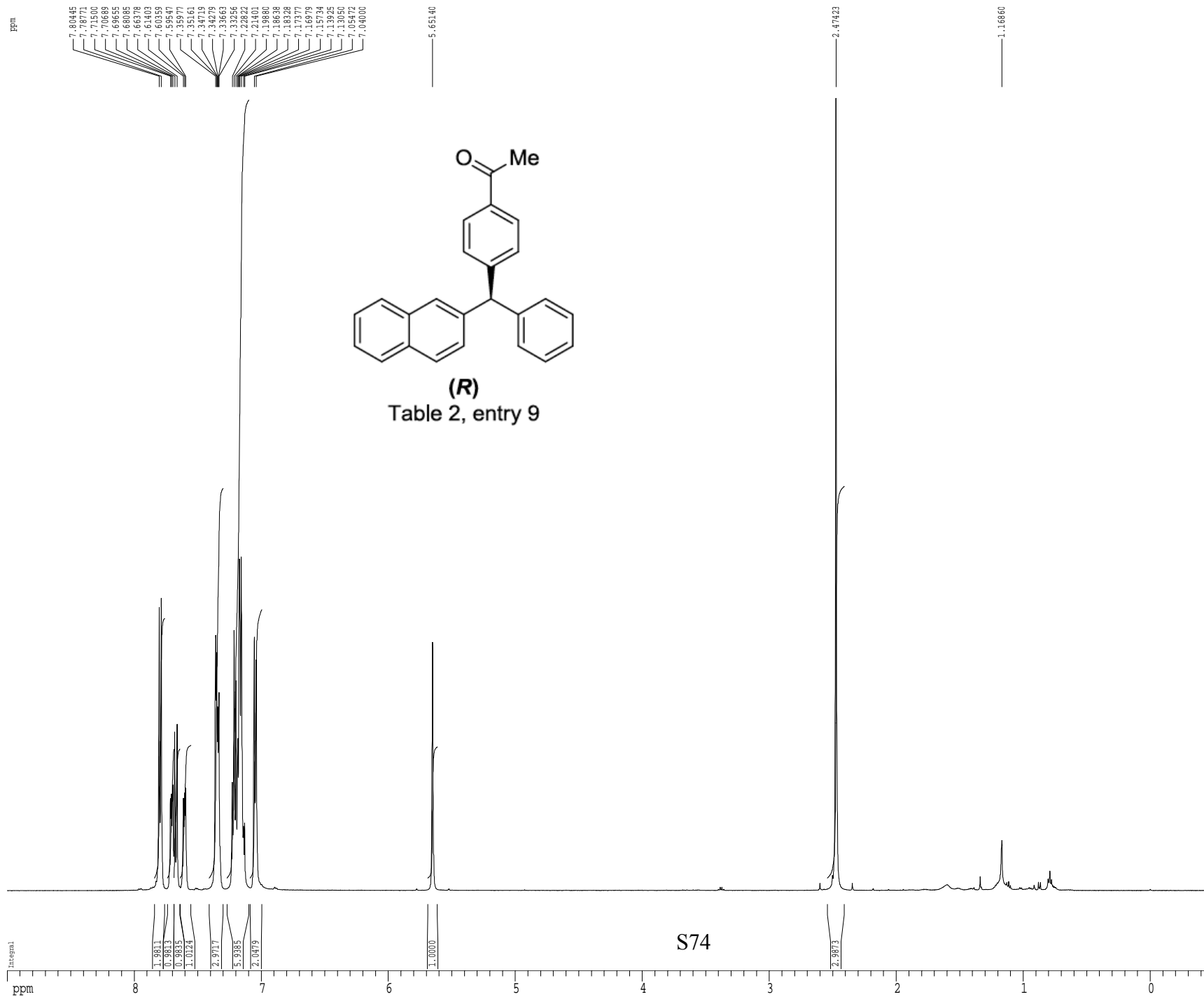
F2 - Processing parameters
 SI 65536
 SF 125.7804099 MHz
 WDW EM
 SSB 0
 LB 1.00 Hz
 GB 0
 PC 2.00

1D NMR plot parameters
 CX 22.80 cm
 CY 15.65 cm
 F1P 220.000 ppm
 F1 27671.69 Hz
 F2P -5.000 ppm
 F2 -628.90 Hz
 PPMCM 9.86842 ppm/cm
 HZCM 1241.25415 Hz/cm

ppm 200 180 160 140 120 100 80 60 40 20 0

S73

¹H spectrum



(R)
Table 2, entry 9

Current Data Parameters
 USER mharri
 NAME MRH-III-283-1HNMR
 EXPNO 3
 PROCNO 1

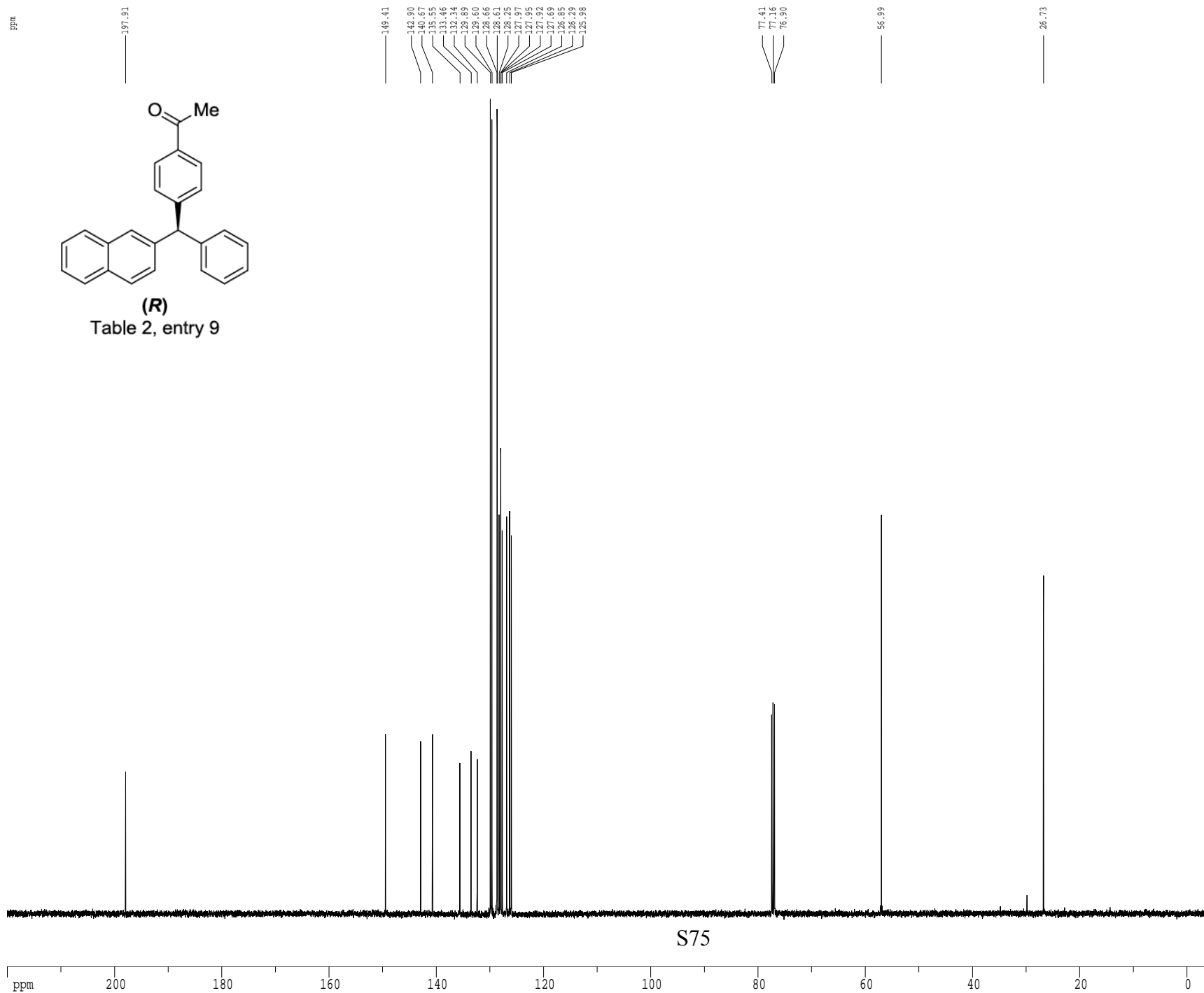
F2 - Acquisition Parameters
 Date_ 20120928
 Time 11.10
 INSTRUM cryo500
 PROBD 5 mm CPTCI 1H-
 PULPROG zg30
 TD 81728
 SOLVENT CDCl3
 NS 1
 DS 2
 SWH 8012.820 Hz
 FIDRES 0.098043 Hz
 AQ 5.0998774 sec
 RG 5
 DW 62.400 usec
 DE 6.00 usec
 TE 298.0 K
 D1 0.10000000 sec
 MCREST 0.00000000 sec
 MCNRK 0.01500000 sec

===== CHANNEL f1 =====
 NUC1 1H
 P1 7.50 usec
 PL1 1.60 dB
 SFO1 500.2235015 MHz

F2 - Processing parameters
 SI 65536
 SF 500.2200951 MHz
 WDW EM
 SSB 0
 LB 0.30 Hz
 GB 0
 PC 4.00

1D NMR plot parameters
 CX 22.80 cm
 CY 15.00 cm
 F1 9.000 ppm
 F2P 4501.98 Hz
 F2 -250.11 Hz
 PPMCM 0.41667 ppm/cm
 HZCM 208.42505 Hz/cm

Z-restored spin-echo 13C spectrum with 1H decoupling



Current Data Parameters
 USER mharri
 NAME MRH-III-283-13CNMR
 EXPNO 3
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20120928
 Time 11.13
 INSTRUM cryo500
 PROBHD 5 mm CPTCI 1H-
 PULPROG SpinEchopg30gp.prd
 TD 65536
 SOLVENT CDCl3T
 NS 106
 DS 16
 SWH 30303.031 Hz
 FIDRES 0.462388 Hz
 AQ 1.0813940 sec
 RG 7298.2
 DW 16.500 usec
 DE 6.00 usec
 TE 298.0 K
 D1 0.2500000 sec
 d11 0.03000000 sec
 D16 0.00020000 sec
 d17 0.00019600 sec
 MCREST 0.00000000 sec
 MCNRX 0.01500000 sec
 P2 31.00 usec

===== CHANNEL f1 =====
 NUC1 13c
 P1 15.50 usec
 P11 500.00 usec
 P12 2000.00 usec
 PL0 120.00 dB
 PL1 -1.00 dB
 SFO1 125.7942548 MHz
 SF1 3.20 dB
 SF2 3.20 dB
 SFOAM1 Crp60,0.5,20.1
 SFOAM2 Crp60comp,4
 SFOFF1 0.00 Hz
 SFOFF2 0.00 Hz

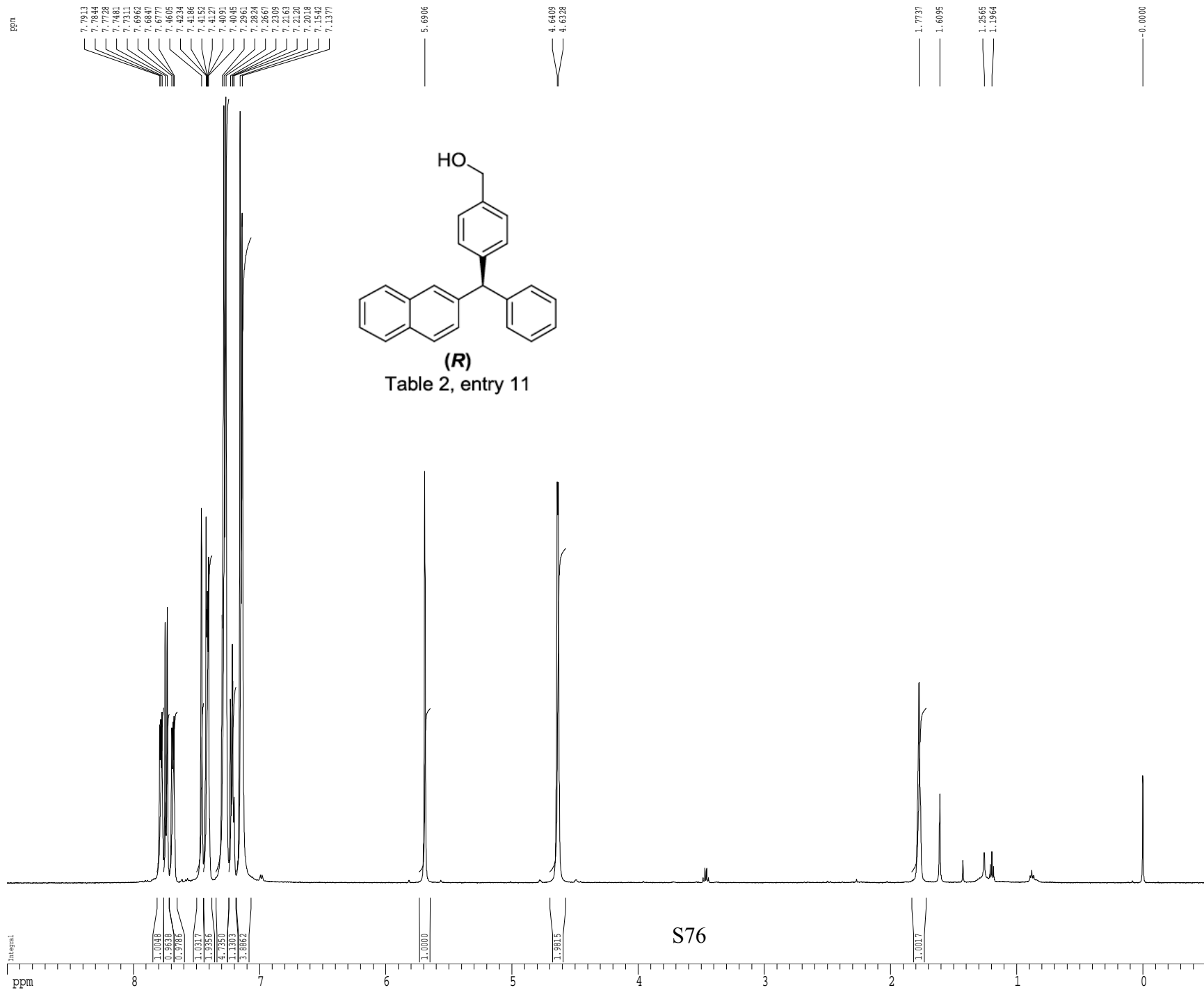
===== CHANNEL f2 =====
 CPDPRG2 waltz16
 NUC2 1H
 PCPD2 100.00 usec
 PL2 1.60 dB
 PL12 24.60 dB
 SFO2 500.2225011 MHz

===== GRADIENT CHANNEL =====
 GENAM1 SINE.100
 GENAM2 SINE.100
 GFX1 0.00 %
 GFX2 0.00 %
 GPY1 0.00 %
 GPY2 0.00 %
 GPZ1 30.00 %
 GPZ2 50.00 %
 p15 500.00 usec
 p16 1000.00 usec

F2 - Processing parameters
 SI 65536
 SF 125.7804177 MHz
 WDW EM
 SSB 0
 LB 1.00 Hz
 GB 0
 PC 2.00

1D NMR plot parameters
 CX 22.80 cm
 CY 15.65 cm
 F1P 220.000 ppm
 F1 27671.69 Hz
 F2P -5.000 ppm
 F2 -628.90 Hz
 PPMCM 9.86842 ppm/cm
 HZCM 1241.25415 Hz/cm

¹H spectrum



Current Data Parameters
 USER mharr
 NAME MRH-IV-26-1HNMR
 EXPNO 2
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20121019
 Time 14:34
 INSTRUM cryo500
 PROBHD 5 mm CPTCI 1H-
 PULPROG zg30
 TD 81728
 SOLVENT CDCl3
 NS 1
 DS 2
 SWH 8012.820 Hz
 FIDRES 0.098043 Hz
 AQ 5.0998774 sec
 RG 3.2
 DW 62.400 usec
 DE 6.00 usec
 TE 298.0 K
 DL 0.10000000 sec
 MCREST 0.00000000 sec
 MCWEX 0.01500000 sec

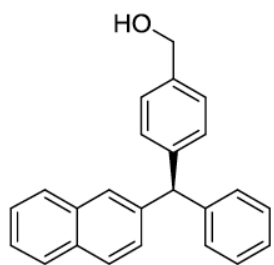
===== CHANNEL f1 =====
 NUC1 1H
 P1 7.50 usec
 PL1 1.60 dB
 SF01 500.2235015 MHz

F2 - Processing parameters
 SI 65536
 SF 500.2200546 MHz
 WDW EM
 SSB 0
 LB 0.30 Hz
 GB 0
 PC 4.00

1D NMR plot parameters
 CX 22.80 cm
 CY 15.00 cm
 F1P 9.000 ppm
 F1 4501.98 Hz
 F2P -0.500 ppm
 F2 -250.11 Hz
 PPMCM 0.41667 ppm/cm
 HZCM 208.42503 Hz/cm

Z-restored spin-echo 13C spectrum with 1H decoupling

ppm



(R)

Table 2, entry 11

143.70
143.36
141.51
139.48
139.06
132.27
129.88
129.64
128.52
128.14
128.04
127.98
127.86
127.67
127.26
126.57
126.14
125.79

77.41
77.16
76.31

65.22

56.78

Current Data Parameters
USER mharri
NAME MRH-IV-26-13CNMR
EXPNO 2
PROCNO 1

F2 - Acquisition Parameters
Date_ 20121019
Time 14.36
INSTRUM cryo500
PROBHD 5 mm CPTCI 1H-
PULPROG SpinEchopg30gp.prd
TD 65536
SOLVENT CDCl3T
NS 101
DS 16
SWH 30303.031 Hz
FIDRES 0.462388 Hz
AQ 1.0813940 sec
RG 4096
DW 16.500 usec
DE 6.00 usec
TE 298.0 K
D1 0.25000000 sec
d11 0.03000000 sec
D16 0.00020000 sec
d17 0.00019600 sec
MCREST 0.00000000 sec
MCMRK 0.01500000 sec
P2 31.00 usec

***** CHANNEL f1 *****
NUC1 13c
P1 15.50 usec
P11 500.00 usec
P12 2000.00 usec
PL0 120.00 dB
PL1 -1.00 dB
SFO1 125.7942548 MHz
SF1 3.20 dB
SF2 3.20 dB
SFOAM1 Crp60,0.5,20.1
SFOAM2 Crp60comp,4
SFOFF1 0.00 Hz
SFOFF2 0.00 Hz

***** CHANNEL f2 *****
CPDPRG2 waltz16
NUC2 1H
PCPD2 100.00 usec
PL2 1.60 dB
PL12 24.60 dB
SFO2 500.2225011 MHz

***** GRADIENT CHANNEL *****
GENAM1 SINE.100
GENAM2 SINE.100
GFX1 0.00 %
GFX2 0.00 %
GPY1 0.00 %
GPY2 0.00 %
GPZ1 30.00 %
GPZ2 50.00 %
p15 500.00 usec
p16 1000.00 usec

F2 - Processing parameters
SI 65536
SF 125.7804168 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 2.00

1D NMR plot parameters
CX 22.80 cm
CY 15.65 cm
F1P 220.000 ppm
F1 27671.69 Hz
F2P -5.000 ppm
F2 -628.90 Hz
PFMCM 9.86842 ppm/cm
HZCM 1241.25415 Hz/cm

ppm

200

180

160

140

120

100

80

60

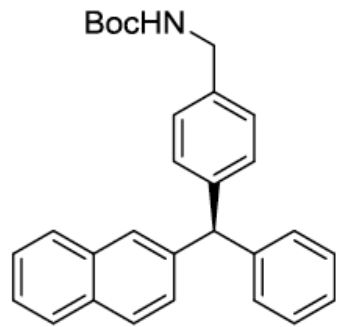
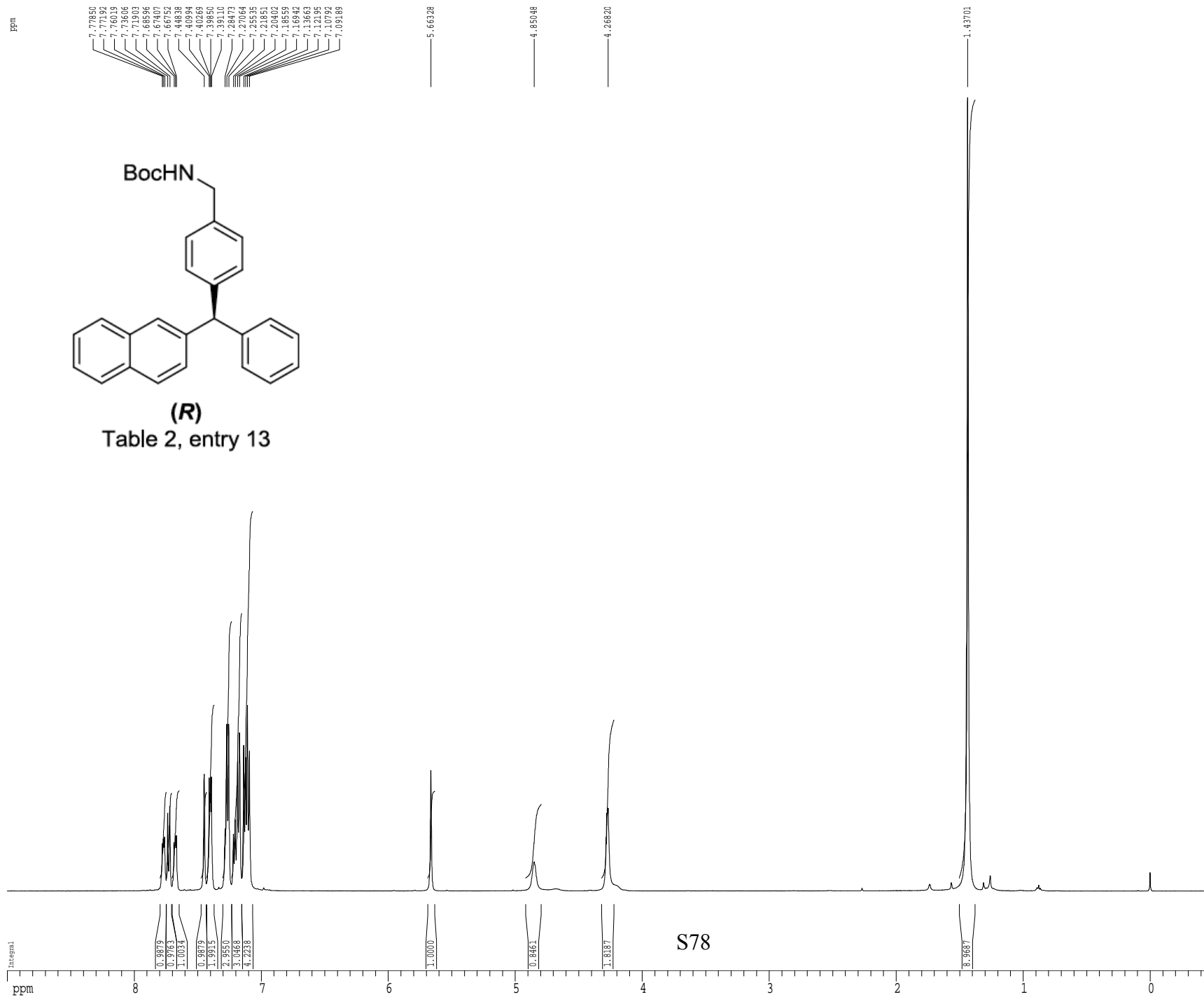
40

20

0

S77

¹H spectrum



(R)
Table 2, entry 13

```

Current Data Parameters
USER          mharri
NAME          MRH-III-294-1HNMR
EXPNO        1
PROCNO       1

F2 - Acquisition Parameters
Date_        20121015
Time         15.33
INSTRUM      cryo500
PROBHD       5 mm CPTCI 1H-
PULPROG      zg30
TD           81728
SOLVENT      CDCl3
NS           1
DS           2
SWH          8012.820 Hz
FIDRES       0.098043 Hz
AQ           5.0998774 sec
RG           5
DW           62.400 usec
DE           6.00 usec
TE           298.0 K
D1           0.10000000 sec
MCREST       0.00000000 sec
MCNRK        0.01500000 sec

===== CHANNEL f1 =====
NUC1         1H
P1           7.50 usec
PL1          1.60 dB
SFO1         500.2235015 MHz

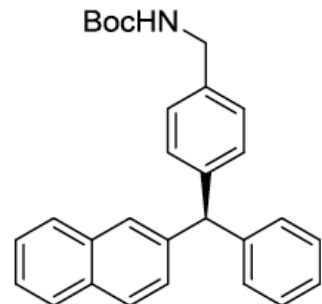
F2 - Processing parameters
SI           65536
SF           500.2200672 MHz
WDW          EM
SSB          0
LB           0.30 Hz
GB           0
PC           4.00

1D NMR plot parameters
CY           22.80 cm
CY           15.00 cm
F1           9.000 ppm
F2           4501.98 Hz
F2P          -0.500 ppm
F2           -250.11 Hz
PPMCM        0.41667 ppm/cm
HZCM         208.42503 Hz/cm
    
```

S78

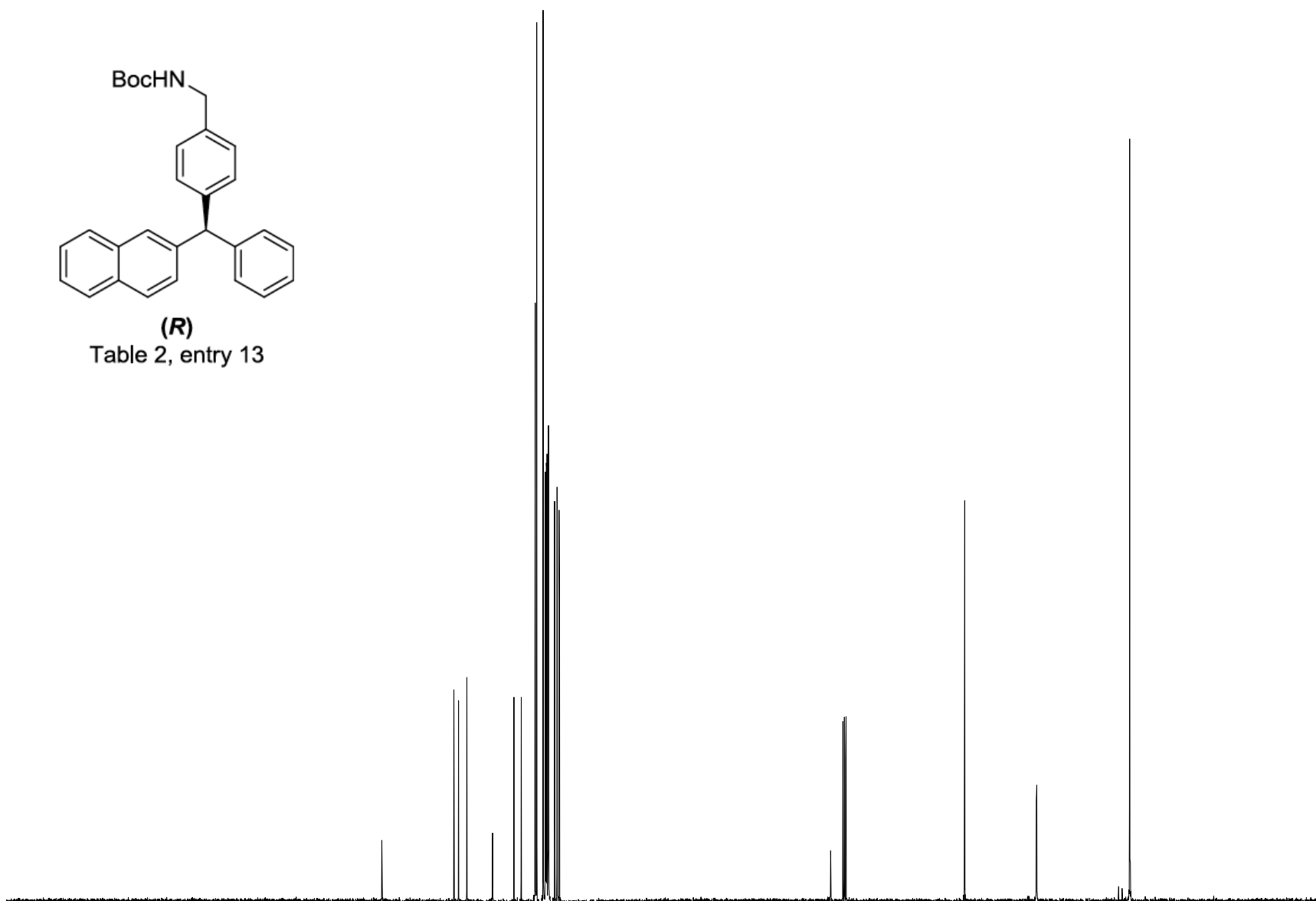
Z-restored spin-echo 13C spectrum with 1H decoupling

ppm



(R)

Table 2, entry 13



Current Data Parameters
 USER mharri
 NAME MRH-III-294-13CNMR
 EXPNO 1
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20121015
 Time 15.35
 INSTRUM cryo500
 PROBHD 5 mm CPTCI 1H-
 PULPROG SpinEchopg30gp.prd
 TD 65536
 SOLVENT CDCl3T
 NS 101
 DS 16
 SWH 30303.031 Hz
 FIDRES 0.462388 Hz
 AQ 1.0813940 sec
 RG 5792.6
 DW 16.500 usec
 DE 6.00 usec
 TE 298.0 K
 D1 0.25000000 sec
 d11 0.03000000 sec
 D16 0.00020000 sec
 d17 0.00019600 sec
 MCREST 0.00000000 sec
 MCMRK 0.01500000 sec
 P2 31.00 usec

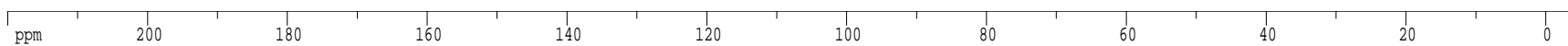
***** CHANNEL f1 *****
 NUC1 13c
 P1 15.50 usec
 P11 500.00 usec
 P12 2000.00 usec
 PL0 120.00 dB
 PL1 -1.00 dB
 SFO1 125.7942548 MHz
 SF1 3.20 dB
 SP2 3.20 dB
 SFOAM1 Crp60,0.5,20.1
 SFOAM2 Crp60comp,4
 SPOFF1 0.00 Hz
 SPOFF2 0.00 Hz

***** CHANNEL f2 *****
 CPDPRG2 waltz16
 NUC2 1H
 PCPD2 100.00 usec
 PL2 1.60 dB
 PL12 24.60 dB
 SFO2 500.2225011 MHz

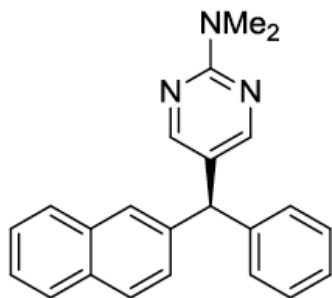
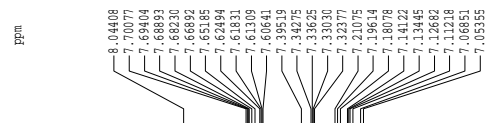
***** GRADIENT CHANNEL *****
 GENAM1 SINE.100
 GENAM2 SINE.100
 GFX1 0.00 %
 GFX2 0.00 %
 GPY1 0.00 %
 GPY2 0.00 %
 GPZ1 30.00 %
 GPZ2 50.00 %
 p15 500.00 usec
 p16 1000.00 usec

F2 - Processing parameters
 SI 65536
 SF 125.7804238 MHz
 HN
 WDW EM
 SSB 0
 LB 1.00 Hz
 GB 0
 PC 2.00

1D NMR plot parameters
 CX 22.80 cm
 CY 15.65 cm
 F1 27671.70 Hz
 F2P -5.000 ppm
 F2 -628.90 Hz
 PPMCM 9.86842 ppm/cm
 HZCM 1241.25427 Hz/cm

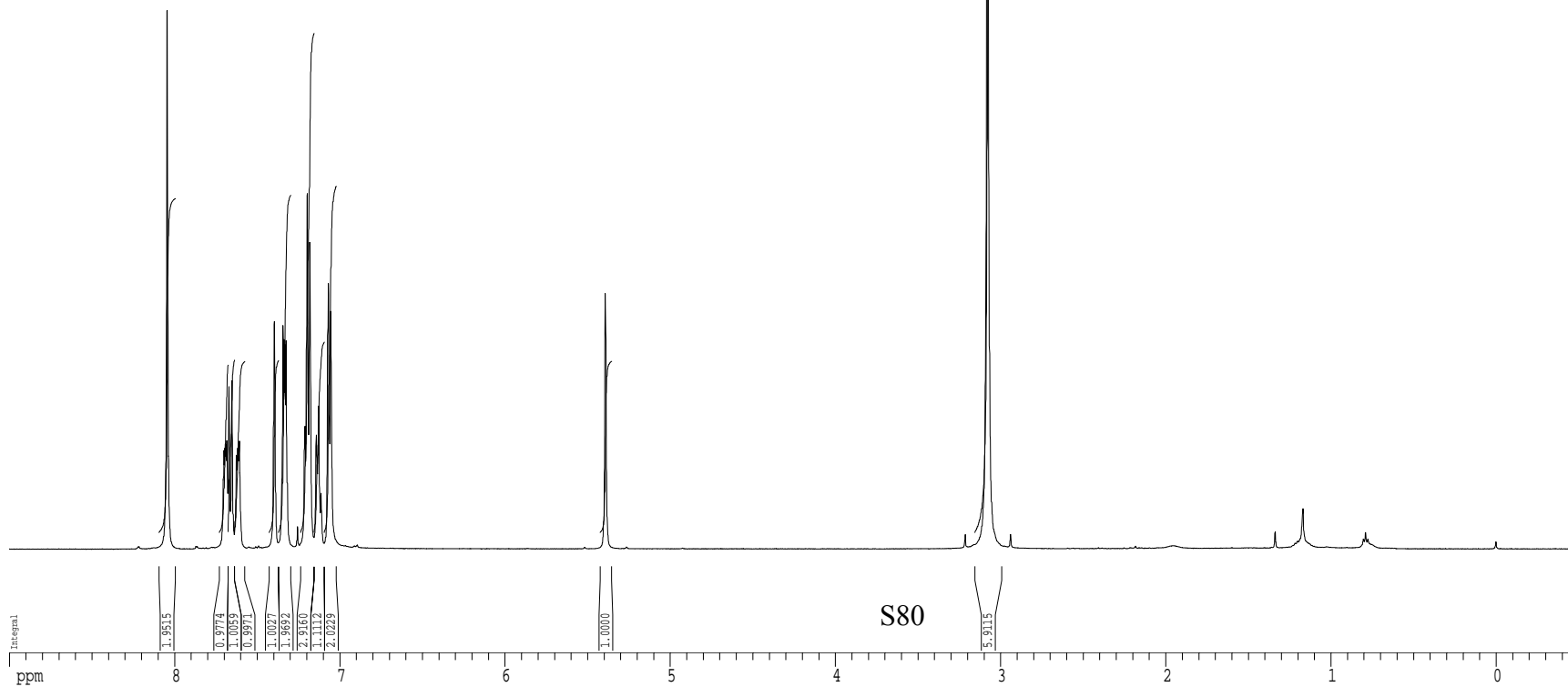


¹H spectrum



(S)

Table 2, entry 15



Current Data Parameters
 USER mharri
 NAME MRH-III-285-1HNMR
 EXPNO 3
 PROCNO 1

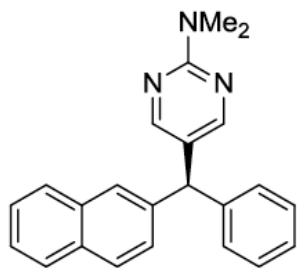
F2 - Acquisition Parameters
 Date_ 20120928
 Time 11.16
 INSTRUM cryo500
 PROBHD 5 mm CPXI 1H-
 PULPROG zg30
 TD 81728
 SOLVENT CDCl3
 DS 2
 NS 1
 SWH 8012.820 Hz
 FIDRES 0.098043 Hz
 AQ 5.0998774 sec
 RG 4.5
 DW 62.400 usec
 DE 6.00 usec
 TE 298.0 K
 D1 0.10000000 sec
 MCREST 0.00000000 sec
 MCNRK 0.01500000 sec

===== CHANNEL f1 =====
 NUC1 1H
 P1 7.50 usec
 PL1 1.60 dB
 SFO1 500.2235015 MHz

F2 - Processing parameters
 SI 65536
 SF 500.2200930 MHz
 WDW EM
 SSB 0
 LB 0.30 Hz
 GB 0
 PC 4.00

1D NMR plot parameters
 CX 22.80 cm
 CY 15.00 cm
 FIP 9.000 ppm
 F1 4501.98 Hz
 F2P -0.500 ppm
 F2 -250.11 Hz
 PPMCM 0.41667 ppm/cm
 HZCM 208.42505 Hz/cm

Z-restored spin-echo 13C spectrum with 1H decoupling



(S)

Table 2, entry 15

Current Data Parameters
 USER mharri
 NAME MRH-III-285-13CNMR
 EXPNO 3
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20120928
 Time 11.19
 INSTRUM cryo500
 PROBHD 5 mm CPTCI 1H-
 PULPROG SpinEchopg30gp.prd
 TD 65536
 SOLVENT CDCl3T
 NS 109
 DS 16
 SWH 30303.031 Hz
 FIDRES 0.462388 Hz
 AQ 1.0813940 sec
 RG 7298.2
 DW 16.500 usec
 DE 6.00 usec
 TE 298.0 K
 D1 0.25000000 sec
 d11 0.03000000 sec
 D16 0.00020000 sec
 d17 0.00019600 sec
 MCREST 0.00000000 sec
 MCWPK 0.01500000 sec
 P2 31.00 usec

***** CHANNEL f1 *****
 NUC1 13c
 P1 15.50 usec
 P11 500.00 usec
 P12 2000.00 usec
 PL0 120.00 dB
 PL1 -1.00 dB
 SFO1 125.7942548 MHz
 SF1 3.20 dB
 SF2 3.20 dB
 SPNAM1 Crp60,0.5,20.1
 SPNAM2 Crp60comp,4
 SPOFF1 0.00 Hz
 SPOFF2 0.00 Hz

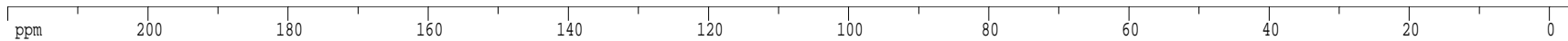
***** CHANNEL f2 *****
 CPDPRG2 waltz16
 NUC2 1H
 PCPD2 100.00 usec
 PL2 1.60 dB
 PL12 24.60 dB
 SFO2 500.2225011 MHz

***** GRADIENT CHANNEL *****
 GENAM1 SINE.100
 GENAM2 SINE.100
 GFX1 0.00 %
 GFX2 0.00 %
 GPY1 0.00 %
 GPY2 0.00 %
 GPZ1 30.00 %
 GPZ2 50.00 %
 p15 500.00 usec
 p16 1000.00 usec

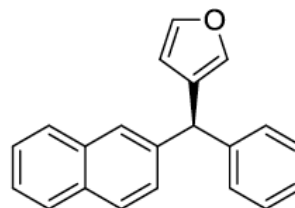
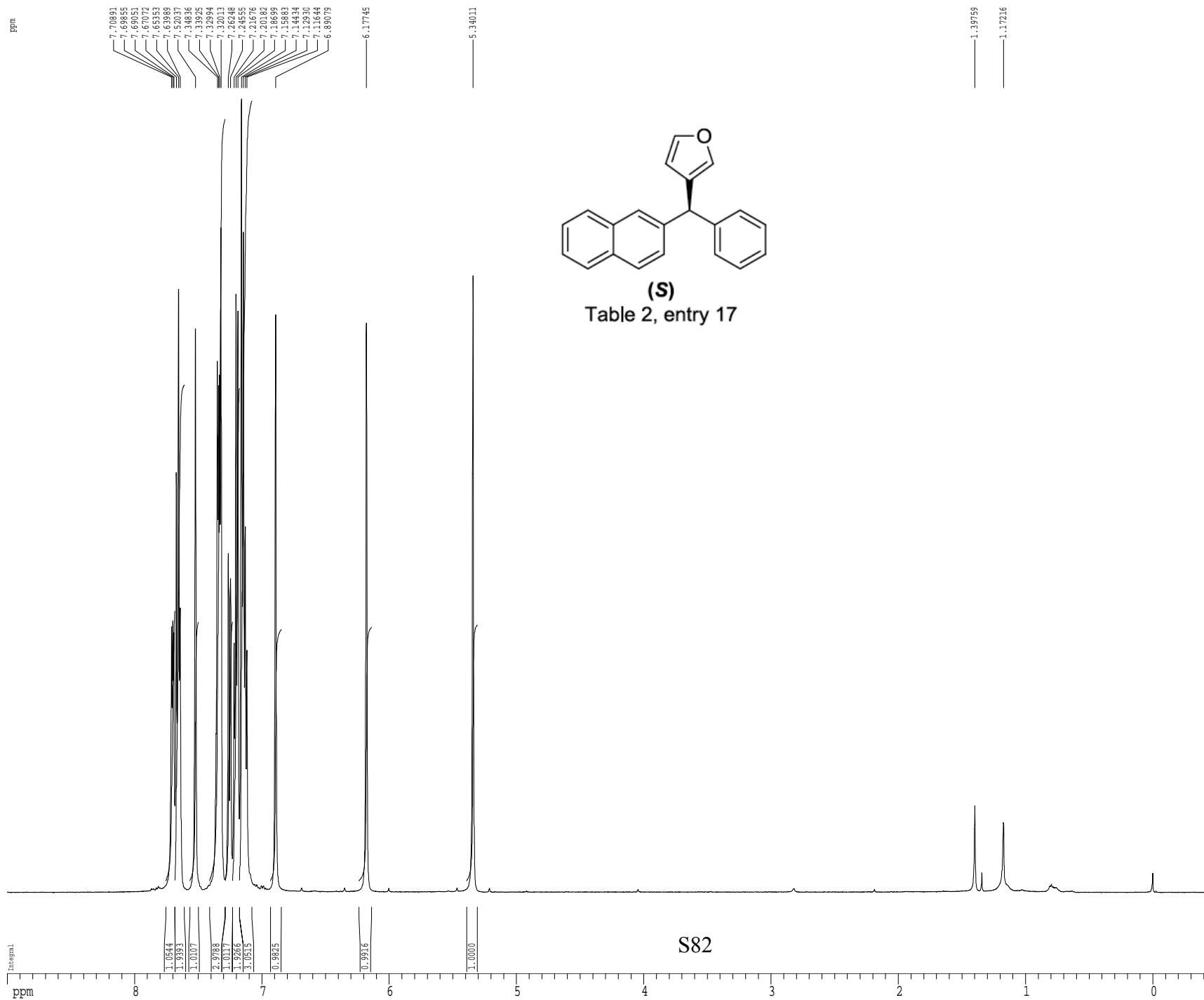
F2 - Processing parameters
 SI 65536
 SF 125.7804177 MHz
 WDW EM
 SSB 0
 LB 1.00 Hz
 GB 0
 PC 2.00

1D NMR plot parameters
 CX 22.80 cm
 CY 15.65 cm
 F1P 220.000 ppm
 F1 27671.69 Hz
 F2P -5.000 ppm
 F2 -628.90 Hz
 PPMCM 9.86842 ppm/cm
 HZCM 1241.25415 Hz/cm

S81



¹H spectrum



(S)

Table 2, entry 17

Current Data Parameters
 USER mharri
 NAME MRH-III-277-1HNMR
 EXPNO 3
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20120928
 Time 11.03
 INSTRUM cryo500
 PROBD 5 mm CPTCI 1H-
 PULPROG zg30
 TD 81728
 SOLVENT CDCl3
 NS 1
 DS 2
 SWH 8012.820 Hz
 FIDRES 0.098043 Hz
 AQ 5.0998774 sec
 RG 5
 DW 62.400 usec
 DE 6.00 usec
 TE 298.1 K
 D1 0.10000000 sec
 MCREST 0.00000000 sec
 MCNRK 0.01500000 sec

===== CHANNEL f1 =====
 NUC1 1H
 P1 7.50 usec
 PL1 1.60 dB
 SF01 500.2235015 MHz

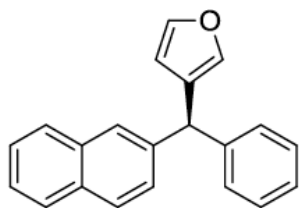
F2 - Processing parameters
 SI 65536
 SF 500.2201019 MHz
 WDW EM
 SSB 0
 LB 0.30 Hz
 GB 0
 PC 4.00

1D NMR plot parameters
 CX 22.80 cm
 CY 15.00 cm
 F1 9.000 ppm
 F1 4501.98 Hz
 F2P -0.500 ppm
 F2 -250.11 Hz
 PPMCM 0.41667 ppm/cm
 HZCM 208.42505 Hz/cm

S82

Z-restored spin-echo 13C spectrum with 1H decoupling

ppm



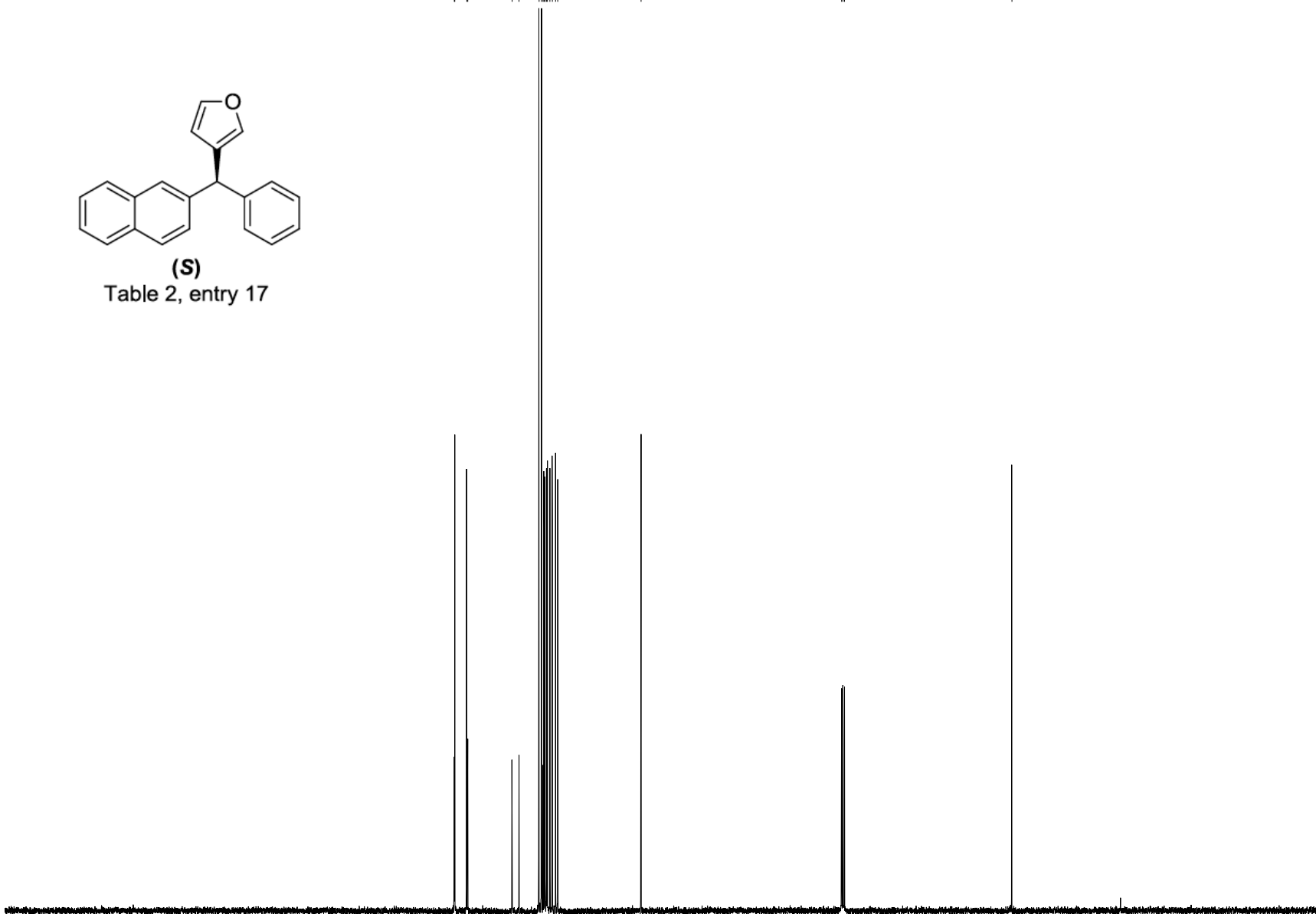
(S)

Table 2, entry 17

143.42
143.33
141.31
141.14
133.55
132.39
128.98
128.56
128.31
128.14
127.97
127.71
127.55
127.11
126.71
126.16
125.79
111.57

77.41
77.16
76.51

48.38



Current Data Parameters
 USER mharri
 NAME MRH-III-277-13CNMR
 EXPNO 3
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20120928
 Time_ 11.07
 INSTRUM cryo500
 PROBHD 5 mm CPTCI 1H-
 PULPROG SpinEchopg30gp.prd
 TD 65536
 SOLVENT CDCl3T
 NS 102
 DS 16
 SWH 30303.031 Hz
 FIDRES 0.462388 Hz
 AQ 1.0813940 sec
 RG 7298.2
 DW 16.500 usec
 DE 6.00 usec
 TE 298.1 K
 D1 0.25000000 sec
 d11 0.03000000 sec
 D16 0.00020000 sec
 d17 0.00019600 sec
 MCREST 0.00000000 sec
 MCWRX 0.01500000 sec
 P2 31.00 usec

***** CHANNEL f1 *****
 NUC1 13c
 P1 15.50 usec
 P11 500.00 usec
 P12 2000.00 usec
 PL0 120.00 dB
 PL1 -1.00 dB
 SFO1 125.7942548 MHz
 SF1 3.20 dB
 SF2 3.20 dB
 SPNAM1 Crp60,0.5,20.1
 SPNAM2 Crp60comp,4
 SPOFF1 0.00 Hz
 SPOFF2 0.00 Hz

***** CHANNEL f2 *****
 CPDPRG2 waltz16
 NUC2 1H
 PCPD2 100.00 usec
 PL2 1.60 dB
 PL12 24.60 dB
 SFO2 500.2225011 MHz

***** GRADIENT CHANNEL *****
 GENAM1 SINE.100
 GENAM2 SINE.100
 GFX1 0.00 %
 GFX2 0.00 %
 GPY1 0.00 %
 GPY2 0.00 %
 GPZ1 30.00 %
 GPZ2 50.00 %
 p15 500.00 usec
 p16 1000.00 usec

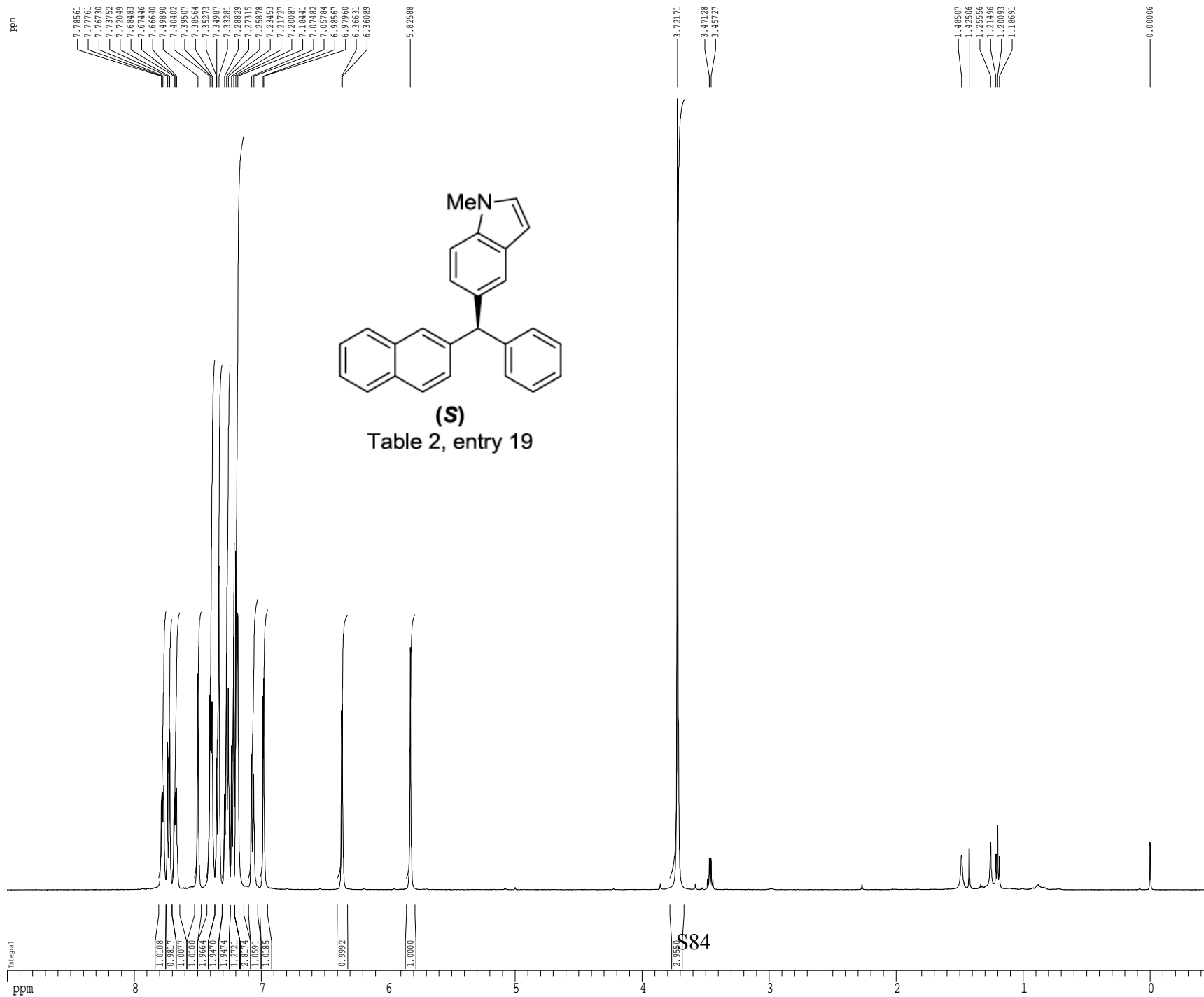
F2 - Processing parameters
 SI 65536
 SF 125.7804159 MHz
 HN
 SSB 0
 LB 1.00 Hz
 GB 0
 PC 2.00

1D NMR plot parameters
 CX 22.80 cm
 CY 15.65 cm
 F1P 220.000 ppm
 F1 27671.69 Hz
 F2P -5.000 ppm
 F2 -628.90 Hz
 PFMC 9.86842 ppm/cm
 HZCM 1241.25415 Hz/cm

S83

ppm 200 180 160 140 120 100 80 60 40 20 0

¹H spectrum



Current Data Parameters
 USER mharri
 NAME MRH-III-267-1HNMR
 EXPNO 1
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20120921
 Time 14.29
 INSTRUM cryo500
 PROBEHD 5 mm CPTCI 1H-
 PULPROG zg30
 TD 81728
 SOLVENT CDCl3
 NS 1
 DS 2
 SWH 8012.820 Hz
 FIDRES 0.098043 Hz
 AQ 5.0998774 sec
 RG 3.6
 DW 62.400 usec
 DE 6.00 usec
 TE 298.0 K
 D1 0.10000000 sec
 MCREST 0.00000000 sec
 MCWPK 0.01500000 sec

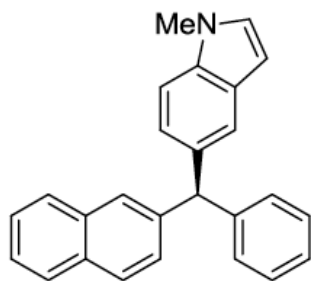
===== CHANNEL f1 =====
 NUC1 1H
 P1 7.50 usec
 PL1 1.60 dB
 SFO1 500.2235015 MHz

F2 - Processing parameters
 SI 65536
 SF 500.2200670 MHz
 WDW EM
 SSB 0
 LB 0.30 Hz
 GB 0
 PC 4.00

1D NMR plot parameters
 CX 22.80 cm
 CY 15.00 cm
 F1 9.000 ppm
 F2 4501.98 Hz
 F2P -0.500 ppm
 F2 -250.11 Hz
 PPMCM 0.41667 ppm/cm
 HZCM 208.42503 Hz/cm

Z-restored spin-echo 13C spectrum with 1H decoupling

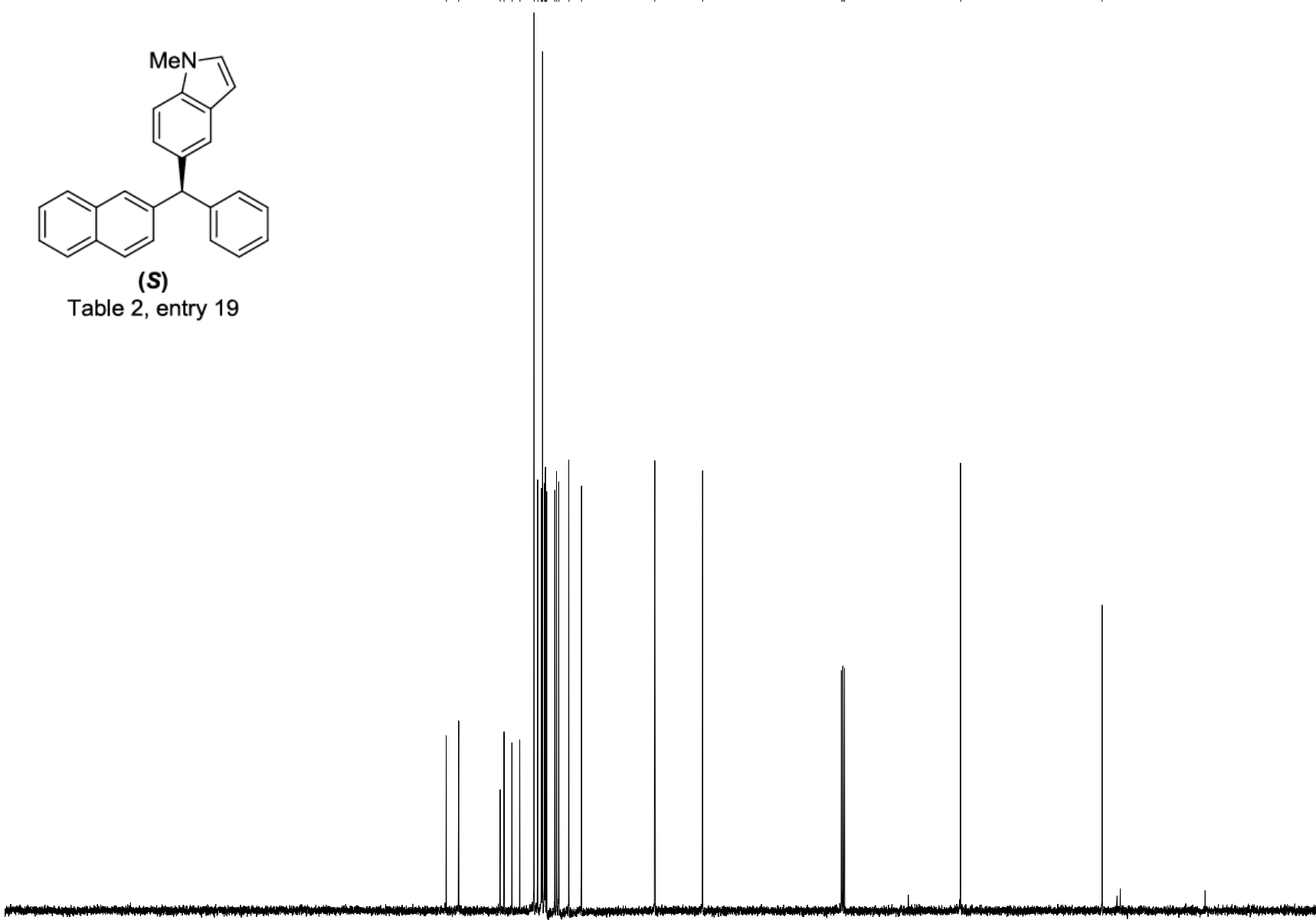
ppm



(S)

Table 2, entry 19

144.74
142.64
135.59
134.91
133.54
132.21
129.80
129.17
128.55
128.52
128.37
128.01
127.88
127.81
127.65
126.27
125.96
125.57
123.86
121.72
109.22
101.09
77.41
77.16
76.51
57.09
32.98



Current Data Parameters
USER mharri
NAME MRH-III-267-13CNMR
EXPNO 2
PROCNO 1

F2 - Acquisition Parameters
Date_ 20120921
Time 14.31
INSTRUM cryo500
PROBHD 5 mm CPTCI 1H-
PULPROG SpinEchopg30gp.prd
TD 65536
SOLVENT CDCl3T
NS 107
DS 15
SWH 30303.031 Hz
FIDRES 0.462388 Hz
AQ 1.0813940 sec
RG 7298.2
DW 16.500 usec
DE 6.00 usec
TE 298.0 K
D1 0.25000000 sec
d11 0.03000000 sec
D16 0.00020000 sec
d17 0.00019600 sec
MCREST 0.00000000 sec
MCWRX 0.01500000 sec
P2 31.00 usec

***** CHANNEL f1 *****
NUC1 13c
P1 15.50 usec
P11 500.00 usec
P12 2000.00 usec
PL0 120.00 dB
PL1 -1.00 dB
SFO1 125.7942548 MHz
SF1 3.20 dB
SF2 3.20 dB
SFOAM1 Crp60,0.5,20.1
SFOAM2 Crp60comp,4
SFOFF1 0.00 Hz
SFOFF2 0.00 Hz

***** CHANNEL f2 *****
CPDPRG2 waltz16
NUC2 1H
PCPD2 100.00 usec
PL2 1.60 dB
PL12 24.60 dB
SFO2 500.2225011 MHz

***** GRADIENT CHANNEL *****
GENAM1 SINE.100
GENAM2 SINE.100
GFX1 0.00 %
GFX2 0.00 %
GPY1 0.00 %
GPY2 0.00 %
GPZ1 30.00 %
GPZ2 50.00 %
p15 500.00 usec
p16 1000.00 usec

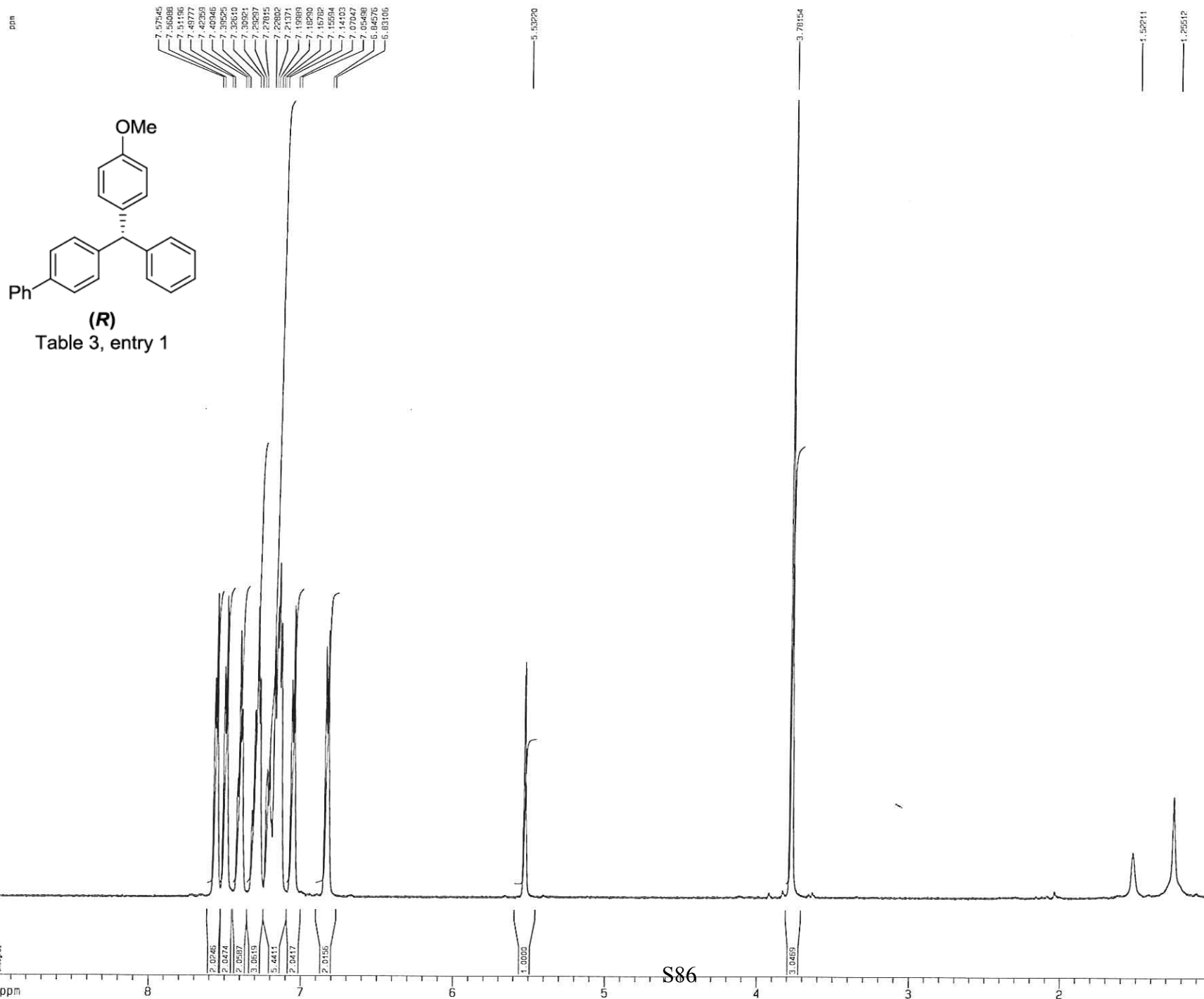
F2 - Processing parameters
SI 65536
SF 125.7804182 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 2.00

1D NMR plot parameters
CX 22.80 cm
CY 15.65 cm
F1 220.000 ppm
F1 27671.69 Hz
F2P -5.000 ppm
F2 -628.90 Hz
PFMCM 9.86842 ppm/cm
HZCM 1241.25415 Hz/cm

S85

ppm 200 180 160 140 120 100 80 60 40 20 0

1H spectrum



Current Data Parameters
 USER jhanna
 NAME LEH-1-116-c2
 EXPRD 1
 PROCNO 1

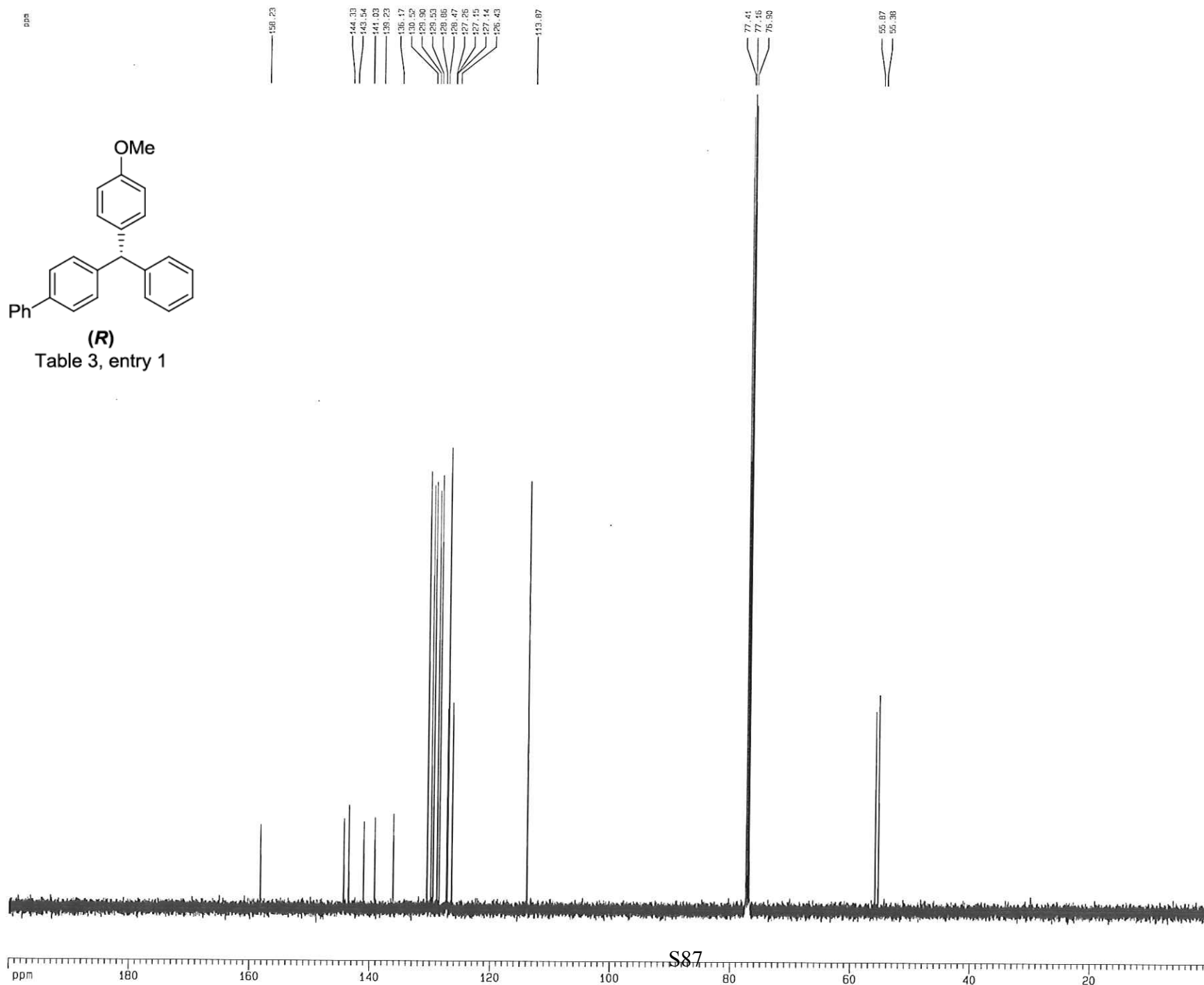
F2 - Acquisition Parameters
 Date_ 20121023
 Time 18.44
 INSTRUM gn500
 PROBD 5 mm broadband
 PULPROG zg30
 TD 81728
 SOLVENT CDCl3
 NS 8
 DS 2
 SWH 8012.820 Hz
 FIDRES 0.098343 Hz
 AQ 5.0398774 sec
 RG 143.7
 DW 62.400 usec
 DE 6.00 usec
 TE 298.0 K
 D1 0.10000000 sec
 MCREST 0.00000000 sec
 MCWK 0.01500000 sec

***** CHANNEL f1 *****
 NUC1 1H
 P1 12.20 usec
 PL1 -5.00 dB
 SFO1 499.4034958 MHz

F2 - Processing parameters
 SI 65536
 SF 499.4000425 MHz
 WDW no
 SSB 0
 LB 0.00 Hz
 GB 0
 PC 1.00

1D NMR plot parameters
 CX 22.80 cm
 CY 15.00 cm
 F1P 9.000 dppm
 F1 4434.60 Hz
 F2P 1.000 dppm
 F2 499.40 Hz
 PPMCM 0.35088 ppm/cm
 HZCM 175.22809 Hz/cm

13C spectrum with 1H decoupling



Current Data Parameters
 USER Ihanna
 NAME LEH-1-118-C13
 EXPNO 1
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20121023
 Time 19.47
 INSTRUM gn500
 PROBHD 5 mm broadband
 PULPROG zgpg30
 TD 65536
 SOLVENT CDCl3
 NS 1024
 DS 4
 SWH 30303.031 Hz
 FIDRES 0.462388 Hz
 AQ 1.0813240 sec
 RG 7298.2
 DM 16.500 usec
 DE 4.50 usec
 TE 298.0 K
 D1 0.25000000 sec
 d11 0.03000000 sec
 MCREST 0.00000000 sec
 MCWK 0.01500000 sec

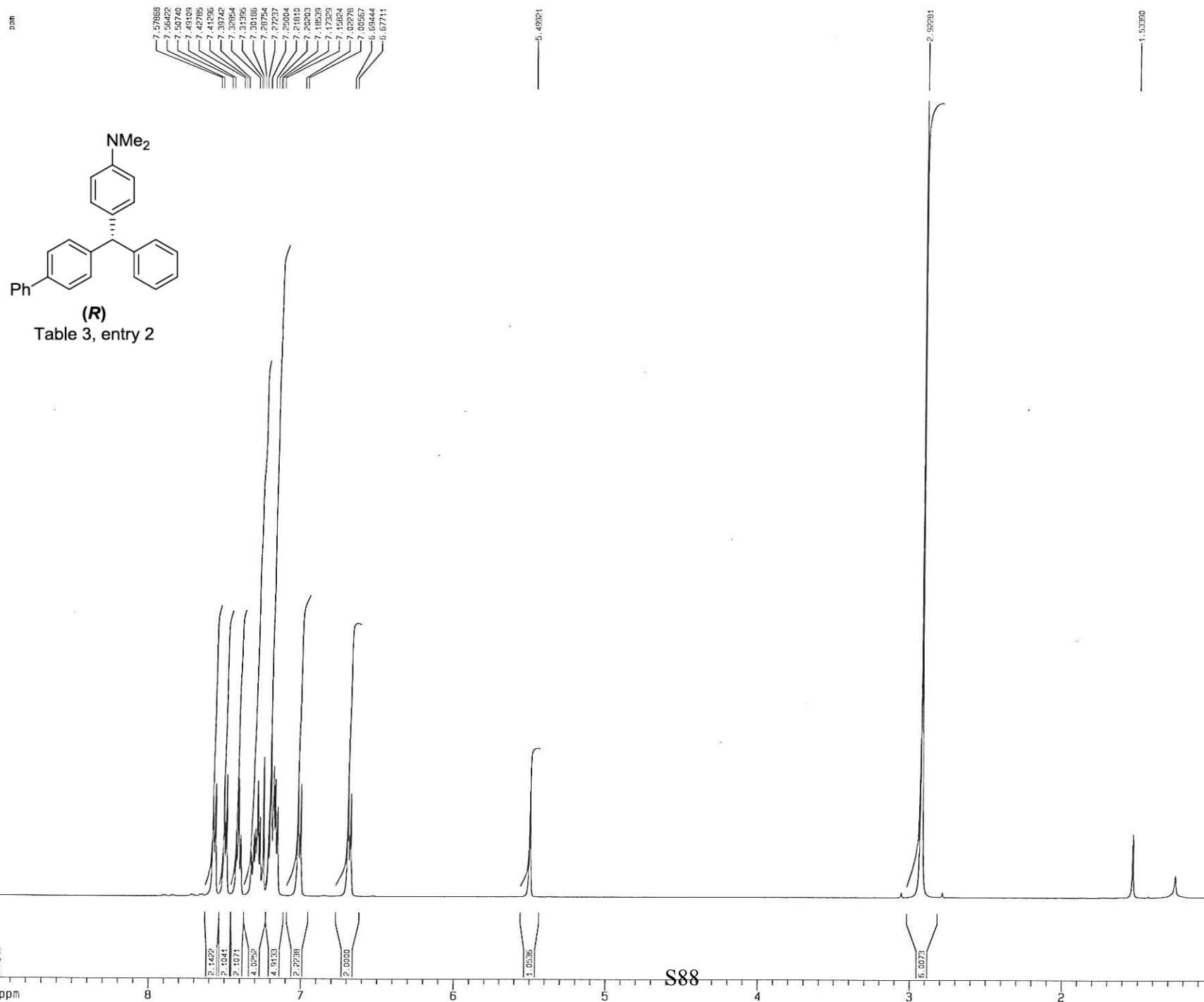
***** CHANNEL f1 *****
 NUC1 13C
 P1 7.70 usec
 PL1 0.00 dB
 SF01 125.5680432 MHz

***** CHANNEL f2 *****
 CPDPRG2 veltz16
 NUC2 1H
 PCPD2 80.00 usec
 PL2 -3.00 dB
 PL12 13.20 dB
 SF02 499.4024970 MHz

F2 - Processing parameters
 SI 65536
 SF 125.5742201 MHz
 WDW no
 SSB 0
 LB 0.00 Hz
 GB 0
 PC 2.00

ID NMR plot parameters
 CX 22.80 cm
 CY 15.65 cm
 F1P 200.000 ppm
 F1 25114.84 Hz
 F2P 0.000 ppm
 F2 0.00 Hz
 PPMCM 8.77193 ppm/cm
 HZCM 1101.52832 Hz/cm

1H spectrum



Current Data Parameters
 USER Ihanna
 NAME LEH-1-148-c3-f25-2
 EXPNO 1
 PROCNO 1

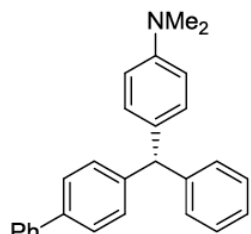
F2 - Acquisition Parameters
 Date_ 20121118
 Time 14.31
 INSTRUM cryo500
 PROBHD 5 mm EPTCI 1H-
 PULPROG zg30
 TD 81728
 SOLVENT CDCl3
 NS 40
 DS 2
 SWH 8012.820 Hz
 FIDRES 0.099043 Hz
 AQ 5.0999774 sec
 RG 5.7
 DK 62.400 usec
 DE 5.00 usec
 TE 298.0 K
 D1 0.1000000 sec
 MCREST 0.0000000 sec
 MCKRK 0.01500000 sec

***** CHANNEL f1 *****
 NUC1 1H
 P1 7.50 usec
 PL1 1.60 dB
 SFO1 500.225015 MHz

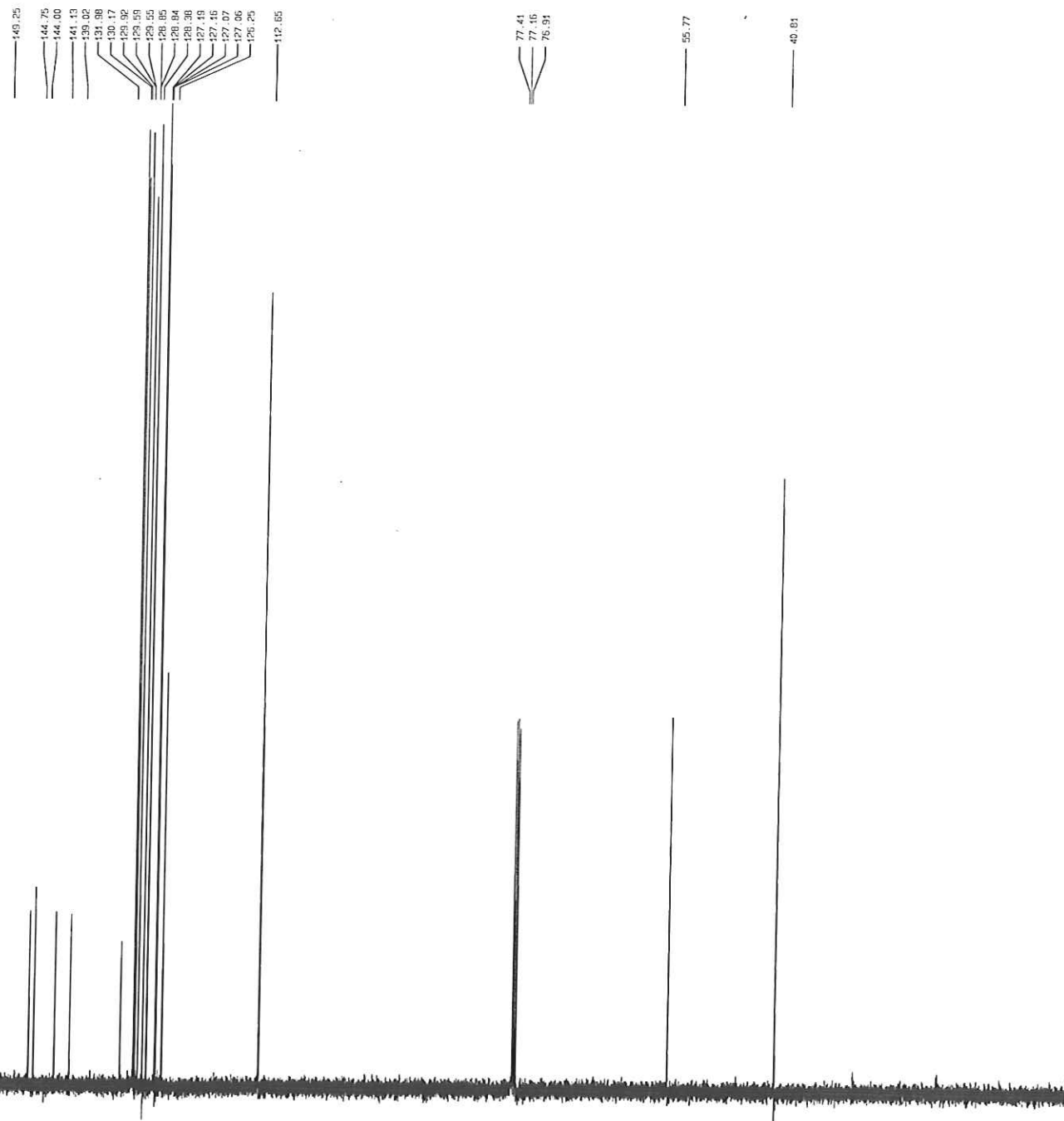
F2 - Processing parameters
 SI 65536
 SF 500.220354 MHz
 WDW no
 SSB 0
 LB 0.00 Hz
 GB 0
 PC 4.00

ID NMR list parameters
 CX 22.80 cm
 CY 15.00 cm
 F1P 9.000 ppm
 F1 4501.98 Hz
 F2P 1.000 ppm
 F2 500.22 Hz
 PPMCW 0.35088 ppm/cm
 HZCW 175.51591 Hz/cm

Z-restored spin-echo ¹³C spectrum with ¹H decoupling



(R)
Table 3, entry 2



149.25
144.75
144.00
141.13
139.02
131.98
130.17
129.92
129.59
129.55
128.85
128.84
128.38
127.18
127.16
127.07
127.06
126.25
112.85

77.41
77.16
76.91

55.77

40.81

```

Current Data Parameters
USER          Inanna
NAME          LEH-1-149-C13
EXPNO         1
PROCNO        1

F2 - Acquisition Parameters
Date_         20121117
Time          23.20
INSTRUM       cryo500
PROBHD        5 mm CPIC1 1H-
PULPROG       Spinechozg30gp.prd
TD            65536
SOLVENT       CDCl3
NS            325
DS            16
SKH           30303.031 Hz
FIDRES        0.482388 Hz
AQ            1.0819940 sec
RG            9195.2
DW            16.500 usec
DE            239.0 K
TE            300.2 K
D1            0.25000000 sec
d11           0.03000000 sec
D15           0.00020000 sec
d17           0.00015600 sec
MCREST        0.00000000 sec
MCMRG         0.01500000 sec
P2            31.00 usec

***** CHANNEL f1 *****
NUC1           13C
P1             15.50 usec
P11            500.00 usec
P12            2000.00 usec
PL0            120.00 dB
PL1            -1.00 dB
SF01           125.7842549 MHz
SR1            3.20 dB
SP2            3.20 dB
SPNAM1         Crp50,0.5,20.1
SPNAM2         Crp500000,4
SPOFF1         0.00 Hz
SPOFF2         0.00 Hz

***** CHANNEL f2 *****
CPDPRG2       waltz16
NUC2           1H
PCPD2         100.00 usec
PL2            1.60 dB
PL12           24.60 dB
SF02           500.2225011 MHz

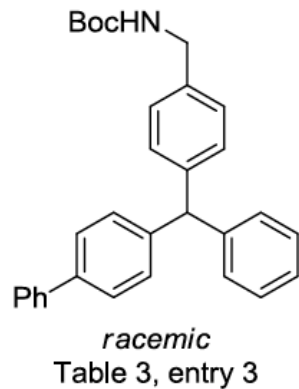
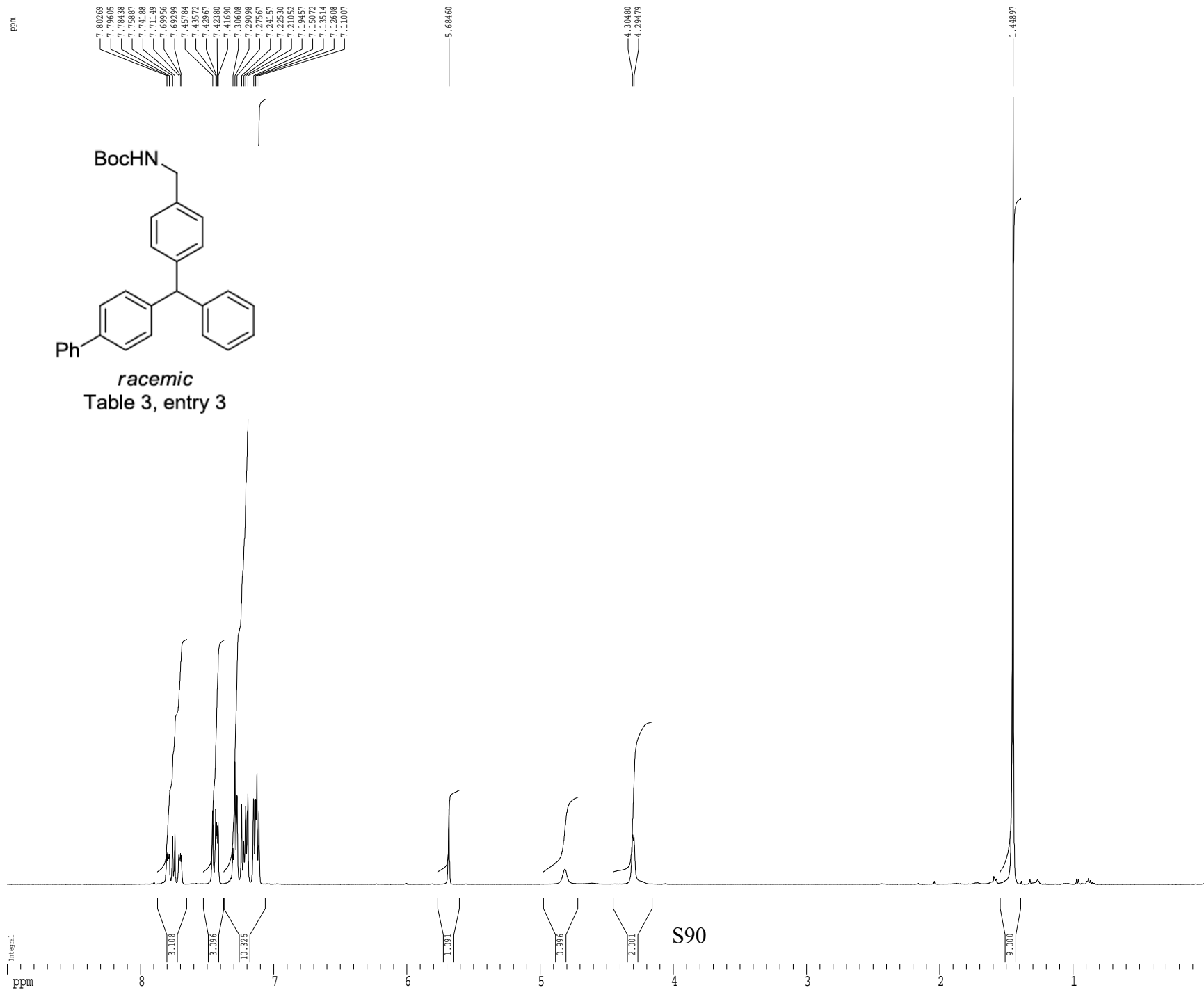
***** GRADIENT CHANNEL *****
GRNAM1        SINE.100
GRNAM2        SINE.100
GPX1           0.00 %
GPX2           0.00 %
GPY1           0.00 %
GPY2           0.00 %
GPZ1           30.00 %
GPZ2           50.00 %
p15            500.00 usec
p16            1000.00 usec

F2 - Processing parameters
SI             65536
SF             125.7842549 MHz
WDW            no
SSB            0
LB             0.00 Hz
GB             0
PC             2.00

ID NMR plot parameters
CX             22.80 cm
CY             15.65 cm
F1P           200.000 ppm
F1             25156.08 Hz
F2P            0.000 ppm
F2             0.00 Hz
PPMCM          8.77193 ppm/cm
HZCM          1103.35991 Hz/cm
    
```

ppm 180 160 140 120 100 80 60 40 20

¹H spectrum



```

Current Data Parameters
USER      lhanna
NAME      LEH-1-179-H1
EXPNO     1
PROCNO    1

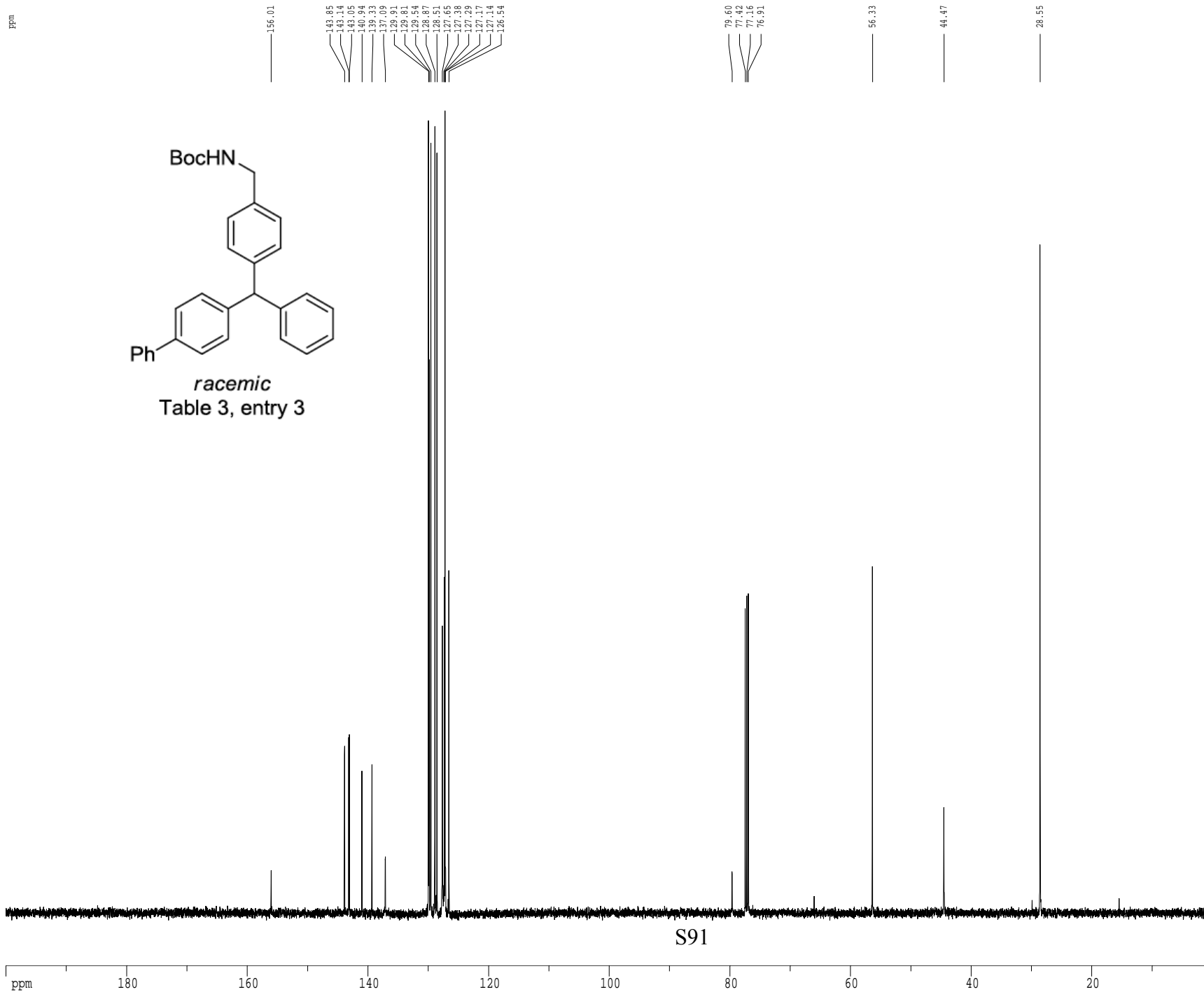
F2 - Acquisition Parameters
Date_     20130112
Time      17.13
INSTRUM   cryo500
PROBHD    5 mm CPTCI 1H-
PULPROG   zg30
TD         81728
SOLVENT   CDCl3
NS         8
DS         2
SWH        8012.820 Hz
FIDRES     0.098043 Hz
AQ          5.0998774 sec
RG          9
DW          62.400 usec
DE          6.00 usec
TE          298.0 K
DL          0.10000000 sec
MCREST     0.00000000 sec
MCWRX      0.01500000 sec

===== CHANNEL f1 =====
NUC1       1H
P1          7.50 usec
PL1         1.60 dB
SF01        500.2235015 MHz

F2 - Processing parameters
SI          65536
SF          500.2200381 MHz
WDW         no
SSB         0
LB          0.00 Hz
GB          0
PC          4.00

1D NMR plot parameters
CX          22.80 cm
CY          15.00 cm
F1P         9.000 ppm
F1          4501.98 Hz
F2P         0.000 ppm
F2          0.00 Hz
PPMCM       0.39474 ppm/cm
HZCM        197.45528 Hz/cm
    
```

Z-restored spin-echo 13C spectrum with 1H decoupling



```

Current Data Parameters
USER          lhanna
NAME          LEH-1-143-c13-2
EXPNO         1
PROCNO        1

F2 - Acquisition Parameters
Date_         20121117
Time          18.06
INSTRUM       cryo500
PROBHD        5 mm CPTCI 1H-
PULPROG       SpinEchopg30gp.prd
TD            65536
SOLVENT       CDCl3
NS            190
DS            16
SWH           30303.031 Hz
FIDRES        0.462388 Hz
AQ            1.0813940 sec
RG            3649.1
DW            16.500 usec
DE            6.00 usec
TE            298.0 K
D1            0.25000000 sec
d11           0.03000000 sec
d16           0.00020000 sec
d17           0.00019600 sec
MCREST        0.00000000 sec
MCONK         0.01500000 sec
P2            31.00 usec

===== CHANNEL f1 =====
NUC1           13c
P1            15.50 usec
P11           500.00 usec
P12           2000.00 usec
PL0           120.00 dB
PL1           -1.00 dB
SFO1          125.7942548 MHz
SF1           3.20 dB
SFO1M1        Crp60,0.5,20.1
SFO1M2        Crp60comp,4
SFOFF1        0.00 Hz
SFOFF2        0.00 Hz

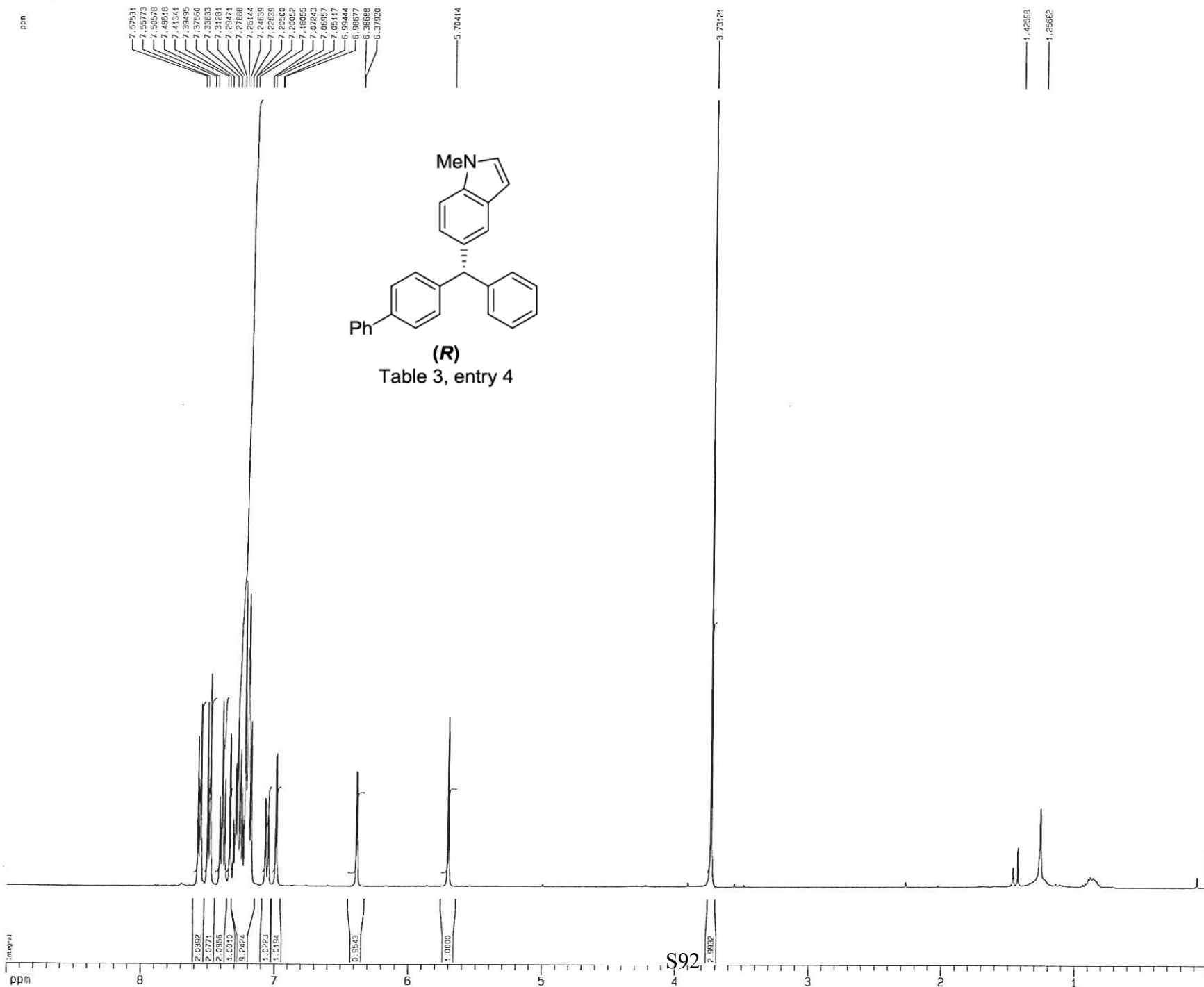
===== CHANNEL f2 =====
CPDPRG2       waltz16
NUC2           1H
PCPD2         100.00 usec
PL2           1.60 dB
PL12          24.60 dB
SFO2          500.2225011 MHz

===== GRADIENT CHANNEL =====
GENAM1        SINE.100
GENAM2        SINE.100
GFX1          0.00 %
GFX2          0.00 %
GPY1          0.00 %
GPY2          0.00 %
GFZ1          30.00 %
GFZ2          50.00 %
p15           500.00 usec
p16           1000.00 usec

F2 - Processing parameters
SI            65536
SF            125.7804122 MHz
WDW           EM
SSB           0
LB            1.00 Hz
GB            0
PC            2.00

1D NMR plot parameters
CX            22.80 cm
CY            15.65 cm
F1P           200.000 ppm
F1            25156.08 Hz
F2P           0.000 ppm
F2            0.00 Hz
PFMCM         8.77193 ppm/cm
HZCM          1103.33691 Hz/cm
    
```

1H spectrum



Current Data Parameters
 USCR Inanna
 NAME LEH-1-137-c2
 EXPNO 1
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20121026
 Time 11.53
 INSTRUM d-x400
 PROBHD 5 mm QNP H/P/P
 PULPROG zg30
 TD 65535
 SOLVENT CDCl3
 NS 8
 DS 2
 SWH 6410.256 Hz
 FIDRES 0.097813 Hz
 AQ 5.1116579 sec
 RG 101.6
 DM 78.000 usec
 DE 4.50 usec
 TE 298.1 K
 D1 0.10000000 sec
 MCREST 0.00000000 sec
 MCWIX 0.01500000 sec

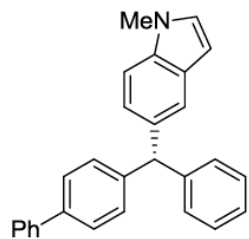
***** CHANNEL f1 *****
 NUC1 1H
 P1 12.00 usec
 PL1 -0.50 dB
 SFO1 400.1326009 MHz

F2 - Processing parameters
 SI 65535
 SF 400.1300472 MHz
 WDM no
 SSB 0
 LB 0.00 Hz
 GB 0
 PC 2.00

ID NMR plot parameters
 CX 22.80 cm
 CY 15.00 cm
 F1P 9.000 ppm
 F1 3501.17 Hz
 F2P 0.000 ppm
 F2 0.00 Hz
 PPMCM 0.39474 ppm/cm
 HZCM 157.94509 Hz/cm

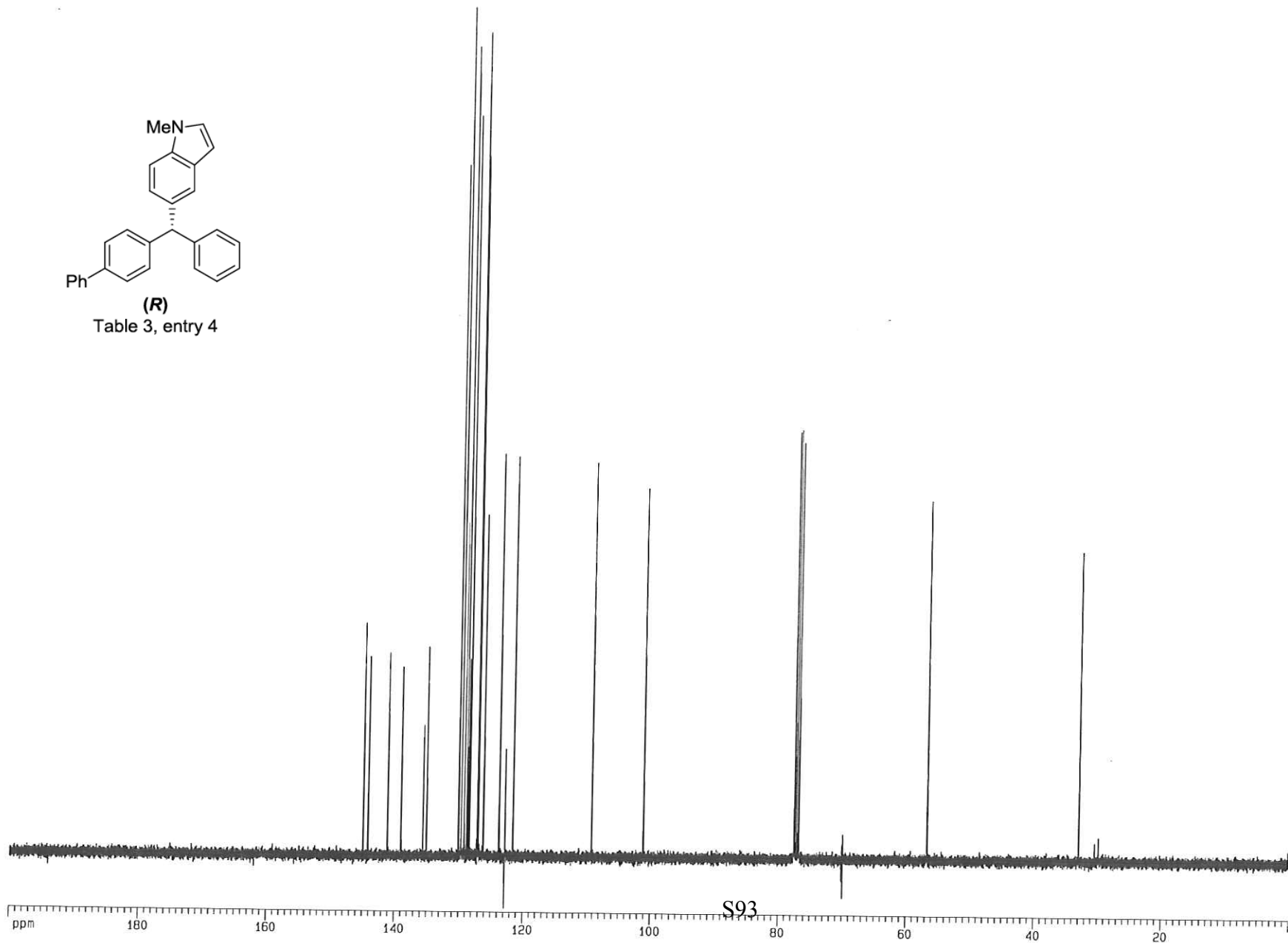
¹³C spectrum with ¹H decoupling

ppm



(R)

Table 3, entry 4



Current Data Parameters
USER Inanna
NAME LEH-1-137-C13
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20121026
Time 12.22
INSTRUM drx400
PROBHD 5 mm QNP H/F/P
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 1024
DS 4
SWH 24154.550 Hz
FIDRES 0.358570 Hz
AQ 1.3556452 sec
RG 14596.5
DM 20.700 usec
DE 20.39 usec
TE 299.1 K
D1 0.10000000 sec
d11 0.03000000 sec
MCRESST 0.00000000 sec
MCWRK 0.01500000 sec

***** CHANNEL f1 *****
NUC1 13C
P1 11.00 usec
PL1 0.00 dB
SF01 100.6237964 MHz

***** CHANNEL f2 *****
CPDPRG2 nlev16
NUC2 1H
PCPD2 80.00 usec
PL2 0.00 dB
PL12 16.20 dB
SF02 400.1328003 MHz

F2 - Processing parameters
SI 65536
SF 100.6127645 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
PC 1.00

1D NMR plot parameters
CX 22.80 cm
CY 15.50 cm
F1P 200.000 ppm
F1 20122.55 Hz
F2P 0.000 ppm
F2 0.00 Hz
PPMCM 8.77193 ppm/cm
HZCM 882.58812 Hz/cm

S93

¹H spectrum

ppm

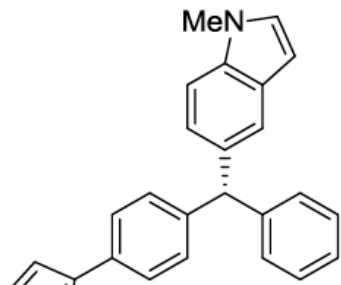
7.5894
7.3378
7.3000
7.2838
7.2585
7.2251
7.1948
7.1801
7.1650
7.1457
7.1357
7.1153
7.1036
7.0868
7.0723
7.0563
6.9584
6.9415
6.8986
6.8928
6.5664
6.2880
6.2825

5.5823

3.6324

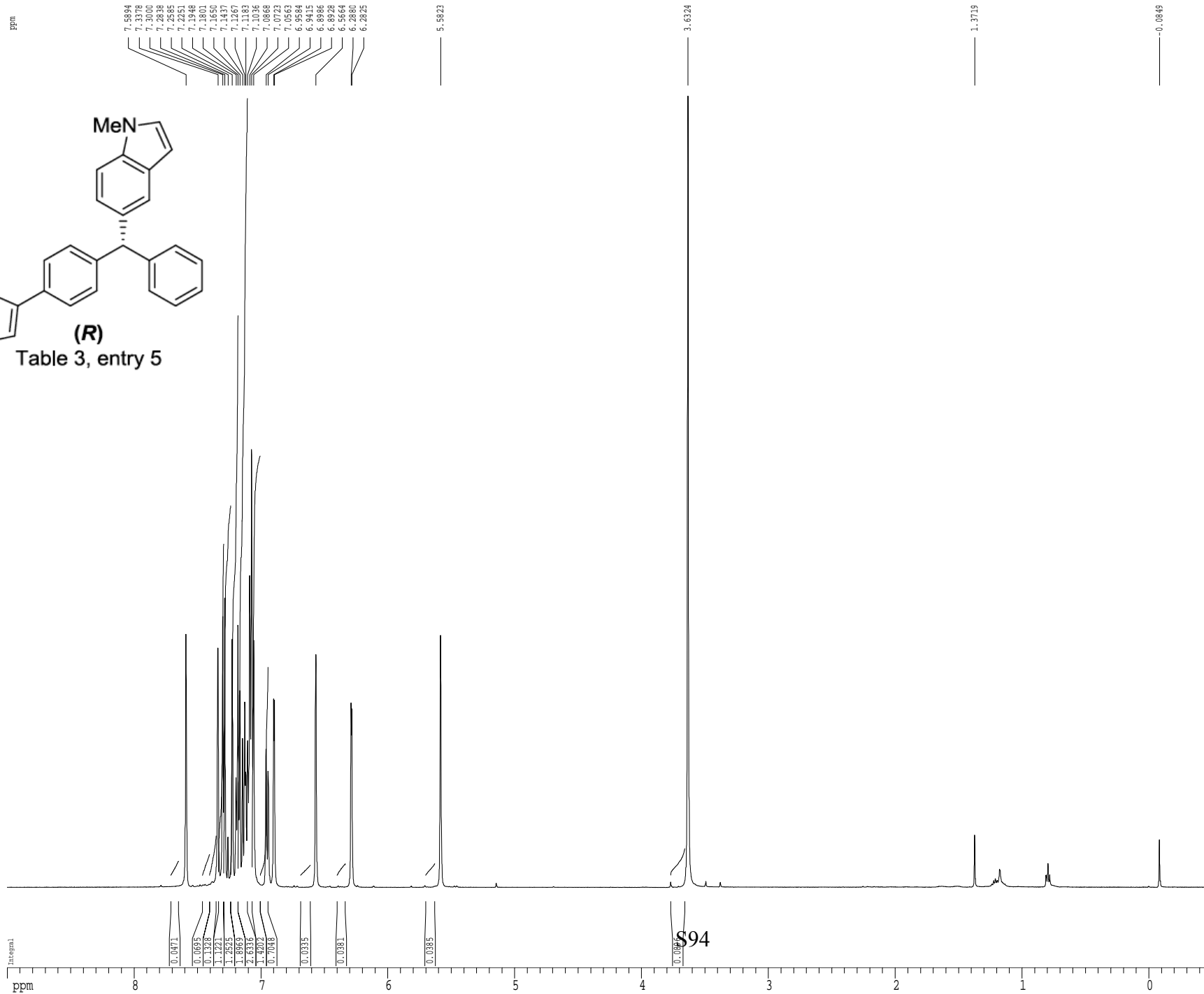
1.3719

-0.0849



(R)

Table 3, entry 5



S94

```

Current Data Parameters
USER          mharr
NAME          MRH-IV-135-1HNMR
EXPNO         2
PROCNO        1

F2 - Acquisition Parameters
Date_         20121118
Time          18.40
INSTRUM       cryo500
PROBHD        5 mm CPTCI 1H-
PULPROG       zg30
TD            81728
SOLVENT       CDCl3
NS            2
DS            2
SWH           8012.820 Hz
FIDRES        0.098043 Hz
AQ            5.0998774 sec
RG            4
DW            62.400 usec
DE            6.00 usec
TE            298.0 K
DL            0.10000000 sec
MCREST        0.00000000 sec
MCWRX         0.01500000 sec

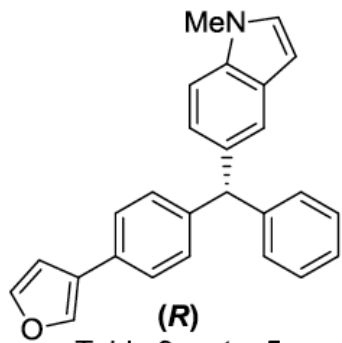
===== CHANNEL f1 =====
NUC1          1H
P1            7.50 usec
PL1           1.60 dB
SF01          500.2235015 MHz

F2 - Processing parameters
SI            65536
SF            500.2201117 MHz
WDW           EM
SSB           0
LB            0.30 Hz
GB            0
PC            4.00

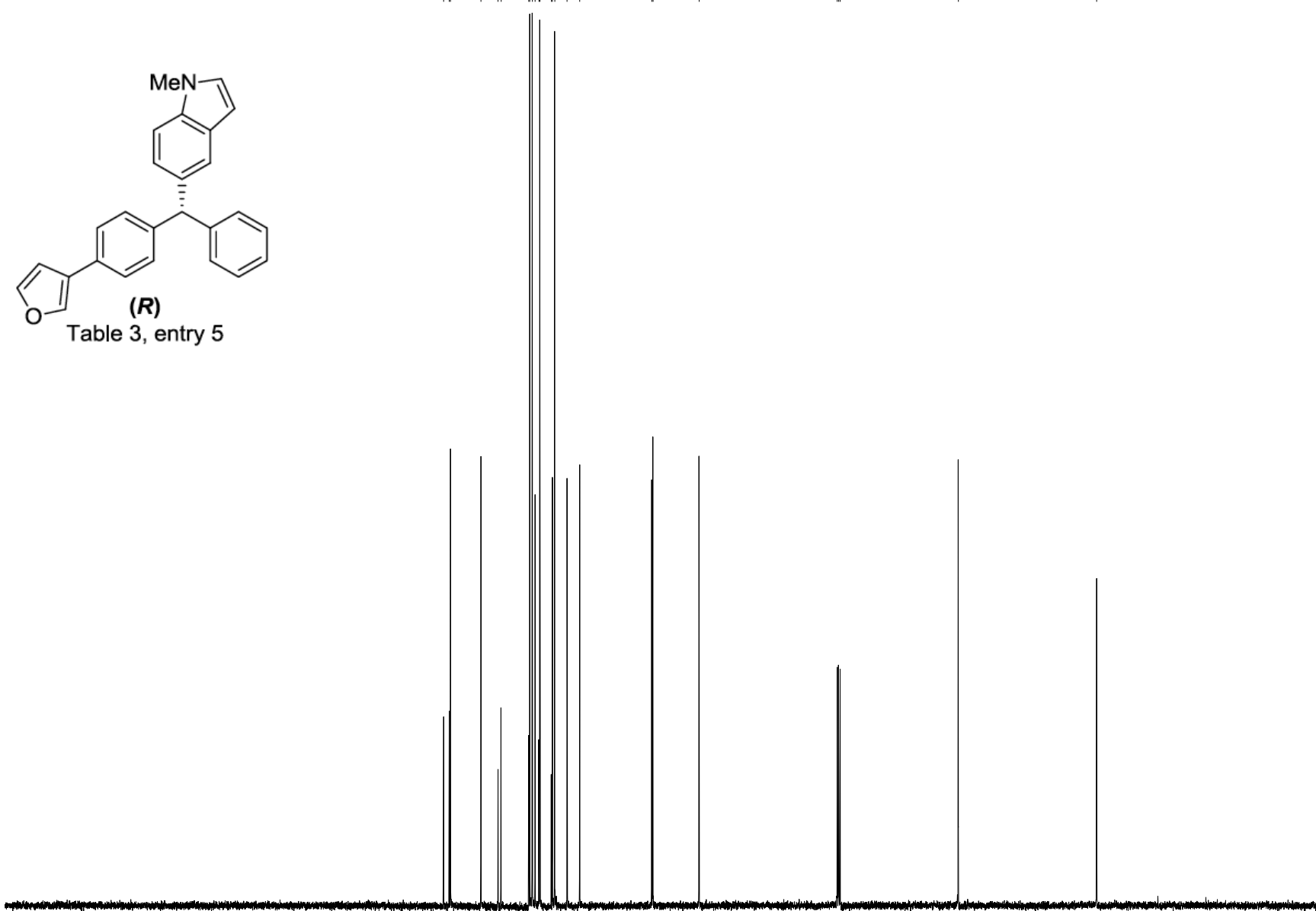
1D NMR plot parameters
CX            22.80 cm
CY            15.00 cm
F1P           9.000 ppm
F1            4501.98 Hz
F2P           -0.500 ppm
F2            -250.11 Hz
PPMCM         0.41667 ppm/cm
HZCM          208.42506 Hz/cm
    
```

Z-restored spin-echo 13C spectrum with 1H decoupling

ppm



144.86
143.88
143.68
138.45
135.55
135.01
130.25
130.09
129.65
129.17
128.53
128.34
128.39
128.11
125.81
123.70
121.52
109.18
108.98
101.06
77.41
77.16
76.90
56.66
32.96



Current Data Parameters
USER mharri
NAME MRH-IV-135-13CNMR
EXPNO 2
PROCNO 1

F2 - Acquisition Parameters
Date_ 20121118
Time 18.46
INSTRUM cryo500
PROBHD 5 mm CPTCI 1H-
PULPROG SpinEchopg30gp.prd
TD 65536
SOLVENT CDCl3T
NS 101
DS 16
SWH 30303.031 Hz
FIDRES 0.462388 Hz
AQ 1.0813940 sec
RG 7298.2
DW 16.500 usec
DE 6.00 usec
TE 298.0 K
D1 0.25000000 sec
d11 0.03000000 sec
D16 0.00020000 sec
d17 0.00019600 sec
MCREST 0.00000000 sec
MCMRK 0.01500000 sec
P2 31.00 usec

***** CHANNEL f1 *****
NUC1 13c
P1 15.50 usec
P11 500.00 usec
P12 2000.00 usec
PL0 120.00 dB
PL1 -1.00 dB
SFO1 125.7942548 MHz
SF1 3.20 dB
SF2 3.20 dB
SFOAM1 Crp60,0.5,20.1
SFOAM2 Crp60comp,4
SFOFF1 0.00 Hz
SFOFF2 0.00 Hz

***** CHANNEL f2 *****
CPDPRG2 waltz16
NUC2 1H
PCPD2 100.00 usec
PL2 1.60 dB
PL12 24.60 dB
SFO2 500.2225011 MHz

***** GRADIENT CHANNEL *****
GENAM1 SINE.100
GENAM2 SINE.100
GFX1 0.00 %
GFX2 0.00 %
GPF1 0.00 %
GPF2 0.00 %
GPF3 0.00 %
GPF4 30.00 %
GPF5 50.00 %
p15 500.00 usec
p16 1000.00 usec

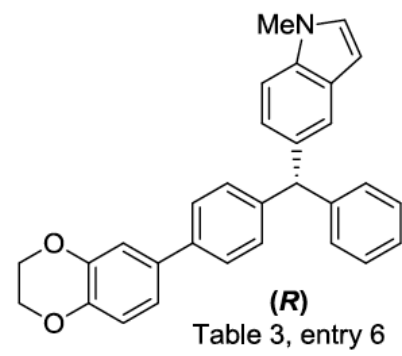
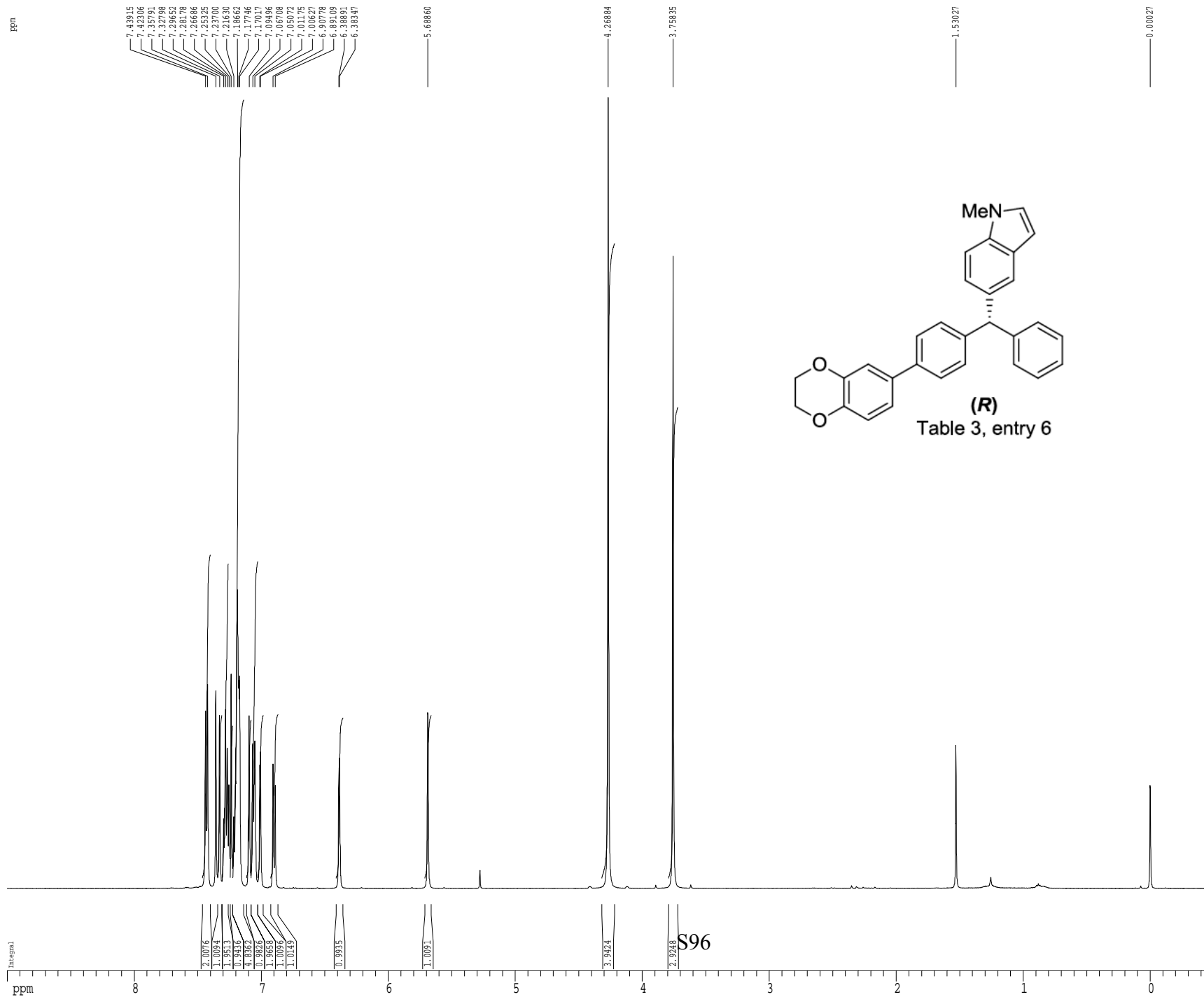
F2 - Processing parameters
SI 65536
SF 125.7804201 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 2.00

1D NMR plot parameters
CX 22.80 cm
CY 15.65 cm
F1P 220.000 ppm
F1 27671.69 Hz
F2P -5.000 ppm
F2 -628.90 Hz
PFMCM 9.86842 ppm/cm
HZCM 1241.25415 Hz/cm

S95

ppm 200 180 160 140 120 100 80 60 40 20 0

¹H spectrum



Current Data Parameters
 USER mharr
 NAME MRH-IV-142-1HNMR
 EXPNO 3
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20121119
 Time 12.04
 INSTRUM cryo500
 PROBHD 5 mm CPTCI 1H-
 PULPROG zg30
 TD 81728
 SOLVENT CDCl3
 NS 4
 DS 2
 SWH 8012.820 Hz
 FIDRES 0.098043 Hz
 AQ 5.0998774 sec
 RG 5
 DW 62.400 usec
 DE 6.00 usec
 TE 298.0 K
 DL 0.10000000 sec
 MCREST 0.00000000 sec
 MCWRR 0.01500000 sec

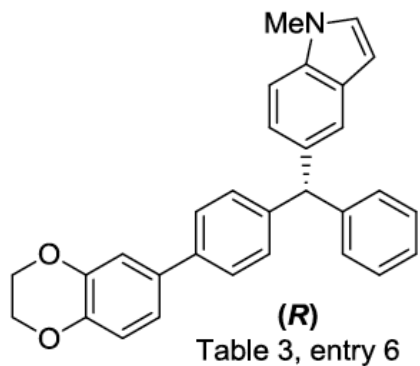
===== CHANNEL f1 =====
 NUC1 1H
 P1 7.50 usec
 PL1 1.60 dB
 SF01 500.2235015 MHz

F2 - Processing parameters
 SI 65536
 SF 500.2200423 MHz
 WDW EM
 SSB 0
 LB 0.30 Hz
 GB 0
 PC 4.00

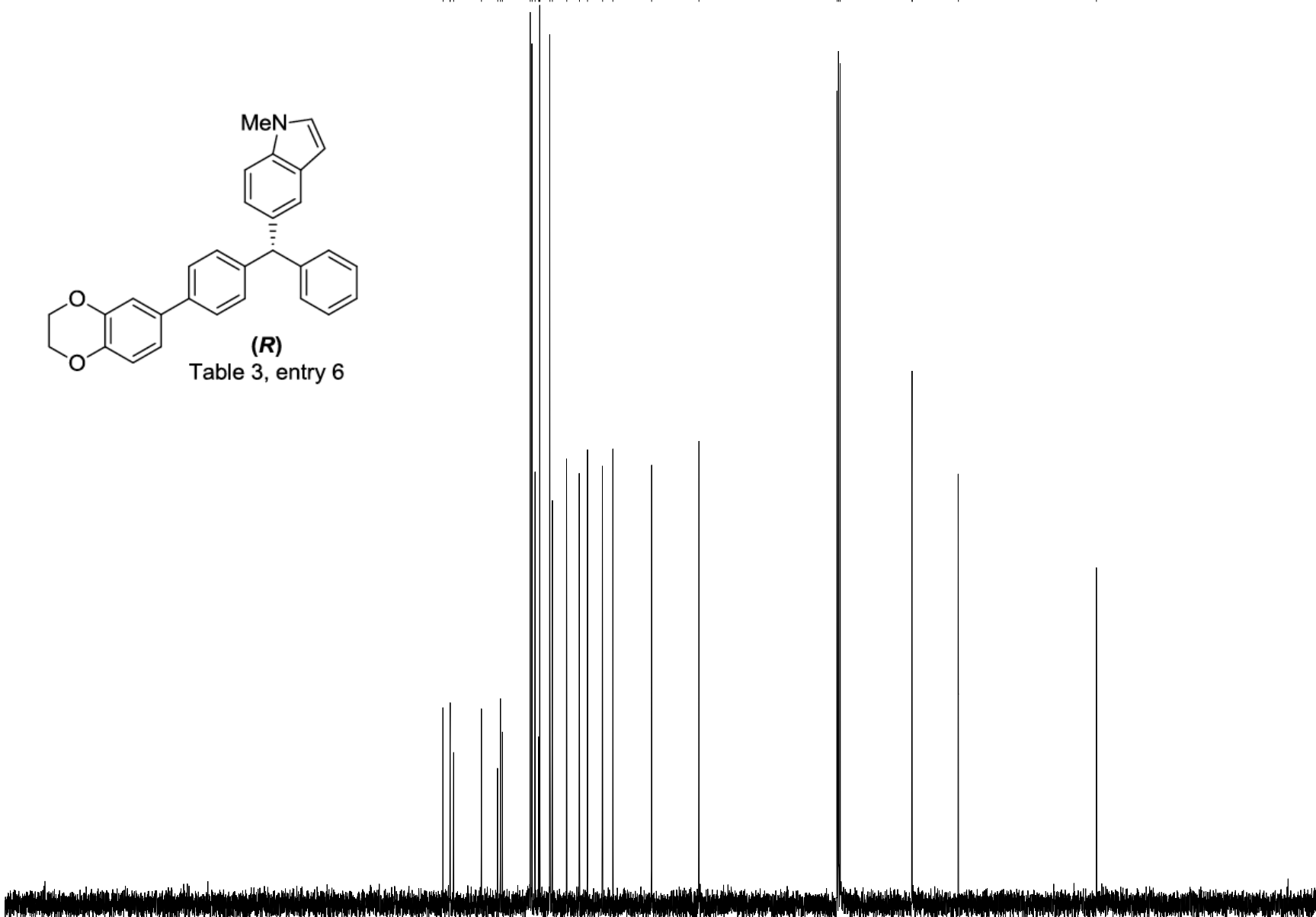
1D NMR plot parameters
 CX 22.80 cm
 CY 15.00 cm
 F1P 9.000 ppm
 F1 4501.98 Hz
 F2P -0.500 ppm
 F2 -250.11 Hz
 PPMCM 0.41667 ppm/cm
 HZCM 208.42502 Hz/cm

Z-restored spin-echo 13C spectrum with 1H decoupling

ppm



144.94
143.75
143.10
142.12
138.32
135.57
135.11
134.76
130.00
129.69
129.18
128.55
128.48
128.35
126.62
126.21
123.75
121.57
120.19
117.61
115.83
109.19
101.08
77.42
77.16
76.51
64.58
64.56
56.66
33.01



Current Data Parameters
USER mharri
NAME MRH-IV-142-13CNMR
EXPNO 2
PROCNO 1

F2 - Acquisition Parameters
Date_ 20121119
Time 12.06
INSTRUM cryo500
PROBHD 5 mm CPTCI 1H-
PULPROG SpinEchopg30gp.prd
TD 65536
SOLVENT CDCl3
NS 99
DS 16
SWH 30303.031 Hz
FIDRES 0.462388 Hz
AQ 1.0813940 sec
RG 7298.2
DW 16.500 usec
DE 6.00 usec
TE 298.0 K
D1 0.25000000 sec
d11 0.03000000 sec
D16 0.00020000 sec
d17 0.00019600 sec
MCREST 0.00000000 sec
MCMRK 0.01500000 sec
P2 31.00 usec

***** CHANNEL f1 *****
NUC1 13C
P1 15.50 usec
P11 500.00 usec
P12 2000.00 usec
PL0 120.00 dB
PL1 -1.00 dB
SFO1 125.7942548 MHz
SF1 3.20 dB
SF2 3.20 dB
SFOAM1 Crp60,0.5,20.1
SFOAM2 Crp60comp,4
SFOFF1 0.00 Hz
SFOFF2 0.00 Hz

***** CHANNEL f2 *****
CPDPRG2 waltz16
NUC2 1H
PCPD2 100.00 usec
PL2 1.60 dB
PL12 24.60 dB
SFO2 500.2225011 MHz

***** GRADIENT CHANNEL *****
GENAM1 SINE.100
GENAM2 SINE.100
GFX1 0.00 %
GFX2 0.00 %
GPF1 0.00 %
GPF2 0.00 %
GPF3 0.00 %
GPF4 30.00 %
GPF5 50.00 %
p15 500.00 usec
p16 1000.00 usec

F2 - Processing parameters
SI 65536
SF 125.7804108 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 2.00

ID NMR plot parameters
CX 22.80 cm
CY 15.65 cm
F1 220.000 ppm
F2 27671.69 Hz
F3 -5.000 ppm
F4 -628.90 Hz
F5 9.86842 ppm/cm
F6 1241.25415 Hz/cm

ppm 200 180 160 140 120 100 80 60 40 20 0

1H spectrum

ppm
 7.8062
 7.8065
 7.79576
 7.78703
 7.77901
 7.75779
 7.74275
 7.73642
 7.72770
 7.69587
 7.65681
 7.64187
 7.64032
 7.63301
 7.62480
 7.61619
 7.58225
 7.56101
 7.52050
 7.49051
 7.48488
 7.47427
 7.46927
 7.46163
 8.91693
 8.91200
 8.93945
 8.73684
 8.72921

5.84101

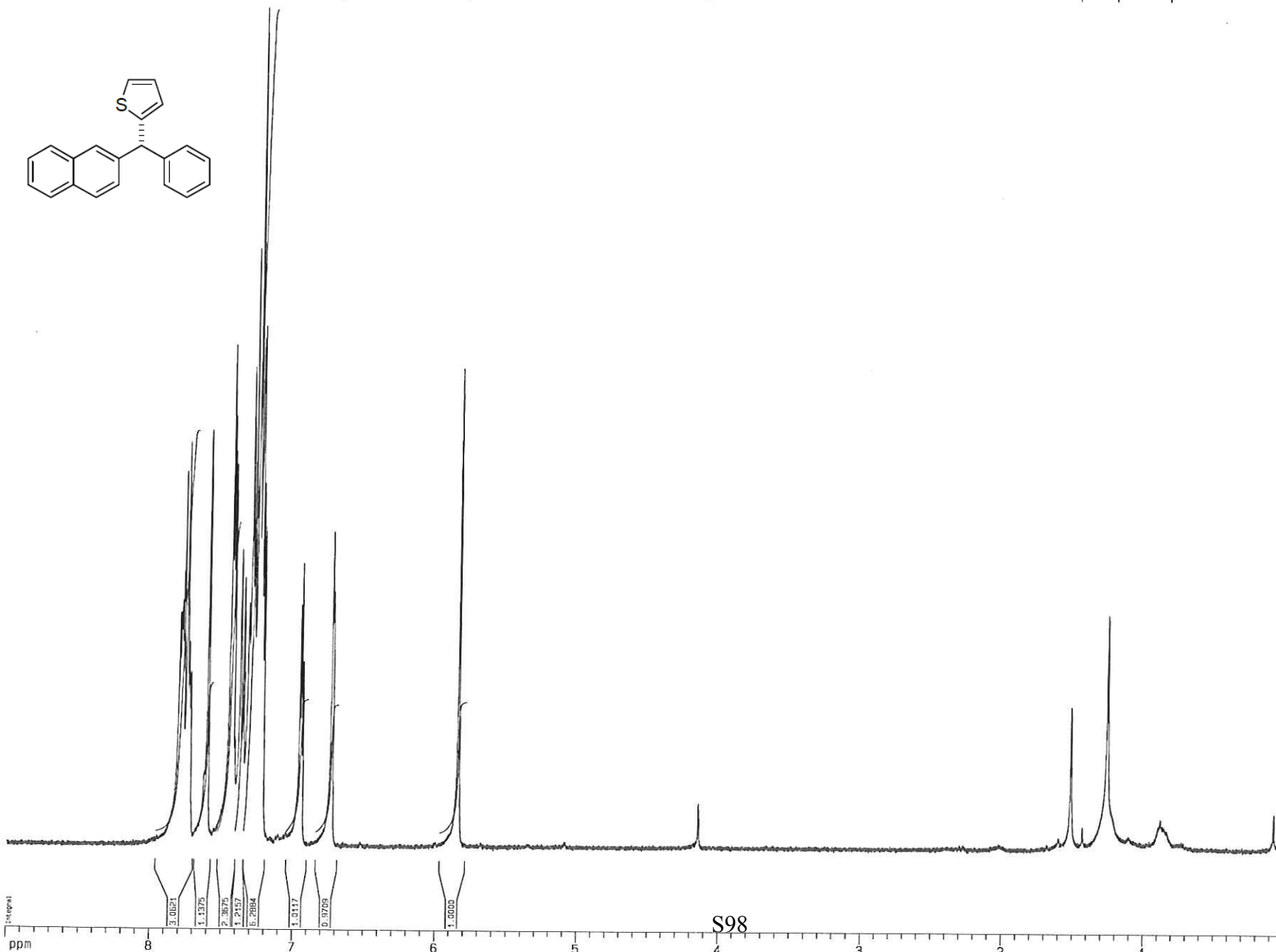
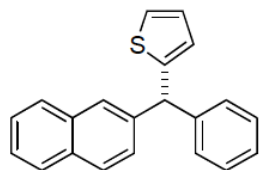
4.14077

1.51279

1.29360

0.88843

0.07471



Current Data Parameters
 USER Inanna
 NAME LEH-1-150-c2-pig
 EXPNO 1
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20121123
 Time 20:31
 INSTRUM d19400
 PROS-D 5 mm QNP H/F/P
 PULPROG zg30
 TD 65536
 SOLVENT CDCl3
 NS 8
 DS 2
 SWH 6410.256 Hz
 FIDRES 0.037813 Hz
 AQ 5.1118579 sec
 RG 256
 DW 78.000 usec
 DE 4.50 usec
 TE 298.1 K
 D1 0.1000000 sec
 MCREST 0.0000000 sec
 MCWK 0.0150000 sec

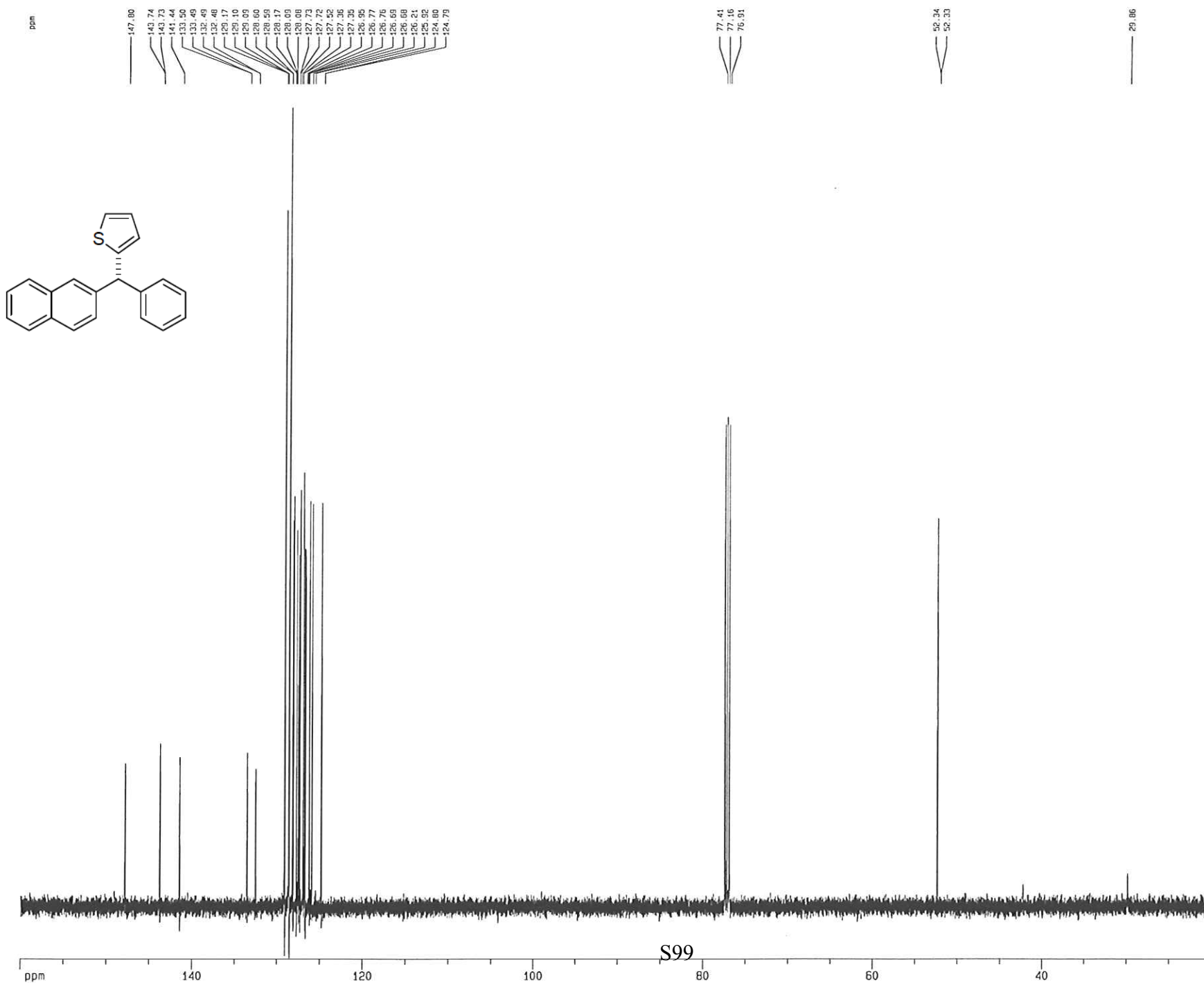
***** CHANNEL f1 *****
 NUC1 1H
 P1 12.00 usec
 PL1 -0.50 dB
 SFO1 400.1326009 MHz

F2 - Processing parameters
 SI 65536
 SF 400.1300295 MHz
 WDW no
 SSB 0
 LB 0.00 Hz
 GB 0
 PC 2.00

1D NMR plot parameters
 CX 22.80 cm
 CY 15.00 cm
 F1P 9.000 ppm
 F1 3601.17 Hz
 F2P 0.000 ppm
 F2 0.00 Hz
 FREQM 0.33474 ppm/cm
 HZCM 157.94508 Hz/cm

S98

Z-restored spin-echo 13C spectrum with 1H decoupling



```

Current Data Parameters
USER Ihanna
NAME LEH-1-150-C13
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20121128
Time 14.18
INSTRUM cryo500
PROBHD 5 mm CPIC1 1H-
PULPROG SpinEchopg30pp.prd
TD 65536
SOLVENT CDCl3
NS 184
DS 15
SWH 30303.031 Hz
FIDRES 0.462388 Hz
AQ 1.0813940 sec
RG 5160.6
DW 15.500 usec
DE 6.00 usec
TE 298.0 K
D1 0.2500000 sec
d11 0.0300000 sec
D16 0.0020000 sec
d17 0.00019600 sec
MCREST 0.0000000 sec
MCMX 0.0150000 sec
P2 31.00 usec

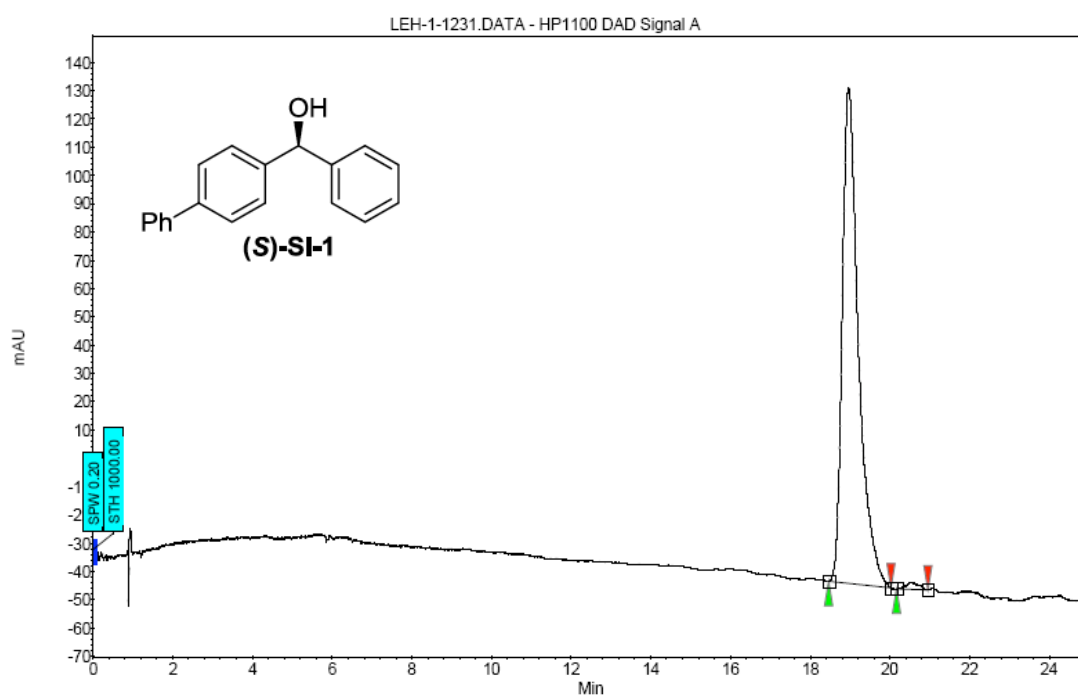
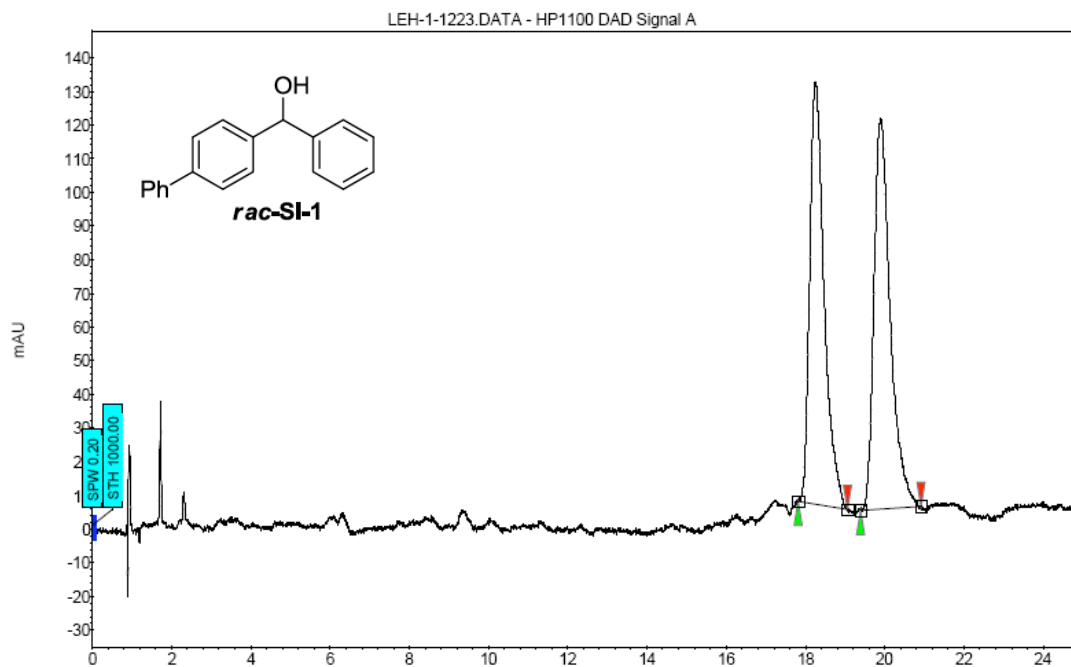
***** CHANNEL f1 *****
NUC1 13C
P1 15.50 usec
P11 500.00 usec
P12 2000.00 usec
PL0 120.00 dB
PL1 -1.00 dB
SFO1 125.7842548 MHz
SP1 3.20 dB
SP2 3.20 dB
SFO2 0.5200000 MHz
SFO3 0.5200000 MHz
SFO4 0.5200000 MHz
SFO5 0.5200000 MHz
SFO6 0.5200000 MHz
SFO7 0.5200000 MHz
SFO8 0.5200000 MHz
SFO9 0.5200000 MHz
SFO10 0.5200000 MHz
SFO11 0.5200000 MHz
SFO12 0.5200000 MHz
SFO13 0.5200000 MHz
SFO14 0.5200000 MHz
SFO15 0.5200000 MHz
SFO16 0.5200000 MHz
SFO17 0.5200000 MHz
SFO18 0.5200000 MHz
SFO19 0.5200000 MHz
SFO20 0.5200000 MHz
SFO21 0.5200000 MHz
SFO22 0.5200000 MHz
SFO23 0.5200000 MHz
SFO24 0.5200000 MHz
SFO25 0.5200000 MHz
SFO26 0.5200000 MHz
SFO27 0.5200000 MHz
SFO28 0.5200000 MHz
SFO29 0.5200000 MHz
SFO30 0.5200000 MHz
SFO31 0.5200000 MHz
SFO32 0.5200000 MHz
SFO33 0.5200000 MHz
SFO34 0.5200000 MHz
SFO35 0.5200000 MHz
SFO36 0.5200000 MHz
SFO37 0.5200000 MHz
SFO38 0.5200000 MHz
SFO39 0.5200000 MHz
SFO40 0.5200000 MHz
SFO41 0.5200000 MHz
SFO42 0.5200000 MHz
SFO43 0.5200000 MHz
SFO44 0.5200000 MHz
SFO45 0.5200000 MHz
SFO46 0.5200000 MHz
SFO47 0.5200000 MHz
SFO48 0.5200000 MHz
SFO49 0.5200000 MHz
SFO50 0.5200000 MHz
SFO51 0.5200000 MHz
SFO52 0.5200000 MHz
SFO53 0.5200000 MHz
SFO54 0.5200000 MHz
SFO55 0.5200000 MHz
SFO56 0.5200000 MHz
SFO57 0.5200000 MHz
SFO58 0.5200000 MHz
SFO59 0.5200000 MHz
SFO60 0.5200000 MHz
SFO61 0.5200000 MHz
SFO62 0.5200000 MHz
SFO63 0.5200000 MHz
SFO64 0.5200000 MHz
SFO65 0.5200000 MHz
SFO66 0.5200000 MHz
SFO67 0.5200000 MHz
SFO68 0.5200000 MHz
SFO69 0.5200000 MHz
SFO70 0.5200000 MHz
SFO71 0.5200000 MHz
SFO72 0.5200000 MHz
SFO73 0.5200000 MHz
SFO74 0.5200000 MHz
SFO75 0.5200000 MHz
SFO76 0.5200000 MHz
SFO77 0.5200000 MHz
SFO78 0.5200000 MHz
SFO79 0.5200000 MHz
SFO80 0.5200000 MHz
SFO81 0.5200000 MHz
SFO82 0.5200000 MHz
SFO83 0.5200000 MHz
SFO84 0.5200000 MHz
SFO85 0.5200000 MHz
SFO86 0.5200000 MHz
SFO87 0.5200000 MHz
SFO88 0.5200000 MHz
SFO89 0.5200000 MHz
SFO90 0.5200000 MHz
SFO91 0.5200000 MHz
SFO92 0.5200000 MHz
SFO93 0.5200000 MHz
SFO94 0.5200000 MHz
SFO95 0.5200000 MHz
SFO96 0.5200000 MHz
SFO97 0.5200000 MHz
SFO98 0.5200000 MHz
SFO99 0.5200000 MHz
SFO100 0.5200000 MHz

***** CHANNEL f2 *****
CPROG2 waltz16
NUC2 1H
PCPD2 100.00 usec
PL2 1.00 dB
PL12 24.50 dB
SFO2 500.2225011 MHz

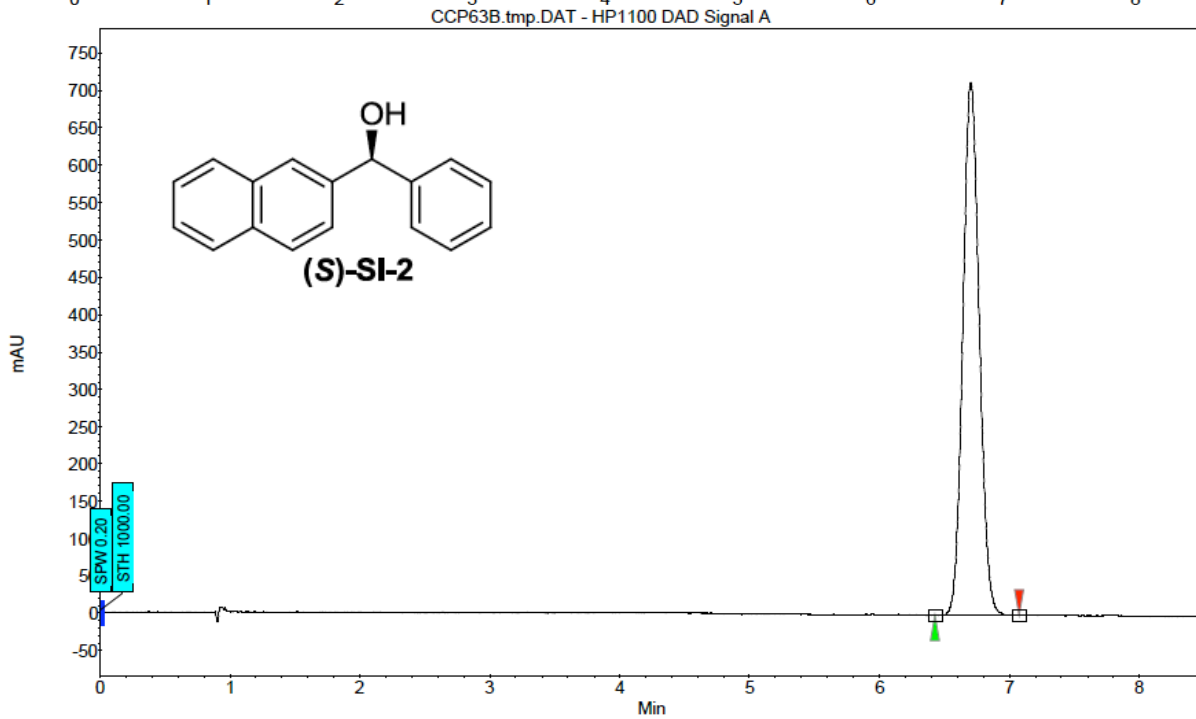
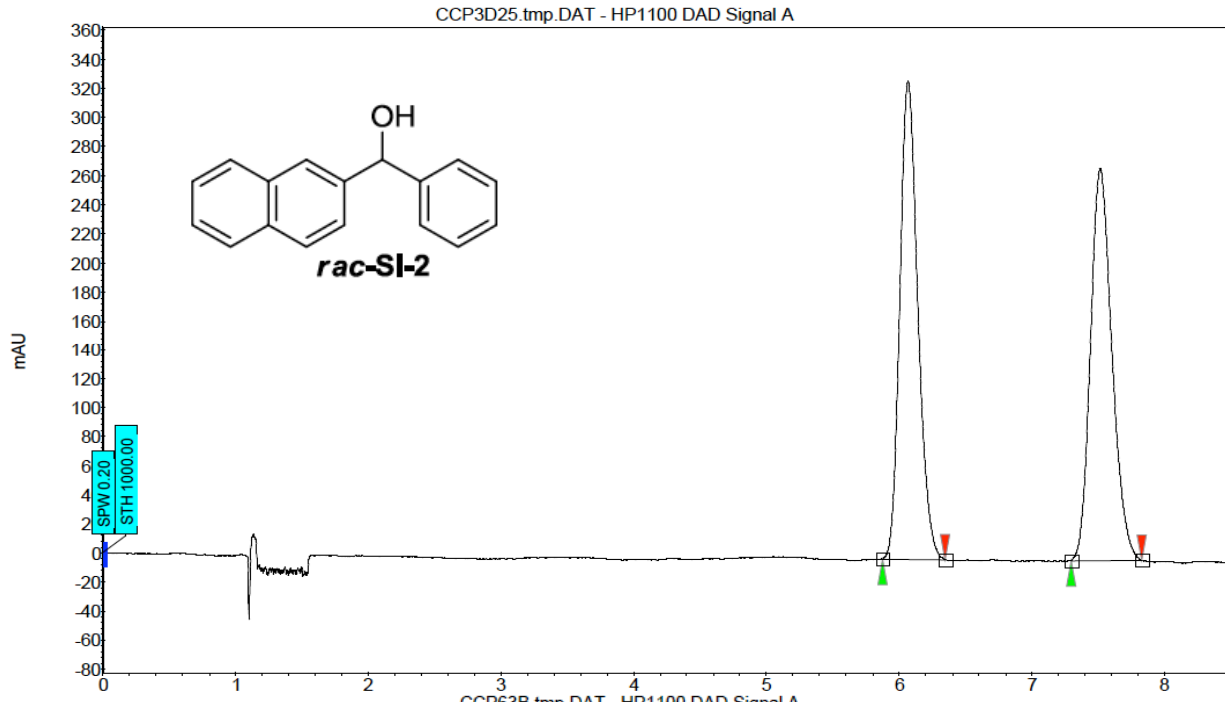
***** GRADIENT CHANNEL *****
GPNAM1 SINE.100
GPNAM2 SINE.100
GPX1 0.00 %
GPX2 0.00 %
GPF1 0.00 %
GPF2 0.00 %
GPZ1 30.00 %
GPZ2 50.00 %
p15 500.00 usec
p16 1000.00 usec

F2 - Processing parameters
SI 65536
SF 125.7804103 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
PC 2.00

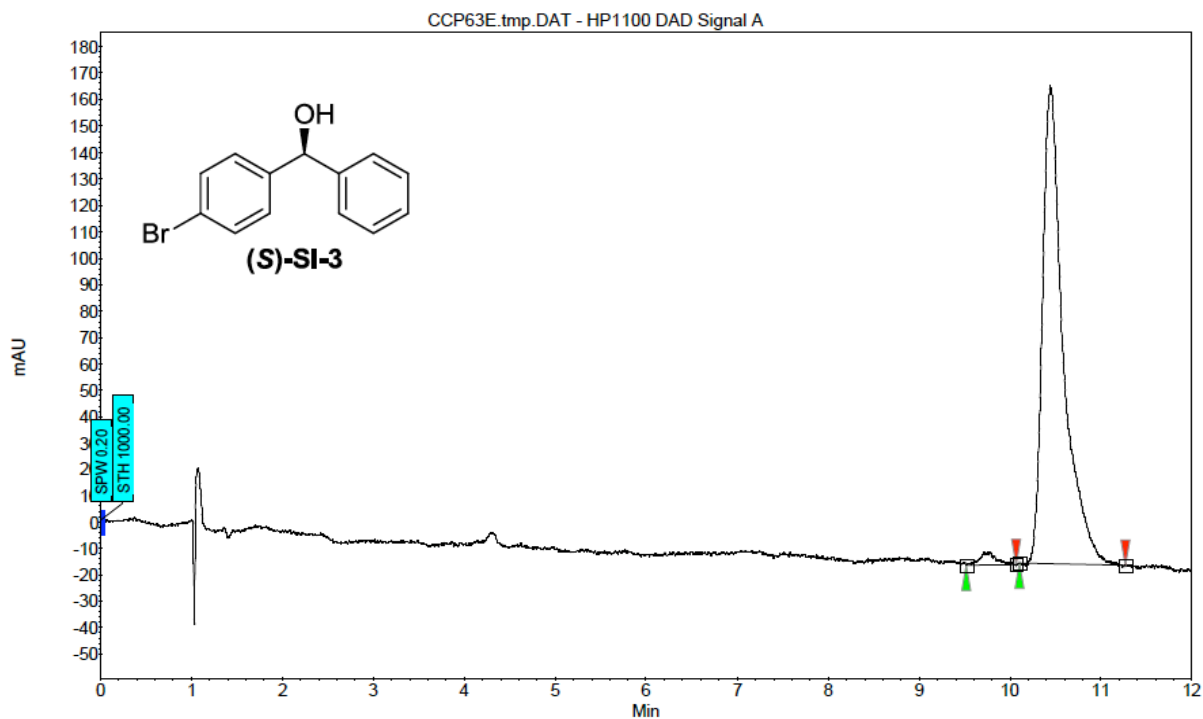
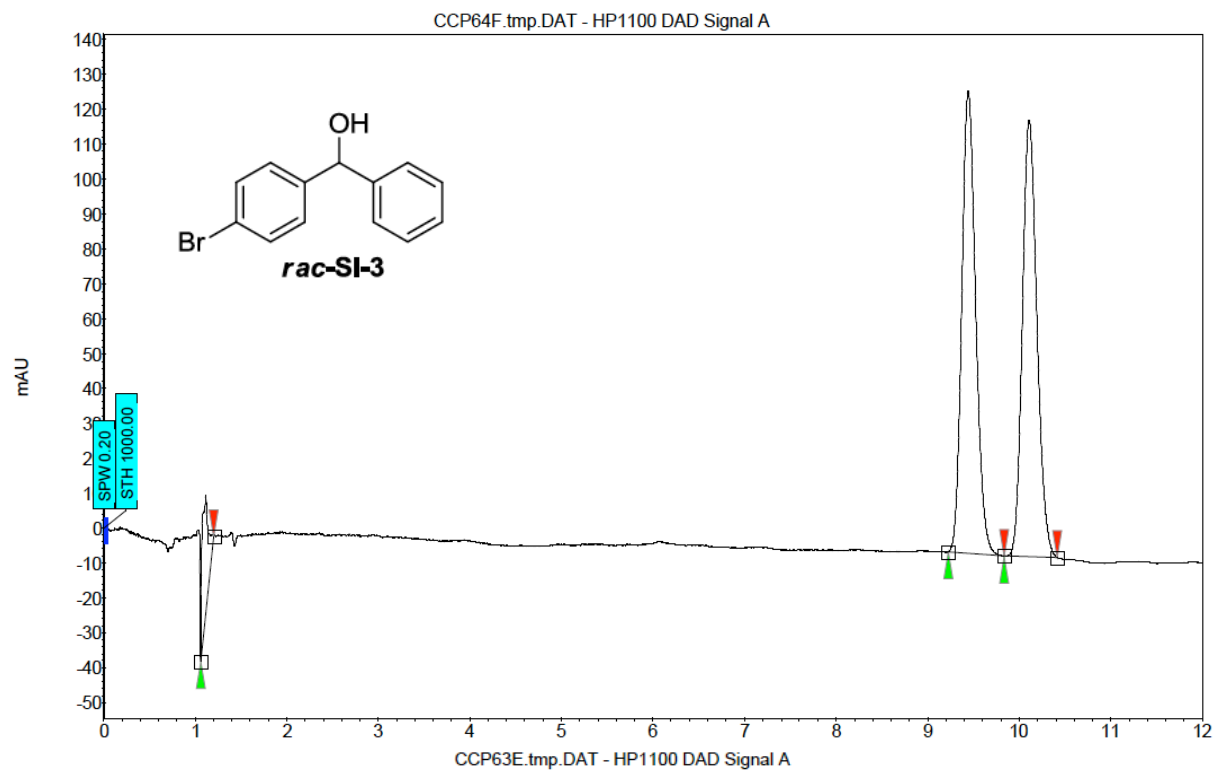
1D NMR plot parameters
CX 22.80 cm
CY 15.65 cm
F1P 150.000 ppm
F1 20124.87 Hz
F2P 20.000 ppm
F2 2515.61 Hz
PPMC 6.14035 ppm/cm
HZCM 772.33588 Hz/cm
    
```



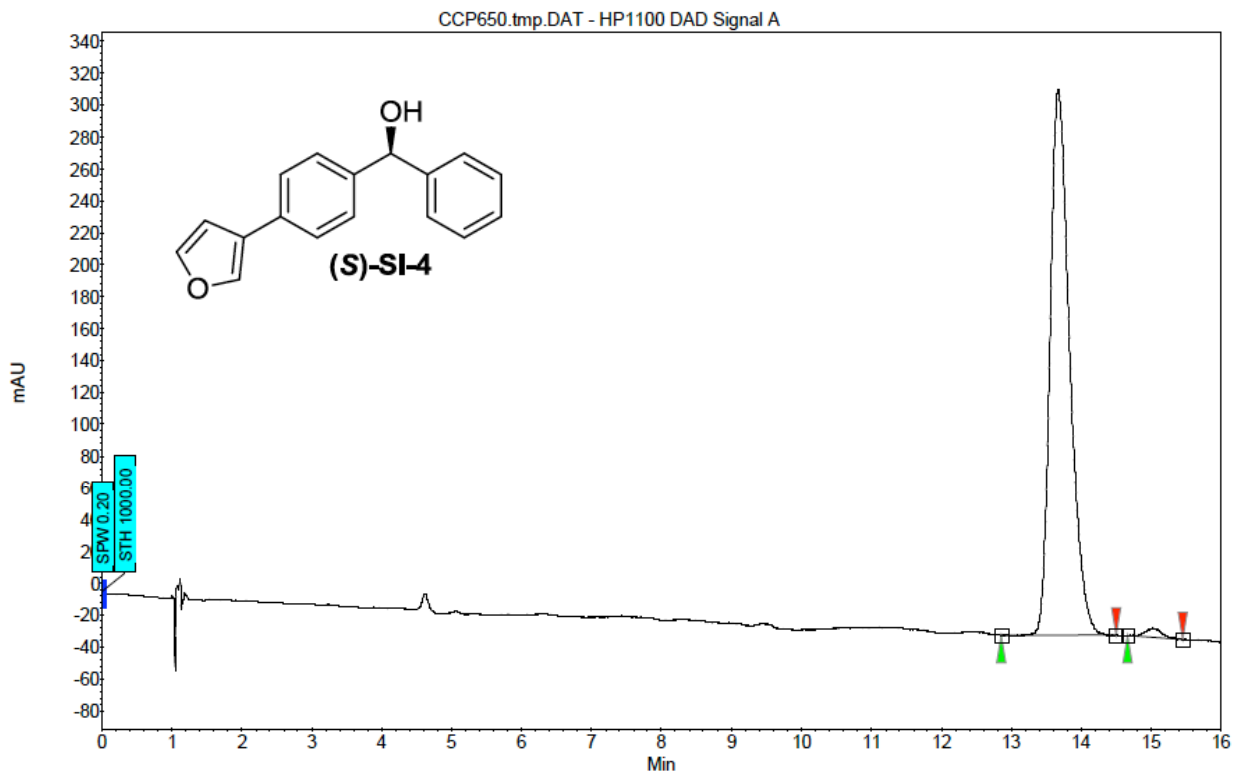
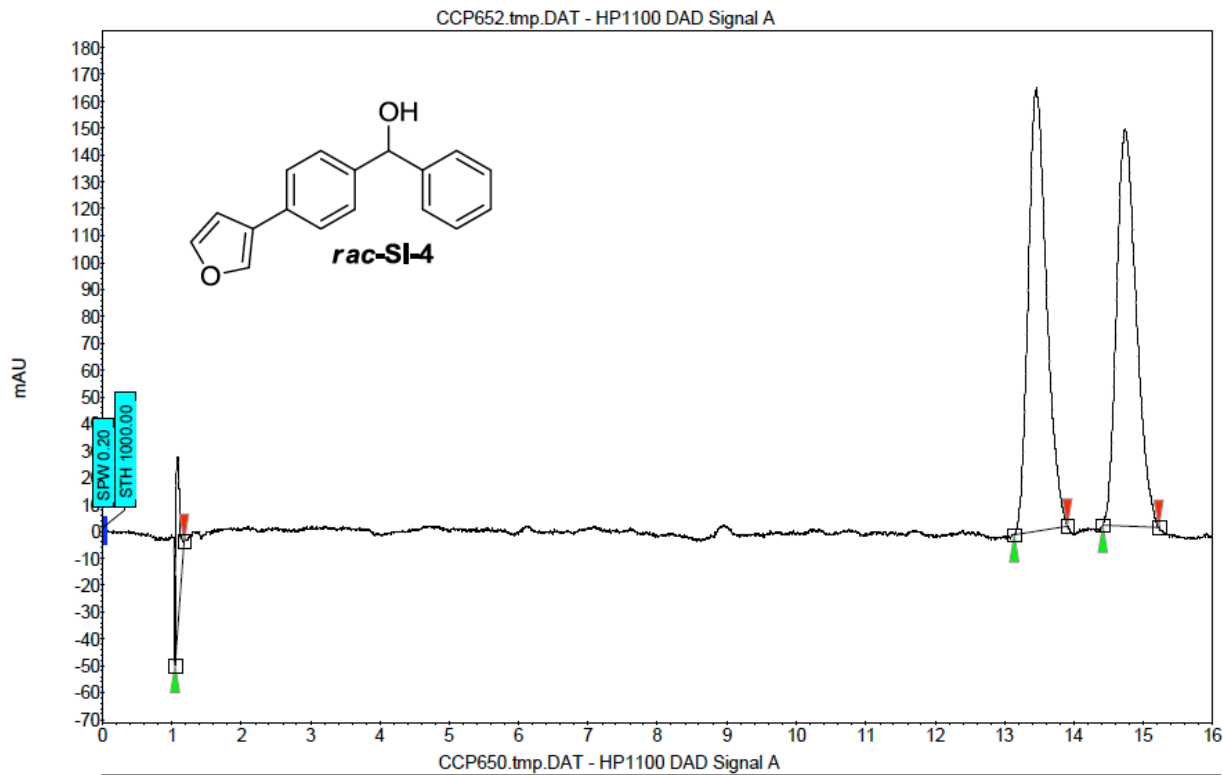
Index	Name	Start	Time	End	RT Offset	Quantity	Height	Area	Area
		[Min]	[Min]	[Min]	[Min]	[% Area]	[μ V]	[μ V.Min]	[%]
1	UNKNOWN	18.46	18.97	20.02	0.00	98.91	175.3	82.1	98.914
2	UNKNOWN	20.17	20.55	20.95	0.00	1.09	2.4	0.9	1.086
Total						100.00	177.7	83.0	100.000



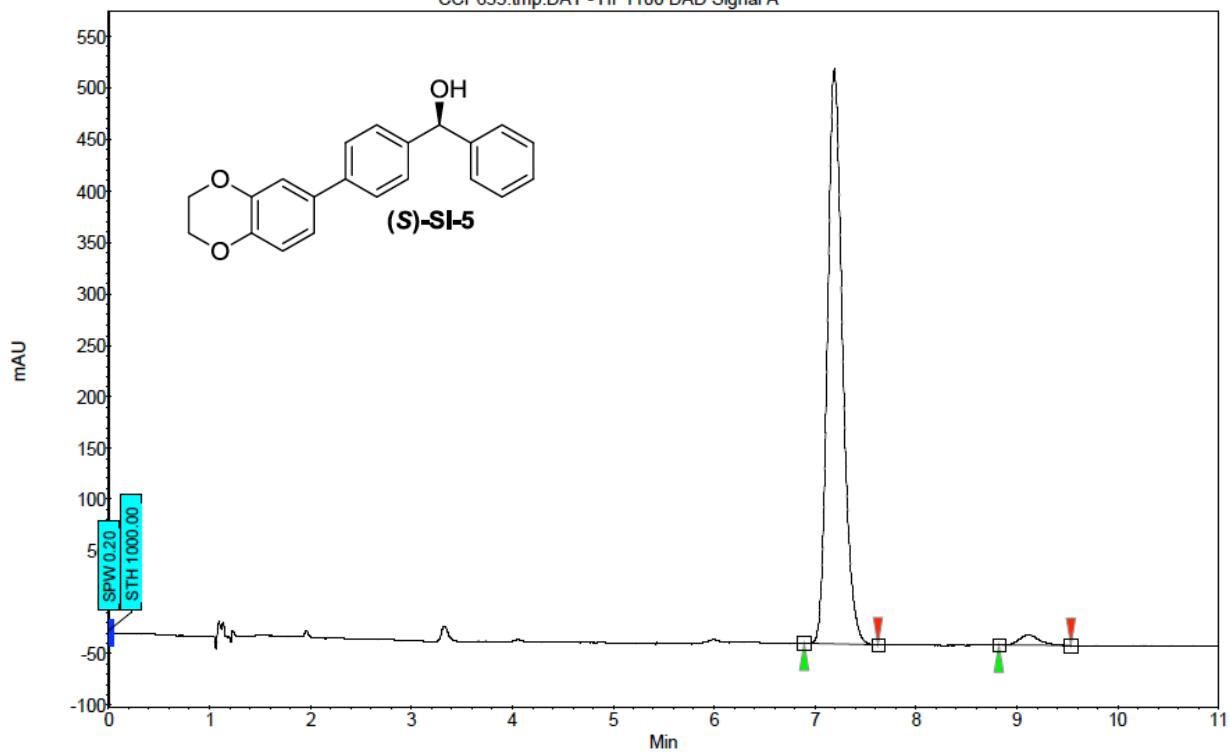
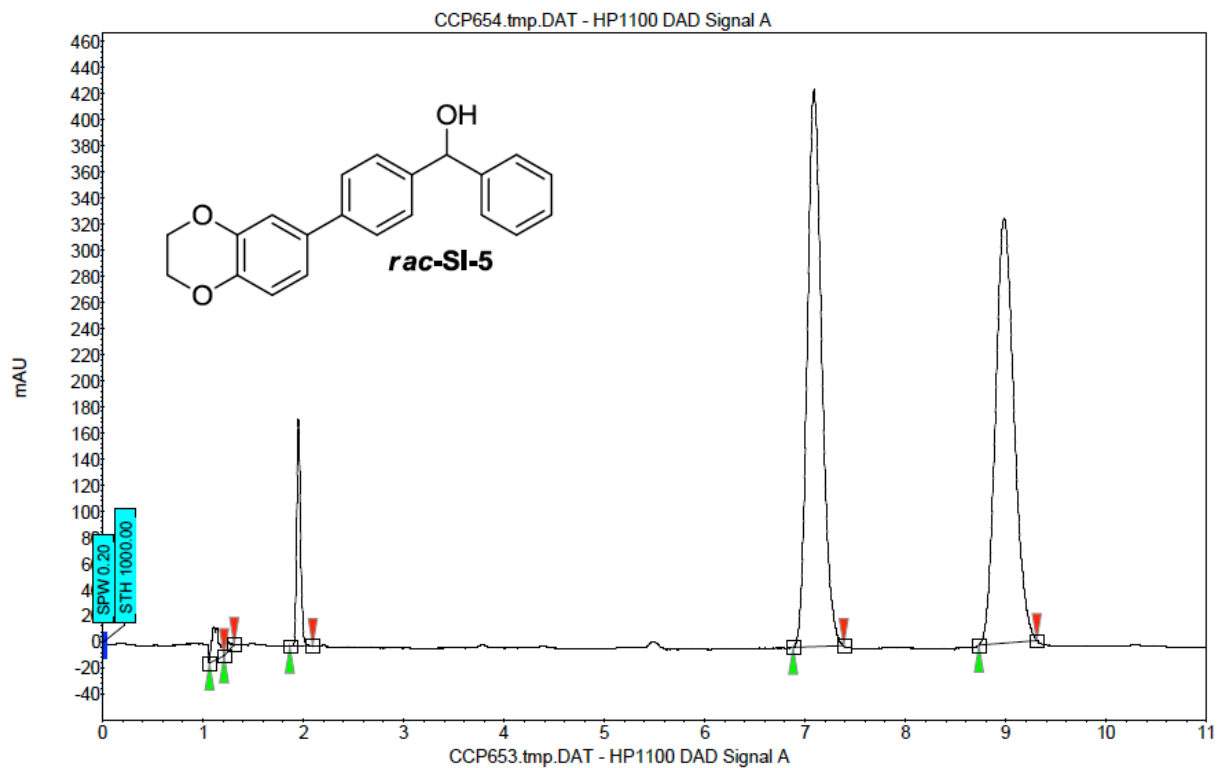
Index	Name	Start Time [Min]	Time [Min]	End [Min]	RT Offset [Min]	Quantity [% Area]	Height [μV]	Area [μV.Min]	Area [%]
1	UNKNOWN	6.43	6.70	7.08	0.00	100.00	712.9	104.3	100.000
Total						100.00	712.9	104.3	100.000



Index	Name	Start	Time	End	RT	Offset	Quantity	Height	Area	Area
		[Min]	[Min]	[Min]	[Min]	[Min]	[% Area]	[μ V]	[μ V.Min]	[%]
2	UNKNOWN	9.52	9.76	10.07		0.00	2.16	4.9	1.1	2.164
1	UNKNOWN	10.10	10.44	11.27		0.00	97.84	181.0	47.8	97.836
Total							100.00	185.8	48.9	100.000

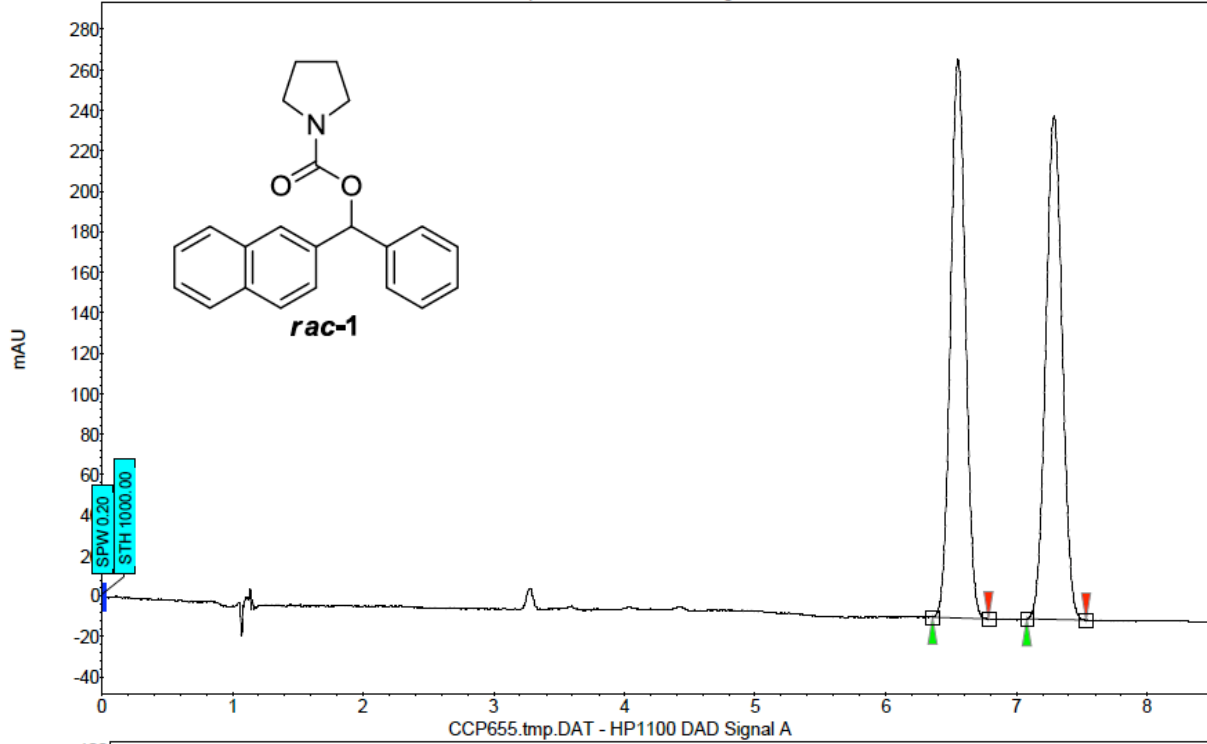


Index	Name	Start	Time	End	RT Offset	Quantity	Height	Area	Area
		[Min]	[Min]	[Min]	[Min]	[% Area]	[μ V]	[μ V.Min]	[%]
1	UNKNOWN	12.86	13.67	14.50	0.00	98.57	341.7	111.0	98.571
2	UNKNOWN	14.66	15.02	15.45	0.00	1.43	5.9	1.6	1.429
Total						100.00	347.6	112.6	100.000

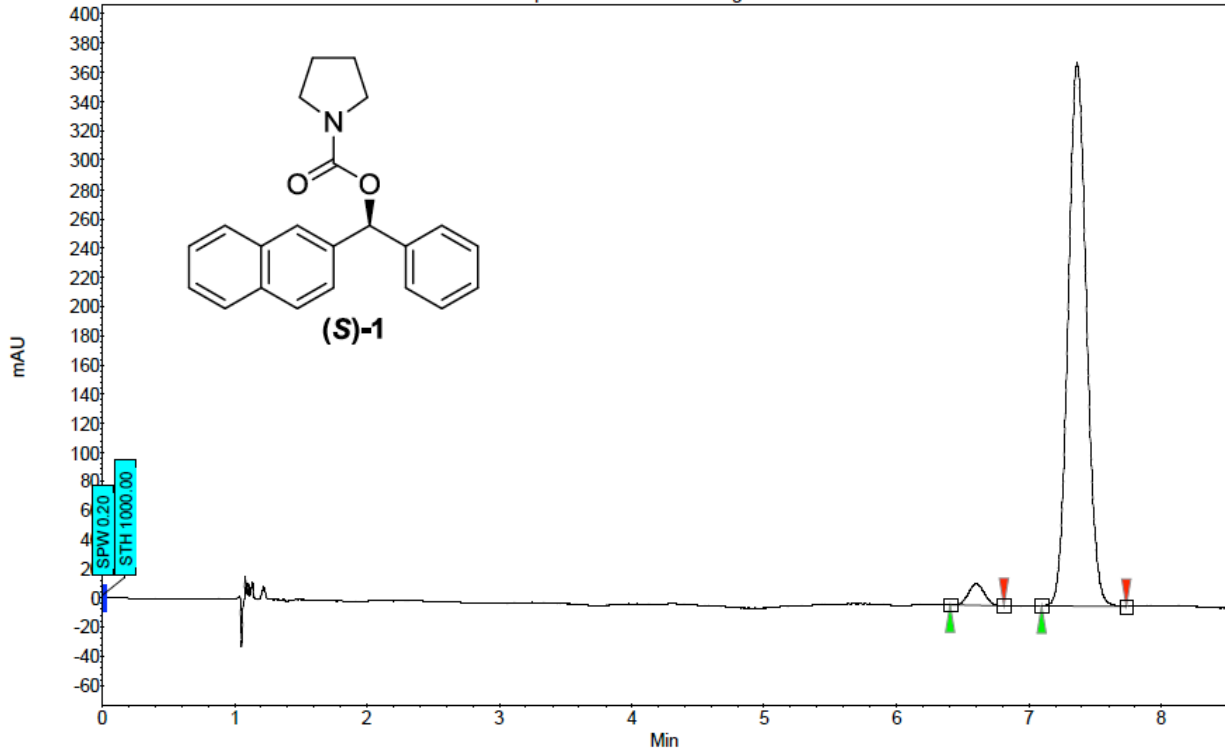


Index	Name	Start [Min]	Time [Min]	End [Min]	RT Offset [Min]	Quantity [% Area]	Height [μV]	Area [μV.Min]	Area [%]
1	UNKNOWN	6.89	7.19	7.62	0.00	97.79	558.2	96.4	97.790
2	UNKNOWN	8.82	9.11	9.53	0.00	2.21	9.8	2.2	2.210
Total						100.00	568.0	98.6	100.000

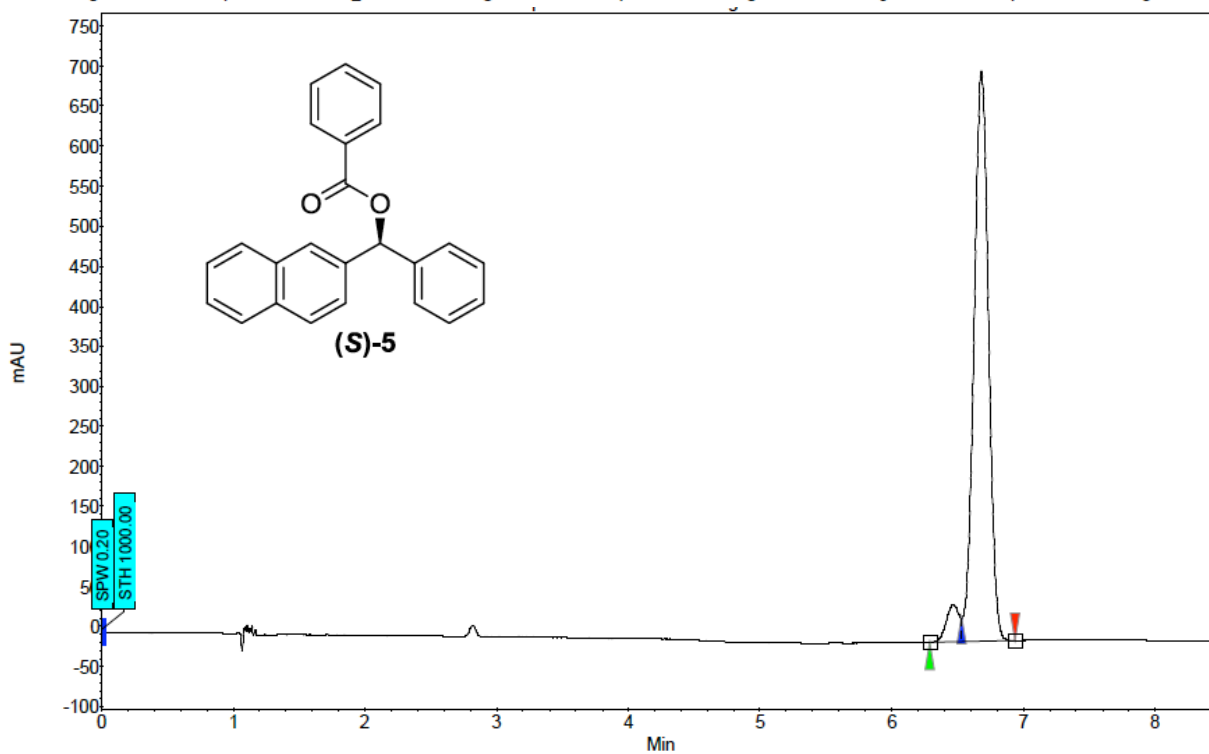
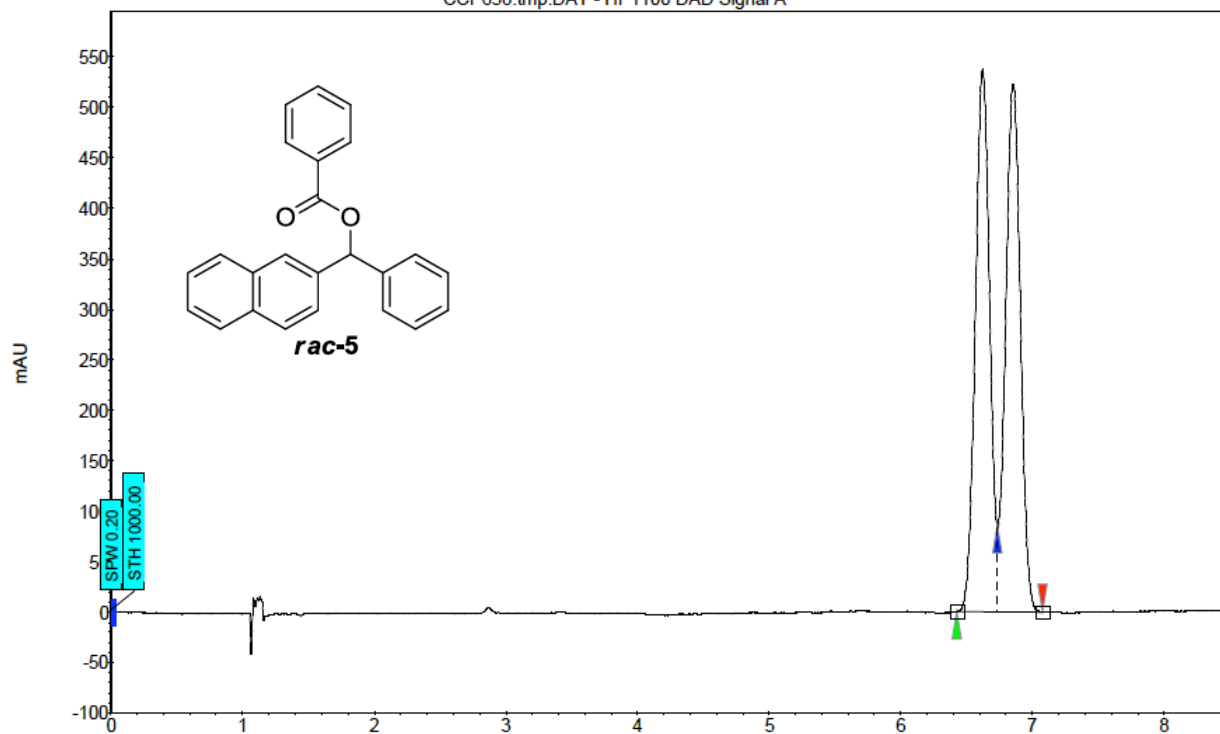
CCP656.tmp.DAT - HP1100 DAD Signal A



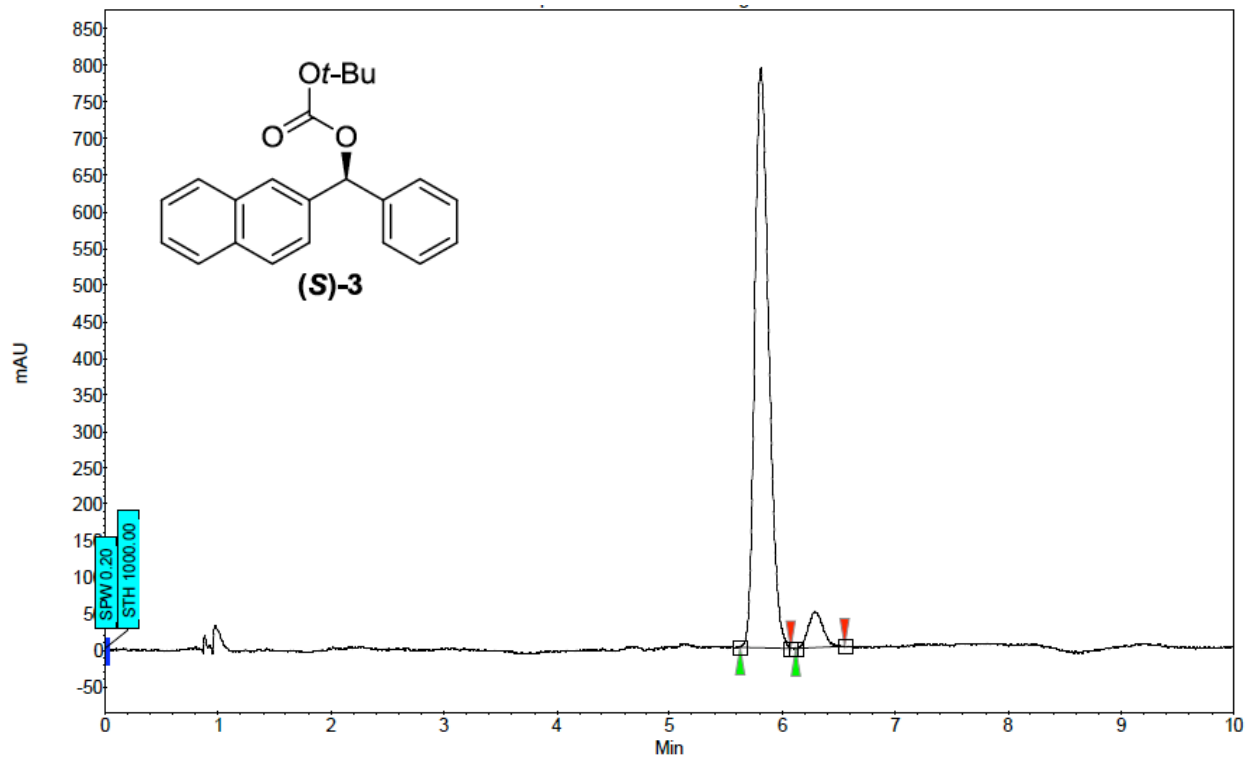
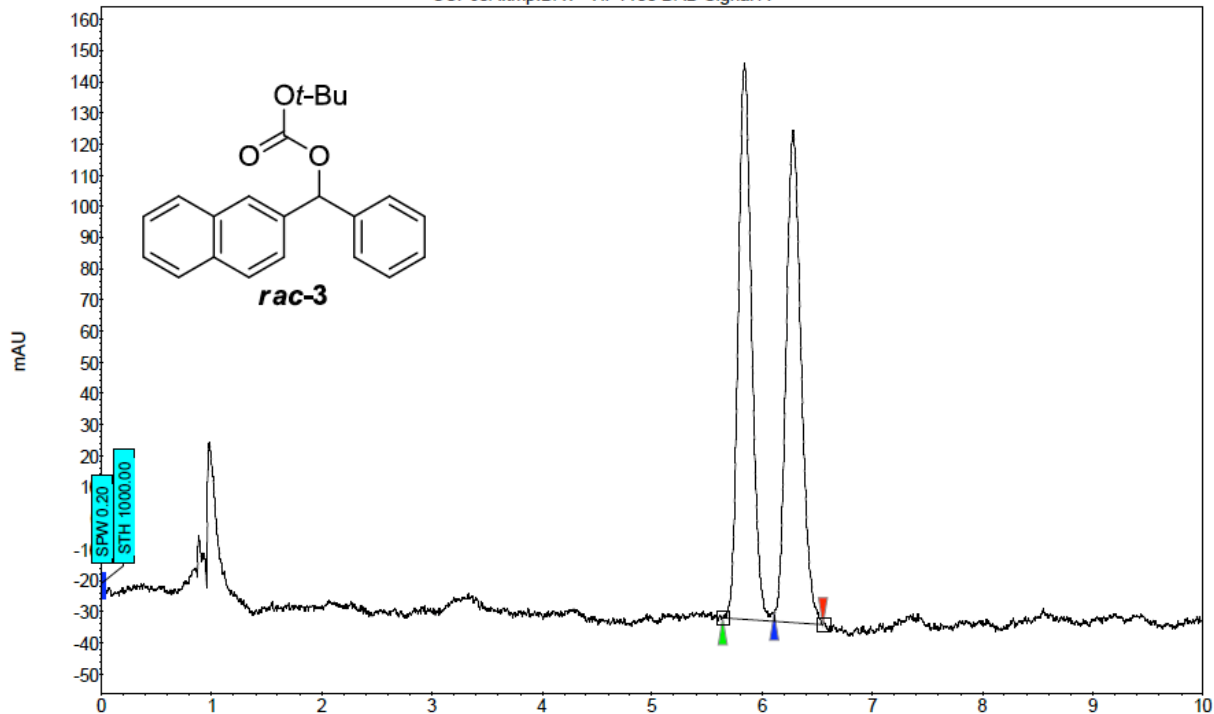
CCP655.tmp.DAT - HP1100 DAD Signal A



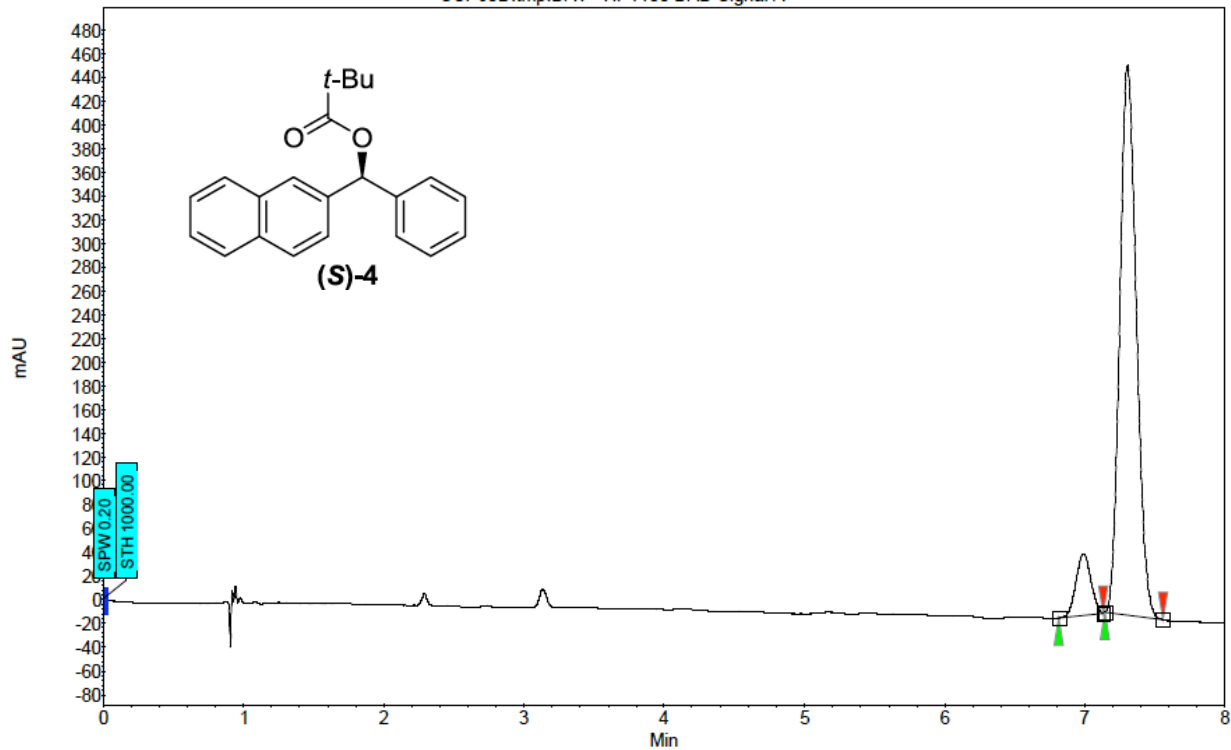
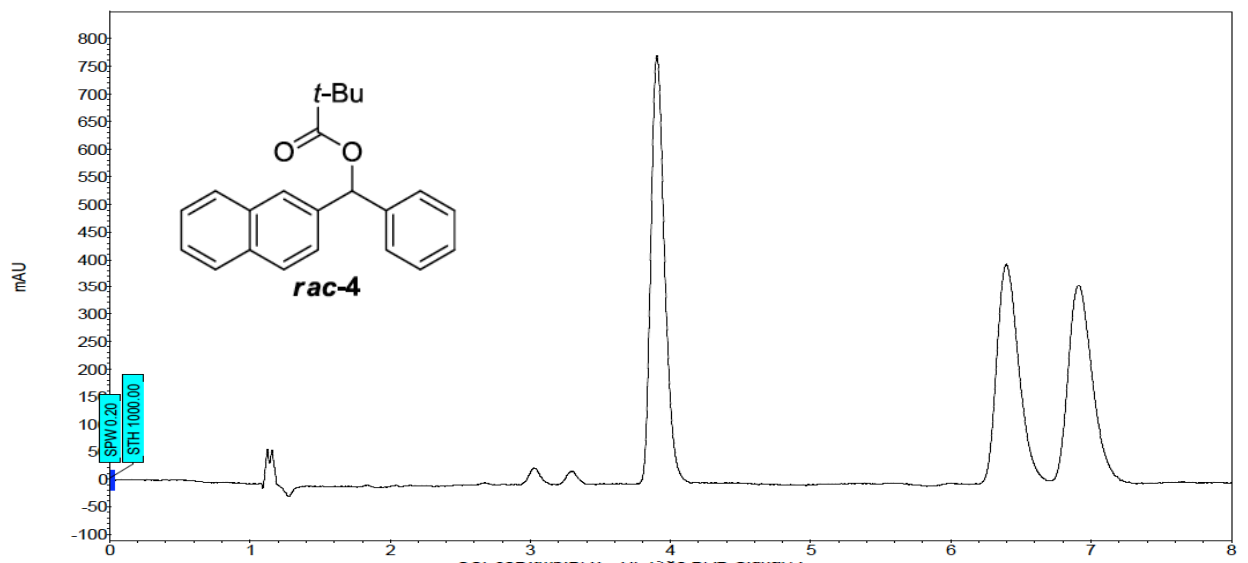
Index	Name	Start [Min]	Time [Min]	End [Min]	RT Offset [Min]	Quantity [% Area]	Height [μV]	Area [μV.Min]	Area [%]
1	UNKNOWN	6.41	6.60	6.81	0.00	3.40	14.9	2.1	3.400
2	UNKNOWN	7.10	7.37	7.74	0.00	96.60	372.0	59.5	96.600
Total						100.00	386.9	61.6	100.000



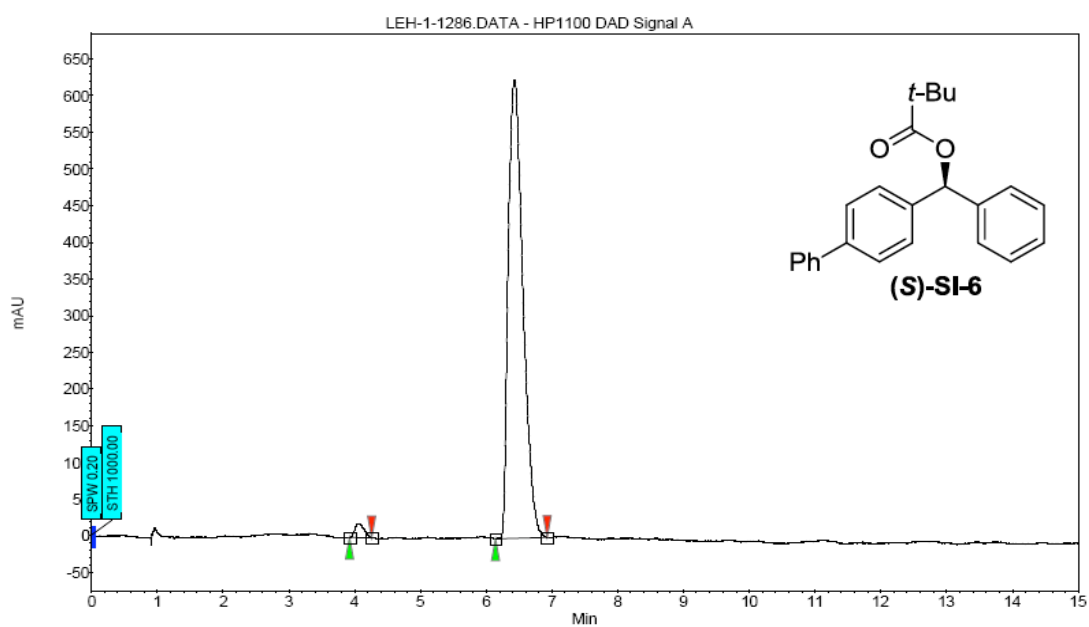
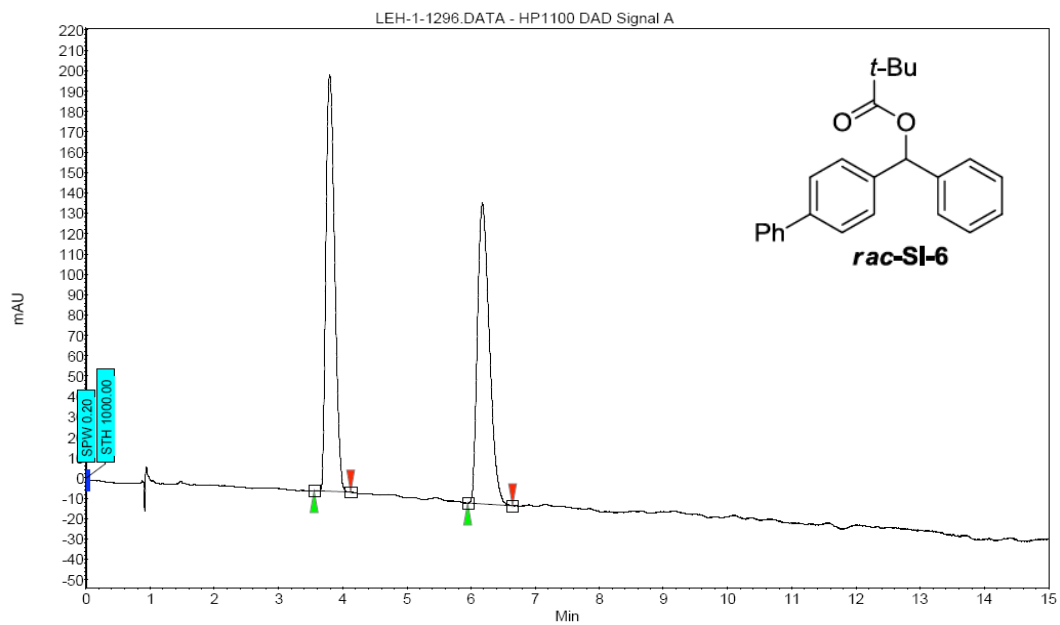
Index	Name	Start [Min]	Time [Min]	End [Min]	RT Offset [Min]	Quantity [% Area]	Height [µV]	Area [µV.Min]	Area [%]
1	UNKNOWN	6.29	6.47	6.53	0.00	5.45	46.2	5.2	5.451
2	UNKNOWN	6.53	6.68	6.94	0.00	94.55	711.7	90.9	94.549
Total						100.00	757.9	96.2	100.000



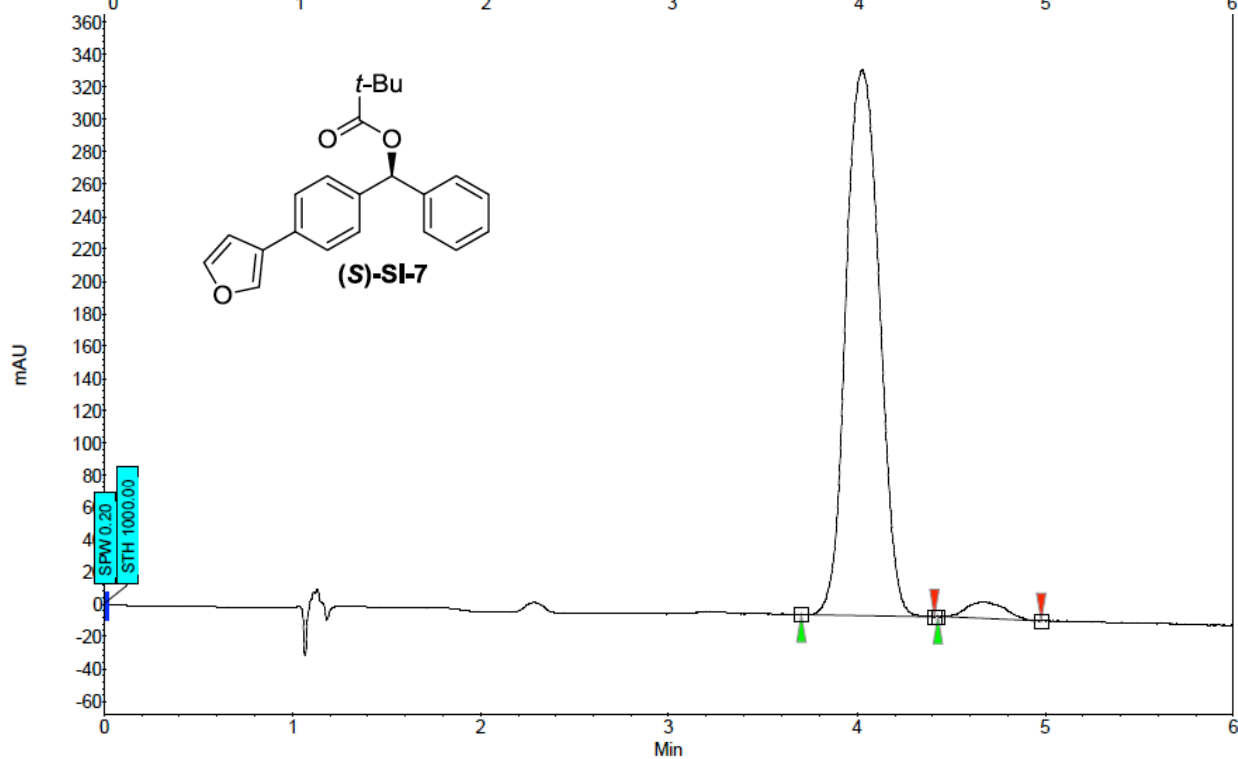
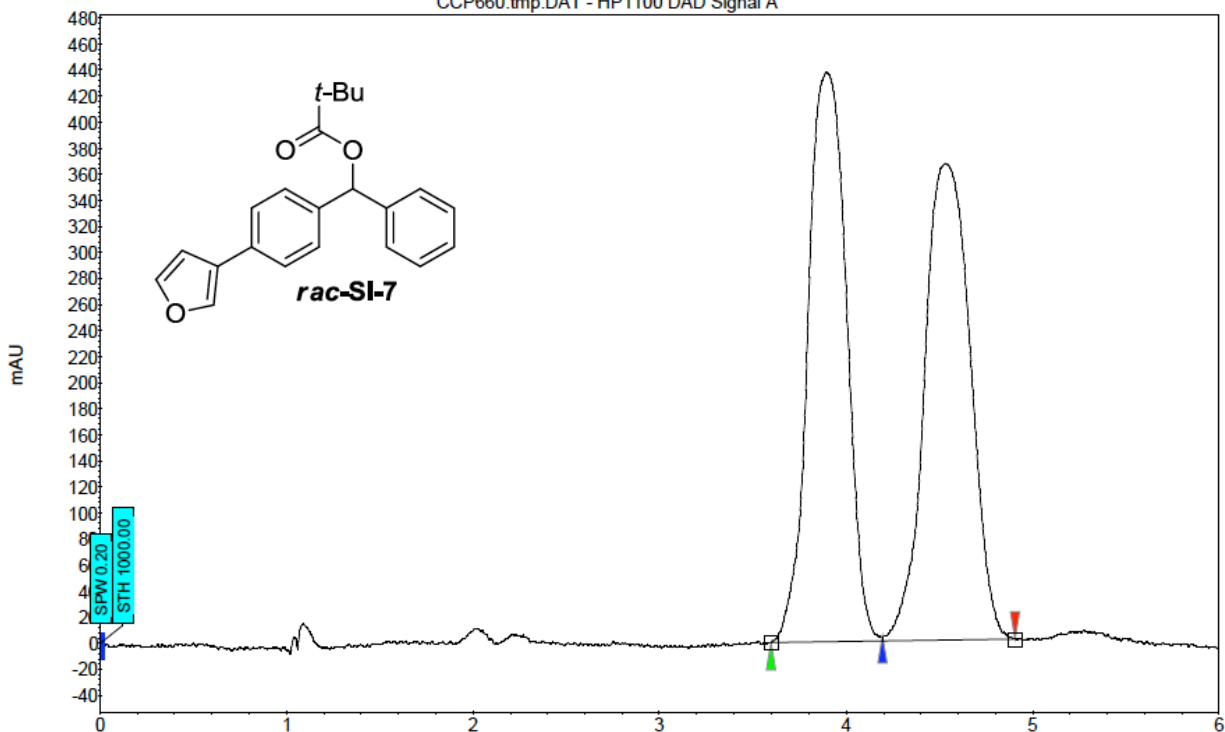
Index	Name	Start Time [Min]	End Time [Min]	RT Offset [Min]	Quantity [% Area]	Height [µV]	Area [µV.Min]	Area [%]	
1	UNKNOWN	5.63	5.81	6.08	0.00	93.79	791.8	113.4	93.788
2	UNKNOWN	6.12	6.29	6.55	0.00	6.21	50.0	7.5	6.212
Total						100.00	841.8	120.9	100.000



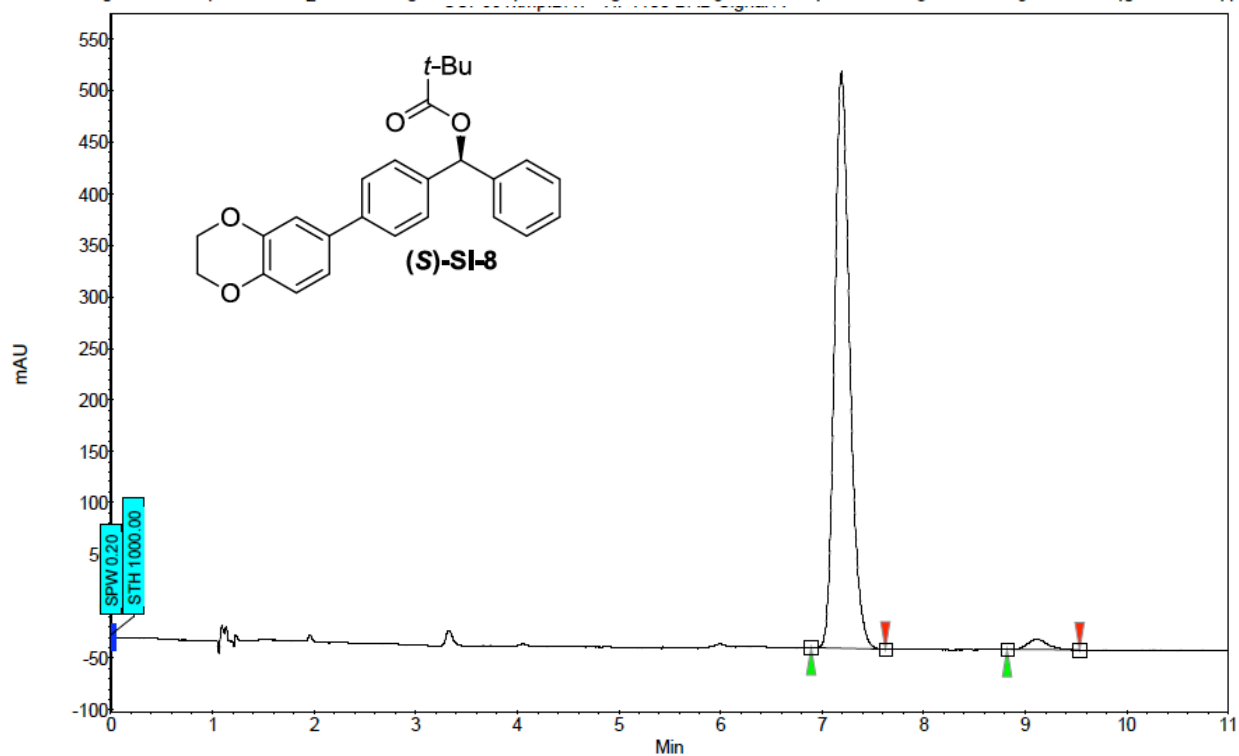
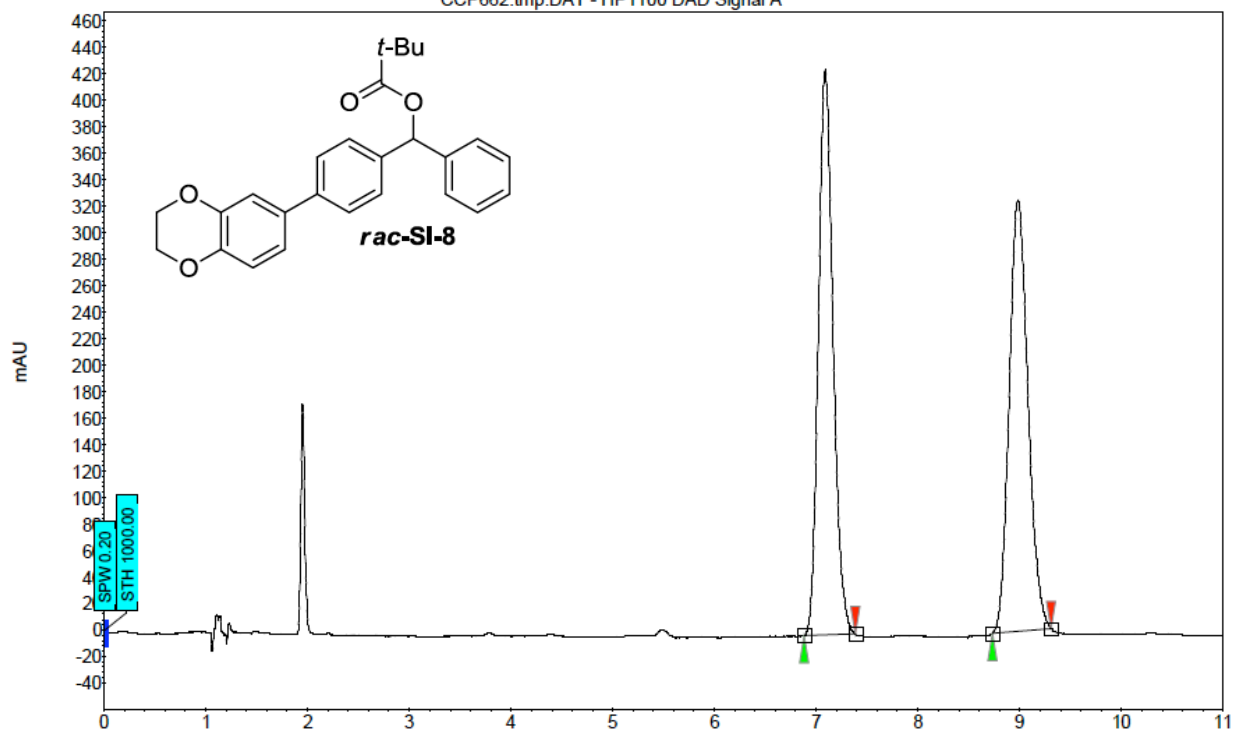
Index	Name	Start Time [Min]	End Time [Min]	RT Offset [Min]	Quantity [% Area]	Height [μV]	Area [μV.Min]	Area [%]
1	UNKNOWN	6.82	6.99	7.13	0.00	9.06	51.7	6.3
2	UNKNOWN	7.14	7.30	7.56	0.00	90.94	463.0	63.5
Total					100.00	514.7	69.9	100.000



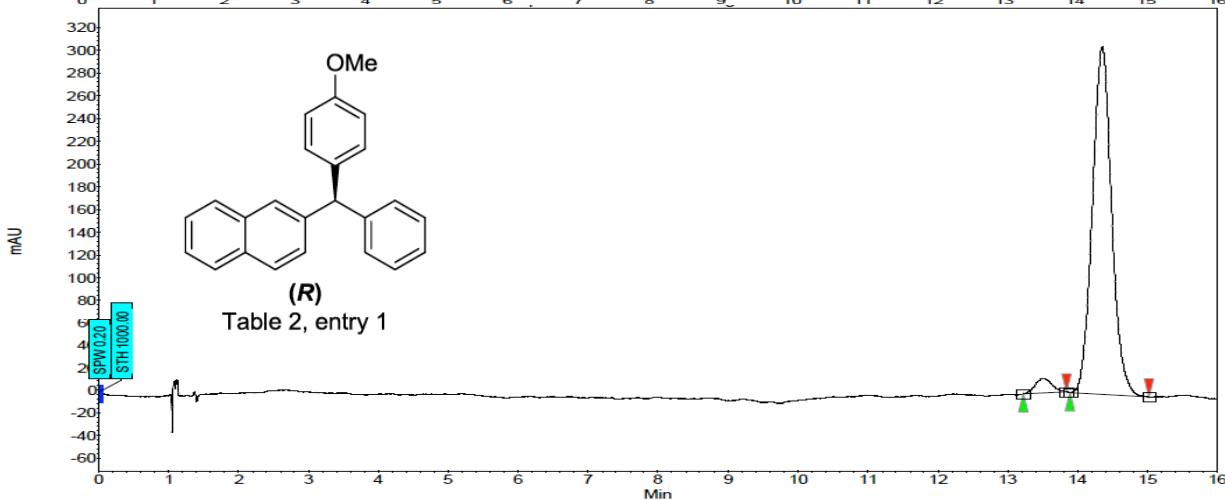
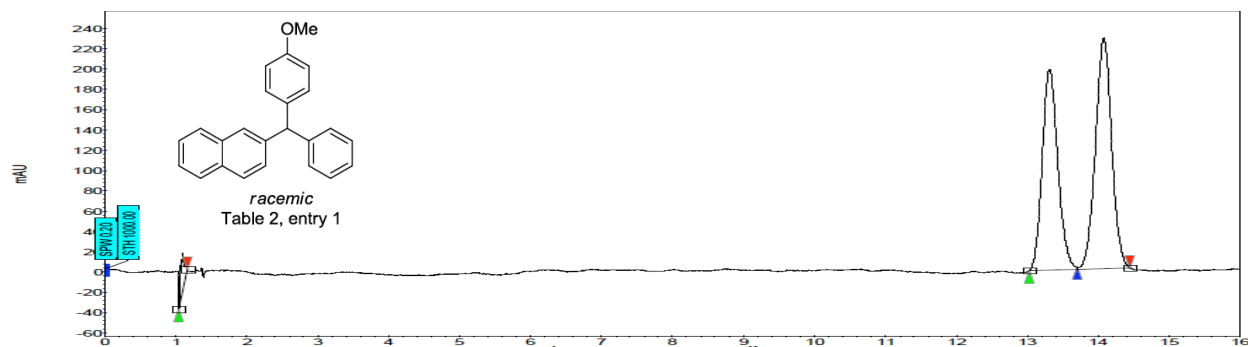
Index	Name	Start Time	End	RT Offset	Quantity	Height	Area	Area
		[Min]	[Min]	[Min]	[Min]	[% Area]	[μ V]	[μ V.Min]
2	UNKNOWN	3.93	4.06	4.26	0.00	1.88	19.0	3.1
1	UNKNOWN	6.14	6.43	6.93	0.00	98.12	625.4	161.3
Total						100.00	644.3	164.4



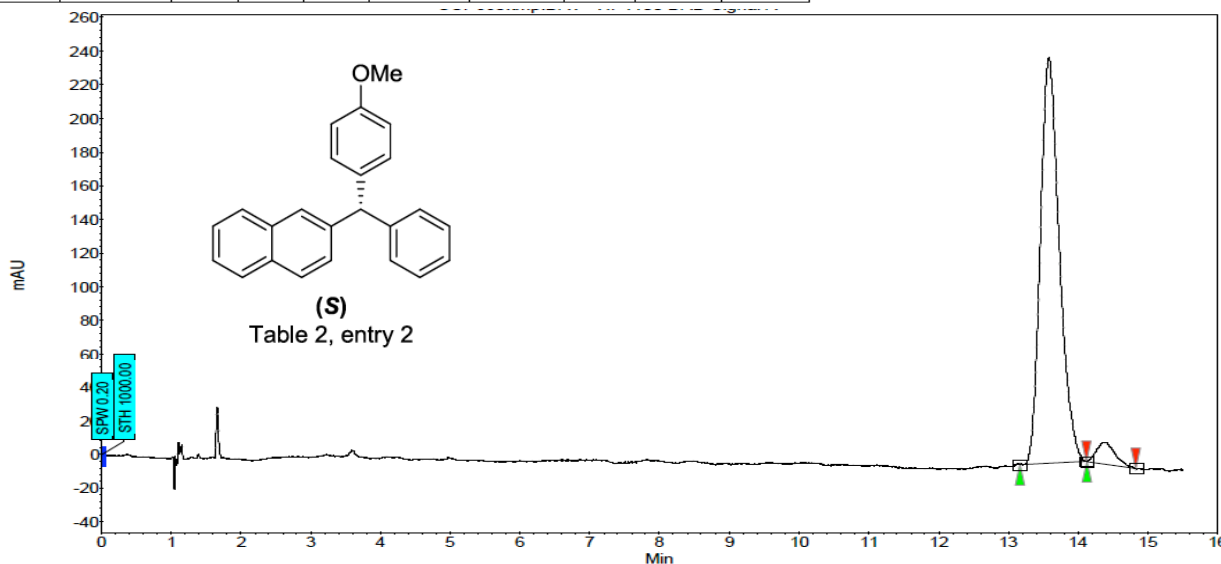
Index	Name	Start	Time	End	RT Offset	Quantity	Height	Area	Area
		[Min]	[Min]	[Min]	[Min]	[% Area]	[μ V]	[μ V.Min]	[%]
1	UNKNOWN	3.70	4.03	4.41	0.00	96.61	337.3	69.5	96.611
2	UNKNOWN	4.43	4.66	4.98	0.00	3.39	10.1	2.4	3.389
Total						100.00	347.4	71.9	100.000



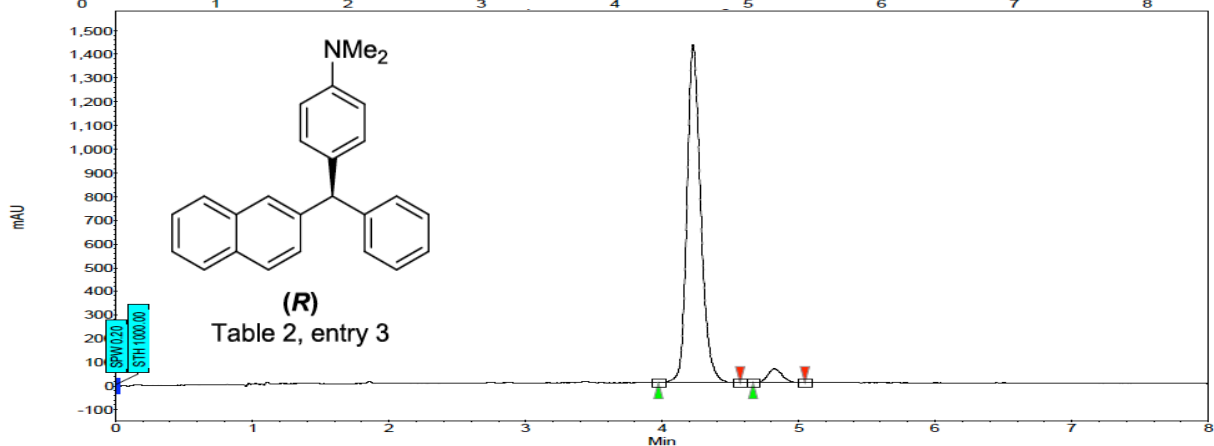
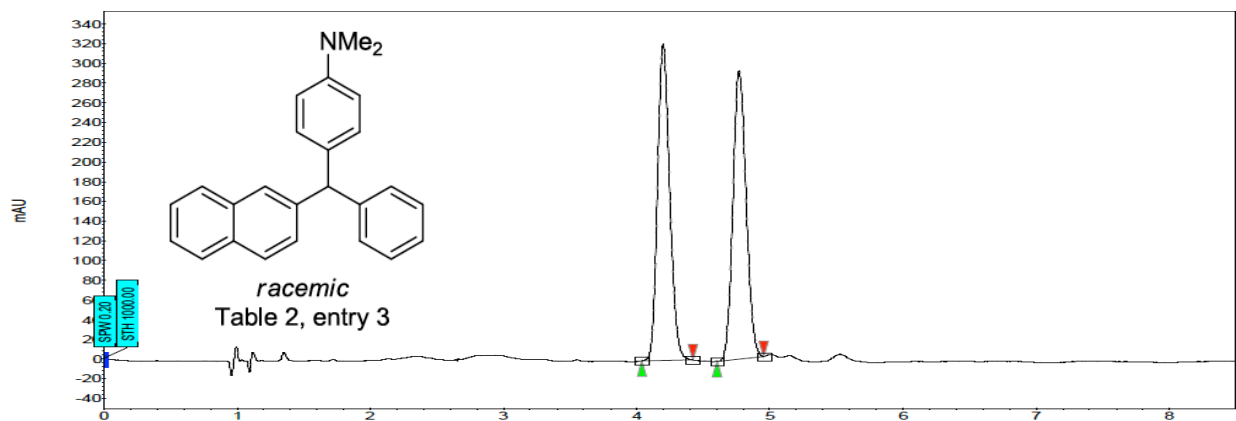
Index	Name	Start Time [Min]	End Time [Min]	RT Offset [Min]	Quantity [% Area]	Height [μV]	Area [μV.Min]	Area [%]
1	UNKNOWN	6.89	7.19	7.62	0.00	97.79	558.2	96.4
2	UNKNOWN	8.82	9.11	9.53	0.00	2.21	9.8	2.2
Total						100.00	568.0	98.6



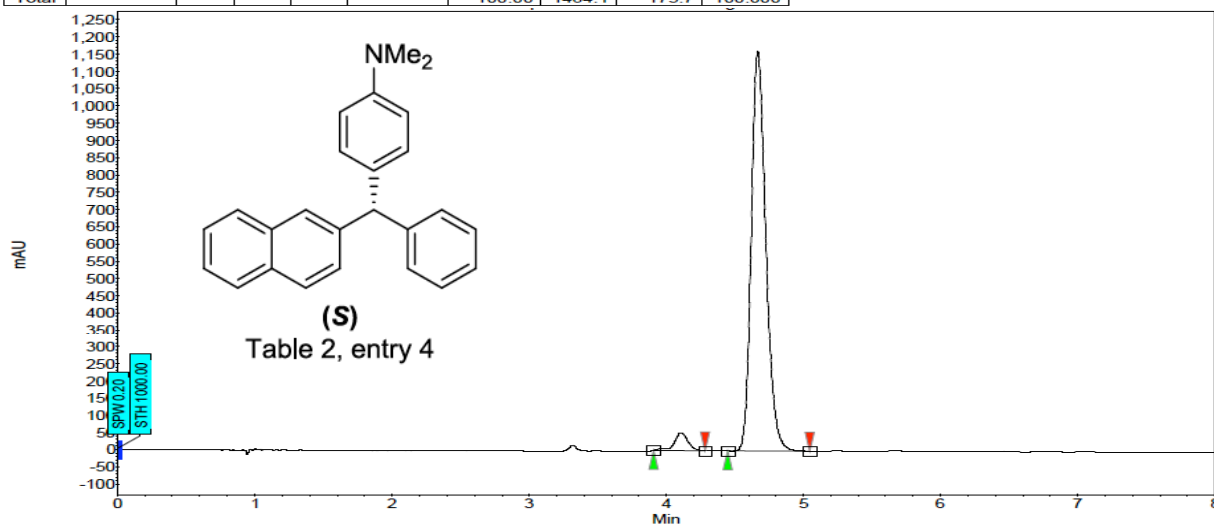
Index	Name	Start Time [Min]	Time [Min]	End [Min]	RT Offset [Min]	Quantity [% Area]	Height [μV]	Area [μV.Min]	Area [%]
1	UNKNOWN	13.22	13.52	13.84	0.00	3.51	13.0	3.6	3.505
2	UNKNOWN	13.89	14.35	15.02	0.00	96.49	306.8	98.0	96.495
Total						100.00	319.8	101.6	100.000



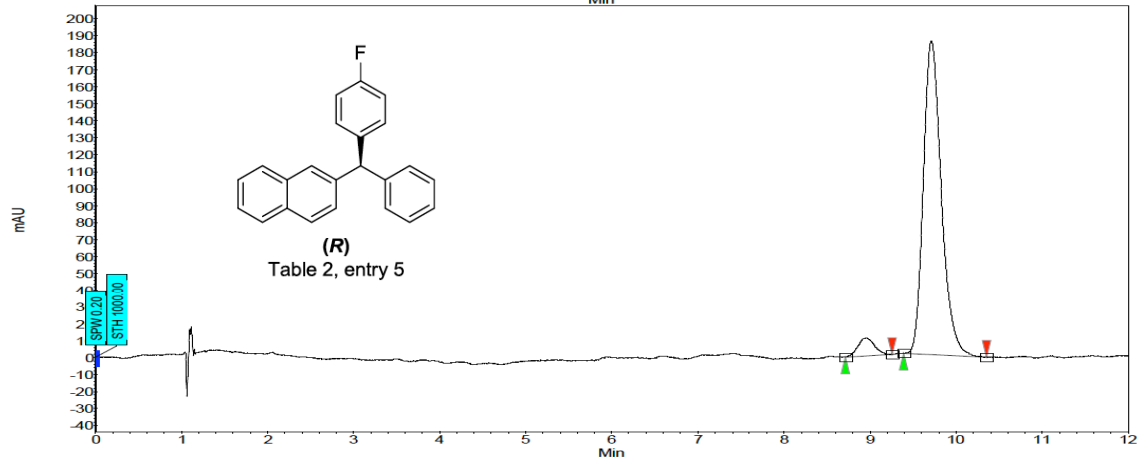
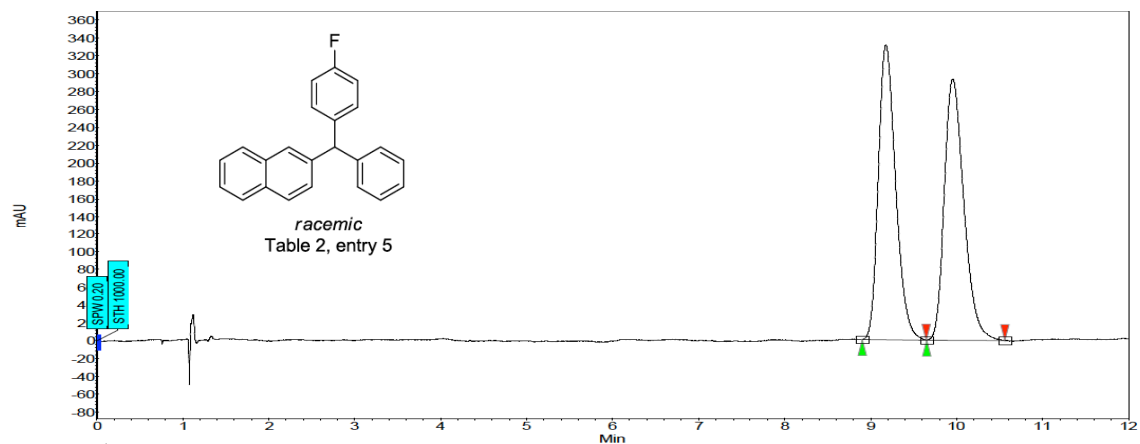
Index	Name	Start Time [Min]	Time [Min]	End [Min]	RT Offset [Min]	Quantity [% Area]	Height [μV]	Area [μV.Min]	Area [%]
1	UNKNOWN	13.16	13.58	14.11	0.00	95.00	241.1	76.1	94.998
2	UNKNOWN	14.12	14.40	14.83	0.00	5.00	12.9	4.0	5.002
Total						100.00	254.0	80.1	100.000



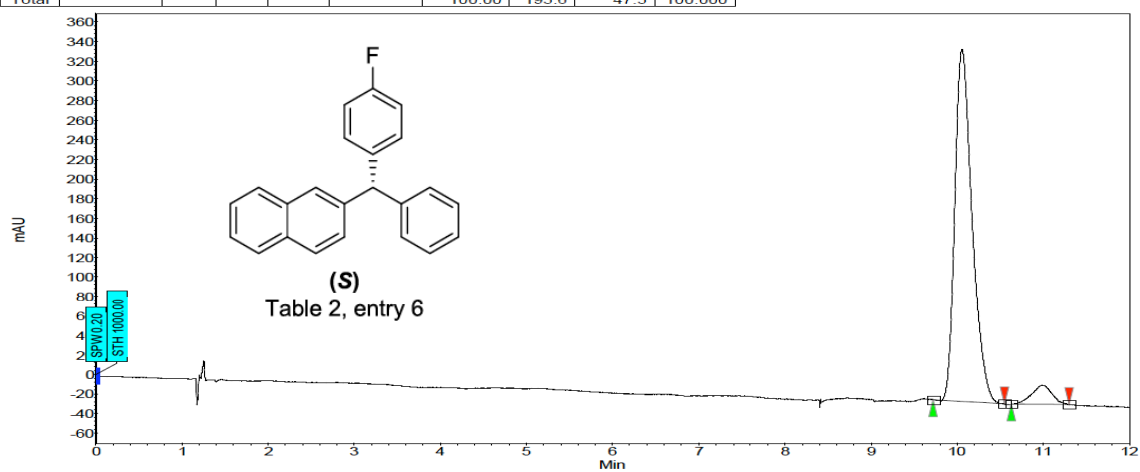
Index	Name	Start [Min]	Time [Min]	End [Min]	RT Offset [Min]	Quantity [% Area]	Height [μV]	Area [μV.Min]	Area [%]
1	UNKNOWN	3.98	4.23	4.57	0.00	95.82	1425.1	166.4	95.819
2	UNKNOWN	4.67	4.83	5.05	0.00	4.18	59.1	7.3	4.181
Total						100.00	1484.1	173.7	100.000



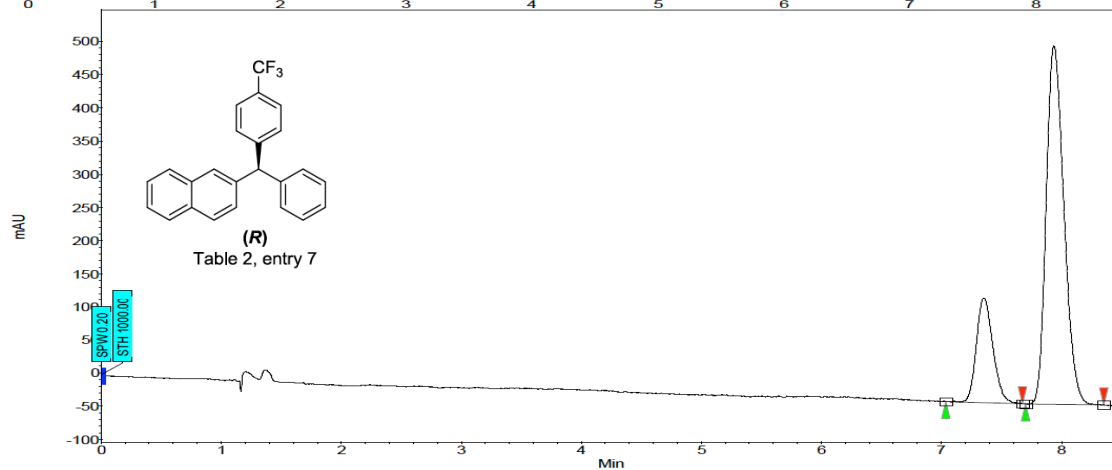
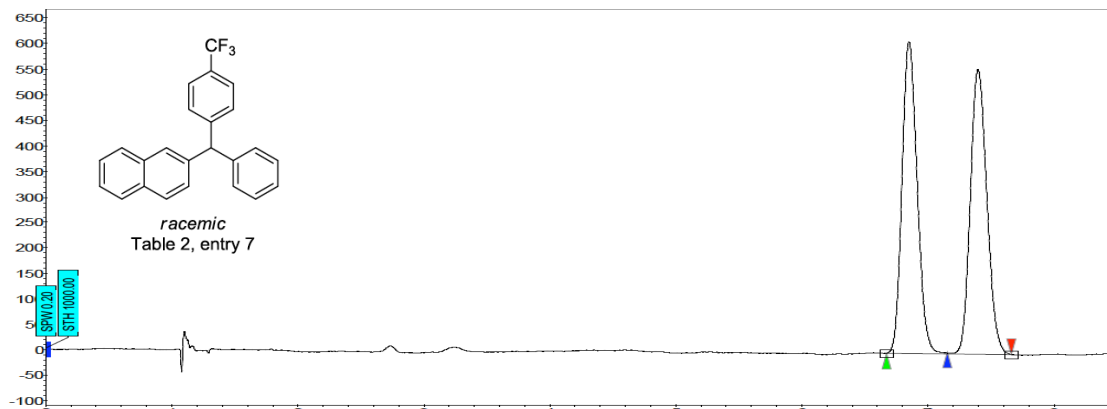
Index	Name	Start [Min]	Time [Min]	End [Min]	RT Offset [Min]	Quantity [% Area]	Height [μV]	Area [μV.Min]	Area [%]
2	UNKNOWN	3.91	4.11	4.29	0.00	4.01	52.1	6.0	4.006
1	UNKNOWN	4.45	4.67	5.05	0.00	95.99	1160.9	143.0	95.994
Total						100.00	1213.0	148.9	100.000



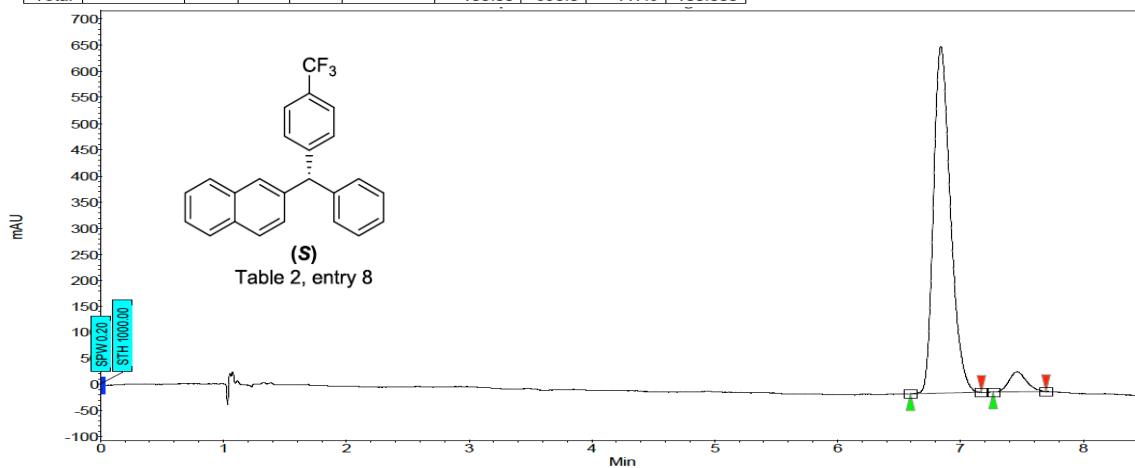
Index	Name	Start [Min]	Time [Min]	End [Min]	RT Offset [Min]	Quantity [% Area]	Height [μV]	Area [μV.Min]	Area [%]
1	UNKNOWN	8.71	8.96	9.26	0.00	4.97	10.6	2.4	4.971
2	UNKNOWN	9.39	9.71	10.35	0.00	95.03	185.0	45.1	95.029
Total						100.00	195.6	47.5	100.000



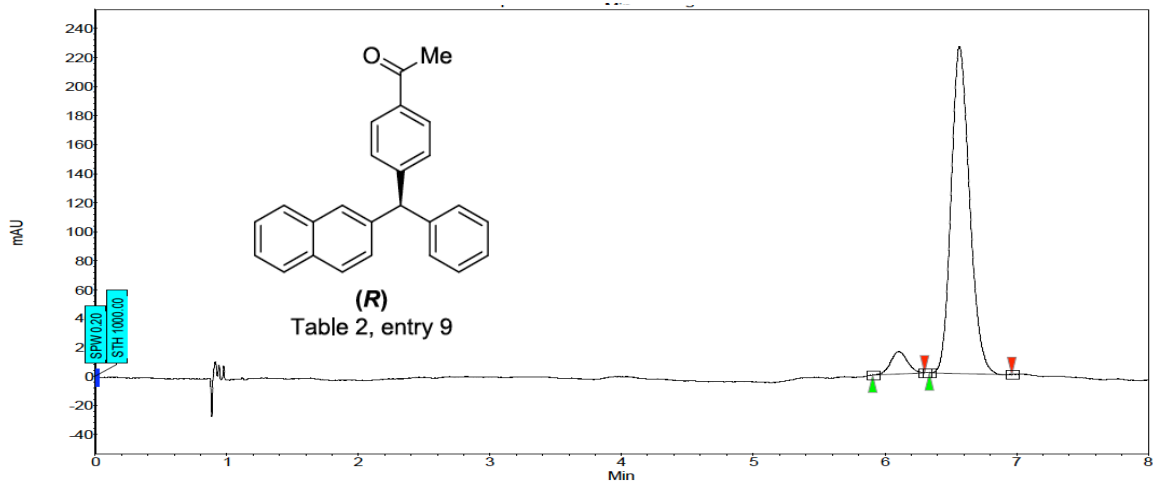
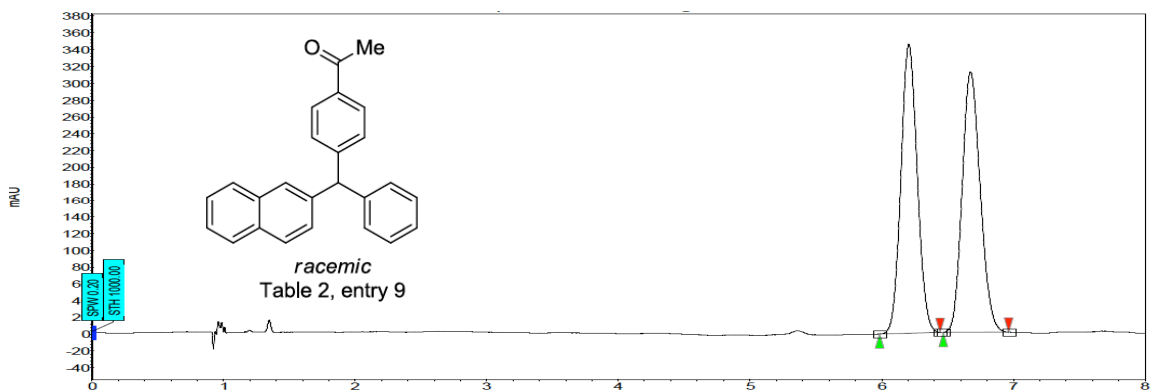
Index	Name	Start [Min]	Time [Min]	End [Min]	RT Offset [Min]	Quantity [% Area]	Height [μV]	Area [μV.Min]	Area [%]
1	UNKNOWN	9.72	10.06	10.55	0.00	94.00	359.5	82.9	94.005
2	UNKNOWN	10.63	10.98	11.30	0.00	6.00	19.5	5.3	5.995
Total						100.00	379.0	88.2	100.000



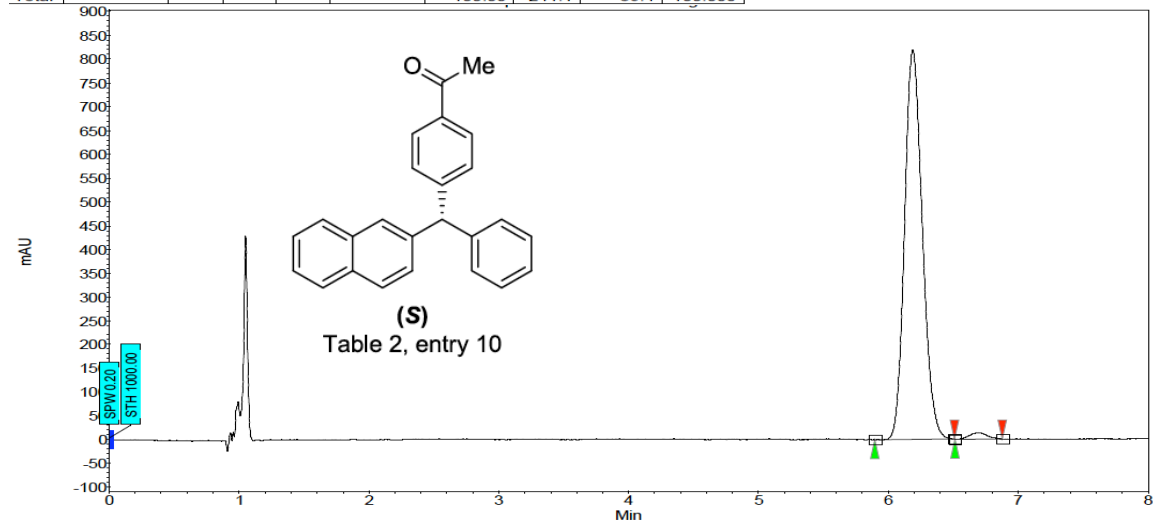
Index	Name	Start Time	End	RT Offset	Quantity	Height	Area	Area
		[Min]	[Min]	[Min]	[Min]	[% Area]	[μ V]	[μ V Min]
1	UNKNOWN	7.04	7.35	7.67	0.00	21.30	157.7	25.0 21.299
2	UNKNOWN	7.70	7.94	8.35	0.00	78.70	540.3	92.5 78.701
Total						100.00	698.0	117.6 100.000



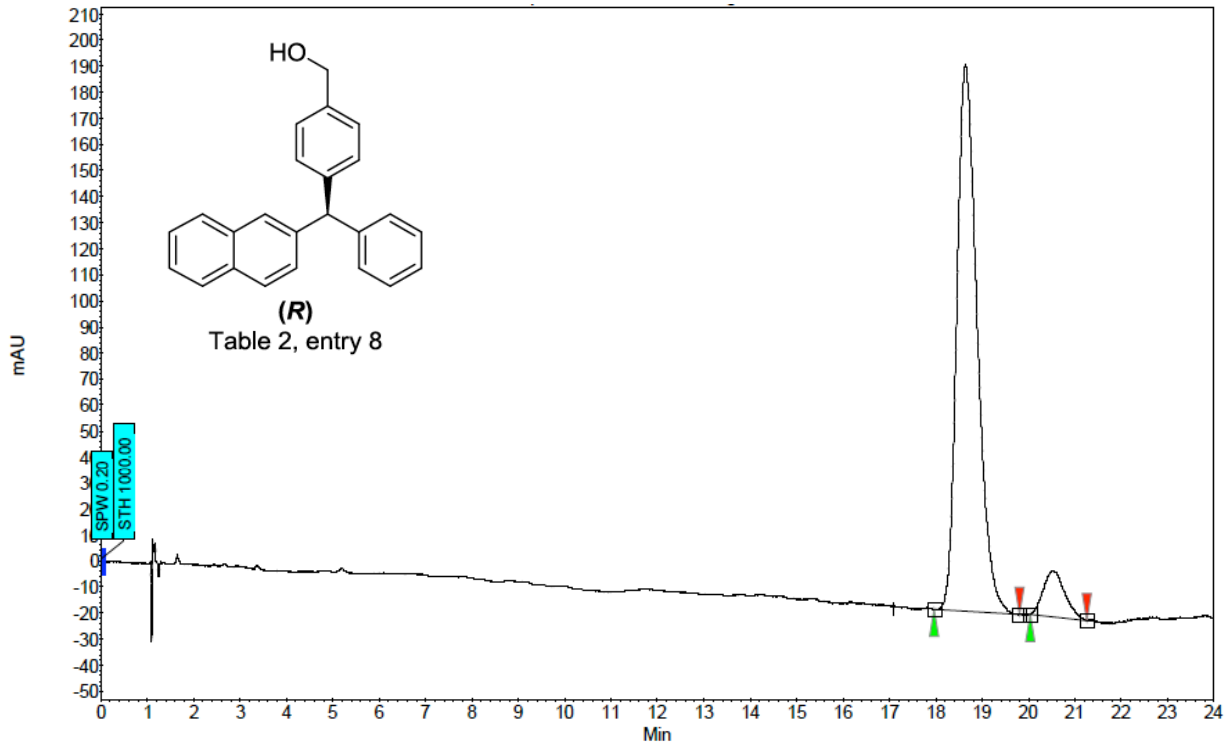
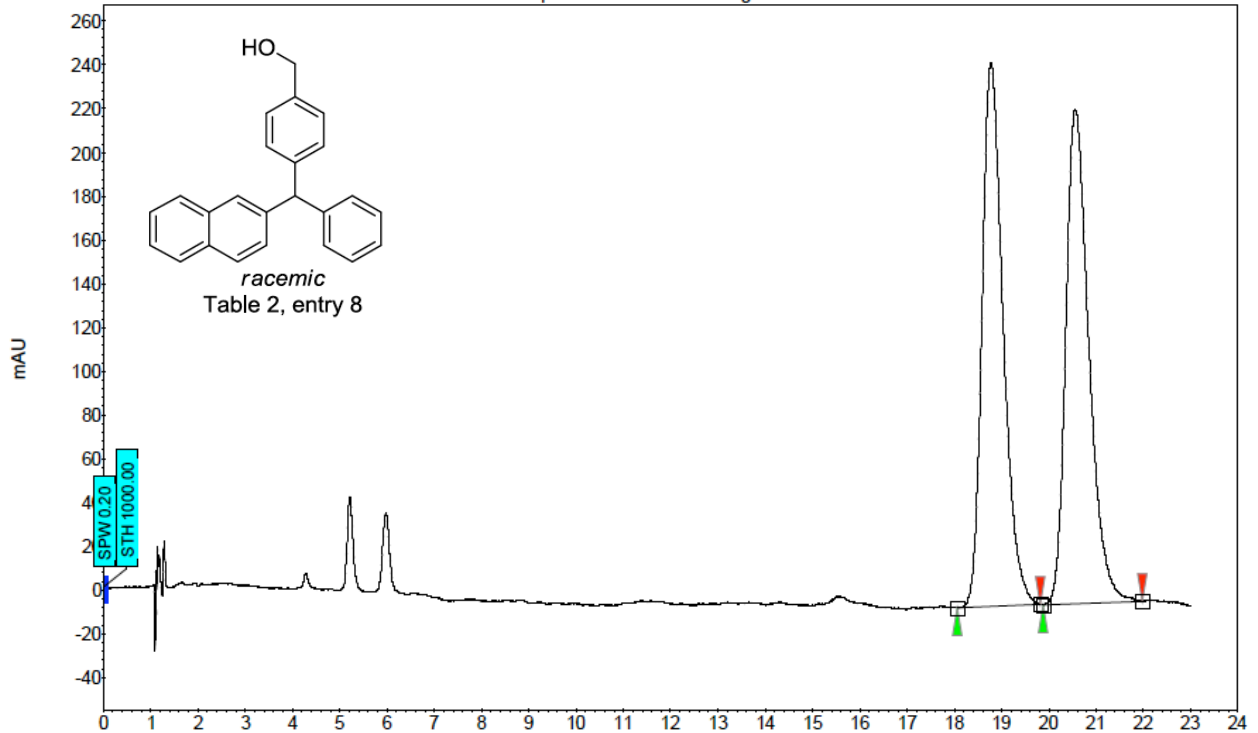
Index	Name	Start Time	End	RT Offset	Quantity	Height	Area	Area
		[Min]	[Min]	[Min]	[Min]	[% Area]	[μ V]	[μ V Min]
1	UNKNOWN	6.59	6.84	7.17	0.00	94.37	663.8	106.4 94.372
2	UNKNOWN	7.27	7.46	7.70	0.00	5.63	38.2	6.3 5.628
Total						100.00	702.1	112.7 100.000



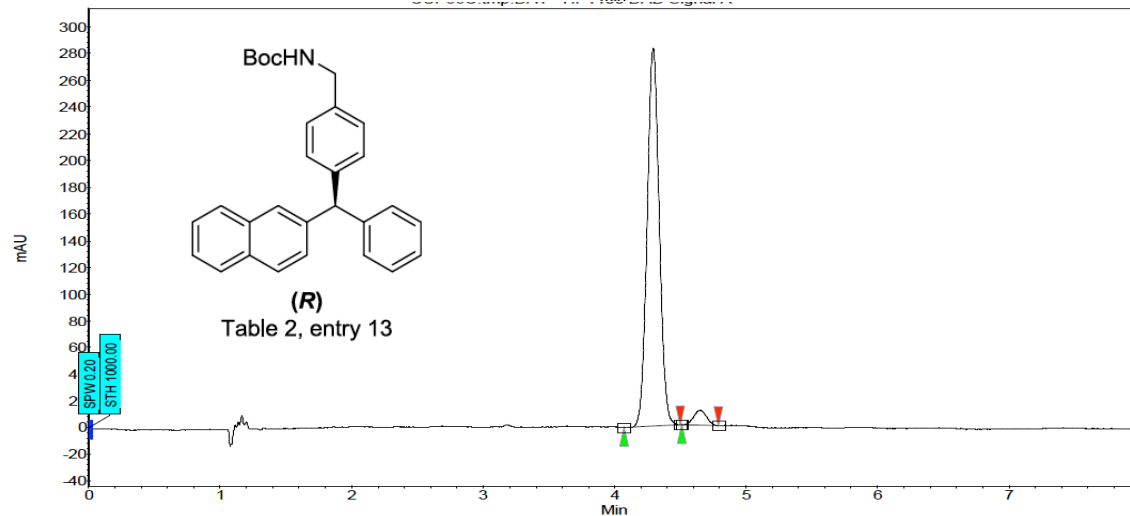
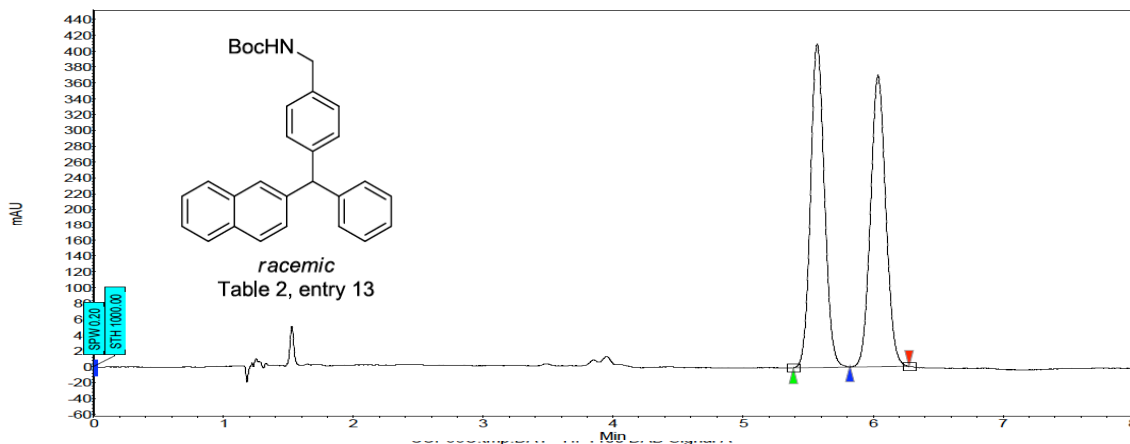
Index	Name	Start	Time	End	RT Offset	Quantity	Height	Area	Area
		[Min]	[Min]	[Min]	[Min]	[% Area]	[μ V]	[μ V.Min]	[%]
2	UNKNOWN	5.91	6.11	6.30	0.00	5.73	15.4	2.3	5.725
1	UNKNOWN	6.34	6.57	6.96	0.00	94.27	225.7	37.1	94.275
Total						100.00	241.1	39.4	100.000



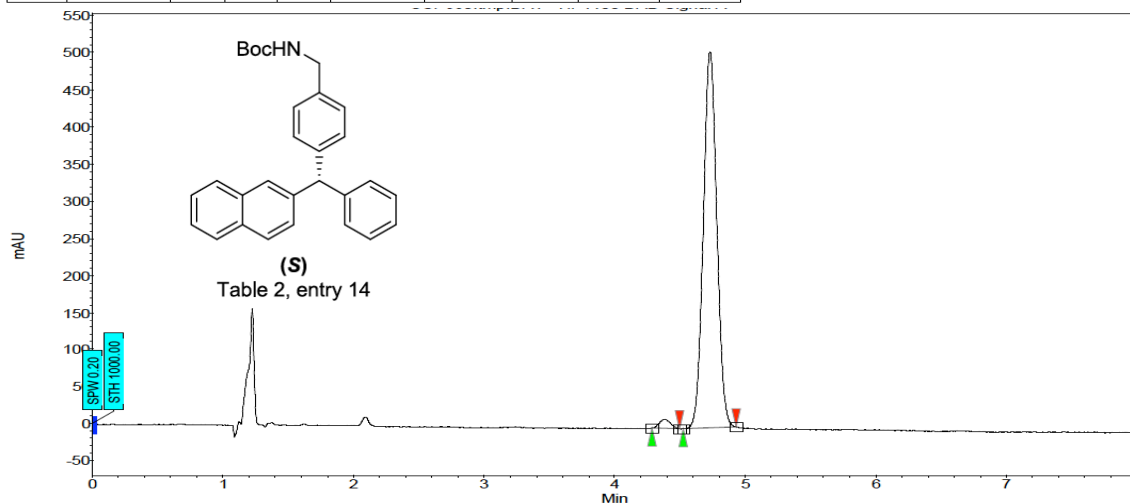
Index	Name	Start	Time	End	RT Offset	Quantity	Height	Area	Area
		[Min]	[Min]	[Min]	[Min]	[% Area]	[μ V]	[μ V.Min]	[%]
1	UNKNOWN	5.90	6.19	6.51	0.00	98.37	818.9	129.1	98.371
2	UNKNOWN	6.51	6.69	6.88	0.00	1.63	13.7	2.1	1.629
Total						100.00	832.6	131.2	100.000



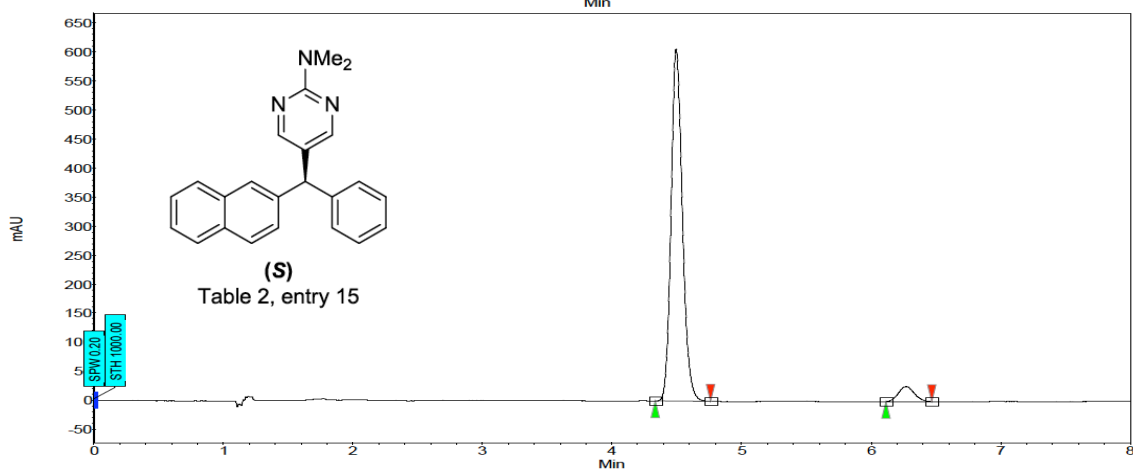
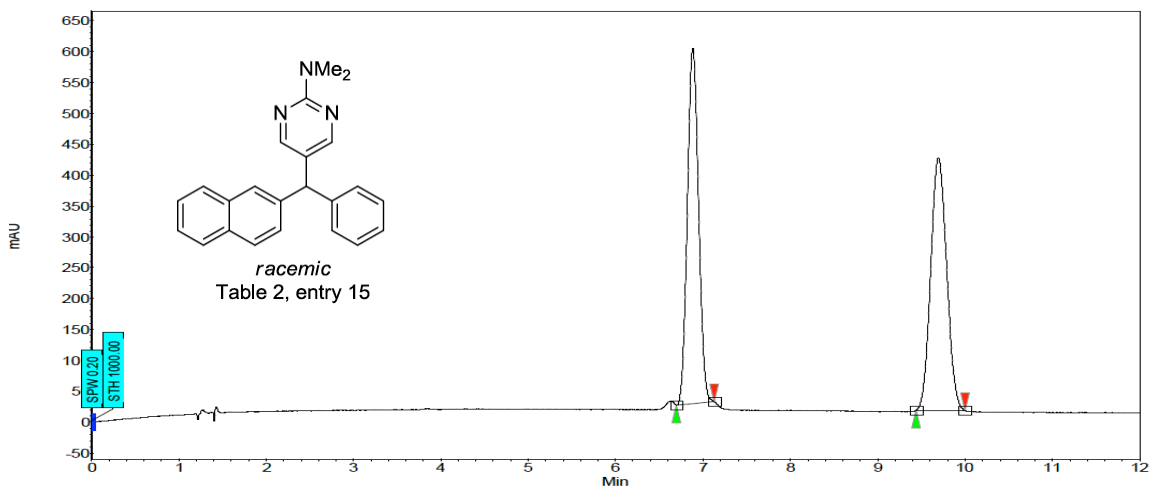
Index	Name	Start	Time	End	RT Offset	Quantity	Height	Area	Area
		[Min]	[Min]	[Min]	[Min]	[% Area]	[μ V]	[μ V.Min]	[%]
1	UNKNOWN	17.97	18.64	19.81	0.00	91.88	209.7	106.3	91.876
2	UNKNOWN	20.04	20.53	21.26	0.00	8.12	17.7	9.4	8.124
Total						100.00	227.3	115.7	100.000



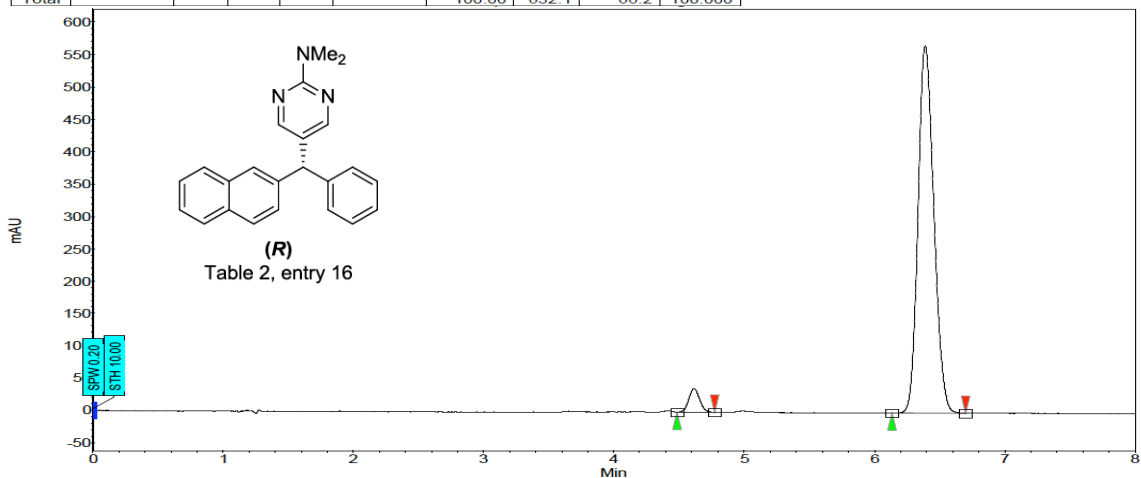
Index	Name	Start [Min]	Time [Min]	End [Min]	RT Offset [Min]	Quantity [% Area]	Height [μV]	Area [μV.Min]	Area [%]
1	UNKNOWN	4.07	4.29	4.50	0.00	96.08	283.0	30.0	96.085
2	UNKNOWN	4.51	4.66	4.79	0.00	3.92	11.0	1.2	3.915
Total						100.00	294.0	31.2	100.000



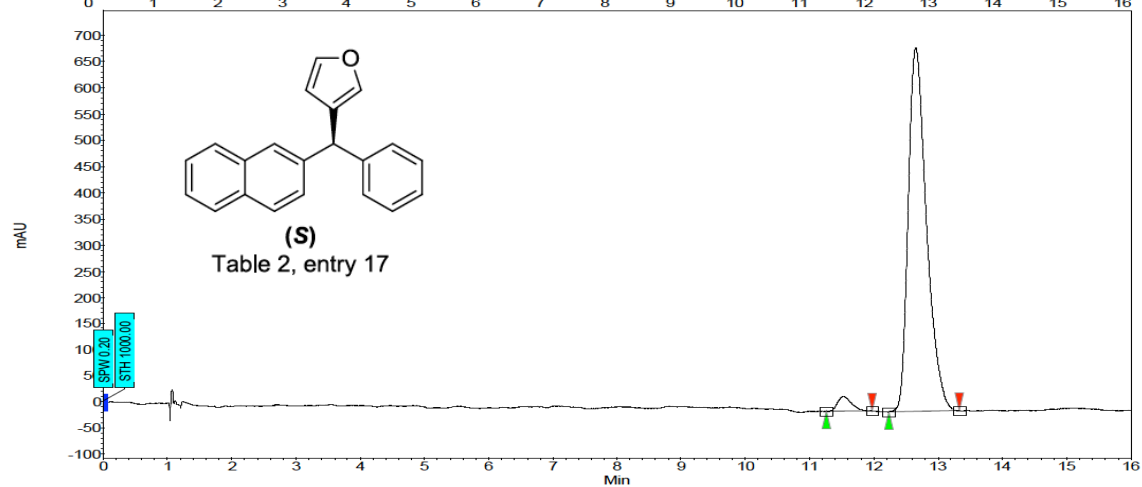
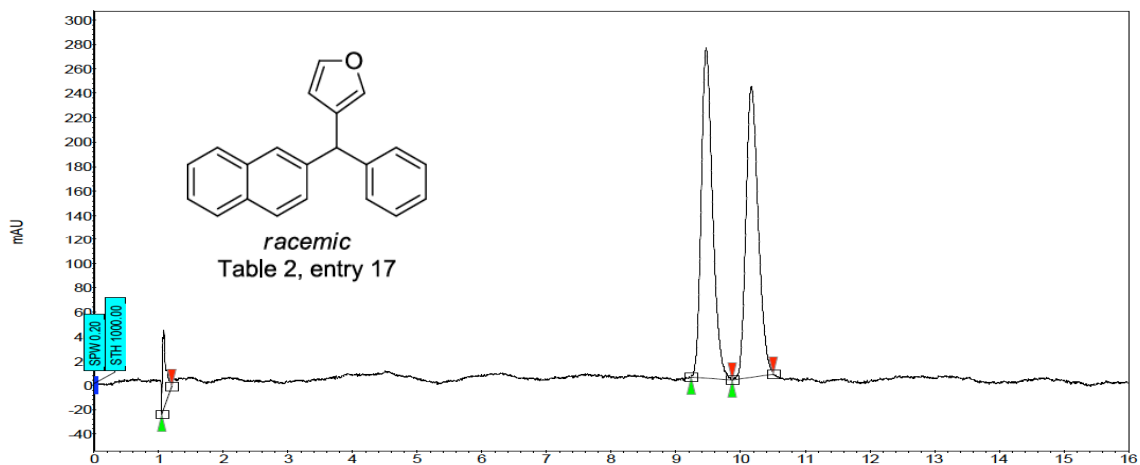
Index	Name	Start [Min]	Time [Min]	End [Min]	RT Offset [Min]	Quantity [% Area]	Height [μV]	Area [μV.Min]	Area [%]
1	UNKNOWN	4.29	4.39	4.50	0.00	1.92	11.9	1.2	1.924
2	UNKNOWN	4.53	4.73	4.93	0.00	98.08	506.5	60.4	98.076
Total						100.00	518.5	61.6	100.000



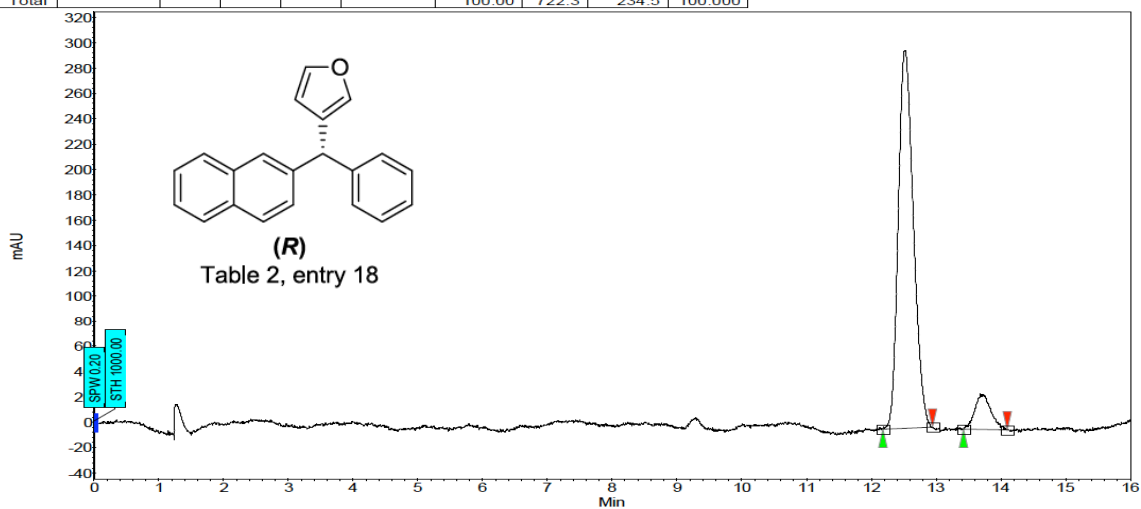
Index	Name	Start Time [Min]	End [Min]	RT Offset [Min]	Quantity [% Area]	Height [μV]	Area [μV.Min]	Area [%]	
1	UNKNOWN	4.33	4.50	4.76	0.00	94.50	606.4	94.501	
2	UNKNOWN	6.12	6.27	6.47	0.00	5.50	25.7	3.6	
Total						100.00	632.1	66.2	100.000



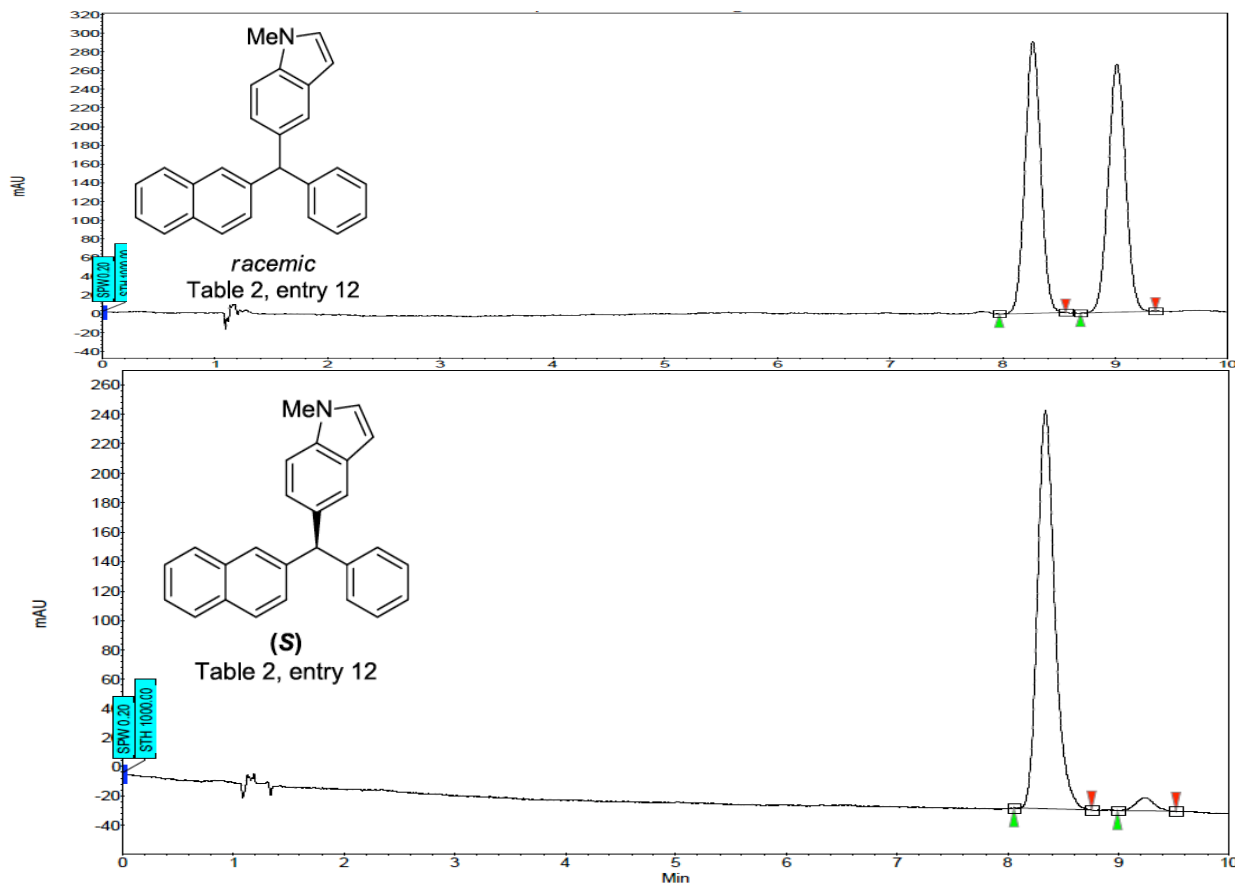
Index	Name	Start Time [Min]	End [Min]	RT Offset [Min]	Quantity [% Area]	Height [μV]	Area [μV.Min]	Area [%]	
1	UNKNOWN	4.48	4.62	4.77	0.00	4.20	3.5	4.201	
2	UNKNOWN	6.13	6.39	6.70	0.00	95.80	567.9	79.6	
Total						100.00	604.4	83.1	100.000



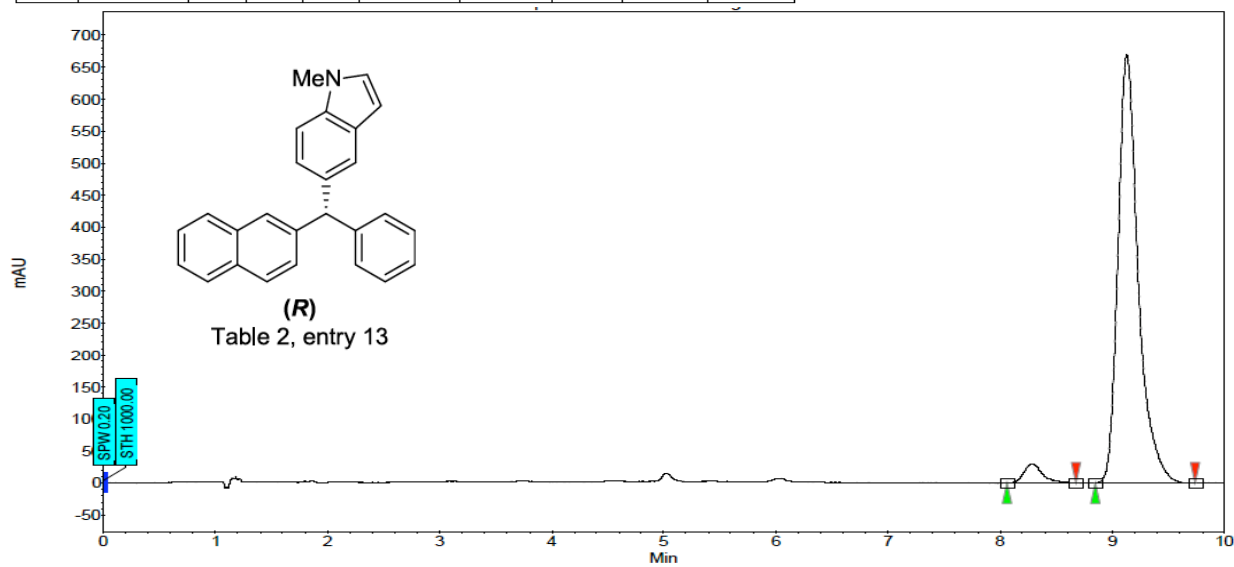
Index	Name	Start [Min]	Time [Min]	End [Min]	RT Offset [Min]	Quantity [% Area]	Height [μV]	Area [μV.Min]	Area [%]
2	UNKNOWN	11.26	11.53	11.97	0.00	2.94	28.1	6.9	2.937
1	UNKNOWN	12.23	12.65	13.32	0.00	97.06	694.1	227.6	97.063
Total						100.00	722.3	234.5	100.000



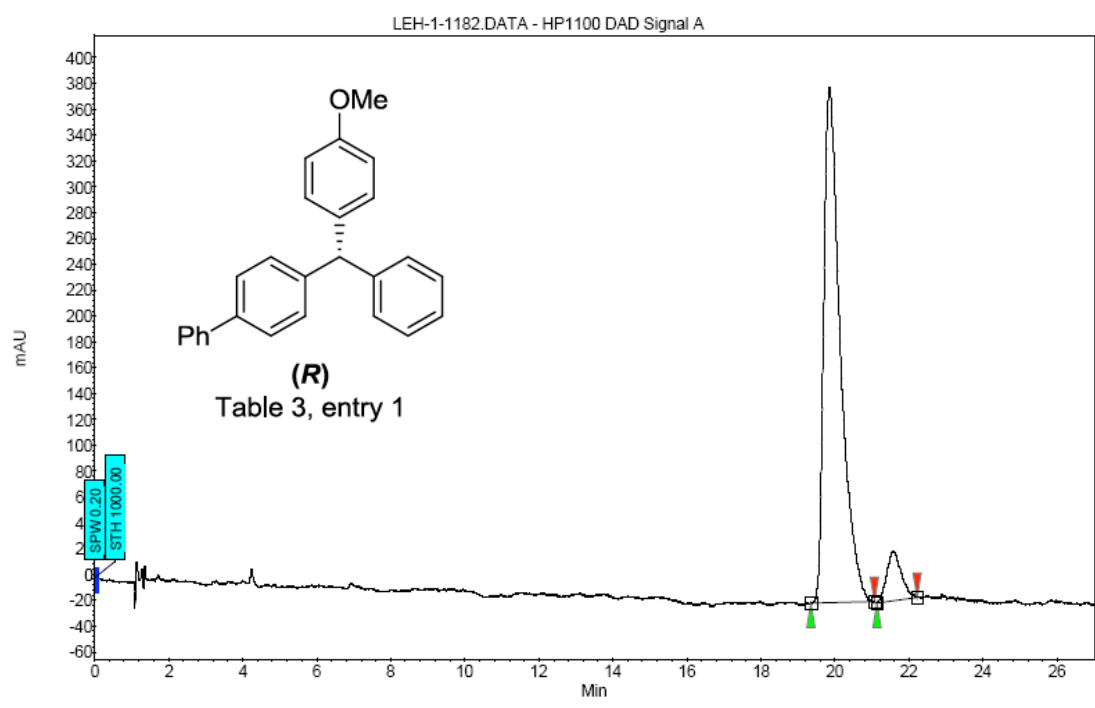
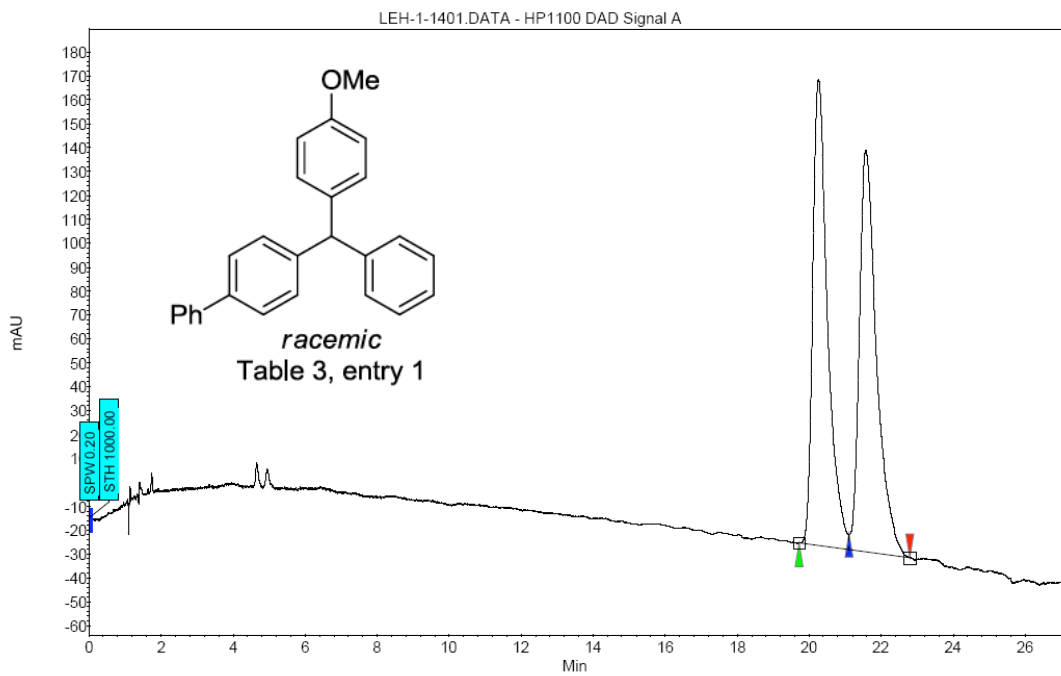
Index	Name	Start [Min]	Time [Min]	End [Min]	RT Offset [Min]	Quantity [% Area]	Height [μV]	Area [μV.Min]	Area [%]
1	UNKNOWN	12.18	12.52	12.95	0.00	90.99	298.5	80.3	90.991
2	UNKNOWN	13.42	13.69	14.10	0.00	9.01	27.8	7.9	9.009
Total						100.00	326.3	88.2	100.000



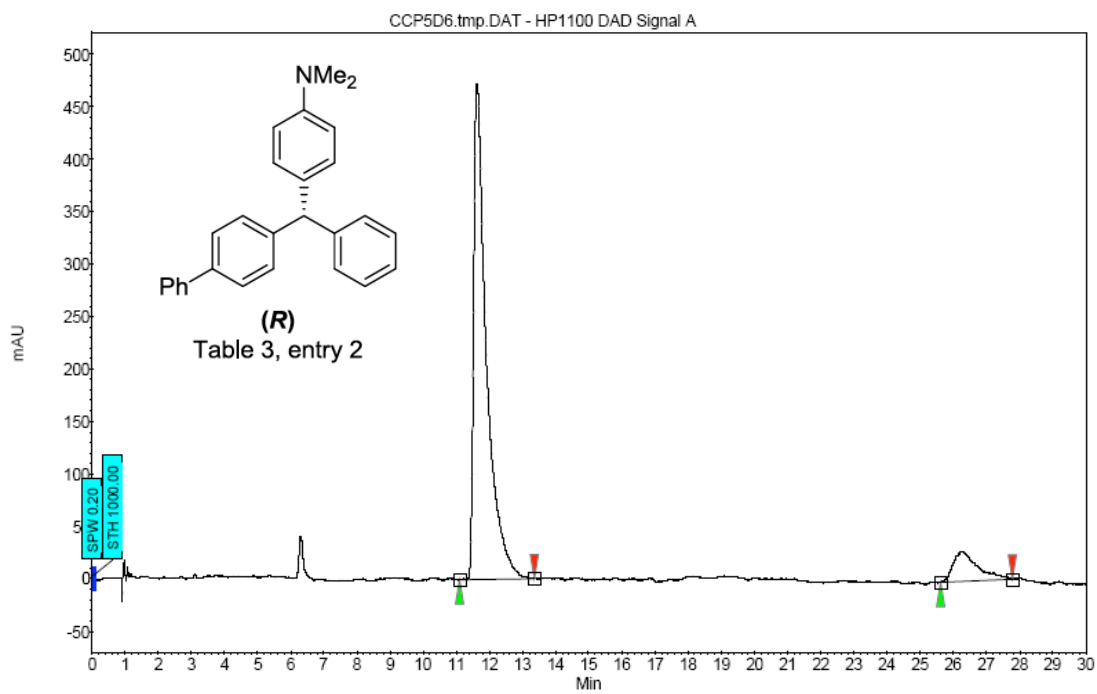
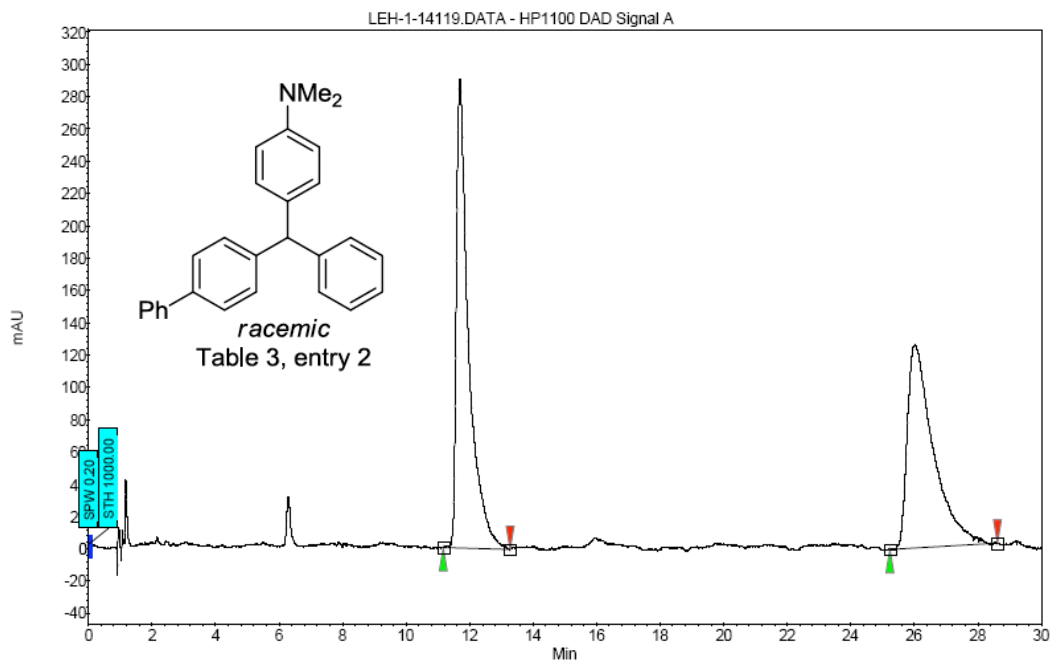
Index	Name	Start [Min]	Time [Min]	End [Min]	RT Offset [Min]	Quantity [% Area]	Height [μV]	Area [μV.Min]	Area [%]
1	UNKNOWN	8.05	8.34	8.76	0.00	96.61	270.9	49.0	96.606
2	UNKNOWN	8.99	9.24	9.52	0.00	3.39	8.9	1.7	3.394
Total						100.00	279.8	50.7	100.000



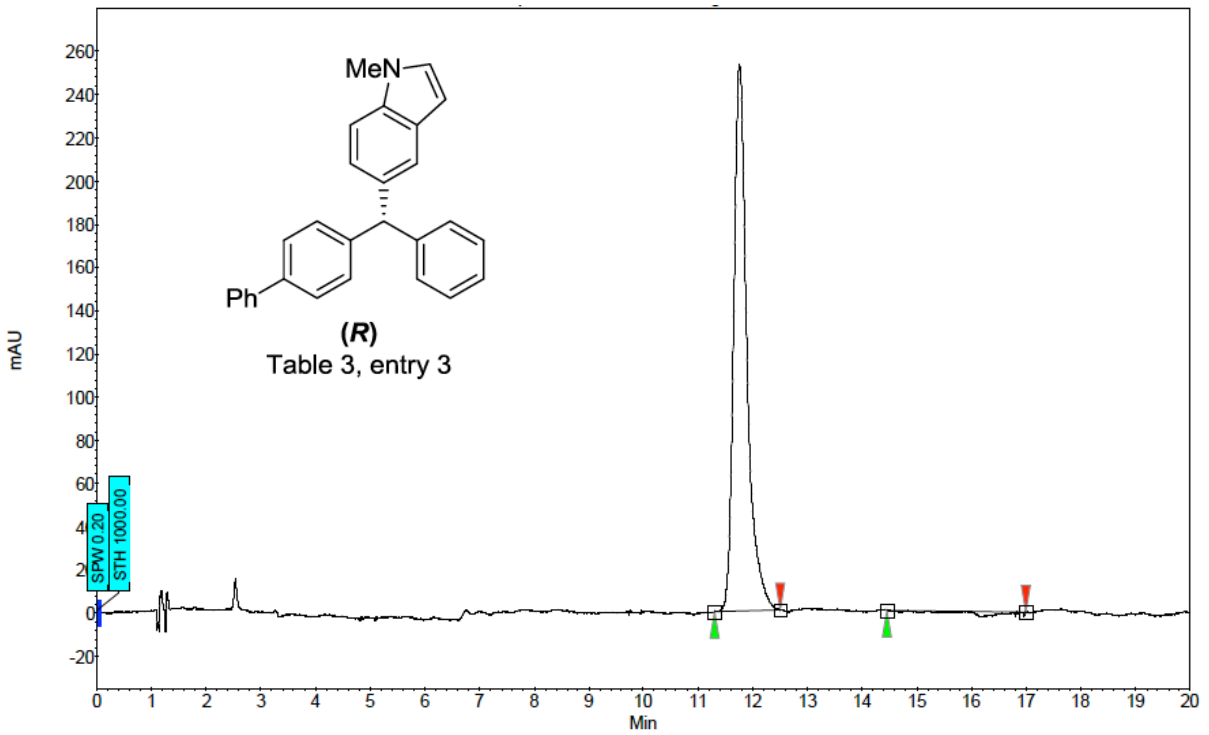
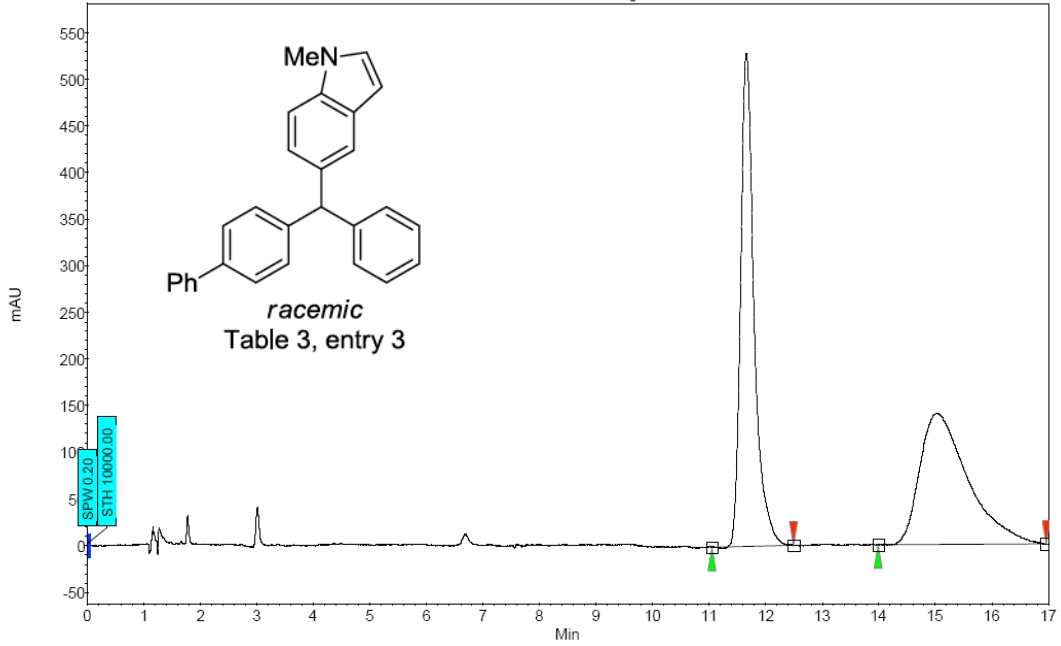
Index	Name	Start [Min]	Time [Min]	End [Min]	RT Offset [Min]	Quantity [% Area]	Height [μV]	Area [μV.Min]	Area [%]
1	UNKNOWN	8.06	8.28	8.67	0.00	3.91	29.8	5.7	3.910
2	UNKNOWN	8.85	9.13	9.73	0.00	96.09	668.3	139.6	96.090
Total						100.00	698.2	145.3	100.000



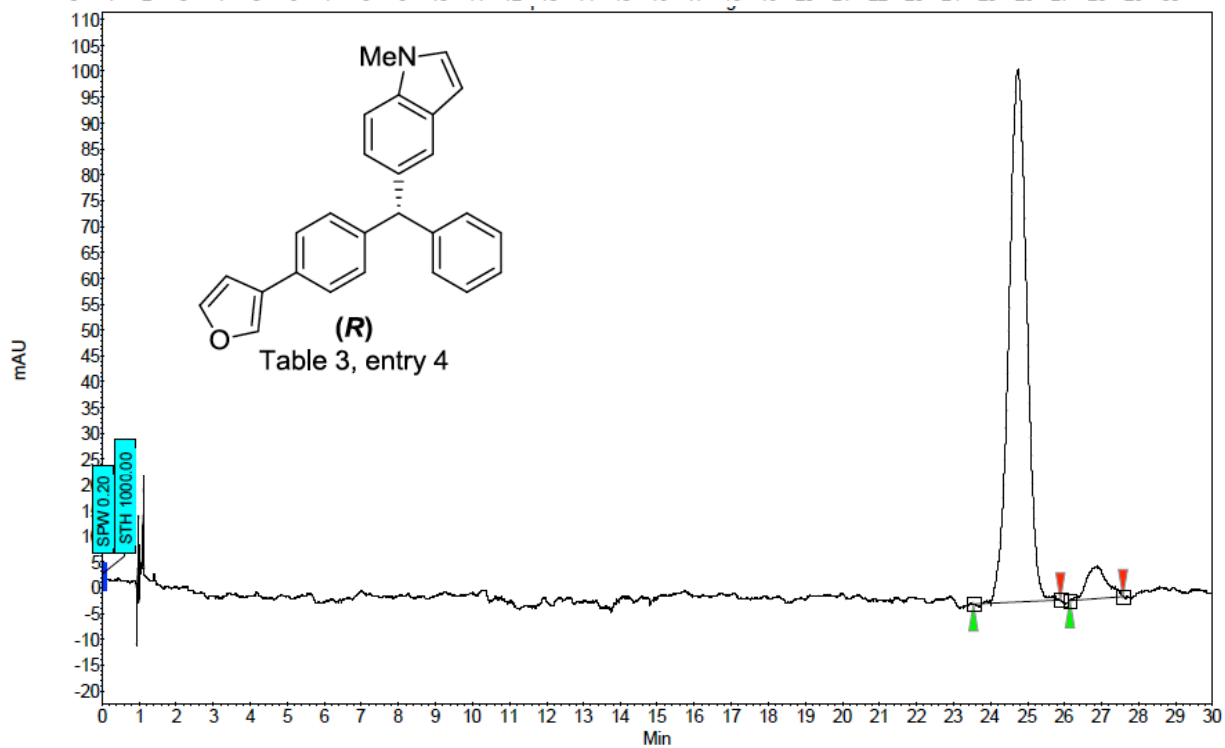
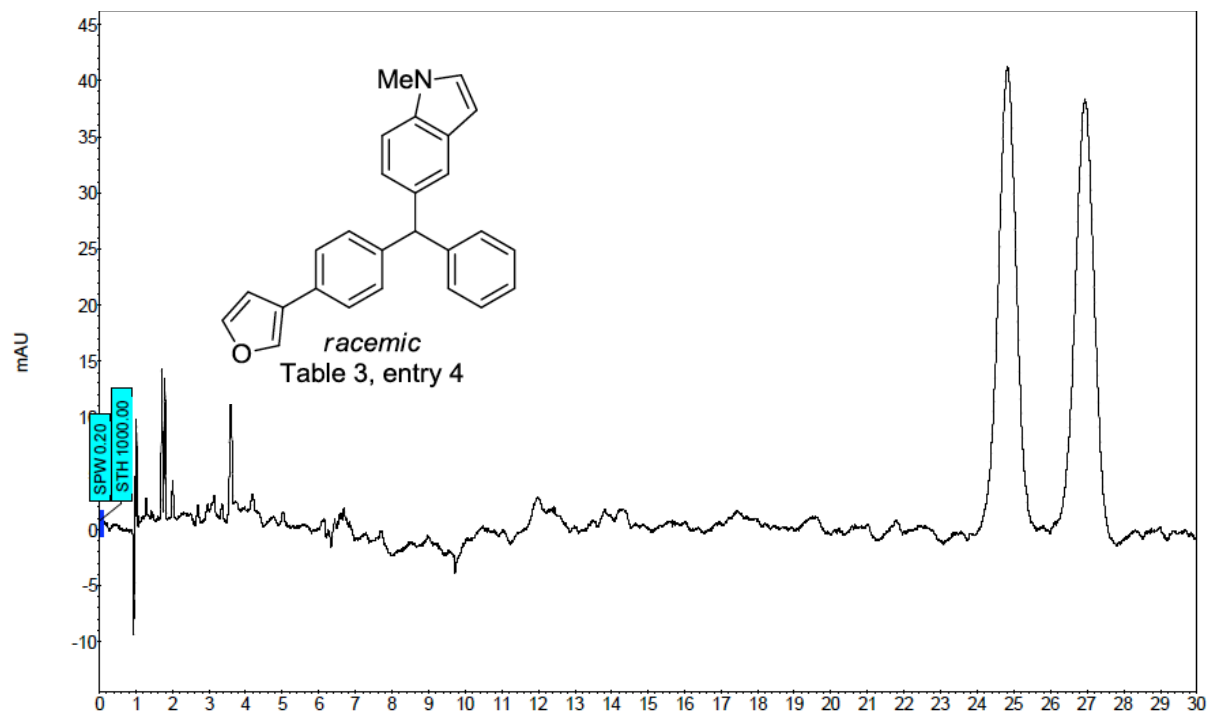
Index	Name	Start [Min]	Time [Min]	End [Min]	RT Offset [Min]	Quantity [% Area]	Height [µV]	Area [µV.Min]	Area [%]
1	UNKNOWN	19.35	19.85	21.07	0.00	92.25	398.9	207.1	92.249
2	UNKNOWN	21.13	21.57	22.22	0.00	7.75	38.6	17.4	7.751
Total						100.00	437.5	224.5	100.000



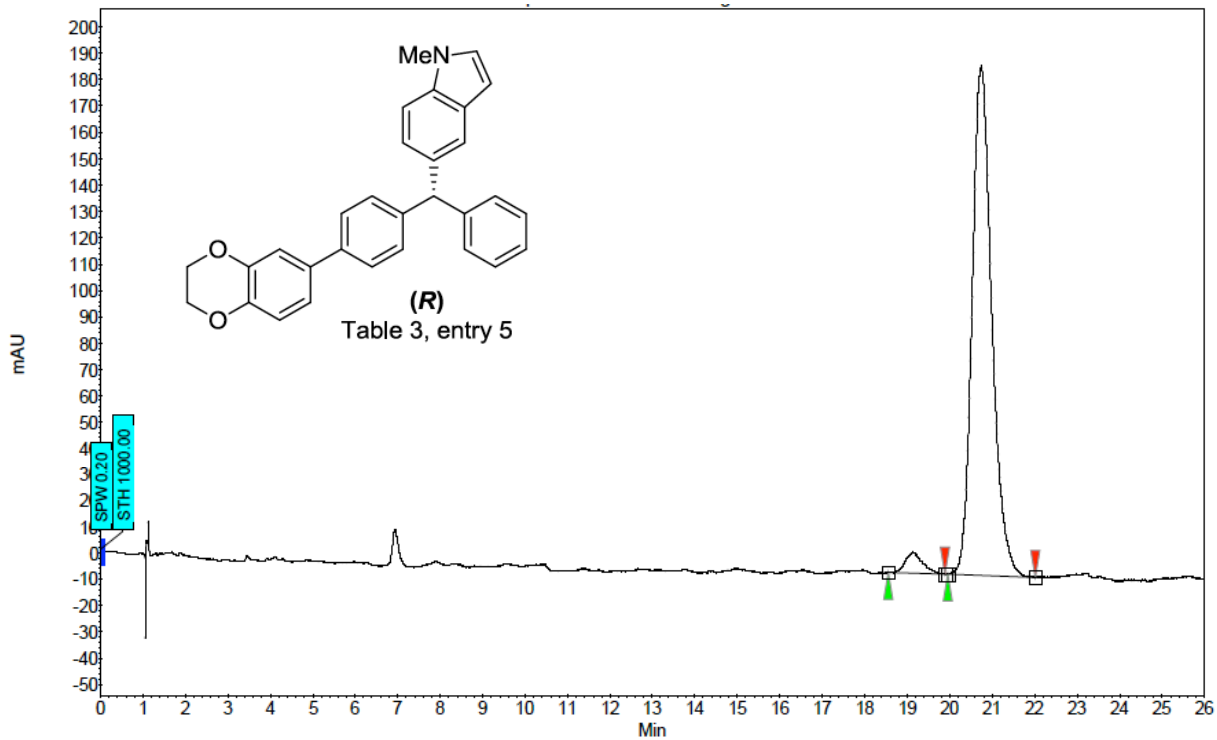
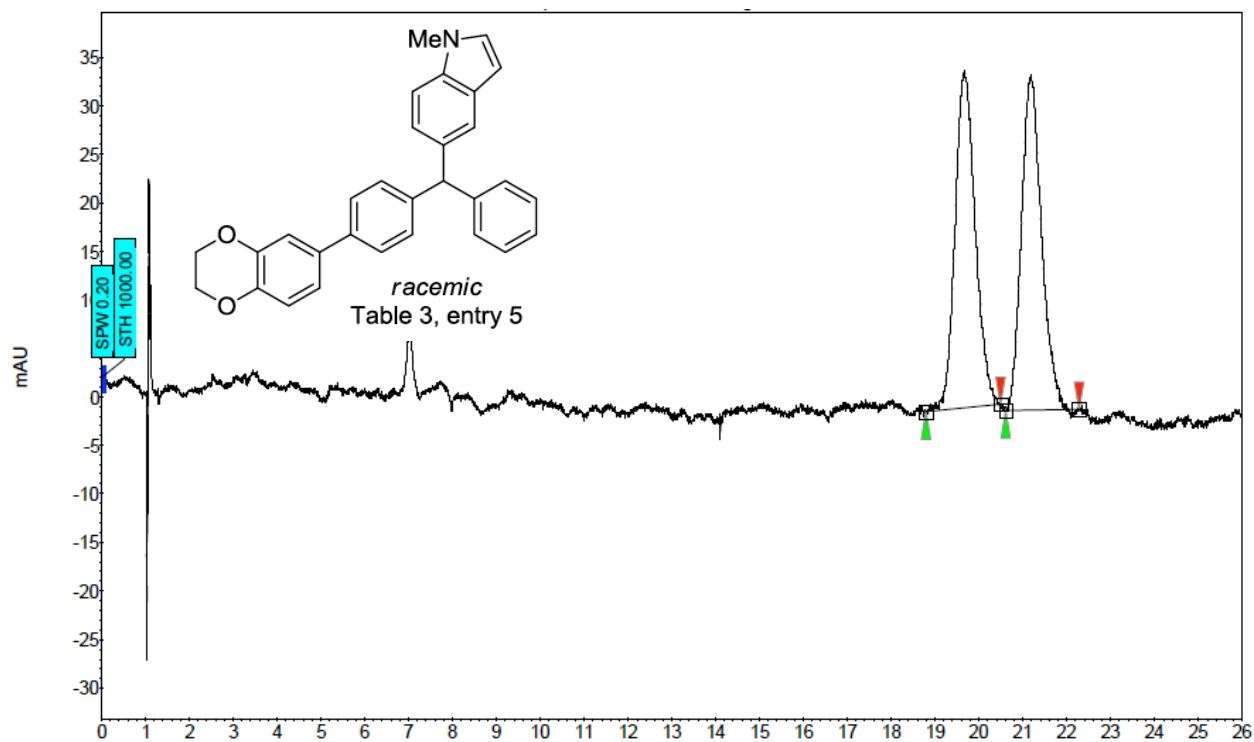
Index	Name	Start	Time	End	RT Offset	Quantity	Height	Area	Area
		[Min]	[Min]	[Min]	[Min]	[% Area]	[μ V]	[μ V.Min]	[%]
1	UNKNOWN	11.09	11.62	13.35	0.00	89.58	472.3	210.6	89.580
2	UNKNOWN	25.61	26.26	27.77	0.00	10.42	28.2	24.5	10.420
Total						100.00	500.5	235.1	100.000



Index	Name	Start [Min]	Time [Min]	End [Min]	RT Offset [Min]	Quantity [% Area]	Height [μV]	Area [μV.Min]	Area [%]
1	UNKNOWN	11.31	11.76	12.51	0.00	97.87	252.3	70.7	97.873
2	UNKNOWN	14.45	16.15	17.00	0.00	2.13	2.3	1.5	2.127
Total						100.00	254.6	72.2	100.000



Index	Name	Start	Time	End	RT Offset	Quantity	Height	Area	Area
		[Min]	[Min]	[Min]	[Min]	[% Area]	[μ V]	[μ V.Min]	[%]
1	UNKNOWN	23.55	24.74	25.88	0.00	93.55	103.0	57.7	93.545
2	UNKNOWN	26.15	26.89	27.58	0.00	6.45	6.2	4.0	6.455
Total						100.00	109.2	61.7	100.000



Index	Name	Start Time [Min]	End Time [Min]	RT Offset [Min]	Quantity [% Area]	Height [μV]	Area [μV.Min]	Area [%]
1	UNKNOWN	18.56	19.13	19.88	0.00	3.46	7.8	3.464
2	UNKNOWN	19.96	20.74	22.01	0.00	96.54	193.6	96.536
Total					100.00	201.4	107.1	100.000