SUPPORTING INFORMATION

SILVER-CATALYZED ENANTIOSELECTIVE PROPARGYLATION REACTIONS OF *N*-SULFONYL KETIMINES

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I. GENERAL PROCEDURES

NMR spectra were recorded on Bruker DRX-400 (400 MHz ^{1}H, 100 MHz ^{13}C, 376.5 MHz ^{19}F), GN-500 (500 MHz ^{1}H, 125.7 MHz ^{13}C, 160.2 MHz ^{11}B), or CRYO-500 (500 MHz ^{1}H, 125.7 MHz ^{13}C) spectrometers. Proton chemical shifts are reported in ppm (δ) relative to internal trimethylsilane (TMS, δ 0.00). Data are reported as follows: chemical shift (multiplicity [singlet (s), broad singlet (br s), doublet (d), apparent doublet (ad), doublet of doublets (dd), doublet of doublets of doublets (ddd), triplet (t), apparent triplet (at), doublet of triplets (dt), triplet of doublets (td), quartet (q), quintet (quint), apparent quintet of doublets (aquintd), sextet (sext), multiplet (m)], coupling constants [Hz], integration). Carbon chemical shifts are reported in ppm (δ) relative to TMS with the solvent resonance as the internal standard ($CDCl_3$, δ 77.16 ppm or DMF-d_7, δ 163.15 ppm). NMR data were collected at 25 °C. Infrared spectra were obtained on a Thermo Scientific Nicolet iS5 spectrometer with an iD5 ATR tip (neat) and are reported in terms of frequency of absorption (cm^{-1}). Analytical thin-layer chromatography (TLC) was performed using Silica Gel 60Å F254 precoated plates (0.25 mm thickness). Visualization was accomplished by irradiation with a UV lamp and/or staining with *p*-anisaldehyde (PAA) or potassium permanganate ($KMnO_4$) 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 with a Rudolph Research Analytical Autopol III Automatic Polarimeter. SFC determinations of enantiopurity were performed on a Berger Analytical instrument using a Daicel™ Chiralpak® column (OD-H, AD-H, AS-H, or (*R*,*R*)-Whelk-O); 100 bar, 215 nm, 50 °C). High resolution mass spectrometry was performed by the University of California, Irvine Mass Spectrometry Center.

All reactions were carried out under a N_2 atmosphere, unless otherwise stated. All glassware was either oven-dried or flame-dried prior to use. *N*,*N*-Dimethylformamide (DMF), tetrahydrofuran (THF), diethyl ether (Et_2O), dichloromethane (CH_2Cl_2), triethylamine (TEA), methanol (MeOH), and *N*,*N*-dimethylacetamide (DMA) were degassed with argon 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 hours) to remove H_2O. Other solvents were purchased "anhydrous" commercially, or were purified as described.

$AgPF_6$ was purchased as a white powder from Strem, stored in the dark in a glove box under an atmosphere of N_2, and discarded upon turning to a brown powder.

(*R*,*R*)-Walphos W001-1 was purchased from Strem or Acros, stored in a glove box under an atmosphere of N_2, and used as received. All other ligands were purchased from Strem or Sigma Aldrich and were stored under N_2 atmosphere and used as received.

Saccharin was purchased from Sigma Aldrich and used as received. All Grignard reagents were titrated with iodine prior to use.[1] *n*-Butyllithium and methyllithium solutions were purchased from Acros, stored at 4 °C, and titrated prior to use.[2]

tert-Butanol was purchased from Fisher and distilled every two weeks over CaH_2 through a short-path distillation head onto activated 4Å mol sieves.

Allenylboronic acid pinacol ester **2** was prepared according to Yoshida and co-workers[3] and distilled every month.

Propargylboronic acid pinacol ester **12** was prepared according to Fandrick and co-workers (vide infra, Section II-G-1).[4] Ethynylmagnesium bromide was purchased from Sigma Aldrich, stored at 4 °C, and used within one week of opening the bottle.

N,*N*-Dimethylformamide-d_7 was purchased from Cambridge Isotope Laboratories and used as received.

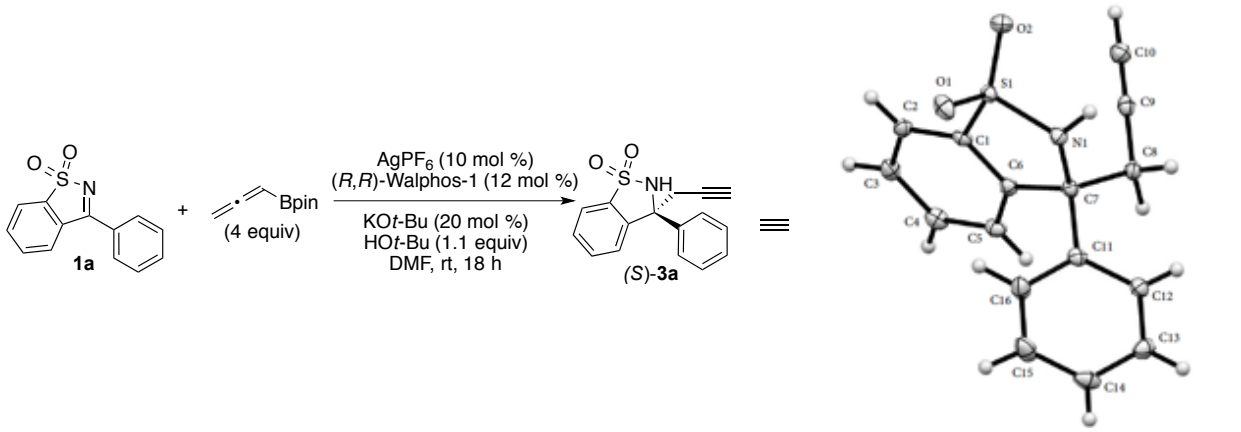
All other chemicals were purchased commercially and used as received.

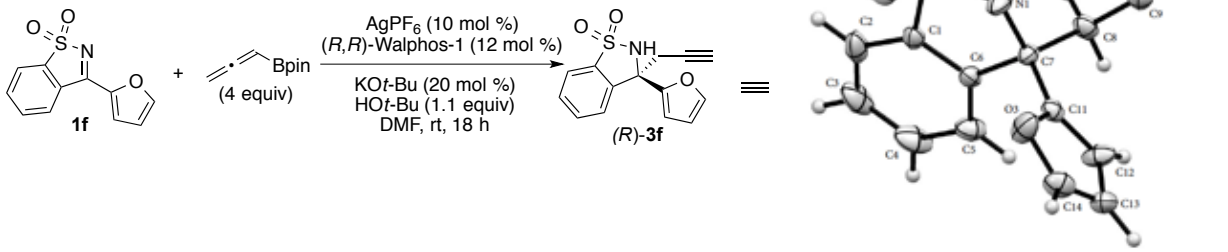
II. EXPERIMENTAL

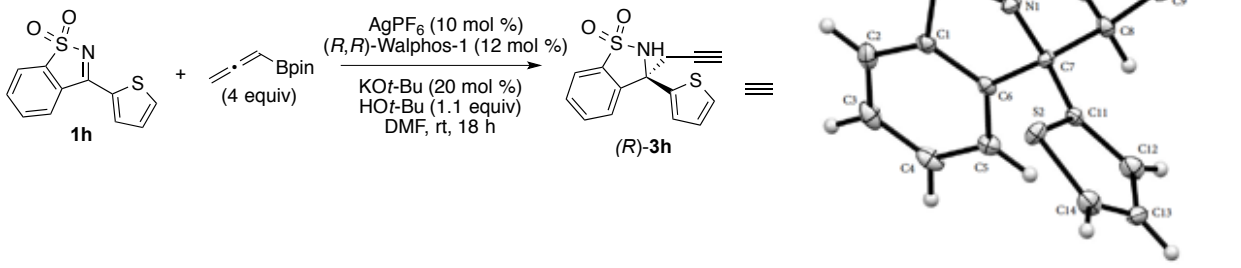
A. STEREOCHEMICAL PROOFS

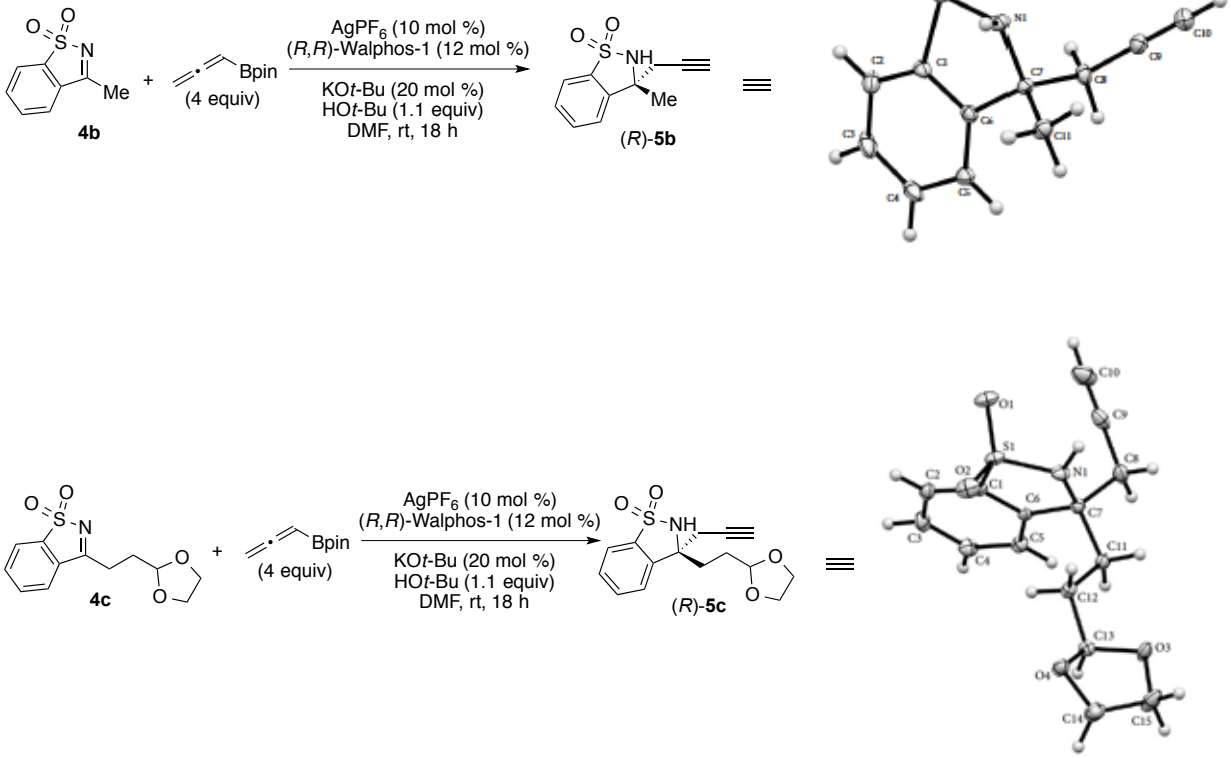
The absolute configurations of products **3a**, **3f**, **3h**, **5b**, and **5c** were assigned by X-ray crystallographic analysis (Scheme SI-1). The absolute configurations of all other products were assigned by analogy. See Section IV for crystallographic data.

Scheme SI-1. Absolute configurations of products determined by X-ray crystallography.





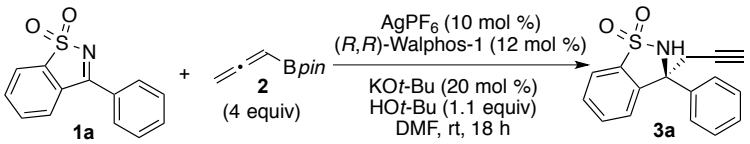



AgPF6 (10 mol %)
(R,R)-Walphos-1 (12 mol %)
KOt-Bu (20 mol %)
HOt-Bu (1.1 equiv)
DMF, rt, 18 h
Bpin
(4 equiv)
4b
(R)-5b
4c
(R)-5c

B. Representative Addition Procedures

Method A: Enantioselective Addition to Ketimines

Note: All manipulations involving silver-catalyzed reactions were performed in the absence of direct light, using vials wrapped in aluminum foil.

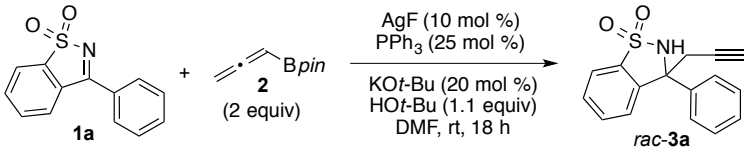


In a glovebox, an oven-dried 1.0 mL conical vial equipped with a triangular stir bar was charged with $AgPF_6$ (5.0 mg, 0.020 mmol, 0.10 equiv) and Walphos W001-1 (22.3 mg, 0.0240 mmol, 0.120 equiv). The vial was sealed with a screw-top cap fit with a septum and removed from the glovebox. Anhydrous DMF (400 μL) was added and the solution was stirred for 5 min at rt. The N_2 line was then removed and the solution was stirred for 30 min at 70 °C, then cooled to rt over 15 min.

To the catalyst solution was added *tert*-butanol (21 μL, 0.22 mmol, 1.1 equiv), followed by potassium *tert*-butoxide (4.5 mg, 0.040 mmol, 0.20 equiv) and phenyl ketimine **1a** (48.6 mg, 0.200 mmol, 1.00 equiv) under a flow of N_2. The reaction was stirred at rt for 5 min to dissolve the ketimine. Allenylboronic acid pinacol ester **2** (72 μL, 0.40 mmol, 2.0 equiv) was added via syringe, followed by another portion of allenylboronic acid pincol ester (72 μL, 0.40 mmol, 2.0 equiv) added via slow addition over 3 h using a syringe pump. The N_2 line was removed and the reaction was stirred at 22 °C for 18 h. The reaction mixture was filtered through a plug of silica gel eluting with 100% Et_2O to remove the catalyst. Et_2O was removed in vacuo and the resulting residue was purified by silica gel chromatography.

Method B: Racemic Standards

Note: All manipulations involving silver-catalyzed reactions were performed in the absence of direct light, using vials wrapped in aluminum foil.

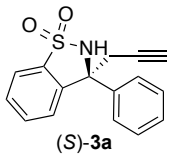


In a glovebox, a flame-dried 7 mL vial equipped with a stir bar was charged with AgF (2.5 mg, 0.020 mmol, 0.10 equiv) and PPh_3 (13 mg, 0.050 mmol, 0.25 equiv). The vial was sealed with a screw-top cap fit with a septum and removed from the glovebox. Anhydrous DMF (800 μL) was added and the solution was stirred for 5 min at rt. The N_2 line was then removed and the solution was stirred for 30 min at 70 °C, then cooled to rt over 15 min.

To the catalyst solution was added *tert*-butanol (21 μL, 0.22 mmol, 1.1 equiv), followed by potassium *tert*-butoxide (4.5 mg, 0.040 mmol, 0.20 equiv) and phenyl ketimine **1a** (48.6 mg, 0.200 mmol, 1.00 equiv) under a flow of N_2. The reaction was stirred at rt for 5 min to dissolve the ketimine. Allenylboronic acid pinacol ester **2** (72 μL, 0.40 mmol, 2.0 equiv) was added via syringe. The N_2 line was removed and the reaction was stirred at 22 °C for 18 h. The reaction mixture was filtered through a plug of silica gel eluting with 100% Et_2O to remove the catalyst. Et_2O was removed in vacuo and the resulting residue was purified by silica gel chromatography.

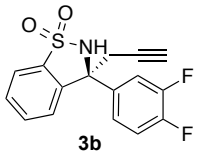
C. CHARACTERIZATION DATA FOR PRODUCTS

Note: The yield of homoallenylic sultam is typically less than 5–10% in these reactions and can be separated from the homopropargylic sultam using the column chromatography conditions specified below. The TLC R_f of the homoallenylic sultam is generally 0.1 higher than the R_f of the homopropargylic sultam. The diagnostic peaks for the homoallenylic sultam are found in the ^{1}H NMR range of δ 5.96 to 5.48 (t, J = 6.6 Hz, 1H) and δ 5.11 to 5.01 (d, J = 6.6 Hz, 2H).



(*S*)-**3a**

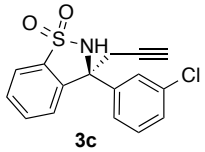
Sultam (*S*)-3a was prepared according to Method A, using the following amounts of reagents: $AgPF_6$ (5.0 mg, 0.020 mmol, 0.10 equiv), Walphos W001-1 (22.3 mg, 0.0240 mmol, 0.120 equiv), *tert*-butanol (21 μL, 0.22 mmol, 1.1 equiv), potassium *tert*-butoxide (4.5 mg, 0.040 mmol, 0.20 equiv), substrate **1a** (48.6 mg, 0.200 mmol, 1.00 equiv), DMF (400 μL), and allenylboronic acid pincol ester (144 μL, 0.800 mmol, 4.00 equiv). The resulting mixture was purified by flash column chromatography using 0–1% TEA/benzene to separate product from unreacted starting material. The mixture was purified again by flash column chromatography using 5–10–20% EtOAc/hexanes (1% TEA) to separate product from excess ligand and afford the title compound as a white solid (43.6 mg, 0.152 mmol, 76%, 99:1 er). **TLC R_f** = 0.2 (20% EtOAc/hexanes, stains pink with PAA); **m.p.** = 139–142 °C; **^{1}H NMR** (500 MHz, $CDCl_3$) δ 7.80 (ad, J = 7.3 Hz, 1H), 7.62–7.53 (m, 4H), 7.41–7.30 (m, 4H), 5.23 (br s, 1H), 3.30 (dd, J = 17.2, 2.6 Hz, 1H), 3.24 (dd, J = 17.2, 2.6 Hz, 1H), 2.06 (t, J = 2.6 Hz, 1H); **^{13}C NMR** (125 MHz, $CDCl_3$) δ 142.3, 140.4, 135.0, 133.5, 130.0, 129.1, 128.7, 126.6, 125.0, 121.5, 78.5, 73.3, 67.1, 31.3; **IR** (neat) 3286, 2923, 1713, 1293, 1165 cm^{-1}; **HRMS** (TOF MS ES+) m / z calcd for $C_{16}H_{13}NO_2S$ $(M + Na)^+$ 306.0565, found 306.0564; **$[\alpha]^{24}_D$** +42 (c 0.7, $CHCl_3$); **SFC** analysis (OD-H, 10% IPA, 3.0 mL/min, 215 nm) indicated 99:1 er: t_R (minor) = 11.5 min, t_R (major) = 14.3 min.



3b

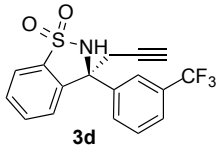
Sultam 3b was prepared according to Method A, using the following amounts of reagents: $AgPF_6$ (5.0 mg, 0.020 mmol, 0.10 equiv), Walphos W001-1 (22.3 mg, 0.0240 mmol, 0.120 equiv), *tert*-butanol (21 μL, 0.22 mmol, 1.1 equiv), potassium *tert*-butoxide (4.5 mg, 0.040 mmol, 0.20 equiv), substrate **1b** (55.9 mg, 0.200 mmol, 1.00 equiv), DMF (400 μL), and allenylboronic acid pincol ester (144 μL, 0.800 mmol, 4.00 equiv). The resulting mixture was purified by flash column chromatography using 0–1% TEA/benzene to separate product from unreacted starting material. The mixture was purified again by flash column chromatography using 5–10–20% EtOAc/hexanes (1% TEA) to separate product from excess ligand and afford the title compound as a pale yellow oil (50.5 mg, 0.158 mmol, 79%, 97:3 er). **TLC R_f** = 0.2

(20% EtOAc/hexanes, stains pink with PAA); **^{1}H NMR** (500 MHz, $CDCl_3$) δ 7.81 (d, *J* = 7.7 Hz, 1H), 7.64 (t, *J* = 7.6 Hz, 1H), 7.60 (t, *J* = 7.6 Hz, 1H), 7.42 (ddd, *J* = 11.6, 7.5, 2.5 Hz, 1H), 7.37–7.37 (d, *J* = 7.7 Hz, 1H), 7.34–7.30 (m, 1H), 7.17 (q, *J* = 8.8 Hz, 1H), 5.35 (br s, 1H), 3.25 (dd, *J* = 17.4, 2.5 Hz, 1H), 3.18 (dd, *J* = 17.4, 2.5 Hz, 1H), 2.10 (t, *J* = 2.5 Hz, 1H); **^{13}C NMR** (125 MHz, $CDCl_3$) δ 151.3 (dd, *J* = 12.5, 7.6 Hz), 149.4 (dd, *J* = 13.0, 9.0 Hz), 141.4, 137.5 (t, *J* = 4.2 Hz), 134.9, 133.6, 130.2, 124.4, 122.8 (dd, *J* = 6.5, 3.7 Hz), 121.6, 117.7 (d, *J* = 17.6 Hz), 116.3 (d, *J* = 19.0 Hz), 77.6, 73.8, 66.1, 31.5; **^{19}F NMR** (376.5 MHz, $CDCl_3$) δ –135.8 (m), –137.7 (m); **IR** (neat) 3281, 2923, 2359, 1518, 1284, 1167 cm^{-1}; **HRMS** (TOF MS ES+) *m* / *z* calcd for $C_{16}H_{11}F_2NO_2S$ $(M + Na)^+$ 342.0376, found 342.0372; **$[\alpha]^{25}_D$** +56 (*c* 0.8, $CHCl_3$); **SFC** analysis (OD-H, 10% IPA, 3.0 mL/min, 215 nm) indicated 97:3 er: t_R (major) = 8.2 min, t_R (minor) = 11.7 min.



3c

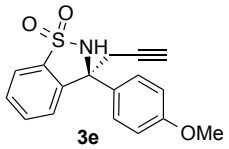
Sultam 3c was prepared according to Method A, using the following amounts of reagents: $AgPF_6$ (5.0 mg, 0.020 mmol, 0.10 equiv), Walphos W001-1 (22.3 mg, 0.0240 mmol, 0.120 equiv), *tert*-butanol (21 μL, 0.22 mmol, 1.1 equiv), potassium *tert*-butoxide (4.5 mg, 0.040 mmol, 0.20 equiv), substrate **1c** (55.5 mg, 0.200 mmol, 1.00 equiv), DMF (400 μL), and allenylboronic acid pincol ester (144 μL, 0.800 mmol, 4.00 equiv). The resulting mixture was purified by flash column chromatography using 0–1% TEA/benzene to separate product from unreacted starting material. The mixture was purified again by flash column chromatography using 5–10–20% EtOAc/hexanes (1% TEA) to separate product from excess ligand and afford the title compound as a clear oil (53.6 mg, 0.168 mmol, 84%, 97:3 er). **TLC R_f** = 0.3 (20% EtOAc/hexanes, stains pink with PAA); **^{1}H NMR** (500 MHz, $CDCl_3$) δ 7.81 (d, *J* = 7.4 Hz, 1H), 7.66–7.54 (m, 3H) 7.48–7.44 (m, 1H), 7.37 (d, *J* = 7.8 Hz, 1H), 7.35–7.29 (m, 2H), 5.30 (br s, 1H), 3.27 (dd, *J* = 17.4, 2.5 Hz, 1H), 3.20 (dd, *J* = 17.4, 2.5 Hz, 1H), 2.09 (t, *J* = 2.5 Hz, 1H); **^{13}C NMR** (125 MHz, $CDCl_3$) δ 142.4, 141.5, 134.95, 134.86, 133.5, 130.3, 130.1, 128.8, 126.8, 124.8, 124.6, 121.5, 77.8, 73.6, 66.5, 31.36; **IR** (neat) 3292, 2923, 2359, 1295, 1165 cm^{-1}; **HRMS** (TOF MS ES+) *m* / *z* calcd for $C_{16}H_{12}ClNO_2S$ $(M + Na)^+$ 340.0175, found 340.0183. **$[a]^{26}_D$** +49 (*c* 1.0, $CHCl_3$); **SFC** analysis (OD-H, 10% MeOH, 3.0 mL/min, 215 nm) indicated 97:3 er: t_R (minor) = 9.5 min, t_R (major) = 10.2 min.



3d

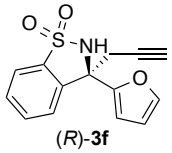
Sultam 3d was prepared according to Method A, using the following amounts of reagents: $AgPF_6$ (5.0 mg, 0.020 mmol, 0.10 equiv), Walphos W001-1 (22.3 mg, 0.0240 mmol, 0.120 equiv), *tert*-butanol (21 μL, 0.22 mmol, 1.1 equiv), potassium *tert*-butoxide (4.5 mg, 0.040 mmol, 0.20 equiv), substrate **1d** (62.3 mg, 0.200 mmol, 1.00 equiv), DMF (400 μL), and allenylboronic acid pincol ester (144 μL, 0.800 mmol, 4.00 equiv). The resulting mixture was purified by flash column chromatography using 0–1% TEA/benzene to separate product from

unreacted starting material. The mixture was purified again by flash column chromatography using 5–10–20% EtOAc/hexanes (1% TEA) to separate product from excess ligand and afford the title compound as a beige solid (49.8 mg, 0.142 mmol, 71%, 98:2 er). **TLC** $\mathbf{R_f}$ = 0.1 (20% EtOAc/hexanes, stains pink with PAA); **m.p.** = 125–127 °C; **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.85–7.82 (m, 2H), 7.78 (d, *J* = 7.9 Hz, 1H), 7.70–7.58 (m, 3H), 7.52 (t, *J* = 7.9 Hz, 1H), 7.37 (d, *J* = 7.9 Hz, 1H), 5.35 (br s, 1H), 3.32 (dd, *J* = 17.3, 2.7 Hz, 1H), 3.24 (dd, *J* = 17.3, 2.7 Hz, 1H), 2.10 (t, *J* = 2.7 Hz, 1H); **^{13}C NMR** (125.7 MHz, $CDCl_3$) δ 141.6, 141.5, 135.1, 133.8, 131.4 (q, *J* = 32.4 Hz), 130.4, 130.3, 129.8, 125.6 (q, *J* = 3.7 Hz), 124.6, 123.9 (q, *J* = 272.4 Hz), 123.3 (q, *J* = 4.0 Hz), 121.8, 77.7, 73.9, 66.7, 31.5; **^{19}F NMR** (376.5 MHz, $CDCl_3$) δ –62.6; **IR** (neat) 3302, 1329, 1164, 1125, 731 cm^{-1}; **HRMS** (TOF MS ES+) *m / z* calcd for $C_{17}H_{12}F_3NO_2S$ $(M + Na)^+$ 374.0439, found 374.0433; $\mathbf{[\alpha]^{26}_D}$ +47 (*c* 1.2, $CDCl_3$); **SFC** analysis (Whelk-O (*R*,*R*), 5% IPA, 3.0 mL/min, 215 nm) indicated 98:2 er: t_R (minor) = 10.8 min, t_R (major) = 11.3 min.



3e

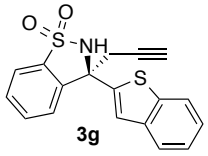
Sultam 3e was prepared according to Method A, using the following amounts of reagents: $AgPF_6$ (5.0 mg, 0.020 mmol, 0.10 equiv), Walphos W001-1 (22.3 mg, 0.0240 mmol, 0.120 equiv), *tert*-butanol (21 μL, 0.22 mmol, 1.1 equiv), potassium *tert*-butoxide (4.5 mg, 0.040 mmol, 0.20 equiv), substrate **1e** (54.7 mg, 0.200 mmol, 1.00 equiv), DMF (400 μL), and allenylboronic acid pincol ester (216 μL, 0.800 mmol, 6.00 equiv). The resulting mixture was purified by flash column chromatography using 0–1% TEA/benzene to separate product from unreacted starting material. The mixture was purified again by flash column chromatography using 5–10–20% EtOAc/hexanes (1% TEA) to separate product from excess ligand and afford the title compound as a clear oil (39.0 mg, 0.124 mmol, 62%, 99:1 er). **TLC** $\mathbf{R_f}$ = 0.1 (20% EtOAc/hexanes, stains purple with PAA); **^{1}H NMR** (500 MHz, $CDCl_3$) δ 7.81 (d, *J* = 7.7 Hz, 1H), 7.60 (t, *J* = 7.2 Hz, 1H), 7.56 (t, *J* = 7.6 Hz, 1H), 7.44 (d, *J* = 8.7 Hz, 2H), 7.35 (d, *J* = 7.7 Hz, 1H), 6.89 (d, *J* = 8.8, 2H), 5.12 (br s, 1H), 3.80 (s, 3H), 3.29–3.20 (m, 2H), 2.07 (t, *J* = 2.5 Hz, 1H); **^{13}C NMR** (125 MHz, $CDCl_3$) δ 159.8, 142.9, 135.1, 133.4, 132.3, 129.9, 128.1, 125.0, 121.4, 114.4, 78.7, 73.2, 66.8, 55.5, 31.4; **IR** (neat) 3272, 2923, 1512, 1293, 1164 cm^{-1}; **HRMS** (TOF MS ES+) *m / z* calcd for $C_{17}H_{15}NO_3S$ $(M + Na)^+$ 336.0670, found 336.0663; $\mathbf{[\alpha]^{24}_D}$ +33 (*c* 0.5, $CHCl_3$); **SFC** analysis (OD-H, 10% IPA, 3.0 mL/min, 215 nm) indicated 99:1 er: t_R (major) = 16.1 min, t_R (minor) = 21.4 min.



(*R*)-**3f**

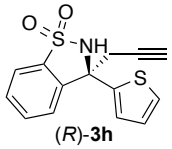
Sultam (*R*)-3f was prepared according to Method A, using the following amounts of reagents: $AgPF_6$ (5.0 mg, 0.020 mmol, 0.10 equiv), Walphos W001-1 (22.3 mg, 0.0240 mmol, 0.120 equiv), *tert*-butanol (21 μL, 0.22 mmol, 1.1 equiv), potassium *tert*-butoxide (4.5 mg, 0.040 mmol, 0.20 equiv), substrate **1f** (46.6 mg, 0.200 mmol, 1.00 equiv), DMF (400 μL), and

allenylboronic acid pincol ester (144 μL, 0.800 mmol, 4.00 equiv). The resulting mixture was purified by flash column chromatography using 0–1% TEA/benzene to separate product from unreacted starting material. The mixture was purified again by flash column chromatography using 5–20–40% EtOAc/hexanes (1% TEA) to separate product from excess ligand and afford the title compound as a white solid (35.4 mg, 0.130 mmol, 65%, 97:3 er). **TLC** $\mathbf{R_f}$ = 0.3 (20% EtOAc/hexanes, stains purple with PAA); **m.p.** = 126–127 °C; **^{1}H NMR** (500 MHz, $CDCl_3$) δ 7.80 (d, *J* = 7.6 Hz, 1H), 7.66 (td, *J* = 7.6, 1.1 Hz, 1H), 7.60–7.58 (m, 2H), 7.40 (d, *J* = 1.1 Hz, 1H), 6.48 (d, *J* = 3.3 Hz, 1H), 6.35 (dd, *J* = 3.3, 1.8 Hz, 1H), 5.29 (br s, 1H), 3.25 (dd, *J* = 17.0, 2.6 Hz, 1H), 3.19 (dd, *J* = 17.0, 2.6 Hz, 1H), 2.07 (t, *J* = 2.6 Hz, 1H); **^{13}C NMR** (125.7 MHz, $CDCl_3$) δ 152.1, 143.6, 139.7, 135.2, 133.5, 130.4, 124.8, 121.5, 110.8, 108.8, 77.8, 73.1, 63.3, 30.6; **IR** (neat) 3283, 1295, 1166, 1132, 735 cm^{-1}; **HRMS** (TOF MS ES+) *m / z* calcd for $C_{14}H_{11}NO_3S$ $(M + Na)^+$ 296.0357, found 296.0363; $\mathbf{[\alpha]^{26}_D}$ +7.0 (*c* 1.0, $CDCl_3$); **SFC** analysis (OD-H, 10% IPA, 3.0 mL/min, 215 nm) indicated 97:3 er: t_R (minor) = 7.2 min, t_R (major) = 8.6 min.



3g

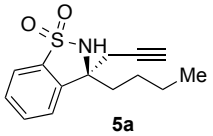
Sultam 3g was prepared according to Method A, using the following amounts of reagents: $AgPF_6$ (5.0 mg, 0.020 mmol, 0.10 equiv), Walphos W001-1 (22.3 mg, 0.0240 mmol, 0.120 equiv), *tert*-butanol (21 μL, 0.22 mmol, 1.1 equiv), potassium *tert*-butoxide (4.5 mg, 0.040 mmol, 0.20 equiv), substrate **1g** (59.9 mg, 0.200 mmol, 1.00 equiv), DMF (400 μL), and allenylboronic acid pincol ester (144 μL, 0.800 mmol, 4.00 equiv). The resulting mixture was purified by flash column chromatography using 0–1% TEA/benzene to separate product from unreacted starting material. The mixture was purified again by flash column chromatography using 5–10–20% EtOAc/hexanes (1% TEA) to separate product from excess ligand and afford the title compound as a pale yellow oil (46.7 mg, 0.138 mmol, 69%, 95:5 er). **TLC** $\mathbf{R_f}$ = 0.3 (20% EtOAc/hexanes, stains purple with PAA); **^{1}H NMR** (500 MHz, $CDCl_3$) δ 7.83 (d, *J* = 7.8 Hz, 1H), 7.77–7.73 (m, 2H), 7.67 (td, *J* = 7.6, 1.1 Hz, 1H), 7.62–7.56 (m, 2H), 7.45 (s, 1H), 7.34 (aquintd, *J* = 7.5, 1.4 Hz, 2H), 5.44 (s, 1H), 3.36 (dd, *J* = 17.1, 2.7 Hz, 1H), 3.29 (dd, *J* = 17.1, 2.7 Hz, 1H), 2.12 (t, *J* = 2.7 Hz, 1H); **^{13}C NMR** (125.7 MHz, $CDCl_3$) δ 145.5, 141.0, 139.9, 139.3, 134.9, 133.7, 130.5, 125.3, 124.9, 124.8, 124.3, 123.0, 122.5, 121.7, 77.8, 73.8, 65.4, 32.8; **IR** (neat) 3288, 1294, 1166, 1131, 726 cm^{-1}; **HRMS** (TOF MS ES+) *m / z* calcd for $C_{18}H_{13}NO_2S_2$ $(M + Na)^+$ 362.0285, found 362.0279; $\mathbf{[\alpha]^{28}_D}$ +17 (*c* 1.1, $CDCl_3$); **SFC** analysis (AS-H, 20% IPA, 3.0 mL/min, 215 nm) indicated 95:5 er: t_R (major) = 11.0 min, t_R (minor) = 16.5 min.



(*R*)-**3h**

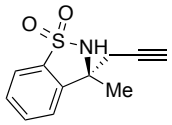
Sultam (*R*)-3h was prepared according to Method A, using the following amounts of reagents: $AgPF_6$ (5.0 mg, 0.020 mmol, 0.10 equiv), Walphos W001-1 (22.3 mg, 0.0240 mmol, 0.120

equiv), *tert*-butanol (21 μL, 0.22 mmol, 1.1 equiv), potassium *tert*-butoxide (4.5 mg, 0.040 mmol, 0.20 equiv), substrate **1h** (49.9 mg, 0.200 mmol, 1.00 equiv), DMF (400 μL), and allenylboronic acid pincol ester (144 μL, 0.800 mmol, 4.00 equiv). The resulting mixture was purified by flash column chromatography using 0–1% TEA/benzene to separate product from unreacted starting material. The mixture was purified again by flash column chromatography using 5–10–20% EtOAc/hexanes (1% TEA) to separate product from excess ligand and afford the title compound as a yellow solid (51.3 mg, 0.177 mmol, 89%, 97:3 er). **TLC** $\mathbf{R_f}$ = 0.3 (20% EtOAc/hexanes, stains purple with PAA); **m.p.** = 155–157 °C; **^{1}H NMR** (500 MHz, $CDCl_3$) δ 7.80 (d, *J* = 7.7 Hz, 1H), 7.66 (td, *J* = 7.6, 1.3 Hz, 1H), 7.59 (td, *J* = 7.5, 1.3 Hz, 1H), 7.54 (d, *J* = 7.8 Hz, 1H), 7.30 (dd, *J* = 5.1, 1.3 Hz, 1H), 7.18 (dd, *J* = 3.7, 1.3 Hz, 1H), 6.98 (dd, *J* = 5.1, 3.7 Hz, 1H), 5.34 (br s, 1H), 3.27 (d, *J* = 2.6 Hz, 2H), 2.09 (t, *J* = 2.6 Hz, 1H); **^{13}C NMR** (125.7 MHz, $CDCl_3$) δ 145.1, 141.6, 135.0, 133.6, 130.3, 127.4, 126.6, 126.2, 124.8, 121.5, 78.0, 73.5, 65.0, 33.2; **IR** (neat) 3305, 2925, 1302, 1168, 906, 728 cm^{-1}; **HRMS** (TOF MS ES+) *m / z* calcd for $C_{14}H_{11}NO_2S_2$ $(M + Na)^+$ 312.0129, found 312.0142; $\mathbf{[\alpha]^{28}_D}$ –12 (*c* 0.9, $CDCl_3$); **SFC** analysis (OD-H, 10% IPA, 3.0 mL/min, 215 nm) indicated 97:3 er: t_R (minor) = 12.5 min, t_R (major) = 15.7 min.



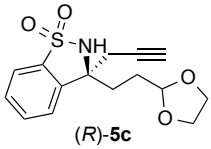
5a

Sultam 5a was prepared according to Method A, using the following amounts of reagents: $AgPF_6$ (5.0 mg, 0.020 mmol, 0.10 equiv), Walphos W001-1 (22.3 mg, 0.0240 mmol, 0.120 equiv), *tert*-butanol (21 μL, 0.22 mmol, 1.1 equiv), potassium *tert*-butoxide (4.5 mg, 0.040 mmol, 0.20 equiv), substrate **4a** (44.7 mg, 0.200 mmol, 1.00 equiv), DMF (400 μL), and allenylboronic acid pincol ester (144 μL, 0.800 mmol, 4.00 equiv). The resulting mixture was purified by flash column chromatography using 0–1% TEA/benzene to separate product from unreacted starting material. The mixture was purified again by flash column chromatography using 5–10% EtOAc/hexanes (1% TEA) to separate product from excess ligand and afford the title compound as a colorless oil (44.3 mg, 0.168 mmol, 84%, 99:1 er). **TLC** $\mathbf{R_f}$ = 0.6 (20% EtOAc/hexanes, stains pink with PAA); **^{1}H NMR** (500 MHz, $CDCl_3$) δ 7.77 (d, *J* = 7.8 Hz, 1H), 7.65 (t, *J* = 7.6, 1.0 Hz, 1H), 7.56 (t, *J* = 7.7, 0.9 Hz, 1H), 7.49 (d, *J* = 7.8 Hz, 1H), 4.87 (s, 1H), 2.82 (dd, *J* = 16.9, 2.7 Hz, 1H), 2.77 (dd, *J* = 16.9, 2.7 Hz, 1H), 2.13 (t, *J* = 2.7 Hz, 1H), 2.14–2.08 (m, 1H), 2.02–1.96 (m, 1H), 1.45–1.35 (m, 1H), 1.34–1.24 (m, 2H), 1.04–0.96 (m, 1H), 0.86 (t, *J* = 7.3 Hz, 3H); **^{13}C NMR** (125.7 MHz, $CDCl_3$) δ 141.9, 135.8, 133.4, 129.8, 123.6, 121.6, 78.8, 72.8, 65.2, 38.5, 31.8, 25.8, 22.7, 14.0; **IR** (neat) 3306, 2959, 1289, 1168, 907, 728 cm^{-1}; **HRMS** (TOF MS ES+) *m / z* calcd for $C_{14}H_{17}NO_2S$ $(M + Na)^+$ 286.0878, found 286.0884; $\mathbf{[\alpha]^{28}_D}$ –2.4 (*c* 1.1, $CDCl_3$); **SFC** analysis (AS-H, 10% IPA, 3.0 mL/min, 215 nm) indicated 99:1 er: t_R (major) = 7.5 min, t_R (minor) = 8.3 min.



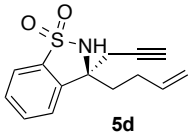
(*R*)-**5b**

Sultam (*R*)-5b was prepared according to Method A, using the following amounts of reagents: $AgPF_6$ (5.0 mg, 0.020 mmol, 0.10 equiv), Walphos W001-1 (22.3 mg, 0.0240 mmol, 0.120 equiv), *tert*-butanol (21 μL, 0.22 mmol, 1.1 equiv), potassium *tert*-butoxide (4.5 mg, 0.040 mmol, 0.20 equiv), substrate **4b** (36.2 mg, 0.200 mmol, 1.00 equiv), DMF (400 μL), and allenylboronic acid pincol ester (144 μL, 0.800 mmol, 4.00 equiv). The resulting mixture was purified by flash column chromatography using 0–1% TEA/benzene to separate product from unreacted starting material. The mixture was purified again by flash column chromatography using 5–10–20% EtOAc/hexanes (1% TEA) to separate product from excess ligand and afford the title compound as a white solid (35.7 mg, 0.161 mmol, 80%, 99:1 er). **TLC R_f** = 0.3 (20% EtOAc/hexanes, stains pink with PAA); **m.p.** = 91–93 °C; **^{1}H NMR** (500 MHz, $CDCl_3$) δ 7.77 (d, *J* = 7.8 Hz, 1H), 7.65 (td, *J* = 7.8, 1.1 Hz, 1H), 7.56 (td, *J* = 7.7, 1.0 Hz, 1H), 7.51 (d, *J* = 7.8 Hz, 1H), 4.93 (s, 1H), 2.82 (dd, *J* = 17.0, 2.7 Hz, 1H), 2.77 (dd, *J* = 17.0, 2.7 Hz, 1H), 2.15 (t, *J* = 2.7 Hz, 1H), 1.76 (s, 3H); **^{13}C NMR** (125.7 MHz, $CDCl_3$) δ 143.3, 135.6, 133.5, 129.8, 123.4, 121.5, 78.8, 72.7, 62.0, 32.6, 26.9; **IR** (neat) 3274, 2980, 2342, 1281, 1156, 1132 cm^{-1}; **HRMS** (TOF MS ES+) *m / z* calcd for $C_{11}H_{11}NO_2S$ $(M + Na)^+$ 244.0408, found 244.0410; **$[\alpha]^{27}_D$** +16 (*c* 0.9, $CDCl_3$); **SFC** analysis (OD-H, 10% IPA, 3.0 mL/min, 215 nm) indicated 99:1 er: t_R (minor) = 5.8 min, t_R (major) = 6.3 min.



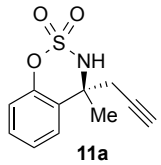
(*R*)-**5c**

Sultam (*R*)-5c was prepared according to Method A, using the following amounts of reagents: $AgPF_6$ (5.0 mg, 0.020 mmol, 0.10 equiv), Walphos W001-1 (22.3 mg, 0.0240 mmol, 0.120 equiv), *tert*-butanol (21 μL, 0.22 mmol, 1.1 equiv), potassium *tert*-butoxide (4.5 mg, 0.040 mmol, 0.20 equiv), substrate **4c** (53.5 mg, 0.200 mmol, 1.00 equiv), DMF (400 μL), and allenylboronic acid pincol ester (144 μL, 0.800 mmol, 4.00 equiv). The resulting mixture was purified by flash column chromatography using 0–1% TEA/benzene to separate product from unreacted starting material. The mixture was purified again by flash column chromatography using 10–20–30% EtOAc/hexanes (1% TEA) to separate product from excess ligand and afford the title compound as a white solid (43.9 mg, 0.143 mmol, 72%, 98:2 er). **TLC R_f** = 0.2 (20% EtOAc/hexanes, stains yellow then pink with PAA); **m.p.** = 128–129 °C; **^{1}H NMR** (500 MHz, $CDCl_3$) δ 7.77 (d, *J* = 7.7 Hz, 1H), 7.64 (t, *J* = 7.7 Hz, 1H), 7.57–7.53 (m, 2H), 5.86 (s, 1H), 4.87 (t, *J* = 3.8 Hz, 1H), 4.03–3.95 (m, 2H), 3.91–3.83 (m, 2H), 2.83 (dd, *J* = 17.0, 2.7 Hz, 1H), 2.77 (dd, *J* = 17.0, 2.7 Hz, 1H), 2.33–2.20 (m, 2H), 2.14 (t, *J* = 2.7 Hz, 1H), 1.70–1.63 (m, 1H), 1.62–1.56 (m, 1H); **^{13}C NMR** (125.7 MHz, $CDCl_3$) δ 141.7, 136.4, 133.3, 129.9, 123.8, 121.5, 103.2, 79.0, 72.7, 65.3, 65.2, 64.6, 32.4, 31.1, 27.5; **IR** (neat) 3269, 2890, 1286, 1164, 1131, 729 cm^{-1}; **HRMS** (TOF MS ES+) *m / z* calcd for $C_{15}H_{17}NO_4S$ $(M + Na)^+$ 330.0776, found 330.0781; **$[\alpha]^{27}_D$** –26 (*c* 1.2, $CDCl_3$); **SFC** analysis (AS-H, 10% IPA, 3.0 mL/min, 215 nm) indicated 98:2 er: t_R (major) = 12.4 min, t_R (minor) = 13.8 min.



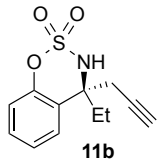
5d

Sultam 5d was prepared according to Method A, using the following amounts of reagents: $AgPF_6$ (5.0 mg, 0.020 mmol, 0.10 equiv), Walphos W001-1 (22.3 mg, 0.0240 mmol, 0.120 equiv), *tert*-butanol (21 μL, 0.22 mmol, 1.1 equiv), potassium *tert*-butoxide (4.5 mg, 0.040 mmol, 0.20 equiv), substrate **4d** (44.3 mg, 0.200 mmol, 1.00 equiv), DMF (400 μL), and allenylboronic acid pincol ester (144 μL, 0.800 mmol, 4.00 equiv). The resulting mixture was purified by flash column chromatography using 0–1% TEA/benzene to separate product from unreacted starting material. The mixture was purified again by flash column chromatography using 5–10% EtOAc/hexanes (1% TEA) to separate product from excess ligand and afford the title compound as a white solid (46.2 mg, 0.177 mmol, 88%, 98:2 er). **TLC R_f** = 0.4 (20% EtOAc/hexanes, stains pink with PAA); **m.p.** = 80–81 °C; **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.78 (d, *J* = 7.8 Hz, 1H), 7.66 (td, *J* = 7.6, 1.3 Hz, 1H), 7.57 (td, *J* = 7.6, 1.0 Hz, 1H), 7.50 (d, *J* = 7.8 Hz, 1H), 5.79–5.69 (m, 1H), 5.02–4.93 (m, 3H), 2.86–2.76 (m, 2H), 2.27–2.16 (m, 2H), 2.15 (t, *J* = 2.6 Hz, 1H), 2.13–2.05 (m, 1H), 1.87–1.76 (m, 1H); **^{13}C NMR** (125.7 MHz, $CDCl_3$) δ 141.4, 137.0, 135.8, 133.4, 129.9, 123.6, 121.6, 115.7, 78.6, 72.9, 65.0, 37.8, 32.0, 28.0; **IR** (neat) 3273, 1285, 1165, 1131, 730 cm^{-1}; **HRMS** (TOF MS ES+) *m / z* calcd for $C_{14}H_{15}NO_2S$ $(M + Na)^+$ 284.0721, found 284.0728; **$[\alpha]^{24}_D$** +0.5 (*c* 1.3, $CDCl_3$); **SFC** analysis (AS-H, 10% IPA, 3.0 mL/min, 215 nm) indicated 98:2 er: t_R (major) = 7.5 min, t_R (minor) = 8.6 min.



11a

Sultam 11a was prepared according to Method A, using the following amounts of reagents: $AgPF_6$ (5.0 mg, 0.020 mmol, 0.10 equiv), Walphos W001-1 (22.3 mg, 0.0240 mmol, 0.120 equiv), *tert*-butanol (21 μL, 0.22 mmol, 1.1 equiv), potassium *tert*-butoxide (4.5 mg, 0.040 mmol, 0.20 equiv), substrate **10a** (39.4 mg, 0.200 mmol, 1.00 equiv), DMF (400 μL), and allenylboronic acid pincol ester (144 μL, 0.800 mmol, 4.00 equiv). The resulting mixture was purified by flash column chromatography using 0–1% TEA/benzene to separate product from unreacted starting material. The mixture was purified again by flash column chromatography using 5–10–20% EtOAc/hexanes (1% TEA) to separate product from excess ligand and afford the title compound as a clear oil (36.1 mg, 0.152 mmol, 76%, 99:1 er). **TLC R_f** = 0.5 (20% EtOAc/hexanes, stains pink with PAA); **^{1}H NMR** (500 MHz, $CDCl_3$) δ 7.33 (td, *J* = 7.7, 2.5 Hz, 1H), 7.27–7.21 (m, 2H), 7.04 (d, *J* = 8.0 Hz, 1H), 4.00 (br s, 1H), 3.04 (dd, *J* = 17.2, 2.5 Hz, 1H), 2.80 (dd, *J* = 17.2, 2.5 Hz, 1H), 2.12 (t, *J* = 2.5 Hz, 1 H), 1.81 (s, 3H); **^{13}C NMR** (125 MHz, $CDCl_3$) δ 150.2, 130.0, 126.5, 125.9, 125.6, 119.5, 78.0, 73.4, 61.8, 33.2, 28.6; **IR** (neat) 3272, 2923, 1512, 1293, 1164 cm^{-1}; **HRMS** (TOF MS ES+) *m / z* calcd for $C_{11}H_{11}NO_3S$ $(M + Na)^+$ 260.0357, found 260.0356; **$[\alpha]^{26}_D$** +35 (*c* 0.9, $CHCl_3$); **SFC** analysis (AD-H, 10% IPA, 3.0 mL/min, 215 nm) indicated 99:1 er: t_R (minor) = 5.4 min, t_R (major) = 6.0 min.



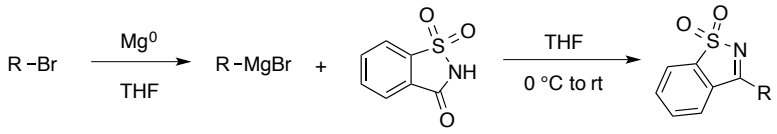
11b

Sultam 11b was prepared according to Method A, using the following amounts of reagents: $AgPF_6$ (5.0 mg, 0.020 mmol, 0.10 equiv), Walphos W001-1 (22.3 mg, 0.0240 mmol, 0.120 equiv), *tert*-butanol (21 μL, 0.22 mmol, 1.1 equiv), potassium *tert*-butoxide (4.5 mg, 0.040 mmol, 0.20 equiv), substrate **10b** (42.2 mg, 0.200 mmol, 1.00 equiv), DMF (400 μL), and allenylboronic acid pincol ester (144 μL, 0.800 mmol, 4.00 equiv). The resulting mixture was purified by flash column chromatography using 0–1% TEA/benzene to separate product from unreacted starting material. The mixture was purified again by flash column chromatography using 5–10–20% EtOAc/hexanes (1% TEA) to separate product from excess ligand and afford the title compound as a clear oil (25.6 mg, 0.102 mmol, 51%, 99:1 er). **TLC R_f** = 0.6 (20% EtOAc/hexanes, stains purple with PAA); **^{1}H NMR** (500 MHz, $CDCl_3$) δ 7.33 (td, *J* = 7.4, 2.4 Hz, 1H), 7.28–7.22 (m, 2H), 7.06 (d, *J* = 8.4 Hz, 1H), 4.71 (br s, 1H), 3.03 (dd, *J* = 17.5, 2.6 Hz, 1H), 2.87 (dd, *J* = 17.9, 3.0 Hz, 1H), 2.22 (sextet, *J* = 7.1 Hz, 1H), 2.11 (t, *J* = 2.5 Hz, 1H), 2.08 (sextet, *J* = 7.2 Hz, 1H), 0.93 (t, *J* = 7.4 Hz, 3H); **^{13}C NMR** (125 MHz, $CDCl_3$) δ 150.7, 129.9, 126.5, 125.9, 125.0, 119.5, 78.4, 73.3, 64.8, 32.9, 30.9, 7.9; **IR** (neat) 3294, 1485, 1417, 1364 cm^{-1}; **HRMS** (TOF MS ES+) *m* / *z* calcd for $C_{12}H_{13}NO_3S$ $(M + Na)^+$ 274.0514, found 274.0509; **$[\alpha]^{26}_D$** +25 (*c* 0.5, $CHCl_3$); **SFC** analysis (AD-H, 10% IPA, 3.0 mL/min, 215 nm) indicated 99:1 er: t_R (minor) = 4.7 min, t_R (major) = 5.1 min.

D. General Procedures for Starting Material Synthesis

Note: When possible, Method D was preferentially used instead of Method C in order to minimize side reactions and obtain reaction mixtures that were easier to purify. The yields for starting material synthesis are unoptimized.

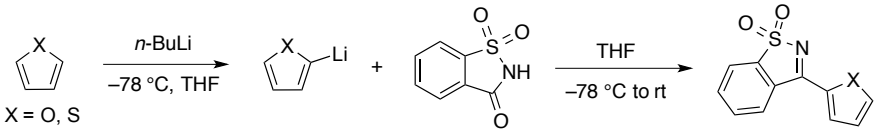
Method C: Grignard Addition into Saccharin



Prepared according to a modified procedure described by Hayashi and co-workers.[5] The Grignard reagent was typically prepared using flame-dried magnesium turnings (2.0 equiv) with a few crystals of I_2 in anhydrous THF (10 mL). The aryl halide (1.0 equiv) was added to the solution until initiation of the Grignard reagent, after which the remaining aryl halide was added dropwise at 0 °C. The reaction was stirred 2 h at rt, then titrated.[1]

The Grignard reagent (2.0 equiv) was then slowly added to a solution of saccharin (1.0 equiv) in THF (6 mL) at 0 °C. The reaction was allowed to warm to rt and stirred at 22 °C overnight. The reaction was quenched at 0 °C with saturated aqueous NH_4Cl (20 mL) and extracted with EtOAc (3 x 20 mL). The combined organic layers were washed with brine, dried over Na_2SO_4, filtered, and concentrated in vacuo. The product was purified either by recrystallization (if the unpurified material was already crystalline) or by flash column chromatography using silica gel (generally, the unpurified material was first adsorbed onto silica).

Method D: Organolithium Addition into Saccharin

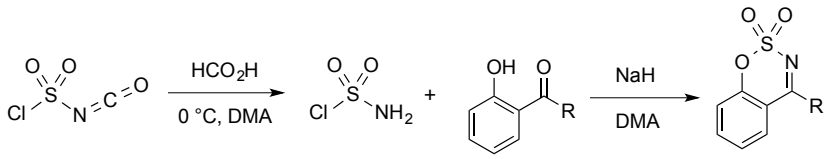


Prepared according to a modifed procedure described by Bode and co-workers.[6] The organolithium reagent was typically prepared by slow addition of *n*-butyllithium (2.75 equiv) to a solution of heterocycle (2.5 equiv) in anhydrous THF (10 mL) at –78 °C. The reaction was stirred at –78 °C for 15 min then used directly in the next step.

To this mixture was slowly added a solution of saccharin (1.0 equiv) in THF (6 mL) via syringe at –78 °C. The reaction was allowed to warm to rt slowly over several hours, then stirred at 22 °C overnight. The reaction was quenched at 0 °C with saturated aqueous NH_4Cl (20 mL) and extracted with EtOAc (3 x 20 mL). The combined organic layers were washed with brine, dried over Na_2SO_4, filtered, and concentrated in vacuo. The product was purified either by recrystallization (if the unpurified material was already crystalline) or by flash column chromatography using silica gel (generally, the unpurified material was first adsorbed onto silica).

Note: In the case of compounds **4a** or **4b**, *n*-butyllithium or methyllithium (2.2 equiv) was slowly added directly to a solution of saccharin (1.0 equiv) in THF at –78 °C.

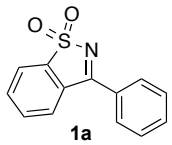
METHOD E: CHLOROSULFONYL ISOCYANATE CONDENSATION



Prepared according to a modified procedure described by Hayashi and co-workers.[7] To a flame-dried round-bottom flask was added chlorosulfonyl isocyanate (2.0 equiv), followed by anhydrous formic acid (2.0 equiv) added dropwise via syringe at 0 °C. Upon addition, a white solid was formed along with vigorous gas evolution. The viscous reaction was stirred for 10 min, until gas evolution ceased.

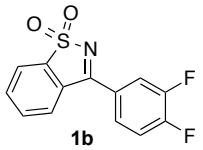
To this mixture was added neat 2-hydroxyketone (1.0 equiv) dropwise via syringe at rt, and the reaction was stirred for 10 min, then cooled to 0 °C. Anhydrous DMA (7 mL) was slowly added and the reaction mixture was warmed to rt and stirred 10 min. One portion of a solution of sodium hydride (1.2 equiv) in DMA (3 mL) was added to the reaction, followed by the other portion after 30 min. The reaction mixture was stirred 1 h at rt, then warmed to 50 °C and stirred 12 h. The reaction was quenched with H_2O (10 mL) and extracted with EtOAc (3 x 10 mL). The combined organic layers were washed with brine, dried over Na_2SO_4, filtered, and concentrated in vacuo. The product was purified by flash column chromatography using silica gel (the unpurified material was first adsorbed onto silica).

E. CHARACTERIZATION DATA FOR STARTING MATERIALS



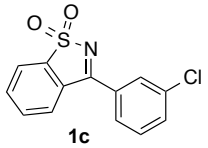
1a

***N*-Sulfonyl ketimine 1a** was prepared according Method C, using the following amounts of reagents: saccharin (1.10 g, 6.00 mmol, 1.00 equiv), phenylmagnesium bromide (6.0 mL, 12 mmol, 2.0 M in THF, 2.0 equiv) and THF (25 mL). The product was recrystallized from hot Et_2O in $CHCl_3$ (1:1 $Et_2O/CHCl_3$) to afford the title compound as a white solid (0.715 g, 2.94 mmol, 49% yield). Analytical data are consistent with literature values.[5] **TLC R_f** = 0.2 (20% EtOAc/hexanes, UV active); **m.p.** = 163–165 ˚C; **^{1}H NMR** (500 MHz, $CDCl_3$) δ 8.03 (d, *J* = 7.5 Hz, 1H), 7.98 (d, *J* = 7.3 Hz, 2H), 7.91 (d, *J* = 7.3 Hz, 1H), 7.80 (t, *J* = 7.3 Hz, 1H), 7.75 (t, *J* = 7.3 Hz, 1H), 7.71 (t, *J* = 7.5 Hz, 1H), 7.62 (t, *J* = 7.6 Hz, 2H); **^{13}C NMR** (125 MHz, $CDCl_3$) δ 171.2, 141.3, 133.8, 133.6, 133.5, 130.7, 130.6, 129.7, 129.4, 126.7, 123.3; **IR** (neat) 1599, 1531, 1332, 1171 cm^{-1}; **HRMS** (TOF MS ES+) *m* / *z* calcd for $C_{13}H_9NO_2S$ $(M + Na)^+$ 266.0252, found 266.0255.



1b

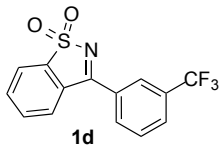
***N*-Sulfonyl ketimine 1b** was prepared according Method C, using the following amounts of reagents: saccharin (1.28 g, 7.00 mmol, 1.00 equiv), 3,4-difluorophenylmagnesium bromide (11.2 mL, 14.0 mmol, 1.25 M in THF, 2.00 equiv) and THF (20 mL). The product was recrystallized from hot Et_2O in $CHCl_3$ (1:1 $Et_2O/CHCl_3$) to afford the title compound as a yellow solid (0.235 g, 0.840 mmol, 12%). Analytical data are consistent with literature values.[5] **TLC R_f** = 0.2 (20% EtOAc/hexanes, UV active); **m.p.** = 170–171 °C; **^{1}H NMR** (500 MHz, $CDCl_3$) δ 8.04 (d, *J* = 7.5 Hz, 1H), 7.89–7.76 (m, 5H), 7.43 (q, *J* = 8.5 Hz, 1H); **^{13}C NMR** (125 MHz, $CDCl_3$) δ 169.0, 153.9 (dd, *J* = 258.9, 12.5 Hz), 150.8 (dd, *J* = 252.9, 13.4 Hz), 141.3, 134.0, 133.9, 130.0, 127.5 (dd, *J* = 6.0, 4.2 Hz), 126.7 (dd, *J* = 7.4, 3.7), 126.3, 123.6, 119.2 (d, *J* = 19.4 Hz), 118.7 (d, *J* = 18.0 Hz); **^{19}F NMR** (376.5 MHz, $CDCl_3$) δ –128.4 (m), –134.3 (dt, *J* = 20.7, 9.2 Hz); **IR** (neat) 1738, 1511, 1335, 1173 cm^{-1}; **HRMS** (TOF MS ES+) *m* / *z* calcd for $C_{13}H_7F_2NO_2S$ $(M + Na)^+$ 302.0063, found 302.0052.



1c

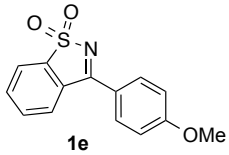
***N*-Sulfonyl ketimine 1c** was prepared according Method C, using the following amounts of reagents: saccharin (0.824 g, 4.50 mmol, 1.00 equiv), 3-chlorophenylmagnesium bromide (6.0 mL, 9.0 mmol, 1.5 M in THF, 2.0 equiv) and THF (15 mL). The product was recrystallized from hot Et_2O in $CHCl_3$ (1:1 $Et_2O/CHCl_3$) to afford the title compound as a white solid (0.700 g, 2.52 mmol, 56%). Analytical data are consistent with literature values.[5] **TLC R_f** = 0.2 (20%

EtOAc/hexanes, UV active); **m.p.** = 149–151 °C; **^{1}H NMR** (500 MHz, $CDCl_3$) δ 8.03 (d, *J* = 7.2 Hz, 1H), 7.96 (s, 1H), 7.87 (t, *J* = 7.5 Hz, 2H), 7.82 (t, *J* = 7.2 Hz, 1H), 7.78 (t, *J* = 7.6 Hz, 1H), 7.68 (d, *J* = 8.3 Hz, 1H), 7.57 (t, *J* = 8.1 Hz, 1H); **^{13}C NMR** (125 MHz, $CDCl_3$) δ 170.0, 141.2, 135.6, 134.0, 133.8, 133.5, 132.2, 130.7, 130.2, 129.5, 127.7, 126.4, 123.5; **IR** (neat) 1537, 1332, 1173 cm^{-1}; **HRMS** (TOF MS ES+) *m* / *z* calcd for $C_{13}H_8ClNO_2S$ $(M + Na)^+$ 299.9862, found 299.9863.



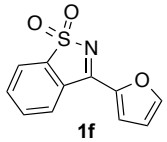
1d

***N*-Sulfonyl ketimine 1d** was prepared according Method C, using the following amounts of reagents: saccharin (0.46 g, 2.5 mmol, 1.0 equiv), (3-(trifluoromethyl)phenyl)magnesium iodide (4.00 mL, 5.00 mmol, 1.25 M in THF, 2.00 equiv) and THF (2.5 mL). The product was recrystallized from hot Et_2O in $CHCl_3$ (1:1 $Et_2O/CHCl_3$) to afford the title compound as a pale yellow solid (0.21 g, 0.68 mmol, 27%). **TLC R_f** = 0.4 (20% EtOAc/hexanes, UV active); **m.p.** = 154–156 °C; **^{1}H NMR** (500 MHz, $CDCl_3$) δ 8.22 (s, 1H), 8.16 (d, *J* = 7.8 Hz, 1H), 8.04 (d, *J* = 7.8 Hz, 1H), 7.96 (d, *J* = 8.0 Hz, 1H), 7.84–7.81 (m, 2H), 7.80–7.76 (m, 2H); **^{13}C NMR** (125.7 MHz, $CDCl_3$) δ 169.9, 141.3, 134.1, 133.9, 132.7, 132.2 (q, *J* = 33.3 Hz), 131.5, 130.14, 130.09, 129.9 (q, *J* = 3.7 Hz), 126.4 (q, *J* = 3.7 Hz), 126.3, 123.55, 123.54 (q, *J* = 272.8 Hz); **^{19}F NMR** (376.5 MHz, $CDCl_3$) δ –62.9; **IR** (neat) 1614, 1325, 1281, 1166, 1123 cm^{-1}; **HRMS** (TOF MS ES+) *m* / *z* calcd for $C_{14}H_8F_3NO_2S$ $(M + Na)^+$ 334.0125, found 334.0131.



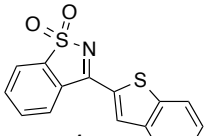
1e

***N*-Sulfonyl ketimine 1e** was prepared according Method C, using the following amounts of reagents: saccharin (1.37 g, 7.50 mmol, 1.00 equiv), 4-methoxyphenylmagnesium bromide (7.5 mL, 15 mmol, 2.0 M in THF, 2.0 equiv) and THF (25 mL). The product was recrystallized from hot Et_2O in $CHCl_3$ (1:1 $Et_2O/CHCl_3$) to afford the title compound as a light green solid (1.00 g, 3.68 mmol, 49%). Analytical data are consistent with literature values.[5] **TLC R_f** = 0.1 (20% EtOAc/hexanes, UV active); **m.p.** = 205–206 °C; **^{1}H NMR** (500 MHz, $CDCl_3$) δ 8.05–8.00 (m, 3H), 7.96 (d, *J* = 7.3 Hz, 1H), 7.76 (dt, *J* = 18.2, 7.5 Hz, 2H), 7.10 (d, *J* = 8.7 Hz, 2H), 3.94 (s, 3H); **^{13}C NMR** (125 MHz, $CDCl_3$) δ 170.1, 164.3, 141.5, 133.6, 133.3, 132.1, 131.1, 126.7, 123.2, 123.0, 114.9, 55.8; **IR** (neat) 1599, 1503, 1316, 1254, 1159 cm^{-1}; **HRMS** (TOF MS ES+) *m* / *z* calcd for $C_{14}H_{11}NO_3S$ $(M + Na)^+$ 296.0357, found 296.0362.



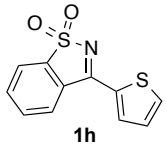
1f

***N*-Sulfonyl ketimine 1f** was prepared according Method D, using the following amounts of reagents: furan (0.910 mL, 12.5 mmol, 2.50 equiv), *n*-BuLi (5.50 mL, 13.8 mmol, 2.50 M in hexanes, 2.75 equiv), saccharin (0.92 g, 5.0 mmol, 1.0 equiv), and THF (15 mL). The product was recrystallized from hot Et_2O in $CHCl_3$ (1:1 $Et_2O/CHCl_3$) to afford the title compound as a yellow solid (0.16 g, 0.70 mmol, 14%). Analytical data are consistent with literature values.[5] **TLC** $\mathbf{R_f}$ = 0.2 (20% EtOAc/hexanes, UV active); **^{1}H NMR** (500 MHz, $CDCl_3$) δ 8.49 (dd, *J* = 5.5, 3.0 Hz, 1H), 7.98 (dd, *J* = 5.5, 3.0 Hz, 1H), 7.91 (s, 1H), 7.79–7.76 (m, 3H), 6.79 (dd, *J* = 3.7, 1.6 Hz, 1H); **^{13}C NMR** (125.7 MHz, $CDCl_3$) δ 158.1, 149.4, 148.2, 141.0, 133.9, 133.6, 129.8, 127.6, 122.8, 122.2, 114.1; **IR** (neat) 1600, 1571, 1516, 1320, 1165 cm^{-1}; **HRMS** (TOF MS ES+) *m / z* calcd for $C_{11}H_7NO_3S$ $(M + Na)^+$ 256.0044, found 256.0043.



1g

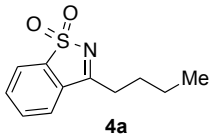
***N*-Sulfonyl ketimine 1g** was prepared according Method D, using the following amounts of reagents: benzothiophene (1.46 mL, 12.5 mmol, 2.50 equiv), *n*-BuLi (5.50 mL, 13.8 mmol, 2.50 M in hexanes, 2.75 equiv), saccharin (0.92 g, 5.0 mmol, 1.0 equiv), and THF (16 mL). The product was purified by flash column chromatography using 20–30–50% EtOAc/hexanes to afford the title compound as a yellow solid (0.29 g, 0.96 mmol, 19%). **TLC** $\mathbf{R_f}$ = 0.2 (20% EtOAc/hexanes, UV active); **m.p.** = 266–268 °C; **^{1}H NMR** (500 MHz, $CDCl_3$) δ 8.46 (s, 1H), 8.30–8.28 (m, 1H), 8.05–8.03 (m, 1H), 7.99 (d, *J* = 8.1 Hz, 1H), 7.96 (d, *J* = 8.2 Hz, 1H), 7.83–7.81 (m, 2H), 7.56 (t, *J* = 7.6 Hz, 1H), 7.50 (t, *J* = 7.6 Hz, 1H); **^{13}C NMR** (125.7 MHz, $CDCl_3$) δ 164.0, 142.9, 141.4, 139.2, 133.9, 133.7, 132.7, 130.4, 128.6, 126.1, 126.0, 125.8, 123.3, 122.9; **IR** (neat) 1593, 1526, 1316, 1171, 744 cm^{-1}; **HRMS** (TOF MS ES+) *m / z* calcd for $C_{15}H_9NO_2S_2$ $(M + Na)^+$ 321.9973, found 321.9986.



1h

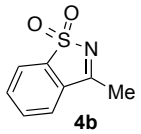
***N*-Sulfonyl ketimine 1h** was prepared according Method C, using the following amounts of reagents: saccharin (0.92 g, 5.0 mmol, 1.0 equiv), 2-thienylmagnesium bromide (8.00 mL, 10.0 mmol, 1.25 M in THF, 2.00 equiv) and THF (5 mL). The product was recrystallized from hot Et_2O in $CHCl_3$ (1:1 $Et_2O/CHCl_3$) to afford the title compound as an orange solid (0.22 g, 0.90 mmol, 18%). Analytical data are consistent with literature values.[6] **TLC** $\mathbf{R_f}$ = 0.2 (20% EtOAc/hexanes, UV active); **^{1}H NMR** (500 MHz, $CDCl_3$) δ 8.23 (d, *J* = 3.9 Hz, 1H), 8.21–8.18 (m, 1H), 8.03–8.00 (m, 1H), 7.88 (d, *J* = 5.0 Hz, 1H), 7.82–7.77 (m, 2H), 7.34 (t, *J* = 4.4 Hz, 1H); **^{13}C NMR** (125.7 MHz, $CDCl_3$) δ 163.1, 141.4, 135.8, 135.3, 134.0, 133.8, 133.6, 130.5,

129.2, 125.8, 123.2; **IR** (neat) 1594, 1416, 1316, 1164, 725 cm^{-1}; **HRMS** (TOF MS ES+) *m / z* calcd for $C_{11}H_7NO_2S_2$ $(M + Na)^+$ 271.9816, found 271.9816.



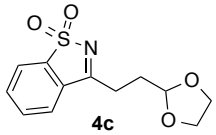
4a

***N*-Sulfonyl ketimine 4a** was prepared according Method D, using the following amounts of reagents: saccharin (0.92 g, 5.0 mmol, 1.0 equiv), *n*-BuLi (5.50 mL, 13.8 mmol, 2.50 M in hexanes, 2.75 equiv), and THF (18 mL). The product was purified by flash column chromatography using 20% EtOAc/hexanes to afford the title compound as a yellow solid (0.40 g, 1.8 mmol, 36%). Analytical data are consistent with literature values.[6] **TLC R_f** = 0.4 (20% EtOAc/hexanes, stains with $KMnO_4$); **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.93–7.90 (m, 1H), 7.77–7.68 (m, 3H), 2.97 (t, *J* = 7.4 Hz, 2H), 1.88 (quint, *J* = 7.4 Hz, 2H), 1.51 (sext, *J* = 7.4 Hz, 2H), 0.99 (t, *J* = 7.4 Hz, 3H); **^{13}C NMR** (125.7 MHz, $CDCl_3$) δ 176.5, 140.0, 134.0, 133.6, 131.5, 124.0, 122.6, 31.0, 27.6, 22.5, 13.9; **IR** (neat) 2342, 1604, 1558, 1332, 1172 cm^{-1}; **HRMS** (TOF MS ES+) *m / z* calcd for $C_{11}H_{13}NO_2S$ $(M + Na)^+$ 246.0565, found 246.0568.



4b

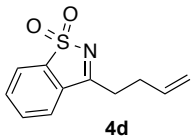
***N*-Sulfonyl ketimine 4b** was prepared according Method D, using the following amounts of reagents: saccharin (1.83 g, 10.0 mmol, 1.00 equiv), MeLi (15.7 mL, 22.0 mmol, 1.40 M in Et_2O, 2.20 equiv) and THF (10 mL). The product was purified by flash column chromatography using 20% EtOAc/hexanes to afford the title compound as a white solid (1.13 g, 6.21 mmol, 62%). Analytical data are consistent with literature values.[6] **TLC R_f** = 0.3 (20% EtOAc/hexanes, stains with $KMnO_4$); **^{1}H NMR** (500 MHz, $CDCl_3$) δ 7.99–7.97 (m, 1H), 7.84–7.79 (m, 2H), 7.76–7.75 (m, 1H), 2.73 (s, 3H); **^{13}C NMR** (125.7 MHz, $CDCl_3$) δ 173.4, 139.7, 134.1, 133.7, 131.7, 124.3, 122.5, 17.7; **IR** (neat) 2341, 1558, 1316, 1168, 771 cm^{-1}; **HRMS** (TOF MS ES+) *m / z* calcd for $C_8H_7NO_2S$ $(M + Na)^+$ 204.0095, found 204.0098.



4c

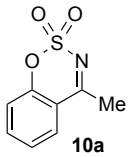
***N*-Sulfonyl ketimine 4c** was prepared according Method C, using the following amounts of reagents: saccharin (1.2 g, 6.5 mmol, 1.0 equiv), (2-(1,3-dioxolan-2-yl)ethyl)magnesium bromide (14.0 mL, 13.0 mmol, 0.900 M in THF, 2.00 equiv) and THF (17 mL). The product was purified by flash column chromatography using 50% EtOAc/hexanes to afford the title compound as a white solid (0.80 g, 3.0 mmol, 46%). **TLC R_f** = 0.5 (50% EtOAc/hexanes, UV active); **m.p.** = 68–70 °C; **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.93–7.90 (m, 1H), 7.77–7.71 (m, 3H), 5.07 (t, *J* = 4.0 Hz, 1H), 4.01–3.95 (m, 2H), 3.93–3.87 (m, 2H), 3.12 (t, *J* = 7.4 Hz, 2H), 2.31 (td, *J* = 7.4, 4.0 Hz, 2H); **^{13}C NMR** (125.7 MHz, $CDCl_3$) δ 176.2, 139.8, 134.0, 133.6, 131.3, 124.1, 122.5,

102.6, 65.2, 29.0, 25.1; **IR** (neat) 1607, 1333, 1172, 909, 726 cm^{-1}; **HRMS** (TOF MS ES+) *m / z* calcd for $C_{12}H_{13}NO_4S$ $(M + Na)^+$ 290.0463, found 290.0465.



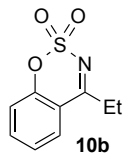
4d

***N*-Sulfonyl ketimine 4d** was prepared according Method C, using the following amounts of reagents: saccharin (0.82 g, 4.5 mmol, 1.0 equiv), 4-butenylmagnesium bromide (5.0 mL, 4.5 mmol, 0.90 M in THF, 1.0 equiv) and THF (6 mL). The product was purified by flash column chromatography using 10–20% EtOAc/hexanes to afford the title compound as a white solid (0.16 g, 0.71 mmol, 16%). Analytical data are consistent with literature values.[8] **TLC** $\mathbf{R_f}$ = 0.3 (20% EtOAc/hexanes, stains with $KMnO_4$); **m.p.** = 81 °C; **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.93–7.88 (m, 1H), 7.78–7.68 (m, 3H), 5.99–5.89 (m, 1H), 5.15 (dd, *J* = 17.1, 1.5 Hz, 1H), 5.09 (dd, *J* = 10.2, 1.4 Hz, 1H), 3.07 (t, *J* = 7.5 Hz, 2H), 2.69–2.63 (m, 2H); **^{13}C NMR** (125.7 MHz, $CDCl_3$) δ 175.7, 139.9, 135.9, 134.1, 133.7, 131.3, 124.0, 122.6, 116.7, 30.6, 29.2; **IR** (neat) 2923, 2257, 1558, 1334, 907, 726 cm^{-1}; **HRMS** (TOF MS ES+) *m / z* calcd for $C_{11}H_{11}NO_2S$ $(M + Na)^+$ 244.0408, found 244.0419.



10a

***N*-Sulfonyl ketimine 10a** was prepared according Method E, using the following amounts of reagents: chlorosulfonyl isocyanate (1.74 mL, 20.0 mmol, 2.00 equiv), anhydrous formic acid (0.750 mL, 20.0 mmol, 2.00 equiv), 2-hydroxyacetophenone (1.20 mL, 10.0 mmol, 1.00 equiv), sodium hydride (0.576 g, 24.0 mmol, 2.4 equiv) and DMA (25 mL). The product was purified by flash column chromatography using 20% EtOAc/hexanes to afford the title compound as a light yellow solid (0.690 g, 3.50 mmol, 34%). Analytical data are consistent with literature values.[7] **TLC** $\mathbf{R_f}$ = 0.3 (20% EtOAc/hexanes, UV active); **m.p.** = 112–114 °C; **^{1}H NMR** (500 MHz, $CDCl_3$) δ 7.81 (dd, *J* = 8.2, 1.5 Hz, 1H), 7.72 (td, *J* = 7.6, 1.6 Hz, 1H), 7.40 (t, *J* = 7.5 Hz, 1H), 7.30 (d, *J* = 8.3 Hz, 1H), 2.74 (s, 3H); **^{13}C NMR** (125 MHz, $CDCl_3$) δ 177.4, 153.6, 137.2, 128.6, 126.0, 119.3, 116.6, 23.9; **IR** (neat) 1594, 1556, 1367, 1323 cm^{-1}; **HRMS** (TOF MS ES+) *m / z* calcd for $C_8H_7NO_3S$ $(M + Na)^+$ 220.0044, found 220.0046.



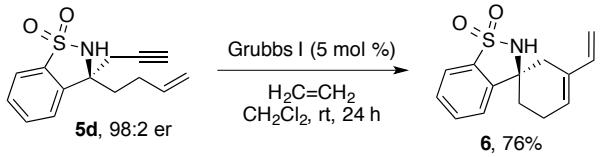
10b

***N*-Sulfonyl ketimine 10b** was prepared according Method E, using the following amounts of reagents: chlorosulfonyl isocyanate (1.74 mL, 20.0 mmol, 2.00 equiv), anhydrous formic acid (0.750 mL, 20.0 mmol, 2.00 equiv), 2-hydroxypropiophenone (1.74 mL, 10.0 mmol, 1.00 equiv),

sodium hydride (0.576 g, 24.0 mmol, 2.4 equiv) and DMA (25 mL). The product was purified by flash column chromatography using 20% EtOAc/hexanes to afford the title compound as a light yellow solid (1.06 g, 5.02 mmol, 50%). Analytical data are consistent with literature values.[7] **TLC R_f** = 0.4 (20% EtOAc/hexanes, UV active); **m.p.** = 79–81 °C **^{1}H NMR** (500 MHz, $CDCl_3$) δ 7.82 (dd, *J* = 8.0, 1.2 Hz, 1H), 7.71 (td, *J* = 7.6, 1.6 Hz, 2H), 7.39 (d, *J* = 7.3 Hz, 1H), 7.30 (t, *J* = 78.0 Hz, 1H), 7.36 (d, *J* = 7.2 Hz, 1H), 3.10 (t, *J* = 7.3 Hz, 2H), 1.36 (t, *J* = 7.2 Hz, 2H); **^{13}C NMR** (125 MHz, $CDCl_3$) δ 180.9, 153.6, 136.9, 1279, 126.0, 119.4, 116.2, 29.4, 9.8; **IR** (neat) 1596, 1553, 1373, 1360 cm^{-1}; **HRMS** (TOF MS ES+) *m / z* calcd for $C_9H_9NO_3S$ $(M + Na)^+$ 234.0201, found 234.0202.

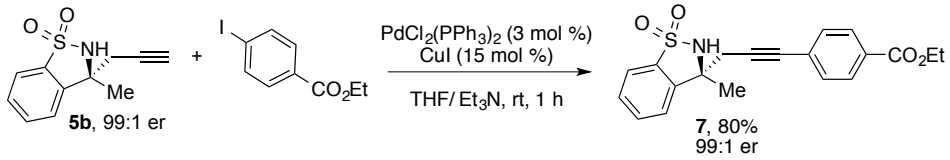
F. Synthetic Transformations of Homopropargylic Sultams (Scheme 3)

1) Enyne Ring-Closing Metathesis to Form 6



Sultam 6 was prepared according to a modified procedure described by Mori and co-workers.[9] To a flame-dried 7 mL reaction vial equipped with a N_2 line and Grubbs 1st generation catalyst (6.0 mg, 0.0070 mmol, 0.050 equiv) was added substrate **5d** (36.8 mg, 0.140 mmol, 1.00 equiv) in anhydrous CH_2Cl_2 (5 mL). The N_2 atmosphere was exchanged with ethylene (1 atm, balloon), taking care to fully purge the vial of N_2. After stirring 24 h at room temperature, the reaction mixture was concentrated in vacuo. The product was purified by flash column chromatography using 5–10% EtOAc/hexanes (1% TEA) to afford the title compound as a colorless oil (28.0 mg, 0.110 mmol, 76%). Enantiomeric ratio could not be determined for the title compound using chiral SFC instrumentation due to lack of separation of the enantiomers. **TLC R_f** = 0.4 (20% EtOAc/hexanes, stains blue with PAA); **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.78 (d, *J* = 7.7 Hz, 1H), 7.66 (t, *J* = 7.6 Hz, 1H), 7.56 (t, *J* = 7.6 Hz, 1H), 7.41 (d, *J* = 8.0 Hz, 1H), 6.42 (dd, *J* = 17.6, 10.9 Hz, 1H), 5.94 (s, 1H), 5.04–4.97 (m, 2H), 4.72 (s, 1H), 2.69 (d, *J* = 17.4 Hz, 1H), 2.58–2.49 (m, 2H), 2.45–2.37 (m, 1H), 2.07–1.94 (m, 2H); **^{13}C NMR** (125.7 MHz, $CDCl_3$) δ 144.6, 138.6, 135.5, 133.6, 133.2, 129.6, 128.7, 123.4, 121.6, 111.7, 61.9, 37.0, 33.3, 23.4; **IR** (neat) 3455, 3251, 2926, 1161, 1061, 744 cm^{-1}; **HRMS** (TOF MS ES+) *m / z* calcd for $C_{14}H_{15}NO_2S$ (M + Na)$^+$ 284.0721, found 284.0712; **$[\alpha]^{24}_D$** –14 (*c* 0.9, $CDCl_3$).

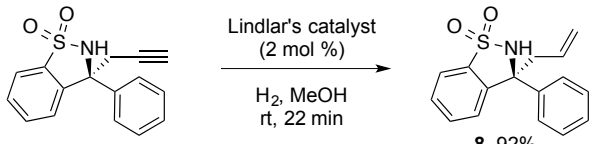
2) Sonogashira Cross-Coupling Reaction to Form 7



Sultam 7 was prepared according to a modified procedure described by Hoppe and co-workers.[10] To a flame-dried 7 mL reaction vial equipped with a N_2 line, substrate **5b** (22.1 mg, 0.100 mmol, 1.00 equiv), bis(triphenylphosphine)palladium(II) dichloride (2.1 mg, 0.0030 mmol, 0.030 equiv), and copper(I) iodide (2.9 mg, 0.015, 0.15 equiv) was added anhydrous THF (0.7 mL) then anhydrous TEA (0.3 mL). Ethyl 4-iodobenzoate (37 µL, 0.20 mmol, 2.0 equiv) was added via syringe. After stirring 1 h at room temperature, the reaction mixture was quenched with 1 M HCl (2 mL), extracted with EtOAc (3 x 2 mL), rinsed with brine, dried with Na_2SO_4, filtered, and concentrated in vacuo. The product was purified by flash column chromatography using 5–30% EtOAc/hexanes (1% TEA) to afford the title compound as a yellow solid (29.8 mg, 0.0807 mmol, 80%, 99:1 er). **TLC R_f** = 0.2 (20% EtOAc/hexanes, UV active, stains pink with PAA); **m.p.** = 162–164 °C; **^{1}H NMR** (500 MHz, $CDCl_3$) δ 7.97 (d, *J* = 8.4 Hz, 2H), 7.80 (d, *J* = 7.7 Hz,

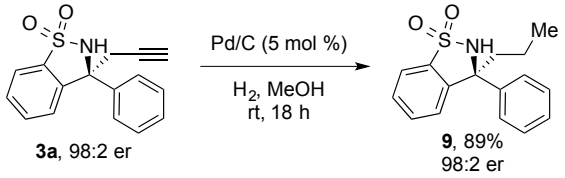
1H), 7.67 (td, *J* = 7.7, 1.0 Hz, 1H), 7.57 (td, *J* = 7.7, 1.0 Hz, 1H), 7.52 (d, *J* = 8.0 Hz, 3H), 7.42 (d, *J* = 8.4, 2H), 4.83 (br s, 1H), 4.38 (q, *J* = 7.1 Hz, 2H), 3.08–3.00 (m, 2H), 1.82 (s, 3H), 1.39 (t, *J* = 7.1 Hz, 3H); **^{13}C NMR** (125.7 MHz, $CDCl_3$) δ 166.0, 143.3, 135.7, 133.4, 131.6, 130.1, 129.8, 129.5, 127.1, 123.3, 121.5, 87.0, 83.9, 62.4, 61.2, 33.7, 27.2, 14.4; **IR** (neat) 3254, 2982, 1713, 1606, 1274, 1172 cm^{-1}; **HRMS** (TOF MS ES+) *m / z* calcd for $C_{20}H_{19}NO_4S$ $(M + Na)^+$ 392.0933, found 392.0932; **$[\alpha]^{24}_D$** +8 (*c* 0.8, $CDCl_3$); **SFC** analysis (OD-H, 20% IPA, 3.0 mL/min, 215 nm) indicated 99:1 er: t_R (minor) = 5.8 min, t_R (major) = 6.3 min.

3) LINDLAR REDUCTION TO FORM **8**



Sultam 8 was prepared according to a modified procedure described by Jarvo and co-workers.[11] To a flame-dried 7 mL reaction vial equipped with a N_2 line, substrate **3a** (41.0 mg, 0.145 mmol, 1.00 equiv), and palladium, 5% on calcium carbonate, lead poisoned (6.4 mg, 2 mol % palladium relative to **3a**) was added anhydrous MeOH (2 mL). The N_2 atmosphere was exchanged with H_2 (1 atm, balloon) and the reaction was allowed to stir at room temperature. After 22 min, the H_2 atmosphere was exchanged with N_2 and the reaction mixture was filtered through a pad of Celite using 50% EtOAc/hexanes, and then concentrated in vacuo. The product was purified by flash column chromatography using 5–10% EtOAc/hexanes (1% TEA) to afford the title compound as a white solid (38.2 mg, 0.134 mmol, 92%, 97:3 er). **TLC R_f** = 0.1 (10% EtOAc/hexanes, stains blue with PAA); **m.p.** = 125–127 °C; **^{1}H NMR** (500 MHz, $CDCl_3$) δ 7.77 (d, *J* = 7.8 Hz, 1H), 7.62–7.58 (m, 3H), 7.52 (t, *J* = 7.7 Hz, 1H), 7.38 (t, *J* = 7.9 Hz, 3H), 7.30 (m, 1H), 5.66–5.57 (m, 1H), 5.28–5.21 (m, 2H), 5.00 (s, 1H), 3.25 (dd, *J* = 14.3, 6.6 Hz, 1H), 3.03 (dd, *J* = 14.3, 7.7 Hz, 1H); **^{13}C NMR** (125.7 MHz, $CDCl_3$) δ 143.2, 141.7, 134.8, 133.5, 131.7, 129.6, 129.1 128.3, 126.3, 124.6, 122.0, 121.6, 67.7, 45.2; **IR** (neat) 3290, 3069, 1295, 1168, 906, 729 cm^{-1}; **HRMS** (TOF MS ES+) *m / z* calcd for $C_{16}H_{15}NO_2S$ $(M + Na)^+$ 308.0721, found 308.0723; **$[\alpha]^{24}_D$** +72 (*c* 0.9, $CDCl_3$); **SFC** analysis (OD-H, 10% IPA, 3.0 mL/min, 215 nm) indicated 97:3 er: t_R (minor) = 9.9 min, t_R (major) = 12.6 min.

4) PD/C REDUCTION TO FORM **9**

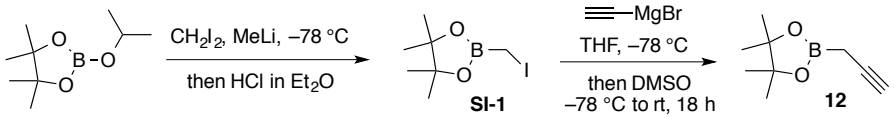


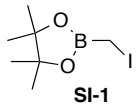
Sultam 9. To a flame-dried 7 mL reaction vial equipped with a N_2 line and palladium, 10% on carbon (6.3 mg, 5 mol % palladium relative to **3a**) was added substrate **3a** (32.3 mg, 0.114 mmol, 1.00 equiv) in anhydrous MeOH (1.0 mL). The vial was evacuated and refilled with N_2 three times. The N_2 atmosphere was exchanged with H_2 (1 atm, balloon) and the reaction was allowed to stir at room temperature. After 18 h, the H_2 atmosphere was exchanged with N_2 and the reaction mixture was filtered through a pad of Celite using MeOH, and then concentrated in vacuo. The product was purified by flash column chromatography using 5–10–20% EtOAc/hexanes (1% TEA) to afford the title compound as a white solid (29.2 mg, 0.102 mmol, 89%, 98:2 er). **TLC** $\mathbf{R_f}$ = 0.5 (20% EtOAc/hexanes, stains blue with PAA); **m.p.** = 160–162 °C; $^{\mathbf{1}}$**H NMR** (400 MHz, $CDCl_3$) δ 7.78 (d, *J* = 7.7 Hz, 1H), 7.60–7.49 (m, 4H), 7.36 (t, *J* = 7.7 Hz, 2H), 7.29 (t, *J* = 7.0 Hz, 2H), 4.86 (br s, 1H), 2.44–2.36 (m, 1H), 2.33–2.26 (m, 1H), 1.57–1.46 (m, 1H), 1.24–1.02 (m, 1H), 0.94 (t, *J* = 7.3 Hz, 3H); $^{\mathbf{13}}$**C NMR** (125.7 MHz, $CDCl_3$) δ 143.6, 142.5, 134.5, 133.5, 129.4, 129.1, 128.2, 126.1, 124.4, 121.4, 68.9, 42.6, 17.5, 14.1; **IR** (neat) 3251, 2958, 1450, 1281, 1157, 765 cm^{-1}; **HRMS** (TOF MS ES+) *m / z* calcd for $C_{16}H_{17}NO_2S$ $(M + Na)^+$ 310.0878, found 310.0883; $\mathbf{[\alpha]^{25}_D}$ +95 (*c* 0.9, $CDCl_3$); **SFC** analysis (OD-H, 10% IPA, 3.0 mL/min, 215 nm) indicated 98:2 er: t_R (minor) = 11.0 min, t_R (major) = 13.9 min.

G. MECHANISTIC STUDIES (TABLE 2)

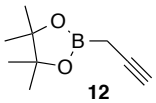
1) SYNTHESIS OF PROPARGYL BOROLANE REAGENT **12**

Scheme SI-2. Synthesis of propargyl borolane **12**.





2-(iodomethyl)-4,4,5,5-tetramethyl-1,3,2-dioxoborolane SI-1 was prepared according to a modified procedure described by Brown and co-workers,[12] using the following amounts of reagents: 2-isopropoxy-4,4,5,5-tetramethyl-1,3,2-dioxaborolane (17.7 mL, 86.9 mmol, 1.00 equiv), diiodomethane (7.00 mL, 86.9 mmol, 1.00 equiv), methyllithium (52.3 mL, 86.9 mmol, 1.66 M in Et_2O, 1.00 equiv), THF (45 mL), and anhydrous HCl (93.0 mL, 93.0 mmol, 1.00 M in Et_2O, 1.07 equiv). The resulting red solution was filtered through a plug of silica gel (40 mL) eluting with 10% Et_2O/pentanes and concentrated in vacuo. Distillation twice through a short path distillation apparatus onto activated 4Å molecular sieves at 13.7 mmHg and T_{vap} = 95 °C provided the title compound as a clear, colorless liquid (7.64 g, 28.5 mmol, 33%). Analytical data are consistent with literature values.[12] **TLC R_f** = 0.7 (10% Et_2O/pentanes, stains blue with PAA); **^{1}H NMR** ($CDCl_3$, 400 MHz) δ 2.16 (s, 2H), 1.28 (s, 12 H); **^{13}C NMR** ($CDCl_3$, 125.7 MHz) δ 84.1, 24.5; **^{11}B NMR** ($CDCl_3$, 160.2 MHz) δ 31.7; **IR** (neat) 2977, 1322, 1142, 844, 673, 577 cm^{-1}; **HRMS** (TOF MS CI+) *m* / *z* calcd for $C_7H_{14}BIO_2$ $(M)^+$ 268.0133, found 268.0143.



Propargylboronic acid pinacol ester 12 was prepared according to a literature procedure by Fandrick and co-workers,[4] using the following amounts of reagents: **SI-1** (2.0 mL, 11 mmol, 1.0 equiv), ethynylmagnesium bromide (19.8 mL, 11.2 mmol, 0.566 M in THF, 1.02 equiv), THF (15 mL), and 1:1 THF/DMSO (24 mL). Distillation using a Kugelrohr distillation apparatus at 3–6 torr and T_{vap} = 60 °C provided the title compound as a 96:4 mixture of propargyl borolane **12** and allenyl borolane **2** as a clear, colorless oil (0.37 g, 2.2 mmol, 20%). Analytical data for allenyl borolane **2** are consistent with literature values.[3] **TLC R_f** = 0.9 (10% Et_2O/hexanes, stains blue with PAA); **^{1}H NMR** (400 MHz, DMF-d_7) δ 4.90 (t, *J* = 7.0 Hz, 1H), 4.71 (d, *J* = 7.0 Hz, 2H), 1.26 (s, 12H). Analytical data for propargyl borolane **12** are consistent with literature values.[4] **^{1}H NMR** (400 MHz, DMF-d_7) δ 2.46 (t, *J* = 2.9 Hz, 1H), 1.78 (d, *J* = 2.9 Hz, 2H), 1.26 (s, 12H); **^{13}C NMR** (125.7 MHz, DMF-d_7) δ 84.9, 81.9, 69.5, 25.3.

Note: We found that in order to obtain high ratios of propargyl borolane to allenyl borolane, it was necessary to use precisely 1.02 equivalents of ethynylmagnesium bromide relative to iodomethyl borolane **SI-1**. Excess Grignard reagent (1.1 equivalents) causes isomerization to allenyl borolane, while fewer than 1.0 equivalents of Grignard reagent results in low conversion to product (Figure SI-1).

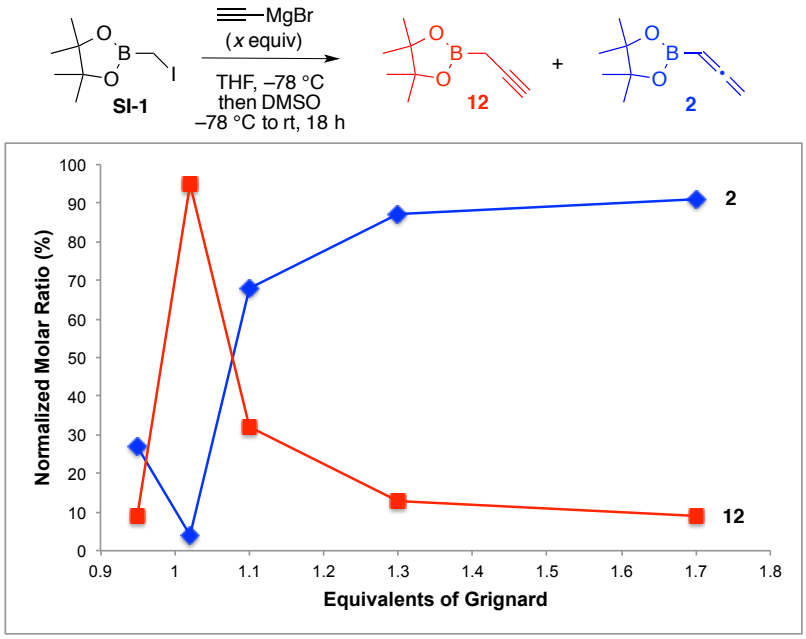
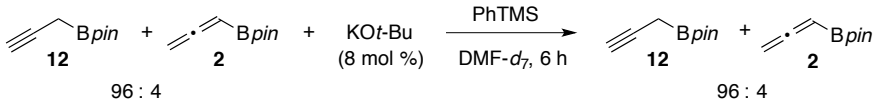


Figure SI-1. Isomerization of propargyl borolane **12** to allenyl borolane **2** in the presence of excess Grignard reagent.

2) CONTROL REACTIONS WITH **12**

I) ISOMERIZATION IN PRESENCE OF 8 MOL % BASE



In a glovebox, a flame-dried vial was charged with potassium *tert*-butoxide (1.7 mg, 0.015 mmol, 0.076 equiv). *N*,*N*-Dimethylformamide-d_7 (+0.05% V/V TMS, 0.6 mL) was added from an ampule opened in the glovebox and the solution was transferred into an oven-dried NMR tube. The NMR tube was capped with a rubber septum, sealed with parafilm, and removed from the glovebox. Phenyltrimethylsilane (PhTMS, internal standard) (17.2 μL, 0.100 mmol, 0.500 equiv) was added via syringe to the NMR tube through the septum, and the NMR tube was inverted to mix. An initial ^{1}H NMR spectrum was collected of the solution, after which propargyl borolane **12** (72 μL, 0.40 mmol, 2.0 equiv) was added via syringe to the NMR tube through the septum, and the NMR tube was inverted to mix. A ^{1}H NMR spectrum was collected (1 minute after adding **12**, Figure SI-2a), followed by sequential ^{1}H NMR spectra collected at the time points listed below:

Time elapsed	Ratio 12 : 2
1 min	96:4
4 min	96:4
7 min	96:4
9 min	96:4
10 min	96:4
15 min	96:4
20 min	96:4
30 min	96:4
40 min	96:4
1 h	96:4
3 h	96:4
6 h	96:4

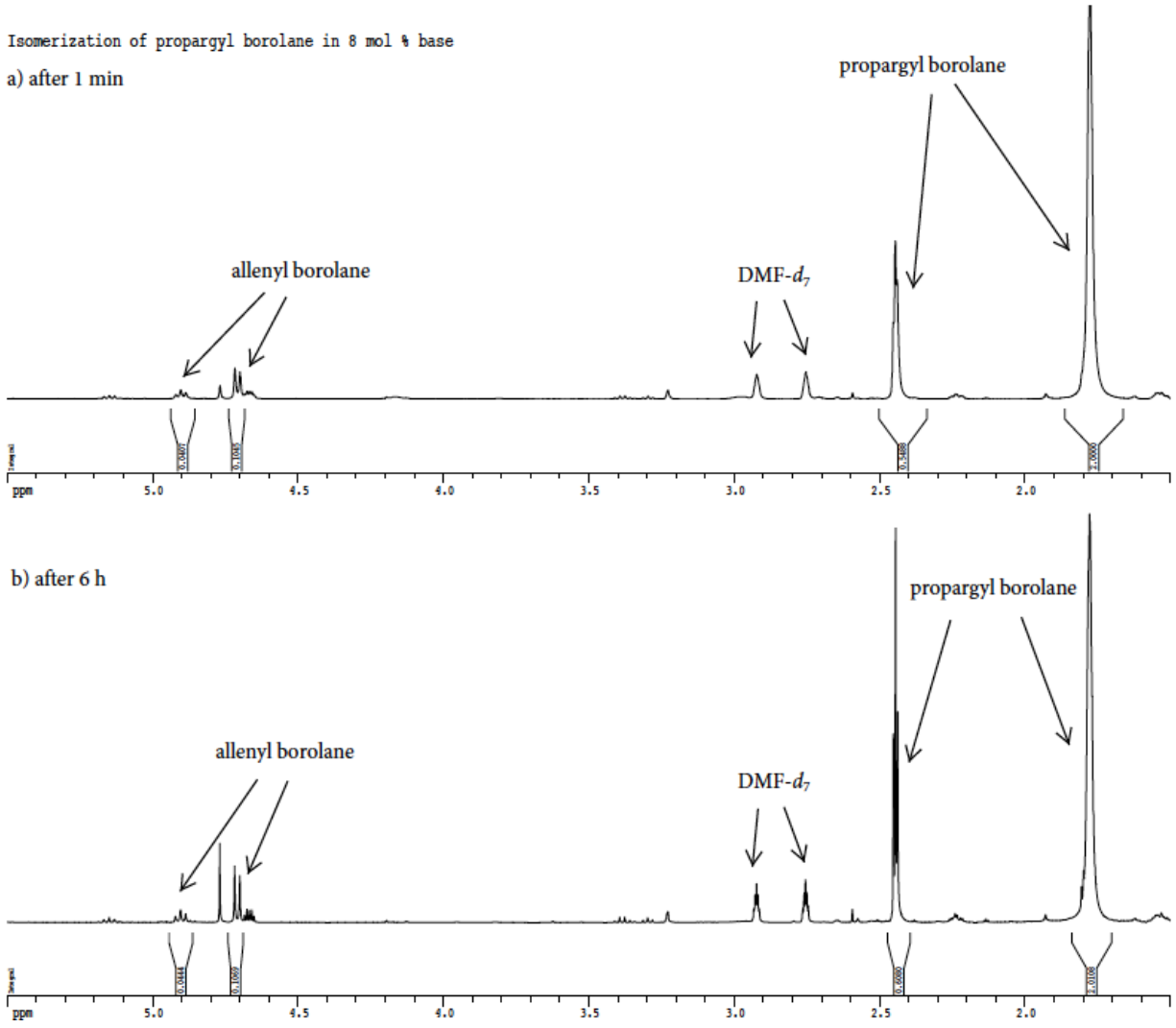
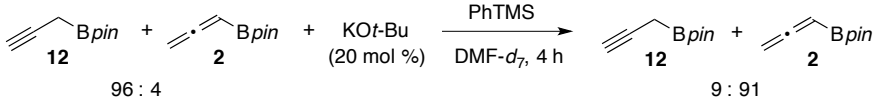


Figure SI-2. Absence of isomerization of propargyl borolane **12** in the presence of 8 mol % KO*t*-Bu at RT.

II) ISOMERIZATION IN PRESENCE OF 20 MOL % BASE



In a glovebox, a flame-dried vial was charged with potassium *tert*-butoxide (4.5 mg, 0.040 mmol, 0.20 equiv). *N*,*N*-Dimethylformamide-d_7 (+0.05% V/V TMS, 0.6 mL) was added from an ampule opened in the glovebox and the solution was transferred into an oven-dried NMR tube. The NMR tube was capped with a rubber septum, sealed with parafilm, and removed from the glovebox. Phenyltrimethylsilane (PhTMS, internal standard) (17.2 μL, 0.100 mmol, 0.500 equiv) was added via syringe to the NMR tube through the septum, and the NMR tube was inverted to mix. An initial ^{1}H NMR spectrum was collected, after which propargyl borolane **12** (72 μL, 0.40 mmol, 2.0 equiv) was added via syringe to the NMR tube through the septum, and the NMR tube was inverted to mix. A ^{1}H NMR spectrum was collected (1 minute after adding **12**, Figure SI-3a), followed by sequential ^{1}H NMR spectra collected at the time points listed below:

Time elapsed	Ratio 12 : 2
1 min	92:8
5 min	87:13
8 min	84:16
10 min	82:18
20 min	69:31
30 min	64:36
35 min	60:40
40 min	57:43
1 h	46:54
2 h	27:73
3 h	18:82
4 h	9:91

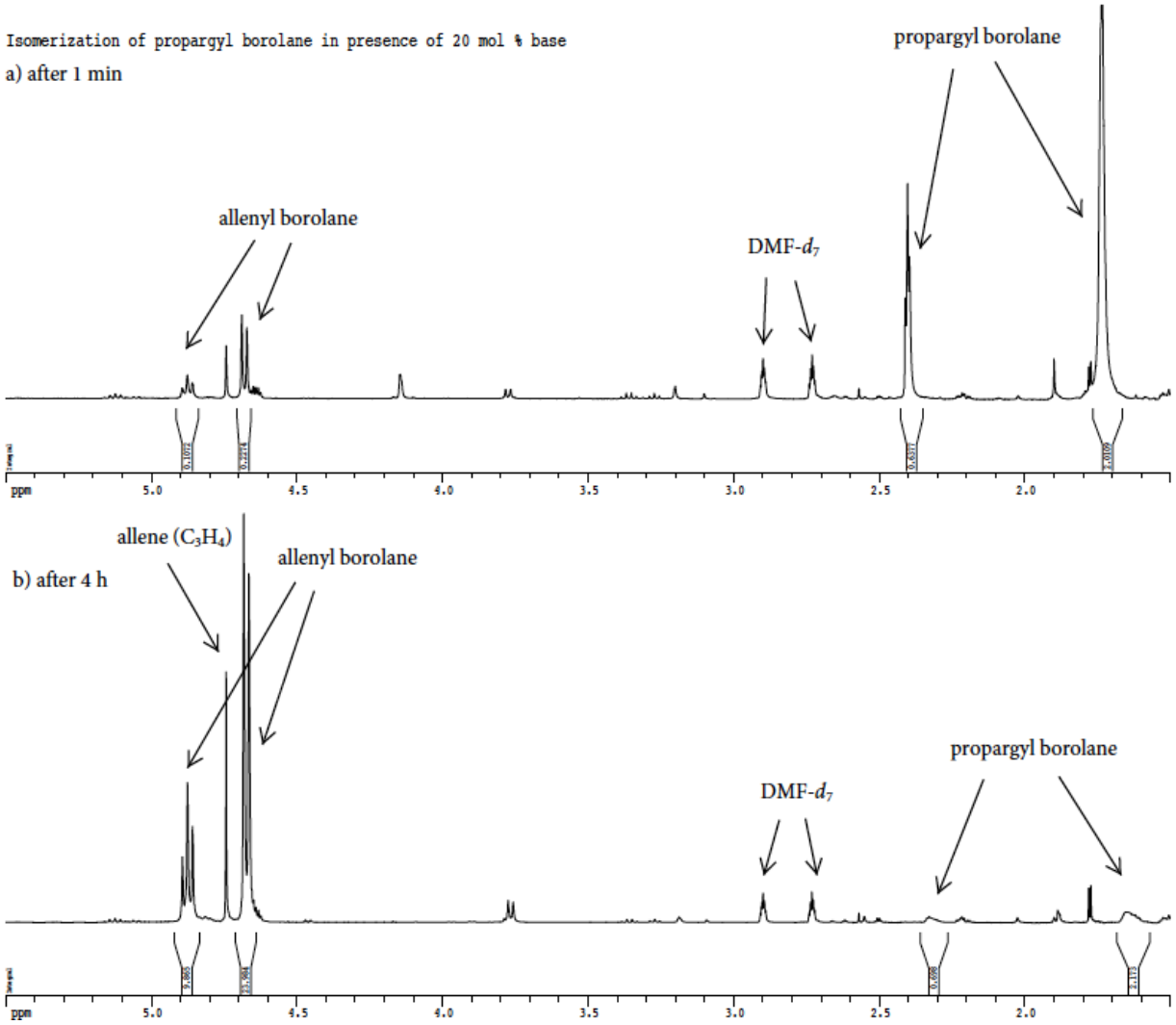


Figure SI-3. Isomerization of propargyl borolane **12** to allenyl borolane **2** in the presence of 20 mol % KO*t*-Bu at RT.

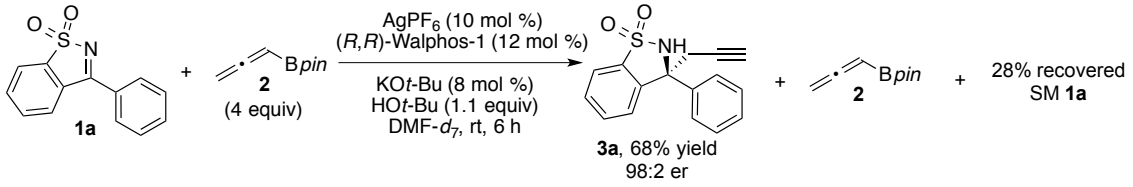
3) MECHANISTIC STUDIES WITH BOROLANE REAGENTS

We were interested in distinguishing between two of the most likely mechanisms for this propargylation reaction: transmetallation of the silver catalyst with the borolane reagent (Mechanism A), or Lewis acid catalysis (Mechanism B). To distinguish between these mechanisms, we examined reactions employing propargyl borolane reagent **12** while lowering the base loading to 8 mol %. This experimental modification was performed to minimize isomerization of **12** to allenyl borolane **2** (see Section 2-G-II, vide supra). We also performed the reactions in deuterated solvent in order to determine the ratio of **12** to **2** by ^{1}H NMR immediately after the reaction.

A control reaction using allenyl borolane **2** (Table 2, entry 1) demonstrated that under these conditions, alkyne **3a** was formed in 68% yield with 28% recovered starting material **1a** (vide infra). Using propargyl borolane **12** in the reaction (Table 2, entry 2) yielded alkyne **3a** in 64% yield with 32% recovered starting material **1a** (vide infra). This product distribution is most consistent with Mechanism A.

Note: All manipulations involving silver-catalyzed reactions were performed in the absence of direct light, using vials and NMR tubes wrapped in aluminum foil.

I) REACTION USING ALLENYL BOROLANE **2** (TABLE 2, ENTRY 1)



In a glovebox, an oven-dried 1.0 mL conical vial equipped with a triangular stir bar was charged with $AgPF_6$ (5.0 mg, 0.020 mmol, 0.10 equiv) and Walphos W001-1 (22.3 mg, 0.0240 mmol, 0.102 equiv). The vial was sealed with a screw-top cap fit with a septum and DMF-d_7 (+0.05% V/V TMS, 400 μL) was added from an ampule opened in the glovebox. The vial was removed from the glovebox and the solution was stirred for 5 min at rt. The N_2 line was then removed and the solution was stirred for 30 min at 70 °C, then cooled to rt over 15 min.

To the catalyst solution was added *tert*-butanol (21 μL, 0.22 mmol, 1.1 equiv), followed by potassium *tert*-butoxide (1.7 mg, 0.015 mmol, 0.076 equiv) and phenyl ketimine **1a** (48.6 mg, 0.200 mmol, 1.00 equiv) under a flow of N_2. The reaction was stirred at rt for 5 min to dissolve the ketimine. Allenylboronic acid pinacol ester **2** (72 μL, 0.40 mmol, 2.0 equiv) was added via syringe, followed by another portion of allenylboronic acid pincol ester (72 μL, 0.40 mmol, 2.0 equiv) added via slow addition over 3 h using a syringe pump. The N_2 line was removed and the reaction was stirred at 22 °C for another 3 h. The reaction mixture in DMF-d_7 was transferred to an NMR tube and the ratio of allenyl borolane **2** to propargyl borolane **12** was determined to be 94:6 by ^{1}H NMR (Figure SI-4). The mixture was then filtered through a plug of silica gel eluting with 100% Et_2O to remove the catalyst. Et_2O was removed in vacuo and the resulting residue

was purified by silica gel chromatography using 0–1% TEA/benzene to separate product from unreacted starting material. The mixture was purified again by flash column chromatography using 5–10–20% EtOAc/hexanes (1% TEA) to separate product from excess ligand and afford alkyne **3a** as a white solid (38.5 mg, 0.136 mmol, 68%, 98:2 er). Analytical data are consistent with the values listed in Section II-C (vide supra). **SFC** analysis (OD-H, 10% IPA, 3.0 mL/min, 215 nm) indicated 98:2 er: t_R (minor) = 11.8 min, t_R (major) = 13.7 min.

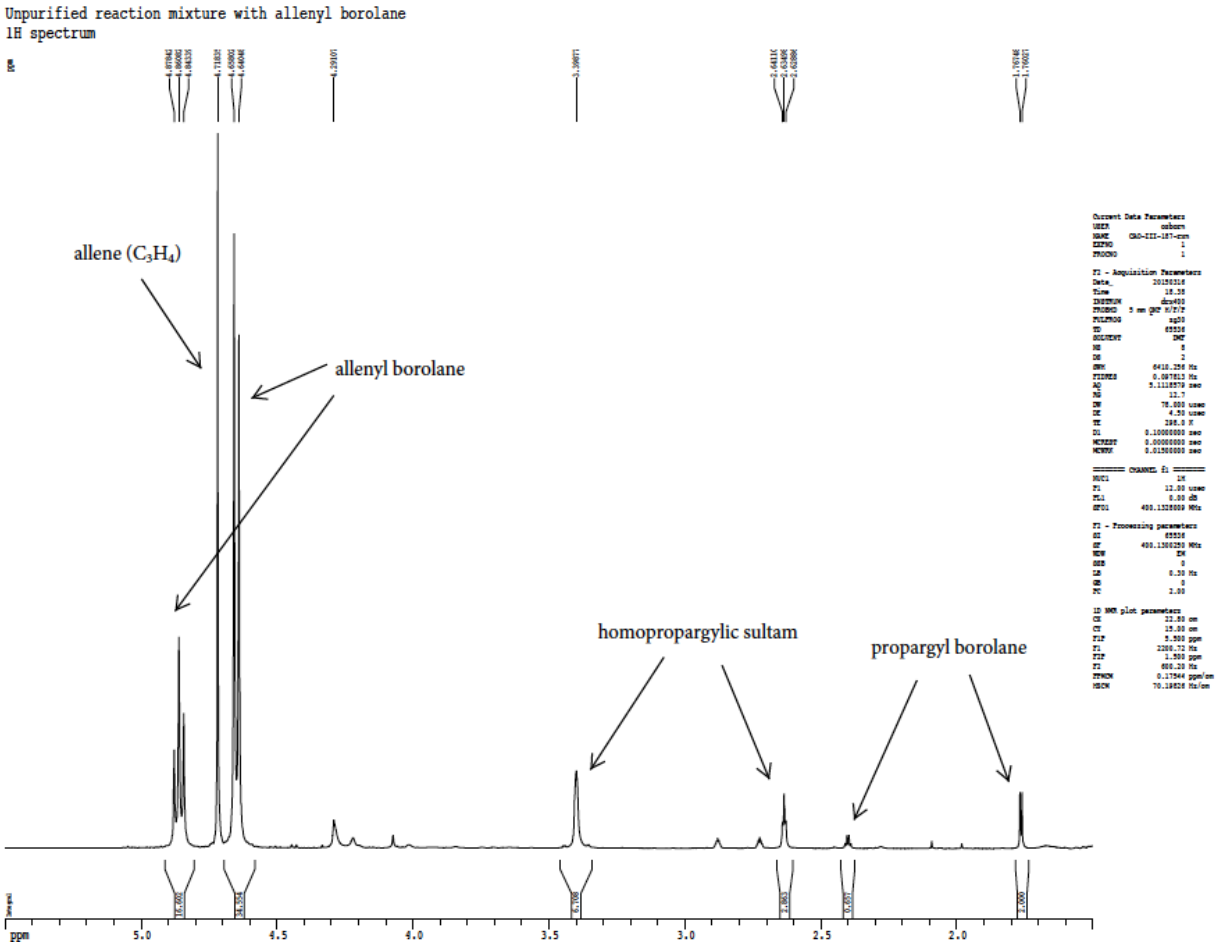
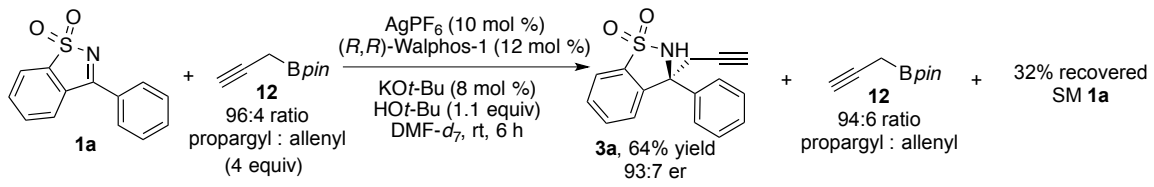


Figure SI-4. Unpurified reaction mixture in DMF-d_7.

II) REACTION USING PROPARGYL BOROLANE **12** (TABLE 2, ENTRY 2)



In a glovebox, an oven-dried 1.0 mL conical vial equipped with a triangular stir bar was charged with $AgPF_6$ (5.0 mg, 0.020 mmol, 0.10 equiv) and Walphos W001-1 (22.3 mg, 0.0240 mmol, 0.120 equiv). The vial was sealed with a screw-top cap fit with a septum and DMF-d_7 (+0.05% V/V TMS, 400 μL) was added from an ampule opened in the glovebox. The vial was removed from the glovebox and the solution was stirred for 5 min at rt. The N_2 line was then removed and the solution was stirred for 30 min at 70 °C, then cooled to rt over 15 min.

To the catalyst solution was added *tert*-butanol (21 μL, 0.22 mmol, 1.1 equiv), followed by potassium *tert*-butoxide (1.7 mg, 0.015 mmol, 0.076 equiv) and phenyl ketimine **1a** (48.6 mg, 0.200 mmol, 1.00 equiv) under a flow of N_2. The reaction was stirred at rt for 5 min to dissolve the ketimine. Propargylboronic acid pinacol ester **12** (72 μL, 0.40 mmol, 2.0 equiv) was added via syringe, followed by another portion of propargylboronic acid pincol ester (72 μL, 0.40 mmol, 2.0 equiv) added via slow addition over 3 h using a syringe pump. The N_2 line was removed and the reaction was stirred at 22 °C for another 3 h. The reaction mixture in DMF-d_7 was transferred to an NMR tube and the ratio of propargyl borolane **12** to allenyl borolane **2** was determined to be 94:6 by ^{1}H NMR (Figure SI-5). The mixture was then filtered through a plug of silica gel eluting with 100% Et_2O to remove the catalyst. Et_2O was removed in vacuo and the resulting residue was purified by silica gel chromatography using 0–1% TEA/benzene to separate product from unreacted starting material. The mixture was purified again by flash column chromatography using 5–10–20% EtOAc/hexanes (1% TEA) to separate product from excess ligand and afford alkyne **3a** as a white solid (36.5 mg, 0.129 mmol, 64%, 93:7 er). Analytical data are consistent with the values listed in Section II-C (vide supra). **SFC** analysis (OD-H, 10% IPA, 3.0 mL/min, 215 nm) indicated 93:7 er: t_R (minor) = 11.8 min, t_R (major) = 13.7 min.

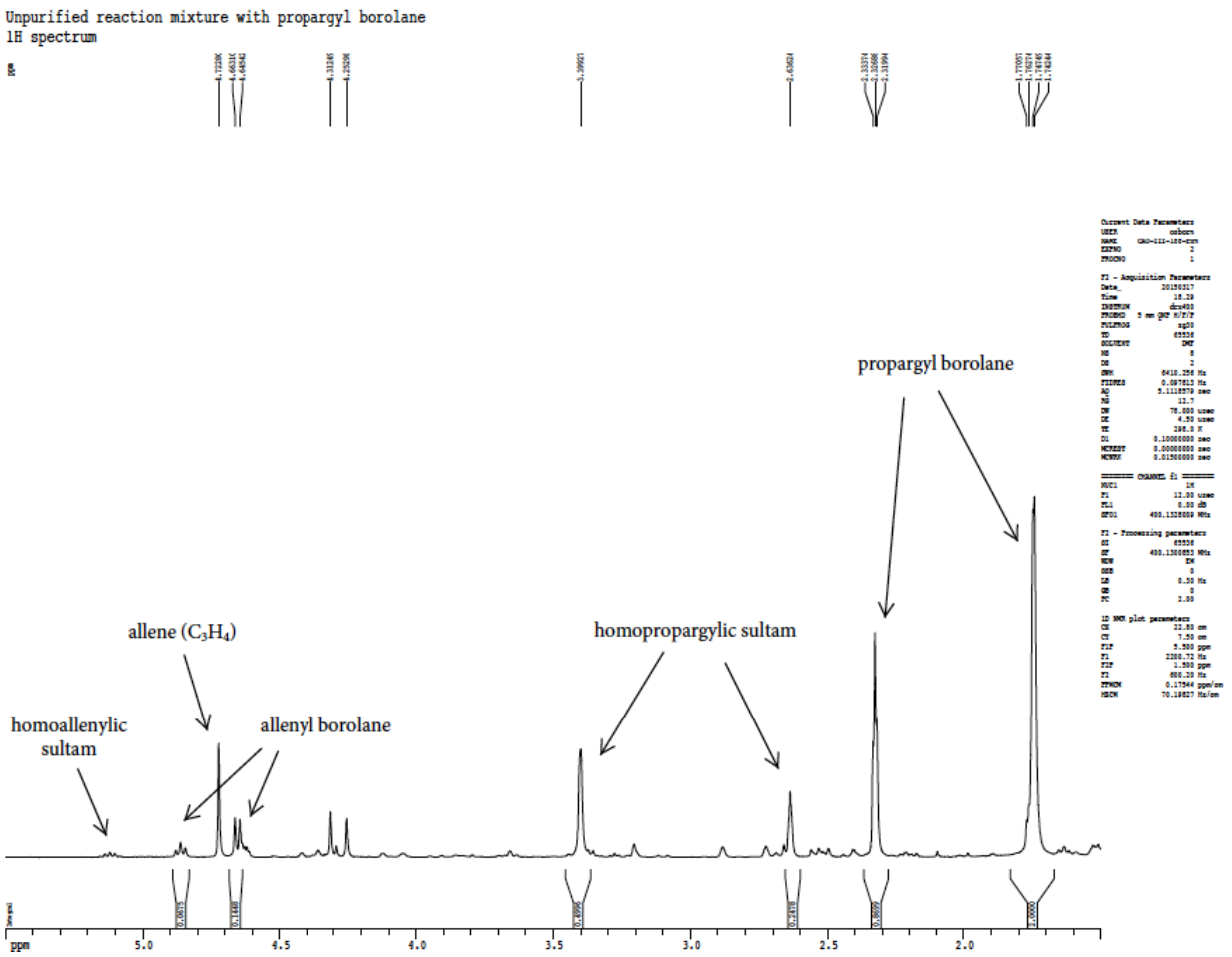


Figure SI-5. Unpurified reaction mixture in DMF-d_7.

III. REFERENCES

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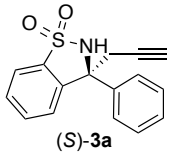
IV. CRYSTALLOGRAPHIC DATA

A. X-ray Data Collection, Structure Solution and Refinement for (*S*)-3a:

CCDC 1405841



(*S*)-**3a**

A single crystal was grown from Et_2O with slow diffusion of pentanes at room temperature. A colorless crystal of approximate dimensions 0.250 x 0.196 x 0.182 mm was mounted on a glass fiber and transferred to a Bruker SMART APEX II diffractometer. The APEX2[1] program package was used to determine the unit-cell parameters and for data collection (60 sec/frame scan time for a sphere of diffraction data). The raw frame data was processed using SAINT[2] and SADABS[3] to yield the reflection data file. Subsequent calculations were carried out using the SHELXTL[4] program. The diffraction symmetry was 4/*m* and the systematic absences were consistent with the tetragonal space group $P4_3$ that was later determined to be correct.

The structure was solved by direct methods and refined on F^2 by full-matrix least-squares techniques. The analytical scattering factors[5] 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 = 0.0661 and Goof = 1.065 for 233 variables refined against 3225 data (0.74 Å), R1 = 0.0269 for those 3084 data with $I > 2.0\sigma(I)$. The absolute structure was assigned by refinement of the Flack parameter[6].

References.

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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.

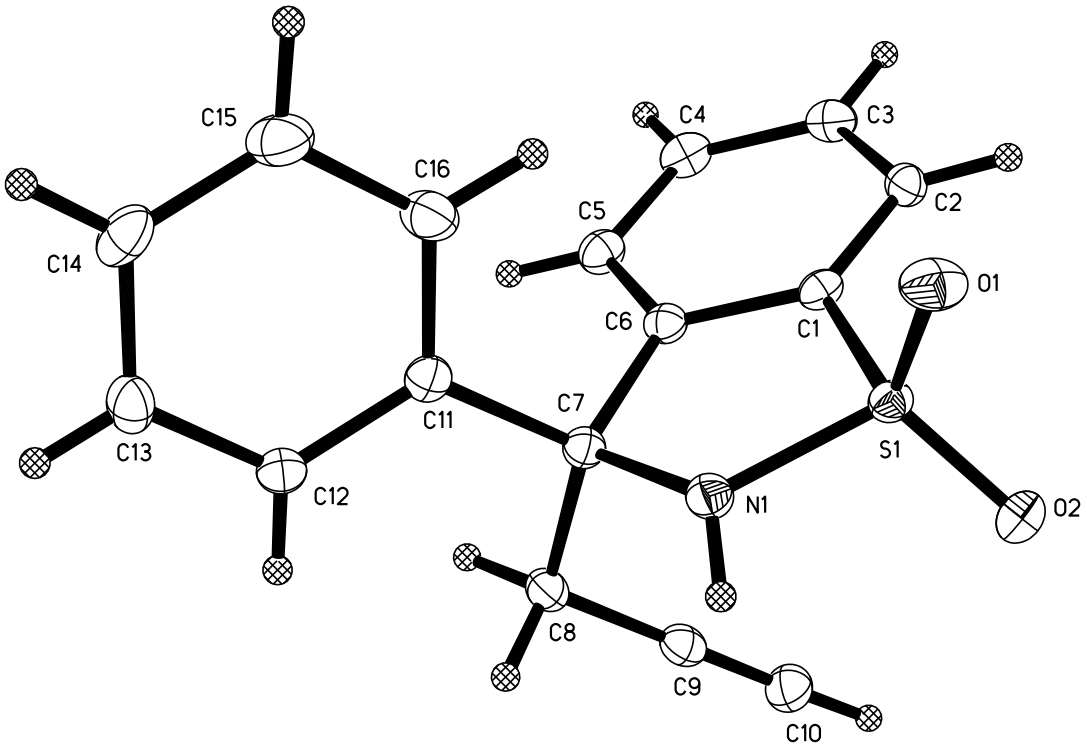

C15
C16
C14
C4
C3
C5
C2
O1
C6
C1
C11
C7
S1
C13
C12
N1
O2
C8
C9
C10

Table 1. Crystal data and structure refinement for erj21.

Identification code	erj21 (Thomas Endean)	
Empirical formula	$C_{16}H_{13}NO_2S$	
Formula weight	283.33	
Temperature	133(2) K	
Wavelength	0.71073 Å	
Crystal system	Tetragonal	
Space group	$P4_3$	
Unit cell dimensions	a = 10.7582(6) Å	a= 90°.
	b = 10.7582(6) Å	b= 90°.
	c = 11.4283(7) Å	g = 90°.
Volume	1322.70(17) $Å^3$	
Z	4	
Density (calculated)	1.423 Mg/m^3	
Absorption coefficient	0.245 mm^{-1}	
F(000)	592	
Crystal color	colorless	
Crystal size	0.250 x 0.196 x 0.182 mm^3	
Theta range for data collection	1.893 to 28.656°	
Index ranges	$-13 \le h \le 13$, $-14 \le k \le 14$, $-14 \le l \le 15$	
Reflections collected	15344	
Independent reflections	3225 [R(int) = 0.0270]	
Completeness to theta = 25.500°	100.0 %	
Absorption correction	Numerical	
Max. and min. transmission	1.0000 and 0.9257	
Refinement method	Full-matrix least-squares on F^2	
Data / restraints / parameters	3225 / 1 / 233	
Goodness-of-fit on F^2	1.065	
Final R indices [I>2sigma(I) = 3084 data]	R1 = 0.0269, wR2 = 0.0645	
R indices (all data, 0.74 Å)	R1 = 0.0295, wR2 = 0.0661	

Absolute structure parameter	-0.04(3)
Largest diff. peak and hole	0.306 and -0.185 e.Å^{-3}

Table 2. Atomic coordinates (x 10^4) and equivalent isotropic displacement parameters ($Å^2$x 10^3) for erj21. U(eq) is defined as one third of the trace of the orthogonalized U^{ij} tensor.

	x	y	z	U(eq)
S(1)	7902(1)	4294(1)	1443(1)	13(1)
O(1)	7584(2)	5501(1)	978(1)	21(1)
O(2)	8458(2)	4312(1)	2587(1)	20(1)
N(1)	6713(2)	3349(2)	1387(2)	15(1)
C(1)	8771(2)	3469(2)	396(2)	13(1)
C(2)	9948(2)	3779(2)	-26(2)	15(1)
C(3)	10465(2)	3013(2)	-878(2)	18(1)
C(4)	9807(2)	1980(2)	-1283(2)	18(1)
C(5)	8630(2)	1691(2)	-859(2)	17(1)
C(6)	8104(2)	2453(2)	-4(2)	14(1)
C(7)	6834(2)	2280(2)	570(2)	13(1)
C(8)	6822(2)	1046(2)	1277(2)	16(1)
C(9)	7837(2)	986(2)	2134(2)	16(1)
C(10)	8653(2)	950(2)	2833(2)	20(1)
C(11)	5777(2)	2333(2)	-340(2)	14(1)
C(12)	4743(2)	1553(2)	-280(2)	16(1)
C(13)	3790(2)	1650(2)	-1105(2)	20(1)
C(14)	3859(2)	2518(2)	-2002(2)	22(1)
C(15)	4885(2)	3302(2)	-2060(2)	24(1)
C(16)	5830(2)	3214(2)	-1232(2)	21(1)

Table 3. Bond lengths [Å] and angles [°] for erj21.

S(1)-O(2)	1.4373(16)
S(1)-O(1)	1.4446(15)
S(1)-N(1)	1.6351(17)
S(1)-C(1)	1.758(2)
N(1)-C(7)	1.487(3)
N(1)-H(1)	0.81(3)
C(1)-C(6)	1.386(3)
C(1)-C(2)	1.395(3)
C(2)-C(3)	1.392(3)
C(2)-H(2)	0.91(3)
C(3)-C(4)	1.396(3)
C(3)-H(3)	0.98(3)
C(4)-C(5)	1.391(3)
C(4)-H(4)	0.94(3)
C(5)-C(6)	1.395(3)
C(5)-H(5)	0.90(3)
C(6)-C(7)	1.527(3)
C(7)-C(11)	1.542(3)
C(7)-C(8)	1.554(3)
C(8)-C(9)	1.469(3)
C(8)-H(8A)	0.94(2)
C(8)-H(8B)	1.01(3)
C(9)-C(10)	1.187(3)
C(10)-H(10)	0.93(4)
C(11)-C(16)	1.393(3)
C(11)-C(12)	1.395(3)
C(12)-C(13)	1.397(3)
C(12)-H(12)	0.89(3)

C(13)-C(14)	1.388(3)
C(13)-H(13)	0.87(3)
C(14)-C(15)	1.391(3)
C(14)-H(14)	0.90(3)
C(15)-C(16)	1.392(3)
C(15)-H(15)	0.92(4)
C(16)-H(16)	0.95(3)

O(2)-S(1)-O(1)	114.86(10)
O(2)-S(1)-N(1)	111.71(10)
O(1)-S(1)-N(1)	111.03(10)
O(2)-S(1)-C(1)	113.88(9)
O(1)-S(1)-C(1)	109.23(9)
N(1)-S(1)-C(1)	94.34(9)
C(7)-N(1)-S(1)	115.92(13)
C(7)-N(1)-H(1)	116.9(19)
S(1)-N(1)-H(1)	112(2)
C(6)-C(1)-C(2)	122.99(19)
C(6)-C(1)-S(1)	110.32(15)
C(2)-C(1)-S(1)	126.66(16)
C(3)-C(2)-C(1)	117.60(19)
C(3)-C(2)-H(2)	118.0(16)
C(1)-C(2)-H(2)	124.4(16)
C(2)-C(3)-C(4)	120.00(19)
C(2)-C(3)-H(3)	119.5(16)
C(4)-C(3)-H(3)	120.5(16)
C(5)-C(4)-C(3)	121.6(2)
C(5)-C(4)-H(4)	119.2(19)
C(3)-C(4)-H(4)	119.2(19)
C(4)-C(5)-C(6)	118.9(2)

C(4)-C(5)-H(5)	121(2)
C(6)-C(5)-H(5)	120(2)
C(1)-C(6)-C(5)	118.95(18)
C(1)-C(6)-C(7)	114.70(17)
C(5)-C(6)-C(7)	126.34(18)
N(1)-C(7)-C(6)	104.68(15)
N(1)-C(7)-C(11)	109.29(16)
C(6)-C(7)-C(11)	111.43(16)
N(1)-C(7)-C(8)	109.52(16)
C(6)-C(7)-C(8)	109.54(16)
C(11)-C(7)-C(8)	112.11(16)
C(9)-C(8)-C(7)	112.23(17)
C(9)-C(8)-H(8A)	106.2(15)
C(7)-C(8)-H(8A)	111.0(14)
C(9)-C(8)-H(8B)	111.6(14)
C(7)-C(8)-H(8B)	109.0(14)
H(8A)-C(8)-H(8B)	106.7(19)
C(10)-C(9)-C(8)	179.2(2)
C(9)-C(10)-H(10)	178(2)
C(16)-C(11)-C(12)	118.56(19)
C(16)-C(11)-C(7)	119.24(18)
C(12)-C(11)-C(7)	122.16(19)
C(11)-C(12)-C(13)	120.5(2)
C(11)-C(12)-H(12)	121.3(17)
C(13)-C(12)-H(12)	118.2(17)
C(14)-C(13)-C(12)	120.6(2)
C(14)-C(13)-H(13)	119(2)
C(12)-C(13)-H(13)	120(2)
C(13)-C(14)-C(15)	119.1(2)
C(13)-C(14)-H(14)	121.6(18)

C(15)-C(14)-H(14)	119.3(18)
C(14)-C(15)-C(16)	120.4(2)
C(14)-C(15)-H(15)	124(2)
C(16)-C(15)-H(15)	116(2)
C(15)-C(16)-C(11)	120.9(2)
C(15)-C(16)-H(16)	120.1(16)
C(11)-C(16)-H(16)	119.0(16)

Table 4. Anisotropic displacement parameters ($Å^2 x\ 10^3$) for erj21. The anisotropic displacement factor exponent takes the form: $-2p^2[\ h^2\ a^{*2}U^{11} + ...\ + 2\ h\ k\ a^*\ b^*\ U^{12}\ ]$

	U^{11}	U^{22}	U^{33}	U^{23}	U^{13}	U^{12}
S(1)	16(1)	12(1)	12(1)	0(1)	0(1)	1(1)
O(1)	30(1)	14(1)	20(1)	2(1)	4(1)	5(1)
O(2)	22(1)	22(1)	14(1)	-2(1)	-3(1)	-2(1)
N(1)	15(1)	16(1)	14(1)	-2(1)	3(1)	-1(1)
C(1)	15(1)	14(1)	11(1)	0(1)	-1(1)	3(1)
C(2)	14(1)	16(1)	16(1)	2(1)	-3(1)	-1(1)
C(3)	12(1)	23(1)	18(1)	4(1)	1(1)	3(1)
C(4)	19(1)	20(1)	16(1)	0(1)	1(1)	6(1)
C(5)	20(1)	16(1)	15(1)	-1(1)	0(1)	2(1)
C(6)	13(1)	14(1)	13(1)	2(1)	-1(1)	1(1)
C(7)	13(1)	15(1)	13(1)	-1(1)	1(1)	0(1)
C(8)	15(1)	15(1)	17(1)	2(1)	0(1)	-2(1)
C(9)	18(1)	13(1)	18(1)	1(1)	2(1)	-1(1)
C(10)	22(1)	20(1)	19(1)	2(1)	-2(1)	-1(1)
C(11)	13(1)	17(1)	13(1)	-2(1)	2(1)	2(1)
C(12)	17(1)	18(1)	14(1)	0(1)	2(1)	0(1)
C(13)	13(1)	25(1)	20(1)	-4(1)	1(1)	-1(1)
C(14)	16(1)	32(1)	16(1)	-2(1)	-3(1)	6(1)
C(15)	22(1)	30(1)	21(1)	9(1)	0(1)	3(1)
C(16)	17(1)	24(1)	22(1)	6(1)	1(1)	-1(1)

Table 5. Hydrogen coordinates (x 10^4) and isotropic displacement parameters ($Å^2$x 10^3) for erj21.

	x	y	z	U(eq)
H(1)	6440(30)	3200(20)	2030(30)	20(7)
H(2)	10390(20)	4460(20)	210(20)	14(6)
H(3)	11290(30)	3210(20)	-1190(30)	22(7)
H(4)	10170(30)	1470(30)	-1850(30)	34(8)
H(5)	8220(30)	1010(30)	-1120(30)	34(8)
H(8A)	6080(20)	960(20)	1700(20)	11(6)
H(8B)	6850(20)	320(20)	710(20)	13(6)
H(10)	9310(30)	910(30)	3360(30)	46(9)
H(13)	3150(30)	1150(30)	-1080(30)	24(7)
H(14)	3250(30)	2590(30)	-2540(30)	23(7)
H(15)	5000(30)	3890(30)	-2640(30)	42(9)
H(16)	6510(30)	3780(30)	-1250(20)	22(7)
H(12)	4680(20)	970(30)	270(30)	20(6)

Table 6. Torsion angles [°] for erj21.

O(2)-S(1)-N(1)-C(7)	-117.33(15)
O(1)-S(1)-N(1)-C(7)	113.04(15)
C(1)-S(1)-N(1)-C(7)	0.52(16)
O(2)-S(1)-C(1)-C(6)	116.77(15)
O(1)-S(1)-C(1)-C(6)	-113.34(15)
N(1)-S(1)-C(1)-C(6)	0.71(16)
O(2)-S(1)-C(1)-C(2)	-65.2(2)
O(1)-S(1)-C(1)-C(2)	64.6(2)
N(1)-S(1)-C(1)-C(2)	178.70(19)
C(6)-C(1)-C(2)-C(3)	-0.8(3)
S(1)-C(1)-C(2)-C(3)	-178.56(16)
C(1)-C(2)-C(3)-C(4)	0.3(3)
C(2)-C(3)-C(4)-C(5)	0.2(3)
C(3)-C(4)-C(5)-C(6)	-0.3(3)
C(2)-C(1)-C(6)-C(5)	0.8(3)
S(1)-C(1)-C(6)-C(5)	178.84(15)
C(2)-C(1)-C(6)-C(7)	-179.80(18)
S(1)-C(1)-C(6)-C(7)	-1.7(2)
C(4)-C(5)-C(6)-C(1)	-0.2(3)
C(4)-C(5)-C(6)-C(7)	-179.56(19)
S(1)-N(1)-C(7)-C(6)	-1.5(2)
S(1)-N(1)-C(7)-C(11)	-120.91(16)
S(1)-N(1)-C(7)-C(8)	115.92(16)
C(1)-C(6)-C(7)-N(1)	2.0(2)
C(5)-C(6)-C(7)-N(1)	-178.63(19)
C(1)-C(6)-C(7)-C(11)	120.00(19)
C(5)-C(6)-C(7)-C(11)	-60.6(3)
C(1)-C(6)-C(7)-C(8)	-115.36(19)

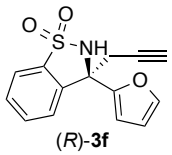
C(5)-C(6)-C(7)-C(8)	64.0(3)
N(1)-C(7)-C(8)-C(9)	-59.2(2)
C(6)-C(7)-C(8)-C(9)	55.0(2)
C(11)-C(7)-C(8)-C(9)	179.28(17)
N(1)-C(7)-C(11)-C(16)	74.1(2)
C(6)-C(7)-C(11)-C(16)	-41.1(2)
C(8)-C(7)-C(11)-C(16)	-164.25(19)
N(1)-C(7)-C(11)-C(12)	-103.7(2)
C(6)-C(7)-C(11)-C(12)	141.10(19)
C(8)-C(7)-C(11)-C(12)	17.9(3)
C(16)-C(11)-C(12)-C(13)	0.6(3)
C(7)-C(11)-C(12)-C(13)	178.41(19)
C(11)-C(12)-C(13)-C(14)	0.4(3)
C(12)-C(13)-C(14)-C(15)	-0.8(3)
C(13)-C(14)-C(15)-C(16)	0.2(3)
C(14)-C(15)-C(16)-C(11)	0.8(4)
C(12)-C(11)-C(16)-C(15)	-1.2(3)
C(7)-C(11)-C(16)-C(15)	-179.1(2)

Table 7. Hydrogen bonds for erj21 [Å and °].

D-H...A	d(D-H)	d(H...A)	d(D...A)	<(DHA)

B. X-ray Data Collection, Structure Solution and Refinement for (*R*)-3f:

CCDC 1405894



(*R*)-**3f**

A single crystal was grown from EtOAc with slow diffusion of pentanes at room temperature. A colorless crystal of approximate dimensions 0.202 x 0.333 x 0.426 mm was mounted on a glass fiber and transferred to a Bruker SMART APEX II diffractometer. The APEX2[1] program package was used to determine the unit-cell parameters and for data collection (15 sec/frame scan time for a sphere of diffraction data). The raw frame data was processed using SAINT[2] and SADABS[3] to yield the reflection data file. Subsequent calculations were carried out using the SHELXTL[4] program. The diffraction symmetry was *mmm* and the systematic absences were consistent with the orthorhombic space group $P2_12_12_1$ that was later determined to be correct.

The structure was solved by direct methods and refined on F^2 by full-matrix least-squares techniques. The analytical scattering factors[5] for neutral atoms were used throughout the analysis. Hydrogen atoms H(1) and H(10) were located from a difference-Fourier map and refined (x,y,z and U_{iso}). The remaining hydrogen atoms were included using a riding model. O(3) and C(12) were disordered and included using partial site-occupancy-factors. The disorder was included to account for the approximate distribution of carbon (50%) and oxygen (50%) over the two sites.

At convergence, wR2 = 0.0909 and Goof = 1.070 for 180 variables refined against 3100 data (0.74 Å), R1 = 0.0363 for those 2870 data with $I > 2.0\sigma(I)$. The absolute structure was assigned by refinement of the Flack parameter[6].

References.

1. APEX2 Version 2014.11-0, Bruker AXS, Inc.; Madison, WI 2014.

2. SAINT Version 8.34a, Bruker AXS, Inc.; Madison, WI 2013.

3. Sheldrick, G. M. SADABS, Version 2014/5, Bruker AXS, Inc.; Madison, WI 2014.

4. Sheldrick, G. M. SHELXTL, Version 2014/7, Bruker AXS, Inc.; Madison, WI 2014.

5. International Tables for Crystallography 1992, Vol. C., Dordrecht: Kluwer Academic Publishers.

6. Parsons, S., Flack, H. D., Wagner, T. Acta Cryst. B69, 249-259, 2013.

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 30% probability level.

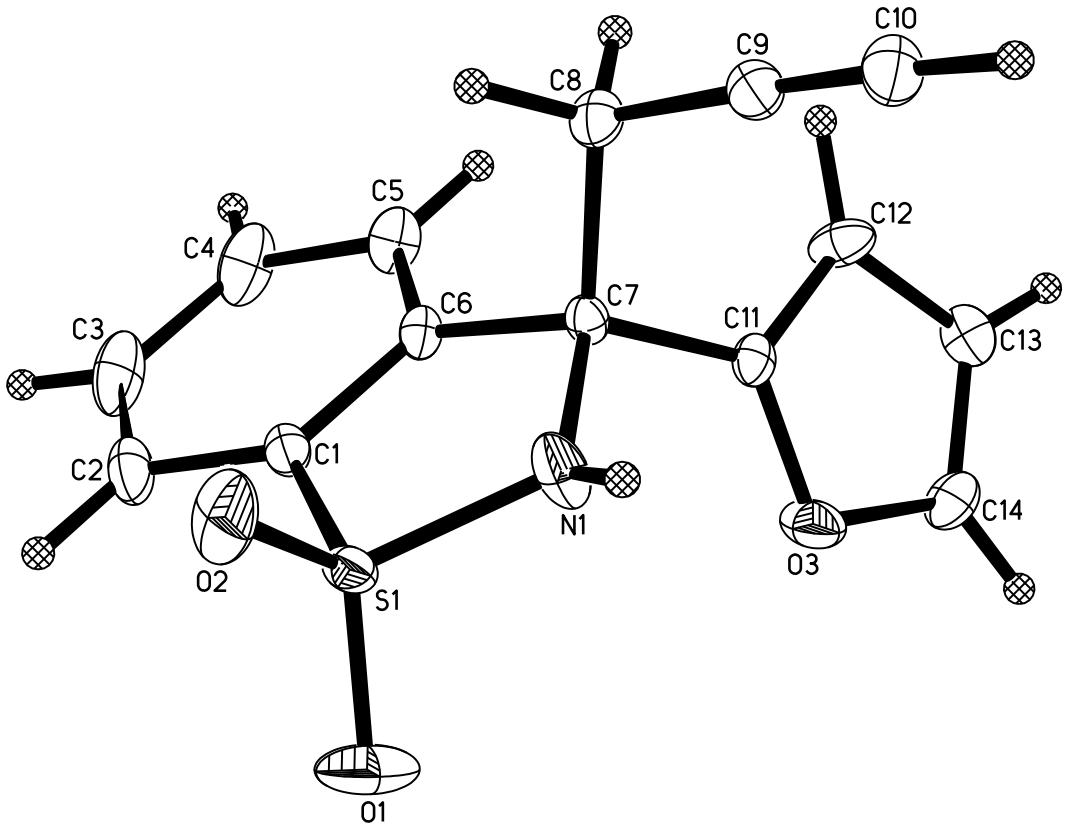

C10
C9
C8
C5
C12
C4
C6
C7
C11
C3
C13
C1
C2
N1
C14
O2
O3
S1
O1

Table 1. Crystal data and structure refinement for erj23.

Identification code	erj23 (Charlotte Osborne)	
Empirical formula	$C_{14}\ H_{11}\ N\ O_3\ S$	
Formula weight	273.30	
Temperature	133(2) K	
Wavelength	0.71073 Å	
Crystal system	Orthorhombic	
Space group	$P2_12_12_1$	
Unit cell dimensions	a = 7.5472(5) Å	a= 90°.
	b = 10.3052(7) Å	b= 90°.
	c = 16.2327(10) Å	g = 90°.
Volume	1262.51(14) $Å^3$	
Z	4	
Density (calculated)	1.438 Mg/m^3	
Absorption coefficient	0.259 mm^{-1}	
F(000)	568	
Crystal color	colorless	
Crystal size	0.426 x 0.333 x 0.202 mm^3	
Theta range for data collection	2.341 to 28.839°	
Index ranges	$-10 \le h \le 10$, $-13 \le k \le 13$, $-21 \le l \le 20$	
Reflections collected	15206	
Independent reflections	3100 [R(int) = 0.0274]	
Completeness to theta = 25.500°	99.9 %	
Absorption correction	Semi-empirical from equivalents	
Max. and min. transmission	0.8621 and 0.8165	
Refinement method	Full-matrix least-squares on F^2	
Data / restraints / parameters	3100 / 0 / 180	
Goodness-of-fit on F^2	1.070	
Final R indices [I>2sigma(I) = 2870 data]	R1 = 0.0363, wR2 = 0.0881	
R indices (all data, 0.74Å)	R1 = 0.0403, wR2 = 0.0909	

Absolute structure parameter	0.04(2)
Largest diff. peak and hole	0.298 and -0.366 e.Å^{-3}

Table 2. Atomic coordinates (x 10^4) and equivalent isotropic displacement parameters ($Å^2$x 10^3) for erj23. U(eq) is defined as one third of the trace of the orthogonalized U^{ij} tensor.

	x	y	z	U(eq)
S(1)	3053(1)	7602(1)	8953(1)	25(1)
N(1)	4347(4)	6393(2)	9154(2)	36(1)
O(1)	1714(3)	7709(3)	9570(1)	58(1)
O(2)	4026(4)	8766(2)	8781(1)	48(1)
O(3)	3159(3)	3886(2)	9539(2)	38(1)
C(1)	2206(3)	6903(2)	8057(2)	24(1)
C(2)	897(3)	7416(3)	7551(2)	37(1)
C(3)	430(4)	6713(3)	6868(2)	47(1)
C(4)	1240(4)	5532(3)	6696(2)	46(1)
C(5)	2551(4)	5035(3)	7203(2)	33(1)
C(6)	3040(3)	5741(2)	7897(1)	22(1)
C(7)	4499(3)	5402(2)	8508(1)	20(1)
C(8)	6309(3)	5461(3)	8061(2)	28(1)
C(9)	7799(3)	5308(2)	8617(2)	28(1)
C(10)	8994(4)	5223(3)	9089(2)	34(1)
C(11)	4214(3)	4100(2)	8891(2)	22(1)
C(12)	4765(3)	2981(2)	8580(1)	32(1)
C(13)	4119(3)	1987(3)	9100(2)	31(1)
C(14)	3160(4)	2544(3)	9680(2)	33(1)

Table 3. Bond lengths [Å] and angles [°] for erj23.

S(1)-O(1)	1.426(2)
S(1)-O(2)	1.435(2)
S(1)-N(1)	1.616(2)
S(1)-C(1)	1.745(3)
N(1)-C(7)	1.468(3)
O(3)-C(11)	1.338(3)
O(3)-C(14)	1.402(4)
C(1)-C(6)	1.378(3)
C(1)-C(2)	1.389(4)
C(2)-C(3)	1.371(5)
C(3)-C(4)	1.390(5)
C(4)-C(5)	1.385(4)
C(5)-C(6)	1.391(4)
C(6)-C(7)	1.522(3)
C(7)-C(11)	1.494(3)
C(7)-C(8)	1.548(3)
C(8)-C(9)	1.451(4)
C(9)-C(10)	1.186(4)
C(11)-C(12)	1.326(3)
C(12)-C(13)	1.414(3)
C(13)-C(14)	1.319(4)
O(1)-S(1)-O(2)	115.75(16)
O(1)-S(1)-N(1)	110.28(16)
O(2)-S(1)-N(1)	112.00(15)
O(1)-S(1)-C(1)	110.97(13)
O(2)-S(1)-C(1)	111.74(12)
N(1)-S(1)-C(1)	94.07(12)

C(7)-N(1)-S(1)	116.09(18)
C(11)-O(3)-C(14)	106.9(2)
C(6)-C(1)-C(2)	123.0(3)
C(6)-C(1)-S(1)	110.40(18)
C(2)-C(1)-S(1)	126.6(2)
C(3)-C(2)-C(1)	117.4(3)
C(2)-C(3)-C(4)	120.8(3)
C(5)-C(4)-C(3)	121.3(3)
C(4)-C(5)-C(6)	118.5(3)
C(1)-C(6)-C(5)	119.1(2)
C(1)-C(6)-C(7)	114.0(2)
C(5)-C(6)-C(7)	126.8(2)
N(1)-C(7)-C(11)	108.4(2)
N(1)-C(7)-C(6)	104.41(19)
C(11)-C(7)-C(6)	111.9(2)
N(1)-C(7)-C(8)	112.1(2)
C(11)-C(7)-C(8)	111.0(2)
C(6)-C(7)-C(8)	108.93(19)
C(9)-C(8)-C(7)	112.8(2)
C(10)-C(9)-C(8)	177.4(3)
C(12)-C(11)-O(3)	110.0(2)
C(12)-C(11)-C(7)	125.2(2)
O(3)-C(11)-C(7)	124.2(2)
C(11)-C(12)-C(13)	107.2(2)
C(14)-C(13)-C(12)	107.5(2)
C(13)-C(14)-O(3)	108.3(2)

Table 4. Anisotropic displacement parameters ($Å^2 x\ 10^3$) for erj23. The anisotropic displacement factor exponent takes the form: $-2p^2[\ h^2\ a^{*2}U^{11} + ...\ + 2\ h\ k\ a^*\ b^*\ U^{12}\ ]$

	U^{11}	U^{22}	U^{33}	U^{23}	U^{13}	U^{12}
S(1)	26(1)	19(1)	30(1)	-2(1)	8(1)	2(1)
N(1)	44(1)	26(1)	38(1)	-13(1)	-19(1)	10(1)
O(1)	38(1)	91(2)	45(1)	-20(1)	19(1)	1(1)
O(2)	74(2)	19(1)	50(1)	4(1)	-6(1)	-12(1)
O(3)	38(1)	28(1)	48(1)	4(1)	21(1)	3(1)
C(1)	19(1)	22(1)	32(1)	6(1)	2(1)	0(1)
C(2)	24(1)	35(2)	53(2)	18(1)	-2(1)	5(1)
C(3)	36(2)	52(2)	53(2)	23(2)	-24(2)	-9(1)
C(4)	50(2)	48(2)	39(2)	10(1)	-26(2)	-16(2)
C(5)	38(2)	30(1)	32(1)	2(1)	-11(1)	-7(1)
C(6)	21(1)	21(1)	25(1)	5(1)	-4(1)	-2(1)
C(7)	23(1)	18(1)	20(1)	-2(1)	-3(1)	2(1)
C(8)	24(1)	28(1)	31(1)	6(1)	0(1)	-4(1)
C(9)	25(1)	25(1)	35(1)	5(1)	3(1)	-2(1)
C(10)	24(1)	38(2)	40(2)	10(1)	-2(1)	-4(1)
C(11)	18(1)	25(1)	23(1)	4(1)	-5(1)	-2(1)
C(12)	44(1)	25(1)	27(1)	-6(1)	12(1)	-10(1)
C(13)	29(1)	22(1)	43(2)	-1(1)	-4(1)	-5(1)
C(14)	36(1)	33(1)	31(1)	9(1)	4(1)	-9(1)

Table 5. Hydrogen coordinates (x 10^4) and isotropic displacement parameters ($Å^2$x 10^3) for erj23.

	x	y	z	U(eq)
H(1)	5160(50)	6570(30)	9490(20)	44(10)
H(2B)	346	8222	7674	45
H(3A)	-457	7037	6507	57
H(4A)	887	5056	6223	55
H(5A)	3105	4230	7079	40
H(8A)	6353	4769	7639	33
H(8B)	6414	6306	7774	33
H(10)	9950(60)	5160(40)	9500(30)	69(13)
H(12A)	5464	2869	8098	38
H(13A)	4336	1084	9042	38
H(14A)	2571	2110	10117	40

Table 6. Hydrogen bonds for erj23 [Å and °].

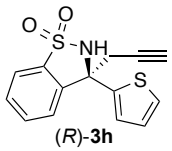
D-H...A	d(D-H)	d(H...A)	d(D...A)	<(DHA)
N(1)-H(1)...O(1)#1	0.84(4)	2.06(4)	2.888(3)	167(3)

Symmetry transformations used to generate equivalent atoms:

#1 x+1/2,-y+3/2,-z+2

C. X-ray Data Collection, Structure Solution and Refinement for (*R*)-3h:

CCDC 1405895



(*R*)-**3h**

A single crystal was grown from EtOAc with slow diffusion of pentanes at room temperature. A colorless crystal of approximate dimensions 0.573 x 0.369 x 0.266 mm was mounted on a glass fiber and transferred to a Bruker SMART APEX II diffractometer. The APEX2[1] program package was used to determine the unit-cell parameters and for data collection (5 sec/frame scan time for a sphere of diffraction data). The raw frame data was processed using SAINT[2] and SADABS[3] to yield the reflection data file. Subsequent calculations were carried out using the SHELXTL[4] program. The diffraction symmetry was *mmm* and the systematic absences were consistent with the orthorhombic space group $P2_12_12_1$ that was later determined to be correct.

The structure was solved by direct methods and refined on F^2 by full-matrix least-squares techniques. The analytical scattering factors[5] for neutral atoms were used throughout the analysis. S(2) and C(12) were disordered and included using partial site-occupancy-factors. The disorder was included to account for the approximate distribution of carbon (25%) / sulfur (75%) at the position of S(2) and carbon (75%) / sulfur (25%) at the position of C(12). H(1) and H(10) were located from a difference-Fourier map and refined (x,y,z and U_{iso}). All other hydrogen atoms were included using a riding model.

At convergence, wR2 = 0.0762 and Goof = 1.076 for 180 variables refined against 3338 data (0.73 Å), R1 = 0.0275 for those 3263 data with $I > 2.0\sigma(I)$. The absolute structure was assigned by refinement of the Flack parameter[6].

References.

1. APEX2 Version 2014.11-0, Bruker AXS, Inc.; Madison, WI 2014.

2. SAINT Version 8.34a, Bruker AXS, Inc.; Madison, WI 2013.

3. Sheldrick, G. M. SADABS, Version 2014/5, Bruker AXS, Inc.; Madison, WI 2014.

4. Sheldrick, G. M. SHELXTL, Version 2014/7, Bruker AXS, Inc.; Madison, WI 2014.

5. International Tables for Crystallography 1992, Vol. C., Dordrecht: Kluwer Academic Publishers.

6. Parsons, S., Flack, H. D., Wagner, T. Acta Cryst. B69, 249-259, 2013.

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.

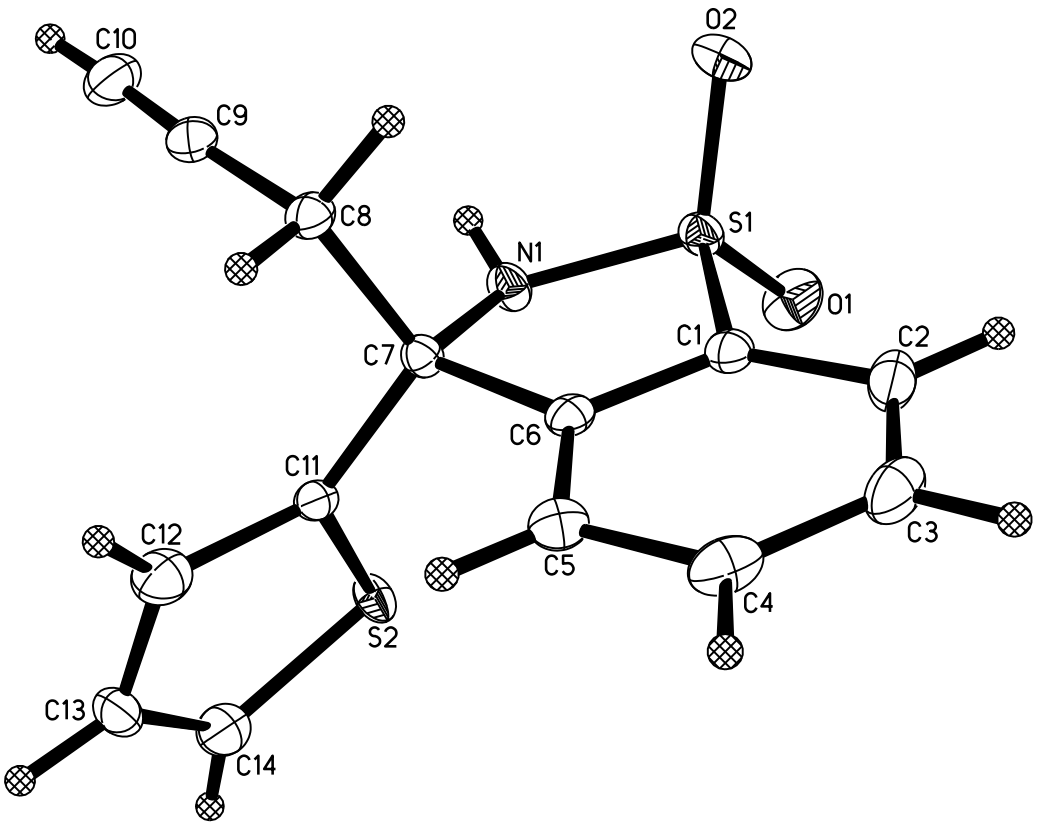

C10
C9
C8
O2
S1
N1
O1
C7
C1
C2
C6
C11
C12
C5
C3
C4
S2
C13
C14

Table 1. Crystal data and structure refinement for erj24.

Identification code	erj24 (Charlotte Osborne)	
Empirical formula	$C_{14}\ H_{11}\ N\ O_2\ S_2$	
Formula weight	289.36	
Temperature	88(2) K	
Wavelength	0.71073 Å	
Crystal system	Orthorhombic	
Space group	$P2_12_12_1$	
Unit cell dimensions	a = 7.5169(3) Å	a= 90°.
	b = 10.5263(5) Å	b= 90°.
	c = 16.5993(8) Å	g = 90°.
Volume	1313.42(10) $Å^3$	
Z	4	
Density (calculated)	1.463 Mg/m^3	
Absorption coefficient	0.401 mm^{-1}	
F(000)	600	
Crystal color	colorless	
Crystal size	0.573 x 0.369 x 0.266 mm^3	
Theta range for data collection	2.291 to 29.140°	
Index ranges	$-9 \le h \le 10$, $-14 \le k \le 14$, $-22 \le l \le 22$	
Reflections collected	16368	
Independent reflections	3338 [R(int) = 0.0221]	
Completeness to theta = 25.500°	99.9 %	
Absorption correction	Numerical	
Max. and min. transmission	0.9277 and 0.8316	
Refinement method	Full-matrix least-squares on F^2	
Data / restraints / parameters	3338 / 0 / 180	
Goodness-of-fit on F^2	1.076	
Final R indices [I>2sigma(I) = 3263 data]	R1 = 0.0275, wR2 = 0.0754	
R indices (all data, 0.73Å)	R1 = 0.0283, wR2 = 0.0762	

Absolute structure parameter	0.040(17)
Largest diff. peak and hole	0.529 and -0.343 e.Å^{-3}

Table 2. Atomic coordinates (x 10^4) and equivalent isotropic displacement parameters ($Å^2$x 10^3) for erj24. U(eq) is defined as one third of the trace of the orthogonalized U^{ij} tensor.

	x	y	z	U(eq)
S(1)	3023(1)	2396(1)	1085(1)	14(1)
S(2)	2903(1)	6156(1)	301(1)	14(1)
O(1)	1668(2)	2128(2)	496(1)	25(1)
O(2)	4157(2)	1338(1)	1302(1)	23(1)
N(1)	4166(2)	3658(2)	827(1)	15(1)
C(1)	2139(3)	3119(2)	1943(1)	14(1)
C(2)	841(3)	2609(2)	2448(1)	20(1)
C(3)	343(3)	3334(2)	3112(1)	23(1)
C(4)	1123(3)	4508(2)	3257(1)	21(1)
C(5)	2439(3)	4994(2)	2750(1)	17(1)
C(6)	2945(3)	4280(2)	2079(1)	13(1)
C(7)	4410(3)	4602(2)	1480(1)	12(1)
C(8)	6241(3)	4446(2)	1908(1)	15(1)
C(9)	7722(3)	4540(2)	1342(1)	18(1)
C(10)	8858(3)	4545(2)	845(2)	24(1)
C(11)	4195(2)	5919(2)	1128(1)	12(1)
C(12)	4882(2)	7120(2)	1456(1)	23(1)
C(13)	4238(3)	8128(2)	940(1)	21(1)
C(14)	3182(3)	7740(2)	320(1)	22(1)

Table 3. Bond lengths [Å] and angles [°] for erj24.

S(1)-O(1)	1.4387(15)
S(1)-O(2)	1.4478(16)
S(1)-N(1)	1.6393(18)
S(1)-C(1)	1.747(2)
S(2)-C(14)	1.682(2)
S(2)-C(11)	1.6990(19)
N(1)-C(7)	1.482(2)
C(1)-C(6)	1.382(3)
C(1)-C(2)	1.394(3)
C(2)-C(3)	1.392(3)
C(3)-C(4)	1.389(3)
C(4)-C(5)	1.396(3)
C(5)-C(6)	1.396(3)
C(6)-C(7)	1.522(3)
C(7)-C(11)	1.513(3)
C(7)-C(8)	1.557(3)
C(8)-C(9)	1.460(3)
C(9)-C(10)	1.187(3)
C(11)-C(12)	1.470(2)
C(12)-C(13)	1.447(3)
C(13)-C(14)	1.363(3)

O(1)-S(1)-O(2)	115.80(10)
O(1)-S(1)-N(1)	110.63(10)
O(2)-S(1)-N(1)	112.31(10)
O(1)-S(1)-C(1)	111.70(10)
O(2)-S(1)-C(1)	110.87(9)
N(1)-S(1)-C(1)	93.38(9)

C(14)-S(2)-C(11)	93.42(10)
C(7)-N(1)-S(1)	114.66(13)
C(6)-C(1)-C(2)	123.30(19)
C(6)-C(1)-S(1)	110.61(14)
C(2)-C(1)-S(1)	126.07(16)
C(3)-C(2)-C(1)	117.0(2)
C(4)-C(3)-C(2)	120.8(2)
C(3)-C(4)-C(5)	121.4(2)
C(6)-C(5)-C(4)	118.5(2)
C(1)-C(6)-C(5)	119.15(18)
C(1)-C(6)-C(7)	114.06(16)
C(5)-C(6)-C(7)	126.69(18)
N(1)-C(7)-C(11)	108.54(15)
N(1)-C(7)-C(6)	103.80(15)
C(11)-C(7)-C(6)	112.32(15)
N(1)-C(7)-C(8)	111.86(16)
C(11)-C(7)-C(8)	111.52(16)
C(6)-C(7)-C(8)	108.58(15)
C(9)-C(8)-C(7)	111.95(16)
C(10)-C(9)-C(8)	174.7(2)
C(12)-C(11)-C(7)	127.37(16)
C(12)-C(11)-S(2)	111.95(13)
C(7)-C(11)-S(2)	120.48(14)
C(13)-C(12)-C(11)	107.10(15)
C(14)-C(13)-C(12)	114.98(18)
C(13)-C(14)-S(2)	112.53(16)

Table 4. Anisotropic displacement parameters ($Å^2 x\ 10^3$) for erj24. The anisotropic displacement factor exponent takes the form: $-2p^2[\ h^2\ a^{*2}U^{11} + ...\ + 2\ h\ k\ a^*\ b^*\ U^{12}\ ]$

	U^{11}	U^{22}	U^{33}	U^{23}	U^{13}	U^{12}
S(1)	15(1)	12(1)	16(1)	-1(1)	-2(1)	0(1)
S(2)	20(1)	11(1)	12(1)	3(1)	-5(1)	-2(1)
O(1)	21(1)	31(1)	24(1)	-9(1)	-7(1)	-2(1)
O(2)	25(1)	14(1)	31(1)	2(1)	0(1)	4(1)
N(1)	18(1)	12(1)	14(1)	-2(1)	3(1)	-1(1)
C(1)	13(1)	15(1)	15(1)	2(1)	-1(1)	2(1)
C(2)	15(1)	23(1)	23(1)	7(1)	-1(1)	-4(1)
C(3)	17(1)	32(1)	21(1)	11(1)	4(1)	1(1)
C(4)	20(1)	30(1)	13(1)	3(1)	3(1)	7(1)
C(5)	17(1)	19(1)	14(1)	0(1)	0(1)	3(1)
C(6)	11(1)	15(1)	12(1)	3(1)	-2(1)	2(1)
C(7)	13(1)	11(1)	12(1)	-1(1)	1(1)	0(1)
C(8)	13(1)	16(1)	16(1)	3(1)	-1(1)	1(1)
C(9)	15(1)	17(1)	22(1)	5(1)	-5(1)	1(1)
C(10)	15(1)	27(1)	29(1)	7(1)	2(1)	2(1)
C(11)	11(1)	13(1)	13(1)	2(1)	1(1)	1(1)
C(12)	21(1)	24(1)	24(1)	7(1)	1(1)	2(1)
C(13)	22(1)	13(1)	28(1)	-2(1)	8(1)	-1(1)
C(14)	20(1)	22(1)	22(1)	10(1)	3(1)	2(1)

Table 5. Hydrogen coordinates (x 10^4) and isotropic displacement parameters ($Å^2$x 10^3) for erj24.

	x	y	z	U(eq)
H(1)	4980(40)	3440(30)	590(20)	34(9)
H(2B)	321	1803	2344	24
H(3A)	-542	3022	3470	28
H(4A)	754	4991	3711	25
H(5A)	2977	5793	2858	20
H(8A)	6368	5112	2325	18
H(8B)	6283	3610	2180	18
H(10)	9730(50)	4590(40)	510(20)	55(11)
H(12A)	5610	7216	1920	27
H(13A)	4530	8995	1027	25
H(14A)	2661	8303	-61	26

Table 6. Hydrogen bonds for erj24 [Å and °].

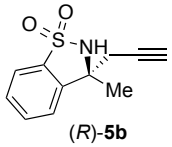
D-H...A	d(D-H)	d(H...A)	d(D...A)	<(DHA)
N(1)-H(1)...O(1)#1	0.76(3)	2.28(3)	3.008(2)	159(3)
C(8)-H(8A)...O(2)#2	0.99	2.65	3.590(3)	158.9
C(13)-H(13A)...O(2)#3	0.95	2.52	3.432(3)	160.2

Symmetry transformations used to generate equivalent atoms:

#1 x+1/2,-y+1/2,-z #2 -x+1,y+1/2,-z+1/2 #3 x,y+1,z

D. X-ray Data Collection, Structure Solution and Refinement for (*R*)-5b:

CCDC 1410049



(*R*)-**5b**

A single crystal was grown from Et_2O with slow diffusion of pentanes at room temperature. A colorless crystal of approximate dimensions 0.284 x 0.299 x 0.489 mm was mounted on a glass fiber and transferred to a Bruker SMART APEX II diffractometer. The APEX2[1] program package was used to determine the unit-cell parameters and for data collection (10 sec/frame scan time for a sphere of diffraction data). The raw frame data was processed using SAINT[2] and SADABS[3] to yield the reflection data file. Subsequent calculations were carried out using the SHELXTL[4] program. The diffraction symmetry was *mmm* and the systematic absences were consistent with the orthorhombic space group $P2_12_12_1$ that was later determined to be correct.

The structure was solved by direct methods and refined on F^2 by full-matrix least-squares techniques. The analytical scattering factors[5] 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 = 0.0679 and Goof = 1.058 for 180 variables refined against 2571 data (0.74Å), R1 = 0.0259 for those 2521 data with $I > 2.0\sigma(I)$. The absolute structure was assigned by refinement of the Flack parameter[6].

References.

1. APEX2 Version 2014.11-0, Bruker AXS, Inc.; Madison, WI 2014.

2. SAINT Version 8.34a, Bruker AXS, Inc.; Madison, WI 2013.

3. Sheldrick, G. M. SADABS, Version 2014/5, Bruker AXS, Inc.; Madison, WI 2014.

4. Sheldrick, G. M. SHELXTL, Version 2014/7, Bruker AXS, Inc.; Madison, WI 2014.

5. International Tables for Crystallography 1992, Vol. C., Dordrecht: Kluwer Academic Publishers.

6. Parsons, S., Flack, H. D., Wagner, T. Acta Cryst. B69, 249-259, 2013.

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.

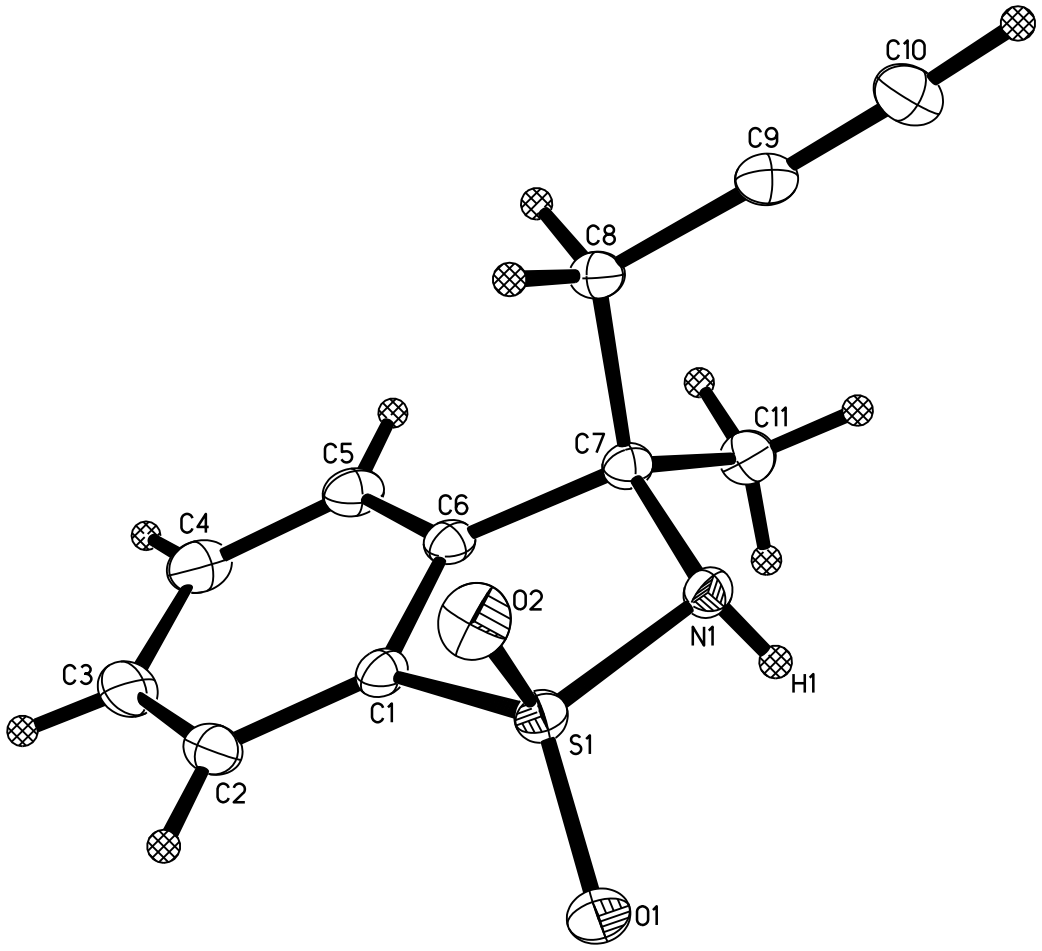

C10
C9
C8
C11
C7
C5
C6
C4
O2
N1
H1
C3
C1
S1
C2
O1

Table 1. Crystal data and structure refinement for erj26.

Identification code	erj26 (Charlotte Osborne)	
Empirical formula	C_{11} H_{11} N O_2 S	
Formula weight	221.27	
Temperature	133(2) K	
Wavelength	0.71073 Å	
Crystal system	Orthorhombic	
Space group	$P2_12_12_1$	
Unit cell dimensions	a = 8.0916(6) Å	a= 90°.
	b = 9.4218(7) Å	b= 90°.
	c = 13.8016(10) Å	g = 90°.
Volume	1052.20(13) $Å^3$	
Z	4	
Density (calculated)	1.397 Mg/m^3	
Absorption coefficient	0.285 mm^{-1}	
F(000)	464	
Crystal color	colorless	
Crystal size	0.489 x 0.299 x 0.284 mm^3	
Theta range for data collection	2.617 to 28.724°	
Index ranges	$-10 \le h \le 10$, $-12 \le k \le 12$, $-18 \le l \le 18$	
Reflections collected	12648	
Independent reflections	2571 [R(int) = 0.0277]	
Completeness to theta = 25.500°	99.9 %	
Absorption correction	Numerical	
Max. and min. transmission	1.0000 and 0.8521	
Refinement method	Full-matrix least-squares on F^2	
Data / restraints / parameters	2571 / 0 / 180	
Goodness-of-fit on F^2	1.058	
Final R indices [I>2sigma(I) = 2521 data]	R1 = 0.0259, wR2 = 0.0672	

R indices (all data, 0.74Å)	R1 = 0.0265, wR2 = 0.0679
Absolute structure parameter	0.01(3)
Largest diff. peak and hole	0.314 and -0.279 e.Å^{-3}

Table 2. Atomic coordinates (x 10^4) and equivalent isotropic displacement parameters ($Å^2$x 10^3) for erj26. U(eq) is defined as one third of the trace of the orthogonalized U^{ij} tensor.

	x	y	z	U(eq)
S(1)	995(1)	3781(1)	7431(1)	14(1)
O(1)	639(2)	4308(2)	8386(1)	21(1)
O(2)	360(2)	2389(1)	7201(1)	22(1)
N(1)	370(2)	4924(2)	6611(1)	14(1)
C(1)	3091(2)	3848(2)	7115(1)	15(1)
C(2)	4403(2)	3209(2)	7592(1)	22(1)
C(3)	5950(3)	3368(2)	7180(1)	26(1)
C(4)	6157(2)	4107(2)	6320(1)	24(1)
C(5)	4813(2)	4700(2)	5835(1)	19(1)
C(6)	3250(2)	4566(2)	6245(1)	14(1)
C(7)	1612(2)	5056(2)	5812(1)	14(1)
C(8)	1157(2)	4018(2)	4982(1)	17(1)
C(9)	-448(2)	4300(2)	4531(1)	18(1)
C(10)	-1745(2)	4511(2)	4149(1)	23(1)
C(11)	1642(3)	6587(2)	5455(1)	19(1)

Table 3. Bond lengths [Å] and angles [°] for erj26.

S(1)-O(1)	1.4377(13)
S(1)-O(2)	1.4438(14)
S(1)-N(1)	1.6418(16)
S(1)-C(1)	1.7524(17)
N(1)-C(7)	1.497(2)
N(1)-H(1)	0.77(3)
C(1)-C(6)	1.384(2)
C(1)-C(2)	1.388(3)
C(2)-C(3)	1.384(3)
C(2)-H(2)	0.93(3)
C(3)-C(4)	1.386(3)
C(3)-H(3)	0.91(3)
C(4)-C(5)	1.394(3)
C(4)-H(4)	0.95(3)
C(5)-C(6)	1.392(2)
C(5)-H(5)	0.94(3)
C(6)-C(7)	1.525(2)
C(7)-C(11)	1.525(2)
C(7)-C(8)	1.550(2)
C(8)-C(9)	1.464(3)
C(8)-H(8A)	1.01(2)
C(8)-H(8B)	0.95(2)
C(9)-C(10)	1.192(3)
C(10)-H(10)	0.92(3)
C(11)-H(11A)	0.98(3)
C(11)-H(11B)	0.93(3)
C(11)-H(11C)	0.97(3)

O(1)-S(1)-O(2)	116.34(8)
O(1)-S(1)-N(1)	110.12(8)
O(2)-S(1)-N(1)	109.58(8)
O(1)-S(1)-C(1)	114.17(8)
O(2)-S(1)-C(1)	108.82(9)
N(1)-S(1)-C(1)	95.88(8)
C(7)-N(1)-S(1)	110.82(12)
C(7)-N(1)-H(1)	113.8(19)
S(1)-N(1)-H(1)	107.4(18)
C(6)-C(1)-C(2)	123.60(16)
C(6)-C(1)-S(1)	108.88(13)
C(2)-C(1)-S(1)	127.35(14)
C(3)-C(2)-C(1)	116.72(17)
C(3)-C(2)-H(2)	120.5(15)
C(1)-C(2)-H(2)	122.6(15)
C(2)-C(3)-C(4)	121.09(18)
C(2)-C(3)-H(3)	121.4(17)
C(4)-C(3)-H(3)	117.5(17)
C(3)-C(4)-C(5)	121.24(18)
C(3)-C(4)-H(4)	120.9(17)
C(5)-C(4)-H(4)	117.9(17)
C(6)-C(5)-C(4)	118.52(17)
C(6)-C(5)-H(5)	121.0(16)
C(4)-C(5)-H(5)	120.5(16)
C(1)-C(6)-C(5)	118.79(16)
C(1)-C(6)-C(7)	114.00(15)
C(5)-C(6)-C(7)	127.10(15)
N(1)-C(7)-C(11)	109.09(14)
N(1)-C(7)-C(6)	105.65(13)
C(11)-C(7)-C(6)	113.48(15)

N(1)-C(7)-C(8)	109.46(14)
C(11)-C(7)-C(8)	111.19(14)
C(6)-C(7)-C(8)	107.79(14)
C(9)-C(8)-C(7)	114.22(15)
C(9)-C(8)-H(8A)	109.7(14)
C(7)-C(8)-H(8A)	108.6(14)
C(9)-C(8)-H(8B)	109.7(15)
C(7)-C(8)-H(8B)	109.1(14)
H(8A)-C(8)-H(8B)	105.1(19)
C(10)-C(9)-C(8)	178.6(2)
C(9)-C(10)-H(10)	177.4(18)
C(7)-C(11)-H(11A)	109.3(16)
C(7)-C(11)-H(11B)	114.0(15)
H(11A)-C(11)-H(11B)	109(2)
C(7)-C(11)-H(11C)	109.1(17)
H(11A)-C(11)-H(11C)	107(2)
H(11B)-C(11)-H(11C)	108(2)

Table 4. Anisotropic displacement parameters ($Å^2 x\ 10^3$) for erj26. The anisotropic displacement factor exponent takes the form: $-2p^2[\ h^2\ a^{*2}U^{11} + ...\ + 2\ h\ k\ a^*\ b^*\ U^{12}\ ]$

	U^{11}	U^{22}	U^{33}	U^{23}	U^{13}	U^{12}
S(1)	13(1)	16(1)	14(1)	1(1)	3(1)	0(1)
O(1)	21(1)	27(1)	14(1)	1(1)	5(1)	4(1)
O(2)	22(1)	17(1)	26(1)	3(1)	4(1)	-4(1)
N(1)	14(1)	15(1)	14(1)	0(1)	2(1)	1(1)
C(1)	12(1)	19(1)	15(1)	-4(1)	1(1)	0(1)
C(2)	20(1)	30(1)	15(1)	-1(1)	-2(1)	5(1)
C(3)	15(1)	41(1)	22(1)	-6(1)	-6(1)	6(1)
C(4)	12(1)	36(1)	23(1)	-9(1)	1(1)	-2(1)
C(5)	16(1)	25(1)	16(1)	-3(1)	3(1)	-4(1)
C(6)	14(1)	16(1)	14(1)	-4(1)	-1(1)	-2(1)
C(7)	14(1)	16(1)	12(1)	0(1)	2(1)	-1(1)
C(8)	18(1)	19(1)	14(1)	-3(1)	-1(1)	0(1)
C(9)	20(1)	20(1)	14(1)	-1(1)	1(1)	-2(1)
C(10)	21(1)	28(1)	19(1)	1(1)	-3(1)	-3(1)
C(11)	23(1)	16(1)	18(1)	2(1)	2(1)	-1(1)

Table 5. Hydrogen coordinates (x 10^4) and isotropic displacement parameters ($Å^2$x 10^3) for erj26.

	x	y	z	U(eq)
H(1)	190(30)	5630(30)	6875(19)	20(6)
H(2)	4280(30)	2750(30)	8185(18)	23(6)
H(3)	6870(30)	2970(30)	7450(20)	28(6)
H(4)	7230(30)	4240(30)	6048(19)	32(7)
H(5)	4960(30)	5170(30)	5240(20)	34(7)
H(8A)	2060(30)	4060(30)	4474(18)	21(6)
H(8B)	1180(30)	3070(20)	5224(17)	18(5)
H(10)	-2740(40)	4630(30)	3840(20)	36(7)
H(11A)	2430(30)	6670(30)	4925(19)	28(6)
H(11B)	1900(30)	7240(30)	5932(18)	23(6)
H(11C)	570(40)	6830(30)	5200(20)	36(7)

Table 6. Torsion angles [°] for erj26.

O(1)-S(1)-N(1)-C(7)	138.66(12)
O(2)-S(1)-N(1)-C(7)	-92.16(13)
C(1)-S(1)-N(1)-C(7)	20.23(13)
O(1)-S(1)-C(1)-C(6)	-126.43(12)
O(2)-S(1)-C(1)-C(6)	101.75(14)
N(1)-S(1)-C(1)-C(6)	-11.27(14)
O(1)-S(1)-C(1)-C(2)	58.3(2)
O(2)-S(1)-C(1)-C(2)	-73.50(18)
N(1)-S(1)-C(1)-C(2)	173.48(17)
C(6)-C(1)-C(2)-C(3)	2.9(3)
S(1)-C(1)-C(2)-C(3)	177.46(15)
C(1)-C(2)-C(3)-C(4)	-1.4(3)
C(2)-C(3)-C(4)-C(5)	-0.7(3)
C(3)-C(4)-C(5)-C(6)	1.6(3)
C(2)-C(1)-C(6)-C(5)	-2.1(3)
S(1)-C(1)-C(6)-C(5)	-177.56(13)
C(2)-C(1)-C(6)-C(7)	174.41(17)
S(1)-C(1)-C(6)-C(7)	-1.05(18)
C(4)-C(5)-C(6)-C(1)	-0.2(3)
C(4)-C(5)-C(6)-C(7)	-176.18(17)
S(1)-N(1)-C(7)-C(11)	-144.84(13)
S(1)-N(1)-C(7)-C(6)	-22.51(16)
S(1)-N(1)-C(7)-C(8)	93.30(15)
C(1)-C(6)-C(7)-N(1)	14.5(2)
C(5)-C(6)-C(7)-N(1)	-169.29(17)
C(1)-C(6)-C(7)-C(11)	134.01(16)
C(5)-C(6)-C(7)-C(11)	-49.8(2)
C(1)-C(6)-C(7)-C(8)	-102.40(17)

C(5)-C(6)-C(7)-C(8)	73.8(2)
N(1)-C(7)-C(8)-C(9)	61.96(19)
C(11)-C(7)-C(8)-C(9)	-58.6(2)
C(6)-C(7)-C(8)-C(9)	176.40(14)

Table 7. Hydrogen bonds for erj26 [Å and °].

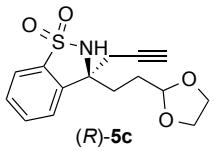
D-H...A	d(D-H)	d(H...A)	d(D...A)	<(DHA)
N(1)-H(1)...O(2)#1	0.77(3)	2.14(3)	2.903(2)	171(3)

Symmetry transformations used to generate equivalent atoms:

#1 -x,y+1/2,-z+3/2

E. X-ray Data Collection, Structure Solution and Refinement for (*R*)-5c:

CCDC 1405843



(*R*)-**5c**

A single crystal was grown from EtOAc with slow diffusion of pentanes at room temperature. A colorless crystal of approximate dimensions 0.288 x 0.160 x 0.108 mm was mounted on a glass fiber and transferred to a Bruker SMART APEX II diffractometer. The APEX2[1] program package was used to determine the unit-cell parameters and for data collection (60 sec/frame scan time for a sphere of diffraction data). The raw frame data was processed using SAINT[2] and SADABS[3] to yield the reflection data file. Subsequent calculations were carried out using the SHELXTL[4] program. The diffraction symmetry was *mmm* and the systematic absences were consistent with the orthorhombic space group $P2_12_12_1$ that was later determined to be correct.

The structure was solved by direct methods and refined on F^2 by full-matrix least-squares techniques. The analytical scattering factors[5] 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 = 0.0706 and Goof = 1.040 for 258 variables refined against 3562 data (0.75 Å), R1 = 0.0294 for those 3283 data with $I > 2.0\sigma(I)$. The absolute structure was assigned by refinement of the Flack parameter[6].

References.

1. APEX2 Version 2014.11-0, Bruker AXS, Inc.; Madison, WI 2014.

2. SAINT Version 8.34a, Bruker AXS, Inc.; Madison, WI 2013.

3. Sheldrick, G. M. SADABS, Version 2014/5, Bruker AXS, Inc.; Madison, WI 2014.

4. Sheldrick, G. M. SHELXTL, Version 2014/7, Bruker AXS, Inc.; Madison, WI 2014.

5. International Tables for Crystallography 1992, Vol. C., Dordrecht: Kluwer Academic Publishers.

6. Flack, H. D. Acta. Cryst., A39, 876-881, 1983.

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.

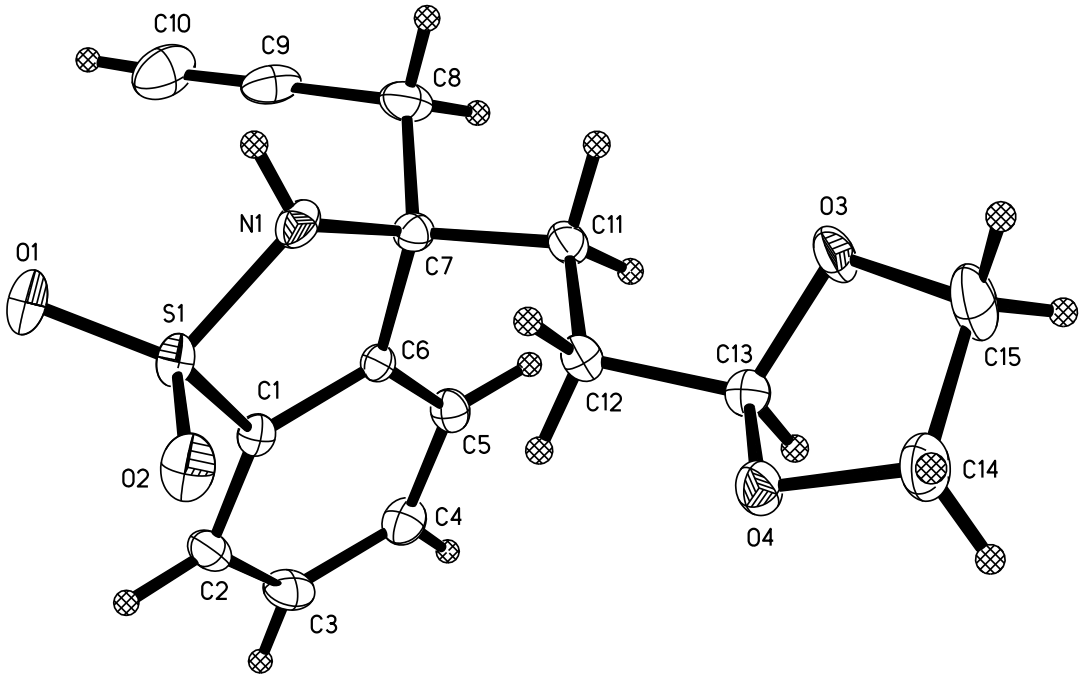

C10
C9
C8
N1
C11
O3
O1
C7
S1
C6
C13
C15
C1
C12
C5
O2
C14
C4
O4
C2
C3

Table 1. Crystal data and structure refinement for erj22.

Identification code	erj22 (Charlotte Osborne)	
Empirical formula	$C_{15}H_{17}NO_4S$	
Formula weight	307.35	
Temperature	133(2) K	
Wavelength	0.71073 Å	
Crystal system	Orthorhombic	
Space group	$P2_12_12_1$	
Unit cell dimensions	a = 7.7171(5) Å	a= 90°.
	b = 7.9486(5) Å	b= 90°.
	c = 23.8160(14) Å	g = 90°.
Volume	1460.88(16) $Å^3$	
Z	4	
Density (calculated)	1.397 Mg/m^3	
Absorption coefficient	0.237 mm^{-1}	
F(000)	648	
Crystal color	colorless	
Crystal size	0.288 x 0.160 x 0.108 mm^3	
Theta range for data collection	1.710 to 28.288°	
Index ranges	$-10 \le h \le 10$, $-10 \le k \le 10$, $-31 \le l \le 31$	
Reflections collected	17581	
Independent reflections	3562 [R(int) = 0.0306]	
Completeness to theta = 25.500°	100.0 %	
Absorption correction	Numerical	
Max. and min. transmission	0.9980 and 0.9400	
Refinement method	Full-matrix least-squares on F^2	
Data / restraints / parameters	3562 / 0 / 258	
Goodness-of-fit on F^2	1.040	
Final R indices [I>2sigma(I) = 3283 data]	R1 = 0.0294, wR2 = 0.0678	
R indices (all data, 0.75 Å)	R1 = 0.0342, wR2 = 0.0706	

Absolute structure parameter	0.07(2)
Largest diff. peak and hole	0.316 and -0.247 e.Å^{-3}

Table 2. Atomic coordinates (x 10^4) and equivalent isotropic displacement parameters ($Å^2$x 10^3) for erj22. U(eq) is defined as one third of the trace of the orthogonalized U^{ij} tensor.

	x	y	z	U(eq)
S(1)	9619(1)	2605(1)	9054(1)	17(1)
N(1)	10456(2)	3214(2)	8456(1)	18(1)
O(1)	10861(2)	1665(2)	9377(1)	25(1)
O(2)	7974(2)	1792(2)	8972(1)	25(1)
O(3)	7309(2)	5275(2)	6830(1)	22(1)
O(4)	5065(2)	4151(2)	7301(1)	20(1)
C(1)	9266(2)	4647(2)	9298(1)	14(1)
C(2)	8545(3)	5077(3)	9810(1)	19(1)
C(3)	8250(3)	6771(3)	9908(1)	22(1)
C(4)	8676(3)	7967(3)	9505(1)	21(1)
C(5)	9407(2)	7505(2)	8996(1)	17(1)
C(6)	9706(2)	5809(2)	8890(1)	13(1)
C(7)	10489(3)	5054(2)	8361(1)	15(1)
C(8)	12367(3)	5666(3)	8281(1)	23(1)
C(9)	13497(3)	5248(3)	8757(1)	24(1)
C(10)	14373(3)	4848(3)	9138(1)	34(1)
C(11)	9414(3)	5506(3)	7836(1)	16(1)
C(12)	7675(3)	4603(3)	7816(1)	16(1)
C(13)	6485(3)	5257(3)	7365(1)	16(1)
C(14)	4400(3)	4455(4)	6752(1)	30(1)
C(15)	5922(3)	5167(4)	6424(1)	30(1)

Table 3. Bond lengths [Å] and angles [°] for erj22.

S(1)-O(2)	1.4379(16)
S(1)-O(1)	1.4383(15)
S(1)-N(1)	1.6361(18)
S(1)-C(1)	1.746(2)
N(1)-C(7)	1.480(3)
O(3)-C(13)	1.425(2)
O(3)-C(15)	1.445(3)
O(4)-C(13)	1.413(2)
O(4)-C(14)	1.425(3)
C(1)-C(2)	1.383(3)
C(1)-C(6)	1.384(3)
C(2)-C(3)	1.385(3)
C(3)-C(4)	1.390(3)
C(4)-C(5)	1.387(3)
C(5)-C(6)	1.391(3)
C(6)-C(7)	1.521(3)
C(7)-C(8)	1.541(3)
C(7)-C(11)	1.543(3)
C(8)-C(9)	1.468(3)
C(9)-C(10)	1.175(3)
C(11)-C(12)	1.523(3)
C(12)-C(13)	1.505(3)
C(14)-C(15)	1.519(3)
O(2)-S(1)-O(1)	115.30(10)
O(2)-S(1)-N(1)	111.35(9)
O(1)-S(1)-N(1)	110.84(10)
O(2)-S(1)-C(1)	108.98(9)
O(1)-S(1)-C(1)	114.11(9)

N(1)-S(1)-C(1)	94.39(9)
C(7)-N(1)-S(1)	115.63(13)
C(13)-O(3)-C(15)	105.46(17)
C(13)-O(4)-C(14)	105.85(16)
C(2)-C(1)-C(6)	123.63(18)
C(2)-C(1)-S(1)	125.90(16)
C(6)-C(1)-S(1)	110.34(14)
C(1)-C(2)-C(3)	117.03(19)
C(2)-C(3)-C(4)	120.6(2)
C(5)-C(4)-C(3)	121.3(2)
C(4)-C(5)-C(6)	118.85(18)
C(1)-C(6)-C(5)	118.58(17)
C(1)-C(6)-C(7)	114.62(16)
C(5)-C(6)-C(7)	126.80(17)
N(1)-C(7)-C(6)	104.84(15)
N(1)-C(7)-C(8)	110.31(17)
C(6)-C(7)-C(8)	110.57(16)
N(1)-C(7)-C(11)	110.20(16)
C(6)-C(7)-C(11)	111.43(15)
C(8)-C(7)-C(11)	109.42(16)
C(9)-C(8)-C(7)	113.10(18)
C(10)-C(9)-C(8)	177.2(3)
C(12)-C(11)-C(7)	112.91(16)
C(13)-C(12)-C(11)	113.39(17)
O(4)-C(13)-O(3)	104.82(15)
O(4)-C(13)-C(12)	109.61(16)
O(3)-C(13)-C(12)	111.66(17)
O(4)-C(14)-C(15)	104.84(18)
O(3)-C(15)-C(14)	104.59(18)

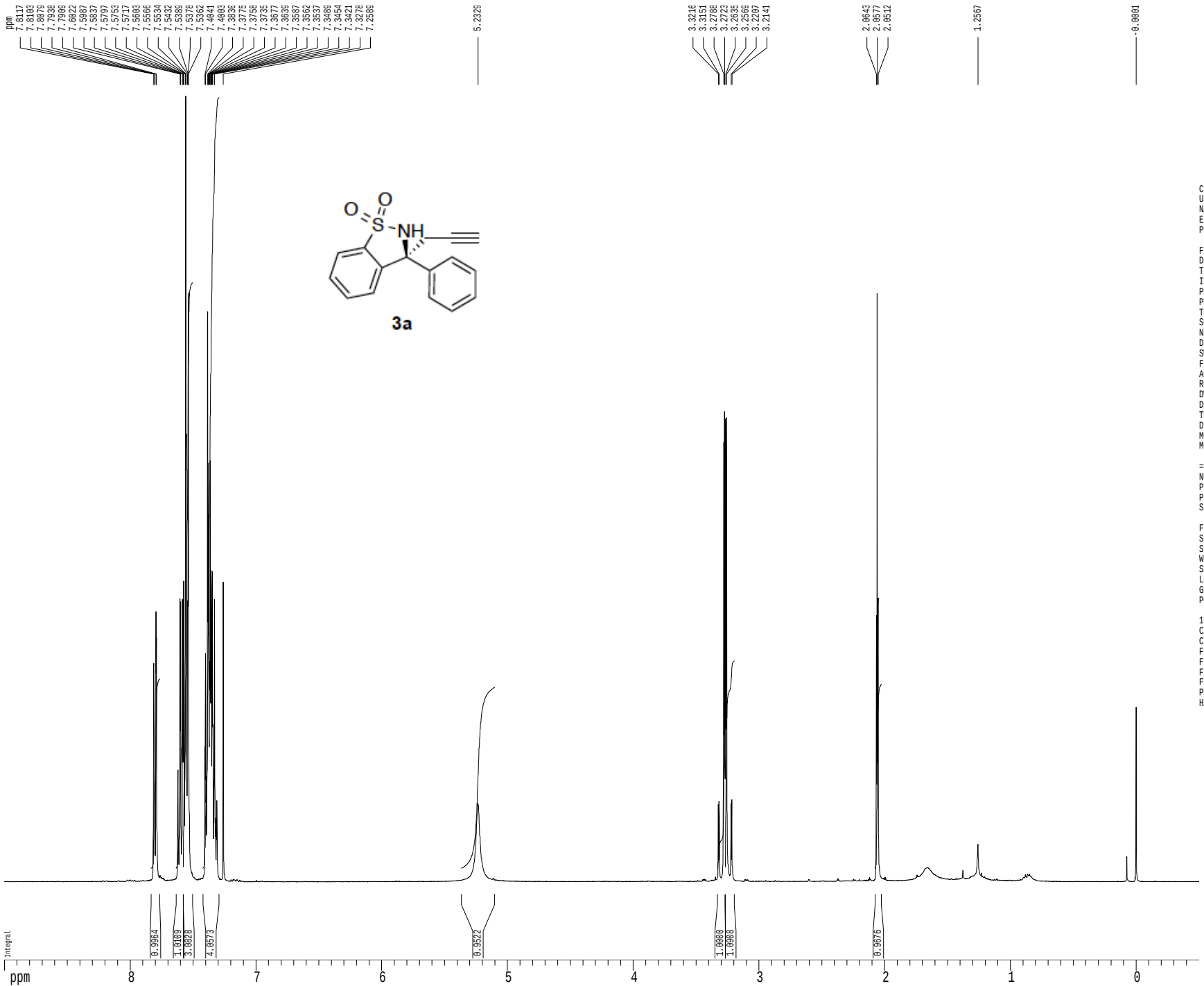
Table 4. Anisotropic displacement parameters ($Å^2 x\ 10^3$) for erj22. The anisotropic displacement factor exponent takes the form: $-2p^2[\ h^2\ a^{*2}U^{11} + ...\ + 2\ h\ k\ a^*\ b^*\ U^{12}\ ]$

	U^{11}	U^{22}	U^{33}	U^{23}	U^{13}	U^{12}
S(1)	19(1)	12(1)	18(1)	1(1)	-6(1)	0(1)
N(1)	21(1)	17(1)	18(1)	-4(1)	-2(1)	6(1)
O(1)	28(1)	20(1)	27(1)	4(1)	-9(1)	5(1)
O(2)	26(1)	18(1)	32(1)	1(1)	-9(1)	-7(1)
O(3)	22(1)	31(1)	12(1)	5(1)	-3(1)	-6(1)
O(4)	17(1)	26(1)	17(1)	4(1)	-4(1)	-5(1)
C(1)	14(1)	13(1)	16(1)	0(1)	-3(1)	-1(1)
C(2)	17(1)	24(1)	15(1)	3(1)	1(1)	-5(1)
C(3)	22(1)	28(1)	15(1)	-6(1)	4(1)	0(1)
C(4)	24(1)	18(1)	21(1)	-4(1)	-2(1)	4(1)
C(5)	18(1)	16(1)	18(1)	1(1)	-2(1)	-2(1)
C(6)	11(1)	17(1)	11(1)	1(1)	-3(1)	-1(1)
C(7)	14(1)	16(1)	14(1)	-1(1)	0(1)	1(1)
C(8)	16(1)	34(1)	18(1)	-2(1)	3(1)	-3(1)
C(9)	14(1)	31(1)	28(1)	-8(1)	3(1)	-1(1)
C(10)	23(1)	43(1)	34(1)	-10(1)	-8(1)	4(1)
C(11)	17(1)	19(1)	12(1)	1(1)	0(1)	-1(1)
C(12)	19(1)	17(1)	14(1)	2(1)	0(1)	-1(1)
C(13)	17(1)	15(1)	16(1)	0(1)	0(1)	0(1)
C(14)	24(1)	45(2)	22(1)	9(1)	-8(1)	-5(1)
C(15)	33(1)	37(1)	19(1)	9(1)	-10(1)	-12(1)

Table 5. Hydrogen coordinates (x 10^4) and isotropic displacement parameters ($Å^2$x 10^3) for erj22.

	x	y	z	U(eq)
H(1)	11280(40)	2610(40)	8381(11)	34(7)
H(2B)	8200(40)	4230(40)	10076(12)	37(8)
H(3A)	7770(30)	7090(30)	10250(10)	20(6)
H(4A)	8440(30)	9070(40)	9586(11)	28(7)
H(5A)	9700(30)	8340(30)	8715(10)	27(7)
H(8A)	12820(30)	5150(30)	7952(11)	23(6)
H(8B)	12330(30)	6880(40)	8216(10)	24(6)
H(10)	15060(40)	4580(40)	9411(13)	51(9)
H(11A)	9240(30)	6750(30)	7845(9)	21(6)
H(11B)	10110(30)	5230(30)	7509(10)	15(5)
H(12A)	7860(30)	3410(30)	7749(10)	20(6)
H(12B)	7070(30)	4720(30)	8154(11)	23(6)
H(13A)	6040(30)	6350(30)	7452(9)	12(5)
H(14A)	3410(40)	5190(40)	6780(12)	40(8)
H(14B)	4030(50)	3470(50)	6598(15)	67(11)
H(15A)	6290(40)	4470(40)	6122(14)	55(10)
H(15B)	5690(30)	6250(30)	6278(11)	27(7)

1H spectrum

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PL1                0.00 dB
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1D NMR plot parameters
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CY                15.00 cm
F1P               9.000 ppm
F1              3601.17 Hz
F2P              -0.500 ppm
F2              -200.06 Hz
PPMCM           0.41667 ppm/cm
HZCM          166.72086 Hz/cm
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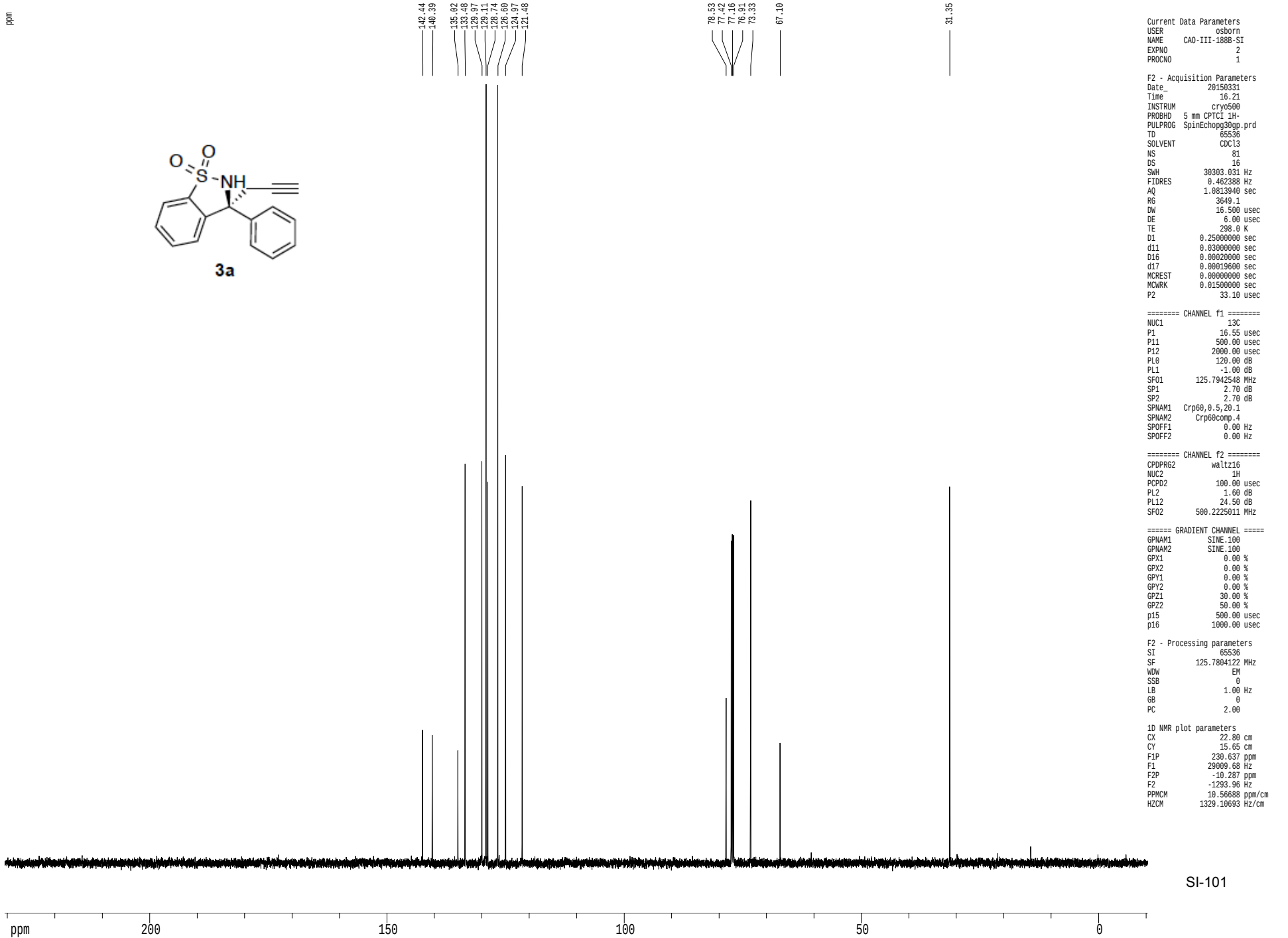
Z-restored spin-echo 13C spectrum with 1H decoupling

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PL1                  -1.00 dB
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SP1                   2.70 dB
SP2                   2.70 dB
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SPNAM2       Crp60comp.4
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SPOFF2                0.00 Hz

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PCPD2              100.00 usec
PL2                  1.60 dB
PL12                24.50 dB
SFO2          500.2225011 MHz

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GPNAM2           SINE.100
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GPX2                  0.00 %
GPY1                  0.00 %
GPY2                  0.00 %
GPZ1                 30.00 %
GPZ2                 50.00 %
p15                 500.00 usec
p16                1000.00 usec

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F1               29009.68 Hz
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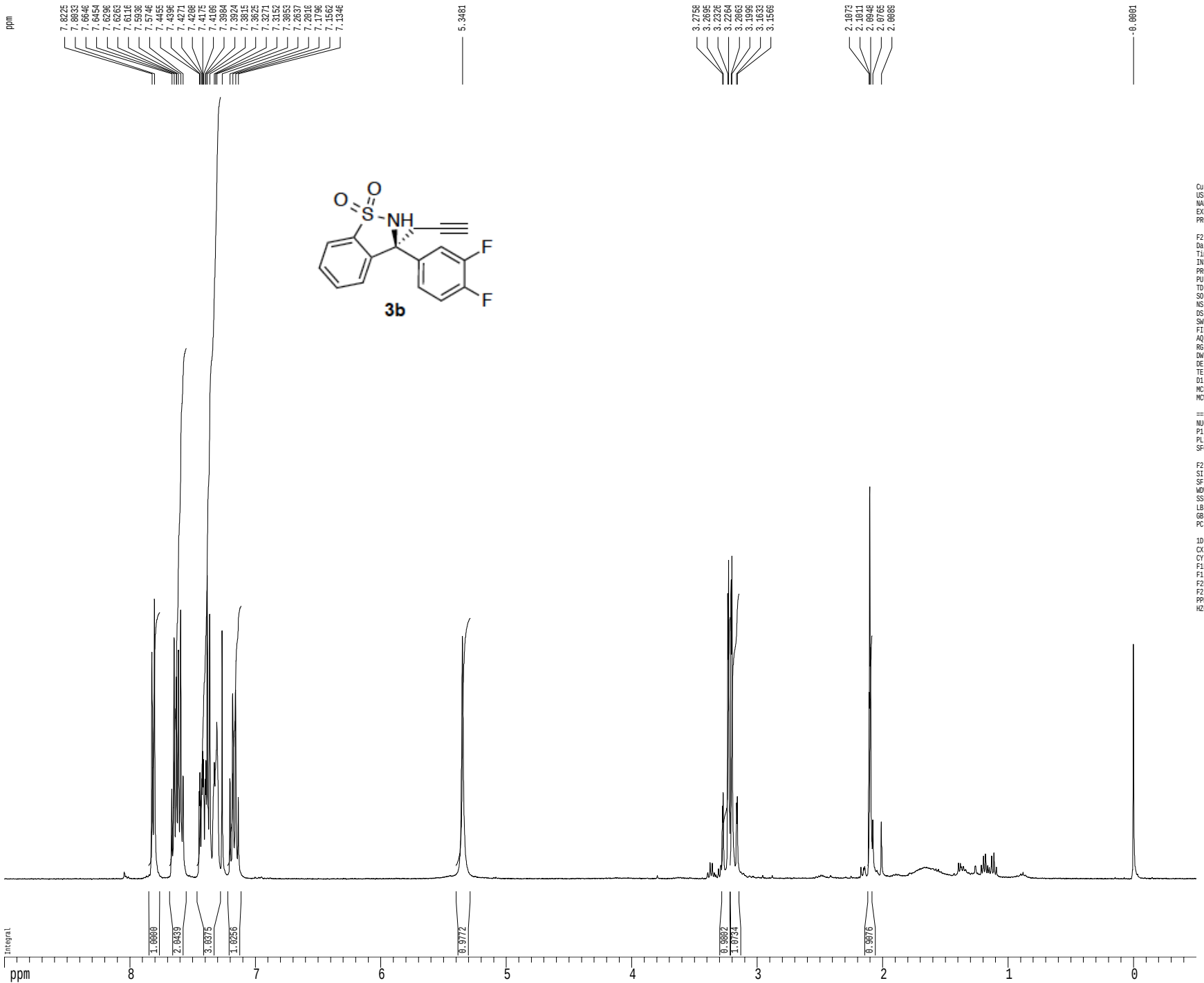
1H spectrum

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DS                     2
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TE                 298.0 K
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MCREST        0.00000000 sec
MCWRK         0.01500000 sec

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PL1                 0.00 dB
SFO1         400.1328009 MHz

F2 - Processing parameters
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SF           400.1300191 MHz
WDW                   EM
SSB                    0
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1D NMR plot parameters
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CY                  7.50 cm
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F1               3601.17 Hz
F2P               -0.500 ppm
F2               -200.06 Hz
PPMCM           0.41667 ppm/cm
HZCM          166.72084 Hz/cm
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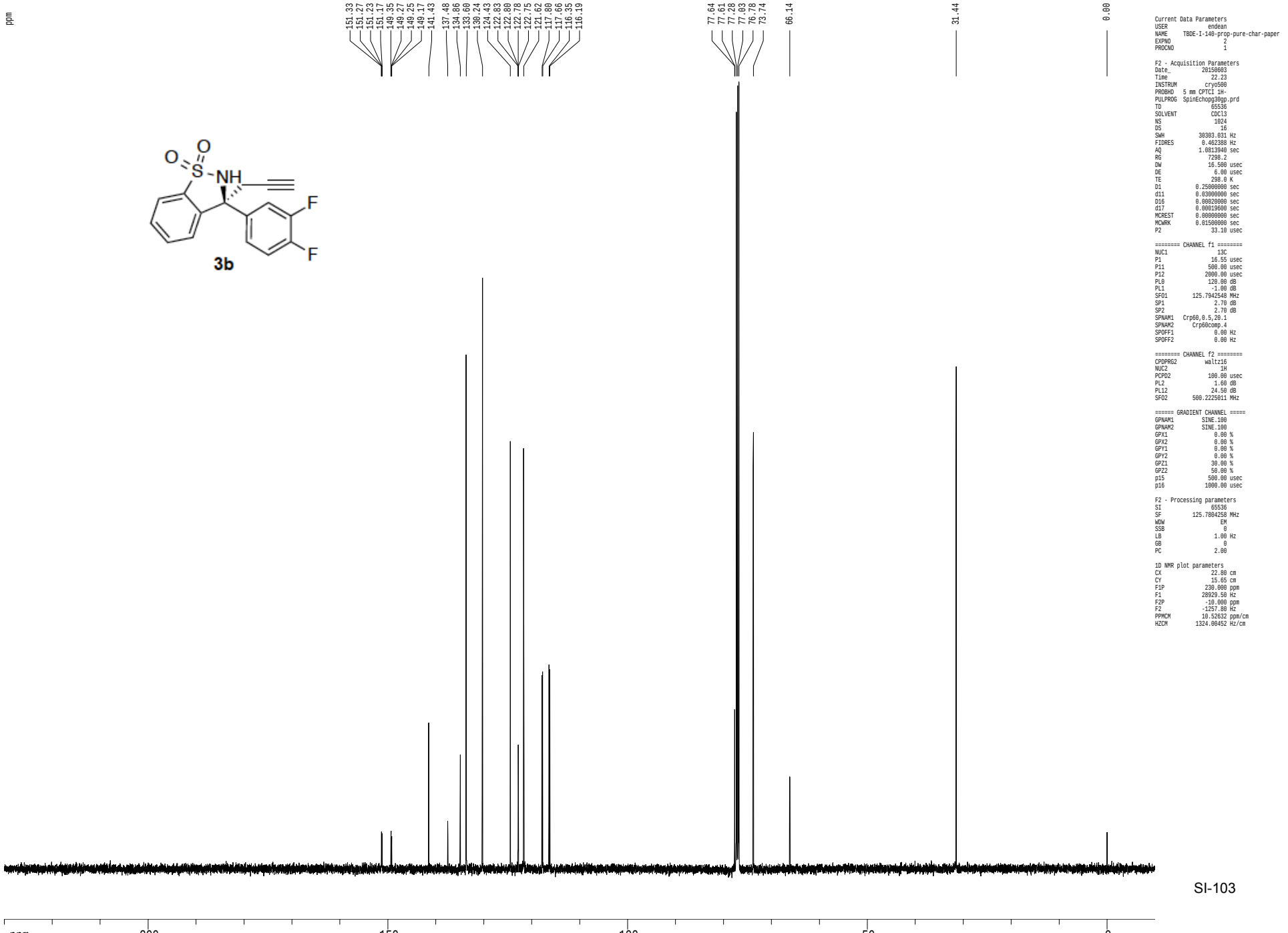
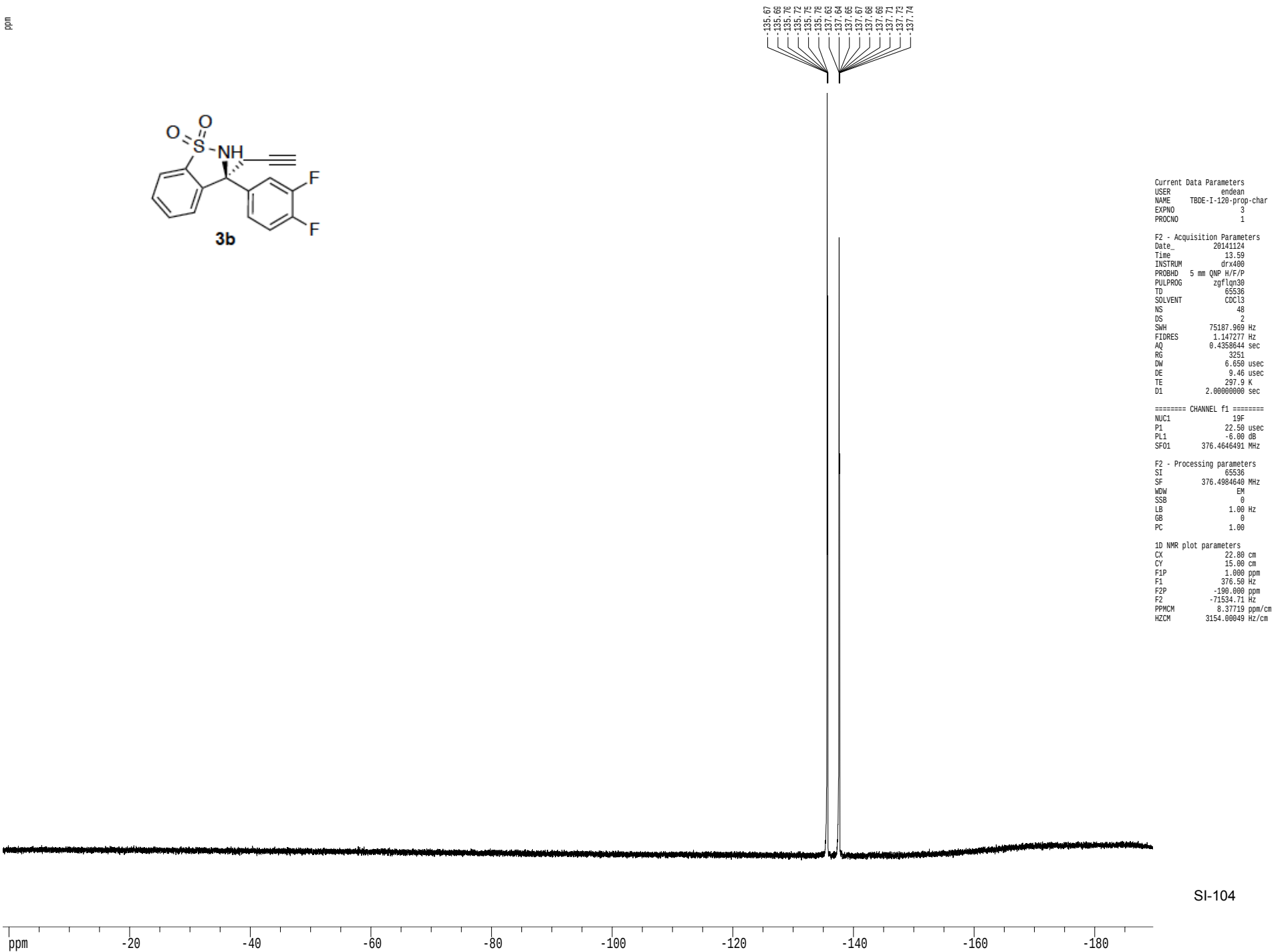
Z-restored spin-echo 13C spectrum with 1H decoupling



19F spectrum



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SFO1 376.4646491 MHz

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1D NMR plot parameters
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CY 15.00 cm
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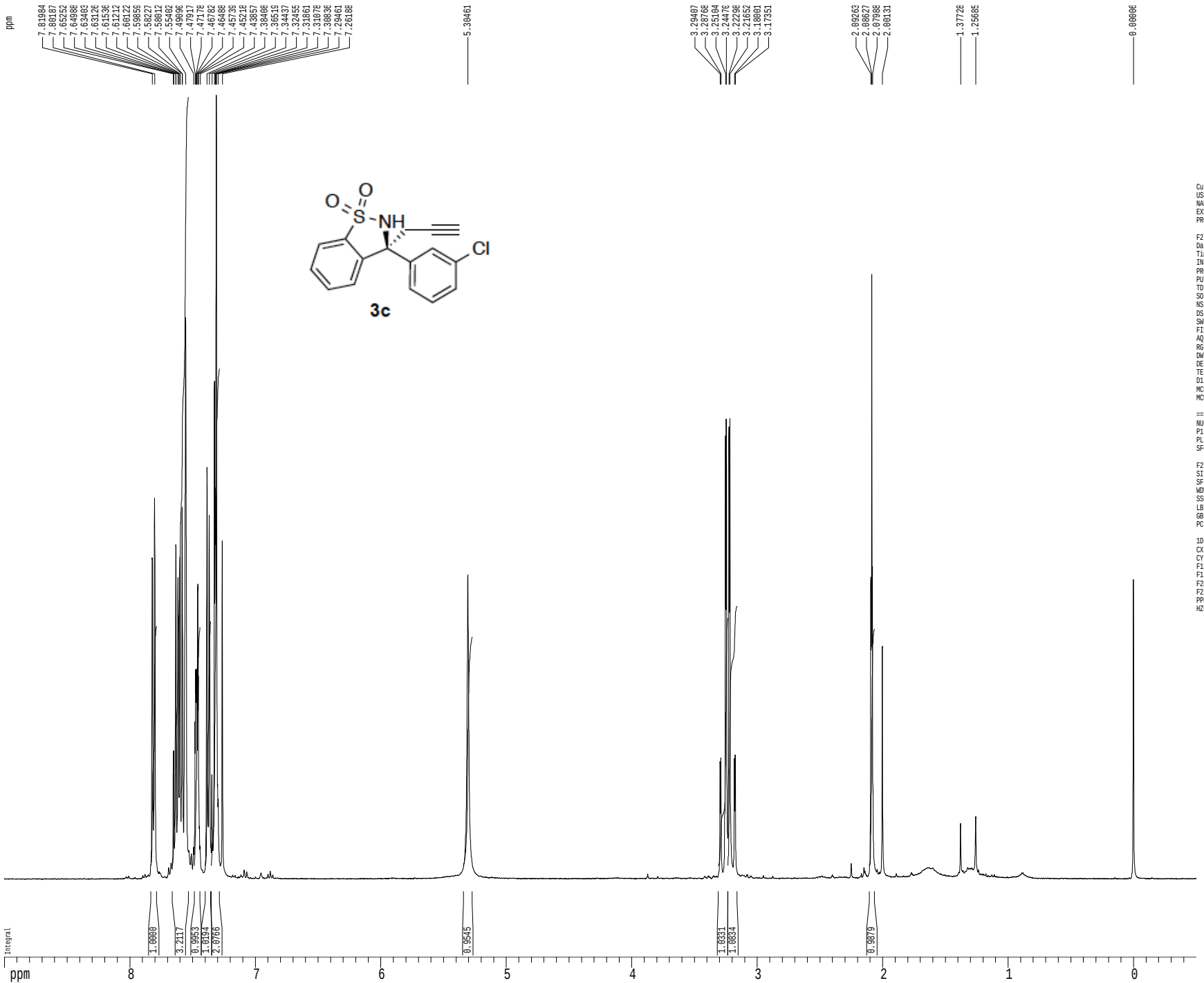
1H spectrum

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RG                   256
DW                 78.000 usec
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TE                 298.0 K
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======== CHANNEL f1 ========
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SFO1          400.1328009 MHz

F2 - Processing parameters
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1D NMR plot parameters
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CY                 15.00 cm
F1P                9.000 ppm
F1               3601.17 Hz
F2P               -0.500 ppm
F2              -200.06 Hz
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HZCM           166.72084 Hz/cm
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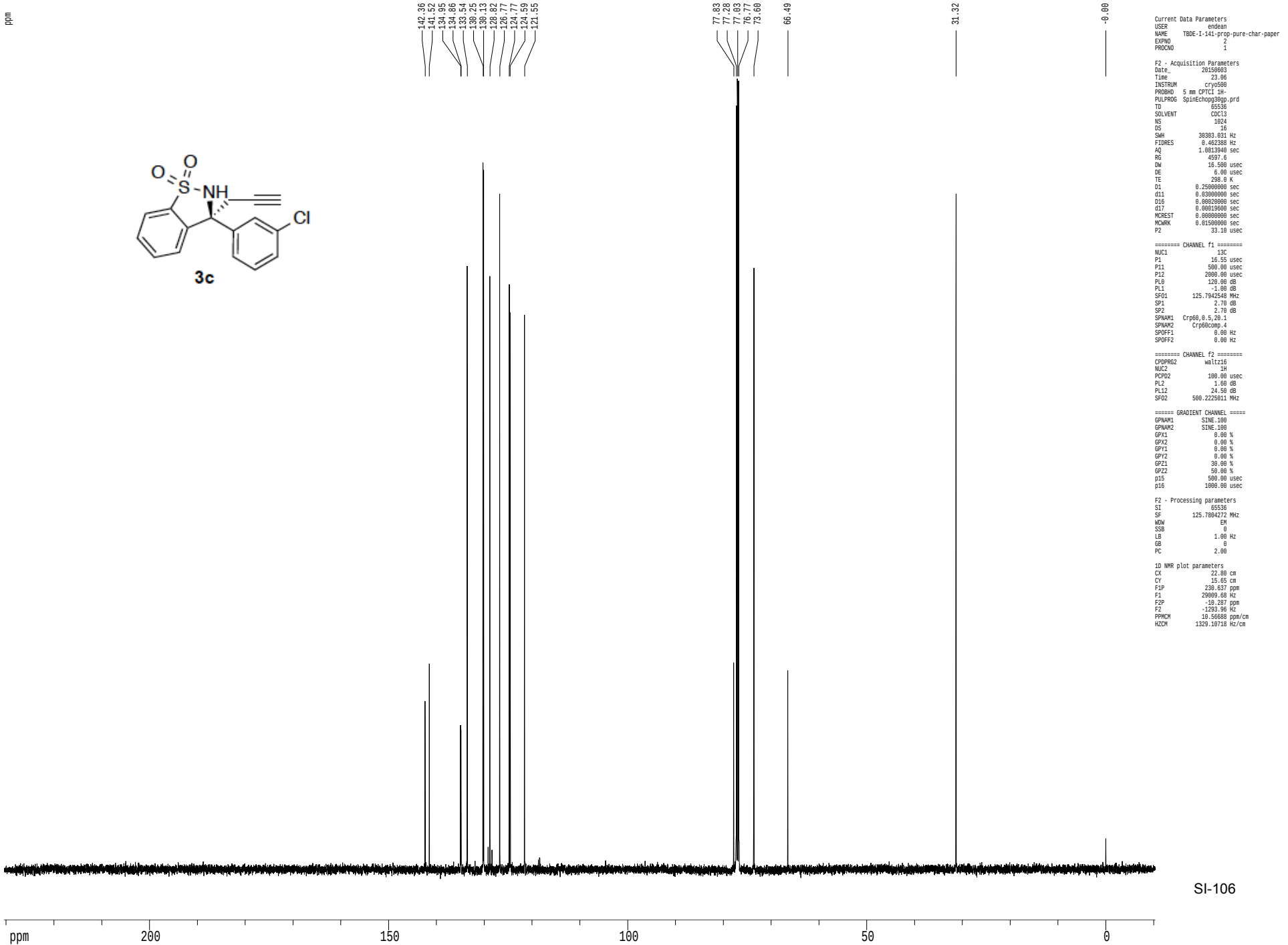
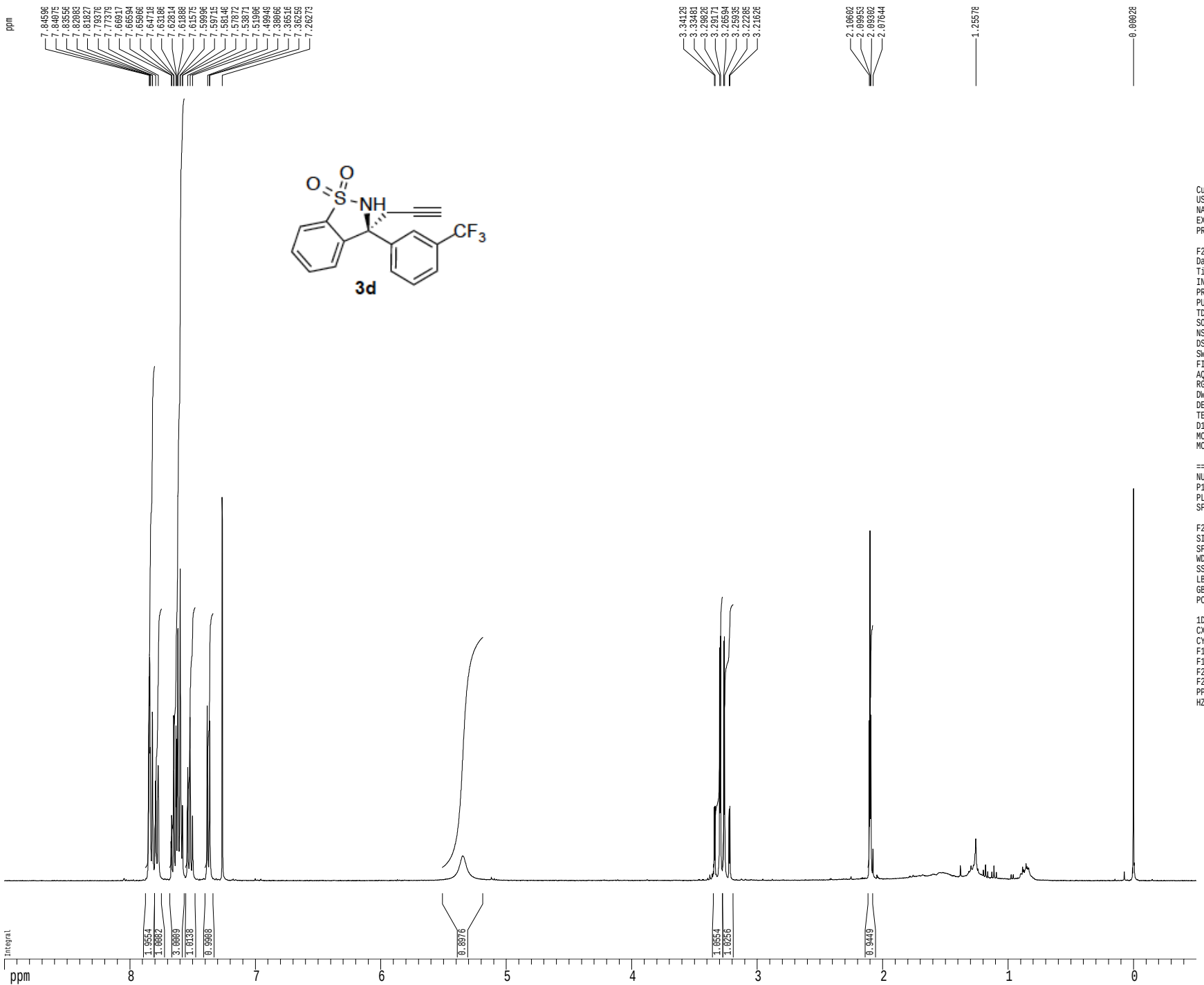
Z-restored spin-echo 13C spectrum with 1H decoupling



1H spectrum



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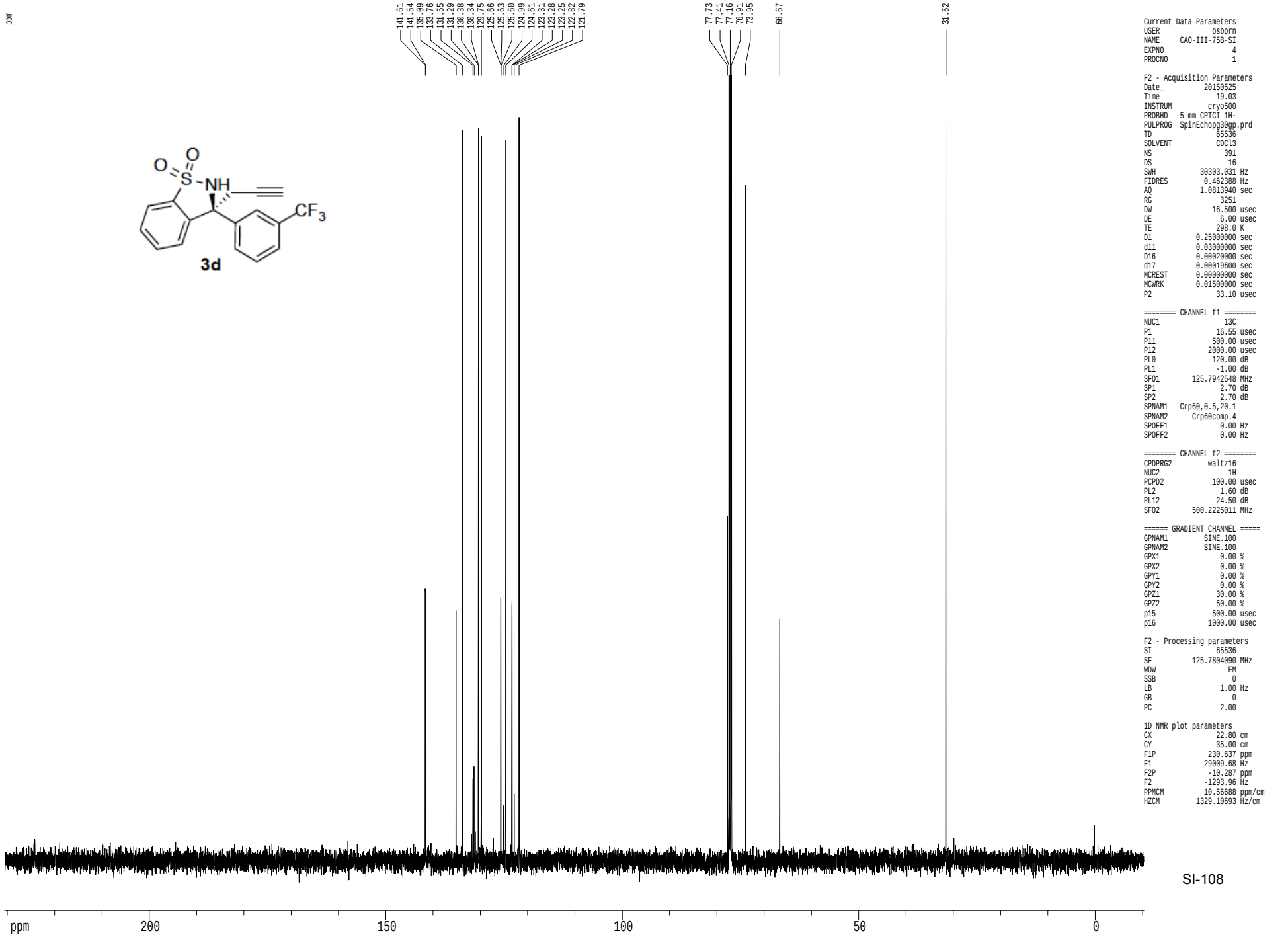
Time 14.35
INSTRUM drx400
PROBHD 5 mm QNP H/F/P
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 8
DS 2
SWH 6410.256 Hz
FIDRES 0.097813 Hz
AQ 5.1118579 sec
RG 362
DW 78.000 usec
DE 4.50 usec
TE 295.8 K
D1 0.10000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec

======== CHANNEL f1 ========
NUC1 1H
P1 12.00 usec
PL1 0.00 dB
SFO1 400.1328009 MHz

F2 - Processing parameters
SI 65536
SF 400.1300197 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 2.00

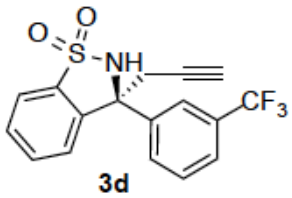
1D NMR plot parameters
CX 22.80 cm
CY 7.50 cm
F1P 9.000 ppm
F1 3601.17 Hz
F2P -0.500 ppm
F2 -200.06 Hz
PPMCM 0.41667 ppm/cm
HZCM 166.72084 Hz/cm

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




```
Current Data Parameters
USER                osborn
NAME      CAO-III-75B-SI
EXPNO                  4
PROCNO                 1

F2 - Acquisition Parameters
Date_           20150525
Time                19.03
INSTRUM           cryo500
PROBHD   5 mm CPTCI 1H-
PULPROG  SpinEchopg30gp.prd
TD                 65536
SOLVENT            CDCl3
NS                   391
DS                    16
SWH            30303.031 Hz
FIDRES         0.462388 Hz
AQ             1.0813940 sec
RG                 3251
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                33.10 usec

======== CHANNEL f1 ========
NUC1                 13C
P1                16.55 usec
P11              500.00 usec
P12             2000.00 usec
PL0              120.00 dB
PL1               -1.00 dB
SFO1         125.7942548 MHz
SP1                2.70 dB
SP2                2.70 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              24.50 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

1D NMR plot parameters
CX                22.80 cm
CY                35.00 cm
F1P             230.637 ppm
F1             29009.68 Hz
F2P             -10.287 ppm
F2             -1293.96 Hz
PPMCM          10.56688 ppm/cm
HZCM         1329.10693 Hz/cm
```

19F spectrum with 1H decoupling

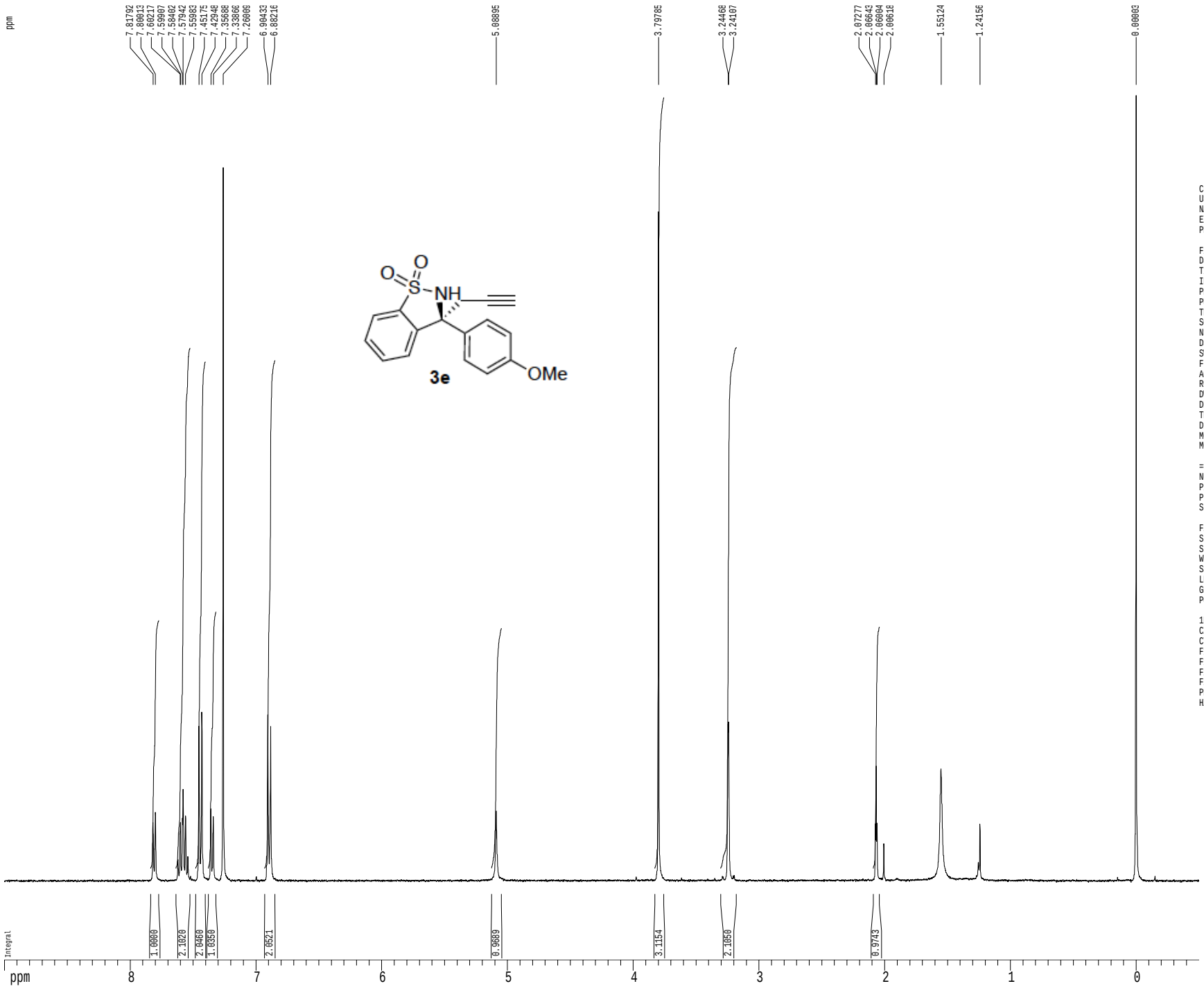


3d

```
Current Data Parameters
USER              osborn
NAME      CAO-III-75B-SI
EXPNO                7
PROCNO               1

F2 - Acquisition Parameters
Date_          20150526
Time              20.53
INSTRUM          drx400
PROBHD   5 mm QNP H/F/P
PULPROG      zgfhigqn30
TD                65536
SOLVENT           CDCl3
NS                   31
DS                    4
SWH           75187.969 Hz
FIDRES         1.147277 Hz
AQ            0.4358644 sec
RG               3649.1
DW                6.650 usec
DE                9.46 usec
TE                298.0 K
D1           2.00000000 sec
d11          0.03000000 sec
d12          0.00002000 sec

======== CHANNEL f1 ========
NUC1                19F
P1                22.50 usec
PL1               -6.00 dB
SFO1        376.4646491 MHz

======== CHANNEL f2 ========
CPDPRG2          waltz16
NUC2                1H
PCPD2             90.00 usec
PL2              120.00 dB
PL12              17.70 dB
SFO2        400.1320007 MHz

F2 - Processing parameters
SI                65536
SF          376.4983852 MHz
WDW                  EM
SSB                   0
LB                 0.30 Hz
GB                    0
PC                 1.00

1D NMR plot parameters
CX                22.80 cm
CY                15.00 cm
F1P               1.000 ppm
F1               376.50 Hz
F2P            -190.000 ppm
F2           -71534.70 Hz
PPMCM          8.37719 ppm/cm
HZCM         3153.99976 Hz/cm
```

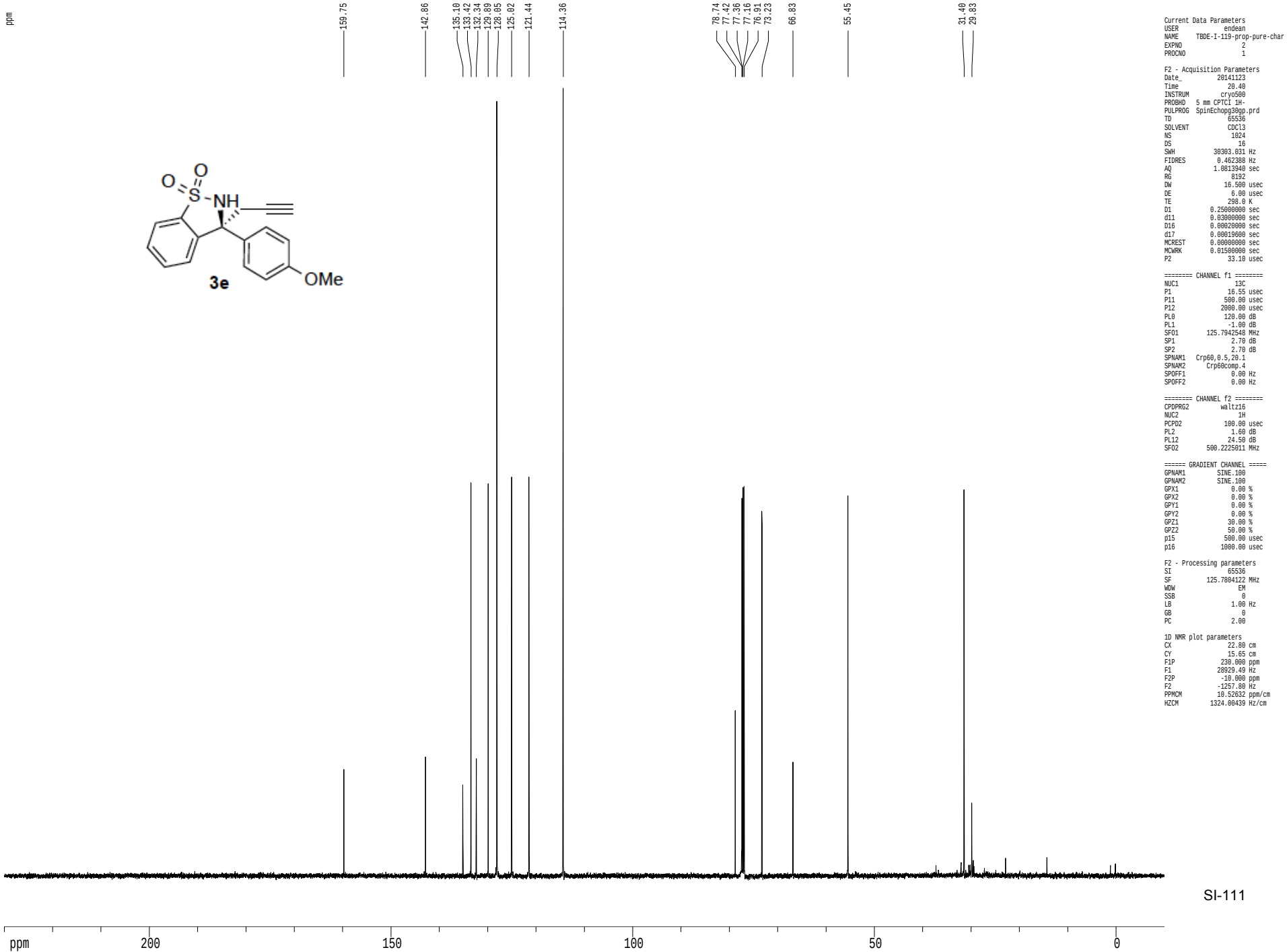

ppm -20 -40 -60 -80 -100 -120 -140 -160 -180

1H spectrum



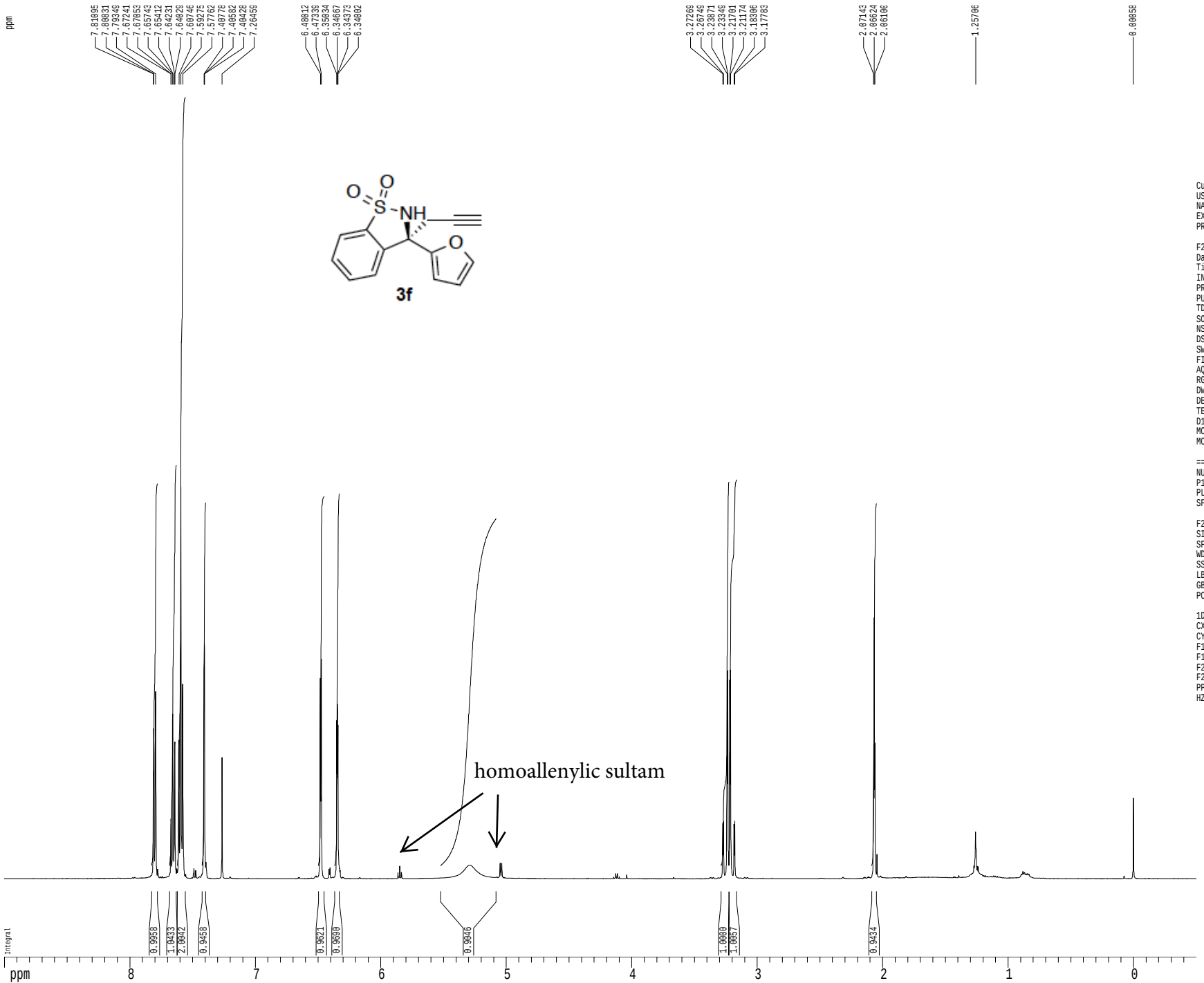

```
Current Data Parameters
USER                endean
NAME      TBDE-I-198-NIH
EXPNO                   1
PROCNO                  1

F2 - Acquisition Parameters
Date_            20150717
Time                14.54
INSTRUM           drx400
PROBHD   5 mm QNP H/F/P
PULPROG              zg30
TD                  65536
SOLVENT             CDCl3
NS                      8
DS                      2
SWH              6410.256 Hz
FIDRES           0.097813 Hz
AQ               5.1118579 sec
RG                   1024
DW                 78.000 usec
DE                  4.50 usec
TE                 298.0 K
D1            0.10000000 sec
MCREST        0.00000000 sec
MCWRK         0.01500000 sec

======== CHANNEL f1 ========
NUC1                   1H
P1                 12.00 usec
PL1                 0.00 dB
SFO1         400.1328009 MHz

F2 - Processing parameters
SI                  65536
SF           400.1300217 MHz
WDW                    EM
SSB                     0
LB                  0.30 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                -0.500 ppm
F2               -200.06 Hz
PPMCM            0.41667 ppm/cm
HZCM           166.72086 Hz/cm
```

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




```
Current Data Parameters
USER              endean
NAME     TBDE-I-119-prop-pure-char
EXPNO                  2
PROCNO                 1

F2 - Acquisition Parameters
Date_          20141123
Time               20.40
INSTRUM         cryo500
PROBHD   5 mm CPTCI 1H-
PULPROG  SpinEchopg30gp.prd
TD                 65536
SOLVENT            CDCl3
NS                  1024
DS                    16
SWH           30303.031 Hz
FIDRES         0.462388 Hz
AQ             1.0813940 sec
RG                  8192
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                33.10 usec

======== CHANNEL f1 ========
NUC1                13C
P1                16.55 usec
P11              500.00 usec
P12             2000.00 usec
PL0              120.00 dB
PL1               -1.00 dB
SFO1         125.7942548 MHz
SP1                2.70 dB
SP2                2.70 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              24.50 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           125.7804122 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              230.000 ppm
F1             28929.49 Hz
F2P              -10.000 ppm
F2             -1257.80 Hz
PPMCM          10.52632 ppm/cm
HZCM         1324.00439 Hz/cm
```

1H spectrum



Current Data Parameters
USER osborn
NAME CAO-III-92-SI
EXPNO 1
PROCNO 1

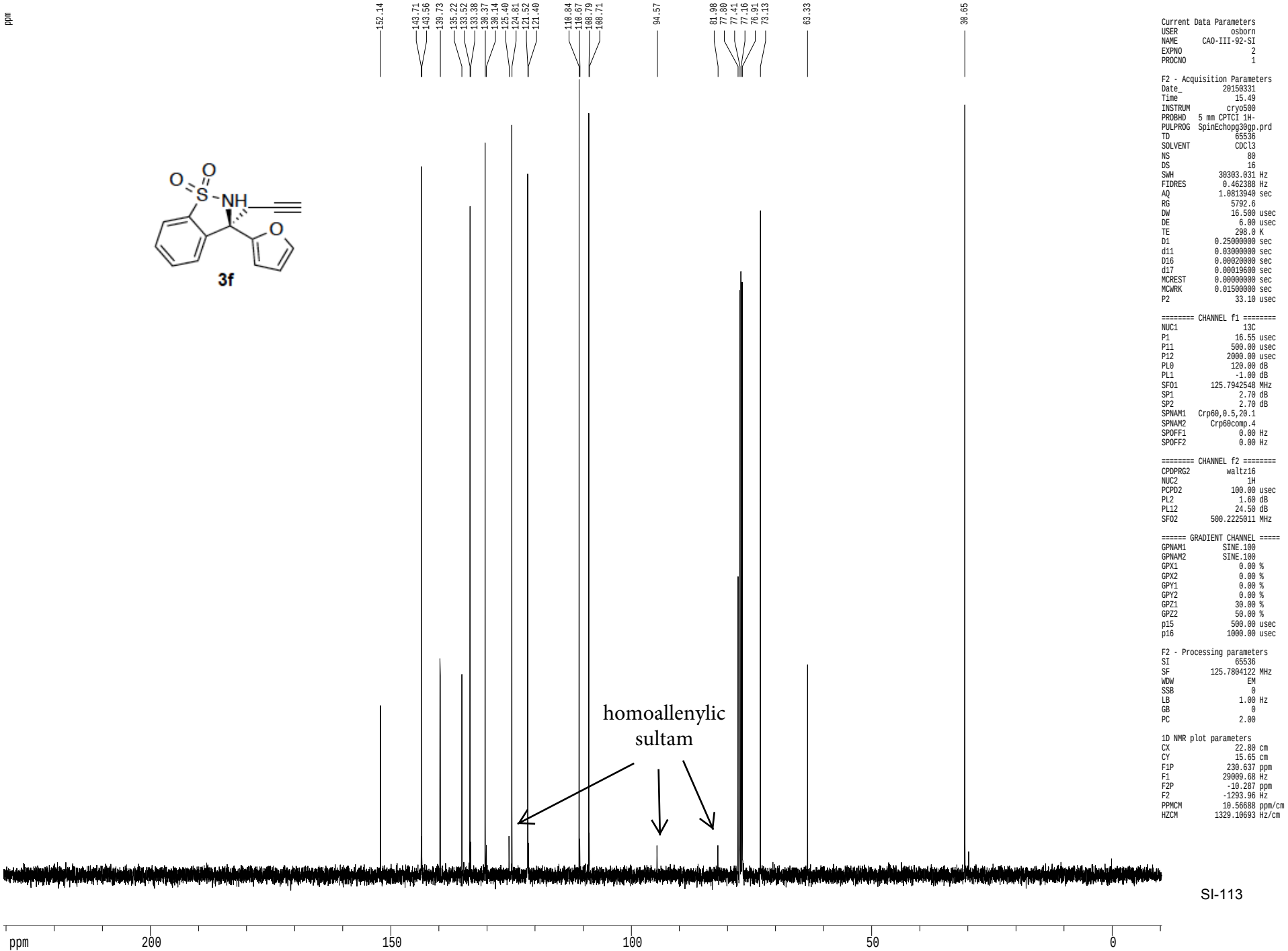
F2 - Acquisition Parameters
Date_ 20150331
Time 15.47
INSTRUM cryo500
PROBHD 5 mm CPTCI 1H-
PULPROG zg30
TD 81728
SOLVENT CDCl3
NS 8
DS 2
SWH 8012.820 Hz
FIDRES 0.098043 Hz
AQ 5.0998774 sec
RG 5
DW 62.400 usec
DE 6.00 usec
TE 298.0 K
D1 0.10000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec

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

F2 - Processing parameters
SI 65536
SF 500.2200287 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 4.00

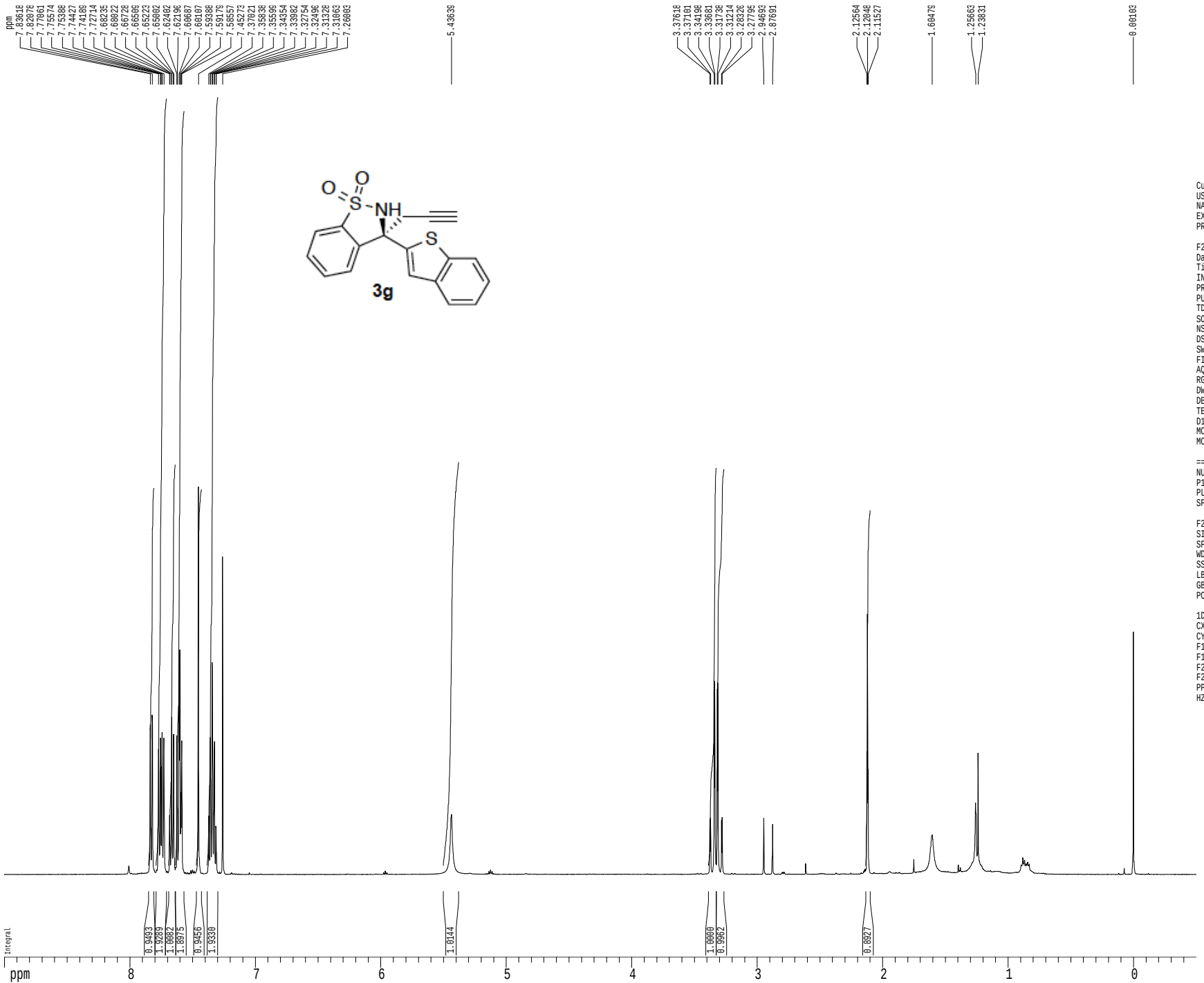
1D NMR plot parameters
CX 22.80 cm
CY 7.50 cm
F1P 9.000 ppm
F1 4501.98 Hz
F2P -0.500 ppm
F2 -250.11 Hz
PPMCM 0.41667 ppm/cm
HZCM 208.42502 Hz/cm

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



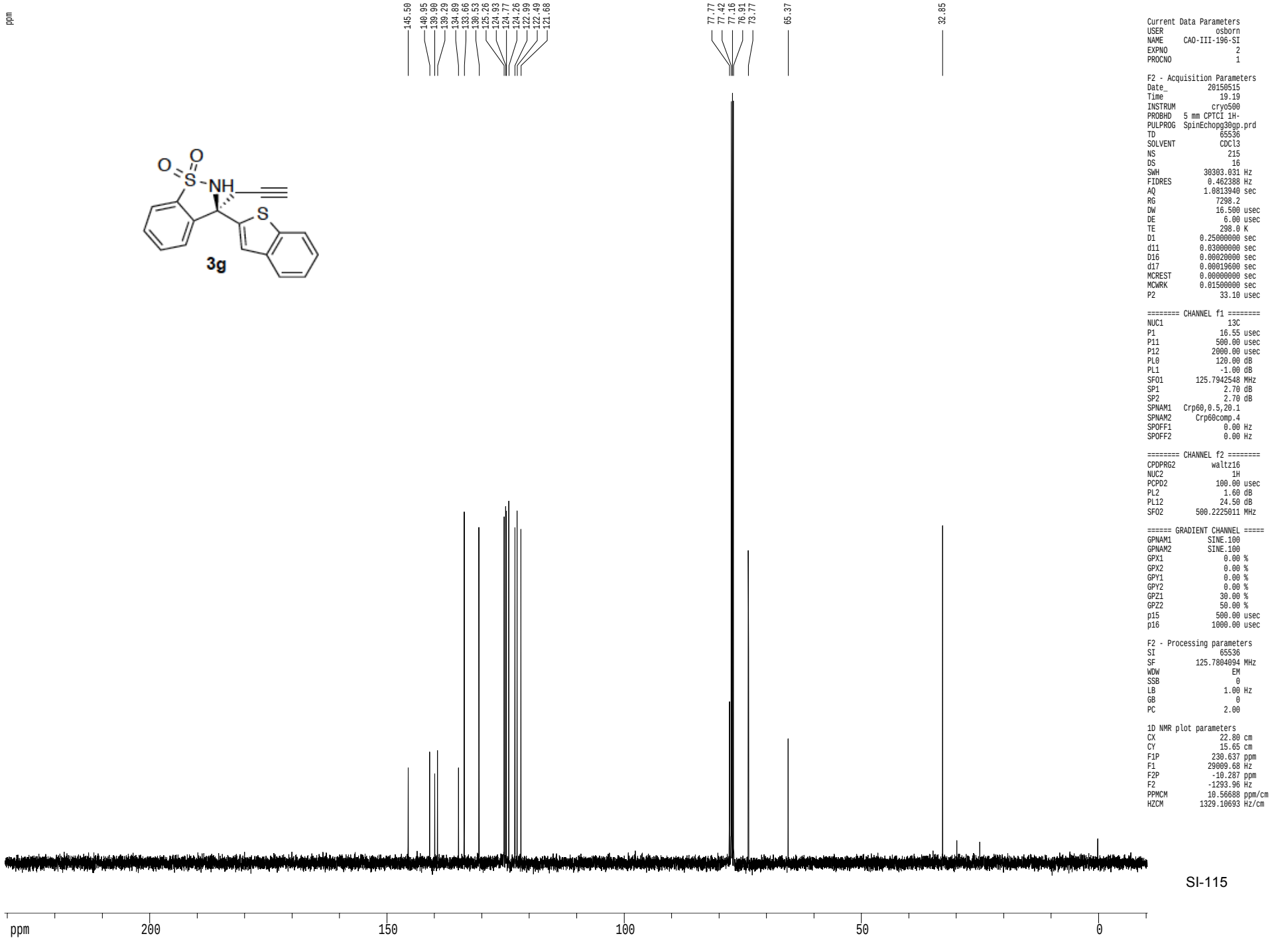

```
Current Data Parameters
USER                osborn
NAME          CAO-III-92-SI
EXPNO                    2
PROCNO                   1

F2 - Acquisition Parameters
Date_             20150331
Time                 15.49
INSTRUM           cryo500
PROBHD    5 mm CPTCI 1H-
PULPROG   SpinEchopg30gp.prd
TD                   65536
SOLVENT              CDCl3
NS                      80
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
MCWRK           0.01500000 sec
P2                  33.10 usec

======== CHANNEL f1 ========
NUC1                   13C
P1                  16.55 usec
P11                500.00 usec
P12               2000.00 usec
PL0                120.00 dB
PL1                 -1.00 dB
SFO1          125.7942548 MHz
SP1                  2.70 dB
SP2                  2.70 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                24.50 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            125.7804122 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               230.637 ppm
F1              29009.68 Hz
F2P               -10.287 ppm
F2              -1293.96 Hz
PPMCM            10.56688 ppm/cm
HZCM           1329.10693 Hz/cm
```

1H spectrum



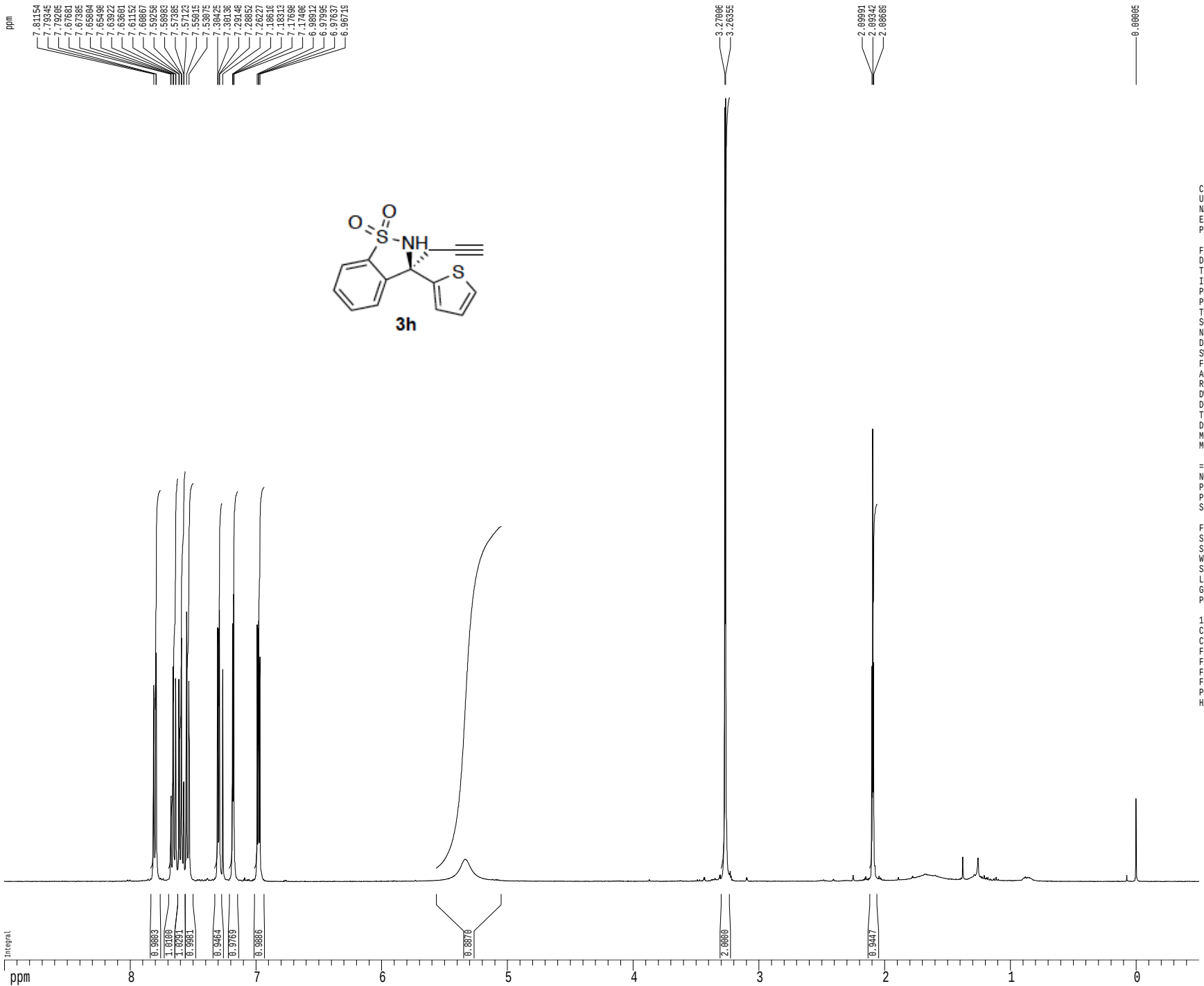

```
Current Data Parameters
USER                osborn
NAME      CAO-III-196-SI
EXPNO                  1
PROCNO                 1

F2 - Acquisition Parameters
Date_           20150515
Time               19.16
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                   3.6
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           500.2200313 MHz
WDW                   EM
SSB                    0
LB                 0.30 Hz
GB                     0
PC                 4.00

1D NMR plot parameters
CX                22.80 cm
CY                 7.50 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



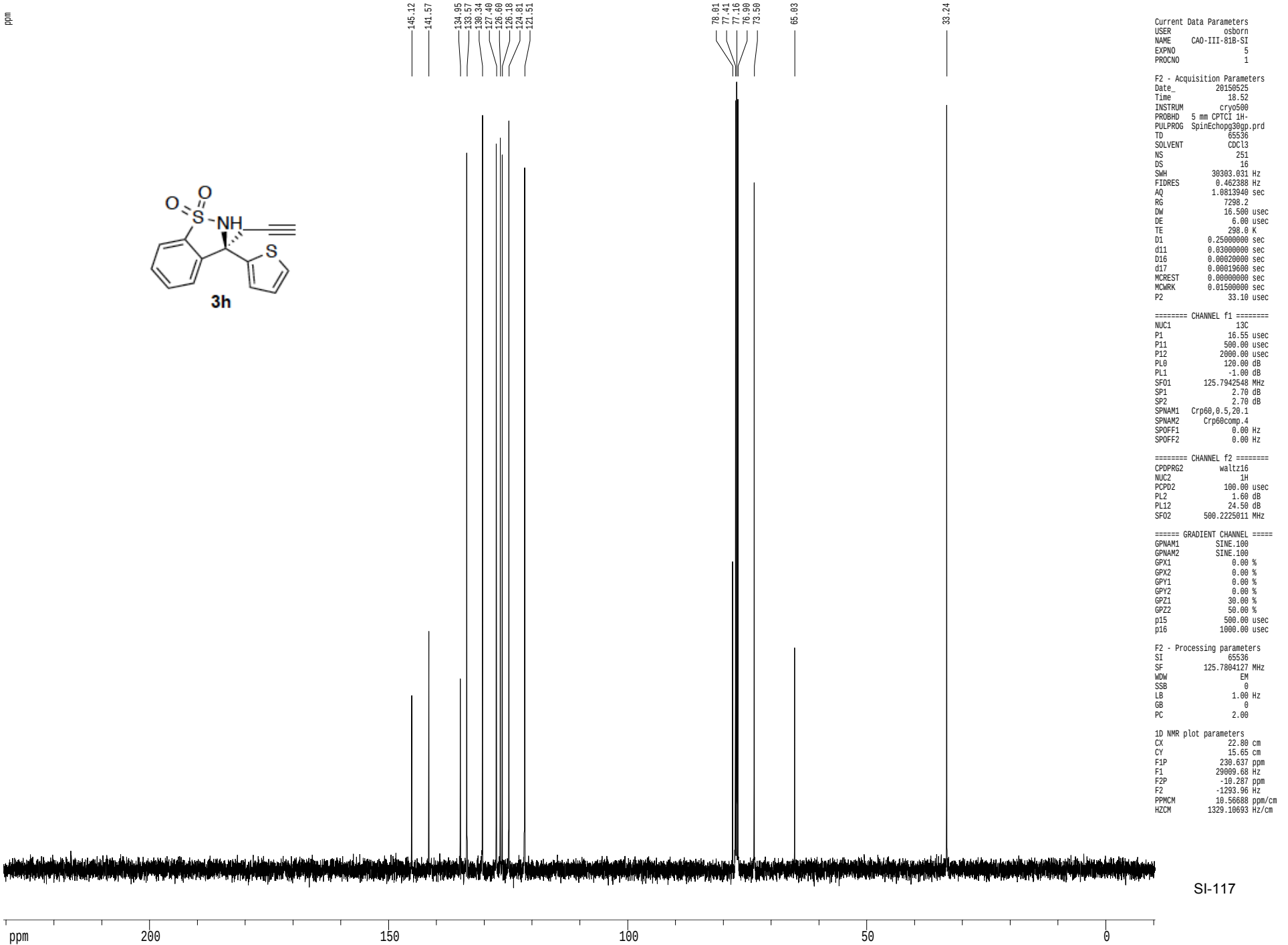

```
Current Data Parameters
USER                osborn
NAME      CAO-III-196-SI
EXPNO                  2
PROCNO                 1

F2 - Acquisition Parameters
Date_           20150515
Time                19.19
INSTRUM           cryo500
PROBHD   5 mm CPTCI 1H-
PULPROG  SpinEchopg30gp.prd
TD                 65536
SOLVENT            CDCl3
NS                   215
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
MCWRK         0.01500000 sec
P2                33.10 usec

======== CHANNEL f1 ========
NUC1                 13C
P1                16.55 usec
P11              500.00 usec
P12             2000.00 usec
PL0              120.00 dB
PL1               -1.00 dB
SFO1         125.7942548 MHz
SP1                2.70 dB
SP2                2.70 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              24.50 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           125.7804094 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              230.637 ppm
F1             29009.68 Hz
F2P              -10.287 ppm
F2             -1293.96 Hz
PPMCM           10.56688 ppm/cm
HZCM          1329.10693 Hz/cm
```

1H spectrum



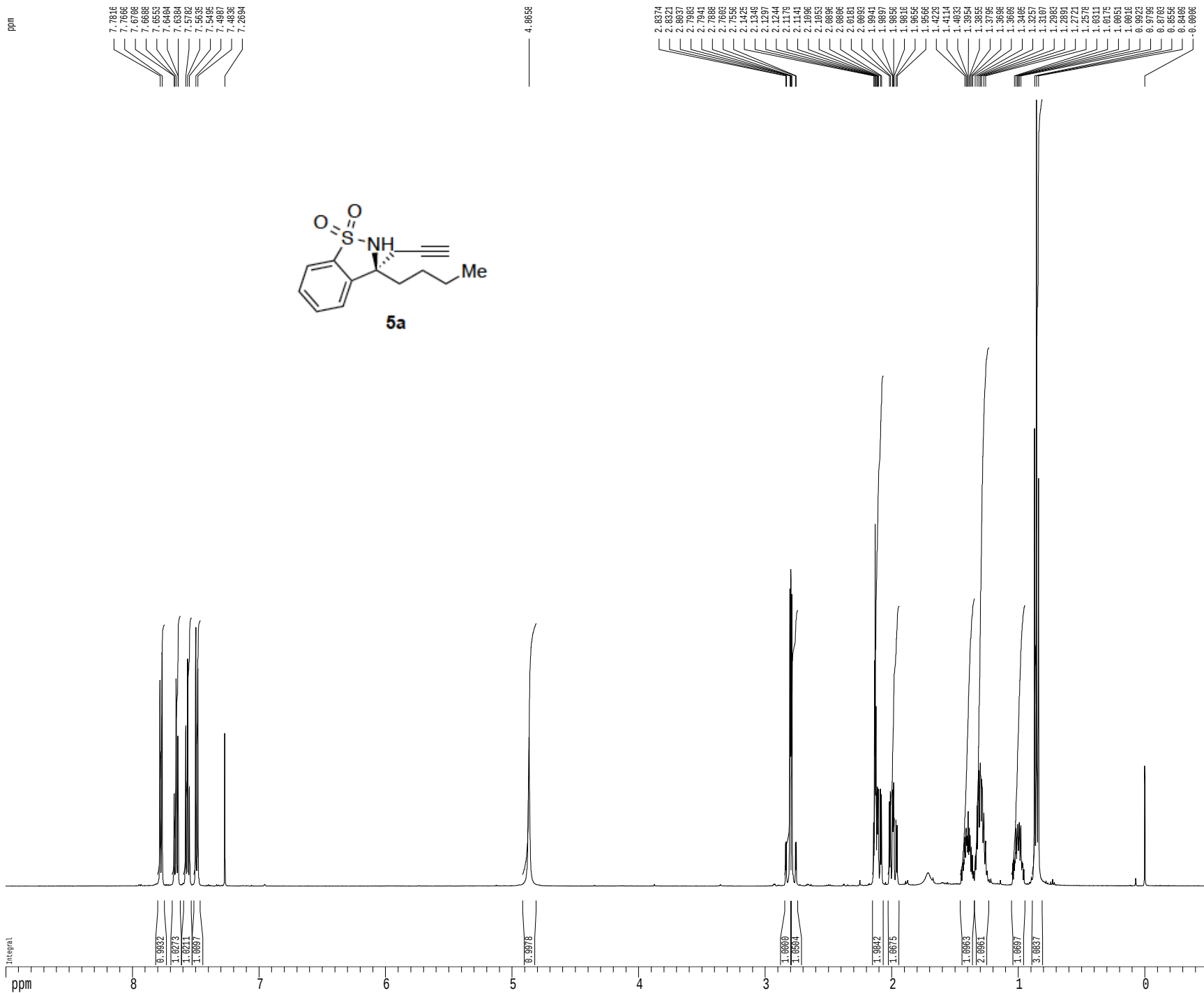

```
Current Data Parameters
USER              osborn
NAME      CAO-III-81B-SI
EXPNO                2
PROCNO               1

F2 - Acquisition Parameters
Date_          20150525
Time              18.04
INSTRUM           drx400
PROBHD   5 mm QNP H/F/P
PULPROG            zg30
TD                65536
SOLVENT           CDCl3
NS                    8
DS                    2
SWH            6410.256 Hz
FIDRES          0.097813 Hz
AQ             5.1118579 sec
RG                228.1
DW                78.000 usec
DE                 4.50 usec
TE                295.8 K
D1           0.10000000 sec
MCREST       0.00000000 sec
MCWRK        0.01500000 sec

======== CHANNEL f1 ========
NUC1                 1H
P1                12.00 usec
PL1                0.00 dB
SFO1         400.1328009 MHz

F2 - Processing parameters
SI                65536
SF           400.1300198 MHz
WDW                  EM
SSB                   0
LB                 0.30 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              -0.500 ppm
F2             -200.06 Hz
PPMCM           0.41667 ppm/cm
HZCM          166.72084 Hz/cm
```

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

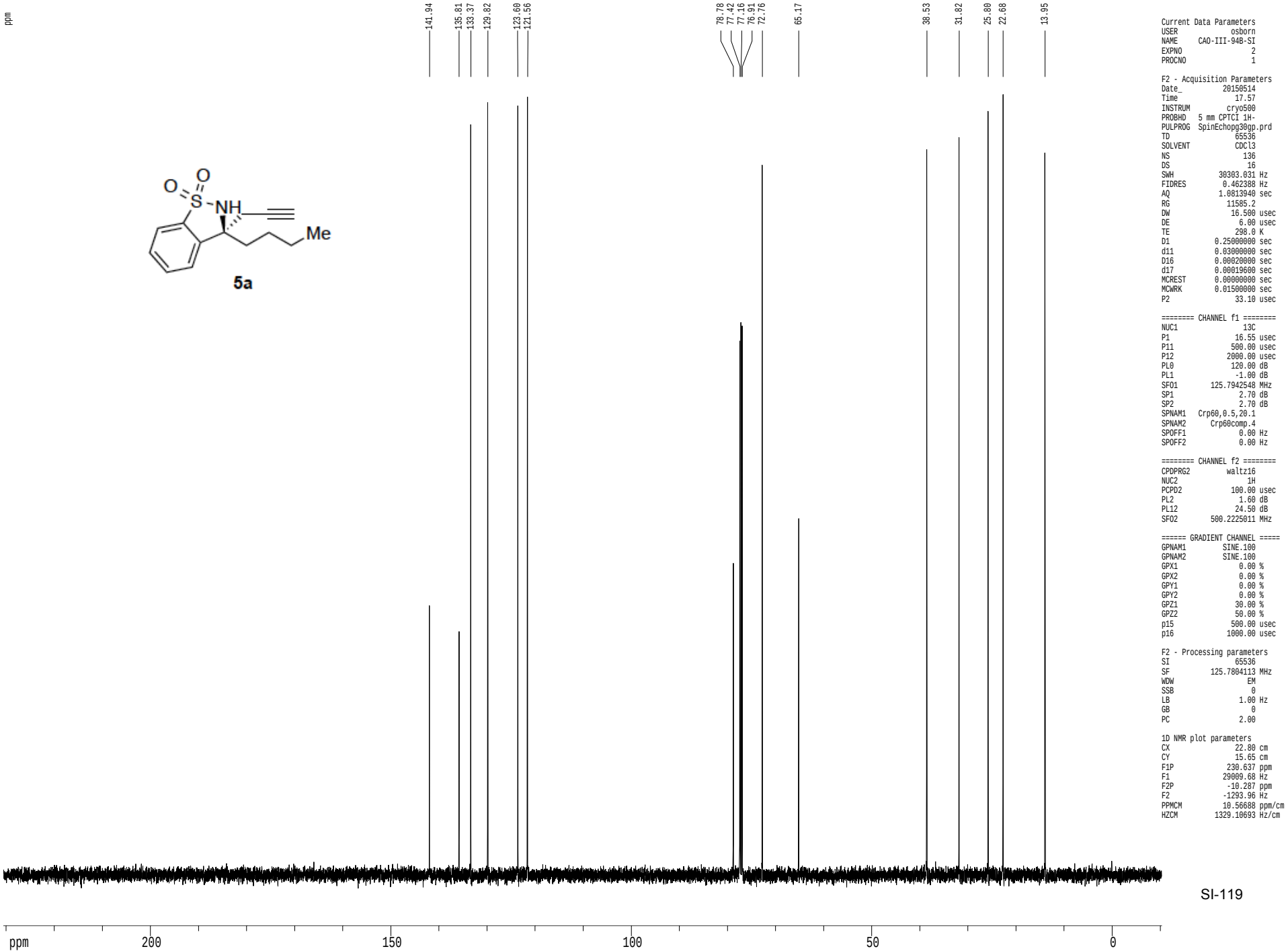



```
Current Data Parameters
USER                osborn
NAME      CAO-III-81B-SI
EXPNO                  5
PROCNO                 1

F2 - Acquisition Parameters
Date_           20150525
Time               18.52
INSTRUM          cry0500
PROBHD   5 mm CPTCI 1H-
PULPROG  SpinEchopg30gp.prd
TD                 65536
SOLVENT            CDCl3
NS                   251
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
MCWRK          0.01500000 sec
P2                33.10 usec

======== CHANNEL f1 ========
NUC1                 13C
P1                16.55 usec
P11              500.00 usec
P12             2000.00 usec
PL0              120.00 dB
PL1               -1.00 dB
SFO1         125.7942548 MHz
SP1               2.70 dB
SP2               2.70 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              24.50 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           125.7804127 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             230.637 ppm
F1             29009.68 Hz
F2P             -10.287 ppm
F2             -1293.96 Hz
PPMCM          10.56688 ppm/cm
HZCM         1329.10693 Hz/cm
```

1H spectrum

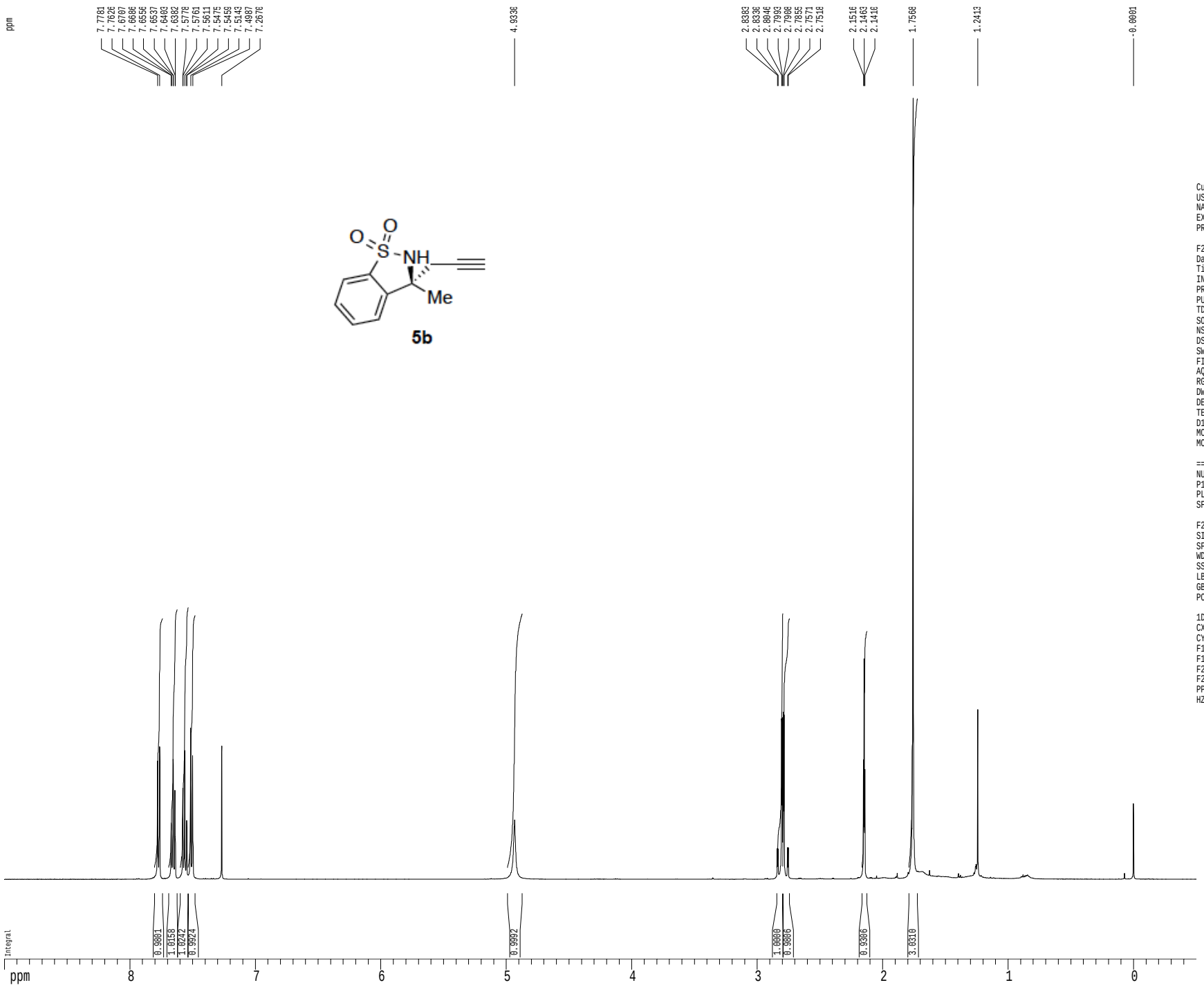

ppm
7.7816
7.7666
7.6708
7.6688
7.6553
7.6404
7.6304
7.5782
7.5635
7.5495
7.4987
7.4836
7.2694
4.8658
2.8374
2.8321
2.8037
2.7983
2.7941
2.7888
2.7663
2.7556
2.1425
2.1349
2.1297
2.1244
2.1173
2.1141
2.1096
2.1053
2.0896
2.0806
2.0181
2.0083
1.9941
1.9897
1.9836
1.9810
1.9656
1.9566
1.4228
1.4114
1.4033
1.3954
1.3855
1.3795
1.3698
1.3608
1.3405
1.3257
1.3107
1.2983
1.2891
1.2721
1.2578
1.0311
1.0175
1.0051
1.0010
0.9923
0.9798
0.8703
0.8556
0.8409
-0.0006
O
O
S
NH
Me
5a
Current Data Parameters
USER osborn
NAME CAO-III-94B-SI
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20150514
Time 17.55
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 3.2
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 500.2200264 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
Integral
0.9932
1.0273
1.0211
1.0097
0.9978
1.0000
1.0504
1.9842
1.0675
1.0963
2.0961
1.0697
3.0837
ppm
8
7
6
5
4
3
2
1
0

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



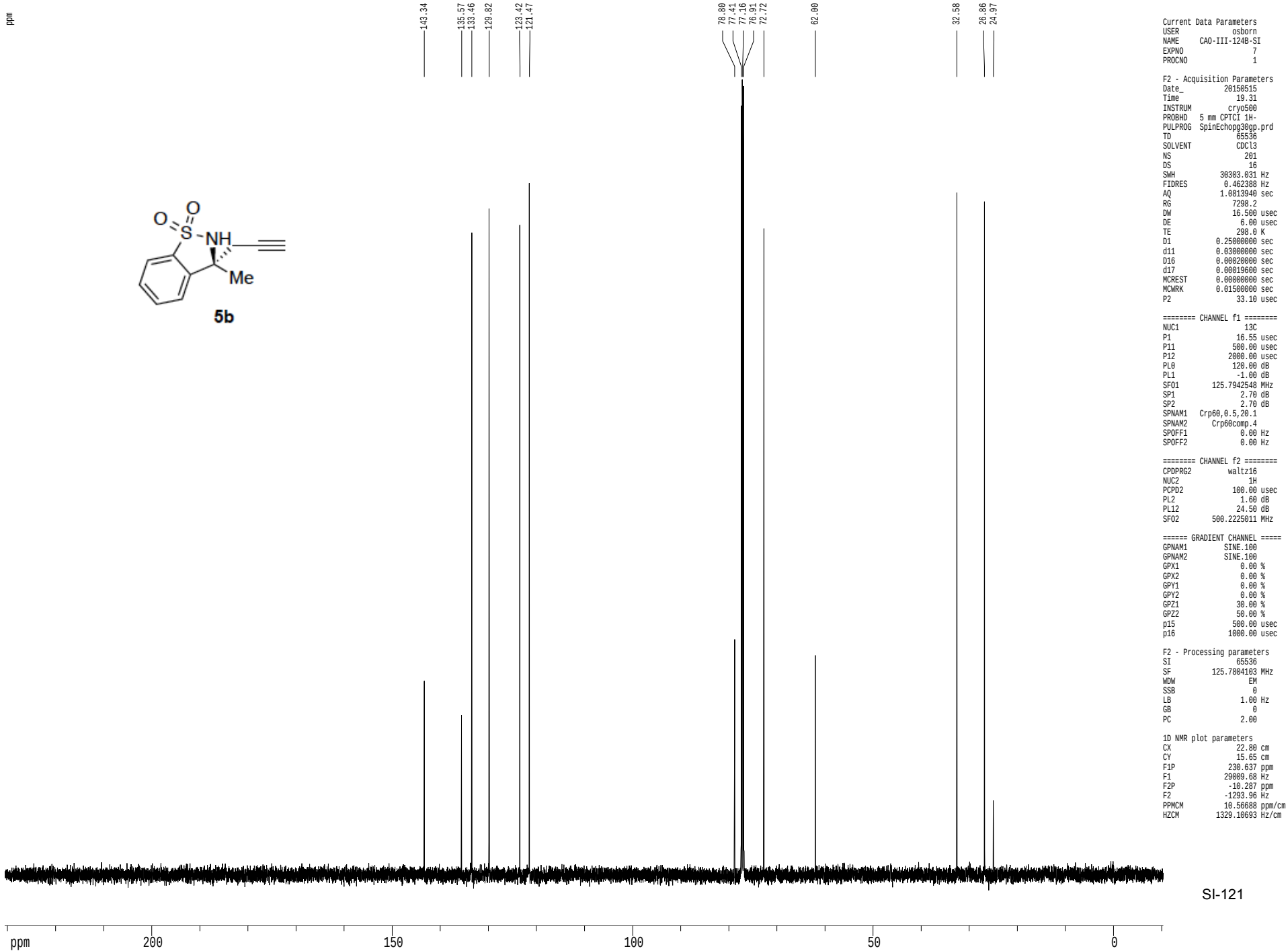

```
Current Data Parameters
USER                osborn
NAME      CAO-III-94B-SI
EXPNO                  2
PROCNO                 1

F2 - Acquisition Parameters
Date_           20150514
Time               17.57
INSTRUM          cryo500
PROBHD   5 mm CPTCI 1H-
PULPROG  SpinEchopg30gp.prd
TD                 65536
SOLVENT            CDCl3
NS                   136
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             0.25000000 sec
d11            0.03000000 sec
D16            0.00020000 sec
d17            0.00019600 sec
MCREST         0.00000000 sec
MCWRK          0.01500000 sec
P2                33.10 usec

======== CHANNEL f1 ========
NUC1                 13C
P1                16.55 usec
P11              500.00 usec
P12             2000.00 usec
PL0              120.00 dB
PL1               -1.00 dB
SFO1         125.7942548 MHz
SP1                2.70 dB
SP2                2.70 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              24.50 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           125.7804113 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             230.637 ppm
F1             29009.68 Hz
F2P             -10.287 ppm
F2             -1293.96 Hz
PPMCM          10.56688 ppm/cm
HZCM         1329.10693 Hz/cm
```

1H spectrum



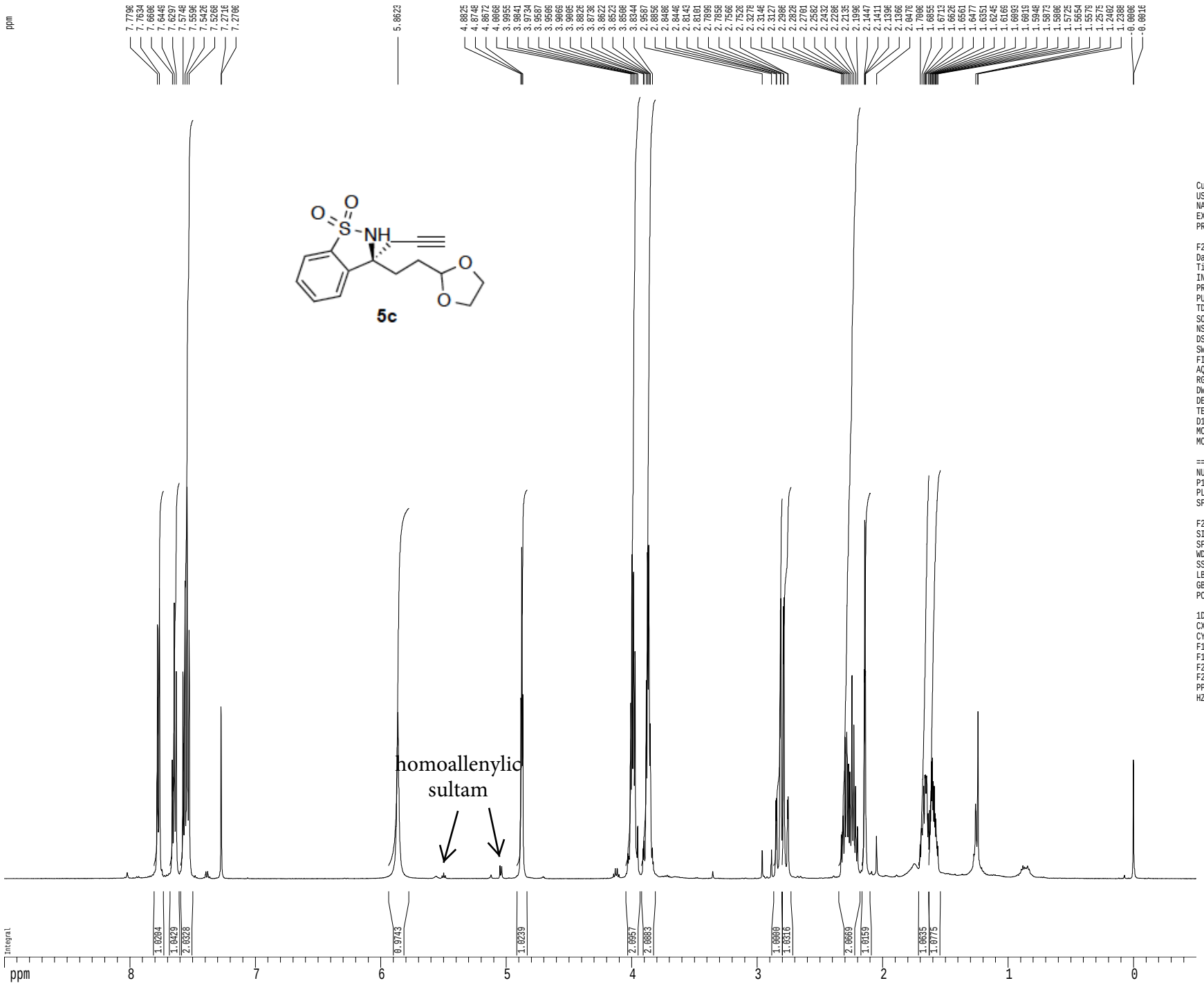

```
Current Data Parameters
USER                osborn
NAME      CAO-III-124B-SI
EXPNO                    3
PROCNO                   1

F2 - Acquisition Parameters
Date_            20150515
Time                16.42
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                      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            500.2200275 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



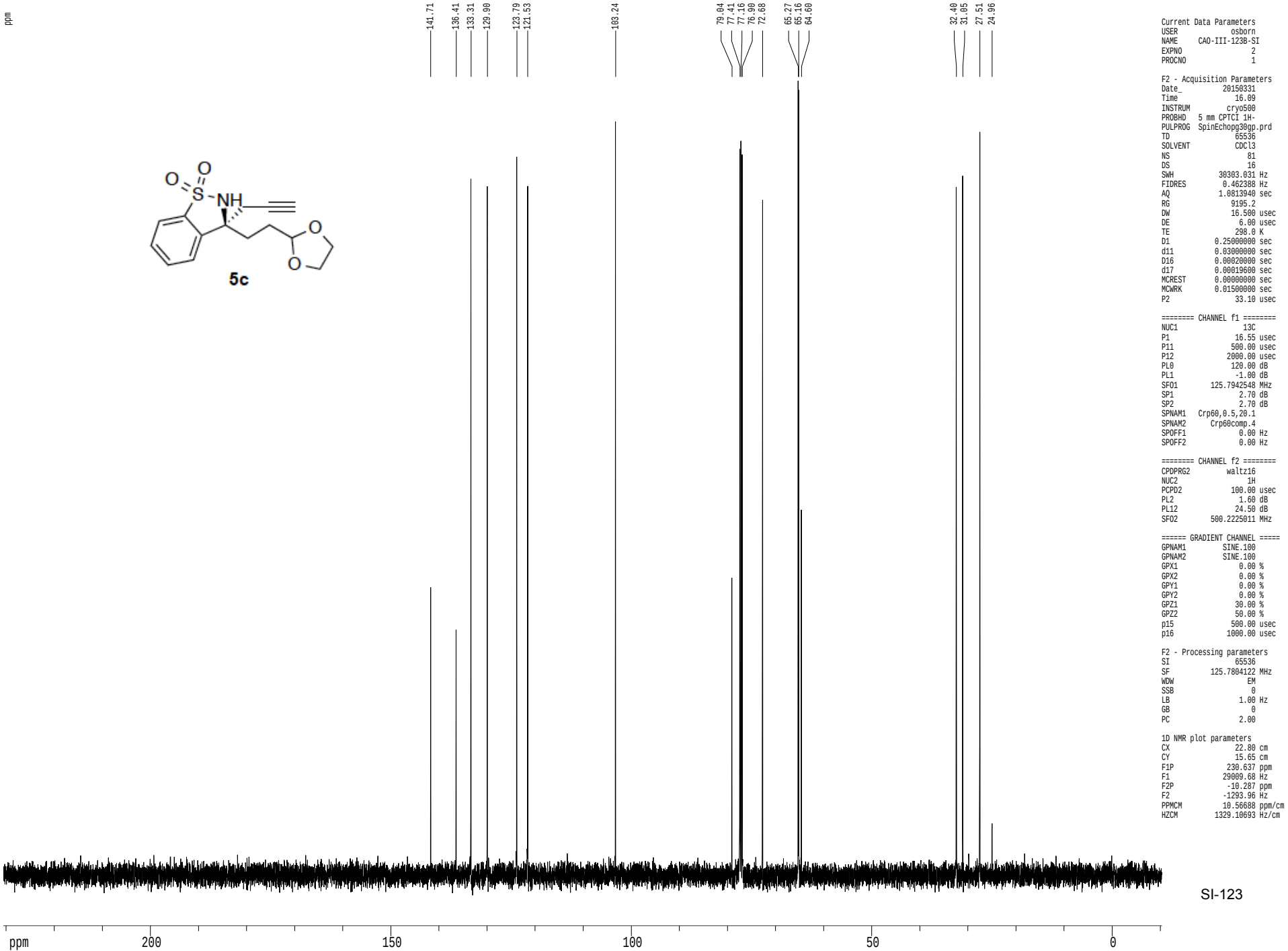

```
Current Data Parameters
USER                osborn
NAME      CAO-III-124B-SI
EXPNO                  7
PROCNO                 1

F2 - Acquisition Parameters
Date_           20150515
Time              19.31
INSTRUM          cryo500
PROBHD   5 mm CPTCI 1H-
PULPROG  SpinEchopg30gp.prd
TD                65536
SOLVENT           CDCl3
NS                  201
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
MCWRK         0.01500000 sec
P2               33.10 usec

======== CHANNEL f1 ========
NUC1                13C
P1               16.55 usec
P11             500.00 usec
P12            2000.00 usec
PL0             120.00 dB
PL1              -1.00 dB
SFO1        125.7942548 MHz
SP1              2.70 dB
SP2              2.70 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             24.50 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          125.7804103 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             230.637 ppm
F1             29009.68 Hz
F2P             -10.287 ppm
F2             -1293.96 Hz
PPMCM          10.56688 ppm/cm
HZCM         1329.10693 Hz/cm
```

1H spectrum



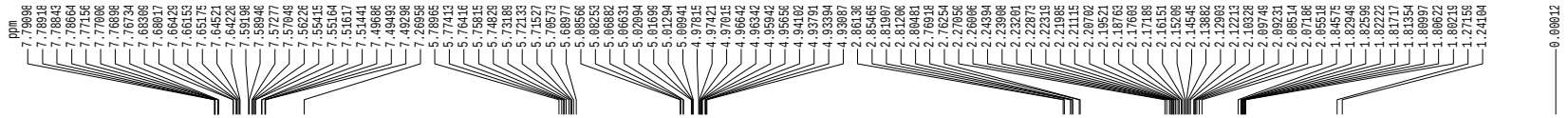

```
Current Data Parameters
USER                osborn
NAME      CAO-III-123B-SI
EXPNO                    1
PROCNO                   1

F2 - Acquisition Parameters
Date_             20150331
Time                 16.06
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                    4.5
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            500.2200253 MHz
WDW                      EM
SSB                       0
LB                   0.30 Hz
GB                        0
PC                   4.00

1D NMR plot parameters
CX                   22.80 cm
CY                    7.50 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
```

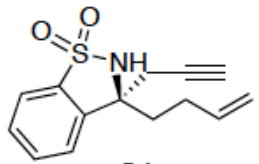
Z-restored spin-echo 13C spectrum with 1H decoupling




```
Current Data Parameters
USER                osborn
NAME      CAO-III-123B-SI
EXPNO                    2
PROCNO                   1

F2 - Acquisition Parameters
Date_             20150331
Time                 16.09
INSTRUM           cryo500
PROBHD   5 mm CPTCI 1H-
PULPROG  SpinEchopg30gp.prd
TD                   65536
SOLVENT              CDCl3
NS                      81
DS                      16
SWH              30303.031 Hz
FIDRES           0.462388 Hz
AQ               1.0813940 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
MCWRK           0.01500000 sec
P2                  33.10 usec

======== CHANNEL f1 ========
NUC1                   13C
P1                  16.55 usec
P11                500.00 usec
P12               2000.00 usec
PL0                120.00 dB
PL1                 -1.00 dB
SFO1          125.7942548 MHz
SP1                  2.70 dB
SP2                  2.70 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                24.50 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            125.7804122 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               230.637 ppm
F1              29009.68 Hz
F2P               -10.287 ppm
F2              -1293.96 Hz
PPMCM            10.56688 ppm/cm
HZCM           1329.10693 Hz/cm
```

1H spectrum



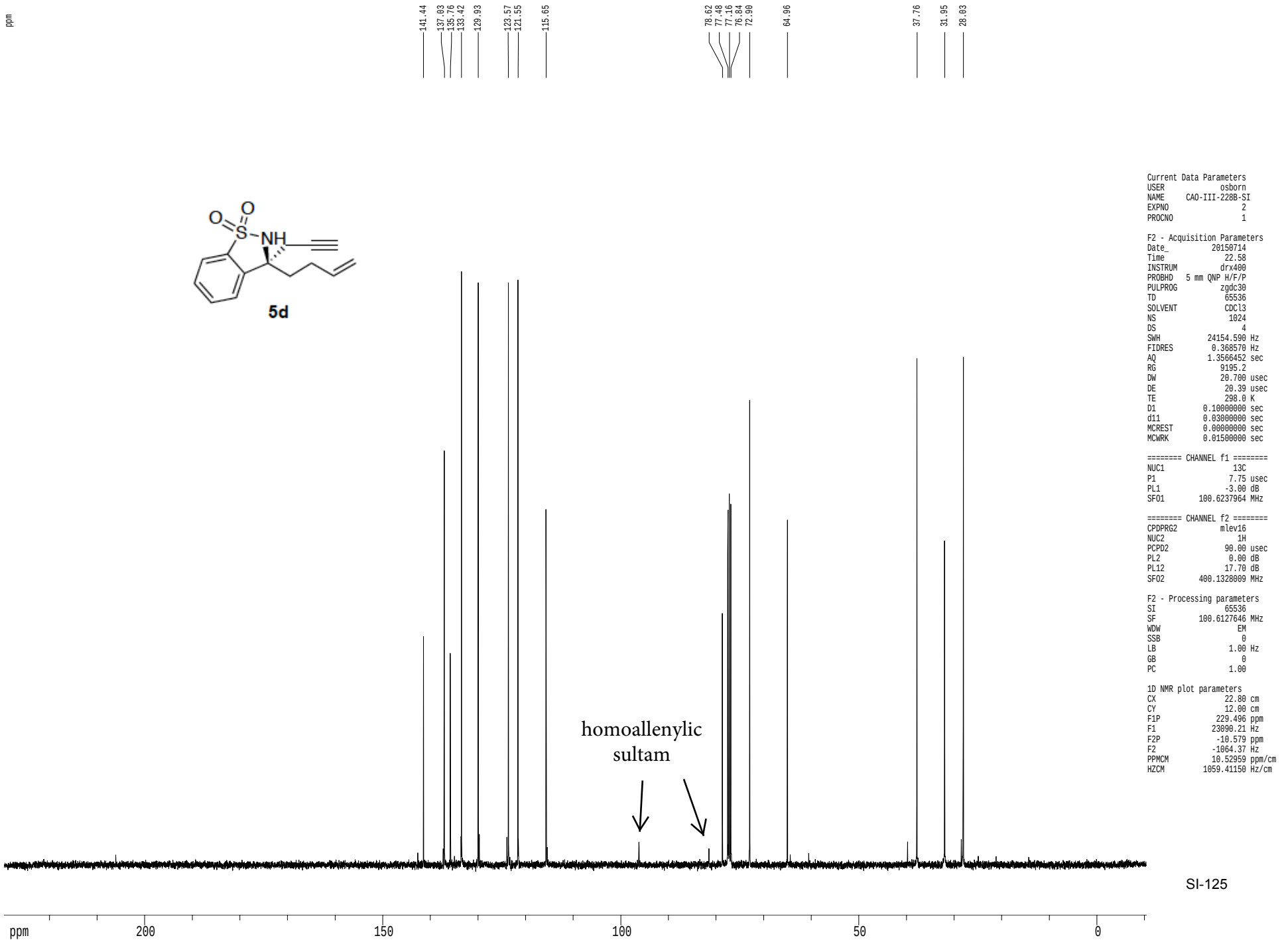


5d

homoallenylic sultam

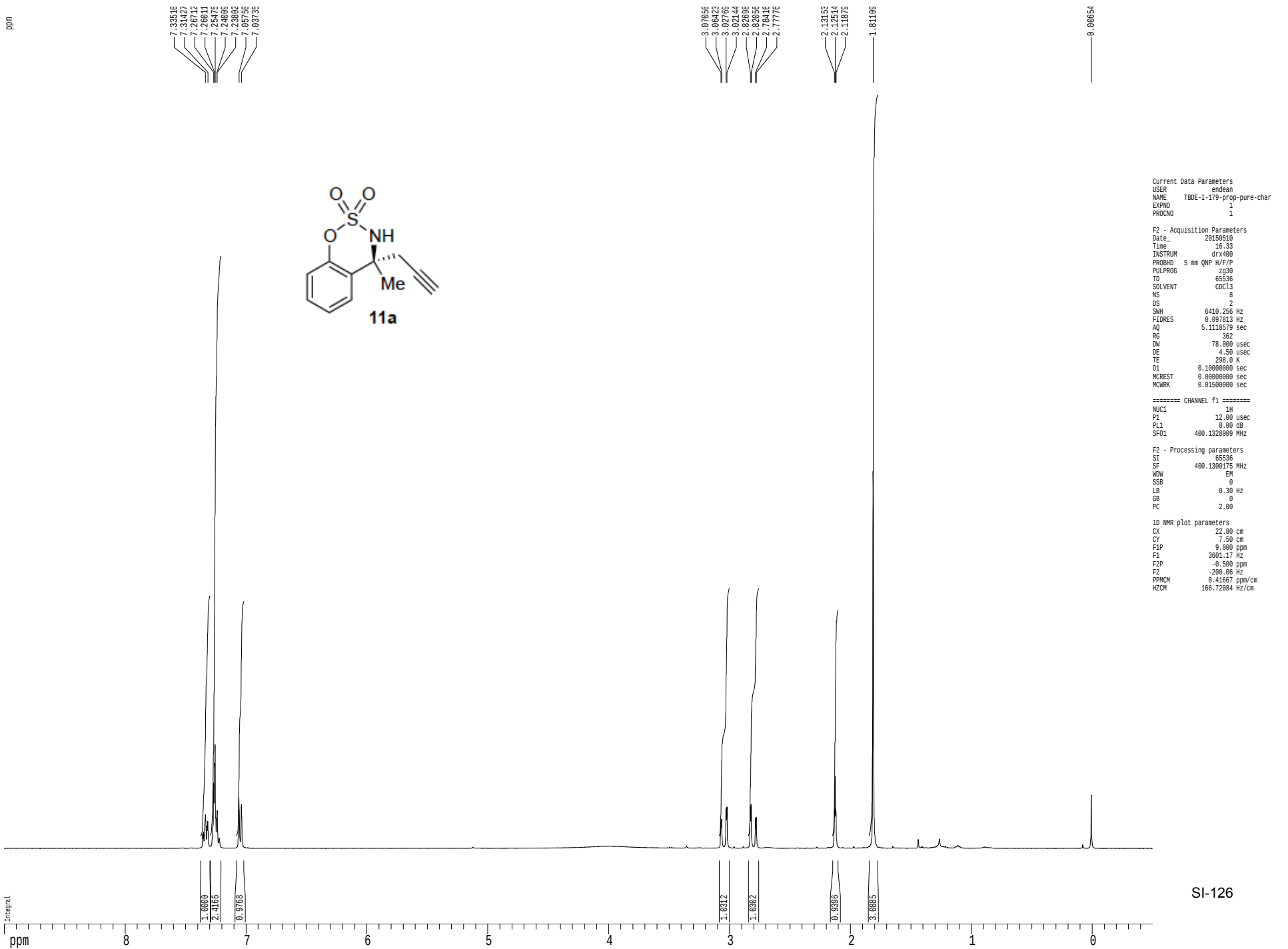
```
Current Data Parameters
USER              osborn
NAME      CAO-III-228B-SI
EXPNO                  3
PROCNO                 1

F2 - Acquisition Parameters
Date_           20150715
Time               9.17
INSTRUM          drx400
PROBHD   5 mm QNP H/F/P
PULPROG            zg30
TD                65536
SOLVENT           CDCl3
NS                     8
DS                     2
SWH           6410.256 Hz
FIDRES         0.097813 Hz
AQ            5.1118579 sec
RG                   128
DW              78.000 usec
DE               4.50 usec
TE               298.0 K
D1           0.10000000 sec
MCREST       0.00000000 sec
MCWRK        0.01500000 sec

======== CHANNEL f1 ========
NUC1                  1H
P1                12.00 usec
PL1                0.00 dB
SFO1        400.1328009 MHz

F2 - Processing parameters
SI                 65536
SF          400.1300178 MHz
WDW                   EM
SSB                    0
LB                0.30 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              -0.500 ppm
F2              -200.06 Hz
PPMCM          0.41667 ppm/cm
HZCM          166.72084 Hz/cm
```

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

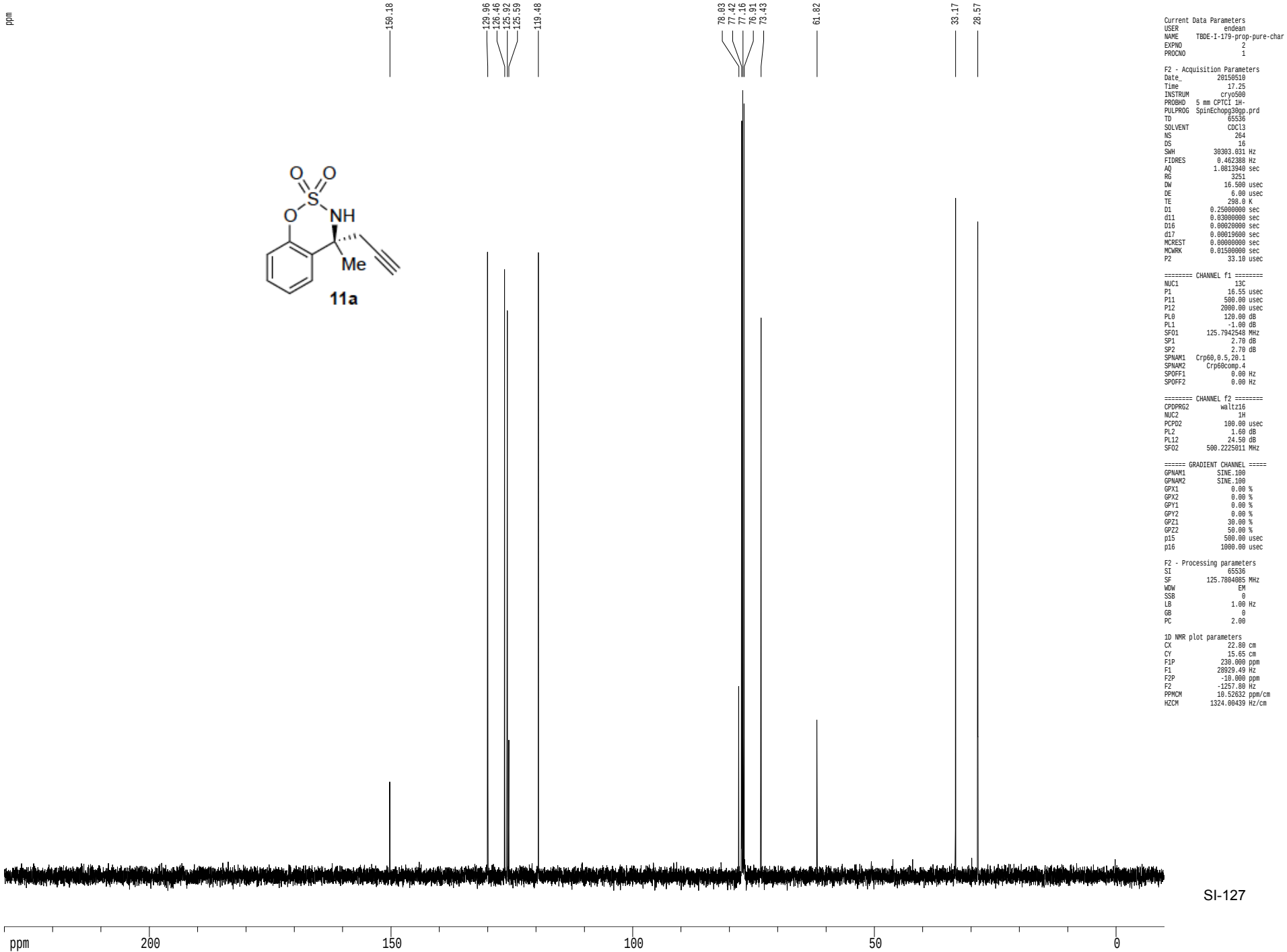



```
Current Data Parameters
USER              osborn
NAME      CAO-III-228B-SI
EXPNO                  2
PROCNO                 1

F2 - Acquisition Parameters
Date_           20150714
Time               22.58
INSTRUM          drx400
PROBHD   5 mm QNP H/F/P
PULPROG          zgdc30
TD                65536
SOLVENT           CDCl3
NS                 1024
DS                    4
SWH           24154.590 Hz
FIDRES         0.368570 Hz
AQ             1.3566452 sec
RG                9195.2
DW                20.700 usec
DE                20.39 usec
TE                298.0 K
D1           0.10000000 sec
d11          0.03000000 sec
MCREST       0.00000000 sec
MCWRK        0.01500000 sec

======== CHANNEL f1 ========
NUC1               13C
P1                7.75 usec
PL1               -3.00 dB
SFO1        100.6237964 MHz

======== CHANNEL f2 ========
CPDPRG2          mlev16
NUC2               1H
PCPD2             90.00 usec
PL2                0.00 dB
PL12              17.70 dB
SFO2        400.1328009 MHz

F2 - Processing parameters
SI                65536
SF           100.6127646 MHz
WDW                  EM
SSB                   0
LB                 1.00 Hz
GB                    0
PC                 1.00

1D NMR plot parameters
CX                22.80 cm
CY                12.00 cm
F1P              229.496 ppm
F1             23090.21 Hz
F2P              -10.579 ppm
F2             -1064.37 Hz
PPMCM          10.52959 ppm/cm
HZCM         1059.41150 Hz/cm
```

1H spectrum

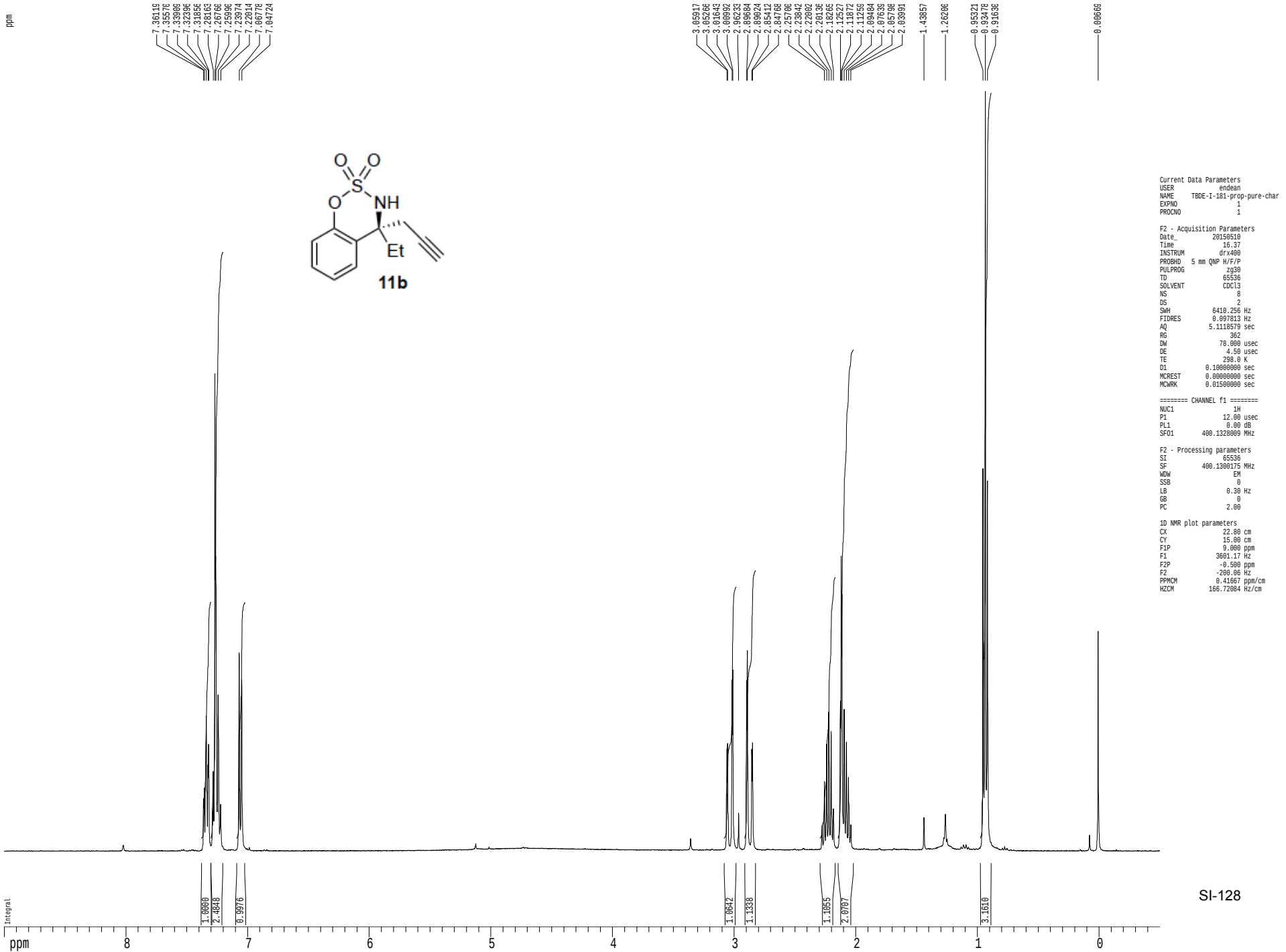

ppm
7.33516
7.31427
7.26712
7.26011
7.25475
7.24006
7.23802
7.05758
7.03735
3.07056
3.06423
3.02769
3.02144
2.82698
2.82056
2.76416
2.77776
2.13153
2.12514
2.11879
1.81108
0.00654
O
O
S
O
NH
Me
11a
Current Data Parameters
USER endean
NAME TBDE-I-179-prop-pure-char
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20150510
Time 16.33
INSTRUM drx400
PROBHD 5 mm QNP H/F/P
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 8
DS 2
SWH 6410.256 Hz
FIDRES 0.097813 Hz
AQ 5.1118579 sec
RG 362
DW 78.000 usec
DE 4.50 usec
TE 298.0 K
D1 0.10000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec
======== CHANNEL f1 ========
NUC1 1H
P1 12.00 usec
PL1 0.00 dB
SFO1 400.1328009 MHz
F2 - Processing parameters
SI 65536
SF 400.1300175 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 2.00
1D NMR plot parameters
CX 22.80 cm
CY 7.50 cm
F1P 9.000 ppm
F1 3601.17 Hz
F2P -0.500 ppm
F2 -200.06 Hz
PPMCM 0.41667 ppm/cm
HZCM 166.72084 Hz/cm
Integral
1.0000
2.4166
0.9768
1.0312
1.0302
0.9396
3.0885
ppm
8
7
6
5
4
3
2
1
0

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

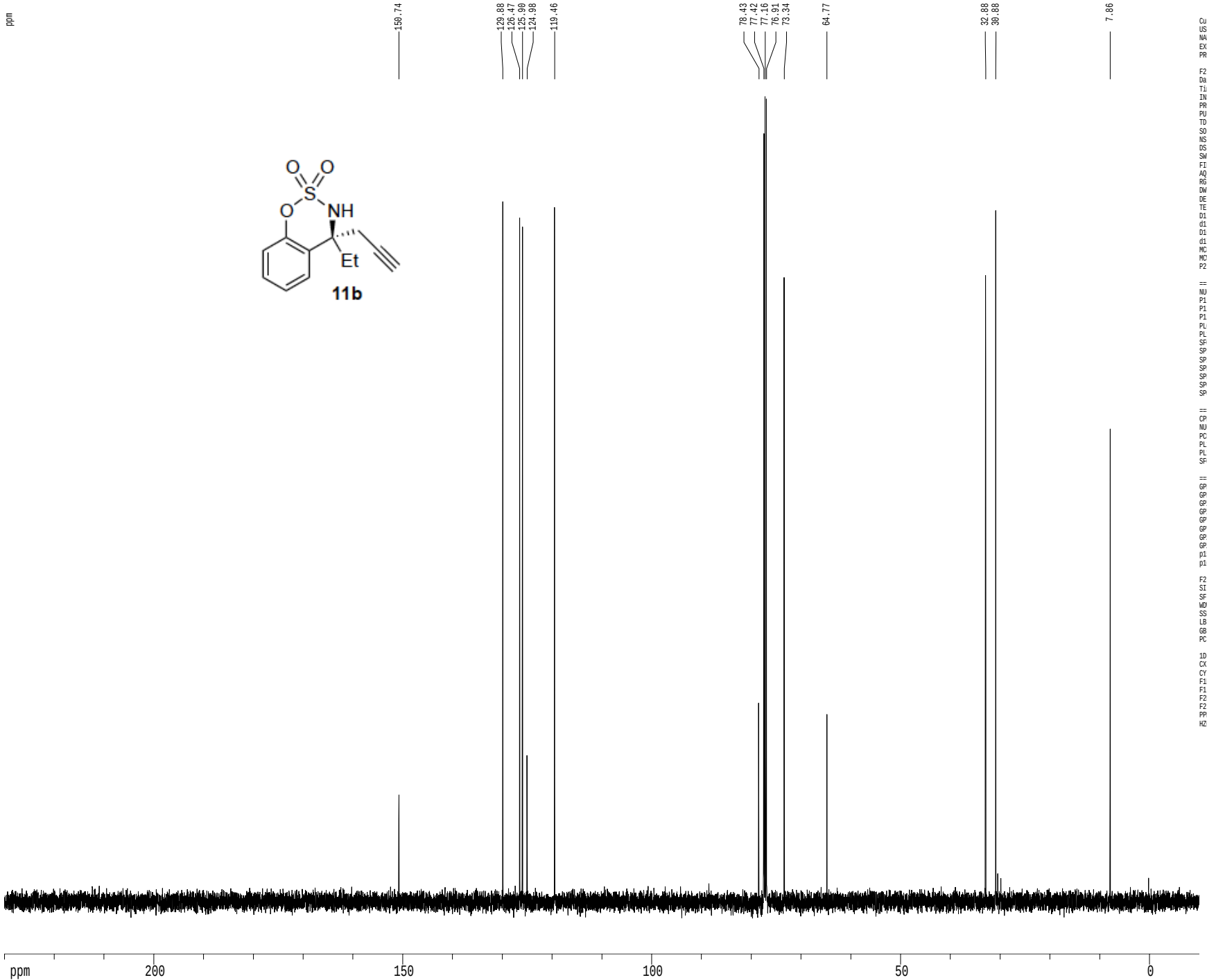



```
Current Data Parameters
USER              endean
NAME      TBDE-I-179-prop-pure-char
EXPNO                  2
PROCNO                 1

F2 - Acquisition Parameters
Date_          20150510
Time               17.25
INSTRUM         cryo500
PROBHD   5 mm CPTCI 1H-
PULPROG  SpinEchopg30gp.prd
TD                 65536
SOLVENT            CDCl3
NS                   264
DS                    16
SWH           30303.031 Hz
FIDRES         0.462388 Hz
AQ             1.0813940 sec
RG                 3251
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                33.10 usec

======== CHANNEL f1 ========
NUC1                13C
P1                16.55 usec
P11              500.00 usec
P12             2000.00 usec
PL0              120.00 dB
PL1               -1.00 dB
SFO1         125.7942548 MHz
SP1                2.70 dB
SP2                2.70 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              24.50 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           125.7804085 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              230.000 ppm
F1             28929.49 Hz
F2P              -10.000 ppm
F2             -1257.80 Hz
PPMCM          10.52632 ppm/cm
HZCM         1324.00439 Hz/cm
```

1H spectrum

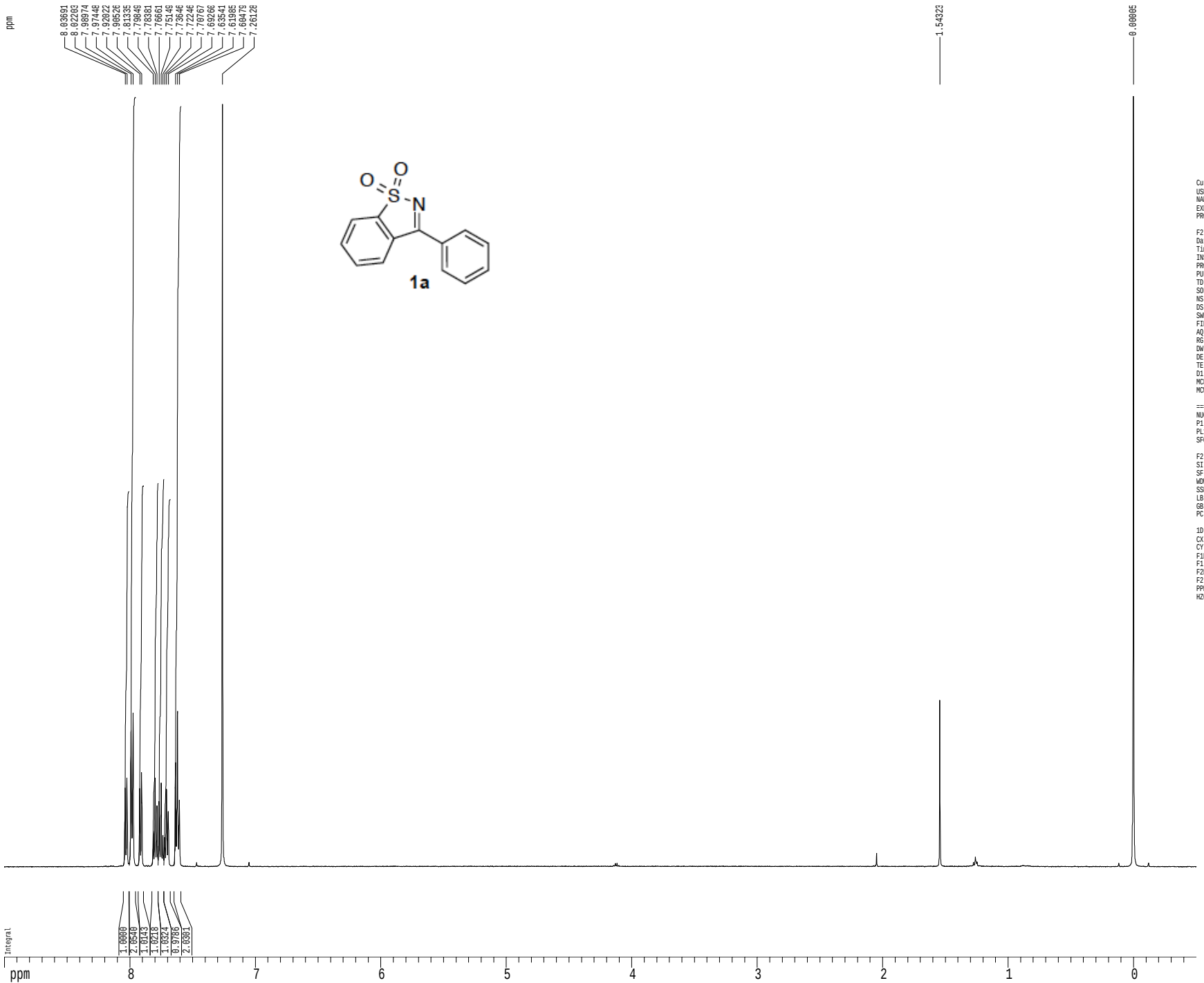

ppm
7.36119
7.35576
7.33906
7.32396
7.31856
7.28163
7.26706
7.25996
7.23974
7.22014
7.06778
7.04724
3.05917
3.05206
3.01643
3.00992
2.96233
2.89684
2.89024
2.85412
2.84768
2.25706
2.23842
2.22092
2.20136
2.18266
2.12527
2.11872
2.11259
2.09484
2.07639
2.05798
2.03991
1.43857
1.26206
0.95321
0.93478
0.91636
0.00669
O
O
S
O
NH
Et
11b
Current Data Parameters
USER endean
NAME TBDE-I-181-prop-pure-char
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20150510
Time 16.37
INSTRUM drx400
PROBHD 5 mm QNP H/F/P
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 8
DS 2
SWH 6410.256 Hz
FIDRES 0.097813 Hz
AQ 5.1118579 sec
RG 362
DW 78.000 usec
DE 4.50 usec
TE 298.0 K
D1 0.10000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec
======== CHANNEL f1 ========
NUC1 1H
P1 12.00 usec
PL1 0.00 dB
SFO1 400.1328009 MHz
F2 - Processing parameters
SI 65536
SF 400.1300175 MHz
WDW EM
SSB 0
LB 0.30 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 -0.500 ppm
F2 -200.06 Hz
PPMCM 0.41667 ppm/cm
HZCM 166.72084 Hz/cm
Integral
1.0000
2.4848
0.9976
1.0642
1.1338
1.1055
2.0707
3.1610
ppm
8
7
6
5
4
3
2
1
0

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



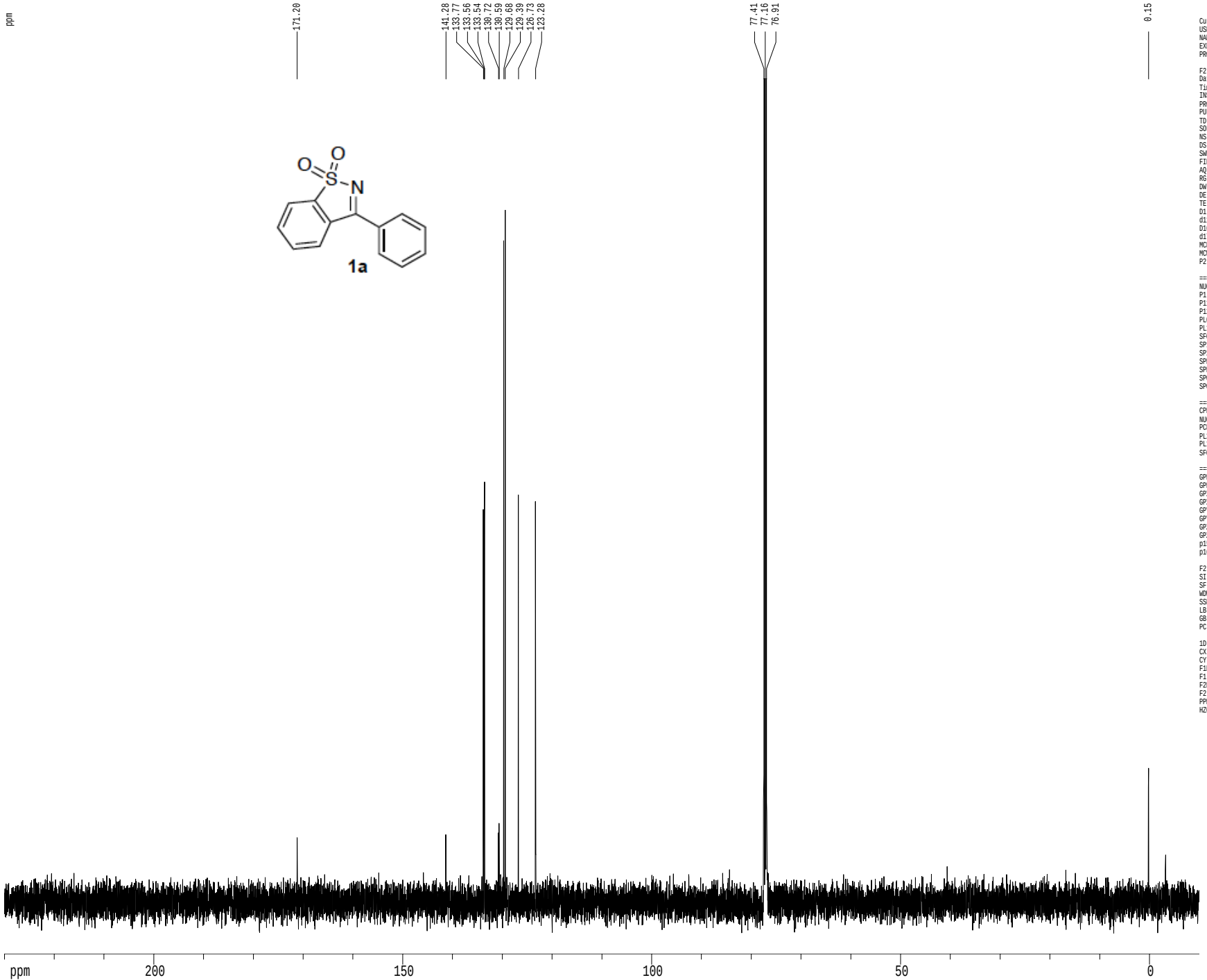

```
Current Data Parameters
USER              endean
NAME     TBDE-I-181-prop-pure-char
EXPNO                  2
PROCNO                 1

F2 - Acquisition Parameters
Date_          20150510
Time              17.36
INSTRUM         cryo500
PROBHD   5 mm CPTCI 1H-
PULPROG  SpinEchopg30gp.prd
TD                65536
SOLVENT           CDCl3
NS                  288
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
MCWRK         0.01500000 sec
P2                33.10 usec

======== CHANNEL f1 ========
NUC1                13C
P1                16.55 usec
P11              500.00 usec
P12             2000.00 usec
PL0              120.00 dB
PL1               -1.00 dB
SFO1         125.7942548 MHz
SP1               2.70 dB
SP2               2.70 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              24.50 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          125.7804085 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              230.000 ppm
F1             28929.49 Hz
F2P              -10.000 ppm
F2             -1257.80 Hz
PPMCM          10.52632 ppm/cm
HZCM         1324.00439 Hz/cm
```

1H spectrum



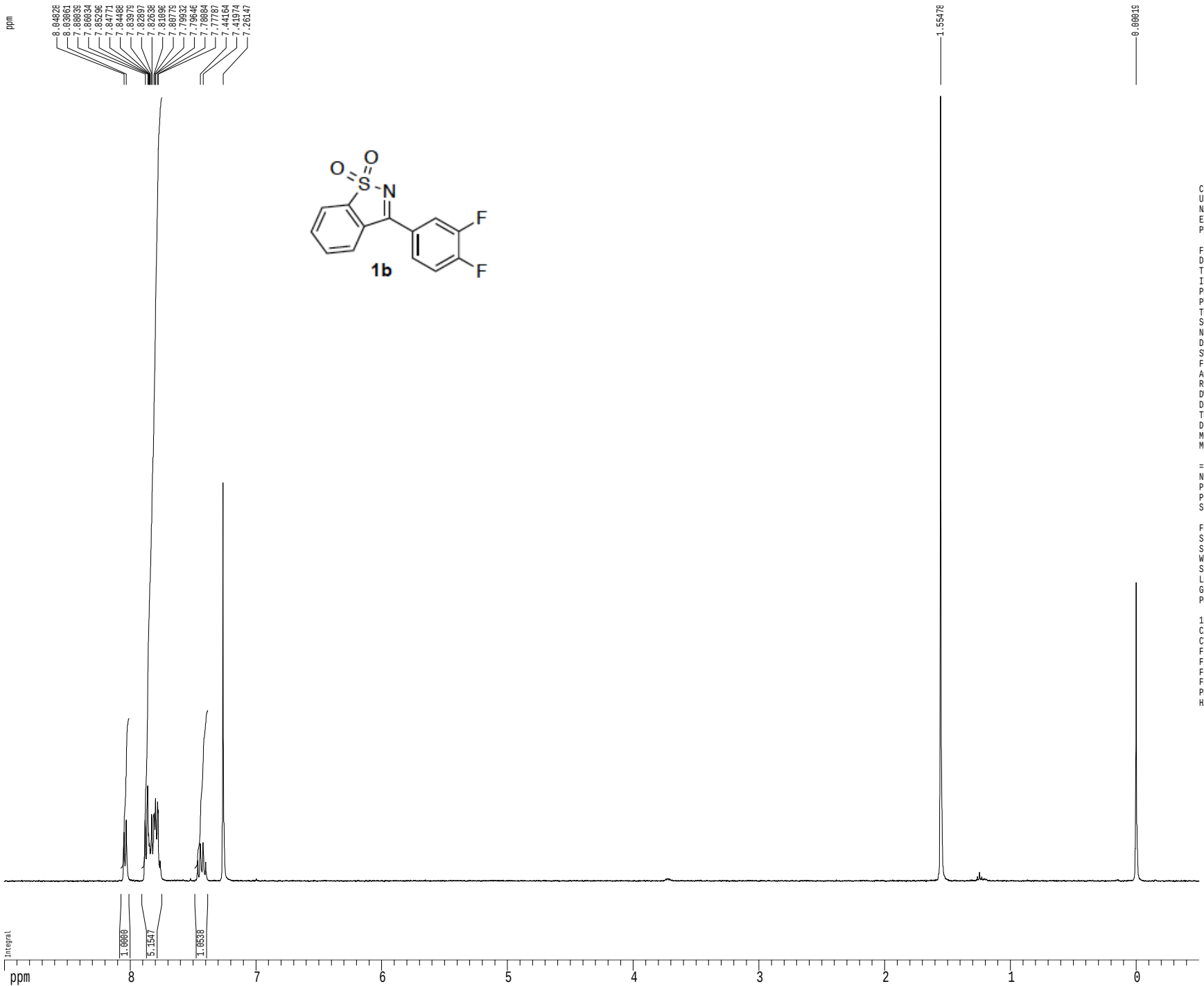

```
Current Data Parameters
USER                endean
NAME      TBDE-I-96-charicterization
EXPNO                  1
PROCNO                 1

F2 - Acquisition Parameters
Date_           20141116
Time                15.12
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                    5.7
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            500.2200327 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




```
Current Data Parameters
USER              endean
NAME     TBDE-I-96-charicterization
EXPNO                  2
PROCNO                 1

F2 - Acquisition Parameters
Date_           20141116
Time              15.16
INSTRUM         cryo500
PROBHD   5 mm CPTCI 1H-
PULPROG  SpinEchopg30gp.prd
TD                65536
SOLVENT            CDCl3
NS                  1024
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
MCWRK        0.01500000 sec
P2                33.10 usec

======= CHANNEL f1 =======
NUC1                13C
P1                16.55 usec
P11              500.00 usec
P12             2000.00 usec
PL0              120.00 dB
PL1               -1.00 dB
SFO1         125.7942548 MHz
SP1                2.70 dB
SP2                2.70 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              24.50 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           125.7804005 MHz
WDW                    EM
SSB                     0
LB                 1.00 Hz
GB                      0
PC                 2.00

1D NMR plot parameters
CX                 22.80 cm
CY                 60.00 cm
F1P              230.000 ppm
F1             28929.49 Hz
F2P               -10.000 ppm
F2             -1257.80 Hz
PPMCM          10.52632 ppm/cm
HZCM         1324.00439 Hz/cm
```

1H spectrum



Current Data Parameters
USER endean
NAME TBDE-I-51-recrys
EXPNO 1
PROCNO 1

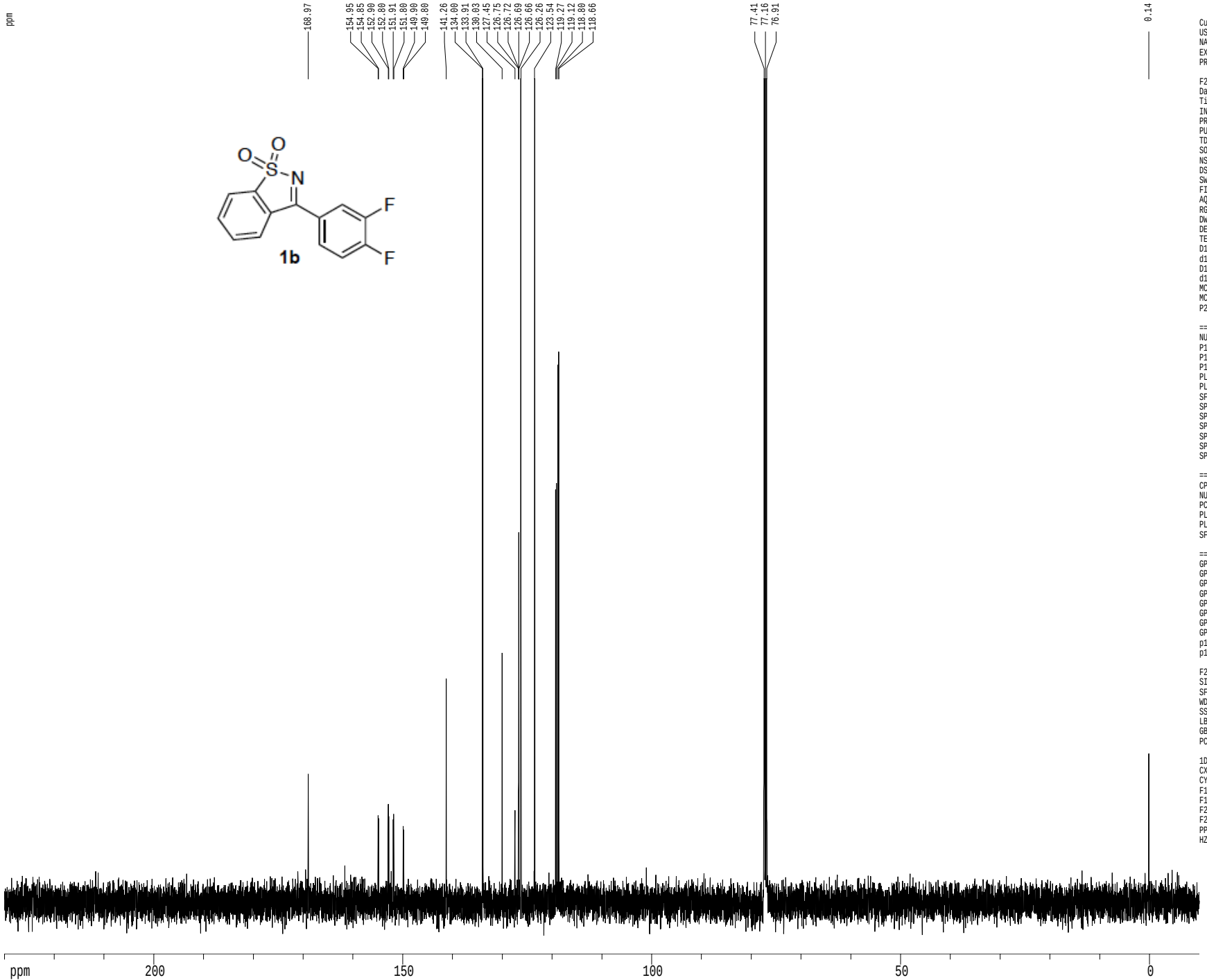
F2 - Acquisition Parameters
Date_ 20141009
Time 11.57
INSTRUM drx400
PROBHD 5 mm QNP H/F/P
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 8
DS 2
SWH 6410.256 Hz
FIDRES 0.097813 Hz
AQ 5.1118579 sec
RG 1290.2
DW 78.000 usec
DE 4.50 usec
TE 298.0 K
D1 0.10000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec

======== CHANNEL f1 ========
NUC1 1H
P1 12.00 usec
PL1 0.00 dB
SFO1 400.1328009 MHz

F2 - Processing parameters
SI 65536
SF 400.1300201 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 2.00

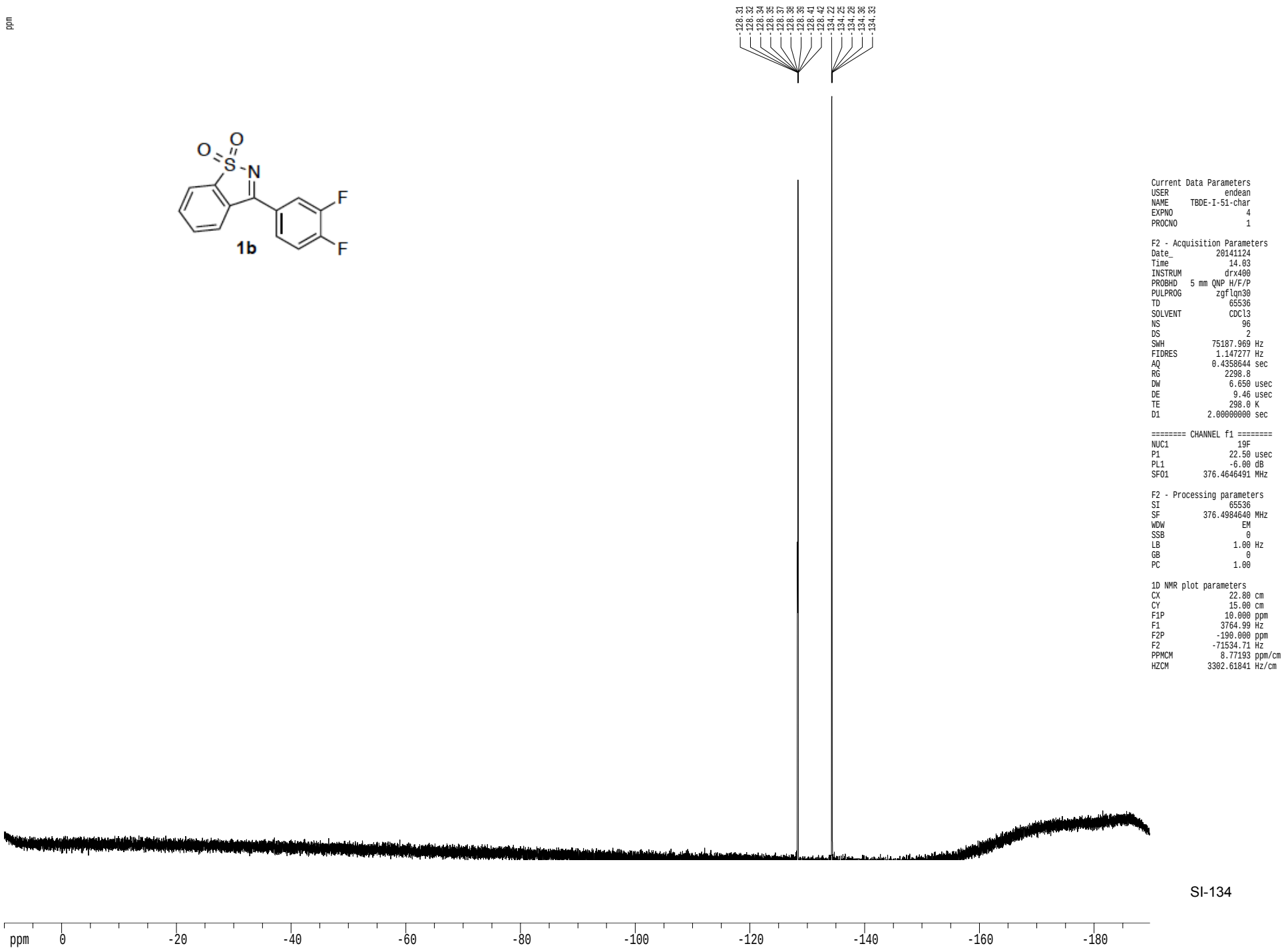
1D NMR plot parameters
CX 22.80 cm
CY 15.00 cm
F1P 9.000 ppm
F1 3601.17 Hz
F2P -0.500 ppm
F2 -200.06 Hz
PPMCM 0.41667 ppm/cm
HZCM 166.72084 Hz/cm

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




```
Current Data Parameters
USER                 endean
NAME      TBDE-I-51-char
EXPNO                     2
PROCNO                    1

F2 - Acquisition Parameters
Date_             20141124
Time                 13.05
INSTRUM            cryo500
PROBHD   5 mm CPTCI 1H-
PULPROG  SpinEchopg30gp.prd
TD                   65536
SOLVENT              CDCl3
NS                    1024
DS                      16
SWH              30303.031 Hz
FIDRES           0.462388 Hz
AQ               1.0813940 sec
RG                  2298.8
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                 33.10 usec

======== CHANNEL f1 ========
NUC1                  13C
P1                 16.55 usec
P11               500.00 usec
P12              2000.00 usec
PL0               120.00 dB
PL1                -1.00 dB
SFO1         125.7942548 MHz
SP1                2.70 dB
SP2                2.70 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               24.50 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           125.7804099 MHz
WDW                     EM
SSB                      0
LB                    1.00 Hz
GB                       0
PC                    2.00

1D NMR plot parameters
CX                   22.80 cm
CY                   60.00 cm
F1P                230.000 ppm
F1                28929.49 Hz
F2P                -10.000 ppm
F2                -1257.80 Hz
PPMCM             10.52632 ppm/cm
HZCM           1324.00439 Hz/cm
```

19F spectrum



Current Data Parameters
USER endean
NAME TBDE-I-51-char
EXPNO 4
PROCNO 1

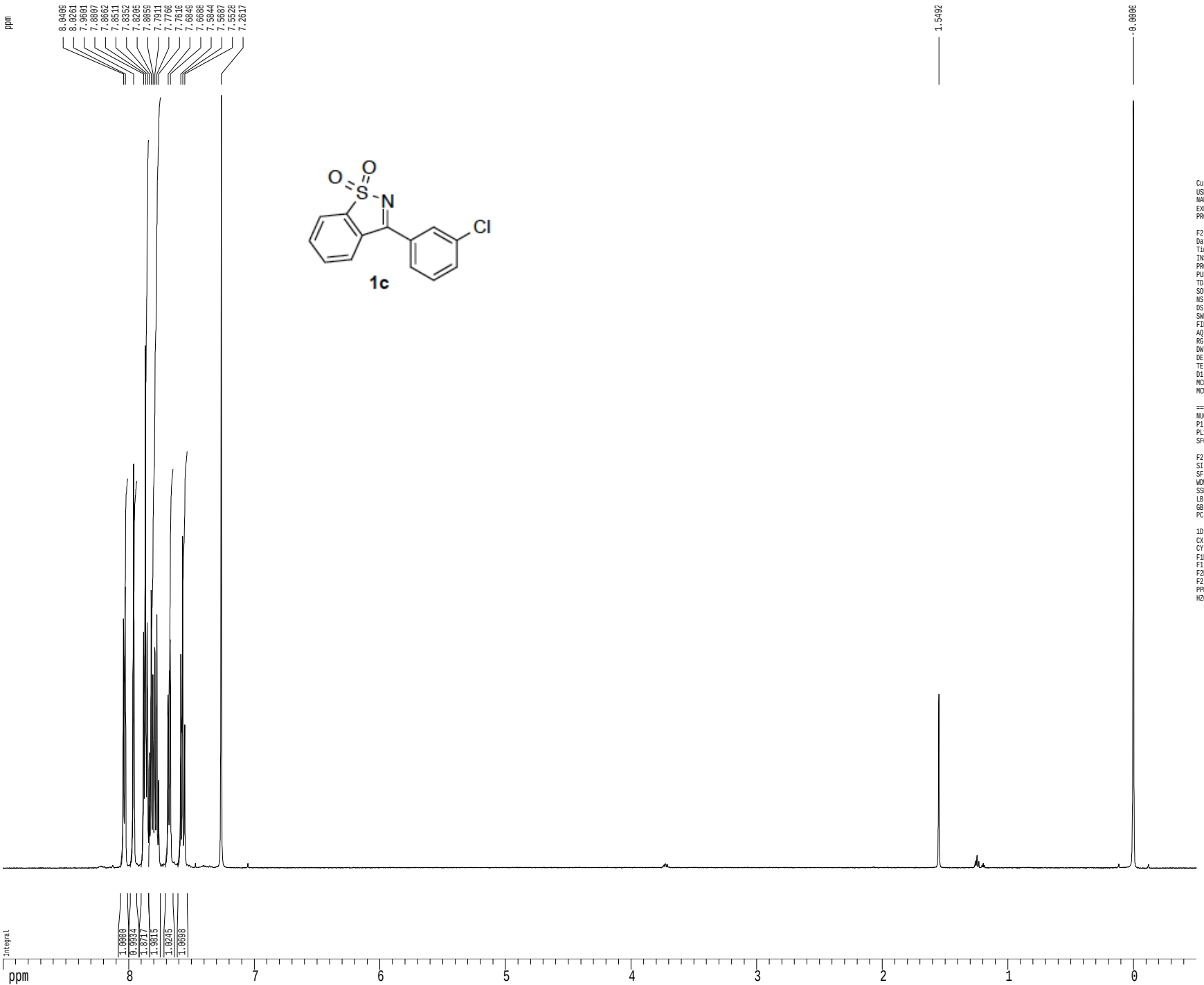
F2 - Acquisition Parameters
Date_ 20141124
Time 14.03
INSTRUM drx400
PROBHD 5 mm QNP H/F/P
PULPROG zgflqn30
TD 65536
SOLVENT CDCl3
NS 96
DS 2
SWH 75187.969 Hz
FIDRES 1.147277 Hz
AQ 0.4358644 sec
RG 2298.8
DW 6.650 usec
DE 9.46 usec
TE 298.0 K
D1 2.00000000 sec

======== CHANNEL f1 ========
NUC1 19F
P1 22.50 usec
PL1 -6.00 dB
SFO1 376.4646491 MHz

F2 - Processing parameters
SI 65536
SF 376.4984640 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.00

1D NMR plot parameters
CX 22.80 cm
CY 15.00 cm
F1P 10.000 ppm
F1 3764.99 Hz
F2P -190.000 ppm
F2 -71534.71 Hz
PPMCM 8.77193 ppm/cm
HZCM 3302.61841 Hz/cm

1H spectrum



Current Data Parameters
USER endean
NAME TBDE-I-81-charicterization
EXPNO 1
PROCNO 1

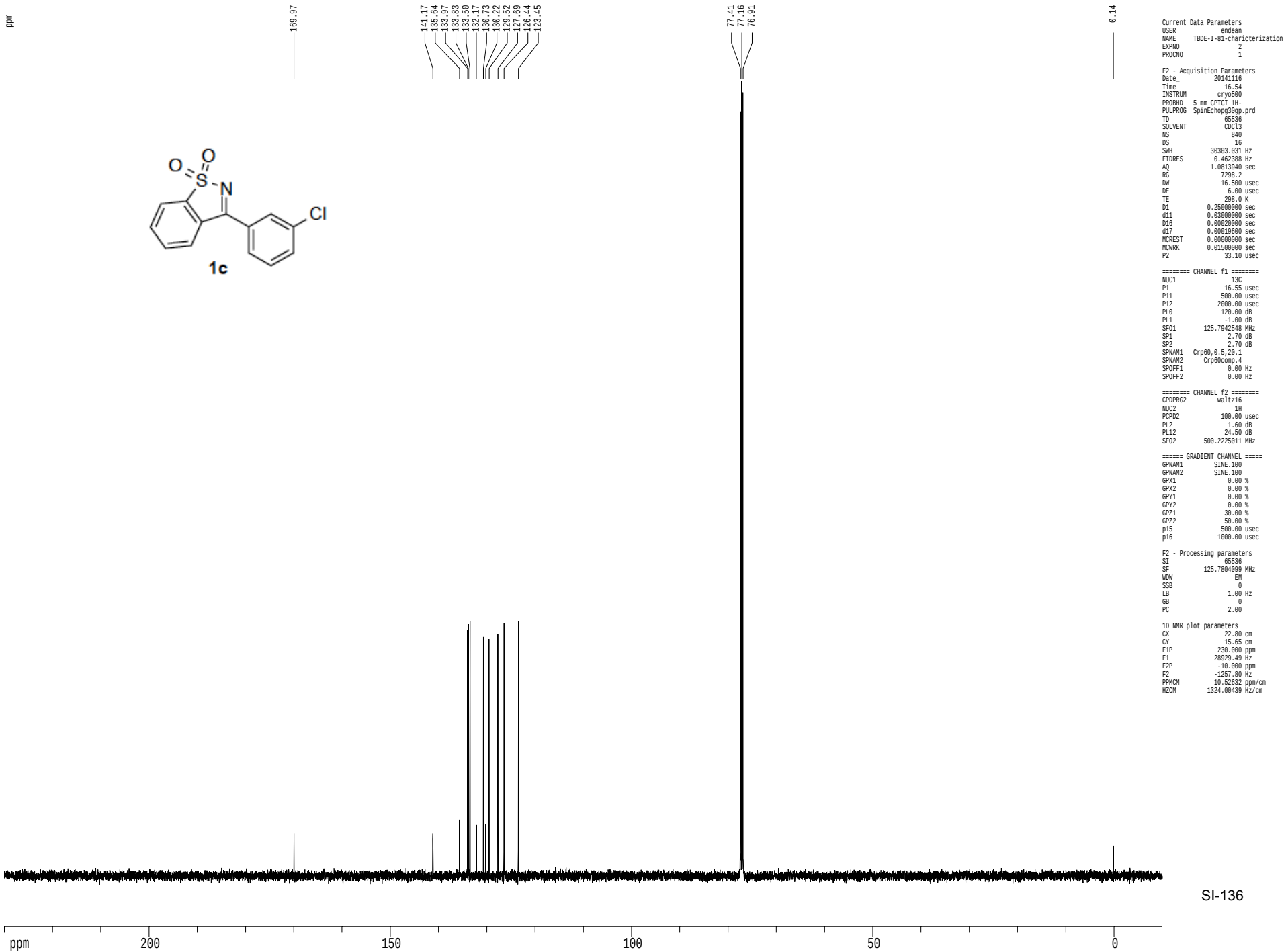
F2 - Acquisition Parameters
Date_ 20141116
Time 16.44
INSTRUM cryo500
PROBHD 5 mm CPTCI 1H-
PULPROG zg30
TD 81728
SOLVENT CDCl3
NS 8
DS 2
SWH 8012.820 Hz
FIDRES 0.098043 Hz
AQ 5.0998774 sec
RG 6.3
DW 62.400 usec
DE 6.00 usec
TE 298.0 K
D1 0.10000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec

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

F2 - Processing parameters
SI 65536
SF 500.2200326 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 4.00

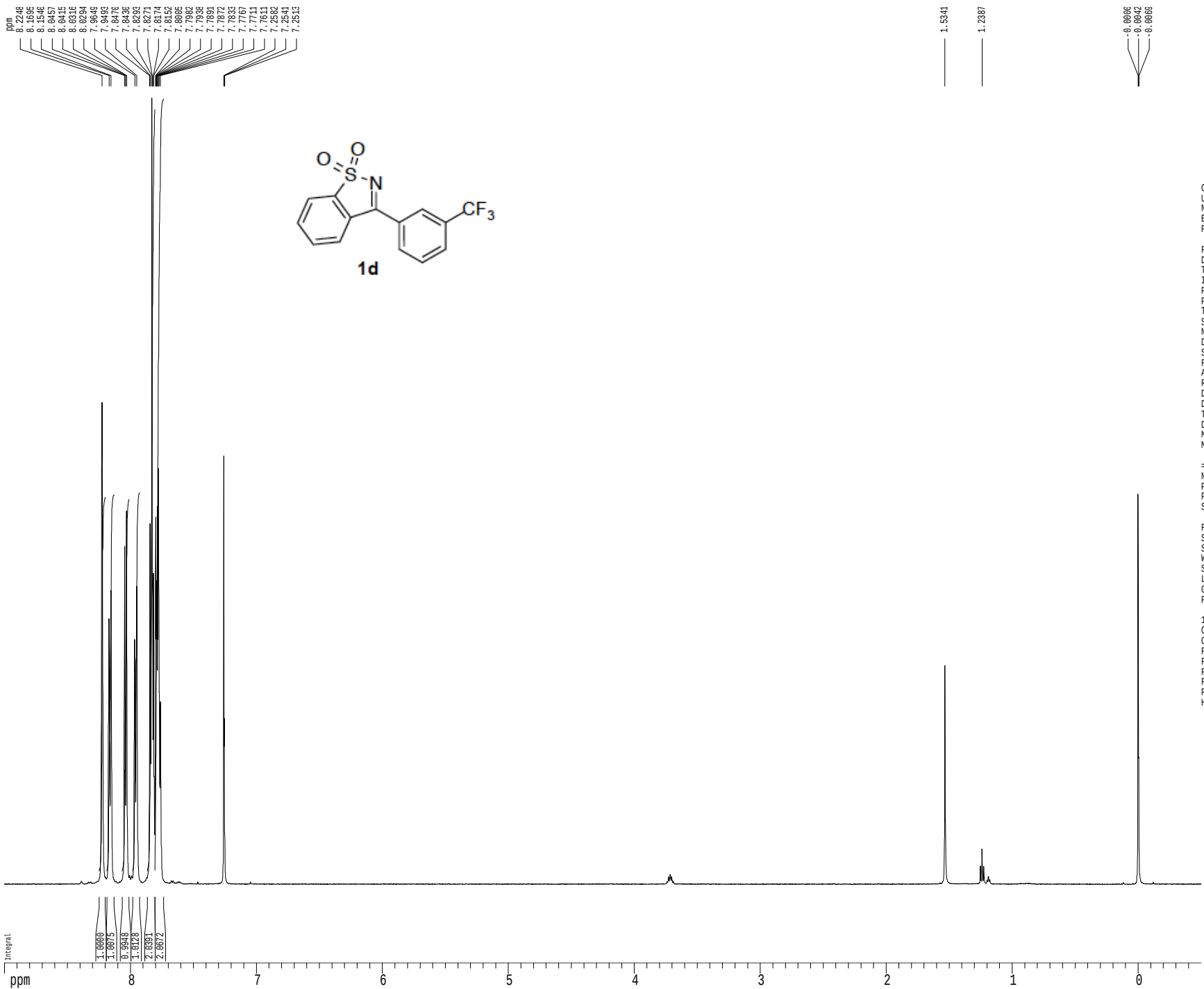
1D NMR plot parameters
CX 22.80 cm
CY 15.00 cm
F1P 9.000 ppm
F1 4501.98 Hz
F2P -0.500 ppm
F2 -250.11 Hz
PPMCM 0.41667 ppm/cm
HZCM 208.42502 Hz/cm

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



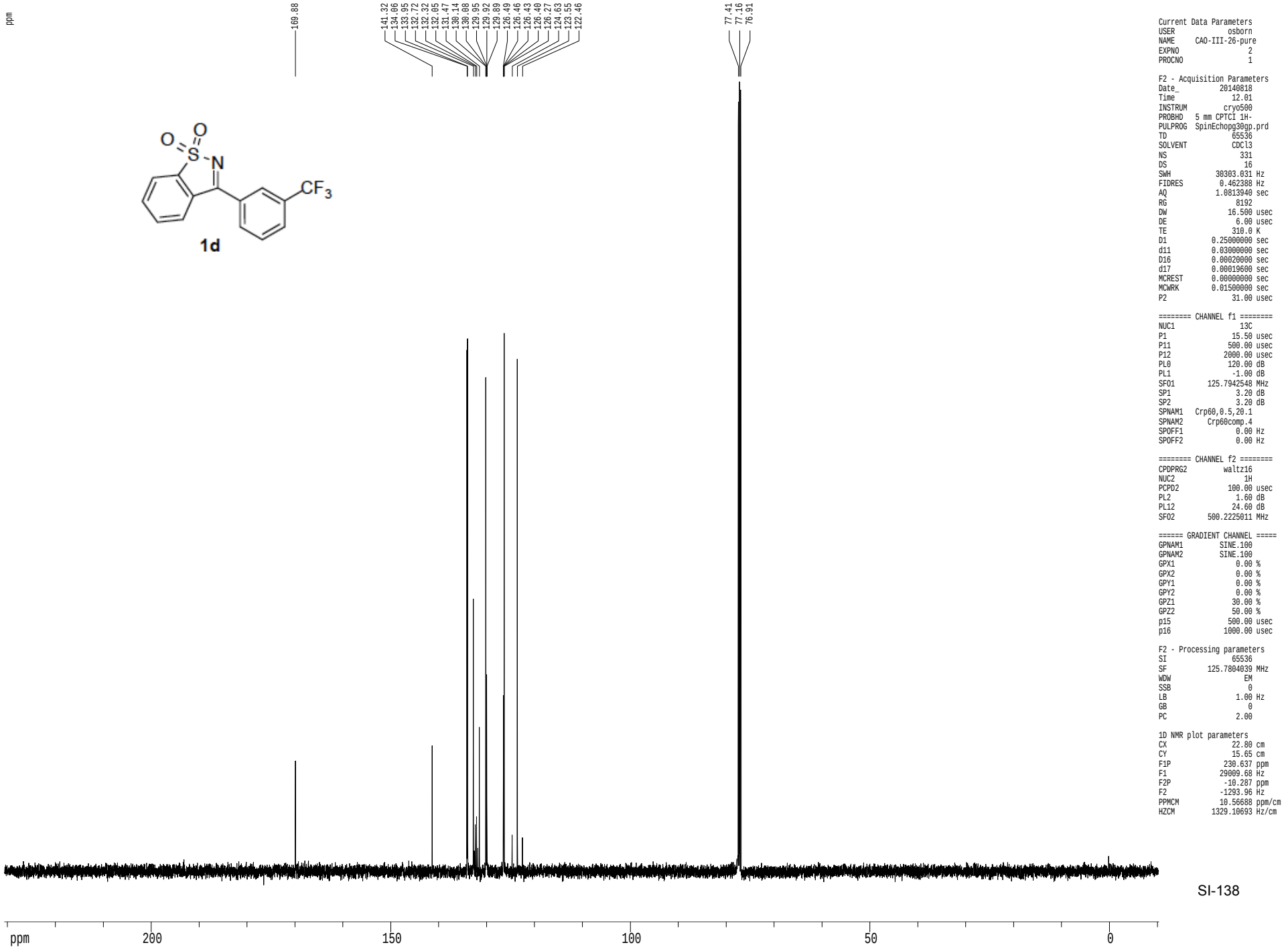

```
Current Data Parameters
USER              endean
NAME     TBDE-I-81-charicterization
EXPNO                  2
PROCNO                 1

F2 - Acquisition Parameters
Date_           20141116
Time              16.54
INSTRUM         cryo500
PROBHD   5 mm CPTCI 1H-
PULPROG  SpinEchopg30gp.prd
TD                65536
SOLVENT            CDCl3
NS                   840
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
MCWRK        0.01500000 sec
P2               33.10 usec

======= CHANNEL f1 =======
NUC1               13C
P1               16.55 usec
P11             500.00 usec
P12            2000.00 usec
PL0             120.00 dB
PL1              -1.00 dB
SFO1        125.7942548 MHz
SP1               2.70 dB
SP2               2.70 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             24.50 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          125.7804099 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             230.000 ppm
F1            28929.49 Hz
F2P             -10.000 ppm
F2            -1257.80 Hz
PPMCM         10.52632 ppm/cm
HZCM        1324.00439 Hz/cm
```

1H spectrum



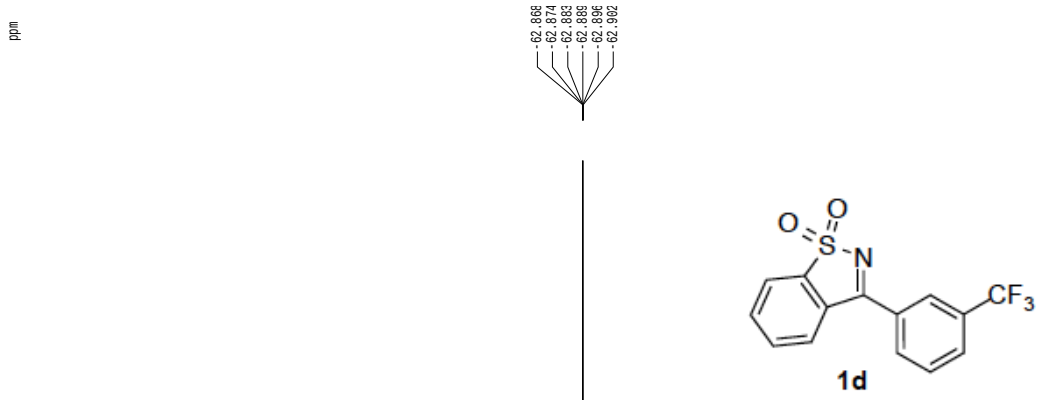

```
Current Data Parameters
USER                osborn
NAME      CAO-III-26-pure
EXPNO                    1
PROCNO                   1

F2 - Acquisition Parameters
Date_            20140818
Time                11.59
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                    6.3
DW                 62.400 usec
DE                  6.00 usec
TE                 310.0 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            500.2200319 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

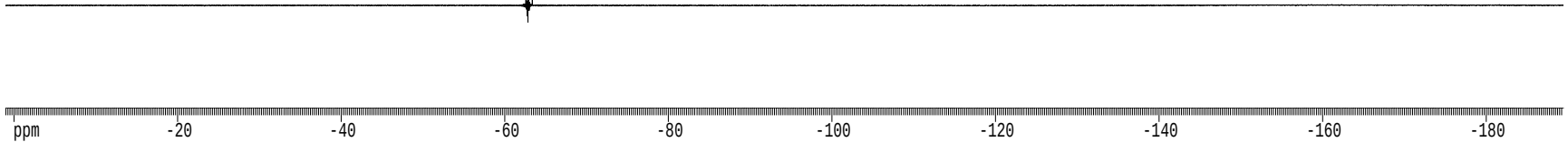



```
Current Data Parameters
USER              osborn
NAME      CAO-III-26-pure
EXPNO                  2
PROCNO                 1

F2 - Acquisition Parameters
Date_          20140818
Time              12.01
INSTRUM         cryo500
PROBHD   5 mm CPTCI 1H-
PULPROG  SpinEchopg30gp.prd
TD                65536
SOLVENT           CDCl3
NS                  331
DS                   16
SWH           30303.031 Hz
FIDRES        0.462388 Hz
AQ            1.0813940 sec
RG                 8192
DW               16.500 usec
DE                6.00 usec
TE                310.0 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
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 ========
CPDPRG2          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          125.7804039 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             230.637 ppm
F1            29009.68 Hz
F2P             -10.287 ppm
F2            -1293.96 Hz
PPMCM          10.56688 ppm/cm
HZCM         1329.10693 Hz/cm
```

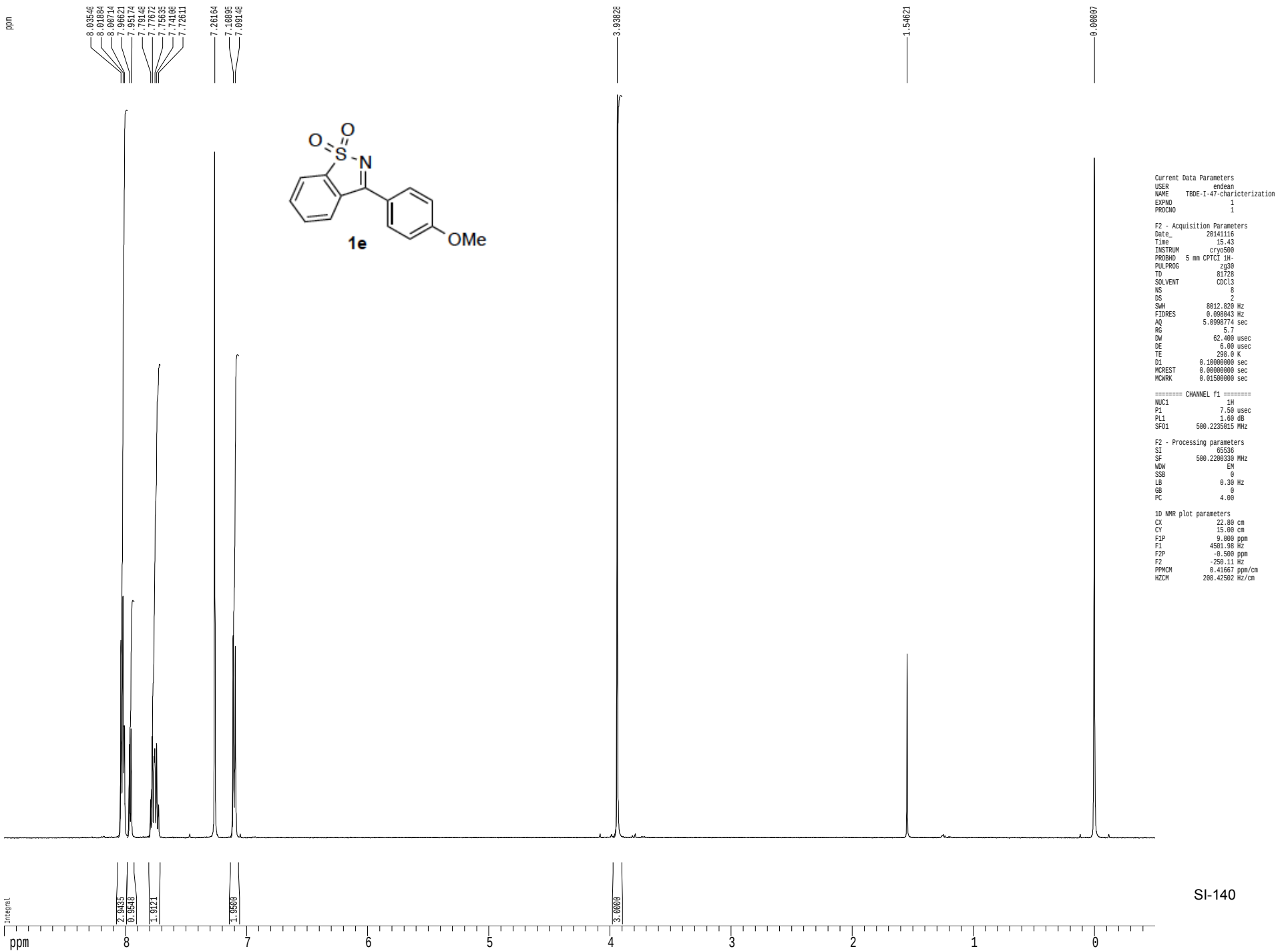
19F spectrum with 1H decoupling




```
Current Data Parameters
USER              osborn
NAME       CAO-III-26-SI
EXPNO                2
PROCNO               1

F2 - Acquisition Parameters
Date_          20150530
Time              19.49
INSTRUM          drx400
PROBHD   5 mm QNP H/F/P
PULPROG      zgfhigqn30
TD                65536
SOLVENT           CDCl3
NS                   31
DS                    4
SWH           75187.969 Hz
FIDRES         1.147277 Hz
AQ            0.4358644 sec
RG               3649.1
DW                6.650 usec
DE                9.46 usec
TE                298.0 K
D1           2.00000000 sec
d11          0.03000000 sec
d12          0.00002000 sec

======== CHANNEL f1 ========
NUC1                 19F
P1                22.50 usec
PL1               -6.00 dB
SFO1        376.4646491 MHz

======== CHANNEL f2 ========
CPDPRG2          waltz16
NUC2                 1H
PCPD2             90.00 usec
PL2              120.00 dB
PL12              17.70 dB
SFO2        400.1320007 MHz

F2 - Processing parameters
SI                65536
SF          376.4983851 MHz
WDW                  EM
SSB                   0
LB                 0.30 Hz
GB                    0
PC                 1.00

1D NMR plot parameters
CX                22.80 cm
CY                15.00 cm
F1P               1.000 ppm
F1              376.50 Hz
F2P            -190.000 ppm
F2           -71534.70 Hz
PPMCM          8.37719 ppm/cm
HZCM         3153.99976 Hz/cm
```

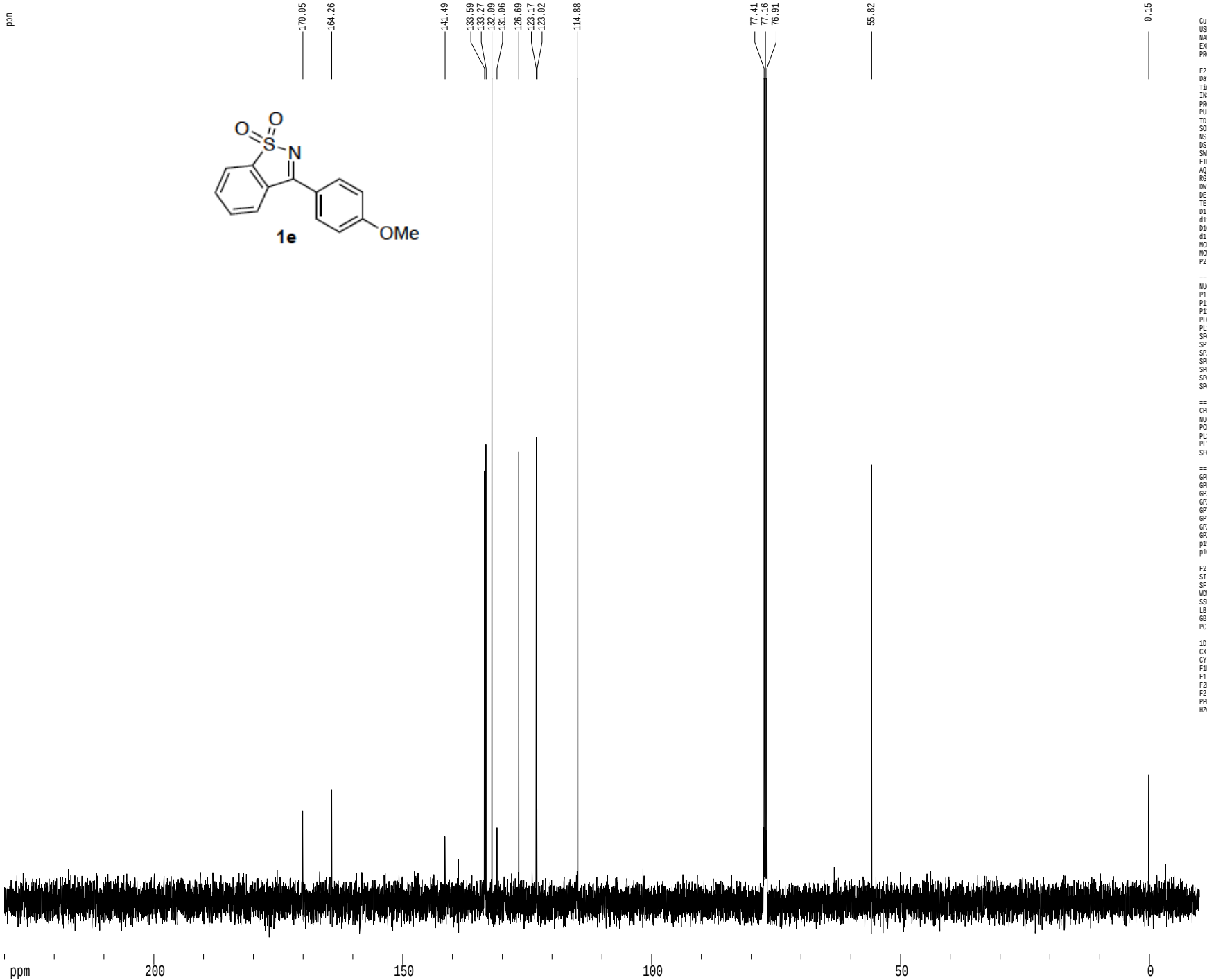


1H spectrum



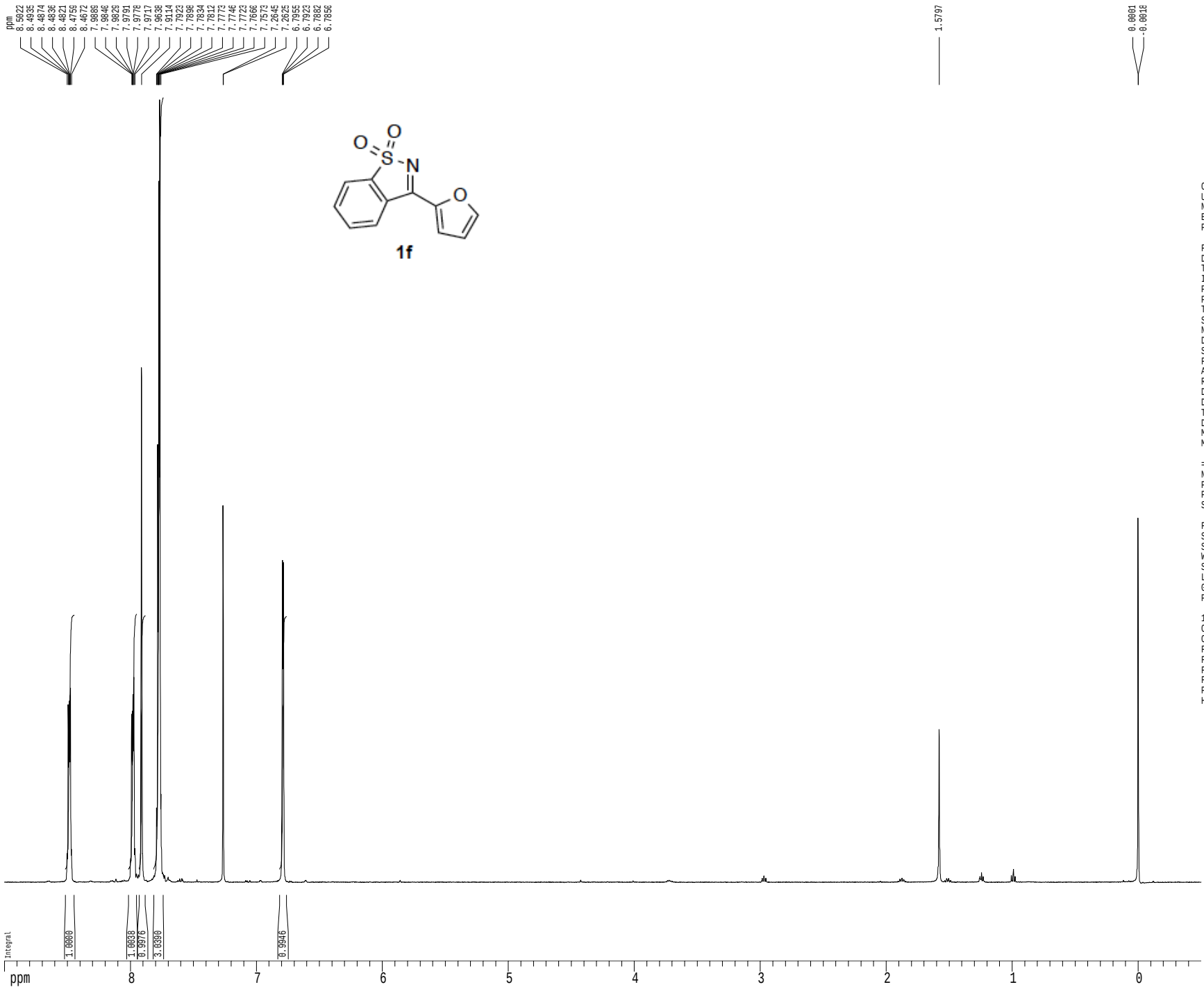

```
Current Data Parameters
USER                endean
NAME      TBDE-I-47-charicterization
EXPNO                    1
PROCNO                   1

F2 - Acquisition Parameters
Date_            20141116
Time                15.43
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                   5.7
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          500.2200330 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



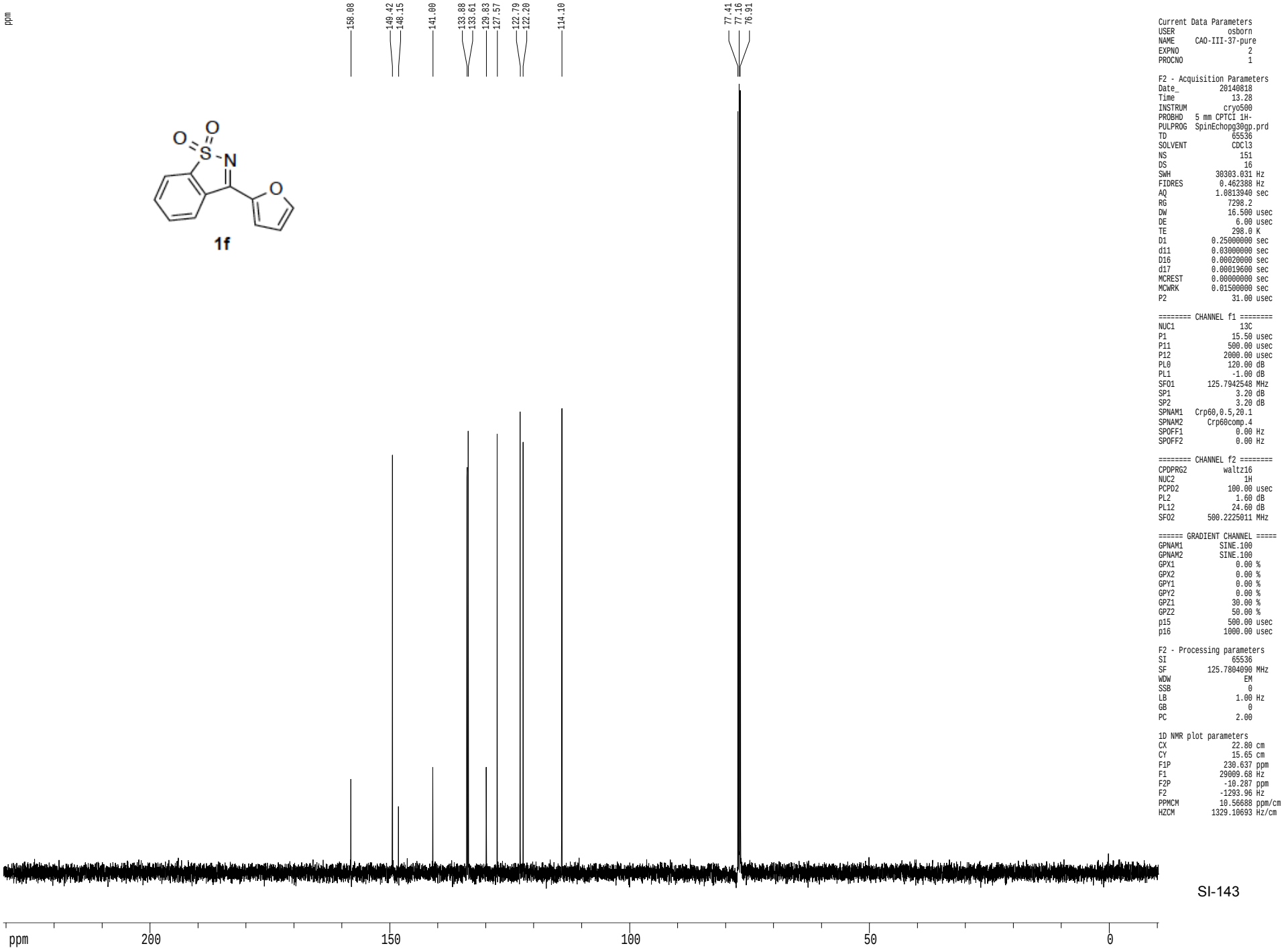

```
Current Data Parameters
USER              endean
NAME     TBDE-I-47-charicterization
EXPNO                  2
PROCNO                 1

F2 - Acquisition Parameters
Date_           20141116
Time              15.53
INSTRUM         cryo500
PROBHD   5 mm CPTCI 1H-
PULPROG  SpinEchopg30gp.prd
TD                65536
SOLVENT           CDCl3
NS                 1024
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
MCWRK        0.01500000 sec
P2                33.10 usec

======== CHANNEL f1 ========
NUC1               13C
P1                16.55 usec
P11              500.00 usec
P12             2000.00 usec
PL0              120.00 dB
PL1               -1.00 dB
SFO1         125.7942548 MHz
SP1               2.70 dB
SP2               2.70 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              24.50 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           125.7804094 MHz
WDW                   EM
SSB                    0
LB                  1.00 Hz
GB                     0
PC                  2.00

1D NMR plot parameters
CX                 22.80 cm
CY                 60.00 cm
F1P              230.000 ppm
F1             28929.49 Hz
F2P              -10.000 ppm
F2             -1257.80 Hz
PPMCM          10.52632 ppm/cm
HZCM         1324.00439 Hz/cm
```

1H spectrum



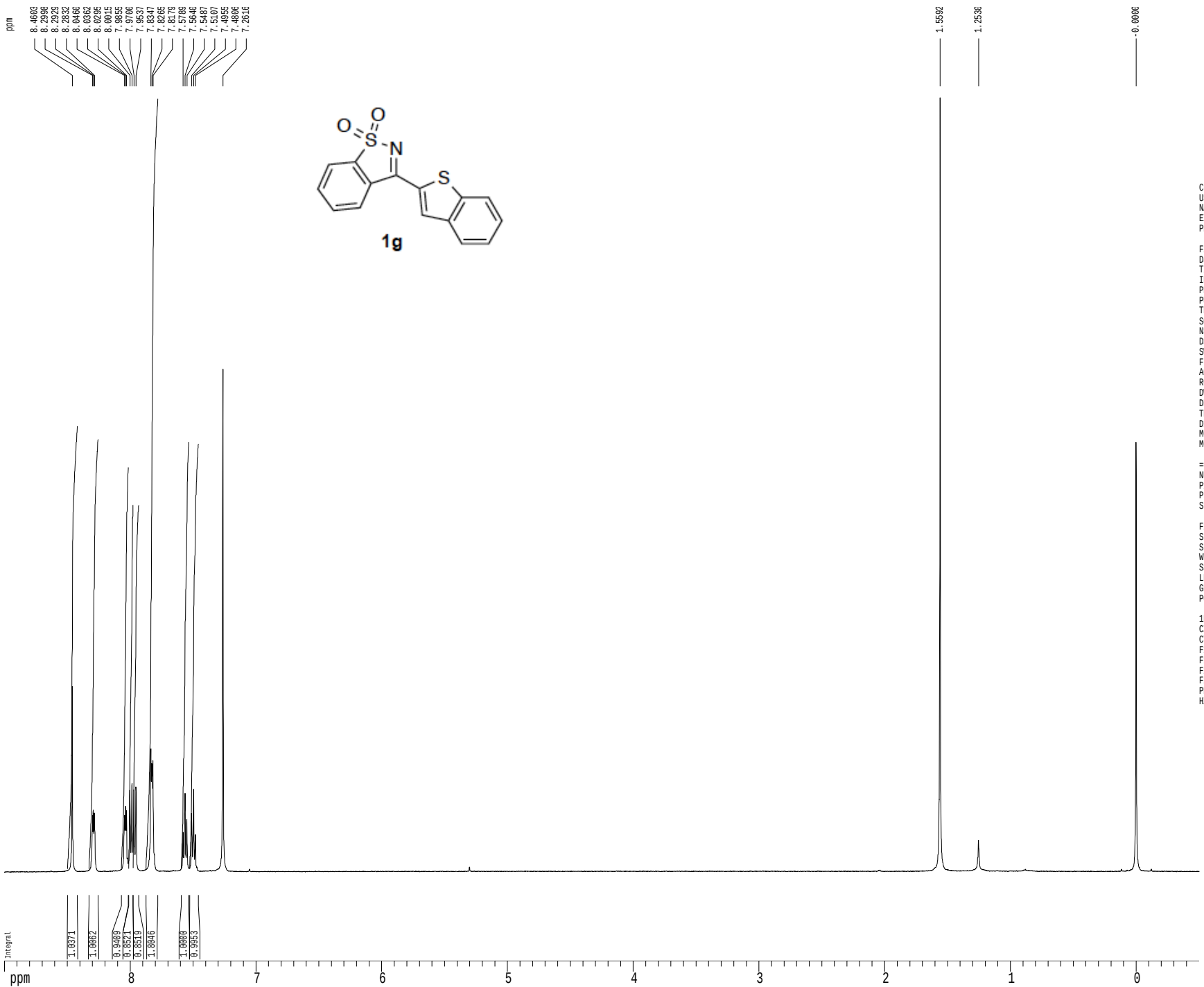

```
Current Data Parameters
USER                osborn
NAME      CAO-III-37-pure
EXPNO                    1
PROCNO                   1

F2 - Acquisition Parameters
Date_            20140818
Time                13.26
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                    6.3
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            500.2200292 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



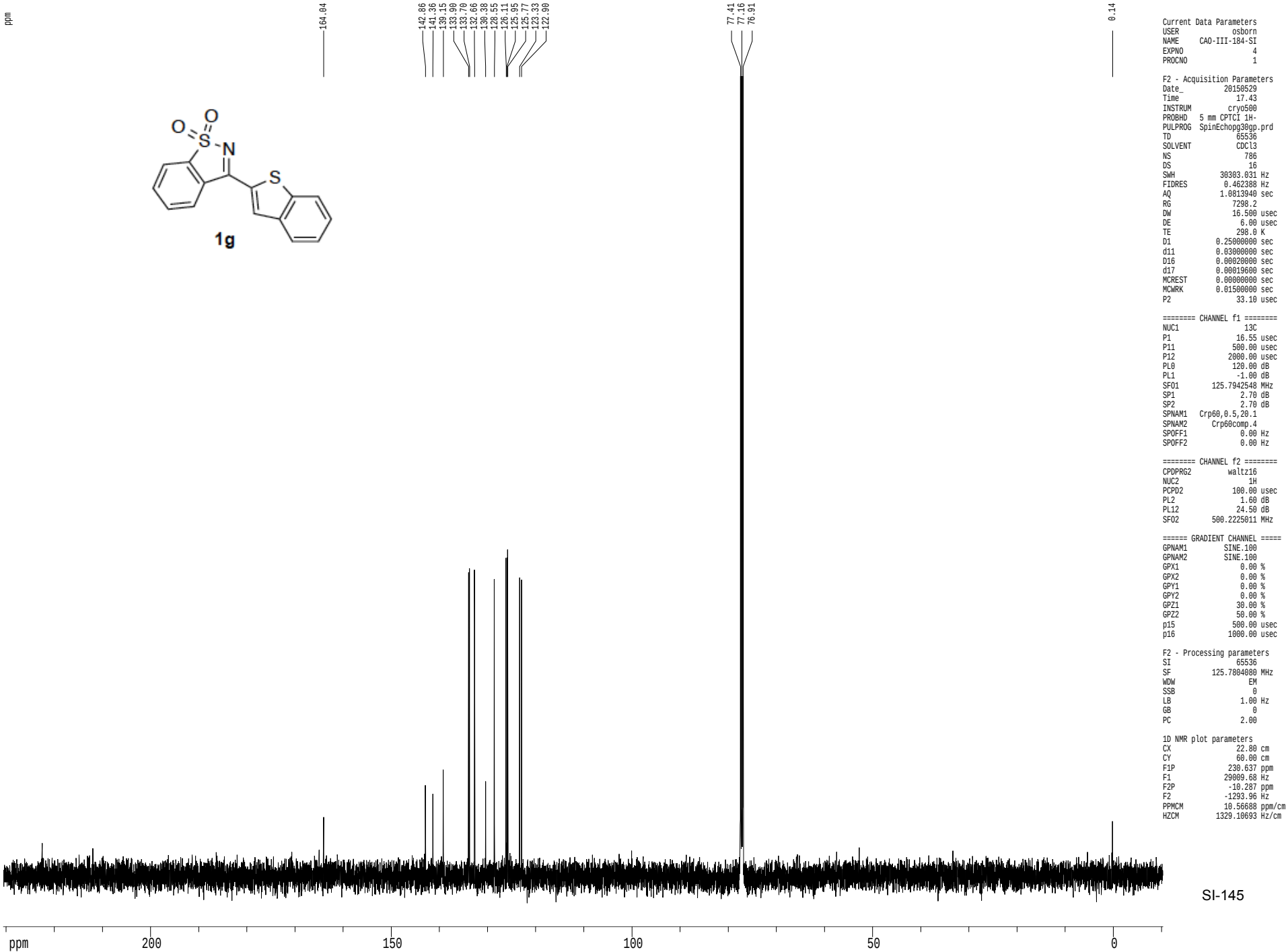

```
Current Data Parameters
USER              osborn
NAME      CAO-III-37-pure
EXPNO                  2
PROCNO                 1

F2 - Acquisition Parameters
Date_           20140818
Time               13.28
INSTRUM          cryo500
PROBHD   5 mm CPTCI 1H-
PULPROG  SpinEchopg30gp.prd
TD                 65536
SOLVENT            CDCl3
NS                   151
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
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
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 ========
CPDPRG2          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

1D NMR plot parameters
CX                 22.80 cm
CY                 15.65 cm
F1P              230.637 ppm
F1             29009.68 Hz
F2P              -10.287 ppm
F2             -1293.96 Hz
PPMCM           10.56688 ppm/cm
HZCM          1329.10693 Hz/cm
```

1H spectrum



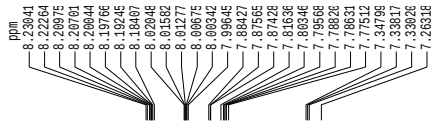

```
Current Data Parameters
USER                osborn
NAME      CAO-III-184-SI
EXPNO                  3
PROCNO                 1

F2 - Acquisition Parameters
Date_           20150529
Time               17.41
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                   6.3
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           500.2200309 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
```

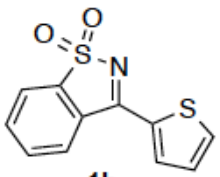
Z-restored spin-echo 13C spectrum with 1H decoupling




```
Current Data Parameters
USER                osborn
NAME      CAO-III-184-SI
EXPNO                  4
PROCNO                 1

F2 - Acquisition Parameters
Date_           20150529
Time               17.43
INSTRUM          cryo500
PROBHD   5 mm CPTCI 1H-
PULPROG  SpinEchopg30gp.prd
TD                 65536
SOLVENT            CDCl3
NS                   786
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
MCWRK          0.01500000 sec
P2                33.10 usec

======== CHANNEL f1 ========
NUC1                 13C
P1                16.55 usec
P11              500.00 usec
P12             2000.00 usec
PL0              120.00 dB
PL1               -1.00 dB
SFO1         125.7942548 MHz
SP1               2.70 dB
SP2               2.70 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              24.50 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           125.7804080 MHz
WDW                  EM
SSB                   0
LB                 1.00 Hz
GB                    0
PC                 2.00

1D NMR plot parameters
CX                22.80 cm
CY                60.00 cm
F1P              230.637 ppm
F1             29009.68 Hz
F2P              -10.287 ppm
F2             -1293.96 Hz
PPMCM           10.56688 ppm/cm
HZCM          1329.10693 Hz/cm
```

1H spectrum

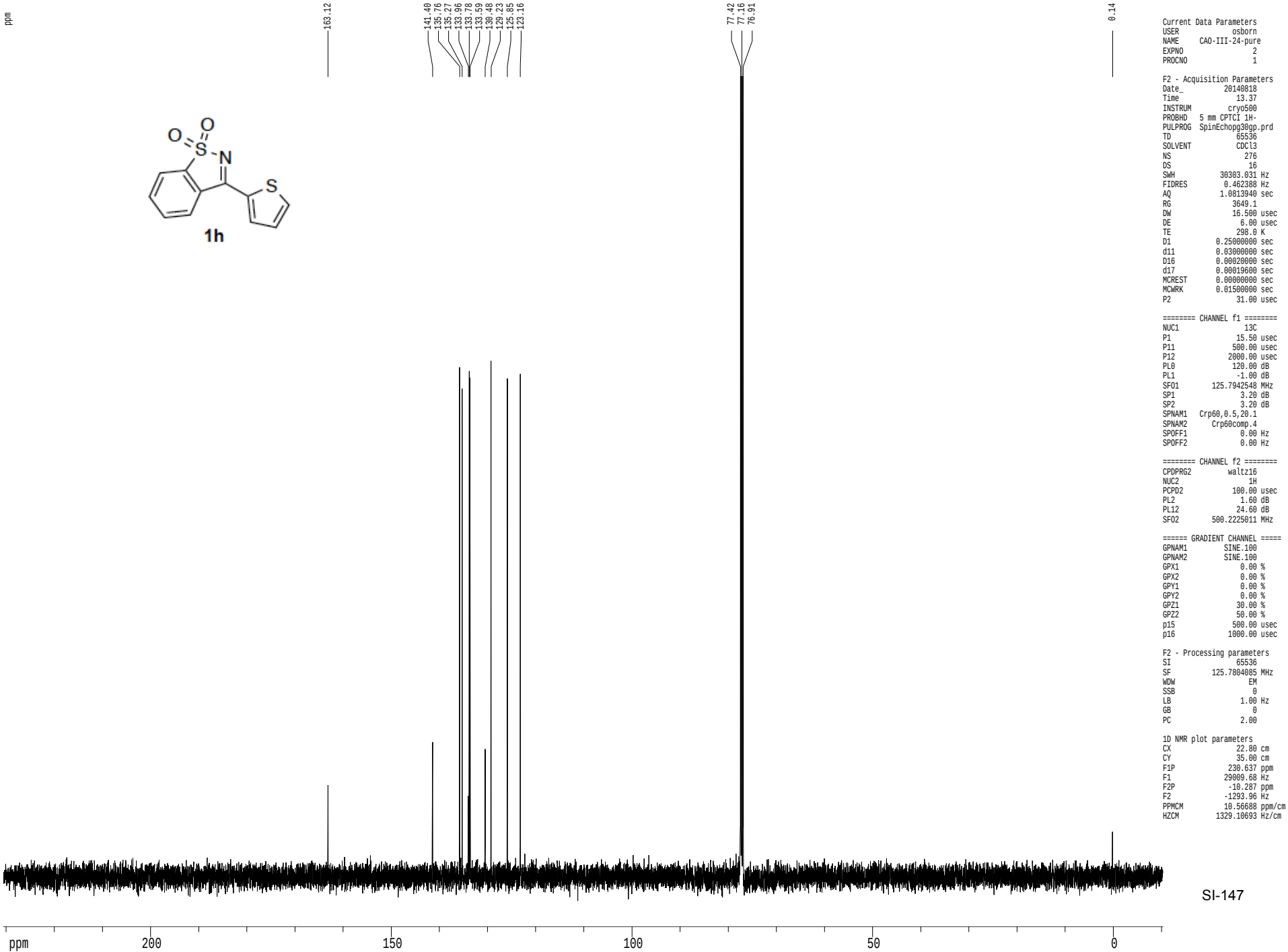




1h

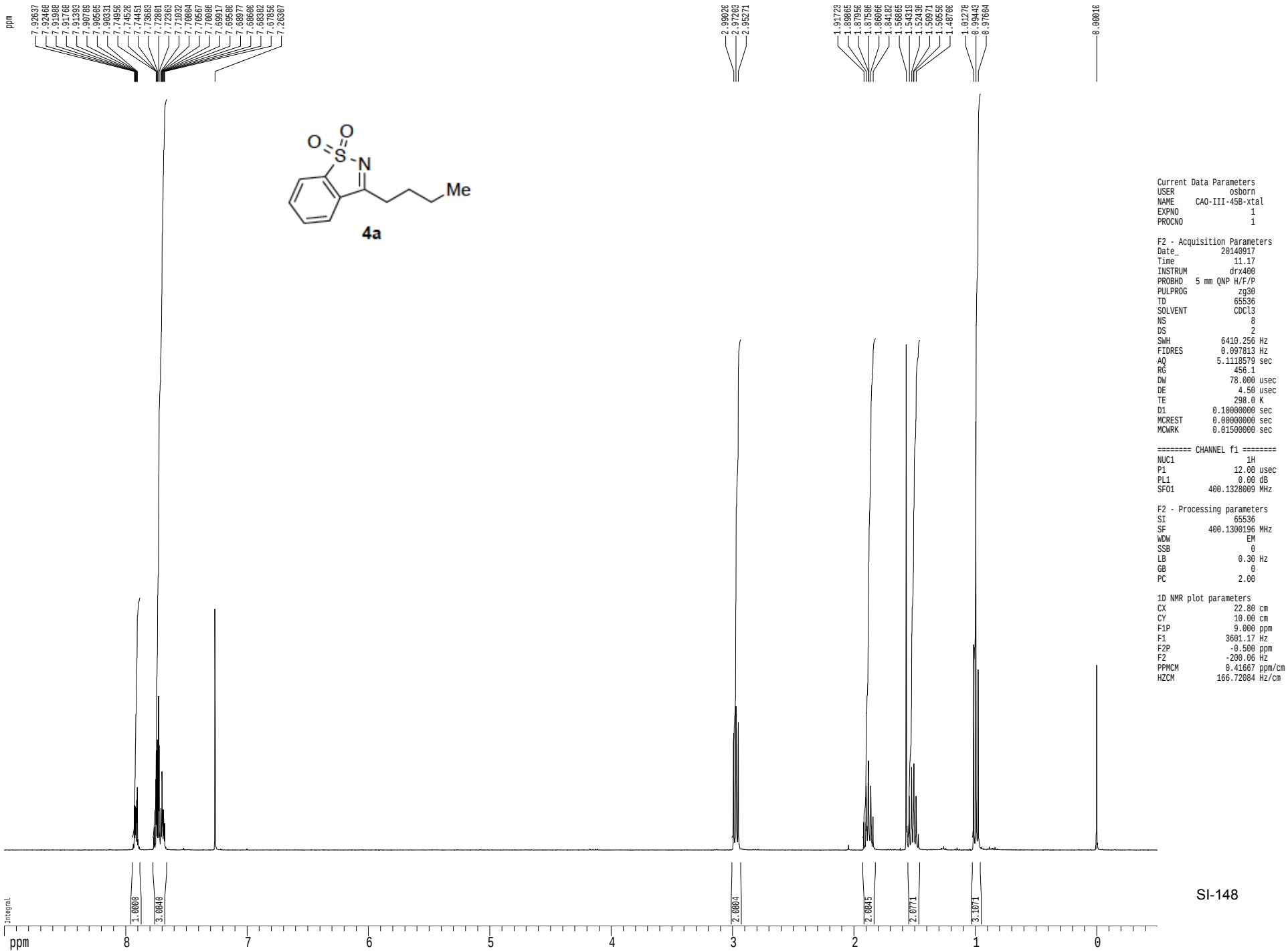

```
Current Data Parameters
USER                osborn
NAME      CAO-III-24-pure
EXPNO                    1
PROCNO                   1

F2 - Acquisition Parameters
Date_            20140818
Time                13.35
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               5.0998774 sec
RG                      8
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            500.2200296 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



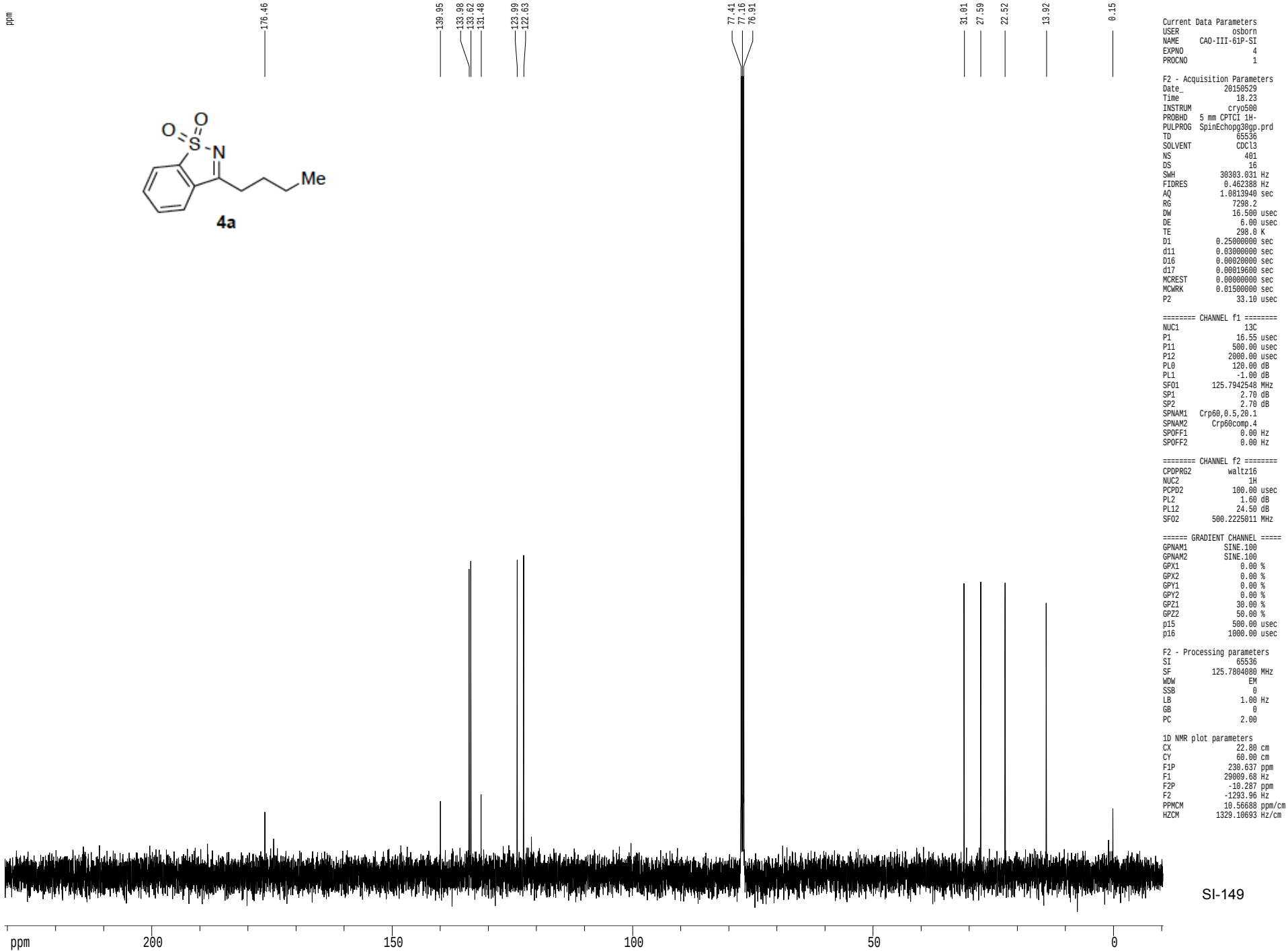

```
Current Data Parameters
USER            osborn
NAME      CAO-III-24-pure
EXPNO                 2
PROCNO                1

F2 - Acquisition Parameters
Date_          20140818
Time              13.37
INSTRUM         cryo500
PROBHD   5 mm CPTCI 1H-
PULPROG  SpinEchopg30gp.prd
TD                65536
SOLVENT           CDCl3
NS                  276
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
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
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 ========
CPDPRG2          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          125.7804085 MHz
WDW                   EM
SSB                    0
LB                  1.00 Hz
GB                     0
PC                  2.00

1D NMR plot parameters
CX                 22.80 cm
CY                 35.00 cm
F1P              230.637 ppm
F1             29009.68 Hz
F2P              -10.287 ppm
F2             -1293.96 Hz
PPMCM           10.56688 ppm/cm
HZCM          1329.10693 Hz/cm
```

1H spectrum



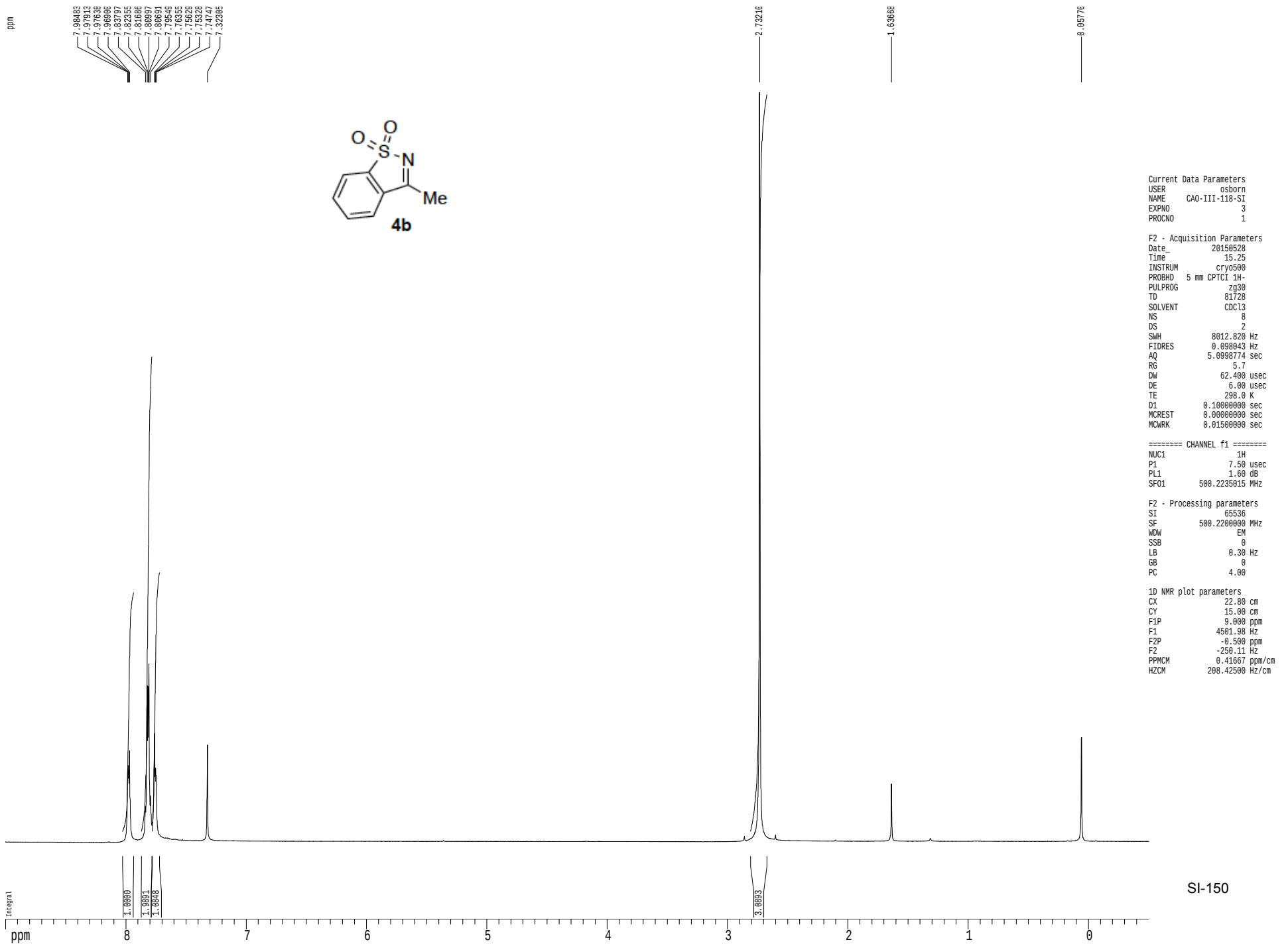

```
Current Data Parameters
USER              osborn
NAME      CAO-III-45B-xtal
EXPNO                1
PROCNO               1

F2 - Acquisition Parameters
Date_          20140917
Time              11.17
INSTRUM          drx400
PROBHD   5 mm QNP H/F/P
PULPROG           zg30
TD               65536
SOLVENT          CDCl3
NS                   8
DS                   2
SWH           6410.256 Hz
FIDRES        0.097813 Hz
AQ            5.1118579 sec
RG               456.1
DW               78.000 usec
DE                4.50 usec
TE               298.0 K
D1          0.10000000 sec
MCREST      0.00000000 sec
MCWRK       0.01500000 sec

======== CHANNEL f1 ========
NUC1                 1H
P1                12.00 usec
PL1                0.00 dB
SFO1        400.1328009 MHz

F2 - Processing parameters
SI                65536
SF          400.1300196 MHz
WDW                  EM
SSB                  0
LB                0.30 Hz
GB                   0
PC                2.00

1D NMR plot parameters
CX                22.80 cm
CY                10.00 cm
F1P               9.000 ppm
F1              3601.17 Hz
F2P              -0.500 ppm
F2              -200.06 Hz
PPMCM          0.41667 ppm/cm
HZCM          166.72084 Hz/cm
```

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



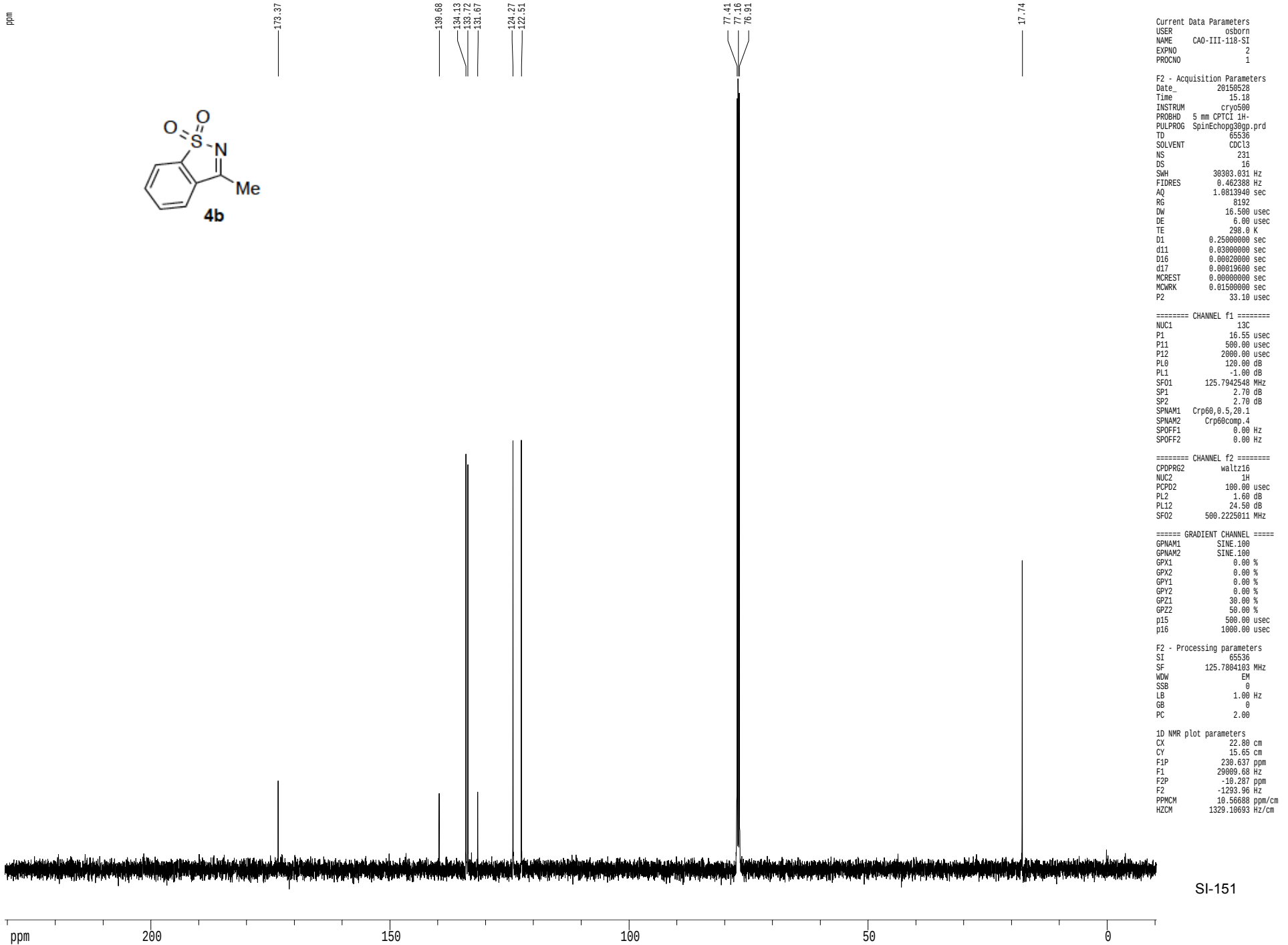

```
Current Data Parameters
USER                osborn
NAME      CAO-III-61P-SI
EXPNO                  4
PROCNO                 1

F2 - Acquisition Parameters
Date_           20150529
Time              18.23
INSTRUM          cryo500
PROBHD   5 mm CPTCI 1H-
PULPROG  SpinEchopg30gp.prd
TD                65536
SOLVENT           CDCl3
NS                  401
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
MCWRK         0.01500000 sec
P2              33.10 usec

======== CHANNEL f1 ========
NUC1                13C
P1               16.55 usec
P11             500.00 usec
P12            2000.00 usec
PL0             120.00 dB
PL1              -1.00 dB
SFO1        125.7942548 MHz
SP1              2.70 dB
SP2              2.70 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             24.50 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          125.7804080 MHz
WDW                 EM
SSB                  0
LB                1.00 Hz
GB                   0
PC                2.00

1D NMR plot parameters
CX               22.80 cm
CY               60.00 cm
F1P            230.637 ppm
F1           29009.68 Hz
F2P            -10.287 ppm
F2           -1293.96 Hz
PPMCM         10.56688 ppm/cm
HZCM        1329.10693 Hz/cm
```

1H spectrum



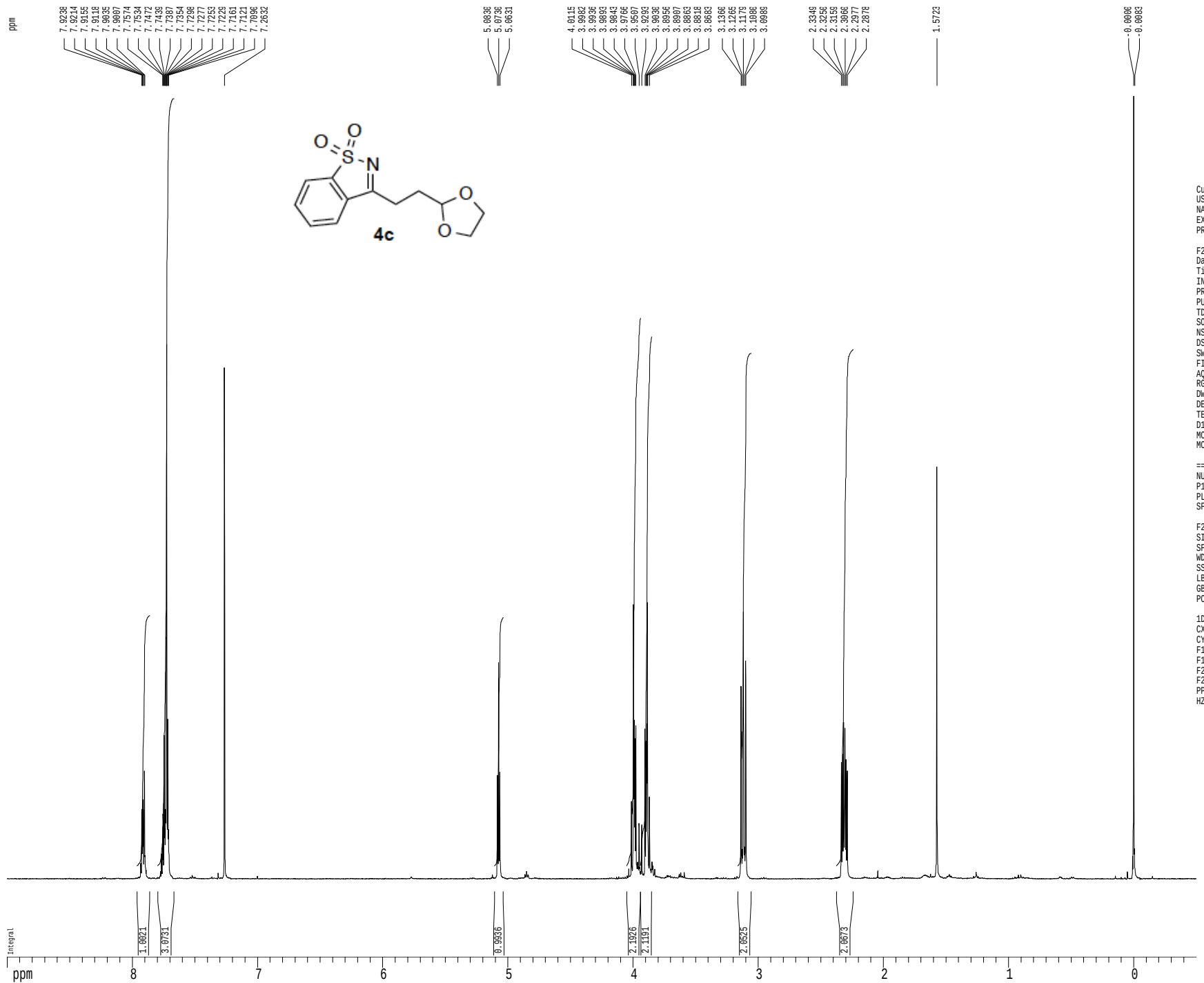

```
Current Data Parameters
USER                osborn
NAME      CAO-III-118-SI
EXPNO                  3
PROCNO                 1

F2 - Acquisition Parameters
Date_           20150528
Time               15.25
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                   5.7
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           500.2200000 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          208.42500 Hz/cm
```

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



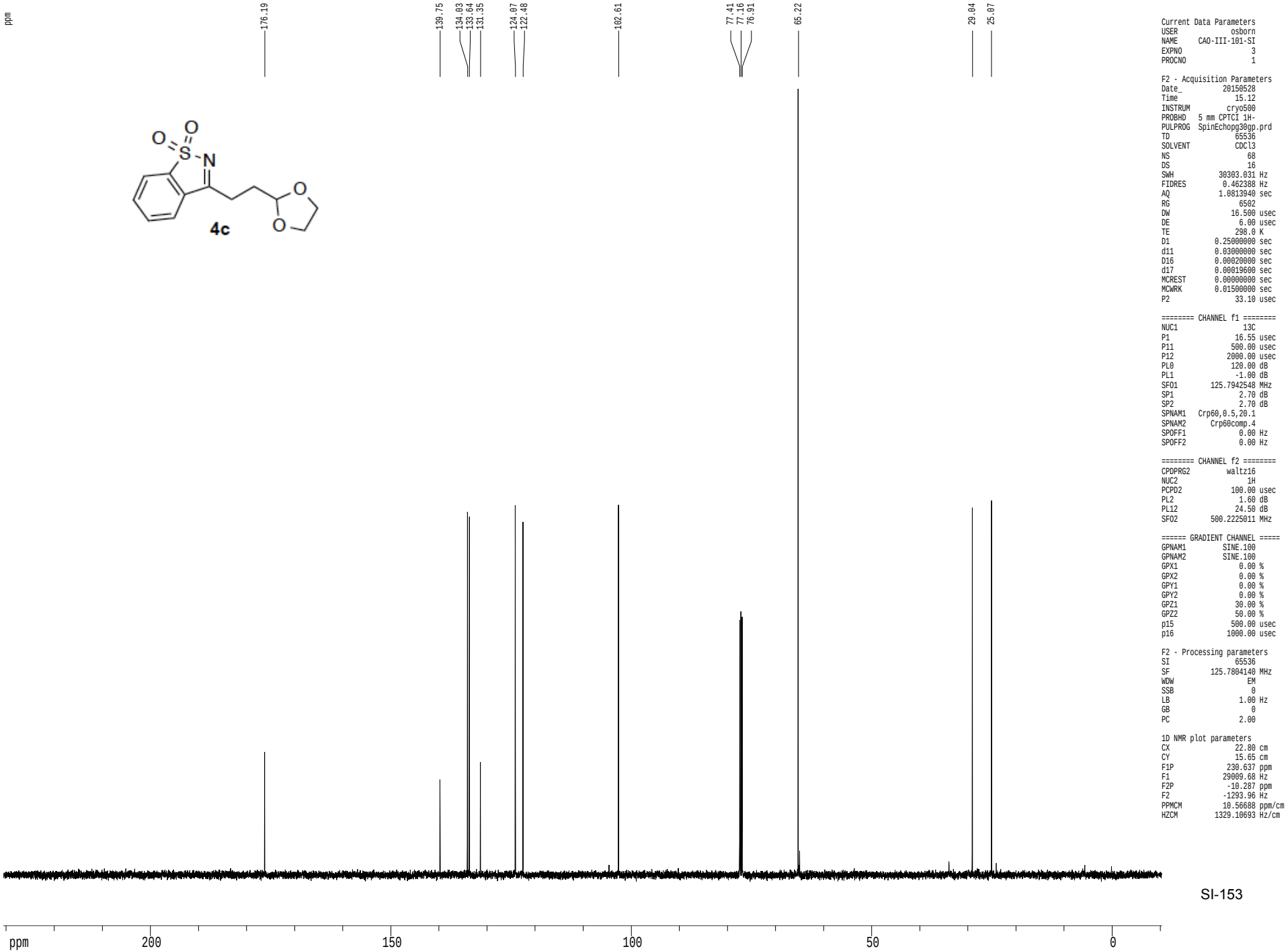

```
Current Data Parameters
USER                osborn
NAME      CAO-III-118-SI
EXPNO                  2
PROCNO                 1

F2 - Acquisition Parameters
Date_           20150528
Time               15.18
INSTRUM          cryo500
PROBHD   5 mm CPTCI 1H-
PULPROG  SpinEchopg30gp.prd
TD                 65536
SOLVENT            CDCl3
NS                   231
DS                    16
SWH            30303.031 Hz
FIDRES         0.462388 Hz
AQ             1.0813940 sec
RG                  8192
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                33.10 usec

======== CHANNEL f1 ========
NUC1                 13C
P1                 16.55 usec
P11               500.00 usec
P12              2000.00 usec
PL0               120.00 dB
PL1                -1.00 dB
SFO1         125.7942548 MHz
SP1                2.70 dB
SP2                2.70 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              24.50 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           125.7804103 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              230.637 ppm
F1             29009.68 Hz
F2P              -10.287 ppm
F2             -1293.96 Hz
PPMCM          10.56688 ppm/cm
HZCM         1329.10693 Hz/cm
```

1H spectrum



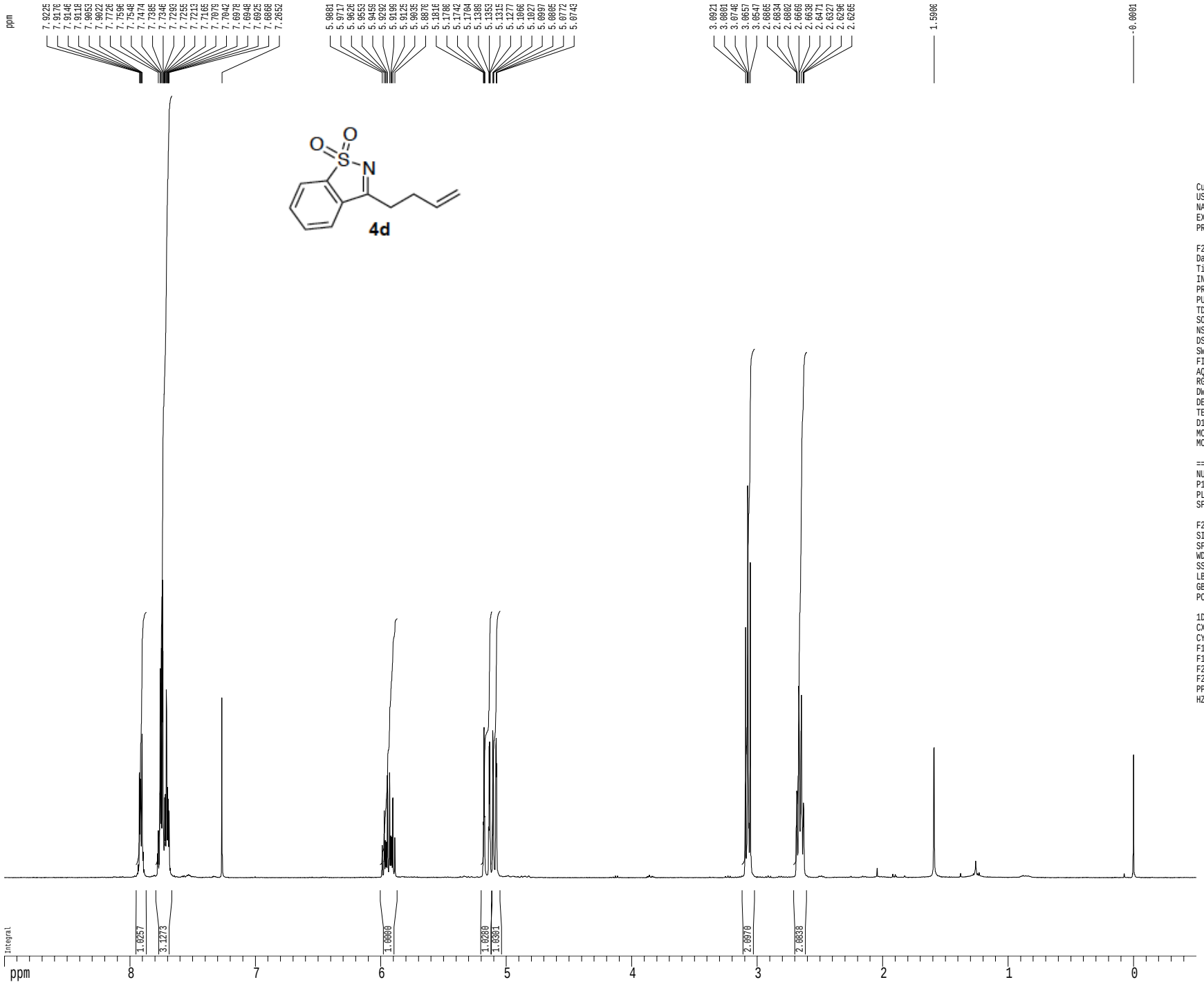

```
Current Data Parameters
USER              osborn
NAME      CAO-III-101-pure
EXPNO                1
PROCNO               1

F2 - Acquisition Parameters
Date_          20141121
Time              12.17
INSTRUM          drx400
PROBHD   5 mm QNP H/F/P
PULPROG           zg30
TD               65536
SOLVENT          CDCl3
NS                   8
DS                   2
SWH           6410.256 Hz
FIDRES         0.097813 Hz
AQ            5.1118579 sec
RG                456.1
DW               78.000 usec
DE                4.50 usec
TE                298.0 K
D1           0.10000000 sec
MCREST       0.00000000 sec
MCWRK        0.01500000 sec

======== CHANNEL f1 ========
NUC1                 1H
P1                12.00 usec
PL1                0.00 dB
SFO1        400.1328009 MHz

F2 - Processing parameters
SI                65536
SF          400.1300196 MHz
WDW                  EM
SSB                  0
LB                0.30 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              -0.500 ppm
F2              -200.06 Hz
PPMCM          0.41667 ppm/cm
HZCM          166.72084 Hz/cm
```

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



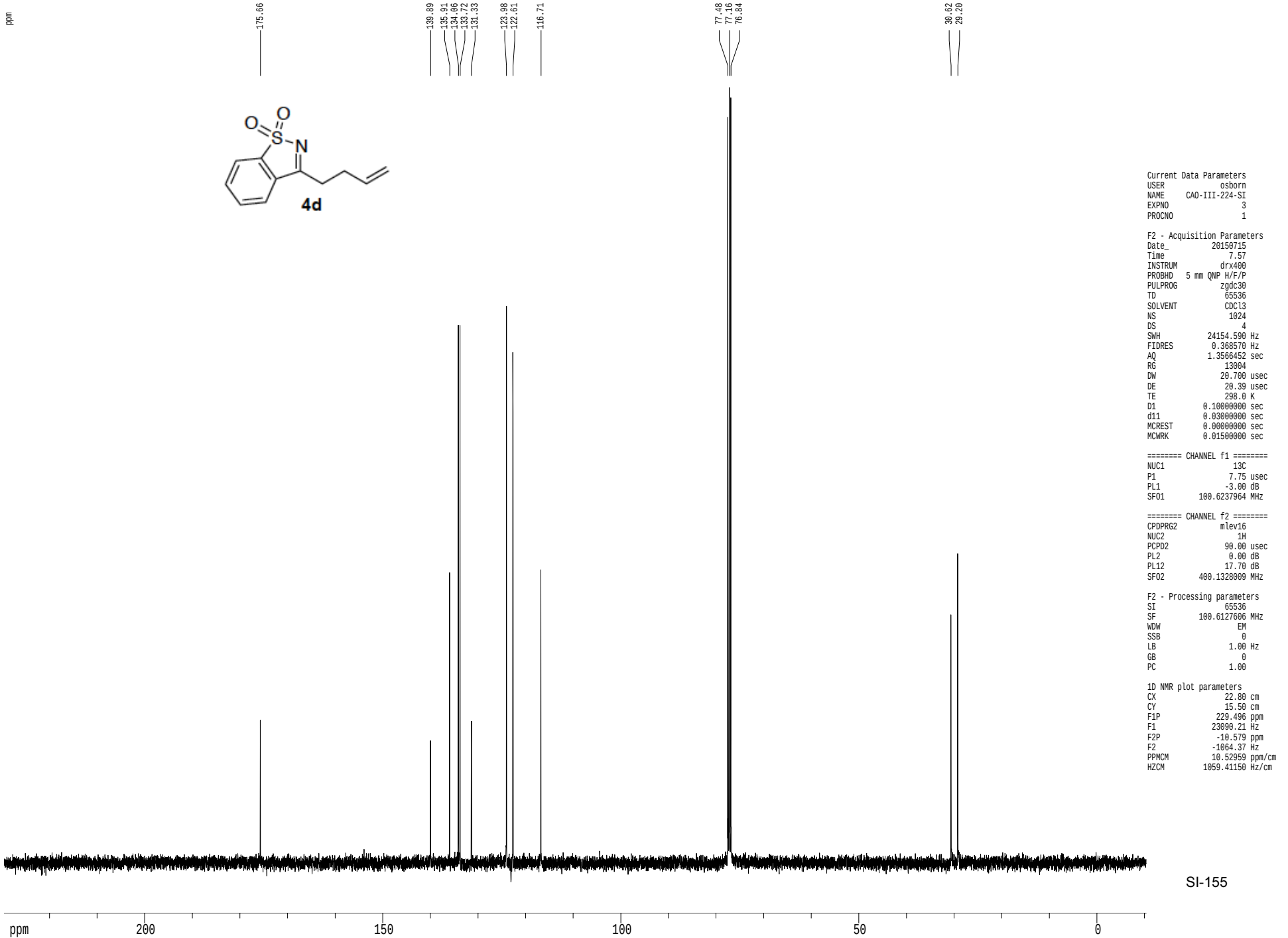

```
Current Data Parameters
USER                osborn
NAME      CAO-III-101-SI
EXPNO                  3
PROCNO                 1

F2 - Acquisition Parameters
Date_           20150528
Time               15.12
INSTRUM          cryo500
PROBHD   5 mm CPTCI 1H-
PULPROG  SpinEchopg30gp.prd
TD                 65536
SOLVENT            CDCl3
NS                    68
DS                    16
SWH            30303.031 Hz
FIDRES         0.462388 Hz
AQ             1.0813940 sec
RG                  6502
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                33.10 usec

======== CHANNEL f1 ========
NUC1                 13C
P1                 16.55 usec
P11               500.00 usec
P12              2000.00 usec
PL0               120.00 dB
PL1                -1.00 dB
SFO1         125.7942548 MHz
SP1                2.70 dB
SP2                2.70 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               24.50 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           125.7804140 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              230.637 ppm
F1             29009.68 Hz
F2P              -10.287 ppm
F2             -1293.96 Hz
PPMCM          10.56688 ppm/cm
HZCM         1329.10693 Hz/cm
```

1H spectrum



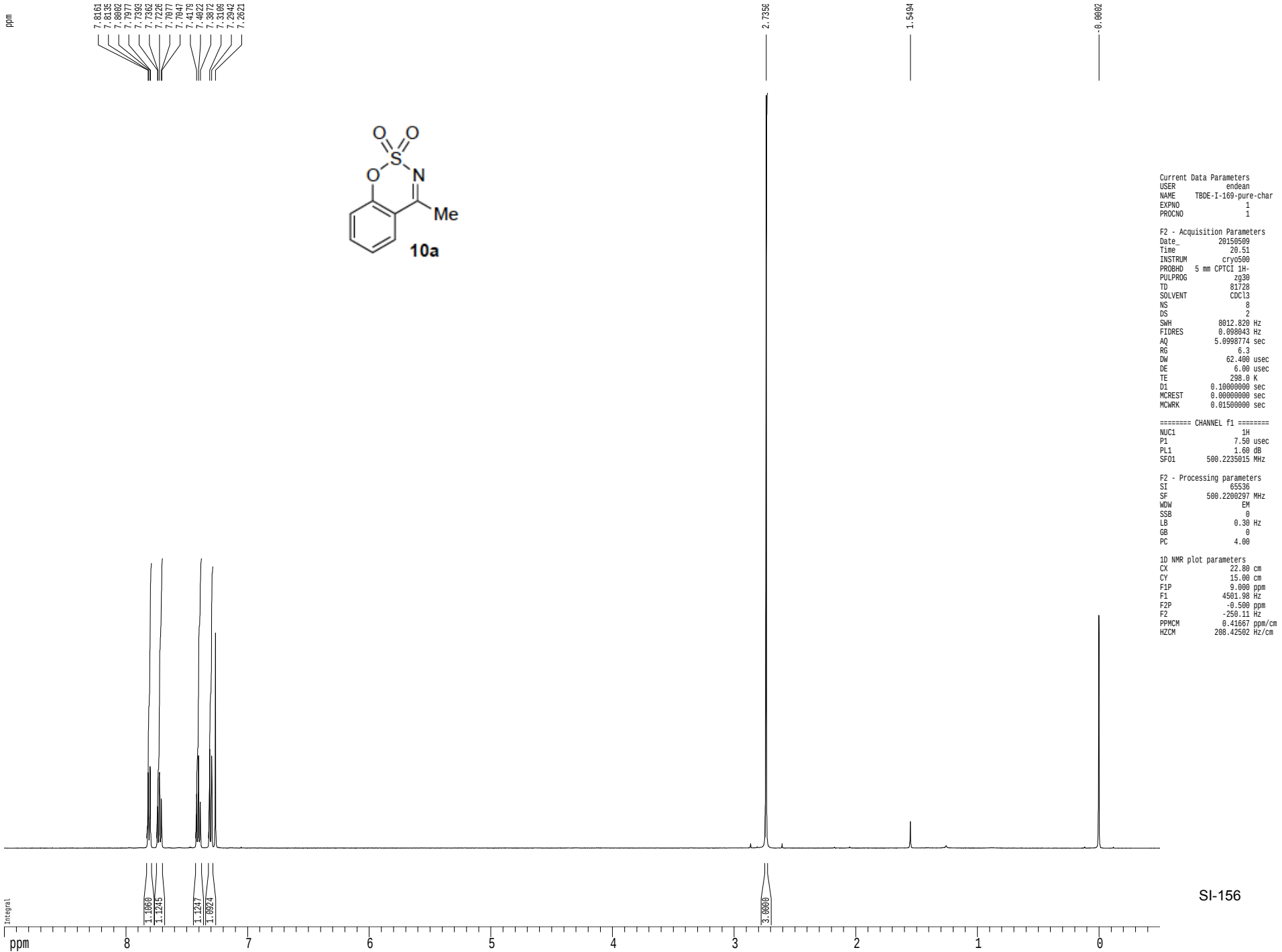

```
Current Data Parameters
USER              osborn
NAME      CAO-III-224-SI
EXPNO                2
PROCNO               1

F2 - Acquisition Parameters
Date_          20150715
Time               7.49
INSTRUM          drx400
PROBHD   5 mm QNP H/F/P
PULPROG           zg30
TD               65536
SOLVENT          CDCl3
NS                   8
DS                   2
SWH           6410.256 Hz
FIDRES        0.097813 Hz
AQ            5.1118579 sec
RG                287.4
DW               78.000 usec
DE                4.50 usec
TE                298.0 K
D1           0.10000000 sec
MCREST       0.00000000 sec
MCWRK        0.01500000 sec

======== CHANNEL f1 ========
NUC1                 1H
P1                12.00 usec
PL1                0.00 dB
SFO1        400.1328009 MHz

F2 - Processing parameters
SI                65536
SF          400.1300196 MHz
WDW                  EM
SSB                  0
LB                0.30 Hz
GB                   0
PC                2.00

1D NMR plot parameters
CX               22.80 cm
CY                7.50 cm
F1P              9.000 ppm
F1             3601.17 Hz
F2P             -0.500 ppm
F2             -200.06 Hz
PPMCM          0.41667 ppm/cm
HZCM         166.72084 Hz/cm
```

13C spectrum with 1H decoupling



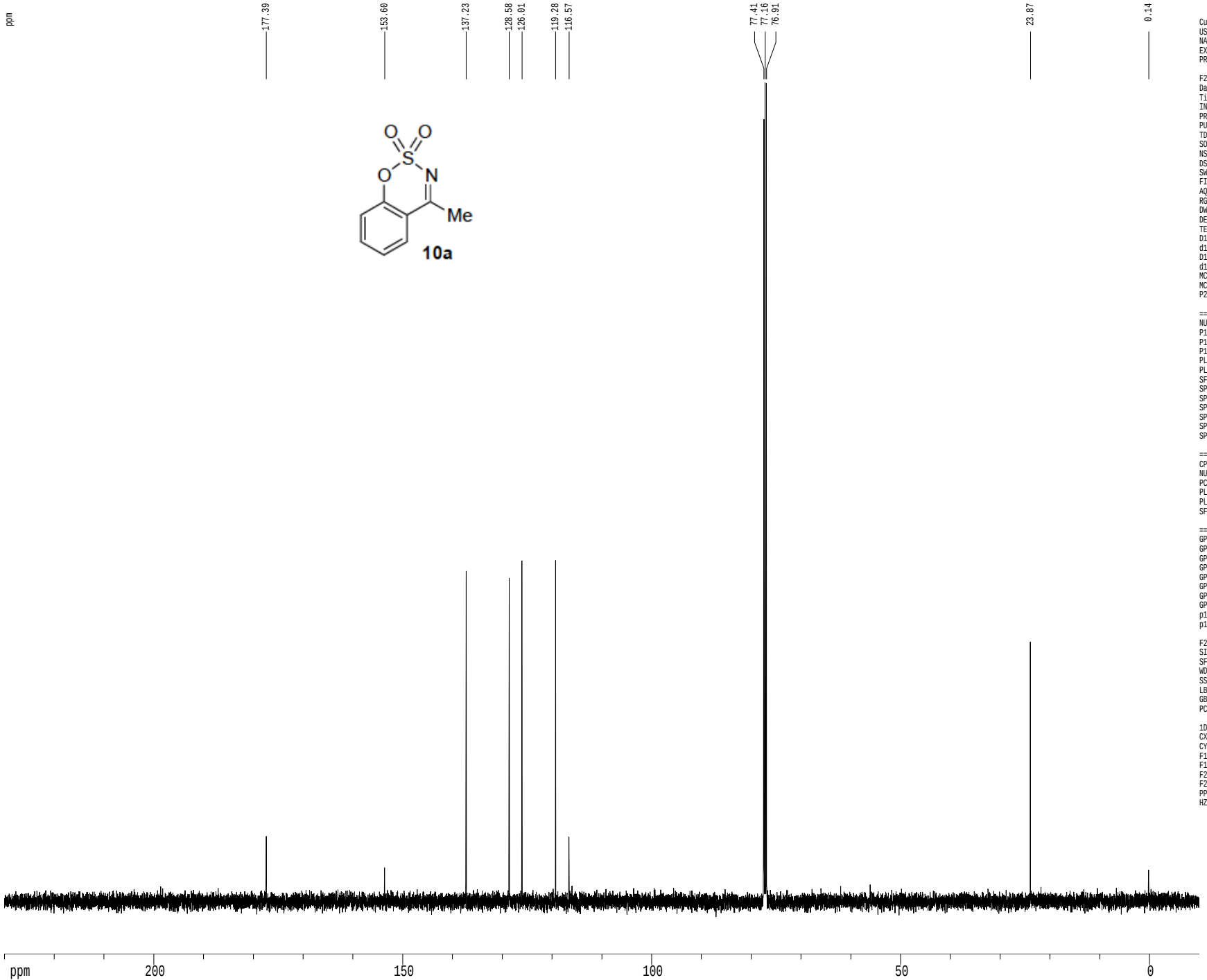

```
Current Data Parameters
USER              osborn
NAME      CAO-III-224-SI
EXPNO                3
PROCNO               1

F2 - Acquisition Parameters
Date_          20150715
Time               7.57
INSTRUM          drx400
PROBHD   5 mm QNP H/F/P
PULPROG          zgdc30
TD                65536
SOLVENT           CDCl3
NS                 1024
DS                    4
SWH           24154.590 Hz
FIDRES         0.368570 Hz
AQ             1.3566452 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
MCWRK        0.01500000 sec

======== CHANNEL f1 ========
NUC1                13C
P1                 7.75 usec
PL1                -3.00 dB
SFO1        100.6237964 MHz

======== CHANNEL f2 ========
CPDPRG2          mlev16
NUC2                1H
PCPD2             90.00 usec
PL2                0.00 dB
PL12              17.70 dB
SFO2        400.1328009 MHz

F2 - Processing parameters
SI                 65536
SF           100.6127606 MHz
WDW                   EM
SSB                    0
LB                 1.00 Hz
GB                     0
PC                 1.00

1D NMR plot parameters
CX                22.80 cm
CY                15.50 cm
F1P              229.496 ppm
F1             23090.21 Hz
F2P              -10.579 ppm
F2             -1064.37 Hz
PPMCM          10.52959 ppm/cm
HZCM         1059.41150 Hz/cm
```

1H spectrum



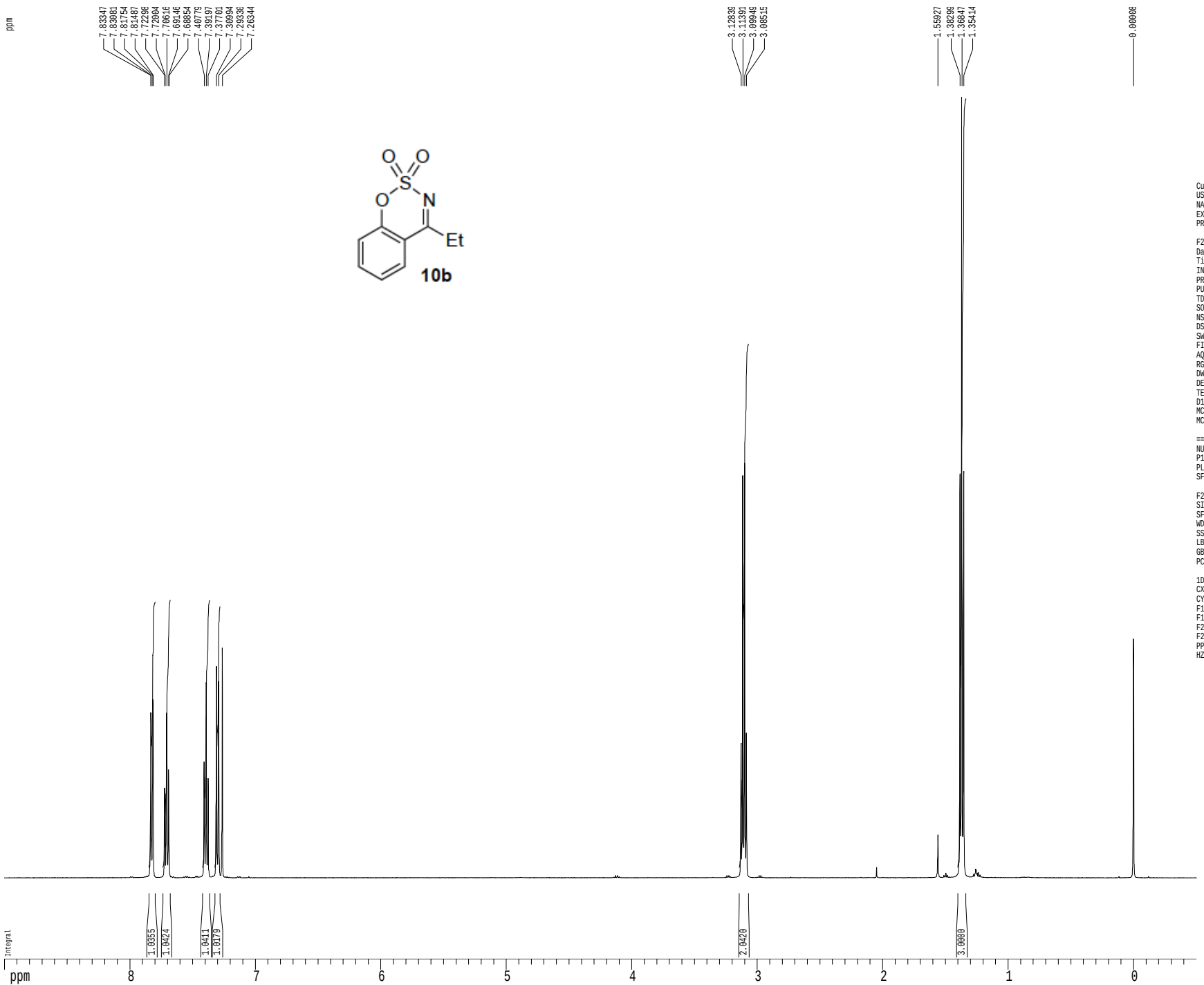

```
Current Data Parameters
USER                 endean
NAME       TBDE-I-169-pure-char
EXPNO                     1
PROCNO                    1

F2 - Acquisition Parameters
Date_              20150509
Time                  20.51
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                        6.3
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              500.2200297 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



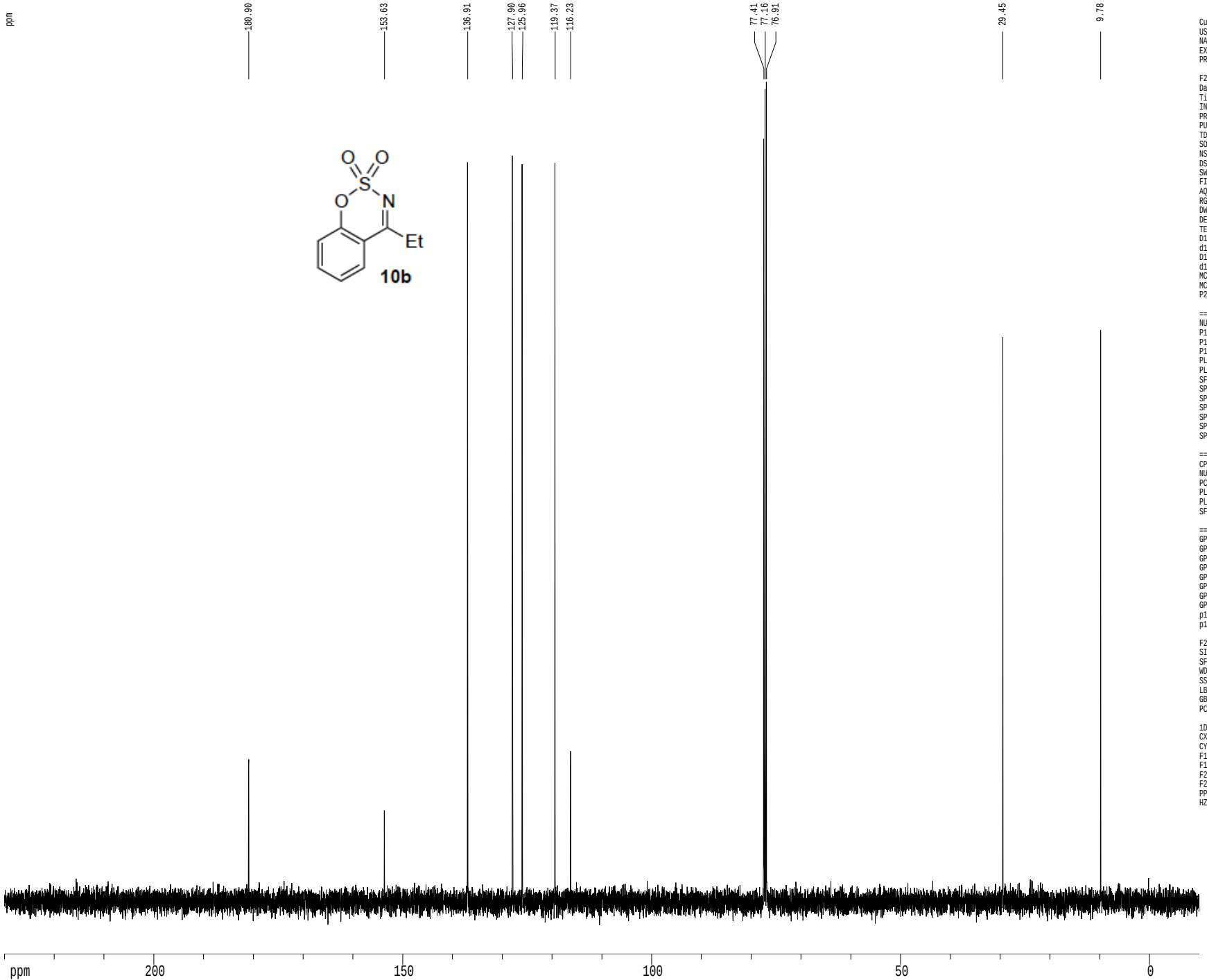

```
Current Data Parameters
USER              endean
NAME      TBDE-I-169-pure-char
EXPNO                  2
PROCNO                 1

F2 - Acquisition Parameters
Date_          20150509
Time              20.55
INSTRUM         cryo500
PROBHD   5 mm CPTCI 1H-
PULPROG  SpinEchopg30gp.prd
TD                65536
SOLVENT           CDCl3
NS                  424
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
MCWRK          0.01500000 sec
P2                33.10 usec

======== CHANNEL f1 ========
NUC1                13C
P1                16.55 usec
P11              500.00 usec
P12             2000.00 usec
PL0              120.00 dB
PL1               -1.00 dB
SFO1        125.7942548 MHz
SP1               2.70 dB
SP2               2.70 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              24.50 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           125.7804080 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              230.000 ppm
F1             28929.49 Hz
F2P              -10.000 ppm
F2             -1257.80 Hz
PPMCM          10.52632 ppm/cm
HZCM         1324.00439 Hz/cm
```

1H spectrum



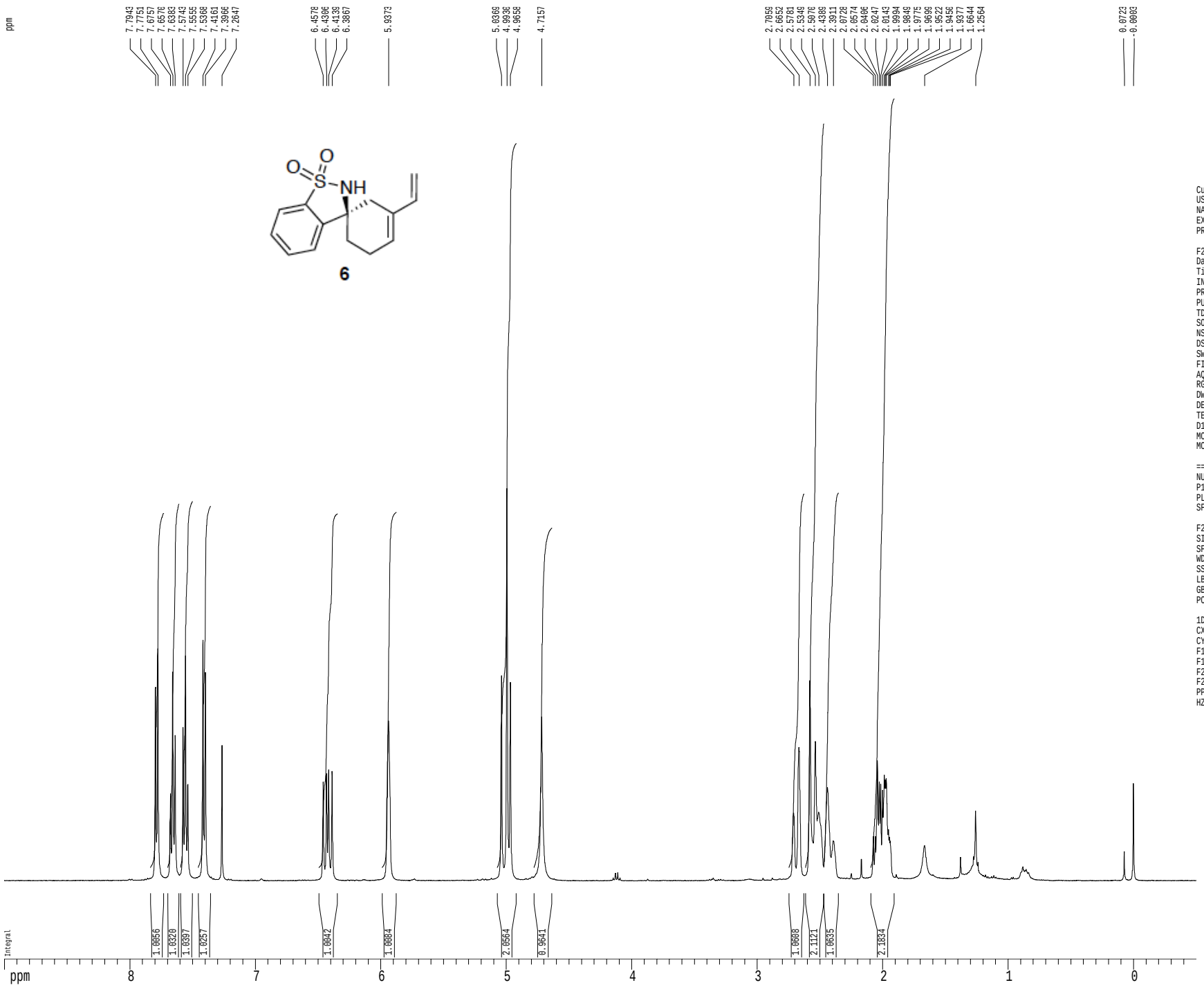

```
Current Data Parameters
USER              endean
NAME      TBDE-I-170-pure-char
EXPNO                1
PROCNO               1

F2 - Acquisition Parameters
Date_          20150509
Time             21.06
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                  7.1
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          500.2200289 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



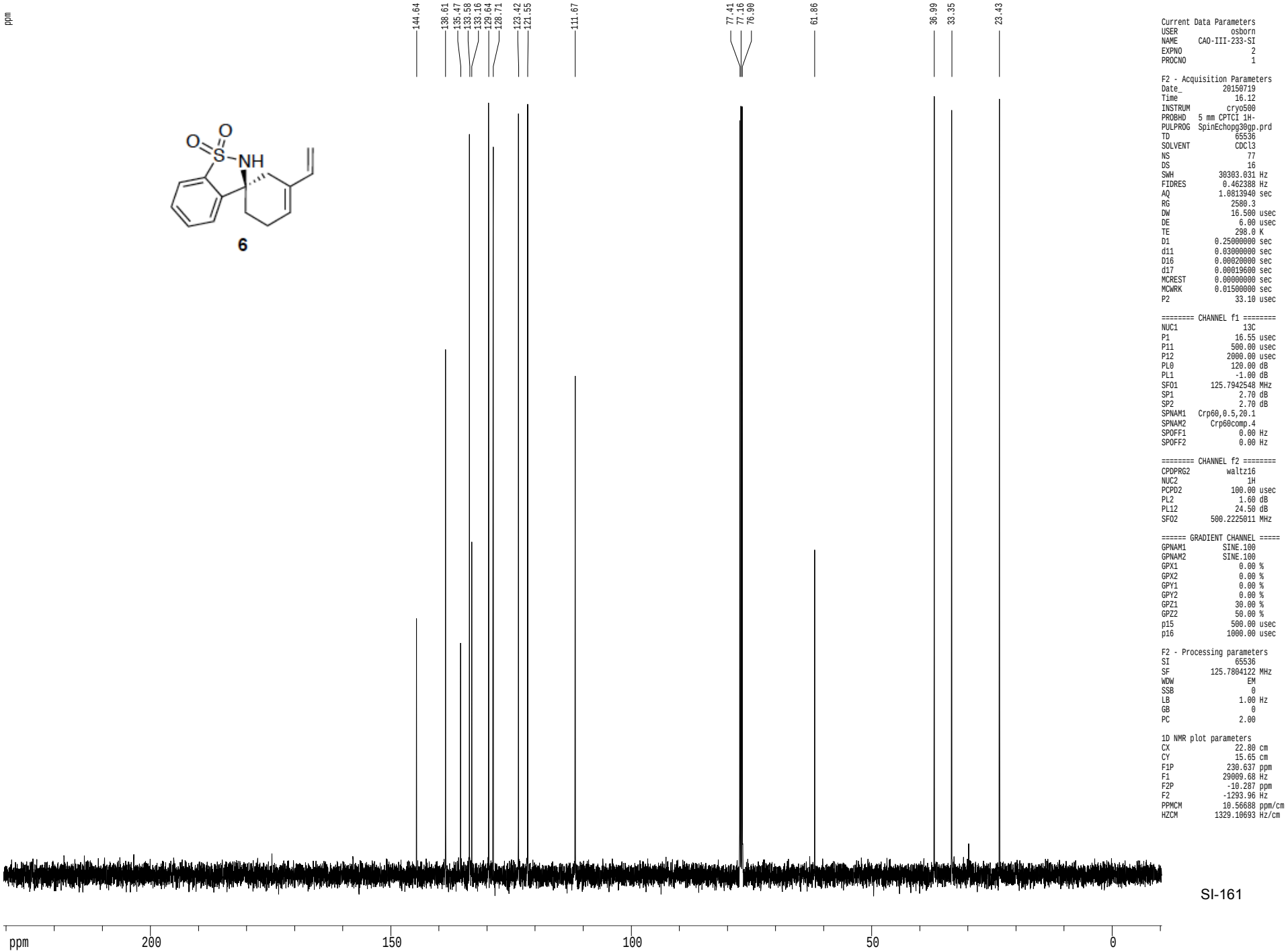

```
Current Data Parameters
USER              endean
NAME      TBDE-I-170-pure-char
EXPNO                  2
PROCNO                 1

F2 - Acquisition Parameters
Date_          20150509
Time              21.12
INSTRUM         cryo500
PROBHD   5 mm CPTCI 1H-
PULPROG  SpinEchopg30gp.prd
TD                65536
SOLVENT           CDCl3
NS                  184
DS                   16
SWH           30303.031 Hz
FIDRES         0.462388 Hz
AQ             1.0813940 sec
RG                 8192
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               33.10 usec

======== CHANNEL f1 ========
NUC1                13C
P1                16.55 usec
P11              500.00 usec
P12             2000.00 usec
PL0              120.00 dB
PL1               -1.00 dB
SFO1        125.7942548 MHz
SP1               2.70 dB
SP2               2.70 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              24.50 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

1D NMR plot parameters
CX                22.80 cm
CY                15.65 cm
F1P              230.000 ppm
F1             28929.49 Hz
F2P              -10.000 ppm
F2             -1257.80 Hz
PPMCM          10.52632 ppm/cm
HZCM         1324.00439 Hz/cm
```

1H spectrum

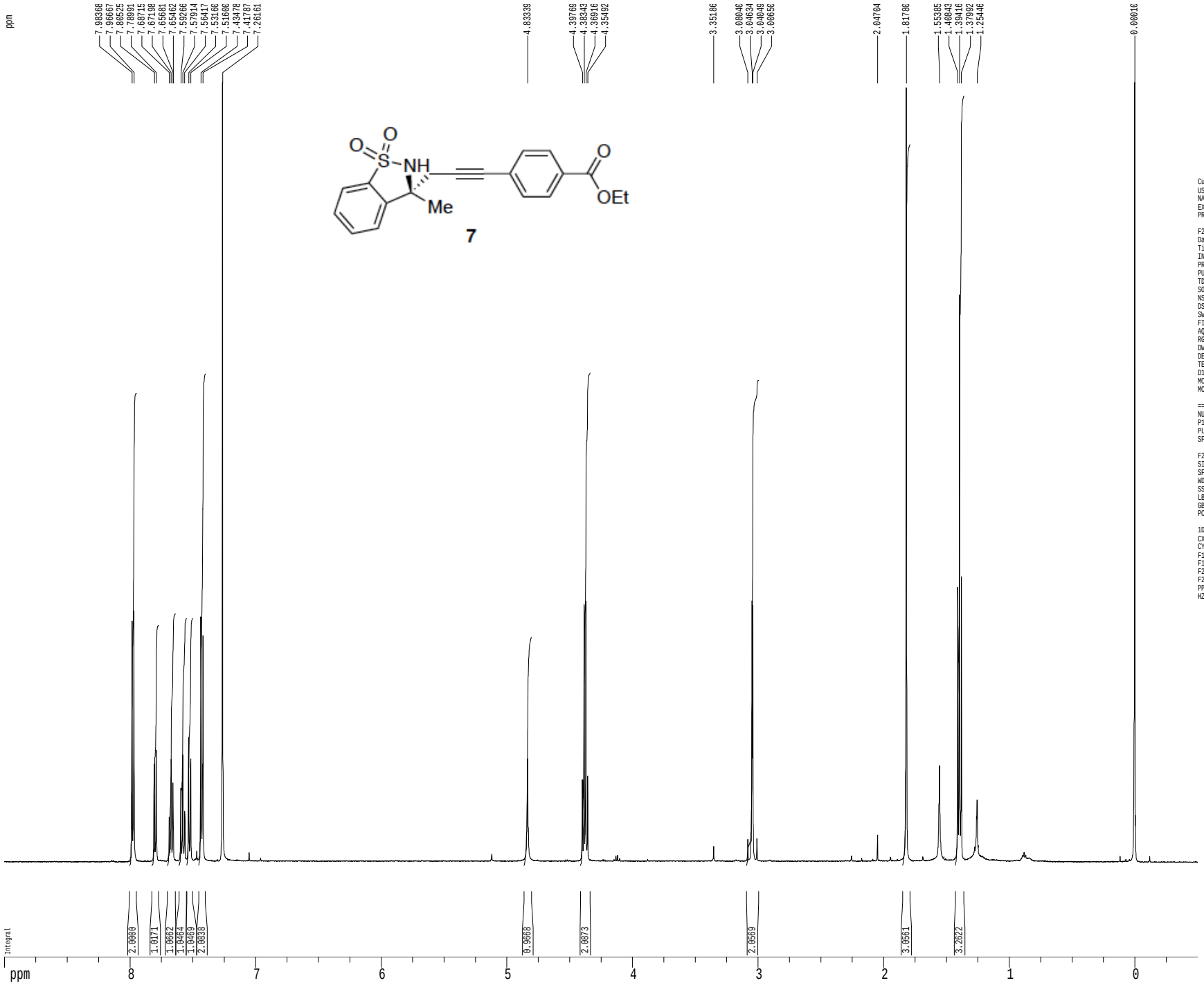



```
Current Data Parameters
USER              osborn
NAME      CAO-III-233-pure
EXPNO                2
PROCNO               1

F2 - Acquisition Parameters
Date_          20150719
Time              15.03
INSTRUM          drx400
PROBHD   5 mm QNP H/F/P
PULPROG           zg30
TD               65536
SOLVENT          CDCl3
NS                   8
DS                   2
SWH           6410.256 Hz
FIDRES        0.097813 Hz
AQ            5.1118579 sec
RG                 181
DW              78.000 usec
DE               4.50 usec
TE               298.0 K
D1          0.10000000 sec
MCREST      0.00000000 sec
MCWRK       0.01500000 sec

======== CHANNEL f1 ========
NUC1                1H
P1              12.00 usec
PL1              0.00 dB
SFO1        400.1328009 MHz

F2 - Processing parameters
SI                 65536
SF          400.1300199 MHz
WDW                  EM
SSB                  0
LB               0.30 Hz
GB                   0
PC                2.00

1D NMR plot parameters
CX                22.80 cm
CY                 7.50 cm
F1P               9.000 ppm
F1             3601.17 Hz
F2P              -0.500 ppm
F2             -200.06 Hz
PPMCM          0.41667 ppm/cm
HZCM          166.72084 Hz/cm
```

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

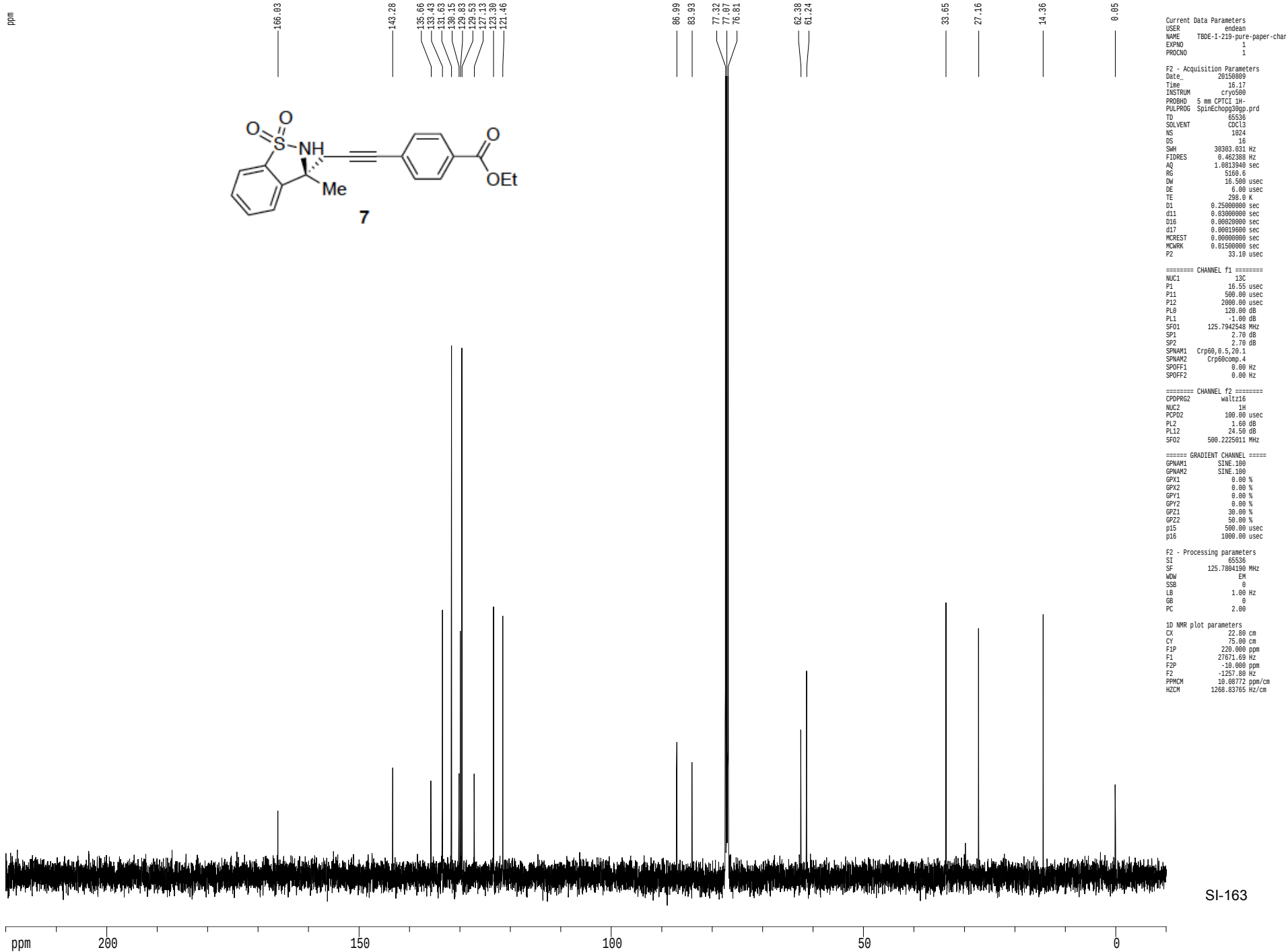


1H spectrum



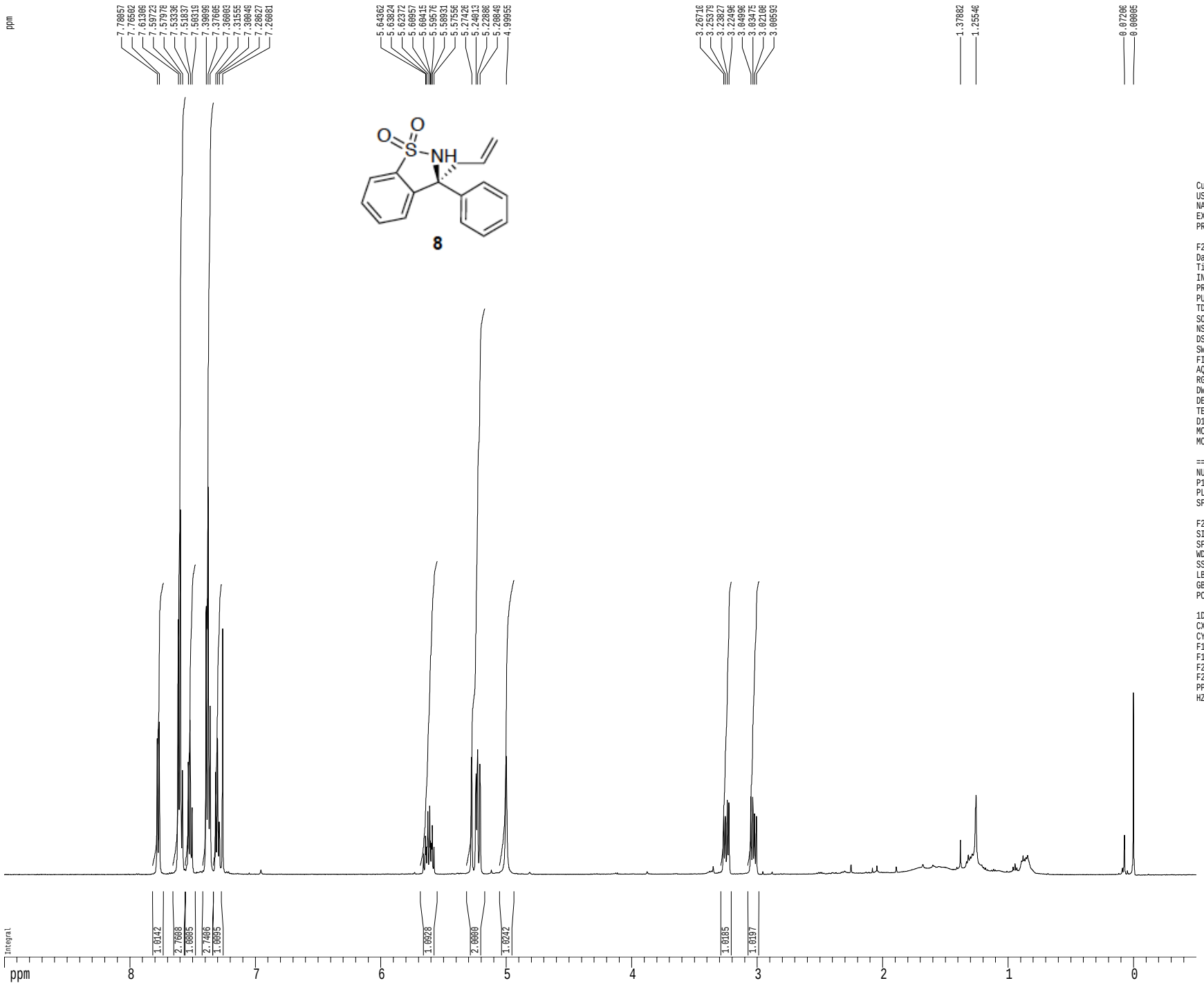

```
Current Data Parameters
USER                endean
NAME      TBDE-I-219-pure-paper-char
EXPNO                    2
PROCNO                   1

F2 - Acquisition Parameters
Date_            20150809
Time                15.49
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                     8
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           500.2200304 MHz
WDW                   EM
SSB                    0
LB                 0.30 Hz
GB                     0
PC                 4.00

1D NMR plot parameters
CX                22.80 cm
CY                30.00 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



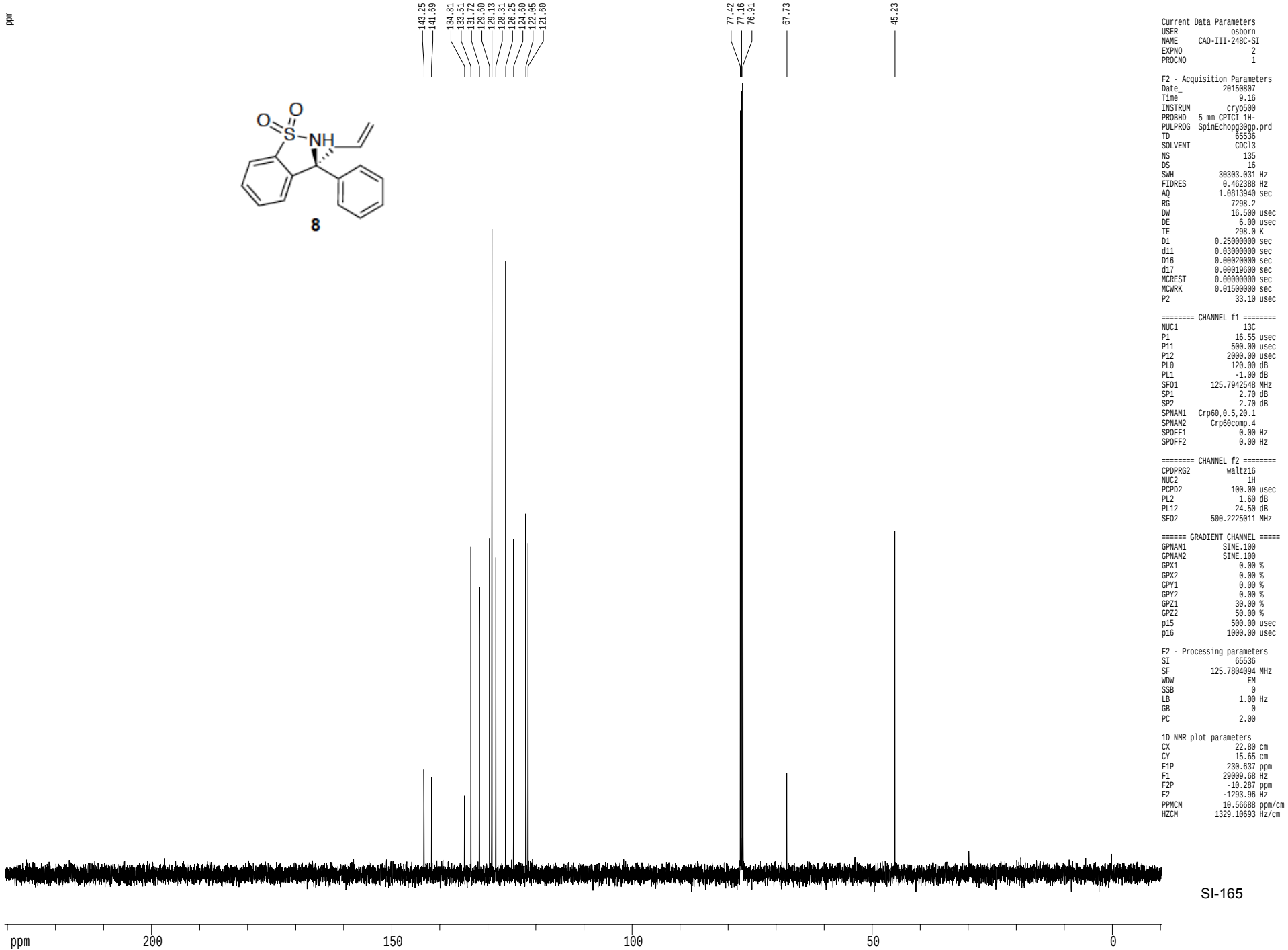

```
Current Data Parameters
USER              endean
NAME     TBDE-I-219-pure-paper-char
EXPNO                  1
PROCNO                 1

F2 - Acquisition Parameters
Date_           20150809
Time              16.17
INSTRUM         cryo500
PROBHD   5 mm CPTCI 1H-
PULPROG  SpinEchopg30gp.prd
TD                65536
SOLVENT           CDCl3
NS                 1024
DS                   16
SWH           30303.031 Hz
FIDRES         0.462388 Hz
AQ            1.0813940 sec
RG               5160.6
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
MCWRK        0.01500000 sec
P2                33.10 usec

======== CHANNEL f1 ========
NUC1               13C
P1                16.55 usec
P11              500.00 usec
P12             2000.00 usec
PL0              120.00 dB
PL1               -1.00 dB
SFO1         125.7942548 MHz
SP1                2.70 dB
SP2                2.70 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              24.50 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           125.7804190 MHz
WDW                  EM
SSB                   0
LB                 1.00 Hz
GB                    0
PC                 2.00

1D NMR plot parameters
CX                22.80 cm
CY                75.00 cm
F1P             220.000 ppm
F1            27671.69 Hz
F2P             -10.000 ppm
F2            -1257.80 Hz
PPMCM          10.08772 ppm/cm
HZCM         1268.83765 Hz/cm
```

1H spectrum



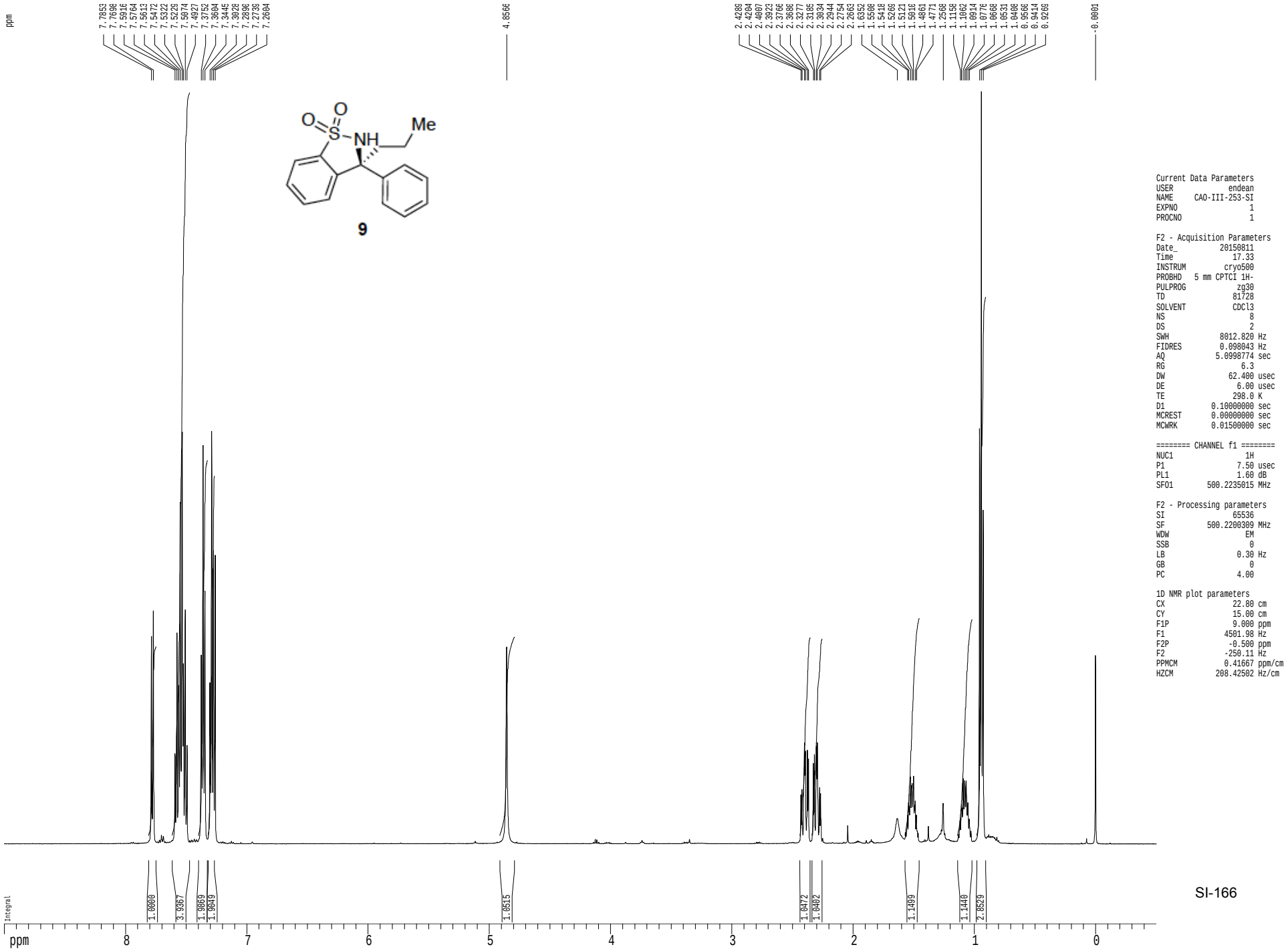

```
Current Data Parameters
USER                osborn
NAME      CAO-III-248C-SI
EXPNO                  1
PROCNO                 1

F2 - Acquisition Parameters
Date_           20150807
Time               9.15
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                     4
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           500.2200310 MHz
WDW                   EM
SSB                    0
LB                  0.30 Hz
GB                     0
PC                  4.00

1D NMR plot parameters
CX                 22.80 cm
CY                  7.50 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



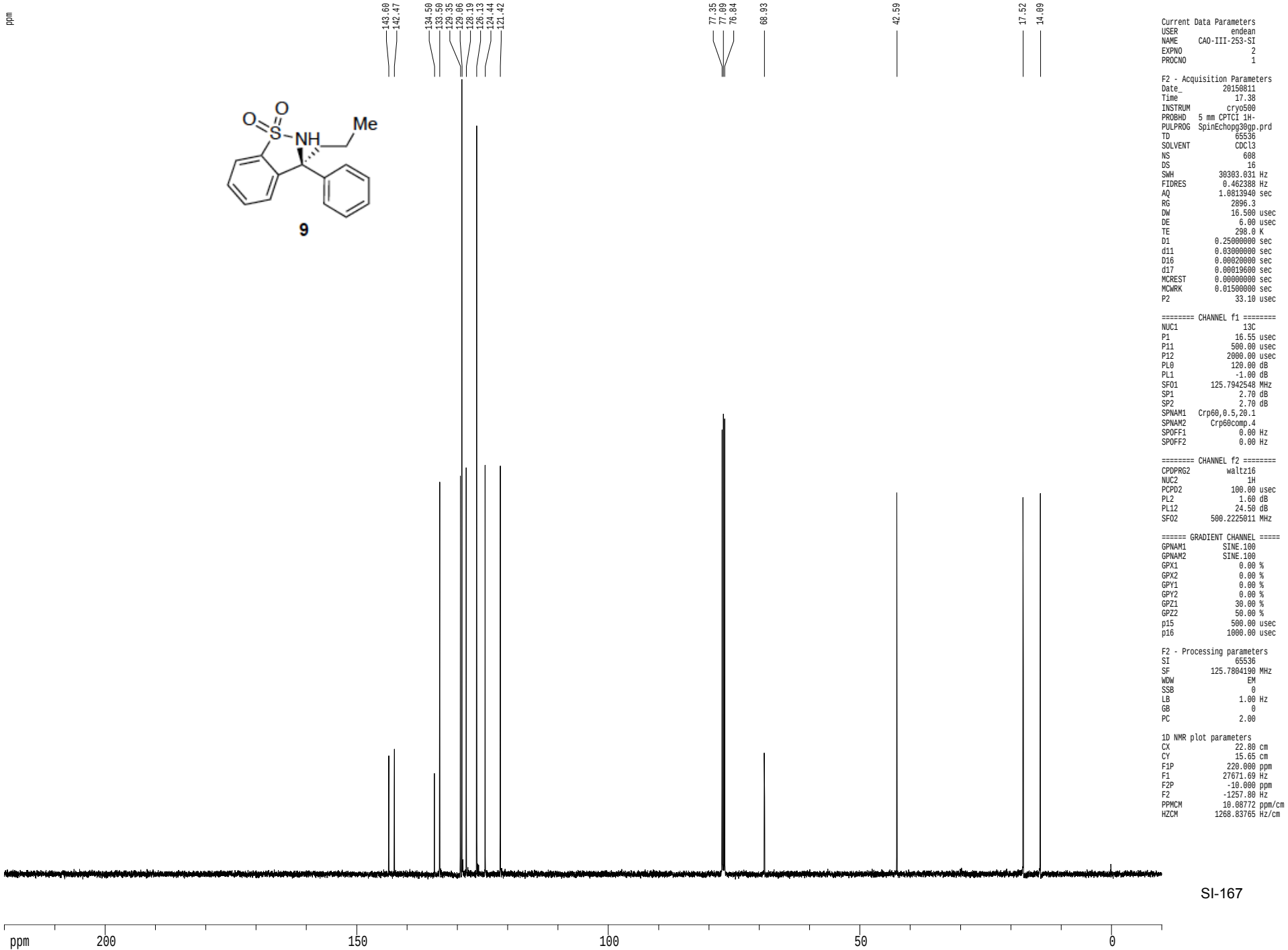

```
Current Data Parameters
USER                osborn
NAME      CAO-III-248C-SI
EXPNO                  2
PROCNO                 1

F2 - Acquisition Parameters
Date_           20150807
Time               9.16
INSTRUM          cryo500
PROBHD   5 mm CPTCI 1H-
PULPROG  SpinEchopg30gp.prd
TD                65536
SOLVENT           CDCl3
NS                  135
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
MCWRK         0.01500000 sec
P2                33.10 usec

======== CHANNEL f1 ========
NUC1                 13C
P1                16.55 usec
P11              500.00 usec
P12             2000.00 usec
PL0              120.00 dB
PL1               -1.00 dB
SFO1         125.7942548 MHz
SP1               2.70 dB
SP2               2.70 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              24.50 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           125.7804094 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              230.637 ppm
F1              29009.68 Hz
F2P              -10.287 ppm
F2              -1293.96 Hz
PPMCM            10.56688 ppm/cm
HZCM          1329.10693 Hz/cm
```

1H spectrum

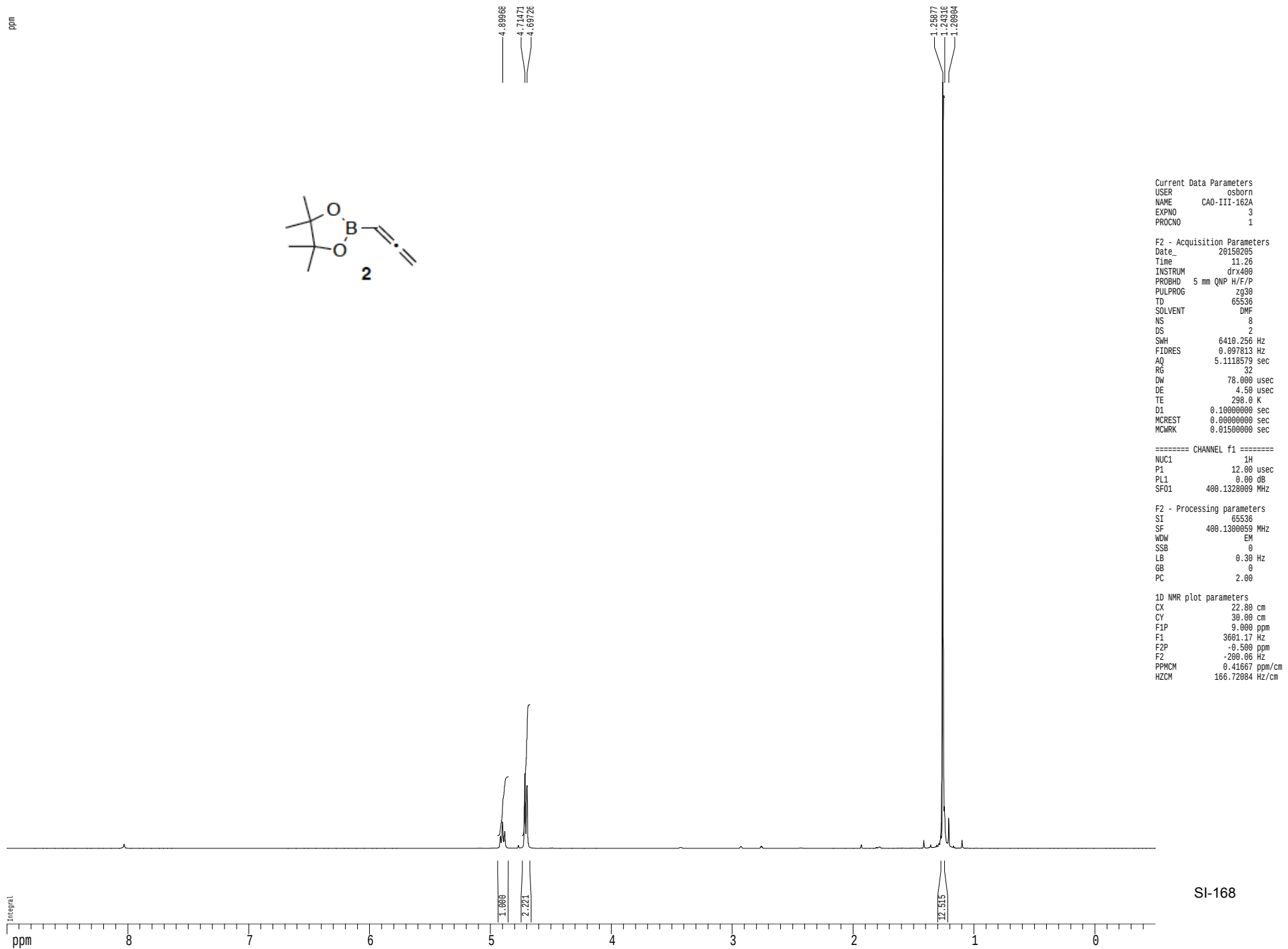



```
Current Data Parameters
USER                endean
NAME      CAO-III-253-SI
EXPNO                  1
PROCNO                 1

F2 - Acquisition Parameters
Date_           20150811
Time               17.33
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                   6.3
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           500.2200309 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



1H spectrum



Current Data Parameters
USER osborn
NAME CAO-III-162A
EXPNO 3
PROCNO 1

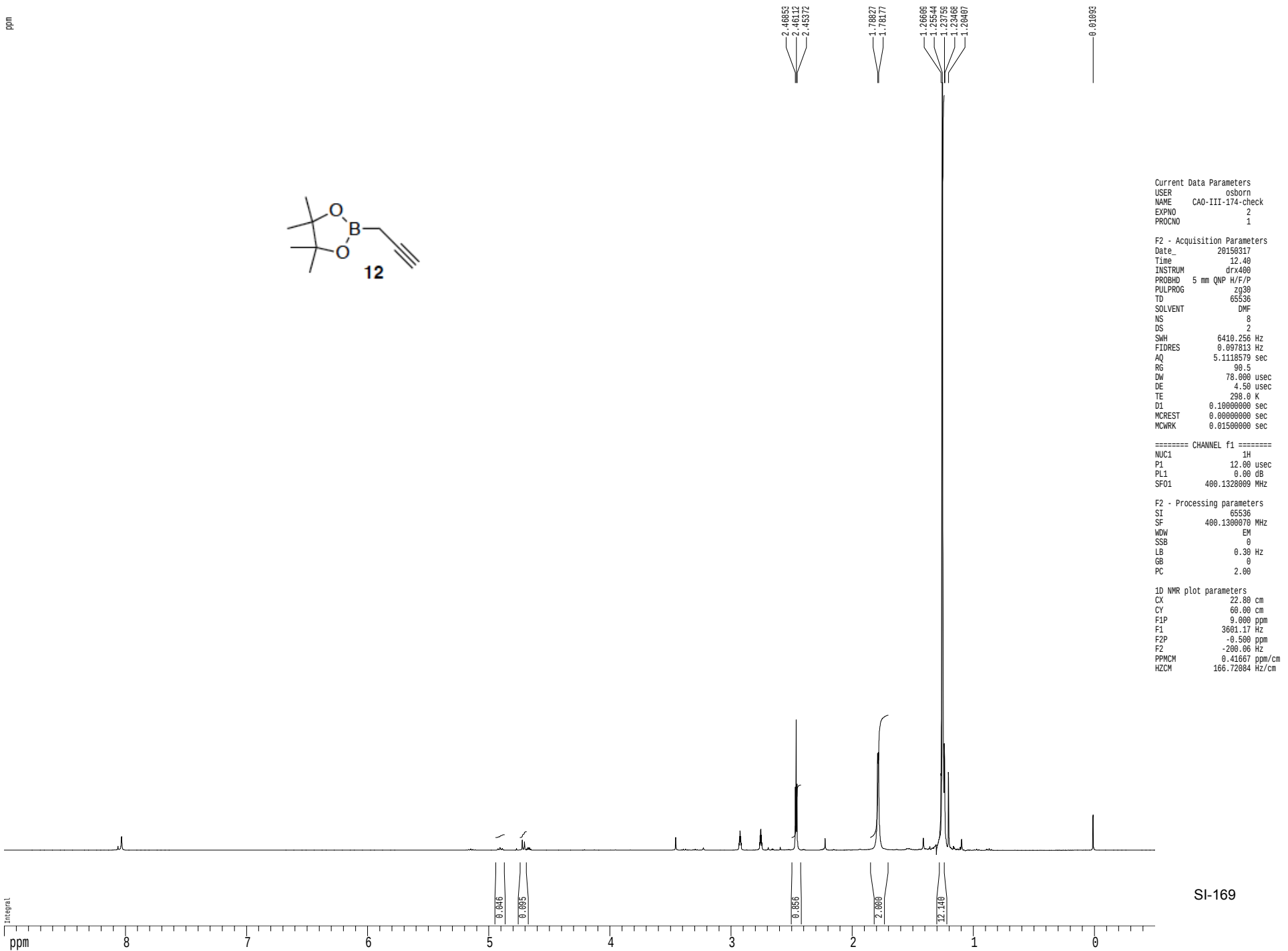
F2 - Acquisition Parameters
Date_ 20150205
Time 11.26
INSTRUM drx400
PROBHD 5 mm QNP H/F/P
PULPROG zg30
TD 65536
SOLVENT DMF
NS 8
DS 2
SWH 6410.256 Hz
FIDRES 0.097813 Hz
AQ 5.1118579 sec
RG 32
DW 78.000 usec
DE 4.50 usec
TE 298.0 K
D1 0.10000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec

======== CHANNEL f1 ========
NUC1 1H
P1 12.00 usec
PL1 0.00 dB
SFO1 400.1328009 MHz

F2 - Processing parameters
SI 65536
SF 400.1300059 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 2.00

1D NMR plot parameters
CX 22.80 cm
CY 30.00 cm
F1P 9.000 ppm
F1 3601.17 Hz
F2P -0.500 ppm
F2 -200.06 Hz
PPMCM 0.41667 ppm/cm
HZCM 166.72084 Hz/cm

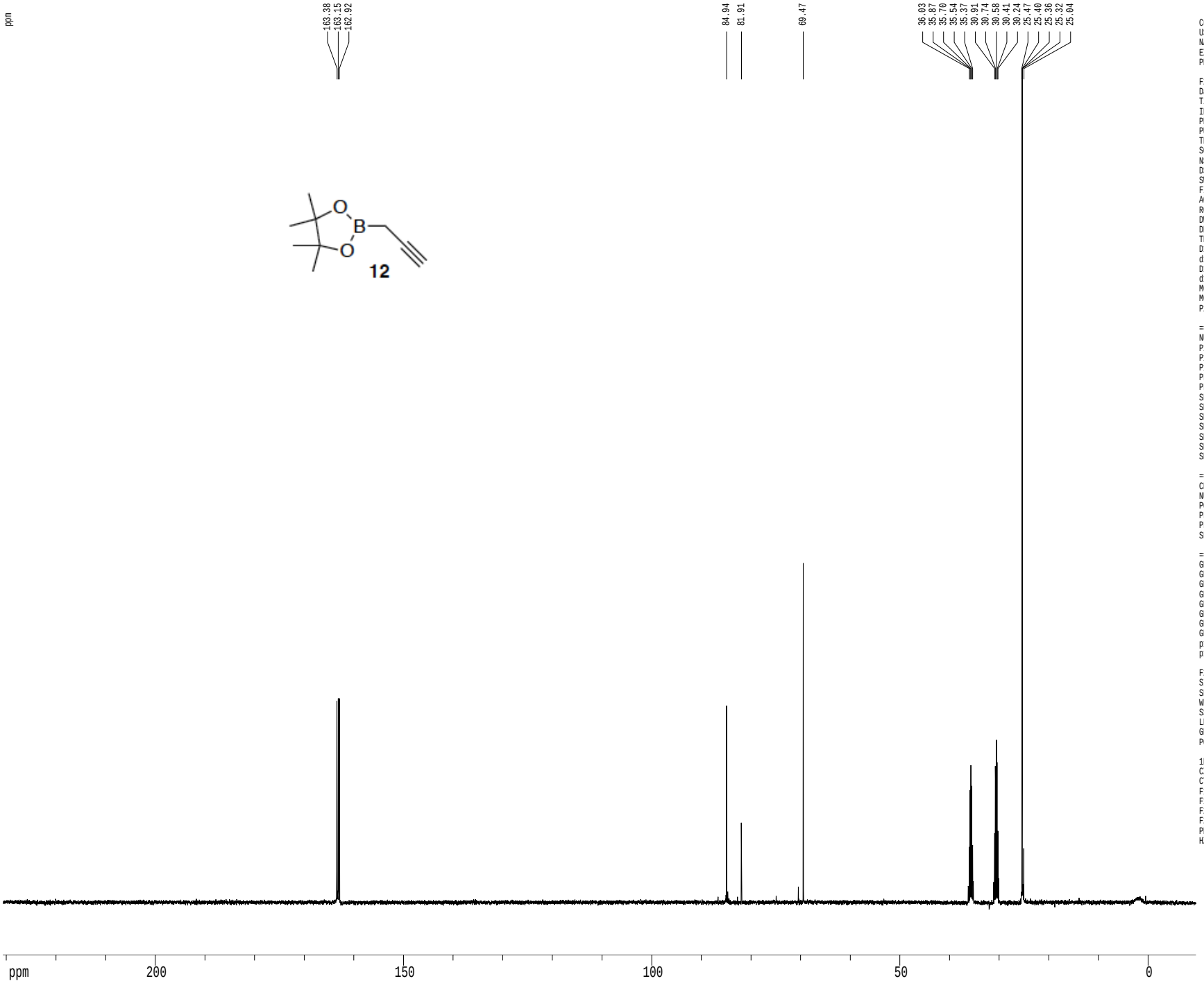
1H spectrum



12

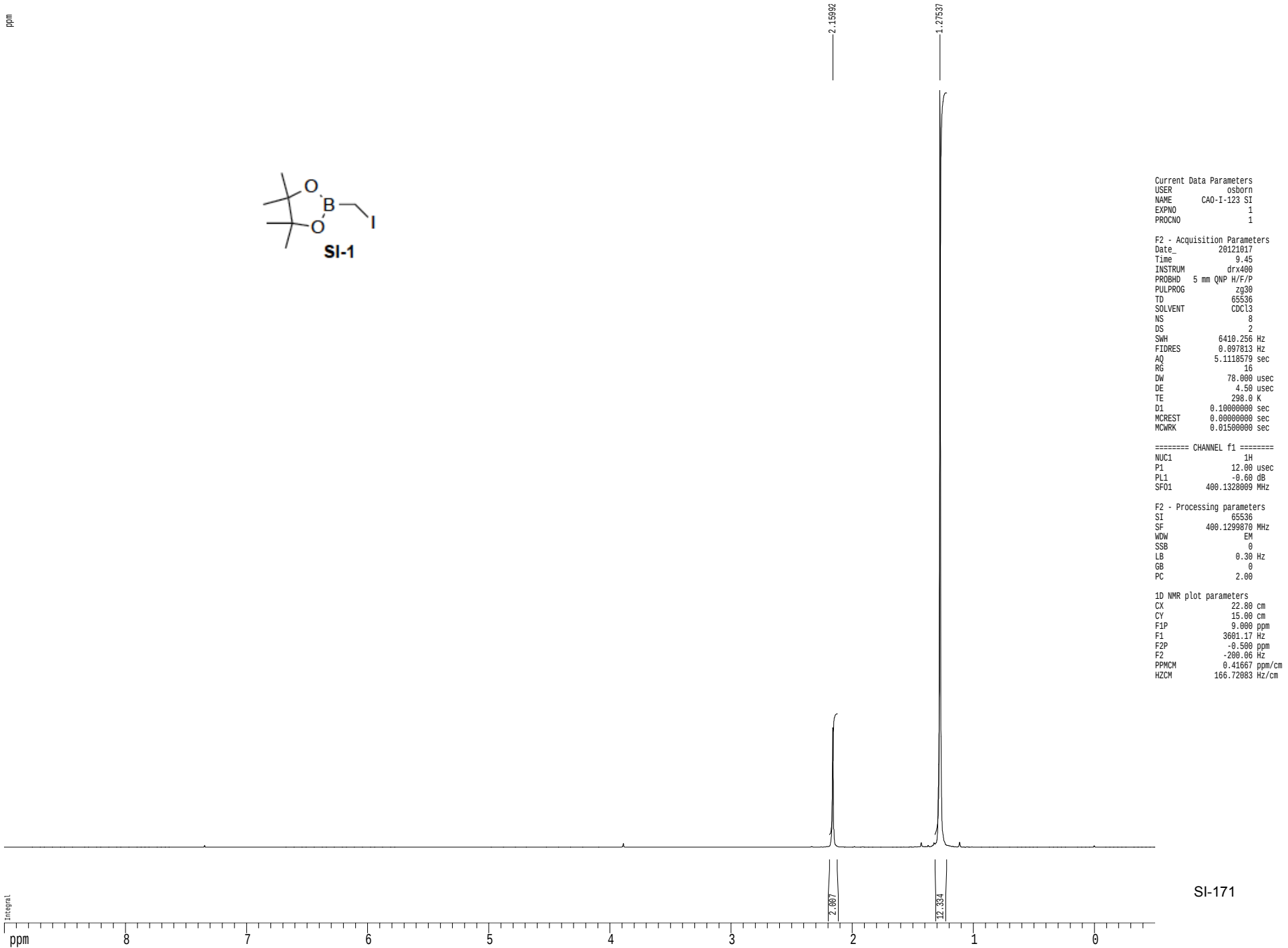
```
Current Data Parameters
USER              osborn
NAME      CAO-III-174-check
EXPNO                 2
PROCNO                1

F2 - Acquisition Parameters
Date_          20150317
Time              12.40
INSTRUM          drx400
PROBHD   5 mm QNP H/F/P
PULPROG           zg30
TD               65536
SOLVENT            DMF
NS                   8
DS                   2
SWH           6410.256 Hz
FIDRES        0.097813 Hz
AQ            5.1118579 sec
RG                90.5
DW              78.000 usec
DE               4.50 usec
TE              298.0 K
D1          0.10000000 sec
MCREST      0.00000000 sec
MCWRK       0.01500000 sec

======== CHANNEL f1 ========
NUC1                 1H
P1                12.00 usec
PL1                0.00 dB
SFO1        400.1328009 MHz

F2 - Processing parameters
SI                65536
SF          400.1300070 MHz
WDW                  EM
SSB                  0
LB                0.30 Hz
GB                   0
PC                2.00

1D NMR plot parameters
CX                22.80 cm
CY                60.00 cm
F1P               9.000 ppm
F1              3601.17 Hz
F2P              -0.500 ppm
F2             -200.06 Hz
PPMCM          0.41667 ppm/cm
HZCM         166.72084 Hz/cm
```

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



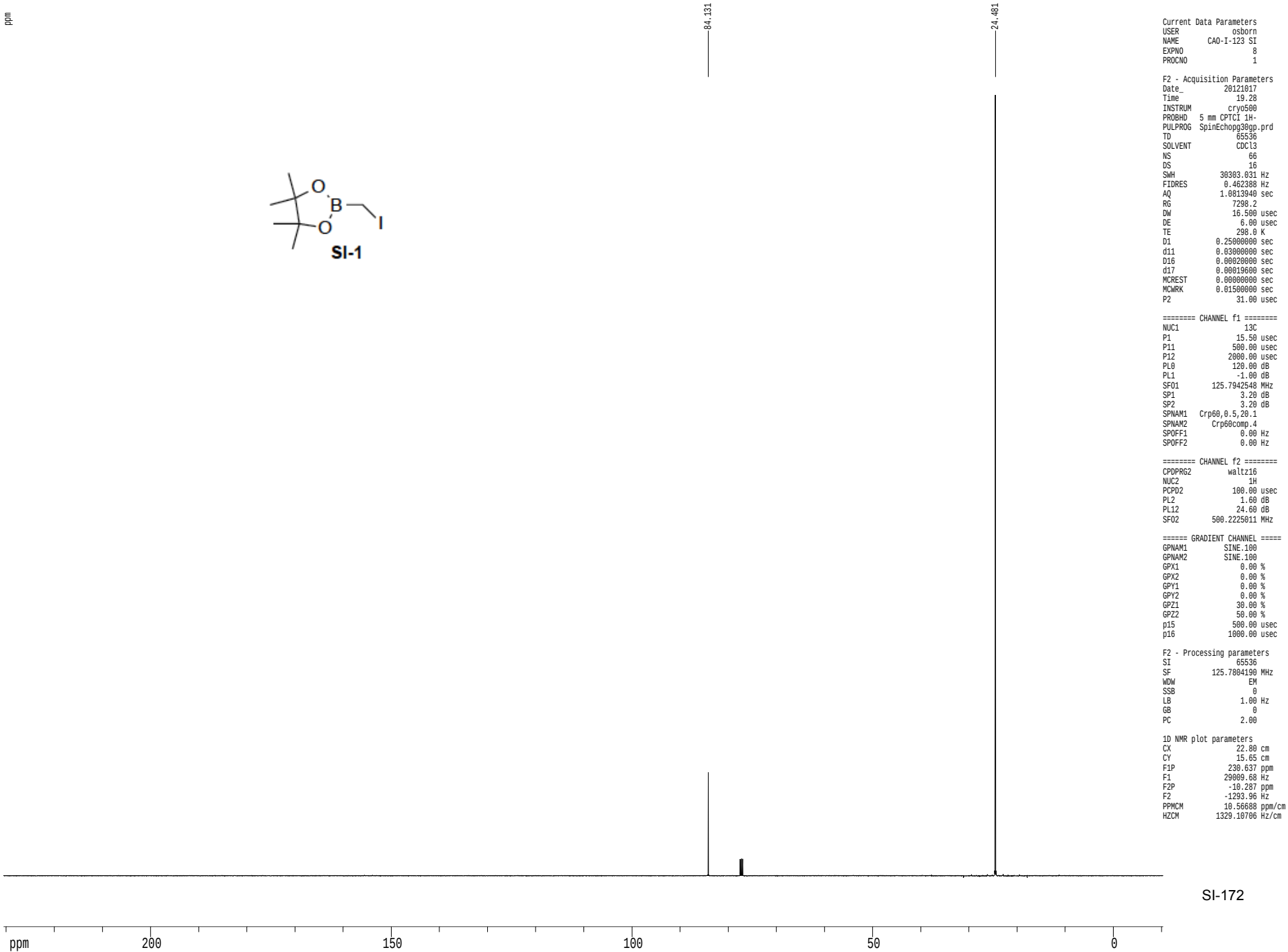

```
Current Data Parameters
USER                osborn
NAME      CAO-III-174-check
EXPNO                   4
PROCNO                  1

F2 - Acquisition Parameters
Date_           20150318
Time              12.57
INSTRUM          cryo500
PROBHD   5 mm CPTCI 1H-
PULPROG  SpinEchopg30gp.prd
TD                65536
SOLVENT           CDCl3
NS                  181
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
MCWRK          0.01500000 sec
P2                33.10 usec

======== CHANNEL f1 ========
NUC1                 13C
P1                16.55 usec
P11              500.00 usec
P12             2000.00 usec
PL0              120.00 dB
PL1               -1.00 dB
SFO1         125.7942548 MHz
SP1               2.70 dB
SP2               2.70 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              24.50 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           125.7803094 MHz
WDW                  EM
SSB                   0
LB                 1.00 Hz
GB                    0
PC                 2.00

1D NMR plot parameters
CX                22.80 cm
CY                35.00 cm
F1P             230.637 ppm
F1             29009.65 Hz
F2P             -10.287 ppm
F2             -1293.96 Hz
PPMCM          10.56688 ppm/cm
HZCM         1329.10596 Hz/cm
```

1H spectrum



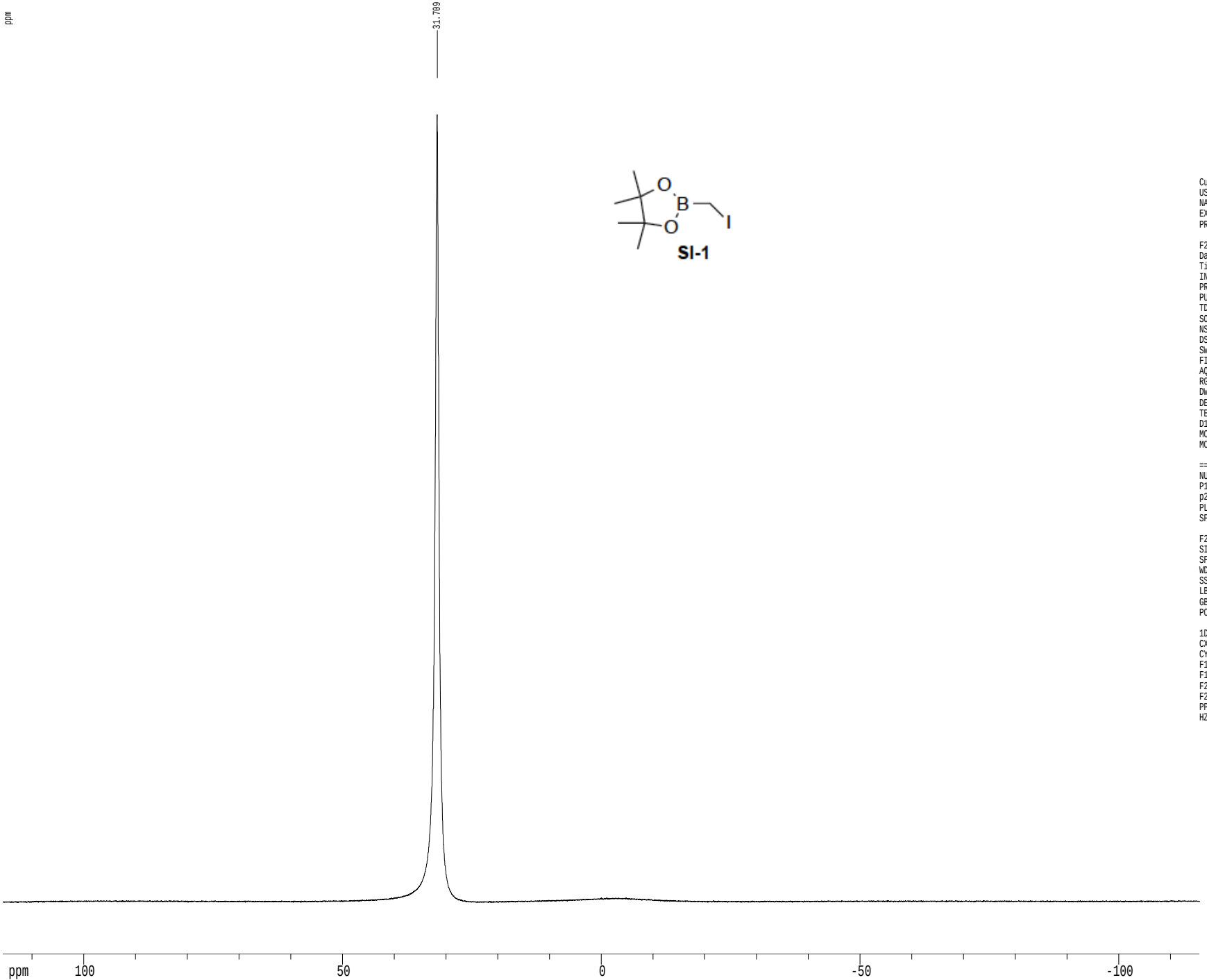

```
Current Data Parameters
USER              osborn
NAME        CAO-I-123 SI
EXPNO                  1
PROCNO                 1

F2 - Acquisition Parameters
Date_          20121017
Time               9.45
INSTRUM          drx400
PROBHD   5 mm QNP H/F/P
PULPROG             zg30
TD                 65536
SOLVENT            CDCl3
NS                     8
DS                     2
SWH             6410.256 Hz
FIDRES          0.097813 Hz
AQ              5.1118579 sec
RG                    16
DW                78.000 usec
DE                 4.50 usec
TE                298.0 K
D1           0.10000000 sec
MCREST       0.00000000 sec
MCWRK        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           400.1299870 MHz
WDW                   EM
SSB                    0
LB                  0.30 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                -0.500 ppm
F2               -200.06 Hz
PPMCM           0.41667 ppm/cm
HZCM          166.72083 Hz/cm
```

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



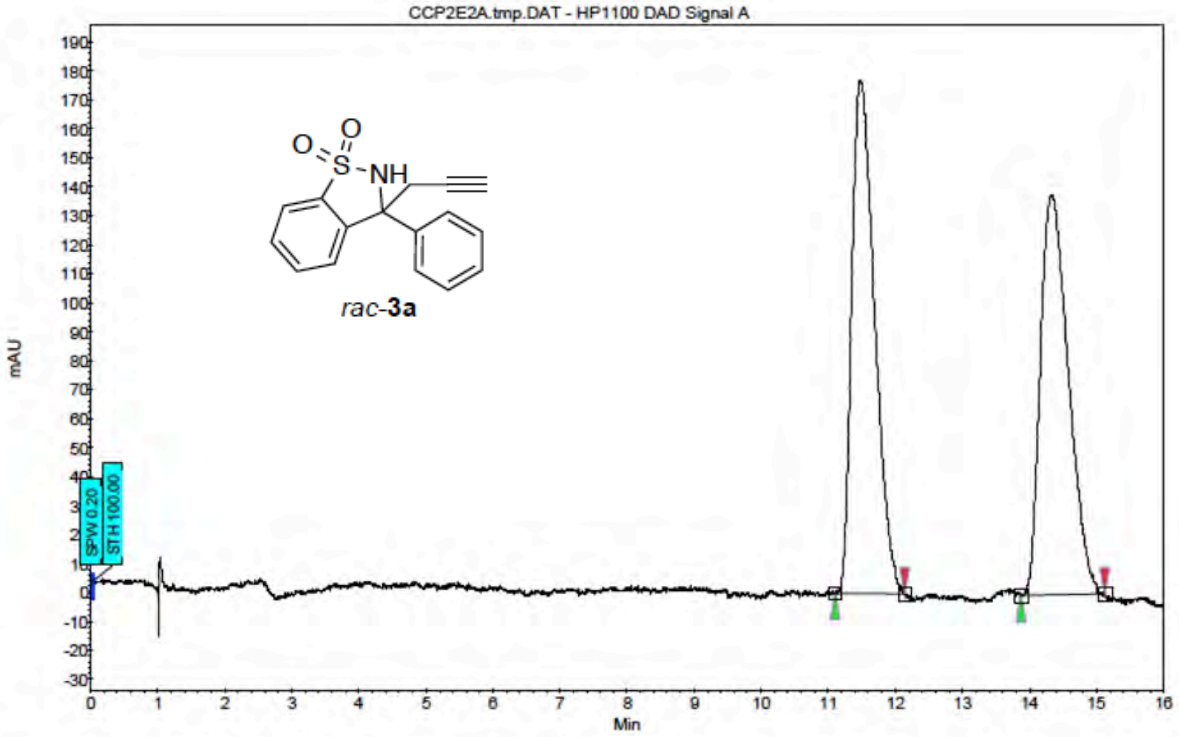

```
Current Data Parameters
USER              osborn
NAME        CAO-I-123 SI
EXPNO                  8
PROCNO                 1

F2 - Acquisition Parameters
Date_           20121017
Time               19.28
INSTRUM          cryo500
PROBHD   5 mm CPTCI 1H-
PULPROG  SpinEchopg30gp.prd
TD                 65536
SOLVENT            CDCl3
NS                    66
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
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
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 ========
CPDPRG2          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           125.7804190 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              230.637 ppm
F1             29009.68 Hz
F2P              -10.287 ppm
F2             -1293.96 Hz
PPMCM           10.56688 ppm/cm
HZCM          1329.10706 Hz/cm
```

11B spectrum with 1H decoupling with background suppression

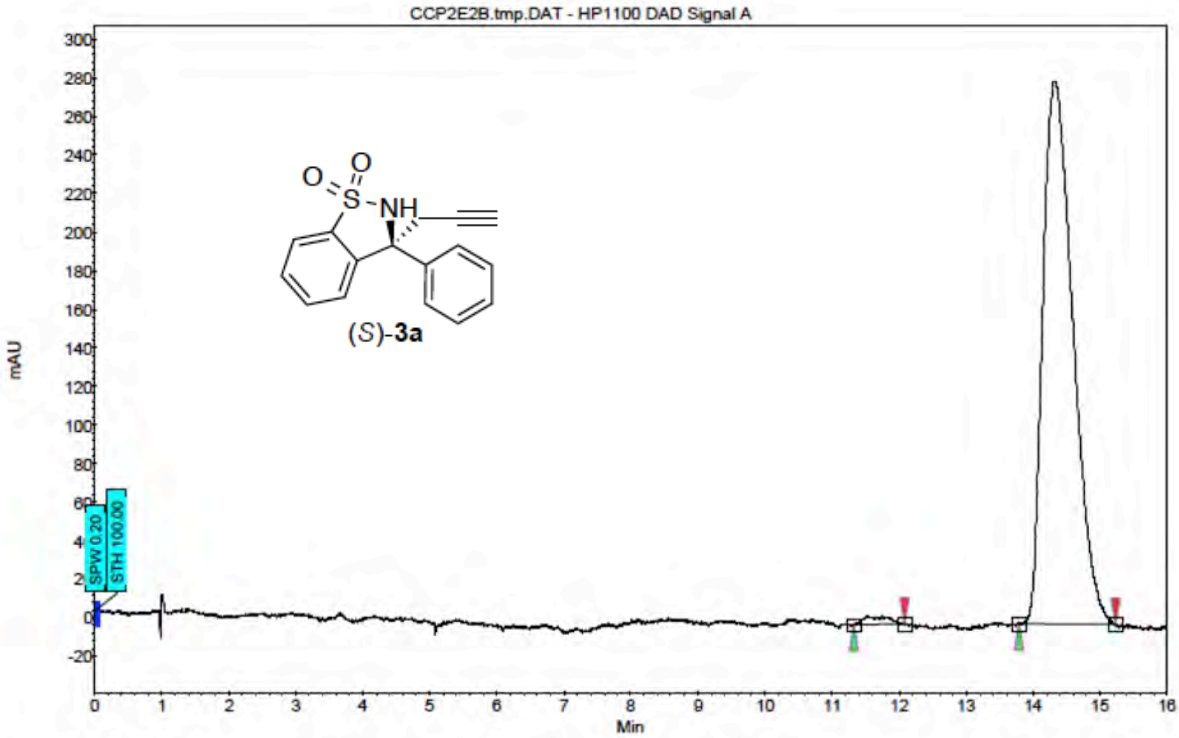


SI-1

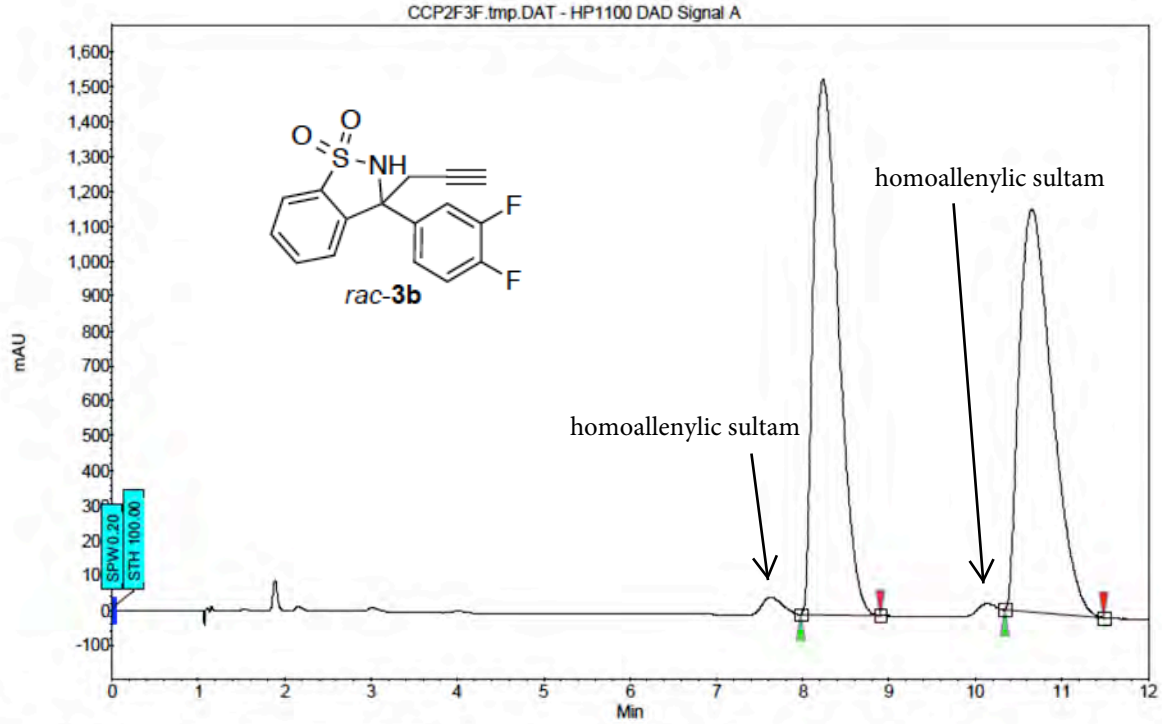
```
Current Data Parameters
USER             osborn
NAME       CAO-I-123 SI
EXPNO                 5
PROCNO                1

F2 - Acquisition Parameters
Date_          20121017
Time              10.21
INSTRUM           gn500
PROBHD   5 mm broadband
PULPROG            zgbs
TD                65536
SOLVENT           CDCl3
NS                   64
DS                    4
SWH           37037.035 Hz
FIDRES         0.565140 Hz
AQ             0.8847860 sec
RG                 90.5
DW                13.500 usec
DE                 6.00 usec
TE                298.0 K
D1           1.00000000 sec
MCREST       0.00000000 sec
MCWRK        0.01500000 sec

======== CHANNEL f1 ========
NUC1               11B
P1                 8.65 usec
p2                17.30 usec
PL1               -3.00 dB
SFO1        160.2273660 MHz

F2 - Processing parameters
SI                65536
SF          160.2273621 MHz
WDW                  EM
SSB                   0
LB                 2.00 Hz
GB                    0
PC                 2.00

1D NMR plot parameters
CX                22.80 cm
CY                15.00 cm
F1P             115.601 ppm
F1             18522.36 Hz
F2P            -115.552 ppm
F2            -18514.67 Hz
PPMCM          10.13829 ppm/cm
HZCM         1624.43140 Hz/cm
```



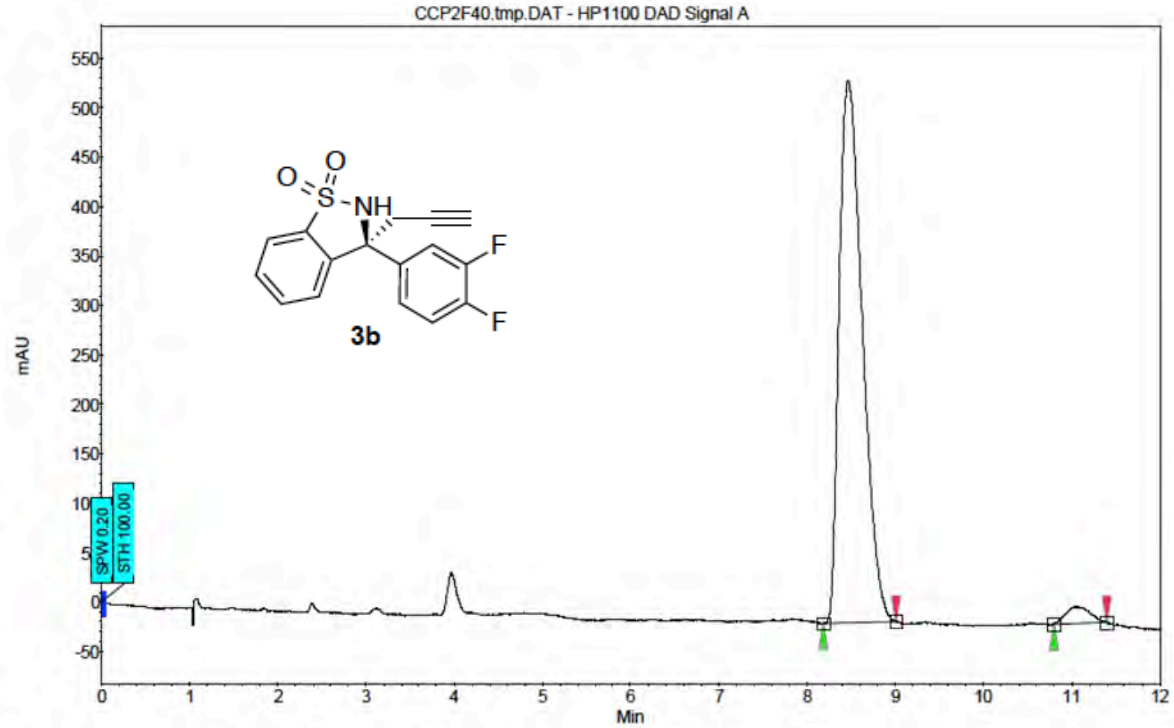
Index	Name	Start	Time	End	RT Offset	Quantity	Height	Area	Area
		[Min]	[Min]	[Min]	[Min]	[% Area]	[µV]	[µV.Min]	[%]
1	UNKNOWN	11.10	11.48	12.14	0.00	51.26	177.2	70.1	51.263
2	UNKNOWN	13.87	14.33	15.12	0.00	48.74	138.2	66.6	48.737



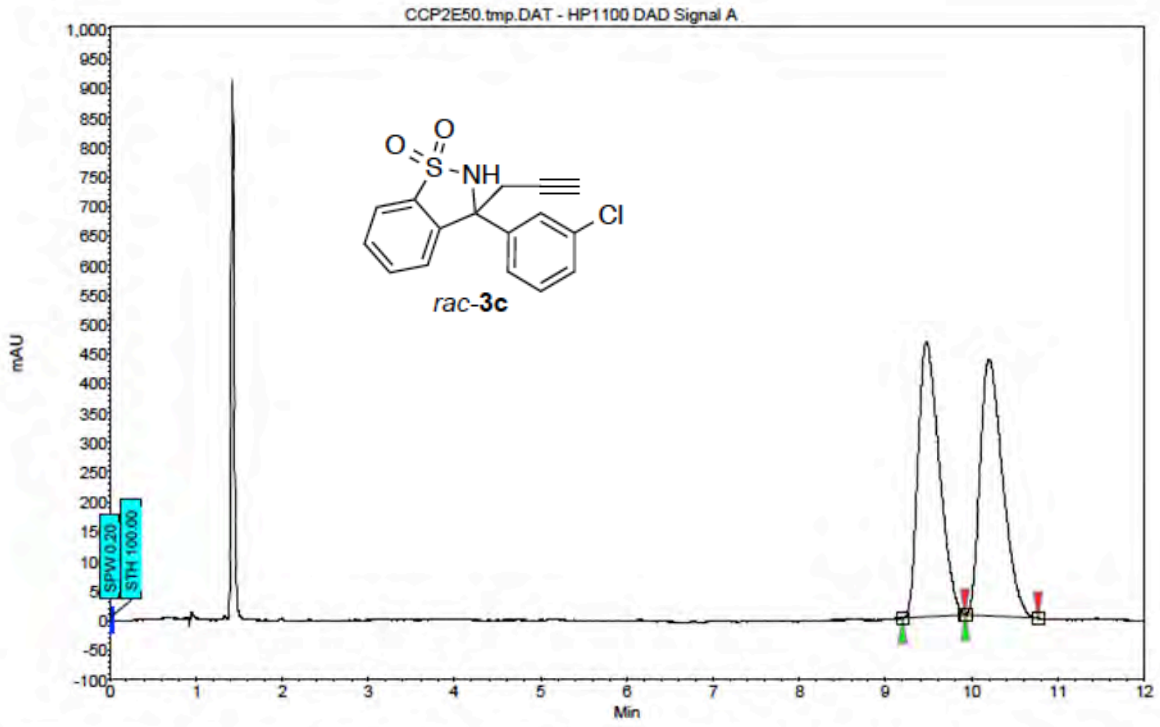
Index	Name	Start	Time	End	RT Offset	Quantity	Height	Area	Area
		[Min]	[Min]	[Min]	[Min]	[% Area]	[µV]	[µV.Min]	[%]
1	UNKNOWN	11.33	11.54	12.08	0.00	1.19	4.6	1.8	1.190
2	UNKNOWN	13.79	14.32	15.23	0.00	98.81	281.6	146.1	98.810
Total						100.00	286.2	147.9	100.000



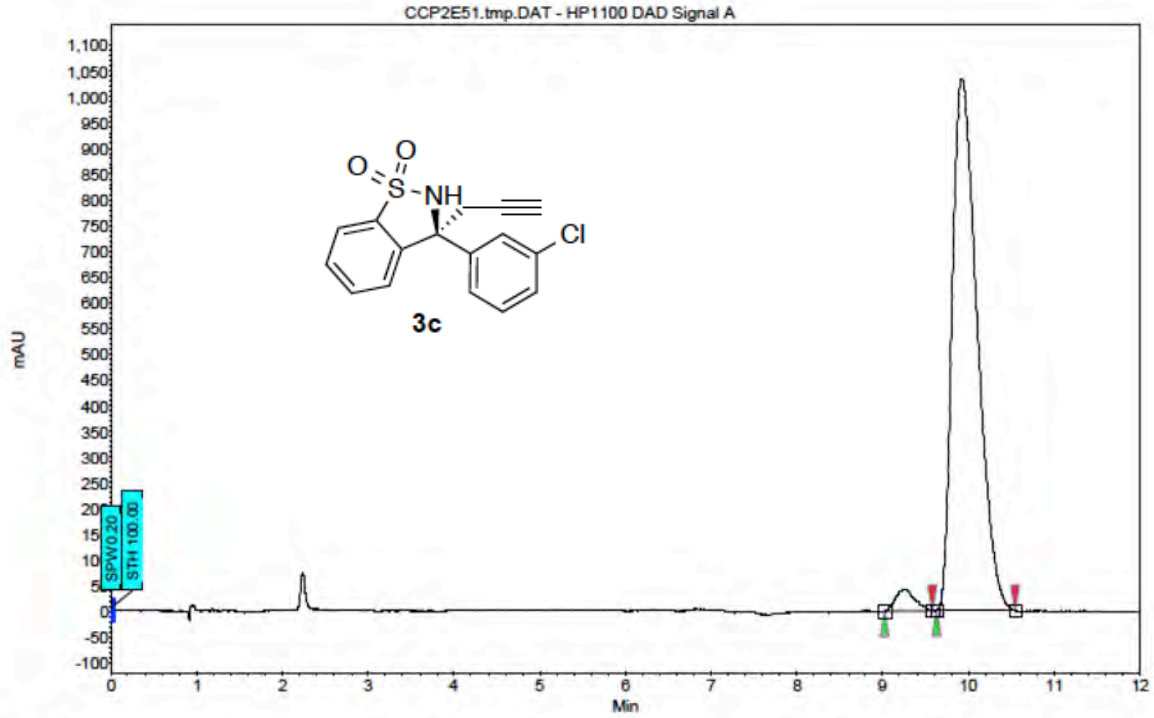
Index	Name	Start	Time	End	RT Offset	Quantity	Height	Area	Area
		[Min]	[Min]	[Min]	[Min]	[% Area]	[µV]	[µV.Min]	[%]
1	UNKNOWN	7.97	8.24	8.90	0.00	50.40	1534.9	516.6	50.402
2	UNKNOWN	10.34	10.65	11.48	0.00	49.60	1156.1	508.4	49.598
Total						100.00	2691.0	1025.0	100.000



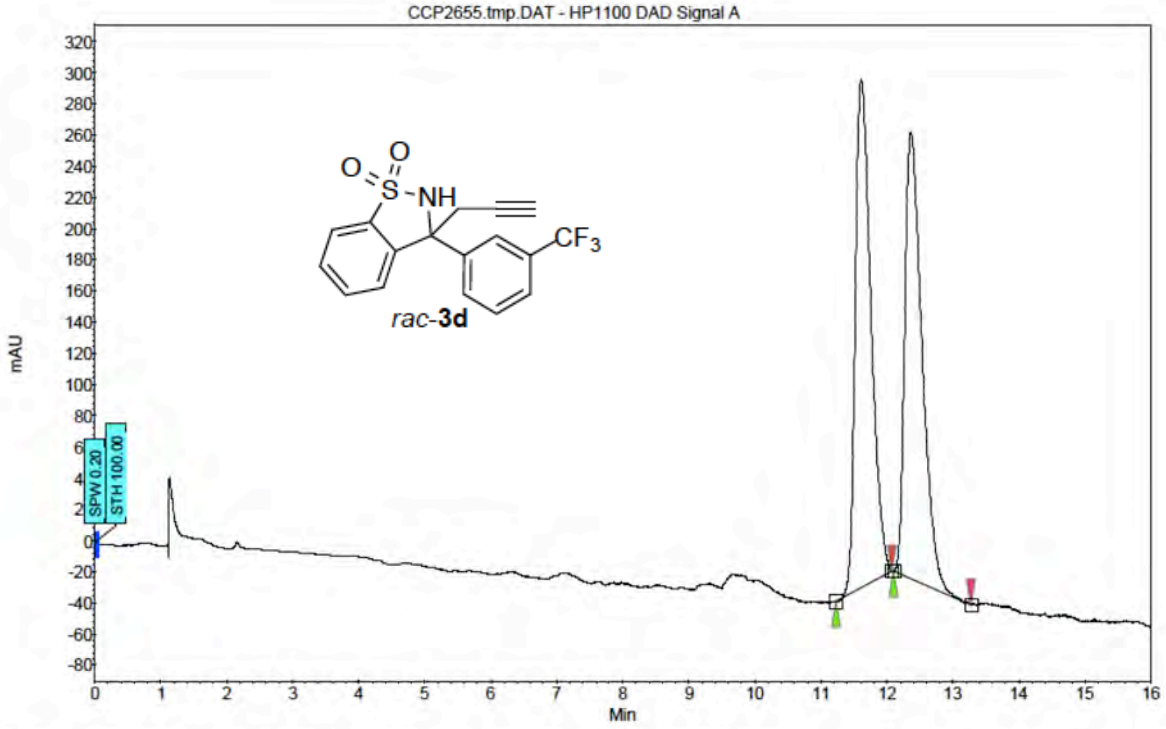
Index	Name	Start	Time	End	RT Offset	Quantity	Height	Area	Area
		[Min]	[Min]	[Min]	[Min]	[% Area]	[µV]	[µV.Min]	[%]
1	UNKNOWN	8.18	8.47	9.01	0.00	96.84	548.7	166.2	96.840
2	UNKNOWN	10.80	11.04	11.40	0.00	3.16	17.4	5.4	3.160
Total						100.00	566.0	171.6	100.000



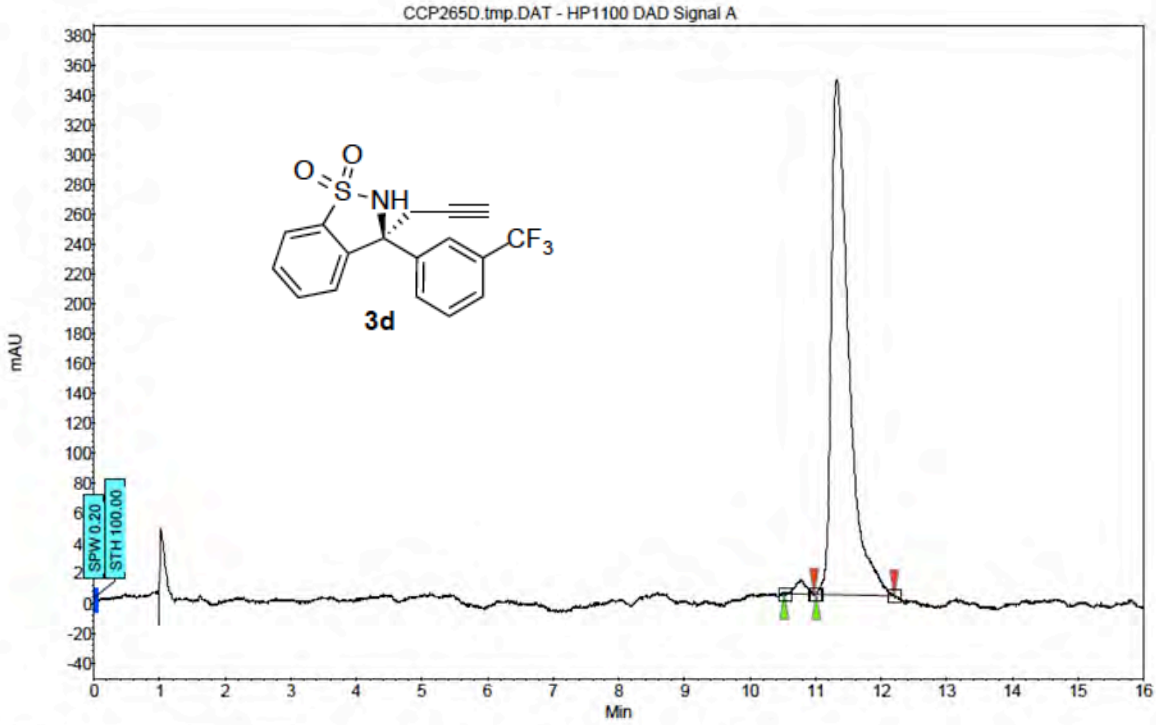
Index	Name	Start	Time	End	RT Offset	Quantity	Height	Area	Area
		[Min]	[Min]	[Min]	[Min]	[% Area]	[µV]	[µV.Min]	[%]
1	UNKNOWN	9.19	9.47	9.91	0.00	50.06	464.5	132.2	50.064
2	UNKNOWN	9.92	10.20	10.77	0.00	49.94	434.4	131.8	49.936
Total						100.00	899.0	264.0	100.000



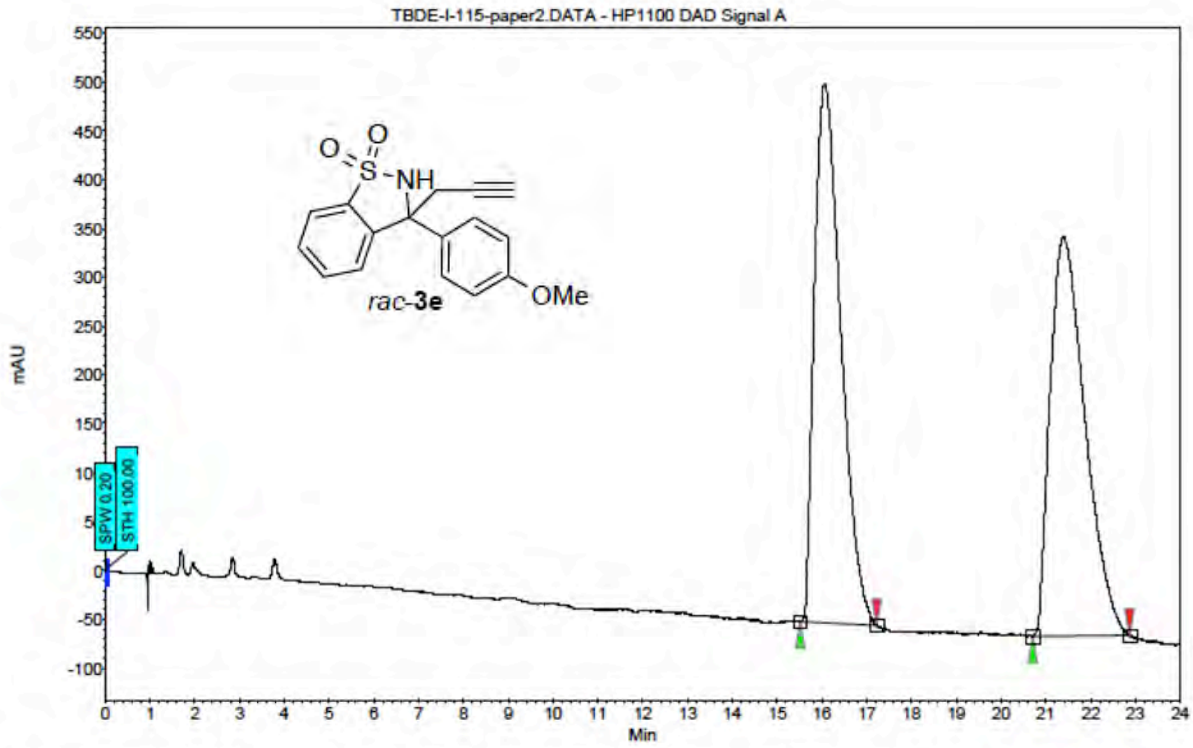
Index	Name	Start	Time	End	RT Offset	Quantity	Height	Area	Area
		[Min]	[Min]	[Min]	[Min]	[% Area]	[µV]	[µV.Min]	[%]
1	UNKNOWN	9.02	9.24	9.58	0.00	3.02	41.6	10.8	3.022
2	UNKNOWN	9.63	9.92	10.54	0.00	96.98	1033.9	347.8	96.978
Total						100.00	1075.5	358.7	100.000



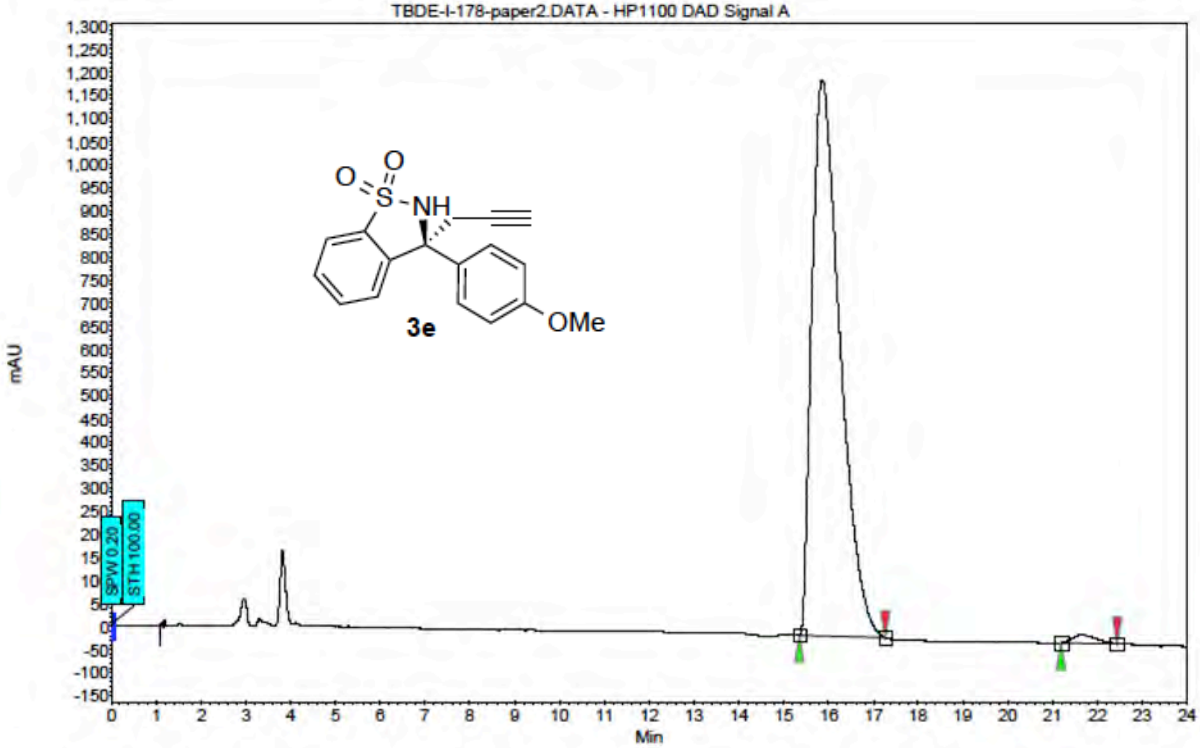
Index	Name	Start	Time	End	RT Offset	Quantity	Height	Area	Area
		[Min]	[Min]	[Min]	[Min]	[% Area]	[µV]	[µV.Min]	[%]
1	UNKNOWN	11.23	11.61	12.07	0.00	50.60	325.9	88.7	50.596
2	UNKNOWN	12.10	12.37	13.27	0.00	49.40	286.1	86.6	49.404
Total						100.00	612.0	175.4	100.000



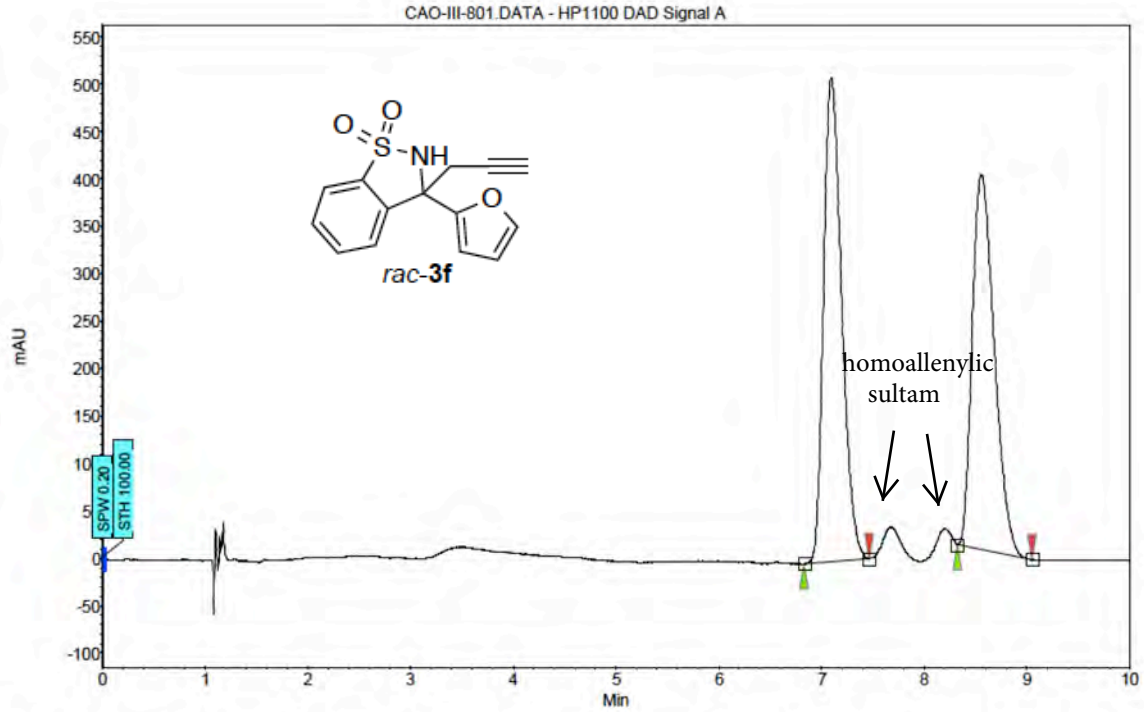
Index	Name	Start	Time	End	RT Offset	Quantity	Height	Area	Area
		[Min]	[Min]	[Min]	[Min]	[% Area]	[µV]	[µV.Min]	[%]
1	UNKNOWN	10.53	10.77	10.98	0.00	1.61	9.2	1.7	1.611
2	UNKNOWN	11.01	11.33	12.20	0.00	98.39	344.7	105.1	98.389
Total						100.00	353.9	106.8	100.000



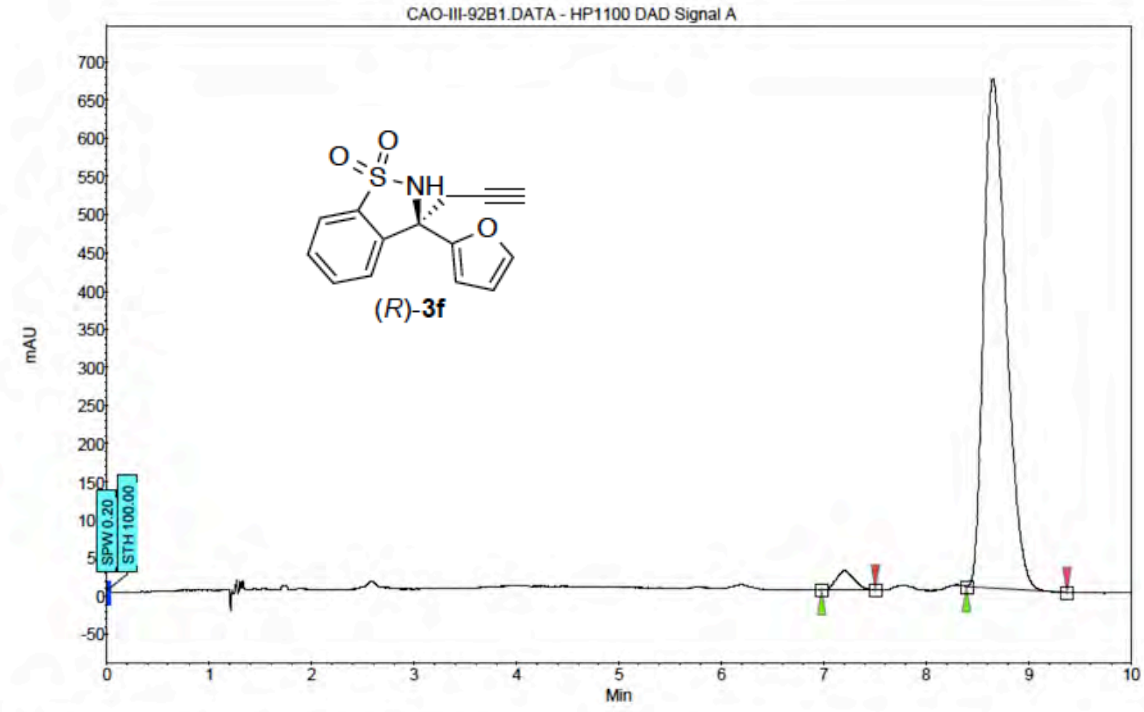
Index	Name	Start	Time	End	RT Offset	Quantity	Height	Area	Area
		[Min]	[Min]	[Min]	[Min]	[% Area]	[µV]	[µV.Min]	[%]
1	UNKNOWN	15.52	16.06	17.23	0.00	50.58	552.1	377.4	50.582
2	UNKNOWN	20.71	21.39	22.87	0.00	49.42	409.4	368.7	49.418
Total						100.00	961.5	746.1	100.000



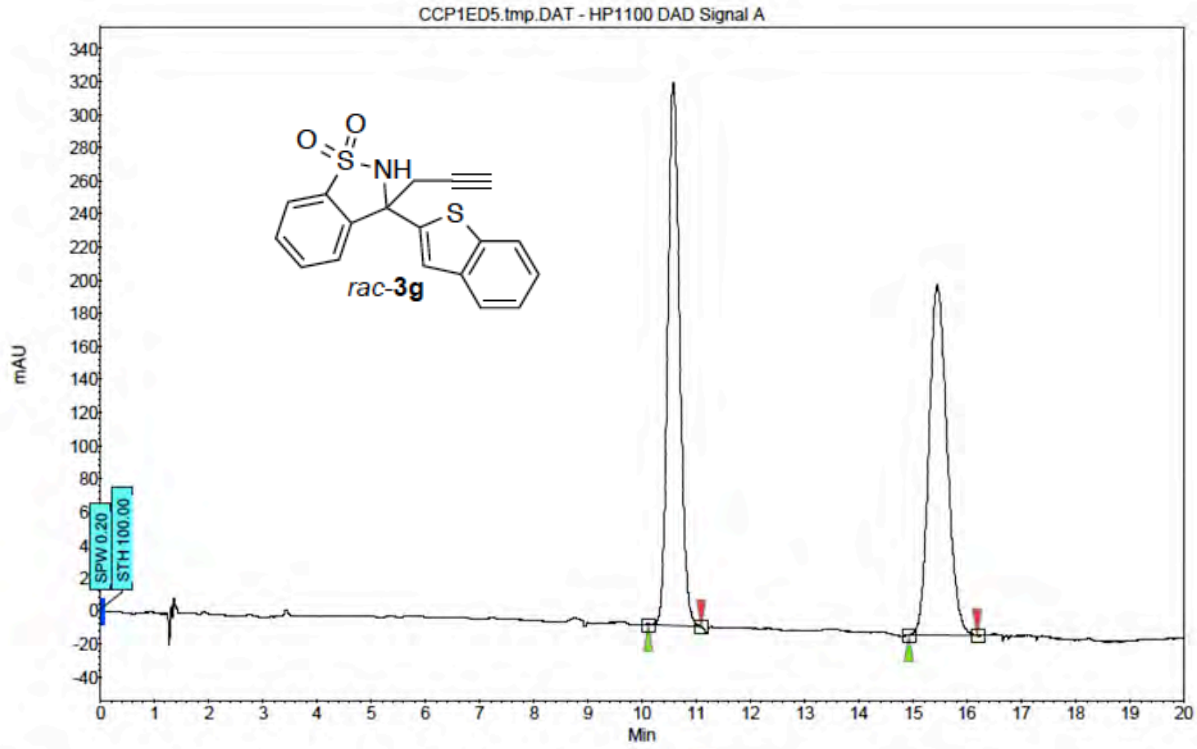
Index	Name	Start	Time	End	RT Offset	Quantity	Height	Area	Area
		[Min]	[Min]	[Min]	[Min]	[% Area]	[µV]	[µV.Min]	[%]
1	UNKNOWN	15.35	15.87	17.26	0.00	98.75	1202.6	870.4	98.747
2	UNKNOWN	21.19	21.68	22.44	0.00	1.25	17.8	11.0	1.253
Total						100.00	1220.4	881.4	100.000



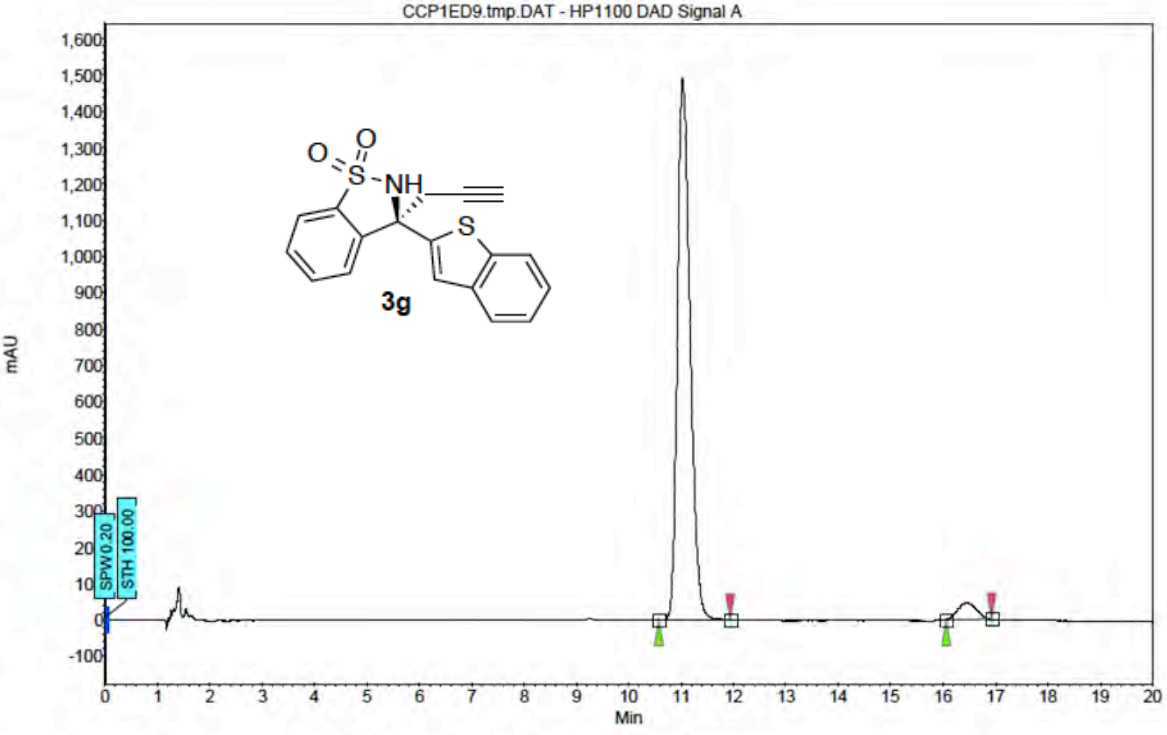
Index	Name	Start	Time	End	RT Offset	Quantity	Height	Area	Area
		[Min]	[Min]	[Min]	[Min]	[% Area]	[µV]	[µV.Min]	[%]
1	UNKNOWN	6.83	7.09	7.46	0.00	51.48	510.0	104.0	51.480
2	UNKNOWN	8.32	8.55	9.05	0.00	48.52	395.3	98.0	48.520
Total						100.00	905.3	202.0	100.000



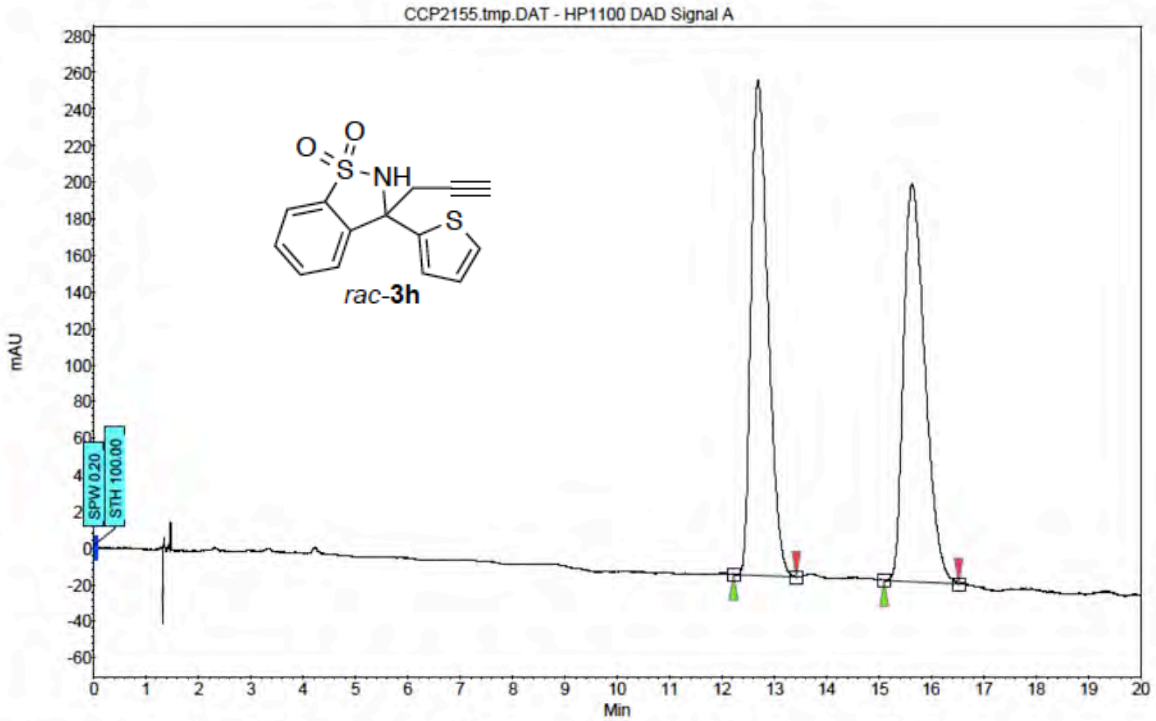
Index	Name	Start	Time	End	RT Offset	Quantity	Height	Area	Area
		[Min]	[Min]	[Min]	[Min]	[% Area]	[µV]	[µV.Min]	[%]
1	UNKNOWN	6.98	7.20	7.50	0.00	2.78	24.7	4.9	2.778
2	UNKNOWN	8.39	8.65	9.37	0.00	97.22	667.3	170.0	97.222
Total						100.00	692.0	174.8	100.000



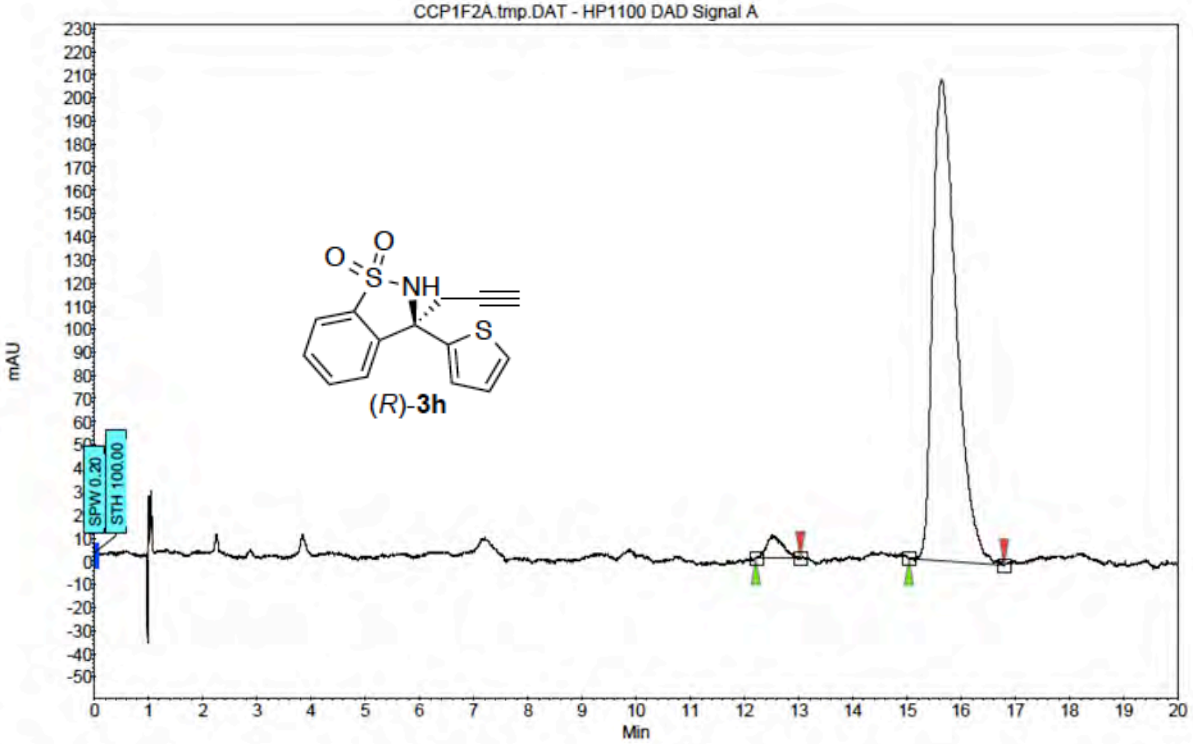
Index	Name	Start	Time	End	RT Offset	Quantity	Height	Area	Area
		[Min]	[Min]	[Min]	[Min]	[% Area]	[µV]	[µV.Min]	[%]
1	UNKNOWN	10.12	10.58	11.09	0.00	50.44	328.0	83.7	50.437
2	UNKNOWN	14.92	15.45	16.18	0.00	49.56	211.6	82.3	49.563
Total						100.00	539.6	166.0	100.000



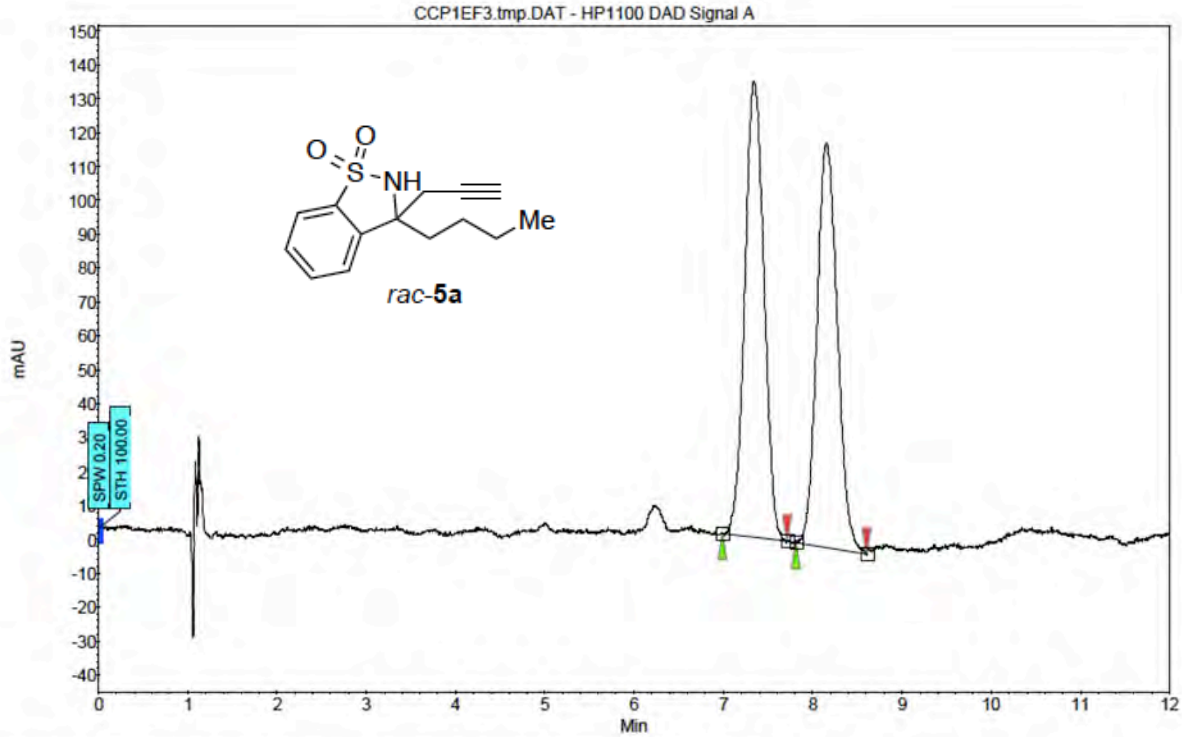
Index	Name	Start	Time	End	RT Offset	Quantity	Height	Area	Area
		[Min]	[Min]	[Min]	[Min]	[% Area]	[µV]	[µV.Min]	[%]
1	UNKNOWN	10.57	11.03	11.94	0.00	95.62	1491.4	417.1	95.617
2	UNKNOWN	16.05	16.46	16.92	0.00	4.38	46.7	19.1	4.383
Total						100.00	1538.0	436.3	100.000



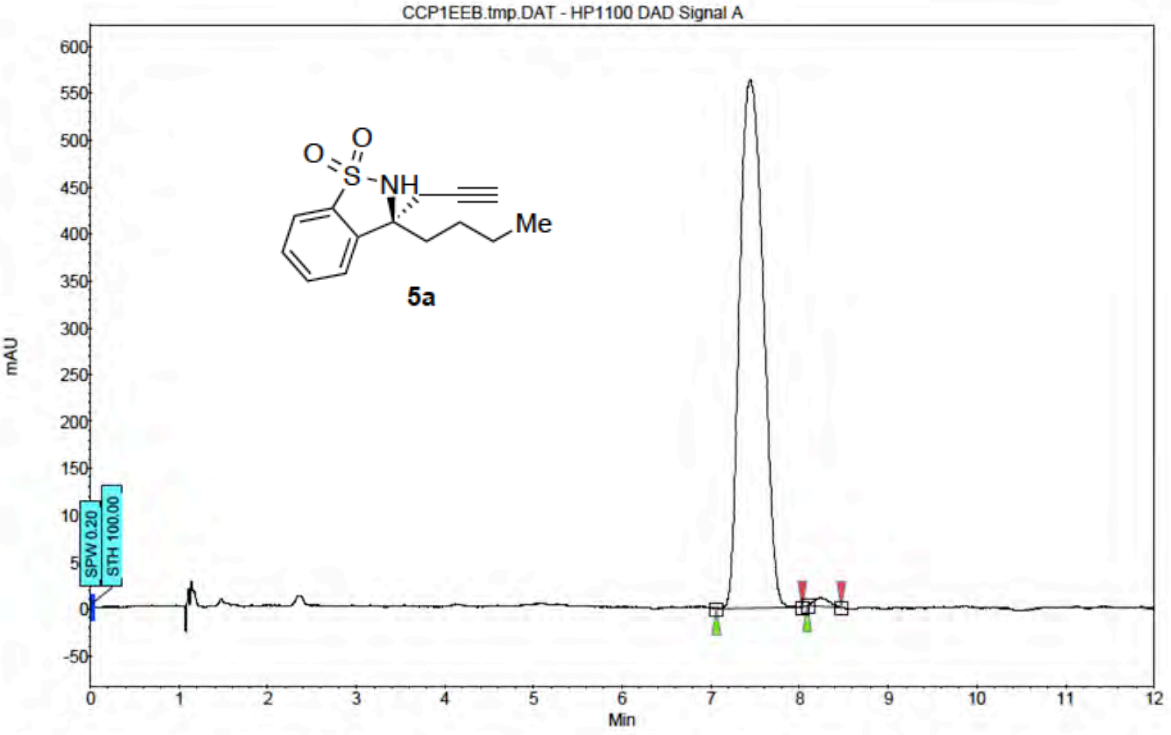
Index	Name	Start	Time	End	RT Offset	Quantity	Height	Area	Area
		[Min]	[Min]	[Min]	[Min]	[% Area]	[µV]	[µV.Min]	[%]
1	UNKNOWN	12.22	12.68	13.42	0.00	49.78	270.8	101.5	49.777
2	UNKNOWN	15.09	15.63	16.51	0.00	50.22	217.6	102.4	50.223
Total						100.00	488.4	203.9	100.000



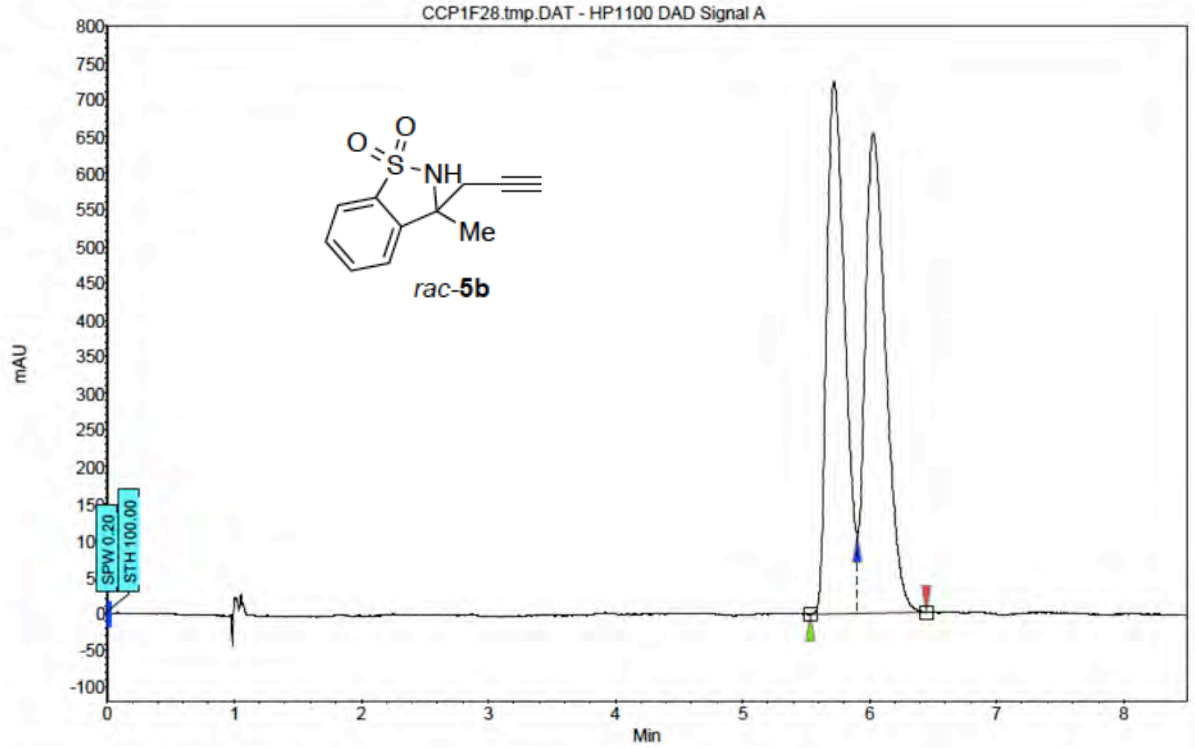
Index	Name	Start	Time	End	RT Offset	Quantity	Height	Area	Area
		[Min]	[Min]	[Min]	[Min]	[% Area]	[µV]	[µV.Min]	[%]
1	UNKNOWN	12.21	12.52	13.03	0.00	3.10	9.8	3.3	3.102
2	UNKNOWN	15.03	15.65	16.79	0.00	96.90	207.5	103.4	96.898
Total						100.00	217.4	106.7	100.000



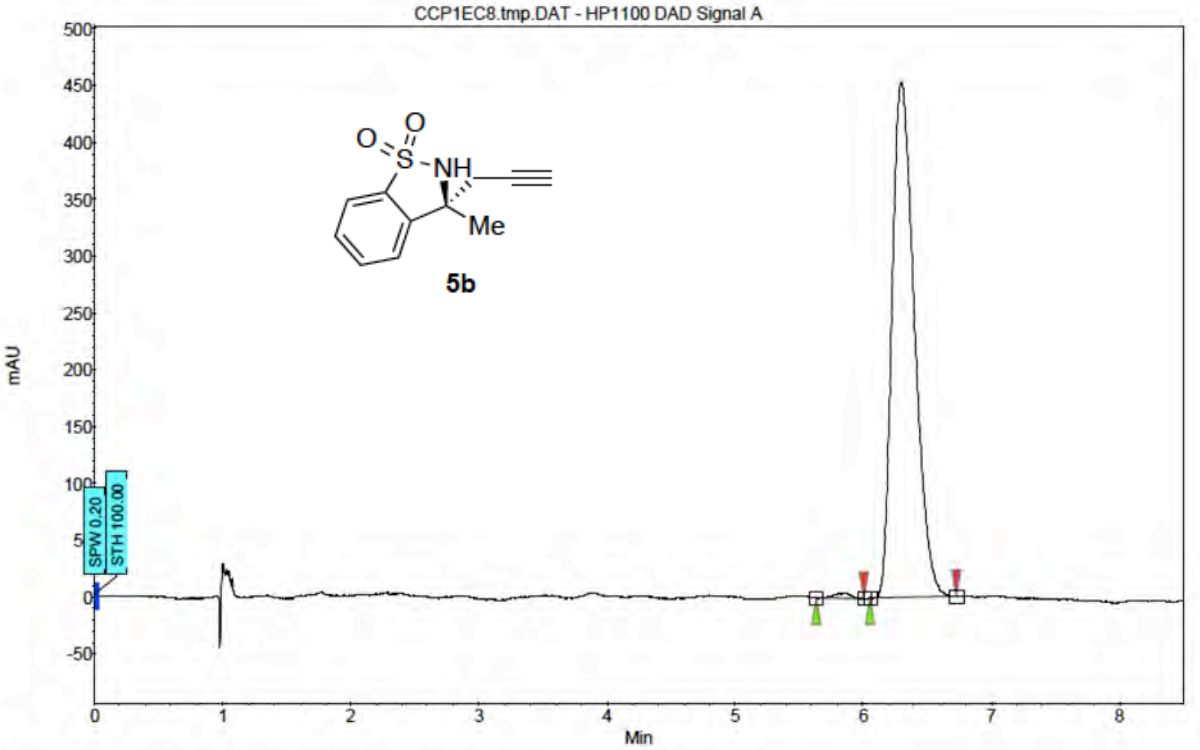
Index	Name	Start	Time	End	RT Offset	Quantity	Height	Area	Area
		[Min]	[Min]	[Min]	[Min]	[% Area]	[µV]	[µV.Min]	[%]
1	UNKNOWN	6.99	7.34	7.72	0.00	51.18	134.7	33.3	51.180
2	UNKNOWN	7.81	8.15	8.61	0.00	48.82	119.6	31.7	48.820
Total						100.00	254.3	65.0	100.000



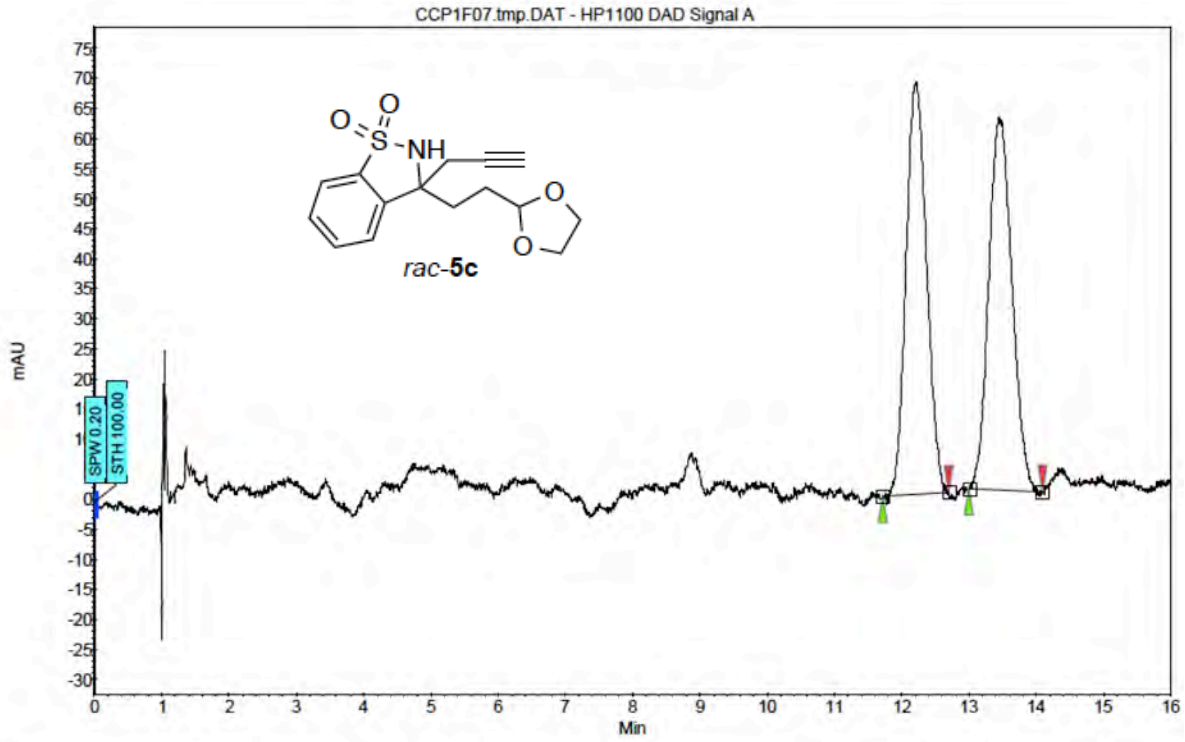
Index	Name	Start	Time	End	RT Offset	Quantity	Height	Area	Area
		[Min]	[Min]	[Min]	[Min]	[% Area]	[µV]	[µV.Min]	[%]
1	UNKNOWN	7.06	7.45	8.03	0.00	98.96	563.5	172.9	98.965
2	UNKNOWN	8.09	8.26	8.47	0.00	1.04	9.5	1.8	1.035
Total						100.00	573.0	174.7	100.000



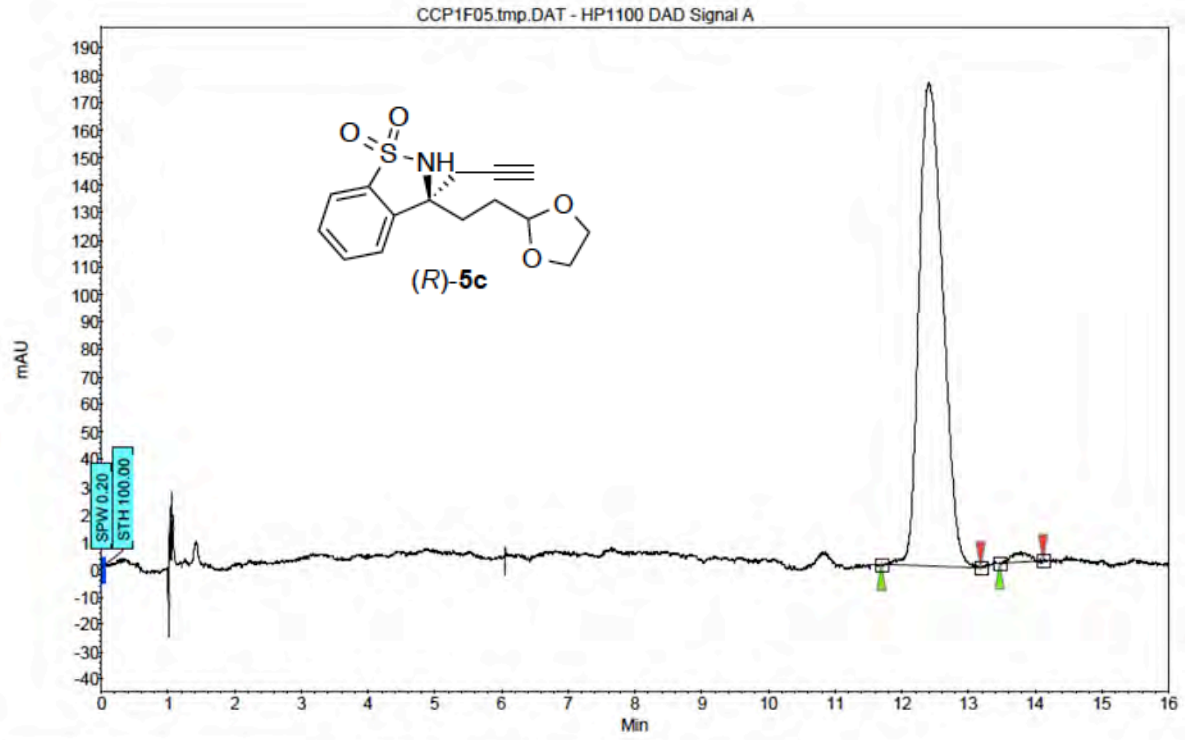
Index	Name	Start	Time	End	RT Offset	Quantity	Height	Area	Area
		[Min]	[Min]	[Min]	[Min]	[% Area]	[µV]	[µV.Min]	[%]
1	UNKNOWN	5.54	5.72	5.90	0.00	49.03	724.8	118.8	49.029
2	UNKNOWN	5.90	6.03	6.45	0.00	50.97	652.4	123.5	50.971
Total						100.00	1377.2	242.4	100.000



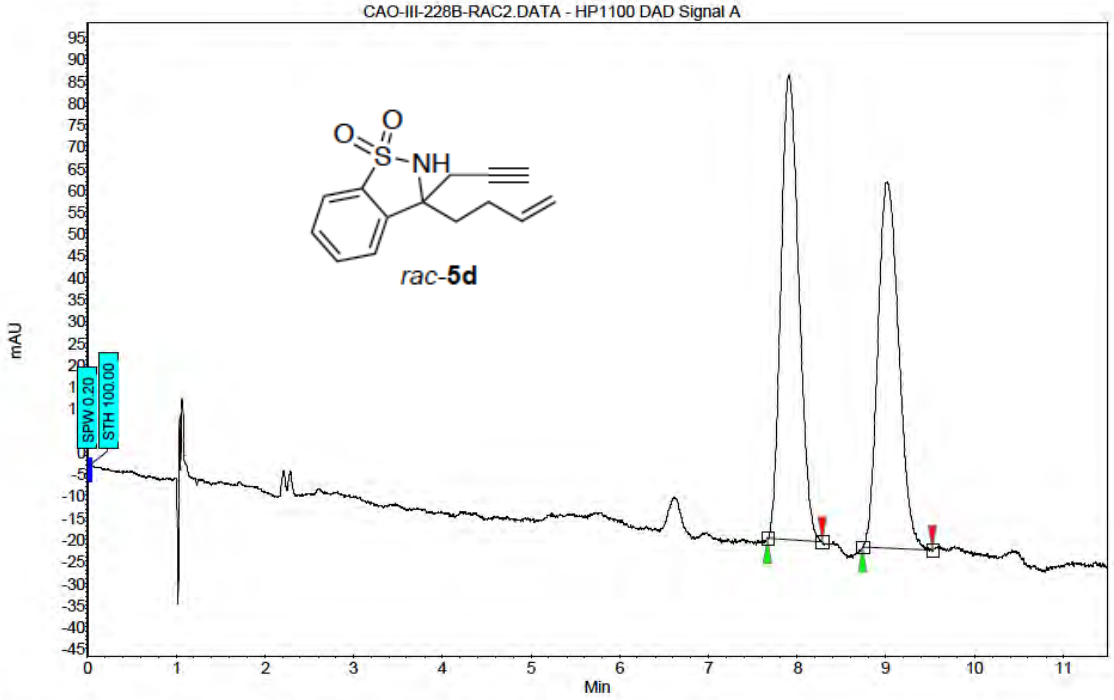
Index	Name	Start	Time	End	RT Offset	Quantity	Height	Area	Area
		[Min]	[Min]	[Min]	[Min]	[% Area]	[µV]	[µV.Min]	[%]
1	UNKNOWN	5.63	5.81	6.00	0.00	0.78	4.6	0.7	0.781
2	UNKNOWN	6.05	6.30	6.72	0.00	99.22	453.1	91.3	99.219
Total						100.00	457.6	92.0	100.000



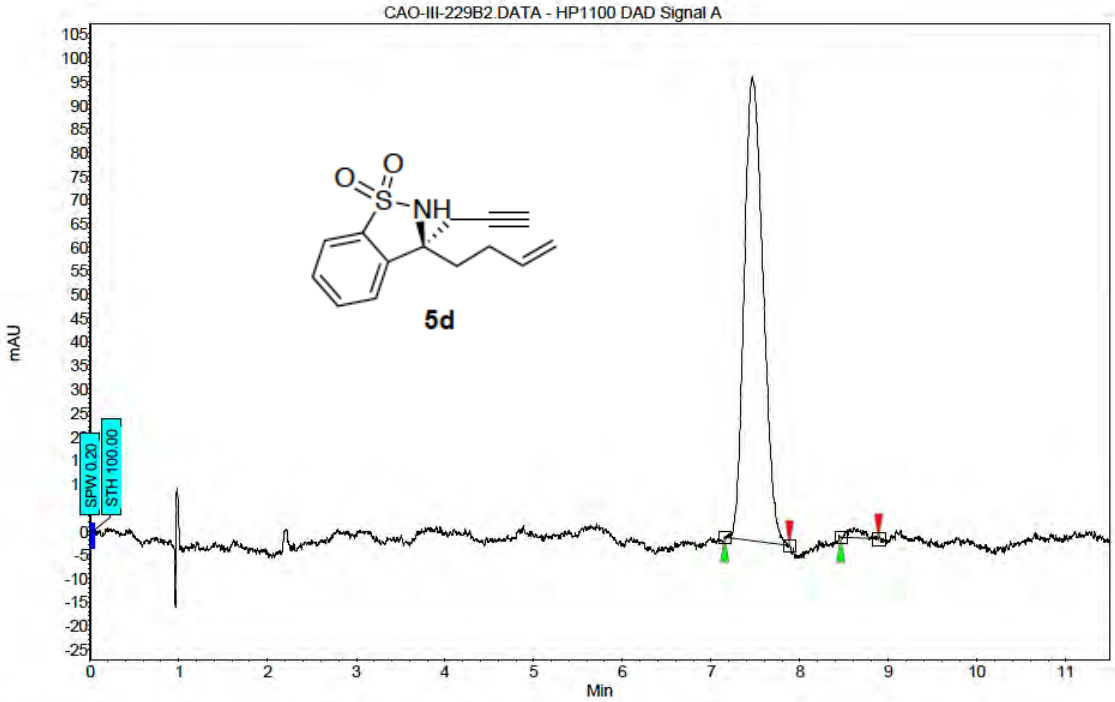
Index	Name	Start	Time	End	RT Offset	Quantity	Height	Area	Area
		[Min]	[Min]	[Min]	[Min]	[% Area]	[µV]	[µV.Min]	[%]
1	UNKNOWN	11.72	12.21	12.69	0.00	49.61	68.6	24.2	49.609
2	UNKNOWN	13.00	13.45	14.09	0.00	50.39	62.0	24.6	50.391
Total						100.00	130.5	48.9	100.000



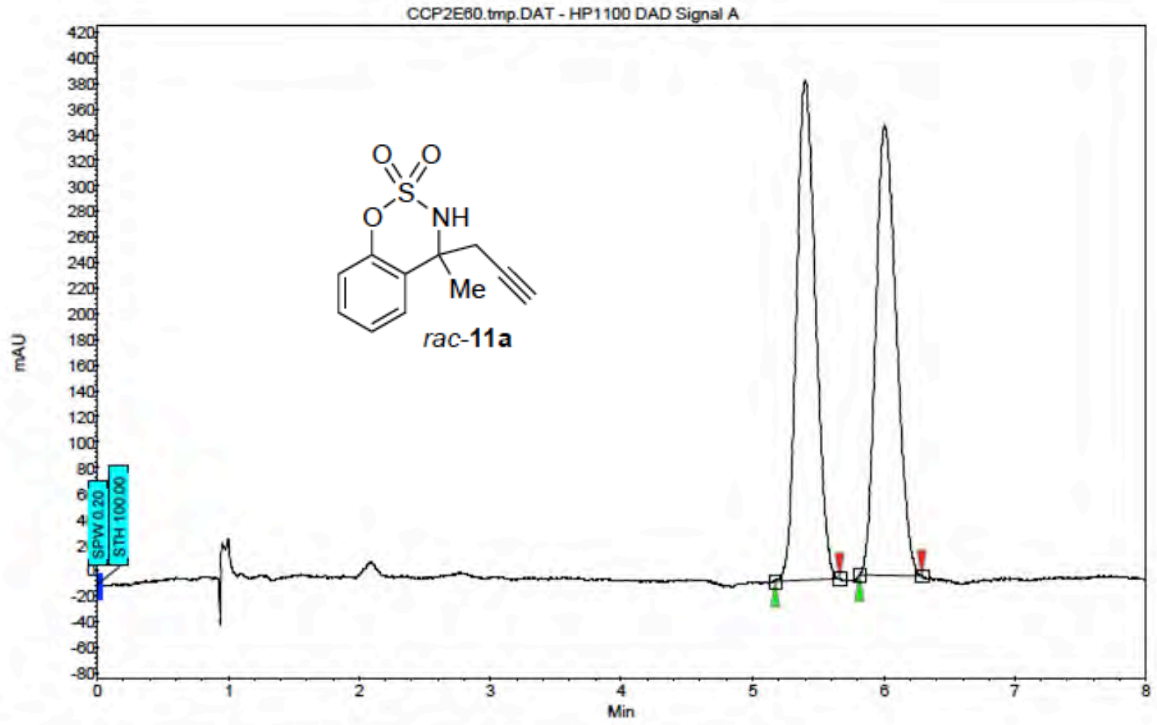
Index	Name	Start	Time	End	RT Offset	Quantity	Height	Area	Area
		[Min]	[Min]	[Min]	[Min]	[% Area]	[µV]	[µV.Min]	[%]
1	UNKNOWN	11.69	12.41	13.18	0.00	98.62	176.3	72.6	98.615
2	UNKNOWN	13.47	13.78	14.11	0.00	1.38	3.5	1.0	1.385
Total						100.00	179.8	73.6	100.000



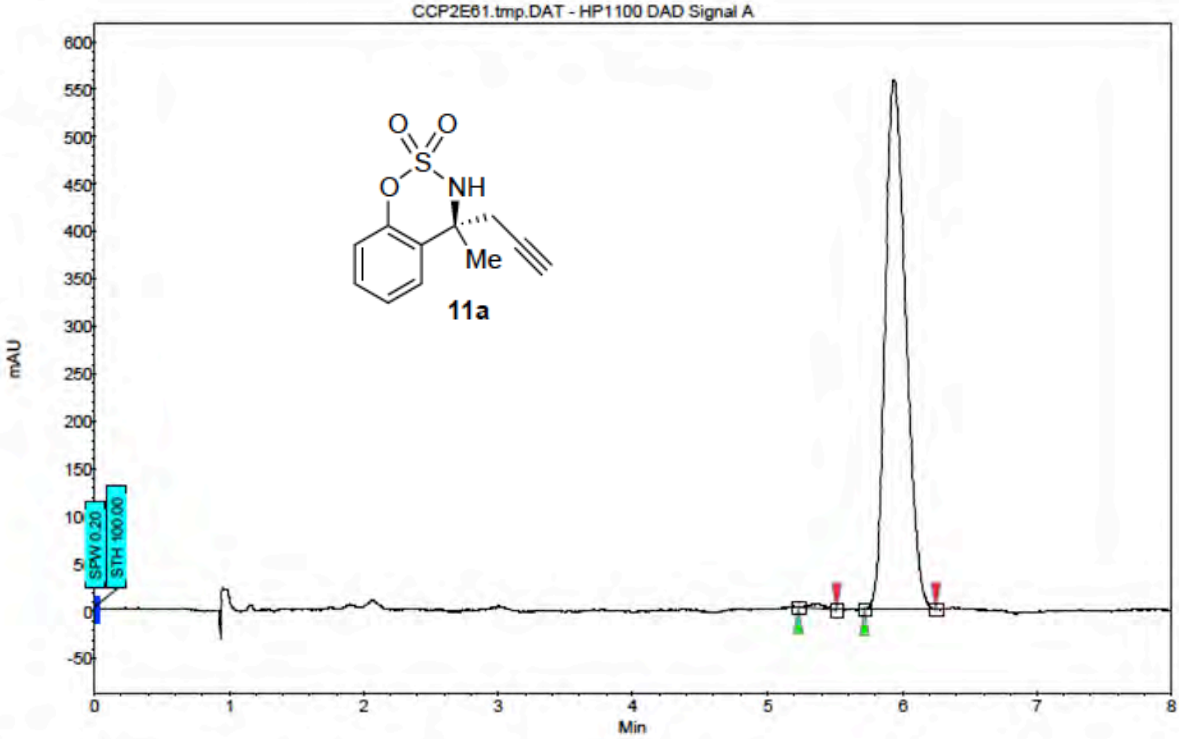
Index	Name	Start	Time	End	RT Offset	Quantity	Height	Area	Area
		[Min]	[Min]	[Min]	[Min]	[% Area]	[µV]	[µV.Min]	[%]
1	UNKNOWN	7.66	7.91	8.28	0.00	52.12	106.2	24.9	52.119
2	UNKNOWN	8.73	9.02	9.52	0.00	47.88	84.0	22.9	47.881
Total						100.00	190.2	47.8	100.000



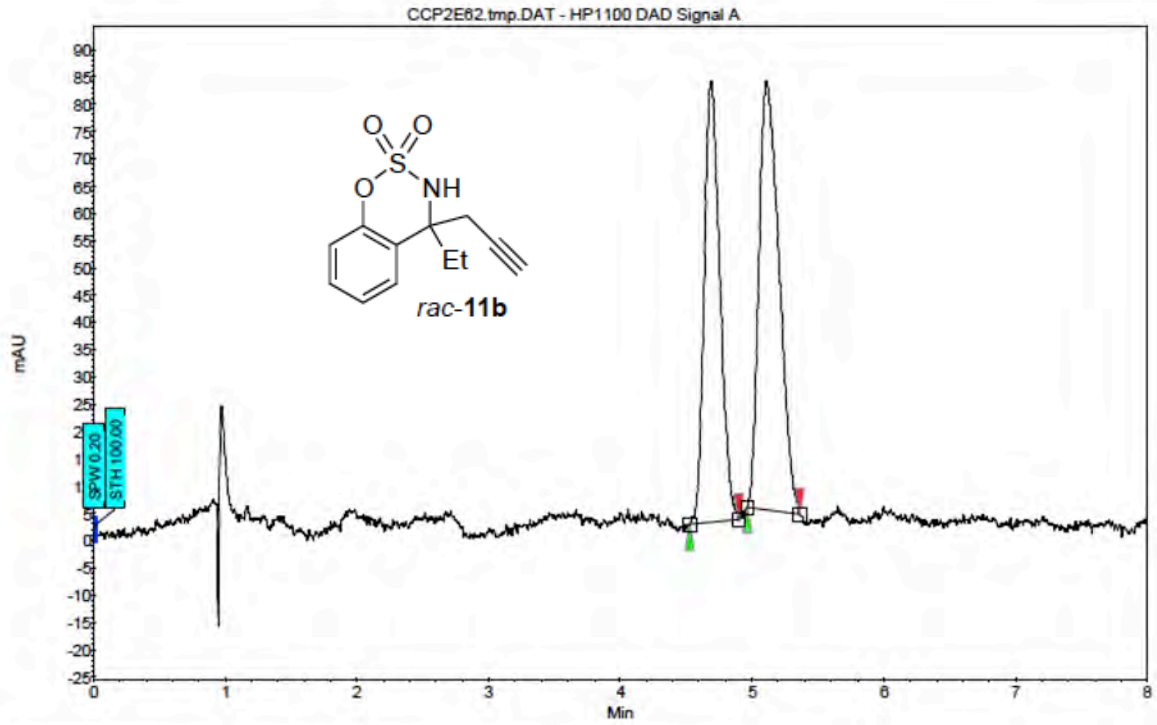
Index	Name	Start	Time	End	RT Offset	Quantity	Height	Area	Area
		[Min]	[Min]	[Min]	[Min]	[% Area]	[µV]	[µV.Min]	[%]
1	UNKNOWN	7.15	7.46	7.88	0.00	98.46	98.0	24.1	98.463
2	UNKNOWN	8.46	8.62	8.89	0.00	1.54	2.3	0.4	1.537
Total						100.00	100.2	24.5	100.000



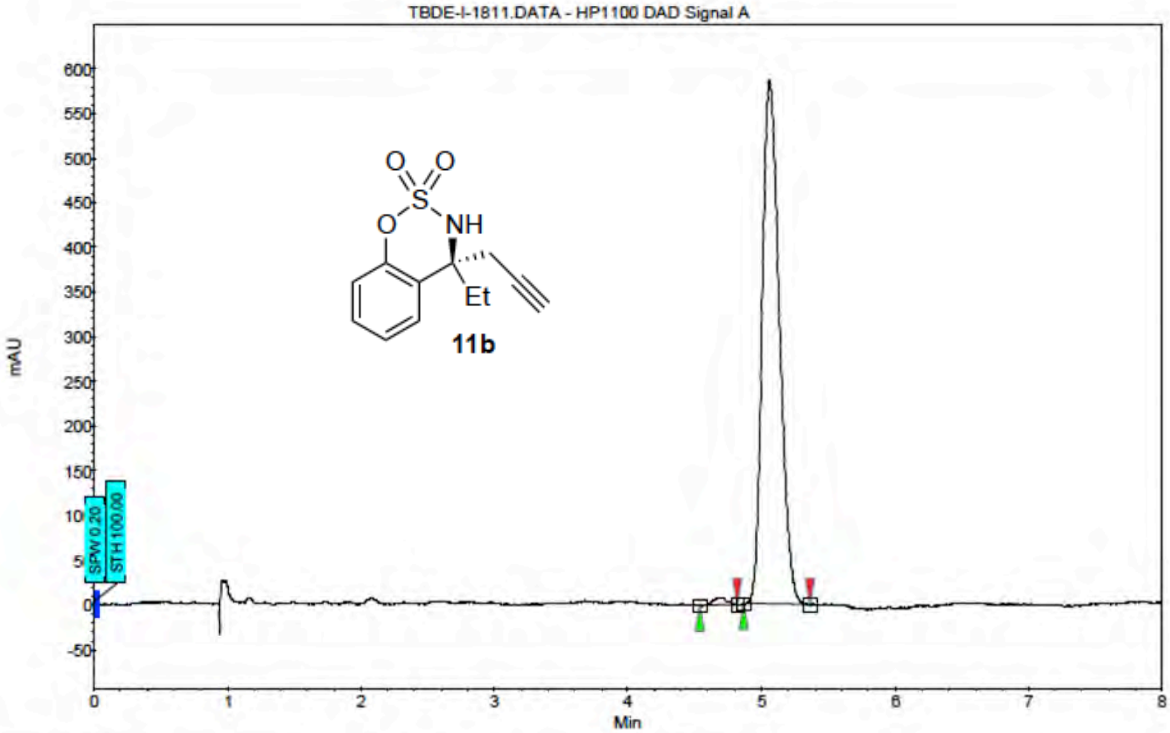
Index	Name	Start	Time	End	RT Offset	Quantity	Height	Area	Area
		[Min]	[Min]	[Min]	[Min]	[% Area]	[µV]	[µV.Min]	[%]
1	UNKNOWN	5.17	5.40	5.66	0.00	50.50	390.2	65.4	50.500
2	UNKNOWN	5.81	6.01	6.29	0.00	49.50	351.5	64.1	49.500
Total						100.00	741.7	129.4	100.000



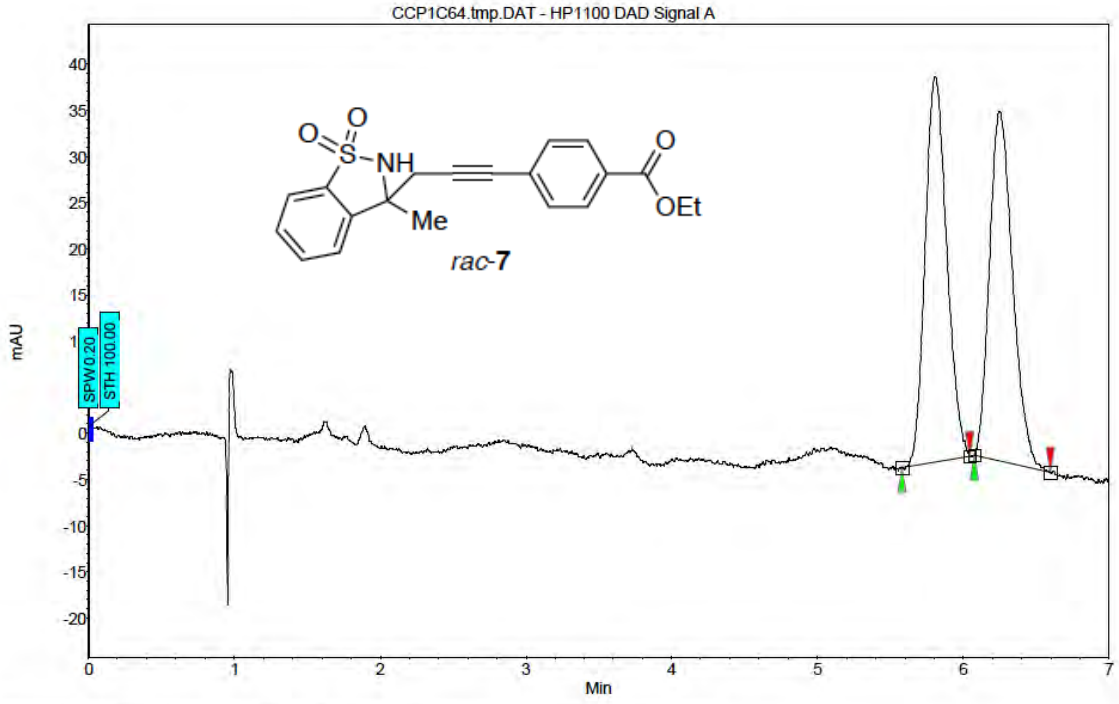
Index	Name	Start	Time	End	RT Offset	Quantity	Height	Area	Area
		[Min]	[Min]	[Min]	[Min]	[% Area]	[µV]	[µV.Min]	[%]
1	UNKNOWN	5.23	5.38	5.52	0.00	0.65	5.1	0.6	0.650
2	UNKNOWN	5.72	5.94	6.25	0.00	99.35	558.9	98.9	99.350
Total						100.00	564.0	99.6	100.000



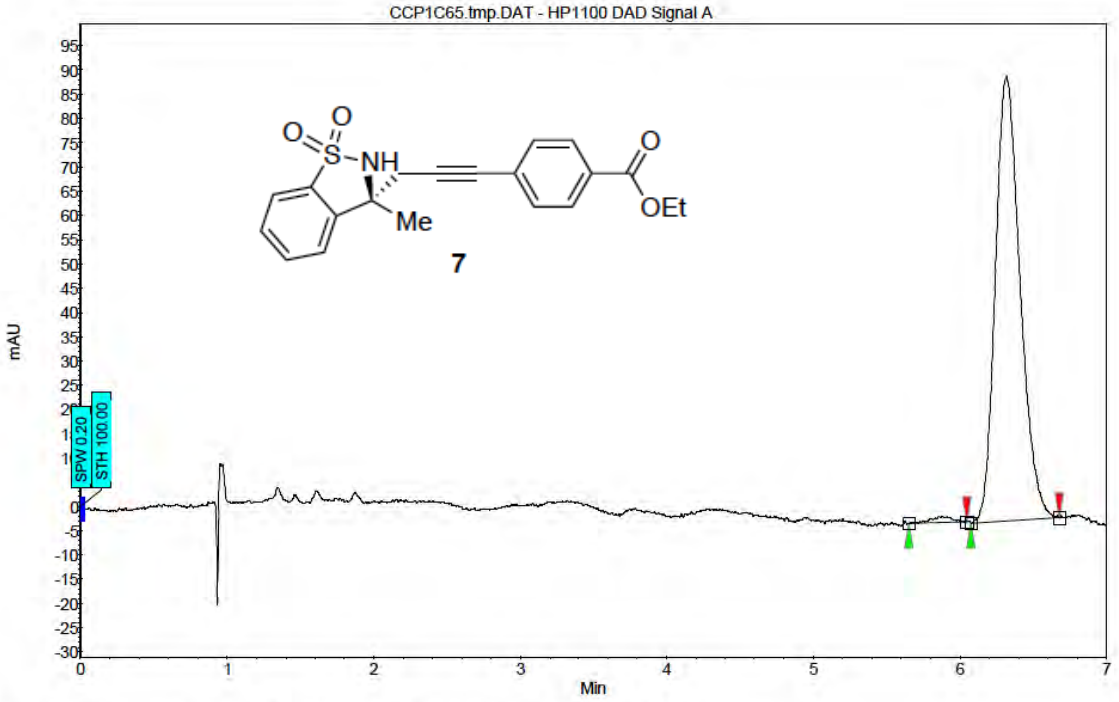
Index	Name	Start	Time	End	RT Offset	Quantity	Height	Area	Area
		[Min]	[Min]	[Min]	[Min]	[% Area]	[µV]	[µV.Min]	[%]
1	UNKNOWN	4.53	4.69	4.90	0.00	44.36	80.8	11.1	44.362
2	UNKNOWN	4.97	5.11	5.36	0.00	55.64	78.7	13.9	55.638
Total						100.00	159.5	25.1	100.000



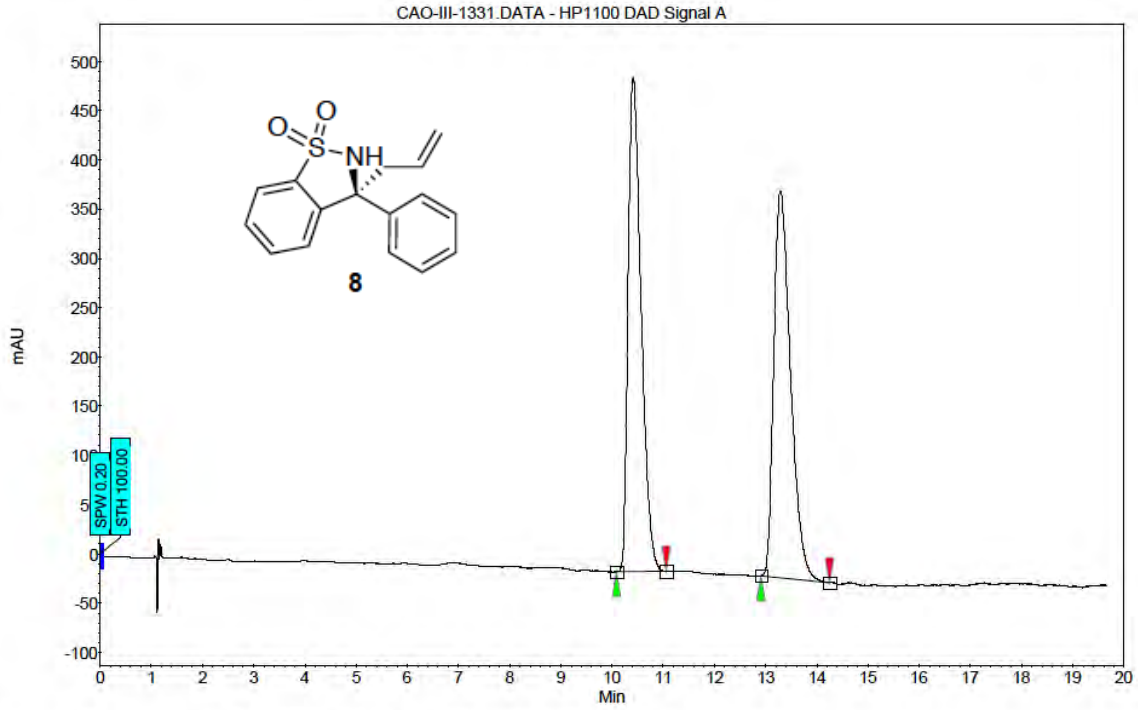
Index	Name	Start	Time	End	RT Offset	Quantity	Height	Area	Area
		[Min]	[Min]	[Min]	[Min]	[% Area]	[µV]	[µV.Min]	[%]
1	UNKNOWN	4.54	4.68	4.82	0.00	0.96	7.7	0.8	0.956
2	UNKNOWN	4.87	5.06	5.36	0.00	99.04	586.0	87.2	99.044
Total						100.00	593.8	88.1	100.000



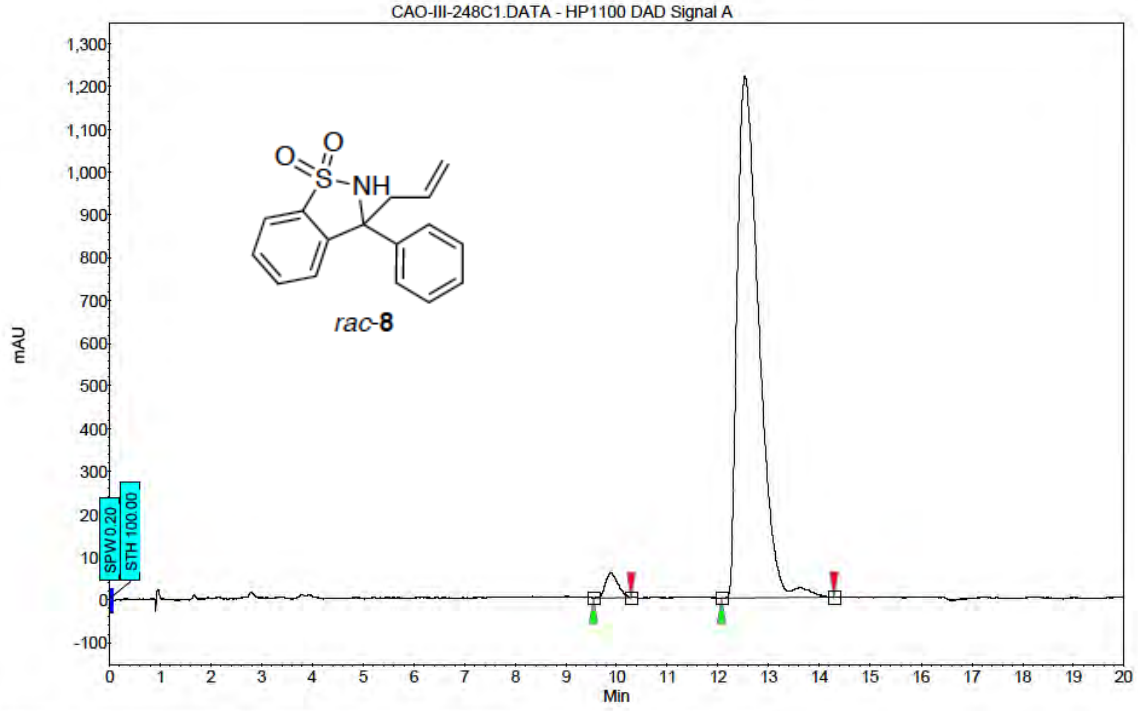
Index	Name	Start	Time	End	RT Offset	Quantity	Height	Area	Area
		[Min]	[Min]	[Min]	[Min]	[% Area]	[µV]	[µV.Min]	[%]
1	UNKNOWN	5.58	5.81	6.04	0.00	50.72	41.7	7.2	50.722
2	UNKNOWN	6.07	6.25	6.60	0.00	49.28	37.8	7.0	49.278
Total						100.00	79.5	14.1	100.000



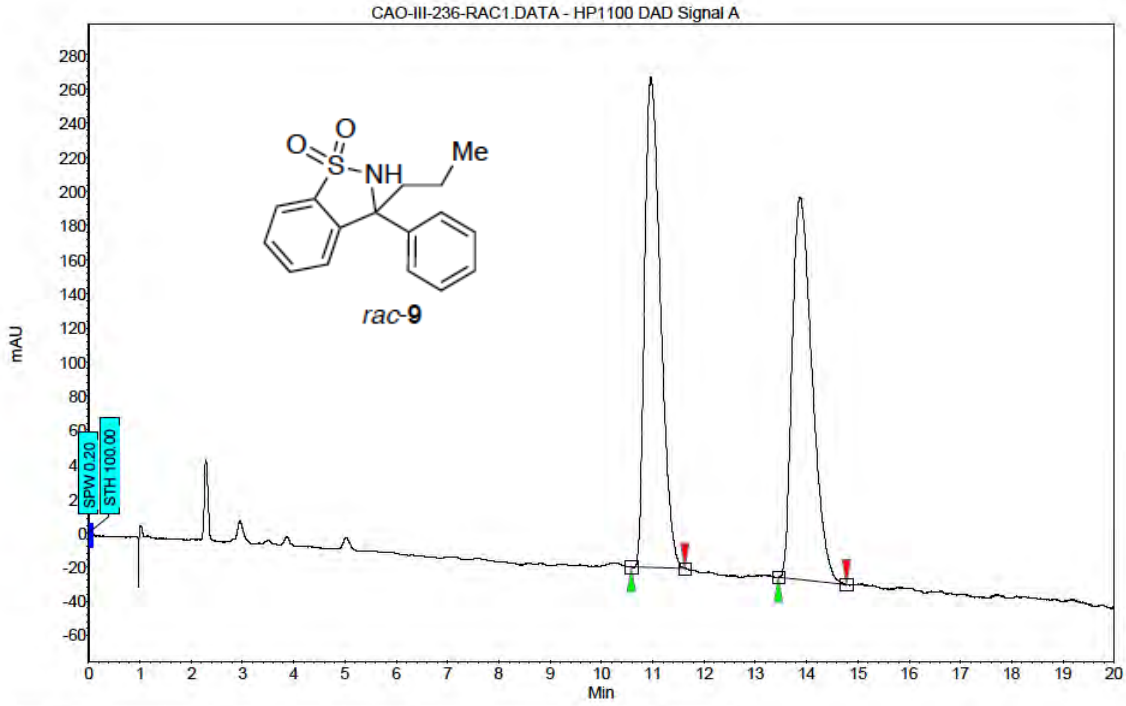
Index	Name	Start	Time	End	RT Offset	Quantity	Height	Area	Area
		[Min]	[Min]	[Min]	[Min]	[% Area]	[µV]	[µV.Min]	[%]
1	UNKNOWN	5.65	5.86	6.04	0.00	1.09	1.3	0.2	1.091
2	UNKNOWN	6.07	6.32	6.68	0.00	98.91	91.6	17.9	98.909
Total						100.00	92.9	18.1	100.000



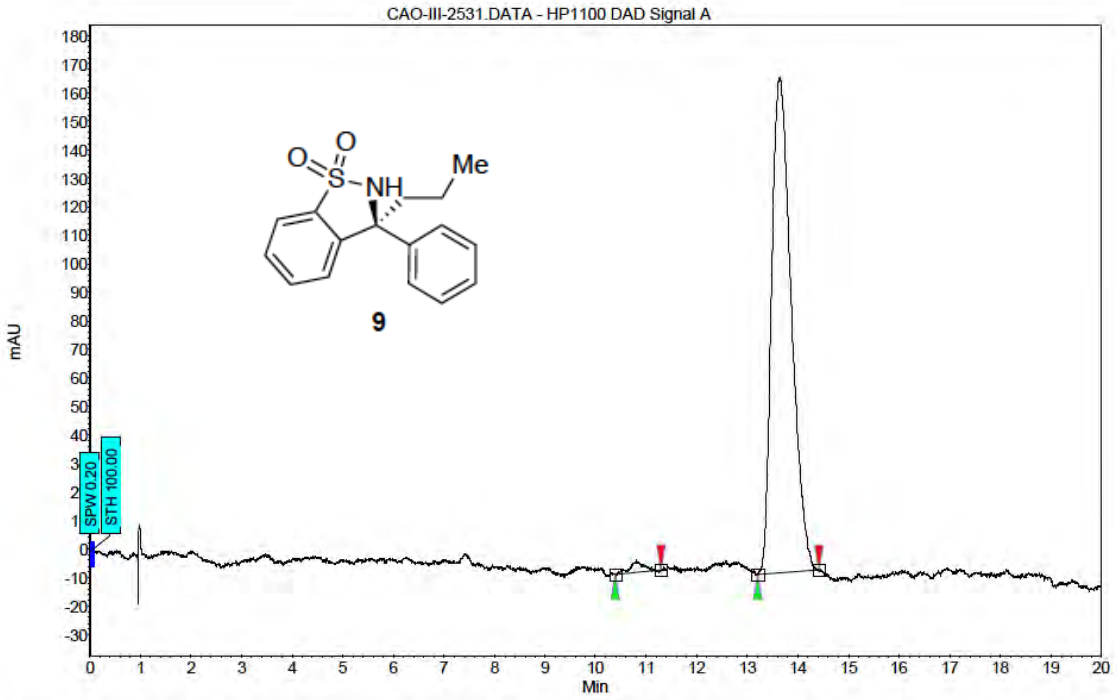
Index	Name	Start	Time	End	RT Offset	Quantity	Height	Area	Area
		[Min]	[Min]	[Min]	[Min]	[% Area]	[µV]	[µV.Min]	[%]
1	UNKNOWN	10.10	10.42	11.06	0.00	49.87	501.6	148.2	49.871
2	UNKNOWN	12.91	13.30	14.25	0.00	50.13	392.5	148.9	50.129
Total						100.00	894.1	297.1	100.000



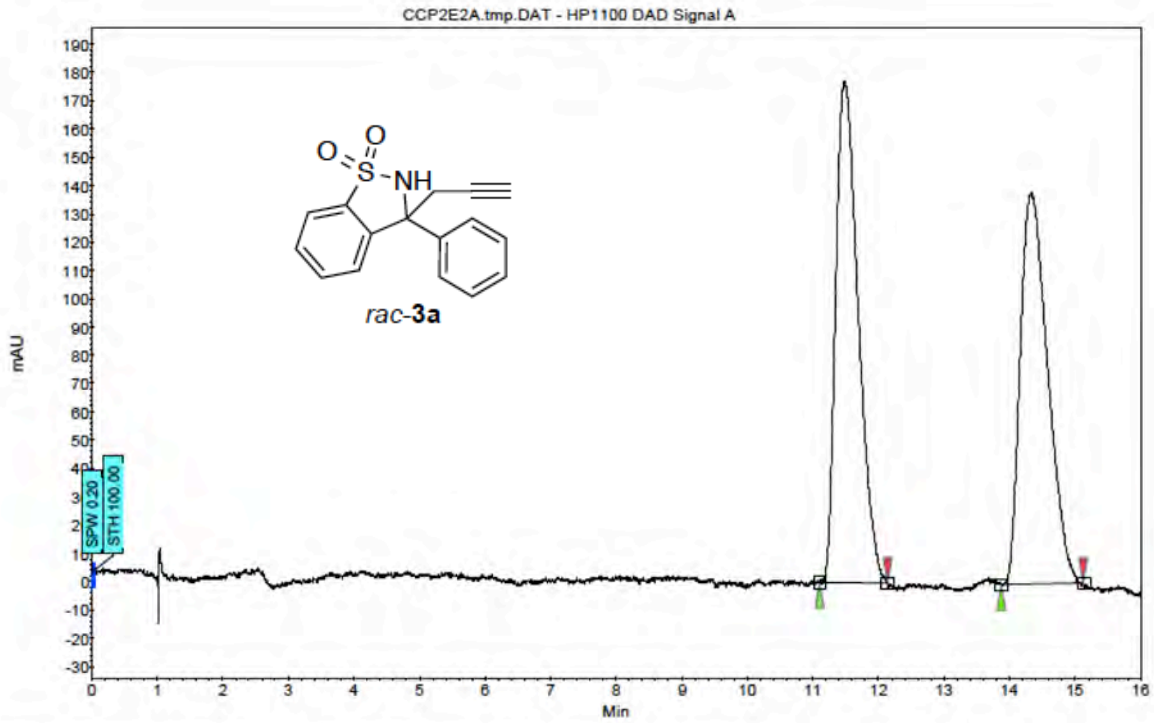
Index	Name	Start	Time	End	RT Offset	Quantity	Height	Area	Area
		[Min]	[Min]	[Min]	[Min]	[% Area]	[µV]	[µV.Min]	[%]
1	UNKNOWN	9.54	9.89	10.29	0.00	2.69	58.5	16.5	2.686
2	UNKNOWN	12.07	12.54	14.29	0.00	97.31	1217.8	598.1	97.314
Total						100.00	1276.3	614.6	100.000



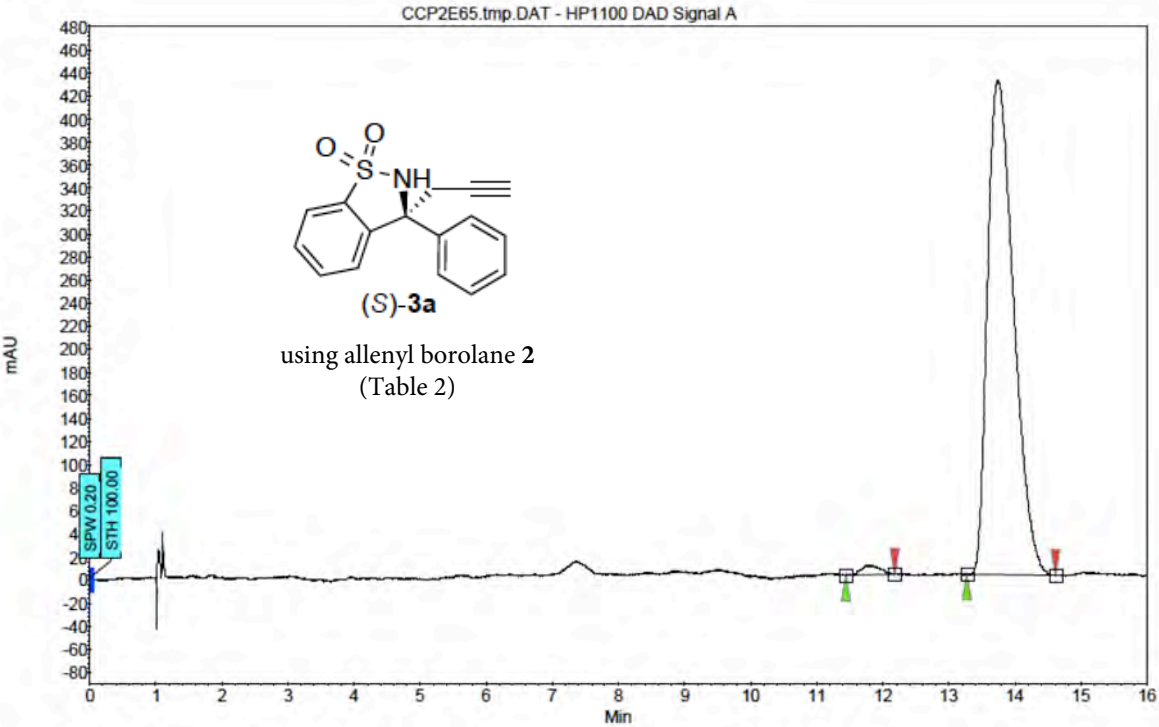
Index	Name	Start	Time	End	RT Offset	Quantity	Height	Area	Area
		[Min]	[Min]	[Min]	[Min]	[% Area]	[µV]	[µV.Min]	[%]
1	UNKNOWN	10.59	10.97	11.63	0.00	50.35	287.2	102.7	50.353
2	UNKNOWN	13.46	13.88	14.78	0.00	49.65	224.0	101.3	49.647
Total						100.00	511.2	204.0	100.000



Index	Name	Start	Time	End	RT Offset	Quantity	Height	Area	Area
		[Min]	[Min]	[Min]	[Min]	[% Area]	[µV]	[µV.Min]	[%]
1	UNKNOWN	10.39	10.81	11.29	0.00	1.46	4.4	1.2	1.464
2	UNKNOWN	13.20	13.63	14.41	0.00	98.54	173.5	77.9	98.536
Total						100.00	177.9	79.1	100.000

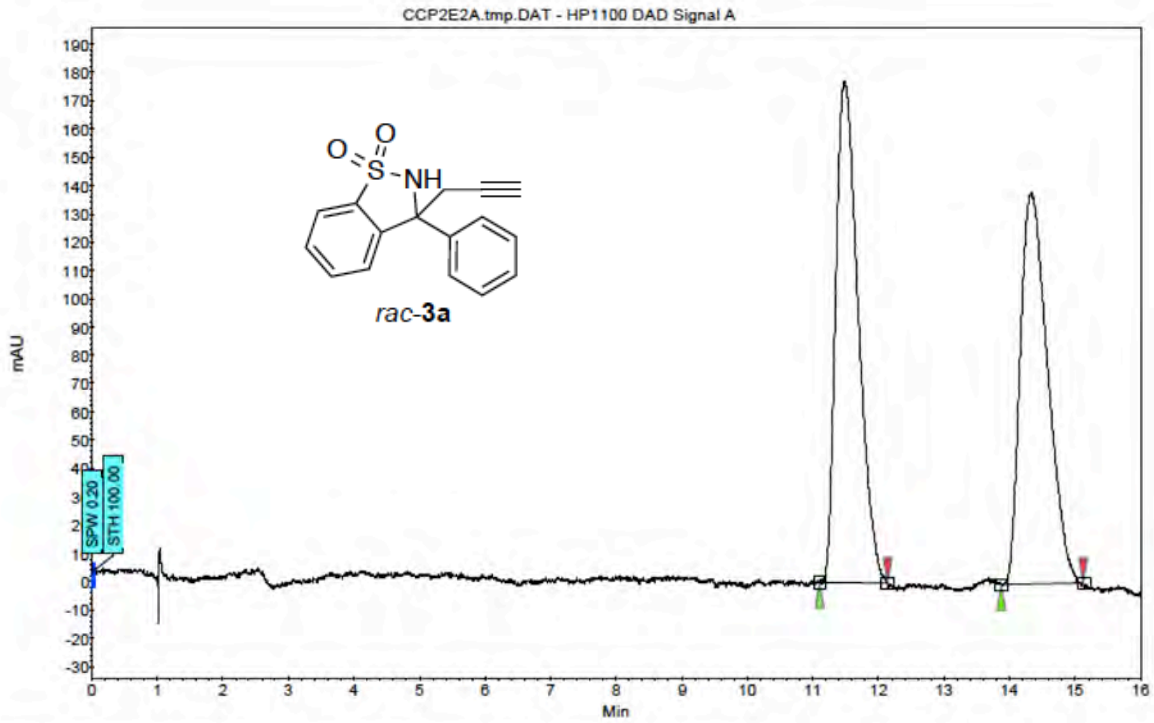


Index	Name	Start	Time	End	RT Offset	Quantity	Height	Area	Area
		[Min]	[Min]	[Min]	[Min]	[% Area]	[µV]	[µV.Min]	[%]
1	UNKNOWN	11.10	11.48	12.14	0.00	51.26	177.2	70.1	51.263
2	UNKNOWN	13.87	14.33	15.12	0.00	48.74	138.2	66.6	48.737
Total						100.00	315.3	136.7	100.000

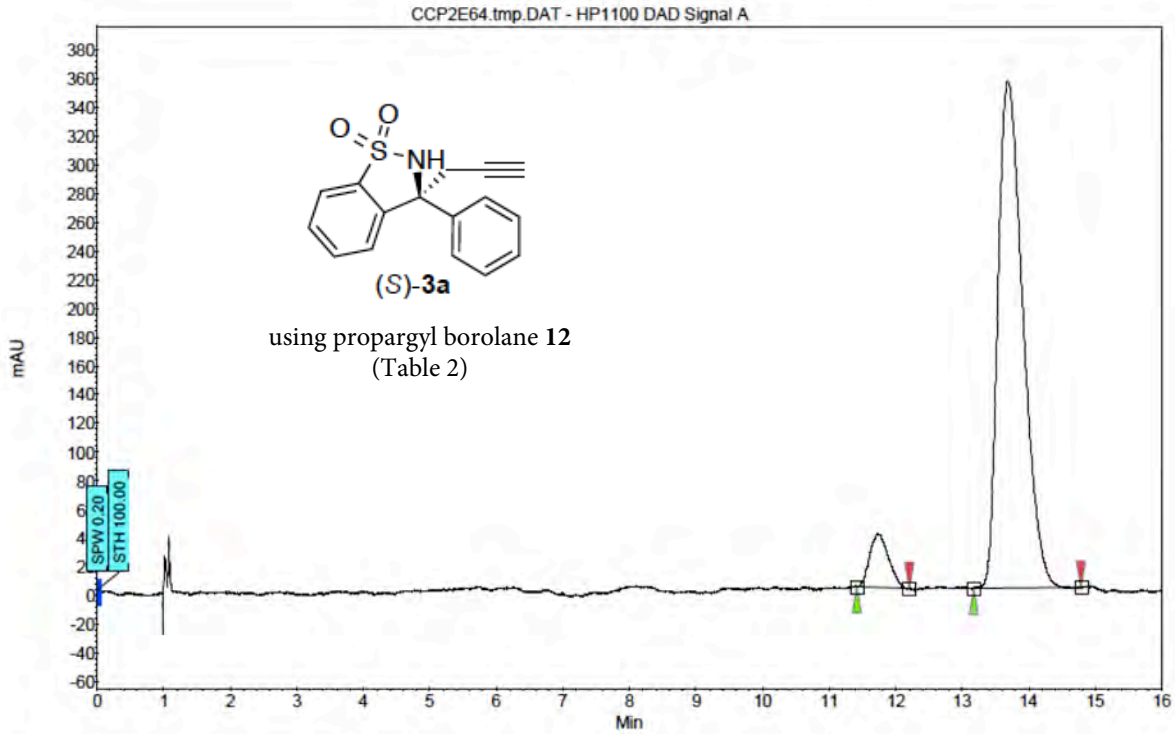


using allenyl borolane **2**
(Table 2)

Index	Name	Start	Time	End	RT Offset	Quantity	Height	Area	Area
		[Min]	[Min]	[Min]	[Min]	[% Area]	[µV]	[µV.Min]	[%]
1	UNKNOWN	11.44	11.78	12.18	0.00	1.38	8.5	2.7	1.379
2	UNKNOWN	13.27	13.73	14.61	0.00	98.62	428.9	196.4	98.621
Total						100.00	437.3	199.2	100.000



Index	Name	Start	Time	End	RT Offset	Quantity	Height	Area	Area
		[Min]	[Min]	[Min]	[Min]	[% Area]	[µV]	[µV.Min]	[%]
1	UNKNOWN	11.10	11.48	12.14	0.00	51.26	177.2	70.1	51.263
2	UNKNOWN	13.87	14.33	15.12	0.00	48.74	138.2	66.6	48.737
Total						100.00	315.3	136.7	100.000



using propargyl borolane **12**
(Table 2)

Index	Name	Start	Time	End	RT Offset	Quantity	Height	Area	Area
		[Min]	[Min]	[Min]	[Min]	[% Area]	[µV]	[µV.Min]	[%]
1	UNKNOWN	11.42	11.74	12.21	0.00	7.32	36.9	12.1	7.325
2	UNKNOWN	13.18	13.68	14.78	0.00	92.68	353.1	153.2	92.675
Total						100.00	390.0	165.3	100.000