Supplementary Material (ESI) for Chemical Science

Quinone Sulfinyl Imines as Versatile Intermediates in Alkaloid Natural Product Synthesis: Total Synthesis of 3-Demethoxyerythratidinone

Kangway V. Chuang, Raul Navarro and Sarah E. Reisman*

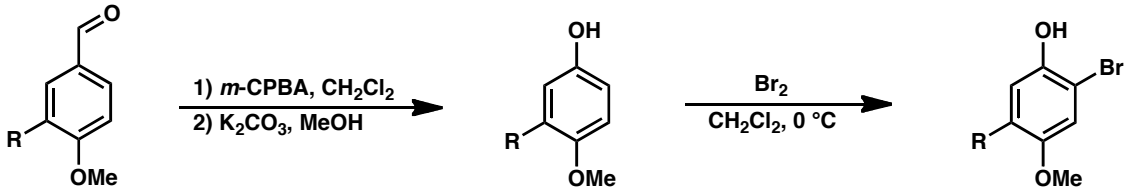
The Warren and Katharine Schlinger Laboratory for Chemistry and Chemical Engineering, Division of Chemistry and Chemical Engineering, California Institute of Technology, Pasadena, California 91125

reisman@caltech.edu

Supporting Information 1 (Experimental Procedures):

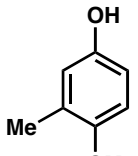
General. Unless otherwise stated, reactions were performed under a nitrogen atmosphere using freshly dried solvents. Tetrahydrofuran (THF), methylene chloride (CH_2Cl_2), diethyl ether (Et_2O), acetonitrile (MeCN), toluene and benzene were dried by passing through activated alumina columns. Dimethylformamide (DMF) was dried over activated molecular sieves, MeOH was distilled over magnesium oxide, dichloroethane (DCE) and triethyl amine (Et_3N) were distilled over calcium hydride. All other commercially obtained reagents were used as received unless specifically indicated. All reactions were monitored by thin-layer chromatography using EMD/Merck silica gel 60 F254 pre-coated plates (0.25 mm). Flash column chromatography was performed either as described by Still et al. (Still, W. C., Kahn, M.; Mitra, A. *J. Org. Chem*. **1978**, *43*, 2923-2925.) using silica gel (partical size 0.032-0.063) purchased from Silicycle or using pre-packaged RediSep®Rf columns on a CombiFlash Rf system (Teledyne ISCO Inc.). Microwave experiments were performed using a Biotage Initiator® microwave reactor. Diastereomeric ratios were determined using an Agilent 1190 or 1290 Series LC/MS (λ = 254 nm) using a ZORBAX Eclipse Plus C18 column (RRHD 1.8 μm, 2.1 x 50 mm, 11,072 plates). Optical rotations were measured on a Jasco P-2000 polarimeter using a 100 mm path-length cell at 589 nm. ^{1}H and ^{13}C NMR spectra were recorded on a Varian Mercury 300 (at 300 MHz and 75 MHz respectively) or a Varian Inova 500 (at 500 MHz and 126 MHz respectively), and are reported relative to internal chloroform (^{1}H, δ = 7.26, ^{13}C, δ = 77.0). Data for ^{1}H NMR spectra are reported as follows: chemical shift (δ ppm) (multiplicity, coupling constant (Hz), integration). Multiplicity and qualifier abbreviations are as follows: s = singlet, d = doublet, t = triplet, q = quartet, m = multiplet, br = broad, app = apparent. IR spectra were recorded on a Perkin Elmer Paragon 1000 spectrometer and are reported in frequency of absorption (cm^{-1}). High-resolution mass spectra were obtained from the Caltech Mass Spectral Facility.

Preparation of 2,4,5-substituted phenols:



S1 S2 S3

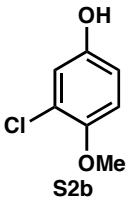
Steps 1 and 2: Baeyer-Villiger Oxidation / Saponification. Preparation of 4-methoxy-3-methylphenol (S2a).



S2a

A 50 mL flask was charged with 4-methoxy-3-methylbenzaldehyde (**S1a**) (500 mg, 3.33 mmol, 1 equiv) and CH_2Cl_2 (11 mL). The resulting solution was cooled to 0 °C in an ice-water bath and *m*-CPBA (1.40 g, 70-75%, 5.66 mmol, 1.7 equiv) was added in 3 portions. The resulting suspension was allowed to warm to room temperature, and was stirred for 2 hours at that temperature. The reaction was quenched with saturated aqueous $NaHCO_3$ (11 mL), and the aqueous layer was extracted with CH_2Cl_2 (3 x 20 mL). The combined organic layers were dried over Na_2SO_4 and concentrated to give a pale yellow oil. The crude formate ester was dissolved in MeOH (17 mL) and cooled to 0 °C. Solid K_2CO_3 (920 mg, 6.66 mmol) was added in one portion, and the resulting solution was stirred at 0 °C for 15 min. The reaction was quenched with aqueous HCl (9 mL of a 2N solution). The organic solvent was removed by rotary evaporation, and resulting aqueous layer was extracted with Et_2O (2 x 30 mL). The combined organic layers were dried over $MgSO_4$, concentrated, and purified by flash chromatography (10% EtOAc/Hexanes) to afford **S2a** (361 mg, 78% yield over 2 steps) as a white solid. ^{1}H NMR (500 MHz, $CDCl_3$) δ 6.70 (d, J = 8.8 Hz, 1H), 6.66 (d, J = 3.2 Hz, 1H), 6.62 (dd, J = 8.7, 3.1 Hz, 1H), 4.77 (s, 1H), 3.78 (s, 3H), 2.19 (s, 3H); ^{13}C NMR (126 MHz, $CDCl_3$) δ 152.0, 149.0, 128.1, 118.0, 112.5, 111.3, 56.0, 16.2; IR (NaCl/thin film): 3350, 2950, 2833, 1501, 1465, 1430, 1286, 1217, 1180, 1034 721 cm^{-1}; HRMS (EI+) calc'd for $C_8H_{10}O_2$ $[M+H]^+$ 138.0681, found 138.0685.

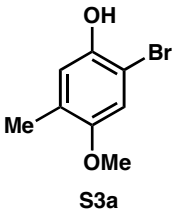
Preparation of 3-chloro-4-methoxyphenol (S2b). Prepared from 11.1 mmol of 3-chloro-4-methoxybenzaldehyde (**S1b**) using the above general procedure. The crude product was purified by flash chromatography (5→20% EtOAc/Hexanes) to give **S2b** (1.10 g, 62% yield) as a beige solid. ^{1}H NMR (500 MHz, $CDCl_3$) δ 6.91 (d, J = 2.9 Hz, 1H), 6.81 (d, J = 8.8 Hz, 1H), 6.70 (dd,



S2b

J = 8.8, 2.9 Hz, 1H), 4.94 (s, 1H), 3.84 (s, 3H); ^{13}C NMR (126 MHz, $CDCl_3$) δ 149.6, 149.4, 123.0, 117.6, 114.2, 113.5, 56.8; IR (NaCl/thin film): 3400, 2947, 2837, 1500, 1437, 1278, 1209, 1180, 1058, 907, 746 cm^{-1}; HRMS (EI+) calc'd for $C_7H_7O_2Cl$ $[M+H]^+$ 158.0135, found 158.0125.

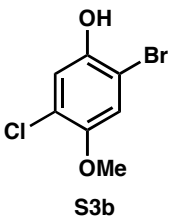
Step 3. Bromination. Preparation of 2-bromo-4-methoxy-5-methylphenol (S3a).



S3a

A 50 mL flask was charged with phenol **S2a** (300 mg, 2.17 mmol, 1 equiv) and CH_2Cl_2 (11 mL). The resulting solution was cooled to 0 °C in an ice-water bath, and bromine (0.117 mL, 2.28 mmol, 1.05 equiv) was added dropwise. (*Caution! A copious amount of HBr gas is generated as the reaction proceeds. A 16-gauge needle was pierced through the septa to allow the reaction to vent*). The reaction was allowed to stir at 0 °C for 30 min, then quenched with saturated aqueous $NaHCO_3$ (11 mL). The organic layer was washed with water (2 x 10 mL), and the combined aqueous layers were extracted with CH_2Cl_2 (20 mL). The combined organic layers were dried over Na_2SO_4, concentrated, and purified by flash chromatography (10% EtOAc/Hexanes) to give **S3a** (440 mg, 93% yield) as a beige solid. The spectral data obtained for **S3a** is consistent with that reported in the literature.[1]

Preparation of 2-bromo-5-chloro-4-methoxyphenol (S3b).

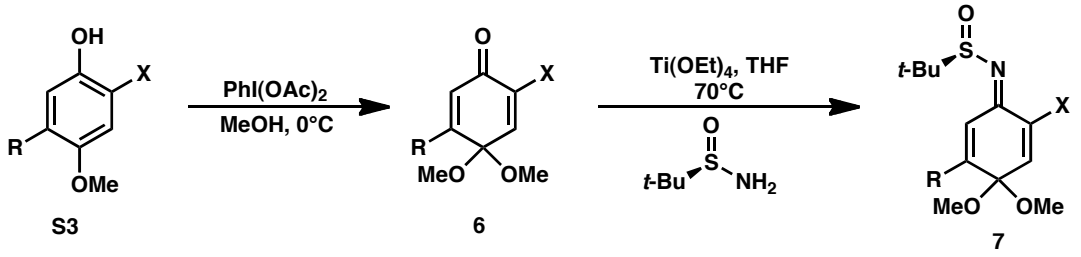


S3b

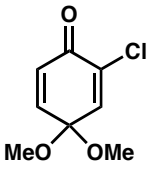
Prepared from 1.26 mmol of 3-chloro-4-methoxyphenol (**S2b**) using the general procedure. **S3b** (288 mg, 96% yield) was isolated as a pale beige solid. The crude product was used without further purification. ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.08 (s, 1H), 7.01 (s, 1H), 5.17 (s, 1H), 3.84 (s, 3H); ^{13}C NMR (126 MHz, $CDCl_3$) δ 149.5, 146.6, 123.0, 117.6, 115.4, 107.6, 56.9; IR (NaCl/thin film): 3248, 2969, 1504, 1442, 1400, 1205, 1182, 1073, 859, 784 cm^{-1}; HRMS (EI–) calc'd for $C_7H_7O_2Cl$ $[M-H]^-$ 234.9167, found 234.9198.

(1) Vyvyan, J.R.; Loitz, C.; Looper, R.E.; Mattingly, C.S.; Peterson, E.A.; Staben, S.T. *J. Org. Chem.* **2004**, *69,* 2461-2468.

General procedure for the preparation of quinone sulfinimine substrates:



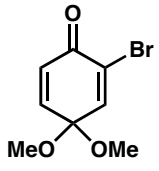
Step 1. Phenolic oxidation. Preparation of chloroquinone 6b.



6b

A 250 mL flask was charged with 2-chloro-4-methoxyphenol (2.00 g, 12.6 mmol, 1.0 equiv) and MeOH (70 mL). The resulting solution was cooled to 0 °C in an ice-water bath and a solution of iodobenzene diacetate (4.47 g, 13.9 mmol, 1.1 equiv) in MeOH (40 mL) was added dropwise via cannula. The reaction was allowed to stir at 0 °C for 10 min, then quenched with saturated aq. $NaHCO_3$ (30mL). The organic solvent was removed by rotary evaporation, and the resulting residue was diluted with Et_2O (60 mL). The aqueous layer was extracted with Et_2O (2 x 50 mL), and the combined organic layers were washed with brine (60 mL), dried over $MgSO_4$, concentrated, and purified by flash chromatography (6:1 Hexanes:EtOAc) to afford **6b** (2.33 g, 98% yield) as a pale yellow oil. ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.01 (d, J = 2.9 Hz, 1H), 6.85 (dd, J = 10.3, 2.9 Hz, 1H), 6.36 (d, J = 10.3 Hz, 1H), 3.38 (s, 6H); ^{13}C NMR (126 MHz, $CDCl_3$) δ 177.9, 143.7, 139.3, 134.0, 128.6, 94.2, 50.6; IR (NaCl/thin film): 2943, 2833, 1684, 1647, 1616, 1331, 1118, 1061, 1036, 1018, 962, 948, 824, 812 cm^{-1}; HRMS (EI+) calc'd for $C_8H_9O_3Cl$ $[M+H]^+$ 188.0240, found 188.0211.

Preparation of bromoquinone 6c.



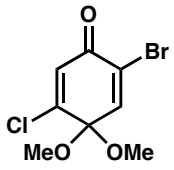
6c

Prepared from 19.7 mmol of 2-bromo-4-methoxyphenol[2] using the general procedure. The quinone product was purified by flash chromatography (10→20% EtOAc/Hexanes) to give **6c** (4.00 g, 87% yield) as a pale yellow solid. ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.25 (d, J = 3.2 Hz, 1H), 6.82 (dd, J = 10.3 Hz, 3.2 Hz, 1H), 6.33 (d, J = 10.3 Hz, 1H), 3.34 (s, 6H); ^{13}C NMR (126 MHz, $CDCl_3$) δ 177.6,

(2) 2-Bromo-4-methoxyphenol is commercially available from TCI America or readily prepared in one step from *p*-methoxyphenol.

143.9, 143.5, 128.0, 125.7, 94.2, 50.5; IR (NaCl/thin film): 3057, 2944, 2834, 1680, 1644, 1612, 1460, 1375, 1332, 1298, 1280, 1221, 1180, 1119, 1062, 1038, 1010, 964, 939, 823, 742 cm^{-1}; HRMS (EI+) calc'd for $C_8H_9O_3Br$ $[M–OMe]^+$ 200.9551, found 200.9551.[3]

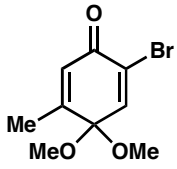
Preparation of dihaloquinone 6d.



6d

Prepared from 0.97 mmol of 2-bromo-5-chloro-4-methoxyphenol (**S3b**) using the general procedure. The quinone product was purified by flash chromatography (5→10% EtOAc/Hexanes) to give **6d** (229 mg, 88% yield) as a white solid. ^1H NMR (500 MHz, $CDCl_3$) δ 7.26 (s, 1H), 6.72 (s, 1H), 3.34 (s, 6H); ^{13}C NMR (126 MHz, $CDCl_3$) δ 176.1, 153.2, 144.2, 129.5, 126.5, 96.4, 51.7; IR (NaCl/thin film): 3435, 3051, 2940, 2841, 1673, 1612, 1458, 1328, 1105, 1071, 997, 755 cm^{-1}; HRMS (EI+) calc'd for $C_8H_8O_3ClBr$ $[M-OMe]^+$ 234.9161, found 234.9160.

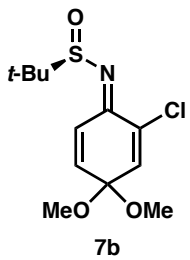
Preparation of bromoquinone 6e.



6e

Prepared from 1.53 mmol of 2-bromo-4-methoxy-5-methylphenol (**S3a**) using the general procedure. The quinone product was purified by flash chromatography (0→20% EtOAc/Hexanes) to give **6e** (350 mg, 93% yield) as a white solid. ^1H NMR (500 MHz, $CDCl_3$) δ 7.23 (s, 1H), 6.33 (q, J = 1.5 Hz, 1H), 3.26 (s, 6H), 1.94 (d, J = 1.5 Hz, 3H); ^{13}C NMR (126 MHz, $CDCl_3$) δ 177.7, 156.8, 144.7, 128.0, 127.2, 97.1, 51.2, 16.6; IR (NaCl/thin film): 3315, 3050, 2936, 2832, 1675, 1609, 1437, 1327, 1226, 1104, 1055, 923, 742 cm^{-1}; HRMS (EI+) calc'd for $C_9H_{11}O_3Br$ $[M-OMe]^+$ 214.9708, found 214.9706.

Step 2. Sulfinamide condensation. Preparation of quinone sulfinimine 7b.



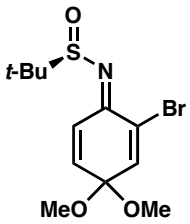
7b

A 50 mL oven-dried Schlenk tube was charged with (*R*)-*tert*-butanesulfinamide (1.78 g, 14.7 mmol, 1.1 equiv) followed by a solution of chloroquinone **6b** (2.64 g, 14.0 mmol, 1.0 equiv) and titanium (IV) ethoxide (6.4 mL, 30.5 mmol, 2.2 equiv) in THF (14 mL). The Schlenk tube was sealed and heated to 70°C in an oil-bath for 72 h while keeping the reaction from light. The reaction was

(3) Quinone monoketals **6b** and **6c** have been previously prepared by employing thallium (III) nitrate as the oxidant: McKillop, A.; Perry, D.H.; Edwards, M. *J. Org. Chem.* **1976**, *41*, 282-287.

allowed to cool to room temperature, diluted with EtOAc, and slowly poured into a stirring solution of brine (40 mL). The resulting suspension was filtered through a plug of celite and the organic layer was washed with brine (2 x 30 mL). The combined aqueous layers were extracted with EtOAc (40 mL), and the combined organic layers were dried over Na_2SO_4, concentrated, and purified by flash chromatography (20% EtOAc/Hexanes) to furnish **7b** (3.82 g, 93% yield) as an orange oil. ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.78 (d, *J* = 10.7 Hz, 1H), 6.68 (d, *J* = 2.4 Hz, 1H), 6.45 (dd, *J* = 10.5, 2.7 Hz, 1H), 3.34 (s, 3H), 3.33 (s, 3H), 1.33 (s, 9H); ^{13}C NMR (126 MHz, $CDCl_3$) δ 156.2, 137.0, 134.0, 133.8, 122.3, 94.2, 60.9, 50.3, 50.2, 23.2; IR (NaCl/thin film): 2961, 2945, 1569, 1457, 1168, 1113, 1082, 1039, 957, 790 cm^{-1}; HRMS (EI+) calc'd for $C_{12}H_{18}NO_3ClS$ $[M+H]^+$ 292.0769, found 292.0769; $[\alpha]_D^{25}$ –344.7 (*c* 0.62, CH_2Cl_2).

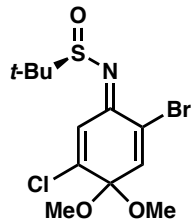
Preparation of quinone sulfinimine 7c.



7c

Prepared from 6.44 mmol of bromoquinone **6c** using the general procedure. The sulfinimine product was purified by flash chromatography (9→33% EtOAc/Hexanes) to yield **7c** (1.91 g, 85% yield) as an orange solid. ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.79 (d, *J* = 10.7 Hz, 1H), 6.94 (d, *J* = 2.4 Hz, 1H), 6.46 (dd, *J* = 10.5, 2.7 Hz, 1H), 3.35 (s, 3H), 3.34 (s, 3H) 1.33 (s, 9H); ^{13}C NMR (126 MHz, $CDCl_3$) δ 156.5, 138.4, 137.0, 125.6, 122.1, 94.5, 61.0, 50.33, 50.25, 23.2; IR (NaCl/thin film): 3198, 2958, 2929, 1669, 1597, 1290, 1057, 956, 886 cm^{-1}; HRMS (EI+) calc'd for $C_{12}H_{18}NO_3BrS$ $[M+H]^+$ 336.0264, found 336.0258; $[\alpha]_D^{25}$ –235.6 (*c* 0.80, CH_2Cl_2).

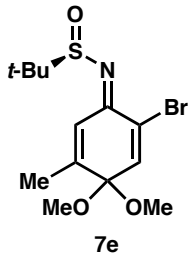
Preparation of quinone sulfinimine 7d.



7d

Prepared from 0.75 mmol of bromoquinone **6d** using the general procedure. The sulfinimine product was purified by flash chromatography (0→20% EtOAc/Hexanes) to yield **7d** (233 mg, 84% yield) as an orange oil. ^{1}H NMR (500 MHz, $CDCl_3$) δ 8.17 (s, 1H), 6.88 (s, 1H), 3.30 (s, 3H), 3.28 (s, 3H), 1.35 (s, 9H); ^{13}C NMR (126 MHz, $CDCl_3$) δ 155.0, 145.4, 138.5, 126.2, 123.3, 96.4, 61.5, 51.6, 51.5, 23.2; IR (NaCl/thin film): 3078, 2947, 2832, 1596, 1561, 1457, 1363, 1234, 1112, 1081, 1001, 977 cm^{-1}; HRMS (EI+) calc'd for $C_{12}H_{17}NO_3SClBr$ $[M+H]^+$ 369.9874 found 369.9873; $[\alpha]_D^{25}$ –346.2 (*c* 1.54, CH_2Cl_2).

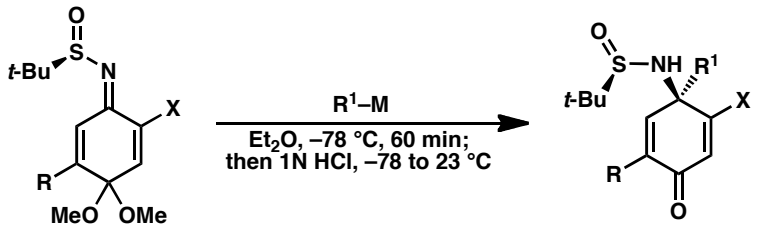
Preparation of quinone sulfinimine 7e.



7e

Prepared from 0.30 mmol of bromoquinone **6e** using the general procedure. The sulfinimine product was purified by flash chromatography (0→20% EtOAc/Hexanes) to yield **7e** (62 mg, 58% yield) as an orange oil. ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.67 (q, J = 1.5 Hz, 1H), 6.88 (s, 1H), 3.22 (s, 3H), 3.20 (s, 3H), 1.90 (d, J = 1.5 Hz, 3H), 1.34 (s, 9H); ^{13}C NMR (126 MHz, $CDCl_3$) δ 156.9, 149.5, 139.4, 127.0, 121.6, 97.2, 60.5, 51.1, 51.0, 23.1, 16.8; IR (NaCl/thin film): 2947, 2830, 1611, 1565, 1456, 1362, 1225, 1109, 1079, 969, 939 cm^{-1}; HRMS (EI+) calc'd for $C_{13}H_{20}NO_3SBr$ $[M+H]^+$ 350.0420, found 350.0423; $[\alpha]_D^{25}$ –261.7 (*c* 0.98, CH_2Cl_2).

General procedures for the diastereoselective addition of organolithium and organomagnesium reagents to quinone sulfinimine substrates:



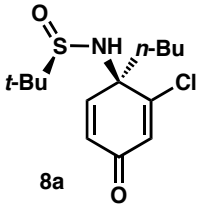
Method A.

An oven-dried 10 mL flask was charged with quinone sulfinimine **7** (0.30 mmol, 1 equiv) and Et_2O (0.6 mL). The resulting solution was cooled to –78 °C in a dry ice-acetone bath, and the organolithium reagent (0.33 mmol, 1.1 equiv) was added dropwise. After stirring at –78 °C for 1 h, the reaction was quenched at that temperature by the addition of aq. 1N HCl (0.6 mL). The reaction mixture was allowed to warm to room temperature and was vigorously stirred for 20 min. The reaction was diluted with EtOAc (30 mL) and washed with saturated aq. $NaHCO_3$ (15 mL). The aqueous layer was extracted with EtOAc (30 mL), and the combined organic layers were washed with brine (20 mL), dried over Na_2SO_4, and concentrated under reduced pressure to provide the crude product, which was analyzed by LC/MS and purified by flash chromatography.

Method B.

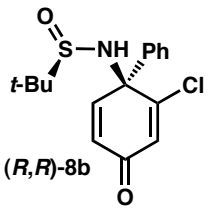
An oven-dried 10 mL flask was charged with aryl or vinyl bromide (0.48 mmol, 2.0 equiv) and Et_2O (0.4 mL). The resulting solution was cooled to –78 °C in a dry-ice acetone bath, and *t*-BuLi (0.99 mmol, 1.7 M in pentane, 4.1 equiv) was added dropwise. The resulting solution was warmed to 0 °C and stirred at that temperature for 45 min. The reaction mixture was re-cooled to –78 °C, and a solution of quinone sulfinimine **7** (0.24 mmol, 1 equiv) in Et_2O (0.5 mL) was added dropwise. The resulting suspension was stirred at –78 °C for 1 h, then quenched at that temperature by the addition of aq. 1N HCl (0.5 mL). Reaction work-up was conducted as described in Method A to obtain the crude product, which was analyzed by LC/MS and purified by flash chromatography.

Sulfinamide 8a. Method A. The reaction was run using quinone sulfinimine **7b** (90 mg, 0.30 mmol) and *n*-BuLi (0.22 mL, 1.5 M in hexanes, 0.33 mmol). The diastereoselectivity was determined by LC/MS: 97:3 d.r. (5→95% MeCN/H_2O, t = 0–7 min, 1 mL/min. Minor diastereomer: t_R = 5.3 min, major diastereomer: t_R = 5.6 min). The crude material was purified by flash chromatography (30→80% EtOAc/Hexanes) to provide **8a** (85 mg, 90% yield) as a pale yellow foam. ^{1}H NMR (500 MHz, $CDCl_3$) δ 6.99 (d, *J* = 10.3 Hz, 1H), 6.53 (d, *J* = 1.5 Hz, 1H), 6.38 (dd, *J* = 10.0, 1.7 Hz, 1H), 3.60 (s, 1H), 2.12 (ddd, *J* = 12.8, 10.5, 6.9 Hz, 1H), 1.67 (ddd, *J* = 12.7, 10.9, 5.5 Hz, 1H), 1.29 (dt, *J* = 14.7, 7.4 Hz, 2H), 1.22 (s, *J* = 5.0 Hz, 9H), 1.12 – 1.01 (m, 2H), 0.86 (t, *J* = 7.4 Hz, 3H); ^{13}C NMR (126 MHz, $CDCl_3$) δ 184.2, 155.9, 150.9, 131.0, 128.7, 62.1, 56.7, 38.8, 25.2, 22.4, 22.3, 13.7; IR (NaCl/thin film): 3198, 2959, 2929, 1660, 1599, 1057, 976, 885 cm^{-1}; HRMS (EI+) calc'd for $C_{14}H_{22}NO_2SCl$ $[M+H]^+$ 304.1133, found 304.1131; $[\alpha]_D^{25}$ –160.7 (*c* 0.50, CH_2Cl_2).



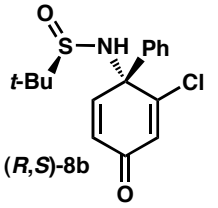
8a

Sulfinamide 8b. Method A. The reaction was run using quinone sulfinimine **7b** (80 mg, 0.27 mmol) and PhLi (0.18 mL, 1.7 M in di-*n*-butyl ether, 0.30 mmol). The diastereoselectivity was determined by LC/MS: 78:22 d.r. (5→95% MeCN/H_2O, t = 0–7 min, 1 mL/min. Minor diastereomer: t_R = 4.9 min, major diastereomer: t_R = 5.1 min). The crude material was purified by flash chromatography (20→80% EtOAc/Hexanes) to give (*R,R*)-**8b** (68 mg, 76% yield) as a pale yellow solid. Major diastereomer: ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.49 – 7.37 (m, 5H), 7.12 (d, *J* = 9.8 Hz, 1H), 6.57 (d, *J* = 1.5 Hz, 1H), 6.40 (dd, *J* = 10.0, 1.7 Hz, 1H), 4.15 (s, 1H), 1.33 (s, 9H); ^{13}C NMR (126 MHz, $CDCl_3$) δ 184.1, 155.9, 150.5, 137.2, 129.8, 129.4, 129.3, 126.34, 126.23, 64.5, 57.4, 22.6; IR (NaCl/thin film): 3186, 2960, 1658, 1596, 1300, 1062, 976 cm^{-1}; HRMS (EI+) calc'd for $C_{14}H_{22}NO_2ClS$ $[M+H]^+$ 324.0820, found 324.0827; $[\alpha]_D^{25}$ –102.4 (*c* 0.80, CH_2Cl_2). The minor diastereomer ((*R,S*)-**8b**) was obtained as a pale yellow oil: ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.48 – 7.32 (m, 5H), 6.80 (d, *J* = 10.3 Hz, 1H), 6.74 (d, *J* = 2.0 Hz, 1H), 6.27 (dd, *J* = 10.0, 1.7 Hz, 1H), 4.53 (s, 1H), 1.36 (s, 9H); ^{13}C NMR (126 MHz, $CDCl_3$) δ 184.1, 158.0, 148.4, 137.5, 129.8, 129.5, 129.2, 126.9, 125.6, 64.3, 57.4, 22.7; IR (NaCl/thin film): 3186, 2960, 1658, 1596, 1491, 1448, 1378, 1364, 1300, 1062, 976, 958 cm^{-1};



(*R,R*)-8b

major diastereomer

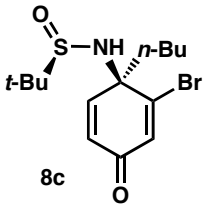


(*R,S*)-8b

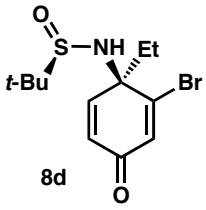
minor diastereomer

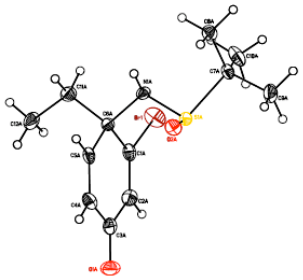
HRMS (EI+) calc'd for $C_{14}H_{22}NO_2ClS$ $[M+H]^+$ 324.0820, found 324.0823; $[\alpha]_D^{25}$ –365.9 (*c* 0.40, CH_2Cl_2).

Sulfinamide 8c. Method A. The reaction was run using quinone sulfinimine **7c** (80 mg, 0.24 mmol) and *n*-BuLi (0.18 mL, 1.5 M in hexanes, 0.26 mmol). The diastereoselectivity was determined by LC/MS: 98:2 d.r. (5→95% MeCN/H_2O, t = 0–7 min, 1 mL/min. Minor diastereomer: t_R = 5.4 min, major diastereomer: t_R = 5.7 min). The crude material was purified by flash chromatography (30→90% EtOAc/Hexanes) to furnish **8c** (73 mg, 88% yield) as a pale yellow foam. ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.10 (d, *J* = 9.8 Hz, 1H), 6.79 (d, *J* = 2.0 Hz, 1H), 6.41 (dd, *J* = 10.0, 1.7 Hz, 1H), 3.61 (s, 1H), 2.17 – 2.07 (m, 1H), 1.70 – 1.59 (m, 2H), 1.35 – 1.26 (m, 2H), 1.24 (s, 9H), 1.10 – 1.00 (m, 2H), 0.87 (t, *J* = 7.3 Hz, 3H); ^{13}C NMR (126 MHz, $CDCl_3$) δ 183.5, 151.1, 150.1, 135.3, 128.6, 62.4, 56.8, 39.8, 25.1, 22.6, 22.3, 13.7; IR (NaCl/thin film): 2946, 1567, 1457, 1179, 1110, 1082, 1039, 962, 764 cm^{-1}; HRMS (EI+) calc'd for $C_{14}H_{22}NO_2SBr$ $[M+H]^+$ 348.0627, found 348.0628; $[\alpha]_D^{25}$ –139.0 (*c* 0.50, CH_2Cl_2).



Sulfinamide 8d.

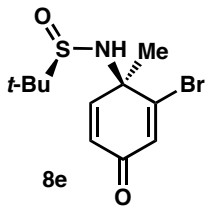




Method A. The reaction was run using quinone sulfinimine **7c** (80 mg, 0.24 mmol) and EtLi (0.52 mL, 0.5 M in 90:10 cyclohexane:benzene, 0.26 mmol). The diastereoselectivity was determined by LC/MS: 98:2 d.r. (5→95% MeCN/H_2O, t = 0–7 min, 1 mL/min. Minor diastereomer: t_R = 4.4 min, major diastereomer: t_R = 4.7 min). The crude material was purified by flash chromatography (50→75% EtOAc/Hexanes) to give **8d** (74 mg, 96% yield) as a pale yellow solid. The solid was recrystallized from CH_2Cl_2/pentane to give crystals suitable for single crystal X-ray diffraction. ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.07 (d, *J* = 10.3 Hz, 1H), 6.81 (d, *J* = 1.5 Hz, 1H), 6.43 (dd, *J* = 10.0, 1.7 Hz, 1H), 2.14 (dq, *J* = 13.1, 7.5 Hz, 1H), 1.72 (dq, *J* = 13.1, 7.4 Hz, 1H), 1.24 (s, 9H), 0.76 (t, *J* = 7.3 Hz, 3H); ^{13}C NMR (126 MHz, $CDCl_3$) δ 183.5,

150.8, 149.7, 135.6, 128.9, 63.0, 56.8, 33.3, 22.6, 7.5; melting point: 60 °C (decomposition); IR (NaCl/thin film): 3196, 2970, 1669, 1597, 1286, 1052, 954 cm^{-1}; HRMS (EI+) calc'd for $C_{12}H_{18}NO_2SBr$ $[M+H]^+$ 320.0314, found 320.0318. $[\alpha]_D^{25}$ –160.7 (*c* 1.20, CH_2Cl_2).

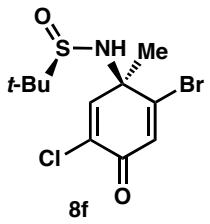
Sulfinamide 8e. Method A. The reaction was run using quinone sulfinimine **7c** (90 mg, 0.27 mmol) and MeLi (0.10 mL, 2.9 M in diethoxymethane, 0.29 mmol). The diastereoselectivity was determined by LC/MS: 98:2 d.r. (5→95% MeCN/H_2O, t = 0–7 min, 1 mL/min. Minor diastereomer: t_R = 4.0 min, major diastereomer: t_R = 4.3 min). The crude material was purified by flash chromatography (50→100% EtOAc/Hexanes) to provide **8e** (75 mg, 91% yield) as a pale yellow solid.



8e

^1H NMR (500 MHz, $CDCl_3$) δ 7.19 (d, *J* = 10.3 Hz, 1H), 6.74 (d, *J* = 1.5 Hz, 1H), 6.33 (dd, *J* = 10.0, 1.7 Hz, 1H), 3.63 (s, 1H), 1.61 (s, 3H), 1.25 (s, 9H); ^{13}C NMR (126 MHz, $CDCl_3$) δ 183.1, 151.9, 151.1, 133.9, 126.7, 58.7, 56.8, 28.1, 22.6; IR (NaCl/thin film): 3139, 2991, 1668, 1636, 1599, 1296, 1048, 960, 884 cm^{-1}; HRMS (EI+) calc'd for $C_{11}H_{16}NO_2SBr$ $[M+H]^+$ 306.0158, found 306.0158; $[\alpha]_D^{25}$ –190.3 (*c* 0.71, CH_2Cl_2).

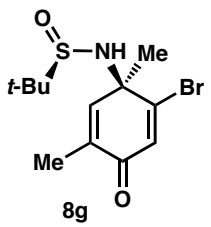
Sulfinamide 8f. Method A. The reaction was run using quinone sulfinimine **7d** (83 mg, 0.22 mmol) and MeLi (0.091 mL, 2.72 M in diethoxymethane, 0.25 mmol). The diastereoselectivity was determined by LC/MS: 97:3 d.r. (5→95% MeCN/H_2O, t = 0–10 min, 1 mL/min. Minor diastereomer: t_R = 3.1 min, major diastereomer: t_R = 3.4 min). The crude material was purified by flash chromatography (25→70% EtOAc/Hexanes) to provide **8f** (70 mg, 92% yield) as a pale yellow solid.



8f

^1H NMR (500 MHz, $CDCl_3$) δ 7.35 (s, 1H), 6.84 (s, 1H), 3.71 (s, 1H), 1.65 (s, 3H), 1.25 (s, 9H); ^{13}C NMR (126 MHz, $CDCl_3$) δ 176.4, 151.2, 147.8, 132.6, 131.0, 60.6, 57.0, 28.2, 22.5; IR (NaCl/thin film): 3126, 2981, 2930, 2868, 1674, 1609, 1365, 1334, 1040, 1005, 892, 873 cm^{-1}; HRMS (EI+) calc'd for $C_{11}H_{15}NO_2SClBr$ $[M+H]^+$ 339.9768, found 339.9765. $[\alpha]_D^{25}$ –138.1 (*c* 1.2, CH_2Cl_2).

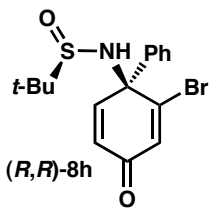
Sulfinamide 8g. Method A. The reaction was run using quinone sulfinimine **7e** (73 mg, 0.21 mmol) and MeLi (0.08 mL, 2.72 M in diethoxymethane, 0.23 mmol). The diastereoselectivity was determined by LC/MS: 98:2 d.r. (5→95% MeCN/H_2O, t = 0–10 min, 1 mL/min. Minor



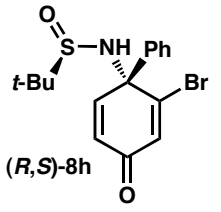
8g

diastereomer: t_R = 3.0 min, major diastereomer: t_R = 3.3 min). The crude material was purified by flash chromatography (30→80% EtOAc/Hexanes) to provide **8g** (61 mg, 91% yield) as a white solid. ^{1}H NMR (500 MHz, $CDCl_3$) δ 6.94 (q, *J* = 1.4 Hz, 1H), 6.72 (s, 1H), 3.59 (s, 1H), 1.93 (d, *J* = 1.5 Hz, 3H), 1.57 (s, 3H), 1.23 (s, 9H); ^{13}C NMR (126 MHz, $CDCl_3$) δ 183.9, 150.7, 147.4, 133.5, 133.5, 59.0, 56.7, 28.4, 22.6, 15.2; IR (NaCl/thin film): 3125, 2989, 2926, 2870, 1663 1649, 1608, 1460, 1365, 1113, 1040, 1015, 901, 892 cm^{-1}; HRMS (EI+) calc'd for $C_{12}H_{18}NO_2SBr$ $[M+H]^+$ 320.0314, found 320.0316. $[\alpha]_D^{25}$ –168.9 (*c* 1.05, CH_2Cl_2).

Sulfinamide 8h. Method A. The reaction was run using quinone sulfinimine **7c** (80 mg, 0.24 mmol) and PhLi (0.15 mL, 1.7 M in di-*n*-butyl ether, 0.26 mmol). The diastereoselectivity was determined by LC/MS: 80:20 d.r. (5→95% MeCN/H$_2$O, t = 0–7 min, 1 min/mL. Minor diastereomer: t_R = 5.0 min, major diastereomer: t_R = 5.2 min). The crude material was purified by flash chromatography (20→80% EtOAc/Hexanes) to yield (*R,R*)-**8h** (65 mg, 74% yield) as a yellow solid. Major diastereomer: ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.48 – 7.35 (m, 5H), 7.20 (d, *J* = 9.8 Hz, 1H), 6.83 (d, *J* = 1.5 Hz, 1H), 6.40 (dd, *J* = 10.0, 1.7 Hz, 1H), 4.20 (s, 1H), 1.34 (s, 9H); ^{13}C NMR (126 MHz, $CDCl_3$) δ 183.4, 150.8, 149.9, 137.6, 133.8, 129.31, 129.25, 126.2, 126.0, 64.8, 57.4, 22.8; IR (NaCl/thin film): 3184, 2960, 1669, 1292, 1059, 954 cm^{-1}; HRMS (EI+) calc'd for $C_{16}H_{18}NO_2SBr$ $[M+H]^+$ 368.0314, found 368.0317. $[\alpha]_D^{25}$ –102.8 (*c* 0.60, CH_2Cl_2). The minor diastereomer ((*R,S*)-**8h**) was obtained as a pale yellow oil. ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.46 – 7.33 (m, 5H), 6.97 (d, *J* = 2.0 Hz, 1H), 6.89 (d, *J* = 9.8 Hz, 1H), 6.29 (dd, *J* = 10.0, 1.7 Hz, 1H), 4.56 (s, 1H), 1.37 (s, 9H); ^{13}C NMR (126 MHz, $CDCl_3$) δ 183.3, 152.5, 148.6, 137.9, 133.6, 129.4, 129.2, 126.8, 125.6, 64.8, 57.4, 22.7; IR (NaCl/thin film): 3287, 2959, 1669, 1295, 1078, 952 cm^{-1}; HRMS (EI+) calc'd for $C_{16}H_{18}NO_2SBr$ $[M+H]^+$ 368.0314, found 368.0313; $[\alpha]_D^{25}$ –281.0 (*c* 0.45, CH_2Cl_2).

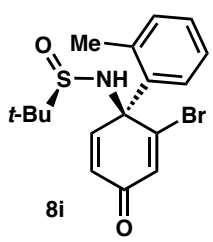


major diastereomer



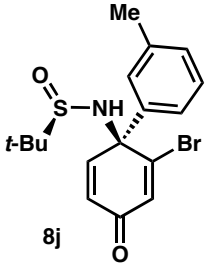
minor diastereomer

Sulfinamide 8i. Method B. The reaction was run using quinone sulfinimine **7c** (81 mg, 0.24 mmol), and *o*-bromotoluene (57 μL, 0.48 mmol). The diastereoselectivity was determined by LC/MS: 97:3 d.r. (5% MeCN/H_2O, t = 0–0.5 min; 5→45% MeCN/H_2O, t = 0.5–10.5 min, 1

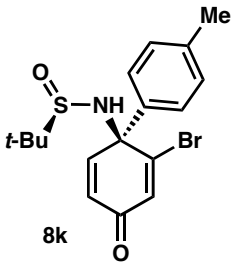


mL/min. Minor diastereomer: t_R = 8.3 min, major diastereomer: t_R = 8.7 min). The crude material was purified by flash chromatography (25→50% EtOAc/Hexanes) to furnish **8i** (79 mg, 86% yield) as a pale yellow foam. ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.72 (dd, *J* = 7.8, 1.5 Hz, 1H), 7.34 (dt, *J* = 7.8, 1.5 Hz, 1H), 7.31 (dt, *J* = 7.3, 1.5 Hz, 1H), 7.16 (dd, *J* = 7.3, 1.5 Hz, 1H), 7.10 (d, *J* = 10.3 Hz, 1H), 6.90 (d, *J* = 1.7 Hz, 1H), 6.48 (dd, *J* = 9.9, 1.7 Hz, 1H), 4.23 (s, 1H), 2.26 (s, 3H), 1.32 (s, 9H); ^{13}C NMR (126 MHz, $CDCl_3$) δ 183.4, 149.3, 148.5, 136.0, 135.7, 134.2, 133.2, 129.3, 127.4, 127.2, 126.9, 64.8, 57.3, 22.7, 20.7; IR (NaCl/thin film): 3188, 2960, 1666, 1641, 1594, 1291, 1082, 1068, 951 cm^{-1}; HRMS (EI+) calc'd for $C_{17}H_{20}NO_2SBr$ $[M+H]^+$ 382.0471, found 382.0469. $[\alpha]_D^{25}$ –107.7 (*c* 0.60, CH_2Cl_2).

Sulfinamide 8j. Method B. The reaction was run using quinone sulfinimine **7c** (81 mg, 0.24 mmol) and *m*-bromotoluene (58 μL, 0.48 mmol). The diastereoselectivity was determined by LC/MS: 91:9 d.r. (5→40% MeCN/H_2O, t = 0–0.5 min; 40→50% MeCN/H_2O, t = 0.5–8.5 min, 1 mL/min. Minor diastereomer: t_R = 5.2 min, major diastereomer: t_R = 5.5 min). The crude material was purified by flash chromatography (25→50% EtOAc/Hexanes) to yield **8j** (73 mg, 79% yield) as a pale yellow foam. ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.33 – 7.29 (m, 1H), 7.29 – 7.26 (m, 1H), 7.19 (d, *J* = 10.3 Hz, 1H), 7.19 (m, 1H), 6.83 (d, *J* = 2.0 Hz, 1H), 6.39 (dd, *J* = 9.8, 1.5 Hz, 1H), 4.19 (s, 1H), 2.37 (s, 3H), 1.34 (s, 9H); ^{13}C NMR (126 MHz, $CDCl_3$) δ 183.5, 150.9, 150.1, 139.3, 137.5, 133.8, 130.1, 129.2, 126.7, 125.9, 123.2, 64.8, 57.4, 22.8, 21.6; IR (NaCl/thin film): 3188, 2959, 1669, 1595, 1292, 1079, 1062, 954 cm^{-1}; HRMS (EI+) calc'd for $C_{17}H_{20}NO_2SBr$ $[M+H]^+$ 382.0471, found 382.0468. $[\alpha]_D^{25}$ –99.1 (*c* 0.60, CH_2Cl_2).

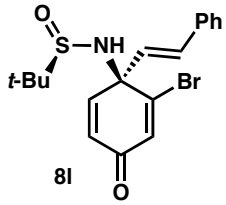


Sulfinamide 8k. Method B. The reaction was run using quinone sulfinimide **7c** (80 mg, 0.24 mmol) and *p*-bromotoluene (81 mg, 0.48 mmol). The diastereoselectivity was determined by LC/MS: 91:9 d.r. (5→30% MeCN/H_2O, t = 0–0.5 min; 30→50% MeCN/H_2O, t = 0.5–10.5 min, 1 mL/min. Minor diastereomer: t_R = 8.2 min, major diastereomer: t_R = 8.7 min). The crude material was purified by flash chromatography (25→60% EtOAc/Hexanes) to provide **8k** (72 mg, 78% yield) as a pale yellow foam. ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.32 (app d, *J* = 8.3 Hz, 2H),

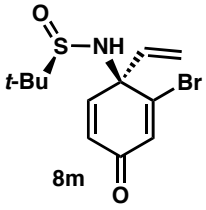


7.22 (d, *J* = 8.3 Hz, 2H), 7.19 (d, *J* = 9.8 Hz, 1H), 6.81 (d, *J* = 1.5 Hz, 1H), 6.38 (dd, *J* = 10.0, 1.7 Hz, 1H), 4.17 (s, 1H), 2.36 (s, 3H), 1.34 (s, 9H); ^{13}C NMR (126 MHz, $CDCl_3$) δ 183.5, 160.0, 150.2, 139.4, 134.6, 133.7, 130.0, 126.1, 125.8, 64.7, 57.4, 22.8, 21.1; IR (NaCl/thin film): 3186, 2959, 2920, 1668, 1292, 1079, 1062, 955 cm^{-1}; HRMS (EI+) calc'd for $C_{17}H_{20}NO_2SBr$ $[M+H]^+$ 382.0471, found 382.0470. $[\alpha]_D^{25}$ –84.7 (*c* 0.50, CH_2Cl_2).

Sulfinamide 8l. Method B. The reaction was run in THF using quinone sulfinimide **7c** (80 mg, 0.24 mmol) using β-bromostyrene[4] (87 mg, 0.48 mmol). The diastereoselectivity was determined by LC/MS: 98:2 d.r. (5→50% MeCN/H_2O, t = 0–10 min; 50→100% MeCN/H_2O, t = 10–13 min, 1 mL/min. Minor diastereomer: t_R = 11.6 min, major diastereomer: t_R = 11.8 min). The crude material was purified by flash chromatography (25→90% EtOAc/Hexanes) to furnish **8l** (64 mg, 68% yield) as a pale yellow solid.[5] ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.42 – 7.37 (m, 2H), 7.37 – 7.29 (m, 3H), 7.25 (d, *J* = 9.8 Hz, 1H), 6.79 (d, *J* = 1.5 Hz, 1H), 6.69 (d, *J* = 16.1 Hz, 1H), 6.44 (dd, *J* = 10.0, 1.7 Hz, 1H), 6.19 (d, *J* = 16.1 Hz, 1H), 3.92 (s, 1H), 1.30 (s, 9H); ^{13}C NMR (126 MHz, $CDCl_3$) δ 183.1, 149.7, 149.0, 135.0, 134.0, 133.8, 129.1, 128.8, 127.02, 127.00, 126.9, 62.4, 57.2, 22.7, 22.4; IR (NaCl/thin film): 3189, 2960, 1669, 1596, 1293, 1060, 955, 735 cm^{-1}; HRMS (EI+) calc'd for $C_{18}H_{20}NO_2SBr$ $[M+H]^+$ 394.0471, found 394.0476. $[\alpha]_D^{25}$ –115.0 (*c* 0.65, CH_2Cl_2).



Sulfinamide 8m. Method A. The reaction was run in THF using quinone sulfinimine **7c** (80 mg, 0.24 mmol) and vinyllithium[6] (0.48 mmol). The diastereoselectivity was determined by LC/MS: 98:2 d.r. (5→50% MeCN/H_2O, t = 0–10 min, 1 mL/min. Minor diastereomer: t_R = 7.9 min, major diastereomer: t_R = 8.3 min). The crude material was purified by flash chromatography (40→90% EtOAc/Hexanes) to yield **8m** (55 mg, 72% yield) as a yellow foam. ^{1}H NMR (500 MHz, $CDCl_3$)



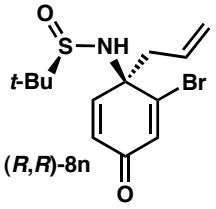
(4) β-bromostyrene was isolated as a 9:1 mixture of E:Z olefin isomers. See: Brown, H.C.; Subrahmanyam, C.; Hamaoka, T.; Ravindran, N.; Bowman, D.H.; Misumi, S.; Unni, M.K.; Somayaji, V.; Bhat, N.G. *J. Org. Chem.* **1989**, *54*, 6068.

(5) The product isolated contained <2% minor diastereomer and 3% *cis* addition product.

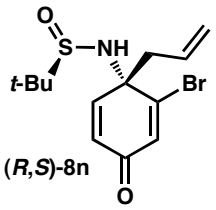
(6) Vinyllithium was prepared by treating a solution of tetravinyl tin (95 μL, 0.52 mmol) in Et_2O (0.3 mL) at 0 °C with *n*-BuLi (0.33 mL, 1.5 M in hexanes, 0.48 mmol). The reaction was allowed to stir 20 min at that temperature, then warmed to room temperature prior to use. See: Liu, H.; Tomooka, C.S.; Xu, S.L. Yerxa, B.R.; Sullivan, R.W.; Xiong, Y.; Moore, H.W. *Org. Synth.* **2004**, *76*, 178.

δ 7.13 (d, *J* = 9.8 Hz, 1H), 6.76 (d, *J* = 1.0 Hz, 1H), 6.39 (dd, *J* = 10.0, 1.2 Hz, 1H), 5.87 (dd, *J* = 17.3, 10.5 Hz, 1H), 5.45 (d, *J* = 10.7 Hz, 1H), 5.45 (d, *J* = 17.1 Hz, 1H), 3.82 (s, 1H), 1.27 (s, 9H); ^{13}C NMR (126 MHz, $CDCl_3$) δ 183.0, 149.6, 148.5, 136.4, 134.1, 127.1, 119.3, 62.6, 57.2, 22.6; IR (NaCl/thin film): 3186, 2959, 1669, 1594, 1294, 1060, 954 cm^{-1}; HRMS (EI+) calc'd for $C_{12}H_{16}NO_2SBr$ $[M+H]^+$ 318.0158, found 318.0161. $[\alpha]_D^{25}$ –175.9 (*c* 0.85, CH_2Cl_2).

Sulfinamide 8n. Method A. The reaction was run in THF using quinone sulfinimide **7c** (80 mg, 0.24 mmol) and allylmagnesium chloride (0.13 mL, 2.0 M in THF, 0.26 mmol). The diastereoselectivity was determined by LC/MS: 87:13 d.r. (5→40% MeCN/H_2O, t = 0–0.5 min; 40→60% MeCN/H_2O, t = 0.5–5.5 min, 1 mL/min. Minor diastereomer: t_R = 3.0 min, major diastereomer: t_R = 3.4 min). The crude material was purified by flash chromatography (30→80% EtOAc/Hexanes) to give (*R,R*)-**8n** (49 mg, 82% yield) as a pale yellow solid.



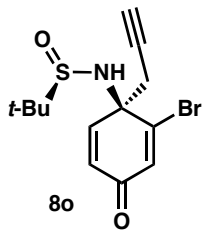
major diastereomer



minor diastereomer

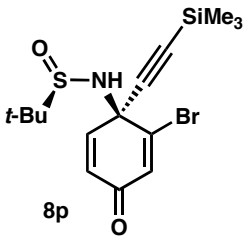
Major diastereomer: ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.14 (d, *J* = 10.0 Hz, 1H), 6.79 (d, *J* = 1.8 Hz, 1H), 6.39 (dd, *J* = 10.0, 1.8 Hz, 1H), 5.52 (dddd, *J* = 17.1, 10.1, 7.7, 7.1 Hz, 1H), 5.26 – 5.22 (m, 1H), 5.22 – 5.20 (m, 1H), 3.77 (s, *J* = 10.4 Hz, 1H), 2.75 (ddt, *J* = 13.2, 7.1, 1.0 Hz, 1H), 2.57 (ddt, *J* = 13.2, 7.8, 1.0 Hz, 1H), 1.25 (s, 9H); ^{13}C NMR (126 MHz, $CDCl_3$) δ 183.2, 150.6, 149.7, 135.4, 128.8, 128.2, 122.1, 61.6, 57.1, 44.6, 22.6. IR (NaCl/thin film): 3196, 2959, 1669, 1597, 1056, 957 cm^{-1}; HRMS (EI+) calc'd for $C_{13}H_{18}NO_2SBr$ $[M+H]^+$ 332.0314, found 332.0316. $[\alpha]_D^{25}$ –129.0 (*c* 0.6, CH_2Cl_2). The minor diastereomer ((*R,S*)-**8n**) was obtained as a pale yellow solid. ^{1}H NMR (500 MHz, $CDCl_3$) δ 6.89 (d, *J* = 10.3 Hz, 1H), 6.76 (d, *J* = 1.5 Hz, 1H), 6.39 (dd, *J* = 10.0, 1.7 Hz, 1H), 5.47 (ddt, *J* = 17.1, 10.3, 7.3 Hz, 1H), 5.20 –5.13 (m, 2H), 3.95 (s, 1H), 2.70 – 2.59 (m, 2H), 1.26 (s, 9H); ^{13}C NMR (126 MHz, $CDCl_3$) δ 183.1, 152.0, 148.3, 133.8, 129.7, 128.7, 121.2, 61.8, 56.9, 43.9, 22.5; IR (NaCl/thin film): 3195, 2956, 1670, 1595, 1070, 955, 883 cm^{-1}; HRMS (EI+) calc'd for $C_{13}H_{18}NO_2S^{81}Br$ $[M+H]^+$ 333.0221, found 333.0209. $[\alpha]_D^{25}$ –95.7 (*c* 0.80, CH_2Cl_2).

Sulfinamide 8o. Method A. The reaction was run using quinone sulfinimine **7c** (80 mg, 0.24 mmol) and propargylmagnesium bromide (0.48 mL, 0.55 M in Et_2O, 0.26 mmol). The diastereoselectivity was determined to be >97:3 by ^{1}H NMR. The crude material was purified by



flash chromatography (25→75% EtOAc/Hexanes) to give **8o** (72 mg, 91% yield) as a white solid. ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.34 (d, J = 10.3 Hz, 1H), 6.85 (d, J = 1.7 Hz, 1H), 6.40 (dd, J = 10.1, 1.8 Hz, 1H), 4.12 (s, 1H), 3.02 (dd, J = 16.6, 2.7 Hz, 1H), 2.61 (dd, J = 16.6, 2.7 Hz, 1H), 2.27 (t, J = 2.7 Hz, 1H), 1.26 (s, 9H); ^{13}C NMR (126 MHz, $CDCl_3$) δ 182.7, 150.0, 147.7, 136.2, 127.6, 76.0, 74.7, 60.1, 57.2, 31.5, 22.6; IR (NaCl/thin film): 3283, 3128, 2962, 1671, 1600, 1377, 1308, 1278, 1047, 1036, 957 cm^{-1}; HRMS (EI+) calc'd for $C_{13}H_{16}NO_2SBr$ $[M+H]^+$ 330.0158, found 330.0159. $[\alpha]_D^{25}$ –94.6 (*c* 1.05, CH_2Cl_2).

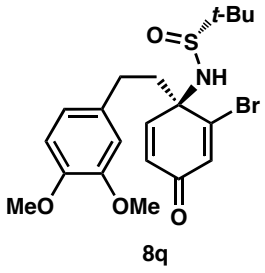
Sulfinamide 8p. Method A. The reaction was run in THF at 0 °C using quinone sulfinimine **7c**



(40 mg, 0.12 mmol) and lithium (trimethylsilyl)acetylide[7] (0.24 mmol). The diastereoselectivity was determined by LC/MS: >98:2 d.r. (30→50% MeCN/H_2O, t = 0–10 min; 50→70% MeCN/H_2O, t = 10–15 min, 1 mL/min. Minor diastereomer: t_R = 11.6 min, major diastereomer: t_R = 12.0 min). The crude material was purified by flash chromatography (10→40% EtOAc/Hexanes) to give **8p** (46 mg, 99% yield) as a pale yellow foam. ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.18 (d, J = 9.8 Hz, 1H), 6.73 (d, J = 1.7 Hz, 1H), 6.36 (dd, J = 9.9, 1.7 Hz, 1H), 4.00 (s, 1H), 1.26 (s, 9H), 0.20 (s, 9H); ^{13}C NMR (126 MHz, $CDCl_3$) δ 182.5, 147.2, 146.1, 133.5, 126.4, 98.1, 93.7, 57.2, 56.5, 22.5, –0.6; IR (NaCl/thin film): 3185, 2960, 1673, 1599, 1292, 1251, 1076, 955, 845 cm^{-1}; HRMS (EI+) calc'd for $C_{15}H_{22}NO_2SSiBr$ $[M+H]^+$ 388.0397, found 388.0401. $[\alpha]_D^{25}$ –41.0 (*c* 0.50, CH_2Cl_2).

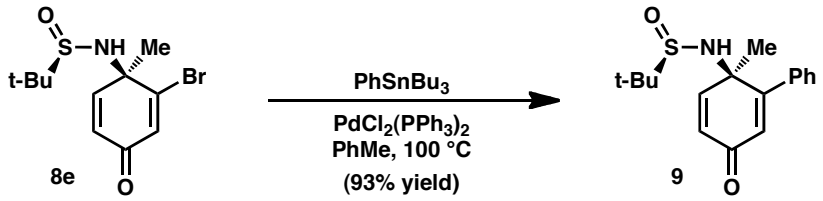
(7) Lithium (trimethylsilyl)acetylide was prepared by treating a solution of trimethylsilylacetylene (37 mL, 0.26 mmol) in THF (0.2 mL) at –78 °C with *n*-BuLi (0.16 mL, 0.24 mmol, 1.5 M in hexanes). The reaction was allowed to stir 10 min at that temperature, then warmed to room temperature prior to use. See: Raminelli, C.; Gargalaka, J.; Silveira, C.C.; Comasseto, J.V. *Tetrahedron* **2007**, *63*, 8801-8809.

Sulfinamide 8q. Method A. The reaction was run in THF using quinone sulfinimine **7c** (270 mg, 0.80 mmol) and (3,4-dimethoxyphenethyl)magnesium bromide[8] (1.6 mL, 0.55 M in THF, 0.88 mmol). The diastereoselectivity was determined by LC/MS: 96:4 d.r. (30→50% MeCN/H_2O, t = 0–10 min, 1 mL/min. Minor diastereomer: t_R = 5.8 min, major diastereomer: t_R = 7.2 min). The crude material was purified by flash chromatography (50→100% Hexanes/EtOAc) to provide **8q** (301 mg, 82% yield) as a pale yellow foam. ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.18 (d, *J* = 10.0 Hz, 1H), 6.87 (d, *J* = 1.7 Hz, 1H), 6.77 (d, *J* = 8.1 Hz, 1H), 6.66 (dd, *J* = 8.1, 2.0 Hz, 1H), 6.62 (d, *J* = 2.0 Hz, 1H), 6.46 (dd, *J* = 10.0, 1.7 Hz, 1H), 3.86 (s, 3H), 3.84 (s, 3H), 3.67 (s, 1H), 2.47 – 2.27 (m, 3H), 2.04 – 1.93 (m, 1H), 1.23 (s, 9H); ^{13}C NMR (126 MHz, $CDCl_3$) δ 183.3, 150.7, 149.6, 149.0, 147.7, 135.6, 132.0, 128.8, 120.1, 111.5, 111.4, 62.2, 56.8, 56.0, 55.9, 41.8, 29.2, 22.5; IR (NaCl/thin film): 3246, 2958, 2835, 1669, 1645, 1596, 1516, 1465, 1258, 1236, 1157, 1060, 1027, 730 cm^{-1}; HRMS (EI+) calc'd for $C_{20}H_{26}NO_4SBr$ $[M+H]^+$ 456.0839, found 456.0841. $[\alpha]_D^{25}$ –63.3 (*c* 1.15, CH_2Cl_2).



8q

Preparation of dienone 9

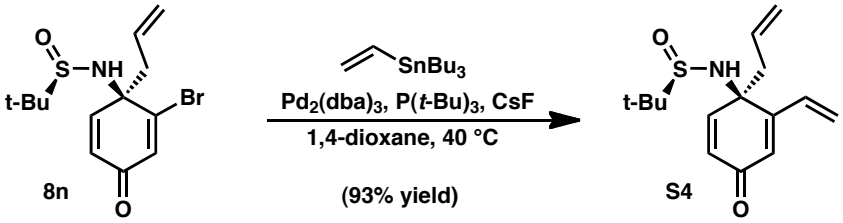


Sulfinamide **8e** (51.9 mg, 0.169 mmol, 1.0 equiv), dichloro-bis(triphenylphosphine)palladium (5.6 mg, 8.0 μmol, 0.05 equiv), and tributylphenylstannane (75 mg, 0.20 mmol, 1.2 equiv) were dissolved in PhMe (1 mL), and the resulting solution was heated to 100°C for 3 hours. The reaction mixture was cooled to room temperature, filtered through a plug of silica gel, and rinsed with EtOAc (15 mL). The filtrate was concentrated in vacuo and purified by flash chromatography (20→70% CH_2Cl_2/EtOAc) to afford phenyldienone **9** as a white solid (47.8 mg, 0.158 mmol, 93% yield). ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.50 – 7.47 (m, 2H), 7.37 – 7.35 (m, 3H), 7.09 (d, *J* = 10.0 Hz, 1H), 6.37 (d, *J* = 2.0 Hz, 1H), 6.30 (dd, *J* =

(8) To a suspension of Mg turnings (239 mg, 9.8 mmol) in THF (1 mL) was added DIBAL-H (1 mol %). The resulting suspension was heated to reflux, and a solution of 3,4-dimethoxyphenethyl bromide (1 g, 4.1 mmol) in THF (4 mL) was added dropwise. The reaction was maintained at reflux for 1.5 hrs, then cooled to room temperature and used for the sulfinimine addition reaction.

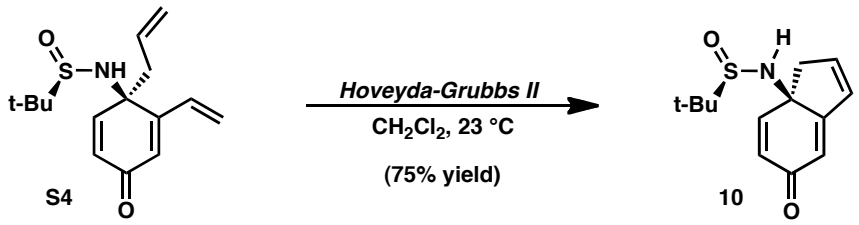
10.0 Hz, 2.0 Hz, 1H), 3.55 (s, 1H), 1.72 (s, 3H), 1.03 (s, 9H); ^{13}C NMR (126 MHz, $CDCl_3$) δ 185.7, 159.5, 154.0, 137.2, 129.5, 129.1, 128.6, 128.2, 126.2, 57.4, 56.6, 28.0, 22.3; IR (NaCl/thin film): 3434, 3151, 2986, 2958, 2930, 2868, 1660, 1626, 1570, 1472, 1457, 1364, 1290, 1274, 1147, 1114, 1040, 893, 813, 763, 705 cm^{-1}; HRMS (ES+) calc'd for $C_{17}H_{22}NO_2S$ $[M+H]^+$ 304.1366, found 304.1358; $[\alpha]_D^{25}$ –134.2 (*c* 0.81, CH_2Cl_2).

Preparation of trienone S4



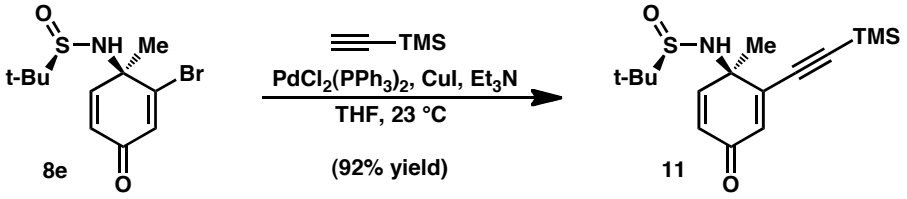
Sulfinamide **8n** (100 mg, 0.30 mmol, 1.0 equiv), tris(dibenzylideneacetone)dipalladium (4.1 mg, 0.0045 mmol, 1.5 mol%), tri-*tert*-butylphosphine (3.7 mg, 0.018 mmol, 6 mol%), cesium fluoride (101 mg, 0.66 mmol, 2.2 equiv), and vinyltributylstannane (93μL, 0.32 mmol, 1.1 equiv), and 1,4-dioxane (3.0 mL) were sequentially added to a Schlenk tube. The solution was then stirred and thoroughly degassed via sequential freeze-pump-thaw cycles (3x), then heated to 40°C for 20 hours. The solution was cooled and filtered through a plug of silica, rinsed with EtOAc (30 mL), and concentrated to afford a brown oil. Flash chromatography (1→5% MeOH/CH_2Cl_2) afforded allyltrienone **S4** (78 mg, 0.28 mmol, 93% yield) as a bright yellow oil. ^{1}H NMR (500 MHz, $CDCl_3$) δ 6.89 (d, *J* = 10.1 Hz, 1H), 6.46 (d, *J* = 1.8 Hz), 6.42 (ddd, *J* = 17.4, 11.0, 0.6 Hz, 1H), 6.23 (dd, *J* = 10.1, 2.0 Hz, 1H), 5.79 (dd, *J* = 17.5, 1.0 Hz, 1H), 5.40 (dd, *J* = 11.0, 1.0 Hz, 1H), 5.45 – 5.35 (m, 1H), 5.08 – 5.00 (m, 2H), 3.88 (s, 1H), 2.55 – 2.43 (m, 2H), 1.11 (s, 9H); ^{13}C NMR (126 MHz, $CDCl_3$) δ 185.9, 155.3, 151.8, 132.1, 129.5, 127.9, 125.7, 121.1, 120.8, 59.0, 56.5, 43.5, 22.5; IR (NaCl/thin film): 3197, 2980, 2960, 2234, 1663, 1624, 1474, 1420, 1390, 1364, 1295, 1192, 1175, 1154, 1057, 992, 9224, 895, 818, 734 cm^{-1}; HRMS (ES+) calc'd for $C_{15}H_{22}NO_2S$ $[M+H]^+$ 280.1366, found 280.1376; $[\alpha]_D^{25}$ –247.5 (*c* 0.92, CH_2Cl_2).

Preparation of bicycle 10



To a solution of trienone **S4** (18 mg, 0.065 mmol, 1.0 equiv) in CH_2Cl_2 (0.75 mL) was added Hoveyda-Grubbs 2nd Generation Catalyst (2.6 mg, 4.6 μmol, 0.06 equiv). The solution was stirred at 23°C for 3 hrs. The reaction mixture was concentrated and purified by flash chromatography (1→5% MeOH/CH_2Cl_2) to afford bicycle **10** (14 mg, 0.058 mmol, 88% yield) as a white crystalline solid. ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.08 (dd, *J* = 9.8, 0.7 Hz, 1H), 6.58 (dt, *J* = 5.2 Hz, 2.6 Hz, 1H), 6.47 (dt, *J* = 5.8, 2.0 Hz, 1H), 6.25 (dd, *J* = 9.8, 1.7 Hz, 1H), 6.13 (d, *J* = 1.5 Hz, 1H), 3.43 (s, 1H), 2.77 (t, *J* = 2.2 Hz, 2H), 1.12 (s, 9H); ^{13}C NMR (126 MHz, $CDCl_3$) δ 186.3, 166.7, 145.5, 143.7, 130.9, 130.6, 120.2, 61.7, 56.4, 43.4, 22.3; IR (NaCl/thin film): 3152, 2979, 2918, 2866, 1726, 1653, 1634, 1597, 1561, 1474, 1457, 1379, 1362, 1289, 1190, 1050, 1037, 929, 891, 865, 811, 740 cm^{-1}; HRMS (ES+) calc'd for $C_{13}H_{17}NO_2S$ $[M+H]^+$ 252.1058, found 252.1061; $[\alpha]_D^{25}$ –80.4 (*c* 0.29, CH_2Cl_2).

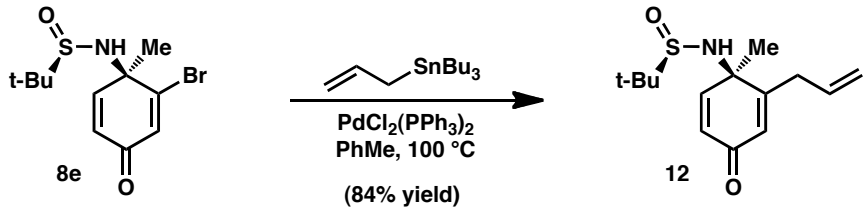
Preparation of dienone 11



A 10 mL flask was charged with sulfinamide **8e** (50 mg, 0.16 mmol, 1.0 equiv), dichlorobis(triphenylphosphine)palladium (6.0 mg, 8 μmol, 0.05 equiv), copper iodide (3.0 mg, 16 μmol, 0.1 equiv), and THF (0.8 mL). Nitrogen was bubbled through the resulting suspension for 20 minutes, then Et_3N (0.8 mL) and ethynyltrimethylsilane (25 μL, 0.18 mmol, 1.1 equiv) were added. The reaction mixture was allowed to stir 1 h at room temperature. The mixture was filtered through Celite, rinsed with EtOAc, concentrated, and purified by flash chromatography (0→70% EtOAc/CH_2Cl_2) to provide dienone **11** (49 mg, 92% yield) as a pale yellow solid. ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.01 (d, *J* = 10.1 Hz, 1H), 6.44 (d, *J* = 2.0 Hz, 1H), 6.28 (dd, *J* = 10.3, 2.0 Hz, 1H), 3.62 (s, 1H), 1.60 (s, 3H), 1.22 (s, 9H), 0.21 (s, 9H); ^{13}C NMR (126 MHz,

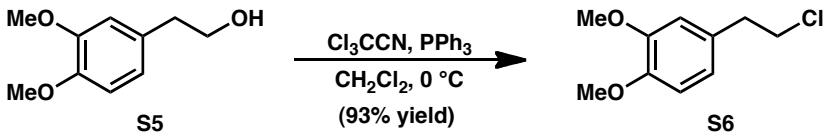
$CDCl_3$) δ 184.7, 152.1, 143.7, 133.6, 127.5, 107.4, 100.8, 56.4, 56.3, 27.9, 22.4, –0.5; IR (NaCl/thin film): 3139, 2960, 2253, 2149, 1662, 1623, 1586, 1364, 1251, 1105, 1043, 897, 843 cm^{-1}; HRMS (ES+) calc'd for $C_{16}H_{25}NO_2SSi$ $[M+H]^+$ 324.1454, found 324.1463; $[\alpha]_D^{25}$ –191.8 (*c* 1.13, CH_2Cl_2).

Preparation of dienone 12



Sulfinamide **8e** (48 mg, 0.16 mmol, 1.0 equiv), dichloro-bis(triphenylphosphine)palladium (5.5 mg, 7.8 μmol, 0.05 equiv), and allyltributyltin (62 mg, 0.19 mmol, 1.2 equiv) were dissolved in PhMe (1 mL), and the resulting solution was heated to 100°C for 3 hours. The reaction mixture was cooled to room temperature, filtered through a plug of silica gel, and rinsed with EtOAc (15 mL). The resulting solution was concentrated in vacuo and the crude residue was purified by flash chromatography (20→70% EtOAc/CH_2Cl_2) to afford allyldienone **12** (35.1 mg, 0.131 mmol, 84% yield) as a white solid. ^{1}H NMR (500 MHz, $CDCl_3$) δ 6.96 (d, *J* = 10.0 Hz, 1H), 6.22 (dd, *J* = 9.8, 2.0 Hz, 1H), 6.19 (app. q, *J* = 1.6 Hz, 1H), 5.75 (m, 1H), 5.20 (dq, *J* = 10.0, 1.2 Hz, 1H), 5.14 (dq, *J* = 17.0, 1.5 Hz, 1H), 3.55 (s, 1H), 3.15 (dddd, *J* = 17.3, 6.3, 2.8, 1.4 Hz, 1H), 2.99 (dddd, *J* = 17.3, 7.3, 2.3, 1.3 Hz, 1H), 1.48 (s, 3H), 1.20 (s, 9H). ^{13}C NMR (126 MHz, $CDCl_3$) δ 185.5, 160.5, 153.6, 133.5, 128.4, 126.7, 118.9, 57.2, 56.4, 34.7, 26.3, 22.5; IR (NaCl/thin film): 3128, 2983, 2964, 2928, 2870, 1672, 1635, 1460, 1419, 1388, 1363, 1285, 1270, 1157, 1064, 1043, 916, 892, 810 cm^{-1}; HRMS (ES+) calc'd for $C_{14}H_{22}NO_2S$ $[M+H]^+$ 268.1366, found 268.1376. $[\alpha]_D^{25}$ –82.7 (*c* 0.70, CH_2Cl_2).

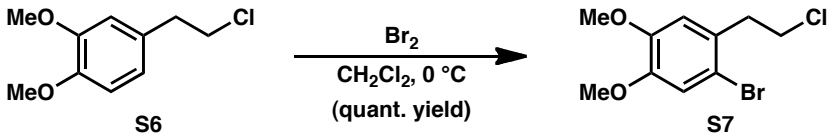
Preparation of 3,4-dimethoxyphenethyl chloride (S6)



To a solution of 3,4-dimethoxyphenethanol (**S5**) (4.72g, 25.9 mmol, 1.0 equiv) in CH_2Cl_2 (250 mL) at 0°C was added triphenylphosphine (13.6 g, 51.8 mmol, 2.0 equiv). The solution was

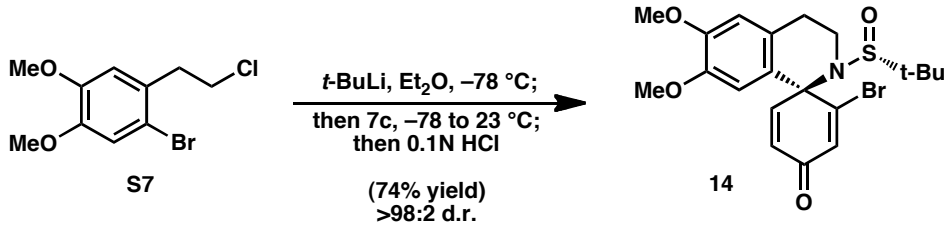
stirred for 10 min, and trichloroacetonitrile (3.89 mL, 38.9 mmol, 1.5 equiv) was added dropwise via syringe over 5 min. The solution was stirred at 0 °C for 10 min and was then slowly warmed to room temperature. After stirring for an additional 45 minutes at room temperature, the reaction mixture was concentrated and purified by flash chromatography (5→20% EtOAc/Hexanes) to afford 3,4-dimethoxyphenethyl chloride (**S6**) (4.82 g, 24.0 mmol, 93 % yield) as a clear colorless oil. ^{1}H NMR (500 MHz, $CDCl_3$) δ 6.81 (d, *J* = 8.30 Hz, 1H), 6.76 (dd, *J* = 8.1, 2.0 Hz, 1H), 6.73 (d, *J* = 2.0 Hz, 1H), 3.87 (s, 3H), 3.86 (s, 3H), 3.68 (t, *J* = 7.5 Hz, 2H), 3.00 (t, *J* = 7.5 Hz, 2H); ^{13}C NMR (126 MHz, $CDCl_3$) δ 148.9, 147.9, 130.6, 120.7, 112.0, 111.2, 55.8, 55.8, 45.1, 38.7; IR (NaCl/thin film): 3000, 2956, 2909, 2867, 2934, 1607, 1591, 1516, 1464, 1418, 1325, 1260, 1232, 1191, 1146, 1027, 914, 854, 809, 767 cm^{-1}; HRMS (ES+) calc'd for $C_{10}H_{13}O_2Cl$ $[M+H]^+$ 200.0604, found 200.0591.

Preparation of 2-bromo-3,4-dimethoxyphenethyl chloride (S7)



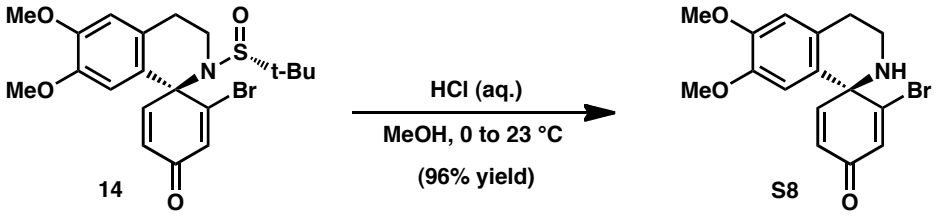
To a solution of 3,4-dimethoxyphenethyl chloride **S6** (4.82 g, 24.0 mmol, 1.0 equiv) in CH_2Cl_2 (240 mL) at 0°C was added bromine (1.25 mL, 24.2 mmol, 1.01 equiv) dropwise via syringe. (*Caution! A copious amount of HBr gas is generated as the reaction proceeds. A 16-gauge needle was pierced through the septa to allow the reaction to vent*). The solution was stirred at 0°C for 10 min, warmed to room temperature, and stirred at that temperature for 20 min. The reaction mixture was quenched with saturated aqueous $Na_2S_2O_3$ (50 mL) and washed with saturated sodium bicarbonate (3 x 100 mL). The combined aqueous layers were back-extracted with CH_2Cl_2 (1 x 50 mL), and the combined organic layers were dried over Na_2SO_4, concentrated, and purified by flash chromatography (5→20% EtOAc/Hexanes) to afford bromide **S7** (6.70 g, 24.0 mmol, quantitative yield) as white needles. ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.01 (s, 1H), 6.77 (s, 1H), 3.87 (s, 3H), 3.86 (s, 3H), 3.71, (t, *J* = 7.3 Hz, 2H), 3.12 (t, *J* = 7.3 Hz, 2H); ^{13}C NMR (126 MHz, $CDCl_3$) δ 148.6, 148.3, 129.2, 115.6, 114.2, 113.9, 56.2, 56.1, 43.5, 39.1; IR (NaCl/thin film): 3009, 2955, 2940, 2906, 2836, 1602, 1576, 1510, 1469, 1461, 1451, 1435, 1382, 1344, 1266, 1254, 1217, 1166, 1033, 959, 856, 834, 865, 834, 802, 759 cm^{-1}; HRMS (ES+) calc'd for $C_{10}H_{12}O_2Cl^{81}Br$ $[M+H]^+$ 279.9689, found 279.9691.

Preparation of sulfinamide 14



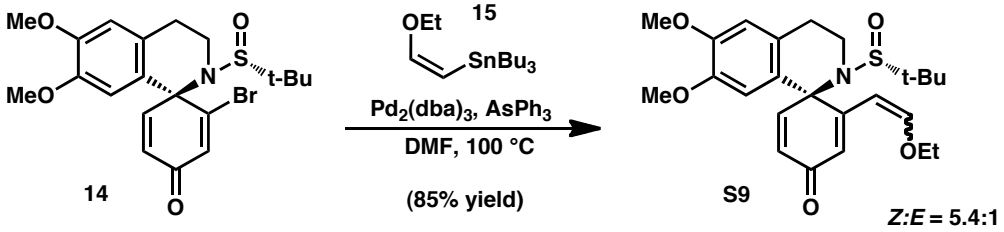
To a solution of aryl bromide **S7** (506 mg, 1.8 mmol) in Et_2O (18 mL) at –78°C was added a solution of *tert*-butyllithium (1.6 M in pentane, 1.31 mL, 2.1 mmol) dropwise via syringe, and the resulting mixture was stirred 2 hrs at –78 °C. A solution of sulfinimine **7c** (495 mg, 1.5 mmol) in Et_2O (3 mL) was added by cannula transfer over 5 min. The reaction mixture was stirred 1 h at –78°C, and allowed to warm to room temperature and stirred for an additional hour. The reaction was quenched by the slow addition of aqueous HCl (0.1 N) and stirred for 30 min at room temperature. The resulting mixture was diluted with EtOAc (60 mL) and washed with saturated aq. $NaHCO_3$ (3 x 20 mL). The combined aqueous layers were back extracted with EtOAc (1 x 25 mL), and the combined organic layers were dried over Na_2SO_4 and concentrated to give a light brown oil. The diastereoselectivity was determined by LC/MS: >98:2 d.r. (5→95% MeCN/H_2O, t = 0–10 min, 1 mL/min. Minor diastereomer: t_R = 3.7 min, major diastereomer: t_R = 4.0 min). Flash chromatography (10→30% EtOAc/CH_2Cl_2) afforded tricyclic dienone **14** (491 mg, 1.08 mmol, 74% yield) as an off-white, flaky solid. ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.30 (d, *J* = 9.8Hz, 1H), 6.77 (d, *J* = 1.5 Hz, 1H), 6.63 (s, 1H), 6.42 (dd, *J* = 9.8, 1.5 Hz, 1H), 6.32 (s, 1H), 3.86 (s, 3H), 3.72 (s, 3H), 3.52 (dt, *J* = 13.2, 4.4 Hz, 1H), 3.33 (ddd, *J* = 13.2, 9.8, 2.9 Hz, 1H), 3.03 (ddd, *J* =15.4, 10.0, 3.9 Hz, 1H), 2.79 (dt, *J* = 15.4, 3.8 Hz, 1H), 1.30 (s, 9H); ^{13}C NMR (126 MHz, $CDCl_3$) δ 183.7, 153.0, 149.6, 149.1, 148.3, 133.5, 128.5, 126.0, 122.7, 111.5, 109.1, 66.7, 59.2, 56.1, 55.8, 38.5, 29.0, 24.4; IR (NaCl/thin film): 2958, 2925, 2855, 1669, 1644, 1594, 1516, 1436, 1363, 1298, 1262, 1230, 1199, 1126, 1076, 1022, 954, 915, 796, 731 cm^{-1}; HRMS (EI+) calc'd for $C_{20}H_{24}BrNO_4S$ $[M+H]^+$ 454.0682, found 454.0697; $[\alpha]_D^{25}$ –17.3 (*c* 0.39, CH_2Cl_2).

Preparation of aminodienone S8



To a solution of dienone **14** (376 mg, 0.83 mmol, 1.0 equiv) in MeOH (4 mL) was added HCl (2N in MeOH, 4 mL) dropwise at 0°C. The solution stirred for 10 min 0 °C, then warmed to 23 °C and stirred for an additional 30 min. The reaction was quenched through the addition of aq. NaOH (10% w/w, 30 mL) and extracted with CH_2Cl_2 (3 x 25 mL). The combined organic layers were dried over Na_2SO_4, concentrated, and purified by flash chromatography (30→80% EtOAc/Hexanes) to afford amine **S8** as a pale tan solid (277 mg, 0.79 mmol, 96% yield). ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.25 (d, *J* = 9.8Hz, 1H), 6.74 (d, *J* = 1.7 Hz, 1H), 6.62 (s, 1H), 6.30 (s, 1H), 6.16 (dd, *J* = 9.8 Hz, 1.8 Hz, 1H), 3.86 (s, 3H), 3.72 (s, 3H), 3.35 (ddd, *J* = 12.1 Hz, 5.2 Hz, 3.3 Hz, 1H), 3.23 (ddd, *J* = 12.9 Hz, 10.4 Hz, 3.5 Hz, 1H), 3.05-2.97 (m, 1H), 2.68 (dt, *J* = 15.6 Hz, 3.4 Hz, 1H), 1.97 (s, 1H); ^{13}C NMR (126 MHz, $CDCl_3$) δ 183.7, 155.5, 151.7, 148.8, 148.0, 132.5, 128.7, 123.1, 122.9, 112.0, 108.8, 61.0, 56.0, 55.8, 39.2, 29.0; IR (NaCl/thin film): 3324, 3055, 2999, 2932, 2832, 1667, 1645, 1610, 1593, 1513, 1464, 1402, 1380, 1363, 1347, 1260, 1226, 1192, 1118, 1039, 1008, 954, 881, 823, 808, 732 cm^{-1}; HRMS (ES+) calc'd for $C_{16}H_{17}BrNO_3$ $[M+H]^+$ 350.0386, found 350.0383. $[\alpha]_D^{25}$ +52.9 (*c* 0.62, CH_2Cl_2).

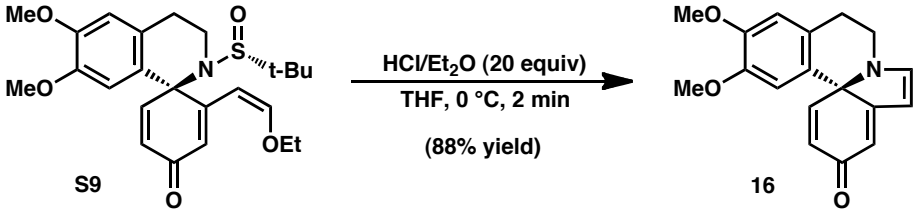
Preparation of trienone S9



To a solution of dienone **14** (238 mg, 0.52 mmol, 1.0 equiv) in DMF (10 mL) was added tris(dibenzylideneacetone)dipalladium (14 mg, 0.016 mmol, 0.030 equiv), triphenylarsine (19 mg, 0.063 mmol, 0.12 equiv) and *cis*-2-ethoxyvinyltributylstannane (**15**) (164 mg, 0.63 mmol, 1.2 equiv). Nitrogen was then bubbled through the solution for 30 minutes, and the reaction was then stirred at 100°C for 1 h. Upon cooling to room temperature, the reaction mixture was passed

through a plug of Celite, rinsed and diluted with Et_2O (40 mL), and washed with H_2O (3x50 mL). The combined organic layers were dried over $MgSO_4$, concentrated, and purified by flash chromatography (35→100% EtOAc/Hexanes) to afford trienone **S9** (~5.4:1 mixture of *Z:E*-isomers by ^{1}H NMR) as a tan solid (199 mg, 0.446 mmol, 85% yield). *Z*-**S9**: ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.14 (d, *J* = 10.0 Hz, 1H), 7.13 (d, *J* = 2.0 Hz, 1H), 6.59 (s, 1H), 6.36 (dd, *J* = 10.0, 2.0 Hz, 1H), 6.34 (s, 1H), 6.28 (d, *J* = 7.3 Hz, 1H), 4.46 (d, *J* = 7.1 Hz, 1H), 3.97 – 3.90 (m, 2H), 3.84 (s, 3H), 3.67 (s, 3H), 3.49 (ddd, *J* = 13.1, 4.3, 3.4 Hz, 1H), 3.17 (ddd, *J* = 13.1, 11.3, 2.8 Hz, 1H), 3.05 (ddd, *J* = 15.5, 11.2, 4.0 Hz, 1H), 2.81 (dt, *J* = 15.5, 3.0 Hz, 1H), 1.28 (t, *J* = 7.1 Hz, 3H), 1.19 (s, 9H); ^{13}C NMR (126 MHz, $CDCl_3$) δ 187.4, 155.4, 153.3, 148.5, 148.1, 147.3, 127.6, 127.2, 126.8, 125.0, 111.3, 109.9, 102.8, 70.4, 63.9, 58.4, 56.0, 55.8, 38.1, 29.0, 24.1, 15.4. IR (NaCl/thin film): 2979, 2959, 2932, 1658, 1625, 1574, 1516, 1464, 1360, 1262, 1249, 1124, 1072, 1038, 1021, 893, 795 cm^{-1}; HRMS (ES+) calc'd for $C_{24}H_{32}NO_5S$ $[M+H]^+$ 446.1996, found 446.2006. *E*-**S9** gave the following diagnostic resonances by ^{1}H NMR (500 MHz, $CDCl_3$): 7.18 (d, *J* = 10.0 Hz, 1H), 6.89 (d, *J* = 13.0 Hz, 1H), 5.09 (d, *J* = 13.2 Hz).

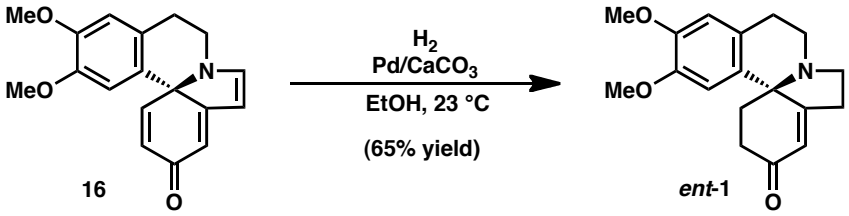
Preparation of enamine 16



To a solution of trienone **S9** (50 mg, 0.11 mmol, 1.0 equiv) in THF (2.2 mL) at 0 °C was added a solution of hydrogen chloride (2.0 M solution in Et_2O, 1.1 mL, 2.2 mmol, 20 equiv) dropwise by syringe. The reaction was allowed to stir 2 min at 0 °C, then quenched by the addition of aq. NaOH (10% w/w, 4 mL) and stirred for an additional 5 min. The mixture was diluted with H_2O (5 mL) and extracted with EtOAc (4 x 10 mL). The combined organic layers were dried over Na_2SO_4, concentrated, and purified by flash chromatography (10→20% EtOAc/ CH_2Cl_2) to afford enamine **16** (29 mg, 0.098 mmol, 88% yield) as a bright orange solid. ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.02 (d, *J* = 9.8 Hz, 1H), 6.99 (d, *J* = 3.4 Hz, 1H), 6.85 (s, 1H), 6.53 (s, 1H), 6.06 (dd, *J* = 9.8 Hz, 2.0 Hz, 1H), 6.03 (d, *J* = 1.5 Hz, 1H), 5.62 (d, *J* = 3.4 Hz, 1H), 3.83 (s, 1H), 3.78 (ddd, *J* = 14.2 Hz, 6.8 Hz, 1.0 Hz, 1H), 3.74 (s, 1H), 3.56 (ddd, *J* = 14.2 Hz, 12.7 Hz, 4.4 Hz, 1H), 2.93 (ddd, *J* = 16.9 Hz, 12.5 Hz, 6.4 Hz, 1H), 2.75 (dd, 16.4 Hz, 4.2 Hz, 1H); ^{13}C

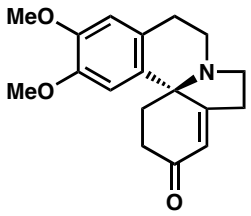
NMR (126 MHz, $CDCl_3$) δ 186.5, 172.8, 152.9, 148.6, 148.0, 143.6, 127.8, 125.7, 124.6, 112.7, 111.4, 107.5, 105.2, 71.3, 55.9, 55.8, 42.1, 28.5; IR (NaCl/thin film): 2992, 2955, 2936, 2835, 1636, 1605, 1571, 1523, 1513, 1455, 1450, 1442, 1402, 1356, 1333, 1256, 1218, 1204, 1190, 1166, 1140, 1111, 1081, 1068, 1039, 1001, 895, 852, 784, 731 cm^{-1}; HRMS (ES+) calc'd for $C_{18}H_{18}NO_3$ $[M+H]^+$ 296.1281, found 296.1272. $[\alpha]^{25}_D$ –1307 (*c* 0.72, CH_2Cl_2).

Preparation of (–)-3-demethoxyerythratidinone (*ent*-1)



To a solution of enamine **16** (20 mg, 0.068 mmol, 1.0 equiv) in EtOH (3.3 mL) was added palladium on $CaCO_3$ (14 mg, 5 wt %, 7.0 μmol, 0.1 equiv). The solution was placed under an atmosphere of hydrogen (balloon) and was allowed to stir 3 h at room temperature. The reaction mixture was filtered through a plug of Celite, rinsed with EtOAc, concentrated, and purified by flash chromatography (0→20% acetone/CH_2Cl_2) to afford (–)-demethoxyerythratidinone (***ent*-1**) as a pale yellow oil (13 mg, 0.043 mmol, 65% yield). ^{1}H NMR (500 MHz, $CDCl_3$) δ 6.65 (s, 1H), 6.56 (s, 1H), 6.11 (app. s, 1H), 3.86 (s, 3H), 3.75 (s, 3H), 3.49 (ddd, *J* = 14.4, 11.7, 6.6 Hz, 1H), 3.24 (dd, *J* = 14.4 Hz, 7.6 Hz, 1H), 3.12 – 3.00 (m, 2H), 2.86 (q, *J* = 7.7 Hz, 1H), 2.77 – 2.68 (m, 1H), 2.62 – 2.50 (m, 3H), 2.46 (dd, *J* = 18.3, 4.2 Hz, 1H), 2.31 (ddd, *J* = 12.5, 5.6, 2.0 Hz, 1H), 2.24 – 2.15 (m, 1H); ^{13}C NMR (126 MHz, $CDCl_3$) δ 199.5, 169.2, 148.3, 146.8, 125.7, 124.8, 123.4, 112.8, 110.3, 63.5, 56.0, 55.9, 45.7, 40.1, 36.1, 32.8, 28.7, 21.4; IR (NaCl/thin film): 2928, 2848, 1667, 1509, 1464, 1329, 1253, 1229, 1205, 1165, 1106 cm^{-1}; HRMS (ES+) calc'd for $C_{18}H_{21}NO_3$ $[M+H]^+$ 300.1600, found 300.1606. $[\alpha]^{25}_D$ – 296.5 (*c* 0.57, $CHCl_3$).

Comparison of spectroscopic data for natural[9] and synthetic 3-demethoxy-erythratidinone.[10,11]



(–)-3-demethoxyerythratidinone *(ent*-1)

^{1}H NMR Data

Natural[12]	Simpkins Synthetic (+)-1 (400 MHz)	Reisman Synthetic (–)-1 (500 MHz)
δ 6.59 (s, 1H)	δ 6.66 (s, 1H)	δ 6.65 (s, 1H)
6.51 (s, 1H)	6.56 (s, 1H)	6.56 (s, 1H)
6.04 (s, 1H)	6.12 (s, 1H)	6.11 (app. s, 1H)
3.79 (s, 3H)	3.88 (s, 3H)	3.86 (s, 3H)
3.68 (s, 3H)	3.76 (s, 3H)	3.75 (s, 3H)
3.52 – 1.85 (m, 12H)	3.52 – 3.45 (m, 1H)	3.49 (ddd, *J* = 14.4, 11.7, 6.6 Hz, 1H)
	3.26 (dd, *J* = 14.4, 7.6 Hz, 1H)	3.24 (dd, *J* = 14.4 Hz, 7.6 Hz, 1H)
	3.12 – 3.03 (m, 2H)	3.12 – 3.00 (m, 2H)
	2.92 – 2.68 (m, 2H)	2.86 (q, *J* = 7.7 Hz, 1H), 2.77 – 2.68 (m, 1H)
	2.68 – 2.43 (m, 4H)	2.62 – 2.50 (m, 3H), 2.46 (dd, *J* = 18.3, 4.2 Hz, 1H)
	2.33 – 2.24 (m, 2H)	2.31 (ddd, *J* = 12.5, 5.6, 2.0 Hz, 1H)
		2.24 – 2.15 (m, 1H)

^{13}C NMR Data

Natural	Simpkins Synthetic (+)-1 (101 MHz)	Reisman Synthetic (–)-1 (126 MHz)
No ^{13}C NMR reported with isolation.	199.3	δ 199.5
	168.5	169.2*
	148.4	148.3
	146.9	146.8
	125.4	125.7
	124.4	124.8*

[9] Isolation of natural (+)-3-demethoxyerythratidinone: D. H. R. Barton, A. A. L. Gunatilaka, R.M. Letcher, A. M. F. T. Lobo and D. A. Widdowson, *J. Chem. Soc., Perk. Trans. 1*, 1973, 874-880.

[10] F. Zhang, N. S. Simpkins and C. Wilson, *Tetrahedron Lett*., 2007, **48**, 5942-5947.

[11] As noted by Simpkins et. al, relatively limited spectroscopic data is available for the natural product despite a number of completed total syntheses. See reference 9.

[12] ^{1}H NMR resonances were converted from the reported units of τ to chemical shift δ by the conversion formula τ = 10 – δ (ppm). As the authors do not list a standard for reference, the converted chemical shifts remain unreferenced.

* Although these chemical shifts exhibit small discrepancies with those reported by Simpkins et. al, they remain in close agreement with other reported data, see: J. M. Joo. R. A. David, Y. Yuan, C. Lee, *Org. Lett.,* 2010, **12**, 5704–5705.

	123.9	123.4
	112.8	112.8
	110.2	110.3
	63.7	63.5
	56.0	56.0
	55.9	55.9
	45.7	45.7
	40.1	40.1
	35.9	36.1
	32.8	32.8
	28.6	28.7
	21.5	21.4

Infrared Data (C=O Stretch)

Natural	Simpkins Synthetic (+)-1	Simpkins Synthetic (+)-1
1667 cm^{-1}	1666 cm^{-1}	1667 cm^{-1}

Optical Rotation

Natural	Simpkins Synthetic (+)-1	Simpkins Synthetic (+)-1
$[\alpha]^{22}_D$ +325 (*c* 0.249, $CHCl_3$).	$[\alpha]^{26}_D$ +316 (*c* 0.4, $CHCl_3$)	$[\alpha]^{25}_D$ –297 (*c* 0.57, $CHCl_3$)

Supplementary Material (ESI) for Chemical Science

Benzoquinone Sulfinyl Imines as Versatile Intermediates in Alkaloid Synthesis: Total Synthesis of (–)-3-Demethoxyerythratidinone

Kangway V. Chuang, Raul Navarro, Sarah E. Reisman*

The Warren and Katharine Schlinger Laboratory for Chemistry and Chemical Engineering, Division of Chemistry and Chemical Engineering, California Institute of Technology, Pasadena, California 91125
reisman@caltech.edu

Supporting Information 2 (Spectral Data):

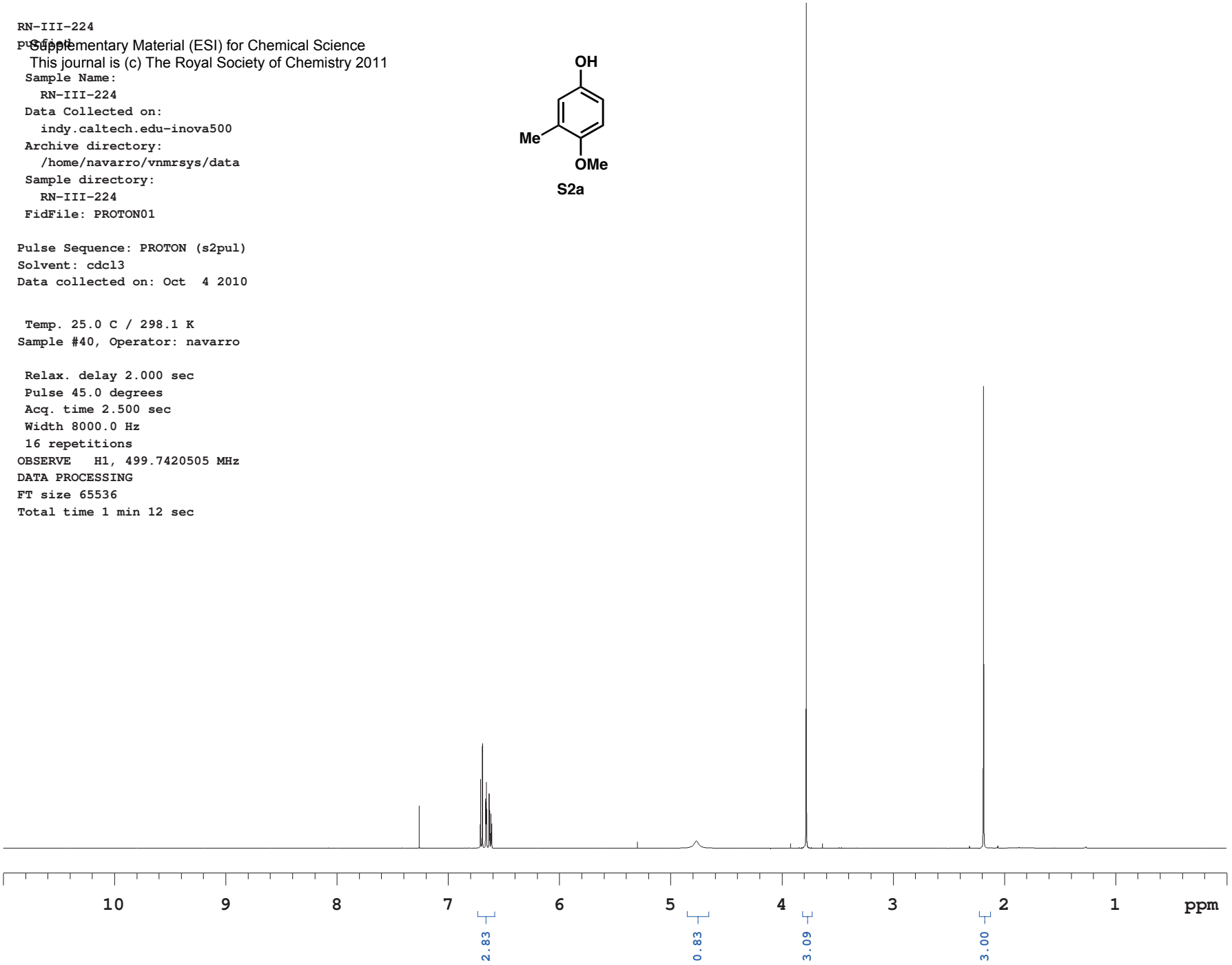
RN-III-224
pulprog
Sample Name:
RN-III-224
Data Collected on:
indy.caltech.edu-inova500
Archive directory:
/home/navarro/vnmrsys/data
Sample directory:
RN-III-224
FidFile: PROTON01

Pulse Sequence: PROTON (s2pul)
Solvent: cdcl3
Data collected on: Oct 4 2010

Temp. 25.0 C / 298.1 K
Sample #40, Operator: navarro

Relax. delay 2.000 sec
Pulse 45.0 degrees
Acq. time 2.500 sec
Width 8000.0 Hz
16 repetitions
OBSERVE H1, 499.7420505 MHz
DATA PROCESSING
FT size 65536
Total time 1 min 12 sec

S2a



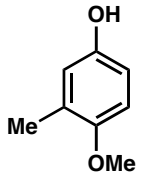
RN-III-224
pulprog

Sample Name:
RN-III-224
Data Collected on:
indy.caltech.edu-inova500
Archive directory:
/home/navarro/vnmrsys/data
Sample directory:
RN-III-224
FidFile: CARBON01

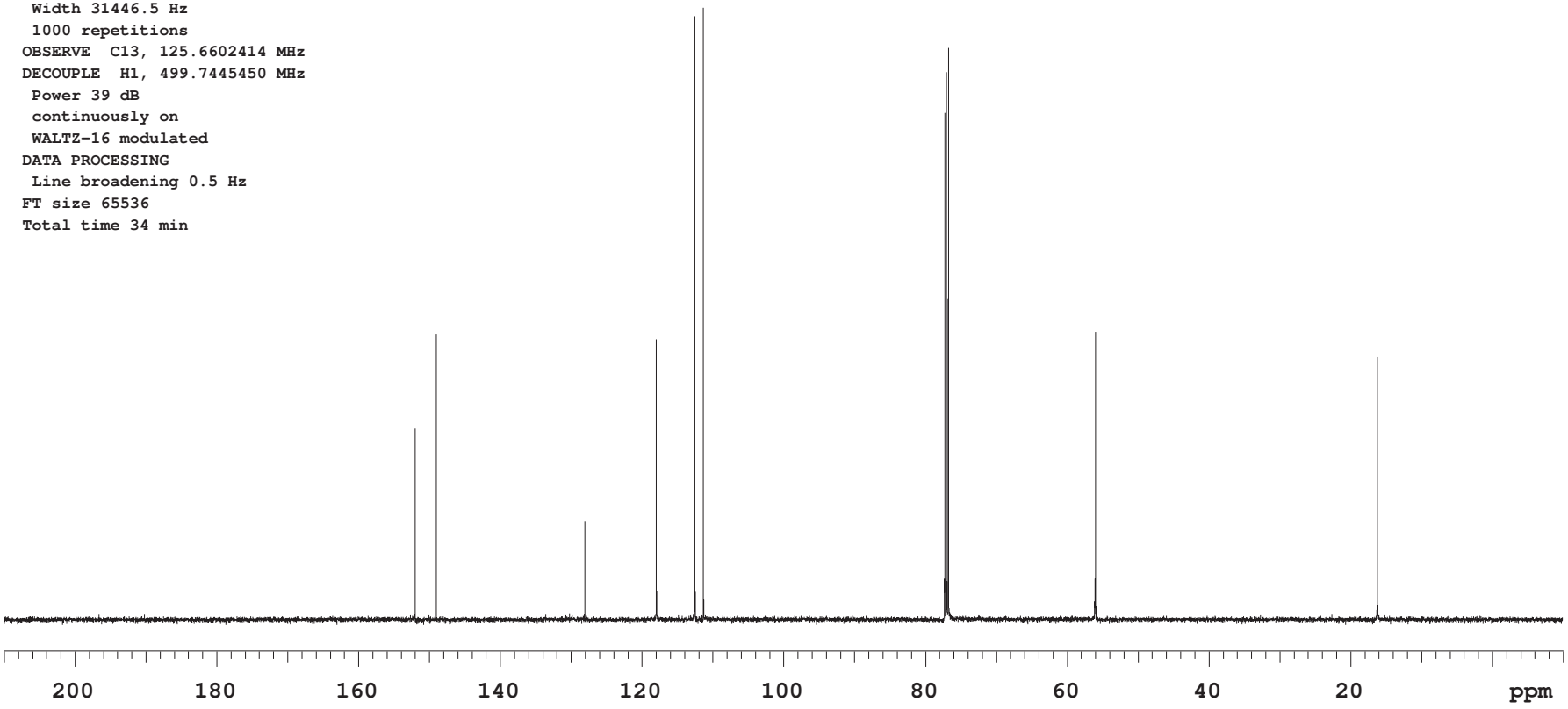
Pulse Sequence: CARBON (s2pul)
Solvent: cdcl3
Data collected on: Oct 4 2010

Temp. 25.0 C / 298.1 K
Sample #40, Operator: navarro

Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.042 sec
Width 31446.5 Hz
1000 repetitions
OBSERVE C13, 125.6602414 MHz
DECOUPLE H1, 499.7445450 MHz
Power 39 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 0.5 Hz
FT size 65536
Total time 34 min



S2a



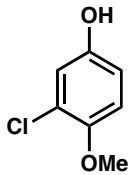
RN-III-214
purified

Sample Name:
RN-III-214
Data Collected on:
indy.caltech.edu-inova500
Archive directory:
/home/navarro/vnmrsys/data
Sample directory:
RN-III-214
FidFile: PROTON01

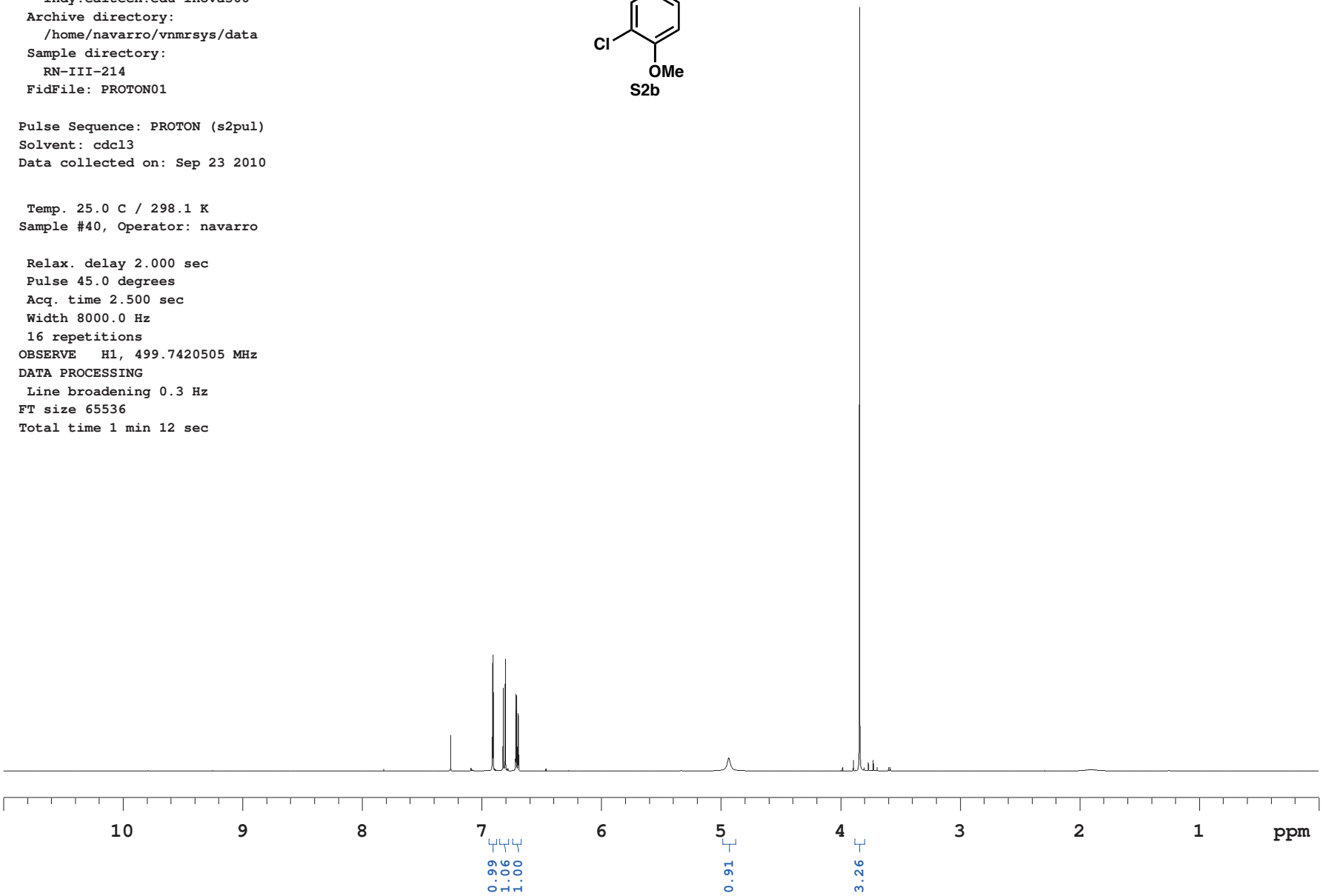
Pulse Sequence: PROTON (s2pul)
Solvent: cdcl3
Data collected on: Sep 23 2010

Temp. 25.0 C / 298.1 K
Sample #40, Operator: navarro

Relax. delay 2.000 sec
Pulse 45.0 degrees
Acq. time 2.500 sec
Width 8000.0 Hz
16 repetitions
OBSERVE H1, 499.7420505 MHz
DATA PROCESSING
Line broadening 0.3 Hz
FT size 65536
Total time 1 min 12 sec



S2b



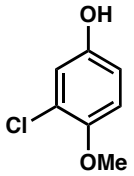
RN-III-214
purified

Sample Name:
RN-III-214
Data Collected on:
indy.caltech.edu-inova500
Archive directory:
/home/navarro/vnmrsys/data
Sample directory:
RN-III-214
FidFile: CARBON01

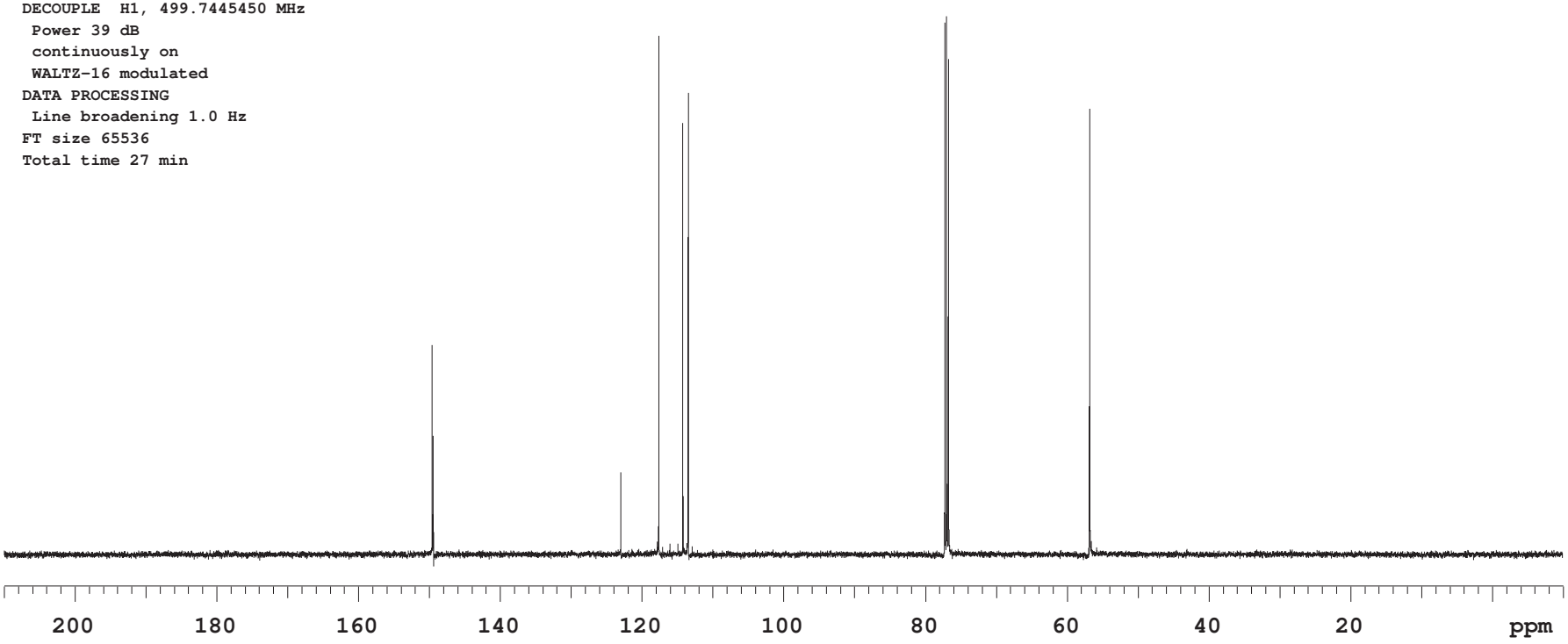
Pulse Sequence: CARBON (s2pul)
Solvent: cdcl3
Data collected on: Sep 23 2010

Temp. 25.0 C / 298.1 K
Sample #40, Operator: navarro

Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.042 sec
Width 31446.5 Hz
800 repetitions
OBSERVE C13, 125.6602414 MHz
DECOUPLE H1, 499.7445450 MHz
Power 39 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
FT size 65536
Total time 27 min



S2b



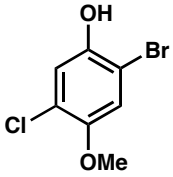
RN-III-215
purified

Sample Name:
RN-III-215
Data Collected on:
indy.caltech.edu-inova500
Archive directory:
/home/navarro/vnmrsys/data
Sample directory:
RN-III-215
FidFile: PROTON01

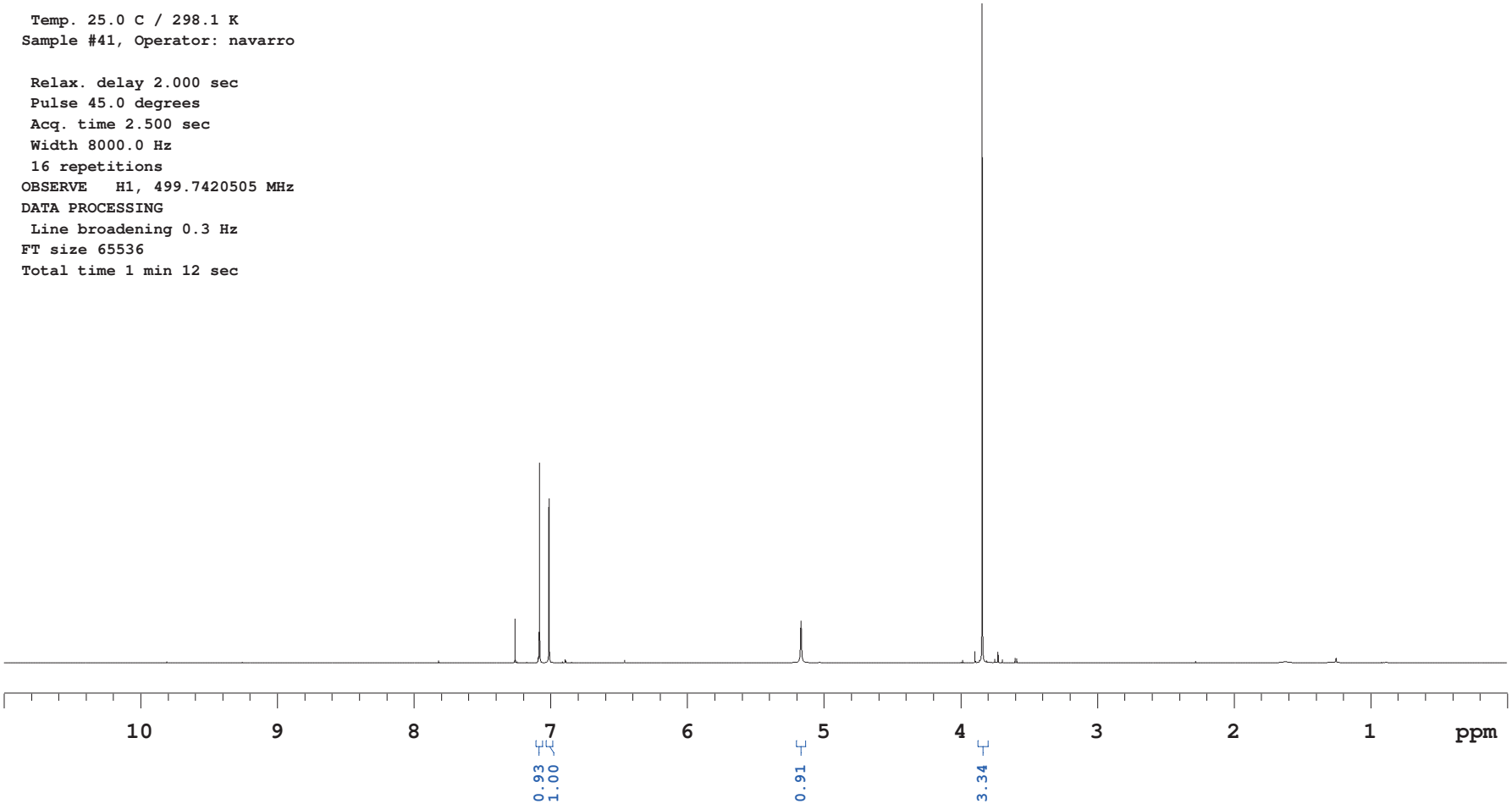
Pulse Sequence: PROTON (s2pul)
Solvent: cdcl3
Data collected on: Sep 23 2010

Temp. 25.0 C / 298.1 K
Sample #41, Operator: navarro

Relax. delay 2.000 sec
Pulse 45.0 degrees
Acq. time 2.500 sec
Width 8000.0 Hz
16 repetitions
OBSERVE H1, 499.7420505 MHz
DATA PROCESSING
Line broadening 0.3 Hz
FT size 65536
Total time 1 min 12 sec



S3b



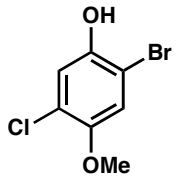
RN-III-215
purified

Sample Name:
RN-III-215
Data Collected on:
indy.caltech.edu-inova500
Archive directory:
/home/navarro/vnmrsys/data
Sample directory:
RN-III-215
FidFile: CARBON01

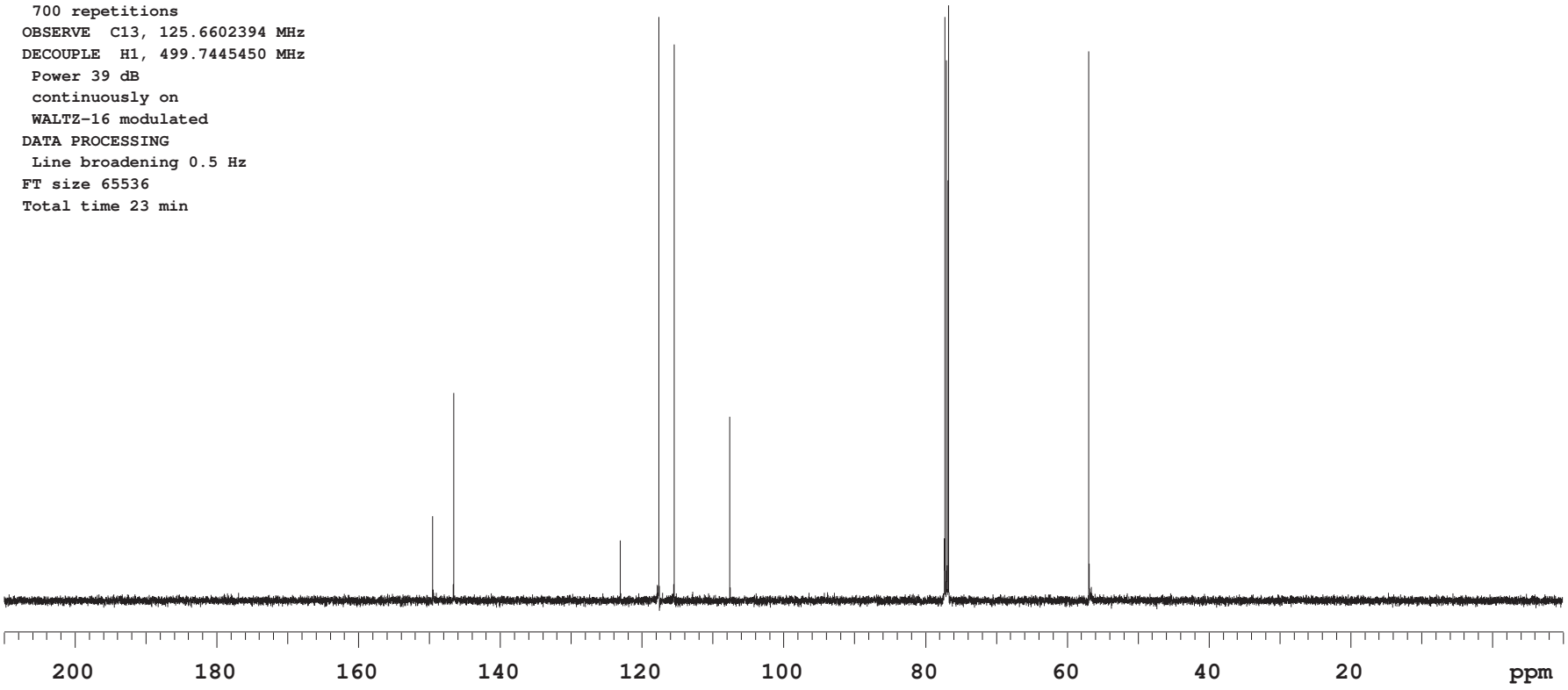
Pulse Sequence: CARBON (s2pul)
Solvent: cdcl3
Data collected on: Sep 23 2010

Temp. 25.0 C / 298.1 K
Sample #41, Operator: navarro

Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.042 sec
Width 31446.5 Hz
700 repetitions
OBSERVE C13, 125.6602394 MHz
DECOUPLE H1, 499.7445450 MHz
Power 39 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 0.5 Hz
FT size 65536
Total time 23 min



S3b



RN-II-282
purified

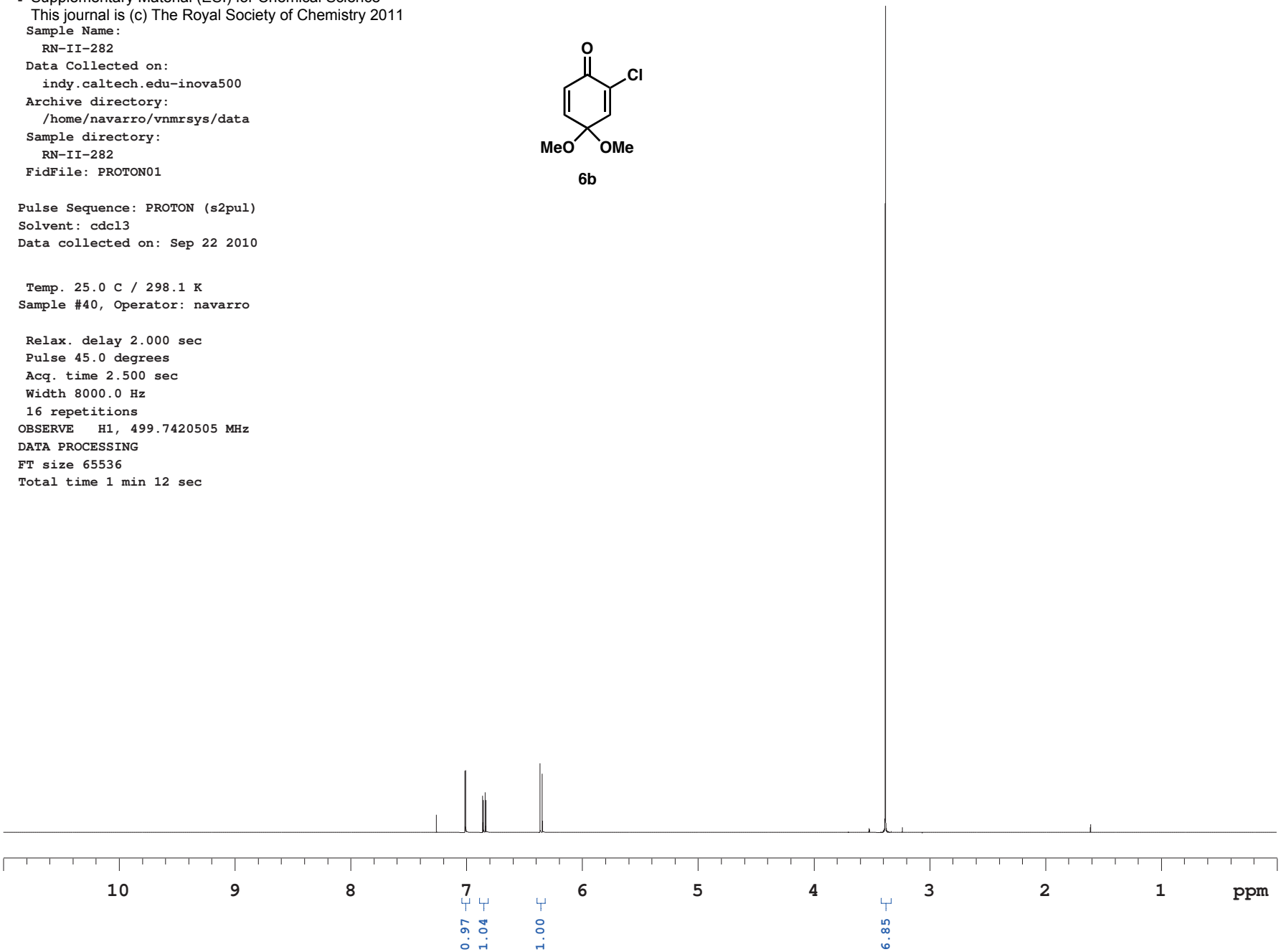
Sample Name:
RN-II-282
Data Collected on:
indy.caltech.edu-inova500
Archive directory:
/home/navarro/vnmrsys/data
Sample directory:
RN-II-282
FidFile: PROTON01

Pulse Sequence: PROTON (s2pul)
Solvent: cdcl3
Data collected on: Sep 22 2010

Temp. 25.0 C / 298.1 K
Sample #40, Operator: navarro

Relax. delay 2.000 sec
Pulse 45.0 degrees
Acq. time 2.500 sec
Width 8000.0 Hz
16 repetitions
OBSERVE H1, 499.7420505 MHz
DATA PROCESSING
FT size 65536
Total time 1 min 12 sec

6b

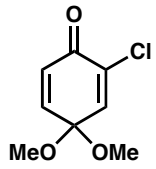


Sample Name:
RN-II-282-Cl
Data Collected on:
indy.caltech.edu-inova500
Archive directory:
/home/navarro/vnmrsys/data
Sample directory:
RN-II-282-Cl
FidFile: RN-II-282-Cl-13C

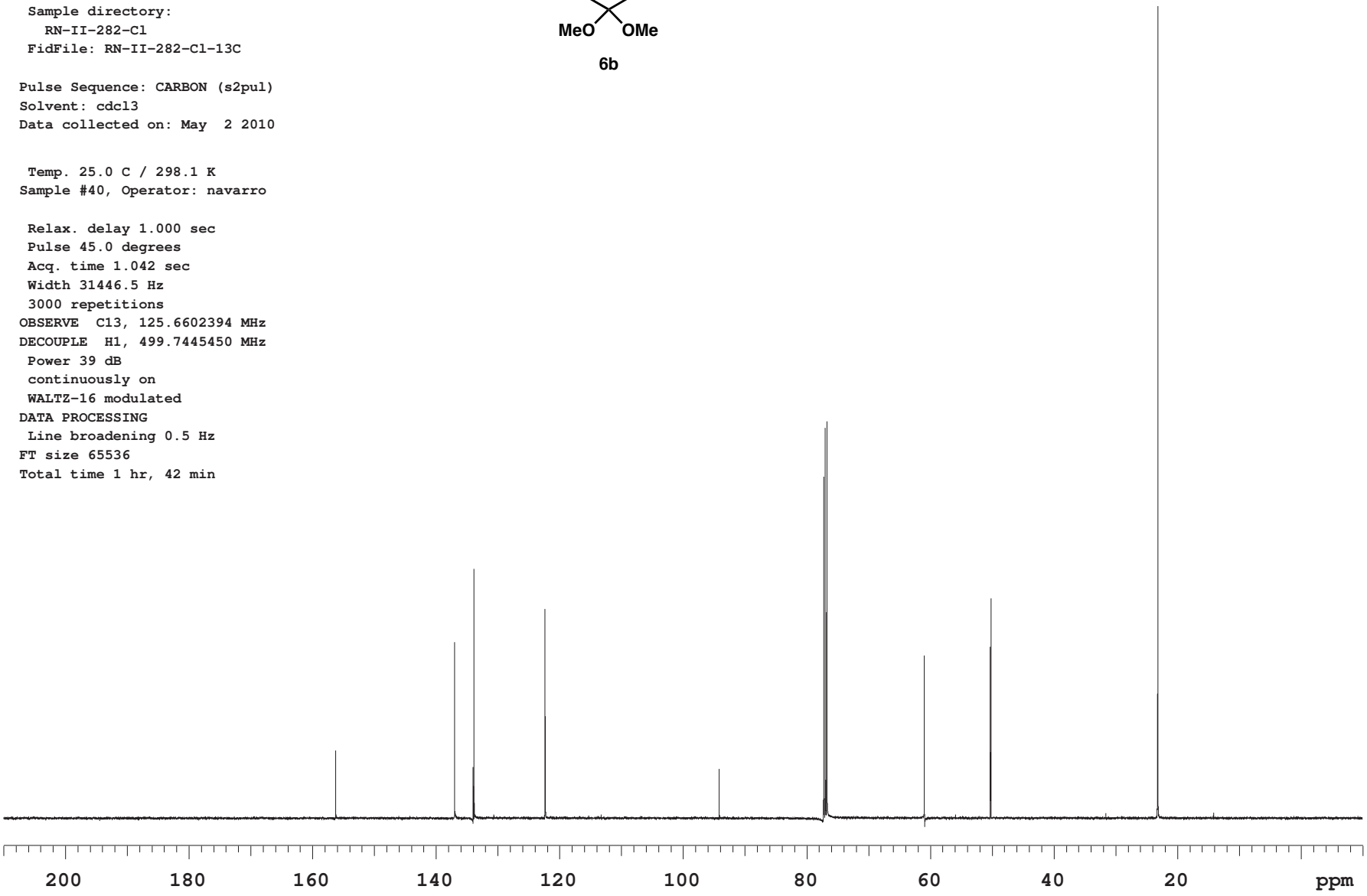
Pulse Sequence: CARBON (s2pul)
Solvent: cdcl3
Data collected on: May 2 2010

Temp. 25.0 C / 298.1 K
Sample #40, Operator: navarro

Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.042 sec
Width 31446.5 Hz
3000 repetitions
OBSERVE C13, 125.6602394 MHz
DECOUPLE H1, 499.7445450 MHz
Power 39 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 0.5 Hz
FT size 65536
Total time 1 hr, 42 min



6b



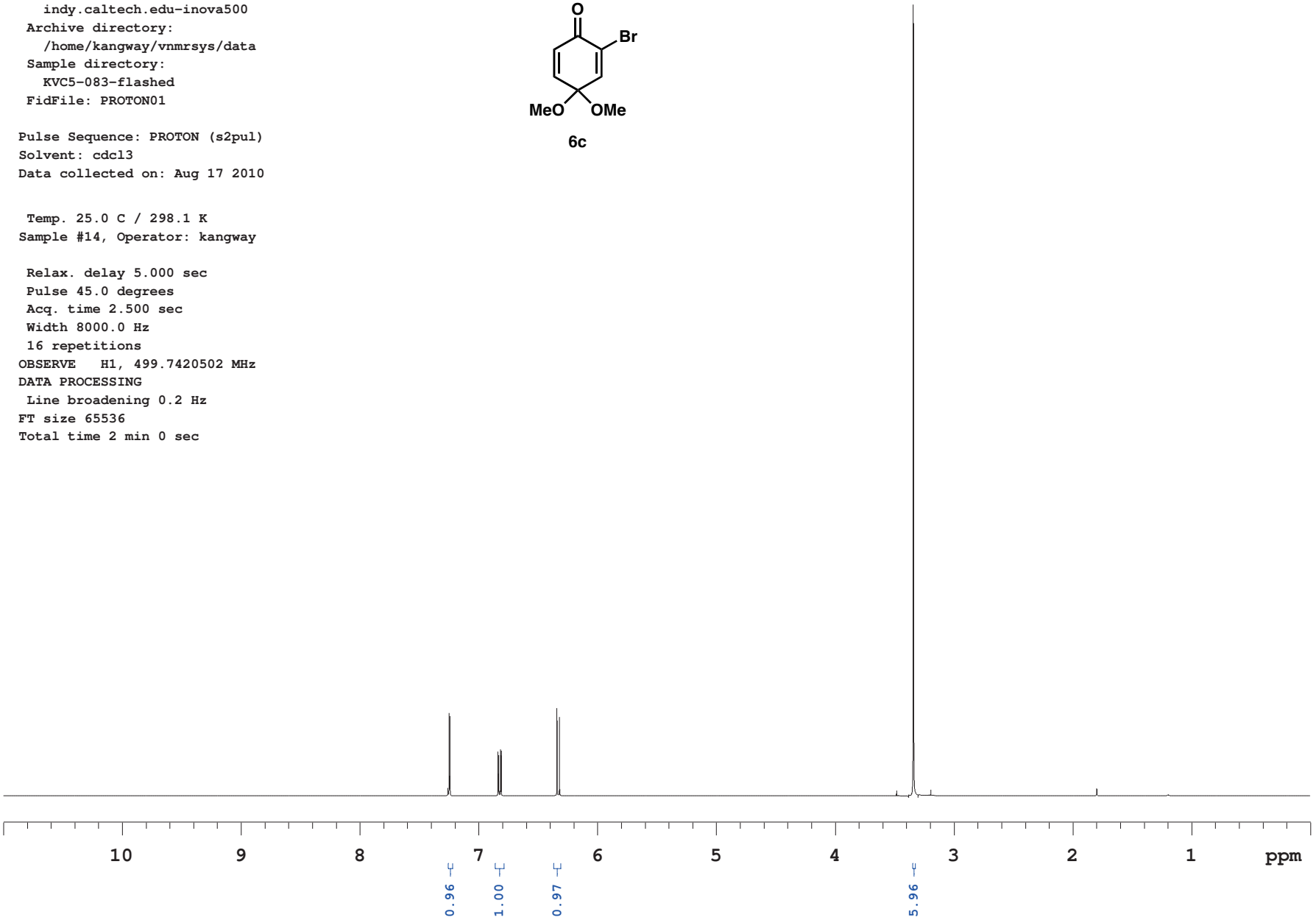
Sample Name:
KVC5-083-flashed
Data Collected on:
indy.caltech.edu-inova500
Archive directory:
/home/kangway/vnmrsys/data
Sample directory:
KVC5-083-flashed
FidFile: PROTON01

Pulse Sequence: PROTON (s2pul)
Solvent: cdcl3
Data collected on: Aug 17 2010

Temp. 25.0 C / 298.1 K
Sample #14, Operator: kangway

Relax. delay 5.000 sec
Pulse 45.0 degrees
Acq. time 2.500 sec
Width 8000.0 Hz
16 repetitions
OBSERVE H1, 499.7420502 MHz
DATA PROCESSING
Line broadening 0.2 Hz
FT size 65536
Total time 2 min 0 sec

6c

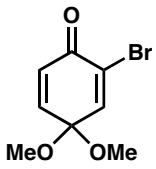


Sample Name:
KVC5-083-flashed
Data Collected on:
indy.caltech.edu-inova500
Archive directory:
/home/kangway/vnmrsys/data
Sample directory:
KVC5-083-flashed
FidFile: CARBON01

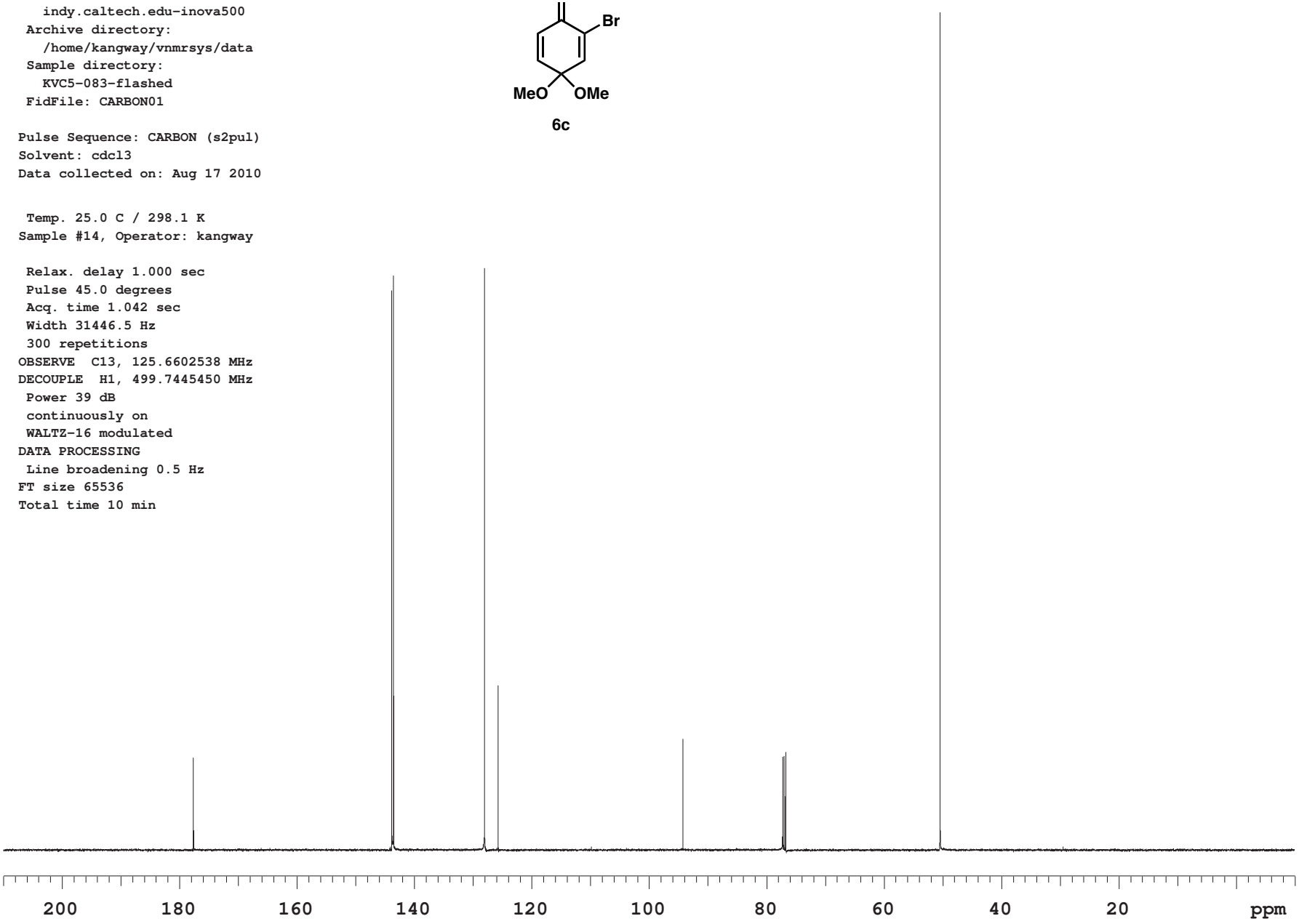
Pulse Sequence: CARBON (s2pul)
Solvent: cdcl3
Data collected on: Aug 17 2010

Temp. 25.0 C / 298.1 K
Sample #14, Operator: kangway

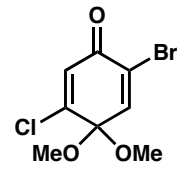
Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.042 sec
Width 31446.5 Hz
300 repetitions
OBSERVE C13, 125.6602538 MHz
DECOUPLE H1, 499.7445450 MHz
Power 39 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 0.5 Hz
FT size 65536
Total time 10 min



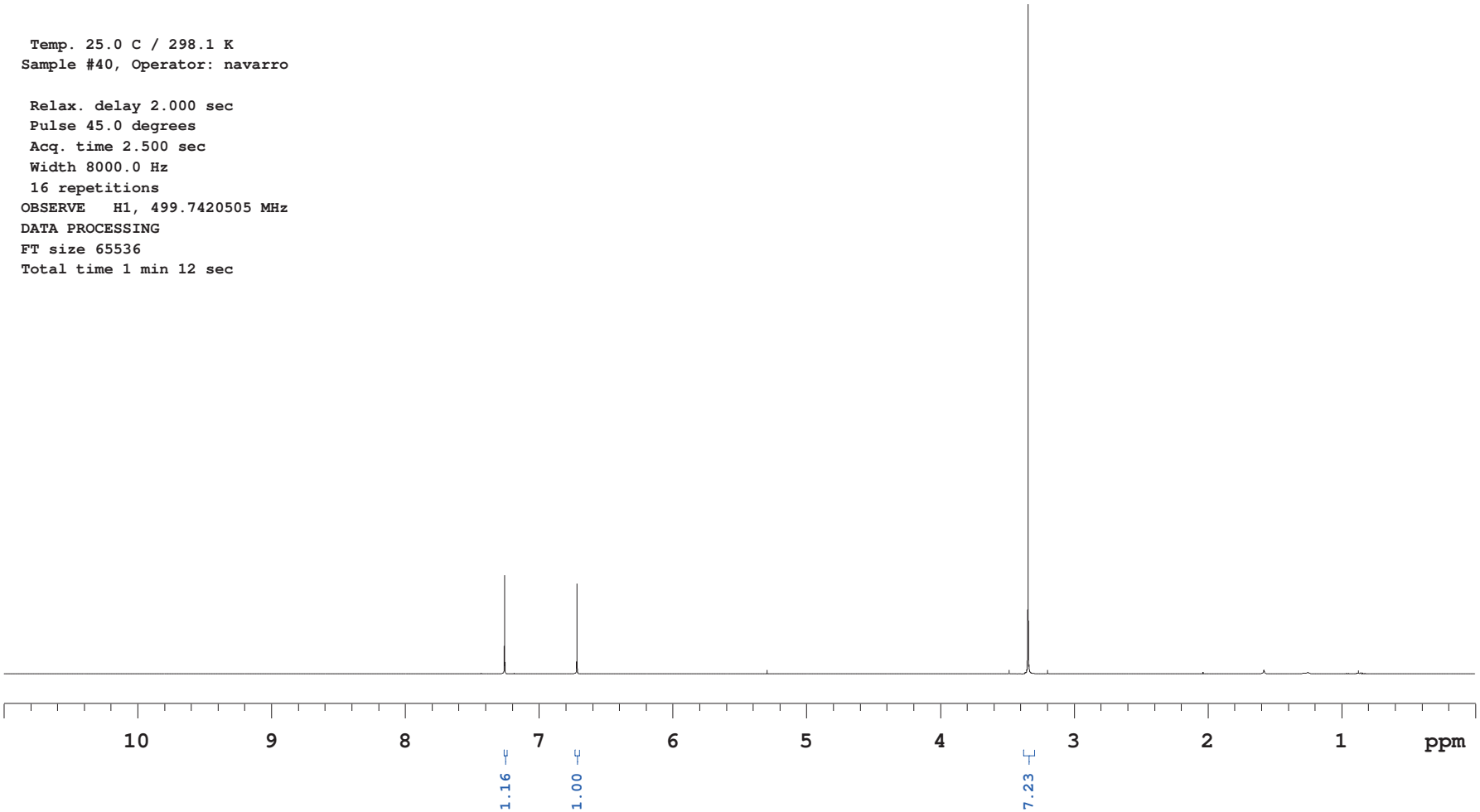
6c



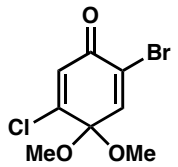
```
 Sample Name:
   RN-III-218
 Data Collected on:
   indy.caltech.edu-inova500
 Archive directory:
   /home/navarro/vnmrsys/data
 Sample directory:
   RN-III-218
 FidFile: PROTON01

Pulse Sequence: PROTON (s2pul)
Solvent: cdcl3
Data collected on: Oct  6 2010


 Temp. 25.0 C / 298.1 K
Sample #40, Operator: navarro

 Relax. delay 2.000 sec
 Pulse 45.0 degrees
 Acq. time 2.500 sec
 Width 8000.0 Hz
 16 repetitions
OBSERVE   H1, 499.7420505 MHz
DATA PROCESSING
FT size 65536
Total time 1 min 12 sec
```



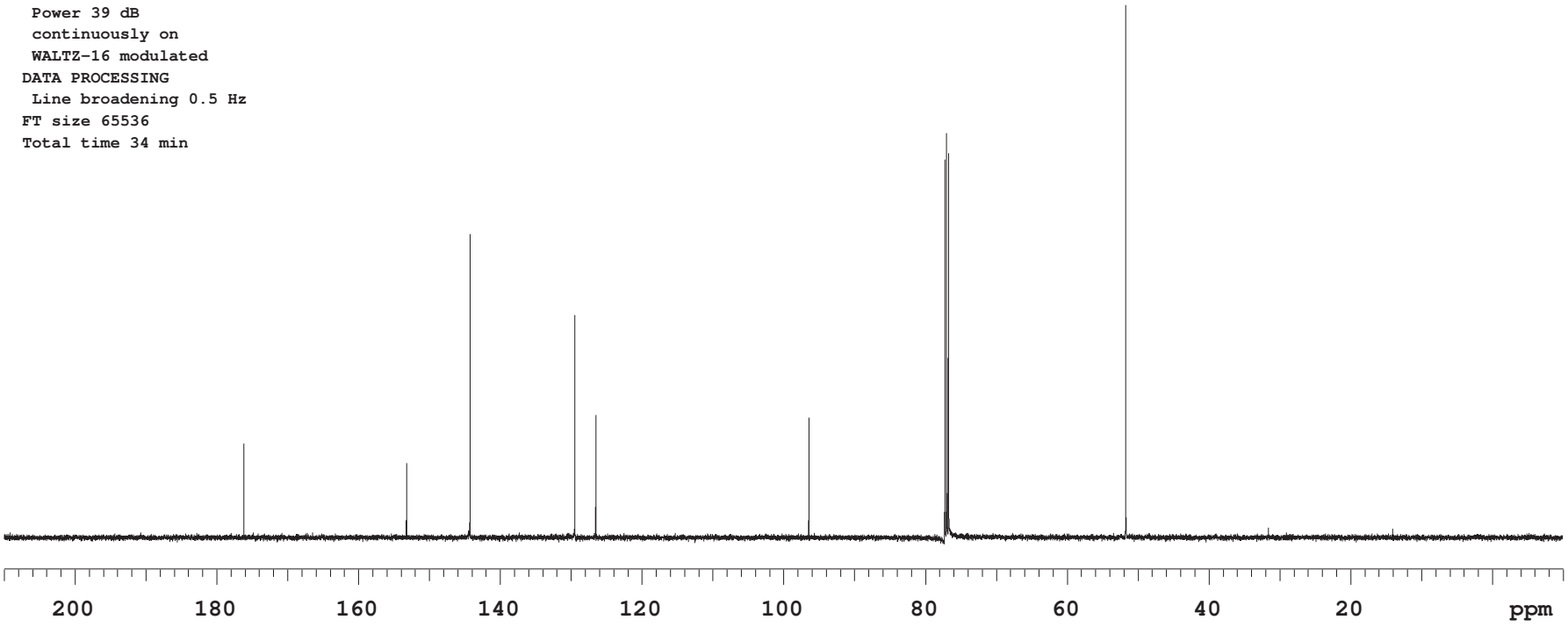
6d



```
 Sample Name:
   RN-III-218
 Data Collected on:
   indy.caltech.edu-inova500
 Archive directory:
   /home/navarro/vnmrsys/data
 Sample directory:
   RN-III-218
 FidFile: CARBON01

Pulse Sequence: CARBON (s2pul)
Solvent: cdcl3
Data collected on: Oct  6 2010


 Temp. 25.0 C / 298.1 K
Sample #40, Operator: navarro

 Relax. delay 1.000 sec
 Pulse 45.0 degrees
 Acq. time 1.042 sec
 Width 31446.5 Hz
 1000 repetitions
OBSERVE  C13, 125.6602404 MHz
DECOUPLE  H1, 499.7445450 MHz
 Power 39 dB
 continuously on
 WALTZ-16 modulated
DATA PROCESSING
 Line broadening 0.5 Hz
FT size 65536
Total time 34 min
```



6d



RN-III-226
purified

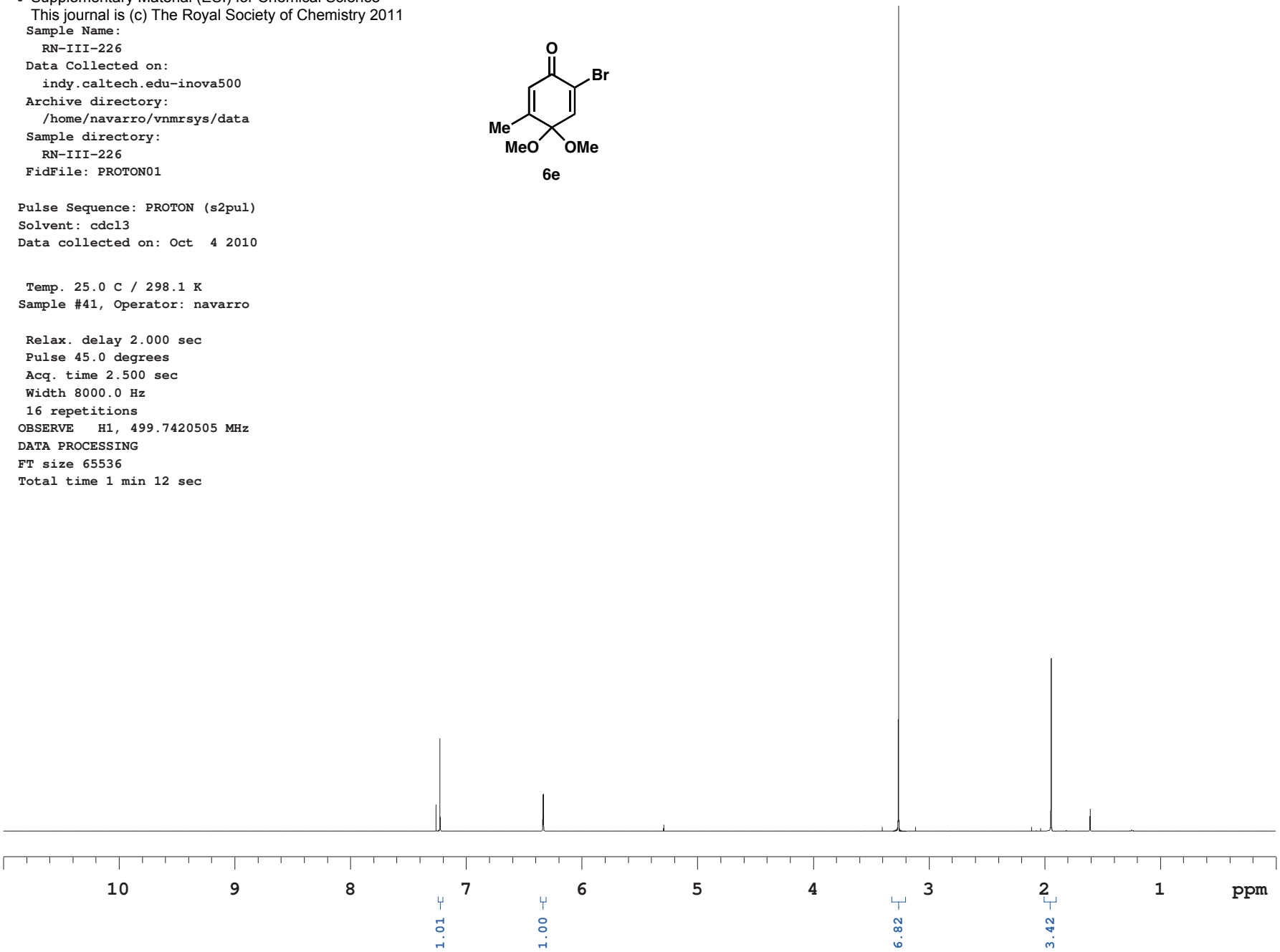
Sample Name:
RN-III-226
Data Collected on:
indy.caltech.edu-inova500
Archive directory:
/home/navarro/vnmrsys/data
Sample directory:
RN-III-226
FidFile: PROTON01

Pulse Sequence: PROTON (s2pul)
Solvent: cdcl3
Data collected on: Oct 4 2010

Temp. 25.0 C / 298.1 K
Sample #41, Operator: navarro

Relax. delay 2.000 sec
Pulse 45.0 degrees
Acq. time 2.500 sec
Width 8000.0 Hz
16 repetitions
OBSERVE H1, 499.7420505 MHz
DATA PROCESSING
FT size 65536
Total time 1 min 12 sec

6e

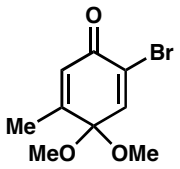


Sample Name:
RN-III-226
Data Collected on:
indy.caltech.edu-inova500
Archive directory:
/home/navarro/vnmrsys/data
Sample directory:
RN-III-226
FidFile: CARBON01

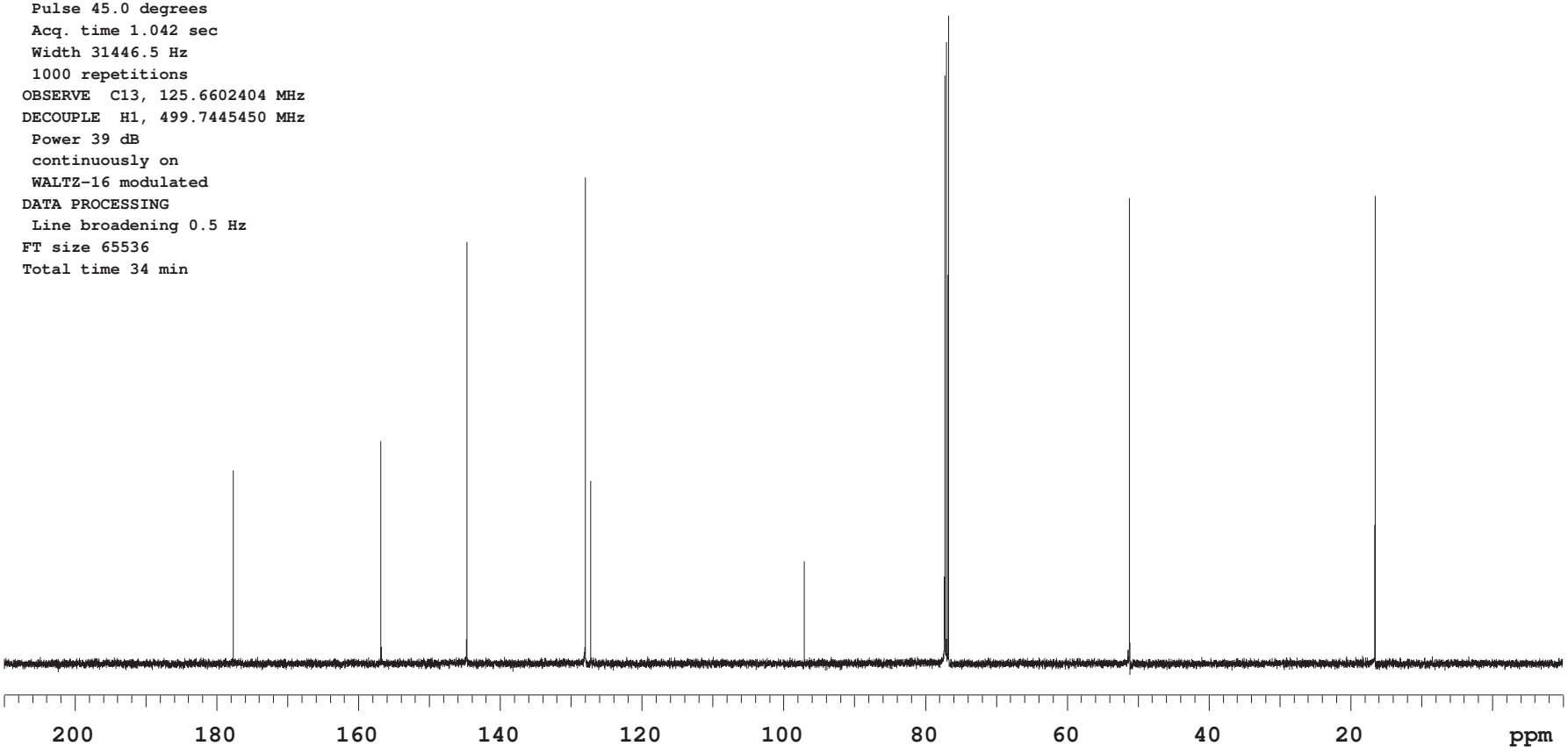
Pulse Sequence: CARBON (s2pul)
Solvent: cdcl3
Data collected on: Oct 4 2010

Temp. 25.0 C / 298.1 K
Sample #41, Operator: navarro

Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.042 sec
Width 31446.5 Hz
1000 repetitions
OBSERVE C13, 125.6602404 MHz
DECOUPLE H1, 499.7445450 MHz
Power 39 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 0.5 Hz
FT size 65536
Total time 34 min



6e

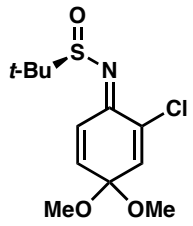


Sample Name:
RN-II-282-Cl
Data Collected on:
indy.caltech.edu-inova500
Archive directory:
/home/navarro/vnmrsys/data
Sample directory:
RN-II-282-Cl
FidFile: RN-II-282-Cl-1H

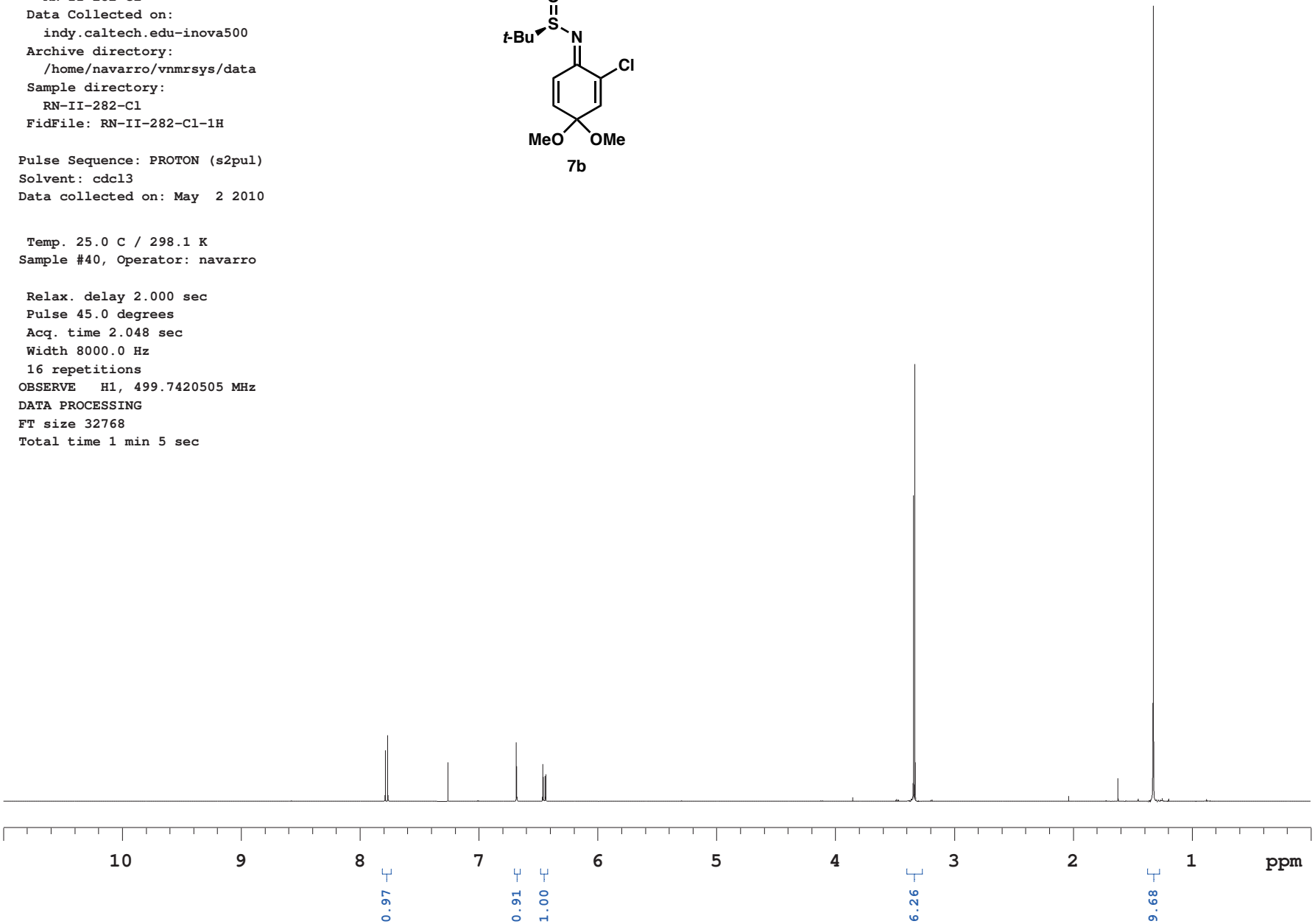
Pulse Sequence: PROTON (s2pul)
Solvent: cdcl3
Data collected on: May 2 2010

Temp. 25.0 C / 298.1 K
Sample #40, Operator: navarro

Relax. delay 2.000 sec
Pulse 45.0 degrees
Acq. time 2.048 sec
Width 8000.0 Hz
16 repetitions
OBSERVE H1, 499.7420505 MHz
DATA PROCESSING
FT size 32768
Total time 1 min 5 sec



7b

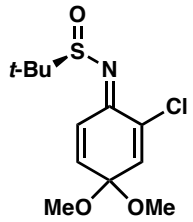


Sample Name:
RN-II-282-Cl
Data Collected on:
indy.caltech.edu-inova500
Archive directory:
/home/navarro/vnmrsys/data
Sample directory:
RN-II-282-Cl
FidFile: RN-II-282-Cl-13C

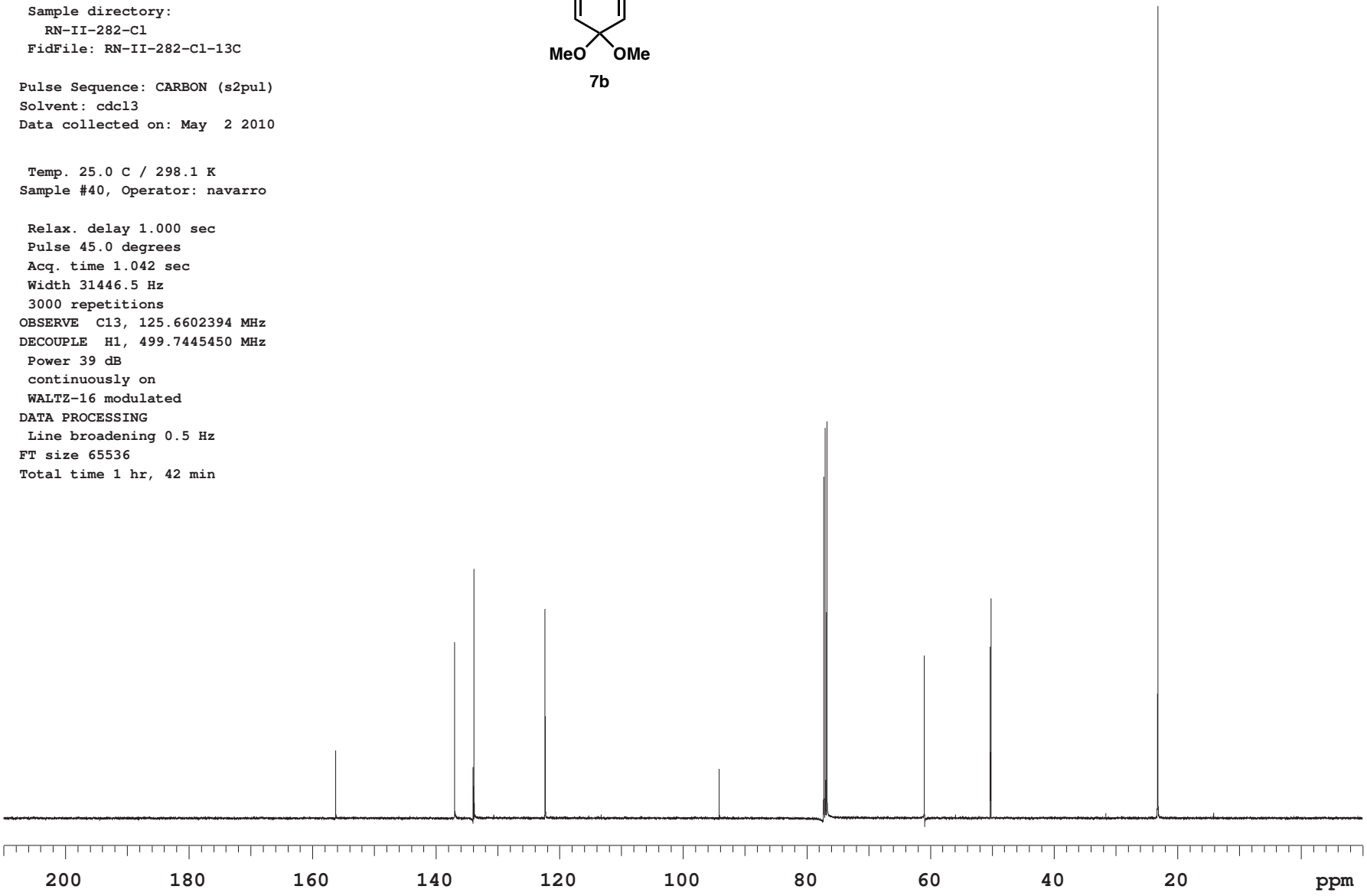
Pulse Sequence: CARBON (s2pul)
Solvent: cdcl3
Data collected on: May 2 2010

Temp. 25.0 C / 298.1 K
Sample #40, Operator: navarro

Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.042 sec
Width 31446.5 Hz
3000 repetitions
OBSERVE C13, 125.6602394 MHz
DECOUPLE H1, 499.7445450 MHz
Power 39 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 0.5 Hz
FT size 65536
Total time 1 hr, 42 min

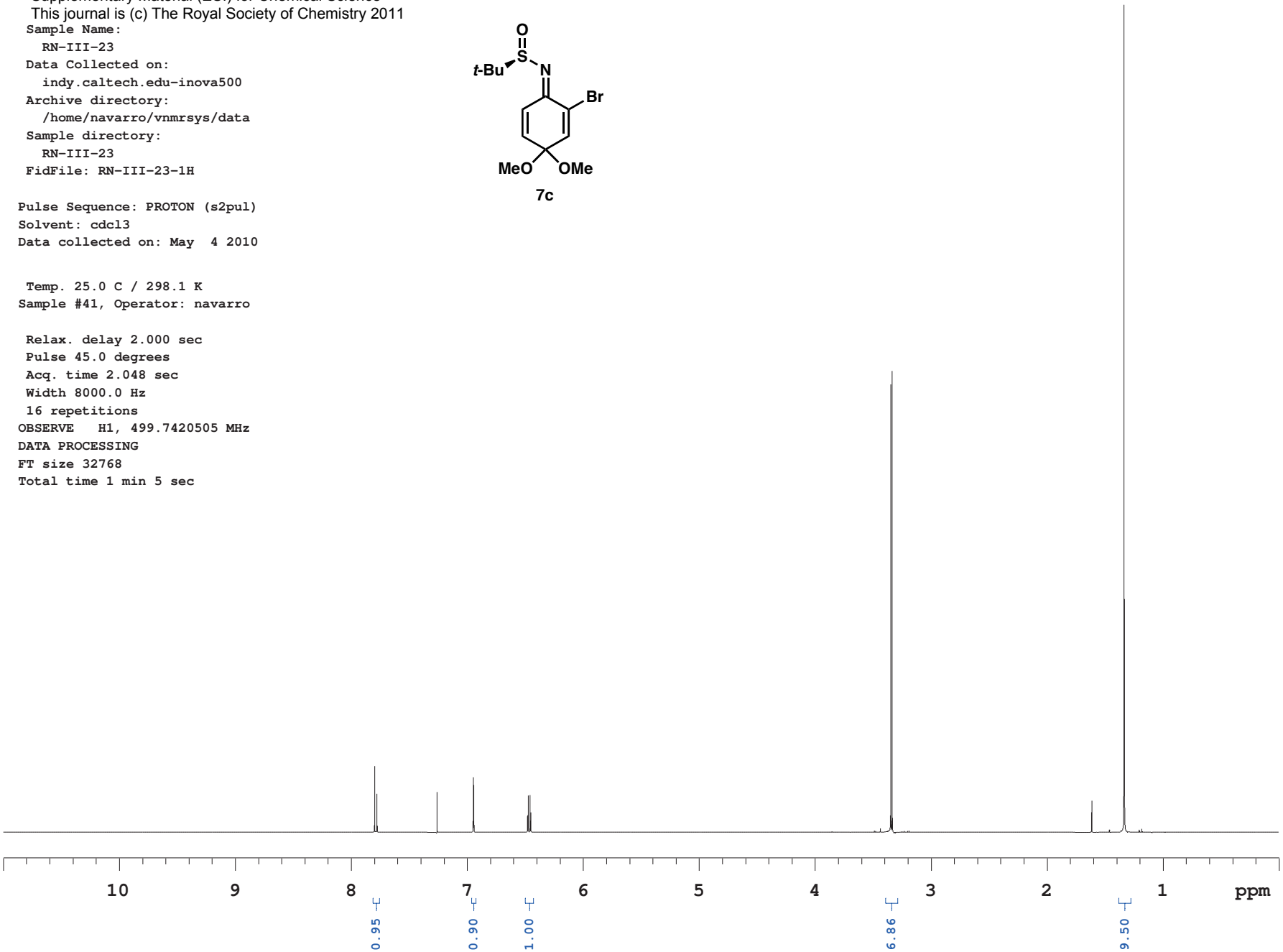


7b



```
 Sample Name:
   RN-III-23
 Data Collected on:
   indy.caltech.edu-inova500
 Archive directory:
   /home/navarro/vnmrsys/data
 Sample directory:
   RN-III-23
 FidFile: RN-III-23-1H

Pulse Sequence: PROTON (s2pul)
Solvent: cdcl3
Data collected on: May  4 2010


 Temp. 25.0 C / 298.1 K
Sample #41, Operator: navarro

 Relax. delay 2.000 sec
 Pulse 45.0 degrees
 Acq. time 2.048 sec
 Width 8000.0 Hz
 16 repetitions
OBSERVE   H1, 499.7420505 MHz
DATA PROCESSING
FT size 32768
Total time 1 min 5 sec
```


7c



RN-III-23
purified

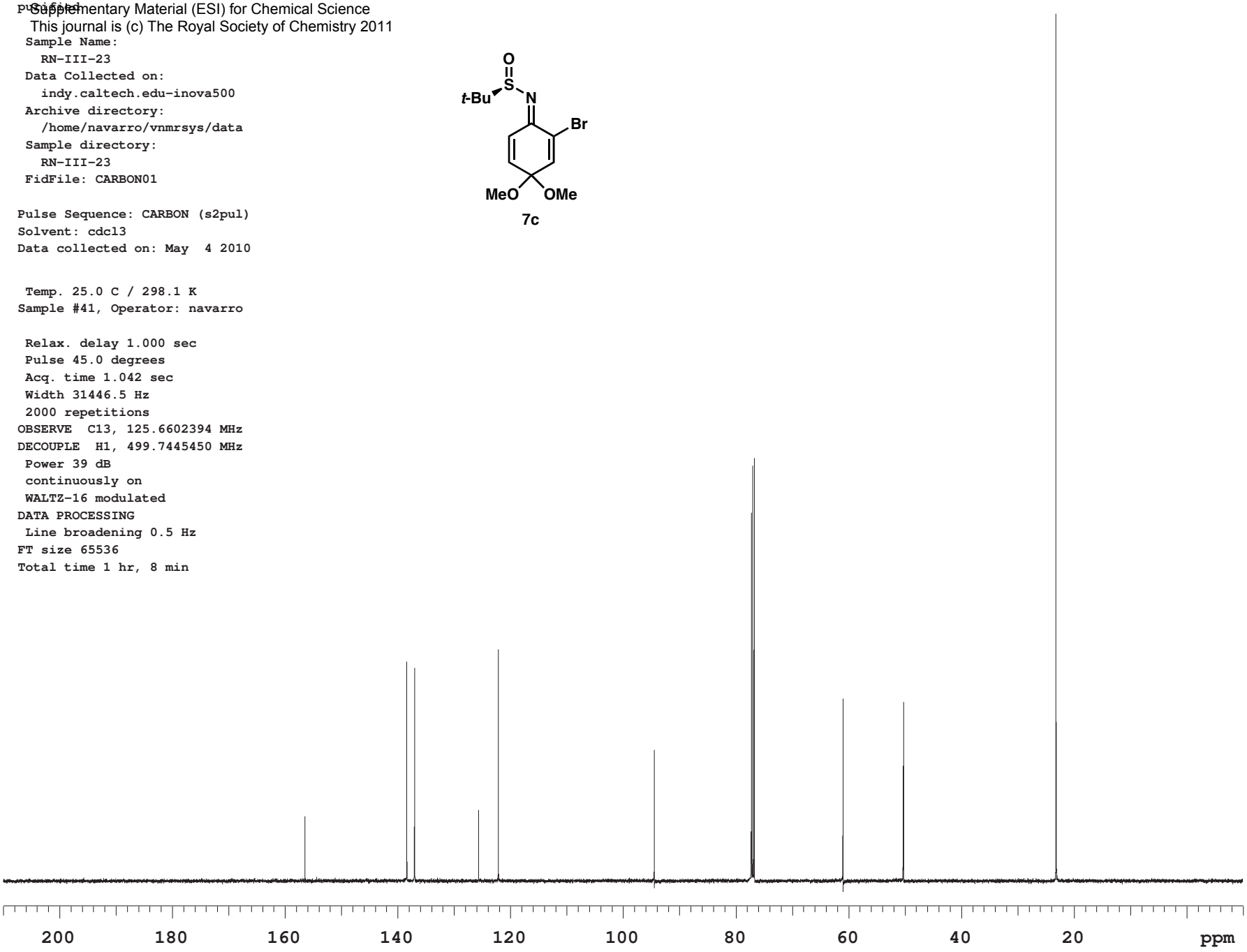
Sample Name:
RN-III-23
Data Collected on:
indy.caltech.edu-inova500
Archive directory:
/home/navarro/vnmrsys/data
Sample directory:
RN-III-23
FidFile: CARBON01

Pulse Sequence: CARBON (s2pul)
Solvent: cdcl3
Data collected on: May 4 2010

Temp. 25.0 C / 298.1 K
Sample #41, Operator: navarro

Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.042 sec
Width 31446.5 Hz
2000 repetitions
OBSERVE C13, 125.6602394 MHz
DECOUPLE H1, 499.7445450 MHz
Power 39 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 0.5 Hz
FT size 65536
Total time 1 hr, 8 min

7c



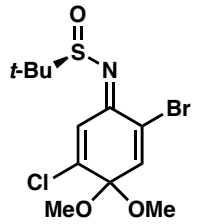
RN-III-219
purified

Sample Name:
RN-III-219
Data Collected on:
indy.caltech.edu-inova500
Archive directory:
/home/navarro/vnmrsys/data
Sample directory:
RN-III-219
FidFile: PROTON01

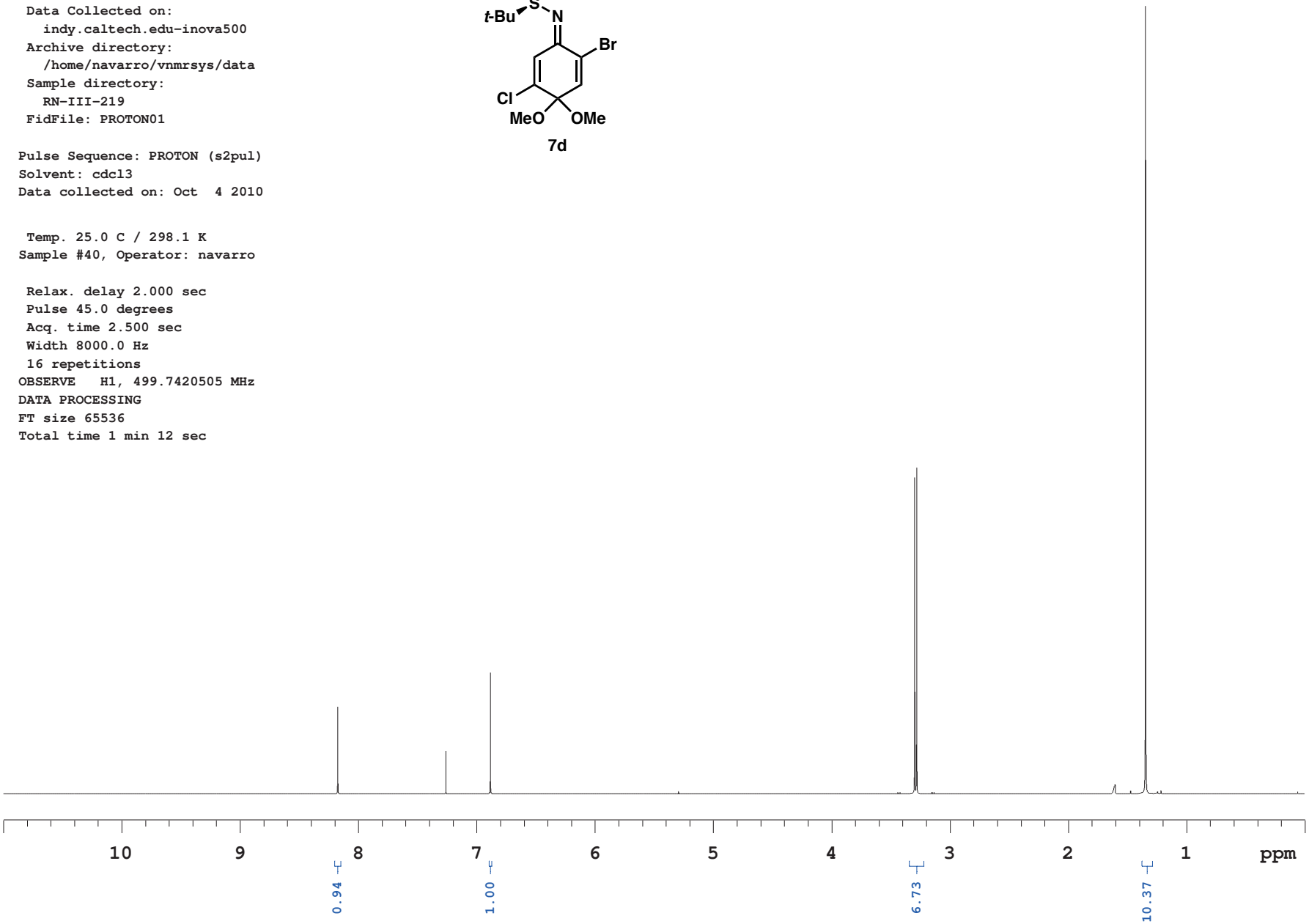
Pulse Sequence: PROTON (s2pul)
Solvent: cdcl3
Data collected on: Oct 4 2010

Temp. 25.0 C / 298.1 K
Sample #40, Operator: navarro

Relax. delay 2.000 sec
Pulse 45.0 degrees
Acq. time 2.500 sec
Width 8000.0 Hz
16 repetitions
OBSERVE H1, 499.7420505 MHz
DATA PROCESSING
FT size 65536
Total time 1 min 12 sec



7d



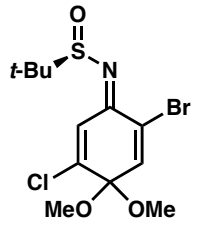
RN-III-219
purified

Sample Name:
RN-III-219
Data Collected on:
indy.caltech.edu-inova500
Archive directory:
/home/navarro/vnmrsys/data
Sample directory:
RN-III-219
FidFile: CARBON01

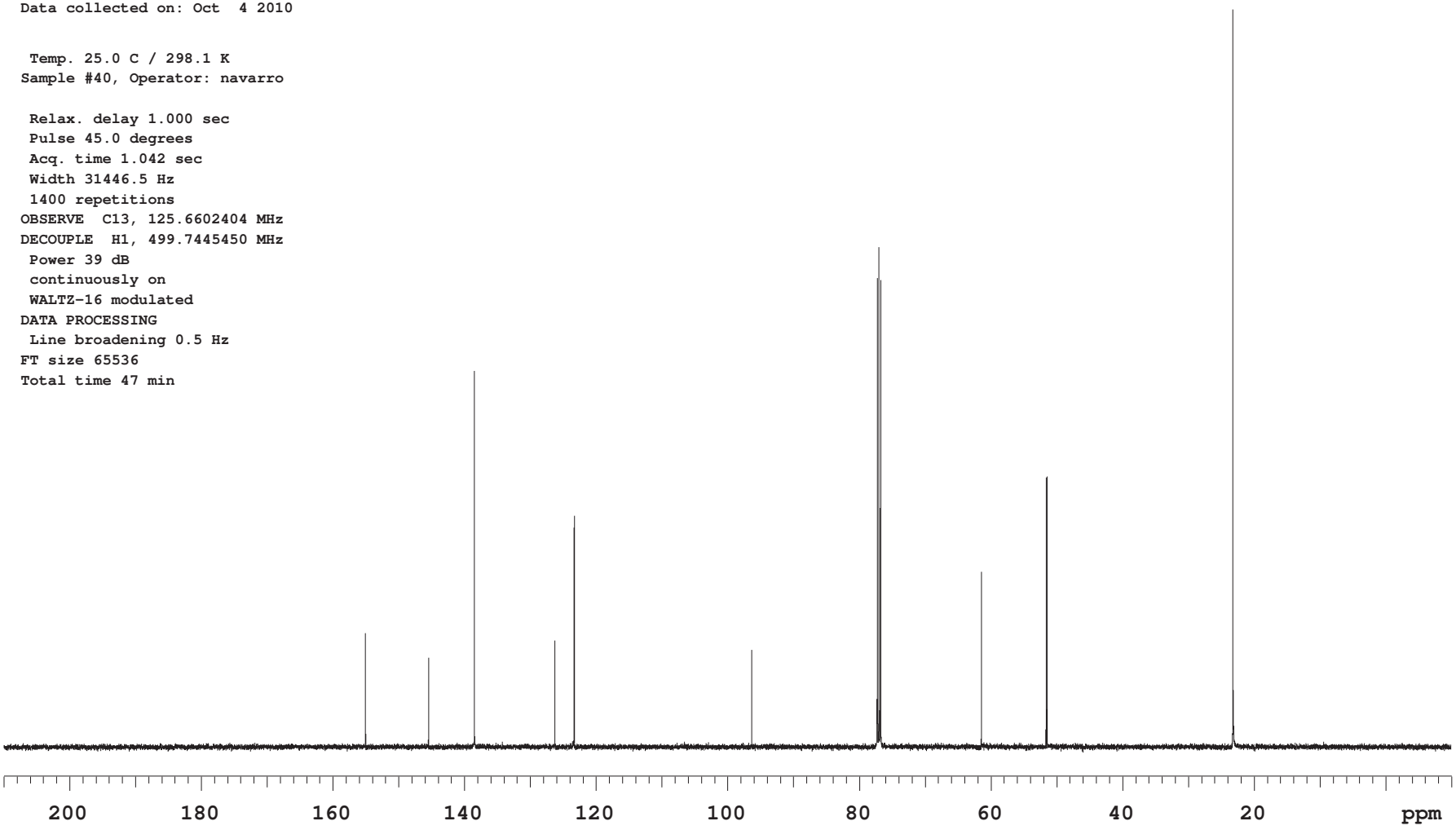
Pulse Sequence: CARBON (s2pul)
Solvent: cdcl3
Data collected on: Oct 4 2010

Temp. 25.0 C / 298.1 K
Sample #40, Operator: navarro

Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.042 sec
Width 31446.5 Hz
1400 repetitions
OBSERVE C13, 125.6602404 MHz
DECOUPLE H1, 499.7445450 MHz
Power 39 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 0.5 Hz
FT size 65536
Total time 47 min



7d



RN-III-208
purified

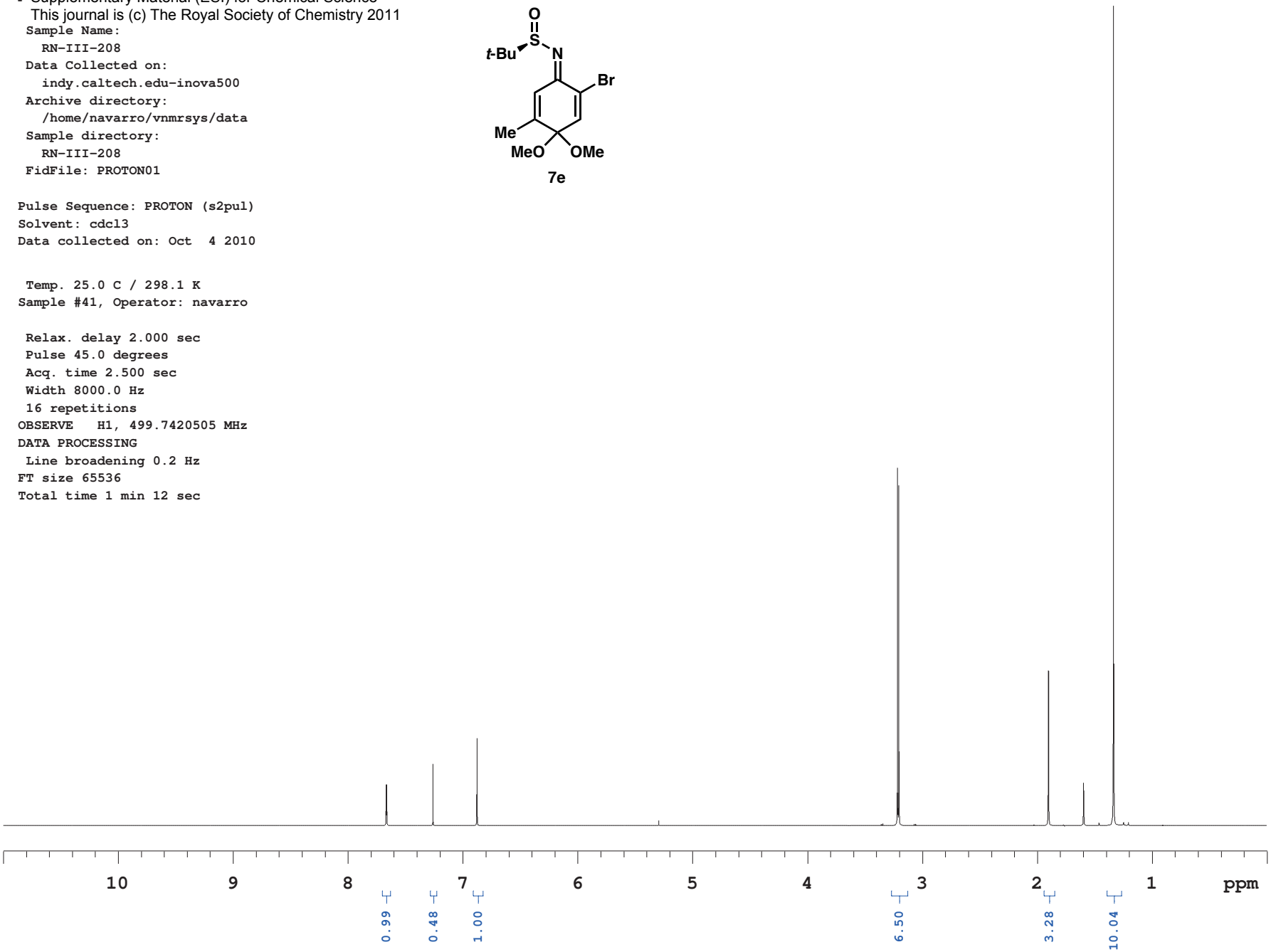
Sample Name:
RN-III-208
Data Collected on:
indy.caltech.edu-inova500
Archive directory:
/home/navarro/vnmrsys/data
Sample directory:
RN-III-208
FidFile: PROTON01

Pulse Sequence: PROTON (s2pul)
Solvent: cdcl3
Data collected on: Oct 4 2010

Temp. 25.0 C / 298.1 K
Sample #41, Operator: navarro

Relax. delay 2.000 sec
Pulse 45.0 degrees
Acq. time 2.500 sec
Width 8000.0 Hz
16 repetitions
OBSERVE H1, 499.7420505 MHz
DATA PROCESSING
Line broadening 0.2 Hz
FT size 65536
Total time 1 min 12 sec

7e



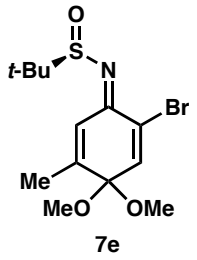
RN-III-208
purified

Sample Name:
RN-III-208
Data Collected on:
indy.caltech.edu-inova500
Archive directory:
/home/navarro/vnmrsys/data
Sample directory:
RN-III-208
FidFile: CARBON01

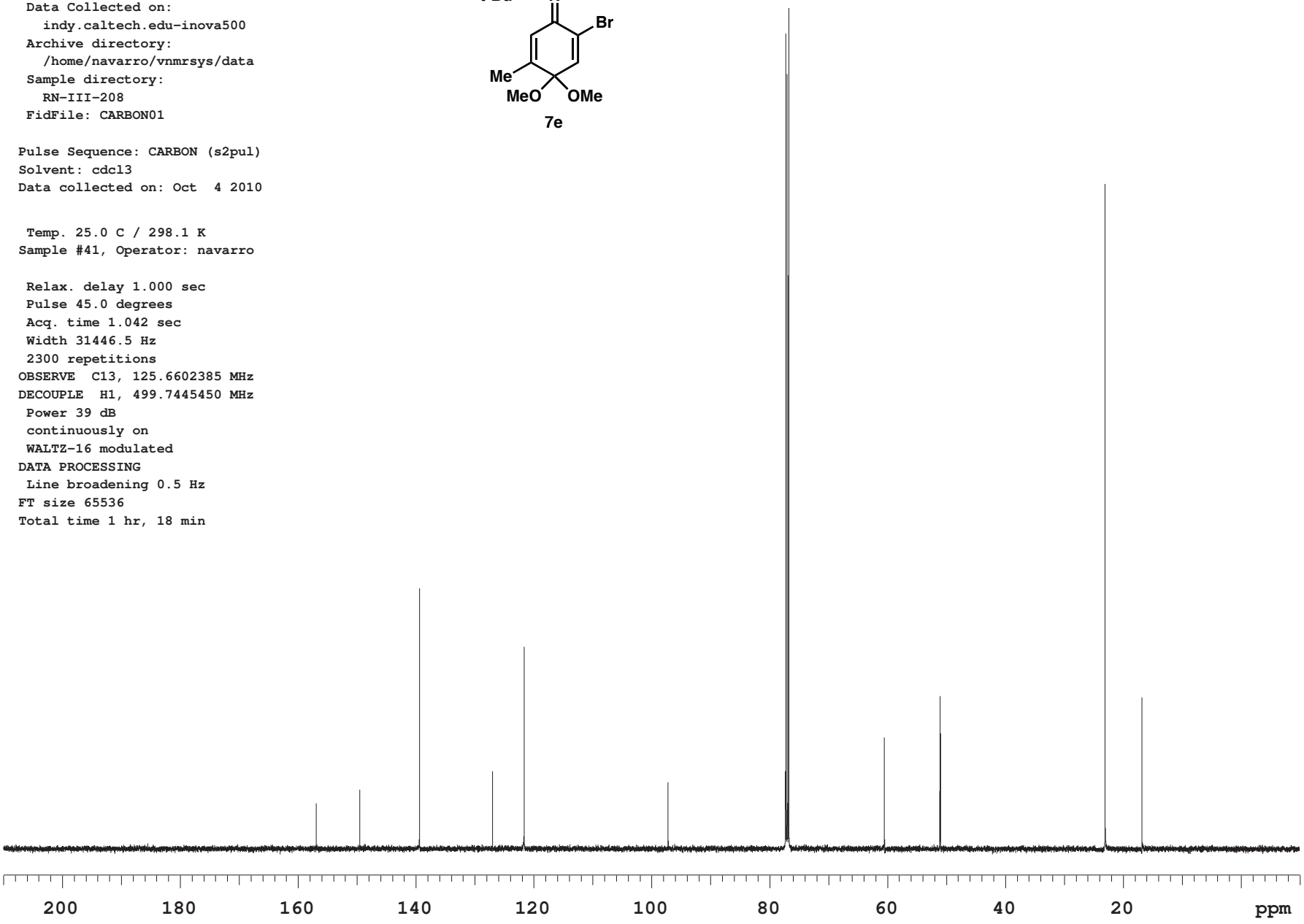
Pulse Sequence: CARBON (s2pul)
Solvent: cdcl3
Data collected on: Oct 4 2010

Temp. 25.0 C / 298.1 K
Sample #41, Operator: navarro

Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.042 sec
Width 31446.5 Hz
2300 repetitions
OBSERVE C13, 125.6602385 MHz
DECOUPLE H1, 499.7445450 MHz
Power 39 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 0.5 Hz
FT size 65536
Total time 1 hr, 18 min



7e



RN-II-287
purified

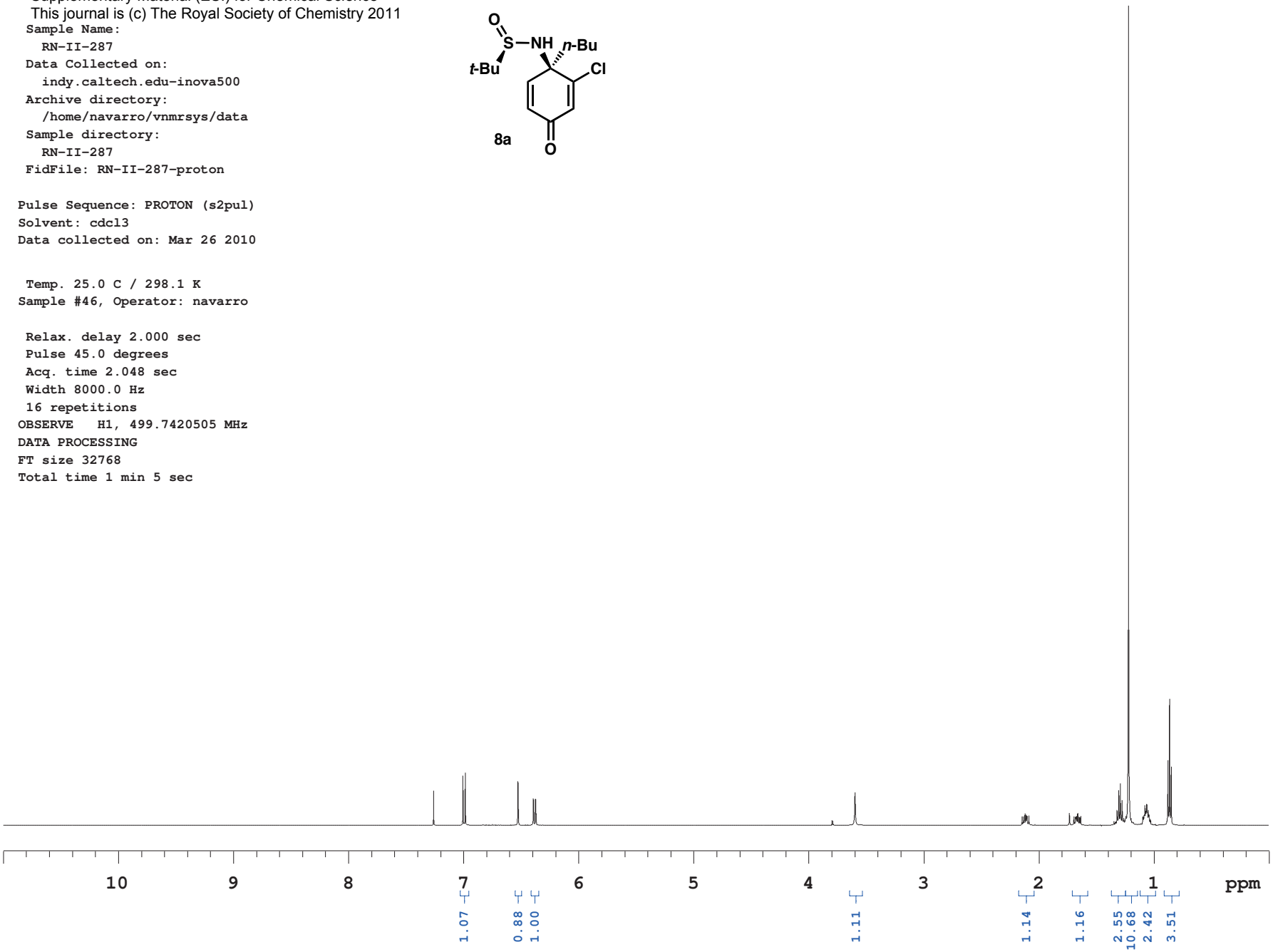
Sample Name:
RN-II-287
Data Collected on:
indy.caltech.edu-inova500
Archive directory:
/home/navarro/vnmrsys/data
Sample directory:
RN-II-287
FidFile: RN-II-287-proton

Pulse Sequence: PROTON (s2pul)
Solvent: cdcl3
Data collected on: Mar 26 2010

Temp. 25.0 C / 298.1 K
Sample #46, Operator: navarro

Relax. delay 2.000 sec
Pulse 45.0 degrees
Acq. time 2.048 sec
Width 8000.0 Hz
16 repetitions
OBSERVE H1, 499.7420505 MHz
DATA PROCESSING
FT size 32768
Total time 1 min 5 sec

8a



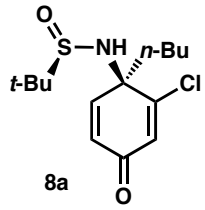
RN-II-287
purified

Sample Name:
RN-II-287
Data Collected on:
indy.caltech.edu-inova500
Archive directory:
/home/navarro/vnmrsys/data
Sample directory:
RN-II-287
FidFile: RN-II-287-carbon

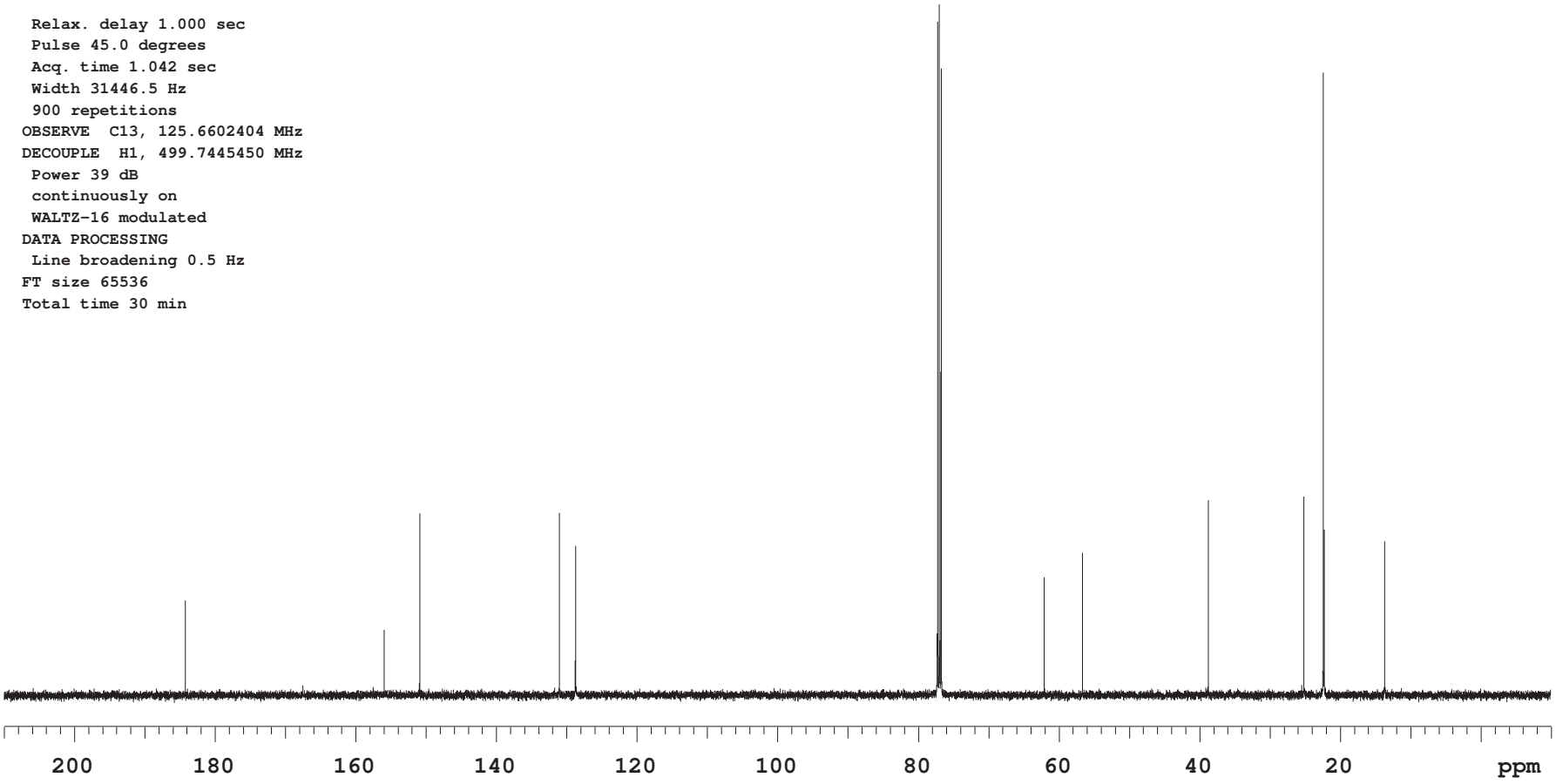
Pulse Sequence: CARBON (s2pul)
Solvent: cdcl3
Data collected on: Mar 26 2010

Temp. 25.0 C / 298.1 K
Sample #46, Operator: navarro

Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.042 sec
Width 31446.5 Hz
900 repetitions
OBSERVE C13, 125.6602404 MHz
DECOUPLE H1, 499.7445450 MHz
Power 39 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 0.5 Hz
FT size 65536
Total time 30 min



8a



RN-II-292
major products
Sample Name:
RN-II-292-A
Data Collected on:
indy.caltech.edu-inova500
Archive directory:
/home/navarro/vnmrsys/data
Sample directory:
RN-II-292-A
FidFile: RN-II-292-major-1H

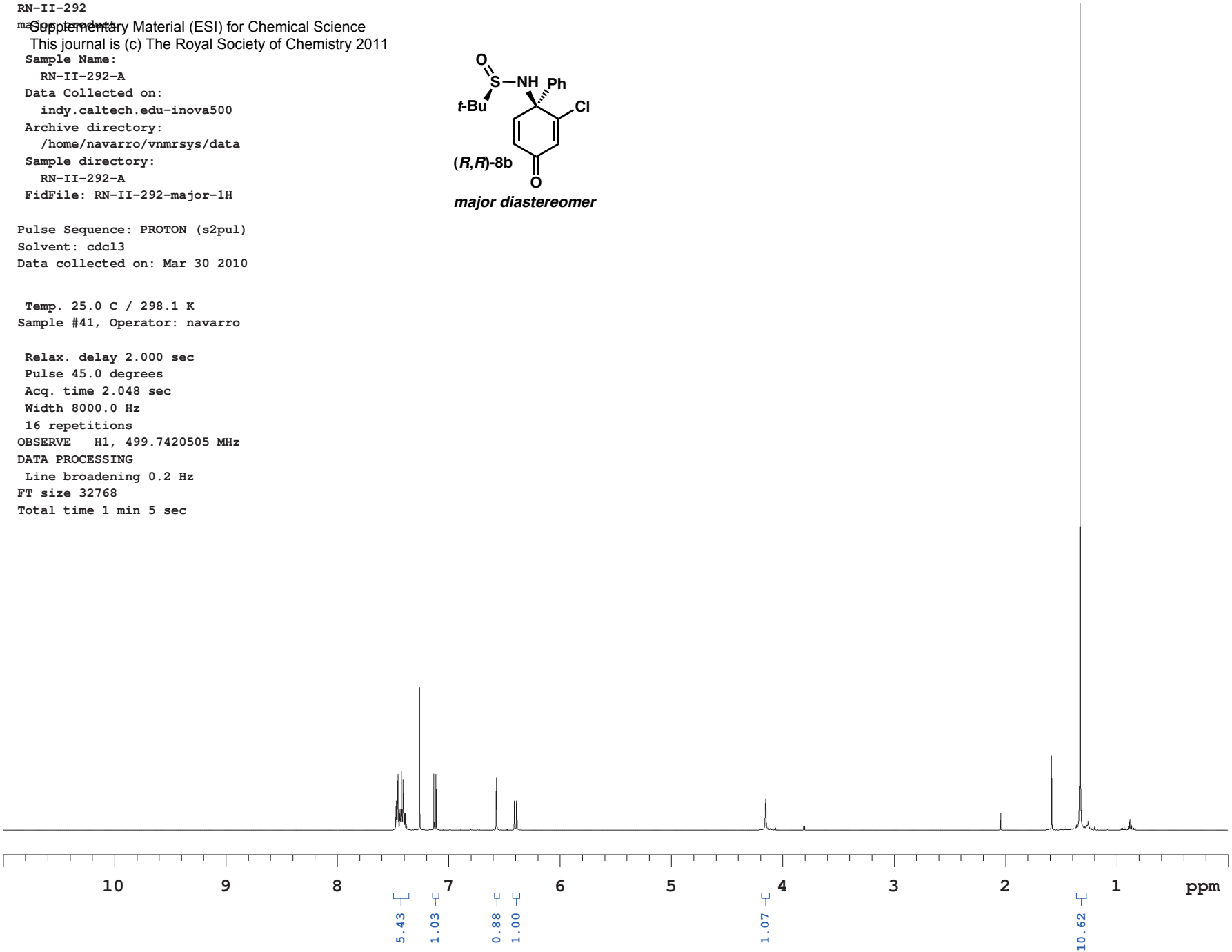
Pulse Sequence: PROTON (s2pul)
Solvent: cdcl3
Data collected on: Mar 30 2010

Temp. 25.0 C / 298.1 K
Sample #41, Operator: navarro

Relax. delay 2.000 sec
Pulse 45.0 degrees
Acq. time 2.048 sec
Width 8000.0 Hz
16 repetitions
OBSERVE H1, 499.7420505 MHz
DATA PROCESSING
Line broadening 0.2 Hz
FT size 32768
Total time 1 min 5 sec

(*R*,*R*)-8b

major diastereomer



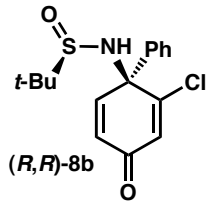
RN-III-54
purified

Sample Name:
RN-III-54
Data Collected on:
indy.caltech.edu-inova500
Archive directory:
/home/navarro/vnmrsys/data
Sample directory:
RN-III-54
FidFile: CARBON02

Pulse Sequence: CARBON (s2pul)
Solvent: cdcl3
Data collected on: Oct 20 2010

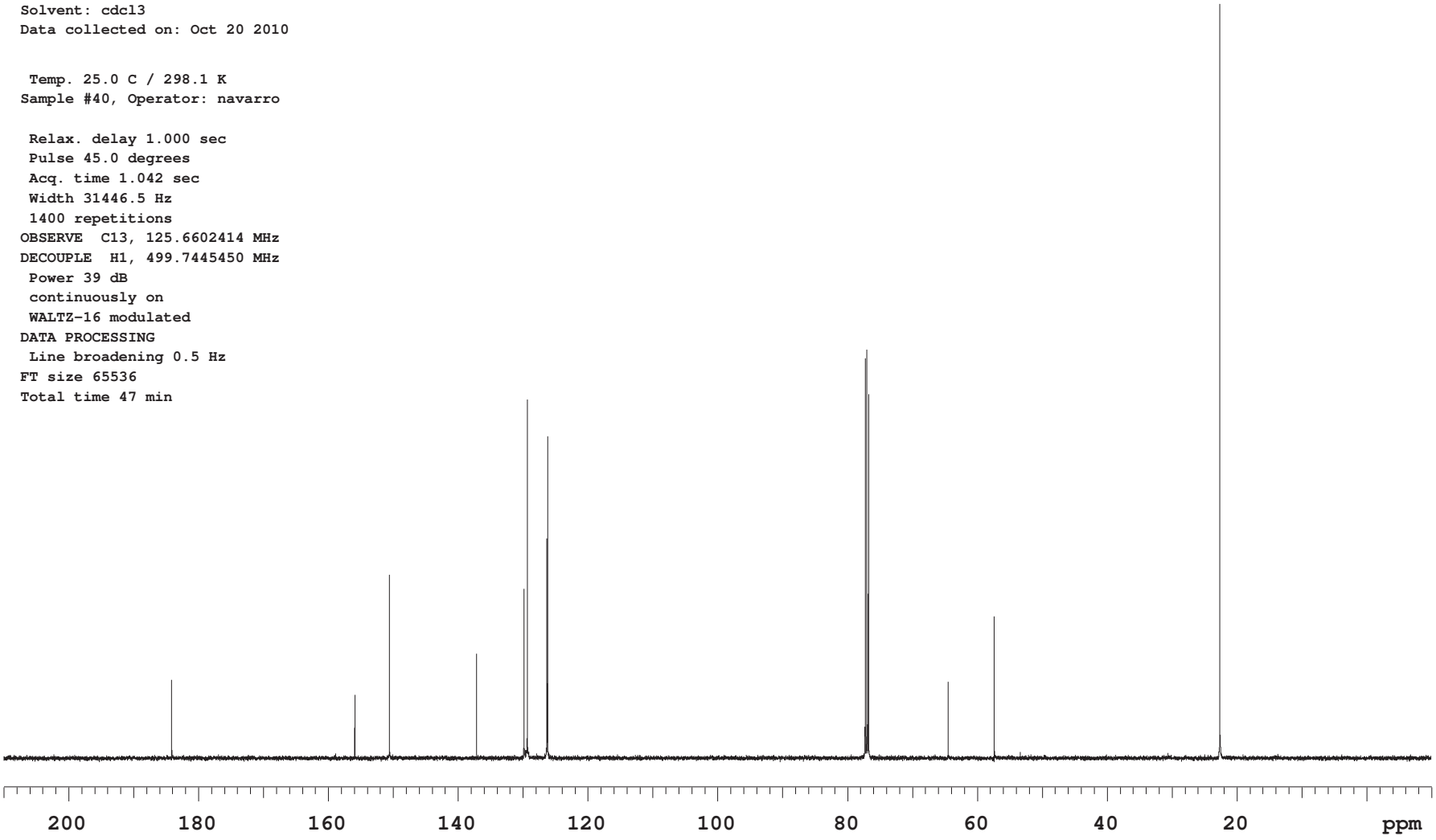
Temp. 25.0 C / 298.1 K
Sample #40, Operator: navarro

Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.042 sec
Width 31446.5 Hz
1400 repetitions
OBSERVE C13, 125.6602414 MHz
DECOUPLE H1, 499.7445450 MHz
Power 39 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 0.5 Hz
FT size 65536
Total time 47 min

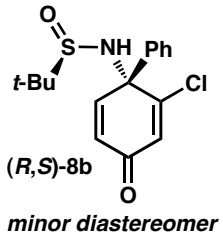


(*R*,*R*)-8b

major diastereomer

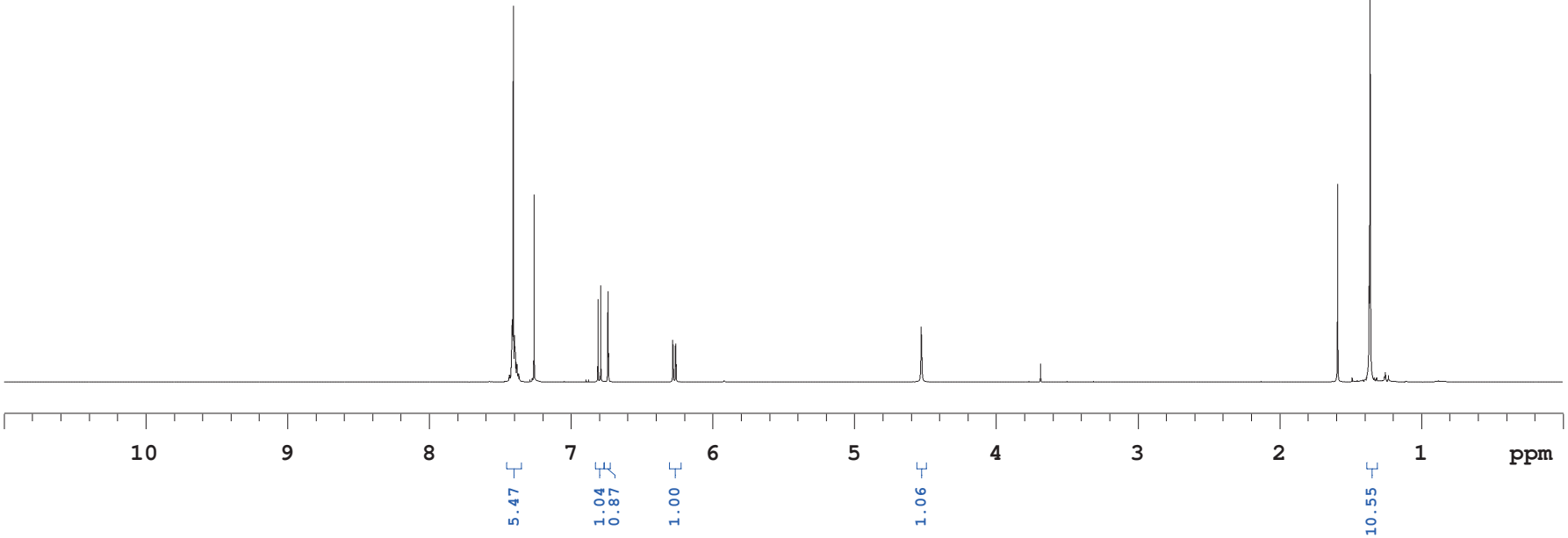


```
RN-III-54
minor
 Sample Name:
   RN-III-54-minor
 Data Collected on:
   indy.caltech.edu-inova500
 Archive directory:
   /home/navarro/vnmrsys/data
 Sample directory:
   RN-III-54-minor
 FidFile: PROTON01

Pulse Sequence: PROTON (s2pul)
Solvent: cdcl3
Data collected on: Jun  6 2010


 Temp. 25.0 C / 298.1 K
Sample #40, Operator: navarro

 Relax. delay 2.000 sec
 Pulse 45.0 degrees
 Acq. time 2.048 sec
 Width 8000.0 Hz
 16 repetitions
OBSERVE   H1, 499.7420505 MHz
DATA PROCESSING
FT size 32768
Total time 1 min 5 sec
```

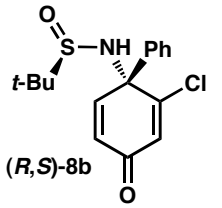


(*R*,*S*)-8b

minor diastereomer

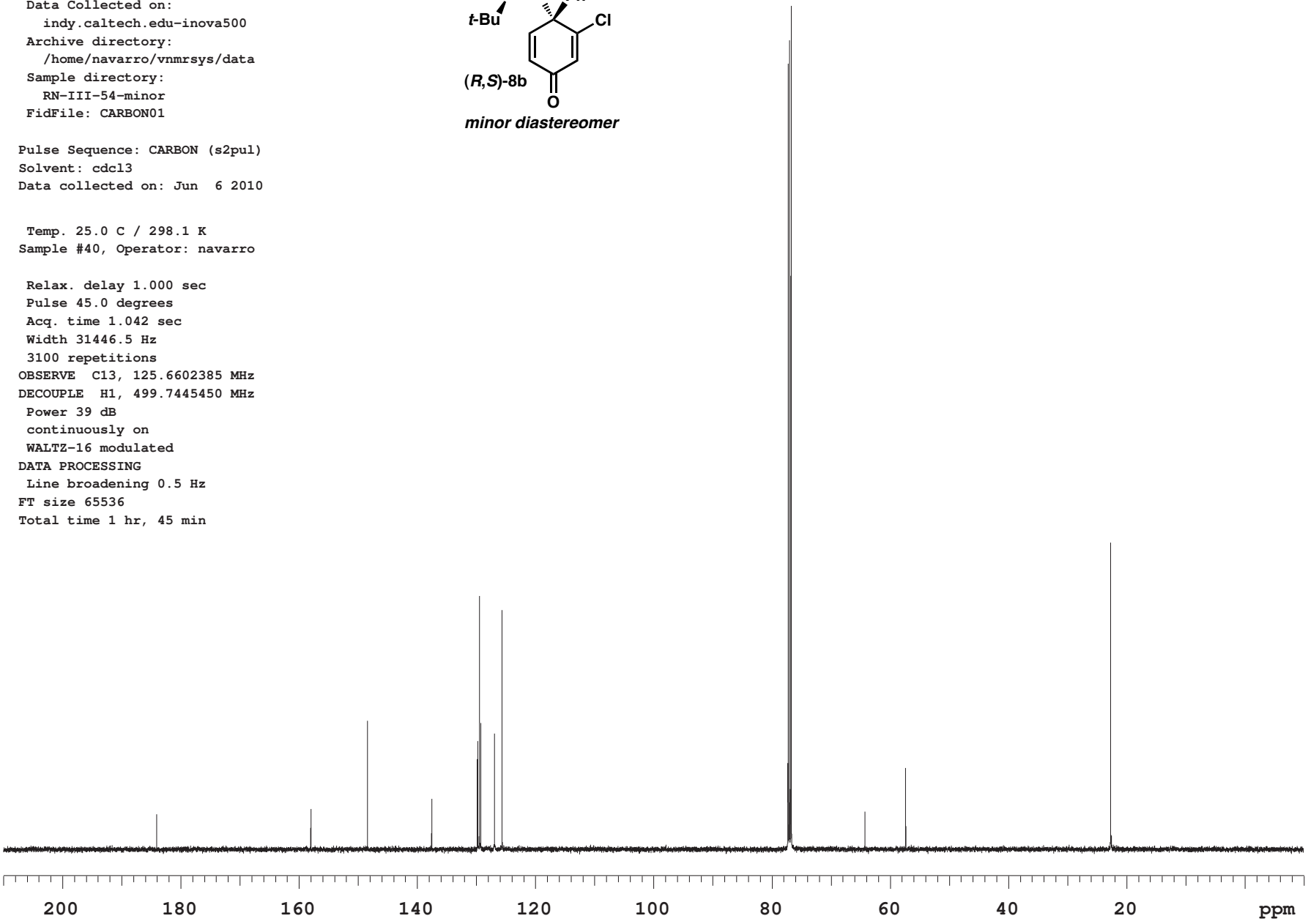


```
RN-III-54
mi-Sos
 Sample Name:
   RN-III-54-minor
 Data Collected on:
   indy.caltech.edu-inova500
 Archive directory:
   /home/navarro/vnmrsys/data
 Sample directory:
   RN-III-54-minor
 FidFile: CARBON01

Pulse Sequence: CARBON (s2pul)
Solvent: cdcl3
Data collected on: Jun  6 2010


 Temp. 25.0 C / 298.1 K
Sample #40, Operator: navarro

 Relax. delay 1.000 sec
 Pulse 45.0 degrees
 Acq. time 1.042 sec
 Width 31446.5 Hz
 3100 repetitions
OBSERVE  C13, 125.6602385 MHz
DECOUPLE  H1, 499.7445450 MHz
 Power 39 dB
 continuously on
 WALTZ-16 modulated
DATA PROCESSING
 Line broadening 0.5 Hz
FT size 65536
Total time 1 hr, 45 min
```



(*R*,*S*)-8b

minor diastereomer



RN-II-288-purified

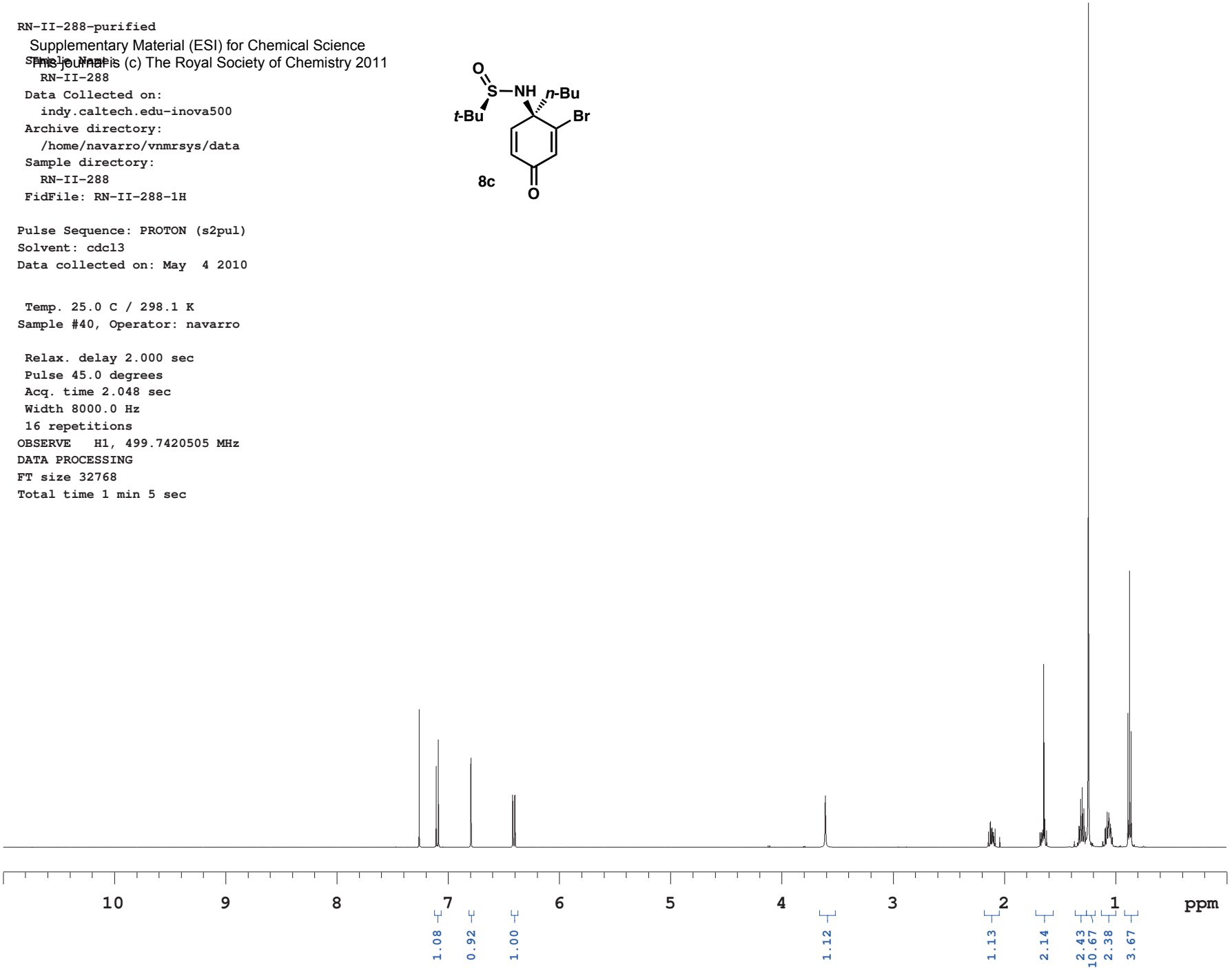
Sample Name:
RN-II-288
Data Collected on:
indy.caltech.edu-inova500
Archive directory:
/home/navarro/vnmrsys/data
Sample directory:
RN-II-288
FidFile: RN-II-288-1H

Pulse Sequence: PROTON (s2pul)
Solvent: cdcl3
Data collected on: May 4 2010

Temp. 25.0 C / 298.1 K
Sample #40, Operator: navarro

Relax. delay 2.000 sec
Pulse 45.0 degrees
Acq. time 2.048 sec
Width 8000.0 Hz
16 repetitions
OBSERVE H1, 499.7420505 MHz
DATA PROCESSING
FT size 32768
Total time 1 min 5 sec

8c



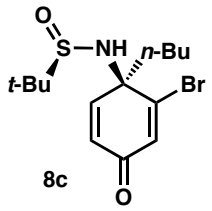
RN-II-288
purified

Sample Name:
RN-II-288-13C
Data Collected on:
indy.caltech.edu-inova500
Archive directory:
/home/navarro/vnmrsys/data
Sample directory:
RN-II-288-13C
FidFile: RN-II-288-13C

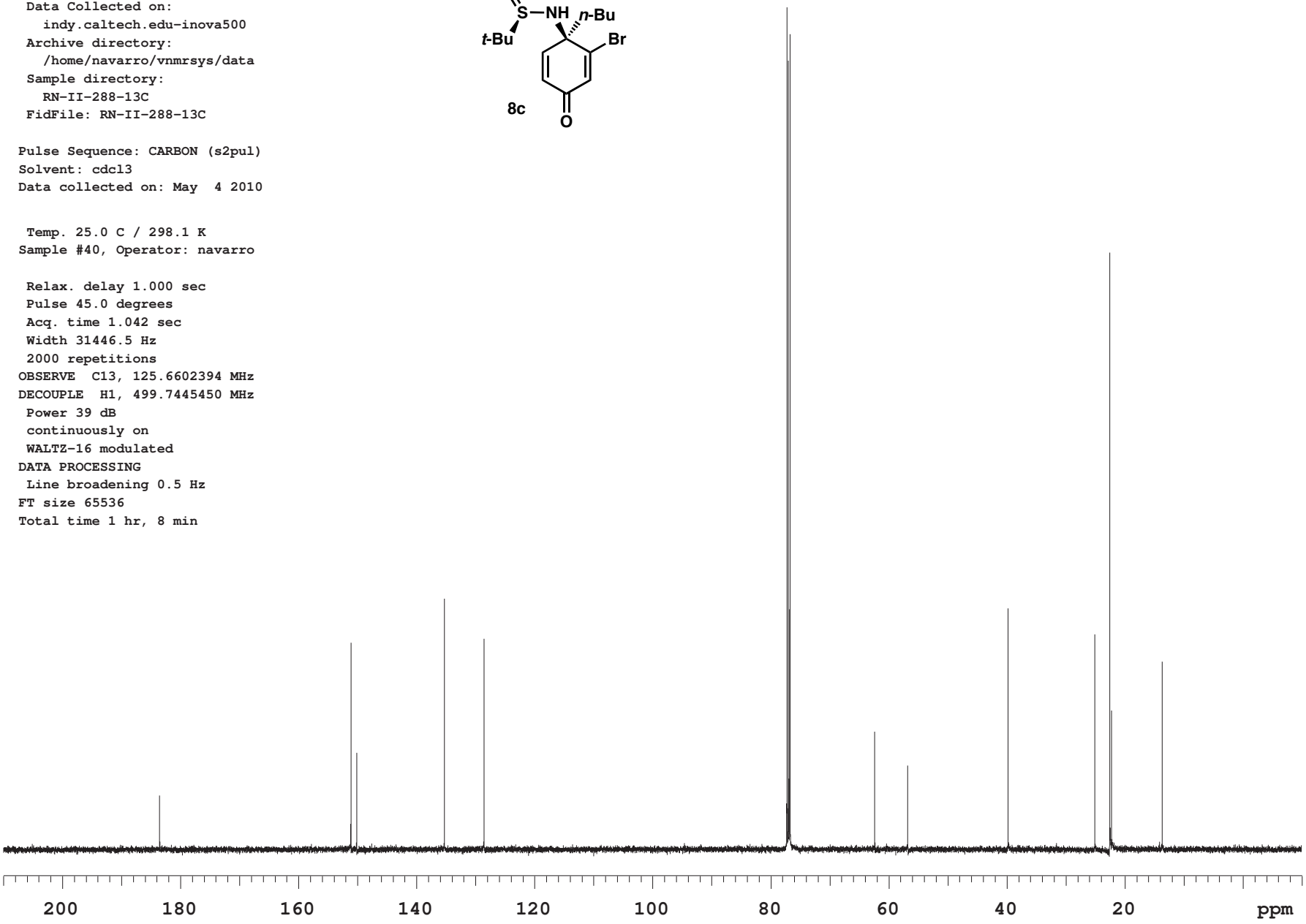
Pulse Sequence: CARBON (s2pul)
Solvent: cdcl3
Data collected on: May 4 2010

Temp. 25.0 C / 298.1 K
Sample #40, Operator: navarro

Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.042 sec
Width 31446.5 Hz
2000 repetitions
OBSERVE C13, 125.6602394 MHz
DECOUPLE H1, 499.7445450 MHz
Power 39 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 0.5 Hz
FT size 65536
Total time 1 hr, 8 min



8c



RN-II-299

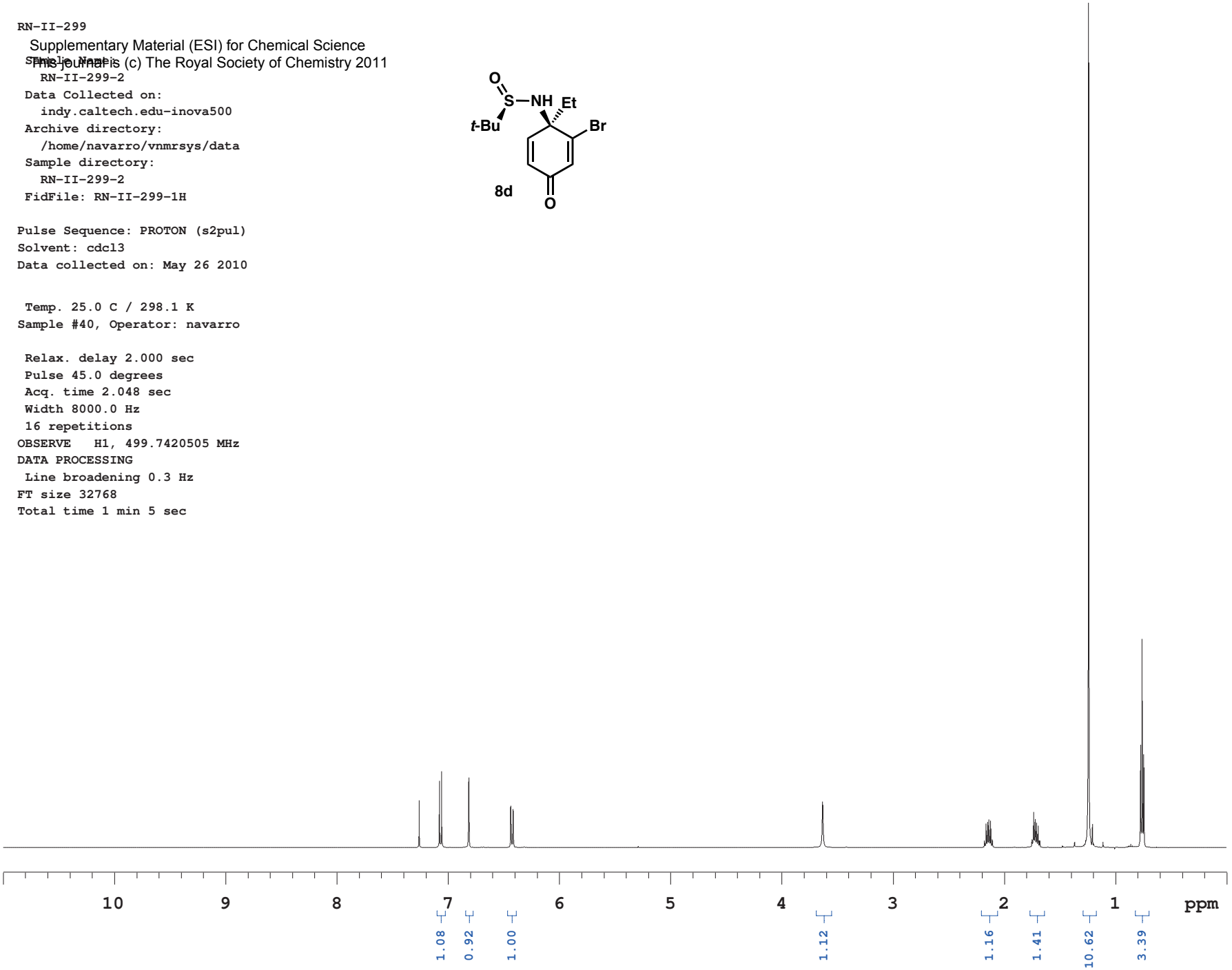
Sample Name:
RN-II-299-2
Data Collected on:
indy.caltech.edu-inova500
Archive directory:
/home/navarro/vnmrsys/data
Sample directory:
RN-II-299-2
FidFile: RN-II-299-1H

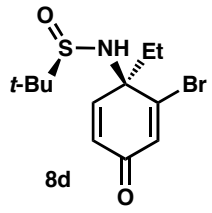
Pulse Sequence: PROTON (s2pul)
Solvent: cdcl3
Data collected on: May 26 2010

Temp. 25.0 C / 298.1 K
Sample #40, Operator: navarro

Relax. delay 2.000 sec
Pulse 45.0 degrees
Acq. time 2.048 sec
Width 8000.0 Hz
16 repetitions
OBSERVE H1, 499.7420505 MHz
DATA PROCESSING
Line broadening 0.3 Hz
FT size 32768
Total time 1 min 5 sec

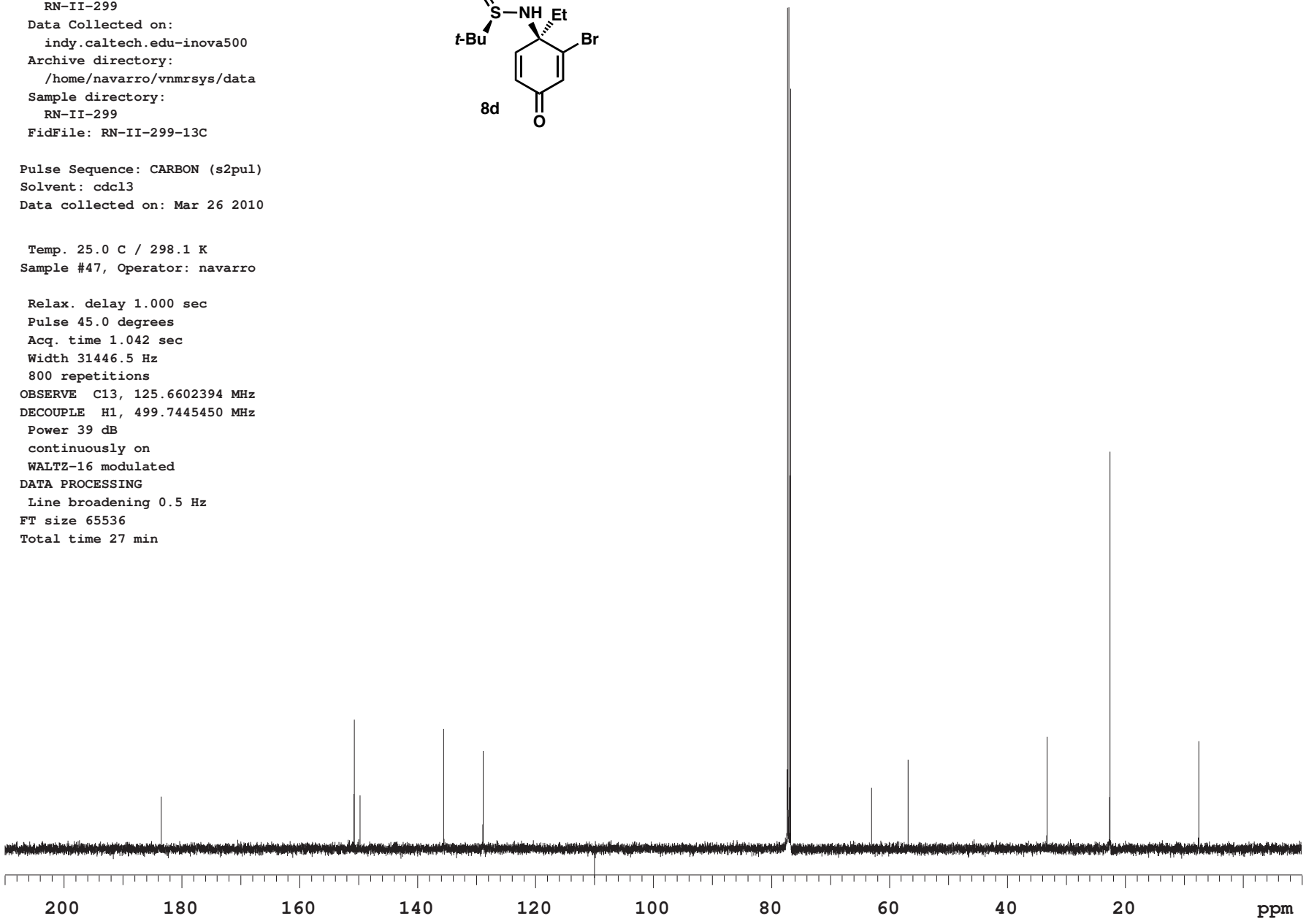
8d





8d

```
 Sample Name:
   RN-II-299
 Data Collected on:
   indy.caltech.edu-inova500
 Archive directory:
   /home/navarro/vnmrsys/data
 Sample directory:
   RN-II-299
 FidFile: RN-II-299-13C

Pulse Sequence: CARBON (s2pul)
Solvent: cdcl3
Data collected on: Mar 26 2010


 Temp. 25.0 C / 298.1 K
Sample #47, Operator: navarro

 Relax. delay 1.000 sec
 Pulse 45.0 degrees
 Acq. time 1.042 sec
 Width 31446.5 Hz
 800 repetitions
OBSERVE  C13, 125.6602394 MHz
DECOUPLE  H1, 499.7445450 MHz
 Power 39 dB
 continuously on
 WALTZ-16 modulated
DATA PROCESSING
 Line broadening 0.5 Hz
FT size 65536
Total time 27 min
```



RN-II-298
purified

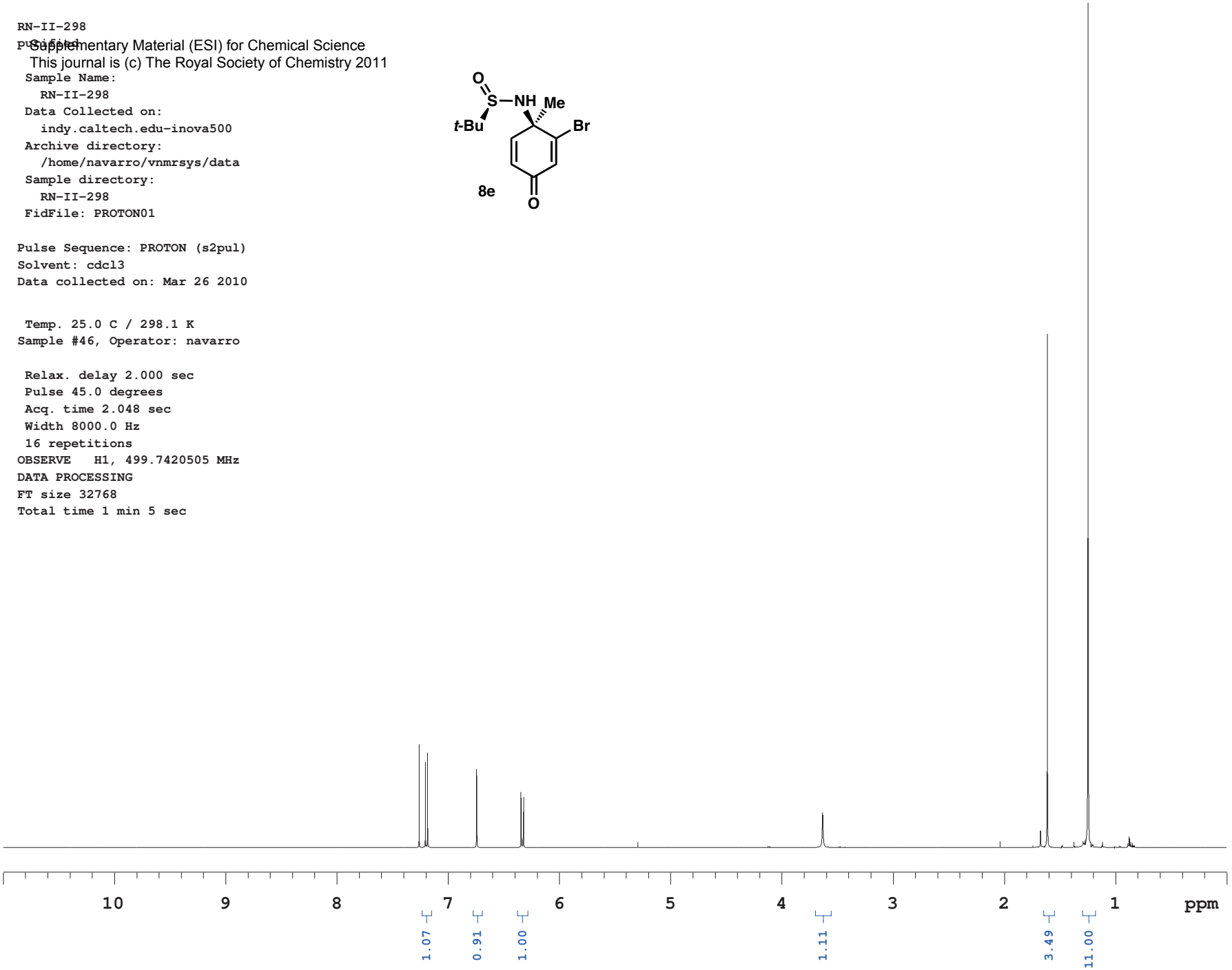
Sample Name:
RN-II-298
Data Collected on:
indy.caltech.edu-inova500
Archive directory:
/home/navarro/vnmrsys/data
Sample directory:
RN-II-298
FidFile: PROTON01

Pulse Sequence: PROTON (s2pul)
Solvent: cdcl3
Data collected on: Mar 26 2010

Temp. 25.0 C / 298.1 K
Sample #46, Operator: navarro

Relax. delay 2.000 sec
Pulse 45.0 degrees
Acq. time 2.048 sec
Width 8000.0 Hz
16 repetitions
OBSERVE H1, 499.7420505 MHz
DATA PROCESSING
FT size 32768
Total time 1 min 5 sec

8e

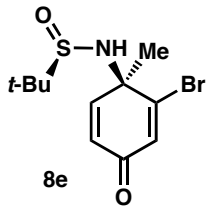


Sample Name:
RN-II-298
Data Collected on:
indy.caltech.edu-inova500
Archive directory:
/home/navarro/vnmrsys/data
Sample directory:
RN-II-298
FidFile: RN-II-298-13C

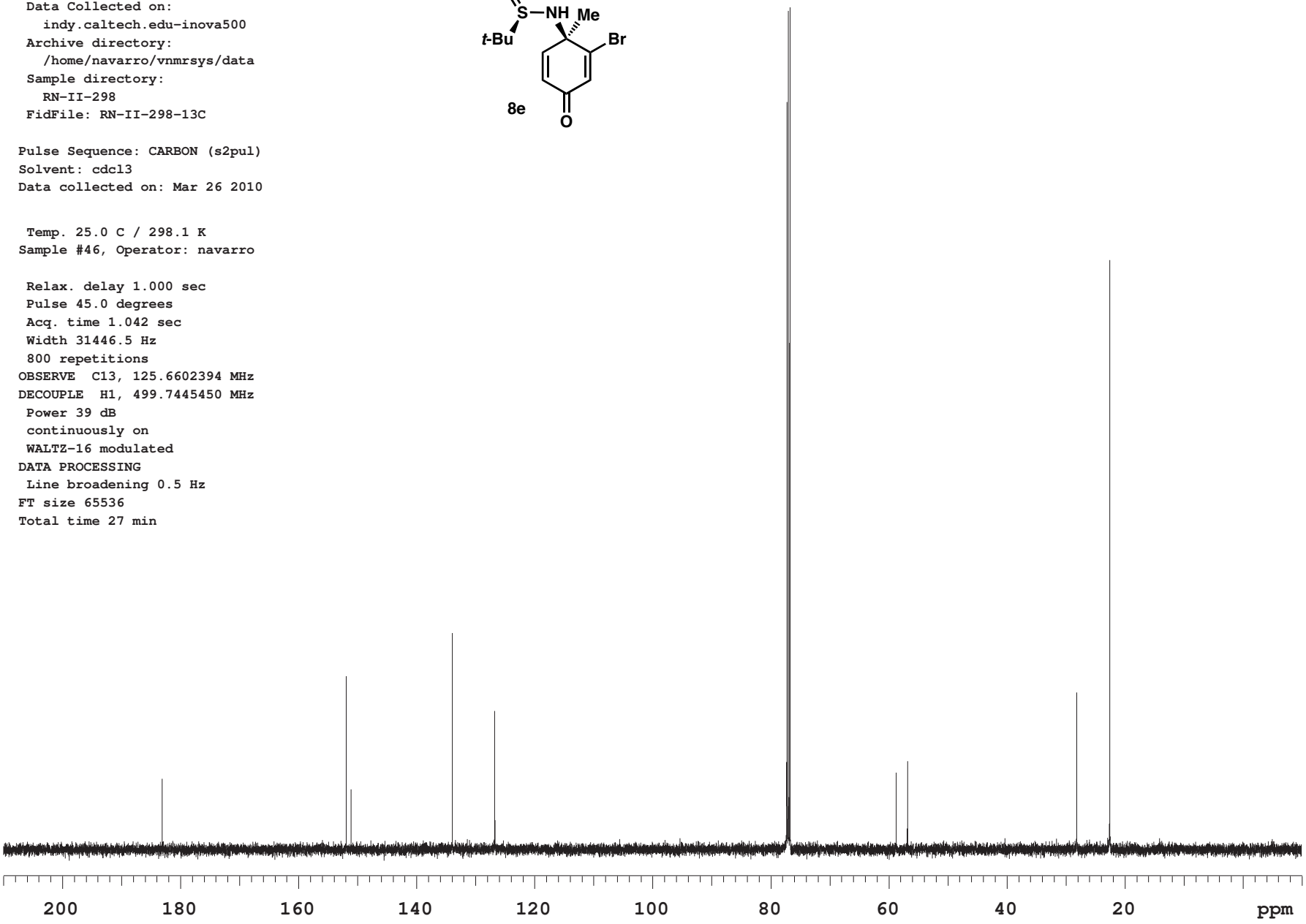
Pulse Sequence: CARBON (s2pul)
Solvent: cdcl3
Data collected on: Mar 26 2010

Temp. 25.0 C / 298.1 K
Sample #46, Operator: navarro

Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.042 sec
Width 31446.5 Hz
800 repetitions
OBSERVE C13, 125.6602394 MHz
DECOUPLE H1, 499.7445450 MHz
Power 39 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 0.5 Hz
FT size 65536
Total time 27 min



8e



RN-III-229
purified

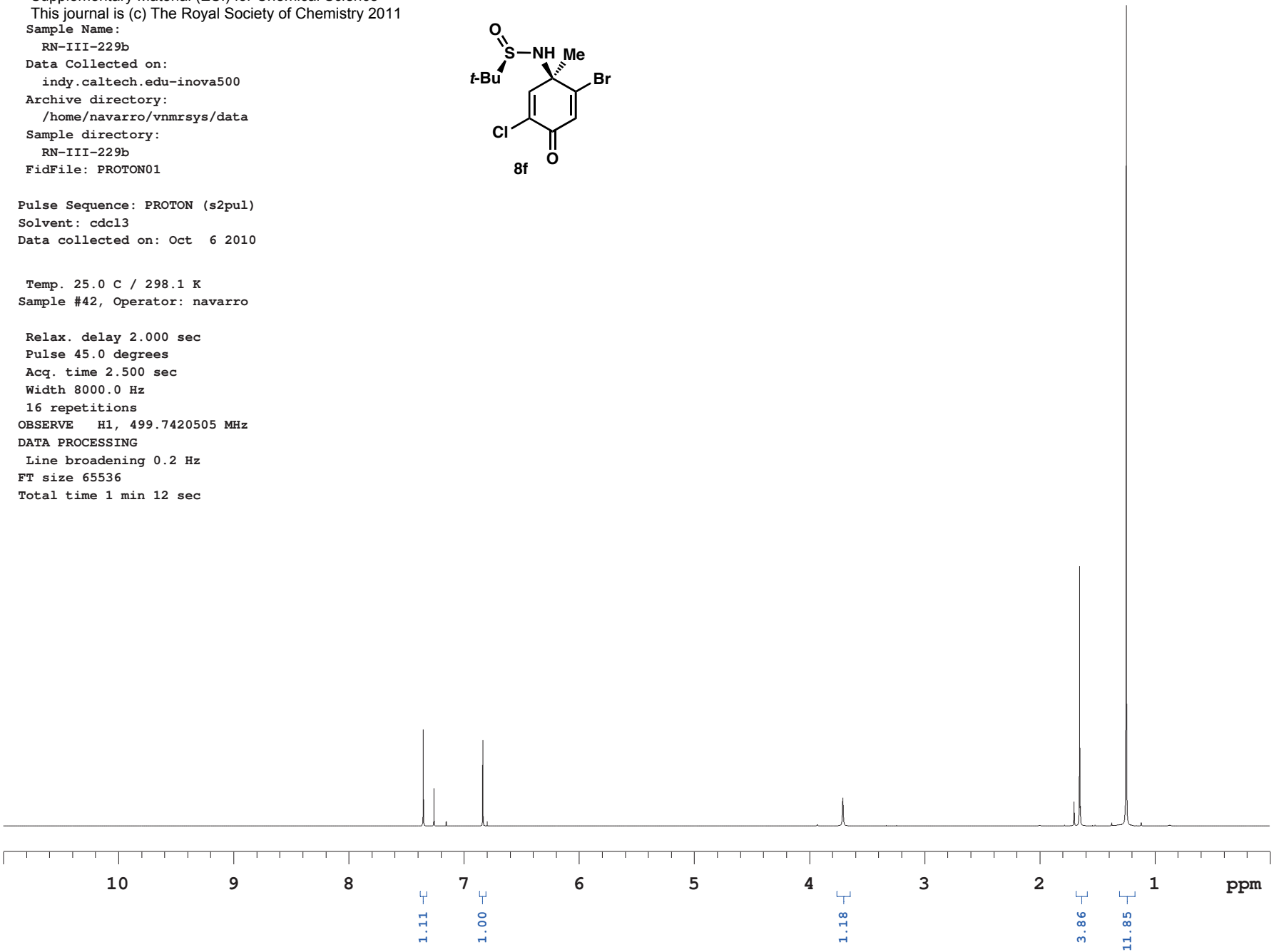
Sample Name:
RN-III-229b
Data Collected on:
indy.caltech.edu-inova500
Archive directory:
/home/navarro/vnmrsys/data
Sample directory:
RN-III-229b
FidFile: PROTON01

Pulse Sequence: PROTON (s2pul)
Solvent: cdcl3
Data collected on: Oct 6 2010

Temp. 25.0 C / 298.1 K
Sample #42, Operator: navarro

Relax. delay 2.000 sec
Pulse 45.0 degrees
Acq. time 2.500 sec
Width 8000.0 Hz
16 repetitions
OBSERVE H1, 499.7420505 MHz
DATA PROCESSING
Line broadening 0.2 Hz
FT size 65536
Total time 1 min 12 sec

8f



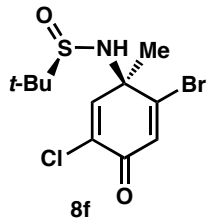
RN-III-229
purified

Sample Name:
RN-III-229b
Data Collected on:
indy.caltech.edu-inova500
Archive directory:
/home/navarro/vnmrsys/data
Sample directory:
RN-III-229b
FidFile: CARBON01

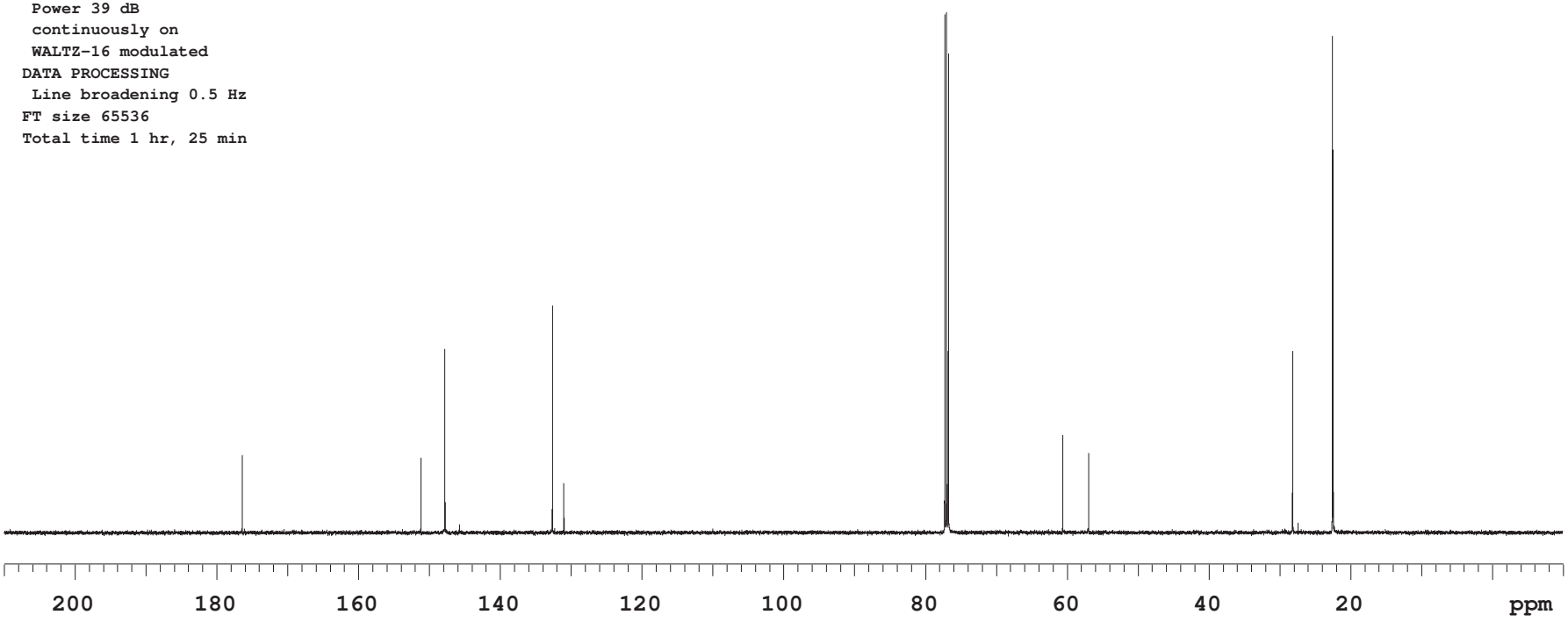
Pulse Sequence: CARBON (s2pul)
Solvent: cdcl3
Data collected on: Oct 6 2010

Temp. 25.0 C / 298.1 K
Sample #42, Operator: navarro

Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.042 sec
Width 31446.5 Hz
2500 repetitions
OBSERVE C13, 125.6602404 MHz
DECOUPLE H1, 499.7445450 MHz
Power 39 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 0.5 Hz
FT size 65536
Total time 1 hr, 25 min



8f



RN-III-228
purified

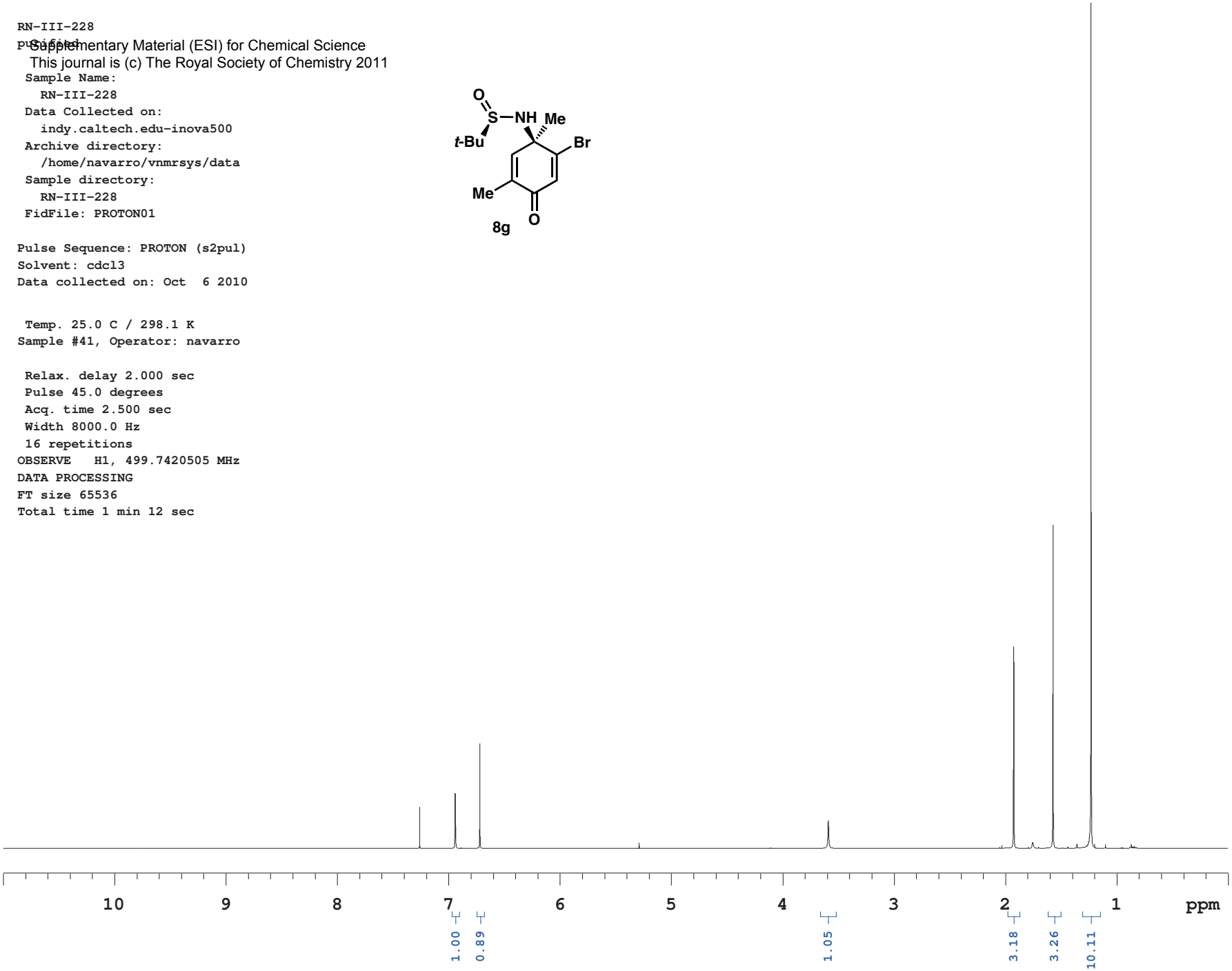
Sample Name:
RN-III-228
Data Collected on:
indy.caltech.edu-inova500
Archive directory:
/home/navarro/vnmrsys/data
Sample directory:
RN-III-228
FidFile: PROTON01

Pulse Sequence: PROTON (s2pul)
Solvent: cdcl3
Data collected on: Oct 6 2010

Temp. 25.0 C / 298.1 K
Sample #41, Operator: navarro

Relax. delay 2.000 sec
Pulse 45.0 degrees
Acq. time 2.500 sec
Width 8000.0 Hz
16 repetitions
OBSERVE H1, 499.7420505 MHz
DATA PROCESSING
FT size 65536
Total time 1 min 12 sec

8g



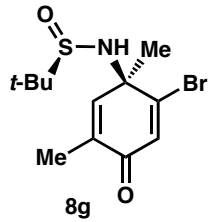
RN-III-228
purified

Sample Name:
RN-III-228
Data Collected on:
indy.caltech.edu-inova500
Archive directory:
/home/navarro/vnmrsys/data
Sample directory:
RN-III-228
FidFile: CARBON01

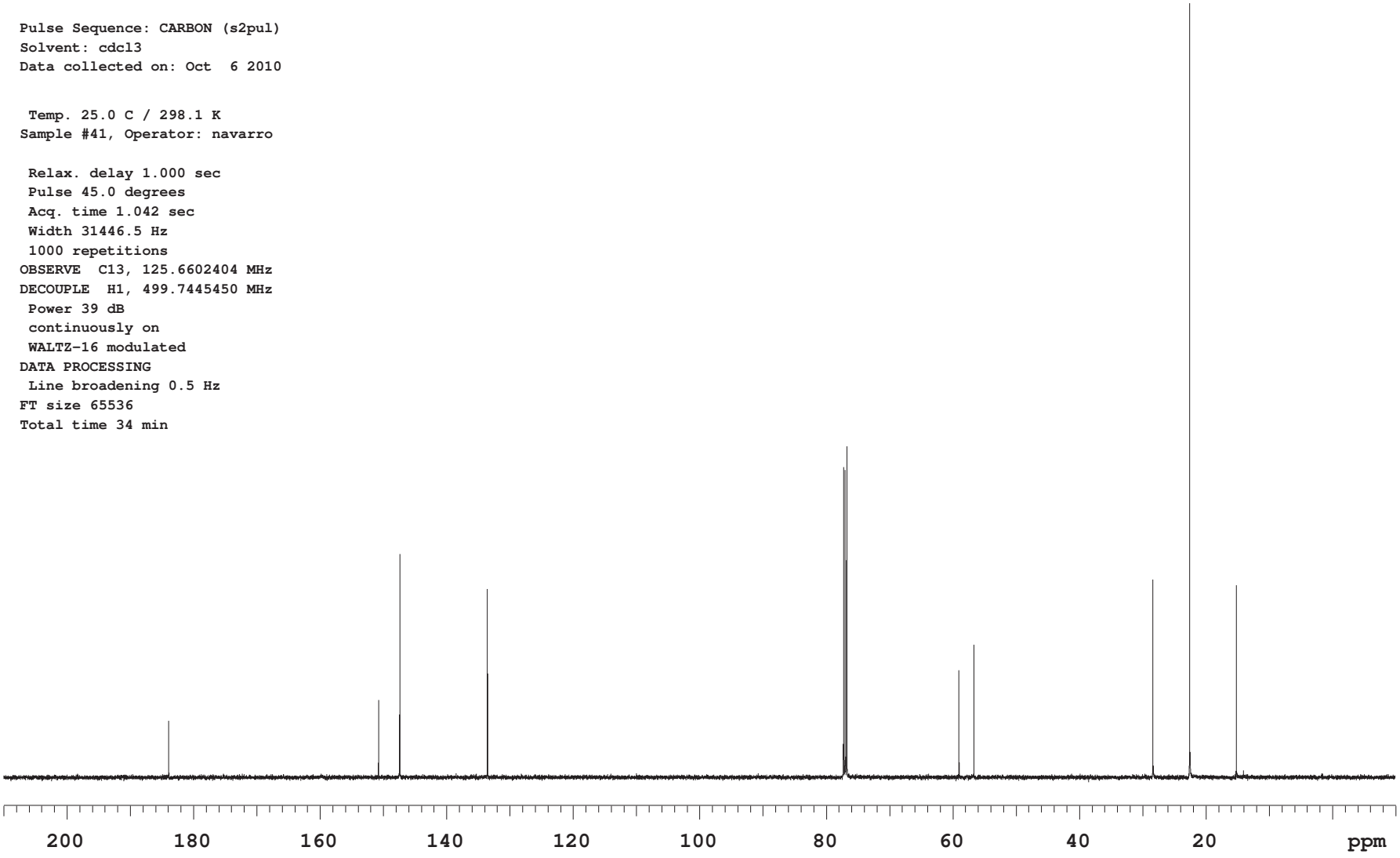
Pulse Sequence: CARBON (s2pul)
Solvent: cdcl3
Data collected on: Oct 6 2010

Temp. 25.0 C / 298.1 K
Sample #41, Operator: navarro

Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.042 sec
Width 31446.5 Hz
1000 repetitions
OBSERVE C13, 125.6602404 MHz
DECOUPLE H1, 499.7445450 MHz
Power 39 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 0.5 Hz
FT size 65536
Total time 34 min



8g



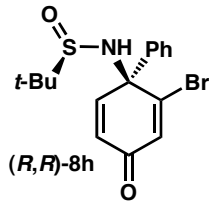
RN-II-293
major products

Sample Name:
RN-II-293-A
Data Collected on:
indy.caltech.edu-inova500
Archive directory:
/home/navarro/vnmrsys/data
Sample directory:
RN-II-293-A
FidFile: RN-II-293-major-1H

Pulse Sequence: PROTON (s2pul)
Solvent: cdcl3
Data collected on: Mar 30 2010

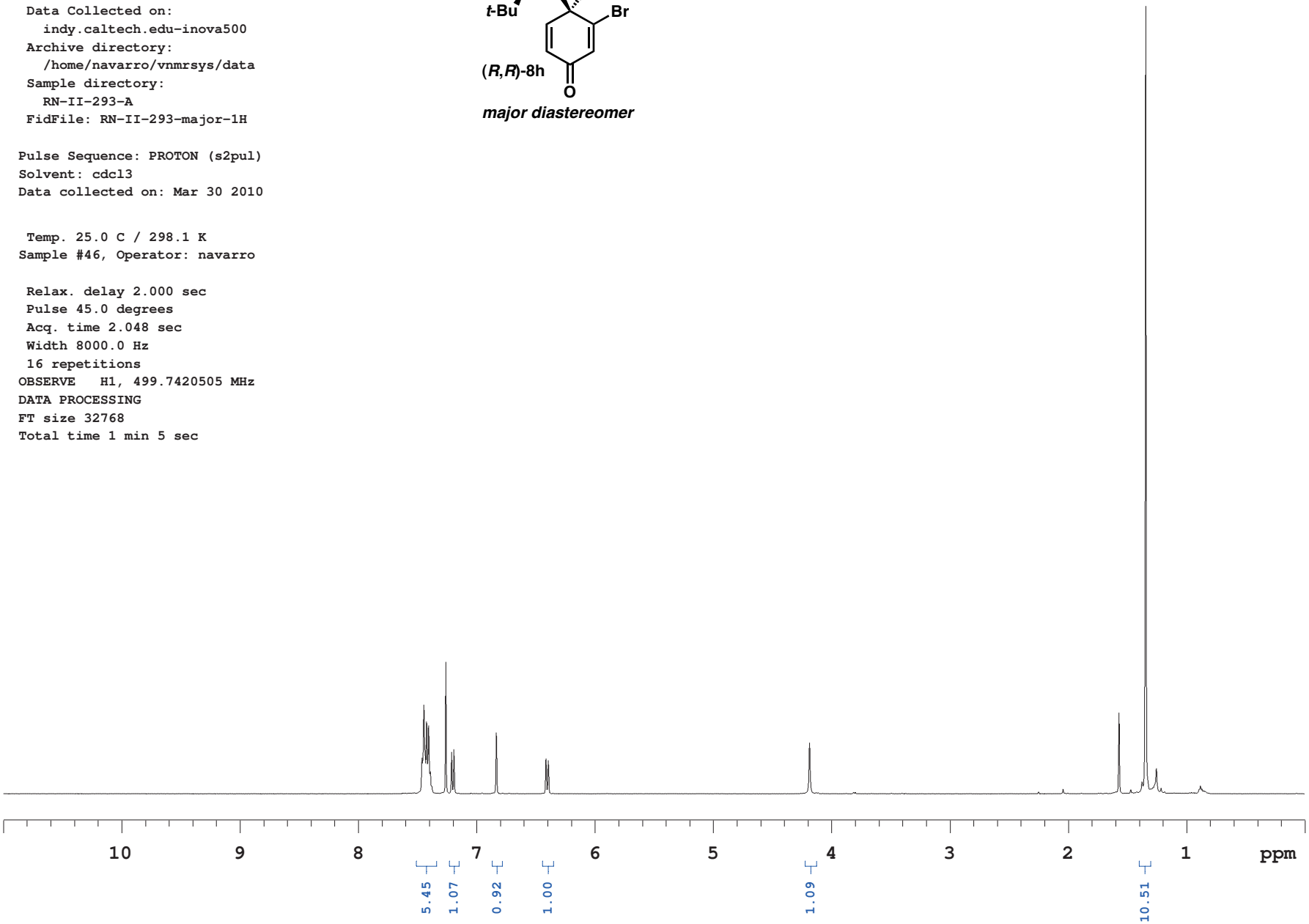
Temp. 25.0 C / 298.1 K
Sample #46, Operator: navarro

Relax. delay 2.000 sec
Pulse 45.0 degrees
Acq. time 2.048 sec
Width 8000.0 Hz
16 repetitions
OBSERVE H1, 499.7420505 MHz
DATA PROCESSING
FT size 32768
Total time 1 min 5 sec



(*R*,*R*)-8h

major diastereomer



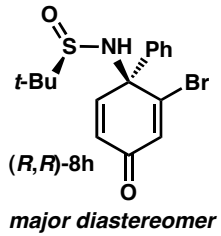
RN-II-293
major product

Sample Name:
RN-II-293
Data Collected on:
indy.caltech.edu-inova500
Archive directory:
/home/navarro/vnmrsys/data
Sample directory:
RN-II-293
FidFile: RN-II-293-major-13C

Pulse Sequence: CARBON (s2pul)
Solvent: cdcl3
Data collected on: Mar 30 2010

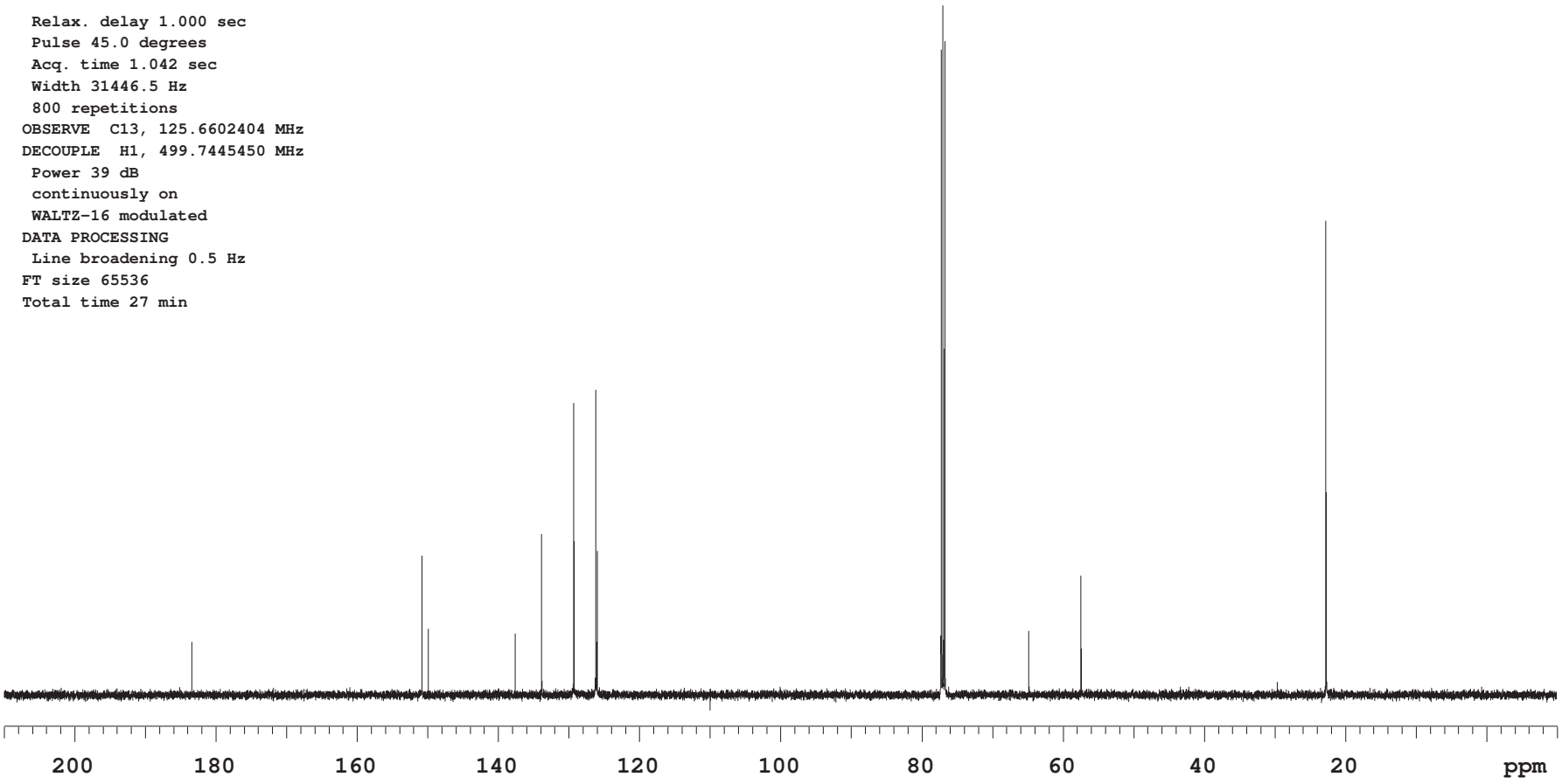
Temp. 25.0 C / 298.1 K
Sample #46, Operator: navarro

Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.042 sec
Width 31446.5 Hz
800 repetitions
OBSERVE C13, 125.6602404 MHz
DECOUPLE H1, 499.7445450 MHz
Power 39 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 0.5 Hz
FT size 65536
Total time 27 min



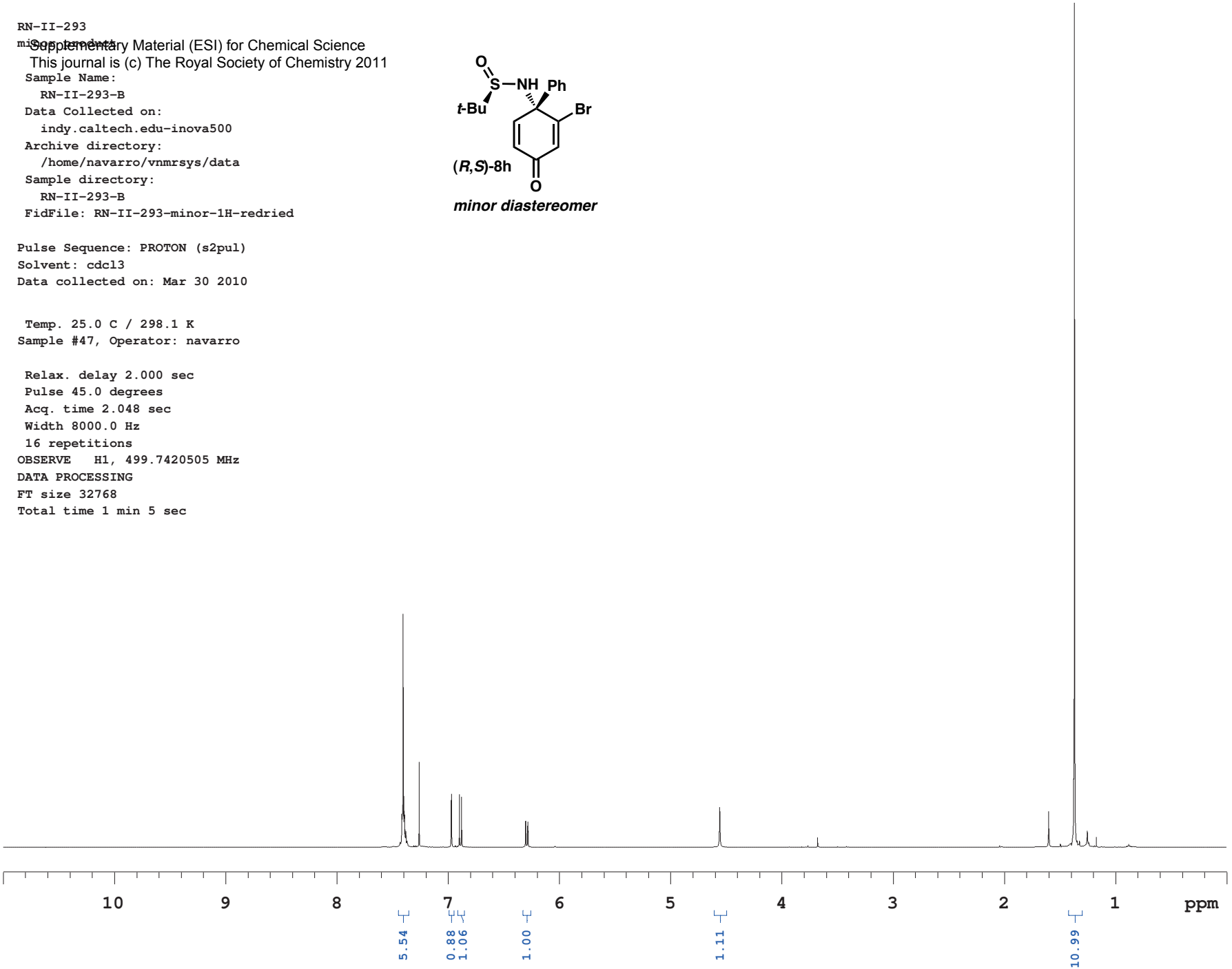
(*R*,*R*)-8h

major diastereomer



```
RN-II-293
minor products
 Sample Name:
   RN-II-293-B
 Data Collected on:
   indy.caltech.edu-inova500
 Archive directory:
   /home/navarro/vnmrsys/data
 Sample directory:
   RN-II-293-B
 FidFile: RN-II-293-minor-1H-redried

Pulse Sequence: PROTON (s2pul)
Solvent: cdcl3
Data collected on: Mar 30 2010


 Temp. 25.0 C / 298.1 K
Sample #47, Operator: navarro

 Relax. delay 2.000 sec
 Pulse 45.0 degrees
 Acq. time 2.048 sec
 Width 8000.0 Hz
 16 repetitions
OBSERVE   H1, 499.7420505 MHz
DATA PROCESSING
FT size 32768
Total time 1 min 5 sec
```


minor diastereomer



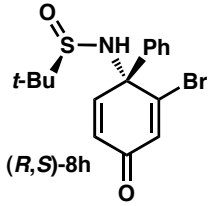
RN-III-293 minor
purified

Sample Name:
RN-II-293-minor
Data Collected on:
indy.caltech.edu-inova500
Archive directory:
/home/navarro/vnmrsys/data
Sample directory:
RN-II-293-minor
FidFile: CARBON01

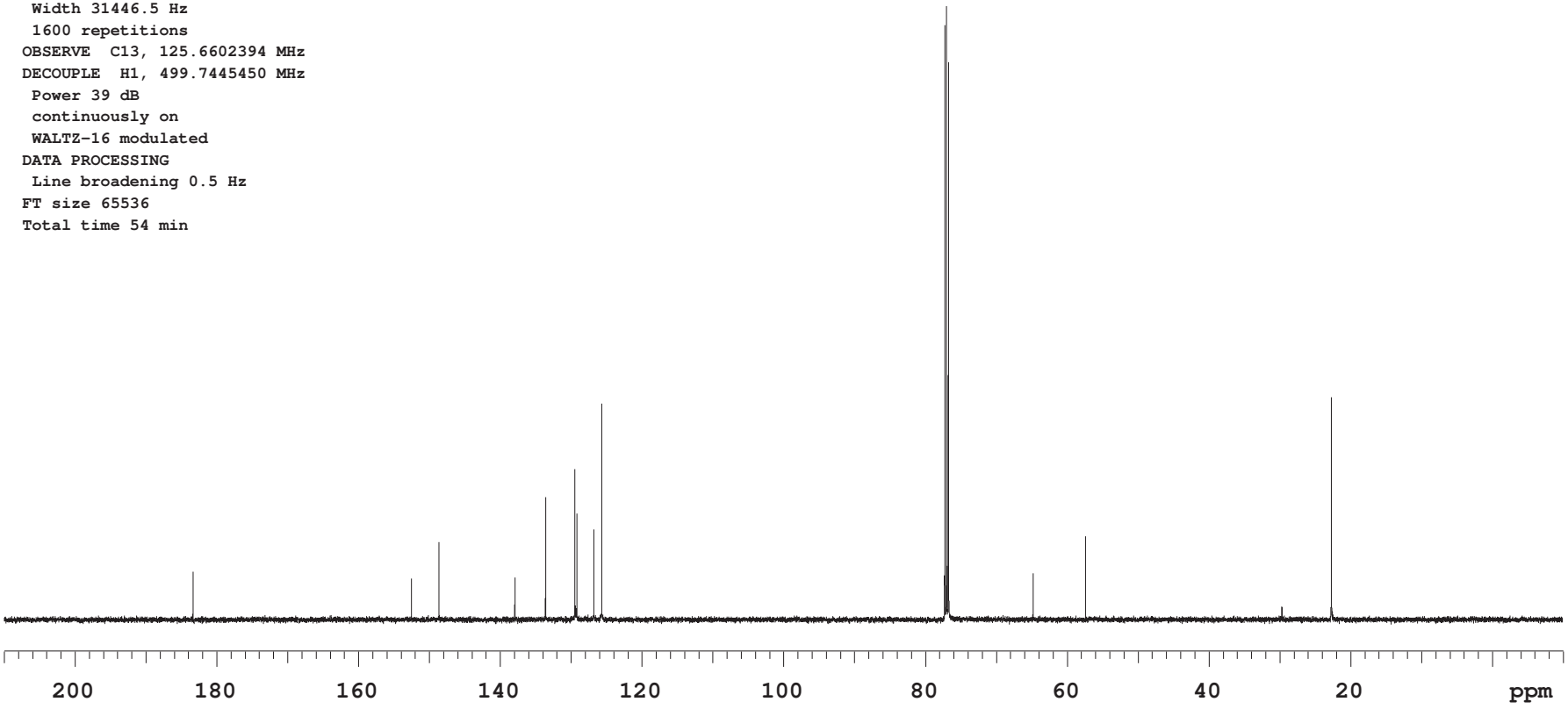
Pulse Sequence: CARBON (s2pul)
Solvent: cdcl3
Data collected on: Oct 20 2010

Temp. 25.0 C / 298.1 K
Sample #41, Operator: navarro

Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.042 sec
Width 31446.5 Hz
1600 repetitions
OBSERVE C13, 125.6602394 MHz
DECOUPLE H1, 499.7445450 MHz
Power 39 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 0.5 Hz
FT size 65536
Total time 54 min



minor diastereomer



RN-III-67

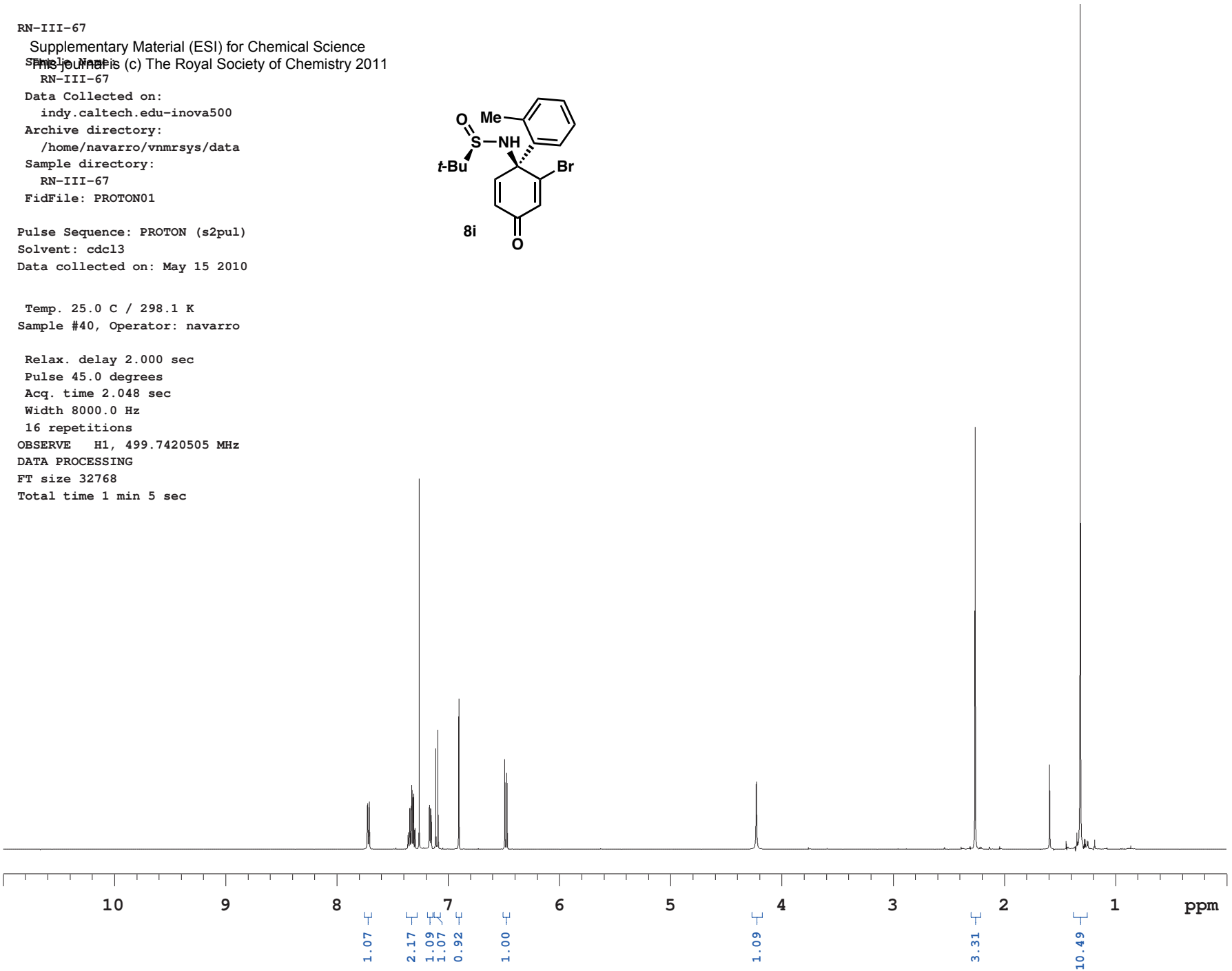
Sample Name:
RN-III-67
Data Collected on:
indy.caltech.edu-inova500
Archive directory:
/home/navarro/vnmrsys/data
Sample directory:
RN-III-67
FidFile: PROTON01

Pulse Sequence: PROTON (s2pul)
Solvent: cdcl3
Data collected on: May 15 2010

Temp. 25.0 C / 298.1 K
Sample #40, Operator: navarro

Relax. delay 2.000 sec
Pulse 45.0 degrees
Acq. time 2.048 sec
Width 8000.0 Hz
16 repetitions
OBSERVE H1, 499.7420505 MHz
DATA PROCESSING
FT size 32768
Total time 1 min 5 sec

8i



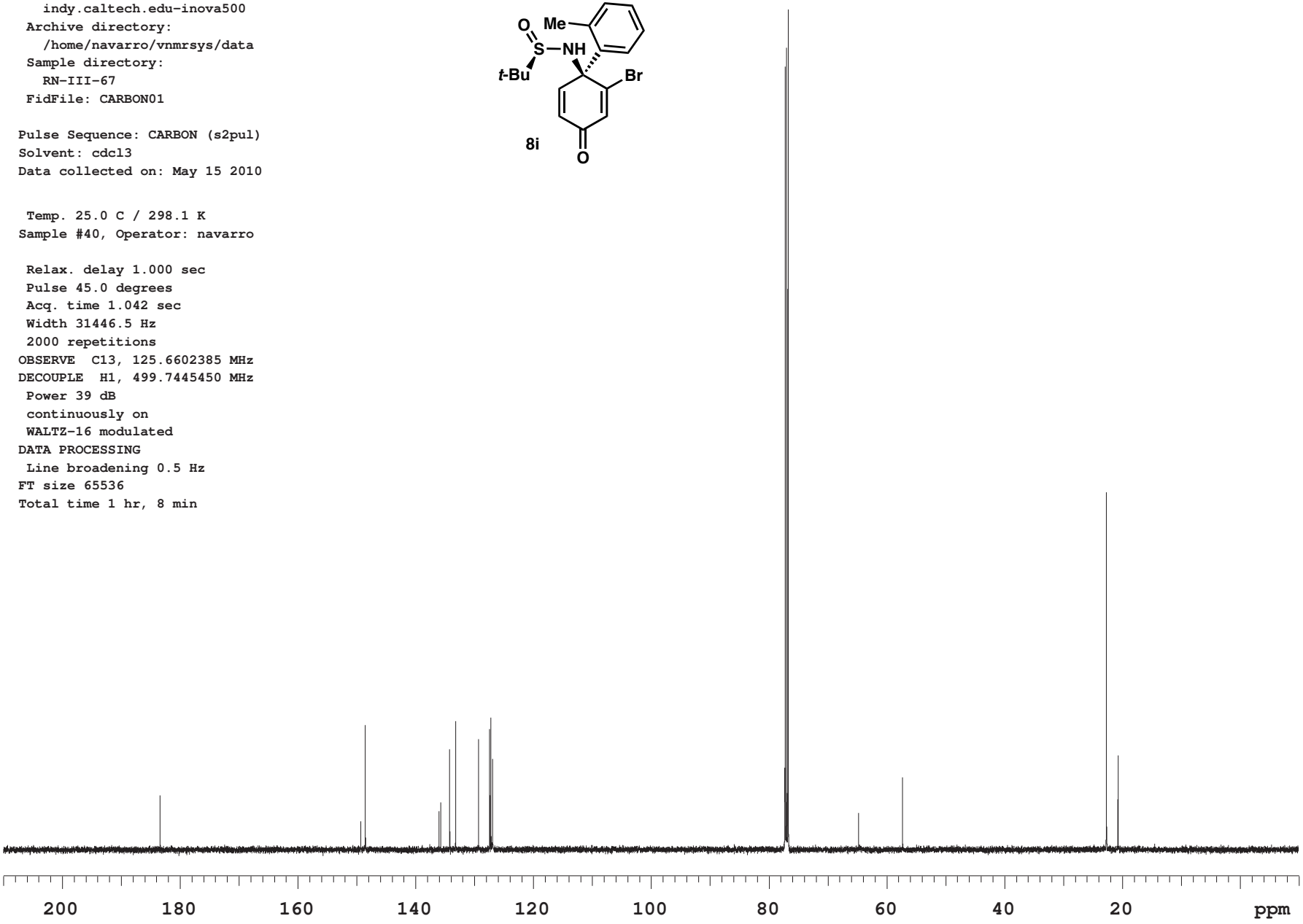
Sample Name:
RN-III-67
Data Collected on:
indy.caltech.edu-inova500
Archive directory:
/home/navarro/vnmrsys/data
Sample directory:
RN-III-67
FidFile: CARBON01

Pulse Sequence: CARBON (s2pul)
Solvent: cdcl3
Data collected on: May 15 2010

Temp. 25.0 C / 298.1 K
Sample #40, Operator: navarro

Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.042 sec
Width 31446.5 Hz
2000 repetitions
OBSERVE C13, 125.6602385 MHz
DECOUPLE H1, 499.7445450 MHz
Power 39 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 0.5 Hz
FT size 65536
Total time 1 hr, 8 min

8i



RN-III-68

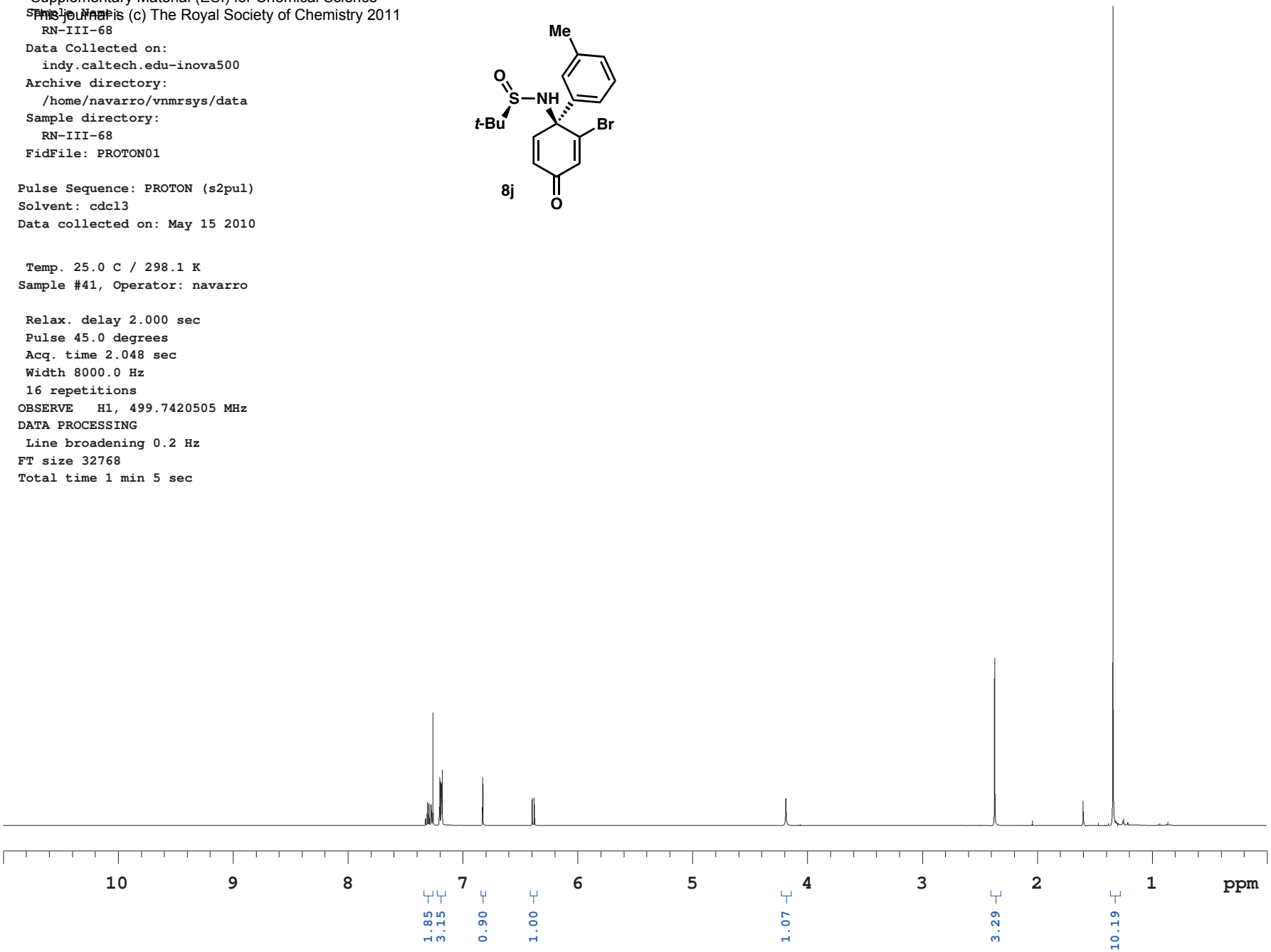
Sample Name:
RN-III-68
Data Collected on:
indy.caltech.edu-inova500
Archive directory:
/home/navarro/vnmrsys/data
Sample directory:
RN-III-68
FidFile: PROTON01

Pulse Sequence: PROTON (s2pul)
Solvent: cdcl3
Data collected on: May 15 2010

Temp. 25.0 C / 298.1 K
Sample #41, Operator: navarro

Relax. delay 2.000 sec
Pulse 45.0 degrees
Acq. time 2.048 sec
Width 8000.0 Hz
16 repetitions
OBSERVE H1, 499.7420505 MHz
DATA PROCESSING
Line broadening 0.2 Hz
FT size 32768
Total time 1 min 5 sec

8j

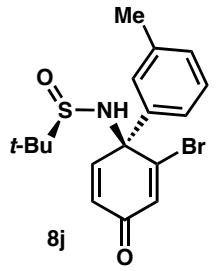


Sample Name:
RN-III-68
Data Collected on:
indy.caltech.edu-inova500
Archive directory:
/home/navarro/vnmrsys/data
Sample directory:
RN-III-68
FidFile: CARBON01

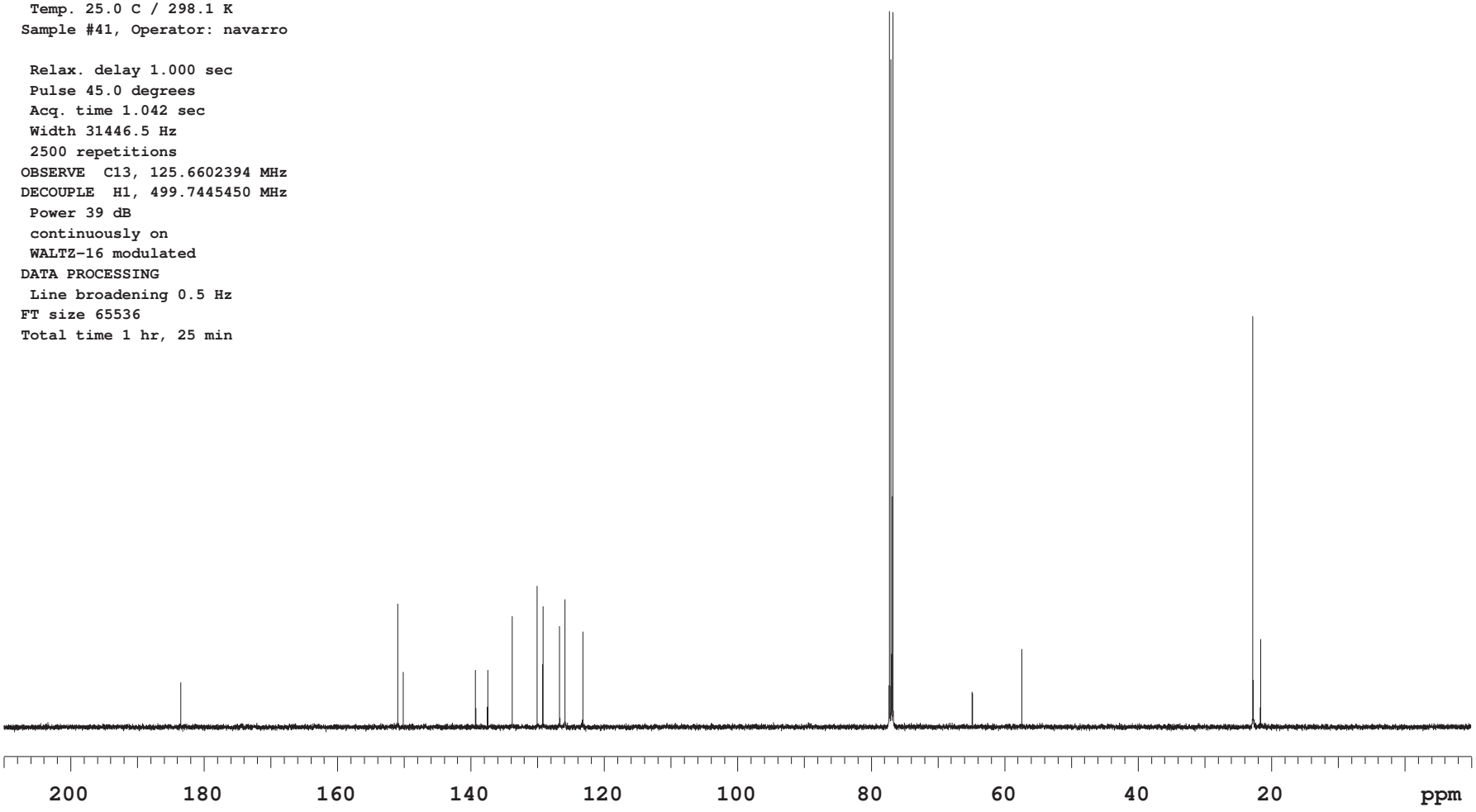
Pulse Sequence: CARBON (s2pul)
Solvent: cdcl3
Data collected on: May 15 2010

Temp. 25.0 C / 298.1 K
Sample #41, Operator: navarro

Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.042 sec
Width 31446.5 Hz
2500 repetitions
OBSERVE C13, 125.6602394 MHz
DECOUPLE H1, 499.7445450 MHz
Power 39 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 0.5 Hz
FT size 65536
Total time 1 hr, 25 min



8j



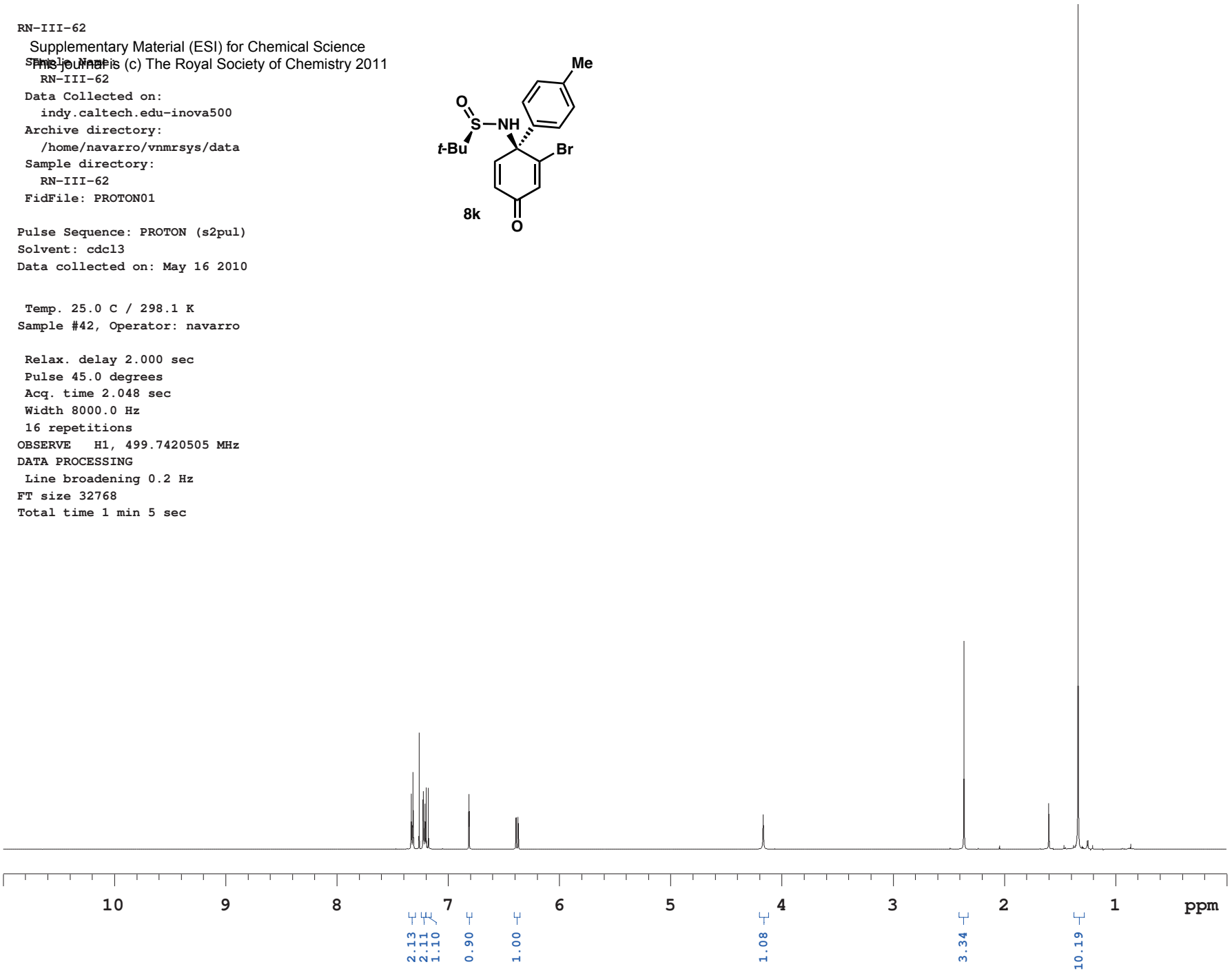
RN-III-62

Sample Name:
RN-III-62
Data Collected on:
indy.caltech.edu-inova500
Archive directory:
/home/navarro/vnmrsys/data
Sample directory:
RN-III-62
FidFile: PROTON01

Pulse Sequence: PROTON (s2pul)
Solvent: cdcl3
Data collected on: May 16 2010

Temp. 25.0 C / 298.1 K
Sample #42, Operator: navarro

Relax. delay 2.000 sec
Pulse 45.0 degrees
Acq. time 2.048 sec
Width 8000.0 Hz
16 repetitions
OBSERVE H1, 499.7420505 MHz
DATA PROCESSING
Line broadening 0.2 Hz
FT size 32768
Total time 1 min 5 sec

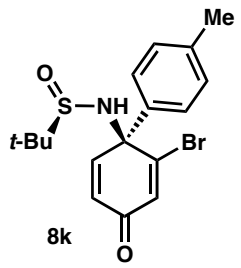


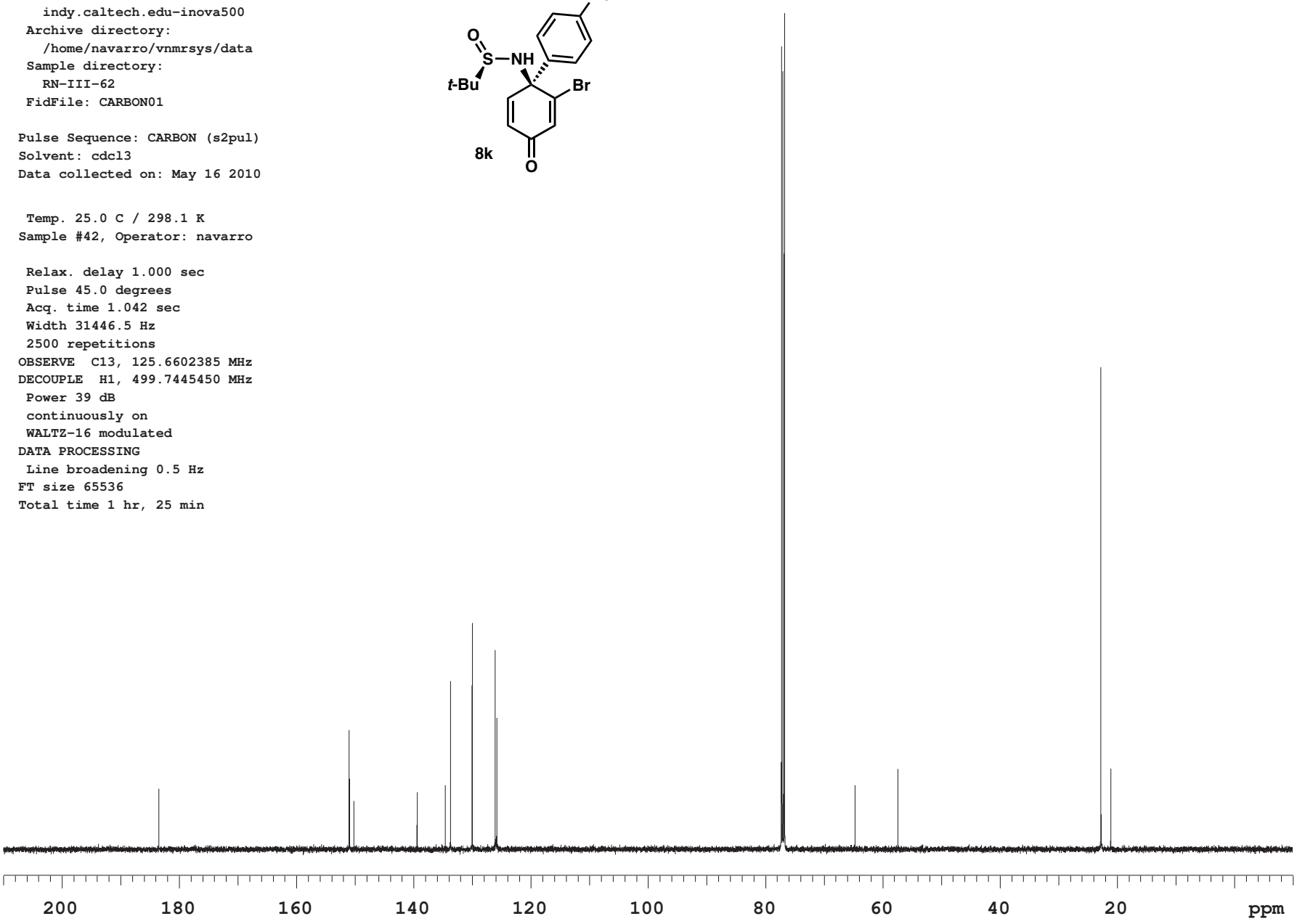
Sample Name:
RN-III-62
Data Collected on:
indy.caltech.edu-inova500
Archive directory:
/home/navarro/vnmrsys/data
Sample directory:
RN-III-62
FidFile: CARBON01

Pulse Sequence: CARBON (s2pul)
Solvent: cdcl3
Data collected on: May 16 2010

Temp. 25.0 C / 298.1 K
Sample #42, Operator: navarro

Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.042 sec
Width 31446.5 Hz
2500 repetitions
OBSERVE C13, 125.6602385 MHz
DECOUPLE H1, 499.7445450 MHz
Power 39 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 0.5 Hz
FT size 65536
Total time 1 hr, 25 min





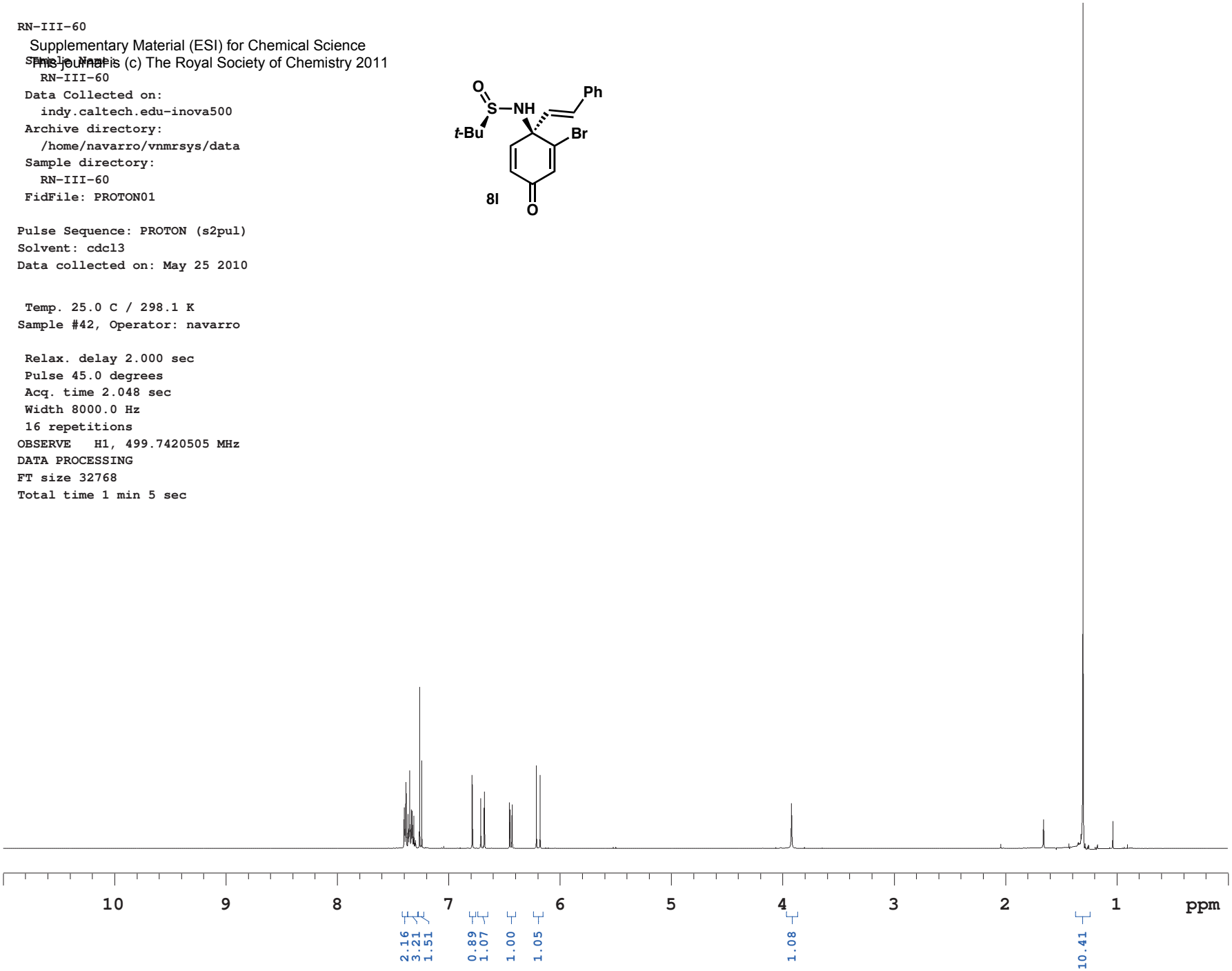
Sample Name:
RN-III-60
Data Collected on:
indy.caltech.edu-inova500
Archive directory:
/home/navarro/vnmrsys/data
Sample directory:
RN-III-60
FidFile: PROTON01

Pulse Sequence: PROTON (s2pul)
Solvent: cdcl3
Data collected on: May 25 2010

Temp. 25.0 C / 298.1 K
Sample #42, Operator: navarro

Relax. delay 2.000 sec
Pulse 45.0 degrees
Acq. time 2.048 sec
Width 8000.0 Hz
16 repetitions
OBSERVE H1, 499.7420505 MHz
DATA PROCESSING
FT size 32768
Total time 1 min 5 sec

8l



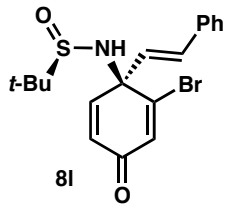
RN-III-60

Sample Name:
RN-III-60
Data Collected on:
indy.caltech.edu-inova500
Archive directory:
/home/navarro/vnmrsys/data
Sample directory:
RN-III-60
FidFile: CARBON01

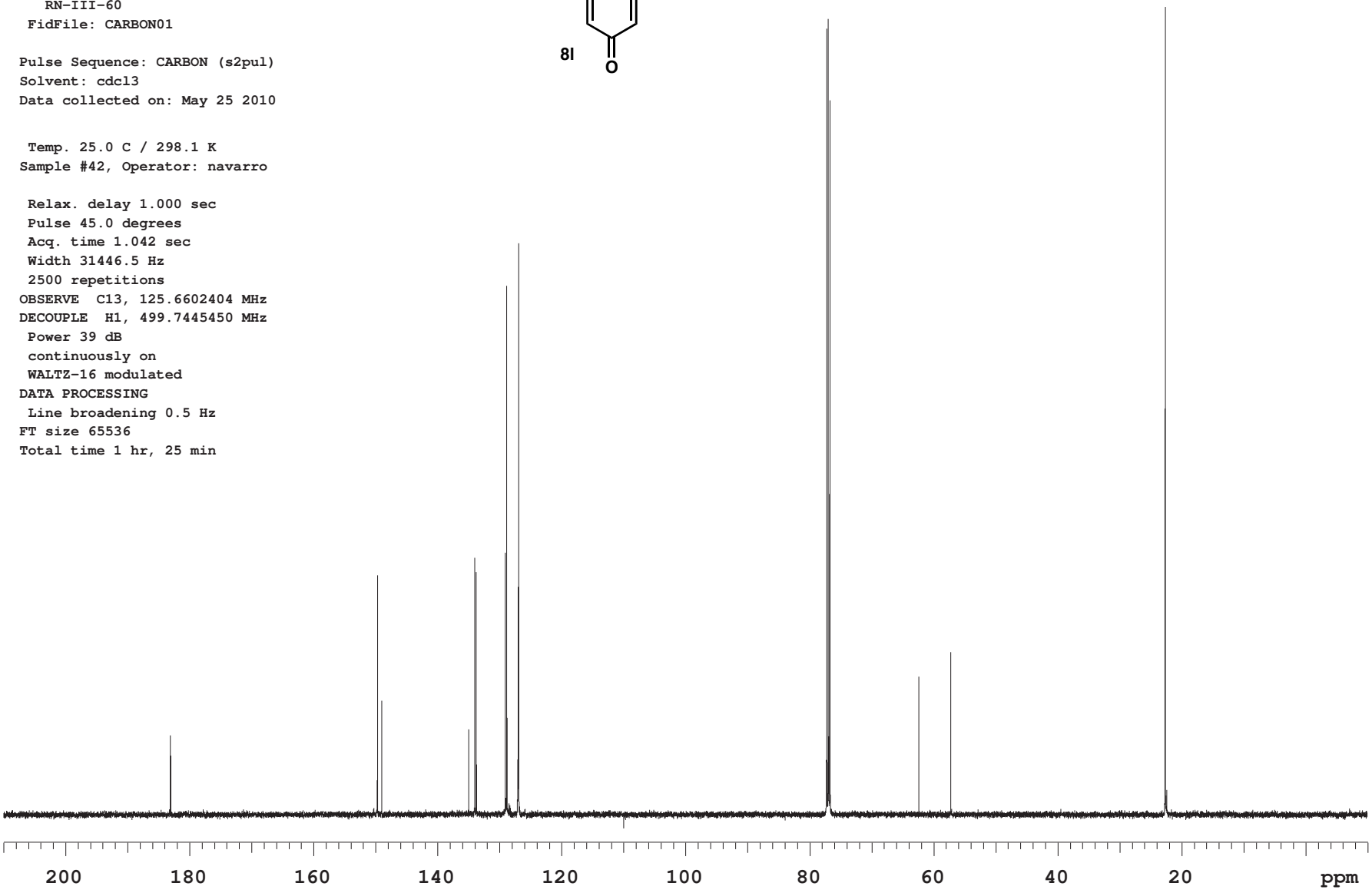
Pulse Sequence: CARBON (s2pul)
Solvent: cdcl3
Data collected on: May 25 2010

Temp. 25.0 C / 298.1 K
Sample #42, Operator: navarro

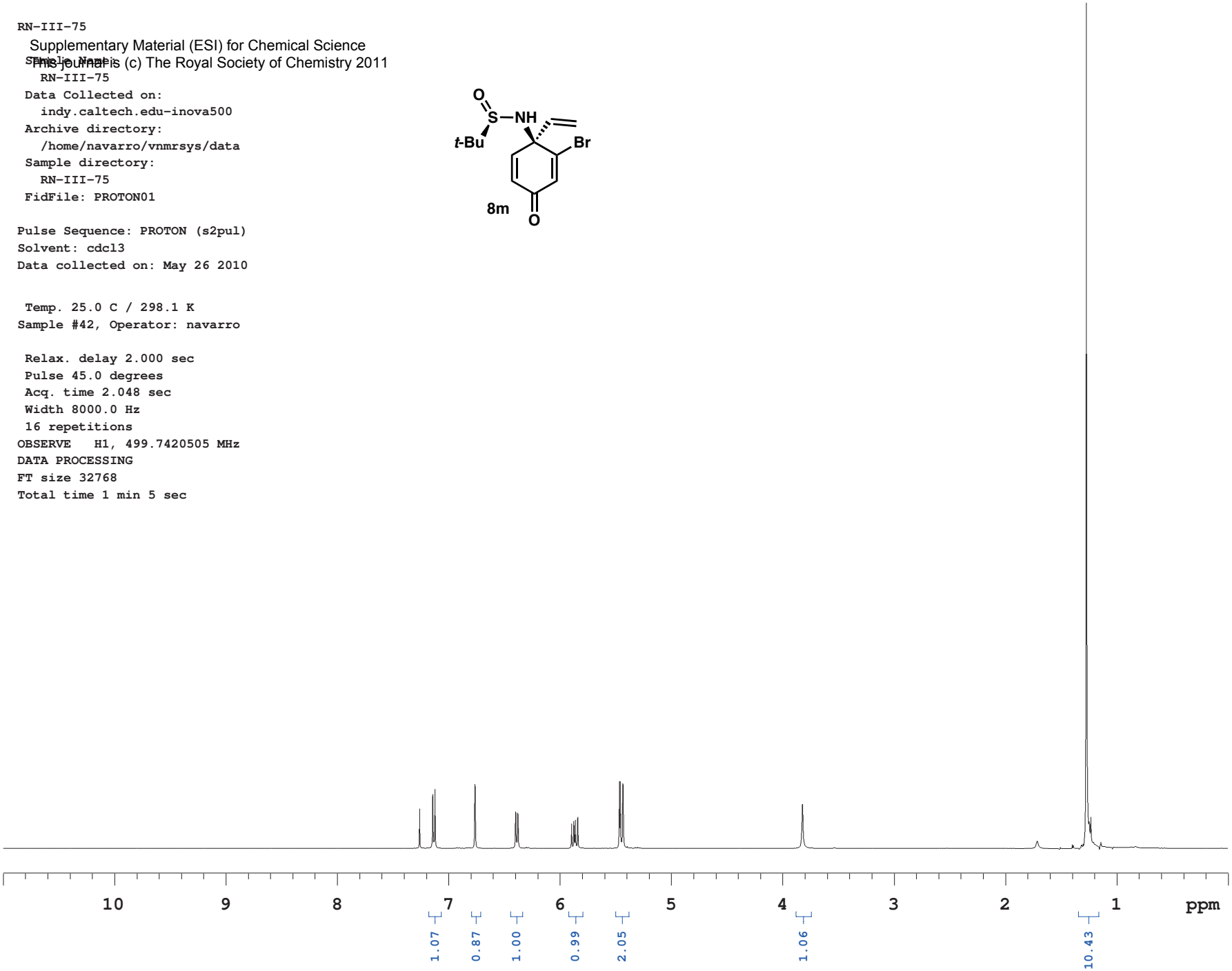
Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.042 sec
Width 31446.5 Hz
2500 repetitions
OBSERVE C13, 125.6602404 MHz
DECOUPLE H1, 499.7445450 MHz
Power 39 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 0.5 Hz
FT size 65536
Total time 1 hr, 25 min



8l



```
RN-III-75
  Sample Name:
    RN-III-75
  Data Collected on:
    indy.caltech.edu-inova500
  Archive directory:
    /home/navarro/vnmrsys/data
  Sample directory:
    RN-III-75
  FidFile: PROTON01

Pulse Sequence: PROTON (s2pul)
Solvent: cdcl3
Data collected on: May 26 2010


  Temp. 25.0 C / 298.1 K
Sample #42, Operator: navarro

  Relax. delay 2.000 sec
  Pulse 45.0 degrees
  Acq. time 2.048 sec
  Width 8000.0 Hz
  16 repetitions
OBSERVE   H1, 499.7420505 MHz
DATA PROCESSING
FT size 32768
Total time 1 min 5 sec
```

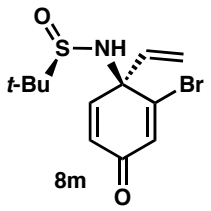



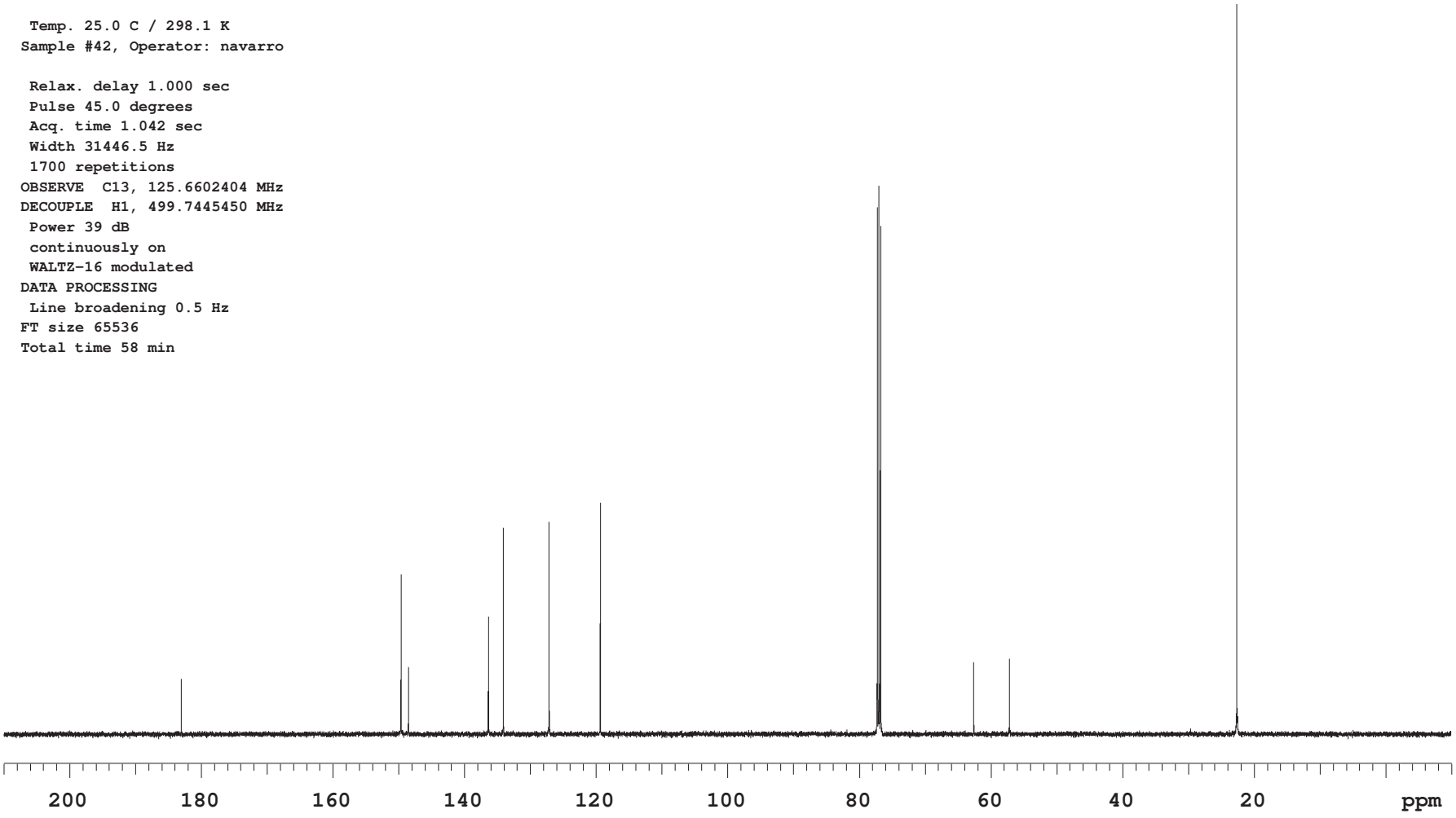
Sample Name:
RN-III-75
Data Collected on:
indy.caltech.edu-inova500
Archive directory:
/home/navarro/vnmrsys/data
Sample directory:
RN-III-75
FidFile: CARBON01

Pulse Sequence: CARBON (s2pul)
Solvent: cdcl3
Data collected on: May 26 2010

Temp. 25.0 C / 298.1 K
Sample #42, Operator: navarro

Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.042 sec
Width 31446.5 Hz
1700 repetitions
OBSERVE C13, 125.6602404 MHz
DECOUPLE H1, 499.7445450 MHz
Power 39 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 0.5 Hz
FT size 65536
Total time 58 min





RN-III-20-2

Sample Name:
RN-III-20-2
Data Collected on:
indy.caltech.edu-inova500
Archive directory:
/home/navarro/vnmrsys/data
Sample directory:
RN-III-20-2
FidFile: PROTON01

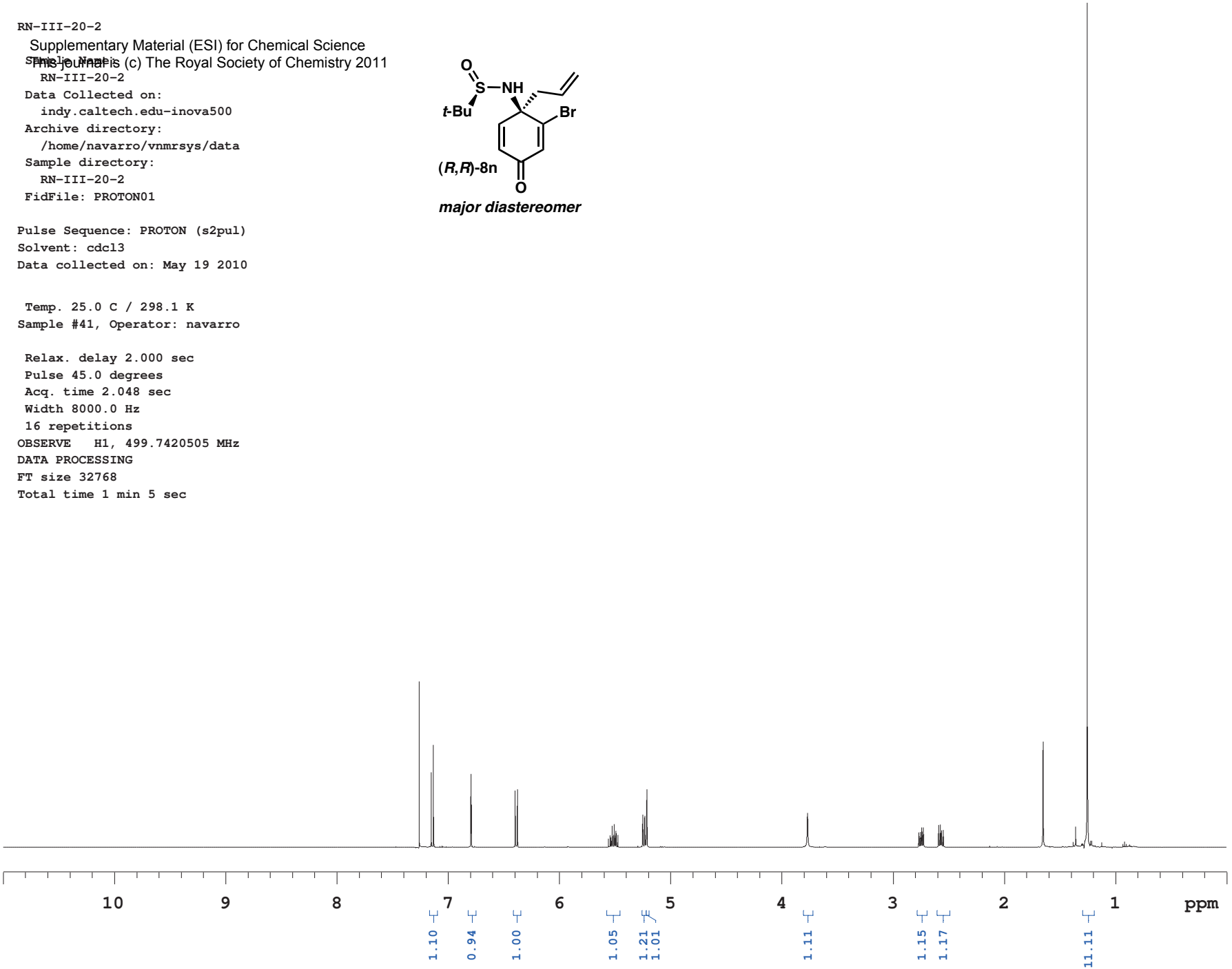
Pulse Sequence: PROTON (s2pul)
Solvent: cdcl3
Data collected on: May 19 2010

Temp. 25.0 C / 298.1 K
Sample #41, Operator: navarro

Relax. delay 2.000 sec
Pulse 45.0 degrees
Acq. time 2.048 sec
Width 8000.0 Hz
16 repetitions
OBSERVE H1, 499.7420505 MHz
DATA PROCESSING
FT size 32768
Total time 1 min 5 sec

(*R*,*R*)-8n

major diastereomer

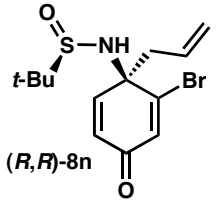


Sample Name:
RN-III-20-2
Data Collected on:
indy.caltech.edu-inova500
Archive directory:
/home/navarro/vnmrsys/data
Sample directory:
RN-III-20-2
FidFile: CARBON01

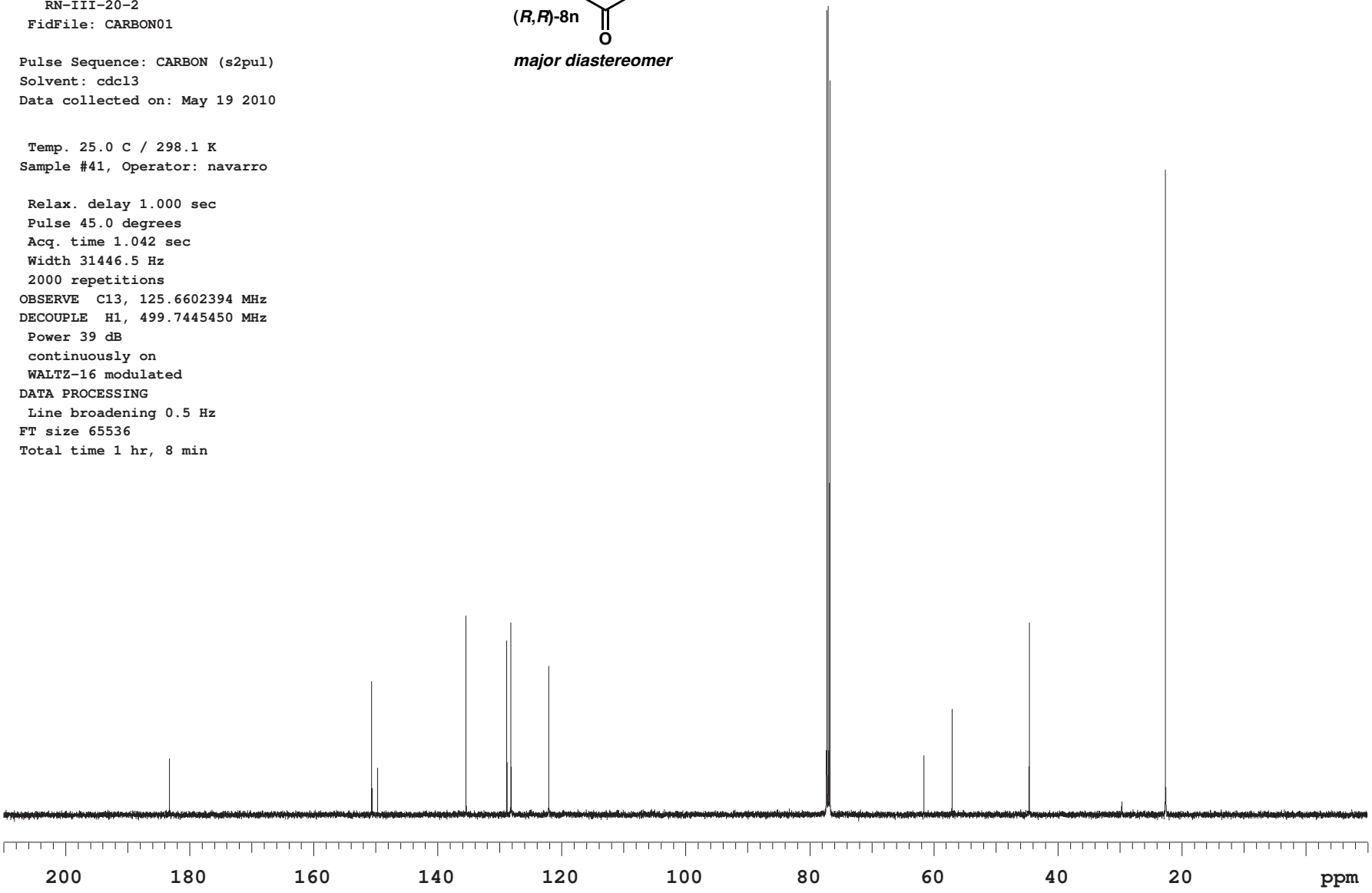
Pulse Sequence: CARBON (s2pul)
Solvent: cdcl3
Data collected on: May 19 2010

Temp. 25.0 C / 298.1 K
Sample #41, Operator: navarro

Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.042 sec
Width 31446.5 Hz
2000 repetitions
OBSERVE C13, 125.6602394 MHz
DECOUPLE H1, 499.7445450 MHz
Power 39 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 0.5 Hz
FT size 65536
Total time 1 hr, 8 min

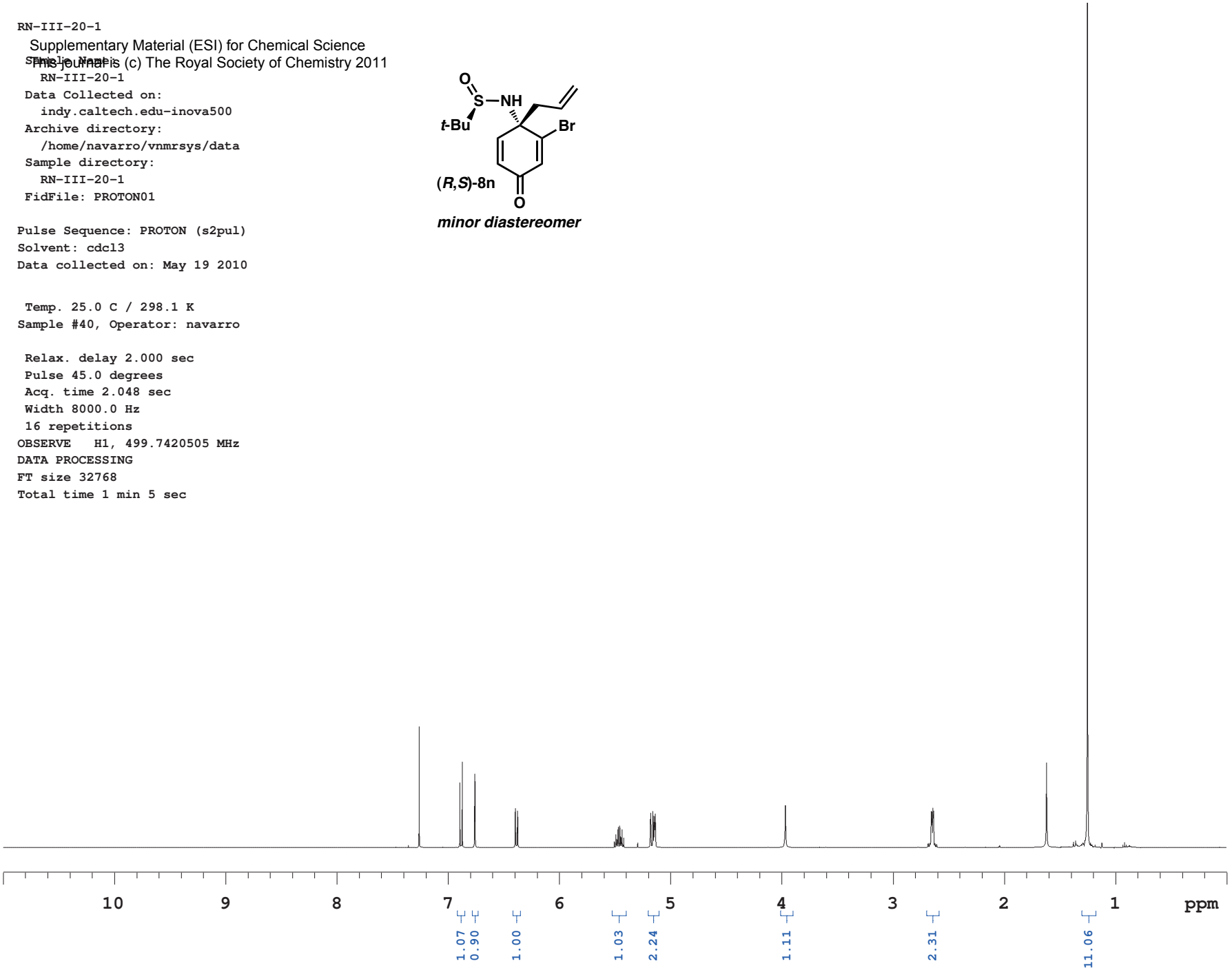


major diastereomer

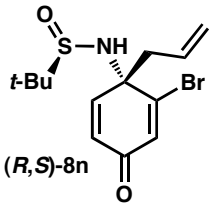


```
RN-III-20-1
 Sample Name:
   RN-III-20-1
 Data Collected on:
   indy.caltech.edu-inova500
 Archive directory:
   /home/navarro/vnmrsys/data
 Sample directory:
   RN-III-20-1
 FidFile: PROTON01

Pulse Sequence: PROTON (s2pul)
Solvent: cdcl3
Data collected on: May 19 2010


 Temp. 25.0 C / 298.1 K
Sample #40, Operator: navarro

 Relax. delay 2.000 sec
 Pulse 45.0 degrees
 Acq. time 2.048 sec
 Width 8000.0 Hz
 16 repetitions
OBSERVE   H1, 499.7420505 MHz
DATA PROCESSING
FT size 32768
Total time 1 min 5 sec
```

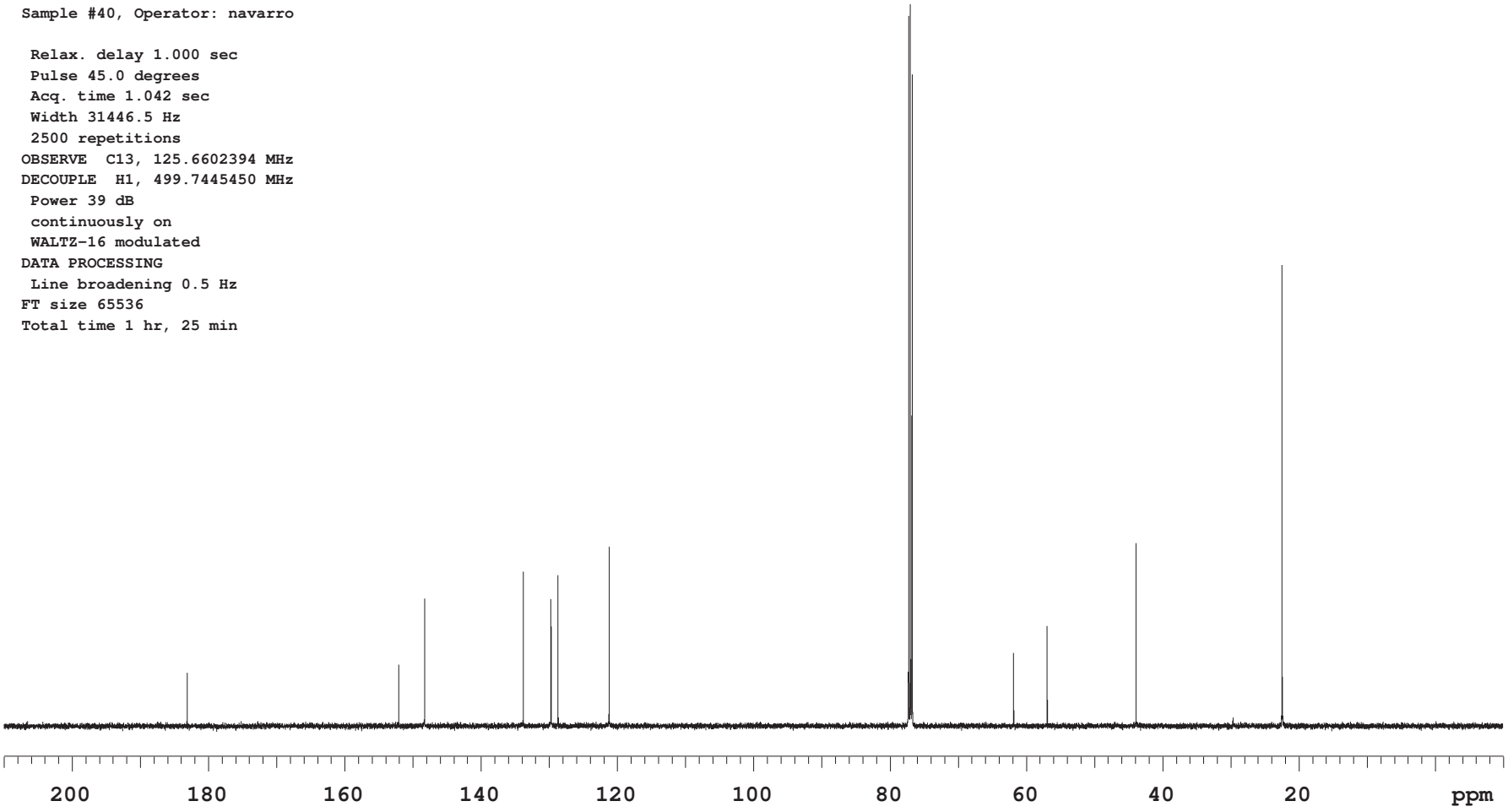

minor diastereomer



```
Sample Name:
  RN-III-20-1
Data Collected on:
  indy.caltech.edu-inova500
Archive directory:
  /home/navarro/vnmrsys/data
Sample directory:
  RN-III-20-1
FidFile: CARBON01

Pulse Sequence: CARBON (s2pul)
Solvent: cdcl3
Data collected on: May 19 2010


Temp. 25.0 C / 298.1 K
Sample #40, Operator: navarro

Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.042 sec
Width 31446.5 Hz
2500 repetitions
OBSERVE  C13, 125.6602394 MHz
DECOUPLE  H1, 499.7445450 MHz
Power 39 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 0.5 Hz
FT size 65536
Total time 1 hr, 25 min
```



minor diastereomer



RN-III-221
purified

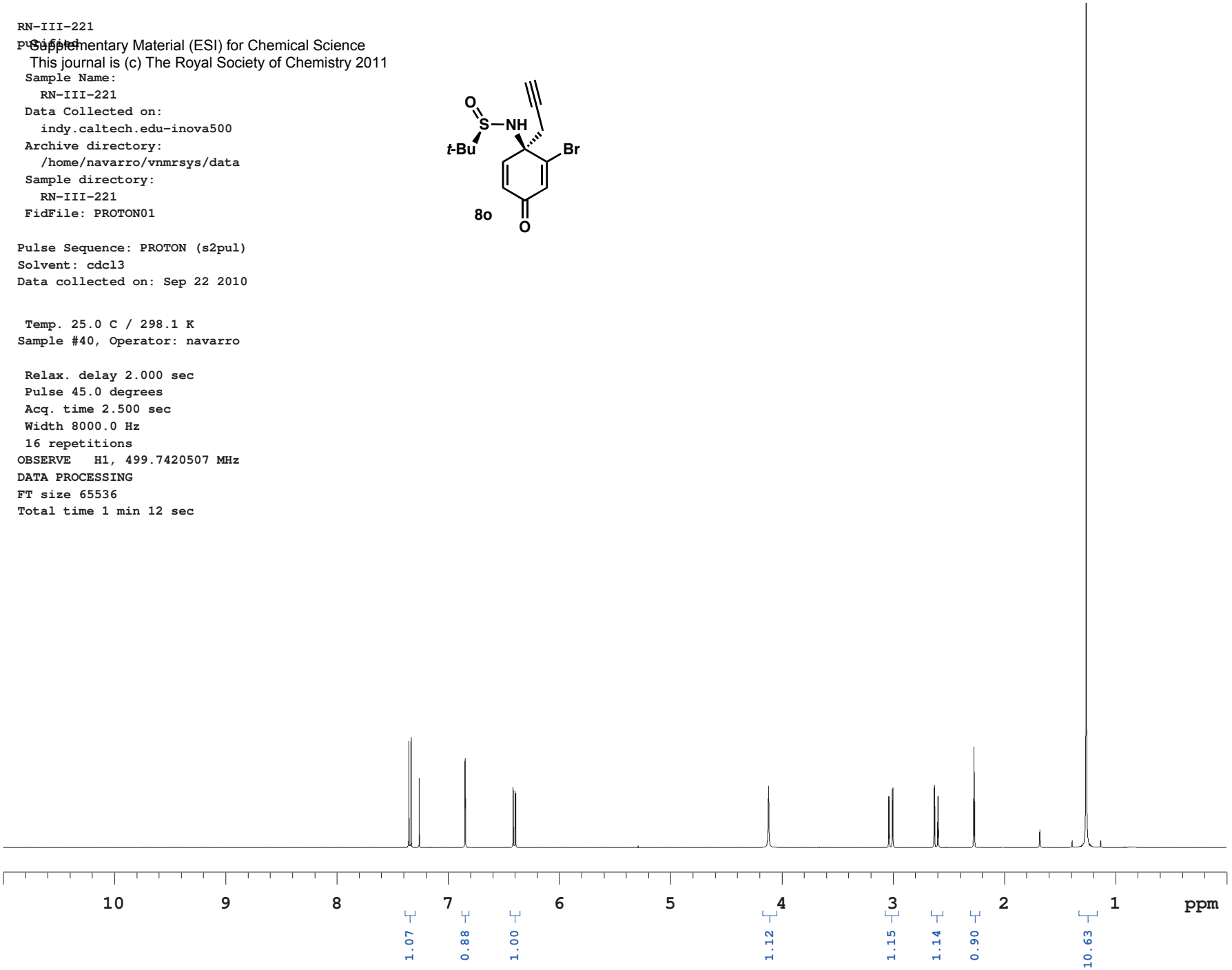
Sample Name:
RN-III-221
Data Collected on:
indy.caltech.edu-inova500
Archive directory:
/home/navarro/vnmrsys/data
Sample directory:
RN-III-221
FidFile: PROTON01

Pulse Sequence: PROTON (s2pul)
Solvent: cdcl3
Data collected on: Sep 22 2010

Temp. 25.0 C / 298.1 K
Sample #40, Operator: navarro

Relax. delay 2.000 sec
Pulse 45.0 degrees
Acq. time 2.500 sec
Width 8000.0 Hz
16 repetitions
OBSERVE H1, 499.7420507 MHz
DATA PROCESSING
FT size 65536
Total time 1 min 12 sec

8o



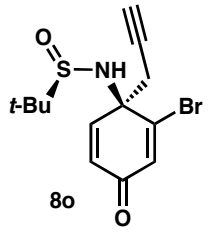
RN-III-221
purified

Sample Name:
RN-III-221
Data Collected on:
indy.caltech.edu-inova500
Archive directory:
/home/navarro/vnmrsys/data
Sample directory:
RN-III-221
FidFile: CARBON01

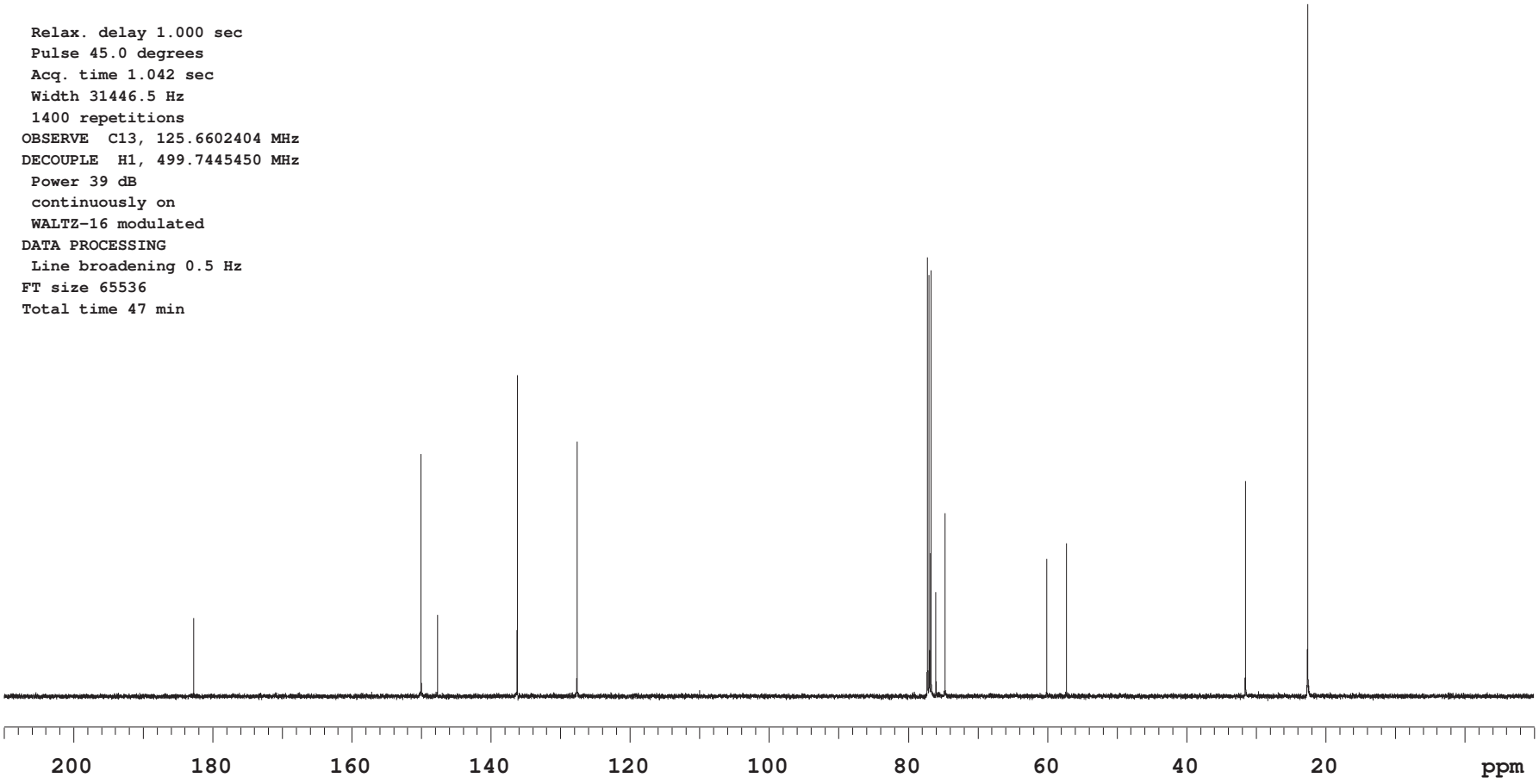
Pulse Sequence: CARBON (s2pul)
Solvent: cdcl3
Data collected on: Sep 22 2010

Temp. 25.0 C / 298.1 K
Sample #40, Operator: navarro

Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.042 sec
Width 31446.5 Hz
1400 repetitions
OBSERVE C13, 125.6602404 MHz
DECOUPLE H1, 499.7445450 MHz
Power 39 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 0.5 Hz
FT size 65536
Total time 47 min



8o



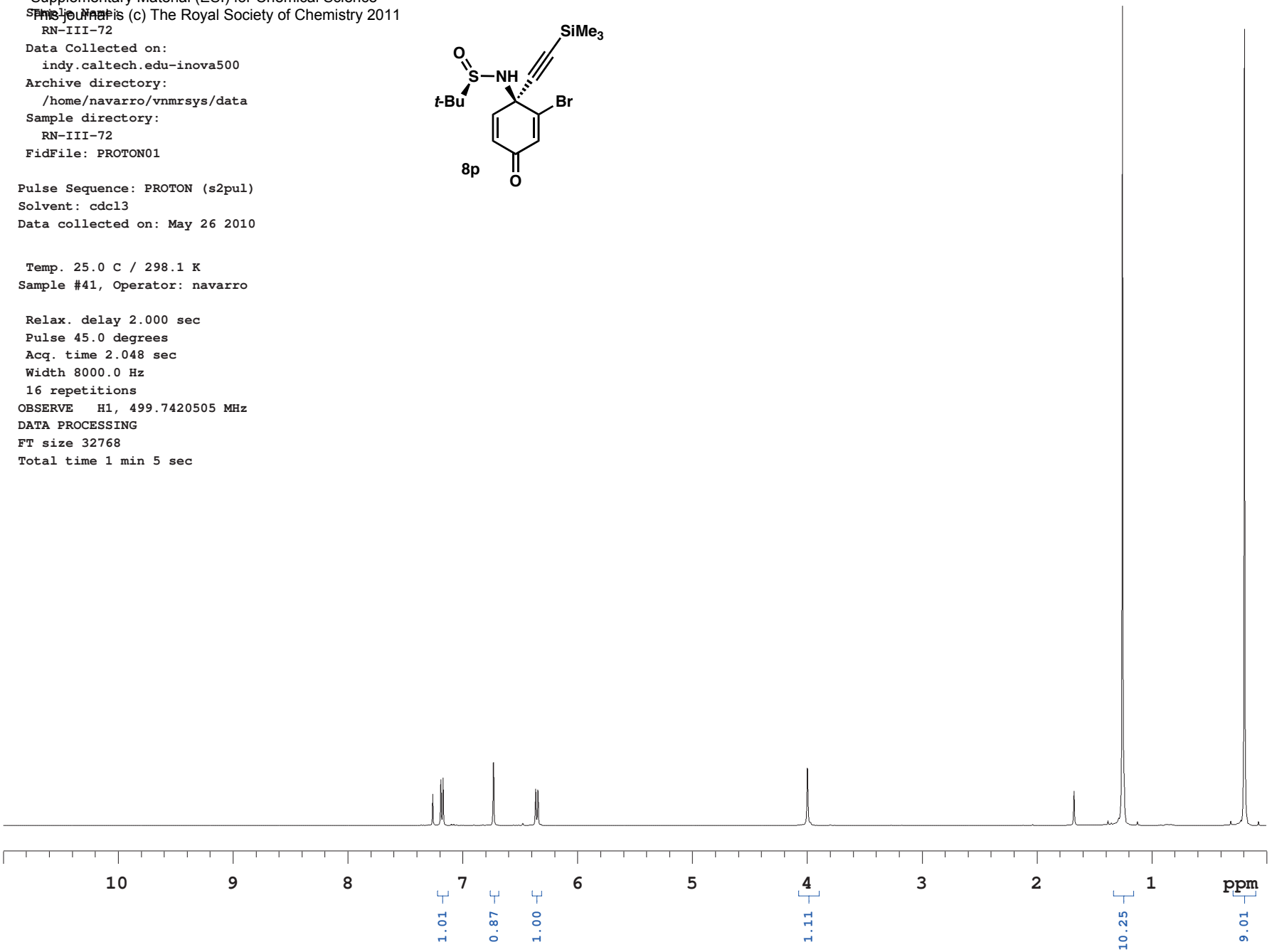
Sample Name:
RN-III-72
Data Collected on:
indy.caltech.edu-inova500
Archive directory:
/home/navarro/vnmrsys/data
Sample directory:
RN-III-72
FidFile: PROTON01

Pulse Sequence: PROTON (s2pul)
Solvent: cdcl3
Data collected on: May 26 2010

Temp. 25.0 C / 298.1 K
Sample #41, Operator: navarro

Relax. delay 2.000 sec
Pulse 45.0 degrees
Acq. time 2.048 sec
Width 8000.0 Hz
16 repetitions
OBSERVE H1, 499.7420505 MHz
DATA PROCESSING
FT size 32768
Total time 1 min 5 sec

8p



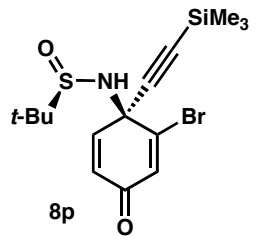
RN-III-86
purified

Sample Name:
RN-III-86
Data Collected on:
indy.caltech.edu-inova500
Archive directory:
/home/navarro/vnmrsys/data
Sample directory:
RN-III-86
FidFile: CARBON01

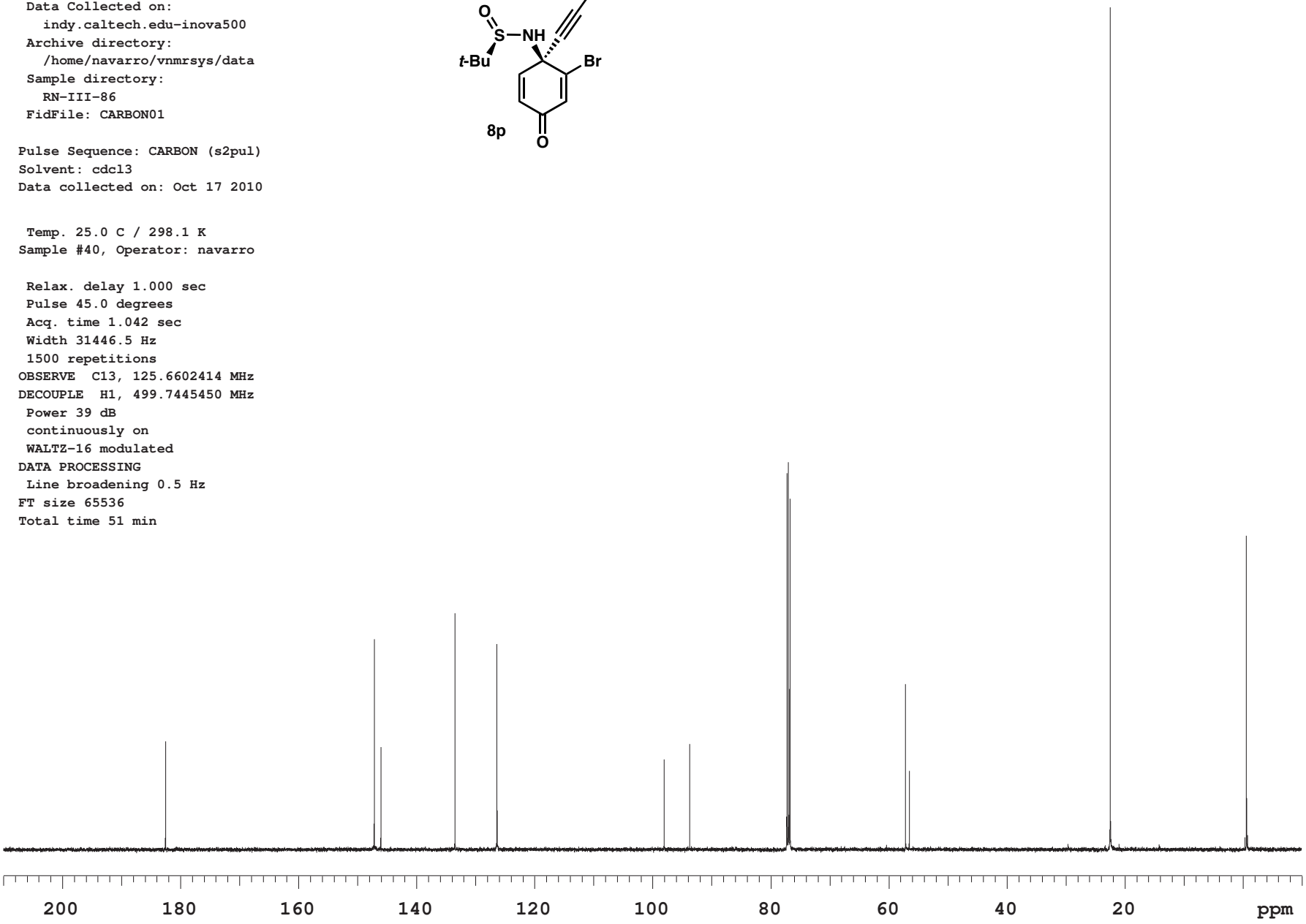
Pulse Sequence: CARBON (s2pul)
Solvent: cdcl3
Data collected on: Oct 17 2010

Temp. 25.0 C / 298.1 K
Sample #40, Operator: navarro

Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.042 sec
Width 31446.5 Hz
1500 repetitions
OBSERVE C13, 125.6602414 MHz
DECOUPLE H1, 499.7445450 MHz
Power 39 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 0.5 Hz
FT size 65536
Total time 51 min



8p



RN-III-174
purified

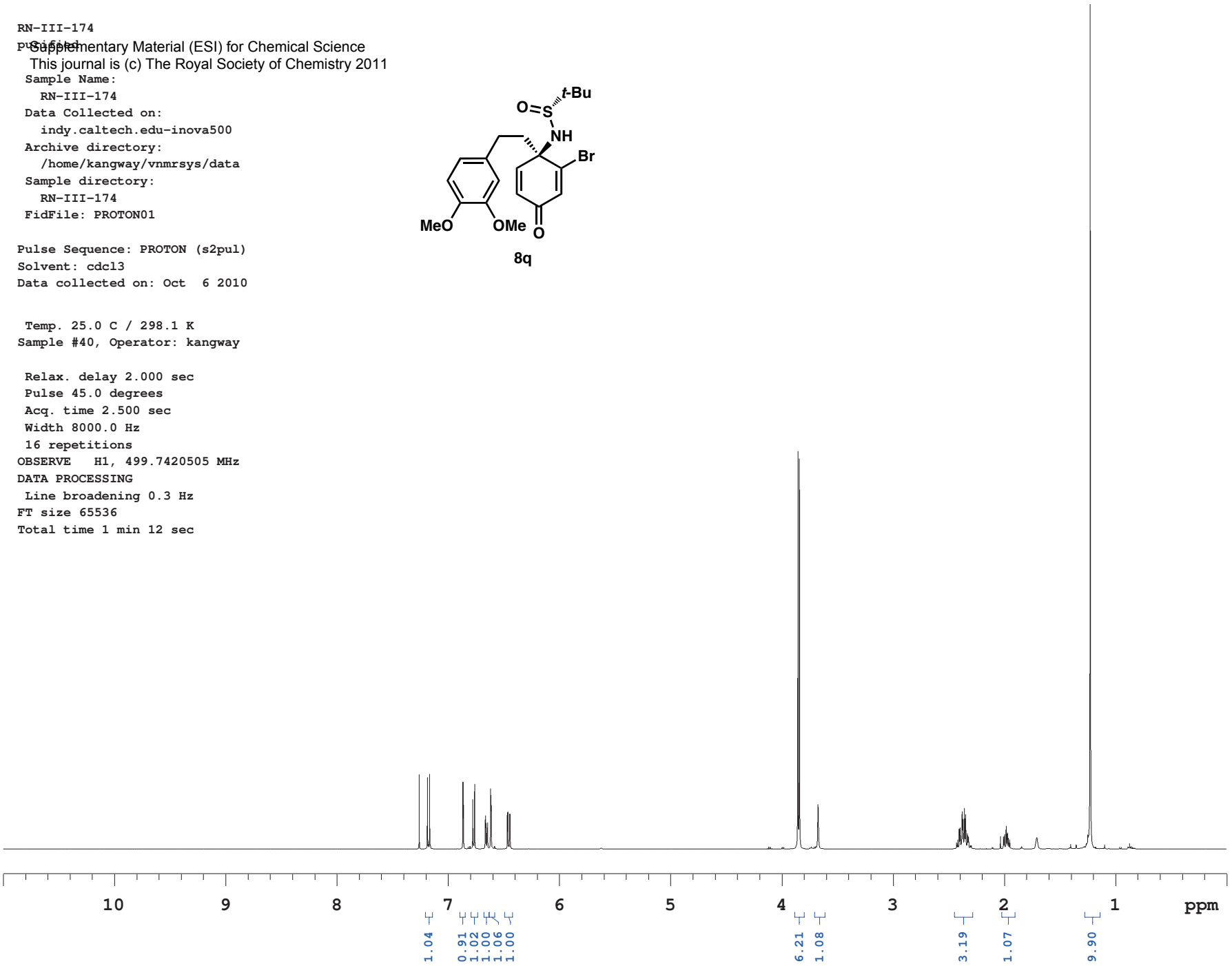
Sample Name:
RN-III-174
Data Collected on:
indy.caltech.edu-inova500
Archive directory:
/home/kangway/vnmrsys/data
Sample directory:
RN-III-174
FidFile: PROTON01

Pulse Sequence: PROTON (s2pul)
Solvent: cdcl3
Data collected on: Oct 6 2010

Temp. 25.0 C / 298.1 K
Sample #40, Operator: kangway

Relax. delay 2.000 sec
Pulse 45.0 degrees
Acq. time 2.500 sec
Width 8000.0 Hz
16 repetitions
OBSERVE H1, 499.7420505 MHz
DATA PROCESSING
Line broadening 0.3 Hz
FT size 65536
Total time 1 min 12 sec

8q



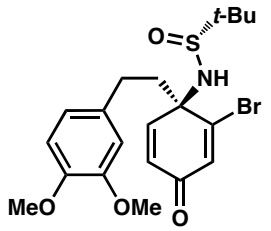
RN-III-174
purified

Sample Name:
RN-III-174
Data Collected on:
indy.caltech.edu-inova500
Archive directory:
/home/kangway/vnmrsys/data
Sample directory:
RN-III-174
FidFile: CARBON01

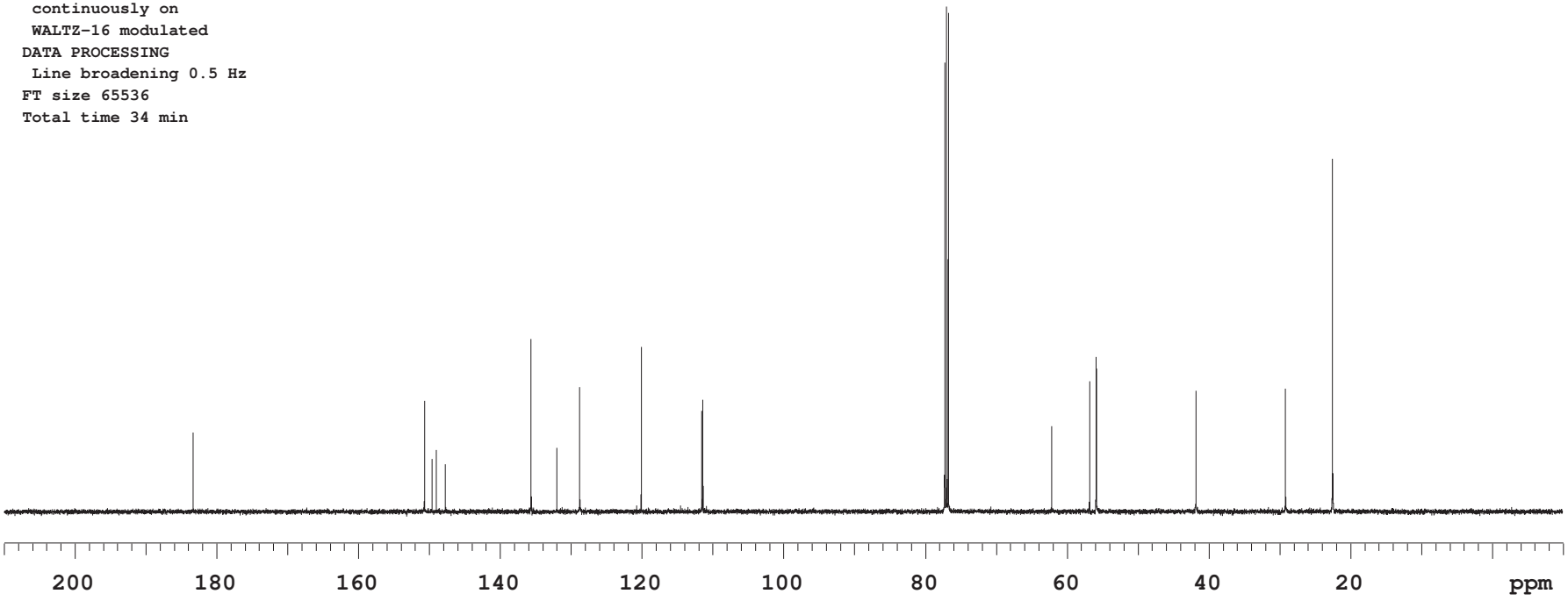
Pulse Sequence: CARBON (s2pul)
Solvent: cdcl3
Data collected on: Oct 6 2010

Temp. 25.0 C / 298.1 K
Sample #40, Operator: kangway

Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.042 sec
Width 31446.5 Hz
1000 repetitions
OBSERVE C13, 125.6602414 MHz
DECOUPLE H1, 499.7445450 MHz
Power 39 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 0.5 Hz
FT size 65536
Total time 34 min



8q

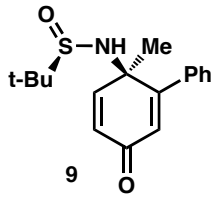


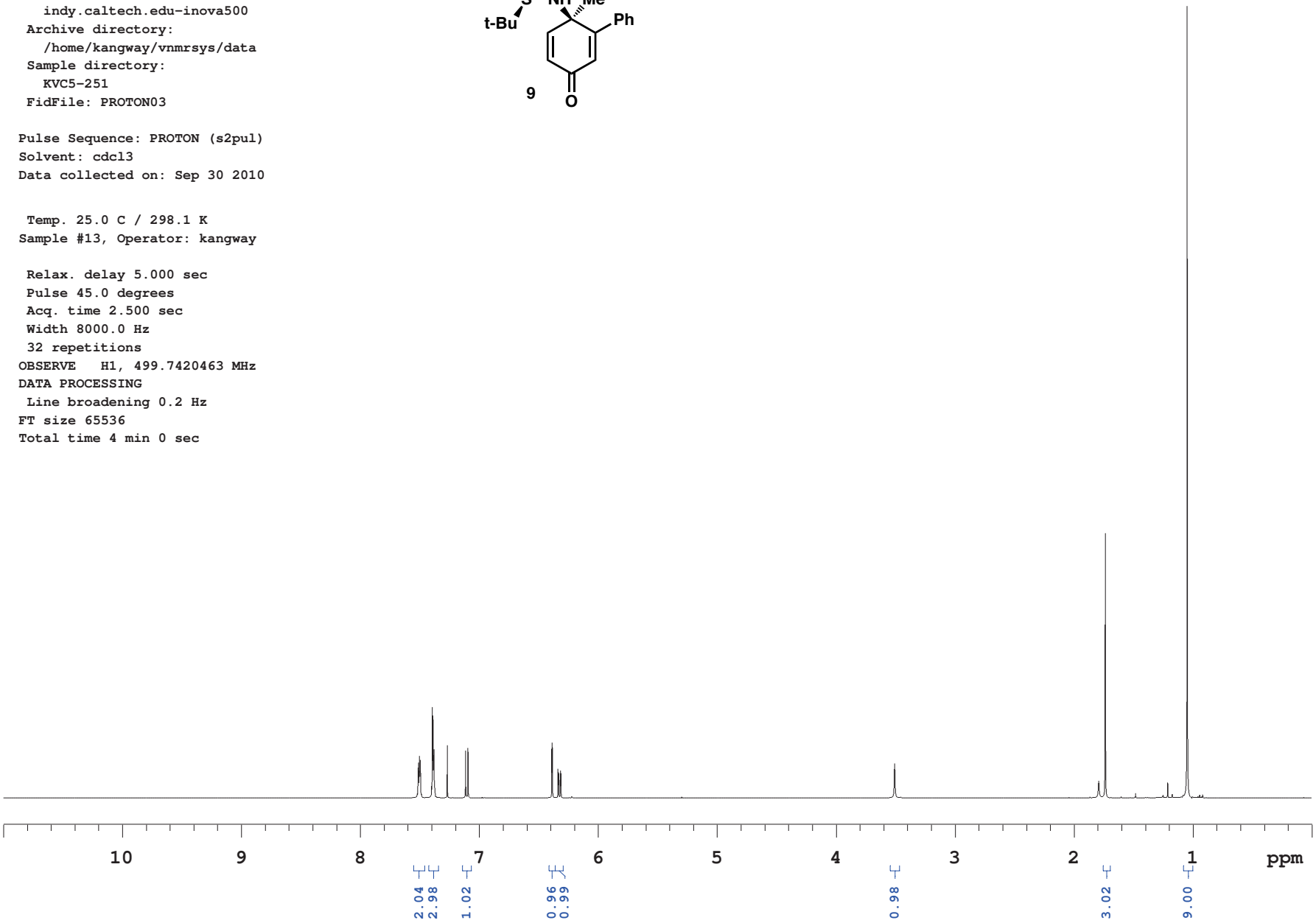
Sample Name:
KVC5-251
Data Collected on:
indy.caltech.edu-inova500
Archive directory:
/home/kangway/vnmrsys/data
Sample directory:
KVC5-251
FidFile: PROTON03

Pulse Sequence: PROTON (s2pul)
Solvent: cdcl3
Data collected on: Sep 30 2010

Temp. 25.0 C / 298.1 K
Sample #13, Operator: kangway

Relax. delay 5.000 sec
Pulse 45.0 degrees
Acq. time 2.500 sec
Width 8000.0 Hz
32 repetitions
OBSERVE H1, 499.7420463 MHz
DATA PROCESSING
Line broadening 0.2 Hz
FT size 65536
Total time 4 min 0 sec



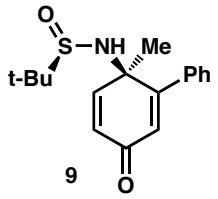


Sample Name:
KVC5-251
Data Collected on:
indy.caltech.edu-inova500
Archive directory:
/home/kangway/vnmrsys/data
Sample directory:
KVC5-251
FidFile: CARBON02

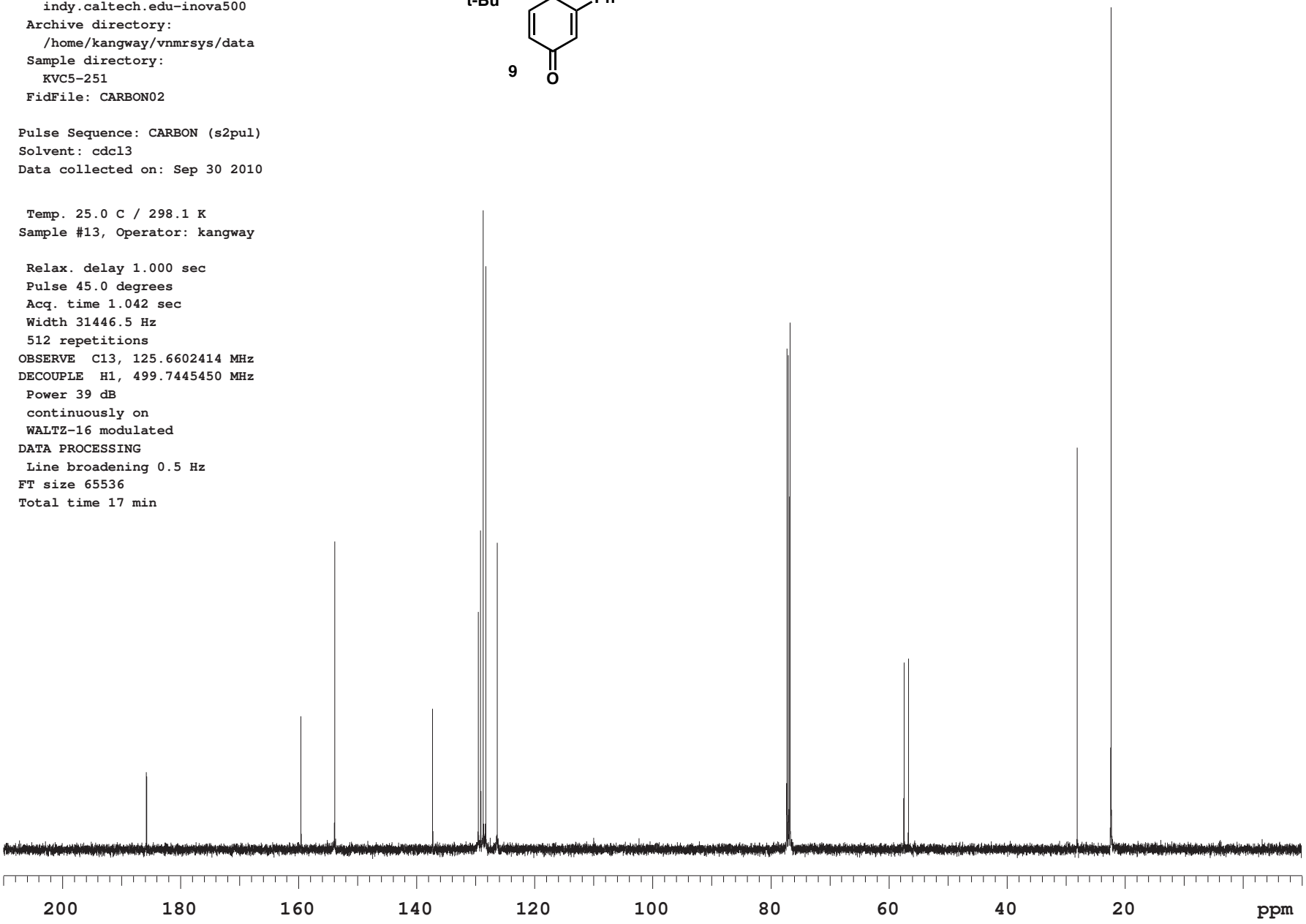
Pulse Sequence: CARBON (s2pul)
Solvent: cdcl3
Data collected on: Sep 30 2010

Temp. 25.0 C / 298.1 K
Sample #13, Operator: kangway

Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.042 sec
Width 31446.5 Hz
512 repetitions
OBSERVE C13, 125.6602414 MHz
DECOUPLE H1, 499.7445450 MHz
Power 39 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 0.5 Hz
FT size 65536
Total time 17 min



9

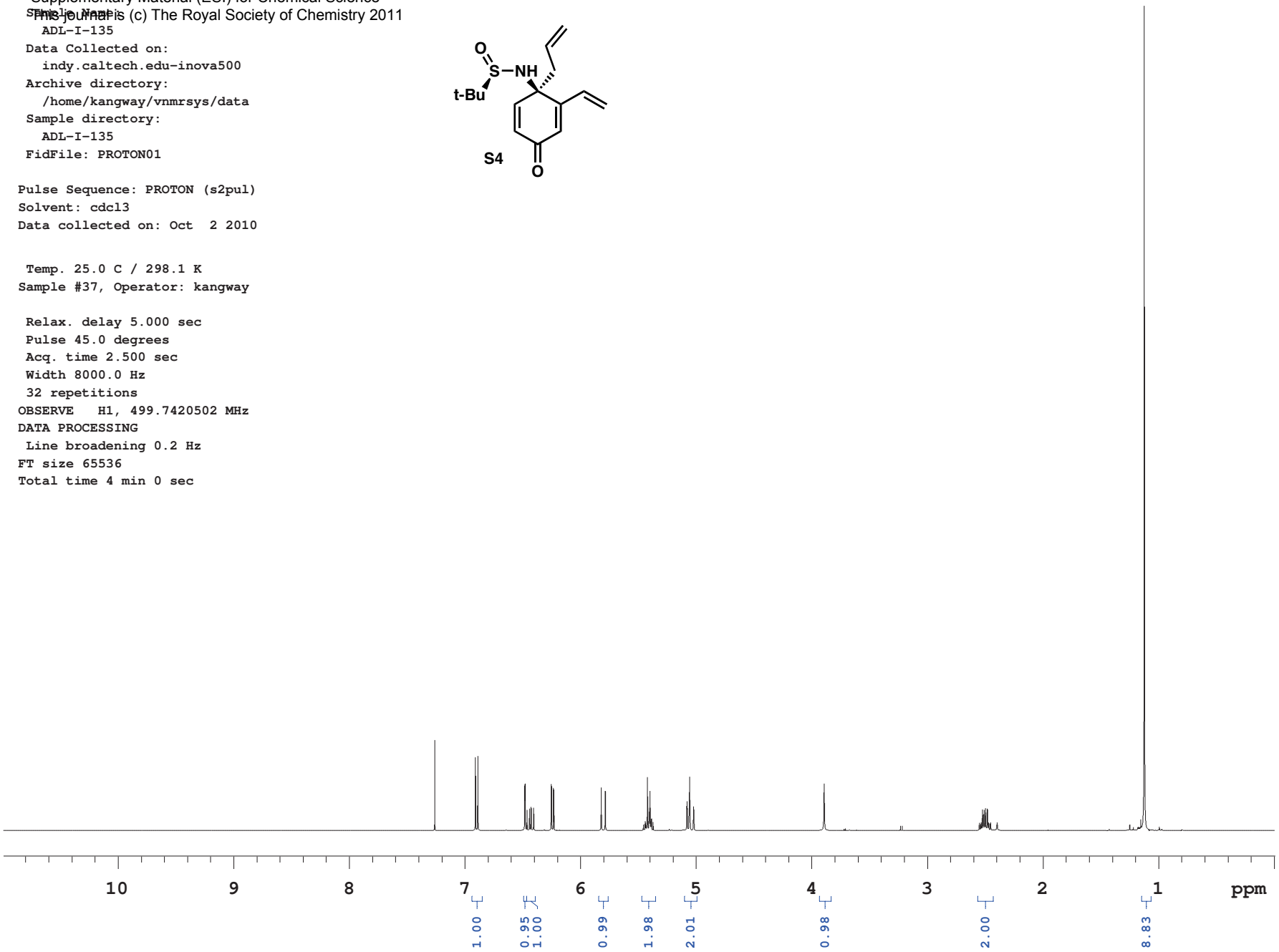


Sample Name:
ADL-I-135
Data Collected on:
indy.caltech.edu-inova500
Archive directory:
/home/kangway/vnmrsys/data
Sample directory:
ADL-I-135
FidFile: PROTON01

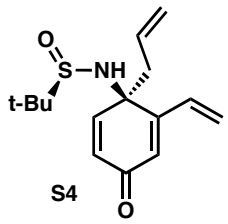
Pulse Sequence: PROTON (s2pul)
Solvent: cdcl3
Data collected on: Oct 2 2010

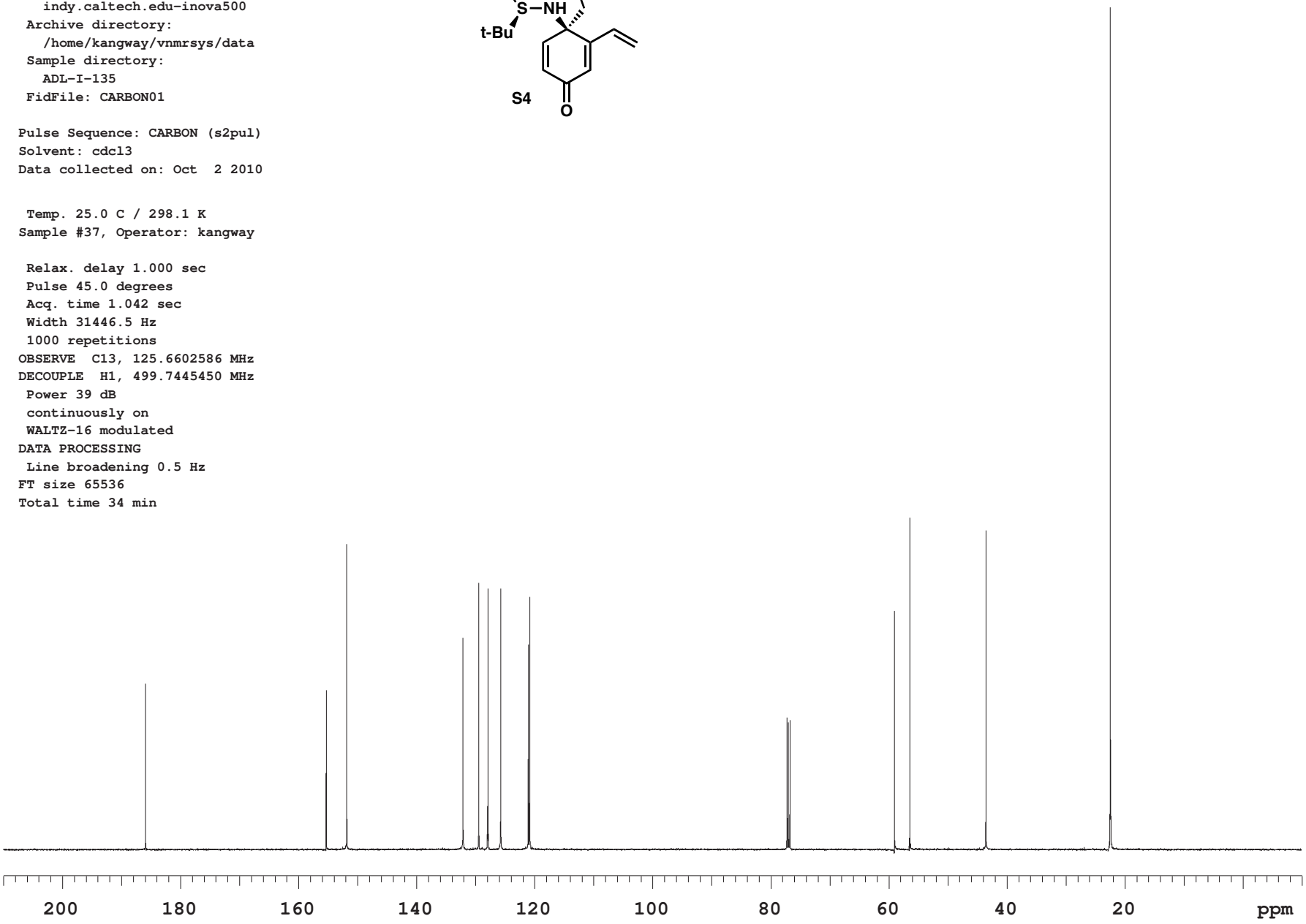
Temp. 25.0 C / 298.1 K
Sample #37, Operator: kangway

Relax. delay 5.000 sec
Pulse 45.0 degrees
Acq. time 2.500 sec
Width 8000.0 Hz
32 repetitions
OBSERVE H1, 499.7420502 MHz
DATA PROCESSING
Line broadening 0.2 Hz
FT size 65536
Total time 4 min 0 sec



```
ADL-I-135
Sample Name:
  ADL-I-135
Data Collected on:
  indy.caltech.edu-inova500
Archive directory:
  /home/kangway/vnmrsys/data
Sample directory:
  ADL-I-135
FidFile: CARBON01

Pulse Sequence: CARBON (s2pul)
Solvent: cdcl3
Data collected on: Oct  2 2010


Temp. 25.0 C / 298.1 K
Sample #37, Operator: kangway

Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.042 sec
Width 31446.5 Hz
1000 repetitions
OBSERVE  C13, 125.6602586 MHz
DECOUPLE  H1, 499.7445450 MHz
Power 39 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 0.5 Hz
FT size 65536
Total time 34 min
```





KVC5-217

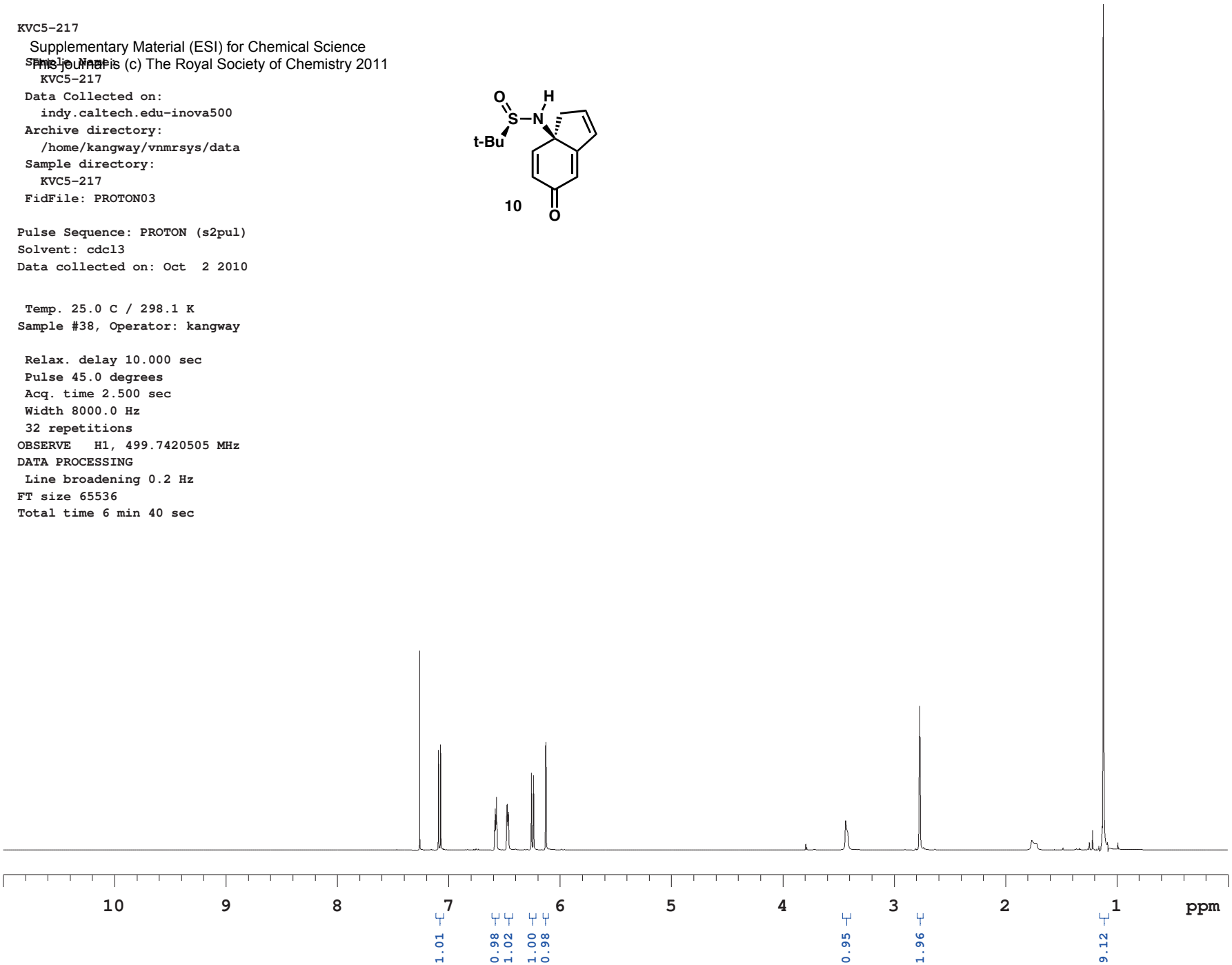
Sample Name:
KVC5-217
Data Collected on:
indy.caltech.edu-inova500
Archive directory:
/home/kangway/vnmrsys/data
Sample directory:
KVC5-217
FidFile: PROTON03

Pulse Sequence: PROTON (s2pul)
Solvent: cdcl3
Data collected on: Oct 2 2010

Temp. 25.0 C / 298.1 K
Sample #38, Operator: kangway

Relax. delay 10.000 sec
Pulse 45.0 degrees
Acq. time 2.500 sec
Width 8000.0 Hz
32 repetitions
OBSERVE H1, 499.7420505 MHz
DATA PROCESSING
Line broadening 0.2 Hz
FT size 65536
Total time 6 min 40 sec

10

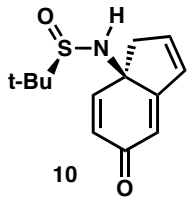


Sample Name:
KVC5-217
Data Collected on:
indy.caltech.edu-inova500
Archive directory:
/home/kangway/vnmrsys/data
Sample directory:
KVC5-217
FidFile: CARBON02

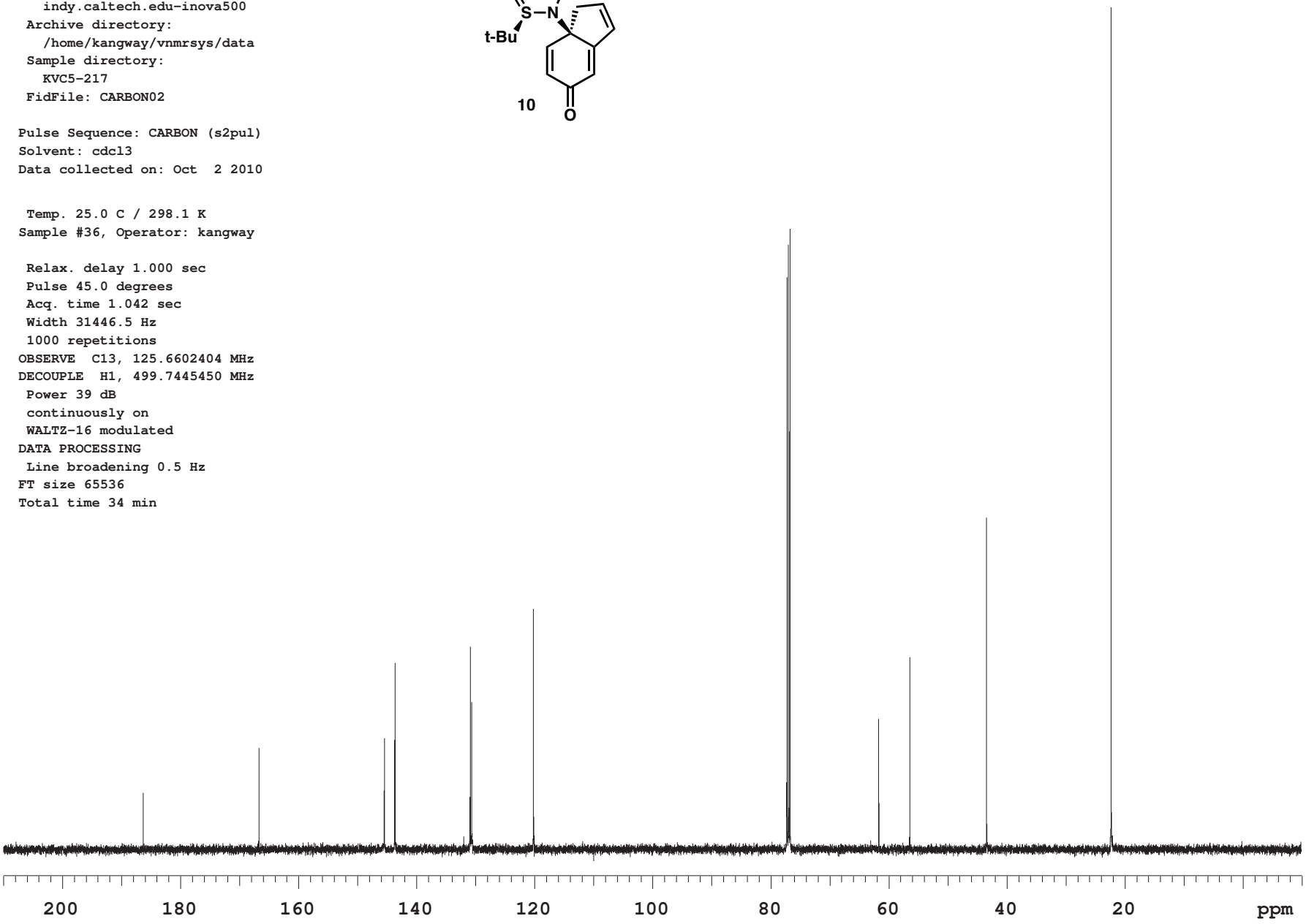
Pulse Sequence: CARBON (s2pul)
Solvent: cdcl3
Data collected on: Oct 2 2010

Temp. 25.0 C / 298.1 K
Sample #36, Operator: kangway

Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.042 sec
Width 31446.5 Hz
1000 repetitions
OBSERVE C13, 125.6602404 MHz
DECOUPLE H1, 499.7445450 MHz
Power 39 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 0.5 Hz
FT size 65536
Total time 34 min



10



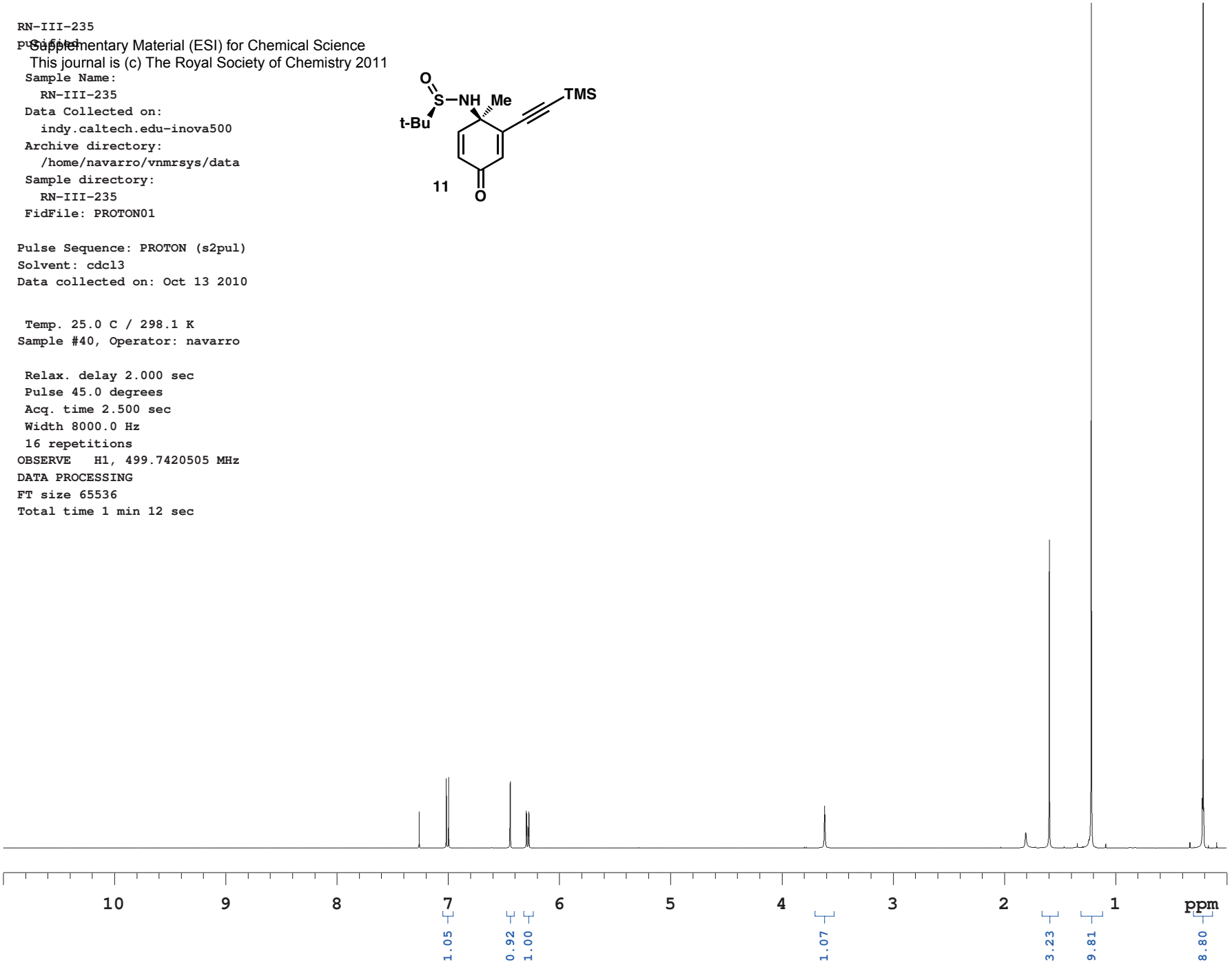
RN-III-235
purified

Sample Name:
RN-III-235
Data Collected on:
indy.caltech.edu-inova500
Archive directory:
/home/navarro/vnmrsys/data
Sample directory:
RN-III-235
FidFile: PROTON01

Pulse Sequence: PROTON (s2pul)
Solvent: cdcl3
Data collected on: Oct 13 2010

Temp. 25.0 C / 298.1 K
Sample #40, Operator: navarro

Relax. delay 2.000 sec
Pulse 45.0 degrees
Acq. time 2.500 sec
Width 8000.0 Hz
16 repetitions
OBSERVE H1, 499.7420505 MHz
DATA PROCESSING
FT size 65536
Total time 1 min 12 sec



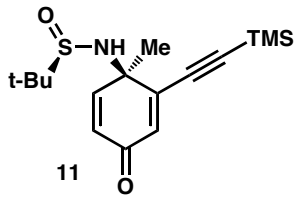
RN-III-235
purified

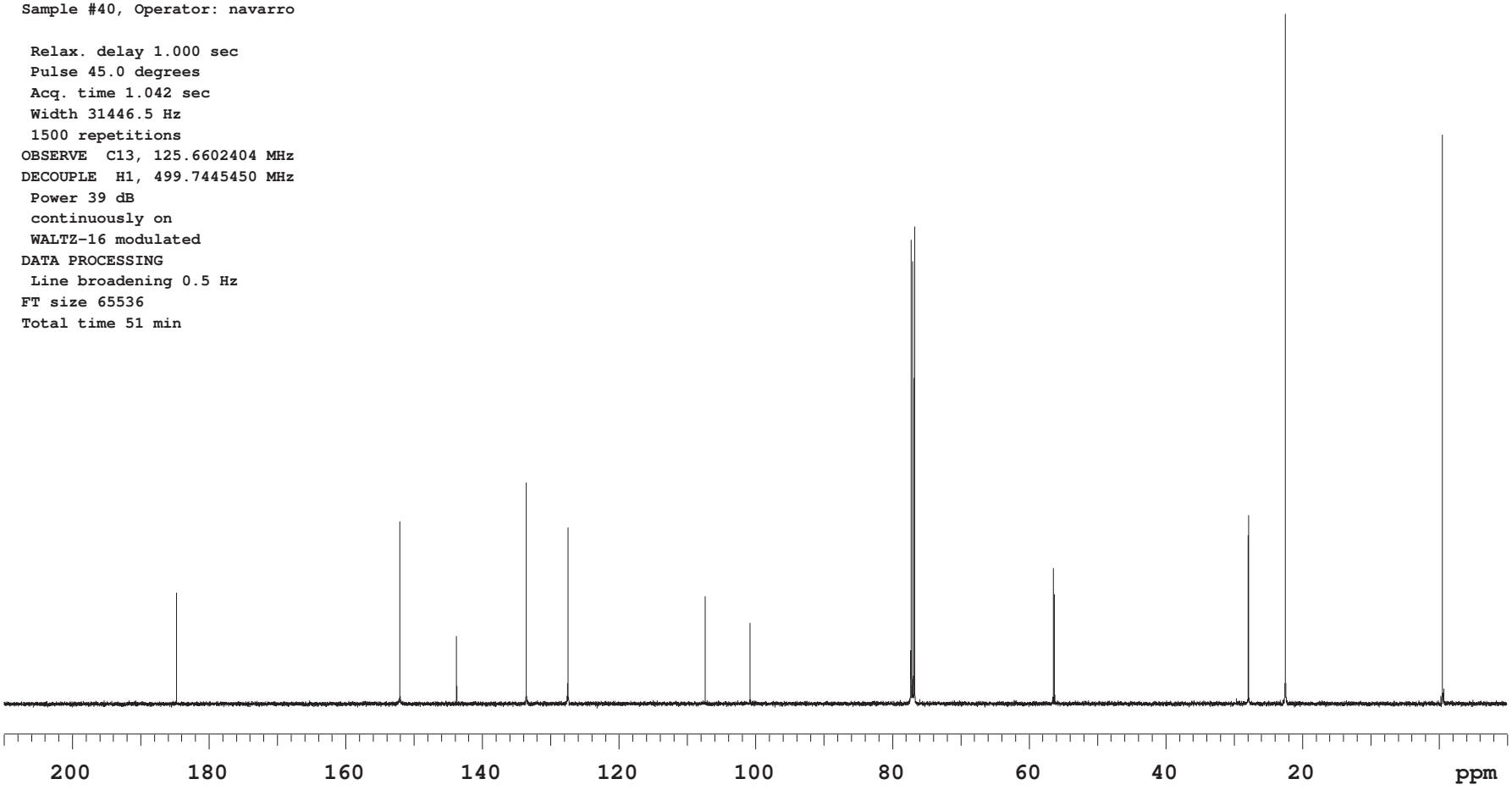
Sample Name:
RN-III-235
Data Collected on:
indy.caltech.edu-inova500
Archive directory:
/home/navarro/vnmrsys/data
Sample directory:
RN-III-235
FidFile: CARBON01

Pulse Sequence: CARBON (s2pul)
Solvent: cdcl3
Data collected on: Oct 13 2010

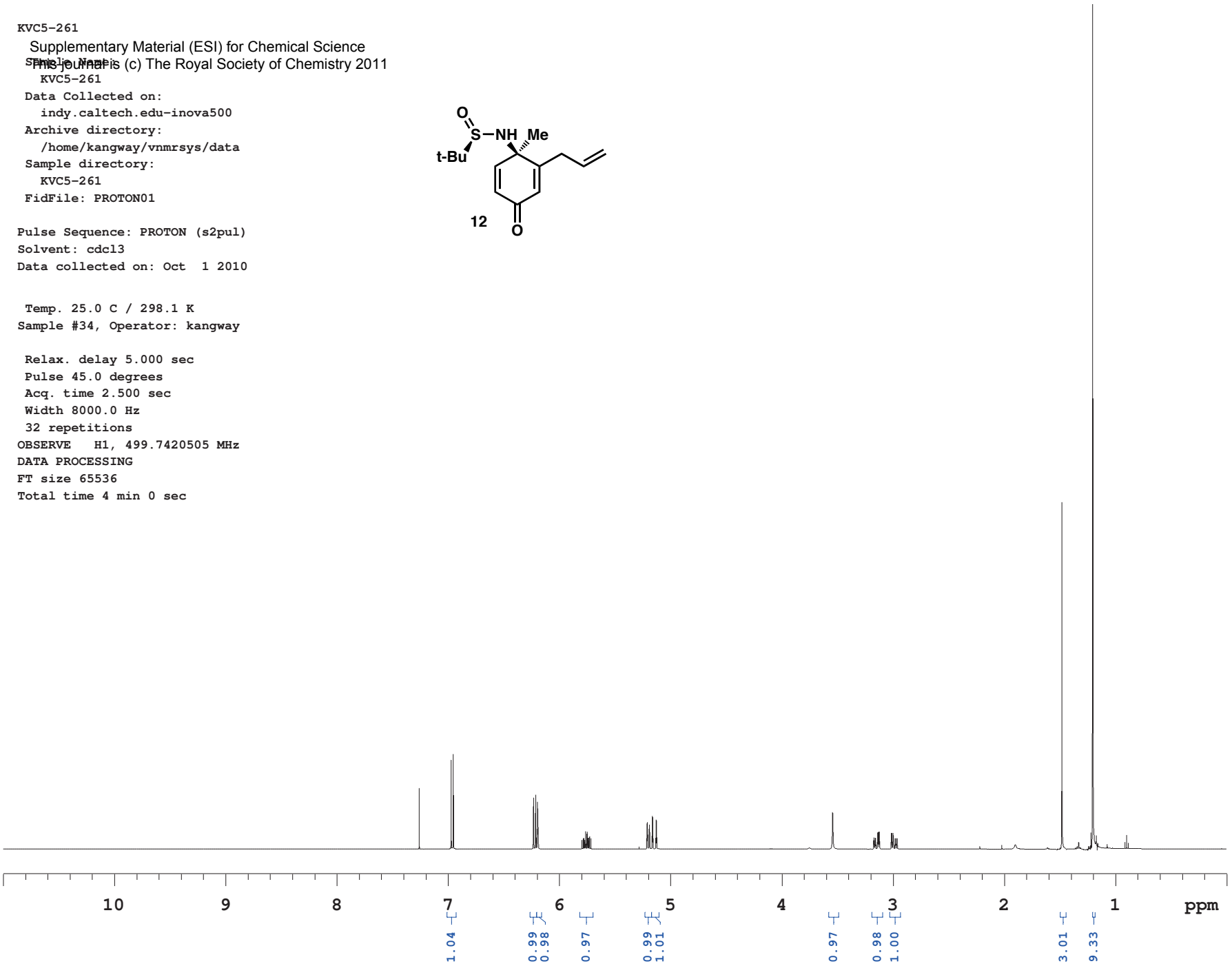
Temp. 25.0 C / 298.1 K
Sample #40, Operator: navarro

Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.042 sec
Width 31446.5 Hz
1500 repetitions
OBSERVE C13, 125.6602404 MHz
DECOUPLE H1, 499.7445450 MHz
Power 39 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 0.5 Hz
FT size 65536
Total time 51 min





```
KVC5-261
Sample Name:
  KVC5-261
Data Collected on:
  indy.caltech.edu-inova500
Archive directory:
  /home/kangway/vnmrsys/data
Sample directory:
  KVC5-261
FidFile: PROTON01

Pulse Sequence: PROTON (s2pul)
Solvent: cdcl3
Data collected on: Oct  1 2010


Temp. 25.0 C / 298.1 K
Sample #34, Operator: kangway

Relax. delay 5.000 sec
Pulse 45.0 degrees
Acq. time 2.500 sec
Width 8000.0 Hz
32 repetitions
OBSERVE   H1, 499.7420505 MHz
DATA PROCESSING
FT size 65536
Total time 4 min 0 sec
```

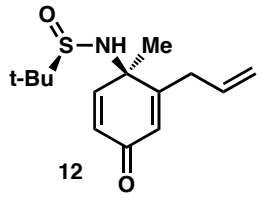



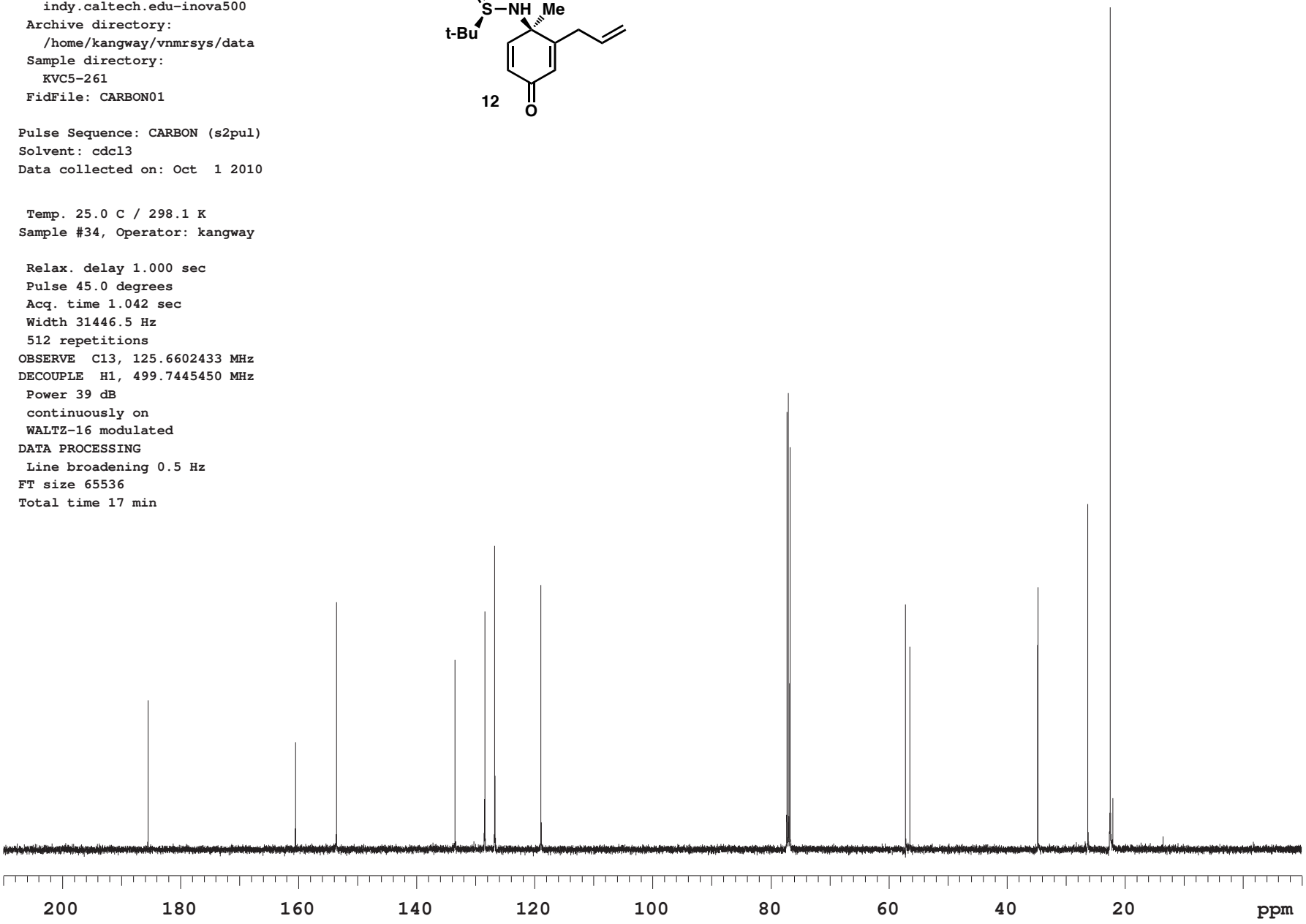
Sample Name:
KVC5-261
Data Collected on:
indy.caltech.edu-inova500
Archive directory:
/home/kangway/vnmrsys/data
Sample directory:
KVC5-261
FidFile: CARBON01

Pulse Sequence: CARBON (s2pul)
Solvent: cdcl3
Data collected on: Oct 1 2010

Temp. 25.0 C / 298.1 K
Sample #34, Operator: kangway

Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.042 sec
Width 31446.5 Hz
512 repetitions
OBSERVE C13, 125.6602433 MHz
DECOUPLE H1, 499.7445450 MHz
Power 39 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 0.5 Hz
FT size 65536
Total time 17 min





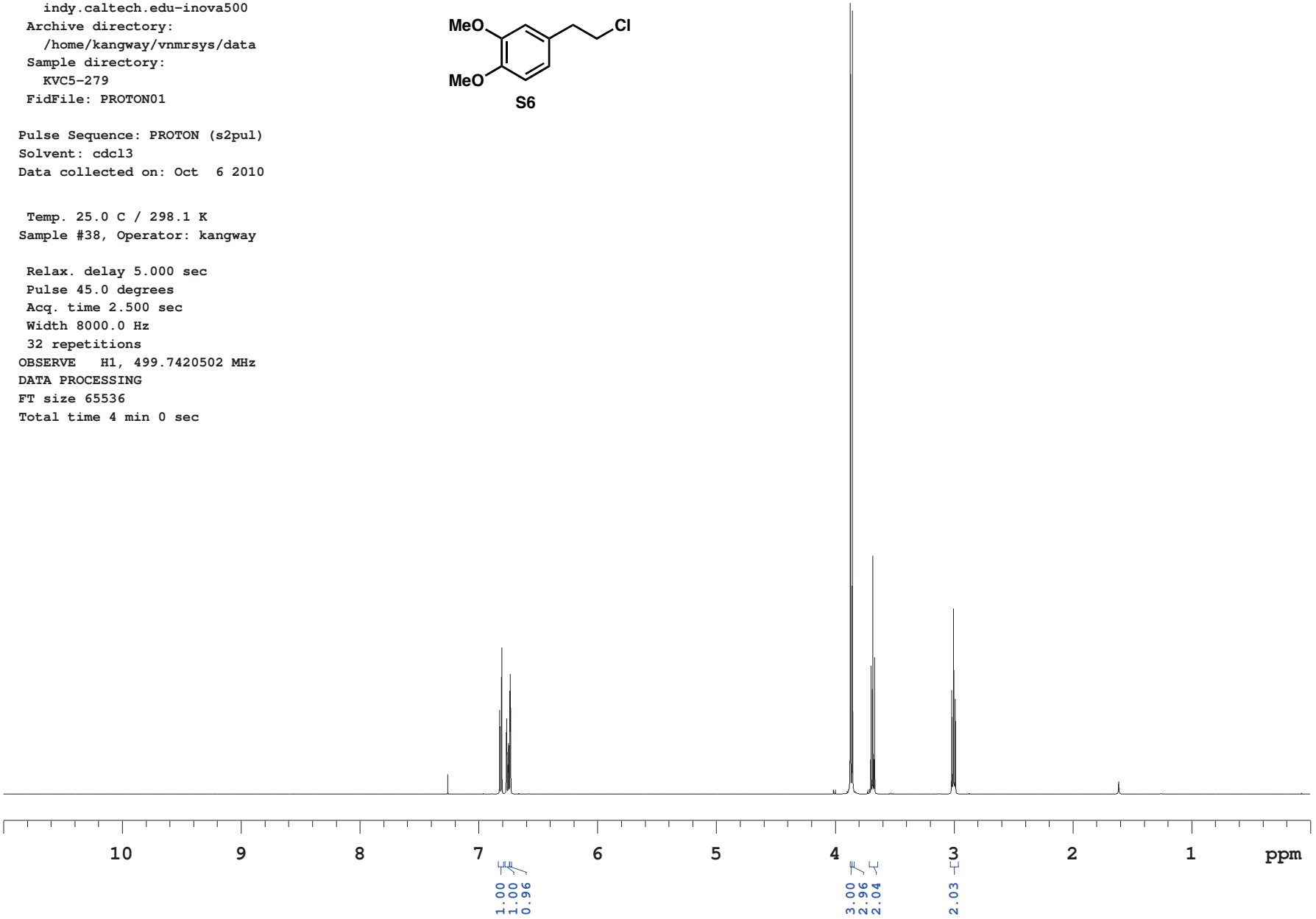
Sample Name:
KVC5-279
Data Collected on:
indy.caltech.edu-inova500
Archive directory:
/home/kangway/vnmrsys/data
Sample directory:
KVC5-279
FidFile: PROTON01

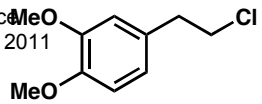
Pulse Sequence: PROTON (s2pul)
Solvent: cdcl3
Data collected on: Oct 6 2010

Temp. 25.0 C / 298.1 K
Sample #38, Operator: kangway

Relax. delay 5.000 sec
Pulse 45.0 degrees
Acq. time 2.500 sec
Width 8000.0 Hz
32 repetitions
OBSERVE H1, 499.7420502 MHz
DATA PROCESSING
FT size 65536
Total time 4 min 0 sec

S6





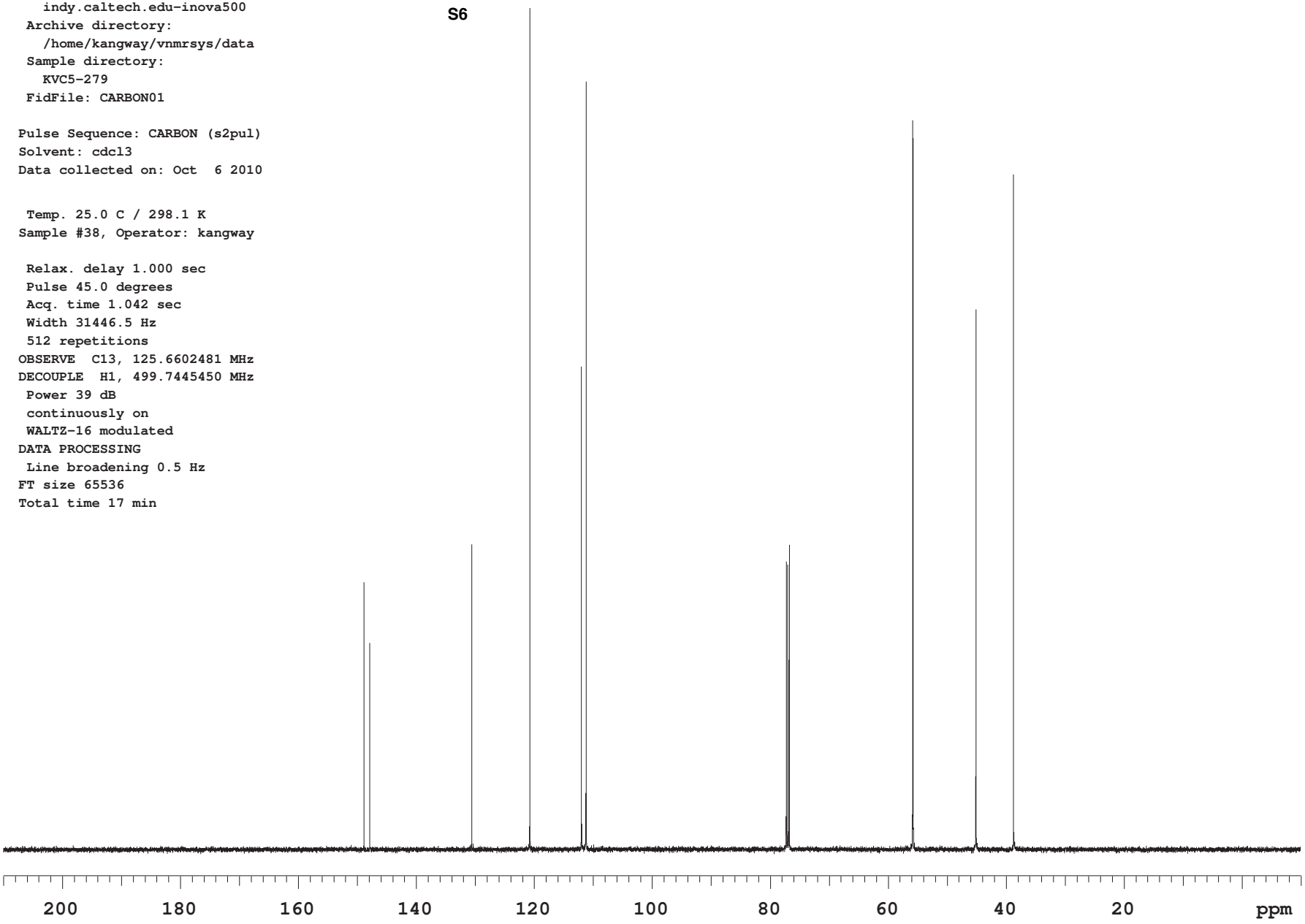
S6

Sample Name:
KVC5-279
Data Collected on:
indy.caltech.edu-inova500
Archive directory:
/home/kangway/vnmrsys/data
Sample directory:
KVC5-279
FidFile: CARBON01

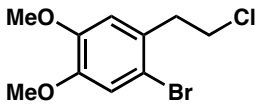
Pulse Sequence: CARBON (s2pul)
Solvent: cdcl3
Data collected on: Oct 6 2010

Temp. 25.0 C / 298.1 K
Sample #38, Operator: kangway

Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.042 sec
Width 31446.5 Hz
512 repetitions
OBSERVE C13, 125.6602481 MHz
DECOUPLE H1, 499.7445450 MHz
Power 39 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 0.5 Hz
FT size 65536
Total time 17 min



Sample Name:
KVC5-281
Data Collected on:
indy.caltech.edu-inova500
Archive directory:
/home/kangway/vnmrsys/data
Sample directory:
KVC5-281
FidFile: PROTON01

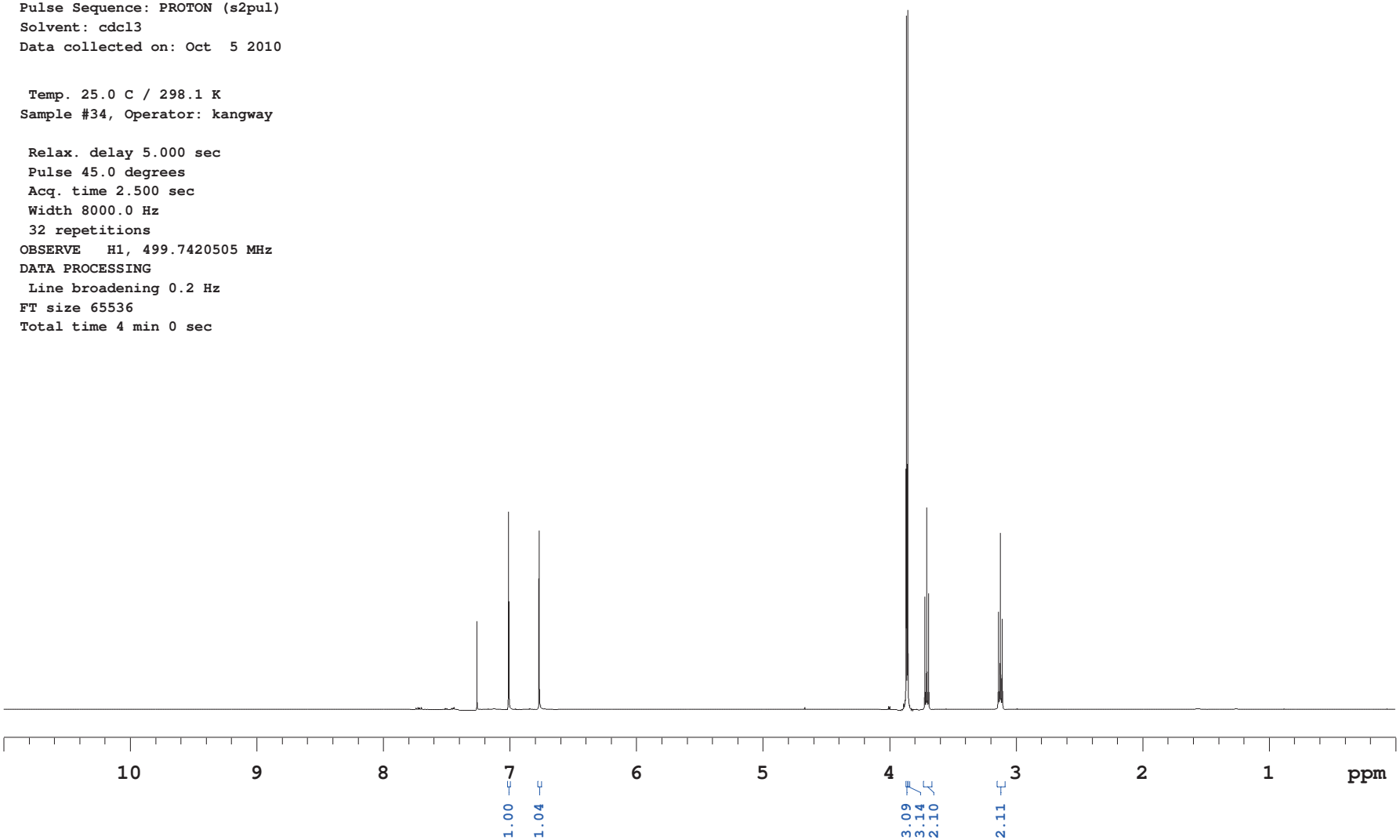


S7

Pulse Sequence: PROTON (s2pul)
Solvent: cdcl3
Data collected on: Oct 5 2010

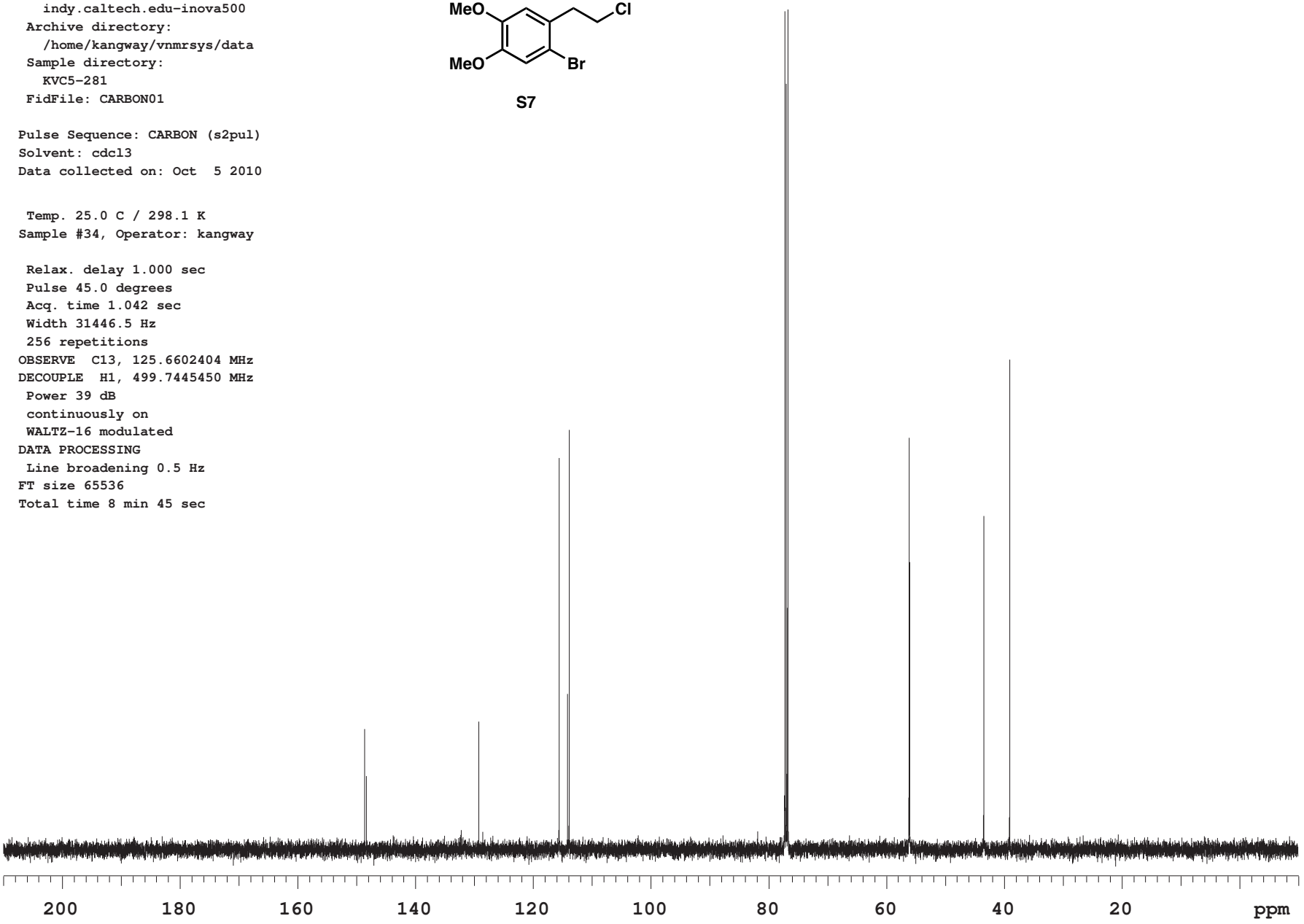
Temp. 25.0 C / 298.1 K
Sample #34, Operator: kangway

Relax. delay 5.000 sec
Pulse 45.0 degrees
Acq. time 2.500 sec
Width 8000.0 Hz
32 repetitions
OBSERVE H1, 499.7420505 MHz
DATA PROCESSING
Line broadening 0.2 Hz
FT size 65536
Total time 4 min 0 sec



```
Sample Name:
  KVC5-281
Data Collected on:
  indy.caltech.edu-inova500
Archive directory:
  /home/kangway/vnmrsys/data
Sample directory:
  KVC5-281
FidFile: CARBON01

Pulse Sequence: CARBON (s2pul)
Solvent: cdcl3
Data collected on: Oct  5 2010


Temp. 25.0 C / 298.1 K
Sample #34, Operator: kangway

Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.042 sec
Width 31446.5 Hz
256 repetitions
OBSERVE  C13, 125.6602404 MHz
DECOUPLE  H1, 499.7445450 MHz
Power 39 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 0.5 Hz
FT size 65536
Total time 8 min 45 sec
```


S7



KVC5-223

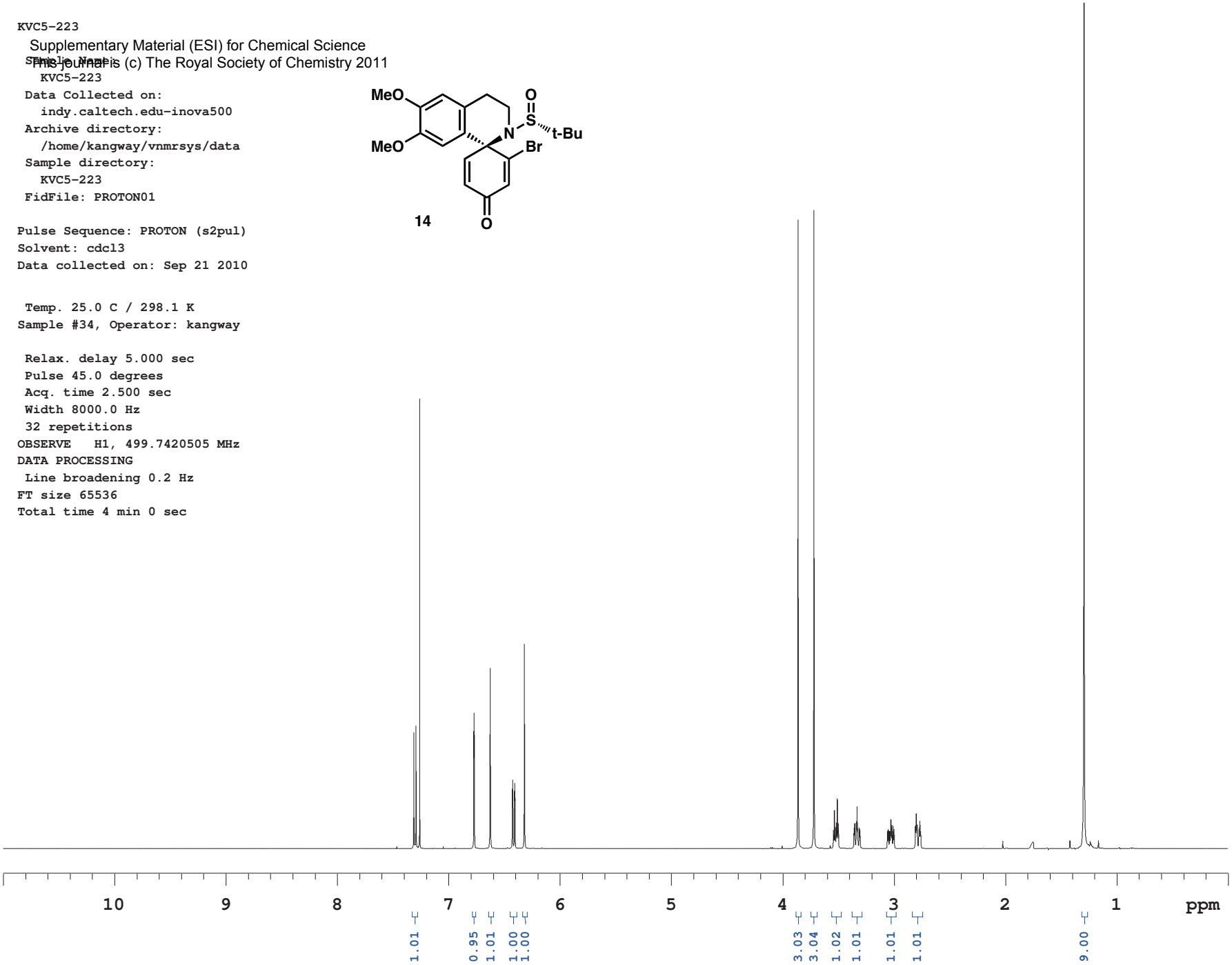
Sample Name:
KVC5-223
Data Collected on:
indy.caltech.edu-inova500
Archive directory:
/home/kangway/vnmrsys/data
Sample directory:
KVC5-223
FidFile: PROTON01

Pulse Sequence: PROTON (s2pul)
Solvent: cdcl3
Data collected on: Sep 21 2010

Temp. 25.0 C / 298.1 K
Sample #34, Operator: kangway

Relax. delay 5.000 sec
Pulse 45.0 degrees
Acq. time 2.500 sec
Width 8000.0 Hz
32 repetitions
OBSERVE H1, 499.7420505 MHz
DATA PROCESSING
Line broadening 0.2 Hz
FT size 65536
Total time 4 min 0 sec

14

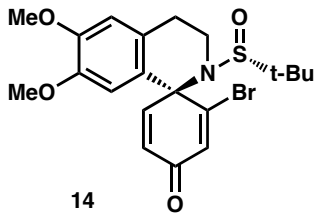


Sample Name:
KVC5-223
Data Collected on:
indy.caltech.edu-inova500
Archive directory:
/home/kangway/vnmrsys/data
Sample directory:
KVC5-223
FidFile: CARBON01

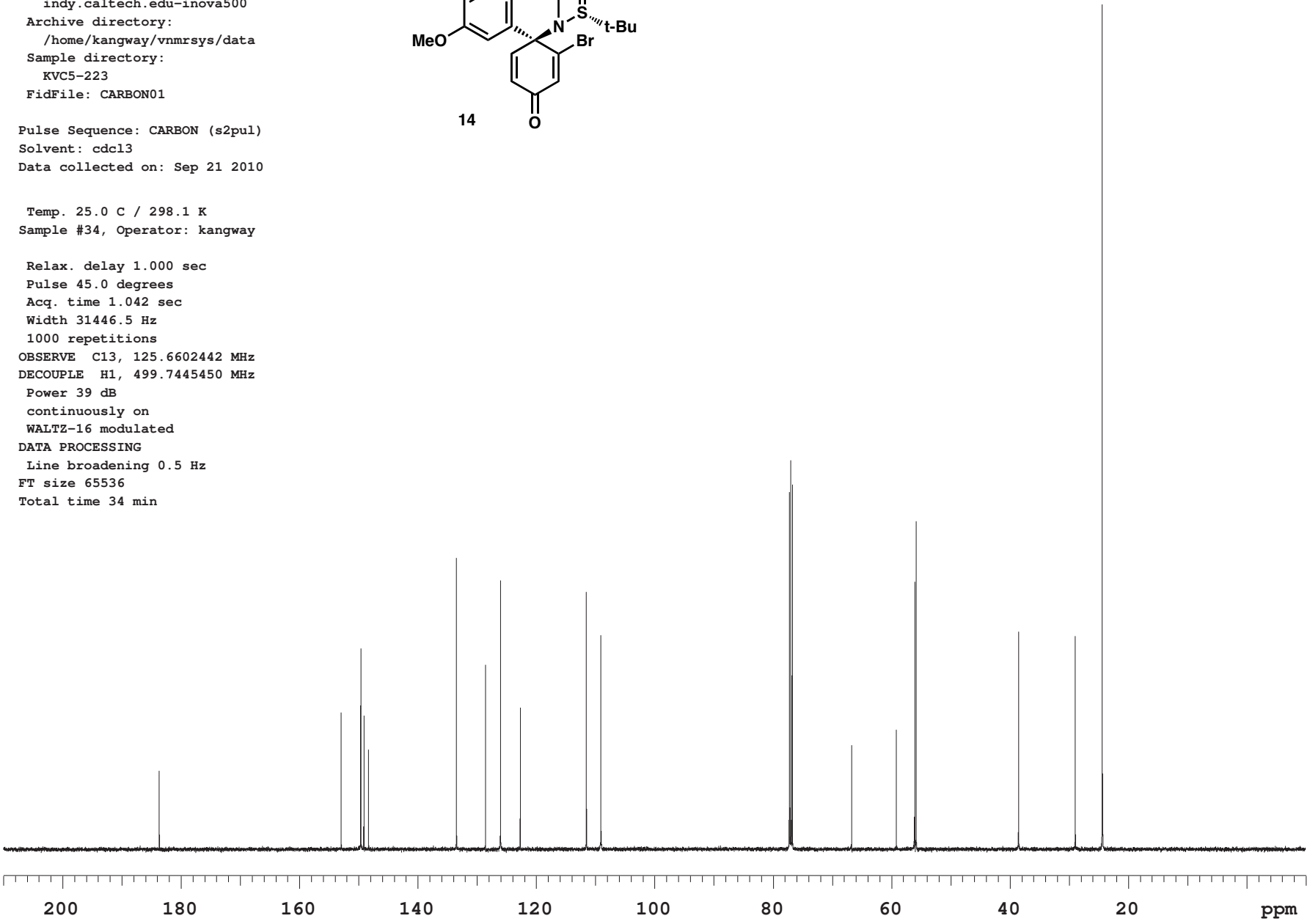
Pulse Sequence: CARBON (s2pul)
Solvent: cdcl3
Data collected on: Sep 21 2010

Temp. 25.0 C / 298.1 K
Sample #34, Operator: kangway

Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.042 sec
Width 31446.5 Hz
1000 repetitions
OBSERVE C13, 125.6602442 MHz
DECOUPLE H1, 499.7445450 MHz
Power 39 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 0.5 Hz
FT size 65536
Total time 34 min



14



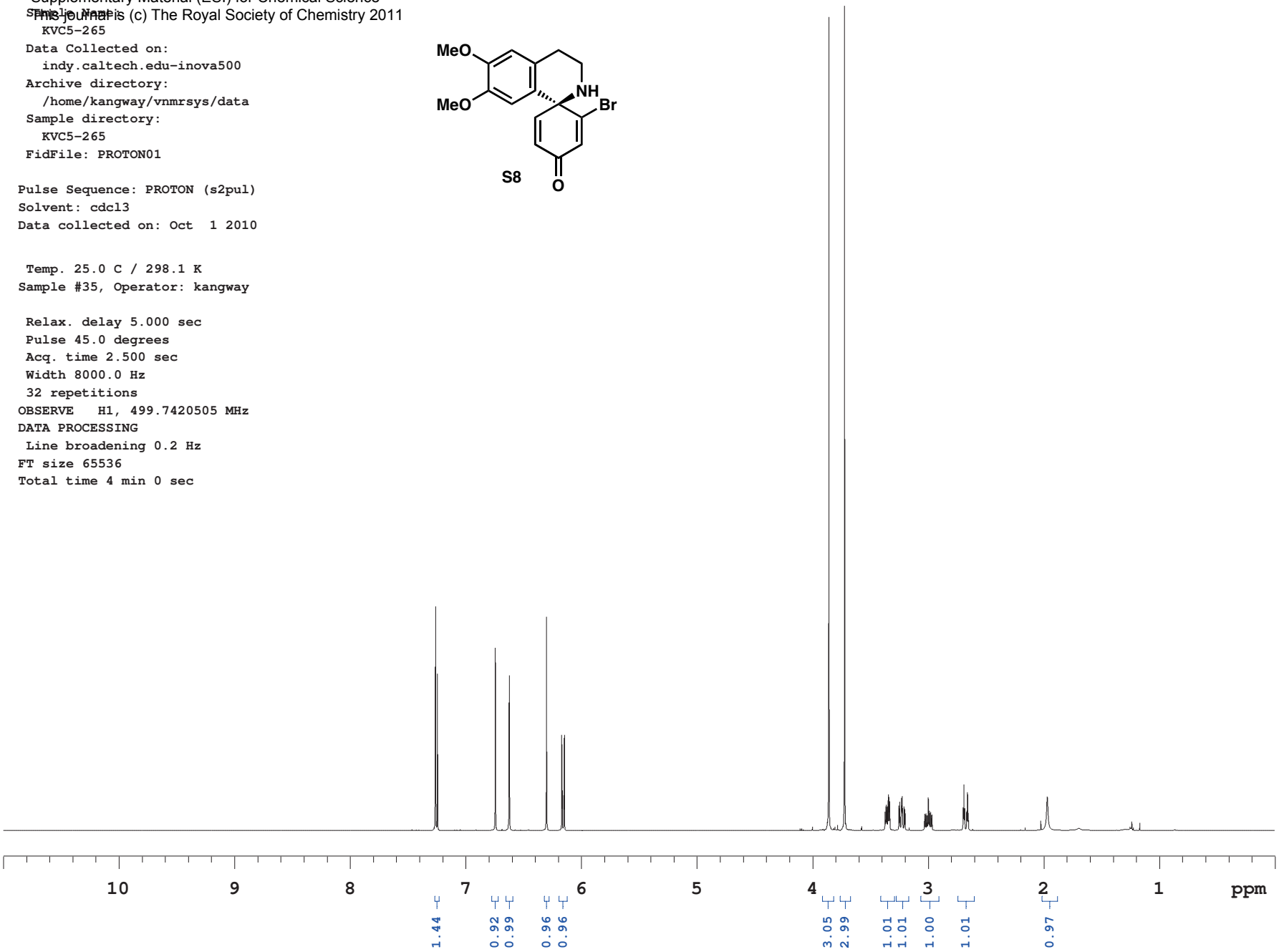
Sample Name:
KVC5-265
Data Collected on:
indy.caltech.edu-inova500
Archive directory:
/home/kangway/vnmrsys/data
Sample directory:
KVC5-265
FidFile: PROTON01

Pulse Sequence: PROTON (s2pul)
Solvent: cdcl3
Data collected on: Oct 1 2010

Temp. 25.0 C / 298.1 K
Sample #35, Operator: kangway

Relax. delay 5.000 sec
Pulse 45.0 degrees
Acq. time 2.500 sec
Width 8000.0 Hz
32 repetitions
OBSERVE H1, 499.7420505 MHz
DATA PROCESSING
Line broadening 0.2 Hz
FT size 65536
Total time 4 min 0 sec

S8

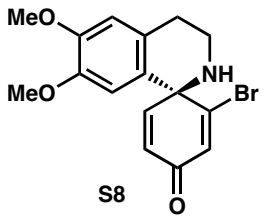


Sample Name:
KVC5-265
Data Collected on:
indy.caltech.edu-inova500
Archive directory:
/home/kangway/vnmrsys/data
Sample directory:
KVC5-265
FidFile: CARBON01

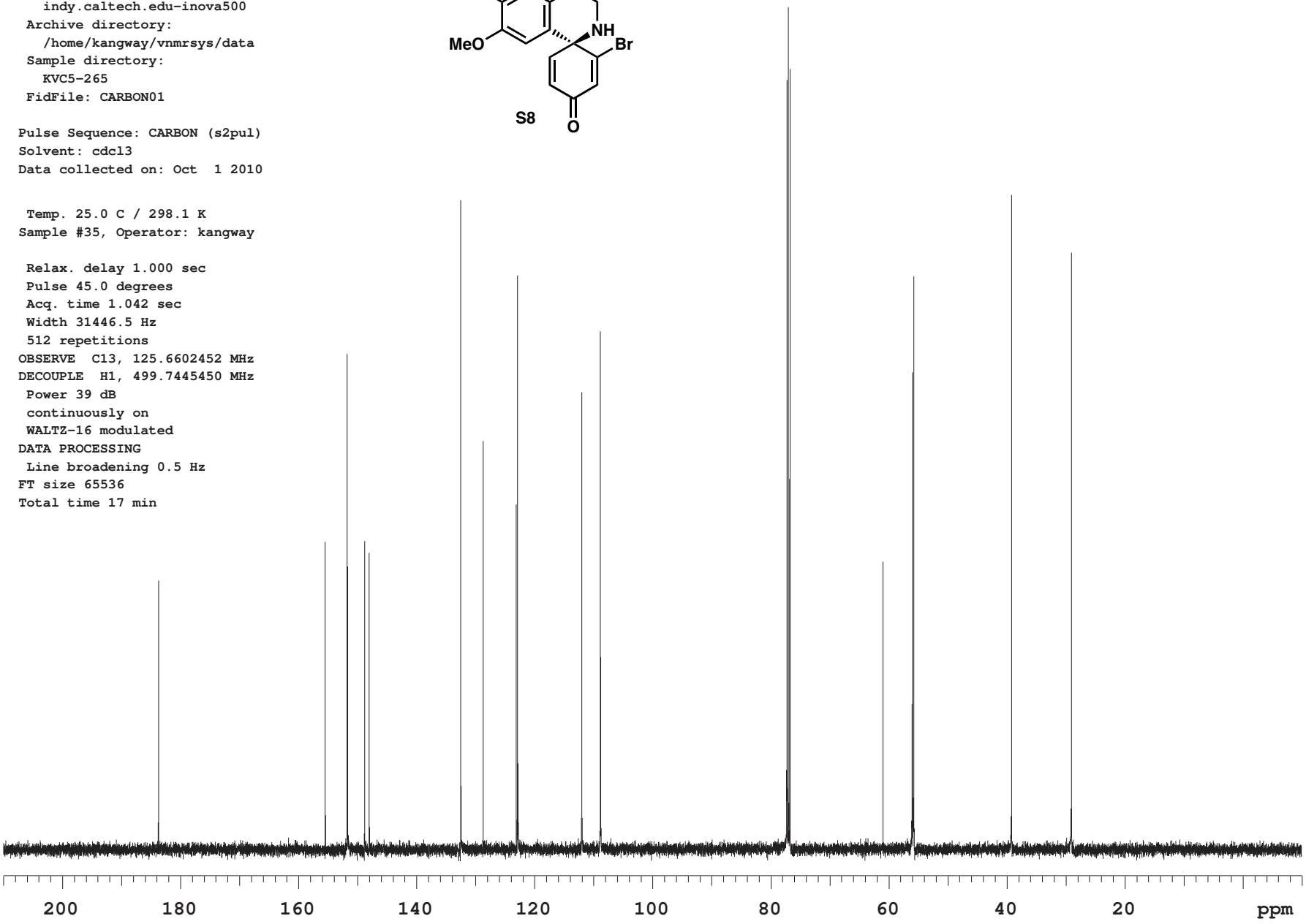
Pulse Sequence: CARBON (s2pul)
Solvent: cdcl3
Data collected on: Oct 1 2010

Temp. 25.0 C / 298.1 K
Sample #35, Operator: kangway

Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.042 sec
Width 31446.5 Hz
512 repetitions
OBSERVE C13, 125.6602452 MHz
DECOUPLE H1, 499.7445450 MHz
Power 39 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 0.5 Hz
FT size 65536
Total time 17 min



S8



KVC5-233

Sample Name:
KVC5-233
Data Collected on:
indy.caltech.edu-inova500
Archive directory:
/home/kangway/vnmrsys/data
Sample directory:
KVC5-233
FidFile: PROTON02

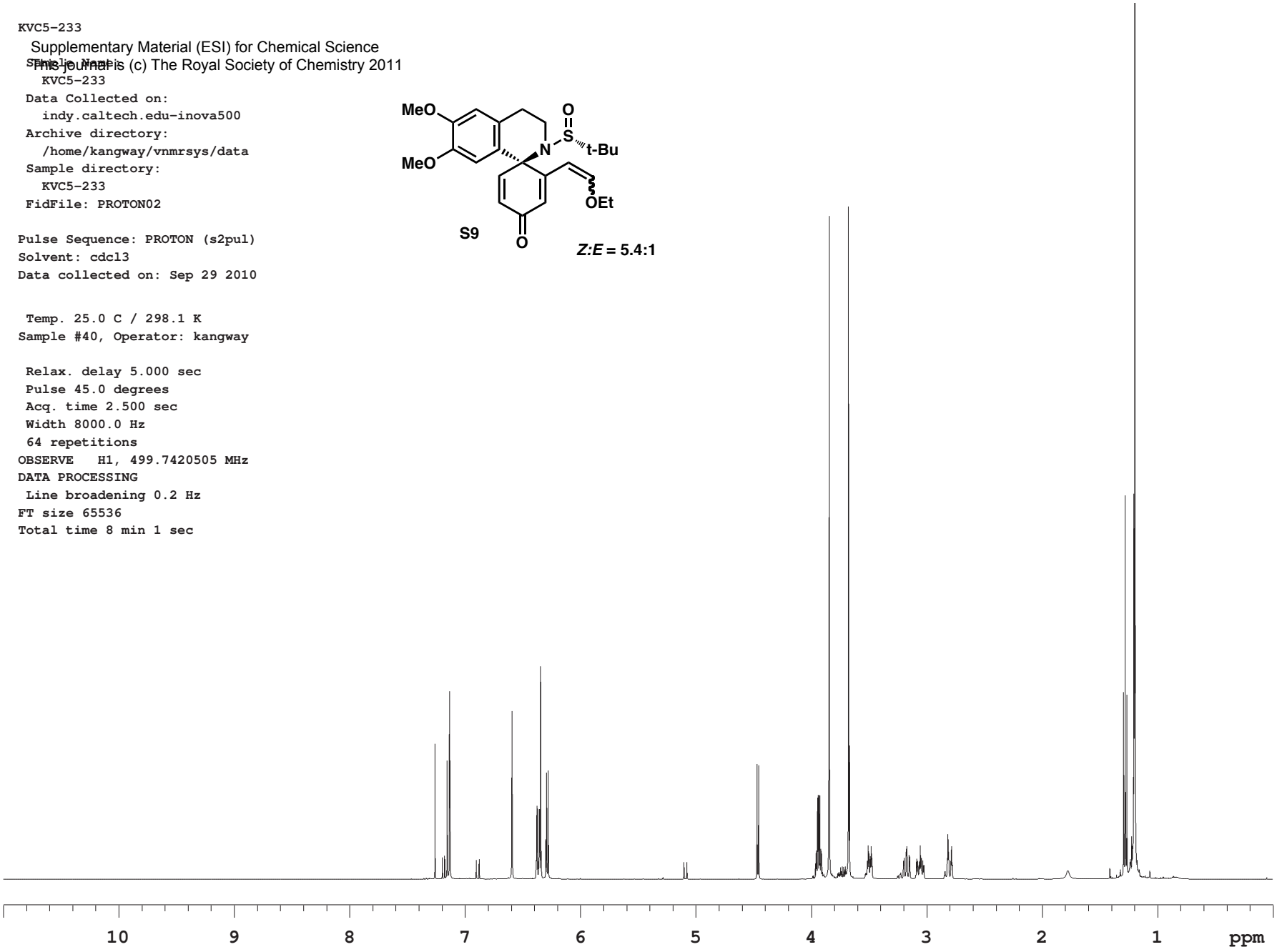
Pulse Sequence: PROTON (s2pul)
Solvent: cdcl3
Data collected on: Sep 29 2010

Temp. 25.0 C / 298.1 K
Sample #40, Operator: kangway

Relax. delay 5.000 sec
Pulse 45.0 degrees
Acq. time 2.500 sec
Width 8000.0 Hz
64 repetitions
OBSERVE H1, 499.7420505 MHz
DATA PROCESSING
Line broadening 0.2 Hz
FT size 65536
Total time 8 min 1 sec

S9

Z:E = 5.4:1

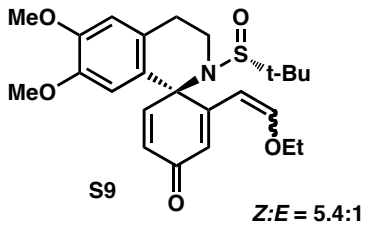


Sample Name:
KVC5-233
Data Collected on:
indy.caltech.edu-inova500
Archive directory:
/home/kangway/vnmrsys/data
Sample directory:
KVC5-233
FidFile: CARBON02

Pulse Sequence: CARBON (s2pul)
Solvent: cdcl3
Data collected on: Sep 29 2010

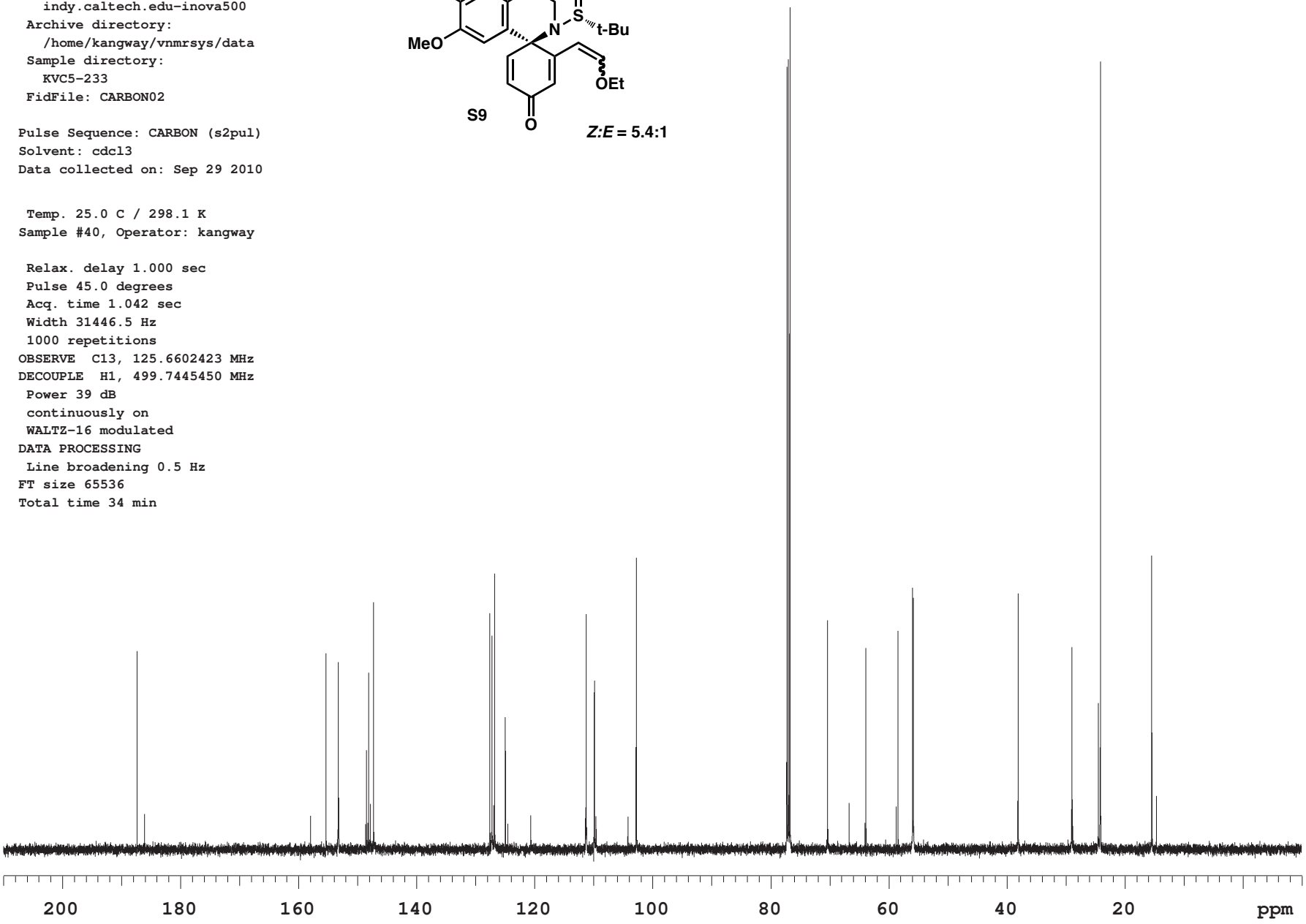
Temp. 25.0 C / 298.1 K
Sample #40, Operator: kangway

Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.042 sec
Width 31446.5 Hz
1000 repetitions
OBSERVE C13, 125.6602423 MHz
DECOUPLE H1, 499.7445450 MHz
Power 39 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 0.5 Hz
FT size 65536
Total time 34 min



S9

Z:E = 5.4:1



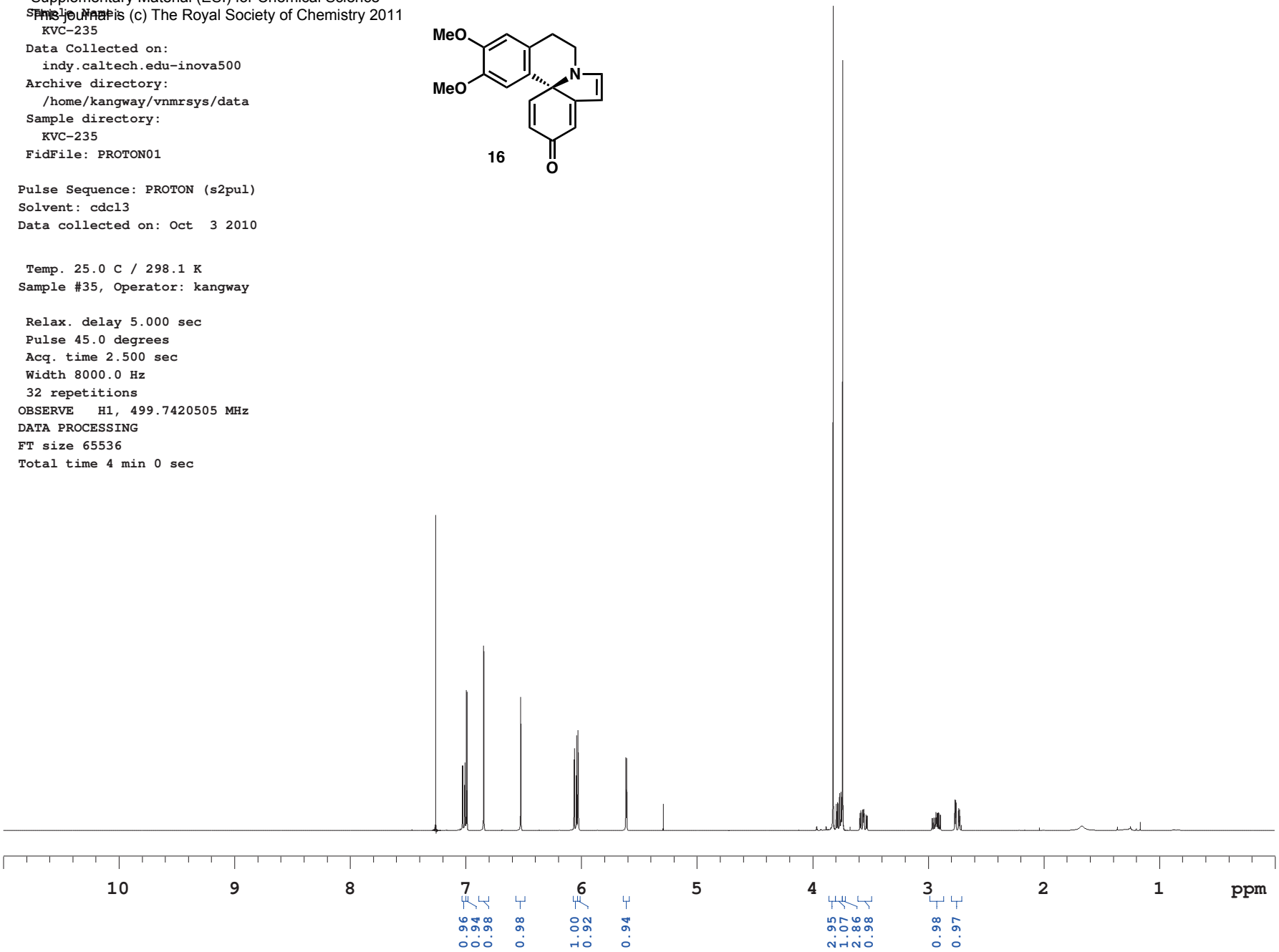
Sample Name:
KVC-235
Data Collected on:
indy.caltech.edu-inova500
Archive directory:
/home/kangway/vnmrsys/data
Sample directory:
KVC-235
FidFile: PROTON01

Pulse Sequence: PROTON (s2pul)
Solvent: cdcl3
Data collected on: Oct 3 2010

Temp. 25.0 C / 298.1 K
Sample #35, Operator: kangway

Relax. delay 5.000 sec
Pulse 45.0 degrees
Acq. time 2.500 sec
Width 8000.0 Hz
32 repetitions
OBSERVE H1, 499.7420505 MHz
DATA PROCESSING
FT size 65536
Total time 4 min 0 sec

16



KVC-235

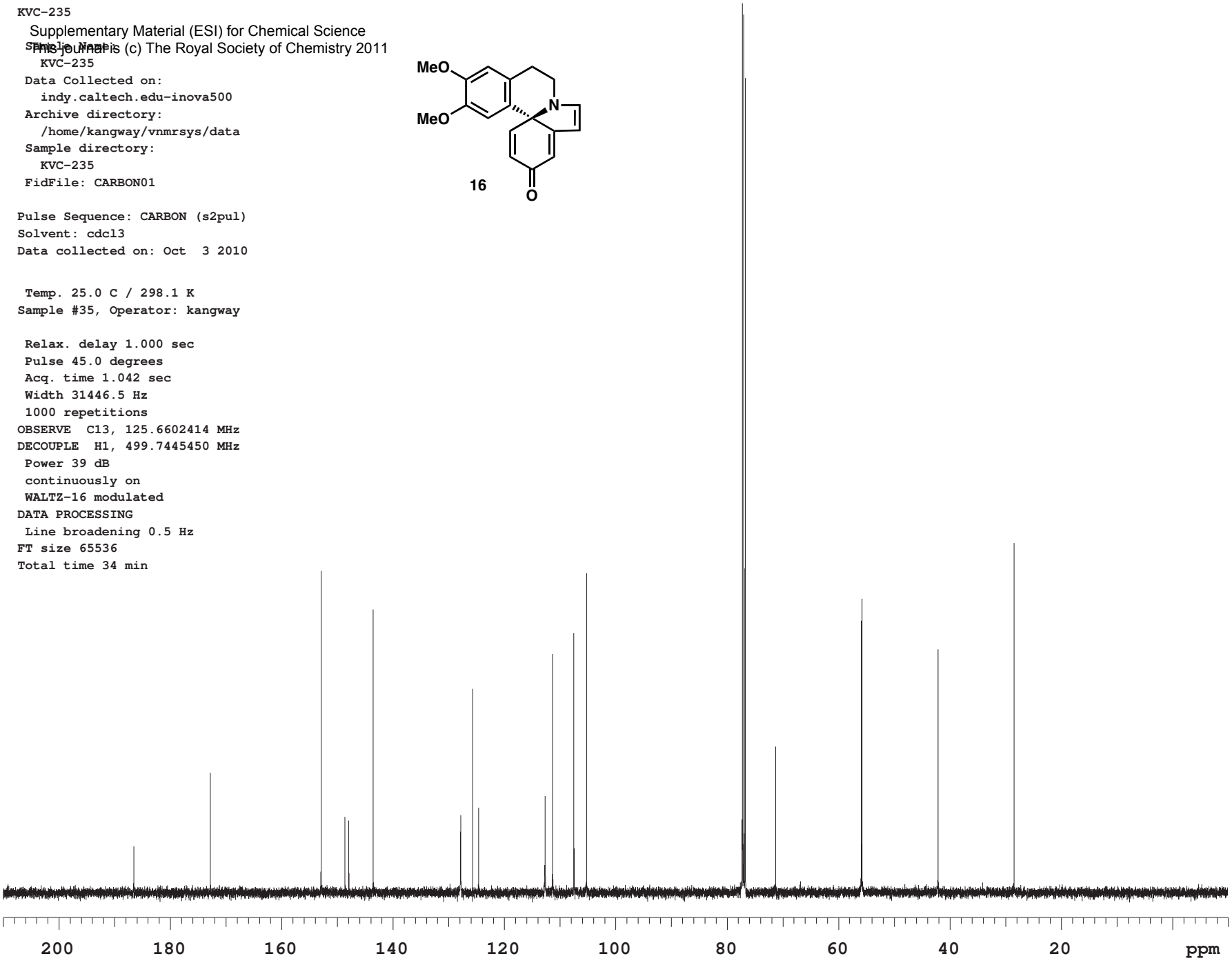
Sample Name:
KVC-235
Data Collected on:
indy.caltech.edu-inova500
Archive directory:
/home/kangway/vnmrsys/data
Sample directory:
KVC-235
FidFile: CARBON01

Pulse Sequence: CARBON (s2pul)
Solvent: cdcl3
Data collected on: Oct 3 2010

Temp. 25.0 C / 298.1 K
Sample #35, Operator: kangway

Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.042 sec
Width 31446.5 Hz
1000 repetitions
OBSERVE C13, 125.6602414 MHz
DECOUPLE H1, 499.7445450 MHz
Power 39 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 0.5 Hz
FT size 65536
Total time 34 min

16



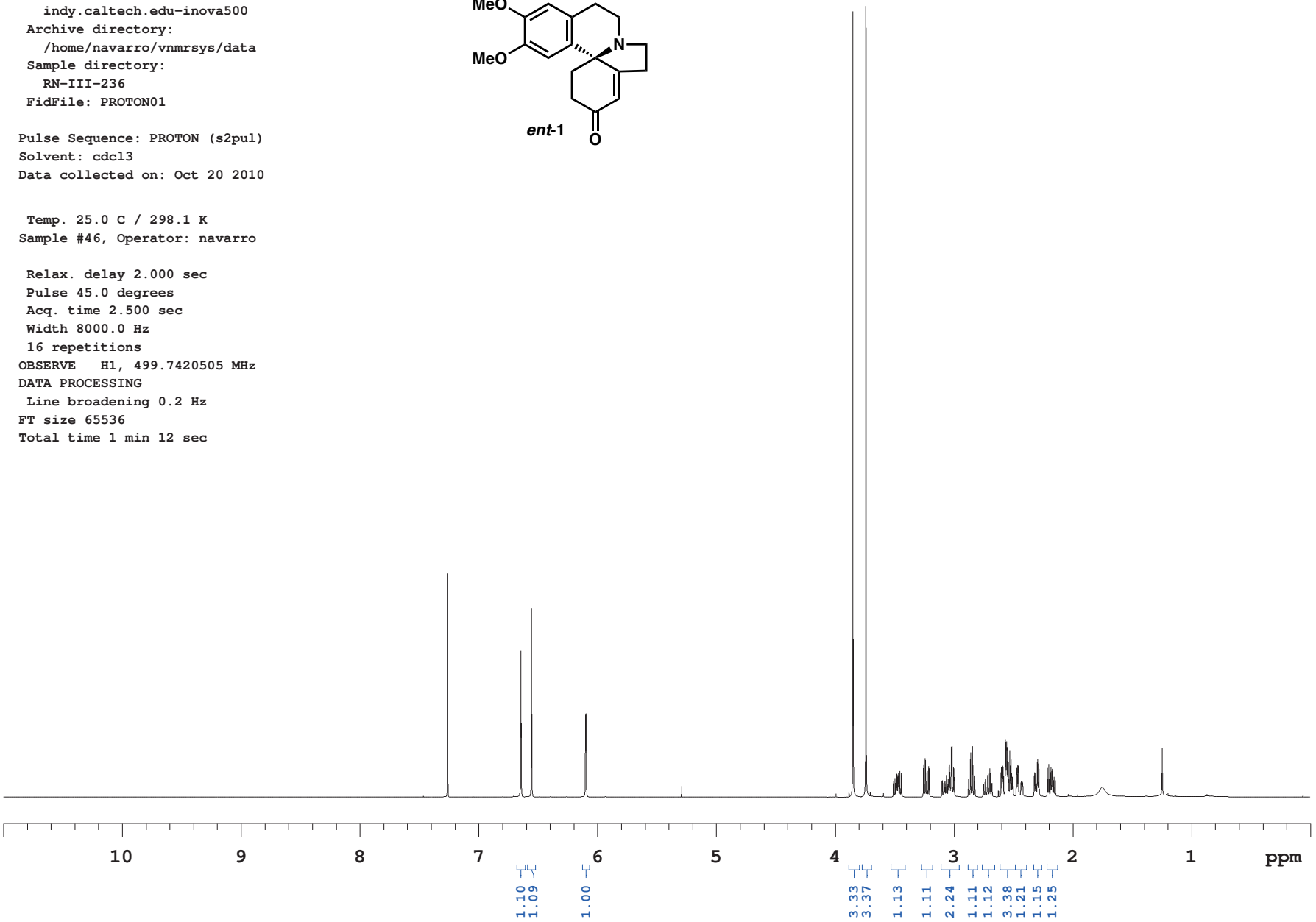
Sample Name:
RN-III-236
Data Collected on:
indy.caltech.edu-inova500
Archive directory:
/home/navarro/vnmrsys/data
Sample directory:
RN-III-236
FidFile: PROTON01

Pulse Sequence: PROTON (s2pul)
Solvent: cdcl3
Data collected on: Oct 20 2010

Temp. 25.0 C / 298.1 K
Sample #46, Operator: navarro

Relax. delay 2.000 sec
Pulse 45.0 degrees
Acq. time 2.500 sec
Width 8000.0 Hz
16 repetitions
OBSERVE H1, 499.7420505 MHz
DATA PROCESSING
Line broadening 0.2 Hz
FT size 65536
Total time 1 min 12 sec

ent-1



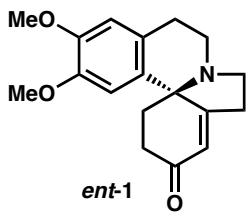
RN-III-236
purified

Sample Name:
RN-III-236
Data Collected on:
indy.caltech.edu-inova500
Archive directory:
/home/navarro/vnmrsys/data
Sample directory:
RN-III-236
FidFile: CARBON01

Pulse Sequence: CARBON (s2pul)
Solvent: cdcl3
Data collected on: Oct 20 2010

Temp. 25.0 C / 298.1 K
Sample #46, Operator: navarro

Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.042 sec
Width 31446.5 Hz
1500 repetitions
OBSERVE C13, 125.6602404 MHz
DECOUPLE H1, 499.7445450 MHz
Power 39 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 0.5 Hz
FT size 65536
Total time 51 min



ent-1