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On the Stereochemistry of Acetylide Additions to Highly Functionalized Biphenylcarboxaldehydes and Multi-component Cycliztions of 1,n-Diynes. Syntheses of Dibenzocyclooctadiene Lignans

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SUPPORTING INFORMATION

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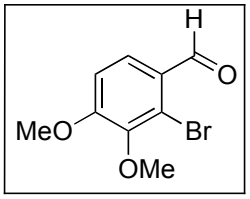
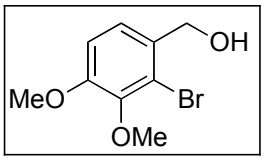
General Methods. All catalyzed reactions were carried out under an inert atmosphere of nitrogen in a Vacuum Atmosphere drybox, or using Schlenk techniques. Methylene chloride (CH_2Cl_2) was distilled from calcium hydride under nitrogen and stored over 4 Å molecular sieves. Tetrahydrofuran (THF) was distilled from sodium benzophenone ketyl under nitrogen and stored over 4 Å molecular sieves. Hexane was distilled from CaH_2 and stored over 4 Å molecular sieves. All chemicals were purchased from Aldrich Chemical Company and used as received unless otherwise mentioned. Analytical TLC was done on E. Merck precoated (0.25 mm) silica gel 60 F_{254} plates. Flash column chromatography was conducted on silica gel 40 (Scientific Adsorbents Incorporated). ^{1}H NMR spectra were recorded on a 500 MHz and or a 400 MHz spectrometer. Coupling constants (*J*) are reported in Hz. Optical rotations were recorded in solution at the sodium D-line using filtered (45 μ nylon filter) solutions.

Compounds previously disclosed. Preparation and spectroscopic details of the following compounds were disclosed in an initial communication[2] describing parts of this work: **1, 2, 4, 8A**. Results of X-ray crystallographic analysis of solid-state structure of **38** was also deposited with that publication.[1] Details of a modified procedure for the synthesis of the [B-Sn]-reagent **3** have been published.[2] Compounds **9**[3] and **10**[4] were prepared by known procedures.

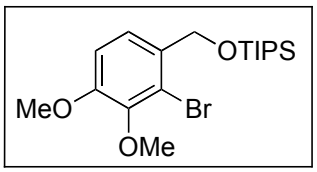
Synthesis of aldehyde 5A from 2-bromo-3-hydroxy-4-dimethoxybenzaldehyde



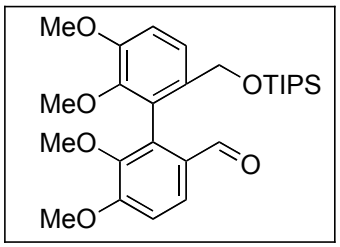
Synthesis of 2-bromo-3,4-dimethoxybenzaldehyde. To a stirred solution of commercially available 2-bromo-3-hydroxy-4-methoxybenzaldehyde (2.0 g, 8.6 mmol) in acetone (35 mL) was added K_2CO_3 (658 mg, 17 mmol) followed by methyl iodide (2.0 mL) ~~were added~~ was further at room temperature, and the mixture was gradually brought to 60 ^{o}C temperature and stirred for 4 h. The solvent was evaporated under reduced pressure to afford the crude product, which was diluted with ethyl acetate and washed successively with water, brine and dried over Na_2SO_4. The organic layer was evaporated under reduced pressure to get the crude product which was purified by column chromatography on silica gel using 20 % EtOAc/hexane as the eluent to yield the product (1.95 g, 8.0 mmol, 93%) as a viscous solid. ^{1}H NMR (400 MHz, $CDCl_3$) δ 10.24 (s, 1 H), 7.72 (d, *J* = 8.8 Hz, 1 H), 6.94 (d, J = 8.8 Hz, 1 H), 3.93 (s, 3 H), 3.84 (s, 3 H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 191.12, 158.86, 146.63, 127.62, 126.69, 123.30, 111.17, 60.86, 56.51; IR (neat, cm^{-1}): 1682, 1558, 1493, 1259, 1023; MS (ESI): m/z: 266.95 $[M+Na]^+$.



Synthesis of 2-bromo-3,4-dimethoxybenzyl alcohol. To a solution of aldehyde from the previous step (1.22 g, 5.0 mmol) in EtOH (20 mL) at 0 °C was added $NaBH_4$ (370 mg, 10.0 mmol) in portions. After stirring at the same temperature for 1 h, the reaction was quenched by the addition of aq. NH_4Cl (5.0 mL). The organic solvent was evaporated under reduced pressure to afford the crude product, which was diluted with ethyl acetate and washed successively with water, brine and dried over Na_2SO_4. The organic layer was evaporated under reduced pressure to afford the crude product which was purified by column chromatography on silica gel using 30 % EtOAc/hexane the eluent to yield the benzyl alcohol (1.08 g, 4.4 mmol) in 88% yield as a viscous oil. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.14 (d, J = 8.4 Hz, 1 H), 6.84 (d, J = 8.4 Hz, 1 H), 4.67 (s, 2 H), 3.86 (s, 3 H), 3.84 (s, 3 H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 153.40, 146.81, 133.07, 124.52, 111.48, 65.39, 60.73, 56.36; IR (neat, cm^{-1}): 1490, 1262, 1036, 823; MS (ESI): m/z: 268.97 $[M+Na]^+$.

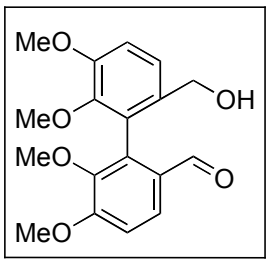


Synthesis of the TIPS ether of 2-bromo-3,4-dimethoxybenzyl alcohol. To a stirred solution of the alcohol (1.0 g, 4.0 mmol) in CH_2Cl_2 (13.0 mL) at 0 °C was added imidazole (457 mg, 6.73 mmol) followed by TIPSCl (1.05 mL, 5.0 mmol) and the reaction mixture was gradually brought to room temperature over 2 h. Afterwards, the reaction mixture was diluted with CH_2Cl_2 and was washed subsequently with water, brine and dried over Na_2SO_4. The solvent was evaporated under reduced pressure to afford the crude product, which was purified by silica gel column chromatography. Elution with 10 % EtOAc/hexane provided product (1.41 g, 3.52 mmol) in 88 % yield as a viscous oil. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.31 (d, J = 8.4 Hz, 1 H), 6.89 (d, J = 8.4 Hz, 1 H), 4.74 (s, 2 H), 3.85 (s, 3 H), 3.83 (s, 3 H), 1.20-1.15 (m, 3 H), 1.09 (s, 18 H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 152.45, 133.74, 122.43, 116.60, 111.55, 64.89, 60.72, 56.38, 18.30, 12.29; IR (neat, cm^{-1}): 1488, 1457, 1297, 1035; MS (ESI): m/z: 427.1 $[M+Na]^+$.



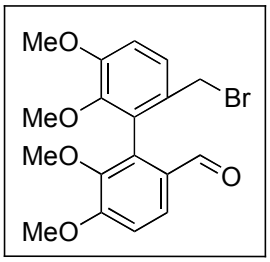
Synthesis of the biphenyl-2'-triisopropylsiloxymethyl-2-carboxaldehyde. To a solution of the TIPS ether from the previous step (806 mg, 2.0 mmol) in THF (8 mL) at –78 °C was added dropwise *n*-BuLi (2.5 M in hexane, 1.04 mL, 2.6 mmol). After stirring at the same temperature for 60 min, trimethylborate (0.33

mL, 3.0 mmol) was added slowly and the solution was allowed to warm to rt overnight. Hydrochloric acid (1 M, 3 mL) was added to the reaction mixture and it was left to stir for 30 min. The volatile materials were removed *in vacuo* and ~~replaced with~~ ether (50 mL) was added. The organic layer was washed with water, brine and dried over Na_2SO_4. The solvent was evaporated under reduced pressure to afford the crude boronic acid, which was used without further purification. To 2-bromo-3,4-dimethoxybenzaldehyde described in the first procedure (396 mg, 1.6 mmol) in DME (6 mL) under an atmosphere of nitrogen was added the crude boronic acid (710 mg) from the above reaction followed by and 2 mL of 2 M sodium carbonate solution. To this mixture was added *tetrakis-(*triphenylphosphine)palladium, and the reaction was heated at reflux for 12 h. The reaction mixture was diluted with diethyl ether and the solution was washed successively with 2 N sodium hydroxide solution, water, brine and dried over Na_2SO_4. The organic layer was evaporated under reduced pressure to afford the crude product which was purified by column chromatography on silica gel using 15 % EtOAc/hexane as the eluent to yield the biphenylcarboxaldehyde (536 mg, 1.1 mmol) in overall 68% yield for two steps as a viscous oil. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.82 (d, J = 8.8 Hz, 1 H), 7.32 (d, J = 8.4 Hz, 1 H), 7.02 (*t*, J = 8.8 Hz, 2 H), 4.39 (d, J = 12 Hz, 1 H), 4.24 (d, J = 12 Hz, 1 H), 3.95 (s, 3 H), 3.89 (s, 3 H), 3.59 (s, 3 H), 3.58 (s, 3 H), 1.08-0.95 (m, 21 H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 191.07, 157.82, 151.56, 146.59, 146.34, 134.63, 133.20, 128.32, 125.99, 124.44, 122.68, 112.49, 111.71, 63.22, 60.63, 60.51, 56.13, 55.94, 18.14, 12.11.



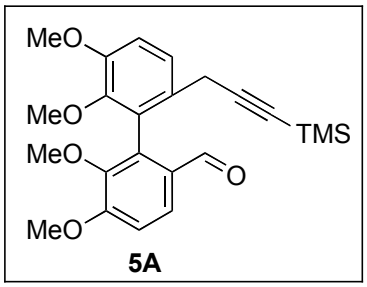
Biphenyl-2'-hydroxymethyl-2-carboxaldehyde. To a stirred solution of the TIPS ether from the previous step (1.27 g, 2.6 mmol) in THF (6.0 mL) was added TBAF (1 M in THF, 3.12 mL, 3.12 mmol) at 0 °C and stirring was continued at the same temperature for 2 h. The solvent was evaporated under reduced pressure to afford the crude product which was purified by silica gel chromatography using 35 % EtOAc/hexane as the eluent to yield the alcohol (742 mg, 2.2 mmol) in 86% yield as a viscous oil. ^{1}H NMR (400 MHz, $CDCl_3$) δ 9.49 (s, 1H), 7.83 (d, J = 8.8 Hz, 1 H), 7.25 (d, J = 7.6 Hz, 1 H), 7.06 (d, J = 8.8 Hz, 1 H), 7.01 (d, J = 8.4 Hz, 1 H), 3.97 (s, 3 H), 3.90 (s, 3 H), 3.60 (s, 6 H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 190.87, 157.90, 152.29, 146.72, 146.13, 134.60, 132.89, 128.42, 127.10, 125.57, 125.15, 112.89, 111.81,

63.71, 60.94, 60.55, 56.16, 55.99; IR (neat, cm^{-1}): 1683, 1491, 1139, 1049, 823, 734; MS (ESI): m/z: 355.11 $[M+Na]^+$.



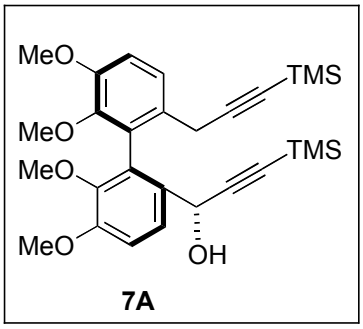
Synthesis of biphenyl-2'-bromomethyl-2-carboxaldehyde: To a stirred solution of benzyl alcohol from the previous step (500 mg, 1.5 mmol) in CH_2Cl_2 (6.0 mL) at 0 °C was added Ph_3P (524 mg, 2.0 mmol) followed by CBr_4 (662 mg, 2.0 mmol) and the reaction mixture was gradually brought to room temperature over 12 h. Afterwards, the reaction mixture was diluted with CH_2Cl_2 and washed subsequently with aqueous 0.2 N HCl, water, brine and dried over Na_2SO_4. The solvent was evaporated under reduced pressure to afford the crude product, which was purified by silica gel column chromatography. Elution with 20% EtOAc/hexane provided the benzyl bromide (485 mg, 1.23 mmol) in 82 % yield as a viscous oil. ^1H NMR (400 MHz, $CDCl_3$) δ 9.52 (s, 1 H), 7.87 (d, *J* = 8.5 Hz, 1 H), 7.25 (d, *J* = 8.5 Hz, 1 H), 7.10 (d, J = 8.5 Hz, 1 H), 6.99 (d, *J* = 8.5 Hz, 1 H), 4.27 (d, *J* = 10.0 Hz, 1 H), 4.12 (d, *J* = 10.0 Hz, 1 H), 3.98 (s, 3 H), 3.91 (s, 3 H), 3.62 (s, 3 H), 3.60 (s, 3 H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 190.75, 157.80, 152.89, 147.04, 146.18, 133.57, 129.50, 128.43, 128.37, 126.27, 124.94, 112.85, 112.12, 60.76, 60.53, 56.14, 56.00, 32.51; IR (neat, cm^{-1}): 1682.5, 1488.7, 1260.4, 1139.6, 1024.9, 814.7; MS (ESI): m/z: 419.02 $[M+Na]^+$.

Synthesis of aldehyde 5A. To a stirred solution of compound benzyl bromide from the previous step (500 mg, 1.26 mmol) in DME (8 mL) at room temperature was added $Pd_2(dba)_3.CHCl_3$ (30 mg, 0.03 mmol) followed by *tris*-2-furylphosphine (30 mg, 0.13 mmol) and the reaction mixture was stirred for 10 min until it became a clear yellow solution. The tributylstannyltrimethylsilylacetylene (619 mg, 1.6 mmol) in DME (1.0 mL) was added to the reaction mixture and stirring was continued at 80 °C for 30 min. Afterwards, the reaction mixture was diluted with CH_2Cl_2 and washed with water, brine and dried over Na_2SO_4. The solvent was evaporated under reduced pressure to afford the crude product, which was purified by silica gel column chromatography. Elution with 20 % EtOAc/hexane provided product **5A** (467 mg, 1.13 mmol) in 90% yield as viscous oil. ^1H NMR (400 MHz, $CDCl_3$) δ 9.47 (s, 1 H), 7.81 (d, *J* = 8.4 Hz, 1 H), 7.29 (d, *J* = 8.4 Hz, 1 H), 7.04 (d, *J* = 8.4 Hz, 1 H), 6.98 (d, *J* = 8.4 Hz, 1 H), 3.96 (s, 3 H),



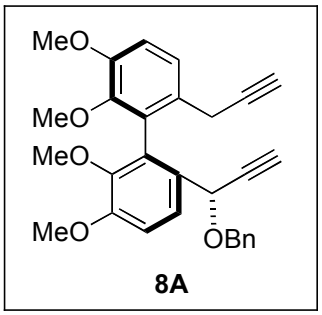
5A

3.89 (s, 3 H), 3.60 (s, 3 H), 3.59 (s, 3 H), 3.26 (d, J = 18.8 Hz, 1 H), 3.12 (d, J = 18.8 Hz, 1 H), 0.07 (s, 9 H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 190.85, 157.94, 151.47, 146.91, 146.26, 134.71, 128.31, 127.31, 124.77, 123.91, 112.76, 111.82, 104.09, 87.21, 60.70, 60.52, 56.12, 56.00, 24.36, 0.14; IR (neat, cm^{-1}): 2838, 1684, 1587, 1487, 1139, 1026; MS (ESI): m/z: 435.16 $[M^+Na]^+$.



7A

Synthesis of compound 7A: To a stirred solution of compound **5A** (412 mg, 1.0 mmol) in dry THF (6 mL) at –78 °C was added lithium trimethylsilylacetylide (0.5 M in THF, 4.0 mL, 2.0 mmol). After stirring at –78 °C for 30 min, the reaction was quenched by the addition of saturated aqueous NH_4Cl solution. The reaction mixture was diluted with CH_2Cl_2 and washed with water, brine and dried over Na_2SO_4. The solvent was evaporated under reduced pressure to afford the crude product, which was purified by silica gel column chromatography. Elution with 25% EtOAc/pentane provided product **7A** (438 mg, 0.86 mmol) in 86 % yield as a viscous oil. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.63 (d, J = 8.8 Hz, 1 H), 7.42 (d, J = 8.8 Hz, 1 H), 7.02 (d, J = 8.8 Hz, 1 H), 6.97 (d, J = 8.8 Hz, 1 H), 4.87 (s, 1 H), 3.89 (s, 3 H), 3.68 (s, 3 H), 3.64 (s, 3 H), 3.60 (s, 3 H), 3.32 (d, J = 20.0 Hz, 1 H), 3.10 (d, J = 20.0 Hz, 1 H), 0.14-0.11(bs, 18 H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 152.78, 151.46, 145.84, 145.75, 132.88, 129.19, 128.98, 128.50, 123.94, 123.64, 112.80, 112.46, 105.26, 104.57, 90.61, 87.61, 62.67, 60.95, 60.71, 56.03, 55.98, 23.88, 0.32, 0.09; IR (neat, cm^{-1}): 2957, 2175, 1488, 1281, 1029, 843; MS (ESI): m/z: 533.21 $[M^+Na]^+$.



8A

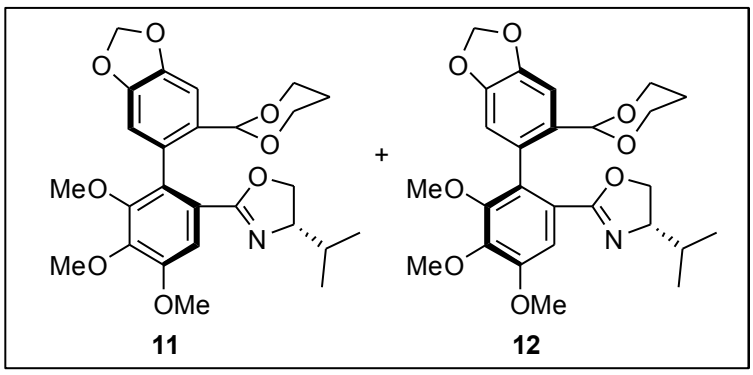
Synthesis of compound 8A: To a stirred solution of the compound **7A** (100 mg, 0.2 mmol) in absolute methanol (2.0 mL) was added K_2CO_3 (13 mg , 0.1 mmol) at 0 °C, and the mixture was gradually brought to room temperature over 12 h. The solvent was evaporated under reduced pressure to afford the crude product which was diluted with ethyl acetate and washed successively with water, brine and dried over Na_2SO_4. The organic layer was evaporated under reduced pressure to afford the crude product which was purified by column chromatography on silica gel using 20 % EtOAc/hexane as the eluent to yield the desilylated diyne (70 mg, 0.18 mmol) in 90 % yield as a viscous oil. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.67 (d, J = 8.5 Hz, 1 H), 7.35 (d, J = 8.5 Hz, 1 H), 7.04 (d, J = 8.5

Hz, 1 H), 6.97 (d, J = 8.5 Hz, 1 H), 4.92 (d, J = 2.0 Hz, 1 H), 3.91 (s, 3 H), 3.88 (s, 3 H), 3.64 (s, 3 H), 3.61 (s, 3 H), 3.24-3.20 (bs, 1 H), 3.25 (dd, J = 19.0, 3.0 Hz, 1 H), 3.08 (dd, J = 19.0, 2.5 Hz, 1 H), 2.48 (d, J = 2.0 Hz, 1 H), 2.05 (t, J = 2.5 Hz); ^{13}C NMR (100 MHz, $CDCl_3$) δ 152.63, 151.40, 145.62, 145.50, 132.63, 129.12, 128.70, 128.31, 124.13, 123.52, 112.63, 112.64, 83.53, 81.81, 73.60, 70.55, 62.13. 60.78, 60.47, 55.78, 55.77, 22.46; MS (ESI): m/z: 389.13 $[M+Na]^+$.

To a solution of the diyne from the previous step (366 mg, 1.0 mmol) in DME (4 mL) at 0 °C was added benzyl bromide (0.48 mL, 4.0 mmol) followed by *n*-tetrabutylammonium iodide (5 mg). After stirring at 0 °C for 20 min, to the above solution was added NaH (50% in paraffin oil, 96 mg, 2.0 mmol). The reaction mixture was stirred at 0 °C for 30 min and quenched with aq. sat. NH_4Cl solution. The reaction mixture was diluted with diethyl ether and washed successively with water, brine and dried over Na_2SO_4. The organic layer was evaporated under reduced pressure to afford the crude product which was purified by column chromatography on silica gel using 10 % EtOAc/hexane as the eluent to get the benzyl ether **8A** (378 mg, 0.83 mmol) in 83% yield as viscous oil. ^{1}H NMR (400 MHz, C_6D_6) δ 7.79 (d, J = 8.8 Hz, 1 H), 7.63 (d, J = 8.8 Hz, 1 H), 7.26 (d, J = 7.2 Hz, 1 H), 7.11 (t, J = 7.2 Hz, 1 H), 7.04 (t, J = 7.2 Hz, 1 H), 6.67 (d, J = 8.8 Hz, 1 H), 6.63 (d, J = 8.8 Hz, 1 H), 5.08 (d, J = 2.0 Hz, 1 H), 4.65 (d, J = 11.6 Hz, 1 H), 4.32 (d, J = 11.6 Hz, 1 H), 3,67 (s, 3 H), 3.65 (s, 3 H), 3.63 (dd, J = 19.6, 1.6 Hz, 1 H), 3.48 dd, J = 19.6, 1.6 Hz, 1 H), 3.34 (s, 3 H), 3.28 (s, 3 H), 2.16 (d, J = 2.4 Hz, 1 H), 1.95 (t, J = 2.8 Hz, 1 H); IR (neat, cm^{-1}): 2938, 1733, 1576, 1489, 1456, 1268; MS (ESI): m/z: 479.18 $[M+Na]^+$.

Synthesis of 9. See reference 3.

Synthesis of 10. See reference 4.



11 **12**

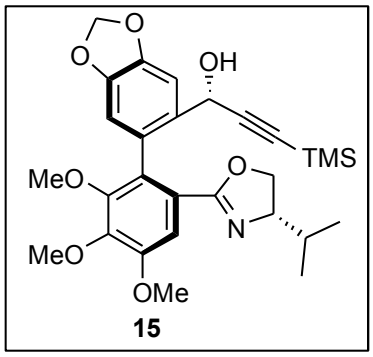
Synthesis of biaryls 11 and 12: To a solution of 4.4 g (15.3 mmol) of aryl bromide **10** in 40 mL of anhydrous THF was added 0.76 g (31.6 mmol) of Mg turnings, and then the reaction was brought to reflux under N_2. A solution of 3.04

g (16.2 mmol) of 1, 2-dibromoethane in 5 mL of THF was added in portions over 1 h. Refluxing was continued until there was complete disappearance of bromide as monitored by TLC (ca. 2 h). To this refluxing solution, a solution of 2.50 g (8.1 mmol) of oxazoline **9** in 20 mL of THF was added, and after refluxing for 18 h, oxazoline was completely consumed. The reaction was cooled to room temperature, quenched with 30 mL of sat. NH_4Cl, separated, the organic portion was washed with brine (2 X 30 mL), dried over $MgSO_4$, and concentrated to obtain a crude brown oil. The crude oil was purified by flash chromatography (EtOAc: hexanes = 1:10 to 2:1) to afford **11** as a yellow oil (1.57 g, 40%) and **12** as a yellow oil (1.77 g, 45%).

Compound **11**: $[\alpha]_D$ -33.6 (*c* 0.5, $CHCl_3$). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.18 (s, 1 H), 7.17 (s, 1 H), 6.62 (s, 1 H), 5.98 (d , *J* = 1.2 Hz, 1 H), 5.94 (d , *J* = 1.2 Hz, 1 H), 5.05 (s, 1 H), 4.11-4.05 (m, 1 H), 3.99-3.96 (m, 1 H), 3.95 (s, 3 H), 3.78-3.64 (m, 3 H), 3.60 (s, 3 H), 3.56-3.50 (m, 1 H), 2.11-2.04 (m, 1 H), 1.66-1.63 (m, 1 H), 1.27-1.22 (m, 1 H), 0.94 (d, 6.4 Hz, 3 H), 0.79 (d, 6.4 Hz, 3 H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 164.14, 152.55, 146.98, 146.94, 144.20, 131.19, 129.02, 127.06, 124, 24, 110.15, 108.32, 106.01, 101.02, 99.87, 72.63, 70.89, 67.21, 67.15, 60.89, 60.79, 60.38, 56.12, 32.78, 25.63, 21.03, 19.32, 18.40; IR (neat, cm^{-1}): 2960, 1649, 1484, 1415, 1369, 1274, 1259, 1108, 750; HRMS (ESI) [$M+H^{+}$]) m/z calcd for $C_{26}H_{32}NO_8$ 486.2122, found 486.2110.

Compound **12** : $[\alpha]_D$ –6.5 (*c* 0.675, $CHCl_3$). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.16 (s, 1 H), 7.15 (s, 1H), 6.66 (s, 1 H), 5.96 (d , *J* = 2 Hz, 2 H), 5.02 (s, 1 H), 4.081-4.00 (m, 4 H), 3.95 (s, 3 H), 3.93 (s, 3 H), 3.85-3.72 (m, 3 H), 3.71-3.65 (dt, *J* = 12 Hz, 2.4 Hz, 1 H), 3.59-3.52 (m, 1 H), 3.56 (s, 3 H), 2.12-2.04 (m, 1 H), 1.65-1.62 (m, 1 H), 1.26-1.23 (m, 1 H), 0.83 (d, 6.8 Hz, 3 H), 0.81 (d, 6.8 Hz, 3 H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 164.07, 152.53, 151.96, 147.05, 144.28, 131.02, 128.82, 126.93, 124.52, 110.32, 108.14, 106.07, 101.03, 99.91, 72.27, 70.59, 67.25, 67.10, 60.95, 60.77, 56.04, 32.66, 25.59, 18.69, 18.25; IR (neat, cm^{-1}): 2959, 1484, 1467, 1415, 1369, 1274, 1259, 1108, 1038, 764, 750. HRMS (ESI) [$M+H]^{+}$) calcd for $C_{26}H_{32}NO_8$ 486.2122, found 486.2113.

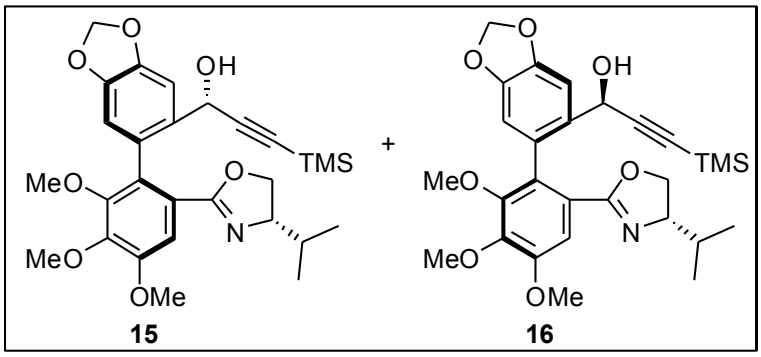
Hydrolysis of 11 to 13, and lithium trimethylsilylacetylide addition to 13. Synthesis of 15.



15

To a solution of 1.33 g (2.74 mmol) of biaryl oxazoline **11** in 30 mL of THF/CH_2Cl_2 (v:v = 1:1) at -10 ºC was added 9.1 mL (27.4 mmol) of 3M HCl solution dropwise. After stirring at this temperature for 1h, TLC showed full conversion of the oxazoline. The temperature was allowed to warm to 0 ºC and 3.79 g (27.4 mmol) of solid K_2CO_3 was added in portions to quench the reaction. Cold ether (0ºC) was used for quick extraction and the combined organic phase was dried over anhydrous $MgSO_4$ at 0 ºC. After filtration and removal of the solvent under vacuum at 0 ºC, the crude aldehyde as a yellow oil was used directly for the next step without further purification. The crude aldehyde was redissolved in 50 mL THF at -78 ºC. To this solution was added a solution of 27.4 mL (0.5 M, 13.7 mmol) of LiC≡CTMS in THF dropwise at -78ºC. After stirring for 30 min at this temperature, the solution was allowed to warm to room temperature before it was quenched with saturated aq. NH_4Cl solution. After ether extraction, drying with anhydrous $MgSO_4$, filtration and removal of the solvent under vacuum, the crude product was obtained as a yellow oil. Flash column (CH_2Cl_2) gave **15** as a light yellow foam (1.20 g, 83% yield). $[\alpha]_D$ 80 (*c* 0.25, $CHCl_3$); ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.41 (s, 1 H), 6.98 (s, 1 H), 6.41 (s, 1 H), 5.98 (d, *J* = 1.2 Hz, 1 H), 5.93 (d, *J* = 1.2 Hz, 1 H), 5.22 (s, 1 H), 4.27 (dd, *J* = 8.4 Hz, 10 Hz, 1 H), 3.97 (m, 1 H), 3.94 (s, 3 H),3.91 (s, 3 H), 3.67 (3 H), 1.37-1.34 (m, 1 H), 0.62 (d, *J* = 6.8 Hz, 3 H), 0.51 (d, *J* = 6.8 Hz, 3 H), 0.17 (s, 9 H).; ^{13}C NMR (100 MHz, $CDCl_3$) δ 163.37, 152.98, 151.97, 147,57,147.06, 144.58, 134.90, 128.79, 127.83, 123.21, 108.82, 108.23, 107.96, 105.67, 101.12, 89.62, 72.14, 70.31, 62.89, 61.01, 60.95, 56.18, 32.63, 18.31, 17.41, -0.07.; IR (neat, cm^{-1}): 3193, 2959, 2176, 1650, 1590, 1501, 1481, 1366, 1250, 1233, 1131, 1104, 1053, 1038, 844. HRMS (ESI) $[M+H]^+$ calcd for $C_{28}H_{36}NO_7Si$ 526.2256, found 526.2257.

A sample was recrystallized from CH_2Cl_2 by slow diffusion of hexane to a concentrated solution at room temperature. Crystallographic Information File is attached to this Supporting Information.

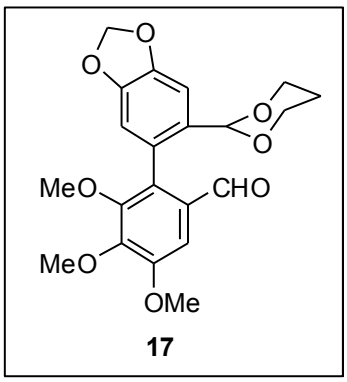


15 **16**

Synthesis of [13 + 14] from 12, and lithium trimethylsilylacetylide addition to [13 + 14]. Synthesis of [15 + 16]. To a solution of 200 mg (0.41 mmol) of biaryl oxazoline **12** in 10 mL of THF/CH_2Cl_2 (v: v = 1:1) at -10 ºC was added 1.4 mL (4.1 mmol) of 3M HCl solution dropwise. After stirring at this temperature for 1 h, TLC showed about 70% conversion of the oxazoline. The temperature was allowed to warm to 0 ºC and 560 mg (4.1 mmol) of solid K_2CO_3 was added by portion to quench the reaction. Cold ether (0 ºC) was used for extraction and the combined organic phase was dried over anhydrous $MgSO_4$ at 0ºC. After filtration and removal of the solvent under vacuum at 0ºC, the crude aldehyde as a yellow oil was used directly for the next step without further purification. The crude aldehyde was redissolved in 10 mL of THF at -78ºC. To this solution was added a solution of 4.1 mL (0.5 M, 2.05 mmol) of LiC≡CTMS in THF dropwise at -78ºC. After stirring for 30 min at this temperature, the solution was allowed to warm to room temperature before it was quenched with saturated aq. NH_4Cl solution. After ether extraction, drying with anhydrous $MgSO_4$, filtration and removal of the solvent under vacuum, the crude product was obtained as a yellow oil. Flash column (CH_2Cl_2) gave a mixture of two diastereomers **16** and **15** (molar ratio 3:1 by ^{1}HNMR) as a white foam (110 mg, 51% yield).

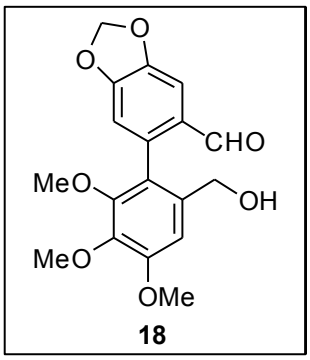
Compound **16** : ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.36 (s, 1H), 6.92 (s, 1H), 6.40 (s, 1H), 6.01 (d, *J* = 1.2 Hz, 1H), 5.98 (d, *J* = 1.2 Hz, 1H), 5.22 (s, 1H), 4.23 (dd, *J* =8.4 Hz, 10 Hz, 1H), 3.94 (s, 3H), 3.91 (s, 3H), 3.73 (m, 1H), 3.67 (3H), 1.73-1.69 (m, 1H), 0.85 (d, *J* = 6.8 Hz, 3H), 0.80 (d, *J* = 6.8 Hz, 3H), 0.18 (s, 9H). ^{13}C NMR (100 MHz, $CDCl_3$) 163.58, 153.07, 152.01, 147.56, 146.98, 144.40, 134.88, 128.31, 127.59, 123.67, 108.73, 108.25, 107.80, 105.56, 101.21, 89.63, 72.36, 70.13, 62.91, 62.75, 61.04, 56.20, 32.07, 18.72, 17.96, -0.07.

A sample was recrystallized from CH_2Cl_2 by slow diffusion of hexane to a concentrated solution at room temperature. Crystallographic Information File is attached to this Supporting Information.



17

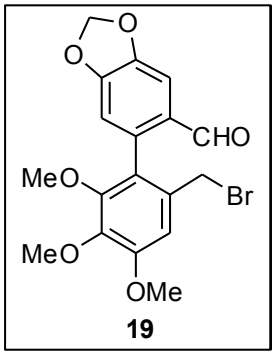
Hydrolysis of oxazolines [11 +12]. Synthesis of 17. A mixture of oxazoline **11** and **12** (2.0 g, 4.1 mmol) and MeOTf (1.36 g, 8.2 mmol) in CH_2Cl_2 (30 mL) was stirred at room temperature for 3 h. To this solution was added at 0°C L-Selectride (1 M in THF, 12.3 mL, 12.3 mmol). The mixture was stirred at 0 °C for 30 min, and then quenched with saturated aq. NH_4Cl solution. Ether extraction, drying with anhydrous $MgSO_4$ and removal of the solvent under vacuum gave an oil. To this oil was added CH_2Cl_2 (50 mL) and silica gel (10 g) were added. The reaction mixture was vigorously stirred for 18 h at room temperature. After removing the solvent under vacuum, the silica gel was loaded onto a column and column chromatography (EtOAc : Hexanes = 1: 5) affored the aldehyde **17** (1.32 g, 80% yield) as a yellow solid. ^{1}H NMR (400 MHz, $CDCl_3$) δ 9.45 (s, 1 H), 7.33 (s,1 H), 7.23 (s, 1 H), 6.66 (s, 1 H), 6.01(d , *J* = 1.2 Hz, 1 H), 5.99(d , *J* = 1.2 Hz, 1 H), 4.91 (s, 1 H), 4.10 (dd, *J* = 5.2 Hz, 7.2Hz, 1 H), 3.99 (s, 3 H), 3.96-3.91 (m, 1 H), 3.65-3.58 (m, 4 H), 3.42 (dt, *J* = 12Hz, , 2.4 Hz, 1 H), 2.09-1.99 (m, 1 H), 1.25-1.18 (m, 1 H).; ^{13}C NMR (100 MHz, $CDCl_3$) δ 190.93, 153.30, 151. 19, 147.91, 147.54, 147.27, 132.28, 131.46, 130.33, 125.16, 110.44, 106.54, 104.53, 101.47, 99.69, 67.28, 66.97, 61.03, 60.95, 56.10, 29.68, 25.40.; IR (neat, cm^{-1}): 2938, 2855, 1686, 1587, 1481, 1414, 1323, 1258, 1243, 1142, 1110, 1039, 764, 750.; HRMS (ESI) [M+Na^+] calcd for $C_{21}H_{22}O_8Na$ 425.1207, found 425.1201.



18

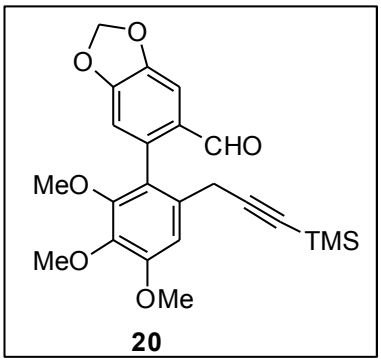
Synthesis of compound 18: To a solution of aldehyde **17** (450 mg, 1.12 mmol) in MeOH (10 mL) was added $NaBH_4$ (85 mg, 2.24 mmol) at 0 ºC. After removing the ice bath, the mixture was allowed to warm to room temperature and stirred for 1h. The reaction was quenched with sat. aq. NH_4Cl solution. Ether extraction, drying with anhydrous $MgSO_4$ and removal of the solvent under vacuum gave a crude oil. This oil was redissolved in THF/CH_2Cl_2 (20 mL, v:v = 1:1) at 0 ºC. To this solution was added 3M HCl solution (3.7 mL, 11.2 mmol) dropwise. After stirring at 0ºC for 1 h, solid K_2CO_3 (1.55 g, 11.2 mmol) was added by portion to quench the reaction. Ether extraction, drying with anhydrous $MgSO_4$ and removal of the solvent under vacuum gave an oil. Purification by flash column (EtOAc : Hexanes = 1: 2) gave **18** as a light yellow glassy solid

(317 mg, 82 % yield). ^{1}H NMR (400 MHz, $CDCl_3$) δ 9.49 (s, 1 H), 7.43 (S, 1 H), 6.92 (s, 1 H), 6.69 (s, 1 H), 6.09 (s, 2 H), 4.31 (dd, *J* = 12.8 Hz, 2 H), 3.92 (s, 3 H), 3.87 (s, 3 H), 3.615 (s, 3 H), 1.89(br, 1 H).; ^{13}C NMR (100 MHz, $CDCl_3$) δ 190.38, 153.81, 152.30, 151.39, 148.01, 141.29, 136.92, 135.05, 129.63, 122.60, 110.92, 106.94, 106.11, 102.15, 62.79, 60.95, 60.85, 56.07.; IR (neat, cm^{-1}): 3429, 2937, 1677, 1611, 1478, 1262, 1139, 1104, 1036, 764, 750.; HRMS (ESI) [M+Na^+] calcd for $C_{18}H_{18}O_7Na$ 369.0945, found 369.0931.



19

Synthesis of benzyl bromide 19: To a solution of PPh_3 (377 mg, 1.44 mmol) in CH_2Cl_2 (10 mL) was added CBr_4 (478 mg, 1.44 mmol) in one portion at room temperature. The mixture was stirred for 30 min and the mixture turned yellow. To the resulting mixture was added a solution of alcohol **18** (250 mg, 0.72 mmol) dropwise. The mixture was stirred for 12 h. Purification by direct flash column (EtOAc:Hexanes = 1: 3) gave **19** as a light yellow oil, which then was further purified by trituration with Et_2O to afford a white solid (276 mg, 94 % yield). ^{1}H NMR (400 MHz, $CDCl_3$) δ 9.50 (s, 1 H), 7.49 (s,1 H), 6.82 (s, 1 H), 6.77 (s, 1 H), 6.13(d , *J* = 1.2 Hz, 1 H), 6.11(d , *J* = 1.2 Hz, 1 H), 4.25 (d, *J* = 10.4 Hz, 1 H), 4.15 (d, *J* = 10.4 Hz, 1 H), 3.93 (s, 3 H), 3.89 (s, 3 H), 3.62 (s, 3 H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 189.98, 153.88, 152.29, 151.66, 148.25, 142.34, 136.09, 131.69, 129.78, 124.47, 110.73, 109.21, 106.25, 102.22, 60.99, 60.85, 56.13, 31.74.; IR (neat, cm^{-1}): 3430, 3003, 2935, 2840, 1681, 1612, 1478, 1403, 1333, 1266, 764, 750.; HRMS (ESI) [M+Na^+] calcd for $C_{18}H_{17}BrO_6Na$ 431.0101, found 431.0104.

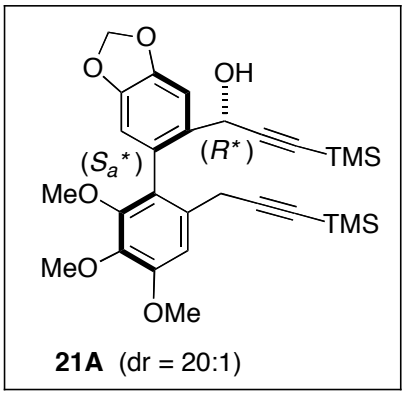


20

Synthesis of aldehyde 20: To a stirred solution of bromide **19** (860 mg, 2.11 mmol) in DME (25 mL) at room temperature was added $Pd_2(dba)_3{\cdot}CHCl_3$ (39 mg, 0.04 mmol) followed by tris-2-furylphosphine (39 mg, 0.02 mmol) and the reaction mixture was stirred for 10 min until it became a clear yellow solution. Then tributyltintrimethylsilylacetylene (845 mg, 2.18 mmol) in DME (3 mL) was added to the reaction mixture and stirring was continued at 80 ºC for 1 h. Afterwards, the reaction mixture was diluted with CH_2Cl_2 and washed with water, brine and dried over Na_2SO_4. The solvent was evaporated under reduced

pressure to afford the crude product, which was purified by silica gel column chromatography. Purification by flash column (EtOAc:Hexanes = 1: 5) gave **20** as a white solid (980 mg, 90 % yield). ^{1}H NMR (400 MHz, $CDCl_3$) δ 9.49(s, 1 H), 7.46 (s,1 H), 6.97 (s, 1 H), 6.67 (s, 1 H), 6.09 (t , *J* = 1.2 Hz, 1 H), 4.15 (d, *J* = 10.4 Hz, 1 H), 3.93 (s, 3 H), 3.88 (s, 3 H), 3.62 (s, 3 H), 3.28 (d, *J* = 19.2 Hz, 1 H), 3.13 (d, 19.2 Hz, 1 H), 0.14 (s, 9 H).; ^{13}C NMR (100 MHz, $CDCl_3$) δ 190.20, 153.60, 152.40, 151.53, 148.02 140. 74, 137.27, 130.63, 129.63, 123.14, 110.82, 107.85, 106.09, 103.55, 102.12, 87.67, 60.96, 60.48, 55.94, 24.85, -0.08.; IR (neat, cm^{-1}): 2958, 2850, 1682, 1613, 1478, 1403, 1242, 845.; HRMS (ESI) [M+Na^+] calcd for $C_{23}H_{26}O_6NaSi$ 449.1391, found 449.1380.

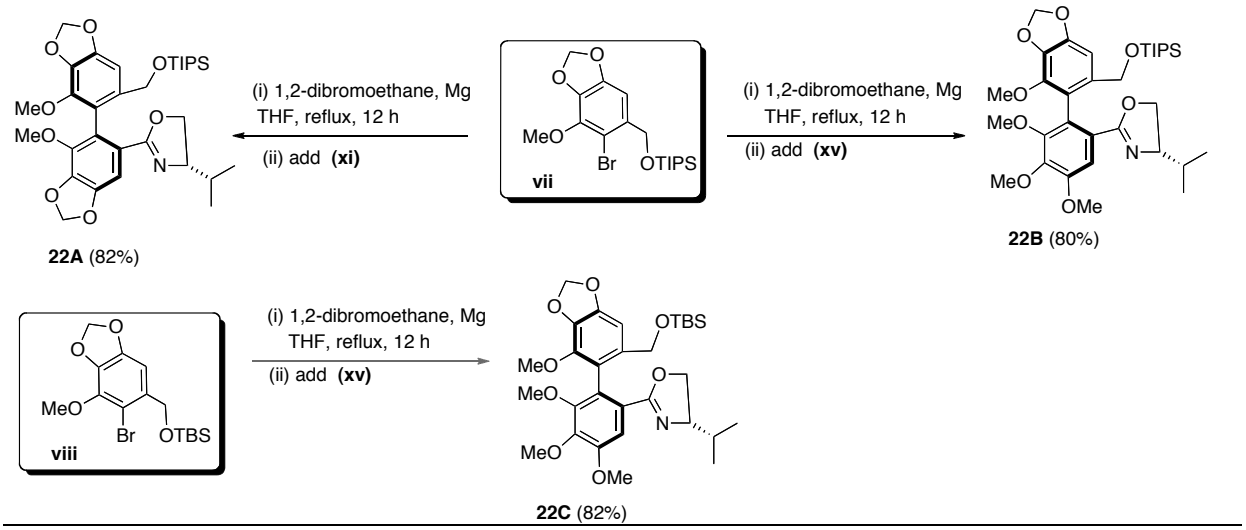
Addition of lithium trimethylsilylacetylide to 20. Synthesis of 21A. To a solution of aldehyde **20** (1.0 g, 2.35 mmol) in THF (50 mL) at -78ºC was added a solution of 27.4 mL (0.5 M, 13.7 mmol) of LiC≡CTMS in THF dropwise. After stirring for 30 min at this temperature, the solution was allowed to warm to room temperature before it was quenched with saturated aq. NH_4Cl solution. After ether extraction, drying with anhydrous $MgSO_4$, filtration and removal of the solvent under vacuum, the crude product was obtained as a yellow oil. Flash column (EtOAc: Hexanes = 1: 4) gave **21A** as a yellow solid (987 mg, 80% yield). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.40 (s, 1H), 7.10 (s, 1H), 6.58 (s, 1H), 6.03(s, 2H), 4.97 (s, 1H), 3.93 (s, 3H), 3.87 (s, 3H), 3.58 (s, 3H), 3.39 (s, 1H), 3.33(d, *J* = 15.6 Hz, 1H), 3.18 (d, *J* = 15.6 Hz, 1H).; ^{13}C NMR (100 MHz, $CDCl_3$) δ 153.15, 150.51, 147.81, 147.66, 140.89, 133.93, 130.92, 127.65, 125.27, 109.88, 108.44, 108.13, 104.69, 104.69, 104. 11, 101.44, 90.75, 87.97, 62.75, 61.47, 61.17, 55.91, 24.60, 0.06, -0.14.; IR (neat, cm^{-1}): 3442, 2958, 2898, 2175, 1599, 1480, 1462, 1402, 1250, 1232, 1140, 1104, 1042, 843, 761.; HRMS (ESI) [M+Na^+] calcd for $C_{28}H_{36}O_6NaSi_2$ 547.1943, found 547.1940.



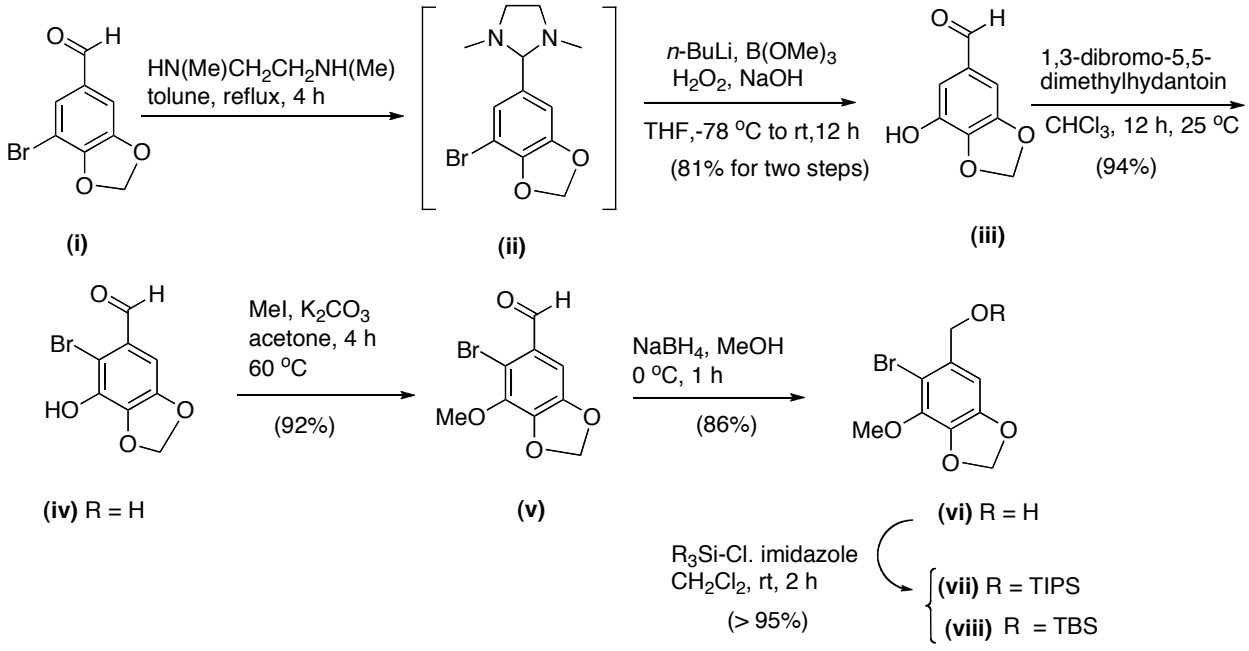
21A (dr = 20:1)

A sample was recrystallized from CH_2Cl_2 by slow diffusion of hexane to a concentrated solution at room temperature. Crystallographic Information File is attached to this Supporting Information.

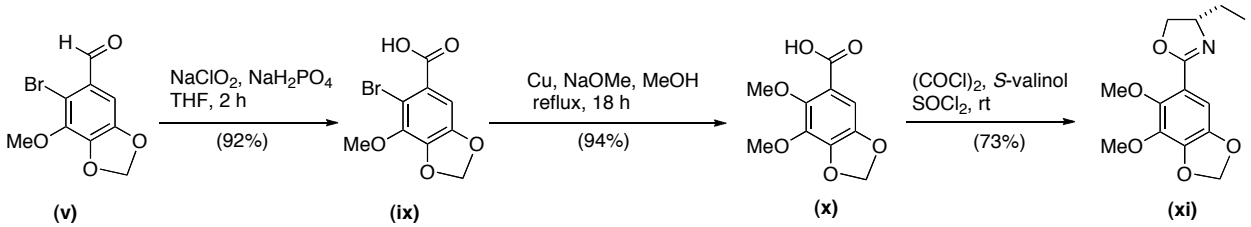
Synthesis of Oxazoline Precursor 22A, 22B and 22C (Scheme 6 in the paper). The oxazoline precursor **22A** was prepared according to the route described in the scheme below:

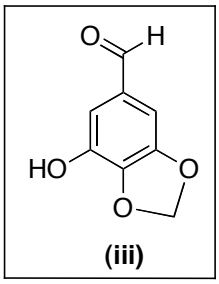


A. Synthesis of Arylbromides (vii) and (viii)

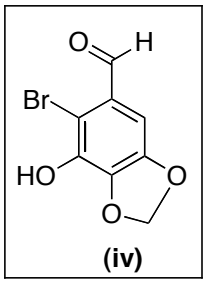


B. Synthesis of Oxazolines (xi) and (xv)



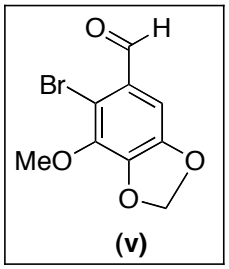

(iii)

Synthesis of the compound (iii). Solution of arylbromide **(i)** (3.68 g, 16.0 mmol) and *N,N'*-dimethylethylenediamine (2.5 mL, 24.0 mmol) in toluene (60 mL) was refluxed at 110 ^{o}C for 2 h with azeotropic removal of the H_2O/toluene mixture by using the Dean Stark trap. After cooling reaction mixture to rt, the organic solvent was evaporated under reduced pressure to afford the crude product **(ii)**. To a solution of compound **(ii)** in THF (60 mL) at –78 °C was added dropwise *n*-BuLi (2.5 M in hexane, 9.6 mL, 24 mmol). After stirring at the same temperature for 15 min, trimethylborate (3.2 mL, 28.8 mmol) was added slowly and the solution was allowed to warm to rt over 2 h. Solution of 0.5 N NaOH (10 mL) followed by 30 % aqueous H_2O_2 (8.0 mL) were added to the reaction mixture and stirring was continued for further 2 h. The reaction mixture was diluted with ethyl acetate and washed successively with water, brine and dried over Na_2SO_4. The organic layer was evaporated under reduced pressure to afford the crude product which was purified by column chromatography on silica gel using 25 % EtOAc/hexane as the eluent to yield **(iii)** in 81% yield (2.02 g, 13 mmol) as solid. ^{1}H NMR (250 MHz, acetone-d_6): δ 9.76 (s, 1 H), 7.12 (d, J = 1.0 Hz, 1 H), 6.94 (d, J = 1. 0 Hz, 1 H), 6.12 (s, 2 H), 2.98-2.73 (bs, 1 H), -0.01 (s, 9 H).; ^{13}C NMR (100 MHz, acetone-d_6): δ 190.05, 149.82, 141.15, 140.02, 132.17, 115.18, 102.44, 100.47; MS (ESI): m/z: 189.02 $[M+Na]^{+}$.


(iv)

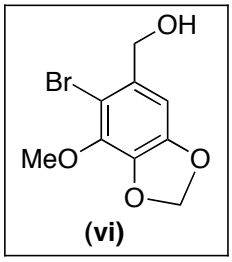
Synthesis of the compound (iv): Substrate **(iii)** (332 mg, 2.0 mmol) in $CHCl_3$ (20 mL) at rt was added solid 1,3-dibromo-5,5-dimethylhydantoin (DBDMH,

291 mg, 1.02 mmol) in parts. Upon initial addition of the DBDMH, the solution became red or deep brown colored, and the next portion of DBDMH was added after the disappearance of color and so on. After stirring at rt for 12 h, the reaction mixture was washed successively with 1 N HCl, water, brine and dried over Na_2SO_4. The organic layer was evaporated under reduced pressure to afford the crude product which was purified by column chromatography on silica gel using 35% EtOAc/hexane as the eluent to yield **(iv)**) in 94 % yield (456 mg, 1.88 mmol) as a white solid. ^{1}H NMR (250 MHz, DMSO-d_6): δ 10.82-10.64 (bs, 1 H), 10.10 (s, 1 H), 6.92 (s, 1 H), 6.17 (s, 2 H).; ^{13}C NMR (100 MHz, DMSO-d_6): δ 190.94, 148.57, 141.39, 138.55, 128.14, 112.67, 103.30, 100.68.; MS (ESI): m/z: 266.92 $[M+Na]^+$.



(v)

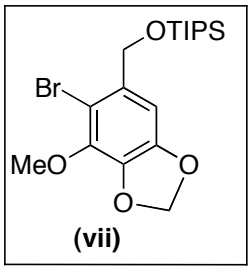
Synthesis of the compound (v): To a stirred solution of the compund **(iv)** (486 mg, 2.0 mmol) in acetone (10 mL) was added K_2CO_3 (552 mg, 4.0 mmol) followed by methyl iodide (1.0 mL) were added at room temperature, and the mixture was gradually brought to 60 ^{o}C temperature and stirred for 4 h. The solvent was evaporated under reduced pressure to afford the crude product, which was diluted with ethyl acetate and washed successively with water, brine and dried over Na_2SO_4. The organic layer was evaporated under reduced pressure to afford the crude product which was purified by column chromatography on silica gel using 15 % EtOAc/hexane as the eluent to get **(v)** in 92% yield (472 mg, 1.84 mmol) as a solid. ^{1}H NMR (250 MHz, $CDCl_3$): δ 10.23 (s, 1 H), 7,14 (s, 1 H), 6.06 (s, 2 H), 4.04 (s, 3 H).; ^{13}C NMR (100 MHz, $CDCl_3$) δ 190.87, 149.38, 143.16, 140.50, 128.78, 115.95, 103.30, 102.90, 60.57.; MS (ESI): m/z: 280.93 $[M+Na]^+$.



(vi)

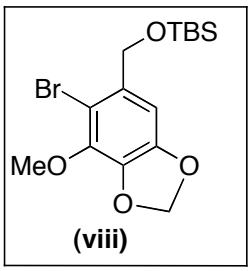
Synthesis of the compound (vi): To a solution of aldehyde **(v)** (1.02 g, 4.0 mmol) in MeOH (15 mL) at 0 °C was added $NaBH_4$ (296 mg, 8.0 mmol) in portions. After stirring at the same temperature for 1 h, the reaction was quenched by the addition of aq. NH_4Cl (5.0 mL). The organic solvent was evaporated under reduced pressure to afford the crude product, which was diluted with ethyl acetate and washed successively with water, brine and dried over Na_2SO_4. The organic layer was evaporated under reduced pressure to afford the crude product which was purified by column chromatography on silica gel using 35 % EtOAc/hexane the eluent to get the

compound **(vi)** in 86% yield (890 mg, 3.5 mmol) as a viscous oil. ^{1}H NMR (400 MHz, $CDCl_3$): δ 6.72 (s, 1 H), 5.95 (s, 2 H), 4.63 (s, 2 H), 4.0 (s, 3 H).; ^{13}C NMR (100 MHz, $CDCl_3$) δ 148.97, 140.63, 136.96, 134.36, 107.3, 103.6, 101.89, 65.48, 60.33.; MS (ESI): m/z: 284.95 $[M+Na]^+$.



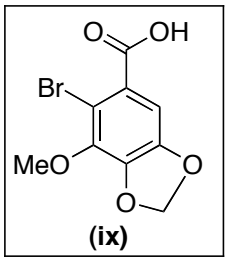
(vii)

Synthesis of the compound (vii): To a stirred solution of compound **(vi)** (647 mg, 2.5 mmol) in CH_2Cl_2 (10 mL) at 0 ^{o}C was added imidazole (340 mg, 5.0 mmol) followed by TIPS-Cl (0.64 mL, 3.0 mmol) and the reaction mixture was gradually brought to room temperature over 2 h. Afterwards, the reaction mixture was diluted with CH_2Cl_2 and washed subsequently with water, brine and dried over Na_2SO_4. The solvent was evaporated under reduced pressure to afford the crude product, which was purified by silica gel column chromatography. Elution with 5 % EtOAc/hexane provided product **(vii)** (1.0 g, 2.37 mmol) in 95% yield as a viscous oil. ^{1}H NMR (250 MHz, $CDCl_3$): δ 6.90 (s, 1 H), 5.94 (s, 2 H), 4.70 (s, 2 H), 4.01 (s, 3 H), 1.18-1.05 (m, 21 H).; ^{13}C NMR (100 MHz, $CDCl_3$) δ 148.96, 140.16, 136.08, 135.42, 104.92, 102.20, 101.64, 65.12, 60.30, 18.26, 12.21.; MS (ESI): 441.08 m/z: $[M+Na]^+$.



(viii)

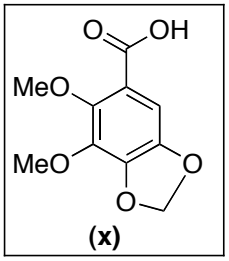
Synthesis of the compound (viii): Following the similar procedure, compound **23B** was synthesized from alcohol **(vi)** in 99% yield as a colorless oil. ^{1}H NMR (400 MHz, $CDCl_3$): δ 6.84 (s, 1H), 5.96 (s, 2H), 4.64 (s, 2H), 4.02 (s, 3H), 0.96 (s, 9H), 0.13 (s, 6H).; ^{13}C NMR (100 MHz, $CDCl_3$) δ 148.71, 140.02, 135.99, 134.98, 105.05, 102.19, 101.45, 64.74, 60.09, 25.95, 18.39, -5.34.; IR (neat, cm^{-1}): 2953, 2884, 2856, 1603, 1480, 1412, 1258, 1128, 838, 766, 750.; HRMS (ESI) $[M+Na^+]$ calcd for $C_{15}H_{23}BrO_4SiNa$ 399.0422, found 399.0424.



(ix)

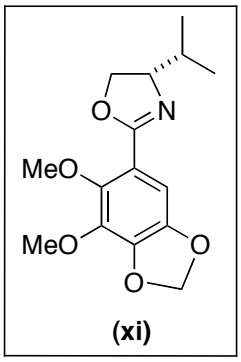
Synthesis of the compound (ix): To a stirred solution of **(v)** (837 mg, 3.2 mmol) in 1:1 *t*-BuOH and H_2O (10 mL), NaH_2PO_4 (1.15 g, 9.6 mmol) followed by $NaClO_2$ (870 mg, 9.6 mmol) were added. After stirring the reaction mixture at the same temperature for 15 min, 2,3-dimethyl-2-butene (32 mL, 1 M in THF) was added to it and stirring was continued at rt for additional 1 h. The reaction mixture was diluted with CH_2Cl_2 and washed subsequently with water, brine and dried over Na_2SO_4. The solvent was evaporated under reduced pressure to

afford the product **(ix)** (815 mg, 3.0 mmol) in 92 % yield, which was used in next without further purification. ^{1}H NMR (250 MHz, $CDCl_3$): δ 7.0 (s, 1 h), 5.94 (s, 2 H), 3.91 (s, 3 H).; ^{13}C NMR (100 MHz, $CDCl_3$) δ 167.67, 148.26, 140.90, 140.42, 127.09, 109.05, 105.9, 102.39, 60.33,; MS (ESI): m/z: 298.92 $[M+Na]^+$.



(x)

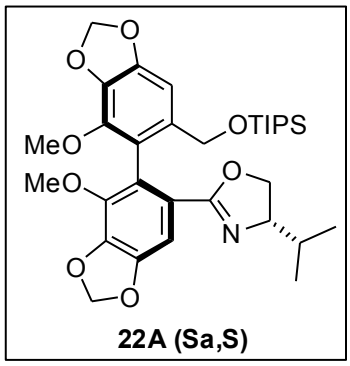
Synthesis of the compound (x). To a stirred solution of Na metal (368 mg, 16 mmol) dissolved in anhydrous methanol (21 mL) was added the acid **(ix)** (815 mg. 3.0 mmol). Once the acid **(ix)** had dissolved, Cu powder (102 mg, 1.6 mmol) was added and the mixture refluxed for 12 h. The mixture was cooled, filtered through a bed of Celite, and concentrated in vacuo to give a crude white solid. This was dissolved in 100 mL of water and acidified to pH 3 with concentrated HCI. The solution was extracted with CH_2Cl_2 (2 x 100 mL), and the combined organic lawyer was washed with water, brine and dried over Na_2SO_4. The solvent was evaporated under reduced pressure to afford the product **(x)** in 94 % yield (637 mg, 2.82 mmol), which was used in next step without further purification. ^{1}H NMR (250 MHz, acetone-d_6): δ 7.0 (s, 1 H), 6.10 (s, 2 H), 4.01 (s, 3 H), 4.91 (s, 3 H), 2.88-2.71 (bs, 1 H).; ^{13}C NMR (100 MHz, $CDCl_3$) δ 165.17, 147.79, 145.79, 142.62, 136.66, 115.17, 104.46, 102.71, 63.01, 60.49; MS (ESI): m/z: 249.03 $[M+Na]^+$.



(xi)

Synthesis of oxazoline (xi): To a solution of the acid **x** (1.18 gm, 5.25 mmol) in anhydrous CH_2Cl_2 (20 mL) was added oxalyl chloride (1.3 mL, 15.75 mmol) and then 2 drops of DMF. After stirring the reaction mixture under N_2 at rt for 12 h, the organic solvents were removed in vacuo to afford the acid chloride, which was then dissolved in anhydrous CH_2Cl_2 (10 mL) and the solution was added to a solution of *S*-valinol (702 mg, 6.8 mmol) and Et_3N (2.72 mL, 21 mmol) in anhydrous CH_2Cl_2 (10 mL) at 0 ^{o}C. The mixture was then stirred at room temperature for 6 h at which time the solvent was evaporated, and the oily residue was dissolved in 50 mL of ethyl acetate. The precipitated trimethylamine hydrochloride was filtered away, and concentration of the filtrate gave the amide. This was dissolved in CH_2Cl_2 (20 mL), and thionyl chloride (5 mL) was added. After stirring at rt, the reaction mixture was cooled to 0 ^{o}C and carefully quenched with H_2O (20 mL) and

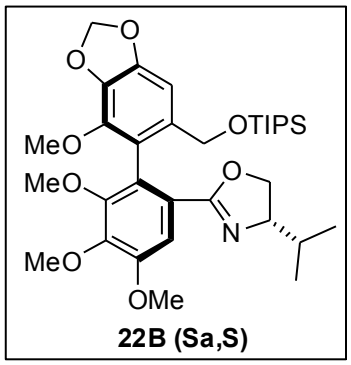
followed by 4 N NaOH (20 mL). After separating organic lawyer, the aqueous layer was extracted with CH_2Cl_2, the combined organic portions was washed with brine and dried over Na_2SO_4, filtered, and the solvent removed. On occasion, this was contaminated with the aliphatic chloride, therefore, to induce ring closure, this mixture was refluxed with K_2CO_3 in (10:1, CH_3CN-H_2O), cooled, the CH_3CN removed, and the aqueous portion extracted with CH_2Cl_2. The solvent was evaporated under reduced pressure to afford the crude which was purified by column chromatography on silica gel using 35 % EtOAc/hexane as the eluent to get the oxazoline **(xi)** in 73% yield (1.13 g, 3.82 mmol) as a viscous oil. ^{1}H NMR (400 MHz, $CDCl_3$): δ 6.91 (s, 1 H), 5.94 (s, 2 H), 4.36-4.31 (m, 1 H), 4.08-4.05 (m, 2 H), 3.98 (s, 3 H), 3.80 (s, 3 H), 1.85-1.81 (m, 1 H), 1.0 (d, J = 3 Hz, 3 H), 0.91 (d, J = 6.5 Hz, 3 H).; ^{13}C NMR (125 MHz, $CDCl_3$): δ 162.01, 148.16, 144.73, 140.68, 138.18, 115.70, 103.71, 102.13, 72.73, 70.11, 62.16, 60.67, 33.06, 19.03, 18.39.; $[\alpha]_D$ – 46.1 (*c* 0.78, $CHCl_3$).; IR (neat): $\tilde{\nu}$ = 2938, 1647, 1614, 1415 cm^{-1}.; MS (ESI): 294.14 m/z: $[M+H]^+$.



22A (Sa,S)

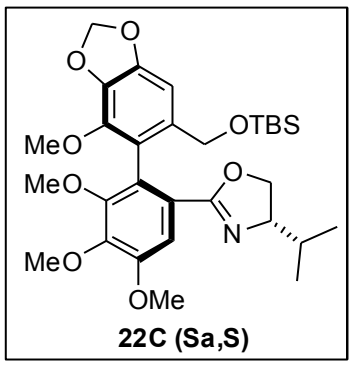
General Synthesis of Enantiopure Biphenyloxazoline (22A): To a solution of aryl bromide **(vii)** (2.143 g, 5.12 mmol) in THF (15 mL) was added Mg turnings (249 mg, 10.24 mmol), and then the reaction was bought to reflux under N_2. A solution of 1,2-dibromoethane (962 mg, 5.12 mmol) in THF (2 mL) was added in portions over 1 h. Refluxing was continued until there was complete disappearance of bromide **(vii)** as monitored by GC. To this refluxing solution, a solution of oxazoline **(xi)** (1.94 g, 3.84 mmol) in THF (7 mL) was added, and reflux was maintained until oxazoline was completely consumed. The reaction mixture was cooled to room temperature and quenched by the addition of a saturated aqueous NH_4Cl solution. The aqueous layer was extracted with EtOAc (2 x 100 mL) and the organic layer washed with water, brine and dried over Na_2SO_4. The solvent was evaporated under reduced pressure to afford the crude product which was purified by silica gel chromatography using 20 % EtOAc/hexane as the eluent to yield the major compound **22A *(Sa,S)*** (1.88 g, 3.14 mmol) in 82 % yield as yellow viscous oil. **Major compound 22A *(Sa,S)*:** $[\alpha]_D$ – 34.2 (*c* 0.88, $CHCl_3$).; ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.05 (s, 1H), 6.88 (s, 1 H), 6.00 (d, *J* = 1.5 Hz, 1 H), 5.98 (d, *J* = 1.5 Hz, 1 H), 5.92 (d, *J* = 1.5 Hz, 1 H), 5.91 (d, *J* = 1.5 Hz, 1 H), 4.38 (d, *J* = 14 Hz,

1 H), 4.35 (d, *J* = 14 Hz, 1 H), 4.03 (dd, *J* = 10, 9.5 Hz, 1 H), 3.81- 3.72 (m, 8 H), 1.57-1.52 (m, 1 H), 1.07-0.98 (m, 21 H), 0.80 (d, *J* =, 8.5 Hz, 3 H), 0.74 (d, *J* = 8 Hz, 3 H).; ^{13}C NMR (100 MHz, $CDCl_3$) δ 163.49, 148.77, 148.67, 141.40, 141.14, 138.95, 135.47, 135.07, 123.44, 123.30, 119.67, 104.74, 101.74, 100.91, 100.80, 72.73, 70.49, 62.90, 59.94, 59.82, 33.08, 18.96, 18.53, 18.26, 12.22.; MS (ESI): m/z: 600.309 $[M+Na]^+$.



22B (Sa,S)

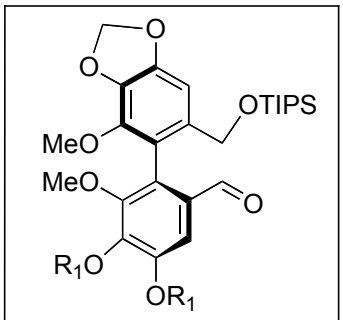
Synthesis of compound 22B *(Sa,S)*: Enantiopure compound **22B *(Sa,S)*** was synthesized from arylbromide **(vii)** and oxazoline **(xv)** using the procedure described for the synthesis of **22A(*Sa,S*)** in 80 % isolated yield. **Major compound 22B *(Sa,S)*** : $[\alpha]_D$ – 43.40 (*c* 1.1, $CHCl_3$).; ^{1}H NMR (400 MHz, $CDCl_3$): δ 2.17 (s, 1 H), 6.89 (s, 1 H), 5.92 (d, *J* = 2.0 Hz, 2 H), 4.30 (dd, *J* = 21.6, 13.6 Hz, 1 H), 4.07 (t, *J* = 8.8 Hz, 1 H), 3.90 (s, 3 H), 3.87 (s, 3 H), 3.83-3.74 (m, 4 H), 6.89 (t, *J* = 8.8 Hz, 1 H), 3.59 (s, 3 H), 1.62-1.56 (m, 1 H), 1.06-0.96 (m, 15 H), 0.82 (d, *J* = 8.2 Hz, 3 H), 0.75 (d, *J* = 8.2 Hz, 3H).; ^{13}C NMR (100 MHz, $CDCl_3$) δ 163.75, 152.62, 151.78, 148.73, 144.43, 141.02, 135.36, 134.81, 124.69, 123.56, 119.63, 108.95, 100.85, 100.57, 72.72, 70.44, 62.93, 61.07, 60.90, 59.68, 56.26, 33.01, 18.99, 18.42, 18.23, 12.15.; IR (neat): $\tilde{\nu}$ = 1652, 1457, 1399, 1049, 737, 685 cm^{-1}.; MS (ESI): 616.338 m/z: $[M+H]^+$. **Minor compound 22B *(Ra,S)***: $[\alpha]_D$ + 13.22 (*c* 1.8, $CHCl_3$).; ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.19 (s, 1 H), 6.88 (s, 1 H), 5.91 (d, *J* = 2.8 Hz, 2 H), 4.31-4.24 (m, 2 H), 4.07-3.78 (m, 11 H), 3.59 (s, 3 H), 1.66-1.50 (m, 1 H), 1.18-0.85 (m, 21 H), 0.77 (d, *J* = 7.8 Hz, 3 H), 0.75 (d, *J* = 7.8 Hz, 3 H).; ^{13}C NMR (125 MHz, $CDCl_3$): δ 152.65, 151.80, 148.77, 141.32, 134.90, 134.76, 119.55, 109.0, 100.811, 100.402, 71.61, 70.36, 62.83, 61.04, 60.88, 59.60, 32.87, 18.83, 18.20, 12.13.; MS (ESI): 616.338 m/z: $[M+H]^+$.



22C (Sa,S)

Synthesis of compound 22C *(Sa,S)*: Enantiopure compound **22C *(Sa,S)*** was synthesized from arylbromide **(viii)** and oxazoline **(xv)** using the procedure described for the synthesis of **22C (*Sa,S*)** in 82 % isolated yield. **Major compound 22C *(Sa,S)***: $[\alpha]_D$ –48.5 (*c* 0.935, $CHCl_3$).; ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.18 (s, 1H), 6.81 (s, 1 H), 5.93-5.92 (m, 2 H), 4.28 (d, *J* = 13.6 Hz, 1 H), 4.21 (d, *J* = 13.6 Hz, 1H), 4.11-4.07 (m, 1H), 3.93 (s, 3H), 3.91 (s, 3H), 3.78 (s, 3 H), 3.69 (t, *J* = 7.6 Hz, 1 H), 3.62 (s, 3 H), 1.62-1.55 (m, 1 H), 0.85 (s, 9 H), 0.83 (d, *J* = 6.8 Hz,

1 H), 0.77(d, *J* = 6.8 Hz, 1 H), -0.05 (s, 3 H), -0.06 (s, 3 H).; ^{13}C NMR (100 MHz, $CDCl_3$) δ 163.59, 152.47, 151.63, 148.52, 144.21, 140.86, 134.92, 134.72, 124.46, 123.36, 119.68,108.72, 100.66, 100.60, 72.52, 70.23, 62.63, 60.85, 60.68, 59.45, 56.01, 32.74, 25.94, 18.74, 18.31, 18.21-5.42, -5.45.; IR (neat, cm^{-1}): 2952, 2856, 1651, 1622, 1454, 1368, 1278, 1079, 980, 775, 749.; HRMS (ESI) $[M+H]^+$ calcd for $C_{30}H_{44}NO_8Si$ 574.2831, found 574.2830.

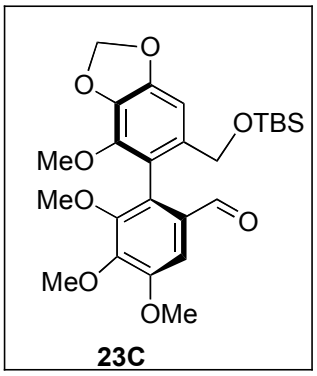


23A 51A $R_1 \sim R_2 = CH_2$
23B 51B $R_1 = R_2 = Me$

Synthesis of the compound 23A: To a solution of **22A *(Sa,S)*** (610 mg, 2.0 mmol) in CH_2Cl_2 (10 mL) at 0 °C was added 2,6-di-*tert*-butylpyridine (573 mg, 3.0 mmol) followed by methyltriflate (0.26 mL, 2.4 mmol), and the mixture was stirred at 0 °C for 30 min. After addition of L-Selectride (1 M in THF, 6.0 mL, 6.0 mmol), the mixture was further stirred at 0°C for 30 min, and then a saturated aqueous solution of citric acid and a small amount of silica gel were added. The reaction mixture was vigorously stirred for 4 h at 0 °C and was brought back to room temperature. The mixture was extracted with CH_2Cl_2. The combined organic extracts were dried over Na_2SO_4 and concentrated under reduced pressure to afford the crude product which was purified by silica gel chromatography using 10% EtOAc/hexane as the eluent to yield compound **(23A,** 935 mg, 1.81 mmol) in 88% yield as a viscous oil. $[\alpha]_D$ – 4.8 (*c* 1.5, $CHCl_3$); ^{1}H NMR (400 MHz, $CDCl_3$): δ 9.48, (s, 1 H), 7.24 (s, 1 H), 6.95 (s, 1 H),), 6.11 (d, *J* = 1.5 Hz, 1 H), 6.10 (d, *J* = 1.2 Hz, 1 H), 6.02 (d, *J* = 1.2 Hz, 1 H), 6.01 (d, *J* = 1.2 Hz, 1 H), 4.38 (d, *J* = 13.5 Hz, 1 H), 4.26 (d, *J* = 13.5 Hz, 1 H), 3.87 (s, 3 H), 3.86 (s, 3 H), 1.08-0.98 (m, 21 H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 190.66, 149.84, 149.60, 142.16, 141.39, 141.15, 135.78, 135.20, 130.16, 129.09, 115.83, 102.21, 101.82, 101.31, 101.05, 63.13, 59.99, 59.70, 27.12, 18.16, 11.96.; IR (neat, cm^{-1}): 2943, 1682, 1475, 1285.; MS (ESI): m/z: 539.209 $[M+Na]^+$.

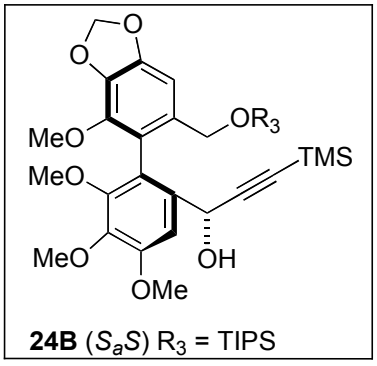
Compound 23B: Following the similar procedure, compound **23B** was synthesized from biphenyloxazoline **22B *(Sa,S)*** in 88 % isolated yield. $[\alpha]_D$ + 55.20 (*c* 0.5, $CHCl_3$).; ^{1}H NMR (400 MHz, $CDCl_3$): δ 9.52 (s, 1 H), 7.32 (s, 1 H), 6.89 (s, 1 H), 5.97 (t, *J* = 2.2 Hz, 2 H), 4.38 (d, *J* = 12.8 Hz, 1 H), 4.17 (d, *J* = 12.8 Hz, 1 H), 3.94 (s, 3 H), 3.93 (s, 3 H), 3.82 (s, 3 H), 3.59 (s, 3 H), 1.01-0.92 (m, 21 H).; ^{13}C NMR (125 MHz, $CDCl_3$) δ 191.41, 153.40, 151.60, 149.81, 147.81, 141.29, 135.86, 134.93, 130.24, 128.23, 115.68, 105.21, 101.76, 101.27, 63.23, 61.22, 61.06,

59.63, 56.28, 18.15, 12.08; IR (neat, cm^{-1}): 1684, 1456, 1323, 1197, 1049, 881, 735.; MS (ESI): m/z: 555.249 $[M+Na]^+$.



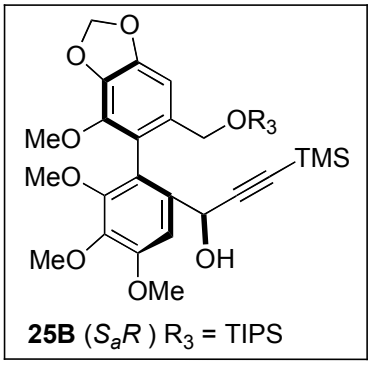
23C

Synthesis of compound 23C. Following the similar procedure, compound **23C** was synthesized from biphenyloxazoline **22C** ***(Sa,S)*** in 90 % yield. $[\alpha]_D$ –13.3 (*c* 0.87, $CHCl_3$). ^1H NMR (400 MHz, $CDCl_3$): δ 9.54 (s, 1H), 7.35 (s, 1H), 6.83 (s, 1H), 6.00 (d, *J* = 1.2 Hz, 1H), 5.99 (d, *J* = 1.2 Hz, 1H), 4.29 (d, *J* = 12.8 Hz, 1H), 4.13 (d, *J* = 12.8 Hz, 1H), 3.97 (s, 3H), 3.96 (s, 3H), 3.84 (s, 3H), 3.63 (s, 3H), 0.83 (s, 9H), -0.07 (s, 3H), -0.08 (s, 3H).; ^{13}C NMR (100 MHz, $CDCl_3$) δ 191.20, 153.23, 151.38, 149.56, 147.17, 135.29, 134.92,130.09,128.07, 115.93, 104.99, 101.88, 101.09,67.96, 62.97, 61.00,60.83, 59.42, 56.05,25.88, 25.61, 18.32, -5.52, -5.54.; IR (neat, cm^{-1}): 2956, 2866, 1645, 1260, 1152, 765, 750.; HRMS (ESI) $[M+Na^+]$ calcd for $C_{25}H_{34}O_8SiNa$ 513.1915, found 513.1917.



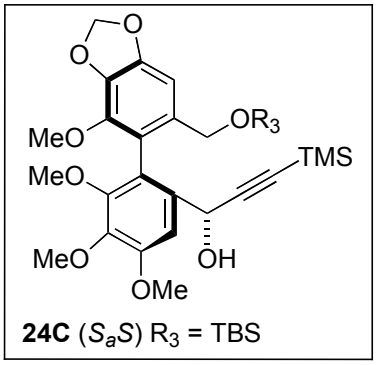
24B (S_aS) R_3 = TIPS

Synthesis of the compound 24B: To a stirred solution of compound **(23B)** (1.06 g, 2.0 mmol) in dry THF (10 mL) at –78 °C was added lithium trimethylsilylacetylide (0.5 M in THF, 8.0 mL, 4.0 mmol). After stirring at –78 °C for 30 min, the reaction was quenched by the addition of aq. sat. NH_4Cl solution. The reaction mixture was diluted with CH_2Cl_2 and washed with water, brine and dried over Na_2SO_4. The solvent was evaporated under reduced pressure to afford the crude product, which was purified by silica gel column chromatography. Elution with 25 % EtOAc/pentane provided the **major product [24B(*Sa,S*)]** (1.01 g, 1.6 mmol) in 80% yield and **minor product [25B(*Sa,R*)]** (151 mg, 0.24 mmol) in 12% yield; **major product [(24B(*Sa,S*)]**: $[\alpha]_D$ – 21.5 (*c* 1, $CHCl_3$).; ^1H NMR (400 MHz, $CDCl_3$): δ 7.27 (s, 1 H), 6.95 (s, 1 H), 5.98 (d, *J* = 1.5 Hz, 1 H), 5.95 (d, *J* = 1.5 Hz, 1 H), 5.01 (d, *J* = 2.0 Hz, 1 H), 4.37 (d, *J* = 13.5 Hz, 1 H), 4.12 (d, *J* = 13.5 Hz, 1 H), 3.92 (s, 3 H), 3.85 (s, 3 H), 3.83 (s, 3 H), 3.60 (s, 3 H), 2.92 (d, *J* = 2.0 Hz, 1 H), 1.04-0.86 (m, 21 H), 0.15 (s, 9 H).; ^{13}C NMR (100 MHz, $CDCl_3$) δ 153.51, 151.30, 149.46, 142.47, 140.36, 136.13, 135.68, 135.21, 120.95, 117.73, 106.93, 104.96, 101.96, 101.27, 90.90, 63.09, 62.94, 61.01, 60.89, 59.96, 56.00, 18.20, 14.38, 12.10, 0.02.; MS (ESI): m/z: 653.2505 $[M+Na]^+$.



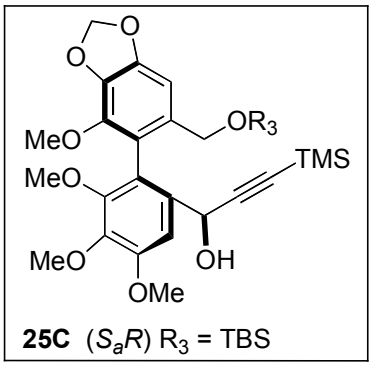
25B (S_aR) R_3 = TIPS

minor product [25B(*Sa*,*R*)]: $[\alpha]_D$ + 4.67 (*c* 0.3, $CHCl_3$); ^1H NMR (400 MHz, $CDCl_3$): δ 7.31 (s, 1 H), 6.98 (s, 1 H), 6.06 (s, 1 H), 6.05 (s, 1 H), 5.16 (s, 1 H), 4.46 (d, *J* = 12.8 Hz, 1 H), 4.33 (d, *J* = 12.8 Hz, 1 H), 4.04 (s, 3 H), 4.02 (s, 3 H), 3.94 (s, 3 H), 3.67 (s, 3 H), 1.31-0.99 (m, 21 H), 0.23 (s, 9 H).; ^{13}C NMR (125 MHz, $CDCl_3$) δ 153.46, 151.03, 149.32, 142.45, 141.18, 136.52, 136.06, 134.33, 121.59, 120.56, 107.36, 105.61, 103.85, 101.33, 90.52, 63.91, 62.47, 61.11, 60.93, 59.75, 53.63, 31.13, 29.92, 14.33, 12.08, 0.13; MS (ESI): m/z: 653.2505 $[M+Na]^+$.



24C (S_aS) R_3 = TBS

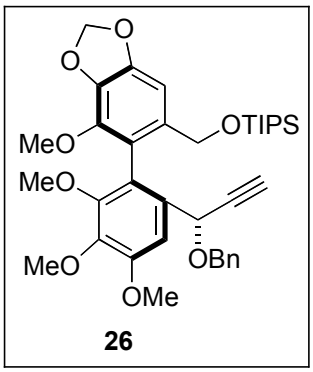
Synthesis of compound 24C: Following the similar procedure, **24C(*Sa*,*S*)** (major product, 90% yield) and **25C(*Sa*,*R*)** (minor product, 6% yield) were synthesized from **23C**. **Major product 24C(*Sa*,*S*)** : $[\alpha]_D$ -23.4 (*c* 1.07, $CHCl_3$).; ^1H NMR (400 MHz, $CDCl_3$): δ 7.28 (s, 1H), 6.88 (s, 1H), 5.99 (d, *J* = 1.2 Hz, 1H), 5.97 (d, *J* = 1.2 Hz, 1H), 5.03 (d, *J* = 1.6 Hz, 1H), 4.29 (d, *J* = 14 Hz, 1H), 4.09 (d, *J* = 14 Hz, 1H), 3.95 (s, 3H), 3.89 (s, 3H), 3.84 (s, 3H), 3.63 (s, 3H), 2.87 (d, *J* = 1.6 Hz, 1H), 0.87 (s, 9H), 0.16 (s, 9H), -0.03 (s, 3H), -0.04 (s, 3H).; ^{13}C NMR (100 MHz, $CDCl_3$) δ 153.37, 151.10, 149.25, 142.25, 140.25, 135.66, 135.49, 135.14, 120.79, 117.82, 106.61, 104.84, 101.97, 90.84, 62.87, 62.66, 60.84, 60.73, 59.77, 55.79, 53.41, 25.98, 18.35, -0.16, -5.37, -5.41. IR (neat, cm^{-1}): 3428, 2929, 2884, 1638, 1320, 1146, 1076.; HRMS (ESI) $[M+Na^+]$ calcd for $C_{30}H_{44}O_8Si_2Na$ 611.2467, found 611.2463.



25C (S_aR) R_3 = TBS

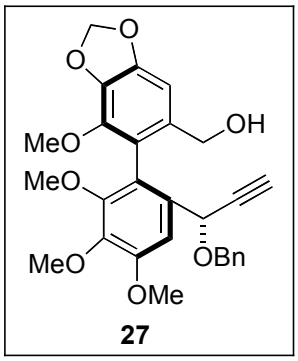
Minor product 25C(*Sa*,*R*): $[\alpha]_D$ -5.6 (*c* 0.9, $CHCl_3$).; ^1H NMR (400 MHz, $CDCl_3$) δ 7.26 (s, 1H), 6.66 (s, 1H), 6.00 (d, *J* = 1.2 Hz, 1H), 5.98 (d, *J* = 1.2 Hz, 1H), 5.08 (d, *J* = 1.6 Hz, 1H), 4.25 (d, *J* = 11.6 Hz, 1H), 4.15 (d, *J* = 11.6 Hz, 1H), 3.95 (s, 3H), 3.87 (s, 3H), 3.82 (s, 3H), 3.65 (d, *J* = 1.6 Hz, 1H), 3.62 (s, 3H), 2.87 (d, *J* = 1.6 Hz, 1H), 0.80 (s, 9H), 0.15 (s, 9H), 0.02 (s, 3H), -0.01 (s, 3H).; ^{13}C NMR (100 MHz, $CDCl_3$) δ 153.26, 150.76, 149.08, 142.11, 141.07, 136.49,136.08, 133.67, 121.32, 120.78, 107.01,105.45, 103.89, 101.17, 90.21, 63.90, 62.08, 60.88, 60.69, 59.57, 55.84, 29.70,26.04, 18.57,-0.09,-5.17, -5.43.; IR (neat, cm^{-1}):

3388, 2940, 2876, 1148, 776.; HRMS (ESI) [M+Na$^+$] calcd for $C_{30}H_{44}O_8Si_2Na$ 611.2467, found 611.2462.



26

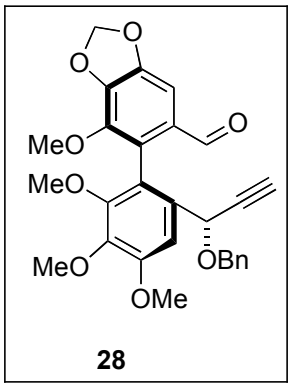
Synthesis of compound 26: To a solution of the substrate **24B(*S*$_a$*R*)** (945 mg, 1.5 mmol) in DME (6 mL) at 0 °C was added benzyl bromide (0.72 mL, 6.0 mmol) followed by tetra-*n*-butylammonium iodide (10 mg). After stirring at 0 °C for 20 min, to the above solution was added NaH (50% in paraffin oil, 145 mg, 3.0 mmol). The reaction mixture was stirred at 0 °C for 30 min and quenched with saturated aq NH_4Cl solution. The reaction mixture was diluted with diethyl ether and washed successively with water, brine and dried over Na_2SO_4. The organic layer was evaporated under reduced pressure to afford the crude product which was purified by column chromatography on silica gel using 15 % EtOAc/hexane as the eluent to get the benzyl ether **26** (875 mg, 1.35 mmol) in 90% yield as viscous oil. $[\alpha]_D$ – 20.6 (*c* 0.9, $CHCl_3$).; ^{1}H NMR (500 MHz, $CDCl_3$): δ 7.26-7.15 (m, 6 H), 6.89 (s, 1 H), 5.94 (d, *J* = 1.5 Hz, 1 H), 5.93 (d, *J* = 1.5 Hz, 1 H), 4.53 (d, *J* = 2.5 Hz, 1 H), 4.56 (d, *J* = 11.5 Hz, 1 H), 4.32-4.18 (m, 3 H), 3.91 (s, 3 H), 3.85 (s, 3 H), 3.75 (s, 3 H), 3.60 (s, 3 H), 2.49 (d, *J* = 2.5 Hz, 1 H), 1.08-0.93 (m, 21 H).; ^{13}C NMR (100 MHz, $CDCl_3$) δ 153.49, 151.23, 149.28, 142.48, 140.88, 137.70, 135.78, 134.62, 133.41, 128.38, 127.74, 122.01, 117.44, 106.74, 100.92, 100.77, 82.51, 75.24, 70.83, 67.56, 62.87, 60.98, 60.84, 59.37, 56.16, 18.24, 18.22, 14.40, 12.12.; MS (ESI): m/z: 671.2866 [M+Na]$^+$.



27

Synthesis of the compound (27): To a stirred solution of compound **26** (648 mg, 1.0 mmol) in THF (4.0 mL) was added TBAF (1 M in THF, 1.5 mL, 1.5 mmol) at 0 ^{o}C and stirring was continued at the same temperature for 2 h. The solvent was evaporated under reduced pressure to afford the crude product, which was purified by silica gel chromatography using 30 % EtOAc/hexane as the eluent to yield the compound **27**: (457 mg, 0.93 mmol) in 93% yield as a viscous oil. $[\alpha]_D$ – 33.5 (*c* 1, $CHCl_3$).; ^{1}H NMR (500 MHz, $CDCl_3$): δ 7.29-7.23 (m, 5 H), 7.15 (s, 1 H), 6.71 (s, 1 H), 5.95 (s, 2 H), 4.73 (d, *J* = 2.0 Hz, 1 H), 4.63 (d, *J* = 11.6 Hz, 1 H), 4.45 (d, *J* = 11.6 Hz,

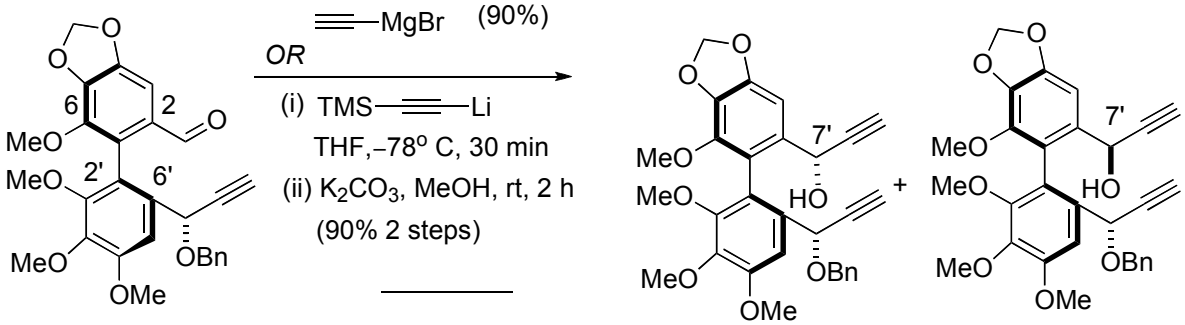
1 H), 4.10-4.08 (bs, 2 H), 3.90 (s, 3 H), 3.87 (s, 3 H), 3.75 (s, 3 H), 3.61 (s, 3 H), 2.47 (d, J = 2.5 Hz, 1 H).; ^{13}C NMR (125 MHz, $CDCl_3$) δ 153.48, 150.92, 149.26, 142.13, 140.54, 137.40, 135.26, 135.06, 133.57, 128.20, 128.05, 127.61, 121.61, 119.04, 106.05, 103.26, 100.93, 81.96, 74.94, 70.63, 67.32, 63.61, 60.92, 60.89, 59.10, 55.90; MS (ESI): m/z: 551.1663 $[M+Na]^+$.



28

Synthesis of compound (28): To a solution of the substrate **27** (240 mg, 0.5 mmol) in CH_2Cl_2 (4 mL) at 0 °C was added Dess-Martin periodinane (318 mg, 0.75 mmol). After stirring at rt for 4 h, the reaction mixture was quenched with aq. sat. $NaHCO_3$ solution. The reaction mixture was diluted with CH_2Cl_2 and washed successively with water, brine and dried over Na_2SO_4. The organic layer was evaporated under reduced pressure to afford the crude product which was purified by column chromatography on silica gel using 15 % EtOAc/hexane as the eluent to get the aldehyde **28** in 90% yield (214 mg, 0.45 mmol) as viscous oil. $[\alpha]_D$ – 11.0 (*c* 1, $CHCl_3$).; ^{1}H NMR (500 MHz, $CDCl_3$): δ 9.39 (s, 1 H), 7.28-7.22 (m, 6 H), 7.15 (s, 1 H), 6.05 (s, 2 H), 4.70 (d, J = 1.6 Hz, 1 H), 4.60 (d, J = 12.0 Hz, 1 H), 4.40 (d, J = 12.0 Hz, 1 H), 3.92 (s, 3 H), 3.87 (s, 3 H), 3.73 (s, 3 H), 3.63 (s, 3 H), 2.49 (d, J = 1.6 Hz, 1 H).; ^{13}C NMR (100 MHz, $CDCl_3$) δ 190.49, 154.32, 151.84, 149.81, 142.15, 141.73, 140.90, 137.37, 133.81, 130.35, 128,43, 128.38, 127.89, 118.84, 105.93, 102.16, 100.77, 81.31, 76.19, 70.81, 67.57, 61.04, 60.87, 59.70, 56.19.; MS (ESI): m/z: 513.1468 $[M+Na]^+$.

Synthesis of compounds (29) and (30):



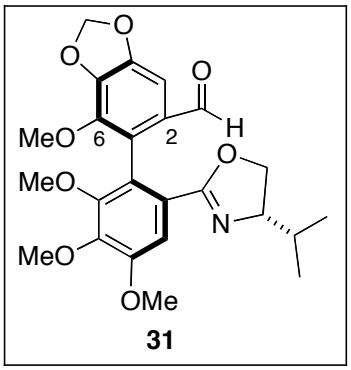
28 (90%) **29** 7'OH (*S*) **30** 7'OH (*R*)

Addition of Ethynyl Magnesium Bromide. To a stirred solution of compound **(28)** (245 mg, 0.5 mmol) in dry THF (4 mL) at 0 °C was added ethynyl magnesium bromide (0.5 M in THF, 2.0 mL, 1.0 mmol). After stirring at 0 °C for 30 min, the reaction was quenched by the addition of aq. sat. NH_4Cl solution. The reaction mixture was diluted with CH_2Cl_2 and washed with water, brine and dried over Na_2SO_4. The solvent was evaporated under reduced pressure to afford the crude product, which was purified by silica gel column chromatography. Elution with 15% EtOAc/hexane provided major product **(29)** (139mg, 0.27 mmol) and minor product (**30**): (92 mg, 0.18 mmol) in 90% combined yield with 3:2 diastereomeric ratio.

Addition of Lithium Trimethylsilylacetylide. To a solution of 200 mg (0.408 mmol) of compound **(28)** in 10 mL THF at -78 ºC was added a solution of 2.4 mL(0.5 M, 1.2 mmol) of LiC≡CTMS in THF dropwise. After stirring for 30 min at this temperature, the solution was allowed to warm to room temperature before it was quenched with saturated aq. NH_4Cl solution. After ether extraction, drying with anhydrous $MgSO_4$, filtration and removal of the solvent under vacuum, the crude product was obtained as a yellow oil, which was then dissolved in MeOH (10 mL). To this solution was added K_2CO_3 (57 mg, 0.408 mmol) and after stirring for 2 hours at room temperature, the solution was quenched with saturated aq. NH_4Cl solution. After removal of the solvent under vaccum, ether was used for extraction. The combined organic phase was dred over anhydrous Na_2SO_4, The solvent was evaporated under reduced pressure to afford the crude product, which was purified by silica gel column chromatography. Elution with 15% EtOAc/hexane provided major product **(29)** (160 mg, 0.24 mmol) and minor product (**30**): (80 mg, 0.12 mmol) in 90 % combined yield with 2:1 diastereomeric ratio.

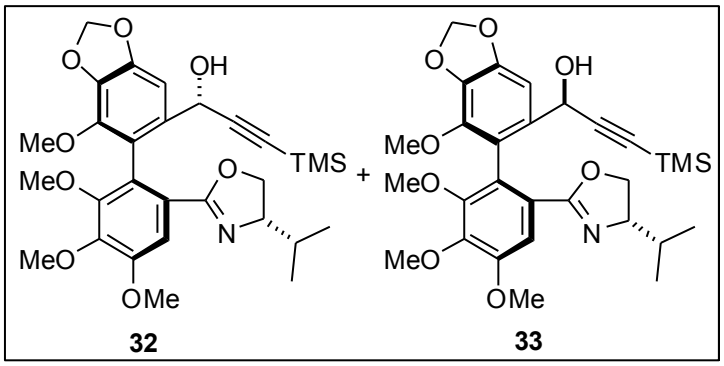
Compound (**29**): $[\alpha]_D$ – 28.6 (*c* 1, $CHCl_3$).; ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.30-7.23 (m, 5 H), 7.17 (s, 1 H), 6.13 (s, 1 H), 5.97 (s, 2 H), 4.92-4.86 (m, 1 H), 4.77 (d, *J* = 2.0 Hz, 1 H), 4.67 (d, *J* = 12.0 Hz, 1 H), 4.49 (d, *J* = 12.0 Hz, 1 H), 3.91 (s, 3 H), 3.86 (s, 3 H), 3.72 (s, 3 H), 3.60 (s, 3 H), 326-3.18 (bs, 1 H), 2.47 (d, *J* = 2.0 Hz, 1 H), 2.46 (d, *J* = 2.4 Hz, 1 H).; ^{13}C NMR (100 MHz, $CDCl_3$) δ 153.97, 150.93, 149.90, 142.28, 140.29, 137.69, 136.10, 135.27, 134.55, 128.52, 128.44, 128.26, 127.93, 120.74, 119.15, 106.06, 102.50, 101.44, 83.53, 81.51, 77.43, 75.60, 73.87, 70.95, 67.59, 63.12, 61.51, 61.31, 59.39, 56.23, 53.62.; MS (ESI): m/z: 539.1608 $[M+Na]^+$.

Compound (30): $[\alpha]_D$ +14.8 (*c* 0.5, $CHCl_3$).; ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.27-7.21 (m, 5 H), 7.11 (s, 1 H), 6.98 (s, 1 H), 5.97 (d, *J* = 1.2 Hz, 1 H), 5.96 (d, *J* = 1.2 Hz, 1 H), 5.0-4.92 (m, 1 H), 4.79 (d, *J* = 2.4 Hz, 1 H), 4.56 (d, *J* = 11.6 Hz, 1 H), 4.38 (d, *J* = 11.6 Hz, 1 H), 3.91 (s, 3 H), 3.87 (s, 3 H), 3.74 (s, 3 H), 3.65 (s, 3 H), 2.59 (d, *J* = 2.4 Hz, 1 H), 2.45 (d, *J* = 2.4 Hz, 1 H), 2.42-2.33 (bs, 1 H).; ^{13}C NMR (100 MHz, $CDCl_3$): δ 153.97, 151.89, 149.78, 142.56, 140.90, 137.46, 136.58, 134.55, 133.34, 128.48, 128.44, 127.90, 121.50, 120.09, 106.79, 102.02, 101.45, 83.75, 82.99, 77.43, 75.87, 74.11, 70.93, 67.82, 61.90, 61.10, 60.99, 59.56, 56.18.; MS (ESI): m/z: 539.1608 $[M+Na]^+$.



31

Synthesis of compound 31: To a solution of biaryl oxazoline **22C** (***Sa,S***) (250 mg, 0.436 mmol) in THF (10 mL) at room temperature was added 1 M TBAF solution (0.52 mL, 0.52 mmol) dropwise. After stirring for 2 h the reaction was quenched by the addition of saturated aq. NH_4Cl solution. The reaction mixture was diluted with CH_2Cl_2 and washed with water, brine and dried over anhydrous $MgSO_4$. The solvent was evaporated under reduced pressure to afford an yellow oil which was then dissolved in CH_2Cl_2 (10 mL). To this solution was added pyridinium chlorochromate (187 mg, 0.87 mmol), p-TsOH·H_2O (165 mg, 0.87 mmol) and Celite (210 mg). After stirring for 18 h, Et_3N (1 mL) was added. The resulting brown suspension was directly loaded into column (EtOAc : hexanes = 1: 2), which affored **31** as a light yellow oil (169 mg, 85% from **22C**). $[\alpha]_D$ -50.4 (*c* 0.635, $CHCl_3$).; ^{1}H NMR (400 MHz, $CDCl_3$): δ 9.45 (s, 1 H), 7.24 (s, 1 H), 7.17 (s, 1 H), 6.05 (d, *J* = 1.6 Hz, 1 H), 6.04 (d, *J* = 1.6 Hz, 1 H), 6.12-6.08 (m, 1 H), 3.93 (s, 3 H), 3.90 (s, 3 H), 3.84-3.77 (m, 4 H), 3.71 (t, *J* = 8 Hz, 1 H), 3.62 (s, 3 H), 1.57-1.52 (m, 1 H), 0.77 (d, *J* = 6.8 Hz, 1 H), 0.73 (d, *J* = 6.8 Hz, 1 H).; ^{13}C NMR (100 MHz, $CDCl_3$) δ 190.30, 162.69, 153.19, 152.11, 149.11, 144.11, 141.66, 141.03, 130.60, 129.99, 124.66, 120.56, 108.56, 101.87, 100.37, 72.74, 70.26, 60.93, 60.69, 59.70, 56.10, 32.78, 18.57, 18.28.; IR (neat, cm^{-1}): 2956, 1682, 1607, 1470, 1392, 1285, 1106.; HRMS (ESI) $[M+H]^+$ calcd for $C_{24}H_{28}NO_8$ 458.1809, found 458.1805.

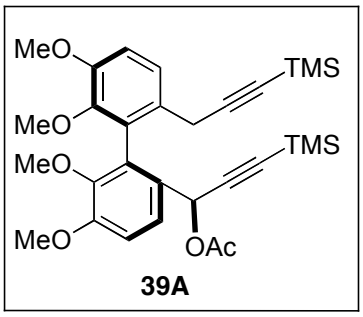


Synthesis of compounds 32 and 33. To a solution of 200 mg (0.438 mmol) of biaryl aldehyde **31** and 203 mg (1.75 mmol, 0.26 mL) of TMEDA in 10 ml THF at -78ºC was added a solution of 2.6 mL(0.5 M, 1.3 mmol) of LiC≡CTMS in THF dropwise. After stirring for 30 min at this temperature, the solution was allowed to warm to room temperature before it was quenched with saturated aq. NH_4Cl solution. After ether extraction, drying with anhydrous $MgSO_4$, filtration and removal of the solvent under vacuum, the crude product was obtained as a yellow oil. Flash column (EtOAc : hexanes = 1: 2) gave **32** as a white foam (183 mg, 75% yield) and **33** as a colorless oil (27 mg, 11%).

Compound **32**: $[\alpha]_D$ 72.5 (*c* 1.03, $CHCl_3$).; ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.16 (s, 1 H), 6.99 (s, 1 H), 6.74 (s, 1 H), 5.96 (d, *J* = 1.2 Hz, 1 H), 5.90 (d, *J* = 1.2 Hz, 1 H), 5.13 (s, 1 H), 4.27 (t, *J* = 8.8 Hz, 1 H), 3.99-3.95 (m, 1 H), 3.93 (s, 3 H), 3.89 (s, 3 H), 3.70 (s, 3 H), 3.66 (s, 3 H), 1.38-1.35 (m, 1 H), 0.64 (d, *J* = 6.8 Hz, 3 H), 0.53 (d, *J* = 6.8 Hz, 3 H). 0.14 (s, 9 H).; ^{13}C NMR (100 MHz, $CDCl_3$): δ 163.73, 152.91, 152.20, 149.08, 144.61, 139.99, 136.59, 135.53, 123.66, 123.53, 121.61, 108.23, 105.68, 102.55, 101.09, 89.45, 72.04, 70.32, 62.89, 60.90, 60.78, 59.59,56.06, 32.75, 18.38, 17.30, -0.10.; IR (neat, cm^{-1}): 3186, 2959, 2899, 1651, 1590,1470, 1366, 1274, 1109,1058, 981, 846.; HRMS (ESI) [M+H$^+$] calcd for $C_{29}H_{38}NO_8Si$ 556.2361, found 556.2363.

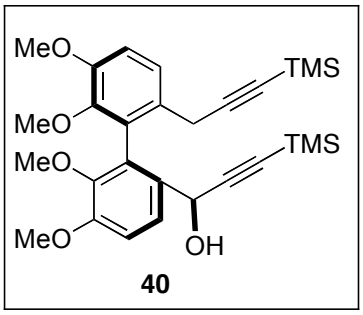
A sample was recrystallized from ethyl acetate/hexane and the solid state structure was determined by X-ray crystallography. Crystallographic Information File is attached to this Supporting Information.

Compound **33**: $[\alpha]_D$ -33.2 (*c* 1.0, $CHCl_3$).; ^{1}H NMR (400 MHz, $CDCl_3$): 7.19 (s, 1H), 7.04 (s, 1H), 5.96 (d, *J* = 1.2 Hz, 1H), 5.95 (d, *J* = 1.2 Hz, 1H), 5.08 (s, 1H), 4.14 (t, *J* = 8Hz, 1H), 3.94-3.89 (s superimposed om a m, 7H), 3.88 (s, 3H), 3.78 (s, 3H), 3.63 (s, 3H), 1.56-1.50 (m, 1H), 0.76 (d, *J* = 0.8 Hz, 3H), 0.74 (d, *J* = 0.8 Hz, 3H), 0.12 (s, 9H).; ^{13}C NMR (100 MHz, $CDCl_3$): δ 163.39, 153.83, 151.55,148.79, 144.29, 140.93, 136.24, 134.09, 124.42, 123.30, 121.77, 108.97, 104.97, 102.61,101.08, 90.39, 72.34, 70.33, 63.83, 61.05, 60.98, 59.48, 56.02, 32.77, 18.37, 18.34, -0.14. IR (neat, cm^{-1}): 2958, 1475, 1402, 1366, 1108, 845, 756.; HRMS (ESI) [M+H]$^+$ calcd for $C_{29}H_{38}NO_8Si$ 556.2361, found 556.2365.



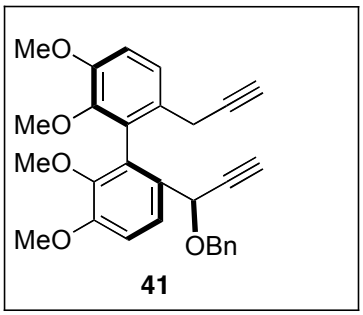
39A

Synthesis of compound 39A: To the alcohol **7A** (510 mg, 1.0 mmol) in THF (4 mL) at 0 °C added acetic acid (0.09 mL, 1.5 mmol), triphenylphosphine (393 mg, 1.5 mmol) followed by diethylazodicarboxylate (0.24 mL, 1.5 mmol), and the mixture was gradually brought to room temperature over 12 h. The solvent was evaporated under reduced pressure to afford the crude product which was purified by column chromatography on silica gel using 20 % EtOAc/hexane as the eluent to yield the major product **39A** $C_6(R)$ (441 mg, 0.8 mmol) in 80% yield and minor product **39B** $C_6(S)$ (83 mg, 0.07 mmol) in 7 % yield as a viscous oil. **Major product 39A** $C_6(R)$: ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.65 (d, J = 8.0 Hz, 1 H), 7.35 (d, J = 8.0 Hz, 1 H), 6.98 (d, J = 8.0 Hz, 1 H), 6.95 (d, J = 8.0 Hz, 1 H), 5.99 (s, 1 H), 3.90 (s, 3 H), 3.87 (s, 3 H), 3.67 (s, 3 H), 3.59 (s, 3 H), 3.23 (d, J = 20.0 Hz, 1 H), 2.98 (d, J = 20.0 Hz, 1 H), 1.84 (s, 3 H), 0.12 (s, 9 H), 0.09 (s, 9 H). **Minor product 39B** $C_6(R)$: ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.55 (d, J = 8.0 Hz, 1 H), 7.37 (d, J = 8.0 Hz, 1 H), 7.0 (d, J = 8.0 Hz, 1 H), 6.95 (d, J = 8.0 Hz, 1 H), 5.81 (s, 1 H), 3.89 (s, 3 H), 3.85 (s, 3 H), 3.62 (s, 3 H), 3.59 (s, 3 H), 3.24 (d, J = 20.0 Hz, 1 H), 3.11 (d, J = 20.0 Hz, 1 H), 1.84 (s, 3 H), 0.13 (s, 18 H).



40

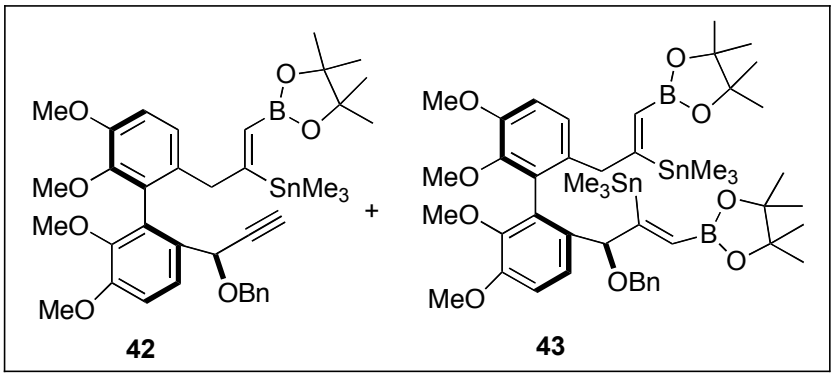
Synthesis of compound 40: To a solution of acetate **39A** (414 mg, 0.75 mmol) in CH_2Cl_2 (3 mL) at –50 °C was added DIBAL-H (1.0 M in hexane, 0.9 mL, 0.9 mmol) dropwise. After stirring at the same temperature for 30 min, the reaction was quenched by the addition of methanol (0.05 mL) followed by 1 N HCl solution (0.5 mL). The aqueous layer was extracted with Et_2O and the organic layer washed with water, brine and dried over Na_2SO_4. The solvent was evaporated under reduced pressure to afford the crude product, which was purified by silica gel chromatography using 25% EtOAc/hexane as the eluent to yield compound **40** (363mg, 0.71mmol) in 95% yield as viscous oil. ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.49 (d, J = 8.0 Hz, 1 H), 7.33 (d, J = 8.0 Hz, 1 H), 7.0 (d, J = 8.0 Hz, 1 H), 6.96 (d, J = 8.0 Hz, 1 H), 5.05 (d, J = 0.4 Hz, 1 H), 3.89 (s, 3 H), 3.88 (s, 3 H), 3.64 (s, 3 H), 3.60 (s, 3 H), 3.27 (d, J = 20.0 Hz, 1 H), 3.18 (d, J = 20.0 Hz, 1 H), 2.75 (d, J = 0.4 Hz, 1 H), 0.1 (s, 9 H), 0.08 (s, 9 H).



41

Synthesis of compound 41: To a solution of the substrate **40** (102 mg, 0.2 mmol) in DME (1 mL) at 0 °C was added benzyl bromide (0.1 mL, 0.8 mmol) followed by tetra-*n*-butylammonium iodide (2 mg). After stirring at 0 °C for 20 min, to the above solution was added NaH (50% in paraffin oil, 30 mg, 0.4 mmol). The reaction mixture was stirred at 0 °C for 30 min and quenched with saturated aq. NH_4Cl solution. The reaction mixture was diluted with diethyl ether and washed successively with water, brine and dried over Na_2SO_4. The organic layer was evaporated under reduced pressure to afford the crude product which was purified by column chromatography on silica gel using 10 % EtOAc/hexane as the eluent to get the benzyl ether **41** (80 mg, 0.17 mmol) in 88% yield as viscous oil. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.63 (d, *J* = 8.0 Hz, 1 H), 7.40 (d, *J* = 8.0 Hz, 1 H), 7.23-7.13 (m, 5 H), 6.98 (d, *J* = 8.0 Hz, 1 H), 6.93 (d, *J* = 8.0 Hz, 1 H), 4.70 (d, *J* = 0.4 Hz, 1 H), 4.55 (d, J = 16.0 Hz, 1 H), 4.10 (d, J = 16.0 Hz, 1 H), 3.87 (s, 6 H), 3.60 (s, 6 H), 3.15-3.13 (m, 2 H), 2.58 (d, *J* = 0.4 Hz, 1 H), 2.07 (t, *J* = 0.4 Hz, 1 H).

Borostannylation of diyne 41: Synthesis of compounds 42 and 43

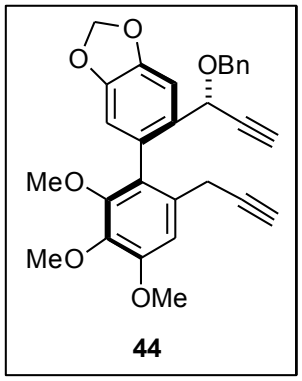


42 **43**

To a solution of 1,3-dimethyl-2-(trimethylstannyl)-2-bora-1,3-diazocyclopentane **3** (60 mg, 0.24 mmol) in benzene (1.5 mL) added $PdCl_2.(PPh_3)_2$ (6 mg, 0.008 mmol) and the mixture was stirred at room temperature for 10 min followed by addition of a solution of diyne **41** (91 mg, 0.2 mmol) in benzene (0.5 mL). After stirring the reaction mixture at room temperature for 6 h., a solution of pinacol (32 mg, 0.26 mmol) in benzene (0.3 mL) followed by *p*-toluenesulfonic acid (*p*-TSA, 54 mg, 0.26 mmol) were added to the reaction mixture. After 6 h, the reaction was quenched by the addition of Et_3N (0.04 mL) and the solvent was evaporated under reduced pressure to afford the crude product, which was purified by silica gel chromatography using 10 % EtOAc/hexane as the eluent to yield compounds **42** (44 mg, 0.06 mmol) and **43** (20 mg, 0.02 mmol) as a viscous

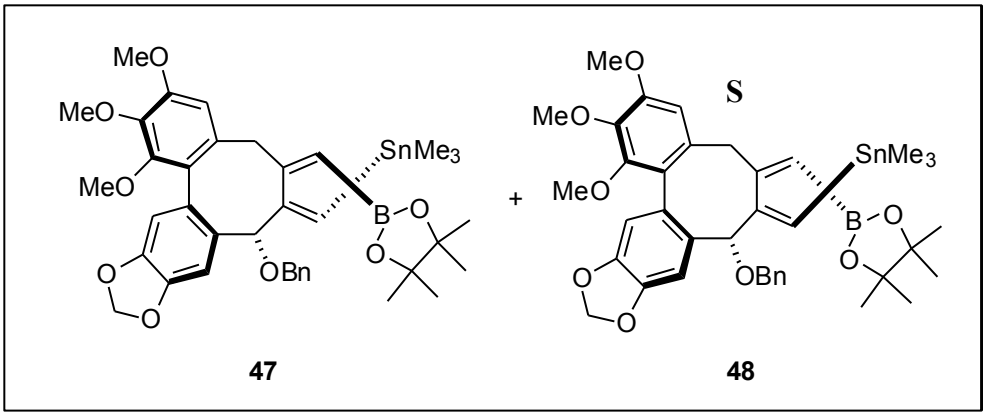
oil. Compound **42**: ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.58 (d, *J* = 8.0 Hz, 1 H), 7.21-7.16 (m, 5 H), 6.97 (d, *J* = 8.0 Hz, 1 H), 6.83 (d, *J* = 8.0 Hz, 1 H), 6.71 (d, *J* = 8.0 Hz, 1 H), 5.89 (s, 1 H), 4.67 (d, *J* = 0.4 Hz, 1 H), 4.59 (d, *J* = 16.0 Hz, 1 H), 4.19 (d, *J* = 16.0 Hz, 1 H), 3.86 (s, 3 H), 3.81 (s, 3 H), 3.61 (s, 3 H), 3.58 (s, 3 H), 3.35 (d, *J* = 18 Hz, 1 H), 3.17 (d, *J* = 18.0 Hz, 1 H), 2.51 (d, *J* = 0.4 Hz, 1 H), 1.13 (s, 12 H), -0.02 (t, *J* = 32.0 Hz, 9 H).

Compound 43: ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.22-7.14 (m, 5 H), 6.88 (d, *J* = 8.0 Hz, 1 H), 6.79 (d, *J* = 8.0 Hz, 1 H), 6.67 (d, *J* = 8.0 Hz, 1 H), 5.91 (s, 1 H), 5.77 (s, 1 H), 4.70 (s, 1 H), 4.21 (d, *J* = 12.0 Hz, 1 H), 4.02 (d, *J* = 12.0 Hz, 1 H), 3.86 (s, 3 H), 3.79 (s, 3 H), 3.61 (s, 6 H), 3.48-3.14 (m, 2 H), 1.24-1.13 (m, 24 H), 0.08-0.01 (m, 18 H).



44

Synthesis of compound 44: To a solution of the alcohol **21A** (903 mg, 1.72 mmol) and BnBr (1.08 g, 6.88 mmol) in DME (10 mL) was added NaH (275 mg, 6.88 mmol, 60% w/w in paraffin oil) at 0ºC. One drop of water was added to the above mixture. The mixture was stirred at 0ºC for 3.5 h, then quenched with saturated aq. NH_4Cl solution. Ether extraction, drying with anhydrous $MgSO_4$ and removal of the solvent under vacuum gave an oil, which was directly used without further purification. The crude oil was dissolved in MeOH (100 mL) followed by the addition of solid K_2CO_3 (950 mg, 6.88 mmol) at 0ºC while stirring. Then the mixture was allowed to warm to room temperature and further stirring for 6h. The reaction was quenched with saturated aq. NH_4Cl solution. Ether extraction, drying with anhydrous $MgSO_4$ and removal of the solvent under vacuum gave an oil. Purification by flash column (EtOAc: hexanes = 1: 15) gave the diyne **44** as a yellow solid (584 mg, 72% yield over two steps). ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.32 (s, 1 H), 7.29-7.20 (m, 5 H), 6.99 (s, 1 H), 6.58 (s, 1 H), 6.02 (s, 2 H), 4.76 (d, *J* = 2 Hz, 1 H), 4.58 (d, *J* = 11.6 Hz, 1 H), 4.20 (d, *J* = 11.6 Hz, 1 H), 3.93 (s, 3 H), 3.80 (s, 3 H), 3.59 (s, 3 H), 3.22 (dd, *J* = 4Hz, 2.8Hz, 2 H), 2.54 (d, *J* = 2 Hz, 1 H), 2.14 (t, *J* = 2.8 Hz, 1 H).; ^{13}C NMR (100 MHz, $CDCl_3$): δ 153.27, 150.93, 147.77, 147.55, 140.84, 137.62, 131.72, 130.86, 128.47, 128.23, 128.16, 128.05, 127.94, 127.55, 125.09, 109.92, 107.52, 107.35, 101.38, 82.30, 82.12, 75.04, 70.70, 70.57, 67.56, 60.86, 60.82, 55.97, 22.95.; IR (neat, cm^{-1}): 3435, 3287, 2955, 1597, 1480, 1462, 1402, 1234, 1040, 749, 700.; HRMS (ESI) [$M+Na^+$] calcd for $C_{29}H_{26}O_6Na$ 493.1622, found 493.1589.



47 **48**

Synthesis of compounds 47 and 48: To a solution of diyne **44** (40 mg, 0.084 mmol) and $PdCl_2(PPh_3)_2$ (3 mg, 0.002 mmol) in benzene (0.6 mL) was added 1,3-dimethyl-2-(trimethylstannyl)-2-bora-1,3-diazocyclopentane (26 mg, 0.1 mmol) in benzene (0.6 mL) and the mixture was stirred at room temperature for 18 h. Then pinacol (15 mg, 0.126 mmol) was added and the mixture was stirred after for 6 h. The solvent was evaporated under reduced pressure to afford the crude product, which was purified by flash column (EtOAc: hexanes = 1: 5) to yield compound **47** (34 mg, 55% yield) and compound **48** (14 mg, 23% yield) as light yellow waxy solids.

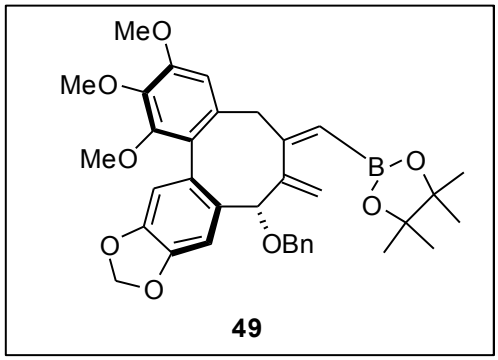
Compound **47**: ^{1}H NMR (400 MHz, C_6D_6) δ 7.70 (s, 1H), 7.43 (d, J = 7.2 Hz, 2H), 7.12 (d, J = 7.2 Hz, 2H), 7.05 (t, J = 7.2 Hz, 1H), 6.84 (s, 1H), 6.83 (s, 1H), 6.51 (S, 1H), 5.70 (s, 1H), 5.29 (d, J = 1.6 Hz, 1H), 5.22 (d, J = 1.6 Hz, 1H), 5.01 (d, J = 12 Hz, 1H), 4.99 (s, 1H), 4.53 (d, J = 12 Hz, 1H), 3.75 (s, 3H), 3.42 (s, 3H), 3.34 (s, 3H), 3.24 (d, J = 11.6 Hz, 1H), 3.16 (d, J = 11.6 Hz, 1H), 1.04 (s, 6H), 1.01 (s, 6H), 0.24 (s, 9H). ^{13}C NMR (100 MHz, C_6D_6) δ 164.62, 160.87, 153.96, 151.60, 148.23, 146.39, 141.96, 139.50, 137.35, 135.73, 128.28, 127.80, 127.68, 127.25, 125.90,123.94, 110..32, 107.90, 104.97, 100.79, 82.84, 79.76, 70.79, 60.47, 60.32, 55.26, 53.09, 45.99, 29.96, 24.92, 24.86, -7.70. IR (neat, cm^{-1}): 2925, 1595, 1480, 1379, 1329, 1271, 1260, 1142, 764, 750. HRMS (ESI) [M+Na^+] calcd for $C_{38}H_{47}BSnO_8Na$ 785.2292, found 785.2291.

A sample, recrystallized from CH_2Cl_2 and ethanol with slow vapor diffusion of hexane was analyzed by X-ray crystallography. Crystallographic Information File is attached to this Supporting Information.

Compound **48**: ^{1}H NMR (400 MHz, C_6D_6) δ 7.52 (s, 1H), 7.37 (d, J = 7.2 Hz, 2H), 7.12-7.10 (d, J = 7.2 Hz, 2H), 7.06-7.04 (), 7.05 (t, J = 7.2 Hz, 1H), 6.97 (s, 1H), 6.51 (s, 1H), 6.47 (s, 1H),

6.15 (d, *J* = 0.8 Hz, 1H), 5.40 (d, *J* = 1.2 Hz, 1H), 5.36 (d, *J* = 1.2 Hz, 1H), 5.31 (s, 1H), 4.73 (d, *J* = 12 Hz, 1H), 4.62 (d, *J* = 12 Hz, 1H), 3.86 (s, 3H), 3.63 (d, *J* = 13.2 Hz, 1H), 3.51 (s, 3H), 3.49 (d, *J* = 13.2 Hz, 1H), 3.41 (s, 3H), 1.04 (s, 6H), 1.03 (s, 6H), 0.35 (s, 9H). ^{13}C NMR (100 MHz, C_6D_6) δ 176.34, 174.86, 153.32, 151.38, 147.58, 146.51, 141.45, 139.46, 134.87, 133.79, 130.67, 128.18, 127.92, 127.68, 127.65, 127.09, 126.75, 110.56, 109.98, 107.93, 100.77, 85.89, 83.07, 82.97, 71.19, 60.65, 60.55, 55.34, 47.90, 29.96, 24.80, 24.68, -5.74. IR (neat, cm^{-1}): 2976, 2924, 2853, 1593, 1477, 1351, 1274, 1260, 1143, 1040, 764, 750. HRMS (ESI) [M+Na$^+$] calcd for $C_{38}H_{47}BSnO_8Na$ 785.2292, found 785.2309.

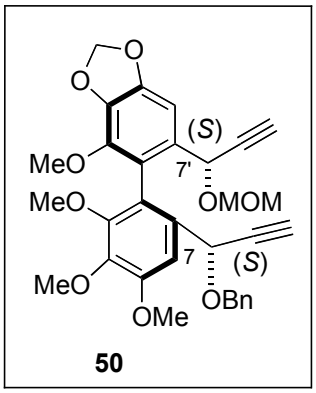
Synthesis of compound 49: To a mixture of cyclized boronesters **47** and **48** (50 mg, 0.068 mmol) in CH_2Cl_2 was added p-TsOH (26 mg, 0.137 mmol) and the mixture was stirred for 2 h. The reaction was quenched with Et_3N (14 mg, 0.137 mmol). Ether extraction, drying with anhydrous $MgSO_4$ and removal of the solvent under vacuum gave an oil. Purification by flash column (EtOAc: hexanes = 1: 5) gave the diene **49** as a yellow solid (33 mg, 81% yield). ^{1}H NMR (400 MHz, C_6D_6): δ 7.62 (s, 1 H), 7.13-7.09 (m, 3 H), 7.05 (t, *J* = 7.2 Hz, 1 H), 6.90 (s, 1 H), 6.50 (s, 1 H), 5.78 (s, 1 H), 5.66 (s, 1 H), 5.39 (d, *J* = 2 Hz, 1 H), 5.33 (d, *J* = 1.2 Hz, 2 H), 4.89 (s, 1 H), 4.65(d, *J* = 12 Hz, 1 H), 4.44 (d, *J* = 12 Hz, 1 H), 4.31 (s, 1 H), 3.81 (s, 3 H), 3.42 (s, 3 H), 3.27 (s, 3 H), 3.16 (s, 2 H), 1.06 (s, 12 H).; ^{13}C NMR (100 MHz, C_6D_6): δ 159.32, 153.15, 150.69, 150.60, 147.64, 146.05, 140.99, 138.80, 135.59, 135.01, 128.57, 128.12, 127.34, 127.26, 127.18, 125.48, 111.58, 109.46, 108.03, 105.12, 101.02, 83.08, 70.04, 61.09, 60.67, 56.05, 44.55, 29.70, 24.92, 24.54. IR (neat, cm^{-1}): 2922, 1612, 1478, 1406, 1328, 1260, 1235, 1195, 1039, 750.; HRMS (ESI) [M+Na$^+$] calcd for $C_{35}H_{39}BO_8Na$ 621.2636, found 621.2634.



49

Since both **47** and **48** gave the same product upon destannylation, these compounds were assigned the atropisomeric structures indicated.

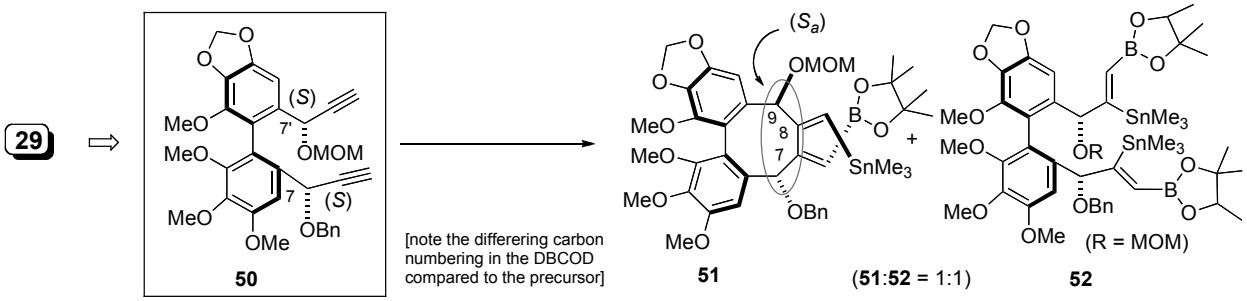
X-ray crystallographic analysis of a sample of **49** recrystallized from CH_2Cl_2 (slow vapor diffusion of hexane) confirmed the relative configuration of the chiral centers. CrystallographicInformation File is attached to this Supporting Information.



50

Synthesis of compound 50: To a solution of the compoud **29** (117 mg, 0.2 mmol) in DME (2mL) at 0 °C was added methoxymethylchloride (0.2 mL). After stirring at 0 °C for 20 min, to the above solution was added NaH (50% in paraffin oil, 37 mg, 0.5 mmol). The reaction mixture was stirred at 0 °C for 1 h and quenched with saturated aq. NH_4Cl solution. The reaction mixture was diluted with diethyl ether and washed successively with water, brine and dried over Na_2SO_4. The organic layer was evaporated under reduced pressure to afford the crude product which was purified by column chromatography on silica gel using 10 % EtOAc/hexane as the eluent to get the compound **50** (100 mg, 0.18 mmol) in 90 % yield as a viscous oil. $[\alpha]_D$ – 26.2 (*c* 1, $CHCl_3$).; ^{1}H NMR (500 MHz, $CDCl_3$): δ 7.22-7.16 (m, 6 H), 6.92 (s, 1 H), 5.97 (d, *J* = 1.6 Hz, 1 H), 5.94 (d, *J* = 1.6 Hz, 1 H), 4.85 (d, *J* = 2.0 Hz, 1 H), 4.83 (d, *J* = 2.0 Hz, 1H), 4.71 (d, *J* = 6.8 Hz, 1 H), 4.55 (d, *J* = 11.2 Hz, 1 H), 4.39 (d, *J* = 6.8 Hz, 1 H), 4.27 (d, *J* = 11.2 Hz, 1 H), 3.92 (s, 3 H), 3.83 (s, 3 H), 3.72 (s, 3 H), 3.69 (s, 3 H), 3.16 (s, 3 H), 2.60 (d, *J* = 2.0 Hz, 1 H), 2.50 (d, *J* = 2.0 Hz, 1H).: ^{13}C NMR (100 MHz, $CDCl_3$): δ 153.66, 151.86, 149.56, 142.44, 141.22, 137.55, 136.34, 133.03, 131.95, 128.63, 128.33, 127.76, 122.03, 120.95, 107.33, 102.17, 101.32, 94.27, 82.11, 82.07, 77.43, 76.26, 75.57, 70.92, 68.31, 64.82, 60.79, 60.77, 59.38, 56.13, 55.91.; MS (ESI): m/z: 583.1866 $[M+Na]^+$.

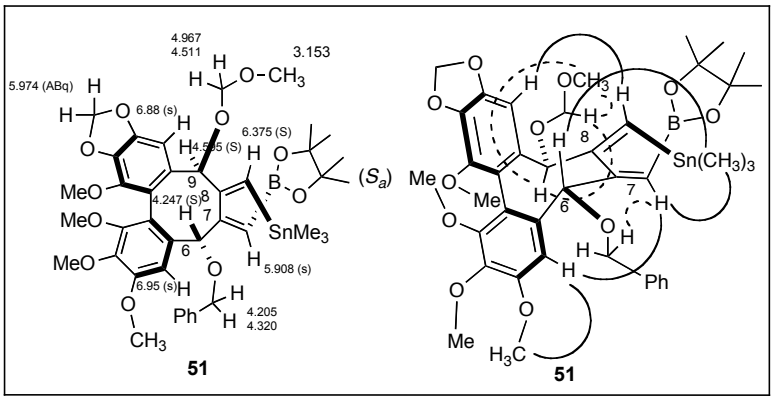
[BSn]-mediated Cyclization of diyne 50: **Synthesis of compounds 51 and 52**:



To a solution of 1,3-dimethyl-2-(trimethylstannyl)-2-bora-1,3-diazocyclopentane **3** (32 mg, 0.12 mmol) in benzene (0.8 mL) added $PdCl_2.(PPh_3)_2$ (1 mg) and the mixture was stirred at room temperature for 10 min and was followed by addition of a solution of diyne **50** (26 mg, 0.05 mmol) in benzene (0.3 mL). After stirring the reaction mixture at rt for 12 h, a solution of pinacol (16 mg, 0.12 mmol) in benzene (0.2 mL) followed by *p*-TSA (24 mg, 0.12 mmol) was

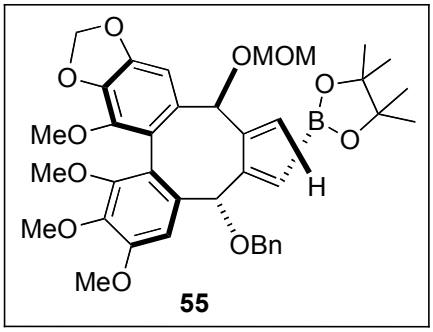
added. After 2 h, the reaction was quenched by the addition of Et_3N (0.02 mL) and the solvent was evaporated under reduced pressure to afford the crude product, which was purified by silica gel chromatography using 10 % EtOAc/hexane as the eluent to yield compound **51** (14 mg, 0.017 mmol) and compound **52** (20 mg, 0.017 mmol) in 70 % combined yield.

Compound 51: $[\alpha]_D$ – 62.5 (*c* 0.8, $CHCl_3$).; ^{1}H NMR (500 MHz, $CDCl_3$): δ 7.26-7.22 (m, 5 H), 6.95 (s, 1 H), 6.88 (s, 1 H), 6.37 (t, *J* = 35 Hz, 1H), 5.98 (d, *J* = 1.0 H, 1 H), 5.97 (d, *J* = 1.0 Hz, 1 H), 5.91 (s, 1 H), 4.97 (d, *J* = 6.5 H,z, 1H), 4.60 (s, 1 H), 4.61 (d, *J* = 6.5 Hz, 1 H), 4.32 (d, *J* = 11.0 Hz, 1 H), 4.24 (s, 1 H), 4.2 (d, *J* = 11.0 Hz, 1 H), 3.93 (s, 3 H), 3.83 (s, 3 H), 3.82 (s, 3 H), 3.51 (s, 3 H), 3.15 (s, 3 H), 1.16 (s, 6 H), 1.16 (s, 6 H), 0.05 (t, *J* = 22 Hz, 9 H).; ^{13}C NMR (125 MHz, $CDCl_3$): δ 164.63, 158.45, 154.32, 151.28, 149.89, 141.33, 140.31, 138.43, 137.84, 137.09, 135.83, 128.33, 128.03, 127.85, 127.53, 127.17, 120.93, 118.87, 103.18, 101.26, 99.27, 94.01, 83.17, 80.00, 74.98, 70.58, 61.27, 60.69, 59.92, 56.17, 55.43, 53.62, 31.13, 29.22, 29.58, 25.36, 24.99, 22.91, 14.32, 1.23.; MS (ESI): m/z: 875.2716 $[M+Na]^+$.

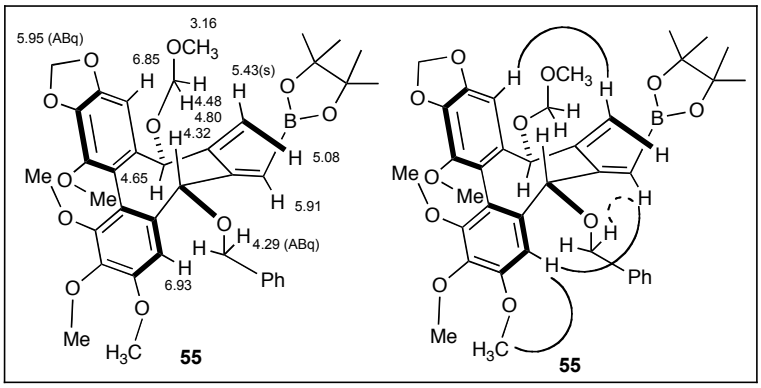


Chemical shifts and nOe's observed for 51 (assignments were done by COSY, after identifying the C*H*(Sn) proton at δ 6.375 which shows coupling to Sn)

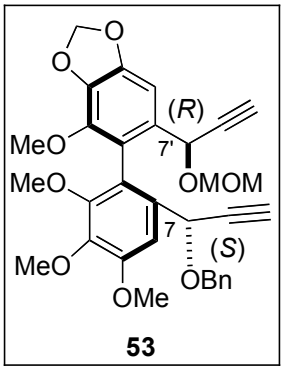
Compound 52: ^{1}H NMR (500 MHz, C_6D_6): δ 7.27 (d, *J* = 7.5 Hz, 2 H), 7.16-7.04 (m, 4 H), 6.87 (s, 1 H), 6.67 (d, *J* = 85 Hz, 1 H), 6.63 (d, *J* = 85 Hz, 1 H), 5.25-5.18 (m, 4 H), 4.60 (d, *J* = 6.0 Hz, 1 H), 4.50 (d, *J* = 6.0 Hz, 1 H), 4.44 (d, *J* = 11.0 Hz, 1 H), 4.07 (d, *J* = 11.0 Hz, 1 H), 3.89 (s, 6 H), 3.70 (s, 3 H), 3.48 (s, 3 H), 3.16 (s, 3 H),1.13-1.06 (m, 24 H), 0.53-0.39 (m, 18 H).; MS (ESI): m/z: 1163.3460 $[M+Na]^+$.



Synthesis of compound 55: Compound **55** was prepared from the compound **51** in 90% isolated yield following the procedure described for the sythesis of the compound **38**. $[\alpha]_D$ – 33.20 (*c* 0.25, $CHCl_3$).; ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.30-7.18 (m, aromatic), 6.92 (s, 1 H), 6.85 (s, 1 H), 5.97 (d, *J* =1.5 Hz, 1 H), 5.93 (d, *J* = 1.5 Hz, 1 H), 5.91 (d, *J* = 0.5 Hz, 1 H), 5.42 (s, 1 H), 5.08 (d, *J* = 2.0 Hz, 1 H), 4.80 (d, *J* = 6.5 Hz, 1 H), 4.65 (s, 1 H), 4.48 (d, *J* = 6.5 Hz, 1 H), 4.32 (s, 1 H), 4.31 (d, *J* = 11.0 Hz, 1 H), 4.27 (d, *J* = 11.0 Hz, 1 H), 3.90 (s, 3 H), 3.86 (s, 3 H), 3.77 (s, 3 H), 3.56 (s, 3 H), 0.85 (s, 6 H), 0.84 (s, 6 H).; ^{13}C NMR (125 MHz, $CDCl_3$) δ 160.26, 154.33, 151.12, 150.20, 149.91, 141.37, 138.53, 136.55, 136.11, 128.33, 127.78, 127.50, 120.58, 119.69, 113.21, 103.87, 101.35, 99.69, 93.89, 83.22, 78.66, 73.15, 70.52, 61.23, 60.83, 59.92, 56.10, 55.43, 53.64, 29.94, 25.14, 24.84, 24.74, 14.35.; MS (ESI) m/z: 711.2550 $[M+Na]^+$.

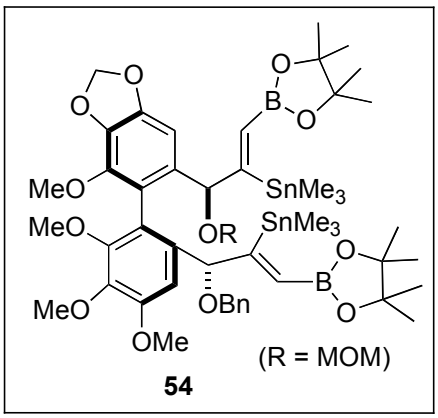


Chemical shifts and nOe's observed for 55



Synthesis of compound 53: Compound **53** was prepared from the compound **30** in 90 % yield following the procedure described for the synthesis of **50**: $[\alpha]_D$ +11.6 (*c* 0.6, $CHCl_3$).; ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.25-7.18 (m, 6 H), 6.98 (s, 1 H), 5.95 (s, 2 H), 4.91 (d, *J* = 2.0 Hz, 1 H), 4.81 (d, *J* = 6.5 Hz, 1 H), 4.79 (d, *J* = 2.5 Hz, 1 H), 4.58 (d, *J* = 11.5 Hz, 1 H), 4.42 (d, *J* = 6.5 Hz, 1 H), 4.20 (d, *J* = 11.5 Hz, 1 H), 3.91 (s, 3 H), 3.85 (s, 3 H), 3.68 (s, 3 H), 3.21 (s, 3 H), 2.59 (d, *J* = 2.0 Hz, 1 H), 2.49 (d, *J* = 2.5 Hz, 1H). ^{13}C NMR (125 MHz, $CDCl_3$): δ 153.51, 152.05, 149.45, 142.6, 141.65,

137.70, 136.42, 132.65, 131.57, 128.64, 128.32, 127.73, 122.32, 12.84, 107.75, 102.14, 101.34, 95.28, 82.28, 81.83, 76.07, 75.53, 71.20, 68.59, 66.0, 61.03, 60.87, 59.43, 56.36, 56.20, 53.64, 29.92, 14.34.; MS (ESI): m/z: 583.1866 $[M+Na]^+$.



54

Attempted [BSn]-Mediated Cyclization of 53, Adduct 54. Compound **54** was prepared from the diyne compound **53** in 85% yield following the procedure described for the synthesis of **52**: ^{1}H NMR (500 MHz, C_6D_6): δ 7.33 (d, J = 7.5 Hz, 2 H), 7.28 (s, 1 H), 7.16-7.05 (m, 3 H), 6.93 (s, 1 H), 6.71 (d, *J* = 85 Hz, 1 H), 6.10 (d, *J* = 85 Hz, 1 H), 5.31 (s, 1 H), 5.26 (s, 1 H), 5.22 (s, 2 H), 4.71 (d, *J* = 6.0 Hz, 1 H), 4.66 (d, *J* = 6.0 Hz, 1 H), 4.49 (d, *J* = 11.0 Hz, 1 H), 4.32 (d, J = 11.0 Hz, 1 H), 3.91 (s, 3 H), 3.84 (s, 3 H), 3.74 (s, 3 H), 3.34 (s, 3 H), 3.25 (s, 3 H), 1.08 (s, 6 H), 1.07 (s, 6 H), 0.98 (s, 12 H), 0.49-0.29 (m, 18 H); MS (ESI): m/z: 1165.2950 $[M+Na]^+$.
No trace of cyclization product(s) were detected under these conditions.

References

(1) Singidi, R. R.; RajanBabu, T. V. *Org. Lett.* **2008**, *10*, 3351.
(2) Singidi, R. R.; Kutney, A. M.; RajanBabu, T. V. *J. Am. Chem. Soc.* **2010**, *132*, 13078.
(3) Meyers, A. I.; Nelson, T. D.; Moorlag, H.; Rawsond D. J.; Meiere, A. *Tetrahedron* **2004**, *60*, 4459.
(4) Meyers, A. I.; Flisak, J. R.; Aitken, R. A. *J. Am. Chem. Soc.* **1987**, *109*, 5446.

9.472
7.826
7.805
7.304
7.283
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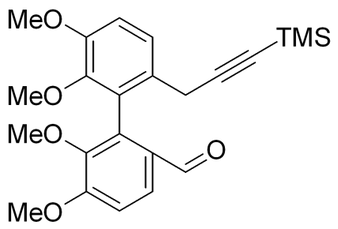
7.053
7.032
6.999
6.978
5.273
3.960
3.887
3.594
3.589
3.285
3.238
3.146
3.098
0.082
0.073
0.064



5A

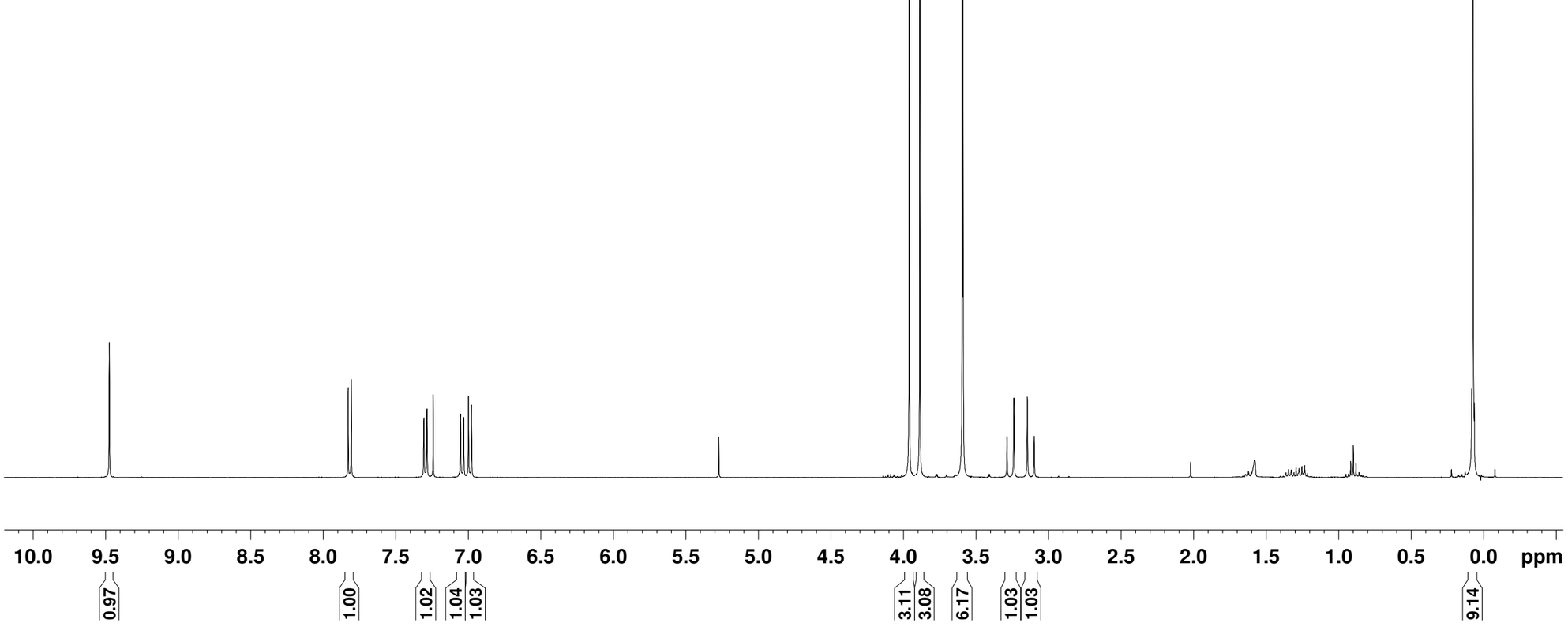
The Ohio State University
Department of Chemistry
NMR Facility
400MHz – 0083

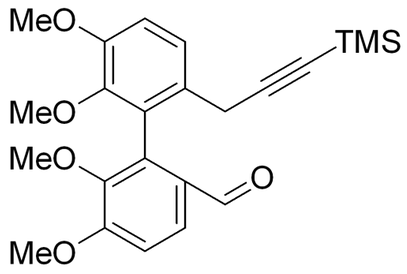
Current Data Parameters
NAME Rs-1-271
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20071012
Time 20.08
INSTRUM spect
PROBHD 5 mm QNP 1H/13
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 8278.146 Hz
FIDRES 0.126314 Hz
AQ 3.9584243 sec
RG 128
DW 60.400 use
DE 6.00 use
TE 0.0 K
D1 1.00000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec

======== CHANNEL f1 =======
NUC1 1H
P1 13.00 use
PL1 0.00 dB
SFO1 400.1324710 MHz

F2 - Processing parameters
SI 32768
SF 400.1300179 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00





5A

The Ohio State University
Department of Chemistry
NMR Facility
400MHz – 0083

```
Current Data Parameters
NAME          Rs-1-271-C
EXPNO                  1
PROCNO                 1

F2 - Acquisition Parameters
Date_           20071012
Time               20.55
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PULPROG          zgpg30
TD                65536
SOLVENT           CDCl3
NS                  755
DS                    4
SWH           23980.814 Hz
FIDRES         0.365918 Hz
AQ             1.3664756 sec
RG                 2048
DW                20.850 use
DE                 6.00 use
TE                  0.0 K
D1            2.00000000 sec
d11           0.03000000 sec
DELTA         1.89999998 sec
MCREST        0.00000000 sec
MCWRK         0.01500000 sec

======== CHANNEL f1 =======
NUC1                13C
P1                10.50 use
PL1                0.00 dB
SFO1        100.6228298 MHz

======== CHANNEL f2 =======
CPDPRG2          waltz16
NUC2                1H
PCPD2              80.00 use
PL2                -6.00 dB
PL12               14.56 dB
PL13               16.50 dB
SFO2        400.1316005 MHz

F2 - Processing parameters
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SF          100.6155318 MHz
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SSB                 0
LB                1.00 Hz
GB                  0
PC                1.00
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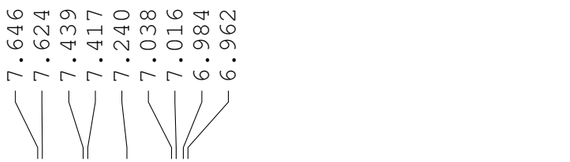


7A

The Ohio State University
Department of Chemistry
NMR Facility
400MHz – 0083

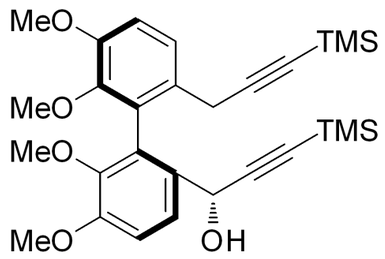
```
Current Data Parameters
NAME            Rs-1-273
EXPNO                  1
PROCNO                 1

F2 - Acquisition Parameters
Date_           20071016
Time                8.55
INSTRUM            spect
PROBHD   5 mm QNP 1H/13
PULPROG             zg30
TD                 65536
SOLVENT            CDCl3
NS                    16
DS                     2
SWH             8278.146 Hz
FIDRES          0.126314 Hz
AQ              3.9584243 sec
RG                  71.8
DW                 60.400 use
DE                  6.00 use
TE                   0.0 K
D1            1.00000000 sec
MCREST        0.00000000 sec
MCWRK         0.01500000 sec

======== CHANNEL f1 =======
NUC1                  1H
P1                13.00 use
PL1                0.00 dB
SFO1         400.1324710 MHz

F2 - Processing parameters
SI                 32768
SF          400.1300176 MHz
WDW                   EM
SSB                    0
LB                  0.30 Hz
GB                     0
PC                  1.00
```

152.78
151.46
145.84
145.75
132.88
129.19
128.98
128.50
123.94
123.64
112.80
112.46
105.26
104.57
90.61
87.61
77.55
77.43
77.23
76.91
62.67
60.95
60.71
56.03
55.98
23.88
0.32
0.09



7A


```
Current Data Parameters
NAME          Rs-1-273-C
EXPNO                  1
PROCNO                 1

F2 - Acquisition Parameters
Date_           20071016
Time                9.02
INSTRUM            spect
PROBHD   5 mm QNP 1H/13
PULPROG          zgpg30
TD                65536
SOLVENT            CDCl3
NS                   450
DS                     4
SWH           23980.814 Hz
FIDRES         0.365918 Hz
AQ            1.3664756 sec
RG               5160.6
DW                20.850 use
DE                 6.00 use
TE                  0.0 K
D1           2.00000000 sec
d11          0.03000000 sec
DELTA        1.89999998 sec
MCREST       0.00000000 sec
MCWRK        0.01500000 sec

======== CHANNEL f1 =======
NUC1                13C
P1                10.50 use
PL1                0.00 dB
SFO1        100.6228298 MHz

======== CHANNEL f2 =======
CPDPRG2         waltz16
NUC2                1H
PCPD2            80.00 use
PL2              -6.00 dB
PL12             14.56 dB
PL13             16.50 dB
SFO2        400.1316005 MHz

F2 - Processing parameters
SI                 32768
SF          100.6127483 MHz
WDW                EM
SSB                 0
LB               1.00 Hz
GB                  0
PC               1.40
```

7.673 7.652 7.364 7.342 7.240 7.053 7.031 6.971 6.950

5.271

4.922 4.918

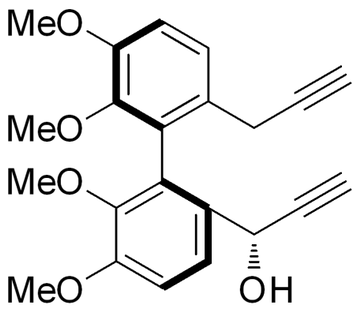
3.904 3.875 3.639 3.607 3.331 3.277 3.270 3.229 3.223 3.108 3.101 3.060 3.054

2.482 2.477

2.055 2.048 2.041 2.018

1.233

0.123



8A

The Ohio State University
Department of Chemistry
NMR Facility
400MHz – 0083

```
Current Data Parameters
NAME            Rs-I-72A
EXPNO                  1
PROCNO                 1

F2 - Acquisition Parameters
Date_           20070601
Time               14.06
INSTRUM            spect
PROBHD   5 mm QNP 1H/13
PULPROG             zg30
TD                 65536
SOLVENT            CDCl3
NS                    16
DS                     2
SWH             8278.146 Hz
FIDRES          0.126314 Hz
AQ              3.9584243 sec
RG                 143.7
DW                60.400 use
DE                 6.00 use
TE                300.2 K
D1             1.00000000 sec
MCREST         0.00000000 sec
MCWRK          0.01500000 sec

======== CHANNEL f1 =======
NUC1                   1H
P1                 13.00 use
PL1                 0.00 dB
SFO1         400.1324710 MHz

F2 - Processing parameters
SI                 32768
SF           400.1300176 MHz
WDW                   EM
SSB                    0
LB                  0.30 Hz
GB                     0
PC                  1.00
```

152.63
151.40
145.62
145.50
132.63
129.12
128.70
128.31
124.13
123.52
112.63
112.24
83.53
81.81
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77.01
76.69
73.60
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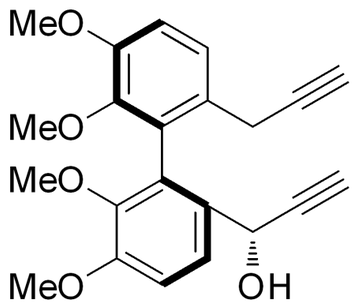
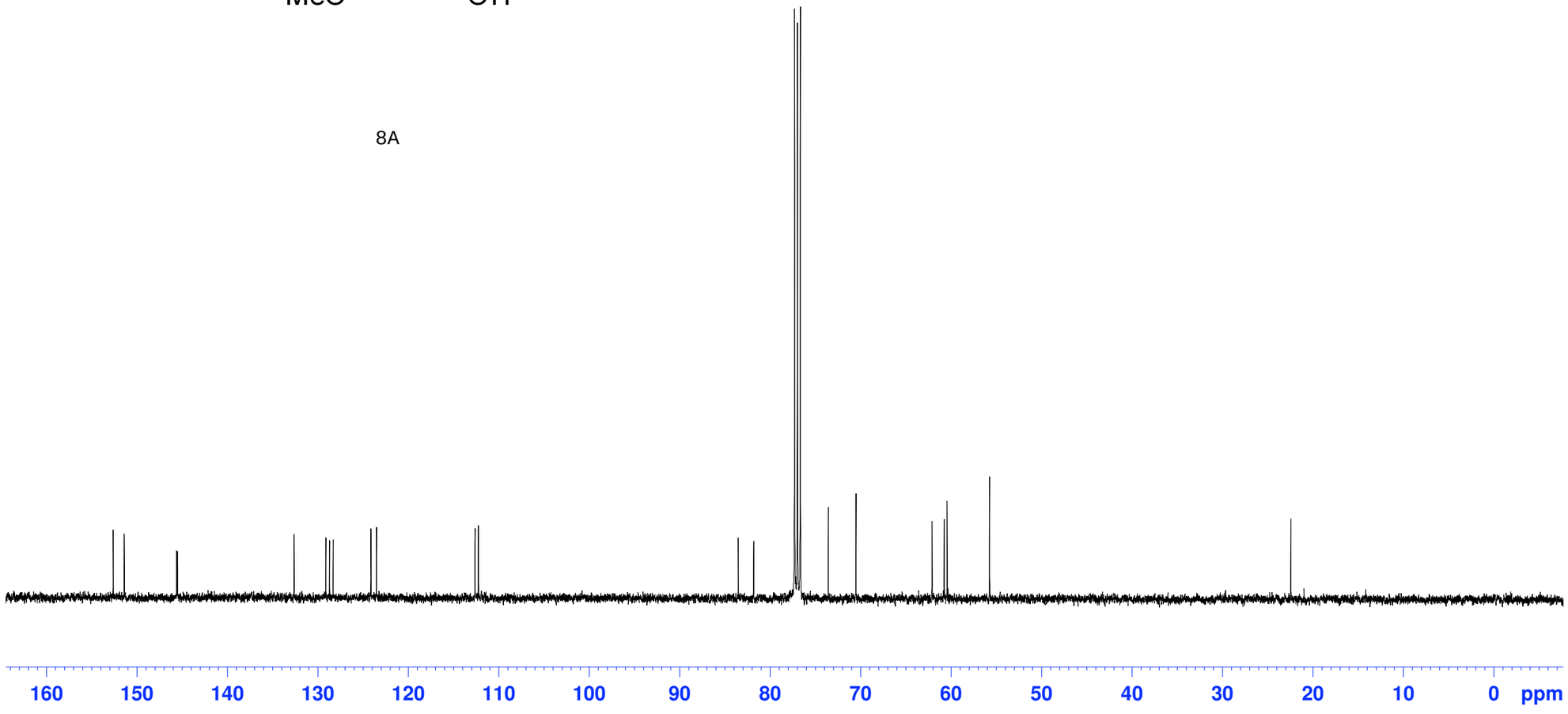
60.78
60.47
55.78
55.77
22.46



8A



Current Data Parameters
NAME Rs-I-72AC
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
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Time 14.39
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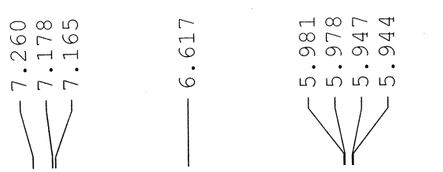
PROBHD 5 mm QNP 1H/13
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 492
DS 4
SWH 23980.814 Hz
FIDRES 0.365918 Hz
AQ 1.3664756 sec
RG 2896.3
DW 20.850 use
DE 6.00 use
TE 300.2 K
D1 2.00000000 sec
d11 0.03000000 sec
DELTA 1.89999998 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec

======== CHANNEL f1 =======
NUC1 13C
P1 10.50 use
PL1 0.00 dB
SFO1 100.6228298 MHz

======== CHANNEL f2 =======
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 use
PL2 -6.00 dB
PL12 14.56 dB
PL13 120.00 dB
SFO2 400.1316005 MHz

F2 - Processing parameters
SI 32768
SF 100.6127715 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40



11

BRUKER

```
Current Data Parameters
NAME          Wgong-I-56A
EXPNO                   2
PROCNO                  1

F2 - Acquisition Parameters
Date_            20090429
Time                17.05
INSTRUM             spect
PROBHD   5 mm QNP 1H/13
PULPROG              zg30
TD                  65536
SOLVENT             CDC13
NS                     16
DS                      2
SWH              8278.146 Hz
FIDRES           0.126314 Hz
AQ               3.9584243 sec
RG                  228.1
DW                 60.400 usec
DE                  6.00 usec
TE                 299.2 K
D1              1.00000000 sec
MCREST          0.00000000 sec
MCWRK           0.01500000 sec

======== CHANNEL f1 ========
NUC1                   1H
P1                  13.00 usec
PL1                  0.00 dB
SFO1          400.1324710 MHz

F2 - Processing parameters
SI                  32768
SF            400.1300096 MHz
WDW                    EM
SSB                     0
LB                   0.30 Hz
GB                      0
PC                   1.00
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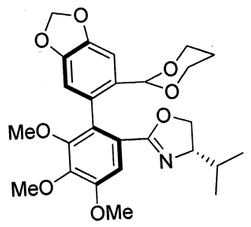

11

BRUKER

```
Current Data Parameters
NAME          wgong-I-56A
EXPNO                   2
PROCNO                  1

F2 - Acquisition Parameters
Date_            20090503
Time                11.26
INSTRUM             spect
PROBHD   5 mm QNP 1H/13
PULPROG           zgpg30
TD                 65536
SOLVENT            CDCl3
NS                   406
DS                     4
SWH            23980.814 Hz
FIDRES          0.365918 Hz
AQ             1.3664756 sec
RG                  1024
DW                20.850 usec
DE                 6.00 usec
TE                300.2 K
D1           2.00000000 sec
d11          0.03000000 sec
DELTA        1.89999998 sec
MCREST       0.00000000 sec
MCWRK        0.01500000 sec

======== CHANNEL f1 ========
NUC1                  13C
P1                10.50 usec
PL1                0.00 dB
SFO1         100.6228298 MHz

======== CHANNEL f2 ========
CPDPRG2          waltz16
NUC2                  1H
PCPD2             80.00 usec
PL2               -6.00 dB
PL12              14.56 dB
PL13              16.50 dB
SFO2         400.1316005 MHz

F2 - Processing parameters
SI                 32768
SF          100.6127690 MHz
WDW                   EM
SSB                    0
LB                 1.00 Hz
GB                     0
PC                  1.00
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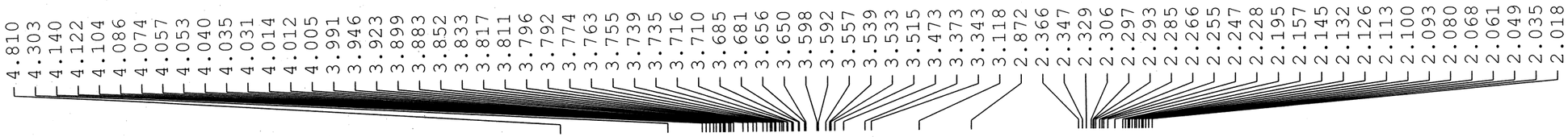

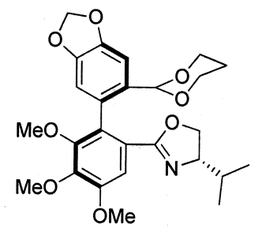

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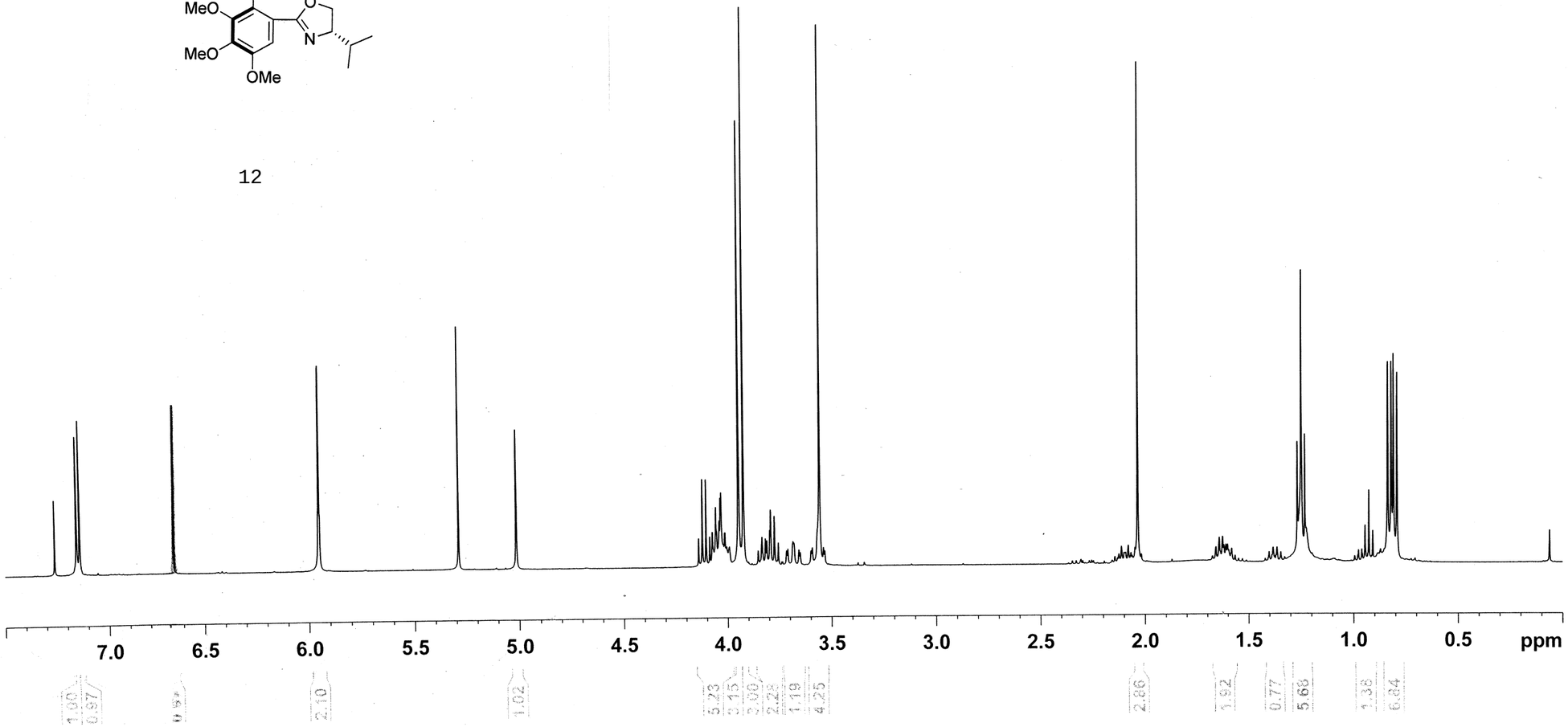


Current Data Parameters
NAME Wgong-I-35B
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters

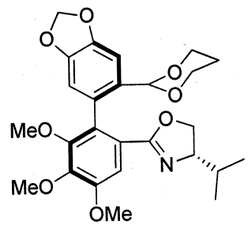
Date_	20080916
Time	15.24
INSTRUM	spect
PROBHD	5 mm QNP 1H/13
PULPROG	zg30
TD	65536
SOLVENT	CDCl3
NS	16
DS	2
SWH	8278.146 Hz
FIDRES	0.126314 Hz
AQ	3.9584243 sec
RG	143.7
DW	60.400 usec
DE	6.00 usec
TE	300.2 K
D1	1.00000000 sec
MCREST	0.00000000 sec
MCWRK	0.01500000 sec

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

NUC1	1H
P1	13.00 usec
PL1	0.00 dB
SFO1	400.1324710 MHz

F2 - Processing parameters

SI	32768
SF	400.1300096 MHz
WDW	EM
SSB	0
LB	0.30 Hz
GB	0
PC	1.00



12

BRUKER

```
Current Data Parameters
NAME      Wgong-I-35B p C13
EXPNO                  412
PROCNO                   1

F2 - Acquisition Parameters
Date_           20110305
Time              10.18
INSTRUM           spect
PROBHD   5 mm QNP 1H/13
PULPROG          zgpg30
TD                65536
SOLVENT           CDCl3
NS                   97
DS                    4
SWH           23980.814 Hz
FIDRES         0.365918 Hz
AQ             1.3664756 sec
RG                5792.6
DW                20.850 usec
DE                 6.00 usec
TE                300.2 K
D1            2.00000000 sec
d11           0.03000000 sec
DELTA         1.89999998 sec
TD0                   1

======== CHANNEL f1 ========
NUC1                 13C
P1                10.50 usec
PL1                0.00 dB
SFO1        100.6228298 MHz

======== CHANNEL f2 ========
CPDPRG2          waltz16
NUC2                 1H
PCPD2             80.00 usec
PL2               -6.00 dB
PL12              14.56 dB
PL13              16.50 dB
SFO2        400.1316005 MHz

F2 - Processing parameters
SI                32768
SF          100.6127690 MHz
WDW                  EM
SSB                   0
LB                 1.00 Hz
GB                    0
PC                 1.40
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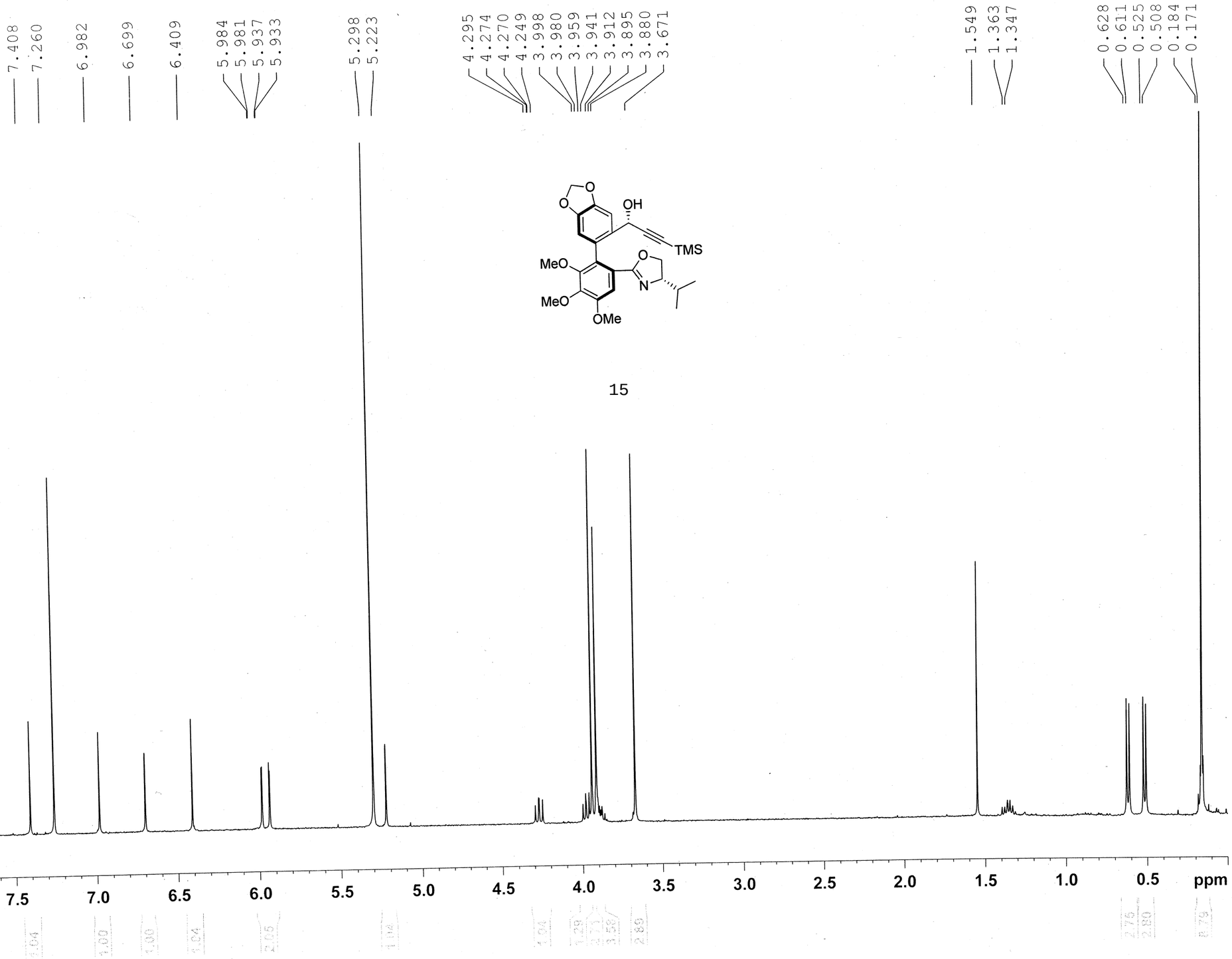


15

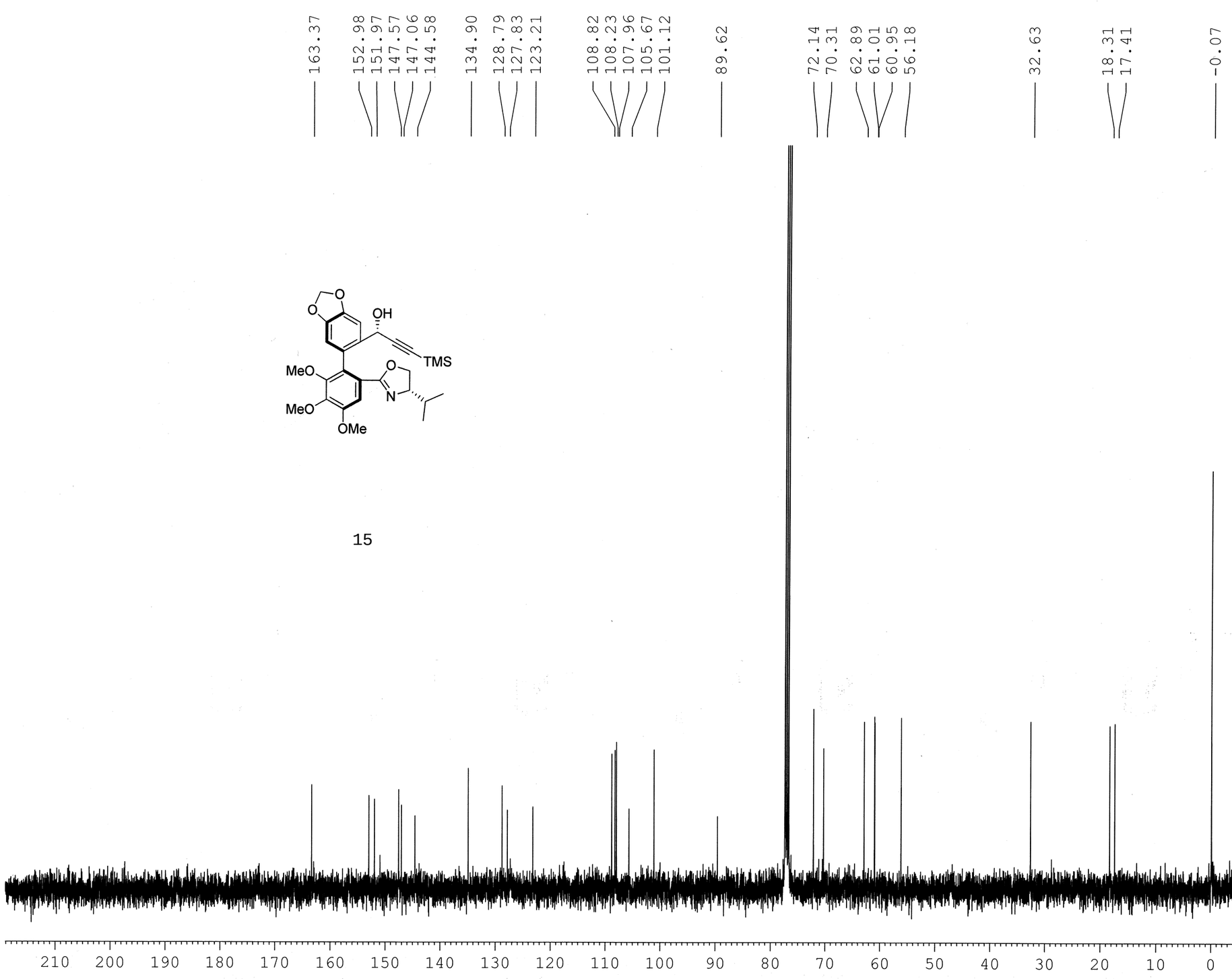
BRUKER

Current Data Parameters
NAME Wgong-I-109
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20081218
Time 12.23
INSTRUM spect
PROBHD 5 mm QNP 1H/13
PULPROG zg30
TD 65536
SOLVENT CDC13
NS 16
DS 2
SWH 8278.146 Hz
FIDRES 0.126314 Hz
AQ 3.9584243 sec
RG 1024
DW 60.400 usec
DE 6.00 usec
TE 299.2 K
D1 1.00000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec

======== CHANNEL f1 ========
NUC1 1H
P1 13.00 usec
PL1 0.00 dB
SFO1 400.1324710 MHz

F2 - Processing parameters
SI 32768
SF 400.1300096 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.40



BRUKER

Current Data Parameters
NAME Wgong-I-103 C13
EXPNO 413
PROCNO 1

F2 - Acquisition Parameters
Date_ 20110305
Time 11.11
INSTRUM spect
PROBHD 5 mm QNP 1H/13
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 337
DS 4
SWH 23980.814 Hz
FIDRES 0.365918 Hz
AQ 1.3664756 sec
RG 4096
DW 20.850 usec
DE 6.00 usec
TE 300.2 K
D1 2.00000000 sec
d11 0.03000000 sec
DELTA 1.89999998 sec
TD0 1

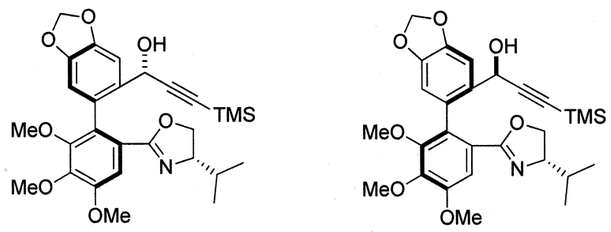
======== CHANNEL f1 ========
NUC1 13C
P1 10.50 usec
PL1 0.00 dB
SFO1 100.6228298 MHz

======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 -6.00 dB
PL12 14.56 dB
PL13 16.50 dB
SFO2 400.1316005 MHz

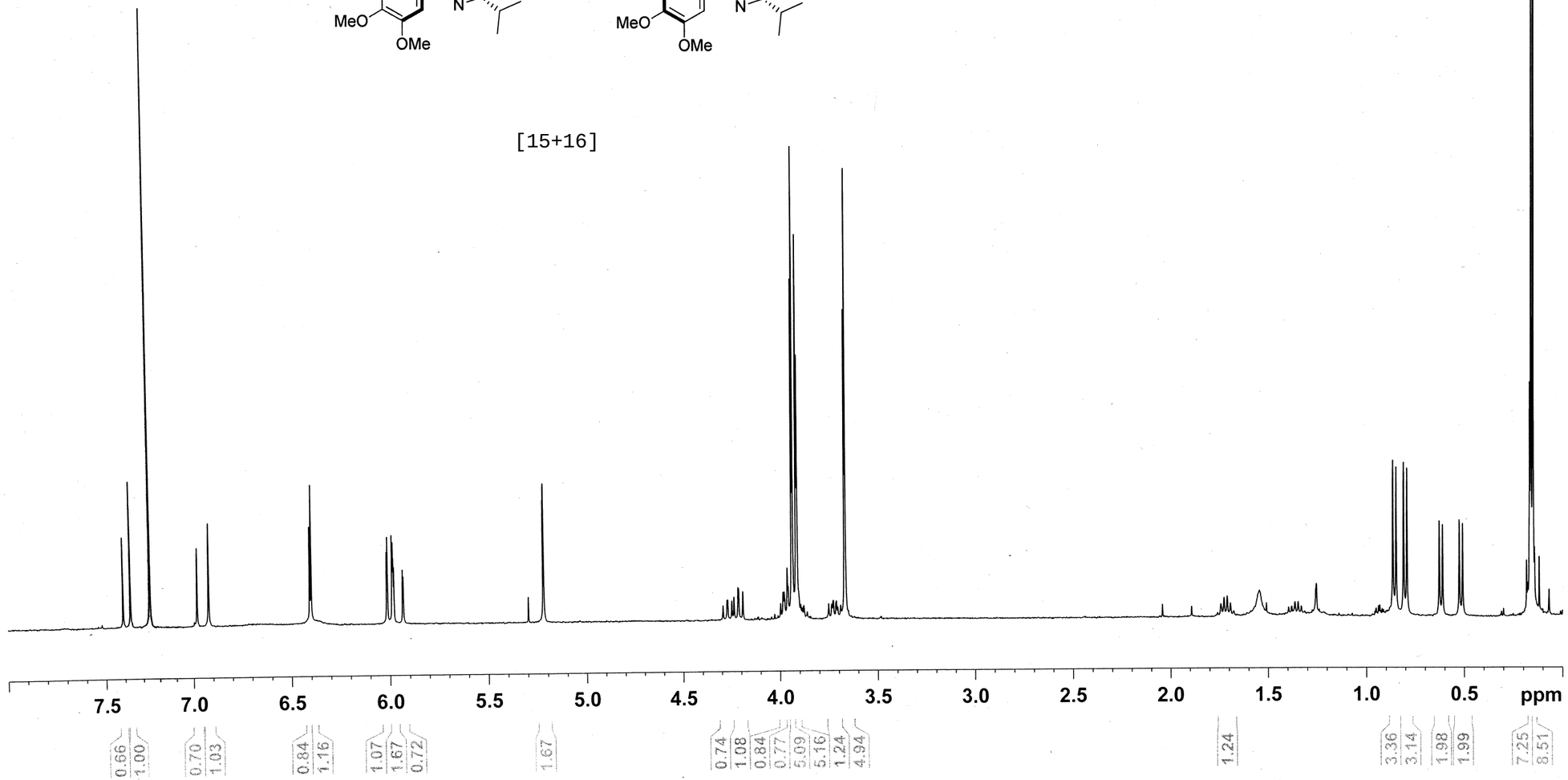
F2 - Processing parameters
SI 32768
SF 100.6127690 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

7.408 7.369 7.260 6.982 6.924 6.408 6.401 6.016 6.013 5.989 5.986 5.980 5.936 5.933 5.298 5.223 5.221 4.294 4.273 4.270 4.249 4.238 4.216 4.213 4.192 3.997 3.985 3.980 3.963 3.959 3.941 3.936 3.918 3.912 3.895 3.880 3.752 3.737 3.728 3.713 3.690 3.671 3.667

1.726 1.710 1.693 1.546 1.510 1.364 1.348 1.254 0.865 0.849 0.811 0.793 0.628 0.612 0.526 0.509 0.184 0.179



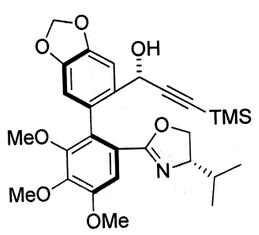
[15+16]

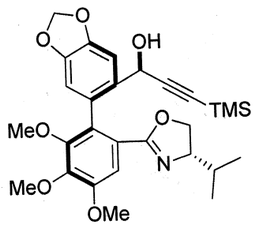


BRUKER

```
Current Data Parameters
NAME          Wgong-I-82
EXPNO                  1
PROCNO                 1

F2 - Acquisition Parameters
Date_           20081113
Time              10.01
INSTRUM           spect
PROBHD   5 mm QNP 1H/13
PULPROG            zg30
TD                65536
SOLVENT           CDCl3
NS                   16
DS                    2
SWH            8278.146 Hz
FIDRES            0.126314 Hz
AQ             3.9584243 sec
RG                 1024
DW                60.400 usec
DE                 6.00 usec
TE                300.2 K
D1             1.00000000 sec
MCREST         0.00000000 sec
MCWRK          0.01500000 sec

======== CHANNEL f1 ========
NUC1                  1H
P1                13.00 usec
PL1                0.00 dB
SFO1          400.1324710 MHz

F2 - Processing parameters
SI                 32768
SF            400.1300099 MHz
WDW                   EM
SSB                    0
LB                0.30 Hz
GB                     0
PC                1.00
```

163.58
153.07
152.01
147.56
146.98
144.40
134.88
128.31
127.59
123.67
108.73
108.25
107.80
105.56
101.21
89.63
77.36
77.04
76.72
72.36
72.16
70.32
70.13
62.91
62.75
61.04
60.95
56.20
32.64
32.07
18.72
18.32
17.96
17.43
-0.07





[15+16] 13C

BRUKER

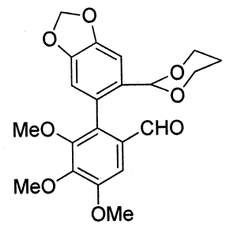
Current Data Parameters
NAME Wgong-II-44 C13
EXPNO 422
PROCNO 1

F2 - Acquisition Parameters
Date_ 20110310
Time 8.32
INSTRUM spect
PROBHD 5 mm QNP 1H/13
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 483
DS 4
SWH 23980.814 Hz
FIDRES 0.365918 Hz
AQ 1.3664756 sec
RG 5160.6
DW 20.850 usec
DE 6.00 usec
TE 300.2 K
D1 2.00000000 sec
d11 0.03000000 sec
DELTA 1.89999998 sec
TD0 1

======== CHANNEL f1 ========
NUC1 13C
P1 10.50 usec
PL1 0.00 dB
SFO1 100.6228298 MHz

======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 -6.00 dB
PL12 14.56 dB
PL13 16.50 dB
SFO2 400.1316005 MHz

F2 - Processing parameters
SI 32768
SF 100.6127677 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40



[17]

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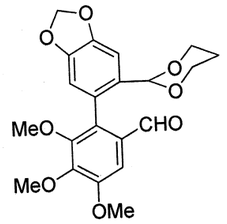
400MHz - 0083

```
Current Data Parameters
NAME          Wgong-I-261
EXPNO                   1
PROCNO                  1

F2 - Acquisition Parameters
Date_            20091020
Time                13.38
INSTRUM             spect
PROBHD   5 mm QNP 1H/13
PULPROG             zg30
TD                 65536
SOLVENT            CDCl3
NS                    16
DS                     2
SWH              8278.146 Hz
FIDRES           0.126314 Hz
AQ               3.9584243 sec
RG                  50.8
DW                60.400 usec
DE                 6.00 usec
TE                300.2 K
D1           1.00000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                  1H
P1                13.00 usec
PL1                0.00 dB
SFO1         400.1324710 MHz

F2 - Processing parameters
SI                 32768
SF           400.1300096 MHz
WDW                   EM
SSB                    0
LB                0.30 Hz
GB                     0
PC                1.00
```



[17]

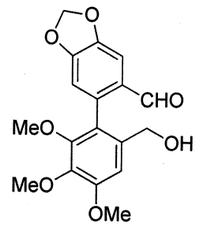
Current Data Parameters
NAME Wgong-I-261 C13
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20091020
Time 13.50
INSTRUM spect
PROBHD 5 mm QNP 1H/13
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 137
DS 4
SWH 23980.814 Hz
FIDRES 0.365918 Hz
AQ 1.3664756 sec
RG 2048
DW 20.850 usec
DE 6.00 usec
TE 300.2 K
D1 2.00000000 sec
d11 0.03000000 sec
DELTA 1.89999998 sec
TD0 1

======== CHANNEL f1 ========
NUC1 13C
P1 10.50 usec
PL1 0.00 dB
SFO1 100.6228298 MHz

======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 -6.00 dB
PL12 14.56 dB
PL13 16.50 dB
SFO2 400.1316005 MHz

F2 - Processing parameters
SI 32768
SF 100.6127690 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.00



[18]

9.492
7.427
7.260
6.915
6.690
6.085
5.285
4.349
4.317
4.302
4.270
3.917
3.895
3.870
3.661
3.609
1.890
1.185

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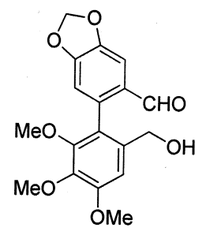
```
Current Data Parameters
NAME          Wgong-I-274
EXPNO                 483
PROCNO                  1

F2 - Acquisition Parameters
Date_          20110509
Time              15.24
INSTRUM           spect
PROBHD   5 mm QNP 1H/13
PULPROG           zg30
TD               65536
SOLVENT          CDCl3
NS                  16
DS                   2
SWH           8278.146 Hz
FIDRES        0.126314 Hz
AQ            3.9584243 sec
RG                101.6
DW             60.400 usec
DE              6.00 usec
TE              300.2 K
D1           1.00000000 sec
TD0                  1

======== CHANNEL f1 ========
NUC1                1H
P1             14.50 usec
PL1             0.00 dB
SFO1       400.1324710 MHz

F2 - Processing parameters
SI                32768
SF         400.1300095 MHz
WDW                EM
SSB                 0
LB              0.30 Hz
GB                  0
PC               1.00
```

190.38
153.81
152.30
151.39
148.01
141.29
136.92
135.05
129.63
122.60
110.92
106.94
106.11
102.15
77.37
77.05
76.73
62.79
60.95
60.85
56.07



[18]

BRUKER

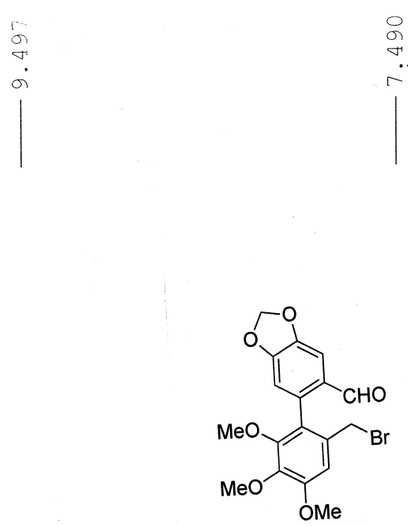
```
Current Data Parameters
NAME      Wgong-I-274 C13
EXPNO                482
PROCNO                 1

F2 - Acquisition Parameters
Date_           20110509
Time              15.12
INSTRUM           spect
PROBHD   5 mm QNP 1H/13
PULPROG          zgpg30
TD                65536
SOLVENT           CDCl3
NS                  170
DS                    4
SWH           23980.814 Hz
FIDRES         0.365918 Hz
AQ            1.3664756 sec
RG               1290.2
DW                20.850 usec
DE                 6.00 usec
TE                300.2 K
D1            2.00000000 sec
d11           0.03000000 sec
DELTA         1.89999998 sec
TD0                   1

======== CHANNEL f1 ========
NUC1                 13C
P1                10.50 usec
PL1                0.00 dB
SFO1        100.6228298 MHz

======== CHANNEL f2 ========
CPDPRG2          waltz16
NUC2                 1H
PCPD2             80.00 usec
PL2               -6.00 dB
PL12              14.56 dB
PL13              16.50 dB
SFO2        400.1316005 MHz

F2 - Processing parameters
SI                32768
SF          100.6127690 MHz
WDW                  EM
SSB                   0
LB                 1.00 Hz
GB                    0
PC                 1.40
```



[19]

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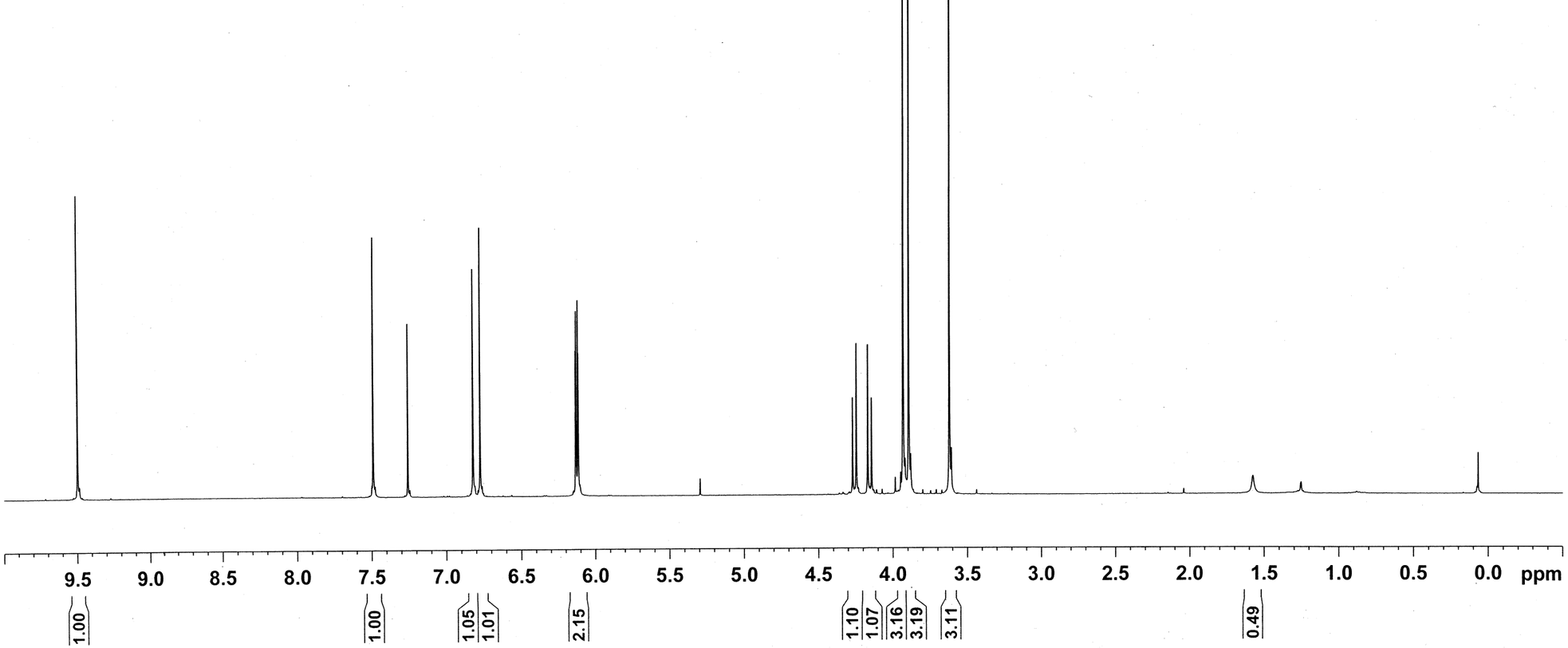
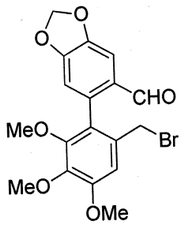
NMR Facility
400MHz - 0083

Current Data Parameters	
NAME	Wgong-II-30
EXPNO	14
PROCNO	1
F2 - Acquisition Parameters	
Date_	20091225
Time	12.27
INSTRUM	spect
PROBHD	5 mm QNP 1H/13
PULPROG	zg30
TD	65536
SOLVENT	CDCl3
NS	16
DS	2
SWH	8278.146 Hz
FIDRES	0.126314 Hz
AQ	3.9584243 sec
RG	322.5
DW	60.400 usec
DE	6.00 usec
TE	300.2 K
D1	1.00000000 sec
TD0	1
======== CHANNEL f1 ========	
NUC1	1H
P1	13.00 usec
PL1	0.00 dB
SFO1	400.1324710 MHz
F2 - Processing parameters	
SI	32768
SF	400.1300102 MHz
WDW	EM
SSB	0
LB	0.30 Hz
GB	0
PC	1.00





[19]

BRUKER

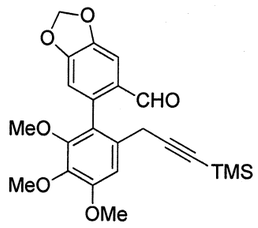
Current Data Parameters
NAME Wgong-II-30 C13
EXPNO 15
PROCNO 1

F2 - Acquisition Parameters
Date_ 20091227
Time 14.38
INSTRUM spect
PROBHD 5 mm QNP 1H/13
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 1024
DS 4
SWH 23980.814 Hz
FIDRES 0.365918 Hz
AQ 1.3664756 sec
RG 4096
DW 20.850 usec
DE 6.00 usec
TE 300.2 K
D1 2.00000000 sec
d11 0.03000000 sec
DELTA 1.89999998 sec
TD0 1

======== CHANNEL f1 ========
NUC1 13C
P1 10.50 usec
PL1 0.00 dB
SFO1 100.6228298 MHz

======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 -6.00 dB
PL12 14.56 dB
PL13 16.50 dB
SFO2 400.1316005 MHz

F2 - Processing parameters
SI 32768
SF 100.6127690 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40



[20]

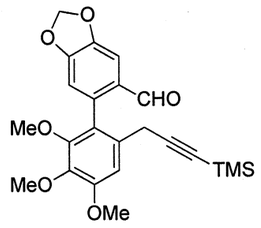
9.493
7.457
7.260
6.970
6.671
6.099
6.095
6.092
5.294
3.934
3.877
3.865
3.621
3.609
3.304
3.256
3.236
3.189
2.040
1.427
1.272
1.251
1.236
0.180
0.143

BRUKER

```
Current Data Parameters
NAME          Wgong-I-185
EXPNO                   2
PROCNO                  1

F2 - Acquisition Parameters
Date_            20090517
Time                11.04
INSTRUM             spect
PROBHD   5 mm QNP 1H/13
PULPROG              zg30
TD                  65536
SOLVENT             CDCl3
NS                     16
DS                      2
SWH              8278.146 Hz
FIDRES           0.126314 Hz
AQ               3.9584243 sec
RG                   143.7
DW                  60.400 usec
DE                   6.00 usec
TE                  298.2 K
D1            1.00000000 sec
MCREST        0.00000000 sec
MCWRK         0.01500000 sec

======== CHANNEL f1 ========
NUC1                   1H
P1                  13.00 usec
PL1                  0.00 dB
SFO1          400.1324710 MHz

F2 - Processing parameters
SI                  32768
SF            400.1300096 MHz
WDW                    EM
SSB                     0
LB                   0.30 Hz
GB                      0
PC                   1.00
```

9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 ppm

1.02 1.00 1.09 1.03 2.17 3.28 3.29 3.26 2.20 9.28



[20]

190.26
153.60
152.40
151.53
148.02
140.74
137.27
130.63
129.63
123.14
110.82
107.85
106.09
103.55
102.12
87.67
77.35
77.03
76.71
60.96
60.84
55.94
29.71
24.85
0.20
-0.08

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Current Data Parameters
NAME Wgong-I-185
EXPNO 2
PROCNO 1

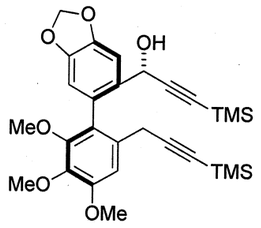
F2 - Acquisition Parameters
Date_ 20090517
Time 11.09
INSTRUM spect
PROBHD 5 mm QNP 1H/13
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 537
DS 4
SWH 23980.814 Hz
FIDRES 0.365918 Hz
AQ 1.3664756 sec
RG 3251
DW 20.850 usec
DE 6.00 usec
TE 298.2 K
D1 2.00000000 sec
d11 0.03000000 sec
DELTA 1.89999998 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec

======== CHANNEL f1 ========
NUC1 13C
P1 10.50 usec
PL1 0.00 dB
SFO1 100.6228298 MHz

======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 -6.00 dB
PL12 14.56 dB
PL13 16.50 dB
SFO2 400.1316005 MHz

F2 - Processing parameters
SI 32768
SF 100.6127690 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.00

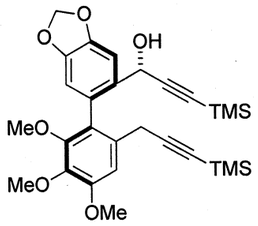
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm



[21A]

BRUKER

```
Current Data Parameters
NAME          Wgong-I-189
EXPNO                   2
PROCNO                  1

F2 - Acquisition Parameters
Date_            20090519
Time               20.23
INSTRUM            spect
PROBHD   5 mm QNP 1H/13
PULPROG             zg30
TD                 65536
SOLVENT            CDCl3
NS                    16
DS                     2
SWH             8278.146 Hz
FIDRES             0.126314 Hz
AQ              3.9584243 sec
RG                 143.7
DW                 60.400 usec
DE                  6.00 usec
TE                 298.2 K
D1              1.00000000 sec
MCREST          0.00000000 sec
MCWRK           0.01500000 sec

======== CHANNEL f1 ========
NUC1                   1H
P1                 13.00 usec
PL1                 0.00 dB
SFO1          400.1324710 MHz

F2 - Processing parameters
SI                  32768
SF            400.1300000 MHz
WDW                    EM
SSB                     0
LB                   0.30 Hz
GB                      0
PC                   1.40
```



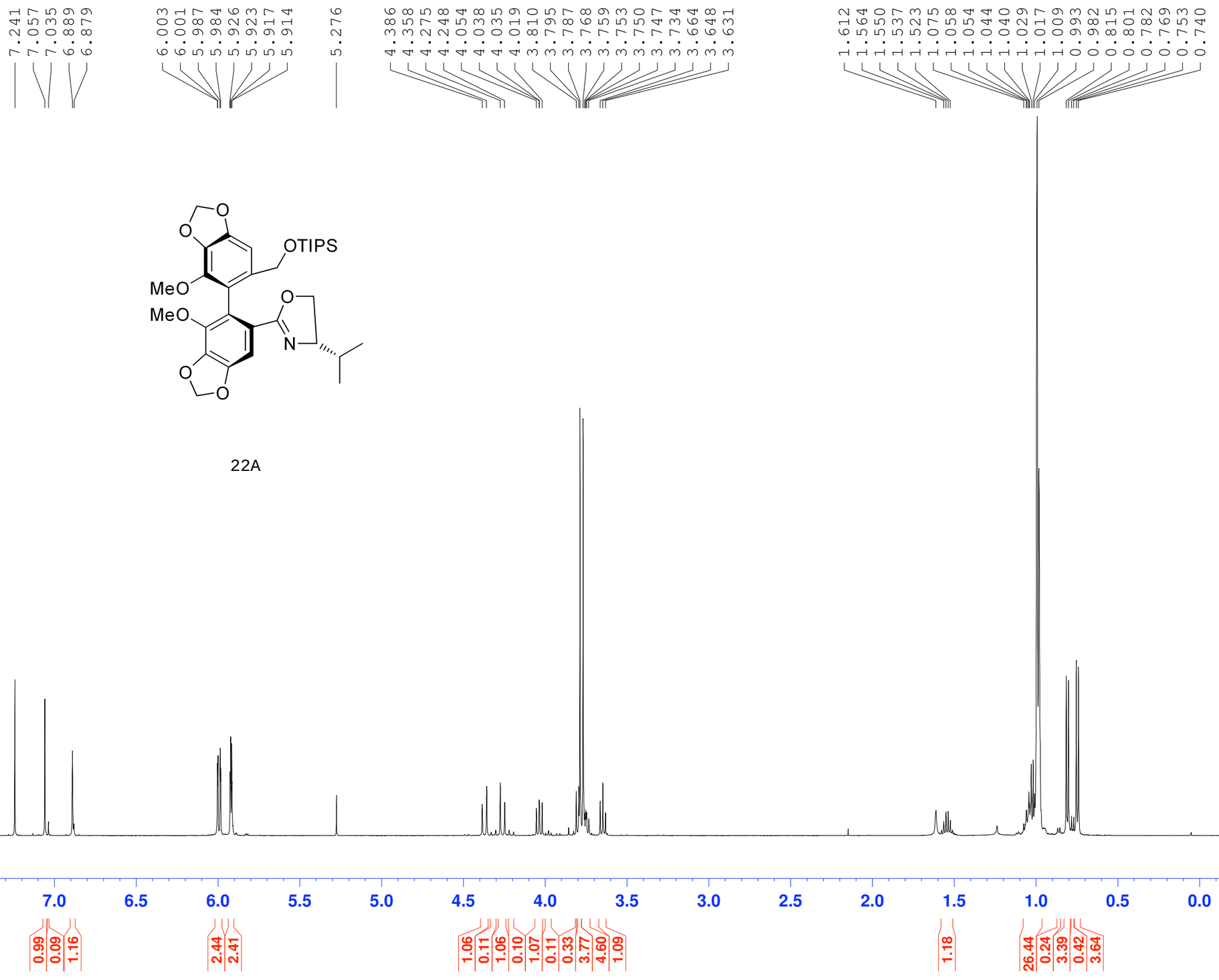
[21A]

153.15
150.51
147.81
147.66
140.89
133.93
130.92
127.65
125.27
109.88
108.44
108.13
104.69
104.11
101.42
90.75
87.97
77.34
77.03
76.71
62.75
61.47
61.17
55.91
53.43
29.71
24.60
1.02
0.00
-0.14

BRUKER

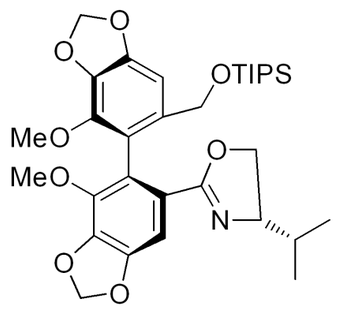
```
Current Data Parameters
NAME         Wgong-I-189
EXPNO                  2
PROCNO                 1

F2 - Acquisition Parameters
Date_           20090519
Time              20.31
INSTRUM           spect
PROBHD   5 mm QNP 1H/13
PULPROG         zgpg30
TD                65536
SOLVENT           CDCl3
NS                  457
DS                    4
SWH           23980.814 Hz
FIDRES         0.365918 Hz
AQ            1.3664756 sec
RG               1448.2
DW               20.850 usec
DE                6.00 usec
TE               298.2 K
D1          2.00000000 sec
d11         0.03000000 sec
DELTA       1.89999998 sec
MCREST      0.00000000 sec
MCWRK       0.01500000 sec

======== CHANNEL f1 ========
NUC1                13C
P1               10.50 usec
PL1               0.00 dB
SFO1        100.6228298 MHz

======== CHANNEL f2 ========
CPDPRG2         waltz16
NUC2                1H
PCPD2            80.00 usec
PL2              -6.00 dB
PL12             14.56 dB
PL13             16.50 dB
SFO2        400.1316005 MHz

F2 - Processing parameters
SI                32768
SF          100.6127690 MHz
WDW                EM
SSB                 0
LB               1.00 Hz
GB                  0
PC               1.00
```

210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm



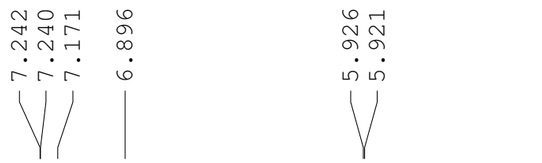

```
Current Data Parameters
NAME            Rs-1-278
EXPNO                  1
PROCNO                 1

F2 - Acquisition Parameters
Date_           20080204
Time               17.55
INSTRUM            spect
PROBHD   5 mm Multinucl
PULPROG             zg30
TD                 65536
SOLVENT            CDCl3
NS                    16
DS                     2
SWH           10330.578 Hz
FIDRES         0.157632 Hz
AQ            3.1719923 sec
RG                203.2
DW               48.400 usec
DE                6.00 usec
TE                300.2 K
D1           1.00000000 sec
MCREST       0.00000000 sec
MCWRK        0.01500000 sec

======== CHANNEL f1 ========
NUC1                  1H
P1                14.80 usec
PL1               -1.00 dB
SFO1         500.0230878 MHz

F2 - Processing parameters
SI                 32768
SF           500.0200213 MHz
WDW                   EM
SSB                    0
LB                0.30 Hz
GB                     0
PC                1.40
```

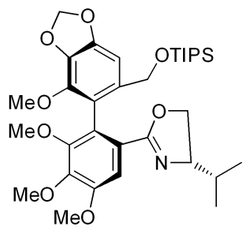
163.49
148.77
148.67
141.40
141.14
138.95
135.47
135.07
123.44
123.30
119.67
104.74
101.74
100.91
100.80
77.56
77.44
77.24
76.92
72.73
70.49
62.90
59.94
59.82
33.08
18.96
18.53
18.26
12.22
4.69



22A


```
Current Data Parameters
NAME                  Rs-1-278
EXPNO                        3
PROCNO                       1

F2 - Acquisition Parameters
Date_                 20080204
Time                     15.37
INSTRUM                  spect
PROBHD    5 mm QNP 1H/13
PULPROG                 zgpg30
TD                       65536
SOLVENT                  CDCl3
NS                         750
DS                           4
SWH                  23980.814 Hz
FIDRES                0.365918 Hz
AQ                   1.3664756 sec
RG                      3649.1
DW                      20.850 use
DE                       6.00 use
TE                      300.2 K
D1                   2.00000000 sec
d11                  0.03000000 sec
DELTA                1.89999998 sec
MCREST               0.00000000 sec
MCWRK                0.01500000 sec

======== CHANNEL f1 =======
NUC1                       13C
P1                       10.50 use
PL1                       0.00 dB
SFO1                100.6228298 MHz

======== CHANNEL f2 =======
CPDPRG2                waltz16
NUC2                       1H
PCPD2                    80.00 use
PL2                      -6.00 dB
PL12                     14.56 dB
PL13                     16.50 dB
SFO2                400.1316005 MHz

F2 - Processing parameters
SI                         32768
SF                  100.6127461 MHz
WDW                           EM
SSB                            0
LB                        1.00 Hz
GB                             0
PC                        1.40
```

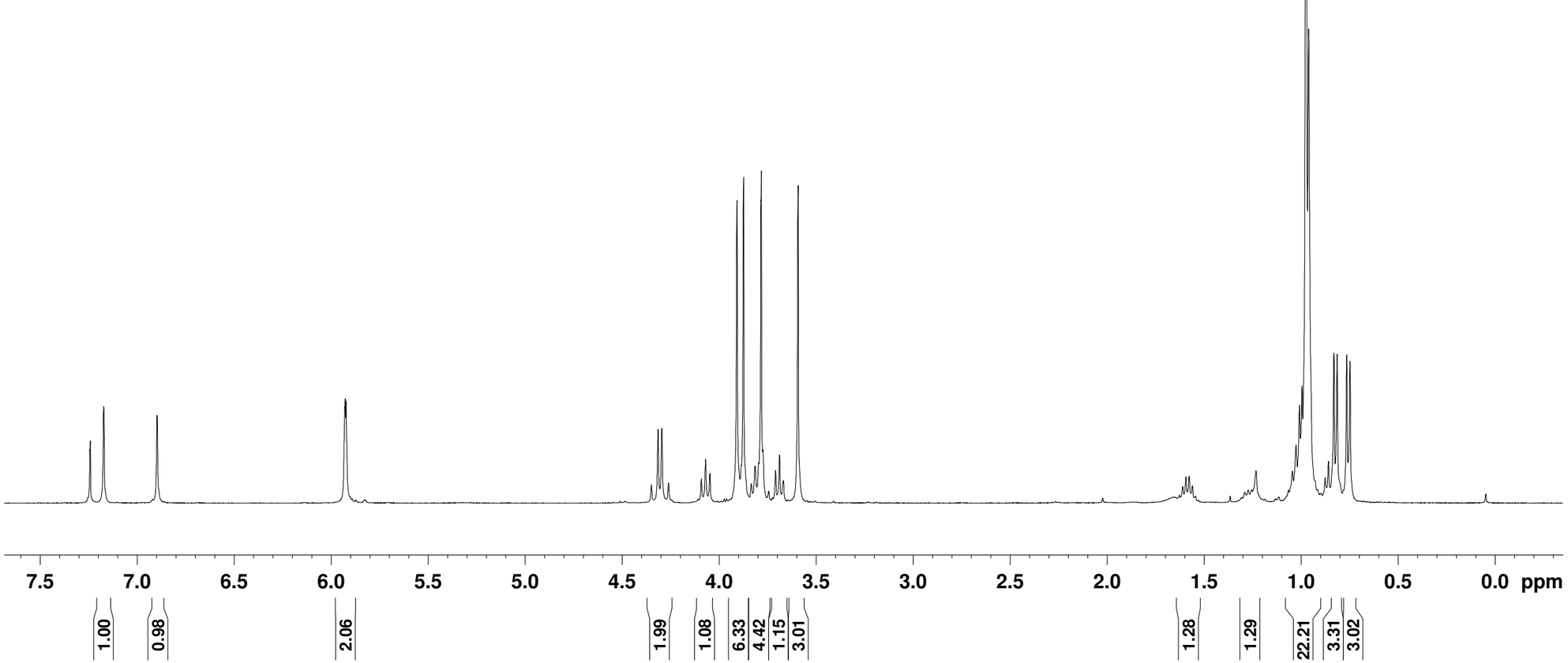




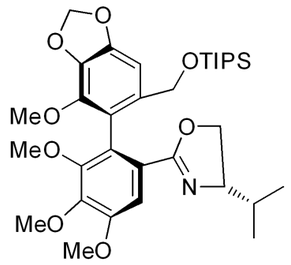
22B

The Ohio State University
Department of Chemistry
NMR Facility
400MHz – 0083

```
Current Data Parameters
NAME                  Rs-2-89MP
EXPNO                         1
PROCNO                        1

F2 - Acquisition Parameters
Date_                  20080105
Time                      11.22
INSTRUM                   spect
PROBHD    5 mm QNP 1H/13
PULPROG                    zg30
TD                        65536
SOLVENT                   CDCl3
NS                           16
DS                            2
SWH                    8278.146 Hz
FIDRES                 0.126314 Hz
AQ                     3.9584243 sec
RG                          128
DW                       60.400 use
DE                        6.00 use
TE                       297.2 K
D1                    1.00000000 sec
MCREST                0.00000000 sec
MCWRK                 0.01500000 sec

======== CHANNEL f1 =======
NUC1                         1H
P1                        13.00 use
PL1                        0.00 dB
SFO1                400.1324710 MHz

F2 - Processing parameters
SI                           32768
SF                  400.1300176 MHz
WDW                             EM
SSB                              0
LB                        0.30 Hz
GB                               0
PC                        1.00
```

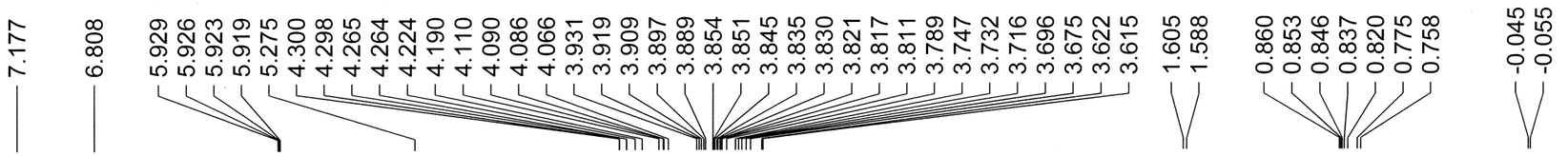


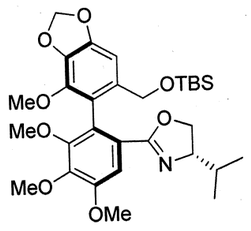
163.75
152.62
151.78
148.73
144.43
141.02
135.36
134.81
124.69
123.56
119.63
108.95
100.85
100.57
77.55
77.23
76.92
72.72
70.44
62.93
61.07
60.90
59.68
56.26
33.01
18.99
18.42
18.23
12.15



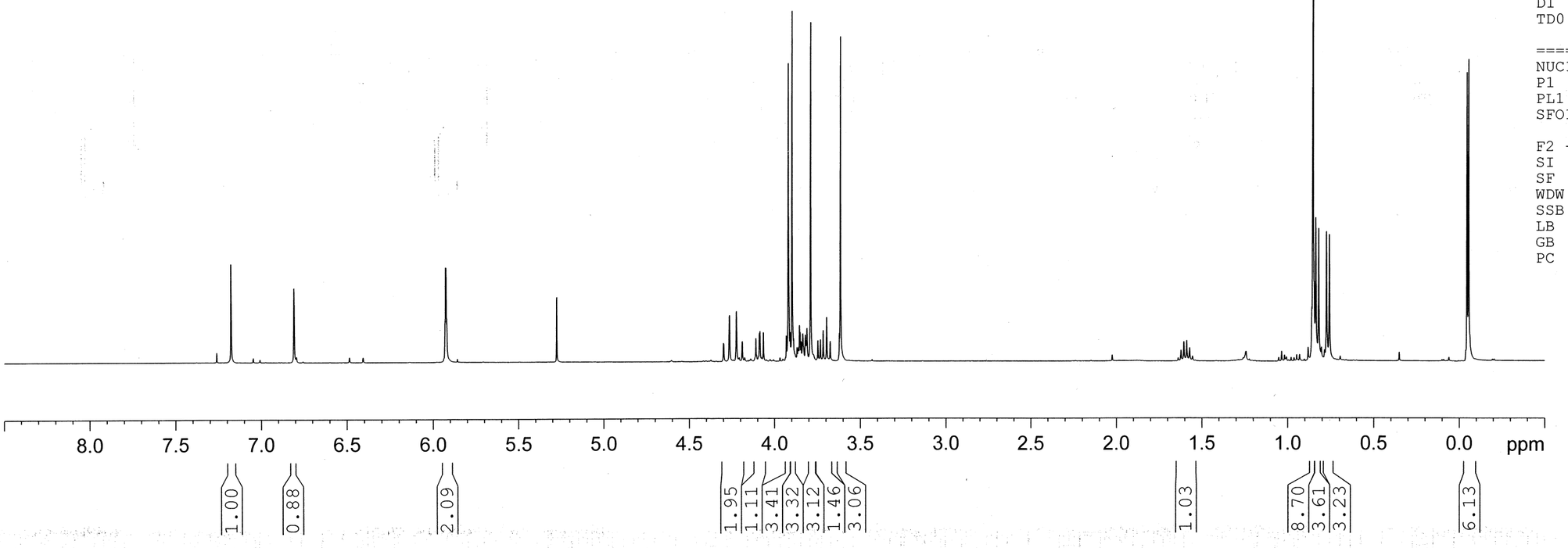
22B


```
Current Data Parameters
NAME                  Rs-2-89MP
EXPNO                         2
PROCNO                        1

F2 - Acquisition Parameters
Date_                  20080105
Time                      11.28
INSTRUM                   spect
PROBHD    5 mm QNP 1H/13
PULPROG                  zgpg30
TD                        65536
SOLVENT                   CDC13
NS                         1000
DS                            4
SWH                   23980.814 Hz
FIDRES                 0.365918 Hz
AQ                    1.3664756 sec
RG                        13004
DW                       20.850 use
DE                        6.00 use
TE                       298.2 K
D1                    2.00000000 sec
d11                   0.03000000 sec
DELTA                 1.89999998 sec
MCREST                0.00000000 sec
MCWRK                 0.01500000 sec

======== CHANNEL f1 =======
NUC1                        13C
P1                        10.50 use
PL1                        0.00 dB
SFO1                100.6228298 MHz

======== CHANNEL f2 =======
CPDPRG2                 waltz16
NUC2                        1H
PCPD2                     80.00 use
PL2                       -6.00 dB
PL12                      14.56 dB
PL13                      16.50 dB
SFO2                400.1316005 MHz

F2 - Processing parameters
SI                        32768
SF                  100.6127483 MHz
WDW                          EM
SSB                           0
LB                         1.00 Hz
GB                            0
PC                         1.40
```





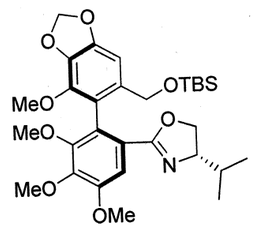
22C



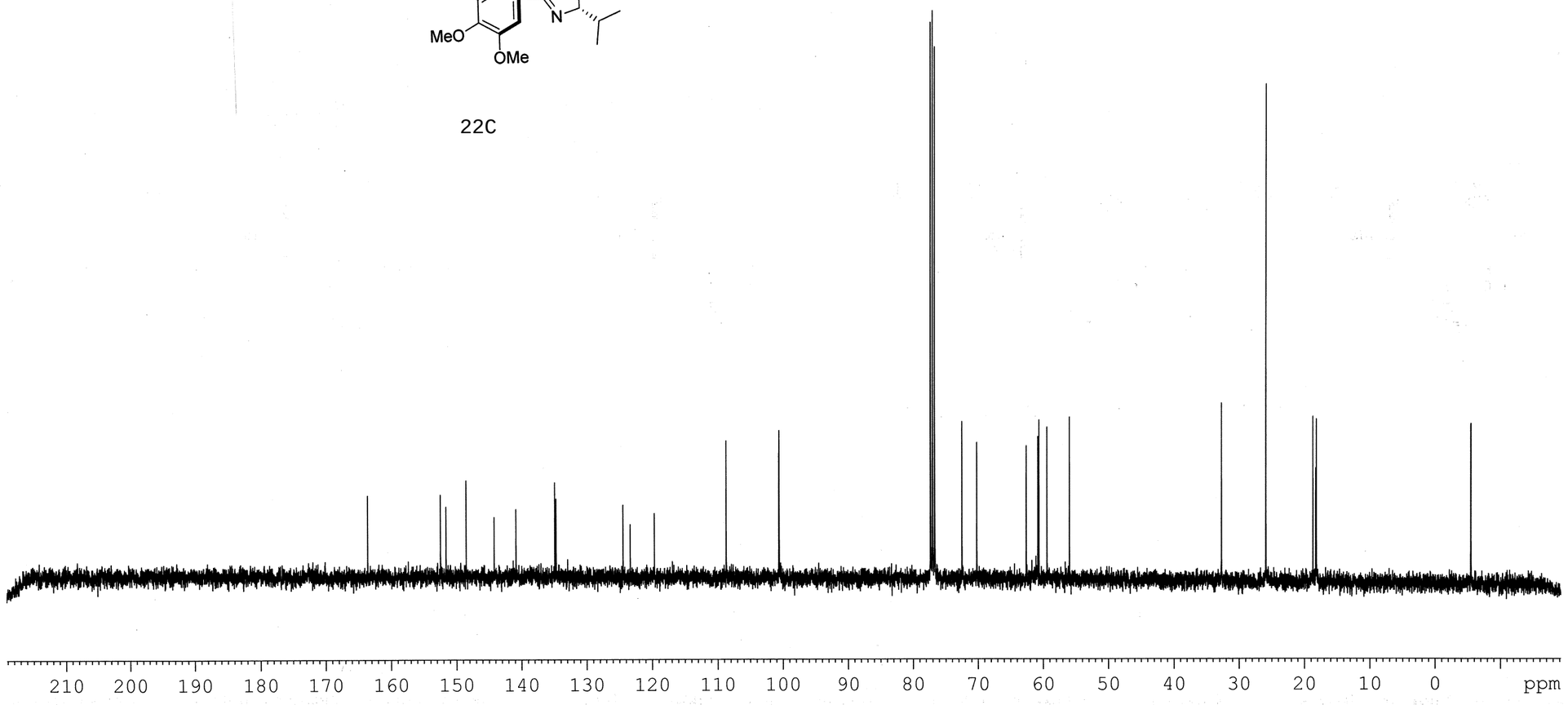
The Ohio State University
Department of Chemistry
NMR Facility

```
Current Data Parameters
NAME       Wgong-III-216
EXPNO                470
PROCNO                 1

F2 - Acquisition Parameters
Date_           20110429
Time               14.54
INSTRUM            spect
PROBHD   5 mm QNP 1H/13
PULPROG             zg30
TD                 65536
SOLVENT            CDCl3
NS                    16
DS                     2
SWH             8278.146 Hz
FIDRES          0.126314 Hz
AQ              3.9584243 sec
RG                  45.3
DW                60.400 usec
DE                 6.00 usec
TE                300.2 K
D1             1.00000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                  1H
P1                 14.50 usec
PL1                 0.00 dB
SFO1          400.1324710 MHz

F2 - Processing parameters
SI                 32768
SF            400.1300093 MHz
WDW                   EM
SSB                    0
LB                  0.30 Hz
GB                     0
PC                  1.00
```

163.59
152.47
151.63
148.52
144.21
140.86
134.92
134.72
124.46
123.36
119.68
108.72
100.66
100.60
77.36
77.04
76.72
72.52
70.23
62.63
60.85
60.68
59.45
56.01
32.74
25.94
18.74
18.31
18.21
-5.42
-5.45



22C



BRUKER

Current Data Parameters
NAME Wgong-III-216 C13
EXPNO 501
PROCNO 1

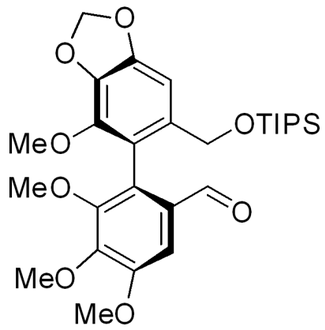
F2 - Acquisition Parameters
Date_ 20110517
Time 16.51
INSTRUM spect
PROBHD 5 mm QNP 1H/13
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 92
DS 4
SWH 23980.814 Hz
FIDRES 0.365918 Hz
AQ 1.3664756 sec
RG 1149.4
DW 20.850 usec
DE 6.00 usec
TE 300.2 K
D1 2.00000000 sec
d11 0.03000000 sec
DELTA 1.89999998 sec
TD0 1

======== CHANNEL f1 ========
NUC1 13C
P1 10.50 usec
PL1 0.00 dB
SFO1 100.6228298 MHz

======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 -6.00 dB
PL12 14.56 dB
PL13 16.50 dB
SFO2 400.1316005 MHz

F2 - Processing parameters
SI 32768
SF 100.6127690 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

9.525
7.322
7.240
6.899
5.981
5.975
5.972
4.394
4.362
4.191
4.158
3.940
3.929
3.823
3.593
2.020
1.230
1.009
0.999
0.992
0.973
0.960
0.941
0.937
0.927

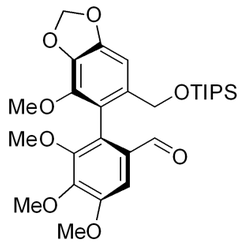


23B

The Ohio State University
Department of Chemistry
NMR Facility
400MHz – 0083

```
Current Data Parameters
NAME                  Rs-2-90
EXPNO                       1
PROCNO                      1

F2 - Acquisition Parameters
Date_               20080106
Time                   21.12
INSTRUM                spect
PROBHD    5 mm QNP 1H/13
PULPROG                 zg30
TD                     65536
SOLVENT                CDCl3
NS                        16
DS                         2
SWH                 8278.146 Hz
FIDRES              0.126314 Hz
AQ                  3.9584243 sec
RG                     101.6
DW                    60.400 use
DE                     6.00 use
TE                    296.2 K
D1                 1.00000000 sec
MCREST             0.00000000 sec
MCWRK              0.01500000 sec

======== CHANNEL f1 =======
NUC1                       1H
P1                     13.00 use
PL1                     0.00 dB
SFO1              400.1324710 MHz

F2 - Processing parameters
SI                         32768
SF                400.1300181 MHz
WDW                           EM
SSB                            0
LB                      0.30 Hz
GB                             0
PC                      1.00
```

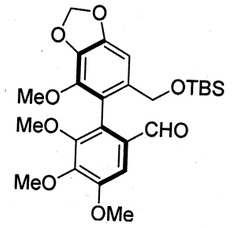
191.41
153.40
151.60
149.81
147.87
141.29
135.86
134.93
130.24
128.23
115.68
105.21
101.76
101.27
77.55
77.24
76.92
63.23
61.22
61.06
59.63
56.28
18.15
12.08



23B

BRUKER

```
Current Data Parameters
NAME                  Rs-2-90
EXPNO                       2
PROCNO                      1

F2 - Acquisition Parameters
Date_              20080106
Time                  21.18
INSTRUM               spect
PROBHD    5 mm QNP 1H/13
PULPROG             zgpg30
TD                    65536
SOLVENT               CDC13
NS                      354
DS                        4
SWH               23980.814 Hz
FIDRES             0.365918 Hz
AQ                1.3664756 sec
RG                     3251
DW                   20.850 use
DE                    6.00 use
TE                   297.2 K
D1                2.00000000 sec
d11               0.03000000 sec
DELTA             1.89999998 sec
MCREST            0.00000000 sec
MCWRK             0.01500000 sec

======== CHANNEL f1 =======
NUC1                    13C
P1                    10.50 use
PL1                    0.00 dB
SFO1           100.6228298 MHz

======== CHANNEL f2 =======
CPDPRG2             waltz16
NUC2                    1H
PCPD2                80.00 use
PL2                  -6.00 dB
PL12                 14.56 dB
PL13                 16.50 dB
SFO2           400.1316005 MHz

F2 - Processing parameters
SI                     32768
SF                100.6127483 MHz
WDW                       EM
SSB                        0
LB                    1.00 Hz
GB                         0
PC                    1.40
```

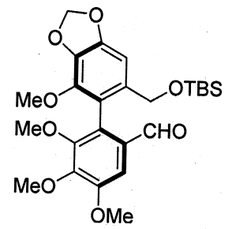


23C

The Ohio State University
Department of Chemistry
NMR Facility

```
Current Data Parameters
NAME       Wgong-III-223
EXPNO                475
PROCNO                 1

F2 - Acquisition Parameters
Date_           20110505
Time              12.18
INSTRUM           spect
PROBHD   5 mm QNP 1H/13
PULPROG           zg30
TD               65536
SOLVENT          CDCl3
NS                  16
DS                   2
SWH           8278.146 Hz
FIDRES        0.126314 Hz
AQ            3.9584243 sec
RG                 181
DW              60.400 usec
DE               6.00 usec
TE              300.2 K
D1         1.00000000 sec
TD0                  1

======== CHANNEL f1 ========
NUC1                1H
P1              14.50 usec
PL1              0.00 dB
SFO1       400.1324710 MHz

F2 - Processing parameters
SI                32768
SF         400.1300098 MHz
WDW                EM
SSB                 0
LB              0.30 Hz
GB                  0
PC               1.00
```

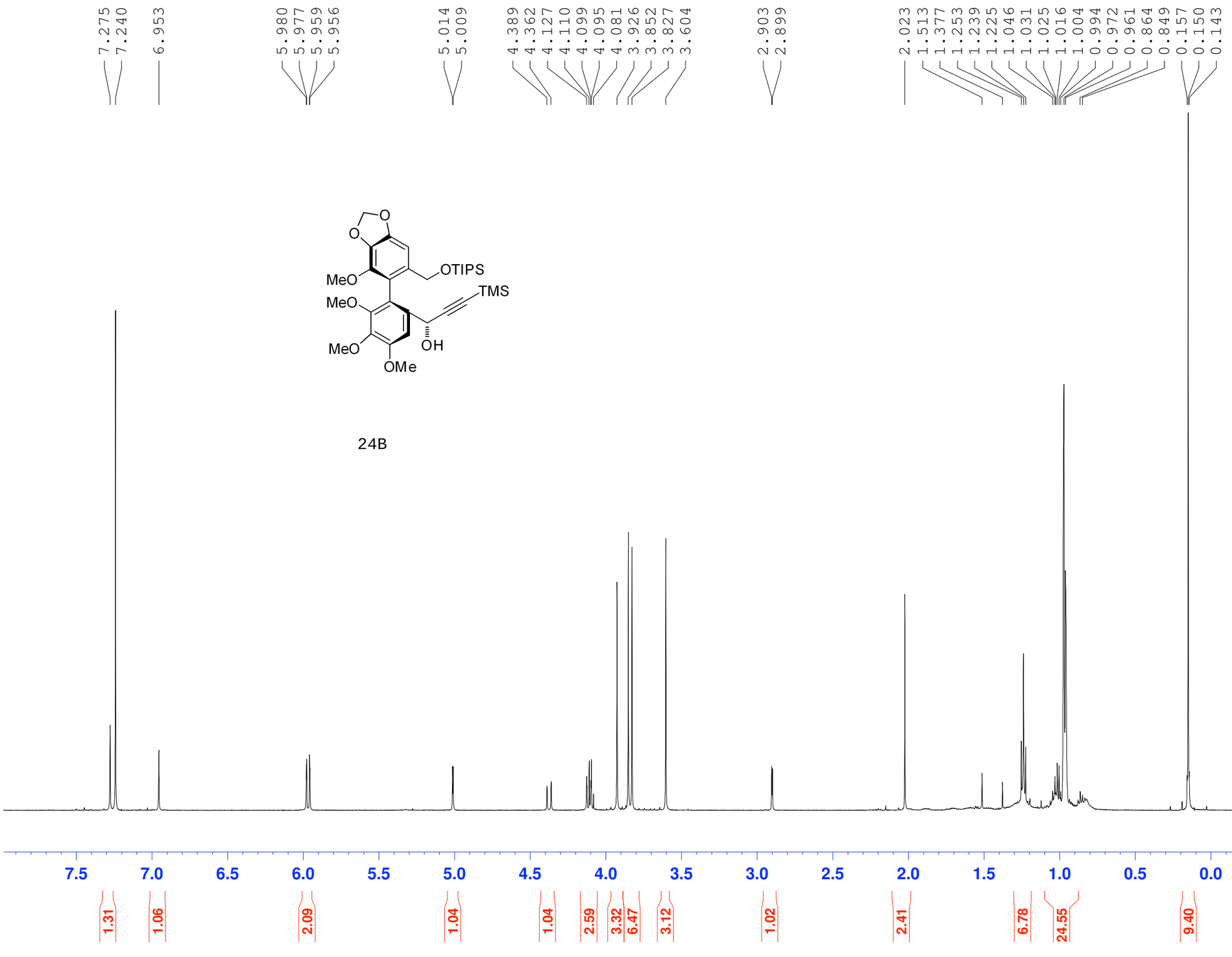
191.20
153.23
151.38
149.56
147.63
141.17
135.29
134.92
130.09
128.07
115.93
104.99
101.88
101.09
77.35
77.03
76.71
67.96
62.97
61.00
60.83
59.42
56.05
25.88
25.61
18.32
-5.52
-5.54



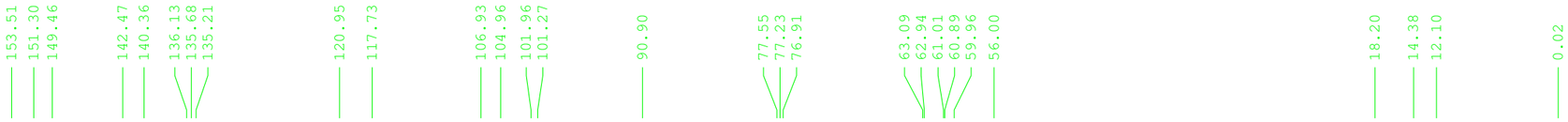
23C

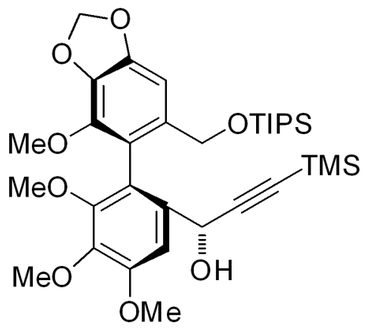
BRUKER

```
Current Data Parameters
NAME      Wgong-III-223 C13
EXPNO                  534
PROCNO                   1

F2 - Acquisition Parameters
Date_           20110526
Time                8.44
INSTRUM            spect
PROBHD   5 mm QNP 1H/13
PULPROG          zgpg30
TD                65536
SOLVENT            CDCl3
NS                   267
DS                     4
SWH            23980.814 Hz
FIDRES          0.365918 Hz
AQ              1.3664756 sec
RG                 1625.5
DW                 20.850 usec
DE                  6.00 usec
TE                 300.2 K
D1             2.00000000 sec
d11            0.03000000 sec
DELTA          1.89999998 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                  13C
P1                 10.50 usec
PL1                 0.00 dB
SFO1         100.6228298 MHz

======== CHANNEL f2 ========
CPDPRG2          waltz16
NUC2                1H
PCPD2              80.00 usec
PL2                -6.00 dB
PL12               14.56 dB
PL13               16.50 dB
SFO2         400.1316005 MHz

F2 - Processing parameters
SI                 32768
SF           100.6127690 MHz
WDW                   EM
SSB                    0
LB                  1.00 Hz
GB                     0
PC                  1.40
```

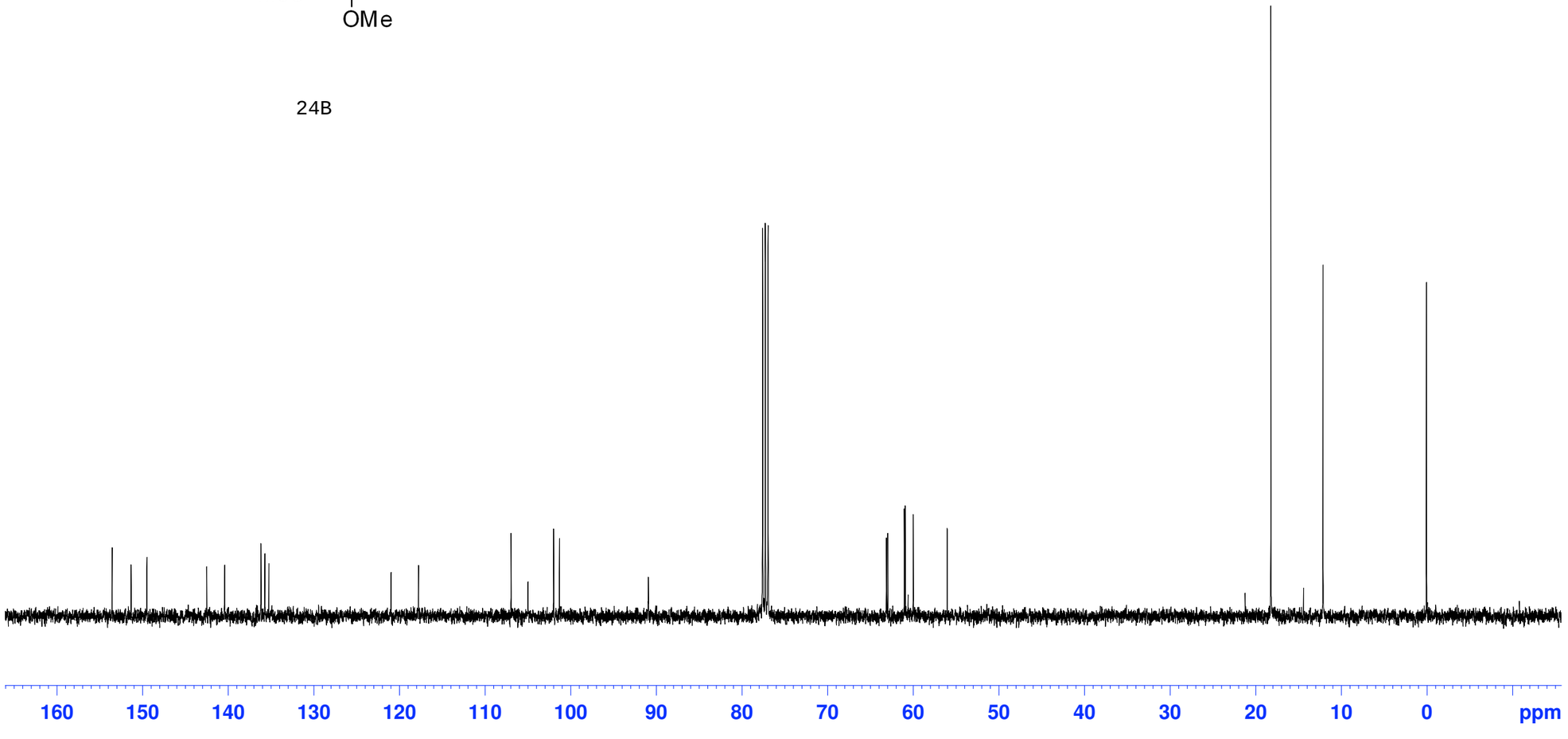



```
Current Data Parameters
NAME            Rs-2-112
EXPNO                  1
PROCNO                 1

F2 - Acquisition Parameters
Date_           20080123
Time               20.08
INSTRUM            spect
PROBHD   5 mm Multinucl
PULPROG             zg30
TD                 65536
SOLVENT            CDCl3
NS                    16
DS                     2
SWH           10330.578 Hz
FIDRES         0.157632 Hz
AQ             3.1719923 sec
RG                 456.1
DW                48.400 usec
DE                 6.00 usec
TE                300.2 K
D1            1.00000000 sec
MCREST        0.00000000 sec
MCWRK         0.01500000 sec

======== CHANNEL f1 ========
NUC1                   1H
P1                 14.80 usec
PL1                -1.00 dB
SFO1          500.0230878 MHz

F2 - Processing parameters
SI                 32768
SF            500.0200216 MHz
WDW                   EM
SSB                    0
LB                 0.30 Hz
GB                     0
PC                 1.00
```

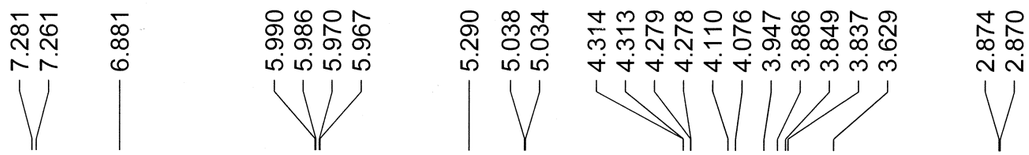


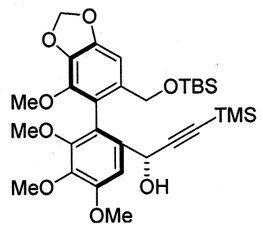


24B




```
Current Data Parameters
NAME       Rs-6-179-MP-1
EXPNO                  3
PROCNO                 1

F2 - Acquisition Parameters
Date_           20101117
Time                9.45
INSTRUM            spect
PROBHD   5 mm QNP 1H/13
PULPROG          zgpg30
TD                65536
SOLVENT           CDCl3
NS                  131
DS                    4
SWH           23980.814 Hz
FIDRES         0.365918 Hz
AQ            1.3664756 sec
RG               4597.6
DW                20.850 use
DE                 6.00 use
TE                300.2 K
D1           2.00000000 sec
d11          0.03000000 sec
DELTA        1.89999998 sec
TD0                   1

======== CHANNEL f1 =======
NUC1                 13C
P1                10.50 use
PL1                0.00 dB
SFO1        100.6228298 MHz

======== CHANNEL f2 =======
CPDPRG2         waltz16
NUC2                1H
PCPD2            80.00 use
PL2              -6.00 dB
PL12             14.56 dB
PL13             16.50 dB
SFO2        400.1316005 MHz

F2 - Processing parameters
SI                 32768
SF          100.6127496 MHz
WDW                   EM
SSB                    0
LB                1.00 Hz
GB                     0
PC                1.40
```





24C

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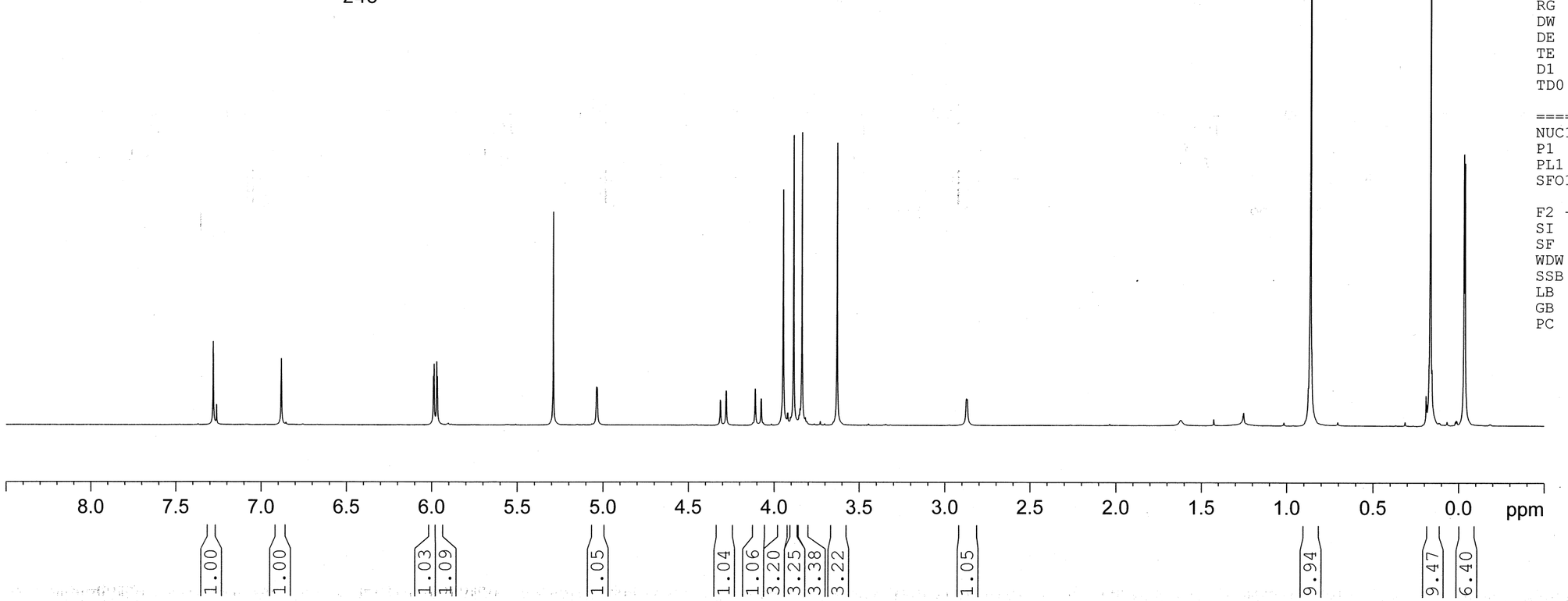
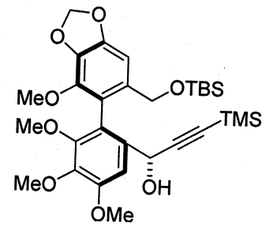
```
Current Data Parameters
NAME      Wgong-III-227-2
EXPNO                 522
PROCNO                  1

F2 - Acquisition Parameters
Date_           20110519
Time              15.21
INSTRUM           spect
PROBHD   5 mm QNP 1H/13
PULPROG            zg30
TD                65536
SOLVENT           CDCl3
NS                   16
DS                    2
SWH            8278.146 Hz
FIDRES         0.126314 Hz
AQ             3.9584243 sec
RG                71.8
DW               60.400 usec
DE                6.00 usec
TE               300.2 K
D1            1.00000000 sec
TD0                   1

======== CHANNEL f1 ========
NUC1                 1H
P1                14.50 usec
PL1                0.00 dB
SFO1         400.1324710 MHz

F2 - Processing parameters
SI                32768
SF           400.1300090 MHz
WDW                  EM
SSB                   0
LB                 0.30 Hz
GB                    0
PC                 1.00
```





24C

153.37
151.10
149.25
142.25
140.25
135.66
135.49
135.14
120.79
117.82
106.61
104.84
101.97
101.11
90.84
77.35
77.03
76.71
62.87
62.66
60.84
60.73
59.77
55.79
53.41
25.98
18.35
-0.16
-5.37
-5.41

BRUKER

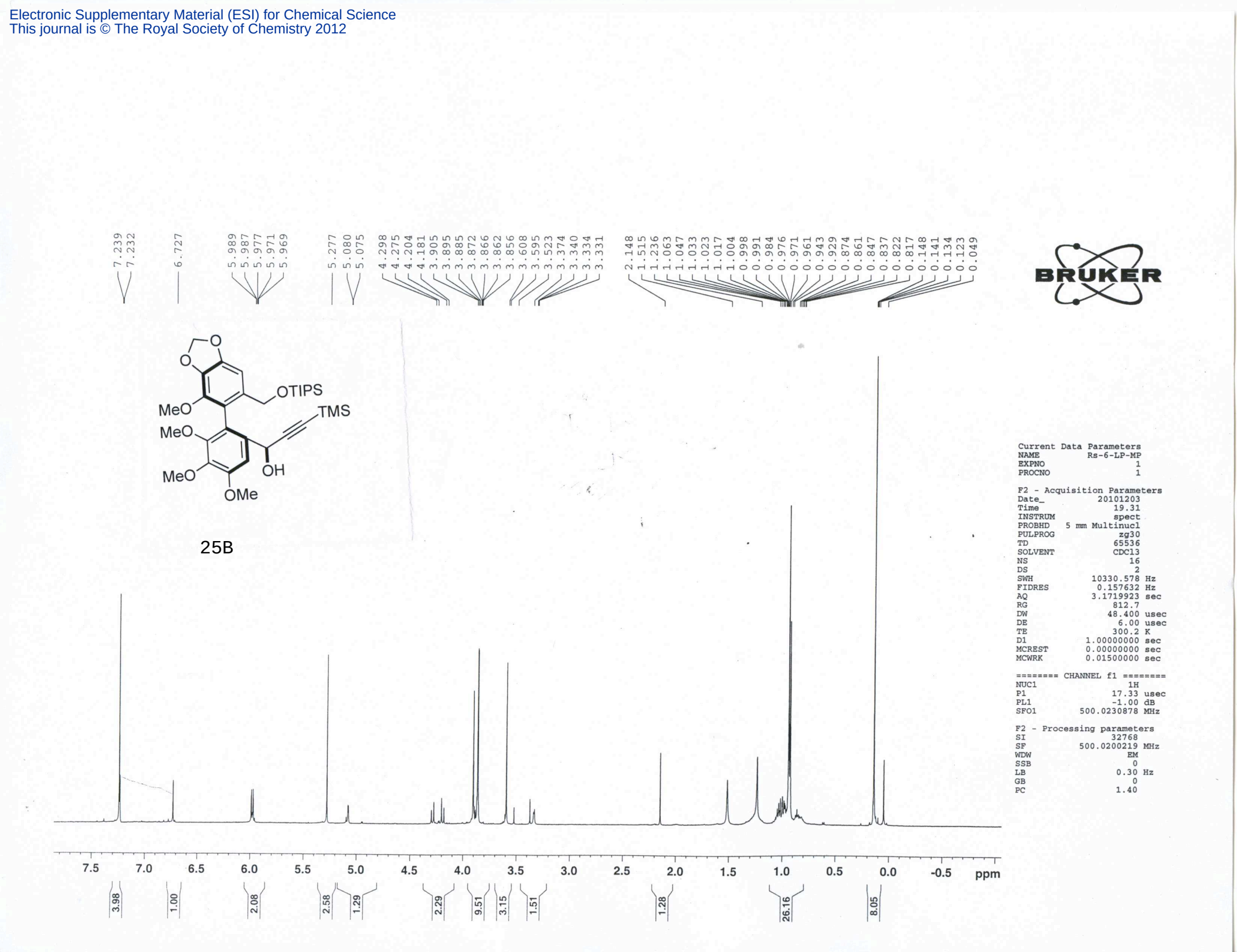
Current Data Parameters
NAME Wgong-III-227-2 C13
EXPNO 523
PROCNO 1

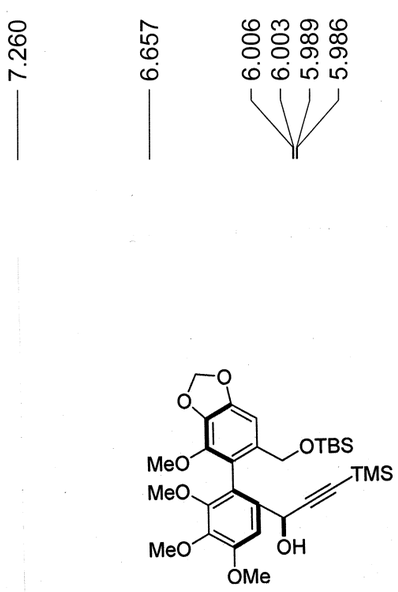
F2 - Acquisition Parameters
Date_ 20110519
Time 15.35
INSTRUM spect
PROBHD 5 mm QNP 1H/13
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 181
DS 4
SWH 23980.814 Hz
FIDRES 0.365918 Hz
AQ 1.3664756 sec
RG 1448.2
DW 20.850 usec
DE 6.00 usec
TE 300.2 K
D1 2.00000000 sec
d11 0.03000000 sec
DELTA 1.89999998 sec
TD0 1

======== CHANNEL f1 ========
NUC1 13C
P1 10.50 usec
PL1 0.00 dB
SFO1 100.6228298 MHz

======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 -6.00 dB
PL12 14.56 dB
PL13 16.50 dB
SFO2 400.1316005 MHz

F2 - Processing parameters
SI 32768
SF 100.6127690 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40





25C

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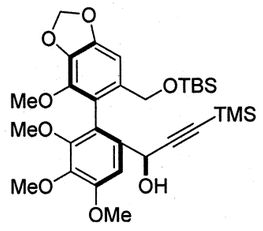
```
Current Data Parameters
NAME          Wgong-III-227-1 p
EXPNO                      532
PROCNO                       1

F2 - Acquisition Parameters
Date_               20110525
Time                   10.20
INSTRUM                spect
PROBHD   5 mm QNP 1H/13
PULPROG                 zg30
TD                     65536
SOLVENT                CDCl3
NS                        16
DS                         2
SWH                 8278.146 Hz
FIDRES              0.126314 Hz
AQ                3.9584243 sec
RG                      80.6
DW                   60.400 usec
DE                    6.00 usec
TE                   300.2 K
D1                1.00000000 sec
TD0                        1

======== CHANNEL f1 ========
NUC1                       1H
P1                    14.50 usec
PL1                    0.00 dB
SFO1          400.1324710 MHz

F2 - Processing parameters
SI                       32768
SF            400.1300093 MHz
WDW                         EM
SSB                          0
LB                    0.30 Hz
GB                           0
PC                    1.00
```

153.26
150.76
149.08
142.11
141.07
136.49
136.08
133.67
121.32
120.78
107.01
105.45
103.89
101.17
90.21
63.90
62.08
60.88
60.69
59.57
55.84
29.70
26.04
18.57
-0.09
-5.17
-5.43



25C

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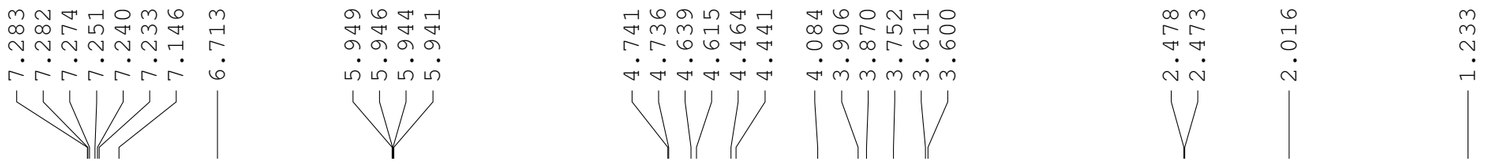
Current Data Parameters
NAME Wgong-III-227-1 p C13
EXPNO 533
PROCNO 1

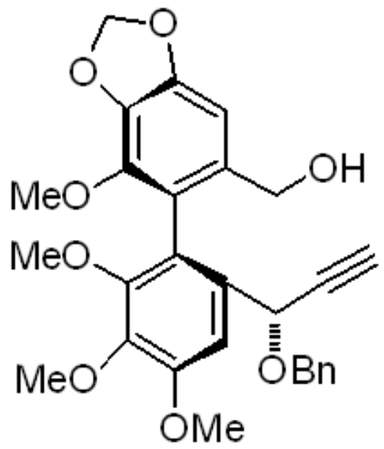
F2 - Acquisition Parameters
Date_ 20110525
Time 10.24
INSTRUM spect
PROBHD 5 mm QNP 1H/13
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 1024
DS 4
SWH 23980.814 Hz
FIDRES 0.365918 Hz
AQ 1.3664756 sec
RG 812.7
DW 20.850 usec
DE 6.00 usec
TE 300.2 K
D1 2.00000000 sec
d11 0.03000000 sec
DELTA 1.89999998 sec
TD0 1

======== CHANNEL f1 ========
NUC1 13C
P1 10.50 usec
PL1 0.00 dB
SFO1 100.6228298 MHz

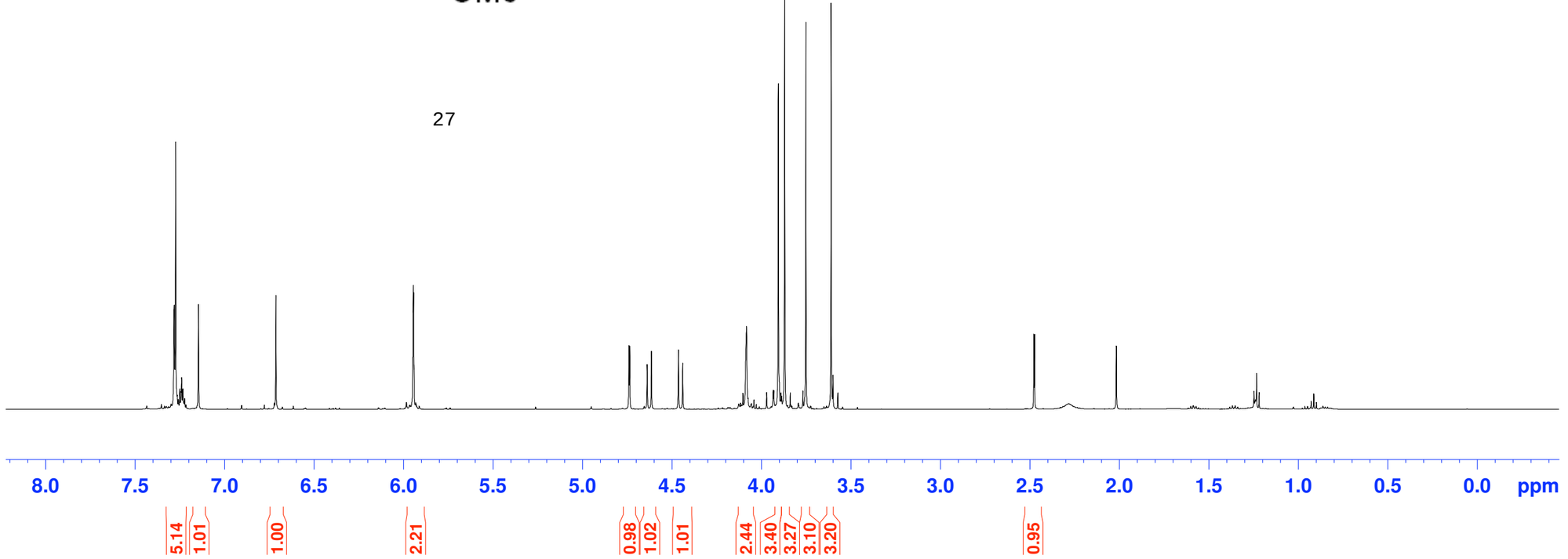
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 -6.00 dB
PL12 14.56 dB
PL13 16.50 dB
SFO2 400.1316005 MHz

F2 - Processing parameters
SI 32768
SF 100.6127690 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40



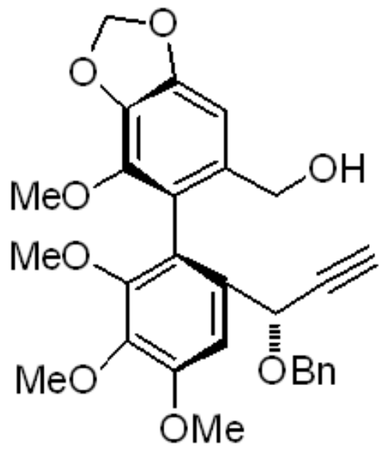


27

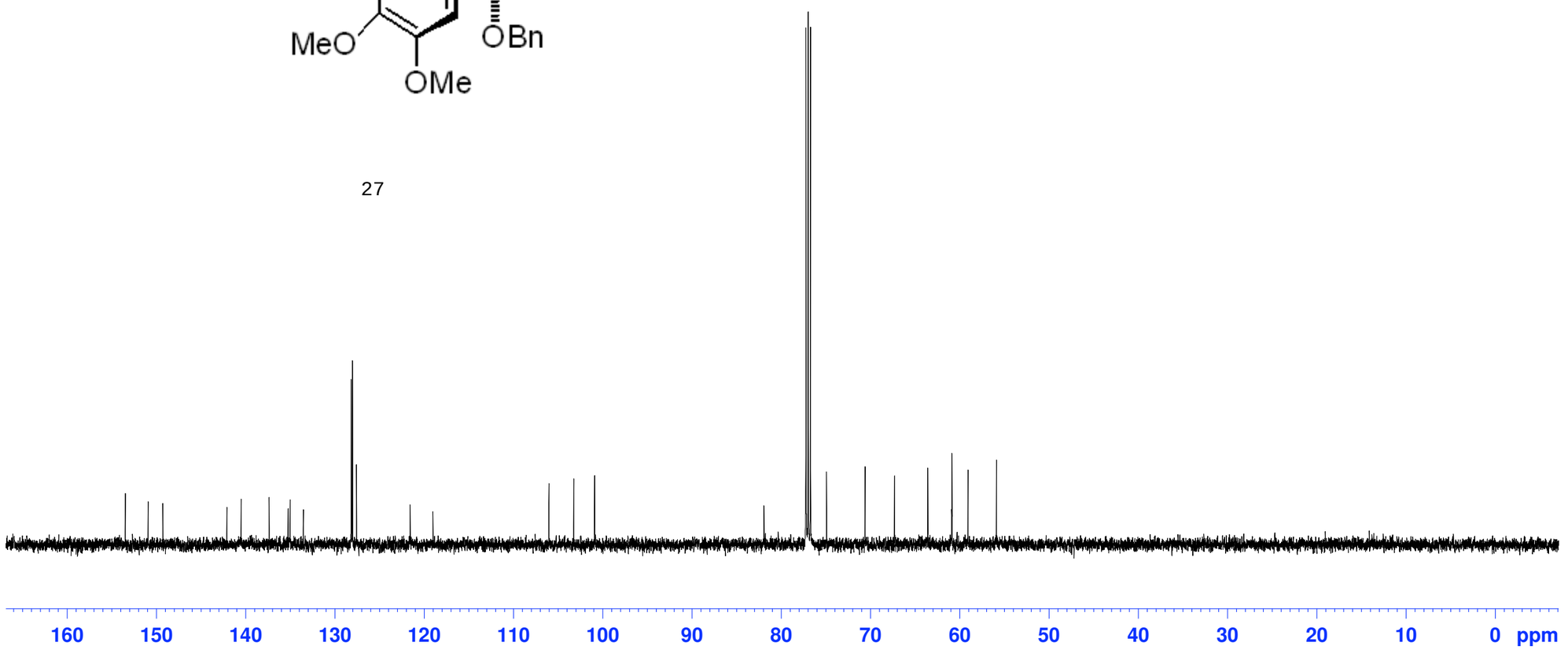



```
Current Data Parameters
NAME         Rs-6-163-MP
EXPNO                  1
PROCNO                 1

F2 - Acquisition Parameters
Date_           20101104
Time               17.58
INSTRUM            spect
PROBHD   5 mm Multinucl
PULPROG             zg30
TD                 65536
SOLVENT            CDCl3
NS                    16
DS                     2
SWH            10330.578 Hz
FIDRES          0.157632 Hz
AQ             3.1719923 sec
RG                  80.6
DW                48.400 usec
DE                 6.00 usec
TE                300.2 K
D1           1.00000000 sec
MCREST       0.00000000 sec
MCWRK        0.01500000 sec

======== CHANNEL f1 ========
NUC1                  1H
P1                17.33 usec
PL1               -1.00 dB
SFO1         500.0230878 MHz

F2 - Processing parameters
SI                 32768
SF           500.0200213 MHz
WDW                   EM
SSB                    0
LB                0.30 Hz
GB                     0
PC                 1.00
```

153.48 150.92 149.26 142.13 140.54 137.40 135.26 135.06 133.57 128.20 128.05 127.61 121.61 119.04 106.05 103.26 100.93 81.96 77.25 77.00 76.75 74.94 70.63 67.32 63.61 60.92 60.89 59.10 55.90

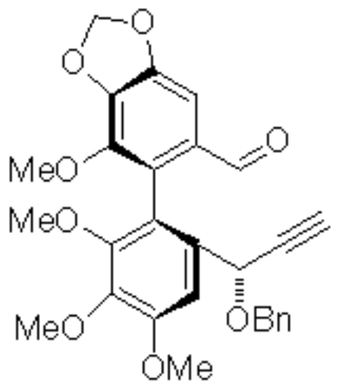


27

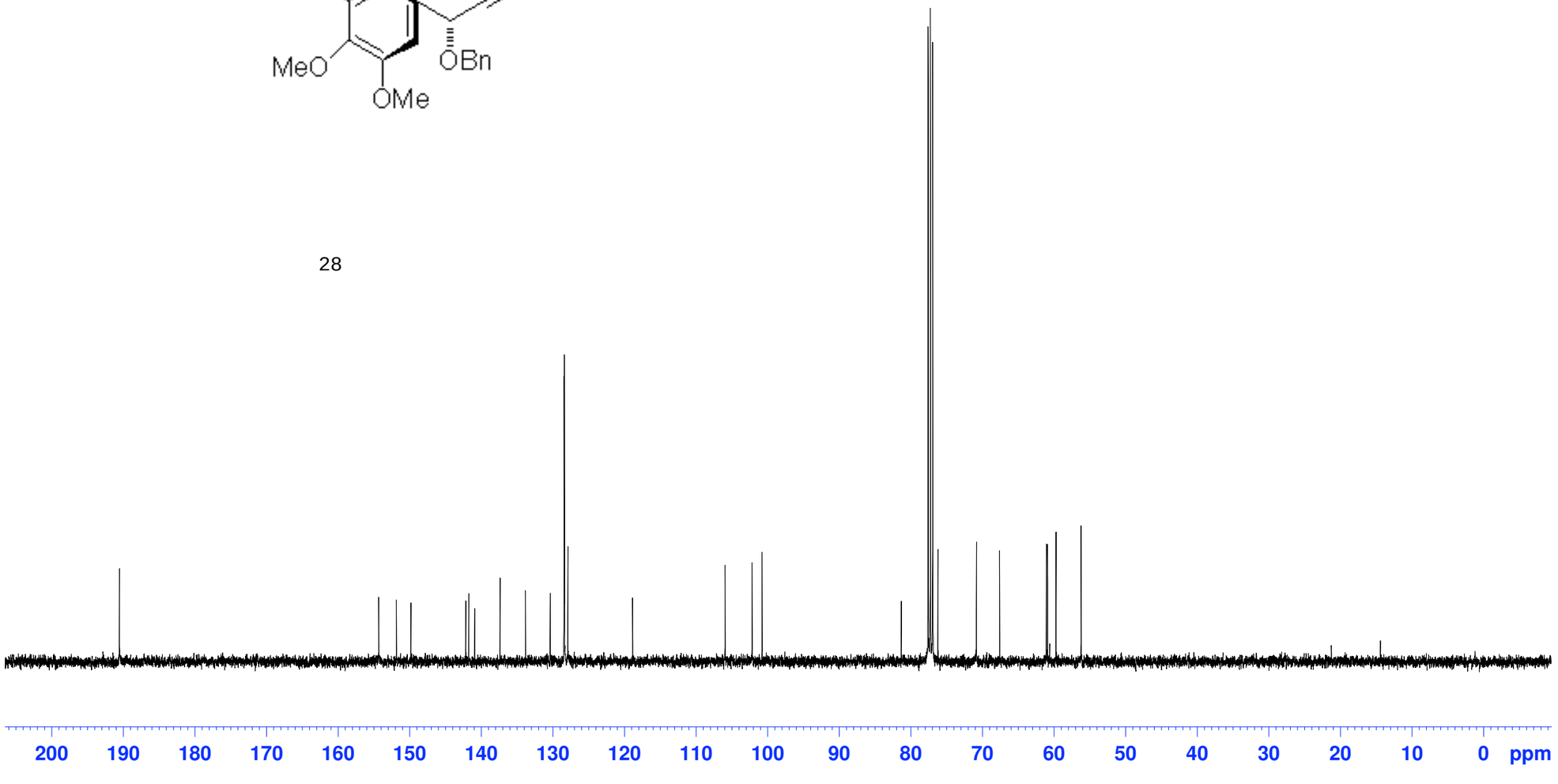



```
Current Data Parameters
NAME         Rs-6-163-MP
EXPNO                  2
PROCNO                 1

F2 - Acquisition Parameters
Date_           20101104
Time               18.01
INSTRUM            spect
PROBHD   5 mm Multinucl
PULPROG          zgpg30
TD                65536
SOLVENT           CDCl3
NS                  317
DS                    4
SWH           30030.029 Hz
FIDRES         0.458222 Hz
AQ            1.0912244 sec
RG                 3251
DW               16.650 usec
DE               12.00 usec
TE                300.2 K
D1           2.00000000 sec
d11          0.03000000 sec
DELTA        1.89999998 sec
MCREST       0.00000000 sec
MCWRK        0.01500000 sec

======== CHANNEL f1 ========
NUC1                13C
P1                27.00 usec
PL1                1.00 dB
SFO1        125.7427020 MHz

======== CHANNEL f2 ========
CPDPRG2         waltz16
NUC2                1H
PCPD2             68.00 usec
PL2               -1.00 dB
PL12              10.87 dB
PL13              22.50 dB
SFO2        500.0220001 MHz

F2 - Processing parameters
SI                32768
SF          125.7301377 MHz
WDW                EM
SSB                 0
LB                1.00 Hz
GB                  0
PC                1.40
```

190.49
154.32
151.84
149.81
142.15
141.73
140.90
137.37
133.81
130.35
128.43
128.38
127.89
118.84
105.93
102.16
100.77
81.31
77.55
77.23
76.92
76.19
70.81
67.57
61.04
60.87
59.70
56.19



28



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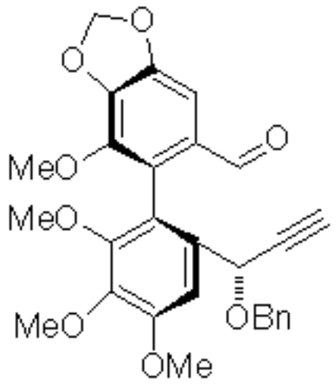
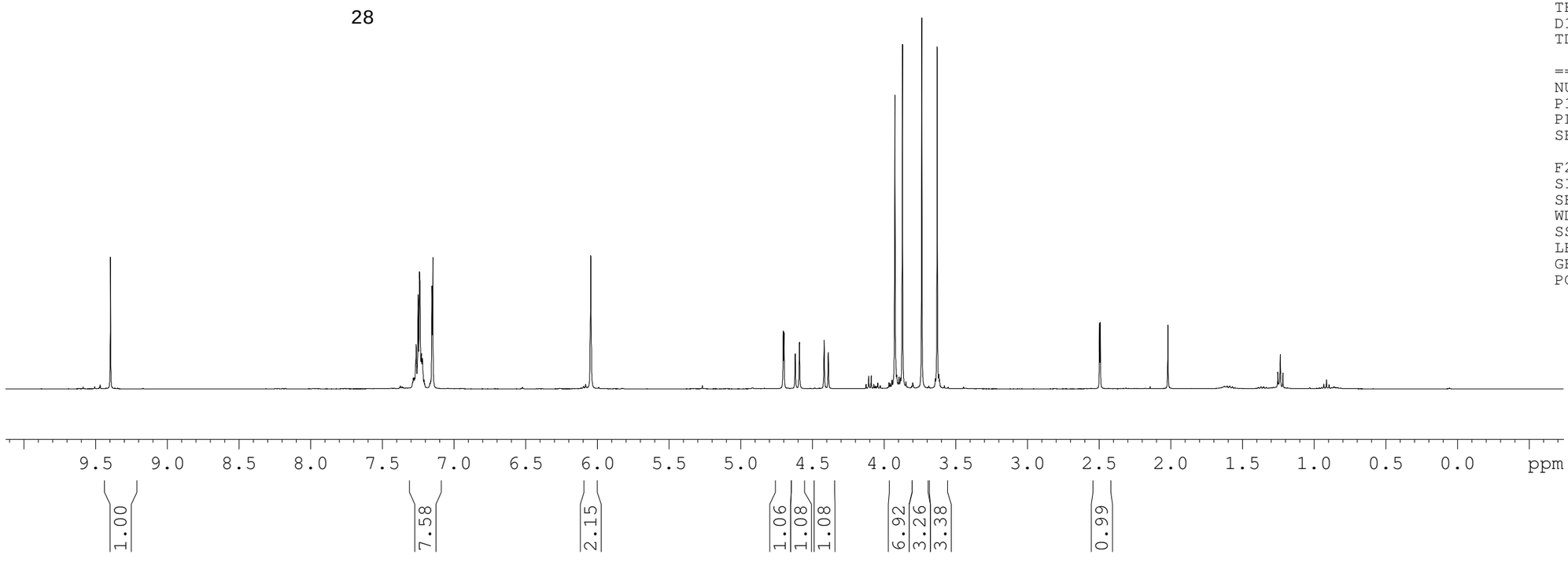
```
Current Data Parameters
NAME            Rs-6-164
EXPNO                  2
PROCNO                 1

F2 - Acquisition Parameters
Date_           20101108
Time               10.10
INSTRUM            spect
PROBHD   5 mm QNP 1H/13
PULPROG          zgpg30
TD                65536
SOLVENT           CDCl3
NS                  374
DS                    4
SWH           23980.814 Hz
FIDRES         0.365918 Hz
AQ            1.3664756 sec
RG               5792.6
DW                20.850 use
DE                 6.00 use
TE                300.2 K
D1           2.00000000 sec
d11          0.03000000 sec
DELTA        1.89999998 sec
TD0                   1

======== CHANNEL f1 =======
NUC1                 13C
P1                10.50 use
PL1                0.00 dB
SFO1        100.6228298 MHz

======== CHANNEL f2 =======
CPDPRG2         waltz16
NUC2                 1H
PCPD2             80.00 use
PL2               -6.00 dB
PL12              14.56 dB
PL13              16.50 dB
SFO2        400.1316005 MHz

F2 - Processing parameters
SI                32768
SF          100.6127508 MHz
WDW                  EM
SSB                   0
LB                 1.00 Hz
GB                    0
PC                 1.40
```

9.39 | 7.26 7.25 7.24 7.24 7.23 7.22 7.15 7.15 | 6.05 | 4.70 4.70 4.62 4.59 4.42 4.39 | 3.92 3.87 3.74 3.63 | 2.50 2.49 | 2.02 | 1.23



28



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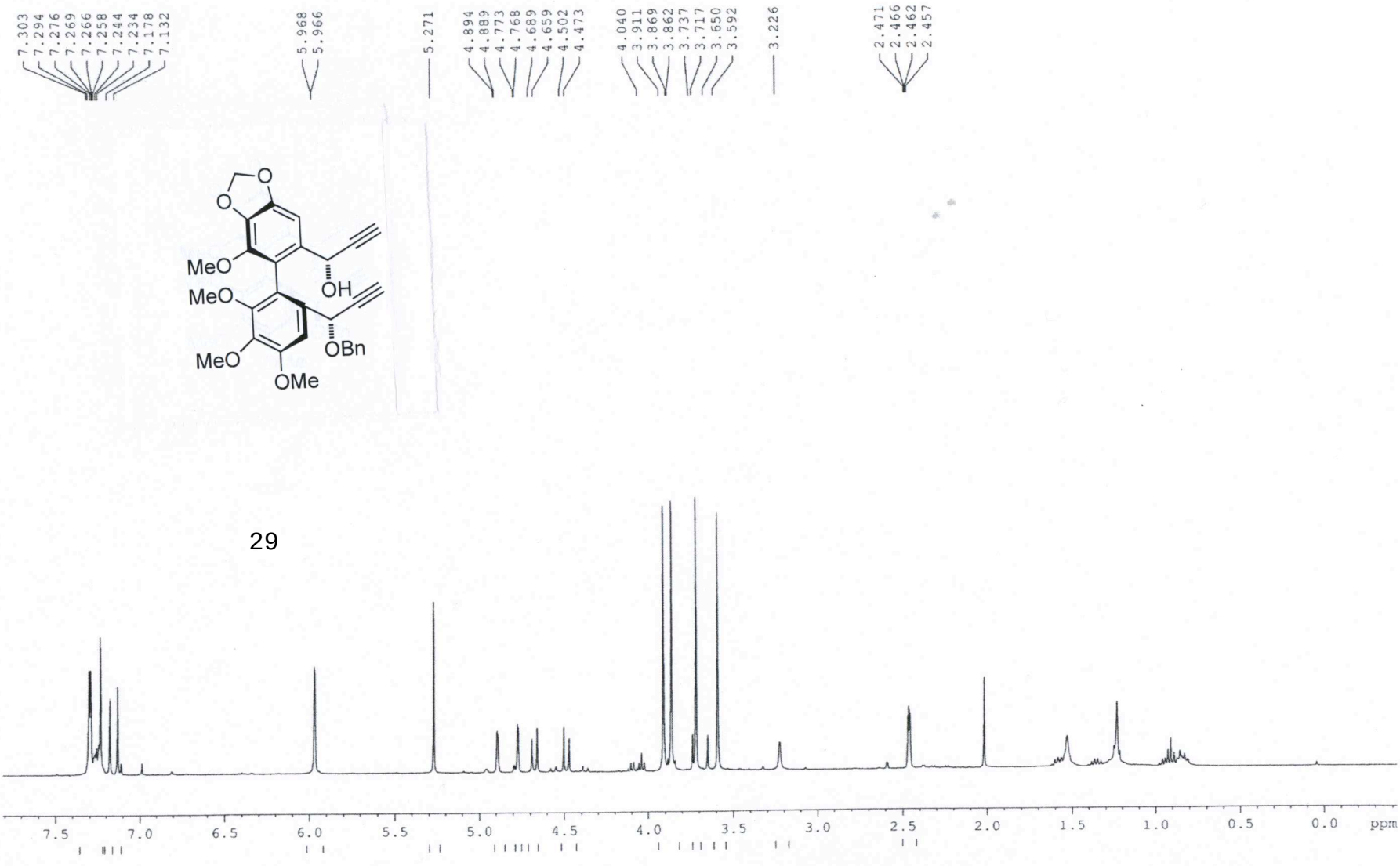
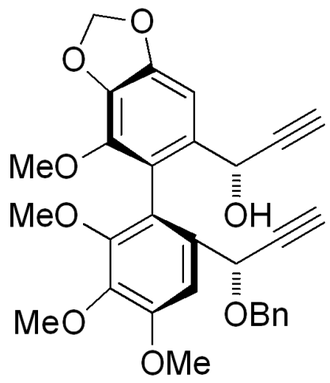
```
Current Data Parameters
NAME                  Rs-6-164
EXPNO                        1
PROCNO                       1

F2 - Acquisition Parameters
Date_                20101108
Time                    10.03
INSTRUM                 spect
PROBHD   5 mm QNP 1H/13
PULPROG                  zg30
TD                      65536
SOLVENT                 CDCl3
NS                         16
DS                          2
SWH                  8278.146 Hz
FIDRES               0.126314 Hz
AQ                   3.9584243 sec
RG                        90.5
DW                    60.400 usec
DE                     6.00 usec
TE                     300.2 K
D1                1.00000000 sec
TD0                          1

======== CHANNEL f1 ========
NUC1                        1H
P1                    13.00 usec
PL1                    0.00 dB
SFO1             400.1324710 MHz

F2 - Processing parameters
SI                       32768
SF               400.1300168 MHz
WDW                         EM
SSB                          0
LB                     0.30 Hz
GB                           0
PC                        1.00
```



153.97 150.93 149.90 142.28 140.29 137.69 136.10 135.27 134.55 128.52 128.44 128.26 127.93 120.74 119.15 106.06 102.50 101.44 83.53 81.51 77.54 77.43 77.22 76.91 75.60 73.87 70.95 67.59 63.12 61.51 61.31 59.39 56.23 53.62



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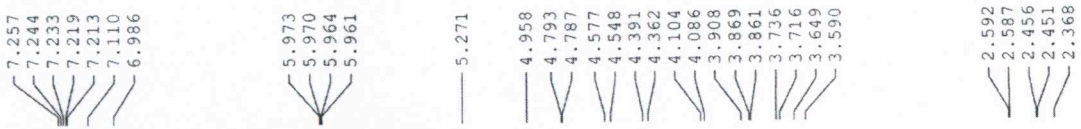
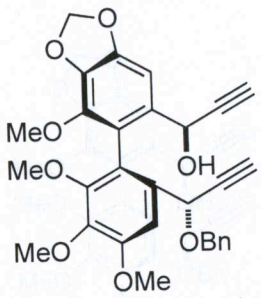
```
Current Data Parameters
NAME          Rs-6-185-MP
EXPNO                   2
PROCNO                  1

F2 - Acquisition Parameters
Date_            20101120
Time                16.33
INSTRUM             spect
PROBHD   5 mm QNP 1H/13
PULPROG           zgpg30
TD                 65536
SOLVENT            CDCl3
NS                  5036
DS                     4
SWH            23980.814 Hz
FIDRES          0.365918 Hz
AQ             1.3664756 sec
RG                  4096
DW                20.850 use
DE                 6.00 use
TE                300.2 K
D1           2.00000000 sec
d11          0.03000000 sec
DELTA        1.89999998 sec
TD0                    1

======== CHANNEL f1 =======
NUC1                  13C
P1                 10.50 use
PL1                 0.00 dB
SFO1        100.6228298 MHz

======== CHANNEL f2 =======
CPDPRG2          waltz16
NUC2                  1H
PCPD2              80.00 use
PL2                -6.00 dB
PL12               14.56 dB
PL13               16.50 dB
SFO2        400.1316005 MHz

F2 - Processing parameters
SI                 32768
SF          100.6127480 MHz
WDW                   EM
SSB                    0
LB                 1.00 Hz
GB                     0
PC                 1.40
```





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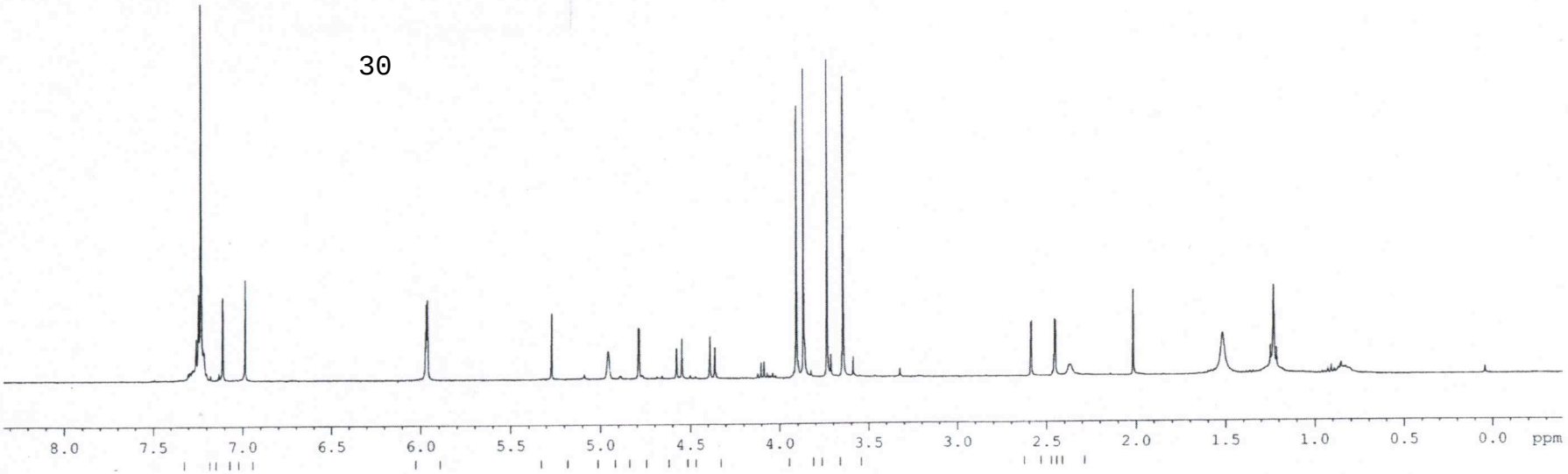
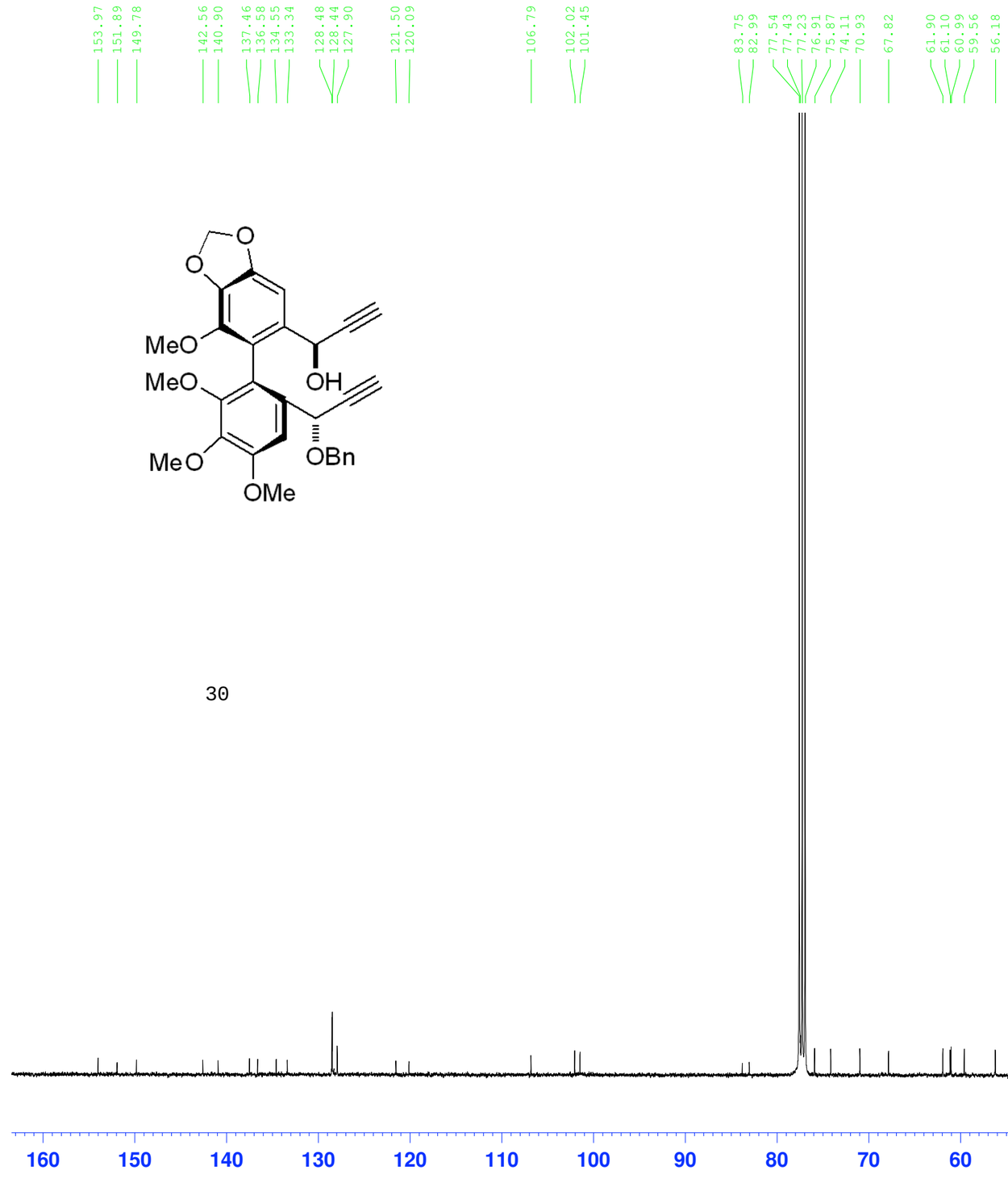




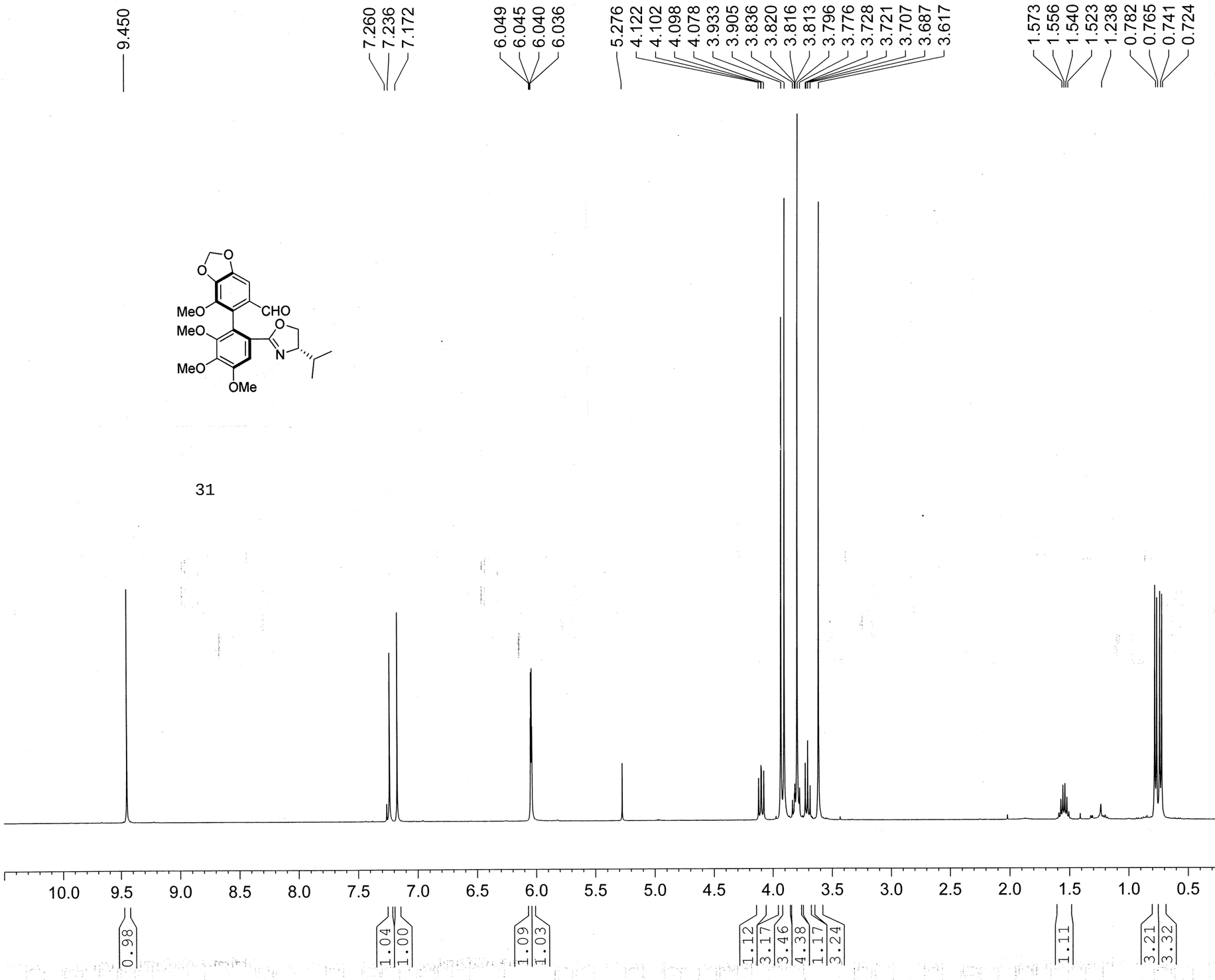
Current Data Parameters
NAME Rs-6-185-LP
EXPNO 2
PROCNO 1

F2 - Acquisition Parameters
Date_ 20101120
Time 21.29
INSTRUM spect
PROBHD 5 mm QNP 1H/13
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 11154
DS 4
SWH 23980.814 Hz
FIDRES 0.365918 Hz
AQ 1.3664756 sec
RG 5792.6
DW 20.850 use
DE 6.00 use
TE 300.2 K
D1 2.00000000 sec
d11 0.03000000 sec
DELTA 1.89999998 sec
TD0 1

======== CHANNEL f1 =======
NUC1 13C
P1 10.50 use
PL1 0.00 dB
SFO1 100.6228298 MHz

======== CHANNEL f2 =======
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 use
PL2 -6.00 dB
PL12 14.56 dB
PL13 16.50 dB
SFO2 400.1316005 MHz

F2 - Processing parameters
SI 32768
SF 100.6127477 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40



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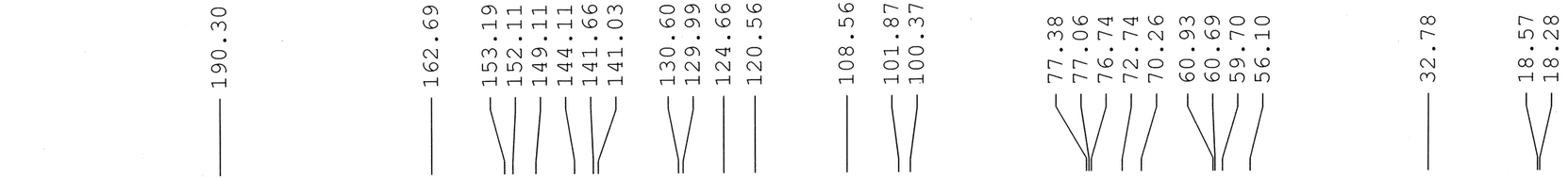
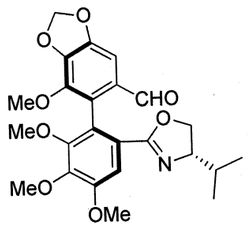
```
Current Data Parameters
NAME      Wgong-III-224 p
EXPNO                504
PROCNO                 1

F2 - Acquisition Parameters
Date_           20110510
Time               12.19
INSTRUM            spect
PROBHD   5 mm QNP 1H/13
PULPROG             zg30
TD                 65536
SOLVENT            CDCl3
NS                    16
DS                     2
SWH             8278.146 Hz
FIDRES          0.126314 Hz
AQ              3.9584243 sec
RG                  71.8
DW                60.400 usec
DE                 6.00 usec
TE                300.2 K
D1             1.00000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                  1H
P1                 14.50 usec
PL1                 0.00 dB
SFO1          400.1324710 MHz

F2 - Processing parameters
SI                 32768
SF            400.1300092 MHz
WDW                   EM
SSB                    0
LB                  0.30 Hz
GB                     0
PC                  1.00
```





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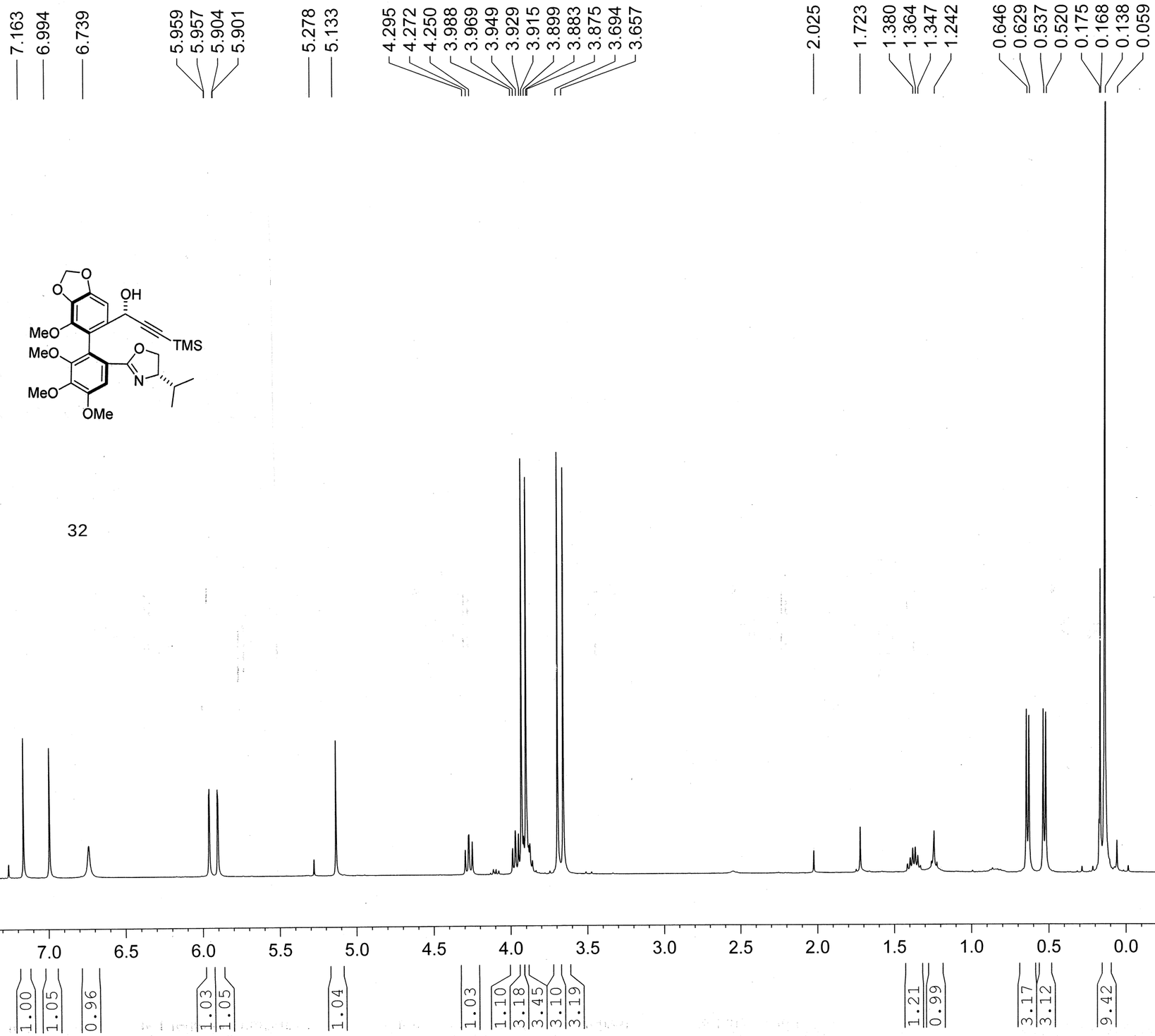
Current Data Parameters
NAME Wgong-III-224 C13
EXPNO 505
PROCNO 1

F2 - Acquisition Parameters
Date_ 20110510
Time 12.24
INSTRUM spect
PROBHD 5 mm QNP 1H/13
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 135
DS 4
SWH 23980.814 Hz
FIDRES 0.365918 Hz
AQ 1.3664756 sec
RG 912.3
DW 20.850 usec
DE 6.00 usec
TE 300.2 K
D1 2.00000000 sec
d11 0.03000000 sec
DELTA 1.89999998 sec
TD0 1

======== CHANNEL f1 ========
NUC1 13C
P1 10.50 usec
PL1 0.00 dB
SFO1 100.6228298 MHz

======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 -6.00 dB
PL12 14.56 dB
PL13 16.50 dB
SFO2 400.1316005 MHz

F2 - Processing parameters
SI 32768
SF 100.6127690 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40



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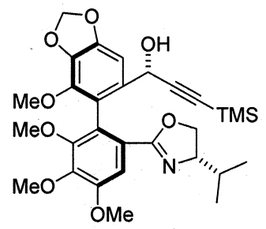
```
Current Data Parameters
NAME       Wgong-III-229
EXPNO                479
PROCNO                 1

F2 - Acquisition Parameters
Date_           20110506
Time               12.06
INSTRUM            spect
PROBHD   5 mm QNP 1H/13
PULPROG            zg30
TD                65536
SOLVENT           CDCl3
NS                   16
DS                    2
SWH           8278.146 Hz
FIDRES        0.126314 Hz
AQ            3.9584243 sec
RG                   57
DW               60.400 usec
DE                6.00 usec
TE               300.2 K
D1            1.00000000 sec
TD0                   1

======== CHANNEL f1 ========
NUC1                 1H
P1               14.50 usec
PL1               0.00 dB
SFO1        400.1324710 MHz

F2 - Processing parameters
SI                32768
SF          400.1300099 MHz
WDW                  EM
SSB                   0
LB                0.30 Hz
GB                    0
PC                 1.00
```



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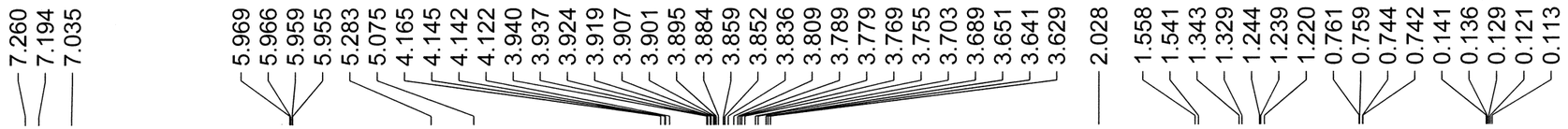
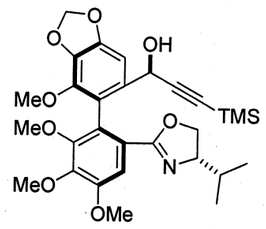
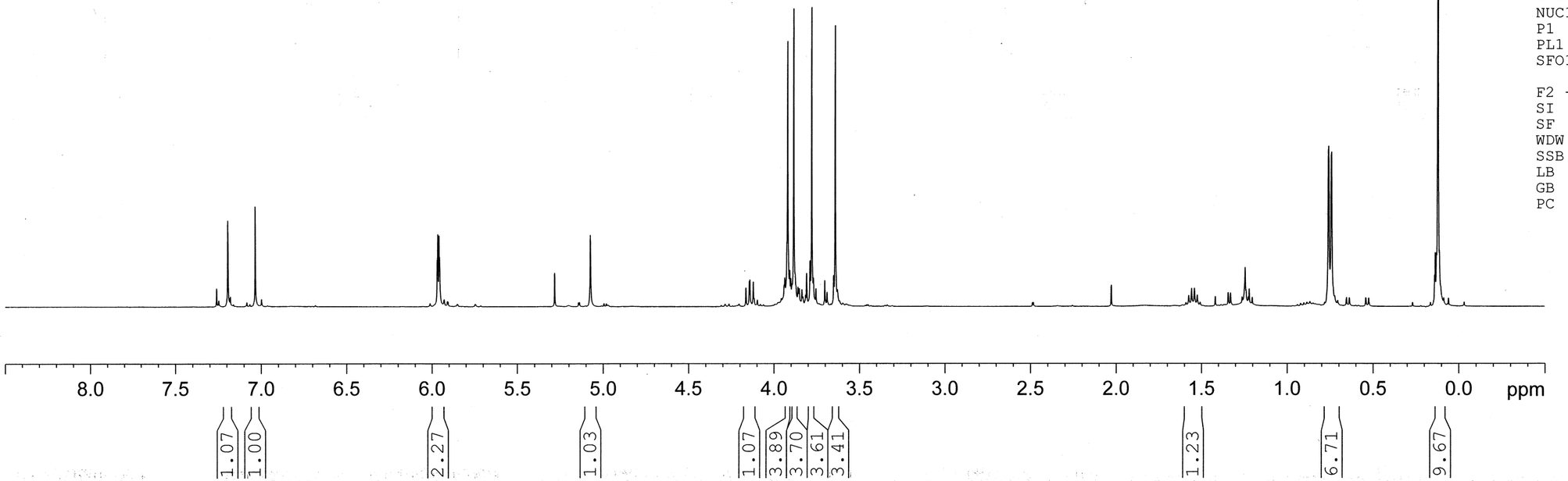
BRUKER

```
Current Data Parameters
NAME      Wgong-III-229 C13
EXPNO                  480
PROCNO                   1

F2 - Acquisition Parameters
Date_           20110506
Time               12.14
INSTRUM            spect
PROBHD   5 mm QNP 1H/13
PULPROG          zgpg30
TD                65536
SOLVENT            CDCl3
NS                   120
DS                     4
SWH            23980.814 Hz
FIDRES          0.365918 Hz
AQ              1.3664756 sec
RG                1149.4
DW                20.850 usec
DE                 6.00 usec
TE                 300.2 K
D1             2.00000000 sec
d11            0.03000000 sec
DELTA          1.89999998 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                  13C
P1                 10.50 usec
PL1                 0.00 dB
SFO1          100.6228298 MHz

======== CHANNEL f2 ========
CPDPRG2           waltz16
NUC2                  1H
PCPD2              80.00 usec
PL2                -6.00 dB
PL12               14.56 dB
PL13               16.50 dB
SFO2          400.1316005 MHz

F2 - Processing parameters
SI                 32768
SF            100.6127690 MHz
WDW                   EM
SSB                    0
LB                  1.00 Hz
GB                     0
PC                  1.40
```





33



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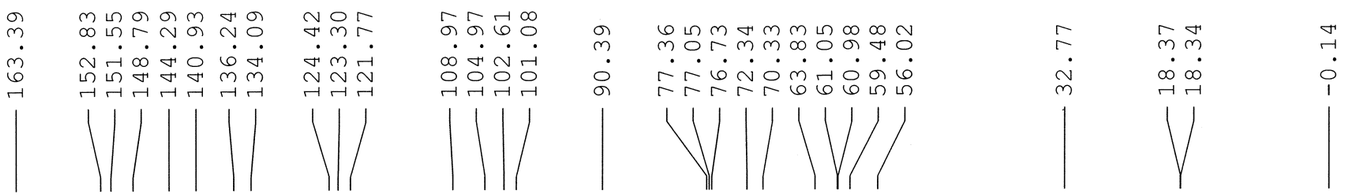
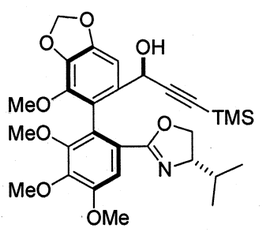
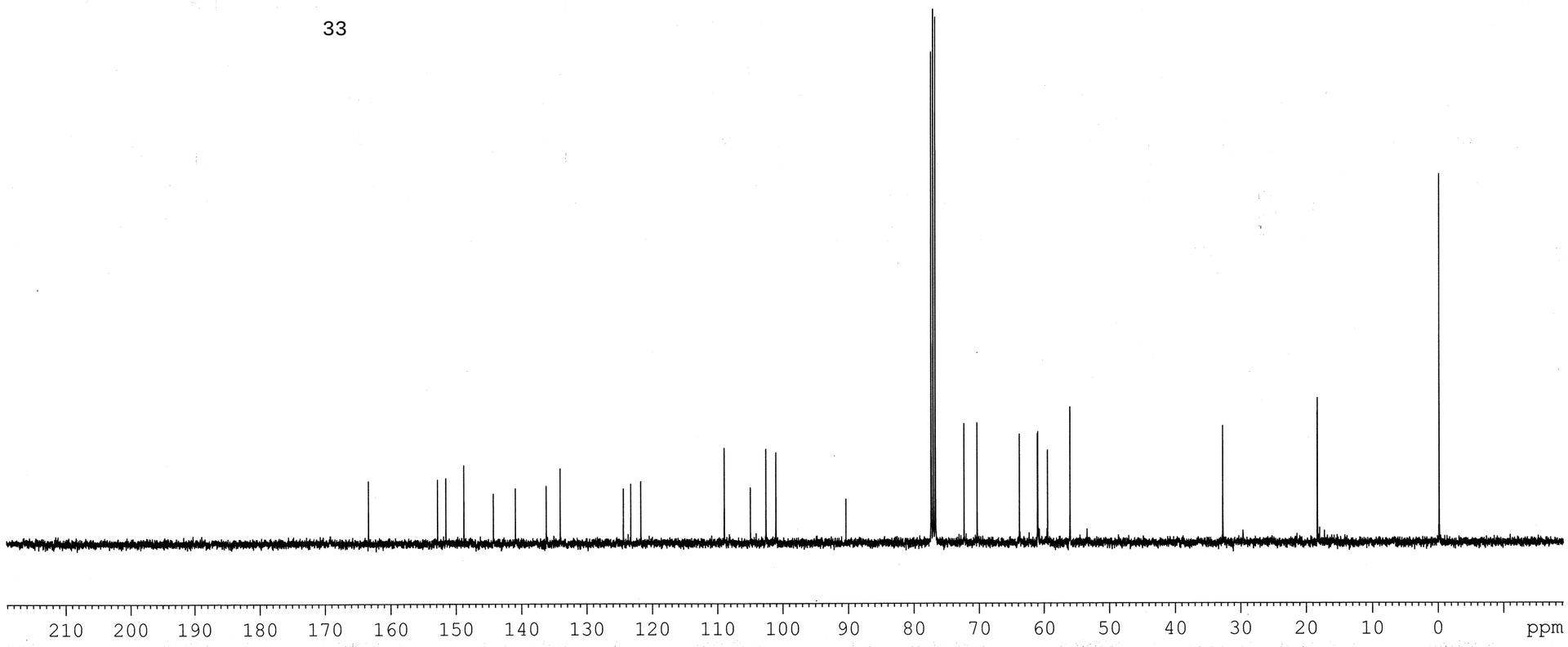
Department of Chemistry
NMR Facility

```
Current Data Parameters
NAME      Wgong-III-240-2 p
EXPNO                  530
PROCNO                   1

F2 - Acquisition Parameters
Date_           20110524
Time              14.39
INSTRUM           spect
PROBHD   5 mm QNP 1H/13
PULPROG            zg30
TD                65536
SOLVENT           CDCl3
NS                   16
DS                    2
SWH            8278.146 Hz
FIDRES         0.126314 Hz
AQ             3.9584243 sec
RG                   64
DW                60.400 usec
DE                 6.00 usec
TE                300.2 K
D1             1.00000000 sec
TD0                  1

======== CHANNEL f1 ========
NUC1                 1H
P1                14.50 usec
PL1                0.00 dB
SFO1         400.1324710 MHz

F2 - Processing parameters
SI                32768
SF           400.1300095 MHz
WDW                  EM
SSB                   0
LB                 0.30 Hz
GB                    0
PC                 1.00
```





33



BRUKER

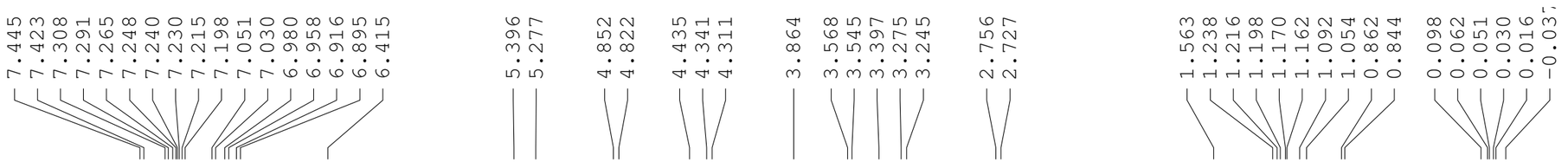
Current Data Parameters
NAME Wgong-III-240-2 p C13
EXPNO 531
PROCNO 1

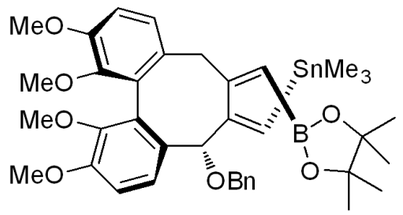
F2 - Acquisition Parameters
Date_ 20110524
Time 14.47
INSTRUM spect
PROBHD 5 mm QNP 1H/13
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 337
DS 4
SWH 23980.814 Hz
FIDRES 0.365918 Hz
AQ 1.3664756 sec
RG 1625.5
DW 20.850 usec
DE 6.00 usec
TE 300.2 K
D1 2.00000000 sec
d11 0.03000000 sec
DELTA 1.89999998 sec
TD0 1

======== CHANNEL f1 ========
NUC1 13C
P1 10.50 usec
PL1 0.00 dB
SFO1 100.6228298 MHz

======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 -6.00 dB
PL12 14.56 dB
PL13 16.50 dB
SFO2 400.1316005 MHz

F2 - Processing parameters
SI 32768
SF 100.6127690 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40





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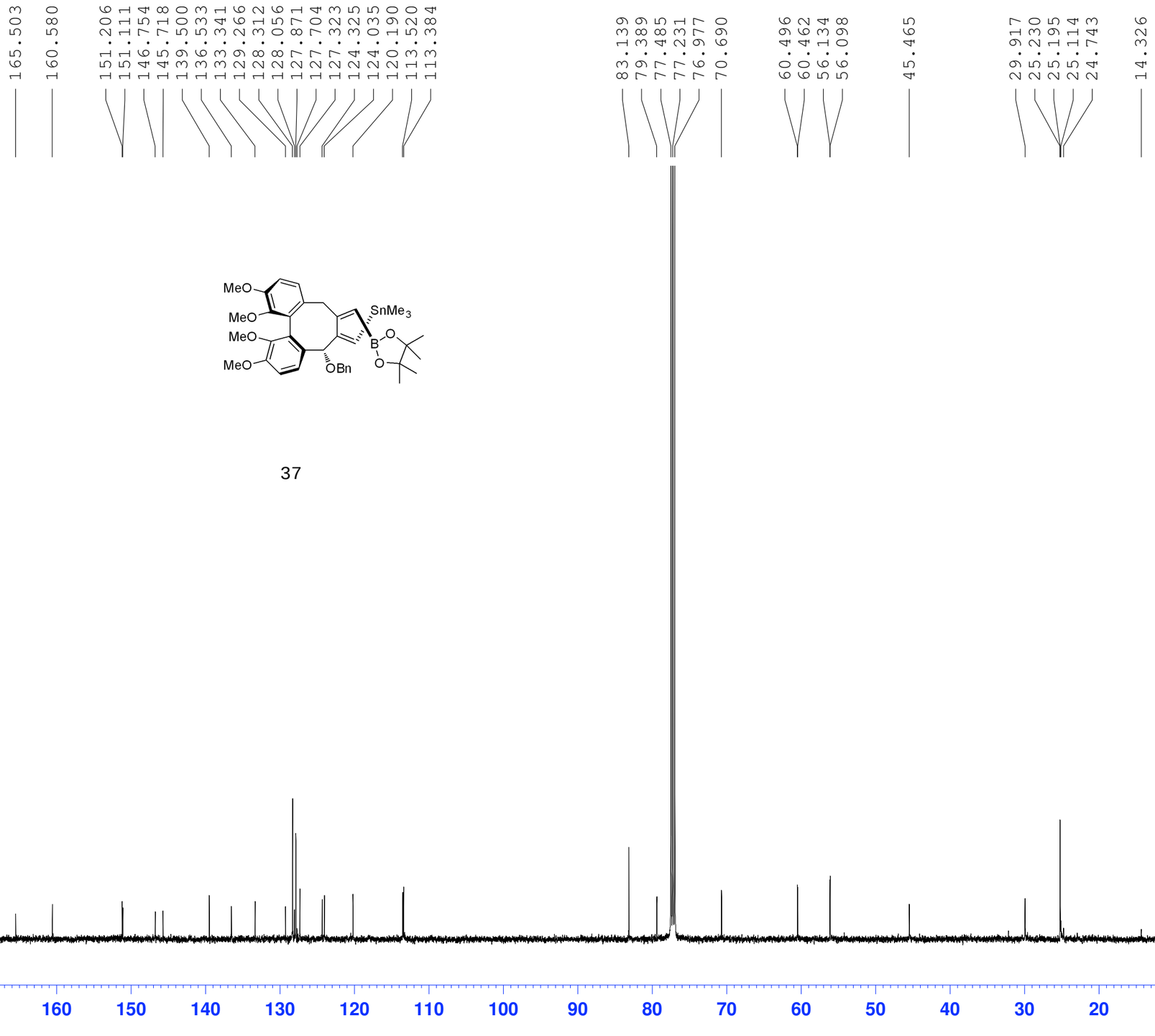
The Ohio State University
Department of Chemistry
NMR Facility
400MHz – 0083

```
Current Data Parameters
NAME      Rs-1-139-1cdcl3
EXPNO                   1
PROCNO                  1

F2 - Acquisition Parameters
Date_           20070713
Time               20.24
INSTRUM            spect
PROBHD   5 mm QNP 1H/13
PULPROG             zg30
TD                 65536
SOLVENT            CDCl3
NS                    16
DS                     2
SWH             8278.146 Hz
FIDRES          0.126314 Hz
AQ              3.9584243 sec
RG                 287.4
DW                60.400 use
DE                 6.00 use
TE                300.2 K
D1           1.00000000 sec
MCREST       0.00000000 sec
MCWRK        0.01500000 sec

======== CHANNEL f1 =======
NUC1                   1H
P1                 13.00 use
PL1                 0.00 dB
SFO1         400.1324710 MHz

F2 - Processing parameters
SI                 32768
SF          400.1300179 MHz
WDW                   EM
SSB                    0
LB                  0.30 Hz
GB                     0
PC                  1.00
```



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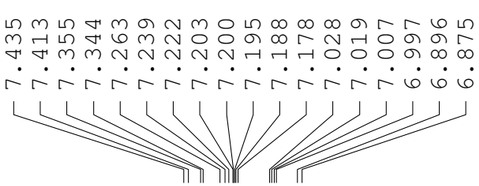
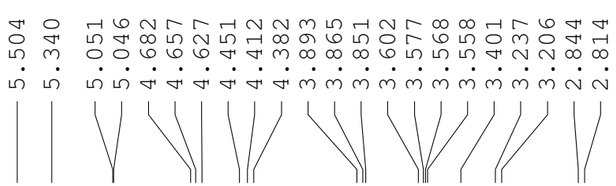
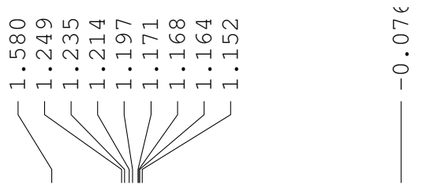
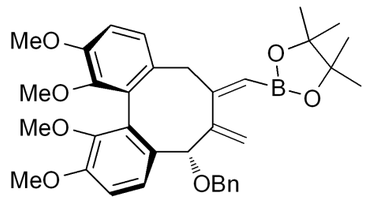
```
Current Data Parameters
NAME       Ra-1-142-C13
EXPNO                 1
PROCNO                1

F2 - Acquisition Parameters
Date_          20070716
Time              16.21
INSTRUM           spect
PROBHD   5 mm Multinucl
PULPROG          zgpg30
TD                65536
SOLVENT           CDCl3
NS                 1083
DS                    4
SWH           30030.029 Hz
FIDRES         0.458222 Hz
AQ            1.0912244 sec
RG               2896.3
DW               16.650 usec
DE                6.00 usec
TE               300.2 K
D1           2.00000000 sec
d11          0.03000000 sec
DELTA        1.89999998 sec
MCREST       0.00000000 sec
MCWRK        0.01500000 sec

======== CHANNEL f1 ========
NUC1                 13C
P1                13.00 usec
PL1                3.00 dB
SFO1         125.7427020 MHz

======== CHANNEL f2 ========
CPDPRG2          waltz16
NUC2                 1H
PCPD2            100.00 usec
PL2               -1.00 dB
PL12              18.80 dB
PL13              22.50 dB
SFO2         500.0220001 MHz

F2 - Processing parameters
SI                32768
SF           125.7301023 MHz
WDW                  EM
SSB                   0
LB                 1.00 Hz
GB                    0
PC                 1.00
```









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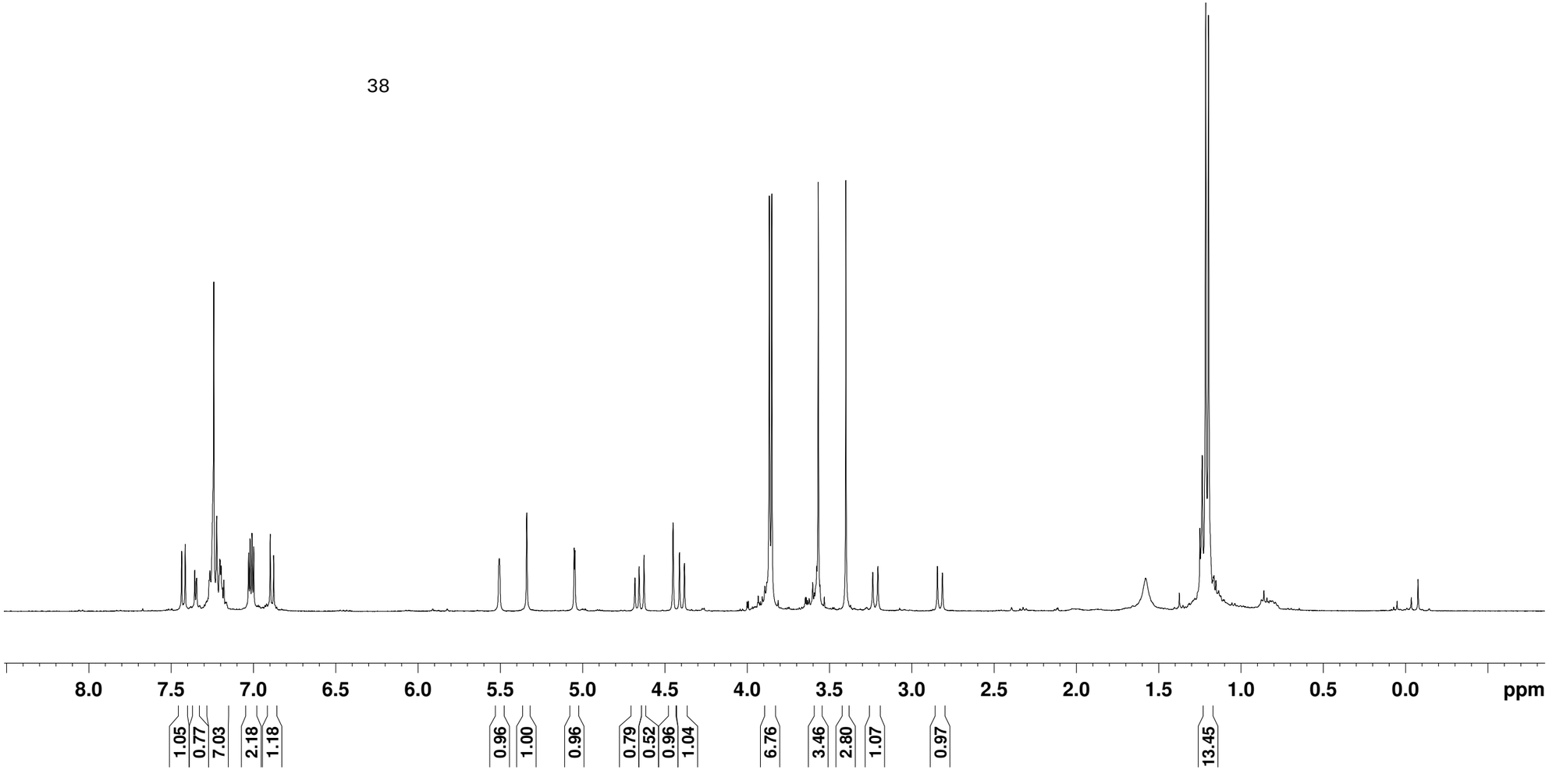
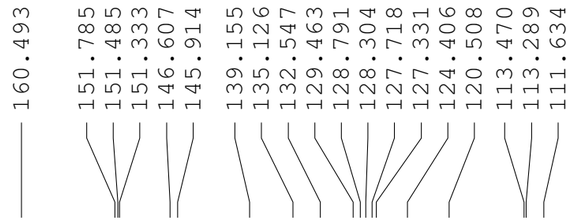
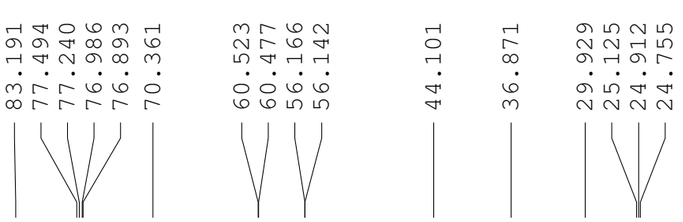
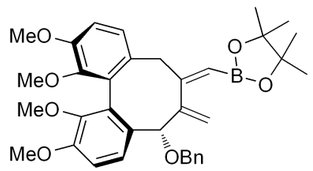
The Ohio State University
Department of Chemistry
NMR Facility
400MHz – 0083

```
Current Data Parameters
NAME            Rs-1-256
EXPNO                  1
PROCNO                 1

F2 - Acquisition Parameters
Date_           20071005
Time                8.23
INSTRUM            spect
PROBHD   5 mm QNP 1H/13
PULPROG             zg30
TD                 65536
SOLVENT            CDCl3
NS                    16
DS                     2
SWH             8278.146 Hz
FIDRES          0.126314 Hz
AQ              3.9584243 sec
RG                   181
DW                60.400 use
DE                 6.00 use
TE                  0.0 K
D1           1.00000000 sec
MCREST       0.00000000 sec
MCWRK        0.01500000 sec

======== CHANNEL f1 =======
NUC1                  1H
P1                13.00 use
PL1                0.00 dB
SFO1         400.1324710 MHz

F2 - Processing parameters
SI                 32768
SF           400.1300181 MHz
WDW                   EM
SSB                    0
LB                0.30 Hz
GB                     0
PC                1.00
```









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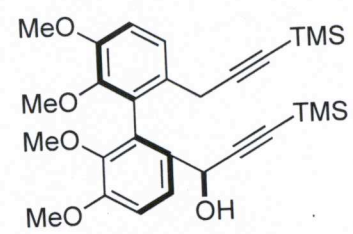
```
Current Data Parameters
NAME       Ra-1-143-C13
EXPNO                 1
PROCNO                1

F2 - Acquisition Parameters
Date_          20070716
Time              17.24
INSTRUM           spect
PROBHD   5 mm Multinucl
PULPROG          zgpg30
TD                65536
SOLVENT           CDCl3
NS                 1695
DS                    4
SWH           30030.029 Hz
FIDRES         0.458222 Hz
AQ            1.0912244 sec
RG               2896.3
DW               16.650 usec
DE                6.00 usec
TE               300.2 K
D1            2.00000000 sec
d11           0.03000000 sec
DELTA         1.89999998 sec
MCREST        0.00000000 sec
MCWRK         0.01500000 sec

======== CHANNEL f1 ========
NUC1                 13C
P1                 13.00 usec
PL1                 3.00 dB
SFO1         125.7427020 MHz

======== CHANNEL f2 ========
CPDPRG2          waltz16
NUC2                 1H
PCPD2             100.00 usec
PL2                -1.00 dB
PL12               18.80 dB
PL13               22.50 dB
SFO2         500.0220001 MHz

F2 - Processing parameters
SI                32768
SF           125.7301005 MHz
WDW                  EM
SSB                   0
LB                 1.00 Hz
GB                    0
PC                 1.40
```



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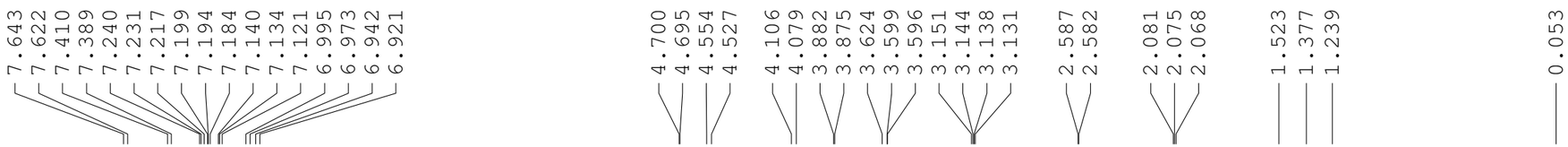
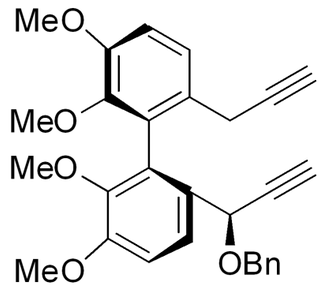
```
Current Data Parameters
NAME                  Rs-2-36
EXPNO                       1
PROCNO                      1

F2 - Acquisition Parameters
Date_              20071120
Time                  12.07
INSTRUM               spect
PROBHD   5 mm QNP 1H/13
PULPROG               zg30
TD                   65536
SOLVENT              CDCl3
NS                      16
DS                       2
SWH                8278.146 Hz
FIDRES             0.126314 Hz
AQ                 3.9584243 sec
RG                     143.7
DW                    60.400 usec
DE                     6.00 usec
TE                    301.2 K
D1                 1.00000000 sec
MCREST             0.00000000 sec
MCWRK              0.01500000 sec

======== CHANNEL f1 ========
NUC1                      1H
P1                     13.00 usec
PL1                     0.00 dB
SFO1           400.1324710 MHz

F2 - Processing parameters
SI                     32768
SF             400.1300181 MHz
WDW                       EM
SSB                        0
LB                      0.30 Hz
GB                         0
PC                      1.00
```





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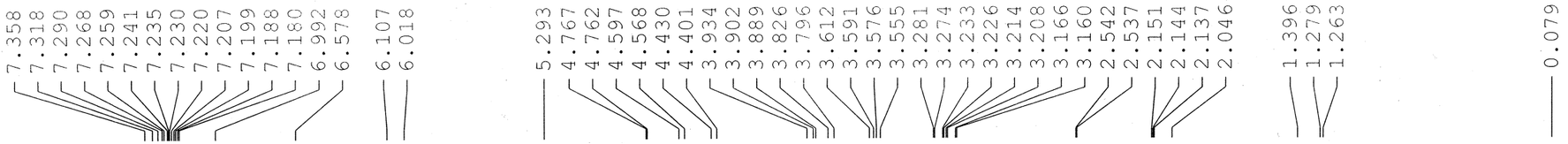
The Ohio State University
Department of Chemistry
NMR Facility
400MHz – 0083

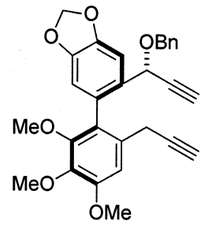
```
Current Data Parameters
NAME           Rs-2-40mp
EXPNO                  1
PROCNO                 1

F2 - Acquisition Parameters
Date_           20071127
Time               13.32
INSTRUM            spect
PROBHD   5 mm QNP 1H/13
PULPROG             zg30
TD                 65536
SOLVENT            CDCl3
NS                    16
DS                     2
SWH             8278.146 Hz
FIDRES          0.126314 Hz
AQ              3.9584243 sec
RG                 228.1
DW                60.400 use
DE                 6.00 use
TE                300.2 K
D1           1.00000000 sec
MCREST       0.00000000 sec
MCWRK        0.01500000 sec

======== CHANNEL f1 =======
NUC1                   1H
P1                 13.00 use
PL1                 0.00 dB
SFO1         400.1324710 MHz

F2 - Processing parameters
SI                 32768
SF          400.1300181 MHz
WDW                   EM
SSB                    0
LB                 0.30 Hz
GB                     0
PC                  1.00
```





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The Ohio State University
Department of Chemistry
NMR Facility
400MHz - 0083

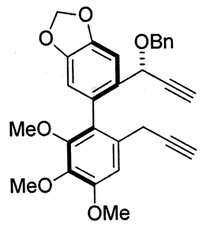
```
Current Data Parameters
NAME      Wgong-II-29 diyne
EXPNO                    9
PROCNO                   1

F2 - Acquisition Parameters
Date_           20091221
Time              14.04
INSTRUM           spect
PROBHD   5 mm QNP 1H/13
PULPROG            zg30
TD                65536
SOLVENT           CDCl3
NS                   16
DS                    2
SWH            8278.146 Hz
FIDRES         0.126314 Hz
AQ             3.9584243 sec
RG                  114
DW                60.400 usec
DE                 6.00 usec
TE                300.2 K
D1           1.00000000 sec
TD0                  1

======== CHANNEL f1 ========
NUC1                 1H
P1                13.00 usec
PL1                0.00 dB
SFO1        400.1324710 MHz

F2 - Processing parameters
SI                 32768
SF          400.1300097 MHz
WDW                  EM
SSB                   0
LB                 0.30 Hz
GB                    0
PC                 1.00
```

153.27
150.93
147.77
147.55
140.84
137.62
131.72
130.86
128.47
128.23
128.16
128.05
127.94
127.55
125.09
109.92
107.52
107.35
101.38
82.30
82.12
77.36
77.24
77.04
76.72
75.04
70.70
70.57
67.56
60.86
60.82
55.97
53.43
22.95



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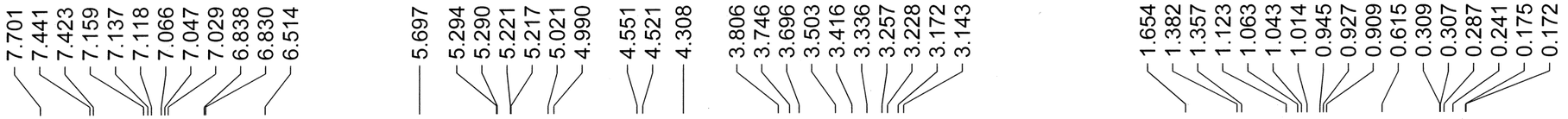
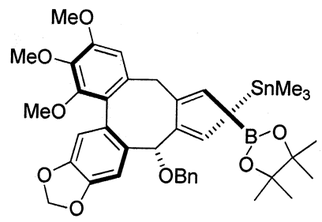
BRUKER

```
Current Data Parameters
NAME      Wgong-II-29 diyne C13
EXPNO                  10
PROCNO                  1

F2 - Acquisition Parameters
Date_           20091221
Time               14.11
INSTRUM            spect
PROBHD   5 mm QNP 1H/13
PULPROG           zgpg30
TD                65536
SOLVENT           CDCl3
NS                  780
DS                    4
SWH           23980.814 Hz
FIDRES         0.365918 Hz
AQ            1.3664756 sec
RG               4597.6
DW                20.850 usec
DE                 6.00 usec
TE                300.2 K
D1           2.00000000 sec
d11          0.03000000 sec
DELTA        1.89999998 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                  13C
P1                 10.50 usec
PL1                 0.00 dB
SFO1         100.6228298 MHz

======== CHANNEL f2 ========
CPDPRG2          waltz16
NUC2                  1H
PCPD2              80.00 usec
PL2                -6.00 dB
PL12               14.56 dB
PL13               16.50 dB
SFO2         400.1316005 MHz

F2 - Processing parameters
SI                 32768
SF          100.6127690 MHz
WDW                   EM
SSB                    0
LB                  1.00 Hz
GB                     0
PC                  1.00
```





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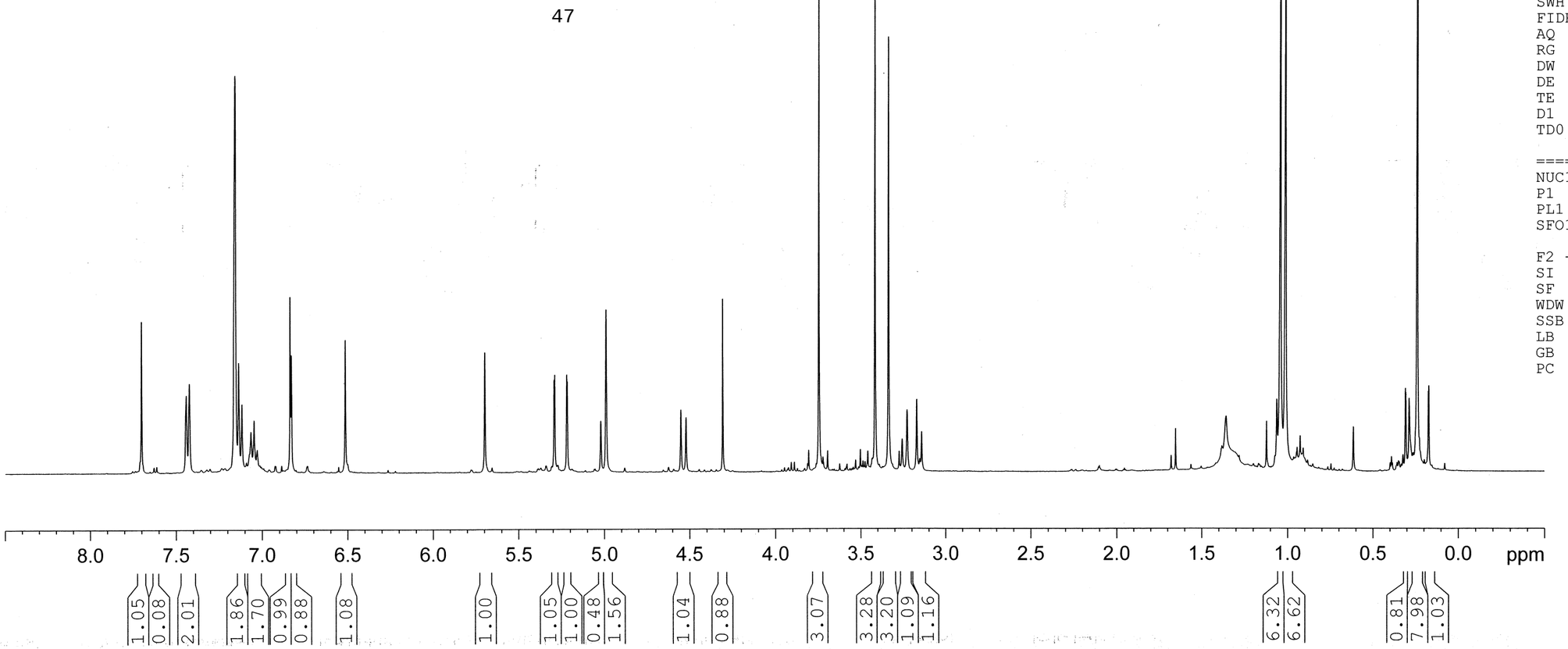
The Ohio State University
Department of Chemistry
NMR Facility

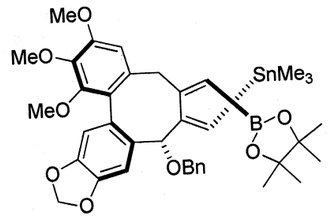
```
Current Data Parameters
NAME       Wgong-II-78-2
EXPNO                 77
PROCNO                 1

F2 - Acquisition Parameters
Date_           20100314
Time              15.43
INSTRUM           spect
PROBHD   5 mm QNP 1H/13
PULPROG           zg30
TD               65536
SOLVENT           C6D6
NS                  16
DS                   2
SWH           8278.146 Hz
FIDRES        0.126314 Hz
AQ            3.9584243 sec
RG                  57
DW             60.400 usec
DE              6.00 usec
TE              300.2 K
D1          1.00000000 sec
TD0                  1

======== CHANNEL f1 ========
NUC1                1H
P1             13.00 usec
PL1             0.00 dB
SFO1       400.1324710 MHz

F2 - Processing parameters
SI                32768
SF         400.1300453 MHz
WDW                EM
SSB                 0
LB              0.30 Hz
GB                  0
PC               1.00
```





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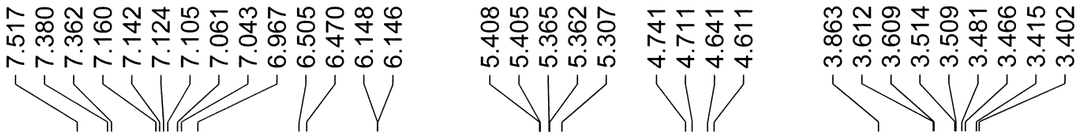
164.62
160.87
153.96
151.60
148.23
146.39
141.96
139.50
137.35
135.73
128.28
128.04
127.80
127.68
127.56
127.25
125.90
123.94
110.32
107.90
104.97
100.79
82.84
79.76
70.79
60.47
60.32
55.26
53.09
45.99
29.96
24.92
24.84
24.70
1.16

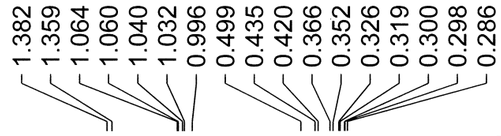
BRUKER

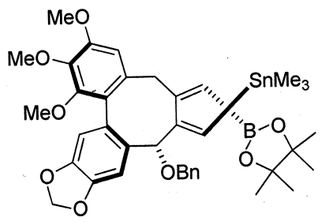
```
Current Data Parameters
NAME      Wgong-II-78-2 C13
EXPNO                   78
PROCNO                   1

F2 - Acquisition Parameters
Date_           20100314
Time               15.54
INSTRUM            spect
PROBHD   5 mm QNP 1H/13
PULPROG          zgpg30
TD                65536
SOLVENT             [redacted] C6D6
NS                  728
DS                    4
SWH           23980.814 Hz
FIDRES         0.365918 Hz
AQ             1.3664756 sec
RG                2896.3
DW                20.850 usec
DE                 6.00 usec
TE                300.2 K
D1           2.00000000 sec
d11          0.03000000 sec
DELTA        1.89999998 sec
TD0                  1

======== CHANNEL f1 ========
NUC1                13C
P1               10.50 usec
PL1               0.00 dB
SFO1        100.6228298 MHz

======== CHANNEL f2 ========
CPDPRG2         waltz16
NUC2                1H
PCPD2           80.00 usec
PL2             -6.00 dB
PL12            14.56 dB
PL13            16.50 dB
SFO2        400.1316005 MHz

F2 - Processing parameters
SI                32768
SF          100.6127690 MHz
WDW                EM
SSB                 0
LB               1.00 Hz
GB                  0
PC               1.00
```

-7.70

210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm







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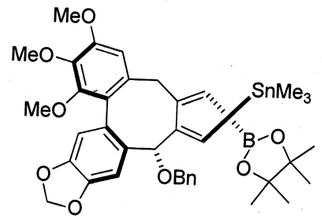
```
Current Data Parameters
NAME      Wgong-II-78-1 p
EXPNO                463
PROCNO                 1

F2 - Acquisition Parameters
Date_           20110427
Time_              12.16
INSTRUM            spect
PROBHD   5 mm QNP 1H/13
PULPROG             zg30
TD                 65536
SOLVENT             C6D6
NS                    16
DS                     2
SWH             8278.146 Hz
FIDRES          0.126314 Hz
AQ              3.9584243 sec
RG                  90.5
DW                60.400 usec
DE                 6.00 usec
TE                300.2 K
D1             1.00000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                  1H
P1                14.50 usec
PL1                0.00 dB
SFO1         400.1324710 MHz

F2 - Processing parameters
SI                 32768
SF           400.1300451 MHz
WDW                   EM
SSB                    0
LB                0.30 Hz
GB                     0
PC                 1.00
```



48

176.34
174.86
153.32
151.38
147.58
146.51
141.45
139.46
134.87
133.79
130.67
127.09
126.75
110.56
109.98
107.93
100.77
85.89
83.07
82.97
71.19
60.65
60.55
55.34
47.90
29.96
24.80
24.68
24.65
24.52
-5.74
-6.37

BRUKER

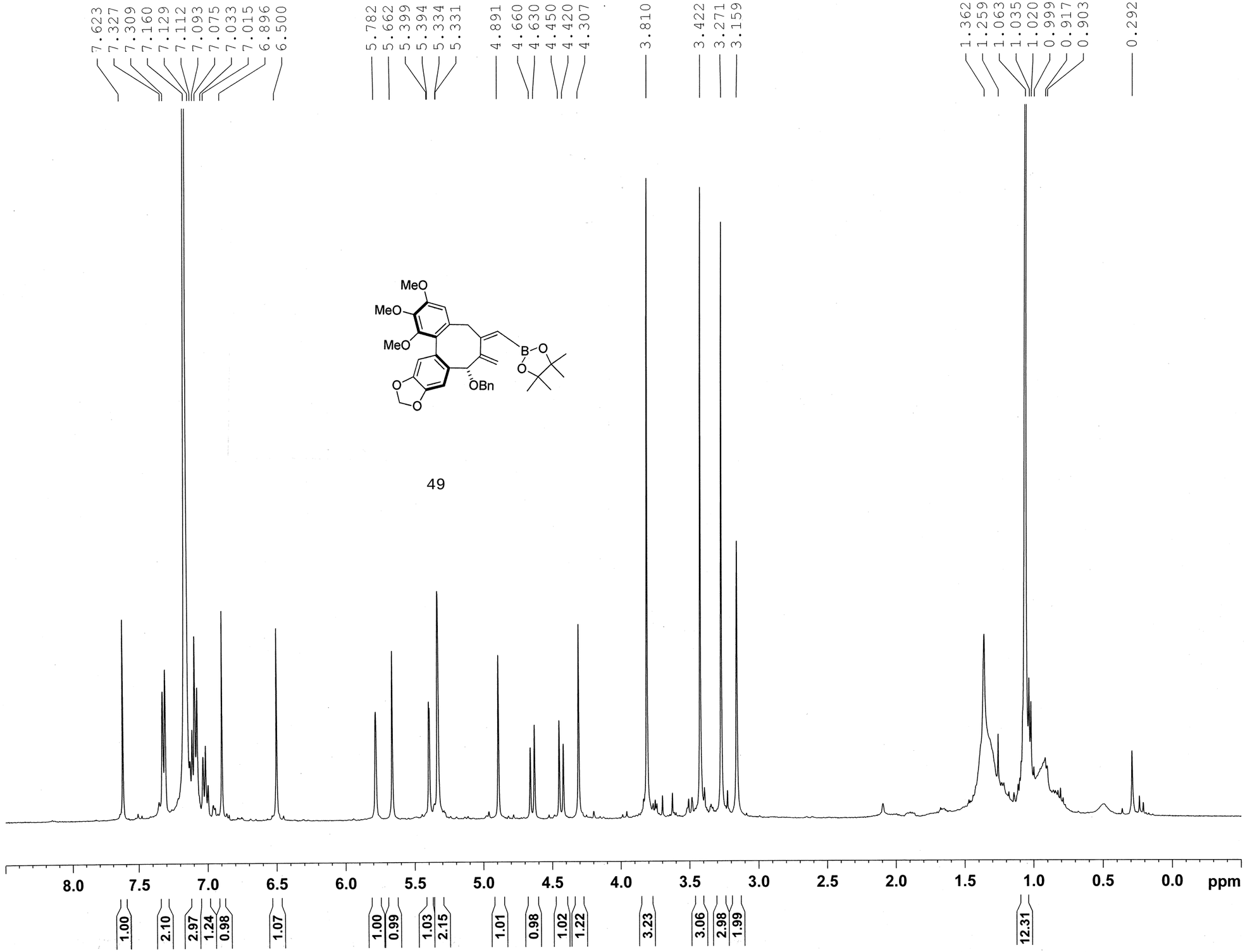
Current Data Parameters
NAME Wgong-II-78-1 p C13
EXPNO 464
PROCNO 1

F2 - Acquisition Parameters
Date_ 20110427
Time 12.29
INSTRUM spect
PROBHD 5 mm QNP 1H/13
PULPROG zgpg30
TD 65536
SOLVENT [redacted] C_6D_6
NS 537
DS 4
SWH 23980.814 Hz
FIDRES 0.365918 Hz
AQ 1.3664756 sec
RG 1625.5
DW 20.850 usec
DE 6.00 usec
TE 300.2 K
D1 2.00000000 sec
d11 0.03000000 sec
DELTA 1.89999998 sec
TD0 1

======== CHANNEL f1 ========
NUC1 13C
P1 10.50 usec
PL1 0.00 dB
SFO1 100.6228298 MHz

======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 -6.00 dB
PL12 14.56 dB
PL13 16.50 dB
SFO2 400.1316005 MHz

F2 - Processing parameters
SI 32768
SF 100.6127690 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40



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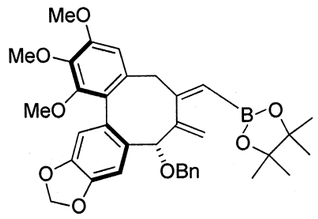
NMR Facility
400MHz - 0083

```
Current Data Parameters
NAME          Wgong-II-67
EXPNO                  78
PROCNO                  1

F2 - Acquisition Parameters
Date_           20100315
Time               15.36
INSTRUM            spect
PROBHD   5 mm QNP 1H/13
PULPROG             zg30
TD                 65536
SOLVENT             C6D6
NS                    16
DS                     2
SWH             8278.146 Hz
FIDRES          0.126314 Hz
AQ              3.9584243 sec
RG                   181
DW                60.400 usec
DE                 6.00 usec
TE                300.2 K
D1             1.00000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                  1H
P1                13.00 usec
PL1                0.00 dB
SFO1          400.1324710 MHz

F2 - Processing parameters
SI                 32768
SF          400.1300454 MHz
WDW                   EM
SSB                    0
LB                0.30 Hz
GB                     0
PC                  1.40
```



49

128.12
127.34
127.18
109.46
101.02
83.08
77.33
77.01
76.69
70.04
29.70
24.92
24.54

BRUKER

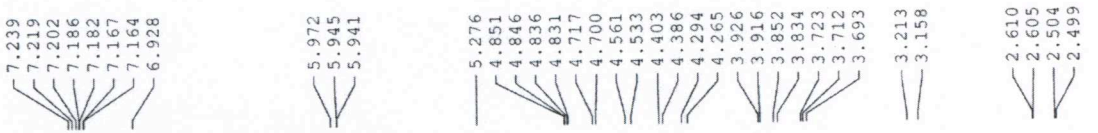
Current Data Parameters
NAME Wgong-II-8 C13
EXPNO 4
PROCNO 1

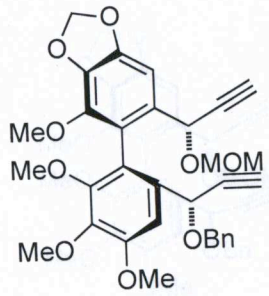
F2 - Acquisition Parameters
Date_ 20091119
Time 12.37
INSTRUM spect
PROBHD 5 mm QNP 1H/13
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 347
DS 4
SWH 23980.814 Hz
FIDRES 0.365918 Hz
AQ 1.3664756 sec
RG 2048
DW 20.850 usec
DE 6.00 usec
TE 302.2 K
D1 2.00000000 sec
d11 0.03000000 sec
DELTA 1.89999998 sec
TD0 1

======== CHANNEL f1 ========
NUC1 13C
P1 10.50 usec
PL1 0.00 dB
SFO1 100.6228298 MHz

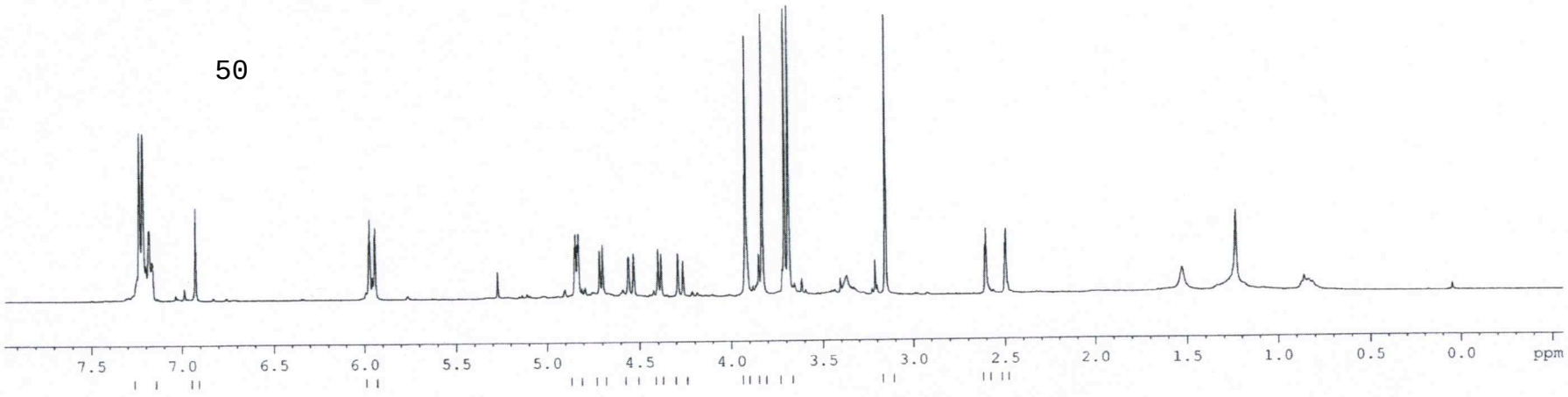
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 -6.00 dB
PL12 14.56 dB
PL13 16.50 dB
SFO2 400.1316005 MHz

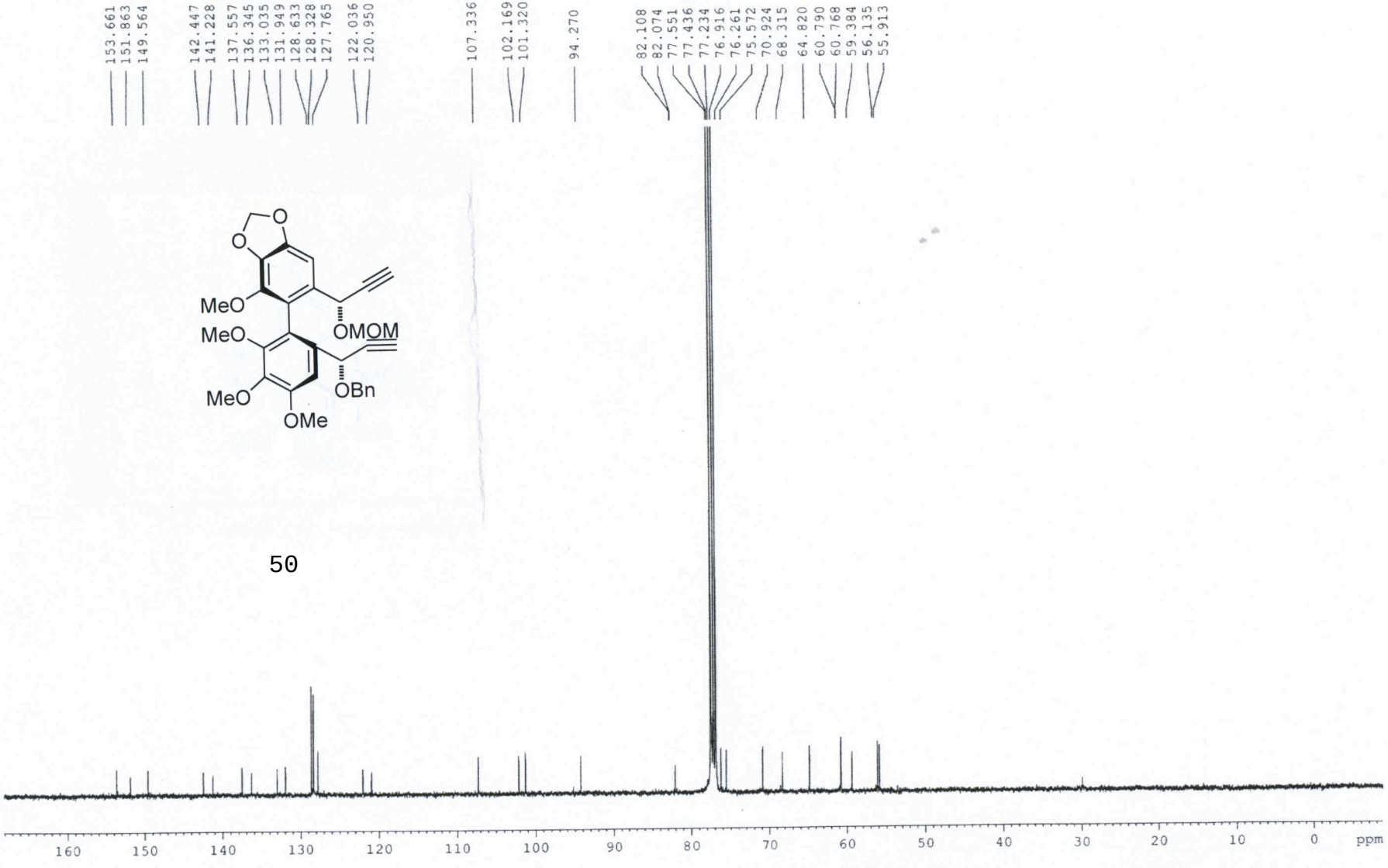
F2 - Processing parameters
SI 32768
SF 100.6127690 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40





50





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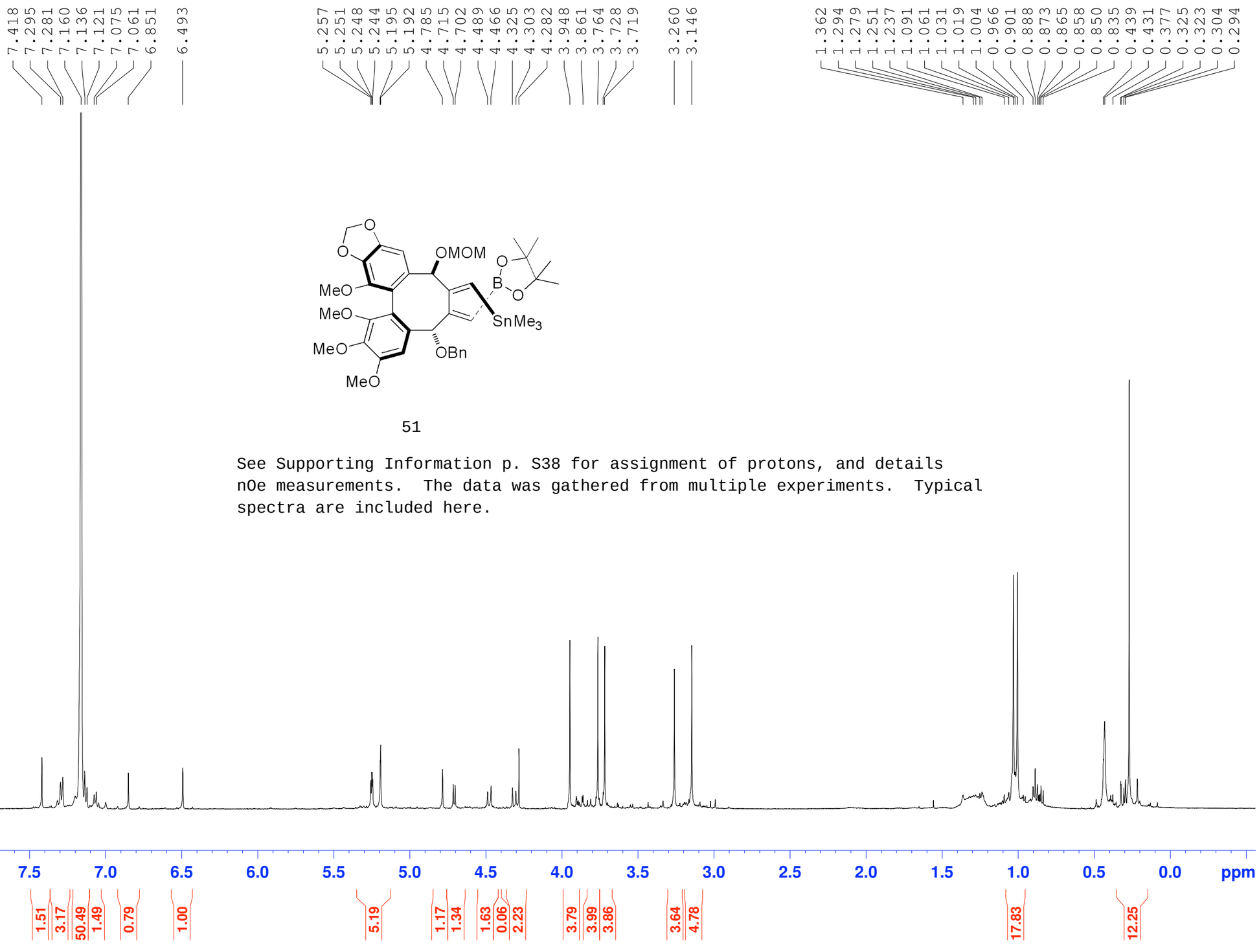

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See Supporting Information p. S38 for assignment of protons, and details nOe measurements. The data was gathered from multiple experiments. Typical spectra are included here.

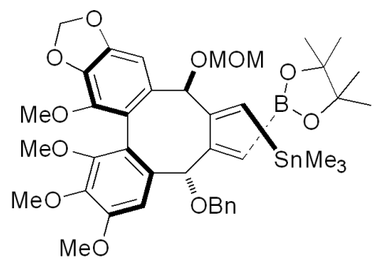
```
Current Data Parameters
NAME          Rs-6-187-3
EXPNO                  1
PROCNO                 1

F2 - Acquisition Parameters
Date_           20101122
Time               16.53
INSTRUM            spect
PROBHD   5 mm Multinucl
PULPROG             zg30
TD                 65536
SOLVENT             C6D6
NS                    16
DS                     2
SWH           10330.578 Hz
FIDRES         0.157632 Hz
AQ            3.1719923 sec
RG                645.1
DW               48.400 usec
DE                6.00 usec
TE               300.2 K
D1            1.00000000 sec
MCREST        0.00000000 sec
MCWRK         0.01500000 sec

======== CHANNEL f1 ========
NUC1                  1H
P1                17.33 usec
PL1               -1.00 dB
SFO1         500.0230878 MHz

F2 - Processing parameters
SI                 32768
SF           500.0200550 MHz
WDW                   EM
SSB                    0
LB                0.30 Hz
GB                     0
PC                1.40
```

164.632
158.454
154.320
151.289
149.891
141.331
140.314
138.434
137.841
137.094
135.833
128.333
128.032
127.850
127.533
127.173
120.935
118.873
103.188
101.262
99.276
94.013
83.176
80.000
77.483
77.229
76.975
74.986
70.587
61.275
60.692
59.927
56.172
55.430
53.627
31.135
29.922
29.581
25.369
24.999
22.910
14.326
1.238



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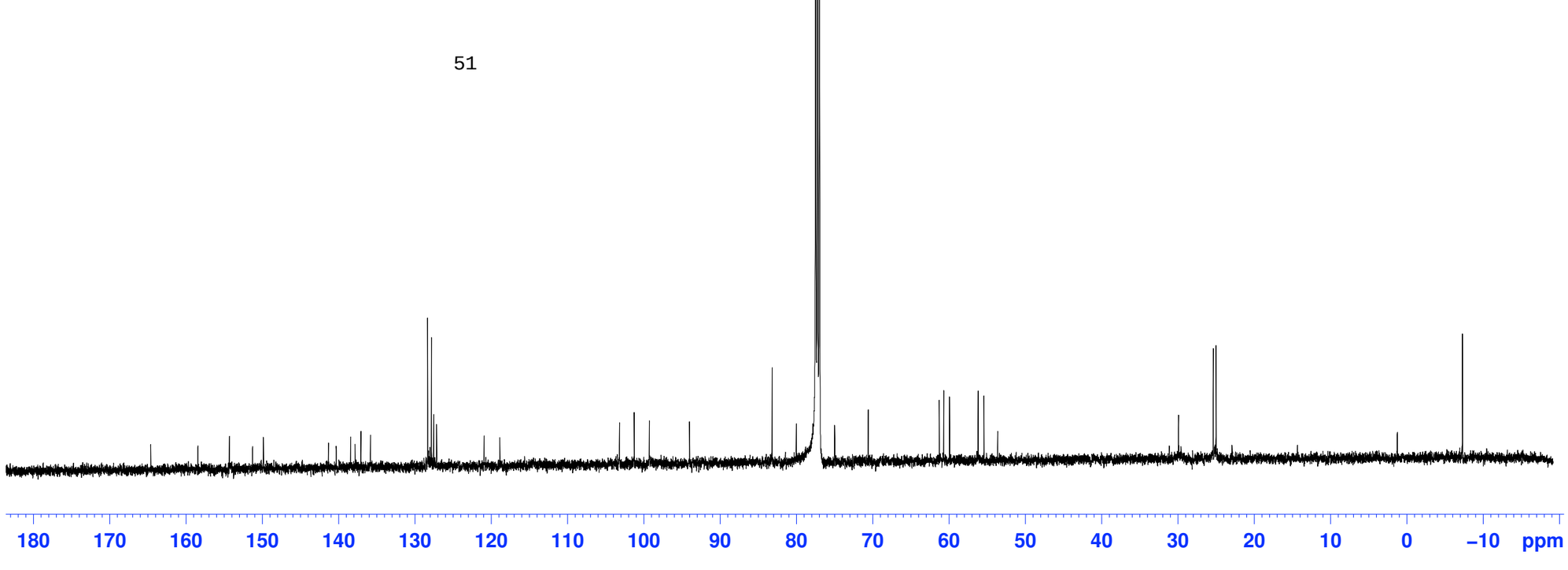
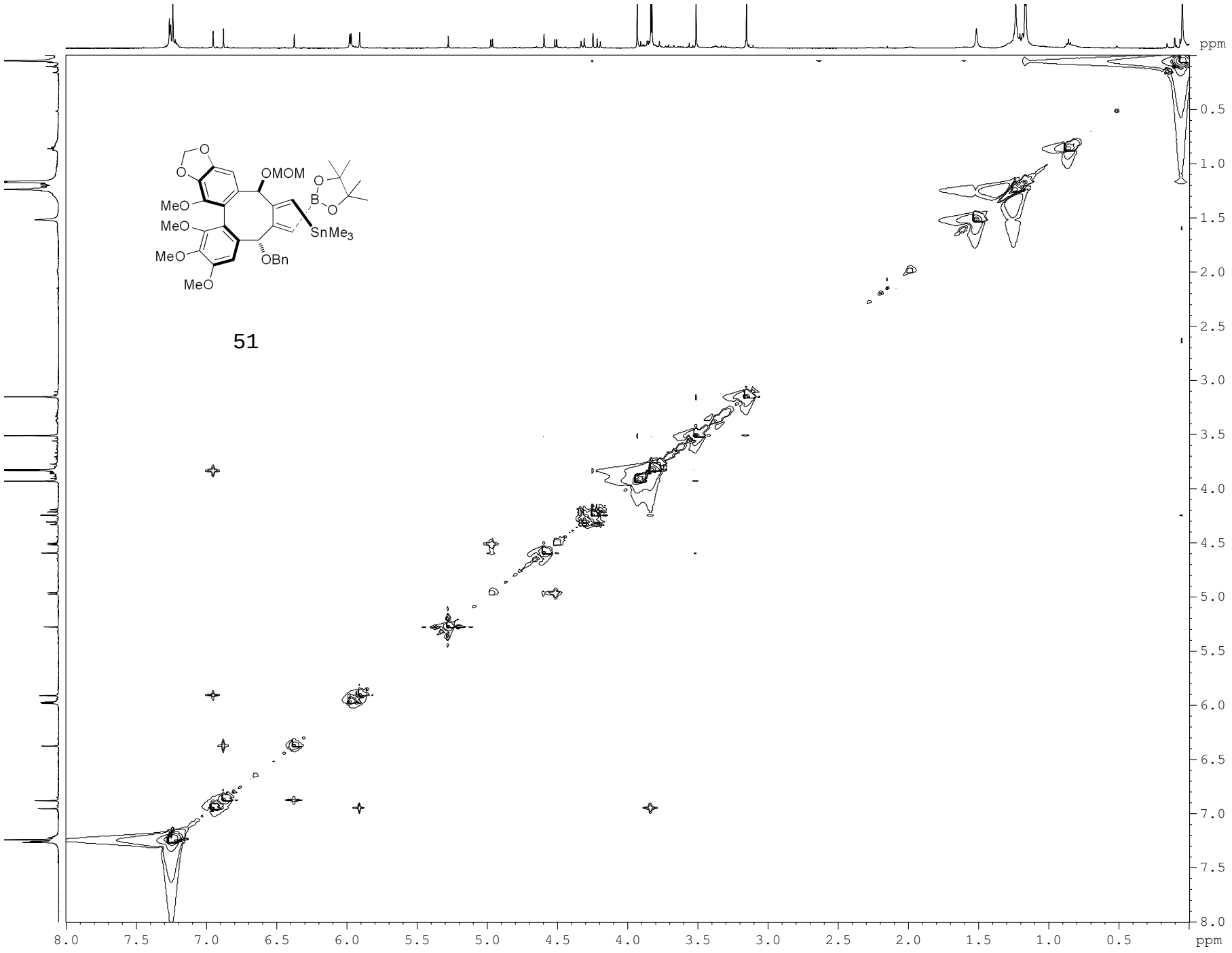
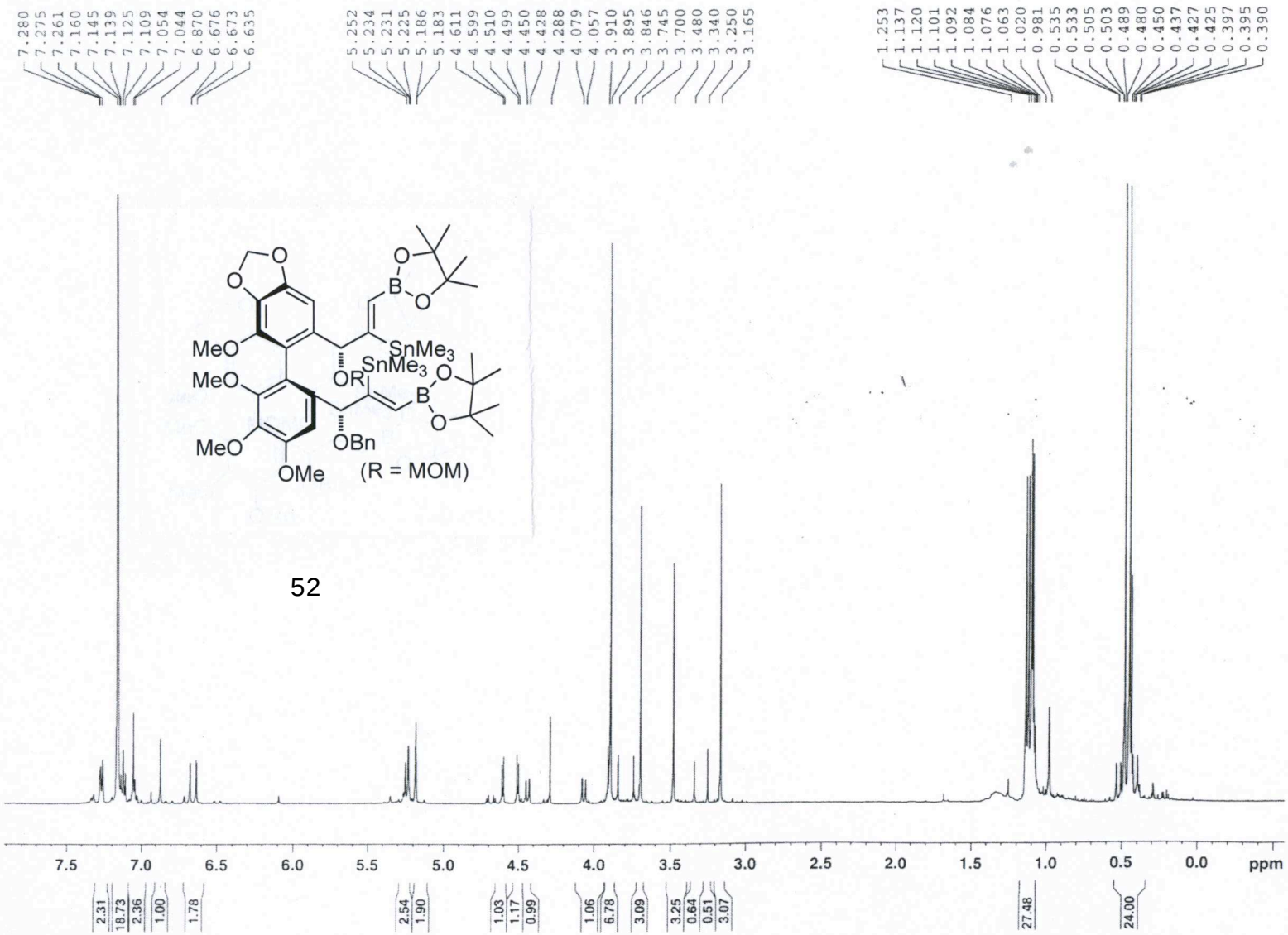



```
Current Data Parameters
NAME          Rs-6-187-3
EXPNO                  3
PROCNO                 1

F2 - Acquisition Parameters
Date_           20101123
Time               19.06
INSTRUM            spect
PROBHD   5 mm Multinucl
PULPROG          zgpg30
TD                65536
SOLVENT           CDCl3
NS                15934
DS                    4
SWH           30030.029 Hz
FIDRES         0.458222 Hz
AQ            1.0912244 sec
RG               4597.6
DW                16.650 usec
DE                12.00 usec
TE                300.2 K
D1           2.00000000 sec
d11          0.03000000 sec
DELTA        1.89999998 sec
MCREST       0.00000000 sec
MCWRK        0.01500000 sec

======== CHANNEL f1 ========
NUC1                13C
P1                27.00 usec
PL1                1.00 dB
SFO1         125.7427020 MHz

======== CHANNEL f2 ========
CPDPRG2          waltz16
NUC2                1H
PCPD2             68.00 usec
PL2               -1.00 dB
PL12              10.87 dB
PL13              22.50 dB
SFO2         500.0220001 MHz

F2 - Processing parameters
SI                 32768
SF           125.7301014 MHz
WDW                   EM
SSB                    0
LB                 1.00 Hz
GB                     0
PC                 1.00
```





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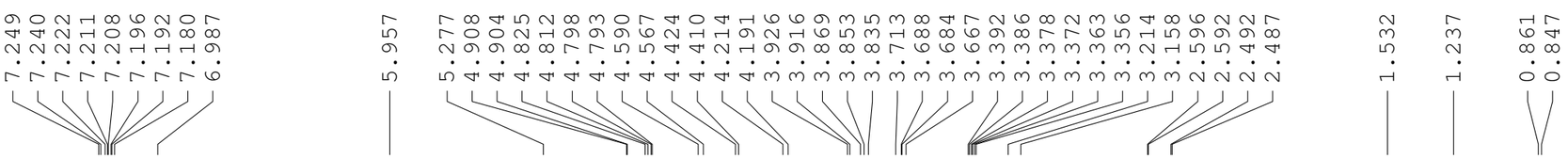
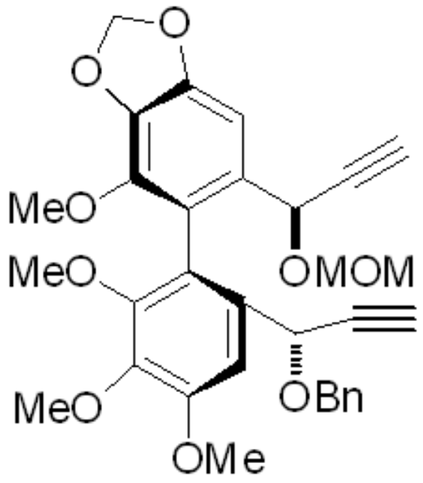
```
Current Data Parameters
NAME            Rs-6-187-1
EXPNO                    1
PROCNO                   1

F2 - Acquisition Parameters
Date_             20101123
Time                 14.15
INSTRUM              spect
PROBHD   5 mm Multinucl
PULPROG              zg30
TD                  65536
SOLVENT               C6D6
NS                      16
DS                       2
SWH              10330.578 Hz
FIDRES               0.157632 Hz
AQ                3.1719923 sec
RG                     287.4
DW                    48.400 usec
DE                     6.00 usec
TE                    300.2 K
D1               1.00000000 sec
MCREST           0.00000000 sec
MCWRK            0.01500000 sec

======== CHANNEL f1 ========
NUC1                    1H
P1                   17.33 usec
PL1                  -1.00 dB
SFO1           500.0230878 MHz

F2 - Processing parameters
SI                   32768
SF             500.0200550 MHz
WDW                     EM
SSB                      0
LB                    0.30 Hz
GB                       0
PC                    1.00
```





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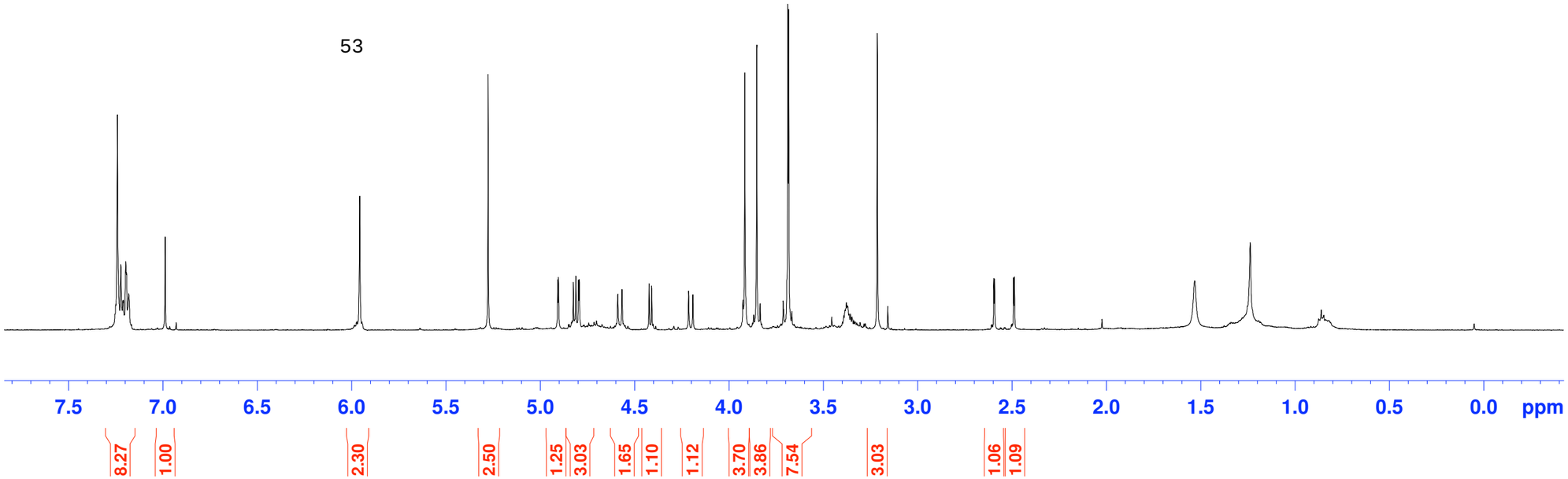
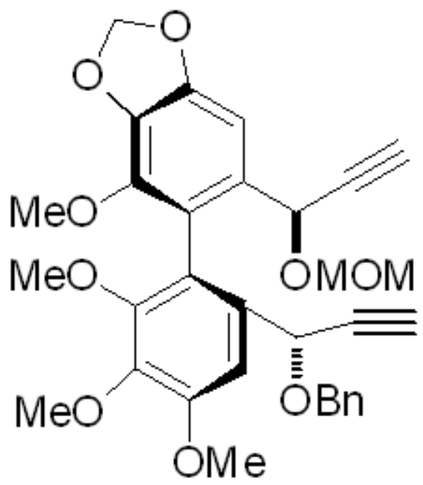


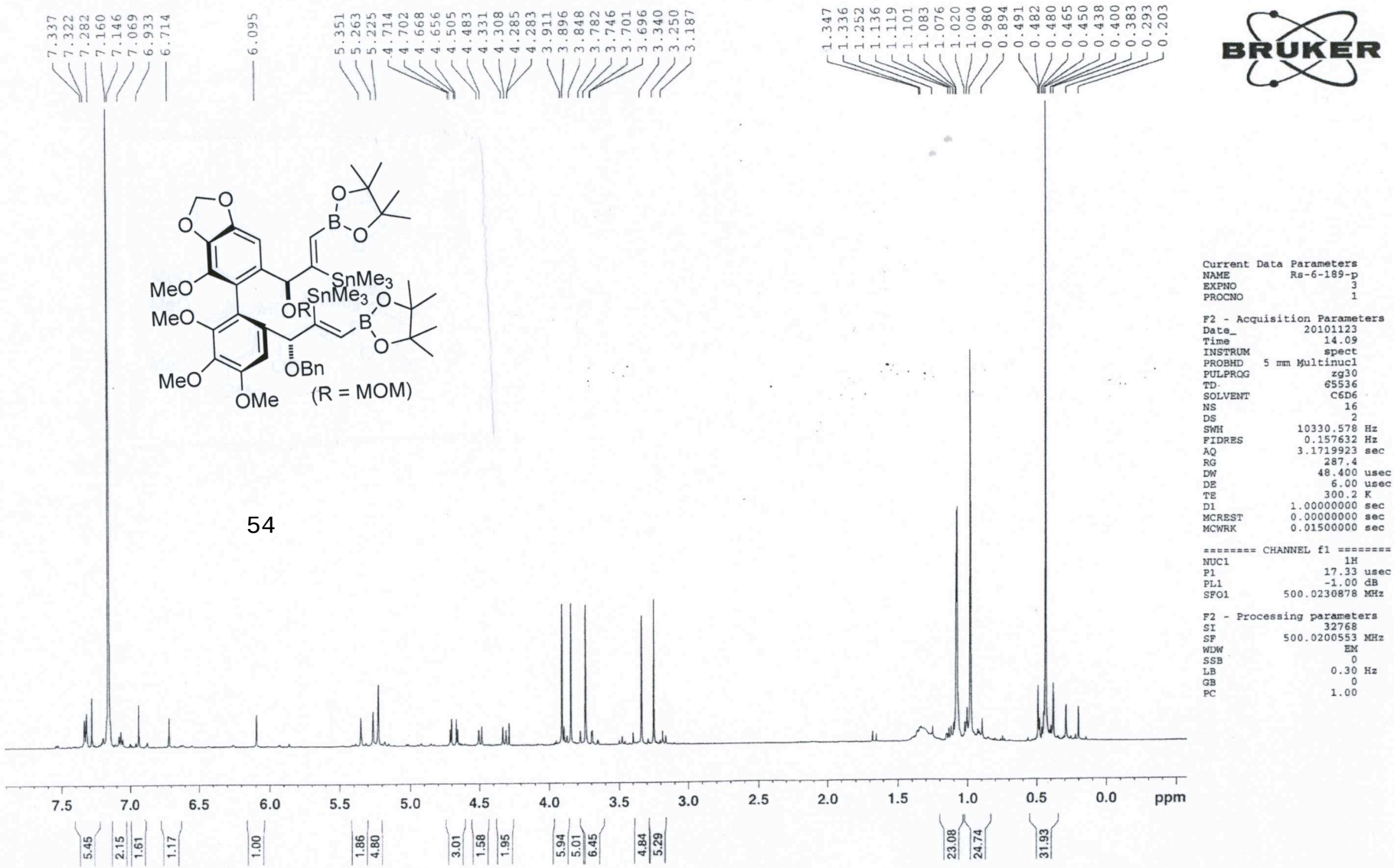

```
Current Data Parameters
NAME            Rs-6-188
EXPNO                  1
PROCNO                 1

F2 - Acquisition Parameters
Date_           20101122
Time               13.58
INSTRUM            spect
PROBHD   5 mm Multinucl
PULPROG             zg30
TD                 65536
SOLVENT            CDCl3
NS                    16
DS                     2
SWH           10330.578 Hz
FIDRES         0.157632 Hz
AQ             3.1719923 sec
RG                  80.6
DW                48.400 usec
DE                 6.00 usec
TE                300.2 K
D1           1.00000000 sec
MCREST       0.00000000 sec
MCWRK        0.01500000 sec

======== CHANNEL f1 ========
NUC1                   1H
P1                 17.33 usec
PL1                -1.00 dB
SFO1         500.0230878 MHz

F2 - Processing parameters
SI                 32768
SF           500.0200216 MHz
WDW                   EM
SSB                    0
LB                  0.30 Hz
GB                     0
PC                  1.00
```

153.516
152.058
149.452
142.599
141.650
137.699
136.426
132.655
131.577
128.647
128.319
127.737
122.319
120.846
107.755
102.144
101.341
95.286
82.283
81.831
77.496
77.243
76.988
76.076
75.531
71.198
68.592
66.006
61.031
60.874
59.431
56.359
56.200
53.641
29.928
14.341

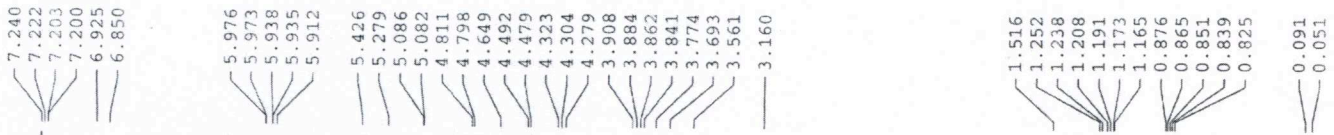


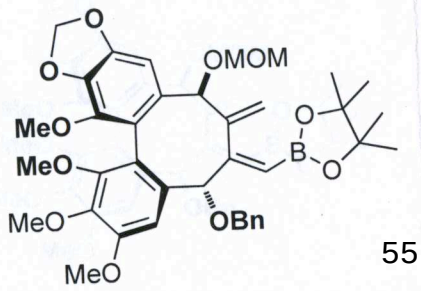
53


```
Current Data Parameters
NAME            Rs-6-188
EXPNO                  2
PROCNO                 1

F2 - Acquisition Parameters
Date_           20101122
Time               14.11
INSTRUM            spect
PROBHD   5 mm Multinucl
PULPROG          zgpg30
TD                65536
SOLVENT           CDCl3
NS                 3160
DS                    4
SWH           30030.029 Hz
FIDRES         0.458222 Hz
AQ            1.0912244 sec
RG               1290.2
DW               16.650 usec
DE               12.00 usec
TE               300.2 K
D1           2.00000000 sec
d11          0.03000000 sec
DELTA        1.89999998 sec
MCREST       0.00000000 sec
MCWRK        0.01500000 sec

======== CHANNEL f1 ========
NUC1               13C
P1               27.00 usec
PL1               1.00 dB
SFO1        125.7427020 MHz

======== CHANNEL f2 ========
CPDPRG2         waltz16
NUC2               1H
PCPD2            68.00 usec
PL2              -1.00 dB
PL12             10.87 dB
PL13             22.50 dB
SFO2        500.0220001 MHz

F2 - Processing parameters
SI                32768
SF          125.7301005 MHz
WDW                EM
SSB                 0
LB               1.00 Hz
GB                  0
PC               1.00
```



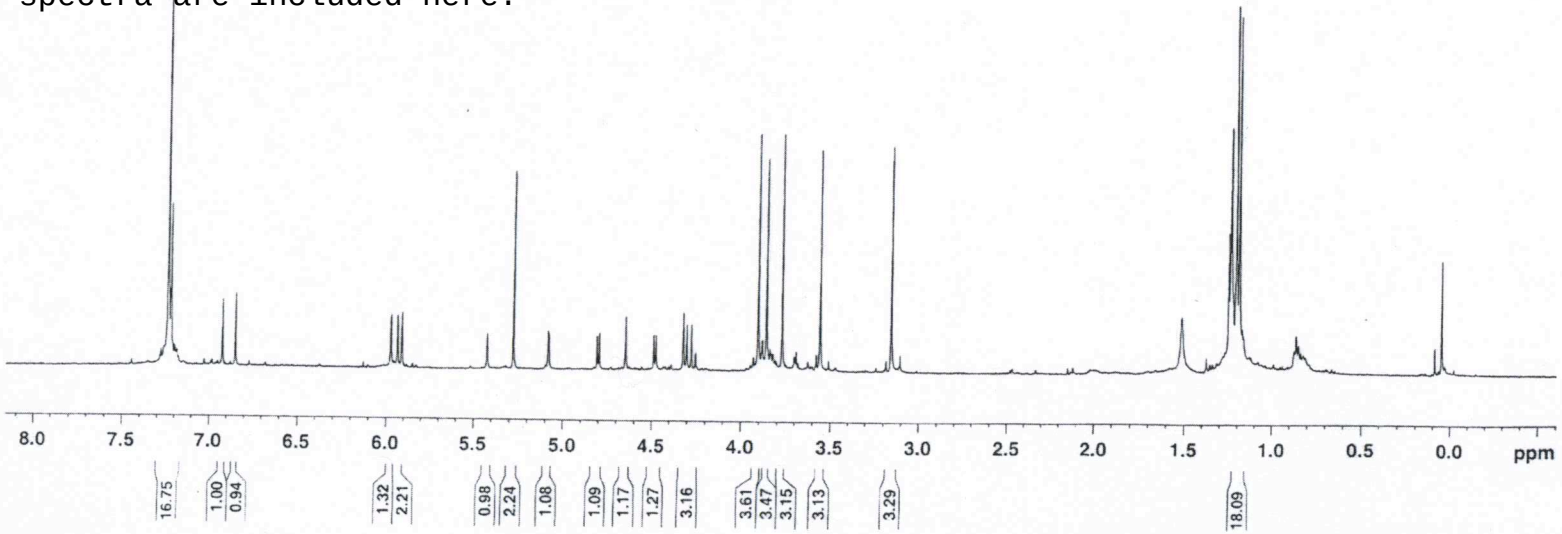

```
Current Data Parameters
NAME                  Rs-6-189-p
EXPNO                          3
PROCNO                         1

F2 - Acquisition Parameters
Date_                   20101123
Time                       14.09
INSTRUM                    spect
PROBHD     5 mm Multinucl
PULPROG                    zg30
TD                        65536
SOLVENT                     C6D6
NS                            16
DS                             2
SWH                    10330.578 Hz
FIDRES                 0.157632 Hz
AQ                     3.1719923 sec
RG                         287.4
DW                        48.400 usec
DE                         6.00 usec
TE                        300.2 K
D1                     1.00000000 sec
MCREST                 0.00000000 sec
MCWRK                  0.01500000 sec

======== CHANNEL f1 ========
NUC1                          1H
P1                        17.33 usec
PL1                       -1.00 dB
SFO1                 500.0230878 MHz

F2 - Processing parameters
SI                         32768
SF                   500.0200553 MHz
WDW                           EM
SSB                            0
LB                          0.30 Hz
GB                             0
PC                          1.00
```





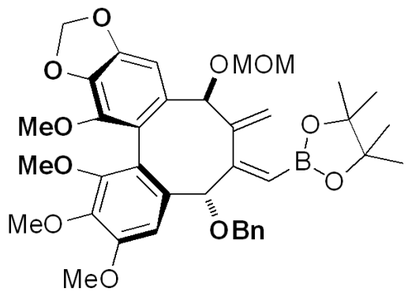
55

See Supporting Information p. S38 for assignment of protons, and details nOe measurements. The data was gathered from multiple experiments. Typical spectra are included here.

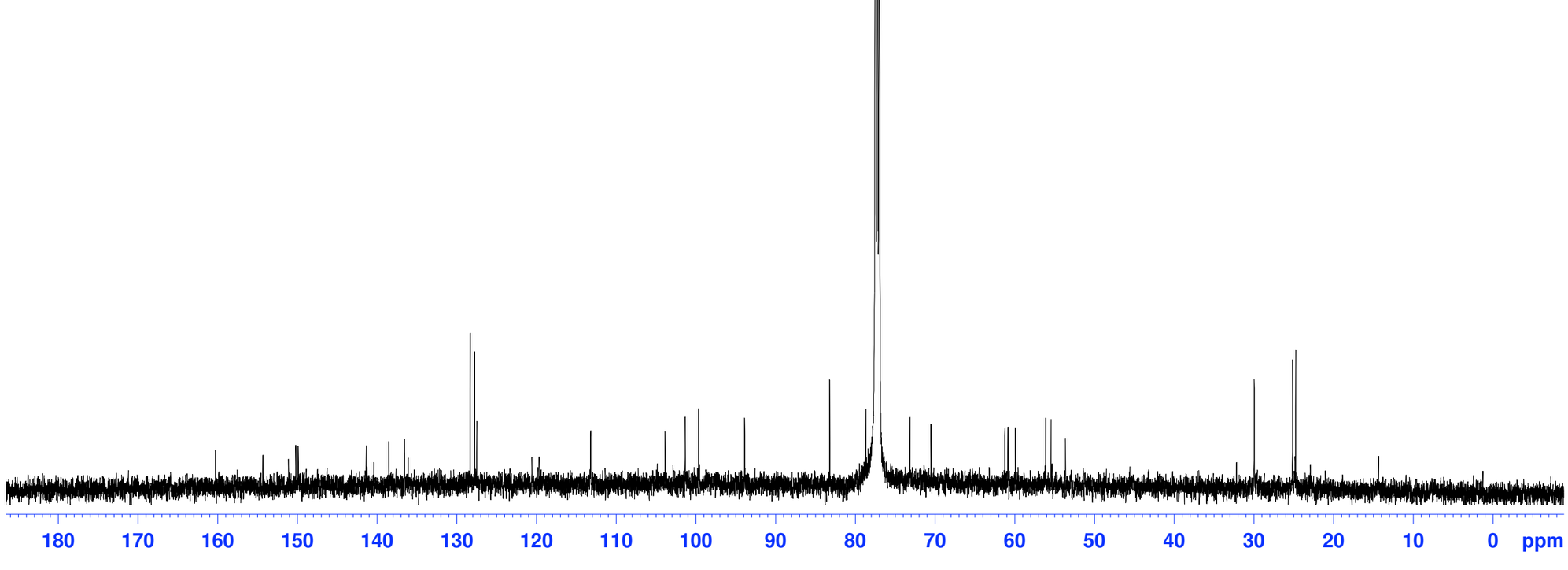



```
Current Data Parameters
NAME                  Rs-2-152
EXPNO                        7
PROCNO                       1

F2 - Acquisition Parameters
Date_                 20080225
Time                     21.37
INSTRUM                  spect
PROBHD    5 mm Multinucl
PULPROG                  zg30
TD                      65536
SOLVENT                 CDCl3
NS                         16
DS                          2
SWH                10330.578 Hz
FIDRES             0.157632 Hz
AQ                 3.1719923 sec
RG                     645.1
DW                    48.400 usec
DE                     6.00 usec
TE                     300.2 K
D1                 1.00000000 sec
MCREST             0.00000000 sec
MCWRK              0.01500000 sec

======== CHANNEL f1 ========
NUC1                       1H
P1                    14.80 usec
PL1                   -1.00 dB
SFO1           500.0230878 MHz

F2 - Processing parameters
SI                      32768
SF             500.0200216 MHz
WDW                        EM
SSB                         0
LB                     0.30 Hz
GB                          0
PC                     1.40
```

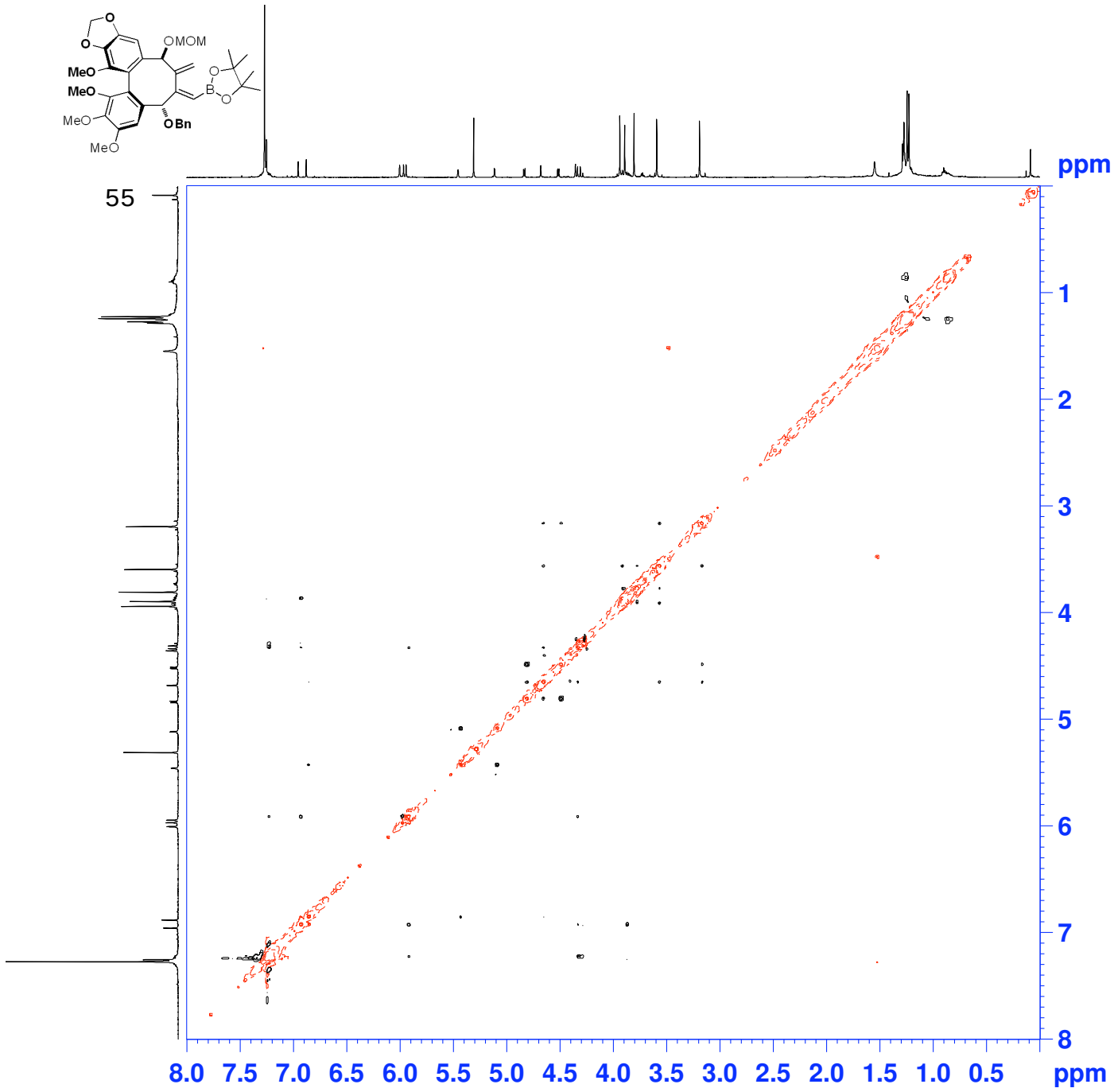
160.293
154.333
151.119
150.198
149.906
141.373
138.530
136.548
136.106
128.333
127.777
127.500
120.584
119.688
113.209
103.871
101.347
99.680
93.892
83.222
78.664
77.501
77.247
76.994
73.146
70.518
61.225
60.830
59.917
56.100
55.433
53.645
29.944
25.138
24.836
24.741
14.347



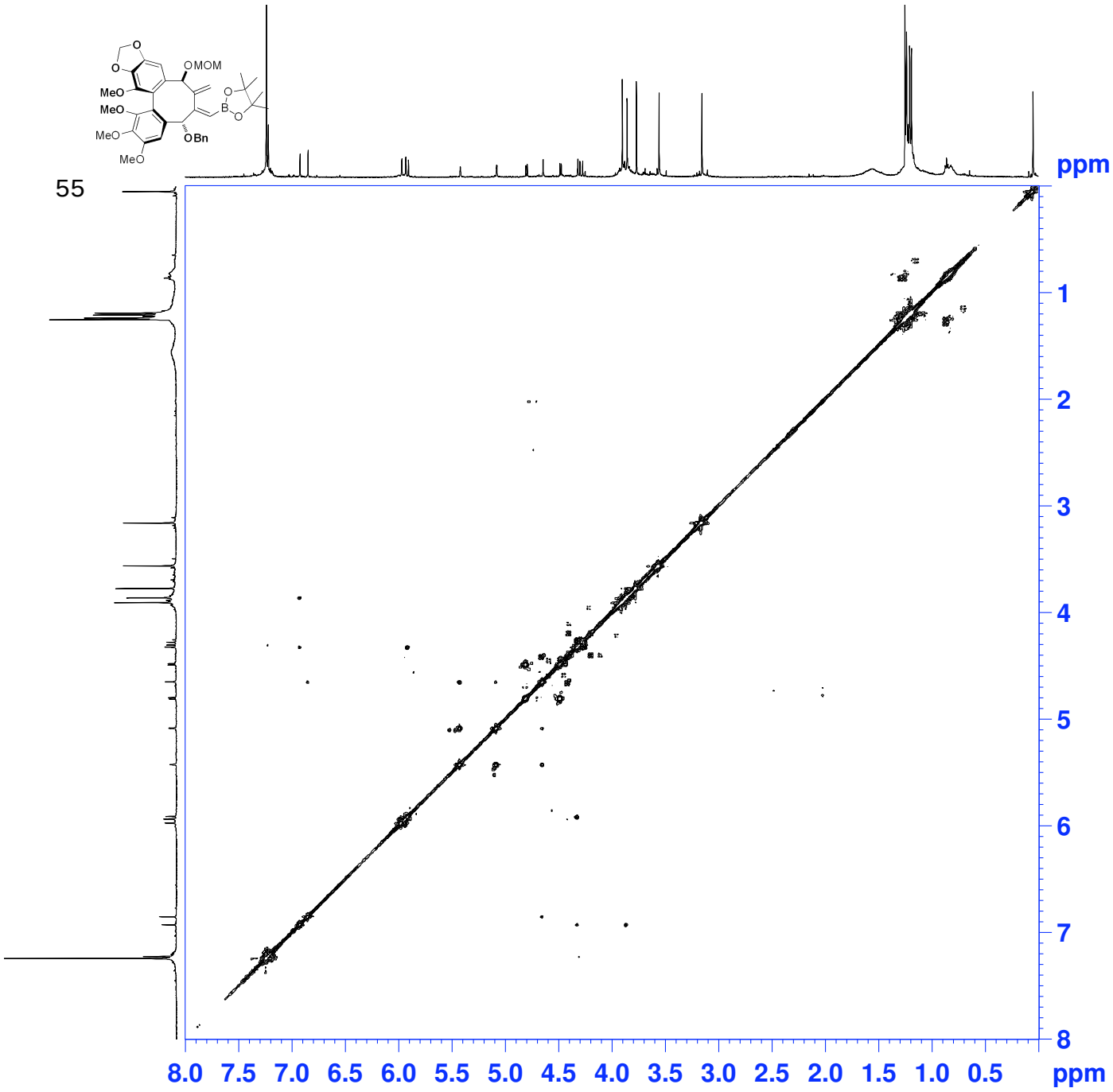
55




```
Current Data Parameters
NAME            Rs-2-152
EXPNO                  6
PROCNO                 1

F2 - Acquisition Parameters
Date_           20080223
Time               16.16
INSTRUM            spect
PROBHD   5 mm Multinucl
PULPROG          zgpg30
TD                65536
SOLVENT           CDCl3
NS                17408
DS                    4
SWH           30030.029 Hz
FIDRES         0.458222 Hz
AQ            1.0912244 sec
RG               2580.3
DW                16.650 usec
DE                12.00 usec
TE                300.2 K
D1           2.00000000 sec
d11          0.03000000 sec
DELTA        1.89999998 sec
MCREST       0.00000000 sec
MCWRK        0.01500000 sec

======== CHANNEL f1 ========
NUC1                13C
P1                12.00 usec
PL1                3.00 dB
SFO1        125.7427020 MHz

======== CHANNEL f2 ========
CPDPRG2          waltz16
NUC2                1H
PCPD2            100.00 usec
PL2               -1.00 dB
PL12              18.80 dB
PL13              22.50 dB
SFO2        500.0220001 MHz

F2 - Processing parameters
SI                 32768
SF          125.7300987 MHz
WDW                EM
SSB                 0
LB                1.00 Hz
GB                  0
PC                1.40
```




```
Current Data Parameters
NAME                  Rs-2-152
EXPNO                        8
PROCNO                       1

F2 - Acquisition Parameters
Date_                 20080225
Time                     21.47
INSTRUM                  spect
PROBHD    5 mm Multinucl
PULPROG             noesygpph
TD                        2048
SOLVENT                  CDCl3
NS                           45
DS                           16
SWH                  5122.951 Hz
FIDRES               2.501441 Hz
AQ                   0.1999348 sec
RG                       912.3
DW                      97.600 usec
DE                       6.00 usec
TE                      300.2 K
d0                   0.00007876 sec
D1                   2.00000000 sec
D8                   2.00000000 sec
D16                  0.00020000 sec
IN0                  0.00019520 sec
MCREST               0.00000000 sec
MCWRK                2.00000000 sec
TAU                  0.99879998 sec

======== CHANNEL f1 ========
NUC1                        1H
P1                       14.80 usec
P2                       29.60 usec
PL1                      -1.00 dB
SFO1                500.0223501 MHz

====== GRADIENT CHANNEL =====
GPNAM1                SINE.100
GPNAM2                SINE.100
GPX1                     0.00 %
GPX2                     0.00 %
GPY1                     0.00 %
GPY2                     0.00 %
GPZ1                    40.00 %
GPZ2                   -40.00 %
P16                   1000.00 usec

F1 - Acquisition parameters
ND0                          1
TD                         214
SFO1                500.0224 MHz
FIDRES              23.939022 Hz
SW                      10.245 ppm
FnMODE                    TPPI

F2 - Processing parameters
SI                        1024
SF                  500.0200216 MHz
WDW                      QSINE
SSB                          2
LB                        0.00 Hz
GB                           0
PC                        1.00

F1 - Processing parameters
SI                        1024
MC2                       TPPI
SF                  500.0200216 MHz
WDW                      QSINE
SSB                          2
LB                        0.00 Hz
GB                           0
```




```
Current Data Parameters
NAME          Rs-2-152A1
EXPNO                  5
PROCNO                 1

F2 - Acquisition Parameters
Date_           20100410
Time                9.18
INSTRUM            spect
PROBHD   5 mm Multinucl
PULPROG         cosygpqf
TD                 2048
SOLVENT             DMSO
NS                     8
DS                     8
SWH             4496.403 Hz
FIDRES          2.195509 Hz
AQ             0.2277876 sec
RG                2298.8
DW              111.200 usec
DE                6.00 usec
TE                300.2 K
d0            0.00000300 sec
D1            1.48689198 sec
d13           0.00000400 sec
D16           0.00020000 sec
IN0           0.00022221 sec
MCREST        0.00000000 sec
MCWRK         1.48689198 sec

======== CHANNEL f1 ========
NUC1                  1H
P0                14.80 usec
P1                14.80 usec
PL1               -1.00 dB
SFO1         500.0220001 MHz

====== GRADIENT CHANNEL =====
GPNAM1          SINE.100
GPNAM2          SINE.100
GPX1                0.00 %
GPX2                0.00 %
GPY1                0.00 %
GPY2                0.00 %
GPZ1               10.00 %
GPZ2               10.00 %
P16              1000.00 usec

F1 - Acquisition parameters
ND0                    1
TD                   512
SFO1            500.022 MHz
FIDRES          8.789447 Hz
SW                9.000 ppm
FnMODE                QF

F2 - Processing parameters
SI                  1024
SF           500.0200216 MHz
WDW                 SINE
SSB                    0
LB                 0.00 Hz
GB                     0
PC                 1.40

F1 - Processing parameters
SI                  1024
MC2                   QF
SF           500.0200216 MHz
WDW                 SINE
SSB                    0
LB                 0.00 Hz
GB                     0
```