Supporting Information

For

Approaches to *N*-Methylwelwitindolinone C Isothiocyanate: Facile Synthesis of the Tetracyclic Core

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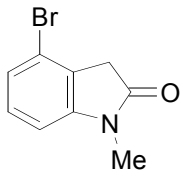
Experimental Section

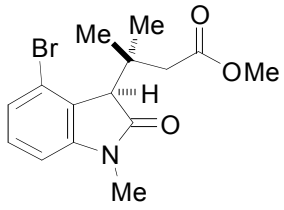
General Melting points were determined using a Thomas-Hoover Uni-melt capillary melting point apparatus and are uncorrected. Infrared (IR) spectra were recorded on a Perkin-Elmer 1600 series FTIR spectrometer and are reported in wavenumbers (cm^{-1}) Oils were analyzed as neat films on sodium chloride plates, and solids were analyzed as solutions in the solvent indicated. Proton nuclear magnetic resonance (^1H NMR) spectra were measured on Varian 500 series (500 MHz) or a Varian Mercury-400 (400 MHz) spectrometer. Carbon magnetic resonance (^{13}C NMR) spectra were measured using the above instruments operating at 126 or 101 MHz, respectively. ^1H NMR and ^{13}C NMR spectra were recorded for samples in $CDCl_3$ and are reported in parts per million (ppm) downfield (δ) using residual chloroform ($CHCl_3$) as an internal standard set to δ 7.26 and δ 77.0 respectively. Proton NMR data are reported in the form: δ (multiplicity [s, singlet; d, doublet; t, triplet; q, quartet; p, pentet; m, multiplet; comp, complex multiplet; br, broad; app, apparent], coupling constants, number of protons). Low resolution chemical ionization mass spectral data (CI) were recorded on a Finnigan TSQ-70 mass spectrometer, and high resolution chemical ionization mass spectra (HRMS) were obtained on a VG ZAB2-E instrument. Analytical thin layer chromatography (TLC) was performed on glass-backed silica gel plates precoated (0.25 mm thick) with 60 F_{254}. Compounds were visualized under UV light and/or staining with ethanolic *p*-anisaldehyde or basic aqueous potassium permanganate. Liquid chromatography was performed using forced flow (flash chromatography) of the indicated solvent system on 230-400 mesh silica gel (E. Merck reagent silica gel 60).

All reagents were reagent grade and used as received unless noted otherwise. Diethyl ether (Et_2O), tetrahydrofuran (THF) and dimethylformamide (DMF) were dried by filtration through alumina according to the procedure described by Grubbs (Pangborn, A. B.; Giardello, M. A.; Grubbs, R. H.; Rosen, R. K.; Timmers, F. J. *Organometallics* **1996**, *15*, 1518-1520.) Toluene was additionally deoxygenated by passing through activated Q5. Triethylamine (Et_3N), diisopropylamine ($^iPr_2NH$), methylene chloride (CH_2Cl_2), allyl bromide, and 1,8-diazabicyclo[5.4.0]undec-7-ene (DBU) were distilled from CaH_2 under nitrogen immediately before use. Xylenes was distilled from CaH_2 under nitrogen and stored over 4Å molecular sieves. Methanol (MeOH) was degassed with a stream of argon, then distilled from CaH_2 and stored under nitrogen.



7

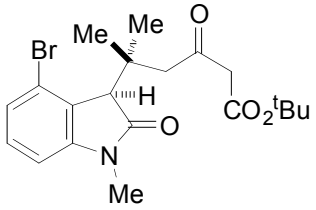
4-Bromo-1-methyl-1,3-dihydro-2-oxindole (7). A suspension of sodium hydride (331 mg, 8.63 mmol, 60% in oil) and xylenes (17.3 mL) was heated to 130 °C. After 15 min, 4-bromo-1,3-dihydro-2-oxindole (**8**, 1.83 g, 8.63 mmol) was added in ~10 equal portions over 5 min. The resulting light orange suspension was heated under reflux for 1 h. Dimethylsulfate (0.818 mL, 8.63 mmol) was added slowly dropwise. The suspension effervesced during addition, then quickly became clear and orange. After 1 h, the reaction was cooled and diluted with EtOAc (30 mL). The organic layer was washed with water (3 x 15 mL), brine (15 mL), dried ($MgSO_4$) and concentrated to provide a yellow solid. The product was purified by flash column chromatography eluting with a solvent gradient (CH_2Cl_2 to 10% Et_2O in CH_2Cl_2) to give 1.29 g (66%) of 7 as a fluffy light yellow solid: mp 138-139 °C; R_f 0.49 (50% EtOAc in hexanes), 0.49 (5% Et_2O in CH_2Cl_2); ^1H NMR (500 MHz, $CDCl_3$) δ 7.15 - 7.10 (comp, 2 H), 6.73 - 6.69 (m, 1 H), 3.43 (s, 2 H), 3.17 (s, 3 H); ^{13}C NMR (126 MHz, $CDCl_3$) δ 173.7, 146.0, 129.4, 125.4, 125.3, 119.0, 106.8, 37.0, 26.5; IR ($CHCl_3$) 3054, 2939, 1717, 1611, 1458, 1340, 1296, 1106, 925, 770, 708 cm^{-1}; mass spectrum (CI) *m/z* 225.9875 [C_9H_9NOBr $(M+H)^+$ requires 225.9868] (base), 175, 147.



9

Methyl-3-(4-bromo-1-methyl-2,3-dihydro-2-oxindol-3-yl)-3-methylbutyrate (9). A suspension of **7** (264 mg, 1.17 mmol), MeOH (0.779 mL) and methyl 3,3-dimethylacrylate (0.765 mL, 5.85 mmol) was treated with sodium methoxide (63.2 mg, 1.17 mmol). A significant portion of the solids dissolved, and the suspension was heated to 55 °C. This yellow reaction turned green after 1 h, then blue after 2 h. After 40 h of heating, the reaction was cooled, and 1 M aqueous HCl (~ 2 mL) and water (15 mL) were added. The mixture was stirred for 5 min and extracted with EtOAc (3 x 20 mL). The combined organic layers were dried ($MgSO_4$) and concentrated to provide an orange oil. The

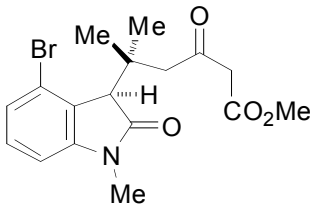
residue was purified by flash column chromatography eluting with 10% EtOAc in benzene (mixed fractions rechromatographed twice) to give 236 mg (62%) of **9** as a light yellow solid: mp 75-77 °C; R_f 0.63 (50% EtOAc in hexanes), 0.38 (10% EtOAc in benzene); ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.13 (dd, J = 7.9, 1.0 Hz, 1 H), 7.08 (td, J = 7.9, 1.0 Hz, 1 H), 6.68 (dd, J = 7.9, 1.0 Hz, 1 H), 3.82 (s, 1 H), 3.66 (s, 3 H), 3.09 (s, 3 H), 2.92 (A of AB, J_{AB} = 15.1 Hz, 1 H), 2.49 (B of AB, J_{AB} = 15.1 Hz, 1 H), 1.31 (s, 3 H), 0.87 (s, 3 H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 176.4, 172.4, 147.2, 129.4, 127.9, 126.6, 120.9, 106.4, 54.0, 51.2, 43.3, 39.6, 27.6, 25.9, 24.2; IR (neat) 2950, 2882, 1713 (br), 1603, 1454, 1330, 1172, 1100, 932, 767 cm^{-1}; mass spectrum (CI) m/z 340.0554 [$C_{15}H_{19}NO_3Br$ $(M+H)^+$ requires 340.0548], 310, 308, 268, 266, 256, 254, 228, 226 (base).



10

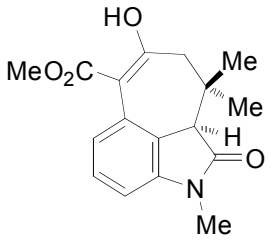
***tert*-Butyl 5-(4-bromo-1-methyl-2,3-dihydro-2-oxindol-3-yl)-5-methyl-3-oxohexanoate (10).** A solution of ^{i}Pr$_2$NH (97.5 μL, 0.696 mmol) and THF (4.46 mL) was cooled to –78 °C and treated with ^{n}BuLi (0.278 mL, 2.50 M in hexanes, 0.696 mmol). After 1 min, the reaction was warmed to 0 °C and stirred 30 min. The clear, colorless solution was cooled to –50 °C and treated with a solution of ^{t}BuOAc (93.8 μL, 0.696 mmol) and THF (0.446 mL) dropwise. The reaction was stirred for 1 h between –45 °C to –30 °C, and a solution of **9** (39.3 mg, 0.116 mmol) was added dropwise. The reaction was stirred for 1 h between –45 °C to –30 °C and then slowly warmed to room temperature over the next 1 h. Saturated aqueous NH_4Cl (5 mL) and water (20 mL) were added, and the mixture was extracted with Et_2O (3 x 20 mL). The combined organic layers were dried ($MgSO_4$) and concentrated to provide a yellow oil. The residue was purified by flash column chromatography eluting with a solvent gradient (10% Et_2O in hexanes to 40% Et_2O in hexanes) to give 35.3 mg (72%) of **10** as a light yellow oil: R_f 0.54 (40% EtOAc in hexanes); ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.20 - 7.08 (comp, 2 H), 6.71 (d, J = 7.3 Hz, 1 H), 3.94 (s, 1 H), 3.44 (A of AB, J_{AB} = 15.3 Hz, 1 H), 3.39 (B of AB, J_{AB} = 15.3 Hz, 1 H), 3.17 (A of AB, J_{AB} = 17.8 Hz, 1 H), 3.11 (s, 3 H), 2.70 (B of AB, J_{AB} = 17.8 Hz, 1 H), 1.47 (s, 9 H), 1.40 (s, 3 H), 0.79 (s, 3 H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 202.2, 176.6, 166.5, 147.1, 129.4, 128.1, 126.7, 120.7, 106.4,

81.7, 53.1, 52.0, 51.3, 39.5, 28.4, 28.0, 25.9, 23.9; IR (neat) 2973, 1713 (br), 1603, 1455, 1368, 1327, 1251, 1158, 1101, 932, 768 cm^{-1}; mass spectrum (CI) *m/z* 424.1109 [$C_{20}H_{27}NO_4Br$ $(M+H)^+$ requires 424.1124] (base), 370, 368, 352, 350, 171.



6

Methyl 5-(4-bromo-1-methyl-2,3-dihydro-2-oxindol-3-yl)-5-methyl-3-oxohexanoate (6). Neat **10** (142 mg, 0.335 mmol) was cooled to 0 °C, and trifluoroacetic acid (0.669 mL) was added. After 1 h, the reaction was concentrated via rotovap (rt water bath), and the residue was dissolved in Et_2O (0.669 mL). The solution was cooled to 0 °C, and an ethereal solution of diazomethane (~0.3M) was added utilizing a flame polished pipette until the yellow color persisted. The reaction was quenched with 1 M aqueous HCl (5 mL), stirred 5 min, then extracted into Et_2O (3 x 5 mL). The combined organic layers were dried ($MgSO_4$) and concentrated to provide a clear light yellow oil. The residue was purified by flash column chromatography eluting with a solvent gradient (20% EtOAc in hexanes to 40% EtOAc in hexanes) to give 121 mg (95%) of **6** as a clear light yellow oil: R_f 0.24 (30% EtOAc in hexanes); ^1H NMR (300 MHz, $CDCl_3$) δ 7.20 - 7.08 (comp, 2 H), 6.71 (dd, J = 7.3, 1.0 Hz, 1 H), 3.95 (s, 1 H), 3.75 (s, 3 H), 3.55 (s, 2 H), 3.23 (A of AB, J_{AB} = 17.9 Hz, 1 H), 3.10 (s, 3 H), 2.63 (B of AB, J_{AB} = 17.9 Hz, 1 H), 1.43 (s, 3 H), 0.74 (s, 3 H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 201.8, 176.6, 167.7, 147.1, 129.4, 128.1, 126.7, 120.7, 106.5, 52.8, 52.3, 51.4, 50.5, 39.5, 28.8, 26.9, 23.9; IR (neat) 2963, 2881, 1747, 1714 (br), 1603, 1454, 1329, 1102, 932, 768 cm^{-1}; mass spectrum (CI) *m/z* 384, 382.0654 [$C_{17}H_{21}NO_4Br$ $(M+H)^+$ requires 382.0654] (base), 352, 350, 304.



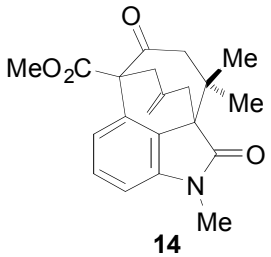
11

Catalysis with [(^{t}Bu$_3$P)$_2$Pd]. Aryl bromide **6** (28.5 mg, 74.6 μmol), *bis*-(tri-*tert*-butylphosphine)palladium (0) (7.6 mg, 14.9 μmol), *bis*-dibenzylidenepalladium (0) (4.3 mg, 7.46 μmol) and sodium *tert*-butoxide (7.9 mg, 82.1 μmol) were added to a dry Kjeldahl-shaped Schlenk flask. The flask was evacuated and backfilled with argon three times, and DMF (0.522 mL) was added. The brown suspension was deoxygenated via a freeze-pump-thaw protocol (3 cycles, 20 min each), and then heated to 75 °C (oil bath temperature). After 3 h, the reaction was cooled, 1 M aqueous HCl (5 mL) was added, and the mixture was extracted with EtOAc (3 x 5 mL). The combined organic layers were dried ($MgSO_4$) and concentrated. The residue was purified by flash column chromatography eluting with a solvent gradient (10% EtOAc in hexanes to 30% EtOAc in hexanes) to give 19.9 mg (88%) of **11** as a white solid.

Catalysis with [^{t}Bu$_2$P(2-biphenyl)]. Aryl bromide **6** (24.2 mg, 63.3 μmol), 2-(di-*tert*-butylphosphino)biphenyl (**27**, 8.3 mg, 27.9 μmol) and sodium *tert*-butoxide (6.7 mg, 69.6 μmol) were added to a dry Kjeldahl-shaped Schlenk flask. The flask was evacuated and backfilled with argon three times and DMF (0.443 mL) was added. The orange suspension was deoxygenated via a freeze-pump-thaw protocol (3 cycles, 20 min each), then heated to 75 °C (oil bath temperature) for 4.5 h. The black reaction was quenched with 1 M aqueous HCl (5 mL) and extracted with EtOAc (3 x 5 mL). The combined organic layers were dried ($MgSO_4$) and concentrated. The residue was purified by flash column chromatography eluting with a solvent gradient (10% EtOAc in hexanes to 30% EtOAc in hexanes) to give 11.0 mg (58%) of **11** as a white solid.

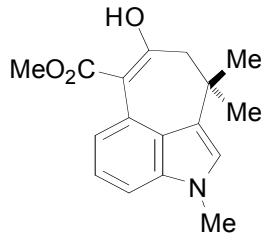
Methyl 7-Hydroxy-2,9,9- trimethyl-1-oxo-2,8,9,9a-tetrahydro-2-azabenzo[cd]azulene-6-carboxylate (11). mp 114-117 °C; R_f 0.56 (40% EtOAc in hexanes); ^{1}H NMR (500 MHz, $CDCl_3$) δ 13.1 (d, *J* = 1.0 Hz, 1 H), 7.25 (td, *J* = 7.8, 0.7 Hz, 1 H), 7.11 (dd, *J* = 7.8, 0.7 Hz, 1 H), 6.66 (d, *J* = 7.8 Hz, 1 H), 3.79 (s, 3 H), 3.16 (s, 3 H), 2.98 (s, 1 H), 2.20 (d, *J* = 12.6 Hz, 1 H), 2.06 (d, *J* = 12.6 Hz, 1 H), 1.51 (s, 3 H), 0.88 (s, 3 H); ^{13}C NMR (126 MHz, $CDCl_3$) δ 177.5, 176.1, 171.7, 143.6, 131.3, 127.5, 126.8, 123.4, 105.5, 101.3, 52.5, 51.8, 48.3, 48.2, 28.6, 26.0, 24.8; IR (neat) 3016, 2928, 2855, 1703,

1644, 1604, 1444, 1370, 1334, 1296, 1236, 770 cm^{-1}; mass spectrum (CI) *m/z* 302.1382 [$C_{17}H_{20}NO_4$ $(M+H)^+$ requires 302.1392] (base), 286, 270.



14

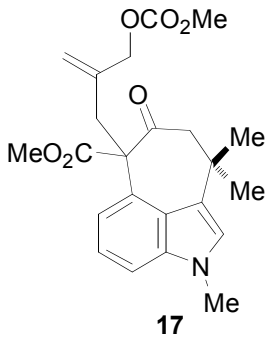
Oxindole-bridged[2.3.3]bicycle 14. To a solution of **11** (14.2 mg, 47.1 μmol) and THF (0.236 mL) was added DBU (8.0 μL, 51.8 μmol). To this orange solution was added carbonate **12** (9.0 μL, 51.8 μmol) and this mixture was added to a solution of tetrakistriphenylphosphine palladium (5.4 mg, 47.1 μmol) and THF (0.236 mL) over 10 min. After 2 h, the reaction was concentrated under reduced vacuum, and the residue was purified by flash chromatography eluting with a solvent gradient (10% EtOAc in hexanes to 20% EtOAc in hexanes) to afford 9.8 mg (59%) of **14** as a white solid: R_f 0.29 (30% EtOAc in hexanes); ^1H NMR (500 MHz) δ 7.29 (t, *J* = 7.8 Hz, 1 H), 6.82 (dd, *J* = 7.8, 0.8 Hz, 1 H), 6.56 (dd, *J* = 7.8, 0.8 Hz, 1 H), 4.91 (br s, 1 H), 4.86 (br s, 1 H), 3.83 (s, 3 H), 3.31 (d, *J* = 13.8 Hz, 1 H), 3.22 (s, 3 H), 2.92 (d, *J* = 14.9 Hz, 1 H), 2.65 (d, *J* = 12.1 Hz, 1 H), 2.53 (d, *J* = 13.8 Hz, 1 H), 2.15 (d, *J* = 12.1 Hz, 1 H), 2.13 (d, *J* = 14.9 Hz, 1 H), 1.44 (s, 3 H), 0.78 (s, 3 H); ^{13}C (126 MHz) δ 208.0, 178.6, 171.1, 143.2, 141.7, 137.3, 129.2, 129.0, 119.9, 118.8, 107.3, 67.2, 56.9, 54.4, 52.6, 45.1, 41.6, 38.7, 28.3, 26.1, 22.9; IR (neat) 3018, 2955, 1746, 1707, 1607, 1470, 1246 cm^{-1}; MS (CI) *m/z* 354.1701 [$C_{21}H_{24}NO_4$ $(M+H)^+$ requires 354.1705] (base), 322.



16

Methyl 7-hydroxyl-2,9,9-trimethyl-8,9-dihydro-2*H*-2-aza-benzo(cd)azulene-6-carboxylate (16). A solution of **11** (45.8 mg, 0.152 mmol) and CH_2Cl_2 (1.52 mL) was cooled to −78 ˚C and Dibal

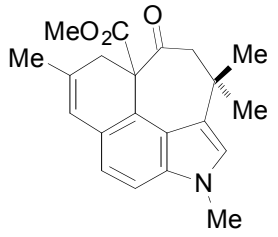
(0.258 mL, 0.258 mmol, 1 M in CH_2Cl_2) was added slowly dropwise. After 2 h, 1 M aqueous HCl (5 mL) and CH_2Cl_2 (5 mL) was added and the mixture was stirred vigorously for 1 h. The layers were separated and the aqueous layer was extracted with CH_2Cl_2 (2 x 5 mL). The combined organic layers were dried ($MgSO_4$) and concentrated to afford a residue that was purified by flash chromatography eluting with a solvent gradient (5% EtOAc in hexanes to 20% EtOAc in hexanes) to afford 12.2 mg of starting material **11** (27% recovery) and 19.7 mg (45%) of **16** as a white solid: mp 143-145 °C (from Et_2O/CH_2Cl_2); R_f 0.36 (20% Et_2O in hexanes); ^{1}H NMR (500 MHz, $CDCl_3$) δ 13.6 (s, 1 H), 7.26 (dd, *J* = 7.8, 1.2 Hz, 1 H), 7.21 (t, *J* = 7.8 Hz, 1 H), 7.15 (dd, *J* = 7.8, 1.2 Hz, 1 H), 6.86 (s, 1 H), 3.84 (s, 3 H), 3.74 (s, 3 H), 2.88 (br d, *J* = 13.1 Hz, 1 H), 2.45 (br d, *J* = 13.1 Hz, 1 H), 1.46 (br s, 3 H), 1.34 (br s, 3 H); ^{13}C NMR (126 MHz, $CDCl_3$) δ 176.3, 173.9, 137.0, 125.4, 125.3, 124.2, 123.3, 121.5, 120.8, 106.9, 102.2, 51.9, 50.2, 34.1, 32.7, 31.5, 29.1; IR (neat) 3008, 2964, 1634, 1594, 1440, 1372, 1332, 1300, 1236, 1074, 1032 cm^{-1}; mass spectrum (CI) *m/z* 286.1445 [$C_{17}H_{20}NO_3$ $(M+H)^+$ requires 286.1443] (base), 254.



17

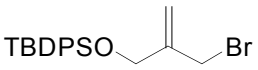
Methyl 6-[2-(methoxycarbonyloxymethyl)prop-2-enyl)-2,9,9-trimethyl-7-oxo-6,7,8,9-tetrahydro-2*H*-2-azabenzo[cd]azulene-6-carboxylate (17). A solution of **16** (7.8 mg, 27 μmol), THF (0.27 mL), carbonate **12** (5.0 μL, 30 μmol), and $(Ph_3P)_4Pd$ (0) (3.1 mg, 2.7 μmol) was stirred for 15 min. EtOAc (5 mL) was added, and the mixture was washed with H_2O (2x5 mL), brine (5 mL), dried ($MgSO_4$) and concentrated to afford a residue that was purified by flash chromatography eluting with a solvent gradient (20% EtOAc in hexanes to 30% EtOAc in hexanes) to afford 8.7 mg (79%) of **17** as a clear colorless residue: R_f 0.15 (20% EtOAc in hexanes); ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.26 (dd, *J* = 8.1, 1.1 Hz, 1 H), 7.21 (t, *J* = 7.8 Hz, 1 H), 7.04 (dd, *J* = 7.2, 1.1 Hz, 1 H), 6.98 (s, 1 H), 5.04 (d, *J* = 1.3 Hz, 1 H), 4.87 (s, 1 H), 4.07 (d, *J* = 13.7 Hz, 1 H), 4.00 (d, *J* = 13.7 Hz, 1 H), 3.77 (s, 3 H), 3.72 (s, 1 H), 3.59 (s, 3 H), 3.35 (d, *J* = 14.4 Hz, 1 H), 3.16 (d, *J* = 14.4 Hz, 1 H), 3.08 (d, *J* = 10.9 Hz, 1 H), 2.83 (d, *J*

= 10.9 Hz, 1 H), 1.42 (S, 3 H), 1.36 (s, 3 H); ^{13}C NMR (126 MHz, $CDCl_3$) δ 207.6, 172.3, 155.2, 139.3, 137.5, 129.1, 125.3, 124.1, 123.8, 121.7, 118.6, 118.1, 108.9, 70.2, 69.8, 58.2, 54.6, 52.8, 43.4, 33.0, 32.9, 32.0, 31.8; IR (neat) 2958, 1745, 1697, 1444, 1266, 1239, 981, 750 cm^{-1}; mass spectrum (CI) *m/z* 413.1836 [$C_{23}H_{27}NO_6$ $(M+H)^+$ requires 413.1838], 338 (base), 320, 278.



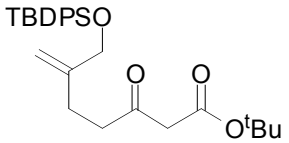
18

Methyl 2,6,10,10-Tetramethyl-8-oxo-2,8,9,10-tetrahydro-7*H*-2-azanaphtho[2,1,8-cde]azulene-7a-carboxylate (18). A solution of **17** (4.3 mg, 10.4 μmol) and $ZnCl_2$ (31 μL, 31 μmol, 1.0 M in Et_2O) in CH_2Cl_2 (0.21 mL) was heated to 60 ˚C in a sealed flask for 2 d. The reaction was diluted with EtOAc (2 mL), and the mixture was washed with H_2O (2 x 2 mL), brine (2 mL). The organic layer was dried ($MgSO_4$) and concentrated, and the residue was purified by flash chromatography eluting with a solvent gradient (10% EtOAc in hexanes to 20% EtOAc in hexanes) to afford 1.7 mg (51%) of **18** as a clear colorless oil: R_f 0.44 (30% EtOAc in hexanes); ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.20 (d, *J* = 8.4 Hz, 1 H), 7.02 (d, *J* = 8.4 Hz, 1 H), 6.96 (s, 1 H), 6.33 (dq, *J* = 4.2, 2.1 Hz, 1 H), 3.74 (s, 3 H), 3.61 (s, 3 H), 3.09 (d, *J* = 10.5 Hz, 1 H), 2.89 (ddq, *J* = 16.8, 4.2, 2.1 Hz, 1 H), 2.72 (d, *J* = 16.8 Hz, 1 H), 2.62 (d, *J* = 10.4 Hz, 1 H), 1.95 (t, *J* = 2.1 Hz, 3 H), 1.49 (s, 3 H), 1.28 (s, 3 H); ^{13}C NMR (126 MHz, $CDCl_3$) δ 203.4, 172.6, 136.5, 132.7, 127.2, 126.2, 124.2, 123.8, 122.9, 121.2, 120.0, 109.0, 66.3, 54.9, 52.9, 35.3, 33.1, 32.9, 32.6, 32.4, 22.9; IR (neat) 2952, 1727, 1712, 1473, 1434, 1227, 1160 cm^{-1}; mass spectrum (CI) *m/z* 338.1752 [$C_{21}H_{24}NO_3$ $(M+H)^+$ requires 338.1756] (base), 320, 278.



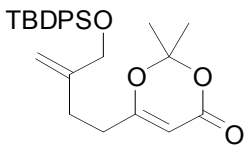
24

2-(*tert*-Butyl-diphenylsilanyloxymethyl)prop-2-en1-yl bromide (24). Triphenylphosphine (0.5263 g, 2.007 mmol) and carbon tetrabromide (0.6635 g, 2.000 mmol) were added to a stirred solution of the corresponding allylic alcohol (0.6615 g, 2.026 mmol) in CH_2Cl_2 (8.0 mL) at 0 ˚C. The reaction mixture was stirred for 40 min and then concentrated *in vacuo*. The crude solid was purified via flash chromatography, eluting with 10% EtOAc in hexanes, to afford 0.8644 g (99%) of **24** as a colorless oil: R_f = 0.51 (10% EtOAc in hexanes); ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.67 (dd, *J* = 8.0, 1.4 Hz, 4 H), 7.44–7.36 (m, 6 H), 5.29 (dd, *J* = 3.0, 1.6 Hz, 1 H), 5.27–5.26 (m, 1 H), 4.29 (t, *J* = 1.3 Hz, 2 H), 4.01 (s, 2 H), 1.06 (s, 9 H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 144.4, 135.5, 133.3, 129.7, 127.7, 114.9, 64.2, 32.7, 26.8, 19.3; IR 3070, 2958, 2930, 2857, 1112 cm^{-1}; HRMS (CI, CH_4) *m/z* calcd for $C_{20}H_{26}OSiBr$ $(M + H)^+$ 389.0936, found 389.0921.



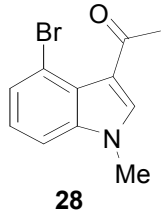
25

***tert*-Butyl 7-*tert*-butyldiphenylsilanyloxy-6-methylidene-hept-3-one-ate (25).** *n*BuLi (1.05 mL, 2.11 M in hexanes, 2.22 mmol) was added to a stirred solution of diisopropylamine (0.32 mL, 2.3 mmol) in THF (7.0 mL) at –78 ˚C. The solution was warmed to 0 ˚C (replaced the dry ice/isopropanol bath with ice), stirred for 40 min, and then *tert*-butylacetoacetate (**23**) (0.18 mL, 1.1 mmol) was added dropwise over 5 min. After 45 min the solution was cooled to –78 ˚C and the allylic bromide **24** (0.3591 g, 0.9222 mmol) in THF (3 mL) was added over 10 min. The reaction mixture was stirred for 1.25 h, warmed to rt (by removing the dry ice/isopropanol bath) and stirred for 2.5 h. Next the solution was poured into saturated NH_4Cl (40 mL) and the resultant aqueous mixture was extracted with EtOAc (3 × 30 mL). The combined organic extracts were washed with brine (40 mL), dried ($MgSO_4$), and concentrated *in vacuo*. The residue was purified by flash chromatography, eluting with 10% EtOAc in hexanes, afforded 0.3013 g (70%) of the product **25** as a colorless oil: R_f = 0.60 (40% EtOAc in hexanes); ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.69–7.66 (comp, 4 H), 7.46–7.36 (comp, 6 H), 5.18–5.17 (m, 1 H), 4.85 (dd, *J* = 2.7, 1.3 Hz, 1 H), 4.10 (s, 2 H), 3.32 (s, 2 H), 2.65 (t, *J* = 7.6 Hz, 2 H), 2.30 (t, *J* = 7.5 Hz, 2 H), 1.46 (s, 9 H), 1.07 (s, 9 H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 202.5, 166.4, 146.5, 135.5, 133.5, 129.7, 127.7, 109.3, 81.9, 66.4, 50.6, 41.1, 27.9, 26.8, 26.2, 19.2; IR (thin film) 3071 (C=C–H), 2931 (C–C–H), 1739 (C=O), 1716 (C=O) cm^{-1}; HRMS (CI, CH_4) *m/z* calcd for $C_{28}H_{39}O_4Si$ $(M + H)^+$ 467.2618, found 467.2615.



26

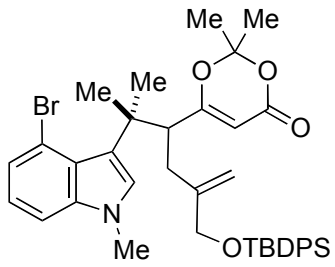
6-(4-*tert*-Butyldiphenylsilyloxy-2-methylidenebuyl)-2,2-dimethyl-4*H*-1,3-dioxin-4-one (26). The β-keto ester **25** (0.1766 g, 0.378 mmol) was dissolved in acetone (0.14 mL, 1.9 mmol), trifluoroacetic acid (0.29 mL, 3.76 mmol), and acetic anhydride (1.8 mL, 19 mmol). After standing for 22 h, saturated aqueous $NaHCO_3$ (60 mL) was added. The solution was then extracted with CH_2Cl_2 (4 × 15 mL), and the combined organic extracts were washed with brine (20 mL), dried (Na_2SO_4), and concentrated *in vacuo*. The residue was purified by flash chromatography, eluting with 30% EtOAc in hexanes to give 0.1135 g (67%) of **26** as a slightly yellow oil: R_f = 0.49 (40% EtOAc in hexanes); ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.66–7.64 (comp, 4 H), 7.44–7.36 (comp, 6 H), 5.17 (s, 1 H), 5.15 (d, *J* = 1.4 Hz, 1 H), 4.86 (d, *J* = 1.2 Hz, 1 H), 4.09 (s, 2 H), 2.35–2.32 (comp, 2 H), 2.31–2.24 (comp, 2 H), 1.63 (s, 6 H), 1.05 (s, 9 H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 171.2, 161.2, 145.9, 135.5, 133.3, 129.8, 127.7, 110.3, 106.3, 93.4, 66.3, 31.8, 28.7, 26.8, 25.0, 19.2; HRMS (CI, CH_4) *m/z* calcd for $C_{27}H_{35}O_4Si$ $(M + H)^+$ 451.2305, found 451.2320.



28

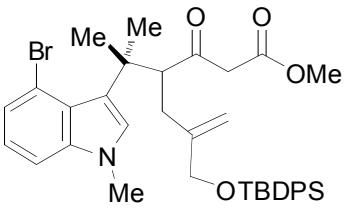
4-Bromo-3-acetyl-1-methyl-1*H*-indole (28). A solution of Me_2AlCl (3.0 mL of 1.0 M in hexanes, 3 mmol) was added dropwise over 10 min to a stirred solution of the 4-bromo-1-methylindole (0.308 g, 1.462 mmol) in CH_2Cl_2 (15 mL), cooled to 0 ˚C. Then reaction mixture was stirred for 45 min, and acetyl chloride (0.15 mL, 2.1 mmol) in CH_2Cl_2 (5 mL) was added over 15 min. After stirring for 1.5 h, pH 7 phosphate buffer (15 mL) was added, followed by H_2O (15 mL). The phases were separated, and the aqueous portion was extracted with CH_2Cl_2 (3 × 15 mL). The combined organic extracts were washed with brine (10 mL), dried (Na_2SO_4), and concentrated *in vacuo*. The product was purified via column chromatography, eluting with 70% EtOAc in hexanes, to afford 0.297 g (80%) of **28** as a white solid: R_f = 0.19 (60% EtOAc in hexanes); mp = 124–125 ˚C; ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.59 (s, 1 H), 7.44 (dd, *J* = 7.7, 0.9 Hz, 1 H), 7.22 (dd, *J* = 8.2, 0.8 Hz, 1 H), 7.08 (t, *J* = 7.9 Hz, 1 H), 3.76 (s, 3 H), 2.52 (s, 3 H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 192.1, 139.0, 136.1, 127.5, 125.2, 124.0, 118.2, 114.9,

108.9, 33.6, 29.9; IR 3106 (C=C–H), 1660 (C=O), 1106 (C–Br) cm^{-1}; HRMS (CI, CH_4) *m/z* calcd for $C_{11}H_{11}NOBr$ $(M + H)^+$ 252.0024, found 252.0032.

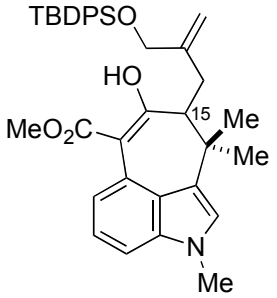


29

***O*-(*tert*-Butyldiphenylsilyl)-2-methylene-4-(2,2-dimethyl-4*H*-1,3-dioxin-4-one)-5-(4-bromo-1-methylindol-3-yl)-5-methyl-hexan-1-ol (29).** A solution of NaHMDS (11.7 mmol, 1.27 M in THF) was added to a stirred solution of the dioxanone **26** (1.78 g, 3.95 mmol) in THF (40 mL) at –78 ºC. After stirring for 45 min, TMSCl (2.5 mL, 19.7 mmol) was added, and the solution was stirred for 50 min. Then the mixture was warmed to rt, and concentrated *in vacuo*. The unpurified silyl ketene acetal **22** was then dissolved in PhMe (10 mL). In a separate flask, TMSOTf (0.35 mL, 1.93 mmol) was added to a solution of the crude indole **21** (1.08 g, 4.03 mmol) in PhMe (40 mL) at –78 ºC. After 2 min, the solution of **22** was added, and the mixture was stirred for 2.5 h. Then the reaction mixture was warmed to rt and stirred for 17 h before it was poured into saturated aqueous $NaHCO_3$ (70 mL) The phases were separated, and the aqueous portion was extracted with EtOAc (3 × 50 mL). The combined organic extracts were then washed with brine (50 mL), dried (Na_2SO_4), and concentrated *in vacuo*. The product was purified via flash chromatography, eluting with 20→35% EtOAc in hexanes to afford 0.96 g (35% from **26**) of **29** as a colorless oil: R_f = 0.39 (40% EtOAc in hexanes); ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.59–7.51 (m, 4 H), 7.40–7.31 (m, 7 H), 7.20 (dd, *J* = 8.2, 1.0 Hz, 1 H), 6.98 (t, *J* = 7.9 Hz, 1 H), 6.89 (s, 1 H, C2), 5.24 (br s, 1 H), 5.09 (s, 1 H), 4.82 (s, 1 H), 4.17 (d, *J* = 12.1 Hz, 1 H), 3.97 (br s, 1 H), 3.67 (s, 3 H), 2.25 (t, *J* = 13.3 Hz, 1 H), 1.68–1.36 (m, 14 H), 0.94 (s, 9 H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 172.6, 161.2, 145.4, 139.9, 135.5, 135.4, 133.54, 133.48, 129.62, 129.58, 127.6, 125.9, 122.4, 122.3, 113.7, 110.3, 109.0, 105.9, 66.0, 48.9, 38.0, 34.1, 33.0, 30.3, 26.7, 25.5, 24.9, 22.3, 19.2, 14.0; IR 2958, 2856, 1724, 1620, 1112 cm^{-1}; HRMS (CI, CH_4) *m/z* calcd for $C_{39}H_{47}O_4SiBr$ $(M + H)^+$ 700.2458, found 700.2462.



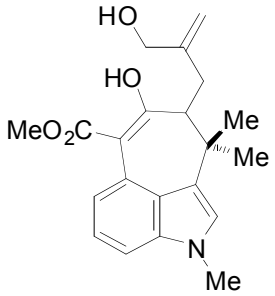
3-Oxo-4-[1',1'–dimethyl-1'(4-bromo-1-methylindol-3-yl)-methyl]-6-methylene-7-(*tert*-butyldiphenylsilyloxy)-heptanoate methyl ester. The dioxanone **29** (960 mg, 1.37 mmol) was heated in a stirred mixture of MeOH (5.5 mL) and PhMe (110 mL) to 110 C. After 21 h, the reaction mixture was cooled to rt and concentrated *in vacuo*. The residue was purified by flash chromatography, eluting with 25% EtOAc in hexanes to afford 871 mg (94%) of ketoester as an oil: R_f = 0.50 (40% EtOAc in hexanes); ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.64–7.52 (m, 4 H), 7.42–7.32 (m, 7 H), 7.26 (d, *J* = 7.9 Hz, 1 H), 7.04 (t, *J* = 7.9 Hz, 1 H), 6.92 (s, 1 H), 5.15 (s, 1 H), 4.84 (s, 1 H), 4.51 (dd, *J* = 11.8, 1.9 Hz, 1 H), 4.04 (br s, 2 H), 3.71 (s, 3 H), 3.58 (s, 3 H), 3.05 (br s, 2 H), 2.52–2.38 (m, 2 H), 1.70–1.46 (m, 7 H), 1.00 (s, 9 H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 207.0, 167.5, 146.3, 140.2, 135.7, 133.8, 129.8, 129.3, 127.9, 126.1, 123.0, 122.5, 122.0, 113.7, 110.6, 109.4, 66.1, 57.5, 52.0, 39.0, 38.1, 33.4, 27.0, 19.5; IR 2955, 2856, 1749, 1708, 1112, 1075 cm^{-1}; HRMS (CI, CH_4) *m/z* calcd for $C_{37}H_{44}O_4NSiBr$ $(M)^+$ 673.2223, found 673.2220.



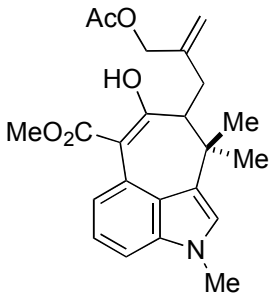
30

(*Z*)-Methyl 8,9-dihydro-7-hydroxy-8-(2-(*tert*-butyldiphenylsilyloxymethyl)allyl)-2,9,9-trimethyl-2*H*-cyclohepta[*cd*]indole-6-carboxylate (30). NaO*t*Bu (0.252 g, 2.624 mmol) was added to a solution of the above ketoester (890 mg, 1.319 mmol) in PhMe (20 mL). The solution was then degassed (freeze-pump-thaw × 2 cycles), and $Pd(OAc)_2$ (30.0 mg, 0.134 mmol) and tri-*tert*-butylphosphine (0.4 mL, 0.4 mmol, 1 M in PhMe) were added. The solution was partitioned into four microwave vials (previously purged with argon twice). Each vial was heated to 150 °C in the microwave for 5 min (80 W, 20 psi), and cooled to rt. The reaction mixtures were combined, added to 0.5 M HCl (30 mL), and extracted with EtOAc (3 × 30 mL). The combined organic extracts were washed with brine (20 mL), dried (Na_2SO_4), and concentrated *in vacuo*. The product was purified via column

chromatography, eluting with 25% EtOAc in hexanes to afford 602 mg (77%) of **31** as a colorless oil: R_f = 0.55 (40% EtOAc in hexanes); ^{1}H NMR (500 MHz, $CDCl_3$) δ 13.64 (s, 1 H), 7.67–7.60 (m, 4 H), 7.43–7.34 (m, 7 H), 7.19–7.13 (m, 2 H), 6.81 (s, 1 H), 5.04 (s, 1 H), 4.67 (s, 1 H), 4.12 (d, *J* = 10.0 Hz, 1 H), 4.03 (d, *J* = 10 Hz, 1 H), 3.83 (s, 3 H), 3.74 (s, 3 H), 2.68 (dd, *J* = 11.7, 3.3 Hz, 1 H), 1.98 (dd, *J* = 13.2, 3.3 Hz, 1 H), 1.62 (dd, *J* = 13.2, 11.7 Hz, 1 H), 1.56 (s, 3 H), 1.42 (s, 3 H), 1.01 (s, 9H).

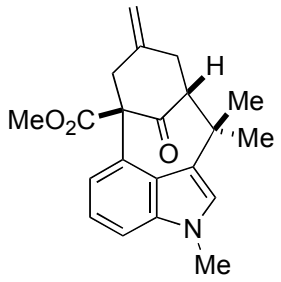


Methyl-8,9-dihydro-7-hydroxy-8-(2-(hydroxymethyl)allyl)-2,9,9-trimethyl-2*H*-cyclohepta[*cd*]indole-6-carboxylate. Triethylamine trihydrofluoride (1.6 mL, 9.8 mmol) was added to a stirred solution of **30** (602 mg, 1.013 mmol) and Et_3N (2.1 mL, 15 mmol) in MeCN (10 mL). After 20 h saturated $NaHCO_3$ (15 mL) and H_2O (15 mL) were added, and the aqueous portion was extracted with CH_2Cl_2 (3 × 25 mL). The combined organic extracts were washed with brine (20 mL), dried (Na_2SO_4), and concentrated *in vacuo*. The residue was purified by flash chromatography, eluting with 40% EtOAc in hexanes to give 240 mg (67%) of allylic alcohol as an oil: R_f = 0.28 (40% EtOAc in hexanes); ^{1}H NMR (400 MHz, $CDCl_3$) δ 13.64 (s, 1 H), 7.25 (dd, *J* = 7.4, 1.1 Hz, 1 H), 7.20 (t, *J* = 7.7 Hz, 1 H), 7.15 (dd, *J* = 7.4, 1.1 Hz, 1 H), 6.84 (s, 1 H), 4.94 (s, 1 H), 4.66 (s, 1 H), 4.02 (br s, 2 H), 3.84 (s, 3 H), 3.75 (s, 3 H), 2.73 (dd, *J* = 7.8, 2.4 Hz, 1 H), 1.98 (dd, *J* = 9.8, 2.4 Hz, 1 H), 1.65 (dd, *J* = 9.8, 7.8 Hz, 1 H), 1.48 (s, 3 H), 1.49 (br s , 1 H), 1.38 (s, 3 H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 178.6, 174.6, 147.2, 137.1, 125.0, 124.9, 123.9, 121.70, 121.66, 121.1, 112.4, 107.3, 102.3, 66.2, 58.5, 52.2, 36.7, 33.0, 32.1, 31.4, 28.5; IR 3416, 2952, 1741, 1633, 1588, 1298, 1224 cm^{-1}; HRMS (CI, CH_4) *m/z* calcd for $C_{21}H_{25}O_4N$ $(M)^+$ 355.1784, found 355.1786.



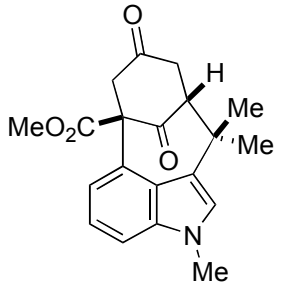
31

Methyl 4-(2-(acetoxymethyl)allyl)-5-hydroxy-1,3,3-trimethyl-3,4-dihydro-1H-cyclohepta[cd]indole-6-carboxylate (32). To a solution of the above alcohol (126 mg, 0.355 mmol) and collidine (50 μL, 0.38 mmol) in CH_2Cl_2 at -78 °C was added AcCl (25 μL, 0.36 mmol) dropwise. The solution was stirred for 3.5 h, whereupon 1 N aqueous HCl (30 mL) was added. The aqueous layer was extracted with CH_2Cl_2 (3 x 25 mL), and the organic layers were combined, dried (Na_2SO_4), and concentrated under reduced pressure. The residue was purified with column chromatography, eluting with 35% EtOAc in hexanes to give 109 mg (78%) of **31** as an oil



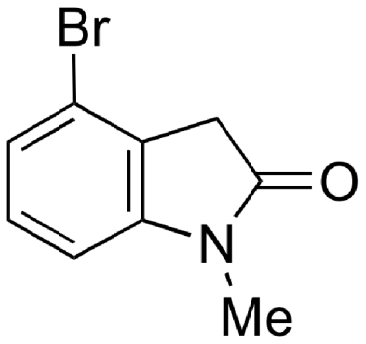
32

3,3-Dimethyl-4,8-methano-6-methylene-12-oxo-cyclonon[cd]-*N*-methyl-indole-8-carboxylate methyl ester (32). Sodium hydride (9.0 mg, 0.23 mmol, 60% dispersion in oil) was added to a solution of the allylic acetate **31** (82.3 mg, 0.207 mmol) in THF (6.0 mL). The solution was degassed (freeze-pump-thaw × 2 cycles) and purged with argon. The $Pd_2(dba)_3$ (8.7 mg, 9.5 μmol) was added, then the solution was heated to 50 °C and stirred for 17 h. After cooling to room temperature, 1 N HCl (20 mL) was added, and the aqueous mixture was extracted with CH_2Cl_2 (3 × 20 mL). The combined organic extracts were washed with brine (20 mL), dried (Na_2SO_4), and concentrated *in vacuo*. The crude product was purified by flash chromatography, eluting with 25% EtOAc in hexanes to furnish 49.9 mg (71%) of **32** as an oil: R_f = 0.51 (40% EtOAc in hexanes); ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.21–7.15 (m, 2 H), 6.92 (s, 1 H), 6.66 (dd, *J* = 6.8, 1.6 Hz, 1 H), 4.42 (s, 1 H), 4.29 (s, 1 H), 3.76 (s, 3 H), 3.73 (s, 3 H), 3.44 (d, *J* = 14.6 Hz, 1 H), 2.88 (d, *J* = 13.8 Hz, 1 H), 2.75 (dd, *J* = 8.9, 4.7 Hz, 1 H), 2.69 (dd, *J* = 15.0, 4.2 Hz, 1 H), 2.62 (dd, *J* = 14.8, 8.9 Hz, 1 H), 1.48 (s, 3 H), 1.23 (s, 3 H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 209.5, 173.1, 140.0, 136.9, 130.9, 126.5, 124.8, 122.1, 120.8, 117.7, 112.9, 108.0, 69.3, 61.3, 52.3, 48.8, 35.6, 34.3, 32.9, 28.8, 14.0; HRMS (CI, CH_4) *m/z* calcd for $C_{21}H_{24}O_3N$ $(M + H)^+$ 338.1756, found 338.1745.



33

3,3-Dimethyl-4,8-methano-6,12-oxo-cyclonon[c.d]-*N*-methyl-indole-8-carboxylate methyl ester (33). The OsO_4 (2.0 mg, 7.9 μmol) and $NaIO_4$ (97.5 mg, 0.456 mmol) were added to a solution of the olefin **32** (16.1 mg, 0.0477 mmol) in THF (2.0 mL) and H_2O (0.5 mL). After stirring for 22 h the solution was diluted with H_2O (15 mL) and extracted with CH_2Cl_2 (3 × 15 mL). The combined organic extracts were washed with brine (20 mL), dried (Na_2SO_4), and concentrated *in vacuo*. The crude product was purified by flash chromatography, eluting with 40% EtOAc in hexanes to give 10.7 mg (66%) of **33** as an oil: R_f = 0.40 (50% EtOAc in hexanes); ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.23–7.21 (m, 2 H), 7.00 (s, 1 H), 6.73 (dd, *J* = 5.8, 2.6 Hz, 1 H), 3.75 (s, 3 H), 3.73 (s, 3 H), 3.51 (dd, *J* = 17.9, 0.7 Hz, 1 H), 3.24 (d, *J* = 17.9 Hz, 1 H), 2.97 (dd, *J* = 9.5, 6.6 Hz, 1 H), 2.82–2.78 (m, 2 H), 1.43 (s, 3 H), 1.33 (s, 3 H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 206.9, 205.6, 171.6, 137.4, 127.9, 127.8, 123.7, 122.6, 119.4, 118.9, 109.1, 66.7, 57.4, 53.9, 52.9, 43.2, 37.0, 32.7, 29.3, 13.9; IR (thin film) 2923, 1737, 1732, 1713, 1454, 1246 cm^{-1}.



7

ss

rwhj i 53 c 1

exp1 s2pul

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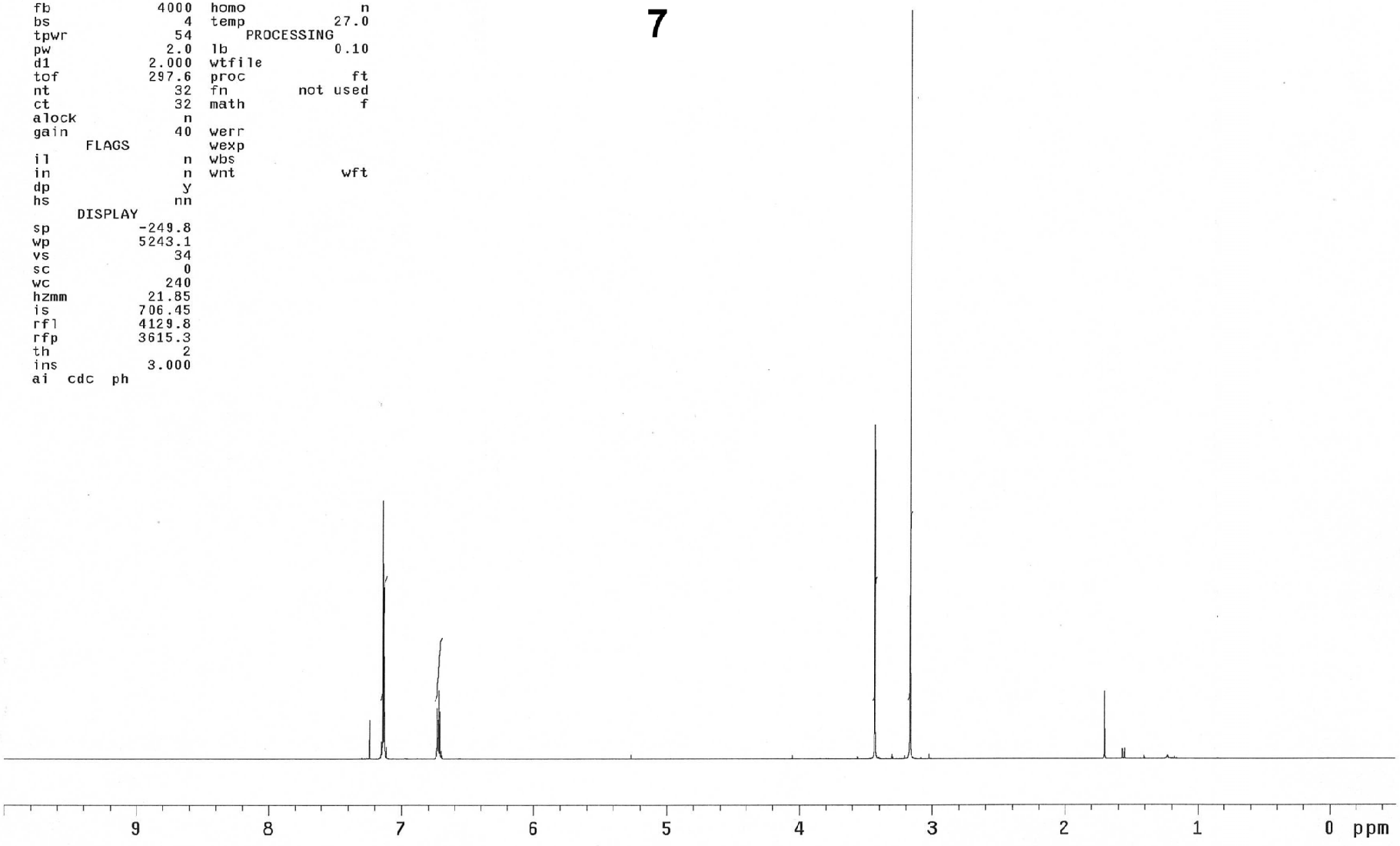
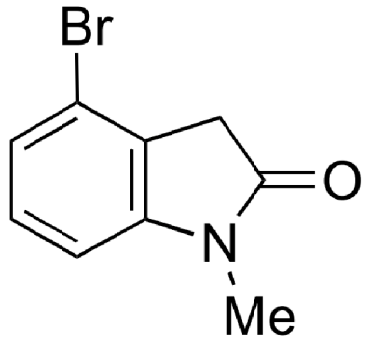
ins	3.000		

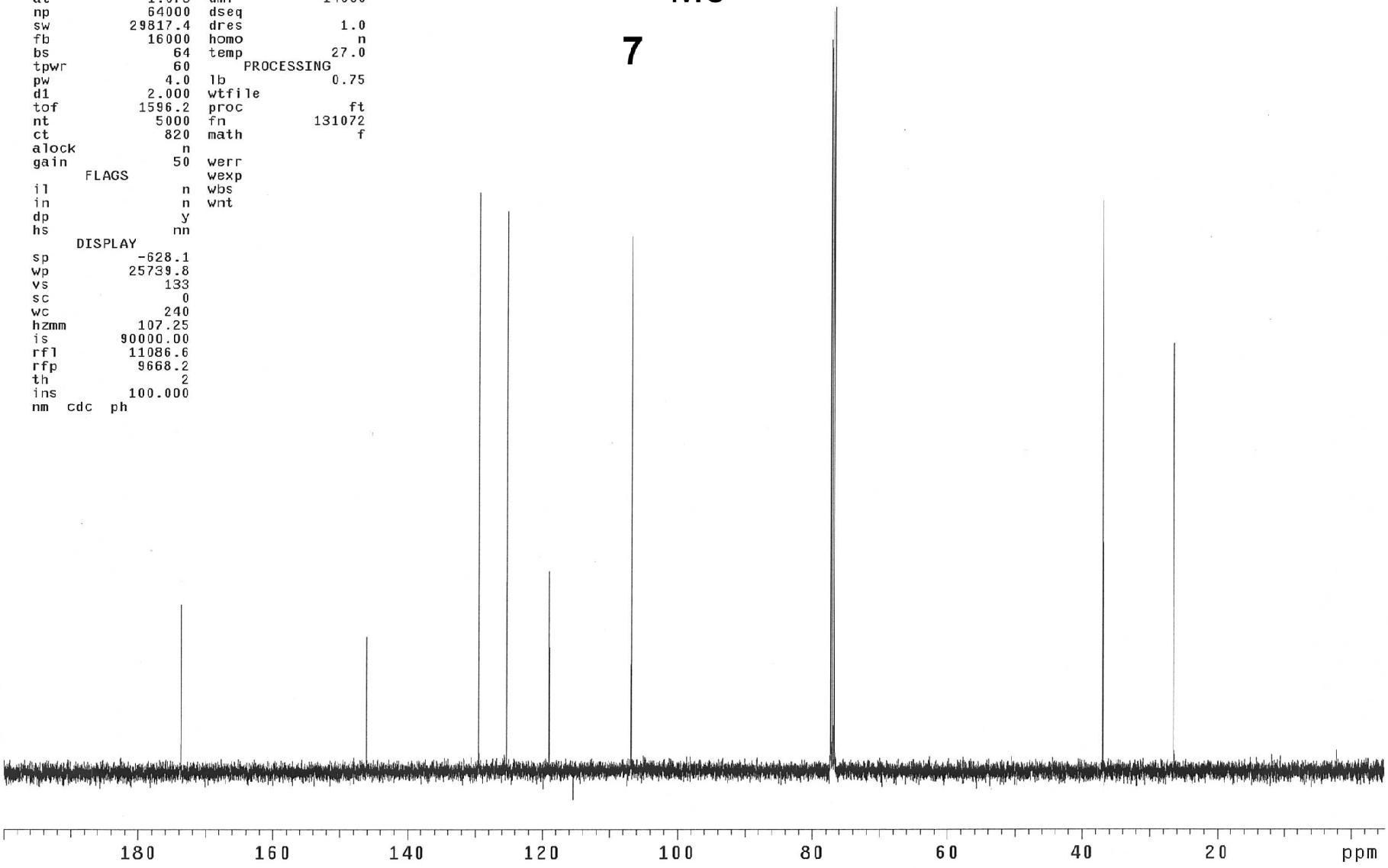
ai cdc ph

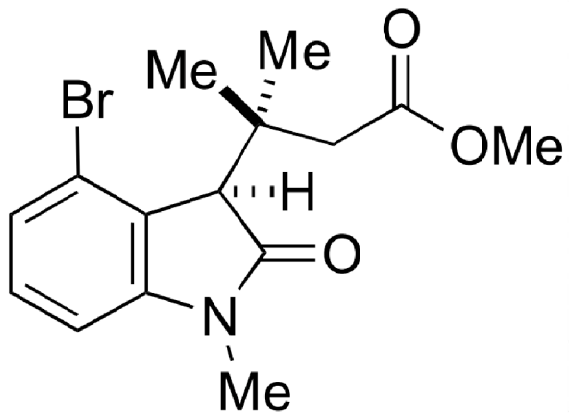




7

```
rwhj i 53 c1

exp4  s2pul

      SAMPLE
date    Apr 18 2002
solvent       CDCl3
file            exp
   ACQUISITION
sfrq        125.574
tn              C13
at              1.073
np              64000
sw           29817.4
fb              16000
bs                 64
tpwr               60
pw                4.0
d1              2.000
tof            1596.2
nt               5000
ct                820
alock               n
gain               50
      FLAGS
il                  n
in                  n
dp                  y
hs                 nn
     DISPLAY
sp             -628.1
wp           25739.8
vs               133
sc                 0
wc               240
hzmm          107.25
is         90000.00
rfl         11086.6
rfp          9668.2
th                 2
ins          100.000
nm  cdc  ph

      DEC. & VT
dfrq        499.349
dn               H1
dpwr             36
dof          -500.0
dm               yyy
dmm                w
dmf            14000
dseq
dres             1.0
homo               n
temp            27.0
     PROCESSING
lb              0.75
wtfile
proc               ft
fn            131072
math               f

werr
wexp
wbs
wnt
```

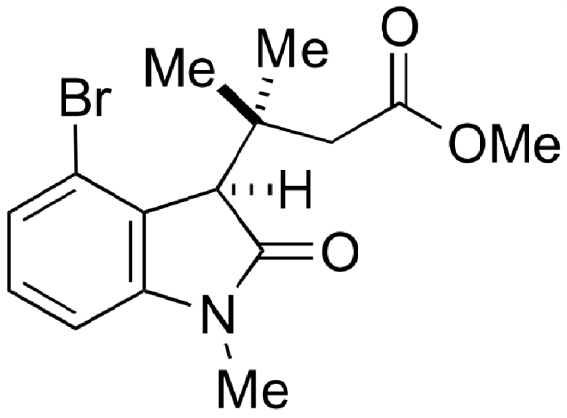




9

ss

```
rwhj I 270 cc 1

exp1  s2pul

      SAMPLE              DEC. & VT
date   Apr 30 2003   dfrq        499.349
solvent      CDCl3   dn               H1
file           exp   dpwr             30
   ACQUISITION       dof               0
sfrq       499.349   dm              nnn
tn              H1   dmm               c
at           4.865   dmf             200
np           64000   dseq
sw          6577.3   dres            1.0
fb            4000   homo              n
bs              32   temp           27.0
tpwr            54       PROCESSING
pw             2.0   lb             0.10
d1           2.000   wtfile
tof          297.6   proc              ft
nt              64   fn         not used
ct              64   math              f
alock            n
gain            40   werr
      FLAGS          wexp
il               n   wbs
in               n   wnt             wft
dp               y
hs              nn
     DISPLAY
sp          -249.8
wp          5243.1
vs              81
sc               0
wc             250
hzmm         20.97
is         1430.08
rfl          504.6
rfp              0
th               7
ins          1.000
ai  cdc  ph
```



9

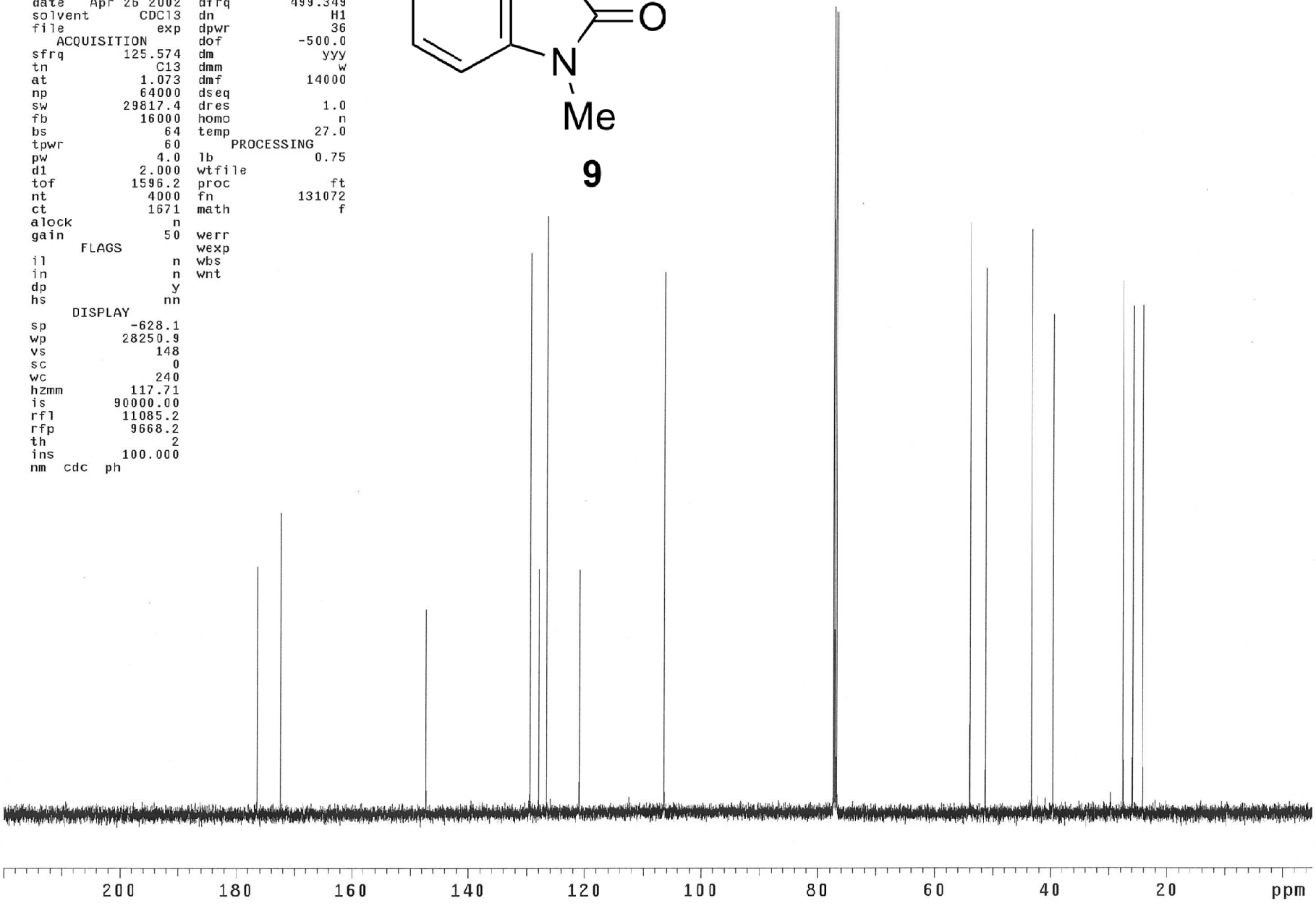
ss

rwhj i 57 c 1

exp4 s2pul

SAMPLE		DEC. & VT	
date	Apr 26 2002	dfrq	499.349
solvent	CDCl3	dn	H1
file	exp	dpwr	36
ACQUISITION		dof	-500.0
sfrq	125.574	dm	yyy
tn	C13	dmm	w
at	1.073	dmf	14000
np	64000	dseq	
sw	29817.4	dres	1.0
fb	16000	homo	n
bs	64	temp	27.0
tpwr	60	PROCESSING	
pw	4.0	lb	0.75
d1	2.000	wtfile	
tof	1596.2	proc	ft
nt	4000	fn	131072
ct	1671	math	f
alock	n		
gain	50	werr	
FLAGS		wexp	
il	n	wbs	
in	n	wnt	
dp	y		
hs	nn		
DISPLAY			
sp	-628.1		
wp	28250.9		
vs	148		
sc	0		
wc	240		
hzmm	117.71		
is	90000.00		
rfl	11085.2		
rfp	9668.2		
th	2		
ins	100.000		

nm cdc ph

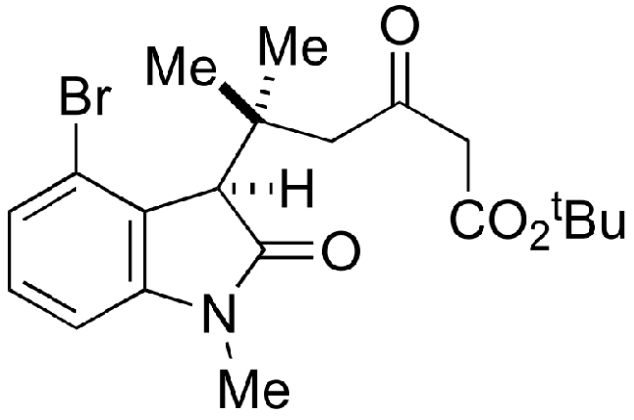


rwhjI142c1_h1

exp1 s2pul

SAMPLE		DEC. & VT	
date	Sep 11 2002	dfrq	499.349
solvent	CDCl3	dn	H1
file	exp	dpwr	30
ACQUISITION		dof	0
sfrq	499.350	dm	nnn
tn	H1	dmm	c
at	4.865	dmf	200
np	77742	dseq	
sw	7989.6	dres	1.0
fb	4000	homo	n
bs	32	temp	27.0
tpwr	54	PROCESSING	
pw	2.0	lb	0.10
d1	2.000	wtfile	
tof	499.2	proc	ft
nt	64	fn	not used
ct	64	math	f
alock	n		
gain	40	werr	
FLAGS		wexp	
il	n	wbs	
in	n	wnt	wft
dp	y		
hs	nn		
DISPLAY			
sp	-757.1		
wp	7739.8		
vs	37		
sc	0		
wc	250		
hzmm	30.96		
is	1145.86		
rfl	1006.9		
rfp	0		
th	2		
ins	1.000		

ai cdc ph

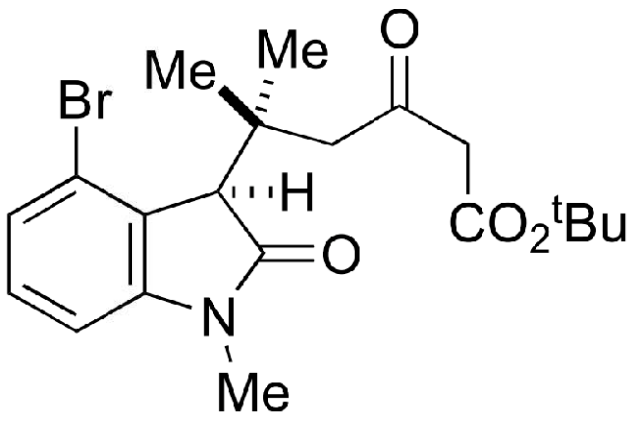


10

rwhj I 142 c 1

exp4 s2pul

SAMPLE		DEC. & VT	
date	Sep 11 2002	dfrq	499.349
solvent	CDCl3	dn	H1
file	exp	dpwr	36
ACQUISITION		dof	-500.0
sfrq	125.575	dm	yyy
tn	C13	dmm	w
at	1.073	dmf	14000
np	70058	dseq	
sw	32639.7	dres	1.0
fb	18000	homo	n
bs	8	temp	27.0
tpwr	60	PROCESSING	
pw	4.0	lb	0.75
d1	2.000	wtfile	
tof	2509.7	proc	ft
nt	6000	fn	131072
ct	6000	math	f
alock	n		
gain	50	werr	
FLAGS		wexp	
il	n	wbs	
in	n	wnt	
dp	y		
hs	nn		
DISPLAY			
sp	-628.0		
wp	28250.9		
vs	327		
sc	0		
wc	250		
hzmm	113.00		
is	90000.00		
rfl	11580.2		
rfp	9668.2		
th	2		
ins	100.000		
nm cdc ph			



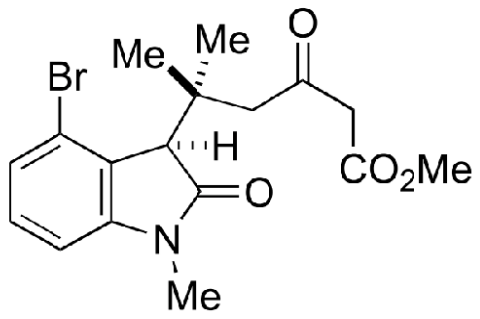
10

ss

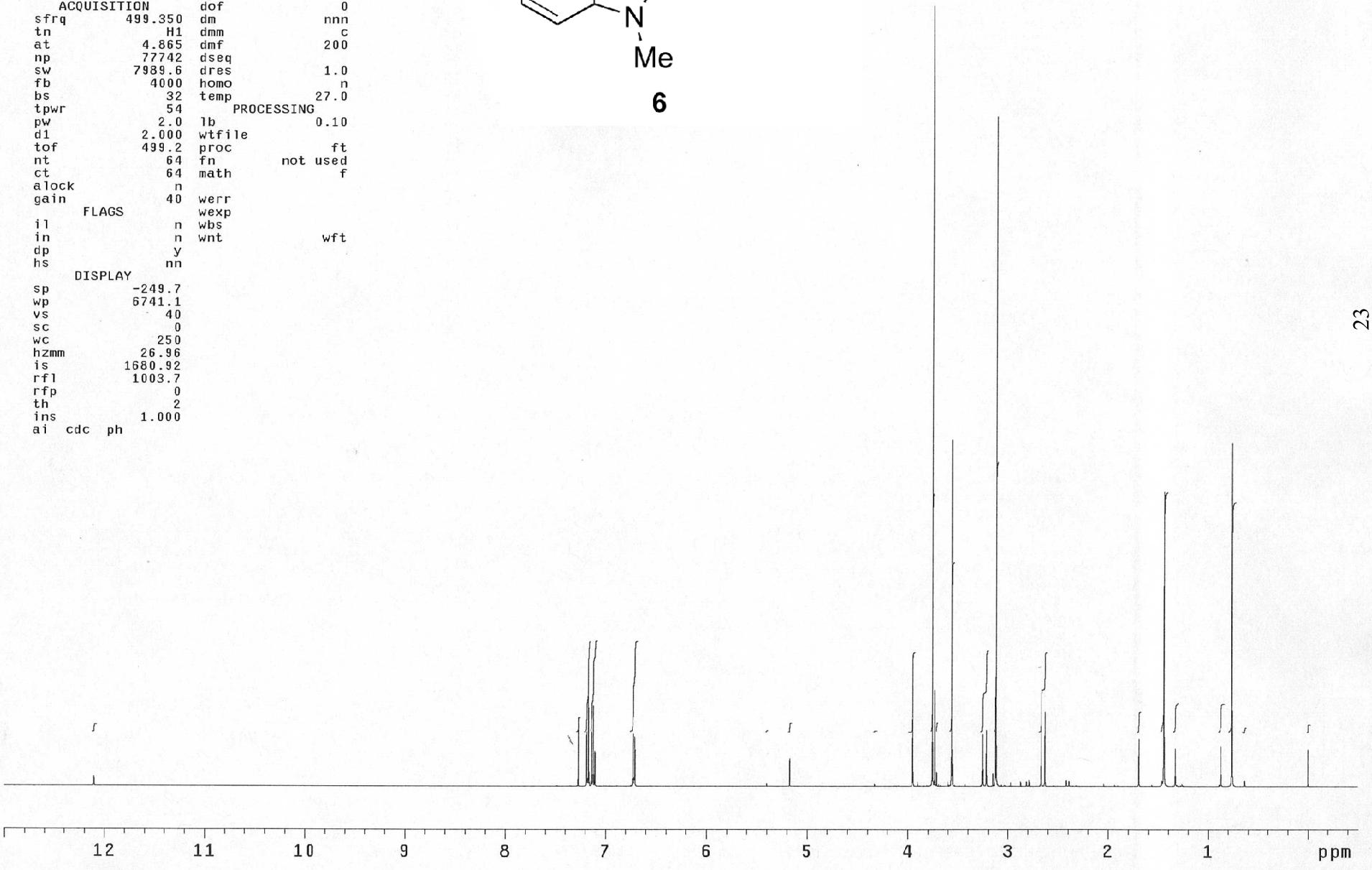
rwhj-I-107-c-1

exp1 s2pul

SAMPLE		DEC. & VT	
date	Aug 28 2002	dfrq	499.349
solvent	CDCl3	dn	H1
file	exp	dpwr	30
ACQUISITION		dof	0
sfrq	499.350	dm	nnn
tn	H1	dmm	c
at	4.865	dmf	200
np	77742	dseq	
sw	7989.6	dres	1.0
fb	4000	homo	n
bs	32	temp	27.0
tpwr	54	PROCESSING	
pw	2.0	lb	0.10
d1	2.000	wtfile	
tof	499.2	proc	ft
nt	64	fn	not used
ct	64	math	f
alock	n		
gain	40	werr	
FLAGS		wexp	
il	n	wbs	
in	n	wnt	wft
dp	y		
hs	nn		
DISPLAY			
sp	-249.7		
wp	6741.1		
vs	40		
sc	0		
wc	250		
hzmm	26.96		
is	1680.92		
rfl	1003.7		
rfp	0		
th	2		
ins	1.000		
ai cdc ph			



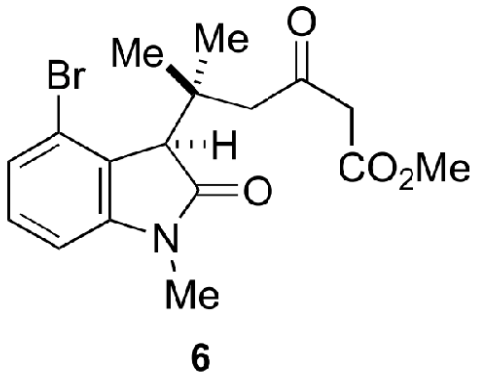
6



rwhj-I-107-c-1

exp4 s2pul

SAMPLE		DEC. & VT	
date	Aug 29 2002	dfrq	499.349
solvent	CDCl3	dn	H1
file	exp	dpwr	36
ACQUISITION		dof	-500.0
sfrq	125.575	dm	yyy
tn	C13	dmm	w
at	1.073	dmf	14000
np	70058	dseq	
sw	32639.7	dres	1.0
fb	18000	homo	n
bs	8	temp	27.0
tpwr	60	PROCESSING	
pw	4.0	lb	0.75
d1	2.000	wtfile	
tof	2509.7	proc	ft
nt	4000	fn	131072
ct	831	math	f
alock	n		
gain	50	werr	
FLAGS		wexp	
il	n	wbs	
in	n	wnt	
dp	y		
hs	nn		
DISPLAY			
sp	-628.0		
wp	28250.9		
vs	143		
sc	0		
wc	250		
hzmm	113.00		
is	90000.00		
rfl	11583.2		
rfp	9668.2		
th	2		
ins	100.000		
nm cdc ph			



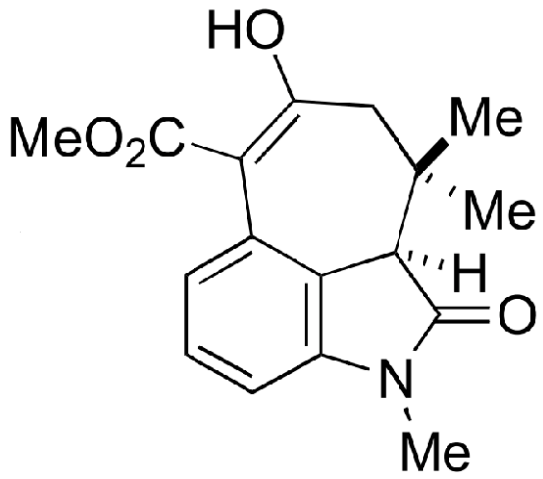
6

rwhj-221-c-1

exp1 s2pul

SAMPLE		DEC. & VT	
date	Feb 3 2003	dfrq	499.349
solvent	CDCl3	dn	H1
file	exp	dpwr	30
ACQUISITION		dof	0
sfrq	499.350	dm	nnn
tn	H1	dmm	c
at	4.865	dmf	200
np	89888	dseq	
sw	9237.9	dres	1.0
fb	5000	homo	n
bs	32	temp	27.0
tpwr	54	PROCESSING	
pw	2.0	lb	0.10
d1	2.000	wtfile	
tof	1123.4	proc	ft
nt	8	fn	not used
ct	8	math	f
alock	n		
gain	40	werr	
FLAGS		wexp	
il	n	wbs	
in	n	wnt	wft
dp	y		
hs	nn		
DISPLAY			
sp	-249.7		
wp	8239.2		
vs	45		
sc	0		
wc	250		
hzmm	32.96		
is	584.67		
rfl	4634.4		
rfp	3615.3		
th	2		
ins	3.000		

ai cdc ph



11

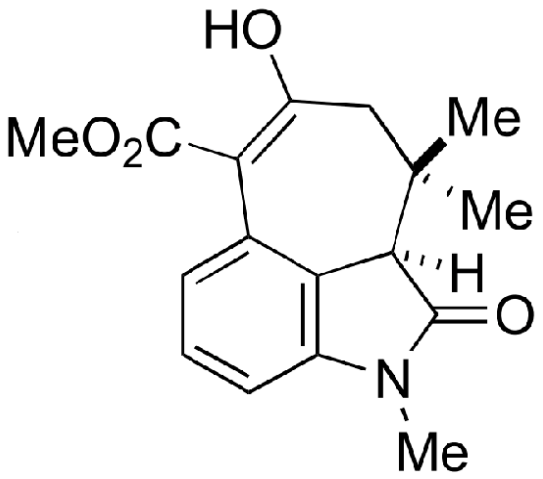
ss

rwhj-221-c-1

exp4 s2pul

SAMPLE		DEC. & VT	
date	Feb 3 2003	dfrq	499.349
solvent	CDCl3	dn	H1
file	exp	dpwr	36
ACQUISITION		dof	-500.0
sfrq	125.574	dm	yyy
tn	C13	dmm	w
at	1.073	dmf	14000
np	64000	dseq	
sw	29817.4	dres	1.0
fb	16000	homo	n
bs	64	temp	27.0
tpwr	60	PROCESSING	
pw	4.0	lb	0.75
d1	2.000	wtfile	
tof	1596.2	proc	ft
nt	2000	fn	131072
ct	642	math	f
alock	n		
gain	50	werr	
FLAGS		wexp	
il	n	wbs	
in	n	wnt	
dp	y		
hs	nn		
DISPLAY			
sp	-628.1		
wp	28250.9		
vs	218		
sc	0		
wc	250		
hzmm	113.00		
is	90000.00		
rfl	11083.9		
rfp	9668.2		
th	2		
ins	100.000		

nm cdc ph



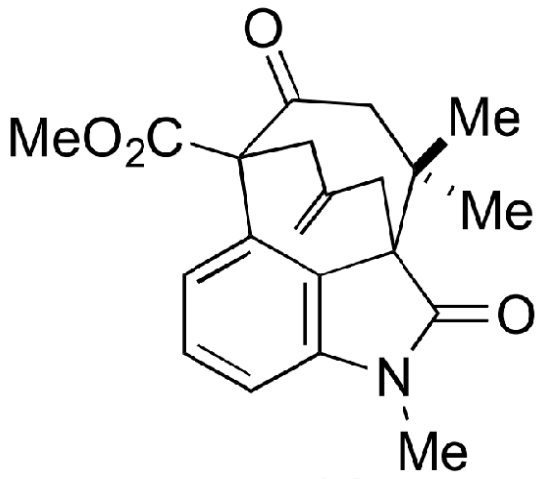
11

ss

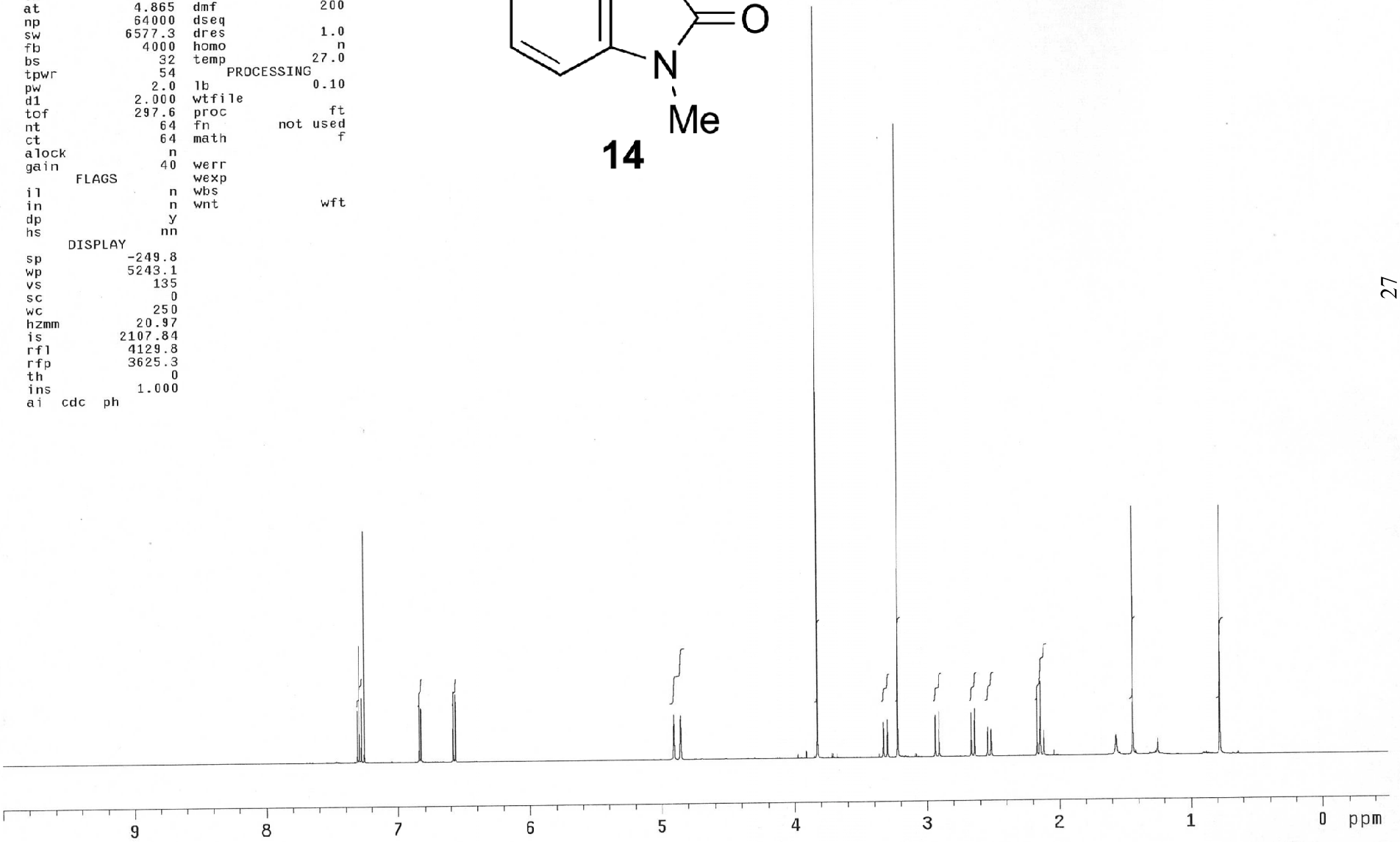
rwhj I 251 c 1

exp1 s2pul

SAMPLE		DEC. & VT	
date	Mar 26 2003	dfrq	499.349
solvent	CDCl3	dn	H1
file	exp	dpwr	30
ACQUISITION		dof	0
sfrq	499.349	dm	nnn
tn	H1	dmm	c
at	4.865	dmf	200
np	64000	dseq	
sw	6577.3	dres	1.0
fb	4000	homo	n
bs	32	temp	27.0
tpwr	54	PROCESSING	
pw	2.0	lb	0.10
d1	2.000	wtfile	
tof	297.6	proc	ft
nt	64	fn	not used
ct	64	math	f
alock	n		
gain	40	werr	
FLAGS		wexp	
il	n	wbs	
in	n	wnt	wft
dp	y		
hs	nn		
DISPLAY			
sp	-249.8		
wp	5243.1		
vs	135		
sc	0		
wc	250		
hzmm	20.97		
is	2107.84		
rfl	4129.8		
rfp	3625.3		
th	0		
ins	1.000		
ai cdc ph			



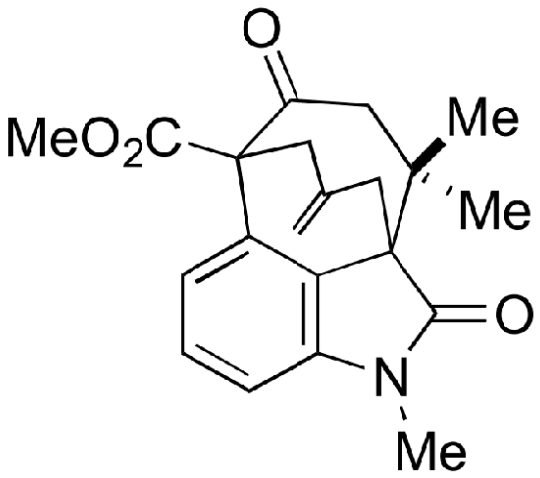
14



rwhj I 251 c 1

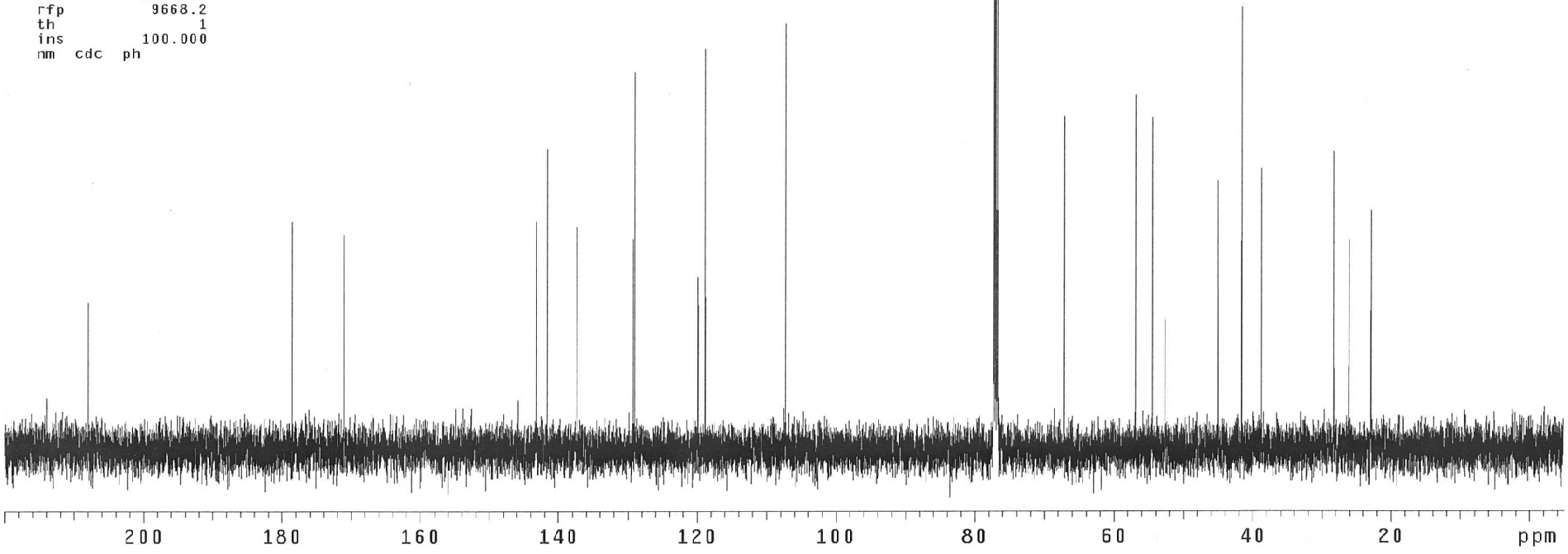
exp9 s2pul

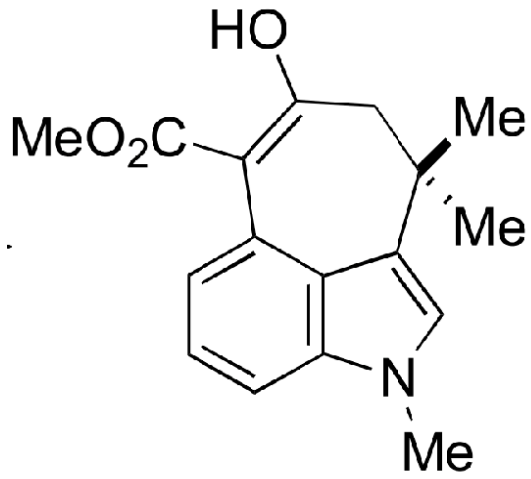
SAMPLE		DEC. & VT	
date	Mar 26 2003	dfrq	499.349
solvent	CDCl3	dn	H1
file	exp	dpwr	44
ACQUISITION		dof	-500.0
sfrq	125.574	dm	yyy
tn	C13	dmm	w
at	1.073	dmf	11000
np	64000	dseq	
sw	29817.4	dres	1.0
fb	16000	homo	n
bs	64	temp	27.0
tpwr	54	PROCESSING	
pw	3.0	lb	0.75
d1	2.000	wtfile	
tof	1596.2	proc	ft
nt	2000	fn	131072
ct	2000	math	f
alock	n		
gain	50	werr	
FLAGS		wexp	
il	n	wbs	
in	n	wnt	
dp	y		
hs	nn		
DISPLAY			
sp	-628.1		
wp	28250.9		
vs	652		
sc	0		
wc	250		
hzmm	113.00		
is	90000.00		
rfl	11081.6		
rfp	9668.2		
th	1		
ins	100.000		
nm cdc ph			



14

ss

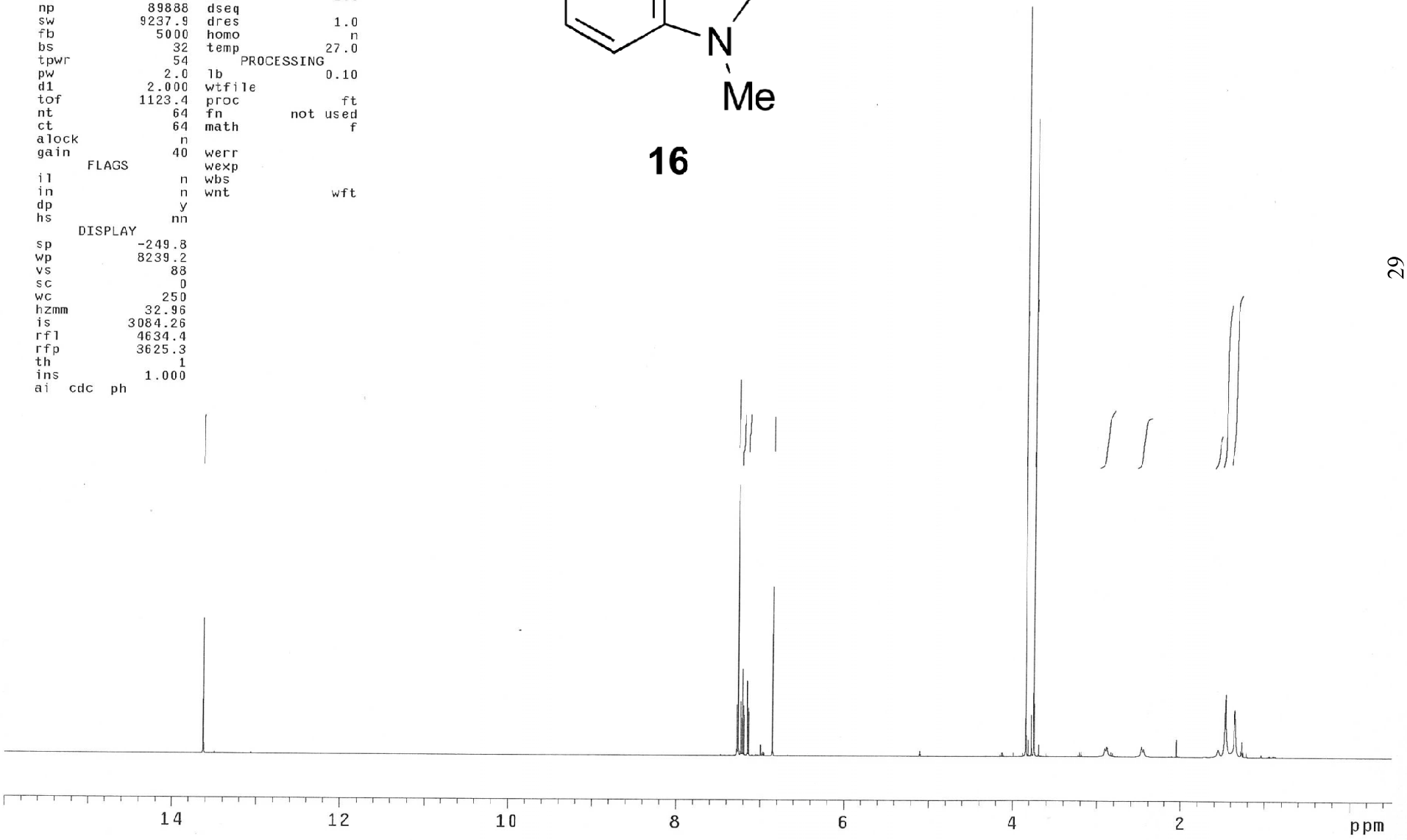




16

ss

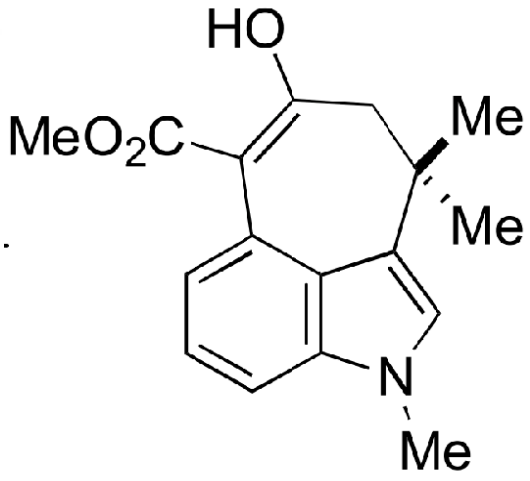
```
rwhj II 50 c 2

exp1  s2pul

      SAMPLE
date    Aug 26 2003
solvent       CDCl3
file             exp
   ACQUISITION
sfrq         499.350
tn                H1
at             4.865
np             89888
sw            9237.9
fb              5000
bs                32
tpwr              54
pw               2.0
d1             2.000
tof           1123.4
nt                64
ct                64
alock              n
gain              40
      FLAGS
il                 n
in                 n
dp                 y
hs                nn
     DISPLAY
sp            -249.8
wp            8239.2
vs                88
sc                 0
wc               250
hzmm           32.96
is           3084.26
rfl           4634.4
rfp           3625.3
th                 1
ins            1.000
ai  cdc  ph

      DEC. & VT
dfrq         499.349
dn                H1
dpwr              30
dof                0
dm               nnn
dmm                c
dmf              200
dseq
dres             1.0
homo               n
temp            27.0
     PROCESSING
lb               0.10
wtfile
proc               ft
fn         not used
math               f
werr
wexp
wbs
wnt              wft
```



rwhj II 50 c 2

exp4 s2pul

SAMPLE		DEC. & VT	
date	Aug 26 2003	dfrq	499.349
solvent	CDCl3	dn	H1
file	exp	dpwr	36
ACQUISITION		dof	-500.0
sfrq	125.575	dm	yyy
tn	C13	dmm	w
at	1.073	dmf	14000
np	70058	dseq	
sw	32639.7	dres	1.0
fb	18000	homo	n
bs	64	temp	27.0
tpwr	60	PROCESSING	
pw	4.0	lb	2.00
d1	2.000	wtfile	
tof	1881.9	proc	ft
nt	14000	fn	not used
ct	13613	math	f
alock	n		
gain	50	werr	
FLAGS		wexp	
il	n	wbs	
in	n	wnt	
dp	y		
hs	nn		
DISPLAY			
sp	-628.0		
wp	25739.7		
vs	781		
sc	0		
wc	250		
hzmm	102.96		
is	90000.00		
rfl	12208.7		
rfp	9668.2		
th	1		
ins	100.000		
nm cdc ph			



16

ss

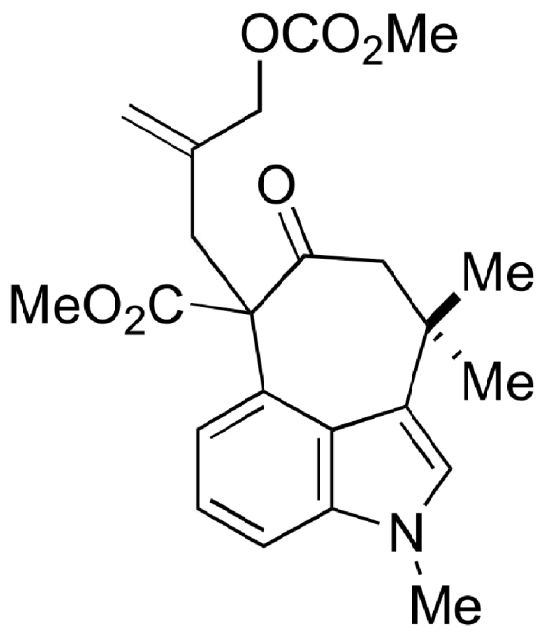
30

rwhj II 55 c 1

exp1 s2pul

SAMPLE		DEC. & VT	
date	Sep 2 2003	dfrq	499.349
solvent	CDCl3	dn	H1
file	exp	dpwr	30
ACQUISITION		dof	0
sfrq	499.350	dm	nnn
tn	H1	dmm	c
at	4.865	dmf	200
np	89888	dseq	
sw	9237.9	dres	1.0
fb	5000	homo	n
bs	32	temp	27.0
tpwr	54	PROCESSING	
pw	2.0	lb	0.10
d1	2.000	wtfile	
tof	1123.4	proc	ft
nt	64	fn	not used
ct	64	math	f
alock	n		
gain	40	werr	
FLAGS		wexp	
il	n	wbs	
in	n	wnt	wft
dp	y		
hs	nn		
DISPLAY			
sp	-249.8		
wp	5243.0		
vs	305		
sc	0		
wc	250		
hzmm	20.97		
is	6546.83		
rfl	1009.3		
rfp	0		
th	1		
ins	1.000		

ai cdc ph



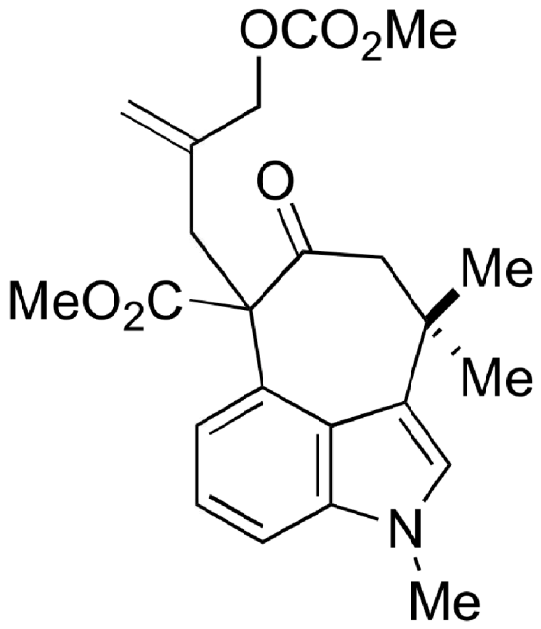
17

rwhj II 55 c 1

exp4 s2pul

SAMPLE		DEC. & VT	
date	Sep 2 2003	dfrq	499.349
solvent	CDCl3	dn	H1
file	exp	dpwr	36
ACQUISITION		dof	-500.0
sfrq	125.575	dm	yyy
tn	C13	dmm	w
at	1.073	dmf	14000
np	70058	dseq	
sw	32639.7	dres	1.0
fb	18000	homo	n
bs	64	temp	27.0
tpwr	60	PROCESSING	
pw	4.0	lb	0.75
d1	2.000	wtfile	
tof	1881.9	proc	ft
nt	10000	fn	not used
ct	10000	math	f
alock	n		
gain	50	werr	
FLAGS		wexp	
il	n	wbs	
in	n	wnt	
dp	y		
hs	nn		
DISPLAY			
sp	-628.0		
wp	28250.9		
vs	1484		
sc	0		
wc	250		
hzmm	113.00		
is	90000.00		
rfl	12207.2		
rfp	9668.2		
th	0		
ins	100.000		

nm cdc ph



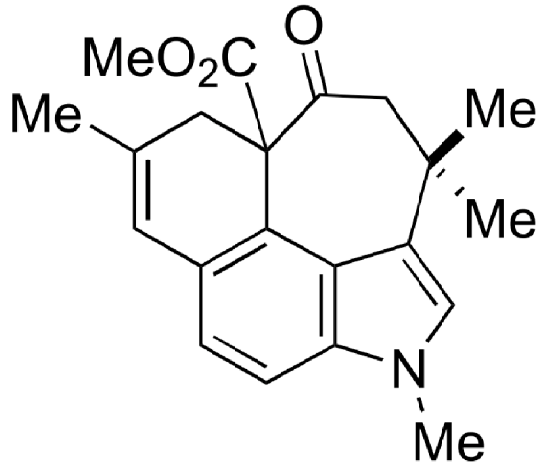
17

ss

RWHJ-II-71-C-1

exp1 s2pul

SAMPLE		DEC. & VT	
date	Sep 26 2003	dfrq	499.349
solvent	CDCl3	dn	H1
file	exp	dpwr	30
ACQUISITION		dof	0
sfrq	499.349	dm	nnn
tn	H1	dmm	c
at	4.865	dmf	200
np	64000	dseq	
sw	6577.3	dres	1.0
fb	4000	homo	n
bs	32	temp	27.0
tpwr	54	PROCESSING	
pw	2.0	lb	0.10
d1	2.000	wtfile	
tof	297.6	proc	ft
nt	64	fn	not used
ct	64	math	f
alock	n		
gain	40	werr	
FLAGS		wexp	
il	n	wbs	
in	n	wnt	wft
dp	y		
hs	nn		
DISPLAY			
sp	-249.8		
wp	5243.1		
vs	1109		
sc	0		
wc	250		
hzmm	20.97		
is	42938.30		
rfl	4129.8		
rfp	3625.3		
th	1		
ins	1.000		
ai cdc ph			



18

jw

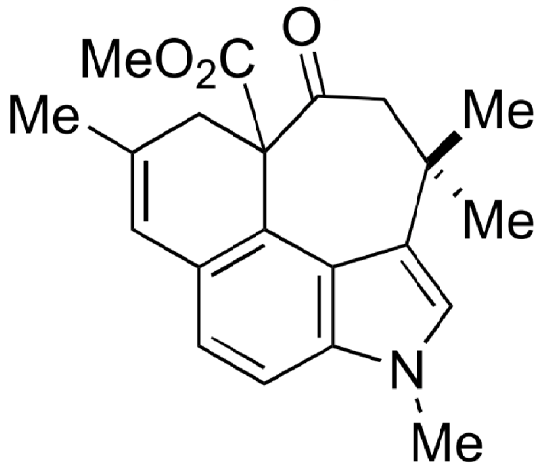
33

9 8 7 6 5 4 3 2 1 0 ppm

rwhj II 71 c1

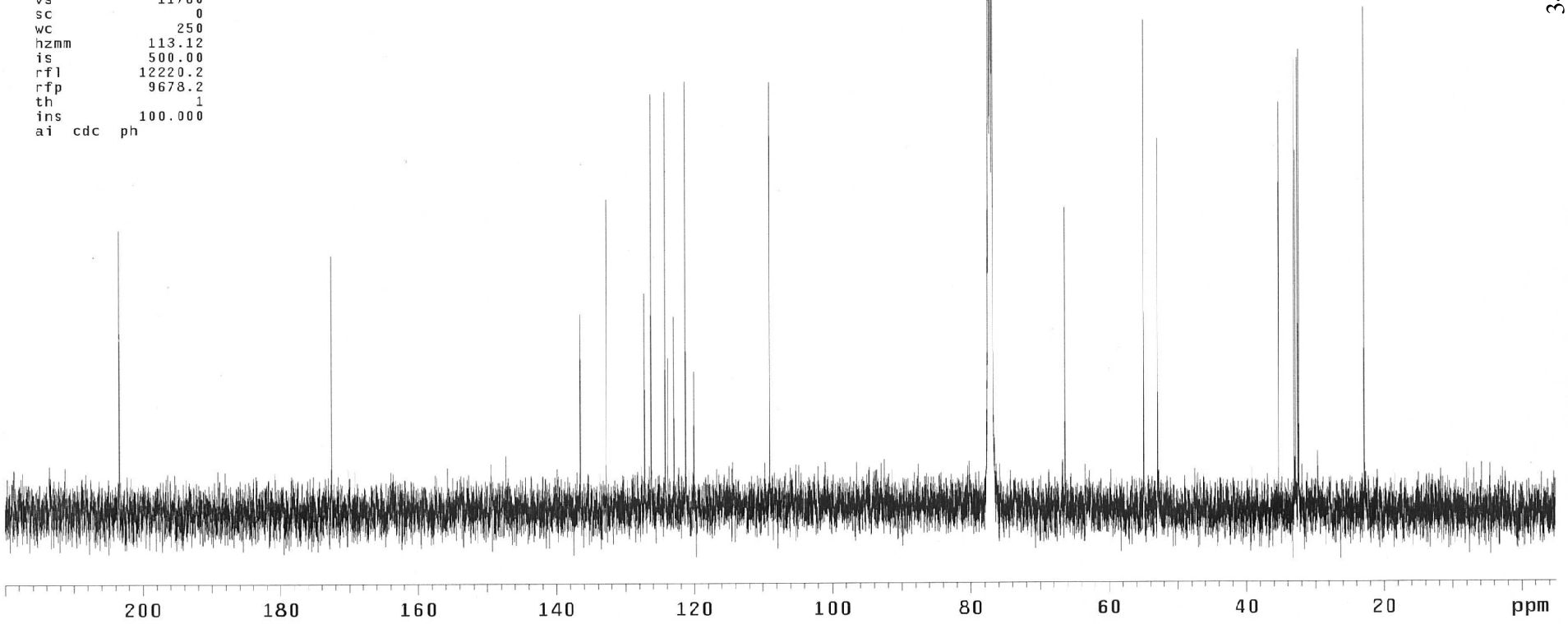
exp4 s2pul

SAMPLE		DEC. & VT	
date	Sep 29 2003	dfrq	499.867
solvent	CDCl3	dn	H1
file	exp	dpwr	37
ACQUISITION		dof	0
sfrq	125.705	dm	yyy
tn	C13	dmm	w
at	1.280	dmf	10582
np	85262	dseq	
sw	33305.6	dres	1.0
fb	18000	homo	n
bs	64	temp	27.0
tpwr	53	PROCESSING	
pw	3.0	lb	1.00
d1	2.000	wtfile	
tof	2198.1	proc	ft
nt	15000	fn	not used
ct	15000	math	f
alock	n		
gain	60	werr	
FLAGS		wexp	
il	n	wbs	
in	n	wnt	
dp	y		
hs	nn		
DISPLAY			
sp	-628.7		
wp	28280.5		
vs	11760		
sc	0		
wc	250		
hzmm	113.12		
is	500.00		
rfl	12220.2		
rfp	9678.2		
th	1		
ins	100.000		
ai cdc ph			



18

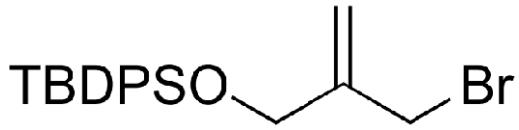
ss



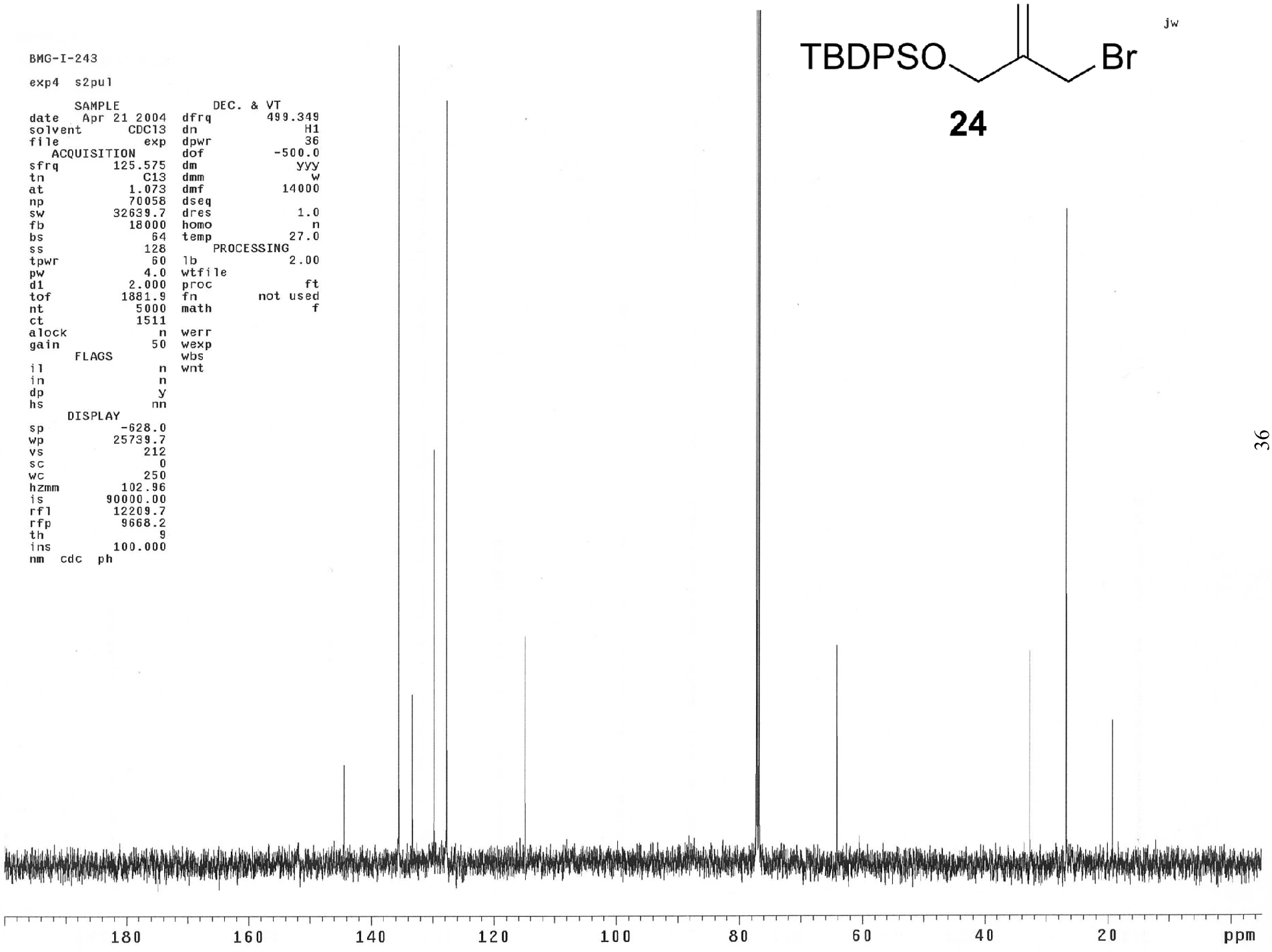
BMG-I-243

exp1 s2pul

SAMPLE		DEC. & VT	
date	Apr 21 2004	dfrq	499.349
solvent	CDCl3	dn	H1
file	exp	dpwr	30
ACQUISITION		dof	0
sfrq	499.349	dm	nnn
tn	H1	dmm	c
at	4.865	dmf	200
np	64000	dseq	
sw	6577.3	dres	1.0
fb	4000	homo	n
bs	32	temp	27.0
ss	4	PROCESSING	
tpwr	54	lb	0.10
pw	2.0	wtfile	
d1	2.000	proc	ft
tof	297.6	fn	not used
nt	32	math	f
ct	32		
alock	n	werr	
gain	40	wexp	
FLAGS		wbs	
il	n	wnt	wft
in	n		
dp	y		
hs	nn		
DISPLAY			
sp	-249.8		
wp	5243.1		
vs	43		
sc	0		
wc	250		
hzmm	20.97		
is	1109.25		
rfl	4130.0		
rfp	3615.3		
th	7		
ins	1.000		
ai cdc ph			



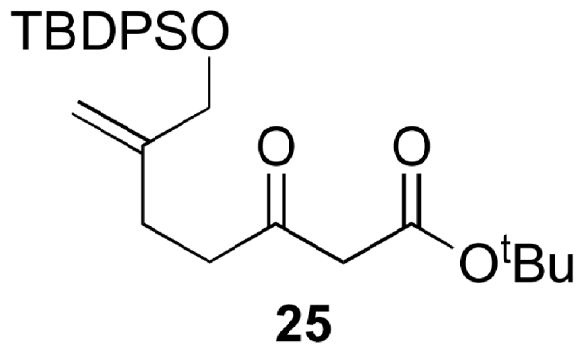
24


BMG-I-243
exp4 s2pul
SAMPLE
date Apr 21 2004
solvent CDCl3
file exp
ACQUISITION
sfrq 125.575
tn C13
at 1.073
np 70058
sw 32639.7
fb 18000
bs 64
ss 128
tpwr 60
pw 4.0
d1 2.000
tof 1881.9
nt 5000
ct 1511
alock n
gain 50
FLAGS
il n
in n
dp y
hs nn
DISPLAY
sp -628.0
wp 25739.7
vs 212
sc 0
wc 250
hzmm 102.96
is 90000.00
rfl 12209.7
rfp 9668.2
th 9
ins 100.000
nm cdc ph
DEC. & VT
dfrq 499.349
dn H1
dpwr 36
dof -500.0
dm yyy
dmm w
dmf 14000
dseq
dres 1.0
homo n
temp 27.0
PROCESSING
lb 2.00
wtfile
proc ft
fn not used
math f
werr
wexp
wbs
wnt
TBDPSO
Br
jw
24
180
160
140
120
100
80
60
40
20
ppm

RWHJ 2 182-c-1 cdcl3

exp1 std1h

SAMPLE		DEC. & VT	
date	Mar 24 2004	dfrq	400.269
solvent	CDCl3	dn	H1
file	exp	dpwr	30
ACQUISITION		dof	0
sfrq	400.269	dm	nnn
tn	H1	dmm	c
at	2.859	dmf	200
np	25130	PROCESSING	
sw	4395.6	lb	0.10
fb	not used	wtfile	
bs	4	proc	ft
tpwr	58	fn	not used
pw	2.0		
d1	2.000	werr	
tof	0.0	wexp	
nt	16	wbs	
ct	16	wnt	
alock	n		
gain	not used		
FLAGS			
il	n		
in	n		
dp	y		
DISPLAY			
sp	-180.1		
wp	4395.6		
vs	150		
sc	0		
wc	250		
hzmm	17.58		
is	956.13		
rfl	180.1		
rfp	0		
th	20		
ins	2.000		
nm cdc ph			



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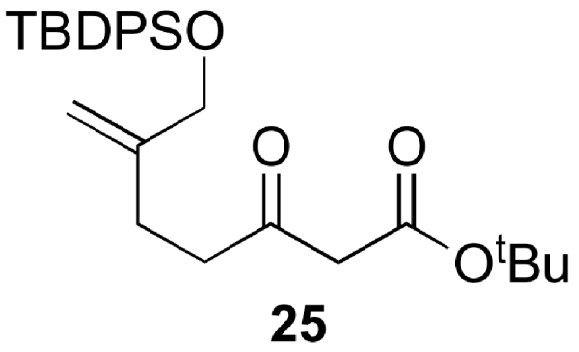
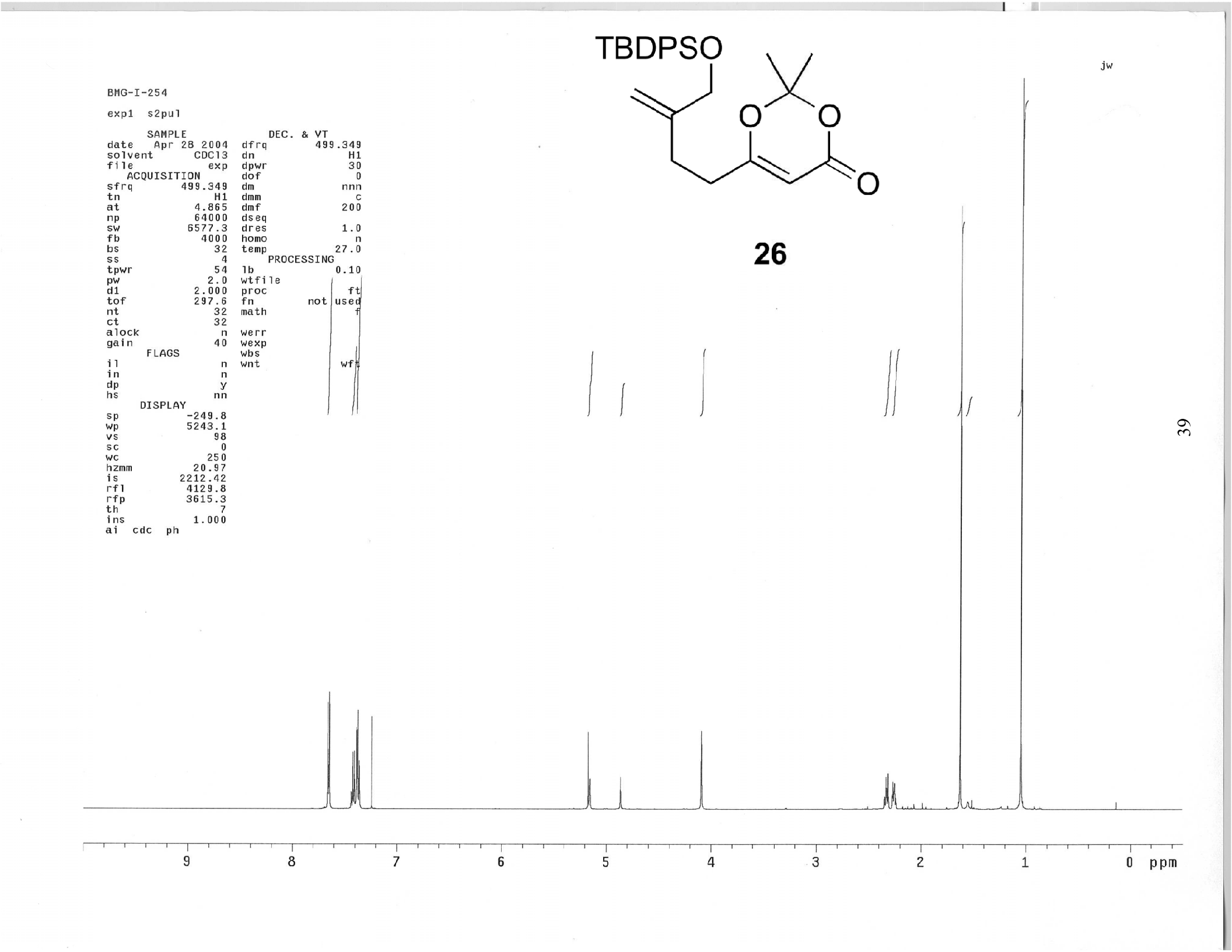
RWHJ 2 171-c-1 cdcl3

exp2 std13c

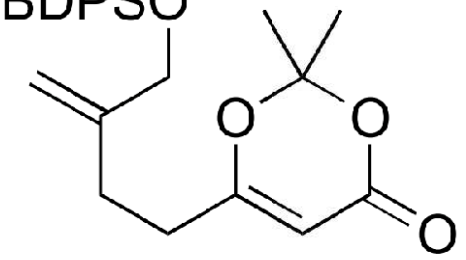
SAMPLE		DEC. & VT	
date	Mar 24 2004	dfrq	400.269
solvent	CDCl3	dn	H1
file	exp	dpwr	38
ACQUISITION		dof	0
sfrq	100.658	dm	yyy
tn	C13	dmm	w
at	1.280	dmf	9300
np	64484	PROCESSING	
sw	25188.9	lb	1.00
fb	13800	wtfile	
bs	16	proc	ft
tpwr	60	fn	not used
pw	3.0		
d1	4.000	werr	
tof	1539.5	wexp	
nt	100000	wbs	
ct	197	wnt	
alock	n		
gain	not used		
FLAGS			
il	n		
in	n		
dp	y		
DISPLAY			
sp	-1553.3		
wp	25188.9		
vs	150		
sc	0		
wc	250		
hzmm	100.76		
is	500.00		
rfl	1553.3		
rfp	0		
th	4		
ins	100.000		
nm cdc ph			



25


TBDPSO
jw
BMG-I-254
exp1 s2pul
SAMPLE
date Apr 28 2004
solvent CDCl3
file exp
ACQUISITION
sfrq 499.349
tn H1
at 4.865
np 64000
sw 6577.3
fb 4000
bs 32
ss 4
tpwr 54
pw 2.0
d1 2.000
tof 297.6
nt 32
ct 32
alock n
gain 40
FLAGS
il n
in n
dp y
hs nn
DISPLAY
sp -249.8
wp 5243.1
vs 98
sc 0
wc 250
hzmm 20.97
is 2212.42
rfl 4129.8
rfp 3615.3
th 7
ins 1.000
ai cdc ph
DEC. & VT
dfrq 499.349
dn H1
dpwr 30
dof 0
dm nnn
dmm c
dmf 200
dseq
dres 1.0
homo n
temp 27.0
PROCESSING
lb 0.10
wtfile
proc ft
fn not used
math f
werr
wexp
wbs
wnt wft
O
O
O
26
9
8
7
6
5
4
3
2
1
0 ppm

TBDPSO



jw

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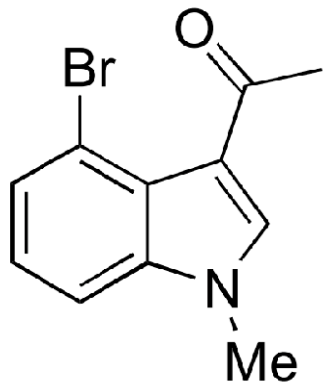
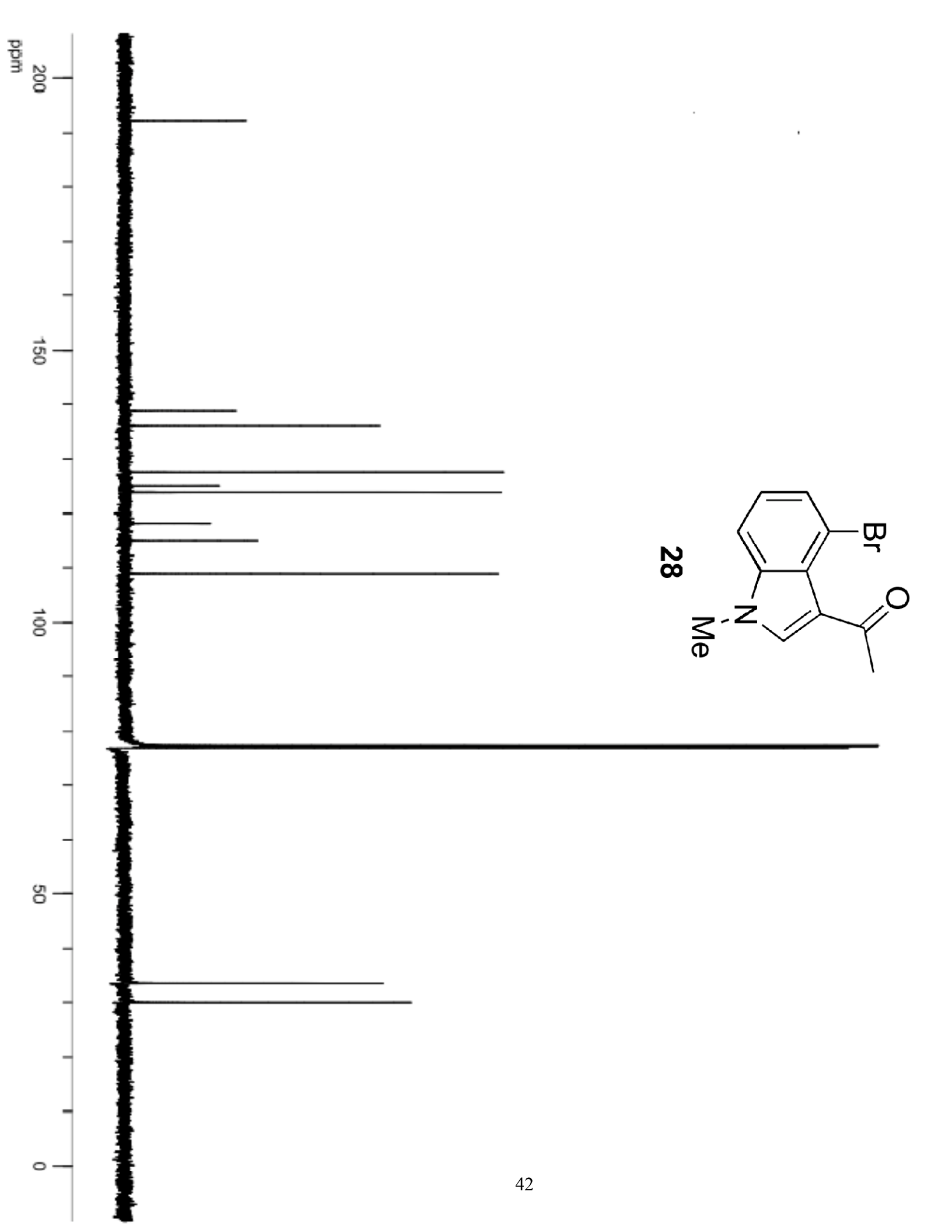
BMG-I-254

exp4 s2pul

SAMPLE		DEC. & VT	
date	Apr 28 2004	dfrq	499.349
solvent	CDCl3	dn	H1
file	exp	dpwr	36
ACQUISITION		dof	-500.0
sfrq	125.575	dm	yyy
tn	C13	dmm	w
at	1.073	dmf	14000
np	70058	dseq	
sw	32639.7	dres	1.0
fb	18000	homo	n
bs	64	temp	27.0
ss	64	PROCESSING	
tpwr	60	lb	2.00
pw	4.0	wtfile	
d1	2.000	proc	ft
tof	1881.9	fn	not used
nt	5000	math	f
ct	3044		
alock	n	werr	
gain	50	wexp	
FLAGS		wbs	
il	n	wnt	
in	n		
dp	y		
hs	nn		
DISPLAY			
sp	-628.0		
wp	25739.7		
vs	324		
sc	0		
wc	250		
hzmm	102.96		
is	90000.00		
rfl	12210.2		
rfp	9668.2		
th	11		
ins	100.000		
nm cdc ph			



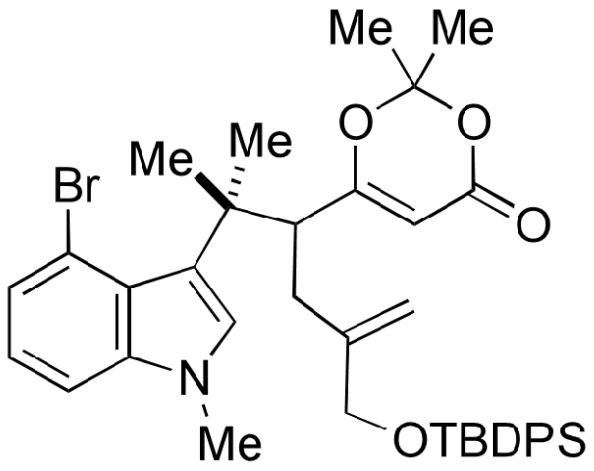
28


ppm
200
150
100
50
0
Br
O
N
Me
28

BMGVI-92

Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
Mercury-400BB "nmr6"

Relax. delay 2.000 sec
Pulse 16.4 degrees
Acq. time 2.856 sec
Width 5602.2 Hz
8 repetitions
OBSERVE H1, 400.2669784 MH
DATA PROCESSING
Line broadening 0.1 Hz
FT size 32768
Total time 0 min, 0 sec



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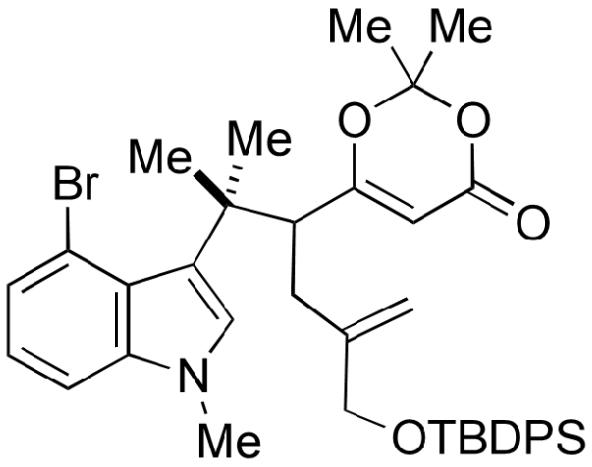
BMG V 170

exp4 s2pul

SAMPLE		DEC. & VT	
date	Jul 11 2005	dfrq	49
solvent	CDCl3	dn	
file	exp	dpwr	
ACQUISITION		dof	
sfrq	125.705	dm	
tn	C13	dmm	
at	1.279	dmf	1
np	85262	dseq	
sw	33333.3	dres	
fb	not used	homo	
bs	64	temp	
tpwr	53	PROCESSINC	
pw	3.0	lb	
d1	2.000	wtfile	
tof	2198.1	proc	
nt	6000	fn	not
ct	6000	math	
alock	n		
gain	60	werr	
FLAGS		wexp	
il	n	wbs	
in	n	wnt	
dp	y		
hs	nn		
DISPLAY			
sp	-628.6		
wp	25766.5		
vs	2711		
sc	0		
wc	250		
hzmm	103.07		
is	500.00		
rfl	12236.5		
rfp	9678.2		
th	6		
ins	100.000		
ai	ph		



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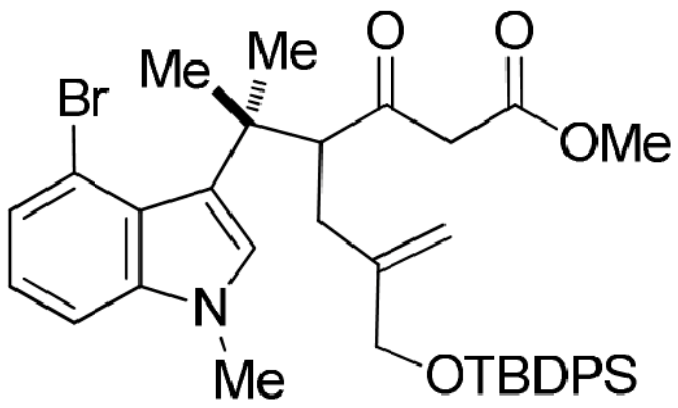
ss

BMGVI-108

Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
Mercury-400BB "nmr6"

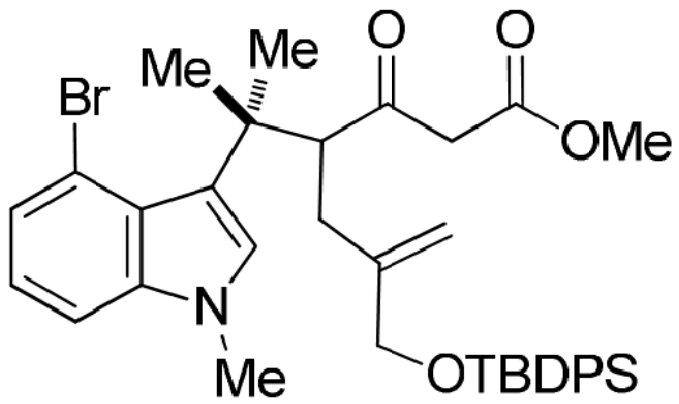
Relax. delay 2.000 sec
Pulse 16.4 degrees
Acq. time 2.856 sec
Width 5602.2 Hz
32 repetitions
OBSERVE H1, 400.2669784 MHz
DATA PROCESSING
Line broadening 0.1 Hz
FT size 32768
Total time 2 min, 40 sec

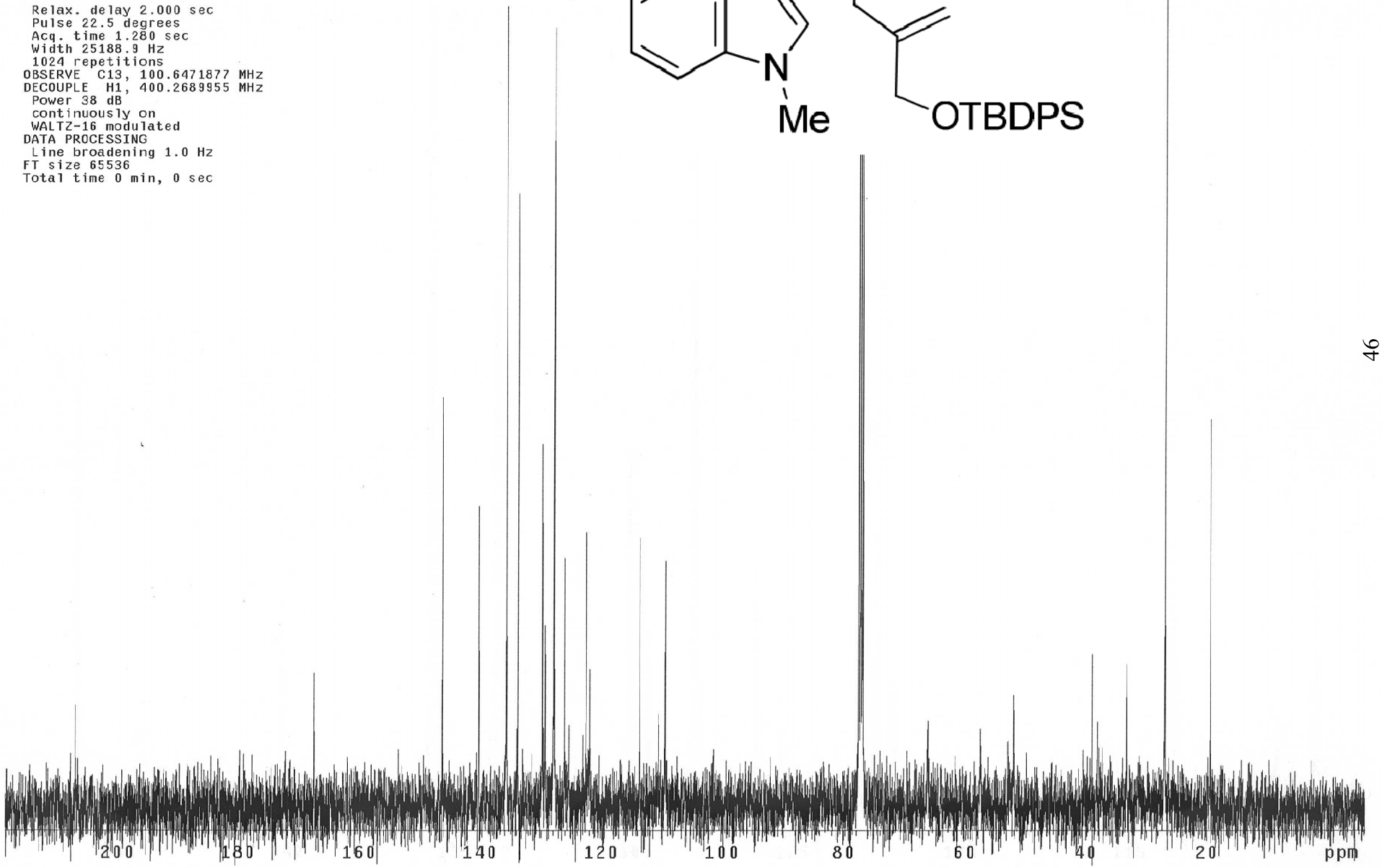


BMGVI-108

Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
Mercury-400BB "nmr6"

Relax. delay 2.000 sec
Pulse 22.5 degrees
Acq. time 1.280 sec
Width 25188.9 Hz
1024 repetitions
OBSERVE C13, 100.6471877 MHz
DECOUPLE H1, 400.2689955 MHz
Power 38 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
FT size 65536
Total time 0 min, 0 sec



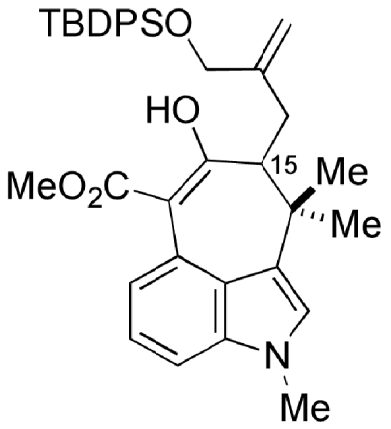


BMGV-198 2nd Column

Pulse Sequence: s2pul

Solvent: CDCl3
Ambient temperature
Mercury-400BB "nmr6"

Relax. delay 2.000 sec
Pulse 16.4 degrees
Acq. time 2.856 sec
Width 6802.7 Hz
8 repetitions
OBSERVE H1, 400.2669784 MHz
DATA PROCESSING
Line broadening 0.1 Hz
FT size 65536
Total time 0 min, 41 sec



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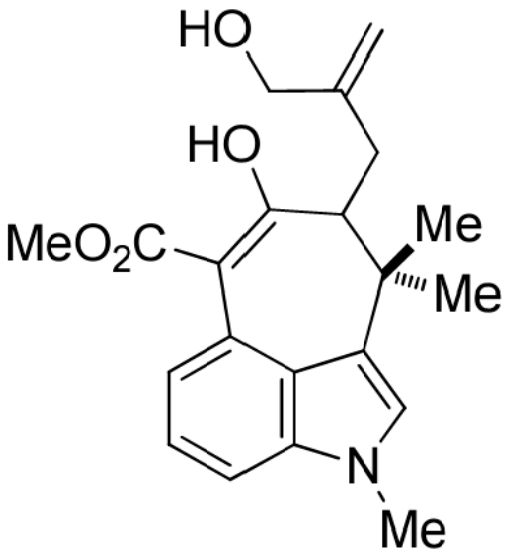
BMGVI-99

Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
Mercury-400BB "nmr6"

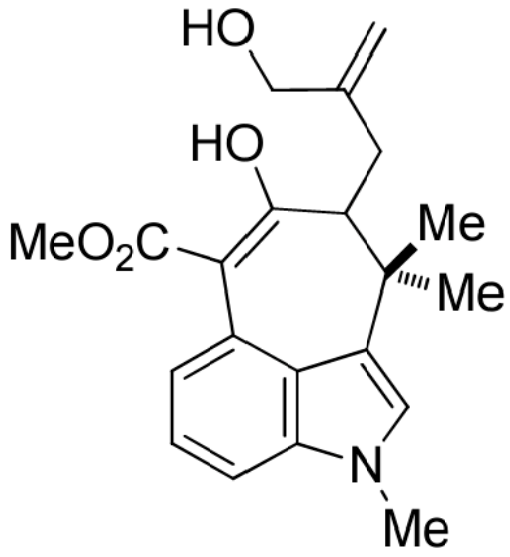
Relax. delay 2.000 sec
Pulse 16.4 degrees
Acq. time 2.856 sec
Width 5602.2 Hz
16 repetitions
OBSERVE H1, 400.2669784 MHz
DATA PROCESSING
Line broadening 0.1 Hz
FT size 32768
Total time 0 min, 0 sec

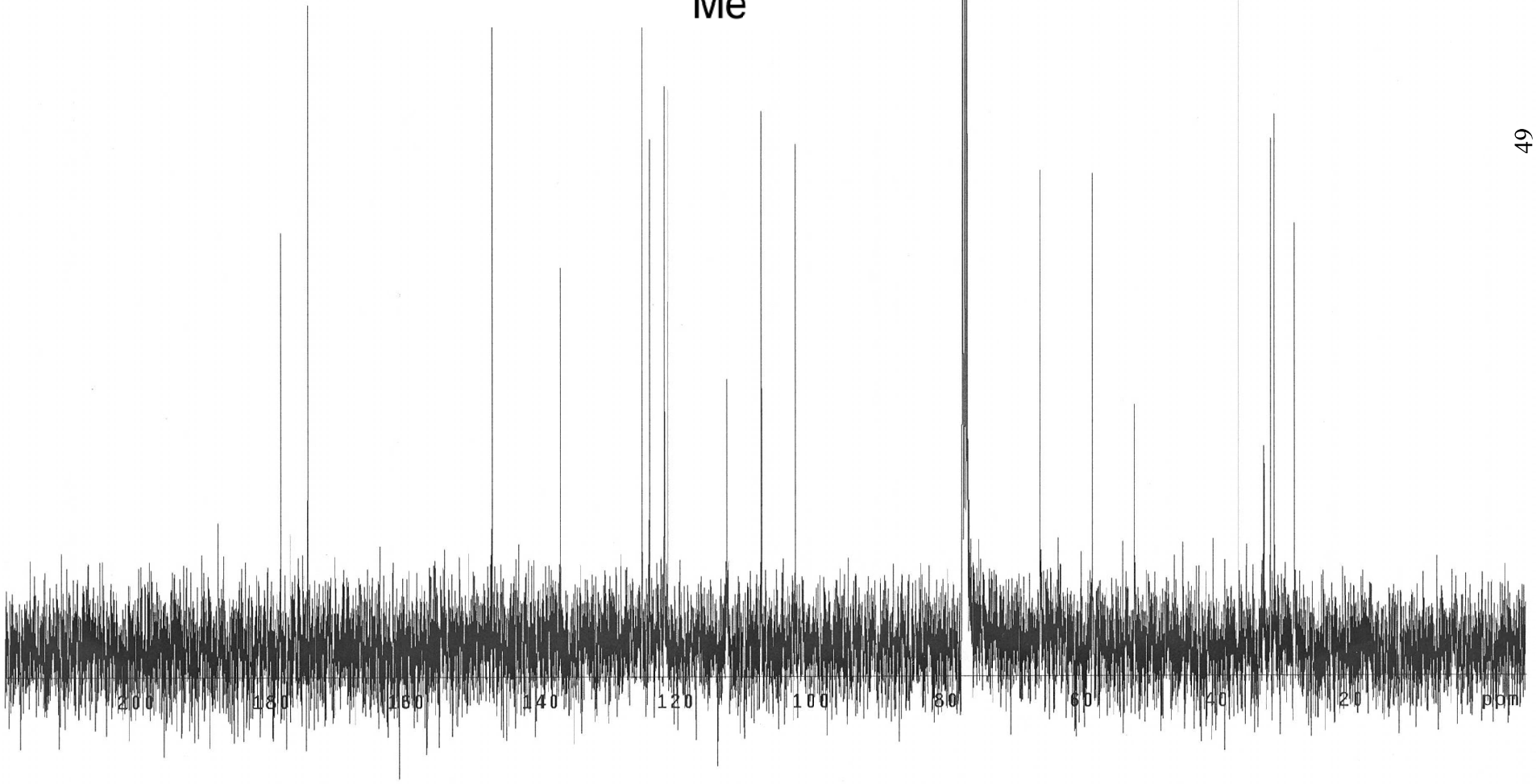


BMGVI-99

Pulse Sequence: s2pul
 Solvent: CDCl3
 Ambient temperature
Mercury-400BB "nmr6"

 Relax. delay 2.000 sec
 Pulse 22.5 degrees
 Acq. time 1.280 sec
 Width 25188.9 Hz
 1024 repetitions
OBSERVE C13, 100.6471877 MHz
DECOUPLE H1, 400.2689955 MHz
 Power 38 dB
 continuously on
 WALTZ-16 modulated
DATA PROCESSING
 Line broadening 1.0 Hz
FT size 65536
Total time 0 min, 0 sec

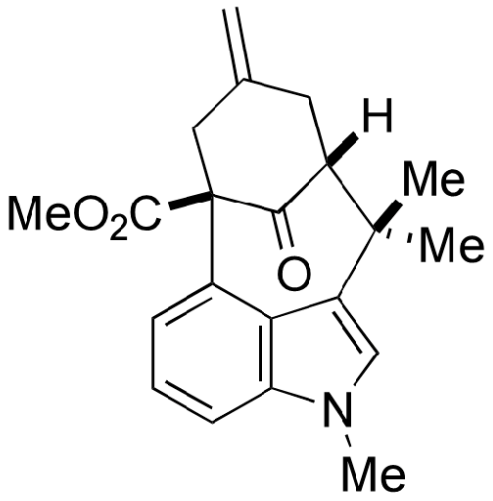




BMG-VI 73
bmgVI73_h1

exp1 s2pul

SAMPLE		DEC. & VT	
date	Oct 26 2005	dfrq	499.867
solvent	CDCl3	dn	H1
file	exp	dpwr	30
ACQUISITION		dof	0
sfrq	499.868	dm	nnn
tn	H1	dmm	c
at	3.996	dmf	200
np	73980	dseq	
sw	9256.0	dres	1.0
fb	not used	homo	n
bs	32	temp	27.0
tpwr	57	PROCESSING	
pw	2.0	lb	0.10
d1	2.000	wtfile	
tof	1124.6	proc	ft
nt	64	fn	not used
ct	64	math	f
alock	n		
gain	30	werr	
FLAGS		wexp	
il	n	wbs	
in	n	wnt	wft
dp	y		
hs	nn		
DISPLAY			
sp	-250.0		
wp	5248.5		
vs	106		
sc	0		
wc	250		
hzmm	20.99		
is	4467.70		
rfl	1025.4		
rfp	0		
th	5		
ins	1.000		
ai	ph		

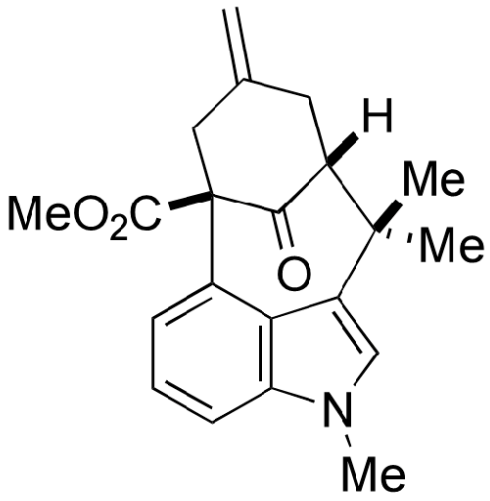


32

ss

50

9 8 7 6 5 4 3 2 1 0 ppm



32

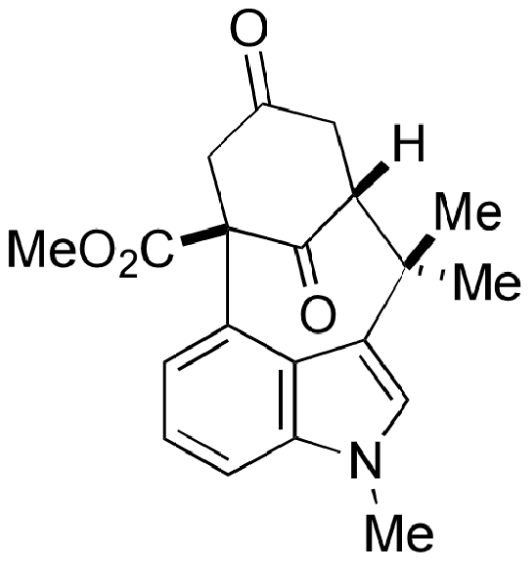
```
BMG-VI 73
bmgVI73_c13

exp4  s2pul

      SAMPLE
date    Oct 26 2005
solvent       CDCl3
file             exp
   ACQUISITION
sfrq        125.705
tn              C13
at              1.279
np              85262
sw          33333.3
fb         not used
bs                64
tpwr              53
pw               3.0
d1             2.000
tof           2198.1
nt             10000
ct             10000
alock              n
gain              60
      FLAGS
il                 n
in                 n
dp                 y
hs                nn
     DISPLAY
sp            -628.6
wp           28280.1
vs              3538
sc                 0
wc               250
hzmm          113.12
is            500.00
rfl          12237.0
rfp           9678.2
th                 5
ins          100.000
ai  cdc  ph

      DEC. & VT
dfrq        499.867
dn               H1
dpwr             37
dof               0
dm              yyy
dmm               w
dmf           10582
dseq
dres            1.0
homo              n
temp           27.0
     PROCESSING
lb              1.00
wtfile
proc              ft
fn         not used
math               f

werr
wexp
wbs
wnt
```

ss

brian
martin
BMG-VI 83
bmgVI83_h1

exp1 s2pul

SAMPLE		DEC. & VT	
date	Nov 8 2005	dfrq	499.867
solvent	CDCl3	dn	H1
file	exp	dpwr	30
ACQUISITION		dof	0
sfrq	499.868	dm	nnn
tn	H1	dmm	c
at	3.996	dmf	200
np	73980	dseq	
sw	9256.0	dres	1.0
fb	not used	homo	n
bs	32	temp	27.0
tpwr	57	PROCESSING	
pw	2.0	lb	0.10
d1	2.000	wtfile	
tof	1124.6	proc	ft
nt	64	fn	not used
ct	64	math	f
alock	n		
gain	30	werr	
FLAGS		wexp	
il	n	wbs	
in	n	wnt	wft
dp	y		
hs	nn		
DISPLAY			
sp	-250.0		
wp	5248.5		
vs	179		
sc	0		
wc	250		
hzmm	20.99		
is	5110.11		
rfl	1025.4		
rfp	0		
th	6		
ins	1.000		
ai ph			



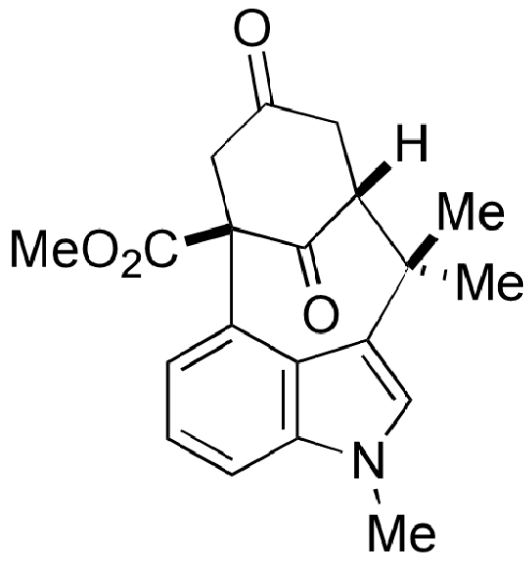
33

ss

brian
martin
BMG-VI 83
bmgVI83_c13

exp4 s2pul

SAMPLE		DEC. & VT	
date	Nov 8 2005	dfrq	499.867
solvent	CDCl3	dn	H1
file	exp	dpwr	37
ACQUISITION		dof	0
sfrq	125.705	dm	yyy
tn	C13	dmm	w
at	1.279	dmf	10582
np	85262	dseq	
sw	33333.3	dres	1.0
fb	not used	homo	n
bs	64	temp	27.0
tpwr	53	PROCESSING	
pw	3.0	lb	1.00
d1	2.000	wtfile	
tof	2198.1	proc	ft
nt	10000	fn	not used
ct	10000	math	f
alock	n		
gain	60	werr	
FLAGS		wexp	
il	n	wbs	
in	n	wnt	
dp	y		
hs	nn		
DISPLAY			
sp	-628.6		
wp	28280.1		
vs	4548		
sc	0		
wc	250		
hzmm	113.12		
is	500.00		
rfl	12236.0		
rfp	9678.2		
th	6		
ins	100.000		
ai cdc ph			



33

ss