Supporting Information

For

Multicomponent Assembly Strategies for the Synthesis of Diverse Tetrahydroisoquinoline Scaffolds

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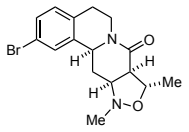
Experimental Section

General Methods. Methanol (MeOH), acetonitrile (CH_3CN), and *N,N*-dimethylformamide (DMF) were dried by filtration through two columns of activated molecular sieves. Tetrahydrofuran (THF) and toluene were passed through two columns of activated neutral alumina prior to use. Triethylamine (Et_3N), *N,N*-diisopropylamine, benzene, dichloromethane (CH_2Cl_2), 1,2-dimethoxyethane (DME), morpholine, piperidine, *trans*-crotonyl chloride, 2-furoyl chloride, phenyl isocyanate, and boron trifluoride diethyl etherate ($BF_3{\cdot}OEt_2$) were freshly distilled over CaH_2. Trimethylsilyl trifluoromethanesulfonate (TMSOTf) was distilled over P_2O_5. Zinc chloride ($ZnCl_2$) was fused by melting under vacuum prior to use. Thionyl chloride ($SOCl_2$) was distilled from triphenylphosphite. All solvents used for palladium-catalyzed cross-coupling reactions were degassed by sparging with nitrogen for 20 min prior to use. All other reagents and solvents were reagent grade and were purchased and used as received unless otherwise noted. Reactions were performed under a nitrogen or argon atmosphere in round-bottom flasks sealed under rubber septa with magnetic stirring, unless otherwise noted. Water sensitive reactions were performed with flame- or oven-dried glassware, stir-bars and steel needles. Reaction temperatures are reported as the temperatures of the bath surrounding the vessel. Sensitive reagents and solvents were transferred using plastic or oven-dried glass syringes and steel needles using standard techniques.

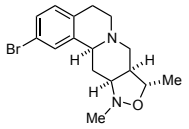
Proton nuclear magnetic resonance (^{1}H NMR) and carbon nuclear magnetic resonance (^{13}C NMR) spectra were acquired in $CDCl_3$ unless otherwise noted. Chemical shifts are reported in parts per million (ppm, δ), downfield from tetramethylsilane (TMS, δ = 0.00 ppm) and are referenced to residual solvent ($CDCl_3$, δ = 7.26 ppm (^{1}H) and 77.16 ppm (^{13}C)). Coupling constants (*J*) are reported in hertz (Hz) and the resonance multiplicity abbreviations used are: s, singlet; d, doublet; t, triplet; dt, doublet of triplets; td, triplet of doublets; dd, doublet of doublets; ddd, doublet of doublet of doublets; m, multiplet; comp, overlapping multiplets of magnetically non-equivalent protons. The abbreviations br and app stand for broad and apparent, respectively. Infrared (IR) spectra were obtained with a Thermo Scientific Nicolet IR-100 FT-IR series spectrometer as thin films on sodium chloride plates. Melting points were determined using a Thomas-Hoover Uni-melt capillary melting point apparatus. Thin-layer chromatography (TLC) was performed on EMD 60 F_{254} glass-backed pre-coated silica gel plates and were visualized

using one or more of the following methods: UV light (254 nm) and staining with basic potassium permanganate ($KMnO_4$) or acidic *p*-anisaldehyde (PAA). Flash chromatography was performed using glass columns and with Silicycle SiliaFlash F60 (40-63 μm) silica gel eluting with the solvents indicated according to the procedure of Still.[1]



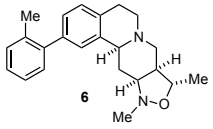
4

4-Bromo-13,15-dimethyl-14-oxa-10,15-diazatetracyclo[8.7.0.0^{2,7}.0^{12,16}]heptadeca-2,4,6-trien-11-one (4). *trans*-Crotonoyl chloride (548 mg, 0.5 mL, 5.24 mmol) was added to a solution of imine **1** (1.0 g, 4.76 mmol) and silyl enol ether **2**[2] (0.90 g, 1.1 mL, 5.71 mmol) in CH_3CN (95 mL) at room temperature. Freshly distilled trimethylsilyl trifluoromethanesulfonate (106 mg, 86 μL, 0.47 mmol) was added, and the reaction was stirred for 0.5 h at room temperature. The mixture was then partitioned between saturated aqueous $NaHCO_3$ (200 mL) and CH_2Cl_2 (200 mL). The layers were separated, and the aqueous layer was extracted with CH_2Cl_2 (2 × 100 mL). The combined organic layers were dried (Na_2SO_4) and concentrated under reduced pressure. The crude amide **3** thus obtained was dissolved in toluene (80 mL) containing *N*-methylhydroxylamine hydrochloride (0.44 g, 5.24 mmol) and Et_3N (577 mg, 0.8 mL, 5.71 mmol), and the mixture was heated at 50 °C for 4 h. The reaction was partitioned between toluene (20 mL) and H_2O (100 mL), and the layers were separated. The aqueous layer was extracted with toluene (2 × 50 mL), and the combined organic layers were dried (Na_2SO_4) and concentrated under reduced pressure. The resultant yellow solid was recrystallized from MeOH to give 1.1 g (66% over 2 steps) of isoxazolidine **4** as small white crystals: mp 190.5-192 °C; ^{1}H NMR (600 MHz) δ 7.34-7.32 (comp, 2 H), 7.05 (d, *J* = 7.8 Hz, 1 H), 4.82-4.77 (m, 1 H), 4.64 (dd, *J* = 12.0, 1.8 Hz, 1 H), 4.33-4.29 (m, 1 H), 3.17 (br s, 1 H), 2.86-2.72 (comp, 4 H), 2.70 (s, 3 H), 2.36 (ddd, *J* = 13.2, 7.2, 2.4 Hz, 1 H), 1.65-1.59 (m, 1 H), 1.52 (d, *J* = 6.0 Hz, 3 H); ^{13}C NMR (150 MHz) δ 169.3, 137.8, 134.3, 130.5, 130.0, 129.1, 120.3, 76.4, 65.0, 53.7, 53.4, 44.0, 38.5, 36.7, 28.8, 20.0; IR (neat) 2966, 2839, 1641, 1432 cm^{-1}; mass spectrum (CI) *m/z* 351.0706 [$C_{16}H_{20}{}^{79}BrN_2O_2$ (M+1) requires 351.0708].[3]



5

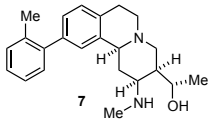
4-Bromo-13,15-dimethyl-14-oxa- 10,15- diazatetracyclo[8.7.0.0^{2,7}.0^{12,16}]heptadeca-2,4,6-triene (5). To a suspension of $NaBH_4$ (57 mg, 1.5 mmol) in anhydrous THF (3 mL) at 0 °C was added $BF_3{\cdot}OEt_2$ (0.25 g, 0.22 mL, 1.8 mmol), and the mixture was stirred at 0 °C for 20 min. A solution of lactam **4** (100 mg, 0.30 mmol) in anhydrous THF (7 mL) was added slowly at 0 °C, and the mixture was warmed to room temperature and stirred for 24 h. A solution of 5 M aqueous HCl (5 mL) was added, and the mixture was heated at 70 °C for 2 h and then cooled to 0 °C. The acidic solution was made basic (pH > 10) with 5 M aqueous NaOH (~7 mL) at 0 °C, and then partitioned with CH_2Cl_2 (10 mL). The layers were separated, and the aqueous layer was extracted with CH_2Cl_2 (2 × 10 mL). The combined organic layers were dried (Na_2SO_4), filtered, and concentrated under reduced pressure. The resultant yellow oil was purified by flash column chromatography eluting with hexanes/EtOAc (1 : 9) to give 79 mg (82%) of **5** as a white solid: mp 140.5-141 °C; ^{1}H NMR (600 MHz) δ 7.31 (d, *J* = 1.8 Hz, 1 H), 7.24 (dd, *J* = 7.8, 1.8 Hz, 1 H), 6.95 (d, *J* = 7.8 Hz, 1 H), 4.31-4.27 (m, 1 H), 3.24 (br s, 1 H), 3.09 (d, *J* = 11.8 Hz, 1 H), 3.06-3.00 (m, 1 H), 2.97-2.96 (m, 1 H), 2.87 (ddd, *J* = 11.4, 5.7, 1.4 Hz, 1 H), 2.75 (s, 3 H), 2.63 (dd, *J* = 11.7, 4.2 Hz, 1 H), 2.63-2.58 (m, 1 H), 2.50-2.47 (m, 1 H), 2.43 (ddd, *J* = 12.0, 11.4, 3.5 Hz, 1 H), 2.43-2.34 (m, 1 H), 1.71-1.67 (m, 1 H), 1.37 (d, *J* = 3.6 Hz, 3 H); ^{13}C NMR (150 MHz) δ 139.8, 133.7, 130.4, 129.1, 128.3, 119.6, 77.0, 65.8, 60.2, 54.1, 51.7, 47.4, 45.9, 35.0, 29.2, 21.3; IR (neat) 2920, 2757, 1114, 908, 731 cm^{-1}; mass spectrum (ESI) *m/z* 337.0910 [$C_{16}H_{22}{}^{79}BrN_2O$ (M+1) requires 337.0916].



6

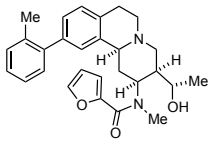
13,15-Dimethyl-4-(2-methylphenyl)-14-oxa-10,15-diazatetracyclo[8.7.0.0^{2,7}.0^{12,16}]-heptadeca-2,4,6-triene (6). A mixture of bromide **5** (50 mg, 0.15 mmol), cesium fluoride (91 mg, 0.59 mmol), 2-methylbenzeneboronic acid (41 mg, 0.30 mmol), and $[PdCl_2(dppf)]{\cdot}CH_2Cl_2$ (6.0 mg, 0.01 mmol) in degassed toluene (1 mL) was heated under reflux for 24 h. The reaction was cooled to room temperature and partitioned between EtOAc (3 mL) and H_2O (3 mL), and the layers were separated. The aqueous layer was extracted with EtOAc (2 × 3 mL), and the

combined organic layers were dried (Na_2SO_4), filtered, and concentrated under reduced pressure. The resultant brown residue was purified by flash column chromatography eluting with EtOAc to give 49 mg (95%) of **6** as a tan solid: mp 151-152 °C; ^{1}H NMR (400 MHz) δ 7.26-7.09 (comp, 7 H), 4.38-4.31 (m, 1 H), 3.24 (br s, 1 H), 3.21-3.13 (comp, 2 H), 3.02-2.98 (m, 1H), 2.91 (ddd, *J* = 11.2, 5.6, 1.6 Hz, 1 H), 2.75 (s, 3 H), 2.75-2.70 (m, 1 H), 2.67 (dd, *J* = 12.2, 4.0 Hz, 1 H), 2.52 (ddd, *J* = 11.6, 11.6, 3.2 Hz, 1 H), 2.53-2.43 (comp, 2 H), 2.26 (s, 3 H), 1.79-1.70 (m, 1 H), 1.38 (d, *J* = 5.6 Hz, 3 H); ^{13}C NMR (100 MHz) δ 141.9, 139.5, 137.3, 135.4, 133.2, 130.3, 129.8, 128.5, 127.1, 126.9, 126.1, 125.7, 77.0, 65.9, 60.6, 54.2, 52.0, 47.4, 46.1, 35.1, 29.5, 21.4, 20.6; IR (neat) 2919, 2755, 1482, 1459, 911, 759, 730; mass spectrum (ESI) *m/z* 349.2273 [$C_{23}H_{29}N_2O$ (M+1) requires 349.2280].



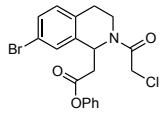
7

2-Methylamino-10-*o*-tolyl-2,3,4,6,7,11*b*-hexahydro-1*H*-pyrido[2,1-*a*]isoquinolin-3-yl)ethanol (7). Sodium borohydride (33 mg, 0.86 mmol) was added to a solution of isoxazolidine **6** (50 mg, 0.14 mmol) and nickel(II) chloride hexahydrate (72 mg, 0.29 mmol) in anhydrous MeOH (2.9 mL), and the reaction was stirred for 5 h at room temperature. The solvent was removed in vacuo, and the black residue was partitioned between concentrated NH_4OH (28 mL) and CH_2Cl_2 (28 mL) and stirred for 12 h at room temperature. The layers were separated, and the aqueous layer was extracted with CH_2Cl_2 (2 × 10 mL). The combined organic layers were dried (Na_2SO_4), filtered, and concentrated under reduced pressure. The resultant yellow oil was purified by flash column chromatography eluting with EtOAc/MeOH (5 : 1) containing 1% Et_3N to give 45 mg (90%) of **7** as a clear gum: ^{1}H NMR (400 MHz) δ 7.26-7.12 (comp, 7 H), 4.49-4.42 (m, 1 H), 3.24 (d, *J* = 10.4 Hz, 1 H), 3.16 (ddd, *J* = 18.0, 12.0, 6.4 Hz, 1 H), 2.96 (dd, *J* = 12.0, 6.4 Hz, 1 H), 2.97-2.92 (m, 1 H), 2.90-2.85 (m, 1 H), 2.72 (dd, *J* = 16.0, 3.2 Hz, 1 H), 2.53 (s, 3 H), 2.46 (ddd, *J* = 12.0, 12.0, 4.0 Hz, 1 H), 2.34-2.28 (comp, 2 H), 2.27 (s, 3 H), 1.95-1.83 (comp, 2 H), 1.23 (d, *J* = 6.0 Hz, 3 H); ^{13}C NMR (100 MHz) δ 141.9, 139.4, 137.4, 135.4, 133.2, 130.3, 129.8, 128.6, 127.2, 127.0, 125.7, 125.2, 67.0, 62.9, 62.5, 57.7, 52.6, 41.9, 34.3, 33.8, 29.5, 21.4, 20.6; IR (neat) 3200, 2926, 2802, 1482, 1118, 910, 760, 731; mass spectrum (ESI) *m/z* 351.2431 [$C_{23}H_{31}N_2O$ (M+1) requires 351.2436].



8

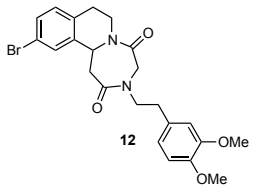
***N*-(3-(1-Hydroxyethyl)-10-*o*-tolyl-2,3,4,6,7,11*b*-hexahydro-1*H*-pyrido[2,1-*a*]isoquinolin-2-yl)-*N*-methylfuran-2-carboxamide (8)**. Freshly distilled 2-furoyl chloride (8.8 mg, 6.7 μL, 0.07 mmol) was added to a solution of amine **7** (22 mg, 0.06 mmol) and Et_3N (7.6 mg, 10 μL, 0.07 mmol) in CH_2Cl_2 (1 mL) at 0 °C. The mixture was warmed to room temperature, and stirred for 3 h. The reaction was partitioned between CH_2Cl_2 (2 mL) and saturated aqueous $NaHCO_3$ (2 mL), and the layers were separated. The aqueous layer was extracted with CH_2Cl_2 (2 × 2 mL), and the combined organic layers were dried (Na_2SO_4), filtered, and concentrated under reduced pressure. The resultant yellow oil was purified by flash column chromatography eluting with hexanes/EtOAc (1 : 1 → 1 : 2) to give 21 mg (77%) of **8** as a pale yellow oil: ^{1}H NMR (400 MHz) δ 7.49 (dd, *J* = 1.6, 0.8 Hz, 1 H), 7.27-7.14 (comp, 7 H), 6.97 (d, *J* = 3.2 Hz, 1 H), 6.48 (dd, *J* = 3.2, 1.6 Hz, 1 H), 4.74 (ddd, *J* = 12.8, 4.8, 4.8 Hz, 1 H), 4.36-4.25 (m, 1 H), 3.47 (d, *J* = 9.6 Hz, 1 H), 3.33 (s, 3 H), 3.23-3.16 (m, 1 H), 3.07-2.97 (comp, 2 H), 2.85 (dd, *J* = 11.4, 2.4 Hz, 1 H), 2.80-2.76 (m, 1 H), 2.58 (ddd, *J* = 12.0, 11.4, 3.2 Hz, 1 H), 2.53-2.32 (comp, 3 H), 2.28 (s, 3 H), 1.25 (d, *J* = 7.2 Hz, 3 H); ^{13}C NMR (100 MHz) δ 161.0, 148.3, 143.8, 141.7, 139.7, 136.8, 135.4, 132.7, 130.4, 129.8, 128.6, 127.4, 127.3, 125.8, 125.5, 116.0, 111.1, 77.2, 73.3, 63.3, 62.7, 56.2, 52.3, 33.6, 33.4, 29.6, 21.3, 20.6; IR (neat) 3395, 2928, 1613, 1485, 1073, 909, 758, 731; mass spectrum (ESI) *m/z* 445.2486 [$C_{28}H_{33}N_2O_3$ (M+1) requires 445.2491].



10

Phenyl 2-(7-bromo-2-(2-chloroacetyl)-1,2,3,4-tetrahydroisoquinolin-1-yl)acetate (10). Trimethylsilyl trifluoromethanesulfonate (27.1 mg, 22 μL, 0.12 mmol) was added to a solution of imine **1** in THF (4.8 mL) at −78 °C. Silyl ketene acetal **9**[4] (743 mg, 3.57 mmol) and chloroacetyl chloride (162 mg, 114 μL, 1.43 mmol) were added, and the reaction was stirred at −78 °C for 22 h. The reaction was partitioned between CH_2Cl_2 (20 mL) and H_2O (20 mL), and the layers were separated. The aqueous layer was extracted with CH_2Cl_2 (2 × 15 mL), and the combined organic layers were dried (Na_2SO_4) and concentrated under reduced pressure. The

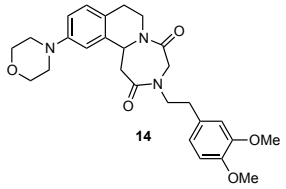
resultant yellow oil was purified by flash column chromatography eluting with hexanes/EtOAc (7 : 3) to give 446 mg (89%) of **10** as a white powder: mp 139-141°C; ^{1}H NMR (600 MHz) (rotamers) δ 7.47 (d, *J* = 1.8 Hz, 0.63 H), 7.42-7.36 (comp, 3.45 H), 7.27-7.28 (m, 0.30 H), 7.23-7.21 (m, 0.67 H), 7.11-7.09 (comp, 1.27 H), 7.06-7.05 (comp, 1.71 H), 6.07 (t, *J* = 6.6 Hz, 0.64 H), 5.48 (dd, *J* = 9.6, 4.8 Hz, 0.36 H), 4.73 (dd, *J* = 13.2, 6.0 Hz, 0.38 H), 4.54 (d, *J* = 12.6 Hz, 0.37 H), 4.19-4.09 (comp, 1.69 H), 3.93 (ddd, *J* = 9.0, 4.8, 3.6 Hz, 0.66 H), 3.73 (ddd, *J* = 13.2, 10.2, 4.2 Hz, 0.65 H), 3.24 (dd, *J* = 16.2, 9.6 Hz, 0.38 H), 3.19 (ddd, *J* = 13.2, 12.0, 4.2 Hz, 0.38 H), 3.10-2.94 (comp, 2.73 H), 2.88 (ddd, *J* = 7.8, 4.2, 4.2 Hz, 0.66 H), 2.77-2.74 (m, 0.37 H); ^{13}C NMR (150 MHz) (rotamers) δ 169.3, 168.6, 166.2, 165.8, 150.5, 150.1, 137.3, 137.0, 133.0, 132.4, 131.3, 131.0, 130.7, 130.5, 130.0, 129.7, 129.5, 129.2, 126.4, 126.0, 121.6, 121.3, 120.5, 120.2, 53.4, 50.1, 41.7, 41.2, 41.1, 40.6, 35.9, 28.6, 27.1; IR (neat) 2946, 1751, 1654, 1485, 1430, 1193, 1136, 816, 750, 690 cm^{-1}; mass spectrum (CI) *m/z* 422.0156 [$C_{19}H_{18}{}^{79}Br^{35}ClNO_3$ (M+1) requires 422.0159].



12

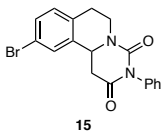
11-Bromo-3-(3,4-dimethoxyphenethyl)-1,3,4,7,8,12*b*-hexahydro-[1,4]diazepino[7,1-*a*]isoquinoline-2,5-dione (12). A solution of amide **10** (30 mg, 0.07 mmol), 3,4-dimethoxyphenethylamine (**11**) (15 mg, 14 μL, 0.09 mmol), and *N,N*-diisopropylethylamine (12 mg, 16 μL, 0.09 mmol) in CH_3CN (1.8 mL) was heated at 75 °C for 24 h. The reaction was cooled to room temperature and partitioned between CH_2Cl_2 (15 mL) and saturated aqueous $NaHCO_3$ (15 mL). The layers were separated, and the aqueous layer was extracted with CH_2Cl_2 (2 × 15 mL). The combined organic layers were dried (Na_2SO_4) and concentrated under reduced pressure, and the resultant yellow oil was purified by flash column chromatography eluting with toluene/EtOAc (2 : 8) to provide 23 mg (69%) of **12** as a white solid: mp 160-161 °C; ^{1}H NMR (600 MHz) δ 7.37-7.35 (comp, 2 H), 7.03 (d, *J* = 7.8 Hz, 1 H), 6.80-6.75 (comp, 3 H), 4.91 (dd, *J* = 12.0, 3.6 Hz, 1 H), 4.30 (ddd, *J* = 9.0, 4.2, 4.2 Hz, 1 H), 4.14 (d, *J* = 16.8 Hz, 1 H), 4.07 (d, *J* = 16.8 Hz, 1 H), 3.88-3.81 (comp, 7 H), 3.67-3.65 (m, 1 H), 3.14 (dd, *J* = 15.6, 3.6 Hz, 1 H), 3.10 (ddd, *J* = 12.6, 10.2, 3.6 Hz, 1 H), 3.02 (dd, *J* = 15.6, 12.0 Hz, 1 H), 2.91 (ddd, *J* = 16.2, 10.8, 5.4 Hz, 1 H), 2.87-2.80 (comp, 2 H), 2.72 (ddd, *J* = 16.2, 4.2, 4.2 Hz, 1H); ^{13}C NMR (150 MHz) δ

168.8, 167.2, 149.0, 147.7, 137.3, 134.3, 130.8, 130.7, 130.5, 128.5, 120.8, 120.4, 112.1, 111.3, 56.0, 55.9, 54.5, 53.9, 50.1, 41.7, 41.4, 33.7, 27.9; IR (neat) 2935, 1662, 1635, 1516, 1465, 1262, 731cm $^{-1}$; mass spectrum (CI) *m/z* 473.1070 [$C_{23}H_{26}{}^{79}BrN_2O_4$ (M+1) requires 473.1076].



14

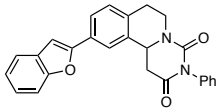
3-(3,4-Dimethoxyphenethyl)-11-morpholino-1,3,4,7,8,12*b*-hexahydro-[1,4]diazepino[7,1-*a*]isoquinoline-2,5-dione (14). A suspension of (±)-BINAP (3.3 mg, 0.01 mmol) in toluene (0.60 mL) was heated at 80 °C until a homogeneous solution was observed (~5 min). The reaction was cooled to room temperature, whereupon $Pd(OAc)_2$ (0.9 mg, 0.01 mmol) was added. The reaction was stirred for 1 min at room temperature. Morpholine (**13**) (7.5 mg, 7.4 µL, 0.09 mmol) was added, followed by bromide **12** (33.5 mg, 0.07 mmol) and NaO*t*-Bu (9.6 mg, 0.10 mmol). The reaction was heated at 80 °C for 2 h and then cooled to room temperature. The mixture was filtered through a pad of celite and concentrated under reduced pressure. The resultant yellow residue was purified by flash column chromatography eluting with EtOAc/MeOH (9 : 1) to yield 24 mg (72%) of **14** as a yellow solid: mp 69-70 °C; ^{1}H NMR (600 MHz) δ 7.07 (d, *J* = 8.4 Hz, 1 H), 6.82 (dd, *J* = 8.4, 2.4 Hz, 1 H), 6.79-6.78 (m, 1 H), 6.76-6.75 (comp, 2 H), 6.73 (d, *J* = 2.4 Hz, 1 H), 4.95 (dd, *J* = 12.0, 3.6 Hz, 1 H), 4.27 (ddd, *J* = 9.6, 4.8, 4.8 Hz, 1 H), 4.22 (d, *J* = 16.8 Hz, 1 H), 4.03 (d, *J* = 16.8 Hz, 1 H), 3.88-3.80 (comp, 11 H), 3.67 (ddd, *J* = 13.8, 8.4, 6.6 Hz, 1 H), 3.18-3.11 (comp, 6 H), 3.01 (dd, *J* = 16.2, 12.0 Hz, 1 H), 2.90 (ddd, *J* = 15.6, 10.8, 5.4 Hz, 1 H), 2.86-2.81 (m, 2 H), 2.70 (ddd, *J* = 15.6, 4.2 Hz, 1 H); ^{13}C NMR (150 MHz) δ 169.2, 167.3, 150.4, 148.9, 147.7, 135.8, 130.7, 129.7, 126.6, 120.8, 115.6, 112.7, 112.0, 111.3, 66.8, 56.0, 55.9, 55.0, 53.9, 50.5, 49.6, 42.2, 41.9, 33.7, 27.5; IR (neat) 2934, 2854, 1659, 1634, 1515, 1450, 1262, 1239, 730 cm^{-1}; mass spectrum (CI) *m/z* 480.2495 [$C_{27}H_{34}N_3O_5$ (M+1) requires 480.2498].



15

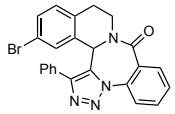
10-Bromo-3-phenyl-6,7-dihydro-1*H*-pyrimido[6,1-*a*]isoquinoline-2,4(3*H*,11*bH*)-dione (**15**). Trimethylsilyl trifluoromethanesulfonate (16 mg, 13 µL, 0.071 mmol) was added slowly to a solution of 7-bromodihydroisoquinoline (**1**) (30 mg, 0.14 mmol) in DME (1.5 mL) at

−40 °C. Silyl ketene acetal **9** (45 mg, 0.21 mmol) was added, and the reaction was stirred for 14 h at −40 °C, whereupon phenyl isocyanate (51 mg, 47 μL, 0.43 mmol) was added. The reaction was warmed to room temperature and stirred for 2 h. The reaction mixture was partitioned between saturated aqueous $NaHCO_3$ (10 mL) and CH_2Cl_2 (10 mL). The layers were separated, and the aqueous layer was extracted with CH_2Cl_2 (2 × 10 mL). The combined organic layers were dried (Na_2SO_4) and concentrated under reduced pressure. The resultant yellow solid was purified by flash chromatography eluting with hexanes/EtOAc (65 : 35) to give 33 mg (63%) of **15** as a white solid: mp 219-220 °C; ^{1}H NMR (500 MHz) δ 7.48-7.45 (comp, 2 H), 7.42-7.39 (comp, 2 H), 7.34-7.32 (m, 1 H), 7.21-7.19 (comp, 2 H), 7.11 (d, *J* = 8.0 Hz, 1 H), 4.99 (dd, *J* = 13.5, 3.5 Hz, 1 H), 4.63 (ddd, *J* = 12.5, 4.5, 2.5 Hz, 1 H), 3.23 (dd, *J* = 16.0, 3.5 Hz, 1 H), 3.11 (ddd, *J* = 12.0, 12.0, 3.5 Hz, 1 H), 2.99 (ddd, *J* = 16.5, 11.5, 5.0 Hz, 1 H), 2.86-2.80 (comp, 2 H); ^{13}C NMR (125 MHz) δ 168.0, 153.5, 135.7, 135.4, 133.3, 130.9, 130.7, 129.2, 128.7, 128.6, 128.5, 120.7, 50.4, 40.7, 40.4, 28.8; IR (neat) 2924, 2862, 1723, 1678, 1431, 1287, 1248, 699 cm^{-1}; mass spectrum (ESI) *m/z* 371.0391 [$C_{18}H_{16}{}^{79}BrN_2O_2$ (M+1) requires 371.0395].



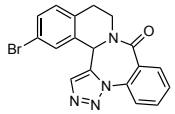
16

10-(Benzofuran-2-yl)-3-phenyl-6,7-dihydro-1*H*-pyrimido[6,1-*a*]isoquinoline-2,4(3*H*,11*bH*)-dione (16). A mixture of bromide **15** (31 mg, 0.08 mmol), cesium carbonate (55 mg, 0.17 mmol), benzofuran-2-ylboronic acid (27 mg, 0.17 mmol), and bis(tri-*tert*-butylphosphine)palladium(0) (0.5 mg, 0.001 mmol) in degassed toluene (1.1 mL) was heated at 90 °C for 3 h. The reaction was cooled to room temperature and filtered through a pad of celite. The filtrate was concentrated under reduced pressure and purified by flash chromatography eluting with hexanes/EtOAc (6 : 4) to provide 31 mg (92%) of **16** as a white solid: mp 240-241 °C (colorless crystals from *i*-PrOH); ^{1}H NMR (400 MHz) δ 7.76-7.72 (comp, 2 H), 7.60 (d, *J* = 7.6 Hz, 1 H), 7.53 (d, *J* = 8.4 Hz, 1 H), 7.51-7.40 (comp, 3 H), 7.34-7.23 (comp, 5 H), 7.03 (s, 1 H), 5.11 (dd, *J* = 13.6, 3.2 Hz, 1 H), 4.67 (ddd, *J* = 7.2, 4.4, 4.4 Hz, 1 H), 3.40 (dd, *J* = 16.8, 3.6 Hz, 1 H), 3.22-3.06 (comp, 2 H), 2.95-2.87 (comp, 2 H); ^{13}C NMR (100 MHz) δ 168.4, 154.9, 153.6, 135.6, 134.6, 134.2, 129.8, 129.6, 129.2, 129.0, 128.7, 128.5, 124.6, 124.1, 123.1, 121.9, 121.0, 111.2, 101.6, 50.8, 40.9, 40.4, 29.1; IR (neat) 3061, 2924, 1723, 1676, 1432, 1249, 751, 698 cm^{-1}; mass spectrum (ESI) *m/z* 409.1552 [$C_{26}H_{21}N_2O_3$ (M+1) requires 409.1552].



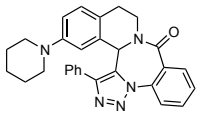
17

20-Bromo-4,5,6,14-tetraazapentacyclo[12.8.0.0^{2,6}.0^{7,12}.0^{7,22}]docosa-2,4,7,9,11,17(22),18,20-octaen-13-one (17). Trimethylsilyl trifluoromethanesulfonate (58 mg, 47 μL, 0.26 mmol) was added slowly to a solution of dihydroisoquinoline **1** (50 mg, 0.24 mmol) in THF (1.8 mL) at −23 °C. The reaction was then cooled to −78 °C. In a separate flask, *n*-BuLi (0.13 mL, 0.31 mmol, 2.36 M in hexanes) was added dropwise to a solution of phenylacetylene (34 mg, 36 μL, 0.33 mmol) in THF (1.0 mL), and the brown solution was stirred 10 min at room temperature. The solution was cooled to −78 °C, whereupon $ZnCl_2$ (0.38 mL, 0.38 mmol, 1 M in THF) was added dropwise, and the yellow solution stirred for 20 min at −78 °C.[7] The solution of freshly prepared organozinc reagent was added dropwise to the solution of activated imine at −78 °C and the reaction stirred 5 h at −78 °C, whereupon *o*-azidobenzoyl chloride[8] (70 mg, 0.43 mmol) in THF (2.0 mL) was added slowly at −78 °C. The bath was removed, and the reaction was stirred at room temperature for 20 h. The reaction mixture was partitioned between saturated aqueous $NaHCO_3$ (20 mL) and CH_2Cl_2 (20 mL). The layers were separated, and the aqueous layer was extracted with CH_2Cl_2 (2 × 20 mL). The combined organic layers were washed with brine (1 × 30 mL), dried (Na_2SO_4) and concentrated under reduced pressure. The resultant yellow solid was purified by flash chromatography eluting with toluene/EtOAc (4 : 1) to give 88 mg (80%) of **17** as a tan solid: mp 270 °C (decomposition); ^{1}H NMR (500 MHz) δ 8.12-8.09 (comp, 2 H), 7.77 (ddd, *J* = 8.1, 7.3, 1.5 Hz, 1 H), 7.62 (td, *J* = 7.8, 1.5 Hz, 1 H), 7.17-7.13 (comp, 2 H), 7.09-7.06 (comp, 2 H), 6.98-6.96 (comp, 3 H), 6.86 (d, *J* = 8.0 Hz, 1 H), 5.90 (s, 1 H), 4.38 (ddd, *J* = 10.7, 5.6, 5.6 Hz, 1 H), 3.63 (ddd, *J* = 13.4, 8.7, 5.6 Hz, 1 H), 2.89-2.78 (comp, 2 H); ^{13}C NMR (125 MHz) δ 165.8, 144.0, 134.4, 133.6, 133.1, 132.7, 131.8, 131.4, 130.9, 129.9, 129.8, 129.4, 129.3, 128.7, 128.1, 127.7, 127.4, 123.1, 120.2, 49.9, 38.8, 28.1; IR (neat) 3052, 2935, 1651, 1401, 760, 698 cm^{-1}; mass spectrum (CI) *m/z* 457.0664 [$C_{24}H_{18}{}^{79}BrN_4O$ (M+1) requires 457.0664].



18

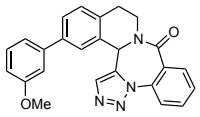
20-Bromo-4,5,6,14-tetraazapentacyclo[12.8.0.0^{2,6}.0^{7,12}.0^{17,22}]docosa-2,4,7(12),8,10,17,19,21-octaen-13-one (18). Trimethylsilyl trifluoromethanesulfonate (58 mg, 47 µL, 0.26 mmol) was added slowly to a solution of dihydroisoquinoline **1** (50 mg, 0.24 mmol) in THF (3.5 mL) at −23 °C. The reaction was then cooled to −78 °C. Ethynylmagnesium bromide (0.62 mL, 0.31 mmol, 0.5 M in THF) was then added dropwise and the reaction stirred 5 h at −78 °C, whereupon *o*-azidobenzoyl chloride[8] (70 mg, 0.43 mmol) in THF (1.0 mL) was added slowly. The cooling bath was removed, and the reaction was stirred at room temperature for 20 h. The reaction mixture was partitioned between saturated aqueous $NaHCO_3$ (20 mL) and CH_2Cl_2 (20 mL). The layers were separated, and the aqueous layer was extracted with CH_2Cl_2 (2 × 20 mL). The combined organic layers were dried (Na_2SO_4) and concentrated under reduced pressure. The resultant yellow oil was purified by flash chromatography eluting with pentane/EtOAc (1 : 1) to give 85 mg (93%) of **18** as a yellow solid: mp 213-215 °C; ^{1}H NMR (600 MHz) δ 8.12 (dd, *J* = 7.8, 1.2 Hz, 1 H), 8.06 (d, *J* = 7.8 Hz, 1 H), 7.77 (m, 1 H), 7.63 (app t, *J* = 7.2 Hz, 1 H), 7.52 (dd, *J* = 8.4, 1.8 Hz, 1 H), 7.37 (m, 1 H), 7.24 (d, *J* = 8.4 Hz, 1 H), 7.17 (s, 1 H), 5.74 (s, 1 H), 3.99 (ddd, *J* = 12.6, 6.6, 6.6 Hz, 1 H), 3.85 (ddd, *J* = 12.6, 6.6, 6.6. Hz, 1 H), 3.00-2.96 (comp, 2 H); ^{13}C NMR (150 MHz) δ 166.0, 139.6, 135.2, 133.2, 132.6, 132.2, 132.0, 131.4, 130.5, 130.4, 130.2, 129.4, 127.3, 122.8, 120.7, 49.2, 40.1, 28.2; IR (neat) 2926, 1637, 1488, 1403, 1250, 760, 732 cm^{-1}; mass spectrum (CI) *m/z* 381.0352 [$C_{18}H_{14}{}^{79}BrN_4O$ (M+1) requires 381.0351].



21

3-Phenyl-20-(piperidin-1-yl)-4,5,6,14-tetraazapentacyclo[12.8.0.0^{2,6}.0^{7,12}.0^{17,22}]-docosa-2,4,7(12),8,10,17,19,21-octaen-13-one (21). A suspension of (±)-BINAP (15 mg, 0.03 mmol) in toluene (2.3 mL) was heated at 80 °C until a homogeneous solution was observed (~5 min). The reaction was cooled to room temperature, whereupon $Pd(OAc)_2$ (3.7 mg, 0.02 mmol)

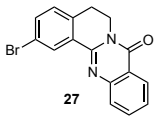
was added. The reaction was stirred for 1 min at room temperature. Piperidine (34 mg, 39 μL, 0.39 mmol) was added, followed by bromide **17** (150 mg, 0.33 mmol) and NaO*t*-Bu (44 mg, 0.46 mmol), and the reaction was heated at 80 °C for 2 h. The reaction mixture was cooled to room temperature, filtered through a pad of celite, and concentrated under reduced pressure. The resultant yellow residue was purified by flash column chromatography eluting with hexanes/EtOAc (1 : 1) to yield 121 mg (80%) of **21** as a yellow solid: mp 267 °C (decomposition); ^{1}H NMR (500 MHz) δ 8.15-8.12 (comp, 2 H), 7.77 (ddd, *J* = 8.0, 7.5, 1.5 Hz, 1 H), 7.64 (ddd, *J* = 7.5, 7.5, 1.0 Hz, 1 H), 7.16-7.12 (m, 1 H), 7.08-7.03 (comp, 4 H), 6.94 (d, *J* = 8.5 Hz, 1 H), 6.68 (dd, *J* = 8.5, 2.4 Hz, 1 H), 6.28 (d, *J* = 2.4 Hz, 1 H), 5.91 (s, 1 H), 4.53 (ddd, *J* = 13.0, 5.0, 5.0 Hz, 1 H), 3.56 (ddd, *J* = 13.5, 10.0, 4.5 Hz, 1 H), 2.93-2.79 (comp, 2 H), 2.73-2.66 (comp, 4 H), 1.52-1.40 (comp, 6 H); ^{13}C NMR (125 MHz) δ 165.8, 151.3, 143.8, 134.3, 132.9, 132.8, 131.7, 129.8, 129.3, 129.1, 128.7, 128.2, 128.0, 127.7, 127.6, 125.6, 123.1, 117.6, 114.9, 50.8, 50.5, 39.2, 27.6, 25.5, 24.1; IR (neat) 3051, 2934, 2853, 2808, 1642, 1512, 1404, 1247, 1130, 761, 734, 696 cm^{-1}; mass spectrum (CI) *m/z* 462.2280 [$C_{29}H_{28}N_5O$ (M+1) requires 462.2294].



22

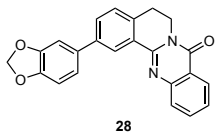
20-(3-Methoxyphenyl)-4,5,6,14-tetraazapentacyclo[12.8.0.0^{2,6}.0^{7,12}.0^{17,22}]docosa-2,4,7(12),8,10,17,19,21-octaen-13-one (22). A solution of bromide **18** (43 mg, 0.11 mmol), cesium carbonate (74 mg, 0.23 mmol), 3-methoxyphenylboronic acid (34 mg, 0.23 mmol), and bis(tri-*tert*-butylphosphine)palladium(0) (0.6 mg, 0.001 mmol) in degassed toluene (1 mL) was heated at 90 °C for 3 h. The reaction was cooled to room temperature, filtered through a pad of celite, and concentrated under reduced pressure. The resultant brown solid was purified by flash chromatography eluting with hexanes/EtOAc (1 : 1) to give 42 mg (90%) of biphenyl **22** as a white solid: mp 225 °C (decomposition); ^{1}H NMR (500 MHz) δ 8.15 (dd, *J* = 8.0, 1.5 Hz, 1 H), 8.07 (dd, *J* = 8.0, 1.0 Hz, 1 H), 7.76 (ddd, *J* = 9.0, 8.0, 1.0 Hz, 1 H), 7.62 (ddd, *J* = 9.0, 8.0, 1.5 Hz, 1 H), 7.61 (dd, *J* = 8.0, 2.0 Hz, 1 H), 7.42-7.41 (comp, 2 H), 7.32 (app t, *J* = 8.0 Hz, 1 H), 7.17 (s, 1 H), 7.09 (ddd, *J* = 7.5, 2.5, 1.5, 1.0 Hz, 1 H), 7.04-7.03 (m, 1 H), 6.88 (ddd, *J* = 8.0, 2.5, 0.5 Hz, 1 H), 5.85 (s, 1 H), 4.02 (ddd, *J* = 12.5, 6.0, 6.0 Hz, 1 H), 3.94-3.89 (m, 1 H), 3.83

(s, 3 H), 3.05 (app t, J = 6.0 Hz, 2 H); ^{13}C NMR (125 MHz) δ 166.2, 160.1, 141.3, 140.3, 140.2, 135.2, 133.0, 132.7, 132.2, 130.3, 129.9, 129.8, 129.4, 129.3, 127.6, 127.5, 126.1, 122.7, 119.4, 113.0, 112.8, 55.3, 49.8, 40.4, 28.3; IR (neat) 2935, 1637, 1606, 1486, 1401, 1227, 785, 760, 734, 699 cm^{-1}; mass spectrum (CI) *m/z* 409.1669 [$C_{25}H_{21}N_4O_2$ (M+1) requires 409.1665].



27

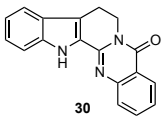
2-Bromo-5*H*-isoquinolino[1,2-*b*]quinazolin-8(6*H*)-one (27). A solution of *o*-azidobenzoic acid (214 mg, 1.3 mmol) in thionyl chloride (6.52 g, 4.0 mL, 55 mmol) was heated under reflux for 3 h. The cooled solution was concentrated under reduced pressure, and the residue was azeotroped with anhydrous benzene (3 × 4 mL).[8] The yellow oil was dissolved in 1,2-dichloroethane (2.0 mL) and added to dihydroisoquinoline **1** (250 mg, 1.19 mmol) in 1,2-dichloroethane (5.0 mL). The yellow solution was heated under reflux for 1.5 h. After cooling to room temperature, the reaction was partitioned between saturated aqueous $NaHCO_3$ (10 mL) and CH_2Cl_2 (10 mL). The layers were separated, and the aqueous layer was extracted with CH_2Cl_2 (3 × 10 mL). The combined organic layers were dried (Na_2SO_4) and concentrated under reduced pressure. The resultant yellow solid was purified by flash chromatography eluting with CH_2Cl_2 to give 242 mg (62%) of **27** as a yellow solid: mp 184-186 °C; ^{1}H NMR (500 MHz) δ 8.55 (d, J = 2.2 Hz, 1 H), 8.22 (dt, J = 8.0, 1.2 Hz, 1 H), 7.69-7.68 (comp, 2 H), 7.50 (dd, J = 8.0, 1.9 Hz, 1 H), 7.40 (ddd, J = 8.0, 4.4, 3.6 Hz, 1 H), 7.09 (d, J = 8.0 Hz, 1 H), 4.32 (t, J = 6.3 Hz, 2 H), 2.98 (t, J = 6.3 Hz, 2 H); ^{13}C NMR (125 MHz) δ 160.5, 146.9, 146.5, 134.7, 133.5, 133.4, 130.3, 129.7, 128.1, 126.7, 125.9, 125.8, 120.4, 119.8, 38.4, 26.0; IR (neat) 3458, 1675, 1556, 1473, 1423, 1239, 772, 693 cm^{-1}; mass spectrum (CI) *m/z* 327.0135 [$C_{16}H_{12}N_2O^{79}Br$ (M+1) requires 327.0133].



28

2-(Benzo[*d*][1,3]dioxol-5-yl)-5*H*-isoquinolino[1,2-*b*]quinazolin-8(6*H*)-one (28). A mixture of quinazolone **27** (50 mg, 0.15 mmol), cesium carbonate (100 mg, 0.31 mmol), benzo[*d*][1,3]dioxol-5-ylboronic acid (51 mg, 0.31 mmol), and bis(tri-*tert*-butylphosphine)-

palladium(0) (0.8 mg, 0.002 mmol) in degassed 1,4-dioxane (0.8 mL) was heated at 90 °C for 3 h. The reaction was cooled to room temperature and filtered through a pad of celite. The filtrate was concentrated under reduced pressure and purified by flash chromatography eluting with CH_2Cl_2 to provide 53 mg (94%) of quinazolone **28** as a cream colored solid: mp 220-221 °C; ^{1}H NMR (500 MHz) δ 8.65 (d, *J* = 1.5 Hz, 1 H), 8.33 (dd, *J* = 8.0, 1.0 Hz, 1 H), 7.81-7.75 (comp, 2 H), 7.63 (dd, *J* = 8.0, 1.5 Hz, 1 H), 7.47 (ddd, *J* = 8.0, 7.0, 1.5 Hz, 1 H), 7.33 (d, *J* = 8.0 Hz, 1 H), 7.18-7.16 (comp, 2 H), 6.92 (d, *J* = 7.5 Hz, 1 H), 6.03 (s, 2 H), 4.44 (t, *J* = 6.5 Hz, 2 H), 3.13 (t, *J* = 6.5 Hz, 2 H); ^{13}C NMR (125 MHz) δ 161.8, 149.3, 148.3, 147.8, 147.4, 140.5, 135.6, 134.6, 134.3, 130.1, 129.9, 128.0, 127.7, 126.9, 126.6, 126.3, 120.83, 120.78, 108.7, 107.7, 101.3, 39.7, 27.2; IR (neat) 2896, 1669, 1477, 1239, 1038, 773 cm^{-1}; mass spectrum (CI) *m/z* 369.1239 [$C_{23}H_{17}N_2O_3$ (M+1) requires 369.1239].



30

Rutaecarpine (30). *N,N*-diisopropylethylamine (34 mg, 46 µL, 0.27 mmol) was added to a suspension of **29** (50 mg, 0.24 mmol) in CH_2Cl_2 (2 mL) at room temperature. *o*-azidobenzoyl chloride (57 mg, 0.32 mmol) in CH_2Cl_2 (2 mL) was added, and the reaction was stirred for 48 h at room temperature. The reaction was partitioned between CH_2Cl_2 (15 mL) and saturated aqueous $NaHCO_3$ (15 mL). The layers were separated, and the aqueous layer was extracted with CH_2Cl_2 (2 × 15 mL). The combined organic layers were dried (Na_2SO_4), filtered, and concentrated under reduced pressure. The resultant yellow solid was purified by flash column chromatography eluting with CH_2Cl_2/EtOAc (99 : 1 → 95 : 5 → 9 : 1) to give 40 mg of **30** (58%) as a tan solid: mp 256-257 °C (Lit. = 256-257 °C);[9] ^{1}H NMR (400 MHz) δ 9.56 (br, 1 H), 8.33 (dd, *J* = 8.0, 1.2 Hz, 1 H), 7.70 (ddd, *J* = 8.4, 6.8, 1.6 Hz, 1 H), 7.66-7.62 (comp, 2 H), 7.42 (ddd, *J* = 8.0, 6.8, 1.2 Hz, 1 H), 7.36-7.28 (comp, 2 H), 7.17 (ddd, *J* = 7.6, 6.4, 1.2 Hz, 1 H), 4.60 (t, *J* = 7.2 Hz, 2 H), 3.24 (t, *J* = 7.2 Hz, 2 H); ^{13}C NMR (100 MHz) δ 161.6, 147.5, 145.0, 138.3, 134.4, 127.2, 127.1, 126.6, 126.2, 125.6, 125.5, 121.2, 120.6, 120.1, 118.4, 112.1, 41.1, 19.7; IR (neat) 3342, 1652, 1603, 1327, 729; mass spectrum (CI) *m/z* 288.1129 [$C_{18}H_{14}N_3O$ (M+1) requires 288.1137].

References

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4. Slougui, N.; Rousseau, G. *Synth. Commun.* **1987**, *17*, 1-11.
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600 MHz nmrox

BAG-01-136

exp1 Proton

SAMPLE		SPECIAL	
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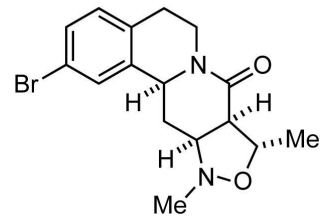
file	exp	spin	20
ACQUISITION		hst	0.008
sw	9615.4	pw90	11.600
at	4.000	alfa	10.000
np	76924	FLAGS	
fb	4000	il	n
bs	32	in	n
ss	4	dp	y
d1	2.000	hs	nn
nt	64	PROCESSING	
ct	64	lb	0.10
TRANSMITTER		fn	262144
tn	H1	DISPLAY	
sfrq	599.753	sp	-299.9
tof	599.7	wp	6297.3
tpwr	61	rfl	1199.9
pw	1.938	rfp	0
DECOUPLER		rp	140.1
dn	C13	lp	9.8
dof	0	PLOT	
dm	nnn	wc	250
dmm	c	sc	0
dpwr	37	vs	179
dmf	35088	th	12
		ai cdc ph	



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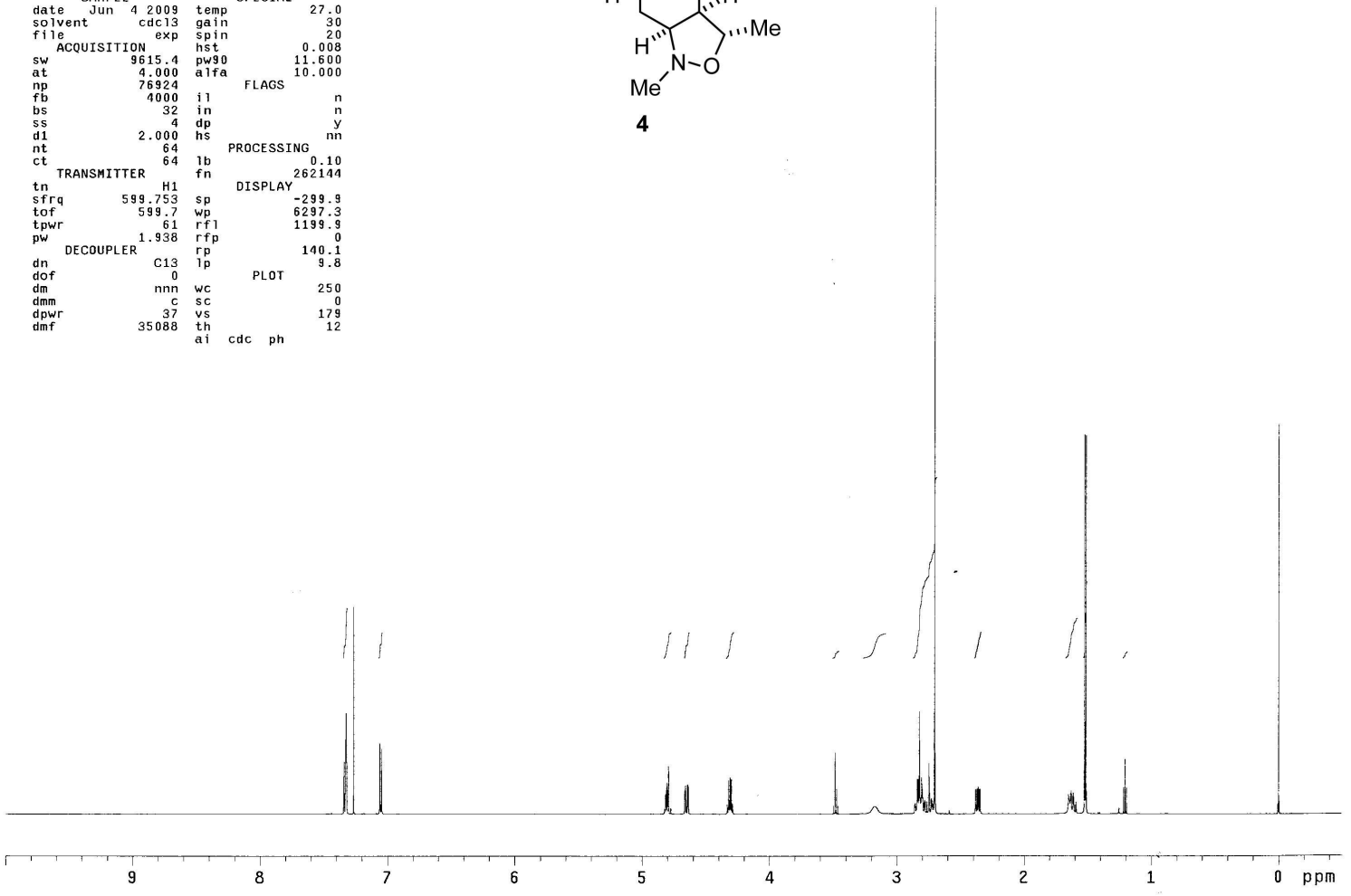

600 MHz nmrox

BAG-01-136

exp4 Carbon

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file	exp	spin	20
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sw	36764.7	pw90	7.800
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np	147058	FLAGS	
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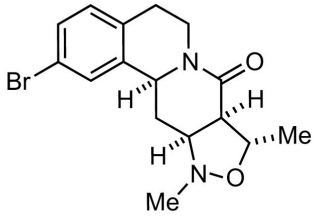
bs	64	in	n
d1	2.000	dp	y
nt	2000	hs	nn
ct	2000	PROCESSING	
TRANSMITTER		lb	0.50
tn	C13	fn	not used
sfrq	150.823	DISPLAY	
tof	1542.3	sp	-754.2
tpwr	58	wp	30915.3
pw	2.600	rfl	2541.8
DECOUPLER		rfp	0
dn	H1	rp	133.6
dof	0	lp	14.3
dm	yyy	PLOT	
dmm	w	wc	250
dpwr	46	sc	0
dmf	15337	vs	69144
		th	68
		ai	cdc ph



4

600 MHz nmrox

kk167

exp1 Proton

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solvent	cdcl3	gain	30
file	exp	spin	20
ACQUISITION		hst	0.008
sw	9615.4	pw90	11.600
at	4.000	alfa	10.000
np	76924	FLAGS	
fb	4000	il	n
bs	32	in	n
d1	2.000	dp	y
nt	64	hs	nn
ct	64	PROCESSING	
TRANSMITTER		lb	0.10
tn	H1	fn	262144
sfrq	599.753	DISPLAY	
tof	599.7	sp	-299.9
tpwr	61	wp	6297.3
pw	1.938	rfl	1200.4
DECOUPLER		rfp	0
dn	C13	rp	46.9
dof	0	lp	12.6
dm	nnn	PLOT	
dmm	c	wc	250
dpwr	37	sc	0
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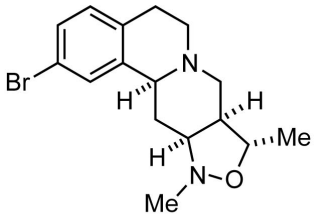
		th	12
		ai	cdc ph



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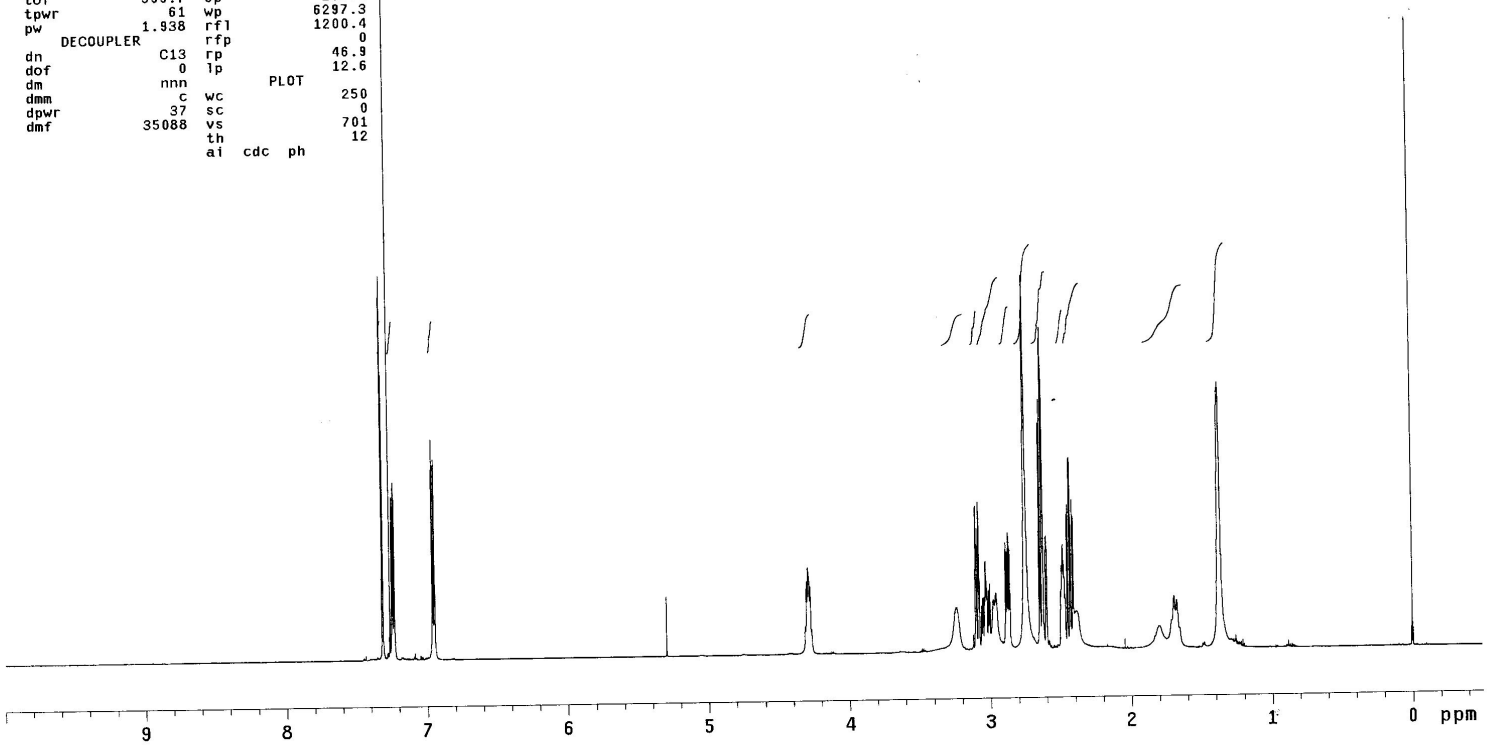

600 MHz nmrox

KK167

exp4 Carbon

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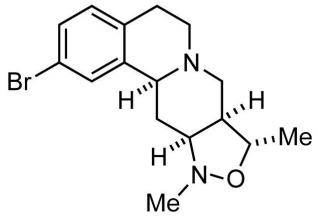
solvent	cdcl3	gain	40
file	exp	spin	20
ACQUISITION		hst	0.008
sw	40322.6	pw90	7.800
at	2.000	alfa	10.000
np	161290	FLAGS	
fb	17000	il	n
bs	64	in	n
d1	2.000	dp	y
nt	6000	hs	nn
ct	6000	PROCESSING	
TRANSMITTER		lb	0.50
tn	C13	fn	not used
sfrq	150.824	DISPLAY	
tof	2296.3	sp	-754.3
tpwr	58	wp	30915.4
pw	2.600	rfl	3568.6
DECOUPLER		rfp	0
dn	H1	rp	133.8
dof	0	lp	-0.0
dm	yyy	PLOT	
dmm	w	wc	250
dpwr	46	sc	0
dmf	15337	vs	26735
		th	6
		ai cdc ph	



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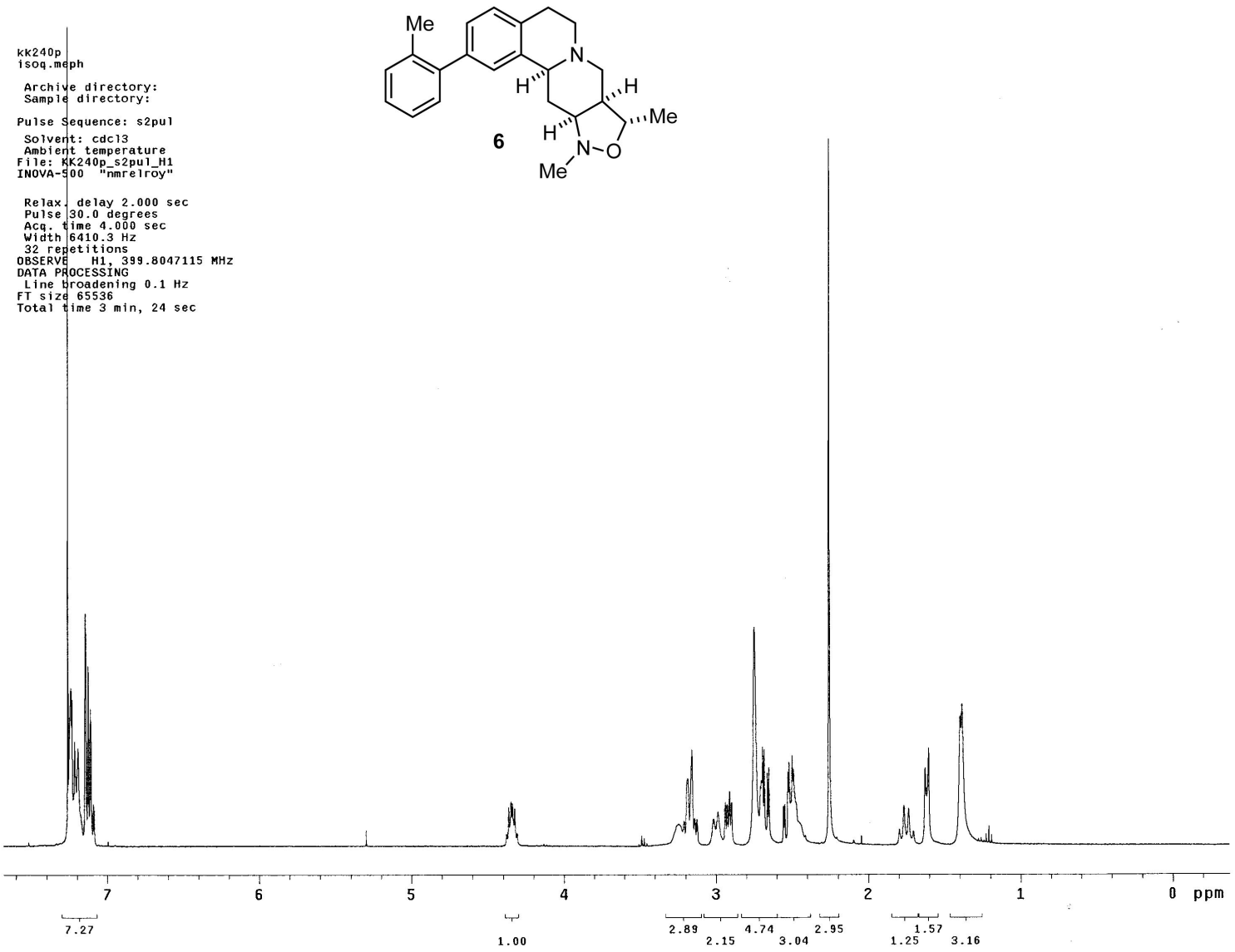
kk240p
isoq.meph
Archive directory:
Sample directory:
Pulse Sequence: s2pul
Solvent: cdcl3
Ambient temperature
File: KK240p_s2pul_H1
INOVA-500 "nmrelroy"
Relax. delay 2.000 sec
Pulse 30.0 degrees
Acq. time 4.000 sec
Width 6410.3 Hz
32 repetitions
OBSERVE H1, 399.8047115 MHz
DATA PROCESSING
Line broadening 0.1 Hz
FT size 65536
Total time 3 min, 24 sec
Me
N
H
H
H
Me
N
O
Me
6
7
6
5
4
3
2
1
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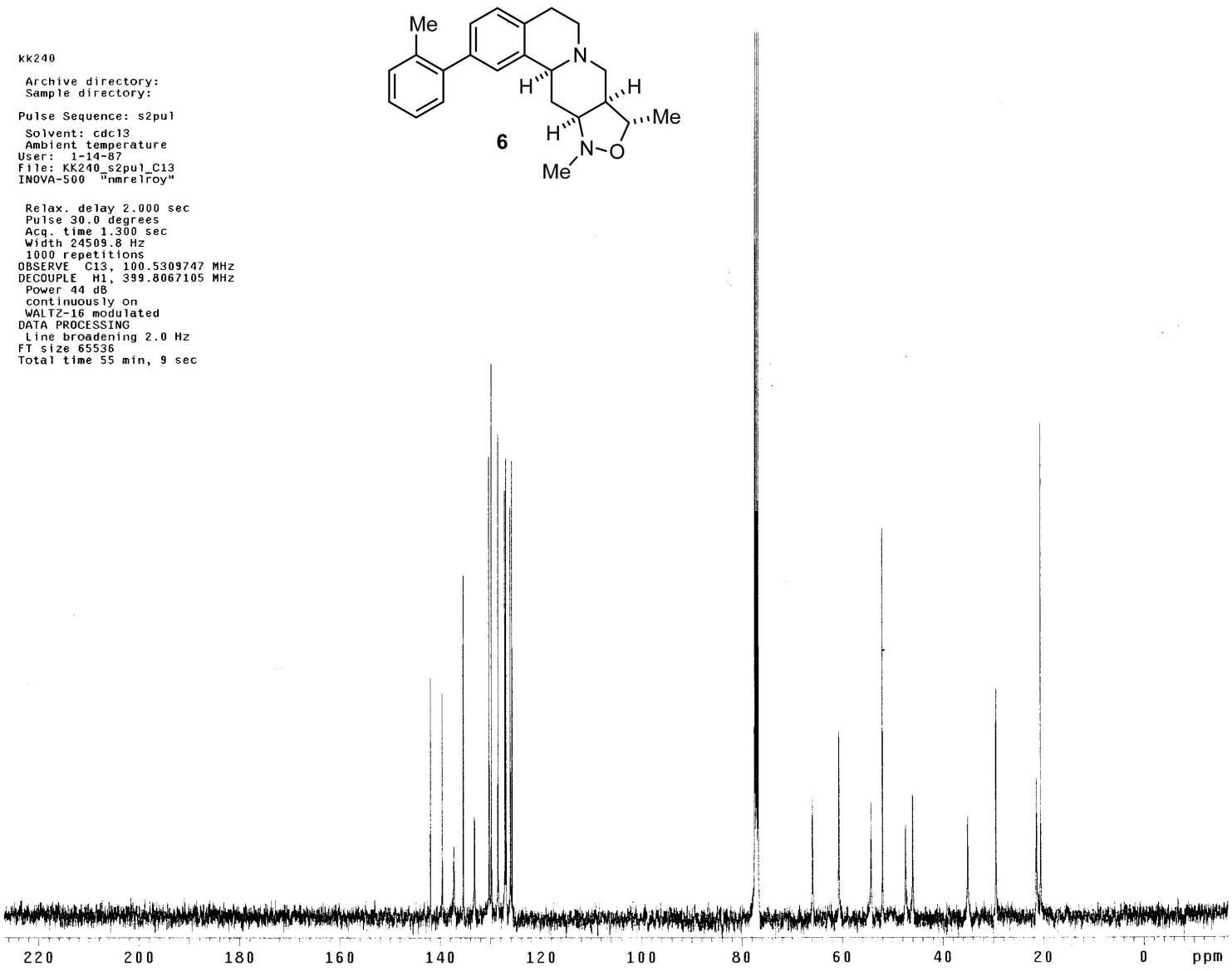
7.27
1.00
2.89
2.15
4.74
3.04
2.95
1.57
1.25
3.16


kk240
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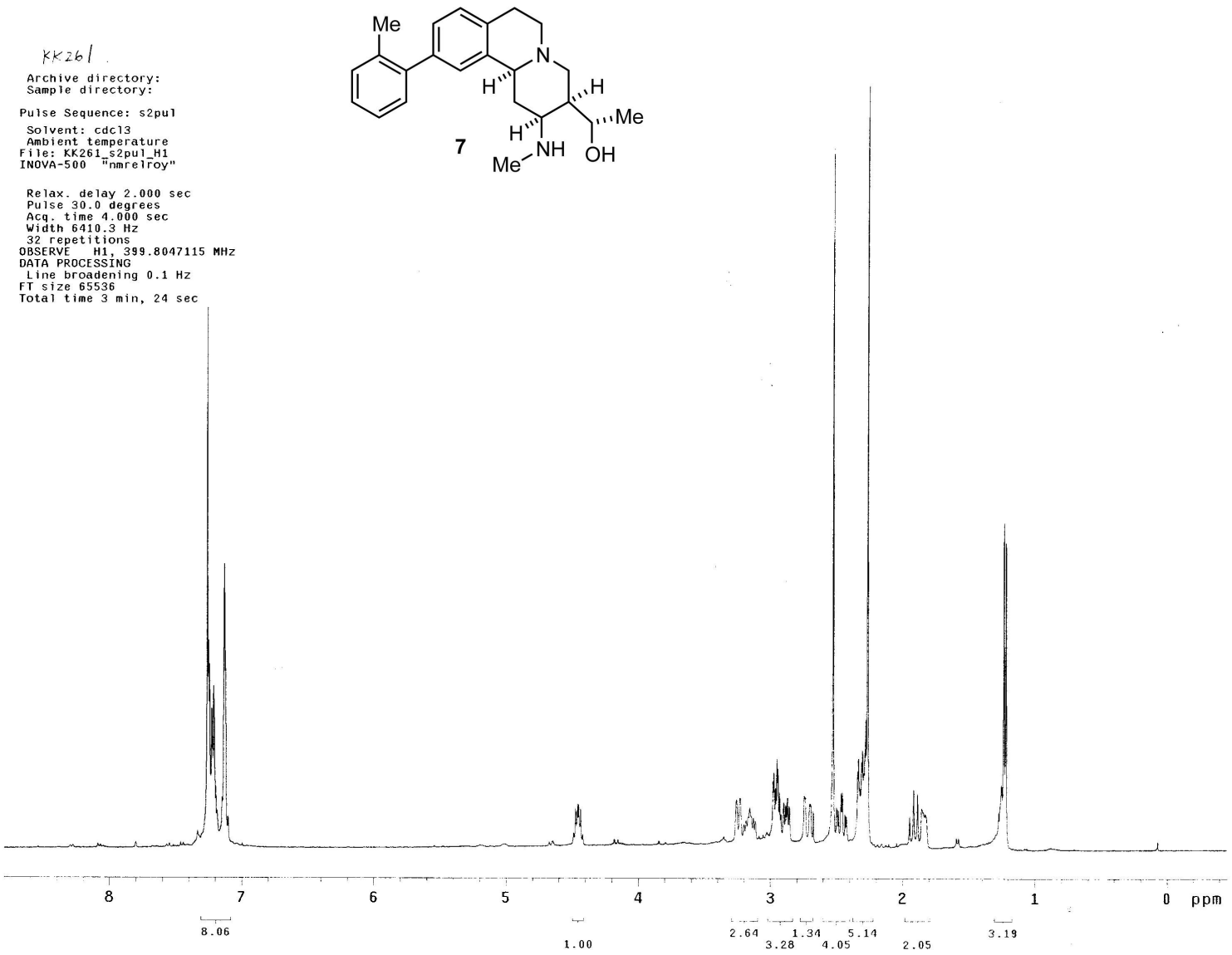
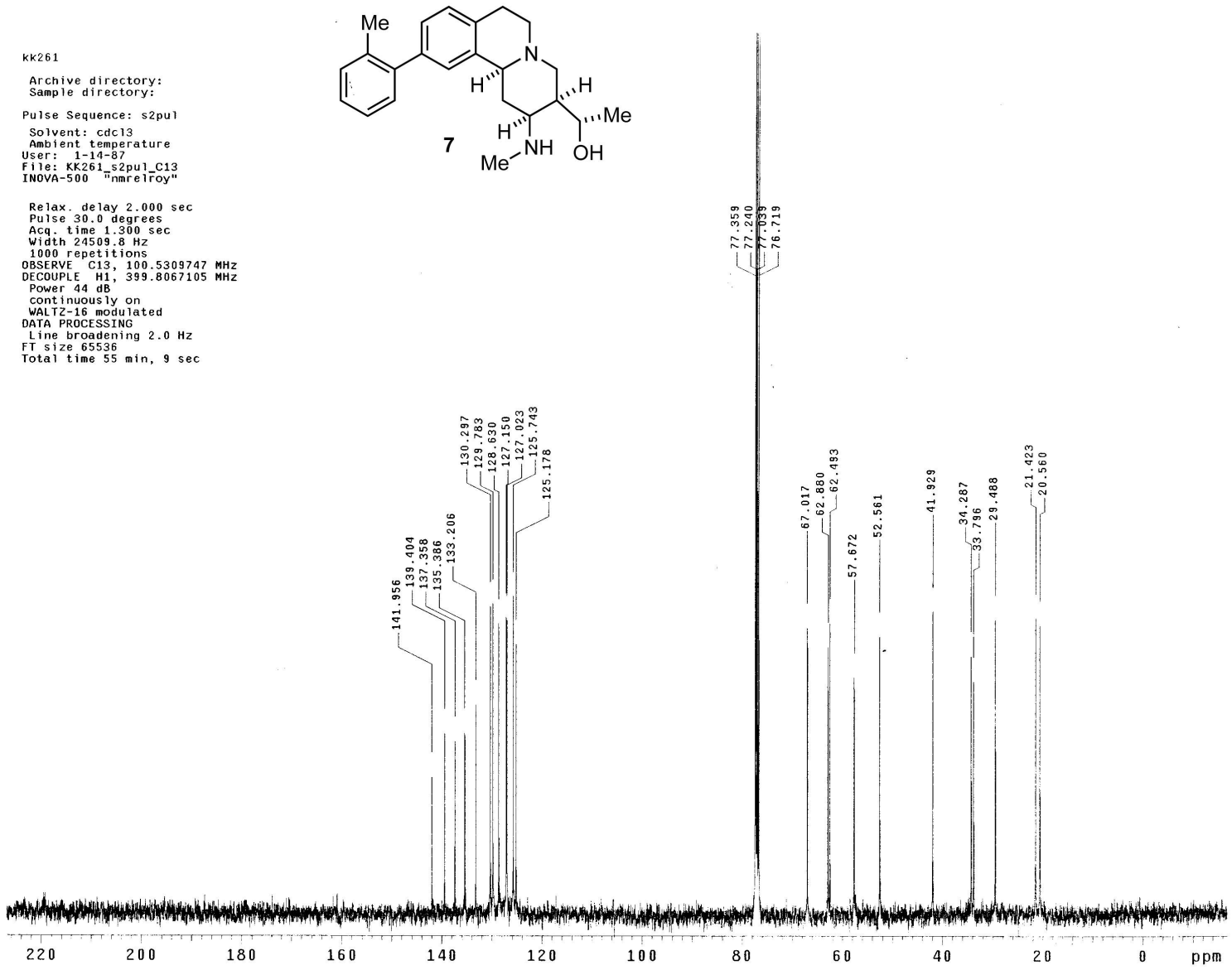
Sample directory:
Pulse Sequence: s2pul
Solvent: cdcl3
Ambient temperature
User: 1-14-87
File: KK240_s2pul_C13
INOVA-500 "nmrelroy"
Relax. delay 2.000 sec
Pulse 30.0 degrees
Acq. time 1.300 sec
Width 24509.8 Hz
1000 repetitions
OBSERVE C13, 100.5309747 MHz
DECOUPLE H1, 399.8067105 MHz
Power 44 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 2.0 Hz
FT size 65536
Total time 55 min, 9 sec
Me
N
H
H
H
Me
N
O
Me
6
220
200
180
160
140
120
100
80
60
40
20
0
ppm


KK261
Archive directory:
Sample directory:
Pulse Sequence: s2pul
Solvent: cdcl3
Ambient temperature
File: KK261_s2pul_H1
INOVA-500 "nmrelroy"
Relax. delay 2.000 sec
Pulse 30.0 degrees
Acq. time 4.000 sec
Width 6410.3 Hz
32 repetitions
OBSERVE H1, 399.8047115 MHz
DATA PROCESSING
Line broadening 0.1 Hz
FT size 65536
Total time 3 min, 24 sec
Me
N
H
H
Me
H
NH
Me
OH
7
8
7
6
5
4
3
2
1
0
ppm
8.06
1.00
2.64
3.28
1.34
4.05
5.14
2.05
3.19


kk261
Archive directory:
Sample directory:
Pulse Sequence: s2pul
Solvent: cdcl3
Ambient temperature
User: 1-14-87
File: KK261_s2pul_C13
INOVA-500 "nmrelroy"
Relax. delay 2.000 sec
Pulse 30.0 degrees
Acq. time 1.300 sec
Width 24509.8 Hz
1000 repetitions
OBSERVE C13, 100.5309747 MHz
DECOUPLE H1, 399.8067105 MHz
Power 44 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 2.0 Hz
FT size 65536
Total time 55 min, 9 sec
Me
N
H
H
H
Me
NH
OH
Me
7
77.359
77.240
77.039
76.719
141.956
139.404
137.358
135.386
133.206
130.297
129.783
128.630
127.150
127.023
125.743
125.178
67.017
62.880
62.493
57.672
52.561
41.929
34.287
33.796
29.488
21.423
20.560
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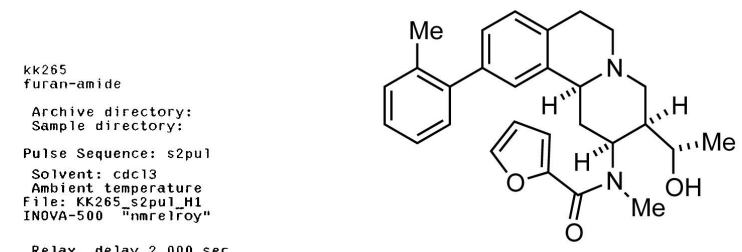
160
140
120
100
80
60
40
20
0
ppm



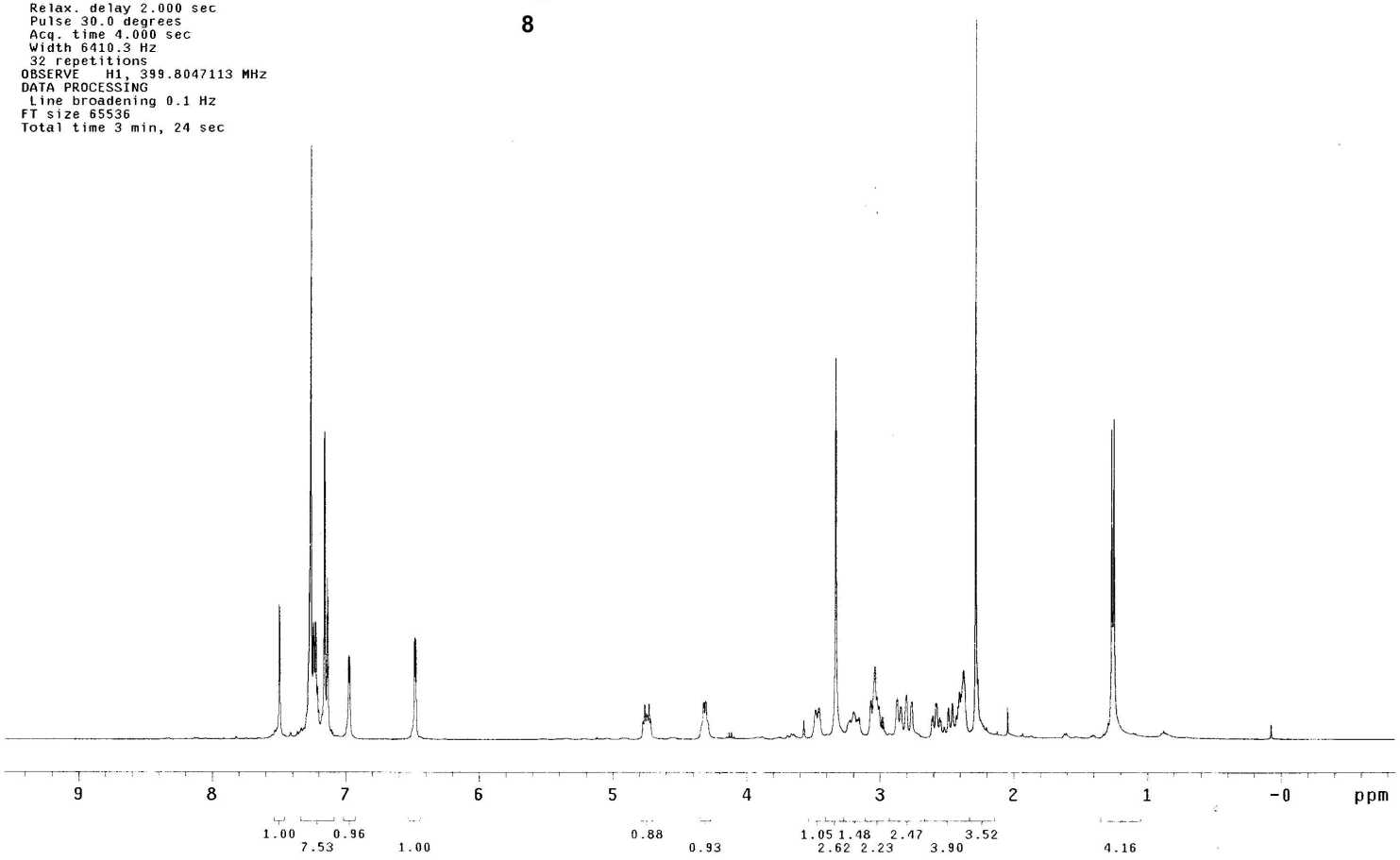
8

kk265
furan-amide

Archive directory:
Sample directory:

Pulse Sequence: s2pul
Solvent: cdcl3
Ambient temperature
File: KK265_s2pul_H1
INOVA-500 "nmrelroy"

Relax. delay 2.000 sec
Pulse 30.0 degrees
Acq. time 4.000 sec
Width 6410.3 Hz
32 repetitions
OBSERVE H1, 399.8047113 MHz
DATA PROCESSING
Line broadening 0.1 Hz
FT size 65536
Total time 3 min, 24 sec

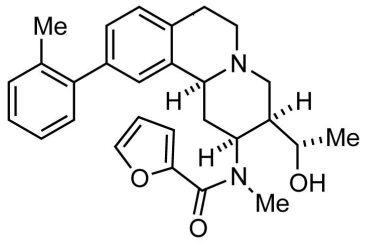


kk265
furan-amide

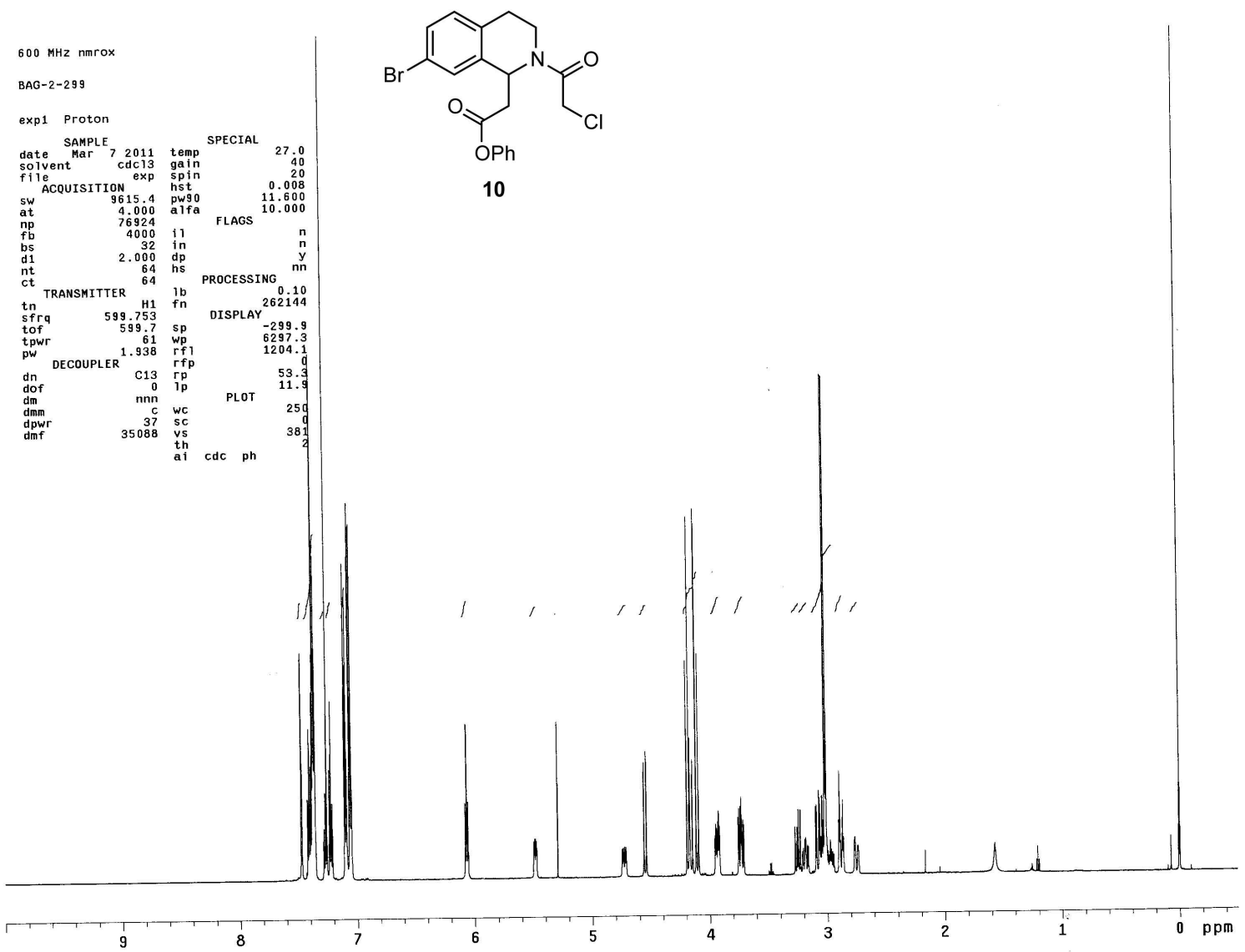
Archive directory:
Sample directory:

Pulse Sequence: s2pul
Solvent: cdcl3
Ambient temperature
User: 1-14-87
File: KK265_s2pul_C13
INOVA-500 "nmrelroy"

Relax. delay 2.000 sec
Pulse 30.0 degrees
Acq. time 1.300 sec
Width 24509.8 Hz
1000 repetitions
OBSERVE C13, 100.5309747 MHz
DECOUPLE H1, 399.8067105 MHz
Power 44 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 2.0 Hz
FT size 65536
Total time 55 min, 9 sec



8

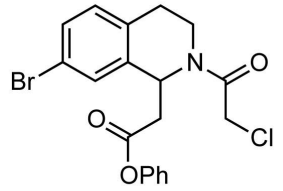

600 MHz nmrox
BAG-2-299
exp1 Proton
SAMPLE
date Mar 7 2011
solvent cdcl3
file exp
ACQUISITION
sw 9615.4
at 4.000
np 76924
fb 4000
bs 32
d1 2.000
nt 64
ct 64
TRANSMITTER
tn H1
sfrq 599.753
tof 599.7
tpwr 61
pw 1.938
DECOUPLER
dn C13
dof 0
dm nnn
dmm c
dpwr 37
dmf 35088
SPECIAL
temp 27.0
gain 40
spin 20
hst 0.008
pw90 11.600
alfa 10.000
FLAGS
il n
in n
dp y
hs nn
PROCESSING
lb 0.10
fn 262144
DISPLAY
sp -299.9
wp 6297.3
rfl 1204.1
rfp 0
rp 53.3
lp 11.9
PLOT
wc 250
sc 0
vs 381
th 2
ai cdc ph
Br
N
O
O
Cl
OPh
10
9
8
7
6
5
4
3
2
1
0 ppm

600 MHz nmrox

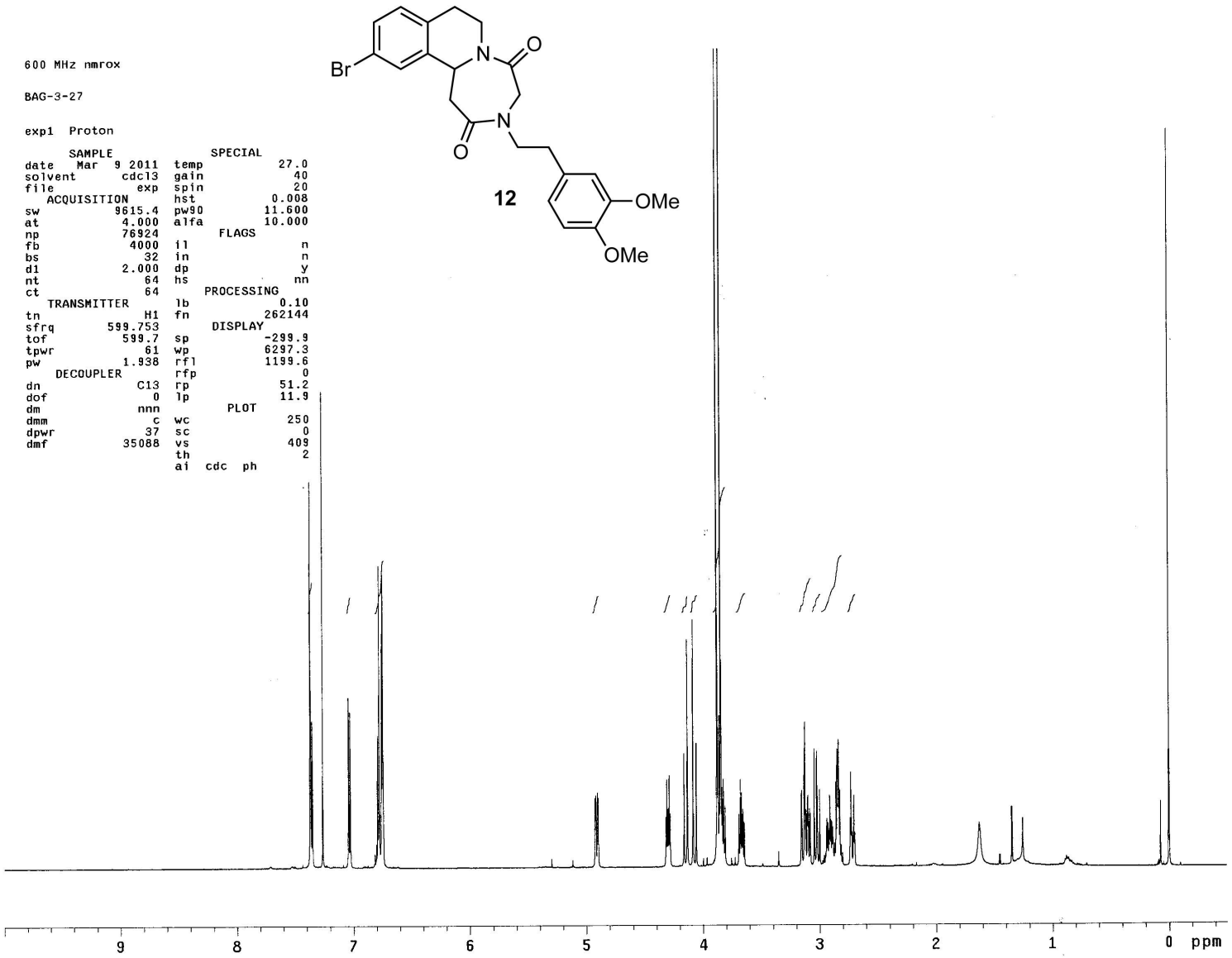
BAG-2-299

exp4 Carbon

SAMPLE		SPECIAL	
date	Mar 7 2011	temp	27.0
solvent	cdcl3	gain	40
file	exp	spin	20
ACQUISITION		hst	0.008
sw	40322.6	pw90	7.800
at	2.000	alfa	10.000
np	161290	**FLAGS**	
fb	17000	il	n
bs	64	in	n
d1	2.000	dp	y
nt	6000	hs	nn
ct	6000	**PROCESSING**	
TRANSMITTER		lb	0.50
tn	C13	fn	not used
sfrq	150.824	**DISPLAY**	
tof	2296.3	sp	-754.3
tpwr	58	wp	30915.4
pw	2.600	rfl	3567.4
DECOUPLER		rfp	0
dn	H1	rp	298.6
dof	0	lp	12.5
dm	yyy	**PLOT**	
dmm	w	wc	250
dpwr	46	sc	0
dmf	15337	vs	17675
		th	68
		ai	cdc ph



10


600 MHz nmrox
BAG-3-27
exp1 Proton
SAMPLE
date Mar 9 2011
solvent cdcl3
file exp
ACQUISITION
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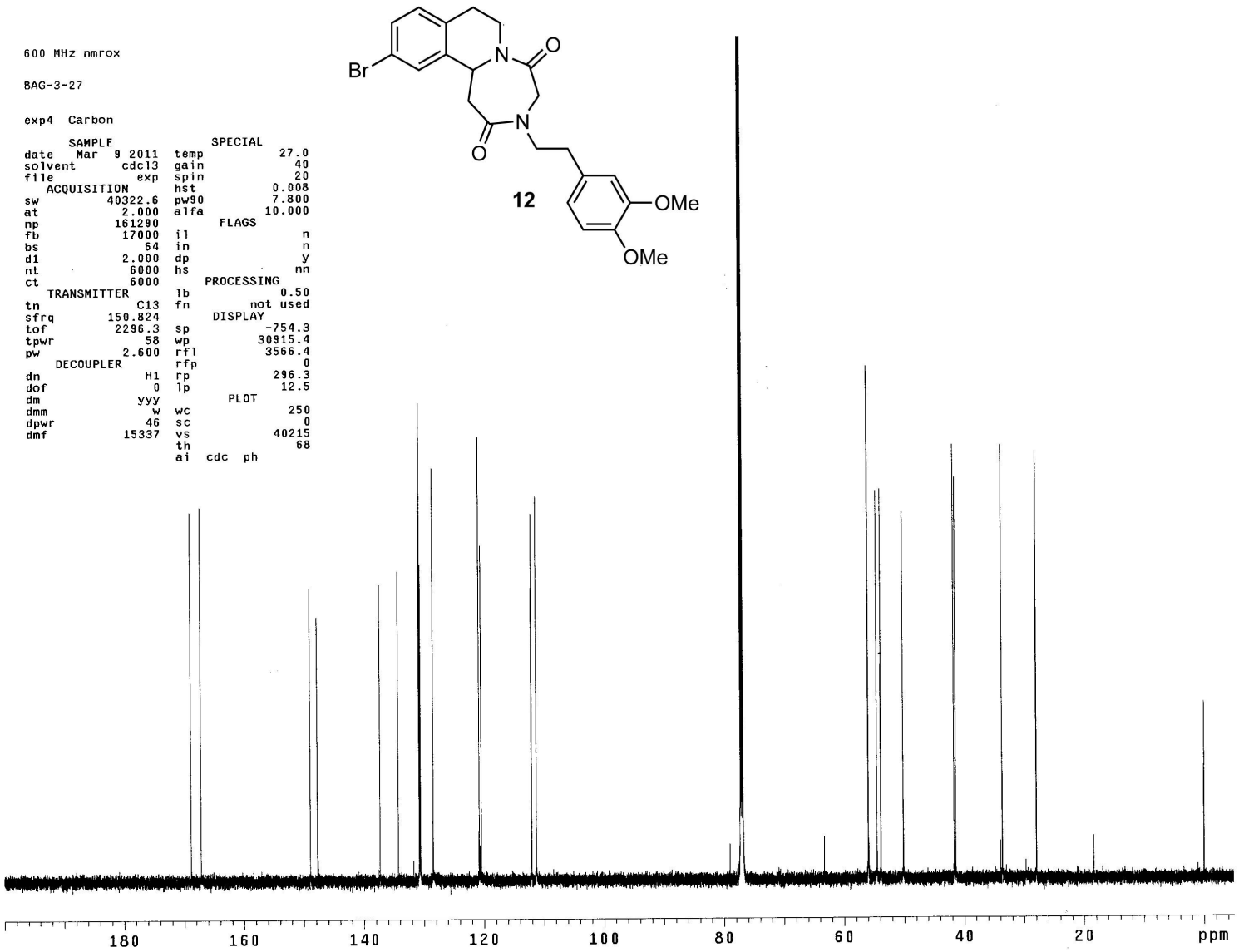
at 4.000
np 76924
fb 4000
bs 32
d1 2.000
nt 64
ct 64
TRANSMITTER
tn H1
sfrq 599.753
tof 599.7
tpwr 61
pw 1.938
DECOUPLER
dn C13
dof 0
dm nnn
dmm c
dpwr 37
dmf 35088
SPECIAL
temp 27.0
gain 40
spin 20
hst 0.008
pw90 11.600
alfa 10.000
FLAGS
il n
in n
dp y
hs nn
PROCESSING
lb 0.10
fn 262144
DISPLAY
sp -299.9
wp 6297.3
rfl 1199.6
rfp 0
rp 51.2
lp 11.9
PLOT
wc 250
sc 0
vs 409
th 2
ai cdc ph
Br
N
O
N
O
12
OMe
OMe
9
8
7
6
5
4
3
2
1
0 ppm


600 MHz nmrox
BAG-3-27
exp4 Carbon
SAMPLE
date Mar 9 2011
solvent cdcl3
file exp
ACQUISITION
sw 40322.6
at 2.000
np 161290
fb 17000
bs 64
d1 2.000
nt 6000
ct 6000
TRANSMITTER
tn C13
sfrq 150.824
tof 2296.3
tpwr 58
pw 2.600
DECOUPLER
dn H1
dof 0
dm yyy
dmm w
dpwr 46
dmf 15337
SPECIAL
temp 27.0
gain 40
spin 20
hst 0.008
pw90 7.800
alfa 10.000
FLAGS
il n
in n
dp y
hs nn
PROCESSING
lb 0.50
fn not used
DISPLAY
sp -754.3
wp 30915.4
rfl 3566.4
rfp 0
rp 296.3
lp 12.5
PLOT
wc 250
sc 0
vs 40215
th 68
ai cdc ph
Br
N
O
N
O
12
OMe
OMe
180
160
140
120
100
80
60
40
20
ppm

600 MHz nmrox

BAG-3-34

exp1 Proton

SAMPLE		SPECIAL	
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/vnmrsys/data/bag_~		hst	0.008
	3_34_h1.fid	pw90	11.600
ACQUISITION		alfa	10.000
sw	9615.4	FLAGS	
at	4.000	il	n
np	76924	in	n
fb	4000	dp	y
bs	32	hs	nn
d1	2.000	PROCESSING	
nt	64	lb	0.10
ct	64	fn	262144
TRANSMITTER		DISPLAY	
tn	H1	sp	-299.9
sfrq	599.753	wp	6297.3
tof	599.7	rfl	1200.2
tpwr	61	rfp	0
pw	1.938	rp	45.9
DECOUPLER		lp	11.0
dn	C13	PLOT	
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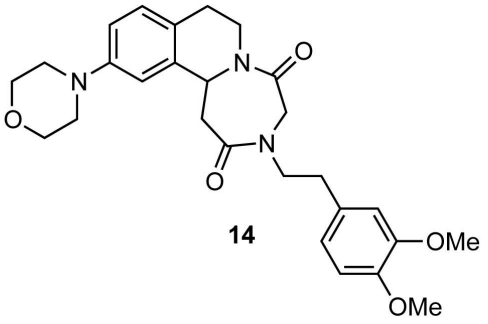
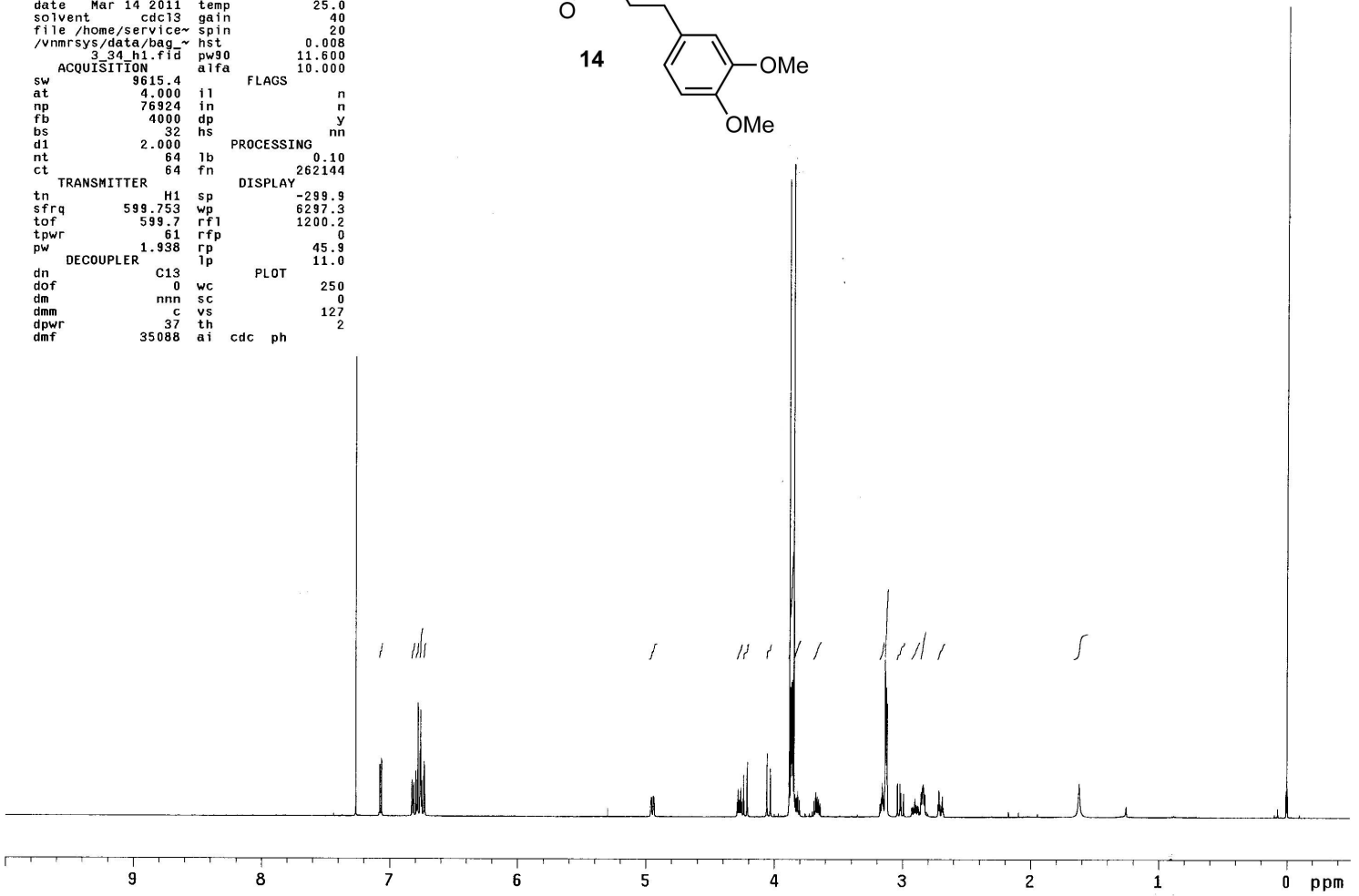
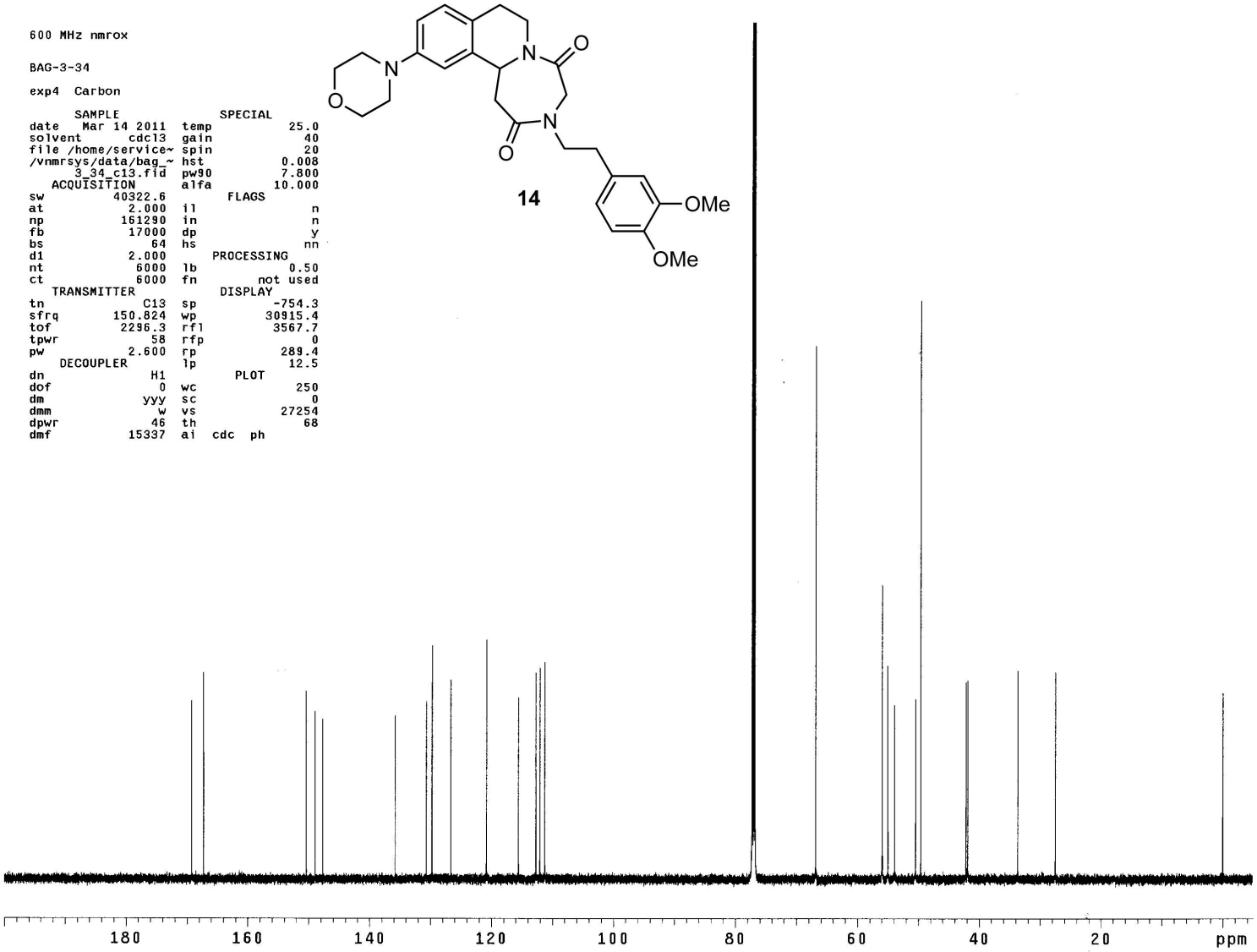
dpwr	37	th	2
dmf	35088	ai cdc ph	



14



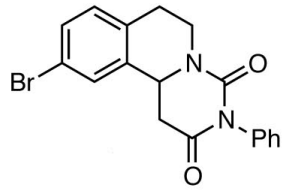

600 MHz nmrox
BAG-3-34
exp4 Carbon
SAMPLE
date Mar 14 2011
solvent cdcl3
file /home/service~
/vnmrsys/data/bag_~
3_34_c13.fid
ACQUISITION
sw 40322.6
at 2.000
np 161290
fb 17000
bs 64
d1 2.000
nt 6000
ct 6000
TRANSMITTER
tn C13
sfrq 150.824
tof 2296.3
tpwr 58
pw 2.600
DECOUPLER
dn H1
dof 0
dm yyy
dmm w
dpwr 46
dmf 15337
SPECIAL
temp 25.0
gain 40
spin 20
hst 0.008
pw90 7.800
alfa 10.000
FLAGS
il n
in n
dp y
hs nn
PROCESSING
lb 0.50
fn not used
DISPLAY
sp -754.3
wp 30915.4
rfl 3567.7
rfp 0
rp 289.4
lp 12.5
PLOT
wc 250
sc 0
vs 27254
th 68
ai cdc ph
14
N
O
N
O
N
O
OMe
OMe
180
160
140
120
100
80
60
40
20
ppm

500 MHz nmr0

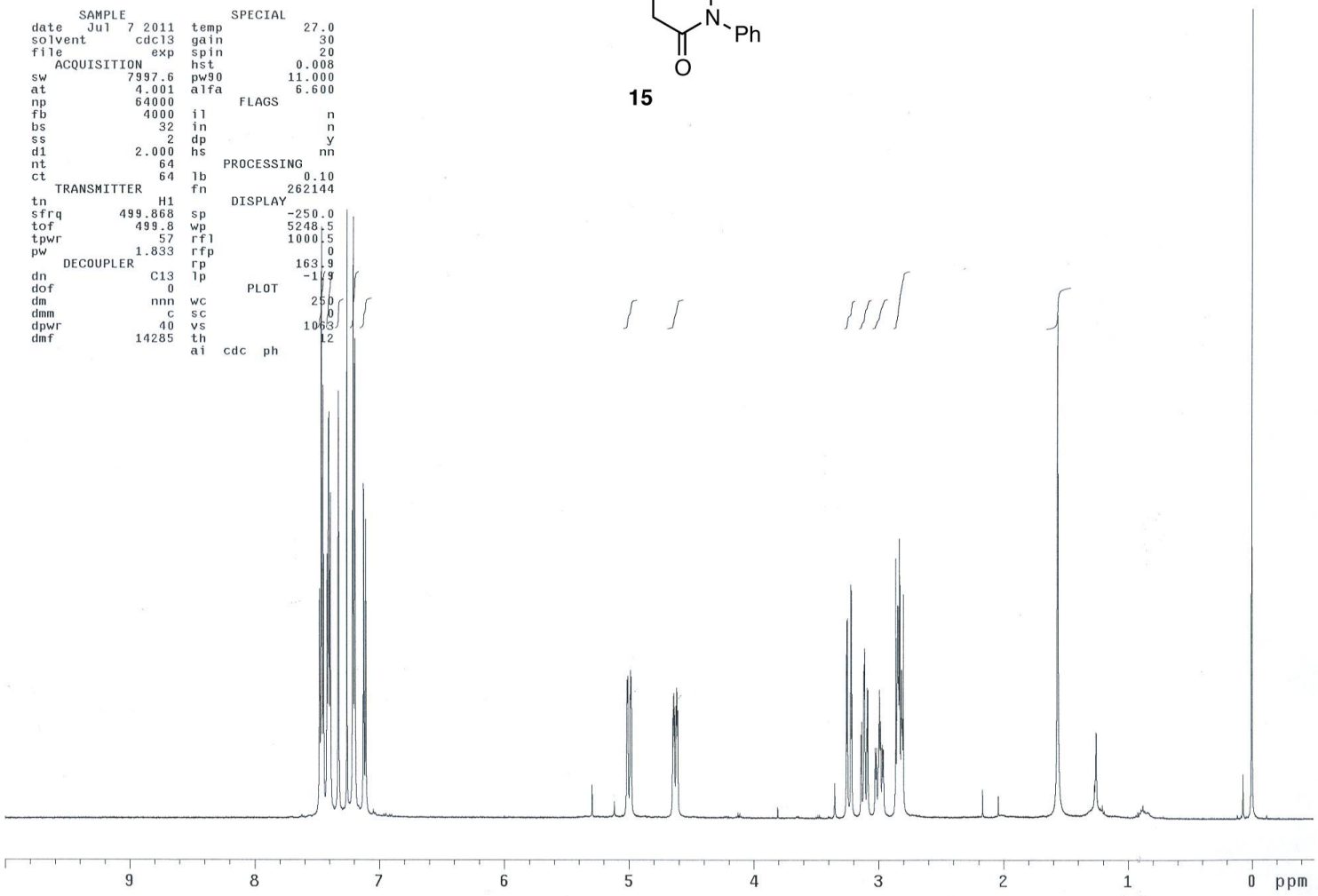
bag_3_116

exp1 Proton

SAMPLE		SPECIAL	
date	Jul 7 2011	temp	27.0
solvent	cdcl3	gain	30
file	exp	spin	20
ACQUISITION		hst	0.008
sw	7997.6	pw90	11.000
at	4.001	alfa	6.600
np	64000	FLAGS	
fb	4000	il	n
bs	32	in	n
ss	2	dp	y
d1	2.000	hs	nn
nt	64	PROCESSING	
ct	64	lb	0.10
TRANSMITTER		fn	262144
tn	H1	DISPLAY	
sfrq	499.868	sp	-250.0
tof	499.8	wp	5248.5
tpwr	57	rfl	1000.5
pw	1.833	rfp	0
DECOUPLER		rp	163.9
dn	C13	lp	-119
dof	0	PLOT	
dm	nnn	wc	250
dmm	c	sc	0
dpwr	40	vs	1068
dmf	14285	th	12
		ai cdc ph	



15

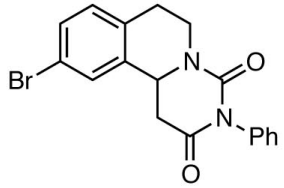


500 MHz nmr0

bag_3_116_c13

exp4 Carbon

SAMPLE		SPECIAL	
date	Jul 7 2011	temp	27.0
solvent	cdcl3	gain	50
file	exp	spin	20
ACQUISITION		hst	0.008
sw	30165.9	pw90	9.500
at	1.958	alfa	10.000
np	118154	FLAGS	
fb	17000	il	n
bs	16	in	n
d1	2.000	dp	y
nt	13000	hs	nn
ct	13000	PROCESSING	
TRANSMITTER		lb	1.00
tn	C13	fn	not used
sfrq	125.704	DISPLAY	
tof	1255.4	sp	-628.7
tpwr	53	wp	25766.4
pw	1.583	rfl	11593.9
DECOUPLER		rfp	9678.2
dn	H1	rp	-125.4
dof	0	lp	-193.1
dm	yyy	PLOT	
dmm	w	wc	250
dpwr	37	sc	0
dmf	10582	vs	15603
		th	16
		ai	cdc ph

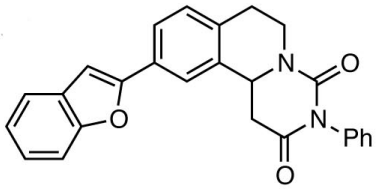


15

BAG-3-140

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
 64 repetitions
OBSERVE H1, 400.2669784 MHz
DATA PROCESSING
 Line broadening 0.1 Hz
FT size 32768
Total time 5 min, 21 sec

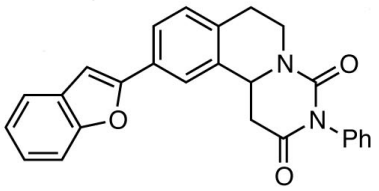


16

BAG-3-140

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
 10000 repetitions
OBSERVE C13, 100.6472130 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 9 hr, 53 min, 41 sec



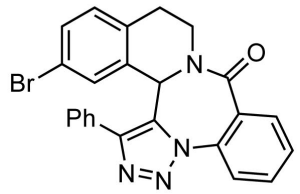
16

500 MHz nmr0

BAG-2-141

exp1 Proton

SAMPLE		SPECIAL	
date	Sep 24 2010	temp	27.0
solvent	cdcl3	gain	30
file	exp	spin	20
ACQUISITION		hst	0.008
sw	7997.6	pw90	11.000
at	4.001	alfa	6.600
np	64000	**FLAGS**	
fb	4000	il	n
bs	32	in	n
d1	2.000	dp	y
nt	64	hs	nn
ct	64	**PROCESSING**	
TRANSMITTER		lb	0.10
tn	H1	fn	262144
sfrq	499.868	**DISPLAY**	
tof	499.7	sp	-250.0
tpwr	57	wp	5248.5
pw	1.833	rfl	996.3
DECOUPLER		rfp	0
dn	C13	rp	173.2
dof	0	lp	-5.2
dm	nnn	**PLOT**	
dmm	c	wc	250
dpwr	40	sc	0
dmf	14285	vs	535
		th	12
		ai	cdc ph



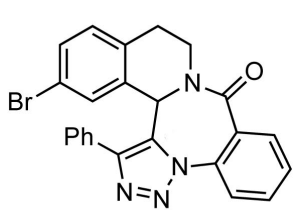
17

500 MHz nmr0

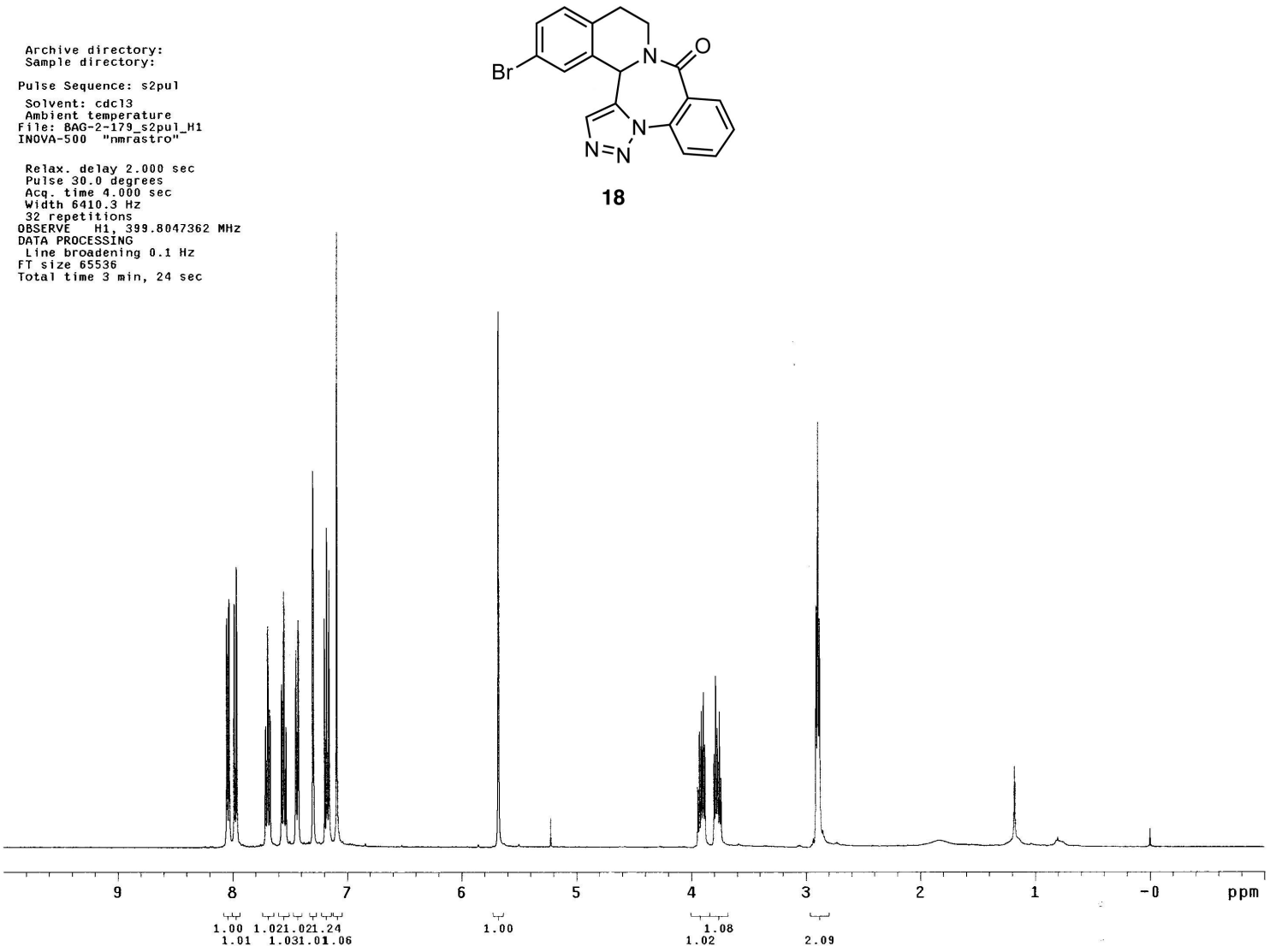
BAG-2-141

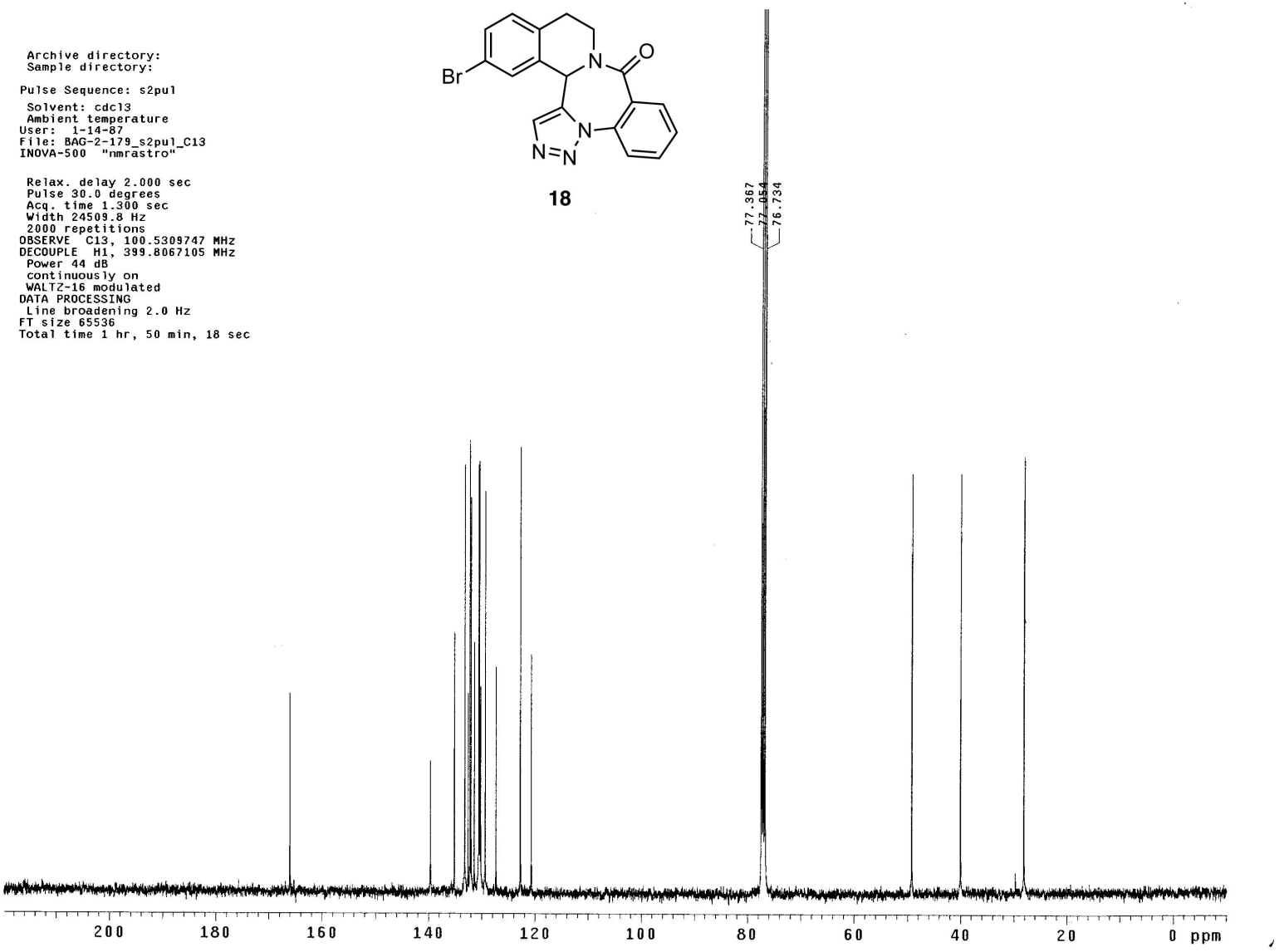
exp4 Carbon

SAMPLE		SPECIAL	
date	Sep 24 2010	temp	27.0
solvent	cdcl3	gain	40
file	exp	spin	20
ACQUISITION		hst	0.008
sw	32679.7	pw90	9.500
at	1.958	alfa	10.000
np	128000	FLAGS	
fb	18000	il	n
bs	16	in	n
d1	2.000	dp	y
nt	8000	hs	nn
ct	0	PROCESSING	
TRANSMITTER		lb	1.00
tn	C13	fn	not used
sfrq	125.705	DISPLAY	
tof	1883.9	sp	-628.8
tpwr	53	wp	25766.4
pw	3.163	rfl	2540.1
DECOUPLER		rfp	0
dn	H1	rp	-103.9
dof	0	lp	-221.5
dm	yyy	PLOT	
dmm	w	wc	250
dpwr	37	sc	0
dmf	10582	vs	16884
		th	5
		ai cdc ph	



17


Archive directory:
Sample directory:
Pulse Sequence: s2pul
Solvent: cdcl3
Ambient temperature
File: BAG-2-179_s2pul_H1
INOVA-500 "nmrastro"
Relax. delay 2.000 sec
Pulse 30.0 degrees
Acq. time 4.000 sec
Width 6410.3 Hz
32 repetitions
OBSERVE H1, 399.8047362 MHz
DATA PROCESSING
Line broadening 0.1 Hz
FT size 65536
Total time 3 min, 24 sec
Br
N
O
N
N=N
18
9
8
7
6
5
4
3
2
1
-0
ppm
1.00
1.01
1.02
1.03
1.02
1.01
1.24
1.06
1.00
1.08
1.02
2.09

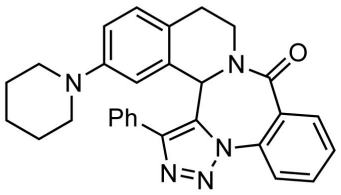

Archive directory:
Sample directory:
Pulse Sequence: s2pul
Solvent: cdcl3
Ambient temperature
User: 1-14-87
File: BAG-2-179_s2pul_C13
INOVA-500 "nmrastro"
Relax. delay 2.000 sec
Pulse 30.0 degrees
Acq. time 1.300 sec
Width 24509.8 Hz
2000 repetitions
OBSERVE C13, 100.5309747 MHz
DECOUPLE H1, 399.8067105 MHz
Power 44 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 2.0 Hz
FT size 65536
Total time 1 hr, 50 min, 18 sec
Br
N
O
N
N=N
18
77.367
77.054
76.734
200
180
160
140
120
100
80
60
40
20
0 ppm

500 MHz nmr0

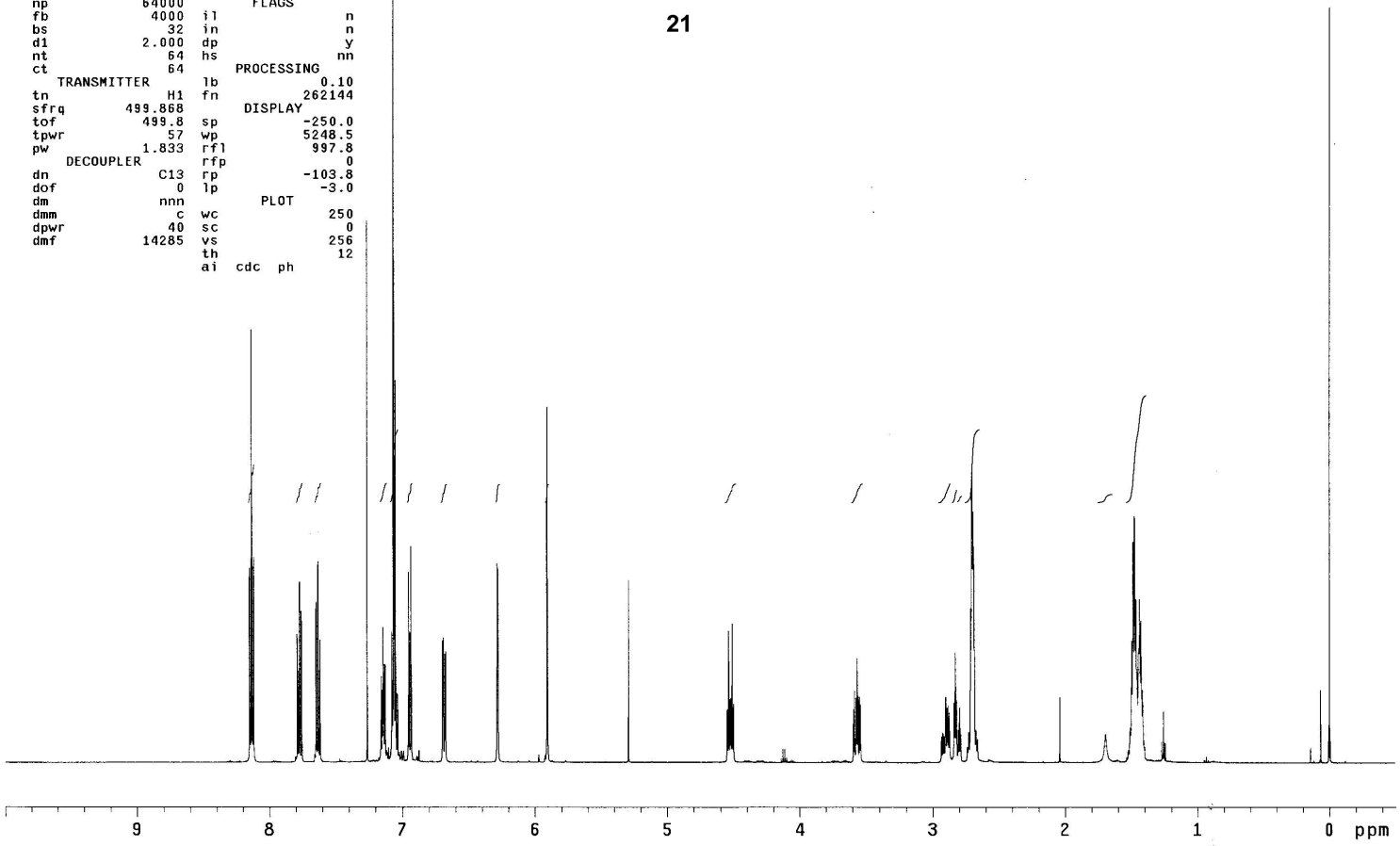
BAG-2-270

exp1 Proton

SAMPLE		SPECIAL	
date	Feb 16 2011	temp	27.0
solvent	CDCl3	gain	30
file	exp	spin	20
ACQUISITION		hst	0.008
sw	7997.6	pw90	11.000
at	4.001	alfa	6.600
np	64000	FLAGS	
fb	4000	il	n
bs	32	in	n
d1	2.000	dp	y
nt	64	hs	nn
ct	64	PROCESSING	
TRANSMITTER		lb	0.10
tn	H1	fn	262144
sfrq	499.868	DISPLAY	
tof	499.8	sp	-250.0
tpwr	57	wp	5248.5
pw	1.833	rfl	997.8
DECOUPLER		rfp	0
dn	C13	rp	-103.8
dof	0	lp	-3.0
dm	nnn	PLOT	
dmm	c	wc	250
dpwr	40	sc	0
dmf	14285	vs	256
		th	12
		ai cdc ph	



21

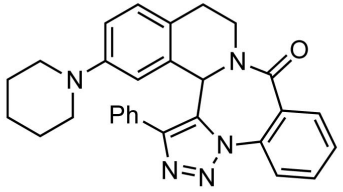


500 MHz nmr0

BAG-2-270

exp4 Carbon

SAMPLE		SPECIAL	
date	Feb 16 2011	temp	27.0
solvent	cdcl3	gain	40
file	exp	spin	20
ACQUISITION		hst	0.008
sw	32679.7	pw90	9.500
at	1.958	alfa	10.000
np	128000	FLAGS	
fb	18000	il	n
bs	16	in	n
d1	2.000	dp	y
nt	2000	hs	nn
ct	2000	PROCESSING	
TRANSMITTER		lb	1.00
tn	C13	fn	not used
sfrq	125.705	DISPLAY	
tof	1883.9	sp	-628.8
tpwr	53	wp	25766.4
pw	3.163	rfl	2541.1
DECOUPLER		rfp	0
dn	H1	rp	-18.2
dof	0	lp	-210.7
dm	yyy	PLOT	
dmm	w	wc	250
dpwr	37	sc	0
dmf	10582	vs	10023
		th	5
		ai	cdc ph



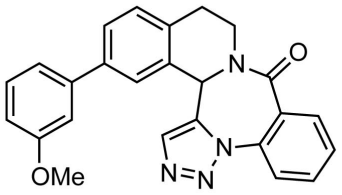
21

500 MHz nmr0

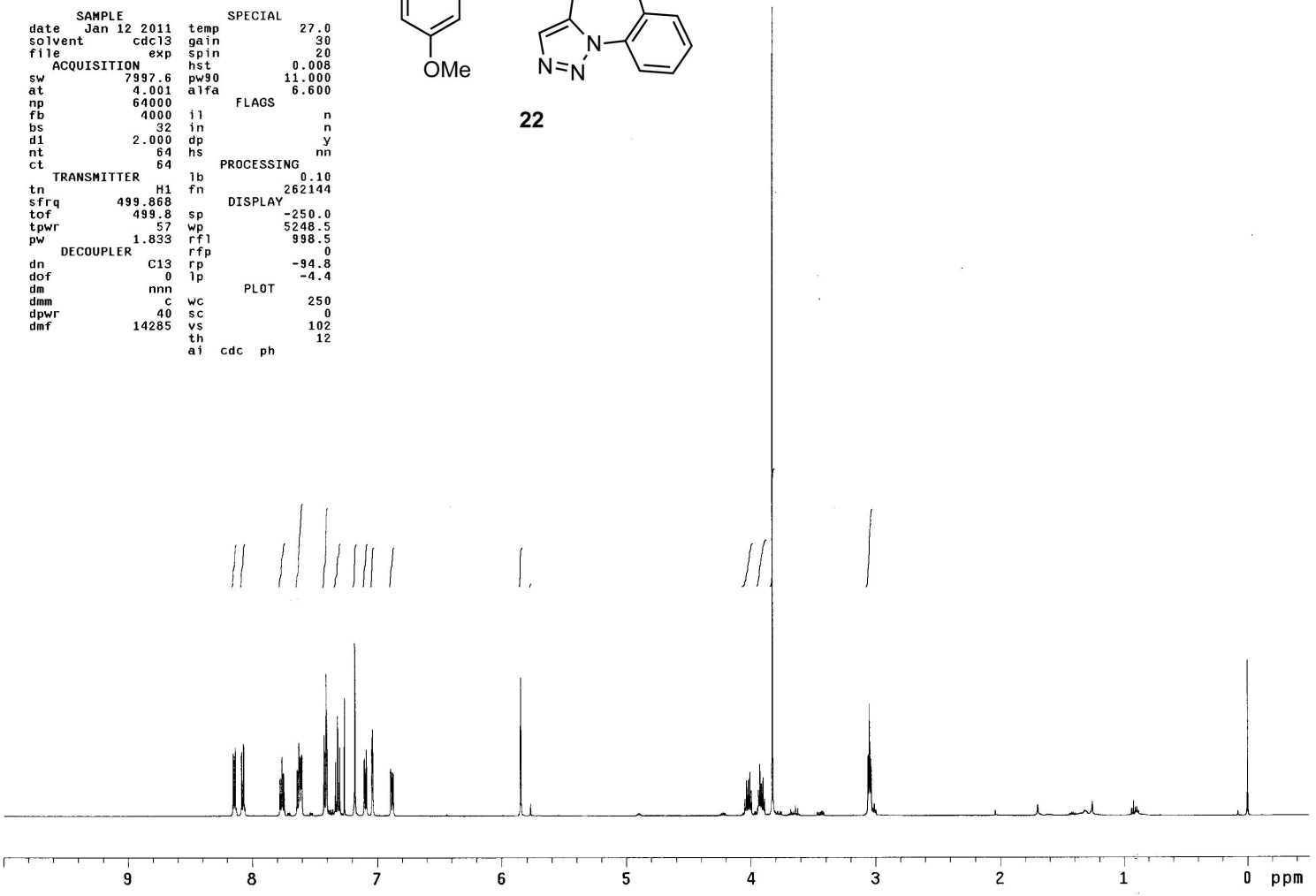
BAG-2-245

exp1 Proton

SAMPLE		SPECIAL	
date	Jan 12 2011	temp	27.0
solvent	cdcl3	gain	30
file	exp	spin	20
ACQUISITION		hst	0.008
sw	7997.6	pw90	11.000
at	4.001	alfa	6.600
np	64000	FLAGS	
fb	4000	il	n
bs	32	in	n
d1	2.000	dp	y
nt	64	hs	nn
ct	64	PROCESSING	
TRANSMITTER		lb	0.10
tn	H1	fn	262144
sfrq	499.868	DISPLAY	
tof	499.8	sp	-250.0
tpwr	57	wp	5248.5
pw	1.833	rfl	998.5
DECOUPLER		rfp	0
dn	C13	rp	-94.8
dof	0	lp	-4.4
dm	nnn	PLOT	
dmm	c	wc	250
dpwr	40	sc	0
dmf	14285	vs	102
		th	12
		ai cdc ph	



22

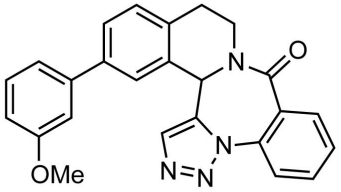


500 MHz nmr0

BAG-2-245

exp4 Carbon

SAMPLE		SPECIAL	
date	Jan 12 2011	temp	27.0
solvent	cdcl3	gain	40
file	exp	spin	20
ACQUISITION		hst	0.008
sw	32679.7	pw90	9.500
at	1.958	alfa	10.000
np	128000	FLAGS	
fb	18000	il	n
bs	16	in	n
d1	2.000	dp	y
nt	4000	hs	nn
ct	4000	PROCESSING	
TRANSMITTER		lb	1.00
tn	C13	fn	not used
sfrq	125.705	DISPLAY	
tof	1883.9	sp	-628.8
tpwr	53	wp	25766.4
pw	3.163	rfl	2541.6
DECOUPLER		rfp	0
dn	H1	rp	-15.9
dof	0	lp	-225.9
dm	yyy	PLOT	
dmm	w	wc	250
dpwr	37	sc	0
dmf	10582	vs	14679
		th	5
		ai	cdc ph



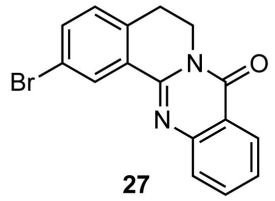
22

500 MHz nmr0

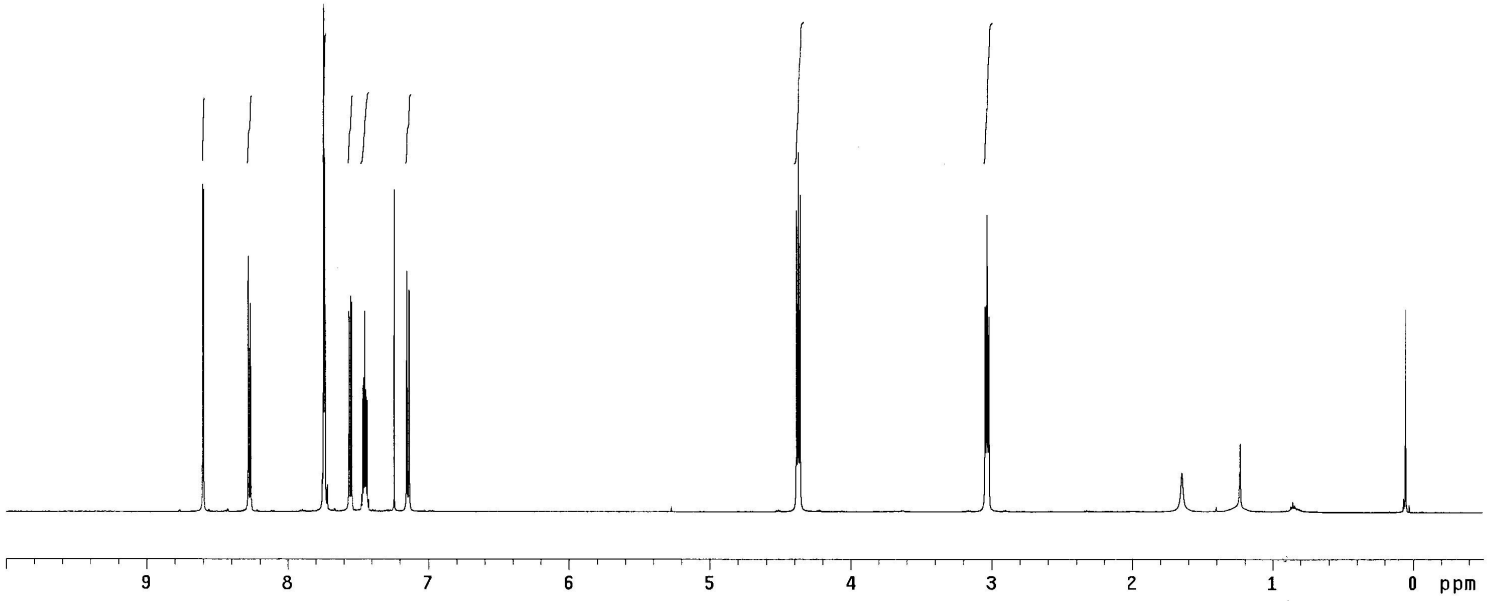
BAG-2-123

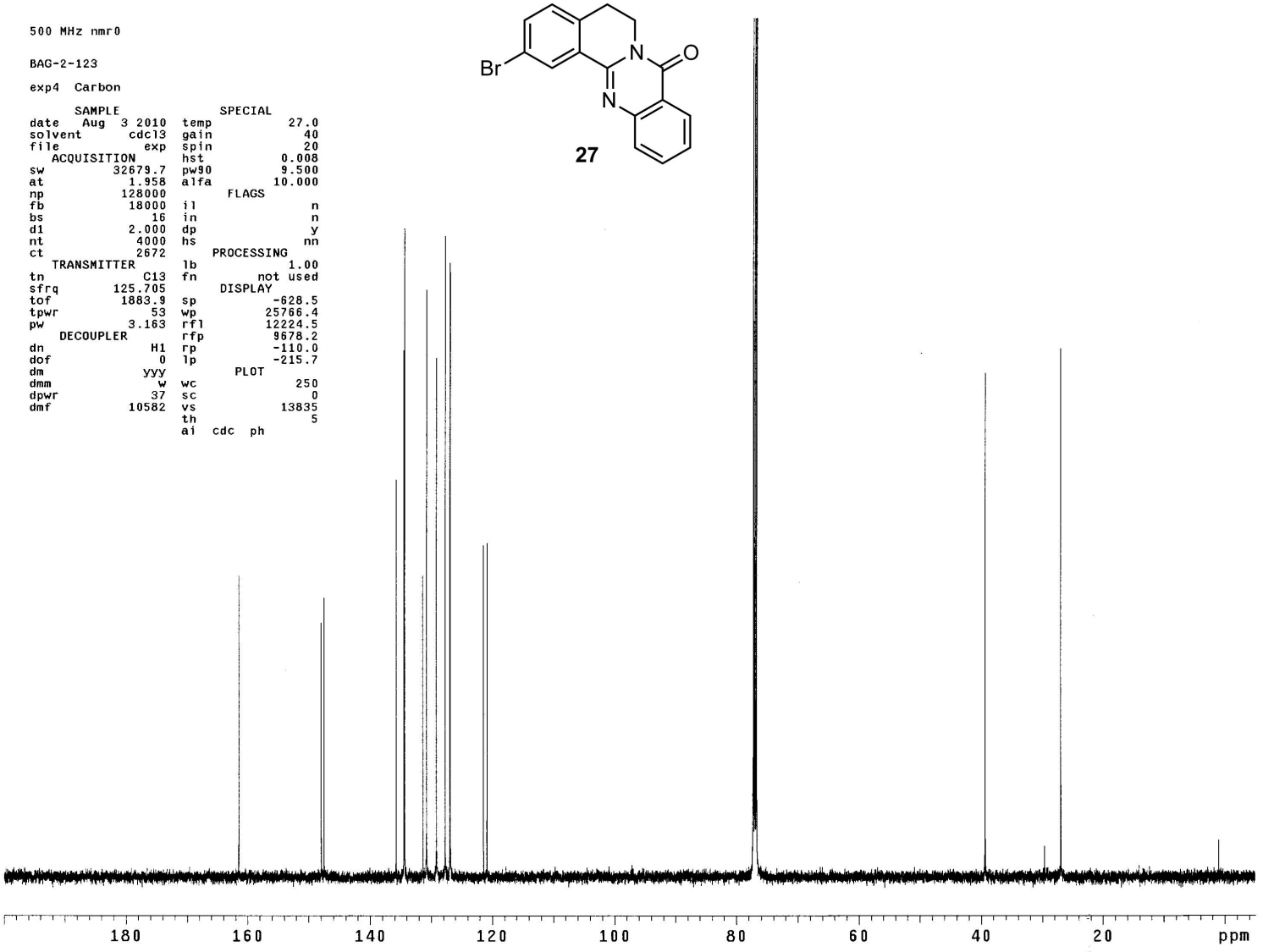
exp1 Proton

SAMPLE		SPECIAL	
date	Aug 3 2010	temp	27.0
solvent	cdcl3	gain	30
file	exp	spin	20
ACQUISITION		hst	0.008
sw	7997.6	pw90	11.000
at	4.001	alfa	6.600
np	64000	FLAGS	
fb	4000	il	n
bs	32	in	n
d1	2.000	dp	y
nt	64	hs	nn
ct	64	PROCESSING	
TRANSMITTER		lb	0.10
tn	H1	fn	262144
sfrq	499.868	DISPLAY	
tof	499.7	sp	-249.9
tpwr	57	wp	5248.5
pw	1.833	rfl	4628.5
DECOUPLER		rfp	3619.0
dn	C13	rp	173.0
dof	0	lp	-6.3
dm	nnn	PLOT	
dmm	c	wc	250
dpwr	40	sc	0
dmf	14285	vs	202
		th	12
		ai cdc ph	



27



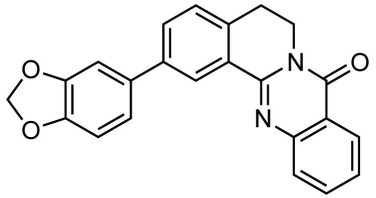

500 MHz nmr0
BAG-2-123
exp4 Carbon
SAMPLE
date Aug 3 2010
solvent cdcl3
file exp
ACQUISITION
sw 32679.7
at 1.958
np 128000
fb 18000
bs 16
d1 2.000
nt 4000
ct 2672
TRANSMITTER
tn C13
sfrq 125.705
tof 1883.9
tpwr 53
pw 3.163
DECOUPLER
dn H1
dof 0
dm yyy
dmm w
dpwr 37
dmf 10582
SPECIAL
temp 27.0
gain 40
spin 20
hst 0.008
pw90 9.500
alfa 10.000
FLAGS
il n
in n
dp y
hs nn
PROCESSING
lb 1.00
fn not used
DISPLAY
sp -628.5
wp 25766.4
rfl 12224.5
rfp 9678.2
lp -215.7
PLOT
wc 250
sc 0
vs 13835
th 5
ai cdc ph
Br
N
O
N
27
180
160
140
120
100
80
60
40
20
ppm

500 MHz nmr0

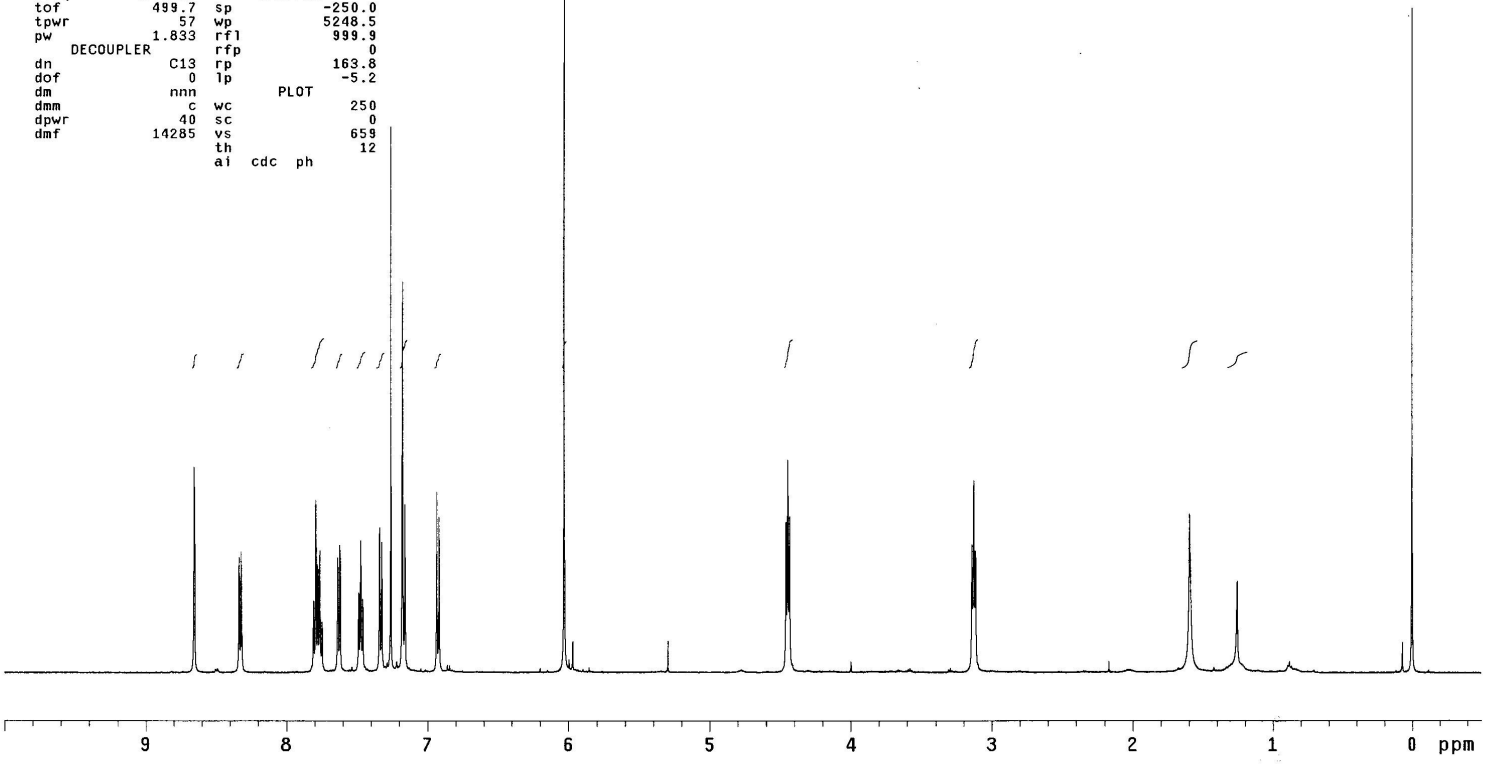
BAG-2-152

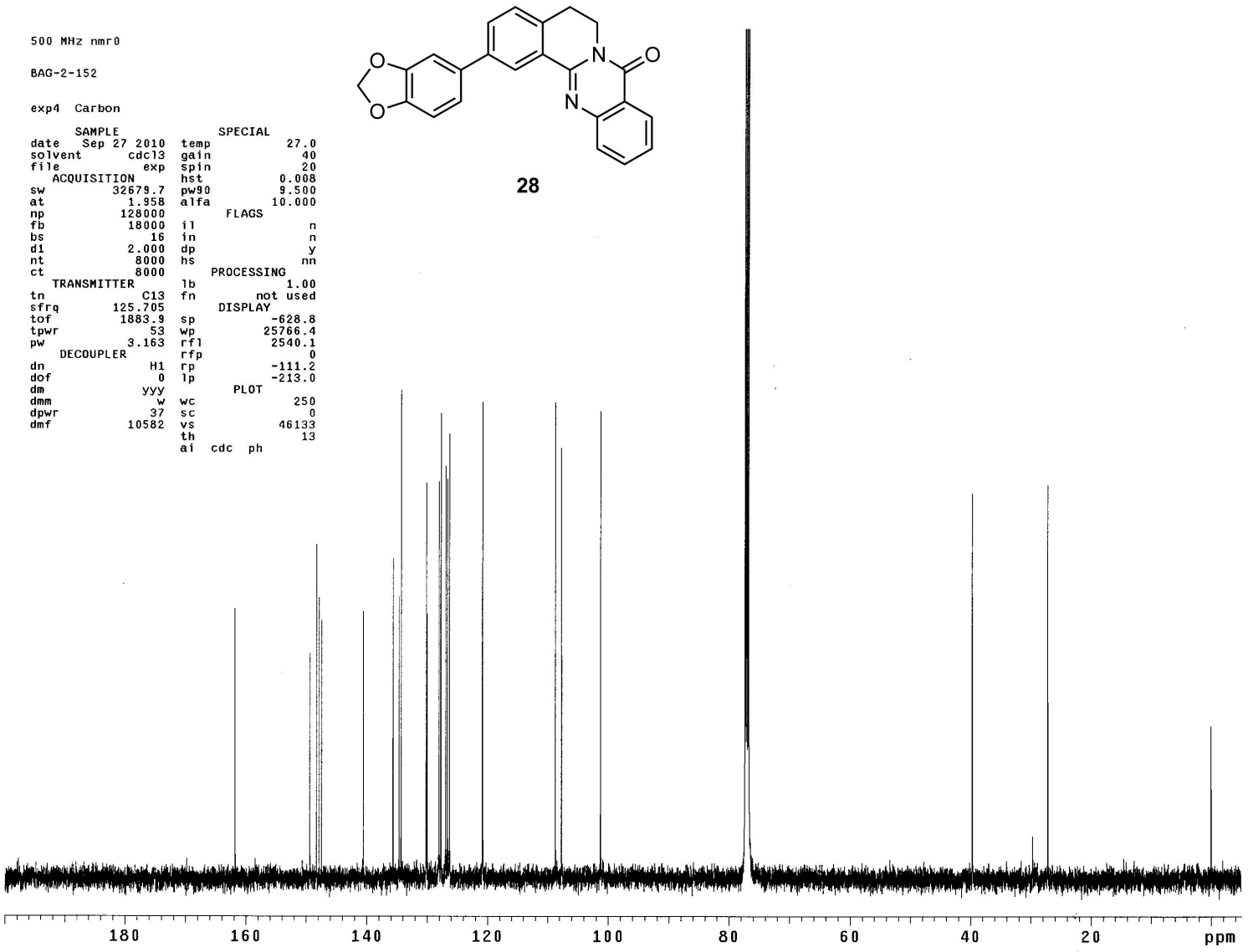
exp1 Proton

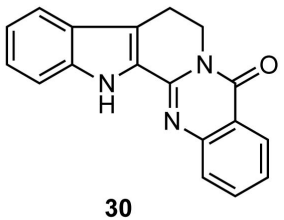
SAMPLE		SPECIAL	
date	Sep 27 2010	temp	27.0
solvent	cdcl3	gain	30
file	exp	spin	20
ACQUISITION		hst	0.008
sw	7997.6	pw90	11.000
at	4.001	alfa	6.600
np	64000	FLAGS	
fb	4000	il	n
bs	32	in	n
d1	2.000	dp	y
nt	64	hs	nn
ct	64	PROCESSING	
TRANSMITTER		lb	0.10
tn	H1	fn	262144
sfrq	499.868	DISPLAY	
tof	499.7	sp	-250.0
tpwr	57	wp	5248.5
pw	1.833	rfl	999.9
DECOUPLER		rfp	0
dn	C13	rp	163.8
dof	0	lp	-5.2
dm	nnn	PLOT	
dmm	c	wc	250
dpwr	40	sc	0
dmf	14285	vs	659
		th	12
		ai cdc ph	



28




500 MHz nmr0
BAG-2-152
exp4 Carbon
SAMPLE
date Sep 27 2010
solvent cdcl3
file exp
ACQUISITION
sw 32679.7
at 1.958
np 128000
fb 18000
bs 16
d1 2.000
nt 8000
ct 8000
TRANSMITTER
tn C13
sfrq 125.705
tof 1883.9
tpwr 53
pw 3.163
DECOUPLER
dn H1
dof 0
dm yyy
dmm w
dpwr 37
dmf 10582
SPECIAL
temp 27.0
gain 40
spin 20
hst 0.008
pw90 9.500
alfa 10.000
FLAGS
il n
in n
dp y
hs nn
PROCESSING
lb 1.00
fn not used
DISPLAY
sp -628.8
wp 25766.4
rfl 2540.1
rfp 0
rp -111.2
lp -213.0
PLOT
wc 250
sc 0
vs 46133
th 13
ai cdc ph
O
O
N
O
N
28
180
160
140
120
100
80
60
40
20
ppm

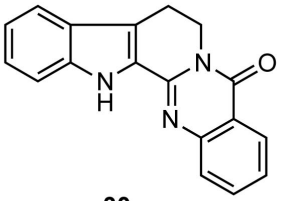


30

```
 Archive directory:
 Sample directory:

Pulse Sequence: s2pul
 Solvent: cdcl3
 Ambient temperature
File: BAG-3-89_s2pul_H1
INOVA-500  "nmrelroy"

 Relax. delay 2.000 sec
 Pulse 30.0 degrees
 Acq. time 4.000 sec
 Width 6410.3 Hz
 32 repetitions
OBSERVE    H1, 399.8047119 MHz
DATA PROCESSING
 Line broadening 0.1 Hz
FT size 65536
Total time 3 min, 24 sec
```



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Archive directory:
Sample directory:

Pulse Sequence: s2pul
Solvent: cdcl3
Ambient temperature
User: 1-14-87
File: BAG-3-89_s2pul_C13
INOVA-500 "nmrelroy"

Relax. delay 2.000 sec
Pulse 30.0 degrees
Acq. time 1.300 sec
Width 24509.8 Hz
2000 repetitions
OBSERVE C13, 100.5309764 MHz
DECOUPLE H1, 399.8067105 MHz
Power 44 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 2.0 Hz
FT size 65536
Total time 1 hr, 50 min, 18 sec