Supporting Information

For

Libraries of 2,3,4,7,11*b*-hexahydro-1*H*-pyrido[2,1-*a*]isoquinolin-2-amine Derivatives via a Multicomponent Assembly Process/1,3-Dipolar Cycloaddition Strategy

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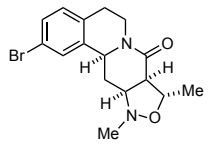
General Methods. Methanol (MeOH) and acetonitrile (CH_3CN) were dried by filtration through two columns of activated molecular sieves. Tetrahydrofuran (THF) and toluene were passed through two columns of activated neutral alumina prior to use. Triethylamine (Et_3N), dichloromethane (CH_2Cl_2), 1,2-dichloroethane (DCE), morpholine, *N*-methylpiperazine, *trans*-crotonoyl chloride, 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. 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.

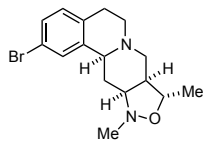
Proton nuclear magnetic resonance (^{1}H NMR) and carbon nuclear magnetic resonance (^{13}C NMR) spectra were acquired in $CDCl_3$ unless otherwise noted. Chemical shifts are reported in parts per million (ppm, δ), downfield from tetramethylsilane (TMS, δ = 0.00 ppm) and are referenced to residual solvent ($CDCl_3$, 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; dddd, doublet of 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. Purity was determined using an LCMS system comprised of an Agilent 1200 Series HPLC and an Agilent 6130 single quadrupole mass spectrometer. Samples were injected onto a Phenomenex Gemini C18 column (5 micron, 2.1 x 50 mm) and eluted at 0.7 mL/min using a gradient of 10-90% acetonitrile, 0.1% formic acid (11 minute linear ramp). Positive mode electrospray ionization was used to verify the identity of the major component, and a UV chromatogram recorded at 214 nm was integrated to determine compound

purity. 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] unless otherwise noted.



10

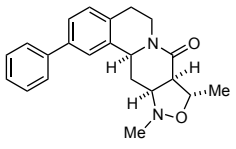
2-Bromo-8,10-dimethyl-5,6,7*a*,8,10,10*a*,11,11*a*-octahydro-9-oxa-6*a*,10-diaza-cyclopenta[*b*]phenanthren-7-one (10). *trans*-Crotonoyl chloride (548 mg, 0.5 mL, 5.24 mmol) was added to a solution of imine **7** (1.0 g, 4.76 mmol) and silyl enol ether **8**[2] (0.90 g, 1.1 mL, 5.71 mmol) in CH_3CN (95 mL) at room temperature. Freshly distilled TMSOTf (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 (pale yellow oil) 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 **10** as small colorless 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), 3.22-3.14 (m, 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] LCMS purity: 98%.



11

2-Bromo-8,10-dimethyl-5,6,7*a*,8,10,10*a*,11,11*a*-octahydro-7*H*-9-oxa-6*a*,10-diaza-cyclopenta[*b*]phenanthrene (11). 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 **10** (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 **11** as a colorless 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), 3.28-3.18 (m, 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]; LCMS purity: 100%.

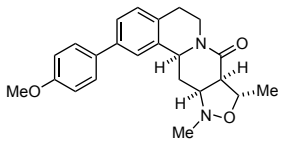
Representative procedure for the formation of 13{1-3} via Suzuki cross-coupling:



13{1}

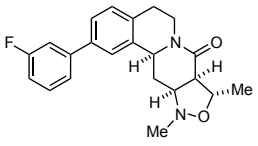
8,10-Dimethyl-2-phenyl-5,6,7*a*,8,10,10*a*,11,11*a*-octahydro-9-oxa-6*a*,10-diaza-cyclopenta[*b*]phenanthren-7-one (13{1}). A mixture of isoxazolidine **10** (1.0 g, 2.9 mmol), cesium carbonate (1.9 g, 5.7 mmol), phenylboronic acid (**12**{1}) (0.7 g, 5.7 mmol), and bis(tri-

tert-butylphosphine)palladium(0) (15 mg, 0.03 mmol) in degassed 1,4-dioxane (14 mL) was heated at 90 °C for 3 h. The reaction was cooled to room temperature, filtered through a pad of celite, and the filtrate was concentrated under reduced pressure. The resultant yellow oil was purified by flash column chromatography eluting with hexanes/EtOAc (1 : 2) to give 0.99 g (99%) of **13**{1} as a fluffy colorless solid: mp 183-183.5 °C (colorless needles from 1 : 1 EtOH : H_2O); ^{1}H NMR (600 MHz) δ 7.57-7.55 (comp, 2 H), 7.46-7.43 (comp, 3 H), 7.37-7.34 (m, 1 H), 7.37 (d, *J* = 1.2 Hz, 1 H), 7.24 (d, *J* = 7.8 Hz, 1 H), 4.87-4.82 (m, 1 H), 4.75 (dd, *J* = 12.0, 2.4 Hz, 1 H), 4.37-4.32 (m, 1 H), 3.25-3.16 (m, 1 H), 2.94-2.80 (comp, 4 H), 2.70 (s, 3 H), 2.44 (ddd, *J* = 13.2, 7.2, 2.4 Hz, 1 H), 1.71-1.65 (m, 1 H), 1.53 (d, *J* = 6.6 Hz, 3 H); ^{13}C NMR (150 MHz) δ 169.4, 140.6, 140.1, 136.0, 134.4, 129.3, 128.9, 127.5, 127.0, 125.8, 124.9, 76.4, 65.2, 54.2, 53.5, 44.0, 38.7, 37.0, 29.0, 20.1; IR (neat) 3485, 1645, 1427 cm^{-1}; mass spectrum (CI) *m/z* 349.1911 [$C_{22}H_{25}N_2O_2$ (M+1) requires 349.1916]; LCMS purity: 97%.



13{2}

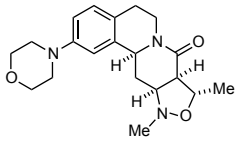
2-(4-Methoxy-phenyl)-8,10-dimethyl-5,6,7*a*,8,10,10*a*,11,11*a*-octahydro-9-oxa-6*a*,10-diaza-cyclopenta[*b*]phenanthren-7-one (13{2}). Prepared according to the representative procedure for the formation of **13**{1-3} via Suzuki cross-coupling. Purification: hexanes/EtOAc (35 : 65 → 0 : 100). Yield: 94% (0.30 g colorless solid). Data: mp 160-160.5 °C (colorless needles from 1 : 1 EtOH : H_2O); ^{1}H NMR (600 MHz) δ 7.49 (d, *J* = 9.0 Hz, 2 H), 7.40 (dd, *J* = 7.8, 1.8 Hz, 1 H), 7.32 (d, *J* = 1.8 Hz, 1 H), 7.22 (d, *J* = 7.8 Hz, 1 H), 6.98 (d, *J* = 9.0 Hz, 2 H), 4.83 (dd, *J* = 8.4, 2.4 Hz, 1 H), 4.74 (dd, *J* = 12.0, 1.8 Hz, 1 H), 4.36-4.32 (m, 1 H), 3.86 (s, 3 H), 3.24-3.16 (m, 1 H), 2.93-2.78 (comp, 4 H), 2.70 (s, 3 H), 2.44 (ddd, *J* = 13.2, 7.2, 2.4 Hz, 1 H), 1.71-1.64 (m, 1 H), 1.53 (d, *J* = 6.0 Hz, 3 H); ^{13}C NMR (150 MHz) δ 169.4, 159.3, 139.7, 135.9, 133.7, 133.1, 129.3, 128.0, 125.4, 124.4, 114.3, 76.4, 65.2, 55.4, 54.3, 53.5, 44.0, 38.7, 37.0, 28.9, 20.1; IR (neat) 1643, 1495, 1425, 1247 cm^{-1}; mass spectrum (CI) *m/z* 379.2019 [$C_{23}H_{27}N_2O_3$ (M+1) requires 379.2022]; LCMS purity: 97%.



13{3}

2-(3-Fluoro-phenyl)-8,10-dimethyl-5,6,7*a*,8,10,10*a*,11,11*a*-octahydro-9-oxa-6*a*,10-diaza-cyclopenta[*b*]phenanthren-7-one (13{3}). Prepared according to the representative procedure for the formation of **13**{1-3} via Suzuki cross-coupling. Purification: hexanes/EtOAc (35 : 65). Yield: Quantitative (51 mg colorless solid). Data: mp 140-141 °C (colorless needles from 1 : 1 EtOH : H_2O); ^{1}H NMR (600 MHz) δ 7.43-7.38 (comp, 2 H), 7.36-7.33 (comp, 2 H), 7.27-7.24 (comp, 2 H), 7.05 (dddd, J = 8.4, 8.4, 2.4, 0.6 Hz, 1 H), 4.88-4.82 (m, 1 H), 4.75 (dd, J = 11.4, 1.2 Hz, 1 H), 4.36-4.32 (m, 1 H), 3.26-3.16 (m, 1 H), 2.94-2.81 (comp, 4 H), 2.71 (s, 3 H), 2.44 (ddd, J = 13.2, 7.2, 2.4 Hz, 1 H), 1.71-1.65 (m, 1 H), 1.53 (d, J = 6.0 Hz, 3 H); ^{13}C NMR (150 MHz) δ 169.4, 163.2 ($J_{C\text{-}F}$ = 244.8 Hz), 142.8 ($J_{C\text{-}F}$ = 7.5 Hz), 138.7 ($J_{C\text{-}F}$ = 2.3 Hz), 136.2, 135.0, 130.3 ($J_{C\text{-}F}$ = 8.3 Hz), 129.5, 125.7, 124.8, 122.6 ($J_{C\text{-}F}$ = 2.9 Hz), 114.3 ($J_{C\text{-}F}$ = 21.3 Hz), 113.9 ($J_{C\text{-}F}$ = 21.8 Hz), 76.4, 65.2, 54.2, 53.5, 44.0, 38.6, 37.0, 29.0, 20.1; IR (neat) 3467, 1645, 1427 cm^{-1} ; mass spectrum (CI) *m/z* 367.1819 [$C_{22}H_{24}FN_2O_2$ (M+1) requires 367.1822]; LCMS purity: 98%.

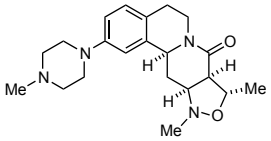
Representative procedure for the formation of 13{4-5} via Buchwald-Hartwig amination:



13{4}

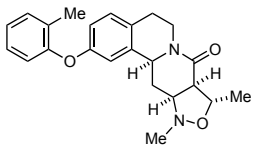
8,10-Dimethyl-2-morpholin-4-yl-5,6,7*a*,8,10,10*a*,11,11*a*-octahydro-9-oxa-6*a*,10-diaza-cyclopenta[*b*]phenanthren-7-one (13{4}). A mixture of $Pd(OAc)_2$ (9.7 mg, 0.04 mmol) and biphenyl-2-yldi-tert-butylphosphine (12.8 mg, 0.04 mmol) in anhydrous toluene (0.5 mL) was stirred for 5 min at room temperature. The catalyst mixture was added to a solution of **10** (0.3 g, 0.85 mmol), morpholine (**12**{4}) (90 μL, 1.03 mmol), and NaO*t*-Bu (0.12 g, 1.11 mmol) in anhydrous toluene (1.4 mL), and the reaction was heated at 100 °C for 3 h. The reaction mixture was then cooled to room temperature, filtered through a pad of celite, and the filtrate was concentrated under reduced pressure. The resultant yellow oil was purified by flash column chromatography eluting with EtOAc/MeOH (95 : 5) to give 0.31 g (Quantitative) of **13**{4} as a

yellow solid: mp 63-64 °C (yellow crystals from 1 : 1 EtOH : H_2O); ^{1}H NMR (500 MHz) δ 7.08 (d, *J* = 8.5 Hz, 1 H), 6.81 (dd, *J* = 8.5, 2.5 Hz, 1 H), 6.67 (d, *J* = 2.5 Hz, 1 H), 4.81-4.79 (m, 1 H), 4.63 (dd, *J* = 12.0, 2.0 Hz, 1 H), 4.35-4.32 (m, 1 H), 3.87-3.85 (comp, 4 H), 3.19-3.10 (comp, 5 H), 2.89-2.73 (comp, 3 H), 2.71-2.67 (comp, 4 H), 2.36 (ddd, *J* = 13.5, 7.5, 2.5 Hz, 1 H), 1.66-1.59 (m, 1 H), 1.52 (d, *J* = 6.5 Hz, 3 H); ^{13}C NMR (125 MHz) δ 169.3, 150.3, 136.2, 129.6, 126.9, 115.0, 113.1, 76.5, 66.9, 65.1, 54.4, 53.4, 49.6, 44.0, 38.9, 37.1, 28.3, 20.1; IR (neat) 2961, 2923, 2853, 1645, 1512, 1422, 1242, 1121 cm^{-1}; mass spectrum (CI) *m/z* 358.2133 [$C_{20}H_{28}N_3O_3$ (M+1) requires 358.2131]; LCMS purity: 97%.



13{5}

8,10-Dimethyl-2-(4-methyl-piperazin-1-yl)-5,6,7*a*,8,10,10*a*,11,11*a*-octahydro-9-oxa-6*a*,10-diaza-cyclopenta[*b*]phenanthren-7-one (13{5}). Prepared according to the representative procedure for the formation of **13**{4-5} via Buchwald-Hartwig amination Purification: EtOAc/MeOH (100 : 0 → 70 : 30 → 65 : 35) with 1% Et_3N. Yield: 88% (0.93 g yellow solid). Data: mp 139-140 °C (yellow needles from 1 : 1 EtOH : H_2O); ^{1}H NMR (600 MHz) δ 7.06 (d, *J* = 8.4 Hz, 1H), 6.82 (dd, *J* = 8.4, 2.4 Hz, 1 H), 6.68 (d, *J* = 2.4 Hz, 1 H), 4.80-4.77 (m, 1 H), 4.63 (dd, *J* = 12.0, 1.2 Hz, 1 H), 4.35-4.30 (m, 1 H), 3.21-3.18 (comp, 5 H), 2.84-2.75 (comp, 3 H), 2.70-2.66 (m, 1 H), 2.70 (s, 3 H), 2.59-2.57 (comp, 4 H), 2.38-2.31 (m, 1 H), 2.36 (s, 3 H), 1.65-1.59 (m, 1 H), 1.52 (d, *J* = 6.6 Hz, 3 H); ^{13}C NMR (150 MHz) δ 169.3, 150.3, 136.1, 129.5, 126.5, 115.3, 113.4, 76.5, 65.2, 55.1, 54.4, 53.5, 49.4, 46.1, 44.1, 38.9, 37.0, 28.3, 20.2; IR (neat) 3480, 1642, 1511, 1424, 1289, 1245 cm^{-1}; mass spectrum (CI) *m/z* 371.2449 [$C_{21}H_{31}N_4O_2$ (M+1) requires 371.2447]; LCMS purity: 100%.

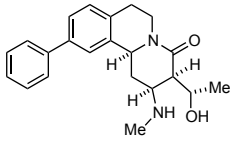


13{6}

8,10-Dimethyl-2-*o*-tolyloxy-5,6,7*a*,8,10,10*a*,11,11*a*-octahydro-9-oxa-6*a*,10-diaza-cyclopenta[*b*]phenanthren-7-one (13{6}). A mixture of $Pd(OAc)_2$ (31 mg, 0.14 mmol) and biphenyl-2-yldi-tert-butylphosphine (42 mg, 0.14 mmol) in anhydrous toluene (0.5 mL) was stirred for 5 min at room temperature. The catalyst mixture was added to a solution of **10** (1.0 g,

2.9 mmol), *o*-cresol (**12**{6}) (0.37 g, 0.35 mL, 3.4 mmol), and K_3PO_4 (1.2 g, 5.7 mmol) in anhydrous toluene (9.5 mL), and the reaction was heated at 100 °C for 17 h. The reaction mixture was then cooled to room temperature, filtered through a pad of celite, and the filtrate was concentrated under reduced pressure. The resultant yellow oil was purified by flash column chromatography eluting with hexanes/EtOAc (45 : 55 → 35 : 65) to give 0.62 g (57%) of **13**{6} as a cream colored solid: mp 159-160 °C (colorless crystals from 1 : 1 EtOH : H_2O); ^{1}H NMR (500 MHz) δ 7.26 (dd, *J* = 7.5, 1.0 Hz, 1 H), 7.17 (ddd, *J* = 7.5, 7.5, 1.0 Hz, 1 H), 7.09-7.06 (comp, 2 H), 6.88 (dd, *J* = 8.0, 0.5 Hz, 1 H), 6.77-6.73 (comp, 2 H), 4.83-4.76 (m, 1 H), 4.60 (dd, *J* = 12.0, 2.0 Hz, 1 H), 4.34-4.28 (m, 1 H), 3.20-3.11 (m, 1 H), 2.89-2.69 (comp, 4 H), 2.68 (s, 3 H), 2.30 (ddd, *J* = 13.0, 7.0, 2.5 Hz, 1 H), 2.25 (s, 3 H), 1.66-1.59 (m, 1 H), 1.52 (d, *J* = 6.0 Hz, 3 H); ^{13}C NMR (125 MHz) δ 169.3, 156.6, 154.4, 137.0, 131.6, 130.1, 129.8, 129.2, 127.2, 124.1, 119.4, 116.2, 114.8, 76.5, 65.0, 54.1, 53.4, 44.2, 38.9, 36.6, 28.5, 20.2, 16.2; IR (neat) 3436, 1646, 1489, 1422, 1248 cm^{-1}; mass spectrum (CI) *m/z* 379.2021 [$C_{23}H_{27}N_2O_3$ (M+1) requires 379.2022] LCMS purity: 95%.

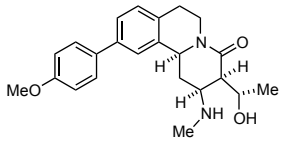
Representative procedure for the formation of 14{1-6} **via *N,O*-bond cleavage:**



14{1}

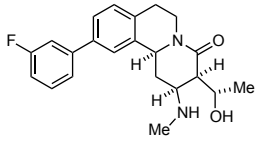
3-(1-Hydroxyethyl)-2-(methylamino)-10-phenyl-2,3,6,7-tetrahydro-1*H*-pyrido[2,1-*a*]isoquinolin-4(11*bH*)-one (14{1}**)**. A mixture of isoxazolidine **13**{1} (1.0 g, 2.9 mmol) in anhydrous MeOH (57 mL) was added $NiCl_2 \cdot 6H_2O$ (1.3 g, 5.7 mmol), followed by $NaBH_4$ (0.64 g, 16.8 mmol). The black mixture was stirred for 3 h at room temperature, and the solvent was evaporated under reduced pressure. The resultant black residue was partitioned between CH_2Cl_2 (50 mL) and concentrated NH_4OH (50 mL). The biphasic mixture was stirred for 3 h at room temperature, and the layers were separated. The aqueous layer was extracted with CH_2Cl_2 (2 × 5 mL), and the combined organic layers were dried (Na_2SO_4), filtered, and concentrated under reduced pressure. The resultant yellow gum was purified by flash column chromatography on Florisil® gel (Fisherbrand, 60-100 mesh) eluting with EtOAc/MeOH (7 : 3) with 1% Et_3N to give 0.88 g (87%) of **14**{1} as a yellow solid: mp 161-163 °C; ^{1}H NMR (500 MHz) δ 7.55-7.52 (comp, 2 H), 7.45-7.41 (comp, 3 H), 7.37-7.32 (comp, 2 H), 7.21 (d, *J* = 7.5 Hz, 1 H), 4.81-4.70

(comp, 2 H), 4.29-4.24 (m, 1 H), 3.37-3.34 (m, 1 H), 2.97-2.88 (comp, 2 H), 2.82-2.75 (m, 1 H), 2.63 (dd, *J* = 7.0, 5.0 Hz, 1 H), 2.60-2.55 (m, 1 H), 2.41 (s, 3 H), 2.12 (ddd, *J* = 13.5, 11.0, 9.5 Hz, 1 H), 1.25 (d, *J* = 6.0 Hz, 3 H); ^{13}C NMR (125 MHz) δ 169.6, 140.7, 139.9, 137.0, 133.7, 129.4, 128.8, 127.4, 127.0, 125.8, 123.6, 66.9, 55.4, 54.6, 49.3, 39.6, 34.3, 33.9, 28.6, 21.4; IR (neat) 3314, 3053, 2926, 2850, 1637, 1486, 1434, 764, 735, 700 cm^{-1}; mass spectrum (CI) *m/z* 351.2074 [$C_{22}H_{27}N_2O_2$ (M+1) requires 351.2073]; LCMS purity: 95%.



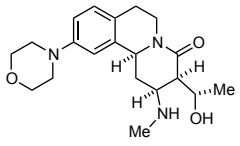
14{2}

3-(1-Hydroxyethyl)-10-(4-methoxyphenyl)-2-(methylamino)-2,3,6,7-tetrahydro-1*H*-pyrido[2,1-*a*]isoquinolin-4(11*bH*)-one (14{2}). Prepared according to the representative procedure for the formation of **14**{1-6} via *N,O*-bond cleavage. Purification: EtOAc/MeOH (100 : 0 → 70 : 30) with 1% Et_3N. Yield: 88% (221 mg yellow solid). Data: ^{1}H NMR (400 MHz) δ 7.50-7.48 (comp, 2 H), 7.41 (d, *J* = 8.0, 1.6 Hz, 1 H), 7.36-7.34 (m, 1 H), 7.22 (d, *J* = 8.0 Hz, 1 H), 7.00-6.97 (comp, 2 H), 4.81-4.72 (comp, 2 H), 4.30 (ddd, *J* = 12.8, 6.4, 6.4, 1 H), 3.86 (s, 3 H), 3.89 (ddd, *J* = 9.6, 4.8, 4.8 Hz, 1 H), 2.97-2.90 (comp, 2 H), 2.84-2.79 (m, 1 H), 2.65 (dd, *J* = 6.8, 4.4 Hz, 1 H), 2.62-2.55 (m, 1 H), 2.43 (s, 3 H), 2.14 (ddd, *J* = 12.8, 10.8, 9.2 Hz, 1 H), 1.27 (d, *J* = 6.4 Hz, 3 H).



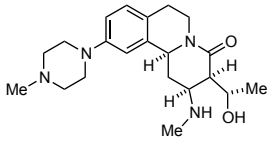
14{3}

10-(3-Fluorophenyl)-3-(1-hydroxyethyl)-2-(methylamino)-2,3,6,7-tetrahydro-1*H*-pyrido[2,1-*a*]isoquinolin-4(11*bH*)-one (14{3}). Prepared according to the representative procedure for the formation of **14**{1-6} via *N,O*-bond cleavage. Purification: EtOAc/MeOH (100 : 0 → 70 : 30). Yield: 83% (255 mg yellow solid). Data: ^{1}H NMR (400 MHz) δ 7.44-7.32 (comp, 4 H), 7.27-7.24 (comp, 2 H), 7.06 (dddd, *J* = 8.4, 8.4, 2.4, 0.8 Hz, 1 H), 4.83-4.73 (comp, 2 H), 4.29 (ddd, *J* = 12.8, 6.4, 6.4 Hz, 1 H), 3.40 (ddd, *J* = 9.6, 4.8, 4.8 Hz, 1 H), 2.98-2.93 (comp, 2 H), 2.85-2.81 (m, 1 H), 2.66 (dd, *J* = 6.8, 4.0 Hz, 1 H), 2.63-2.55 (m, 1 H), 2.44 (s, 3 H), 2.14 (ddd, *J* = 13.2, 10.8, 9.2 Hz, 1 H), 1.24 (d, *J* = 6.4 Hz, 3 H).



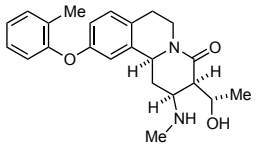
14{4}

3-(1-Hydroxyethyl)-2-(methylamino)-10-morpholino-2,3,6,7-tetrahydro-1*H*-pyrido[2,1-*a*]isoquinolin-4(11*bH*)-one (14{4}). Prepared according to the representative procedure for the formation of **14**{1-6} via *N,O*-bond cleavage. Purification: EtOAc/MeOH (100 : 0 → 80 : 20). Yield: 81% (204 mg yellow solid). Data: ^{1}H NMR (400 MHz) δ 7.08 (d, *J* = 8.4 Hz, 1 H), 6.82 (dd, *J* = 8.4, 2.4 Hz, 1 H), 6.71 (d, *J* = 2.4 Hz, 1 H), 4.78-4.74 (m, 1 H), 4.64 (dd, *J* = 10.8, 6.0 Hz, 1 H), 4.32-4.25 (m, 1 H), 3.88-3.85 (comp, 4 H), 3.40-3.35 (m, 1 H), 3.19-3.13 (comp, 4 H), 2.88-2.80 (comp, 2 H), 2.72-2.68 (m, 1 H), 2.63 (dd, *J* = 6.4, 4.4 Hz, 1 H), 2.53-2.47 (m, 1 H), 2.43 (s, 3 H), 2.06 (ddd, *J* = 12.8, 10.8, 9.2 Hz, 1 H), 1.26 (d, *J* = 6.4 Hz, 3 H).



14{5}

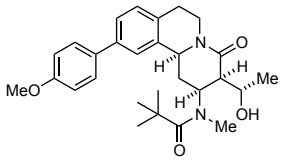
3-(1-Hydroxyethyl)-2-(methylamino)-10-(4-methylpiperazin-1-yl)-2,3,6,7-tetrahydro-1*H*-pyrido[2,1-*a*]isoquinolin-4(11*bH*)-one (14{5}). Prepared according to the representative procedure for the formation of **14**{1-6} via *N,O*-bond cleavage. Purification: EtOAc/MeOH (60 : 40) with 1% Et_3N. Yield: 90% (1.0 g yellow gum). Data: ^{1}H NMR (500 MHz) δ 7.00 (d, *J* = 8.5 Hz, 1 H), 6.77 (dd, *J* = 8.5, 2.0 Hz, 1 H), 6.67 (d, *J* = 2.0 Hz, 1 H), 4.71-4.65 (m, 1 H), 4.58 (dd, *J* = 10.5, 5.5 Hz, 1 H), 4.24-4.20 (m, 1 H), 3.34-3.29 (m, 1 H), 3.14-3.12 (comp, 4 H), 2.84-2.73 (comp, 2 H), 2.67-2.62 (m, 1 H), 2.57 (dd, *J* = 6.5, 5.0 Hz, 1 H), 2.54-2.52 (comp, 4 H), 2.47-2.44 (m, 1 H), 2.37 (s, 3 H), 2.30 (s, 3 H), 2.01 (ddd, *J* = 13.5, 11.0, 9.5 Hz, 1 H), 1.21 (d, *J* = 6.5 Hz, 3 H); ^{13}C NMR (125 MHz) δ 169.6, 150.1, 137.0, 129.5, 125.8, 115.3, 112.4, 66.8, 55.3, 55.0, 54.6, 49.3, 49.2, 46.0, 39.7, 34.6, 33.9, 28.0, 21.3; IR (neat) 3410, 2937, 2806, 1622, 1452, 1142, 1115, 1010, 793, 730 cm^{-1}; mass spectrum (CI) *m/z* 373.2602 [$C_{21}H_{33}N_4O_2$ (M+1) requires 373.2604]; LCMS purity: 96%.



14{6}

3-(1-Hydroxyethyl)-2-(methylamino)-10-(*o*-tolyloxy)-2,3,6,7-tetrahydro-1*H*-pyrido[2,1-*a*]isoquinolin-4(11*bH*)-one (14{6}). Prepared according to the representative procedure for the formation of **14**{1-6} via *N,O*-bond cleavage. Purification: EtOAc/MeOH (70 : 30). Yield: 92% (0.7 g yellow solid). Data: mp 55-56 °C (dec.); ^{1}H NMR (500 MHz) δ 7.24-2.23 (m, 1 H), 7.16-7.13 (m, 1 H), 7.06-7.04 (comp, 2 H), 6.85 (d, *J* = 8.0 Hz, 1 H), 6.75 (d, *J* = 2.0 Hz, 1 H), 6.71 (dd, *J* = 8.0, 2.0 Hz, 1 H), 4.75-4.71 (m, 1 H), 4.60 (dd, *J* = 10.5, 6.0 Hz, 1 H), 4.27-4.22 (m, 1 H), 3.31 (ddd, *J* = 9.0, 4.0, 4.0 Hz, 1H), 2.88-2.79 (comp, 2 H), 2.74-2.67 (m, 1 H), 2.60 (dd, *J* = 7.0, 5.0 Hz, 1 H), 2.41-2.37 (comp, 4 H), 2.22 (s, 3 H), 2.07-2.00 (m, 1 H), 1.24 (d, *J* = 6.0 Hz, 3 H); ^{13}C NMR (125 MHz) δ 169.5, 156.5, 154.3, 137.9, 131.5, 130.1, 129.8, 128.5, 127.2, 124.0, 119.3, 116.1, 113.8, 67.0, 55.4, 54.6, 49.3, 39.7, 34.0, 33.9, 28.2, 21.5, 16.1; IR (neat) 3435, 2920, 2850, 1637, 1436, 1281, 1114, 757, 734 cm^{-1}; mass spectrum (CI) *m/z* 381.2181 [$C_{23}H_{29}N_2O_3$ (M+1) requires 381.2178].

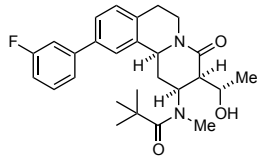
Representative procedure for the formation of amides:



16{2,1}

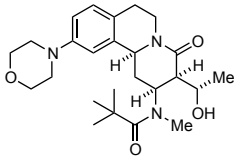
***N*-(3-(1-Hydroxyethyl)-10-(4-methoxyphenyl)-4-oxo-2,3,4,6,7,11*b*-hexahydro-1*H*-pyrido[2,1-*a*]isoquinolin-2-yl)-*N*-methylpivalamide (16{2,1}).** Pivaloyl chloride (**15**{1}) (11 mg, 11 μL, 0.09 mmol) was added to a solution of 1,3-amino alcohol **14**{2} (30 mg, 0.08 mmol) and triethylamine (9.6 mg, 13 μL, 0.10 mmol) in anhydrous CH_2Cl_2 (0.40 mL) at 0 °C, and the solution was stirred for 45 min at 0 °C. Saturated $NaHCO_3$ (2 mL) and CH_2Cl_2 (2 mL) were added and the layers were separated. The aqueous layer was extracted with CH_2Cl_2 (2 × 2 mL), and the organic layer was dried (Na_2SO_4), filtered, and concentrated under reduced pressure. The resultant yellow oil was purified by flash column chromatography eluting with EtOAc to give 28 mg (76%) of **16**{2,1} as a cream colored solid: ^{1}H NMR (400 MHz) δ 7.49 (d, *J* = 8.8 Hz, 2 H), 7.42 (dd, *J* = 7.6, 1.6 Hz, 1 H), 7.32 (d, *J* = 1.6 Hz, 1 H), 7.25 (d, *J* = 7.6 Hz, 1 H), 6.99 (d, *J* =

8.8 Hz, 2 H), 5.67 (ddd, *J* = 10.0, 5.2, 5.2 Hz, 1 H), 4.74-4.64 (comp, 2 H), 4.49-4.43 (m, 1 H), 3.99-3.96 (m, 1 H), 3.86 (s, 3 H), 3.13-3.06 (m, 1 H), 2.98 (s, 3 H), 2.90-2.88 (comp, 2 H), 2.97 (ddd, *J* = 14.8, 10.0, 4.4 Hz, 1 H), 2.58-2.53 (m, 1 H), 1.73 (ddd, *J* = 14.4, 12.0, 5.2 Hz, 1 H), 1.44 (dd, *J* = 6.4 Hz, 3 H), 1.27 (s, 9 H); LCMS purity: 96%.



16{3,1}

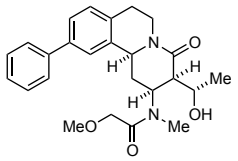
***N*-(10-(3-Fluorophenyl)-3-(1-hydroxyethyl)-4-oxo-2,3,4,6,7,11*b*-hexahydro-1*H*-pyrido[2,1-*a*]isoquinolin-2-yl)-*N*-methylpivalamide (16{3,1})**. Prepared according to the representative procedure for the formation of amides. Purification: EtOAc. Yield: 65% (40 mg colorless solid). Data: mp 203-204 °C (colorless crystals from 1 : 1 EtOH : H_2O); ^{1}H NMR (600 MHz) δ 7.44 (dd, *J* = 7.8, 1.8 Hz, 1 H), 7.41 (ddd, *J* = 7.8, 7.8, 6.0 Hz, 1 H), 7.36 (d, *J* = 1.8 Hz, 1 H), 7.33 (ddd, *J* = 7.8, 1.8, 1.2 Hz, 1 H), 7.29 (d, *J* = 7.8 Hz, 1H), 7.25 (ddd, *J* = 10.2, 2.4, 1.8 Hz, 1 H), 7.08-7.04 (m, 1 H), 5.67 (ddd, *J* = 9.6, 6.0, 6.0 Hz, 1 H), 4.73 (dd, *J* = 12.0, 4.2 Hz, 1 H), 4.67 (ddd, *J* = 8.4, 8.4, 4.2 Hz, 1 H), 4.40 (d, *J* = 2.4 Hz, 1 H), 4.00-3.96 (m, 1 H), 3.11 (ddd, *J* = 13.2, 9.0, 6.0 Hz, 1 H), 2.99 (s, 3 H), 2.93-2.91 (comp, 2 H), 2.70 (ddd, *J* = 15.0, 10.2, 4.8 Hz, 1 H), 2.57-2.55 (m, 1 H), 1.73 (ddd, *J* = 15.0, 12.0, 6.0 Hz, 1 H), 1.44 (d, *J* = 6.0 Hz, 3 H), 1.27 (s, 9 H); ^{13}C NMR (150 MHz) δ 180.0, 171.6, 163.2 ($J_{C\text{-}F}$ = 244.8 Hz), 142.7 ($J_{C\text{-}F}$ = 7.7 Hz), 139.0 ($J_{C\text{-}F}$ = 2.1 Hz), 135.8, 134.7, 130.4 ($J_{C\text{-}F}$ = 8.6 Hz), 129.5, 126.0, 124.9, 122.6 ($J_{C\text{-}F}$ = 2.9 Hz), 114.4 ($J_{C\text{-}F}$ = 21.2 Hz), 113.9 ($J_{C\text{-}F}$ = 21.7 Hz), 64.7, 53.3, 51.3, 48.3, 39.5, 38.1, 36.6, 31.8, 29.0, 28.3, 21.2; IR (neat) 3377, 2970, 2244, 1649, 1613, 1481, 1406, 1093, 732 cm^{-1}; mass spectrum (CI) *m/z* 453.2549 [$C_{27}H_{34}FN_2O_3$ (M+1) requires 453.2553]; LCMS purity: 96%.



16{4,1}

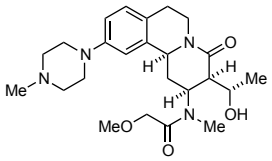
***N*-(3-(1-Hydroxyethyl)-10-morpholino-4-oxo-2,3,4,6,7,11*b*-hexahydro-1*H*-pyrido[2,1-*a*]isoquinolin-2-yl)-*N*-methylpivalamide (16{4,1})**. Prepared according to the representative procedure for the formation of amides. Purification: EtOAc/MeOH (100 : 0 → 90 : 10). Yield: 67% (20 mg yellow solid). Data: ^{1}H NMR (400 MHz) δ 7.11 (d, *J* = 8.8 Hz, 1 H),

6.82 (dd, *J* = 8.8, 2.8 Hz, 1 H), 6.66 (d, *J* = 2.8 Hz, 1 H), 5.69-5.64 (m, 1 H), 4.66-4.58 (comp, 2 H), 4.46-4.40 (m, 1 H), 3.98-3.96 (m, 1 H), 3.88-3.86 (comp, 4 H), 3.14-3.12 (comp, 4 H), 3.04-2.96 (m, 1 H), 2.96 (s, 3 H), 2.79-2.78 (comp, 2 H), 2.62 (ddd, *J* = 14.0, 9.6, 4.0 Hz, 1 H), 2.52-2.48 (m, 1 H), 1.66 (ddd, *J* = 14.4, 12.0, 5.2 Hz, 1 H), 1.43 (d, *J* = 6.4 Hz, 3 H), 1.27 (s, 9 H).



16{1,2}

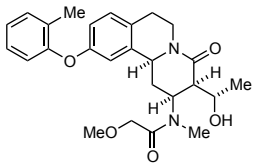
***N*-(3-(1-Hydroxyethyl)-4-oxo-10-phenyl-2,3,4,6,7,11*b*-hexahydro-1*H*-pyrido[2,1-*a*]isoquinolin-2-yl)-2-methoxy-*N*-methylacetamide (16{1,2})**. Prepared according to the representative procedure for the formation of amides. Purification: EtOAc/MeOH (100 : 0 → 90 : 10). Yield: 64% (26 mg colorless solid). Data: mp 93-94 °C; ^{1}H NMR (400 MHz) δ 7.56-7.55 (comp, 2 H), 7.48-7.44 (comp, 3 H), 7.40-7.36 (comp, 2 H), 7.28 (d, *J* = 8.0 Hz, 1 H), 5.66-5.60 (m, 1 H), 4.74 (dd, *J* = 12.0, 4.4 Hz, 1 H), 4.66 (ddd, *J* = 12.8, 4.0, 4.0 Hz, 1 H), 4.26 (d, *J* = 3.2 Hz, 1 H), 4.07 (s, 3 H), 3.42 (s, 3 H), 3.13 (ddd, *J* = 14.0, 13.2, 6.8 Hz, 1 H), 2.93-2.90 (comp, 2 H), 2.83 (s, 3 H), 2.76 (ddd, *J* = 14.8, 10.4, 4.8 Hz, 1 H), 2.57-2.55 (m, 1 H), 1.74 (ddd, *J* = 14.8, 11.6, 4.8 Hz, 1 H), 1.42 (d, *J* = 6.0 Hz, 3 H); ^{13}C NMR (125 MHz) δ 171.4, 171.0, 140.4, 140.3, 135.3, 133.5, 129.3, 128.9, 127.6, 127.0, 126.1, 124.8, 71.3, 64.5, 59.2, 53.1, 50.8, 46.9, 38.1, 36.5, 29.7, 28.9, 20.8; IR (neat) 3433, 2929, 1647, 1412, 1103, 732 cm^{-1}; mass spectrum (CI) *m/z* 423.2275 [$C_{25}H_{31}N_2O_4$ (M+1) requires 423.2284]; LCMS purity: 94%.



16{5,2}

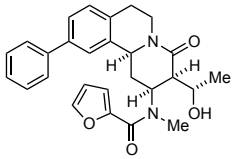
***N*-(3-(1-Hydroxyethyl)-10-(4-methylpiperazin-1-yl)-4-oxo-2,3,4,6,7,11*b*-hexahydro-1*H*-pyrido[2,1-*a*]isoquinolin-2-yl)-2-methoxy-*N*-methylacetamide (16{5,2})**. Prepared according to the representative procedure for the formation of amides. Purification: EtOAc/MeOH (60 : 40) with 1% Et_3N. Yield: 84% (33 mg yellow solid). Data: mp 84-85 °C; ^{1}H NMR (600 MHz) δ 7.09 (d, *J* = 8.4 Hz, 1 H), 6.84 (dd, *J* = 8.4, 2.4 Hz, 1 H), 6.67 (d, *J* = 2.4 Hz, 1 H), 5.62-5.60 (m, 1 H), 4.63-4.58 (comp, 2 H), 4.07 (s, 3 H), 3.41 (s, 3 H), 3.19-3.17 (comp, 4 H), 3.03 (ddd, *J* = 12.6, 8.4, 6.6 Hz, 1 H), 2.81 (s, 3 H), 2.79-2.76 (comp, 3 H), 2.67 (ddd, *J* =

15.0, 10.2, 4.8 Hz, 1 H), 2.60-2.58 (comp, 4 H), 2.51-2.49 (m, 1 H), 2.36 (s, 3 H), 1.67 (ddd, *J* = 15.0, 12.0, 5.4 Hz, 1 H), 1.41 (d, *J* = 6.0 Hz, 3 H); ^{13}C NMR (150 MHz) δ 171.4, 171.0, 150.5, 135.5, 129.5, 125.5, 115.4, 113.4, 71.4, 64.6, 59.2, 55.0, 53.3, 50.8, 49.2, 46.8, 46.1, 38.4, 36.7, 29.7, 28.3, 20.8; IR (neat) 3411, 2935, 2822, 1648, 1424, 1247, 1104, 731 cm^{-1}; mass spectrum (ESI) *m/z* 445.2811 [$C_{24}H_{37}N_4O_4$ (M+1) requires 445.2815]; LCMS purity: 96%.



16{6,2}

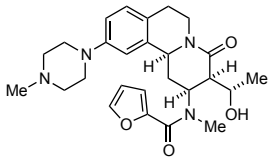
***N*-(3-(1-Hydroxyethyl)-4-oxo-10-(*o*-tolyloxy)-2,3,4,6,7,11*b*-hexahydro-1*H*-pyrido[2,1-*a*]isoquinolin-2-yl)-2-methoxy-*N*-methylacetamide (16{6,2})**. Prepared according to the representative procedure for the formation of amides. Purification: EtOAc/MeOH (100 : 0 → 90 : 10). Yield: 79% (31 mg yellow solid). Data: ^{1}H NMR (400 MHz) δ 7.28-7.26 (m, 1 H), 7.20-7.16 (m, 1 H), 7.14-7.08 (comp, 2 H), 6.88 (dd, *J* = 8.0, 1.2 Hz, 1 H), 6.79 (dd, *J* = 8.4, 2.4 Hz, 1 H), 6.66 (d, *J* = 2.4 Hz, 1 H), 5.60-5.54 (m, 1 H), 4.64-4.55 (comp, 2 H), 4.19 (d, *J* = 2.8 Hz, 1 H), 4.06 (s, 3 H), 3.42 (s, 3 H), 3.07 (ddd, *J* = 14.8, 8.4, 8.4 Hz, 1 H), 2.84-2.81 (comp, 5 H), 2.59 (ddd, *J* = 14.8, 10.0, 4.4 Hz, 1 H), 2.49-2.46 (m, 1 H), 2.23 (s, 3 H), 1.66 (ddd, *J* = 14.8, 12.0, 5.2 Hz, 1 H), 1.39 (d, *J* = 6.0 Hz, 3 H); LCMS purity: 97%.



16{1,3}

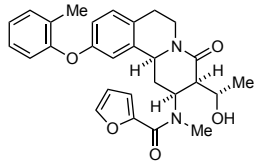
***N*-(3-(1-Hydroxyethyl)-4-oxo-10-phenyl-2,3,4,6,7,11*b*-hexahydro-1*H*-pyrido[2,1-*a*]isoquinolin-2-yl)-*N*-methylfuran-2-carboxamide (16{1,3})**. Prepared according to the representative procedure for the formation of amides. Purification: EtOAc. Yield: 44% (20 mg yellow solid). Data: mp 98-100 °C; ^{1}H NMR (500 MHz) δ 7.57-7.55 (comp, 2 H), 7.49-7.44 (comp, 4 H), 7.39-7.36 (comp, 2 H), 7.29 (d, *J* = 8.0 Hz, 1 H), 7.05 (d, *J* = 3.5 Hz, 1 H), 6.48 (dd, *J* = 3.5, 2.0 Hz, 1 H), 5.71-5.61 (m, 1 H), 4.77 (dd, *J* = 12.0, 4.5 Hz, 1 H), 4.72-4.68 (m, 1 H), 4.14-4.11 (m, 1 H), 3.13-3.09 (comp, 2 H), 2.95-2.92 (comp, 2 H), 2.80 (ddd, *J* = 14.5, 10.0, 4.5 Hz, 1 H), 2.66-2.63 (m, 1 H), 2.17 (s, 3 H), 1.86 (ddd, *J* = 14.5, 12.0, 5.5 Hz, 1 H), 1.45 (d, *J* = 6.5 Hz, 3 H); ^{13}C NMR (125 MHz) δ 171.3, 161.8, 144.5, 140.4, 140.3, 135.4, 133.7, 129.3,

128.9, 127.6, 127.0, 126.1, 124.9, 117.9, 111.5, 64.7, 53.4, 51.0, 48.3, 38.2, 36.8, 32.1, 30.9, 29.0, 21.2; IR (neat) 3417, 2932, 1647, 1487, 1409, 1070, 887, 834, 762, 734, 699 cm^{-1}; mass spectrum (ESI) *m/z* 445.2125 [$C_{27}H_{30}N_2O_4$ (M+1) requires 445.2127]; LCMS purity: 95%.



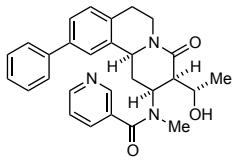
16{5,3}

***N*-(3-(1-Hydroxyethyl)-10-(4-methylpiperazin-1-yl)-4-oxo-2,3,4,6,7,11*b*-hexahydro-1*H*-pyrido[2,1-*a*]isoquinolin-2-yl)-*N*-methylfuran-2-carboxamide (16**{5,3}**)**. Prepared according to the representative procedure for the formation of amides. Purification: EtOAc/MeOH (60 : 40) with 1% Et_3N. Yield: 85% (39 mg yellow oil). Data: ^{1}H NMR (400 MHz) δ 7.49 (dd, *J* = 1.6, 0.8 Hz, 1 H), 7.10 (d, *J* = 8.4 Hz, 1 H), 7.04 (d, *J* = 3.2 Hz, 1 H), 6.84 (dd, *J* = 8.4, 2.4 Hz, 1 H), 6.70 (d, *J* = 2.4 Hz, 1 H), 6.48 (dd, *J* = 3.6, 1.6 Hz, 1 H), 5.65-5.63 (m, 1 H), 4.67-4.62 (comp, 2 H), 4.15-4.08 (m, 1 H), 3.22-3.20 (comp, 4 H), 3.12-2.99 (comp, 4 H), 2.82-2.78 (comp, 2 H), 2.72 (ddd, *J* = 14.4, 10.0, 4.4 Hz, 1 H), 2.65-2.58 (comp, 5 H), 2.39 (s, 3 H), 1.79 (ddd, *J* = 14.4, 12.0, 5.6 Hz, 1 H), 1.43 (d, *J* = 6.4 Hz, 3 H); LCMS purity: 95%.



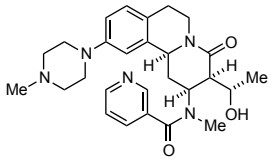
16{6,3}

***N*-(3-(1-Hydroxyethyl)-4-oxo-10-(*o*-tolyloxy)-2,3,4,6,7,11*b*-hexahydro-1*H*-pyrido[2,1-*a*]isoquinolin-2-yl)-*N*-methylfuran-2-carboxamide (16**{6,3}**)**. Prepared according to the representative procedure for the formation of amides. Purification: EtOAc. Yield: 69% (31 mg yellow oil). Data: ^{1}H NMR (400 MHz) δ 7.49 (dd, *J* = 2.0, 0.8 Hz, 1 H), 7.28-7.26 (m, 1 H), 7.20-7.07 (comp, 3 H), 7.04 (d, *J* = 3.2 Hz, 1 H), 6.89 (dd, *J* = 8.0, 1.2 Hz, 1 H), 6.79 (dd, *J* = 8.4, 2.4 Hz, 1 H), 6.69 (d, *J* = 2.4 Hz, 1 H), 6.48 (dd, *J* = 3.6, 1.6 Hz, 1 H), 5.68-5.55 (m, 1 H), 4.65 (ddd, *J* = 12.8, 4.0, 4.0 Hz, 1 H), 4.60 (dd, *J* = 12.0, 4.4 Hz, 1 H), 4.13-4.09 (m, 1 H), 3.09-3.04 (comp, 4 H), 2.85-2.82 (comp, 2 H), 2.64 (ddd, *J* = 14.8, 10.0, 4.4 Hz, 1 H), 2.58-2.54 (m, 1 H), 2.28 (s, 3 H), 1.79 (ddd, *J* = 14.8, 12.4, 6.0 Hz, 1 H), 1.42 (d, *J* = 6.0 Hz, 3 H); LCMS purity: 91%.



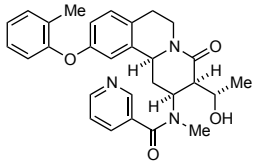
16{1,4}

***N*-(3-(1-Hydroxyethyl)-4-oxo-10-phenyl-2,3,4,6,7,11*b*-hexahydro-1*H*-pyrido[2,1-*a*]isoquinolin-2-yl)-*N*-methylnicotinamide (16**{1,4}**)**. Prepared according to the representative procedure for the formation of amides with the following modification: 2.2 equivalents of Et_3N were used, rather than 1.2 equivalents. Purification: EtOAc/MeOH (90 : 10) with 1% Et_3N. Yield: 81% (38 mg yellow solid). Data: mp 130-132 °C; ^{1}H NMR (400 MHz) δ 8.66-8.64 (comp, 2 H), 7.75 (d, *J* = 7.6 Hz, 1 H), 7.56 (d, *J* = 7.2 Hz, 2 H), 7.50-7.45 (comp, 3 H), 7.40-7.28 (comp, 4 H), 5.77-5.75 (m, 1 H), 4.78-4.76 (m, 1 H), 4.69-4.66 (m, 1 H), 4.35-4.33 (m, 1 H), 4.34-4.32 (m, 1 H), 3.14-3.08 (m, 1 H), 2.97-2.88 (comp, 3 H), 2.85 (s, 3 H), 2.67-2.64 (m, 1 H), 1.86-1.78 (m, 1 H), 1.45 (d, *J* = 6.4 Hz, 3 H); ^{13}C NMR (125 MHz) δ 171.8, 170.4, 150.9, 147.9, 140.4, 140.3, 135.2, 134.8, 133.5, 131.9, 129.3, 128.9, 127.6, 127.0, 126.1, 124.8, 123.3, 64.9, 53.2, 49.8, 46.8, 38.2, 37.0, 33.3, 28.9, 20.5; IR (neat) 3419, 2932, 1634, 1486, 1409, 1073, 765, 733, 700 cm^{-1}; mass spectrum (ESI) *m/z* 456.2280 [$C_{28}H_{31}N_3O_3$ (M+1) requires 456.2287]; LCMS purity: 94%.



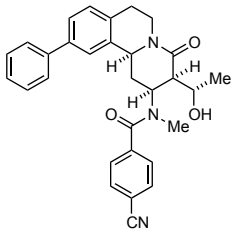
16{5,4}

***N*-(3-(1-Hydroxyethyl)-10-(4-methylpiperazin-1-yl)-4-oxo-2,3,4,6,7,11*b*-hexahydro-1*H*-pyrido[2,1-*a*]isoquinolin-2-yl)-*N*-methylnicotinamide (16**{5,4}**)**. Prepared according to the representative procedure for the formation of amides, with the following modification: 2.2 equivalents of Et_3N were used, rather than 1.2 equivalents. Purification: EtOAc/MeOH (55 : 45) with 1% Et_3N. Yield: 99% (47 mg yellow oil). Data: ^{1}H NMR (400 MHz) δ 8.65-8.63 (comp, 2 H), 7.77-7.75 (m, 1 H), 7.33 (ddd, *J* = 7.6, 4.8, 0.8 Hz, 1 H), 7.10 (d, *J* = 8.0 Hz, 1 H), 6.85 (dd, *J* = 8.4, 2.4 Hz, 1 H), 6.72-6.70 (m, 1 H), 5.76-5.74 (m, 1 H), 4.66-4.62 (comp, 2 H), 4.34-4.32 (m, 1 H), 3.22-3.19 (comp, 4 H), 3.02-2.96 (comp, 2 H), 2.83-2.79 (comp, 5 H), 2.62-2.59 (comp, 5 H), 2.37 (s, 3 H), 1.79-1.76 (m, 1 H), 1.44-1.43 (comp, 3 H); LCMS purity: 94%.



16{6,4}

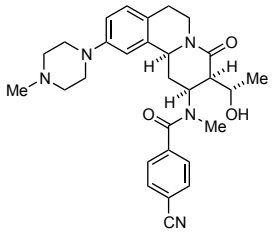
***N*-(3-(1-Hydroxyethyl)-4-oxo-10-(*o*-tolyloxy)-2,3,4,6,7,11*b*-hexahydro-1*H*-pyrido[2,1-*a*]isoquinolin-2-yl)-*N*-methylnicotinamide (16{6,4})**. Prepared according to the representative procedure for the formation of amides, with the following modification: 2.2 equivalents of Et_3N were used, rather than 1.2 equivalents. Purification: EtOAc/MeOH (90 : 10). Yield: 80% (37 mg yellow solid). Data: ^{1}H NMR (400 MHz) δ 8.65-8.64 (comp, 2 H), 7.77-7.75 (m, 1 H), 7.33 (ddd, *J* = 7.6, 4.8, 0.8 Hz, 1 H), 7.29-7.26 (m, 1 H), 7.22-7.18 (m, 1 H), 7.14-7.09 (comp, 2 H), 6.90 (dd, *J* = 8.0, 0.8 Hz, 1H), 6.81-6.79 (m, 1 H), 6.72-6.67 (m, 1 H), 5.73-5.68 (m, 1 H), 4.67-4.64 (m, 1 H), 4.60-4.58 (m, 1 H), 4.32-4.29 (comp, 2 H), 3.05-3.02 (m, 1 H), 2.97-2.93 (m, 1 H), 2.83 (s, 3 H), 2.76-2.70 (m, 1 H), 2.58-2.54 (m, 1 H), 2.42 (s, 3 H), 1.76-1.69 (m, 1 H), 1.42 (d, *J* = 6.0 Hz, 3 H); LCMS purity: 90%.



16{1,5}

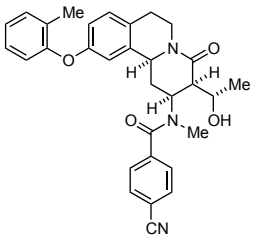
4-Cyano-*N*-(3-(1-hydroxyethyl)-4-oxo-10-phenyl-2,3,4,6,7,11*b*-hexahydro-1*H*-pyrido[2,1-*a*]isoquinolin-2-yl)-*N*-methylbenzamide (16{1,5}). Prepared according to the representative procedure for the formation of amides, with the following modifications: Upon addition of the *p*-cyanobenzoyl chloride to the reaction mixture, the reaction was stirred for 48 h at room temperature rather than for 3 h at 0 °C. After stirring for 24 h at room temperature, an extra 0.5 equivalent of *p*-cyanobenzoyl chloride was added. Purification: EtOAc/MeOH (100 : 0 → 90 : 10). Yield: 74% (31 mg colorless solid). Data: mp 100-101 °C; ^{1}H NMR (400 MHz) δ 7.68 (d, *J* = 8.0 Hz, 2 H), 7.57-7.55 (comp, 2 H), 7.52-7.45 (comp, 6 H), 7.42-7.37 (m, 1 H), 7.30-7.28 (m, 1 H), 5.79-5.75 (m, 1 H), 4.75 (dd, *J* = 12.8, 4.8 Hz, 1 H), 4.70-4.67 (m, 1 H), 4.41-4.38 (m, 1 H), 4.29-4.23 (m, 1 H), 3.16-3.09 (m, 1 H), 2.97-2.91 (comp, 3 H), 2.78 (s, 3 H), 2.65-2.62 (m, 1 H), 1.83-1.76 (m, 1 H), 1.44 (d, *J* = 6.0 Hz, 3 H); ^{13}C NMR (125 MHz) δ 171.0, 169.8, 139.4, 139.4, 139.3, 134.2, 132.4, 131.4, 128.4, 127.9, 126.7, 126.6, 126.0, 125.2, 123.8,

117.1, 112.6, 64.0, 52.2, 48.4, 45.4, 37.3, 36.2, 32.1, 27.9, 19.2; IR (neat) 3430, 2918, 1634, 1410, 1068, 912, 732 cm^{-1}; mass spectrum (CI) *m/z* 480.2289 [$C_{30}H_{30}N_3O_3$ (M+1) requires 480.2287]; LCMS purity: 96%.



16{5,5}

4-Cyano-*N*-(3-(1-hydroxyethyl)-10-(4-methylpiperazin-1-yl)-4-oxo-2,3,4,6,7,11*b*-hexahydro-1*H*-pyrido[2,1-*a*]isoquinolin-2-yl)-*N*-methylbenzamide (16{5,5}). Prepared according to the representative procedure for the formation of amides, with the following modifications: Upon addition of the *p*-cyanobenzoyl chloride to the reaction mixture, the reaction was stirred for 48 h at room temperature rather than for 3 h at 0 °C. After stirring for 24 h at room temperature, an extra 0.5 equivalent of *p*-cyanobenzoyl chloride was added. Purification: EtOAc/MeOH (60 : 40) with 1% Et_3N. Yield: 89% (32 mg colorless solid). Data: ^{1}H NMR (400 MHz) δ 7.68 (d, *J* = 8.0 Hz, 2 H), 7.51 (d, *J* = 8.0 Hz, 2 H), 7.09 (d, *J* = 8.4 Hz, 1 H), 6.84 (dd, *J* = 8.4, 2.0 Hz, 1 H), 6.70 (d, *J* = 2.0 Hz, 1 H), 5.77-5.72 (m, 1 H), 4.65-4.59 (comp, 2 H), 4.40-4.37 (m, 1 H), 3.28-3.22 (comp, 4 H), 3.04-3.00 (m, 1 H), 2.86-2.75 (comp, 6 H), 2.69-2.63 (comp, 4 H), 2.60-2.57 (m, 1 H), 2.42 (s, 3 H), 1.74-1.66 (m, 1H), 1.41 (d, *J* = 6.4 Hz, 3 H); LCMS purity: 95%.

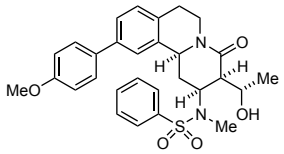


16{6,5}

4-Cyano-*N*-(3-(1-hydroxyethyl)-4-oxo-10-(*o*-tolyloxy)-2,3,4,6,7,11*b*-hexahydro-1*H*-pyrido[2,1-*a*]isoquinolin-2-yl)-*N*-methylbenzamide (16{6,5}). Prepared according to the representative procedure for the formation of amides, with the following modifications: Upon addition of the *p*-cyanobenzoyl chloride to the reaction mixture, the reaction was stirred for 48 h at room temperature rather than for 3 h at 0 °C. After stirring for 24 h at room temperature, an extra 0.5 equivalent of *p*-cyanobenzoyl chloride was added. Purification: EtOAc/MeOH (90 :

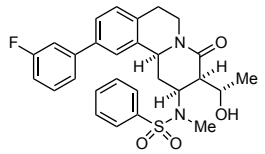
10). Yield: 88% (32 mg colorless solid). Data: mp 126-127 °C; ^{1}H NMR (500 MHz) δ 7.68 (d, *J* = 8.5 Hz, 2 H), 7.51 (d, *J* = 8.5 Hz, 1 H), 7.29-7.27 (m, 1 H), 7.21-7.18 (m, 1 H), 7.14-7.10 (comp, 2 H), 6.90 (d, *J* = 8.0 Hz, 1 H), 6.80 (dd, *J* = 8.0, 2.5 Hz, 1 H), 6.67 (d, *J* = 2.5 Hz, 1 H), 5.72-5.68 (m, 1 H), 4.67-4.62 (m, 1 H), 4.58-4.56 (m, 1 H), 4.39-4.35 (m, 1 H), 4.21-4.20 (m, 1 H), 3.08-3.03 (m, 1 H), 2.86-2.80 (comp, 2 H), 2.76 (s, 3 H), 2.75-2.70 (m, 1 H), 2.56-2.54 (m, 1 H), 2.24 (s, 3 H), 1.72-1.66 (m, 1 H), 1.40 (d, *J* = 6.5 Hz, 3 H); ^{13}C NMR (125 MHz) δ 172.0, 170.7, 157.2, 154.0, 140.4, 136.1, 132.4, 131.7, 130.2, 130.1, 128.1, 127.6, 127.3, 124.5, 119.9, 118.1, 116.4, 114.3, 113.6, 65.0, 53.0, 49.3, 46.2, 38.4, 37.1, 33.1, 28.4, 20.1, 16.2; IR (neat) 3431, 2932, 2231, 1638, 1488, 1410, 1251, 731 cm^{-1}; mass spectrum (ESI) *m/z* 510.2386 [$C_{31}H_{32}N_3O_4$ (M+1) requires 510.2393]; LCMS purity: 92%.

Representative procedure for the formation of sulfonamides:



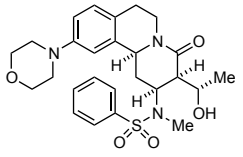
16{2,6}

***N*-(3-(1-Hydroxyethyl)-10-(4-methoxyphenyl)-4-oxo-2,3,4,6,7,11*b*-hexahydro-1*H*-pyrido[2,1-*a*]isoquinolin-2-yl)-*N*-methylbenzenesulfonamide (16{2,6})**. Phenyl sulfonyl chloride (**15**{6}) (22 mg, 16 μL, 0.12 mmol) was added to a solution of 1,3-amino alcohol **14**{2} (43 mg, 0.11 mmol) and Et_3N (14 mg, 19 μL, 0.14 mmol) in anhydrous CH_2Cl_2 (0.6 mL), and was stirred for 2 h at room temperature. Saturated $NaHCO_3$ (5 mL) and CH_2Cl_2 (5 mL) were added, and the layers were separated. The aqueous layer was extracted with CH_2Cl_2 (2 × 5 mL), and the organic layer was dried (Na_2SO_4), filtered, and concentrated under reduced pressure. The resultant yellow oil was purified by flash column chromatography eluting with EtOAc to give 38 mg (64%) of **16**{2,6} as a yellow solid: ^{1}H NMR (400 MHz) δ 7.80-7.77 (comp, 2 H), 7.54-7.46 (comp, 3 H), 7.43 (d, *J* = 8.8 Hz, 2 H), 7.38 (dd, *J* = 7.6, 1.6 Hz, 1 H), 7.19 (d, *J* = 7.6 Hz, 1 H), 7.10 (d, *J* = 1.6 Hz, 1 H), 7.00 (d, *J* = 8.8 Hz, 2 H), 5.03-4.97 (m, 1 H), 4.60-4.54 (comp, 2 H), 4.31 (ddd, *J* = 8.4, 6.0, 4.4 Hz, 1 H), 3.87 (s, 3 H), 3.71 (d, *J* = 4.0 Hz, 1 H), 3.07-3.01 (m, 1 H), 2.82-2.79 (m, 1 H), 2.75 (s, 3 H), 2.51 (dd, *J* = 8.4, 6.8 Hz, 1 H), 2.16 (ddd, *J* = 14.8, 10.0, 4.4 Hz, 1 H), 1.50 (d, *J* = 6.0 Hz, 3 H), 1.33-1.24 (m, 1 H); LCMS purity: 92%.



16{3,6}

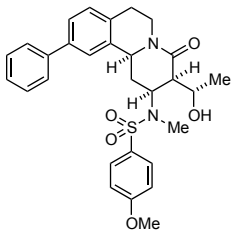
***N*-(10-(3-Fluorophenyl)-3-(1-hydroxyethyl)-4-oxo-2,3,4,6,7,11*b*-hexahydro-1*H*-pyrido[2,1-*a*]isoquinolin-2-yl)-*N*-methylbenzenesulfonamide (16**{3,6}**)**. Prepared according to the representative procedure for the formation of sulfonamides. Purification: EtOAc. Yield: 92% (64 mg yellow solid). Data: mp 194-195.5^{o} C (colorless needles from 1 : 1 EtOH : H_2O); ^{1}H NMR (400 MHz) δ 8.07-8.04 (m, 1 H), 7.82-7.79 (comp, 2 H), 7.77-7.74 (m, 1 H), 7.66-7.63 (m, 1 H), 7.58-7.54 (m, 1 H), 7.52-7.47 (m, 1 H), 7.42-7.39 (m, 1 H), 7.29-7.26 (m, 1 H), 7.17 (ddd, *J* = 10.4, 2.4, 2.0 Hz, 1 H), 7.11 (d, *J* = 1.2 Hz, 1 H), 7.08 (dddd, *J* = 8.4, 8.4, 2.8, 1.2 Hz, 1 H), 5.00 (ddd, *J* = 10.0, 5.6, 5.6 Hz, 1 H), 4.59-4.50 (comp, 2 H), 4.33-4.30 (m, 1 H), 3.69 (d, *J* = 3.2 Hz, 1 H), 3.09 (ddd, *J* = 14.0, 10.0, 4.4 Hz, 1 H), 2.84-2.79 (comp, 2 H), 2.75 (s, 3 H), 2.54-2.50 (m, 1 H), 2.17 (ddd, *J* = 14.4, 10.0, 4.4 Hz, 1 H), 1.50 (d, *J* = 6.4 Hz, 3 H), 1.28 (ddd, *J* = 14.8, 12.0, 5.6 Hz,1 H); ^{13}C NMR (100 MHz) δ 170.6, 163.2 ($J_{C\text{-}F}$ = 244.7 Hz), 142.6 ($J_{C\text{-}F}$ = 7.5 Hz), 138.9, 135.3, 134.5, 133.2, 130.4 ($J_{C\text{-}F}$ = 8.1 Hz), 129.7, 129.5, 129.4, 127.0 ($J_{C\text{-}F}$ = 5.9 Hz), 126.0, 124.6, 122.5, 114.4 ($J_{C\text{-}F}$ = 20.8 Hz), 113.8 ($J_{C\text{-}F}$ = 22.3 Hz), 64.5, 52.9, 51.5, 50.8, 38.1, 34.6, 29.6, 29.0, 21.7; IR (neat) 3517, 2934, 1654, 1421, 1331, 1159, 736 cm^{-1}; mass spectrum (CI) *m/z* 509.1917 [$C_{28}H_{30}FN_2O_4S$ (M+1) requires 509.1910]; LCMS purity: 92%.



16{4,6}

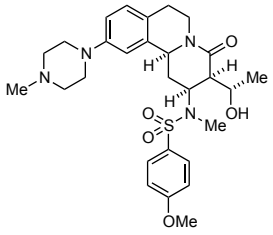
***N*-(3-(1-Hydroxyethyl)-10-morpholino-4-oxo-2,3,4,6,7,11*b*-hexahydro-1*H*-pyrido[2,1-*a*]isoquinolin-2-yl)-*N*-methylbenzenesulfonamide (16**{4,6}**)**. Prepared according to the representative procedure for the formation of sulfonamides. Purification: EtOAc. Yield: 61% (30 mg yellow solid). Data: ^{1}H NMR (400 MHz) δ 7.80-7.78 (comp, 2 H), 7.56-7.55 (comp, 3 H), 7.05 (d, *J* = 8.4 Hz, 1 H), 6.78 (dd, *J* = 8.4, 2.4 Hz, 1 H), 6.46 (d, *J* = 2.4 Hz, 1 H), 4.99 (ddd, *J* = 10.0, 5.6, 5.6 Hz, 1 H), 4.55 (ddd, *J* = 12.4, 3.6, 3.6 Hz, 1 H), 4.45 (dd, *J* = 12.0, 4.4 Hz, 1 H), 4.29 (ddd, *J* = 8.4, 6.0, 4.4 Hz, 1 H), 3.88-3.88 (comp, 4 H), 3.68 (d, *J* = 4.4 Hz, 1 H), 3.09-3.06 (comp, 4 H), 2.98-2.91 (m, 1 H), 2.73 (s, 3 H), 2.71-2.69 (m, 1 H), 2.47 (dd, *J* = 8.0, 7.2 Hz,

1 H), 2.12 (ddd, *J* = 14.4, 10.0, 4.0 Hz, 1 H), 1.49 (d, *J* = 5.6 Hz, 3 H), 1.27 (ddd, *J* = 14.4, 12.0, 5.2 Hz, 1 H).



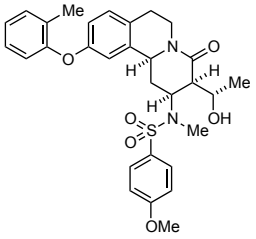
16{1,7}

***N*-(3-(1-Hydroxyethyl)-4-oxo-10-phenyl-2,3,4,6,7,11*b*-hexahydro-1*H*-pyrido[2,1-*a*]isoquinolin-2-yl)-4-methoxy-*N*-methylbenzenesulfonamide (16**{1,7}**)**. Prepared according to the representative procedure for the formation of sulfonamides. Purification: pentane/EtOAc (45 : 55). Yield: 78% (36 mg colorless solid). Data: ^{1}H NMR (400 MHz) δ 7.73-7.69 (comp, 2 H), 7.52-7.36 (comp, 6 H), 7.23 (d, *J* = 8.0 Hz, 1 H), 7.16 (d, *J* = 1.2 Hz, 1 H), 6.95-6.91 (comp, 2 H), 5.01-4.95 (m, 1 H), 4.60-4.52 (comp, 2 H), 4.33-4.29 (m, 1 H), 3.76 (s, 3 H), 3.76-3.72 (m, 1 H), 3.08 (ddd, *J* = 14.4, 10.0, 4.4 Hz, 1 H), 2.86-2.79 (comp, 2 H), 2.72 (s, 3 H), 2.53-2.49 (m, 1 H), 2.17 (ddd, *J* = 14.4, 10.0, 4.4 Hz, 1 H), 1.50 (d, *J* = 6.0 Hz, 3 H), 1.30 (ddd, *J* = 14.4, 12.0, 5.2 Hz, 1 H); LCMS purity: 96%.



16{5,7}

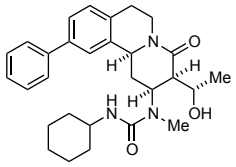
***N*-(3-(1-Hydroxyethyl)-10-(4-methylpiperazin-1-yl)-4-oxo-2,3,4,6,7,11*b*-hexahydro-1*H*-pyrido[2,1-*a*]isoquinolin-2-yl)-4-methoxy-*N*-methylbenzenesulfonamide (16**{5,7}**)**. Prepared according to the representative procedure for the formation of sulfonamides. Purification: EtOAc/MeOH (65 : 35) with 1% Et_3N. Yield: 55% (25 mg yellow solid). Data: ^{1}H NMR (300 MHz) δ 7.69 (d, *J* = 9.0 Hz, 2 H), 7.03 (d, *J* = 8.4 Hz, 1 H), 6.92 (d, *J* = 9.0 Hz, 2 H), 6.78 (dd, *J* = 8.4, 2.1 Hz, 1 H), 6.50 (d, *J* = 2.1 Hz, 1 H), 5.00-4.93 (m, 1 H), 4.55-4.42 (comp, 2 H), 4.33-4.24 (m, 1 H), 3.83 (s, 3 H), 3.14-3.12 (comp, 4 H), 2.99-2.90 (m 1 H), 2.69 (s, 3 H), 2.69-2.60 (comp, 2 H), 2.59-2.57 (comp, 4 H), 2.48-2.43 (m, 1 H), 2.37 (s, 3 H), 2.13 (ddd, *J* = 14.4, 10.2, 4.5 Hz, 1 H), 1.47 (d, *J* = 6.0 Hz, 3 H), 1.32-1.21 (m, 1 H); LCMS purity: 98%.



16{6,7}

***N*-(3-(1-Hydroxyethyl)-4-oxo-10-(*o*-tolyloxy)-2,3,4,6,7,11*b*-hexahydro-1*H*-pyrido[2,1-*a*]isoquinolin-2-yl)-4-methoxy-*N*-methylbenzenesulfonamide (16**{6,7}**)**. Prepared according to the representative procedure for the formation of sulfonamides. Purification: pentane/EtOAc (3 : 7). Yield: 75% (34 mg colorless solid). Data: mp 198-199 °C; ^{1}H NMR (500 MHz) δ 7.68 (d, *J* = 9.0 Hz, 2 H), 7.26-7.25 (m, 1 H), 7.18-7.15 (m, 1 H), 7.08 (ddd, *J* = 7.5, 7.5, 1.0 Hz, 1 H), 7.04 (d, *J* = 8.0 Hz, 1 H), 6.92 (d, *J* = 9.0 Hz, 2 H), 6.80 (dd, *J* = 8.0, 1.0 Hz, 1 H), 6.70 (dd, *J* = 8.5, 2.5 Hz, 1 H), 6.49 (d, *J* = 2.5 Hz, 1 H), 4.89 (ddd, *J* = 10.0, 6.5, 5.5 Hz, 1 H), 4.54 (ddd, *J* = 13.0, 4.5, 4.5 Hz, 1 H), 4.40 (dd, *J* = 13.0, 4.5 Hz, 1 H), 4.26 (ddd, *J* = 8.5, 6.0, 4.0 Hz, 1 H), 3.83 (s, 3 H), 3.65 (d, *J* = 4.0 Hz, 1 H), 2.99-2.93 (m, 1 H), 2.76-2.65 (comp, 2 H), 2.68 (s, 3 H), 2.42-2.39 (m, 1 H), 2.18 (s, 3 H), 2.01 (ddd, *J* = 14.0, 10.0, 4.0 Hz, 1 H), 1.45 (d, *J* = 6.0 Hz, 3 H), 1.25-1.19 (m, 1 H);^{13}C NMR (125 MHz) δ 170.6, 163.2, 156.8, 154.1, 136.1, 131.6, 130.2, 130.0, 129.8, 129.2, 128.4, 127.2, 124.4, 119.5, 116.2, 114.6, 114.5, 64.5, 55.6, 52.8, 51.5, 50.5, 38.2, 34.7, 29.5, 28.5, 21.6, 16.1; IR (neat) 3519, 2933, 1650, 1596, 1497, 1418, 1332, 1257, 1185, 1153, 1092, 730 cm^{-1}; mass spectrum (CI) *m/z* 551.2217 [$C_{30}H_{35}N_2O_6S$ (M+1) requires 551.2216]; LCMS purity: 97%.

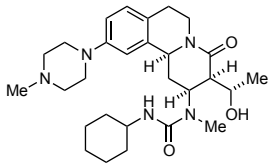
Representative procedure for the formation of ureas:



16{1,8}

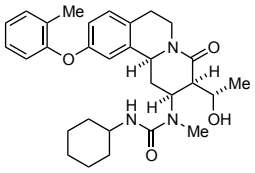
3-Cyclohexyl-1-(3-(1-hydroxyethyl)-4-oxo-10-phenyl-2,3,4,6,7,11*b*-hexahydro-1*H*-pyrido[2,1-*a*]isoquinolin-2-yl)-1-methylurea (16{1,8}**)**. A mixture of cyclohexyl isocyanate (**15**{8}) (12 mg, 12 μL, 0.094 mmol) and1,3-amino alcohol **14**{1} (30 mg, 0.086 mmol) in CH_2Cl_2 (0.5 mL) was stirred for 1.5 h at room temperature. The solvent was removed *in vacuo*,

and the resultant colorless residue was purified by flash column chromatography eluting with pentane/EtOAc (1 : 1) to give 32 mg (72%) of urea **16**{1,8} as a colorless solid: mp 120-121 °C; ^{1}H NMR (500 MHz) δ 7.57-7.55 (comp, 2 H), 7.47-7.44 (comp, 3 H), 7.39-7.36 (comp, 2 H), 7.28 (d, *J* = 8.0 Hz, 1 H), 5.50-5.46 (m, 1 H), 5.09 (br s, 1 H), 4.73 (dd, *J* = 12.0, 5.0 Hz, 1 H), 4.59 (ddd, *J* = 13.0, 4.0, 4.0 Hz, 1 H), 4.28 (d, *J* = 7.0 Hz, 1 H), 3.99-3.97 (m, 1 H), 3.65-3.61 (m, 1 H), 3.20-3.14 (m, 1 H), 2.92-2.89 (comp, 2 H), 2.77 (ddd, *J* = 15.5, 10.5, 5.0 Hz, 1 H), 2.68 (s, 3 H), 2.44 (dd, *J* = 9.0, 6.5 Hz, 1 H), 1.97-1.89 (m, 1 H), 1.75-1.59 (comp, 6 H), 1.47 (d, *J* = 6.0 Hz, 3 H), 1.39-1.31 (comp, 2 H), 1.19-1.03 (comp, 2 H); ^{13}C NMR (125 MHz) δ 171.5, 158.5, 140.4, 140.3, 135.7, 133.6, 129.2, 128.9, 127.6, 127.0, 126.0, 125.0, 64.3, 53.1, 52.7, 49.8, 47.7, 37.9, 37.3, 33.9, 29.5, 29.0, 25.6, 25.0, 21.3; IR (neat) 3332, 2931, 2855, 1650, 1531, 1422 cm^{-1}; mass spectrum (ESI) *m/z* 498.2728 [$C_{29}H_{37}N_3O_3Na$ (M + Na) requires 498.2733]; LCMS purity: 92%.



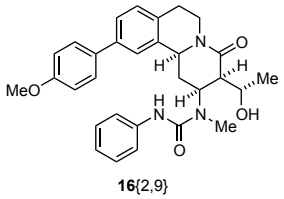
16{5,8}

3-Cyclohexyl-1-(3-(1-hydroxyethyl)-10-(4-methylpiperazin-1-yl)-4-oxo-2,3,4,6,7,11*b*-hexahydro-1*H*-pyrido[2,1-*a*]isoquinolin-2-yl)-1-methylurea (16{5,8}**)**. Prepared according to the representative procedure for the formation of ureas. Purification: EtOAc/MeOH (7 : 3). Yield: 62% (27 mg colorless solid). Data: ^{1}H NMR (400 MHz) δ 7.10 (d, *J* = 8.0 Hz, 1 H), 6.83 (dd, *J* = 8.0, 2.4 Hz, 1 H), 6.69 (d, *J* = 2.4 Hz, 1 H), 5.48-5.43 (m, 1 H), 5.08 (br s, 1 H), 4.61-4.53 (comp, 2 H), 4.27 (d, *J* = 8.0 Hz, 1 H), 3.98-3.95 (m, 1 H), 3.65-3.59 (m, 1 H), 3.34-3.24 (comp, 4 H), 3.10-3.04 (m, 1 H), 2.79-2.68 (comp, 6 H), 2.66 (s, 3 H), 2.53-2.44 (comp, 3 H), 2.39 (dd, *J* = 8.4, 5.6 Hz, 1 H), 1.97-1.88 (comp, 2 H), 1.72-1.60 (comp, 4 H), 1.45 (d, *J* = 6.0 Hz, 3 H), 1.37-1.05 (comp, 6 H); LCMS purity: 98%.



16{6,8}

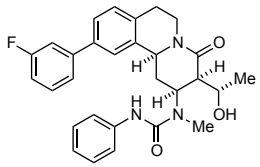
3-Cyclohexyl-1-(3-(1-hydroxyethyl)-4-oxo-10-(*o*-tolyloxy)-2,3,4,6,7,11*b*-hexahydro-1*H*-pyrido[2,1-*a*]isoquinolin-2-yl)-1-methylurea (16{6,8}). Prepared according to the representative procedure for the formation of ureas. Purification: pentane/EtOAc (1 : 1). Yield: 78% (31 mg colorless solid). Data: ^{1}H NMR (400 MHz) δ 7.28-7.26 (m, 1 H), 7.20-7.08 (comp, 3 H), 6.88 (d, *J* = 8.0 Hz, 1 H), 6.78 (dd, *J* = 8.0, 1.2 Hz, 1 H), 6.67 (d, *J* = 1.2 Hz, 1 H), 5.45-5.41 (m, 1 H), 5.02 (br s, 1 H), 4.58-4.54 (comp, 2 H), 4.27 (d, *J* = 8.4 Hz, 1 H), 3.97-3.94 (m, 1 H), 3.64-3.62 (m, 1 H), 3.15-3.08 (m, 1 H), 2.83-2.81 (comp, 2 H), 2.66 (s, 3 H), 2.61 (ddd, *J* = 14.8, 10.4, 4.4 Hz, 1 H), 2.38-2.34 (m, 1 H), 2.23 (s, 3 H), 1.96-1.89 (comp, 2 H), 1.68-1.59 (comp, 3 H), 1.44 (d, *J* = 6.0 Hz, 3 H), 1.37-1.26 (comp, 3 H), 1.15-1.04 (comp, 3 H); LCMS purity: 97%.



16{2,9}

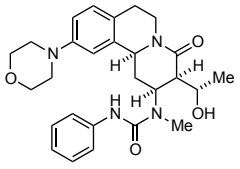
1-(3-(1-Hydroxyethyl)-10-(4-methoxyphenyl)-4-oxo-2,3,4,6,7,11*b*-hexahydro-1*H*-pyrido[2,1-*a*]isoquinolin-2-yl)-1-methyl-3-phenylurea (16{2,9}). Prepared according to the representative procedure for the formation of ureas, with the following modification: Instead of removing the CH_2Cl_2 *in vacuo* at the end of the reaction, 2 mL of hexanes were added, resulting in a colorless precipitate. The colorless powder was collected via filtration, and was sufficiently pure. Yield: 69% (14 mg colorless powder). Data: mp 215-216 °C (colorless crystals from 1 : 1 EtOH : H_2O); ^{1}H NMR (600 MHz) δ 7.49 (d, *J* = 9.0 Hz, 2 H), 7.43 (dd, *J* = 8.4, 2.4 Hz, 1 H), 7.34-7.32 (comp, 3 H), 7.30-7.26 (comp, 3 H), 7.06-7.03 (m, 1 H), 6.99 (d, *J* = 9.0 Hz, 2 H), 6.58 (br s, 1 H), 5.50-5.47 (m, 1 H), 4.73 (dd, *J* = 12.0, 4.2 Hz, 1 H), 4.63 (ddd, *J* = 12.6, 4.2, 4.2 Hz, 1 H), 4.42 (br s, 1 H), 4.19-4.14 (m, 1 H), 3.86 (s, 3 H), 3.19-3.14 (m, 1 H), 2.93-2.89 (comp, 2 H), 2.86 (s, 3 H), 2.80 (ddd, *J* = 15.0, 10.2, 4.8 Hz, 1 H), 2.54-2.52 (m, 1 H), 1.77 (ddd, *J* = 14.4,

12.0, 4.8 Hz, 1 H), 1.48 (d, J = 6.0 Hz, 3 H); ^{13}C NMR (150 MHz) δ 171.4, 159.4, 156.8, 140.0, 138.5, 135.4, 133.0, 132.9, 129.2, 128.9, 128.0, 125.7, 124.5, 123.6, 120.4, 114.4, 64.8, 55.4, 53.2, 51.7, 48.2, 38.1, 37.3, 29.9, 28.9, 21.4; IR (neat) 3319, 2928, 1645, 1610, 1536, 1496, 1443, 1246 cm^{-1}; mass spectrum (ESI) *m/z* 500.2543 [$C_{30}H_{34}N_3O_4$ (M+1) requires 500.2549]; LCMS purity: 93%.



16{3,9}

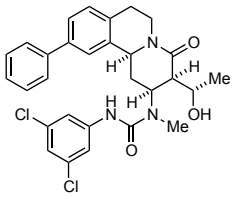
1-(10-(3-Fluorophenyl)-3-(1-hydroxyethyl)-4-oxo-2,3,4,6,7,11*b*-hexahydro-1*H*-pyrido[2,1-*a*]isoquinolin-2-yl)-1-methyl-3-phenylurea (16{3,9}**)**. Prepared according to the representative procedure for the formation of ureas, with the following modification: Instead of removing the CH_2Cl_2 *in vacuo* at the end of the reaction, 2 mL of hexanes were added, resulting in a colorless precipitate. The colorless powder was collected via filtration, and was sufficiently pure. Yield: 86% (34 mg colorless powder). Data: ^{1}H NMR (400 MHz) δ 7.47 (dd, J = 8.0, 2.0 Hz, 1 H), 7.43-7.39 (m, 1 H), 7.36-7.29 (comp, 7 H), 7.26-7.24 (m, 1 H), 7.07-7.05 (comp, 2 H), 6.56 (br s, 1 H), 5.53-5.49 (m, 1 H), 4.75 (dd, J = 11.6, 4.0 Hz, 1 H), 4.65 (ddd, J = 13.2, 4.4, 4.4 Hz, 1 H), 4.42 (br s, 1 H), 4.18-4.14 (m, 1 H), 3.20-3.14 (m, 1 H), 2.95-2.93 (comp, 2 H), 2.87 (s, 3 H), 2.81 (ddd, J = 15.2, 10.4, 4.4 Hz, 1 H), 2.56-2.52 (m, 1 H), 1.78 (ddd, J = 15.2, 12.8, 5.2 Hz, 1 H), 1.48 (d, J = 6.0 Hz, 3 H).



16{4,9}

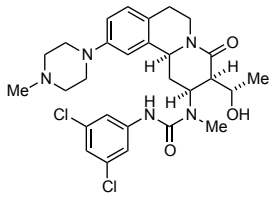
1-(3-(1-Hydroxyethyl)-10-morpholino-4-oxo-2,3,4,6,7,11*b*-hexahydro-1*H*-pyrido[2,1-*a*]isoquinolin-2-yl)-1-methyl-3-phenylurea (16{4,9}**)**. Prepared according to the representative procedure for the formation of ureas, with the following modification: Instead of removing the CH_2Cl_2 *in vacuo* at the end of the reaction, 2 mL of hexanes were added, resulting in a colorless precipitate. The colorless powder was collected via filtration, and was sufficiently pure. Yield: 74% (24 mg colorless powder). Data: ^{1}H NMR (400 MHz) δ 7.36-7.29 (comp, 4 H), 7.12 (d, J = 8.4 Hz, 1 H), 7.08-7.04 (m, 1 H), 6.83 (dd, J = 8.4, 2.4 Hz, 1 H), 6.67 (d, J = 2.4 Hz, 1 H), 6.56

(br s, 1 H), 5.50-5.45 (m, 1 H), 4.64-4.59 (comp, 2 H), 4.45 (br s, 1 H), 4.17-4.12 (m, 1 H), 3.88-3.86 (comp, 4 H), 3.15-3.12 (comp, 4 H), 3.11-3.04 (m, 1 H), 2.85 (s, 3 H), 2.81-2.79 (comp, 2 H), 2.72 (ddd, *J* = 14.8, 10.4, 4.4 Hz, 1 H), 2.51-2.47 (m, 1 H), 1.71 (ddd, *J* = 14.8, 12.0, 5.2 Hz, 1 H), 1.47 (d, *J* = 6.0 Hz, 3 H).



16{1,10}

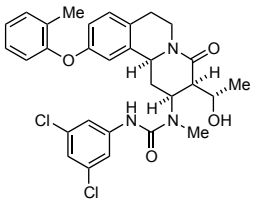
3-(3,5-Dichlorophenyl)-1-(3-(1-hydroxyethyl)-4-oxo-10-phenyl-2,3,4,6,7,11*b*-hexahydro-1*H*-pyrido[2,1-*a*]isoquinolin-2-yl)-1-methylurea (16{1,10}**)**. Prepared according to the representative procedure for the formation of ureas. Purification: pentane/EtOAc (3 : 7). Yield: 73% (37 mg colorless powder). Data: mp 118-119 °C; ^{1}H NMR (500 MHz) δ 7.56-7.54 (comp, 2 H), 7.49-7.44 (comp, 3 H), 7.38-7.35 (comp, 2 H), 7.30-7.29 (comp, 3 H), 6.99-6.98 (m, 1 H), 5.37-5.31 (m, 1 H), 4.73 (dd, *J* = 12.0, 4.0 Hz, 1 H), 4.63 (ddd, *J* = 13.0, 4.0, 4.0 Hz, 1 H), 4.22-4.18 (m, 1 H), 3.85 (br s, 1 H), 3.19-3.13 (m, 1 H), 2.93-2.90 (comp, 2 H), 2.81 (s, 3 H), 2.77 (ddd, *J* = 15.0, 10.5, 4.5 Hz, 1 H), 2.56-2.53 (m, 1 H), 1.77 (ddd, *J* = 14.5, 12.0, 5.0 Hz, 1 H), 1.50 (d, *J* = 6.0 Hz, 3 H); ^{13}C NMR (125 MHz) δ 171.1, 156.1, 141.0, 140.4, 140.3, 135.2, 135.0, 133.6, 129.3, 128.9, 127.6, 127.0, 126.2, 124.9, 122.9, 117.9, 65.2, 53.1, 51.1, 48.8, 38.2, 37.2, 29.8, 28.9, 21.8,; IR (neat) 3332, 2933, 1645, 1586, 1486, 1414, 1325 cm^{-1}; mass spectrum (ESI) *m/z* 538.1657 [$C_{29}H_{29}N_3O_3{}^{35}Cl_2$ (M+1) requires 538.1664]; LCMS purity: 98%.



16{5,10}

3-(3,5-Dichlorophenyl)-1-(3-(1-hydroxyethyl)-10-(4-methylpiperazin-1-yl)-4-oxo-2,3,4,6,7,11*b*-hexahydro-1*H*-pyrido[2,1-*a*]isoquinolin-2-yl)-1-methylurea (16{5,10}**)**. Prepared according to the representative procedure for the formation of ureas. Purification: EtOAc/MeOH (6 : 4) with 1% Et_3N. Yield: 61% (30 mg colorless powder). Data: ^{1}H NMR (400 MHz) δ 7.28 (d, *J* = 1.6 Hz, 1 H), 7.12-7.04 (comp, 2 H), 7.00-6.98 (m, 1 H), 6.85-6.80 (m, 1 H), 6.67 (d, *J* = 2.4 Hz, 1 H), 5.34-5.29 (m, 1 H), 4.64-4.57 (comp, 2 H), 4.23-4.16 (m, 1 H), 3.86 (br

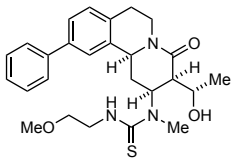
s, 1 H), 3.19-3.17 (comp, 4 H), 3.09-3.03 (m, 1 H), 2.82-2.67 (comp, 6 H), 2.60-2.56 (comp, 4 H), 2.52-2.48 (m, 1 H), 2.36 (s, 3 H), 1.75-1.67 (comp, 2 H), 1.49 (d, *J* = 6.0 Hz, 3 H); LCMS purity: 98%.



16{6,10}

3-(3,5-Dichlorophenyl)-1-(3-(1-hydroxyethyl)-4-oxo-10-(*o*-tolyloxy)-2,3,4,6,7,11*b*-hexahydro-1*H*-pyrido[2,1-*a*]isoquinolin-2-yl)-1-methylurea (16{6,10}**)**. Prepared according to the representative procedure for the formation of ureas. Purification: pentane/EtOAc (3 : 7). Yield: 48% (24 mg colorless powder). Data: ^{1}H NMR (400 MHz) δ 7.28-7.25 (comp, 3 H), 7.20-7.07 (comp, 3 H), 6.98-6.97 (m, 1 H), 6.88 (d, *J* = 8.0 Hz, 1 H), 6.81-6.79 (m, 1 H), 6.65-6.64 (m, 1 H), 5.30-5.22 (m, 1 H), 4.61-4.53 (comp, 2 H), 4.20-4.15 (m, 1 H), 3.78 (br s, 1 H), 3.12-3.05 (m, 1 H), 2.84-2.82 (comp, 2 H), 2.78 (s, 3 H), 2.60 (ddd, *J* = 14.0, 10.0, 3.6 Hz, 1 H), 2.47-2.44 (m, 1 H), 2.22 (s, 3 H), 1.73-1.65 (m, 1 H), 1.47 (d, *J* = 6.0 Hz, 3 H); LCMS purity: 98%.

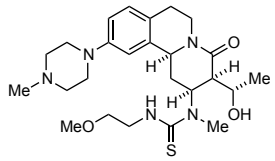
Representative procedure for the formation of thioureas:



16{1,11}

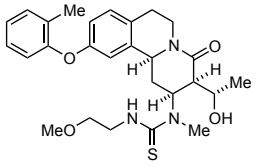
1-(3-(1-Hydroxyethyl)-4-oxo-10-phenyl-2,3,4,6,7,11*b*-hexahydro-1*H*-pyrido[2,1-*a*]isoquinolin-2-yl)-3-(2-methoxyethyl)-1-methylthiourea (16{1,11}**)**. A mixture of 1-isothiocyanato-2-methoxyethane (**15**{11}) (12 mg, 11 µL, 0.103 mmol) and 1,3-amino alcohol **14**{1} (30 mg, 0.086 mmol) in CH_2Cl_2 (0.5 mL) was stirred for 1.5 h at room temperature. The solvent was removed *in vacuo*, and the resultant colorless residue was purified by flash column chromatography eluting with EtOAc/MeOH (100 : 0 → 90 : 10) to give 17 mg (42%) of **16**{1,11} as a yellow solid: mp 151.5-153 °C; ^{1}H NMR (600 MHz) δ 7.55-7.53 (comp, 2 H), 7.47-7.43 (comp, 3 H), 7.38-7.35 (comp, 2 H), 7.27 (d, *J* = 8.4 Hz, 1 H), 6.59-6.56 (m, 1 H), 6.00 (br s, 1 H), 4.71 (dd, *J* = 12.0, 3.6 Hz, 1 H), 4.66 (ddd, *J* = 13.2, 4.2, 4.2 Hz, 1 H), 4.13-4.07 (comp, 2 H), 3.90-3.79 (comp, 2 H), 3.54-3.53 (m, 2 H), 3.34 (s, 3 H), 3.17-3.12 (m, 1 H), 2.92-

2.84 (comp, 2 H), 2.87 (s, 3 H), 2.67-2.66 (m, 1 H), 1.63-1.57 (comp, 2 H), 1.45 (d, *J* = 6.6 Hz, 3 H); ^{13}C NMR (150 MHz) δ 182.3, 171.7, 140.4, 140.3, 135.3, 133.6, 129.2, 128.9, 127.6, 127.0, 126.0, 125.0, 70.6, 65.2, 58.8, 53.6, 53.4, 50.8, 46.1, 38.3, 37.5, 31.6, 29.0, 21.0; IR (neat) 3332, 2931, 1644, 1530, 1486, 1425, 1328, 1187, 1101, 764, 734, 699 cm^{-1}; mass spectrum (CI) *m/z* 468.2320 [$C_{26}H_{34}N_3O_3S$ (M+1) requires 468.2321]; LCMS purity: 98%.



16{5,11}

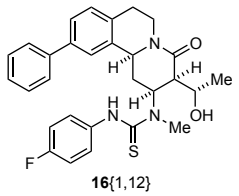
1-(3-(1-Hydroxyethyl)-10-(4-methylpiperazin-1-yl)-4-oxo-2,3,4,6,7,11*b*-hexahydro-1*H*-pyrido[2,1-*a*]isoquinolin-2-yl)-3-(2-methoxyethyl)-1-methylthiourea (16{5,11}**)**. Prepared according to the representative procedure for the formation of thioureas. Purification: EtOAc/MeOH (65 : 35) with 1% Et_3N. Yield: 65% (43 mg yellow oil). Data: ^{1}H NMR (400 MHz) δ 7.07 (d, *J* = 8.4 Hz, 1 H), 6.82 (dd, *J* = 8.4, 2.4 Hz, 1 H), 6.68 (d, *J* = 2.4 Hz, 1 H), 6.58-6.53 (m, 1 H), 6.00-5.96 (m, 1 H), 4.64-4.55 (comp, 2 H), 4.13-4.06 (comp, 2 H), 3.91-3.78 (comp, 2 H), 3.55-3.53 (m, 2 H), 3.35 (s, 3 H), 3.20-3.17 (comp, 4 H), 3.08-3.02 (m, 1 H), 2.85 (s, 3 H), 2.82-2.75 (comp, 3 H), 2.62-2.58 (comp, 5 H), 2.37 (s, 3 H), 1.52 (ddd, *J* = 8.4, 6.4, 3.2 Hz, 1 H), 1.43 (d, *J* = 6.4 Hz, 3 H); LCMS purity: 97%.



16{6,11}

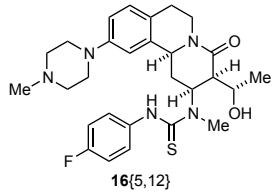
1-(3-(1-Hydroxyethyl)-4-oxo-10-(*o*-tolyloxy)-2,3,4,6,7,11*b*-hexahydro-1*H*-pyrido[2,1-*a*]isoquinolin-2-yl)-3-(2-methoxyethyl)-1-methylthiourea (16{6,11}**)**. Prepared according to the representative procedure for the formation of thioureas. Purification: EtOAc/MeOH (95 : 5). Yield: 87% (34 mg yellow solid). Data: ^{1}H NMR (400 MHz) δ 7.27-7.25 (m, 1 H), 7.20-7.16 (m, 1 H), 7.12-7.05 (comp, 2 H), 6.87 (dd, *J* = 8.0, 1.2 Hz, 1 H), 6.78 (dd, *J* = 8.0, 2.0 Hz, 1 H), 6.67 (d, *J* = 2.0 Hz, 1 H), 6.52-6.46 (m, 1 H), 6.04-6.00 (m, 1 H), 4.65 (ddd, *J* = 13.2, 4.4, 4.4 Hz, 1 H), 4.54 (dd, *J* = 12.0, 3.6 Hz, 1 H), 4.09-4.03 (comp, 2 H), 3.91-3.77 (comp, 2 H), 3.54-3.55 (comp, 2 H), 3.35 (s, 3 H), 3.10-3.03 (m, 1 H), 2.86 (s, 3 H), 2.83-2.79 (comp, 2 H), 2.68 (ddd, *J*

= 14.4, 10.4, 4.0 Hz, 1 H), 2.60-2.56 (m, 1 H), 2.22 (s, 3 H), 1.53 (ddd, *J* = 12.4, 10.4, 6.4 Hz, 1 H), 1.42 (d, *J* = 6.4 Hz, 3 H); LCMS purity: 91%.



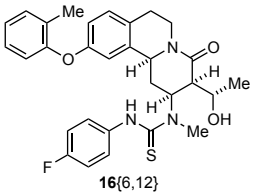
16{1,12}

3-(4-Fluorophenyl)-1-(3-(1-hydroxyethyl)-4-oxo-10-phenyl-2,3,4,6,7,11*b*-hexahydro-1*H*-pyrido[2,1-*a*]isoquinolin-2-yl)-1-methylthiourea (16{1,12}**)**. Prepared according to the representative procedure for the formation of thioureas. Purification: pentane/EtOAc (2 : 8). Yield: 75% (33 mg yellow solid). Data: ^{1}H NMR (400 MHz) δ 7.56-7.53 (comp, 2 H), 7.48-7.42 (comp, 3 H), 7.38-7.34 (comp, 2 H), 7.27 (d, *J* = 8.0 Hz, 1 H), 7.24-7.22 (comp, 2 H), 7.02-6.97 (comp, 2 H), 6.35 (br s, 1 H), 4.75-4.65 (comp, 2 H), 4.24-4.18 (m, 1 H), 4.07-4.03 (m, 1 H), 3.16-3.09 (m, 1 H), 3.04 (s, 3 H), 2.94-2.82 (comp, 3 H), 2.69-2.66 (m, 1 H), 1.68 (ddd, *J* = 14.4, 12.4, 6.4 Hz, 1 H), 1.47 (d, *J* = 6.0 Hz, 3 H); LCMS purity: 96%.



16{5,12}

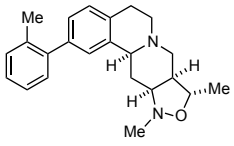
3-(4-Fluorophenyl)-1-(3-(1-hydroxyethyl)-10-(4-methylpiperazin-1-yl)-4-oxo-2,3,4,6,7,11*b*-hexahydro-1*H*-pyrido[2,1-*a*]isoquinolin-2-yl)-1-methylthiourea (16{5,12}**)**. Prepared according to the representative procedure for the formation of thioureas. Purification: EtOAc/MeOH (60 : 40) with 1% Et_3N. Yield: 74% (31 mg yellow solid). Data: mp 194-195 °C; ^{1}H NMR (600 MHz) δ 7.43 (br s, 1 H), 7.26-7.21 (comp, 2 H), 7.09 (d, *J* = 8.4 Hz, 1 H), 7.06-7.00 (comp, 2 H), 6.83 (dd, *J* = 8.4, 2.4 Hz, 1 H), 6.69 (d, *J* = 2.4 Hz, 1 H), 6.41 (br s , 1 H), 4.65 (ddd, *J* = 12.6, 4.2, 4.2 Hz, 1 H), 4.57 (dd, *J* = 12.0, 3.6 Hz, 1 H), 4.29-4.25 (m, 1 H), 3.20-3.18 (comp, 4 H), 3.08-3.03 (m, 1 H), 3.03 (s, 3 H), 2.87-2.77 (comp, 3 H), 2.65-2.59 (comp, 6 H), 2.37 (s, 3 H), 1.61 (ddd, *J* = 14.4, 12.0, 6.0 Hz, 1 H), 1.46 (d, *J* = 6.0 Hz, 3 H); ^{13}C NMR (150 MHz) δ 181.9, 171.6, 160.8 ($J_{C\text{-}F}$ = 245.4 Hz), 150.5, 135.5, 135.2, 129.4, 127.9, 127.3 ($J_{C\text{-}F}$ = 8.6 Hz), 125.5, 115.5 ($J_{C\text{-}F}$ = 22.7 Hz), 115.4, 113.5, 65.4, 64.3, 55.0, 53.5, 49.2, 46.0, 38.6, 37.7, 33.1, 28.4, 21.0;IR (neat) 3270, 2935, 1652, 1426, 1247, 1098, 733 cm^{-1}; mass spectrum (CI) *m/z* 526.2640 [$C_{28}H_{37}FN_5O_2S$ (M+1) requires 526.2652]; LCMS purity: 95%.



16{6,12}

3-(4-Fluorophenyl)-1-(3-(1-hydroxyethyl)-4-oxo-10-(*o*-tolyloxy)-2,3,4,6,7,11*b*-hexahydro-1*H*-pyrido[2,1-*a*]isoquinolin-2-yl)-1-methylthiourea (16{6,12}**)**. Prepared according to the representative procedure for the formation of thioureas. Purification: pentane/EtOAc (20 : 80). Yield: 68% (38 mg yellow solid). Data: mp 115-117 °C; ^{1}H NMR (600 MHz) δ 7.43 (br s, 1 H), 7.27-7.15 (comp, 4 H), 7.12 (d, *J* = 8.4 Hz, 1 H), 7.11-7.00 (comp, 3 H), 6.88 (dd, *J* = 8.4, 1.2 Hz, 1 H), 6.79 (dd, *J* = 7.8, 2.4 Hz, 1 H), 6.69 (d, *J* = 3.0 Hz, 1 H), 4.67 (ddd, *J* = 13.2, 4.2, 4.2 Hz, 1 H), 4.55 (dd, *J* = 12.0, 3.6 Hz, 1 H), 4.27-4.22 (m, 1 H), 3.81 (br s, 1 H), 3.10-3.03 (m, 1 H), 3.04 (s, 3 H), 2.85-2.83 (comp, 2 H), 2.74 (ddd, *J* = 13.8, 9.6, 3.6 Hz, 1 H), 2.62-2.60 (m, 1 H), 2.23 (s, 3 H), 1.64-1.59 (comp, 2 H), 1.45 (d, *J* = 6.6 Hz, 3 H); ^{13}C NMR (150 MHz) δ 183.8, 171.5, 160.8 ($J_{C\text{-}F}$ = 244.2 Hz), 157.1, 154.4, 136.0, 135.5, 131.7, 130.1, 130.0, 128.3, 127.9, 127.3, 124.4, 119.8, 116.4, 115.5 ($J_{C\text{-}F}$ = 22.7 Hz), 114.6, 65.4, 53.7, 53.2, 50.2, 38.5, 37.5, 33.1, 28.6, 21.1, 16.2; LCMS purity: 94%.

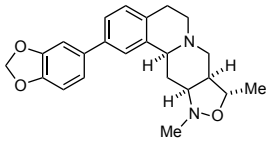
Representative procedure for the formation of 17{7} and **17**{8} **via Suzuki cross-coupling**:



17{7}

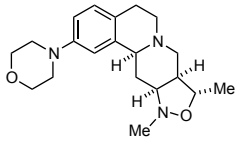
8,10-Dimethyl-2-*o*-tolyl-5,6,7*a*,8,10,10*a*,11,11*a*-octahydro-7*H*-9-oxa-6*a*,10-diaza-cyclopenta[*b*]phenanthrene (17{7}**)**. A mixture of bromide **11** (50 mg, 0.15 mmol), cesium fluoride (91 mg, 0.59 mmol), 2-methylbenzeneboronic acid (**12**{7}) (41 mg, 0.30 mmol), and $[PdCl_2(dppf)]\cdot CH_2Cl_2$ (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 **17**{7} 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), 3.30-3.13 (comp, 3 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 cm^{-1}; mass spectrum (ESI) *m/z* 349.2273 [$C_{23}H_{29}N_2O$ (M+1) requires 349.2280]; LCMS purity: 100%.



17{8}

2-Benzo[1,3]dioxol-5-yl-8,10-dimethyl-5,6,7*a*,8,10,10*a*,11,11*a*-octahydro-7*H*-9-oxa-6*a*,10-diaza-cyclopenta[*b*]phenanthrene (17{8}). Prepared according to the representative procedure for the formation of **17**{7} and **17**{8} via Suzuki cross-coupling. Purification: EtOAc. Yield: 82% (366 mg tan solid). Data: : mp 188-189 °C; ^{1}H NMR (400 MHz) δ 7.34-7.33 (m, 1 H), 7.28 (dd, *J* = 8.0, 1.6 Hz, 1 H), 7.12 (d, *J* = 8.0 Hz, 1 H), 7.02-6.99 (comp, 2 H), 6.85 (d, *J* = 8.0 Hz, 1 H), 5.98 (s, 2 H), 4.37-4.30 (m, 1 H), 3.32-3.22 (m, 1 H), 3.18-3.09 (comp, 2 H), 2.99 (d, *J* = 11.6 Hz, 1 H), 2.89 (dd, *J* = 11.6, 4.8 Hz, 1 H), 2.77 (s, 3 H), 2.76-2.71 (m, 1 H), 2.67 (ddd, *J* =12.4, 12.4, 4.0 Hz, 1 H), 2.60-2.44 (comp, 3 H),1.81-1.72 (m, 1 H), 1.39 (d, *J* = 5.6 Hz, 3 H); ^{13}C NMR (100 MHz) δ 148.1, 146.9, 138.7, 137.9, 135.7, 133.5, 129.2, 124.7, 123.7, 120.4, 108.5, 107.6, 101.1, 77.3, 65.9, 60.6, 54.2, 52.0, 47.4, 46.0, 35.0, 29.4, 21.4; IR (neat) 2960, 2915, 2825, 2766, 1485, 1230 cm^{-1}; mass spectrum (ESI) *m/z* 379.2019 [$C_{23}H_{27}N_2O_3$ (M+1) requires 379.2022]; LCMS purity: 96%.

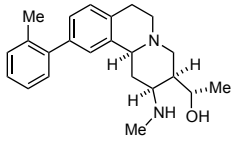


17{4}

8,10-Dimethyl-2-morpholin-4-yl-5,6,7*a*,8,10,10*a*,11,11*a*-octahydro-7*H*-9-oxa-6*a*,10-diaza-cyclopenta[*b*]phenanthrene (17{4}). A suspension of (±)-BINAP (69 mg, 0.11 mmol) in toluene (6.0 mL) was heated at 80 °C until a homogeneous solution was observed (~5 min). The reaction was cooled to room temperature, whereupon $Pd(OAc)_2$ (20 mg, 0.09 mmol) was added. The reaction was stirred for 1 min at room temperature. The catalyst solution was added to a solution of bromide **11** (300 mg, 0.89 mmol), morpholine (**12**{4}) (117 mg, 0.12 mL, 1.34

mmol), and Cs_2CO_3 (580 mg, 1.78 mmol) in toluene (7 mL). The reaction mixture was heated at 100 °C and for 6 h, whereupon H_2O (13 mL) and EtOAc (13 mL) were added. The mixture was filtered through a pad of Celite, layers were separated. The aqueous layer was extracted with EtOAc (3 × 13 mL), and the combined organic layers were washed with brine, dried (Na_2SO_4), filtered, and concentrated under reduced pressure. The residue was purified by flash column chromatography eluting with CH_2Cl_2/MeOH (20 : 1) to give 248 mg (81%) of **17**{4} as a light brown solid: mp 159-160 °C; ^{1}H NMR (400 MHz) δ 6.98 (d, *J* = 8.0 Hz, 1 H), 6.74-6.71 (comp, 2 H), 4.33-4.27 (m, 1 H), 3.83-3.81 (comp, 4 H), 3.28-3.20 (m, 1 H), 3.08-2.95 (comp, 7 H), 2.85 (dd, *J* = 10.8, 5.2 Hz, 1 H), 2.74 (s, 3 H), 2.64-2.55 (comp, 2 H), 2.50-2.39 (comp, 3 H), 1.74-1.65 (m, 1 H), 1.36 (d, *J* = 5.6 Hz, 3 H); ^{13}C NMR (100 MHz) δ 149.6, 138.1, 129.4, 126.5, 114.7, 112.5, 77.0, 66.9, 65.9, 60.8, 54.2, 52.2, 50.0, 47.4, 45.9, 35.1, 28.8, 21.4; IR (neat) 3400 , 2960, 2916, 2854, 2817, 2755, 1613, 1509 cm^{-1}; mass spectrum (ESI) *m/z* 344.2333 [$C_{20}H_{30}N_3O_2$ (M+1) requires 344.2338]; LCMS purity: 100%.

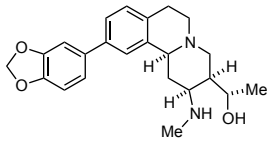
Representative procedure for the formation of 18{7}, 18{8}, and 18{4} via *N,O*-bond cleavage:



18{7}

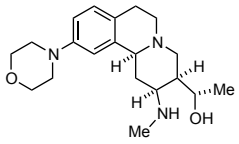
1-(2-(Methylamino)-10-*o*-tolyl-2,3,4,6,7,11*b*-hexahydro-1*H*-pyrido[2,1-*a*]isoquinolin-3-yl)ethanol (18{7}). Sodium borohydride (33 mg, 0.86 mmol) was added to a solution of isoxazolidine **11** (50 mg, 0.14 mmol) and $NiCl_2 \cdot 6H_2O$ (72 mg, 0.29 mmol) in anhydrous MeOH (2.9 mL), and the reaction 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) with 1% Et_3N to give 45 mg (90%) of **18**{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 cm^{-1}; mass spectrum (ESI) *m/z* 351.2431 [$C_{23}H_{31}N_2O$ (M+1) requires 351.2436]; LCMS purity: 99%.



18{8}

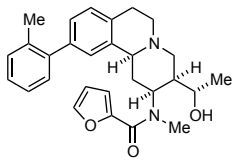
1-(10-(Benzo[*d*][1,3]dioxol-5-yl)-2-(methylamino)-2,3,4,6,7,11*b*-hexahydro-1*H*-pyrido[2,1-*a*]isoquinolin-3-yl)ethanol (18{8}). Prepared according to the representative procedure for the formation of **18**{7}, **18**{8}, and **18**{4} via *N,O*-bond cleavage. Purification: EtOAc/MeOH (5 : 1) with 1% Et_3N. Yield: 84% (80 mg colorless foam). Data: ^{1}H NMR (400 MHz) δ 7.33-7.27 (comp, 2 H), 7.13 (d, *J* = 8.0 Hz, 1 H), 7.03-6.99 (comp, 2 H), 6.86 (d, *J* = 7.6 Hz, 1 H), 5.99 (s, 2 H), 4.47-4.40 (m, 1 H), 3.25-3.22 (m, 1 H), 3.17-3.09 (m, 1 H), 2.98-2.91 (comp, 2 H), 2.86 (dd, *J* = 11.6, 5.6 Hz, 1 H), 2.70 (dd, *J* = 16.4, 2.8 Hz, 1 H), 2.54 (s, 3 H), 2.43 (ddd, *J* = 11.6, 11.6, 3.6 Hz, 1 H), 2.35-2.30 (comp, 2 H), 1.96-1.87 (m, 1 H), 1.82-1.78 (comp, 2 H), 1.21 (d, *J* = 6.0 Hz, 3 H); LCMS purity: 100%.



18{4}

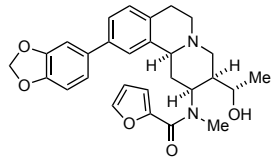
1-(2-(Methylamino)-10-morpholino-2,3,4,6,7,11*b*-hexahydro-1*H*-pyrido[2,1-*a*]isoquinolin-3-yl)ethanol (18{4}). Prepared according to the representative procedure for the formation of **18**{7}, **18**{8}, and **18**{4} via *N,O*-bond cleavage. Purification: CH_2Cl_2/MeOH (5 : 1) with 1% Et_3N. Yield: 90% (286 mg colorless powder). Data: mp 157-158 °C; ^{1}H NMR (400 MHz, CD_3OD) δ 6.97 (d, *J* = 8.4 Hz, 1 H), 6.82 (d, *J* = 2.8 Hz, 1 H), 6.80 (dd, *J* = 8.4, 2.8 Hz, 1 H), 4.48-4.41 (m, 1 H), 3.83-3.80 (comp, 4 H), 3.17-3.14 (m, 1 H), 3.09-3.06 (comp, 4 H), 3.04-2.84 (comp, 4 H), 2.58 (dd, *J* = 15.6, 3.2 Hz, 1 H), 2.47 (s, 3 H), 2.45-2.30 (comp, 3 H), 1.82-1.80 (m, 1 H), 1.74-1.65 (m, 1 H), 1.20 (d, *J* = 6.0 Hz, 3 H); ^{13}C NMR (100 MHz, CD_3OD) δ 149.7, 137.9, 129.0, 126.4, 114.7, 111.9, 67.4, 66.6, 62.9, 62.7,57.1, 52.5, 50.0, 41.1, 33.4, 33.0,

28.4, 20.4; IR (neat) 3434, 2958, 2801, 2530, 1612, 1507, 1450, 1119 cm^{-1}; mass spectrum (ESI) *m/z* 346.2489 [$C_{20}H_{32}N_3O_2$ (M+1) requires 346.2489]; LCMS purity: 100%.



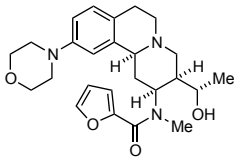
19{7,3}

***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 (19**{7,3}**)**. Prepared according to the representative procedure for the formation of amides. Purification: hexanes/EtOAc (1 : 1 → 1 : 2). Yield: 77% (21 mg yellow oil). Data: ^{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 cm^{-1}; mass spectrum (ESI) *m/z* 445.2486 [$C_{28}H_{33}N_2O_3$ (M+1) requires 445.2491]; LCMS purity: 82%.



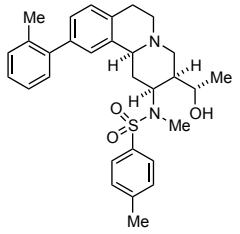
19{8,3}

***N*-(10-(benzo[*d*][1,3]dioxol-5-yl)-3-(1-hydroxyethyl)-2,3,4,6,7,11*b*-hexahydro-1*H*-pyrido[2,1-*a*]isoquinolin-2-yl)-*N*-methylfuran-2-carboxamide (19**{8,3}**)**. Prepared according to the representative procedure for the formation of amides. Purification: hexanes/EtOAc (1 : 3). Yield: 67% (32 mg yellow oil). Data: ^{1}H NMR (400 MHz) δ 7.51 (dd, *J* = 1.6, 0.8 Hz, 1 H), 7.36-7.31 (comp, 2 H), 7.16 (d, *J* = 7.6 Hz, 1 H), 7.03-6.99 (comp, 3 H), 6.87 (d, *J* = 7.6 Hz, 1 H), 6.49 (dd, *J* = 3.6, 2.0 Hz, 1 H), 5.99 (s, 2 H), 4.80-4.74 (m, 1 H), 4.33-4.27 (m, 1 H), 3.48-3.44 (m, 1 H), 3.35 (s, 3 H), 3.21-3.12 (m, 1 H), 3.05-2.98 (comp, 2 H), 2.85 (dd, *J* = 11.6, 2.8 Hz, 1 H), 2.77-2.73 (m, 1 H), 2.54 (ddd, *J* = 11.6, 11.6, 3.2 Hz, 1 H), 2.50-2.37 (comp, 3 H), 1.25 (d, *J* = 6.8 Hz, 3 H); LCMS purity: 97%.



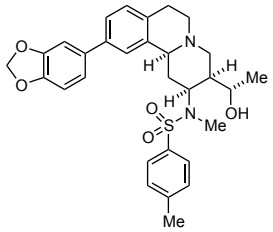
19{4,3}

***N*-(3-(1-Hydroxyethyl)-10-morpholino-2,3,4,6,7,11*b*-hexahydro-1*H*-pyrido[2,1-*a*]isoquinolin-2-yl)-*N*-methylfuran-2-carboxamide (19**{4,3}**)**. Prepared according to the representative procedure for the formation of amides. Purification: hexanes/EtOAc (1 : 2 → 0 : 100). Yield: 99% (32 mg yellow solid). Data: ^{1}H NMR (400 MHz) δ 7.51 (dd, *J* = 2.0, 0.8 Hz, 1 H), 7.04 (d, *J* = 8.4 Hz, 1 H), 7.00 (d, *J* = 3.2 Hz, 1 H), 6.80-6.78 (m, 1 H), 6.72-6.71 (m, 1 H), 6.49 (dd, *J* = 3.6, 2.0 Hz, 1 H), 4.79-4.70 (m, 1 H), 4.32-4.28 (m, 1 H), 3.87-3.85 (comp, 4 H), 3.37-3.34 (comp, 4 H), 3.16-2.91 (comp, 7 H), 2.88-2.79 (m, 1 H), 2.67-2.63 (m, 1 H), 2.53-2.30 (comp, 4 H), 1.24 (d, *J* = 6.8 Hz, 3 H); LCMS purity: 99%.



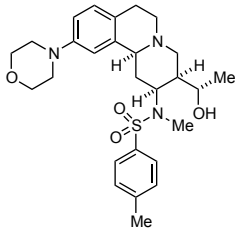
19{7,13}

***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*,4-dimethylbenzenesulfonamide (19**{7,13}**)**. Prepared according to the representative procedure for the formation of sulfonamides. Purification: hexanes/EtOAc (2 : 1). Yield: 62% (24 mg tan oil). Data: ^{1}H NMR (400 MHz) δ 7.73 (d, *J* = 8.4 Hz, 2 H), 7.30 (d, *J* = 8.4 Hz, 2 H), 7.28-7.21 (comp, 3 H), 7.14 (dd, *J* = 6.4, 2.0 Hz, 1 H), 7.11-7.08 (comp, 2 H), 6.71-6.70 (m, 1 H), 4.34-4.28 (m, 1 H), 4.15 (ddd, *J* = 12.8, 4.8, 4.8 Hz, 1 H), 3.24-3.21 (m, 1 H), 3.12 (ddd, *J* = 17.2, 12.4, 6.0 Hz, 1 H), 3.02 (dd, *J* = 12.0, 2.4 Hz, 1 H), 2.96 (dd, *J* = 11.2, 4.8 Hz, 1 H), 2.92 (s, 3 H), 2.76-2.74 (m, 1 H), 2.72 (dd, *J* = 12.0, 3.2 Hz, 1 H), 2.49 (ddd, *J* = 12.0, 12.0, 3.6 Hz, 1 H), 2.32 (s, 3 H), 2.19 (s, 3 H), 2.11-2.00 (comp, 2 H), 1.65 (ddd, *J* = 12.4, 4.0, 4.0 Hz, 1 H), 1.38 (d, *J* = 6.8 Hz, 3 H); ^{13}C NMR (100 MHz) δ 143.5, 141.7, 139.5, 136.4, 135.5, 135.3, 132.7, 130.4, 129.8, 129.7, 128.6, 127.3, 127.2, 127.1, 125.8, 125.2, 72.8, 63.1, 62.6, 57.7, 52.1, 45.0, 32.6, 32.1, 29.6, 21.5, 21.4, 20.5; IR (neat) 3384, 2926, 2814, 2755, 2250, 1482 1338, 1164 cm^{-1}; mass spectrum (ESI) *m/z* 505.2520 [$C_{30}H_{37}N_2O_3S$ (M+1) requires 505.2519]; LCMS purity: 97%.



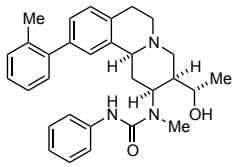
19{8,13}

***N*-(10-(Benzo[*d*][1,3]dioxol-5-yl)-3-(1-hydroxyethyl)-2,3,4,6,7,11*b*-hexahydro-1*H*-pyrido[2,1-*a*]isoquinolin-2-yl)-*N*,4-dimethylbenzenesulfonamide (19**{8,13}**)**. Prepared according to the representative procedure for the formation of sulfonamides. Purification: hexanes/EtOAc (3 : 2). Yield: 55% (19 mg colorless solid). Data: ^{1}H NMR (400 MHz) δ 7.76 (d, *J* = 8.4 Hz, 2 H), 7.35 (d, *J* = 8.4 Hz, 2 H), 7.26 (dd, *J* = 8.0, 1.6 Hz, 1 H), 7.09 (d, *J* = 8.0 Hz, 1 H), 6.91-6.81 (comp, 4 H), 5.99 (s, 2 H), 4.30-4.24 (m, 1 H), 4.17 (ddd, *J* = 12.8, 4.8 Hz, 1 H), 3.22-3.17 (m, 1 H), 3.07 (ddd, *J* = 17.2, 12.0, 5.6 Hz, 1 H), 3.00 (dd, *J* = 11.8, 2.4 Hz, 1 H), 2.94-2.90 (m, 1 H), 2.92 (s, 3 H), 2.17-2.64 (comp, 2 H), 2.44 (ddd, *J* = 12.0, 12.0, 3.6 Hz, 1 H), 2.41 (s, 3 H), 2.07-1.97 (comp, 2 H), 1.67 (ddd, *J* = 12.4, 4.0, 4.0 Hz, 1 H), 1.34 (d, *J* = 6.8 Hz, 3 H); LCMS purity: 99%.



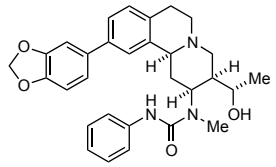
19{4,13}

***N*-(3-(1-Hydroxyethyl)-10-morpholino-2,3,4,6,7,11*b*-hexahydro-1*H*-pyrido[2,1-*a*]isoquinolin-2-yl)-*N*,4-dimethylbenzenesulfonamide (19**{4,13}**)**. Prepared according to the representative procedure for the formation of sulfonamides. Purification: hexanes/EtOAc (1 : 1). Yield: 73% (26 mg yellow oil). Data: ^{1}H NMR (400 MHz) δ 7.78 (d, *J* = 8.4 Hz, 2 H), 7.35 (d, *J* = 8.4 Hz, 2 H), 6.98 (d, *J* = 8.4 Hz, 1 H), 6.73 (dd, *J* = 8.4, 2.4 Hz, 1 H), 6.31 (d, *J* = 2.4 Hz, 1 H), 4.31-4.19 (comp, 2 H), 3.95-3.80 (comp, 4 H), 3.17-3.14 (m, 1 H), 3.05-2.95 (comp, 5 H), 2.94 (s, 3 H), 2.91-2.87 (m, 1 H), 2.67 (dd, *J* = 12.0, 3.2 Hz, 1 H), 2.58 (dd, *J* = 16.0, 2.8 Hz, 1 H), 2.45 (s, 3 H), 2.43-2.36 (m, 1 H), 2.07-1.98 (comp, 2 H), 1.69 (ddd, *J* = 12.4, 4.0, 4.0 Hz, 1 H), 1.31 (d, *J* = 6.8 Hz, 3 H); LCMS purity: 96%.



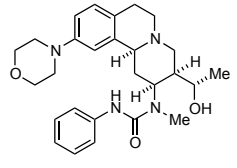
19{7,9}

1-(3-(1-Hydroxyethyl)-10-*o*-tolyl-2,3,4,6,7,11*b*-hexahydro-1*H*-pyrido[2,1-*a*]isoquinolin-2-yl)-1-methyl-3-phenylurea (19{7,9}). Prepared according to the representative procedure for the formation of ureas. Purification: hexanes/EtOAc (10 : 1 → 1 : 1). Yield: 55% (18 mg yellow oil). Data: ^{1}H NMR (400 MHz) δ 7.33 (d, *J* = 8.8 Hz, 2 H), 7.24-7.03 (comp, 9 H), 6.98-6.94 (m, 1 H), 6.48 (br s, 1 H), 4.56-4.47 (m, 1 H), 4.26-4.16 (m, 1 H), 3.44-3.34 (m, 1 H), 3.19-3.06 (m, 1 H), 3.03 (s, 3 H), 2.99-2.90 (comp, 2 H), 2.77-2.65 (comp, 2 H), 2.56-2.45 (m, 1 H), 2.35-2.25 (comp, 2 H), 2.20 (s, 3 H), 2.19-2.12 (m, 1 H), 1.21 (d, *J* = 6.8 Hz, 3 H); LCMS purity: 99%.



19{8,9}

1-(10-(Benzo[*d*][1,3]dioxol-5-yl)-3-(1-hydroxyethyl)-2,3,4,6,7,11*b*-hexahydro-1*H*-pyrido[2,1-*a*]isoquinolin-2-yl)-1-methyl-3-phenylurea (19{8,9}). Prepared according to the representative procedure for the formation of ureas. Purification: CH_2Cl_2/MeOH (98 : 2). Yield: 70% (23 mg colorless oil). Data: ^{1}H NMR (400 MHz) δ 7.43-7.40 (comp, 2 H), 7.34-7.25 (comp, 4 H), 7.16 (d, *J* = 8.0 Hz, 1 H), 7.06-7.00 (comp, 3 H), 6.87 (dd, *J* = 7.6, 0.4 Hz, 1 H), 6.62 (br s, 1 H), 5.99 (s, 2 H), 4.64-4.58 (m, 1 H), 4.31-4.23 (m, 1 H), 3.48-3.42 (m, 1 H), 3.20-3.12 (m, 1 H), 3.12 (s, 3 H), 3.05-2.95 (comp, 2 H), 2.82-2.71 (comp, 2 H), 2.52 (ddd, *J* = 11.6, 11.6, 3.2 Hz, 1 H), 2.47-2.33 (comp, 2 H), 2.24-2.18 (m, 1 H), 1.28 (d, *J* = 6.8 Hz, 3 H); LCMS purity: 99%.

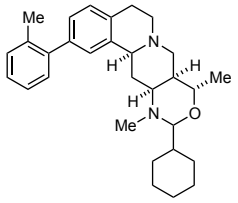


19{4,9}

1-(3-(1-Hydroxyethyl)-10-morpholino-2,3,4,6,7,11*b*-hexahydro-1*H*-pyrido[2,1-*a*]isoquinolin-2-yl)-1-methyl-3-phenylurea (19{4,9}). Prepared according to the representative procedure for the formation of ureas. Purification: hexanes/EtOAc (1 : 1 → 1 : 2). Yield: 99%

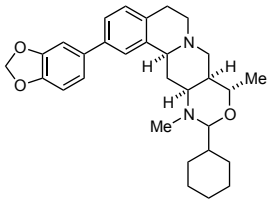
(34 mg yellow oil). Data: ^{1}H NMR (400 MHz) δ 7.43-7.40 (comp, 2 H), 7.31-7.27 (comp, 2 H), 7.06-7.01 (comp, 2 H), 6.78 (dd, *J* = 8.0, 2.4 Hz, 1 H), 6.70 (d, *J* = 2.4 Hz, 1 H), 6.64 (br s, 1 H), 4.60-4.55 (m, 1 H), 4.29-4.21 (m, 1 H), 3.87-3.84 (comp, 4 H), 3.40-3.35 (m, 1 H), 3.16-2.92 (comp, 10 H), 2.76 (dd, *J* = 11.6, 3.2 Hz, 1 H), 2.66-2.61 (m, 1 H), 2.47 (ddd, *J* = 12.0, 12.0, 3.2 Hz, 1 H), 2.37-2.32 (comp, 2 H), 2.20-2.16 (m, 1 H), 1.27 (d, *J* = 7.2 Hz, 3 H); ^{13}C NMR (100 MHz) δ 155.8, 149.9, 139.1, 137.8, 129.6, 128.9, 126.2, 123.0, 119.9, 114.8, 112.3, 72.9, 67.2, 66.9, 62.8, 55.8, 52.2, 49.9, 42.2, 34.5, 32.0, 29.1, 21.3; IR (neat) 3330, 2962, 2818, 1641, 1530, 1502, 1445 cm^{-1}; mass spectrum (ESI) *m/z* 487.2676 [$C_{27}H_{36}N_4O_3Na$ (M+Na) requires 487.2685]; LCMS purity: 98%.

Representative procedure for the formation of *N,O*-acetals:



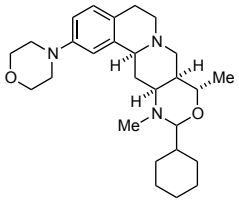
20{7,14}

10-Cyclohexyl-8,11-dimethyl-2-*o*-tolyl-5,7*a*,8,10,11,11*a*,12,12*a*-octahydro-6*H*,7*H*-9-oxa-6*a*,11-diaza-benzo[*a*]anthracene (20{7,14}). A solution of amino alcohol **18**{1} (50 mg, 0.14 mmol) and cyclohexanecarboxaldehyde (**15**{14}) (18 mg, 20 μL, 0.16 mmol) in 1,2-dichloroethane (2.0 mL) was heated under reflux for 24 h. The solvent was removed *in vacuo* and the residue was purified by flash column chromatography eluting with hexanes/EtOAc (7 : 1) to give 63 mg (99%) of **20**{7,14} as a yellow oil: ^{1}H NMR (400 MHz) δ 7.26-7.19 (comp, 5 H), 7.14-7.12 (comp, 2 H), 4.15-4.08 (m, 1 H), 4.07 (d, *J* = 8.8 Hz, 1 H), 3.19-3.10 (comp, 2 H), 2.99 (dd, *J* = 12.4, 2.0 Hz, 1 H), 3.00-2.93 (m, 1 H), 2.87 (dd, *J* = 11.8, 4.8 Hz, 1 H), 2.75-2.69 (m, 1 H), 2.46 (s, 3 H), 2.41 (ddd, *J* = 12.0, 12.0, 3.6 Hz, 1 H), 2.32 (dd, *J* = 12.8, 4.0 Hz, 1 H), 2.29 (s, 3 H), 2.29-2.22 (m, 1 H), 2.21-2.12 (m, 1 H), 2.10-1.91 (comp, 2 H), 1.78-0.81 (comp, 10 H), 1.26 (d, *J* = 6.0 Hz, 3 H); ^{13}C NMR (100 MHz) δ 141.8, 139.4, 137.8, 135.2, 133.0, 130.3, 129.8, 128.4, 127.1, 126.9, 125.9, 125.7, 89.7, 71.8, 63.1, 61.4, 57.4, 52.4, 38.8, 36.0, 35.0, 29.8, 29.7, 28.3, 26.5, 25.8, 20.6, 19.6; IR (neat) 2923, 2852, 1451, 1089, 1001, 758, 731 cm^{-1}; mass spectrum (ESI) *m/z* 445.3214 [$C_{30}H_{41}N_2O$ (M+1) requires 445.3219]; LCMS purity: 100%.



20{8,14}

2-Benzo[1,3]dioxol-5-yl-10-cyclohexyl-8,11-dimethyl-5,7*a*,8,10,11,11*a*,12,12*a*-octahydro-6*H*,7*H*-9-oxa-6*a*,11-diaza-benzo[*a*]anthracene (20{8,14}**)**. Prepared according to the representative procedure for the formation of *N,O*-acetals. Purification: hexanes/EtOAc (10 : 1 → 5 : 1). Yield: 84% (63 mg yellow oil). Data: ^{1}H NMR (400 MHz) δ 7.39 (d, *J* = 0.8 Hz, 1 H), 7.30 (dd, *J* = 8.0, 2.0 Hz, 1 H), 7.13 (d, *J* = 8.0 Hz, 1 H), 7.05-7.01 (comp, 2 H), 6.86 (d, *J* = 7.6 Hz, 1 H), 5.99 (s, 2 H), 4.15-4.09 (m, 1 H), 4.09 (d, *J* = 8.8 Hz, 1 H), 3.18-3.12 (m, 1 H), 3.13 (ddd, *J* = 17.2, 12.4, 6.0 Hz, 1 H), 3.04-2.97 (comp, 2 H), 2.87-2.83 (m, 1 H), 2.68 (dd, *J* = 16.4, 2.4 Hz, 1 H), 2.49 (s, 3 H), 2.42-2.30 (comp, 3 H), 2.25-2.16 (m, 1 H), 2.02-1.94 (comp, 2 H), 1.77-1.58 (comp, 4 H), 1.54-1.46 (m, 1 H), 1.31-1.08 (comp, 3 H), 1.25 (d, *J* = 6.0 Hz, 3 H), 0.94-0.82 (comp, 2 H); ^{13}C NMR (100 MHz) δ 148.1, 146.9, 138.5, 138.4, 135.6, 133.4, 129.3, 124.5, 123.5, 120.4, 108.5, 107.6, 101.1, 89.7, 71.8, 63.1, 61.5, 57.4, 52.3, 38.8, 35.9, 35.0, 30.0, 29.5, 28.2, 26.5, 25.8, 19.6; IR (neat) 2923, 2852, 2807, 2752, 1483, 1229, 1040 cm^{-1}; mass spectrum (ESI) *m/z* 475.2958 [$C_{30}H_{39}N_2O_3$ (M+1) requires 475.2955]; LCMS purity: 99%.

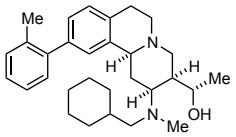


20{4,14}

10-Cyclohexyl-8,11-dimethyl-2-morpholin-4-yl-5,7*a*,8,10,11,11*a*,12,12*a*-octahydro-6*H*,7*H*-9-oxa-6*a*,11-diaza-benzo[*a*]anthracene (20{4,14}**)**. Prepared according to the representative procedure for the formation of *N,O*-acetals. Purification: hexanes/EtOAc (3 : 1). Yield: 81% (68 mg yellow oil). Data: ^{1}H NMR (400 MHz) δ 7.00 (d, *J* = 8.0 Hz, 1 H), 6.77 (d, *J* = 2.4 Hz, 1 H), 6.74 (dd, *J* = 8.0, 2.4 Hz, 1 H), 4.13-4.07 (m, 1 H), 4.08 (d, *J* = 9.2 Hz, 1 H), 3.86-3.83 (comp, 4 H), 3.15-2.95 (comp, 7 H), 2.81 (dd, *J* = 11.6, 5.2 Hz, 1 H), 2.36-2.12 (comp, 5 H), 2.03-1.87 (comp, 3 H), 1.75-1.61 (comp, 5 H), 1.51-1.47 (m, 1 H), 1.29-1.08 (comp, 4 H), 1.24 (d, *J* = 6.0 Hz, 3 H), 0.90-0.83 (comp, 3 H); ^{13}C NMR (100 MHz) δ 149.6, 138.5, 129.5, 126.5, 114.5, 112.4, 89.6, 71.9, 66.9, 63.3, 61.6, 57.3, 52.4, 49.9, 38.8, 35.8, 35.0, 30.0, 28.9,

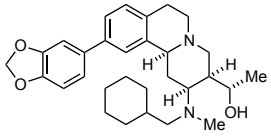
28.2, 26.4, 25.8, 19.5; IR (neat) 2924, 2853, 1451, 1122, 732 cm^{-1}; mass spectrum (ESI) *m/z* 440.3273 [$C_{27}H_{42}N_3O_2$ (M+1) requires 440.3277]; LCMS purity: 100%.

Representative procedure for reductive amination:



21{7,14}

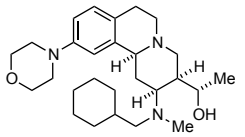
1-(2-((Cyclohexylmethyl)(methyl)amino)-10-*o*-tolyl-2,3,4,6,7,11*b*-hexahydro-1*H*-pyrido[2,1-*a*]isoquinolin-3-yl)ethanol (21{7,14}). $NaBH_3CN$ (7.6 mg, 0.12 mmol) was added to a solution of **20**{1,14} (36 mg, 0.08 mmol) and AcOH (7.3 mg, 6.9 μL, 0.12 mmol) in 1,2-dichloroethane (2 mL), and the reaction was stirred for 24 h at room temperature. Saturated aqueous $NaHCO_3$ (2 mL) was added, and the layers were separated. The aqueous layer was extracted with CH_2Cl_2 (2 × 2 mL). The combined organic layers were dried (Na_2SO_4), filtered, and concentrated under reduced pressure. The residue was purified by flash column chromatography eluting with hexanes/EtOAc (1 : 3) to give 30 mg (83%) of **21**{7,14} as a yellow oil: ^{1}H NMR (400 MHz) δ 7.30-7.10 (comp, 7 H), 4.47-4.40 (m, 1 H), 3.21-3.15 (m, 1 H), 3.13 (d, *J* = 11.6 Hz, 1 H), 2.97 (dd, *J* = 11.6, 3.6 Hz, 1 H), 2.86 (dd, *J* = 11.2, 6.0 Hz, 1 H), 2.71 (dd, *J* = 16.4, 3.2 Hz, 1 H), 2.59-2.39 (comp, 4 H), 2.33-2.26 (comp, 3 H), 2.28 (s, 3 H), 1.96-1.48 (comp, 10 H), 1.33-1.10 (comp, 3 H), 1.22 (d, *J* = 6.0 Hz, 3 H), 0.99-0.0.76 (comp, 2 H); IR (neat) 2924, 2860, 1117, 759, 730 cm^{-1}; mass spectrum (ESI) *m/z* 447.3369 [$C_{30}H_{43}N_2O$ (M+1) requires 447.3375]; LCMS purity: 97%.



21{8,14}

1-(10-(Benzo[*d*][1,3]dioxol-5-yl)-2-((cyclohexylmethyl)(methyl)amino)-2,3,4,6,7,11*b*-hexahydro-1*H*-pyrido[2,1-*a*]isoquinolin-3-yl)ethanol (21{8,14}). Prepared according to the representative procedure for reductive amination. Purification: CH_2Cl_2/MeOH (95 : 5). Yield: 70% (19 mg colorless oil). Data: ^{1}H NMR (400 MHz) δ 7.32 (d, *J* = 1.6 Hz, 1 H), 7.29 (dd, *J* = 8.0, 1.6 Hz, 1 H), 7.13 (d, *J* = 8.0 Hz, 1 H), 7.04-7.01 (comp, 2 H), 6.88 (d, *J* = 8.0 Hz, 1 H),

5.99 (s, 2 H), 4.46-4.39 (m, 1 H), 3.18-3.09 (comp, 2 H), 2.96 (dd, *J* = 11.6, 2.4 Hz, 1 H), 2.84 (dd, *J* = 11.2, 5.2 Hz, 1 H), 2.71-2.63 (comp, 3 H), 2.40 (ddd, *J* = 11.6, 11.6, 3.6 Hz, 1 H), 2.32 (s, 3 H), 2.33-2.29 (m, 1 H), 1.92-1.57 (comp, 10 H), 1.25-1.13 (comp, 3 H), 1.21 (d, *J* = 5.6 Hz, 3 H), 0.99-0.86 (comp, 2 H); ^{13}C NMR (100 MHz) δ 148.0, 146.9, 138.7, 138.2, 135.9, 133.8, 129.4, 125.0, 122.9, 120.6, 108.6, 107.7, 101.1, 77.2, 67.8, 66.9, 62.9, 58.0, 52.3, 41.8, 35.6, 31.3, 29.7, 29.6, 26.6, 26.1, 21.6; IR (neat) 3200, 2925, 2851, 2797, 1483, 1229, 1040, 731 cm^{-1}; mass spectrum (ESI) *m/z* 477.3115 [$C_{30}H_{41}N_2O_3$ (M+1) requires 477.3112]; LCMS purity: 100%.



21{4,14}

1-(2-((Cyclohexylmethyl)(methyl)amino)-10-morpholino-2,3,4,6,7,11*b*-hexahydro-1*H*-pyrido[2,1-*a*]isoquinolin-3-yl)ethanol (21{4,14}**)**. Prepared according to the representative procedure for reductive amination. Purification: EtOAc. Yield: 64% (25 mg yellow oil). Data: ^{1}H NMR (400 MHz) δ 7.01 (d, *J* = 8.8 Hz, 1 H), 6.78-6.73 (comp, 2 H), 4.47-4.39 (m, 1 H), 3.88-3.86 (comp, 4 H), 3.16-2.97 (comp, 6 H), 2.94 (dd, *J* = 12.0, 2.4 Hz, 1 H), 2.79 (dd, *J* = 10.4, 5.6 Hz, 1 H), 2.65 (ddd, *J* = 12.8, 3.6, 3.6 Hz, 1 H), 2.57-2.55 (m, 1 H), 2.49-2.46 (comp, 2 H), 2.39 (s, 3 H), 2.38-2.27 (comp, 2 H), 1.96-1.90 (m, 1 H), 1.90-1.56 (comp, 8 H), 1.21 (d, *J* = 6.0 Hz, 3 H), 1.02-0.81 (comp, 4 H); LCMS purity: 98%.

Lipinski's Rule Data:

Compound #	Molecular Weight	Clog P	H-bond donors	H-bond acceptors	Lipinski Rule of 5
10	351.24	1.9	0	3	Satisfied
11	337.26	2.66	0	3	Satisfied
13{1}	348.44	2.78	0	3	Satisfied
13{2}	378.46	2.62	0	4	Satisfied
13{3}	366.43	2.92	0	3	Satisfied
13{4}	357.45	1.02	0	5	Satisfied
13{5}	370.49	1.08	0	5	Satisfied
13{6}	378.46	3.14	0	3	Satisfied
14{1}	350.45	2.26	2	3	Satisfied
14{2]	380.48	3.07	2	5	Satisfied
14{3}	368.45	3.16	2	4	Satisfied

14{4}	359.47	1.17	2	6	Satisfied
14{5}	372.5	0.57	2	5	Satisfied
14{6}	380.49	3.37	2	5	Satisfied
16{2,1}	464.6	3.51	1	4	Satisfied
16{3,1}	452.56	3.81	1	3	Satisfied
16{4,1}	443.58	1.91	1	5	Satisfied
16{1,2}	422.52	1.7	1	4	Satisfied
16{5,2}	444.57	0.6	1	6	Satisfied
16{6,2}	452.54	2.06	1	4	Satisfied
16{1,3}	444.52	2.79	1	3	Satisfied
16{5,3}	466.57	1.09	1	5	Satisfied
16{6,3}	474.55	3.15	1	3	Satisfied
16{1,4}	455.55	2.51	1	4	Satisfied
16{5,4}	477.6	0.82	1	6	Satisfied
16{6,4}	485.57	2.87	1	4	Satisfied
16{1,5}	479.57	3.58	1	4	Satisfied
16{5,5}	501.62	1.89	1	6	One Violation
16{6,5}	509.6	3.95	1	4	One Violation
16{2,6}	520.64	3.32	1	5	One Violation
16{3,6}	508.6	3.62	1	4	One Violation
16{4,6}	499.62	1.72	1	6	Satisfied
16{1,7}	520.64	3.32	1	5	One Violation
16{5,7}	542.69	1.63	1	7	One Violation
16{6,7}	550.67	3.69	1	5	One Violation
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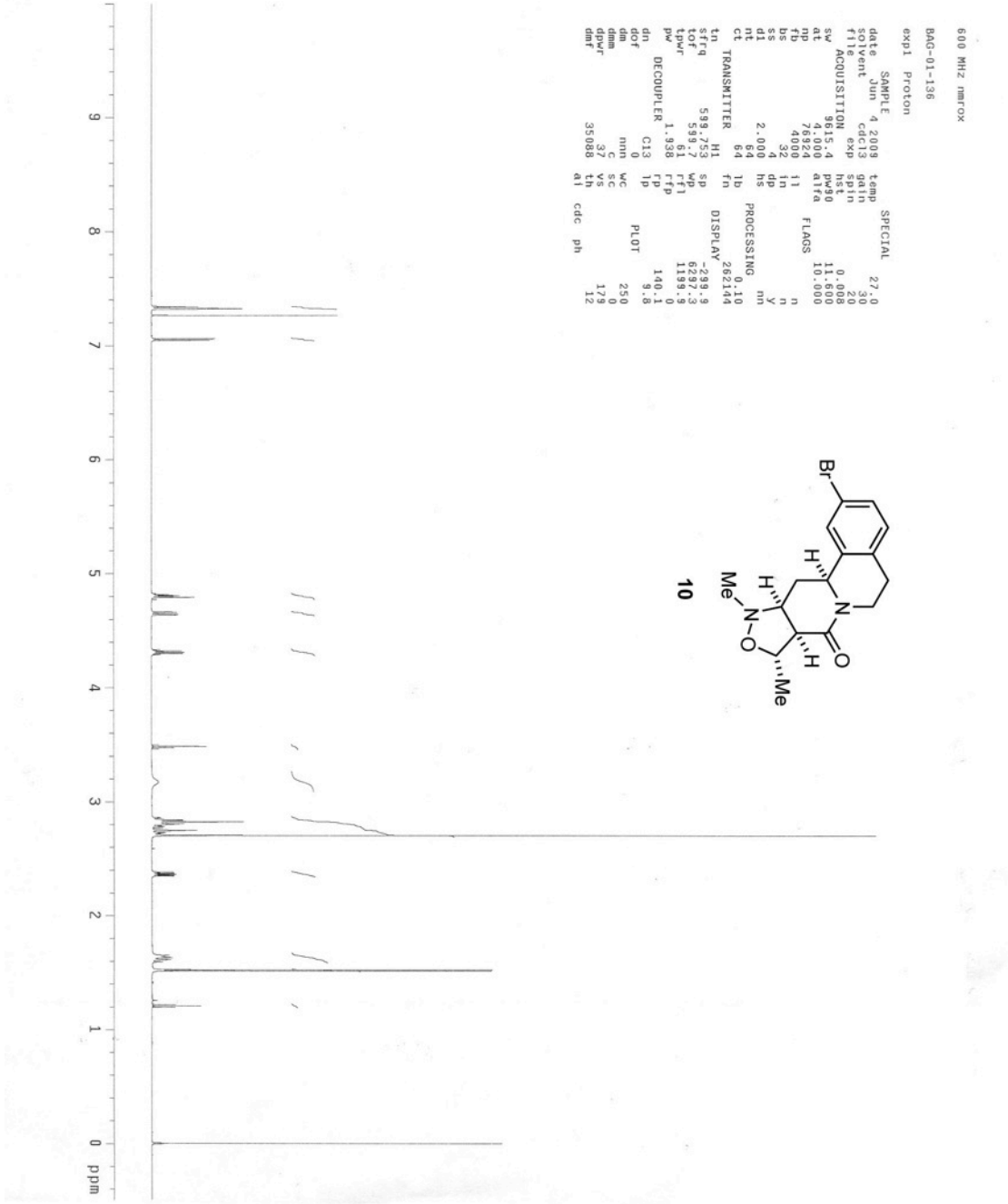
16{5,8}	497.67	1.87	2	5	Satisfied
16{6,8}	505.65	3.93	2	3	One Violation
16{2,9}	499.6	3.62	2	4	Satisfied
16{3,9}	487.57	3.92	2	3	Satisfied
16{4,9}	478.58	2.02	2	5	Satisfied
16{1,10}	538.47	4.99	2	3	One Violation
16{5,10}	560.52	3.29	2	5	One Violation
16{6,10}	568.49	5.35	2	3	Violated
16{1,11}	467.62	2.6	2	3	Satisfied
16{5,11}	489.67	0.91	2	5	Satisfied
16{6,11}	497.65	2.97	2	3	Satisfied
16{1,12}	503.63	4.81	2	2	One Violation
16{5,12}	525.68	2.93	2	4	One Violation
16{6,12}	533.66	5.18	2	2	Violated
17{7}	348.48	4.05	0	3	Satisfied
17{8}	378.46	3.16	0	5	Satisfied
17{4}	343.46	1.78	0	5	Satisfied
18{7}	350.5	3.54	2	3	Satisfied
18{8}	380.48	2.65	2	5	Satisfied
18{4}	345.48	1.27	2	5	Satisfied
19{7,3}	444.57	4.06	1	3	Satisfied

19{8,3}	474.55	3.17	1	5	Satisfied
19{4,3}	439.55	1.79	1	5	Satisfied
19{7,13}	504.68	5.27	1	4	Violated
19{8,13}	534.67	4.38	1	6	One Violation
19{4,13}	499.67	3	1	6	Satisfied
19{7,5}	469.62	5.06	2	3	One Violation
19{8,5}	499.6	4.17	2	5	Satisfied
19{4,5}	464.61	3.44	2	7	Satisfied
20{7,14}	444.65	6.72	0	3	One Violation
20{8,14}	474.63	5.83	0	5	One Violation
20{4,14}	439.63	4.45	0	5	Satisfied
21{7,14}	446.67	6.04	1	3	One Violation
21{8,14}	476.65	5.15	1	5	One Violation
21{4,14}	441.65	3.77	1	5	Satisfied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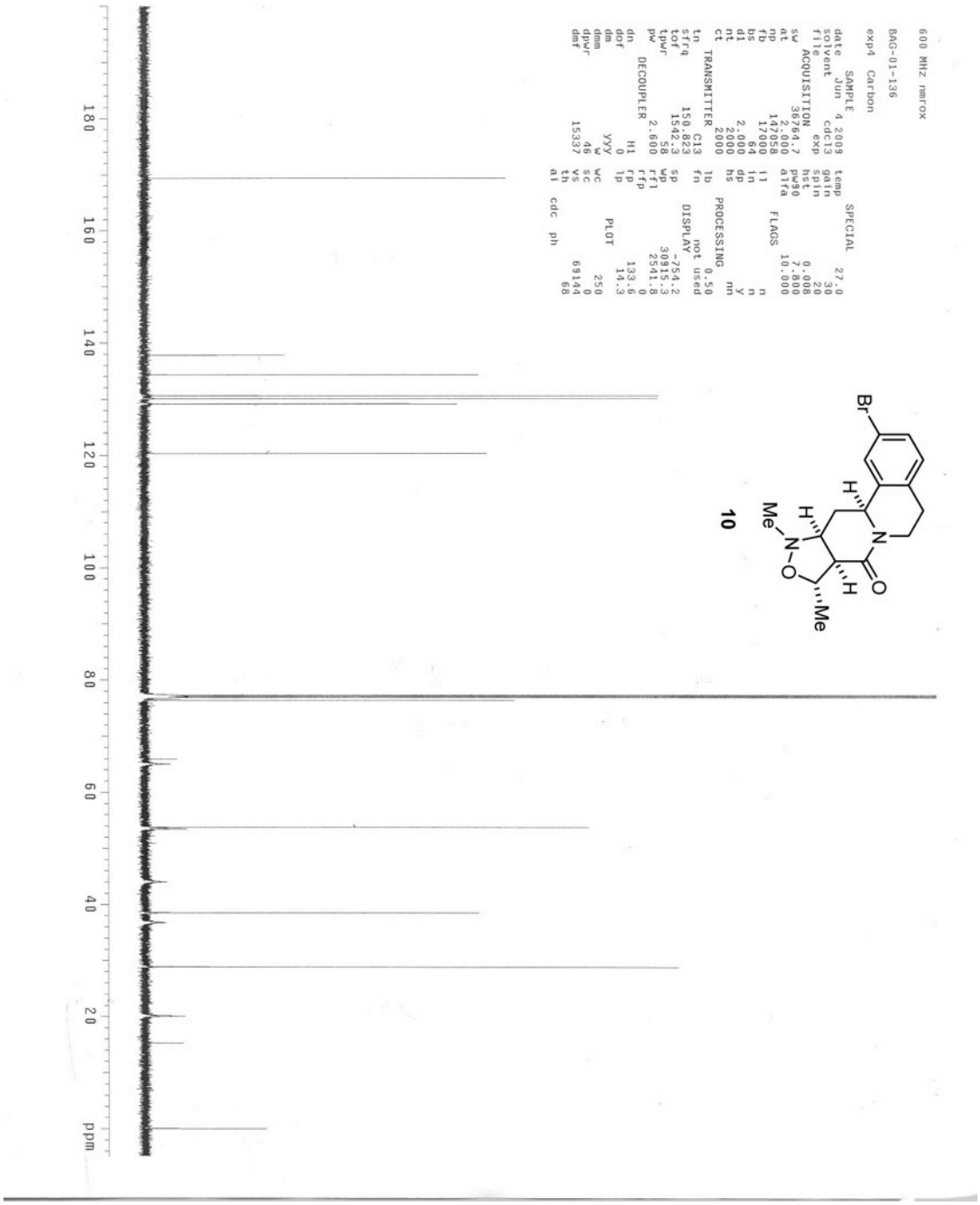
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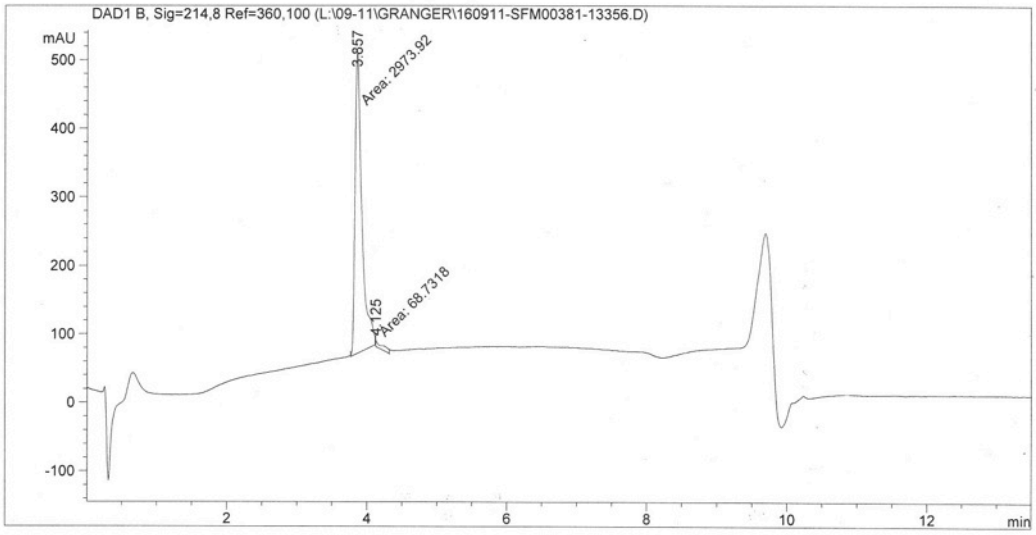


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Use Multiplier & Dilution Factor with ISTDs


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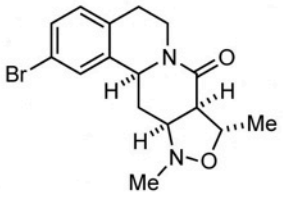
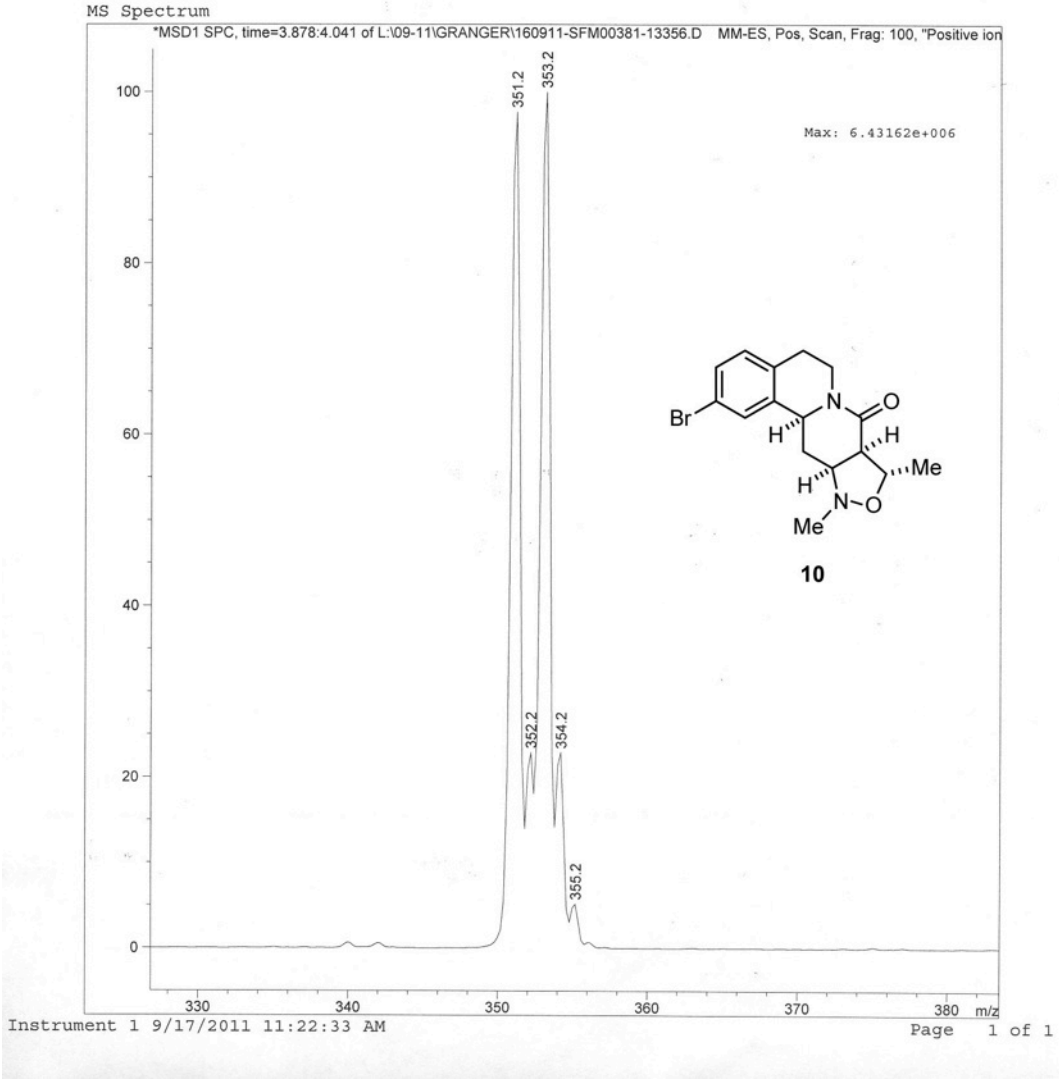
   1   3.857 MM    0.1121 2973.91821  442.11911  97.7411
   2   4.125 MM    0.1001   68.73178   10.16864   2.2589

Totals :                  3042.64999  452.28775
```



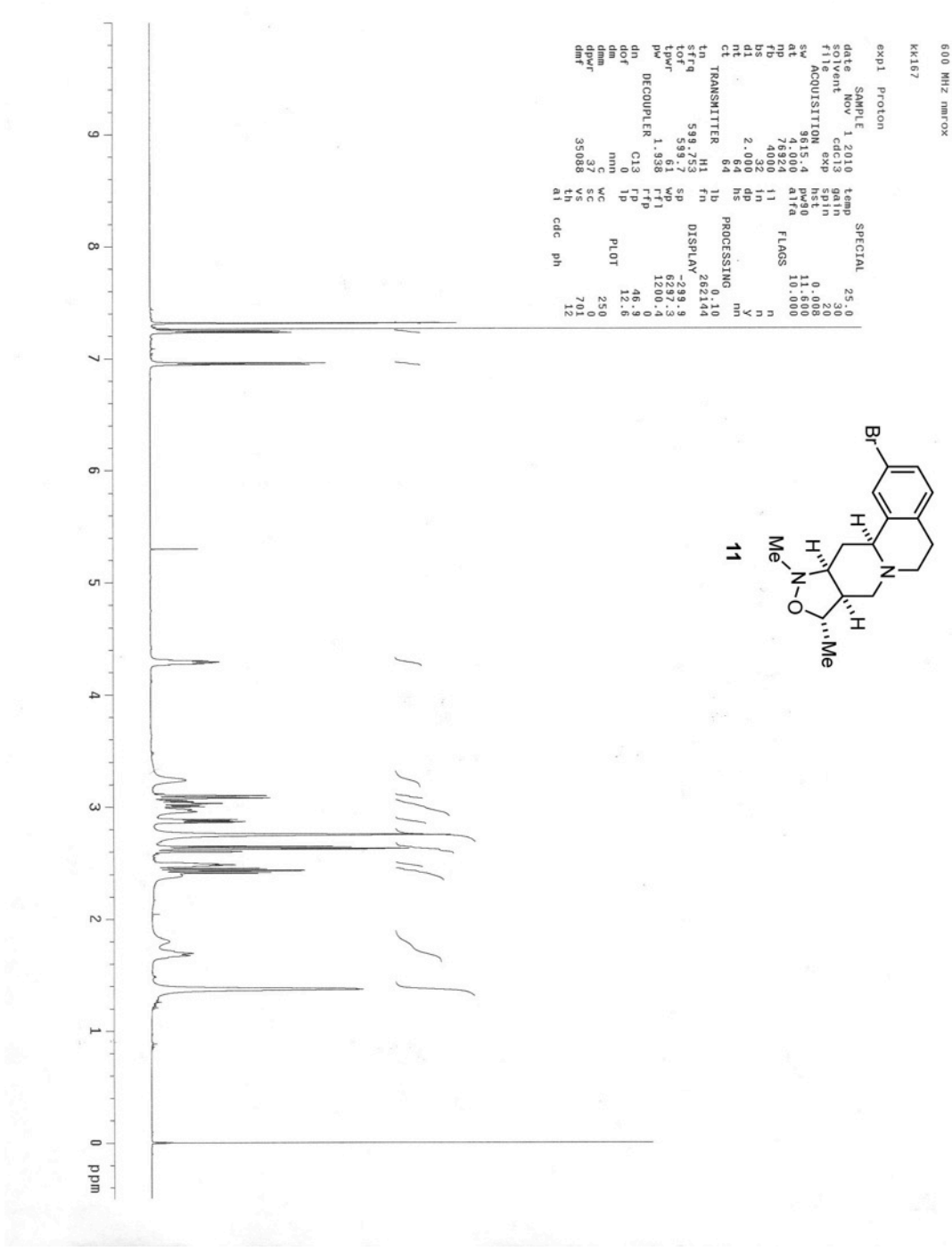
10

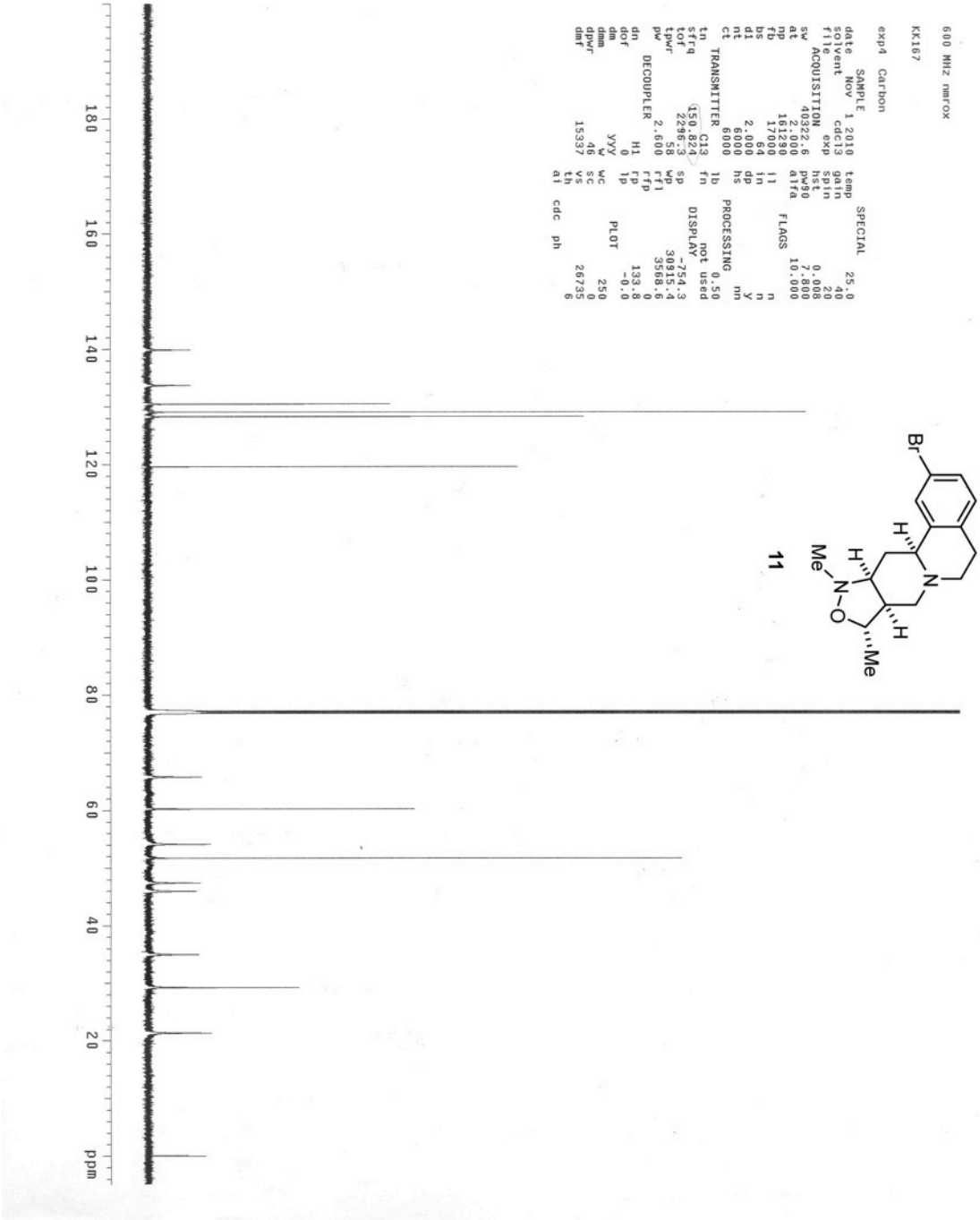
```
Print of window 79: MS Spectrum
Data File  : L:\09-11\GRANGER\160911-SFM00381-13356.D
Sample Name : SFM0038
=====================================================================
Acq. Operator   : brettag35@mail.utexas.edu
Acq. Instrument : LCMS                          Location : Vial 37
Injection Date  : 9/16/2011 10:17:10 PM              Inj :   1
                                                Inj Volume : 1.0 µl
Acq. Method     : C:\CHEM32\1\METHODS\SP NIH.M
Last changed    : 9/16/2011 10:16:49 PM by brettag35@mail.utexas.edu
                  (modified after loading)
Analysis Method : C:\CHEM32\1\METHODS\DEF_LC.M
Last changed    : 11/20/2006 4:14:44 AM
Sample Info     : Easy-Access Method: 'SP NIH'
```



Instrument 1 9/17/2011 11:22:33 AM

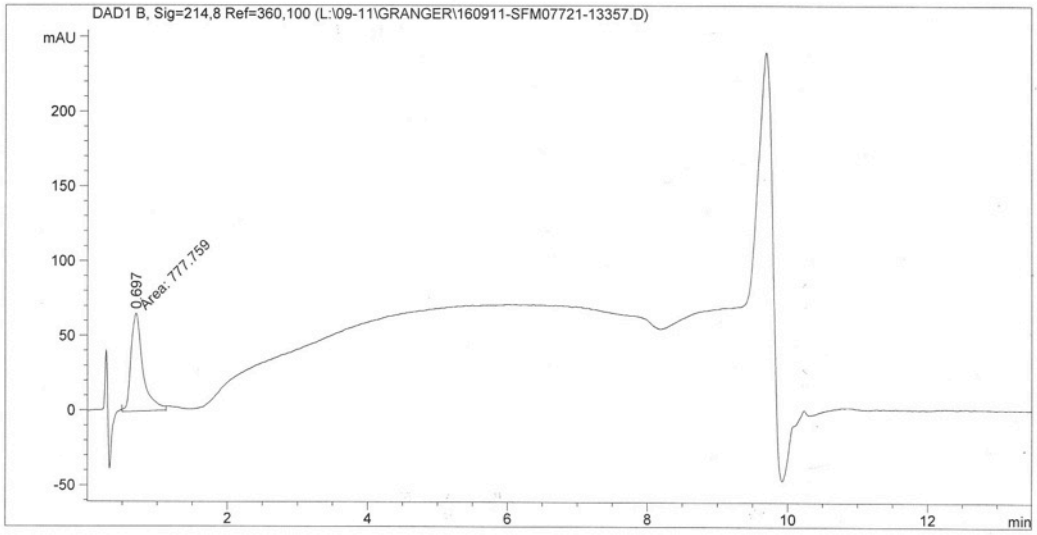
Page 1 of 1


600 MHz rmrox
kk167
exp1 Proton
SAMPLE
date Nov 1 2010
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 nn0
dm nnn
dmm c
dpwr 37
dmf 35088
temp
gain
spin
hst
pw90
a1fa
FLAGS
PROCESSING
lb
fn
DISPLAY
sp
wp
rfl
rfp
rp
lp
PLOT
wc
sc
vs
th
cdc
SPECIAL
25.0
30
20
0.008
11.600
10.000
n
n
y
nn
0.10
262144
-299.9
6297.3
1200.4
0
46.9
12.8
250
0
701
12
ph
Br
H
H
Me
N
N
O
H
Me
11
9
8
7
6
5
4
3
2
1
0
ppm


600 MHz rmrox
KK167
exp4 Carbon
SAMPLE
date Nov 1 2010
solvent cdcl3
file exp
ACQUISITION
sw 40322.6
at 2.000
np 161290
fb 17000
bs 64
bs 2
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 3568.6
rfp 0
rp 133.8
lp -0.0
PLOT
wc 250
sc 0
vs 26735
th 6
al
cdc
ph
Br
H
H
Me
N
O
N
H
Me
11
180
160
140
120
100
80
60
40
20
ppm

Data File L:\09-11\GRANGER\160911-SFM07721-13357.D
Sample Name: SFM0772

```
==========================================================================
Acq. Operator    : brettag35@mail.utexas.edu
Acq. Instrument : LCMS                          Location : Vial 38
Injection Date   : 9/16/2011 10:32:35 PM
                                                Inj Volume : 1.0 µl
Acq. Method      : C:\CHEM32\1\METHODS\SP NIH.M
Last changed     : 9/16/2011 10:32:14 PM by brettag35@mail.utexas.edu
                   (modified after loading)
Analysis Method : C:\CHEM32\1\METHODS\DEF_LC.M
Last changed     : 11/20/2006 4:14:44 AM
Sample Info      : Easy-Access Method: 'SP NIH'
```




```
==========================================================================
                          Area Percent Report
==========================================================================

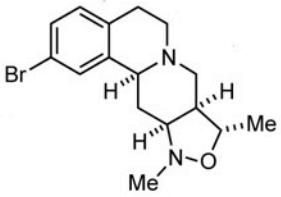
Sorted By             :      Signal
Multiplier:                   :      1.0000
Dilution:                     :      1.0000
Use Multiplier & Dilution Factor with ISTDs


Signal 1: DAD1 B, Sig=214,8 Ref=360,100

Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]   [mAU*s]     [mAU]       %
----|-------|----|-------|----------|----------|--------|
   1   0.697 MM    0.1970  777.75891   65.79211 100.0000

Totals :                   777.75891   65.79211

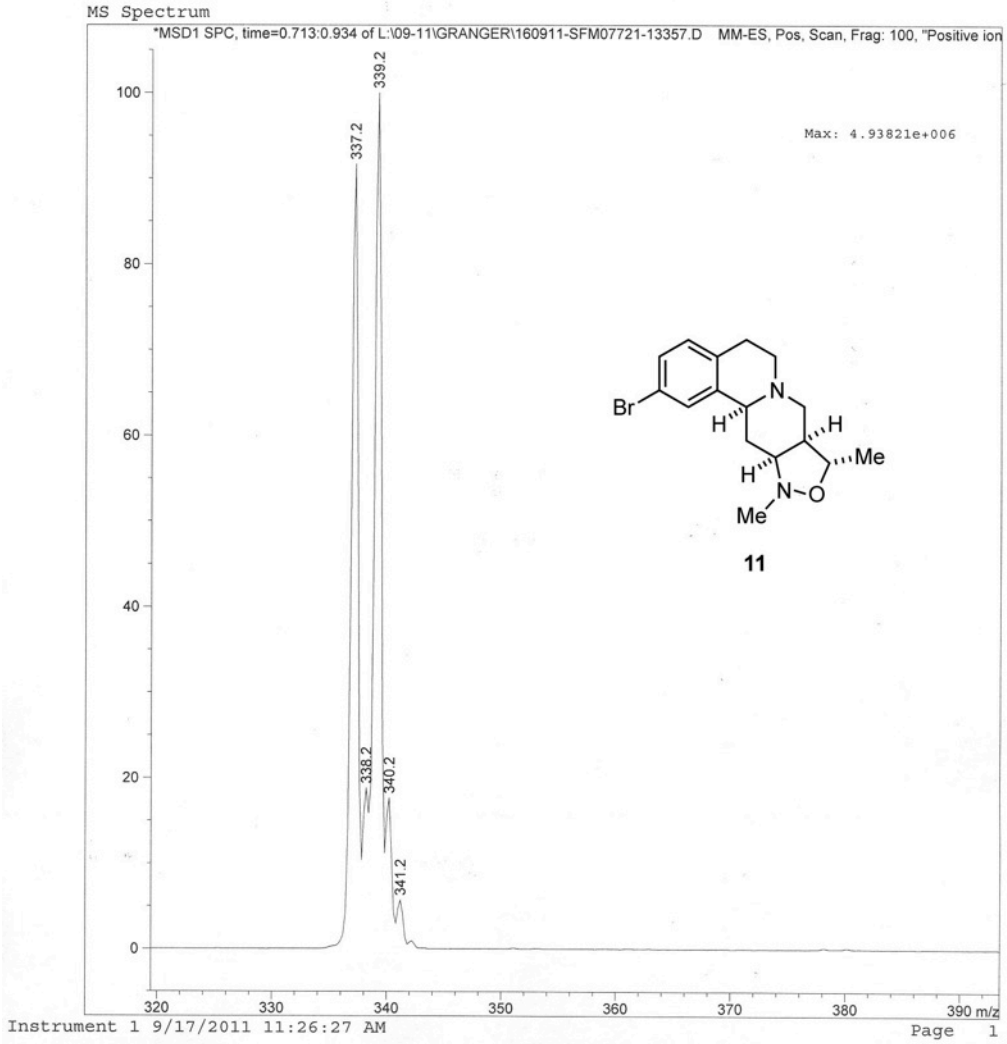
==========================================================================
```

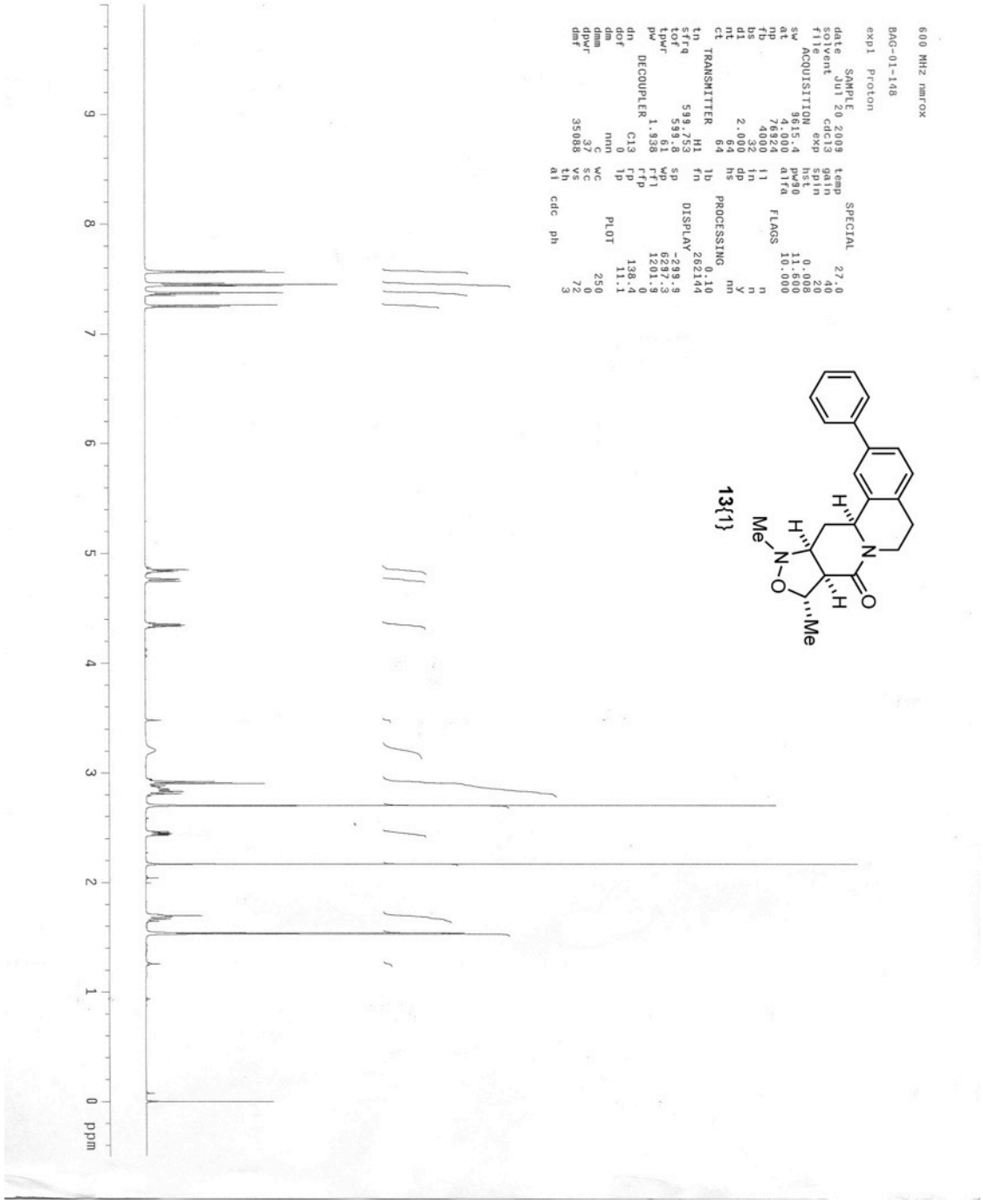


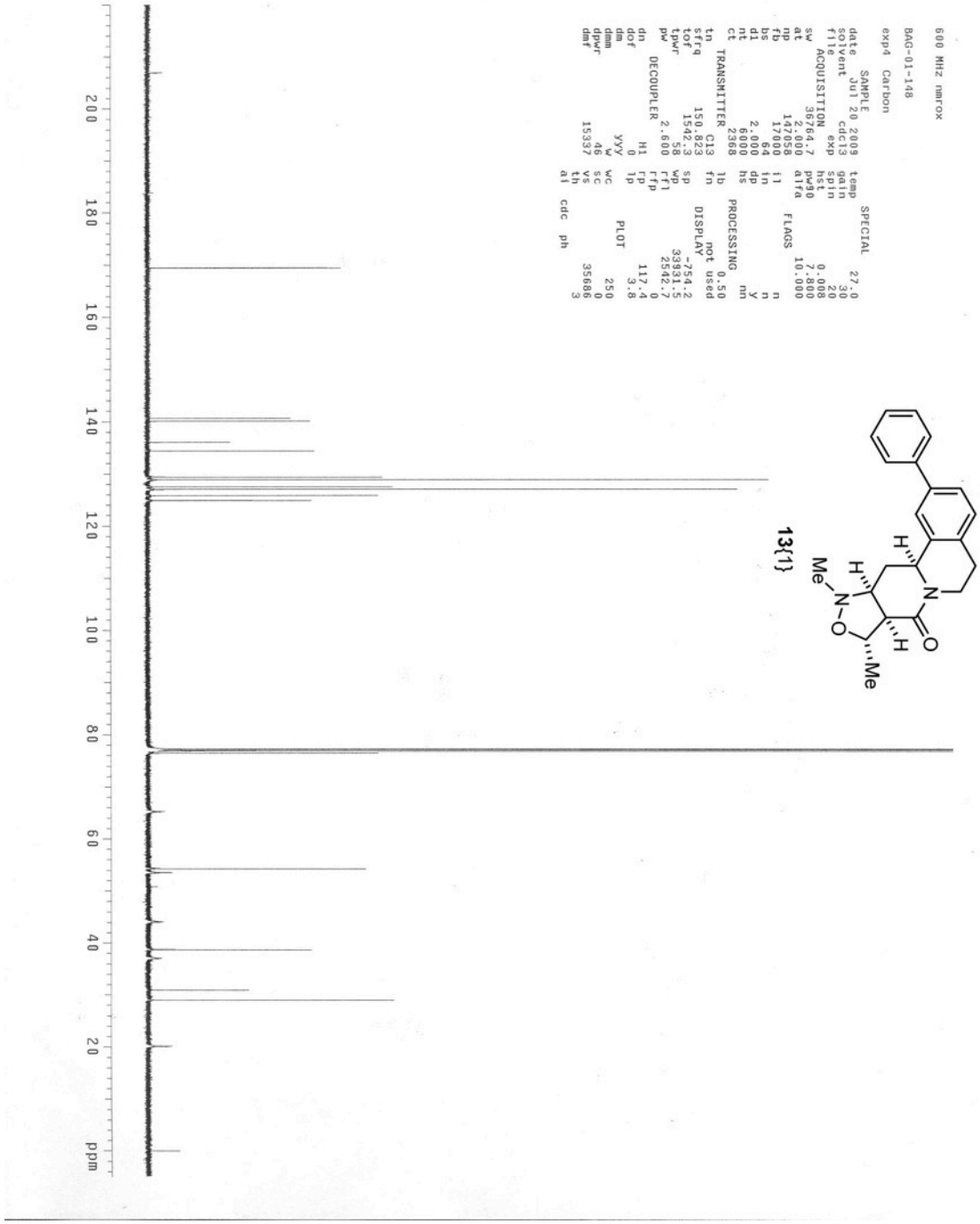
11

Instrument 1 9/17/2011 11:26:04 AM Page 1 of 1

```
Print of window 79: MS Spectrum
Data File  : L:\09-11\GRANGER\160911-SFM07721-13357.D
Sample Name : SFM0772
======================================================================
Acq. Operator   : brettag35@mail.utexas.edu
Acq. Instrument : LCMS                          Location : Vial 38
Injection Date  : 9/16/2011 10:32:35 PM              Inj :   1
                                                Inj Volume : 1.0 µl
Acq. Method     : C:\CHEM32\1\METHODS\SP NIH.M
Last changed    : 9/16/2011 10:32:14 PM by brettag35@mail.utexas.edu
                  (modified after loading)
Analysis Method : C:\CHEM32\1\METHODS\DEF_LC.M
Last changed    : 11/20/2006 4:14:44 AM
Sample Info     : Easy-Access Method: 'SP NIH'
```

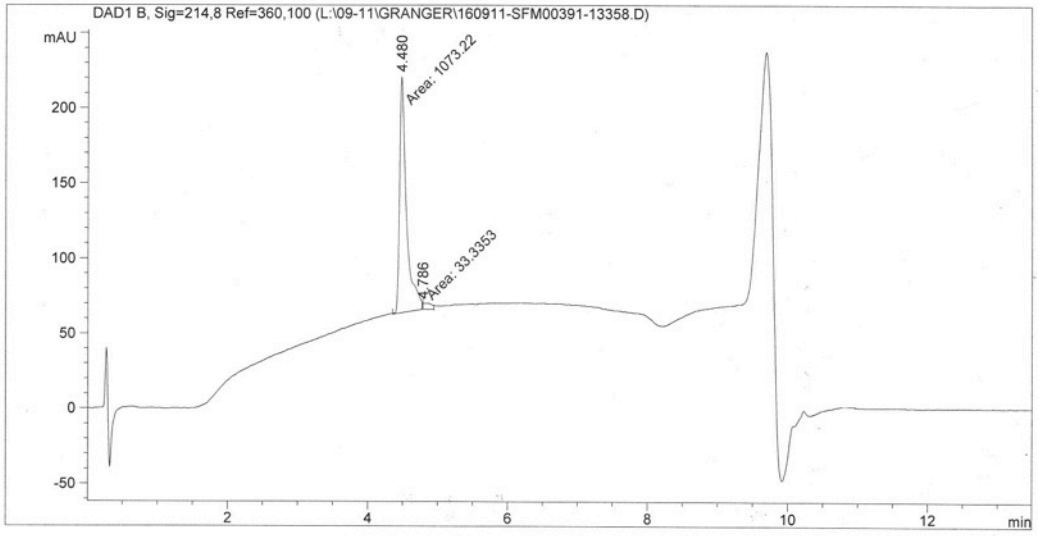



600 MHz nmrox
BAG-01-148
exp1 Proton
SAMPLE
date Jul 20 2009
solvent cdcl3
file exp
ACQUISITION
sw 9615.4
at 4.000
np 76924
fb 4000
bs 32
d1 2.000
nt 64
ct 64
TRANSMITTER
tn H1
sfrq 599.753
tof 599.8
tpwr 61
pw 1.938
DECOUPLER
dn C13
dof 0
dm nnn
dmm c
dpwr 37
dmf 35088
temp 27.0
gain 40
spin 20
hst 0.008
ppm 11.008
dfrq 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 1201.9
rfp 0
rp 138.4
lp 11.1
PLOT
wc 250
sc 0
vs 72
th 3
al cdc ph
13{1}
Me
Me
H
H
H
N
N
O
O
9
8
7
6
5
4
3
2
1
0 ppm


600 MHz nmrox
BAG-01-148
exp4 Carbon
SAMPLE
date Jul 20 2009
solvent cdcl3
file
ACQUISITION
sw 36764.7
at 2.000
np 147058
fb 17000
bs 64
d1 2.000
nt 6000
ct 2368
TRANSMITTER
tn C13
sfrq 150.823
tof 1542.3
tpwr 58
pw 2.600
DECOUPLER
dn H1
dof 0
dm yyy
dmm w
dpwr 46
dmf 15337
SPECIAL
temp 27.0
gain 30
spin 20
hst 0.008
ph90 7.800
alfa 10.000
FLAGS
il n
in n
dp y
hs nn
PROCESSING
lb 0.50
fn not used
DISPLAY
sp -754.2
wp 33931.5
rfl 2542.7
rfp 0
rp 117.4
lp 3.8
PLOT
wc 250
sc 0
vs 35686
th 3
al cdc ph
13{1}
Me
Me
H
H
H
N
N
O
O
200
180
160
140
120
100
80
60
40
20
ppm

Data File L:\09-11\GRANGER\160911-SFM00391-13358.D
Sample Name: SFM0039

```
=====================================================================
Acq. Operator    : brettag35@mail.utexas.edu
Acq. Instrument  : LCMS                          Location : Vial 39
Injection Date   : 9/16/2011 10:47:42 PM
                                                 Inj Volume : 1.0 µl
Acq. Method      : C:\CHEM32\1\METHODS\SP NIH.M
Last changed     : 9/16/2011 10:47:26 PM by brettag35@mail.utexas.edu
                   (modified after loading)
Analysis Method  : C:\CHEM32\1\METHODS\DEF_LC.M
Last changed     : 11/20/2006 4:14:44 AM
Sample Info      : Easy-Access Method: 'SP NIH'
```



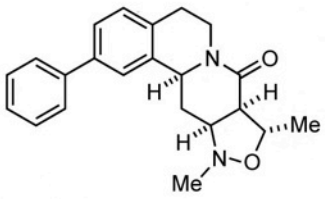

```
=====================================================================
                        Area Percent Report
=====================================================================

Sorted By             :      Signal
Multiplier:                   :      1.0000
Dilution:                     :      1.0000
Use Multiplier & Dilution Factor with ISTDs


Signal 1: DAD1 B, Sig=214,8 Ref=360,100

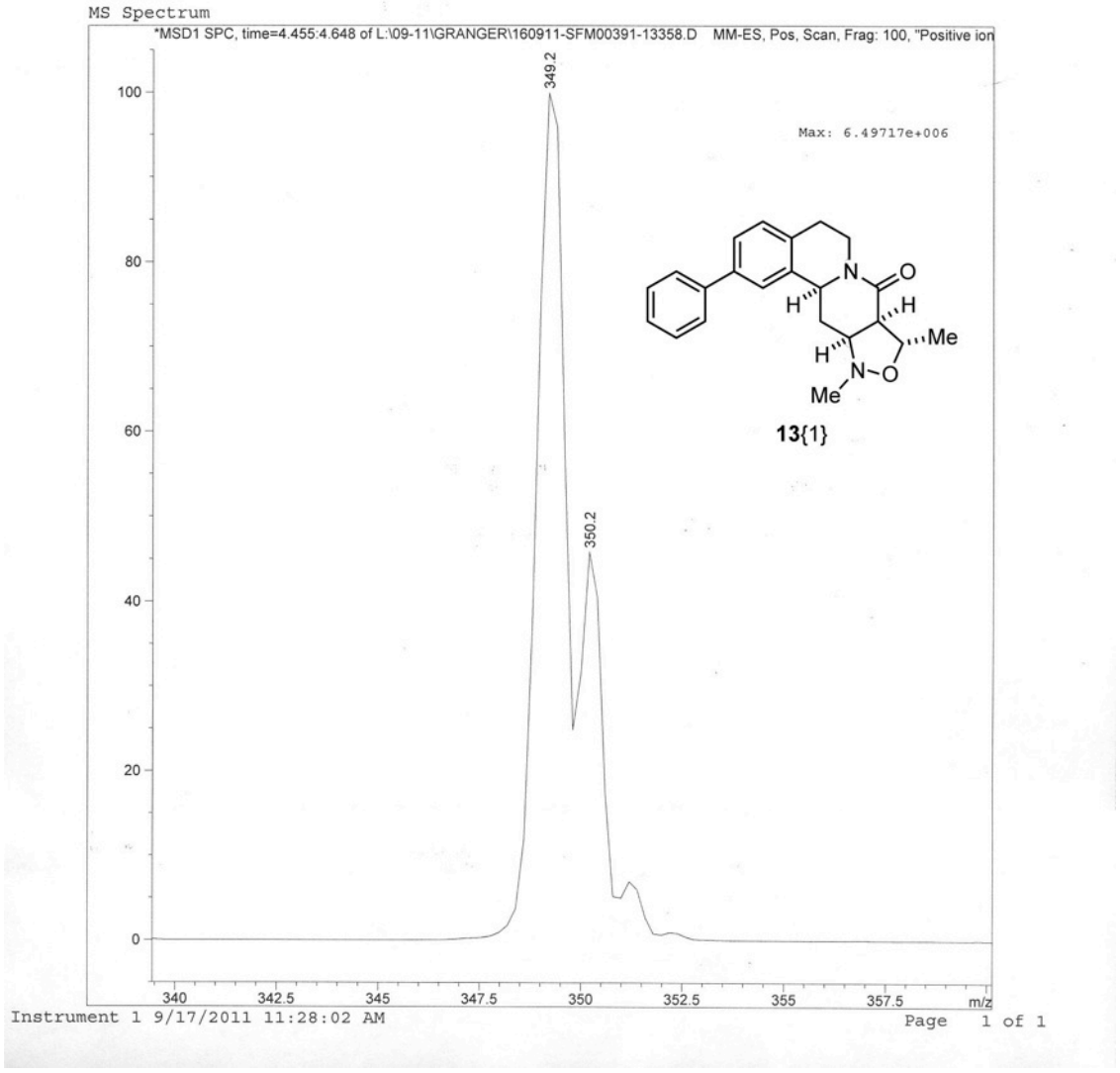
Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]    [mAU*s]     [mAU]      %
----|-------|----|-------|----------|----------|--------|
   1   4.480 MM    0.1135 1073.22461  157.65465  96.9875
   2   4.786 MM    0.1069   33.33529    4.29844   3.0125

Totals :                  1106.55990  161.95308
```

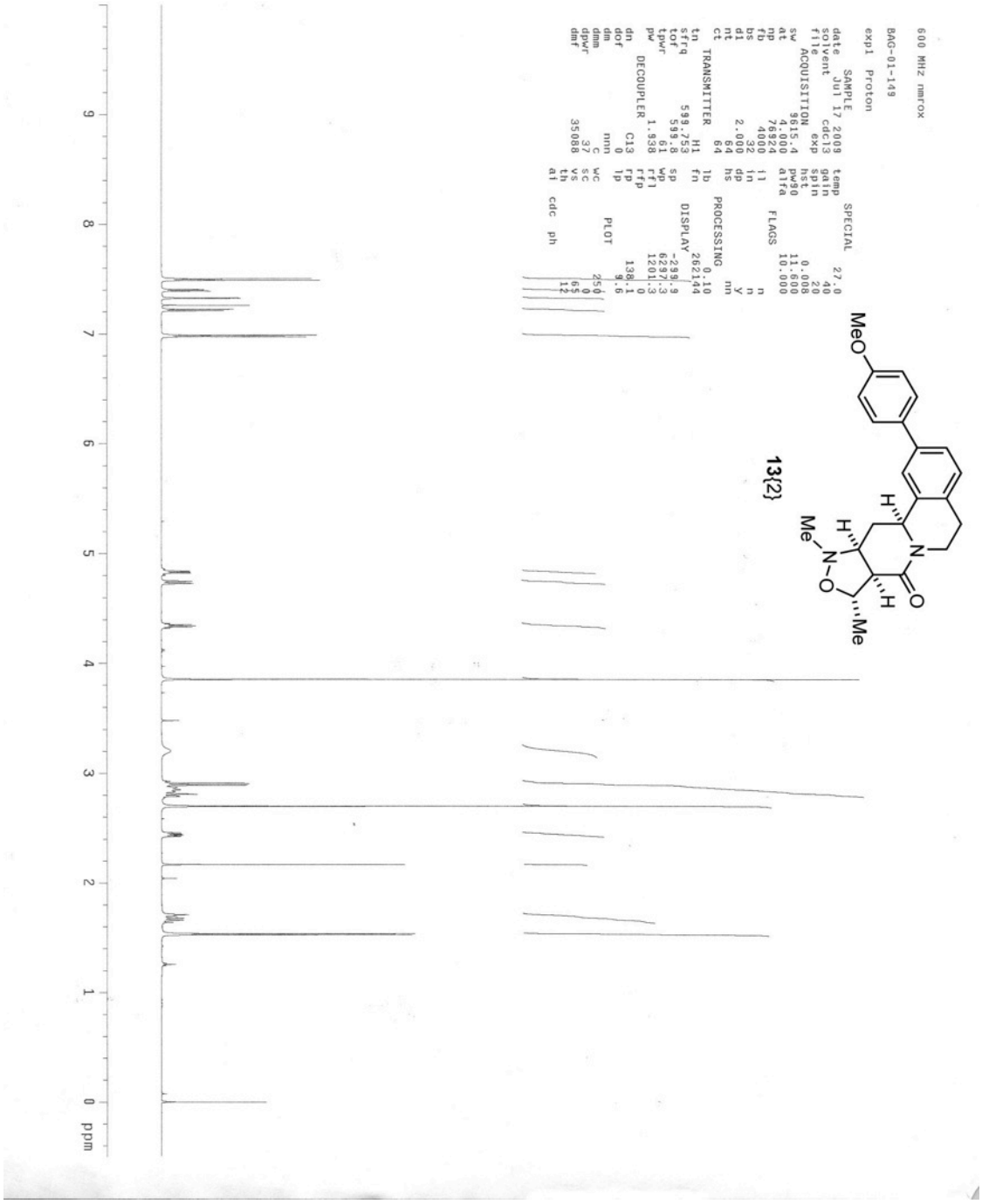


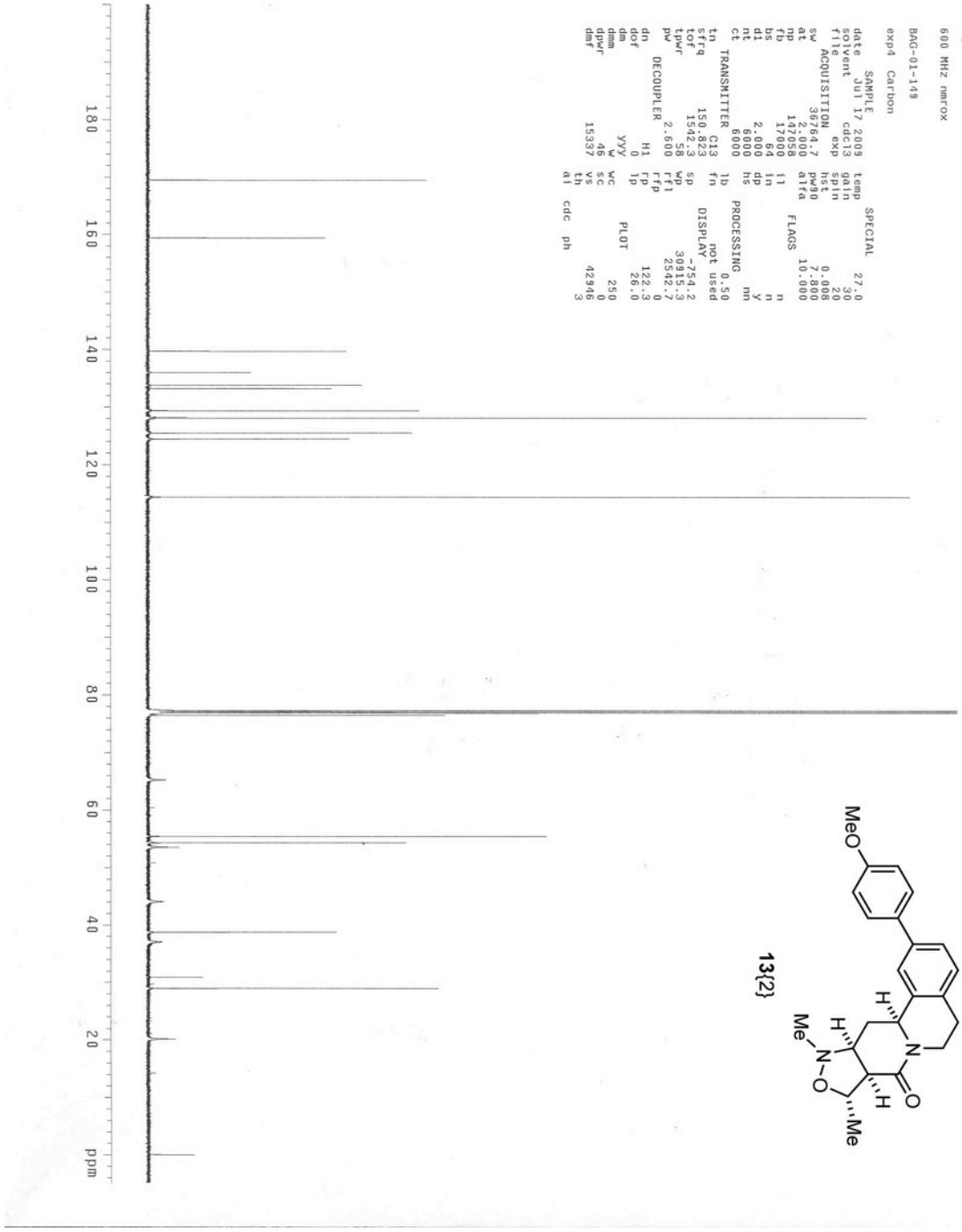
13{1}

```
Print of window 79: MS Spectrum
Data File  : L:\09-11\GRANGER\160911-SFM00391-13358.D
Sample Name : SFM0039
======================================================================
Acq. Operator   : brettag35@mail.utexas.edu
Acq. Instrument : LCMS                          Location : Vial 39
Injection Date  : 9/16/2011 10:47:42 PM              Inj :   1
                                                Inj Volume : 1.0 µl
Acq. Method     : C:\CHEM32\1\METHODS\SP NIH.M
Last changed    : 9/16/2011 10:47:26 PM by brettag35@mail.utexas.edu
                  (modified after loading)
Analysis Method : C:\CHEM32\1\METHODS\DEF_LC.M
Last changed    : 11/20/2006 4:14:44 AM
Sample Info     : Easy-Access Method: 'SP NIH'
```



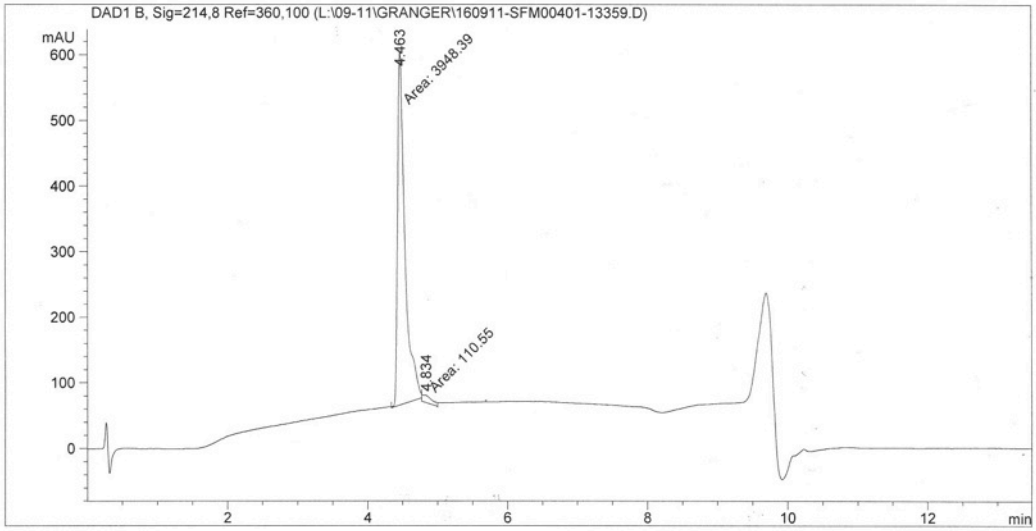
13{1}


600 MHz nmrox
BAG-01-149
exp1 Proton
SAMPLE
date Jul 17 2009
solvent cdcl3
file
ACQUISITION
sw 6515.4
at 4.000
np 76924
fb 4000
bs 32
d1 2.000
nt 64
ct 64
TRANSMITTER
tn H1
sfrq 599.753
tof 599.8
tpwr 61
pw 1.938
DECOUPLER
dn C13
dof 0
dm nnn
dmm c
dpwr 37
dmf 35088
temp SPECIAL 27.0
gain 40
spin 20
hst 0.008
pw90 11.000
alfa 10.000
FLAGS
ii n
in n
dp y
hs nn
PROCESSING
lb 0.10
fn 26214
DISPLAY
sp -299.9
wp 6297.3
rfl 1201.3
rfp 0
rp 138.1
lp 9.6
PLOT
wc 250
sc 0
vs 65
th 12
al
cdc ph
MeO
H
H
Me
N
O
O
H
Me
13{2}
9
8
7
6
5
4
3
2
1
0 ppm


600 MHz nmrox
BAG-01-149
exp4 Carbon
SAMPLE
date Jul 17 2009
solvent cdcl3
file exp
ACQUISITION
sw 38764.7
at 2.000
np 147058
fb 17000
bs 64
d1 2.000
nt 6000
ct 6000
TRANSMITTER
tn C13
sfrq 150.823
tof 1542.3
tpwr 58
pw 2.600
DECOUPLER
dn H1
dof 0
dm yyy
dmm w
dpwr 46
dmf 15337
SPECIAL
temp 27.0
gain 30
spin 20
hst 0.008
ph90 7.000
atfa 10.000
FLAGS
il n
in n
dp y
hs nn
PROCESSING
lb 0.50
fn not used
DISPLAY
sp -754.2
wp 3915.3
rfl 2542.7
rfp 0
rp 122.3
lp 26.0
PLOT
wc 250
sc 0
vs 42946
th 3
al cdc ph
MeO
H
N
O
H
Me
N
O
H
Me
13{2}
180
160
140
120
100
80
60
40
20
ppm

Data File L:\09-11\GRANGER\160911-SFM00401-13359.D
Sample Name: SFM0040

```
    ======================================================================
    Acq. Operator   : brettag35@mail.utexas.edu
    Acq. Instrument : LCMS                          Location : Vial 40
    Injection Date  : 9/16/2011 11:02:41 PM
                                                   Inj Volume : 1.0 µl
    Acq. Method     : C:\CHEM32\1\METHODS\SP NIH.M
    Last changed    : 9/16/2011 11:02:26 PM by brettag35@mail.utexas.edu
                      (modified after loading)
    Analysis Method : C:\CHEM32\1\METHODS\DEF_LC.M
    Last changed    : 11/20/2006 4:14:44 AM
    Sample Info     : Easy-Access Method: 'SP NIH'
```



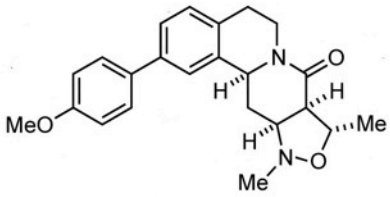

```
    ======================================================================
                              Area Percent Report
    ======================================================================

    Sorted By             :      Signal
    Multiplier:                   :      1.0000
    Dilution:                     :      1.0000
    Use Multiplier & Dilution Factor with ISTDs


    Signal 1: DAD1 B, Sig=214,8 Ref=360,100

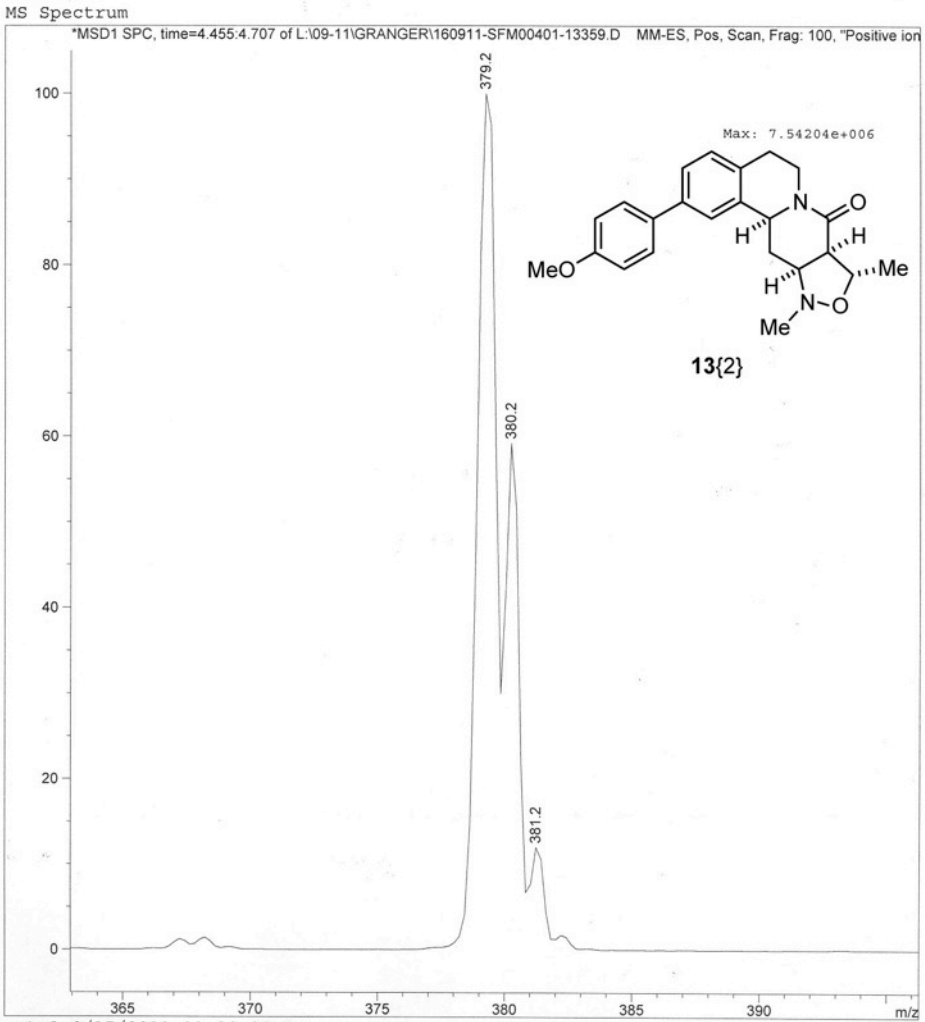
    Peak RetTime Type  Width     Area      Height     Area
      #   [min]        [min]   [mAU*s]     [mAU]       %
    ----|-------|----|-------|----------|----------|--------|
       1   4.463 MM    0.1212 3948.38672  542.93597  97.2764
       2   4.834 MM    0.1661  110.54974   11.09314   2.7236

    Totals :                  4058.93646  554.02912
```

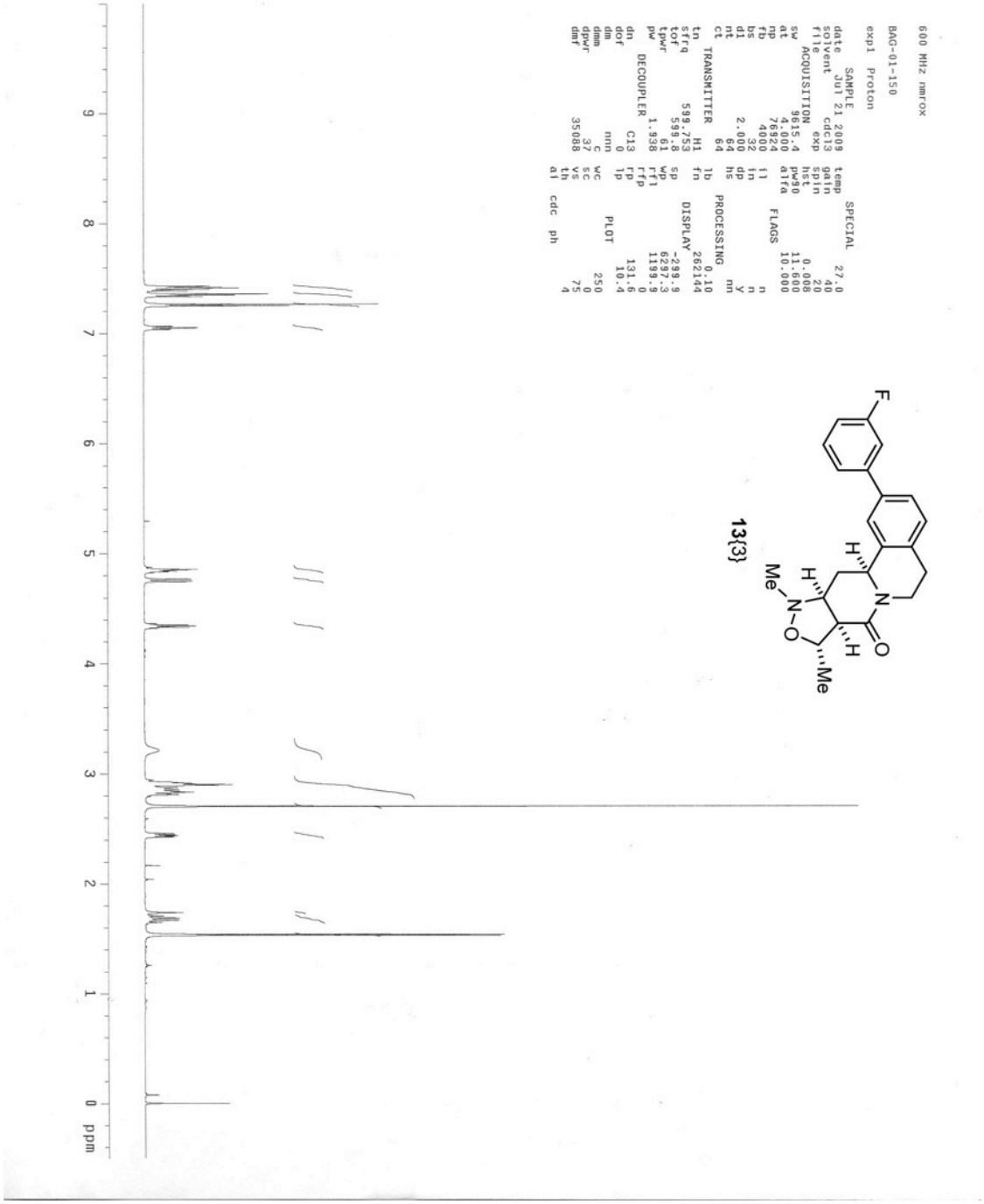


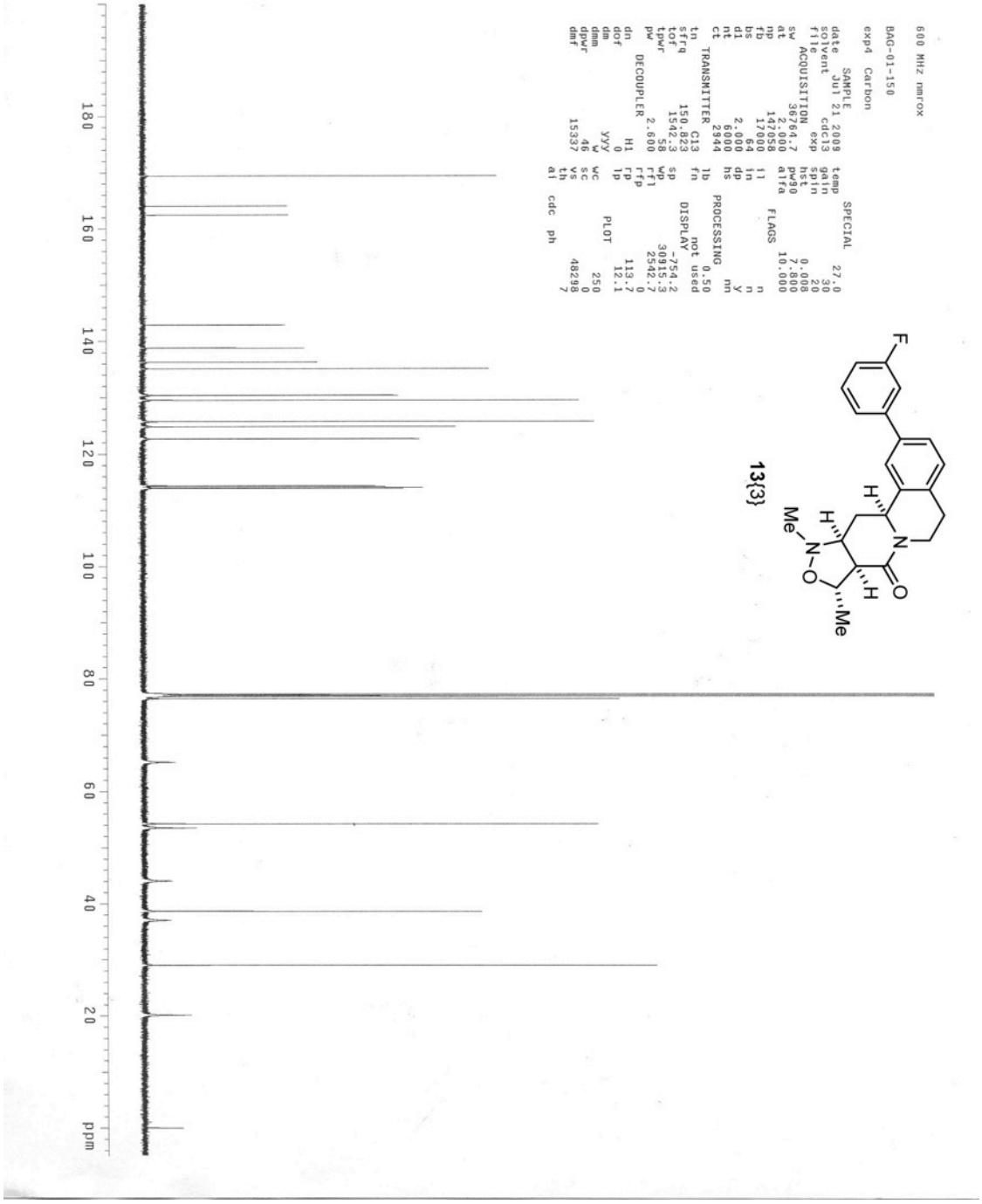
13{2}

```
Print of window 79: MS Spectrum
Data File  : L:\09-11\GRANGER\160911-SFM00401-13359.D
Sample Name : SFM0040
======================================================================
Acq. Operator   : brettag35@mail.utexas.edu
Acq. Instrument : LCMS                          Location : Vial 40
Injection Date  : 9/16/2011 11:02:41 PM              Inj :   1
                                                Inj Volume : 1.0 µl
Acq. Method     : C:\CHEM32\1\METHODS\SP NIH.M
Last changed    : 9/16/2011 11:02:26 PM by brettag35@mail.utexas.edu
                  (modified after loading)
Analysis Method : C:\CHEM32\1\METHODS\DEF_LC.M
Last changed    : 11/20/2006 4:14:44 AM
Sample Info     : Easy-Access Method: 'SP NIH'
```



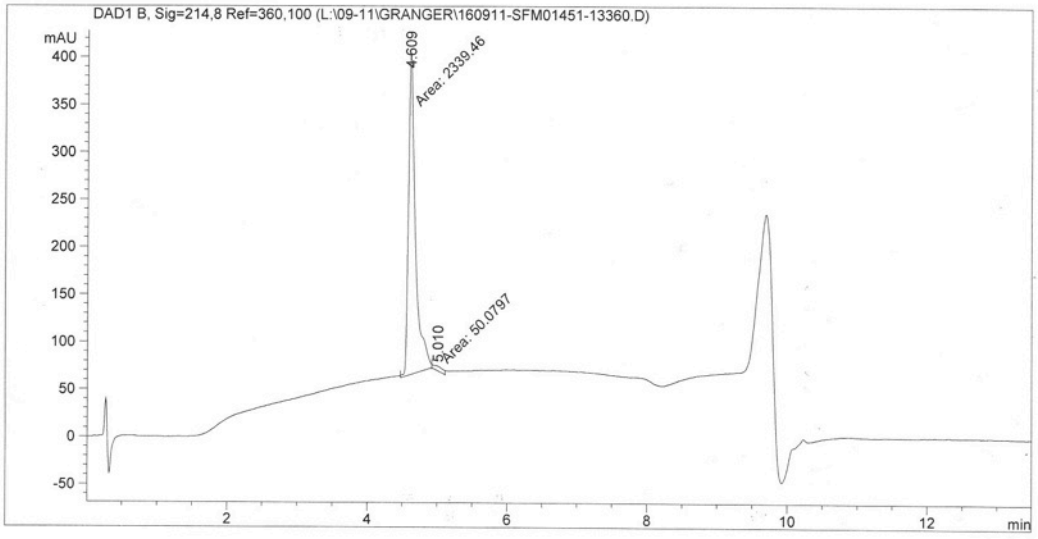
13{2}


600 MHz nmrox
BAG-01-150
exp1 Proton
SAMPLE
date Jul 21 2009
solvent cdcl3
file
ACQUISITION
sw 9615.4
at 1.000
np 76924
4000
fb
bs 32
d1 2.000
nt 64
ct 64
TRANSMITTER
tn H1
sfrq 599.753
599.8
tof 61
tpwr
pw 1.938
DECOUPLER
dn C13
dof 0
dm nnn
dmm c
dpwr 37
dmf 35088
temp SPECIAL 27.0
gain 40
spin 20
hst 0.008
pw90 11.000
alfa 10.000
FLAGS
i1 n
in n
dp y
hs nn
PROCESSING
lb 0.10
fn 262144
DISPLAY
sp -299.9
wp 6297.3
rfl 1199.9
rfp 0
rp 131.6
lp 10.4
PLOT
wc 250
sc 0
vs 75
th 4
al cdc ph
F
H
N
O
H
Me
N
O
Me
13{3}
9
8
7
6
5
4
3
2
1
0 ppm


600 MHz nmrox
BAG-01-150
exp4 Carbon
SAMPLE
date Jul 21 2009
solvent cdcl3
file exp
ACQUISITION
sw 36764.7
at 2.000
np 147058
fb 17000
bs 64
d1 2.000
nt 6000
ct 2944
TRANSMITTER
tn C13
sfrq 150.823
tof 1542.3
tpwr 58
pw 2.600
DECOUPLER
dn H1
dof 0
dm yyy
dmm w
dmf 46
dpwr 15337
SPECIAL
temp 27.0
gain 30
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 -754.2
wp 3015.3
rfl 2542.7
rfp 0
rp 113.7
lp 12.1
PLOT
wc 250
sc 0
vs 48298
th 7
at
cdc
ph
13{3}
F
H
N
O
H
Me
N
O
H
Me
180
160
140
120
100
80
60
40
20
ppm

Data File L:\09-11\GRANGER\160911-SFM01451-13360.D
Sample Name: SFM0145

```
=====================================================================
Acq. Operator    : brettag35@mail.utexas.edu
Acq. Instrument  : LCMS                          Location : Vial 41
Injection Date   : 9/16/2011 11:17:44 PM
                                                 Inj Volume : 1.0 µl
Acq. Method      : C:\CHEM32\1\METHODS\SP NIH.M
Last changed     : 9/16/2011 11:17:29 PM by brettag35@mail.utexas.edu
                   (modified after loading)
Analysis Method  : C:\CHEM32\1\METHODS\DEF_LC.M
Last changed     : 11/20/2006 4:14:44 AM
Sample Info      : Easy-Access Method: 'SP NIH'
```



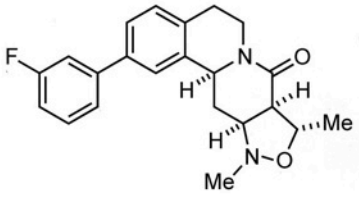

```
=====================================================================
                        Area Percent Report
=====================================================================

Sorted By             :      Signal
Multiplier:                   :      1.0000
Dilution:                     :      1.0000
Use Multiplier & Dilution Factor with ISTDs


Signal 1: DAD1 B, Sig=214,8 Ref=360,100

Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]   [mAU*s]     [mAU]       %
----|-------|----|-------|----------|----------|--------|
   1   4.609 MM    0.1145 2339.45825  340.49762  97.9042
   2   5.010 MM    0.1514   50.07967    4.97597   2.0958

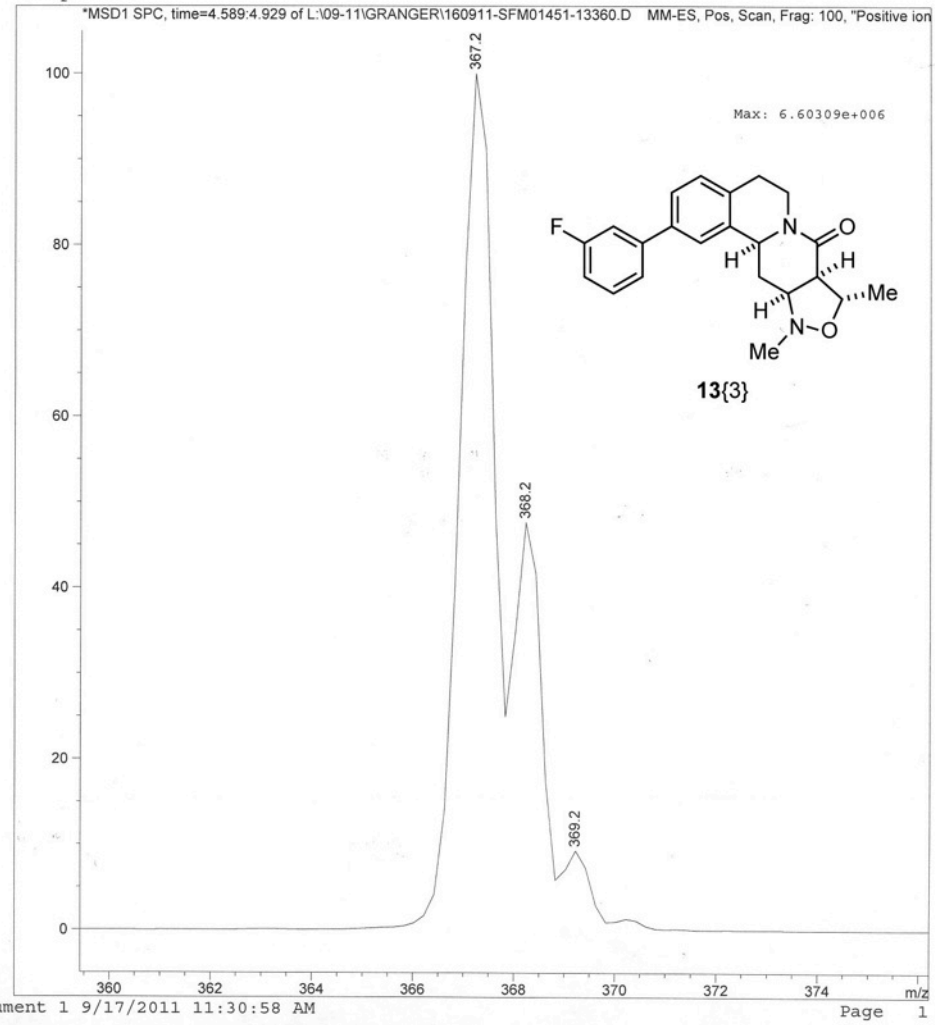
Totals :                  2389.53792  345.47359
```



13{3}

```
Print of window 79: MS Spectrum
Data File  : L:\09-11\GRANGER\160911-SFM01451-13360.D
Sample Name : SFM0145
======================================================================
Acq. Operator   : brettag35@mail.utexas.edu
Acq. Instrument : LCMS                          Location : Vial 41
Injection Date  : 9/16/2011 11:17:44 PM              Inj :   1
                                                Inj Volume : 1.0 µl
Acq. Method     : C:\CHEM32\1\METHODS\SP NIH.M
Last changed    : 9/16/2011 11:17:29 PM by brettag35@mail.utexas.edu
                  (modified after loading)
Analysis Method : C:\CHEM32\1\METHODS\DEF_LC.M
Last changed    : 11/20/2006 4:14:44 AM
Sample Info     : Easy-Access Method: 'SP NIH'
```

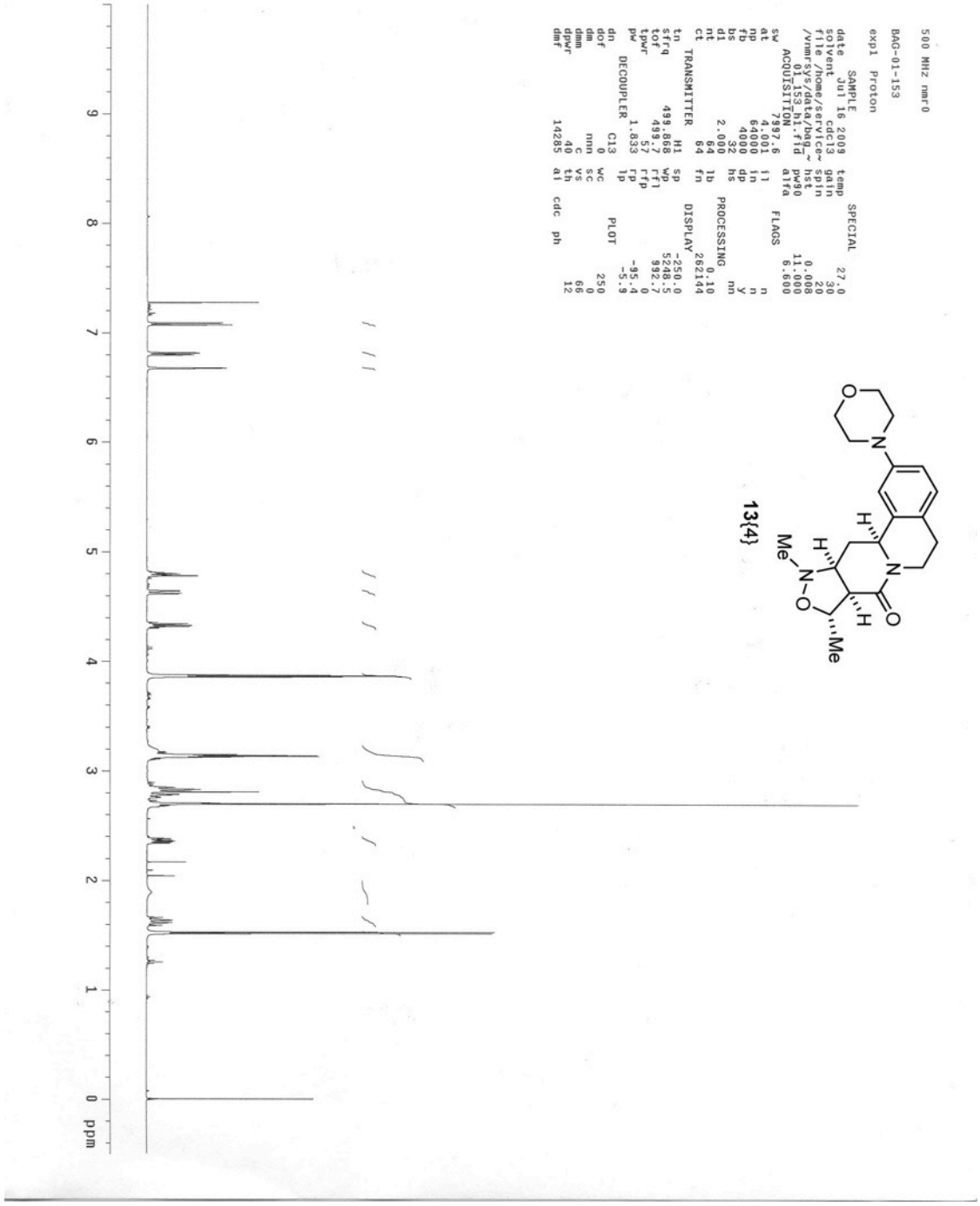
MS Spectrum

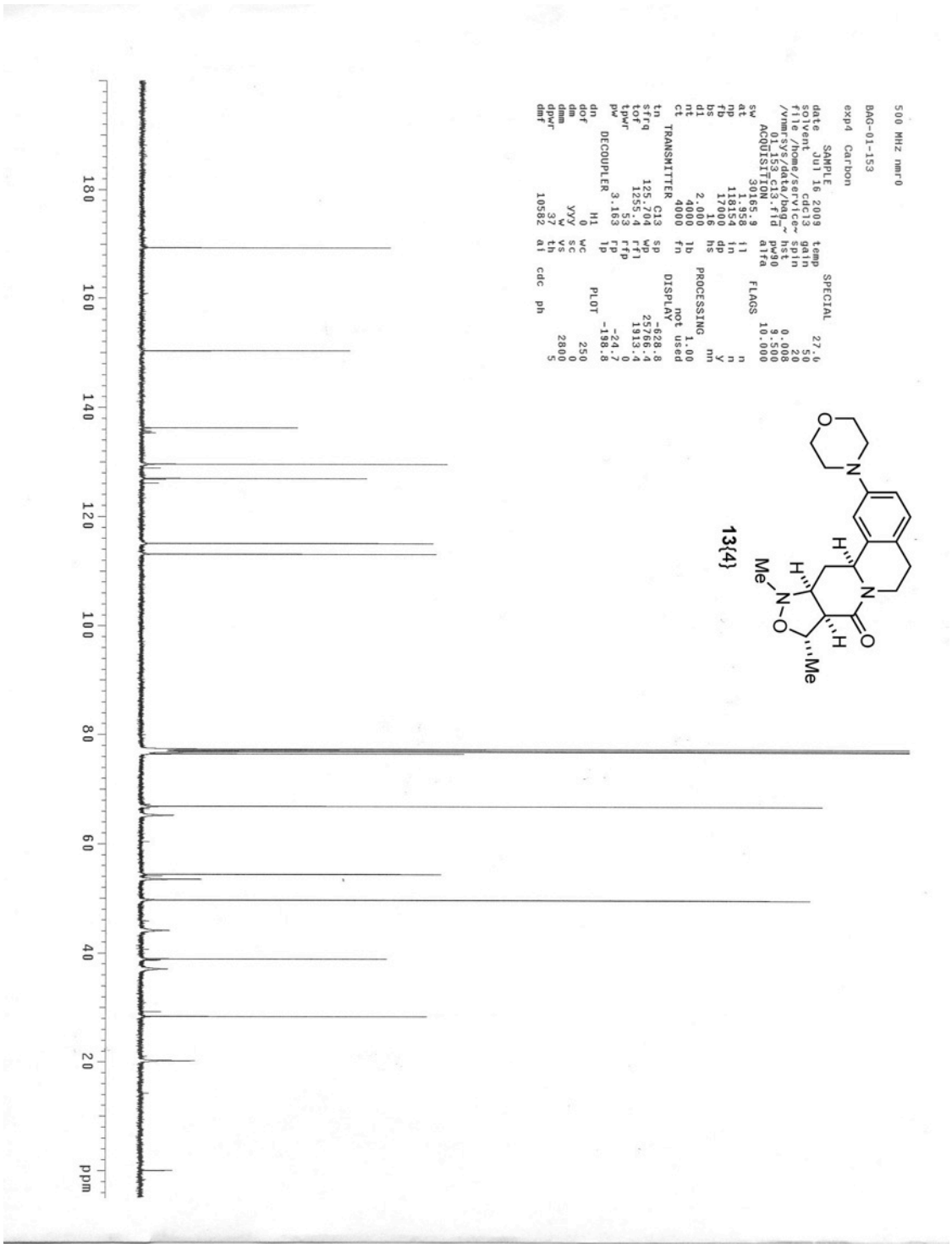


13{3}

Instrument 1 9/17/2011 11:30:58 AM

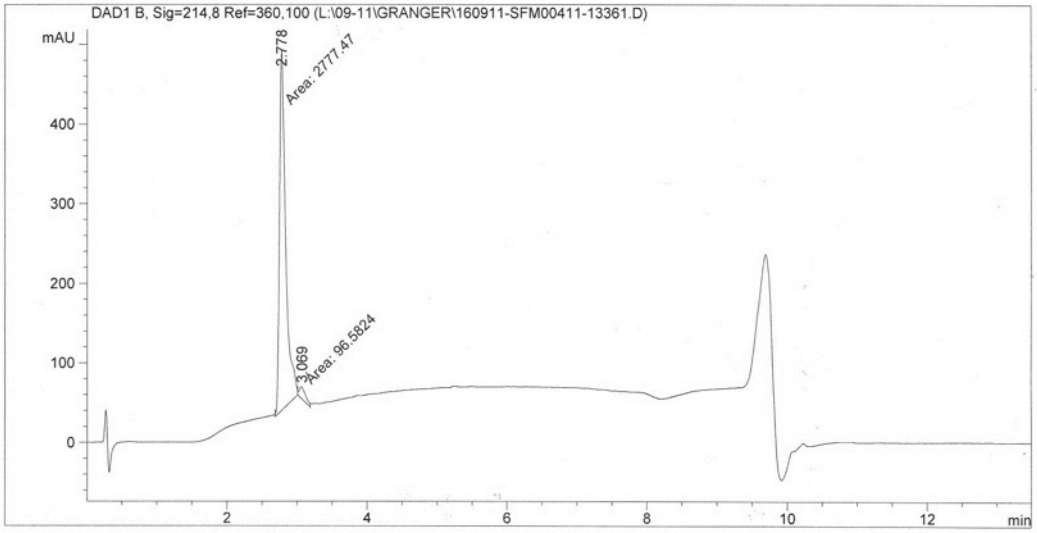
Page 1 of 1


500 MHz nmr0
BAG-01-153
exp1 Proton
date SAMPLE 2009 temp SPECIAL 27.0
solvent Jul 16 cdcl3 gain 30
file /home/service~ spin 20
/vnmrsys/data/bag~ hst 0.008
01-153_h1.fid pw90 11.000
ACQUISITION FLAGS 6.600
sw 7997.6
at 4.001 fl n
np 64000 in n
fb 40032 dp y
bs 32 hs nn
d1 2.000 PROCESSING
nt 64 lb 0.10
ct 64 fn 262144
TRANSMITTER DISPLAY
tn H1 sp -250.0
sfrq 499.868 wp 5248.5
tof 499.7 rfl 992.7
tpwr 57 rfp 0
pw 1.833 lp -95.0
DECOUPLER PLOT -5.9
dn C13
dof 0 wc 250
dm nnn sc 0
dmm c vs 66
dpwr 40 th 12
dmf 14285 al cdc ph
13{4}
Me
Me
9
8
7
6
5
4
3
2
1
0 ppm


500 MHz nmr0
BAG-01-153
exp4 Carbon
date SAMPLE 2009 temp SPECIAL 27.4
solvent jul 16 cdcl3 gain 50
file /home/service~ spin 20
/vnmrsys/data/baq~ hst 0.008
01_153_c13.fid pw90 9.500
ACQUISITION FLAGS 10.000
sw 30165.9 fl n
at 1.958 in n
np 118154 dp y
fb 17000 hs nn
bs 160 PROCESSING
d1 2.000 1b 1.00
nt 2000 fn not used
ct 4000 DISPLAY
TRANSMITTER -628.8
tn C13 sp 25766.4
sfrq 125.704 wp 1913.4
tof 1255.4 rfl 0
tpwr 53 rfp -24.7
pw 3.163 lp -198.8
DECOUPLER PLOT
dn H1 250
dof 0 wc 0
dm yyy sc 2800
dmm w vs 5
dpwr 37 th
dmf 10582 al cdc ph
13(4)
Me
Me
180
160
140
120
100
80
60
40
20
ppm

Data File L:\09-11\GRANGER\160911-SFM00411-13361.D
Sample Name: SFM0041

```
    ======================================================================
    Acq. Operator    : brettag35@mail.utexas.edu
    Acq. Instrument  : LCMS                              Location : Vial 42
    Injection Date   : 9/16/2011 11:32:45 PM
                                                      Inj Volume : 1.0 µl
    Acq. Method      : C:\CHEM32\1\METHODS\SP NIH.M
    Last changed     : 9/16/2011 11:32:30 PM by brettag35@mail.utexas.edu
                       (modified after loading)
    Analysis Method  : C:\CHEM32\1\METHODS\DEF_LC.M
    Last changed     : 11/20/2006 4:14:44 AM
    Sample Info      : Easy-Access Method: 'SP NIH'
```



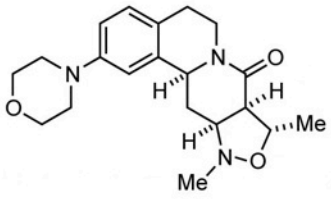

```
    ======================================================================
                              Area Percent Report
    ======================================================================

    Sorted By             :      Signal
    Multiplier:                   :      1.0000
    Dilution:                     :      1.0000
    Use Multiplier & Dilution Factor with ISTDs


    Signal 1: DAD1 B, Sig=214,8 Ref=360,100

    Peak RetTime Type  Width     Area      Height     Area
      #   [min]        [min]   [mAU*s]     [mAU]       %
    ----|-------|----|-------|----------|----------|--------|
       1   2.778 MM    0.1017 2777.47241  455.14041  96.6395
       2   3.069 MM    0.1066   96.58239   15.10584   3.3605

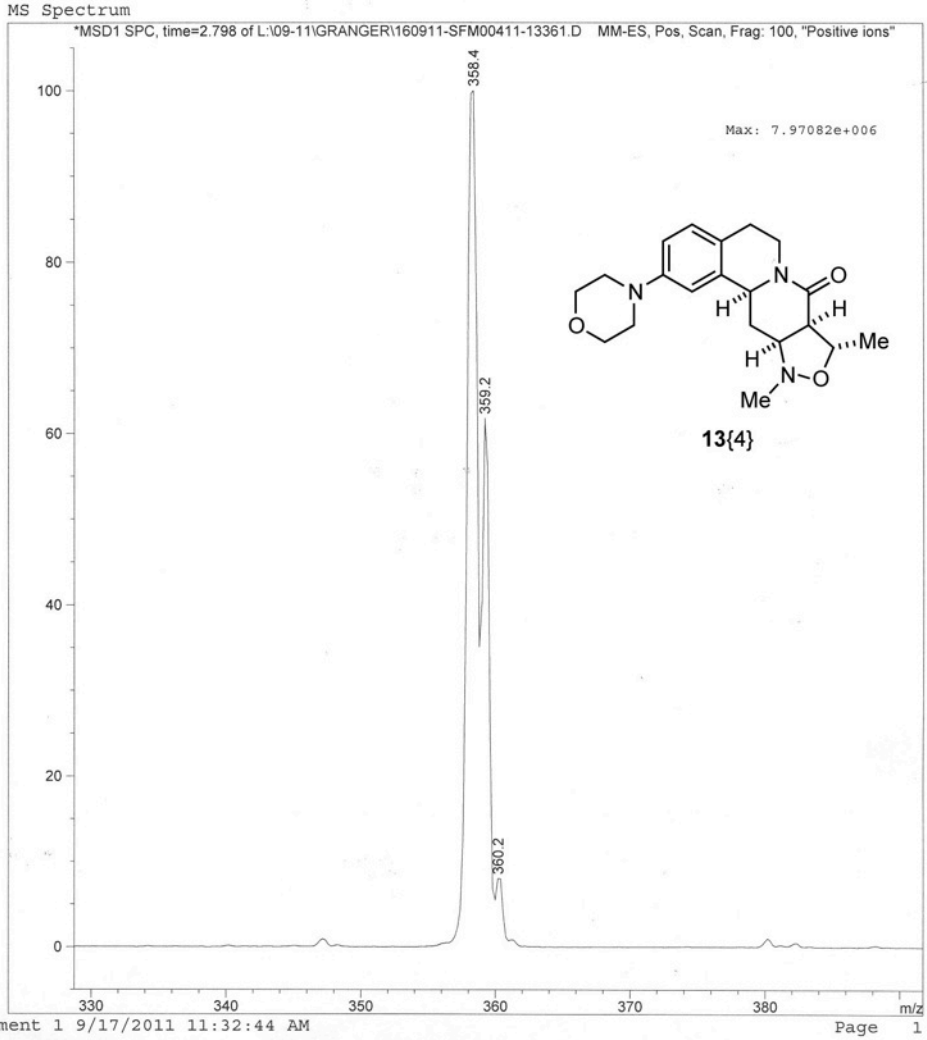
    Totals :                  2874.05480  470.24625
```



13{4}

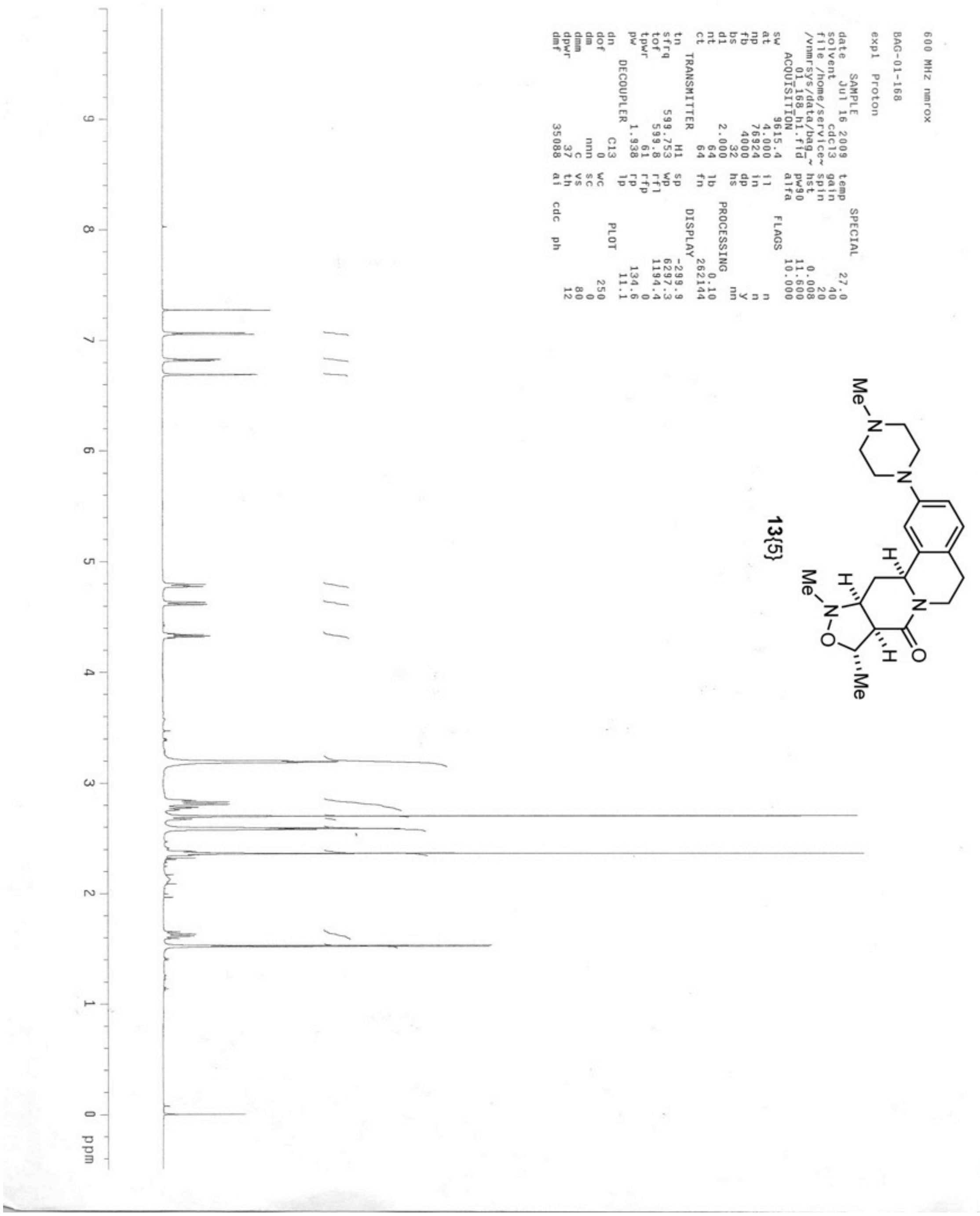
```
Print of window 79: MS Spectrum
Data File  : L:\09-11\GRANGER\160911-SFM00411-13361.D
Sample Name : SFM0041
======================================================================
Acq. Operator   : brettag35@mail.utexas.edu
Acq. Instrument : LCMS                          Location : Vial 42
Injection Date  : 9/16/2011 11:32:45 PM              Inj :   1
                                                Inj Volume : 1.0 µl
Acq. Method     : C:\CHEM32\1\METHODS\SP NIH.M
Last changed    : 9/16/2011 11:32:30 PM by brettag35@mail.utexas.edu
                  (modified after loading)
Analysis Method : C:\CHEM32\1\METHODS\DEF_LC.M
Last changed    : 11/20/2006 4:14:44 AM
Sample Info     : Easy-Access Method: 'SP NIH'
```

MS Spectrum

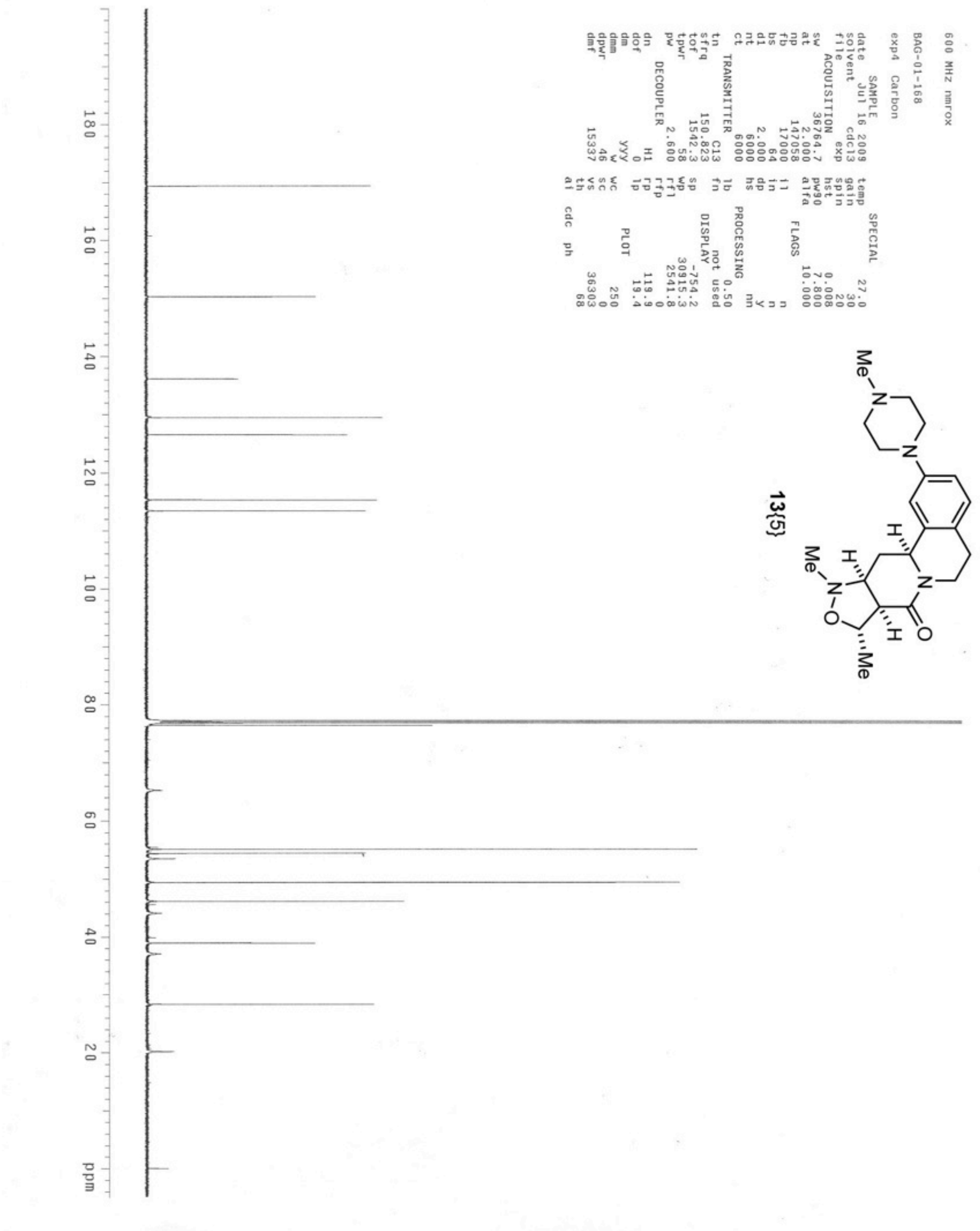


Instrument 1 9/17/2011 11:32:44 AM

Page 1 of 1

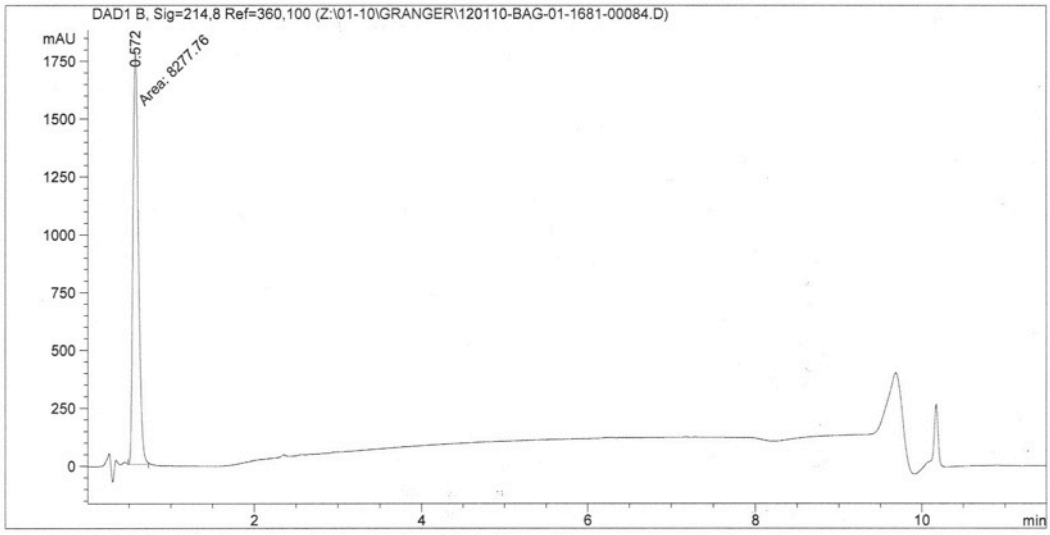


13{5}


600 MHz nmrox
BAG-01-168
exp4 Carbon
SAMPLE
date Jul 16 2009
solvent cdcl3
file exp
ACQUISITION
sw 36764.7
at 2.000
np 147058
fb 17000
bs 64
d1 2.000
nt 6000
ct 6000
TRANSMITTER
tn C13
sfrq 150.823
tof 1542.3
tpwr 58
pw 2.600
DECOUPLER
dn H1
dof 0
dm yyy
dmm w
dpwr 46
dmf 15337
SPECIAL
temp 27.0
gain 30
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 -754.2
wp 30915.3
rfl 2541.8
rfp 0
rp 119.9
lp 19.4
PLOT
wc 250
sc 0
vs 36303
th 68
ai cdc ph
Me
N
O
H
13{5}
180
160
140
120
100
80
60
40
20
ppm

Data File Z:\01-10\GRANGER\120110-BAG-01-1681-00084.D
Sample Name: BAG-01-168

```
==========================================================================
Acq. Operator   : brettag35@gmail.com
Acq. Instrument : LCMS                          Location : Vial 85
Injection Date  : 1/12/2010 9:06:27 PM
                                               Inj Volume : 1.0 µl
Acq. Method     : C:\CHEM32\1\METHODS\SP NIH.M
Last changed    : 1/12/2010 9:06:17 PM by brettag35@gmail.com
                  (modified after loading)
Analysis Method : C:\CHEM32\1\METHODS\DEF_LC.M
Last changed    : 11/20/2006 4:14:44 AM
Sample Info     : Easy-Access Method: 'SP NIH'
```




```
==========================================================================
                          Area Percent Report
==========================================================================

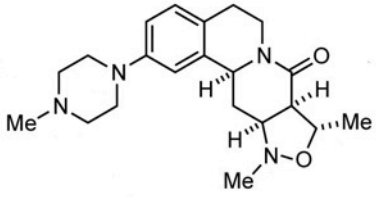
Sorted By             :      Signal
Multiplier:                   :      1.0000
Dilution:                     :      1.0000
Use Multiplier & Dilution Factor with ISTDs


Signal 1: DAD1 B, Sig=214,8 Ref=360,100

Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]   [mAU*s]     [mAU]       %
----|-------|----|-------|----------|----------|--------|
   1   0.572 MM    0.0769 8277.76172 1793.66711 100.0000

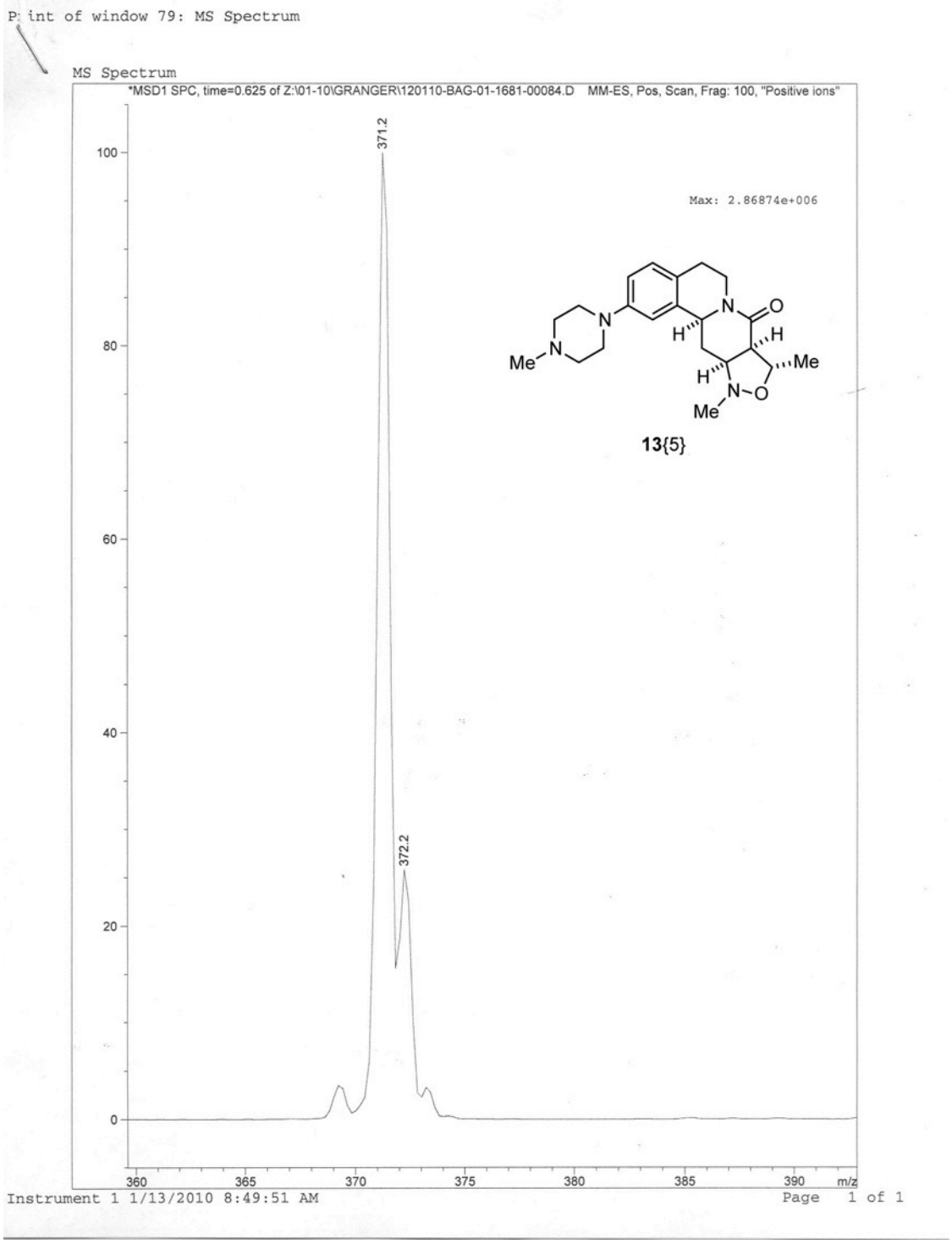
Totals :                  8277.76172 1793.66711

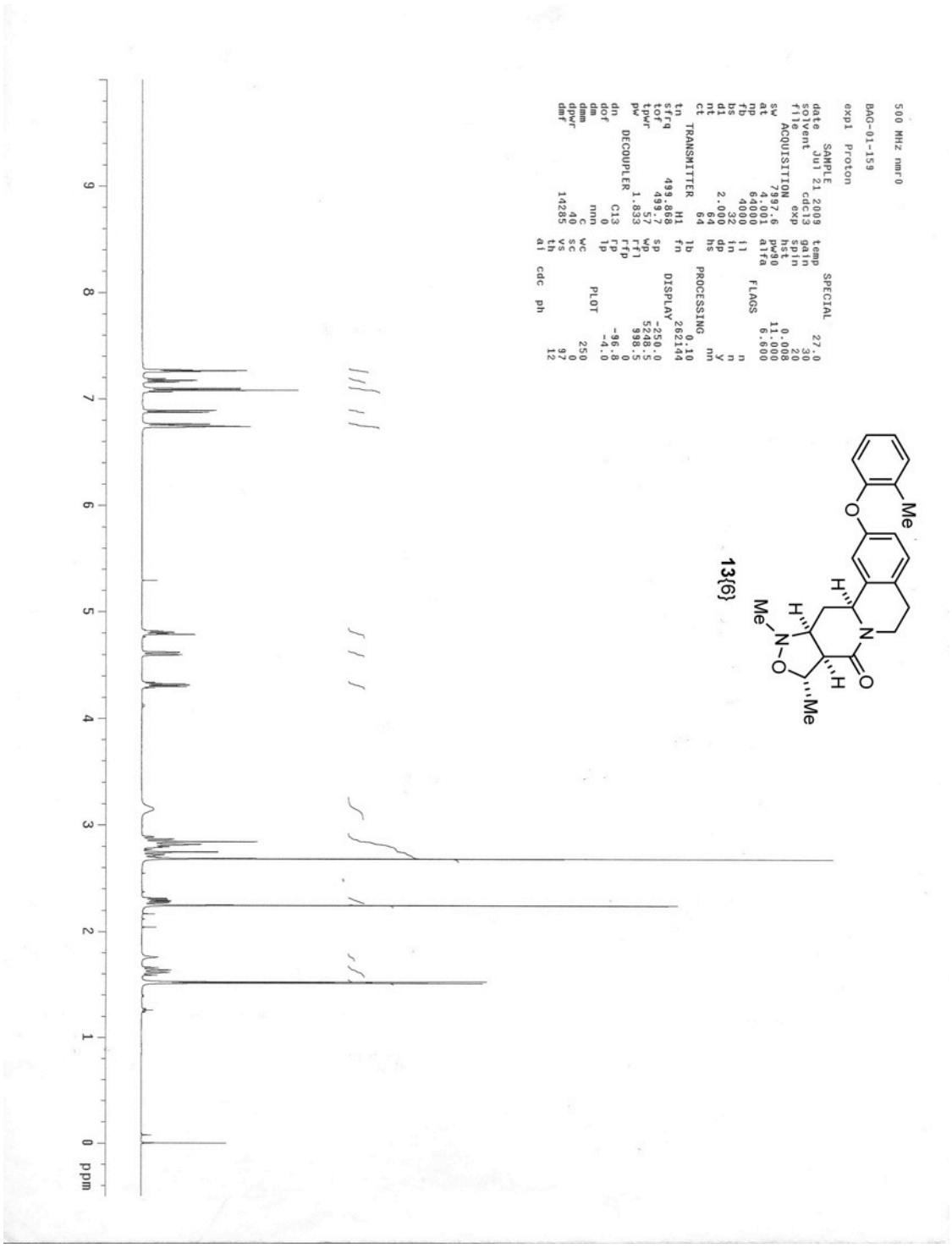
==========================================================================
```

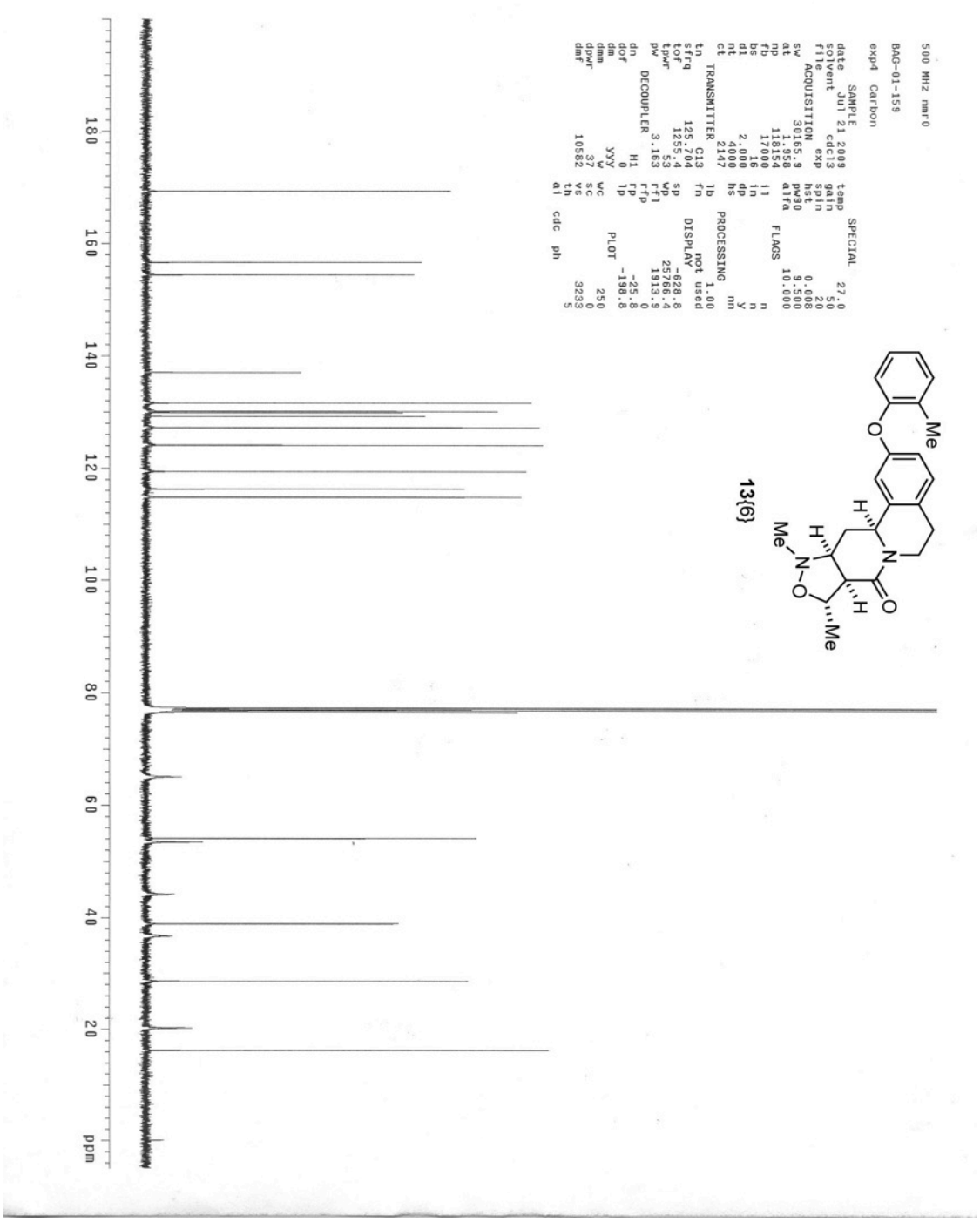


13{5}

Instrument 1 1/13/2010 8:51:18 AM Page 1 of 1

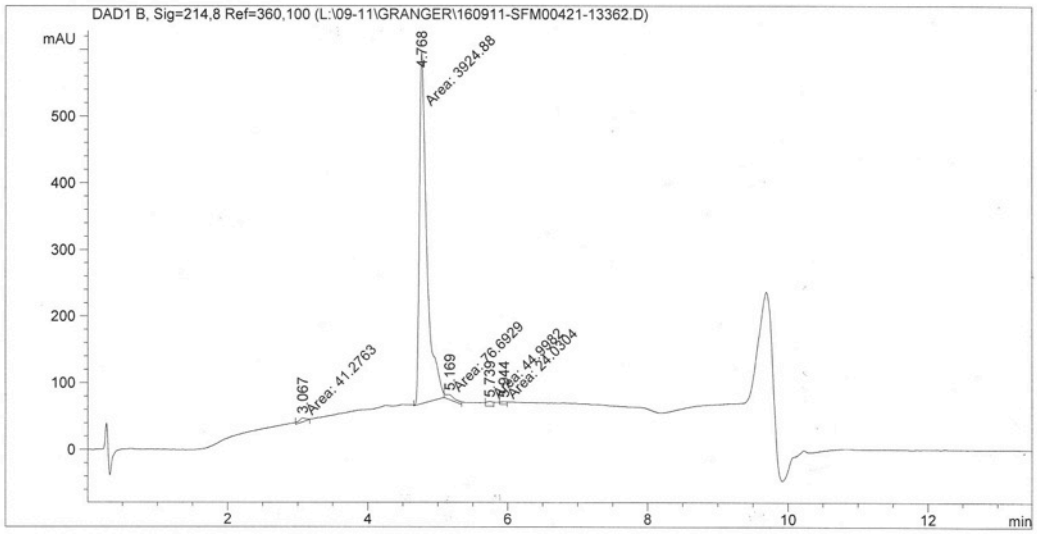

P: int of window 79: MS Spectrum
MS Spectrum
*MSD1 SPC, time=0.625 of Z:\01-10\GRANGER\120110-BAG-01-1681-00084.D MM-ES, Pos, Scan, Frag: 100, "Positive ions"
371.2
372.2
Max: 2.86874e+006
100
80
60
40
20
0
360
365
370
375
380
385
390
m/z
Me
N
O
H
Me
13{5}
Instrument 1 1/13/2010 8:49:51 AM
Page 1 of 1


500 MHz nmr0
BAG-01-159
exp1 Proton
SAMPLE
date Jul 21 2009
solvent cdcl3
file exp
ACQUISITION
sw 7997.6
at 4.001
np 64000
fb 4000
bs 32
d1 2.000
nt 64
ct 64
TRANSMITTER
tn H1
sfrq 499.868
tof 499.7
tpwr 57
pw 1.833
DECOUPLER
dn C13
dof 0
dm nnn
dmm c
dpwr 40
dmf 14285
temp 27.0
gain 30
spin 20
hst 0.008
pw90 11.000
alfa 6.600
FLAGS
il n
in n
dp y
hs nn
PROCESSING
lb 0.10
fn 262144
DISPLAY
sp -250.0
wp 5248.5
rfl 998.5
rfp 0
rp -96.8
lp -4.0
PLOT
wc 250
sc 0
vs 97
th 12
al cdc ph
Me
O
H
N
O
H
Me
N
O
H
Me
13{6}
9
8
7
6
5
4
3
2
1
0 ppm


500 MHz rmr0
BAG-01-159
exp4 Carbon
SAMPLE
date Jul 21 2009
solvent cdcl3
file exp
ACQUISITION
sw 30165.9
at 1.958
np 118154
fb 17000
bs 16
bs 2.000
d1 1.000
nt 2147
ct
TRANSMITTER
tn C13
sfrq 125.704
tof 1255.4
tpwr 53
pw 3.163
DECOUPLER
dn H1
dof 0
dm yyy
dmm w
dpwr 37
dmf 10582
SPECIAL
temp 27.0
gain 50
spin 20
hst 0.008
pw90 9.500
a1fa FLAGS 10.000
lb n
fn n
PROCESSING y
lb nn
fn 1.00
DISPLAY not used
sp -628.8
wp 25766.4
rfl 1913.9
rfp 0
lp -25.0
PLOT -198.8
wc 250
sc 0
vs 3233
th 5
al cdc ph
Me
O
H
N
O
H
Me
N
O
H
Me
13{6}
180
160
140
120
100
80
60
40
20
ppm

Data File L:\09-11\GRANGER\160911-SFM00421-13362.D
Sample Name: SFM0042

```
Acq. Operator   : brettag35@mail.utexas.edu
Acq. Instrument : LCMS                          Location : Vial 43
Injection Date  : 9/16/2011 11:47:44 PM
                                                Inj Volume : 1.0 µl
Acq. Method     : C:\CHEM32\1\METHODS\SP NIH.M
Last changed    : 9/16/2011 11:47:29 PM by brettag35@mail.utexas.edu
                  (modified after loading)
Analysis Method : C:\CHEM32\1\METHODS\DEF_LC.M
Last changed    : 11/20/2006 4:14:44 AM
Sample Info     : Easy-Access Method: 'SP NIH'
```

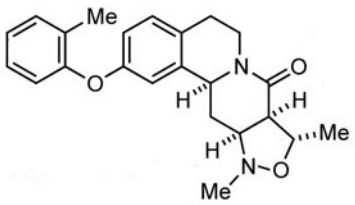



```
=====================================================================
                        Area Percent Report
=====================================================================

Sorted By             :      Signal
Multiplier:                   :      1.0000
Dilution:                     :      1.0000
Use Multiplier & Dilution Factor with ISTDs

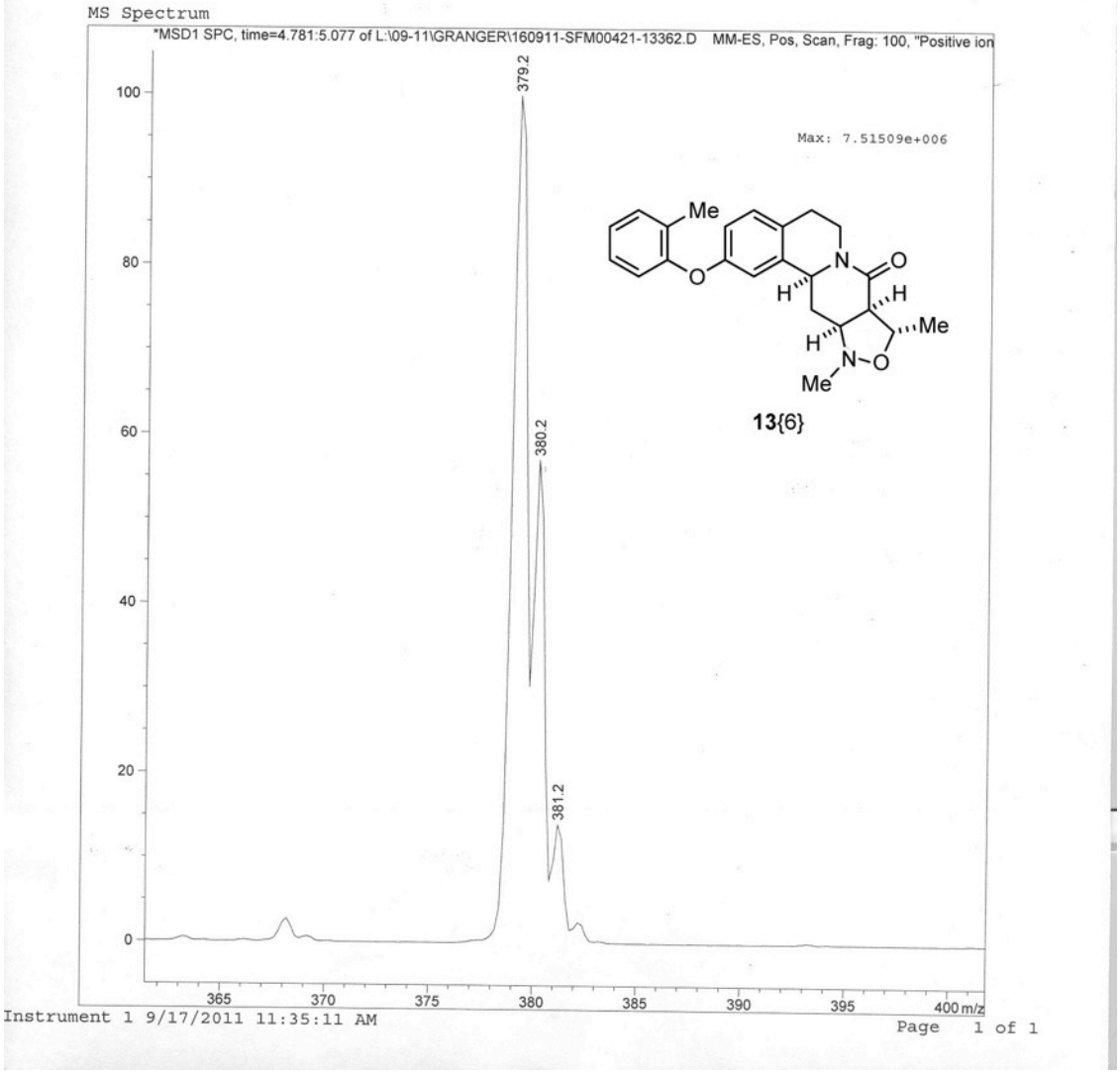

Signal 1: DAD1 B, Sig=214,8 Ref=360,100

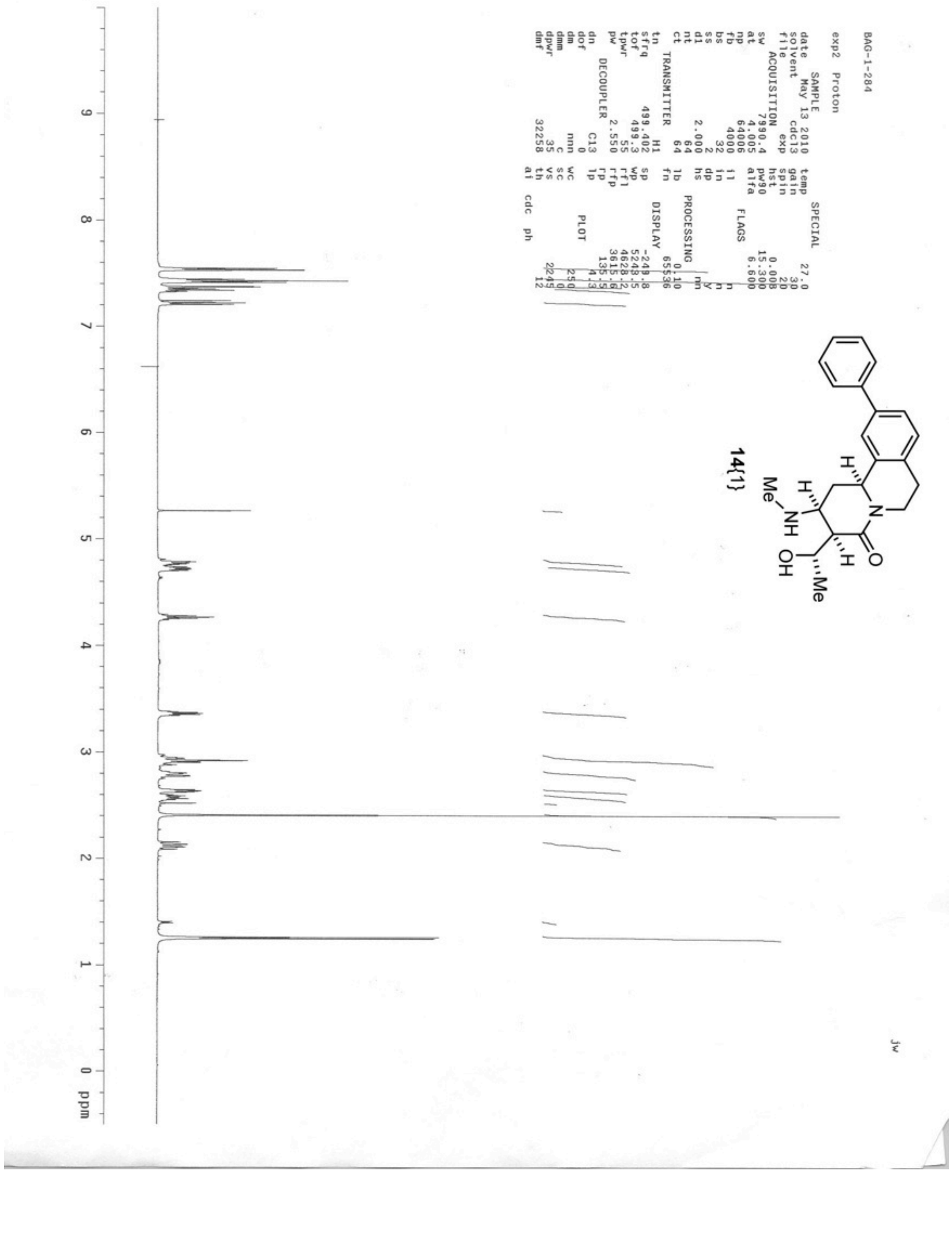
Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]   [mAU*s]     [mAU]       %
----|-------|----|-------|----------|----------|--------|
   1   3.067 MM    0.1132   41.27634    6.07784   1.0038
   2   4.768 MM    0.1237 3924.88403  528.73779  95.4523
   3   5.169 MM    0.1719   76.69287    7.43584   1.8652
   4   5.739 MM    0.1087   44.99820    6.89999   1.0943
   5   5.944 MM    0.1012   24.03038    3.95608   0.5844
```



13{6}

```
Print of window 79: MS Spectrum
Data File  : L:\09-11\GRANGER\160911-SFM00421-13362.D
Sample Name : SFM0042
======================================================================
Acq. Operator   : brettag35@mail.utexas.edu
Acq. Instrument : LCMS                          Location : Vial 43
Injection Date  : 9/16/2011 11:47:44 PM              Inj :   1
                                                Inj Volume : 1.0 µl
Acq. Method     : C:\CHEM32\1\METHODS\SP NIH.M
Last changed    : 9/16/2011 11:47:29 PM by brettag35@mail.utexas.edu
                  (modified after loading)
Analysis Method : C:\CHEM32\1\METHODS\DEF_LC.M
Last changed    : 11/20/2006 4:14:44 AM
Sample Info     : Easy-Access Method: 'SP NIH'
```

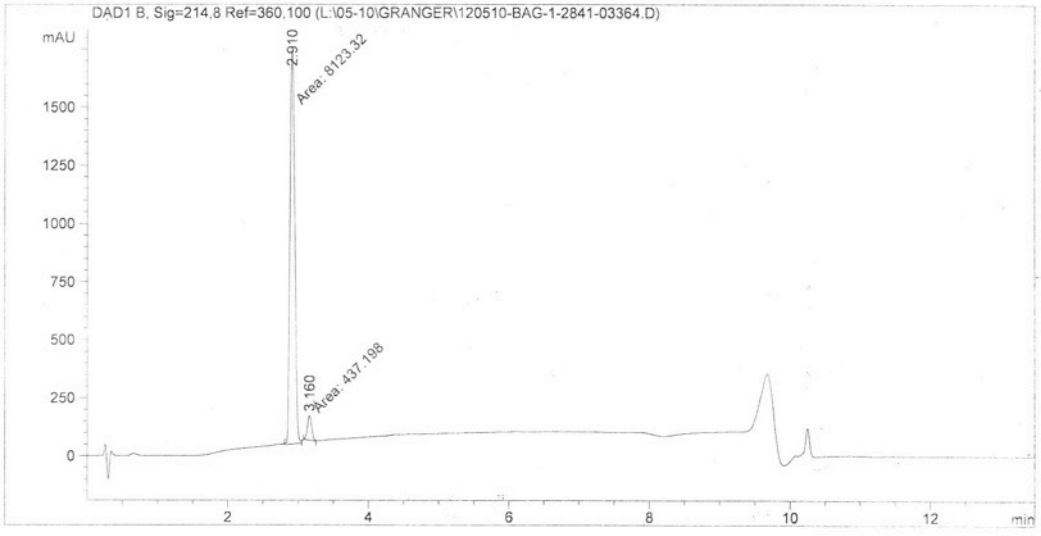



BAG-1-284
exp2 Proton
14{1}
ppm

BAG-1-284
exp4 Carbon
SAMPLE
May 13 2010
cdcl3
exp
ACQUISITION
30143.2
1.062
64024
17000
64
2.000
600
600
PROCESSING
1.00
not used
DISPLAY
-628.1
25742.4
11596.2
9669.2
-95.5
-212.5
PLOT
250
0
1863
8
SPECIAL
27.0
50
20
0.008
15.500
10.000
FLAGS
n
n
y
nn
14{1}
Me
NH
OH
H
N
O
Me
jw
180
160
140
120
100
80
60
40
20
ppm

Data File L:\05-10\GRANGER\120510-BAG-1-2841-03364.D
Sample Name: BAG-1-284

```
==========================================================================
Acq. Operator   : brettag35@mail.utexas.edu
Acq. Instrument : LCMS                          Location : Vial 16
Injection Date  : 5/12/2010 2:14:17 PM
                                                Inj Volume : 1.0 µl
Acq. Method     : C:\CHEM32\1\METHODS\SP NIH.M
Last changed    : 5/12/2010 2:14:01 PM by brettag35@mail.utexas.edu
                  (modified after loading)
Analysis Method : C:\CHEM32\1\METHODS\DEF_LC.M
Last changed    : 11/20/2006 4:14:44 AM
Sample Info     : Easy-Access Method: 'SP NIH'
```



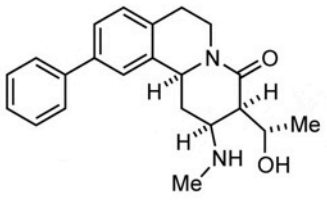

```
==========================================================================
                        Area Percent Report
==========================================================================

Sorted By             :      Signal
Multiplier:                   :      1.0000
Dilution:                     :      1.0000
Use Multiplier & Dilution Factor with ISTDs


Signal 1: DAD1 B, Sig=214,8 Ref=360,100

Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]    [mAU*s]     [mAU]      %
----|-------|----|-------|----------|----------|--------|
   1   2.910 MM    0.0796 8123.32373 1701.59070  94.8929
   2   3.160 MM    0.0693  437.19791  105.10128   5.1071

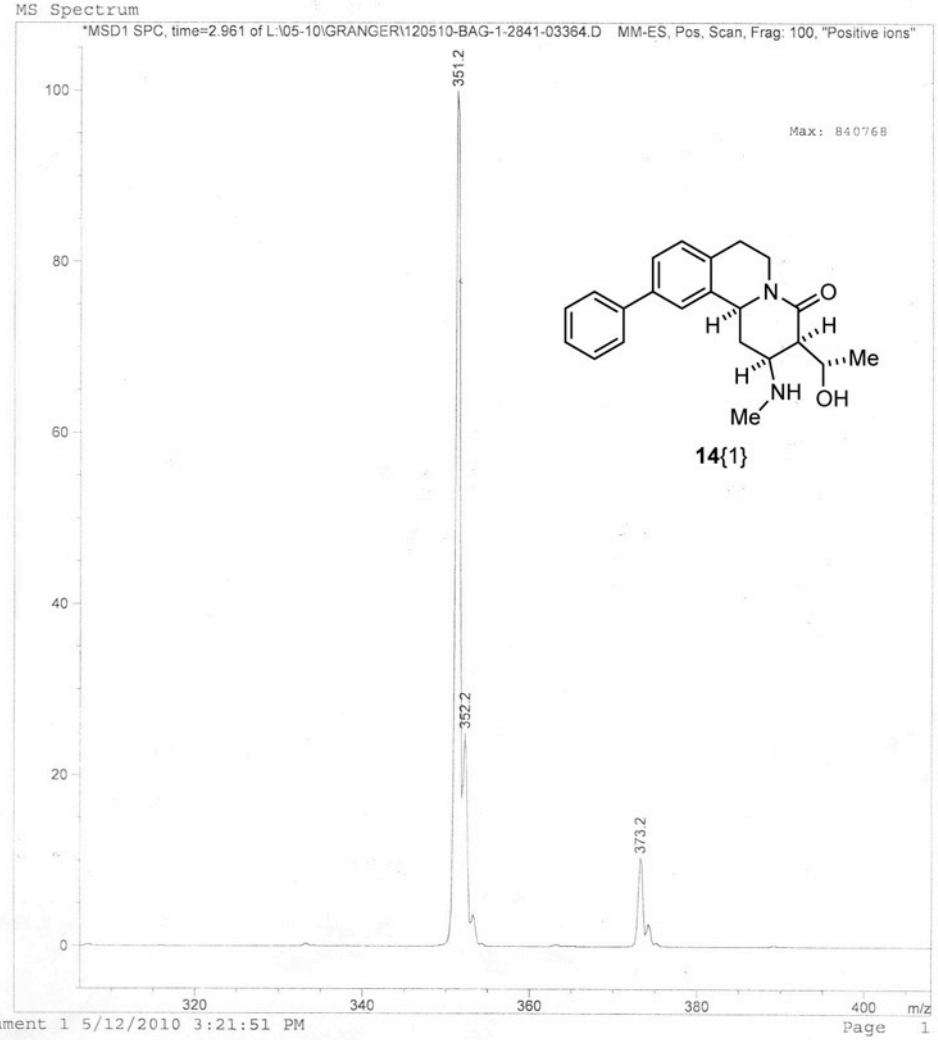
Totals :                  8560.52164 1806.69198
```



14{1}

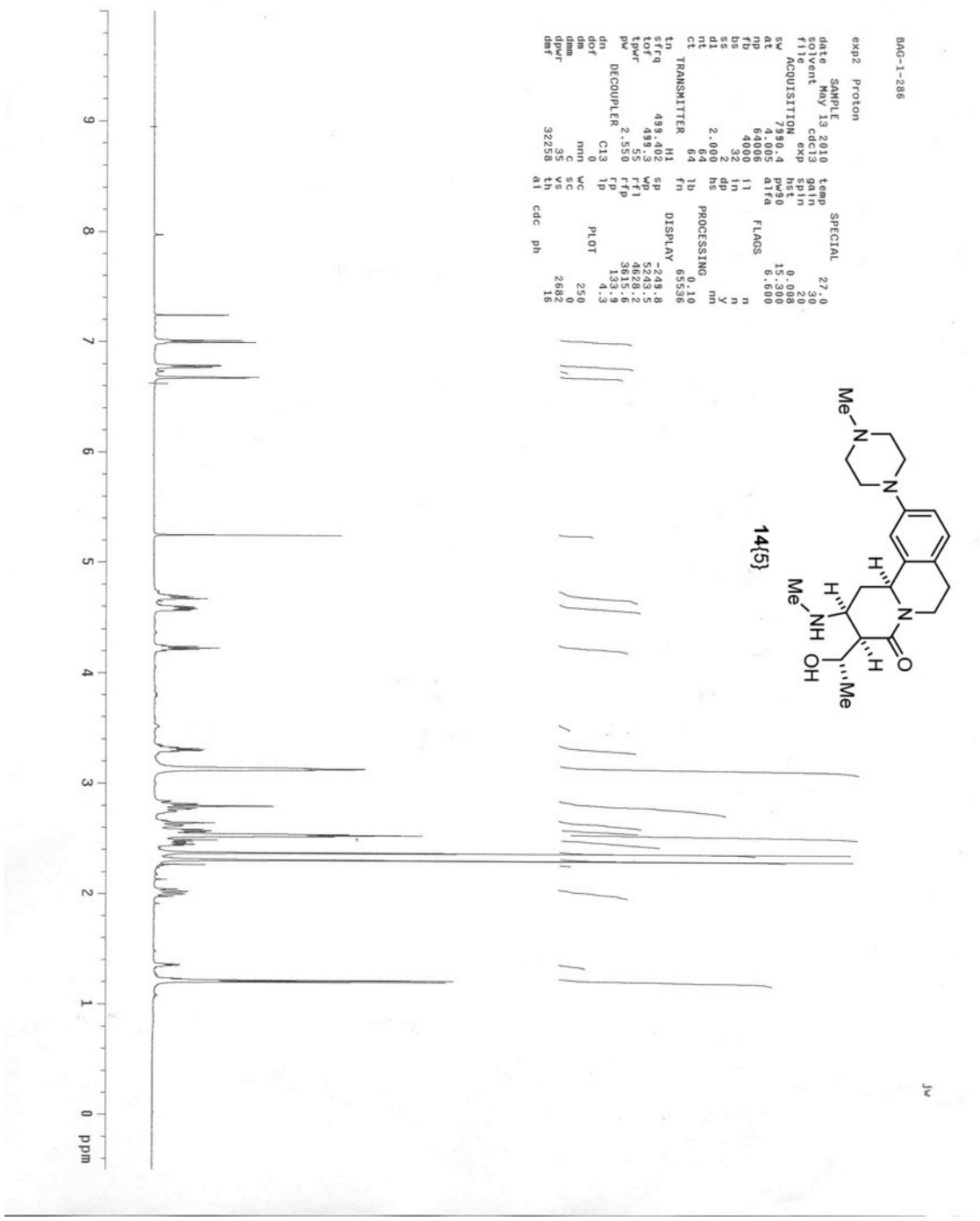
```
Print of window 79: MS Spectrum
Data File   : L:\05-10\GRANGER\120510-BAG-1-2841-03364.D
Sample Name : BAG-1-284
=====================================================================
Acq. Operator    : brettag35@mail.utexas.edu
Acq. Instrument : LCMS                          Location : Vial 16
Injection Date  : 5/12/2010 2:14:17 PM             Inj :   1
                                                Inj Volume : 1.0 µl
Acq. Method      : C:\CHEM32\1\METHODS\SP NIH.M
Last changed     : 5/12/2010 2:14:01 PM by brettag35@mail.utexas.edu
                   (modified after loading)
Analysis Method : C:\CHEM32\1\METHODS\DEF_LC.M
Last changed     : 11/20/2006 4:14:44 AM
Sample Info      : Easy-Access Method: 'SP NIH'
```

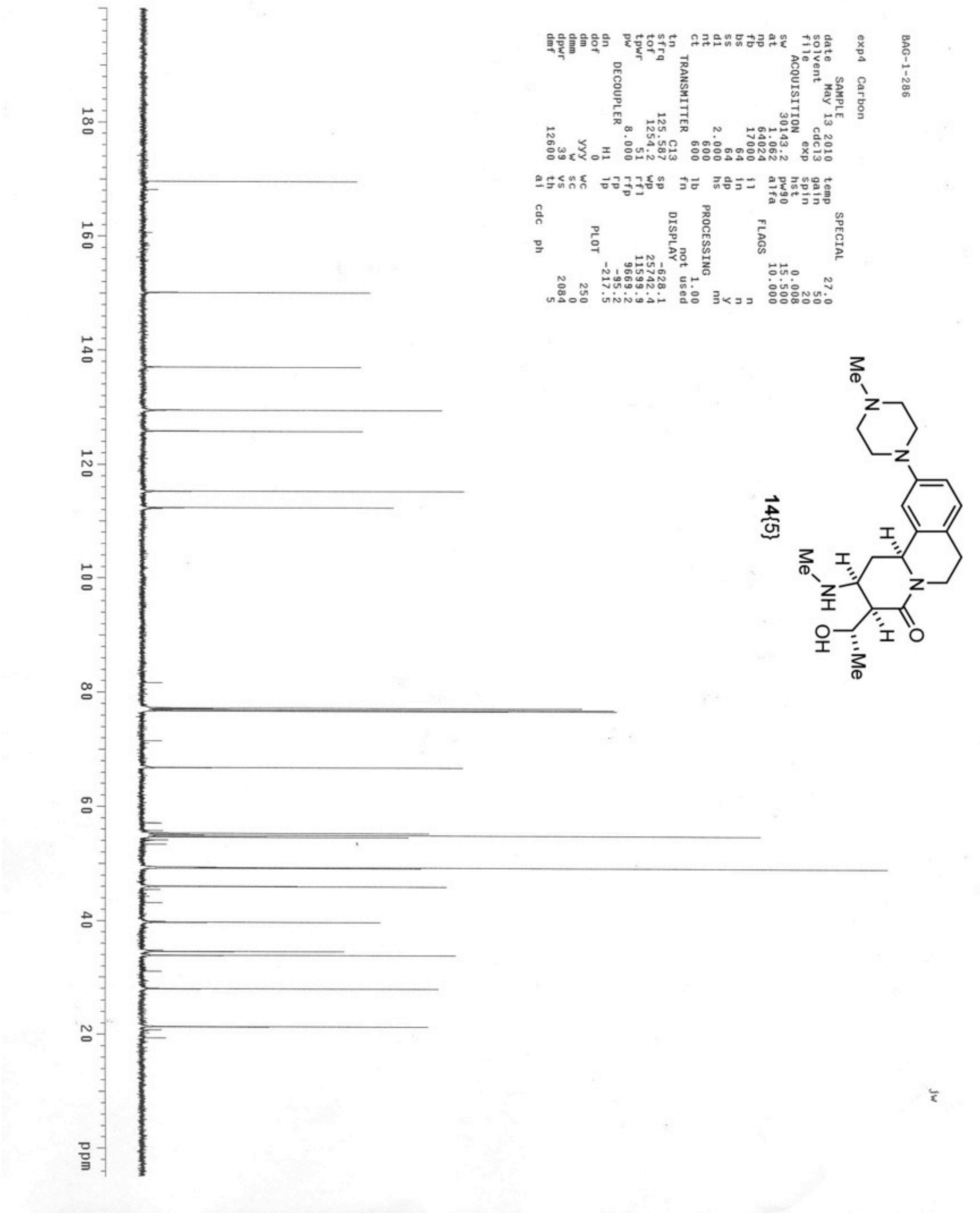
MS Spectrum



14{1}

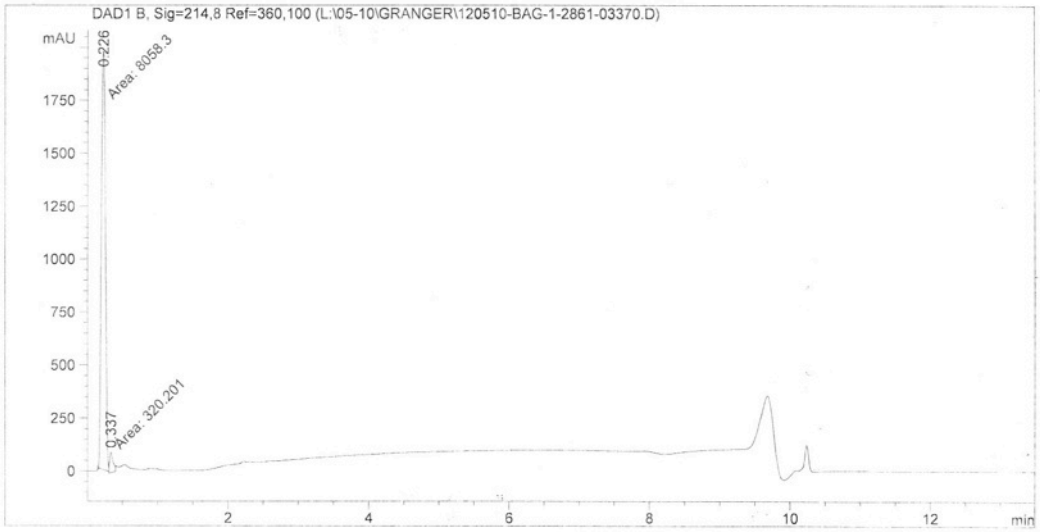
Instrument 1 5/12/2010 3:21:51 PM


BAG-1-286
exp2 Proton
SAMPLE
date May 13 2010
solvent cdcl3
file exp
ACQUISITION
sw 7990.4
at 4.005
np 64006
fb 4000
bs 32
ss 2
sf 2.000
nt 64
ct 64
TRANSMITTER
tn H1
sfrq 499.402
tof 499.3
tpwr 55
pw 2.550
DECOUPLER
dn C13
dof 0
dm nnn
dmm c
dpwr 35
dmf 32258
temp
gain
spin
hst
pw90
alfa
FLAGS
il
in
dp
hs
PROCESSING
lb
fn
DISPLAY
sp
wp
rfl
rfp
rp
lp
PLOT
wc
sc
vs
th
al
SPECIAL
27.0
30
20
0.008
15.300
6.600
n
n
y
nn
0.10
65536
-249.8
5243.5
4628.2
3615.6
133.9
4.3
250
0
2682
16
cdc ph
14{5}
Me
N
N
H
H
Me
NH
OH
H
Me
O
N
JW
9
8
7
6
5
4
3
2
1
0
ppm


BAG-1-286
exp4 Carbon
14{5}
ppm

Data File L:\05-10\GRANGER\120510-BAG-1-2861-03370.D
Sample Name: BAG-1-286

```
==========================================================================
Acq. Operator   : brettag35@mail.utexas.edu
Acq. Instrument : LCMS                          Location : Vial 22
Injection Date  : 5/12/2010 5:14:10 PM
                                                Inj Volume : 1.0 µl
Acq. Method     : C:\CHEM32\1\METHODS\SP NIH.M
Last changed    : 5/12/2010 5:13:56 PM by brettag35@mail.utexas.edu
                  (modified after loading)
Analysis Method : C:\CHEM32\1\METHODS\DEF_LC.M
Last changed    : 11/20/2006 4:14:44 AM
Sample Info     : Easy-Access Method: 'SP NIH'
```



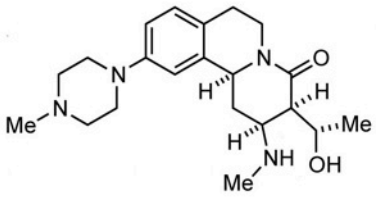

```
==========================================================================
                        Area Percent Report
==========================================================================

Sorted By             :      Signal
Multiplier:                   :      1.0000
Dilution:                     :      1.0000
Use Multiplier & Dilution Factor with ISTDs


Signal 1: DAD1 B, Sig=214,8 Ref=360,100

Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]   [mAU*s]     [mAU]       %
----|-------|----|-------|----------|----------|--------|
   1   0.226 MM    0.0675 8058.30127 1988.87952  96.1783
   2   0.337 MM    0.0551  320.20087   96.86745   3.8217

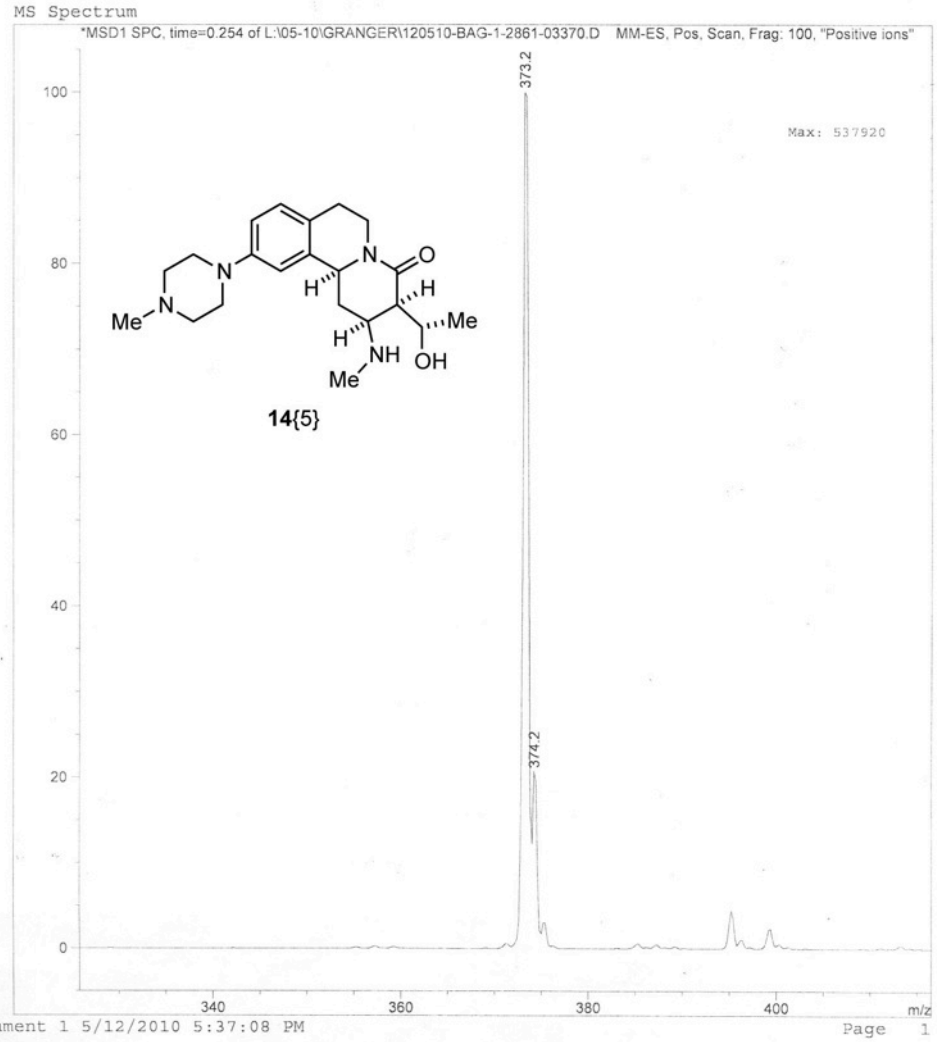
Totals :                  8378.50214 2085.74697
```



14{5}

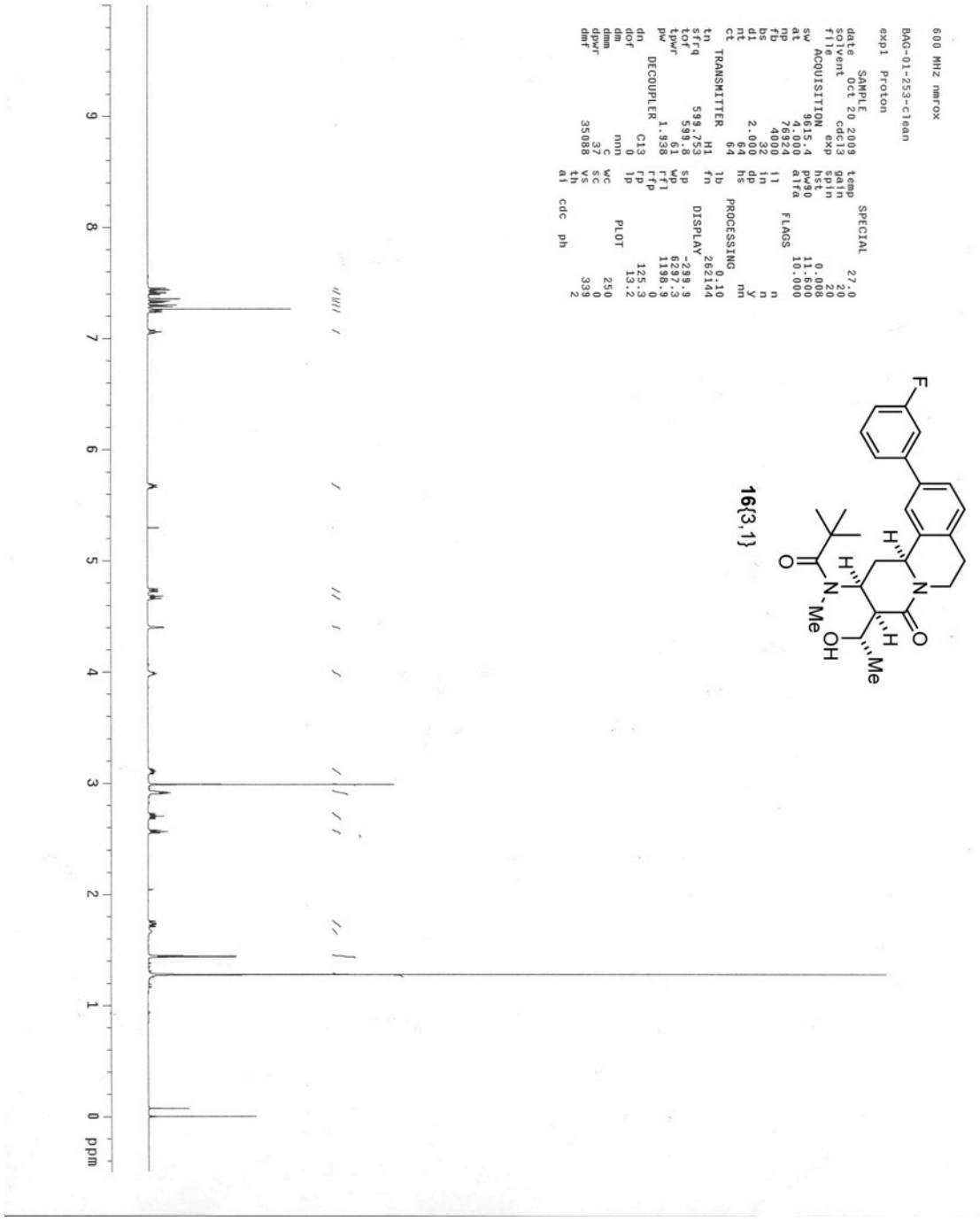
nt of window 79: MS Spectrum
a File : L:\05-10\GRANGER\120510-BAG-1-2861-03370.D
Sample Name : BAG-1-286

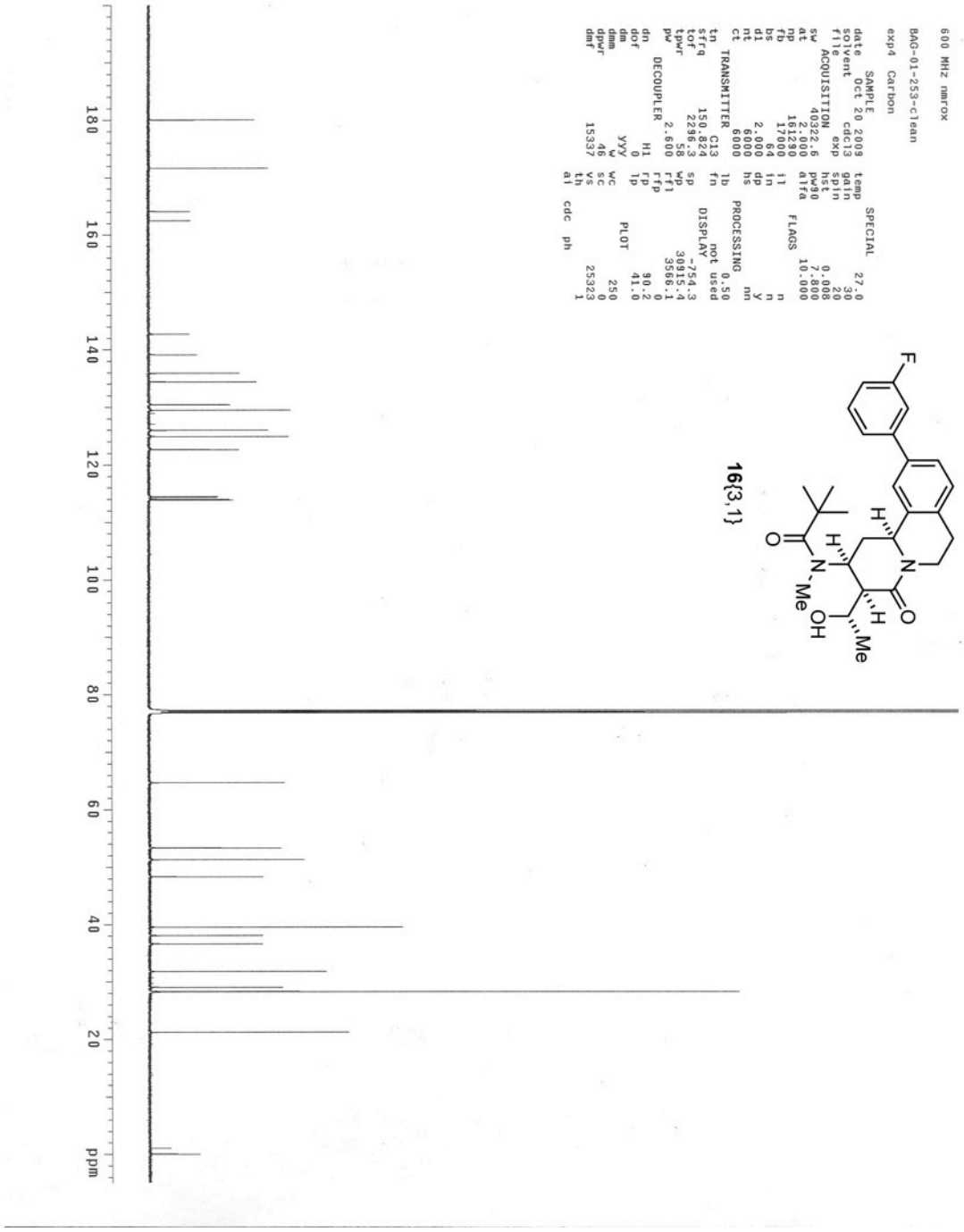
```
Acq. Operator    : brettag35@mail.utexas.edu
Acq. Instrument : LCMS                          Location : Vial 22
Injection Date  : 5/12/2010 5:14:10 PM              Inj :   1
                                                Inj Volume : 1.0 µl
Acq. Method      : C:\CHEM32\1\METHODS\SP NIH.M
Last changed     : 5/12/2010 5:13:56 PM by brettag35@mail.utexas.edu
                   (modified after loading)
Analysis Method : C:\CHEM32\1\METHODS\DEF_LC.M
Last changed     : 11/20/2006 4:14:44 AM
Sample Info      : Easy-Access Method: 'SP NIH'
```



Instrument 1 5/12/2010 5:37:08 PM

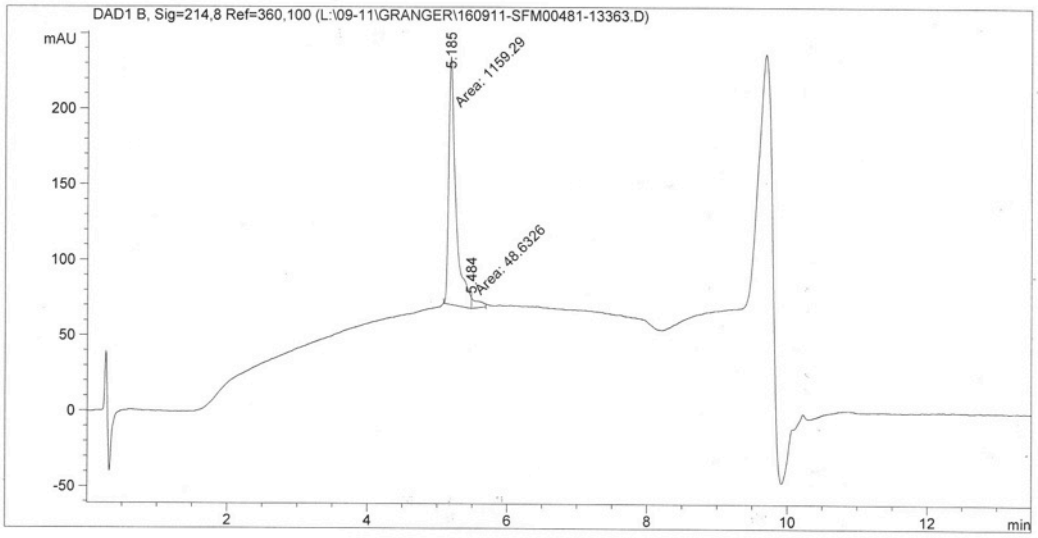
Page 1 of 1


600 MHz nmrox
BAG-01-253-clean
exp1 Proton
SAMPLE
date Oct 20 2009
solvent cdc13
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 599.8
tpwr 61
pw 1.938
DECOUPLER
dn C13
dof 0
dm nnn
dmm c
dmf 37
dpwr 35088
SPECIAL
temp 27.0
gain 20
spin 20
hst 0.008
pw90 11.600
atfa 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 1198.9
rfp 0
rp 125.3
lp 13.2
PLOT
wc 250
sc 0
vs 339
th 2
al cdc ph
16{3,1}
F
H
N
O
H
N
Me
O
H
Me
OH
9
8
7
6
5
4
3
2
1
0
ppm


600 MHz nmrox
BAG-01-253-clean
exp4 Carbon
SAMPLE
date Oct 20 2009
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 30
spin 20
hst 0.008
ppm 7.800
dfrq 10.000
FLAGS
il n
in n
dp y
hs nn
PROCESSING
lb 0.50
fn not used
DISPLAY
sp -754.3
wp 3091.4
rfl 3566.1
rfp 0
rp 90.2
lp 41.0
PLOT
wc 250
sc 0
vs 25323
th 1
al cdc ph
F
H
N
O
H
Me
OH
N
Me
O
16{3,1}
180
160
140
120
100
80
60
40
20
ppm

Data File L:\09-11\GRANGER\160911-SFM00481-13363.D
Sample Name: SFM0048

```
======================================================================
Acq. Operator   : brettag35@mail.utexas.edu
Acq. Instrument : LCMS                          Location : Vial 44
Injection Date  : 9/17/2011 12:02:44 AM
                                                Inj Volume : 1.0 µl
Acq. Method     : C:\CHEM32\1\METHODS\SP NIH.M
Last changed    : 9/17/2011 12:02:29 AM by brettag35@mail.utexas.edu
                  (modified after loading)
Analysis Method : C:\CHEM32\1\METHODS\DEF_LC.M
Last changed    : 11/20/2006 4:14:44 AM
Sample Info     : Easy-Access Method: 'SP NIH'
```



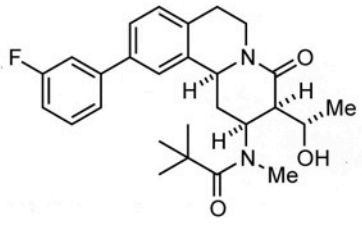

```
======================================================================
                        Area Percent Report
======================================================================

Sorted By             :      Signal
Multiplier:                   :      1.0000
Dilution:                     :      1.0000
Use Multiplier & Dilution Factor with ISTDs


Signal 1: DAD1 B, Sig=214,8 Ref=360,100

Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]   [mAU*s]     [mAU]       %
----|-------|----|-------|----------|----------|--------|
   1   5.185 MM    0.1174 1159.29321  164.63277  95.9739
   2   5.484 MM    0.1018   48.63261    7.05262   4.0261

Totals :                  1207.92582  171.68539
```



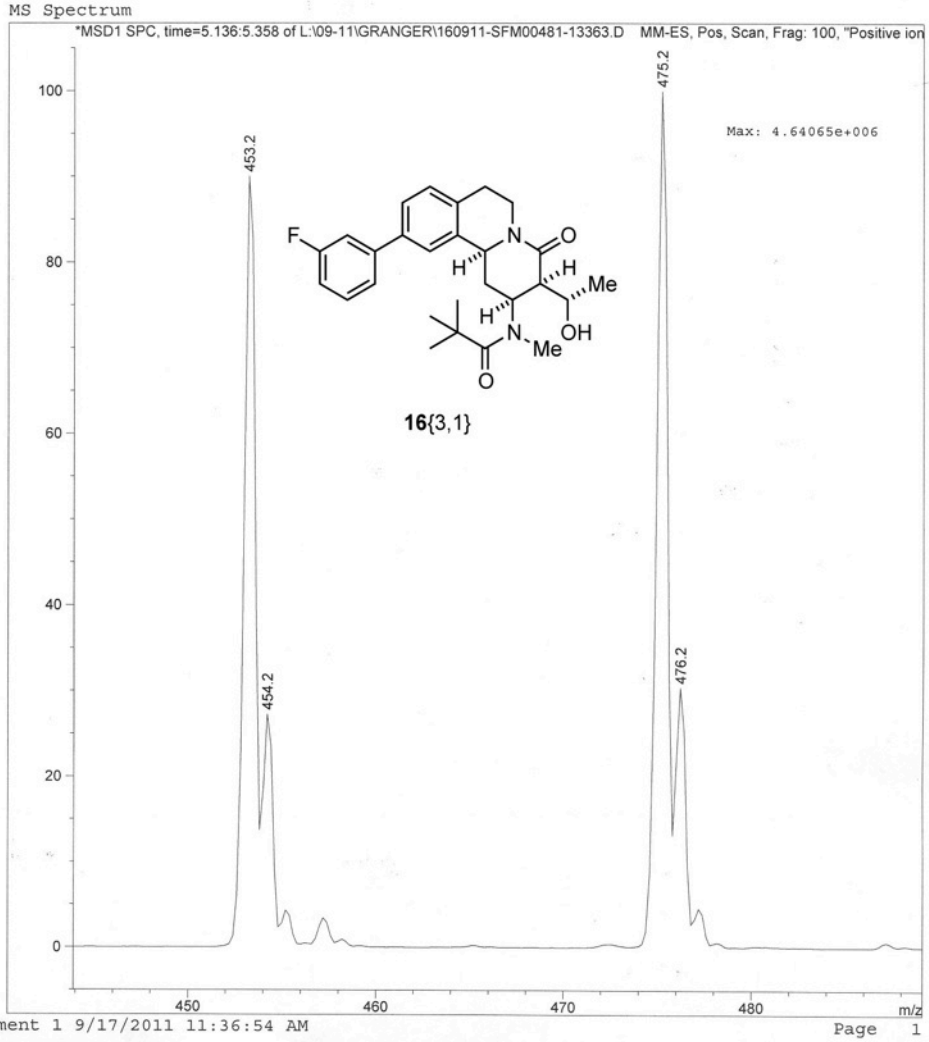
16{3,1}

Instrument 1 9/17/2011 11:36:14 AM

Page 1 of 2

```
Print of window 79: MS Spectrum
Data File  : L:\09-11\GRANGER\160911-SFM00481-13363.D
Sample Name : SFM0048
======================================================================
Acq. Operator   : brettag35@mail.utexas.edu
Acq. Instrument : LCMS                          Location : Vial 44
Injection Date  : 9/17/2011 12:02:44 AM              Inj :   1
                                                Inj Volume : 1.0 µl
Acq. Method     : C:\CHEM32\1\METHODS\SP NIH.M
Last changed    : 9/17/2011 12:02:29 AM by brettag35@mail.utexas.edu
                  (modified after loading)
Analysis Method : C:\CHEM32\1\METHODS\DEF_LC.M
Last changed    : 11/20/2006 4:14:44 AM
Sample Info     : Easy-Access Method: 'SP NIH'
```

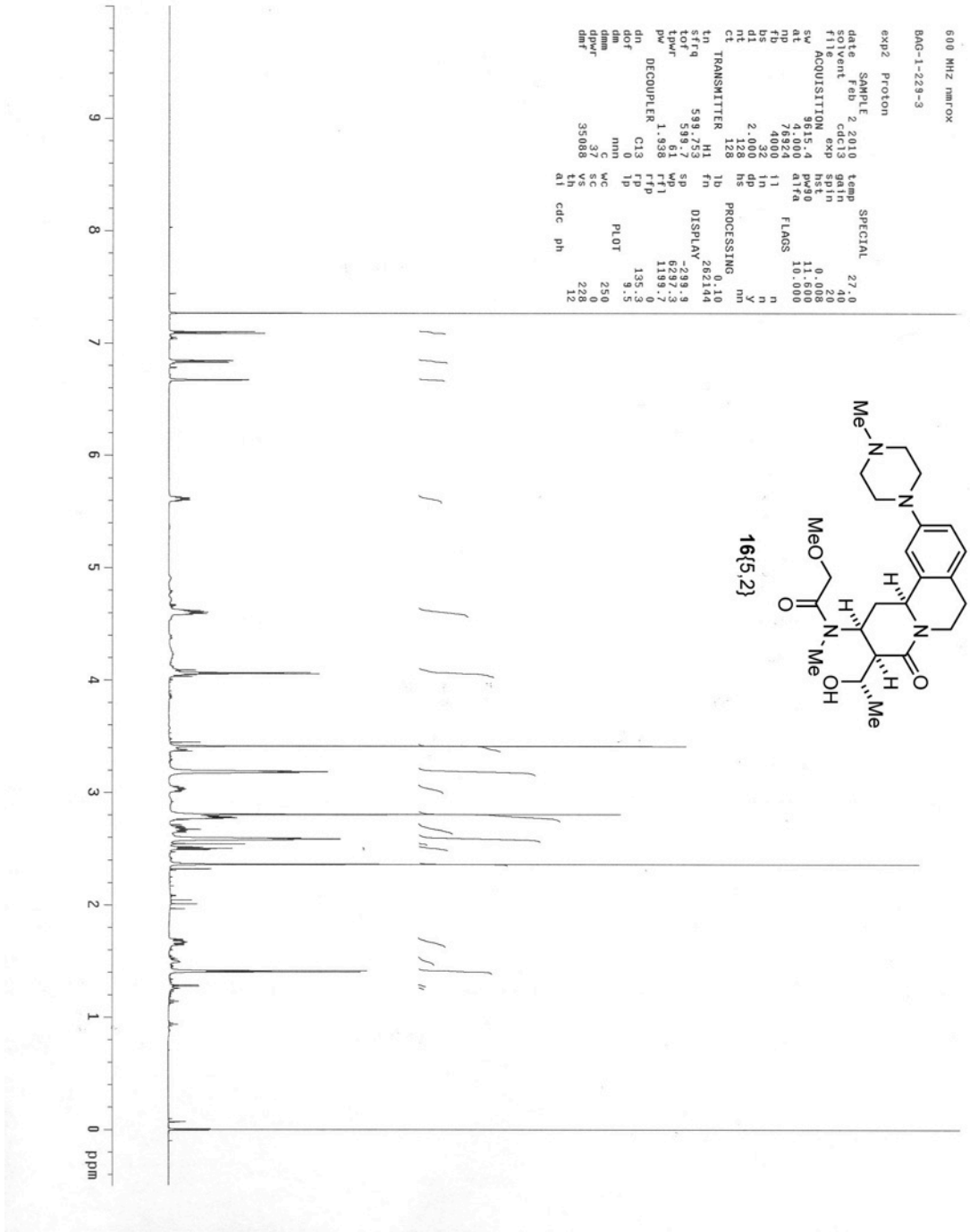
MS Spectrum

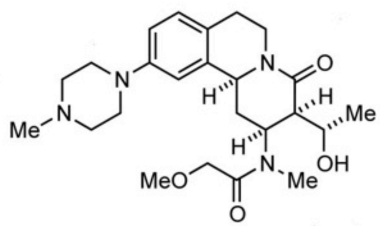


16{3,1}

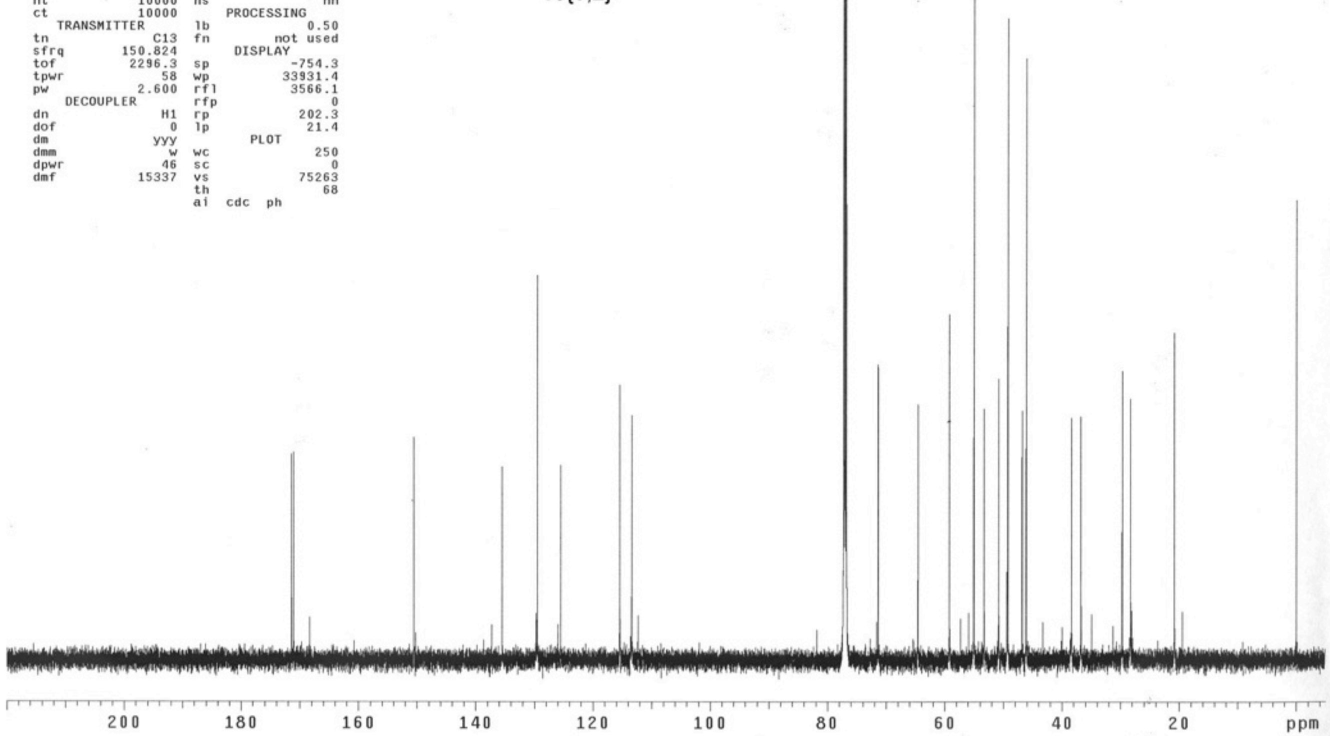
Instrument 1 9/17/2011 11:36:54 AM

Page 1 of 1


600 MHz rmrox
BAG-1-229-3
exp2 Proton
SAMPLE
date Feb 2 2010
solvent cdcl3
file exp
ACQUISITION
sw 9615.4
at 4.000
np 76924
fb 4000
bs 32
ss 2
d1 2.000
nt 128
ct 128
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 1199.7
rfp 0
rp 135.9
lp 3.5
PLOT
wc 250
sc 0
vs 228
th 12
al cdc ph
16{5,2}
9
8
7
6
5
4
3
2
1
0 ppm



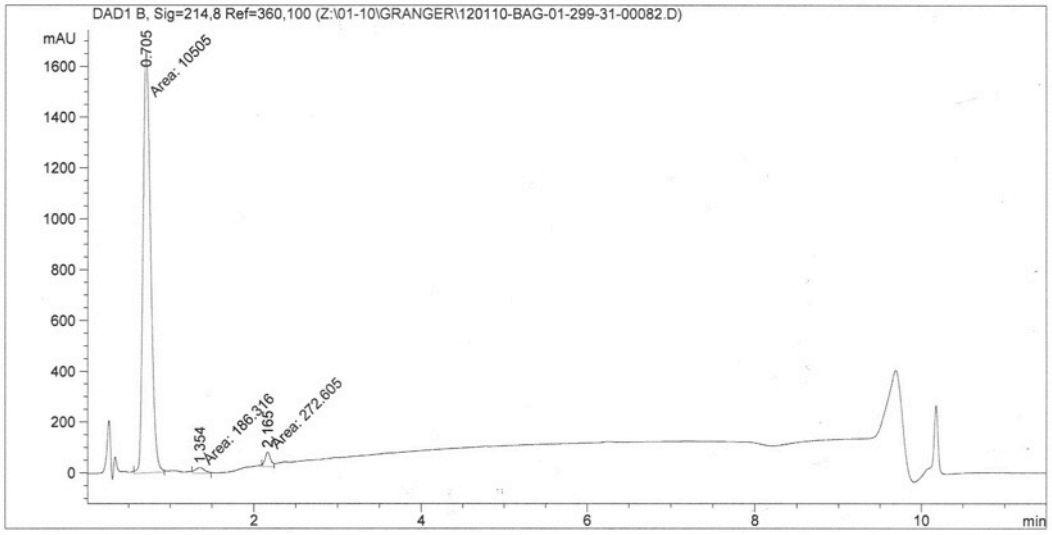
16{5,2}



600 MHz nmrox
BAG-1-229-3
exp4 Carbon
SAMPLE
date Feb 1 2010
solvent cdcl3
file exp
ACQUISITION
sw 40322.6
at 2.000
np 161290
fb 17000
bs 64
d1 2.000
nt 10000
ct 10000
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 33931.4
rfl 3566.1
rfp 0
rp 202.3
lp 21.4
PLOT
wc 250
sc 0
vs 75263
th 68
ai cdc ph
200
180
160
140
120
100
80
60
40
20
ppm

Data File Z:\01-10\GRANGER\120110-BAG-01-299-31-00082.D
Sample Name: BAG-01-299-3

```
    ======================================================================
    Acq. Operator    : brettag35@gmail.com
    Acq. Instrument  : LCMS                          Location : Vial 83
    Injection Date   : 1/12/2010 8:40:32 PM
                                                     Inj Volume : 1.0 µl
    Acq. Method      : C:\CHEM32\1\METHODS\SP NIH.M
    Last changed     : 1/12/2010 8:40:21 PM by brettag35@gmail.com
                       (modified after loading)
    Analysis Method  : C:\CHEM32\1\METHODS\DEF_LC.M
    Last changed     : 11/20/2006 4:14:44 AM
    Sample Info      : Easy-Access Method: 'SP NIH'
```



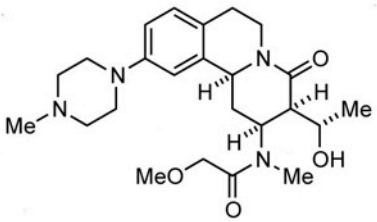

```
    ======================================================================
                              Area Percent Report
    ======================================================================

    Sorted By             :      Signal
    Multiplier:                   :      1.0000
    Dilution:                     :      1.0000
    Use Multiplier & Dilution Factor with ISTDs


    Signal 1: DAD1 B, Sig=214,8 Ref=360,100

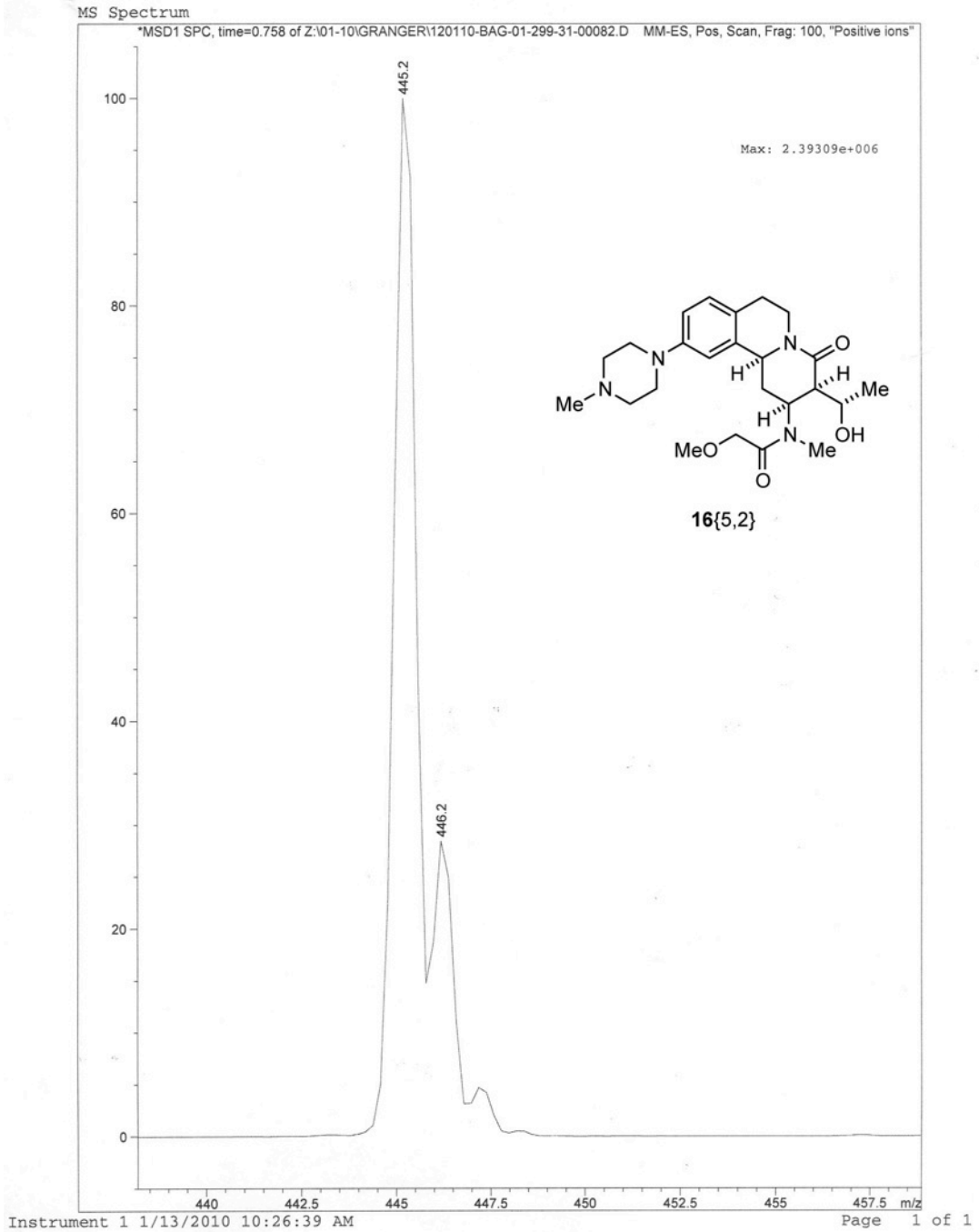
    Peak RetTime Type  Width     Area      Height     Area
      #   [min]        [min]   [mAU*s]     [mAU]       %
    ----|-------|----|-------|----------|----------|--------|
       1   0.705 MM    0.1055 1.05050e4  1660.28857  95.8143
       2   1.354 MM    0.1374  186.31616   22.59595   1.6994
       3   2.165 MM    0.0779  272.60535   58.30742   2.4864

    Totals :                  1.09639e4  1741.19193
```

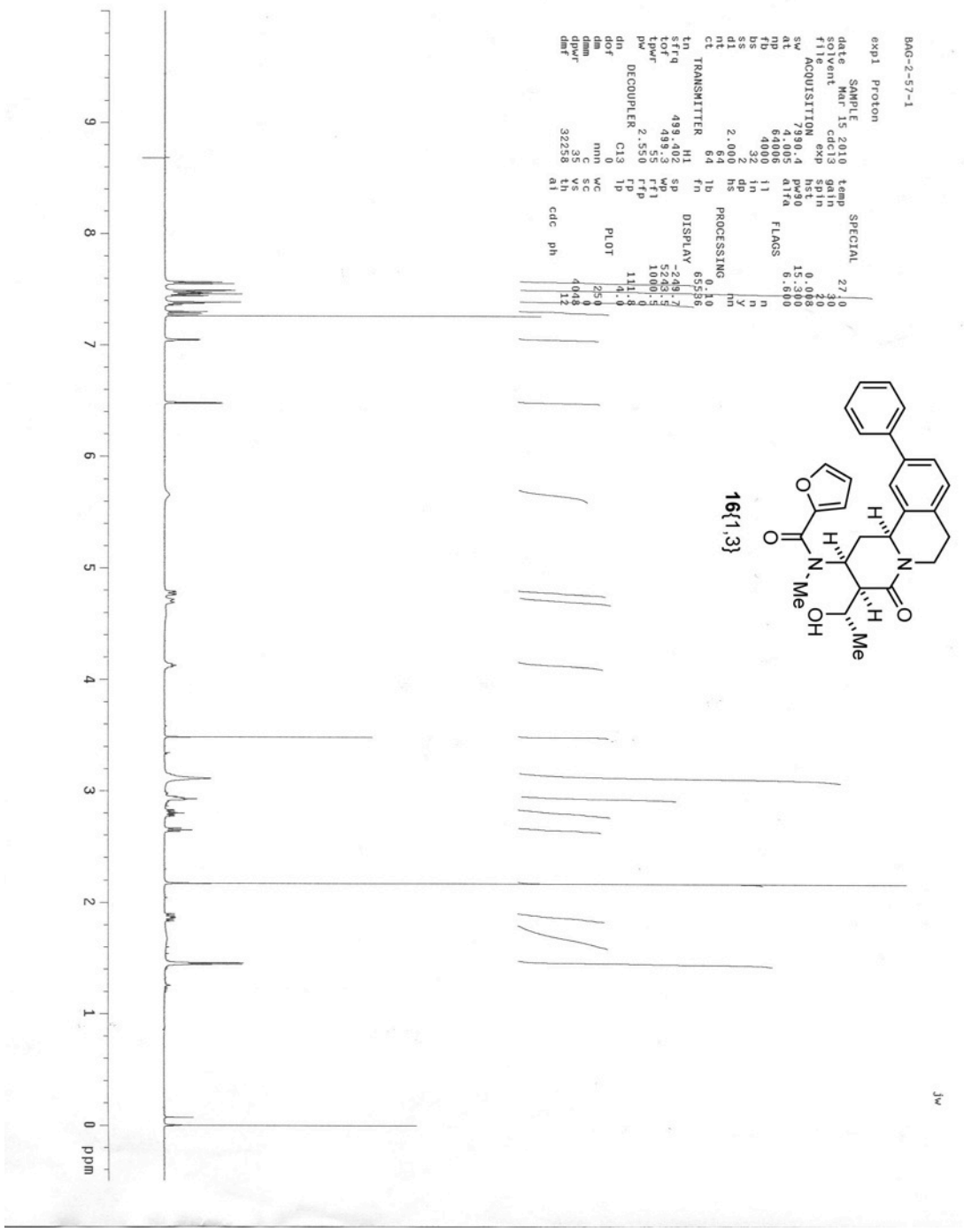


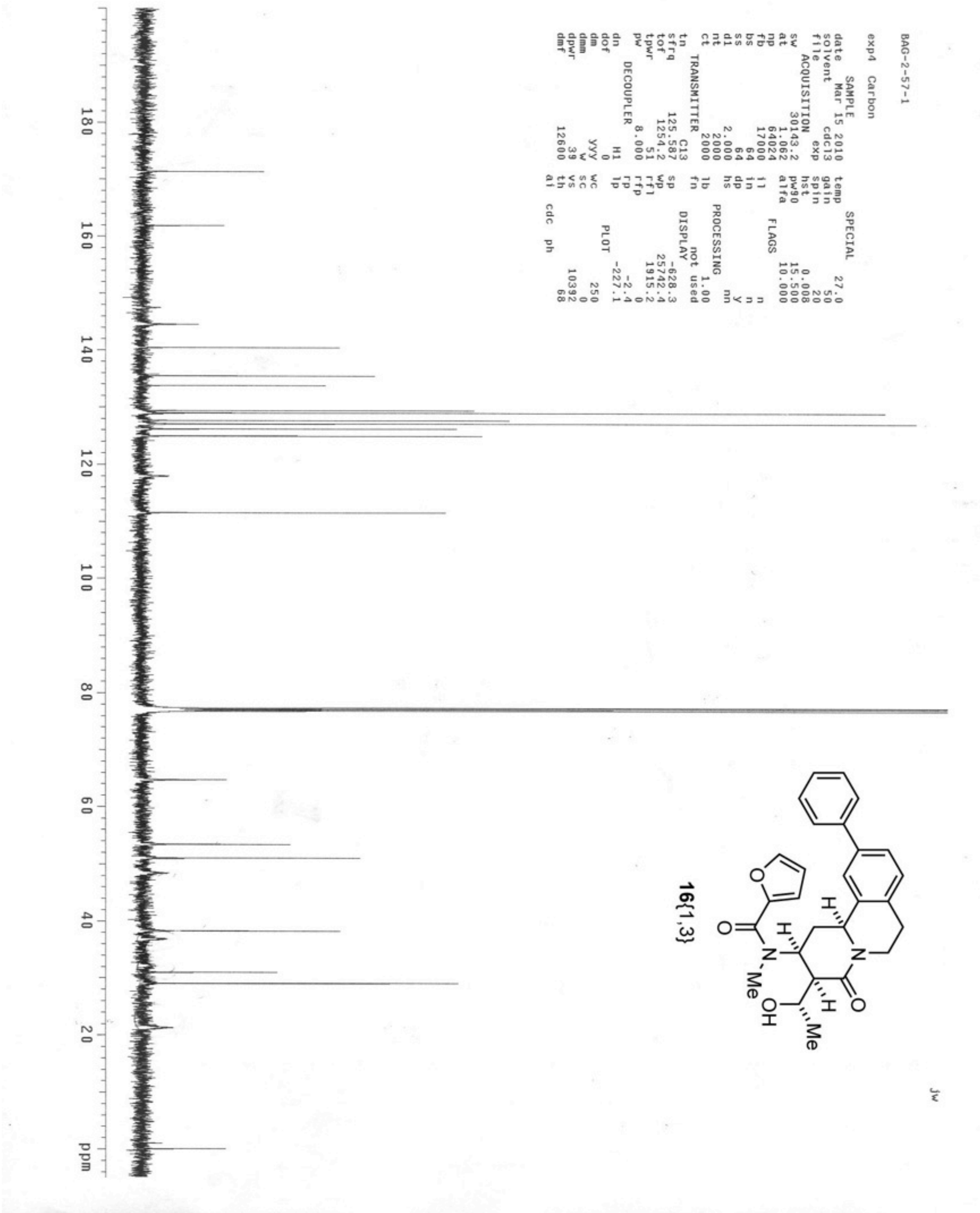
16{5,2}

Print of window 79: MS Spectrum



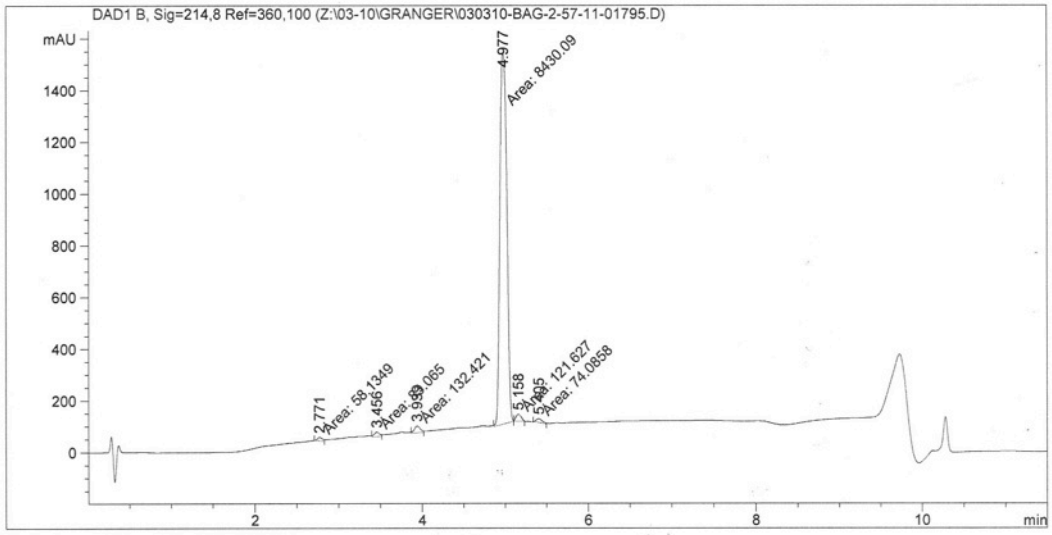
Instrument 1 1/13/2010 10:26:39 AM Page 1 of 1


BAG-2-57-1
exp1 Proton
16{1,3}
ppm


BAG-2-57-1
exp4 Carbon
16{1,3}
180
160
140
120
100
80
60
40
20
ppm

Data File Z:\03-10\GRANGER\030310-BAG-2-57-11-01795.D
Sample Name: BAG-2-57-1

```
==========================================================================
Acq. Operator   : brettag35@mail.utexas.edu
Acq. Instrument : LCMS                          Location : Vial 30
Injection Date  : 3/3/2010 9:01:24 PM
                                                Inj Volume : 1.0 µl
Acq. Method     : C:\CHEM32\1\METHODS\SP NIH.M
Last changed    : 3/3/2010 9:01:13 PM by brettag35@mail.utexas.edu
                  (modified after loading)
Analysis Method : C:\CHEM32\1\METHODS\DEF_LC.M
Last changed    : 3/4/2010 9:46:25 AM
                  (modified after loading)
Sample Info     : Easy-Access Method: 'SP NIH'
```

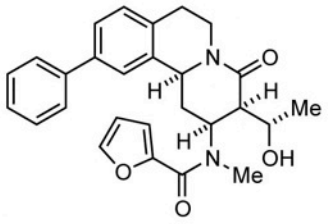



```
==========================================================================
                        Area Percent Report
==========================================================================

Sorted By             :      Signal
Multiplier:                   :      1.0000
Dilution:                     :      1.0000
Use Multiplier & Dilution Factor with ISTDs

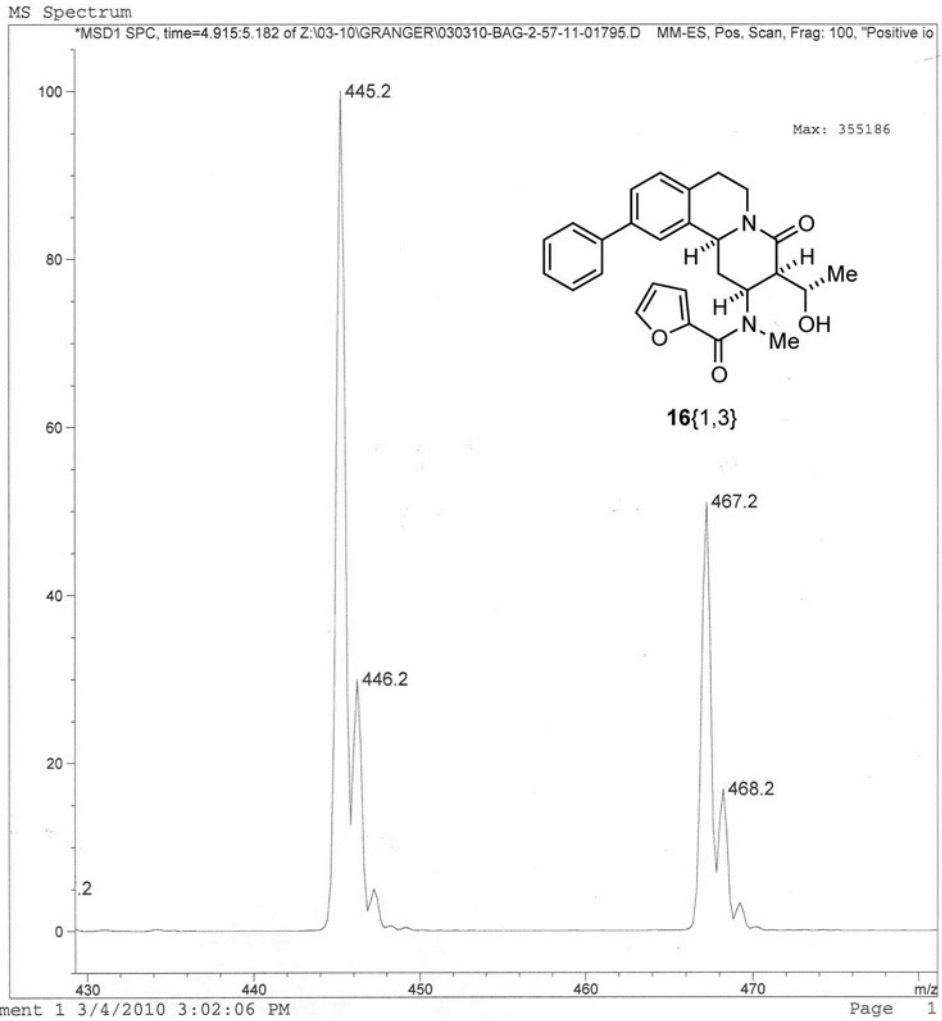

Signal 1: DAD1 B, Sig=214,8 Ref=360,100

Peak RetTime Type  Width     Area      Height     Area
  #   [min]       [min]    [mAU*s]     [mAU]       %
----|-------|----|-------|----------|----------|--------|
   1   2.771 MM    0.0725   58.13486   13.36314   0.6532
   2   3.456 MM    0.0758   83.06499   18.27118   0.9334
   3   3.939 MM    0.0844  132.42076   26.14830   1.4880
   4   4.977 MM    0.0975 8430.08887 1441.72473  94.7262
   5   5.158 MM    0.0737  121.62691   27.50731   1.3667
```

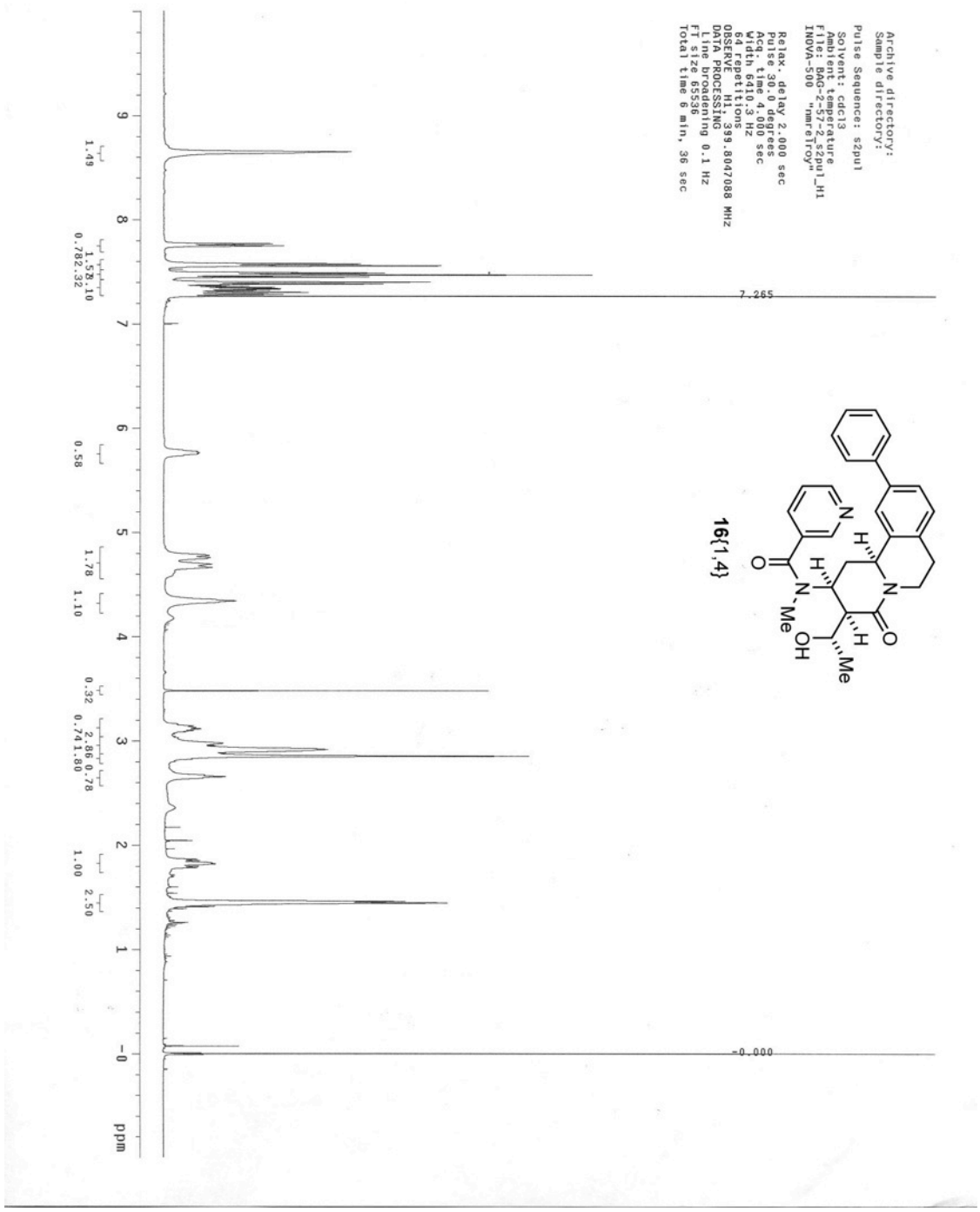


16{1,3}

```
Print of window 79: MS Spectrum
Data File  : Z:\03-10\GRANGER\030310-BAG-2-57-11-01795.D
Sample Name : BAG-2-57-1
=====================================================================
Acq. Operator   : brettag35@mail.utexas.edu
Acq. Instrument : LCMS                          Location : Vial 30
Injection Date  : 3/3/2010 9:01:24 PM               Inj :   1
                                                Inj Volume : 1.0 µl
Acq. Method     : C:\CHEM32\1\METHODS\SP NIH.M
Last changed    : 3/3/2010 9:01:13 PM by brettag35@mail.utexas.edu
                  (modified after loading)
Analysis Method : C:\CHEM32\1\METHODS\DEF_LC.M
Last changed    : 3/4/2010 9:46:25 AM
                  (modified after loading)
Sample Info     : Easy-Access Method: 'SP NIH'
```



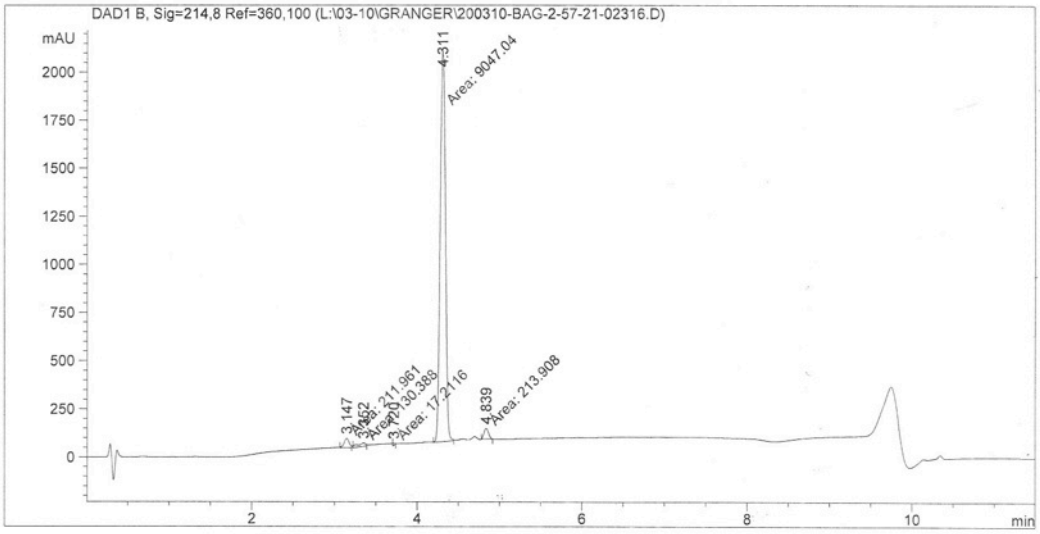
Instrument 1 3/4/2010 3:02:06 PM Page 1 of 1


Archive directory:
Sample directory:
Pulse Sequence: s2pul
Solvent: cdcl3
Ambient temperature
File: BAG-2-57-2_s2pul_H1
INOVA-500 "nmrelroy"
Relax. delay 2.000 sec
Pulse 30.0 degrees
Acq. time 4.000 sec
Width 6410.3 Hz
64 repetitions
OBSERVE H1, 399.8047088 MHz
DATA PROCESSING
Line broadening 0.1 Hz
FT size 65536
Total time 8 min, 36 sec
7.265
0.000
16(1,4)
1.49
1.52
1.10
0.78
0.58
1.78
1.10
0.32
2.86
0.78
1.80
1.00
2.50
ppm

BAG-2-57-2
exp2 Carbon
SAMPLE
date May 10 2010
solvent cdcl3
file exp
ACQUISITION
sw 30143.2
at 1.062
np 64024
fb 17000
bs 64
ss 64
d1 2.000
nt 2000
ct 1419
TRANSMITTER
tn C13
sfrq 125.587
tof 1254.2
tpwr 51
pw 8.000
DECOUPLER
dn H1
dof 0
dm yyy
dmm w
dpwr 39
dmf 12600
SPECIAL
temp 27.0
gain 50
spin 20
hst 0.008
pw90 15.500
alfa 10.000
FLAGS
il n
in n
dp y
hs nn
PROCESSING
lb 1.00
fn not used
DISPLAY
sp -628.1
wp 25742.4
rfl 11591.6
rfp 9669.2
rp -88.5
lp -229.9
PLOT
wc 250
sc 0
vs 4542
th 68
al cdc ph
16{1,4}
JW
180
160
140
120
100
80
60
40
20
ppm

Data File L:\03-10\GRANGER\200310-BAG-2-57-21-02316.D
Sample Name: BAG-2-57-2

```
==========================================================================
Acq. Operator   : brettag35@mail.utexas.edu
Acq. Instrument : LCMS                          Location : Vial 30
Injection Date  : 3/20/2010 2:10:54 PM
                                                Inj Volume : 1.0 µl
Acq. Method     : C:\CHEM32\1\METHODS\SP NIH.M
Last changed    : 3/20/2010 2:10:42 PM by brettag35@mail.utexas.edu
                  (modified after loading)
Analysis Method : C:\CHEM32\1\METHODS\DEF_LC.M
Last changed    : 11/20/2006 4:14:44 AM
Sample Info     : Easy-Access Method: 'SP NIH'
```

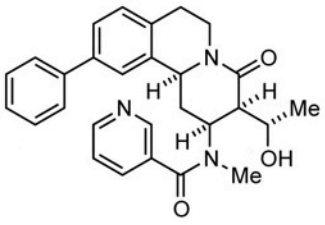



```
==========================================================================
                          Area Percent Report
==========================================================================

Sorted By             :      Signal
Multiplier:                   :      1.0000
Dilution:                     :      1.0000
Use Multiplier & Dilution Factor with ISTDs


Signal 1: DAD1 B, Sig=214,8 Ref=360,100

Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]   [mAU*s]     [mAU]       %
----|-------|----|-------|----------|----------|--------|
   1   3.147 MM    0.0702  211.96133   50.29136   2.2032
   2   3.352 MM    0.1167  130.38808   18.62537   1.3553
   3   3.720 MM    0.0251   17.21155   11.40865   0.1789
   4   4.311 MM    0.0740 9047.04102 2038.54919  94.0391
   5   4.839 MM    0.0638  213.90776   55.88526   2.2235
```



16{1,4}

Instrument 1 3/20/2010 3:04:01 PM

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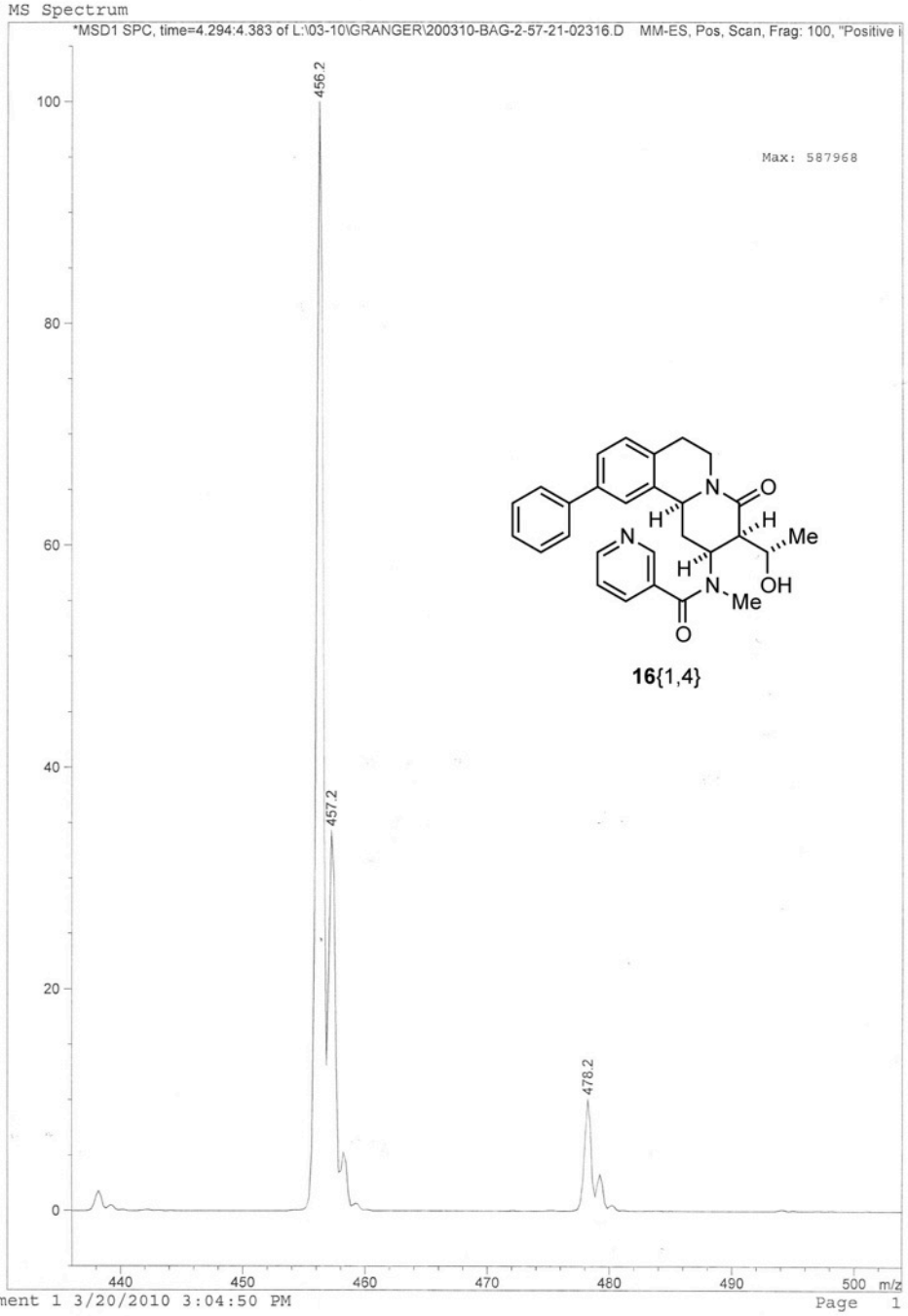
Print of window 79: MS Spectrum

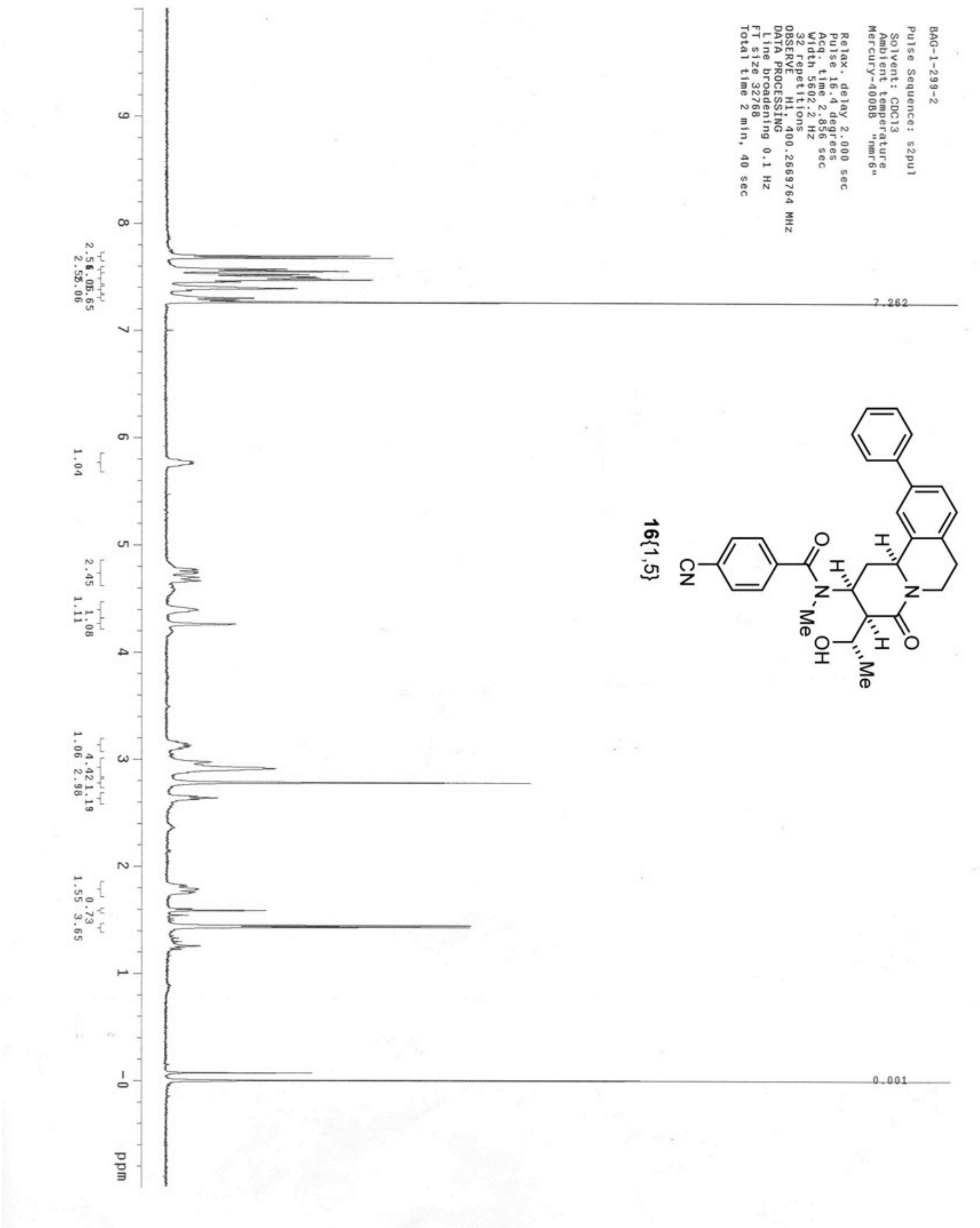
MS Spectrum

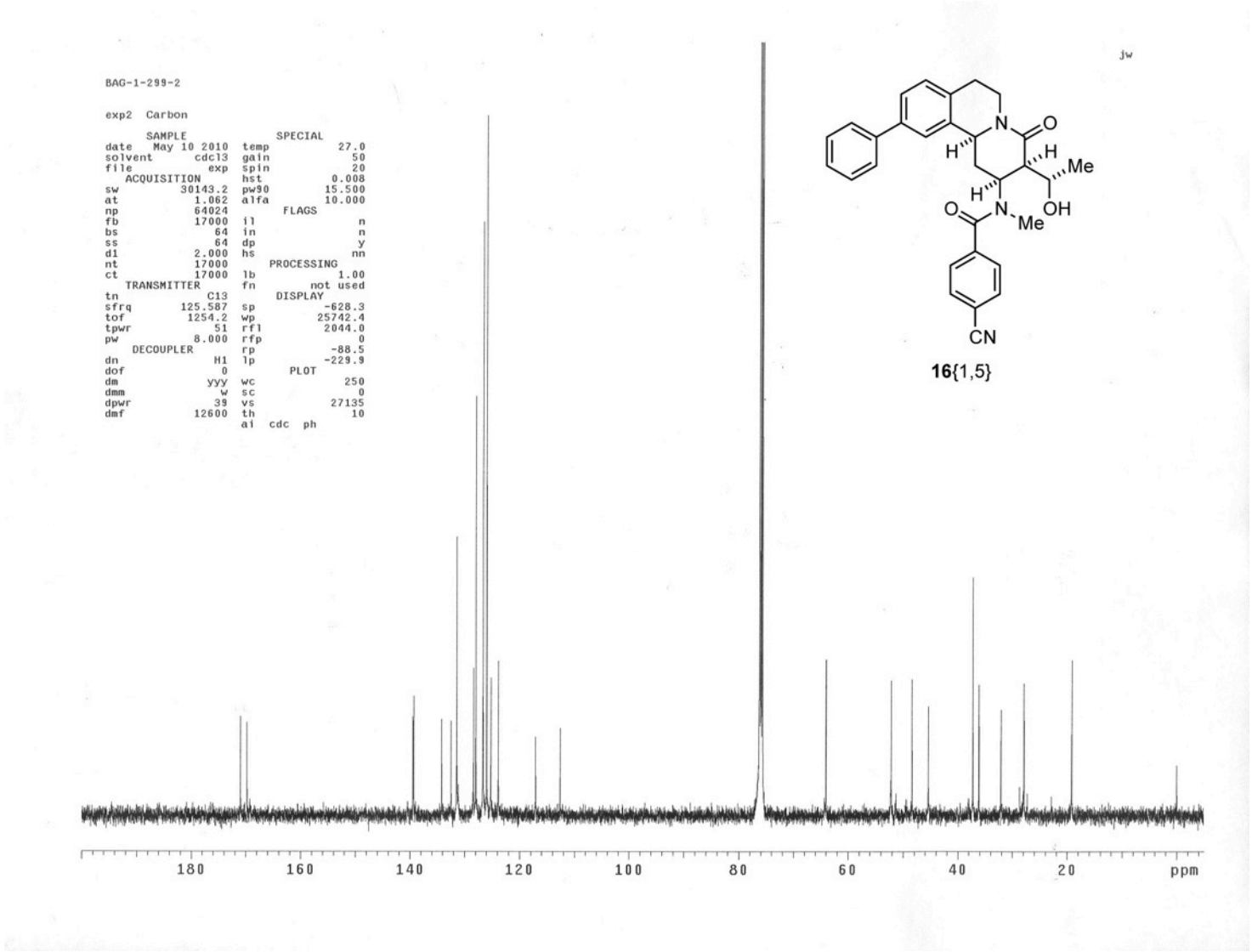


16{1,4}

Instrument 1 3/20/2010 3:04:50 PM

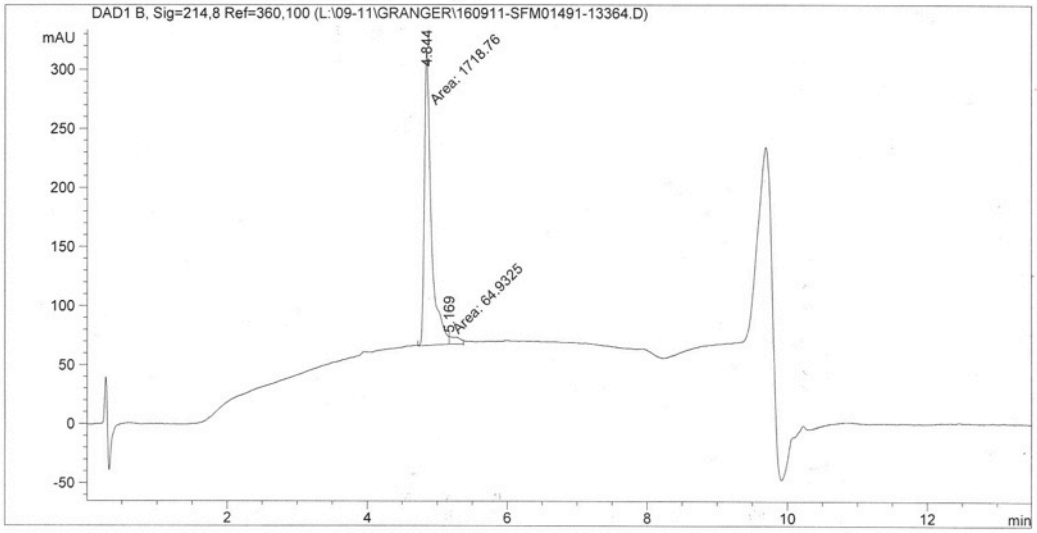
Page 1 of 1


BAG-1-299-2
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, 400.2669764 MHz
DATA PROCESSING
Line broadening 0.1 Hz
FT size 32768
Total time 2 min, 40 sec
7.262
0.001
16{1,5}
ppm


BAG-1-299-2
exp2 Carbon
SAMPLE
date May 10 2010
solvent cdcl3
file exp
ACQUISITION
sw 30143.2
at 1.062
np 64024
fb 17000
bs 64
ss 64
d1 2.000
nt 17000
ct 17000
TRANSMITTER
tn C13
sfrq 125.587
tof 1254.2
tpwr 51
pw 8.000
DECOUPLER
dn H1
dof 0
dm yyy
dmm w
dpwr 39
dmf 12600
SPECIAL
temp 27.0
gain 50
spin 20
hst 0.008
pw90 15.500
alfa 10.000
FLAGS
il n
in n
dp y
hs nn
PROCESSING
lb 1.00
fn not used
DISPLAY
sp -628.3
wp 25742.4
rfl 2044.0
rfp 0
rp -88.5
lp -229.9
PLOT
wc 250
sc 0
vs 27135
th 10
ai cdc ph
jw
16{1,5}
180
160
140
120
100
80
60
40
20
ppm

Data File L:\09-11\GRANGER\160911-SFM01491-13364.D
Sample Name: SFM0149

```
==========================================================================
Acq. Operator   : brettag35@mail.utexas.edu
Acq. Instrument : LCMS                          Location : Vial 45
Injection Date  : 9/17/2011 12:17:43 AM
                                                Inj Volume : 1.0 µl
Acq. Method     : C:\CHEM32\1\METHODS\SP NIH.M
Last changed    : 9/17/2011 12:17:28 AM by brettag35@mail.utexas.edu
                  (modified after loading)
Analysis Method : C:\CHEM32\1\METHODS\DEF_LC.M
Last changed    : 11/20/2006 4:14:44 AM
Sample Info     : Easy-Access Method: 'SP NIH'
```



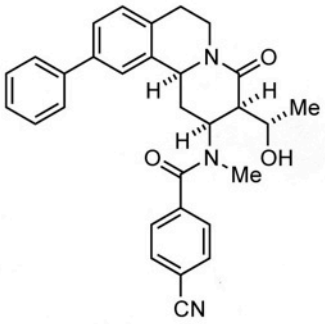

```
==========================================================================
                          Area Percent Report
==========================================================================

Sorted By             :      Signal
Multiplier:                   :      1.0000
Dilution:                     :      1.0000
Use Multiplier & Dilution Factor with ISTDs


Signal 1: DAD1 B, Sig=214,8 Ref=360,100

Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]   [mAU*s]     [mAU]       %
----|-------|----|-------|----------|----------|--------|
   1   4.844 MM    0.1142 1718.76440  250.80269  96.3597
   2   5.169 MM    0.1222   64.93247    6.38380   3.6403

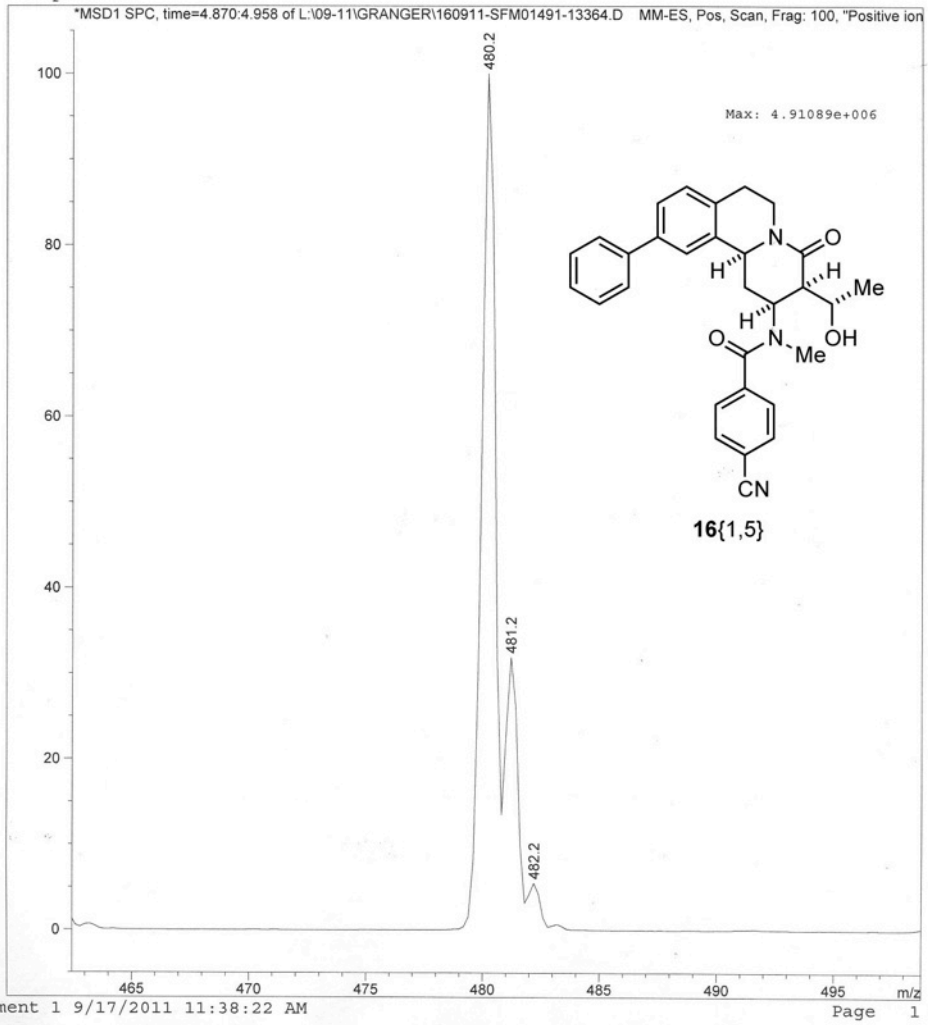
Totals :                  1783.69688  257.18649
```



16{1,5}

```
Print of window 79: MS Spectrum
Data File  : L:\09-11\GRANGER\160911-SFM01491-13364.D
Sample Name : SFM0149
======================================================================
Acq. Operator   : brettag35@mail.utexas.edu
Acq. Instrument : LCMS                          Location : Vial 45
Injection Date  : 9/17/2011 12:17:43 AM              Inj :   1
                                                Inj Volume : 1.0 µl
Acq. Method     : C:\CHEM32\1\METHODS\SP NIH.M
Last changed    : 9/17/2011 12:17:28 AM by brettag35@mail.utexas.edu
                  (modified after loading)
Analysis Method : C:\CHEM32\1\METHODS\DEF_LC.M
Last changed    : 11/20/2006 4:14:44 AM
Sample Info     : Easy-Access Method: 'SP NIH'
```

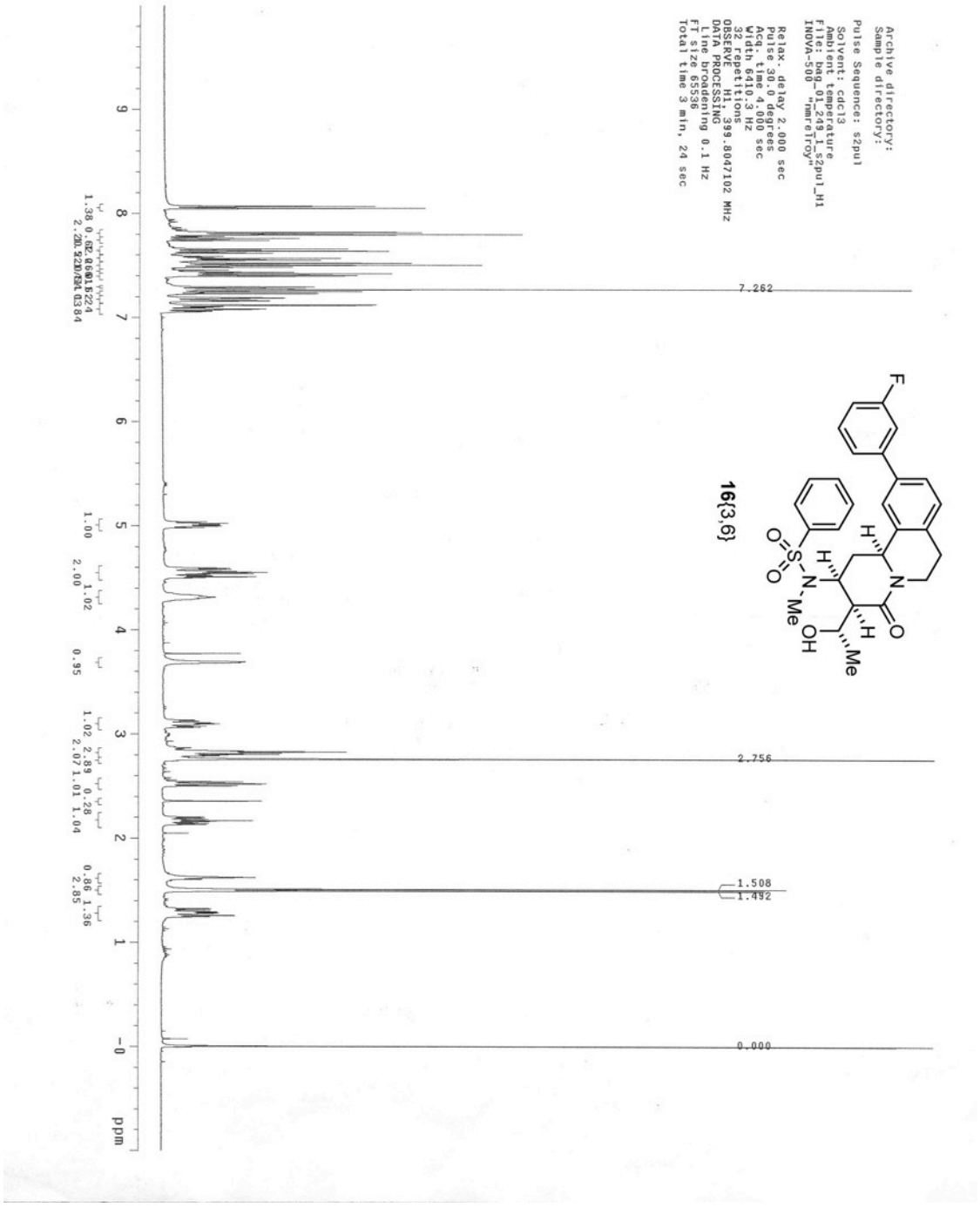
MS Spectrum

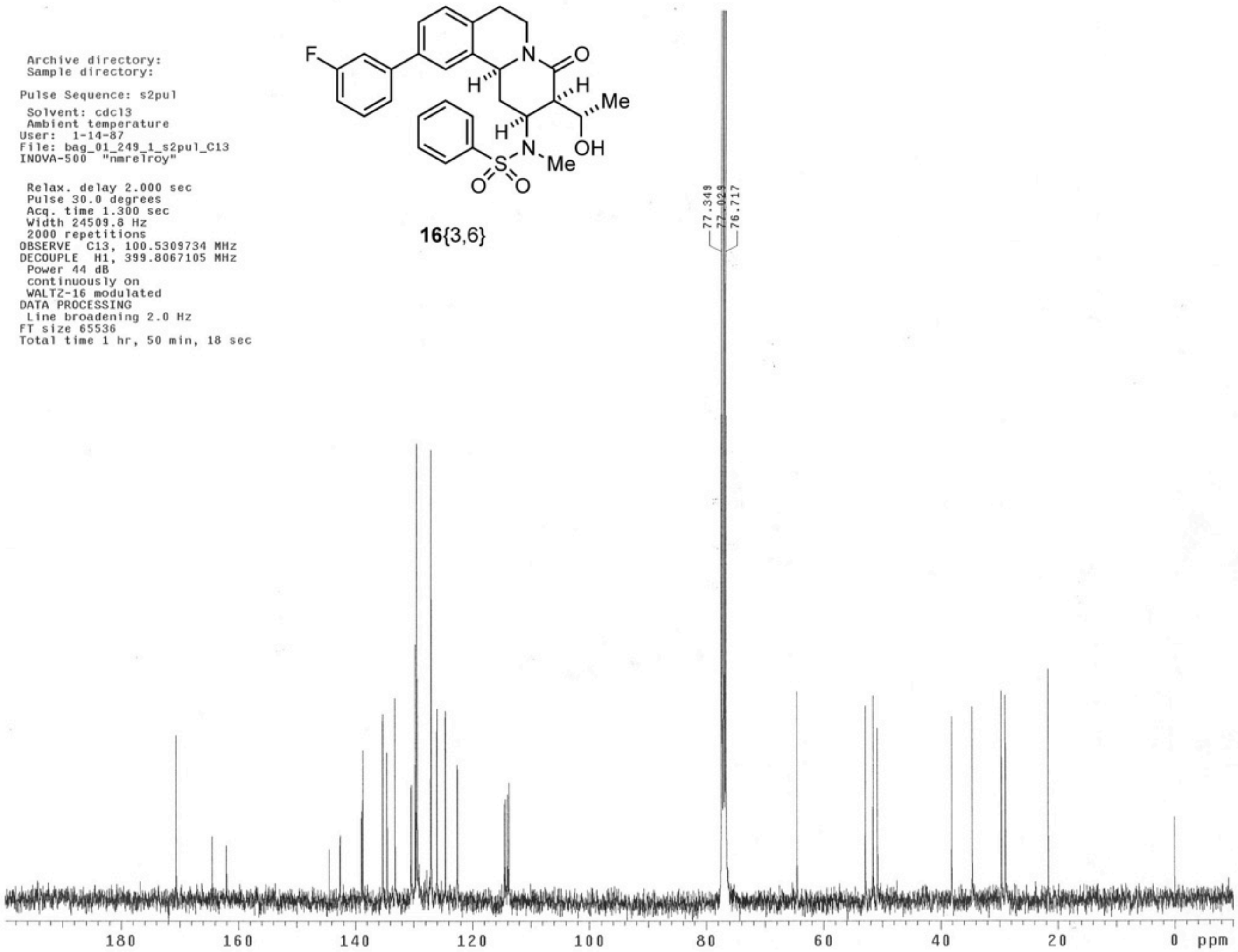


16{1,5}

Instrument 1 9/17/2011 11:38:22 AM

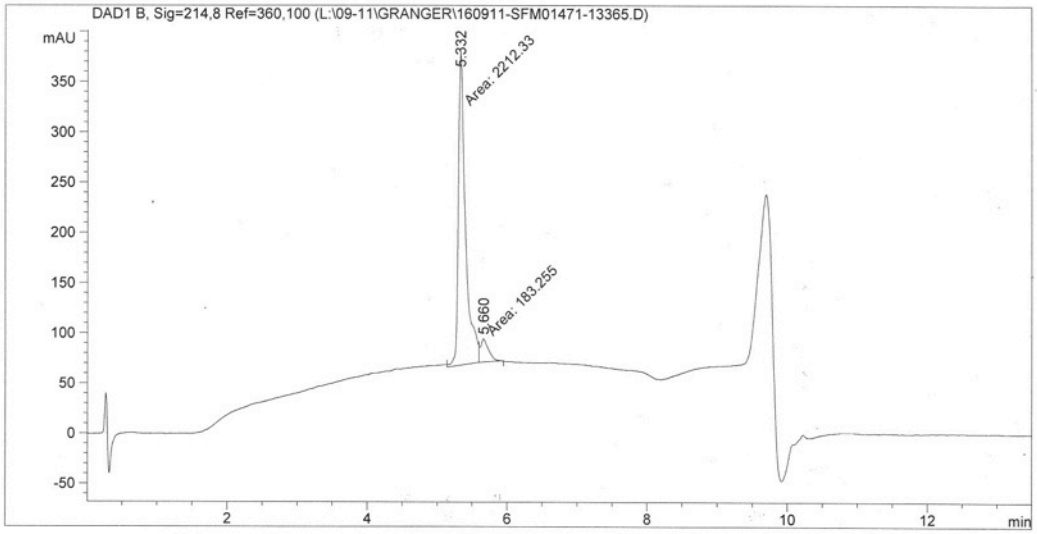
Page 1 of 1


Archive directory:
Sample directory:
Pulse Sequence: s2pul
Solvent: cdcl3
Ambient temperature
File: bag_01_249_1_s2pul_H1
INOVA-500 "nmrTroy"
Relax. delay 2.000 sec
Pulse 30.0 degrees
Acq. time 4.000 sec
Width 6410.3 Hz
32 repetitions
OBSERVE H1, 399.8047102 MHz
DATA PROCESSING
Line broadening 0.1 Hz
FT size 65536
Total time 3 min, 24 sec
16{3,6}
7.262
2.756
1.508
1.492
0.000
ppm


Archive directory:
Sample directory:
Pulse Sequence: s2pul
Solvent: cdcl3
Ambient temperature
User: 1-14-87
File: bag_01_249_1_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, 100.5309734 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
16{3,6}
77.349
77.029
76.717
180
160
140
120
100
80
60
40
20
0
ppm

Data File L:\09-11\GRANGER\160911-SFM01471-13365.D
Sample Name: SFM0147

```
======================================================================
Acq. Operator   : brettag35@mail.utexas.edu
Acq. Instrument : LCMS                          Location : Vial 46
Injection Date  : 9/17/2011 12:32:43 AM
                                                Inj Volume : 1.0 µl
Acq. Method     : C:\CHEM32\1\METHODS\SP NIH.M
Last changed    : 9/17/2011 12:32:29 AM by brettag35@mail.utexas.edu
                  (modified after loading)
Analysis Method : C:\CHEM32\1\METHODS\DEF_LC.M
Last changed    : 11/20/2006 4:14:44 AM
Sample Info     : Easy-Access Method: 'SP NIH'
```



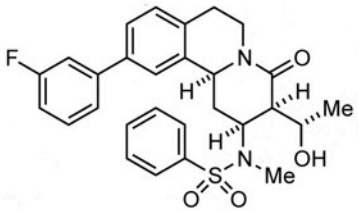

```
======================================================================
                        Area Percent Report
======================================================================

Sorted By             :      Signal
Multiplier:                   :      1.0000
Dilution:                     :      1.0000
Use Multiplier & Dilution Factor with ISTDs


Signal 1: DAD1 B, Sig=214,8 Ref=360,100

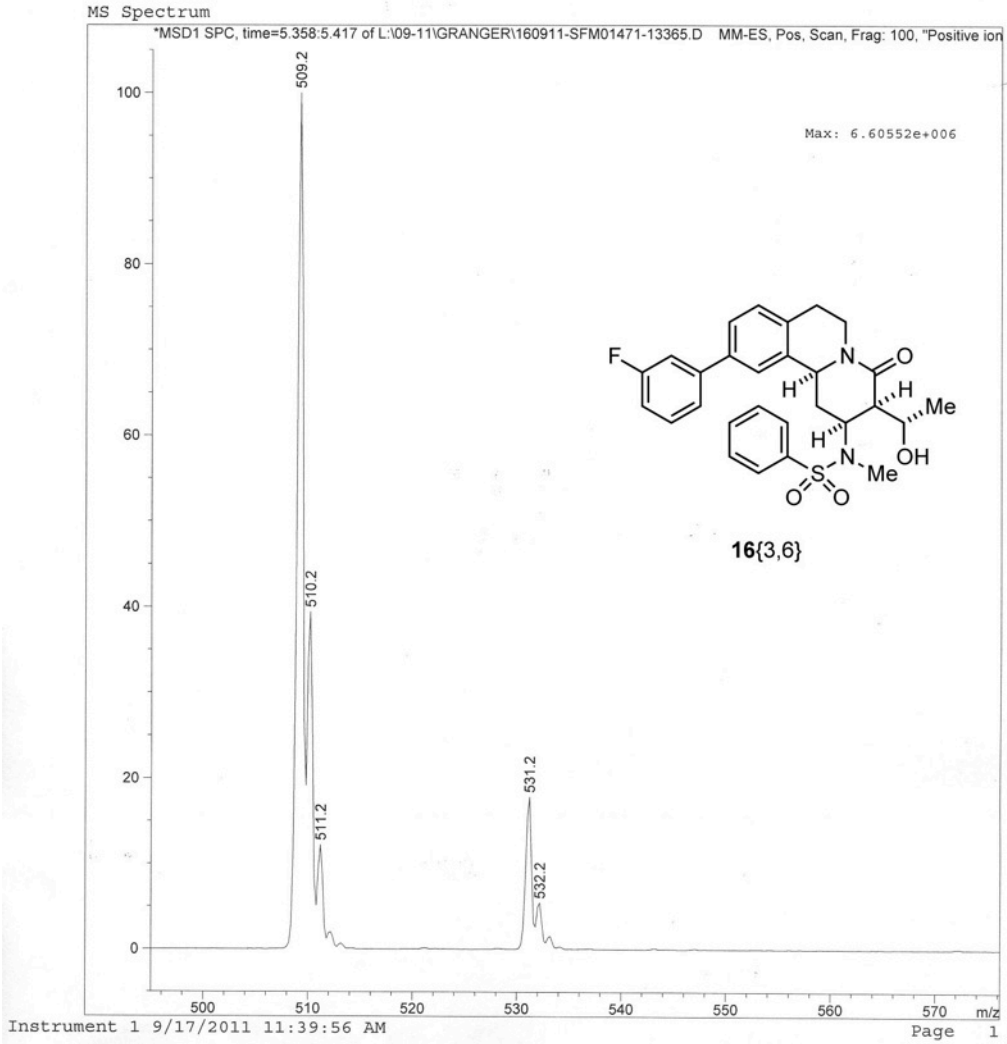
Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]   [mAU*s]     [mAU]       %
----|-------|----|-------|----------|----------|--------|
   1   5.332 MM    0.1180 2212.32764  312.42572  92.3503
   2   5.660 MM    0.1316  183.25522   23.20903   7.6497

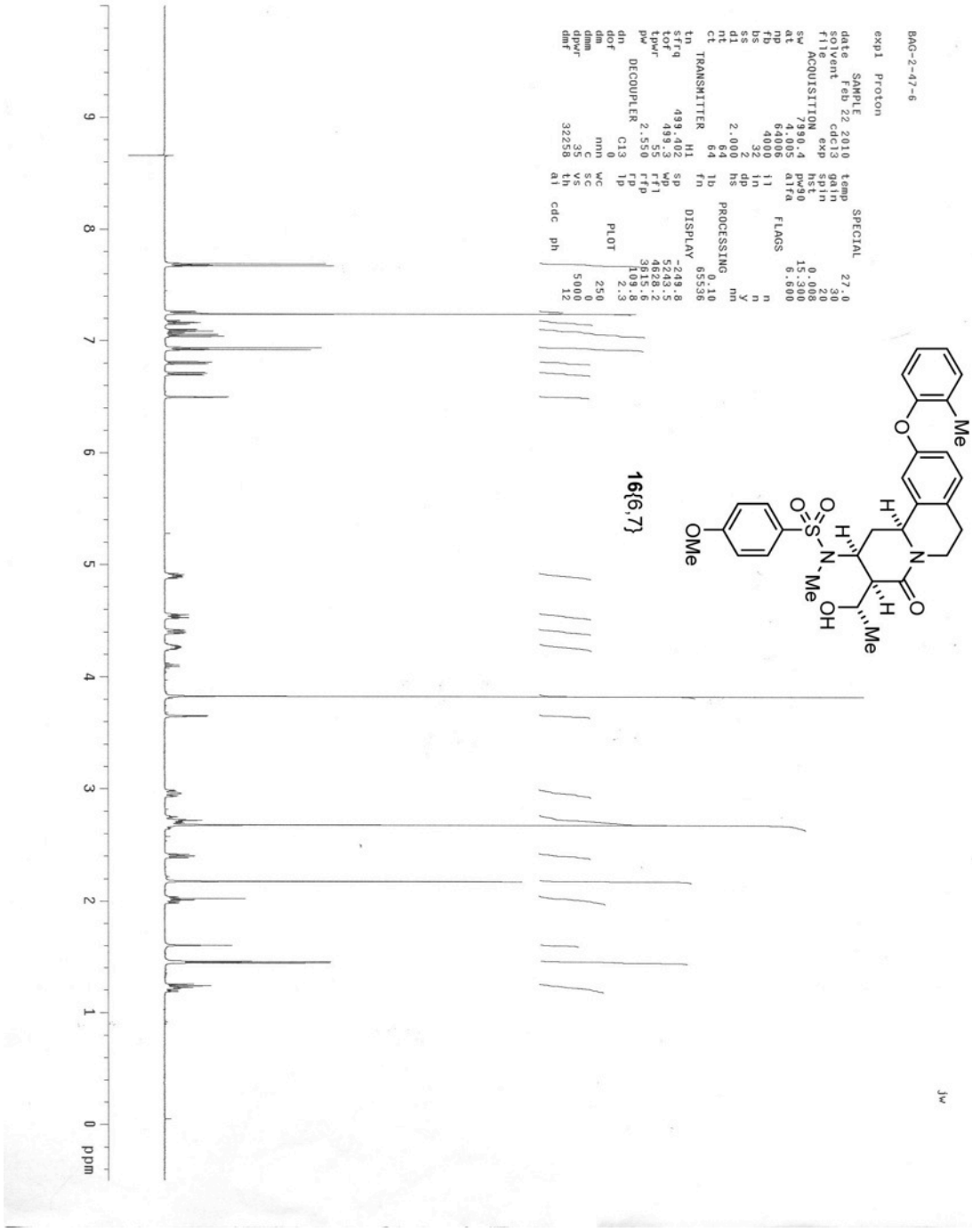
Totals :                  2395.58286  335.63475
```

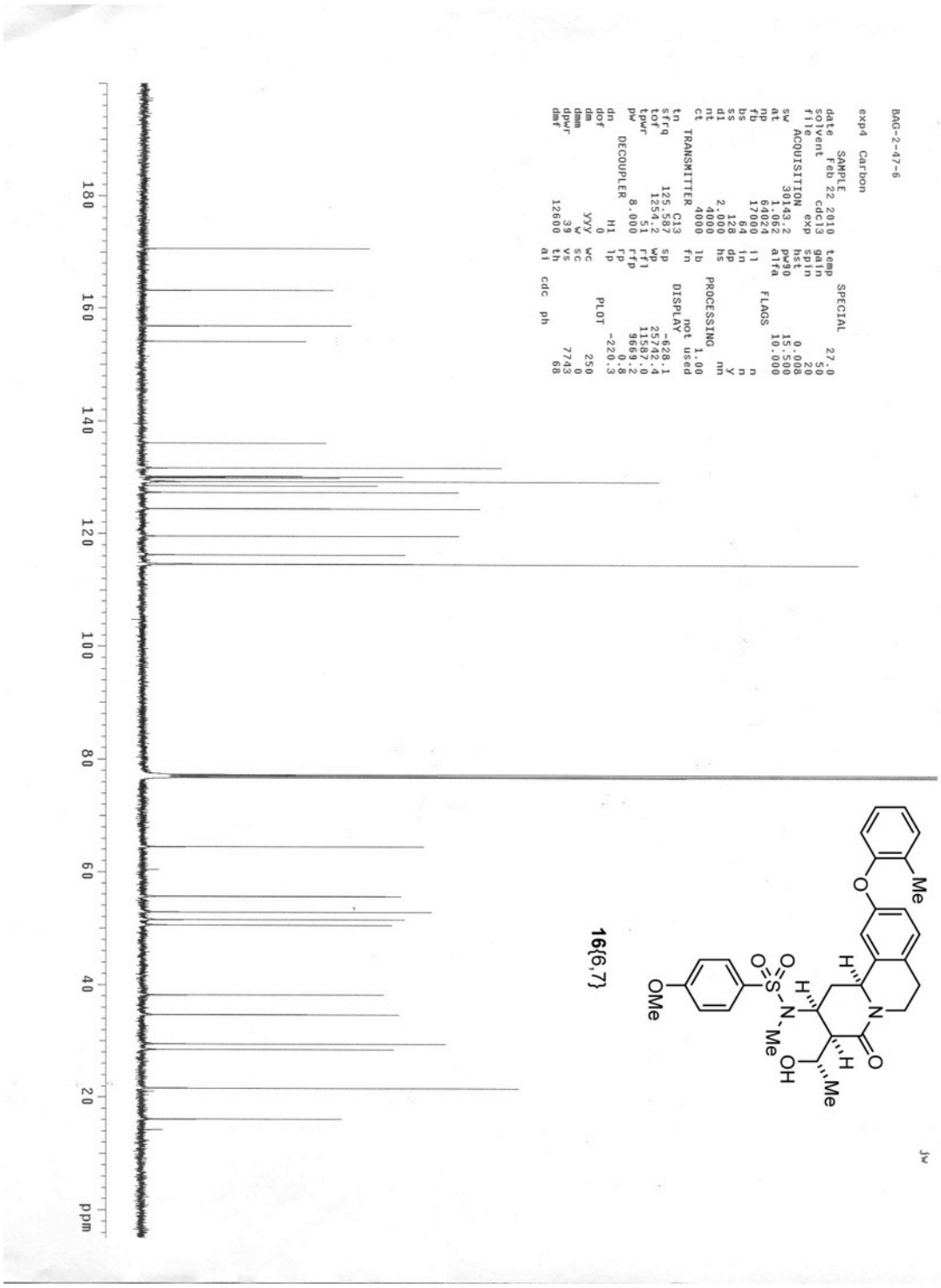


16{3,6}

```
Print of window 79: MS Spectrum
Data File  : L:\09-11\GRANGER\160911-SFM01471-13365.D
Sample Name : SFM0147
======================================================================
Acq. Operator   : brettag35@mail.utexas.edu
Acq. Instrument : LCMS                          Location : Vial 46
Injection Date  : 9/17/2011 12:32:43 AM             Inj :   1
                                                Inj Volume : 1.0 µl
Acq. Method     : C:\CHEM32\1\METHODS\SP NIH.M
Last changed    : 9/17/2011 12:32:29 AM by brettag35@mail.utexas.edu
                  (modified after loading)
Analysis Method : C:\CHEM32\1\METHODS\DEF_LC.M
Last changed    : 11/20/2006 4:14:44 AM
Sample Info     : Easy-Access Method: 'SP NIH'
```

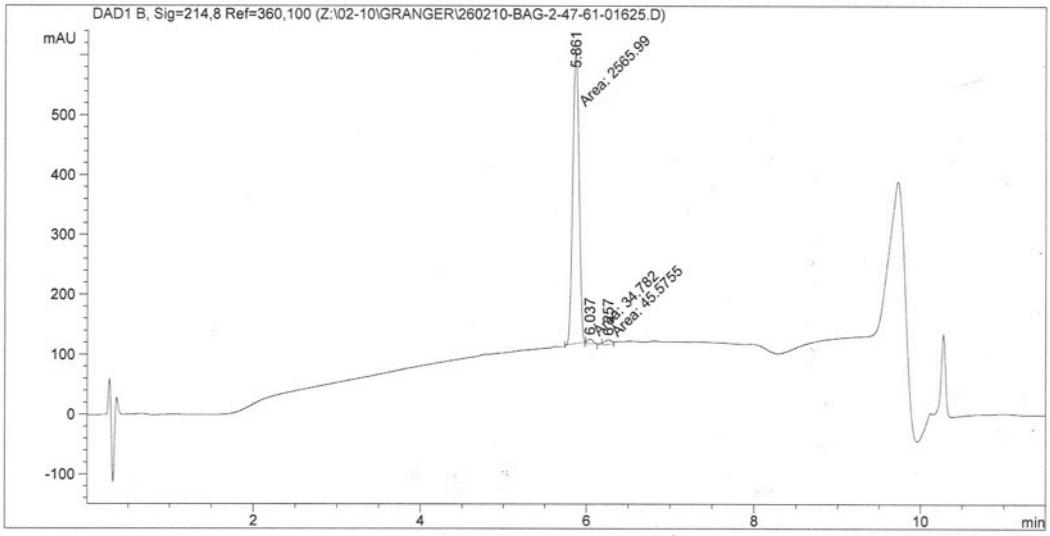



BAG-2-47-6
exp1 Proton
SAMPLE
date Feb 22 2010
solvent cdcl3
file exp
ACQUISITION
sw 7990.4
at 4.005
np 64006
fb 4000
bs 32
ss 2
sl 2.002
nt 64
ct 64
TRANSMITTER
tn H1
sfrq 499.402
tof 499.3
tpwr 55
pw 2.550
DECOUPLER
dn C13
dof 0
dm nnn
dmm c
dpwr 35
dmf 32258
temp
spin
hst
pw90
FLAGS
il
in
dp
hs
PROCESSING
lb
fn
DISPLAY
sp
wp
rfl
rfp
PLOT
wc
sc
vs
th
cdc
SPECIAL
27.0
20
0.008
15.300
6.600
n
n
y
nn
0.10
65536
-249.8
5243.5
4628.2
3615.6
109.8
2.3
250
0
5000
12
ph
Me
O
H
N
O
H
Me
O
S
N
Me
OH
OMe
16{6.7}
9
8
7
6
5
4
3
2
1
0 ppm


BAG-2-47-6
exp4 Carbon
SAMPLE
date Feb 22 2010
solvent cdcl3
file exp
ACQUISITION
sw 30143.2
at 1.062
np 64024
fb 17000
bs 64
ss 128
sd 2.000
nt 4000
ct 4000
TRANSMITTER
tn C13
sfrq 125.587
tof 1254.2
tpwr 51
pw 8.000
DECOUPLER
dn H1
dof 0
dm yyy
dmm w
dpwr 39
dmf 12600
SPECIAL
temp 27.0
spin 50
hst 20
pw90 0.008
alfa 15.500
FLAGS 10.000
il n
in n
dp y
hs nn
PROCESSING
lb 1.00
fn not used
DISPLAY
sp -628.1
wp 25742.4
rfl 11587.0
rfp 9669.2
rp 0.8
lp -220.3
PLOT
wc 250
sc 0
vs 7743
th 68
al cdc ph
Me
O
H
O
S
N
Me
OH
H
Me
H
N
O
OMe
16{6,7}
Jw
180
160
140
120
100
80
60
40
20
ppm

Data File Z:\02-10\GRANGER\260210-BAG-2-47-61-01625.D
Sample Name: BAG-2-47-6

```
Acq. Operator    : brettag35@mail.utexas.edu
Acq. Instrument : LCMS                          Location : Vial 55
Injection Date  : 2/26/2010 9:09:26 PM
                                                Inj Volume : 1.0 µl
Acq. Method     : C:\CHEM32\1\METHODS\SP NIH.M
Last changed    : 2/26/2010 9:09:15 PM by brettag35@mail.utexas.edu
                  (modified after loading)
Analysis Method : C:\CHEM32\1\METHODS\DEF_LC.M
Last changed    : 2/25/2010 3:55:13 PM
                  (modified after loading)
Sample Info     : Easy-Access Method: 'SP NIH'
```



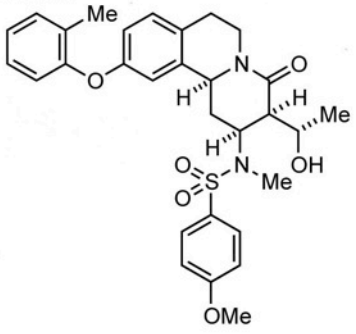
Area Percent Report

```
Sorted By             :      Signal
Multiplier:                   :      1.0000
Dilution:                     :      1.0000
Use Multiplier & Dilution Factor with ISTDs


Signal 1: DAD1 B, Sig=214,8 Ref=360,100

Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]   [mAU*s]     [mAU]       %
----|-------|----|-------|----------|----------|--------|
   1   5.861 MM    0.0875 2565.99365  488.58817  96.9635
   2   6.037 MM    0.0752   34.78198    7.71055   1.3143
   3   6.257 MM    0.1006   45.57550    7.54726   1.7222

Totals :                  2646.35113  503.84597
```

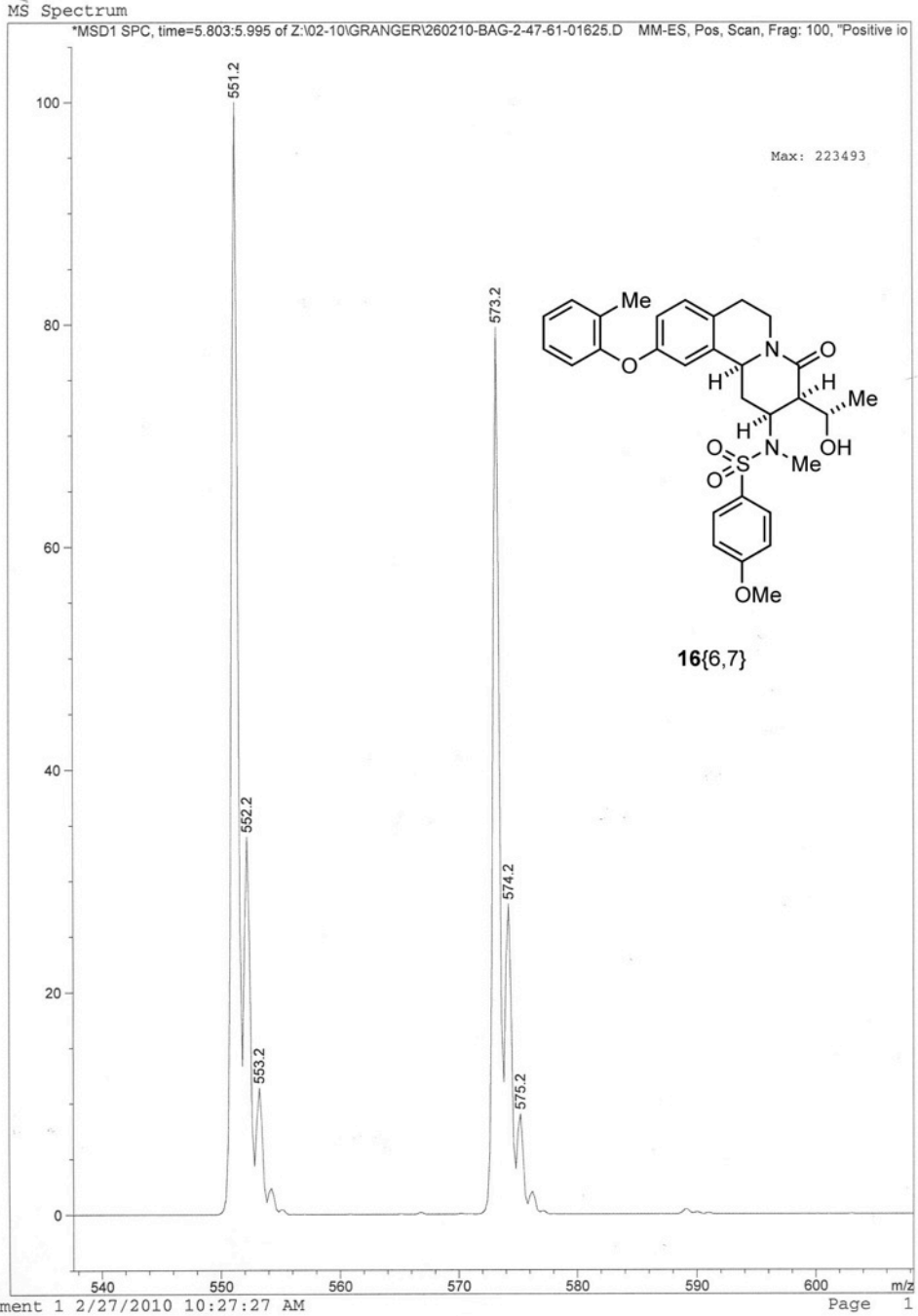


16{6,7}

Instrument 1 2/27/2010 10:25:55 AM

Print of window 79: MS Spectrum

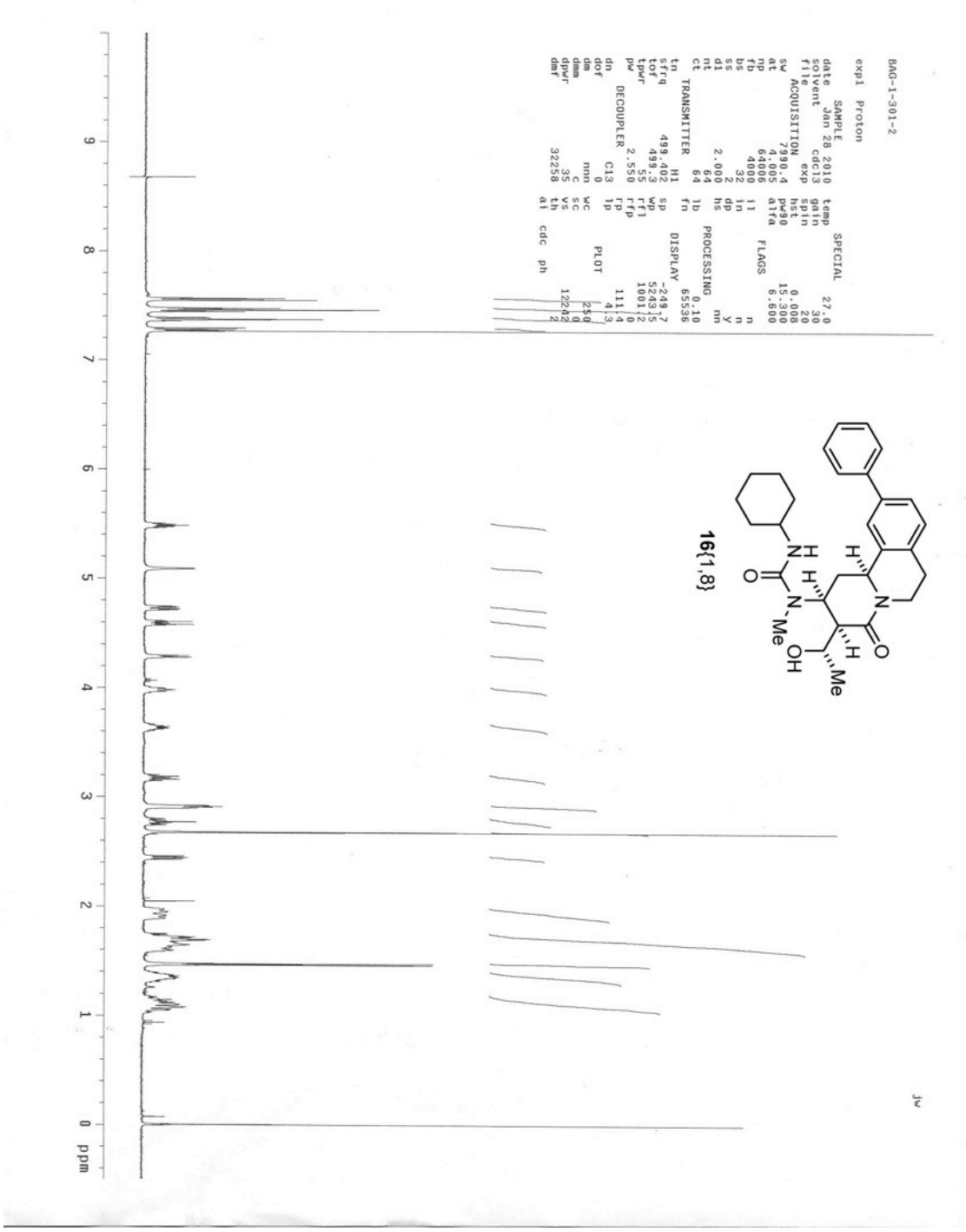
MS Spectrum

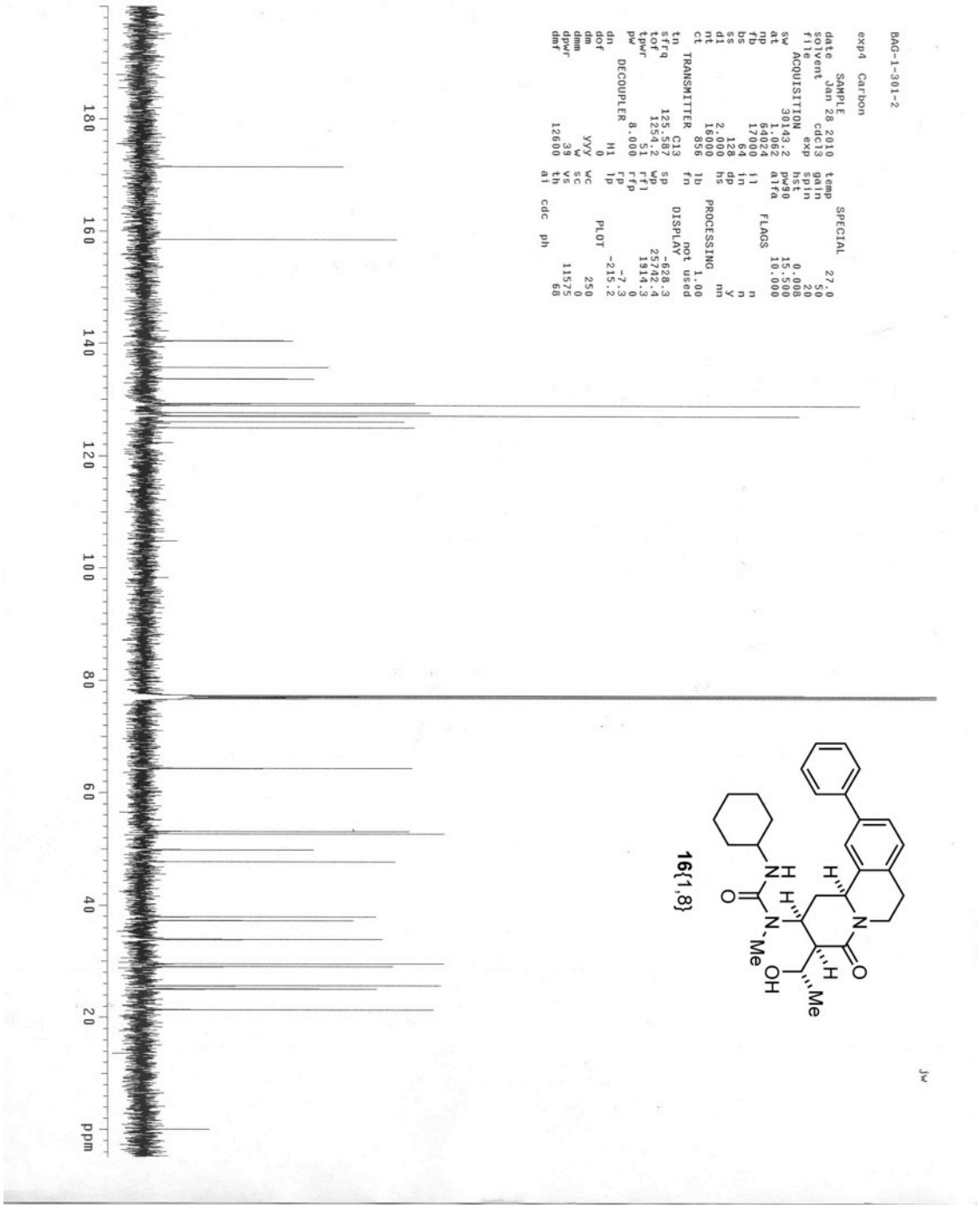


16{6,7}

Instrument 1 2/27/2010 10:27:27 AM

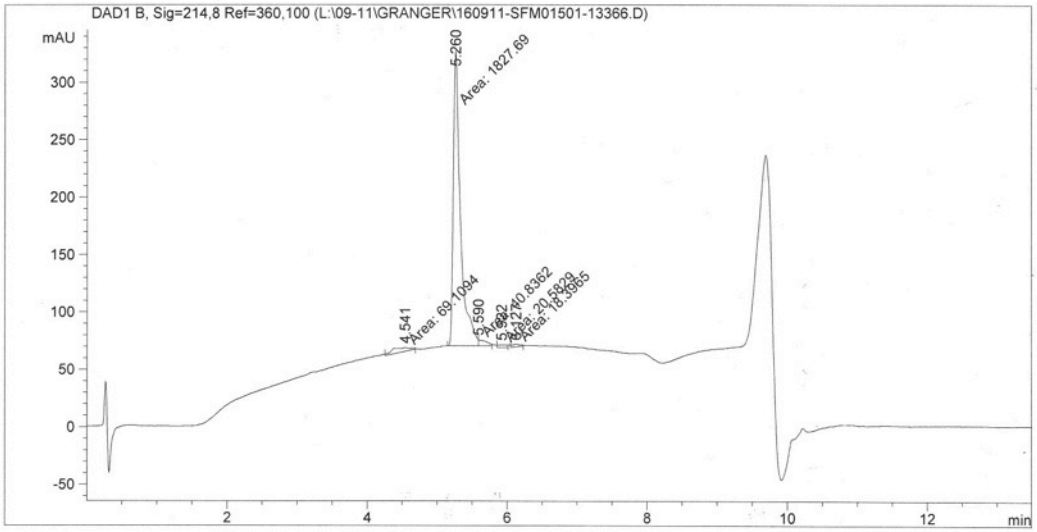
Page 1 of 1


BAG-1-301-2
exp1 Proton
SAMPLE
date Jan 28 2010
solvent
file
ACQUISITION
sw
at
np
fb
bs
ss
d1
nt
ct
TRANSMITTER
tn H1
sfrq 499.402
tof 499.3
tpwr 55
pw 2.550
DECOUPLER
dn C13
dof 0
dm nnn
dmm c
dpwr 35
dmf 32258
temp
gain
spin
hst
pw90
alfa
FLAGS
PROCESSING
lb
fn
DISPLAY
sp
wp
rfl
rfp
PLOT
wc
sc
vs
th
cdc
ph
SPECIAL
27.0
30
20
0.008
15.300
6.600
n
n
y
nn
0.10
65536
-249.7
5243.5
1001.2
111.0
41.3
250.0
12242.2
16{1,8}
jw
9
8
7
6
5
4
3
2
1
0 ppm


BAG-1-301-2
exp4 Carbon
SAMPLE
date Jan 28 2010
solvent
file
ACQUISITION
sw 30143.2
at 1.062
np 64024
fb 17000
bs 4
ss 128
d1 2.000
nt 16000
ct 856
TRANSMITTER
tn C13
sfrq 125.587
tof 1254.2
tpwr 51
pw 8.000
DECOUPLER
dn H1
dof 0
dm yyy
dmm w
dpwr 39
dmf 12600
SPECIAL
temp 27.0
gain 50
spin 20
hst 0.008
pw90 15.500
alfa 10.000
FLAGS
il n
in n
dp y
hs nn
PROCESSING
lb 1.00
fn not used
DISPLAY
sp -628.3
wp 25742.4
rfl 1914.3
rfp 0
rlp -7.3
tp -215.2
PLOT
wc 250
sc 0
vs 11575
th 68
ai cdc ph
16{1,8}
180
160
140
120
100
80
60
40
20
ppm

Data File L:\09-11\GRANGER\160911-SFM01501-13366.D
Sample Name: SFM0150

```
    ======================================================================
    Acq. Operator    : brettag35@mail.utexas.edu
    Acq. Instrument  : LCMS                          Location : Vial 47
    Injection Date   : 9/17/2011 12:47:43 AM
                                                     Inj Volume : 1.0 µl
    Acq. Method      : C:\CHEM32\1\METHODS\SP NIH.M
    Last changed     : 9/17/2011 12:47:28 AM by brettag35@mail.utexas.edu
                       (modified after loading)
    Analysis Method  : C:\CHEM32\1\METHODS\DEF_LC.M
    Last changed     : 11/20/2006 4:14:44 AM
    Sample Info      : Easy-Access Method: 'SP NIH'
```

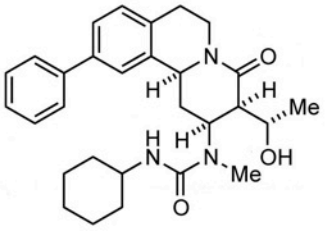



```
    ======================================================================
                              Area Percent Report
    ======================================================================

    Sorted By             :      Signal
    Multiplier:                   :      1.0000
    Dilution:                     :      1.0000
    Use Multiplier & Dilution Factor with ISTDs

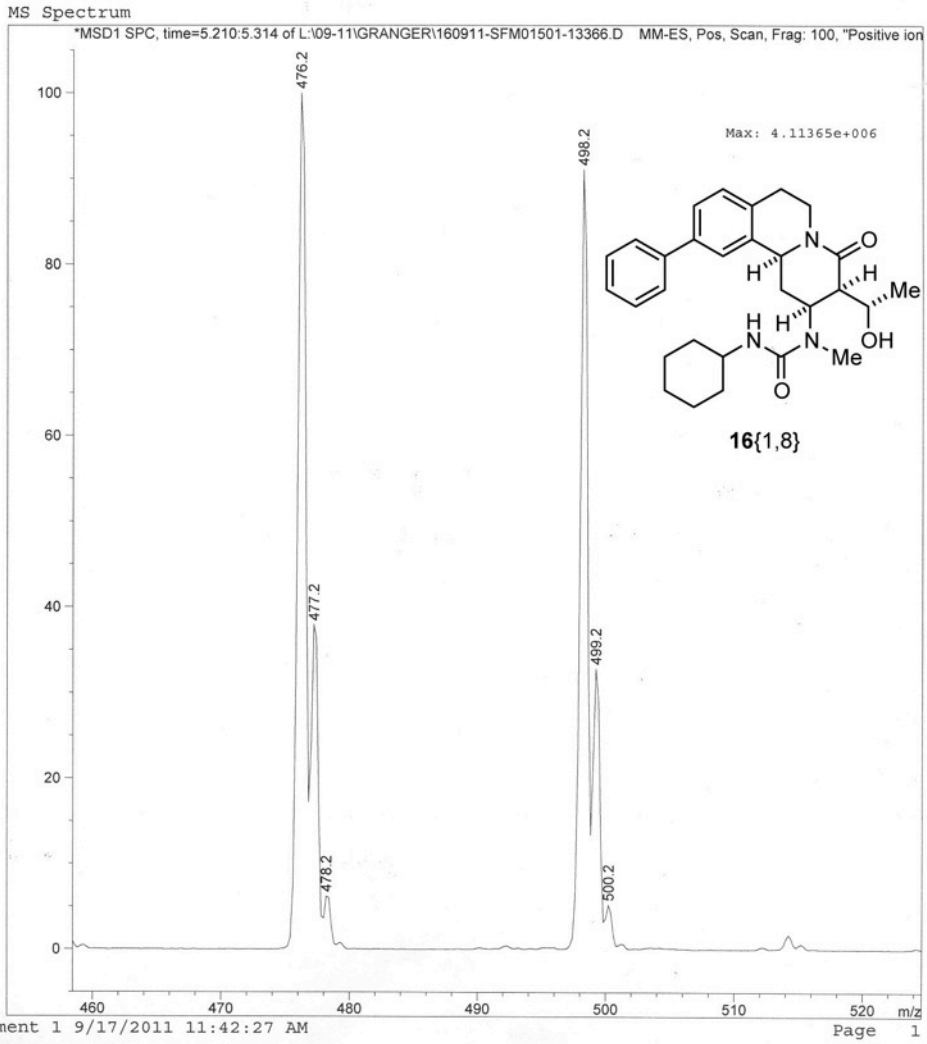

    Signal 1: DAD1 B, Sig=214,8 Ref=360,100

    Peak RetTime Type  Width     Area      Height     Area
      #   [min]       [min]   [mAU*s]     [mAU]       %
    ----|-------|----|-------|----------|----------|--------|
       1   4.541 MM    0.3433   69.10943    3.35512   3.4964
       2   5.260 MM    0.1184 1827.68750  257.19733  92.4656
       3   5.590 MM    0.0993   40.83616    5.43133   2.0660
       4   5.922 MM    0.1474   20.58289    2.32797   1.0413
       5   6.127 MM    0.1284   18.39647    2.38740   0.9307
```



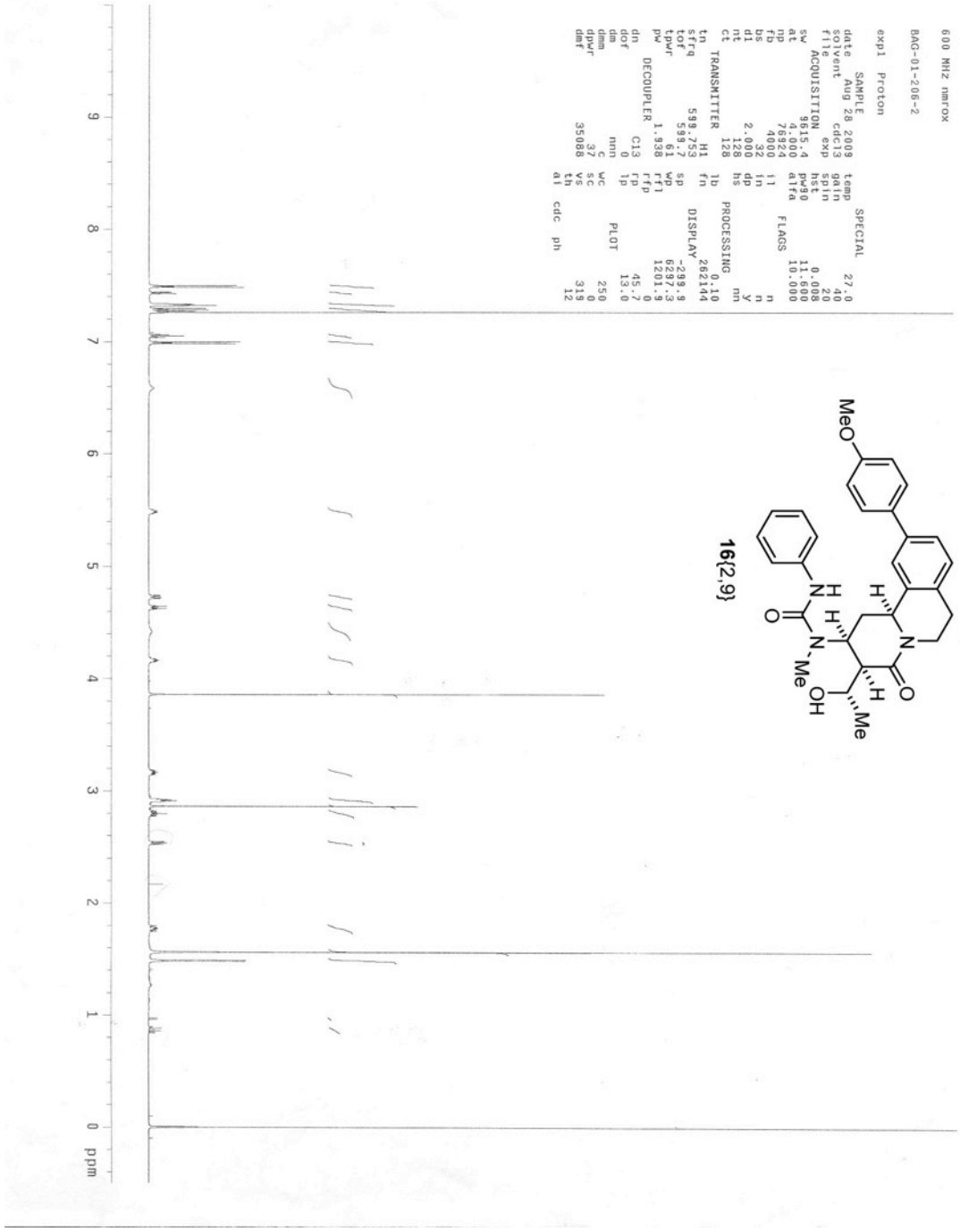
16{1,8}

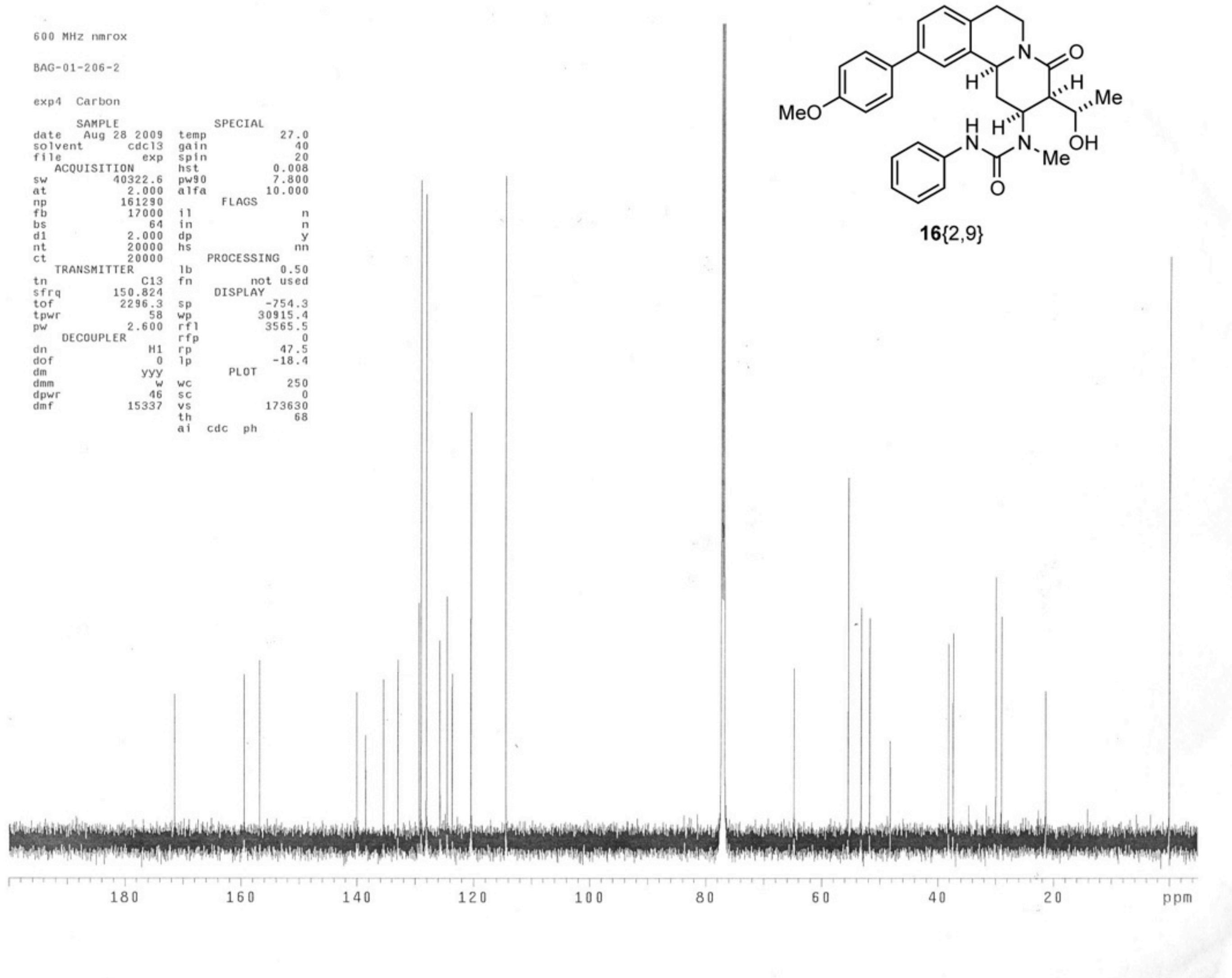
```
Print of window 79: MS Spectrum
Data File  : L:\09-11\GRANGER\160911-SFM01501-13366.D
Sample Name : SFM0150
======================================================================
Acq. Operator   : brettag35@mail.utexas.edu
Acq. Instrument : LCMS                          Location : Vial 47
Injection Date  : 9/17/2011 12:47:43 AM              Inj :   1
                                                Inj Volume : 1.0 µl
Acq. Method     : C:\CHEM32\1\METHODS\SP NIH.M
Last changed    : 9/17/2011 12:47:28 AM by brettag35@mail.utexas.edu
                  (modified after loading)
Analysis Method : C:\CHEM32\1\METHODS\DEF_LC.M
Last changed    : 11/20/2006 4:14:44 AM
Sample Info     : Easy-Access Method: 'SP NIH'
```



Instrument 1 9/17/2011 11:42:27 AM

Page 1 of 1

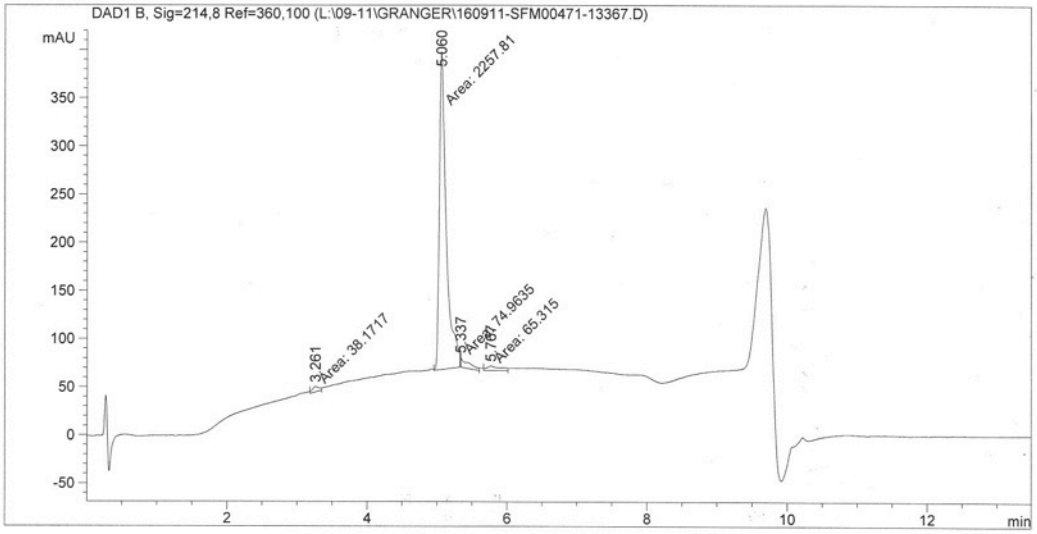

600 MHz nmrox
BAG-01-206-2
exp1 Proton
SAMPLE
date Aug 28 2009
solvent cdcl3
file ACQUISITION
sw 9615.4
at 4.000
np 76924
fb 4000
bs 32
d1 2.000
nt 128
ct 128
TRANSMITTER
tn H1
sfrq 599.753
tof 599.7
tpwr 61
pw 1.938
DECOUPLER
dn C13
dof 0
dm nnn
dmm c
dpwr 37
dmf 35088
SPECIAL
temp 27.0
gain 20
spin 20
hst 0.008
pw90 11.600
atfa 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 1201.9
rfp 0
rp 45.7
lp 13.0
PLOT
wc 250
sc 0
vs 319
th 12
al cdc ph
MeO
H
N
O
H
H
N
N
Me
O
OH
H
Me
16{2,9}
9
8
7
6
5
4
3
2
1
0 ppm


600 MHz nmrox
BAG-01-206-2
exp4 Carbon
SAMPLE
date Aug 28 2009
solvent cdcl3
file exp
ACQUISITION
sw 40322.6
at 2.000
np 161290
fb 17000
bs 64
d1 2.000
nt 20000
ct 20000
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 3565.5
rfp 0
rp 47.5
lp -18.4
PLOT
wc 250
sc 0
vs 173630
th 68
ai cdc ph
MeO
H
H
H
H
N
N
N
O
O
Me
Me
OH
180
160
140
120
100
80
60
40
20
ppm

16{2,9}

Data File L:\09-11\GRANGER\160911-SFM00471-13367.D
Sample Name: SFM0047

```
    ======================================================================
    Acq. Operator   : brettag35@mail.utexas.edu
    Acq. Instrument : LCMS                          Location : Vial 48
    Injection Date  : 9/17/2011 1:02:48 AM
                                                    Inj Volume : 1.0 µl
    Acq. Method     : C:\CHEM32\1\METHODS\SP NIH.M
    Last changed    : 9/17/2011 1:02:33 AM by brettag35@mail.utexas.edu
                      (modified after loading)
    Analysis Method : C:\CHEM32\1\METHODS\DEF_LC.M
    Last changed    : 11/20/2006 4:14:44 AM
    Sample Info     : Easy-Access Method: 'SP NIH'
```



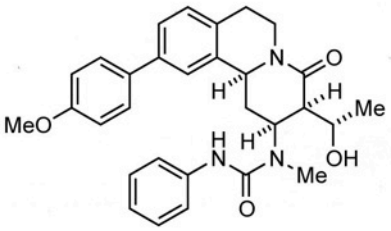

```
    ======================================================================
                              Area Percent Report
    ======================================================================

    Sorted By             :      Signal
    Multiplier:                   :      1.0000
    Dilution:                     :      1.0000
    Use Multiplier & Dilution Factor with ISTDs


    Signal 1: DAD1 B, Sig=214,8 Ref=360,100

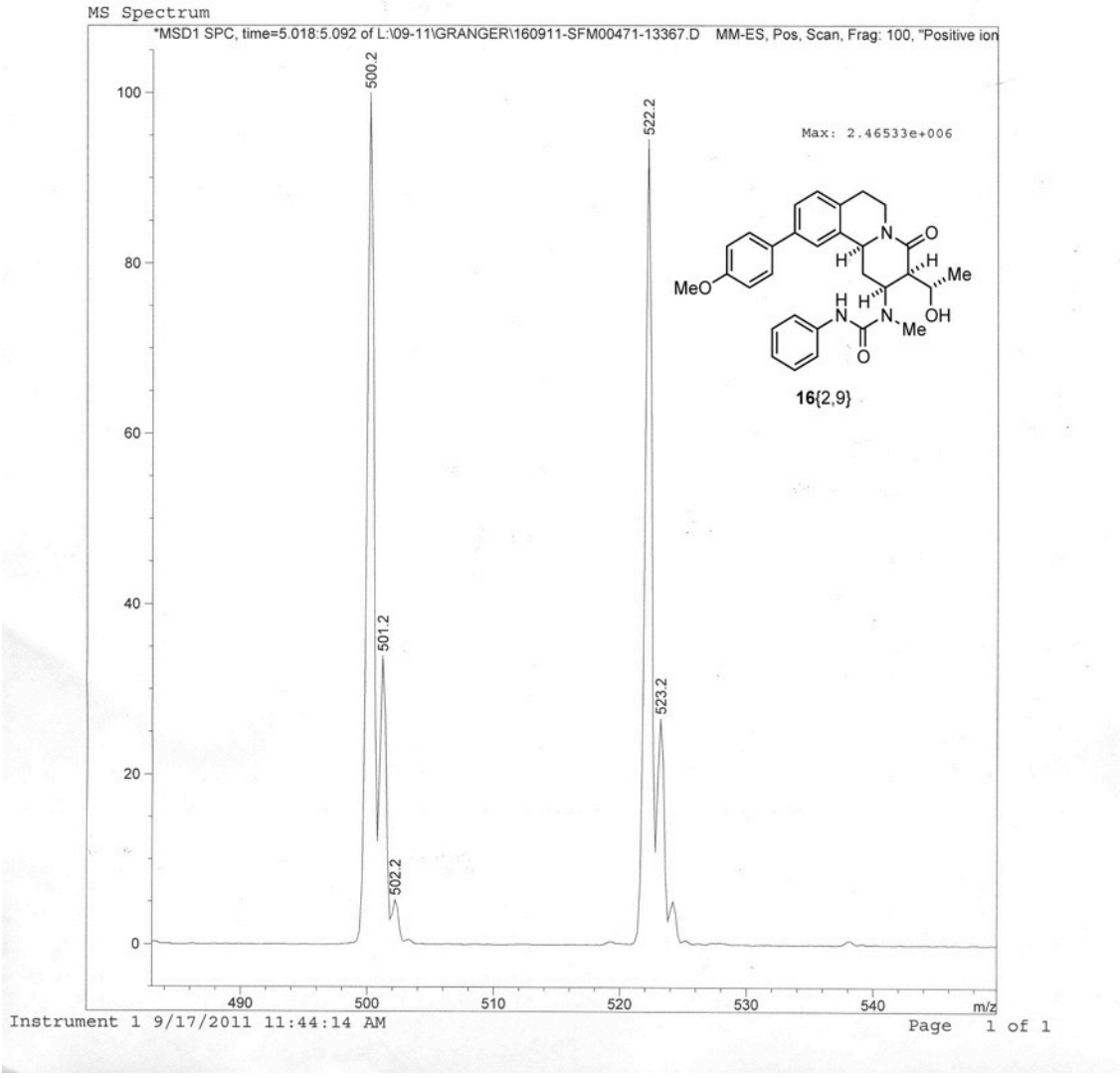
    Peak RetTime Type  Width     Area      Height     Area
      #   [min]        [min]   [mAU*s]     [mAU]       %
    ----|-------|----|-------|----------|----------|--------|
       1   3.261 MM    0.1077   38.17173    5.90643   1.5668
       2   5.060 MM    0.1138 2257.80518  330.67264  92.6752
       3   5.337 MM    0.1083   74.96353   10.07144   3.0770
       4   5.767 MM    0.2310   65.31505    4.71266   2.6810

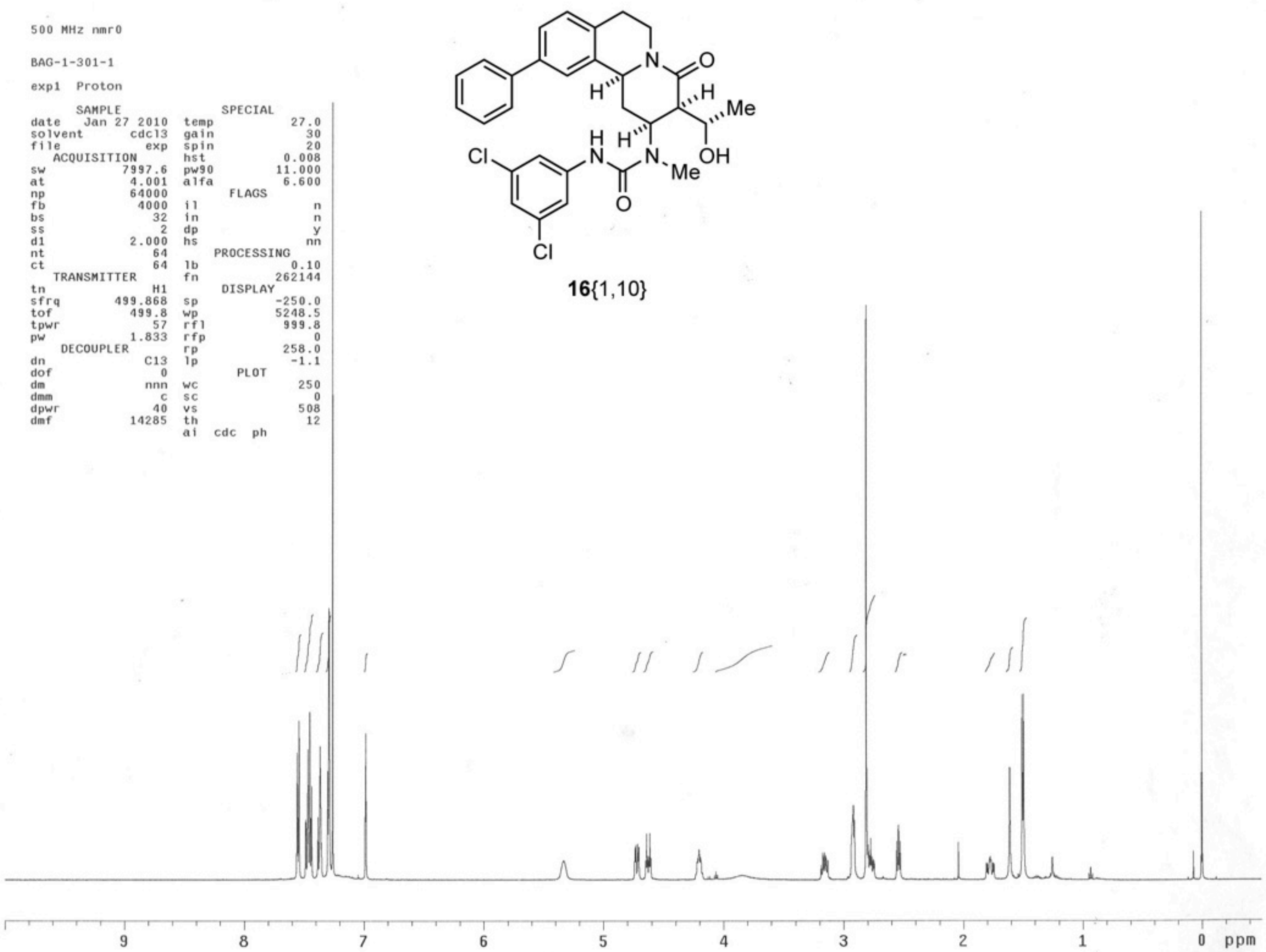
    Totals :                   2436.25548  351.36318
```



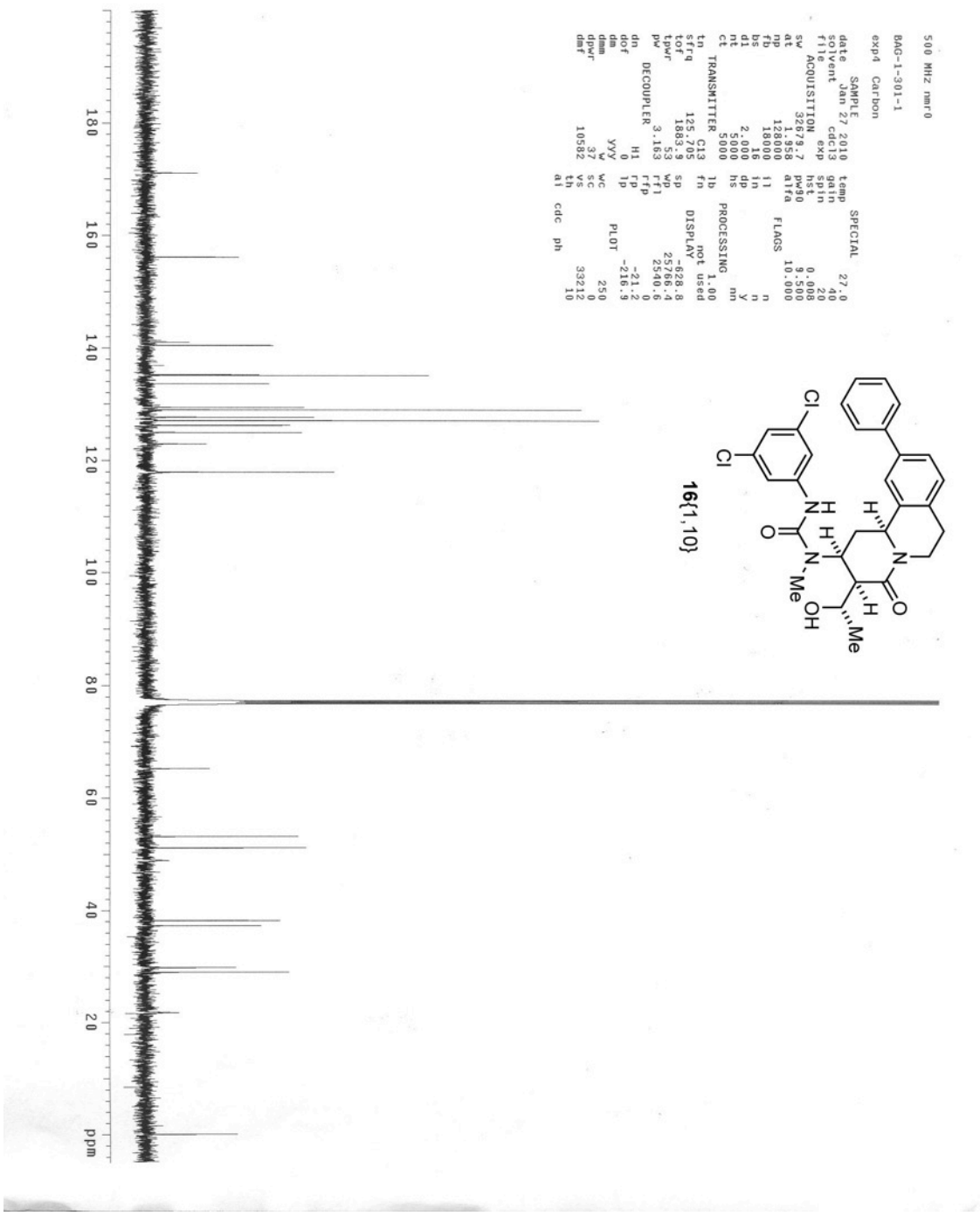
16{2,9}

```
Print of window 79: MS Spectrum
Data File  : L:\09-11\GRANGER\160911-SFM00471-13367.D
Sample Name : SFM0047
======================================================================
Acq. Operator   : brettag35@mail.utexas.edu
Acq. Instrument : LCMS                          Location : Vial 48
Injection Date  : 9/17/2011 1:02:48 AM               Inj :   1
                                                Inj Volume : 1.0 µl
Acq. Method     : C:\CHEM32\1\METHODS\SP NIH.M
Last changed    : 9/17/2011 1:02:33 AM by brettag35@mail.utexas.edu
                  (modified after loading)
Analysis Method : C:\CHEM32\1\METHODS\DEF_LC.M
Last changed    : 11/20/2006 4:14:44 AM
Sample Info     : Easy-Access Method: 'SP NIH'
```



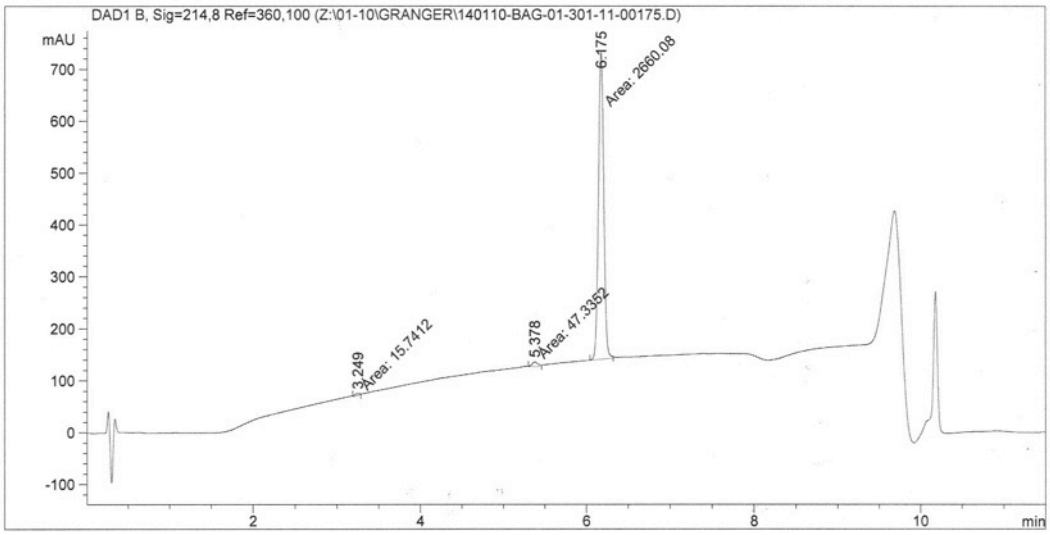

500 MHz nmr0
BAG-1-301-1
exp1 Proton
SAMPLE
date Jan 27 2010
solvent cdcl3
file exp
ACQUISITION
sw 7997.6
at 4.001
np 64000
fb 4000
bs 32
ss 2
d1 2.000
nt 64
ct 64
TRANSMITTER
tn H1
sfrq 499.868
tof 499.8
tpwr 57
pw 1.833
DECOUPLER
dn C13
dof 0
dm nnn
dmm c
dpwr 40
dmf 14285
SPECIAL
temp 27.0
gain 30
spin 20
hst 0.008
pw90 11.000
alfa 6.600
FLAGS
il n
in n
dp y
hs nn
PROCESSING
lb 0.10
fn 262144
DISPLAY
sp -250.0
wp 5248.5
rfl 999.8
rfp 0
rp 258.0
lp -1.1
PLOT
wc 250
sc 0
vs 508
th 12
ai cdc ph
Cl
Cl
H
N
O
N
Me
OH
Me
H
H
H
O
N
9
8
7
6
5
4
3
2
1
0 ppm

16{1,10}


500 MHz nmr0
BAG-1-301-1
exp4 Carbon
SAMPLE
date Jan 27 2010
solvent cdcl3
file exp
ACQUISITION
sw 32679.7
at 1.958
np 128000
fb 18000
bs 16
d1 2.000
nt 5000
ct 5000
TRANSMITTER
tn C13
sfrq 125.705
tof 1883.9
tpwr 53
pw 3.163
DECOUPLER
dn H1
dof 0
dm yyy
dmm w
dmf 37
dpwr 10582
SPECIAL
temp 27.0
gain 40
spin 20
hst 0.008
pw90 9.500
atfa FLAGS 10.000
n
n
y
nn
PROCESSING
lb 1.00
fn not used
DISPLAY
sp -628.8
wp 25766.4
rfl 2540.6
rfp 0
rp -21.2
lp -216.9
PLOT
250
0
wc 33212
sc 10
vs
th
cdc ph
al
Cl
Cl
N
H
O
N
Me
OH
H
H
Me
O
16{1,10}
180
160
140
120
100
80
60
40
20
ppm

Data File Z:\01-10\GRANGER\140110-BAG-01-301-11-00175.D
Sample Name: BAG-01-301-1

```
    ======================================================================
    Acq. Operator    : brettag35@gmail.com
    Acq. Instrument : LCMS                              Location : Vial 18
    Injection Date   : 1/14/2010 2:52:54 PM
                                                      Inj Volume : 1.0 µl
    Acq. Method      : C:\CHEM32\1\METHODS\SP NIH.M
    Last changed     : 1/14/2010 2:52:43 PM by brettag35@gmail.com
                       (modified after loading)
    Analysis Method : C:\CHEM32\1\METHODS\DEF_LC.M
    Last changed     : 11/20/2006 4:14:44 AM
    Sample Info      : Easy-Access Method: 'SP NIH'
```



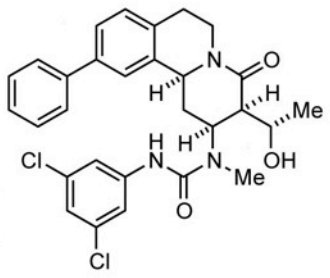

```
    ======================================================================
                              Area Percent Report
    ======================================================================

    Sorted By             :      Signal
    Multiplier:                   :      1.0000
    Dilution:                     :      1.0000
    Use Multiplier & Dilution Factor with ISTDs


    Signal 1: DAD1 B, Sig=214,8 Ref=360,100

    Peak RetTime Type  Width     Area      Height     Area
      #   [min]        [min]   [mAU*s]     [mAU]       %
    ----|-------|----|-------|----------|----------|--------|
       1   3.249 MM    0.0656   15.74118    4.00183   0.5780
       2   5.378 MM    0.0882   47.33524    8.94904   1.7382
       3   6.175 MM    0.0745 2660.08301  594.81671  97.6837

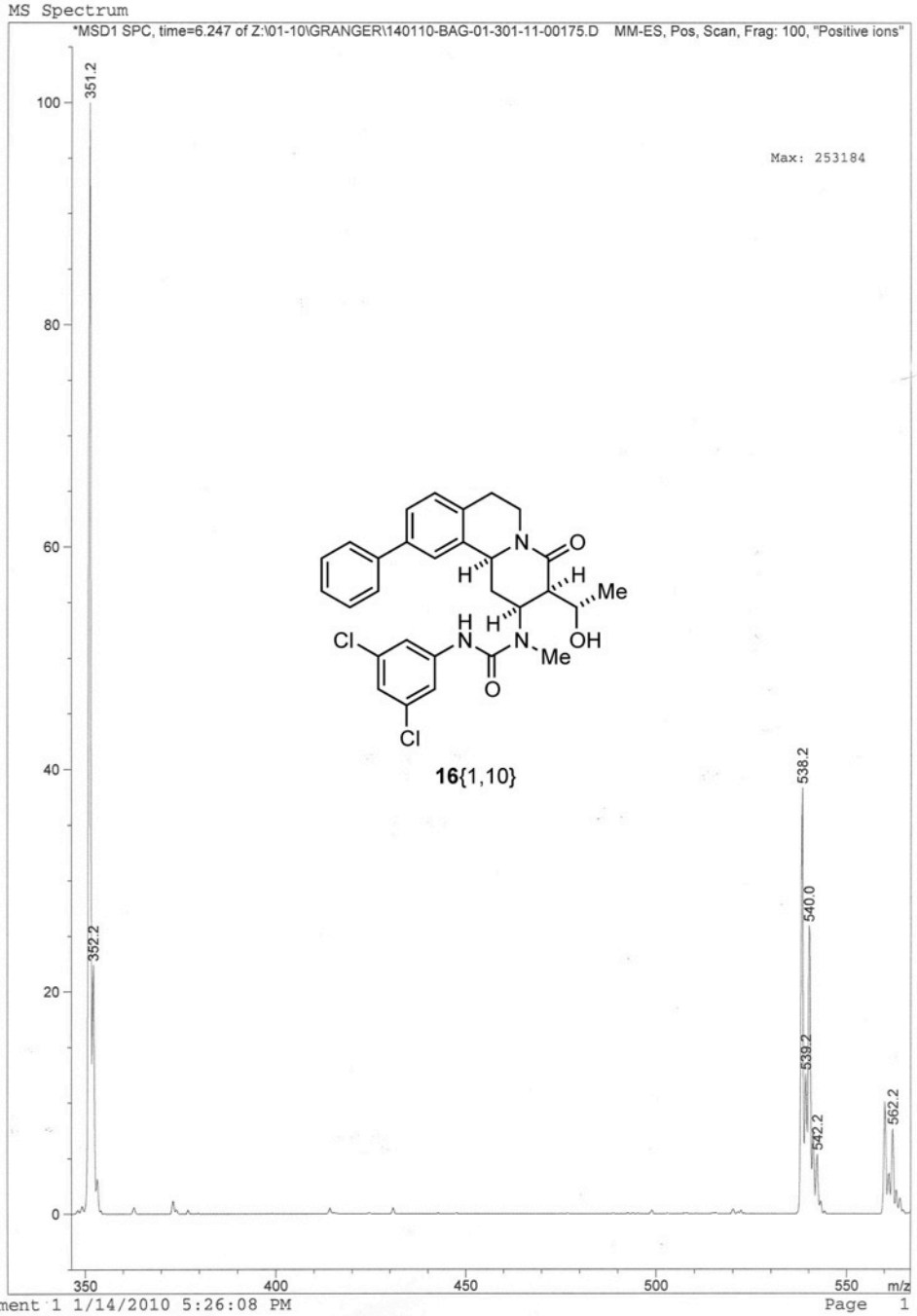
    Totals :                   2723.15943  607.76759
```



16{1,10}

Print of window 79: MS Spectrum

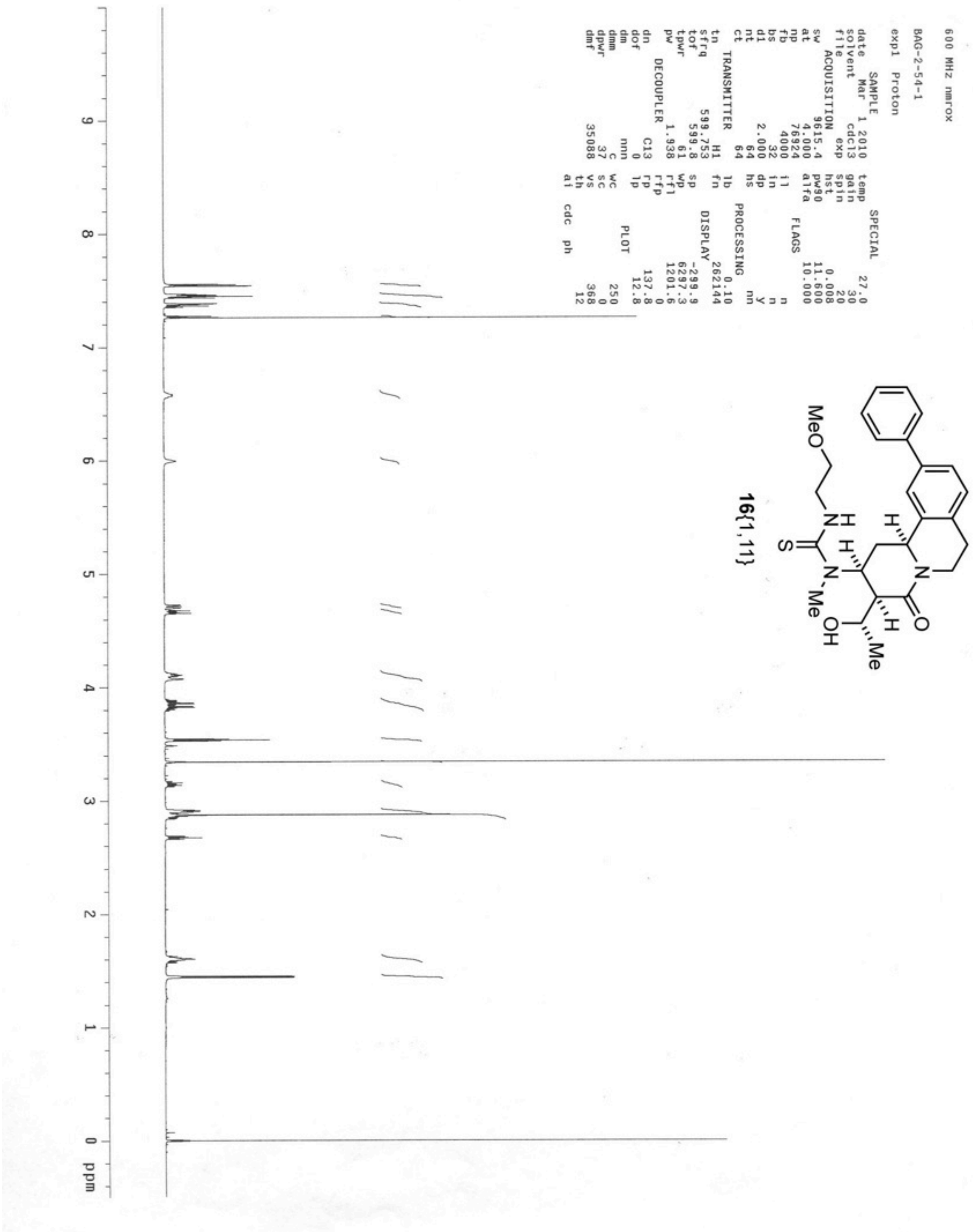
MS Spectrum

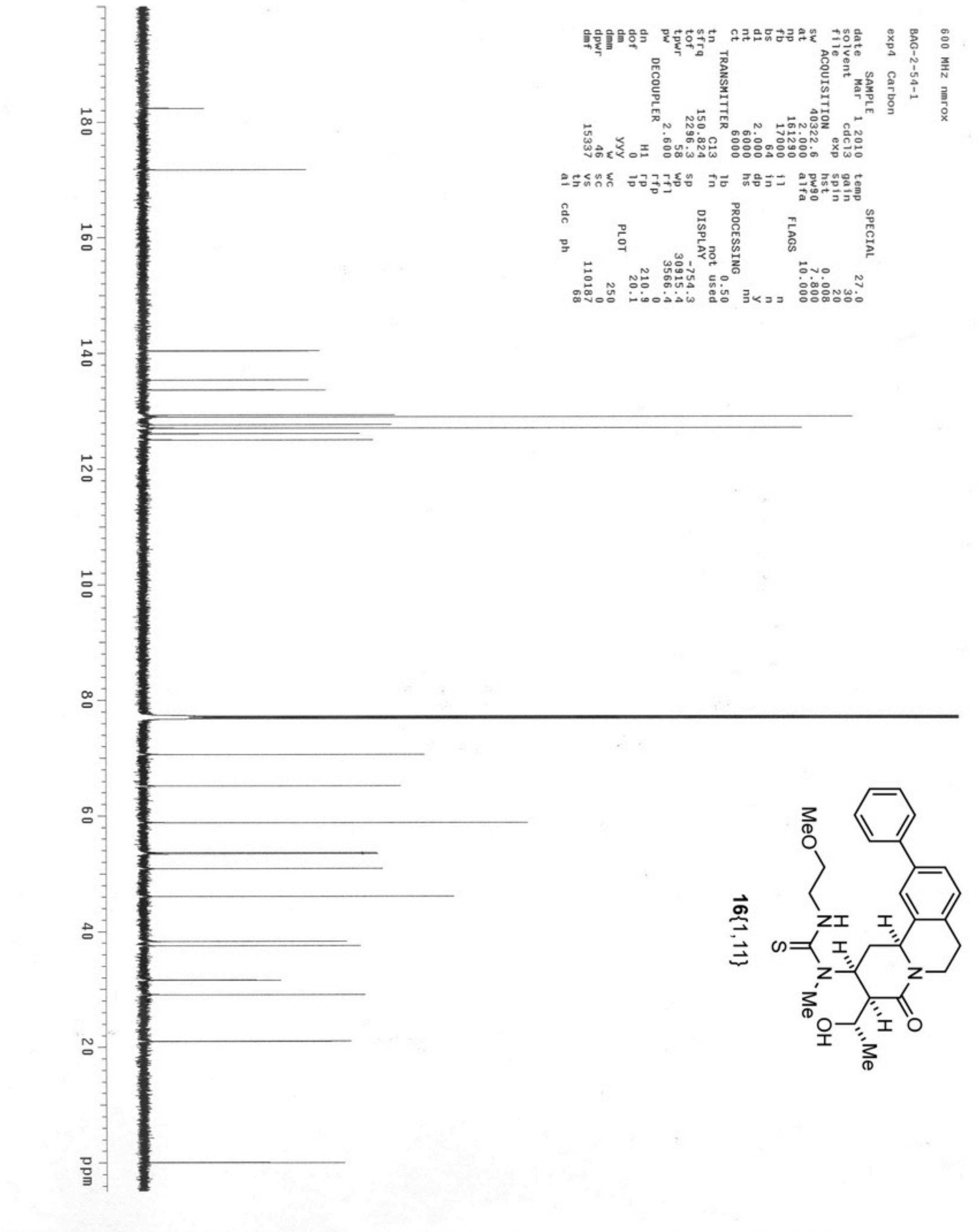


16{1,10}

Instrument 1 1/14/2010 5:26:08 PM

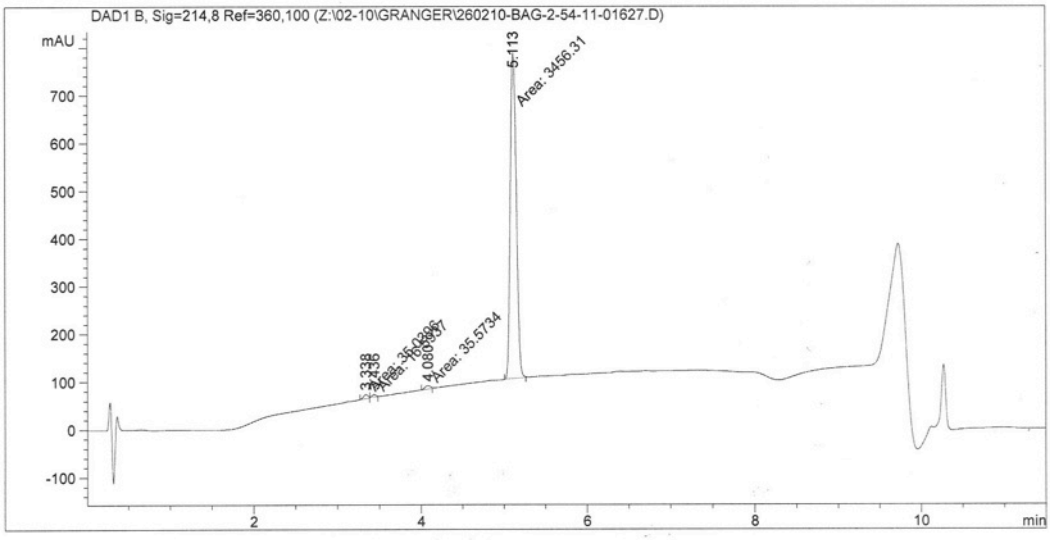
Page 1 of 1


600 MHz nmrox
BAG-2-54-1
exp1 Proton
16{1,11}
ppm


600 MHz nmrox
BAG-2-54-1
exp4 Carbon
180
160
140
120
100
80
60
40
20
ppm
MeO
16{1,11}

Data File Z:\02-10\GRANGER\260210-BAG-2-54-11-01627.D
Sample Name: BAG-2-54-1

```
Acq. Operator    : brettag35@mail.utexas.edu
Acq. Instrument : LCMS                          Location : Vial 57
Injection Date  : 2/26/2010 9:35:17 PM
                                                Inj Volume : 1.0 µl
Acq. Method     : C:\CHEM32\1\METHODS\SP NIH.M
Last changed    : 2/26/2010 9:35:06 PM by brettag35@mail.utexas.edu
                  (modified after loading)
Analysis Method : C:\CHEM32\1\METHODS\DEF_LC.M
Last changed    : 2/25/2010 3:55:13 PM
                  (modified after loading)
Sample Info     : Easy-Access Method: 'SP NIH'
```

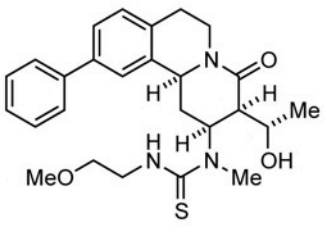



```
                          Area Percent Report

Sorted By             :      Signal
Multiplier:                   :      1.0000
Dilution:                     :      1.0000
Use Multiplier & Dilution Factor with ISTDs

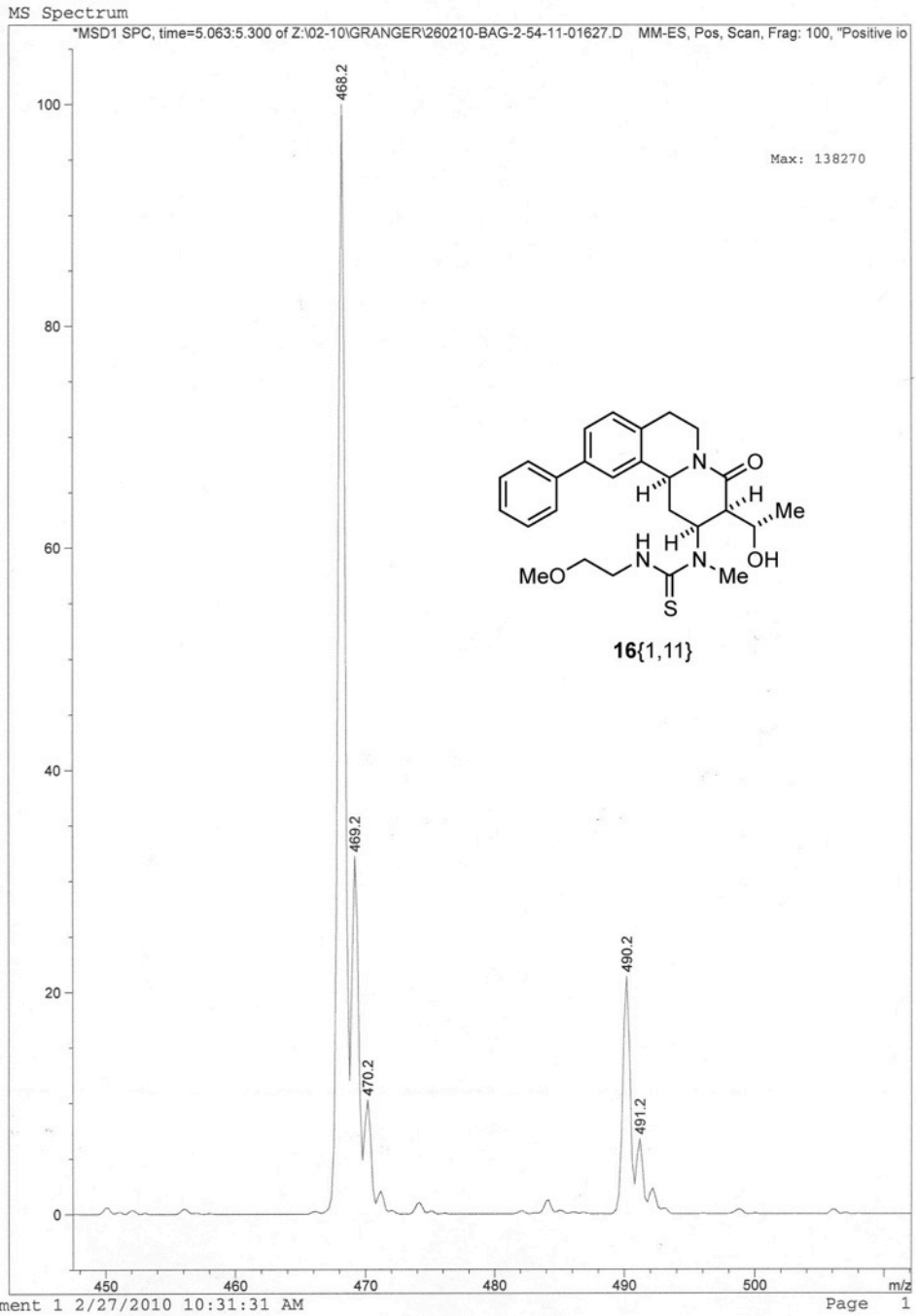

Signal 1: DAD1 B, Sig=214,8 Ref=360,100

Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]   [mAU*s]     [mAU]       %
----|-------|----|-------|----------|----------|--------|
   1   3.338 MM    0.0629   35.03965    9.28029   0.9888
   2   3.436 MM    0.0513   16.59366    5.39154   0.4683
   3   4.080 MM    0.0796   35.57336    7.45282   1.0039
   4   5.113 MM    0.0841 3456.31445  684.99646  97.5390
```



16{1,11}

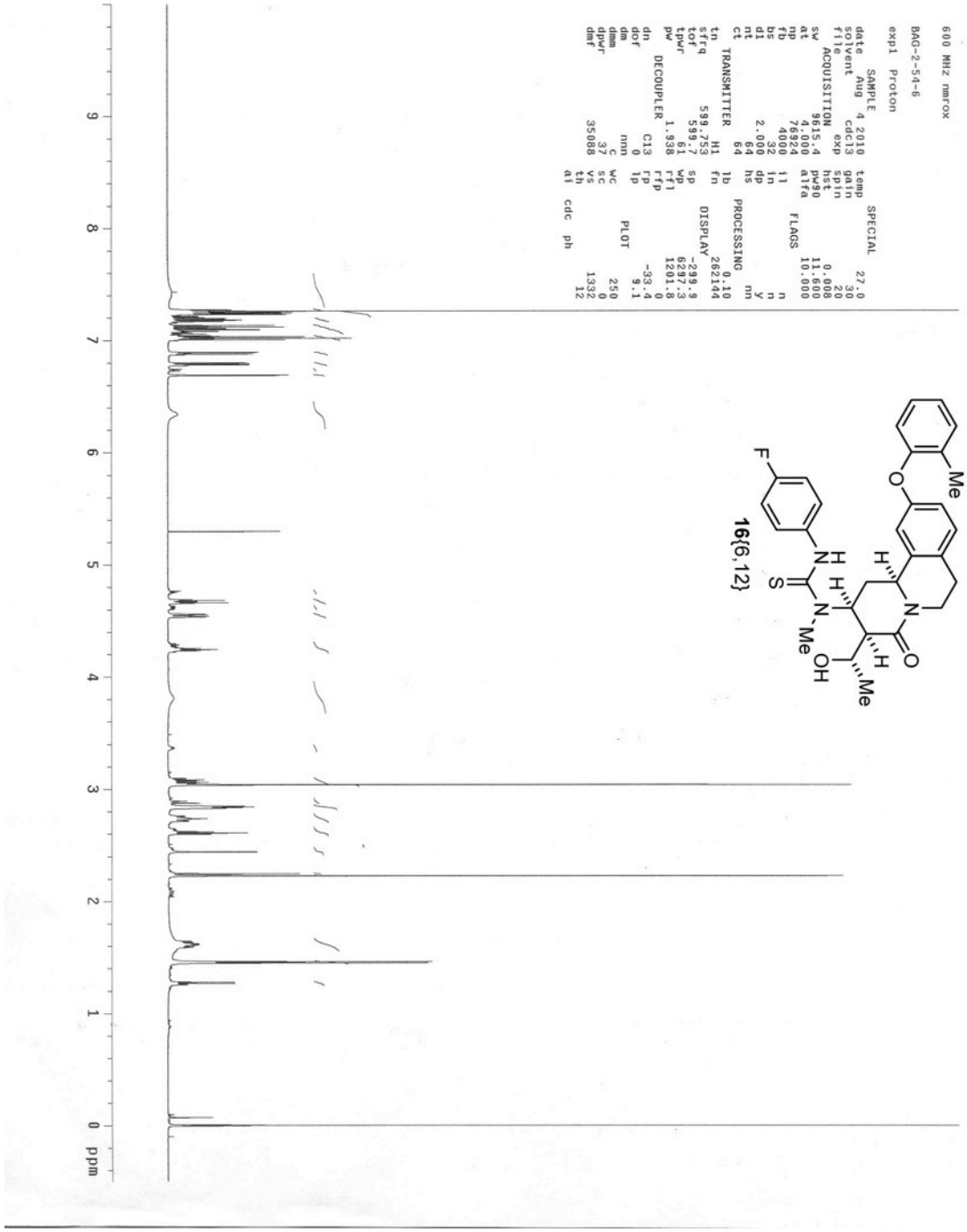
Print of window 79: MS Spectrum

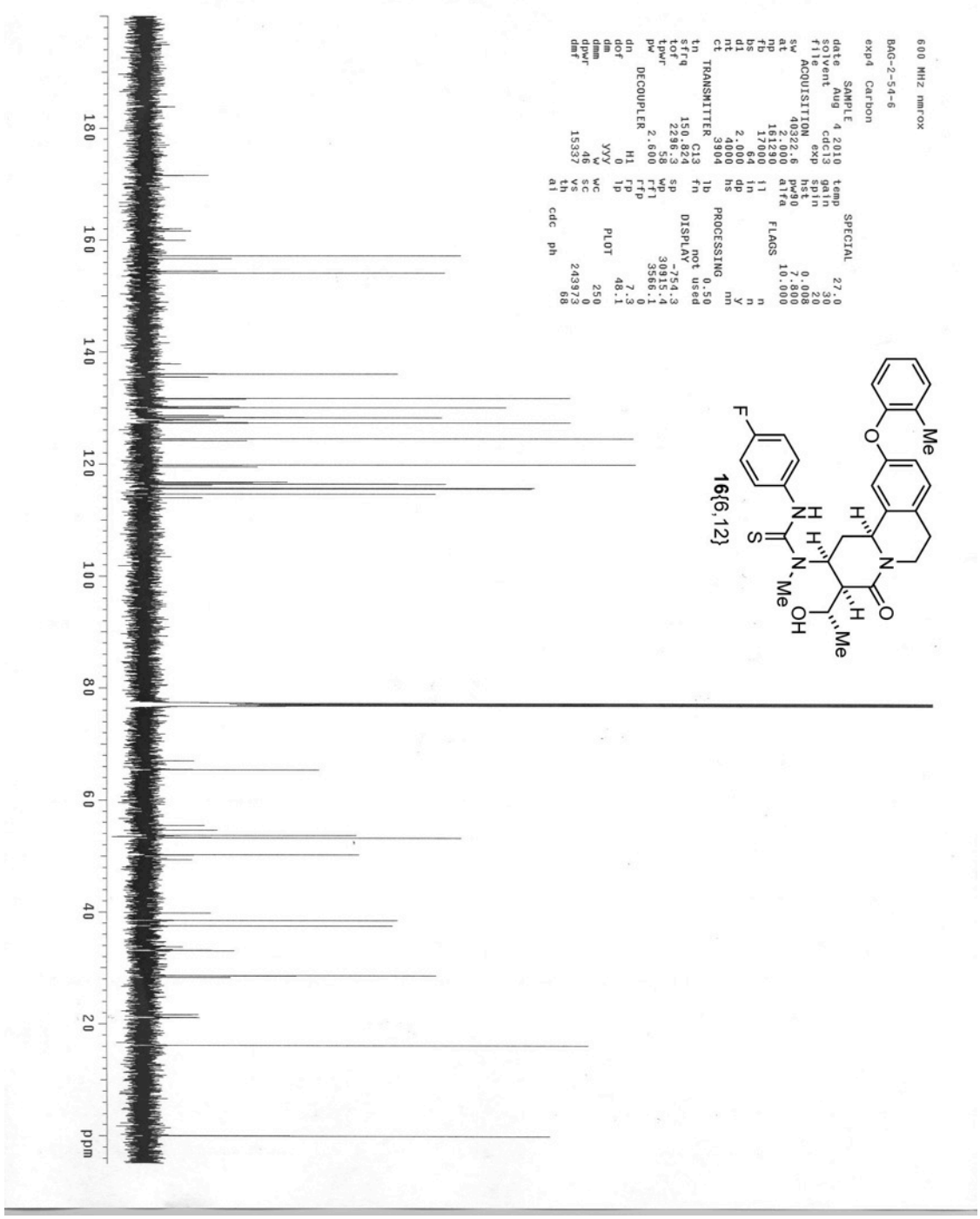


16{1,11}

Instrument 1 2/27/2010 10:31:31 AM

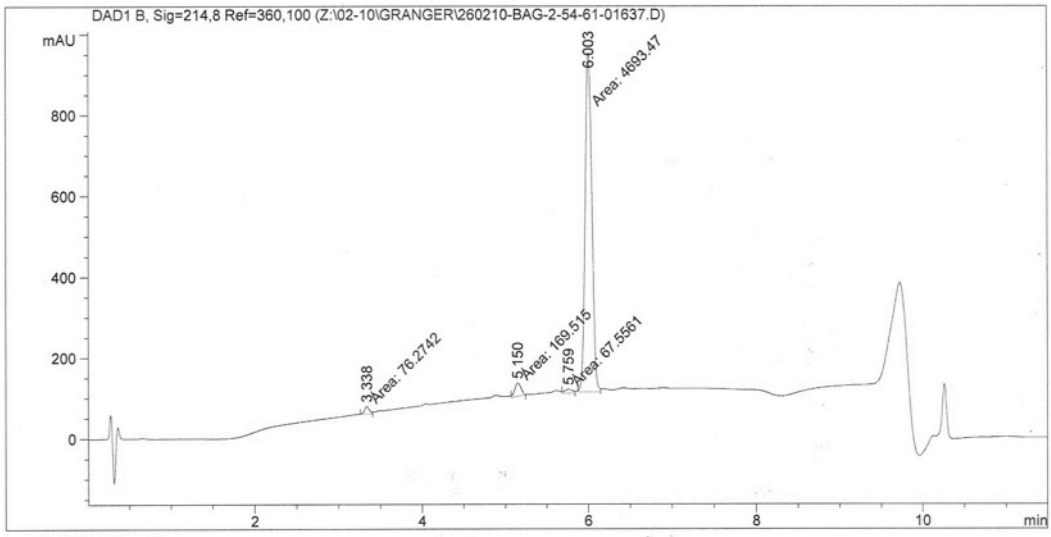
Page 1 of 1


600 MHz rmrox
BAG-2-54-6
exp1 Proton
SAMPLE
date Aug 4 2010
solvent
file
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
dof
dm
dmm
dmf
temp
gain
spin
hst
pw90
atfa
FLAGS
il
in
dp
hs
PROCESSING
lb 0.10
fn 262144
DISPLAY
sp -299.9
wp 6297.3
rfl 1201.8
rfp 0
rp -33.4
lp 9.1
PLOT
wc 250
sc 0
vs 1332
th 12
SPECIAL
27.0
30
0.008
11.600
10.000
n
n
y
nn
C13
0
nnn
37
35088
al cdc ph
16{6,12}
Me
F
O
H
N
S
N
Me
OH
H
Me
O
9
8
7
6
5
4
3
2
1
0 ppm


600 MHz nmrox
BAG-2-54-6
exp4 Carbon
SAMPLE
date Aug 4 2010
solvent cdcl3
file exp
ACQUISITION
sw 40322.6
at 2.000
np 161290
fb 17000
bs 64
d1 2.000
nt 4000
ct 3904
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
temp 27.0
gain 30
spin 20
hst 0.008
pw90 7.800
atfa 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
rf1 3566.1
rfp 0
rp 7.3
lp 48.1
PLOT
wc 250
sc 0
vs 243973
th 68
al cdc ph
Me
O
H
N
O
H
Me
OH
N
Me
H
N
S
F
16{6,12}
180
160
140
120
100
80
60
40
20
ppm

Data File Z:\02-10\GRANGER\260210-BAG-2-54-61-01637.D
Sample Name: BAG-2-54-6

```
==========================================================================
Acq. Operator    : brettag35@mail.utexas.edu
Acq. Instrument : LCMS                              Location : Vial 67
Injection Date  : 2/26/2010 11:45:29 PM
                                                   Inj Volume : 1.0 µl
Acq. Method      : C:\CHEM32\1\METHODS\SP NIH.M
Last changed     : 2/26/2010 11:45:17 PM by brettag35@mail.utexas.edu
                   (modified after loading)
Analysis Method : C:\CHEM32\1\METHODS\DEF_LC.M
Last changed     : 2/25/2010 3:55:13 PM
                   (modified after loading)
Sample Info      : Easy-Access Method: 'SP NIH'
```

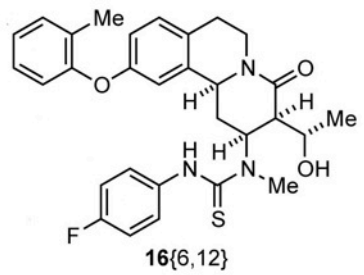



```
==========================================================================
                        Area Percent Report
==========================================================================

Sorted By             :      Signal
Multiplier:                   :      1.0000
Dilution:                     :      1.0000
Use Multiplier & Dilution Factor with ISTDs

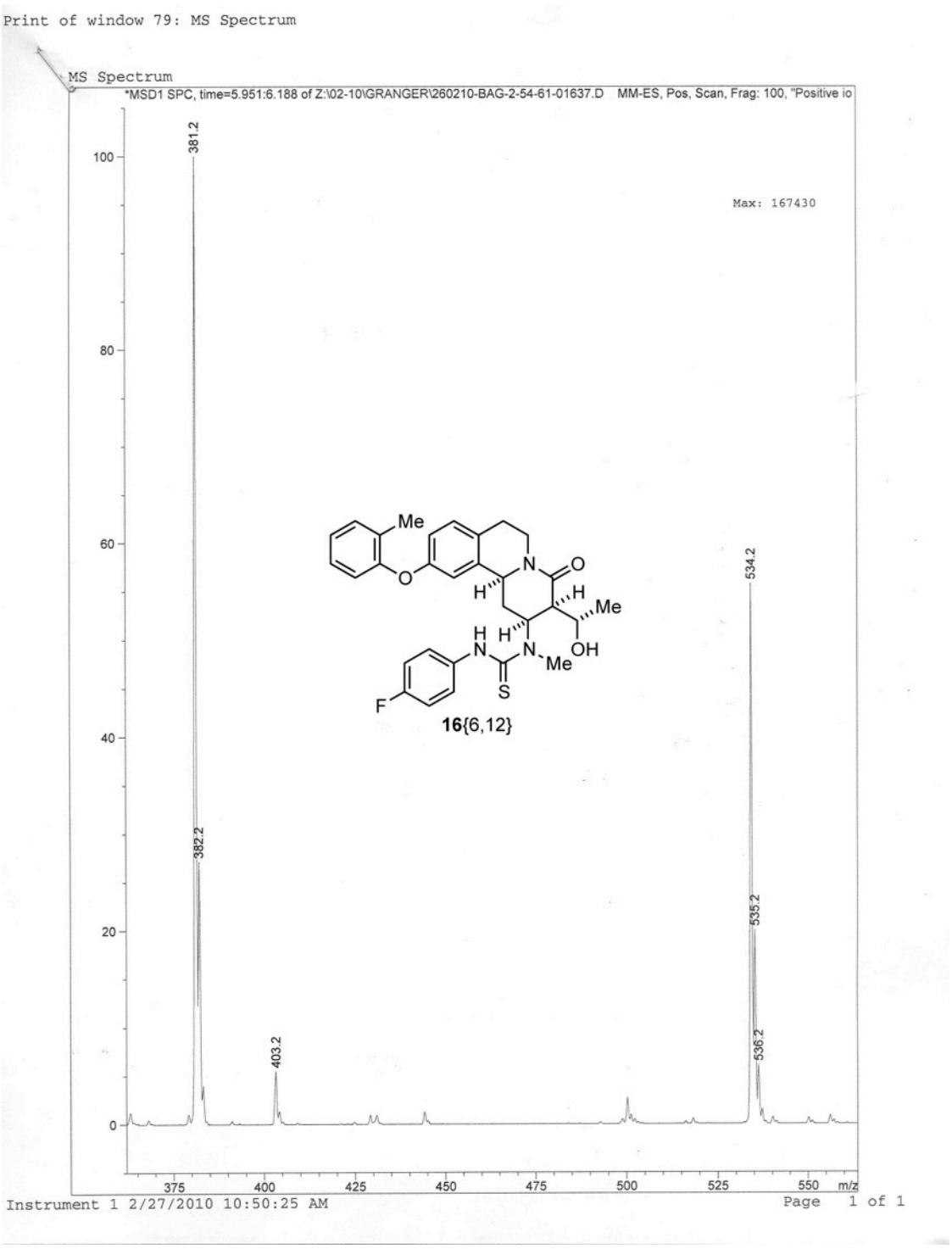

Signal 1: DAD1 B, Sig=214,8 Ref=360,100

Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]   [mAU*s]     [mAU]       %
----|-------|----|-------|----------|----------|--------|
   1   3.338 MM    0.0686   76.27418   18.52308   1.5234
   2   5.150 MM    0.0873  169.51480   32.37020   3.3857
   3   5.759 MM    0.1135   67.55612    9.92377   1.3493
   4   6.003 MM    0.0927 4693.47461  843.95105  93.7416
```

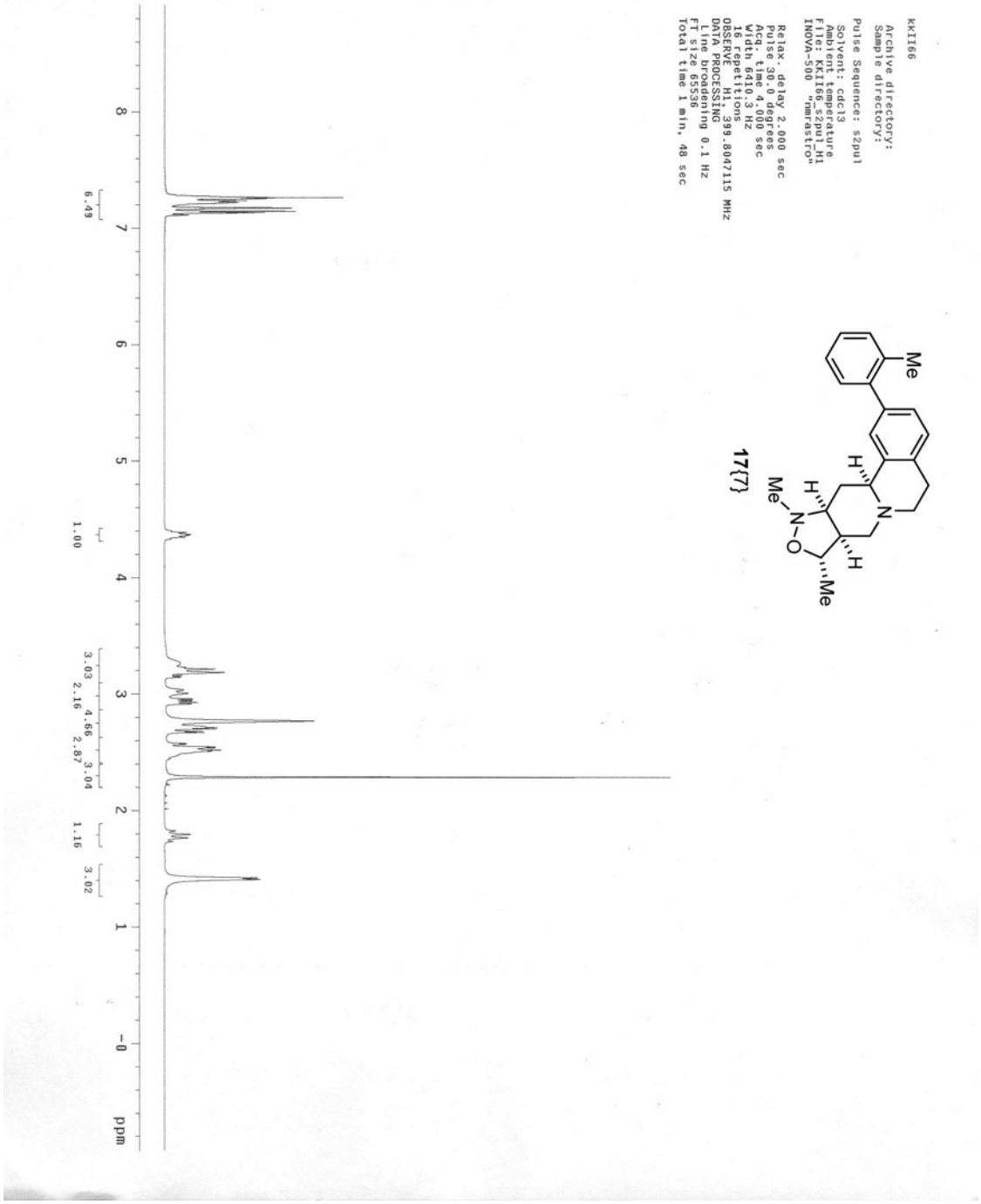


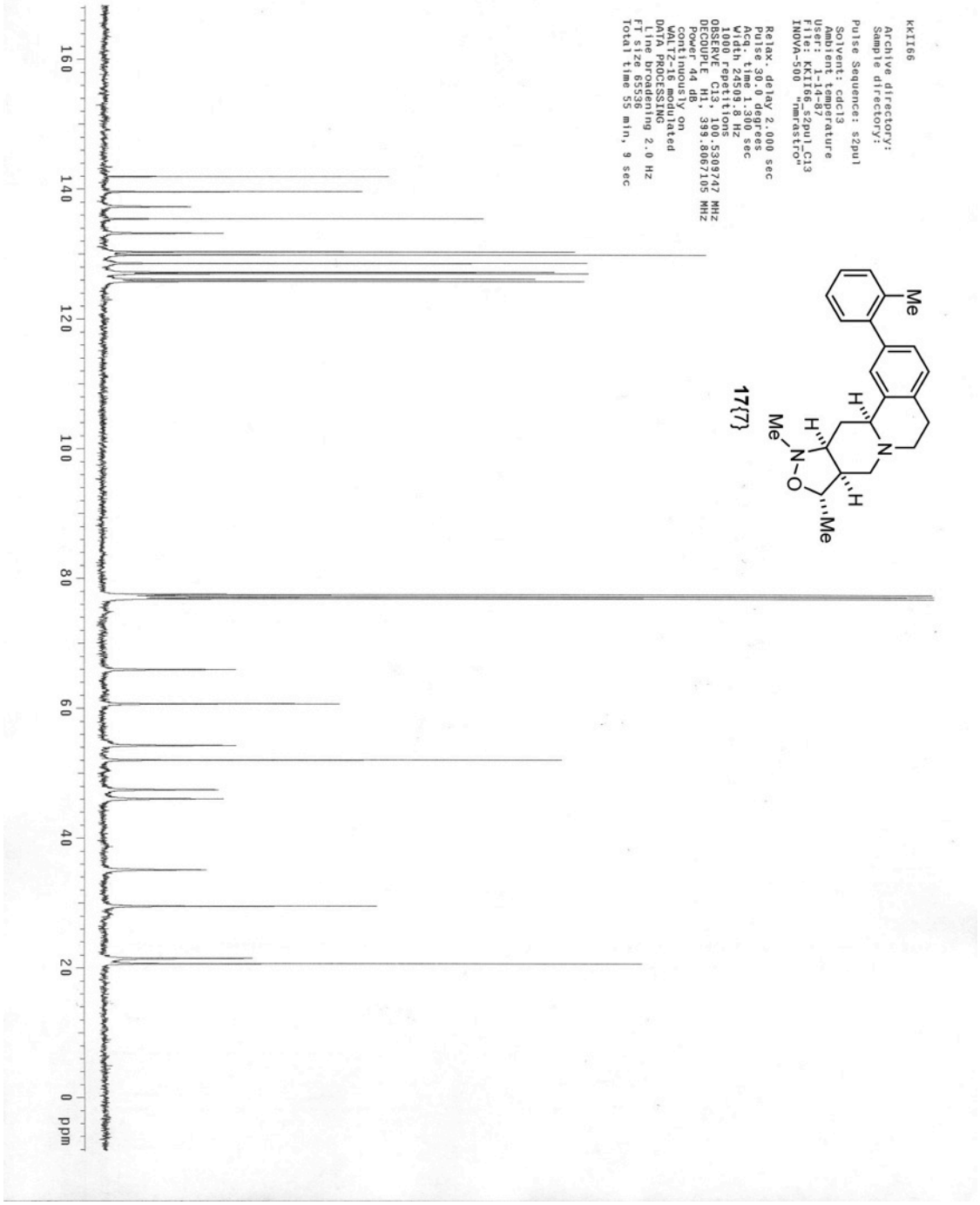
16{6,12}

Print of window 79: MS Spectrum



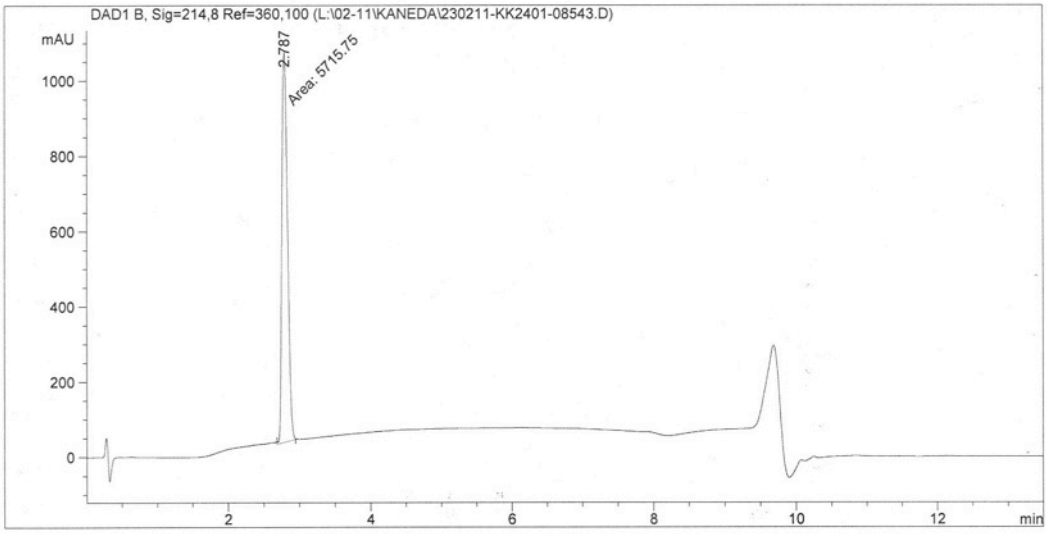
Instrument 1 2/27/2010 10:50:25 AM Page 1 of 1


kkI166
Archive directory:
Sample directory:
Pulse Sequence: s2pul
Solvent: cdcl3
Ambient temperature
File: kkI166_s2pul_H1
INOVA-500 "nmrastro"
Relax. delay 2.000 sec
Pulse 30.0 degrees
Acq. time 4.000 sec
Width 6410.3 Hz
16 repetitions
OBSERVE H1, 399.8047115 MHz
DATA PROCESSING
Line broadening 0.1 Hz
FT size 65536
Total time 1 min, 48 sec
Me
H
H
N
Me
N
O
H
Me
17{7}
8
7
6
5
4
3
2
1
-0
ppm
6.49
1.00
3.03
2.16
4.66
2.87
3.04
1.16
3.02


kkI166
Archive directory:
Sample directory:
Pulse Sequence: s2pul
Solvent: cdcl3
Ambient temperature
Usem: 1-14-07
File: KkI166_s2pul_C13
INOVA-500 "marastro"
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
WALTZ16 modulated
DATA PROCESSING
Line broadening 2.0 Hz
FT size 65536
Total time 55 min, 9 sec
Me
H
H
N
H
Me
N
O
Me
17{7}
160
140
120
100
80
60
40
20
0 ppm

Data File L:\02-11\KANEDA\230211-KK2401-08543.D
Sample Name: kk240

```
    ========================================================================
    Acq. Operator    : kyosuke.kaneda@cm.utexas.edu
    Acq. Instrument : LCMS                              Location : Vial 44
    Injection Date   : 2/23/2011 9:33:14 PM
                                                      Inj Volume : 1.0 µl
    Acq. Method      : C:\CHEM32\1\METHODS\SP NIH.M
    Last changed     : 2/23/2011 9:32:59 PM by kyosuke.kaneda@cm.utexas.edu
                       (modified after loading)
    Analysis Method : C:\CHEM32\1\METHODS\DEF_LC.M
    Last changed     : 11/20/2006 4:14:44 AM
    Sample Info      : Easy-Access Method: 'SP NIH'
```




```
    ========================================================================
                              Area Percent Report
    ========================================================================

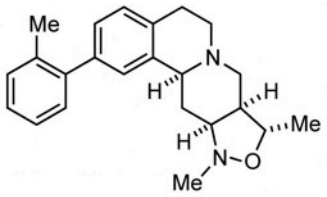
    Sorted By             :      Signal
    Multiplier:                   :      1.0000
    Dilution:                     :      1.0000
    Use Multiplier & Dilution Factor with ISTDs


    Signal 1: DAD1 B, Sig=214,8 Ref=360,100

    Peak RetTime Type  Width     Area      Height     Area
      #   [min]        [min]   [mAU*s]     [mAU]       %
    ----|-------|----|-------|----------|----------|--------|
       1   2.787 MM    0.0917 5715.75342 1039.32715 100.0000

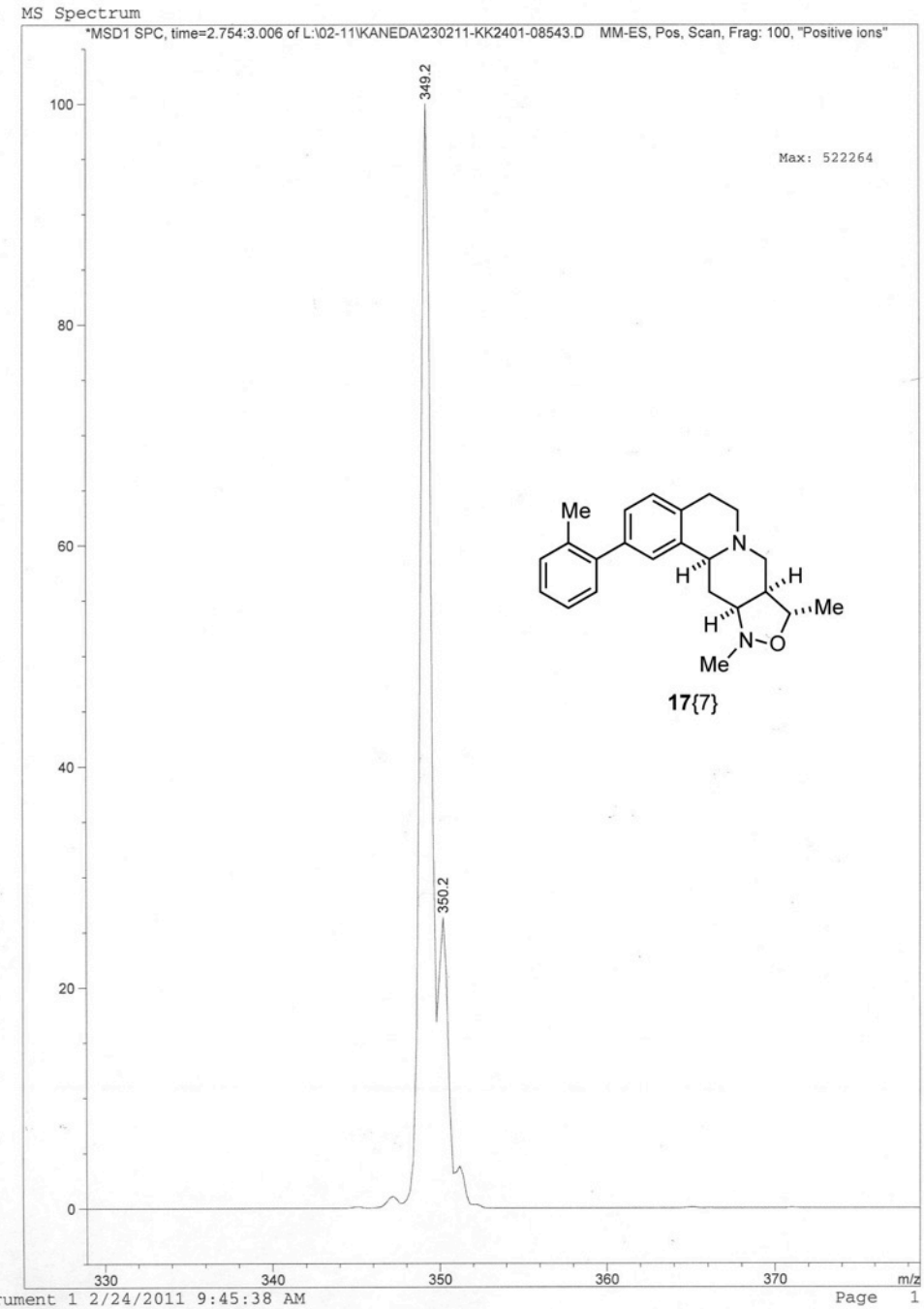
    Totals :                  5715.75342 1039.32715

    ========================================================================
```



17{7}

Print of window 79: MS Spectrum



17{7}

Instrument 1 2/24/2011 9:45:38 AM

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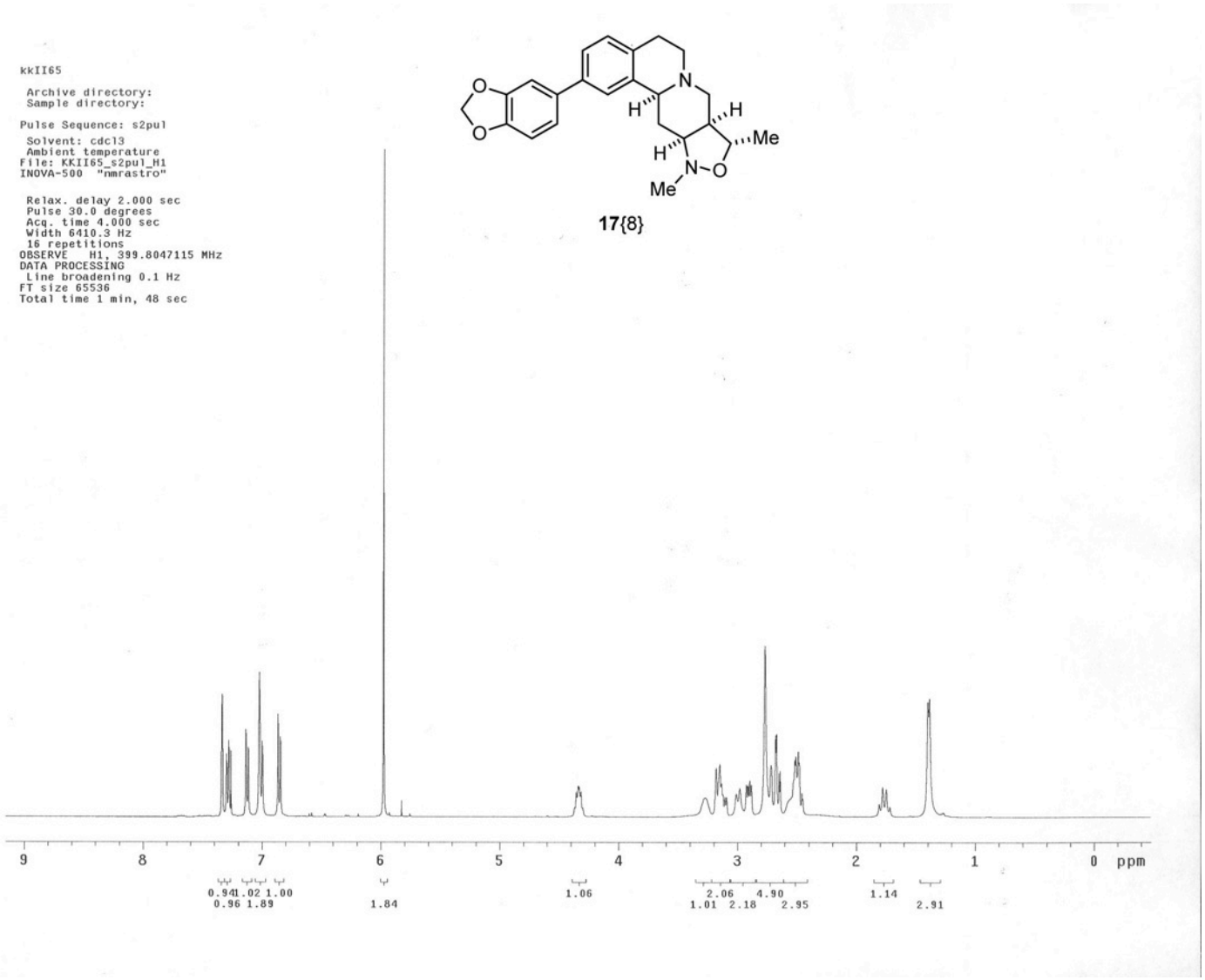
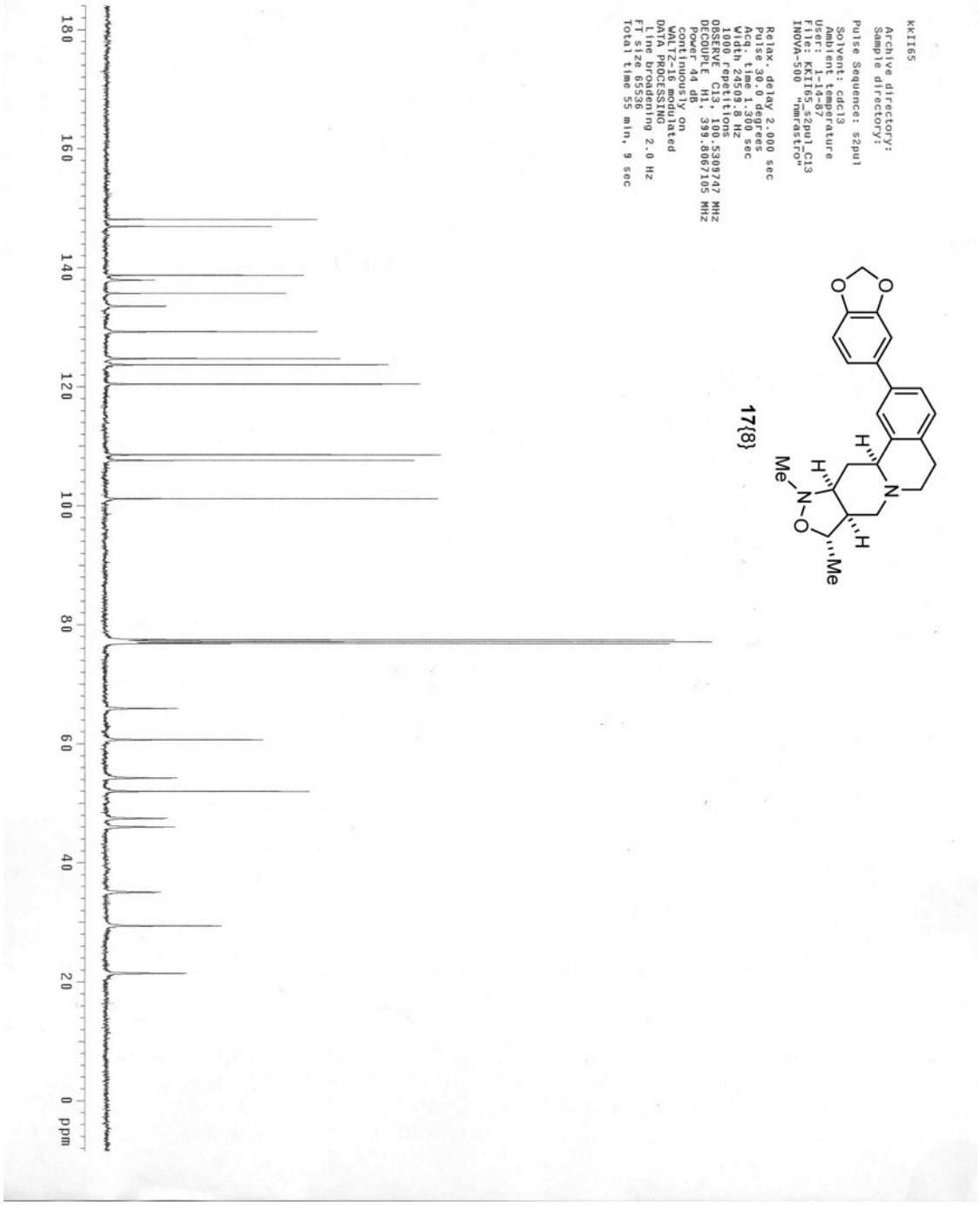
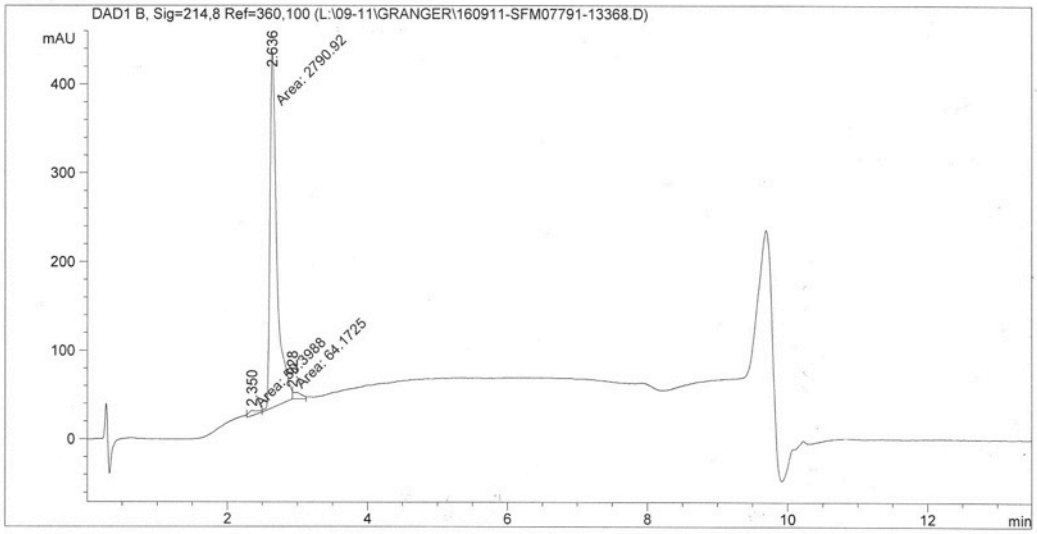

kkII65
Archive directory:
Sample directory:
Pulse Sequence: s2pul
Solvent: cdcl3
Ambient temperature
File: KKII65_s2pul_H1
INOVA-500 "nmrastro"
Relax. delay 2.000 sec
Pulse 30.0 degrees
Acq. time 4.000 sec
Width 6410.3 Hz
16 repetitions
OBSERVE H1, 399.8047115 MHz
DATA PROCESSING
Line broadening 0.1 Hz
FT size 65536
Total time 1 min, 48 sec
O
O
N
H
H
H
N
O
Me
Me
17{8}
9
8
7
6
5
4
3
2
1
0
ppm
0.94 1.02 1.00
0.96 1.89
1.84
1.06
2.06 4.90
1.01 2.18 2.95
1.14
2.91


kkI165
Archive directory:
Sample directory:
Pulse Sequence: s2pul
Solvent: cdcl3
Ambient temperature
User: 1-14-87
File: KKI165_s2pul_C13
INOVA-500 "nmrastro"
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
17{8}
Me
H
H
N
N
O
H
Me
180
160
140
120
100
80
60
40
20
0 ppm

Data File L:\09-11\GRANGER\160911-SFM07791-13368.D
Sample Name: SFM0779

```
    ======================================================================
    Acq. Operator   : brettag35@mail.utexas.edu
    Acq. Instrument : LCMS                          Location : Vial 49
    Injection Date  : 9/17/2011 1:17:48 AM
                                                    Inj Volume : 1.0 µl
    Acq. Method     : C:\CHEM32\1\METHODS\SP NIH.M
    Last changed    : 9/17/2011 1:17:33 AM by brettag35@mail.utexas.edu
                      (modified after loading)
    Analysis Method : C:\CHEM32\1\METHODS\DEF_LC.M
    Last changed    : 11/20/2006 4:14:44 AM
    Sample Info     : Easy-Access Method: 'SP NIH'
```



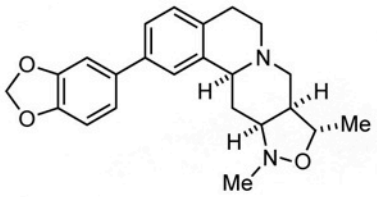

```
    ======================================================================
                              Area Percent Report
    ======================================================================

    Sorted By             :      Signal
    Multiplier:                   :      1.0000
    Dilution:                     :      1.0000
    Use Multiplier & Dilution Factor with ISTDs


    Signal 1: DAD1 B, Sig=214,8 Ref=360,100

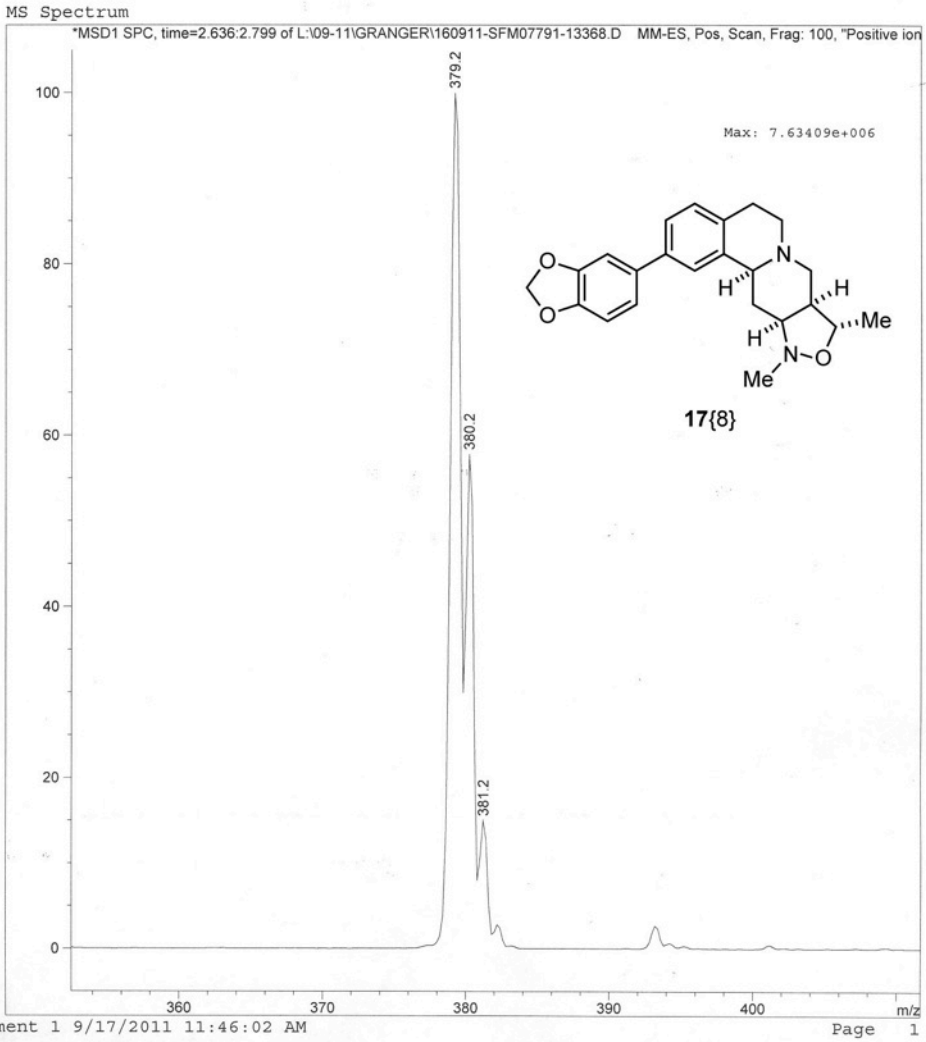
    Peak RetTime Type  Width     Area      Height     Area
      #   [min]        [min]   [mAU*s]     [mAU]      %
    ----|-------|----|-------|----------|----------|--------|
       1   2.350 MM    0.1429   50.39883    5.87624   1.7346
       2   2.636 MM    0.1153 2790.91650  403.48798  96.0567
       3   2.928 MM    0.1039   64.17245    8.46443   2.2087

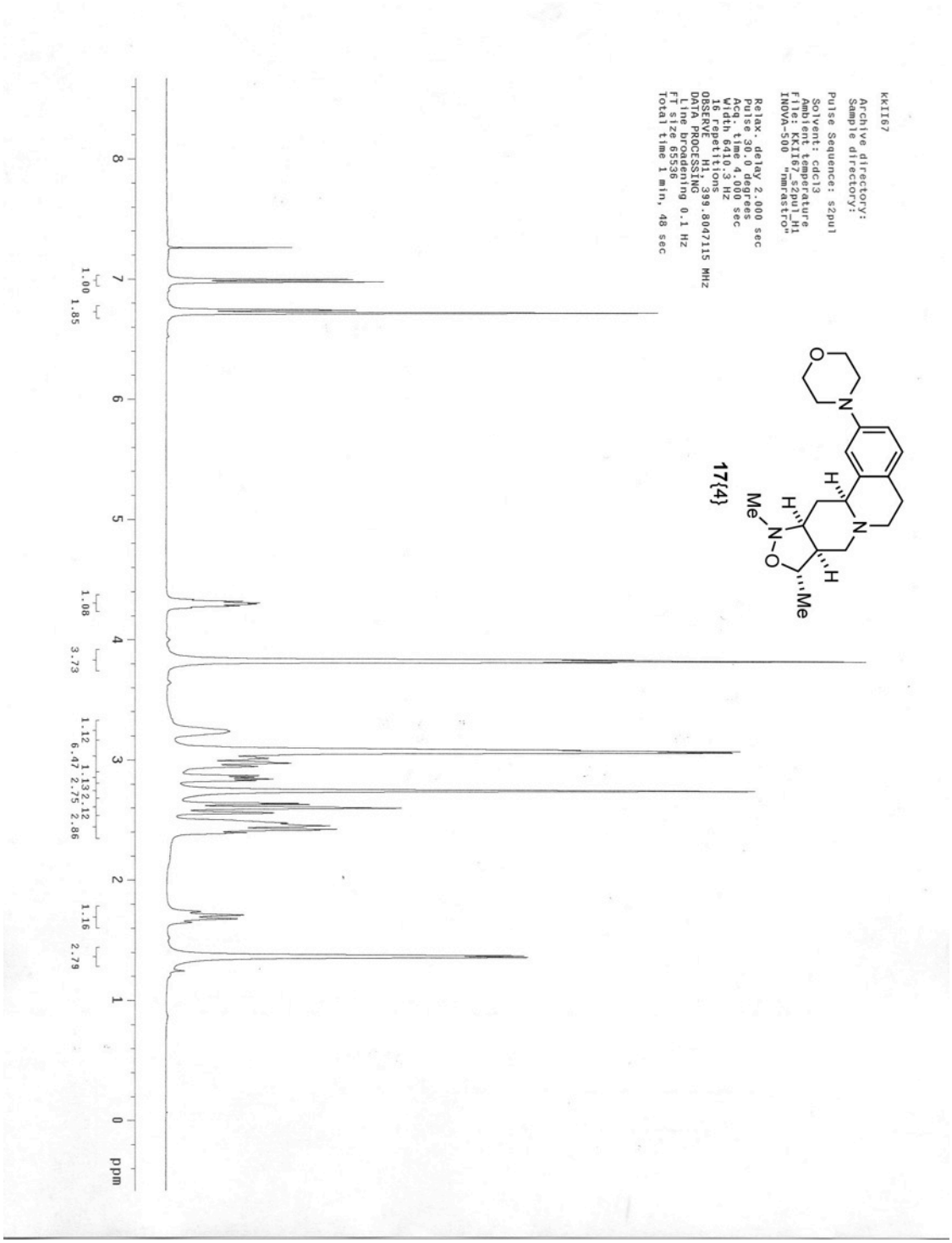
    Totals :                  2905.48779  417.82865
```

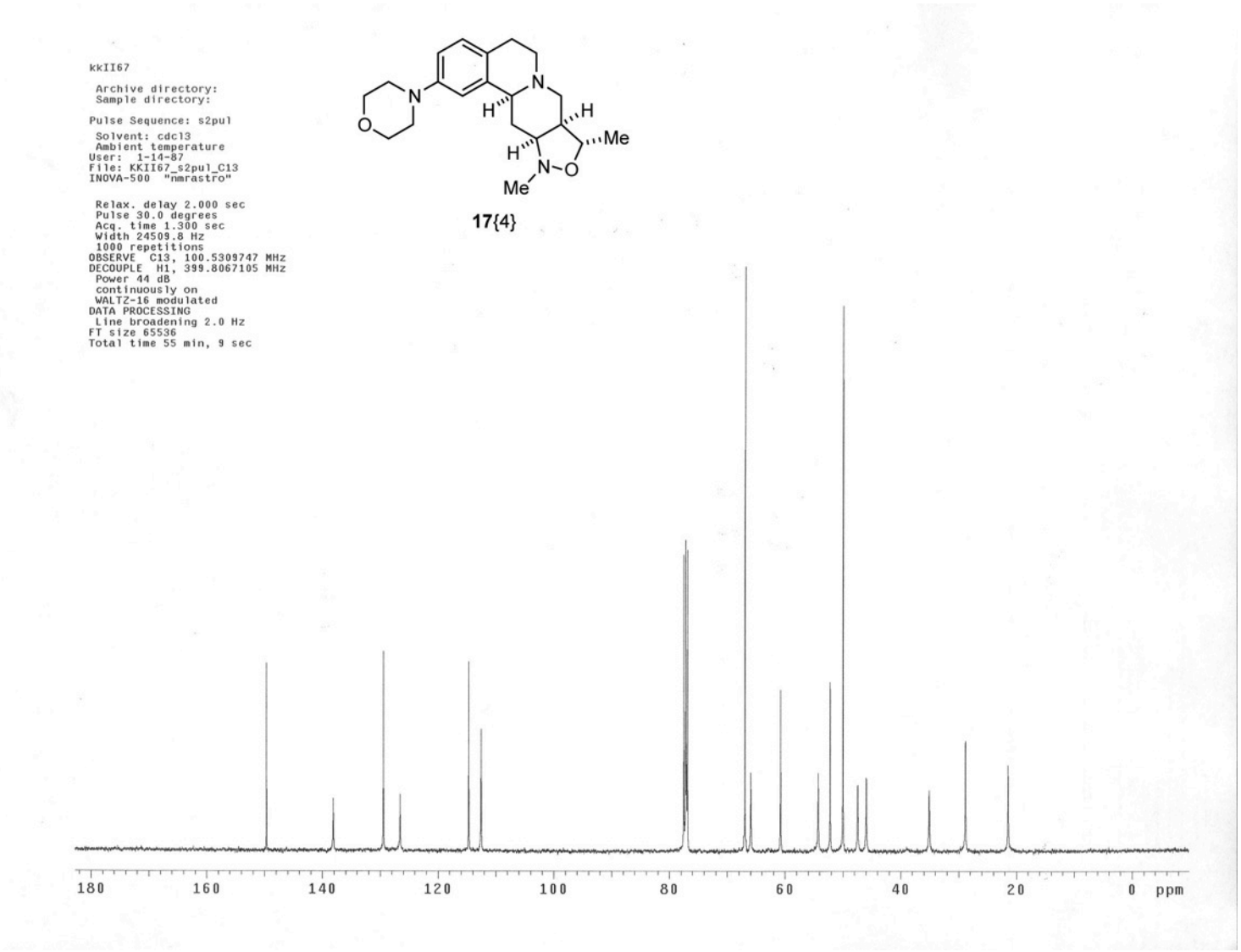


17{8}

```
Print of window 79: MS Spectrum
Data File  : L:\09-11\GRANGER\160911-SFM07791-13368.D
Sample Name : SFM0779
======================================================================
Acq. Operator   : brettag35@mail.utexas.edu
Acq. Instrument : LCMS                          Location : Vial 49
Injection Date  : 9/17/2011 1:17:48 AM               Inj :   1
                                                Inj Volume : 1.0 µl
Acq. Method     : C:\CHEM32\1\METHODS\SP NIH.M
Last changed    : 9/17/2011 1:17:33 AM by brettag35@mail.utexas.edu
                  (modified after loading)
Analysis Method : C:\CHEM32\1\METHODS\DEF_LC.M
Last changed    : 11/20/2006 4:14:44 AM
Sample Info     : Easy-Access Method: 'SP NIH'
```

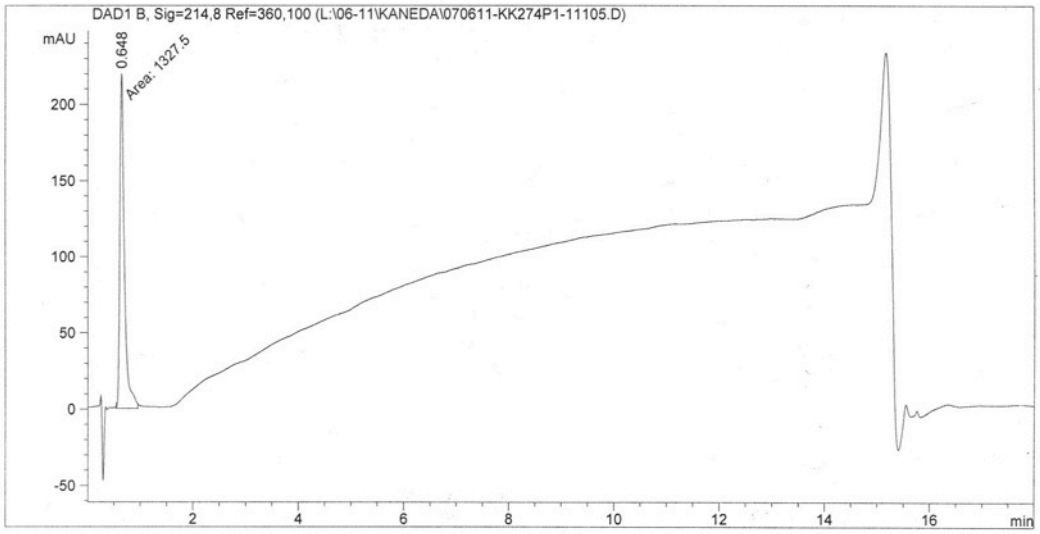



kkI167
Archive directory:
Sample directory:
Pulse Sequence: s2pul
Solvent: cdcl3
Ambient temperature
File: FKI167_s2pul_H1
INOVA-500 "marastro"
Relax. delay 2.000 sec
Pulse 30.0 degrees
Acq. time 4.000 sec
Width 6410.3 Hz
16 repetitions
OBSERVE H1, 399.8047115 MHz
DATA PROCESSING
Line broadening 0.1 Hz
FT size 65536
Total time 1 min, 48 sec
17{4}
Me
Me
H
H
H
O
N
N
O
N
8
7
6
5
4
3
2
1
0
ppm
1.00
1.85
1.08
3.73
1.12
6.47
1.13
2.75
2.12
2.86
1.16
2.79


kkII67
Archive directory:
Sample directory:
Pulse Sequence: s2pul
Solvent: cdcl3
Ambient temperature
User: 1-14-87
File: KKII67_s2pul_C13
INOVA-500 "nmrastro"
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
17{4}
180
160
140
120
100
80
60
40
20
0
ppm

Data File L:\06-11\KANEDA\070611-KK274P1-11105.D
Sample Name: kk274p

```
    ======================================================================
    Acq. Operator    : kyosuke.kaneda@cm.utexas.edu
    Acq. Instrument  : LCMS                              Location : Vial 28
    Injection Date   : 6/7/2011 10:16:47 AM
                                                      Inj Volume : 1.0 µl
    Acq. Method      : C:\CHEM32\1\METHODS\LCMS 12MIN GRADIENT.M
    Last changed     : 6/7/2011 10:16:26 AM by kyosuke.kaneda@cm.utexas.edu
                       (modified after loading)
    Analysis Method  : C:\CHEM32\1\METHODS\DEF_LC.M
    Last changed     : 11/20/2006 4:14:44 AM
    Sample Info      : Easy-Access Method: 'LCMS 12MIN GRADIENT'
```




```
    ======================================================================
                              Area Percent Report
    ======================================================================

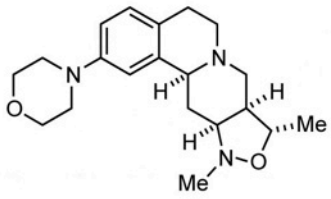
    Sorted By             :      Signal
    Multiplier:                   :      1.0000
    Dilution:                     :      1.0000
    Use Multiplier & Dilution Factor with ISTDs


    Signal 1: DAD1 B, Sig=214,8 Ref=360,100

    Peak RetTime Type  Width     Area      Height     Area
      #   [min]        [min]   [mAU*s]     [mAU]       %
    ----|-------|----|-------|----------|----------|--------|
       1   0.648 MM    0.1004 1327.50012  220.27107 100.0000

    Totals :                  1327.50012  220.27107

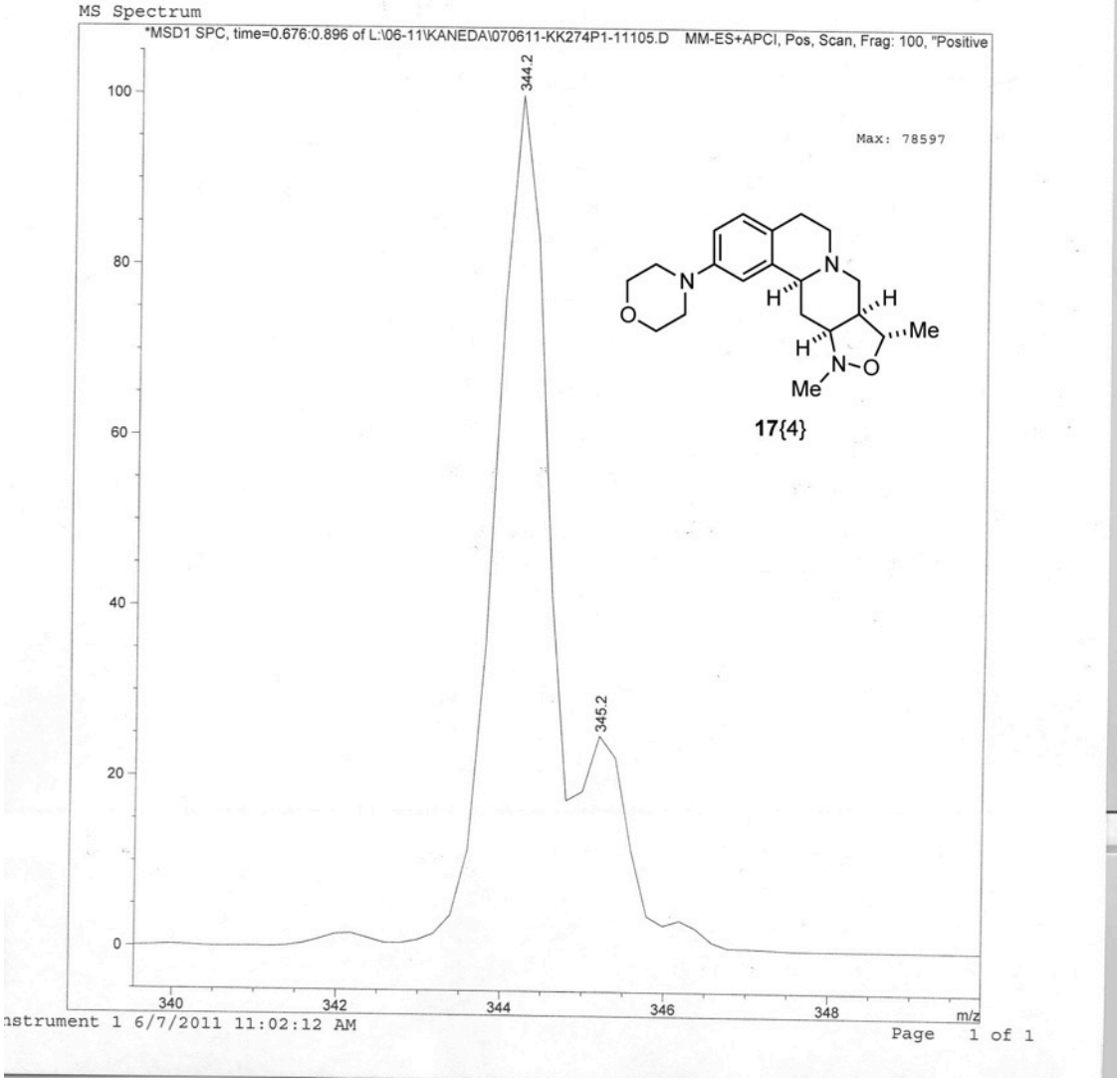
    ======================================================================
```

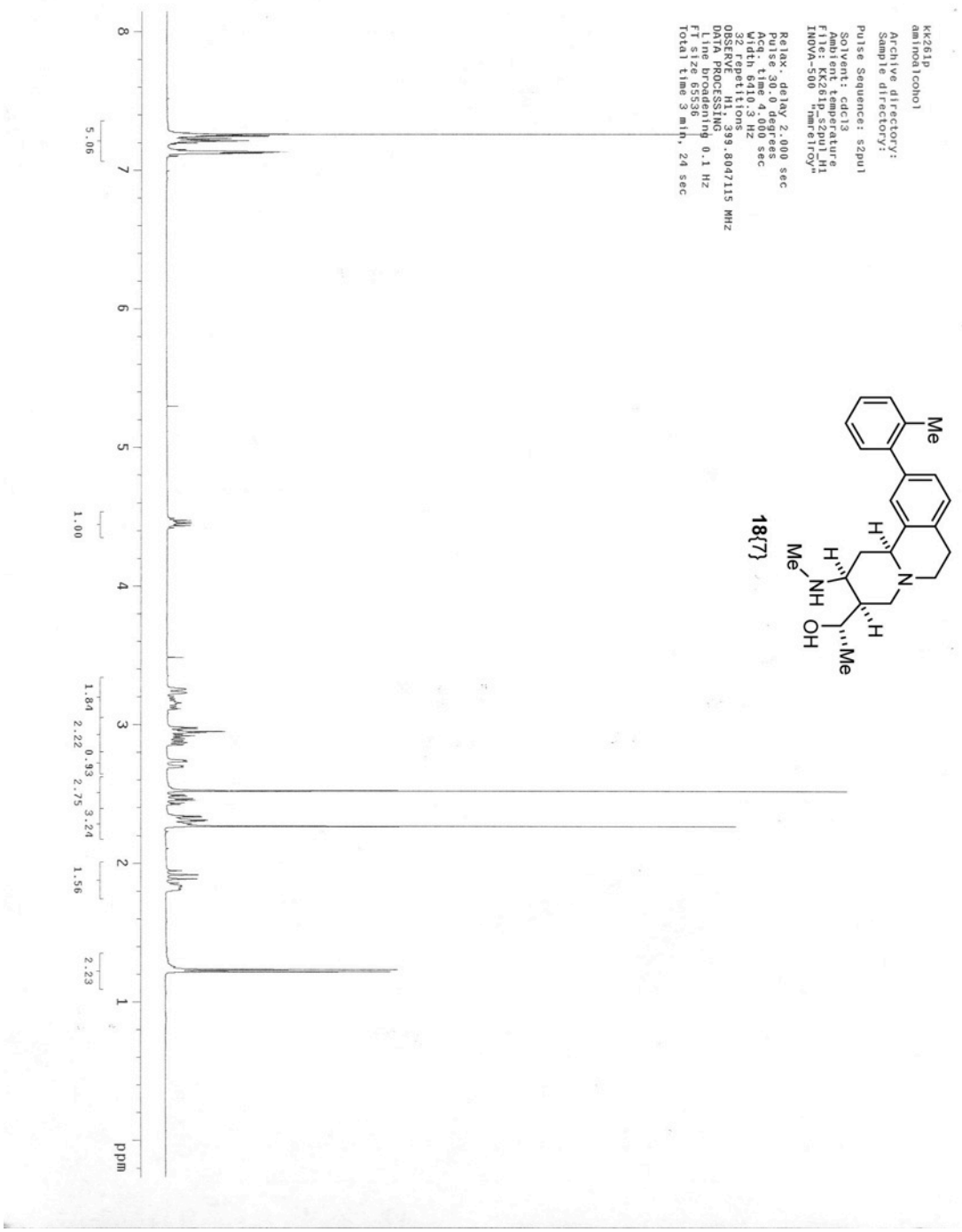


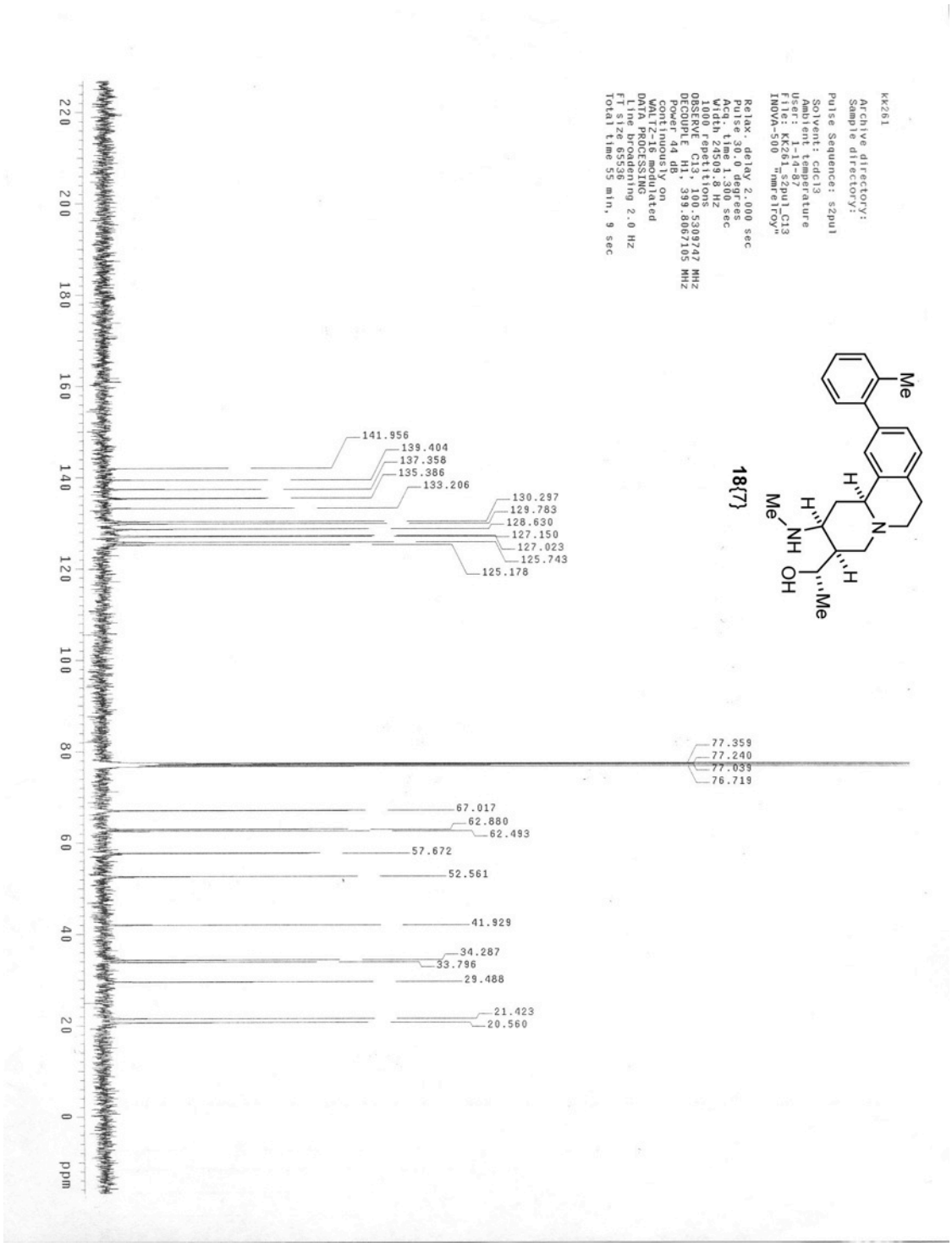
17{4}

Instrument 1 6/7/2011 11:01:36 AM Page 1 of 1

```
Print of window 79: MS Spectrum
Data File  : L:\06-11\KANEDA\070611-KK274P1-11105.D
Sample Name : kk274p
======================================================================
Acq. Operator   : kyosuke.kaneda@cm.utexas.edu
Acq. Instrument : LCMS                          Location : Vial 28
Injection Date  : 6/7/2011 10:16:47 AM             Inj :   1
                                                Inj Volume : 1.0 µl
Acq. Method     : C:\CHEM32\1\METHODS\LCMS 12MIN GRADIENT
Last changed    : 6/7/2011 10:16:26 AM by kyosuke.kaneda@cm.utexas.edu
                  (modified after loading)
Analysis Method : C:\CHEM32\1\METHODS\DEF_LC.M
Last changed    : 11/20/2006 4:14:44 AM
Sample Info     : Easy-Access Method: 'LCMS 12MIN GRADIENT'
```

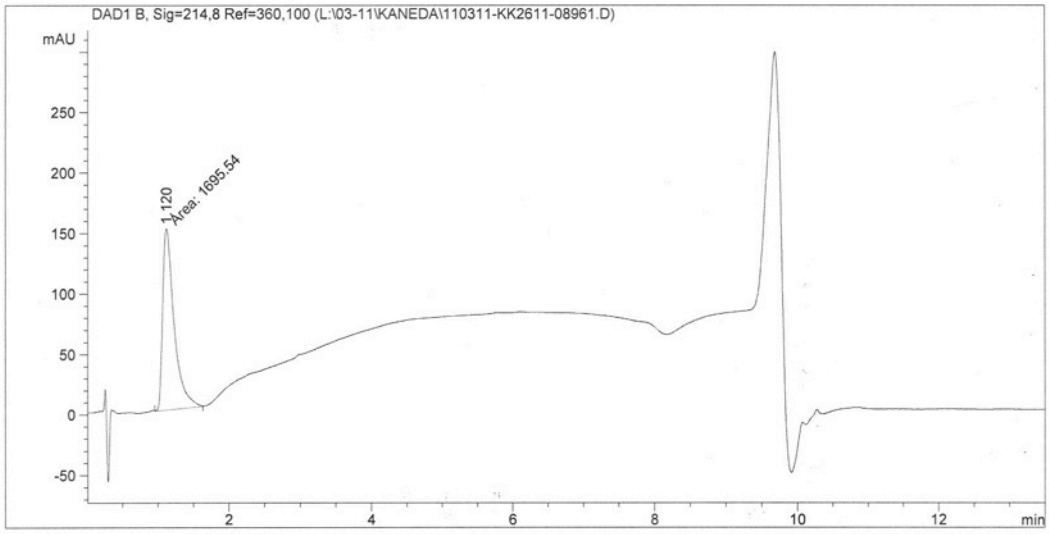



kk261p
aminoalcohol
Archive directory:
Sample directory:
Pulse Sequence: s2pul
Solvent: cdcl3
Ambient temperature
File: KK261p_s2pul_H1
INOVA-500 "marelroy"
Relax. delay 2.000 sec
Pulse 30.0 degrees
Acq. time 4.000 sec
Width 6410.3 Hz
32 repetitions
OBSERVE H1, 399.8047115 MHz
DATA PROCESSING
Line broadening 0.1 Hz
FT size 65536
Total time 3 min, 24 sec
Me
H
N
H
Me
NH
OH
H
Me
18{7}
8
7
6
5
4
3
2
1
ppm
5.06
1.00
1.84
2.22
0.93
2.75
3.24
1.56
2.23


kk261
Archive directory:
Sample directory:
Pulse Sequence: s2pul
Solvent: cdcl3
Ambient temperature
User: 1-14-87
File: KK261_s2pul_C13
INOVA-500 "mmrelroy"
Relax. delay 2.000 sec
Pulse 30.0 degrees
Acq. time 1.300 sec
Width 24509.8 Hz
1000 repetitions
OBSERVE C13, 100.530747 MHz
DECOUPLE H1, 399.806705 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
18{7}
Me
H
H
N
Me
NH
OH
H
Me
141.956
139.404
137.358
135.386
133.206
130.297
129.783
128.630
127.150
127.023
125.743
125.178
77.359
77.240
77.039
76.719
67.017
62.880
62.493
57.672
52.561
41.929
34.287
33.796
29.488
21.423
20.560
220
200
180
160
140
120
100
80
60
40
20
0
ppm

Data File L:\03-11\KANEDA\110311-KK2611-08961.D
Sample Name: kk261

```
==========================================================================
Acq. Operator   : kyosuke.kaneda@cm.utexas.edu
Acq. Instrument : LCMS                          Location : Vial 16
Injection Date  : 3/11/2011 7:42:24 PM
                                               Inj Volume : 1.0 µl
Acq. Method     : C:\CHEM32\1\METHODS\SP NIH.M
Last changed    : 3/11/2011 7:42:10 PM by kyosuke.kaneda@cm.utexas.edu
                  (modified after loading)
Analysis Method : C:\CHEM32\1\METHODS\DEF_LC.M
Last changed    : 11/20/2006 4:14:44 AM
Sample Info     : Easy-Access Method: 'SP NIH'
```




```
==========================================================================
                          Area Percent Report
==========================================================================

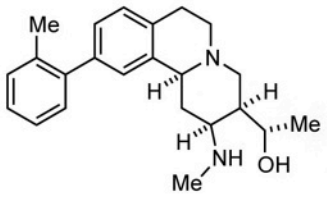
Sorted By             :      Signal
Multiplier:                   :      1.0000
Dilution:                     :      1.0000
Use Multiplier & Dilution Factor with ISTDs


Signal 1: DAD1 B, Sig=214,8 Ref=360,100

Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]   [mAU*s]     [mAU]       %
----|-------|----|-------|----------|----------|--------|
   1   1.120 MM    0.1880 1695.53796  150.33015 100.0000

Totals :                  1695.53796  150.33015

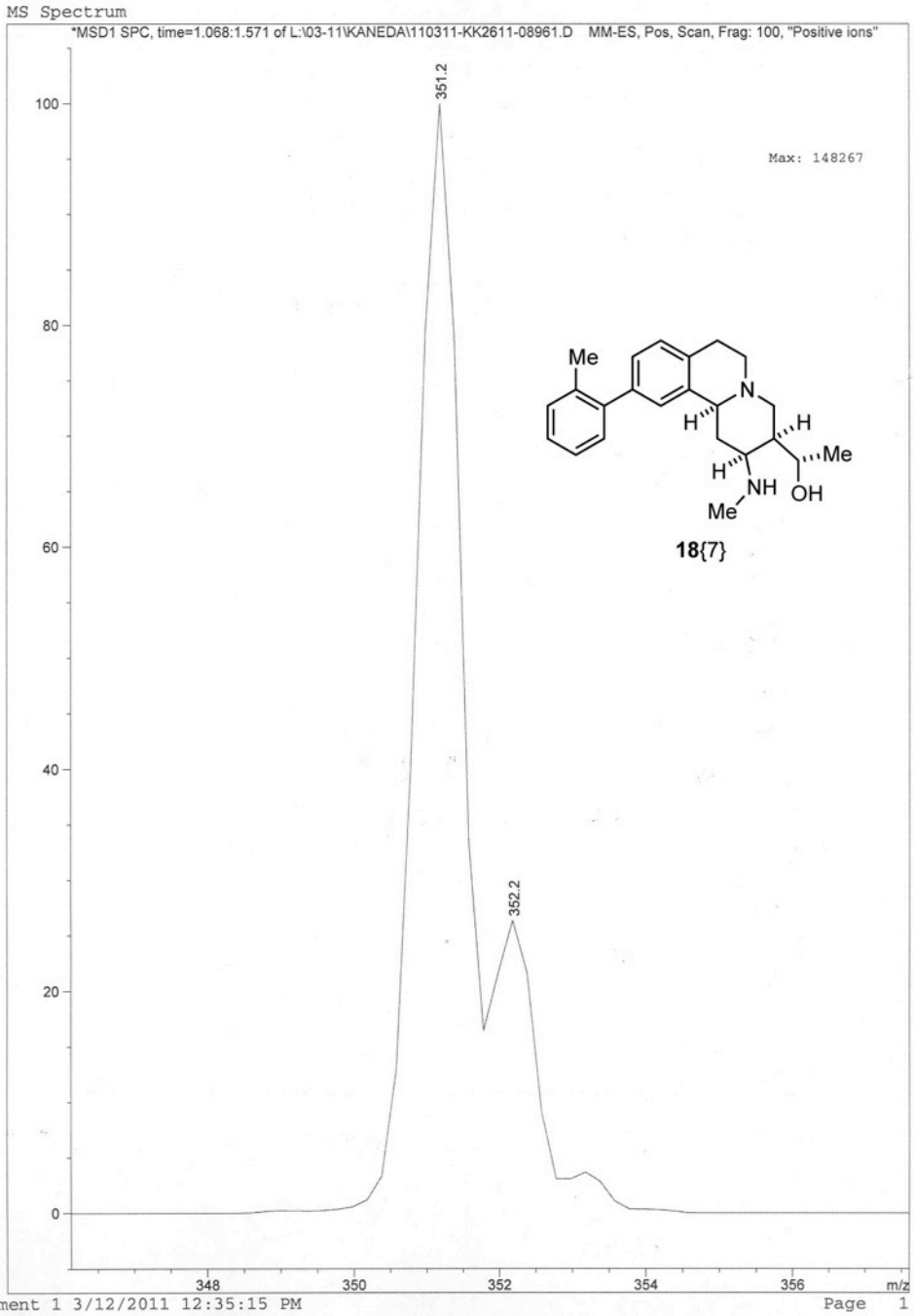
==========================================================================
```



18{7}

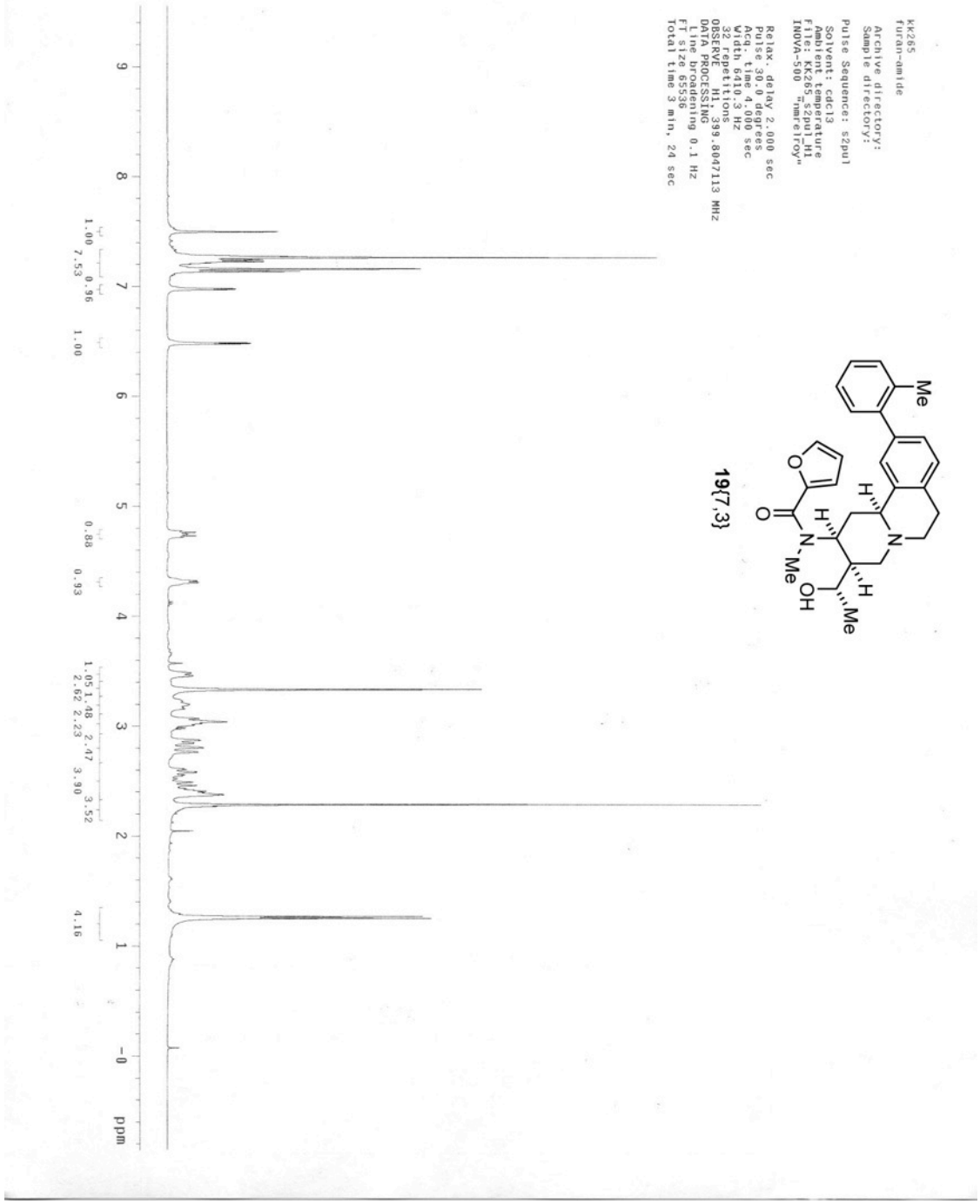
```
Instrument 1 3/12/2011 12:34:16 PM                          Page   1 of 1
```

Print of window 79: MS Spectrum



Instrument 1 3/12/2011 12:35:15 PM

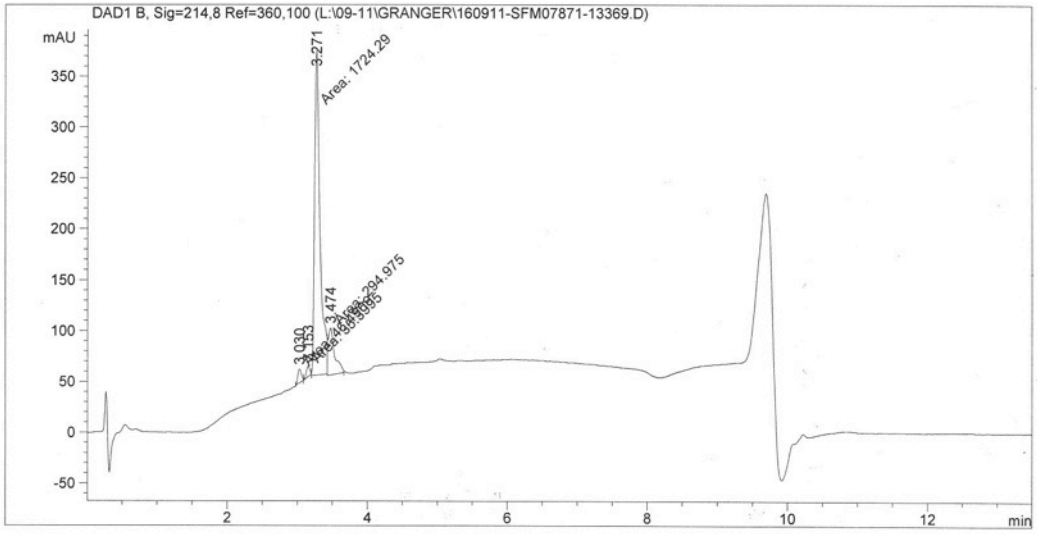
Page 1 of 1


kk265
furan-amide
Archive directory:
Sample directory:
Pulse Sequence: s2pul
Solvent: cdcl3
Ambient temperature
File: KK265_s2pul_H1
INOVA-500 "marelroy"
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
19(7,3)
Me
H
N
H
O
O
N
Me
OH
H
Me
9
8
7
6
5
4
3
2
1
-0
ppm
1.00
7.53
0.96
1.00
0.88
0.93
1.05
1.48
2.47
3.52
2.62
2.23
3.90
4.16

kk265
furan-amide
Archive directory:
Sample directory:
Pulse Sequence: s2pul
Solvent: cdcl3
Ambient temperature
User: ll-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, 100.530747 MHz
DECOUPLE H1, 399.886705 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
H
H
N
N
Me
OH
H
Me
O
O
19{7,3}
220
200
180
160
140
120
100
80
60
40
20
0
ppm

Data File L:\09-11\GRANGER\160911-SFM07871-13369.D
Sample Name: SFM0787

```
======================================================================
Acq. Operator   : brettag35@mail.utexas.edu
Acq. Instrument : LCMS                          Location : Vial 50
Injection Date  : 9/17/2011 1:32:48 AM
                                                Inj Volume : 1.0 µl
Acq. Method     : C:\CHEM32\1\METHODS\SP NIH.M
Last changed    : 9/17/2011 1:32:33 AM by brettag35@mail.utexas.edu
                  (modified after loading)
Analysis Method : C:\CHEM32\1\METHODS\DEF_LC.M
Last changed    : 11/20/2006 4:14:44 AM
Sample Info     : Easy-Access Method: 'SP NIH'
```



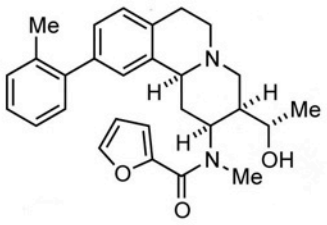

```
======================================================================
                        Area Percent Report
======================================================================

Sorted By             :      Signal
Multiplier:                   :      1.0000
Dilution:                     :      1.0000
Use Multiplier & Dilution Factor with ISTDs


Signal 1: DAD1 B, Sig=214,8 Ref=360,100

Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]   [mAU*s]     [mAU]       %
----|-------|----|-------|----------|----------|--------|
   1   3.030 MM    0.0562   46.49094   13.78585   2.2126
   2   3.153 MM    0.0556   35.39946   10.61999   1.6848
   3   3.271 MM    0.0899 1724.28870  319.64722  82.0639
   4   3.474 MM    0.1059  294.97543   46.44121  14.0387

Totals :                  2101.15453  390.49427
```



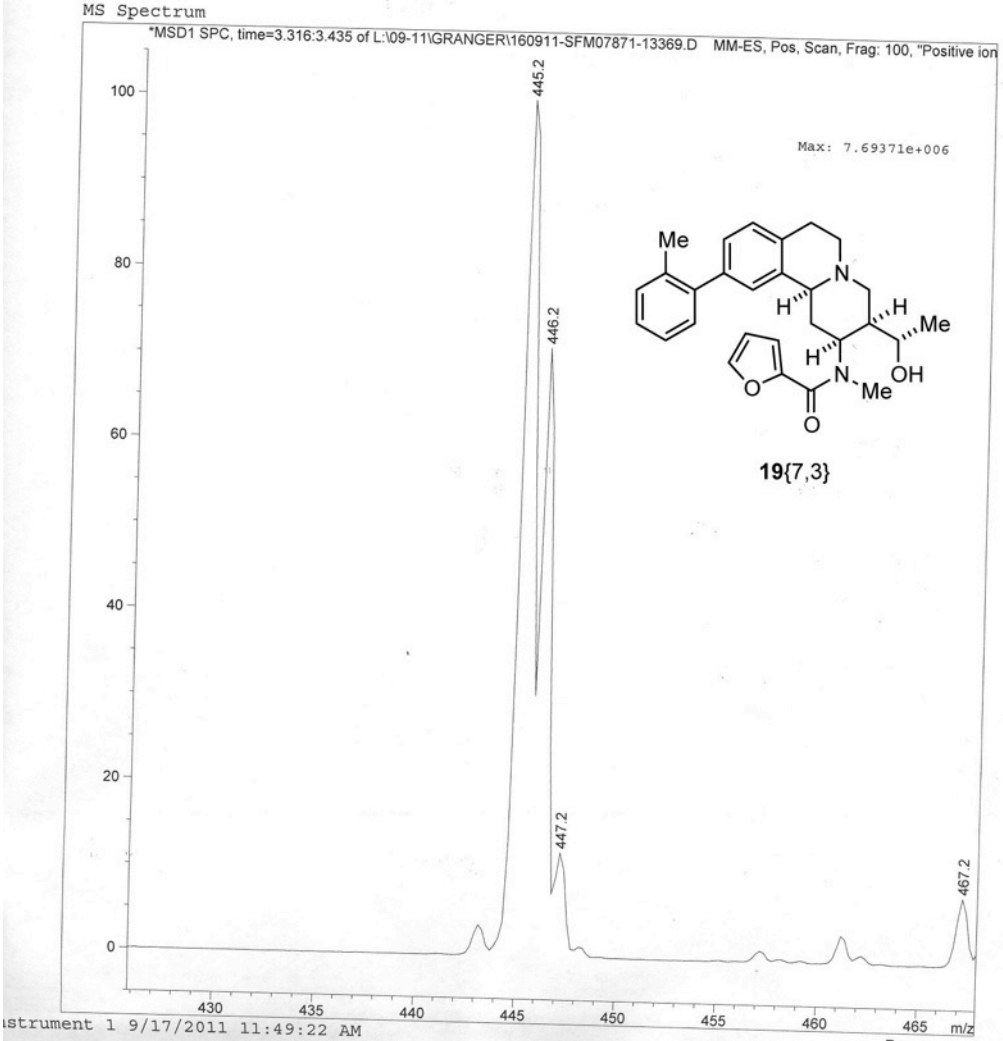
19{7,3}

Print of window 79: MS Spectrum
Data File : L:\09-11\GRANGER\160911-SFM07871-13369.D
Sample Name : SFM0787

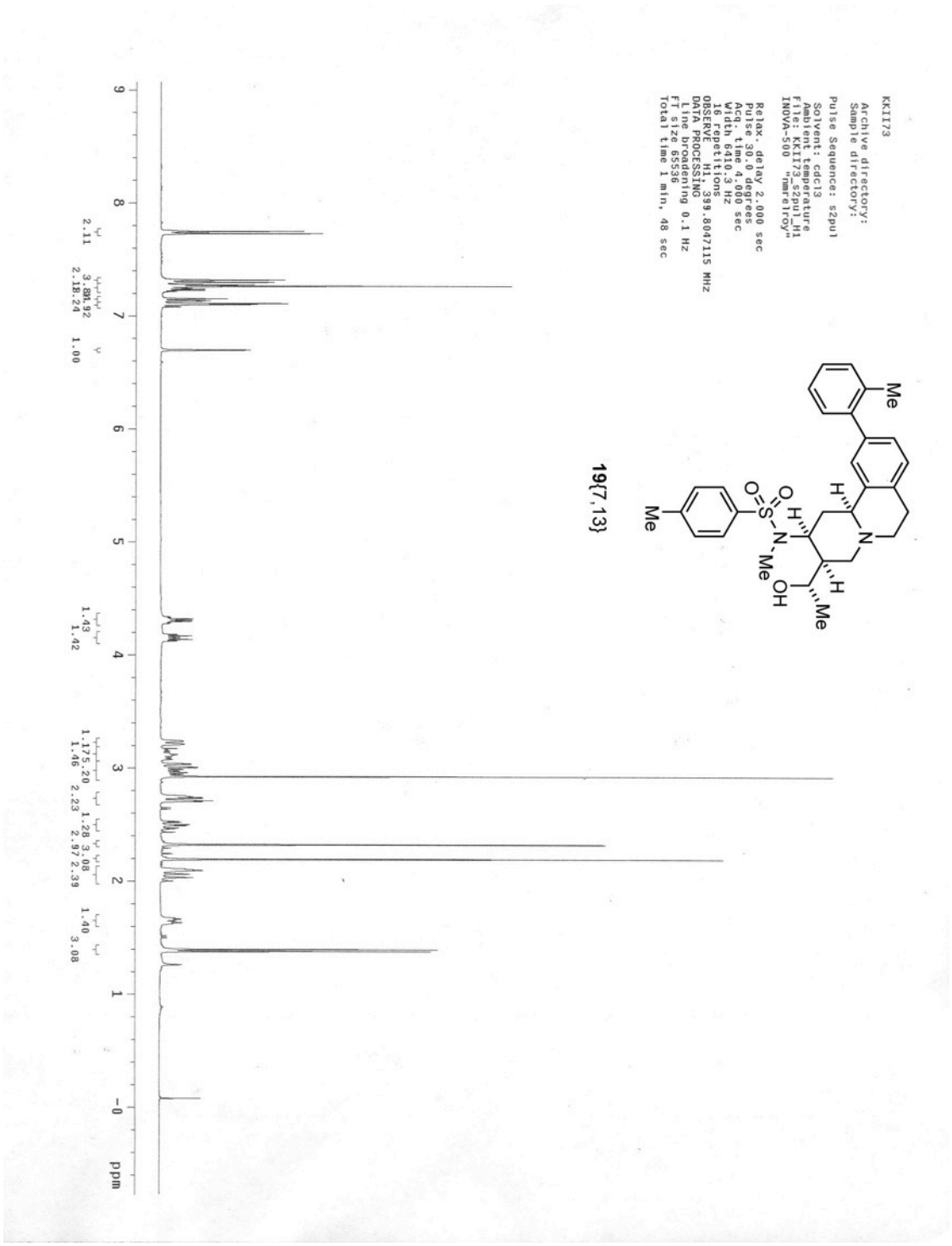
Acq. Operator : brettag35@mail.utexas.edu
Acq. Instrument : LCMS
Injection Date : 9/17/2011 1:32:48 AM
Location : Vial 50
Inj : 1
Inj Volume : 1.0 µl

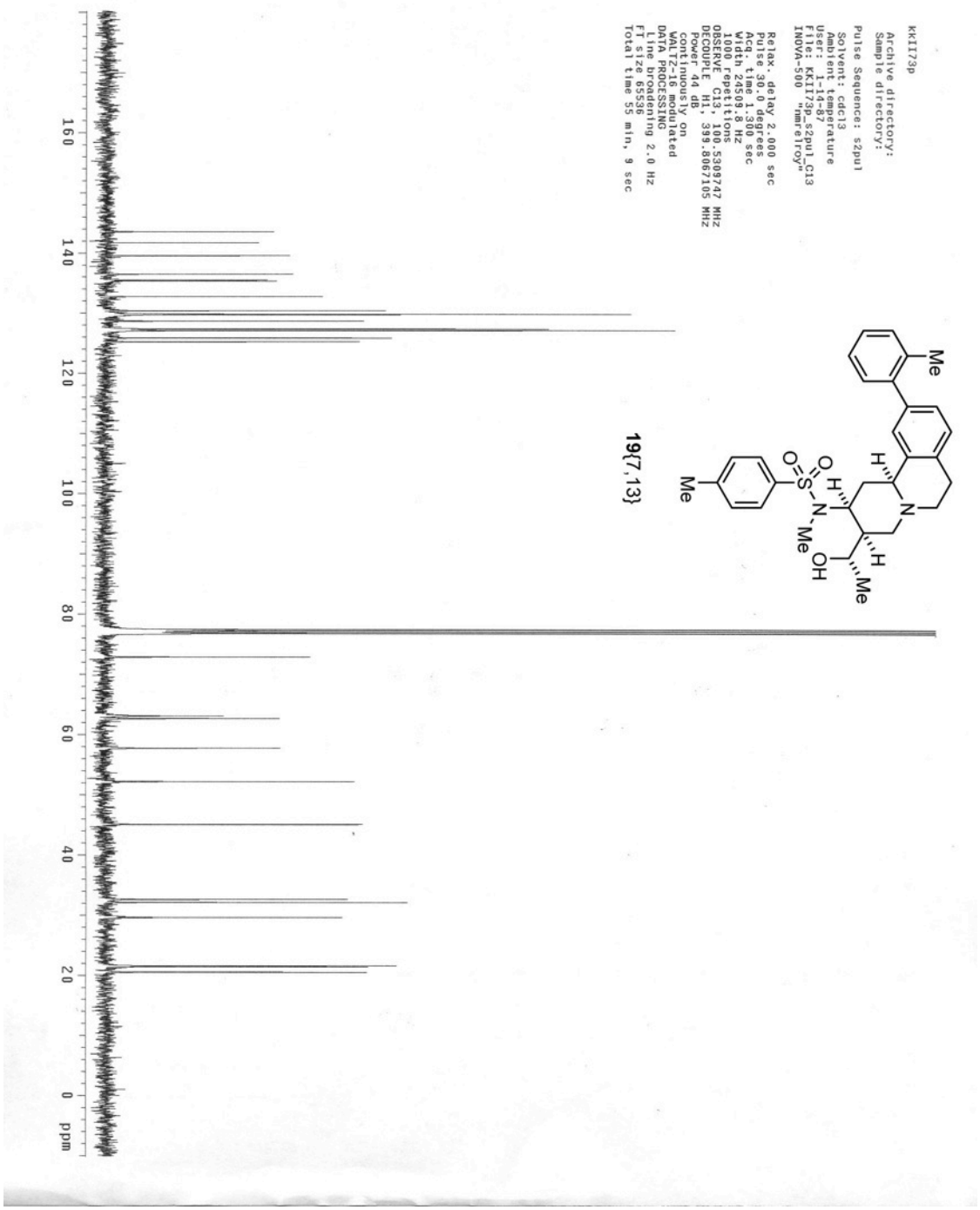
Acq. Method : C:\CHEM32\1\METHODS\SP NIH.M
Last changed : 9/17/2011 1:32:33 AM by brettag35@mail.utexas.edu
(modified after loading)
Analysis Method : C:\CHEM32\1\METHODS\DEF_LC.M
Last changed : 11/20/2006 4:14:44 AM
Sample Info : Easy-Access Method: 'SP NIH'

MS Spectrum



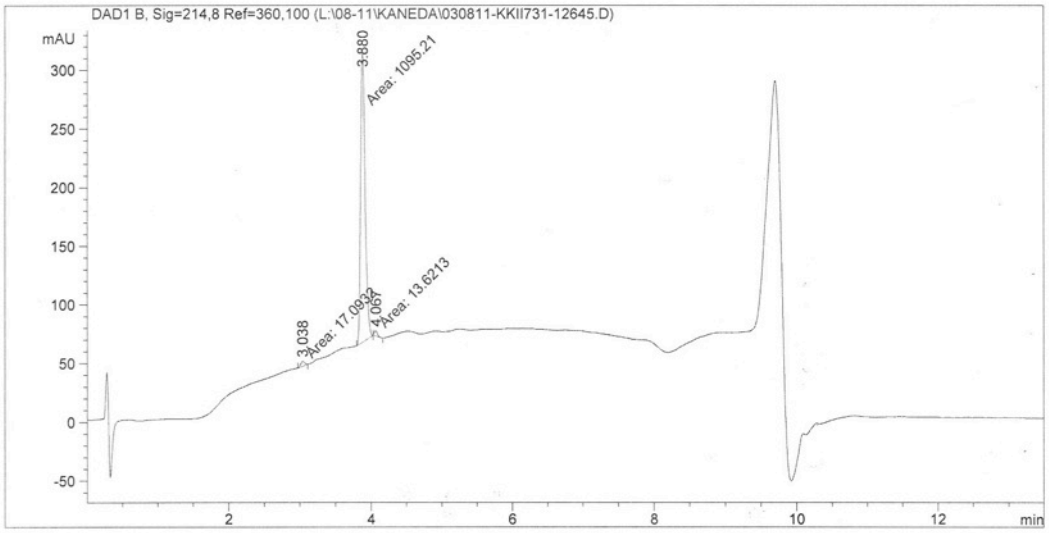
19{7,3}


KKII73
Archive directory:
Sample directory:
Pulse Sequence: s2pul
Solvent: cdcl3
Ambient temperature
File: KKII73_s2pul_H1
INOVA-500 "marelroy"
Relax. delay 2.000 sec
Pulse 30.0 degrees
Acq. time 4.000 sec
Width 6410.3 Hz
16 repetitions
OBSERVE H1, 399.8047115 MHz
DATA PROCESSING
Line broadening 0.1 Hz
FT size 65536
Total time 1 min, 48 sec
Me
H
N
O
S
N
Me
OH
H
Me
Me
19{7,13}
9
8
7
6
5
4
3
2
1
-0
ppm
2.11
3.81
3.92
2.18
2.24
1.00
1.43
1.42
1.175
1.20
1.46
2.23
1.28
3.08
2.97
2.39
1.40
3.08


KKII73p
Archive directory:
Sample directory:
Pulse Sequence: s2pul
Solvent: cdcl3
Ambient temperature
User: 1-14-87
File: KKII73p_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 40 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 2.0 Hz
FT size 65536
Total time 55 min, 9 sec
Me
H
O
H
S
N
Me
OH
H
Me
N
Me
19{7,13}
160
140
120
100
80
60
40
20
0
ppm

Data File L:\08-11\KANEDA\030811-KKII731-12645.D
Sample Name: KKII73

```
==========================================================================
Acq. Operator   : kyosuke.kaneda@cm.utexas.edu
Acq. Instrument : LCMS                          Location : Vial 2
Injection Date  : 8/3/2011 11:53:54 AM
                                                Inj Volume : 1.0 µl
Acq. Method     : C:\CHEM32\1\METHODS\SP NIH.M
Last changed    : 8/3/2011 11:53:33 AM by kyosuke.kaneda@cm.utexas.edu
                  (modified after loading)
Analysis Method : C:\CHEM32\1\METHODS\DEF_LC.M
Last changed    : 11/20/2006 4:14:44 AM
Sample Info     : Easy-Access Method: 'SP NIH'
```



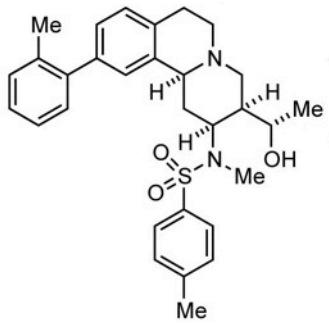

```
==========================================================================
                        Area Percent Report
==========================================================================

Sorted By             :      Signal
Multiplier:                   :      1.0000
Dilution:                     :      1.0000
Use Multiplier & Dilution Factor with ISTDs


Signal 1: DAD1 B, Sig=214,8 Ref=360,100

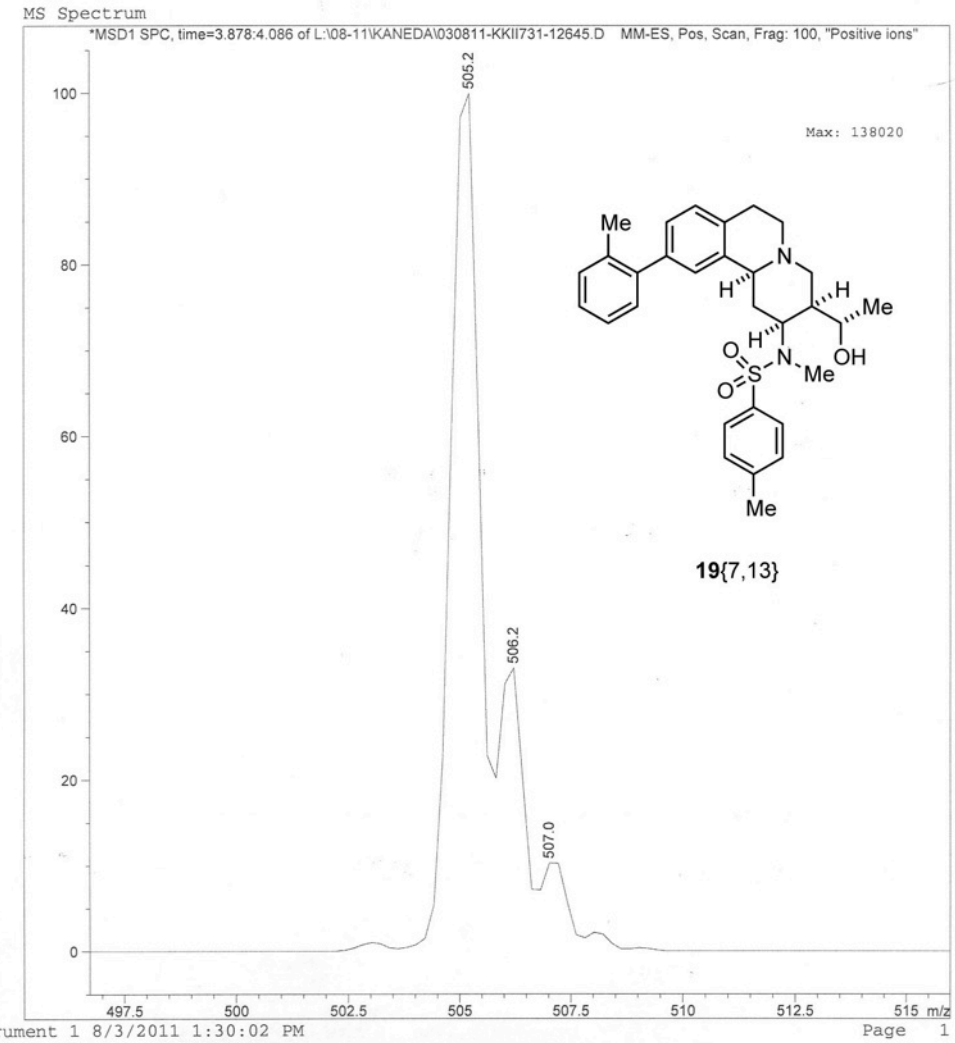
Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]   [mAU*s]     [mAU]       %
----|-------|----|-------|----------|----------|--------|
   1   3.038 MM    0.0648   17.09324    4.39901   1.5182
   2   3.880 MM    0.0734 1095.20935  248.54048  97.2721
   3   4.067 MM    0.0508   13.62129    4.46536   1.2098

Totals :                  1125.92388  257.40485
```

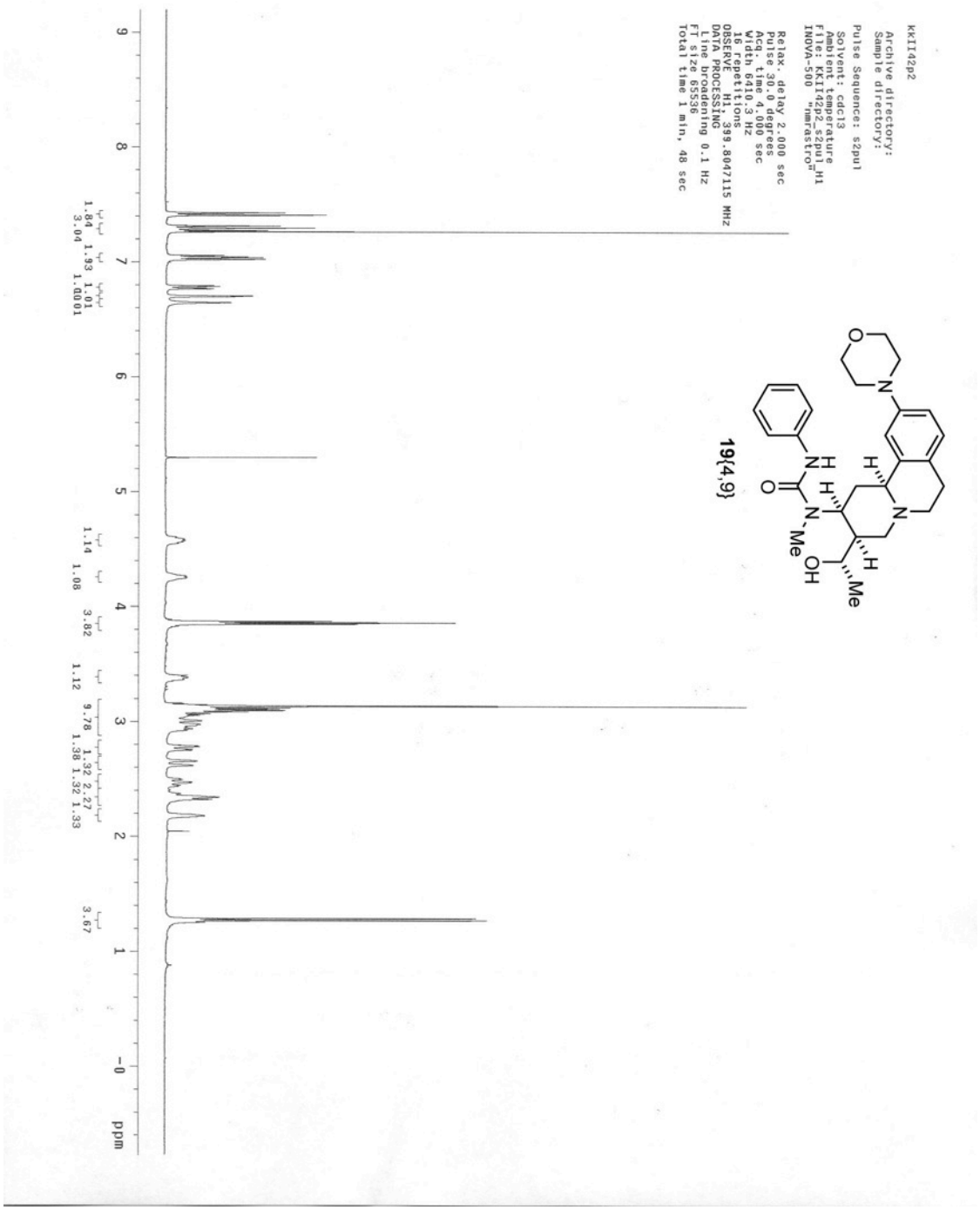


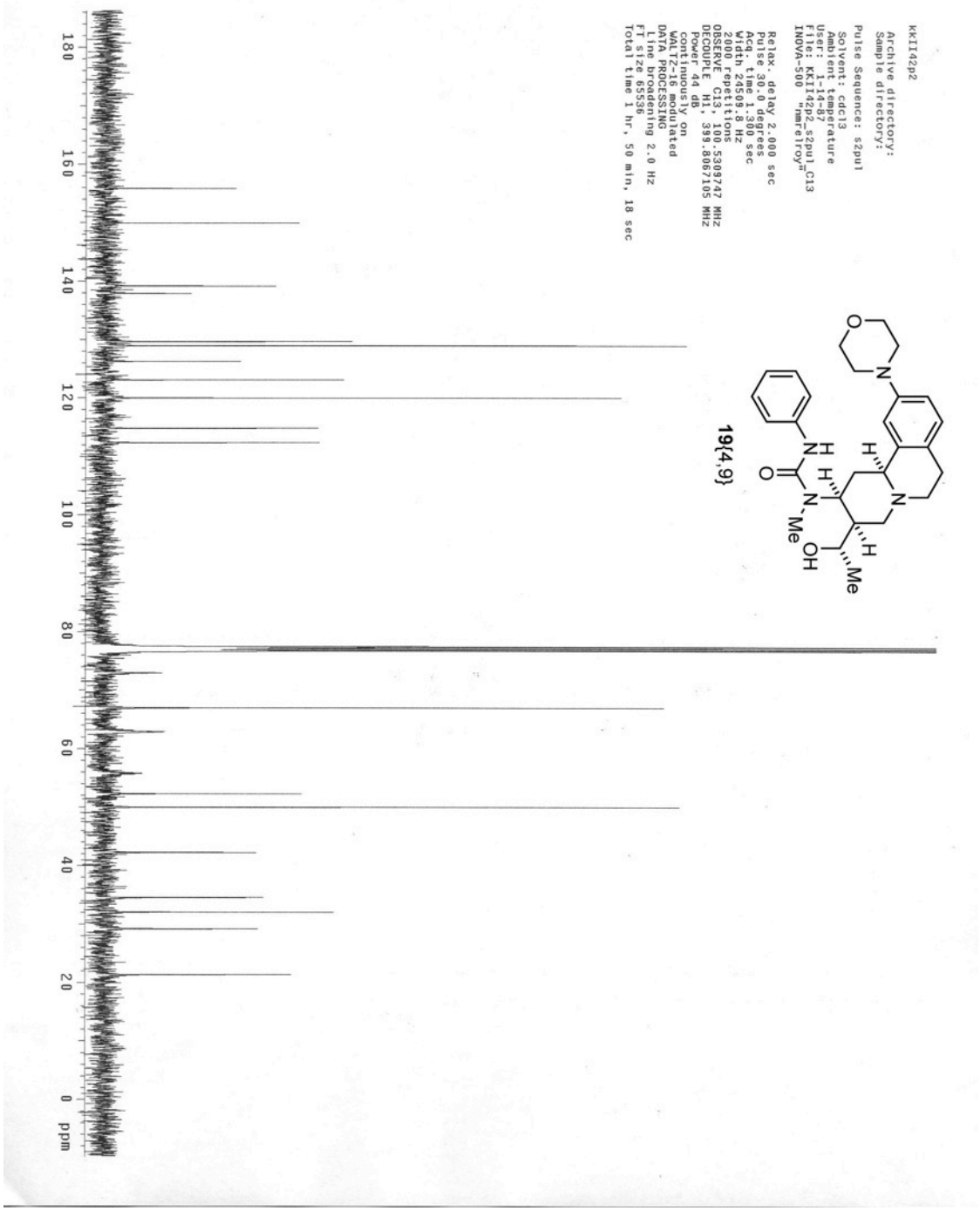
19{7,13}

```
Print of window 79: MS Spectrum
Data File  : L:\08-11\KANEDA\030811-KKII731-12645.D
Sample Name : KKII73
==================================================================
Acq. Operator   : kyosuke.kaneda@cm.utexas.edu
Acq. Instrument : LCMS                          Location : Vial 2
Injection Date  : 8/3/2011 11:53:54 AM               Inj :   1
                                                Inj Volume : 1.0 µl
Acq. Method     : C:\CHEM32\1\METHODS\SP NIH.M
Last changed    : 8/3/2011 11:53:33 AM by kyosuke.kaneda@cm.utexas.edu
                  (modified after loading)
Analysis Method : C:\CHEM32\1\METHODS\DEF_LC.M
Last changed    : 11/20/2006 4:14:44 AM
Sample Info     : Easy-Access Method: 'SP NIH'
```



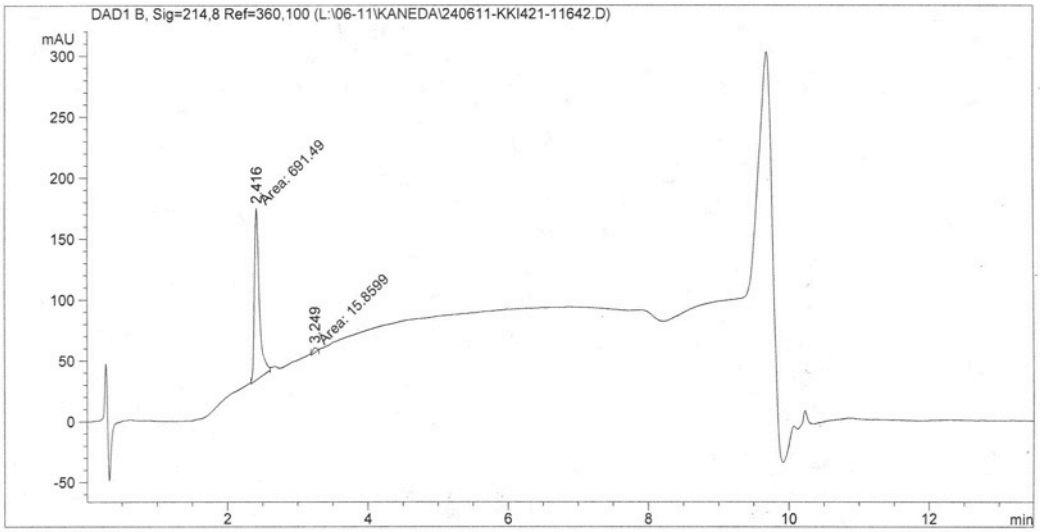
19{7,13}


kkII42p2
Archive directory:
Sample directory:
Pulse Sequence: s2pul
Solvent: cdcl3
Ambient temperature
File: KKII42p2.s2pul H1
INOVA-500 "nmrastro"
Relax. delay 2.000 sec
Pulse 30.0 degrees
Acq. time 4.000 sec
16 repetitions
Width 6410.3 Hz
OBSERVE H1, 399.8047115 MHz
DATA PROCESSING
Line broadening 0.1 Hz
FT size 65536
Total time 1 min, 48 sec
19{4,9}
Me
OH
ppm


kkII42p2
Archive directory:
Sample directory:
Pulse Sequence: s2pul
Solvent: cdcl3
Ambient temperature
User: 1-14-87
File: KkII42p2_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, 100.5309747 MHz
DECOUPLE H1, 399.8067105 MHz
Power 41 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 2.0 Hz
FT size 65536
Total time 1 hr, 50 min, 18 sec
19(4,9)
180
160
140
120
100
80
60
40
20
0
ppm

Data File L:\06-11\KANEDA\240611-KKI421-11642.D
Sample Name: kkI42

```
==========================================================================
Acq. Operator    : kyosuke.kaneda@cm.utexas.edu
Acq. Instrument : LCMS                              Location : Vial 54
Injection Date  : 6/24/2011 8:40:09 PM
                                                   Inj Volume : 1.0 µl
Acq. Method     : C:\CHEM32\1\METHODS\SP NIH.M
Last changed    : 6/24/2011 8:39:55 PM by kyosuke.kaneda@cm.utexas.edu
                  (modified after loading)
Analysis Method : C:\CHEM32\1\METHODS\DEF_LC.M
Last changed    : 11/20/2006 4:14:44 AM
Sample Info     : Easy-Access Method: 'SP NIH'
```



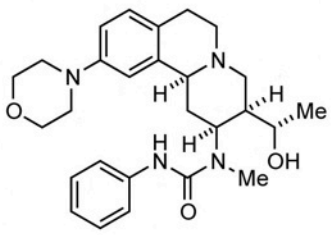

```
==========================================================================
                          Area Percent Report
==========================================================================

Sorted By             :      Signal
Multiplier:                   :      1.0000
Dilution:                     :      1.0000
Use Multiplier & Dilution Factor with ISTDs


Signal 1: DAD1 B, Sig=214,8 Ref=360,100

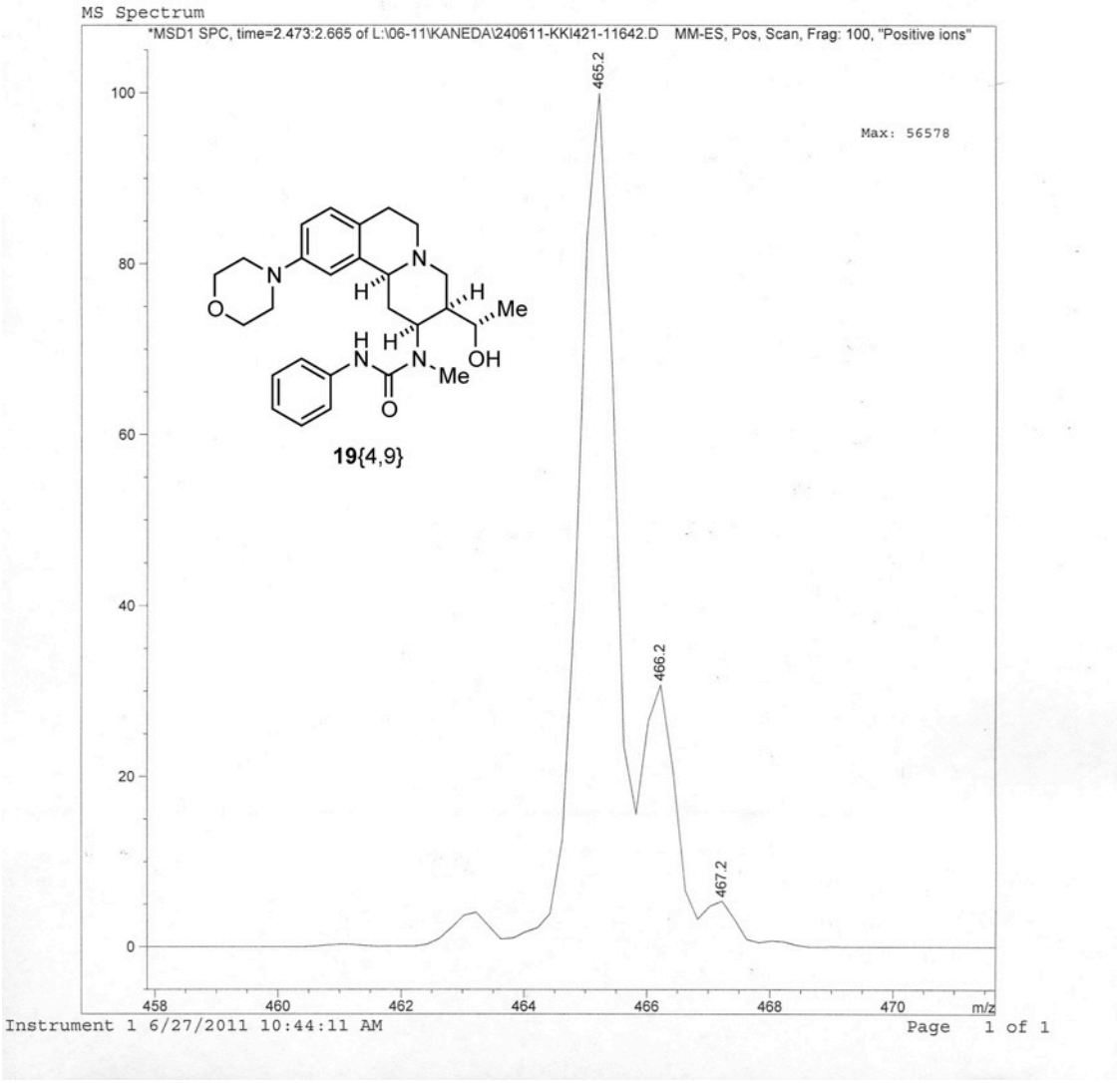
Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]   [mAU*s]     [mAU]      %
----|-------|----|-------|----------|----------|--------|
   1   2.416 MM    0.0814  691.48956  141.54823  97.7578
   2   3.249 MM    0.0661   15.85988    3.99740   2.2422

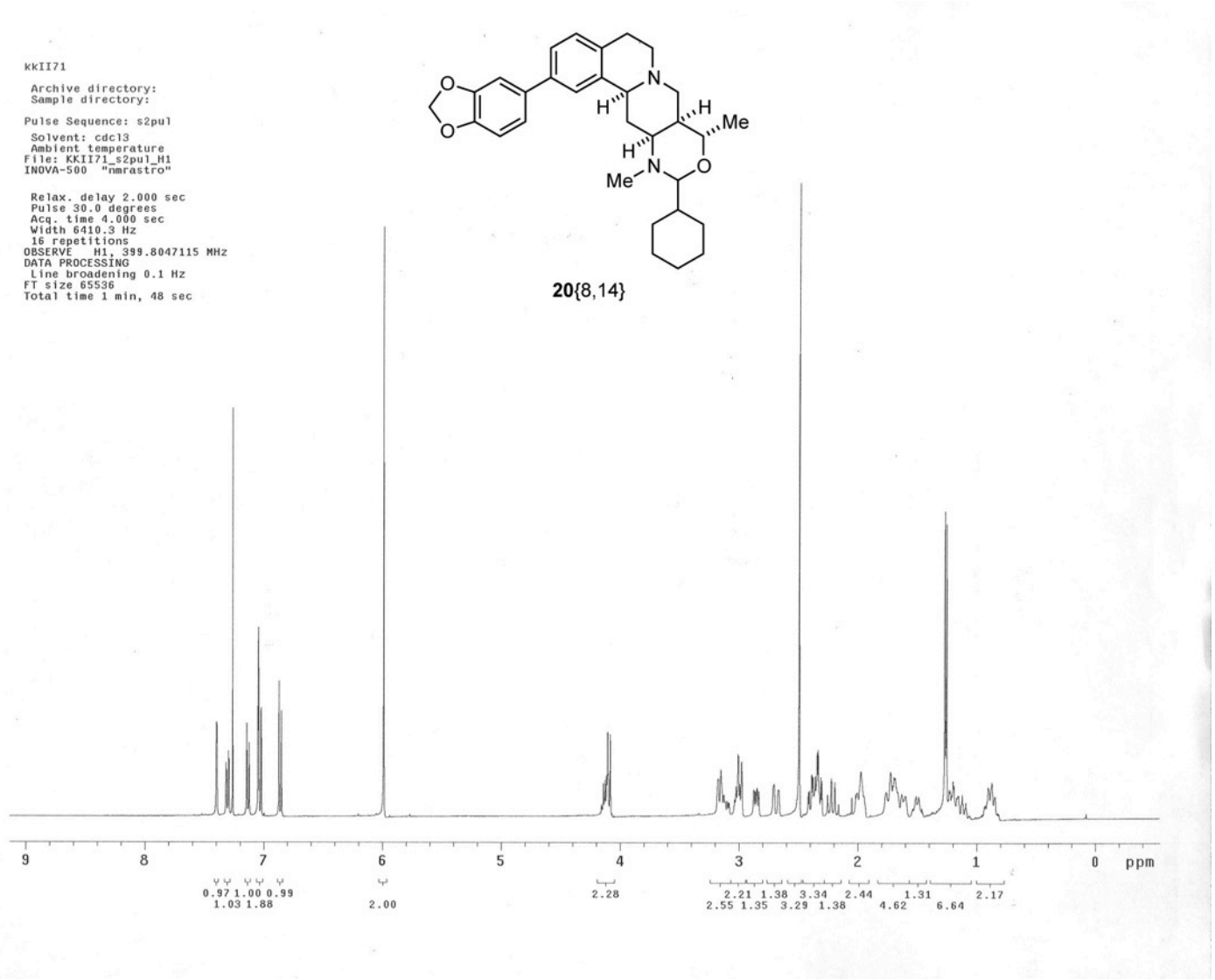
Totals :                   707.34944  145.54563
```

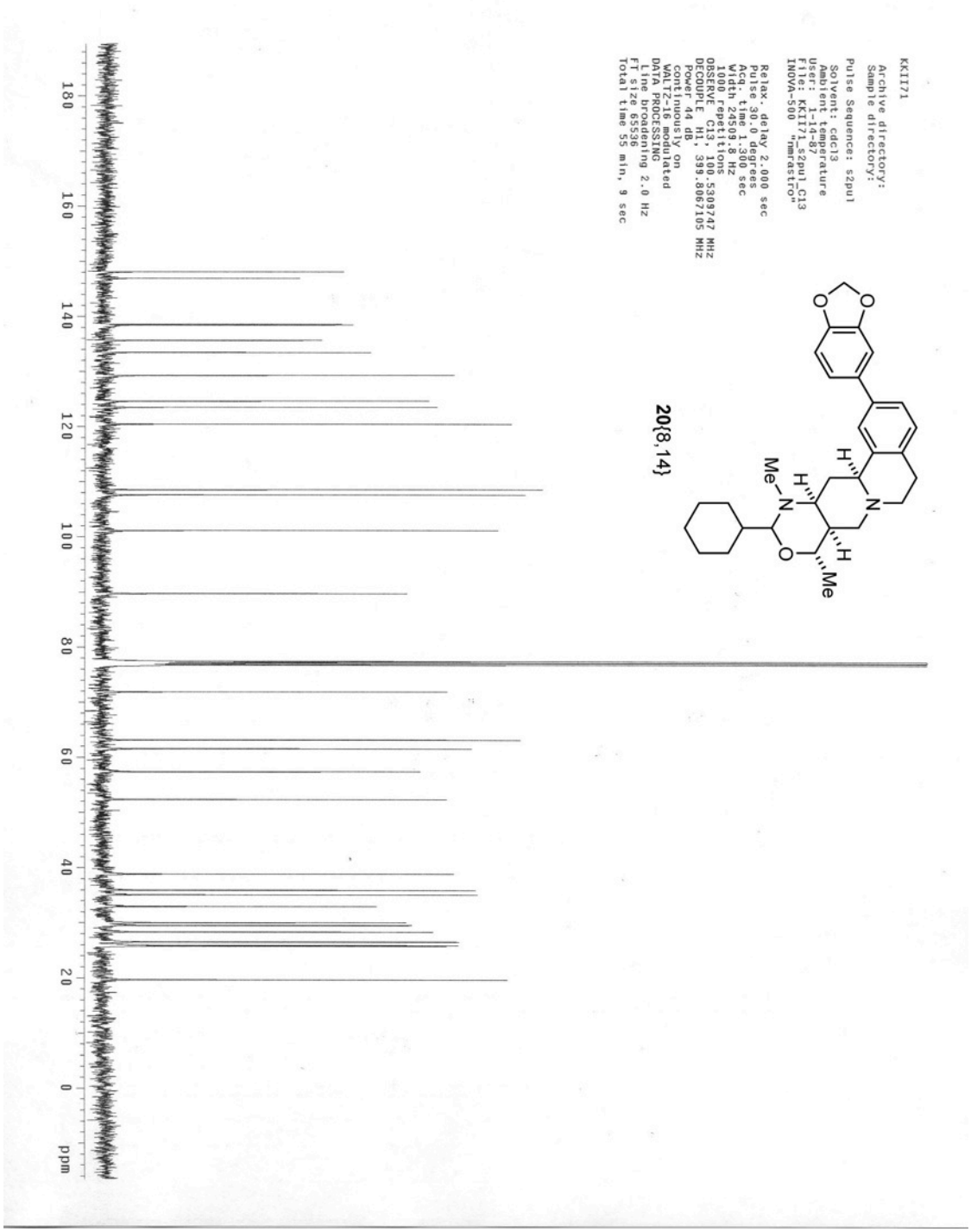


19{4,9}

```
Print of window 79: MS Spectrum
Data File  : L:\06-11\KANEDA\240611-KKI421-11642.D
Sample Name : kkI42
======================================================================
Acq. Operator   : kyosuke.kaneda@cm.utexas.edu
Acq. Instrument : LCMS                          Location : Vial 54
Injection Date  : 6/24/2011 8:40:09 PM               Inj :   1
                                                Inj Volume : 1.0 µl
Acq. Method     : C:\CHEM32\1\METHODS\SP NIH.M
Last changed    : 6/24/2011 8:39:55 PM by kyosuke.kaneda@cm.utexas.edu
                  (modified after loading)
Analysis Method : C:\CHEM32\1\METHODS\DEF_LC.M
Last changed    : 11/20/2006 4:14:44 AM
Sample Info     : Easy-Access Method: 'SP NIH'
```

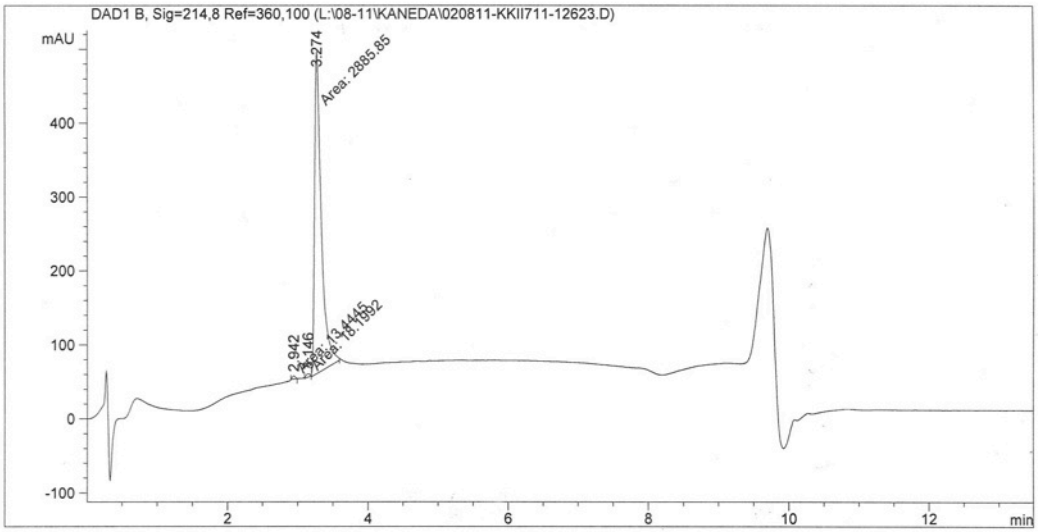



kkII71
Archive directory:
Sample directory:
Pulse Sequence: s2pul
Solvent: cdcl3
Ambient temperature
File: KKII71_s2pul_H1
INOVA-500 "nmrastro"
Relax. delay 2.000 sec
Pulse 30.0 degrees
Acq. time 4.000 sec
Width 6410.3 Hz
16 repetitions
OBSERVE H1, 399.8047115 MHz
DATA PROCESSING
Line broadening 0.1 Hz
FT size 65536
Total time 1 min, 48 sec
20{8,14}
9
8
7
6
5
4
3
2
1
0
ppm
0.97 1.00 0.99
1.03 1.88
2.00
2.28
2.21 1.38 3.34 2.44 1.31 2.17
2.55 1.35 3.29 1.38 4.62 6.64


KKII71
Archive directory:
Sample directory:
Pulse Sequence: s2pul
Solvent: cdcl3
Ambient temperature
User: 1-14-87
File: KKII71_s2pul_C13
INOVA-500 "nmrastro"
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 34 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 2.0 Hz
FT size 65536
Total time 55 min, 9 sec
20{8,14}
H
H
Me
N
N
H
Me
O
O
O
180
160
140
120
100
80
60
40
20
0
ppm

Data File L:\08-11\KANEDA\020811-KKII711-12623.D
Sample Name: KKII71

```
==========================================================================
Acq. Operator    : kyosuke.kaneda@cm.utexas.edu
Acq. Instrument : LCMS                              Location : Vial 45
Injection Date  : 8/2/2011 10:39:55 PM
                                                   Inj Volume : 1.0 µl
Acq. Method     : C:\CHEM32\1\METHODS\SP NIH.M
Last changed    : 8/2/2011 10:39:40 PM by kyosuke.kaneda@cm.utexas.edu
                  (modified after loading)
Analysis Method : C:\CHEM32\1\METHODS\DEF_LC.M
Last changed    : 11/20/2006 4:14:44 AM
Sample Info     : Easy-Access Method: 'SP NIH'
```



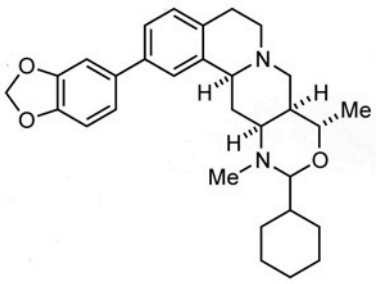

```
==========================================================================
                          Area Percent Report
==========================================================================

Sorted By             :      Signal
Multiplier:                   :      1.0000
Dilution:                     :      1.0000
Use Multiplier & Dilution Factor with ISTDs


Signal 1: DAD1 B, Sig=214,8 Ref=360,100

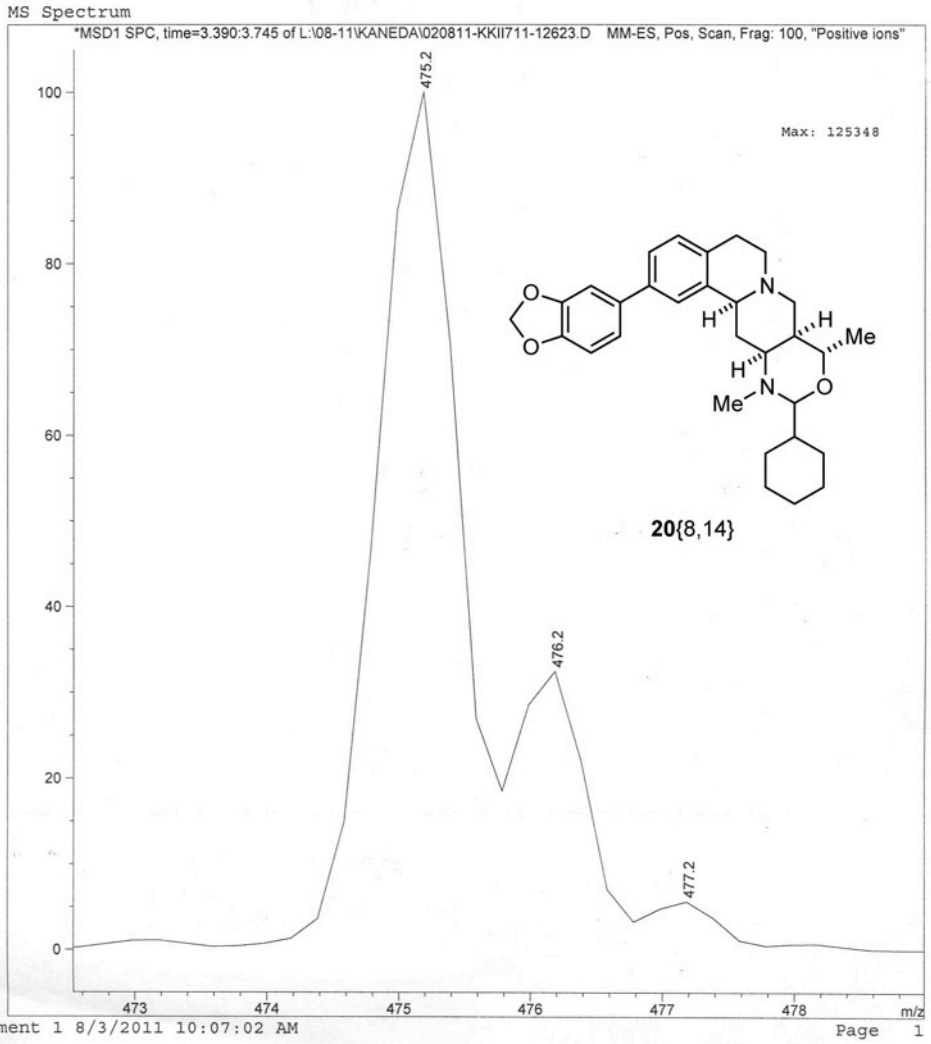
Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]   [mAU*s]     [mAU]       %
----|-------|----|-------|----------|----------|--------|
   1   2.942 MM    0.0501   13.44451    4.47037   0.4608
   2   3.146 MM    0.0614   18.19924    4.94061   0.6238
   3   3.274 MM    0.1099 2885.85205  437.74823  98.9154

Totals :                  2917.49580  447.15921
```

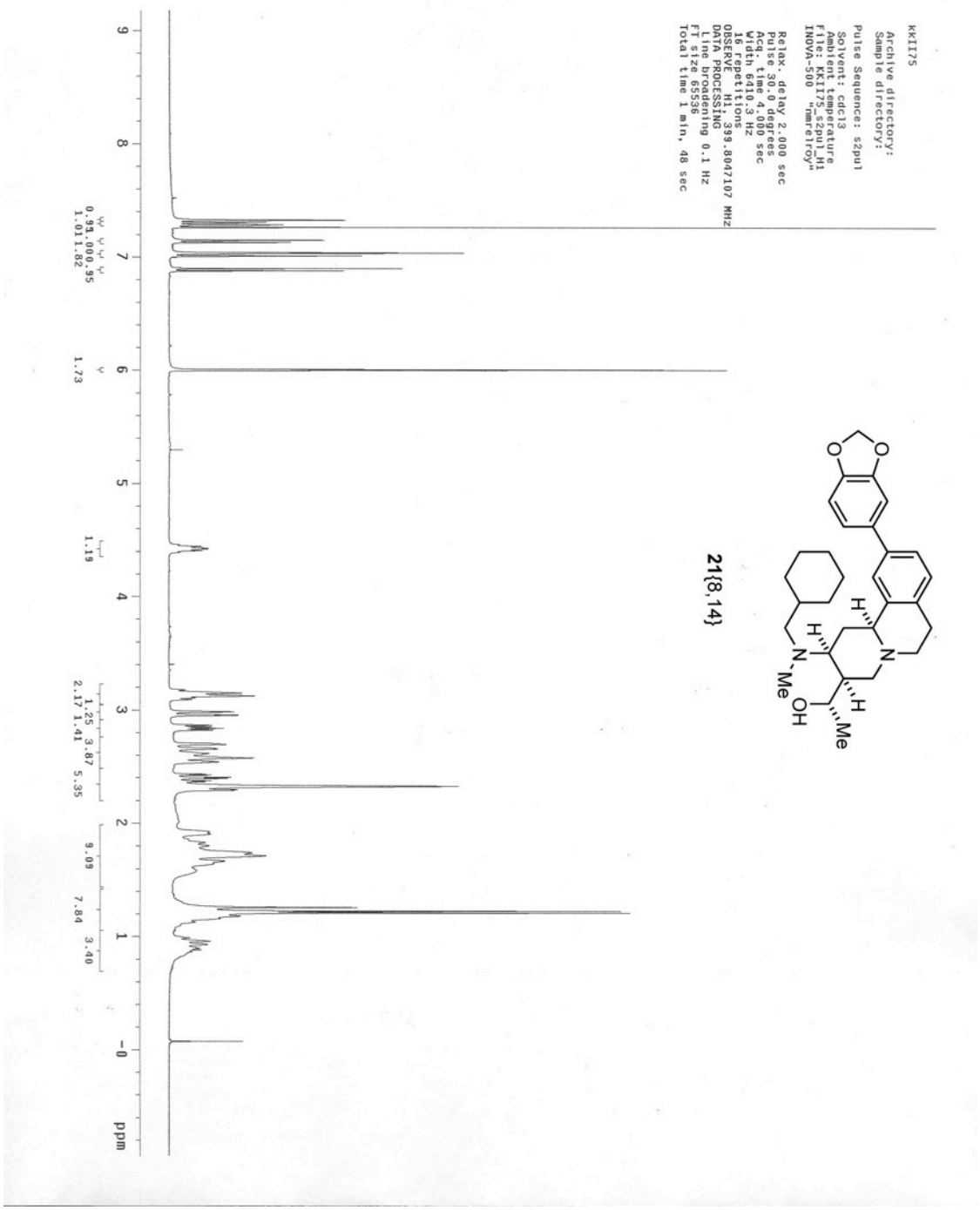


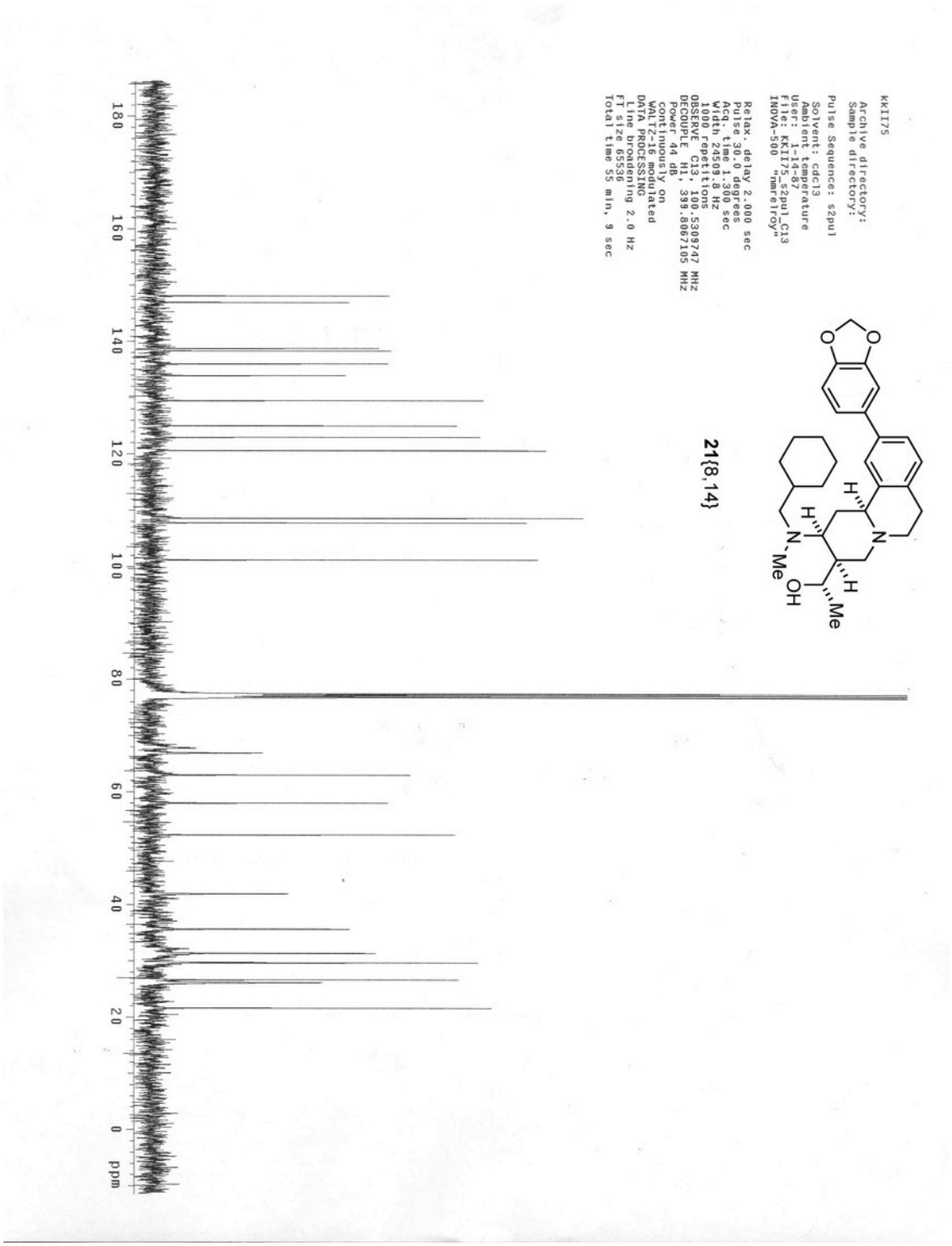
20{8,14}

```
Print of window 79: MS Spectrum
Data File  : L:\08-11\KANEDA\020811-KKII711-12623.D
Sample Name : KKII71
======================================================================
Acq. Operator   : kyosuke.kaneda@cm.utexas.edu
Acq. Instrument : LCMS                          Location : Vial 45
Injection Date  : 8/2/2011 10:39:55 PM               Inj :   1
                                                Inj Volume : 1.0 µl
Acq. Method     : C:\CHEM32\1\METHODS\SP NIH.M
Last changed    : 8/2/2011 10:39:40 PM by kyosuke.kaneda@cm.utexas.edu
                  (modified after loading)
Analysis Method : C:\CHEM32\1\METHODS\DEF_LC.M
Last changed    : 11/20/2006 4:14:44 AM
Sample Info     : Easy-Access Method: 'SP NIH'
```



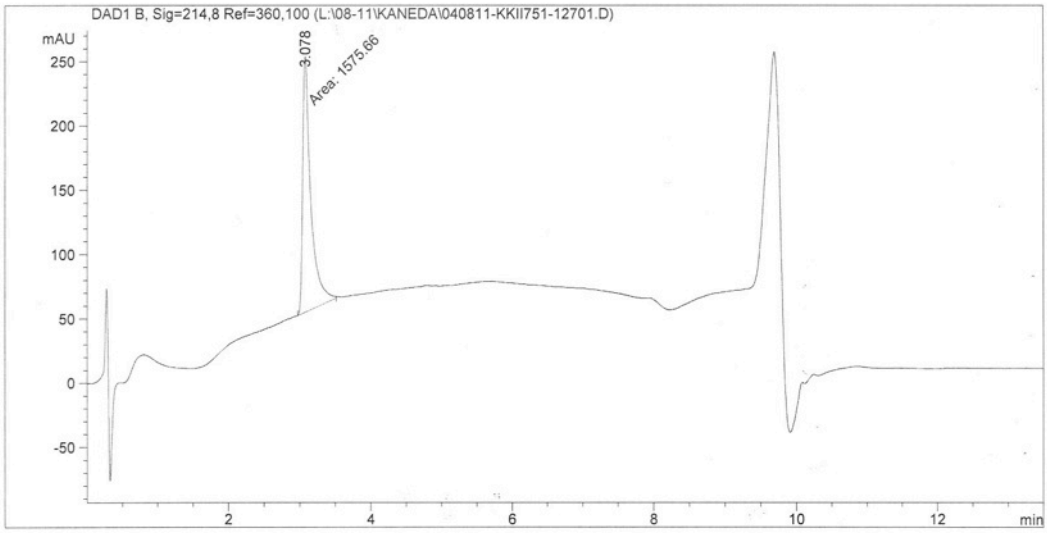
Instrument 1 8/3/2011 10:07:02 AM Page 1 of 1


KKII75
Archive directory:
Sample directory:
Pulse Sequence: s2pul
Solvent: cdcl3
Ambient temperature
File: KKII75_s2pul_H1
INOVA-500 "marelroy"
Relax. delay 2.000 sec
Pulse 30.0 degrees
Acq. time 4.000 sec
Width 6410.3 Hz
16 repetitions
OBSERVE H1, 399.8047107 MHz
DATA PROCESSING
Line broadening 0.1 Hz
FT size 65536
Total time 1 min, 48 sec
21{8,14}
H
H
N
N
Me
H
Me
OH
0.99 1.00 0.95
1.01 1.82
1.73
1.19
1.25 3.87
2.17 1.41 5.35
9.09
7.84
3.40
9
8
7
6
5
4
3
2
1
-0
ppm


kkII75
Archive directory:
Sample directory:
Pulse Sequence: s2pul
Solvent: cdcl3
Ambient temperature
User: lt-14-87
File: KKII75_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
21{8,14}
H
H
N
N
Me
OH
H
Me
180
160
140
120
100
80
60
40
20
0
ppm

Data File L:\08-11\KANEDA\040811-KKII751-12701.D
Sample Name: KKII75

```
=====================================================================
Acq. Operator   : kyosuke.kaneda@cm.utexas.edu
Acq. Instrument : LCMS                          Location : Vial 58
Injection Date  : 8/4/2011 4:06:05 PM
                                                Inj Volume : 1.0 µl
Acq. Method     : C:\CHEM32\1\METHODS\SP NIH.M
Last changed    : 8/4/2011 4:05:51 PM by kyosuke.kaneda@cm.utexas.edu
                  (modified after loading)
Analysis Method : C:\CHEM32\1\METHODS\DEF_LC.M
Last changed    : 11/20/2006 4:14:44 AM
Sample Info     : Easy-Access Method: 'SP NIH'
```




```
=====================================================================
                        Area Percent Report
=====================================================================

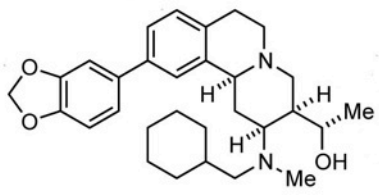
Sorted By             :      Signal
Multiplier:                   :      1.0000
Dilution:                     :      1.0000
Use Multiplier & Dilution Factor with ISTDs


Signal 1: DAD1 B, Sig=214,8 Ref=360,100

Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]   [mAU*s]     [mAU]      %
----|-------|----|-------|----------|----------|--------|
   1   3.078 MM    0.1315 1575.65979  199.70491 100.0000

Totals :                  1575.65979  199.70491

=====================================================================
```



21{8,14}

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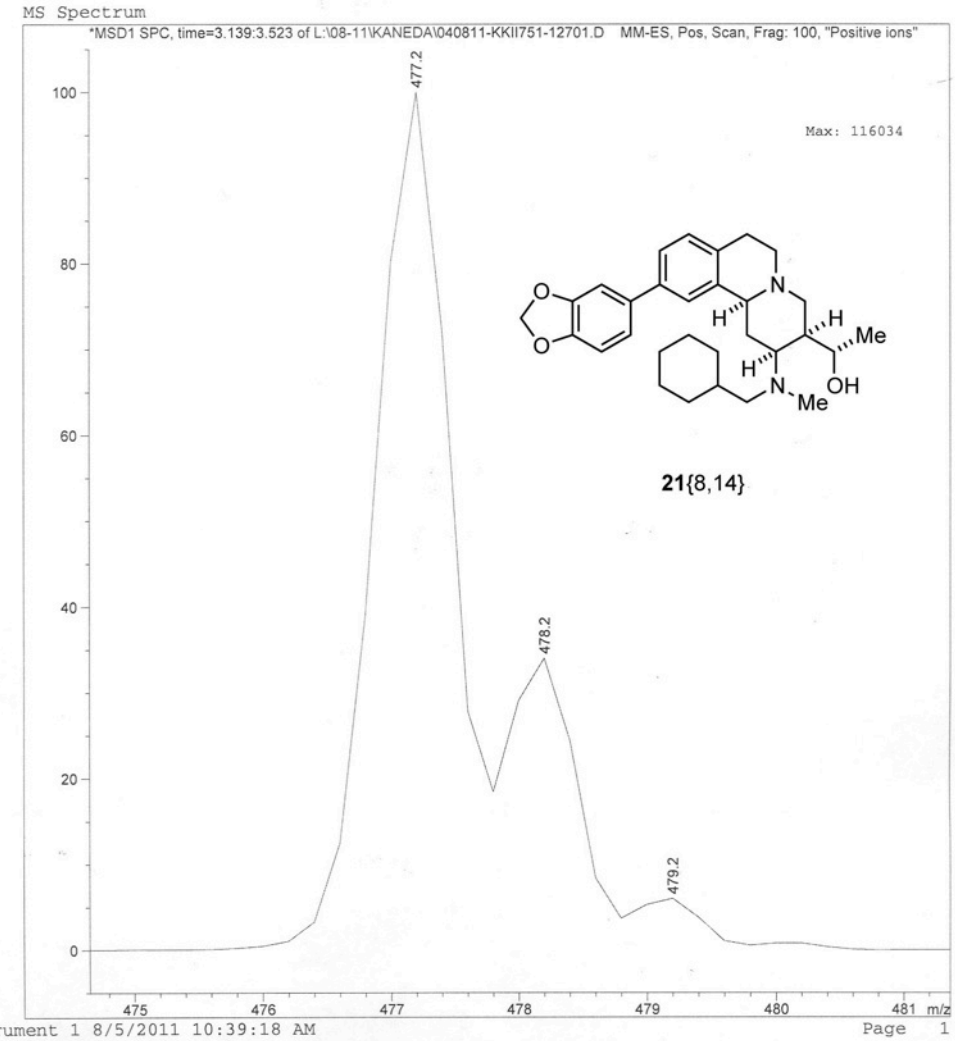
```
Print of window 79: MS Spectrum
Data File  : L:\08-11\KANEDA\040811-KKII751-12701.D
Sample Name : KKII75
======================================================================
Acq. Operator   : kyosuke.kaneda@cm.utexas.edu
Acq. Instrument : LCMS                          Location : Vial 58
Injection Date  : 8/4/2011 4:06:05 PM                Inj :   1
                                                Inj Volume : 1.0 µl
Acq. Method     : C:\CHEM32\1\METHODS\SP NIH.M
Last changed    : 8/4/2011 4:05:51 PM by kyosuke.kaneda@cm.utexas.edu
                  (modified after loading)
Analysis Method : C:\CHEM32\1\METHODS\DEF_LC.M
Last changed    : 11/20/2006 4:14:44 AM
Sample Info     : Easy-Access Method: 'SP NIH'
```



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