Electronic Supplementary Material (ESI) for ChemComm.

Expedient Synthesis of Tetrahydroquinoline-3-Spirohydantoin Derivatives *via* Lewis Acid-Catalyzed *tert*-Amino Effect Reaction

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Supporting Information

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1. General Methods

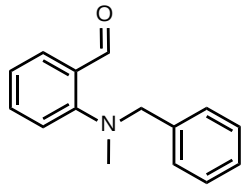
All of the solvents and reagents used were obtained commercially and used as such unless noted otherwise. ^{1}H NMR spectra were recorded in $CDCl_3$ or DMSO-*d6* solutions at 300 K using a Bruker Ultrashield 300 MHz instrument or a Bruker Ultrashield 400 MHz instrument. ^{13}C NMR spectra were recorded in DMSO-*d6* solutions at 300 K and 126 MHz using a Bruker DRX-500 500 MHz instrument with a QNP cryoprobe or at 101 MHz using a Bruker Ultrashield 400 MHz instrument or at 75.5 MHz using a Bruker Ultrashield 300 MHz instrument. ^{19}F NMR spectra were recorded at 282 MHz in $CDCl_3$ or DMSO-*d6* solutions at 300 K using a Bruker Ultrashield 300 MHz instrument. Chemical shifts are reported as parts per million relative to TMS (0.00) for ^{1}H and ^{13}C NMR and $CFCl_3$ for ^{19}F NMR. NMR spectra were processed via Spectrus Processor, ACDLabs. High-resolution mass spectra (HRMS) were obtained using a hybrid quadrupole time-of-flight mass spectrometer (microTOFq II, Bruker Daltonics) in ESI^+ mode. Silica gel chromatographies were performed on an ISCO Combiflash Companion Instruments using ISCO RediSep® Flash Cartridges (particle size: 35-70 microns) or Silacycle SiliaSep® Flash Cartridges (particle size: 40-63 microns). All compounds tested possessed a purity of ≥ 95%. When not indicated, compound intermediates and reagents were purchased from chemical supply houses. Compounds were determined to be greater than 95% pure via analysis by reversed phase UPLC-MS using a Waters Acquity UPLC instrument with DAD and ELSD and a UPLC HSS T3, 2.1 x 30 mm, 1.8 um column and a gradient of 2 to 98% CH_3CN in water with 0.1% formic acid over 2.0 min at 1 mL/min. Injection volume was 1 µL and the column temperature was 30 °C. Detection was based on electrospray ionization (ESI) in positive and negative polarity using Waters ZQ mass spectrometer (Milford, MA, USA), diode-array UV detector from 210 to 400 nm, and evaporative light scattering detector (Sedex 75, Sedere, Alfortville Cedex, France). HPLC purification of spirohydantoins were performed using Gilson preparative HPLC with 333/334 pumps, 215 autosampler, and 156 UV/VIS running Trilution 3.0 software using Waters Atlantis T3 19x100mm 5µm column.

2. General procedure for the synthesis of 2- dialkylamino benzaldehydes *via* S_nAr displacement

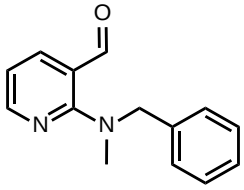
To a 250-mL round bottom flask equipped with magnetic stirrer and reflux condenser were added 2-fluorobenzaldehyde (1 equiv), amine (1-5 equiv) and K_2CO_3 (1.2-2 equiv). The mixture was suspended in DMF and the resulting mixture was heated to reflux until all the aldehyde has been consumed as determined by UPLC-MS. The reaction was then cooled to rt and quenched with water and extracted with EtOAc 3x. The combined organic layers were washed with brine, dried over $MgSO_4$, filtered, and concentrated *in vacuo*. The crude material

was often pure enough and was used without further purification. Otherwise, pure 2-dialkylaminobenzaldehydes can be obtained *via* purification using automated flash chromatography.

Representative Aldehyde Examples:

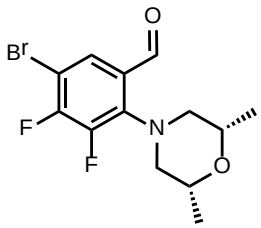


2-(Benzyl(methyl)amino)benzaldehyde. To a 500-mL round bottom flask equipped with magnetic stirrer and reflux condenser were added 2-fluorobenzaldehyde (10.0 g, 80.57 mmol), *N*-methyl-1-phenylmethanamine (11.7 g, 96.69 mmol) and K_2CO_3 (13.5 g, 96.69 mmol). The mixture was suspended in 100 mL DMF and the resulting mixture was heated to reflux until all the aldehyde had been consumed. After 6 h, the reaction was then cooled to rt and quenched with water and extracted with EtOAc 3x. The combined organic layers were washed with brine, dried over $MgSO_4$, filtered, and concentrated *in vacuo*. The crude material was eluted through a short silica plug with CH_2Cl_2 to afford the pure product, 2-(benzyl(methyl)amino)benzaldehyde (18.1 g, >99% yield) as yellow oil. ^{1}H NMR(400 MHz, $CDCl_3$) δ 10.43 (s, 1H), 7.84 (dd, *J*=9.0, 3.0 Hz, 1H), 7.50 (t, J=9.0Hz), 7.28-7.35 (m, 5H), 7.12 (t, 2H), 4.36 (s, 2H), 2.84 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 191.2, 155.7, 137.4, 134.7, 130.2, 128.6, 128.0, 127.5, 121.7, 119.6, 62.4, 42.4; HRMS (ES) MH^+ calcd for $C_{15}H_{16}NO$ 225.1154, found 225.1157.



2-(Benzyl(methyl)amino)nicotinaldehyde. To a 100-mL round bottom flask equipped with magnetic stirrer and reflux condenser were added 2-fluoronicotinalldehyde (0.969 g, 7.99

mmol), *N*-methyl-1-phenylmethanamine (1.00 g, 7.99 mmol) and K_2CO_3 (2.21 g, 15.99 mmol). The mixture was suspended in 20 mL DMF and the resulting mixture was heated to reflux until all the aldehyde has been consumed. After 6 h, the reaction was then cooled to rt and quenched with water and extracted with EtOAc 3x. The combined organic layers were washed with brine, dried over $MgSO_4$, filtered, and concentrated *in vacuo*. The material was pure enough and was used for next step without further purification affording 2-(benzyl(methyl)amino)nicotinaldehyde (1.81 g, 97%) as yellow oil. ^{1}H NMR (500 MHz, $CDCl_3$) δ 10.03 (s, 1H), 8.38 (d, *J*=5.0 Hz, 1H), 8.02 (d, *J*=5 Hz, 1H), 7.29-7.37 (m, 5H), 6.85 (d, *J*=5.0 Hz, 1H), 4.85 (s, 2H), 3.04 (s, 3H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 189.6, 160.7, 152.4, 141.3, 137.7, 128.7, 127.6, 127.3, 117.5, 114.2, 59.9, 40.5; HRMS (ES) MH^+ calcd for $C_{14}H_{15}N_2O$ 227.1140, found 227.1147.

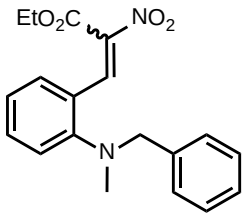


5-Bromo-2-((2S,6R)-2,6-dimethylmorpholino)-3,4-difluorobenzaldehyde, 50. To a 250-mL round bottom flask equipped with magnetic stirrer and reflux condenser were added 5-bromo-2,3,4-trifluorobenzaldehyde (1.00 g, 4.18 mmol), cis-2,6-dimethylmorpholine (0.497 g, 4.18 mmol) and *N*,*N*-diisopropylethylamine (0.877 mL, 5.02 mmol). The mixture was suspended in 20 mL CH_3CN, and the resulting mixture was heated to reflux until all the aldehyde has been consumed. After 5 h, the reaction was then cooled to rt and quenched with water and extracted with EtOAc 3x. The combined organic layers were washed with brine, dried over $MgSO_4$, filtered, and concentrated *in vacuo*. The crude material was purified using ISCO (isocratic, 5% hexane/EtOAc) to afford the product 5-bromo-2-((2R,6S)-2,6-dimethylmorpholino)-3,4-difluorobenzaldehyde, **50** (1.00 g, 72% yield) as yellow solid. ^{1}H NMR (300 MHz, $CDCl_3$) δ 10.3 (s, 1H), 7.83 (dd, J_{FH}=9.0, 3.0 Hz, 1H), 3.84 (m, 2H), 3.04 (m, 4H), 1.23 (d, *J*=9.0 Hz, 6H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 189.6, 154.3 (dd, J_{CF}=257.3, 14.3 Hz), 150.3 (dd, J_{CF}=255.0, 13.5 Hz) 142.4, 128.8, 128.1, 105.8, 105.7, 72.2, 58.2, 18.7; ^{19}F NMR (282 MHz, $CDCl_3$) δ -119.0, -141.3; HRMS (ES) MH^+ calcd for $C_{13}H_{15}BrF_2NO_2$ 334.0210, found 334.0700.

3. General procedure for the Knoevenagel condensation between 2-dialkylaminobenzaldehydes and ethyl nitroacetate[1]

A mixture of ethyl nitroacetate (1 equiv) and 2-dialkylamino benzaldehyde (1 equiv) in a 250-mL round bottom flask was dissolved in 20 mL anhydrous THF. The resulting solution was cooled to 0 ˚C using an ice/water bath followed by careful addition of $TiCl_4$ solution (1M in DCM or toluene, 2 equiv) *via* syringe. After 5 min of stirring, 4-methylmorpholine (4 equiv) was added to the brownish mixture dropwise over 10 min. The reaction was then stirred for additional 4 h; then water was added to quench the reaction. The mixture was extracted with EtOAc 3x, and the combined organic layers were washed with brine, dried over Na_2SO_4, filtered and concentrated *in vacuo*. The crude material was purified using ISCO automated flash chromatography with hexane/EtOAc as solvent system.

Representative Example: Synthesis of ethyl 3-(2-(benzyl(methyl)amino)phenyl)-2-nitroacrylate, 4



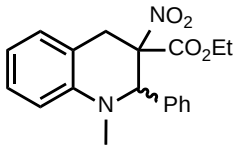
Ethyl 3-(2-(benzyl(methyl)amino)phenyl)-2-nitroacrylate, 4. A mixture of ethyl nitroacetate (0.591 g, 4.44 mmol) and 2-(benzyl(methyl)amino)benzaldehyde (1.00 g, 4.44 mmol) in a 250-mL round bottom flask was dissolved in 20 mL anhydrous THF. The resulting solution was cooled to 0 ºC using an ice/water bath followed by careful addition of $TiCl_4$ solution (1M in DCM) *via* syringe (8.9 mL, 8.88 mmol). After 5 min of stirring, 4-methylmorpholine (1.95 mL, 17.7 mmol) was added to the brownish mixture dropwise over 10 min. The reaction was then stirred for additional 4 h; then water was added to quench the reaction. The mixture was extracted with EtOAc 3x, and the combined organic layers were washed with brine, dried over Na_2SO_4, filtered, and concentrated *in vacuo*. The crude material was purified using ISCO automated flash chromatography with hexane/EtOAc as solvent system to afford the product **4** (1.10 g, 73% yield) as orange oil and as a mixture of *E/Z* isomers

(~1.4:1). The material was used for next step as a mixture of regioisomers. ^{1}H NMR (500 MHz, $CDCl_3$) δ ppm 8.57 (s, 1 H) 8.12 (s, 1 H) 7.25 - 7.45 (m, 21 H) 7.05 - 7.13 (m, 5 H) 4.32 - 4.43 (m, 5 H) 4.18 (s, 5 H) 2.76 (s, 7 H) 1.28 - 1.40 (m, 8 H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 189.6, 142.4, 128.8, 128.1, 105.8, 105.7, 72.2, 58.2, 18.7; HRMS (ES) MH^-calcd for $C_{19}H_{19}N_2O_4$ 339.1350, found 339.2305.

4. Chemoselective study for the T-reaction: General procedure for the $Mg(OTf)_2$-catalyzed T-reaction.

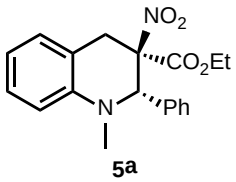
A mixture of Knoevenagel adduct (mixture of *E*/*Z* isomers) and $Mg(OTf)_2$ was weighed in a 25-ml one-necked round bottom flask equipped with magnetic stirrer. The mixture was dissolved with 10 mL CH_3CN and the resulting orange solution was heated to reflux until all the starting material has been converted to the T-reaction products. When the conversion was completed (as monitored by UPLC-MS), the reaction was cooled to rt and then concentrated *in vacuo*. Diastereomeric ratio and regioisomeric ratio were determined using ^{1}H NMR analysis of the crude mixture.

Representative Example: Synthesis of ethyl 1-methyl-3-nitro-2-phenyl-1,2,3,4-tetrahydroquinoline-3-carboxylate, 5



A mixture of Knoevenagel adduct above (0.50 g, 1.47 mmol) and $Mg(OTf)_2$ (0.047 g, 0.15 mmol) was weighed in a 25-ml one-necked round bottom flask equipped with magnetic stirrer. The mixture was dissolved with 10 mL CH_3CN and the resulting orange solution was heated to reflux until all the starting material has been converted to the T-reaction products as monitored by UPLC-MS. After ~0.5 h, the reaction was cooled to rt then concentrated *in vacuo*. The adduct was used for next step as a mixture of *E/Z* isomers. Diastereomeric ratio and regioisomeric ratio were determined using ^{1}H NMR analysis of the crude mixture. Pure tetrahydroquinoline-nitroester product was obtained after purification using ISCO automated flash chromatography using 20% EtOAc in hexanes as solvent system to afford the product

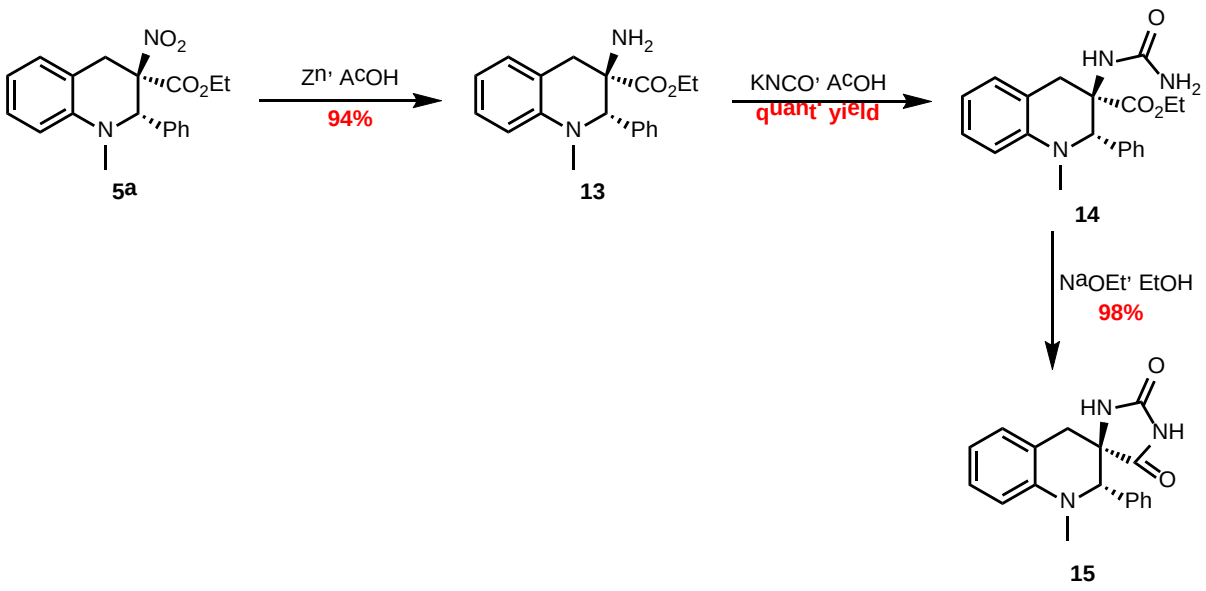
(0.492 g, 98%) as yellow oil. The oil was re-dissolved in 1:1 hexane/EtOAc and the major diastereomer crystallized quickly to afford the pure tetrahydroquinoline nitroester **5a** (57% yield after recrystallization). The structure of **5a** was confirmed by X-ray crystallographic analysis.



5a

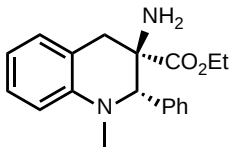
(2S,3R)-ethyl 1-methyl-3-nitro-2-phenyl-1,2,3,4-tetrahydroquinoline-3-carboxylate, 5a. ^{1}H NMR (400 MHz, $CDCl_3$) δ ppm 7.19 - 7.36 (m, 6 H) 7.08 (d, *J*=7.5 Hz, 1 H) 6.72 (t, *J*=7.2 Hz, 1 H) 6.66 (d, *J*=8.3 Hz, 1 H) 5.41 (d, *J*=2.0 Hz, 1 H) 4.11 (qd, *J*=7.1, 1.0 Hz, 2 H) 3.70 (d, *J*=17.3 Hz, 1 H) 3.59 (d, *J*=17.3 Hz, 1 H) 2.96 (s, 3 H) 1.13 - 1.23 (m, 3 H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 164.5, 143.4, 137.7, 129.2, 128.9, 128.8, 127.8, 116.8, 114.8, 109.9, 92.9, 65.8, 63.2, 37.3, 30.4, 13.6; HRMS (ES) M-H calcd for $C_{19}H_{19}N_2O_4$ 339.1350, found 339.1379.

5. Procedure for the synthesis of spirohydantoin (2'S,3'R)-1'-methyl-2'-phenyl-2',4'-dihydro-1'H-spiro[imidazolidine-4,3'-quinoline]-2,5-dione, 15



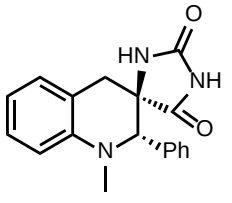
To a 25 mL round bottom flask equipped with a magnetic stirrer were added **5a** (0.1 g, 0.29 mmol) and zinc dust (0.096g, 1.47 mmol). The mixture was suspended in acetic acid (5 mL)

and the resulting mixture was stirred at rt. After 2 h, the reaction was filtered through celite and concentrated *in vacuo* to afford the amine derivative **13**, which was re-dissolved in acetic acid (5 mL). The solution was charged with potassium cyanate (0.024 g, 0.29 mmol) and the reaction was stirred overnight at rt. The reaction was then quenched with water and extracted with EtOAc 3x. The combined organic layers were washed with brine, dried over $MgSO_4$, filtered and concentrated *in vacuo* to afford the crude urea **14**. The urea was then transferred to a 25 mL round bottom flask equipped with magnetic stirrer, then dissolved in absolute EtOH. The solution was treated with NaOEt in EtOH and, after 30 min, quenched with saturated aqueous NH_4Cl solution and then extracted with EtOAc 3x. The combined organic layers were washed with brine, dried over $MgSO_4$, filtered and then concentrated *in vacuo* to afford the spirohydantoin **15** (0.085g, 98% yield) as an off-white solid.



13

(2S,3R)-ethyl 3-amino-1-methyl-2-phenyl-1,2,3,4-tetrahydroquinoline-3-carboxylate, 13. ^{1}H NMR (500 MHz, $CDCl_3$) δ ppm 7.61 - 7.83 (m, 2 H) 7.23 - 7.34 (m, 3 H) 7.07 - 7.18 (m, 2 H) 6.98 (d, *J*=7.6 Hz, 2 H) 6.76 (d, *J*=8.2 Hz, 1 H) 6.64 (t, *J*=7.3 Hz, 1 H) 4.74 (s, 1 H) 4.02 (dq, *J*=10.7, 7.1 Hz, 1 H) 3.89 (dq, *J*=10.7, 7.1 Hz, 1 H) 3.48 (d, *J*=17.7 Hz, 1 H) 3.10 (d, *J*=17.7 Hz, 1 H) 2.93 (s, 3 H) 0.98 (t, *J*=7.3 Hz, 3 H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 166.2, 141.2, 128.6, 127.3, 127.1, 126.9, 125.1, 115.9, 112.8, 109.1, 64.9, 61.3, 58.3, 35.4, 29.1, 11.4; HRMS (ES) MH^+ calcd for $C_{19}H_{23}N_2O_2$ 311.1754, found 311.1752.



15

(2'S,3'R)-1'-methyl-2'-phenyl-2',4'-dihydro-1'H-spiro[imidazolidine-4,3'-quinoline]-2,5-dione, 15. ^{1}H NMR (400 MHz, DMSO-d_6) δ ppm 10.57 (s, 1 H) 8.20 (s, 1 H) 7.21 - 7.29 (m, 3 H) 7.12 (t, *J*=7.7 Hz, 1 H) 6.98 - 7.08 (m, 3 H) 6.69 (d, *J*=8.5 Hz, 1 H) 6.62 - 6.67 (m, 1 H) 4.43 (s, 1 H) 3.05 (d, *J*=16.3 Hz, 1 H) 2.77 (s, 3 H) 2.75 (d, *J*=16.3 Hz, 1 H); ^{13}C NMR (100

MHz, $CDCl_3$) δ 173.1, 154.9, 143.7, 137.0, 127.8, 126.8, 126.6, 116.9, 115.0, 109.6, 67.7, 60.4, 36.6, 31.7; HRMS (ES) MH^+ calcd for $C_{18}H_{19}N_3O_2$ 308.1394, found 308.1381.

6. General procedure for the synthesis of spirohydantoins from Knoevenagel adducts.

To a 25 mL round bottom flask equipped with magnetic stirrer were added the Knoevenagel adducts and $Mg(OTf)_2$. The mixture was dissolved in CH_3CN and heated to reflux until all the starting material was consumed after which the reaction was cooled to rt then concentrated *in vacuo*. The crude material was then re-dissolved in acetic acid and Zn dust was added. After 1-2 h of stirring at rt, the reaction was filtered thru celite and concentrated *in vacuo* to afford the amine derivatives as a mixture of diastereomers. The diastereomers can either be separated at the amine stage (**Procedure A**) or at the spirohydantoin stage (**Procedure B**) *via* chromatography to afford the pure amines or spirohydantoin products.

Procedure A: To a 25 mL round bottom flask equipped with magnetic stirrer were added the diasteromerically pure amine from above and potassium cyanate (1-2 equiv). The mixture was dissolved in acetic acid and the resulting solution was stirred overnight at rt. The reaction was then concentrated *in vacuo,* re-dissolved in EtOAc and washed with water and brine. The organic layer was separated and dried over $MgSO_4$, filtered and then concentrated *in vacuo* to afford the crude urea derivative. This material was then transferred to a 25 mL round bottom flask, dissolved in EtOH, and then 21% wt NaOEt in EtOH (1-2 equiv) was added. The resulting brown solution was stirred at rt for about 30 min to 1 h until all the urea has been converted to the spirohydantoin derivative (as monitored by UPLC-MS). When the reaction was complete, solution was concentrated *in vacuo,* quenched with saturated aqueous NH_4Cl solution and then extracted with EtOAc 3x. The combined organic layers were washed with brine, dried over $MgSO_4$, filtered, and concentrated in vacuo. The crude residue was purified using chromatography to afford the pure spirohydantoin product as white powdery solid.

Procedure B. To a 25 mL round bottom flask equipped with magnetic stirrer were added the diastereomeric mixture of amine from above and potassium cyanate (1-2 equiv). The mixture was dissolved in acetic acid and the resulting solution was stirred overnight at rt. The reaction was then concentrated in vacuo, then re-dissolved in EtOAc and washed with water and brine. The organic layer was separated and dried over $MgSO_4$, filtered and then concentrated *in vacuo*

to afford the crude urea derivative. This material (diastereomeric mixture of urea derivatives) was then transferred to a 25 mL round bottom flask, dissolved in EtOH, and 21% NaOEt in EtOH (1-2 equiv) was added. The resulting brown solution was stirred at rt for about 30 min to 1 h until all the urea has been converted to the spirohydantoin derivative (as monitored by UPLC-MS). When reaction was complete, the solution was concentrated *in vacuo*, quenched with saturated aqueous NH_4Cl solution and then extracted with EtOAc 3x. The combined organic layers were washed with brine, dried over $MgSO_4$, filtered, and concentrated *in vacuo*. The crude residue was purified using chromatography to afford the two spirohydantoin diastereomers as white powdery solids.

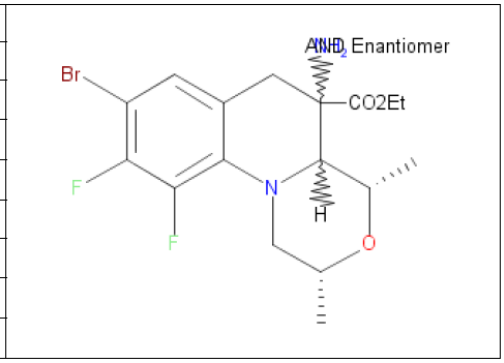
7. Procedure for the synthesis of spirohydantoins 23 and *epi*-C3-23

A mixture of ethyl nitroacetate (0.40 g, 0.35 mL, 2.99 mmol) and **19** (1.0 g, 2.99 mmol) in a 250-mL round bottom flask was dissolved in 20 mL anhydrous THF. The resulting solution was cooled to 0 ^oC using an ice/water bath followed by careful addition of $TiCl_4$ solution (6 mL, 5.99 mmol, 1M in DCM) *via* syringe. After 5 min of stirring, the brownish mixture was treated with 4-methylmorpholine (1.3 mL, 11.9 mmol) by dropwise addition over 10 min. The reaction was stirred for additional 12 h and then water was added to quench the reaction. The mixture was extracted with EtOAc 3x, and the combined organic layers were washed with brine, dried over Na_2SO_4, filtered and then concentrated *in vacuo*. The crude material was purified using ISCO automated flash chromatography with hexane/EtOAc as solvent system to afford **20** (1.14 g, 2.54 mmol, 85% yield) as red orange oil and as mixture of *E/Z* regioisomers.

To a 25 mL round bottom flask equipped with magnetic stirrer were added **20** (1.0 g, 2.23 mmol) and $Mg(OTf)_2$ (0.072 g, 0.22 mmol). The mixture was dissolved in CH_3CN and heated to 120 ^oC until all the starting material was consumed. After 6 h, the reaction was cooled to rt and then concentrated *in vacuo*. The crude material was then re-dissolved in acetic acid and Zn dust (0.437 g, 6.68 mmol) was added. After 3 h of stirring at rt, the reaction was filtered thru celite and concentrated *in vacuo*. The crude was purified using ISCO automated flash chromatography using DCM/EtOAc as solvent system to afford **22** (0.914 g, 98% yield) as a mixture of diastereomers. The diastereomers were separated using Xbridge C18 column (19 mm x 150 mm 5 µm) mobile phase A: H_2O with 0.2% NH_4OH, mobile phase B: CH_3CN, gradient 50-80% B over 5 min. The latter material was contaminated with presumably a third diastereomer and purified final material was obtained in the subsequent step.

Purification conditions (Achiral)

Column Info: Xbridge C18	19mm x 150mm 5µm
Mobile Phase A	**H2O with 0.2 % NH4OH pH10**
Mobile Phase B	**Acetonitrile**
Gradient (%B)	50-80%B over 5 min.
Flow Rate	20 ml/min
Concentration	mg/mL in _____
Loading (mg/injection)	
Column Temperature	room temp
Outlet Pressure (SFC)	N/A



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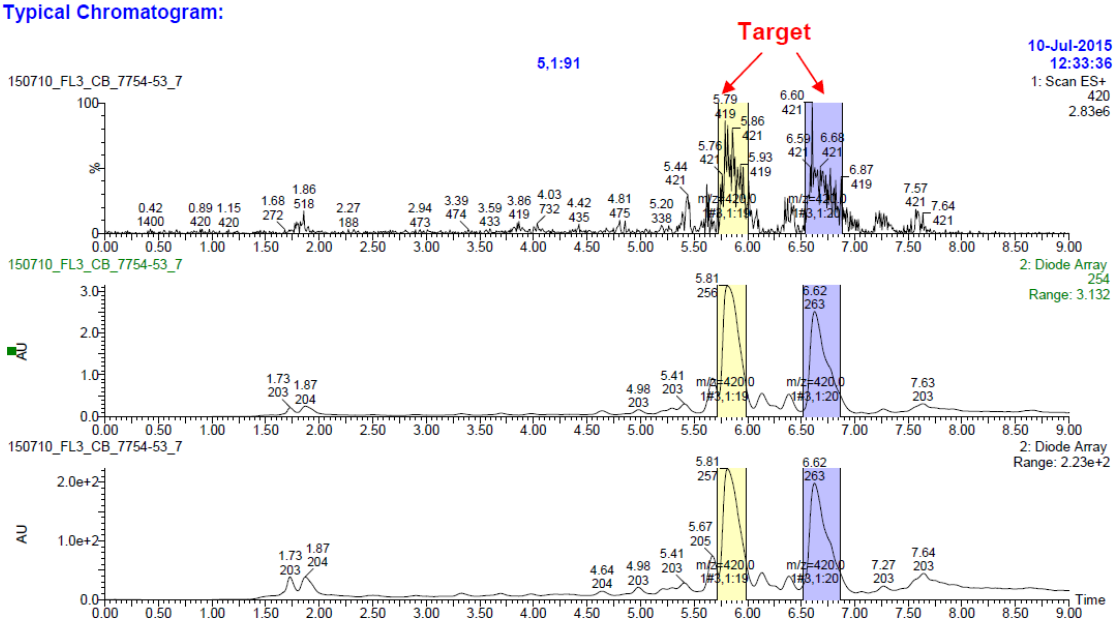
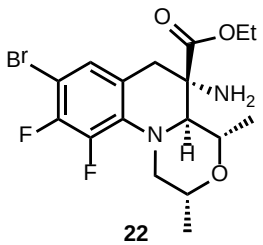
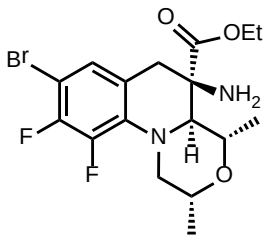




22

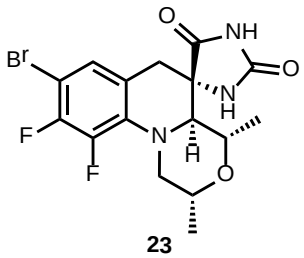
***rac*-(2R,4S,4aS,5S)-Ethyl 5-amino-8-bromo-9,10-difluoro-2,4-dimethyl-1,2,4,4a,5,6-hexahydro-[1,4]oxazino[4,3-a]quinoline-5-carboxylate, 22**. ^{1}H NMR (300 MHz, CD_3OD) δ ppm 7.05 (d, *J*=7.2 Hz, 1 H) 4.79 (s, 3 H) 4.07 - 4.34 (m, 3 H) 3.53 - 3.82 (m, 2 H) 3.14 - 3.40 (m, 2 H) 2.87 - 3.09 (m, 2 H) 2.67 - 2.86 (m, 1 H) 1.24 (t, *J*=7.2 Hz, 3 H) 1.12 (t, *J*=6.8 Hz, 6 H); ^{13}C NMR (75 MHz, CD_3OD) δ 174.3, 151.1 (dd, *Jcf*=240.0, 15.0 Hz), 144.0 (dd, *Jcf*=244.5, 16.5 Hz), 133.7, 128.5, 122.1, 97.4 (d, *Jcf*=18.5 Hz), 74.5, 72.3, 70.3, 62.7, 58.3, 57.5, 36.3,

18.5, 18.4, 14.2; ^{19}F NMR (282 MHz, CD_3OD) δ -134.9, -148.5; HRMS (ES) MH^+ calcd for $C_{17}H_{22}BrF_2N_2O_3$ 419.0737, found 419.0741.



***epi*-C3-22**

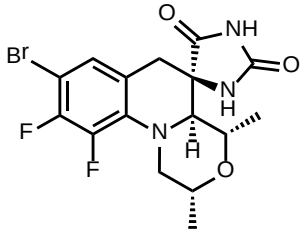
***rac*-(2R,4S,4aS,5R)-Ethyl 5-amino-8-bromo-9,10-difluoro-2,4-dimethyl-1,2,4,4a,5,6-hexahydro-[1,4]oxazino[4,3-a]quinoline-5-carboxylate, *epi*-C3-22**. ^{1}H NMR (300 MHz, METHANOL-d_4) δ ppm 6.97 (d, *J*=7.0 Hz, 1 H) 4.15 - 4.35 (m, 3 H) 4.03 (ddd, *J*=13.0, 2.1, 1.0 Hz, 1 H) 3.84 (ddd, *J*=10.2, 6.2, 2.3 Hz, 1 H) 3.59 - 3.74 (m, 1 H) 3.47 (d, *J*=8.7 Hz, 1 H) 3.20 - 3.39 (m, 1 H) 3.08 (d, *J*=14.7 Hz, 1 H) 2.78 - 2.99 (m, 1 H) 2.58 (d, *J*=14.7, 1 H) 1.35 (t, *J*=9 Hz, 3 H), 1.18 (d, *J*=6 Hz, 3H), 1.13 (d, *J*=6 Hz, 3H); ^{13}C NMR (75 MHz, CD_3OD) δ 176.2, 151.3 (dd, *J*cf=241.5, 15.0 Hz), 143.8 (dd, *J*cf=243.8, 15.8 Hz), 134.4, 127.9, 121.9, 97.1 (d, *J*cf=18.8 Hz), 75.5, 73.3, 68.1, 63.3, 59.5, 57.9, 41.6, 18.7, 18.6, 14.4; ^{19}F NMR (282 MHz, CD_3OD) δ -134.8, -149.5; HRMS (ES) MH^+ calcd for $C_{17}H_{22}BrF_2N_2O_3$ 419.0737, found 419.0772.



23

To a 25 mL round bottom flask equipped with magnetic stirrer were added **22** (0.1 g, 0.24 mmol) and potassium cyanate (0.023 g, 0.29 mmol). The mixture was dissolved in acetic acid (10 mL) and the resulting solution was stirred overnight at rt. The reaction was concentrated *in vacuo*, then re-dissolved in EtOAc and washed with water and brine. The organic layer was separated and dried over $MgSO_4$, filtered, and concentrated *in vacuo* to afford the crude urea derivative. This material was then transferred to a 25 mL round bottom flask, dissolved in 10

mL EtOH, and then 21% NaOEt in EtOH (0.30 mL, 0.29 mmol) was added. The resulting brown solution was stirred at rt for about 30 min to 1 h until all the urea has been converted to the spirohydantoin derivative (as monitored by UPLC-MS). When the reaction was complete, solution was concentrated *in vacuo*, quenched with saturated aqueous NH_4Cl solution and then extracted with EtOAc 3x. The combined organic layers were washed with brine, dried over $MgSO_4$, filtered, and concentrated *in vacuo*. The crude residue was purified using chromatography to afford the pure spirohydantoin product **23** (0.092 g, 93% yield) as white powdery solid. ^{1}H NMR (500 MHz, DMSO-d_6) δ ppm 10.87 (s, 1 H) 8.37 (s, 1 H) 7.12 (d, *J*=6.9 Hz, 1 H) 3.88 (d, *J*=13.2 Hz, 1 H) 3.65 - 3.77 (m, 1 H) 3.43 - 3.53 (m, 1 H) 3.10 (d, *J*=9.1 Hz, 1 H) 2.85 - 2.97 (m, 3 H) 1.18 (t, *J*=7.1 Hz, 1 H) 0.88 (d, *J*=5 Hz, 3 H) 0.85 (d, *J*=5 Hz, 3 H); ^{13}C NMR (125 MHz, DMSO-d6) δ 173.4, 155.6, 148.2 (dd, *J*cf=243.8, 15.0 Hz), 140.7 (dd, *J*cf=242.5, 15.0 Hz), 133.1, 125.5, 121.9, 97.9 (d, *J*cf=18.8 Hz), 72.6, 71.2, 66.3, 62.6, 55.9, 37.2, 18.3, 18.1; ^{19}F NMR (470 MHz, DMSO-d6) δ -73.43; HRMS (ES) MH^- calcd for $C_{16}H_{15}BrF_2N_3O_3$ 414.0270, found 414.0267.



***epi*-C3-23**

To a 25 mL round bottom flask equipped with magnetic stirrer were added ***epi*-C3-22** (0.1 g, 0.24 mmol) and potassium cyanate (0.023 g, 0.29 mmol). The mixture was dissolved in acetic acid (10 mL) and the resulting solution was stirred overnight at rt. The reaction was concentrated *in vacuo*, re-dissolved in EtOAc and washed with water and brine. The organic layer was separated and dried over $MgSO_4$, filtered, and concentrated *in vacuo* to afford the crude urea derivative. This material was then transferred to a 25 mL round bottom flask, dissolved in 10 mL EtOH, and 21% NaOEt in EtOH (0.30 mL, 0.29 mmol) was added. The resulting brown solution was stirred at rt for about 30 min to 1 h until all the urea has been converted to the spirohydantoin derivative (as monitored by UPLC-MS). When the reaction was complete, solution was concentrated *in vacuo*, quenched with saturated aqueous NH_4Cl solution and then extracted with EtOAc 3x. The combined organic layers were washed with brine, dried over $MgSO_4$, filtered, and concentrated *in vacuo*. The crude residue was purified

using chromatography to afford the pure spirohydantoin product **23** (0.094 g, 95% yield) as white powdery solid. ^{1}H NMR (500 MHz, DMSO-d_6) δ ppm 10.93 (s, 1 H) 8.05 (s, 1 H) 7.05 (d, *J*=6.9 Hz, 1 H) 3.74 (d, *J*=13.6 Hz, 1 H) 3.50 - 3.59 (m, 1 H) 3.22 - 3.32 (m, 1 H) 3.16 - 3.21 (m, 1 H) 2.75 - 2.85 (m, 1 H) 2.57 - 2.69 (m, 2 H) 2.35 - 2.41 (m, 1 H) 0.96 (d, *J*=6.0 Hz, 3 H) 0.80 (d, *J*=6.3 Hz, 3 H); ^{13}C NMR (125 MHz, DMSO-d6) δ 175.7, 156.1, 148.6 (dd, *J*cf=238.8, 13.8 Hz), 141.4 (dd, *J*cf=243.8, 17.5 Hz), 133.1, 126.3, 121.6, 94.9 (d, *J*cf=18.8 Hz), 72.8, 71.2, 64.9, 63.2, 55.5, 37.5, 18.1, 17.8; ^{19}F NMR (470 MHz, DMSO-d6) δ -73.40; HRMS (ES) MH^- calcd for $C_{16}H_{15}BrF_2N_3O_3$ 414.0270, found 414.0279.

8. References

[1] Wortman, L.; Koppitz, M.; Menzenbach, M.; Kosemund, D.; Schmees, N.; Muhn, H.-P.; Frenzel, T.; Liesener, F. P.; Schrey, A. K.; Kuehne, R.; PCT Int. Appl. (2009), WO 2009013354.

9. X-ray crystallography data for 5a

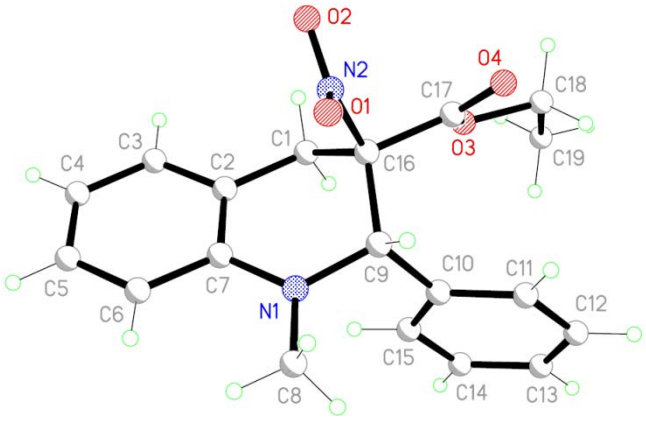


Table 1. Crystal data and structure refinement for **5a**.

Identification code	**5a**
Empirical formula	C19 H20 N2 O4
Formula weight	340.37
Temperature	296(2) K
Wavelength	0.71073 Å
Crystal system	Monoclinic
Space group	P2(1)/n

Unit cell dimensions	a = 7.7910(4) Å	□= 90°.
	b = 13.1164(6) Å	□= 96.8950(10)°.
	c = 16.9526(8) Å	□ = 90°.
Volume	1719.85(14) Å^3	
Z	4	
Density (calculated)	1.315 Mg/m^3	
Absorption coefficient	0.093 mm^{-1}	
F(000)	720	
Crystal size	0.28 x 0.16 x 0.10 mm^3	
Theta range for data collection	1.97 to 30.57°.	
Index ranges	-11<=h<=11, -18<=k<=18, -24<=l<=24	
Reflections collected	27526	
Independent reflections	5271 [R(int) = 0.0306]	
Completeness to theta = 30.57°	99.9 %	
Absorption correction	None	
Refinement method	Full-matrix least-squares on F^2	
Data / restraints / parameters	5271 / 0 / 226	
Goodness-of-fit on F^2	1.048	
Final R indices [I>2sigma(I)]	R1 = 0.0515, wR2 = 0.1438	
R indices (all data)	R1 = 0.0798, wR2 = 0.1659	
Largest diff. peak and hole	0.357 and -0.276 e.Å^{-3}	

Table 2. Atomic coordinates (x 10^4) and equivalent isotropic displacement parameters ($Å^2$x 10^3) for **5a**. U(eq) is defined as one third of the trace of the orthogonalized U^{ij} tensor.

	x	y	z	U(eq)
O(1)	4407(2)	6984(1)	2379(1)	72(1)
O(2)	6449(2)	6918(2)	1647(1)	95(1)
O(3)	3372(2)	6646(1)	-243(1)	55(1)
O(4)	3292(2)	5624(1)	813(1)	72(1)
N(1)	2192(2)	8778(1)	1659(1)	45(1)
N(2)	4966(2)	7087(1)	1749(1)	51(1)
C(1)	4638(2)	8275(1)	609(1)	41(1)
C(2)	4881(2)	9185(1)	1147(1)	39(1)
C(3)	6307(2)	9817(1)	1146(1)	49(1)
C(4)	6539(2)	10659(1)	1636(1)	58(1)
C(5)	5335(2)	10856(1)	2145(1)	56(1)
C(6)	3912(2)	10238(1)	2171(1)	48(1)
C(7)	3647(2)	9389(1)	1666(1)	38(1)
C(8)	937(2)	8967(2)	2206(1)	60(1)
C(9)	2019(2)	7783(1)	1271(1)	38(1)
C(10)	601(2)	7773(1)	570(1)	38(1)
C(11)	-437(2)	6917(1)	425(1)	49(1)
C(12)	-1676(2)	6874(2)	-234(1)	60(1)
C(13)	-1902(2)	7688(2)	-748(1)	62(1)
C(14)	-903(3)	8547(2)	-602(1)	58(1)
C(15)	338(2)	8594(1)	54(1)	47(1)
C(16)	3752(2)	7425(1)	1011(1)	38(1)
C(17)	3463(2)	6447(1)	520(1)	47(1)
C(18)	2853(3)	5819(2)	-799(1)	73(1)
C(19)	2348(4)	6301(2)	-1579(1)	91(1)

Table 3. Bond lengths [Å] and angles [°] for **5a**.

O(1)-N(2)	1.2074(19)
O(2)-N(2)	1.210(2)
O(3)-C(17)	1.312(2)
O(3)-C(18)	1.462(2)
O(4)-C(17)	1.203(2)
N(1)-C(7)	1.3865(19)
N(1)-C(8)	1.447(2)
N(1)-C(9)	1.4604(19)
N(2)-C(16)	1.5405(19)
C(1)-C(2)	1.500(2)
C(1)-C(16)	1.516(2)
C(2)-C(3)	1.387(2)
C(2)-C(7)	1.404(2)
C(3)-C(4)	1.381(3)
C(4)-C(5)	1.373(3)
C(5)-C(6)	1.378(3)
C(6)-C(7)	1.405(2)
C(9)-C(10)	1.522(2)
C(9)-C(16)	1.543(2)
C(10)-C(15)	1.388(2)
C(10)-C(11)	1.388(2)
C(11)-C(12)	1.388(2)
C(12)-C(13)	1.376(3)
C(13)-C(14)	1.375(3)
C(14)-C(15)	1.384(2)
C(16)-C(17)	1.531(2)
C(18)-C(19)	1.475(4)

C(17)-O(3)-C(18)	117.81(15)
C(7)-N(1)-C(8)	120.66(13)
C(7)-N(1)-C(9)	123.48(12)
C(8)-N(1)-C(9)	114.09(13)
O(1)-N(2)-O(2)	123.89(15)
O(1)-N(2)-C(16)	119.95(14)
O(2)-N(2)-C(16)	116.14(14)
C(2)-C(1)-C(16)	110.04(12)

C(3)-C(2)-C(7)	119.71(14)
C(3)-C(2)-C(1)	121.17(14)
C(7)-C(2)-C(1)	119.12(13)
C(4)-C(3)-C(2)	121.64(16)
C(5)-C(4)-C(3)	118.61(16)
C(4)-C(5)-C(6)	121.44(16)
C(5)-C(6)-C(7)	120.48(16)
N(1)-C(7)-C(2)	120.31(13)
N(1)-C(7)-C(6)	121.56(14)
C(2)-C(7)-C(6)	118.11(14)
N(1)-C(9)-C(10)	112.25(12)
N(1)-C(9)-C(16)	111.33(12)
C(10)-C(9)-C(16)	110.65(11)
C(15)-C(10)-C(11)	118.52(14)
C(15)-C(10)-C(9)	121.60(13)
C(11)-C(10)-C(9)	119.86(13)
C(12)-C(11)-C(10)	120.65(16)
C(13)-C(12)-C(11)	120.12(17)
C(14)-C(13)-C(12)	119.73(16)
C(13)-C(14)-C(15)	120.43(17)
C(14)-C(15)-C(10)	120.53(16)
C(1)-C(16)-C(17)	114.64(12)
C(1)-C(16)-N(2)	107.97(12)
C(17)-C(16)-N(2)	103.73(12)
C(1)-C(16)-C(9)	111.44(12)
C(17)-C(16)-C(9)	109.50(12)
N(2)-C(16)-C(9)	109.18(11)
O(4)-C(17)-O(3)	126.13(16)
O(4)-C(17)-C(16)	122.95(15)
O(3)-C(17)-C(16)	110.88(13)
O(3)-C(18)-C(19)	106.51(18)

Symmetry transformations used to generate equivalent atoms:

Table 4. Anisotropic displacement parameters ($Å^2 x\ 10^3$) for **5a**. The anisotropic displacement factor exponent takes the form: $-2\pi^2 [h^2 a^{*2} U^{11} + ... + 2 h k a^* b^* U^{12}]$

	U^{11}	U^{22}	U^{33}	U^{23}	U^{13}	U^{12}
O(1)	70(1)	101(1)	44(1)	20(1)	-2(1)	6(1)
O(2)	54(1)	148(2)	81(1)	33(1)	4(1)	42(1)
O(3)	72(1)	45(1)	47(1)	-12(1)	2(1)	2(1)
O(4)	104(1)	35(1)	75(1)	4(1)	-1(1)	5(1)
N(1)	43(1)	48(1)	47(1)	-12(1)	12(1)	-3(1)
N(2)	50(1)	53(1)	47(1)	8(1)	-3(1)	7(1)
C(1)	42(1)	42(1)	37(1)	-2(1)	8(1)	0(1)
C(2)	41(1)	38(1)	37(1)	2(1)	1(1)	1(1)
C(3)	46(1)	50(1)	52(1)	3(1)	6(1)	-5(1)
C(4)	54(1)	48(1)	69(1)	-1(1)	-2(1)	-11(1)
C(5)	58(1)	44(1)	61(1)	-13(1)	-8(1)	0(1)
C(6)	49(1)	46(1)	46(1)	-9(1)	-3(1)	7(1)
C(7)	38(1)	37(1)	36(1)	1(1)	-2(1)	5(1)
C(8)	54(1)	67(1)	61(1)	-12(1)	22(1)	0(1)
C(9)	39(1)	37(1)	38(1)	2(1)	5(1)	-1(1)
C(10)	37(1)	38(1)	41(1)	1(1)	6(1)	2(1)
C(11)	45(1)	45(1)	57(1)	2(1)	5(1)	-5(1)
C(12)	47(1)	62(1)	69(1)	-12(1)	-1(1)	-8(1)
C(13)	51(1)	78(1)	54(1)	-12(1)	-8(1)	10(1)
C(14)	64(1)	59(1)	51(1)	6(1)	-3(1)	17(1)
C(15)	50(1)	40(1)	51(1)	4(1)	1(1)	5(1)
C(16)	41(1)	36(1)	37(1)	1(1)	0(1)	5(1)
C(17)	50(1)	38(1)	53(1)	-2(1)	0(1)	9(1)
C(18)	94(2)	53(1)	68(1)	-27(1)	-7(1)	7(1)
C(19)	114(2)	101(2)	56(1)	-27(1)	5(1)	-23(2)

Table 5. Hydrogen coordinates (x 10^4) and isotropic displacement parameters ($Å^2$x 10^3) for **5a**.

	x	y	z	U(eq)
H(1A)	5753	8041	481	49
H(1B)	3942	8464	117	49
H(3A)	7128	9670	807	59
H(4A)	7490	11084	1622	69
H(5A)	5482	11420	2480	67
H(6A)	3122	10384	2525	57
H(8A)	1125	9633	2435	89
H(8B)	1062	8463	2619	89
H(8C)	-207	8931	1925	89
H(9A)	1692	7292	1662	46
H(11A)	-301	6368	774	59
H(12A)	-2355	6294	-329	72
H(13A)	-2728	7657	-1191	75
H(14A)	-1062	9100	-947	70
H(15A)	1000	9181	149	57
H(18A)	1887	5445	-630	87
H(18B)	3806	5350	-828	87
H(19A)	1994	5784	-1965	136
H(19B)	3315	6668	-1738	136
H(19C)	1407	6763	-1540	136

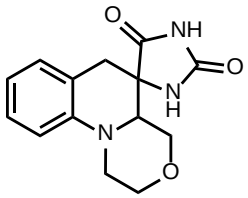
Table 6. Torsion angles [°] for **5a**.

C(16)-C(1)-C(2)-C(3)	146.62(14)
C(16)-C(1)-C(2)-C(7)	-32.65(18)
C(7)-C(2)-C(3)-C(4)	-1.1(2)
C(1)-C(2)-C(3)-C(4)	179.67(15)
C(2)-C(3)-C(4)-C(5)	1.3(3)
C(3)-C(4)-C(5)-C(6)	-0.4(3)
C(4)-C(5)-C(6)-C(7)	-0.7(3)
C(8)-N(1)-C(7)-C(2)	178.38(15)
C(9)-N(1)-C(7)-C(2)	14.5(2)
C(8)-N(1)-C(7)-C(6)	-3.3(2)
C(9)-N(1)-C(7)-C(6)	-167.20(14)
C(3)-C(2)-C(7)-N(1)	178.37(14)
C(1)-C(2)-C(7)-N(1)	-2.4(2)
C(3)-C(2)-C(7)-C(6)	0.0(2)
C(1)-C(2)-C(7)-C(6)	179.25(13)
C(5)-C(6)-C(7)-N(1)	-177.47(15)
C(5)-C(6)-C(7)-C(2)	0.9(2)
C(7)-N(1)-C(9)-C(10)	-114.38(15)
C(8)-N(1)-C(9)-C(10)	80.74(17)
C(7)-N(1)-C(9)-C(16)	10.28(19)
C(8)-N(1)-C(9)-C(16)	-154.59(14)
N(1)-C(9)-C(10)-C(15)	39.36(19)
C(16)-C(9)-C(10)-C(15)	-85.68(17)
N(1)-C(9)-C(10)-C(11)	-142.22(14)
C(16)-C(9)-C(10)-C(11)	92.74(16)
C(15)-C(10)-C(11)-C(12)	1.8(2)
C(9)-C(10)-C(11)-C(12)	-176.65(15)
C(10)-C(11)-C(12)-C(13)	-0.8(3)
C(11)-C(12)-C(13)-C(14)	-0.4(3)
C(12)-C(13)-C(14)-C(15)	0.5(3)
C(13)-C(14)-C(15)-C(10)	0.6(3)
C(11)-C(10)-C(15)-C(14)	-1.7(2)
C(9)-C(10)-C(15)-C(14)	176.74(15)
C(2)-C(1)-C(16)-C(17)	-179.03(12)
C(2)-C(1)-C(16)-N(2)	-64.00(15)
C(2)-C(1)-C(16)-C(9)	55.90(15)

O(1)-N(2)-C(16)-C(1)	132.59(17)
O(2)-N(2)-C(16)-C(1)	-49.1(2)
O(1)-N(2)-C(16)-C(17)	-105.38(18)
O(2)-N(2)-C(16)-C(17)	73.0(2)
O(1)-N(2)-C(16)-C(9)	11.3(2)
O(2)-N(2)-C(16)-C(9)	-170.38(17)
N(1)-C(9)-C(16)-C(1)	-45.24(16)
C(10)-C(9)-C(16)-C(1)	80.32(14)
N(1)-C(9)-C(16)-C(17)	-173.13(12)
C(10)-C(9)-C(16)-C(17)	-47.57(15)
N(1)-C(9)-C(16)-N(2)	73.94(15)
C(10)-C(9)-C(16)-N(2)	-160.50(12)
C(18)-O(3)-C(17)-O(4)	6.3(3)
C(18)-O(3)-C(17)-C(16)	-171.39(15)
C(1)-C(16)-C(17)-O(4)	156.34(17)
N(2)-C(16)-C(17)-O(4)	38.9(2)
C(9)-C(16)-C(17)-O(4)	-77.6(2)
C(1)-C(16)-C(17)-O(3)	-25.87(18)
N(2)-C(16)-C(17)-O(3)	-143.35(13)
C(9)-C(16)-C(17)-O(3)	100.21(15)
C(17)-O(3)-C(18)-C(19)	163.28(19)

Symmetry transformations used to generate equivalent atoms:

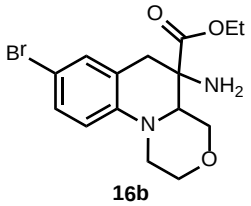
10. Spectral data and full characterization of spirohydantoin products.



17a

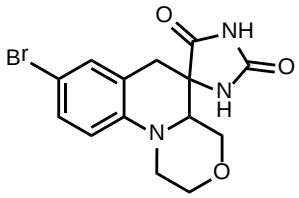
2,4,4a,6-tetrahydro-1H-spiro[[1,4]oxazino[4,3-a]quinoline-5,4'-imidazolidine]-2',5'-dione, 17a. Spirohydantoin **17a** was synthesized *via* **Procedure B**. To a 25 mL round bottom

flask equipped with magnetic stirrer were added the diastereomeric mixture of amine (0.39 g, 1.41 mmol) from above and potassium cyanate (0.137 g, 1.69 mmol). The mixture was dissolved in acetic acid (10 mL) and the resulting solution was stirred overnight at rt. The reaction was then concentrated *in vacuo*, then re-dissolved in EtOAc and washed with water and brine. The organic layer was separated and dried over $MgSO_4$, filtered, and concentrated *in vacuo* to afford the crude urea derivative. This material (diastereomeric mixture of urea derivatives) was then transferred to a 25 mL round bottom flask, dissolved in EtOH, and NaOEt in EtOH (-.6 mL, 1.69 mmol) was added. The resulting brown solution was stirred at rt for about 30 min to 1 h until all the urea has been converted to the spirohydantoin derivative (as monitored by UPLC-MS). When the reaction was complete, solution was concentrated *in vacuo*, quenched with saturated aqueous NH_4Cl solution and then extracted with EtOAc 3x. The combined organic layers were washed with brine, dried over $MgSO_4$, filtered, and concentrated *in vacuo*. The crude residue was purified using chromatography to afford the two spirohydantoin diastereomers as white powdery solids **17a-dia-1** (0.2 g, 52% yield): ^{1}H NMR (400 MHz, DMSO-d_6) δ ppm 10.73 (s, 1 H) 8.04 - 8.11 (m, 1 H) 7.09 (t, *J*=7.4 Hz, 1 H) 6.97 (d, *J*=7.2 Hz, 1 H) 6.89 (d, *J*=8.0 Hz, 1 H) 6.69 (t, *J*=7.4 Hz, 1 H) 3.96 (d, *J*=9.0 Hz, 1 H) 3.78 - 3.92 (m, 2 H) 3.49 (td, *J*=11.7, 2.5 Hz, 1 H) 3.36 (br. s., 2 H) 3.14 - 3.25 (m, 2 H) 2.79 - 3.01 (m, 2 H) 2.83 (td, *J*=12.0, 3.0 Hz, 1 H) ; ^{13}C NMR (100 MHz, DMSO-d_6) δ 174.2, 156.1, 144.4, 129.0, 127.1, 119.7, 118.1, 112.4, 66.1, 65.8, 59.6, 58.9, 46.6, 35.5; HRMS (ES) MH^+ calcd for $C_{14}H_{16}N_3O_3$ 274.1186, found 274.1195. **17a-dia-2** (0.13 g, 34 % yield): ^{1}H NMR (400 MHz, DMSO-d_6) δ ppm 10.91 (s, 1 H) 8.32 (s, 1 H) 7.23 (s, 1 H) 7.06 - 7.18 (m, 1 H) 7.00 (d, *J*=7.4 Hz, 1 H) 6.93 (d, *J*=8.3 Hz, 1 H) 6.74 (t, *J*=7.3 Hz, 1 H) 3.95 (dd, *J*=11.4, 3.4 Hz, 1 H) 3.76 (d, *J*=11.0 Hz, 1 H) 3.65 (d, *J*=8.5 Hz, 1 H) 3.53 (td, *J*=11.7, 2.8 Hz, 1 H) 3.14 - 3.37 (m, 4 H) 2.67 - 2.82 (m, 2 H); ^{13}C NMR (100 MHz, DMSO-d_6) δ 175.6, 156.4, 144.8, 129.6, 127.3, 119.6, 118.5, 112.7, 66.1, 65.8, 60.2, 56.5, 45.6, 37.0; HRMS (ES) MH^+ calcd for $C_{14}H_{16}N_3O_3$ 274.1186, found 274.1189.



16b

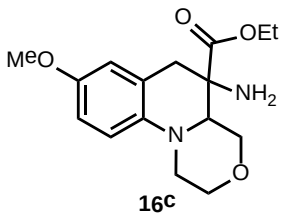
Ethyl 5-amino-8-bromo-1,2,4,4a,5,6-hexahydro-[1,4]oxazino[4,3-a]quinoline-5-carboxylate, 16b. Diastereomers were separated using Atlantis T3 column (19 mm x100 mm 5µm) + Jupiter Proteo (21 mm x100 mm 4µm); mobile phase A: H_2O with 20 mM ammonium acetate pH 8, mobile phase B: CH_3CN, gradient 40-60% B in 20 min. **Diastereomer 1 (16b)**: ^{1}H NMR (400 MHz, Solvent) δ ppm 7.34 (dd, *J*=8.9, 2.1 Hz, 1 H) 7.27 (d, *J*=2.0 Hz, 1 H) 6.89 (d, *J*=9.0 Hz, 1 H) 4.39 (q, *J*=7.2 Hz, 2 H) 3.81 - 4.02 (m, 4 H) 3.45 - 3.67 (m, 3 H) 3.20 - 3.39 (m, 2 H) 1.36 (t, *J*=7.2 Hz, 3 H); ^{13}C NMR (100 MHz, CD_3CO_2D) δ 167.4, 141.6, 132.5, 131.2, 120.8, 115.8, 111.2, 65.1, 64.1, 63.4, 58.5, 48.3, 31.0, 13.1; HRMS (ES) MH^+ calcd for $C_{15}H_{21}BrN_2O_3$ 355.0613, found 355.0645.



17b

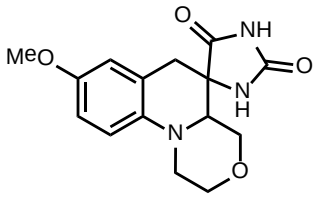
8-Bromo-2,4,4a,6-tetrahydro-1H-spiro[[1,4]oxazino[4,3-a]quinoline-5,4'-imidazolidine]-2',5'-dione, 17b. To a 25 mL round bottom flask equipped with magnetic stirrer were added the diasteromerically pure amine **16b** (0.08 g, 0.23 mmol) from above and potassium cyanate (0.037 g, 0.45 mmol). The mixture was dissolved in acetic acid (5 mL) and the resulting solution was stirred overnight at rt. The reaction was concentrated *in vacuo*, re-dissolved in EtOAc and washed with water and brine. The organic layer was separated, dried over $MgSO_4$, filtered, and concentrated *in vacuo* to afford the crude urea derivative. This material was then transferred to a 25 mL round bottom flask, dissolved in EtOH, and NaOEt in EtOH (0.08 mL, 0.21 mmol) was added. The resulting brown solution was stirred at rt for about 30 min to 1 h until all the urea has been converted to the spirohydantoin derivative (as monitored by UPLC-MS). When the reaction was complete, solution was concentrated *in vacuo*, quenched with saturated aqueous NH_4Cl solution and then extracted with EtOAc 3x. The combined organic layers were washed with brine, dried over $MgSO_4$, filtered, and concentrated *in vacuo*. The crude residue was purified using chromatography (20-75% Water/ CH_3CN /TFA) to afford the pure spirohydantoin product **17b** (0.047 g, 76% yield) as white powdery solid. ^{1}H NMR (400 MHz, DMSO-d_6) δ ppm 10.78 (br 2, 1H), 8.12 (s, 1 H) 7.22 (d, *J*=8.9 Hz, 1 H) 7.16 (s, 1 H) 6.85 (d, *J*=9.0 Hz, 1 H) 3.95 (d, *J*=7.0 Hz, 1 H) 3.76 - 3.92 (m, 2 H) 3.47 (td, *J*=11.7, 2.6 Hz, 1 H) 3.34 (br. s., 3 H) 3.14 - 3.26 (m, 2 H) 2.81 - 3.03 (m, 3 H) 2.06 - 2.13 (m, 1 H); ^{13}C NMR

(100 MHz, DMSO-d_6) δ 174.0, 156.1, 143.7, 131.1, 129.5, 122.4, 114.4, 108.9, 66.0, 65.7, 59.2, 58.7, 46.5, 34.9; HRMS (ES) MH^+ calcd for $C_{14}H_{15}BrN_3O_3$ 352.0291, found 352.0282.



16c

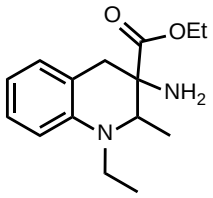
Ethyl 5-amino-8-methoxy-1,2,4,4a,5,6-hexahydro-[1,4]oxazino[4,3-a]quinoline-5-carboxylate, 16c. Diastereomers were separated using Atlantis T3 column (19 mm x100 mm 5µm) + Jupiter Proteo (21 mm x100 mm 4µm); mobile phase A: H_2O with 20 mM ammonium formate pH 4, mobile phase B: CH_3CN, gradient 20-40% B in 20 min. **Diastereomer 1 (16c)**: ^{1}H NMR (400 MHz, CD_3CO_2D) δ ppm 6.95 (d, *J*=9.3 Hz, 1 H) 6.86 (dd, *J*=9.0, 2.8 Hz, 1 H) 6.71 (d, *J*=2.5 Hz, 1 H) 4.44 (q, *J*=7.0 Hz, 2 H) 4.10 (dd, *J*=11.3, 2.8 Hz, 1 H) 3.64 - 3.87 (m, 8 H) 3.49 (dd, *J*=11.0, 3.8 Hz, 1 H) 3.32 (d, *J*=17.6 Hz, 1 H) 2.85 - 3.06 (m, 1 H) 1.39 (t, *J*=7.2 Hz, 3 H); ^{13}C NMR (100 MHz, CD_3CO_2D) δ 168.1, 165.6, 153.9, 137.9, 118.7, 114.8, 114.4, 66.2, 64.8, 63.8, 58.4, 56.3, 54.9, 46.3, 35.5, 13.1; HRMS (ES) MH^+ calcd for $C_{16}H_{23}BrN_2O_4$ 307.1613, found 307.1644.



17c

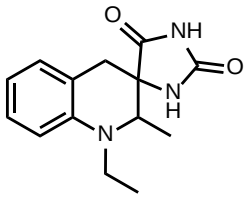
8-Methoxy-2,4,4a,6-tetrahydro-1H-spiro[[1,4]oxazino[4,3-a]quinoline-5,4'-imidazolidine]-2',5'-dione, 17c. To a 25 mL round bottom flask equipped with magnetic stirrer were added **16c** (0.09 g, 0.29 mmol) from above and potassium cyanate (0.048 g, 0.59 mmol). The mixture was dissolved in acetic acid (10 mL) and the resulting solution was stirred overnight at rt. The reaction was concentrated *in vacuo*, re-dissolved in EtOAc and washed with water and brine. The organic layer was separated and dried over $MgSO_4$, filtered, and concentrated *in vacuo* to afford the crude urea derivative. This material was then transferred to a 25 mL round bottom flask, dissolved in EtOH, and NaOEt in EtOH (0.10 mL, 0.29 mmol) was added. The resulting brown solution was stirred at rt for about 30 min to 1 h until all the urea has been converted to the spirohydantoin derivative (as monitored by UPLC-MS). When

the reaction was complete, solution was concentrated *in vacuo*, quenched with saturated aqueous NH_4Cl solution and then extracted with EtOAc 3x. The combined organic layers were washed with brine, dried over $MgSO_4$, filtered, and concentrated *in vacuo*. The crude residue was purified using chromatography (20-75% Water/ CH_3CN /TFA) to afford the pure spirohydantoin product **17b** (0.073 g, 84% yield) as white powdery solid. ^{1}H NMR (400 MHz, DMSO-d_6) δ ppm 10.9 (br, s) 8.29 (s, 1 H) 6.87 (d, *J*=9.3 Hz, 1 H) 6.72 (dd, *J*=9.0, 3.0 Hz, 1 H) 6.64 (d, *J*=3.0 Hz, 1 H) 3.93 (dd, *J*=11.4, 3.4 Hz, 1 H) 3.55 - 3.72 (m, 4 H) 3.54 (td, *J*=9.0, 3.0 Hz, 1 H) 3.19 - 3.47 (m, 2 H) 3.09 (dd, *J*=10.4, 3.1 Hz, 1 H) 2.64 - 2.77 (m, 2 H) ; ^{13}C NMR (100 MHz, DMSO-d_6) δ 175.6, 156.4, 152.2, 138.9, 120.9, 114.7, 113.9, 112.9, 66.1, 65.7, 60.5, 57.0, 55.2, 46.6, 37.2; HRMS (ES) MH^+ calcd for $C_{15}H_{18}N_3O_4$ 304.1292, found 304.1303.



16d

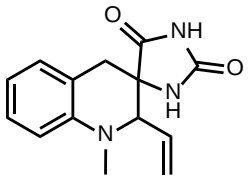
Ethyl 3-amino-1-ethyl-2-methyl-1,2,3,4-tetrahydroquinoline-3-carboxylate, 16d. The major diastereomers was isolated using SFC with DEAP (4.6 mm x250 mm 5µm); mobile phase A: CO_2, mobile phase B: EtOH. **Diastereomer 1 (16d)**: ^{1}H NMR (400 MHz, $CDCl_3$) δ ppm 6.97 - 7.15 (m, 2 H) 6.64 (t, *J*=7.1 Hz, 1 H) 6.59 (d, *J*=7.9 Hz, 1 H) 4.03 - 4.21 (m, 2 H) 3.59 - 3.77 (m, 1 H) 3.33 - 3.50 (m, 1 H) 3.16 - 3.29 (m, 2 H) 2.75 (d, *J*=16.1 Hz, 1 H) 1.09 - 1.22 (m, 9 H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 175.3, 142.6, 129.1, 127.1, 119.9, 116.2, 111.0, 60.9, 58.3, 43.8, 34.3, 14.0, 13.0, 12.8; HRMS (ES) MH^+ calcd for $C_{15}H_{23}N_2O_2$ 263.1715, found 263.1722.



17d

1'-Ethyl-2'-methyl-2',4'-dihydro-1'H-spiro[imidazolidine-4,3'-quinoline]-2,5-dione, 17d. To a 25 mL round bottom flask equipped with magnetic stirrer were added the diasteromerically pure amine **16d** (0.21 g, 0.80 mmol) from above and potassium cyanate (0.13 g, 1.6 mmol). The mixture was dissolved in acetic acid (10 mL) and the resulting solution was

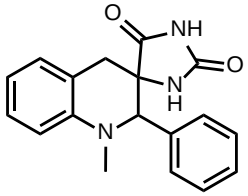
stirred overnight at rt. The reaction was concentrated *in vacuo*, re-dissolved in EtOAc and washed with water and brine. The organic layer was separated and dried over $MgSO_4$, filtered, and concentrated *in vacuo* to afford the crude urea derivative. This material was then transferred to a 25 mL round bottom flask, dissolved in EtOH, and NaOEt in EtOH (0.35 mL, 0.94 mmol) was added. The resulting brown solution was stirred at rt for about 30 min to 1 h until all the urea has been converted to the spirohydantoin derivative (as monitored by UPLC-MS). When the reaction was complete, the solution was concentrated *in vacuo*, quenched with saturated aqueous NH_4Cl solution and then extracted with EtOAc 3x. The combined organic layers were washed with brine, dried over $MgSO_4$, filtered, and concentrated *in vacuo*. The crude residue was purified using chromatography to afford the pure spirohydantoin product **17d** (0.179 g, 88% yield) as white powdery solid. ^{1}H NMR (400 MHz, DMSO-d_6) δ ppm 10.66 (s, 1 H) 8.04 (s, 1 H) 7.02 (t, *J*=7.7 Hz, 1 H) 6.93 (d, *J*=7.1 Hz, 1 H) 6.65 (d, *J*=8.0 Hz, 1 H) 6.53 (t, *J*=7.0 Hz, 1 H) 3.41 - 3.52 (m, 2 H) 3.15 - 3.31 (m, 1 H) 2.92 - 3.02 (d, *J*=20 Hz, 1 H) 2.78 – 2.83 (d, *J*=20 Hz, 1 H) 2.48 - 2.57 (m, 1 H) 1.00 - 1.13 (m, 5 H); ^{13}C NMR (100 MHz, DMSO-d_6) δ 174.9, 141.4, 130.2, 128.2, 116.9, 116.0, 116.6, 61.9, 59.1, 44.6, 32.1, 14.3, 13.5; HRMS (ES) MH^+ calcd for $C_{14}H_{18}N_3O_2$ 260.1354, found 260.1370.



17e

1'-Methyl-2'-vinyl-2',4'-dihydro-1'H-spiro[imidazolidine-4,3'-quinoline]-2,5-dione, 17e. Spirohydantoin **17e** was synthesized *via* **Procedure B**. To a 25 mL round bottom flask equipped with magnetic stirrer were added the diastereomeric mixture of amine from above and potassium cyanate (1.2 equiv). The mixture was dissolved in acetic acid and the resulting solution was stirred overnight at rt. The reaction was concentrated *in vacuo*, re-dissolved in EtOAc and washed with water and brine. The organic layer was separated and dried over $MgSO_4$, filtered, and concentrated *in vacuo* to afford the crude urea derivative. This material (diastereomeric mixture of urea derivatives) was then transferred to a 25 mL round bottom flask, dissolved in EtOH, and NaOEt in EtOH was added. The resulting brown solution was stirred at rt for about 30 min to 1 h until all the urea has been converted to the spirohydantoin derivative (as monitored by UPLC-MS). When the reaction was complete, solution was

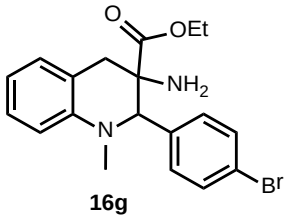
concentrated *in vacuo*, quenched with saturated aqueous NH_4Cl solution and then extracted with EtOAc 3x. The combined organic layers were washed with brine, dried over $MgSO_4$, filtered, and concentrated *in vacuo*. The crude residue was purified using chromatography to afford the two spirohydantoin diastereomers as white powdery solids. **17e-dia-1** (0.2 g, 52% yield): ^{1}H NMR (400 MHz, DMSO-d_6) δ ppm 10.68 (s, 1 H) 8.09 (s, 1 H) 7.08 (t, *J*=7.4 Hz, 1 H) 6.95 (d, *J*=7.1 Hz, 1 H) 6.68 (d, *J*=8.0 Hz, 1 H) 6.61 (t, *J*=7.0 Hz, 1 H) 5.67 (ddd, *J*=17.2, 10.2, 8.3 Hz, 1 H) 5.33 (dd, *J*=12.0, 4.0 Hz, 1 H) 5.22 (d, *J*=16.0 Hz, 1 H) 3.87 (d, *J*=8.0 Hz, 1 H) 3.06 (d, *J*=16.1 Hz, 1 H) 2.82 (s, 3H) 2.74 - 2.78 (d, *J*=16.1 Hz, 1 H); ^{13}C NMR (100 MHz, DMSO-d_6) δ 175.6, 156.5, 144.8, 134.4, 128.9, 127.4, 120.4, 118.5, 116.4, 111.6, 65.1, 61.9, 36.2, 34.9; HRMS (ES) MH^+ calcd for $C_{14}H_{16}N_3O_2$ 258.1198, found 258.1201. **17e-dia-2** (0.13 g, 34 % yield): ^{1}H NMR (400 MHz, DMSO-d_6) δ ppm 10.76 (s, 1 H) 8.08 - 8.15 (m, 1 H) 7.08 (t, *J*=7.3 Hz, 1 H) 6.95 (d, *J*=7.3 Hz, 1 H) 6.57 - 6.65 (m, 2 H) 5.82 (ddd, *J*=16.9, 10.5, 5.9 Hz, 1 H) 5.11 (dt, *J*=10.3, 1.5 Hz, 1 H) 4.92 (dt, *J*=17.2, 1.6 Hz, 1 H) 3.88 (dd, *J*=5.9, 1.4 Hz, 1 H) 3.10 (d, *J*=16.3 Hz, 1 H) 2.91 (s, 3 H) 2.59 - 2.74 (d, *J*=16.3 Hz, 1 H); ^{13}C NMR (100 MHz, DMSO-d_6) δ 175.3, 156.1, 143.6, 132.9, 129.0, 127.5, 116.9, 116.0, 116.0, 110.6, 66.7, 60.7, 37.8, 32.6; HRMS (ES) MH^+ calcd for $C_{14}H_{16}N_3O_2$ 258.1198, found 258.1199.



17f

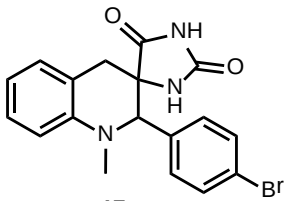
1'-Methyl-2'-phenyl-2',4'-dihydro-1'H-spiro[imidazolidine-4,3'-quinoline]-2,5-dione, 17f. Spirohydantoin **17f** was synthesized *via* **Procedure B**. To a 25 mL round bottom flask equipped with magnetic stirrer were added the diastereomeric mixture of amine (0.4 g, 1.29 mmol) and potassium cyanate (0.125 g, 1.55 mmol). The mixture was dissolved in acetic acid (10 mL) and the resulting solution was stirred overnight at rt. The reaction was then concentrated *in vacuo*, then re-dissolved in EtOAc and washed with water and brine. The organic layer was separated and dried over $MgSO_4$, filtered, and concentrated *in vacuo* to afford the crude urea derivative. This material (diastereomeric mixture of urea derivatives) was then transferred to a 25 mL round bottom flask, dissolved in EtOH (10 mL), and NaOEt in EtOH (0.6 mL, 1.55 mmol) was added. The resulting brown solution was stirred at rt for about 30 min to 1 h until all the urea has been converted to the spirohydantoin derivative (as monitored

by UPLC-MS). When the reaction was complete, the solution was concentrated *in vacuo*, quenched with saturated aqueous NH_4Cl solution and then extracted with EtOAc 3x. The combined organic layers were washed with brine, dried over $MgSO_4$, filtered, and concentrated *in vacuo*. The crude residue was purified using chromatography to afford the two spirohydantoin diastereomers as white powdery solids. **17f-dia-1** (0.245 g, 62% yield, product identical to spirohydantoin **15**); **17f-dia-2** (0.144 g, 36 % yield): ^{1}H NMR (400 MHz, DMSO-d_6) δ ppm 10.33 (br. s., 1 H) 8.20 (s, 1 H) 7.25 - 7.36 (m, 5 H) 7.12 (t, *J*=7.3 Hz, 1 H) 7.01 (d, *J*=7.3 Hz, 1 H) 6.80 (d, *J*=8.0 Hz, 1 H) 6.66 (t, *J*=7.0 Hz, 1 H) 4.56 (s, 1 H) 3.27 - 3.31 (d, *J*=15.8 Hz, 1 H) 2.80 (d, *J*=15.8 Hz, 1 H) 2.63 (s, 3 H); ^{13}C NMR (100 MHz, DMSO-d_6) δ 174.4, 154.9, 144.9, 135.7, 127.8, 127.7, 126.8, 126.5, 117.8, 115.6, 111.8, 65.6, 61.7, 36.6, 34.9; HRMS (ES) MH^+ calcd for $C_{18}H_{18}N_3O_2$ 308.1394, found 308.1381.



16g

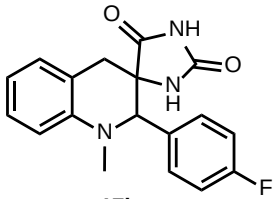
Ethyl 3-amino-2-(4-bromophenyl)-1-methyl-1,2,3,4-tetrahydroquinoline-3-carboxylate, 16g. The major diastereomer was isolated using SFC with Viridis Silica (4.6 mm x 150 mm 5µm); mobile phase A: CO_2, mobile phase B: EtOH, gradient 10-30% B in 5 min. **Diastereomer 1 (16g)**: ^{1}H NMR (400 MHz, $CDCl_3$) δ ppm 7.27 - 7.47 (d, *J*=8.0 Hz, 2 H) 7.15 - 7.24 (m, 1 H) 7.00 (d, *J*=8.0 Hz, 2 H) 6.82 - 6.99 (m, 1 H) 6.50 - 6.72 (m, 2 H) 4.58 (s, 1 H) 4.02 (q, *J*=7.0 Hz, 3 H) 3.20 (d, *J*=15.6 Hz, 1 H) 2.73 (s, 3 H) 2.61 (d, *J*=15.6 Hz, 1 H) 1.05 (t, *J*=8.0 Hz, 3 H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 171.6, 143.3, 138.0, 131.6, 130.3, 128.8, 128.5, 122.2, 117.3, 116.9, 110.1, 70.2, 62.0, 58.9, 37.7, 30.8, 13.7; HRMS (ES) MH^+ calcd for $C_{19}H_{22}BrN_2O_2$ 389.0820, found 389.0991.



17g

2'-(4-Bromophenyl)-1'-methyl-2',4'-dihydro-1'H-spiro[imidazolidine-4,3'-quinoline]-2,5-dione, 17g. To a 25 mL round bottom flask equipped with magnetic stirrer were added the diasteromerically pure amine **16g** (0.04 g, 0.10 mmol) from above and potassium cyanate (0.017 g, 0.21 mmol). The mixture was dissolved in acetic acid (5 mL) and the resulting

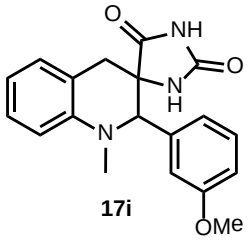
solution was stirred overnight at rt. The reaction was concentrated *in vacuo*, re-dissolved in EtOAc and washed with water and brine. The organic layer was separated and dried over $MgSO_4$, filtered, and concentrated *in vacuo* to afford the crude urea derivative. This material was then transferred to a 25 mL round bottom flask, dissolved in EtOH, and NaOEt in EtOH (0.35 mL, 0.94 mmol) was added. The resulting brown solution was stirred at rt for about 30 min to 1 h until all the urea has been converted to the spirohydantoin derivative (as monitored by UPLC-MS). When the reaction was complete, solution was concentrated *in vacuo*, quenched with saturated aqueous NH_4Cl solution and then extracted with EtOAc 3x. The combined organic layers were washed with brine, dried over $MgSO_4$, filtered, and concentrated *in vacuo*. The crude residue was purified using chromatography to afford the pure spirohydantoin product **17g** (0.03 g, 78% yield) as a white powdery solid. ^{1}H NMR (400 MHz, DMSO-d_6) δ ppm 10.42 (s, 1 H) 8.21 (s, 1 H) 7.55 (d, *J*=7.8 Hz, 2 H) 7.22 (d, *J*=7.8 Hz, 2 H) 7.13 (t, *J*=7.3 Hz, 1 H) 7.01 (d, *J*=7.0 Hz, 1 H) 6.80 (d, *J*=8.0 Hz, 1 H) 6.67 (t, *J*=7.1 Hz, 1 H) 4.58 (s, 1 H) 3.24 (d, *J*=15.8 Hz, 1 H) 2.80 (d, *J*=15.8 Hz, 1 H) 2.63 (s, 3 H) ; ^{13}C NMR (100 MHz, DMSO-d_6) δ 175.3, 156.0, 145.9, 136.4, 130.9, 128.9, 127.6, 121.3, 118.9, 117.0, 112.9, 66.1, 62.6, 54.8, 37.8, 35.6; HRMS (ES) MH^+ calcd for $C_{18}H_{17}BrN_3O_2$ 386.0499, found 386.0511.



17h

2'-(4-Fluorophenyl)-1'-methyl-2',4'-dihydro-1'H-spiro[imidazolidine-4,3'-quinoline]-2,5-dione, 17h. Spirohydantoin **17h** was synthesized *via* **Procedure B**. To a 25 mL round bottom flask equipped with magnetic stirrer were added the diastereomeric mixture of amine (0.449 g, 1.37 mmol) from above and potassium cyanate (0.133 g, 1.64 mmol). The mixture was dissolved in acetic acid (10 mL) and the resulting solution was stirred overnight at rt. The reaction was then concentrated *in vacuo*, then re-dissolved in EtOAc and washed with water and brine. The organic layer was separated and dried over $MgSO_4$, filtered, and concentrated *in vacuo* to afford the crude urea derivative. This material (diastereomeric mixture of urea derivatives) was then transferred to a 25 mL round bottom flask, dissolved in EtOH (10 mL), and NaOEt in EtOH (0.6 mL, 1.64 mmol) was added. The resulting brown solution was stirred at rt for about 30 min to 1 h until all the urea has been converted to the spirohydantoin derivative (as monitored by UPLC-MS). When the reaction was complete, solution was concentrated *in*

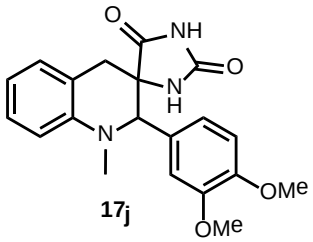
vacuo, quenched with saturated aqueous NH_4Cl solution and then extracted with EtOAc 3x. The combined organic layers were washed with brine, dried over $MgSO_4$, filtered, and concentrated *in vacuo*. The crude residue was purified using chromatography to afford the two spirohydantoin diastereomers as white powdery solids. **17h-dia-1** (0.245 g, 61% yield): ^{1}H NMR (500 MHz, DMSO-d_6) δ ppm 10.62 (br. s., 1 H) 8.23 (s, 1 H) 7.00 - 7.16 (m, 6 H) 6.69 (d, *J*=8.1 Hz, 1 H) 6.65 (t, *J*=7.3 Hz, 1 H) 4.49 (s, 1 H) 3.34 (s, 5 H) 3.03 (d, *J*=16.4 Hz, 1 H) 2.77 (s, 3H) 2.72 - 2.83 (d, *J*=16.4 Hz, 1 H); ^{19}F NMR (470 MHz, DMSO-d_6) δ -73.4; ^{13}C NMR (125 MHz, $CDCl_3$) δ 174.2, 160.6 (d, J_{cf}=241.3 Hz), 156.1, 144.6, 134.4, 129.8, 129.0, 127.7, 117.3, 116.2, 114.6 (d, J_{cf}=21.3 Hz), 110.7, 67.8, 61.3, 37.6, 32.5; HRMS (ES) MH^+ calcd for $C_{17}H_{18}FN_3O_2$ 326.1299, found 326.1293. **17h-dia-2** (0.145 g, 33 % yield):^{1}H NMR (500 MHz, DMSO-d_6) δ ppm 10.5 (br s, 1H), 8.36 (s, 1 H) 7.43 (t, *J*=6.5 Hz, 2 H) 7.24 - 7.34 (m, 3 H) 7.14 (d, *J*=7.3 Hz, 1 H) 6.94 (d, *J*=8.2 Hz, 1 H) 6.80 (t, *J*=7.2 Hz, 1 H) 4.72 (s, 1 H) 3.40 (d, *J*=15.8 Hz, 1 H) 2.93 (d, *J*=15.8 Hz, 1 H) 2.75 (s, 3 H) ; ^{19}F NMR (470 MHz, DMSO-d_6) δ -73.4; ^{13}C NMR (125 MHz, $CDCl_3$) δ 175.4, 162.8 (d, J_{cf}=242.5 Hz), 156.1, 145.9, 133.0, 130.7, 128.9, 127.6, 118.9, 116.9, 114.9 (d, J_{cf}=21.3 Hz), 112.9, 65.9, 62.7, 37.6, 35.8; HRMS (ES) MH^+ calcd for $C_{17}H_{18}FN_3O_2$ 326.1299, found 326.1300.



17i

2'-(3-Methoxyphenyl)-1'-methyl-2',4'-dihydro-1'H-spiro[imidazolidine-4,3'-quinoline]-2,5-dione, 17i. Spirohydantoin **17i** was synthesized *via* **Procedure B**. To a 25 mL round bottom flask equipped with magnetic stirrer were added the diastereomeric mixture of amine (0.33 g, 0.97 mmol) and potassium cyanate (0.157 g, 1.94 mmol). The mixture was dissolved in acetic acid (5 mL) and the resulting solution was stirred overnight at rt. The reaction was then heated to 90 ^{o}C for 24 h until all the urea has been converted to the spirohydantoin derivative (as monitored by UPLC-MS). When the reaction was complete, the solution was cooled to rt, diluted with water and then extracted with EtOAc 3x. The combined organic layers were washed with brine, dried over $MgSO_4$, filtered, and concentrated *in vacuo*. The crude residue was purified using chromatography to afford the two spirohydantoin diastereomers as white powdery solids. **17i-dia-1** (0.209 g, 64% yield): ^{1}H NMR (500 MHz, DMSO-d_6) δ ppm 10.78 (s, 1 H) 8.40 (s, 1 H) 7.28 - 7.43 (m, 2 H) 7.21 (d, *J*=7.3 Hz, 1 H) 7.03 (d, *J*=8.1 Hz, 1

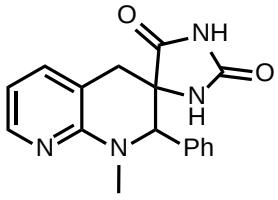
H) 6.78 - 6.92 (m, 4 H) 4.60 (s, 1 H) 3.86 (s, 3 H) 3.27 (d, *J*=16.4 Hz, 1 H) 3.00 (d, *J*=16.4 Hz, 1 H) 2.96 (s, 3H); ^{13}C NMR (100 MHz, DMSO-d_6) δ 175.5, 158.9, 156.1, 146.1, 138.4, 128.9, 127.6, 121.1, 118.9, 116.9, 114.4, 113.6, 112.8, 66.6, 62.8, 55.0, 37.6, 36.1; HRMS (ES) MH^+ calcd for $C_{19}H_{20}N_3O_3$ 338.1499, found 338.1488. **17i-dia-2** (0.087 g, 26 % yield): ^{1}H NMR (400 MHz, DMSO-d_6) δ ppm 10.59 (br. s., 1 H) 8.19 (s, 1 H) 7.07 - 7.23 (m, 2 H) 7.02 (d, *J*=7.0 Hz, 1 H) 6.77 - 6.91 (m, 1 H) 6.58 - 6.73 (m, 4 H) 4.40 (s, 1 H) 3.66 (s, 3 H) 3.08 (d, *J*=16.3 Hz, 1 H) 2.81 (d, *J*=16.3 Hz, 1 H) 2.77 (s, 3 H); ^{13}C NMR (100 MHz, DMSO-d_6) δ 174.2, 158.6, 156.0, 144.8, 139.7, 128.9, 128.8, 127.6, 120.1, 117.6, 116.2, 114.4, 112.5, 110.7, 68.6, 61.5, 54.8, 37.7, 32.9; HRMS (ES) MH^+ calcd for $C_{19}H_{20}N_3O_3$ 338.1499, found 338.1504.



17j

2'-(3,4-Dimethoxyphenyl)-1'-methyl-2',4'-dihydro-1'H-spiro[imidazolidine-4,3'-quinoline]-2,5-dione, 17j. Spirohydantoin **17j** was synthesized *via* **Procedure B**. To a 25 mL round bottom flask equipped with magnetic stirrer were added the diastereomeric mixture of amine (0.46g, 1.24 mmol) from above and potassium cyanate (0.201 g, 2.48 mmol). The mixture was dissolved in acetic acid (10 mL) and the resulting solution was stirred overnight at rt. The reaction was then concentrated *in vacuo*, re-dissolved in EtOAc and washed with water and brine. The organic layer was separated and dried over $MgSO_4$, filtered, and concentrated *in vacuo* to afford the crude urea derivative. This material (diastereomeric mixture of urea derivatives) was then transferred to a 25 mL round bottom flask, dissolved in EtOH, and NaOEt in EtOH (0.6 mL, 1.45 mmol) was added. The resulting brown solution was stirred at rt for about 30 min to 1 h until all the urea has been converted to the spirohydantoin derivative (as monitored by UPLC-MS). When the reaction was complete, the solution was concentrated *in vacuo*, quenched with saturated aqueous NH_4Cl solution and then extracted with EtOAc 3x. The combined organic layers were washed with brine, dried over $MgSO_4$, filtered, and concentrated *in vacuo*. The crude residue was purified using chromatography to afford the two spirohydantoin diastereomers as white powdery solids. **17j-dia-1** (0.165 g, 37% yield^{1}H NMR (500 MHz, DMSO-d_6) δ 10.30 (s, 1 H) 8.25 (s, 1 H) 7.13 (t, *J*=7.8 Hz, 1 H) 7.01 (d, *J*=7.7 Hz, 1 H) 6.99 (d, *J*=8.1 Hz, 1 H) 6.89 (d, *J*=8.5 Hz, 1 H) 6.81 (d, *J*=8.2 Hz, 1 H) 6.75 (d, *J*=8.1 Hz,

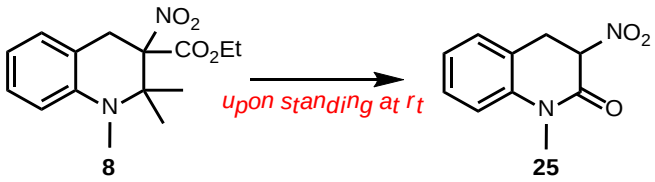
1 H) 6.68 (t, *J*=7.2 Hz, 1 H) 4.48 (s, 1 H) 3.75 (s, 3 H) 3.71 (s, 3 H) 3.28 (d, *J*=15.8 Hz, 1 H) 2.78 (d, *J*=15.8 Hz, 1 H) 2.64 (s, 3 H); ^{13}C NMR (125 MHz, DMSO-d_6) δ 175.6, 158.9, 156.3, 148.5, 148.2, 146.1, 128.9, 128.8, 127.6, 121.1, 118.9, 116.8, 112.9, 112.0, 110.7, 66.2, 62.9, 55.6, 55.3, 37.3, 36.2; HRMS (ES) MH^+ calcd for $C_{20}H_{22}N_3O_4$ 368.1605, found 368.1613. **17j-dia-2** (0.220 g, 50 % yield): ^{1}H NMR (500 MHz, DMSO-d_6) δ 10.52 (s, 1 H) 8.14 (s, 1 H) 7.13 (t, *J*=7.9 Hz, 1 H) 7.00 (d, *J*=7.3 Hz, 1 H) 6.83 (d, *J*=8.2 Hz, 1 H) 6.57 - 6.75 (m, 4 H) 4.34 (s, 1 H) 3.72 (s, 3H), 3.60 (s, 3H), 3.06 (d, *J*=16.4 Hz, 1 H) 2.69 - 2.85 (d, *J*=16.4 Hz, 1 H) 2.76 (s, 3 H); ^{13}C NMR (125 MHz, DMSO-d_6) δ 174.4, 156.1, 148.2, 147.7, 145.0, 130.3, 128.8, 127.6, 119.9, 117.8, 116.2, 112.3, 111.0, 110.9, 68.2, 61.7, 55.3 (2 C), 37.7, 33.2; HRMS (ES) MH^+ calcd for $C_{20}H_{22}N_3O_4$ 368.1605, found 368.1597.



17k

1'-methyl-2'-phenyl-2',4'-dihydro-1'H-spiro[imidazolidine-4,3'-[1,8]naphthyridine]-2,5-dione, 17k. Spirohydantoin **17k** was synthesized *via* **Procedure B**. To a 25 mL round bottom flask equipped with magnetic stirrer were added the diastereomeric mixture of amine (0.46g, 1.24 mmol) from above and potassium cyanate (0.201 g, 2.48 mmol). The mixture was dissolved in acetic acid (10 mL) and the resulting solution was stirred overnight at rt. The reaction was then concentrated *in vacuo,* re-dissolved in EtOAc and washed with water and brine. The organic layer was separated and dried over $MgSO_4$, filtered, and concentrated *in vacuo* to afford the crude urea derivative. This material (diastereomeric mixture of urea derivatives) was then transferred to a 25 mL round bottom flask, dissolved in EtOH, and NaOEt in EtOH (0.6 mL, 1.45 mmol) was added. The resulting brown solution was stirred at rt for about 30 min to 1 h until all the urea has been converted to the spirohydantoin derivative (as monitored by UPLC-MS). When the reaction was complete, the solution was concentrated *in vacuo,* quenched with saturated aqueous NH_4Cl solution and then extracted with EtOAc 3x. The combined organic layers were washed with brine, dried over $MgSO_4$, filtered, and concentrated *in vacuo*. The crude residue was purified using chromatography to afford the two spirohydantoin diastereomers as white powdery solids. **17k-dia-1** (0.165 g, 37% yield): ^{1}H

NMR (500 MHz, DMSO-d_6) δ ppm 10.62 (br. s., 1 H) 8.36 (s, 1 H) 8.04 (dd, *J*=5.0, 1.6 Hz, 1 H) 7.25 - 7.34 (m, 4 H) 7.02 - 7.08 (m, 2 H) 6.61 (dd, *J*=7.1, 4.9 Hz, 1 H) 4.60 (s, 1 H) 3.34 (s, 6 H) 3.06 (d, *J*=16.4 Hz, 1 H) 2.91 (s, 3 H) 2.82 (d, *J*=16.1 Hz, 1 H); ^{13}C NMR (125 MHz, DMSO-d_6) δ 174.0, 156.0, 154.9, 146.1, 137.5, 136.1, 127.9, 127.6, 112.9, 112.2, 68.3, 60.9, 35.4, 32.4; HRMS (ES) MH^+ calcd for $C_{17}H_{17}N_4O_2$ 309.1346, found 309.1354. **17k-dia-2** (0.220 g, 50 % yield): ^{1}H NMR (500 MHz, DMSO-d_6) δ 10.43 (br. s., 1 H) 8.24 (s, 1 H) 8.04 (dd, *J*=4.7, 1.3 Hz, 1 H) 7.30 - 7.39 (m, 4 H) 7.25 (d, *J*=6.6 Hz, 2 H) 6.63 (dd, *J*=7.3, 5.0 Hz, 1 H) 4.75 (s, 1 H) 3.25 (d, *J*=15.8 Hz, 1 H) 2.85 (d, *J*=15.8 Hz, 1 H) 2.79 (s, 3 H); ^{13}C NMR (100 MHz, DMSO-d_6) δ 175.4, 155.9, 146.0, 136.2, 128.8, 128.1, 127.9, 113.9, 112.6, 66.2, 61.7, 34.9, 29.9; HRMS (ES) MH^+ calcd for $C_{17}H_{17}N_4O_2$ 309.1346, found 309.1359.



8 25

Ethyl 1,2,2-trimethyl-3-nitro-1,2,3,4-tetrahydroquinoline-3-carboxylate, 8. ^{1}H NMR (400 MHz, $CDCl_3$) δ ppm 7.09 (t, *J*=8.0 Hz, 1 H) 7.00 (d, *J*=8.0 Hz, 1 H) 6.68 (t, *J*=8.0 Hz, 1 H) 6.63 (d, *J*=8.0 Hz, 1 H) 4.13 - 4.26 (m, 2 H) 3.70 (d, *J*=17.6 Hz, 1 H) 3.43 (d, *J*=17.6 Hz, 1 H) 2.80 (s, 3 H) 1.59 (s, 3 H) 1.36 (s, 3 H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 165.6, 144.6, 128.3, 127.7, 118.5, 117.7, 113.4, 95.5, 62.6, 59.3, 34.5, 31.1, 23.2, 22.6, 13.8; HRMS (ES) MH^+ calcd for $C_{15}H_{20}N_2O$ 292.1423, not determined due to instability.

1-methyl-3-nitro-3,4-dihydroquinolin-2(1H)-one, 25. This material was formed from the decomposition. of tetrahydroquinoline-nitroester **8** after standing at rt overnight. ^{1}H NMR (400 MHz, $CDCl_3$) δ ppm 7.37 (t, *J*=7.8 Hz, 1 H) 7.22 - 7.29 (m, 1 H) 7.13 (t, *J*=7.6 Hz, 1 H) 7.09 (d, *J*=8.2 Hz, 1 H) 5.37 (dd, *J*=9.8, 5.5 Hz, 1 H) 3.80 (dd, *J*=15.7, 9.9 Hz, 1 H) 3.48 (s, 3H) 3.39 - 3.44 (dd, *J*=12.0, 4.0 Hz, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 160.8, 138.8, 128.9, 128.7, 124.2, 120.4, 115.4, 83.6, 30.9, 30.5; HRMS (ES) MH^+ calcd for $C_{10}H_{10}N_2O_3$ 206.0691, did not ionize either by ES+ or ES-.

11. ^{1}H NMR and ^{13}C NMR spectra of spirohydantoin products.

9/2/2015 3:45:31 PM

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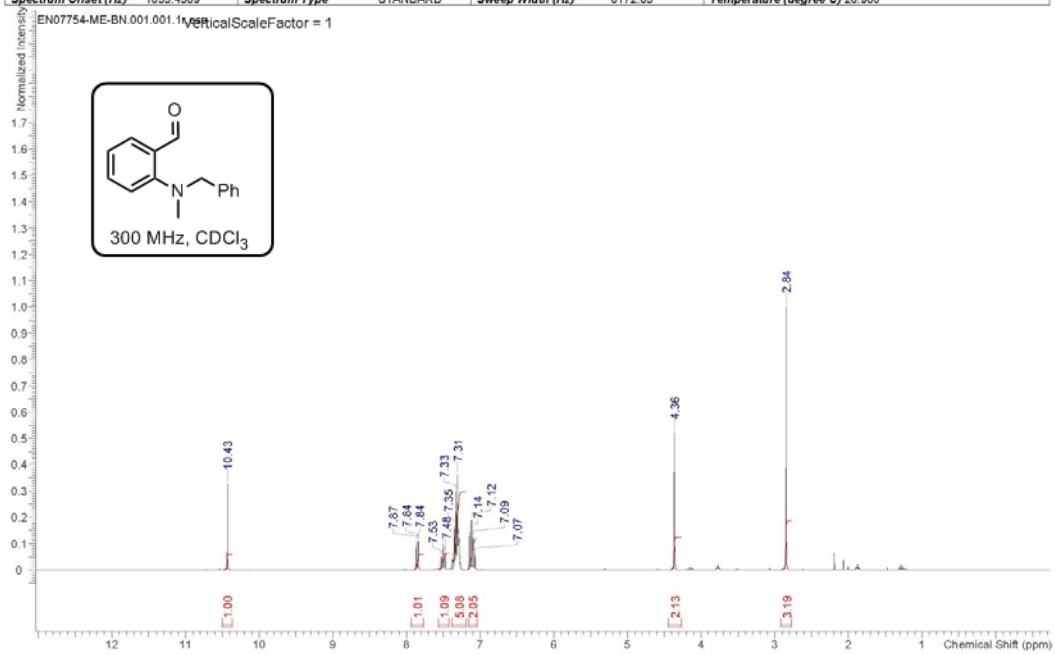

9/2/2015 3:06:38 PM

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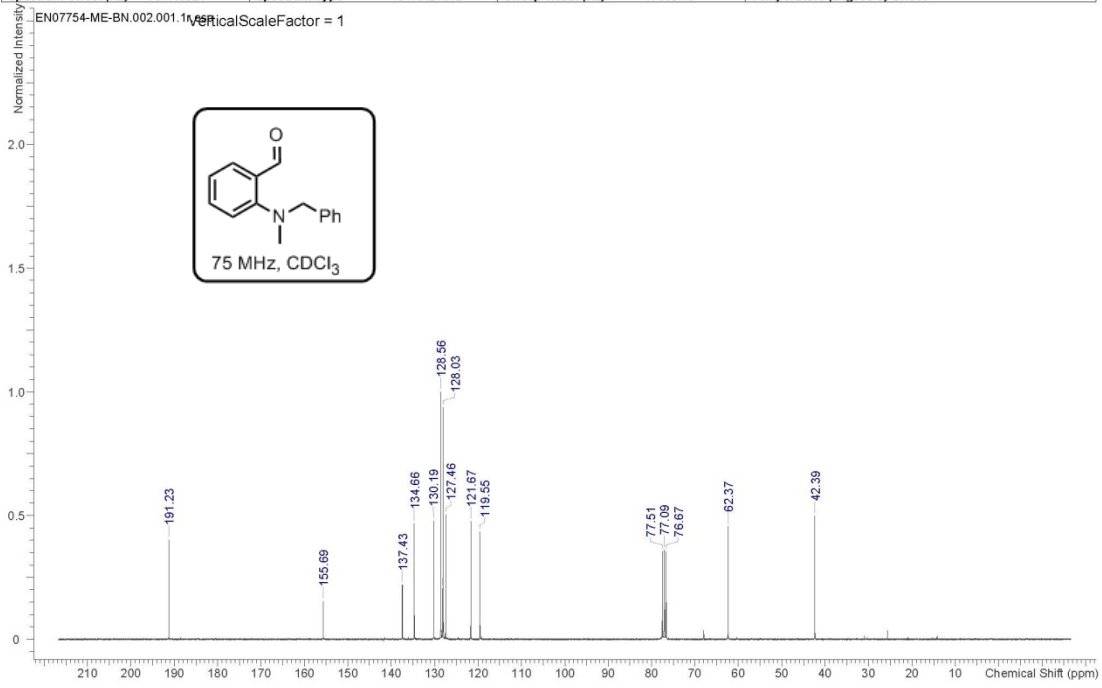

9/2/2015 3:43:48 PM

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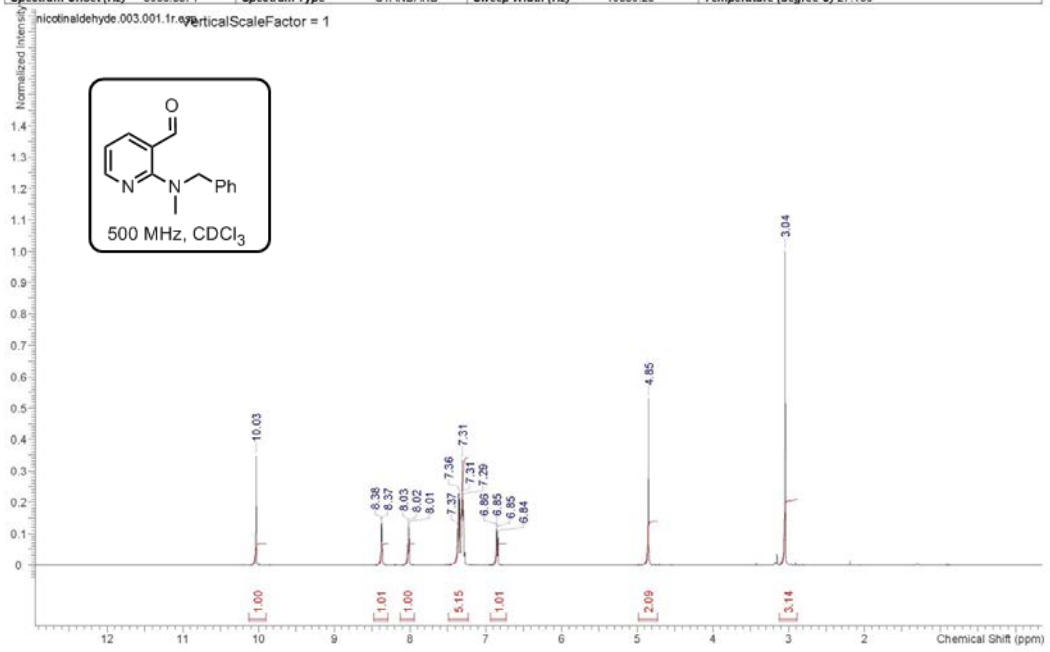

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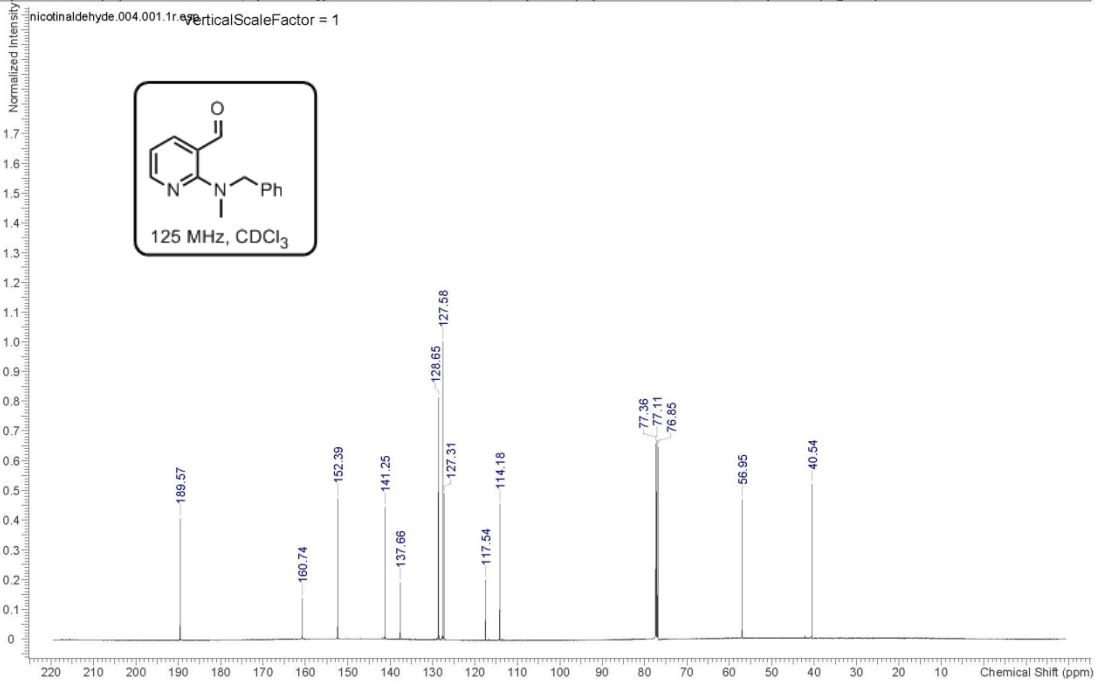
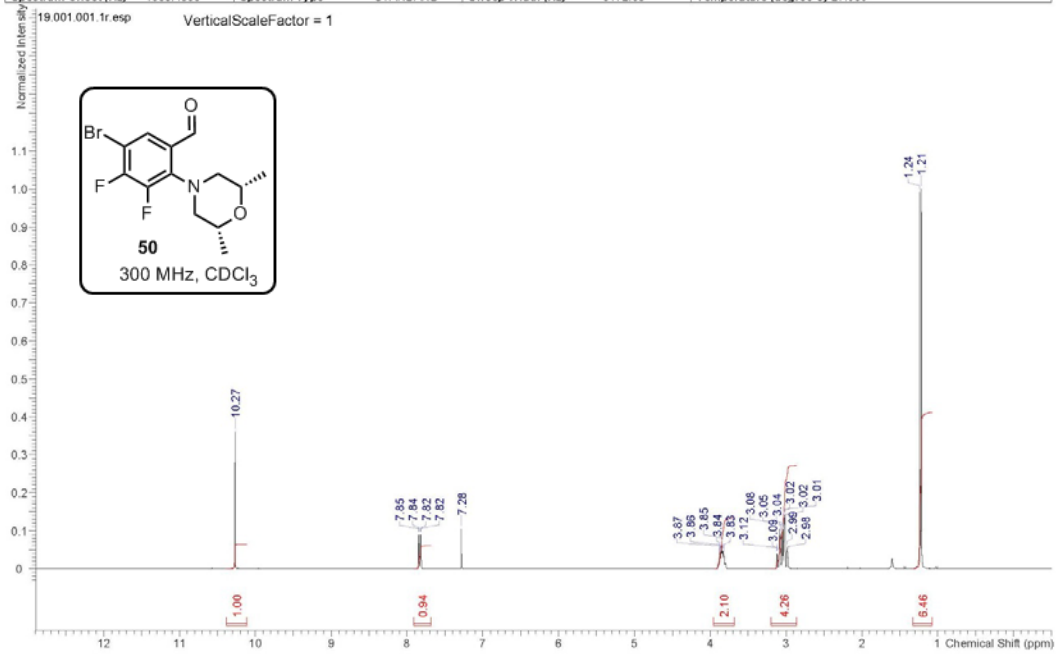
Date Stamp	11 Aug 2015 15:35:28			*File Name*	C:\Users\kmwk459\Desktop\OL-NMR\nicotinaldehyde\4\pdata\1\1r		
Frequency (MHz)	125.77	*Nucleus*	13C	*Number of Transients*	512	*Origin*	spect
Original Points Count	32768	*Owner*	usbodlab	*Points Count*	32768	*Pulse Sequence*	zgpg30
Receiver Gain	5792.60	*SW(cyclical) (Hz)*	30030.03	*Solvent*	CHLOROFORM-d		
Spectrum Offset (Hz)	12578.0596	*Spectrum Type*	STANDARD	*Sweep Width (Hz)*	30029.11	*Temperature (degree C)*	27.160



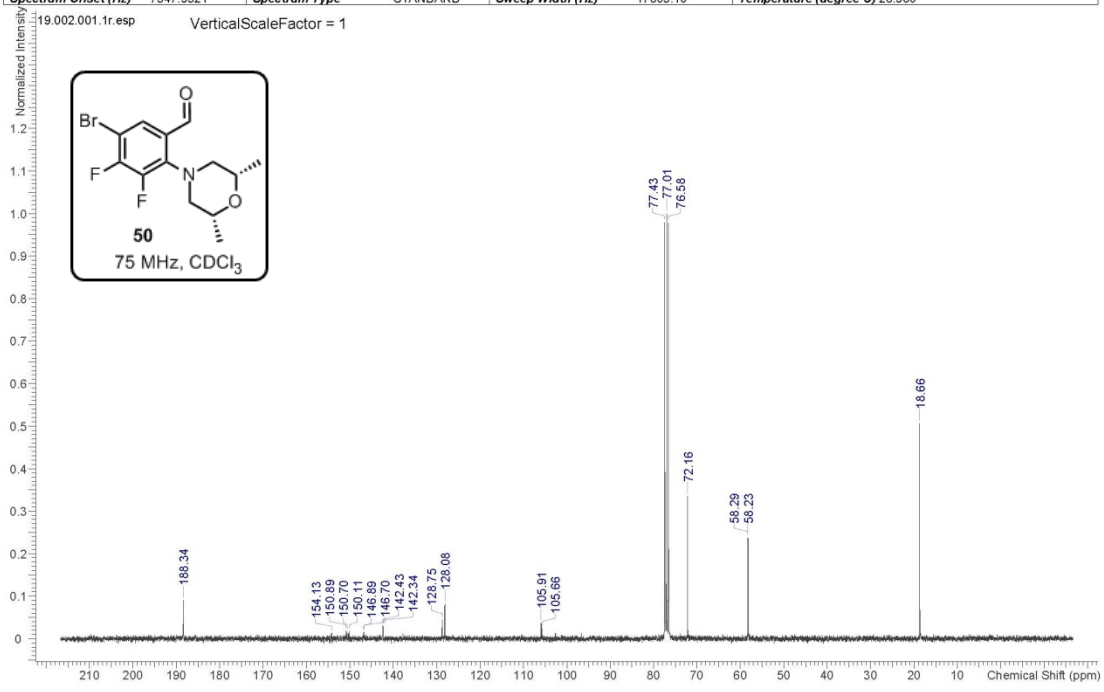
9/2/2015 3:56:19 PM

OriginalDateForRelativeTime 2015-06-04T18:30:24				***Multiplets Integrals Sum***	0.00	***Number of Nuclei***	0 H's
Acquisition Time (sec)	2.6542	***Comment***	A3 300MHz proton_QNP_32 CDCl3 {C:\u} fbriones 37			***Date***	04 Jun 2015 18:30:24
Date Stamp	04 Jun 2015 18:30:24			***File Name***	C:\Users\kmwk459\Desktop\OL-NMR\19\1\pdata\1\1r		
Frequency (MHz)	300.13	***Nucleus***	1H	***Number of Transients***	32	***Origin***	spect
Original Points Count	16384	***Owner***	usbodlab	***Points Count***	32768	***Pulse Sequence***	zg30
Receiver Gain	574.70	***SW(cyclical) (Hz)***	6172.84	***Solvent***	CHLOROFORM-d		
Spectrum Offset (Hz)	1853.4569	***Spectrum Type***	STANDARD	***Sweep Width (Hz)***	6172.65	***Temperature (degree C)***	27.060



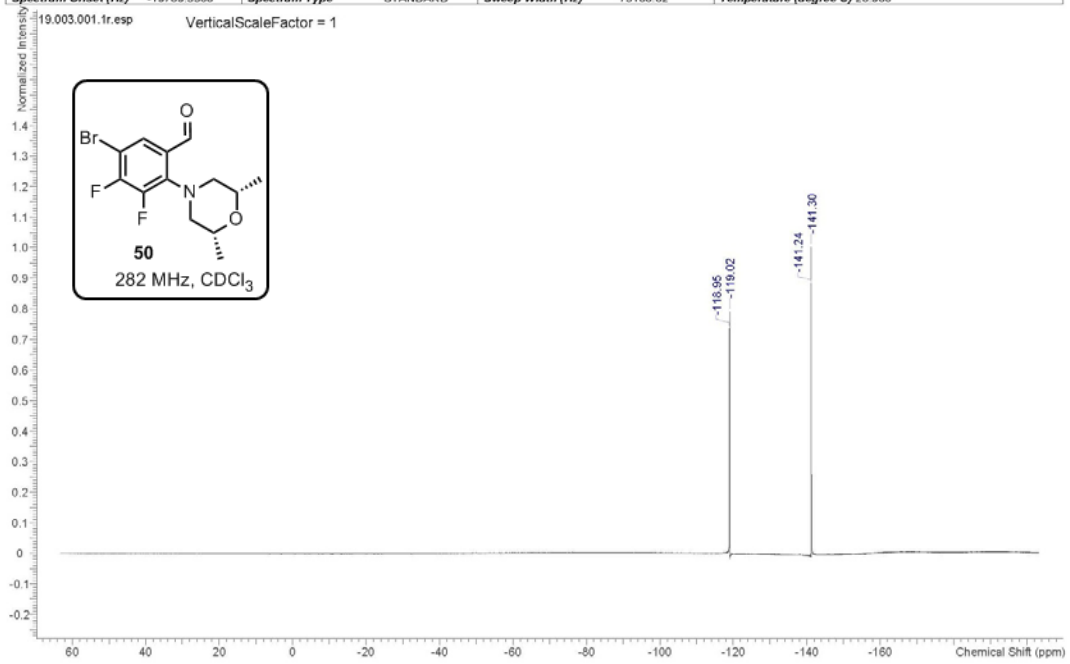
9/2/2015 4:00:34 PM

OriginalDateForRelativeTime 2015-06-05T05:33:52				***Number of Nuclei***	0 C's		
Acquisition Time (sec)	1.8612	***Comment***	A3 300MHz carbon_QNP_2500 CDCl3 {C:\u} fbriones 37			***Date***	05 Jun 2015 05:33:52
Date Stamp	05 Jun 2015 05:33:52			***File Name***	C:\Users\kmwk459\Desktop\OL-NMR\19\2\pdata\1\1r		
Frequency (MHz)	75.48	***Nucleus***	13C	***Number of Transients***	2500	***Origin***	spect
Original Points Count	32768	***Owner***	usbodlab	***Points Count***	32768	***Pulse Sequence***	zgpg30
Receiver Gain	3251.00	***SW(cyclical) (Hz)***	17605.63	***Solvent***	CHLOROFORM-d		
Spectrum Offset (Hz)	7547.9321	***Spectrum Type***	STANDARD	***Sweep Width (Hz)***	17605.10	***Temperature (degree C)***	26.960



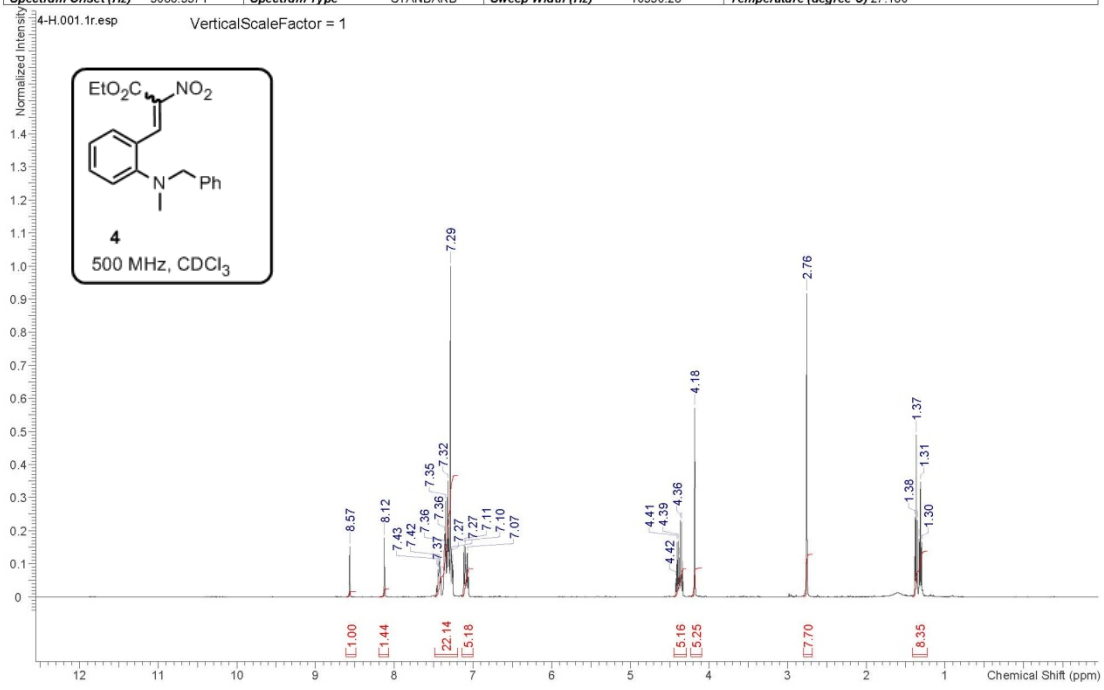
9/2/2015 3:57:29 PM

OriginalDateForRelativeTime 2015-06-05T12:19:12				*Multiplets Integrals Sum*	0.00	*Number of Nuclei*	0 F's
Acquisition Time (sec)	0.8716	*Comment*	A3 300MHz fluorine_dec_QNP_64 CDCl3 {C:\u} fbriones 37			*Date*	05 Jun 2015 12:19:12
Date Stamp	05 Jun 2015 12:19:12			*File Name*	C:\Users\kmwk459\Desktop\OL-NMR\19\3\pdata\1\1r		
Frequency (MHz)	282.38	*Nucleus*	19F	*Number of Transients*	64	*Origin*	spect
Original Points Count	65536	*Owner*	usbodlab	*Points Count*	65536	*Pulse Sequence*	zgfhigqn
Receiver Gain	1625.50	*SW(cyclical) (Hz)*	75187.97	*Solvent*	CHLOROFORM-d		
Spectrum Offset (Hz)	-19769.5508	*Spectrum Type*	STANDARD	*Sweep Width (Hz)*	75186.82	*Temperature (degree C)*	26.960



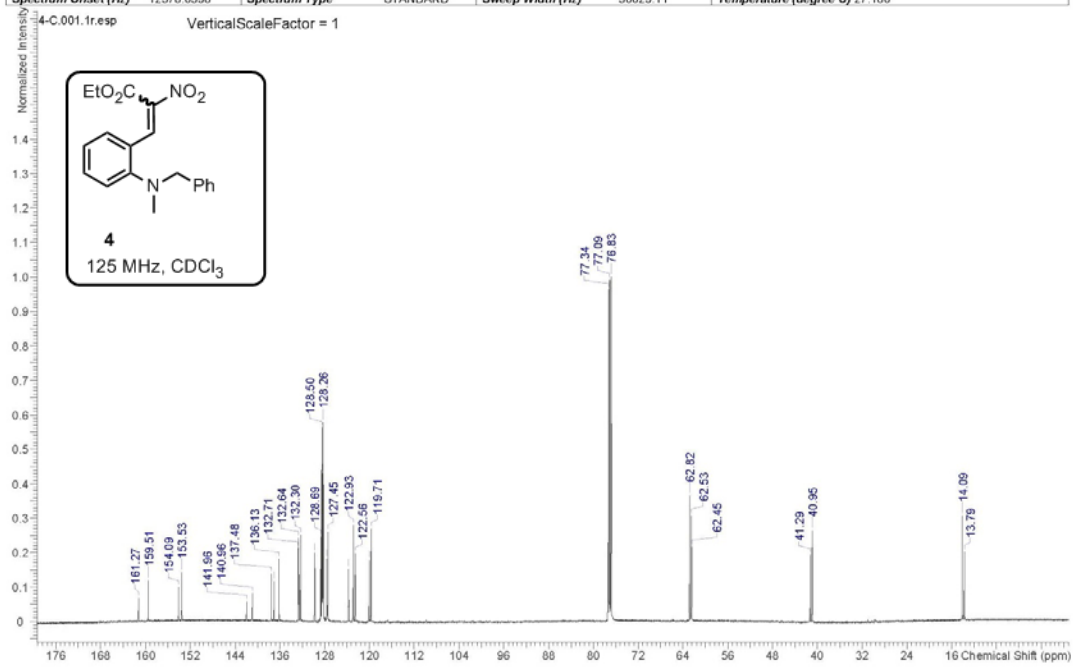
9/2/2015 4:06:21 PM

OriginalDateForRelativeTime 2015-08-12T13:10:24				*Multiplets Integrals Sum*	0.00	*Number of Nuclei*	0 H's
Acquisition Time (sec)	3.1719	*Comment*	PROTON CDCl3 {C:\Bruker\TOPSPIN1.3} fbriones 48			*Date*	12 Aug 2015 13:10:24
Date Stamp	12 Aug 2015 13:10:24			*File Name*	C:\Users\kmwk459\Desktop\OL-NMR\4-H\pdata\1\1r		
Frequency (MHz)	500.13	*Nucleus*	1H	*Number of Transients*	16	*Origin*	spect
Original Points Count	32768	*Owner*	usbodlab	*Points Count*	32768	*Pulse Sequence*	zg30
Receiver Gain	362.00	*SW(cyclical) (Hz)*	10330.58	*Solvent*	CHLOROFORM-d		
Spectrum Offset (Hz)	3088.5571	*Spectrum Type*	STANDARD	*Sweep Width (Hz)*	10330.26	*Temperature (degree C)*	27.160



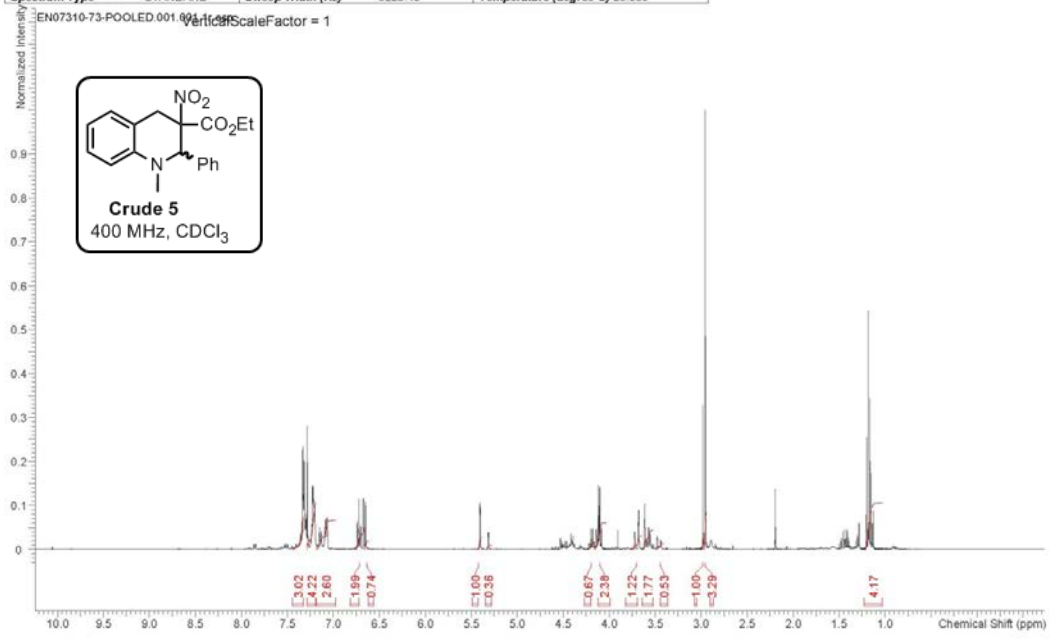
9/2/2015 4:07:59 PM

OriginalDateForRelativeTime 2015-08-12T12:53:20				*Multiplets Integrals Sum* 0.00		*Number of Nuclei* 0 C's	
Acquisition Time (sec) 1.0912	*Comment* C13CPD CDCl3 {C:\Bruker\TOPSPIN1.3} fbriones 34					*Date* 12 Aug 2015 12:53:20	
Date Stamp 12 Aug 2015 12:53:20				*File Name* C:\Users\kmwk459\Desktop\OL-NMR\4-C\pdata\1\1r			
Frequency (MHz) 125.77	*Nucleus* 13C			*Number of Transients* 512		*Origin* spect	
Original Points Count 32768	*Owner* usbodlab			*Points Count* 32768		*Pulse Sequence* zgpg30	
Receiver Gain 1149.40	*SW(cyclical) (Hz)* 30030.03			*Solvent* CHLOROFORM-d			
Spectrum Offset (Hz) 12578.0596	*Spectrum Type* STANDARD			*Sweep Width (Hz)* 30029.11		*Temperature (degree C)* 27.160	



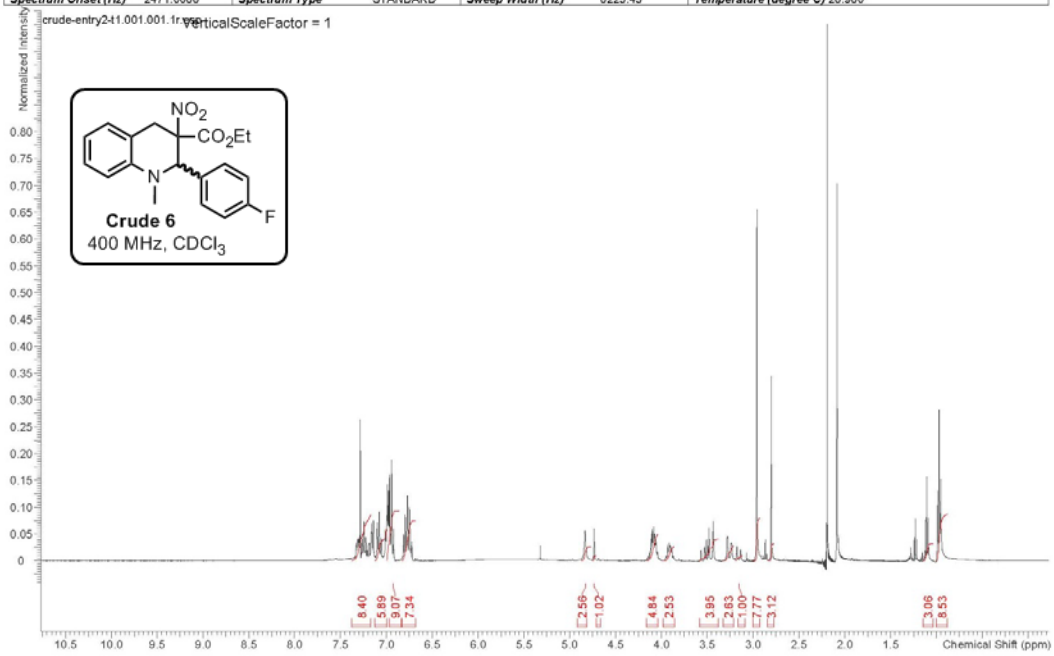
9/4/2015 4:44:51 PM

OriginalDateForRelativeTime 2014-03-30T13:38:08				*Multiplets Integrals Sum* 0.00		*Number of Nuclei* 0 H's	
Acquisition Time (sec) 0.9961	*Comment* proton_QNP_32 CDCl3 {C:\u} fbriones 14					*Date* 30 Mar 2014 13:38:08	
Date Stamp 30 Mar 2014 13:38:08							
File Name \\netapp2\nmr_archive\GHP_C3_400\data\fbriones\nmr\EN07310-73-POOLED\1\pdata\1\1r						*Frequency (MHz)* 400.13	
Nucleus 1H	*Number of Transients* 32		*Origin* spect			*Original Points Count* 8192	
Owner usbodlab	*Points Count* 32768		*Pulse Sequence* zg30			*Receiver Gain* 181.00	
SW(cyclical) (Hz) 8223.68	*Solvent* CHLOROFORM-d					*Spectrum Offset (Hz)* 2471.0088	
Spectrum Type STANDARD	*Sweep Width (Hz)* 8223.43		*Temperature (degree C)* 26.960				



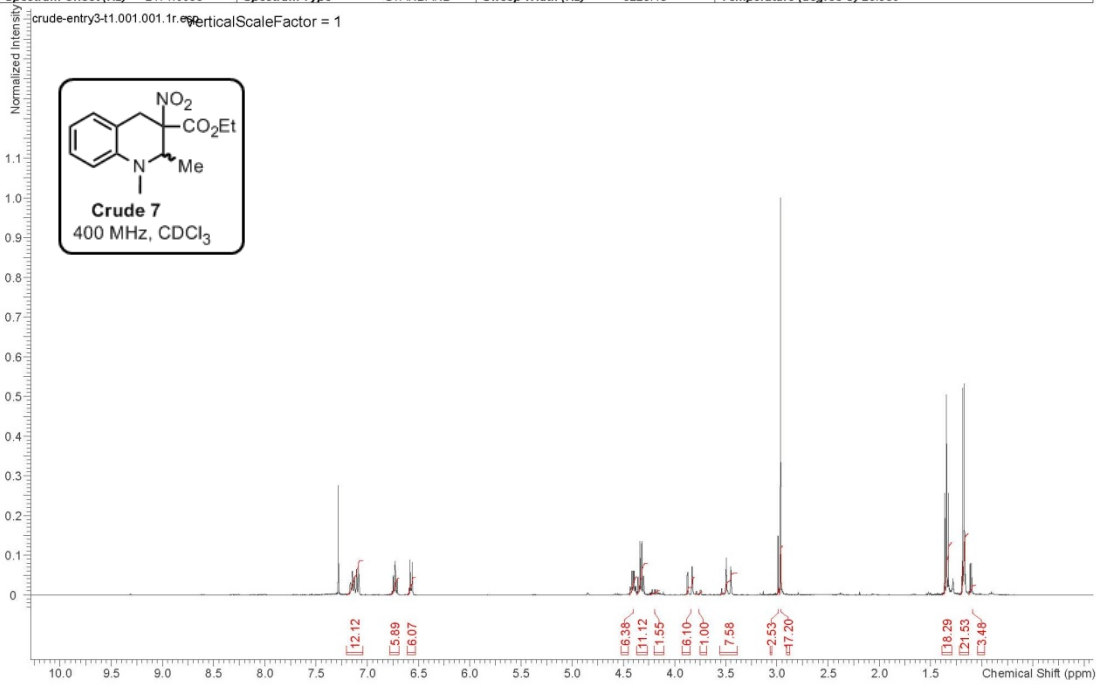
9/2/2015 4:23:56 PM

OriginalDateForRelativeTime 2014-11-19T16:07:12			*Multiplets Integrals Sum* 0.00	*Number of Nuclei* 0 H's	
Acquisition Time (sec) 0.9961	*Comment*	proton_QNP_32 CDCl3 {C:\u} fbriones 44		*Date*	19 Nov 2014 16:07:12
Date Stamp 19 Nov 2014 16:07:12			*File Name*	C:\Users\kmwk459\Desktop\OL-NMR\crude-entry2-t1\1\pdata\1\1r	
Frequency (MHz) 400.13	*Nucleus*	1H	*Number of Transients* 32	*Origin*	spect
Original Points Count 8192	*Owner*	usbodlab	*Points Count* 32768	*Pulse Sequence*	zg30
Receiver Gain 161.30	*SW(cyclical) (Hz)*	8223.68	*Solvent* CHLOROFORM-d		
Spectrum Offset (Hz) 2471.0088	*Spectrum Type*	STANDARD	*Sweep Width (Hz)* 8223.43	*Temperature (degree C)* 26.960	



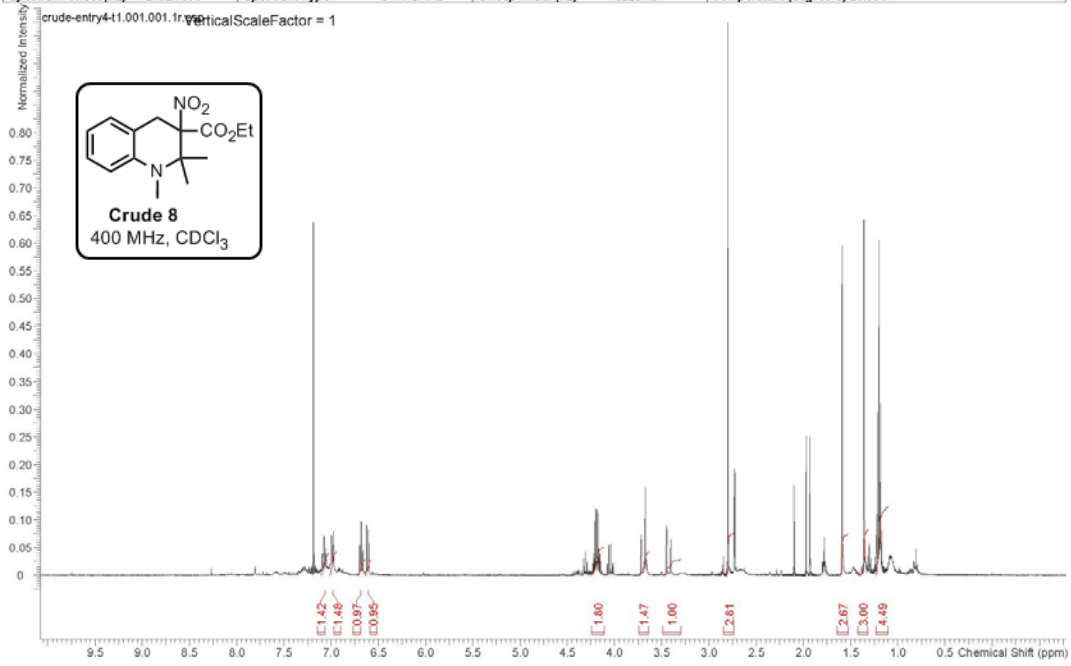
9/2/2015 4:26:29 PM

OriginalDateForRelativeTime 2014-10-20T09:54:08			*Multiplets Integrals Sum* 0.00	*Number of Nuclei* 0 H's	
Acquisition Time (sec) 0.9961	*Comment*	proton_QNP_32 CDCl3 {C:\u} fbriones 11		*Date*	20 Oct 2014 09:54:08
Date Stamp 20 Oct 2014 09:54:08			*File Name*	C:\Users\kmwk459\Desktop\OL-NMR\crude-entry3-t1\1\pdata\1\1r	
Frequency (MHz) 400.13	*Nucleus*	1H	*Number of Transients* 32	*Origin*	spect
Original Points Count 8192	*Owner*	usbodlab	*Points Count* 32768	*Pulse Sequence*	zg30
Receiver Gain 181.00	*SW(cyclical) (Hz)*	8223.68	*Solvent* CHLOROFORM-d		
Spectrum Offset (Hz) 2471.0088	*Spectrum Type*	STANDARD	*Sweep Width (Hz)* 8223.43	*Temperature (degree C)* 26.960	



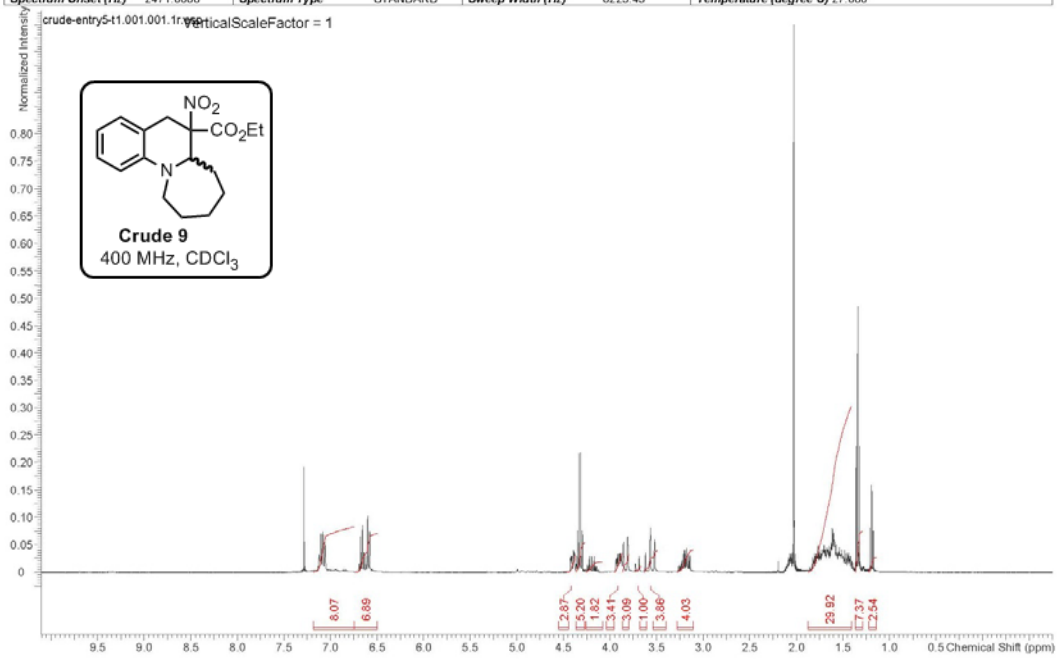
9/2/2015 4:29:52 PM

OriginalDateForRelativeTime 2014-11-05T16:32:48				*Multiplets Integrals Sum* 0.00		*Number of Nuclei* 0 H's	
Acquisition Time (sec) 0.9961	*Comment*	proton_longdelay_QNP_16 CDCl3 {C:\u} fbriones 3				*Date*	05 Nov 2014 16:32:48
Date Stamp 05 Nov 2014 16:32:48				*File Name*	C:\Users\kmwk459\Desktop\OL-NMR\crude-entry4-t1\1\pdata\1\1r		
Frequency (MHz) 400.13	*Nucleus*	1H		*Number of Transients*	16	*Origin*	spect
Original Points Count 8192	*Owner*	usbodlab		*Points Count*	32768	*Pulse Sequence*	zg30
Receiver Gain 181.00	*SW(cyclical) (Hz)*	8223.68		*Solvent*	CHLOROFORM-d		
Spectrum Offset (Hz) 2432.7966	*Spectrum Type*	STANDARD		*Sweep Width (Hz)*	8223.43	*Temperature (degree C)*	26.960



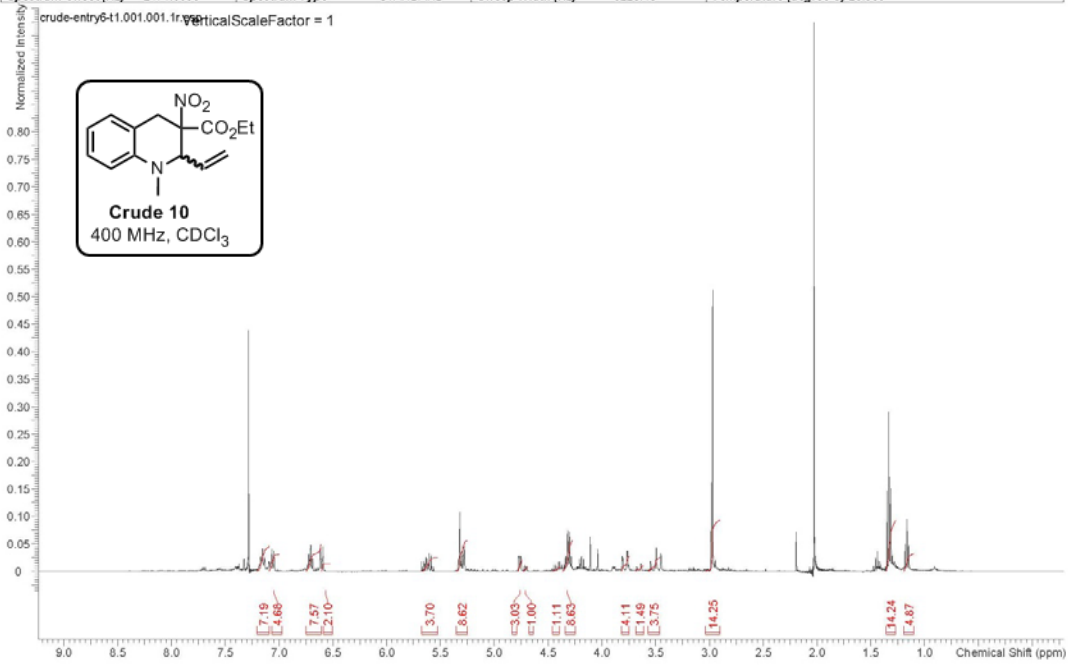
9/2/2015 4:32:14 PM

OriginalDateForRelativeTime 2014-12-05T15:45:52				*Multiplets Integrals Sum* 0.00		*Number of Nuclei* 0 H's	
Acquisition Time (sec) 0.9961	*Comment*	proton_QNP_32 CDCl3 {C:\u} fbriones 21				*Date*	05 Dec 2014 15:45:52
Date Stamp 05 Dec 2014 15:45:52				*File Name*	C:\Users\kmwk459\Desktop\OL-NMR\crude-entry5-t1\1\pdata\1\1r		
Frequency (MHz) 400.13	*Nucleus*	1H		*Number of Transients*	32	*Origin*	spect
Original Points Count 8192	*Owner*	usbodlab		*Points Count*	32768	*Pulse Sequence*	zg30
Receiver Gain 181.00	*SW(cyclical) (Hz)*	8223.68		*Solvent*	CHLOROFORM-d		
Spectrum Offset (Hz) 2471.0088	*Spectrum Type*	STANDARD		*Sweep Width (Hz)*	8223.43	*Temperature (degree C)*	27.060



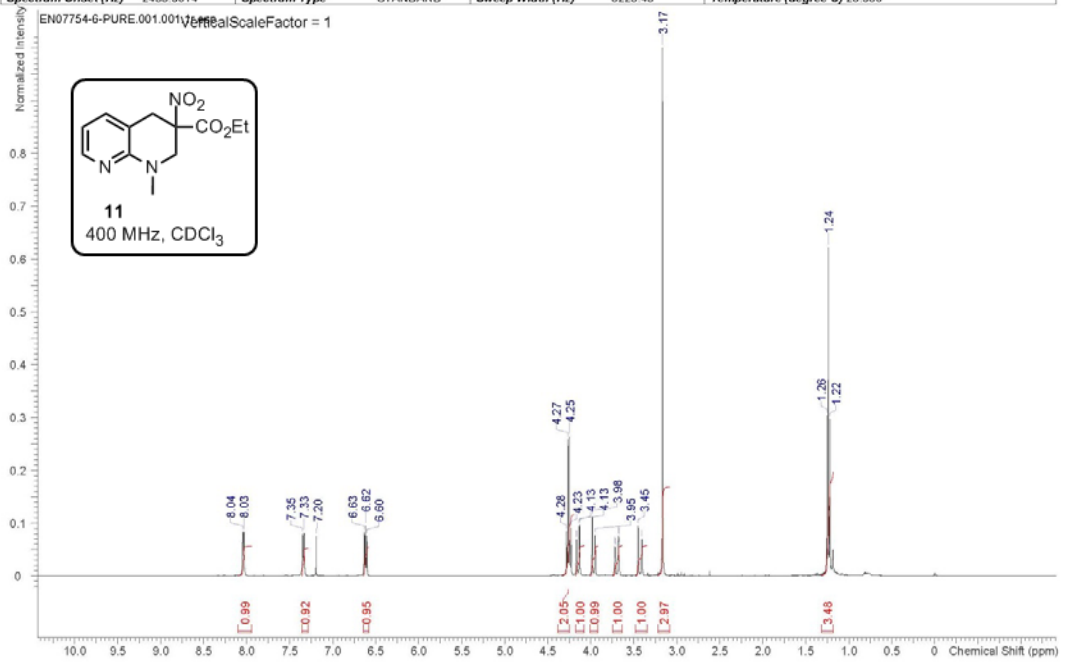
9/2/2015 4:34:43 PM

OriginalDateForRelativeTime 2015-01-13T10:49:20			*Multiplets Integrals Sum* 0.00	*Number of Nuclei* 0 H's	
Acquisition Time (sec) 0.9961	*Comment*	proton_longdelay_QNP_16 CDCl3 {C:\u} fbriones 49		*Date*	13 Jan 2015 10:49:20
Date Stamp 13 Jan 2015 10:49:20			*File Name*	C:\Users\kmwk459\Desktop\OL-NMR\crude-entry6-t1\1\pdata\1\1r	
Frequency (MHz) 400.13	*Nucleus*	1H	*Number of Transients* 16	*Origin*	spect
Original Points Count 8192	*Owner*	usbodlab	*Points Count* 32768	*Pulse Sequence*	zg30
Receiver Gain 228.10	*SW(cyclical) (Hz)*	8223.68	*Solvent*	CHLOROFORM-d	
Spectrum Offset (Hz) 2471.0088	*Spectrum Type*	STANDARD	*Sweep Width (Hz)*	8223.43	*Temperature (degree C)* 26.960



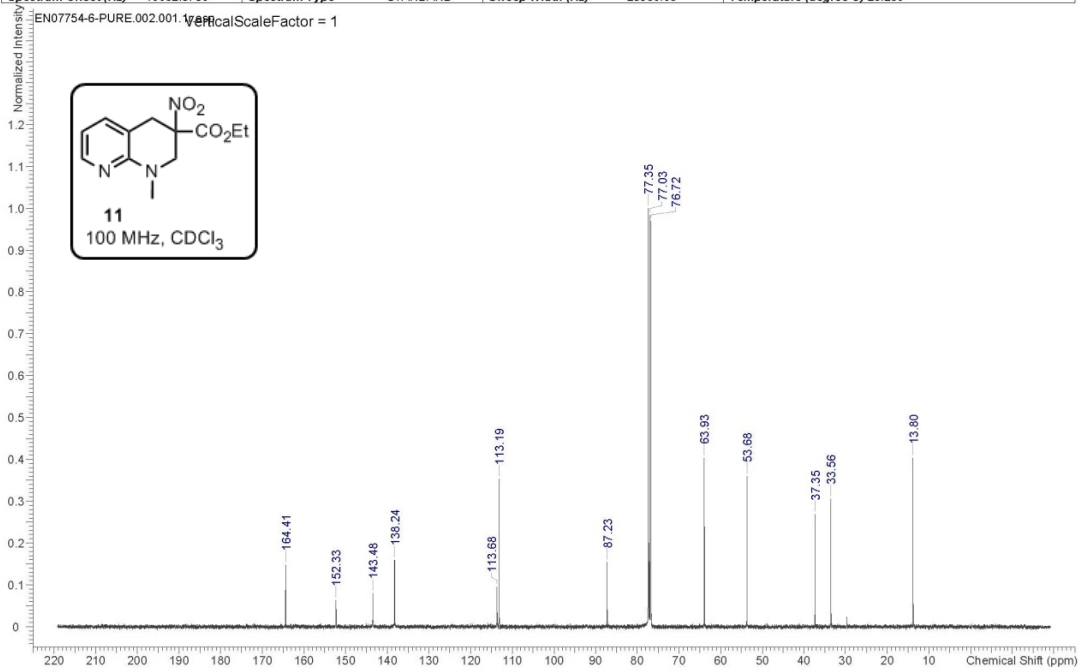
9/2/2015 4:37:28 PM

OriginalDateForRelativeTime 2015-03-06T17:02:40			*Multiplets Integrals Sum* 0.00	*Number of Nuclei* 0 H's	
Acquisition Time (sec) 0.9961	*Comment*	proton_longdelay_QNP_16 CDCl3 {C:\u} fbriones 43		*Date*	06 Mar 2015 17:02:40
Date Stamp 06 Mar 2015 17:02:40			*File Name*	C:\Users\kmwk459\Desktop\OL-NMR\EN07754-6-PURE\1\pdata\1\1r	
Frequency (MHz) 400.13	*Nucleus*	1H	*Number of Transients* 16	*Origin*	spect
Original Points Count 8192	*Owner*	usbodlab	*Points Count* 32768	*Pulse Sequence*	zg30
Receiver Gain 128.00	*SW(cyclical) (Hz)*	8223.68	*Solvent*	CHLOROFORM-d	
Spectrum Offset (Hz) 2435.9014	*Spectrum Type*	STANDARD	*Sweep Width (Hz)*	8223.43	*Temperature (degree C)* 26.960



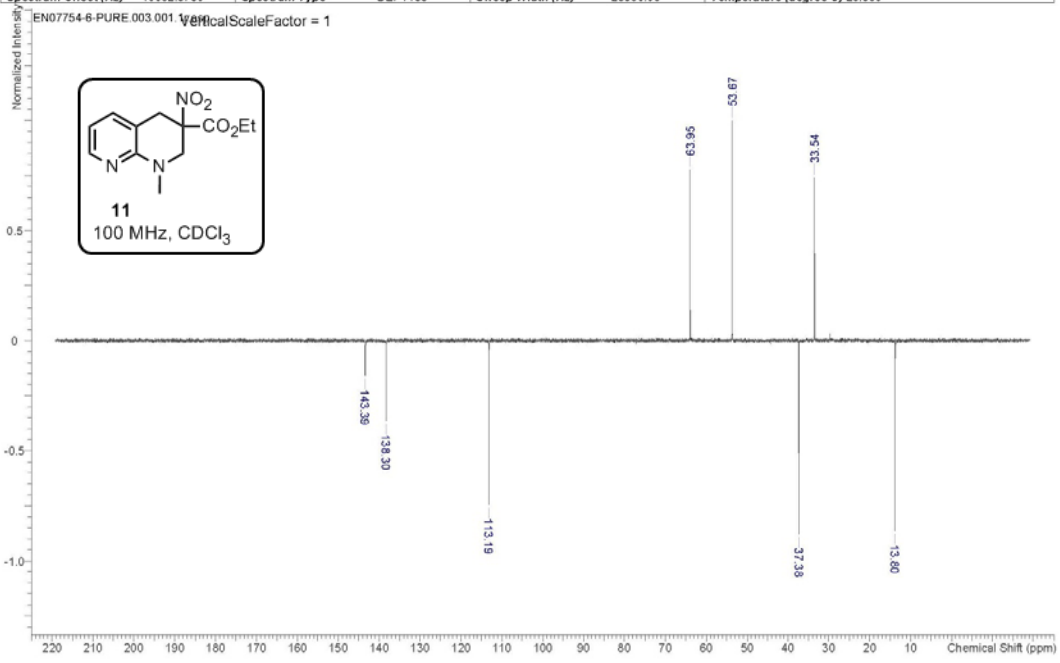
9/2/2015 4:38:11 PM

OriginalDateForRelativeTime 2015-03-06T21:48:32				***Number of Nuclei***	0 C's
Acquisition Time (sec)	1.3664	***Comment***	carbon_QNP_2500 CDCl3 {C:\u} fbriones 43	***Date***	06 Mar 2015 21:48:32
Date Stamp	06 Mar 2015 21:48:32			***File Name***	C:\Users\kmwk459\Desktop\OL-NMR\EN07754-6-PURE\2\pdata\1\1r
Frequency (MHz)	100.62	***Nucleus***	13C	***Number of Transients***	2500
Original Points Count	32768	***Owner***	usbodlab	***Points Count***	32768
Receiver Gain	16384.00	***SW(cyclical) (Hz)***	23980.81	***Solvent***	CHLOROFORM-d
Spectrum Offset (Hz)	10062.3730	***Spectrum Type***	STANDARD	***Sweep Width (Hz)***	23980.08



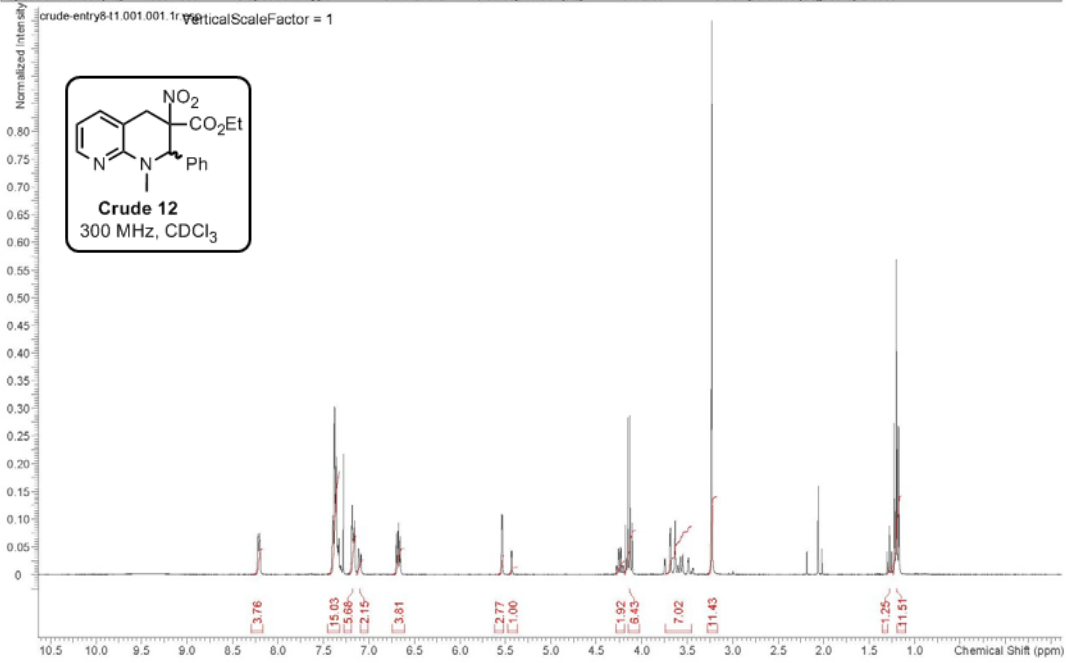
9/2/2015 4:39:04 PM

OriginalDateForRelativeTime 2015-03-08T16:05:20				***Number of Nuclei***	0 C's
Acquisition Time (sec)	1.3664	***Comment***	dept_QNP_2000 CDCl3 {C:\u} fbriones 43	***Date***	08 Mar 2015 16:05:20
Date Stamp	08 Mar 2015 16:05:20			***File Name***	C:\Users\kmwk459\Desktop\OL-NMR\EN07754-6-PURE\3\pdata\1\1r
Frequency (MHz)	100.62	***Nucleus***	13C	***Number of Transients***	2000
Original Points Count	32768	***Owner***	usbodlab	***Points Count***	32768
Receiver Gain	16384.00	***SW(cyclical) (Hz)***	23980.81	***Solvent***	CHLOROFORM-d
Spectrum Offset (Hz)	10062.3730	***Spectrum Type***	DEPT135	***Sweep Width (Hz)***	23980.08



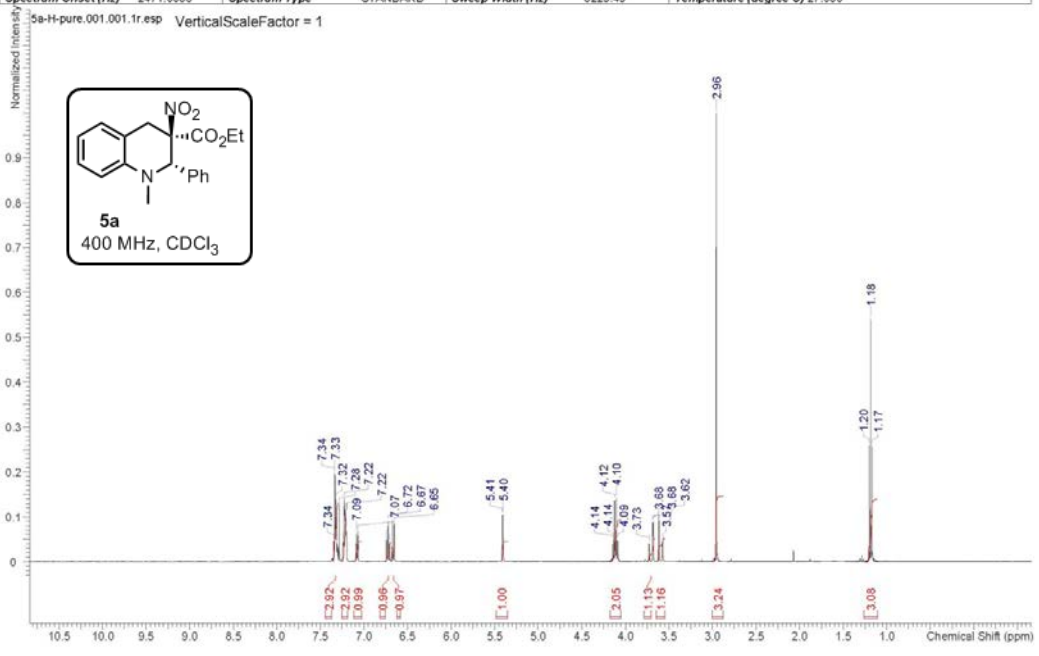
9/2/2015 4:40:12 PM

OriginalDateForRelativeTime 2015-08-19T16:01:04			*Multiplets Integrals Sum* 0.00	*Number of Nuclei* 0 H's	
Acquisition Time (sec) 2.6542	*Comment*	A3 300MHz proton_longdelay_QNP_16 CDCl3 {C:\u} fbriones 47		*Date*	19 Aug 2015 16:01:04
Date Stamp 19 Aug 2015 16:01:04			*File Name*	C:\Users\kmwk459\Desktop\OL-NMR\crude-entry8-t1\1\pdata\1\1r	
Frequency (MHz) 300.13	*Nucleus*	1H	*Number of Transients* 16	*Origin*	spect
Original Points Count 16384	*Owner*	usbodlab	*Points Count* 32768	*Pulse Sequence*	zg30
Receiver Gain 456.10	*SW(cyclical) (Hz)*	6172.84	*Solvent* CHLOROFORM-d		
Spectrum Offset (Hz) 1853.4569	*Spectrum Type*	STANDARD	*Sweep Width (Hz)* 6172.65	*Temperature (degree C)* 27.060	



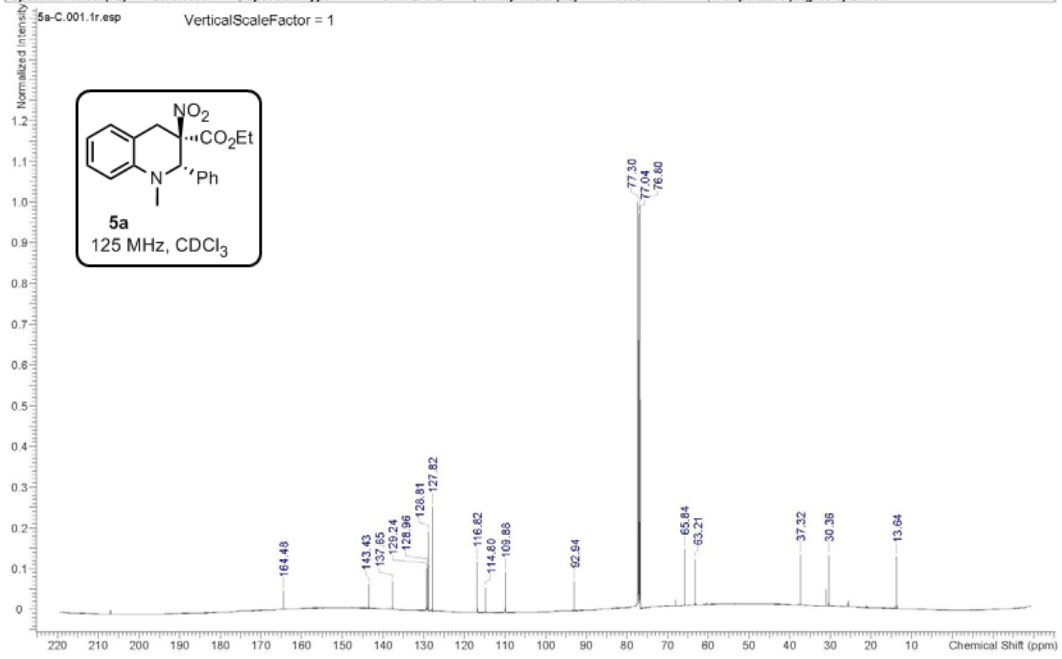
9/2/2015 4:50:21 PM

OriginalDateForRelativeTime 2014-04-03T13:33:52			*Multiplets Integrals Sum* 0.00	*Number of Nuclei* 0 H's	
Acquisition Time (sec) 0.9961	*Comment*	proton_QNP_32 CDCl3 {C:\u} fbriones 3		*Date*	03 Apr 2014 13:33:52
Date Stamp 03 Apr 2014 13:33:52			*File Name*	C:\Users\kmwk459\Desktop\OL-NMR\5a-H-pure\1\pdata\1\1r	
Frequency (MHz) 400.13	*Nucleus*	1H	*Number of Transients* 32	*Origin*	spect
Original Points Count 8192	*Owner*	usbodlab	*Points Count* 32768	*Pulse Sequence*	zg30
Receiver Gain 161.30	*SW(cyclical) (Hz)*	8223.68	*Solvent* CHLOROFORM-d		
Spectrum Offset (Hz) 2471.0088	*Spectrum Type*	STANDARD	*Sweep Width (Hz)* 8223.43	*Temperature (degree C)* 27.060	



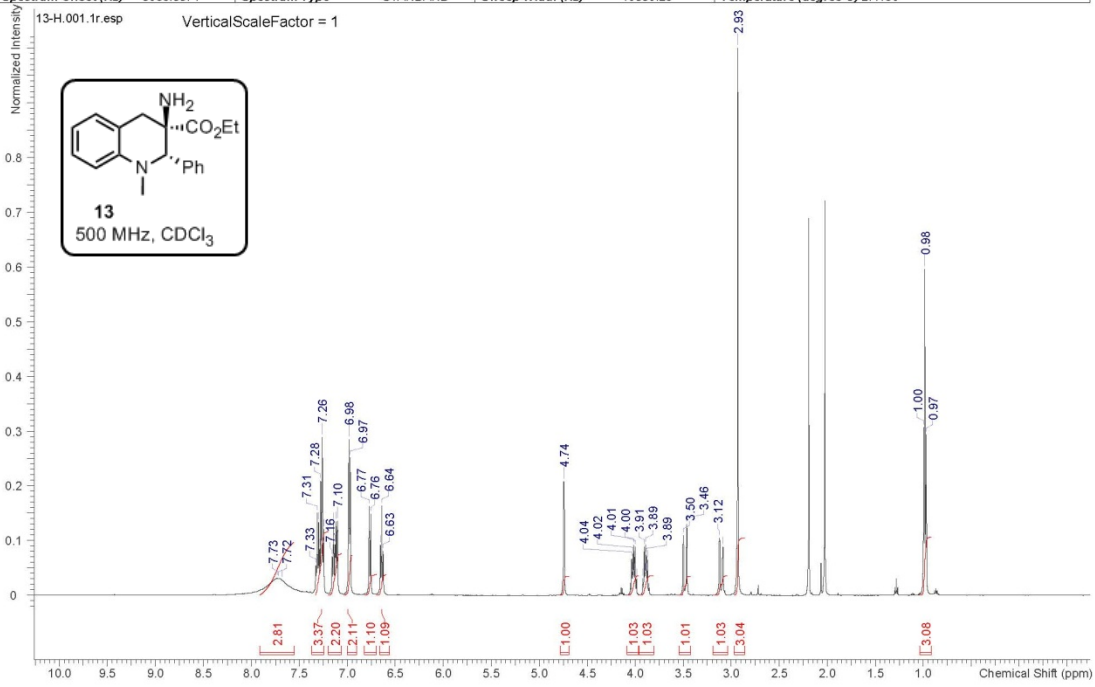
9/2/2015 4:52:08 PM

OriginalDateForRelativeTime 2015-08-19T01:17:52			*Multiplets Integrals Sum* 0.00	*Number of Nuclei*	0 C's
Acquisition Time (sec) 1.0912	*Comment*	C13CPD-long CDCl3 {C:\Bruker\TOPSPIN1.3} fbriones 53		*Date*	19 Aug 2015 01:17:52
Date Stamp 19 Aug 2015 01:17:52			*File Name*	C:\Users\kmwk459\Desktop\OL-NMR\5a-C\pdata\1\1r	
Frequency (MHz) 125.77	*Nucleus*	13C	*Number of Transients* 8192	*Origin*	spect
Original Points Count 32768	*Owner*	usbodlab	*Points Count* 32768	*Pulse Sequence*	zgpg30
Receiver Gain 4096.00	*SW(cyclical) (Hz)*	30030.03	*Solvent* CHLOROFORM-d		
Spectrum Offset (Hz) 12578.0596	*Spectrum Type*	STANDARD	*Sweep Width (Hz)* 30029.11	*Temperature (degree C)* 27.160	



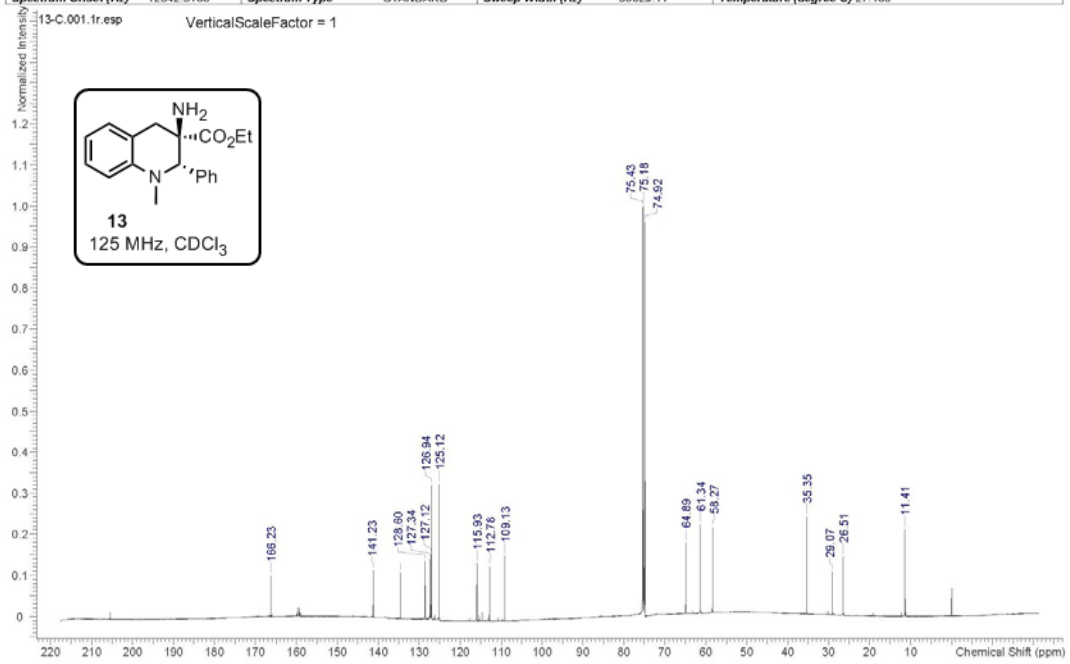
9/2/2015 4:56:34 PM

OriginalDateForRelativeTime 2015-08-24T16:48:00			*Multiplets Integrals Sum* 0.00	*Number of Nuclei*	0 H's
Acquisition Time (sec) 3.1719	*Comment*	PROTON CDCl3 {C:\Bruker\TOPSPIN1.3} fbriones 4		*Date*	24 Aug 2015 16:48:00
Date Stamp 24 Aug 2015 16:48:00			*File Name*	C:\Users\kmwk459\Desktop\OL-NMR\13-H\pdata\1\1r	
Frequency (MHz) 500.13	*Nucleus*	1H	*Number of Transients* 64	*Origin*	spect
Original Points Count 32768	*Owner*	usbodlab	*Points Count* 32768	*Pulse Sequence*	zg30
Receiver Gain 80.60	*SW(cyclical) (Hz)*	10330.58	*Solvent* CHLOROFORM-d		
Spectrum Offset (Hz) 3088.5571	*Spectrum Type*	STANDARD	*Sweep Width (Hz)* 10330.26	*Temperature (degree C)* 27.160	



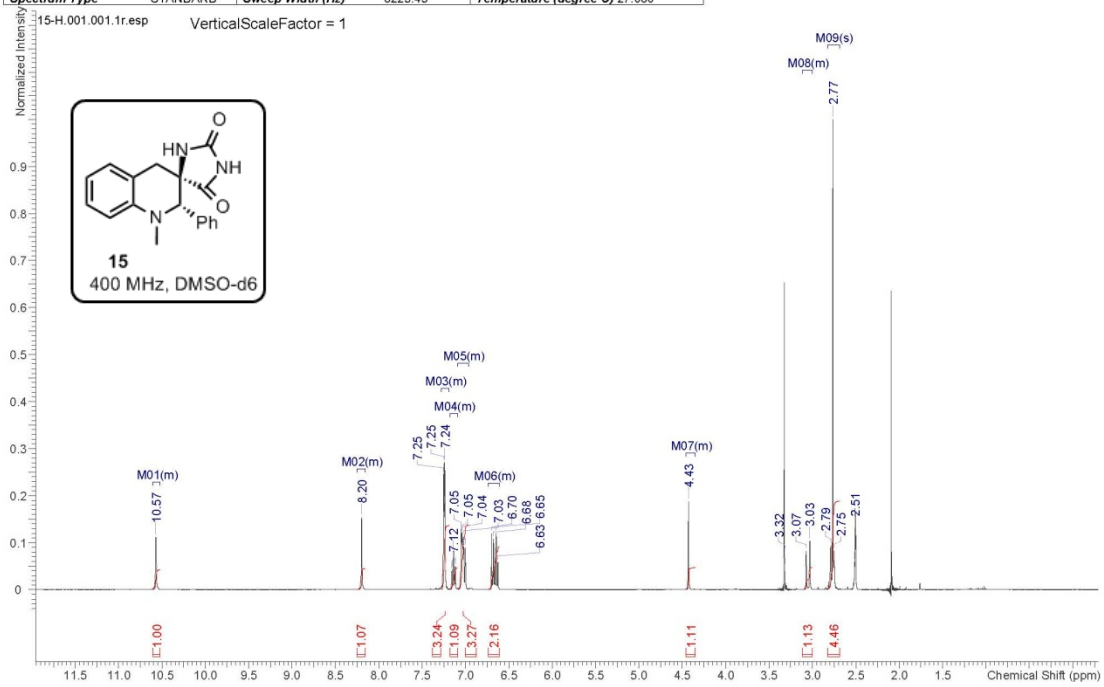
9/2/2015 4:58:50 PM

OriginalDateForRelativeTime 2015-08-25T01:17:52				***Multiplets Integrals Sum*** 0.00		***Number of Nuclei*** 0 C's	
Acquisition Time (sec)	1.0912	***Comment***	C13CPD-long CDCl3 {C:\Bruker\TOPSPIN1.3} fbriones 4			***Date***	25 Aug 2015 01:17:52
Date Stamp	25 Aug 2015 01:17:52			***File Name***	C:\Users\kmwk459\Desktop\OL-NMR\13-C\pdata\1\1r		
Frequency (MHz)	125.77	***Nucleus***	13C	***Number of Transients***	8192	***Origin***	spect
Original Points Count	32768	***Owner***	usbodlab	***Points Count***	32768	***Pulse Sequence***	zgpg30
Receiver Gain	3251.00	***SW(cyclical) (Hz)***	30030.03	***Solvent***	CHLOROFORM-d		
Spectrum Offset (Hz)	12342.5166	***Spectrum Type***	STANDARD	***Sweep Width (Hz)***	30029.11	***Temperature (degree C)***	27.160



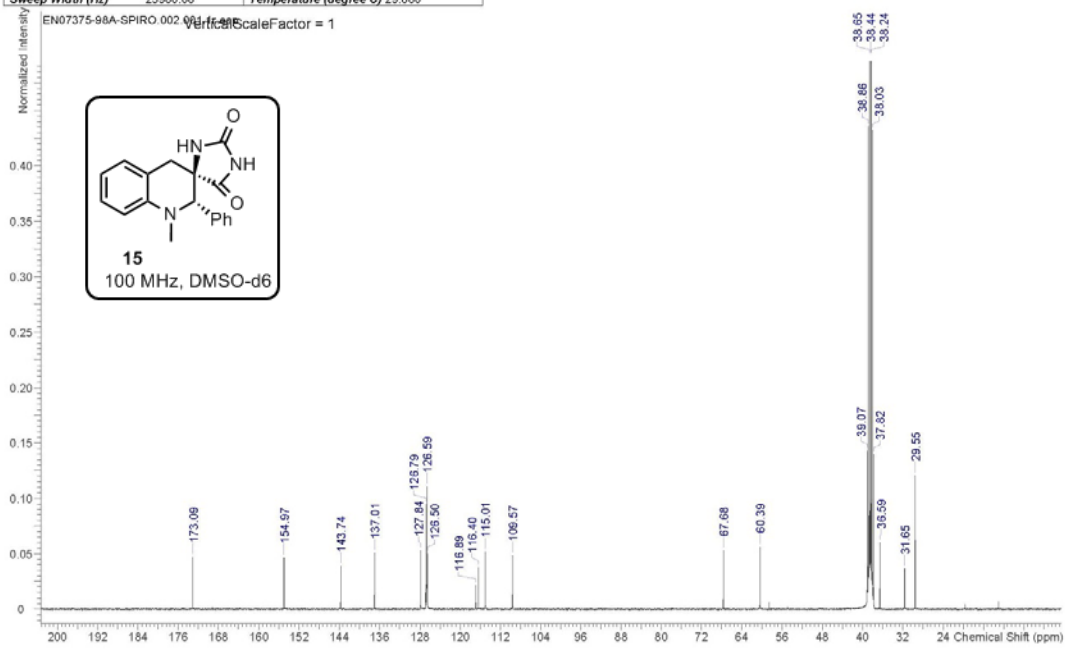
9/2/2015 5:04:29 PM

OriginalDateForRelativeTime 2014-04-28T14:59:12				***Multiplets Integrals Sum*** 18.55		***Number of Nuclei*** 17 H's	
Acquisition Time (sec)	0.9961	***Comment***	proton_QNP_128 DMSO {C:\u} fbriones 3			***Date***	28 Apr 2014 14:59:12
Date Stamp	28 Apr 2014 14:59:12			***File Name***	C:\Users\kmwk459\Desktop\OL-NMR\15-H\1\pdata\1\1r		
Frequency (MHz)	400.13	***Nucleus***	1H	***Number of Transients***	128	***Origin***	spect
Original Points Count	8192	***Owner***	usbodlab	***Points Count***	32768	***Pulse Sequence***	zg30
Receiver Gain	128.00	***SW(cyclical) (Hz)***	8223.68	***Solvent***	DMSO-d6	***Spectrum Offset (Hz)***	2471.0088
Spectrum Type	STANDARD	***Sweep Width (Hz)***	8223.43	***Temperature (degree C)***	27.060		



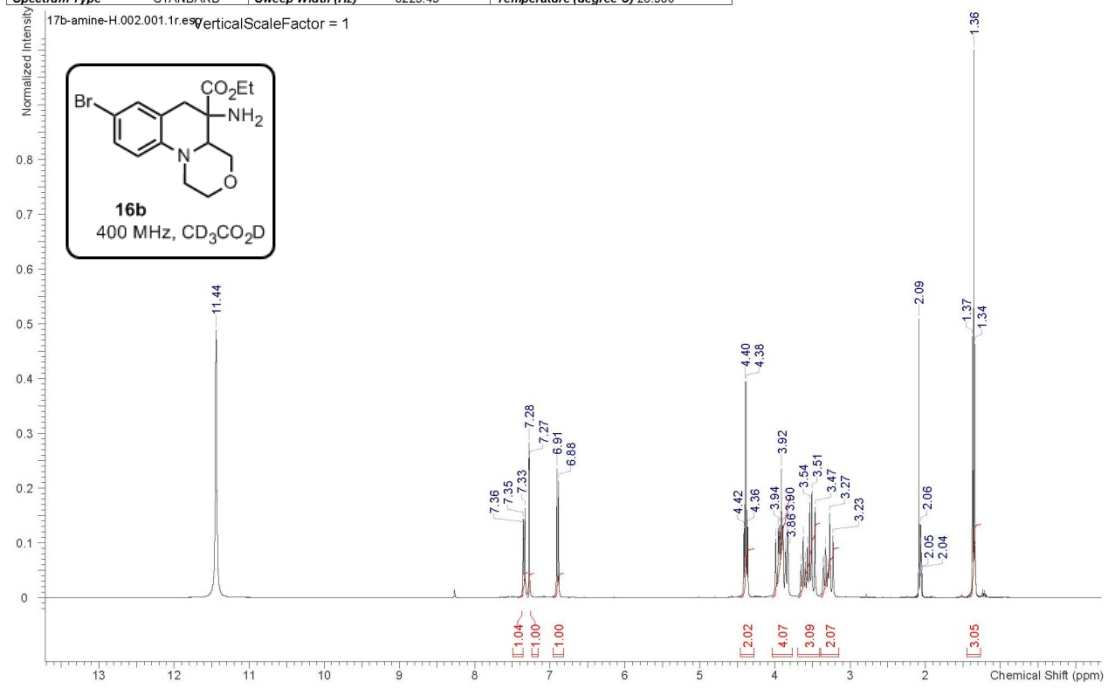
9/3/2015 10:36:07 AM

OriginalDateForRelativeTime 2014-07-12T07:01:20				*Number of Nuclei*	0 C's
Acquisition Time (sec)	1.3664	*Comment*	carbon_QNP_8000 DMSO {C:\u} fbriones 41	*Date*	12 Jul 2014 07:01:20
Date Stamp	12 Jul 2014 07:01:20				
File Name	\\netapp2\nmr_archive\GHP_C3_400\data\fbriones\nmr\EN07375-98A-SPIRO\2\pdata\1\1r			*Frequency (MHz)*	100.62
Nucleus	13C	*Number of Transients*	8000	*Origin*	spect
Owner	usbodlab	*Points Count*	32768	*Pulse Sequence*	zgpg30
SW(cyclical) (Hz)	23980.81	*Solvent*	DMSO-d6	*Spectrum Offset (Hz)*	9902.5947
Sweep Width (Hz)	23980.08	*Temperature (degree C)*	29.660		



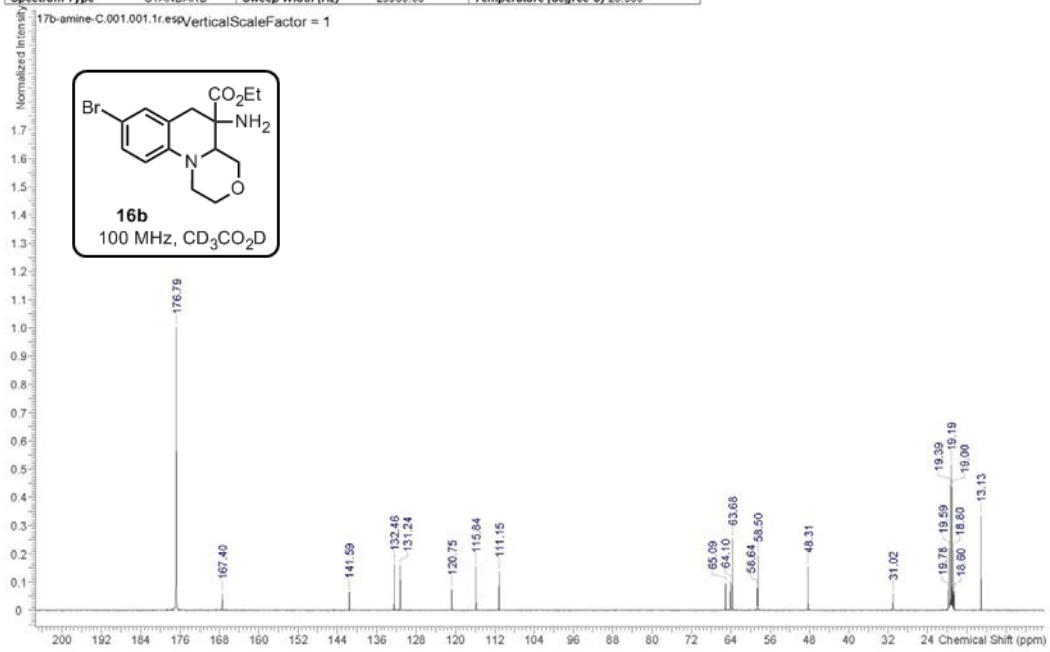
9/3/2015 12:03:55 PM

OriginalDateForRelativeTime 2014-04-28T14:35:44				*Multiplets Integrals Sum* 0.00	*Number of Nuclei* 0 H's
Acquisition Time (sec)	0.9961	*Comment*	proton_longdelay_QNP_16 Acetic {C:\u} fbriones 30	*Date*	28 Apr 2014 14:35:44
Date Stamp	28 Apr 2014 14:35:44		*File Name*	C:\Users\kmwk459\Desktop\OL-NMR\Table 3\amines\17b-amine-H\2\pdata\1\1r	
Frequency (MHz)	400.13	*Nucleus*	1H	*Number of Transients*	16
Original Points Count	8192	*Owner*	usbodlab	*Points Count*	32768
Receiver Gain	32.00	*SW(cyclical) (Hz)*	8223.68	*Solvent*	Acetic
Spectrum Type	STANDARD	*Sweep Width (Hz)*	8223.43	*Temperature (degree C)*	26.960



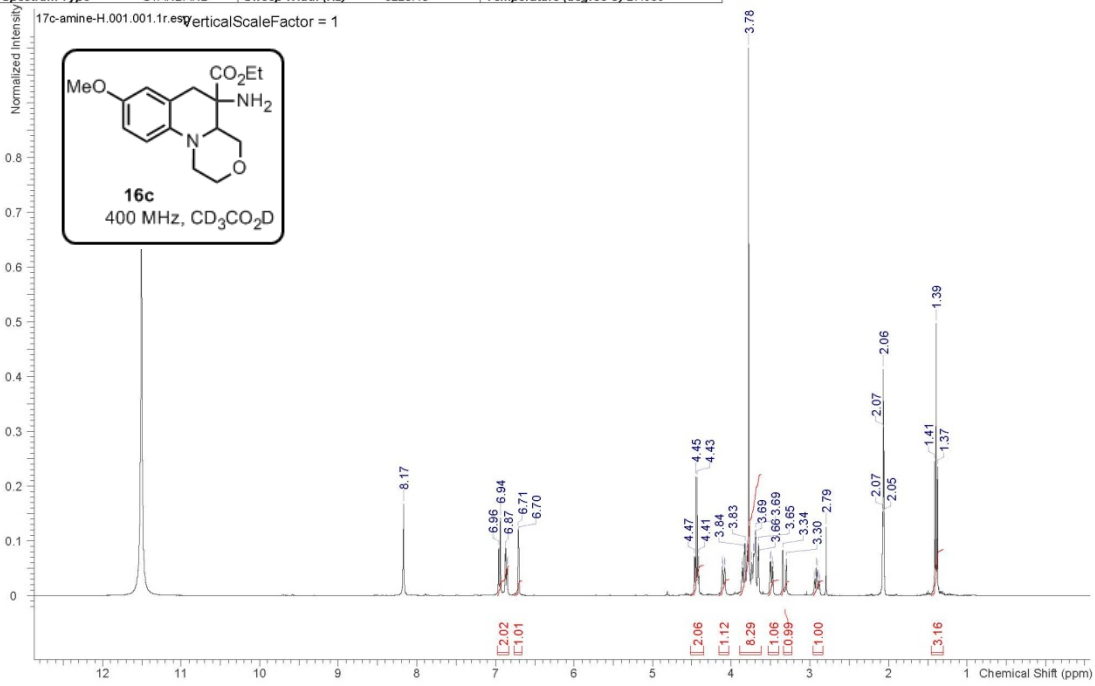
9/3/2015 12:04:33 PM

OriginalDateForRelativeTime 2014-04-28T22:22:56			*Number of Nuclei*	0 C's	
Acquisition Time (sec) 1.3664	*Comment*	carbon_QNP_2500 Acetic {C:\u} fbriones 30		*Date*	28 Apr 2014 22:22:56
Date Stamp 28 Apr 2014 22:22:56			*File Name*	C:\Users\kmwk459\Desktop\OL-NMR\Table 3\amines\17b-amine-C\1\pdata\1\1r	
Frequency (MHz) 100.62	*Nucleus*	13C	*Number of Transients*	2500	*Origin* spect
Original Points Count 32768	*Owner*	usbodlab	*Points Count*	32768	*Pulse Sequence* zgpg30
Receiver Gain 16384.00	*SW(cyclical) (Hz)*	23980.81	*Solvent*	Acetic	*Spectrum Offset (Hz)* 10062.3730
Spectrum Type STANDARD	*Sweep Width (Hz)*	23980.08	*Temperature (degree C)*	28.560	



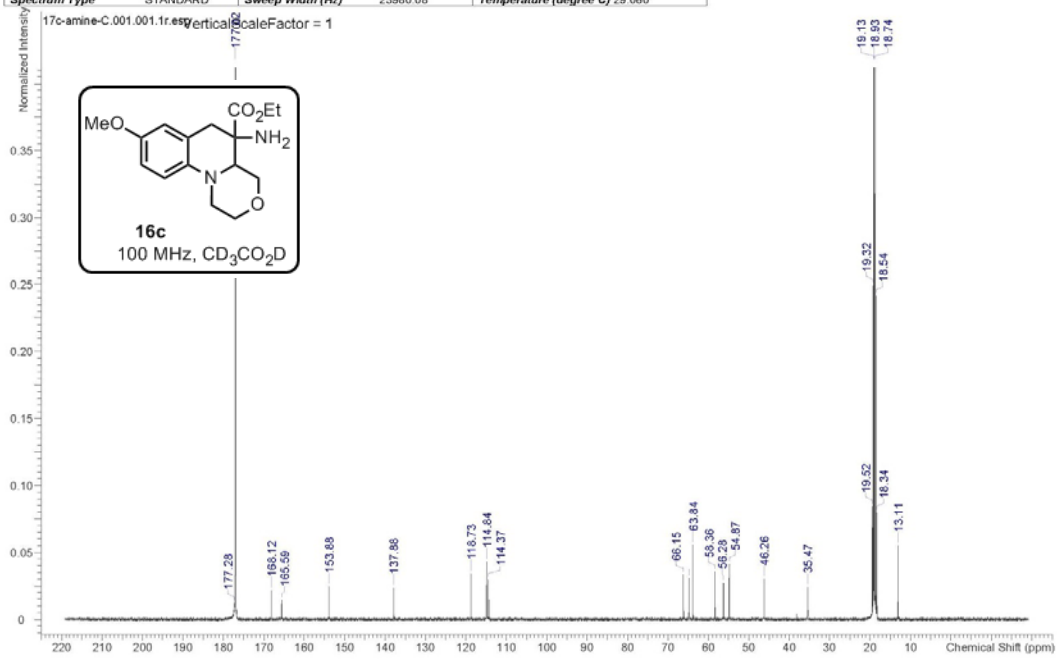
9/3/2015 12:06:02 PM

OriginalDateForRelativeTime 2014-05-07T13:48:48			*Multiplets Integrals Sum*	0.00	*Number of Nuclei*	0 H's	
Acquisition Time (sec) 0.9961	*Comment*	proton_longdelay_QNP_16 Acetic {C:\u} fbriones 13			*Date*	07 May 2014 13:48:48	
Date Stamp 07 May 2014 13:48:48			*File Name*	C:\Users\kmwk459\Desktop\OL-NMR\Table 3\amines\17c-amine-H\1\pdata\1\1r			
Frequency (MHz) 400.13	*Nucleus*	1H	*Number of Transients*	16	*Origin*	spect	
Original Points Count 8192	*Owner*	usbodlab	*Points Count*	32768	*Pulse Sequence*	zg30	
Receiver Gain 71.80	*SW(cyclical) (Hz)*	8223.68	*Solvent*	Acetic	*Spectrum Offset (Hz)*	2471.0088	
Spectrum Type STANDARD	*Sweep Width (Hz)*	8223.43	*Temperature (degree C)*	27.060			



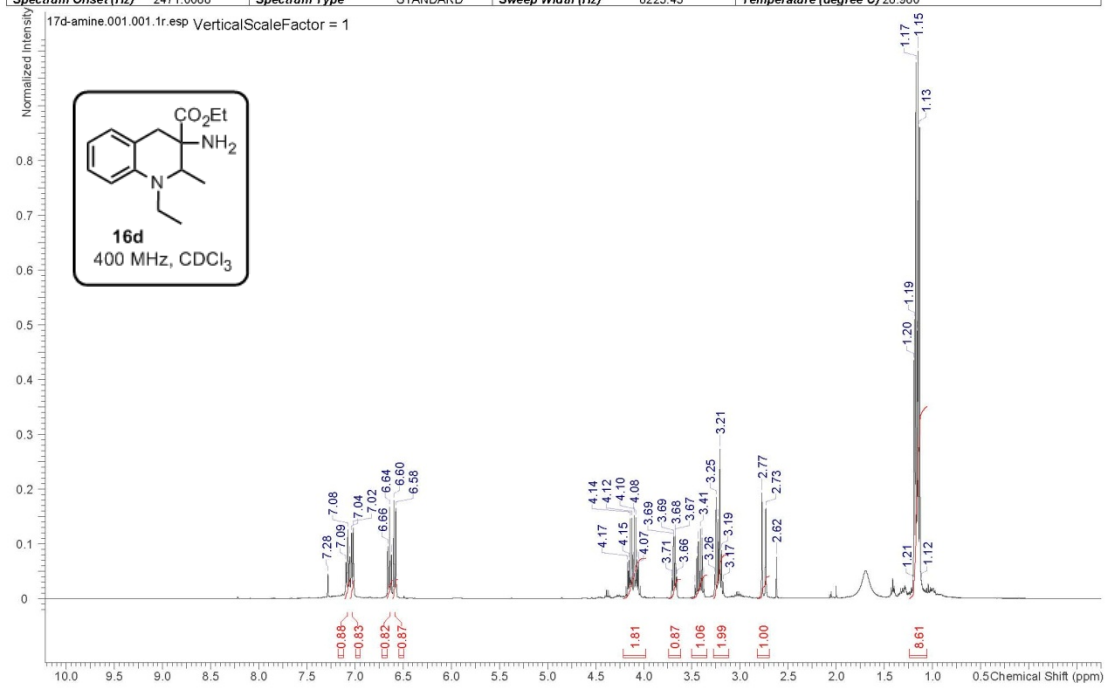
9/3/2015 12:06:50 PM

OriginalDateForRelativeTime 2014-05-07T23:37:36				*Number of Nuclei*	0 C's
Acquisition Time (sec)	1.3664	*Comment*	carbon_QNP_2500 Acetic {C:\u} fbriones 13	*Date*	07 May 2014 23:37:36
Date Stamp	07 May 2014 23:37:36			*File Name*	C:\Users\kmwk459\Desktop\OL-NMR\Table 3\amines\17c-amine-C\1\pdata\1\1r
Frequency (MHz)	100.62	*Nucleus*	13C	*Number of Transients*	2500
Original Points Count	32768	*Owner*	usbodlab	*Points Count*	32768
Receiver Gain	14596.50	*SW(cyclical) (Hz)*	23980.81	*Solvent*	Acetic
Spectrum Type	STANDARD	*Sweep Width (Hz)*	23980.08	*Temperature (degree C)*	29.060
				Origin	spect
				Pulse Sequence	zgpg30
				Spectrum Offset (Hz)	10062.3730



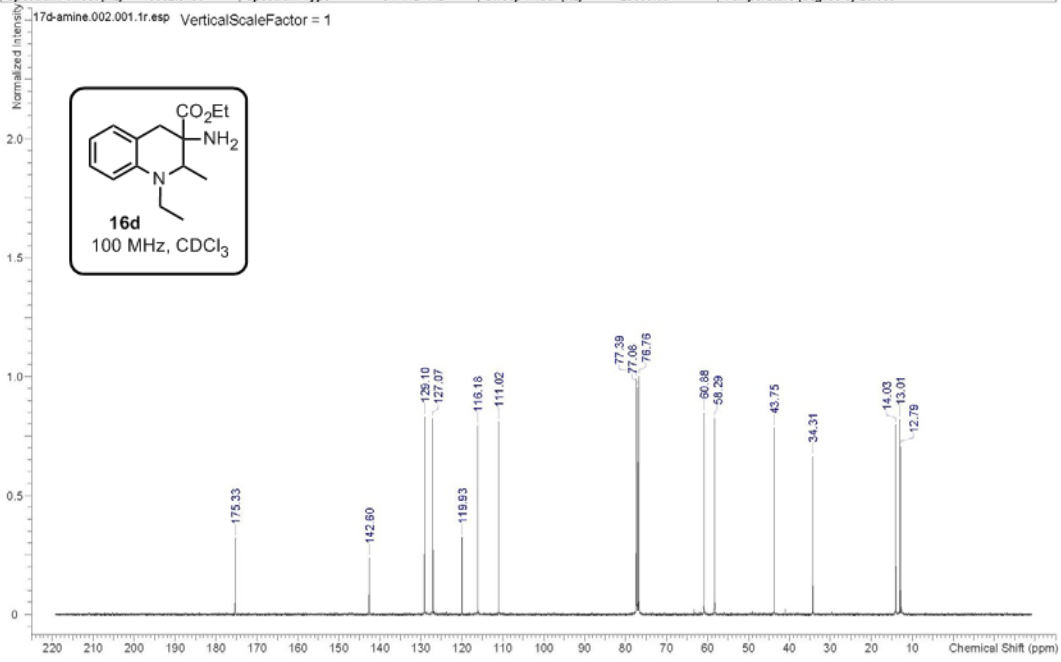
9/3/2015 12:09:17 PM

OriginalDateForRelativeTime 2014-05-21T17:32:48				*Multiplets Integrals Sum*	0.00	*Number of Nuclei*	0 H's
Acquisition Time (sec)	0.9961	*Comment*	proton_QNP_128 CDCl3 {C:\u} fbriones 45			*Date*	21 May 2014 17:32:48
Date Stamp	21 May 2014 17:32:48			*File Name*	C:\Users\kmwk459\Desktop\OL-NMR\Table 3\amines\1\pdata\1\1r		
Frequency (MHz)	400.13	*Nucleus*	1H	*Number of Transients*	128	*Origin*	spect
Original Points Count	8192	*Owner*	usbodlab	*Points Count*	32768	*Pulse Sequence*	zg30
Receiver Gain	45.30	*SW(cyclical) (Hz)*	8223.68	*Solvent*	CHLOROFORM-d		
Spectrum Offset (Hz)	2471.0088	*Spectrum Type*	STANDARD	*Sweep Width (Hz)*	8223.43	*Temperature (degree C)*	26.960



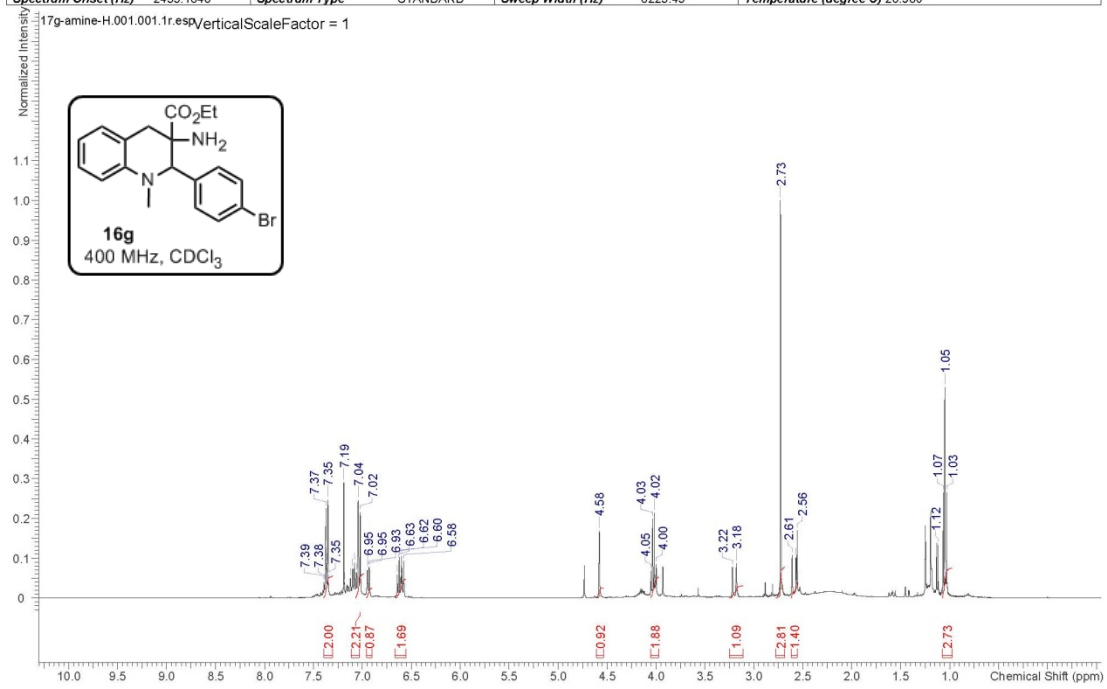
9/3/2015 12:09:49 PM

OriginalDateForRelativeTime 2014-05-22T05:27:28			*Number of Nuclei*	0 C's	
Acquisition Time (sec)	1.3664	*Comment*	carbon_QNP_2500 CDCl3 {C:\u} fbriones 45	*Date*	22 May 2014 05:27:28
Date Stamp	22 May 2014 05:27:28		*File Name*	C:\Users\kmwk459\Desktop\OL-NMR\Table 3\amines\17d-amine\2\pdata\1\1r	
Frequency (MHz)	100.62	*Nucleus*	13C	*Number of Transients*	2500	*Origin*	spect
Original Points Count	32768	*Owner*	usbodlab	*Points Count*	32768	*Pulse Sequence*	zgpg30
Receiver Gain	16384.00	*SW(cyclical) (Hz)*	23980.81	*Solvent*	CHLOROFORM-d		
Spectrum Offset (Hz)	10062.3730	*Spectrum Type*	STANDARD	*Sweep Width (Hz)*	23980.08	*Temperature (degree C)*	29.060



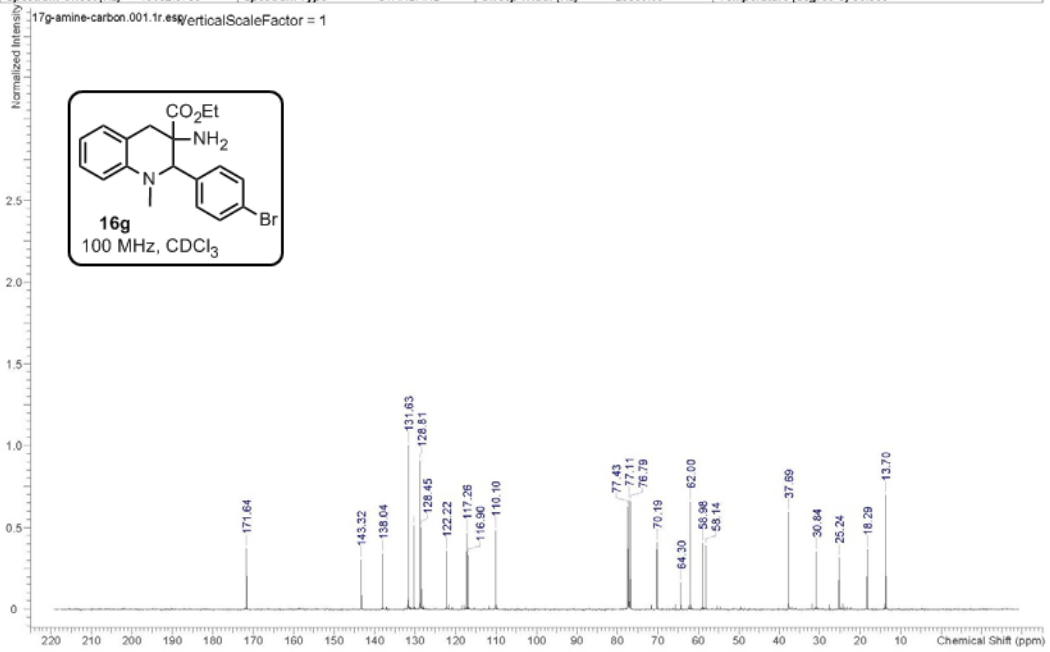
9/3/2015 12:13:18 PM

OriginalDateForRelativeTime 2014-06-16T10:36:48				*Multiplets Integrals Sum*	0.00	*Number of Nuclei*	0 H's
Acquisition Time (sec)	0.9961	*Comment*	proton_QNP_128 CDCl3 {C:\u} fbriones 36			*Date*	16 Jun 2014 10:36:48
Date Stamp	16 Jun 2014 10:36:48			*File Name*	C:\Users\kmwk459\Desktop\OL-NMR\Table 3\amines\17g-amine-H\1\pdata\1\1r		
Frequency (MHz)	400.13	*Nucleus*	1H	*Number of Transients*	128	*Origin*	spect
Original Points Count	8192	*Owner*	usbodlab	*Points Count*	32768	*Pulse Sequence*	zg30
Receiver Gain	161.30	*SW(cyclical) (Hz)*	8223.68	*Solvent*	CHLOROFORM-d		
Spectrum Offset (Hz)	2433.1646	*Spectrum Type*	STANDARD	*Sweep Width (Hz)*	8223.43	*Temperature (degree C)*	26.960



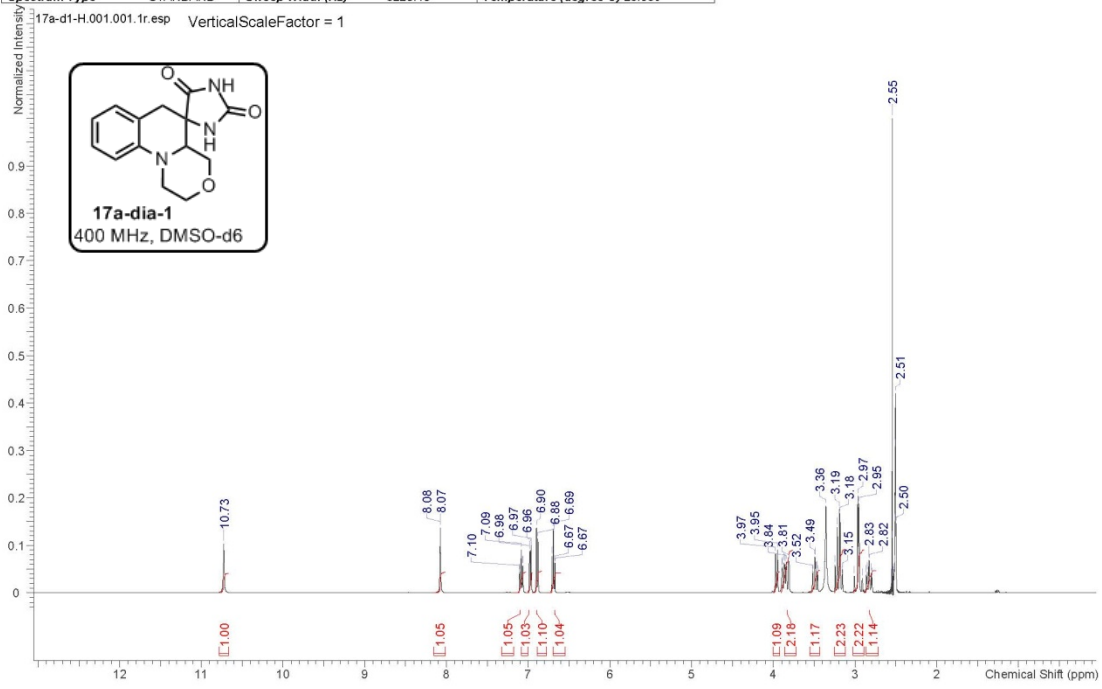
9/3/2015 12:14:06 PM

OriginalDateForRelativeTime 2014-06-16T21:48:48			*Number of Nuclei*	0 C's	
Acquisition Time (sec) 1.3664	*Comment*	carbon_QNP_2500 CDCl3 {C:\u} fbriones 24		*Date*	16 Jun 2014 21:48:48
Date Stamp 16 Jun 2014 21:48:48			*File Name*	C:\Users\kmwk459\Desktop\OL-NMR\Table 3\amines\17g-amine-carbon\pdata\1\1r	
Frequency (MHz) 100.62	*Nucleus*	13C	*Number of Transients*	2500	*Origin* spect
Original Points Count 32768	*Owner*	usbodlab	*Points Count*	32768	*Pulse Sequence* zgpg30
Receiver Gain 18390.40	*SW(cyclical) (Hz)*	23980.81	*Solvent*	CHLOROFORM-d	
Spectrum Offset (Hz) 10062.3730	*Spectrum Type*	STANDARD	*Sweep Width (Hz)*	23980.08	*Temperature (degree C)* 30.360



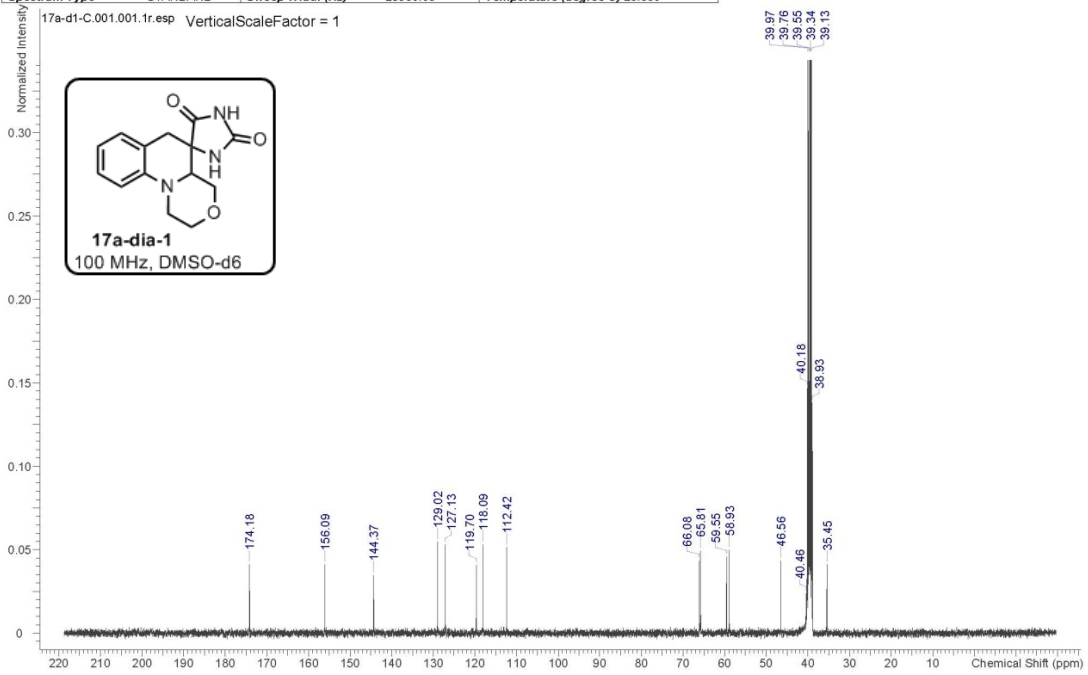
9/3/2015 1:03:01 PM

OriginalDateForRelativeTime 2014-04-30T17:02:56				*Multiplets Integrals Sum*	0.00	*Number of Nuclei*	0 H's
Acquisition Time (sec) 0.9961	*Comment*	proton_QNP_128 DMSO {C:\u} fbriones 43				*Date*	30 Apr 2014 17:02:56
Date Stamp 30 Apr 2014 17:02:56				*File Name*	C:\Users\kmwk459\Desktop\OL-NMR\Table 3\17a-d1-H\1\pdata\1\1r		
Frequency (MHz) 400.13	*Nucleus*	1H		*Number of Transients*	128	*Origin*	spect
Original Points Count 8192	*Owner*	usbodlab		*Points Count*	32768	*Pulse Sequence*	zg30
Receiver Gain 181.00	*SW(cyclical) (Hz)*	8223.68		*Solvent*	DMSO-d6	*Spectrum Offset (Hz)*	2471.0088
Spectrum Type STANDARD	*Sweep Width (Hz)*	8223.43		*Temperature (degree C)* 26.960			



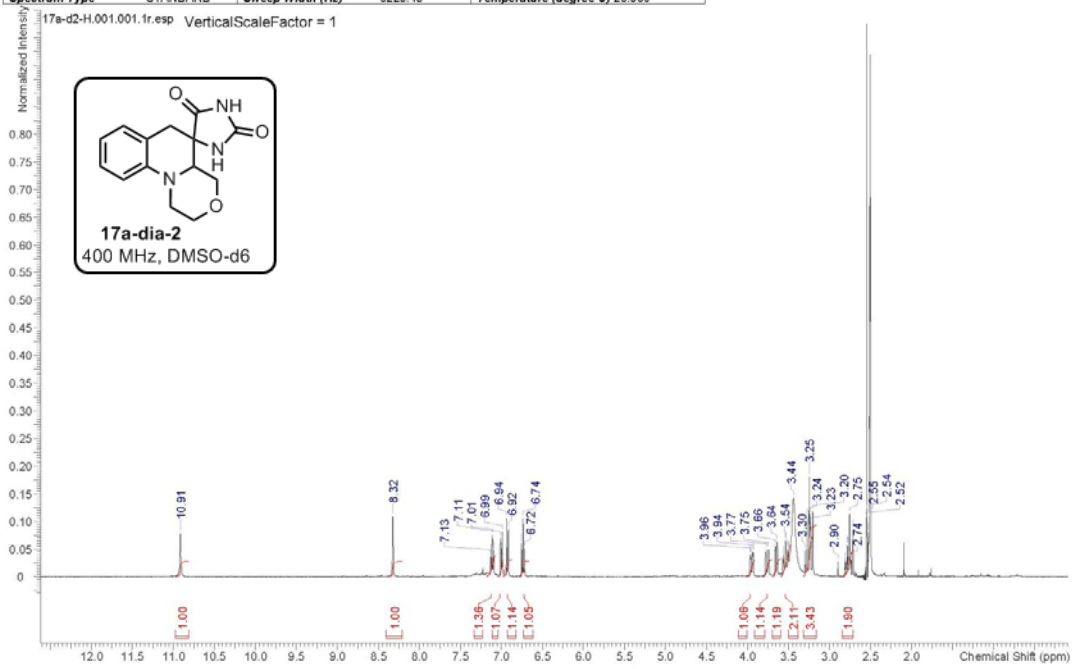
9/3/2015 1:03:36 PM

OriginalDateForRelativeTime 2014-05-01T01:26:24				*Number of Nuclei*	0 C's		
Acquisition Time (sec)	1.3664	*Comment*	carbon_QNP_2500 DMSO {C:\u} fbriones 18			*Date*	01 May 2014 01:26:24
Date Stamp	01 May 2014 01:26:24			*File Name*	C:\Users\kmwk459\Desktop\OL-NMR\Table 3\17a-d1-C\1\pdata\1\1r		
Frequency (MHz)	100.62	*Nucleus*	13C	*Number of Transients*	2500	*Origin*	spect
Original Points Count	32768	*Owner*	usbodlab	*Points Count*	32768	*Pulse Sequence*	zgpg30
Receiver Gain	8192.00	*SW(cyclical) (Hz)*	23980.81	*Solvent*	DMSO-d6	*Spectrum Offset (Hz)*	10012.0518
Spectrum Type	STANDARD	*Sweep Width (Hz)*	23980.08	*Temperature (degree C)*	28.860		



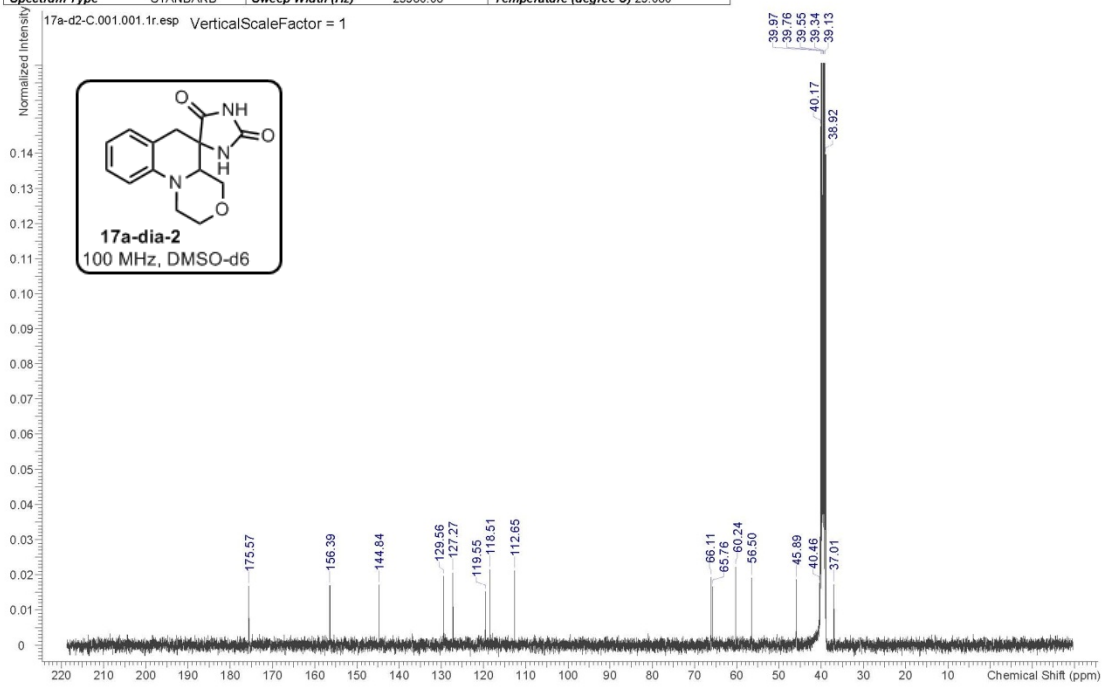
9/3/2015 1:07:48 PM

OriginalDateForRelativeTime 2014-05-01T16:24:32				*Multiplets Integrals Sum*	0.00	*Number of Nuclei*	0 H's
Acquisition Time (sec)	0.9961	*Comment*	proton_longdelay_QNP_16 DMSO {C:\u} fbriones 52			*Date*	01 May 2014 16:24:32
Date Stamp	01 May 2014 16:24:32			*File Name*	C:\Users\kmwk459\Desktop\OL-NMR\Table 3\17a-d2-H\1\pdata\1\1r		
Frequency (MHz)	400.13	*Nucleus*	1H	*Number of Transients*	16	*Origin*	spect
Original Points Count	8192	*Owner*	usbodlab	*Points Count*	32768	*Pulse Sequence*	zg30
Receiver Gain	181.00	*SW(cyclical) (Hz)*	8223.68	*Solvent*	DMSO-d6	*Spectrum Offset (Hz)*	2471.0088
Spectrum Type	STANDARD	*Sweep Width (Hz)*	8223.43	*Temperature (degree C)*	26.960		



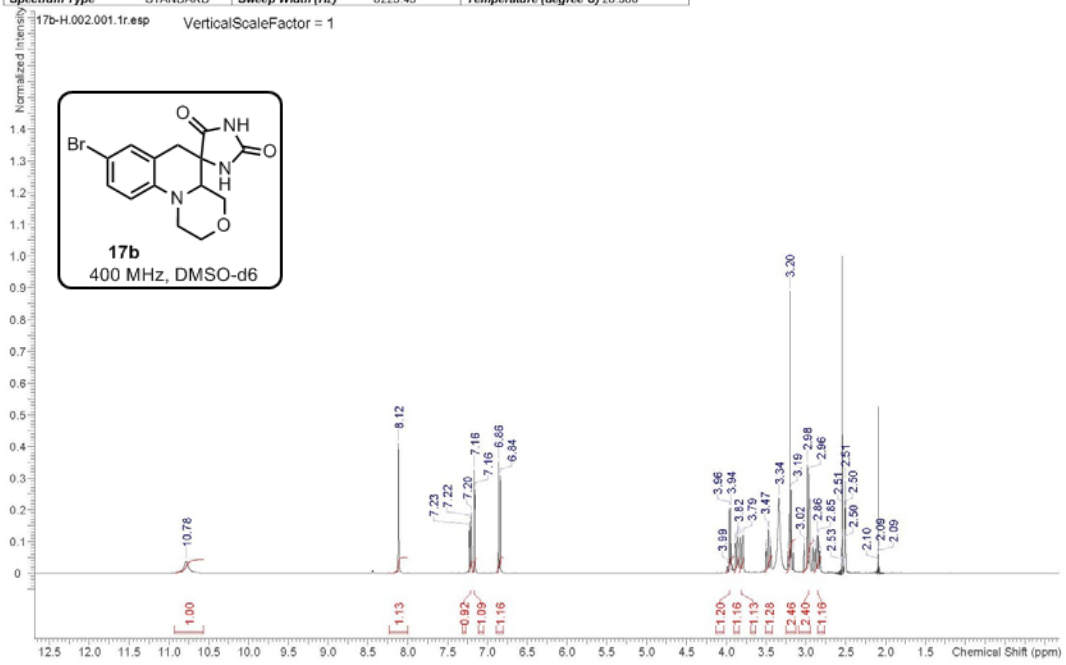
9/3/2015 1:08:49 PM

OriginalDateForRelativeTime	2014-05-01T21:46:40			*Number of Nuclei*	0 C's		
Acquisition Time (sec)	1.3664	*Comment*	carbon_QNP_2500 DMSO {C:\u} fbriones 52			*Date*	01 May 2014 21:46:40
Date Stamp	01 May 2014 21:46:40			*File Name*	C:\Users\kmwk459\Desktop\OL-NMR\Table 3\17a-d2-C\1\pdata\1\1r		
Frequency (MHz)	100.62	*Nucleus*	13C	*Number of Transients*	2500	*Origin*	spect
Original Points Count	32768	*Owner*	usbodlab	*Points Count*	32768	*Pulse Sequence*	zgpg30
Receiver Gain	9195.20	*SW(cyclical) (Hz)*	23980.81	*Solvent*	DMSO-d6	*Spectrum Offset (Hz)*	10012.0518
Spectrum Type	STANDARD	*Sweep Width (Hz)*	23980.08	*Temperature (degree C)*	29.060		



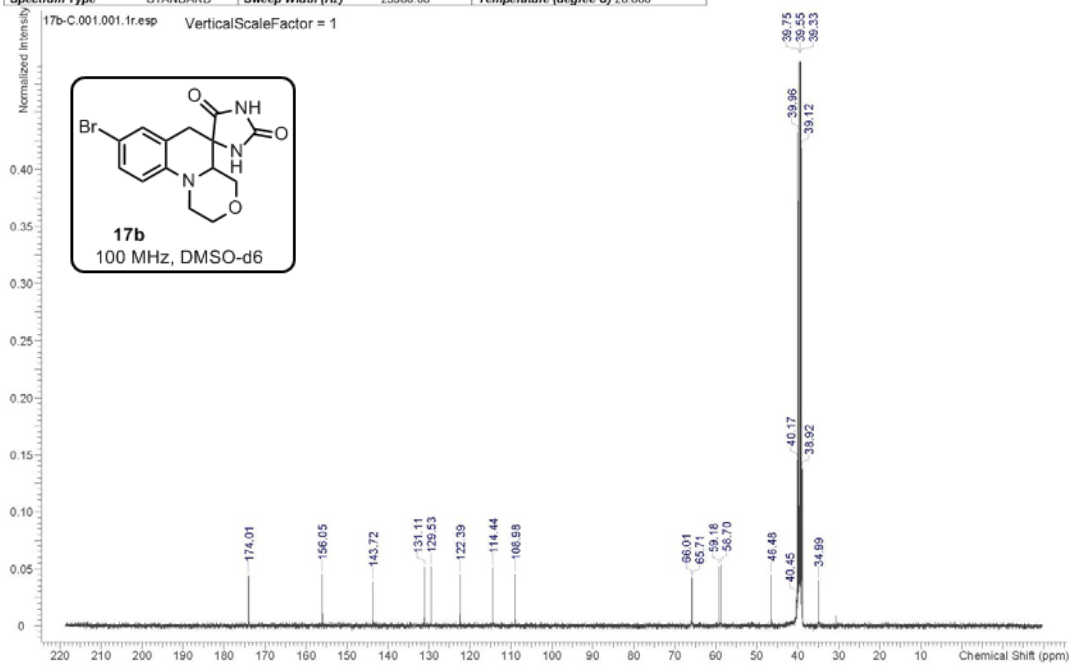
9/3/2015 1:12:34 PM

OriginalDateForRelativeTime	2014-05-15T16:28:48			*Multiplets Integrals Sum*	0.00	*Number of Nuclei*	0 H's
Acquisition Time (sec)	0.9961	*Comment*	proton_QNP_128 DMSO {C:\u} fbriones 43			*Date*	15 May 2014 16:28:48
Date Stamp	15 May 2014 16:28:48			*File Name*	C:\Users\kmwk459\Desktop\OL-NMR\Table 3\17b-H\2\pdata\1\1r		
Frequency (MHz)	400.13	*Nucleus*	1H	*Number of Transients*	128	*Origin*	spect
Original Points Count	8192	*Owner*	usbodlab	*Points Count*	32768	*Pulse Sequence*	zg30
Receiver Gain	161.30	*SW(cyclical) (Hz)*	8223.68	*Solvent*	DMSO-d6	*Spectrum Offset (Hz)*	2471.0088
Spectrum Type	STANDARD	*Sweep Width (Hz)*	8223.43	*Temperature (degree C)*	26.960		



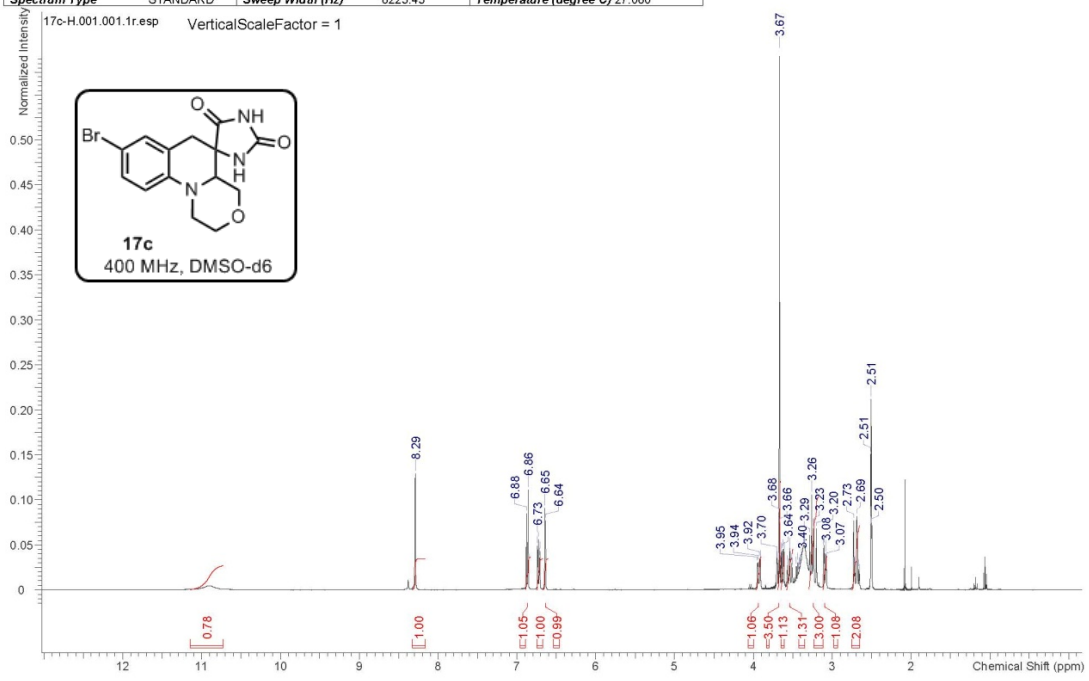
9/3/2015 1:13:21 PM

OriginalDateForRelativeTime 2014-05-15T23:37:36				*Number of Nuclei*	0 C's
Acquisition Time (sec)	1.3664	*Comment*	carbon_QNP_2500 DMSO {C:\u} fbriones 43	*Date*	15 May 2014 23:37:36
Date Stamp	15 May 2014 23:37:36		*File Name*	C:\Users\kmwk459\Desktop\OL-NMR\Table 3\17b-C\1\pdata\1\1r	
Frequency (MHz)	100.62	*Nucleus*	13C	*Number of Transients*	2500	*Origin*	spect
Original Points Count	32768	*Owner*	usbodlab	*Points Count*	32768	*Pulse Sequence*	zgpg30
Receiver Gain	6502.00	*SW(cyclical) (Hz)*	23980.81	*Solvent*	DMSO-d6	*Spectrum Offset (Hz)*	10012.0518
Spectrum Type	STANDARD	*Sweep Width (Hz)*	23980.08	*Temperature (degree C)*	28.860		



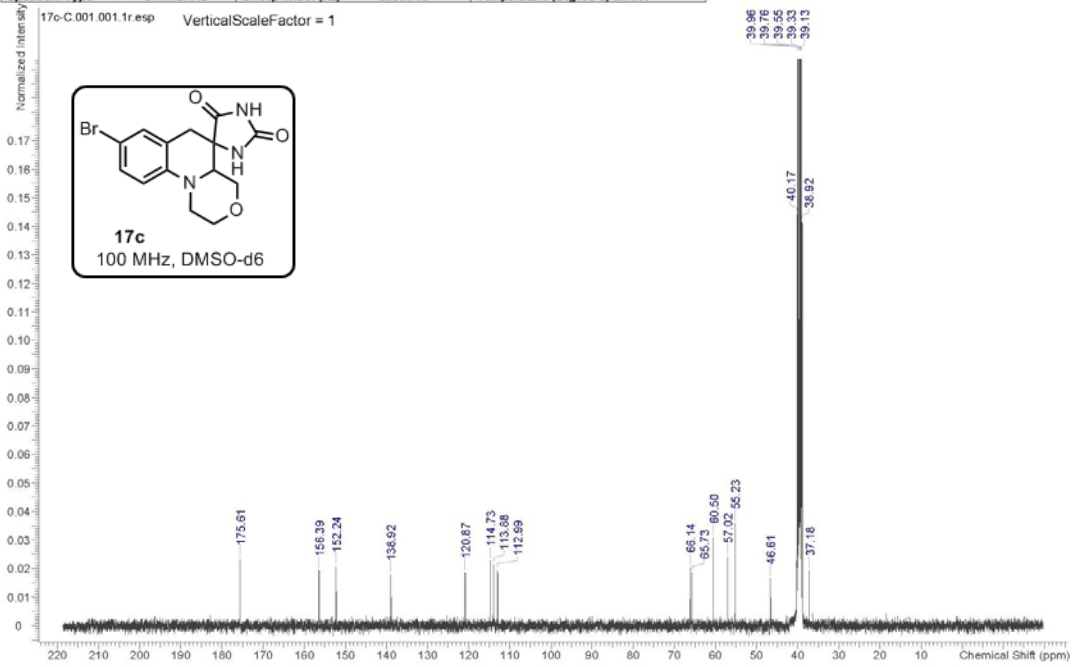
9/3/2015 1:20:08 PM

OriginalDateForRelativeTime 2014-05-16T10:15:28				*Multiplets Integrals Sum*	0.00	*Number of Nuclei*	0 H's
Acquisition Time (sec)	0.9961	*Comment*	proton_QNP_128 DMSO {C:\u} fbriones 3			*Date*	16 May 2014 10:15:28
Date Stamp	16 May 2014 10:15:28			*File Name*	C:\Users\kmwk459\Desktop\OL-NMR\Table 3\17c-H\1\pdata\1\1r		
Frequency (MHz)	400.13	*Nucleus*	1H	*Number of Transients*	128	*Origin*	spect
Original Points Count	8192	*Owner*	usbodlab	*Points Count*	32768	*Pulse Sequence*	zg30
Receiver Gain	181.00	*SW(cyclical) (Hz)*	8223.68	*Solvent*	DMSO-d6	*Spectrum Offset (Hz)*	2471.0088
Spectrum Type	STANDARD	*Sweep Width (Hz)*	8223.43	*Temperature (degree C)*	27.060		



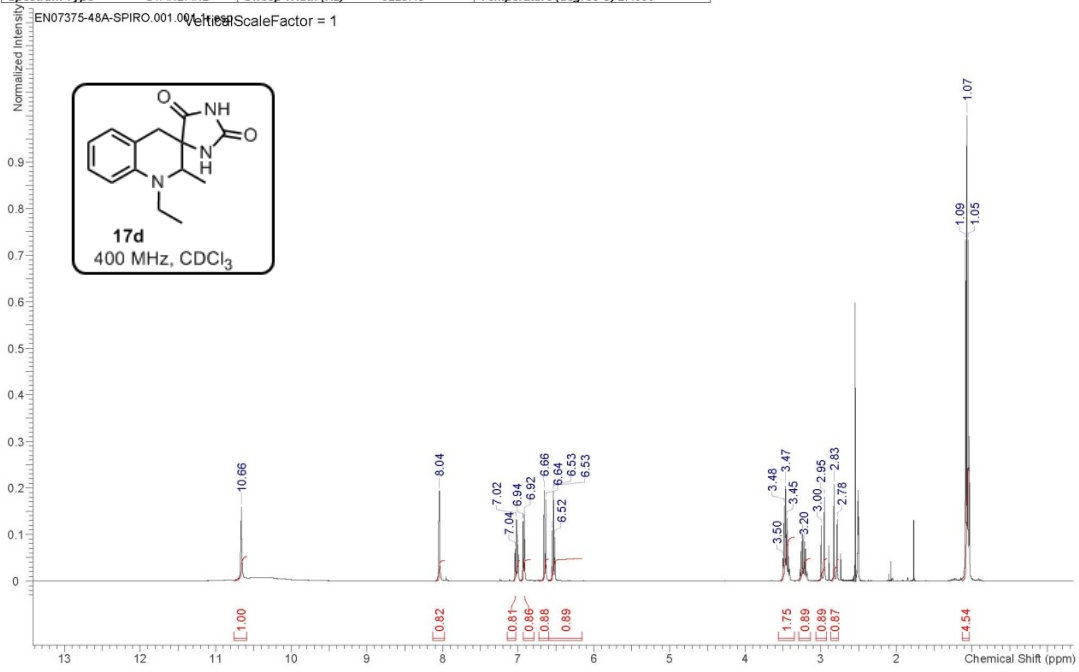
9/3/2015 1:14:20 PM

OriginalDateForRelativeTime 2014-05-16T21:48:48				*Number of Nuclei*	0 C's
Acquisition Time (sec)	1.3664	*Comment*	carbon_QNP_2500 DMSO {C:\u} fbriones 3	*Date*	16 May 2014 21:48:48
Date Stamp	16 May 2014 21:48:48		*File Name*	C:\Users\kmwk459\Desktop\OL-NMR\Table 3\17c-C\1\pdata\1\1r	
Frequency (MHz)	100.62	*Nucleus*	13C	*Number of Transients*	2500	*Origin*	spect
Original Points Count	32768	*Owner*	usbodlab	*Points Count*	32768	*Pulse Sequence*	zgpg30
Receiver Gain	13004.00	*SW(cyclical) (Hz)*	23980.81	*Solvent*	DMSO-d6	*Spectrum Offset (Hz)*	10012.0518
Spectrum Type	STANDARD	*Sweep Width (Hz)*	23980.08	*Temperature (degree C)*	28.860		



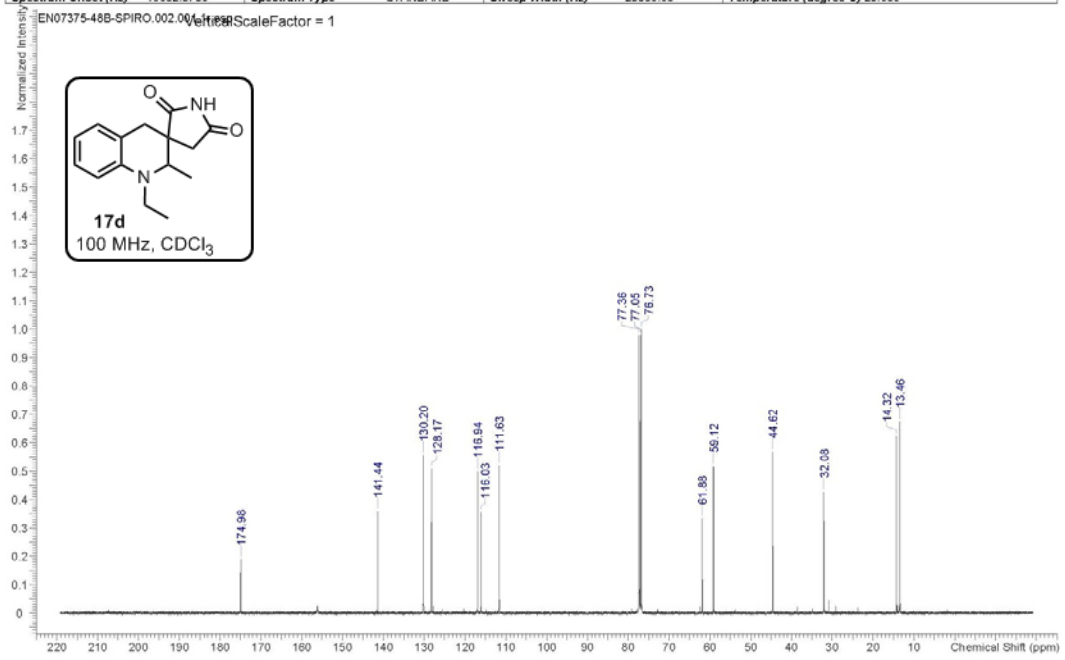
9/3/2015 1:27:40 PM

OriginalDateForRelativeTime 2014-06-04T11:00:16				*Multiplets Integrals Sum*	0.00	*Number of Nuclei*	0 H's
Acquisition Time (sec)	0.9961	*Comment*	proton_QNP_128 DMSO {C:\u} fbriones 40			*Date*	04 Jun 2014 11:00:16
Date Stamp	04 Jun 2014 11:00:16			*File Name*	C:\Users\kmwk459\Desktop\OL-NMR\Table 3\EN07375-48A-SPIRO\1\pdata\1\1r		
Frequency (MHz)	400.13	*Nucleus*	1H	*Number of Transients*	128	*Origin*	spect
Original Points Count	8192	*Owner*	usbodlab	*Points Count*	32768	*Pulse Sequence*	zg30
Receiver Gain	90.50	*SW(cyclical) (Hz)*	8223.68	*Solvent*	DMSO-d6	*Spectrum Offset (Hz)*	2471.0088
Spectrum Type	STANDARD	*Sweep Width (Hz)*	8223.43	*Temperature (degree C)*	27.060		



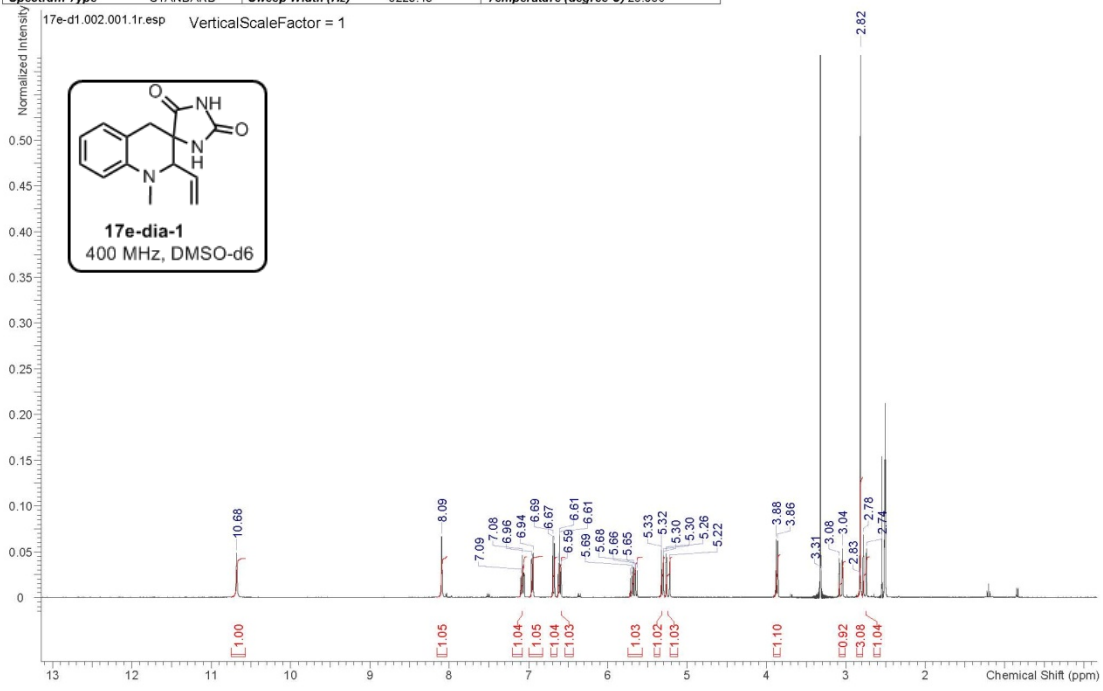
9/3/2015 1:28:26 PM

OriginalDateForRelativeTime 2014-06-04T21:48:48			*Number of Nuclei*	0 C's	
Acquisition Time (sec) 1.3664	*Comment*	carbon_QNP_2500 CDCl3 {C:\u} fbriones 42		*Date*	04 Jun 2014 21:48:48
Date Stamp 04 Jun 2014 21:48:48			*File Name*	C:\Users\kmwk459\Desktop\OL-NMR\Table 3\EN07375-48B-SPIRO\2\pdata\1\1r	
Frequency (MHz) 100.62	*Nucleus*	13C	*Number of Transients*	2500	*Origin* spect
Original Points Count 32768	*Owner*	usbodlab	*Points Count*	32768	*Pulse Sequence* zgpg30
Receiver Gain 16384.00	*SW(cyclical) (Hz)*	23980.81	*Solvent*	CHLOROFORM-d	
Spectrum Offset (Hz) 10062.3730	*Spectrum Type*	STANDARD	*Sweep Width (Hz)*	23980.08	*Temperature (degree C)* 29.060



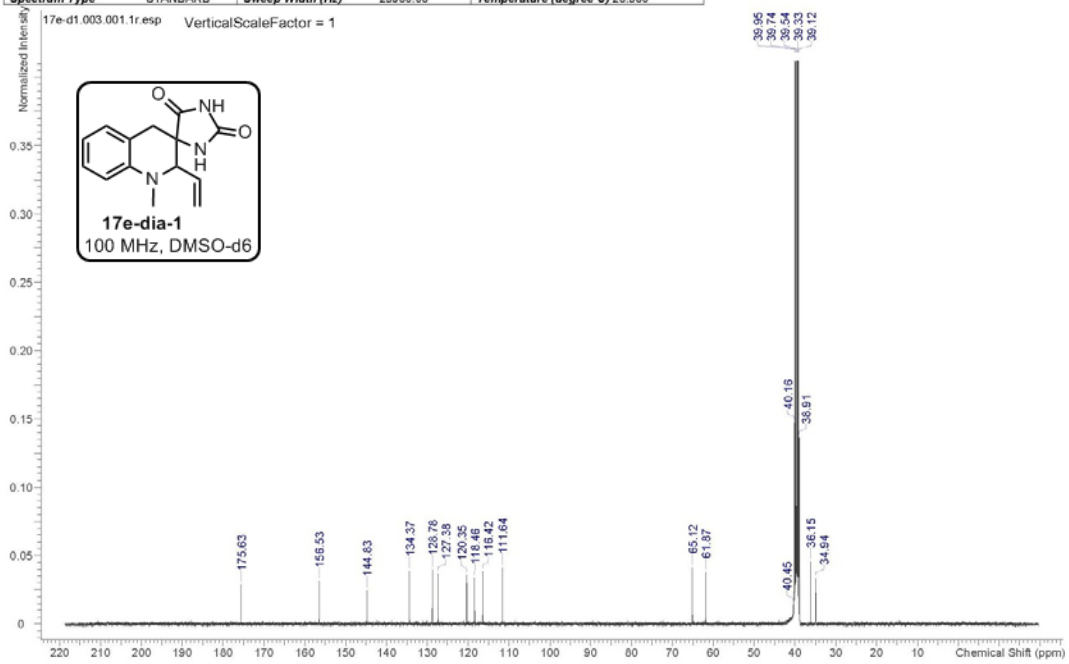
9/3/2015 1:39:17 PM

OriginalDateForRelativeTime 2015-02-20T09:53:52			*Multiplets Integrals Sum*	0.00	*Number of Nuclei* 0 H's
Acquisition Time (sec) 0.9961	*Comment*	proton_longdelay_QNP_16 DMSO {C:\u} fbriones 1		*Date*	20 Feb 2015 09:53:52
Date Stamp 20 Feb 2015 09:53:52			*File Name*	C:\Users\kmwk459\Desktop\OL-NMR\Table 3\17e-d1\2\pdata\1\1r	
Frequency (MHz) 400.13	*Nucleus*	1H	*Number of Transients*	16	*Origin* spect
Original Points Count 8192	*Owner*	usbodlab	*Points Count*	32768	*Pulse Sequence* zg30
Receiver Gain 181.00	*SW(cyclical) (Hz)*	8223.68	*Solvent*	DMSO-d6	*Spectrum Offset (Hz)* 2471.0088
Spectrum Type STANDARD	*Sweep Width (Hz)*	8223.43	*Temperature (degree C)*	26.960	



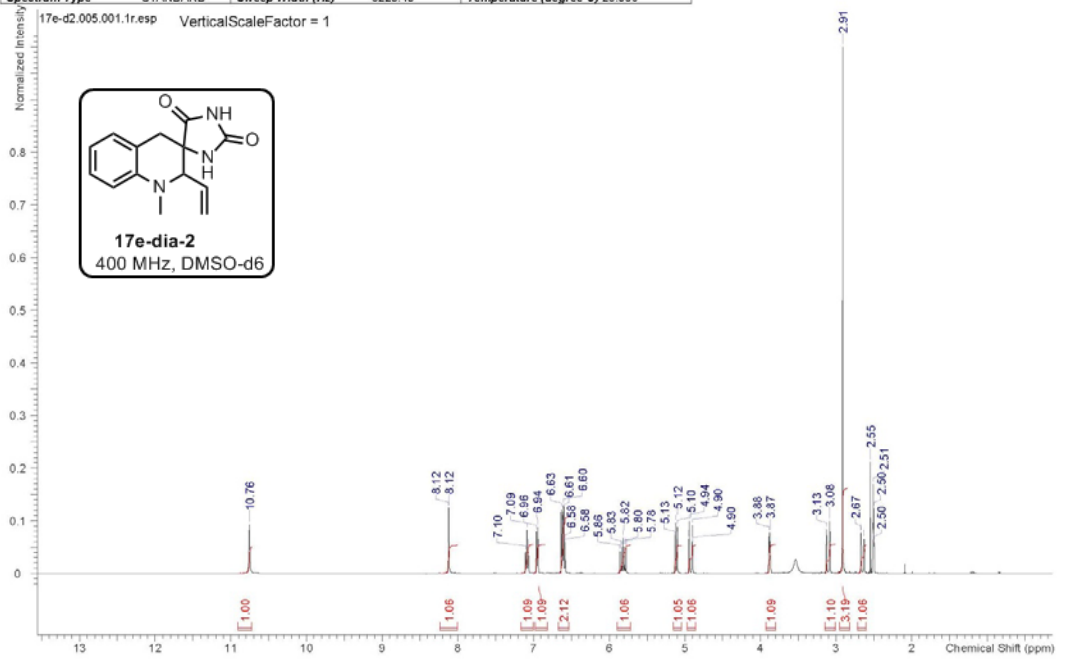
9/3/2015 1:39:59 PM

OriginalDateForRelativeTime 2015-02-21T01:30:24				*Number of Nuclei*	0 C's
Acquisition Time (sec)	1.3664	*Comment*	carbon_QNP_8000 DMSO {C:\u} fbriones 1	*Date*	21 Feb 2015 01:30:24
Date Stamp	21 Feb 2015 01:30:24			*File Name*	C:\Users\kmwk459\Desktop\OL-NMR\Table 3\17e-d1\3\pdata\1\1r
Frequency (MHz)	100.62	*Nucleus*	13C	*Number of Transients*	8000
Original Points Count	32768	*Owner*	usbodlab	*Points Count*	32768
Receiver Gain	13004.00	*SW(cyclical) (Hz)*	23980.81	*Solvent*	DMSO-d6
Spectrum Type	STANDARD	*Sweep Width (Hz)*	23980.08	*Temperature (degree C)*	28.360
Origin	spect				
Pulse Sequence	zgpg30				
Spectrum Offset (Hz)	10012.0518				



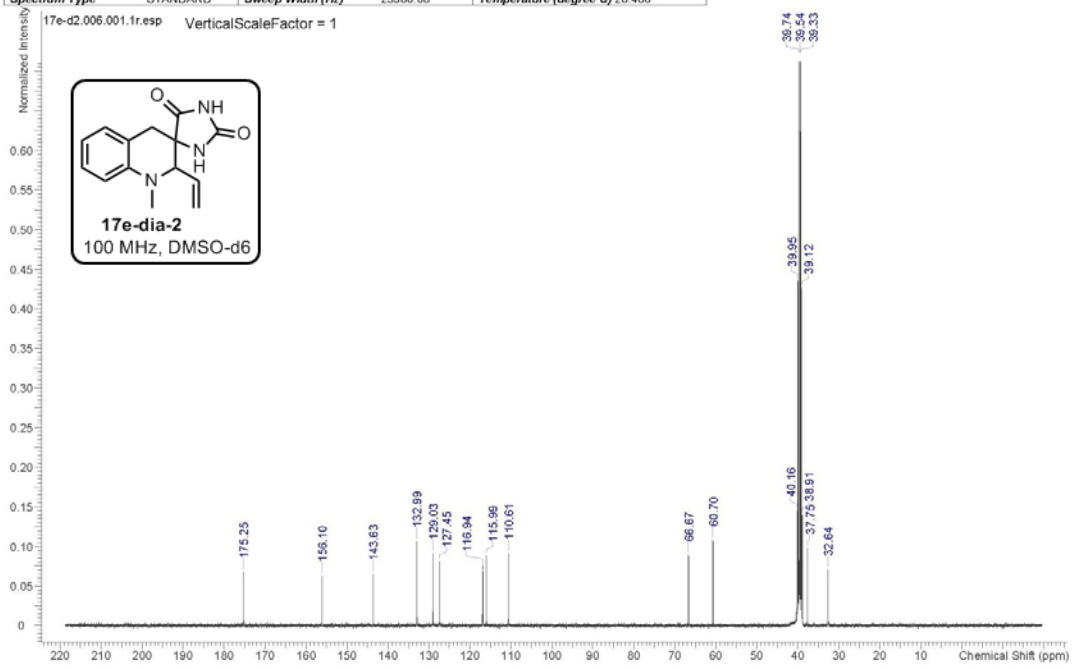
9/3/2015 1:46:11 PM

OriginalDateForRelativeTime 2015-02-23T15:56:32				*Multiplets Integrals Sum*	0.00
Number of Nuclei	0 H's				
Acquisition Time (sec)	0.9961	*Comment*	proton_longdelay_QNP_16 DMSO {C:\u} fbriones 20	*Date*	23 Feb 2015 15:56:32
Date Stamp	23 Feb 2015 15:56:32			*File Name*	C:\Users\kmwk459\Desktop\OL-NMR\Table 3\17e-d2\5\pdata\1\1r
Frequency (MHz)	400.13	*Nucleus*	1H	*Number of Transients*	16
Original Points Count	8192	*Owner*	usbodlab	*Points Count*	32768
Receiver Gain	161.30	*SW(cyclical) (Hz)*	8223.68	*Solvent*	DMSO-d6
Spectrum Type	STANDARD	*Sweep Width (Hz)*	8223.43	*Temperature (degree C)*	26.960
Origin	spect				
Pulse Sequence	zg30				
Spectrum Offset (Hz)	2471.0088				



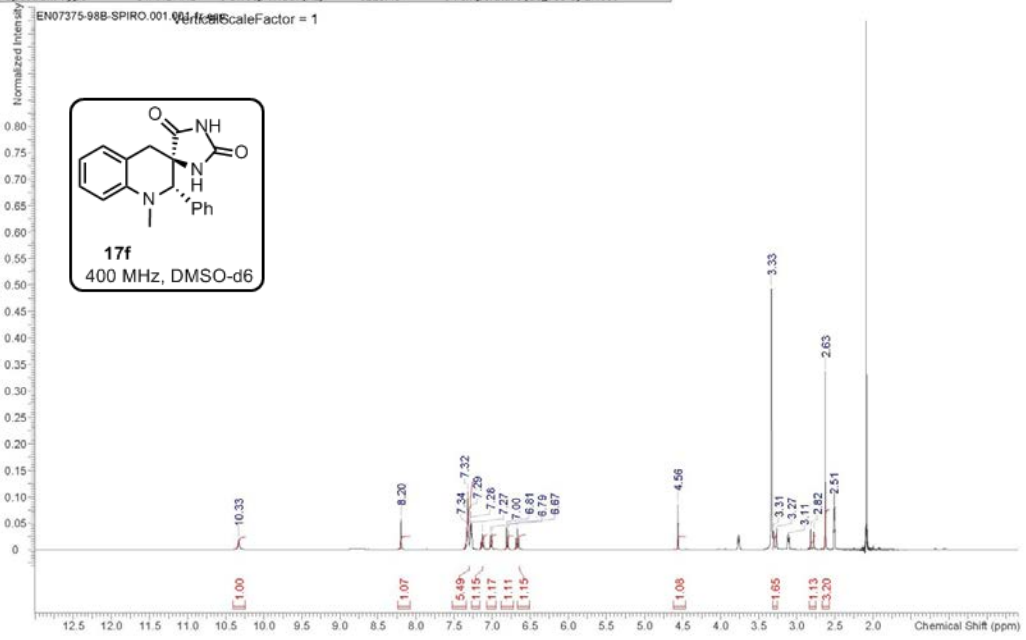
9/3/2015 1:44:54 PM

OriginalDateForRelativeTime 2015-02-23T21:48:32				*Number of Nuclei*	0 C's		
Acquisition Time (sec)	1.3664	*Comment*	carbon_QNP_2500 DMSO {C:\u} fbriones 20			*Date*	23 Feb 2015 21:48:32
Date Stamp	23 Feb 2015 21:48:32			*File Name*	C:\Users\kmwk459\Desktop\OL-NMR\Table 3\17e-d2\6\pdata\1\1r		
Frequency (MHz)	100.62	*Nucleus*	13C	*Number of Transients*	2500	*Origin*	spect
Original Points Count	32768	*Owner*	usbodlab	*Points Count*	32768	*Pulse Sequence*	zgpg30
Receiver Gain	9195.20	*SW(cyclical) (Hz)*	23980.81	*Solvent*	DMSO-d6	*Spectrum Offset (Hz)*	10012.0518
Spectrum Type	STANDARD	*Sweep Width (Hz)*	23980.08	*Temperature (degree C)*	28.460		



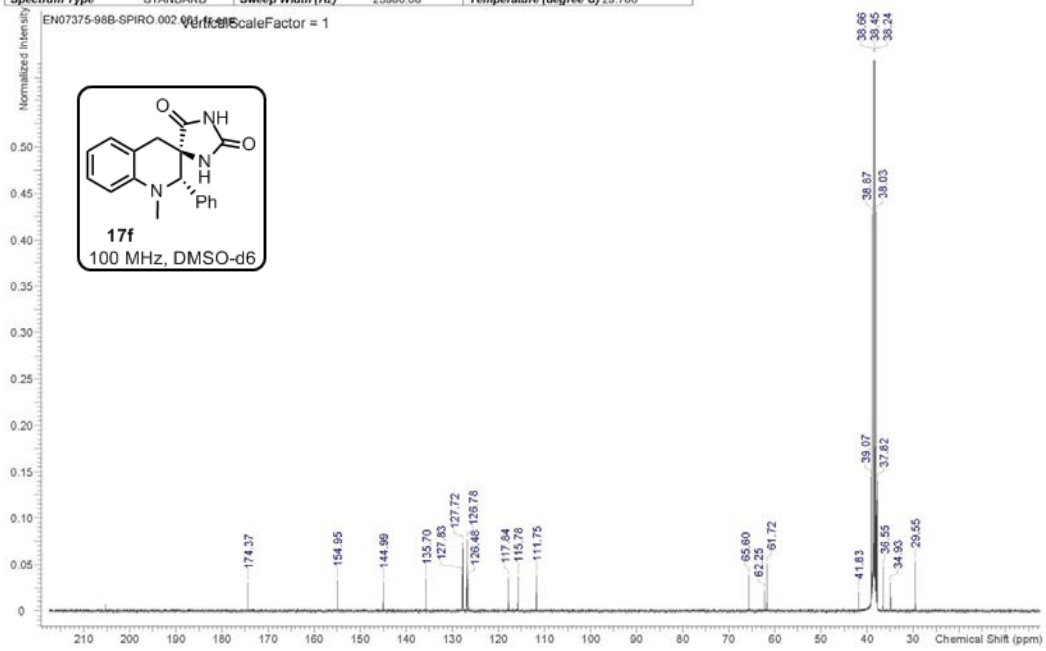
9/3/2015 1:51:39 PM

OriginalDateForRelativeTime 2014-07-11T13:55:12				*Multiplets Integrals Sum*	0.00	*Number of Nuclei*	0 H's
Acquisition Time (sec)	0.9961	*Comment*	proton_longdelay_QNP_16 DMSO {C:\u} fbriones 5			*Date*	11 Jul 2014 13:55:12
Date Stamp	11 Jul 2014 13:55:12			*File Name*	C:\Users\kmwk459\Desktop\OL-NMR\EN07375-98B-SPIRO\1\pdata\1\1r		
Frequency (MHz)	400.13	*Nucleus*	1H	*Number of Transients*	16	*Origin*	spect
Original Points Count	8192	*Owner*	usbodlab	*Points Count*	32768	*Pulse Sequence*	zg30
Receiver Gain	128.00	*SW(cyclical) (Hz)*	8223.68	*Solvent*	DMSO-d6	*Spectrum Offset (Hz)*	2471.0088
Spectrum Type	STANDARD	*Sweep Width (Hz)*	8223.43	*Temperature (degree C)*	27.060		



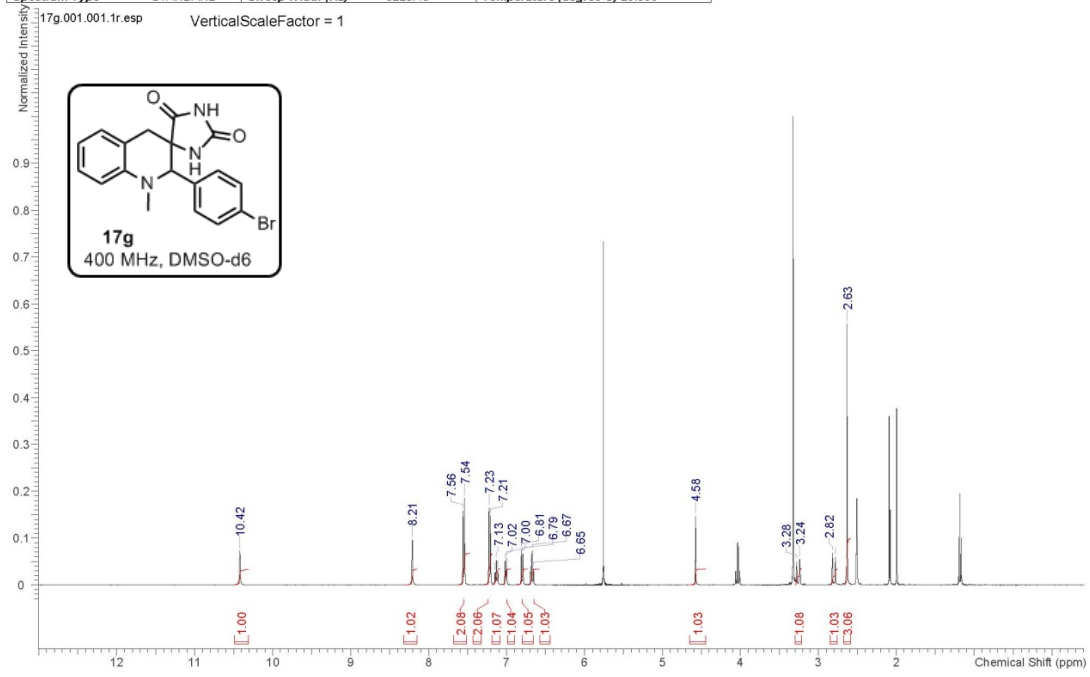
9/3/2015 1:53:00 PM

OriginalDateForRelativeTime	2014-07-12T12:34:08			*Number of Nuclei*	0 C's		
Acquisition Time (sec)	1.3664	*Comment*	carbon_QNP_8000 DMSO {C:\u} fbriones 5			*Date*	12 Jul 2014 12:34:08
Date Stamp	12 Jul 2014 12:34:08			*File Name*	C:\Users\kmwk459\Desktop\OL-NMR\EN07375-98B-SPIRO\2\pdata\1\1r		
Frequency (MHz)	100.62	*Nucleus*	13C	*Number of Transients*	8000	*Origin*	spect
Original Points Count	32768	*Owner*	usbodlab	*Points Count*	32768	*Pulse Sequence*	zgpg30
Receiver Gain	14596.50	*SW(cyclical) (Hz)*	23980.81	*Solvent*	DMSO-d6	*Spectrum Offset (Hz)*	9901.6484
Spectrum Type	STANDARD	*Sweep Width (Hz)*	23980.08	*Temperature (degree C)*	29.760		



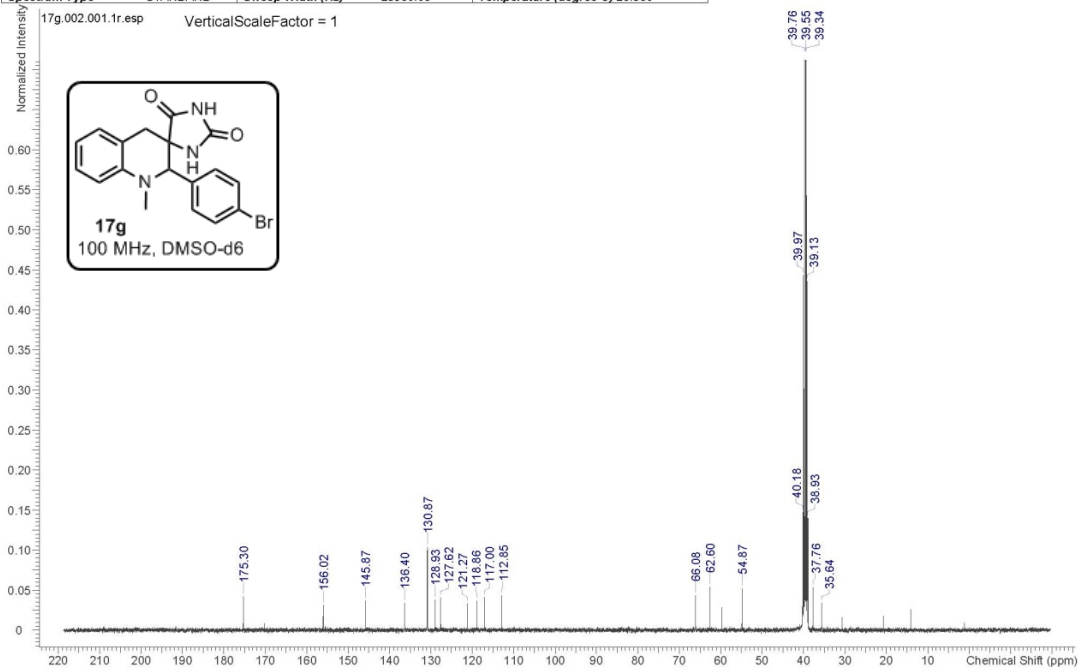
9/3/2015 1:57:52 PM

OriginalDateForRelativeTime	2014-06-20T14:35:44			*Multiplets Integrals Sum*	0.00	*Number of Nuclei*	0 H's
Acquisition Time (sec)	0.9961	*Comment*	proton_longdelay_QNP_16 DMSO {C:\u} fbriones 22			*Date*	20 Jun 2014 14:35:44
Date Stamp	20 Jun 2014 14:35:44			*File Name*	C:\Users\kmwk459\Desktop\OL-NMR\Table 3\17g\1\pdata\1\1r		
Frequency (MHz)	400.13	*Nucleus*	1H	*Number of Transients*	16	*Origin*	spect
Original Points Count	8192	*Owner*	usbodlab	*Points Count*	32768	*Pulse Sequence*	zg30
Receiver Gain	161.30	*SW(cyclical) (Hz)*	8223.68	*Solvent*	DMSO-d6	*Spectrum Offset (Hz)*	2471.0088
Spectrum Type	STANDARD	*Sweep Width (Hz)*	8223.43	*Temperature (degree C)*	26.960		



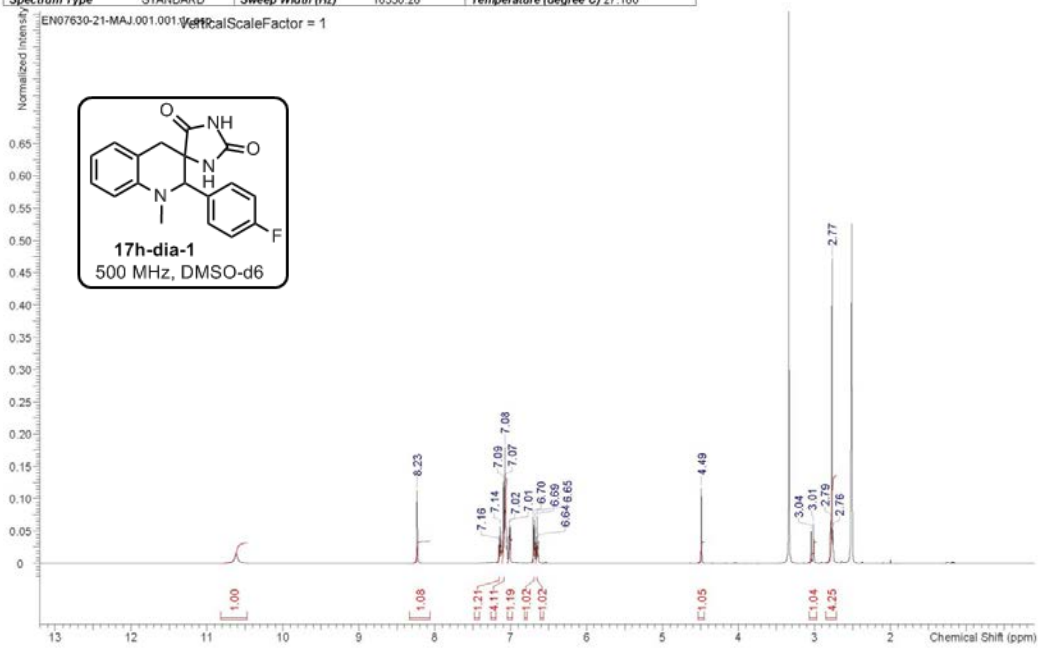
9/3/2015 3:23:23 PM

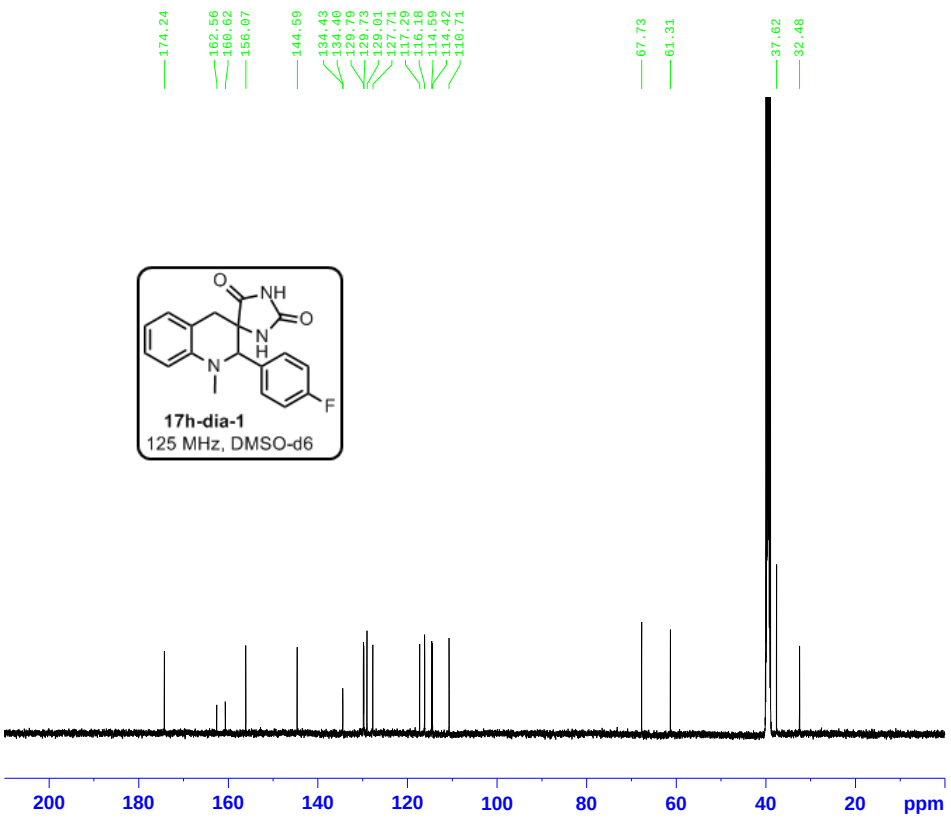
OriginalDateForRelativeTime 2014-06-21T03:21:36				*Number of Nuclei*	0 C's		
Acquisition Time (sec)	1.3664	*Comment*	carbon_QNP_2500 DMSO {C:\u} fbriones 22			*Date*	21 Jun 2014 03:21:36
Date Stamp	21 Jun 2014 03:21:36			*File Name*	C:\Users\kmwk459\Desktop\OL-NMR\Table 3\17g\2\pdata\1\1r		
Frequency (MHz)	100.62	*Nucleus*	13C	*Number of Transients*	2500	*Origin*	spect
Original Points Count	32768	*Owner*	usbodlab	*Points Count*	32768	*Pulse Sequence*	zgpg30
Receiver Gain	9195.20	*SW(cyclical) (Hz)*	23980.81	*Solvent*	DMSO-d6	*Spectrum Offset (Hz)*	10012.0518
Spectrum Type	STANDARD	*Sweep Width (Hz)*	23980.08	*Temperature (degree C)*	29.560		



9/3/2015 2:05:05 PM

OriginalDateForRelativeTime 2015-08-31T12:00:00				*Multiplets Integrals Sum*	0.00	*Number of Nuclei*	0 H's
Acquisition Time (sec)	3.1719	*Comment*	PROTON DMSO {C:\Bruker\TOPSPIN1.3} fbriones 50			*Date*	31 Aug 2015 12:00:00
Date Stamp	31 Aug 2015 12:00:00			*File Name*	C:\Users\kmwk459\Desktop\OL-NMR\Table 3\EN07630-21-MAJ\1\pdata\1\1r		
Frequency (MHz)	500.13	*Nucleus*	1H	*Number of Transients*	32	*Origin*	spect
Original Points Count	32768	*Owner*	usbodlab	*Points Count*	32768	*Pulse Sequence*	zg30
Receiver Gain	287.40	*SW(cyclical) (Hz)*	10330.58	*Solvent*	DMSO-d6	*Spectrum Offset (Hz)*	3088.5571
Spectrum Type	STANDARD	*Sweep Width (Hz)*	10330.26	*Temperature (degree C)*	27.160		






```
NAME      EN07630-21-MAJ
EXPNO                 4
PROCNO                1
Date_          20150831
Time              14.03
INSTRUM           spect
PROBHD   5 mm CPQNP 1H/
PULPROG          zgpg30
TD                65418
SOLVENT             DMSO
NS                  512
DS                    4
SWH           30030.029 Hz
FIDRES        0.459048 Hz
AQ            1.0892597 sec
RG               2896.3
DW               16.650 usec
DE               35.00 usec
TE               300.2 K
D1           2.00000000 sec
d11          0.03000000 sec
DELTA        1.89999998 sec
TD0                   1

======== CHANNEL f1 ========
NUC1                 13C
P1                12.00 usec
PL1                2.20 dB
SF01        125.7703643 MHz

======== CHANNEL f2 ========
CPDPRG2          waltz16
NUC2                 1H
PCPD2             80.00 usec
PL2                1.20 dB
PL12              15.74 dB
PL13              15.89 dB
SFO2        500.1320005 MHz
SI                32768
SF          125.7578519 MHz
WDW                  EM
SSB                   0
LB                 1.00 Hz
GB                    0
PC                 1.40
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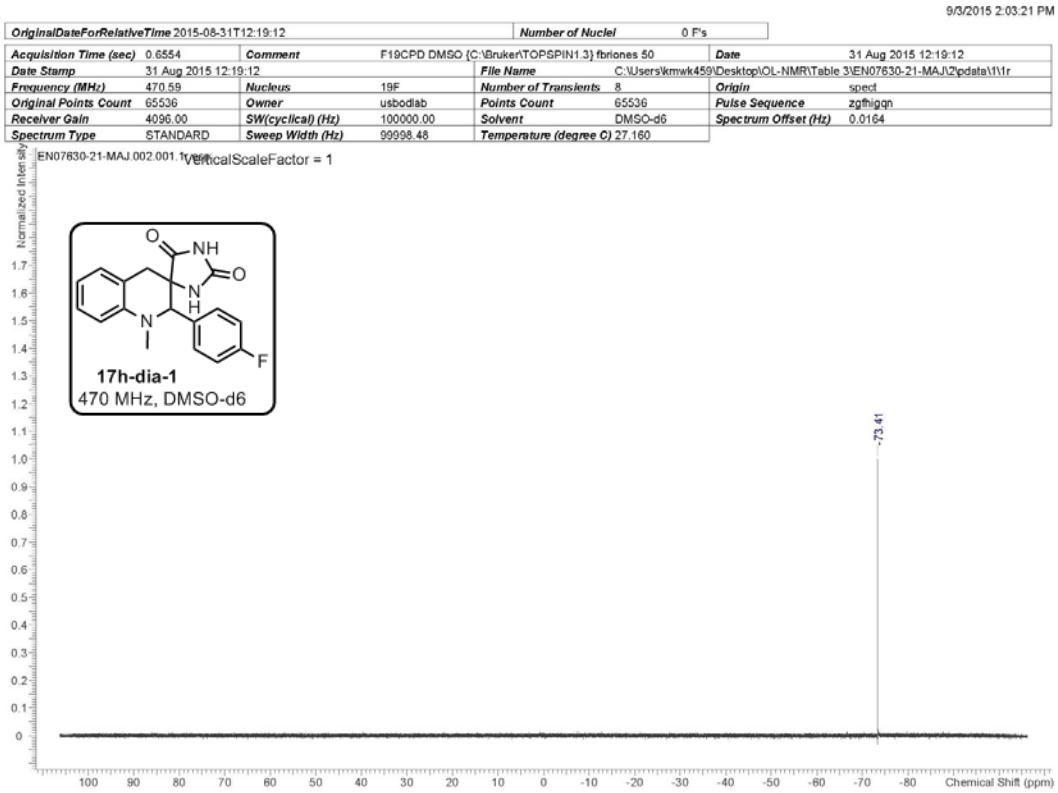
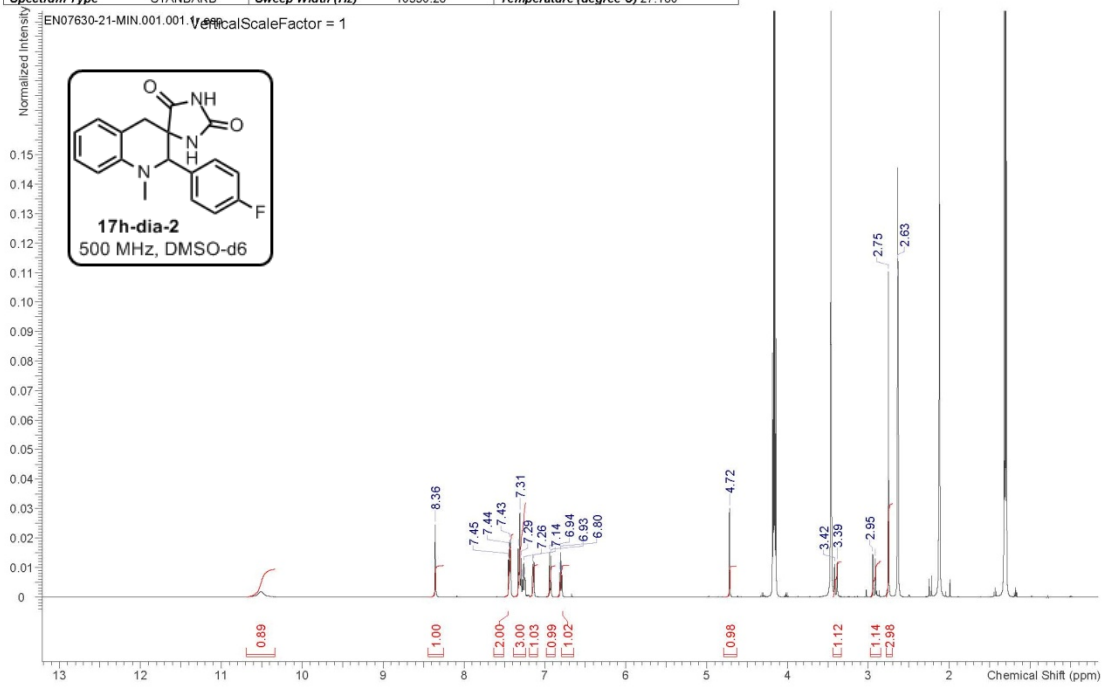
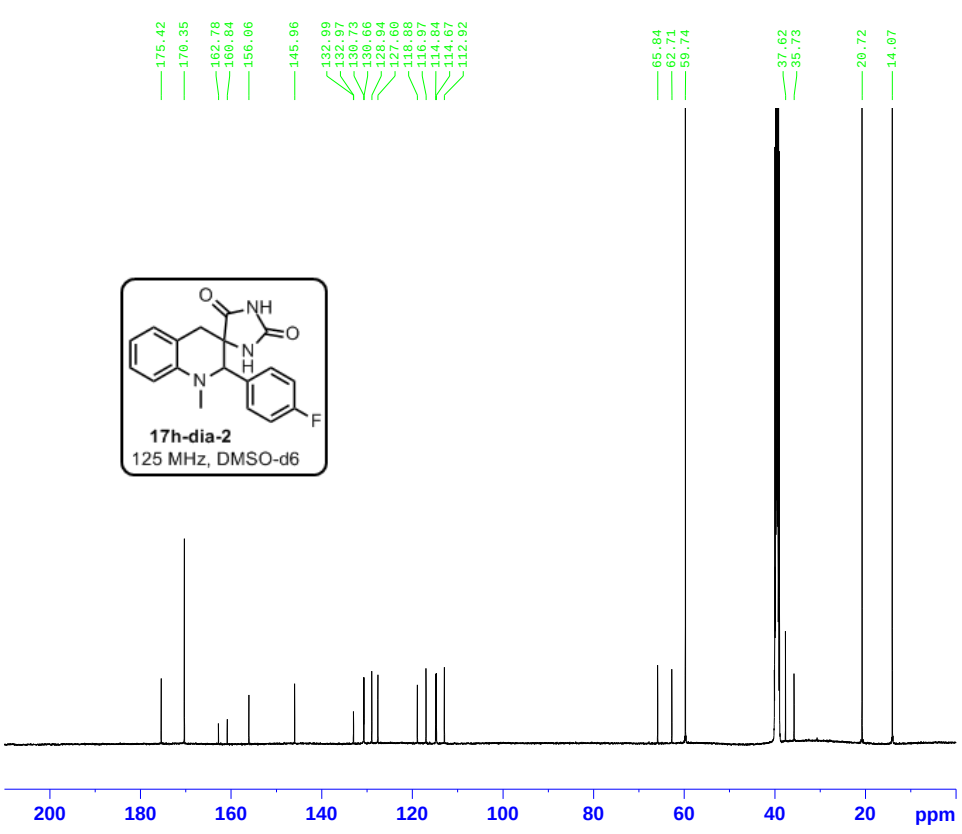


9/3/2015 2:11:48 PM

OriginalDateForRelativeTime	2015-08-31T12:08:32			*Multiplets Integrals Sum*	0.00	*Number of Nuclei*	0 H's
Acquisition Time (sec)	3.1719	*Comment*	PROTON DMSO {C:\Bruker\TOPSPIN1.3} fbriones 51			*Date*	31 Aug 2015 12:08:32
Date Stamp	31 Aug 2015 12:08:32			*File Name*	C:\Users\kmwk459\Desktop\OL-NMR\Table 3\EN07630-21-MIN\1\pdata\1\1r		
Frequency (MHz)	500.13	*Nucleus*	1H	*Number of Transients*	32	*Origin*	spect
Original Points Count	32768	*Owner*	usbodlab	*Points Count*	32768	*Pulse Sequence*	zg30
Receiver Gain	143.70	*SW(cyclical) (Hz)*	10330.58	*Solvent*	DMSO-d6	*Spectrum Offset (Hz)*	3152.1794
Spectrum Type	STANDARD	*Sweep Width (Hz)*	10330.26	*Temperature (degree C)*	27.160		





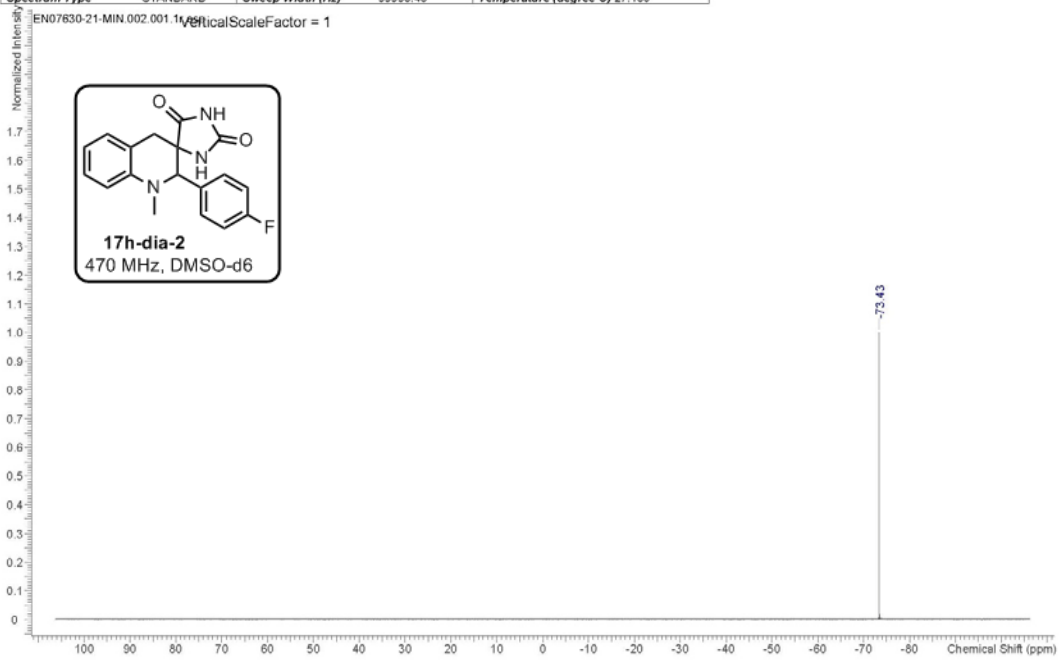

```
NAME          EN07630-21-MIN
EXPNO                      4
PROCNO                     1
Date_               20150901
Time                    1.18
INSTRUM                spect
PROBHD    5 mm CPQNP 1H/
PULPROG              zgpg30
TD                     65418
SOLVENT                DMSO
NS                      8192
DS                         4
SWH                30030.029 Hz
FIDRES             0.459048 Hz
AQ                 1.0892597 sec
RG                      4096
DW                    16.650 usec
DE                    35.00 usec
TE                    300.2 K
D1                 2.00000000 sec
d11                0.03000000 sec
DELTA              1.89999998 sec
TD0                        1

======== CHANNEL f1 ========
NUC1                     13C
P1                     12.00 usec
PL1                     2.20 dB
SFO1             125.7703643 MHz

======== CHANNEL f2 ========
CPDPRG2              waltz16
NUC2                     1H
PCPD2                 80.00 usec
PL2                    1.20 dB
PL12                  15.74 dB
PL13                  15.89 dB
SFO2             500.1320005 MHz
SI                     32768
SF               125.7578519 MHz
WDW                      EM
SSB                       0
LB                     1.00 Hz
GB                        0
PC                     1.40
```

9/3/2015 2:12:22 PM

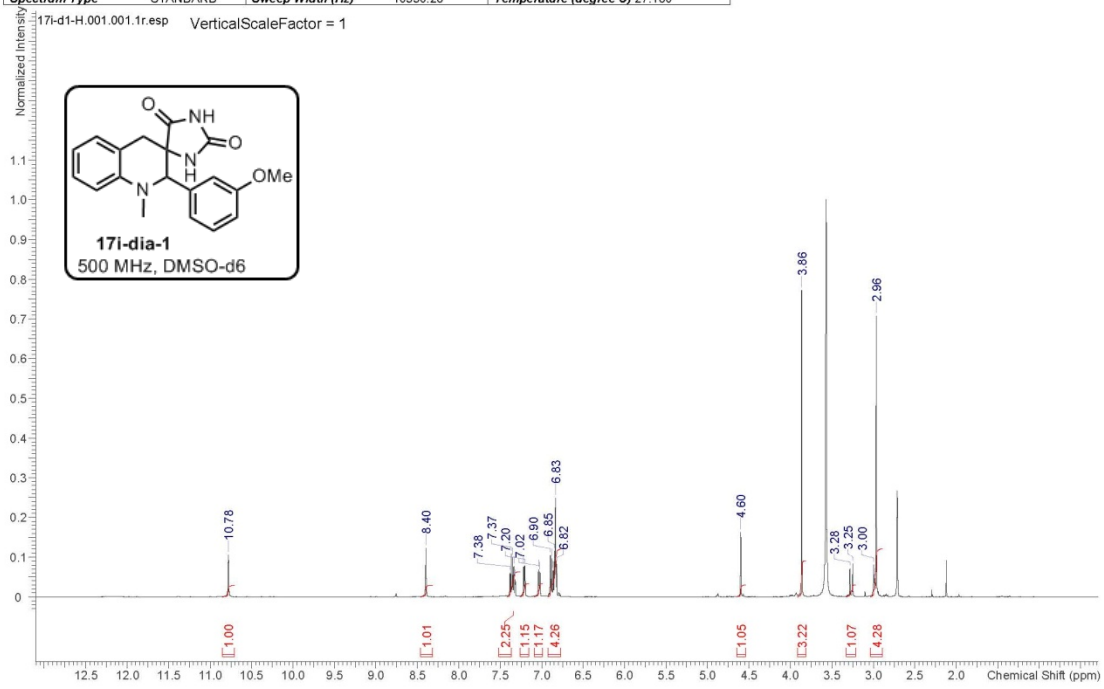
OriginalDateForRelativeTime 2015-08-31T12:25:36				*Number of Nuclei*	0 F's
Acquisition Time (sec)	0.6554	*Comment*	F19CPD DMSO {C:\Bruker\TOPSPIN1.3} fbriones 51	*Date*	31 Aug 2015 12:25:36
Date Stamp	31 Aug 2015 12:25:36		*File Name*	C:\Users\kmwk459\Desktop\OL-NMR\Table 3\EN07630-21-MIN\2\pdata\1\1r	
Frequency (MHz)	470.59	*Nucleus*	19F	*Number of Transients*	8	*Origin*	spect
Original Points Count	65536	*Owner*	usbodlab	*Points Count*	65536	*Pulse Sequence*	zgfhigqn
Receiver Gain	7298.20	*SW(cyclical) (Hz)*	100000.00	*Solvent*	DMSO-d6	*Spectrum Offset (Hz)*	0.0164
Spectrum Type	STANDARD	*Sweep Width (Hz)*	99998.48	*Temperature (degree C)*	27.160		



17h-dia-2
470 MHz, DMSO-d6

9/3/2015 2:19:36 PM

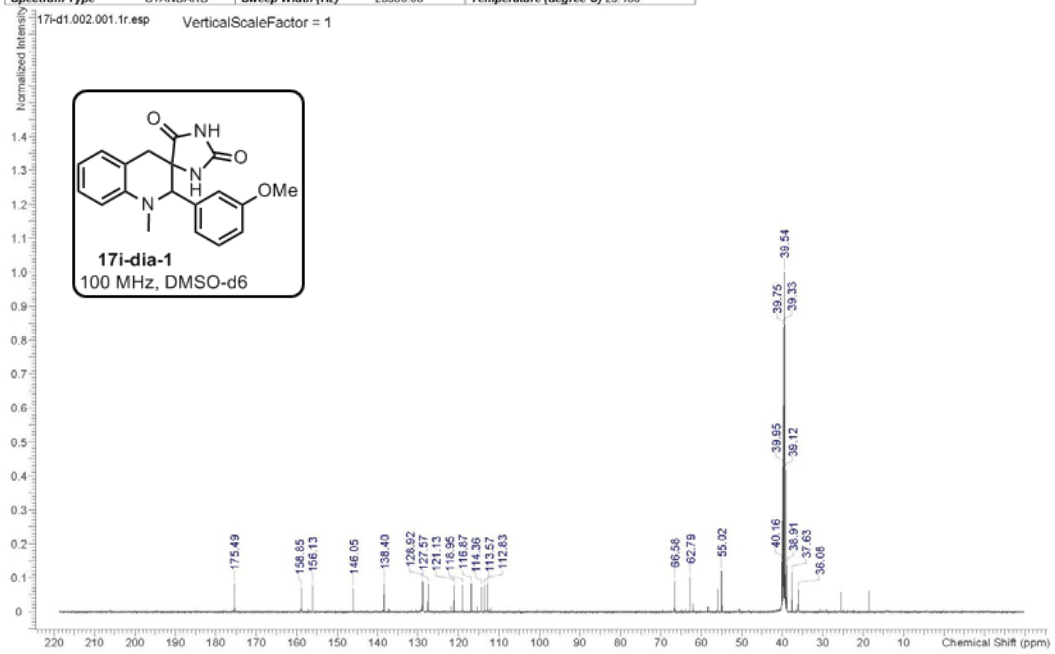
OriginalDateForRelativeTime 2015-08-24T11:06:40				*Multiplets Integrals Sum*	0.00	*Number of Nuclei*	0 H's
Acquisition Time (sec)	3.1719	*Comment*	PROTON DMSO {C:\Bruker\TOPSPIN1.3} fbriones 58			*Date*	24 Aug 2015 11:06:40
Date Stamp	24 Aug 2015 11:06:40			*File Name*	C:\Users\kmwk459\Desktop\OL-NMR\Table 3\17i-d1-H\1\pdata\1\1r		
Frequency (MHz)	500.13	*Nucleus*	1H	*Number of Transients*	64	*Origin*	spect
Original Points Count	32768	*Owner*	usbodlab	*Points Count*	32768	*Pulse Sequence*	zg30
Receiver Gain	143.70	*SW(cyclical) (Hz)*	10330.58	*Solvent*	DMSO-d6	*Spectrum Offset (Hz)*	3188.8694
Spectrum Type	STANDARD	*Sweep Width (Hz)*	10330.26	*Temperature (degree C)*	27.160		



17i-dia-1
500 MHz, DMSO-d6

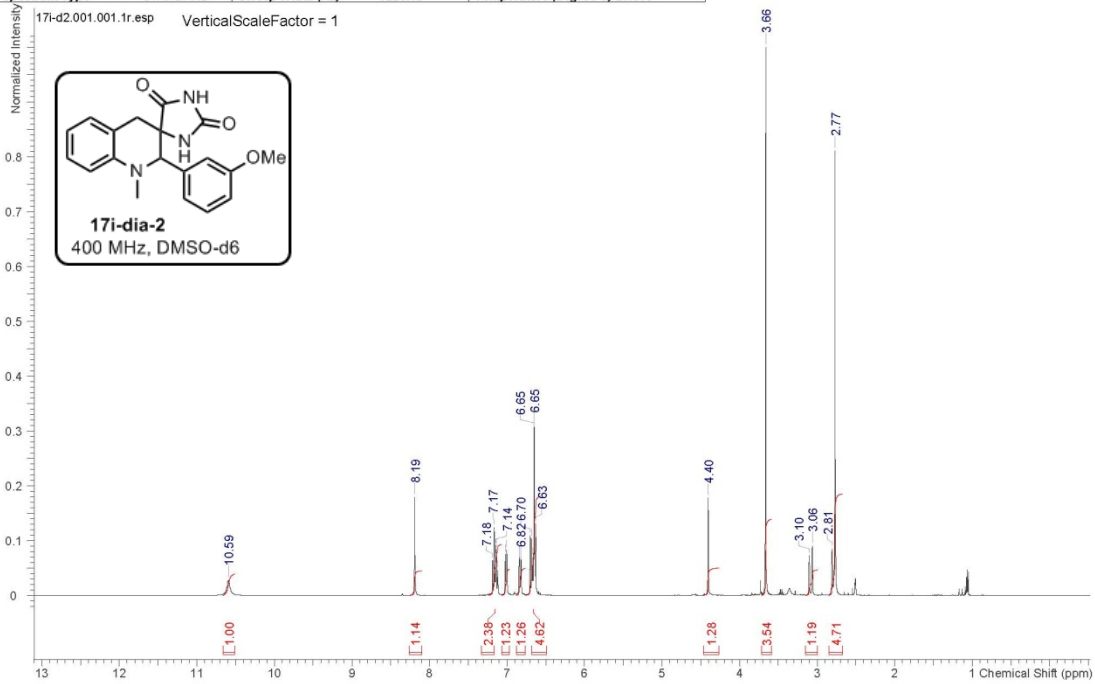
9/3/2015 2:23:01 PM

OriginalDateForRelativeTime 2014-05-29T21:48:48				*Number of Nuclei*	0 C's		
Acquisition Time (sec)	1.3664	*Comment*	carbon_QNP_2500 DMSO {C:\u} fbriones 33			*Date*	29 May 2014 21:48:48
Date Stamp	29 May 2014 21:48:48			*File Name*	C:\Users\kmwk459\Desktop\OL-NMR\Table 3\17i-d1\2\pdata\1\1r		
Frequency (MHz)	100.62	*Nucleus*	13C	*Number of Transients*	2500	*Origin*	spect
Original Points Count	32768	*Owner*	usbodlab	*Points Count*	32768	*Pulse Sequence*	zgpg30
Receiver Gain	11585.20	*SW(cyclical) (Hz)*	23980.81	*Solvent*	DMSO-d6	*Spectrum Offset (Hz)*	10012.0518
Spectrum Type	STANDARD	*Sweep Width (Hz)*	23980.08	*Temperature (degree C)*	29.160		



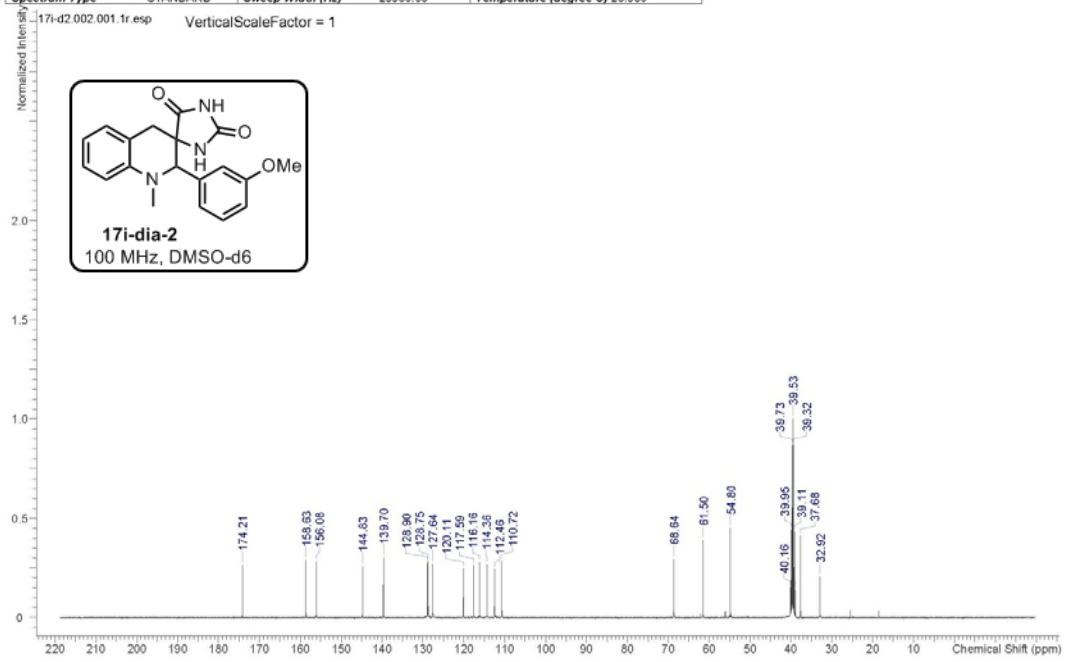
9/3/2015 2:21:49 PM

OriginalDateForRelativeTime 2014-05-29T16:41:36				*Multiplets Integrals Sum*	0.00	*Number of Nuclei*	0 H's
Acquisition Time (sec)	0.9961	*Comment*	proton_QNP_128 DMSO {C:\u} fbriones 37			*Date*	29 May 2014 16:41:36
Date Stamp	29 May 2014 16:41:36			*File Name*	C:\Users\kmwk459\Desktop\OL-NMR\Table 3\17i-d2\1\pdata\1\1r		
Frequency (MHz)	400.13	*Nucleus*	1H	*Number of Transients*	128	*Origin*	spect
Original Points Count	8192	*Owner*	usbodlab	*Points Count*	32768	*Pulse Sequence*	zg30
Receiver Gain	40.30	*SW(cyclical) (Hz)*	8223.68	*Solvent*	DMSO-d6	*Spectrum Offset (Hz)*	2471.0088
Spectrum Type	STANDARD	*Sweep Width (Hz)*	8223.43	*Temperature (degree C)*	26.960		



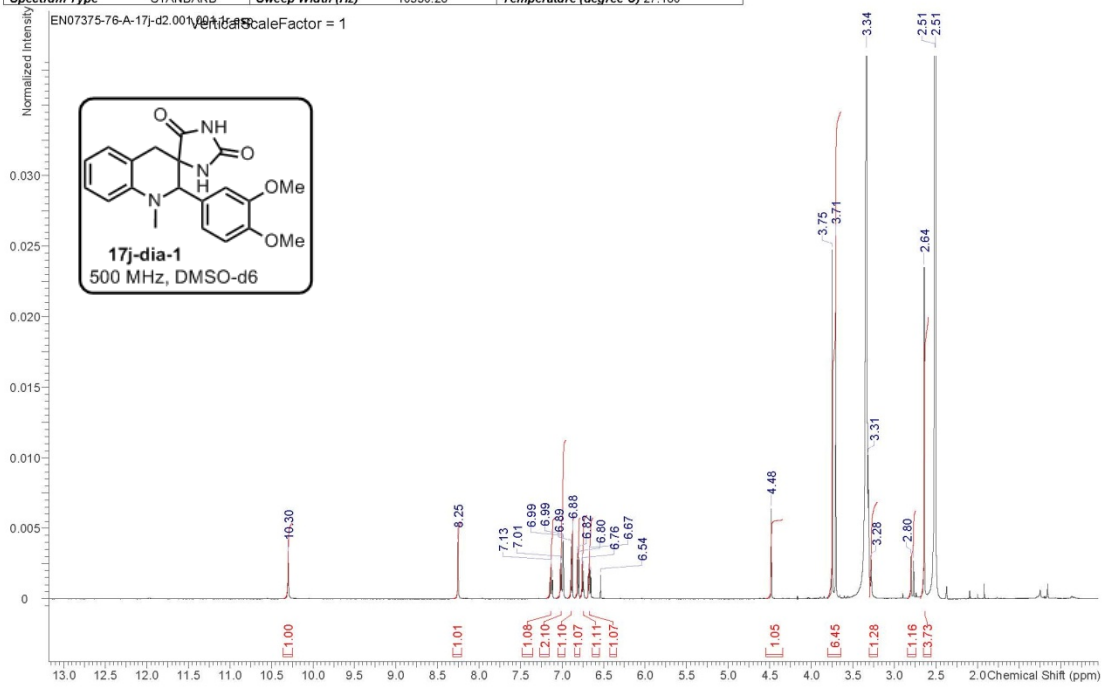
9/3/2015 2:23:32 PM

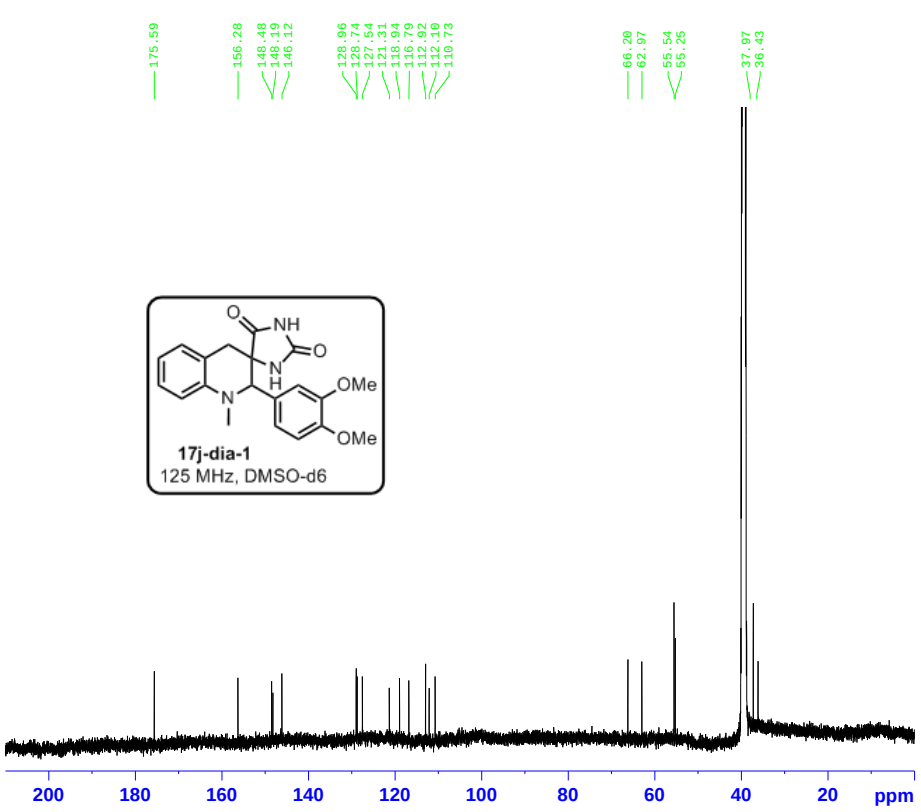
OriginalDateForRelativeTime 2014-05-29T23:35:28				*Number of Nuclei*	0 C's
Acquisition Time (sec)	1.3664	*Comment*	carbon_QNP_2500 DMSO {C:\u} fbriones 37	*Date*	29 May 2014 23:35:28
Date Stamp	29 May 2014 23:35:28			*File Name*	C:\Users\kmwk459\Desktop\OL-NMR\Table 3\17i-d2\2\pdata\1\1r
Frequency (MHz)	100.62	*Nucleus*	13C	*Number of Transients*	2500
Original Points Count	32768	*Owner*	usbodlab	*Points Count*	32768
Receiver Gain	9195.20	*SW(cyclical) (Hz)*	23980.81	*Solvent*	DMSO-d6
Spectrum Type	STANDARD	*Sweep Width (Hz)*	23980.08	*Temperature (degree C)*	28.960
				Origin	spect
				Pulse Sequence	zgpg30
				Spectrum Offset (Hz)	10012.0518



9/3/2015 2:32:31 PM

OriginalDateForRelativeTime 2015-08-25T11:10:56				*Multiplets Integrals Sum*	0.00	*Number of Nuclei*	0 H's
Acquisition Time (sec)	3.1719	*Comment*	PROTON DMSO {C:\Bruker\TOPSPIN1.3} fbriones 34			*Date*	25 Aug 2015 11:10:56
Date Stamp	25 Aug 2015 11:10:56			*File Name*	C:\Users\kmwk459\Desktop\OL-NMR\Table 3\EN07375-76-A-17j-d2\1\pdata\1\1r		
Frequency (MHz)	500.13	*Nucleus*	1H	*Number of Transients*	64	*Origin*	spect
Original Points Count	32768	*Owner*	usbodlab	*Points Count*	32768	*Pulse Sequence*	zg30
Receiver Gain	287.40	*SW(cyclical) (Hz)*	10330.58	*Solvent*	DMSO-d6	*Spectrum Offset (Hz)*	3088.5571
Spectrum Type	STANDARD	*Sweep Width (Hz)*	10330.26	*Temperature (degree C)*	27.160		



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NAME      EN07754-17J-76A
EXPNO                   3
PROCNO                  1
Date_            20150904
Time                 1.18
INSTRUM             spect
PROBHD    5 mm CPQNP 1H/
PULPROG           zgpg30
TD                 65418
SOLVENT              DMSO
NS                   8192
DS                      4
SWH             30030.029 Hz
FIDRES          0.459048 Hz
AQ              1.0892597 sec
RG                 2896.3
DW                 16.650 usec
DE                 35.00 usec
TE                 300.2 K
D1            2.00000000 sec
d11           0.03000000 sec
DELTA         1.89999998 sec
TD0                     1

======== CHANNEL f1 ========
NUC1                  13C
P1                 12.00 usec
PL1                 2.20 dB
SFO1         125.7703643 MHz

======== CHANNEL f2 ========
CPDPRG2           waltz16
NUC2                   1H
PCPD2              80.00 usec
PL2                 1.20 dB
PL12               15.74 dB
PL13               15.89 dB
SFO2         500.1320005 MHz
SI                  32768
SF           125.7578519 MHz
WDW                    EM
SSB                     0
LB                   1.00 Hz
GB                      0
PC                   1.40
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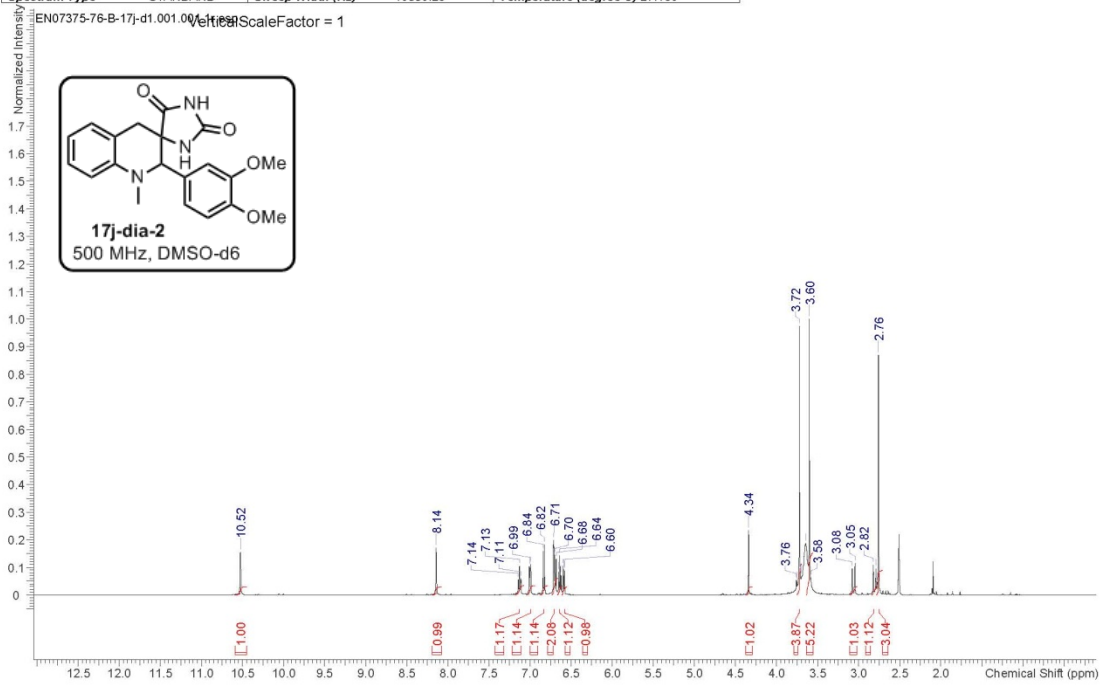
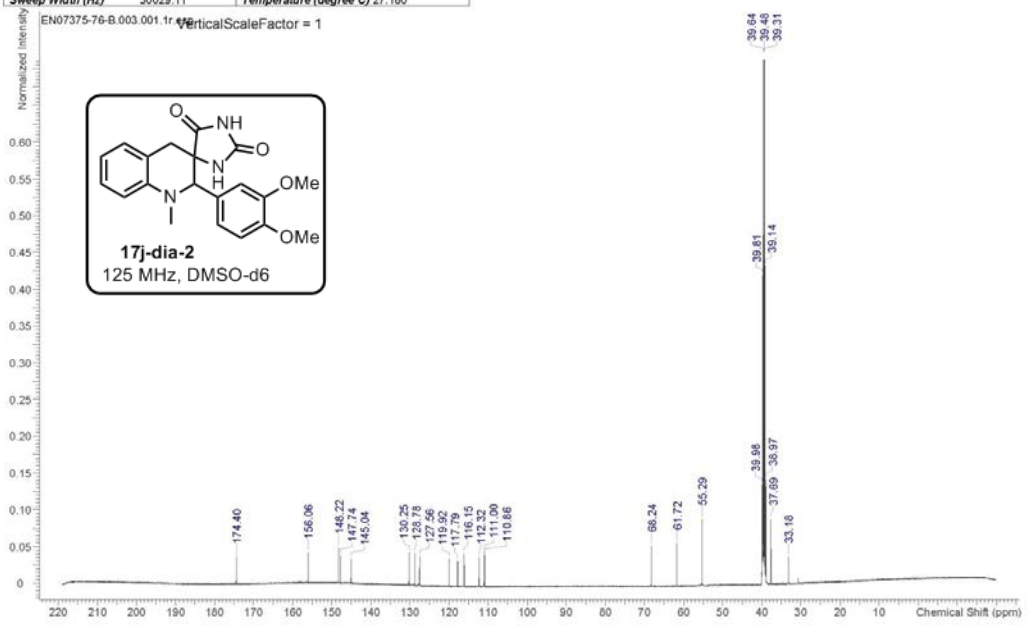
9/3/2015 2:25:42 PM

OriginalDateForRelativeTime 2015-08-25T11:23:44				***Multiplets Integrals Sum***	0.00	***Number of Nuclei***	0 H's
Acquisition Time (sec)	3.1719	***Comment***	PROTON DMSO {C:\Bruker\TOPSPIN1.3} fbriones 35			***Date***	25 Aug 2015 11:23:44
Date Stamp	25 Aug 2015 11:23:44			***File Name***	C:\Users\kmwk459\Desktop\OL-NMR\Table 3\EN07375-76-B-17j-d1\1\pdata\1\1r		
Frequency (MHz)	500.13	***Nucleus***	1H	***Number of Transients***	64	***Origin***	spect
Original Points Count	32768	***Owner***	usbodlab	***Points Count***	32768	***Pulse Sequence***	zg30
Receiver Gain	90.50	***SW(cyclical) (Hz)***	10330.58	***Solvent***	DMSO-d6	***Spectrum Offset (Hz)***	3088.5571
Spectrum Type	STANDARD	***Sweep Width (Hz)***	10330.26	***Temperature (degree C)***	27.160		



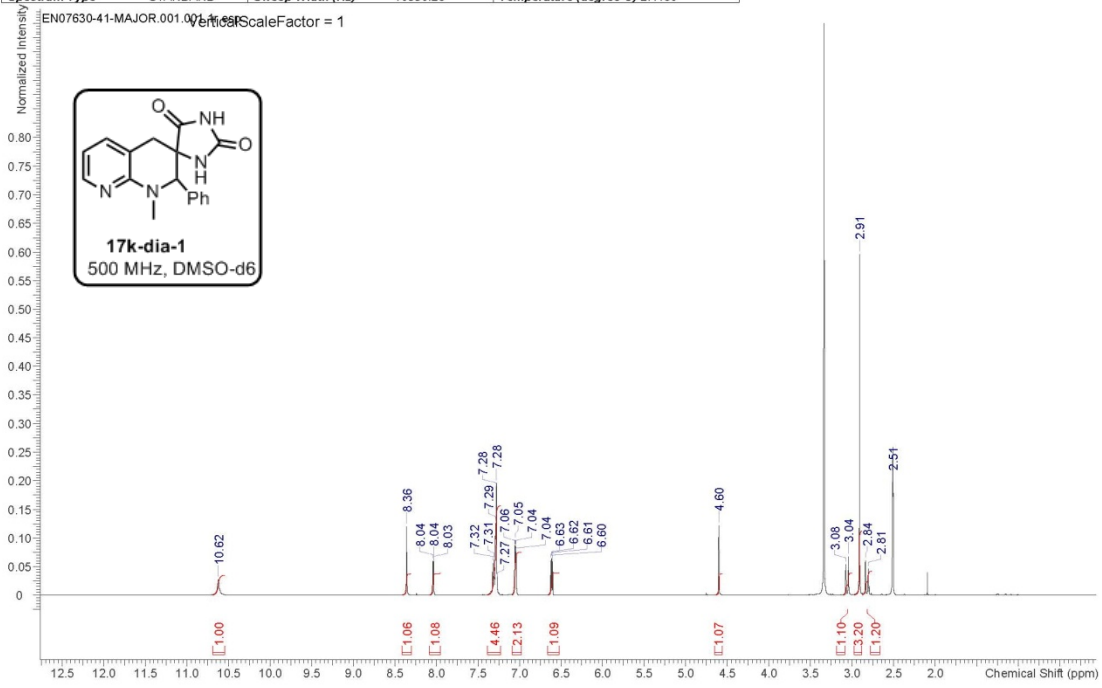
9/4/2015 11:54:04 AM

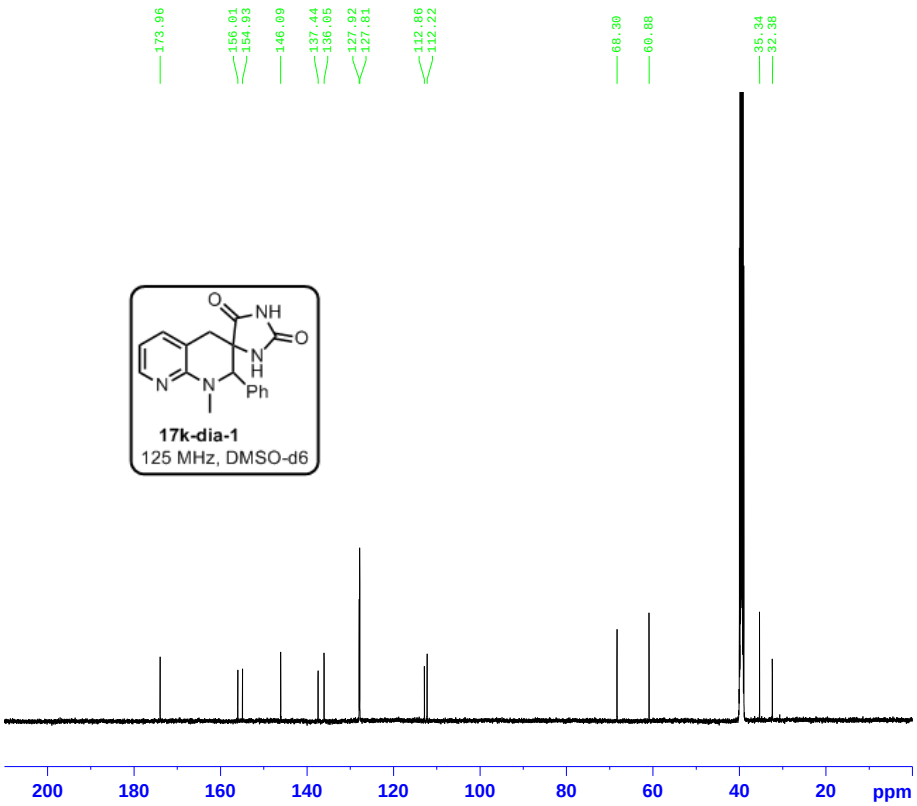
OriginalDateForRelativeTime 2015-09-04T11:32:16			*Multiplets Integrals Sum* 0.00	*Number of Nuclei*	0 C's
Acquisition Time (sec) 1.0912	*Comment*	C13CPD DMSO {C:\Bruker\TOPSPIN1.3} fbriones 18		*Date*	04 Sep 2015 11:32:16
Date Stamp 04 Sep 2015 11:32:16					
File Name	\\netapp2\nmr_archive\GHP_B00_500cryo\data\fbriones\nmr\EN07375-76-B\3\pdata\1\1r			*Frequency (MHz)*	125.77
Nucleus 13C	*Number of Transients*	512	*Origin* spect	*Original Points Count*	32768
Owner usbodlab	*Points Count*	32768	*Pulse Sequence* zgpg30	*Receiver Gain*	4096.00
SW(cyclical) (Hz) 30030.03	*Solvent*	DMSO-d6	*Spectrum Offset (Hz)* 12515.1611	*Spectrum Type*	STANDARD
Sweep Width (Hz) 30029.11	*Temperature (degree C)* 27.160				



9/3/2015 4:38:09 PM

OriginalDateForRelativeTime 2015-08-20T16:16:00				*Multiplets Integrals Sum*	0.00	*Number of Nuclei*	0 H's
Acquisition Time (sec) 3.1719	*Comment*	PROTON DMSO {C:\Bruker\TOPSPIN1.3} fbriones 2				*Date*	20 Aug 2015 16:16:00
Date Stamp 20 Aug 2015 16:16:00				*File Name*	C:\Users\kmwk459\Desktop\OL-NMR\Table 3\EN07630-41-MAJOR\1\pdata\1\1r		
Frequency (MHz) 500.13	*Nucleus*	1H		*Number of Transients*	16	*Origin*	spect
Original Points Count 32768	*Owner*	usbodlab		*Points Count*	32768	*Pulse Sequence*	zg30
Receiver Gain 228.10	*SW(cyclical) (Hz)*	10330.58		*Solvent*	DMSO-d6	*Spectrum Offset (Hz)*	3088.5571
Spectrum Type STANDARD	*Sweep Width (Hz)*	10330.26		*Temperature (degree C)*	27.160		



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NAME      EN07630-41-MAJOR
EXPNO                    3
PROCNO                   1
Date_             20150903
Time                 16.16
INSTRUM              spect
PROBHD    5 mm CPQNP 1H/
PULPROG           zgpg30
TD                 65418
SOLVENT             DMSO
NS                   512
DS                     4
SWH            30030.029 Hz
FIDRES          0.459048 Hz
AQ              1.0892597 sec
RG                3649.1
DW                16.650 usec
DE                35.00 usec
TE                300.2 K
D1           2.00000000 sec
d11          0.03000000 sec
DELTA        1.89999998 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                  13C
P1                 12.00 usec
PL1                 2.20 dB
SFO1         125.7703643 MHz

======== CHANNEL f2 ========
CPDPRG2           waltz16
NUC2                  1H
PCPD2              80.00 usec
PL2                 1.20 dB
PL12               15.74 dB
PL13               15.89 dB
SFO2         500.1320005 MHz
SI                 32768
SF           125.7578519 MHz
WDW                   EM
SSB                    0
LB                  1.00 Hz
GB                     0
PC                  1.40
```
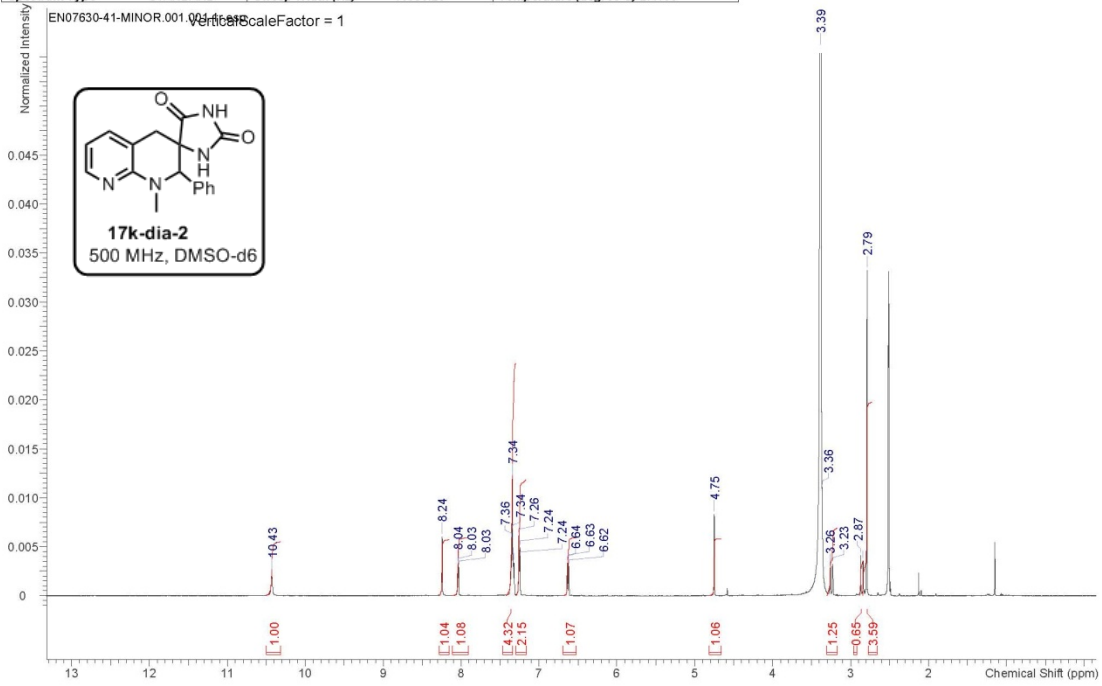
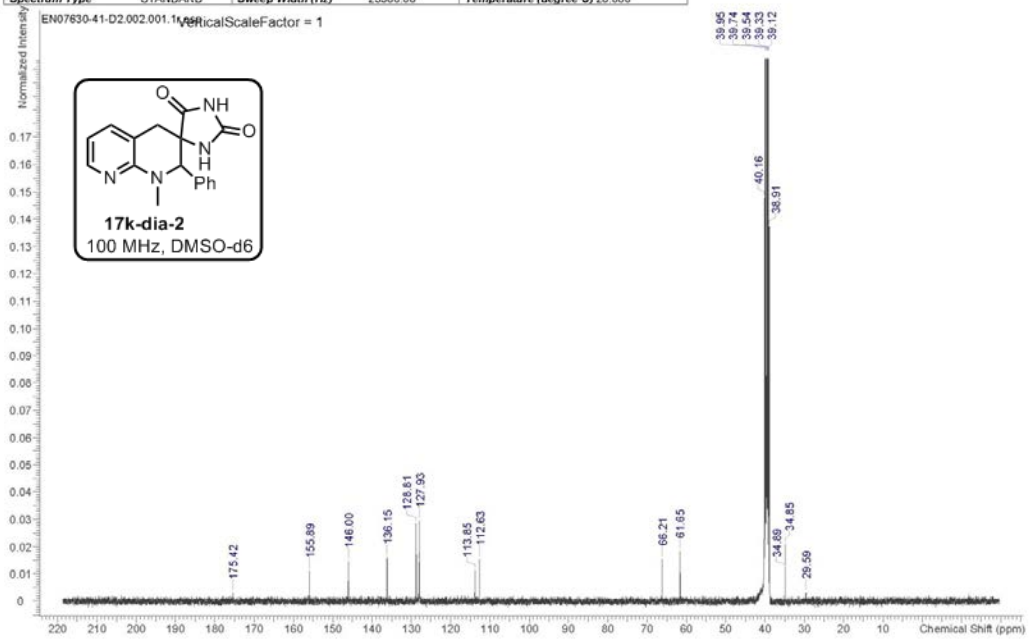

9/3/2015 4:40:48 PM

OriginalDateForRelativeTime 2015-08-20T15:50:24				***Multiplets Integrals Sum***	0.00	***Number of Nuclei***	0 H's
Acquisition Time (sec)	3.1719	***Comment***	PROTON DMSO {C:\Bruker\TOPSPIN1.3} fbriones 1			***Date***	20 Aug 2015 15:50:24
Date Stamp	20 Aug 2015 15:50:24			***File Name***	C:\Users\kmwk459\Desktop\OL-NMR\Table 3\EN07630-41-MINOR\1\pdata\1\1r		
Frequency (MHz)	500.13	***Nucleus***	1H	***Number of Transients***	16	***Origin***	spect
Original Points Count	32768	***Owner***	usbodlab	***Points Count***	32768	***Pulse Sequence***	zg30
Receiver Gain	128.00	***SW(cyclical) (Hz)***	10330.58	***Solvent***	DMSO-d6	***Spectrum Offset (Hz)***	3088.5571
Spectrum Type	STANDARD	***Sweep Width (Hz)***	10330.26	***Temperature (degree C)***	27.160		



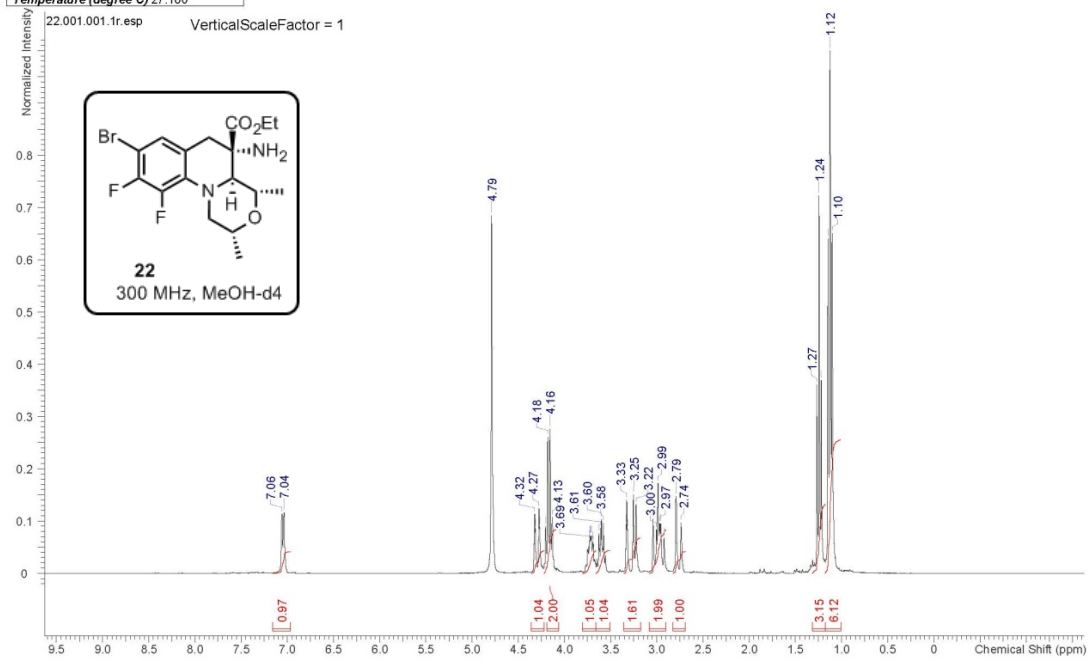
9/3/2015 4:54:17 PM

OriginalDateForRelativeTime 2014-12-11T01:30:24				*Number of Nuclei*	0 C's
Acquisition Time (sec)	1.3664	*Comment*	carbon_QNP_8000 DMSO {C:\u} fbriones 11	*Date*	11 Dec 2014 01:30:24
Date Stamp	11 Dec 2014 01:30:24			*File Name*	C:\Users\kmwk459\Desktop\OL-NMR\Table 3\EN07630-41-D2\2\pdata\1\1r
Frequency (MHz)	100.62	*Nucleus*	13C	*Number of Transients*	8000
Original Points Count	32768	*Owner*	usbodlab	*Points Count*	32768
Receiver Gain	18390.40	*SW(cyclical) (Hz)*	23980.81	*Solvent*	DMSO-d6
Spectrum Type	STANDARD	*Sweep Width (Hz)*	23980.08	*Temperature (degree C)*	28.060
Origin	spect				
Pulse Sequence	zgpg30				
Spectrum Offset (Hz)	10012.0518				



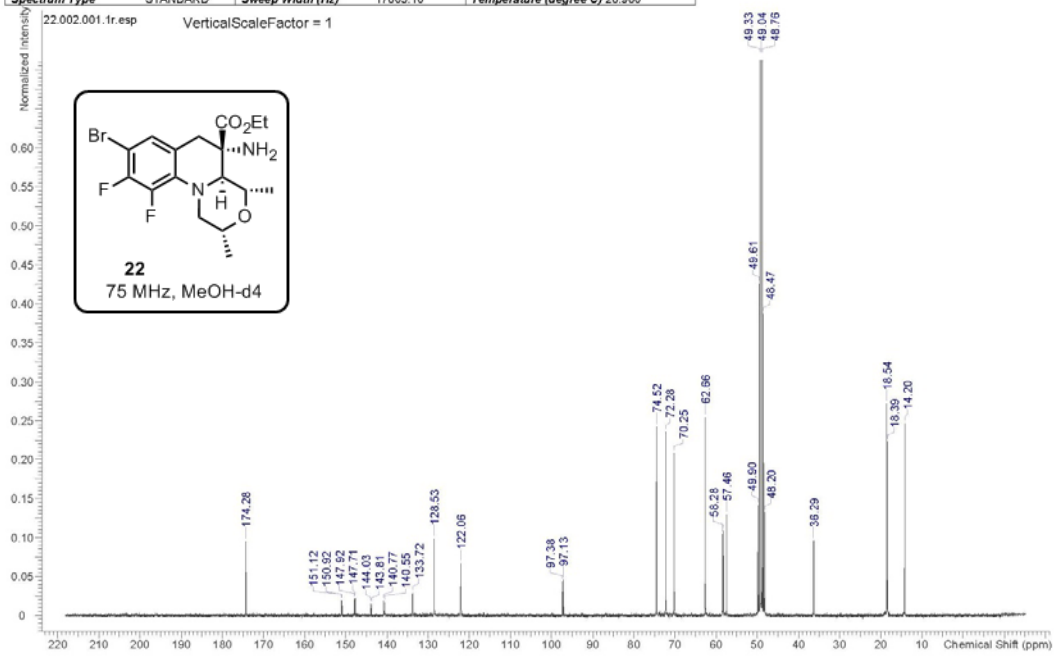
9/2/2015 5:09:51 PM

OriginalDateForRelativeTime 2015-07-17T14:08:00				*Multiplets Integrals Sum*	0.00	*Number of Nuclei*	0 H's
Acquisition Time (sec)	2.6542	*Comment*	A3 300MHz proton_longdelay_QNP_16 MeOD {C:\u} fbriones 9				
Date	17 Jul 2015 14:08:00			*Date Stamp*	17 Jul 2015 14:08:00		
File Name	C:\Users\kmwk459\Desktop\OL-NMR\22\1\pdata\1\1r			*Frequency (MHz)*	300.13	*Nucleus*	1H
Number of Transients	16	*Origin*	spect	*Original Points Count*	16384	*Owner*	usbodlab
Points Count	32768	*Pulse Sequence*	zg30	*Receiver Gain*	228.10	*SW(cyclical) (Hz)*	6172.84
Solvent	METHANOL-d4	*Spectrum Offset (Hz)*	1853.4569	*Spectrum Type*	STANDARD	*Sweep Width (Hz)*	6172.65
Temperature (degree C)	27.160						



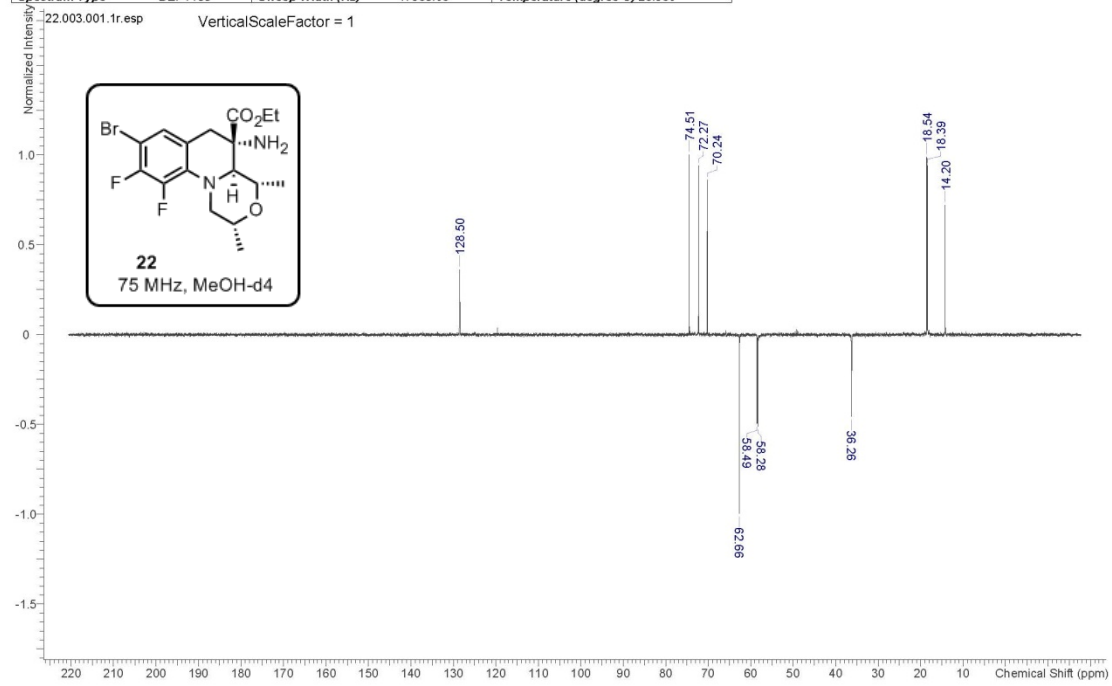
9/2/2015 5:10:31 PM

OriginalDateForRelativeTime 2015-07-18T01:07:12				*Number of Nuclei*	0 C's		
Acquisition Time (sec)	1.8612	*Comment*	A3 300MHz carbon_QNP_8000 MeOD {C:\u} fbriones 9			*Date*	18 Jul 2015 01:07:12
Date Stamp	18 Jul 2015 01:07:12			*File Name*	C:\Users\kmwk459\Desktop\OL-NMR\22\2\pdata\1\1r		
Frequency (MHz)	75.48	*Nucleus*	13C	*Number of Transients*	8000	*Origin*	spect
Original Points Count	32768	*Owner*	usbodlab	*Points Count*	32768	*Pulse Sequence*	zgpg30
Receiver Gain	2048.00	*SW(cyclical) (Hz)*	17605.63	*Solvent*	METHANOL-d4	*Spectrum Offset (Hz)*	7655.1299
Spectrum Type	STANDARD	*Sweep Width (Hz)*	17605.10	*Temperature (degree C)*	26.960		



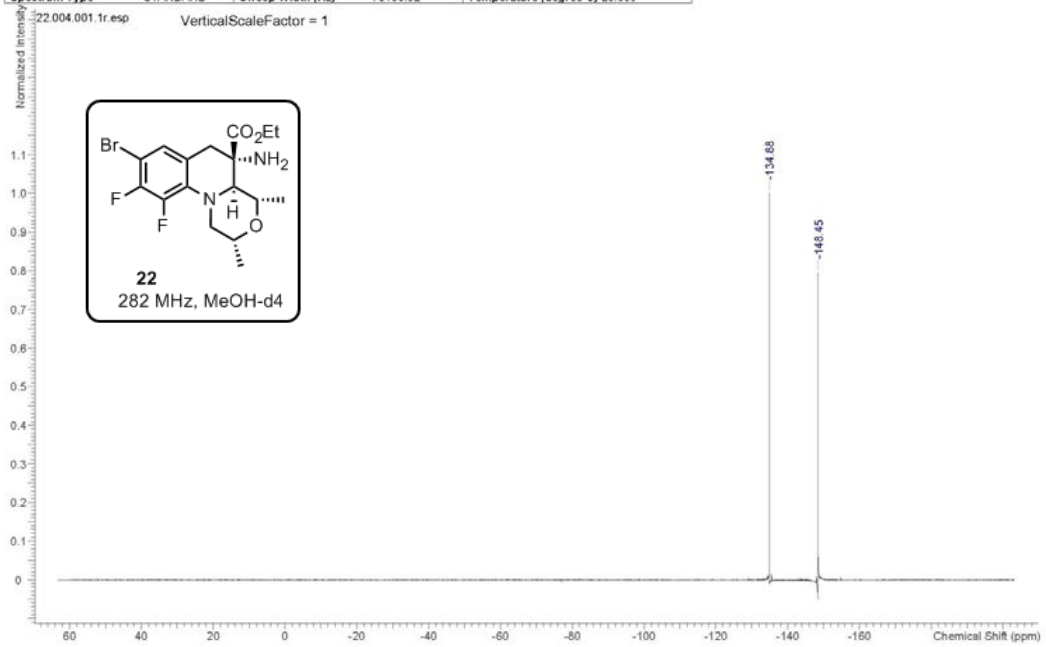
9/2/2015 5:11:09 PM

OriginalDateForRelativeTime 2015-07-18T10:06:56				*Number of Nuclei*	0 C's		
Acquisition Time (sec)	1.8219	*Comment*	A3 300MHz dept_QNP_2000 MeOD {C:\u} fbriones 9			*Date*	18 Jul 2015 10:06:56
Date Stamp	18 Jul 2015 10:06:56			*File Name*	C:\Users\kmwk459\Desktop\OL-NMR\22\3\pdata\1\1r		
Frequency (MHz)	75.48	*Nucleus*	13C	*Number of Transients*	2000	*Origin*	spect
Original Points Count	32768	*Owner*	usbodlab	*Points Count*	32768	*Pulse Sequence*	dept135
Receiver Gain	16384.00	*SW(cyclical) (Hz)*	17985.61	*Solvent*	METHANOL-d4	*Spectrum Offset (Hz)*	7655.1504
Spectrum Type	DEPT135	*Sweep Width (Hz)*	17985.06	*Temperature (degree C)*	26.960		



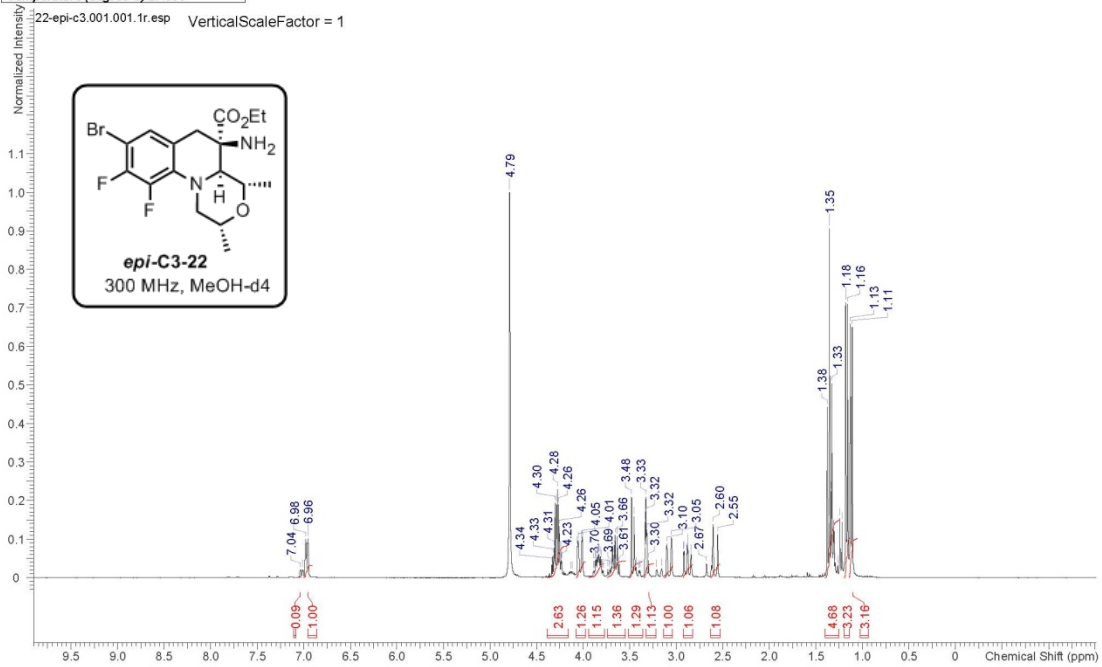
9/2/2015 5:11:46 PM

OriginalDateForRelativeTime 2015-07-18T10:09:04				*Number of Nuclei*	0 F's
Acquisition Time (sec)	0.8716	*Comment*	A3 300MHz fluorine_dec_QNP_64 MeOD {C:\u} fbriones 9	*Date*	18 Jul 2015 10:09:04
Date Stamp	18 Jul 2015 10:09:04		*File Name*	C:\Users\kmwk459\Desktop\OL-NMR\22\4\pdata\1\1r	
Frequency (MHz)	282.38	*Nucleus*	19F	*Number of Transients*	64	*Origin*	spect
Original Points Count	65536	*Owner*	usbodlab	*Points Count*	65536	*Pulse Sequence*	zgfhigqn
Receiver Gain	4597.60	*SW(cyclical) (Hz)*	75187.97	*Solvent*	METHANOL-d4	*Spectrum Offset (Hz)*	-19769.5508
Spectrum Type	STANDARD	*Sweep Width (Hz)*	75186.82	*Temperature (degree C)*	26.860		



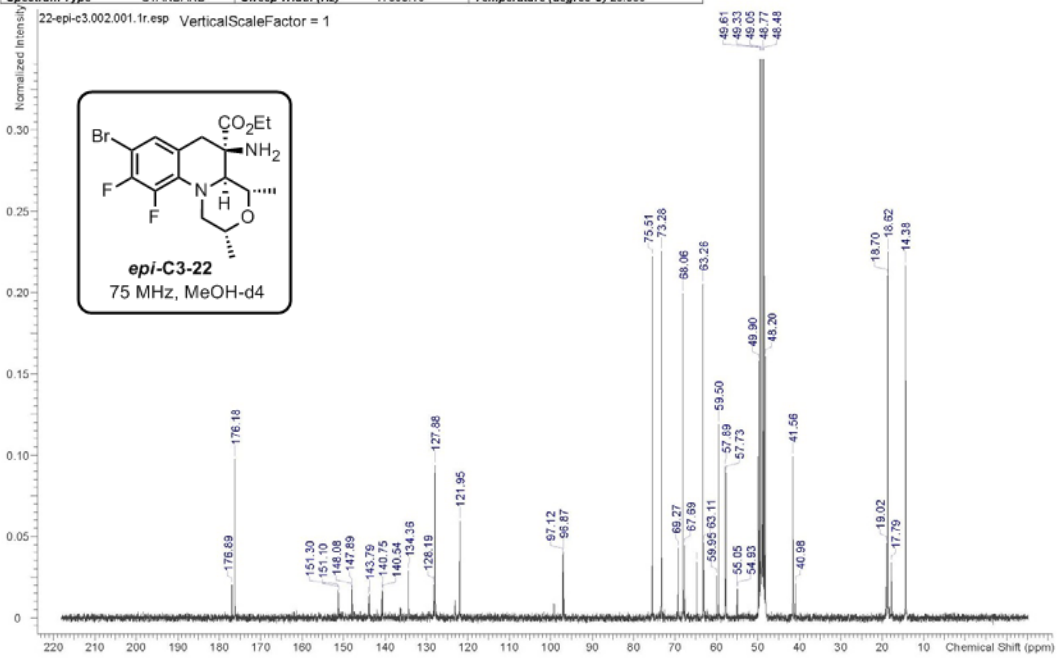
9/2/2015 5:13:24 PM

OriginalDateForRelativeTime 2015-07-17T14:18:40				*Multiplets Integrals Sum*	0.00	*Number of Nuclei*	0 H's
Acquisition Time (sec)	2.6542	*Comment*	A3 300MHz proton_longdelay_QNP_16 MeOD {C:\u} fbriones 10				
Date	17 Jul 2015 14:18:40			*Date Stamp*	17 Jul 2015 14:18:40		
File Name	C:\Users\kmwk459\Desktop\OL-NMR\22-epi-c3\1\pdata\1\1r			*Frequency (MHz)*	300.13	*Nucleus*	1H
Number of Transients	16	*Origin*	spect	*Original Points Count*	16384	*Owner*	usbodlab
Points Count	32768	*Pulse Sequence*	zg30	*Receiver Gain*	228.10	*SW(cyclical) (Hz)*	6172.84
Solvent	METHANOL-d4	*Spectrum Offset (Hz)*	1853.4569	*Spectrum Type*	STANDARD	*Sweep Width (Hz)*	6172.65
Temperature (degree C)	27.060						



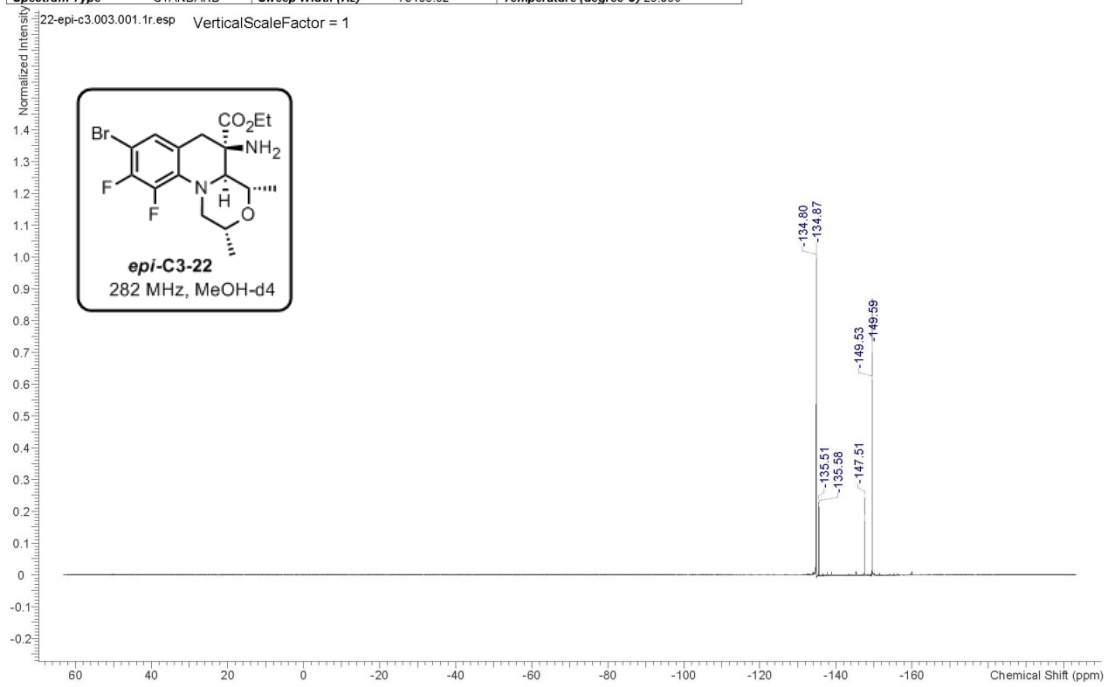
9/2/2015 5:14:07 PM

OriginalDateForRelativeTime 2015-07-18T07:50:24				*Number of Nuclei*	0 C's		
Acquisition Time (sec)	1.8612	*Comment*	A3 300MHz carbon_QNP_8000 MeOD {C:\u} fbriones 10			*Date*	18 Jul 2015 07:50:24
Date Stamp	18 Jul 2015 07:50:24			*File Name*	C:\Users\kmwk459\Desktop\OL-NMR\22-epi-c3\2\pdata\1\1r		
Frequency (MHz)	75.48	*Nucleus*	13C	*Number of Transients*	8000	*Origin*	spect
Original Points Count	32768	*Owner*	usbodlab	*Points Count*	32768	*Pulse Sequence*	zgpg30
Receiver Gain	3251.00	*SW(cyclical) (Hz)*	17605.63	*Solvent*	METHANOL-d4	*Spectrum Offset (Hz)*	7655.1299
Spectrum Type	STANDARD	*Sweep Width (Hz)*	17605.10	*Temperature (degree C)*	26.960		



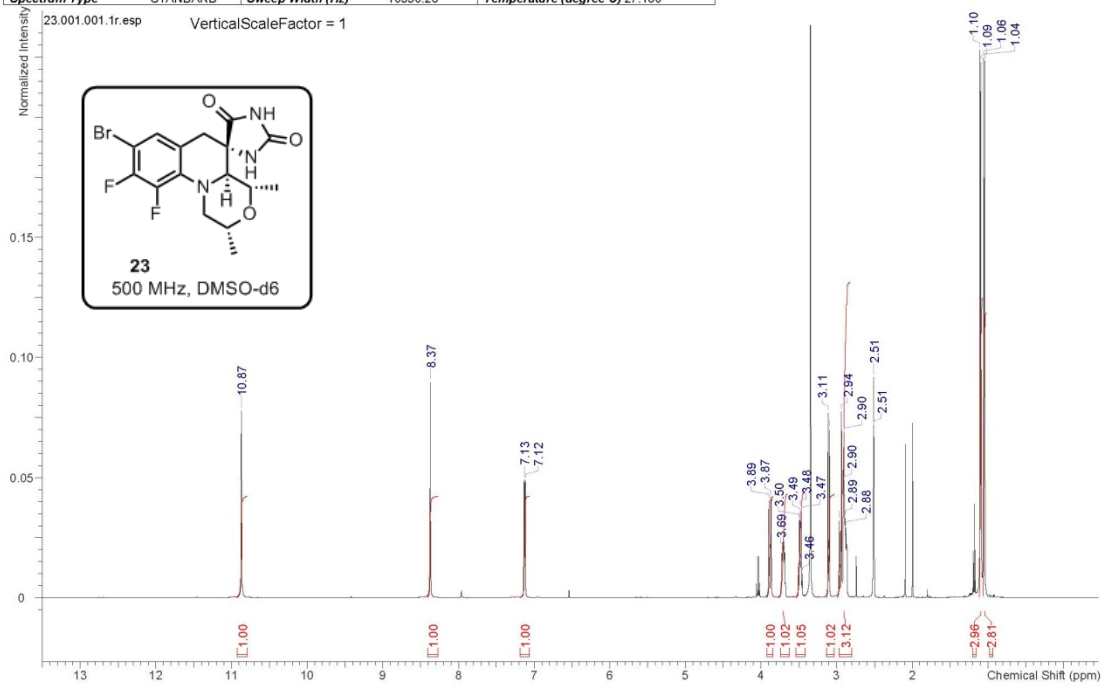
9/2/2015 5:14:49 PM

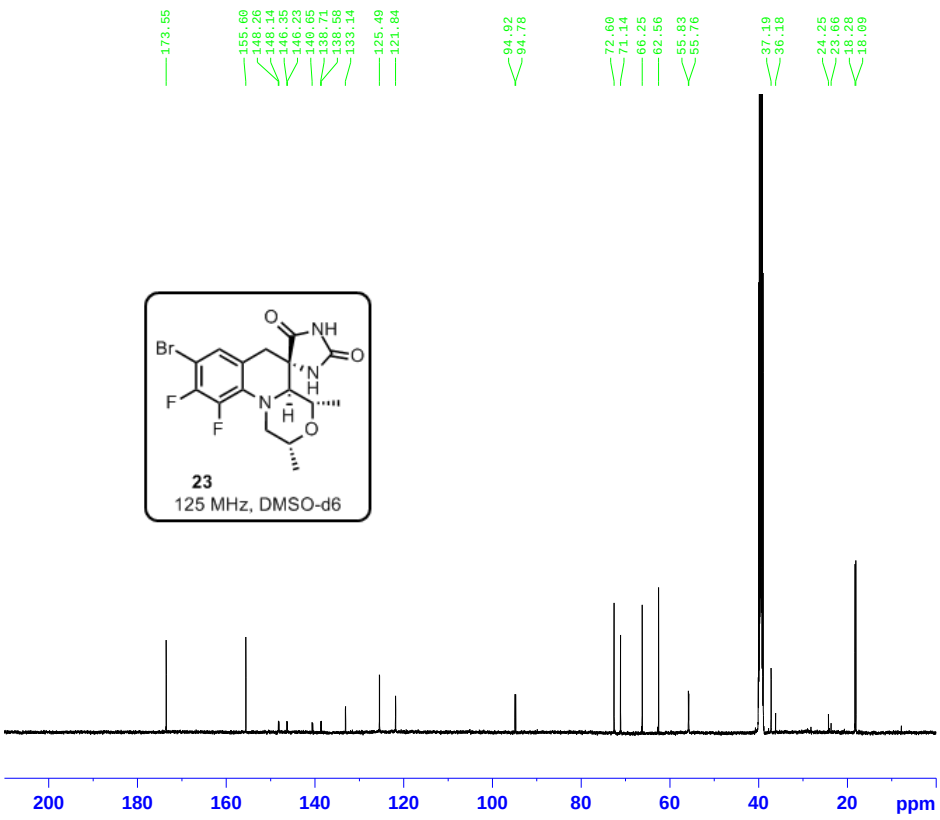
OriginalDateForRelativeTime 2015-07-18T10:19:44				*Multiplets Integrals Sum*	0.00	*Number of Nuclei*	0 F's
Acquisition Time (sec)	0.8716	*Comment*	A3 300MHz fluorine_dec_QNP_64 MeOD {C:\u} fbriones 10			*Date*	18 Jul 2015 10:19:44
Date Stamp	18 Jul 2015 10:19:44			*File Name*	C:\Users\kmwk459\Desktop\OL-NMR\22-epi-c3\3\pdata\1\1r		
Frequency (MHz)	282.38	*Nucleus*	19F	*Number of Transients*	64	*Origin*	spect
Original Points Count	65536	*Owner*	usbodlab	*Points Count*	65536	*Pulse Sequence*	zgfhigqn
Receiver Gain	5792.60	*SW(cyclical) (Hz)*	75187.97	*Solvent*	METHANOL-d4	*Spectrum Offset (Hz)*	-19769.5508
Spectrum Type	STANDARD	*Sweep Width (Hz)*	75186.82	*Temperature (degree C)*	26.960		



9/2/2015 8:32:21 PM

OriginalDateForRelativeTime	2015-07-31T16:16:00			*Multiplets Integrals Sum*	0.00	*Number of Nuclei*	0 H's
Acquisition Time (sec)	3.1719	*Comment*	PROTON DMSO {C:\Bruker\TOPSPIN1.3} fbriones 30			*Date*	31 Jul 2015 16:16:00
Date Stamp	31 Jul 2015 16:16:00			*File Name*	C:\Users\kmwk459\Desktop\OL-NMR\23\1\pdata\1\1r		
Frequency (MHz)	500.13	*Nucleus*	1H	*Number of Transients*	32	*Origin*	spect
Original Points Count	32768	*Owner*	usbodlab	*Points Count*	32768	*Pulse Sequence*	zg30
Receiver Gain	161.30	*SW(cyclical) (Hz)*	10330.58	*Solvent*	DMSO-d6	*Spectrum Offset (Hz)*	3088.5571
Spectrum Type	STANDARD	*Sweep Width (Hz)*	10330.26	*Temperature (degree C)*	27.160		

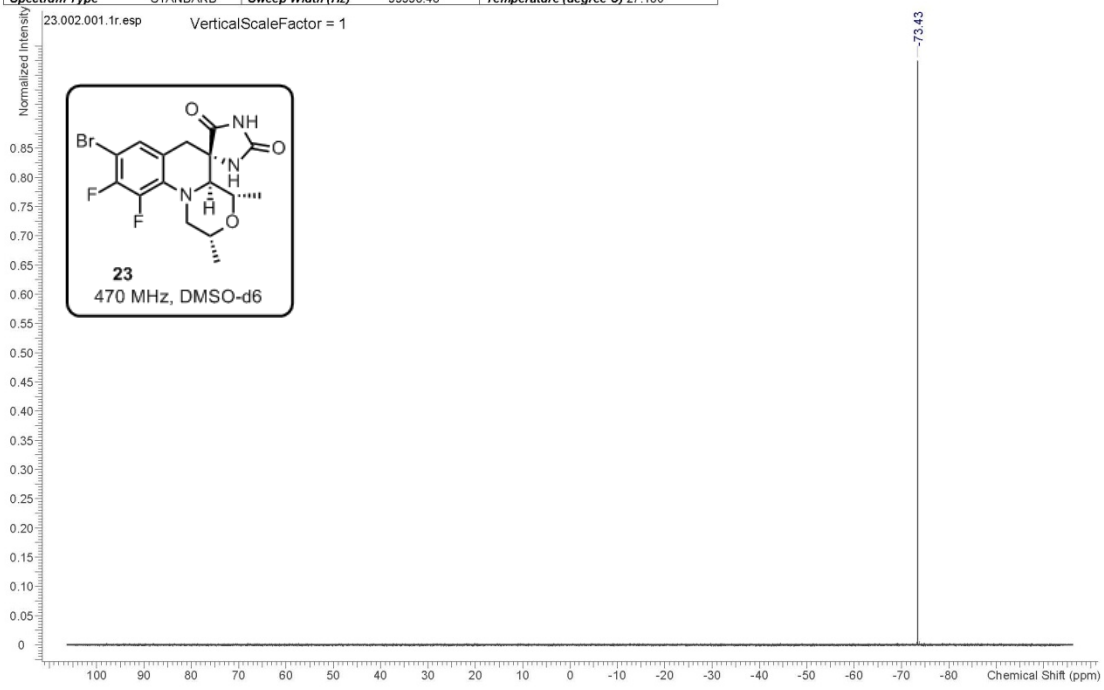


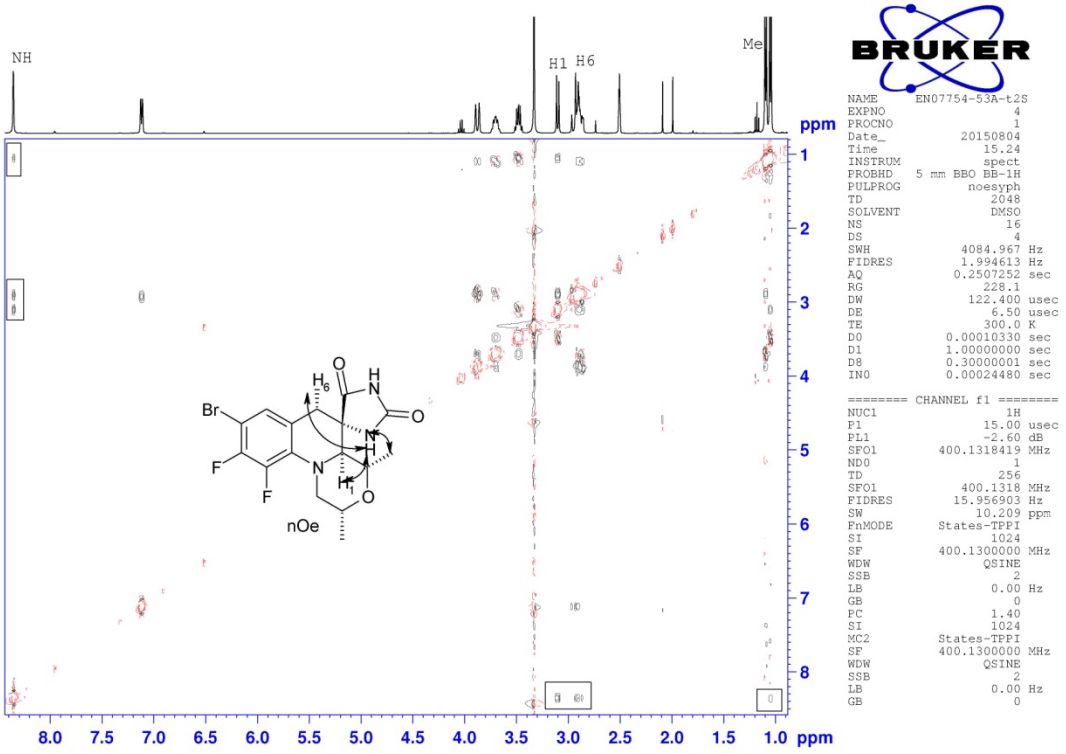



```
NAME          EN07754-6534
EXPNO                    4
PROCNO                   1
Date_             20150903
Time                 10.35
INSTRUM              spect
PROBHD   5 mm CPQNP 1H/
PULPROG             zgpg30
TD                   65418
SOLVENT               DMSO
NS                    1024
DS                       4
SWH             30030.029 Hz
FIDRES           0.459048 Hz
AQ              1.0892597 sec
RG                 3649.1
DW                16.650 usec
DE                35.00 usec
TE                 300.2 K
D1            2.00000000 sec
d11           0.03000000 sec
DELTA         1.89999998 sec
TD0                      1

======== CHANNEL f1 ========
NUC1                   13C
P1                  12.00 usec
PL1                  2.20 dB
SFO1          125.7703643 MHz

======== CHANNEL f2 ========
CPDPRG2             waltz16
NUC2                    1H
PCPD2               80.00 usec
PL2                  1.20 dB
PL12                15.74 dB
PL13                15.89 dB
SFO2          500.1320005 MHz
SI                   32768
SF            125.7578519 MHz
WDW                     EM
SSB                      0
LB                    1.00 Hz
GB                       0
PC                    1.40
```

9/2/2015 8:32:58 PM

OriginalDateForRelativeTime 2015-08-01T01:05:04				*Number of Nuclei*	0 F's
Acquisition Time (sec)	0.6554	*Comment*	F19CPD DMSO {C:\Bruker\TOPSPIN1.3} fbriones 30	*Date*	01 Aug 2015 01:05:04
Date Stamp	01 Aug 2015 01:05:04		*File Name*	C:\Users\kmwk459\Desktop\OL-NMR\23\2\pdata\1\1r	
Frequency (MHz)	470.59	*Nucleus*	19F	*Number of Transients*	8	*Origin*	spect
Original Points Count	65536	*Owner*	usbodlab	*Points Count*	65536	*Pulse Sequence*	zgfhigqn
Receiver Gain	3649.10	*SW(cyclical) (Hz)*	100000.00	*Solvent*	DMSO-d6	*Spectrum Offset (Hz)*	0.0164
Spectrum Type	STANDARD	*Sweep Width (Hz)*	99998.48	*Temperature (degree C)*	27.160		



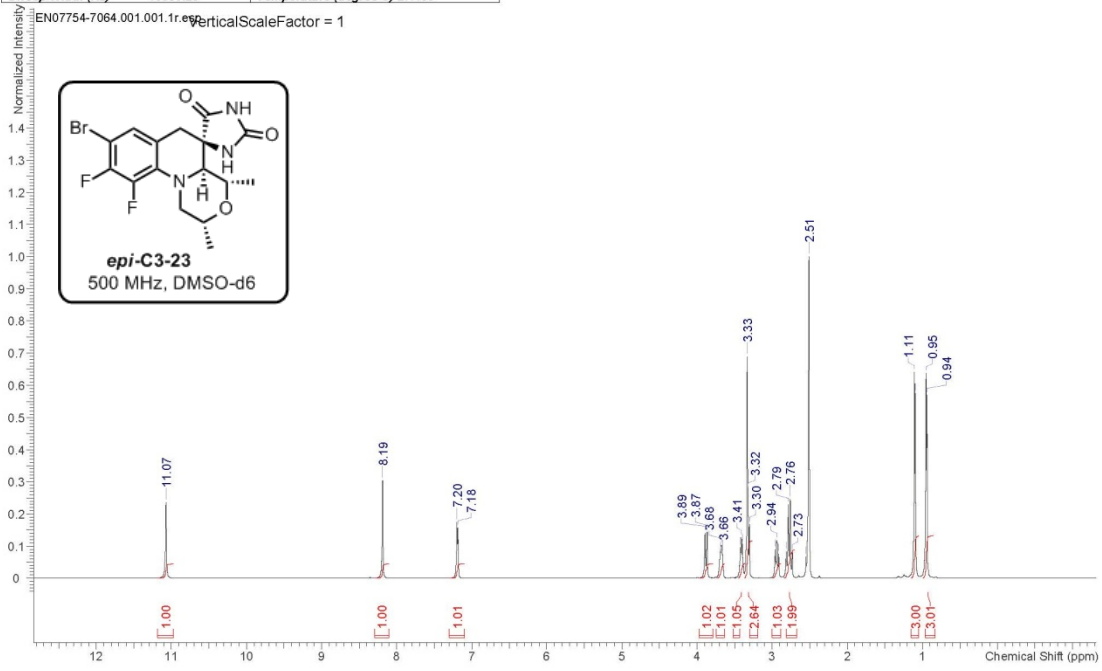


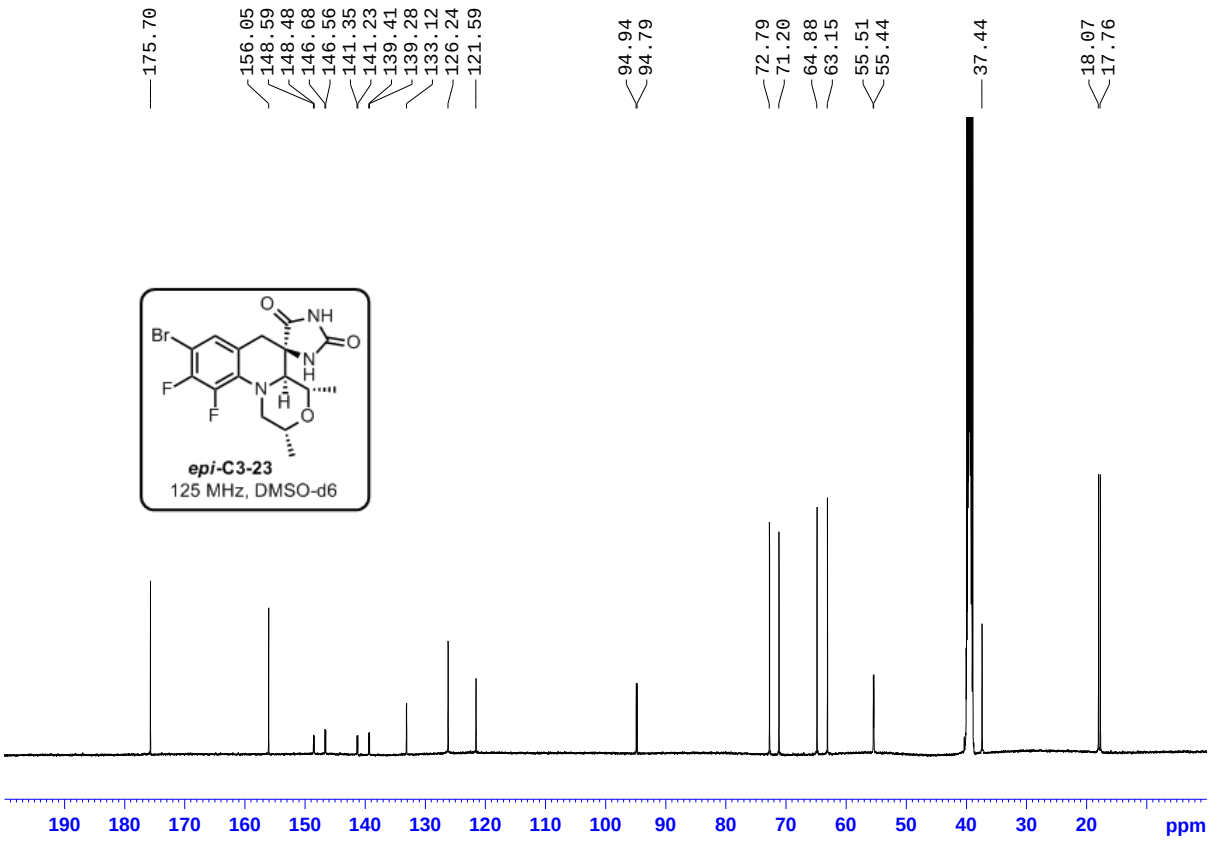
BRUKER

```
NAME          EN07754-53A-t2S
EXPNO                       4
PROCNO                      1
Date_              20150804
Time                   15.24
INSTRUM                spect
PROBHD   5 mm BBO BB-1H
PULPROG             noesyph
TD                       2048
SOLVENT                 DMSO
NS                        16
DS                         4
SWH                4084.967 Hz
FIDRES             1.994613 Hz
AQ                 0.2507252 sec
RG                     228.1
DW                   122.400 usec
DE                     6.50 usec
TE                    300.0 K
D0                 0.00010330 sec
D1                 1.00000000 sec
D8                 0.30000001 sec
IN0                0.00024480 sec

======== CHANNEL f1 ========
NUC1                       1H
P1                      15.00 usec
PL1                     -2.60 dB
SFO1               400.1318419 MHz
ND0                        1
TD                       256
SFO1                400.1318 MHz
FIDRES             15.956903 Hz
SW                     10.209 ppm
FnMODE          States-TPPI
SI                       1024
SF                 400.1300000 MHz
WDW                     QSINE
SSB                        2
LB                      0.00 Hz
GB                         0
PC                      1.40
SI                       1024
MC2             States-TPPI
SF                 400.1300000 MHz
WDW                     QSINE
SSB                        2
LB                      0.00 Hz
GB                         0
```

9/3/2015 10:18:37 AM

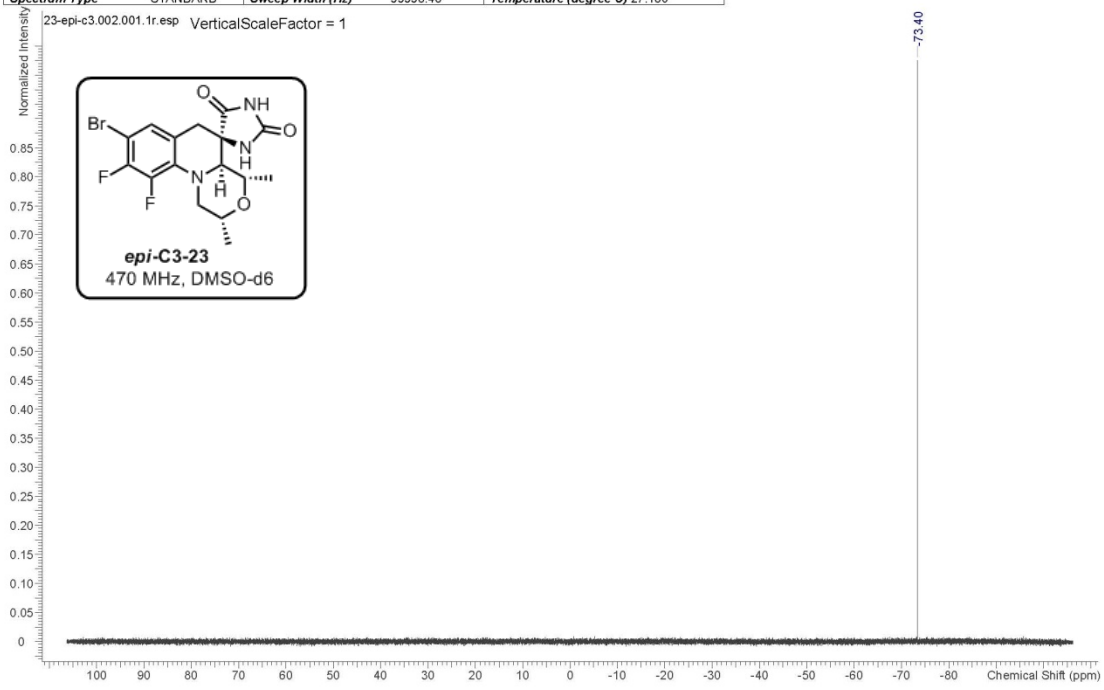
OriginalDateForRelativeTime 2015-08-31T11:49:20			*Multiplets Integrals Sum* 0.00	*Number of Nuclei*	0 H's
Acquisition Time (sec)	3.1719	*Comment*	PROTON DMSO {C:\Bruker\TOPSPIN1.3} fbriones 35	*Date*	31 Aug 2015 11:49:20
Date Stamp	31 Aug 2015 11:49:20				
File Name	\\netapp2\nmr_archive\GHP_B00_500cryo\data\fbriones\nmr\EN07754-7064\1\pdata\1\1r			*Frequency (MHz)*	500.13
Nucleus	1H	*Number of Transients*	32	*Origin* spect	*Original Points Count* 32768
Owner	usbodlab	*Points Count*	32768	*Pulse Sequence* zg30	*Receiver Gain* 203.20
SW(cyclical) (Hz)	10330.58	*Solvent*	DMSO-d6	*Spectrum Offset (Hz)* 3088.5571	*Spectrum Type* STANDARD
Sweep Width (Hz)	10330.26	*Temperature (degree C)* 27.160			

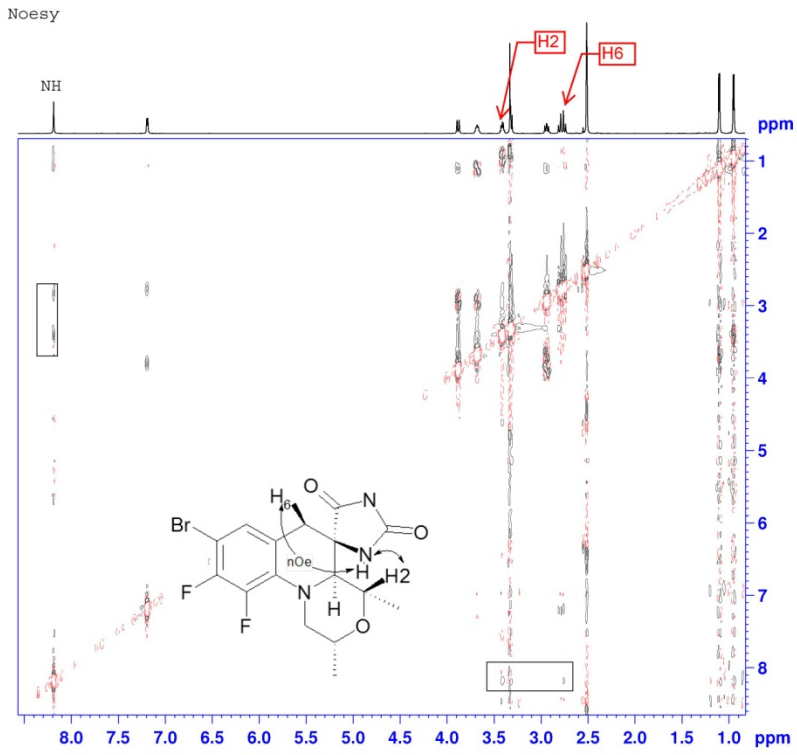




9/2/2015 8:23:14 PM

OriginalDateForRelativeTime 2015-08-01T08:22:24				***Number of Nuclei***	0 F's
Acquisition Time (sec)	0.6554	***Comment***	F19CPD DMSO {C:\Bruker\TOPSPIN1.3} fbriones 31	***Date***	01 Aug 2015 08:22:24
Date Stamp	01 Aug 2015 08:22:24			***File Name***	C:\Users\kmwk459\Desktop\OL-NMR\23-epi-c3\2\pdata\1\1r
Frequency (MHz)	470.59	***Nucleus***	19F	***Number of Transients***	8
Original Points Count	65536	***Owner***	usbodlab	***Points Count***	65536
Receiver Gain	3649.10	***SW(cyclical) (Hz)***	100000.00	***Solvent***	DMSO-d6
Spectrum Type	STANDARD	***Sweep Width (Hz)***	99998.48	***Temperature (degree C)***	27.160
				Origin	spect
				Pulse Sequence	zgfhigqn
				Spectrum Offset (Hz)	0.0164



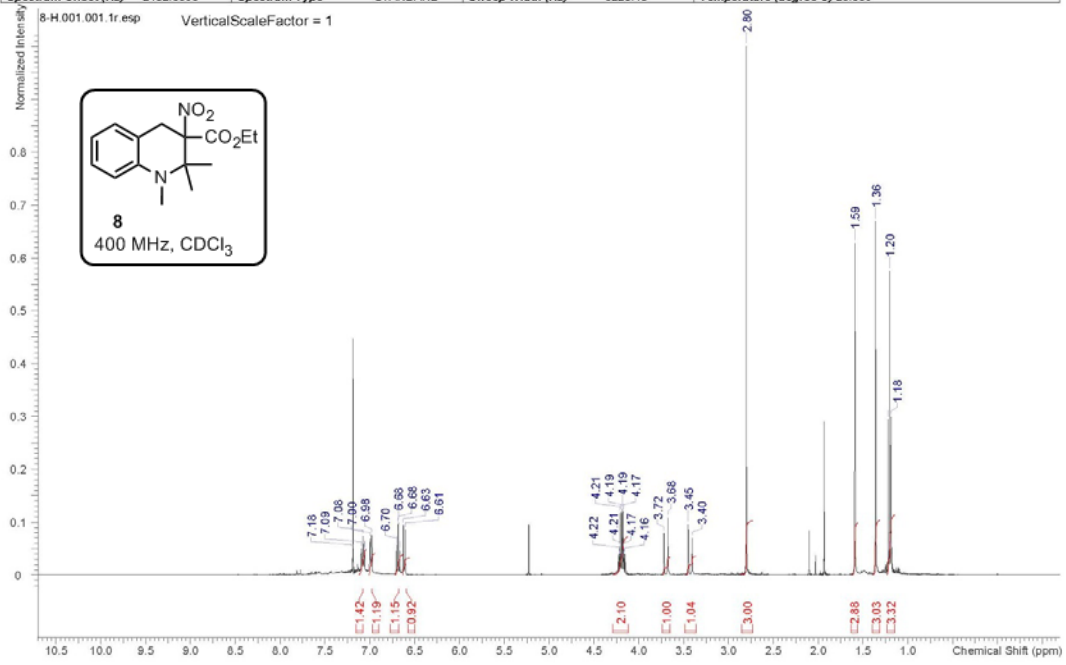


BRUKER

```
NAME                EN07754-91
EXPNO                        3
PROCNO                       1
Date_                 20150901
Time                     10.12
INSTRUM                  spect
PROBHD     5 mm TCI 1H-13
PULPROG              noesyph
TD                        2048
SOLVENT                   DMSO
NS                           8
DS                           4
SWH                 6127.451 Hz
FIDRES              2.991920 Hz
AQ                  0.1671668 sec
RG                         144
DW                     81.600 usec
DE                     30.00 usec
TE                     300.1 K
D0                  0.00007147 sec
D1                  2.00000000 sec
D8                  0.30000001 sec
IN0                 0.00016330 sec

======== CHANNEL f1 ========
NUC1                       1H
P1                     8.00 usec
PL1                    4.70 dB
PL1W                5.87285280 W
SFO1              599.8032989 MHz
ND0                          1
TD                         236
SFO1                599.8033 MHz
FIDRES              25.946711 Hz
SW                     10.209 ppm
FnMODE           States-TPPI
SI                        2048
SF                599.8000000 MHz
WDW                      QSINE
SSB                          2
LB                      0.00 Hz
GB                           0
PC                      1.40
SI                        2048
MC2              States-TPPI
SF                599.8000000 MHz
WDW                      QSINE
SSB                          2
LB                      0.00 Hz
GB                           0
```

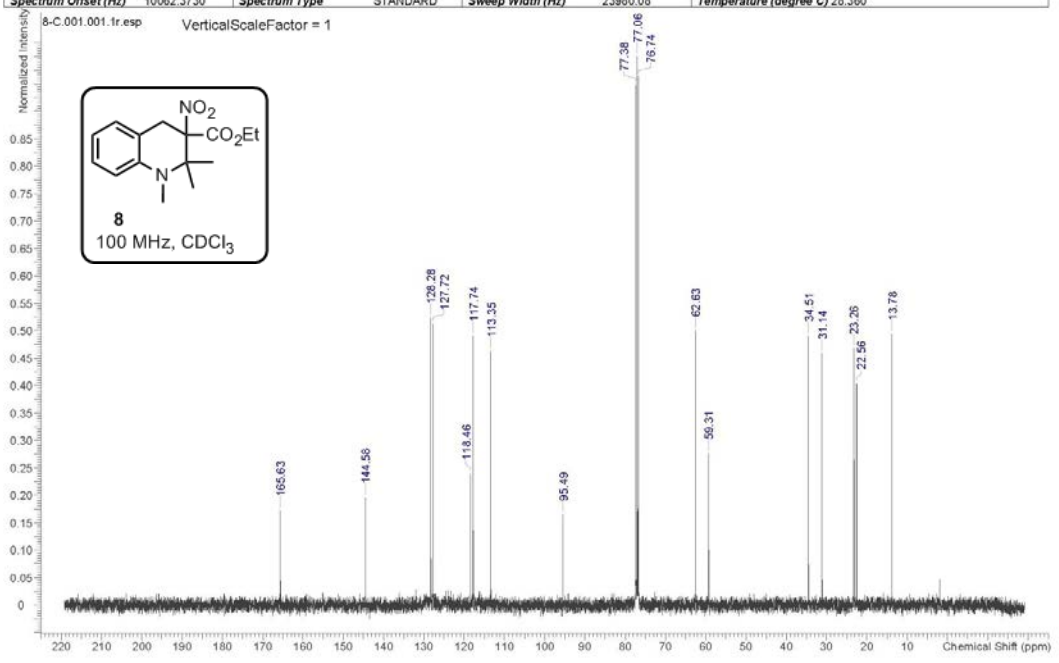
9/2/2015 9:20:49 PM

OriginalDateForRelativeTime 2014-12-08T16:41:20				*Multiplets Integrals Sum* 0.00		*Number of Nuclei* 0 H's	
Acquisition Time (sec) 0.9961	*Comment*	proton_longdelay_QNP_16 CDCl3 {C:\u} fbriones 33				*Date*	08 Dec 2014 16:41:20
Date Stamp 08 Dec 2014 16:41:20				*File Name*	C:\Users\kmwk459\Desktop\OL-NMR\8-H\1\pdata\1\1r		
Frequency (MHz) 400.13	*Nucleus*	1H		*Number of Transients*	16	*Origin*	spect
Original Points Count 8192	*Owner*	usbodlab		*Points Count*	32768	*Pulse Sequence*	zg30
Receiver Gain 181.00	*SW(cyclical) (Hz)*	8223.68		*Solvent*	CHLOROFORM-d		
Spectrum Offset (Hz) 2432.8806	*Spectrum Type*	STANDARD		*Sweep Width (Hz)*	8223.43	*Temperature (degree C)*	26.960



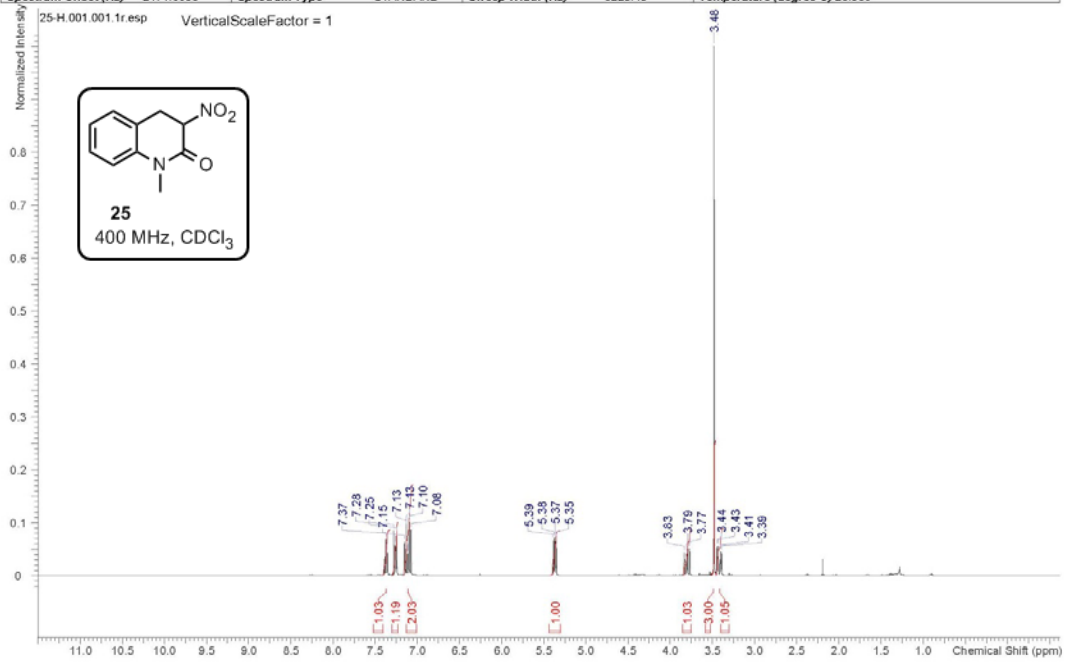
9/2/2015 9:21:20 PM

OriginalDateForRelativeTime 2014-12-08T17:11:12				*Number of Nuclei* 0 C's			
Acquisition Time (sec) 1.3664	*Comment*	carbon_QNP_256 CDCl3 {C:\u} fbriones 27				*Date*	08 Dec 2014 17:11:12
Date Stamp 08 Dec 2014 17:11:12				*File Name*	C:\Users\kmwk459\Desktop\OL-NMR\8-C\1\pdata\1\1r		
Frequency (MHz) 100.62	*Nucleus*	13C		*Number of Transients*	256	*Origin*	spect
Original Points Count 32768	*Owner*	usbodlab		*Points Count*	32768	*Pulse Sequence*	zgpg30
Receiver Gain 18390.40	*SW(cyclical) (Hz)*	23980.81		*Solvent*	CHLOROFORM-d		
Spectrum Offset (Hz) 10062.3730	*Spectrum Type*	STANDARD		*Sweep Width (Hz)*	23980.08	*Temperature (degree C)*	28.360



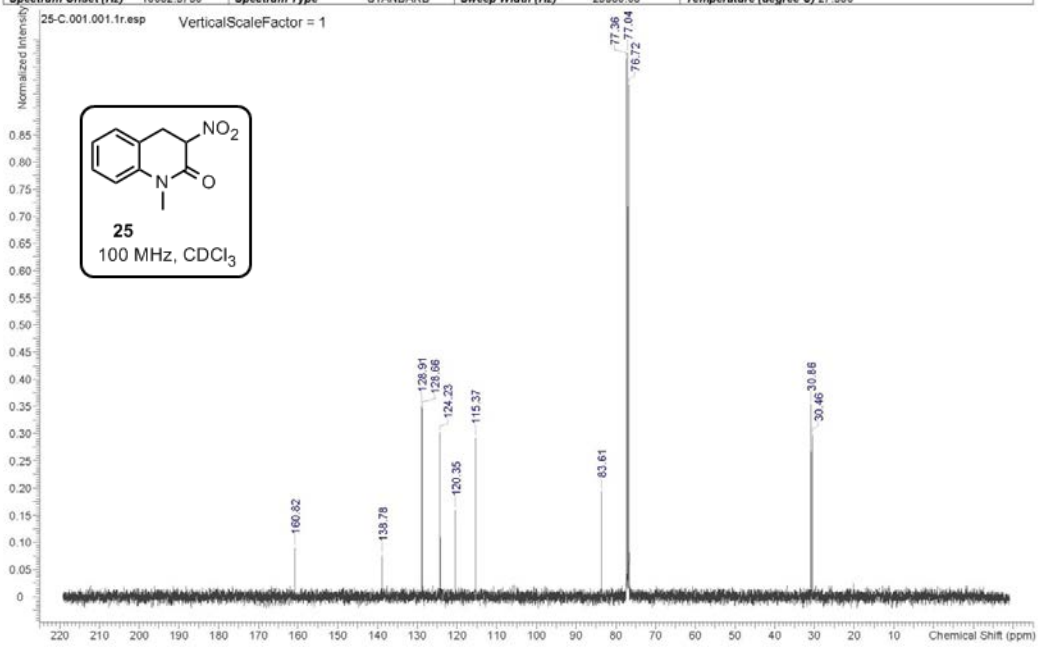
9/2/2015 7:30:44 PM

OriginalDateForRelativeTime 2014-10-24T13:25:20				*Multiplets Integrals Sum* 0.00		*Number of Nuclei* 0 H's	
Acquisition Time (sec) 0.9961	*Comment*	proton_longdelay_QNP_16 CDCl3 {C:\u} fbriones 6				*Date*	24 Oct 2014 13:25:20
Date Stamp 24 Oct 2014 13:25:20				*File Name*	C:\Users\kmwk459\Desktop\OL-NMR\25-H\1\pdata\1\1r		
Frequency (MHz) 400.13	*Nucleus*	1H		*Number of Transients*	16	*Origin*	spect
Original Points Count 8192	*Owner*	usbodlab		*Points Count*	32768	*Pulse Sequence*	zg30
Receiver Gain 128.00	*SW(cyclical) (Hz)*	8223.68		*Solvent*	CHLOROFORM-d		
Spectrum Offset (Hz) 2471.0088	*Spectrum Type*	STANDARD		*Sweep Width (Hz)*	8223.43	*Temperature (degree C)*	26.960



9/2/2015 7:32:11 PM

OriginalDateForRelativeTime 2014-10-24T13:53:04				*Number of Nuclei* 0 C's			
Acquisition Time (sec) 1.3664	*Comment*	carbon_QNP_256 CDCl3 {C:\u} fbriones 6				*Date*	24 Oct 2014 13:53:04
Date Stamp 24 Oct 2014 13:53:04				*File Name*	C:\Users\kmwk459\Desktop\OL-NMR\25-C\1\pdata\1\1r		
Frequency (MHz) 100.62	*Nucleus*	13C		*Number of Transients*	256	*Origin*	spect
Original Points Count 32768	*Owner*	usbodlab		*Points Count*	32768	*Pulse Sequence*	zgpg30
Receiver Gain 14596.50	*SW(cyclical) (Hz)*	23980.81		*Solvent*	CHLOROFORM-d		
Spectrum Offset (Hz) 10062.3730	*Spectrum Type*	STANDARD		*Sweep Width (Hz)*	23980.08	*Temperature (degree C)*	27.560



9/2/2015 7:33:00 PM

OriginalDateForRelativeTime 2015-08-19T23:20:32				***Number of Nuclei***	0 C's		
Acquisition Time (sec)	1.8219	***Comment***	A3 300MHz dept_ONP_2000 CDCl3 {C:\u} fbriones 48			***Date***	19 Aug 2015 23:20:32
Date Stamp	19 Aug 2015 23:20:32			***File Name***	C:\Users\kmwk459\Desktop\OL-NMR\25-dept\1\pdata\1\1r		
Frequency (MHz)	75.48	***Nucleus***	13C	***Number of Transients***	2000	***Origin***	spect
Original Points Count	32768	***Owner***	usbodlab	***Points Count***	32768	***Pulse Sequence***	dept135
Receiver Gain	16384.00	***SW(cyclical) (Hz)***	17985.61	***Solvent***	CHLOROFORM-d		
Spectrum Offset (Hz)	7547.9526	***Spectrum Type***	DEPT135	***Sweep Width (Hz)***	17985.06	***Temperature (degree C)***	26.960