

Extending the scope of oleic acid catalysis in diversity-oriented synthesis of chromene and pyrimidine based scaffolds

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Supporting information

Materials and methods

All the chemicals were received from sigma Aldrich, Loba chemicals, Merck, Avra synthesis or SD Fine chemicals. The Oleic acid was obtained from (Pdt No: 61821625001730) Merck chemicals, and its used as such any further purification The 2-hydroxychalcone were synthesized through reported Literature. Melting points were found *via* microscopic melting point apparatus and those were uncorrected. Subsequently the characterization of NMR (H^1) were determined by a Bruker Av-300MHz spectrometer TMS as internal standard and $CDCl_3$, DMSO- d_6 as external solvent. Labomed LX- 400 Microscope were used for images. All the reactions were carried out to the 10 ML round bottom flask with magnetic stirrer.

General procedure for synthesis of 4H-chromene derivatives synthesis (3a-g)

The 2-hydroxychalcone (1.0 mmol) and indole (1mmol, 117 mg) were added to the mixture of oleic acid (40 μ L) in water at room temperature. Then the reaction mixture was refluxed 4 hours. The progress of the reaction was monitored by TLC. After completion of reaction, the reaction mixture was cooled to room temperature. Then the reaction mixture was extracted with ethyl acetate (2×20 mL) and the combined extracts were washed with water (1×10 mL) and dried over anhydrous sodium sulphate. The solvents were removed under reduced pressure and the crude residue was purified through the silica gel column chromatography (eluent ethyl acetate, hexane solvent mixture). Eventually pale red solid was obtained with good yield.

3-(2-phenyl-4H-chromen-4yl)-1H-indole (3a)

Yield = 228 mg (71 %) Brown solid. Mp 99-101 $^{\circ}$ C (Lit 100-102 $^{\circ}$ C)¹. H^1 NMR (300 MHz, $CDCl_3$, δ ppm): 5.17 (d, $J = 3.9$ Hz, 1H), 5.68 (d, $J = 4.2$ Hz, 1H), 6.92 (td, $J = 7.8, 1.8$ Hz, 1H), 7.02-7.19 (m, 6H), 7.31-7.39 (m, 4H), 7.62 (d, $J = 7.8$ Hz, 1H), 7.72 (d, $J = 7.2$ Hz, 2H), 7.99(brs, 1H).

2-methyl-3-(2-phenyl-4H-chromen-4yl)-1H-indole (3b)

Yield = 217 mg (67 %) Brown solid, Mp 97-99 $^{\circ}$ C (Lit 97-98 $^{\circ}$ C)¹. H^1 NMR (300 MHz, $CDCl_3$, δ ppm): 2.39 (s, 3H), 5.19 (d, $J = 3.6$ Hz, 1H), 5.57 (d, $J = 3.6$ Hz, 1H), 6.86-6.98 (m, 3H), 7.04-7.15 (m, 3H), 7.27-7.40 (m, 4H), 7.45 (d, $J = 7.8$ Hz, 1H), 7.72 (dd, $J = 7.8, 1.2$ Hz, 2H), 7.78(brs, 1H)

5-methoxy-3-(2-phenyl-4H-chromen-4yl)-1H-indole (3c)

Yield = 220mg mg (68 %) Gum-like red (lit)¹ oil., H¹ NMR (300 MHz, CDCl₃, δ ppm): 3.74 (s, 3H), 5.13 (d, *J* = 3.9 Hz, 1H), 5.67 (d, *J* = 3.7 Hz, 1H), 6.82 (dd, *J* = 8.7, 2.4 Hz, 1H), 6.93 (td, *J* = 7.8, 1.8 Hz, 1H), 7.06 (t, *J* = 2.7 Hz, 2H), 7.11-7.23 (m, 3H), 7.31-7.47 (m, 3H), 7.72 (dd, *J* = 7.8, 1.2 Hz, 2H), 7.91 (brs, 1H).

3-[2-(2-bromophenyl)-4H-chromen-4-yl]-1H-indole (3d)

Yield = 242 mg (60 %) Gum-like red (lit)¹. H¹ NMR (300 MHz, CDCl₃, δ ppm): 5.17 (d, *J* = 3.9 Hz, 1H), 5.35 (d, *J* = 3.9 Hz, 1H), 6.92 (dd, *J* = 7.8, 1.2 Hz, 1H), 7.04-7.08 (m, 2H), 7.10-7.15 (m, 3H), 7.20 (td, *J* = 7.8, 1.8 Hz, 2H), 7.30 (td, *J* = 7.5, 1.2 Hz, 1H), 7.35 (d, *J* = 8.1 Hz, 1H), 7.47 (dd, *J* = 7.8, 1.5 Hz, 1H), 7.62 (dd, *J* = 8.1, 1.2 Hz, 1H), 7.72 (d, *J* = 7.8 Hz, 1H), 8.01 (brs, 1H)

3-[2-(4-chlorophenyl)-4H-chromen-4-yl]-1H-indole (3e)

Yield = 246 mg (69 %) Brown solid, Mp 80-81 °C (Lit 84-86 °C)¹. H¹ NMR (300 MHz, CDCl₃, δ ppm): 5.16 (d, *J* = 4.2 Hz, 1H), 5.66 (d, *J* = 3.9 Hz, 1H), 6.93 (td, *J* = 6.9, 1.5 Hz, 1H), 7.02-7.08 (m, 2H), 7.13 (td, *J* = 7.8, 1.5 Hz, 2H), 7.17-7.20 (m, 2H), 7.33 (dt, *J* = 8.7, 2.4 Hz, 2H), 7.37 (d, *J* = 0.6 Hz, 1H), 7.59 (d, *J* = 7.8 Hz, 1H), 7.65 (dt, *J* = 8.7, 2.4 Hz, 2H), 8.03 (brs, 1H)

3-[2-(4-methoxyphenyl)-4H-chromen-4-yl]-1H-indole (3f)

Yield = 247 mg (70 %) Brown solid, Mp 97-98 °C (Lit 97-98 °C)¹. H¹ NMR (300 MHz, CDCl₃, δ ppm): 3.82 (s, 3H), 5.15 (d, *J* = 3.9 Hz, 1H), 5.55 (d, *J* = 3.9 Hz, 1H), 6.87-6.95 (m, 3H), 7.02-7.21 (m, 6H), 7.35 (d, *J* = 8.1 Hz, 1H), 7.61-7.68 (m, 3H), 8.02 (brs, 1H).

3-[2-(4-methylphenyl)-4H-chromen-4-yl]-1H-indole (3g)

Yield = 235 mg (70 %) Gum-like red oil (lit)¹. H¹ NMR (300 MHz, CDCl₃, δ ppm): 2.36 (s, 3H), 5.15 (d, *J* = 3.9 Hz, 1H), 5.62 (d, *J* = 3.9 Hz, 1H), 6.90 (td, *J* = 8.1, 1.8 Hz, 1H), 7.04 (td, *J* = 7.8, 0.9 Hz, 2H), 7.09-7.24 (m, 6H), 7.34 (d, *J* = 7.8 Hz, 1H), 7.61 (dd, *J* = 8.1, 0.9 Hz, 3H), 7.98 (brs, 1H).

General procedure for synthesis of 4H-chromene derivatives synthesis (3h-n)

To a stirred solution of 2-hydroxy aldehyde (1 mmol), malononitrile (1 mmol, 66 μL) and indole (1 mmol, 117 mg) were added to the oleic acid (40 μL) in water (3 mL) at room temperature. The reaction mixture was stirred further 3 hours. After completion of the reaction, the reaction mixture was extracted with ethyl acetate (2 x 20 mL) and the combined extracts were washed with water (1 x 10 mL). The organic layer was dried over sodium sulphate, filtered and the solvent were removed under reduced pressure. After that the crude was purified through

the silica gel column chromatography eluted by ethyl acetate, hexane solvent mixture. Eventually dark yellow solid was obtained with good yield.

2-amino-4-(1H-indol-3-yl)-4H-chromene-3-carbonitrile (3h)

Yield = 236 mg (82 %) Yellow solid Mp 192–194 °C (Lit 193–194 °C)² H¹ NMR (300 MHz, DMSO(d₆), δ ppm): 4.99 (s, 1H), 6.83-6.89 (m, 3H), 6.99-7.08 (m, 4H), 7.19-7.24 (m, 2H), 7.29-7.35 (m, 2H), 10.93 (s, 1H).

2-amino-4-(1-methyl-1H-indol-3-yl)-4H-chromene-3-carbonitrile (3i)

Yield = 256 mg (85 %) Yellow solid. Mp 200-202 °C (Lit 202°C)² H¹ NMR (300 MHz, DMSO(d₆), δ ppm): 3.74 (s, 3H), 4.99 (s, 1H), 6.89-7.13 (m, 6H), 7.21 (t, *J* = 8.1 Hz, 1H), 7.28 (d, *J* = 9.9 Hz, 2H), 7.38 (d, *J* = 8.4 Hz, 1H).

3-amino-1-(1H-indol-3-yl)-1H-benzo[f]chromene-2-carbonitrile (3j)

Yield = 259 mg (77 %) Yellow solid. Mp 212–214 °C (Lit 215–217 °C)² H¹ NMR (300 MHz, DMSO(d₆), δ ppm): 5.62 (s, 1H), 6.79-6.86 (m, 3H), 6.96 (t, *J* = 7.2 Hz, 1H), 7.20 (d, *J* = 8.1 Hz, 1H), 7.27 (d, *J* = 8.1 Hz, 1H), 7.35-7.44 (m, 4H), 7.84-7.90 (m, 2H), 8.10 (d, *J* = 7.8 Hz, 1H), 10.90 (s, 1H).

2-amino-4-(1-methyl-1H-pyrrol-2-yl)-4H-chromene-3-carbonitrile (3k)

Yield = 210 mg (85%) Yellow solid. Mp 196-198 °C, H¹ NMR (300 MHz, DMSO(d₆), δ ppm): 3.43 (s, 3H), 4.97 (s, 1H), 5.76-5.87 (m, 2H), 6.59 (s, 1H), 6.92-7.11 (m, 5H), 7.56 (t, *J* = 7.5 Hz, 1H).

2-amino-4-(1H-pyrrol-2-yl)-4H-chromene-3-carbonitrile (3l)

Yield = 200 mg (84%) Gray colour solid. Mp 195-197°C (Lit 195-197 °C)² H¹ NMR (300 MHz, DMSO(d₆), δ ppm): 4.75 (s, 1H), 5.82-5.88 (m, 2H), 6.59 (s, 1H), 6.88 (s, 2H), 6.98-7.09 (m, 2H), 7.16-7.24 (m, 2H), 10.69 (s, 1H).

2-amino-4-(2-methyl-1H-indol-3-yl)-4H-chromene-3-carbonitrile (3m)

Yield = 238 mg (79 %) Yellow solid. Mp 186–188°C (Lit 187–188 °C)² H¹ NMR (300 MHz, DMSO (d₆), δ ppm): 2.44 (s, 3H), 5.06 (s, 1H), 6.76-6.79 (m, 3H), 6.90-7.06 (m, 5H), 7.16-7.24 (m, 2H), 10.85 (s, 1H).

2-amino-4-(6-methoxy-1H-indol-3-yl)-4H-chromene-3-carbonitrile (3n)

Yield = 257 mg (81 %) Yellow solid. Mp 199-201 °C (Lit 198-200 °C)² H¹ NMR (300 MHz, DMSO(d₆), δ ppm): 3.65 (s, 3H), 4.96 (s, 1H), 6.68-6.76 (m, 2H), 6.83 (s, 2H), 6.99-7.12 (m, 3H), 7.21 (t, *J* = 10.5 Hz, 3H), 10.76 (s, 1H).

Intermediate isolation from the reaction mixture (Scheme 3 in main article)

5-[(2-hydroxyphenyl)methylidene]-1,3-diazinane-2,4,6-trione (6)

Yield = 180 mg (78 %) Orange solid. Mp >260 °C ^1H NMR (300 MHz, DMSO(d₆), δ ppm): 6.82 (t, $J = 7.5$ Hz, 1H), 6.93 (d, $J = 8.1$ Hz, 1H), 7.37 (t, $J = 7.5$ Hz, 1H), 8.16 (d, $J = 8.1$ Hz, 1H), 8.61 (s, 1H), 10.63 (s, 1H), 11.15 (s, 1H), 11.31 (s, 1H).

General procedure for synthesis of Synthesis of 5-(2,3,4,5-tetrahydro-1H-chromeno-[2,3-d]pyrimidin-5-yl)pyrimidiones (7a-c)

To a stirred solution of salicylaldehyde or 5-bromo salicylaldehyde (0.5 mmol) and barbituric acid or thiobarbituric acid (1 mmol) in water (3 mL), oleic acid (40 μL) was added and the mixture was heated at 50 °C for 1 hour. After completion the reaction indicated by TLC, the reaction mixture was cooled and filtered. The precipitate was washed with ethanol to afford pure product.

5-(2,4-Dioxo-2,3,4,5-tetrahydro-1H-chromeno[2,3-d]pyrimidin-5-yl)pyrimidine-2,4,6(1H,3H,5H)-trione (7a)

Yield = 157 mg (92 %) white solid. Mp >225°C (Lit 268-270 °C)³ ^1H NMR (300 MHz, DMSO(d₆), δ ppm): 3.85 (d, $J = 2.1$ Hz, 1H), 4.71 (d, $J = 2.1$ Hz, 1H), 7.08- 7.24 (m, 3H), 7.31- 7.36 (m, 1H), 10.99 (s, 1H), 11.19 (s, 1H), 11.30 (s, 1H), 11.98 (s, 1H).

5-{4-oxo-2-sulfanylidene-1H,2H,3H,4H,5H-chromeno[2,3-d]pyrimidin-5-yl}-2-sulfanylidene-1,3-diazinane-4,6-dione (7b)

Yield = 170 mg (91 %) white solid. Mp >225°C (Lit > 300 °C)³ ^1H NMR (300 MHz, DMSO (d₆), δ ppm): 5.12 (s, 1H), 6.99 (d, $J = 7.5$ Hz, 1H), 7.11 (s, 2H), 7.22 (s, 1H), 11.94 (brs, 1H), 12.24 (brs, 1H), 12.34 (s, 1H), 13.29 (s, 1H).

5-(2,4-Dioxo-7-bromo-2,3,4,5-tetrahydro-1H-chromeno[2,3-d]pyrimidin-5-yl)pyrimidine-2,4,6(1H,3H,5H)-trione (7c)

Yield = 185 mg (88 %) white solid. Mp >225°C (Lit > 240-242 °C)³ ^1H NMR (300 MHz, DMSO(d₆), δ ppm): 3.93 (brs, 1H), 4.72 (brs, 1H), 7.10 (s, 1H), 7.29 (s, 1H), 7.52 (d, $J = 3.9$ Hz, 1H), 11.10 (s, 1H), 11.23 (s, 1H), 11.37 (s, 1H), 12.06 (s, 1H)

General procedure for synthesis of C-glycosylated pyrimidine-fused heterocycle (8)

To a stirred mixture of barbituric acid (1 mmol, 128 mg), D- glucose (0.5 mmol, 90 mg) and oleic acid (40 μ L) in ethanol (3 mL) at 50 $^{\circ}$ C was stirred 12 h. After completion the reaction confirmed by TLC, the reaction mixture cooled at room temperature and filtered. The mixture washed with ethanol thrice and evaporated reduced pressure affords a pure product.

5-((1R,2S,3S,4S)-1,2,3,4,5-Pentahydroxypentyl)-5,9-dihydro-2H-pyrano[2,3-d:6,5-d']dipyrimidine-2,4,6,8(1H,3H,7H)-tetraone (8)

Yield = 284 mg (71 %) white solid. Mp 200-201 $^{\circ}$ C (Lit 202-204 $^{\circ}$ C)⁴ 1 H NMR (300 MHz, DMSO(d₆), δ ppm): 2.50-2.98 (m, 2H), 3.11 (t, J = 7.1 Hz, 1H), 3.26-3.30 (m, 1H), 3.39-3.35 (m, 2H), 3.61-3.70 (m, 5H), 3.89(s, 1H), 11.07 (s, 2H), 11.12 (s, 1H), 11.21(s, 1H).

General procedure for synthesis of pyrazolo pyranopyrimidine derivatives synthesis 10a-f

To a stirred solution of hydrazine hydrate (1 mmol, 50 μ L) and ethylacetoacetate (1 mmol, 130 μ L) in water (3 mL), oleic acid (40 μ L) was added and the mixture was refluxed over 30 min. Next an appropriate amount aromatic aldehyde (1 mmol), barbituric acid (128 mg, 1mmol) were added in the reaction mixture was further refluxed for an appropriate time. After completion the reaction indicated by TLC, the reaction mixture was cooled and filtered. The precipitate was washed with ethanol to afford pure product.

3-Methyl-4-(4-nitrophenyl)-1,4-dihydropyrazolo[4',3':5,6]pyrano[2,3-d]pyrimidine-5,7(6H,8H)-dione(10 a)

Yield = 296 mg (87 %) white solid. Mp >225 $^{\circ}$ C (Lit 233-234 $^{\circ}$ C)⁵ 1 H NMR (300 MHz, DMSO (d₆), δ ppm): 2.25 (s, 3H), 5.51 (s, 1H), 7.31(d, J = 8.7 Hz, 2H), 8.11 (d, J = 8.4 Hz, 2H), 10.25 (s, 2H), 13.40 (brs, 1H).

3-Methyl-4-(3-nitrophenyl)-1,4-dihydropyrazolo[4',3':5,6]pyrano[2,3-d]pyrimidine-5,7(6H,8H)-dione (10 b)

Yield = 283 mg (83 %) white solid. Mp >225 $^{\circ}$ C (Lit 266-267 $^{\circ}$ C)⁵ 1 H NMR (300 MHz, DMSO (d₆), δ ppm): 2.09 (s, 3H), 5.30 (s, 1H), 7.31(s, 2H), 7.63 (s, 1H), 7.91 (s, 1H), 10.05 (s, 2H), 13.19 (brs, 1H).

3-Methyl-4-phenyl-1,4-dihydropyrazolo[4',3':5,6]pyrano[2,3-d]pyrimidine-5,7(6H,8H)-dione (10c)

Yield = 230 mg (78 %) white solid. Mp 216-218°C (Lit 218-219°C)⁵ H¹ NMR (300 MHz, DMSO (d₆), δ ppm): 2.23 (s, 3H), 5.43 (s, 1H), 7.04-7.13(m, 3H), 7.18-7.23 (m, 2H), 10.18 (s, 2H), 13.14 (brs, 1H).

3-Methyl-4-(4-methylphenyl)-1,4-dihydropyrazolo[4',3':5,6]pyrano[2,3-d]pyrimidine-5,7(6H,8H)-dione(10 d)

Yield = 254 mg (82 %) white solid. Mp 197-198 °C (Lit 200-201°C)⁵ H¹ NMR (300 MHz, DMSO (d₆), δ ppm): 2.08 (s, 3H), 2.11 (s, 3H), 4.73 (s, 1H), 7.03-7.24 (m, 4H), 10.93 (s, 2H), 11.74 (brs, 1H).

3-Methyl-4-(2-nitrophenyl)-6,8-dihydropyrazolo[4, '3':5,6]pyrano[2,3-d]pyrimidine-5,7(1H,4H)-dione (10 e)

Yield = 291mg (85 %) pale yellow solid. Mp 207-208°C (Lit 208-209°C)⁵ H¹ NMR (300 MHz, DMSO (d₆), δ ppm): 2.21 (s, 3H), 5.77 (s, 1H), 7.33-7.41 (m, 2H), 7.50-7.76 (m, 2H) 10.16 (s, 2H), 13.19 (brs, 1H)

4-(4-Chlorophenyl)-3-methyl-6,8-dihydropyrazolo[4',3'5,6]-pyrano[2,3-d]pyrimidine-5,7(1H,4H)-dione(10 f)

Yield = 264 mg (80 %) white solid. Mp 220-222°C (Lit 222-223°C)⁵ H¹ NMR (300 MHz, DMSO (d₆), δ ppm): 2.08 (s, 3H), 2.11 (s, 3H), 4.73 (s, 1H), 7.03-7.24 (m, 4H), 10.93 (s, 2H), 11.74 (brs, 1H).

Procedure for synthesis of curcumin-based pyranof[2,3-d]pyrimidine 11:

To a magnetically stirred solution of curcumin (0.5 mmol, 184 mg), 4-chlorobenzaldehyde (0.5 mmol, 70 mg) and barbituric acid (0.5 mmol, 62 mg) in EtOH (2 ml) was oleic acid (40 μL) at reflux temperature of EtOH. The mixture was stirred for 6 h. After completion of the reaction (monitored by TLC), the reaction mixture cooled down to room temperature and then the precipitate filtered, washed with EtOH (3 x 5 mL) thrice and dried under vacuum.

Progress of the reaction monitored as photograph.



A

B

C

- A) Initial reaction mixture of curcumin, 4-chlorobenzaldehyde, barbituric acid and oleic acid in ethanol
- B) Homogeneous solution of the reaction mixture after 2 hours.
- C) After completion of the reaction precipitate was formed

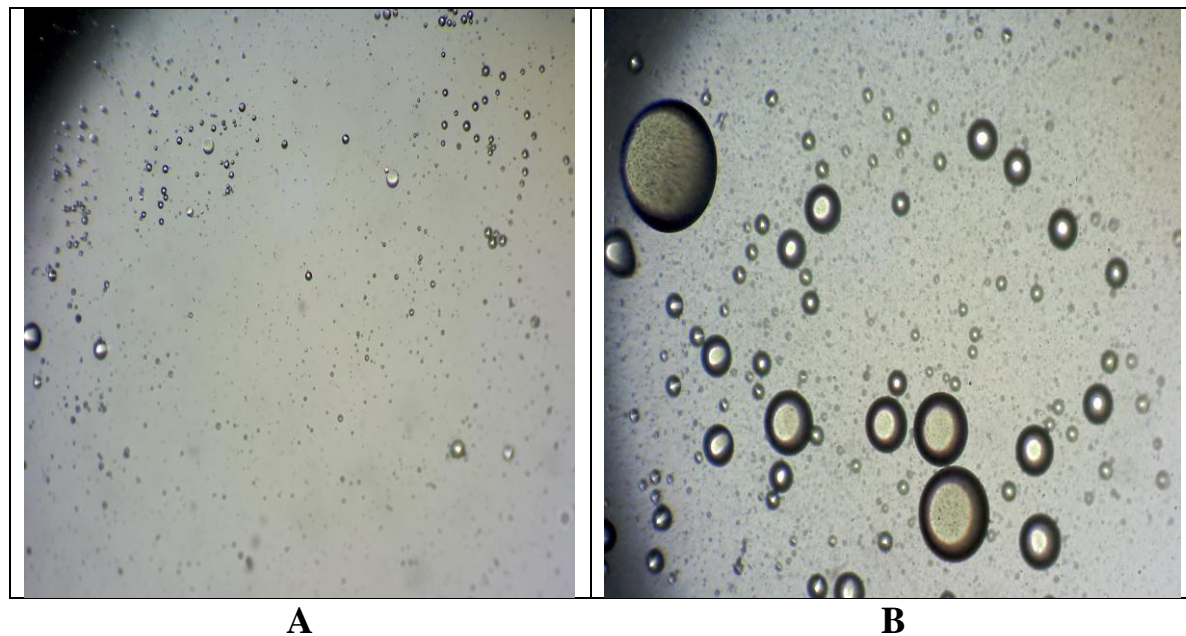
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Yield = 261 mg (87 %) yellow solid. Mp 228-230 °C (lit 152-156 °C) ⁶ H¹ NMR (300 MHz, DMSO(d₆), δ ppm): 3.71 (s, 6H), 5.19 (s, 1H), 6.14(d, *J* = 15.6, 1H), 6.50 (d, *J* = 8.1 Hz, 2H), 6.61-6.82 (m, 5H), 6.88-6.97 (m, 2H), 7.23-7.38 (m, 4H), 9.09 (s, 1H), 9.67 (s, 1H), 11.25 (s, 1H), 11.32 (s, 1H).

References:

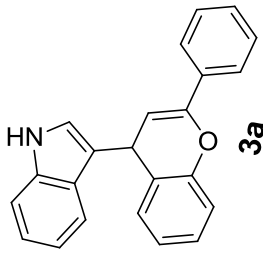
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Light microscopic images of oleic acid in water



- A) Light microscopic image of oleic acid(40 μ L) in water (3 mL)
- B) Light microscopic image of Indole (1 mmol), salicylaldehyde (1 mmol) and oleic acid (40 μ L) in water (3 mL)

7.991
7.737
7.713
7.637
7.611
7.399
7.377
7.367
7.351
7.340
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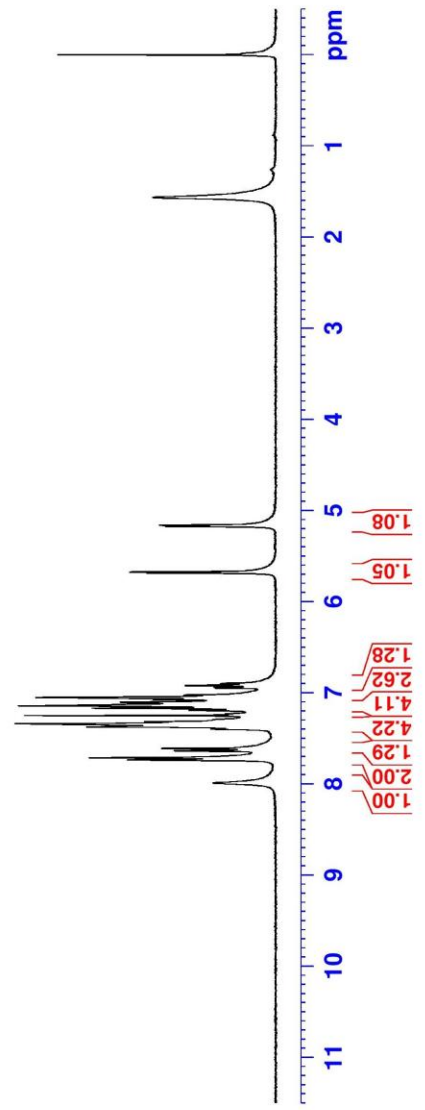


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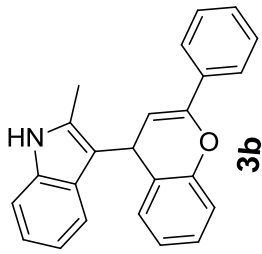
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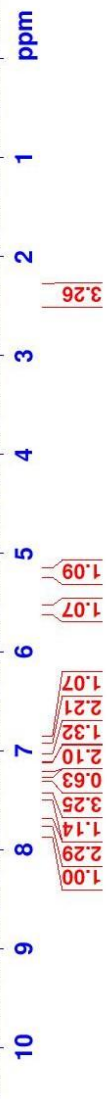


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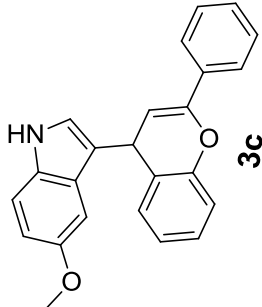
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 NS 16
 DS 2
 SWH 6188.119 Hz
 FIDRES 0.094423 Hz
 AQ 5.2953587 sec
 RG 287
 DW 80.800 usec
 DE 6.00 usec
 TE 300.0 K
 D1 1.00000000 sec
 TD0 1

===== CHANNEL f1 =====
 NUC1 1H
 P1 8.60 usec
 PL1 -2.00 dB
 SF01 300.1318534 MHz

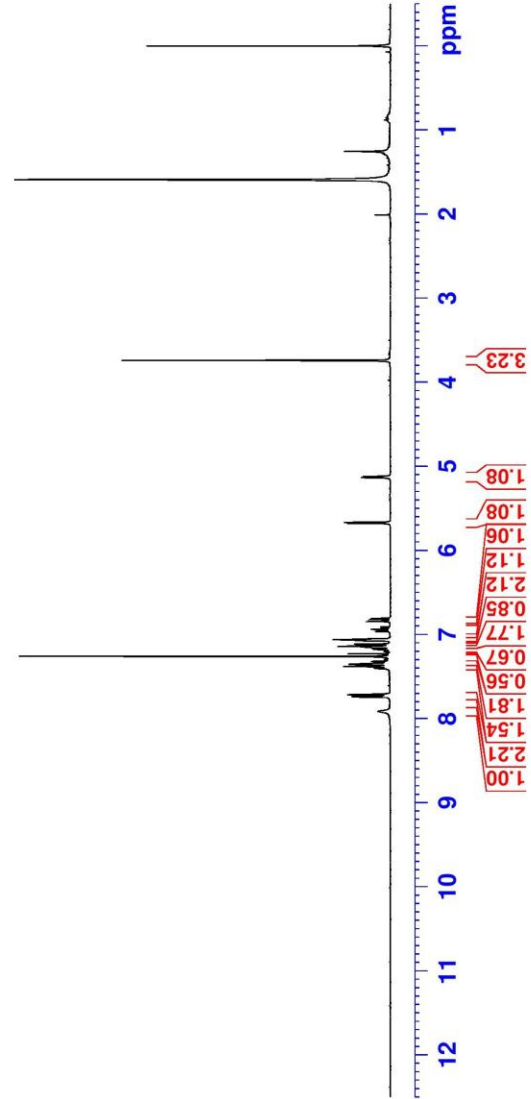
F2 - Processing parameters
 SI 32768
 SF 300.1300082 MHz
 WDW EM
 SSB 0
 LB 0.30 Hz
 GB 0
 PC 1.00



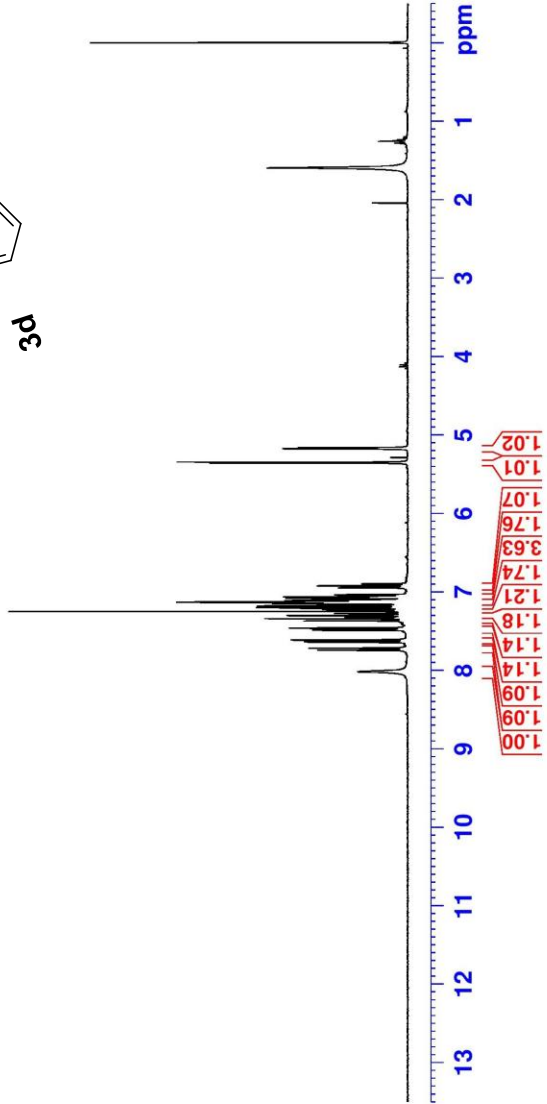
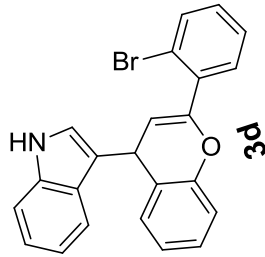
3.740
 5.121
 5.134
 5.667
 5.680
 6.809
 6.817
 6.838
 6.846
 6.912
 6.918
 6.938
 6.960
 6.966
 7.052
 7.061
 7.072
 7.116
 7.138
 7.144
 7.151
 7.155
 7.173
 7.178
 7.201
 7.205
 7.229
 7.298
 7.322
 7.332
 7.340
 7.345
 7.355
 7.380
 7.396
 7.402
 7.409
 7.712
 7.716
 7.738
 7.744
 7.916



Current Data Parameters
 NAME 1247f
 EXPNO 1
 PROCNO 1
 F2 - Acquisition Parameters
 Date_ 20141217
 Time 15.59
 INSTRUM spect
 PROBHD 5 mm BBO BB-1H
 PULPROG zg30
 TD 65536
 SOLVENT CDCl3
 NS 16
 DS 2
 SWH 6188.119 Hz
 FIDRES 0.094423 Hz
 AQ 5.2953587 sec
 RG 406
 DW 80.800 usec
 DE 6.00 usec
 TE 300.0 K
 D1 1.00000000 sec
 TD0 1
 ===== CHANNEL f1 =====
 NUC1 1H
 P1 8.60 usec
 PL1 -2.00 dB
 SFO1 300.1318534 MHz
 F2 - Processing parameters
 SI 32768
 SF 300.1300059 MHz
 WDW EM
 SSB 0
 LB 0.30 Hz
 GB 0
 PC 1.00



7.740
7.714
7.639
7.635
7.612
7.608
7.492
7.487
7.467
7.461
7.371
7.344
7.304
7.300
7.279
7.251
7.235
7.229
7.209
7.203
7.184
7.180
7.157
7.153
7.149
7.136
7.128
7.116
7.114
7.110
7.108
7.104
7.081
7.078
7.070
7.066
6.925
5.360
5.347
5.177
5.164
0.000



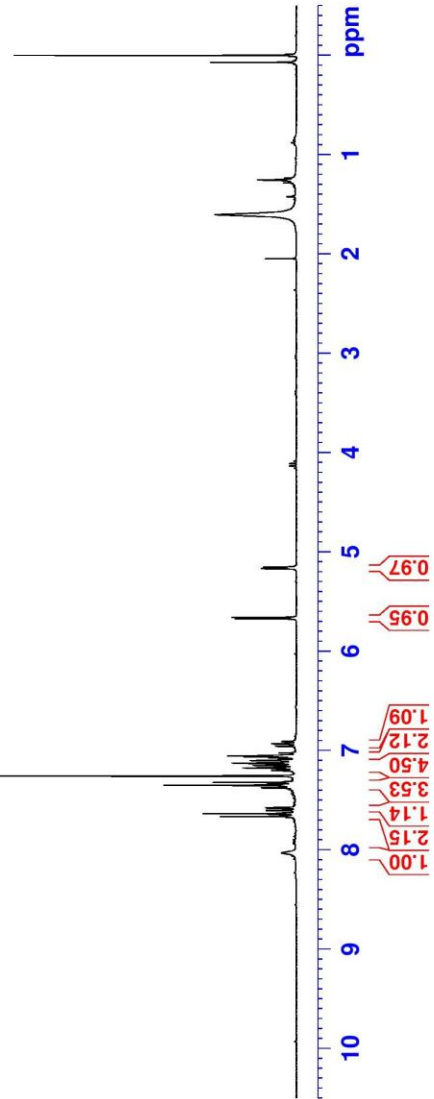
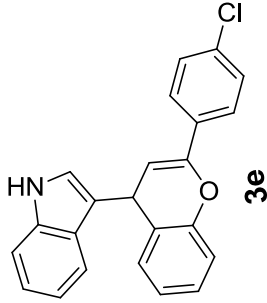
Current Data Parameters
 NAME 1200K
 EXPNO 1
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20141213
 Time 14.41
 INSTRUM spect
 PROBHD 5 mm BBO BB-1H
 PULPROG zg30
 TD 65536
 SOLVENT CDCl3
 NS 16
 DS 2
 SWH 6188.119 Hz
 FIDRES 0.094423 Hz
 AQ 5.2953587 sec
 RG 203
 DW 80.800 usec
 DE 6.00 usec
 TE 300.0 K
 D1 1.0000000 sec
 TD0 1

===== CHANNEL f1 =====
 NUC1 1H
 P1 8.60 usec
 PL1 -2.00 dB
 SFO1 300.1318534 MHz

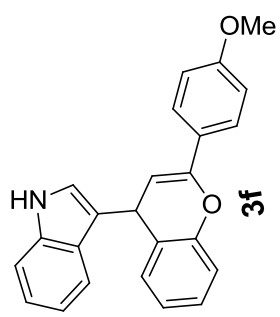
F2 - Processing parameters
 SI 32768
 SF 300.1300089 MHz
 WDW EM
 SSB 0
 LB 0.30 Hz
 GB 0
 PC 1.00

5.154
 5.168
 5.662
 5.675
 6.911
 6.929
 6.932
 6.934
 6.955
 6.960
 7.025
 7.028
 7.049
 7.052
 7.055
 7.063
 7.075
 7.078
 7.101
 7.104
 7.127
 7.132
 7.152
 7.156
 7.176
 7.179
 7.200
 7.203
 7.259
 7.323
 7.330
 7.352
 7.361
 7.376
 7.378
 7.578
 7.604
 7.637
 7.644
 7.660
 7.666
 8.033

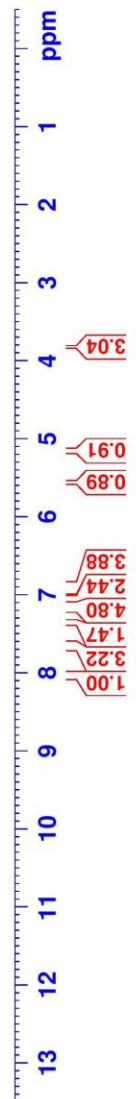


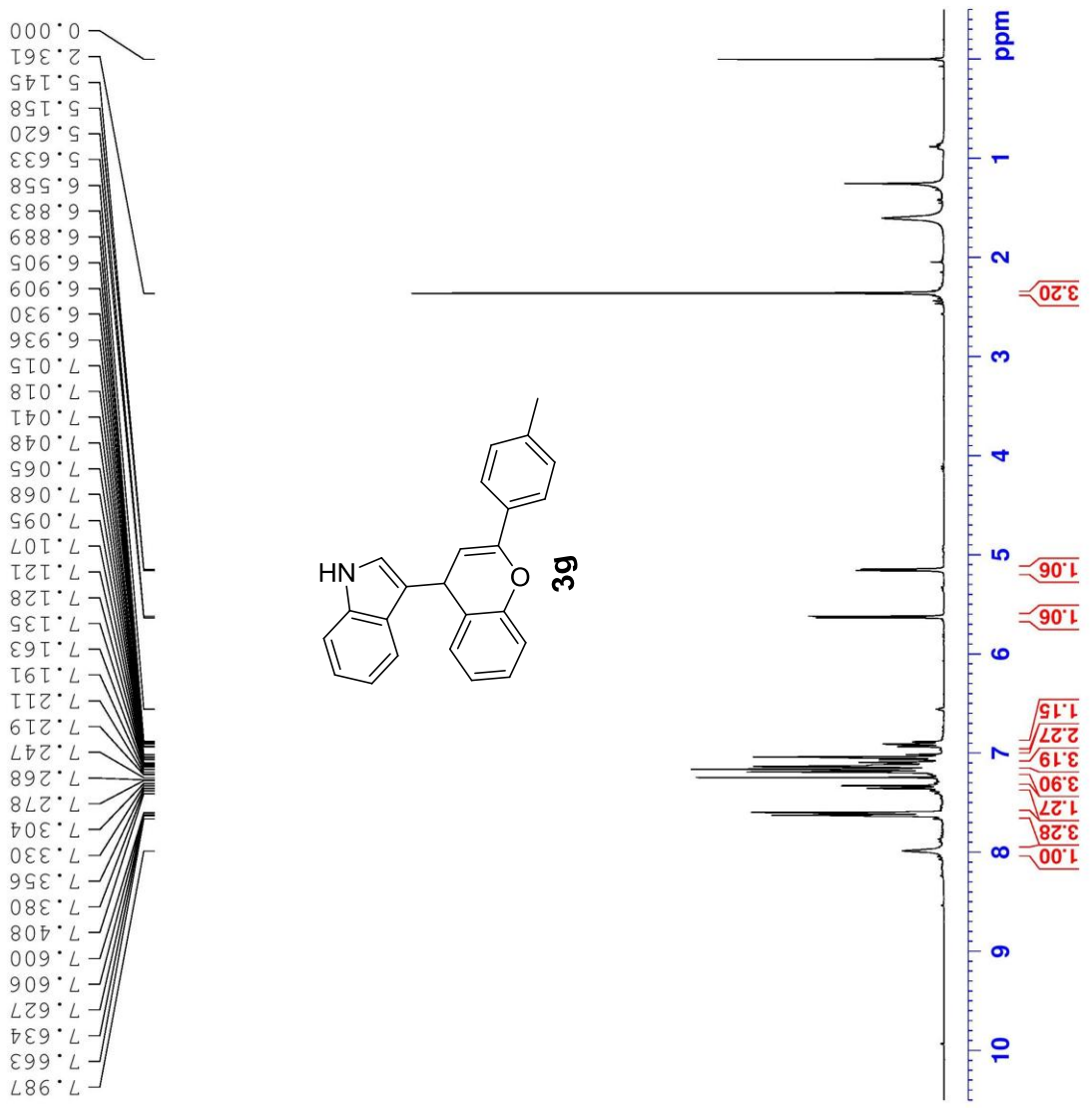
Current Data Parameters
 NAME 1200M
 EXPNO 1
 PROCNO 1
 F2 - Acquisition Parameters
 Date_ 20141215
 Time 14.15
 INSTRUM spect
 PROBHD 5 mm BBO BB-1H
 PULPROG zg30
 TD 65536
 SOLVENT CDCl3
 NS 16
 DS 2
 SWH 6188.119 Hz
 FIDRES 0.094423 Hz
 AQ 5.2953587 sec
 RG 362
 DW 80.800 usec
 DE 6.00 usec
 TE 300.0 K
 D1 1.0000000 sec
 TD0 1
 ===== CHANNEL f1 =====
 NUC1 1H
 P1 8.60 usec
 PL1 -2.00 dB
 SFO1 300.1318534 MHz
 F2 - Processing parameters
 SI 32768
 SF 300.1300067 MHz
 WDW EM
 SSB 0
 LB 0.30 Hz
 GB 0
 PC 1.00

8.023
 7.680
 7.670
 7.663
 7.647
 7.640
 7.609
 7.367
 7.340
 7.257
 7.210
 7.193
 7.189
 7.166
 7.142
 7.138
 7.129
 7.124
 7.099
 7.072
 7.069
 7.061
 7.053
 7.046
 7.022
 7.019
 6.951
 6.940
 6.934
 6.927
 6.917
 6.887
 6.878
 5.563
 5.550
 5.500
 5.156
 5.143
 3.827



Current Data Parameters
 NAME 1247C
 EXPNO 1
 PROCNO 1
 F2 - Acquisition Parameters
 Date_ 20141216
 Time 15.42
 INSTRUM spect
 PROBHD 5 mm BBO BB-1H
 PULPROG zg30
 TD 65536
 SOLVENT CDCl3
 NS 16
 DS 2
 SWH 6188.119 Hz
 FIDRES 0.094423 Hz
 AQ 5.2953587 sec
 RG 287
 DW 80.800 usec
 DE 6.00 usec
 TE 300.0 K
 D1 1.00000000 sec
 TD0 1
 ===== CHANNEL f1 =====
 NUC1 1H
 P1 8.60 usec
 PL1 -2.00 dB
 SFO1 300.1318534 MHz
 F2 - Processing parameters
 SI 32768
 SF 300.1300071 MHz
 WDW EM
 SSB 0
 LB 0.30 Hz
 GB 0
 PC 1.00



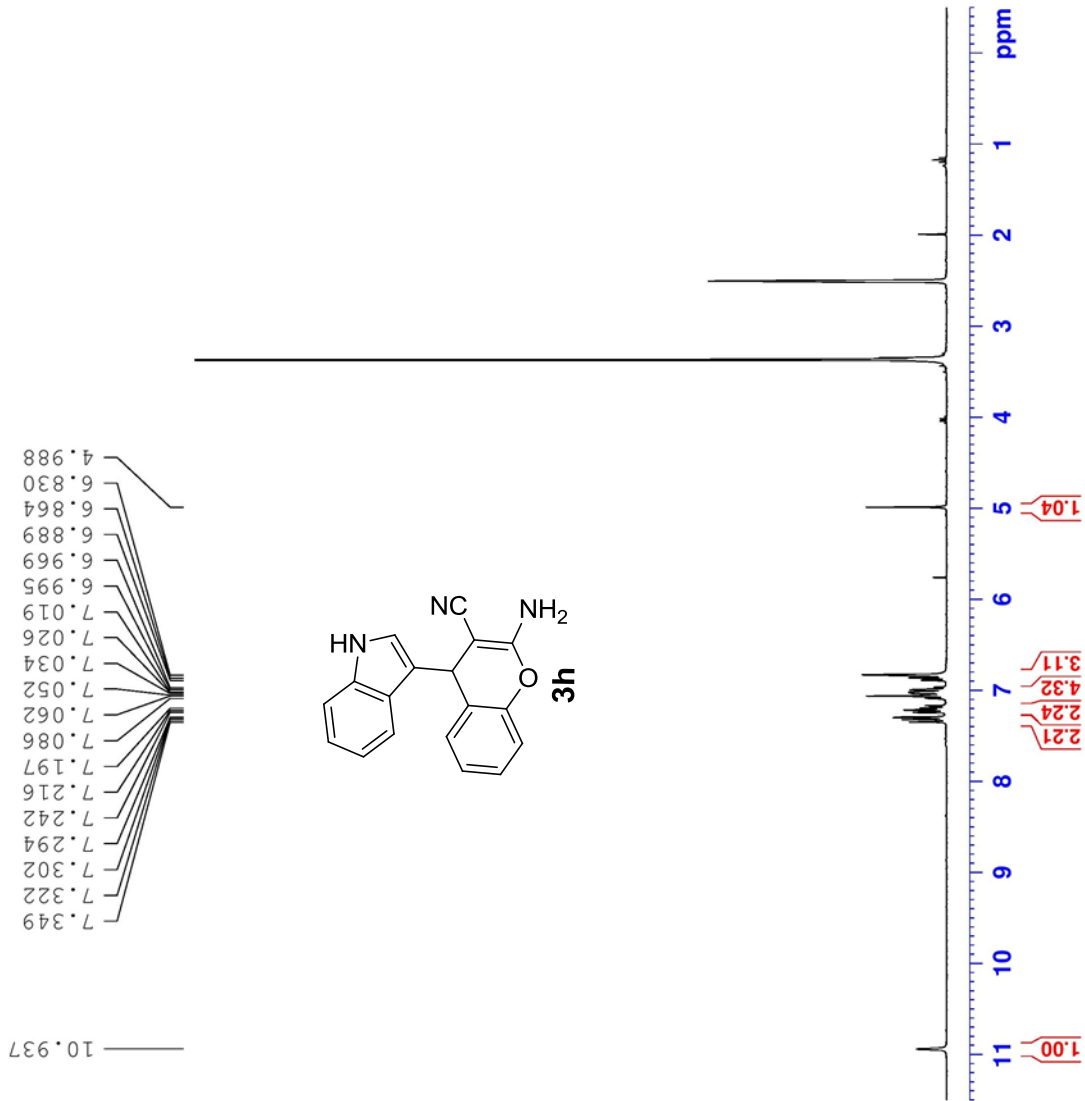


Current Data Parameters
 NAME 12001
 EXPNO 1
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20141206
 Time 15.16
 INSTRUM spect
 PROBHD 5 mm BBO BB-1H
 PULPROG zg30
 TD 65536
 SOLVENT CDCl3
 NS 16
 DS 2
 SWH 6188.119 Hz
 FIDRES 0.094423 Hz
 AQ 5.2953587 sec
 RG 256
 DW 80.800 usec
 DE 6.00 usec
 TE 300.0 K
 D1 1.0000000 sec
 TD0 1

===== CHANNEL f1 =====
 NUC1 1H
 P1 8.60 usec
 PL1 -2.00 dB
 SFO1 300.1318534 MHz

F2 - Processing parameters
 SI 32768
 SF 300.1300101 MHz
 WDW EM
 SSB 0
 LB 0.30 Hz
 GB 0
 PC 1.00

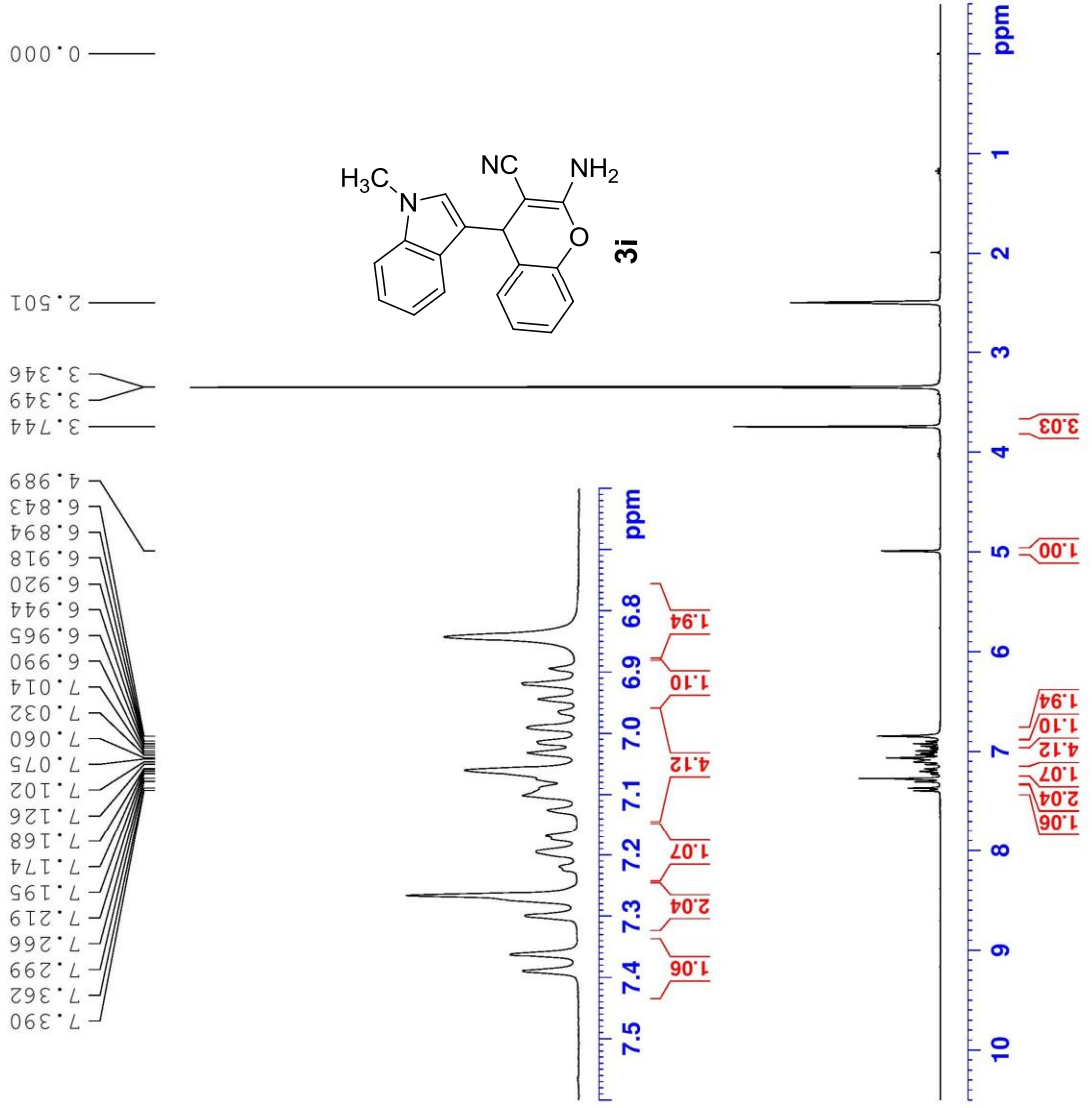


Current Data Parameters
 NAME 1200J
 EXPNO 1
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20141213
 Time 10.58
 INSTRUM spect
 PROBHD 5 mm BBO BB-1H
 PULPROG zg30
 TD 65536
 SOLVENT DMSO
 NS 16
 DS 2
 SWH 6188.119 Hz
 FIDRES 0.094423 Hz
 AQ 5.2953587 sec
 RG 228
 DW 80.800 usec
 DE 6.00 usec
 TE 300.0 K
 D1 1.0000000 sec
 TD0 1

===== CHANNEL f1 =====
 NUC1 1H
 P1 8.60 usec
 PL1 -2.00 dB
 SFO1 300.1318534 MHz

F2 - Processing parameters
 SI 32768
 SF 300.1300000 MHz
 WDW EM
 SSB 0
 LB 0.30 Hz
 GB 0
 PC 1.00

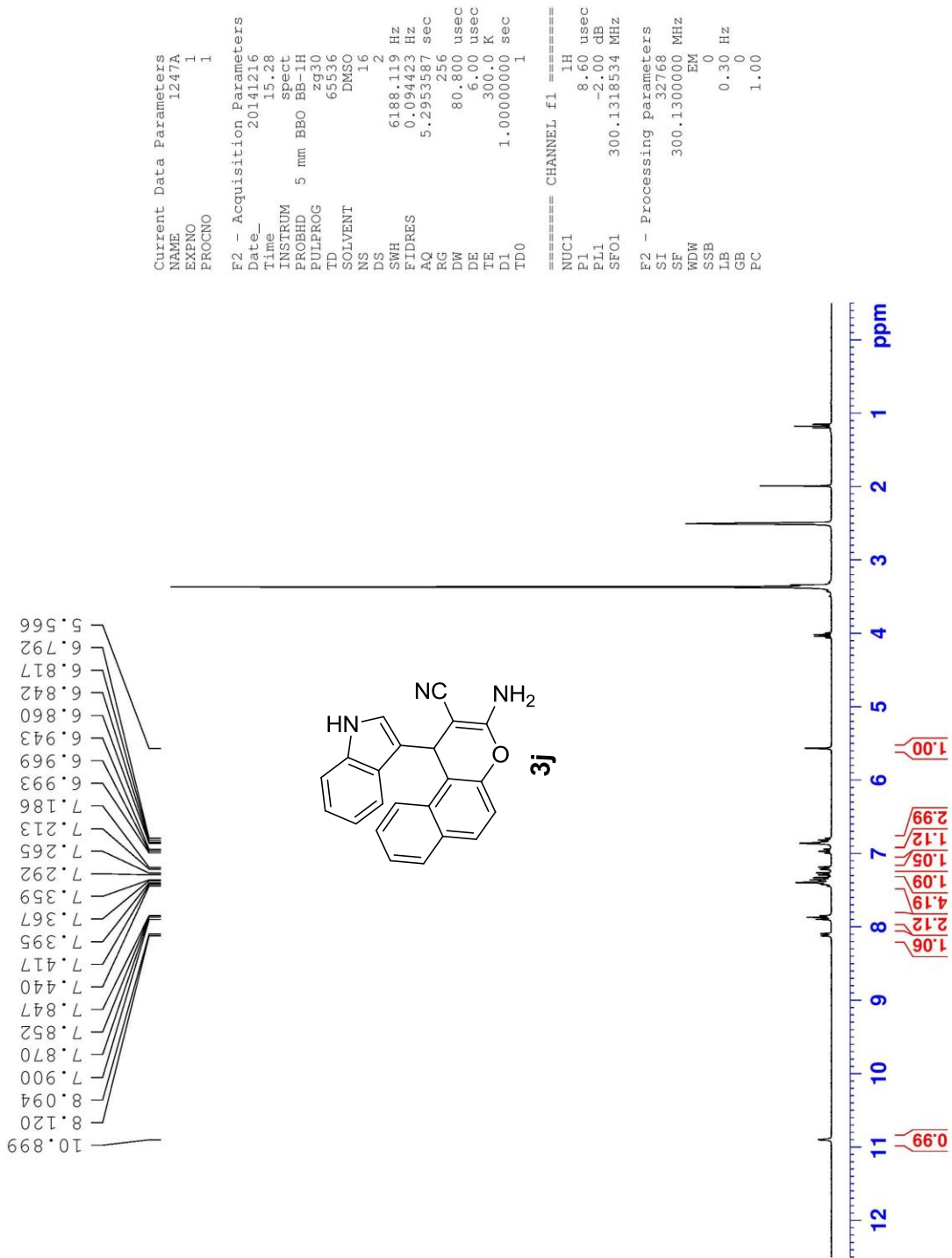


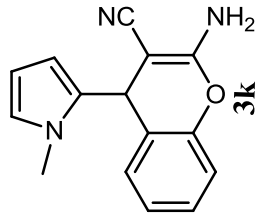
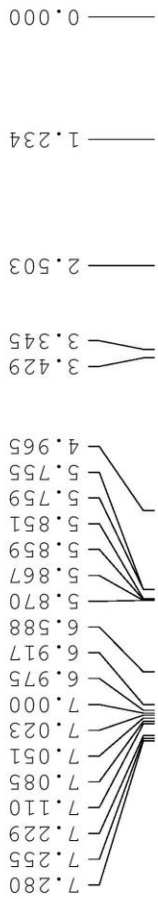
Current Data Parameters
 NAME 0805L
 EXPNO 1
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20150703
 Time 16.24
 INSTRUM spect
 PROBHD 5 mm BBO BB-1H
 PULPROG zg30
 TD 65536
 SOLVENT DMSO
 NS 16
 DS 2
 SWH 6188.119 Hz
 FIDRES 0.094423 Hz
 AQ 5.2953587 sec
 RG 287
 DW 80.800 usec
 DE 6.00 usec
 TE 300.0 K
 D1 1.0000000 sec
 TD0 1

===== CHANNEL f1 =====
 NUC1 1H
 P1 8.60 usec
 PL1 -2.00 dB
 SFO1 300.1318534 MHz

F2 - Processing parameters
 SI 32768
 SF 300.1300008 MHz
 WDW EM
 SSB 0
 LB 0.30 Hz
 GB 0
 PC 1.00



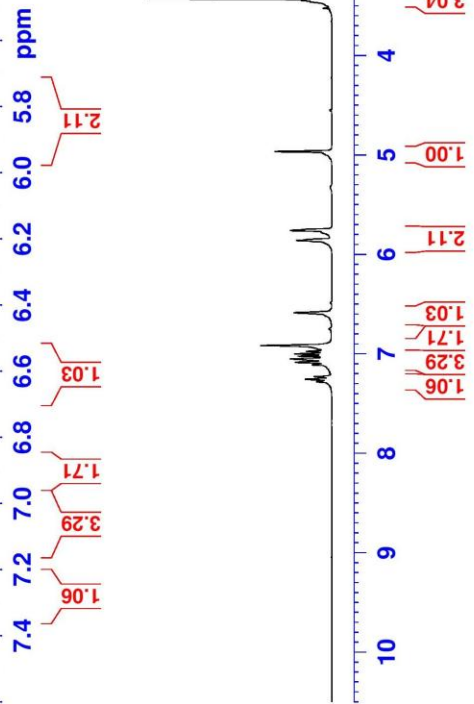


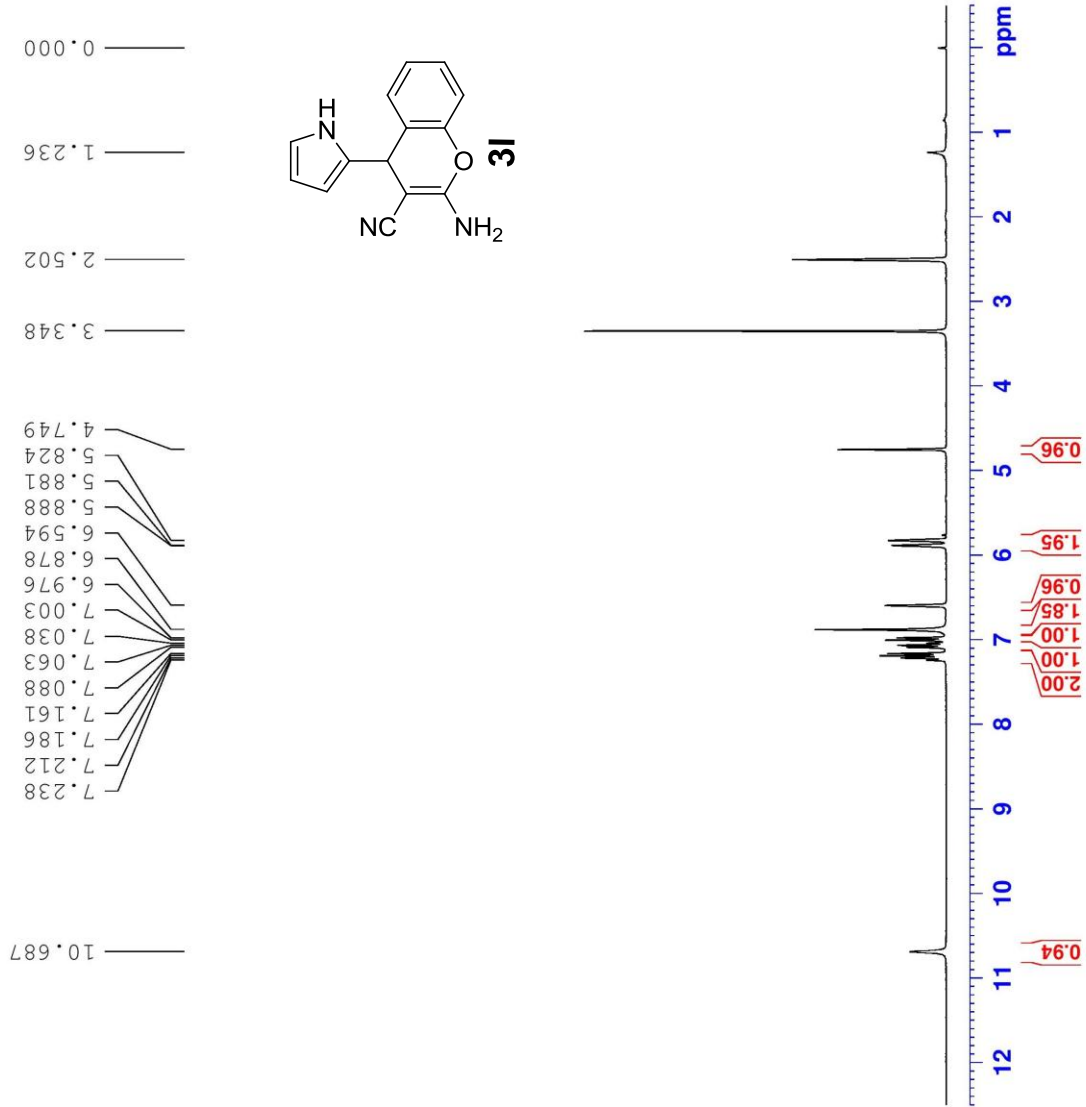
Current Data Parameters
 NAME 0369H-1
 EXPNO 1
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20150708
 Time 14.19
 INSTRUM spect
 PROBHD 5 mm BBO BB-1H
 PULPROG zg30
 TD 65536
 SOLVENT DMSO
 NS 16
 DS 2
 SWH 6188.119 Hz
 FIDRES 0.094423 Hz
 AQ 5.2953587 sec
 RG 228
 DW 80.800 usec
 DE 6.00 usec
 TE 300.0 K
 D1 1.00000000 sec
 TD0 1

===== CHANNEL f1 =====
 NUC1 1H
 P1 8.60 usec
 PL1 -2.00 dB
 SF01 300.1318534 MHz

F2 - Processing parameters
 SI 32768
 SF 300.1299999 MHz
 WDW EM
 SSB 0
 LB 0.30 Hz
 GB 0
 PC 1.00





```

Current Data Parameters
NAME      0369H-2
EXPNO    1
PROCNO   1

F2 - Acquisition Parameters
Date_    20150714
Time     14.55
INSTRUM  spect
PROBHD   5 mm BBO BB-1H
PULPROG  zg30
TD       65536
SOLVENT  DMSO
NS       16
DS       2
SWH      6188.119 Hz
FIDRES   0.094423 Hz
AQ       5.2953587 sec
RG       322
DE       80.800 usec
TE       300.0 K
D1       1.00000000 sec
TD0      1

===== CHANNEL f1 =====
NUC1     1H
P1       9.00 usec
PL1      -2.00 dB
SFO1     300.1318534 MHz

F2 - Processing parameters
SI       32768
SF       300.1300000 MHz
WDW      EM
SSB      0
LB       0.30 Hz
GB       0
PC       1.00
  
```

```

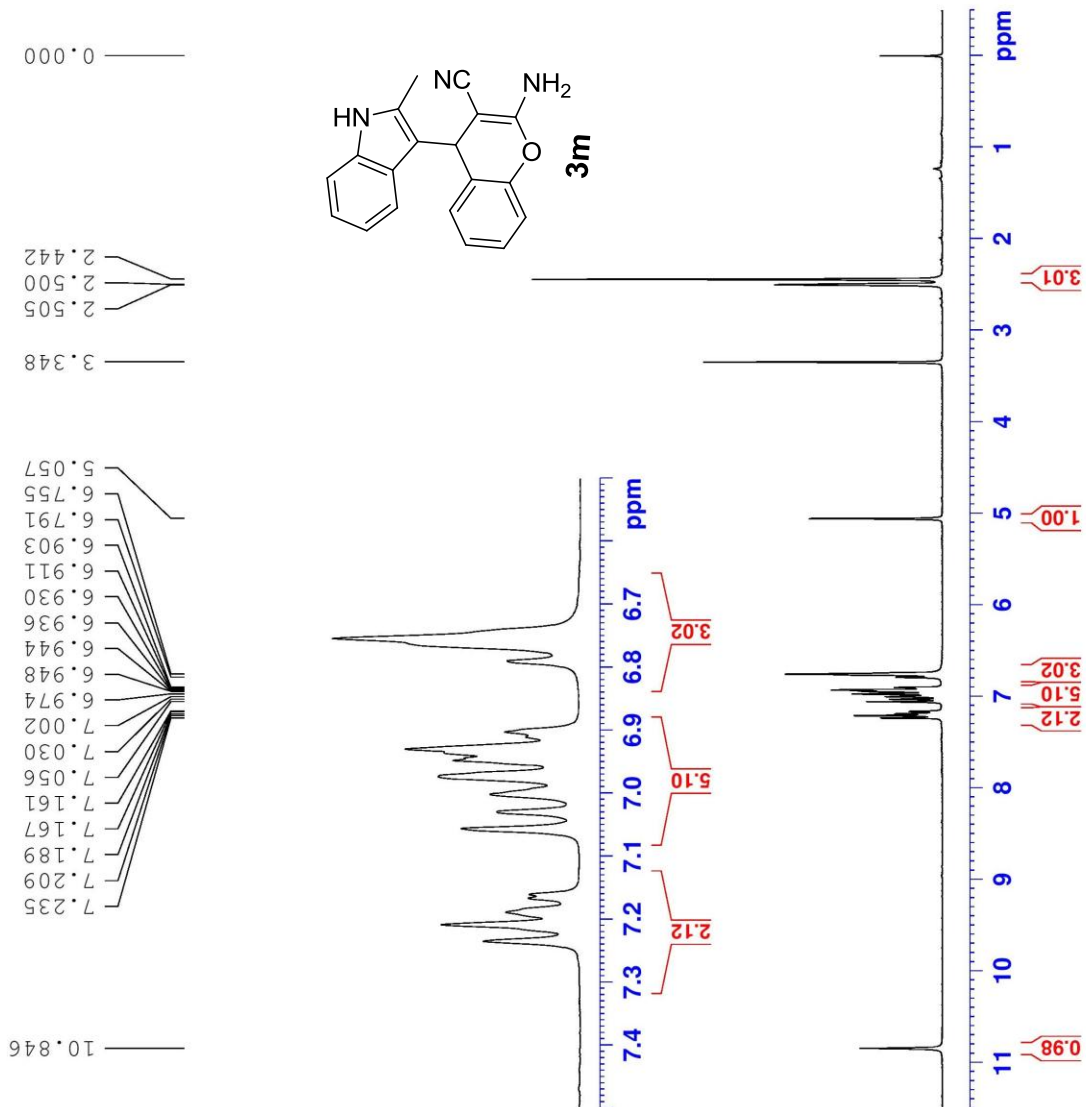
Current Data Parameters
NAME      0805Q
EXPNO    1
PROCNO   1

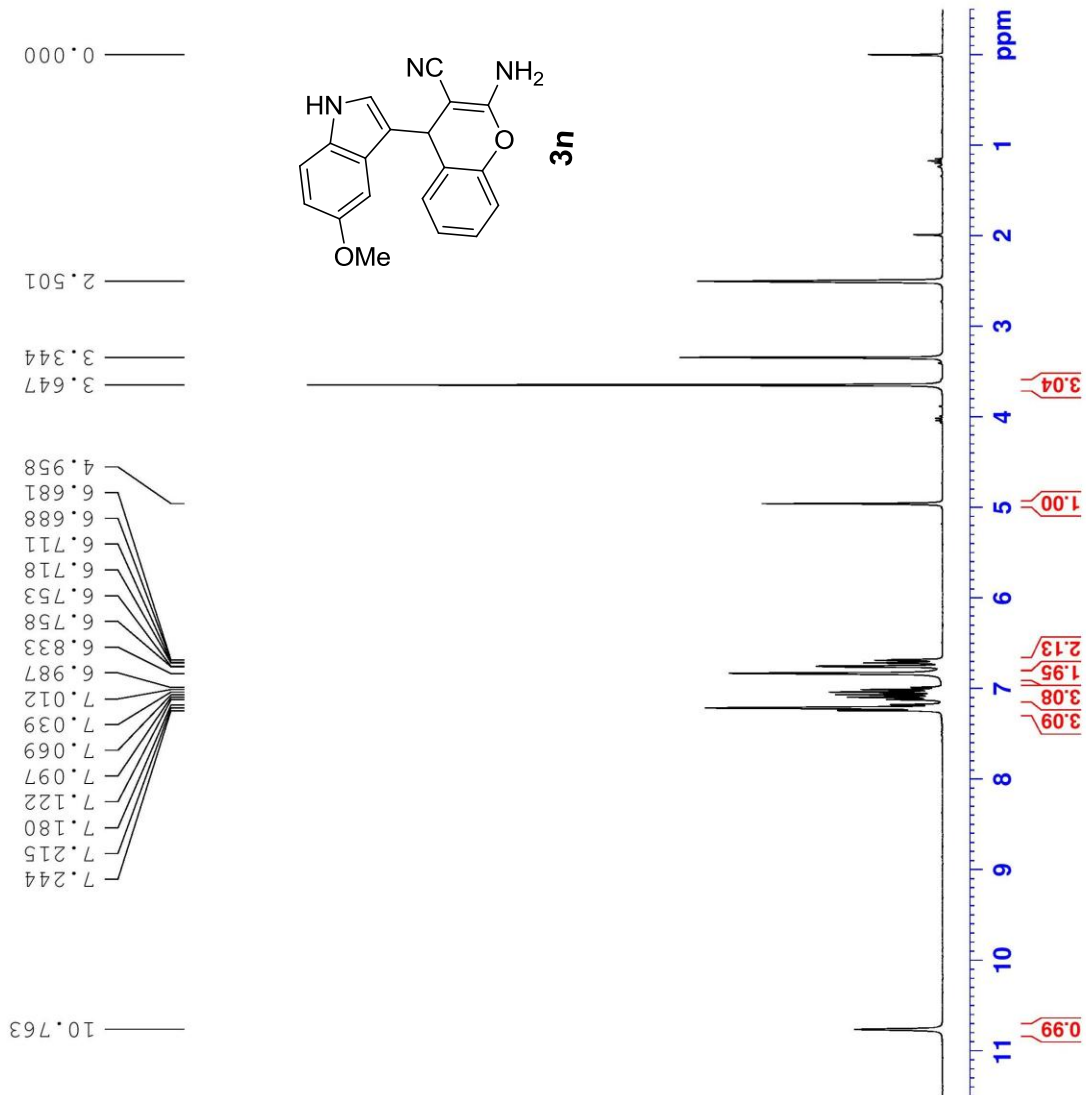
F2 - Acquisition Parameters
Date_    20150704
Time     10.48
INSTRUM  spect
PROBHD   5 mm BBO BB-1H
PULPROG  zg30
TD       65536
SOLVENT  DMSO
NS       16
DS       2
SWH      6188.119 Hz
FIDRES   0.094423 Hz
AQ       5.2953587 sec
RG       256
DW       80.800 usec
DE       6.00 usec
TE       300.0 K
D1       1.0000000 sec
TD0      1

===== CHANNEL f1 =====
NUC1     1H
P1       8.60 usec
PL1     -2.00 dB
SFO1    300.1318534 MHz

F2 - Processing parameters
SI       32768
SF       300.1300010 MHz
WDW      EM
SSB      0
LB       0.30 Hz
GB       0
PC       1.00

```



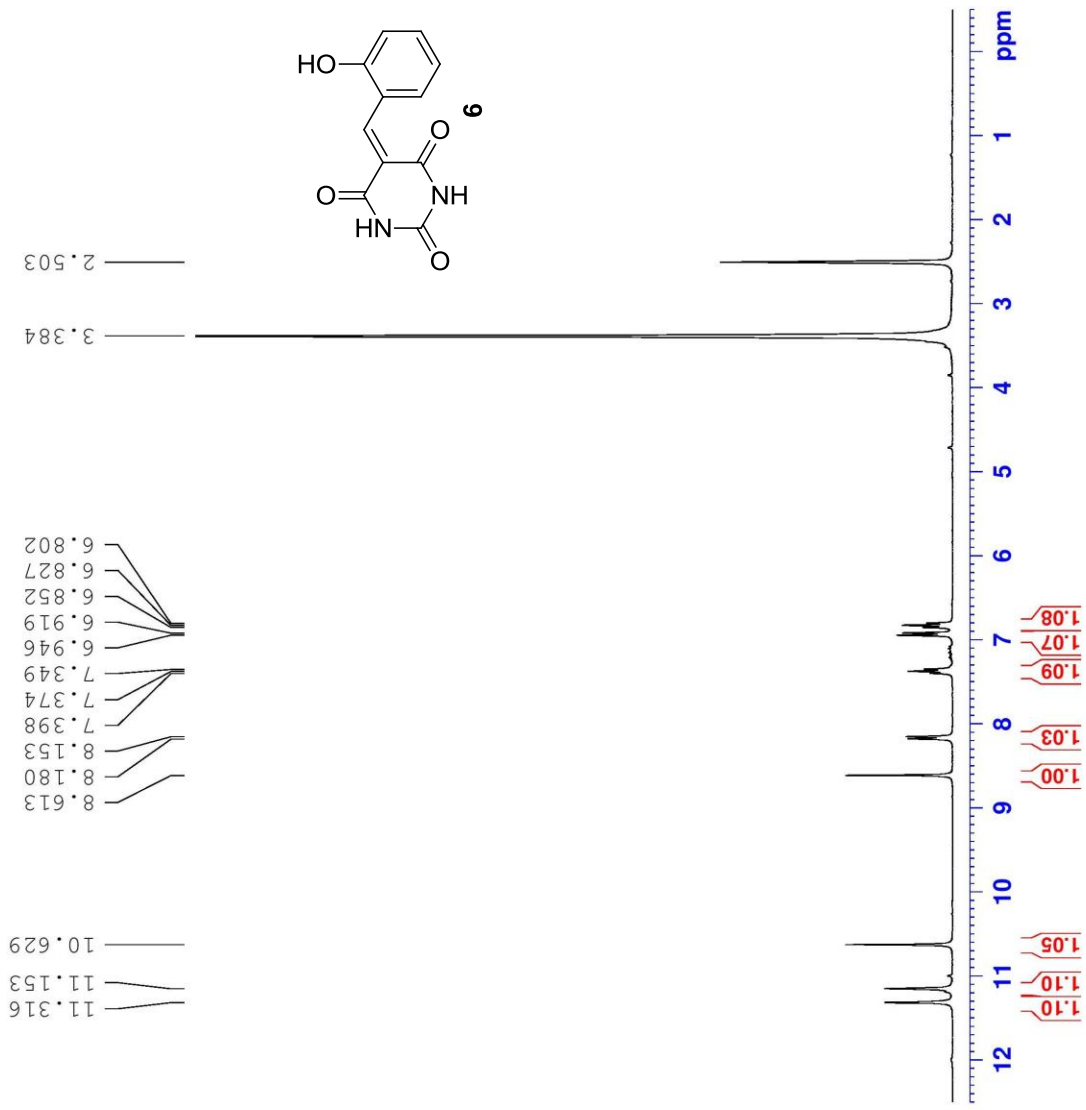


Current Data Parameters
 NAME 0805P
 EXPNO 1
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20150704
 Time 10.38
 INSTRUM spect
 PROBHD 5 mm BBO BB-1H
 PULPROG zg30
 TD 65536
 SOLVENT DMSO
 NS 16
 DS 2
 SWH 6188.119 Hz
 FIDRES 0.094423 Hz
 AQ 5.2953587 sec
 RG 256
 DW 80.800 usec
 DE 6.00 usec
 TE 300.0 K
 D1 1.0000000 sec
 TD0 1

===== CHANNEL f1 =====
 NUC1 1H
 P1 8.60 usec
 PL1 -2.00 dB
 SFO1 300.1318534 MHz

F2 - Processing parameters
 SI 32768
 SF 300.1300004 MHz
 WDW EM
 SSB 0
 LB 0.30 Hz
 GB 0
 PC 1.00



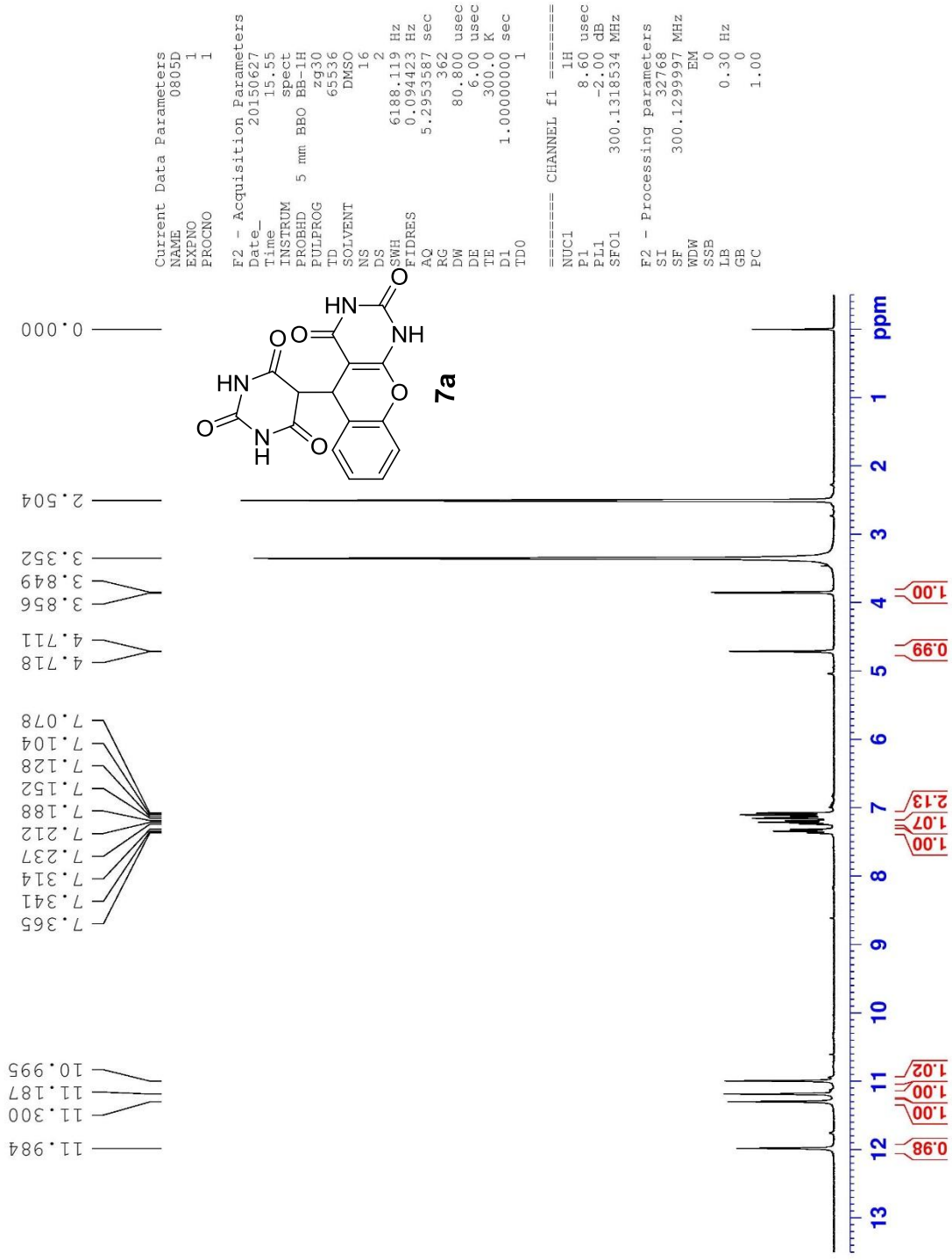
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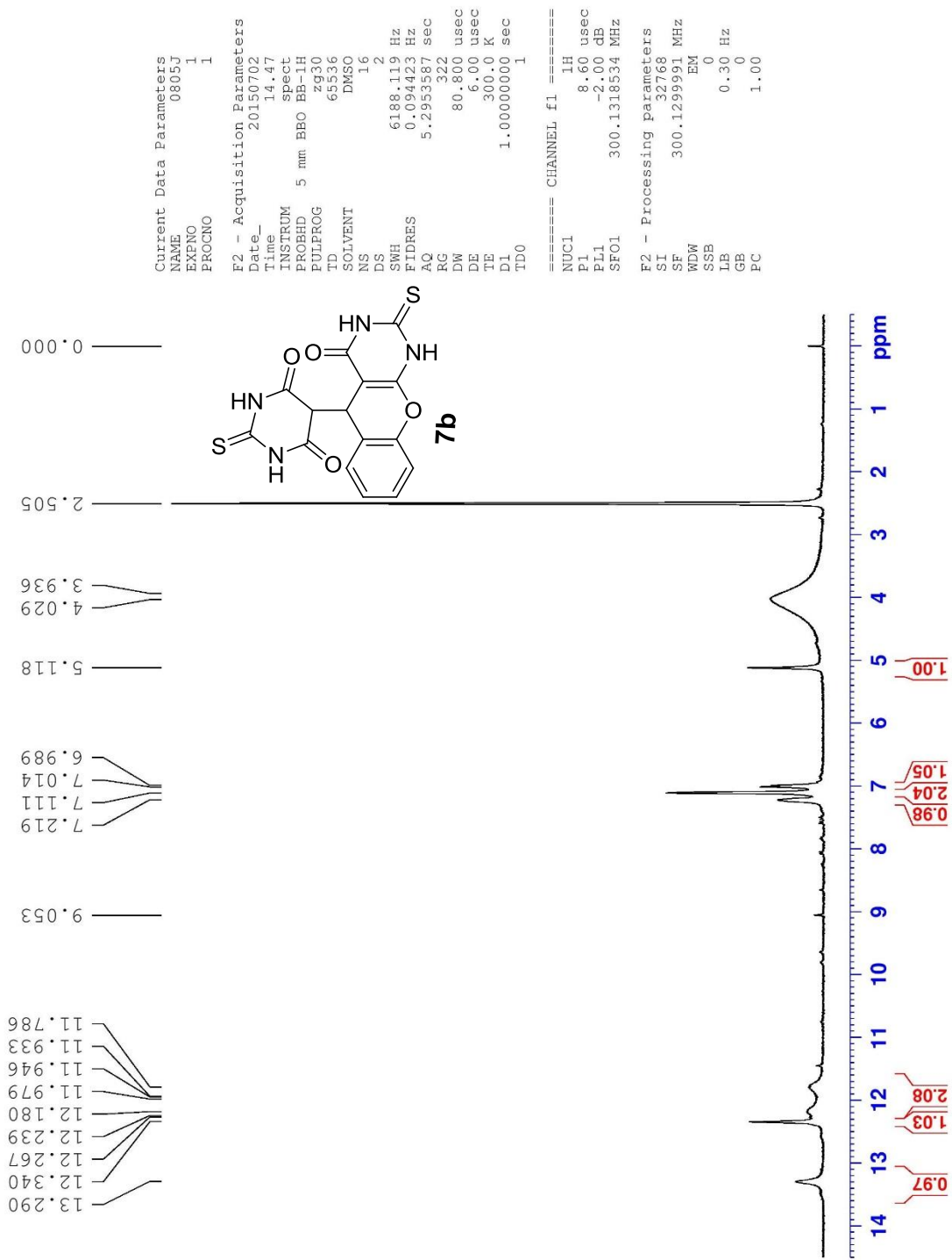
Current Data Parameters
NAME      1211D
EXPNO    1
PROCNO   1

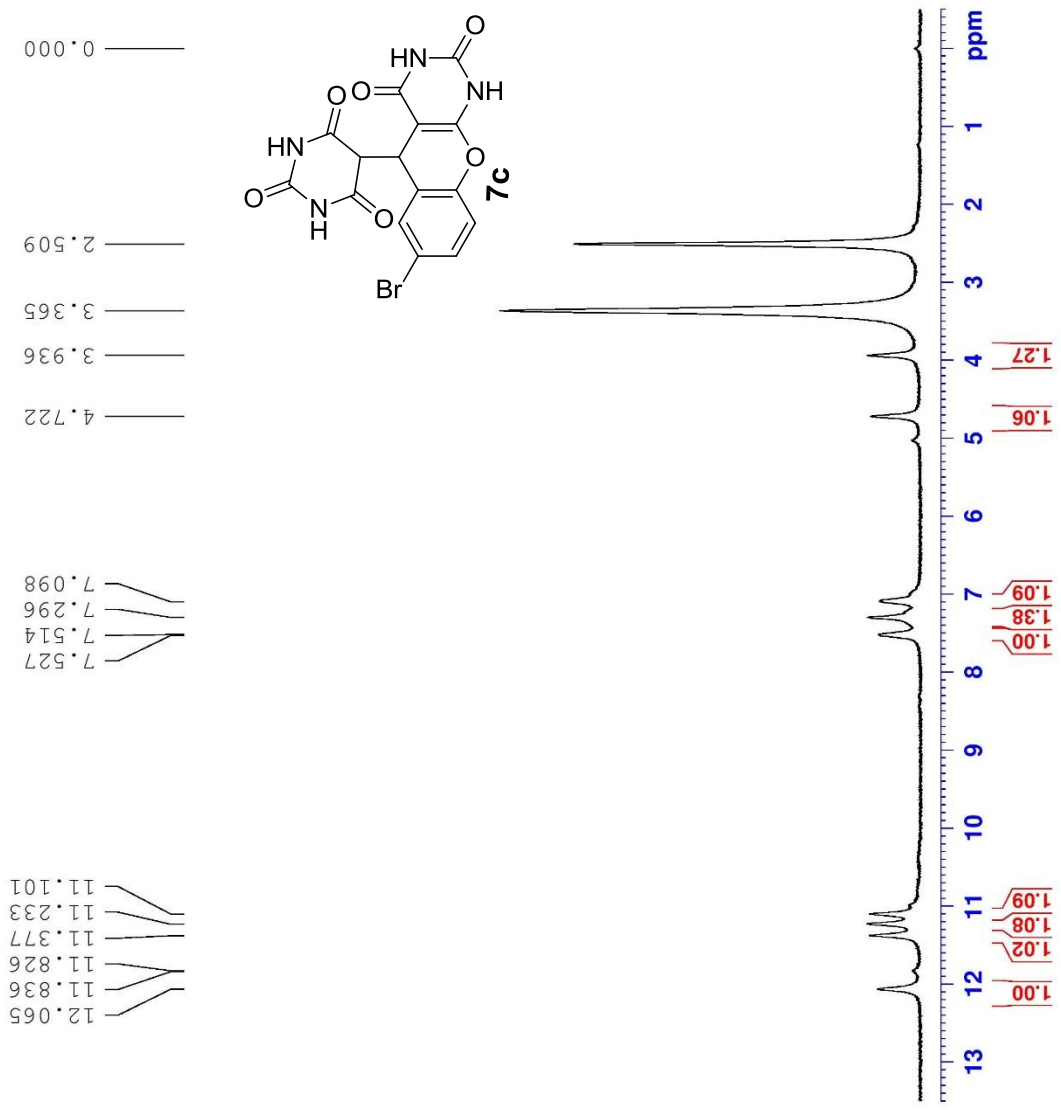
F2 - Acquisition Parameters
Date_    20151226
Time     15.03
INSTRUM  spect
PROBHD   5 mm BBO BB-1H
PULPROG  zg30
TD       65536
SOLVENT  DMSO
NS       16
DS       2
SWH      6188.119 Hz
FIDRES   0.094423 Hz
AQ       5.2953587 sec
RG       228
DW       80.800 usec
DE       6.00 usec
TE       300.0 K
D1       1.0000000 sec
TD0      1

===== CHANNEL f1 =====
NUC1     1H
P1       9.00 usec
PL1      -2.00 dB
SFO1     300.1318534 MHz

F2 - Processing parameters
SI       32768
SF       300.1300000 MHz
WDW      EM
SSB      0
LB       0.30 Hz
GB       0
PC       1.00
  
```



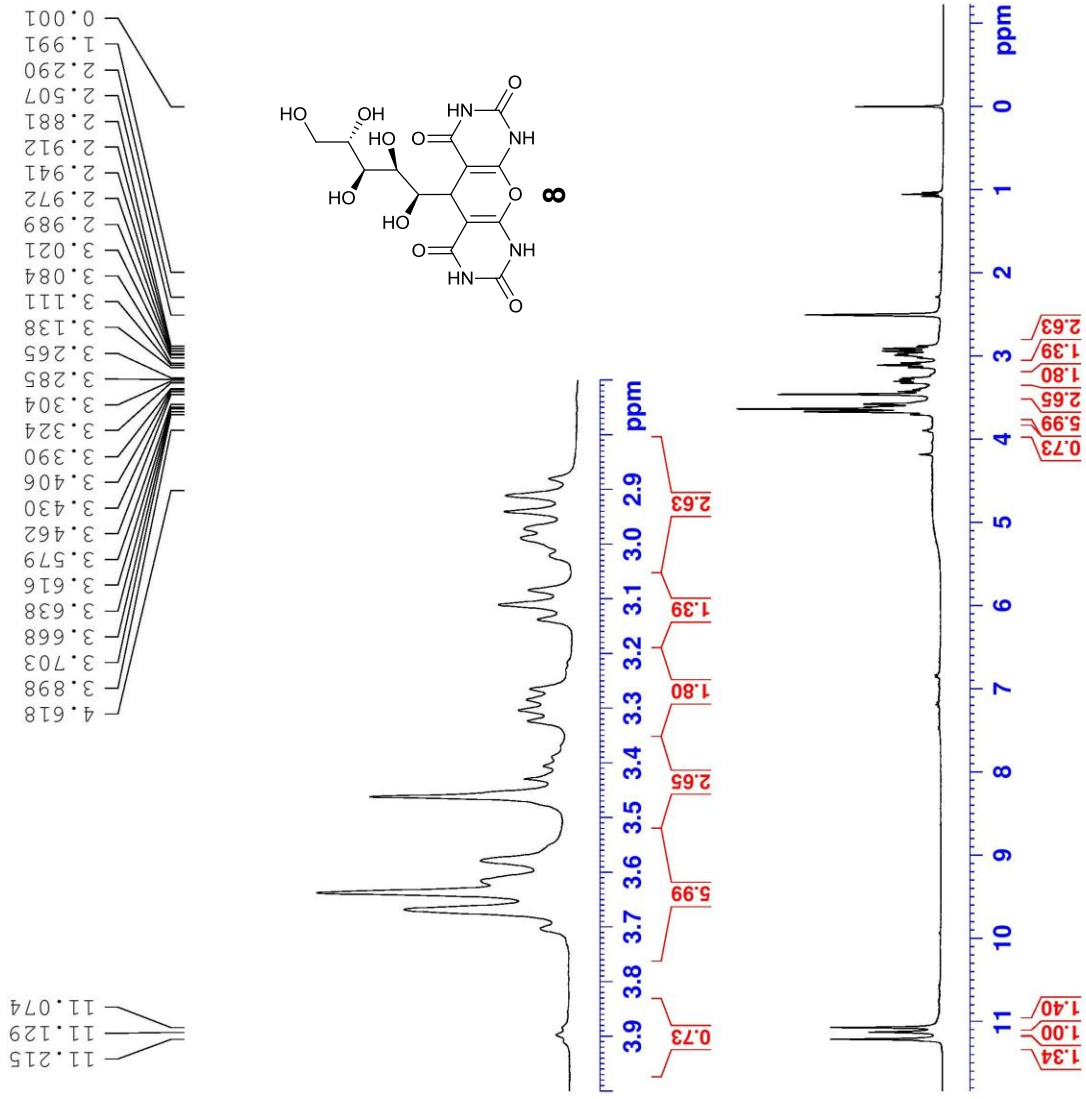


Current Data Parameters
 NAME 0805H
 EXPNO 1
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20150702
 Time 11.47
 INSTRUM spect
 PROBHD 5 mm BBO BB-1H
 PULPROG zg30
 TD 65536
 SOLVENT DMSO
 NS 16
 DS 2
 SWH 6188.119 Hz
 FIDRES 0.094423 Hz
 AQ 5.2953587 sec
 RG 322
 DW 80.800 usec
 DE 6.00 usec
 TE 300.0 K
 D1 1.0000000 sec
 TD0 1

===== CHANNEL f1 =====
 NUC1 1H
 P1 8.60 usec
 PL1 -2.00 dB
 SFO1 300.1318534 MHz

F2 - Processing parameters
 SI 32768
 SF 300.1299973 MHz
 WDW EM
 SSB 0
 LB 0.30 Hz
 GB 0
 PC 1.00

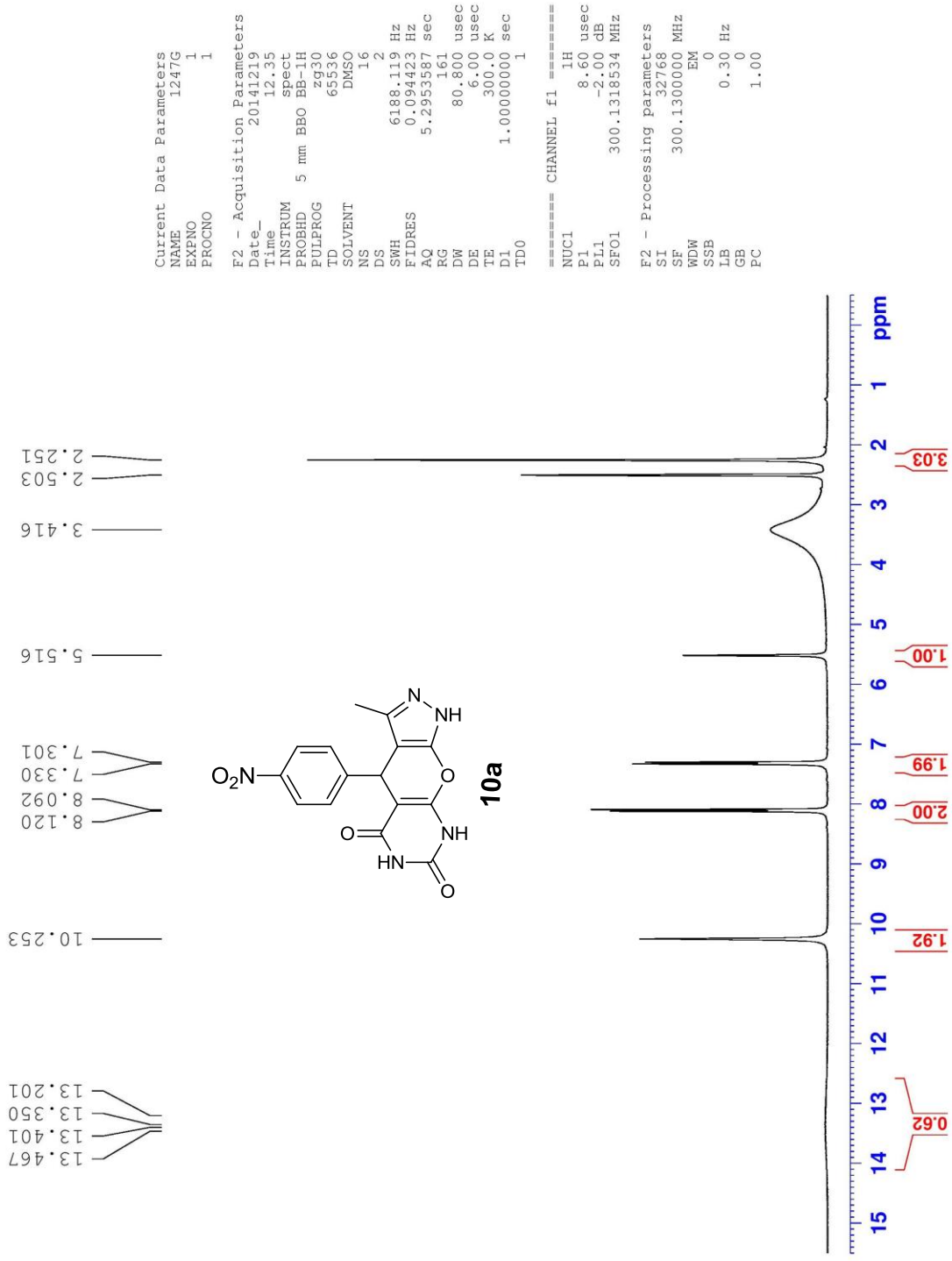


Current Data Parameters
 NAME 0170F
 EXPNO 1
 PROCNO 1

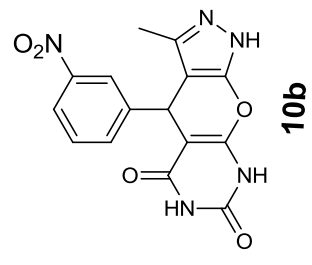
F2 - Acquisition Parameters
 Date_ 20150226
 Time 12.59
 INSTRUM spect
 PROBHD 5 mm BBO BB-1H
 PULPROG zg30
 TD 65536
 SOLVENT DMSO
 NS 16
 DS 2
 SWH 6188.119 Hz
 FIDRES 0.094423 Hz
 AQ 5.2953587 sec
 RG 181
 DW 80.800 usec
 DE 6.00 usec
 TE 300.0 K
 D1 1.0000000 sec
 TD0 1

===== CHANNEL f1 =====
 NUC1 1H
 P1 8.60 usec
 PL1 -2.00 dB
 SFO1 300.1318534 MHz

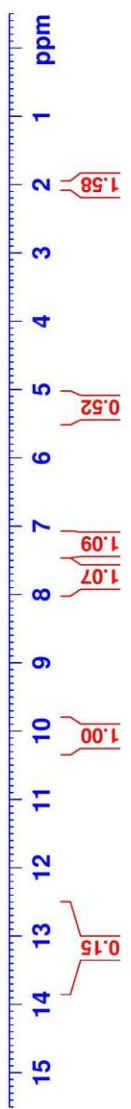
F2 - Processing parameters
 SI 32768
 SF 300.1299986 MHz
 WDW EM
 SSB 0
 LB 0.30 Hz
 GB 0
 PC 1.00

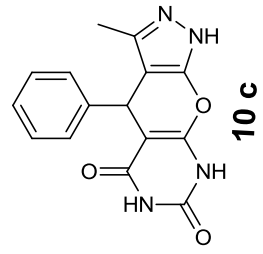
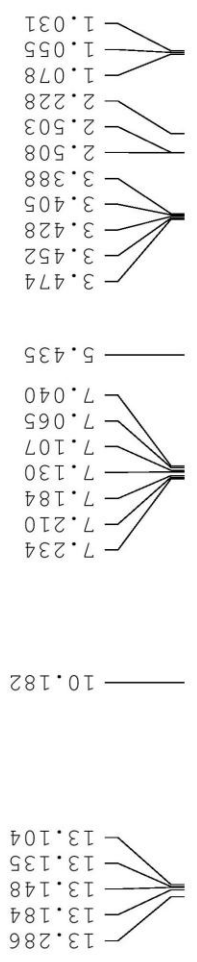


13.326
 13.283
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 13.155
 13.099
 13.071
 10.052
 8.719
 8.700
 8.507
 8.174
 8.148
 8.128
 8.104
 7.913
 7.798
 7.798
 7.790
 7.634
 7.517
 7.355
 7.310
 7.150
 7.080
 7.065
 7.035
 5.306
 3.162
 2.901
 2.281
 2.092
 2.039
 1.930
 1.864
 1.822
 1.010



Current Data Parameters
 NAME 12000
 EXPNO 1
 PROCNO 1
 F2 - Acquisition Parameters
 Date_ 20141216
 Time 15.20
 INSTRUM spect
 PROBHD 5 mm BBO BB-IH
 PULPROG zg30
 TD 65536
 SOLVENT DMSO
 NS 16
 DS 2
 SWH 6188.119 Hz
 FIDRES 0.094423 Hz
 AQ 5.2953587 sec
 RG 161
 DW 80.800 usec
 DE 6.00 usec
 TE 300.0 K
 D1 1.00000000 sec
 TD0 1
 ===== CHANNEL f1 =====
 NUC1 1H
 P1 8.60 usec
 PL1 -2.00 dB
 SF01 300.1318534 MHz
 F2 - Processing parameters
 SI 32768
 SF 300.1300664 MHz
 WDW EM
 SSB 0
 LB 0.30 Hz
 GB 0
 PC 1.00



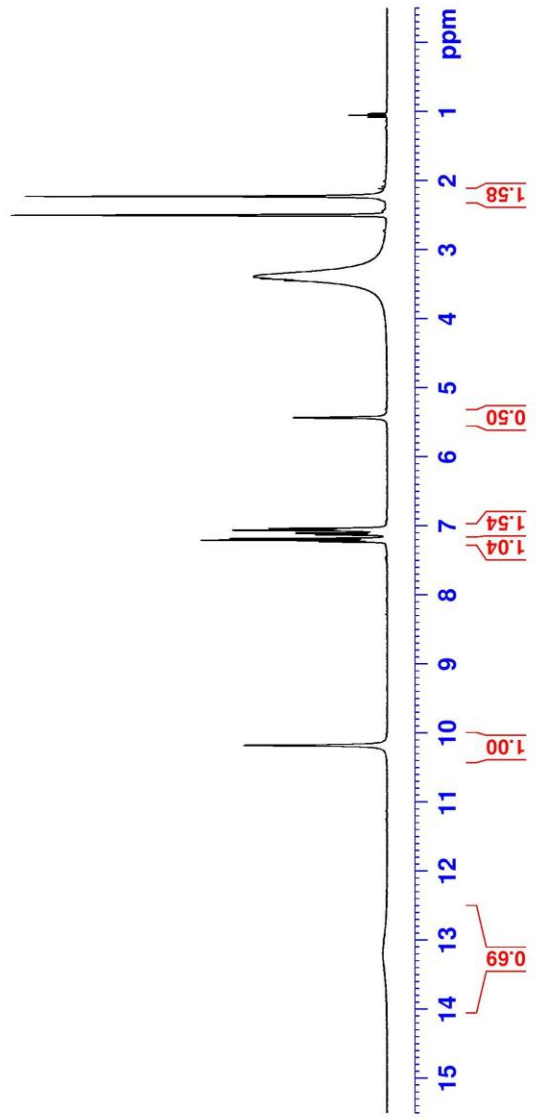


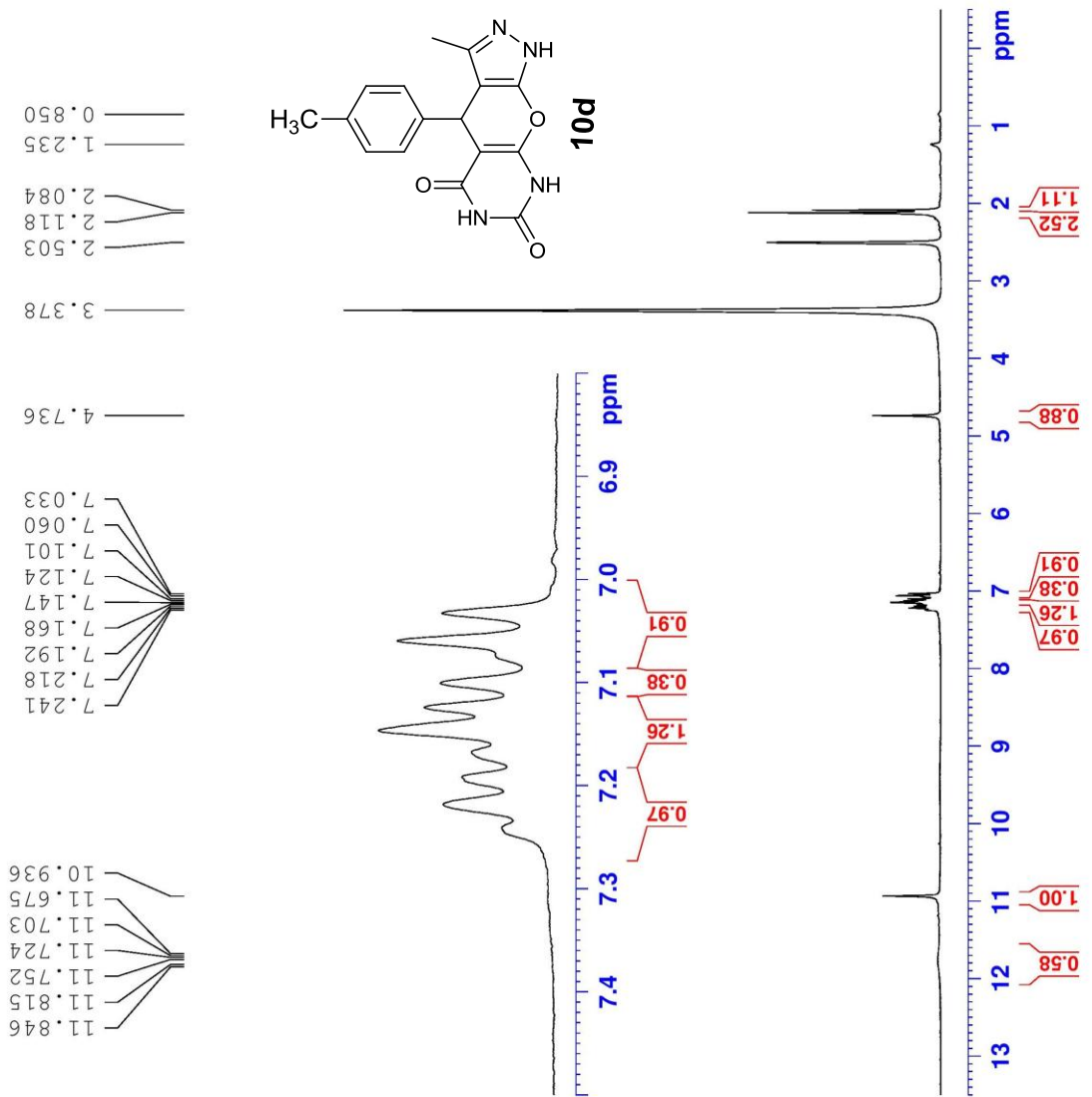
Current Data Parameters
 NAME 1200N
 EXPNO 1
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20141215
 Time 14.48
 INSTRUM spect
 PROHD 5 mm BBO BB-IH
 PULPROG zg30
 TD 65536
 SOLVENT DMSO
 NS 16
 DS 2
 SWH 6188.119 Hz
 FIDRES 0.094423 Hz
 AQ 5.2953587 sec
 RG 203
 DW 80.800 usec
 DE 6.00 usec
 TE 300.0 K
 D1 1.00000000 sec
 TD0 1

===== CHANNEL f1 =====
 NUC1 1H
 P1 8.60 usec
 PL1 -2.00 dB
 SF01 300.1318534 MHz

F2 - Processing parameters
 SI 32768
 SF 300.1300000 MHz
 WDW EM
 SSB 0
 LB 0.30 Hz
 GB 0
 PC 1.00



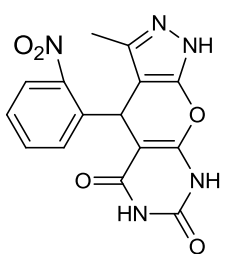
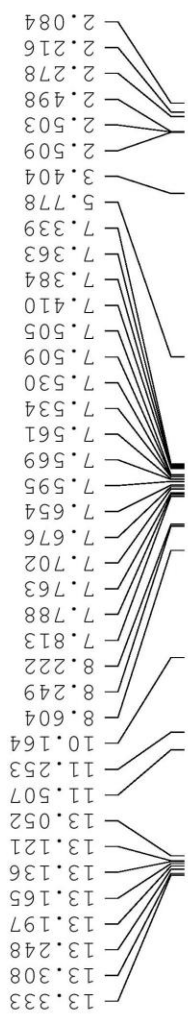


Current Data Parameters
 NAME 12471
 EXPNO 1
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20141222
 Time 14.18
 INSTRUM spect
 PROBHD 5 mm BBO BB-1H
 PULPROG zg30
 TD 65536
 SOLVENT DMSO
 NS 16
 DS 2
 SWH 6188.119 Hz
 FIDRES 0.094423 Hz
 AQ 5.2953587 sec
 RG 181
 DW 80.800 usec
 DE 6.00 usec
 TE 300.0 K
 D1 1.00000000 sec
 TD0 1

===== CHANNEL f1 =====
 NUC1 1H
 P1 8.60 usec
 PL1 -2.00 dB
 SF01 300.1318534 MHz

F2 - Processing parameters
 SI 32768
 WDW EM
 SSB 0
 LB 0
 GB 0
 PC 1.00

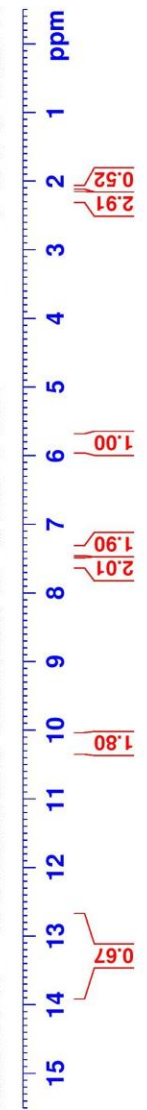


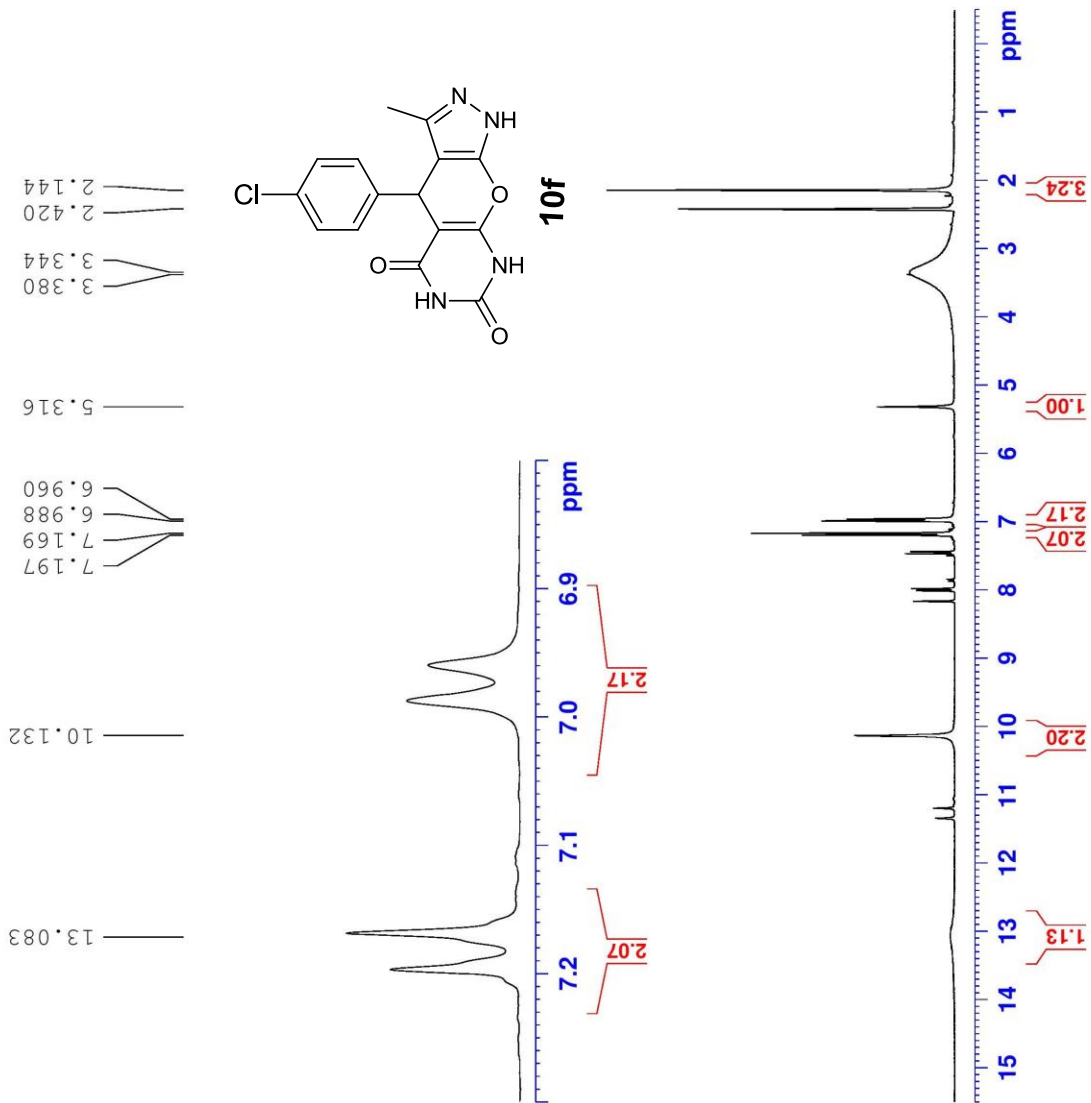
Current Data Parameters
 NAME 0170B
 EXPNO 1
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20150217
 Time 10.50
 INSTRUM spect
 PROBHD 5 mm BBO BB-1H
 PULPROG zg30
 TD 65536
 SOLVENT DMSO
 NS 16
 DS 2
 SWH 6188.119 Hz
 FIDRES 0.094423 Hz
 AQ 5.2953587 sec
 RG 203
 DW 80.800 usec
 DE 6.00 usec
 TE 300.0 K
 D1 1.00000000 sec
 TD0 1

===== CHANNEL f1 =====
 NUC1 1H
 P1 8.60 usec
 PL1 -2.00 dB
 SFO1 300.1318534 MHz

F2 - Processing parameters
 SI 32768
 SF 300.1300000 MHz
 WDW EM
 SSB 0
 LB 0.30 Hz
 GB 0
 PC 1.00



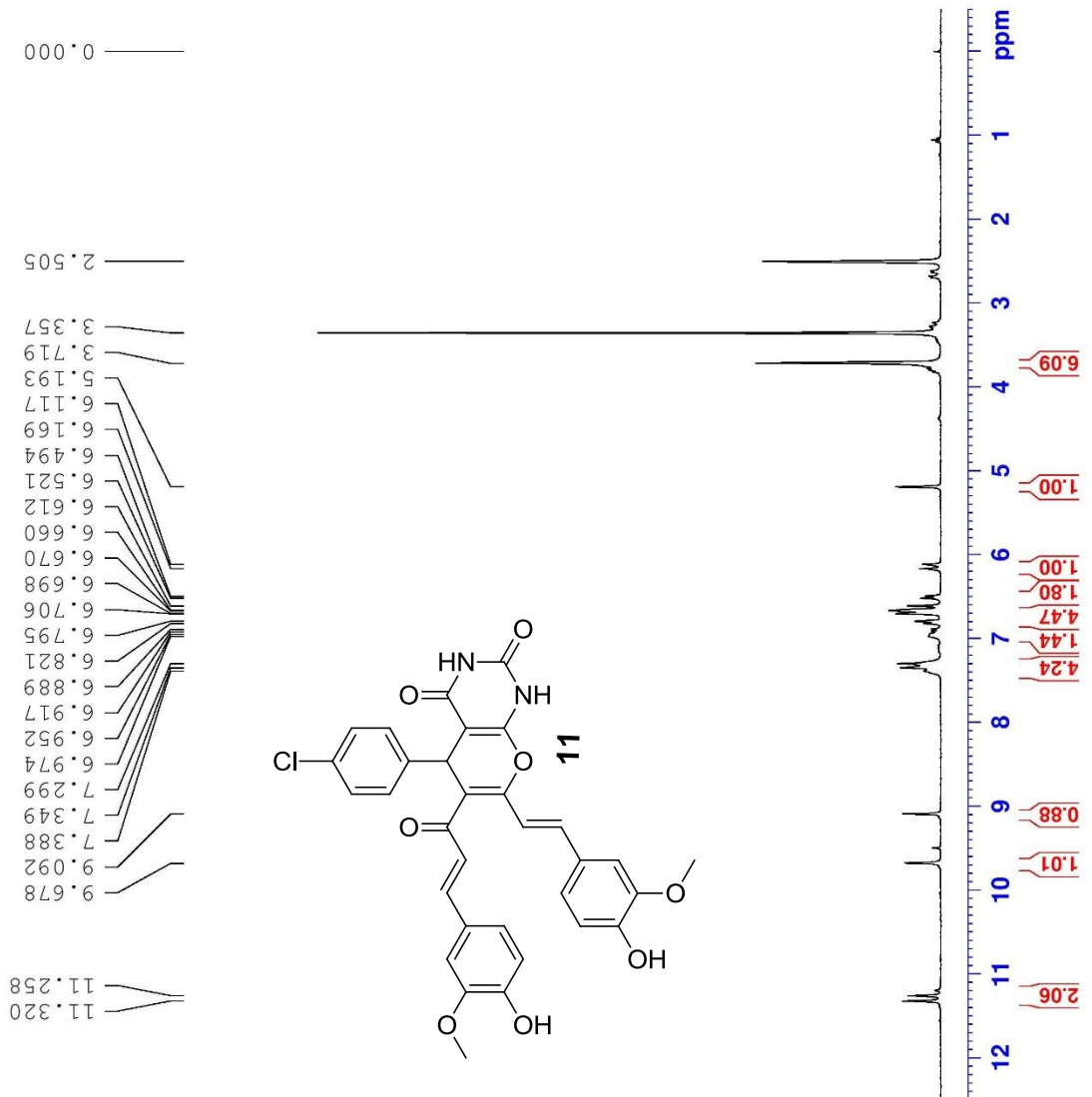


Current Data Parameters
 NAME 1247D
 EXPNO 1
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20141217
 Time 15.46
 INSTRUM spect
 PROBHD 5 mm BBO BB-1H
 PULPROG zg30
 TD 65536
 SOLVENT DMSO
 NS 16
 DS 2
 SWH 6188.119 Hz
 FIDRES 0.094423 Hz
 AQ 5.2953587 sec
 RG 181
 DE 80.800 usec
 TE 300.0 K
 D1 1.0000000 sec
 TD0 1

===== CHANNEL f1 =====
 NUC1 1H
 P1 8.60 usec
 PL1 -2.00 dB
 SFO1 300.1318534 MHz

F2 - Processing parameters
 SI 32768
 SF 300.1300248 MHz
 WDW EM
 SSB 0
 LB 0.30 Hz
 GB 0
 PC 1.00



Current Data Parameters
NAME 0805E
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20150701
Time 14.52
INSTRUM spect
PROBHD 5 mm BBO BB-1H
PULPROG zg30
TD 65536
SOLVENT DMSO
NS 16
DS 2
SWH 6188.119 Hz
FIDRES 0.094423 Hz
AQ 5.2953587 sec
RG 256
DE 80.800 usec
TE 6.00 usec
D1 300.0 K
D1 1.00000000 sec
TD0 1

==== CHANNEL f1 =====
NUC1 1H
P1 8.60 usec
PL1 -2.00 dB
SF01 300.1318534 MHz

F2 - Processing parameters
SI 32768
SF 300.1299994 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00