Supporting Information

Discovery of a Potent Anti-tumor Agent through Regioselective Mono-*N*-acylation of 7*H*-Pyrrolo[3,2-*f*]quinazoline-1,3-diamine

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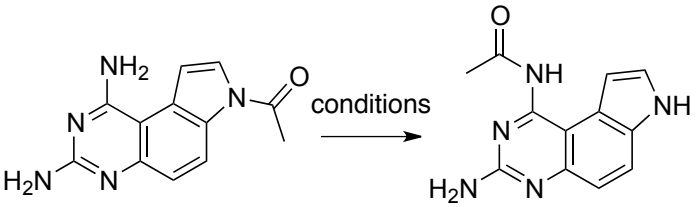
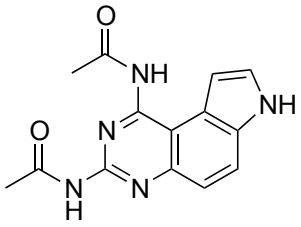
Table S2. The effect of bases on the yield of converting **2a** to **4a**.



2a **4a**

entry	base	temp. (°C)	yield (%)[a]
1	NaH	25	24
2	LDA	25	0
3	NaO^{t}Bu	25	25
4	LiHMDS	25	35[b]
5	LiHMDS	0	45
6	LiHMDS	-20	40

[a] Isolated yields. [b] Containing ca.10% of **4a'** as assessed by ^{1}H NMR.



4a'

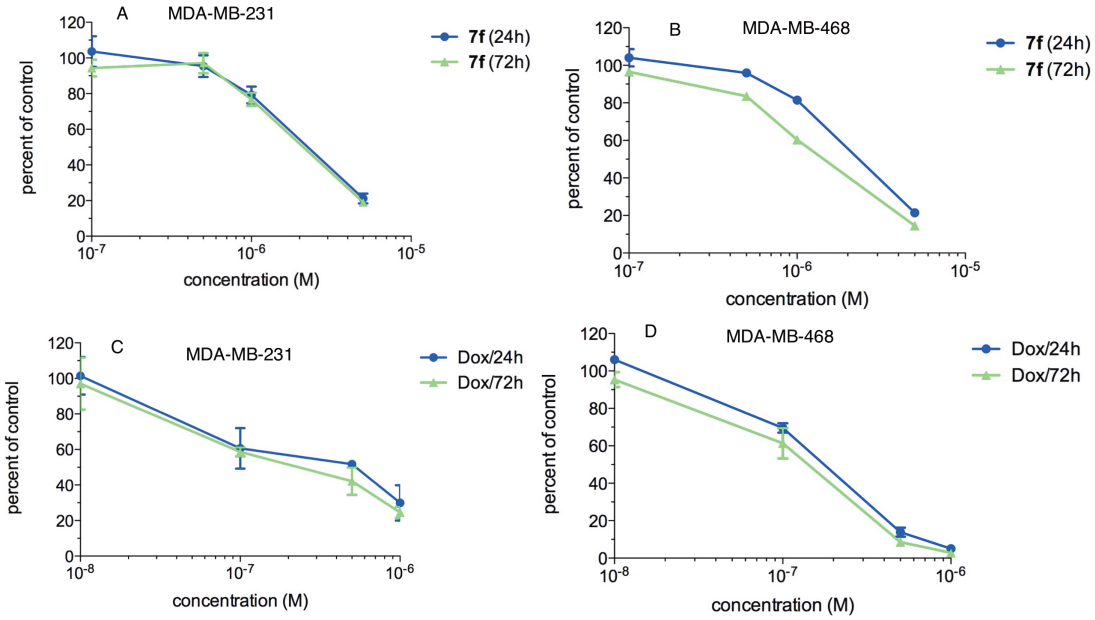


Figure S1. Compound **7f** and Dox inhibited TNBC cell growth after 24 h exposure. MDA-MB-231 (A and C) and MDA-MB-468 (B and D) cells were treated with different concentrations of **7f** (A and B) or Dox (C and D) for either 24 h or 72 h. For those cells treated with drugs for 24 h, the cells were further incubated in drug-free media for 48 h. At the end of the 72 h incubation period, the number of viable cells was quantified by an MTT assay. The data are presented as mean ± SD of a representative experiment performed in triplicates.

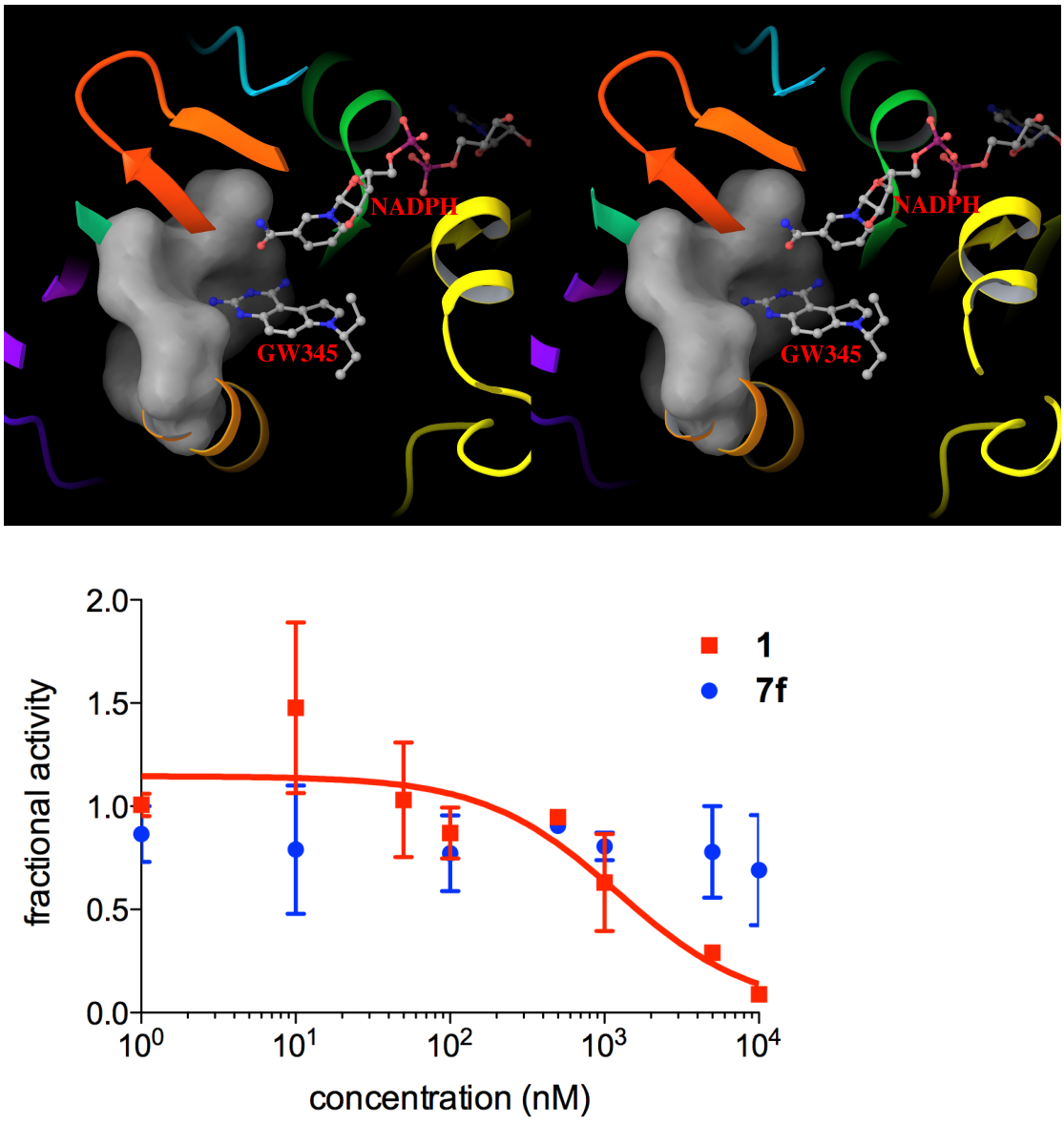


Figure S2. Compound **7f** did not inhibit human DHFR. Top: Crystal structure of GW345 bound to DHFR from *C. albicans* (PDB: 1AOE).[1] GW345 and NADPH are presented as ball-and-stick model. The molecular surface is presented for residues close to N^3 of GW345 to emphasize the sterics. The rest of the protein is shown as ribbons. The GW345 binding sites in DHFR from human and *C. albicans* are almost identical (see Figure 5 of reference[1]). The image is programed for wall-eyed viewing. Bottom: The human DHFR inhibition assay was performed with the DHFR inhibition kit from Sigma. See experimental section for details.

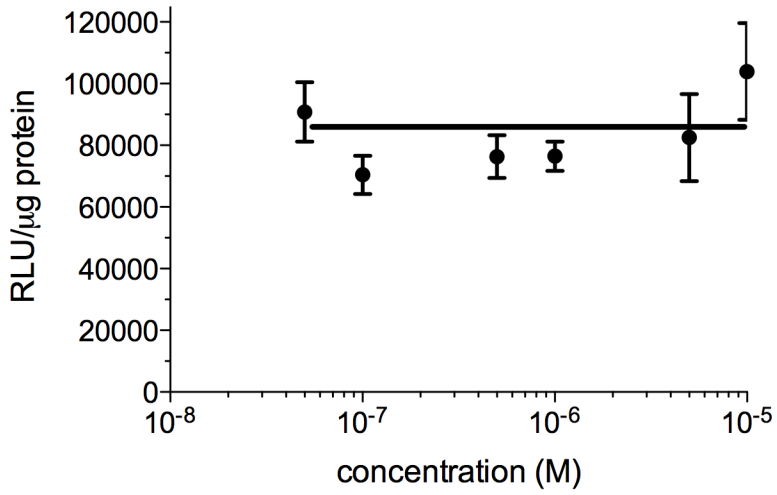
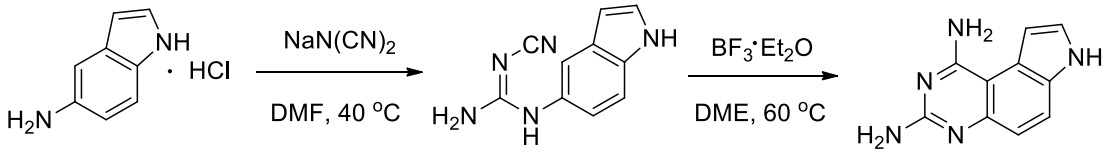


Figure S3. Compound **7f** did not inhibit CREB-mediated gene transcription in HEK 293T cells. HEK 293T cells were transfected with a CREB *renilla* luciferase reporter (CRE-RLuc). Then the cells were treated with increasing concentrations of **7f** for 30 min before the addition of forskolin at a final concentration of 10 μM. The cells were further incubated for 5 h before cell lysis and *renilla* luciferase activity measurement. The *renilla* luciferase activity was normalized to the protein concentration of the cell lysates and was expressed as relative luciferase unit (RLU)/μg of proteins.

Experimental Procedures

Synthesis of compound **1**[2]



8 **9** **1**

$NaN(CN)_2$ (7.3 g, 81.5 mmol) was added to a stirred solution of **8** (5.5 g, 32.6 mmol, prepared by treating a methanolic solution of 5-aminoindole with 1.5 equiv HCl in Et_2O) in DMF (55 mL). The reaction mixture was stirred at 40 °C for 4 h. DMF was removed and the residue was treated with H_2O (50 mL) for overnight. The gray solid was collected by filtration and dried in vacuum for 1 d to give compound **9** (6.3 g, 97% yield), which was used for the next step without further purification. The characterization data were consistent with literature reported values: ^{1}H NMR (400 MHz, DMSO-d_6) δ 11.12 (s, 1 H), 8.86 (s, 1 H), 7.46 (s, 1 H), 7.35-7.33 (m, 2 H), 6.94 (dd, J = 8.8 Hz, 2.0 Hz, 1 H), 6.74 (s, 2 H), 6.40 (s, 1 H). Boron trifluoride (18.8 mL, 152 mmol) was added dropwise to a stirred suspension of **9** (6.3 g, 31.6 mmol) in DME (600 mL) at 60 °C. The resulting mixture was stirred at 60 °C for 4 h. Then the solvent was removed and the residue was suspended in MeOH (60 mL) and treated with NH_4OH (40 mL) for 2 h. The solvents were removed in *vacuo* and the residue was purified by column chromatography on silica gel, eluting with 3:1 DCM:MeOH with 1% NH_4OH to give a yellow solid, which was treated with 1 N NaOH (50 mL) at room temperature for overnight. Then the solid was collected to give compound **1** as a white to pale yellow solid (5.2 g, 89%). The characterization data were consistent

with literature reported values: ^{1}H NMR (400 MHz, DMSO-d_6) δ 11.55 (s, 1 H), 7.64 (d, J = 8.8 Hz, 1 H), 7.43 (t, J = 2.8 Hz, 1 H), 7.03-7.00 (m, 2 H), 6.65 (brs, 2 H), 5.65 (s, 2 H); ^{13}C NMR (100 MHz, DMSO-d_6) δ 162.0, 159.1, 150.1, 130.4, 124.8, 120.0, 119.1, 119.0, 102.5, 102.0.

1-(1,3-Diamino-7H-pyrrolo[3,2-f]quinazolin-7-yl)propan-1-one (***2b***). The title compound was prepared as described for **2a** from 20.0 mg (0.10 mmol) of **1** and compound **2b** (20.0 mg, 78%) was obtained as a yellowish solid: mp 210-212 ºC; ^{1}H NMR (400 MHz, DMSO-d_6) δ 8.59 (d, J = 9.2 Hz, 1 H), 8.03 (d, J = 3.6 Hz, 1 H), 7.39 (d, J = 4.0 Hz, 1 H), 7.18 (d, J = 9.2 Hz, 1 H), 6.90 (s, 2 H), 5.92 (s, 2 H), 3.12 (q, J = 7.2 Hz, 2 H), 1.19 (t, J = 7.6 Hz, 3 H); HRMS (ESI) Calcd for $C_{13}H_{14}N_5O^+$ $(M + H)^+$ 256.11929; Found 256.11893.

1-(1,3-Diamino-7H-pyrrolo[3,2-f]quinazolin-7-yl)butan-1-one (***2c***). The title compound was prepared as described for **2a** from 65.0 mg (0.33 mmol) of **1** and compound **2c** (65.0 mg, 73%) was obtained as a yellowish solid: mp 196-198 ºC; ^{1}H NMR (400 MHz, DMSO-d_6) δ 8.59 (d, J = 9.2 Hz, 1 H), 8.05 (d, J = 3.6 Hz, 1 H), 7.39 (d, J = 4.0 Hz, 1 H), 7.18 (d, J = 8.8 Hz, 1 H), 6.90 (brs, 2 H), 5.92 (s, 2 H), 3.07 (t, J = 7.2 Hz, 2 H), 1.73 (sextet, J = 7.2 Hz, 2 H), 0.99 (t, J = 7.2 Hz, 3 H); HRMS (ESI) Calcd for $C_{14}H_{16}N_5O^+$ $(M + H)^+$ 270.13494; Found 270.13507.

1-(1,3-Diamino-7H-pyrrolo[3,2-f]quinazolin-7-yl)-2-methylpropan-1-one (***2d***). The title compound was prepared as described for **2a** from 20.0 mg (0.10 mmol) of **1** and compound **2d** (21.0 mg, 78%) was obtained as a yellowish solid: mp 200-202 ºC. ^{1}H NMR (400 MHz, DMSO-d_6) δ 8.59 (d, J = 9.2 Hz, 1 H), 8.14 (d, J = 4.0 Hz, 1 H),

7.41 (d, J = 3.6 Hz, 1 H), 7.18 (d, J = 9.2 Hz, 1 H), 6.90 (s, 2 H), 5.92 (s, 2 H), 3.63 (septet, J = 6.8 Hz, 1 H), 1.24 (d, J = 6.8 Hz, 6 H); ^{13}C NMR (100 MHz, DMSO-d_6) δ 176.5, 162.3, 160.0, 151.5, 129.4, 126.5, 124.0, 122.2, 121.8, 108.1, 102.6, 32.9, 19.6; HRMS (ESI) Calcd for $C_{14}H_{16}N_5O^+$ $(M + H)^+$ 270.13494; Found 270.13498.

*5-(tert-Butyldimethylsilyloxy)-1-(1,3-diamino-7H-pyrrolo[3,2-f]quinazolin-7-yl)penta n-1-one (**2e**)*. DCC (262 mg, 1.27 mmol) was added to a stirred solution of 5-(*tert*-butyldimethylsilyloxy)pentanoic acid (247 mg, 1.06 mmol), NHS (146 mg, 1.27 mmol) and DMAP (14 mg, 0.117 mmol) in dry THF (5 mL) at 0 ºC. The resulting mixture was stirred for 24 h at room temperature. The solid was filtered off and the solvent was removed in vacuo. The residue was purified by column chromatography on silica gel, eluting with 2:1 hexanes:EtOAc to give **3e** (280 mg, 80%) as a colorless oil: ^{1}H NMR (400 MHz, $CDCl_3$) δ 3.64 (t, J = 6.4 Hz, 2 H), 2.84-2.83 (m, 4 H), 2.64 (t, J = 7.6 Hz, 2 H), 1.85-1.78 (m, 2 H), 1.65-1.58 (m, 2 H), 0.88 (s, 9 H), 0.04 (s, 6 H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 169.3, 168.8, 62.5, 31.8, 30.8, 26.1, 25.7, 21.4, 18.4, -5.2; HRMS (ESI) Calcd for $C_{15}H_{28}NO_5Si^+$ $(M + H)^+$ 330.17313; Found 330.17282. The title compound **2e** was prepared as described for **2a** from 100 mg (0.50 mmol) of **1** with the following exception: NHS ester **3e** was used as the acylating reagent. When the reaction was complete, the solvent was removed and the residue was purified by column chromatography on silica gel, eluting with THF to give the desired compound **2e** (135 mg, 65%) as a yellowish solid: mp 173-175 ºC. ^{1}H NMR (400 MHz, DMSO-d_6) δ 8.58 (d, J = 9.2 Hz, 1 H), 8.04 (d, J = 4.0 Hz, 1 H), 7.39 (d, J = 4.0 Hz, 1 H), 7.18 (d, J = 9.2 Hz, 1 H), 6.92 (brs,

2 H), 5.94 (s, 2 H), 3.64 (t, J = 6.4 Hz, 2 H), 3.11 (t, J = 6.8 Hz, 2 H), 1.78-1.72 (m, 2 H), 1.65-1.55 (m, 2 H), 0.86 (s, 9 H), 0.03 (s, 6 H); ^{13}C NMR (100 MHz, DMSO-d_6) δ 172.5, 162.2, 159.9, 151.4, 129.3, 126.6, 123.9, 122.0, 121.7, 107.8, 102.7, 62.3, 34.6, 31.6, 25.9, 20.8, 18.0, -5.3; HRMS (ESI) Calcd for $C_{21}H_{32}N_5O_2Si^+$ $(M + H)^+$ 414.23198; Found 414.23187.

(1,3-Diamino-7H-pyrrolo[3,2-f]quinazolin-7-yl)(naphthalen-2-yl)methanone (***2f***). NHS (748 mg, 6.5 mmol) and EDCI·HCl (1.25 g, 6.5 mmol) were added to a stirred solution of 2-naphthoic acid (861 mg, 5.0 mmol) in dry DMF (8 mL). The resulting mixture was stirred overnight. Then the solvent was removed and the residue was treated with H_2O (15 mL). The white solid was collected by filtration to give compound **3f** (1.31 g, 97%): mp 148-150 ºC. ^{1}H NMR (400 MHz, DMSO-d_6) δ 8.87 (s, 1 H), 8.25 (d, J = 8.4 Hz, 1 H), 8.17 (d, J = 8.8 Hz, 1 H), 8.09 (d, J = 8.4 Hz, 1 H), 8.04 (d, J = 8.4 Hz, 1 H), 7.78 (t, J = 7.6 Hz, 1 H), 7.70 (t, J = 7.2 Hz, 1 H), 2.93 (s, 4 H); ^{13}C NMR (100 MHz, DMSO-d_6) δ 170.5, 162.0, 135.9, 132.5, 132.0, 130.0, 129.8, 129.4, 128.0, 127.7, 124.5, 121.7, 25.6. The title compound was prepared as described for **2a** from 100 mg (0.50 mmol) of **1** with the following exception: NHS ester **3f** was used as the acylating reagent. Compound **2f** (160 mg, 91%) was obtained as a yellowish solid: mp 188-190 ºC. ^{1}H NMR (400 MHz, DMSO-d_6) δ 8.50 (d, J = 9.2 Hz, 1 H), 8.43 (s, 1 H), 8.16-8.13 (m, 2 H), 8.08 (d, J = 8.0 Hz, 1 H), 7.87 (d, J = 8.8 Hz, 1 H), 7.72 (t, J = 8.0 Hz, 1 H), 7.67 (t, J = 7.6 Hz, 1 H), 7.61 (d, J = 3.6 Hz, 1 H), 7.44 (d, J = 3.2 Hz, 1 H), 7.25 (d, J = 9.2 Hz, 1 H), 6.94 (s, 2 H), 5.98 (s, 2 H); ^{13}C NMR (100 MHz, DMSO-d_6) δ 168.5, 162.3, 160.0, 151.6, 134.5, 131.9, 131.0, 130.6,

129.8, 129.3, 128.7, 128.6, 128.5, 127.9, 127.3, 125.5, 124.4, 122.0, 121.5, 108.2, 102.7; HRMS (ESI) Calcd for $C_{21}H_{16}N_5O^+$ $(M + H)^+$ 354.13494; Found 354.13490.

*N-(3-Amino-7H-pyrrolo[3,2-f]quinazolin-1-yl)propionamide (**4b**)*. The title compound was prepared as described for **4a** (Method A) from 53.0 mg (0.21 mmol) of **2b**, and after column chromatography on silica gel, eluting with 15:1 DCM:MeOH containing 1% DIPEA, compound **4b** (17.0 mg, 32%) was obtained as a yellowish solid: mp 226-228 ºC. ^{1}H NMR (400 MHz, DMSO-d_6) δ 11.59 (s, 1 H), 10.19 (s, 1 H), 7.80 (d, J = 8.8 Hz, 1 H), 7.42 (t, J = 2.8 Hz, 1 H), 7.17 (d, J = 8.8 Hz, 1 H), 6.68 (brs, 1 H), 6.39 (s, 2 H), 2.53 (q, J = 7.6 Hz, 2 H), 1.12 (t, J = 7.6 Hz, 3 H); ^{13}C NMR (100 MHz, DMSO-d_6) δ 173.1, 159.1, 157.1, 152.1, 130.6, 124.8, 121.2, 119.9, 118.8, 109.3, 103.6, 29.0, 9.4; HRMS (ESI) Calcd for $C_{13}H_{14}N_5O^+$ $(M + H)^+$ 256.11929; Found 256.11941.

*N-(3-Amino-7H-pyrrolo[3,2-f]quinazolin-1-yl)butyramide (**4c**)*. The title compound was prepared as described for **4a** (Method A) from 47.0 mg (0.175 mmol) of **2c**, and after column chromatography on silica gel, eluting with 15:1 DCM:MeOH containing 1% DIPEA, compound **4c** (15.0 mg, 32%) was obtained as a yellowish solid: mp 224-226 ºC. ^{1}H NMR (400 MHz, DMSO-d_6) δ 11.59 (s, 1 H), 10.17 (s, 1 H), 7.80 (d, J = 8.8 Hz, 1 H), 7.42 (t, J = 2.8 Hz, 1 H), 7.17 (d, J = 8.8 Hz, 1 H), 6.70 (brs, 1 H), 6.36 (s, 2 H), two protons were buried in residual DMSO signal, 1.65 (sextet, J = 7.6 Hz, 2 H), 0.96 (t, J = 7.2 Hz, 3 H); ^{13}C NMR (100 MHz, DMSO-d_6) δ 172.2, 159.1, 157.1, 152.1, 130.6, 124.7, 121.2, 119.9, 118.8, 109.4, 103.6, 37.7, 18.2, 13.9; HRMS (ESI) Calcd for $C_{14}H_{16}N_5O^+$ $(M + H)^+$, 270.13494; Found 270.13497.

N-(3-Amino-7H-pyrrolo[3,2-f]quinazolin-1-yl)isobutyramide ***(4d)***. The title compound was prepared as described for **4a** (Method A) from 52.0 mg (0.193 mmol) of **2d**, and after column chromatography on silica gel, eluting with 10:1 DCM:MeOH containing 1% DIPEA, compound **4d** (15.0 mg, 27%) was obtained as a yellowish solid: mp 232-234 ºC. ^{1}H NMR (400 MHz, DMSO-d_6) δ 11.59 (s, 1 H), 10.21 (s, 1 H), 7.80 (d, J = 9.2 Hz, 1 H), 7.43 (t, J = 2.8 Hz, 1 H), 7.17 (d, J = 9.2 Hz, 1 H), 6.70 (brs, 1 H), 6.41 (s, 2 H), 2.85 (septet, J = 6.8 Hz, 1 H), 1.18 (d, J = 7.2 Hz, 6 H); ^{13}C NMR (100 MHz, DMSO-d_6) δ 175.9, 159.1, 157.3, 152.2, 130.6, 124.7, 121.2, 119.9, 118.8, 109.7, 103.6, 34.3, 19.3; HRMS (ESI) Calcd for $C_{14}H_{16}N_5O^+$ $(M + H)^+$, 270.13494; Found 270.13467.

N-(3-Amino-7H-pyrrolo[3,2-f]quinazolin-1-yl)-5-(tert-butyldimethylsilyloxy) pentanamide ***(4e)***. The title compound was prepared as described for **4a** (Method A) from 261 mg (0.631 mmol) of **2e**, and after column chromatography on silica gel, eluting with 20:1 DCM:MeOH containing 1% DIPEA, compound **4e** (100 mg, 38%) was obtained as a yellowish solid: mp 172-174 ºC. ^{1}H NMR (400 MHz, DMSO-d_6) δ 11.61 (s, 1 H), 10.23 (s, 1 H), 7.82 (d, J = 9.2 Hz, 1 H), 7.42 (s, 1 H), 7.17 (d, J = 8.8 Hz, 1 H), 6.70 (s, 1 H), 6.48 (brs, 2 H), 3.61 (t, J = 6.0 Hz, 2 H), two protons were buried in residual DMSO signal, 1.73-1.62 (m, 2 H), 1.58-1.52 (m, 2 H), 0.86 (s, 9 H), 0.03 (s, 6 H); ^{13}C NMR (100 MHz, DMSO-d_6) δ 172.3, 159.1, 157.1, 152.2, 130.7, 124.7, 121.3, 120.0, 118.9, 109.4, 103.6, 62.3, 35.5, 32.0, 25.9, 21.3, 18.0, -5.2; HRMS (ESI) Calcd for $C_{21}H_{32}N_5O_2Si^+$ $(M + H)^+$ 414.23198; Found 414.23145.

N-(3-Amino-7H-pyrrolo[3,2-f]quinazolin-1-yl)-2-naphthamide ***(4f)*** and

*N-(3-Amino-2,7-dihydro-1H-pyrrolo[3,2-f]quinazolin-1-ylidene)-2-naphthamide (**4f′**).* The title compound was prepared as described for **4a** (Method B) from 80 mg (0.226 mmol) of **2f**, and after column chromatography on silica gel, eluting with 20:1 DCM:MeOH containing 1% DIPEA, a yellow solid was obtained (40 mg, 50%), which exists as a 1:1 tautomeric mixture of **4f** and **4f'** in DMSO-d_6: mp 230-232 ºC. ^{1}H NMR (400 MHz, DMSO-d_6) δ 14.37 (s, 1 H), 11.64 (s, 1 H), 11.57 (s, 1 H), 11.11 (s, 1 H), 8.93 (s, 1 H), 8.78 (s, 1 H), 8.44 (d, *J* = 8.4 Hz, 1 H), 8.14-8.02 (m, 7 H), 7.87-7.83 (m, 2 H), 7.78 (brs, 1 H), 7.71-7.62 (m, 5 H), 7.30 (brs, 1 H), 7.25-7.23 (m, 3 H), 7.09 (d, *J* = 8.4 Hz, 1 H), 6.57 (s, 1 H), 6.50 (s, 2 H); ^{13}C NMR (100 MHz, DMSO-d_6) δ 177.7, 166.4, 159.3, 158.7, 157.8, 152.3, 149.7, 148.4, 135.8, 134.7, 132.4, 131.6, 131.2, 130.7, 129.7, 129.3, 128.8, 128.4, 128.2, 127.9, 127.8, 127.0, 126.7, 126.3, 125.6, 125.0, 124.4, 123.0, 121.5, 120.1, 118.9, 118.5, 110.6, 107.7, 105.4, 103.4; HRMS (ESI) Calcd for $C_{21}H_{32}N_5O_2Si^+$ $(M + H)^+$ 354.13494; Found 354.13475. When a mixture of **4f** and **4f′** in DMSO-d_6 (375 μL) was treated with an aqueous solution NaOH (125 μL, 0.4 N), it became **4f** and all the active protons disappeared due to the H-D exchange:[3] ^{1}H NMR (400 MHz) 8.61 (s, 1 H), 8.33 (dd, *J* = 8.4 Hz, 1.2 Hz, 1 H), 7.97-7.86 (m, 3 H), 7.64 (d, *J* = 8.8 Hz, 1 H), 7.53-7.48 (m, 2 H), 7.15 (d, *J* = 2.8 Hz, 1 H), 7.00 (d, *J* = 2.4 Hz, 1 H), 6.94 (d, *J* = 8.8 Hz, 1 H); ^{13}C NMR (100 MHz) 171.6, 166.6, 159.2, 148.9, 139.4, 134.1, 133.6, 133.2, 129.3, 128.4, 128.1, 127.4, 127.3, 127.1, 126.6, 123.6, 120.7, 117.0, 110.9, 104.9.

*N-(1-Hydroxy-7H-pyrrolo[3,2-f]quinazolin-3-yl)propionamide (**6b**).* The title compound was prepared as described for **6a** from 30 mg of **5** and compound **6b** (32

mg, 84%) was obtained as a brown solid: mp 262-264 °C. ^{1}H NMR (400 MHz, DMSO-d_6) δ 12.01 (s, 1 H), 11.58 (s, 1 H), 11.46 (s, 1 H), 7.83 (d, J = 8.8 Hz, 1 H), 7.55 (t, J = 2.4 Hz, 1 H), 7.26-7.18 (m, 2 H), 2.47 (q, J = 7.2 Hz, 2 H), 1.09 (t, J = 7.6 Hz, 3 H); ^{13}C NMR (100 MHz, DMSO-d_6) δ 176.9, 160.4, 144.9, 144.8, 132.7, 127.3, 123.7, 119.6, 119.0, 111.1, 103.0, 29.4, 8.9; HRMS (ESI) Calcd for $C_{13}H_{13}N_4O_2^+$ (M + H)$^+$ 257.10330; Found 257.10318.

*N-(1-Hydroxy-7H-pyrrolo[3,2-f]quinazolin-3-yl)butyramide (**6c**)*. The title compound was prepared as described for **6a** from 35 mg of **5** and compound **6c** (40 mg, 85%) was obtained as a brown solid: mp 270-272 °C. ^{1}H NMR (400 MHz, DMSO-d_6) δ 12.01 (s, 1 H), 11.65 (s, 1 H), 11.45 (s, 1 H), 7.83 (d, J = 9.2 Hz, 1 H), 7.55 (t, J = 2.4 Hz, 1 H), 7.26-7.21 (m, 2 H), 2.44 (t, J = 7.6 Hz, 2 H), 1.63 (sextet, J = 7.2 Hz, 2 H), 0.93 (t, J = 7.2 Hz, 3 H); ^{13}C NMR (100 MHz, DMSO-d_6) δ 176.1, 160.4, 144.9, 144.8, 132.8, 127.3, 123.7, 119.6, 119.0, 111.1, 103.0, 37.9, 18.1, 13.5; HRMS (ESI) Calcd for $C_{14}H_{15}N_4O_2^+$ (M + H)$^+$ 271.11895; Found 271.11874.

*N-(1-Hydroxy-7H-pyrrolo[3,2-f]quinazolin-3-yl)isobutyramide (**6d**)*. The title compound was prepared as described for **6a** from 30 mg of **5** and compound **6d** (27 mg, 67%) was obtained as a brown solid: mp 240-242 °C. ^{1}H NMR (400 MHz, DMSO-d_6) δ 12.01 (s, 1 H), 11.65 (s, 1 H), 11.45 (s, 1 H), 7.84 (d, J = 8.8 Hz, 1 H), 7.55 (t, J = 2.8 Hz, 1 H), 7.28-7.20 (m, 2 H), 2.77 (septet, J = 7.2 Hz, 1 H), 1.13 (d, J = 7.2 Hz, 6 H); ^{13}C NMR (100 MHz, DMSO-d_6) δ 180.1, 160.3, 145.0, 144.8, 132.8, 127.3, 123.7, 119.5, 119.0, 111.1, 103.0, 34.8, 19.0; HRMS (ESI) Calcd for $C_{14}H_{15}N_4O_2^+$ (M + H)$^+$, 271.11895; Found 271.11881.

*5-[(tert-Butyldimethylsilyl)oxy]-N-(1-hydroxy-7H-pyrrolo[3,2-f]quinazolin-3-yl)penta namide (**6e**)*. A mixture of **5** (100.0 mg, 0.5 mmol) and **3e** (247 mg, 0.75 mmol) in dry DMF (5 mL) was stirred at 90 ºC for 4 h. Then the solvent was removed and the residue was purified by column chromatography on silica gel, eluting with 2:1 DCM:EtOAc containing 1% DIPEA to give compound **6e,** which was further washed with Et_2O (3 mL) to give the desired compound as a white solid (130 mg, 62%): mp 188-190 ºC. ^{1}H NMR (400 MHz, DMSO-d_6) δ 12.0 (s, 1 H), 11.6 (s, 1 H), 11.5 (s, 1 H), 7.83 (d, J = 8.8 Hz, 1 H), 7.55 (brs, 1 H), 7.23-7.21 (m, 2 H), 3.60 (t, J = 6.0 Hz, 2 H), 2 protons were buried in residual DMSO signal, 1.70-1.60 (m, 2 H), 1.55-1.45 (m, 2 H), 0.86 (s, 9 H), 0.03 (s, 6 H); ^{13}C NMR (100 MHz, DMSO-d_6) δ 176.2, 160.4, 144.9, 144.8, 132.8, 127.3, 123.7, 119.6, 119.0, 111.1, 103.0, 62.1, 35.7, 31.6, 25.9, 21.0, 18.0, -5.2; HRMS (ESI) Calcd for $C_{21}H_{31}N_4O_3Si^+$ $(M + H)^+$ 415.21599; Found 415.21555.

*N-(1-Hydroxy-7H-pyrrolo[3,2-f]quinazolin-3-yl)-2-naphthamide (**6f**)*. The title compound was prepared as described for **6e** from 40 mg of **5** and **3f**, and after column chromatography on silica gel, eluting with 3:1 EtOAc:DCM containing 1% DIPEA, compound **6f** (40 mg, 67%) was obtained as a yellowish solid: mp 226-228 ºC. ^{1}H NMR (400 MHz, DMSO-d_6) δ 12.5 (brs, 1 H), 11.9 (brs, 1 H), 11.7 (s, 1 H), 8.8 (s, 1 H), 8.16 (d, J = 8.0 Hz, 1 H), 8.10-8.01 (m, 3 H), 7.89 (d, J = 8.8 Hz, 1 H), 7.69-7.60 (m, 3 H), 7.34 (d, J = 8.4 Hz, 1 H), 7.25 (s, 1 H); ^{13}C NMR (100 MHz, DMSO-d_6) δ 160.7, 134.8, 132.9, 132.1, 129.7, 129.4, 128.4, 128.0, 127.9, 127.7, 127.0, 124.8, 123.9, 119.8, 102.9; HRMS (ESI) Calcd for $C_{21}H_{15}N_4O_2^+$ $(M + H)^+$ 355.11895; Found

355.11893.

*N-(1-Amino-7H-pyrrolo[3,2-f]quinazolin-3-yl)propionamide (**7b**)*. The title compound was prepared as described for **7a** from 32 mg of **6b**, and after column chromatography on silica gel, eluting with 1.5:1 EtOAc:THF containing 1% DIPEA, compound **7b** (14 mg, 44%) was obtained as a yellowish solid: mp 228-230 ºC. ^{1}H NMR (400 MHz, DMSO-d_6) δ 11.79 (s, 1 H), 9.73 (s, 1 H), 7.83 (d, J = 8.8 Hz, 1 H), 7.56 (t, J = 2.4 Hz, 1 H), 7.27 (d, J = 8.8 Hz, 1 H), 7.24 (brs, 1 H), 7.10 (brs, 2 H), 2.56 (q, J = 7.6 Hz, 2 H), 1.07 (t, J = 7.6 Hz, 3 H); ^{13}C NMR (100 MHz, DMSO-d_6) δ 172.8, 161.9, 152.4, 148.4, 131.9, 125.6, 120.0, 119.6, 119.4, 104.8, 102.5, 29.5, 9.6; HRMS (ESI) Calcd for $C_{13}H_{14}N_5O^+$ $(M + H)^+$ 256.11929; Found 256.11913.

*N-(1-Amino-7H-pyrrolo[3,2-f]quinazolin-3-yl)butyramide (**7c**)*. The title compound was prepared as described for **7a** from 35 mg of **6c**, and after column chromatography on silica gel, eluting with 1.5:1 EtOAc:THF containing 1% DIPEA, compound **7c** (13 mg, 37%) was obtained as a yellowish solid: mp 262-264 ºC. ^{1}H NMR (400 MHz, DMSO-d_6) δ 11.79 (s, 1 H), 9.74 (s, 1 H), 7.83 (d, J = 8.8 Hz, 1 H), 7.56 (t, J = 2.4 Hz, 1 H), 7.27 (d, J = 9.2 Hz, 1 H), 7.24 (brs, 1 H), 7.09 (brs, 2 H), two protons were buried in residual DMSO signal, 1.60 (sextet, J = 7.2 Hz, 2 H), 0.93 (t, J = 7.6 Hz, 3 H); ^{13}C NMR (100 MHz, DMSO-d_6) δ 171.8, 161.9, 152.4, 148.4, 131.9, 125.6, 120.0, 119.6, 119.4, 104.8, 102.5, 38.1, 18.4, 13.8; HRMS (ESI) Calcd for $C_{14}H_{16}N_5O^+$ $(M + H)^+$ 270.13494; Found 270.13474.

*N-(1-Amino-7H-pyrrolo[3,2-f]quinazolin-3-yl)isobutyramide (**7d**)*. The title compound was prepared as described for **7a** from 35 mg of **6d**, and after column

chromatography on silica gel, eluting with 2:1 EtOAc:THF containing 1% DIPEA, compound **7d** (14 mg, 40%) was obtained as a yellowish solid: mp 288-290 ºC. ^{1}H NMR (400 MHz, DMSO-d_6) δ 11.79 (s, 1 H), 9.79 (s, 1 H), 7.83 (d, J = 9.2 Hz, 1 H), 7.56 (t, J = 2.8 Hz, 1 H), 7.27 (d, J = 9.2 Hz, 1 H), 7.24 (brs, 1 H), 7.08 (brs, 2 H), 2.92 (brs, 1 H), 1.08 (d, J = 6.8 Hz, 6 H); ^{13}C NMR (100 MHz, DMSO-d_6) δ 175.3, 162.0, 152.4, 148.4, 131.9, 125.6, 120.0, 119.6, 119.4, 105.0, 102.6, 34.1, 19.5; HRMS (ESI) Calcd for $C_{14}H_{16}N_5O^+$ $(M + H)^+$ 270.13494; Found 270.13484.

*N-(1-Amino-7H-pyrrolo[3,2-f]quinazolin-3-yl)-5-[(tert-butyldimethylsilyl)oxy]pentan amide (**7e**)*. The title compound was prepared as described for **7a** from 80 mg of **6e**, and after column chromatography on silica gel, eluting with 4:1 EtOAc:THF containing 1% DIPEA, compound **7e** (40 mg, 50%) was obtained as a light green solid after treating with NH_4OH (3 mL) and hexanes (3 mL) successively: mp 140-142 ºC. ^{1}H NMR (400 MHz, DMSO-d_6) δ 11.77 (s, 1 H), 9.74 (s, 1 H), 7.83 (d, J = 8.8 Hz, 1 H), 7.56 (t, J = 2.4 Hz, 1 H), 7.26 (d, J = 9.2 Hz, 1 H), 7.23 (s, 1 H), 7.08 (brs, 2 H), 3.60 (t, J = 6.0 Hz, 2 H), two protons were buried in residual DMSO signal, 1.65-1.57 (m, 2 H), 1.54-1.47 (m, 2 H), 0.85 (s, 9 H), 0.02 (s, 6 H); ^{13}C NMR (100 MHz, DMSO-d_6) δ 172.0, 162.9, 152.4, 148.4, 131.9, 125.6, 120.0, 119.6, 119.4, 104.9, 102.6, 62.4, 35.9, 32.0, 25.9, 21.5, 18.0, -5.2; HRMS (ESI) Calcd for $C_{21}H_{32}N_5O_2Si^+$ $(M + H)^+$ 414.23198; Found 414.23145.

*N-(1-Amino-7H-pyrrolo[3,2-f]quinazolin-3-yl)-2-naphthamide (**7f**)*. The title compound was prepared as described for **7a** from 40 mg of **6f**, and after column chromatography on silica gel, eluting with 20:1 EtOAc:THF containing 1% DIPEA,

compound **7f** (10 mg, 25%) was obtained as a yellow solid: mp 234-236 °C. ^{1}H NMR (400 MHz, DMSO-d_6) δ 11.85 (s, 1 H), 10.61 (brs, 1 H), 8.65 (s, 1 H), 8.08-7.99 (m, 4 H), 7.90 (d, J = 8.8 Hz, 1 H), 7.66-7.60 (m, 3 H), 7.39-7.20 (m, 4 H); ^{13}C NMR (100 MHz, DMSO-d_6) δ 162.3, 152.8, 134.4, 132.4, 132.3, 132.2, 129.2, 128.5, 127.9, 127.8, 127.7, 126.7, 125.9, 124.8, 119.8, 119.4, 105.3, 102.7; HRMS (ESI) Calcd for $C_{21}H_{16}N_5O^+$ $(M + H)^+$ 354.13494; Found 354.13478.

Biology. General information. pCRE-RLuc was described previously.[4] MDA-MB-231 and MDA-MB-468 cells were obtained from Development Therapeutics Program at the National Cancer Institute. The cells were maintained in Dulbecco's modified Eagle medium (DMEM, Invitrogen, Carlsbad, CA) supplemented with 10% (v/v) fetal bovine serum (FBS), 10 μg/mL penicillin and 10 μg/mL streptomycin (Invitrogen, Carlsbad, CA) at 37 °C under 5% CO_2. HMEC was from Lonza (Walkersville, MA) and cultured following the manufacture's instructions.

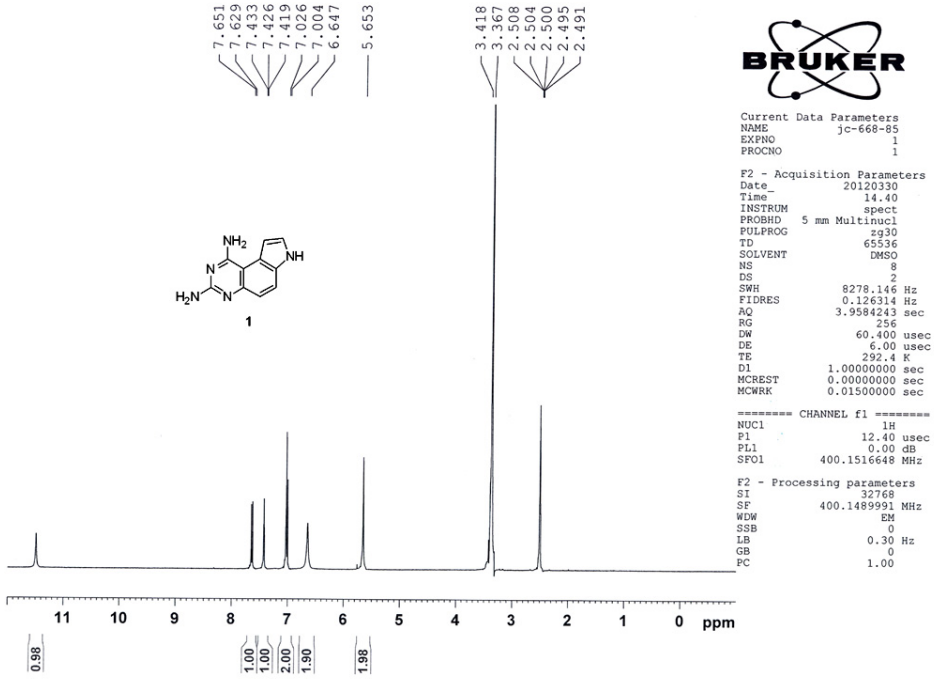
Inhibition of human DHFR. The human DHFR assay was carried out with DHFR assay kit (Sigma) following manufacture's instructions with minor modifications. Briefly, different concentrations of a compound were incubated with human DHFR (0.1875 mU) and reduced nicotinamide adenine dinucleotide phosphate (NADPH, 60 μM) in the provided 1X assay buffer for 2 min at room temperature. Then dihydrofolic acid (DHF, 50 μM) was added to initiate the reduction reaction, which was immediately monitored by absorbance at 340 nm every 12 s for 3 min. The final reaction volume was 100 μL and the final DMSO concentration was 1%. The reaction

velocities were calculated as the slopes from the absorbance-time curves. The data were presented as mean ± SE of two independent experiments.

MTT assays. The MTT assays for MDA-MB-231, MDA-MB-468 and HMEC were performed as described.[5] The GI_{50}s were calculated using Prism 5.0 as described before.[5]

Inhibition of CREB-mediated gene transcription. The inhibition of CREB-mediated gene transcription in HEK 293T cells by a CREB reporter assay was done as previously described.[4]

Molecular Modeling. All the molecular modeling work was conducted in the Schrödinger modeling suite (Portland, OR). The structures were optimized at HF/6-31G** level of theory in Jaguar. The structural minima were confirmed by the absence of any negative vibrational frequencies. The MEP surfaces were generated by mapping the electrostatic potentials onto the electron densities and were normalized from -50 kcal/mol to +50 kcal/mol.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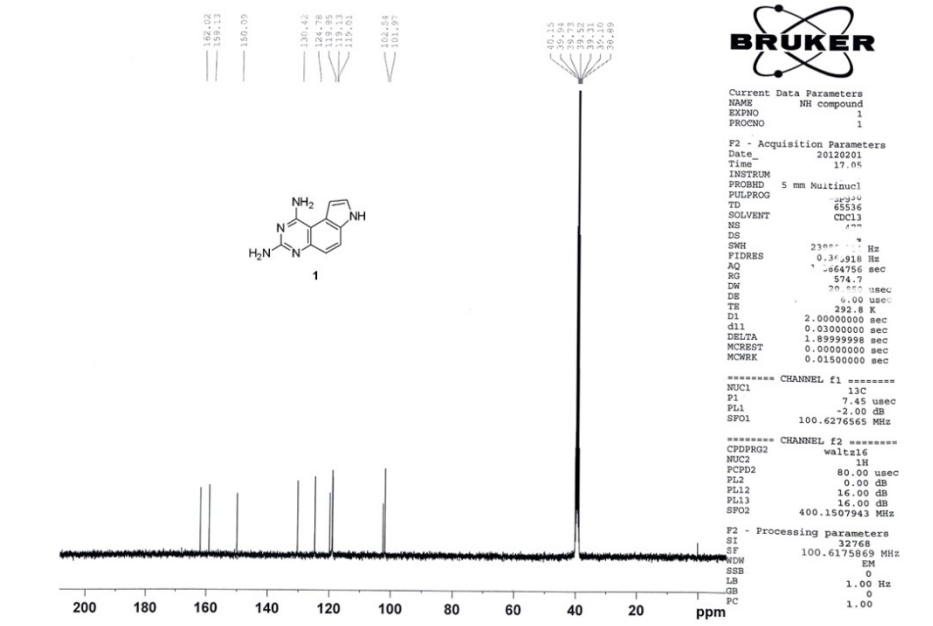
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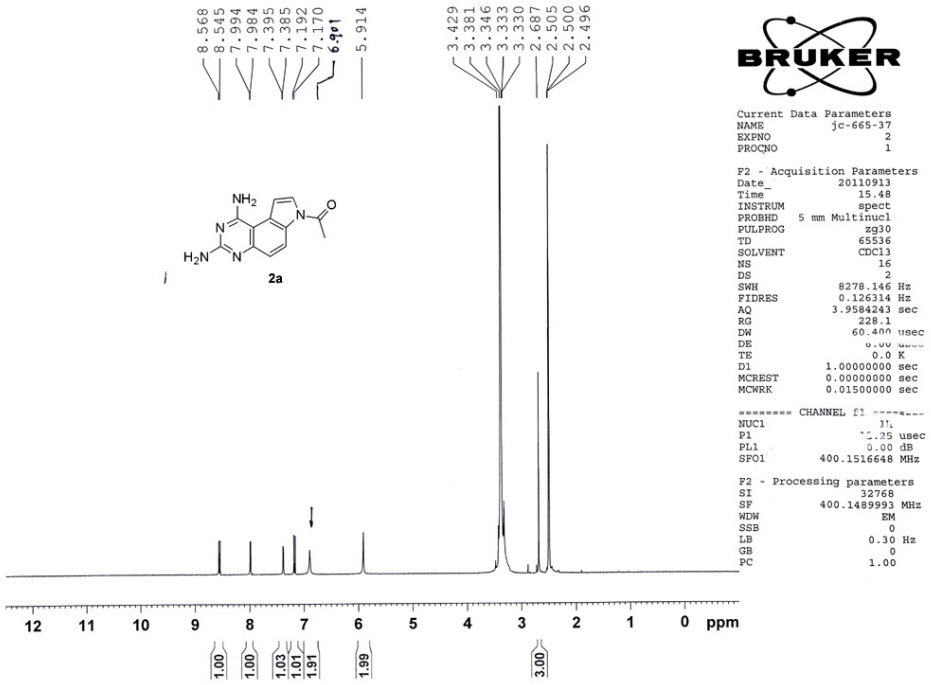
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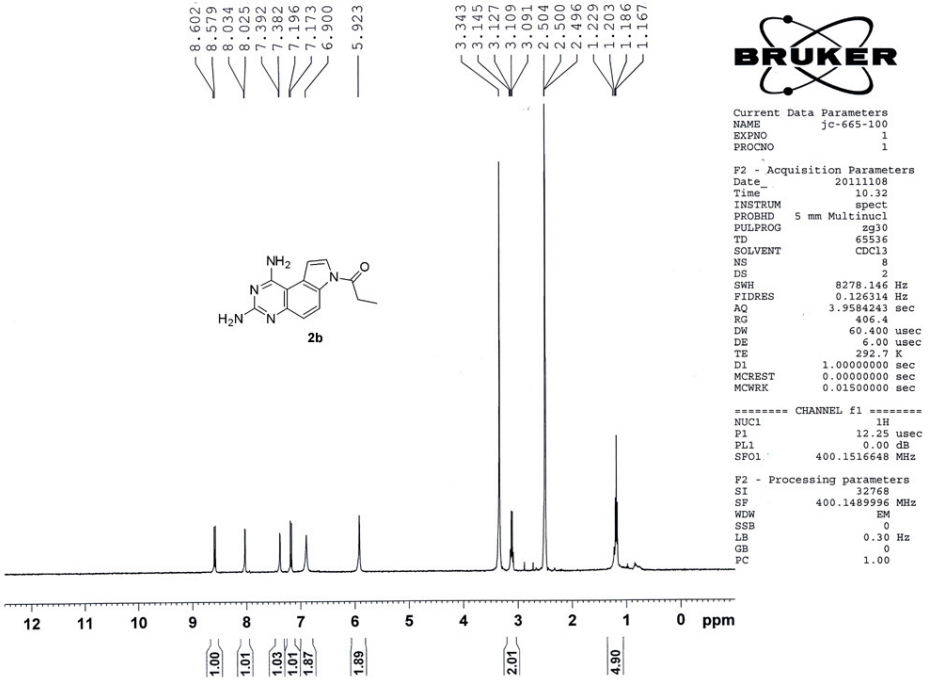
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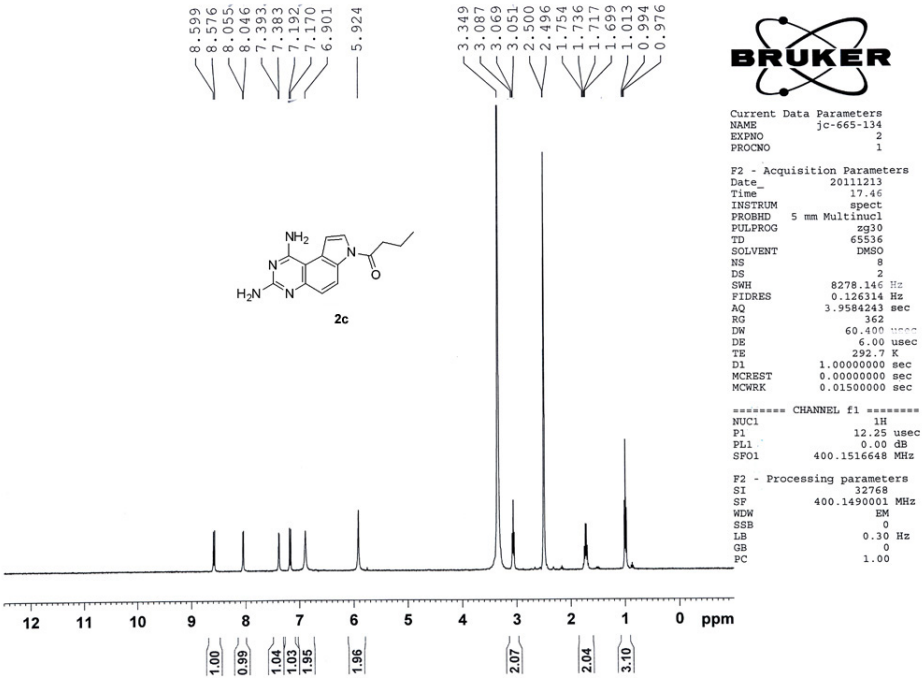
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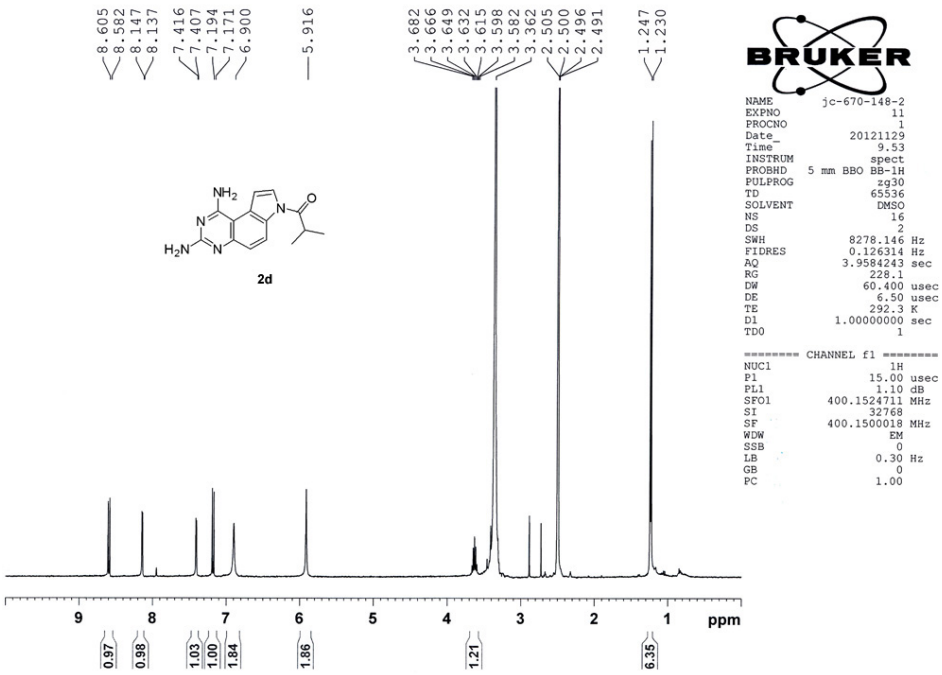
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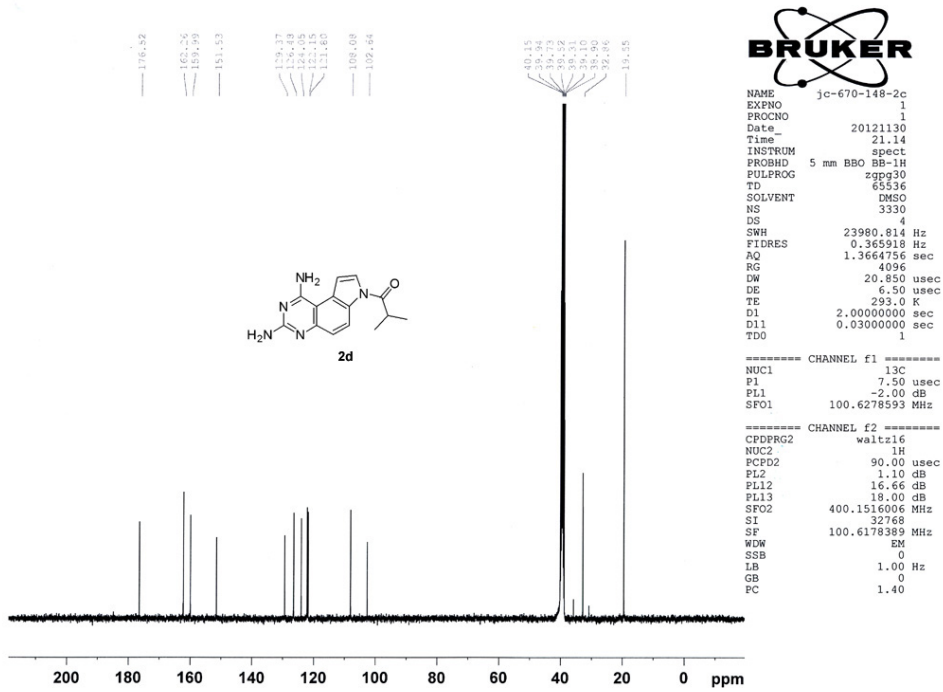
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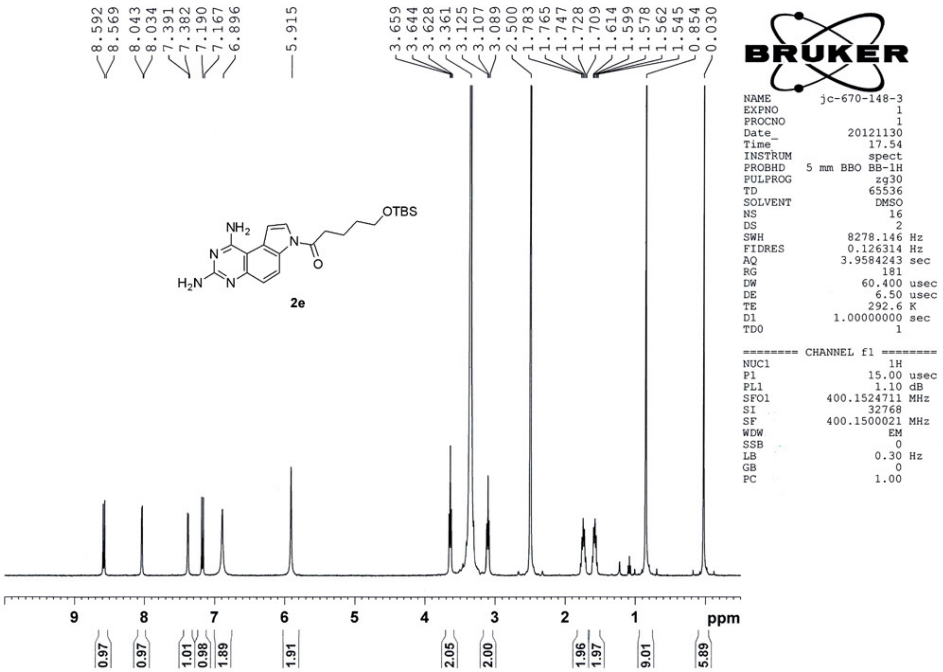
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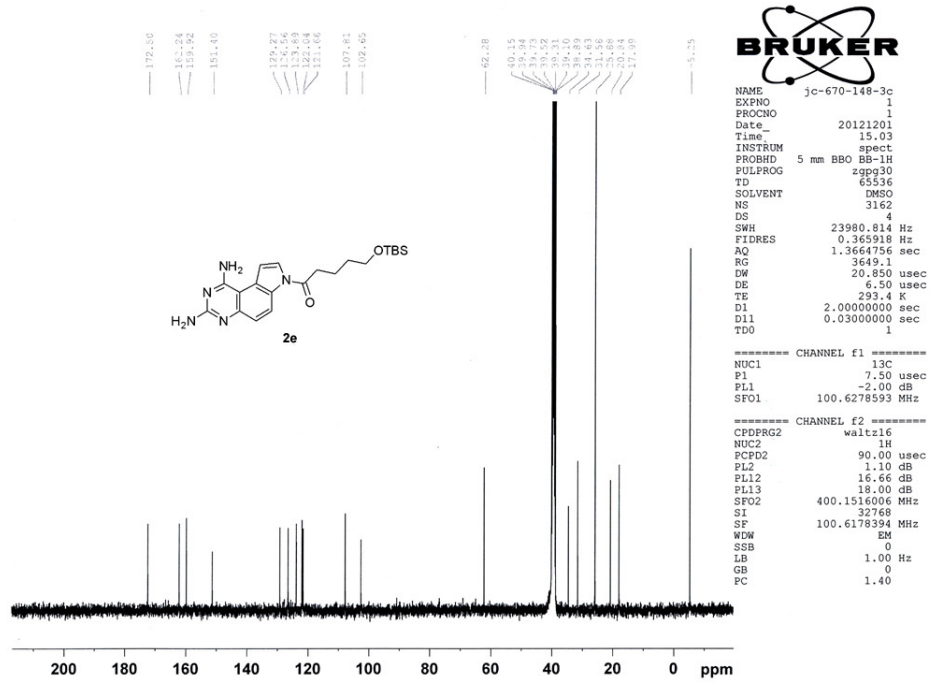
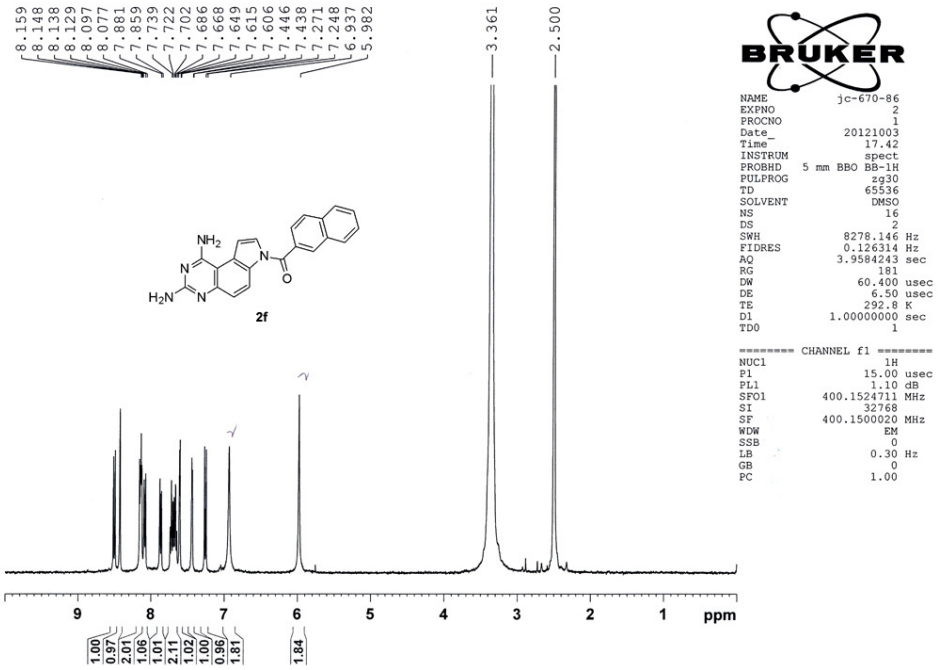
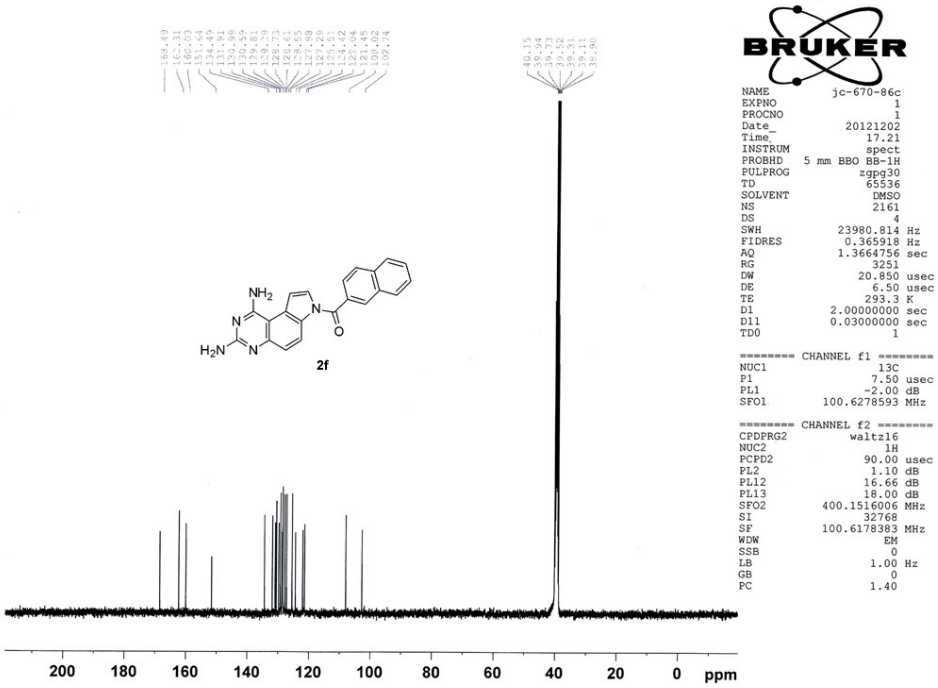
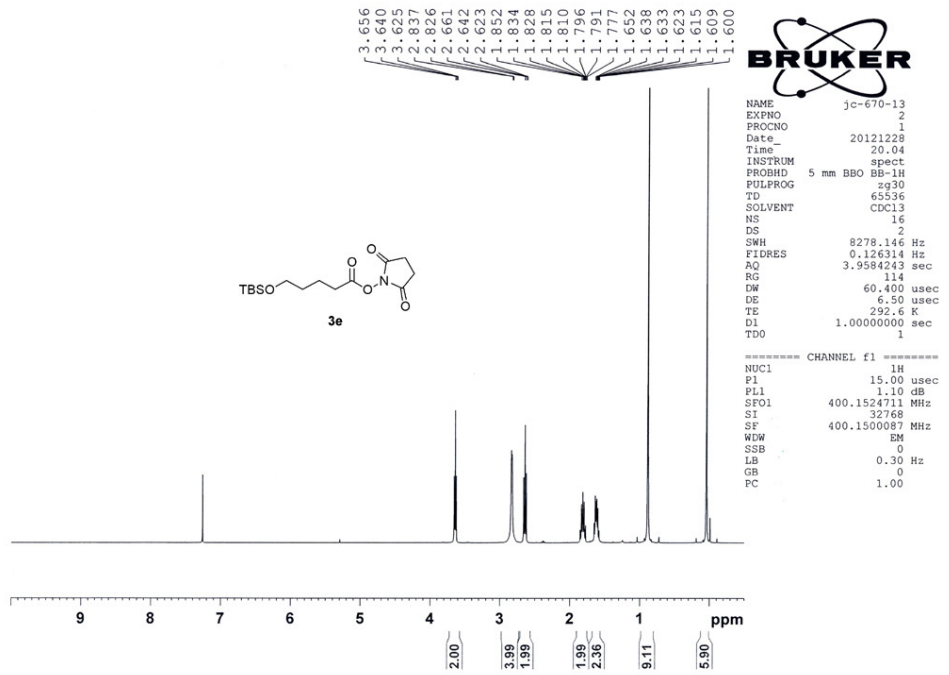
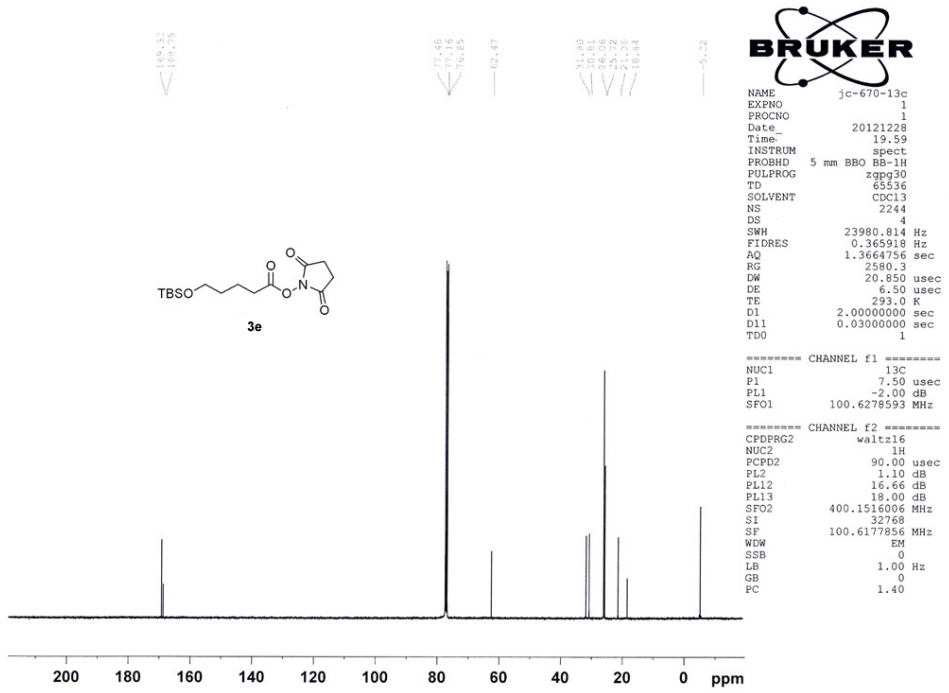
8.034
7.391
7.382
7.190
7.167
6.896
5.915
3.659
3.644
3.628
3.361
3.125
3.107
3.089
2.500
1.783
1.765
1.747
1.728
1.709
1.614
1.599
1.578
1.562
1.545
0.854
0.030
OTBS
NH2
N
N
H2N
O
2e
9
8
7
6
5
4
3
2
1
ppm
0.97
0.97
1.01
0.98
1.89
1.91
2.05
2.00
1.96
1.97
9.01
5.89
BRUKER
NAME jc-670-148-3
EXPNO 1
PROCNO 1
Date_ 20121130
Time_ 17.54
INSTRUM spect
PROBHD 5 mm BBO BB-1H
PULPROG zg30
TD 65536
SOLVENT DMSO
NS 16
DS 2
SWH 8278.146 Hz
FIDRES 0.126314 Hz
AQ 3.9584243 sec
RG 181
DW 60.400 usec
DE 6.50 usec
TE 292.6 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 15.00 usec
PL1 1.10 dB
SFO1 400.1524711 MHz
SI 32768
SF 400.1500021 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

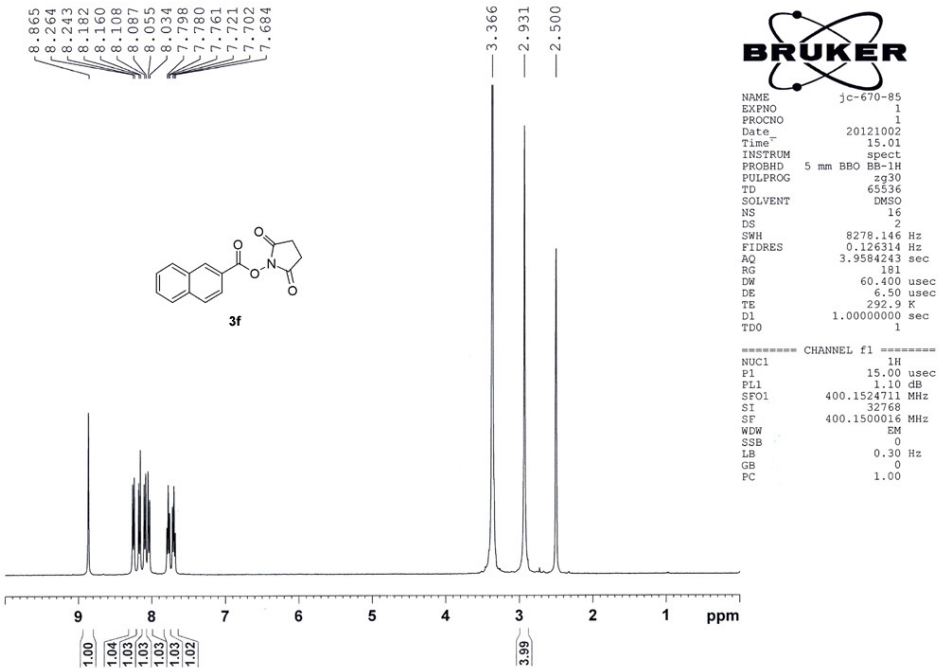

172.50
162.24
159.92
151.40
129.27
126.56
125.89
123.04
121.66
107.81
102.65
62.28
40.15
39.94
39.73
39.52
39.31
39.10
38.89
34.63
31.50
25.88
20.84
17.99
5.25
OTBS
NH2
N
N
H2N
O
2e
200
180
160
140
120
100
80
60
40
20
0
ppm
BRUKER
NAME jc-670-148-3c
EXPNO 1
PROCNO 1
Date_ 20121201
Time_ 15.03
INSTRUM spect
PROBHD 5 mm BBO BB-1H
PULPROG zgpg30
TD 65536
SOLVENT DMSO
NS 3162
DS 4
SWH 23980.814 Hz
FIDRES 0.365918 Hz
AQ 1.3664756 sec
RG 3649.1
DW 20.850 usec
DE 6.50 usec
TE 293.4 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 7.50 usec
PL1 -2.00 dB
SFO1 100.6278593 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 90.00 usec
PL2 1.10 dB
PL12 16.66 dB
PL13 18.00 dB
SFO2 400.1516006 MHz
SI 32768
SF 100.6178394 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

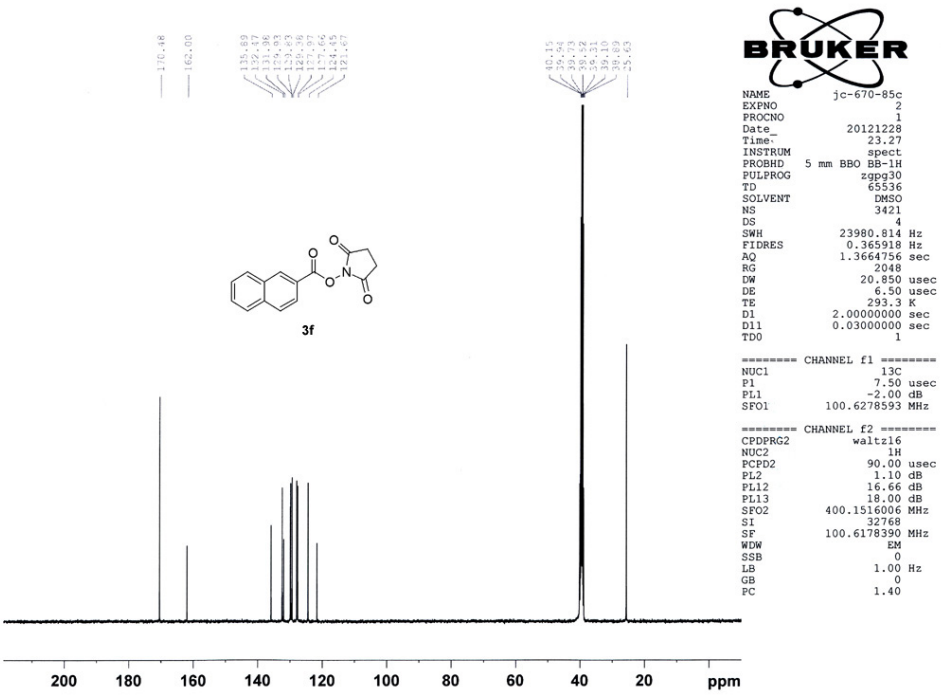

8.159
8.148
8.138
8.129
8.097
8.077
7.881
7.859
7.739
7.722
7.702
7.686
7.668
7.649
7.615
7.606
7.446
7.438
7.271
7.248
6.937
5.982
3.361
2.500
NH2
N
N
H2N
O
2f
9
8
7
6
5
4
3
2
1
ppm
1.00
0.97
2.01
1.06
1.01
2.11
1.02
1.00
0.96
1.81
1.84
BRUKER
NAME jc-670-86
EXPNO 2
PROCNO 1
Date_ 20121003
Time 17.42
INSTRUM spect
PROBHD 5 mm BBO BB-1H
PULPROG zg30
TD 65536
SOLVENT DMSO
NS 16
DS 2
SWH 8278.146 Hz
FIDRES 0.126314 Hz
AQ 3.9584243 sec
RG 181
DW 60.400 usec
DE 6.50 usec
TE 292.8 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 15.00 usec
PL1 1.10 dB
SFO1 400.1524711 MHz
SI 32768
SF 400.1500020 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

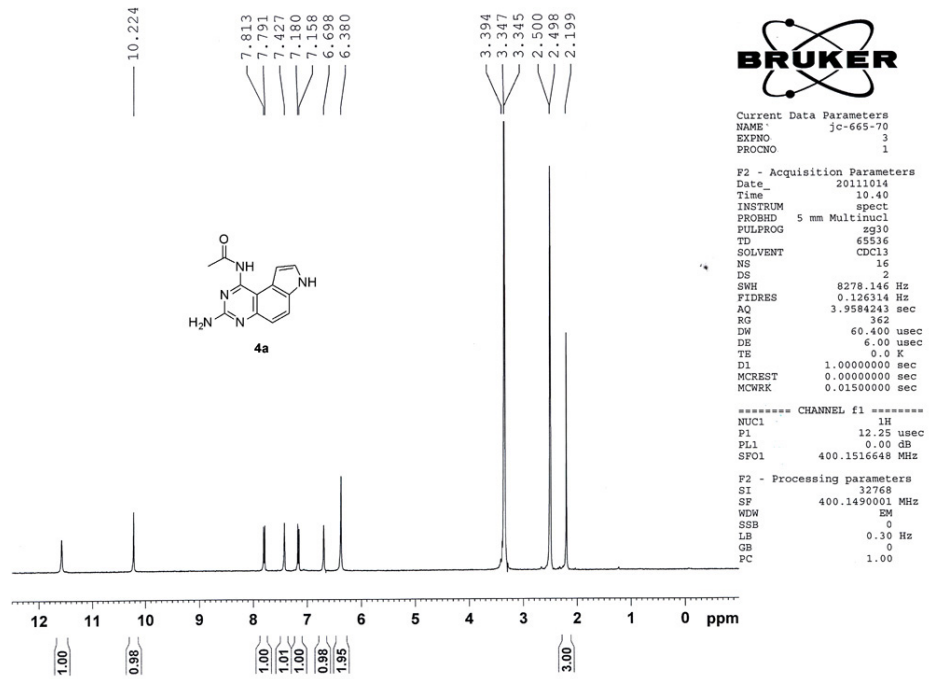

168.49
160.31
160.03
151.64
134.45
131.41
130.99
130.55
129.81
129.39
128.73
128.61
126.55
125.88
125.21
124.42
122.04
121.45
121.02
100.74
40.15
39.94
39.73
39.52
39.32
39.11
38.90
NH2
N
N
H2N
O
2f
200
180
160
140
120
100
80
60
40
20
0
ppm
BRUKER
NAME jc-670-86c
EXPNO 1
PROCNO 1
Date_ 20121202
Time 17.21
INSTRUM spect
PROBHD 5 mm BBO BB-1H
PULPROG zgpg30
TD 65536
SOLVENT DMSO
NS 2161
DS 4
SWH 23980.814 Hz
FIDRES 0.365918 Hz
AQ 1.3664756 sec
RG 3251
DW 20.850 usec
DE 6.50 usec
TE 293.3 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 7.50 usec
PL1 -2.00 dB
SFO1 100.6278593 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 90.00 usec
PL2 1.10 dB
PL12 16.66 dB
PL13 18.00 dB
SFO2 400.1516006 MHz
SI 32768
SF 100.6178383 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

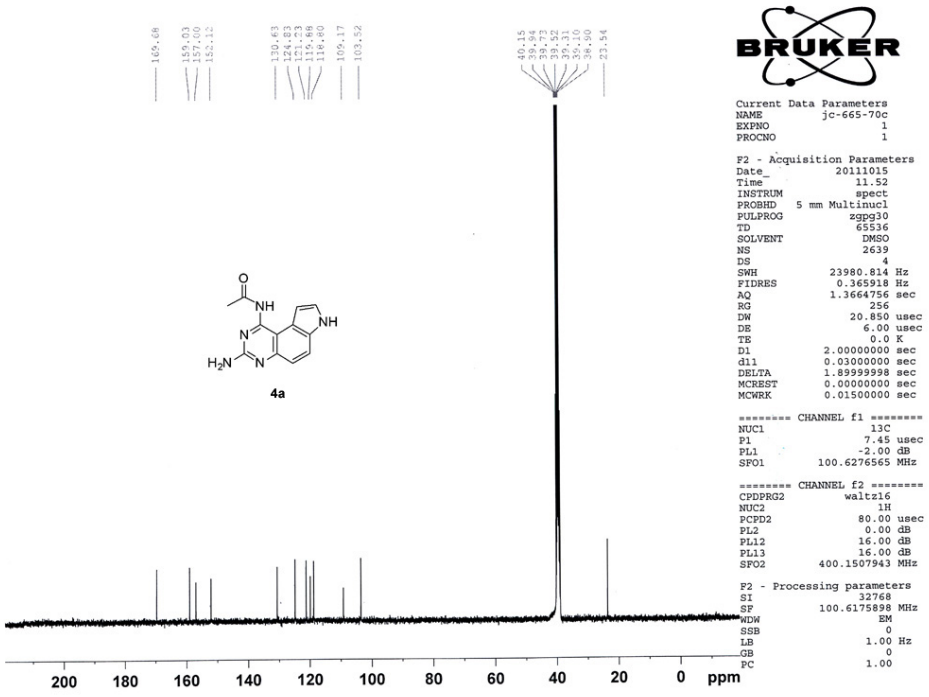

3.656
3.640
3.625
2.837
2.826
2.661
2.642
2.623
1.852
1.834
1.828
1.815
1.810
1.796
1.791
1.777
1.652
1.638
1.633
1.623
1.615
1.609
1.600
TBSO
3e
BRUKER
NAME jc-670-13
EXPNO 2
PROCNO 1
Date_ 20121228
Time_ 20.04
INSTRUM spect
PROBHD 5 mm BBO BB-1H
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 8278.146 Hz
FIDRES 0.126314 Hz
AQ 3.9584243 sec
RG 114
DW 60.400 usec
DE 6.50 usec
TE 292.6 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 15.00 usec
PL1 1.10 dB
SFO1 400.1524711 MHz
SI 32768
SF 400.1500087 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
9 8 7 6 5 4 3 2 1 ppm
2.00 3.99 1.99 1.99 2.36 9.11 5.90

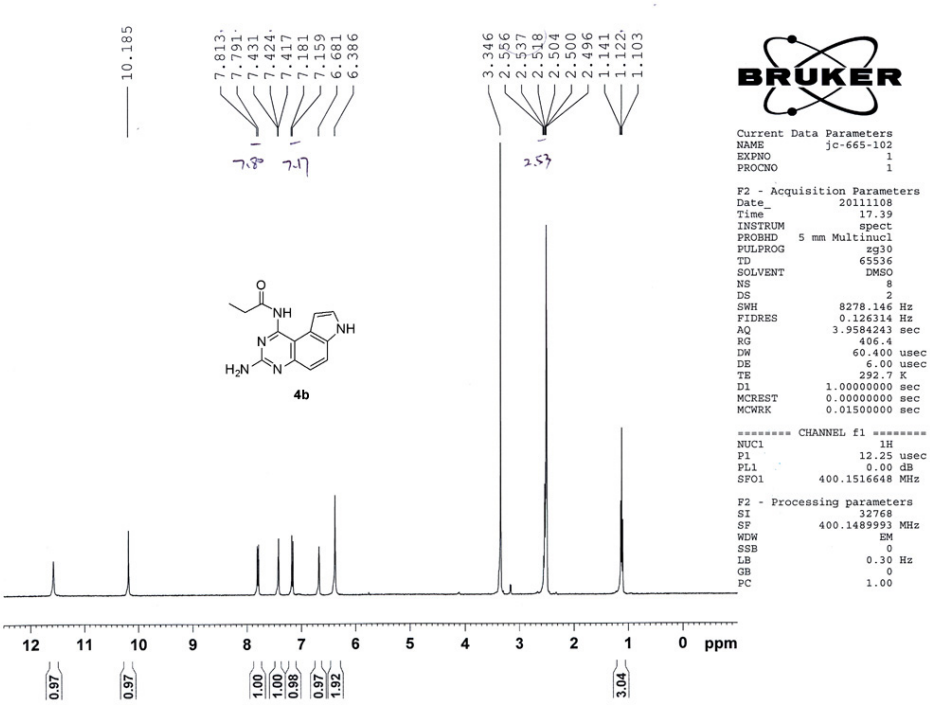

169.32
169.75
77.46
77.16
76.85
63.47
31.40
30.81
26.06
25.72
21.36
18.44
-5.22
TBSO
3e
BRUKER
NAME jc-670-13c
EXPNO 1
PROCNO 1
Date_ 20121228
Time_ 19.59
INSTRUM spect
PROBHD 5 mm BBO BB-1H
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 2244
DS 4
SWH 23980.814 Hz
FIDRES 0.365918 Hz
AQ 1.3664756 sec
RG 2580.3
DW 20.850 usec
DE 6.50 usec
TE 293.0 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 7.50 usec
PL1 -2.00 dB
SFO1 100.6278593 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 90.00 usec
PL2 1.10 dB
PL12 16.66 dB
PL13 18.00 dB
SFO2 400.1516006 MHz
SI 32768
SF 100.6177856 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
200 180 160 140 120 100 80 60 40 20 0 ppm

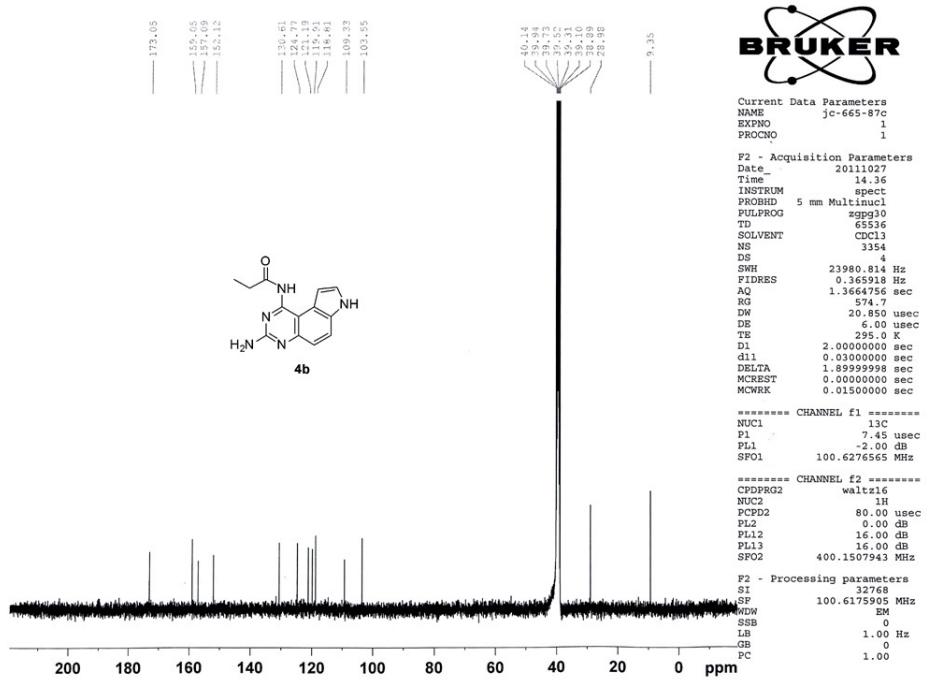

8.865
8.264
8.243
8.182
8.160
8.108
8.087
8.055
8.034
7.798
7.780
7.761
7.721
7.702
7.684
3.366
2.931
2.500
3f
9
8
7
6
5
4
3
2
1
ppm
1.00
1.04
1.03
1.03
1.03
1.03
1.02
3.99
BRUKER
NAME jc-670-85
EXPNO 1
PROCNO 1
Date_ 20121002
Time_ 15.01
INSTRUM spect
PROBHD 5 mm BBO BB-1H
PULPROG zg30
TD 65536
SOLVENT DMSO
NS 16
DS 2
SWH 8278.146 Hz
FIDRES 0.126314 Hz
AQ 3.9584243 sec
RG 181
DW 60.400 usec
DE 6.50 usec
TE 292.9 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 15.00 usec
PL1 1.10 dB
SFO1 400.1524711 MHz
SI 32768
SF 400.1500016 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

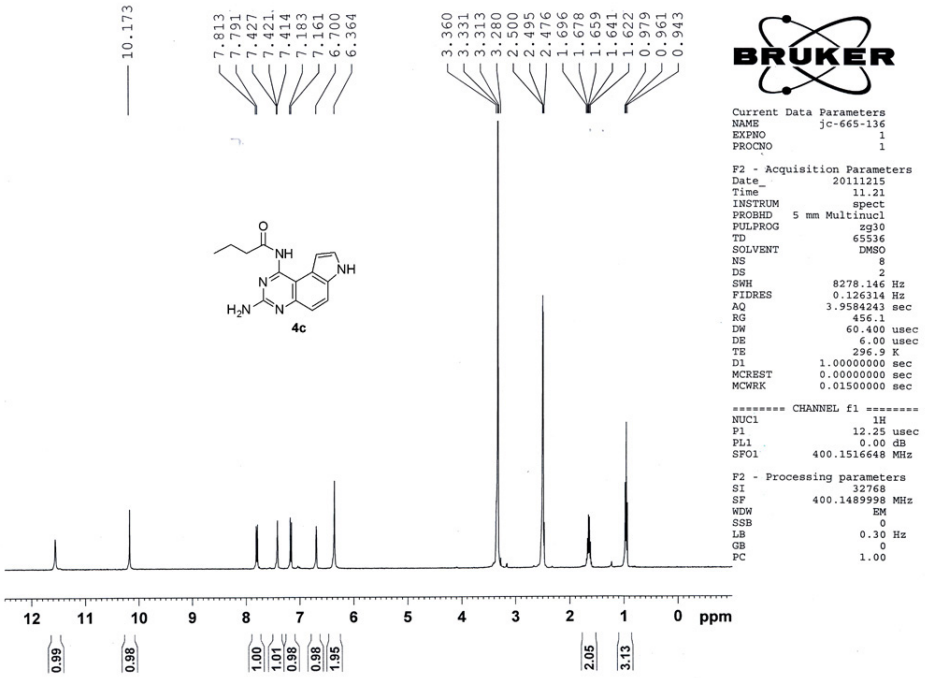

170.48
162.00
135.89
132.47
131.96
131.83
129.36
127.97
127.66
124.66
121.67
40.15
39.94
39.73
39.52
39.31
39.10
38.89
38.68
25.63
3f
200
180
160
140
120
100
80
60
40
20
ppm
BRUKER
NAME jc-670-85c
EXPNO 2
PROCNO 1
Date_ 20121228
Time_ 23.27
INSTRUM spect
PROBHD 5 mm BBO BB-1H
PULPROG zgpg30
TD 65536
SOLVENT DMSO
NS 3421
DS 4
SWH 23980.814 Hz
FIDRES 0.365918 Hz
AQ 1.3664756 sec
RG 2048
DW 20.850 usec
DE 6.50 usec
TE 293.3 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 7.50 usec
PL1 -2.00 dB
SFO1 100.6278593 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 90.00 usec
PL2 1.10 dB
PL12 16.66 dB
PL13 18.00 dB
SFO2 400.1516006 MHz
SI 32768
SF 100.6178390 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40


10.224
7.813
7.791
7.427
7.180
7.158
6.698
6.380
3.394
3.347
3.345
2.500
2.498
2.199
4a
12 11 10 9 8 7 6 5 4 3 2 1 0 ppm
1.00 0.98 1.00 1.01 1.00 0.98 1.95 3.00
BRUKER
Current Data Parameters
NAME jc-665-70
EXPNO 3
PROCNO 1
F2 - Acquisition Parameters
Date_ 20111014
Time 10.40
INSTRUM spect
PROBHD 5 mm Multinucl
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 8278.146 Hz
FIDRES 0.126314 Hz
AQ 3.9584243 sec
RG 362
DW 60.400 usec
DE 6.00 usec
TE 0.0 K
D1 1.00000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec
======== CHANNEL f1 ========
NUC1 1H
P1 12.25 usec
PL1 0.00 dB
SFO1 400.1516648 MHz
F2 - Processing parameters
SI 32768
SF 400.1490001 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00


165.68
159.03
157.00
153.12
130.63
124.83
122.73
117.68
116.80
109.17
103.52
40.15
39.94
39.73
39.52
39.31
39.10
38.90
23.54
4a
200 180 160 140 120 100 80 60 40 20 0 ppm
BRUKER
Current Data Parameters
NAME jc-665-70c
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20111015
Time 11.52
INSTRUM spect
PROBHD 5 mm Multinucl
PULPROG zgpg30
TD 65536
SOLVENT DMSO
NS 2639
DS 4
SWH 23980.814 Hz
FIDRES 0.365918 Hz
AQ 1.3664756 sec
RG 256
DW 20.850 usec
DE 6.00 usec
TE 0.0 K
D1 2.00000000 sec
d11 0.03000000 sec
DELTA 1.89999998 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec
======== CHANNEL f1 ========
NUC1 13C
P1 7.45 usec
PL1 -2.00 dB
SFO1 100.6276565 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 0.00 dB
PL12 16.00 dB
PL13 16.00 dB
SFO2 400.1507943 MHz
F2 - Processing parameters
SI 32768
SF 100.6175898 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.00


10.185
7.813
7.791
7.431
7.424
7.417
7.181
7.159
6.681
6.386
3.346
2.556
2.537
2.518
2.504
2.500
2.496
1.141
1.122
1.103
7.80
7.17
2.53
4b
12 11 10 9 8 7 6 5 4 3 2 1 0 ppm
0.97 0.97 1.00 1.00 0.98 0.97 1.92 3.04
BRUKER
Current Data Parameters
NAME jc-665-102
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20111108
Time 17.39
INSTRUM spect
PROBHD 5 mm Multinucl
PULPROG zg30
TD 65536
SOLVENT DMSO
NS 8
DS 2
SWH 8278.146 Hz
FIDRES 0.126314 Hz
AQ 3.9584243 sec
RG 406.4
DW 60.400 usec
DE 6.00 usec
TE 292.7 K
D1 1.00000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec
======== CHANNEL f1 ========
NUC1 1H
P1 12.25 usec
PL1 0.00 dB
SFO1 400.1516648 MHz
F2 - Processing parameters
SI 32768
SF 400.1489993 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00


173.05
159.05
157.09
152.12
130.61
124.79
121.19
119.51
116.61
109.39
103.55
40.14
39.94
39.73
39.52
39.31
39.10
38.89
28.98
9.35
4b
200 180 160 140 120 100 80 60 40 20 0 ppm
BRUKER
Current Data Parameters
NAME jc-665-87c
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20111027
Time 14.36
INSTRUM spect
PROBHD 5 mm Multinucl
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 3354
DS 4
SWH 23980.814 Hz
FIDRES 0.365918 Hz
AQ 1.3664756 sec
RG 574.7
DW 20.850 usec
DE 6.00 usec
TE 295.0 K
D1 2.00000000 sec
d11 0.03000000 sec
DELTA 1.89999998 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec
======== CHANNEL f1 ========
NUC1 13C
P1 7.45 usec
PL1 -2.00 dB
SFO1 100.6276565 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 0.00 dB
PL12 16.00 dB
PL13 16.00 dB
SFO2 400.1507943 MHz
F2 - Processing parameters
SI 32768
SF 100.6175905 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.00



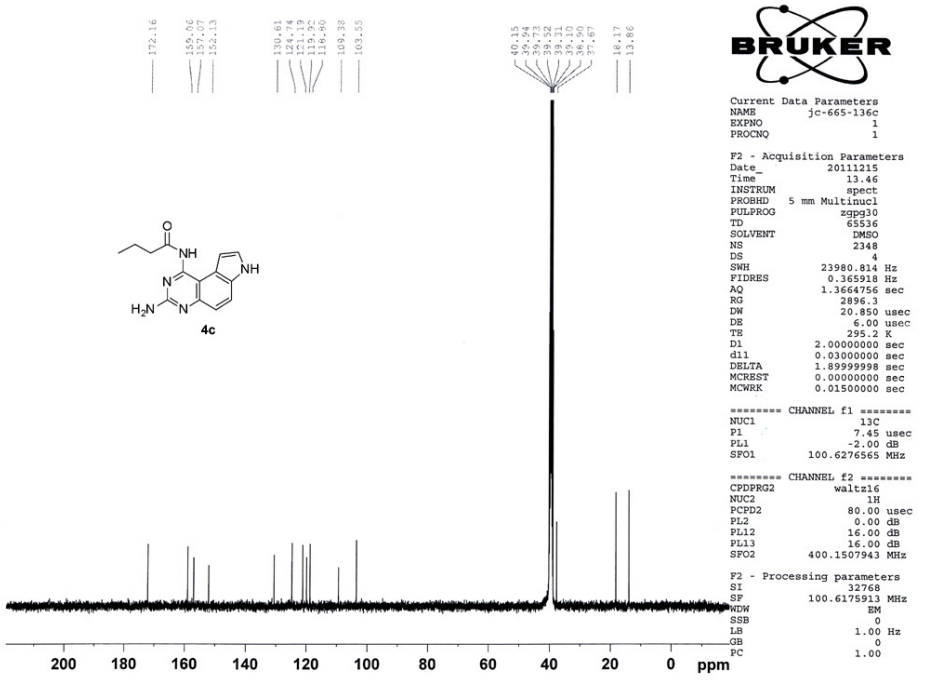
BRUKER

Current Data Parameters
NAME jc-665-136
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20111215
Time 11.21
INSTRUM spect
PROBHD 5 mm Multinucl
PULPROG zg30
TD 65536
SOLVENT DMSO
NS 8
DS 2
SWH 8278.146 Hz
FIDRES 0.126314 Hz
AQ 3.9584243 sec
RG 456.1
DW 60.400 usec
DE 6.00 usec
TE 296.9 K
D1 1.00000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec

======== CHANNEL f1 ========
NUC1 1H
P1 12.25 usec
PL1 0.00 dB
SFO1 400.1516648 MHz

F2 - Processing parameters
SI 32768
SF 400.1489998 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00



BRUKER

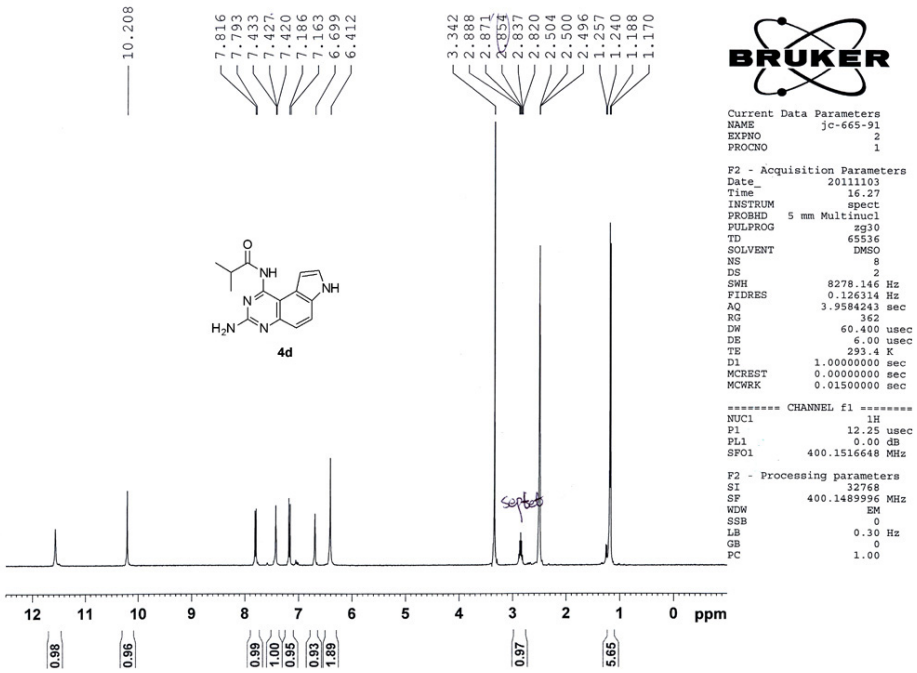
Current Data Parameters
NAME jc-665-136c
EXPNO 1
PROCNO 1

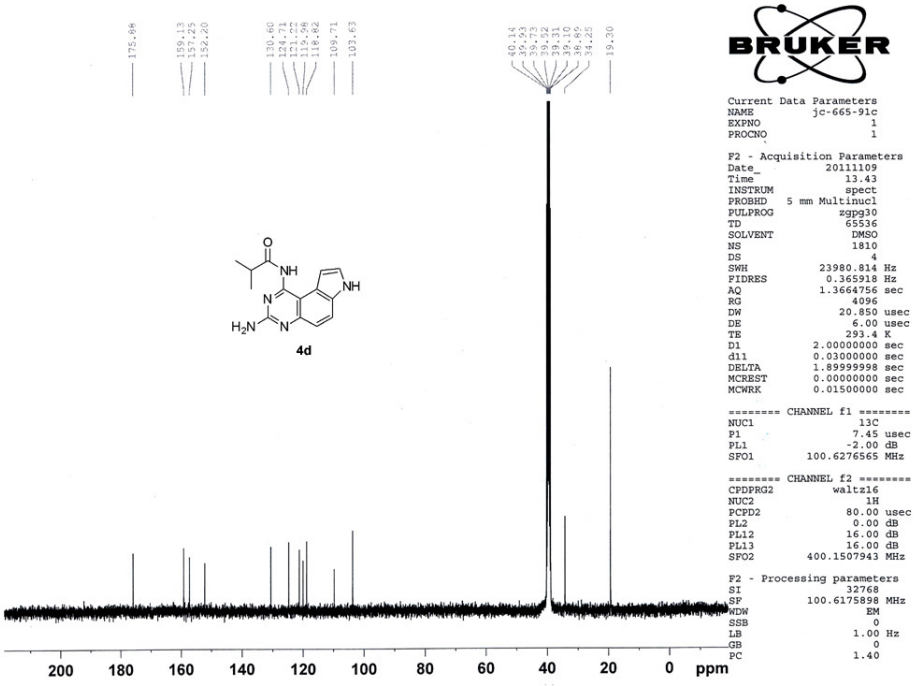
F2 - Acquisition Parameters
Date_ 20111215
Time 13.46
INSTRUM spect
PROBHD 5 mm Multinucl
PULPROG zgpg30
TD 65536
SOLVENT DMSO
NS 2348
DS 4
SWH 23980.814 Hz
FIDRES 0.365918 Hz
AQ 1.3664756 sec
RG 2896.3
DW 20.850 usec
DE 6.00 usec
TE 295.2 K
D1 2.00000000 sec
d11 0.03000000 sec
DELTA 1.89999998 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec

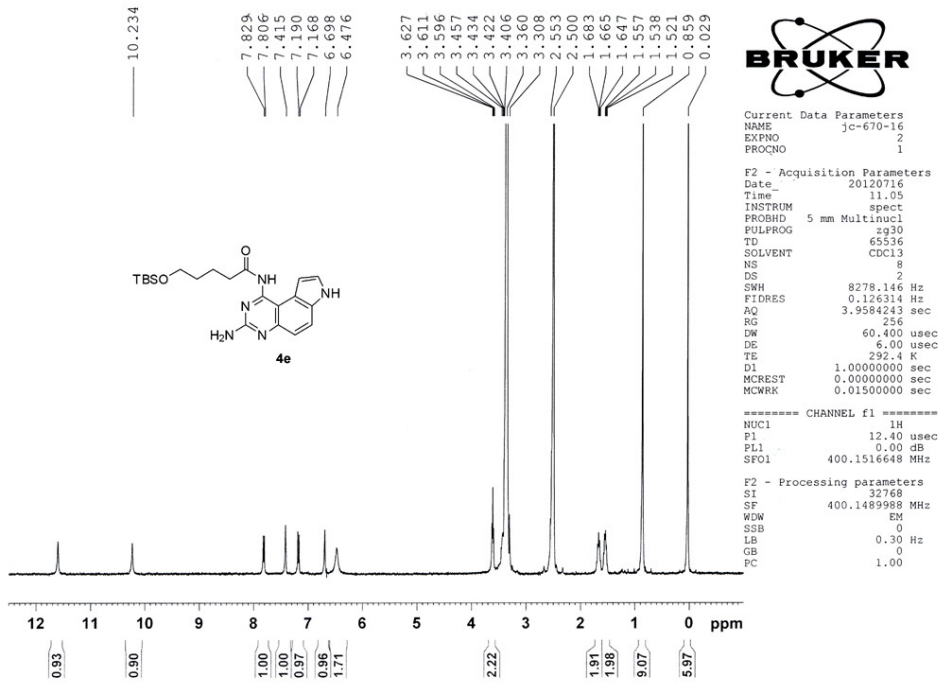
======== CHANNEL f1 ========
NUC1 13C
P1 7.45 usec
PL1 -2.00 dB
SFO1 100.6276565 MHz

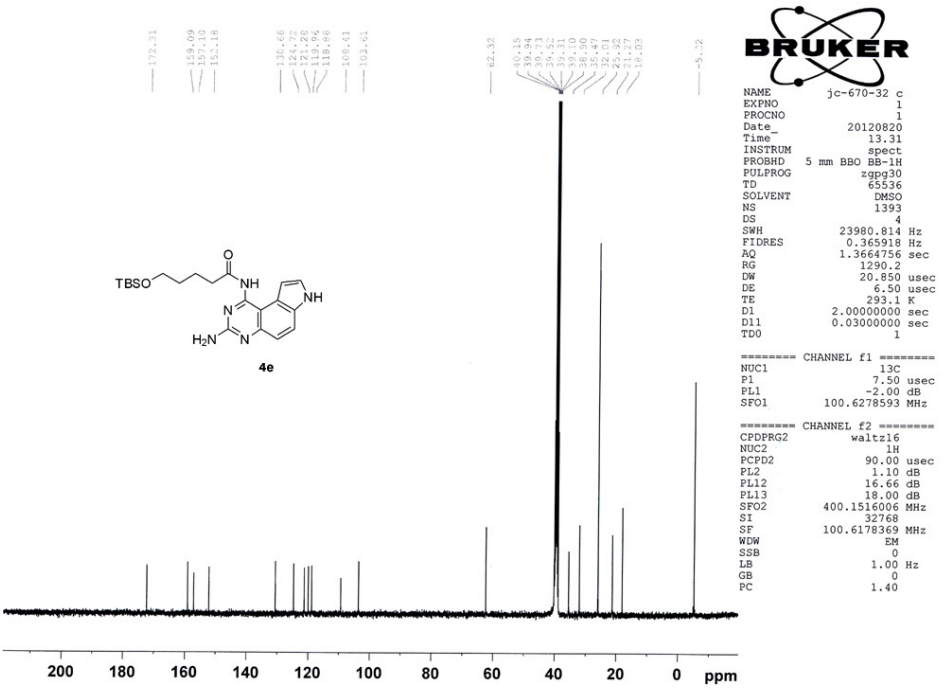
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 0.00 dB
PL12 16.00 dB
PL13 16.00 dB
SFO2 400.1507943 MHz

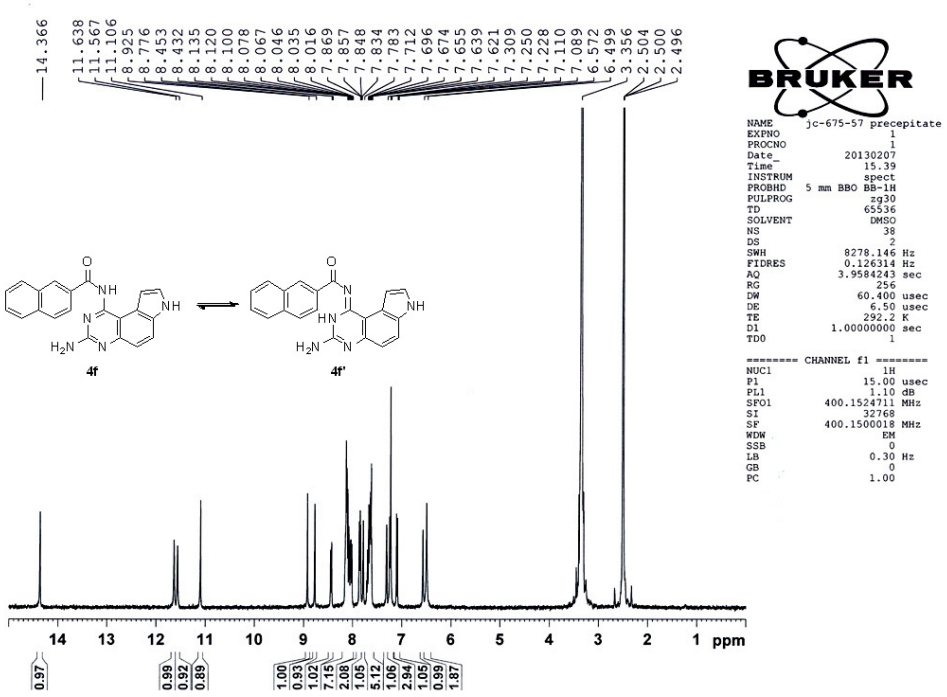
F2 - Processing parameters
SI 32768
SF 100.6175913 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.00

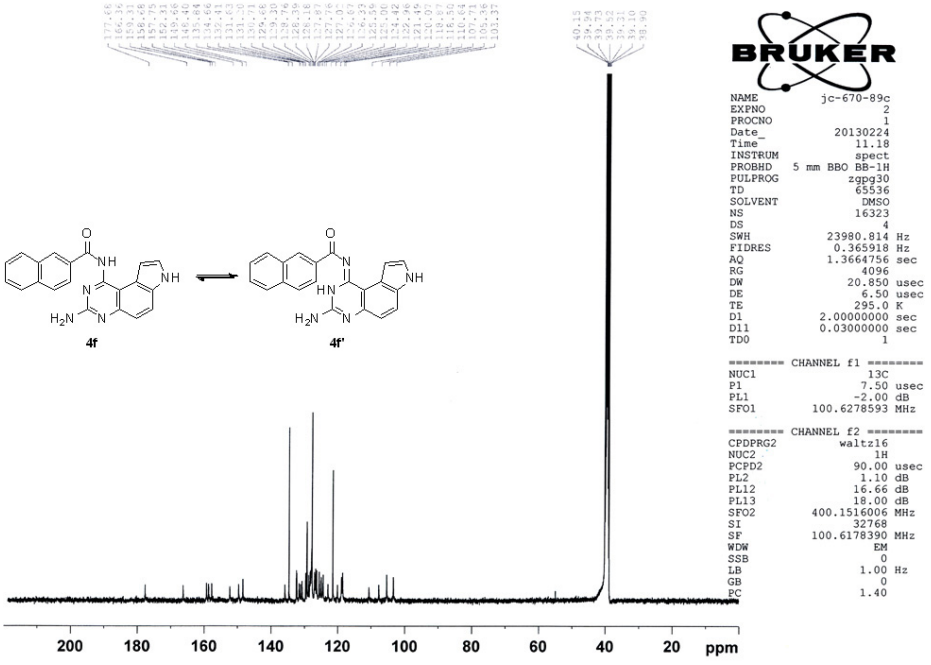

10.208
7.816
7.793
7.433
7.427
7.420
7.186
7.163
6.699
6.412
3.342
2.888
2.871
2.854
2.837
2.820
2.504
2.500
2.496
1.257
1.240
1.188
1.170
4d
12 11 10 9 8 7 6 5 4 3 2 1 0 ppm
0.98 0.96 0.99 1.00 0.95 0.93 1.89 0.97 5.65
BRUKER
Current Data Parameters
NAME jc-665-91
EXPNO 2
PROCNO 1
F2 - Acquisition Parameters
Date_ 20111103
Time 16.27
INSTRUM spect
PROBHD 5 mm Multinucl
PULPROG zg30
TD 65536
SOLVENT DMSO
NS 8
DS 2
SWH 8278.146 Hz
FIDRES 0.126314 Hz
AQ 3.9584243 sec
RG 362
DW 60.400 usec
DE 6.00 usec
TE 293.4 K
D1 1.00000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec
======== CHANNEL f1 ========
NUC1 1H
P1 12.25 usec
PL1 0.00 dB
SFO1 400.1516648 MHz
F2 - Processing parameters
SI 32768
SF 400.1489996 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

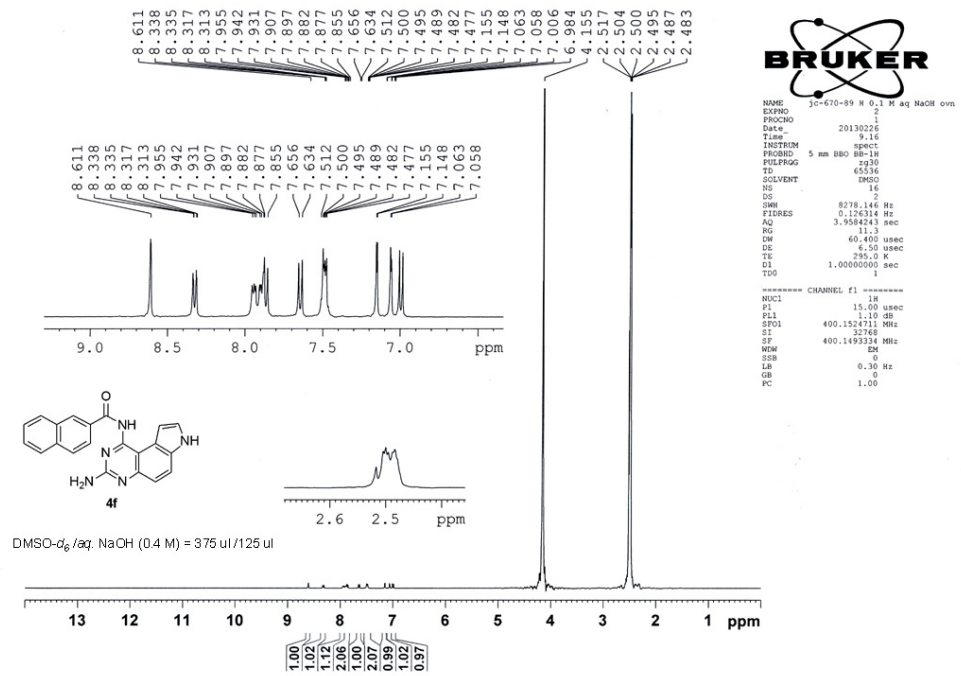

175.86
159.13
157.25
152.20
130.60
124.71
121.22
115.96
118.82
109.71
103.63
40.14
39.93
39.73
39.52
39.31
39.10
36.80
34.25
19.30
4d
200 180 160 140 120 100 80 60 40 20 0 ppm
BRUKER
Current Data Parameters
NAME jc-665-91c
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20111109
Time 13.43
INSTRUM spect
PROBHD 5 mm Multinucl
PULPROG zgpg30
TD 65536
SOLVENT DMSO
NS 1810
DS 4
SWH 23980.814 Hz
FIDRES 0.365918 Hz
AQ 1.3664756 sec
RG 4096
DW 20.850 usec
DE 6.00 usec
TE 293.4 K
D1 2.00000000 sec
d11 0.03000000 sec
DELTA 1.89999998 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec
======== CHANNEL f1 ========
NUC1 13C
P1 7.45 usec
PL1 -2.00 dB
SFO1 100.6276565 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 0.00 dB
PL12 16.00 dB
PL13 16.00 dB
SFO2 400.1507943 MHz
F2 - Processing parameters
SI 32768
SF 100.6175898 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

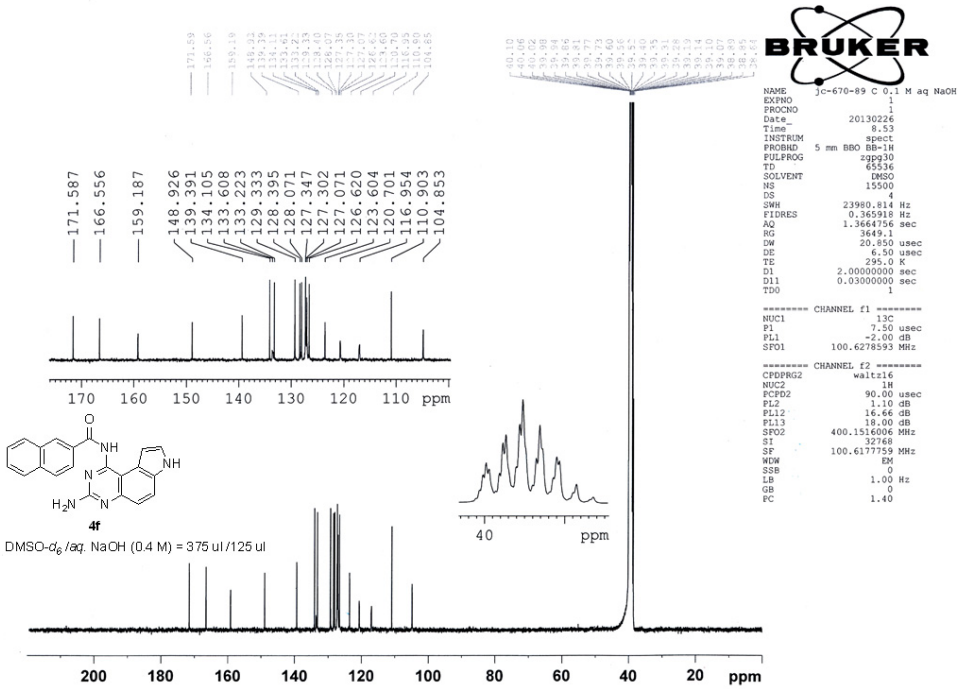

10.234
7.829
7.806
7.415
7.190
7.168
6.698
6.476
3.627
3.611
3.596
3.457
3.434
3.422
3.406
3.360
3.308
2.553
2.500
1.683
1.665
1.647
1.557
1.538
1.521
0.859
0.029
TBSO
NH
NH
N
N
H2N
4e
12 11 10 9 8 7 6 5 4 3 2 1 0 ppm
0.93
0.90
1.00
1.00
0.97
0.96
1.71
2.22
1.91
1.98
9.07
5.97
BRUKER
Current Data Parameters
NAME jc-670-16
EXPNO 2
PROCNO 1
F2 - Acquisition Parameters
Date_ 20120716
Time 11.05
INSTRUM spect
PROBHD 5 mm Multinucl
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 8
DS 2
SWH 8278.146 Hz
FIDRES 0.126314 Hz
AQ 3.9584243 sec
RG 256
DW 60.400 usec
DE 6.00 usec
TE 292.4 K
D1 1.00000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec
======== CHANNEL f1 ========
NUC1 1H
P1 12.40 usec
PL1 0.00 dB
SFO1 400.1516648 MHz
F2 - Processing parameters
SI 32768
SF 400.1489988 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

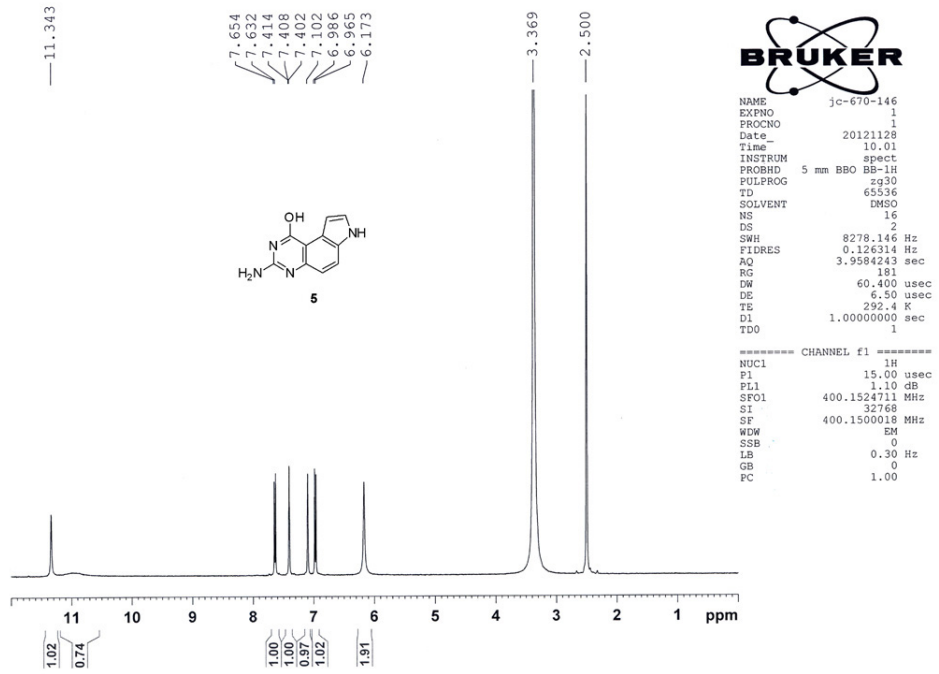

172.31
159.09
157.10
152.18
130.66
124.72
121.26
119.96
118.66
109.41
103.61
62.32
40.15
39.94
39.73
39.52
39.31
39.10
38.90
35.47
32.01
25.82
21.27
18.03
-5.32
TBSO
NH
NH
N
N
H2N
4e
200 180 160 140 120 100 80 60 40 20 0 ppm
BRUKER
NAME jc-670-32 c
EXPNO 1
PROCNO 1
Date_ 20120820
Time 13.31
INSTRUM spect
PROBHD 5 mm BBO BB-1H
PULPROG zgpg30
TD 65536
SOLVENT DMSO
NS 1393
DS 4
SWH 23980.814 Hz
FIDRES 0.365918 Hz
AQ 1.3664756 sec
RG 1290.2
DW 20.850 usec
DE 6.50 usec
TE 293.1 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 7.50 usec
PL1 -2.00 dB
SFO1 100.6278593 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 90.00 usec
PL2 1.10 dB
PL12 16.66 dB
PL13 18.00 dB
SFO2 400.1516006 MHz
SI 32768
SF 100.6178369 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

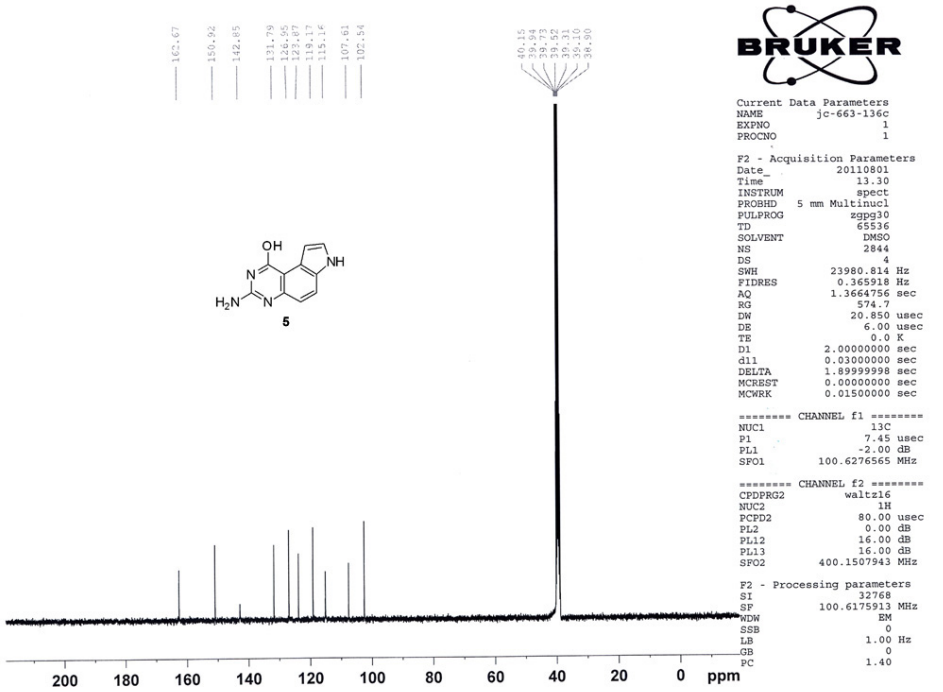

14.366
11.638
11.567
11.106
8.925
8.776
8.453
8.432
8.135
8.120
8.100
8.078
8.067
8.046
8.035
8.016
7.869
7.857
7.848
7.834
7.783
7.712
7.696
7.674
7.655
7.639
7.621
7.309
7.250
7.228
7.110
7.089
6.572
6.499
3.356
2.504
2.500
2.496
4f
4f'
BRUKER
NAME jc-675-57 precepitate
EXPNO 1
PROCNO 1
Date_ 20130207
Time 15.39
INSTRUM spect
PROBHD 5 mm BBO BB-1H
PULPROG zg30
TD 65536
SOLVENT DMSO
NS 38
DS 2
SWH 8278.146 Hz
FIDRES 0.126314 Hz
AQ 3.9584243 sec
RG 256
DW 60.400 usec
DE 6.50 usec
TE 292.2 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 15.00 usec
PL1 1.10 dB
SFO1 400.1524711 MHz
SI 32768
SF 400.1500018 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
14 13 12 11 10 9 8 7 6 5 4 3 2 1 ppm
0.97 0.99 0.92 0.89 1.00 0.93 1.02 7.15 2.08 1.05 5.12 1.06 2.94 1.05 0.99 1.87

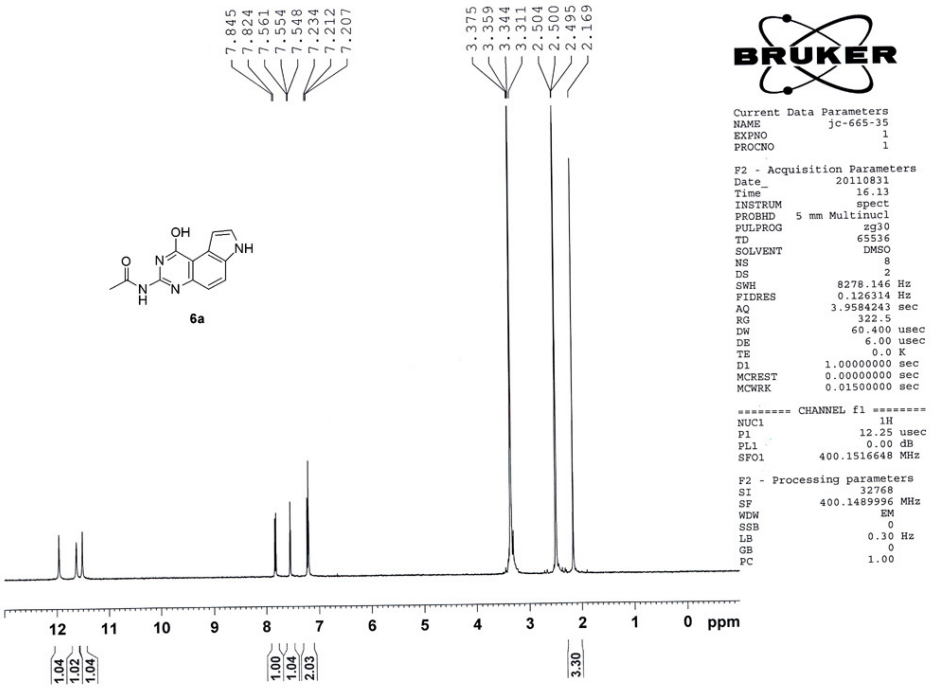

4f
4f'
BRUKER
NAME jc-670-89c
EXPNO 2
PROCNO 1
Date_ 20130224
Time 11.18
INSTRUM spect
PROBHD 5 mm BBO BB-1H
PULPROG zgpg30
TD 65536
SOLVENT DMSO
NS 16323
DS 4
SWH 23980.814 Hz
FIDRES 0.365918 Hz
AQ 1.3664756 sec
RG 4096
DW 20.850 usec
DE 6.50 usec
TE 295.0 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 7.50 usec
PL1 -2.00 dB
SFO1 100.6278593 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 90.00 usec
PL2 1.10 dB
PL12 16.66 dB
PL13 18.00 dB
SFO2 400.1516006 MHz
SI 32768
SF 100.6178390 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
200 180 160 140 120 100 80 60 40 20 ppm

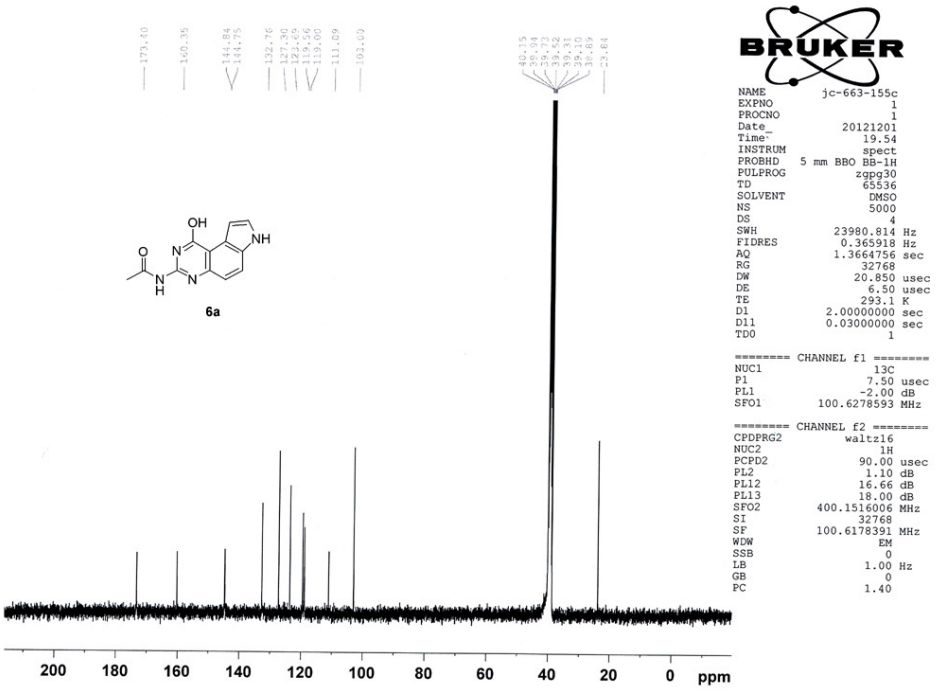

8.611
8.338
8.335
8.317
8.313
7.955
7.942
7.931
7.907
7.897
7.882
7.877
7.855
7.656
7.634
7.512
7.500
7.495
7.489
7.482
7.477
7.155
7.148
7.063
7.058
7.006
6.984
4.155
2.517
2.504
2.500
2.495
2.487
2.483
BRUKER
NAME jc-670-89 H 0.1 M aq NaOH ovn
EXPNO 2
PROCNO 1
Date_ 20130226
Time_ 9.16
INSTRUM spect
PROBHD 5 mm BBO BB-1H
PULPROG zg30
TD 65536
SOLVENT DMSO
NS 16
DS 2
SWH 8278.146 Hz
FIDRES 0.126314 Hz
AQ 3.9584243 sec
RG 11.3
DW 60.400 usec
DE 6.50 usec
TE 295.0 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 15.00 usec
PL1 1.10 dB
SFO1 400.1524711 MHz
SI 32768
SF 400.1493334 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
9.0 8.5 8.0 7.5 7.0 ppm
2.6 2.5 ppm
4f
DMSO-d6 /aq. NaOH (0.4 M) = 375 ul /125 ul
13 12 11 10 9 8 7 6 5 4 3 2 1 ppm
1.00 1.02 1.12 2.06 1.00 2.07 0.99 1.02 0.97

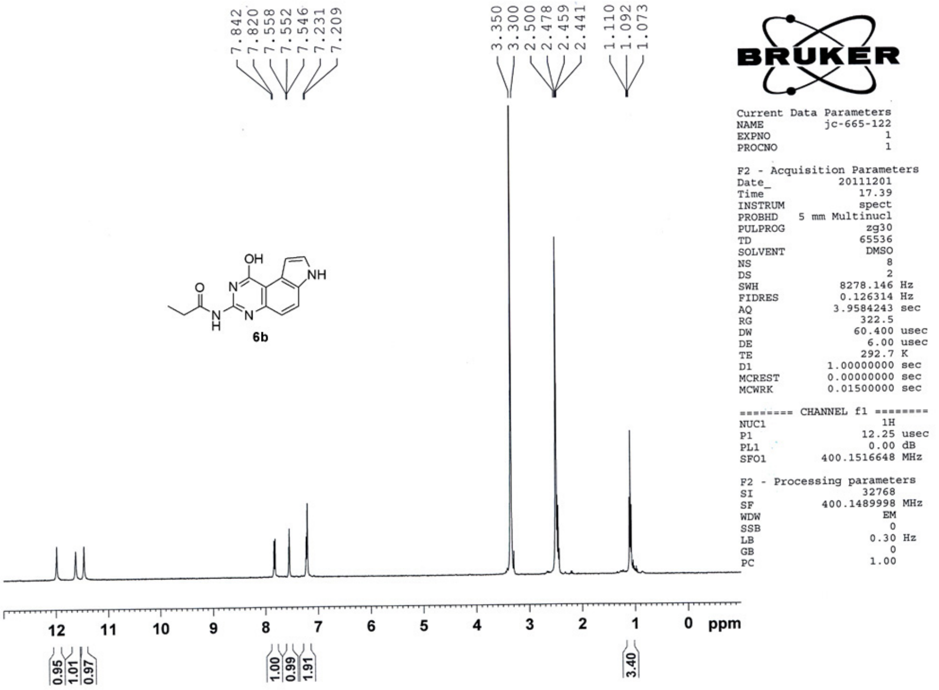

171.587
166.556
159.187
148.926
139.391
134.105
133.608
133.223
129.333
128.395
128.071
127.347
127.302
127.071
126.620
123.604
120.701
116.954
110.903
104.853
BRUKER
NAME jc-670-89 C 0.1 M aq NaOH
EXPNO 1
PROCNO 1
Date_ 20130226
Time_ 9.53
INSTRUM spect
PROBHD 5 mm BBO BB-1H
PULPROG zgpg30
TD 65536
SOLVENT DMSO
NS 15500
DS 4
SWH 23980.814 Hz
FIDRES 0.365918 Hz
AQ 1.3664756 sec
RG 3649.1
DW 20.850 usec
DE 6.50 usec
TE 295.0 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 7.50 usec
PL1 -2.00 dB
SFO1 100.6278593 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 90.00 usec
PL2 1.10 dB
PL12 16.66 dB
PL13 18.00 dB
SFO2 400.1516006 MHz
SI 32768
SF 100.6177759 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
170 160 150 140 130 120 110 ppm
40 ppm
4f
DMSO-d6 /aq. NaOH (0.4 M) = 375 ul /125 ul
200 180 160 140 120 100 80 60 40 20 ppm

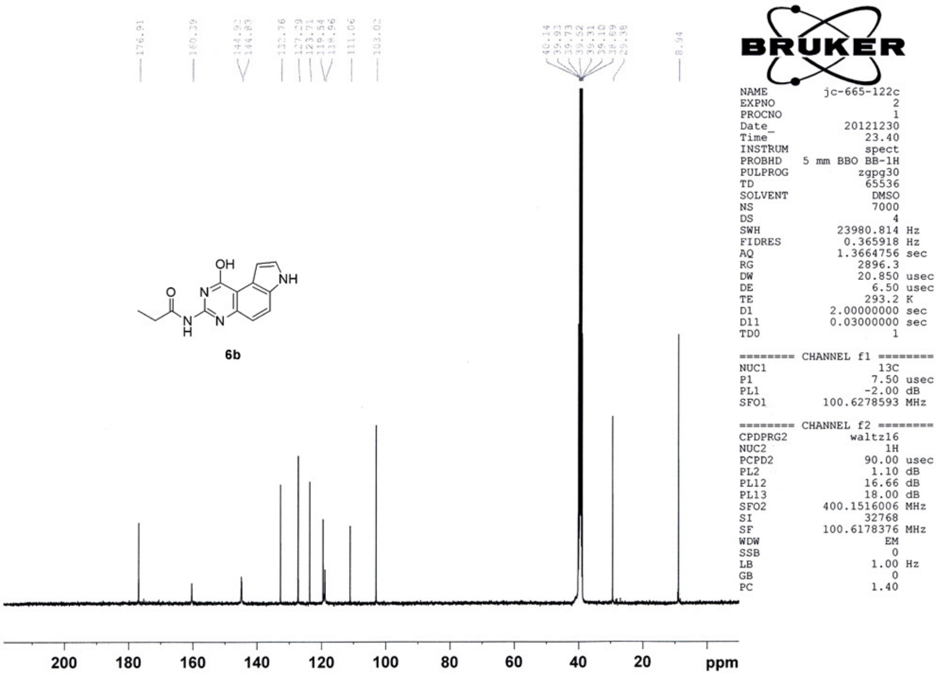

11.343
7.654
7.632
7.414
7.408
7.402
7.102
6.986
6.965
6.173
3.369
2.500
OH
NH
N
N
H_2N
5
11
10
9
8
7
6
5
4
3
2
1
ppm
1.02
0.74
1.00
1.00
0.97
1.02
1.91
BRUKER
NAME jc-670-146
EXPNO 1
PROCNO 1
Date_ 20121128
Time_ 10.01
INSTRUM spect
PROBHD 5 mm BBO BB-1H
PULPROG zg30
TD 65536
SOLVENT DMSO
NS 16
DS 2
SWH 8278.146 Hz
FIDRES 0.126314 Hz
AQ 3.9584243 sec
RG 181
DW 60.400 usec
DE 6.50 usec
TE 292.4 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 15.00 usec
PL1 1.10 dB
SFO1 400.1524711 MHz
SI 32768
SF 400.1500018 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

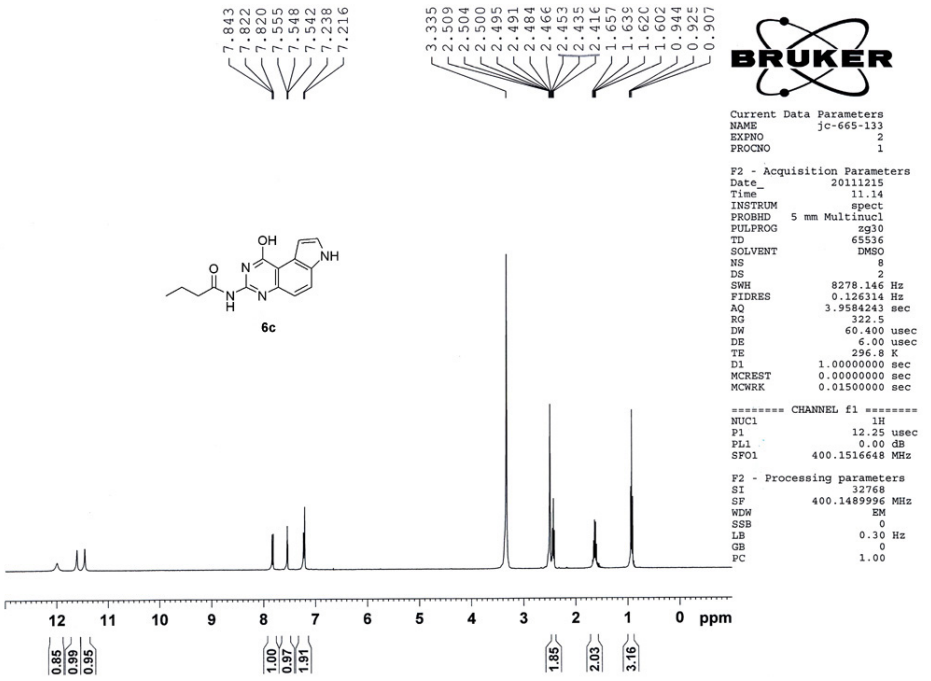

162.67
150.92
142.85
131.79
126.95
123.67
119.17
115.14
107.61
102.54
40.15
39.94
39.73
39.52
39.31
39.10
38.90
OH
NH
N
N
H_2N
5
200
180
160
140
120
100
80
60
40
20
0
ppm
BRUKER
Current Data Parameters
NAME jc-663-136c
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20110801
Time 13.30
INSTRUM spect
PROBHD 5 mm Multinucl
PULPROG zgpg30
TD 65536
SOLVENT DMSO
NS 2844
DS 4
SWH 23980.814 Hz
FIDRES 0.365918 Hz
AQ 1.3664756 sec
RG 574.7
DW 20.850 usec
DE 6.00 usec
TE 0.0 K
D1 2.00000000 sec
d11 0.03000000 sec
DELTA 1.89999998 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec
======== CHANNEL f1 ========
NUC1 13C
P1 7.45 usec
PL1 -2.00 dB
SFO1 100.6276565 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 0.00 dB
PL12 16.00 dB
PL13 16.00 dB
SFO2 400.1507943 MHz
F2 - Processing parameters
SI 32768
SF 100.6175913 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

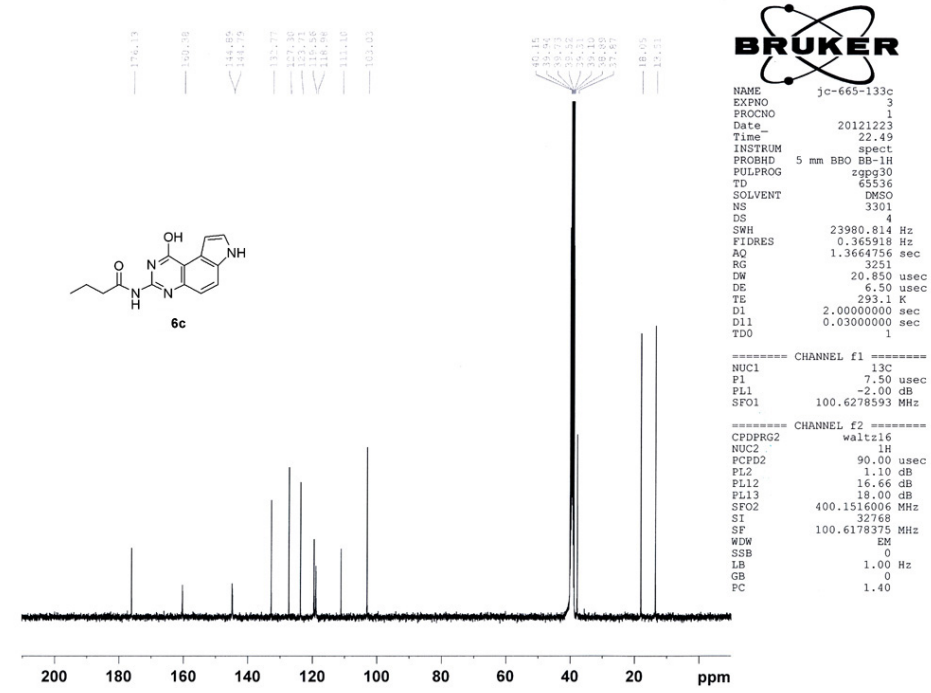

7.845
7.824
7.561
7.554
7.548
7.234
7.212
7.207
3.375
3.359
3.344
3.311
2.504
2.500
2.495
2.169
OH
NH
O
N
N
H
6a
12
11
10
9
8
7
6
5
4
3
2
1
0
ppm
1.04
1.02
1.04
1.00
1.04
2.03
3.30
BRUKER
Current Data Parameters
NAME jc-665-35
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20110831
Time 16.13
INSTRUM spect
PROBHD 5 mm Multinucl
PULPROG zg30
TD 65536
SOLVENT DMSO
NS 8
DS 2
SWH 8278.146 Hz
FIDRES 0.126314 Hz
AQ 3.9584243 sec
RG 322.5
DW 60.400 usec
DE 6.00 usec
TE 0.0 K
D1 1.00000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec
======== CHANNEL f1 ========
NUC1 1H
P1 12.25 usec
PL1 0.00 dB
SFO1 400.1516648 MHz
F2 - Processing parameters
SI 32768
SF 400.1489996 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

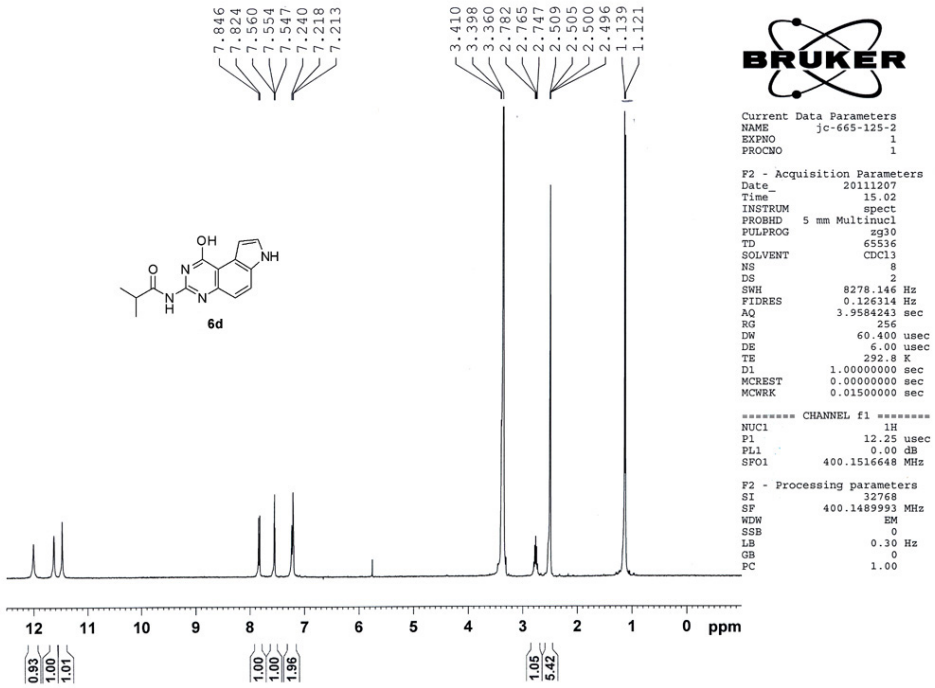

173.40
160.35
144.84
144.75
132.76
127.30
123.69
119.56
119.00
111.09
103.03
40.15
39.94
39.73
39.52
39.31
39.10
38.89
23.64
OH
NH
O
N
N
H
6a
200
180
160
140
120
100
80
60
40
20
0
ppm
BRUKER
NAME jc-663-155c
EXPNO 1
PROCNO 1
Date_ 20121201
Time 19.54
INSTRUM spect
PROBHD 5 mm BBO BB-1H
PULPROG zgpg30
TD 65536
SOLVENT DMSO
NS 5000
DS 4
SWH 23980.814 Hz
FIDRES 0.365918 Hz
AQ 1.3664756 sec
RG 32768
DW 20.850 usec
DE 6.50 usec
TE 293.1 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 7.50 usec
PL1 -2.00 dB
SFO1 100.6278593 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 90.00 usec
PL2 1.10 dB
PL12 16.66 dB
PL13 18.00 dB
SFO2 400.1516006 MHz
SI 32768
SF 100.6178391 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40


7.842
7.820
7.558
7.552
7.546
7.231
7.209
3.350
3.300
2.500
2.478
2.459
2.441
1.110
1.092
1.073
BRUKER
Current Data Parameters
NAME jc-665-122
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20111201
Time 17.39
INSTRUM spect
PROBHD 5 mm Multinucl
PULPROG zg30
TD 65536
SOLVENT DMSO
NS 8
DS 2
SWH 8278.146 Hz
FIDRES 0.126314 Hz
AQ 3.9584243 sec
RG 322.5
DW 60.400 usec
DE 6.00 usec
TE 292.7 K
D1 1.00000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec
======== CHANNEL f1 ========
NUC1 1H
P1 12.25 usec
PL1 0.00 dB
SFO1 400.1516648 MHz
F2 - Processing parameters
SI 32768
SF 400.1489998 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
OH
NH
O
N
N
H
6b
12 11 10 9 8 7 6 5 4 3 2 1 0 ppm
0.95
1.01
0.97
1.00
0.99
1.91
3.40


BRUKER
NAME jc-665-122c
EXPNO 2
PROCNO 1
Date_ 20121230
Time 23.40
INSTRUM spect
PROBHD 5 mm BBO BB-1H
PULPROG zgpg30
TD 65536
SOLVENT DMSO
NS 7000
DS 4
SWH 23980.814 Hz
FIDRES 0.365918 Hz
AQ 1.3664756 sec
RG 2896.3
DW 20.850 usec
DE 6.50 usec
TE 293.2 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 7.50 usec
PL1 -2.00 dB
SFO1 100.6278593 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 90.00 usec
PL2 1.10 dB
PL12 16.66 dB
PL13 18.00 dB
SFO2 400.1516006 MHz
SI 32768
SF 100.6178376 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
OH
NH
O
N
N
H
6b
200 180 160 140 120 100 80 60 40 20 ppm


7.843
7.822
7.820
7.555
7.548
7.542
7.238
7.216
3.335
2.509
2.504
2.500
2.495
2.491
2.484
2.466
2.453
2.435
2.416
1.657
1.639
1.620
1.602
0.944
0.925
0.907
OH
NH
O
N
N
N
H
6c
12
11
10
9
8
7
6
5
4
3
2
1
0
ppm
0.85
0.99
0.95
1.00
0.97
1.91
1.85
2.03
3.16
BRUKER
Current Data Parameters
NAME jc-665-133
EXPNO 2
PROCNO 1
F2 - Acquisition Parameters
Date_ 20111215
Time 11.14
INSTRUM spect
PROBHD 5 mm Multinucl
PULPROG zg30
TD 65536
SOLVENT DMSO
NS 8
DS 2
SWH 8278.146 Hz
FIDRES 0.126314 Hz
AQ 3.9584243 sec
RG 322.5
DW 60.400 usec
DE 6.00 usec
TE 296.8 K
D1 1.00000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec
======== CHANNEL f1 ========
NUC1 1H
P1 12.25 usec
PL1 0.00 dB
SFO1 400.1516648 MHz
F2 - Processing parameters
SI 32768
SF 400.1489996 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00


176.13
160.36
144.85
144.79
131.77
127.30
123.71
119.86
116.86
111.10
103.03
40.15
35.44
30.73
26.02
21.31
18.60
15.51
OH
NH
O
N
N
N
H
6c
200
180
160
140
120
100
80
60
40
20
ppm
BRUKER
NAME jc-665-133c
EXPNO 3
PROCNO 1
Date_ 20121223
Time 22.49
INSTRUM spect
PROBHD 5 mm BBO BB-1H
PULPROG zgpg30
TD 65536
SOLVENT DMSO
NS 3301
DS 4
SWH 23980.814 Hz
FIDRES 0.365918 Hz
AQ 1.3664756 sec
RG 3251
DW 20.850 usec
DE 6.50 usec
TE 293.1 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 7.50 usec
PL1 -2.00 dB
SFO1 100.6278593 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 90.00 usec
PL2 1.10 dB
PL12 16.66 dB
PL13 18.00 dB
SFO2 400.1516006 MHz
SI 32768
SF 100.6178375 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40



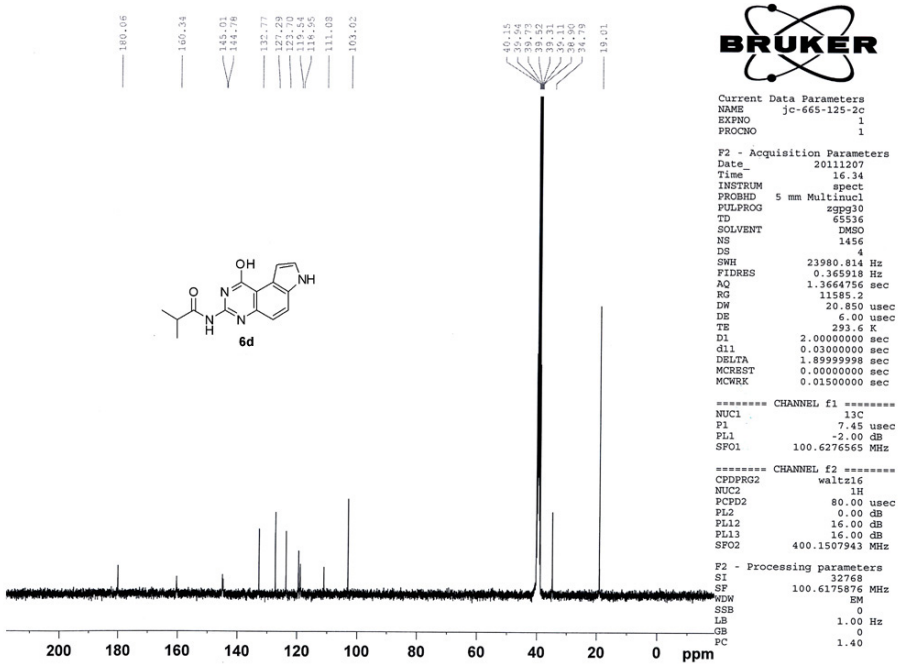
BRUKER

Current Data Parameters
NAME jc-665-125-2
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20111207
Time 15.02
INSTRUM spect
PROBHD 5 mm Multinucl
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 8
DS 2
SWH 8278.146 Hz
FIDRES 0.126314 Hz
AQ 3.9584243 sec
RG 256
DW 60.400 usec
DE 6.00 usec
TE 292.8 K
D1 1.00000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec

======== CHANNEL f1 ========
NUC1 1H
P1 12.25 usec
PL1 0.00 dB
SFO1 400.1516648 MHz

F2 - Processing parameters
SI 32768
SF 400.1489993 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00



BRUKER

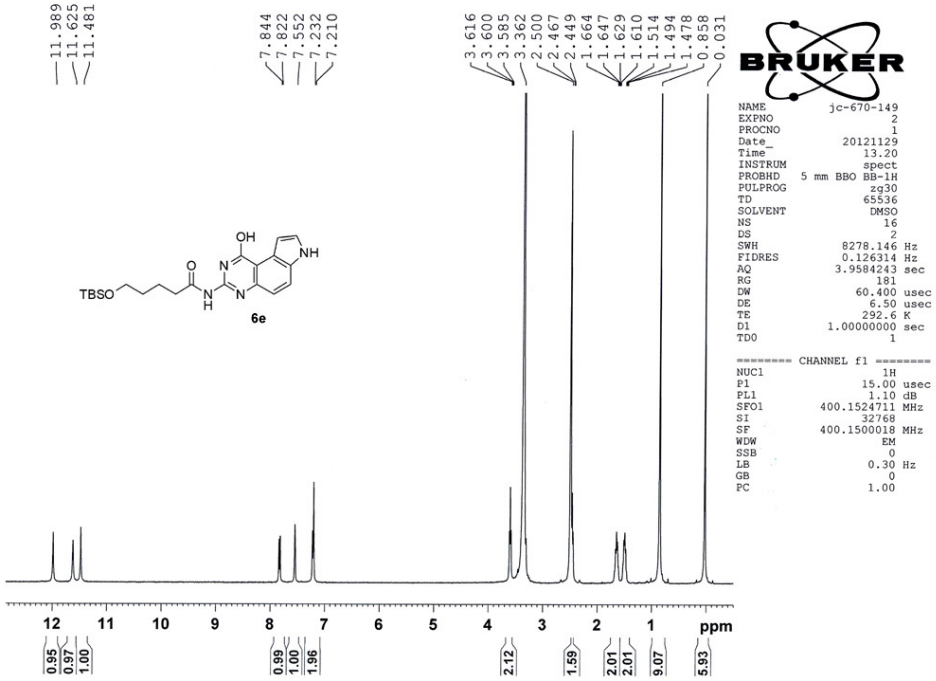
Current Data Parameters
NAME jc-665-125-2c
EXPNO 1
PROCNO 1

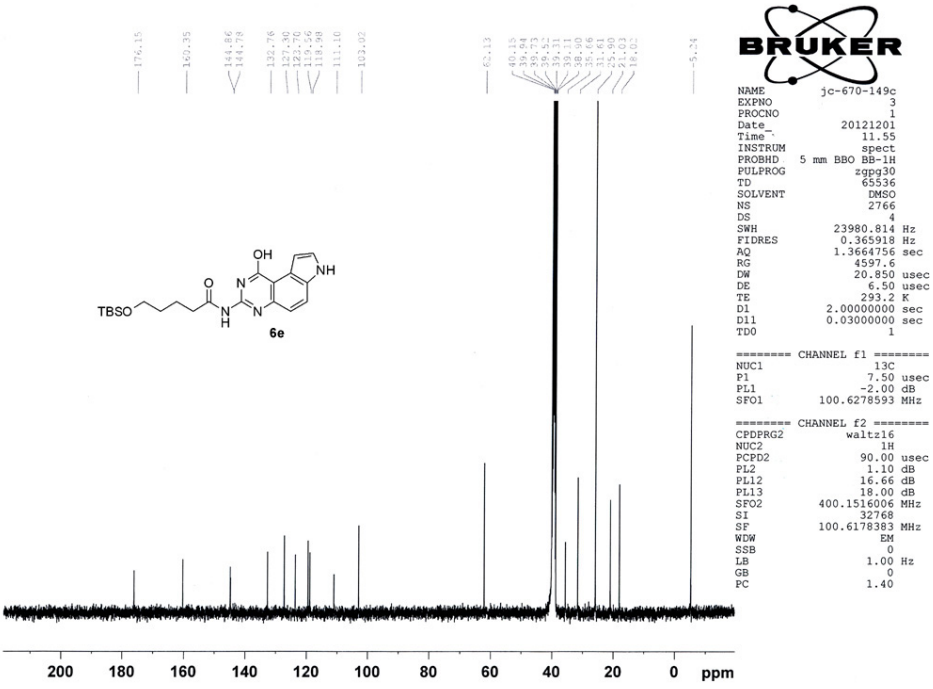
F2 - Acquisition Parameters
Date_ 20111207
Time 16.34
INSTRUM spect
PROBHD 5 mm Multinucl
PULPROG zgpg30
TD 65536
SOLVENT DMSO
NS 1456
DS 4
SWH 23980.814 Hz
FIDRES 0.365918 Hz
AQ 1.3664756 sec
RG 11585.2
DW 20.850 usec
DE 6.00 usec
TE 293.6 K
D1 2.00000000 sec
d11 0.03000000 sec
DELTA 1.89999998 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec

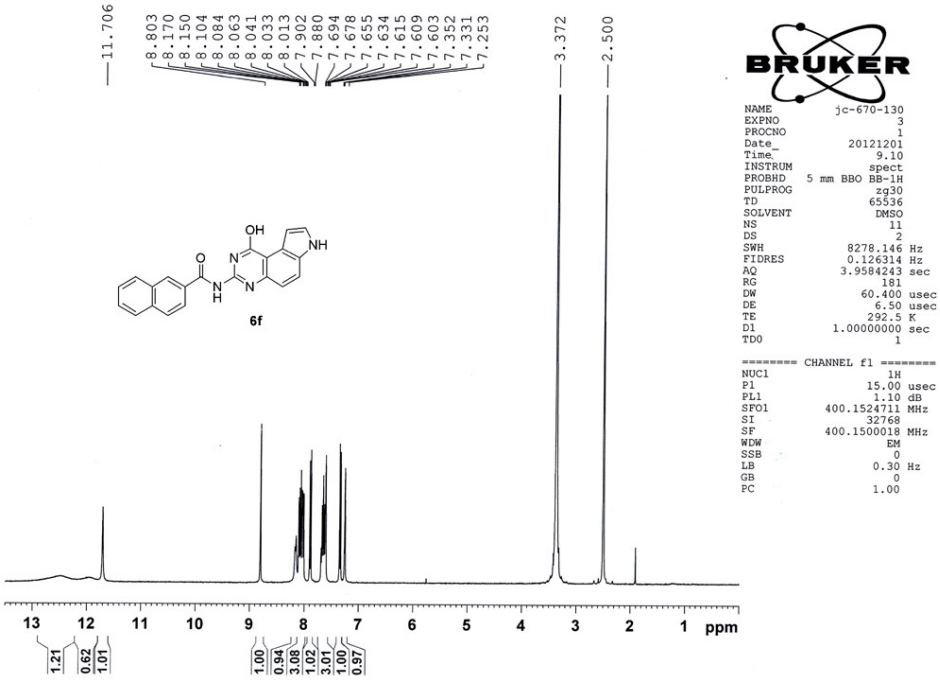
======== CHANNEL f1 ========
NUC1 13C
P1 7.45 usec
PL1 -2.00 dB
SFO1 100.6276565 MHz

======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 0.00 dB
PL12 16.00 dB
PL13 16.00 dB
SFO2 400.1507943 MHz

F2 - Processing parameters
SI 32768
SF 100.6175876 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

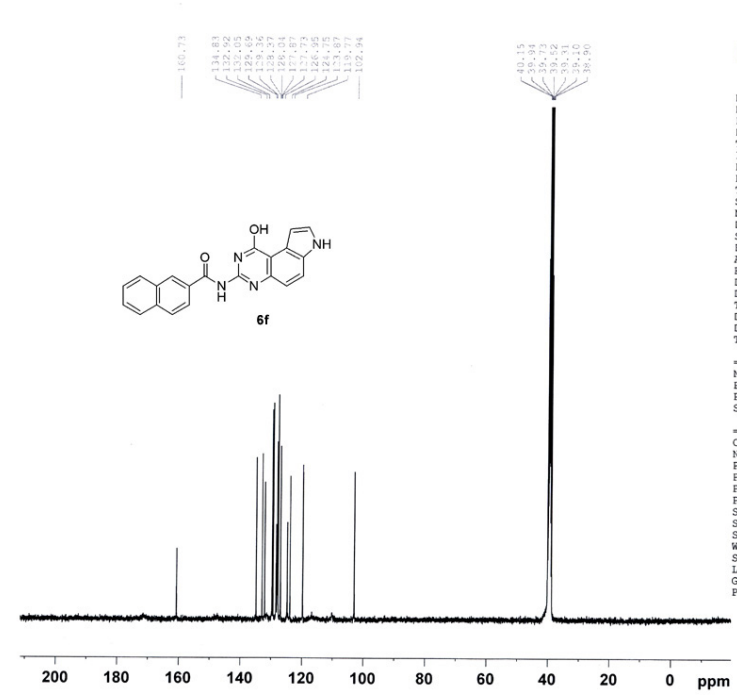

11.989
11.625
11.481
7.844
7.822
7.552
7.232
7.210
3.616
3.600
3.585
3.362
2.500
2.467
2.449
1.664
1.647
1.629
1.610
1.514
1.494
1.478
0.858
0.031
OH
NH
N
O
N
H
N
TBSO
6e
BRUKER
NAME jc-670-149
EXPNO 2
PROCNO 1
Date_ 20121129
Time 13.20
INSTRUM spect
PROBHD 5 mm BBO BB-1H
PULPROG zg30
TD 65536
SOLVENT DMSO
NS 16
DS 2
SWH 8278.146 Hz
FIDRES 0.126314 Hz
AQ 3.9584243 sec
RG 181
DW 60.400 usec
DE 6.50 usec
TE 292.6 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 15.00 usec
PL1 1.10 dB
SFO1 400.1524711 MHz
SI 32768
SF 400.1500018 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
12 11 10 9 8 7 6 5 4 3 2 1 ppm
0.95 0.97 1.00 0.99 1.00 1.96 2.12 1.59 2.01 2.01 9.07 5.93


176.15
160.35
144.86
144.79
132.76
127.30
125.70
119.56
118.98
111.10
103.02
62.13
40.15
39.94
39.73
39.52
39.31
39.10
38.89
38.68
31.61
25.90
21.03
18.02
-5.24
OH
NH
N
O
N
H
N
TBSO
6e
BRUKER
NAME jc-670-149c
EXPNO 3
PROCNO 1
Date_ 20121201
Time 11.55
INSTRUM spect
PROBHD 5 mm BBO BB-1H
PULPROG zgpg30
TD 65536
SOLVENT DMSO
NS 2766
DS 4
SWH 23980.814 Hz
FIDRES 0.365918 Hz
AQ 1.3664756 sec
RG 4597.6
DW 20.850 usec
DE 6.50 usec
TE 293.2 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 7.50 usec
PL1 -2.00 dB
SFO1 100.6278593 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 90.00 usec
PL2 1.10 dB
PL12 16.66 dB
PL13 18.00 dB
SFO2 400.1516006 MHz
SI 32768
SF 100.6178383 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
200 180 160 140 120 100 80 60 40 20 0 ppm



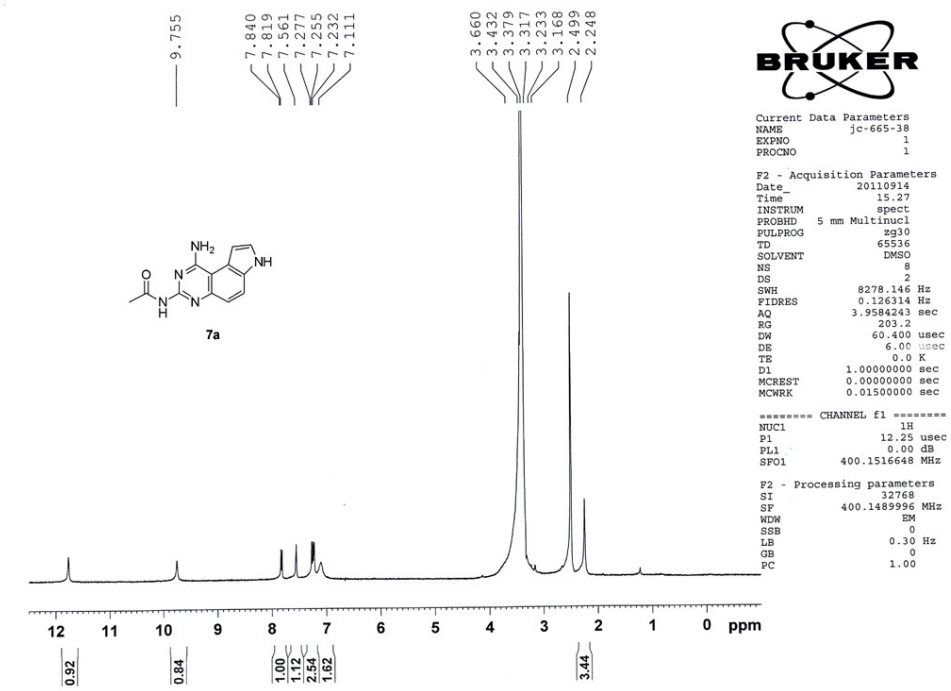
BRUKER

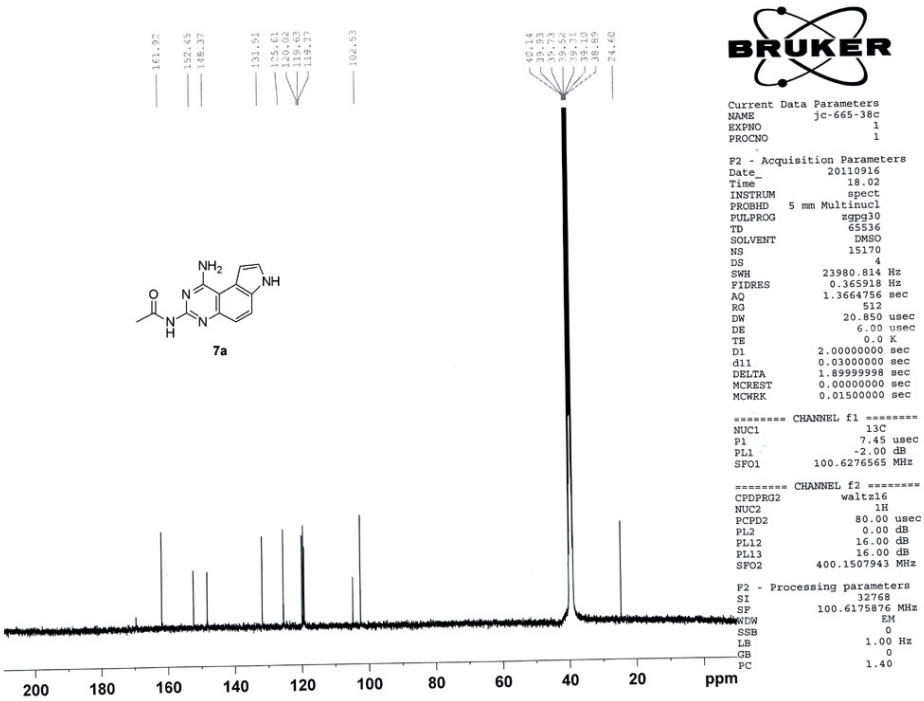
NAME	jc-670-130
EXPNO	3
PROCNO	1
Date_	20121201
Time_	9.10
INSTRUM	spect
PROBHD	5 mm BBO BB-1H
PULPROG	zg30
TD	65536
SOLVENT	DMSO
NS	11
DS	2
SWH	8278.146 Hz
FIDRES	0.126314 Hz
AQ	3.9584243 sec
RG	181
DW	60.400 usec
DE	6.50 usec
TE	292.5 K
D1	1.00000000 sec
TD0	1
======== CHANNEL f1 ========	
NUC1	1H
P1	15.00 usec
PL1	1.10 dB
SFO1	400.1524711 MHz
SI	32768
SF	400.1500018 MHz
WDW	EM
SSB	0
LB	0.30 Hz
GB	0
PC	1.00

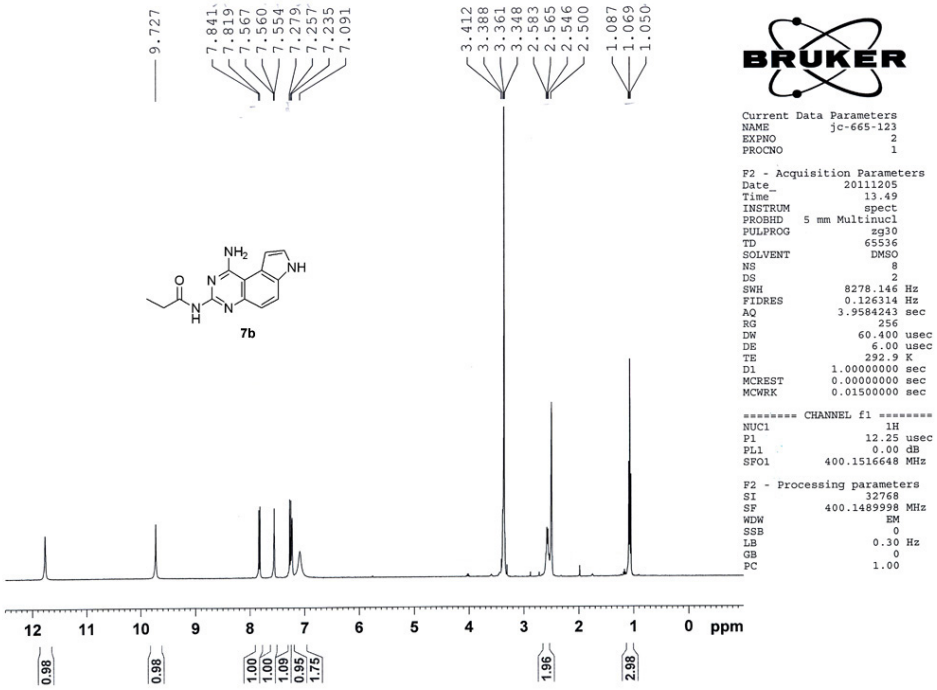


BRUKER

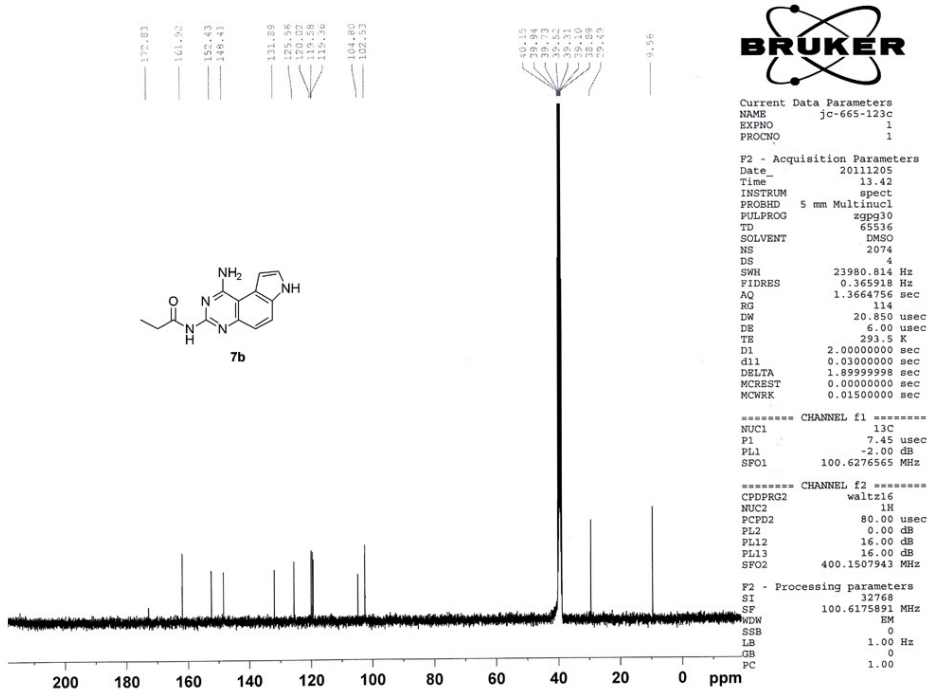
NAME	jc-665-130c
EXPNO	3
PROCNO	1
Date_	20121225
Time_	17.19
INSTRUM	spect
PROBHD	5 mm BBO BB-1H
PULPROG	zgpg30
TD	65536
SOLVENT	DMSO
NS	6000
DS	4
SWH	23980.814 Hz
FIDRES	0.365918 Hz
AQ	1.3664756 sec
RG	1290.2
DW	20.850 usec
DE	6.50 usec
TE	293.1 K
D1	2.00000000 sec
D11	0.03000000 sec
TD0	1
======== CHANNEL f1 ========	
NUC1	13C
P1	7.50 usec
PL1	-2.00 dB
SFO1	100.6278593 MHz
======== CHANNEL f2 ========	
CPDPRG2	waltz16
NUC2	1H
PCPD2	90.00 usec
PL2	1.10 dB
PL12	16.66 dB
PL13	18.00 dB
SFO2	400.1516006 MHz
SI	32768
SF	100.6178375 MHz
WDW	EM
SSB	0
LB	1.00 Hz
GB	0
PC	1.40


9.755
7.840
7.819
7.561
7.277
7.255
7.232
7.111
3.660
3.432
3.379
3.317
3.233
3.168
2.499
2.248
7a
12 11 10 9 8 7 6 5 4 3 2 1 0 ppm
0.92
0.84
1.00
1.12
2.54
1.62
3.44
BRUKER
Current Data Parameters
NAME jc-665-38
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20110914
Time 15.27
INSTRUM spect
PROBHD 5 mm Multinucl
PULPROG zg30
TD 65536
SOLVENT DMSO
NS 8
DS 2
SWH 8278.146 Hz
FIDRES 0.126314 Hz
AQ 3.9584243 sec
RG 203.2
DW 60.400 usec
DE 6.00 usec
TE 0.0 K
D1 1.00000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec
======== CHANNEL f1 ========
NUC1 1H
P1 12.25 usec
PL1 0.00 dB
SFO1 400.1516648 MHz
F2 - Processing parameters
SI 32768
SF 400.1489996 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

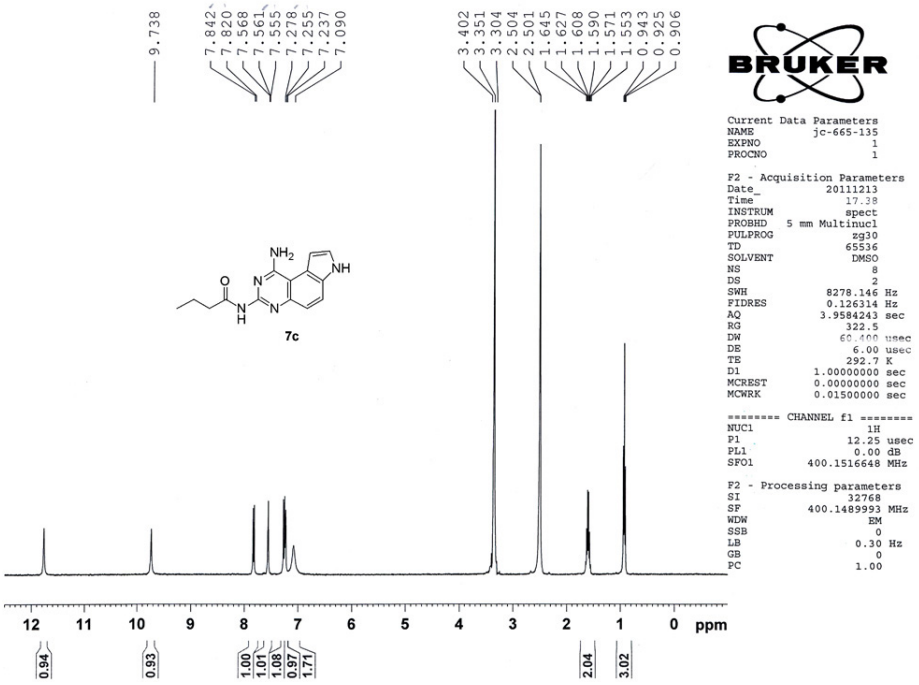

161.92
152.45
148.37
131.91
125.61
120.02
119.63
119.37
102.93
40.14
39.83
39.73
39.52
39.31
39.10
38.89
24.60
7a
200 180 160 140 120 100 80 60 40 20 ppm
BRUKER
Current Data Parameters
NAME jc-665-38c
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20110916
Time 18.02
INSTRUM spect
PROBHD 5 mm Multinucl
PULPROG zgpg30
TD 65536
SOLVENT DMSO
NS 15170
DS 4
SWH 23980.814 Hz
FIDRES 0.365918 Hz
AQ 1.3664756 sec
RG 512
DW 20.850 usec
DE 6.00 usec
TE 0.0 K
D1 2.00000000 sec
d11 0.03000000 sec
DELTA 1.89999998 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec
======== CHANNEL f1 ========
NUC1 13C
P1 7.45 usec
PL1 -2.00 dB
SFO1 100.6276565 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 0.00 dB
PL12 16.00 dB
PL13 16.00 dB
SFO2 400.1507943 MHz
F2 - Processing parameters
SI 32768
SF 100.6175876 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

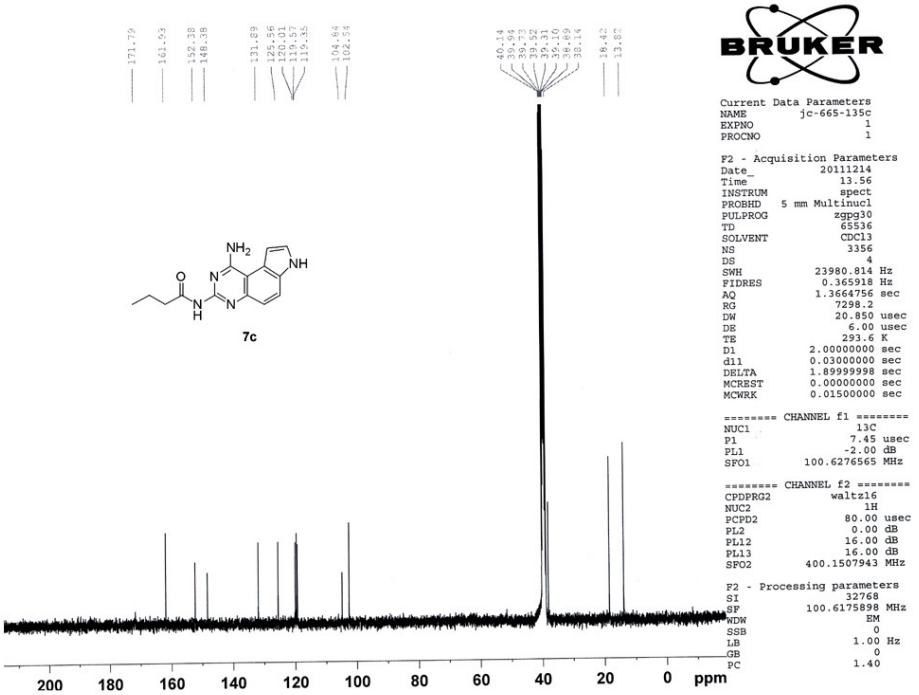


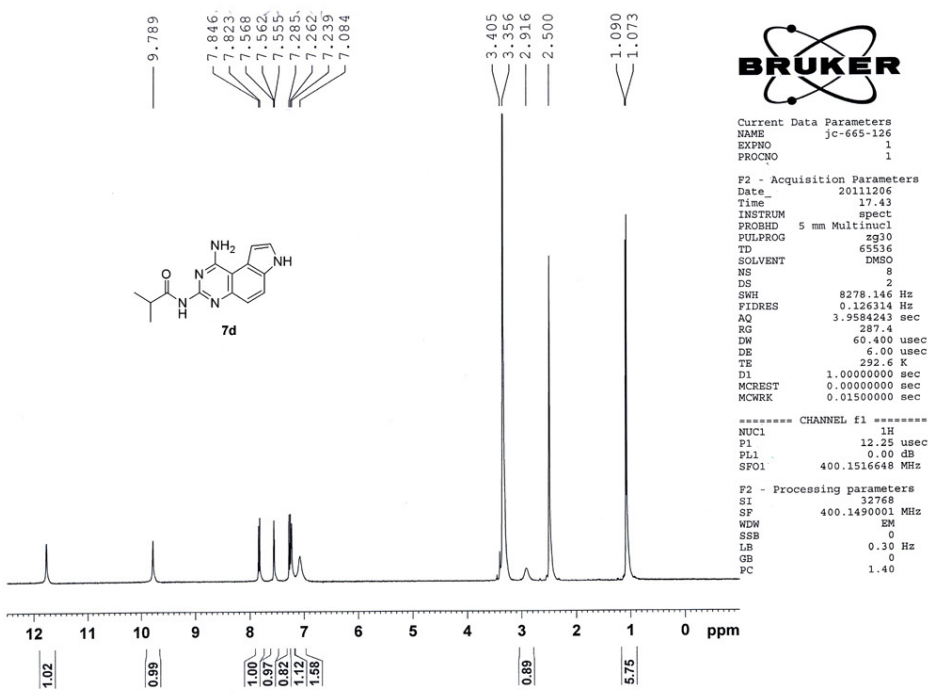

```
Current Data Parameters
NAME          jc-665-123
EXPNO                  2
PROCNO                 1

F2 - Acquisition Parameters
Date_           20111205
Time               13.49
INSTRUM            spect
PROBHD   5 mm Multinucl
PULPROG             zg30
TD                 65536
SOLVENT             DMSO
NS                     8
DS                     2
SWH           8278.146 Hz
FIDRES        0.126314 Hz
AQ            3.9584243 sec
RG                   256
DW               60.400 usec
DE                6.00 usec
TE                292.9 K
D1           1.00000000 sec
MCREST       0.00000000 sec
MCWRK        0.01500000 sec

======== CHANNEL f1 ========
NUC1                  1H
P1                12.25 usec
PL1                0.00 dB
SFO1         400.1516648 MHz

F2 - Processing parameters
SI                 32768
SF           400.1489998 MHz
WDW                   EM
SSB                    0
LB                0.30 Hz
GB                     0
PC                  1.00
```

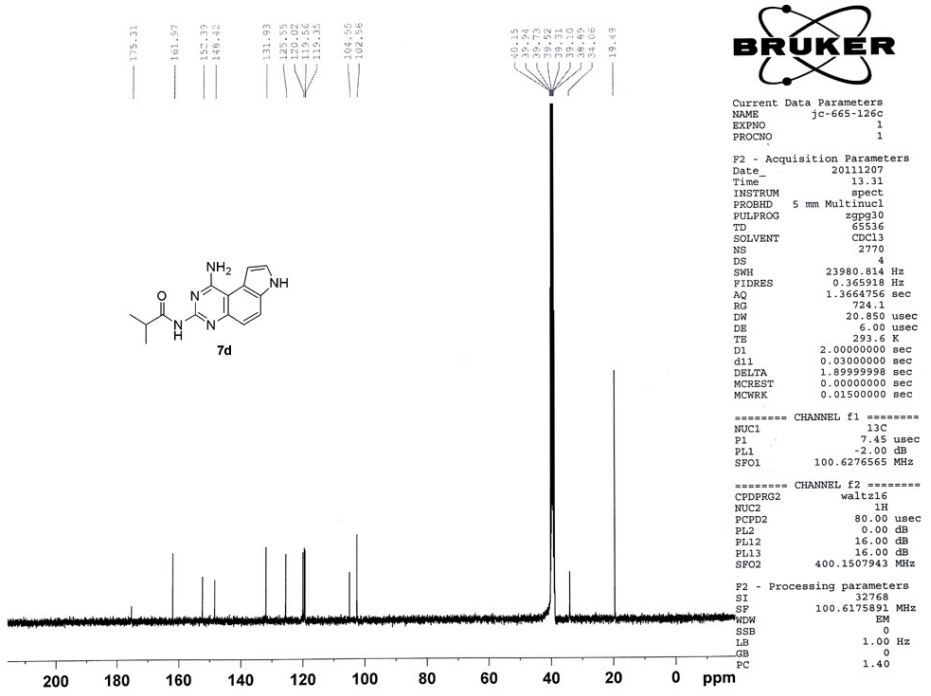



```
Current Data Parameters
NAME        jc-665-123c
EXPNO                 1
PROCNO                1

F2 - Acquisition Parameters
Date_           20111205
Time               13.42
INSTRUM            spect
PROBHD   5 mm Multinucl
PULPROG           zgpg30
TD                 65536
SOLVENT             DMSO
NS                  2074
DS                     4
SWH          23980.814 Hz
FIDRES        0.365918 Hz
AQ            1.3664756 sec
RG                   114
DW               20.850 usec
DE                6.00 usec
TE                293.5 K
D1           2.00000000 sec
d11          0.03000000 sec
DELTA        1.89999998 sec
MCREST       0.00000000 sec
MCWRK        0.01500000 sec

======== CHANNEL f1 ========
NUC1                 13C
P1                7.45 usec
PL1               -2.00 dB
SFO1         100.6276565 MHz

======== CHANNEL f2 ========
CPDPRG2          waltz16
NUC2                  1H
PCPD2            80.00 usec
PL2                0.00 dB
PL12              16.00 dB
PL13              16.00 dB
SFO2         400.1507943 MHz

F2 - Processing parameters
SI                 32768
SF           100.6175891 MHz
WDW                   EM
SSB                    0
LB                1.00 Hz
GB                     0
PC                  1.00
```


9.738
7.842
7.820
7.568
7.561
7.555
7.278
7.255
7.237
7.090
3.402
3.351
3.304
2.501
1.645
1.627
1.608
1.590
1.571
1.553
0.943
0.925
0.906
7c
12 11 10 9 8 7 6 5 4 3 2 1 0 ppm
0.94
0.93
1.00
1.01
1.08
0.97
1.71
2.04
3.02
Current Data Parameters
NAME jc-665-135
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20111213
Time_ 17.38
INSTRUM spect
PROBHD 5 mm Multinucl
PULPROG zg30
TD 65536
SOLVENT DMSO
NS 8
DS 2
SWH 8278.146 Hz
FIDRES 0.126314 Hz
AQ 3.9584243 sec
RG 322.5
DW 60.400 usec
DE 6.00 usec
TE 292.7 K
D1 1.00000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec
======== CHANNEL f1 ========
NUC1 1H
P1 12.25 usec
PL1 0.00 dB
SFO1 400.1516648 MHz
F2 - Processing parameters
SI 32768
SF 400.1489993 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00


171.79
161.93
152.38
148.38
131.89
125.56
120.01
119.57
119.35
104.84
102.54
40.14
39.94
39.73
39.52
39.31
39.10
38.89
38.14
18.42
13.82
7c
200 180 160 140 120 100 80 60 40 20 0 ppm
Current Data Parameters
NAME jc-665-135c
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20111214
Time_ 13.56
INSTRUM spect
PROBHD 5 mm Multinucl
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 3356
DS 4
SWH 23980.814 Hz
FIDRES 0.365918 Hz
AQ 1.3664756 sec
RG 7298.2
DW 20.850 usec
DE 6.00 usec
TE 293.6 K
D1 2.00000000 sec
d11 0.03000000 sec
DELTA 1.89999998 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec
======== CHANNEL f1 ========
NUC1 13C
P1 7.45 usec
PL1 -2.00 dB
SFO1 100.6276565 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 0.00 dB
PL12 16.00 dB
PL13 16.00 dB
SFO2 400.1507943 MHz
F2 - Processing parameters
SI 32768
SF 100.6175898 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40


9.789
7.846
7.823
7.568
7.562
7.555
7.285
7.262
7.239
7.084
3.405
3.356
2.916
2.500
1.090
1.073
7d
12 11 10 9 8 7 6 5 4 3 2 1 0 ppm
1.02
0.99
1.00
0.97
0.82
1.12
1.58
0.89
5.75
BRUKER
Current Data Parameters
NAME jc-665-126
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20111206
Time 17.43
INSTRUM spect
PROBHD 5 mm Multinucl
PULPROG zg30
TD 65536
SOLVENT DMSO
NS 8
DS 2
SWH 8278.146 Hz
FIDRES 0.126314 Hz
AQ 3.9584243 sec
RG 287.4
DW 60.400 usec
DE 6.00 usec
TE 292.6 K
D1 1.00000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec
======== CHANNEL f1 ========
NUC1 1H
P1 12.25 usec
PL1 0.00 dB
SFO1 400.1516648 MHz
F2 - Processing parameters
SI 32768
SF 400.1490001 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.40


175.31
161.97
152.39
148.42
131.93
125.55
120.02
119.56
119.38
104.98
102.96
40.15
39.94
39.73
39.52
39.31
39.10
38.89
34.06
19.49
7d
200 180 160 140 120 100 80 60 40 20 0 ppm
BRUKER
Current Data Parameters
NAME jc-665-126c
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20111207
Time 13.31
INSTRUM spect
PROBHD 5 mm Multinucl
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 2770
DS 4
SWH 23980.814 Hz
FIDRES 0.365918 Hz
AQ 1.3664756 sec
RG 724.1
DW 20.850 usec
DE 6.00 usec
TE 293.6 K
D1 2.00000000 sec
d11 0.03000000 sec
DELTA 1.89999998 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec
======== CHANNEL f1 ========
NUC1 13C
P1 7.45 usec
PL1 -2.00 dB
SFO1 100.6276565 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 0.00 dB
PL12 16.00 dB
PL13 16.00 dB
SFO2 400.1507943 MHz
F2 - Processing parameters
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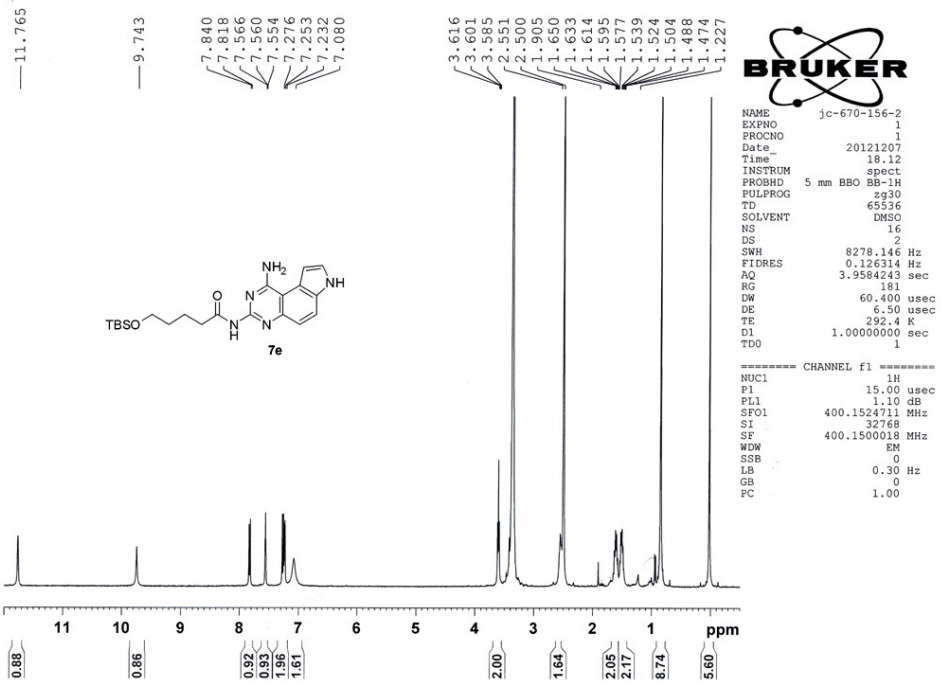
SF 100.6175891 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40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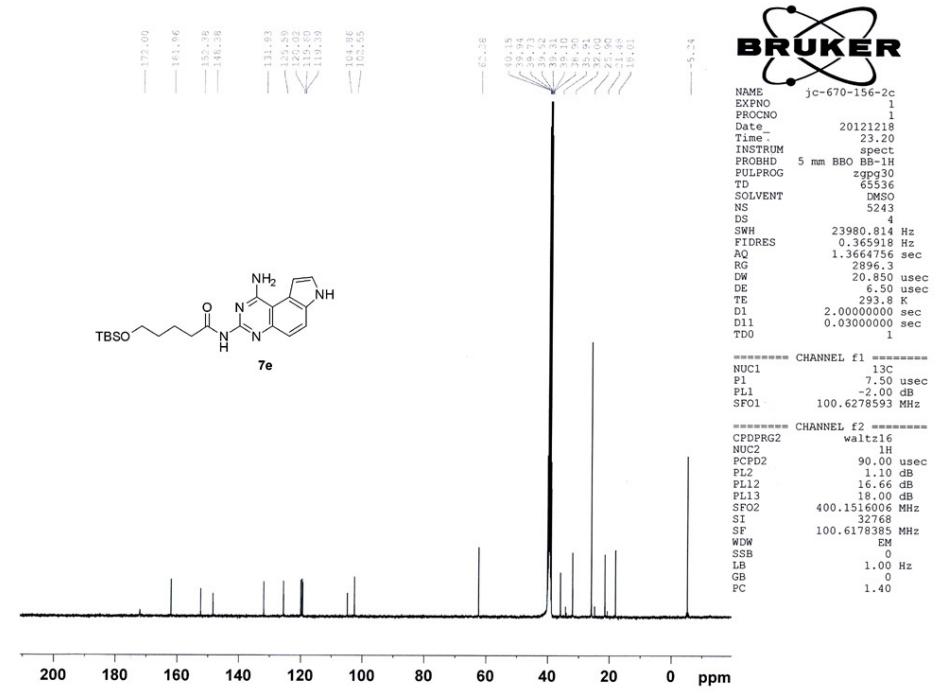
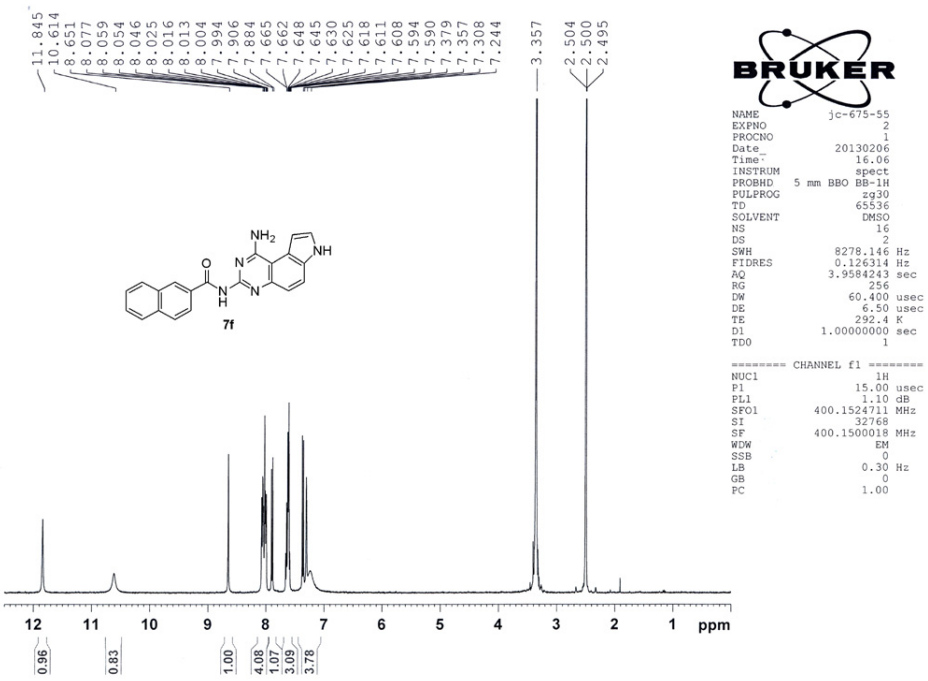
9.743
7.840
7.818
7.566
7.560
7.554
7.276
7.253
7.232
7.080
3.616
3.601
3.585
2.551
2.500
1.905
1.650
1.633
1.614
1.595
1.577
1.539
1.524
1.504
1.488
1.474
1.227
BRUKER
NAME jc-670-156-2
EXPNO 1
PROCNO 1
Date_ 20121207
Time 18.12
INSTRUM spect
PROBHD 5 mm BBO BB-1H
PULPROG zg30
TD 65536
SOLVENT DMSO
NS 16
DS 2
SWH 8278.146 Hz
FIDRES 0.126314 Hz
AQ 3.9584243 sec
RG 181
DW 60.400 usec
DE 6.50 usec
TE 292.4 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 15.00 usec
PL1 1.10 dB
SFO1 400.1524711 MHz
SI 32768
SF 400.1500018 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
NH2
NH
O
N
N
N
H
TBSO
7e
11
10
9
8
7
6
5
4
3
2
1
ppm
0.88
0.86
0.92
0.93
1.96
1.61
2.00
1.64
2.05
2.17
8.74
5.60


172.00
161.96
157.38
148.38
131.93
125.89
122.02
119.80
119.39
104.88
102.55
67.38
60.15
39.94
39.73
39.52
39.31
39.10
38.90
35.91
27.90
27.00
18.01
-5.24
BRUKER
NAME jc-670-156-2c
EXPNO 1
PROCNO 1
Date_ 20121218
Time 23.20
INSTRUM spect
PROBHD 5 mm BBO BB-1H
PULPROG zgpg30
TD 65536
SOLVENT DMSO
NS 5243
DS 4
SWH 23980.814 Hz
FIDRES 0.365918 Hz
AQ 1.3664756 sec
RG 2896.3
DW 20.850 usec
DE 6.50 usec
TE 293.8 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 7.50 usec
PL1 -2.00 dB
SFO1 100.6278593 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 90.00 usec
PL2 1.10 dB
PL12 16.66 dB
PL13 18.00 dB
SFO2 400.1516006 MHz
SI 32768
SF 100.6178385 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
NH2
NH
O
N
N
N
H
TBSO
7e
200
180
160
140
120
100
80
60
40
20
0
ppm


11.845
10.614
8.651
8.077
8.059
8.054
8.046
8.025
8.016
8.013
8.004
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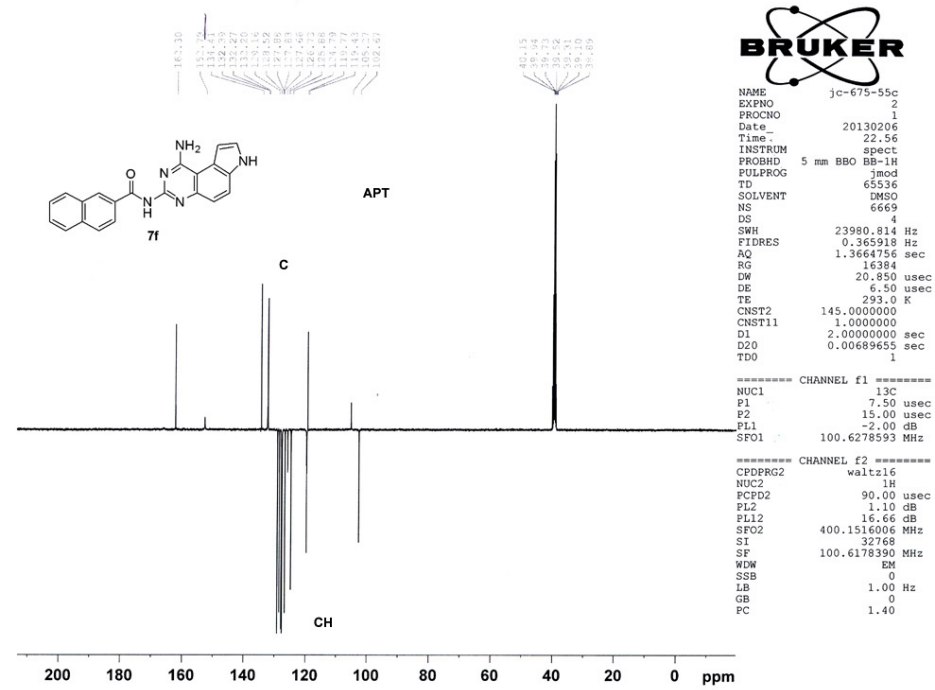
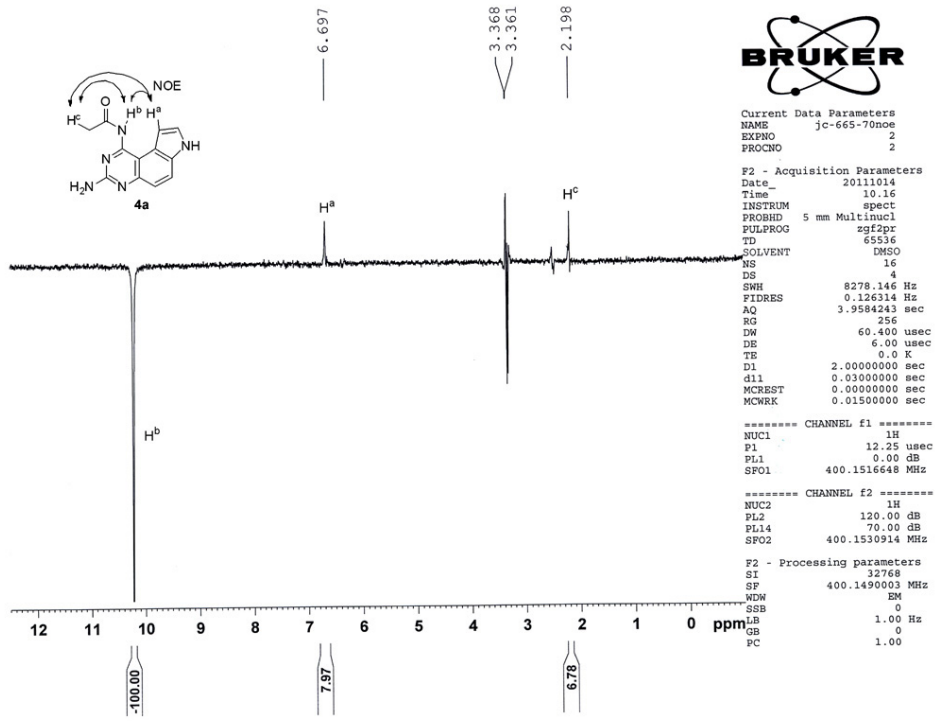
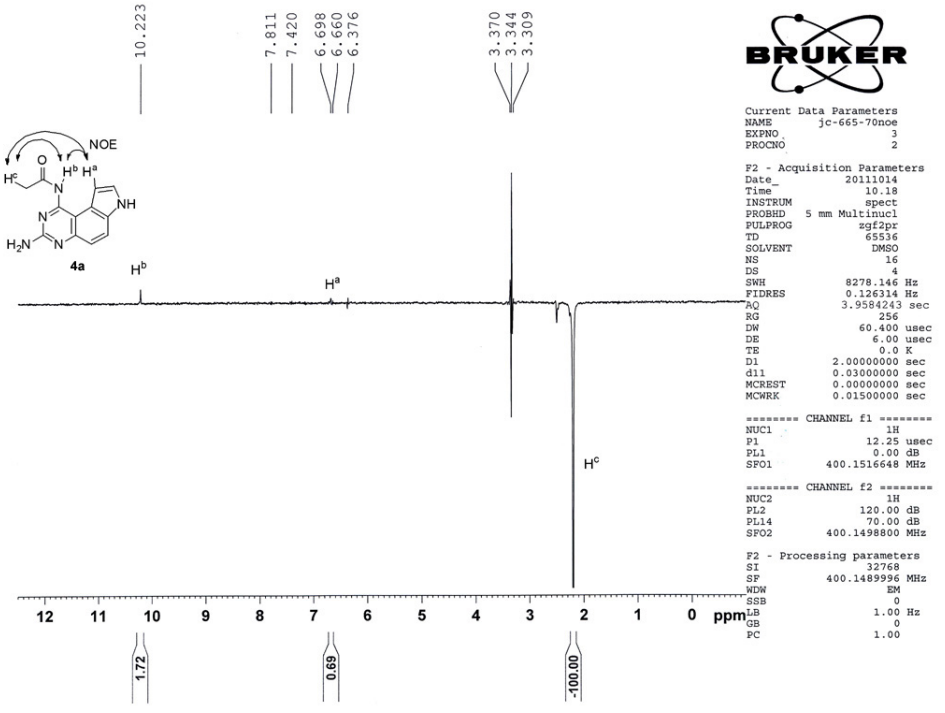
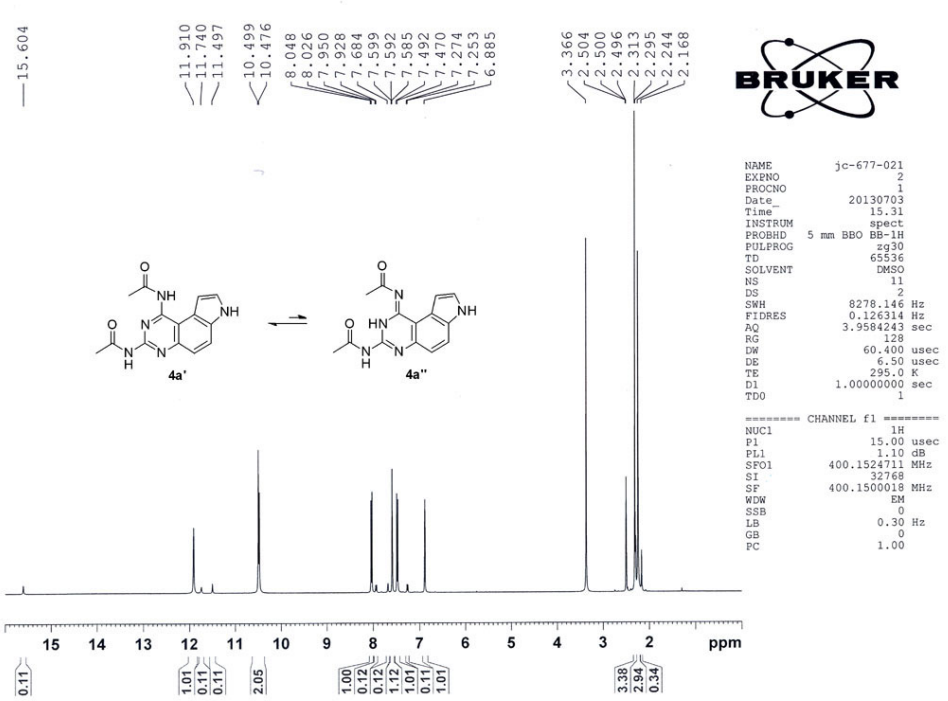
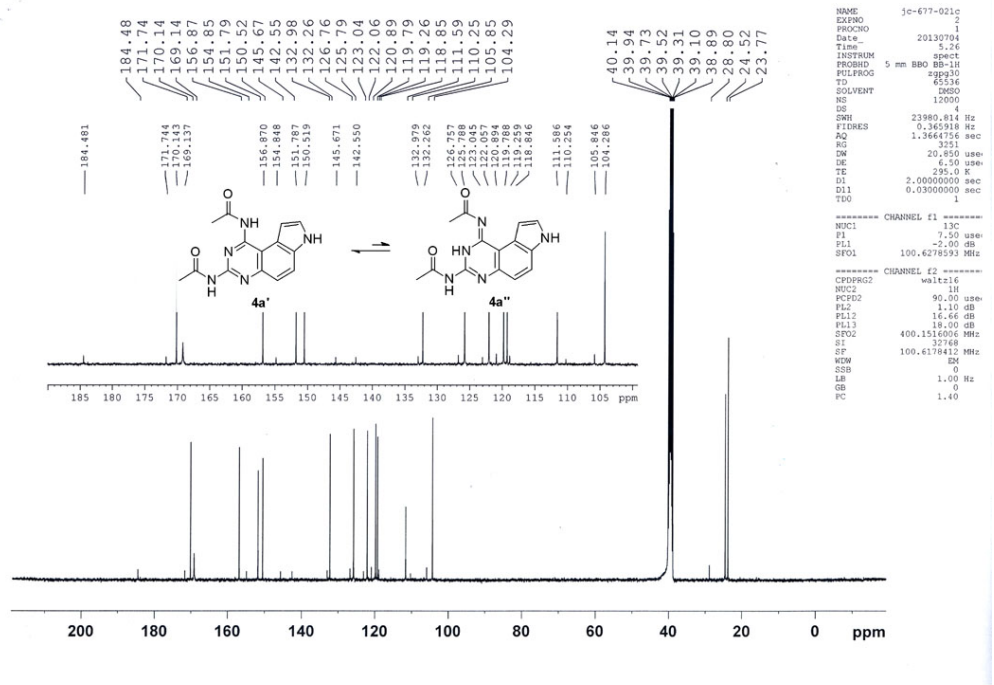
7.906
7.884
7.665
7.662
7.648
7.645
7.630
7.625
7.618
7.611
7.608
7.594
7.590
7.379
7.357
7.308
7.244
3.357
2.504
2.500
2.495
7f
12
11
10
9
8
7
6
5
4
3
2
1
ppm
0.96
0.83
1.00
4.08
1.07
3.09
3.78
BRUKER
NAME jc-675-55
EXPNO 2
PROCNO 1
Date_ 20130206
Time_ 16.06
INSTRUM spect
PROBHD 5 mm BBO BB-1H
PULPROG zg30
TD 65536
SOLVENT DMSO
NS 16
DS 2
SWH 8278.146 Hz
FIDRES 0.126314 Hz
AQ 3.9584243 sec
RG 256
DW 60.400 usec
DE 6.50 usec
TE 292.4 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 15.00 usec
PL1 1.10 dB
SFO1 400.1524711 MHz
SI 32768
SF 400.1500018 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

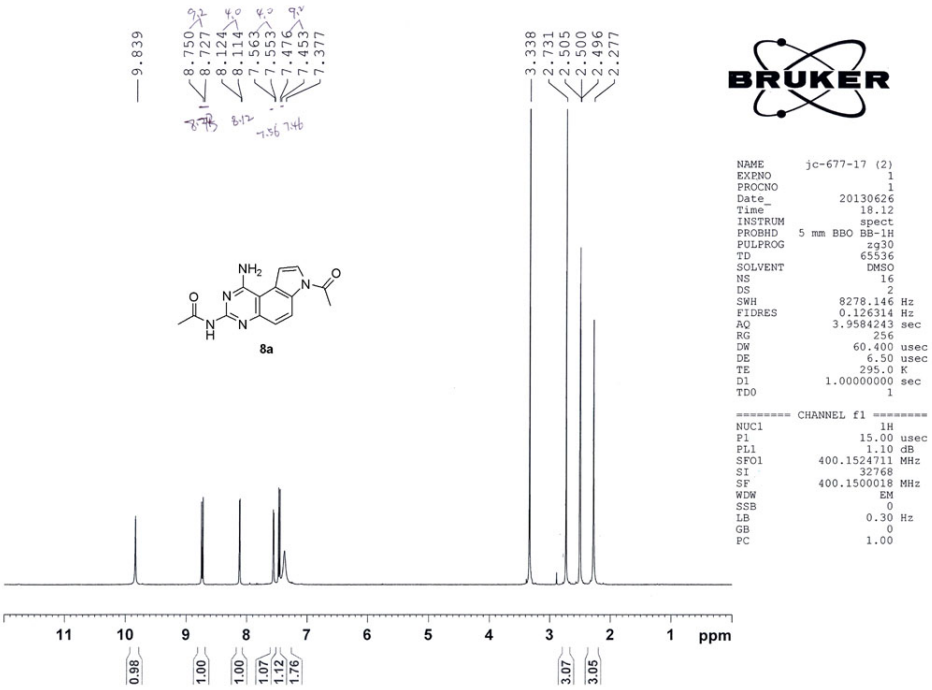

7f
APT
C
CH
200
180
160
140
120
100
80
60
40
20
0
ppm
BRUKER
NAME jc-675-55c
EXPNO 2
PROCNO 1
Date_ 20130206
Time_ 22.56
INSTRUM spect
PROBHD 5 mm BBO BB-1H
PULPROG jmod
TD 65536
SOLVENT DMSO
NS 6669
DS 4
SWH 23980.814 Hz
FIDRES 0.365918 Hz
AQ 1.3664756 sec
RG 16384
DW 20.850 usec
DE 6.50 usec
TE 293.0 K
CNST2 145.0000000
CNST11 1.0000000
D1 2.00000000 sec
D20 0.00689655 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 7.50 usec
P2 15.00 usec
PL1 -2.00 dB
SFO1 100.6278593 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 90.00 usec
PL2 1.10 dB
PL12 16.66 dB
SFO2 400.1516006 MHz
SI 32768
SF 100.6178390 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40


6.697
3.368
3.361
2.198
NOE
4a
Ha
Hb
Hc
-100.00
7.97
6.78
ppm
BRUKER
Current Data Parameters
NAME jc-665-70noe
EXPNO 2
PROCNO 2
F2 - Acquisition Parameters
Date_ 20111014
Time 10.16
INSTRUM spect
PROBHD 5 mm Multinucl
PULPROG zgf2pr
TD 65536
SOLVENT DMSO
NS 16
DS 4
SWH 8278.146 Hz
FIDRES 0.126314 Hz
AQ 3.9584243 sec
RG 256
DW 60.400 usec
DE 6.00 usec
TE 0.0 K
D1 2.00000000 sec
d11 0.03000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec
======== CHANNEL f1 ========
NUC1 1H
P1 12.25 usec
PL1 0.00 dB
SFO1 400.1516648 MHz
======== CHANNEL f2 ========
NUC2 1H
PL2 120.00 dB
PL14 70.00 dB
SFO2 400.1530914 MHz
F2 - Processing parameters
SI 32768
SF 400.1490003 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.00

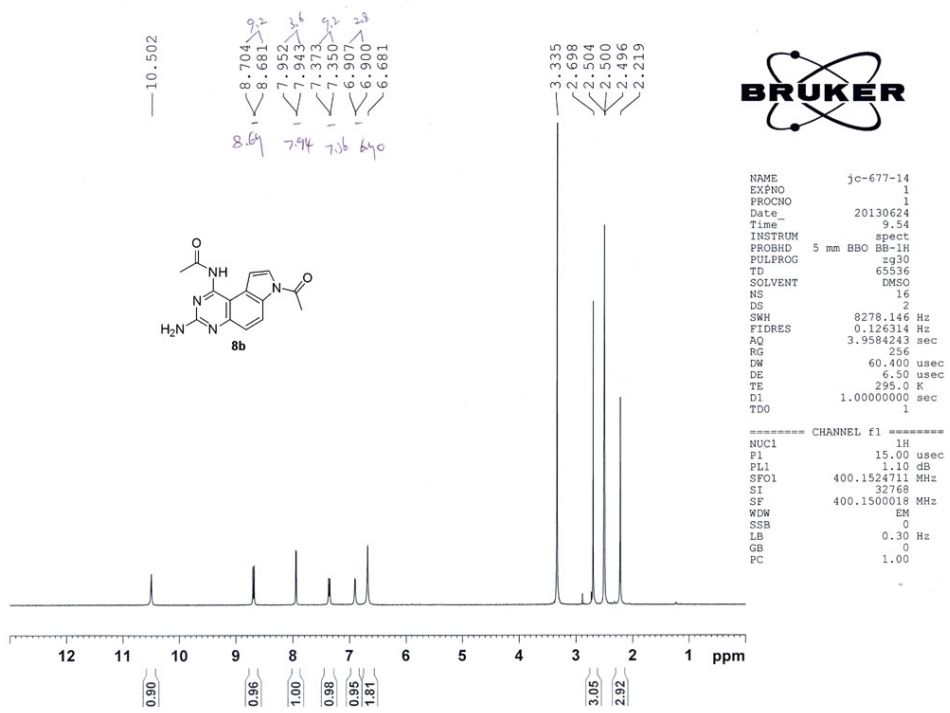

10.223
7.811
7.420
6.698
6.660
6.376
3.370
3.344
3.309
NOE
4a
Hb
Ha
Hc
1.72
0.69
-100.00
ppm
BRUKER
Current Data Parameters
NAME jc-665-70noe
EXPNO 3
PROCNO 2
F2 - Acquisition Parameters
Date_ 20111014
Time 10.18
INSTRUM spect
PROBHD 5 mm Multinucl
PULPROG zgf2pr
TD 65536
SOLVENT DMSO
NS 16
DS 4
SWH 8278.146 Hz
FIDRES 0.126314 Hz
AQ 3.9584243 sec
RG 256
DW 60.400 usec
DE 6.00 usec
TE 0.0 K
D1 2.00000000 sec
d11 0.03000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec
======== CHANNEL f1 ========
NUC1 1H
P1 12.25 usec
PL1 0.00 dB
SFO1 400.1516648 MHz
======== CHANNEL f2 ========
NUC2 1H
PL2 120.00 dB
PL14 70.00 dB
SFO2 400.1498800 MHz
F2 - Processing parameters
SI 32768
SF 400.1489996 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.00

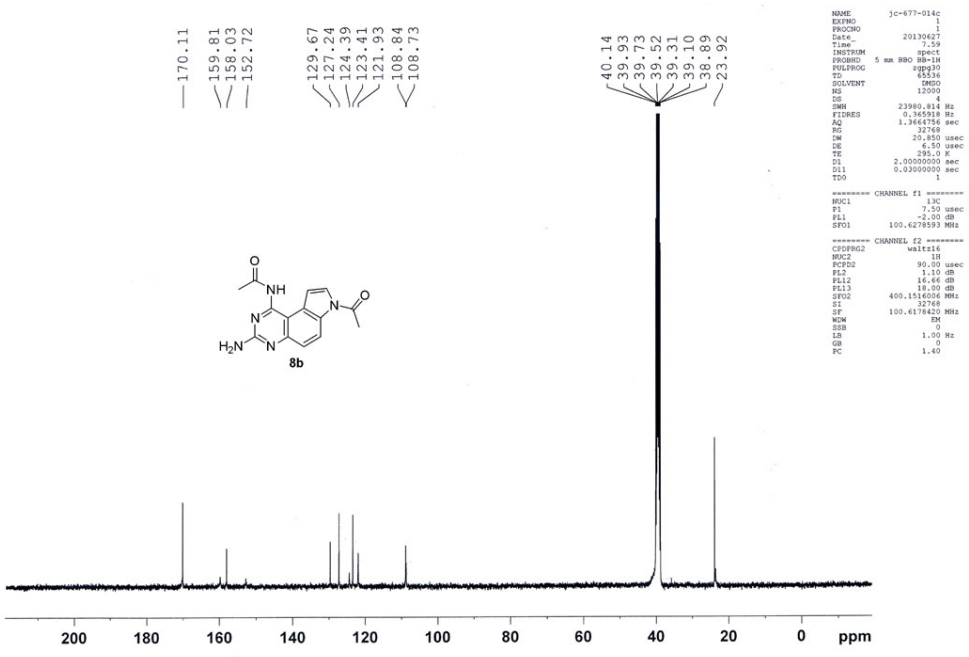

15.604
11.910
11.740
11.497
10.499
10.476
8.048
8.026
7.950
7.928
7.684
7.599
7.592
7.585
7.492
7.470
7.274
7.253
6.885
3.366
2.504
2.500
2.496
2.313
2.295
2.244
2.168
BRUKER
NAME jc-677-021
EXPNO 2
PROCNO 1
Date_ 20130703
Time 15.31
INSTRUM spect
PROBHD 5 mm BBO BB-1H
PULPROG zg30
TD 65536
SOLVENT DMSO
NS 11
DS 2
SWH 8278.146 Hz
FIDRES 0.126314 Hz
AQ 3.9584243 sec
RG 128
DW 60.400 usec
DE 6.50 usec
TE 295.0 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 15.00 usec
PL1 1.10 dB
SFO1 400.1524711 MHz
SI 32768
SF 400.1500018 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
4a'
4a''
15 14 13 12 11 10 9 8 7 6 5 4 3 2 ppm
0.11 1.01 0.11 0.11 2.05 1.00 0.12 0.12 1.12 1.01 0.11 1.01 3.38 2.94 0.34

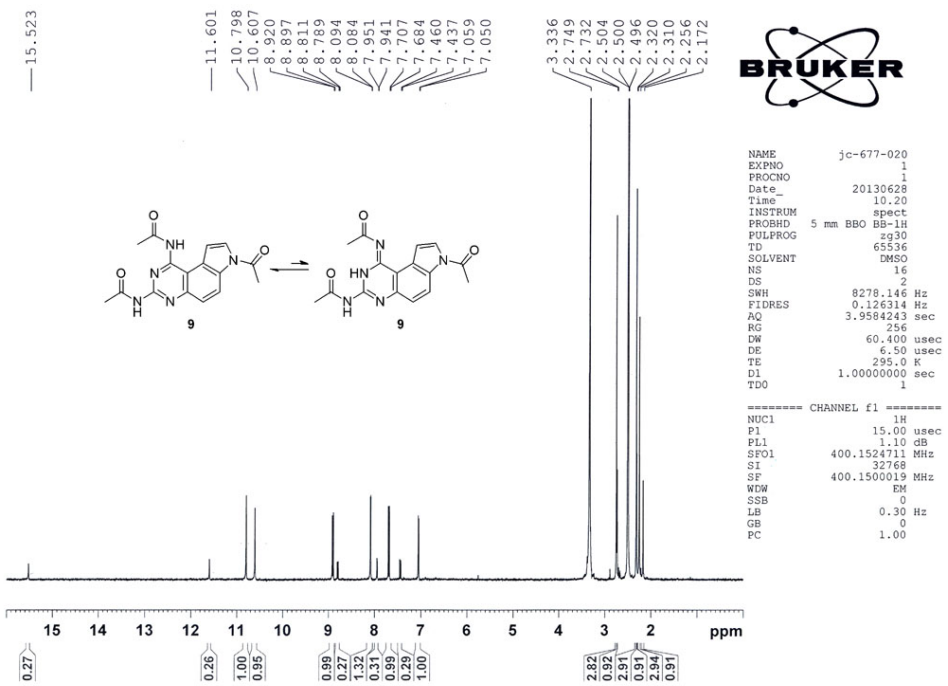

184.48
171.74
170.14
169.14
156.87
154.85
151.79
150.52
145.67
142.55
132.98
132.26
126.76
125.79
123.04
122.06
120.89
119.79
119.26
118.85
111.59
110.25
105.85
104.29
40.14
39.94
39.73
39.52
39.31
39.10
38.89
28.80
24.52
23.77
NAME jc-677-021c
EXPNO 2
PROCNO 1
Date_ 20130704
Time 5.26
INSTRUM spect
PROBHD 5 mm BBO BB-1H
PULPROG zgpg30
TD 65536
SOLVENT DMSO
NS 12000
DS 4
SWH 23980.814 Hz
FIDRES 0.365918 Hz
AQ 1.3664756 sec
RG 3251
DW 20.850 usec
DE 6.50 usec
TE 295.0 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 7.50 usec
PL1 -2.00 dB
SFO1 100.6278593 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 90.00 usec
PL2 1.10 dB
PL12 16.66 dB
PL13 18.00 dB
SFO2 400.1516006 MHz
SI 32768
SF 100.6178412 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
4a'
4a''
185 180 175 170 165 160 155 150 145 140 135 130 125 120 115 110 105 ppm
200 180 160 140 120 100 80 60 40 20 0 ppm



NAME	jc-677-17 (2)
EXPNO	1
PROCNO	1
Date_	20130626
Time	18.12
INSTRUM	spect
PROBHD	5 mm BBO BB-1H
PULPROG	zg30
TD	65536
SOLVENT	DMSO
NS	16
DS	2
SWH	8278.146 Hz
FIDRES	0.126314 Hz
AQ	3.9584243 sec
RG	256
DW	60.400 usec
DE	6.50 usec
TE	295.0 K
D1	1.00000000 sec
TD0	1
======== CHANNEL f1 ========	
NUC1	1H
P1	15.00 usec
PL1	1.10 dB
SFO1	400.1524711 MHz
SI	32768
SF	400.1500018 MHz
WDW	EM
SSB	0
LB	0.30 Hz
GB	0
PC	1.00


10.502
8.704
8.681
7.952
7.943
7.373
7.350
6.907
6.900
6.681
3.335
2.698
2.504
2.500
2.496
2.219
8b
12 11 10 9 8 7 6 5 4 3 2 1 ppm
0.90
0.96
1.00
0.98
0.95
1.81
3.05
2.92


170.11
159.81
158.03
152.72
129.67
127.24
124.39
123.41
121.93
108.84
108.73
40.14
39.93
39.73
39.52
39.31
39.10
38.89
23.92
8b
200 180 160 140 120 100 80 60 40 20 0 ppm


15.523
11.601
10.798
10.607
8.920
8.897
8.811
8.789
8.094
8.084
7.951
7.941
7.707
7.684
7.460
7.437
7.059
7.050
3.336
2.749
2.732
2.504
2.500
2.496
2.320
2.310
2.256
2.172
9
9
BRUKER
NAME jc-677-020
EXPNO 1
PROCNO 1
Date_ 20130628
Time 10.20
INSTRUM spect
PROBHD 5 mm BBO BB-1H
PULPROG zg30
TD 65536
SOLVENT DMSO
NS 16
DS 2
SWH 8278.146 Hz
FIDRES 0.126314 Hz
AQ 3.9584243 sec
RG 256
DW 60.400 usec
DE 6.50 usec
TE 295.0 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 15.00 usec
PL1 1.10 dB
SFO1 400.1524711 MHz
SI 32768
SF 400.1500019 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
15 14 13 12 11 10 9 8 7 6 5 4 3 2 ppm
0.27
0.26
1.00
0.95
0.99
0.27
1.32
0.31
0.99
0.29
1.00
2.82
0.92
2.91
0.91
2.94
0.91

References cited.

1. M. Whitlow, A. J. Howard, D. Stewart, K. D. Hardman, L. F. Kuyper, D. P. Baccanari, M. E. Fling and R. L. Tansik, *J. Biol. Chem.*, 1997, **272**, 30289-30298.

2. M. L. Jones, L. F. Kuyper, V. L. Styles and J. M. Caddell, *J. Heterocycl. Chem.*, 1994, **31**, 1681-1683.

3. The tautomeric mixture became a single tautomer **4f** upon treatment with an aqueous NaOH solution (see supporting information for its NMR spectra). In addition, all the active protons in **4f** disappeared in its ^{1}H NMR due to H-D exchange with HDO generated from reaction of NaOH with DMSO-d_6. For the same reason, the signals from the residual solvents in both ^{1}H NMR and ^{13}C NMR spectra became very complicated.

4. B. X. Li and X. Xiao, *ChemBioChem*, 2009, **10**, 2721-2724.

5. B. X. Li, K. Yamanaka and X. Xiao, *Bioorg. Med. Chem.*, 2012, **20**, 6811-6820.