

Silica sulfuric acid/ethylene glycol as an efficient catalyst for the synthesis of benzo[4,5]imidazo[1,2-*a*]pyrimidine-3-carbonitrile derivatives

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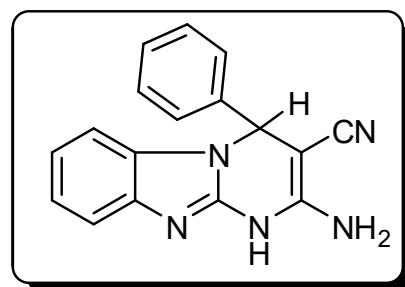
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Experimental Part:

All melting points are recorded on digital Gallen Kamp MFB-595 instrument and may be uncorrected. The IR spectra (KBr) (cm^{-1}) were measured on a JASCO spectrophotometer. ^1H NMR spectra were recorded on Bruker spectrometers (at 400 MHz) and are reported relative to deuterated solvent signals in deuterated dimethylsulfoxide ($\text{DMSO}-d_6$). ^{13}C NMR spectra were recorded on Bruker Spectrometers in deuterated dimethylsulfoxide ($\text{DMSO}-d_6$).

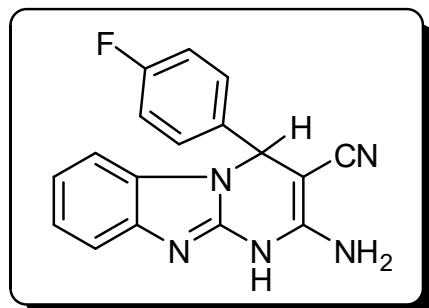
Synthesis of 2-amino-4-phenyl-1,4-dihydrobenzo[4,5]imidazo[1,2-*a*] pyrimidine-3-carbonitrile (1a):



To the mixture of 2-aminobenzimidazole (1 mmol), malononitrile (1 mmol) and benzaldehyde (1 mmol) in ethylene glycol (5 mL), SSA (42.6 mg, 0.11 mol %) was added. The mixture was stirred at room temperature for 5 minutes. After completion of the reaction (TLC), 10 mL EtOAc was added to the reaction mixture and the catalyst was recovered by filtration. The organic layer was dried over Na_2SO_4 ; the solvent was evaporated and purified by recrystallization from ethanol to give compounds **1a**. Yield 96%; m.p. 227-229 °C (Lit. 236-238,^[1] 233-236^[2], 225-227^[3]); IR: ν/cm^{-1} : 3431, 3320, 3214 (NH₂ & NH), 2920 (CH-

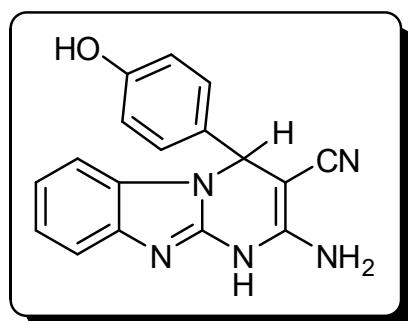
aliph), 2189 (C≡N), 1682 (C=N); Anal. calcd for C₁₇H₁₃N₅ (287.32): C, 71.06; H, 4.56; N, 24.37; Found: C, 71.21; H, 4.51; N, 24.43 %.

Synthesis of 2-amino-4-(4-fluorophenyl)-1,4-dihydrobenzo[4,5]imidazo[1,2-*a*]pyrimidine-3-carbonitrile (1b)



To the mixture of 2-aminobenzimidazole (1 mmol), malononitrile (1 mmol) and 4-fluorobenzaldehyde (1 mmol) in ethylene glycol (5 mL), SSA (42.6 mg, 0.11 mol %) was added. The mixture was heated at 50 °C for 5 minutes. After completion of the reaction (TLC), 10 mL EtOAc was added to the reaction mixture and the catalyst was recovered by filtration. The organic layer was dried over Na₂SO₄; the solvent was evaporated and purified by recrystallization from ethanol to give compounds **1b**. Yield 87%; m.p. 232 °C (Lit. 254-256,^[1] 252-266,^[2] 260-262^[3]); IR: ν/cm^{-1} : 3431, 3324, 3219 (NH₂ & NH), 2922 (CH-aliph), 2188 (C≡N), 1680 (C=N); Anal. calcd for C₁₇H₁₂FN₅ (305.31): C, 66.88; H, 3.96; N, 22.94; Found: C, 66.71; H, 4.01; N, 22.78 %.

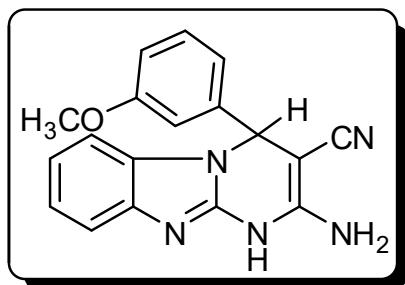
Synthesis of 2-amino-4-(4-hydroxyphenyl)-1,4-dihydrobenzo[4,5]imidazo[1,2-*a*]pyrimidine-3-carbonitrile (1c)



To the mixture of 2-aminobenzimidazole (1 mmol), malononitrile (1 mmol) and 4-hydroxybenzaldehyde (1 mmol) in ethylene glycol (5 mL), SSA (42.6 mg, 0.11 mol %) was added. The mixture was heated at 50 °C for 15 minutes. After completion of the reaction (TLC), 10 mL EtOAc was added to the reaction mixture and the catalyst was recovered by filtration. The organic layer was dried over Na₂SO₄; the solvent was evaporated and purified by recrystallization from ethanol to give compounds **1c**. Yield 83%; m.p. 222 °C; IR: ν/cm^{-1} : 3481, 3382, 3326 (OH, NH₂ & NH), 2911 (CH-aliph), 2188 (C≡N), 1679 (C=N); ¹H NMR

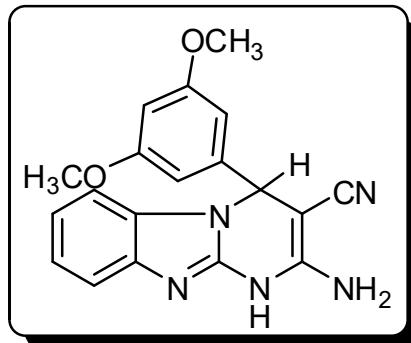
(400 MHz, DMSO): δ /ppm = 5.13 (s, 1H, CH), 6.75 (m, 3H, Ar-H & NH₂), 6.94 (m, 1H, Ar-H), 7.00 (t, 1H, Ar-H), 7.11 (d, 2H, Ar-H), 7.21 (d, 1H, Ar-H), 7.65 (d, 1H, Ar-H), 7.85 (d, 1H, Ar-H), 8.58 (br, 1H, OH); ¹³C NMR (101 MHz, DMSO): 53.5, 63.1, 112.8, 115.8 (2C), 116.5, 119.7, 120.3, 123.8, 127.8 (2C), 129.8, 133.6, 144.1, 149.4, 152.3, 157.6; Anal. calcd for C₁₇H₁₃N₅O (303.32): C, 67.32; H, 4.32; N, 23.09; Found: C, 67.46; H, 4.29; N, 22.98 %.

Synthesis of 2-amino-4-(3-methoxyphenyl)-1,4-dihydrobenzo[4,5]imidazo[1,2-*a*]pyrimidine-3-carbonitrile (1d)



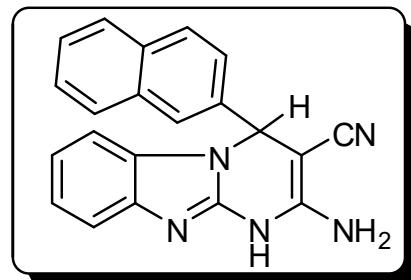
To the mixture of 2-aminobenzimidazole (1 mmol), malononitrile (1 mmol) and 3-methoxybenzaldehyde (1 mmol) in ethylene glycol (5 mL), SSA (42.6 mg, 0.11 mol %) was added. The mixture was stirred at room temperature for 10 minutes. After completion of the reaction (TLC), 10 mL EtOAc was added to the reaction mixture and the catalyst was recovered by filtration. The organic layer was dried over Na₂SO₄; the solvent was evaporated and purified by recrystallization from ethanol to give compounds **1d**. Yield 83%; m.p. 225 °C; IR: ν /cm⁻¹: 3427, 3314, 3214 (NH₂ & NH), 2918 (CH-aliph), 2191 (C≡N), 1680 (C=N); ¹H NMR (400 MHz, DMSO): δ /ppm = 3.71 (s, 3H, OCH₃), 5.20 (s, 1H, CH), 6.86 (m, 5H, 3Ar-H & NH₂), 7.02 (m, 1H, Ar-H), 7.13 (m, 1H, Ar-H), 7.19-7.35 (m, 2H, Ar-H), 7.64 (m, 1H, Ar-H), 8.63 (br, 1H, NH); ¹³C NMR (101 MHz, DMSO): 49.6 (CH), 56.5 (OCH₃), 61.4, 112.6, 112.8, 112.9, 114.1, 116.6, 119.5, 120.3, 123.4, 129.5, 131.7, 144.1, 149.9, 151.1, 152.8, 153.5; Anal. calcd for C₁₈H₁₅N₅O (317.34): C, 68.13; H, 4.76; N, 22.07; Found: C, 68.31; H, 4.81; N, 22.23 %.

Synthesis of 2-amino-4-(2,5-dimethoxyphenyl)-1,4-dihydrobenzo [4,5]imidazo[1,2-*a*]pyrimidine-3-carbonitrile (1e)



To the mixture of 2-aminobenzimidazole (1 mmol), malononitrile (1 mmol) and 2,5-dimethoxybenzaldehyde (1 mmol) in ethylene glycol (5 mL), SSA (42.6 mg, 0.11 mol %) was added. The mixture was heated at 50 °C for 15 minutes. After completion of the reaction (TLC), 10 mL EtOAc was added to the reaction mixture and the catalyst was recovered by filtration. The organic layer was dried over Na₂SO₄; the solvent was evaporated and purified by recrystallization from ethanol to give compounds **1e**. Yield 97%; m.p. 223 °C; IR: ν/cm^{-1} : 3418, 3310, 3137 (NH₂ & NH), 2927 (CH_{-aliph}), 2179 (C≡N), 1690 (C=N); ¹H NMR (400 MHz, DMSO): δ/ppm = 3.60 (s, 3H, OCH₃), 3.64 (s, 3H, OCH₃), 5.34 (s, 1H, CH), 6.66 (m, 1H, Ar-H), 6.73 (br, 2H, NH₂), 6.83 (m, 1H, Ar-H), 6.93 (m, 1H, Ar-H), 7.00 (m, 1H, Ar-H), 7.12 (m, 1H, Ar-H), 7.23 (m, 1H, Ar-H), 7.63 (m, 1H, Ar-H), 8.28 (br, 1H, NH); ¹³C NMR (101 MHz, DMSO): 49.5 (CH), 55.7 (OCH₃), 56.3 (OCH₃), 61.8, 112.7, 112.8, 112.9, 114.0, 116.5, 119.5, 120.3, 123.7, 129.8, 131.8, 144.1, 149.9, 151.0, 152.8, 153.4; Anal. calcd for C₁₉H₁₇N₅O₂ (347.37): C, 65.69; H, 4.93; N, 20.16; Found: C, 65.81; H, 5.01; N, 20.32 %.

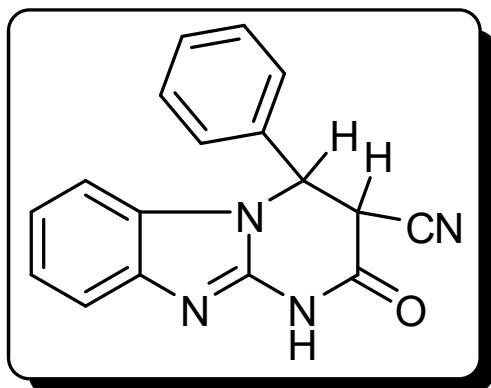
Synthesis of 2-amino-4-(naphthalene-2-yl)-1,4-dihydrobenzo[4,5]imidazo[1,2-a]pyrimidine-3-carbonitrile (**1f**)



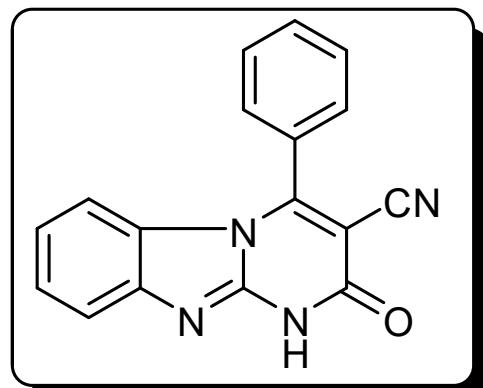
To the mixture of 2-aminobenzimidazole (1 mmol), malononitrile (1 mmol) and 2-naphthaldehyde (1 mmol) in ethylene glycol (5 mL), SSA (42.6 mg, 0.11 mol %) was added. The mixture was heated at 50 °C for 15 minutes. After completion of the reaction (TLC), 10 mL EtOAc was added to the reaction mixture and the catalyst was recovered by filtration. The organic layer was dried over Na₂SO₄; the solvent was evaporated and purified by recrystallization from ethanol to give compounds **1f**. Yield 97%; m.p. 237 °C (Lit. 228-229^{1,2}); IR: ν/cm^{-1} : 3430, 3327, 3137 (NH₂ & NH), 2917 (CH_{-aliph}), 2185 (C≡N), 1679 (C=N); ¹H NMR (400 MHz, DMSO): δ/ppm = 5.44 (s, 1H, CH), 6.89 (br, 2H, NH₂), 7.02 (t, 1H, Ar-H),

7.12 (t, 1H, Ar-H), 7.24 (d, 1H, Ar-H), 7.50 (m, 3H, Ar-H), 7.68 (d, 1H, Ar-H), 7.80 (s, 1H, Ar-H), 7.92 (m, 3H, Ar-H), 8.73 (br, 1H, NH); ^{13}C NMR (101 MHz, DMSO): 54.1, 62.3, 112.9, 116.6, 119.7, 120.4, 123.8, 124.9, 125.1, 126.7, 127.0, 128.1, 128.4, 129.2, 129.8, 133.1, 140.6, 144.1, 149.7, 152.2; Anal. calcd for $\text{C}_{21}\text{H}_{15}\text{N}_5$ (337.38): C, 74.76; H, 4.48; N, 20.76; Found: C, 74.54; H, 4.52; N, 20.58 %.

Synthesis of 2-oxo-4-phenyl-1,2,3,4-tetrahydrobenzo[4,5]imidazo [1,2-*a*]pyrimidine-3-carbonitrile (2a) and 2-oxo-4-phenyl-1,2-dihydropyrimido[1,2-*a*] benzimidazole-3-carbonitrile (3a)



2a



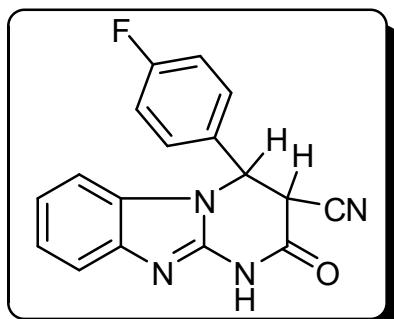
3a

To the mixture of 2-aminobenzimidazole (1 mmol), ethyl cyanoacetate (1 mmol) and benzaldehyde (1 mmol) in ethylene glycol (5 mL), SSA (42.6 mg, 0.11 mol %) was added. The mixture was heated at 120 °C for 35 min. After completion of the reaction (TLC), 10 mL EtOAc was added to the reaction mixture and the catalyst was recovered by filtration. The organic layer was dried over Na_2SO_4 ; the solvent was evaporated. The mixture of two products was separated by column chromatography on silica-gel using pet. ether/ethyl acetate (60/40) as eluent.

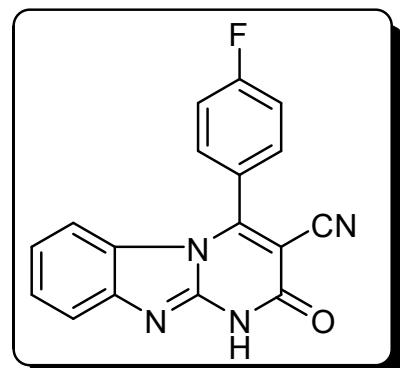
2-Oxo-4-phenyl-1,2,3,4-tetrahydrobenzo[4,5]imidazo [1,2-*a*]pyrimidine-3-carbonitrile (2a): Yield 50%; m.p. 290 °C; (Lit. 252 °C); IR: ν/cm^{-1} : 3439 (NH), 2926, 2852 (CH-aliph), 2227 (C≡N), 1665 (C=O), 1631 (C=N); Anal. Calcd. for $\text{C}_{17}\text{H}_{12}\text{N}_4\text{O}$ (288.30): C, 70.82; H, 4.20; N, 19.43; Found: C, 70.73; H, 3.89; N, 19.03 %.

2-oxo-4-phenyl-1,2-dihydropyrimido[1,2-*a*] benzimidazole-3-carbonitrile (3a): Yield 18%; m.p. >300 °C; IR: ν/cm^{-1} : 3289 (NH), 2209 (C≡N), 1698 (C=O), 1637 (C=N); ^1H NMR (400 MHz, DMSO): δ/ppm = 7.11 (m, 1H, Ar-H), 7.25 (m, 2H, Ar-H), 7.41 (m, 1H, Ar-H), 7.53 (m, 2H, Ar-H), 7.60 (m, 1H, Ar-H), 7.87 (br, 1H, NH); 8.40 (d, 1H, J = 6.8 Hz, Ar-H); ^{13}C NMR (101 MHz, DMSO): 111.8, 115.6, 116.0, 121.4, 122.4, 125.4, 128.6 (2C), 129.0 (2C), 130.4, 132.5, 138.7, 152.3, 153.2, 161.0, 166.9; Anal. Calcd. for $\text{C}_{17}\text{H}_{10}\text{N}_4\text{O}$ (286.29): C, 71.32; H, 3.52; N, 19.57; Found: C, 71.41; H, 3.47; N, 19.44 %.

Synthesis of 4-(4-fluorophenyl)-2-oxo-1,2,3,4-tetrahydrobenzo[4,5] imidazo[1,2-*a*]pyrimidine-3-carbonitrile (2b) and 4-(4-fluorophenyl)-2-oxo-1,2-dihydropyrimido[1,2-*a*]benzimidazole-3-carbonitrile (3b)



2b



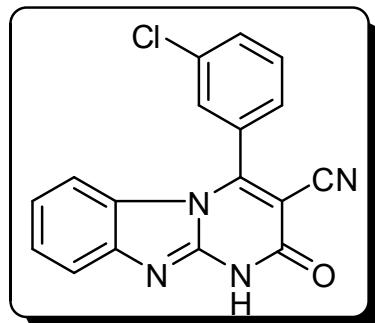
3b

To the mixture of 2-aminobenzimidazole (1 mmol), ethyl cyanoacetate (1 mmol) and 4-fluorobenzaldehyde (1 mmol) in ethylene glycol (5 mL), SSA (42.6 mg, 0.11 mol %) was added. The mixture was heated at 120 °C for 35 min. After completion of the reaction (TLC), 10 mL EtOAc was added to the reaction mixture and the catalyst was recovered by filtration. The organic layer was dried over Na₂SO₄; the solvent was evaporated. The mixture of two products was separated by column chromatography on silica-gel using pet. ether/ethyl acetate (60/40) as eluent.

4-(4-Fluorophenyl)-2-oxo-1,2,3,4-tetrahydrobenzo[4,5] imidazo[1,2-*a*]pyrimidine-3-carbonitrile (2b): Yield 44%; m.p. 288 °C; IR: v/cm⁻¹: 3353 (NH), 2962, 2887 (CH-aliph), 2224 (C≡N), 1681 (C=O), 1603 (C=N); ¹H NMR (400 MHz, DMSO): δ/ppm = 5.91 (d, 1H, J = 8.0 Hz, C₃-H), 6.88 (t, 1H, J = 7.6 Hz, Ar-H), 7.05 (m, 1H, Ar-H), 7.25 (t, 1H, J = 7.6 Hz, Ar-H), 7.34 (m, 1H, Ar-H), 7.50 (d, 1H, J = 8.0 Hz, C₄-H), 7.61 (m, 2H, Ar-H), 7.77 (br, 1H, NH), 7.86 (m, 2H, Ar-H); ¹³C NMR (101 MHz, DMSO): 95.5, 111.9 (2C), 116.4, 117.4, 120.9, 121.4 (2C), 125.7, 128.3, 131.6, 134.7, 141.4, 151.9, 153.9, 154.8, 164.2; Anal. Calcd for C₁₇H₁₁FN₄O (306.29): C, 66.66; H 3.62; N, 18.29; Found: C, 66.63; H 3.42; N, 18.32 %.

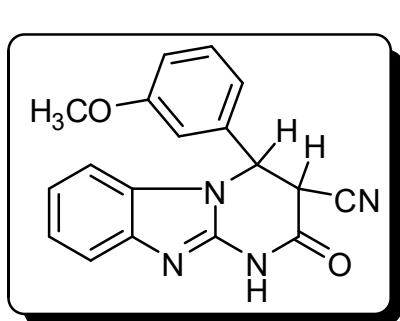
4-(4-Fluorophenyl)-2-oxo-1,2-dihydropyrimido[1,2-*a*]benzimidazole-3-carbonitrile (3b): Yield 19%; m.p. > 300 °C; IR: v/cm⁻¹: 3301 (NH), 2210 (C≡N), 1698 (C=O), 1635 (C=N); ¹H NMR (400 MHz, DMSO): δ/ppm = 6.80-7.70 (m, 7H, Ar-H), 7.94 (br, 1H, NH), 8.38 (d, 1H, J= 6.0 Hz, Ar-H); Anal. Calcd. for C₁₇H₉FN₄O (304.28): C, 67.10; H, 2.98; N, 18.41; Found: C, 67.23; H, 3.01; N, 18.32 %.

Synthesis of 4-(3-chlorophenyl)-2-oxo-1,2-dihydrobenzo[4,5]imidazo[1,2-*a*]pyrimidine-3-carbonitrile (3c)

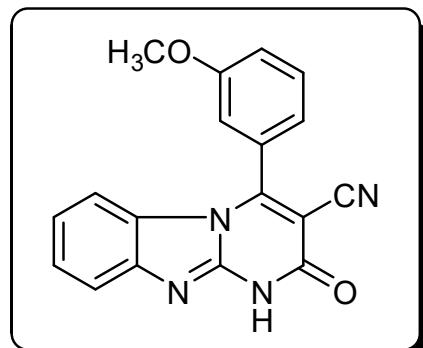


To the mixture of 2-aminobenzimidazole (1 mmol), ethyl cyanoacetate (1 mmol) and 3-chlorobenzaldehyde (1 mmol) in ethylene glycol (5 mL), SSA (42.6 mg, 0.11 mol %) was added. The mixture was heated at 120 °C for 35 min. After completion of the reaction (TLC), 10 mL EtOAc was added to the reaction mixture and the catalyst was recovered by filtration. The organic layer was dried over Na₂SO₄; the solvent was evaporated. The mixture of two products was separated by column chromatography on silica-gel using pet. ether/ethyl acetate (60/40) as eluent. Yield 83%; m.p. 342 °C; IR: ν/cm^{-1} : 3362 (NH), 2208 (C≡N), 1698 (C=O), 1647 (C=N); ¹H NMR (400 MHz, DMSO): δ/ppm = 7.17 (m, 1H, Ar-H), 7.31 (m, 2H, Ar-H), 7.44 (m, 1H, Ar-H), 7.50-7.65 (m, 2H, Ar-H), 7.86 (m, 1H, Ar-H), 8.22 (br, 1H, NH), 8.40 (d, 1H, J = 8.4 Hz, Ar-H); ¹³C NMR (101 MHz, DMSO): 81.2, 111.8, 115.5, 119.0, 122.0, 123.1, 125.8, 127.5, 128.7, 130.7, 133.4, 140.4, 151.5, 152.5, 160.5, 165.4; Anal. Calcd. for C₁₇H₉ClN₄O (320.73): C, 63.66; H, 2.83; N, 17.47; Found: C, 63.73; H, 2.79; N, 17.54 %.

Synthesis of 4-(3-methoxyphenyl)-2-oxo-1,2,3,4-tetrahydrobenzo[4,5]imidazo[1,2-*a*]pyrimidine-3-carbonitrile (2d) and 4-(3-methoxyphenyl)-2-oxo-1,2-dihydropyrimido[1,2-*a*]benzimidazole-3-carbonitrile (3d)



2d



3d

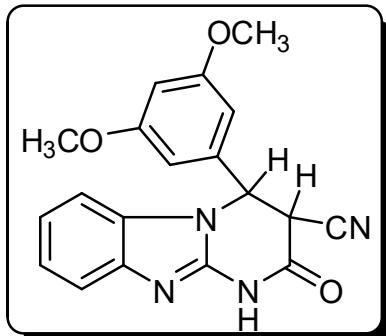
To the mixture of 2-aminobenzimidazole (1 mmol), ethyl cyanoacetate (1 mmol) and 3-methoxybenzaldehyde (1 mmol) in ethylene glycol (5 mL), SSA (42.6 mg, 0.11 mol %) was added. The mixture was heated at 120 °C for 50 min. After completion of the reaction (TLC),

10 mL EtOAc was added to the reaction mixture and the catalyst was recovered by filtration. The organic layer was dried over Na_2SO_4 ; the solvent was evaporated. The mixture of two products was separated by column chromatography on silica-gel using pet. ether/ethyl acetate (60/40) as eluent.

4-(3-Methoxyphenyl)-2-oxo-1,2,3,4-tetrahydrobenzo[4,5]imidazo[1,2-*a*]-pyrimidine-3-carbonitrile (2d): Yield 47%; m.p. 323 °C; IR: ν/cm^{-1} : 3432 (NH), 2923 (CH-aliph), 2232 (CN), 1675 (C=O), 1601 (C=N); ^1H NMR (400 MHz, DMSO): δ/ppm = 3.83 (s, 3H, OCH_3), 5.89 (d, 1H, J = 8.2 Hz, CH), 6.96 (t, 1H, J = 7.7 Hz), 7.10-7.40 (m, 7H, Ar-H + NH), 7.49 (d, 1H, J = 7.8 Hz, CH), 7.67 (t, 1H, J = 7.8 Hz, Ar-H); ^{13}C NMR (101 MHz, DMSO): 56.0, 113.2, 114.1, 115.2, 117.8, 120.5, 122.2, 126.2, 127.9, 130.9, 131.6, 148.9, 155.8, 160.2; Anal. Calcd for $\text{C}_{18}\text{H}_{14}\text{N}_4\text{O}_2$ (318.33): C, 67.91; H, 4.43; N, 17.60; Found: C, 68.07; H, 4.39; N, 17.72 %.

4-(3-Methoxyphenyl)-2-oxo-1,2-dihydropyrimido[1,2-*a*]benzimidazole-3-carbonitrile (3d): Yield 16%; m.p. 294 °C; IR: ν/cm^{-1} : 3301 (NH), 2204 (C≡N), 1694 (C=O), 1637 (C=N); ^1H NMR (400 MHz, DMSO): δ/ppm = 3.83 (s, 3H, OCH_3), 7.11 (m, 2H, Ar-H), 7.20 - 7.30 (m, 2H, Ar-H), 7.37 - 7.50 (m, 2H, Ar-H), 7.61 (d, 1H, J = 7.9 Hz, Ar-H), 8.03 (br, 1H, NH), 8.41 (d, 1H J = 7.9 Hz, Ar-H); ^{13}C NMR (101 MHz, DMSO): 55.7, 80.0, 111.8, 114.3, 115.6, 116.1, 119.5, 121.3, 122.5, 125.4, 127.9, 129.8, 132.3, 140.0, 152.2, 153.3, 159.4, 161.0, 166.6; Anal. Calcd for $\text{C}_{18}\text{H}_{12}\text{N}_4\text{O}_2$ (316.31): C, 68.35; H, 3.82; N, 17.71; Found: C, 68.43; H, 3.78; N, 17.58 %.

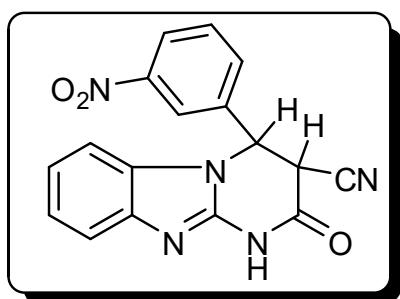
Synthesis of 4-(2,5-dimethoxyphenyl)-2-oxo-1,2,3,4-tetrahydrobenzo[4,5]imidazo[1,2-*a*]-pyrimidine-3-carbonitrile (2e)



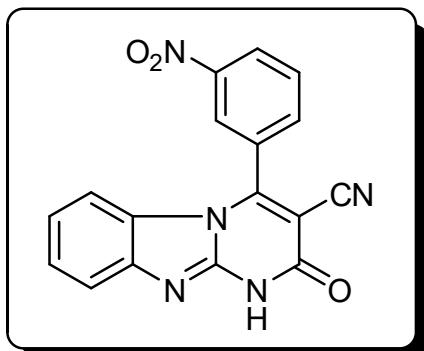
To the mixture of 2-aminobenzimidazole (1 mmol), ethyl cyanoacetate (1 mmol) and 2,5-dimethoxybenzaldehyde (1 mmol) in ethylene glycol (5 mL), SSA (42.6 mg, 0.11 mol %) was added. The mixture was heated at 120 °C for 80 min. After completion of the reaction (TLC), 10 mL EtOAc was added to the reaction mixture and the catalyst was recovered by filtration. The organic layer was dried over Na_2SO_4 ; the solvent was evaporated. The mixture of two products was separated by column chromatography on silica-gel using pet. ether/ethyl acetate (60/40) as eluent. Yield 51%; m.p. 320 °C; IR: ν/cm^{-1} : 3442 (NH), 2946

(CH_{-aliph}), 2230 (C≡N), 1663 (C=O); ¹H NMR (400 MHz, DMSO): δ/ppm = 3.69 (s, 3H, OCH₃), 3.78 (s, 3H, OCH₃), 6.07 (d, 1H, *J* = 8.2 Hz, CH), 7.02 (m, 2H, Ar-H), 7.27 (br, 1H, NH), 7.31-7.37 (m, 5H, Ar-H), 7.50 (d, 1H, *J* = 7.9 Hz, CH); ¹³C NMR (101 MHz, DMSO): 56.3, 57.0, 112.5, 114.3, 115.0, 115.6, 118.5, 119.2, 122.6, 126.5, 127.8, 150.6, 154.0; Anal. Calcd for C₁₉H₁₆N₄O₃ (348.36): C, 65.51; H, 4.63; N, 16.08; Found: C, 65.63; H, 4.69; N, 15.86 %.

Synthesis of 4-(3-nitrophenyl)-2-oxo-1,2,3,4-tetrahydrobenzo[4,5]imidazo[1,2-*a*]-pyrimidine-3-carbonitrile (2f) and 4-(3-nitrophenyl)-2-oxo-1,2-dihydropyrimido[1,2-*a*]benzimidazole-3-carbonitrile (3f)



2f



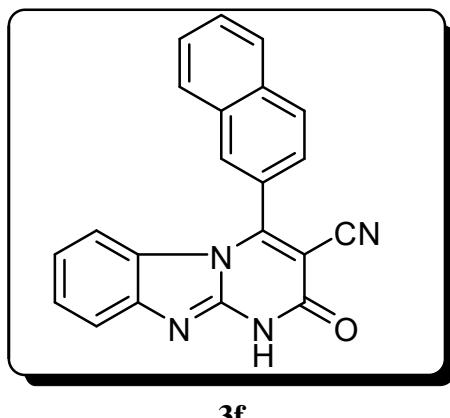
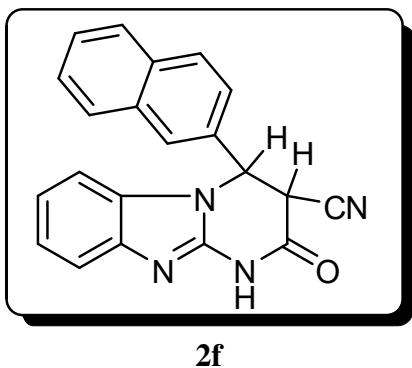
3f

To the mixture of 2-aminobenzimidazole (1 mmol), ethyl cyanoacetate (1 mmol) and 3-nitrobenzaldehyde (1 mmol) in ethylene glycol (5 mL), SSA (42.6 mg, 0.11 mol %) was added. The mixture was heated at 120 °C for 10 min. After completion of the reaction (TLC), 10 mL EtOAc was added to the reaction mixture and the catalyst was recovered by filtration. The organic layer was dried over Na₂SO₄; the solvent was evaporated. The mixture of two products was separated by column chromatography on silica-gel using pet. ether/ethyl acetate (60/40) as eluent.

4-(3-Nitrophenyl)-2-oxo-1,2,3,4-tetrahydrobenzo[4,5]imidazo[1,2-*a*]-pyrimidine-3-carbonitrile (2f): Yield 58%; >300 °C; IR: ν/cm^{-1} : 3434 (NH), 2911 (CH_{-aliph}), 2230 (CN), 1681 (C=O), 1607 (C≡N); ¹H NMR (400 MHz, DMSO): δ/ppm = 6.00-6.95 (m, 2H, CH & Ar-H), 7.10-7.40 (m, 6H, Ar-H + NH), 7.49 -7.67 (m, 3H, Ar-H); Anal. Calcd for C₁₇H₁₆N₅O₃ (333.30): C, 61.26; H, 3.33; N, 21.01; Found: C, 61.19; H, 3.28; N, 20.94 %.

4-(3-Nitrophenyl)-2-oxo-1,2-dihydropyrimido[1,2-*a*]benzimidazole-3-carbonitrile (3f): Yield 37%; >300 °C; IR: ν/cm^{-1} : 3312 (NH), 2208 (C≡N), 1671 (C=O), 1637 (C≡N); ¹H NMR (400 MHz, DMSO): δ/ppm = 7.20 - 7.50 (m, 6H, Ar-H), 7.61 (d, 1H, *J* = 7.9 Hz, Ar-H), 8.03 (br, 1H, NH), 8.41 (d, 1H *J* = 7.9 Hz, Ar-H); Anal. Calcd for C₁₇H₉N₅O₃ (331.29): C, 61.63; H, 2.74; N, 21.14; Found: C, 61.49; H, 2.68; N, 21.24 %.

Synthesis of 4-(naphthalene-2-yl)-2-oxo-1,2,3,4-tetrahydrobenzo[4,5]imidazo[1,2-*a*]-pyrimidine-3-carbonitrile (2g) and 4-(naphthalen-2-yl)-2-oxo-1,2-dihydropyrimido[1,2-*a*]benzimidazole-3-carbonitrile (3g)



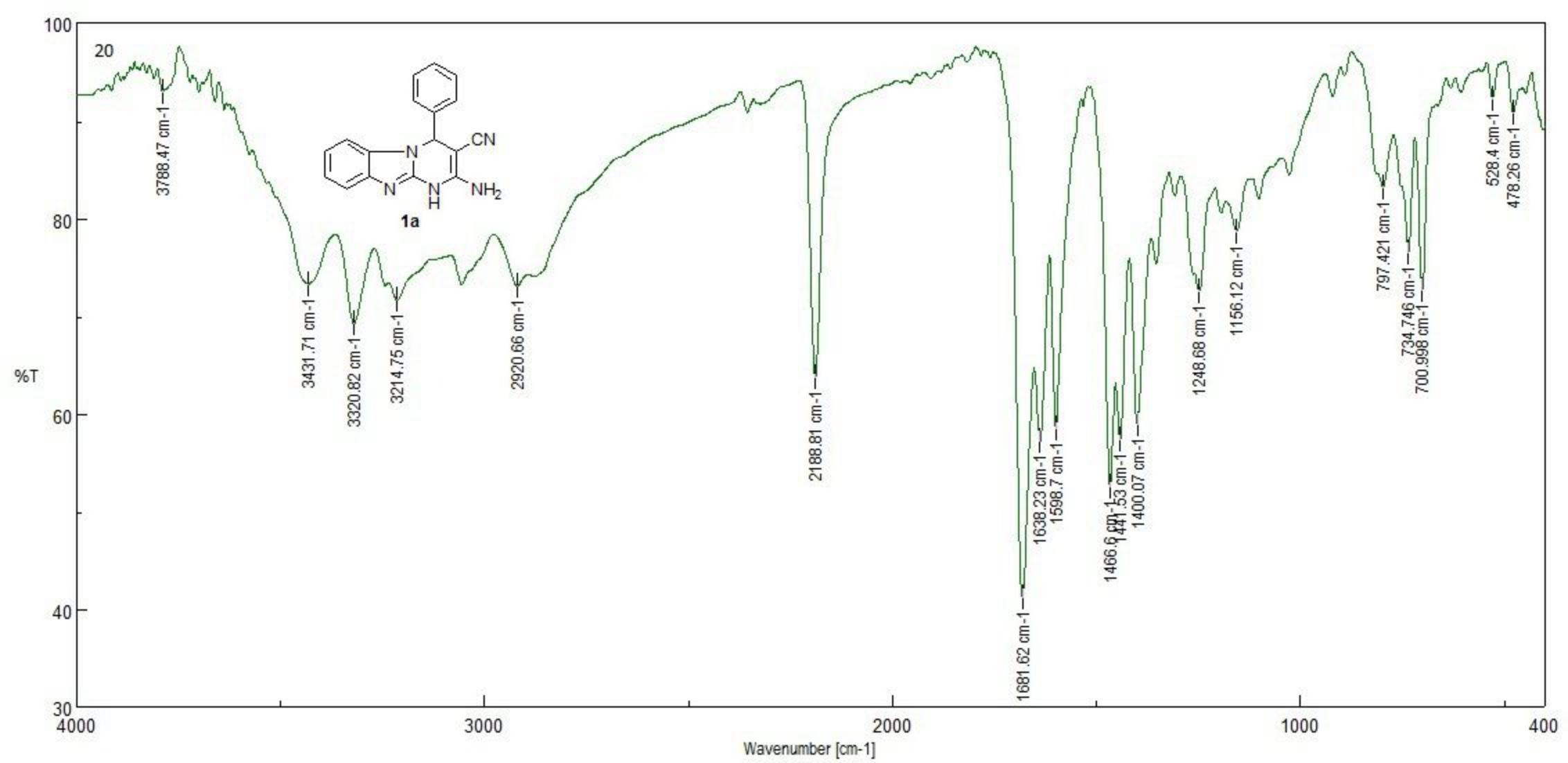
To the mixture of 2-aminobenzimidazole (1 mmol), ethyl cyanoacetate (1 mmol) and 2-naphthaldehyde (1 mmol) in ethylene glycol (5 mL), SSA (42.6 mg, 0.11 mol %) was added. The mixture was heated at 120 °C for 60 min. After completion of the reaction (TLC), 10 mL EtOAc was added to the reaction mixture and the catalyst was recovered by filtration. The organic layer was dried over Na₂SO₄; the solvent was evaporated. The mixture of two products was separated by column chromatography on silica-gel using pet. ether/ethyl acetate (60/40) as eluent.

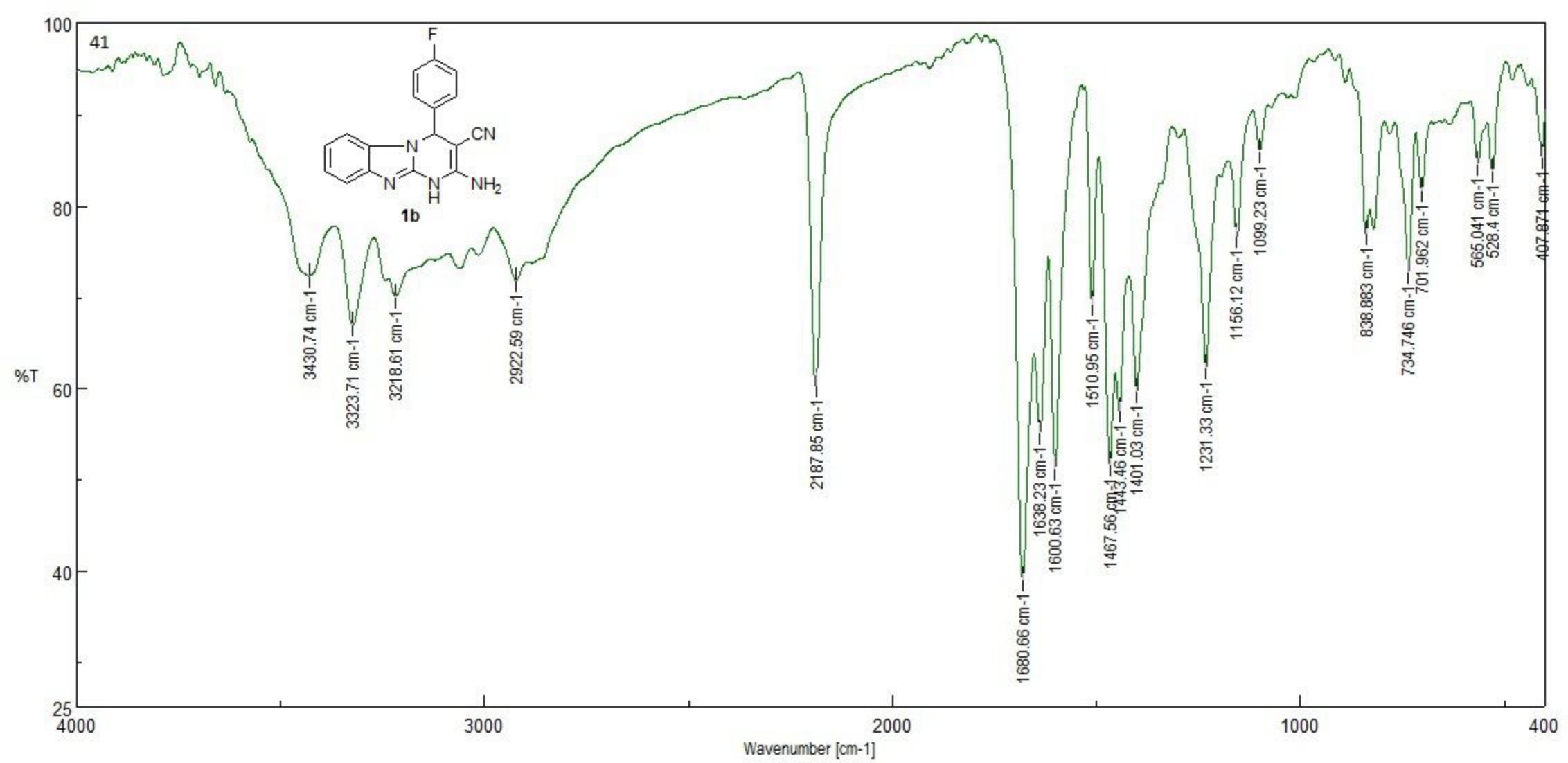
4-(Naphthalene-2-yl)-2-oxo-1,2,3,4-tetrahydrobenzo[4,5]imidazo[1,2-*a*]-pyrimidine-3-carbonitrile (2f): Yield 73%; m.p. 309 °C; IR: ν/cm^{-1} : 3255 (NH), 2927 (CH-aliph), 2216 (C≡N), 1684 (C=O), 1615 (C=N); ¹H NMR (400 MHz, DMSO): δ/ppm = 5.81 (d, 1H, *J* = 7.4 Hz, CH), 6.72 (m, 1H, Ar-H), 7.06 (m, 2H, Ar-H), 7.21 (m, 1H, Ar-H), 7.37 (m, 2H, Ar-H), 7.50 (d, 1H, *J* = 6.9 Hz, CH), 7.65-7.81 (m, 2H, Ar-H), 8.12 (m, 2H, Ar-H), 8.27 (d, 1H, *J* = 7.6 Hz, Ar-H), 8.39 (br, 1H, NH); Anal. Calcd for C₂₁H₁₄N₄O (338.36): C, 74.54; H 4.17; N, 16.56; Found: C, 74.33; H 4.02; N, 16.22 %.

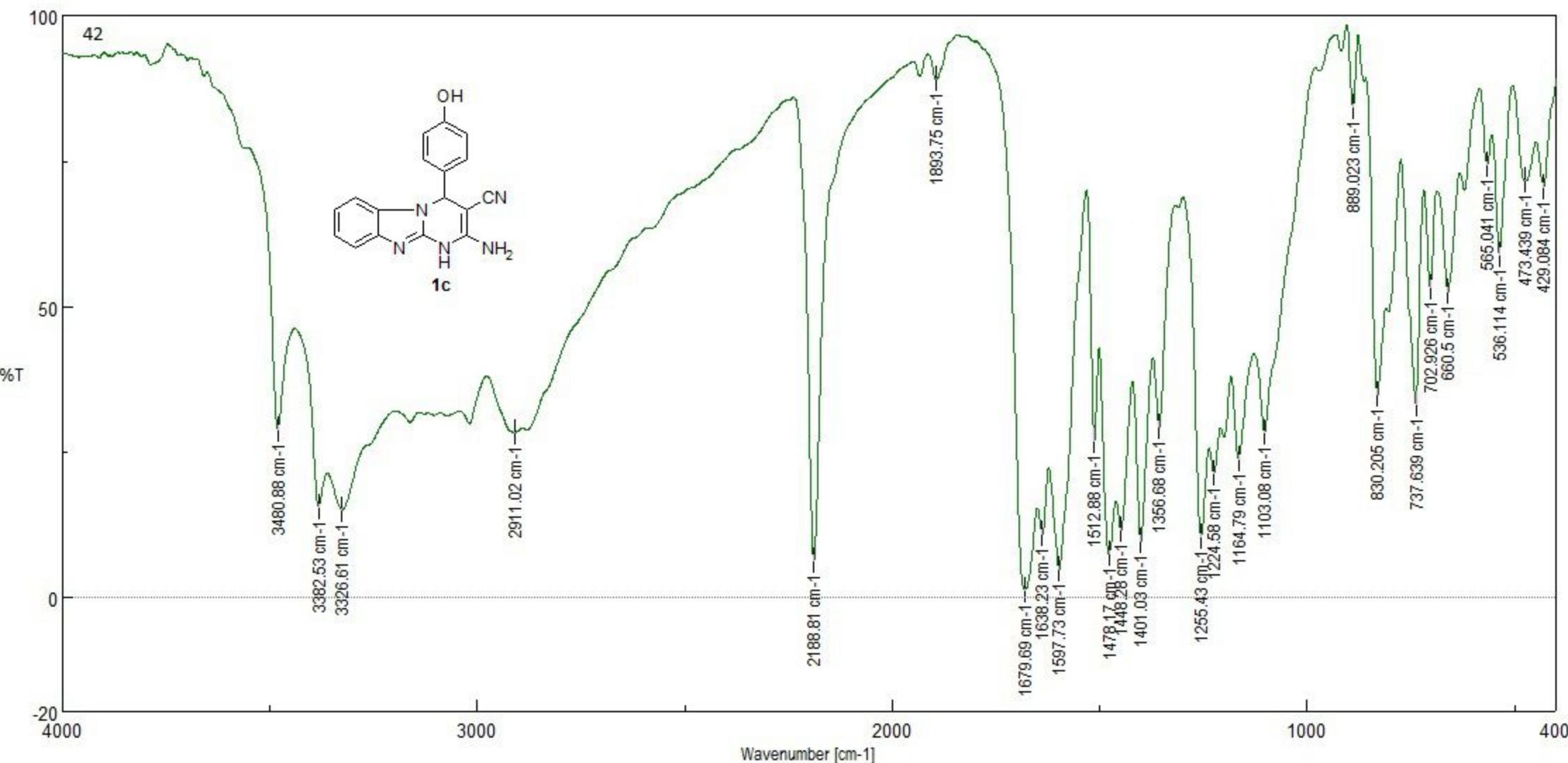
4-(Naphthalen-2-yl)-2-oxo-1,2-dihydropyrimido[1,2-*a*]benzimidazole-3-carbonitrile (3f): Yield 9%; m.p. 290 °C; IR: ν/cm^{-1} : 3302 (NH), 2209 (C≡N), 1688 (C=O), 1644 (C=N); ¹H NMR (400 MHz, DMSO): δ/ppm = 6.69 (m, 1H, Ar-H), 6.89 (m, 1H, Ar-H), 7.00-8.00 (m, 8H, Ar-H), 8.21(br., 1H, NH), 8.45 (m, 1H, Ar-H); ¹³C NMR (101 MHz, DMSO): 111.8, 115.7, 115.8, 121.6, 122.5, 125.5, 126.4, 126.8, 126.9, 127.1, 127.8, 128.1, 128.8, 129.2, 132.4, 132.7, 133.9, 136.0, 152.2, 160.9, 166.9; Anal. Calcd for C₂₁H₁₂N₄O (336.35): C, 74.99; H, 3.60; N, 16.66; Found: C, 74.78; H, 3.54; N, 16.73 %.

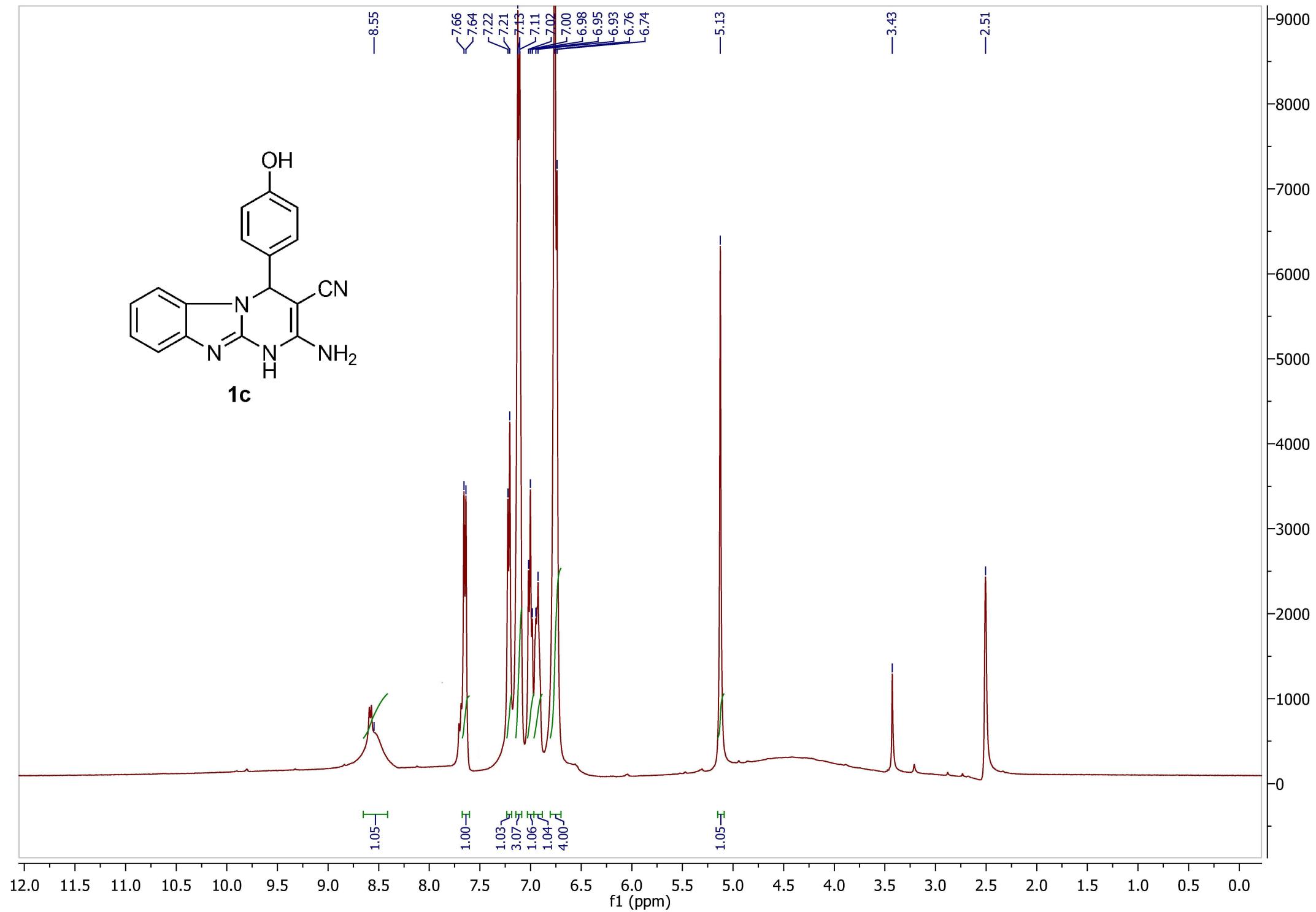
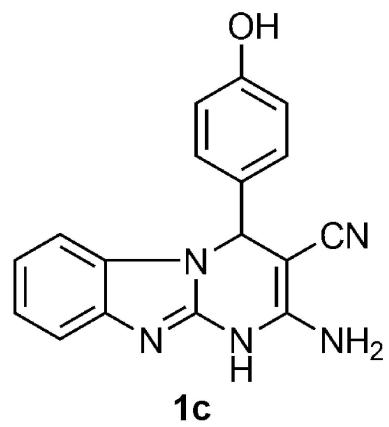
References

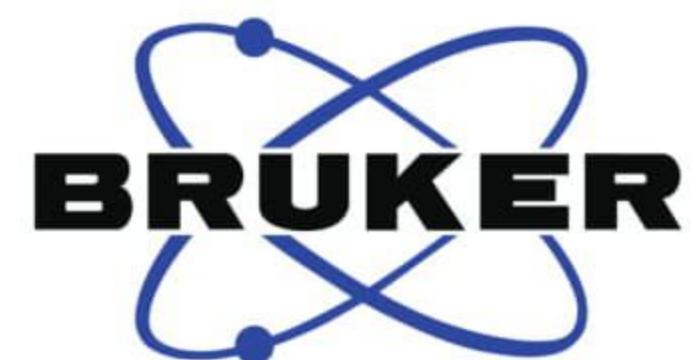
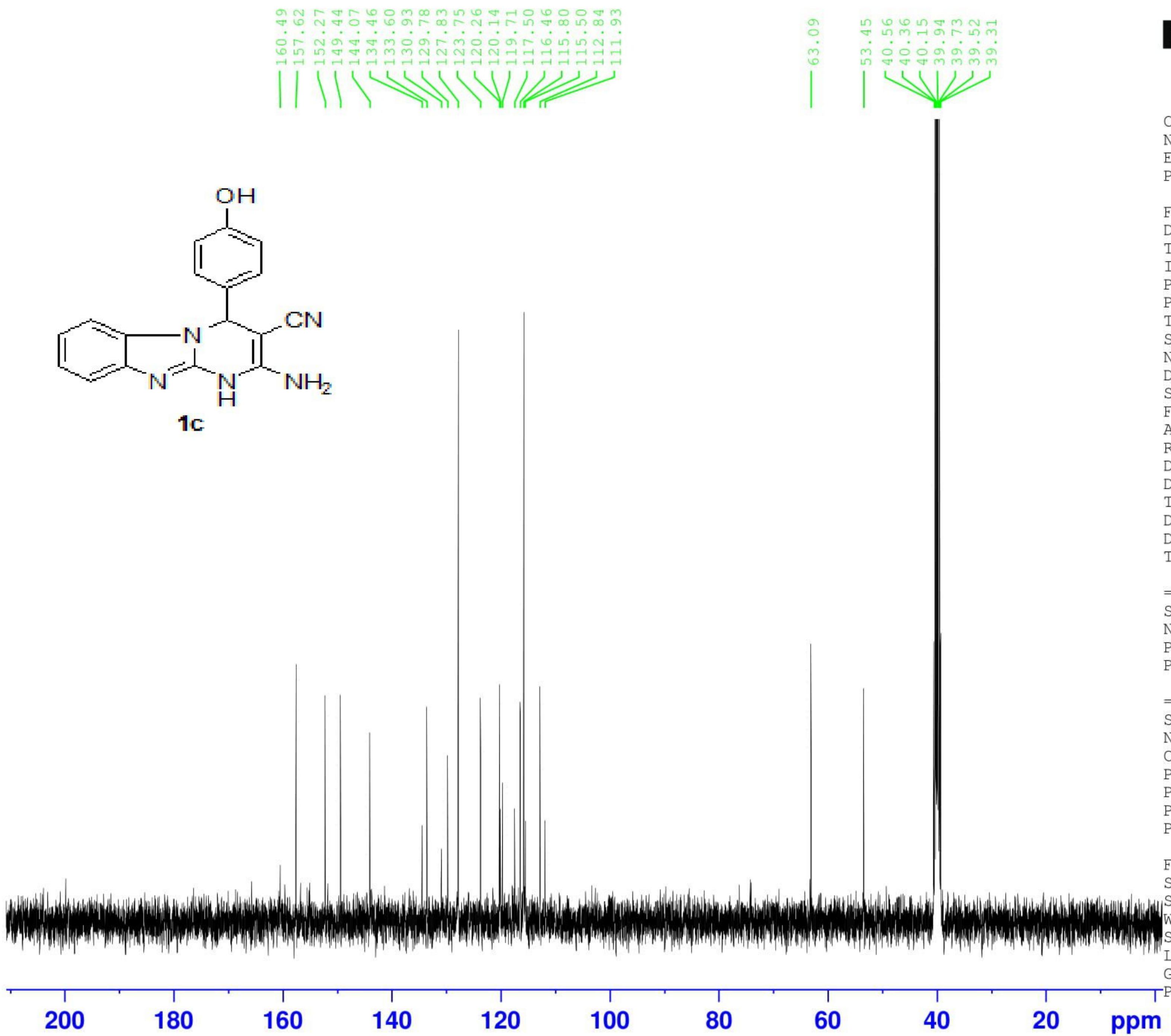
- [1] M. Makhsoos, F. Shirini, M. Seddighi, M. Mazloumi, polycyclic aromatic compounds, **2018**, *in press*
- [2] M. Abedini, F. Shirini, M. Mousapour, O.G. Jolodar, *Res. Chem. Intermed.*, **2016**, 42, 6221.
- [3] Shirini, F.; Seddighi, M.; Goli-Jolodar O.; *J. Iranian Chem. Soc.* **2016**, 13 (11), 2013-2018











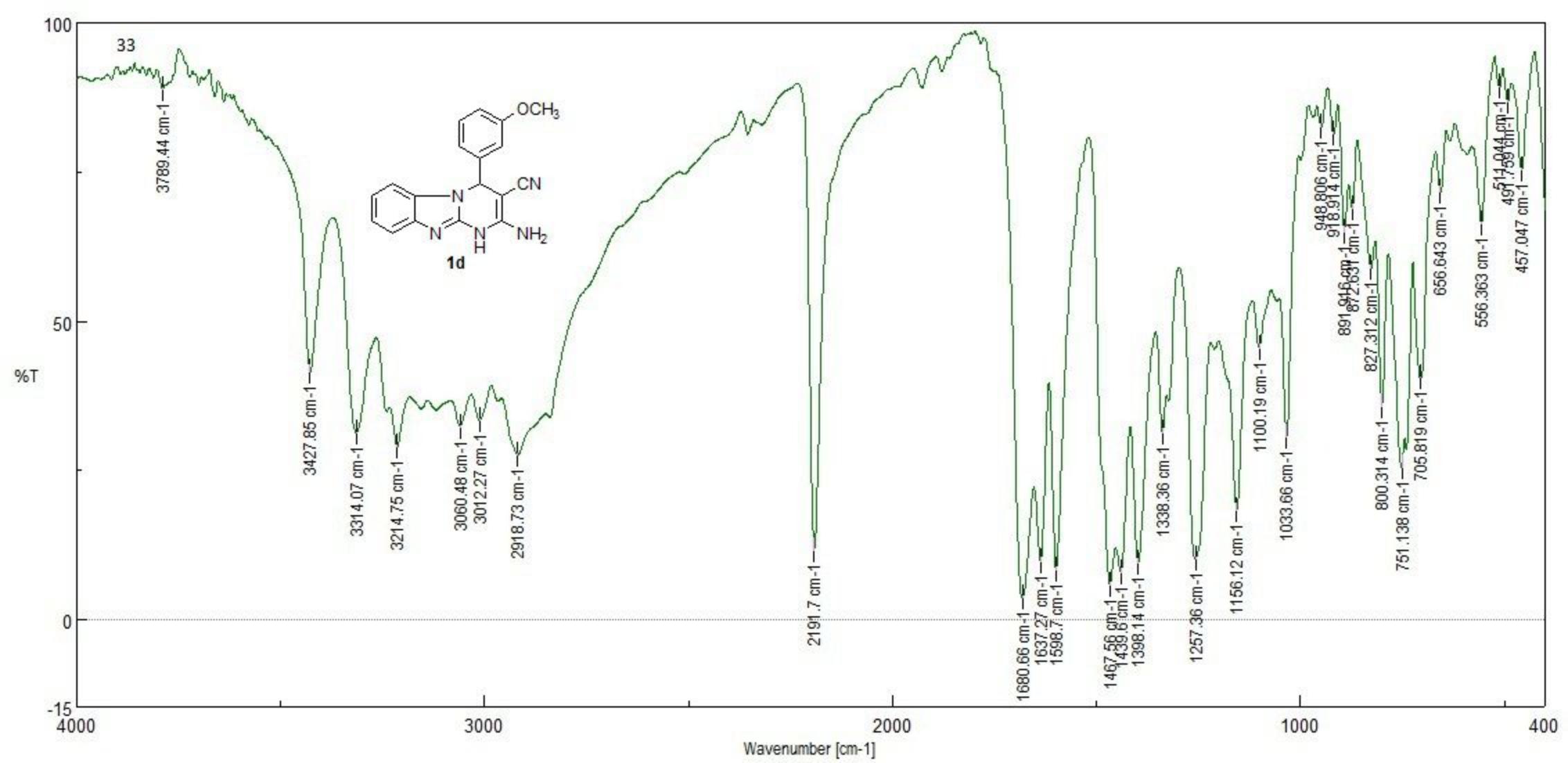
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EXPNO 2
PROCNO 1

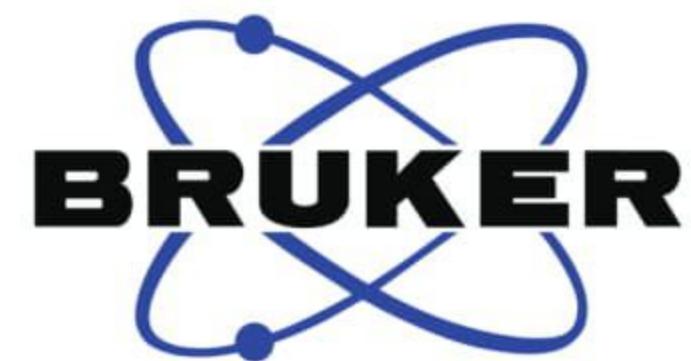
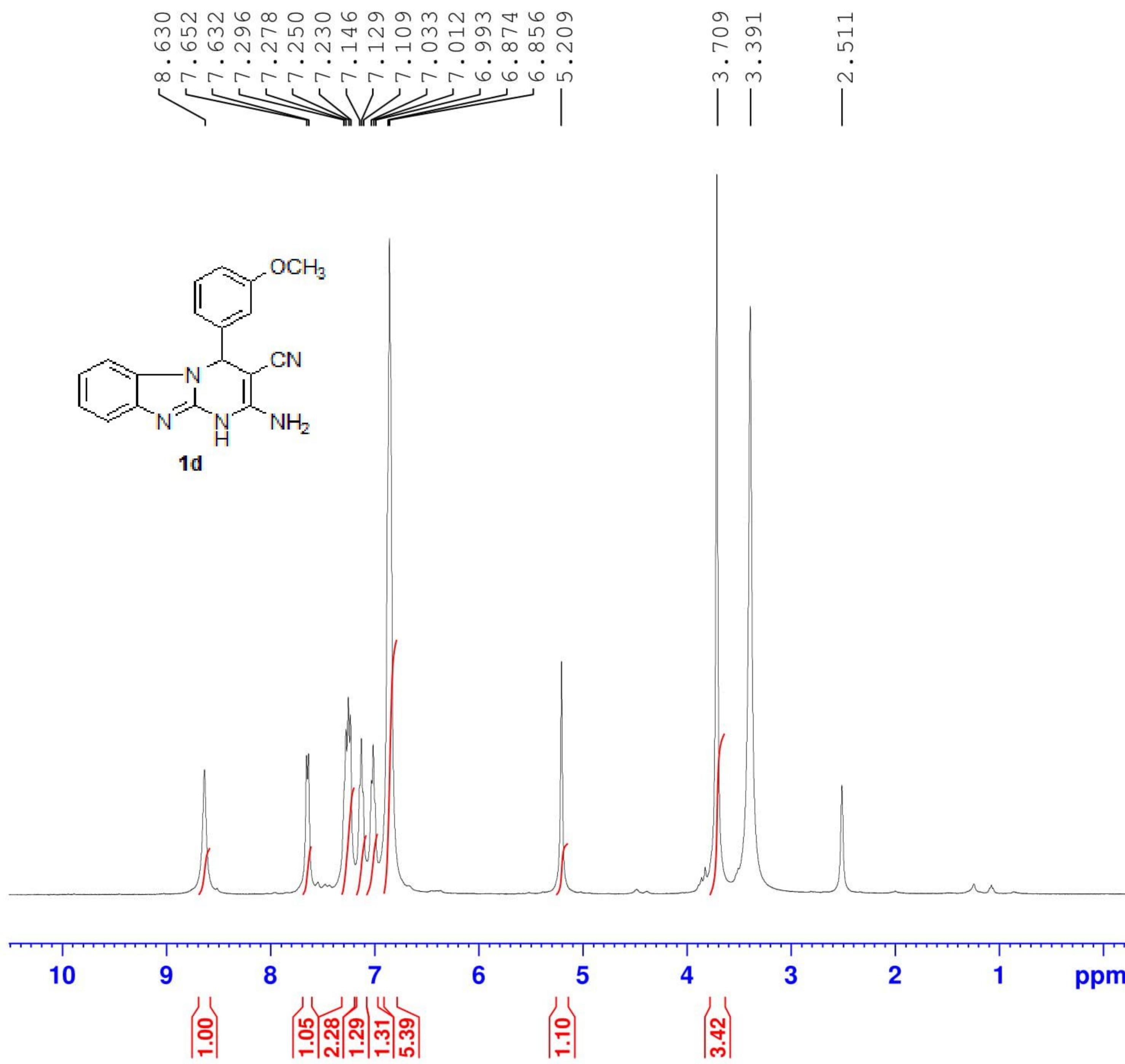
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Time 11.19
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TD 65536
SOLVENT DMSO
NS 295
DS 4
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3631488 sec
RG 205.37
DW 20.800 usec
DE 6.50 usec
TE 299.0 K
D1 2.0000000 sec
D11 0.0300000 sec
TD0 1

===== CHANNEL f1 =====
SFO1 100.6278588 MHz
NUC1 13C
P1 10.00 usec
PLW1 47.00000000 W

===== CHANNEL f2 =====
SFO2 400.1516006 MHz
NUC2 1H
CPDPRG[2] waltz16
PCPD2 90.00 usec
PLW2 18.00000000 W
PLW12 0.34722000 W
PLW13 0.28125000 W

F2 - Processing parameters
SI 32768
SF 100.6177975 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40



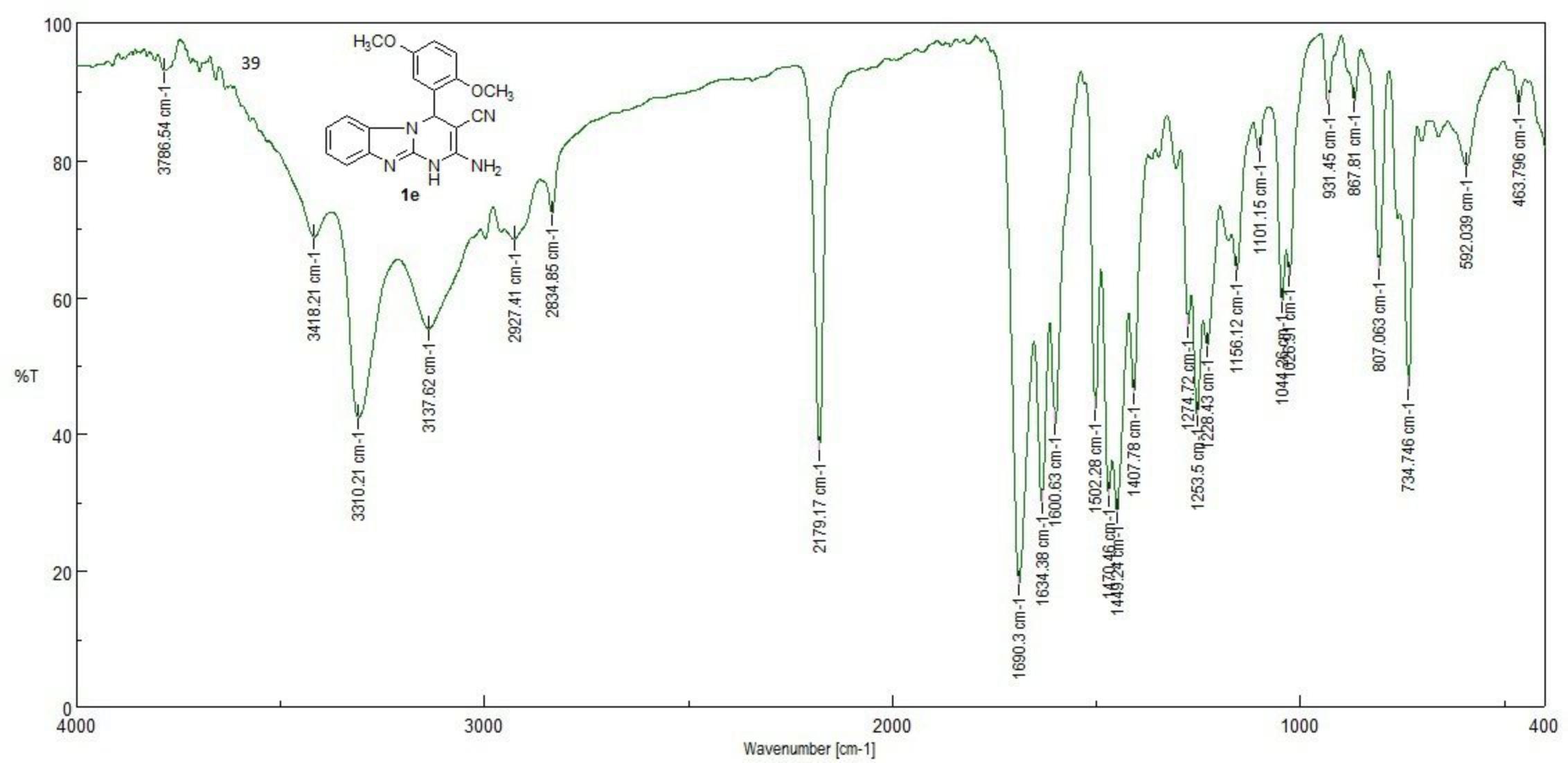


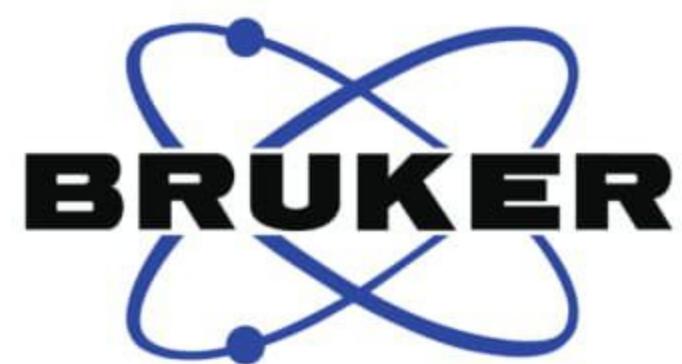
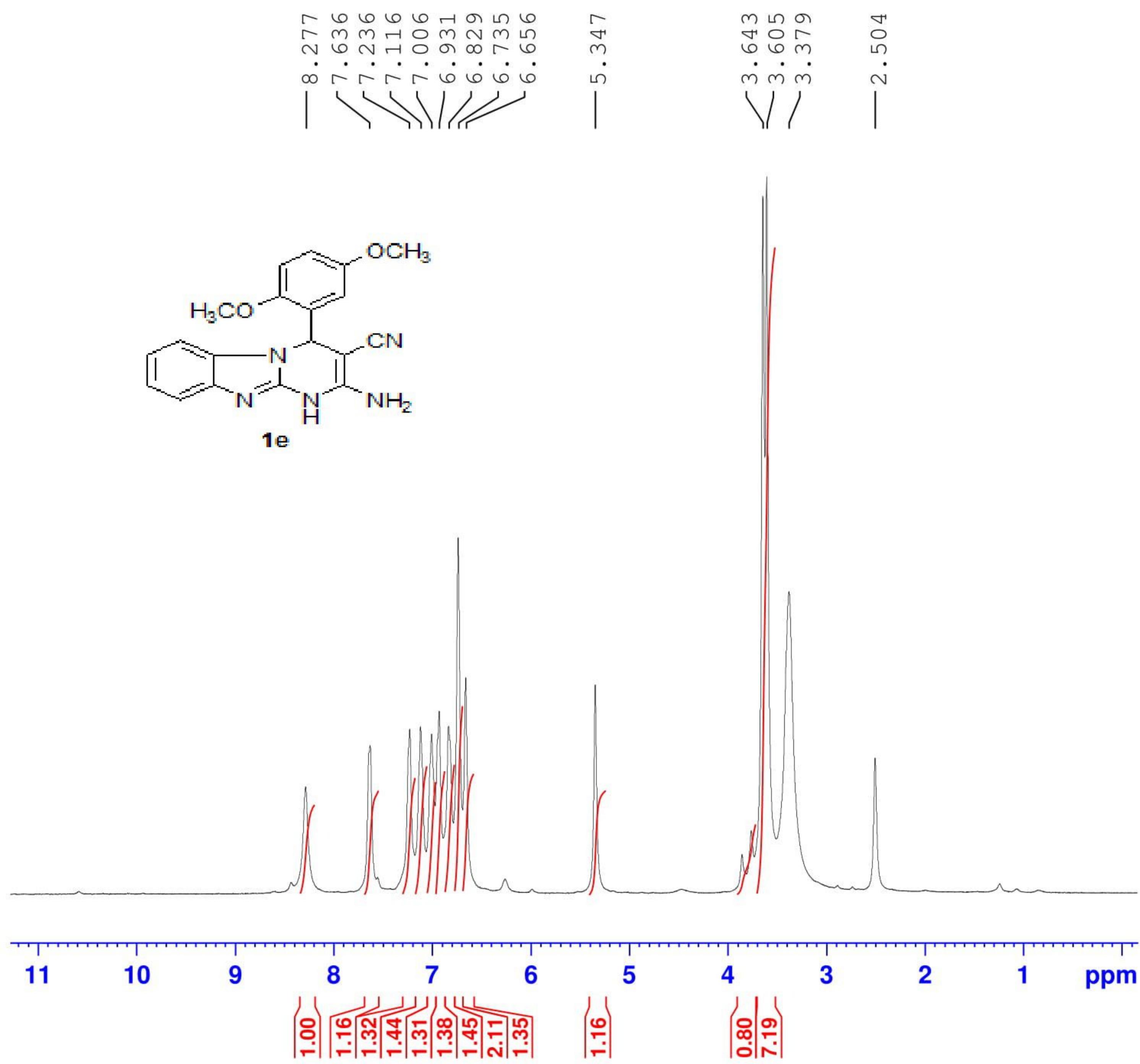
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 NAME nagwa-33
 EXPNO 1
 PROCNO 1

F2 - Acquisition Parameters
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 PULPROG zg30
 TD 65536
 SOLVENT DMSO
 NS 59
 DS 2
 SWH 8012.820 Hz
 FIDRES 0.122266 Hz
 AQ 4.0894465 sec
 RG 205.37
 DW 62.400 usec
 DE 6.50 usec
 TE 299.0 K
 D1 1.0000000 sec
 TD0 1

===== CHANNEL f1 =====
 SFO1 400.1524711 MHz
 NUC1 1H
 P1 12.00 usec
 PLW1 18.00000000 W

F2 - Processing parameters
 SI 65536
 SF 400.1500000 MHz
 WDW EM
 SSB 0
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 PC 1.00



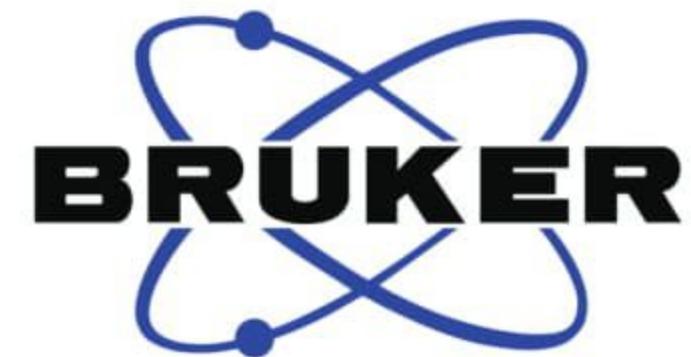
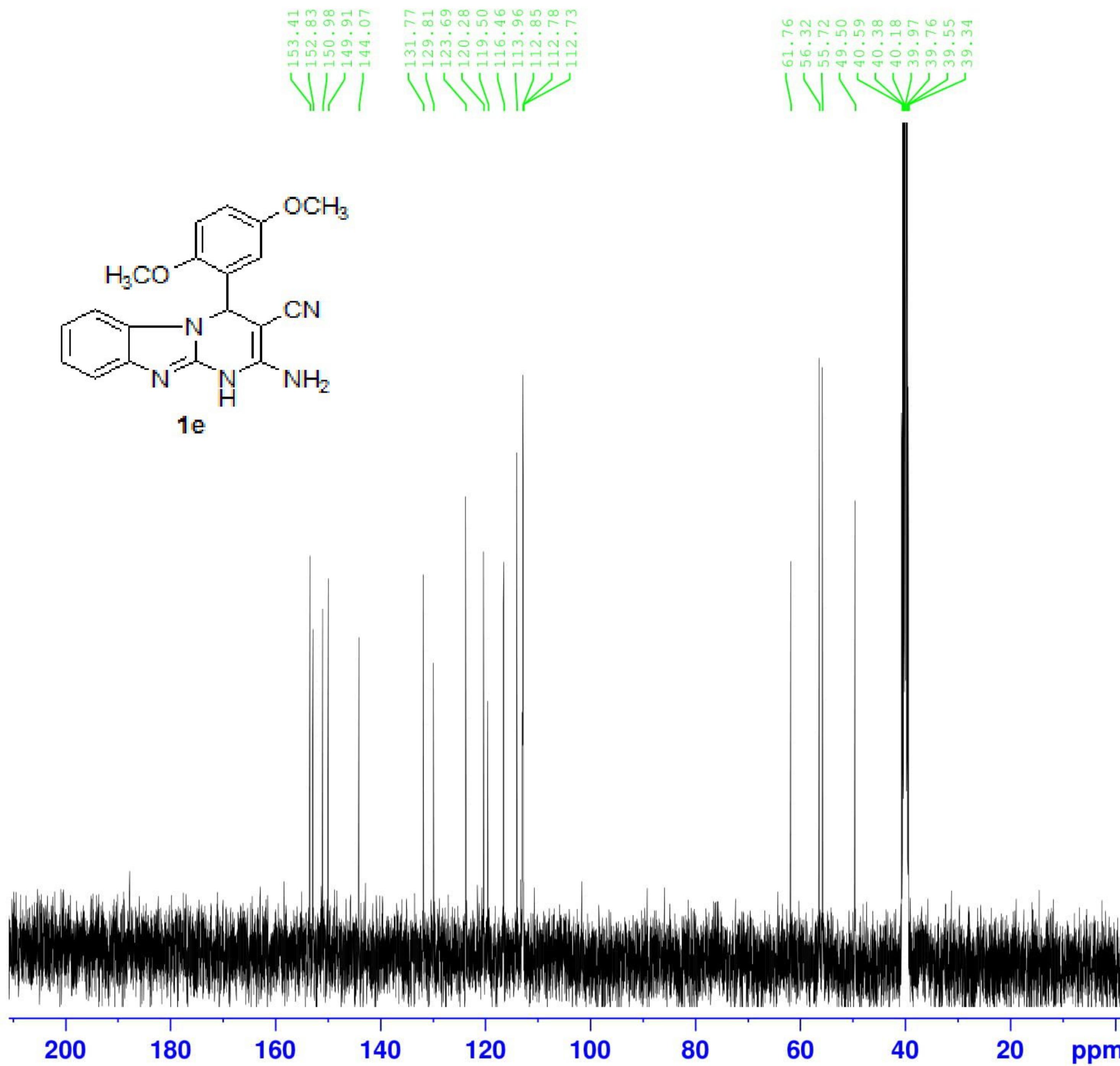


Current Data Parameters
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 EXPNO 1
 PROCNO 1

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 PULPROG zg30
 TD 65536
 SOLVENT DMSO
 NS 64
 DS 2
 SWH 8012.820 Hz
 FIDRES 0.122266 Hz
 AQ 4.0894465 sec
 RG 205.37
 DW 62.400 usec
 DE 6.50 usec
 TE 299.0 K
 D1 1.0000000 sec
 TD0 1

===== CHANNEL f1 =====
 SFO1 400.1524711 MHz
 NUC1 1H
 P1 12.00 usec
 PLW1 18.00000000 W

F2 - Processing parameters
 SI 65536
 SF 400.1500000 MHz
 WDW EM
 SSB 0
 LB 0.30 Hz
 GB 0
 PC 1.00



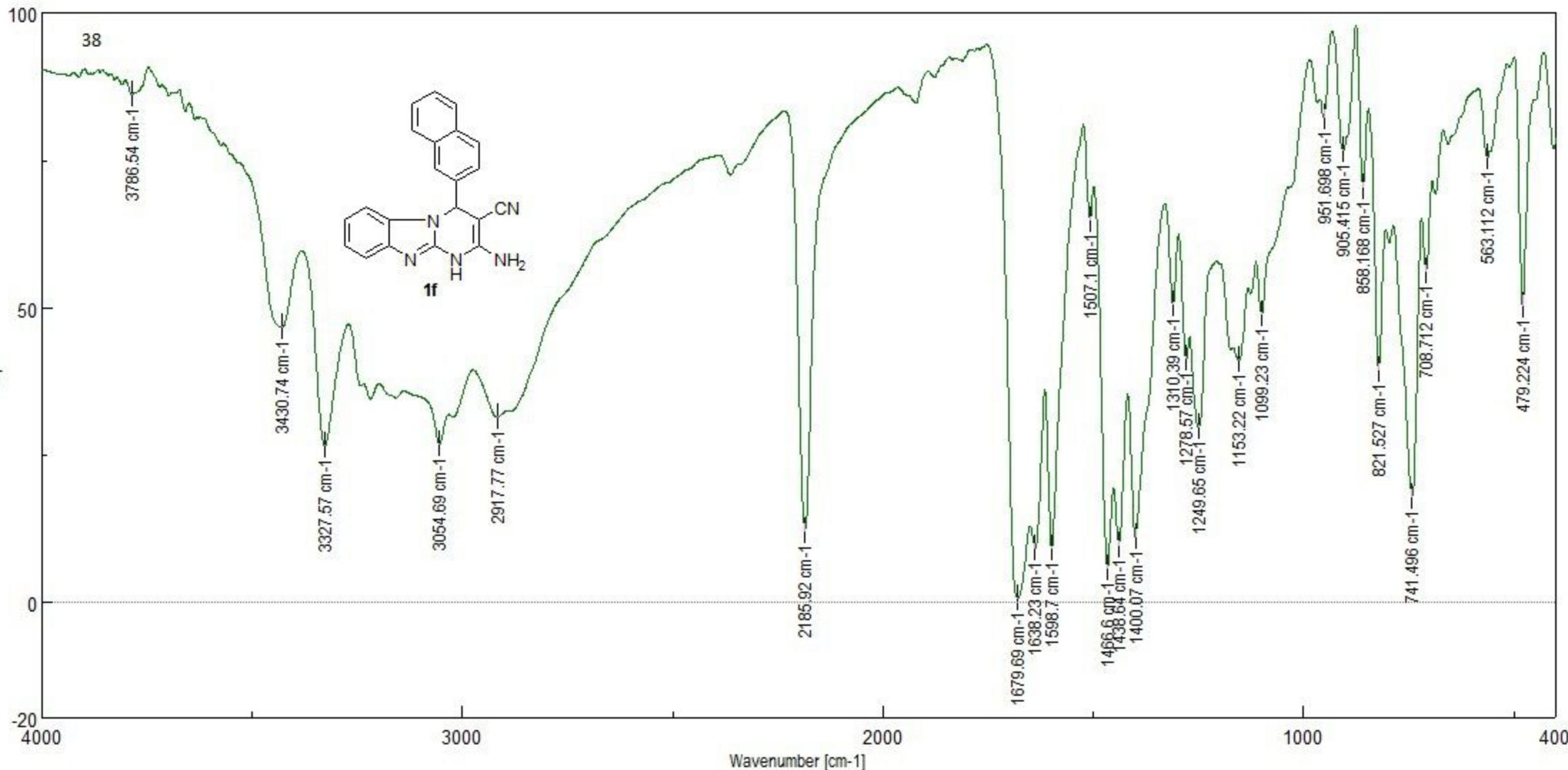
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 PROCNO 1

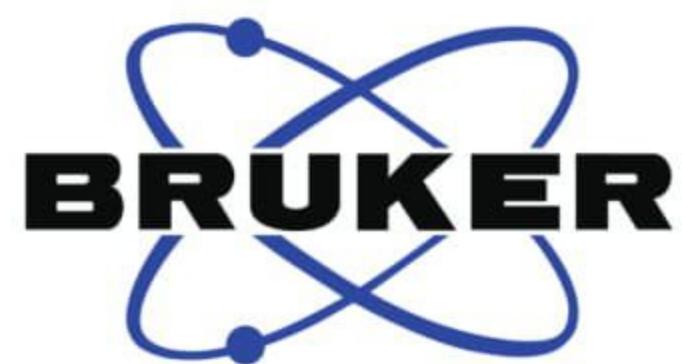
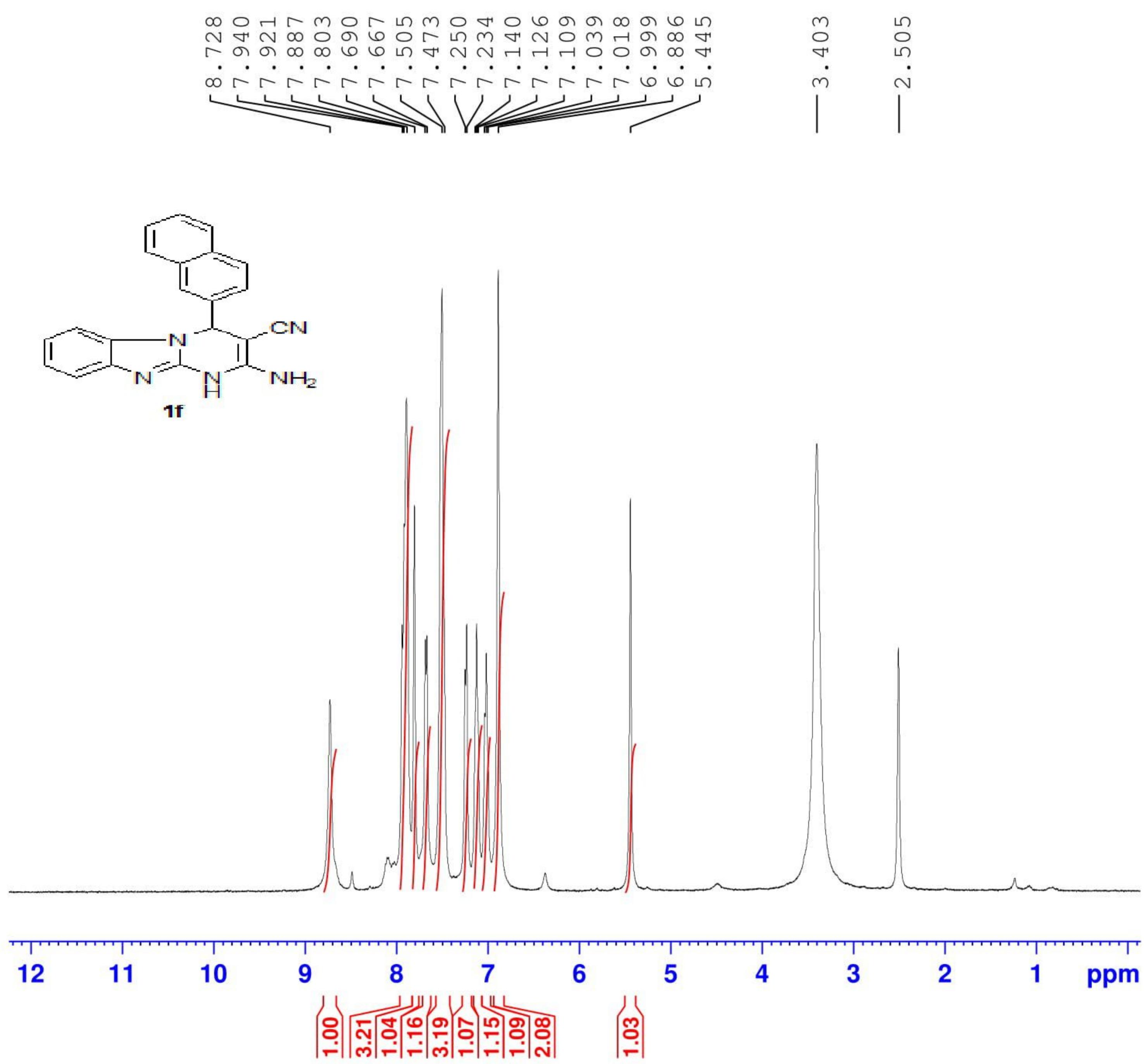
F2 - Acquisition Parameters
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 PULPROG zgpg30
 TD 65536
 SOLVENT DMSO
 NS 540
 DS 4
 SWH 24038.461 Hz
 FIDRES 0.366798 Hz
 AQ 1.3631488 sec
 RG 205.37
 DW 20.800 usec
 DE 6.50 usec
 TE 299.0 K
 D1 2.0000000 sec
 D11 0.0300000 sec
 TD0 1

===== CHANNEL f1 =====
 SFO1 100.6278588 MHz
 NUC1 13C
 P1 10.00 usec
 PLW1 47.00000000 W

===== CHANNEL f2 =====
 SFO2 400.1516006 MHz
 NUC2 1H
 CPDPRG[2] waltz16
 PCPD2 90.00 usec
 PLW2 18.00000000 W
 PLW12 0.34722000 W
 PLW13 0.28125000 W

F2 - Processing parameters
 SI 32768
 SF 100.6177975 MHz
 WDW EM
 SSB 0
 LB 1.00 Hz
 GB 0
 PC 1.40



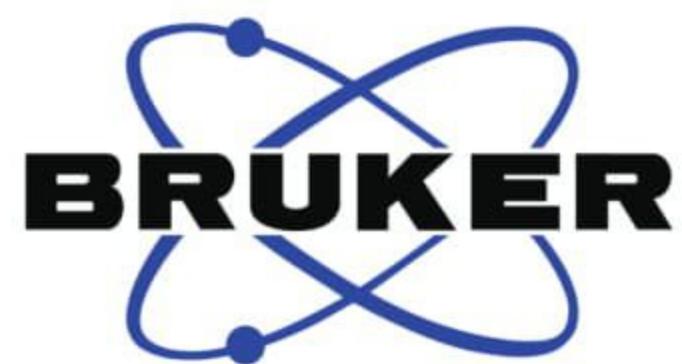
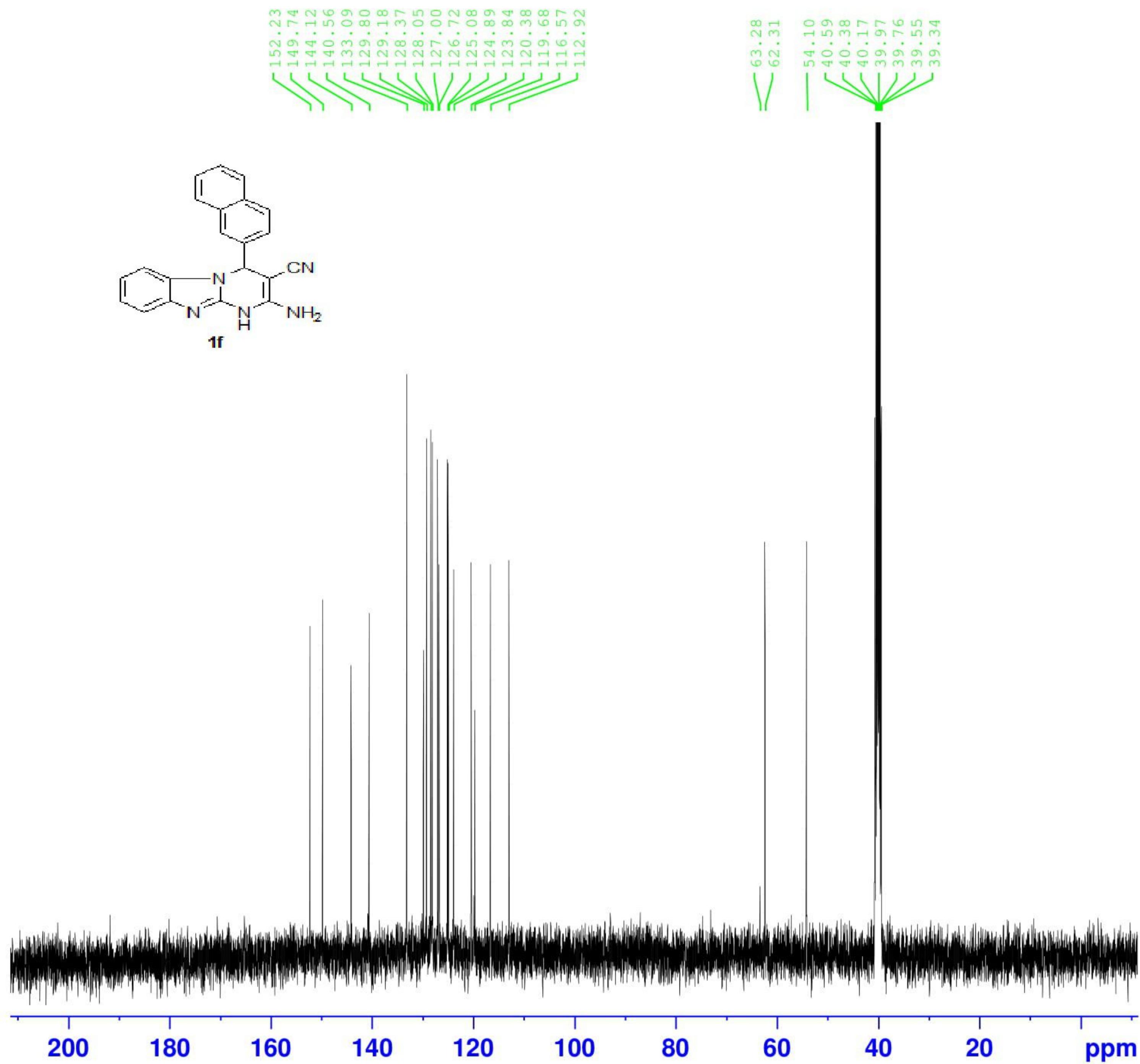


Current Data Parameters
 NAME nagwa-morad-38
 EXPNO 1
 PROCNO 1

F2 - Acquisition Parameters
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 PULPROG zg30
 TD 65536
 SOLVENT DMSO
 NS 24
 DS 2
 SWH 8012.820 Hz
 FIDRES 0.122266 Hz
 AQ 4.0894465 sec
 RG 205.37
 DW 62.400 usec
 DE 6.50 usec
 TE 298.0 K
 D1 1.0000000 sec
 TD0 1

===== CHANNEL f1 =====
 SFO1 400.1524711 MHz
 NUC1 1H
 P1 12.00 usec
 PLW1 18.00000000 W

F2 - Processing parameters
 SI 65536
 SF 400.1500000 MHz
 WDW EM
 SSB 0
 LB 0.30 Hz
 GB 0
 PC 1.00



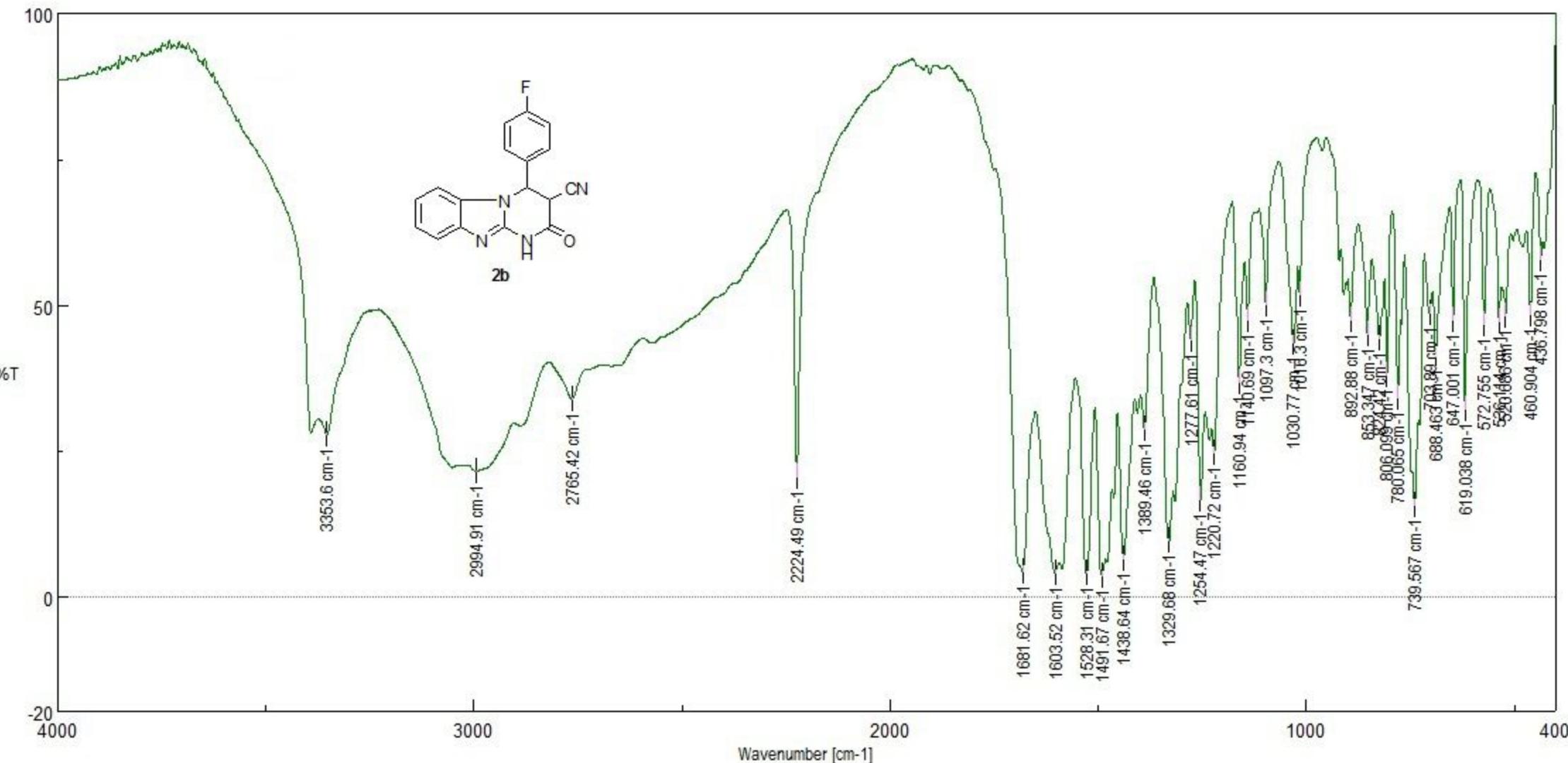
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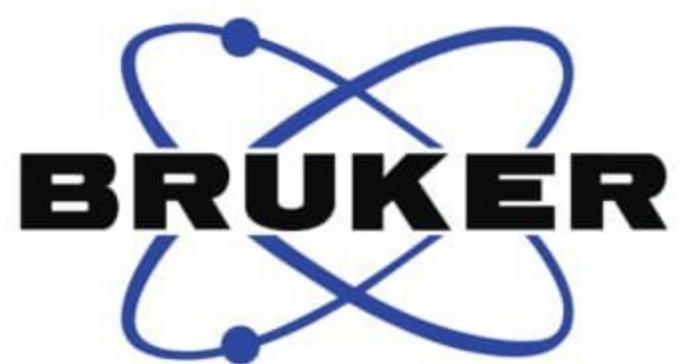
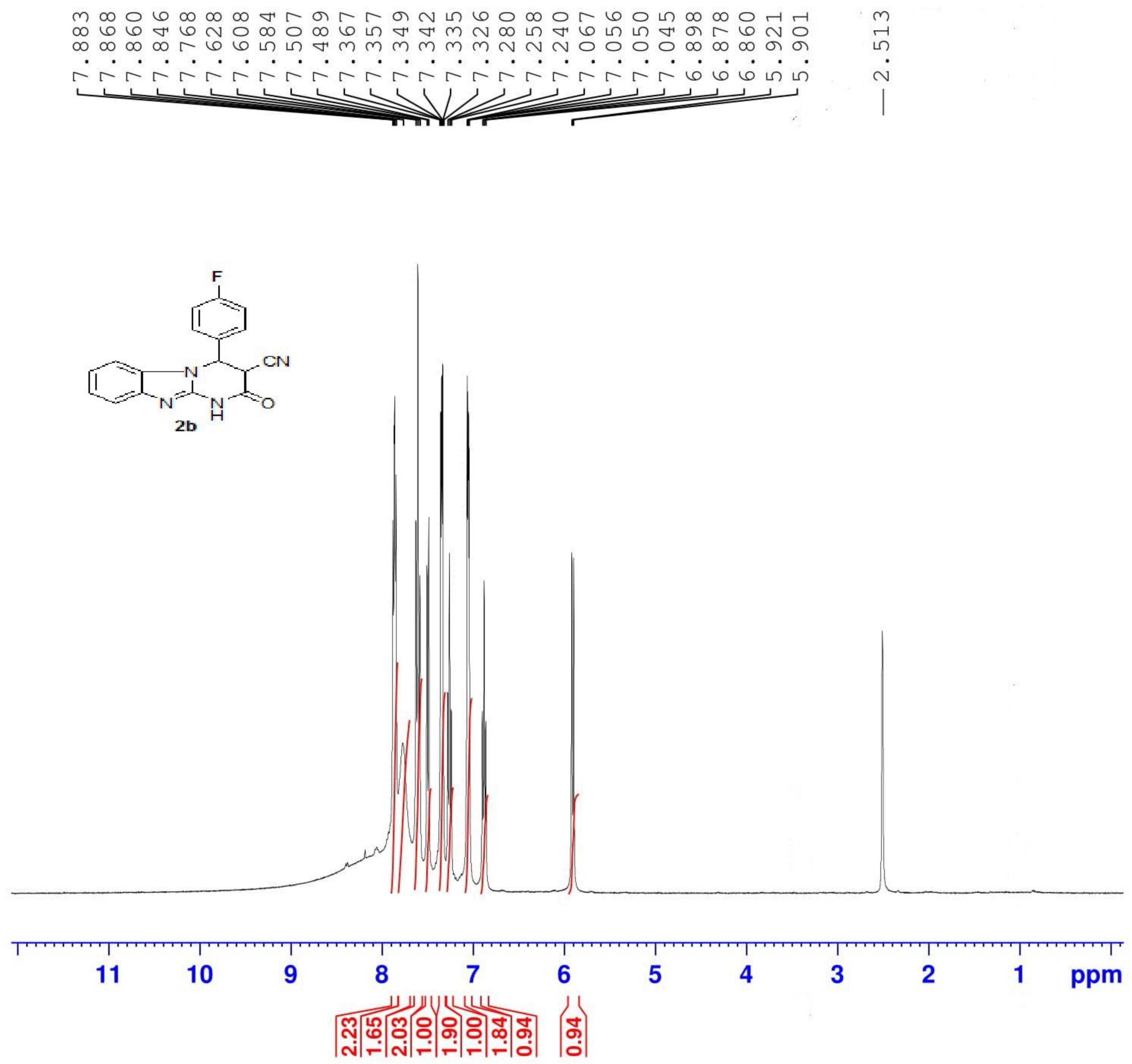
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 PULPROG zgpg30
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 SOLVENT DMSO
 NS 1344
 DS 4
 SWH 24038.461 Hz
 FIDRES 0.366798 Hz
 AQ 1.3631488 sec
 RG 205.37
 DW 20.800 usec
 DE 6.50 usec
 TE 298.0 K
 D1 2.0000000 sec
 D11 0.0300000 sec
 TD0 1

===== CHANNEL f1 =====
 SFO1 100.6278588 MHz
 NUC1 13C
 P1 10.00 usec
 PLW1 47.00000000 W

===== CHANNEL f2 =====
 SFO2 400.1516006 MHz
 NUC2 1H
 CPDPRG[2] waltz16
 PCPD2 90.00 usec
 PLW2 18.00000000 W
 PLW12 0.34722000 W
 PLW13 0.28125000 W

F2 - Processing parameters
 SI 32768
 SF 100.6177975 MHz
 WDW EM
 SSB 0
 LB 1.00 Hz
 GB 0
 PC 1.40



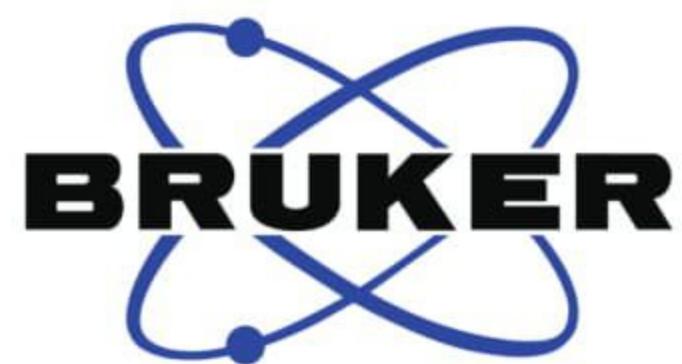
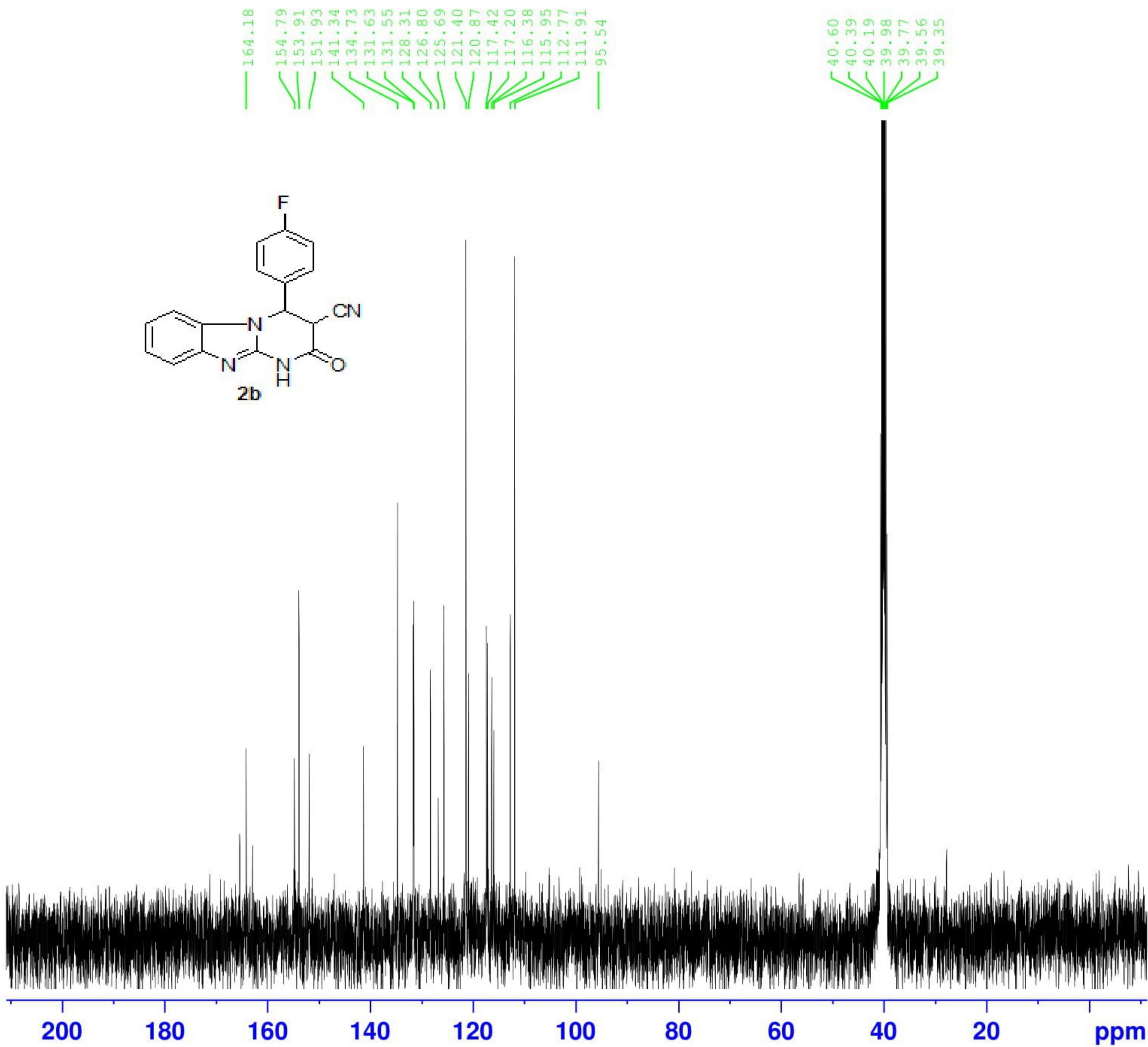


Current Data Parameters
 NAME nagwa-morad-44
 EXPNO 1
 PROCNO 1

F2 - Acquisition Parameters
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 PULPROG zg30
 TD 65536
 SOLVENT DMSO
 NS 38
 DS 2
 SWH 8012.820 Hz
 FIDRES 0.122266 Hz
 AQ 4.0894465 sec
 RG 205.37
 DW 62.400 usec
 DE 6.50 usec
 TE 299.0 K
 D1 1.0000000 sec
 TD0 1

===== CHANNEL f1 =====
 SFO1 400.1524711 MHz
 NUC1 1H
 P1 12.00 usec
 PLW1 18.00000000 W

F2 - Processing parameters
 SI 65536
 SF 400.1500000 MHz
 WDW EM
 SSB 0
 LB 0.30 Hz
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 PC 1.00



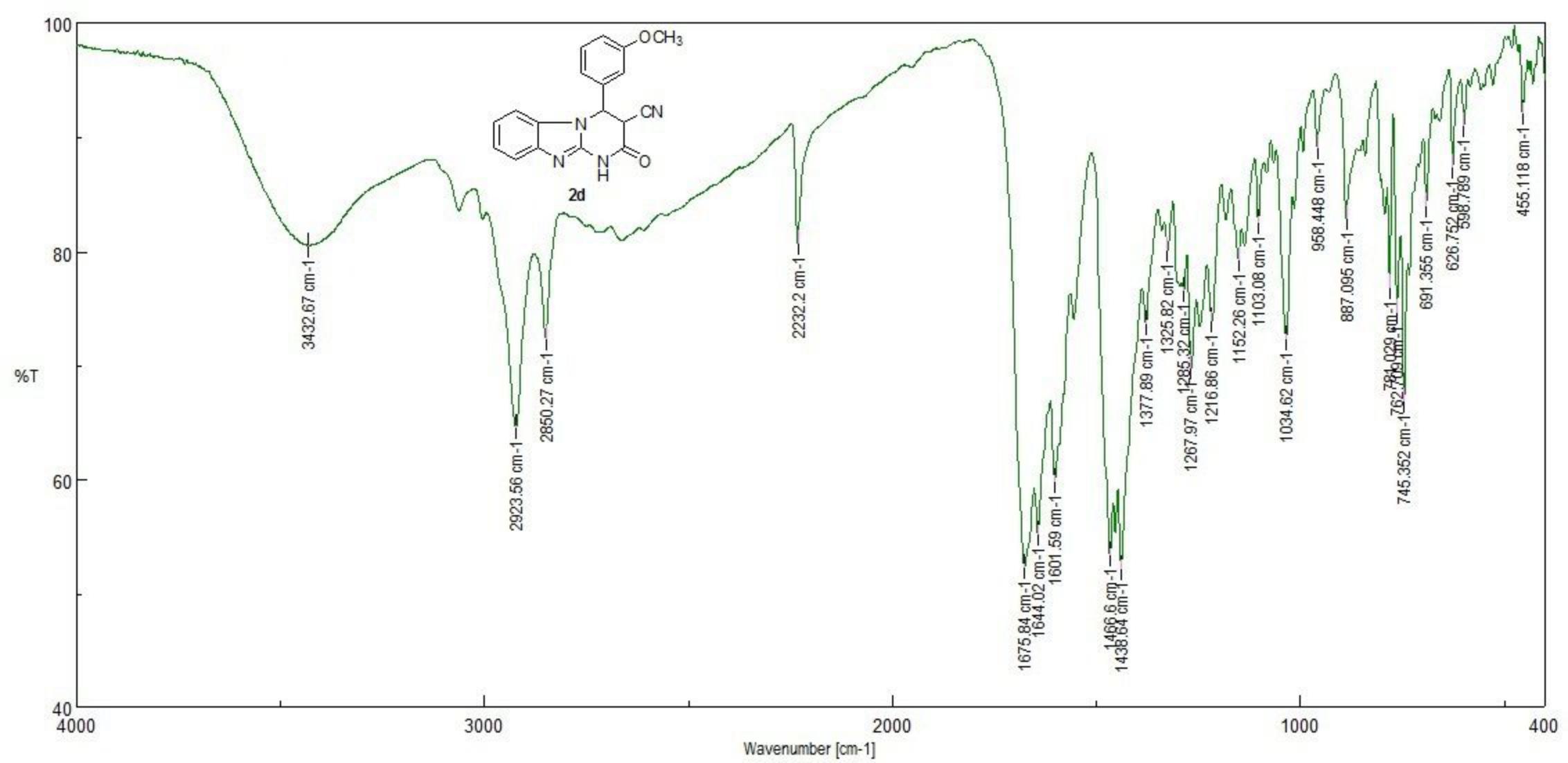
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 EXPNO 2
 PROCNO 1

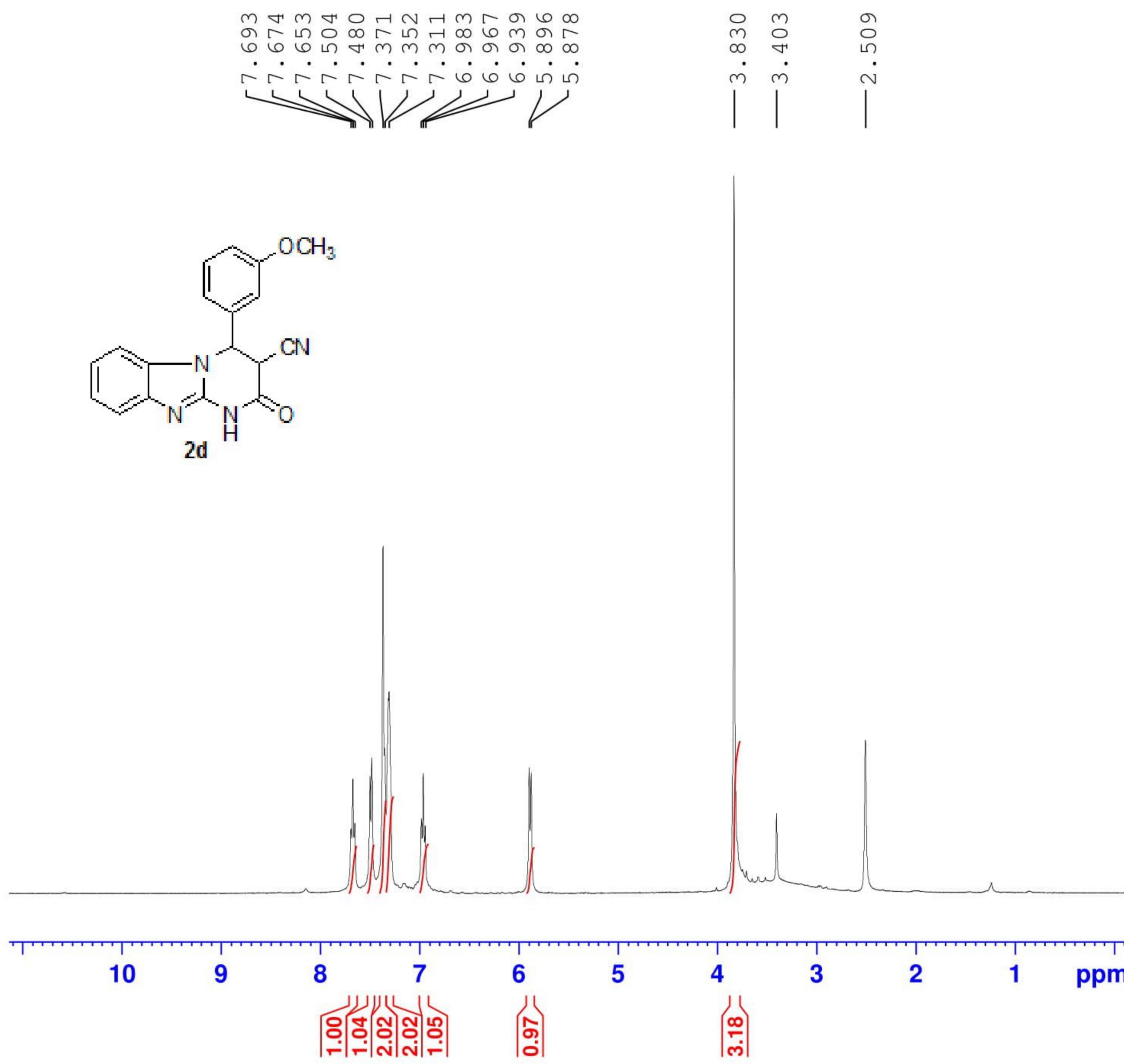
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 PULPROG zgpg30
 TD 65536
 SOLVENT DMSO
 NS 600
 DS 4
 SWH 24038.461 Hz
 FIDRES 0.366798 Hz
 AQ 1.3631488 sec
 RG 205.37
 DW 20.800 usec
 DE 6.50 usec
 TE 299.0 K
 D1 2.0000000 sec
 D11 0.0300000 sec
 TD0 1

===== CHANNEL f1 =====
 SFO1 100.6278588 MHz
 NUC1 13C
 P1 10.00 usec
 PLW1 47.00000000 W

===== CHANNEL f2 =====
 SFO2 400.1516006 MHz
 NUC2 1H
 CPDPRG[2] waltz16
 PCPD2 90.00 usec
 PLW2 18.00000000 W
 PLW12 0.34722000 W
 PLW13 0.28125000 W

F2 - Processing parameters
 SI 32768
 SF 100.6177975 MHz
 WDW EM
 SSB 0
 LB 1.00 Hz
 GB 0
 PC 1.40



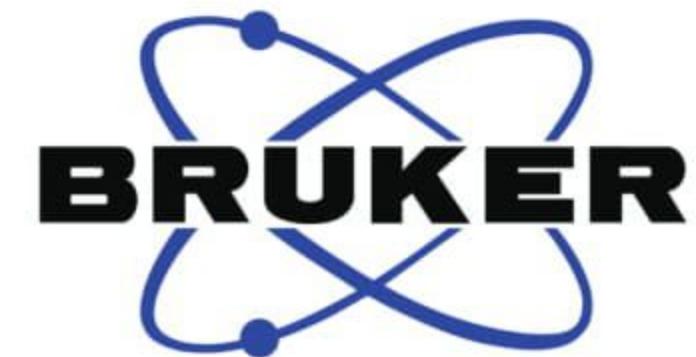
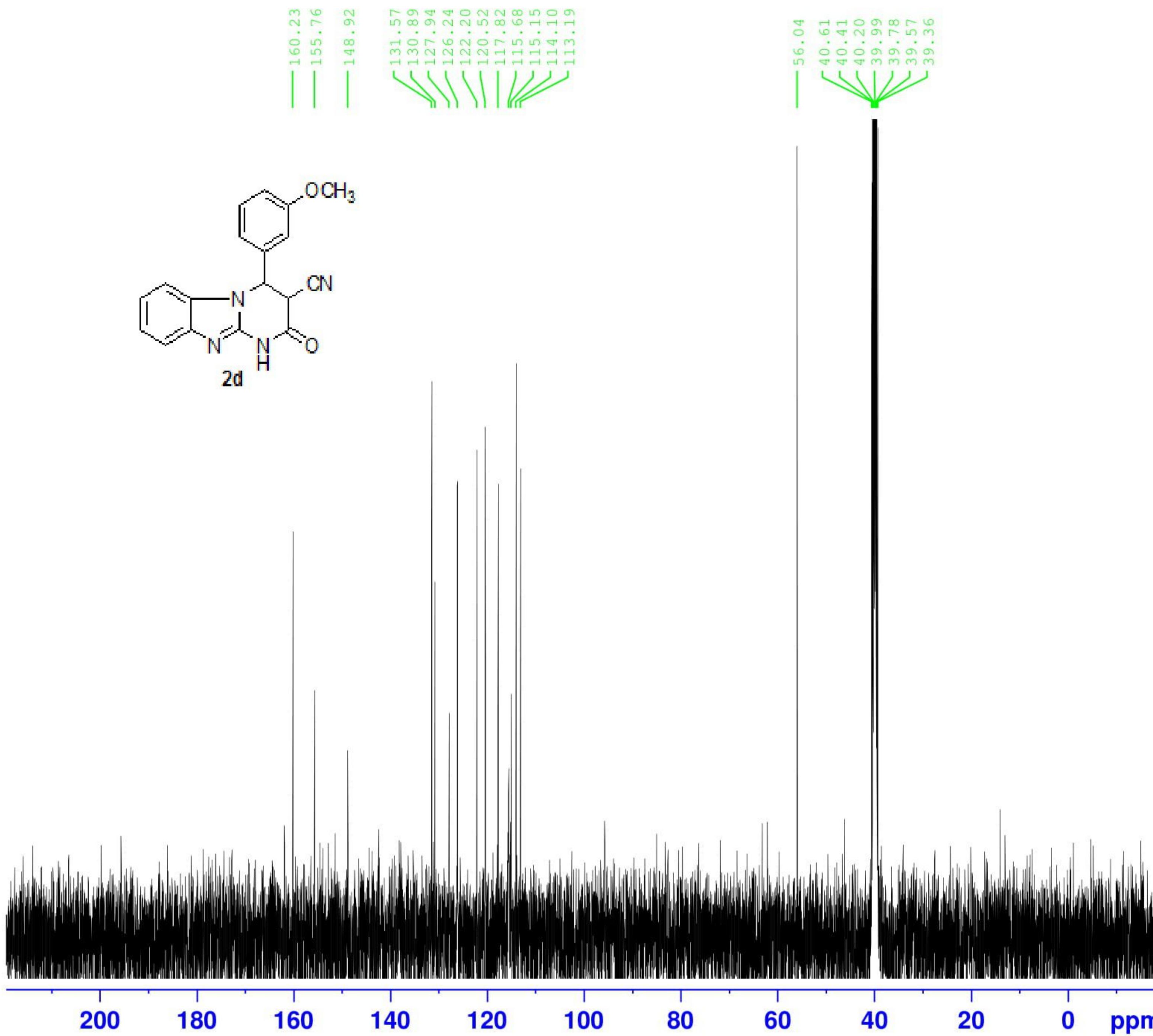


Current Data Parameters
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 PROCNO 1

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 PULPROG zg30
 TD 65536
 SOLVENT DMSO
 NS 46
 DS 2
 SWH 8012.820 Hz
 FIDRES 0.122266 Hz
 AQ 4.0894465 sec
 RG 205.37
 DW 62.400 usec
 DE 6.50 usec
 TE 299.0 K
 D1 1.0000000 sec
 TD0 1

===== CHANNEL f1 =====
 SFO1 400.1524711 MHz
 NUC1 1H
 P1 12.00 usec
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F2 - Processing parameters
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 WDW EM
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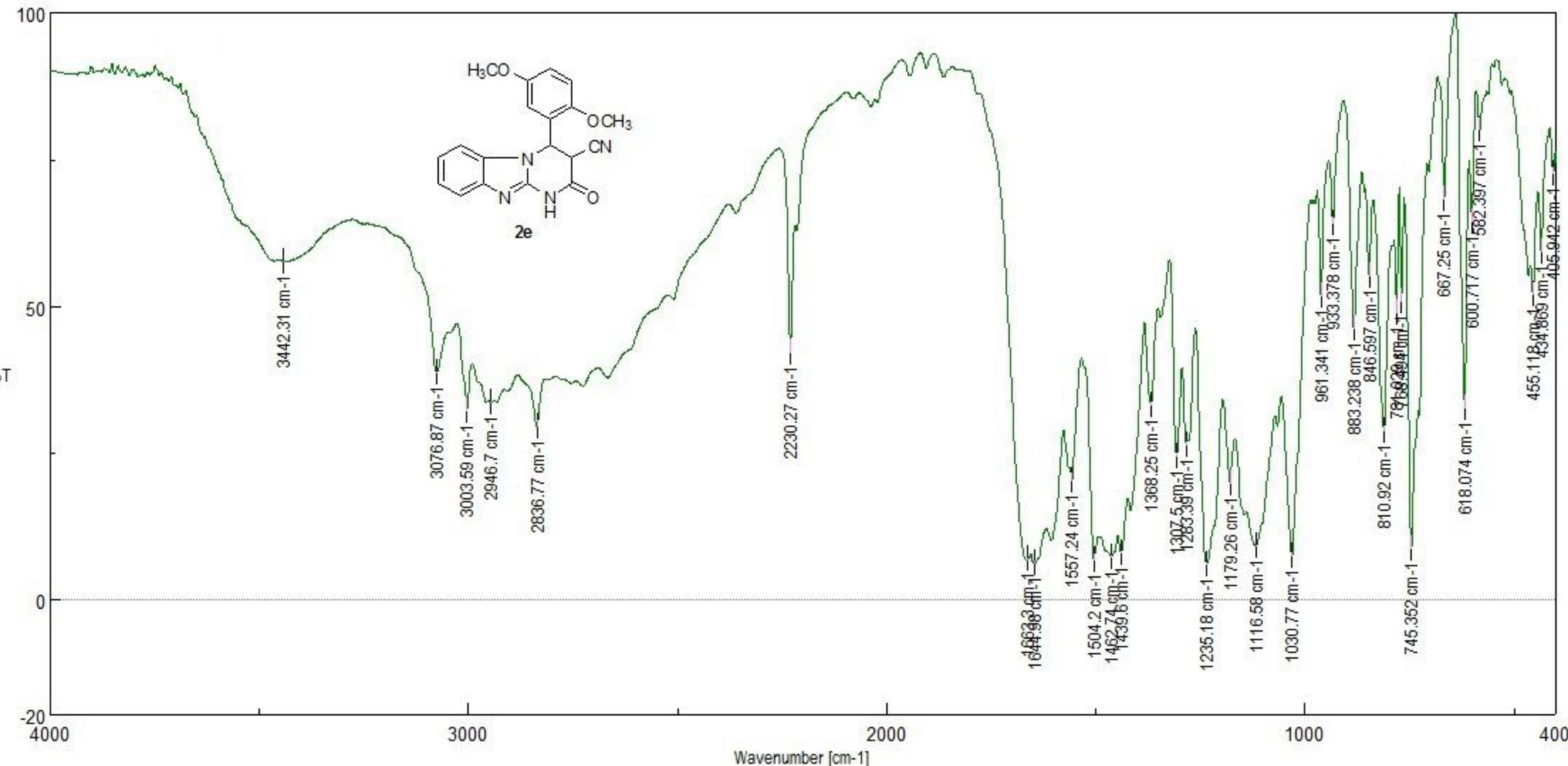
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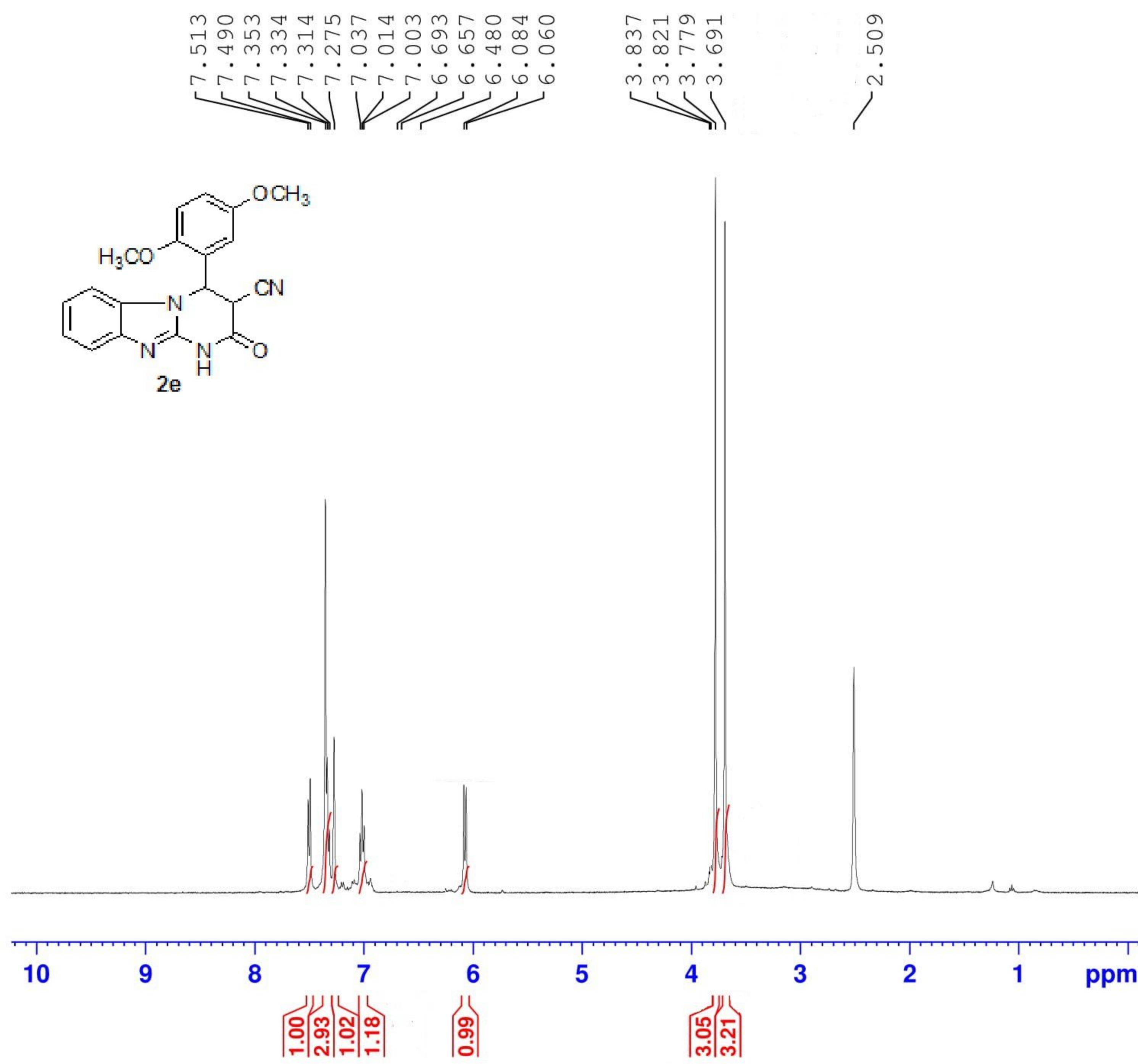
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 PULPROG zgpg30
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 SOLVENT DMSO
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 FIDRES 0.366798 Hz
 AQ 1.3631488 sec
 RG 205.37
 DW 20.800 usec
 DE 6.50 usec
 TE 299.0 K
 D1 2.00000000 sec
 D11 0.03000000 sec
 TD0 1

===== CHANNEL f1 =====
 SFO1 100.6278588 MHz
 NUC1 13C
 P1 10.00 usec
 PLW1 47.00000000 W

===== CHANNEL f2 =====
 SFO2 400.1516006 MHz
 NUC2 1H
 CPDPRG[2] waltz16
 PCPD2 90.00 usec
 PLW2 18.00000000 W
 PLW12 0.34722000 W
 PLW13 0.28125000 W

F2 - Processing parameters
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 WDW EM
 SSB 0
 LB 1.00 Hz
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 PC 1.40



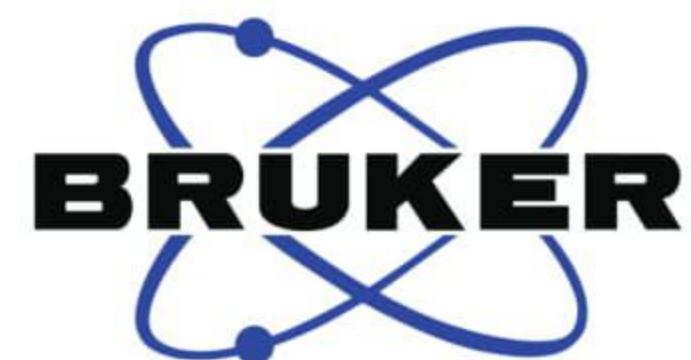
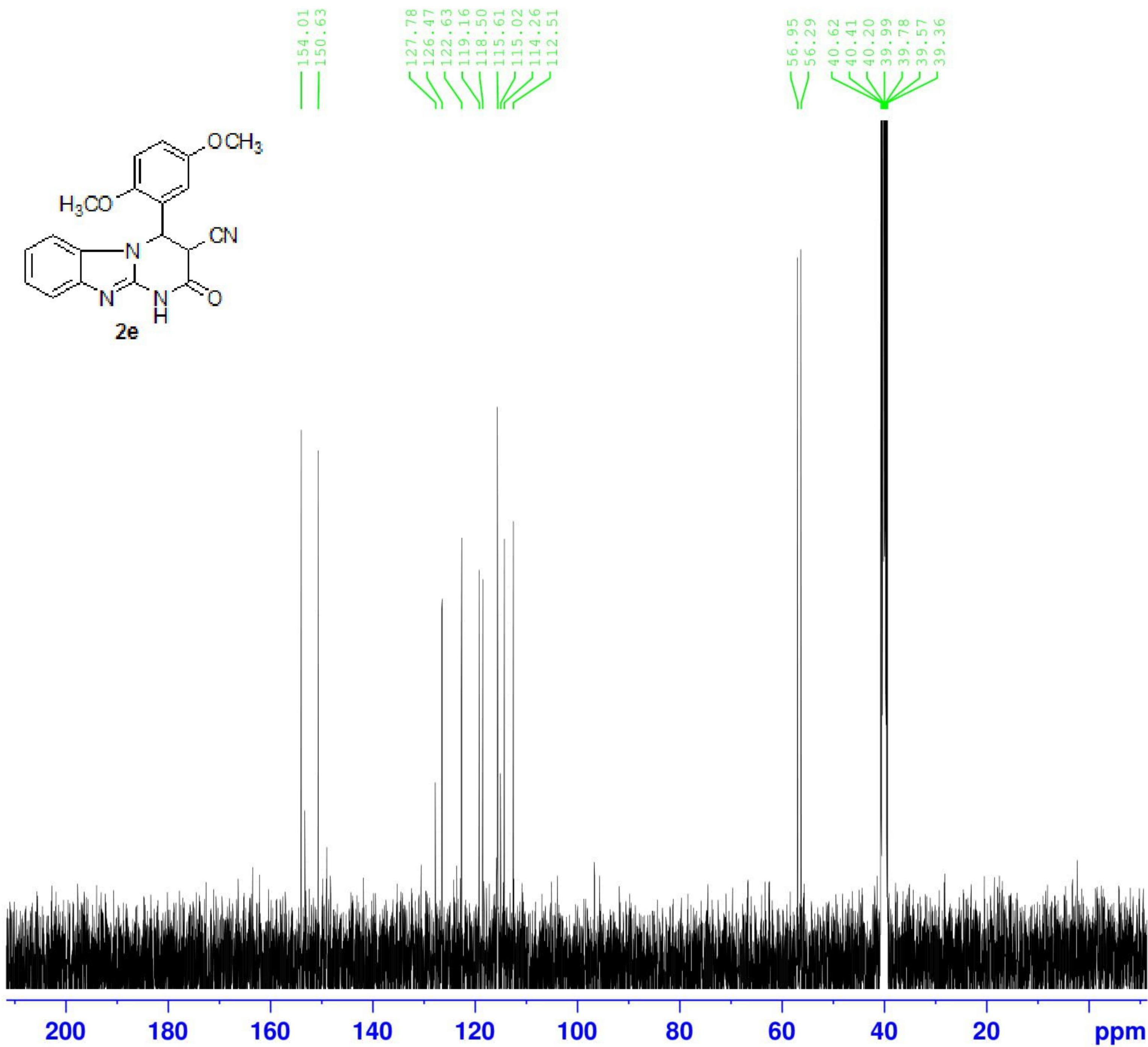


Current Data Parameters
 NAME nagwa-morad-47
 EXPNO 1
 PROCNO 1

F2 - Acquisition Parameters
 Date 20180422
 Time 9.36
 INSTRUM spect
 PROBHD 5 mm PABBO BB/
 PULPROG zg30
 TD 65536
 SOLVENT DMSO
 NS 24
 DS 2
 SWH 8012.820 Hz
 FIDRES 0.122266 Hz
 AQ 4.0894465 sec
 RG 205.37
 DW 62.400 usec
 DE 6.50 usec
 TE 299.0 K
 D1 1.0000000 sec
 TD0 1

===== CHANNEL f1 =====
 SFO1 400.1524711 MHz
 NUC1 1H
 P1 12.00 usec
 PLW1 18.00000000 W

F2 - Processing parameters
 SI 65536
 SF 400.1500000 MHz
 WDW EM
 SSB 0
 LB 0.30 Hz
 GB 0
 PC 1.00



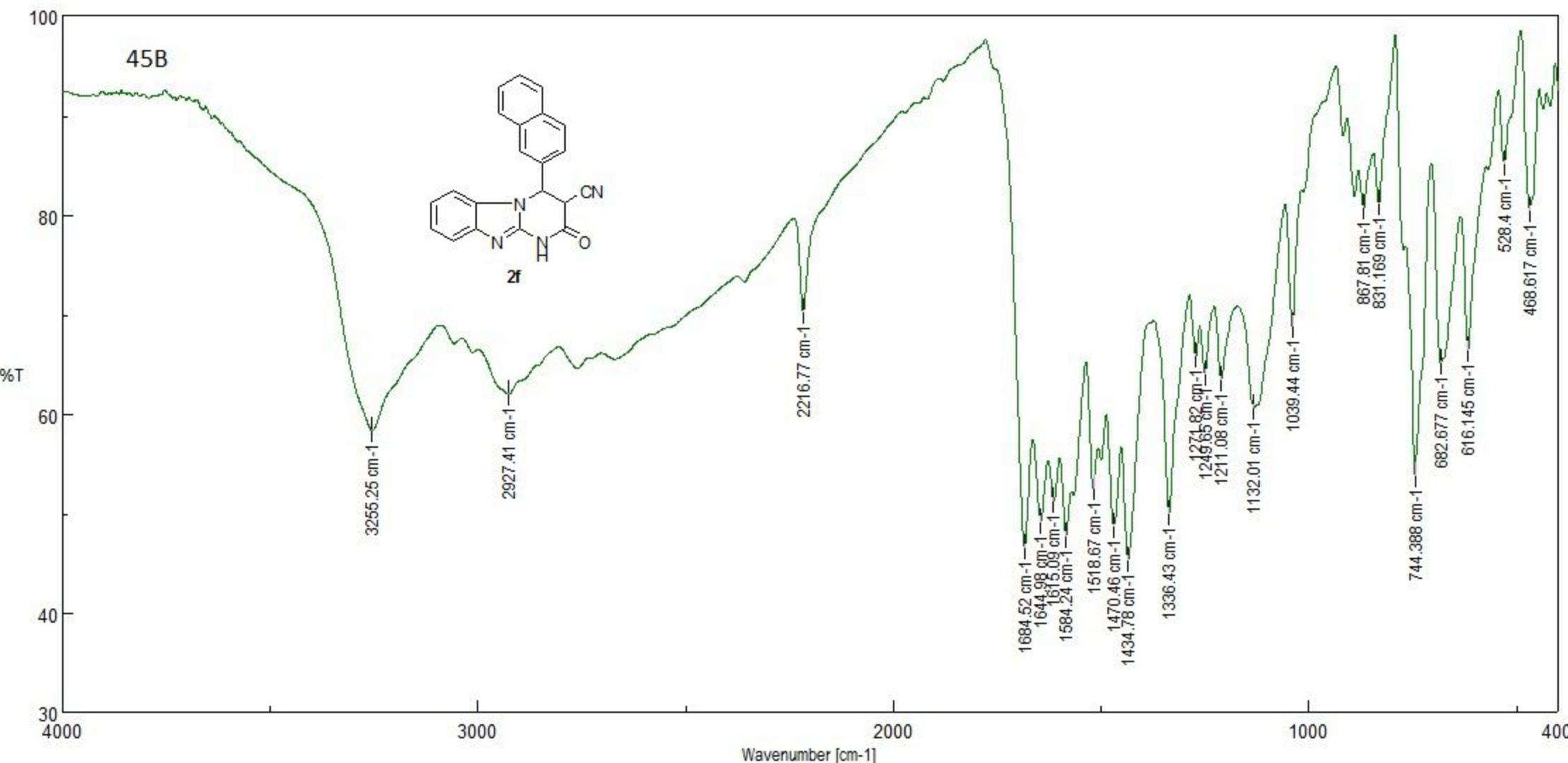
Current Data Parameters
 NAME nagwa-morad-47
 EXPNO 2
 PROCNO 1

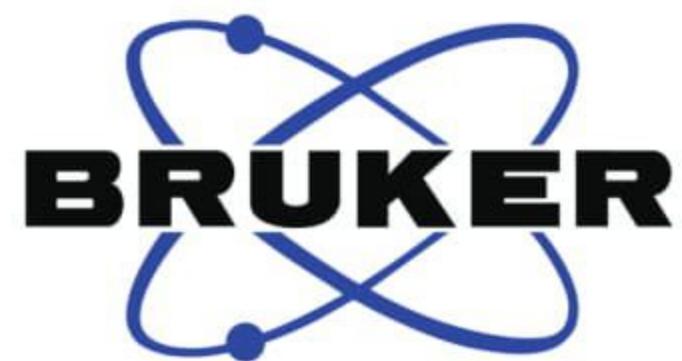
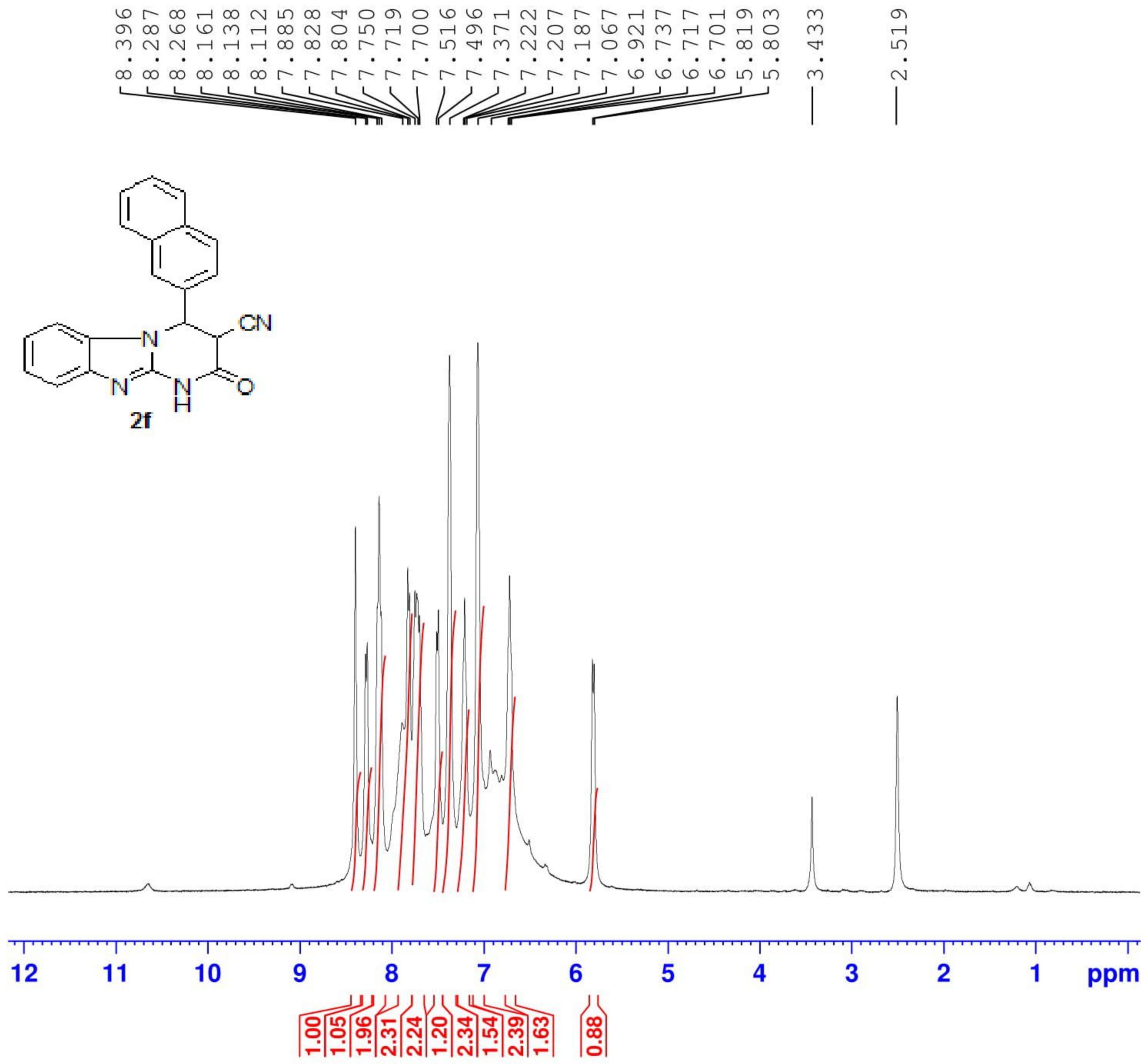
F2 - Acquisition Parameters
 Date_ 20180422
 Time 10.55
 INSTRUM spect
 PROBHD 5 mm PABBO BB/
 PULPROG zgpg30
 TD 65536
 SOLVENT DMSO
 NS 1348
 DS 4
 SWH 24038.461 Hz
 FIDRES 0.366798 Hz
 AQ 1.3631488 sec
 RG 205.37
 DW 20.800 usec
 DE 6.50 usec
 TE 299.0 K
 D1 2.00000000 sec
 D11 0.03000000 sec
 TD0 1

===== CHANNEL f1 =====
 SFO1 100.6278588 MHz
 NUC1 13C
 P1 10.00 usec
 PLW1 47.00000000 W

===== CHANNEL f2 =====
 SFO2 400.1516006 MHz
 NUC2 1H
 CPDPRG[2] waltz16
 PCPD2 90.00 usec
 PLW2 18.00000000 W
 PLW12 0.34722000 W
 PLW13 0.28125000 W

F2 - Processing parameters
 SI 32768
 SF 100.6177975 MHz
 WDW EM
 SSB 0
 LB 1.00 Hz
 GB 0
 PC 1.40





Current Data Parameters
 NAME nagwa-morad-45B
 EXPNO 1
 PROCNO 1

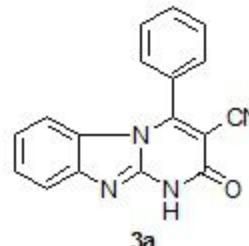
F2 - Acquisition Parameters
 Date_ 20180104
 Time 9.40
 INSTRUM spect
 PROBHD 5 mm PABBO BB/
 PULPROG zg30
 TD 65536
 SOLVENT DMSO
 NS 31
 DS 2
 SWH 8012.820 Hz
 FIDRES 0.122266 Hz
 AQ 4.0894465 sec
 RG 205.37
 DW 62.400 usec
 DE 6.50 usec
 TE 299.0 K
 D1 1.0000000 sec
 TD0 1

===== CHANNEL f1 =====
 SFO1 400.1524711 MHz
 NUC1 1H
 P1 12.00 usec
 PLW1 18.00000000 W

F2 - Processing parameters
 SI 65536
 SF 400.1500000 MHz
 WDW EM
 SSB 0
 LB 0.30 Hz
 GB 0
 PC 1.00

100

43



%T

40

20

4000

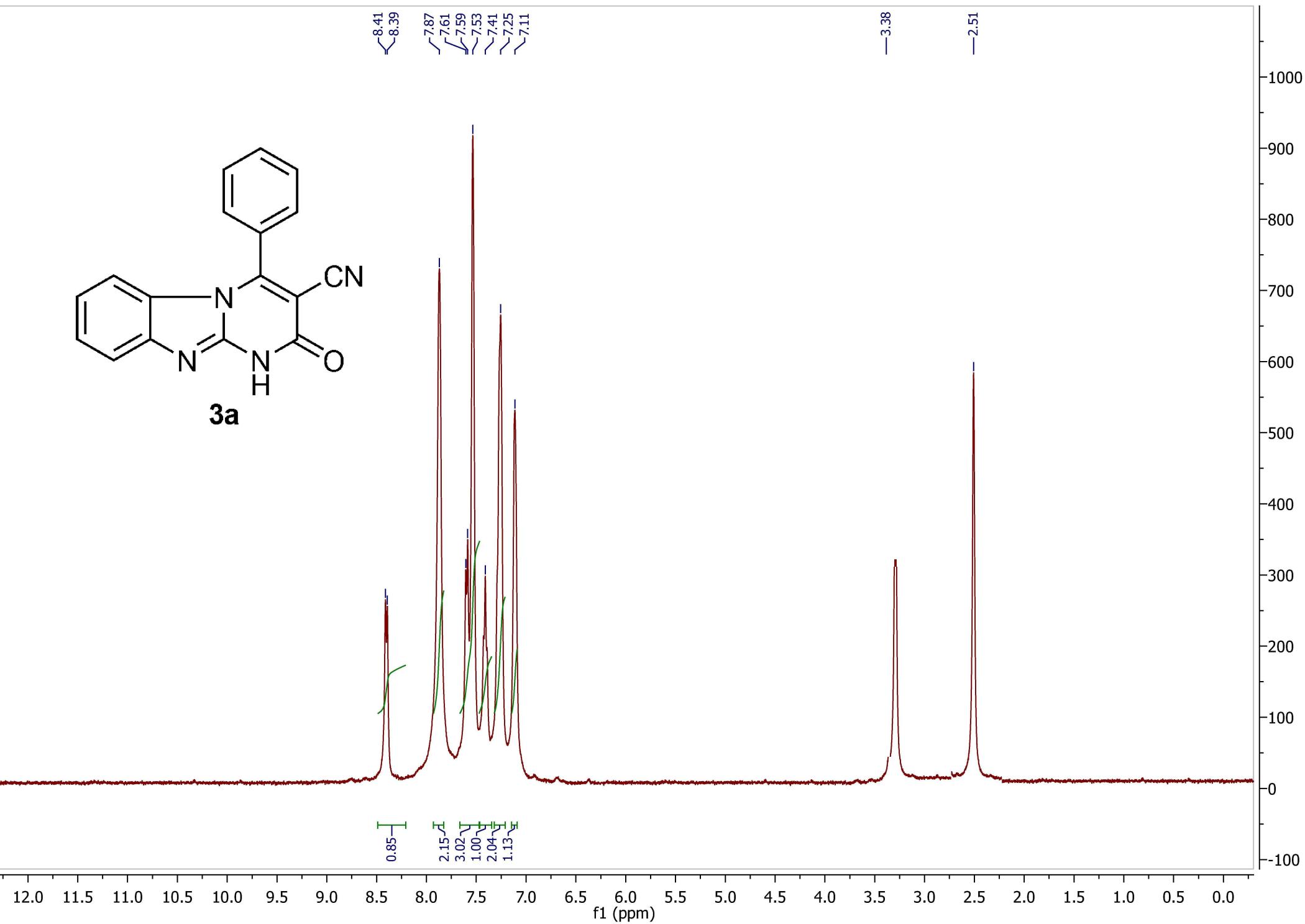
3000

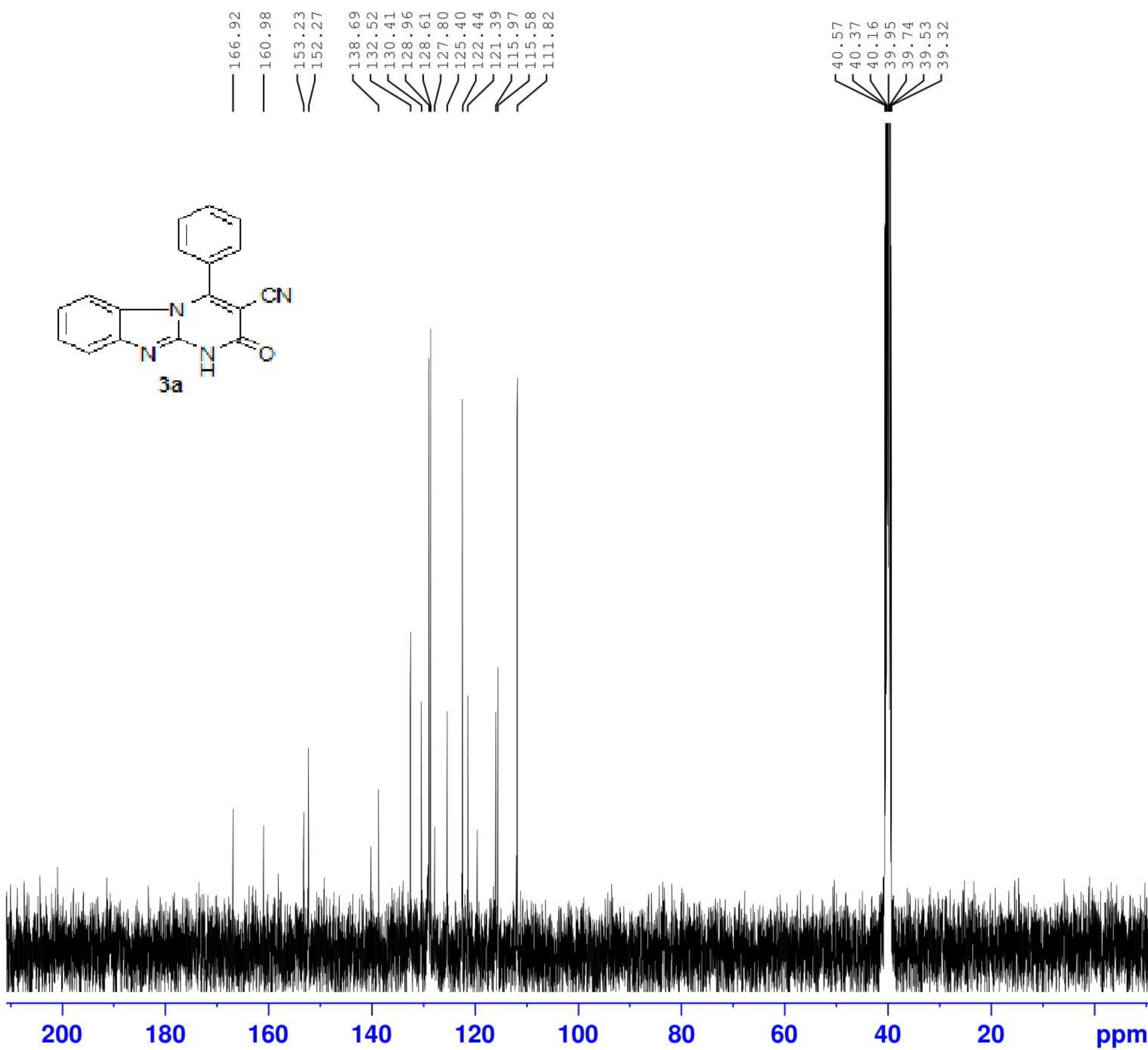
2000

1000

400

Wavenumber [cm⁻¹]





Current Data Parameters
 NAME nagwa-43A
 EXPNO 2
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20181118
 Time 9.38
 INSTRUM spect
 PROBHD 5 mm PABBO BB/
 PULPROG zgpg30
 TD 65536
 SOLVENT DMSO
 NS 1024
 DS 4
 SWH 24038.461 Hz
 FIDRES 0.366798 Hz
 AQ 1.3631488 sec
 RG 205.37
 DW 20.800 usec
 DE 6.50 usec
 TE 299.0 K
 D1 2.0000000 sec
 D11 0.0300000 sec
 TD0 1

===== CHANNEL f1 =====
 SFO1 100.6278588 MHz
 NUC1 13C
 P1 10.00 usec
 PLW1 47.00000000 W

===== CHANNEL f2 =====
 SFO2 400.1516006 MHz
 NUC2 1H
 CPDPRG[2] waltz16
 PCPD2 90.00 usec
 PLW2 18.00000000 W
 PLW12 0.34722000 W
 PLW13 0.28125000 W

F2 - Processing parameters
 SI 32768
 SF 100.6177975 MHz
 WDW EM
 SSB 0
 LB 1.00 Hz
 GB 0
 PC 1.40

100

44

%T

50

0

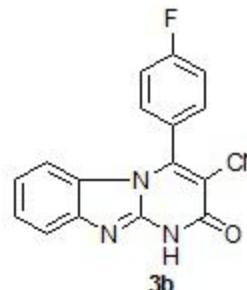
-5

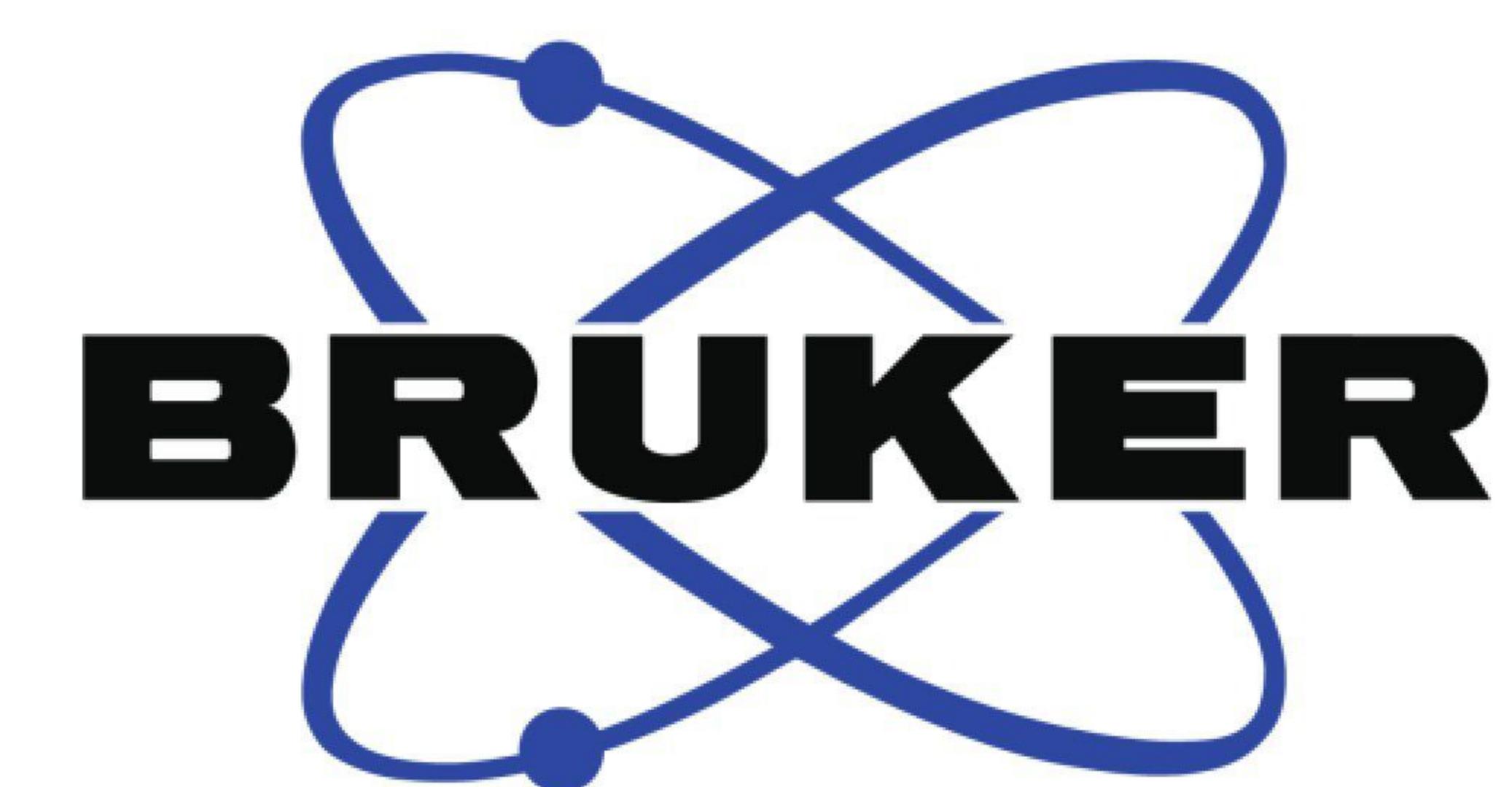

3000

2000

1000

400

Wavenumber [cm⁻¹]3301.54 cm⁻¹3001.66 cm⁻¹2210.02 cm⁻¹1698.02 cm⁻¹1605.45 cm⁻¹1534.1 cm⁻¹1440.56 cm⁻¹1229.4 cm⁻¹1156.12 cm⁻¹1099.23 cm⁻¹739.567 cm⁻¹898.666 cm⁻¹838.883 cm⁻¹665.321 cm⁻¹616.145 cm⁻¹474.403 cm⁻¹

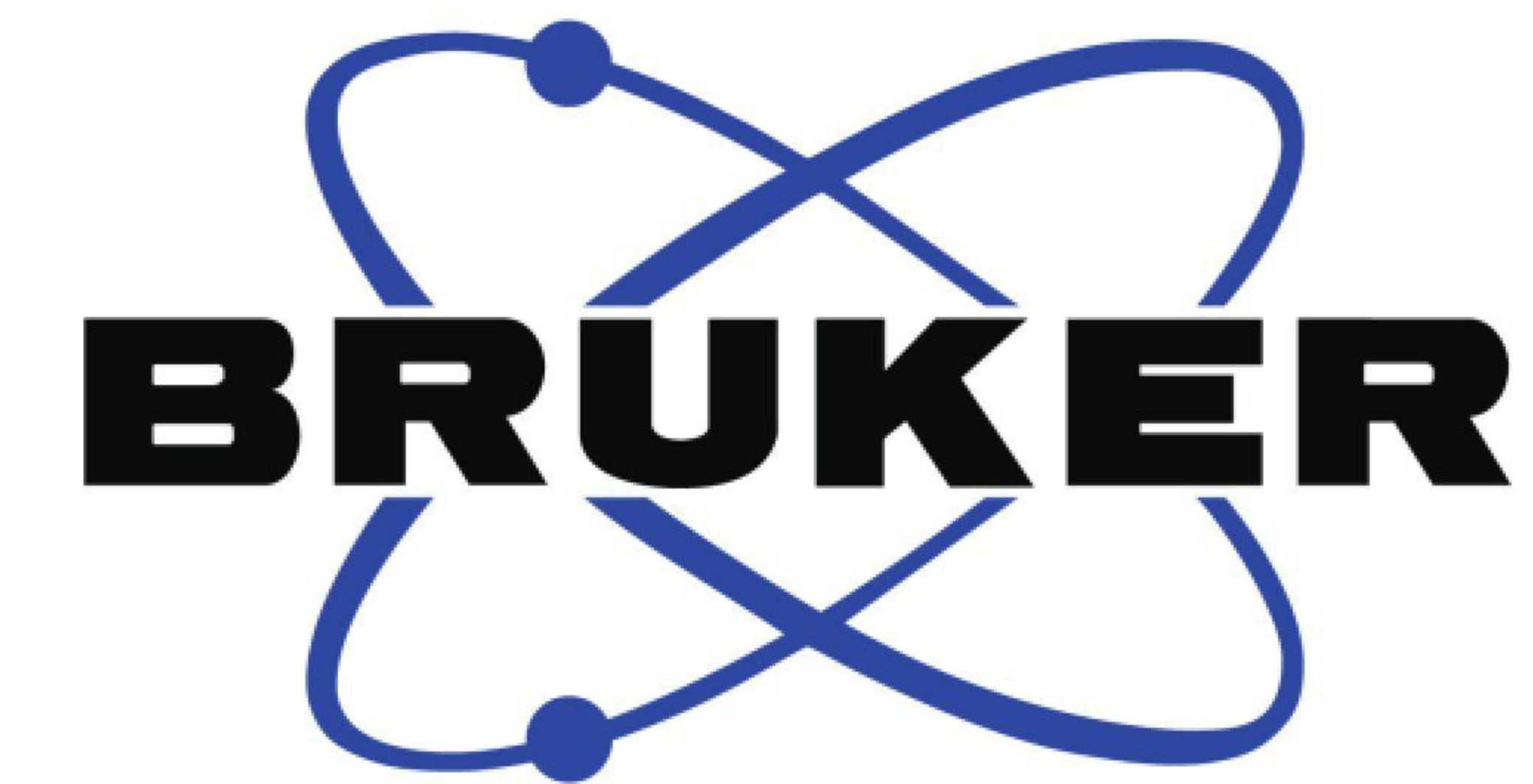



Current Data Parameters
 NAME nagwa-44A
 EXPNO 1
 PROCNO 1

F2 - Acquisition Parameters
 Date 20181118
 Time 12.56
 INSTRUM spect
 PROBHD 5 mm PABBO BB/
 PULPROG zg30
 TD 65536
 SOLVENT DMSO
 NS 64
 DS 2
 SWH 8012.820 Hz
 FIDRES 0.122266 Hz
 AQ 4.0894465 sec
 RG 205.37
 DW 62.400 usec
 DE 6.50 usec
 TE 299.0 K
 D1 1.0000000 sec
 TD0 1

===== CHANNEL f1 =====
 SFO1 400.1524711 MHz
 NUC1 1H
 P1 12.00 usec
 PLW1 18.00000000 W

F2 - Processing parameters
 SI 65536
 SF 400.1500000 MHz
 WDW EM
 SSB 0
 LB 0.30 Hz
 GB 0
 PC 1.00



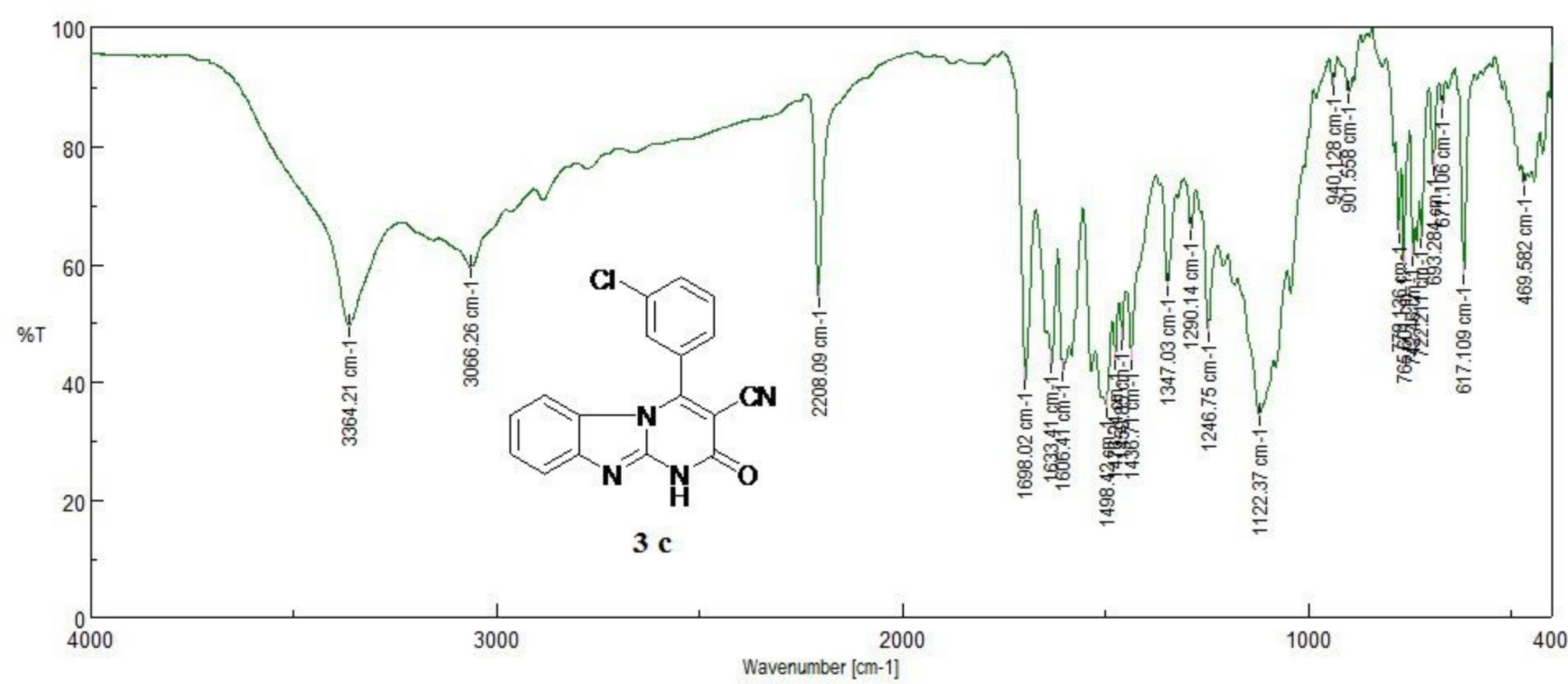
Current Data Parameters
 NAME nagwa-44A
 EXPNO 2
 PROCNO 1

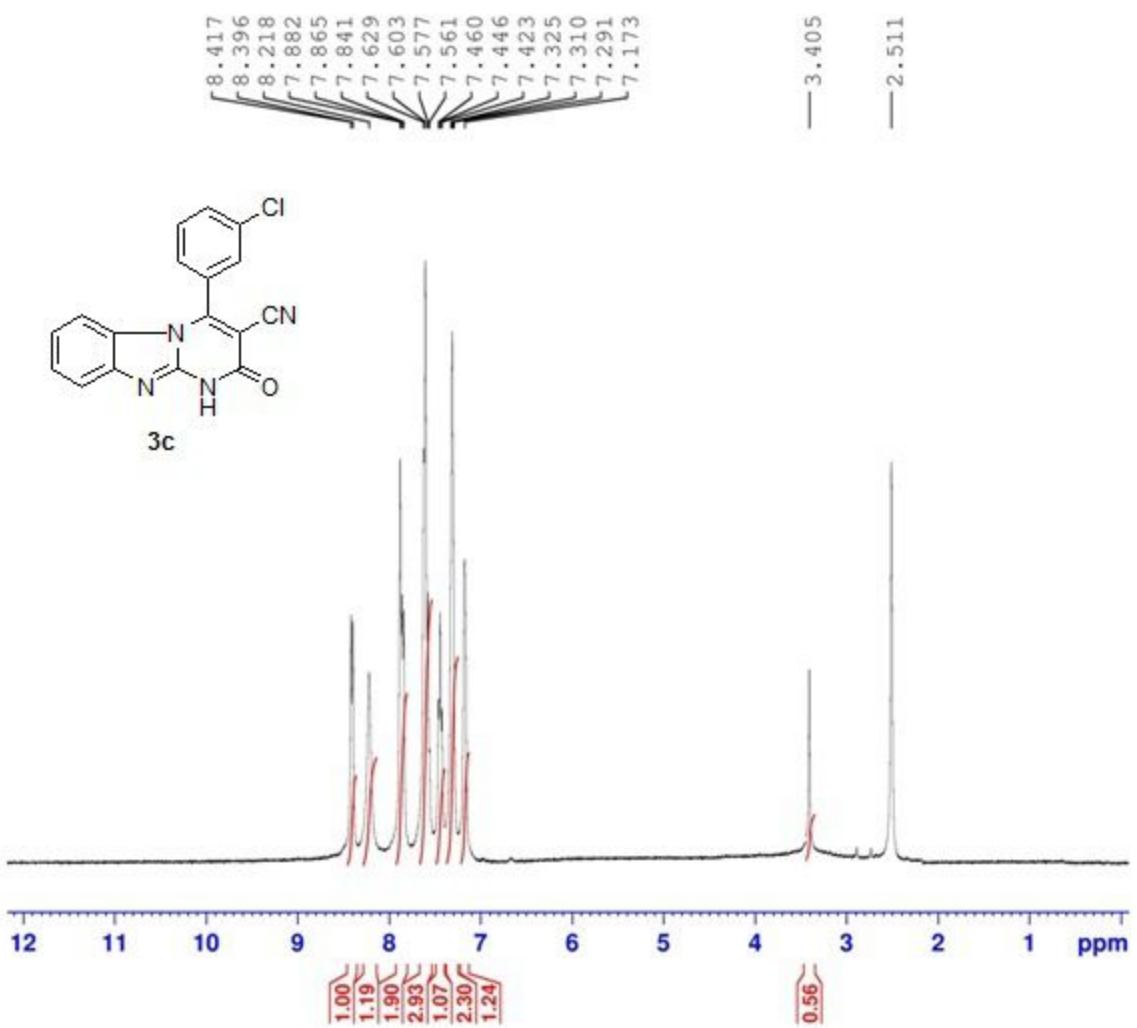
F2 - Acquisition Parameters
 Date_ 20181118
 Time 15.50
 INSTRUM spect
 PROBHD 5 mm PABBO BB/
 PULPROG zgpg30
 TD 65536
 SOLVENT DMSO
 NS 3000
 DS 4
 SWH 24038.461 Hz
 FIDRES 0.366798 Hz
 AQ 1.3631488 sec
 RG 205.37
 DW 20.800 usec
 DE 6.50 usec
 TE 300.0 K
 D1 2.0000000 sec
 D11 0.0300000 sec
 TD0 1

===== CHANNEL f1 =====
 SFO1 100.6278588 MHz
 NUC1 13C
 P1 10.00 usec
 PLW1 47.0000000 W

===== CHANNEL f2 =====
 SFO2 400.1516006 MHz
 NUC2 1H
 CPDPRG[2] waltz16
 PCPD2 90.00 usec
 PLW2 18.00000000 W
 PLW12 0.34722000 W
 PLW13 0.28125000 W

F2 - Processing parameters
 SI 32768
 SF 100.6177975 MHz
 WDW EM
 SSB 0
 LB 1.00 Hz
 GB 0
 PC 1.40



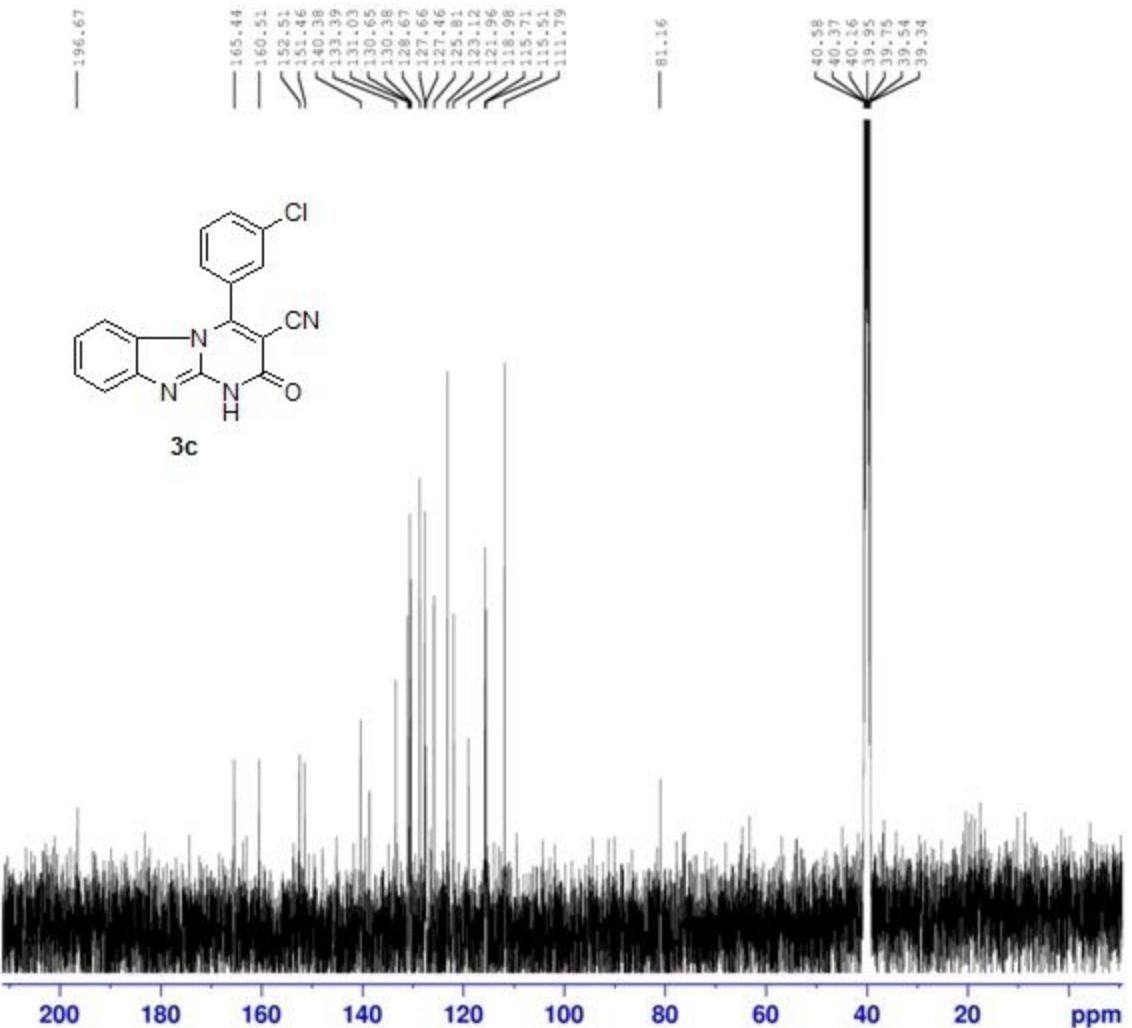


Current Data Parameters
 NAME nagwa-48A
 EXPNO 2
 PROCNO 1

F2 - Acquisition Parameters
 Date 20181121
 Time 9.28
 INSTRUM spect
 PROBHD 5 mm PABBO BB/
 PULPROG zg30
 TD 65536
 SOLVENT DMSO
 NS 30
 DS 2
 SWH 8012.820 Hz
 FIDRES 0.122266 Hz
 AQ 4.0894465 sec
 RG 205.37
 DW 62.400 usec
 DE 6.50 usec
 TE 300.0 K
 D1 1.0000000 sec
 TDO 1

===== CHANNEL f1 =====
 SF01 400.1524711 MHz
 NUC1 1H
 PI 12.00 usec
 PLW1 18.00000000 W

F2 - Processing parameters
 SI 65536
 SF 400.1500000 MHz
 WDW EM
 SSB 0
 LB 0.30 Hz
 GB 0
 PC 1.00



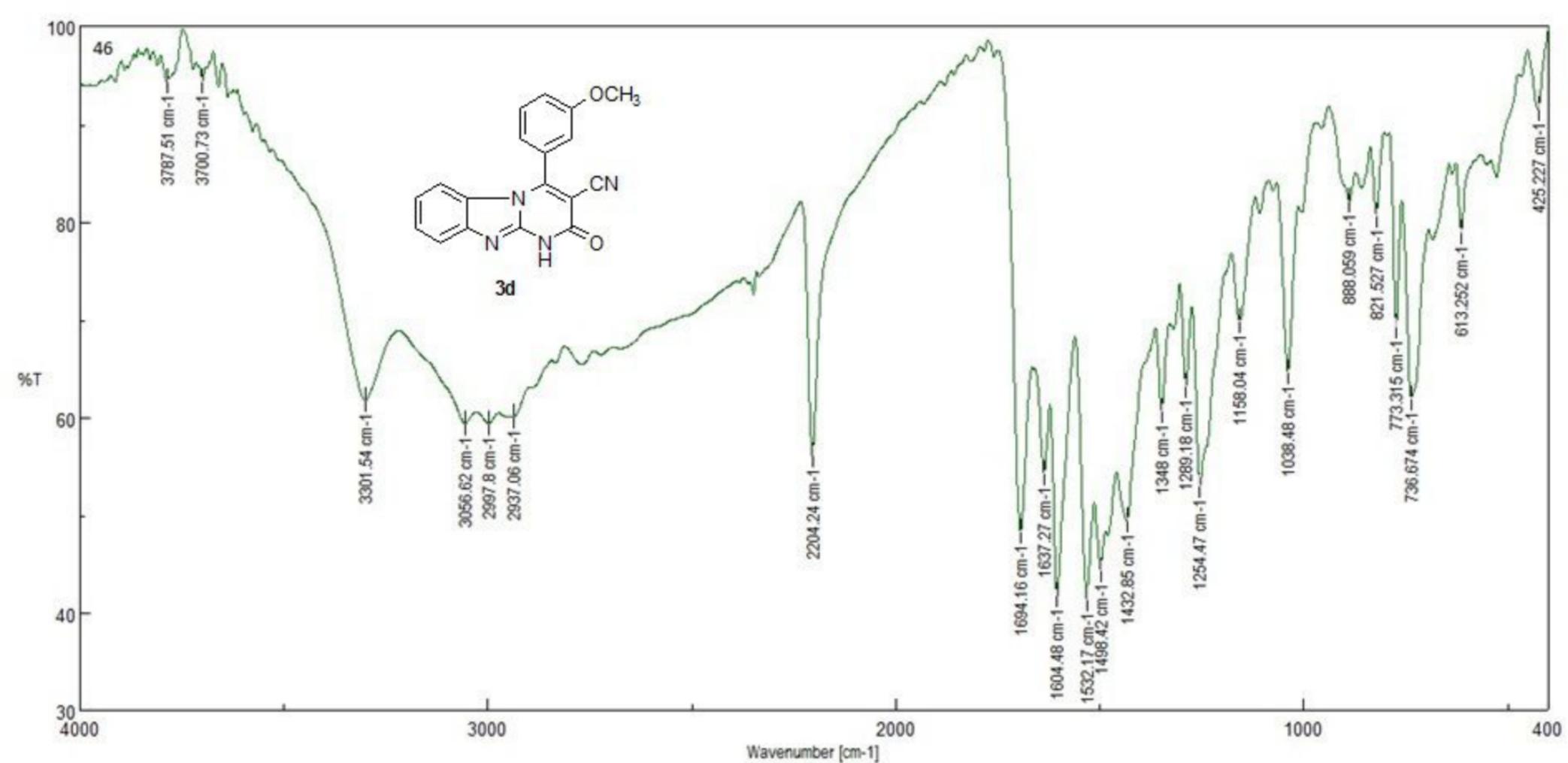
Current Data Parameters
 NAME nagwa-48A
 EXPNO 1
 PROCNO 1

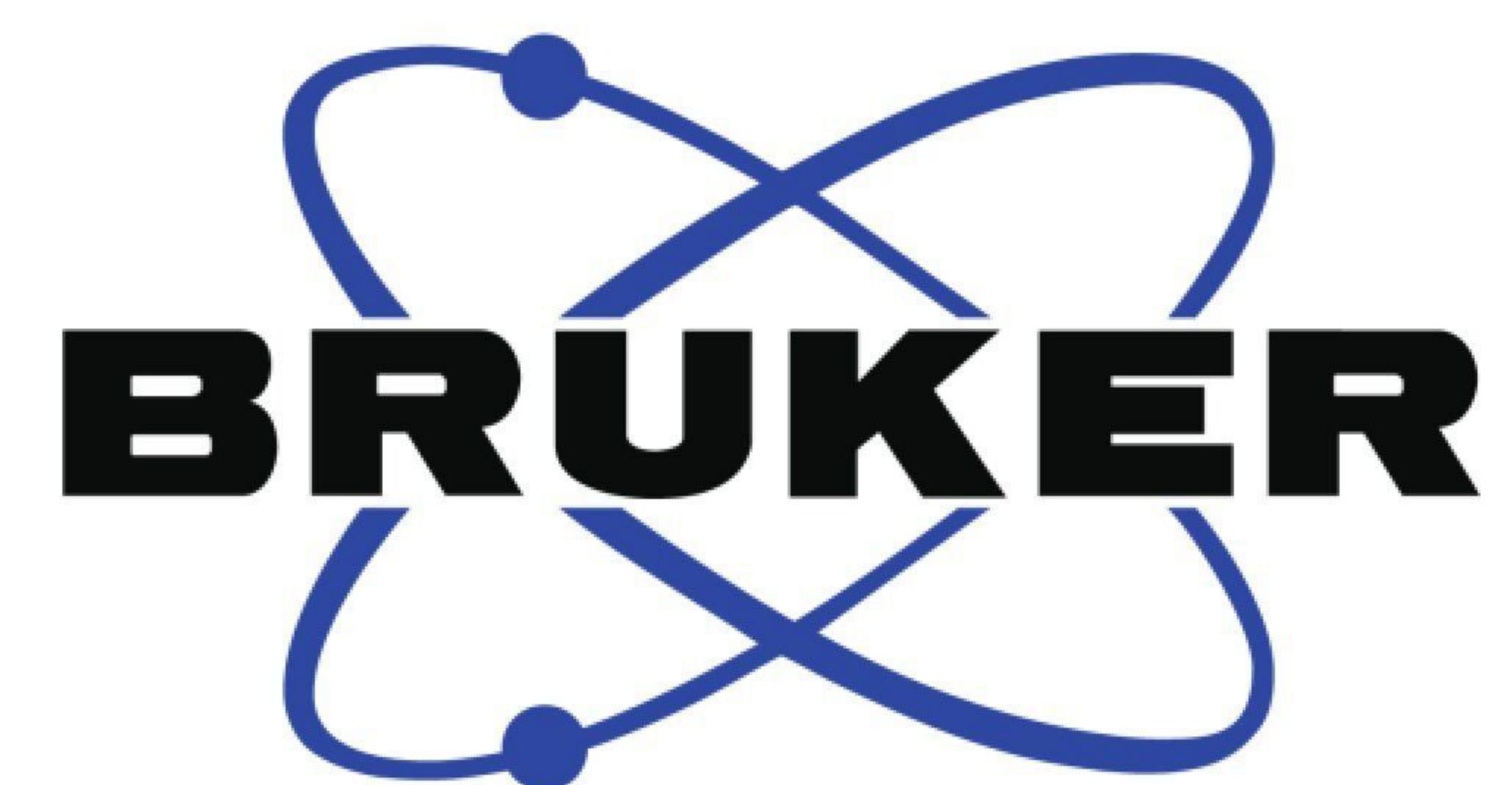

F2 - Acquisition Parameters
 Date_ 20181121
 Time 9.23
 INSTRUM spect
 PROBHD 5 mm PABBO BB/
 PULPROG zgpg30
 TD 65536
 SOLVENT DMSO
 NS 1038
 DS 4
 SWH 24038.461 Hz
 FIDRES 0.366798 Hz
 AQ 1.3631488 sec
 RG 205.37
 DW 20.800 usec
 DE 6.50 usec
 TE 300.0 K
 D1 2.00000000 sec
 D11 0.03000000 sec
 TDO 1

----- CHANNEL f1 -----
 SFO1 100.6278588 MHz
 NUC1 13C
 PI 10.00 usec
 PLW1 47.00000000 W

----- CHANNEL f2 -----
 SFO2 400.1516006 MHz
 NUC2 1H
 CPDPRG[2] waltz16
 PCPD2 90.00 usec
 PLW2 18.00000000 W
 PLW12 0.34722000 W
 PLW13 0.28125000 W

F2 - Processing parameters
 SI 32768
 SF 100.6177975 MHz
 WDW EM
 SSB 0
 LB 1.00 Hz
 GB 0
 FC 1.40



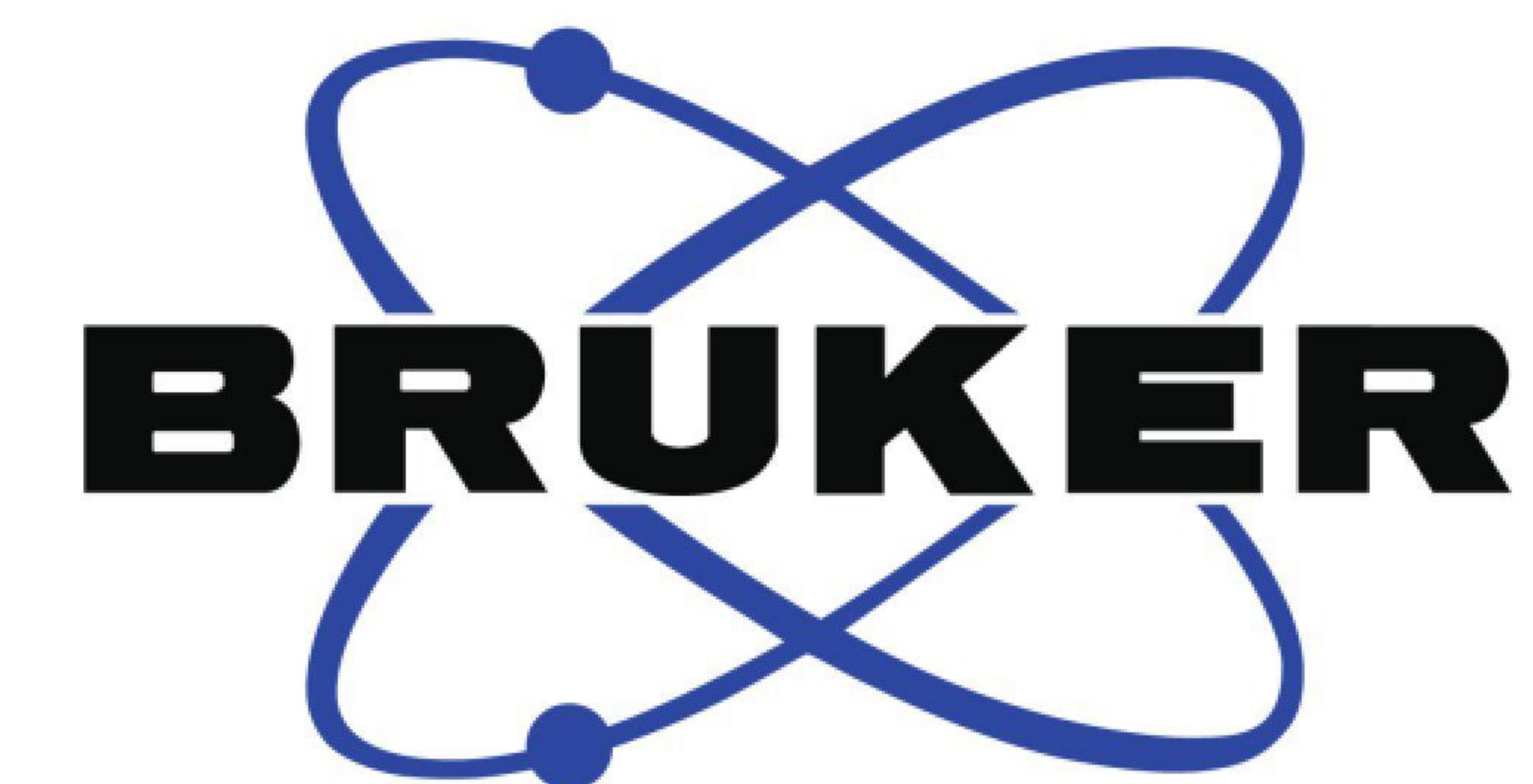



Current Data Parameters
 NAME nagwa-46H
 EXPNO 1
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20181113
 Time 8.13
 INSTRUM spect
 PROBHD 5 mm PABBO BB/
 PULPROG zg30
 TD 65536
 SOLVENT DMSO
 NS 32
 DS 2
 SWH 8012.820 Hz
 FIDRES 0.122266 Hz
 AQ 4.0894465 sec
 RG 205.37
 DW 62.400 usec
 DE 6.50 usec
 TE 300.0 K
 D1 1.0000000 sec
 TD0 1

===== CHANNEL f1 =====
 SFO1 400.1524711 MHz
 NUC1 1H
 P1 12.00 usec
 PLW1 18.00000000 W

F2 - Processing parameters
 SI 65536
 SF 400.1500000 MHz
 WDW EM
 SSB 0
 LB 0.30 Hz
 GB 0
 PC 1.00



Current Data Parameters
 NAME nagwa-46H
 EXPNO 2
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20181113
 Time 8.56
 INSTRUM spect
 PROBHD 5 mm PABBO BB/
 PULPROG zgpg30
 TD 65536
 SOLVENT DMSO
 NS 713
 DS 4
 SWH 24038.461 Hz
 FIDRES 0.366798 Hz
 AQ 1.3631488 sec
 RG 205.37
 DW 20.800 usec
 DE 6.50 usec
 TE 300.0 K
 D1 2.00000000 sec
 D11 0.03000000 sec
 TD0 1

===== CHANNEL f1 =====
 SFO1 100.6278588 MHz
 NUC1 13C
 P1 10.00 usec
 PLW1 47.00000000 W

===== CHANNEL f2 =====
 SFO2 400.1516006 MHz
 NUC2 1H
 CPDPRG[2] waltz16
 PCPD2 90.00 usec
 PLW2 18.00000000 W
 PLW12 0.34722000 W
 PLW13 0.28125000 W

F2 - Processing parameters
 SI 32768
 SF 100.6177975 MHz
 WDW EM
 SSB 0
 LB 1.00 Hz
 GB 0
 PC 1.40