

Electronic Supplementary Information

Visible-light-promoted sulfenylation of 6-aminouracils under catalyst-free condition

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GLOSSARY

Sl. No.	Abbreviation	Full forms
1.	BHT	Butylated hydroxytoluene
2.	Br	Bromo
3.	°C	Degree centigrade
4.	CDCl ₃	Deuterated chloroform
5.	CFL	Compact Fluorescent Light
6.	Cl	Chloro
7.	cm ⁻¹	Centimeter inverse
8.	δ	Chemical shifts
9.	DMF	Dimethylformamide
10.	DMS	dimethylsulfide
11.	DMSO	Dimethyl sulfoxide
12.	DMSO-d ₆	Dimethyl sulfoxide-deuterated
13.	DNA	Deoxyribonucleic acid
14.	ESI-HRMS	High resolution electrospray ionization mass spectrometry
15.	eq.	Equivalent
16.	F	Floro
17.	FT-IR	Fourier Transform Infrared Spectroscopy
18.	g	Gram
19.	h	Hour
20.	HIV	Human immunodeficiency virus
21.	Hz	Hertz
22.	I ₂	Iodine
23.	I.R.	Infrared
24.	J	Coupling constants
25.	KBr	Potassium bromide
26.	KMnO ₄	Potassium permanganate
27.	LC/MS	Liquid Chromatography/mass spectrometry
28.	LED	light-emitting diode
29.	Me	Methyl
30.	MHz	Megahertz
31.	mg	Milligram
32.	mmol	Millimole

33.	mL	Milliliter
34.	M.P.	Melting point
35.	MW	Microwave
36.	NCS	N-chlorosuccinimide
37.	nm	Nanometer
38.	NMR	Nuclear Magnetic Resonance
39.	O ₂	Oxygen
40.	Ph	Phenyl
41.	ppm	Parts per million
42.	Pr	Propyl
43.	Q-TOF	Quadrupole Time-of-Flight
44.	RNA	Ribonucleic acid
45.	r.t.	Room temperature
46.	TEMPO	(2,2,6,6-tetramethylpiperidin-1-yl)oxidanyl
47.	TLC	Thin layer chromatography
48.	TMS	Tetramethylsilane
49.	UV	Ultraviolet
50.	W	Watt

On-Off mechanistic experiments

In a round bottom flask equipped with a stir bar were added the compound **1** (1 mmol), **2** (1 mmol) and DMSO (2 mL) as solvent. The reaction was carried out under CFL (20 W) irradiation in open air for 2 h. Then, the reaction mixture was placed under dark conditions for another 0.5 h. The procedure was repeated for 10 h in total. Between the intervals, the reaction was monitored to calculate the reaction yield.

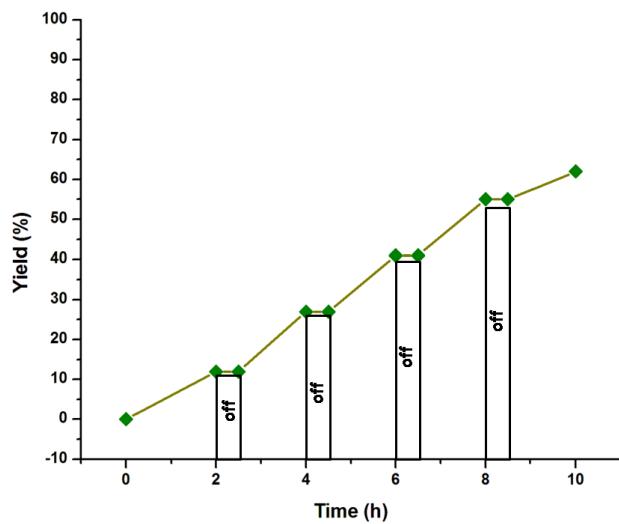


Figure. The “On/Off” experiment showing the effect of visible light on the reaction

Control reaction with TEMPO

To a small round bottom flask equipped with a stir bar were added the compound **1** (1 mmol), **2** (1 mmol), TEMPO (1.5 eq.) and DMSO (2 mL) as solvent. The reaction was carried out under CFL (20 W) irradiation in open air for 8 h. The reaction mixture was analyzed by High resolution mass spectrometry (HRMS) in which the presence of 2,2,6,6-tetramethyl-1-((phenylthio)oxy)piperidine was observed ($[M+H]^+ = 266.1572$), suggesting the formation of thiyl radical in the reaction. The spectrum recorded is presented below.

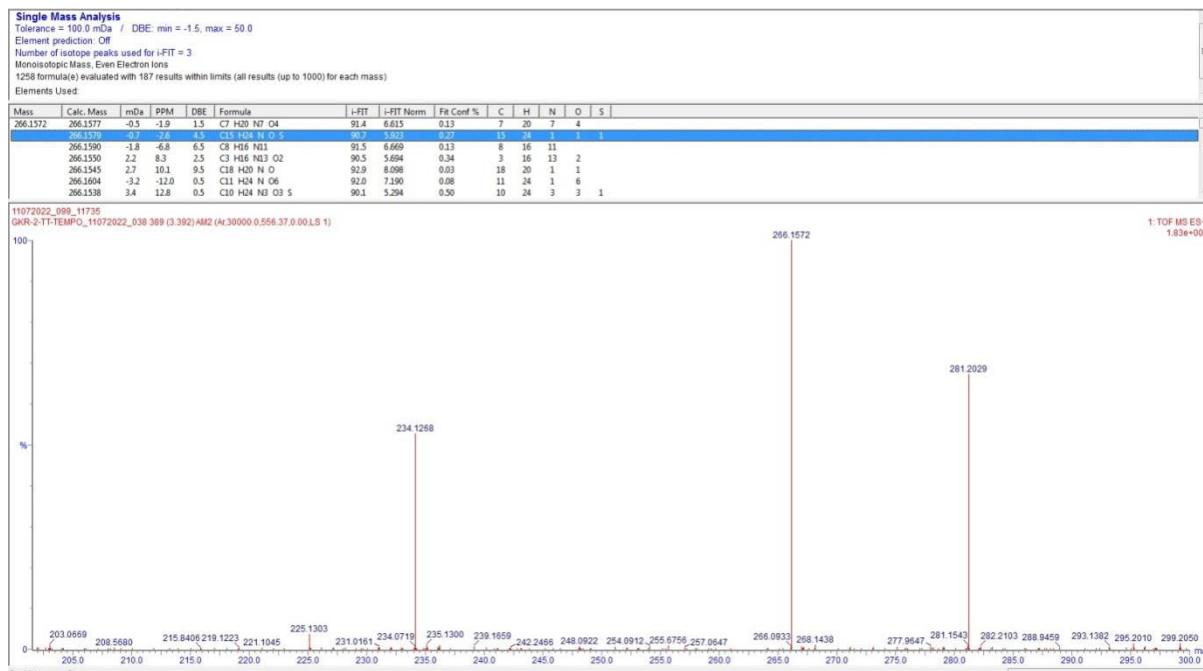


Figure. The adduct formed between thiyl radical and TEMPO identified by HRMS

An image of the photoreactor for the reaction



Figure. 20 W CFL bulb utilized for the photochemical reaction

Characterization of the products

 3a	6-Amino-1,3-dimethyl-5-(phenylthio)pyrimidine-2,4(1H,3H)-dione:³ White solid; Yield 70 %, 184 mg; M.P.:143-145 °C; IR (KBr): 3361, 3247, 3190, 3025, 2946, 2897, 1665, 1562, 1515, 1489, 1420, 1389, 1231, 1192, 1093, 1024, 931, 843, 754, 703, 653 cm ⁻¹ ; ¹ H NMR (300 MHz, CDCl ₃): δ 7.23-7.18 (m, 2H), 7.12-7.10 (m, 3H), 5.93 (bs, 2H), 3.47 (s, 3H), 3.36 (s, 3H); ¹³ C NMR (75 MHz, CDCl ₃): δ 162.2, 157.0, 150.9, 136.9, 129.0, 125.7, 125.5, 77.2, 30.5, 28.9; HRMS (ESI) exact mass calculated for C ₁₂ H ₁₃ N ₃ O ₂ S [M + H] ⁺ : 264.0801; found: 264.0800.
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<p>3b</p>	<p>6-Amino-5-((4-chlorophenyl)thio)-1,3-dimethylpyrimidine-2,4(1H,3H)-dione:^{2, 3} White solid; Yield 93 %, 277 mg; M.P.: 245-247 °C; IR (KBr): 3477, 3341, 3150, 2873, 1701, 1625, 1583, 1549, 1497, 1077, 763, 615 cm⁻¹; ¹H NMR (300 MHz, CDCl₃): δ 7.73-7.70 (m, 1H), 7.52-7.54 (m, 1H), 7.20-7.18 (m, 1H), 7.09-7.07 (m, 1H), 5.83 (bs, 2H), 3.51 (s, 3H), 3.37 (s, 3H); ¹³C NMR (125 MHz, DMSO-<i>d</i>₆): δ 161.0, 157.7, 150.7, 137.3, 129.2, 128.5, 126.8, 73.7, 30.9, 28.1; HRMS (ESI) exact mass calculated for C₁₂H₁₂ClN₃O₂S [M + H]⁺: 298.0412; found: 298.0414.</p>
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<p>3c</p>	<p>6-Amino-5-((4-bromophenyl)thio)-1,3-dimethylpyrimidine-2,4(1H,3H)-dione:³ White solid; Yield 86 %, 294 mg; M.P.: 244-248 °C; IR (KBr): 3744, 3707, 3670, 3643, 3608, 2881, 1517, 1491, 842, 544 cm⁻¹; ¹H NMR (400 MHz, DMSO-<i>d</i>₆): δ 7.42 (d, J = 8.6 Hz, 2H), 7.33 (bs, 2H), 7.07 (d, J = 8.6 Hz, 2H), 3.37 (s, 3H), 3.16 (s, 3H); ¹³C NMR (125 MHz, DMSO-<i>d</i>₆): δ 160.9, 157.7, 150.7, 137.9, 131.4, 127.1, 117.3, 73.6, 30.9, 28.1; HRMS (ESI) exact mass calculated for C₁₂H₁₂BrN₃O₂S [M + H]⁺: 341.9906; found: 341.9908.</p>
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<p>3d</p>	<p>6-Amino-5-((3-bromophenyl)thio)-1,3-dimethylpyrimidine-2,4(1H,3H)-dione: Brown gummy solid; Yield 68 %, 233mg; IR (KBr): 2882, 1761, 1668, 1576, 1523, 1479, 1410, 1323, 264, 1222, 1105, 1035, 947, 901, 832, 785, 693, 562, 498 cm⁻¹; ¹H NMR (400 MHz, DMSO-<i>d</i>₆): δ 7.38 (bs, 2H), 7.29-7.25 (m, 2H), 7.21-7.17 (m, 1H), 7.12-7.20 (m, 1H), 3.36 (s, 3H), 3.16 (s, 3H); ¹³C NMR (100 MHz, DMSO-<i>d</i>₆): δ 161.1, 157.9, 150.9, 141.4, 130.7, 127.6, 127.0, 124.1, 122.2, 73.1, 31.0, 28.3; HRMS (ESI) exact mass calculated for C₁₂H₁₂BrN₃O₂S [M + H]⁺: 341.9906; found: 341.9908.</p>
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<p>3e</p>	<p>6-Amino-5-((4-fluorophenyl)thio)-1,3-dimethylpyrimidine-2,4(1H,3H)-dione: Light brown gummy solid; Yield 78 %, 219 mg; IR (KBr): 2882, 1666, 1567, 1536, 1475, 1388, 1245, 1165, 1139, 1103, 1045, 854, 776, 688, 636, 593, 506, 483 cm⁻¹; ¹H NMR (500 MHz, CDCl₃): δ 7.09-7.06 (m, 2H), 6.86-6.83 (m, 2H), 5.82 (bs, 2H), 3.41 (s, 3H), 3.28 (s, 3H); ¹³C NMR (125 MHz, CDCl₃): δ 161.9, 161.6 (d, J = 245.4 Hz), 156.6, 150.9, 131.3 (d, J = 2.8 Hz), 128.6 (d, J = 8.3 Hz), 116.1 (d, J = 22.1 Hz), 79.5, 31.4, 28.9; HRMS (ESI) exact mass calculated for C₁₂H₁₂FN₃O₂S [M + H]⁺: 282.0707; found: 282.0708.</p>
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<p>3f</p>	<p>6-Amino-5-((2-fluorophenyl)thio)-1,3-dimethylpyrimidine-2,4(1H,3H)-dione: Off-white solid; Yield 72 %, 203mg; M.P.: 237-239°C; IR (KBr): 2878, 1658, 1561, 1532, 1471, 1382, 1241, 1161, 1135, 1101, 1041, 850, 771, 682, 632, 587, 499, 473 cm⁻¹; ¹H NMR (400 MHz, CDCl₃): δ 7.14-7.09 (m, 2H), 7.02-6.94 (m, 2H), 6.11 (bs, 2H), 3.48 (s, 3H), 3.34 (s, 3H); ¹³C NMR (100 MHz, CDCl₃): δ 162.2, 160.1 (d, J = 243.3 Hz), 157.0, 150.9, 129.0, 127.8 (d, J = 7.7 Hz), 124.7 (d, J = 2.9 Hz), 123.1 (d, J = 17.3 Hz), 115.5 (d, J = 21.7 Hz), 76.1, 31.9, 30.7; HRMS (ESI) exact mass calculated for C₁₂H₁₂FN₃O₂S [M + H]⁺: 282.0707; found: 282.0709.</p>
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<p>3g</p>	<p>6-Amino-1,3-dimethyl-5-(<i>p</i>-tolylthio)pyrimidine-2,4(1<i>H</i>,3<i>H</i>)-dione:^{2,3,5,6,7} White solid; Yield 89 %, 202 mg; M.P.: 207-209 °C; IR (KBr): 3328, 3252, 3081, 3051, 2882, 2762, 1867, 1676, 1574, 1523, 1478, 1396, 1327, 1134, 1031, 854, 825, 774, 701, 650, 544, 518 cm⁻¹; ¹H NMR (400 MHz, DMSO-<i>d</i>₆): δ 7.28 (bs, 2H), 7.06-7.00 (m, 4H), 3.36 (s, 3H), 3.15 (s, 3H), 2.22 (s, 3H); ¹³C NMR (100 MHz, DMSO-<i>d</i>₆): δ 161.1, 157.7, 150.8, 134.5, 134.1, 129.4, 125.5, 74.7, 30.9, 28.2, 20.5; HRMS (ESI) exact mass calculated for C₁₃H₁₅N₃O₂S [M + H]⁺: 278.0958; found: 278.0955.</p>
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<p>3h</p>	<p>6-amino-5-((2,4-dimethylphenyl)thio)-1,3-dimethylpyrimidine-2,4(1<i>H</i>,3<i>H</i>)-dione: Yellow solid; Yield 83 %, 242 mg; M.P.: 211-213 °C; IR (KBr): 3348, 3256, 3134, 3048, 2886, 2764, 1864, 1688, 1656, 1600, 1584, 1540, 1500, 1486, 1427, 1376, 1324, 1146, 1101, 1054, 1026, 853, 772, 703, 657, 529, 498 cm⁻¹; ¹H NMR (400 MHz, DMSO-<i>d</i>₆): δ 7.21 (bs, 2H), 6.96 (s, 1H), 6.87-6.84 (m, 1H), 6.70-6.68 (m, 1H), 3.35 (s, 3H), 3.15 (s, 3H), 2.54 (s, 3H), 2.30 (s, 3H); ¹³C NMR (100 MHz, DMSO-<i>d</i>₆): δ 161.0, 157.6, 150.8, 133.8, 133.3, 133.1, 130.6, 126.9, 123.5, 73.2, 30.9, 28.2, 20.3, 19.3; HRMS (ESI) exact mass calculated for C₁₄H₁₇N₃O₂S [M + H]⁺: 292.1114; found: 292.1111.</p>
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<p>3i</p>	<p>6-amino-1,3-dimethyl-5-((4-nitrophenyl)thio)pyrimidine-2,4(1<i>H</i>,3<i>H</i>)-dione:³ Yellow solid; Yield 80 %, 247 mg; M.P.: 289-291 °C; IR (KBr): 3398, 3186, 2872, 2749, 1849, 1687, 1610, 1594, 1534, 1516, 1468, 1430, 1368, 1330, 1203, 1110, 1074, 1026, 857, 768, 707, 667, 604, 557, 477 cm⁻¹; ¹H NMR (400 MHz, DMSO-<i>d</i>₆): δ 8.09-8.07 (m, 2H), 7.44 (bs, 2H), 7.34-7.32 (m, 2H), 3.38 (s, 3H), 3.16 (s, 3H); ¹³C NMR (100 MHz, DMSO-<i>d</i>₆): δ 160.9, 157.8, 150.8, 149.1, 144.5, 125.0, 123.8, 71.9, 31.0, 29.0; HRMS (ESI) exact mass calculated for C₁₂H₁₂N₄O₄S [M + H]⁺: 309.0652; found: 309.0650.</p>
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<p>3j</p>	<p>6-Amino-5-((3-methoxyphenyl)thio)-1,3-dimethylpyrimidine-2,4(1<i>H</i>,3<i>H</i>)-dione:² White solid; Yield 92 %, 269.8 mg; M.P.: 219-221 °C; IR (KBr): 3328, 3186, 2862, 2770, 1854, 1690, 1590, 1548, 1483, 1439, 1377, 1290, 1213, 1100, 1087, 1016, 860, 811, 758, 669, 610, 543, 497 cm⁻¹; ¹H NMR (400 MHz, DMSO-<i>d</i>₆): δ 7.34 (bs, 2H), 7.30 (s, 1H), 7.17-7.13 (m, 1H), 6.69-6.65 (m, 2H), 3.70 (s, 3H), 3.37 (s, 3H), 3.16 (s, 3H); ¹³C NMR (100 MHz, DMSO-<i>d</i>₆): δ 161.1, 159.7, 157.8, 150.8, 139.6, 129.7, 117.3, 110.9, 110.1, 74.0, 55.1, 31.0, 28.3; HRMS (ESI) exact mass calculated for C₁₃H₁₅N₃O₃S [M + H]⁺: 294.0907; found: 294.0905.</p>
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<p>3k</p>	<p>6-Amino-1,3-dimethyl-5-(naphthalen-2-ylthio)pyrimidine-2,4(1<i>H</i>,3<i>H</i>)-dione:^{2,3} White solid; Yield 90 %, 282 mg; M.P.: 299-300 °C; IR (KBr): 3355, 3263, 3067, 1667, 1563, 1520, 1465, 1387, 1346, 1287, 1236, 1118, 1036, 950, 902, 864, 829, 779, 753, 691, 652, 548, 519, 494 cm⁻¹; ¹H NMR (400 MHz, DMSO-<i>d</i>₆): δ 7.84-7.78 (m, 3H), 7.56 (m, 1H), 7.47-7.38 (m, 2H), 7.37 (bs, 2H), 7.30-7.28 (m, 1H), 3.39 (s, 3H), 3.18 (s, 3H); ¹³C NMR (100 MHz, DMSO-<i>d</i>₆): δ 161.2, 157.9, 150.9, 135.8, 133.5, 131.0, 128.2, 127.7, 126.8, 126.5, 125.1, 124.2, 122.1, 73.9, 31.0, 28.3; HRMS (ESI) exact mass calculated for C₁₆H₁₅N₃O₂S [M + H]⁺: 314.0958; found: 314.0959.</p>
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<p>3l</p>	<p>6-Amino-5-(phenylthio)-1,3-dipropylpyrimidine-2,4(1H,3H)-dione: White solid; Yield 89 %, 284 mg; M.P.: 250-252 °C; IR (KBr): 3366, 3258, 3180, 3090, 3026, 2946, 2891, 1672, 1562, 1520, 1482, 1447, 1419, 1389, 1289, 1251, 1192, 1139, 1090, 1035, 919, 842, 809, 753, 708, 653, 497 cm⁻¹; ¹H NMR (400 MHz, DMSO-<i>d</i>₆): δ 7.37 (bs, 2H), 7.27-7.23 (m, 2H), 7.10-7.06 (m, 3H), 3.90 (t, <i>J</i> = 7.3 Hz, 2H), 3.76 (t, <i>J</i> = 8.0 Hz, 2H), 1.62-1.47 (m, 4H), 0.88 (t, <i>J</i> = 7.3 Hz, 3H), 0.82 (t, <i>J</i> = 8.0 Hz, 3H); ¹³C NMR (75 MHz, CDCl₃): δ 162.3, 156.9, 150.6, 128.9, 125.3, 124.9, 76.1, 45.3, 43.5, 21.1, 20.9, 11.2, 10.9; HRMS (ESI) exact mass calculated for C₁₆H₂₁N₃O₂S [M + H]⁺: 320.1427; found: 320.1431.</p>
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<p>3m</p>	<p>6-amino-5-((4-chlorophenyl)thio)-1,3-dipropylpyrimidine-2,4(1H,3H)-dione: Off-white solid; Yield 94 %, 333mg; M.P.: 265-267 °C; IR (KBr): 2947, 2888, 1681, 1636, 1518, 1480, 1443, 1416, 1374, 1327, 1232, 1135, 1016, 914, 840, 790, 702, 616, 549 cm⁻¹; ¹H NMR (500 MHz, DMSO-<i>d</i>₆): δ 7.36 (bs, 2H), 7.31-7.29 (m, 2H), 7.09-7.07 (m, 2H), 3.89 (t, <i>J</i> = 7.4 Hz, 2H), 3.74 (t, <i>J</i> = 7.4 Hz, 2H), 1.59-1.47 (m, 4H), 0.88 (t, <i>J</i> = 7.4 Hz, 3H), 0.82 (t, <i>J</i> = 7.4 Hz, 3H); ¹³C NMR (100 MHz, CDCl₃): δ 161.7, 156.3, 150.5, 134.9, 131.5, 129.1, 127.1, 77.8, 45.5, 43.7, 21.2, 21.1, 11.3, 11.1; HRMS (ESI) exact mass calculated for C₁₆H₂₀ClN₃O₂S [M + H]⁺: 354.1038; found: 354.1037.</p>
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<p>3n</p>	<p>6-Amino-5-((4-bromophenyl)thio)-1,3-dipropylpyrimidine-2,4(1H,3H)-dione: White solid; Yield 76 %, 303mg; M.P.: 266-268 °C; IR (KBr): 2948, 2884, 1681, 1636, 1565, 1521, 1479, 1443, 1417, 1235, 1140, 1095, 1015, 840, 791, 734, 697, 488 cm⁻¹; ¹H NMR (500 MHz, DMSO-<i>d</i>₆): δ 7.43-7.42 (m, 2H), 7.37 (bs, 2H), 7.03-7.01 (m, 2H), 3.88 (t, <i>J</i> = 7.5 Hz, 2H), 3.74 (t, <i>J</i> = 7.3 Hz, 2H), 1.60-1.47 (m, 4H), 0.88 (t, <i>J</i> = 7.3 Hz, 2H), 0.82 (t, <i>J</i> = 7.5 Hz, 2H); ¹³C NMR (100 MHz, CDCl₃): δ 161.7, 156.4, 150.5, 135.6, 132.0, 127.3, 119.3, 77.5, 45.5, 43.7, 21.2, 21.1, 11.3, 11.1; HRMS (ESI) exact mass calculated for C₁₆H₂₀BrN₃O₂S [M + H]⁺: 398.0532; found: 398.0530.</p>
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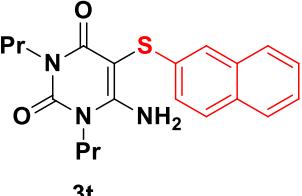
<p>3o</p>	<p>6-Amino-5-((3-bromophenyl)thio)-1,3-dipropylpyrimidine-2,4(1H,3H)-dione: Light brown solid; Yield 70 %, 279 mg; M.P.: 270-272 °C; IR (KBr): 3324, 3276, 3023, 2947, 2888, 1672, 1634, 1561, 1517, 1476, 1447, 1417, 1372, 1311, 1238, 1213, 1119, 917, 871, 797, 707, 635, 536, 492 cm⁻¹; ¹H NMR (500 MHz, DMSO-<i>d</i>₆): δ 7.39 (bs, 2H), 7.29-7.27 (m, 1H), 7.22-7.19 (m, 1H), 7.17 (m, 1H), 7.10-1.09 (m, 1H), 3.90 (t, <i>J</i> = 7.4 Hz, 2H), 3.76 (t, <i>J</i> = 7.1 Hz, 2H), 1.59-1.50 (m, 4H), 0.88 (t, <i>J</i> = 7.1 Hz, 3H), 0.82 (t, <i>J</i> = 7.4 Hz, 3H); ¹³C NMR (125 MHz, DMSO-<i>d</i>₆): δ 160.6, 157.0, 150.4, 141.1, 130.7, 127.5, 126.8, 123.9, 122.1, 73.4, 44.5, 42.4, 20.7, 20.5, 11.0, 10.5; HRMS (ESI) exact mass calculated for C₁₆H₂₀BrN₃O₂S [M + H]⁺: 398.0532; found: 398.0534.</p>
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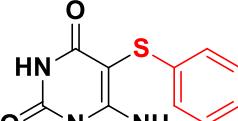
<p>3p</p>	<p>6-amino-5-((4-fluorophenyl)thio)-1,3-dipropylpyrimidine-2,4(1H,3H)-dione: Brown liquid; Yield 80 %, 270 mg; IR (KBr): 3345, 3269, 3187, 2999, 2920, 2887, 2768, 1671, 1629, 1559, 1518, 1477, 1443, 1411, 1378, 1348, 1327, 1279, 1231, 1161, 1136, 1086, 958, 910, 859, 796, 728, 699, 613, 543, 499 cm⁻¹; ¹H NMR (100 MHz, CDCl₃): δ 7.08-6.86 (m, 4H), 6.31 (bs, 2H), 3.96-3.86 (m, 4H), 1.71-1.60 (m, 4H), 0.94-0.88 (m, 6H); ¹³C NMR (100 MHz, CDCl₃): δ 162.1, 161.2 (d, <i>J</i> = 246.6 Hz), 156.6, 150.5, 131.4 (d, <i>J</i> = 2.9 Hz), 127.4 (d, <i>J</i> = 7.7 Hz), 116.1 (d, <i>J</i> = 22.2 Hz), 77.7, 45.5, 43.6, 21.1 (2C), 11.3, 11.0; HRMS (ESI) exact mass calculated for C₁₆H₂₀FN₃O₂S [M + H]⁺: 338.1333; found: 338.1335.</p>
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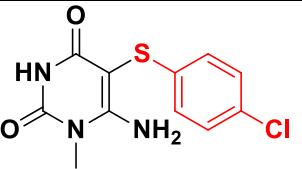
<p>3q</p>	<p>6-Amino-5-((3-fluorophenyl)thio)-1,3-dipropylpyrimidine-2,4(1H,3H)-dione: Yellow solid; Yield 78 %, 263 mg; M.P.: 269-271 °C; IR (KBr): 3324, 3276, 3192, 3014, 2946, 2892, 2779, 1680, 1637, 1562, 1520, 1481, 1450, 1417, 1388, 1353, 1331, 1289, 1235, 1165, 1140, 1088, 967, 918, 861, 808, 734, 708, 619, 547, 500 cm⁻¹; ¹H NMR (400 MHz, DMSO-d₆): δ 7.41 (bs, 2H), 7.32-7.26 (m, 1H), 6.94-6.88 (m, 2H), 6.86-6.83 (m, 1H), 3.89 (t, <i>J</i> = 7.3 Hz, 2H), 3.75 (t, <i>J</i> = 8.0 Hz, 2H), 1.61-1.47 (m, 4H), 0.88 (t, <i>J</i> = 7.3 Hz, 3H), 0.82 (t, <i>J</i> = 8.0 Hz, 3H); ¹³C NMR (100 MHz, DMSO-d₆): δ 162.7 (d, <i>J</i> = 244.7 Hz), 160.7, 157.1, 150.5, 141.4 (d, <i>J</i> = 7.7 Hz), 130.6 (d, <i>J</i> = 8.7 Hz), 120.8, 111.5 (d, <i>J</i> = 18.3 Hz), 111.3 (d, <i>J</i> = 21.2 Hz), 73.3, 44.6, 42.5, 20.8, 20.6, 11.2, 10.7; HRMS (ESI) exact mass calculated for C₁₆H₂₀FN₃O₂S [M + H]⁺: 338.1333; found: 338.1336.</p>
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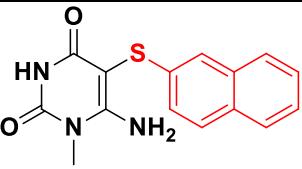
<p>3r</p>	<p>6-Amino-5-((2-fluorophenyl)thio)-1,3-dipropylpyrimidine-2,4(1H,3H)-dione: White solid; Yield 73 %, 246 mg; M.P.: 264-266 °C; IR (KBr): 3341, 3263, 3183, 2991, 2916, 2883, 2762, 1669, 1623, 1553, 1516, 1473, 1437, 1407, 1372, 1339, 1325, 1273, 1227, 1156, 1130, 1082, 952, 899, 851, 792, 722, 691, 611, 541, 493 cm⁻¹; ¹H NMR (400 MHz, DMSO-d₆): δ 7.43 (bs, 2H), 7.20-7.05 (m, 3H), 6.85-6.81 (m, 1H), 3.89 (t, <i>J</i> = 7.3 Hz, 2H), 3.74 (t, <i>J</i> = 7.3 Hz, 2H), 1.59-1.46 (m, 4H), 0.88 (t, <i>J</i> = 7.3 Hz, 3H), 0.81 (t, <i>J</i> = 7.3 Hz, 3H); ¹³C NMR (100 MHz, DMSO-d₆): δ 160.7, 158.7 (d, <i>J</i> = 240.8 Hz), 157.3, 150.5, 126.1 (d, <i>J</i> = 7.7 Hz), 125.7 (d, <i>J</i> = 2.9 Hz), 125.1 (d, <i>J</i> = 16.4 Hz), 126.9 (d, <i>J</i> = 2.9 Hz), 115.2 (d, <i>J</i> = 21.2 Hz), 70.6, 44.6, 42.5, 20.8, 20.7, 11.2, 10.7; HRMS (ESI) exact mass calculated for C₁₆H₂₀FN₃O₂S [M + H]⁺: 338.1333; found: 338.1337.</p>
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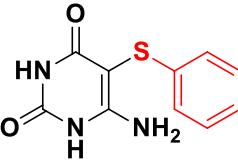
<p>3s</p>	<p>6-Amino-5-((4-nitrophenyl)thio)-1,3-dipropylpyrimidine-2,4(1H,3H)-dione: Light yellow solid; Yield 86 %, 313 mg; M.P.: 278-280 °C; IR (KBr): 3438, 3354, 3198, 3176, 3078, 3026, 2943, 2887, 1873, 1687, 1590, 1560, 1531, 1498, 1443, 1385, 1329, 1247, 1120, 1089, 1045, 855, 745, 713, 669, 501 cm⁻¹; ¹H NMR (400 MHz, DMSO-d₆): δ 8.11-8.08 (m, 2H), 7.48 (bs, 2H), 7.29-7.27 (m, 2H), 3.89 (t, <i>J</i> = 6.1 Hz, 2H), 3.75 (t, <i>J</i> = 6.1 Hz, 2H), 1.58-1.49 (m, 4H), 0.89 (t, <i>J</i> = 7.3 Hz, 3H), 0.82 (t, <i>J</i> = 7.3 Hz, 3H); ¹³C NMR (100 MHz, DMSO-d₆): δ 160.5, 157.0, 150.4, 149.0, 144.5, 124.9, 123.9, 72.0, 44.6, 42.6, 20.8, 20.6, 11.2, 10.7; HRMS (ESI) exact mass calculated for C₁₆H₂₀N₄O₄S [M + H]⁺: 365.1278; found: 365.1280.</p>
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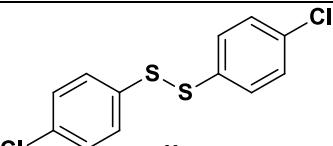
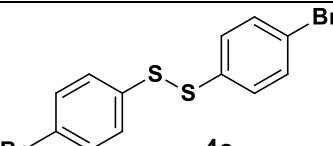
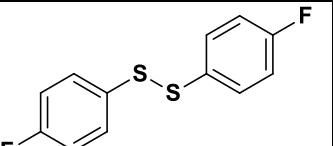
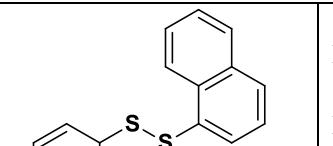
 3t	<p>6-Amino-5-(naphthalen-2-ylthio)-1,3-dipropylpyrimidine-2,4(1H,3H)-dione: Brown gummy liquid; Yield 92 %, 340 mg; IR (KBr): 3369, 3024, 2887, 2455, 1930, 1867, 1565, 1522, 1478, 1421, 1390, 1314, 1244, 1163, 1101, 1042, 985, 921, 868, 837, 792, 712, 692, 657, 582, 494 cm⁻¹; ¹H NMR (500 MHz, CDCl₃): δ 7.57-7.55 (m, 1H), 7.47-7.43 (m, 2H), 7.30-7.24 (m, 3H), 6.99-6.98 (m, 1H), 6.28 (bs, 2H), 3.78 (m, 4H), 1.56-1.52 (m, 4H), 0.81-0.71 (m, 6H); ¹³C NMR (125 MHz, CDCl₃): δ 162.2, 156.8, 150.6, 133.9, 133.7, 131.6, 128.6, 127.7, 126.8, 126.5, 125.3, 124.0, 123.1, 77.1, 45.4, 43.6, 21.1 (2C), 11.2, 10.8; HRMS (ESI) exact mass calculated for C₂₀H₂₃N₃O₂S [M + H]⁺: 370.1584; found: 370.1582.</p>
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 3u	<p>6-Amino-1-methyl-5-(phenylthio)pyrimidine-2,4(1H,3H)-dione:⁷ Off-white solid; Yield 63 %, 157 mg; M.P.: 278-280 °C; IR (KBr): 3391, 3301, 3121, 2965, 2883, 1717, 1612, 1553, 1491, 1417, 1011, 845, 751, 631 cm⁻¹; ¹H NMR (500 MHz, DMSO-d₆): δ 10.8 (bs, 1H), 7.27-7.23 (m, 4H), 7.10-7.09 (m, 3H), 3.30 (s, 3H); ¹³C NMR (125 MHz, DMSO-d₆): δ 161.5, 159.0, 150.3, 138.1, 128.7, 124.9, 124.6, 74.0, 29.8; HRMS (ESI) exact mass calculated for C₁₁H₁₁N₃O₂S [M + H]⁺: 250.0645; found: 250.0642.</p>
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 3v	<p>6-Amino-5-((4-chlorophenyl)thio)-1-methylpyrimidine-2,4(1H,3H)-dione:² White solid; Yield 67 %, 190 mg; M.P.: 293-295°C; IR (KBr): 2882, 1682, 1568, 1531, 1483, 1404, 1213, 1105, 1032, 846, 777, 516 cm⁻¹; ¹H NMR (400 MHz, DMSO-d₆): δ 11.1 (bs, 1H), 7.81 (bs, 2H), 7.65-7.63 (m, 2H), 7.62-7.59 (m, 2H), 3.16 (s, 3H); ¹³C NMR (100 MHz, DMSO-d₆): δ 162.4, 156.6, 151.2, 147.7, 136.2, 129.1, 128.7, 126.6, 126.2, 75.1, 31.4; HRMS (ESI) exact mass calculated for C₁₁H₁₀ClN₃O₂S [M + H]⁺: 284.0255; found: 284.0258.</p>
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 3w	<p>6-Amino-1-methyl-5-(naphthalen-2-ylthio)pyrimidine-2,4(1H,3H)-dione:² Off-white solid; Yield 66 %, 198 mg; M.P.: 297-299 °C; IR (KBr): 2881, 1682, 1566, 1525, 1478, 1417, 1323, 1252, 1116, 1133, 1088, 1045, 873, 843, 785, 717, 691, 667, 557, 527, 491 cm⁻¹; ¹H NMR (500 MHz, DMSO-d₆): δ 11.05 (bs, 1H), 8.28 (m, 1H), 8.10-8.08 (m, 1H), 8.06-8.05 (m, 1H), 8.00-7.98 (m, 1H), 7.82 (bs, 2H), 7.63-7.61 (m, 3H), 3.15 (s, 3H); ¹³C NMR (125 MHz, DMSO-d₆): δ 159.7, 156.1, 150.0, 142.2, 133.6, 132.2, 129.3, 128.4, 127.8, 127.6, 127.2, 123.9, 120.9, 85.6, 28.2; HRMS (ESI) exact mass calculated for C₁₅H₁₃N₃O₂S [M + H]⁺: 300.0801; found: 300.0803.</p>
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 3x	<p>6-Amino-5-(phenylthio)pyrimidine-2,4(1H,3H)-dione:⁷ White solid; Yield 59 %, 139 mg; M.P.: >300°C; IR (KBr): 3390, 3408, 3283, 2958, 2879, 2787, 1713, 1627, 1608, 1558, 1482, 1400, 1006, 843, 749, 627 cm⁻¹; ¹H NMR (400 MHz, DMSO-d₆): δ 10.6 (bs, 1H), 10.4 (bs, 1H), 7.27-7.23 (m, 2H), 7.11-7.06 (m, 3H), 6.64 (bs, 2H); ¹³C NMR (100 MHz, DMSO-d₆): δ 163.2, 158.0, 149.9, 138.3, 128.9, 124.9, 124.7, 73.2; HRMS (ESI) exact mass calculated for C₁₀H₉N₃O₂S [M + H]⁺: 236.0488; found: 236.0485.</p>
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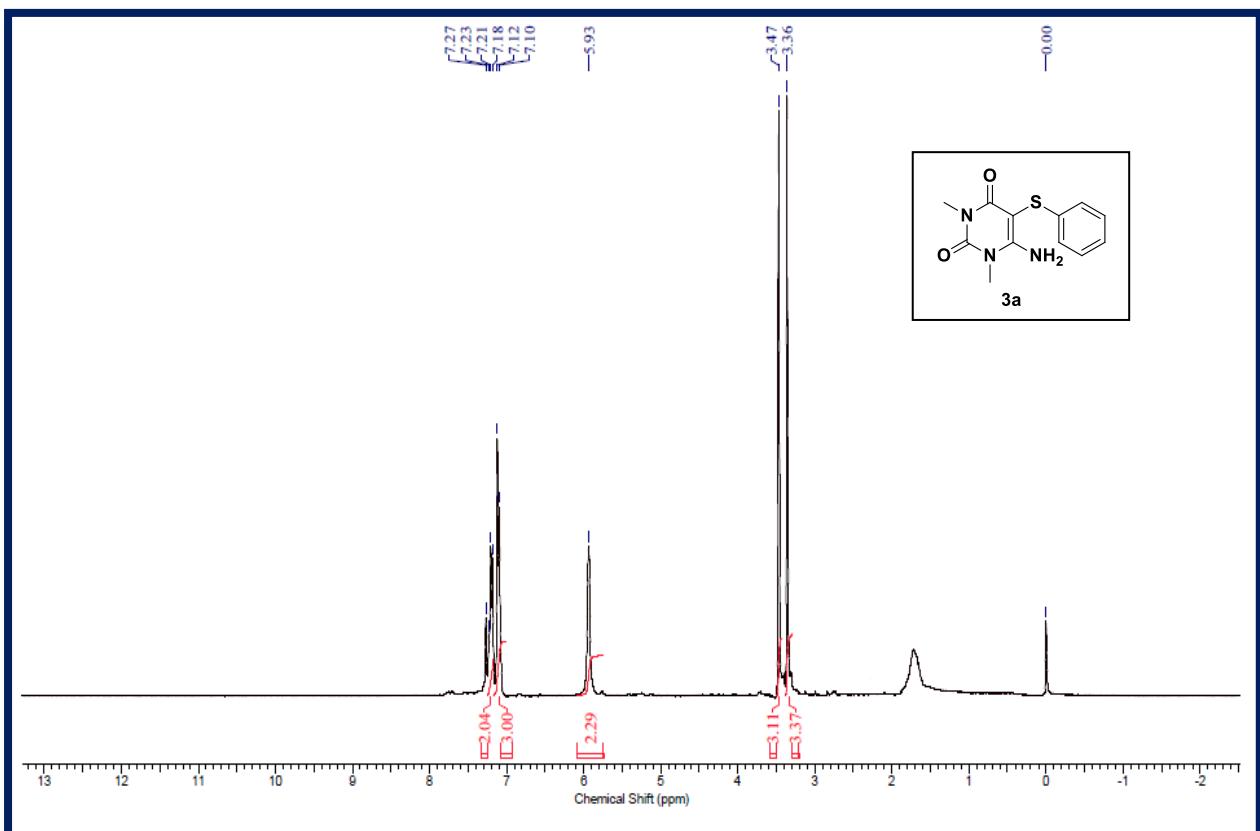
 4b	<p>1,2-Bis(4-chlorophenyl)disulfane:^{1,4} White solid; Yield 76%, 218 mg; M.P.: 68-70 °C; IR (KBr): 2880, 1577, 1487, 1400, 1302, 1130, 1017, 961, 848, 751, 701, 560, 524 cm⁻¹; ¹H NMR (500 MHz, DMSO-<i>d</i>₆): δ 7.52-7.50 (m, 4H), 7.44-7.41 (m, 4H); ¹³C NMR (125 MHz, DMSO-<i>d</i>₆): δ 134.6, 132.7, 129.6, 129.3; HRMS (ESI) exact mass calculated for C₁₂H₈Cl₂S₂[M + H]⁺: 286.9517; found: 286.9514.</p>
 4c	<p>1,2-Bis(4-bromophenyl)disulfane:⁴ White solid; Yield 80%, 301 mg; M.P.: 90-93 °C; IR (KBr): 2876, 1574, 1479, 1394, 1301, 1131, 1095, 1013, 841, 730, 698, 509, 482 cm⁻¹; ¹H NMR (400 MHz, CDCl₃): δ 7.42-7.39 (m, 4H), 7.33-7.30 (m, 4H); ¹³C NMR (100 MHz, CDCl₃): δ 135.6, 132.1, 129.3, 121.5; HRMS (ESI) exact mass calculated for C₁₂H₈Br₂S₂[M + H]⁺: 374.8507; found: 374.8504.</p>
 4d	<p>1,2-Bis(4-fluorophenyl)disulfane: Off-white solid; Yield 78 %, 198 mg; M.P.: 74-76°C; IR (KBr): 3880, 3816, 3683, 2882, 2328, 1606, 1535, 1414, 1278, 1171, 1127, 854, 667, 642, 444 cm⁻¹; ¹H NMR (400 MHz, CDCl₃): δ 7.45-7.41 (m, 4H), 7.02-6.97 (m, 4H); ¹³C NMR (100 MHz, CDCl₃): δ 162.6 (d, <i>J</i> = 248.5 Hz), 132.1 (d, <i>J</i> = 2.9 Hz), 131.2 (d, <i>J</i> = 8.7 Hz), 116.4 (d, <i>J</i> = 22.2 Hz); HRMS (ESI) exact mass calculated for C₁₂H₈F₂S₂[M + H]⁺: 255.0108; found: 255.0105.</p>
 4e	<p>1,2-Di(naphthalen-1-yl)disulfane:^{1,4} White solid; Yield 82 %, 261 mg; M.P.: 138-140°C; IR (KBr): 2878, 1586, 1500, 1467, 1409, 1383, 1271, 1121, 1088, 1015, 853, 839, 775, 707, 671, 537, 517, 471 cm⁻¹; ¹H NMR (300 MHz, CDCl₃): δ 7.98 (m, 2H), 7.80-7.72 (m, 6H), 7.63-7.60 (m, 2H), 7.46-7.45 (m, 4H); ¹³C NMR (125 MHz, CDCl₃): δ 134.1, 133.4, 132.4, 128.9, 127.7, 127.4, 126.7, 126.4, 126.2, 125.5; HRMS (ESI) exact mass calculated for C₂₀H₁₄S₂[M + H]⁺: 319.0610; found: 319.0607.</p>

References:

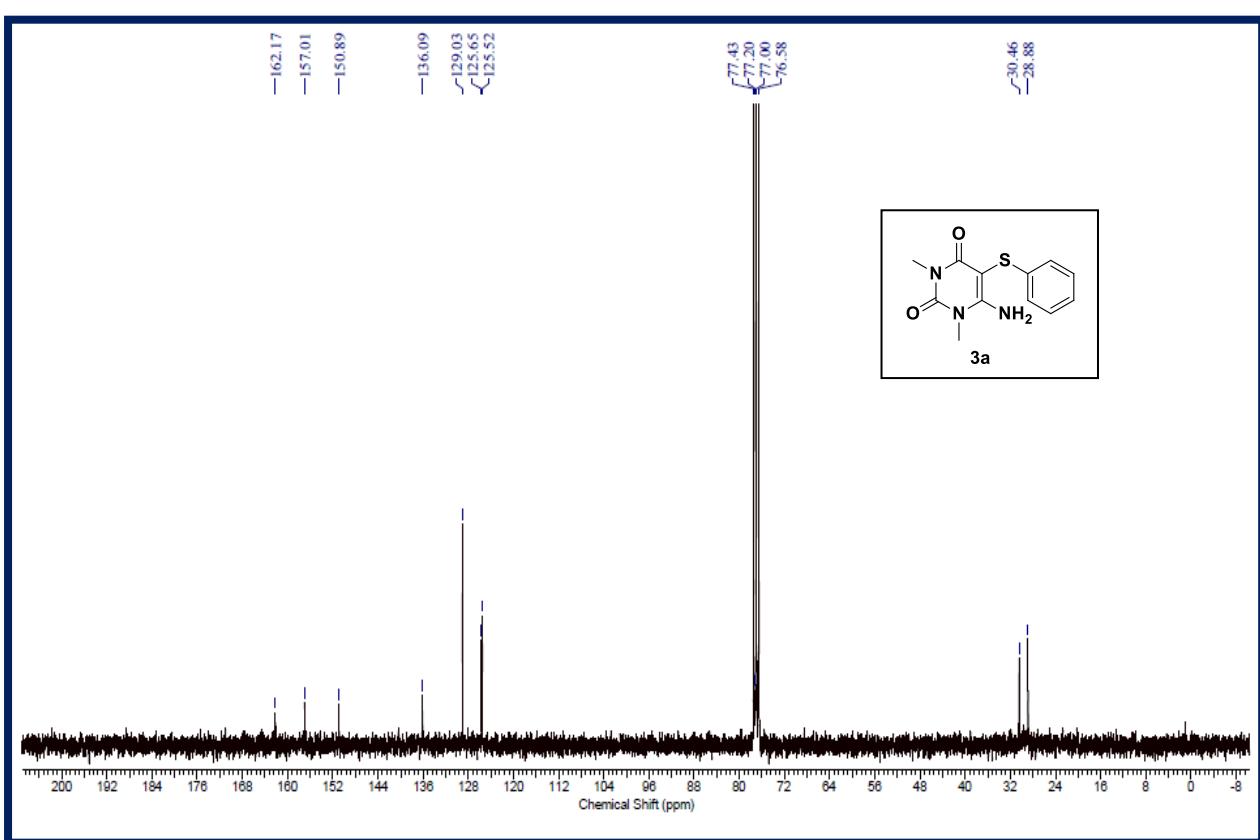
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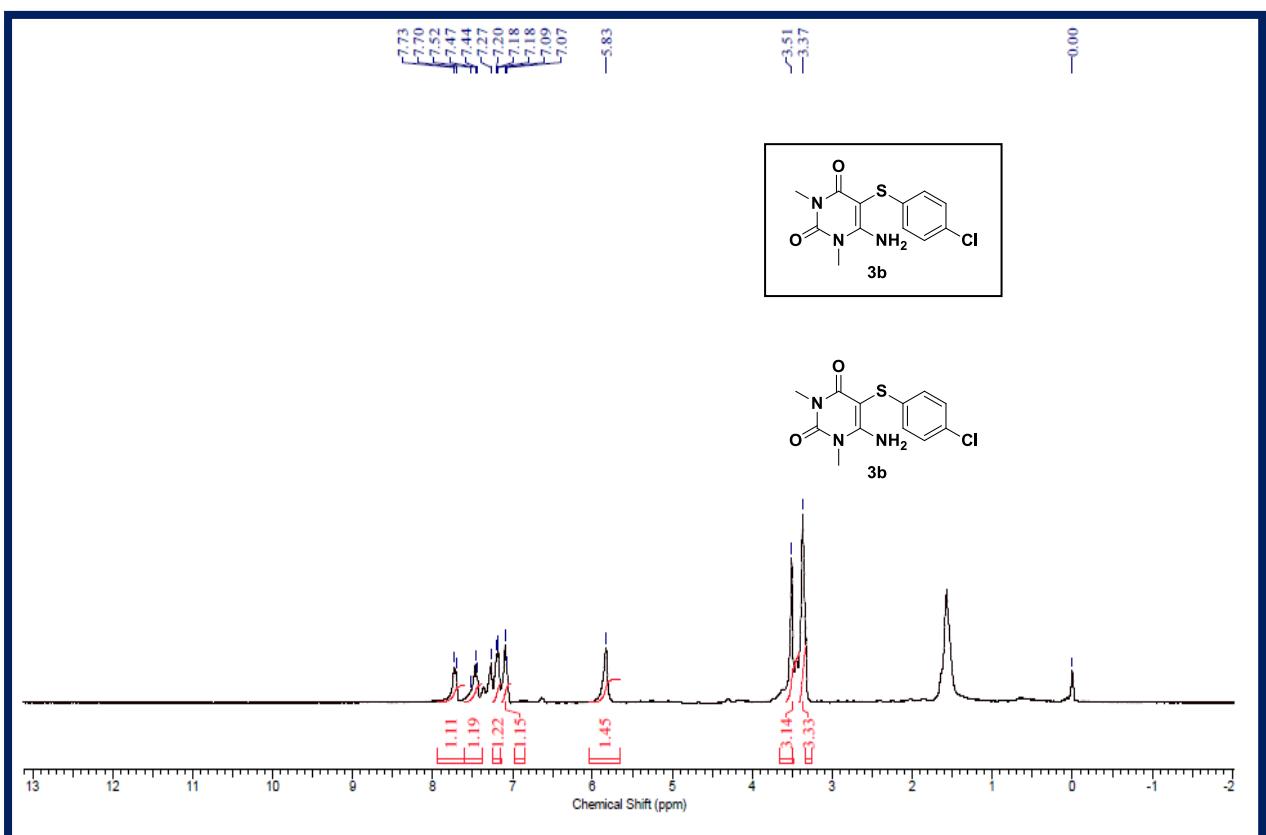
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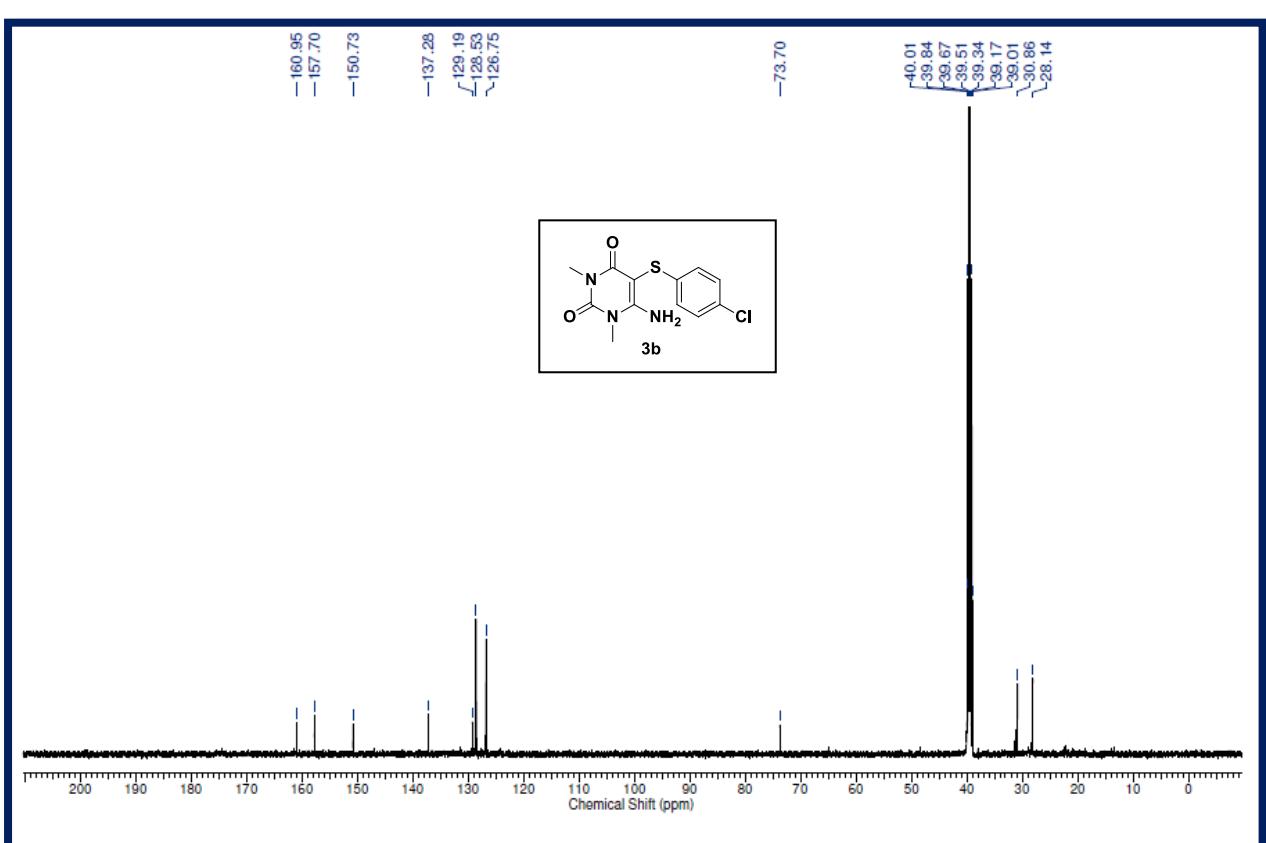
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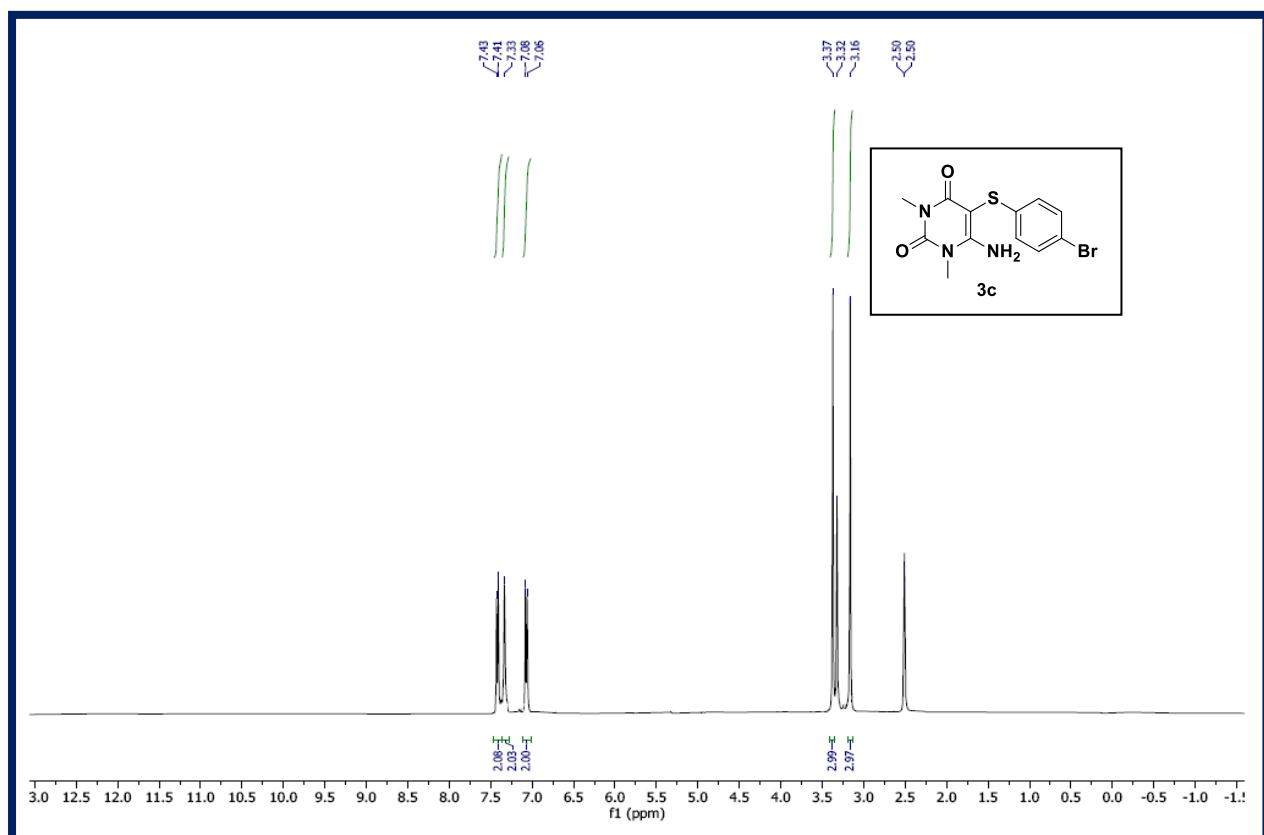
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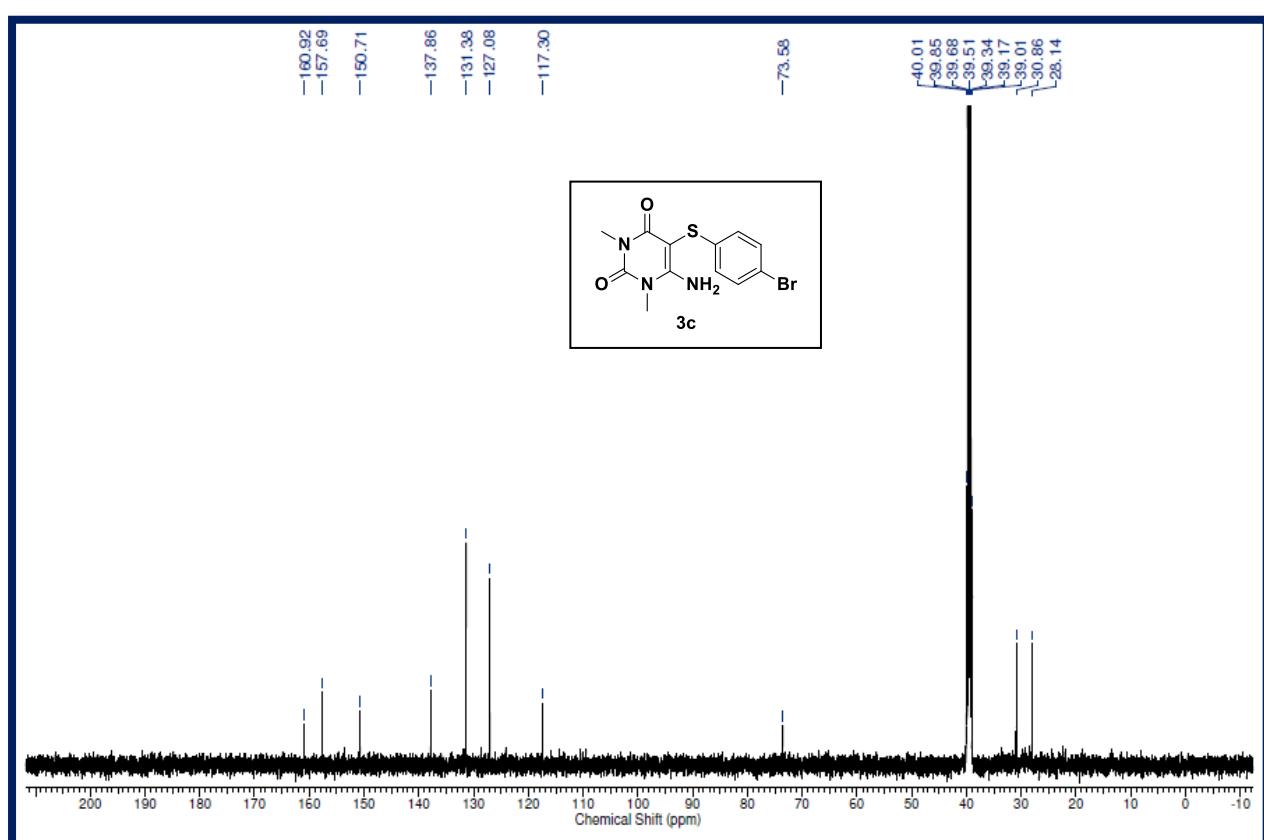
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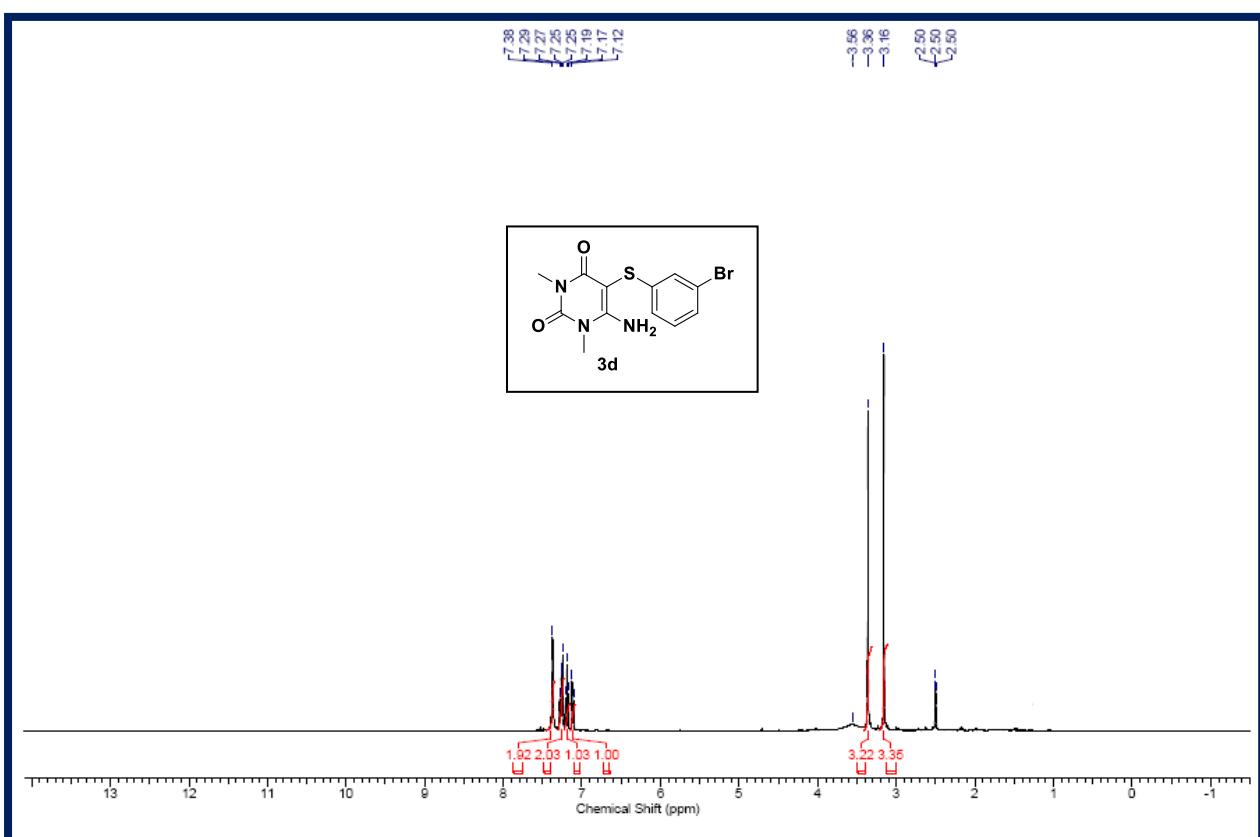
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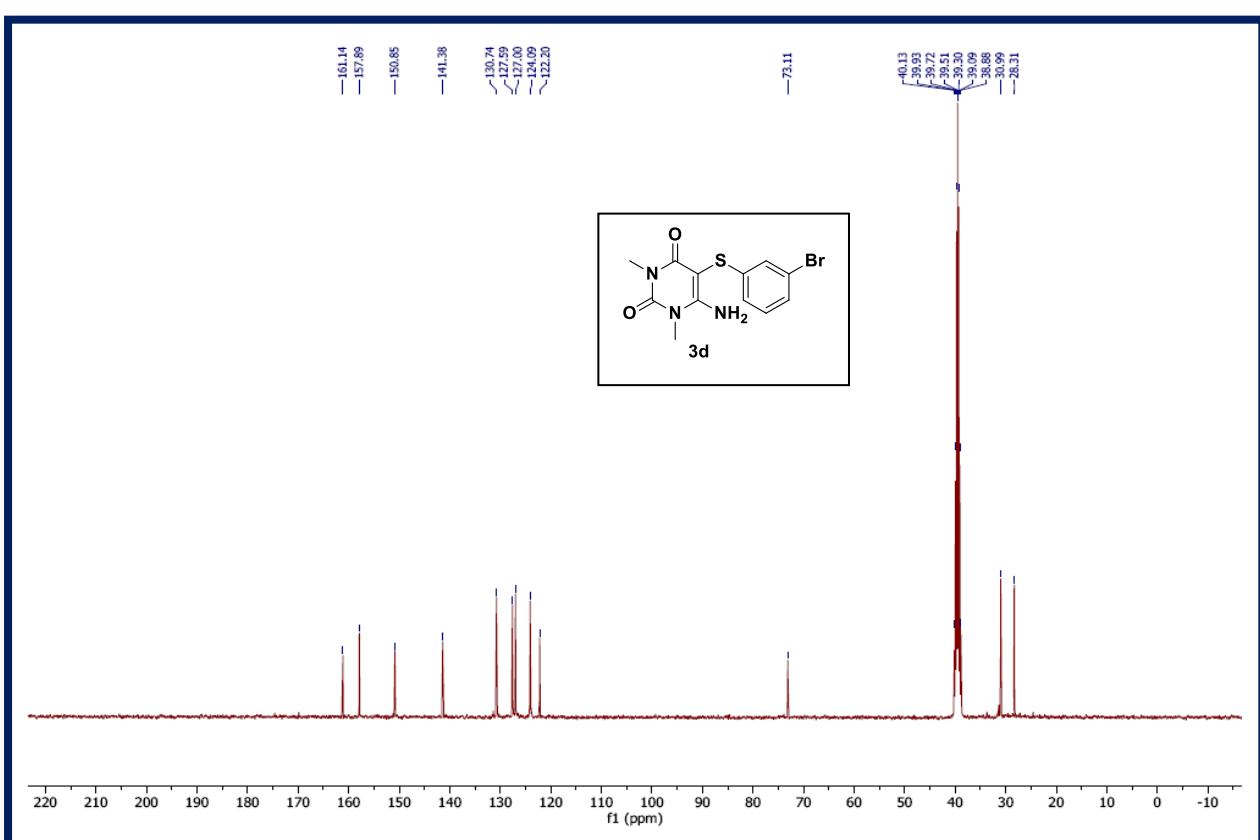
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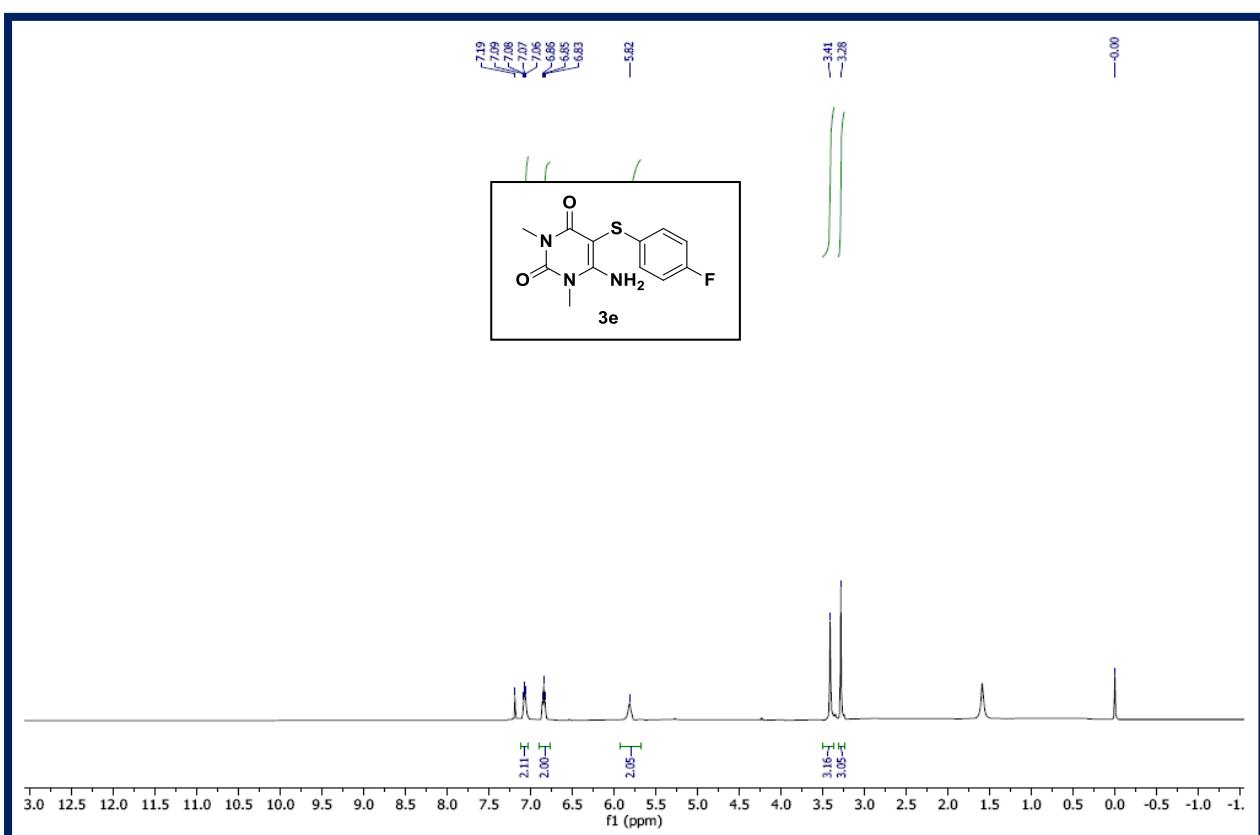
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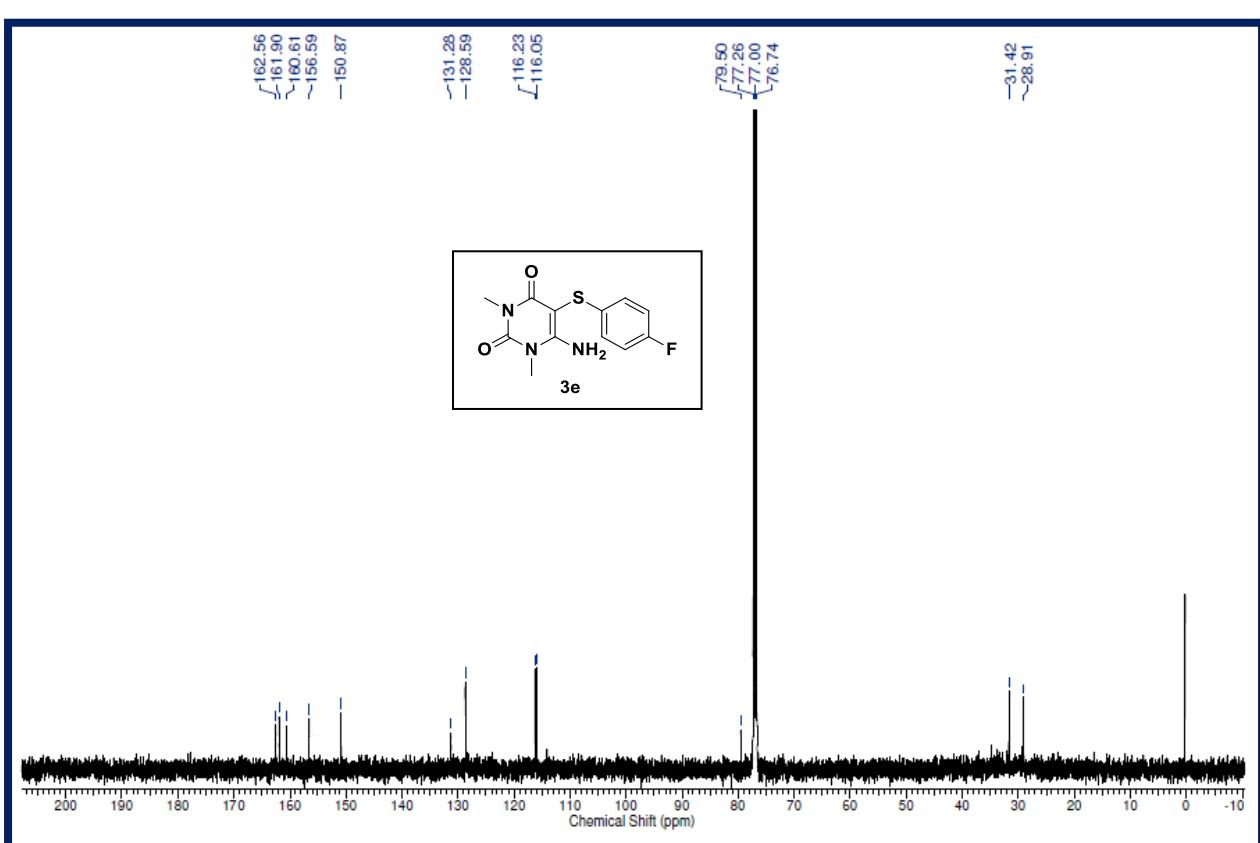
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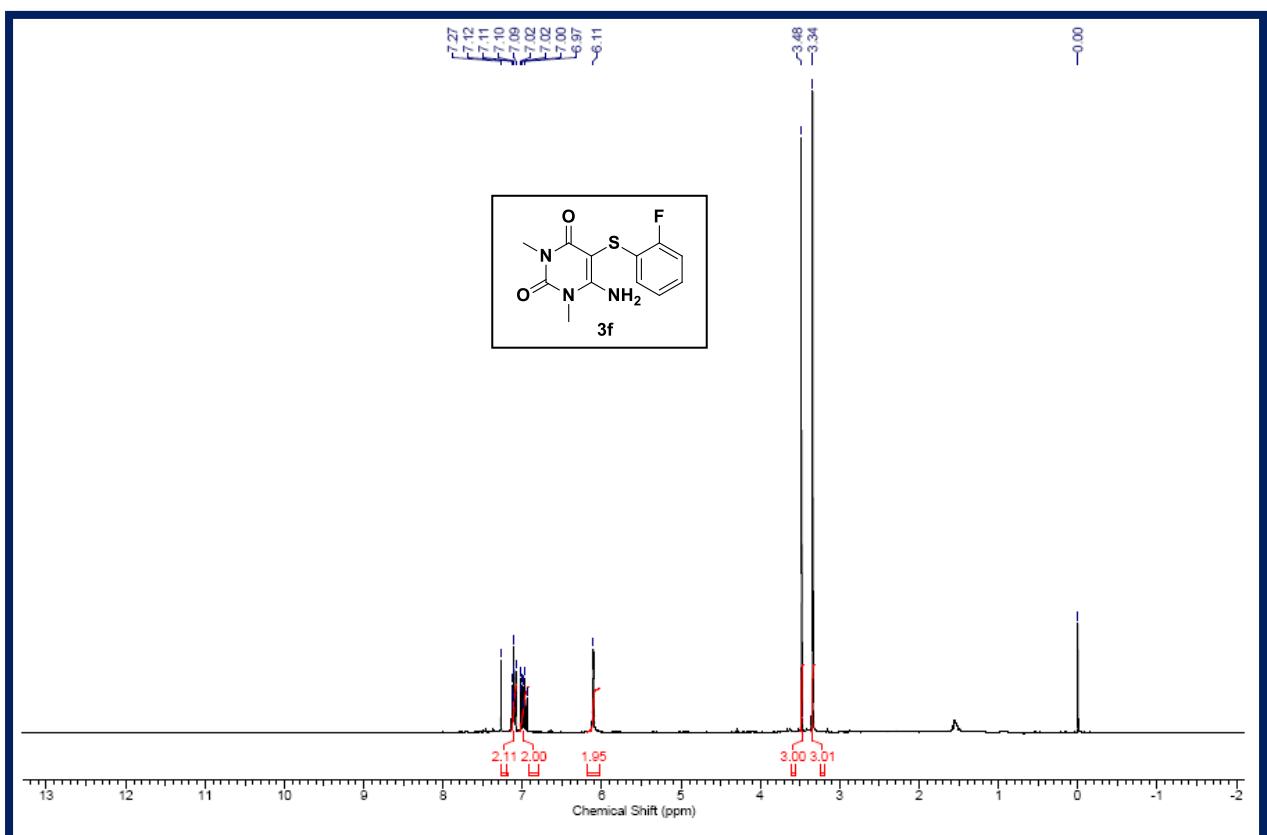
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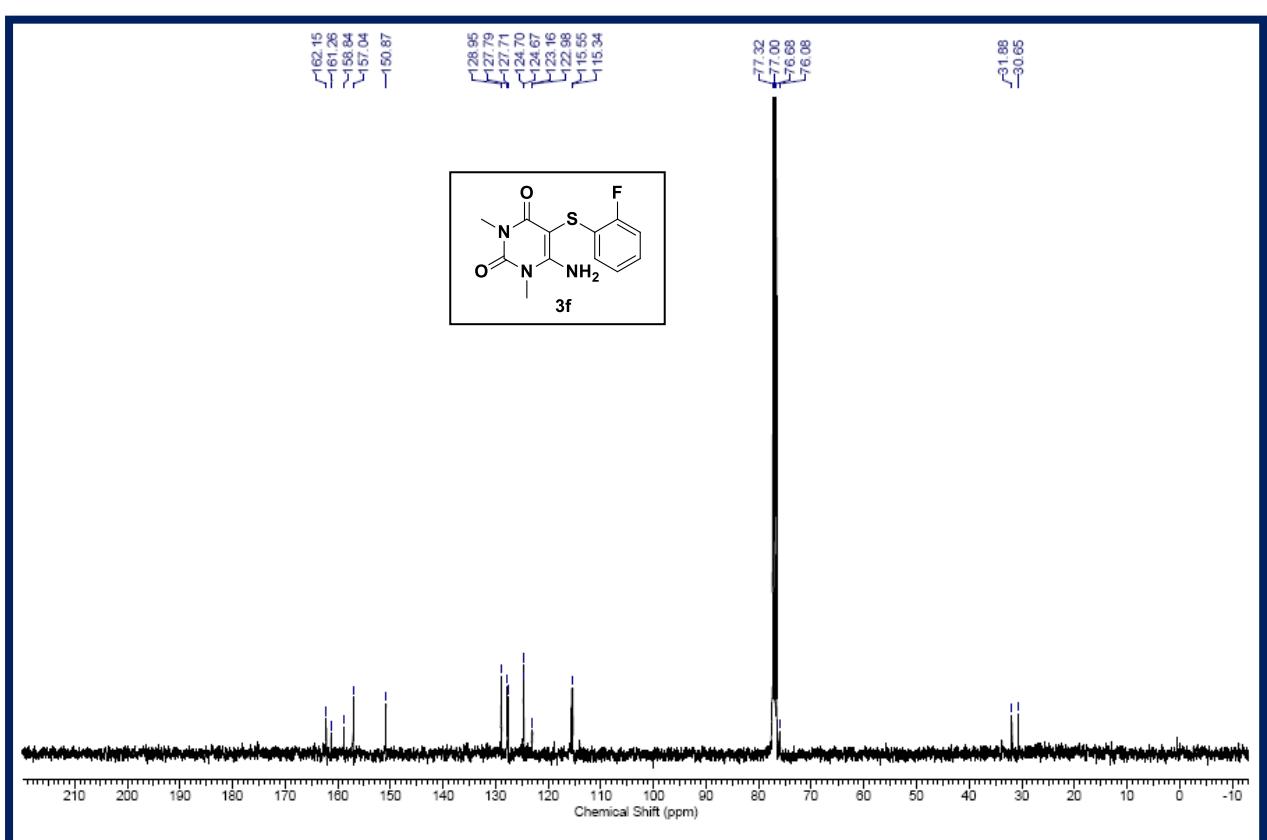
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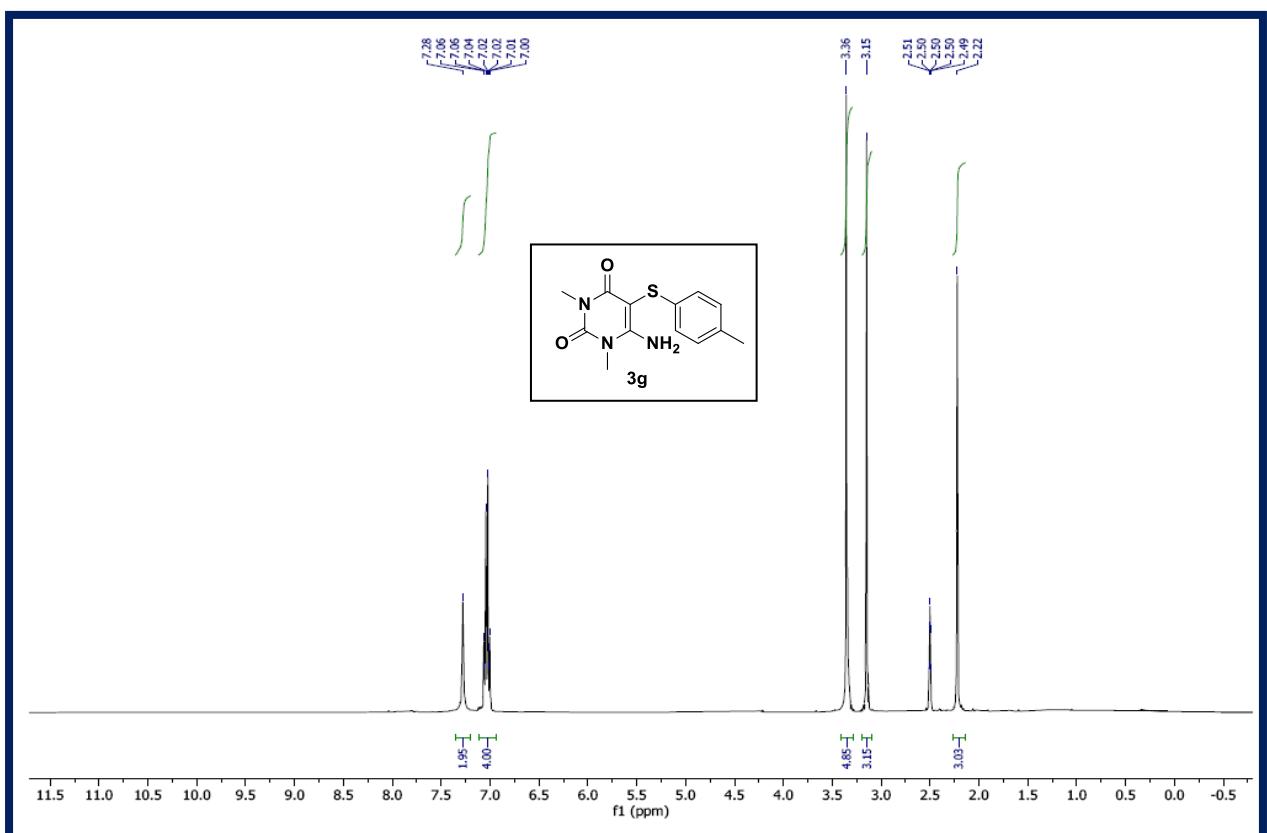
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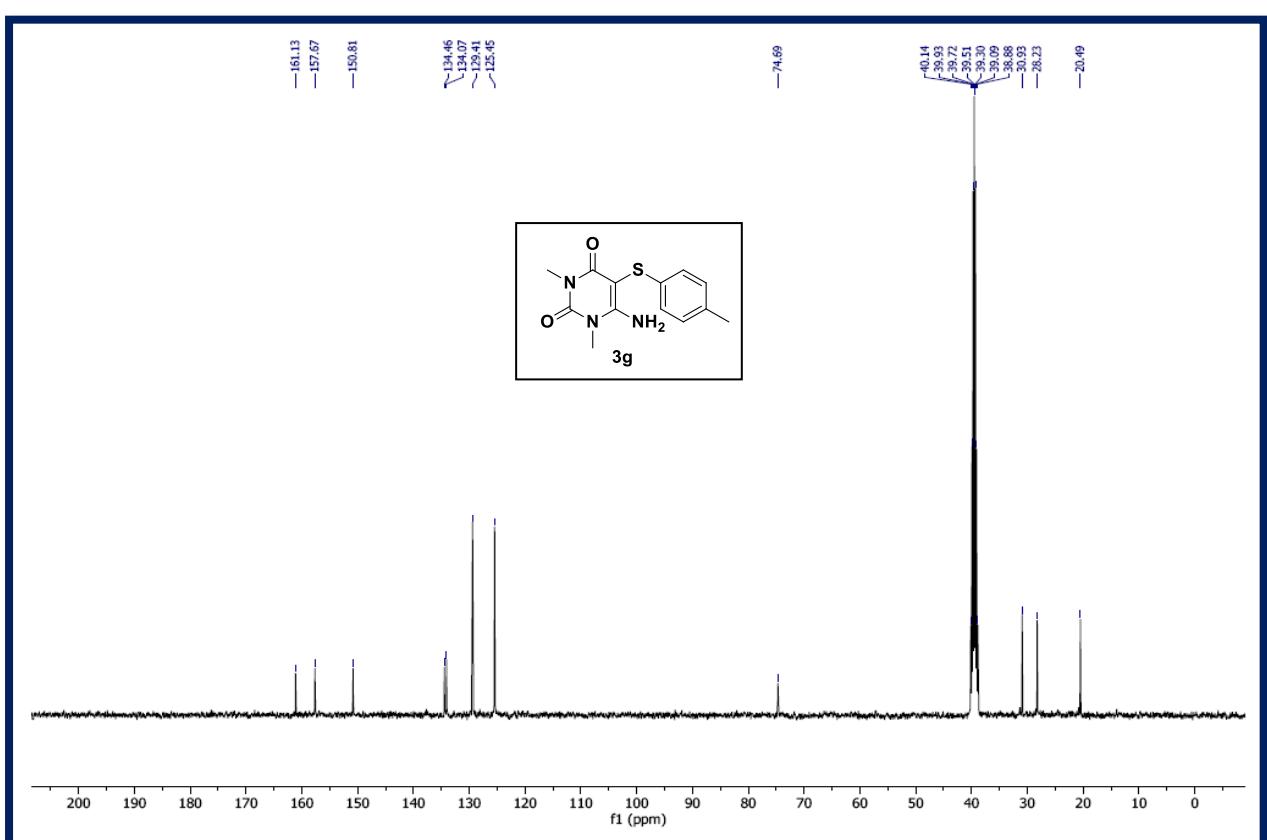
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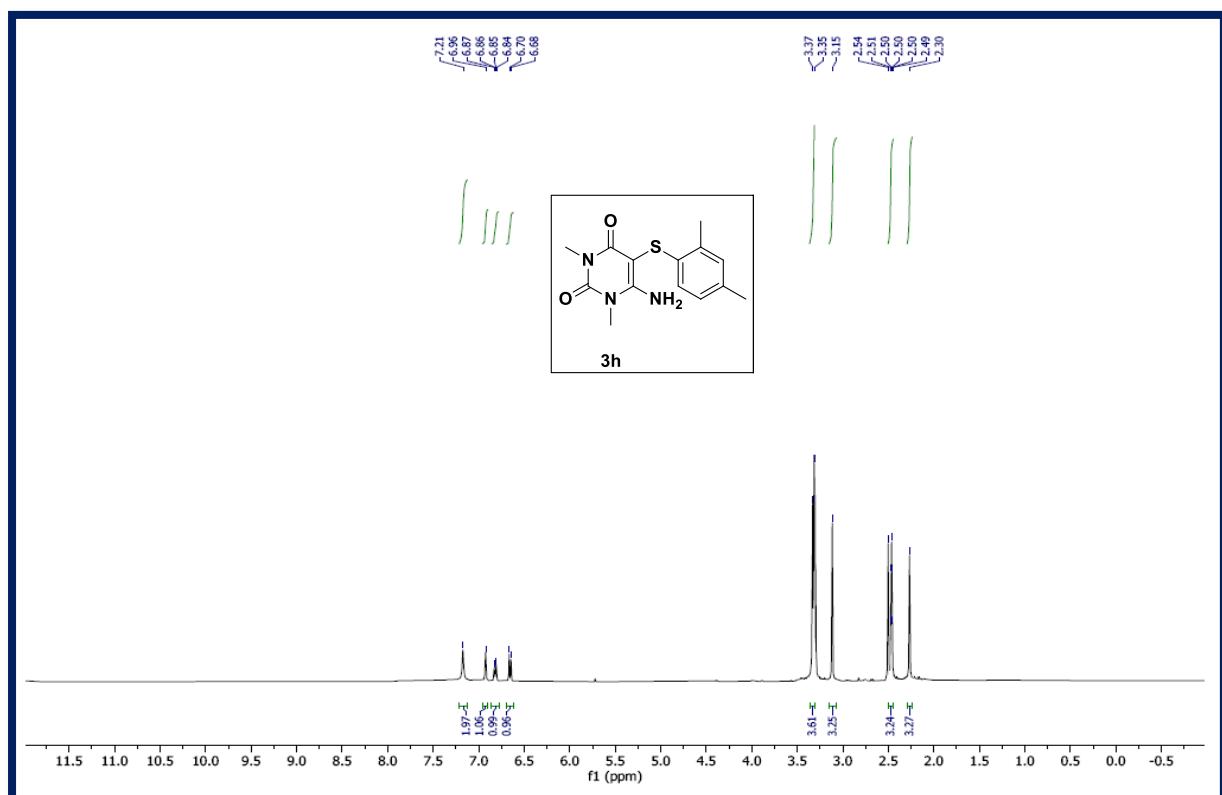
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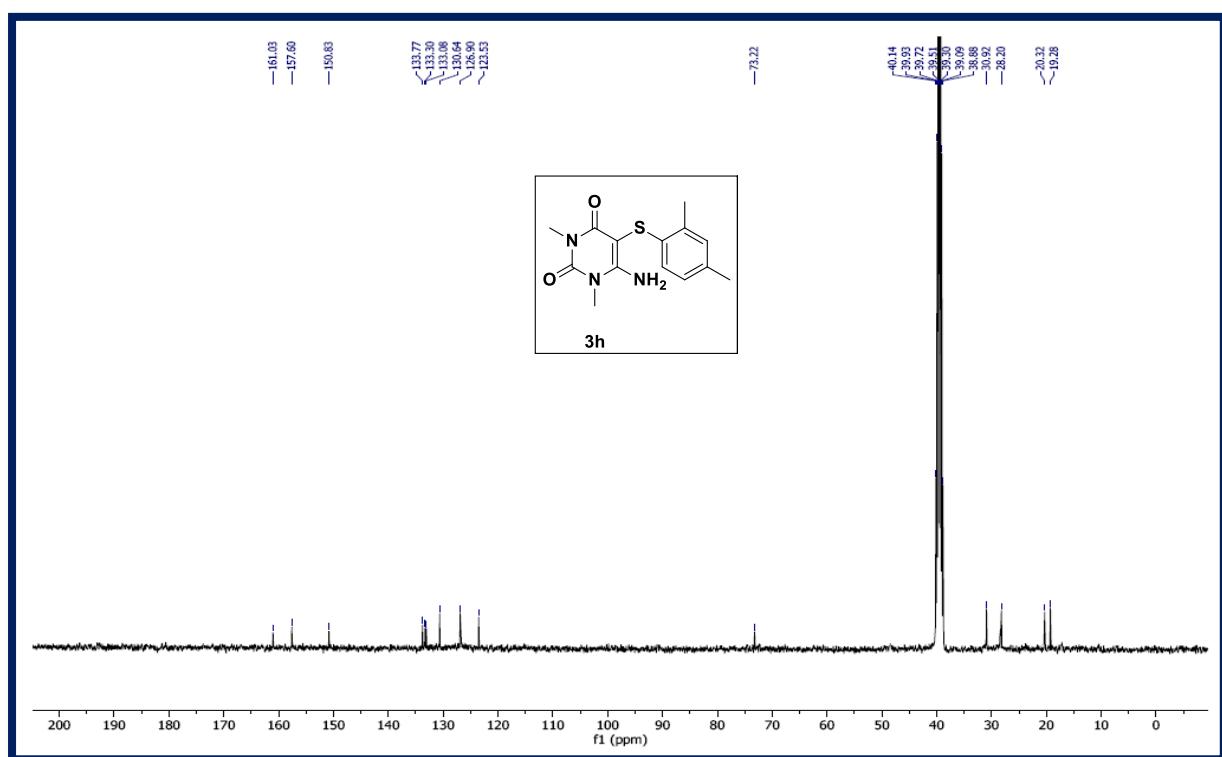
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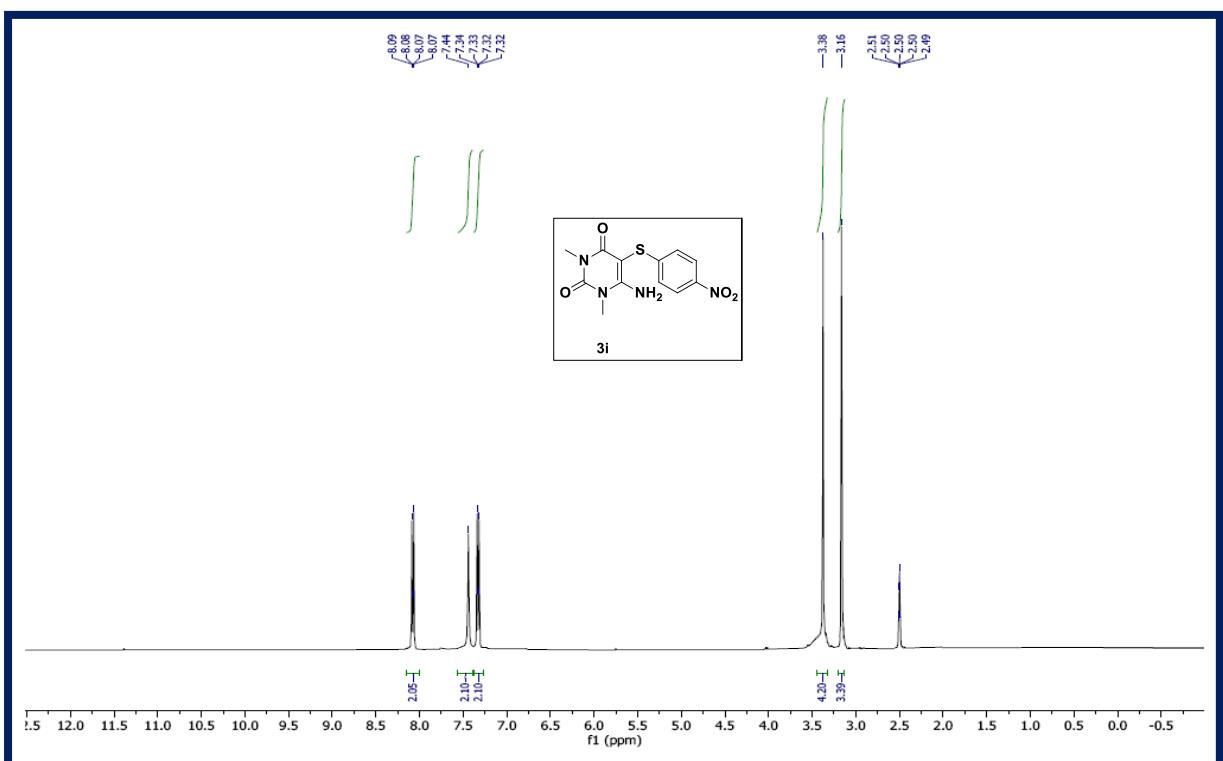
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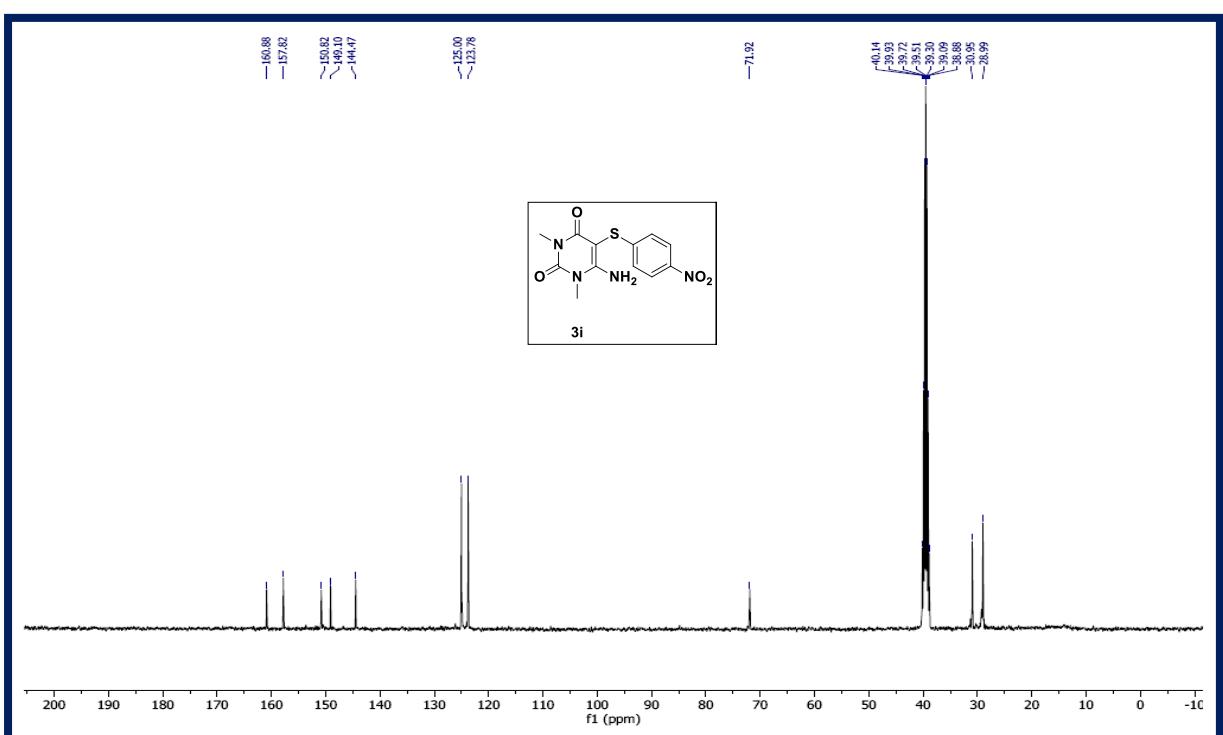
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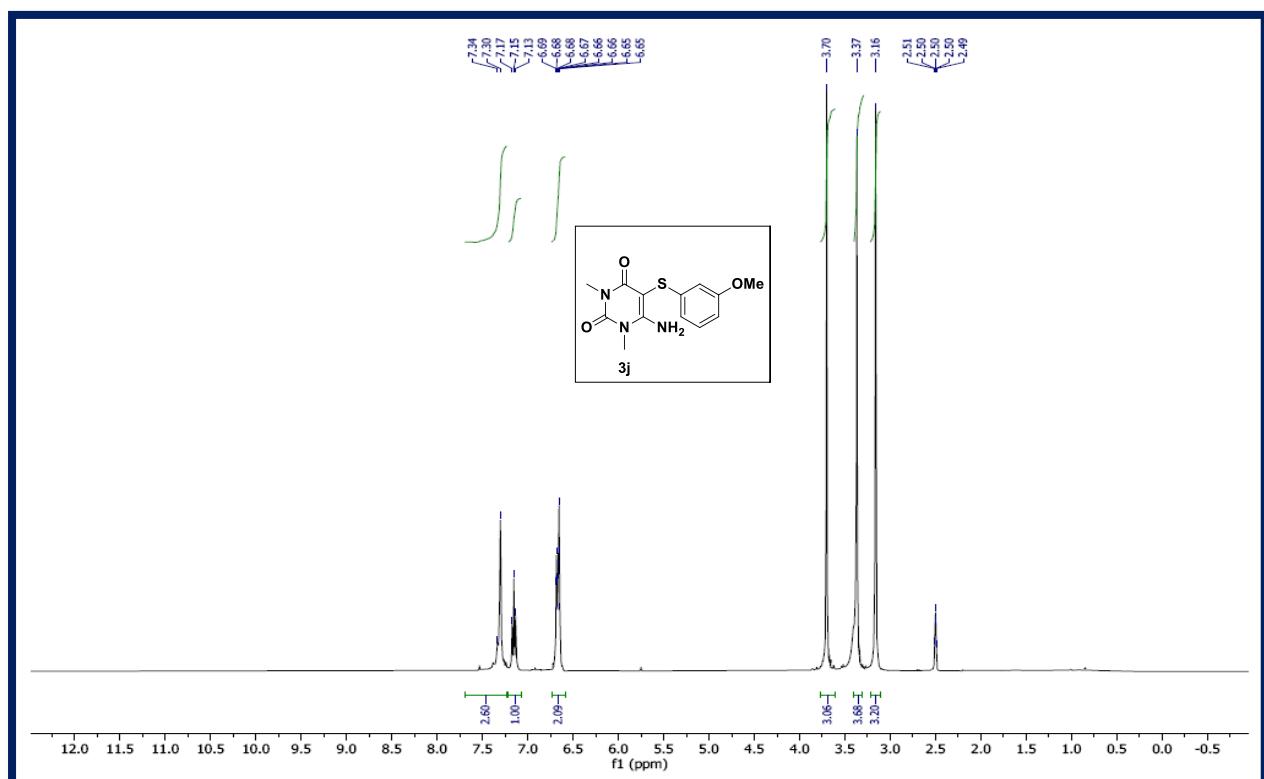
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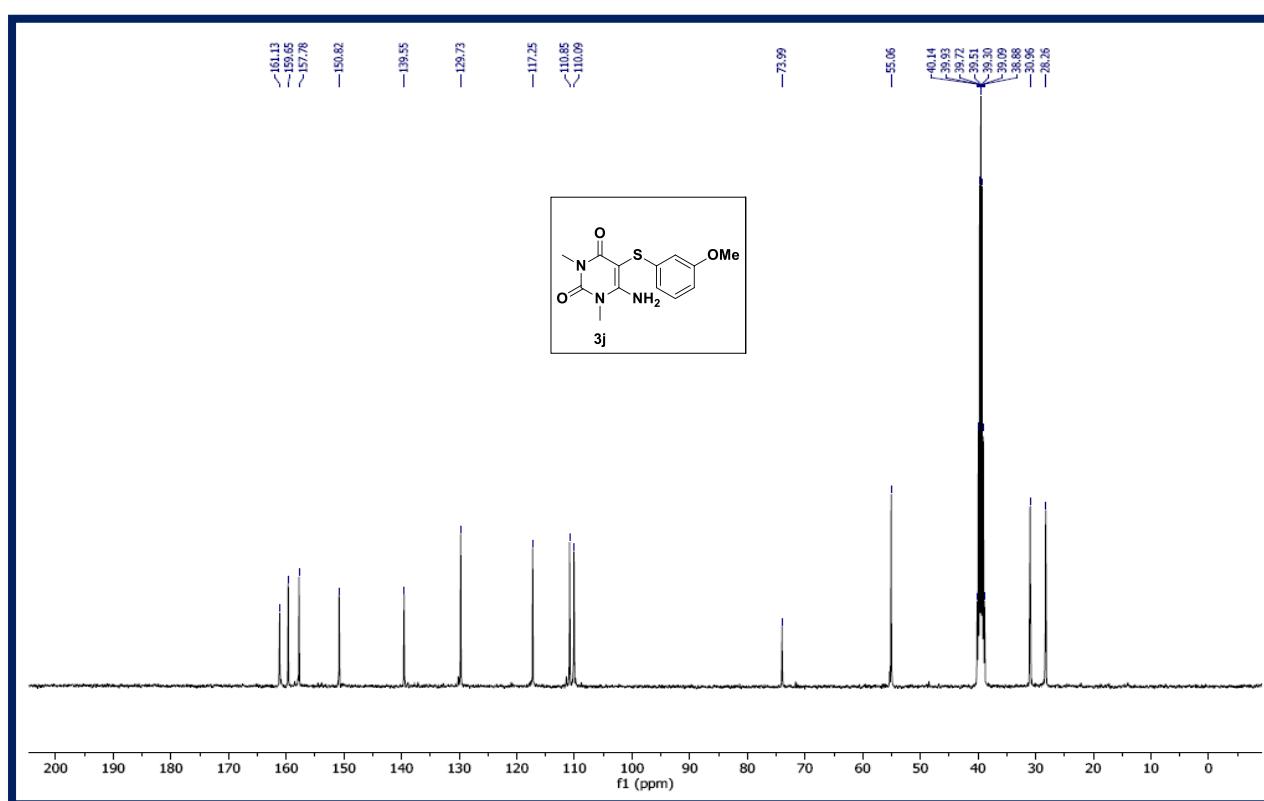
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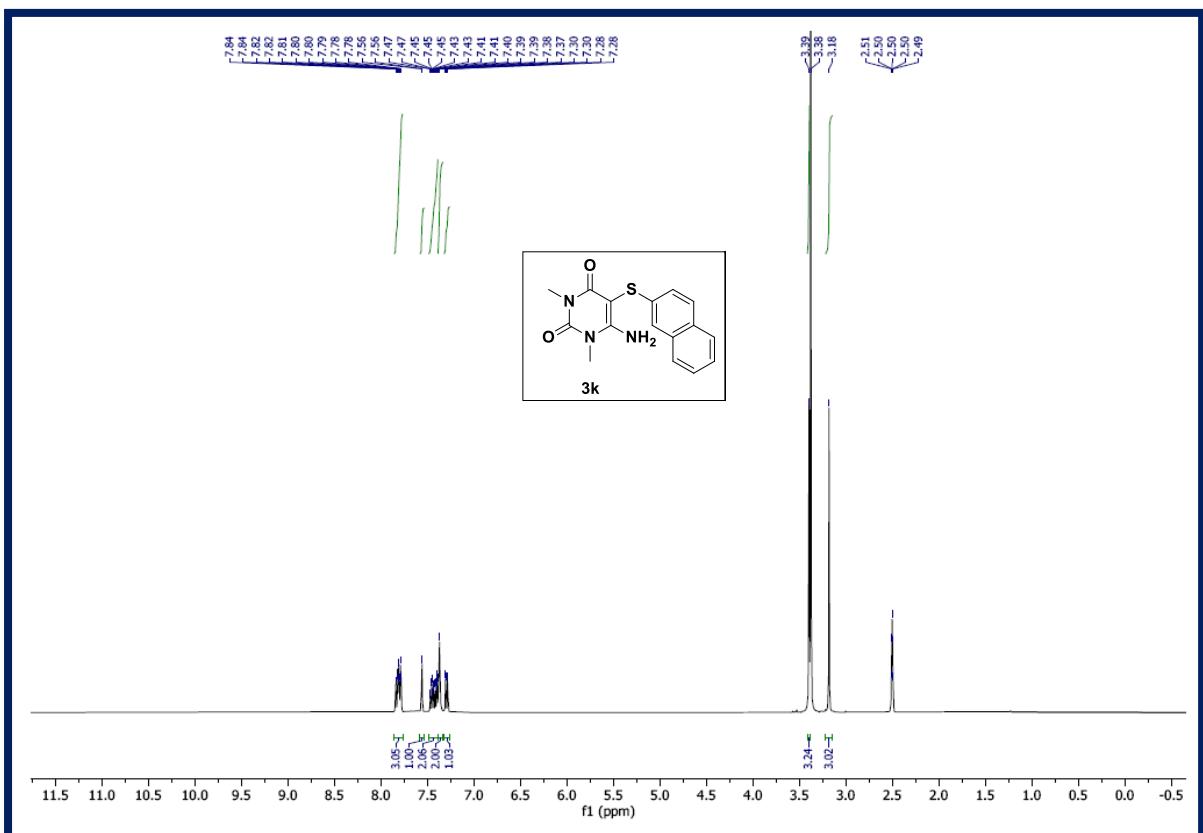
¹H NMR 3j



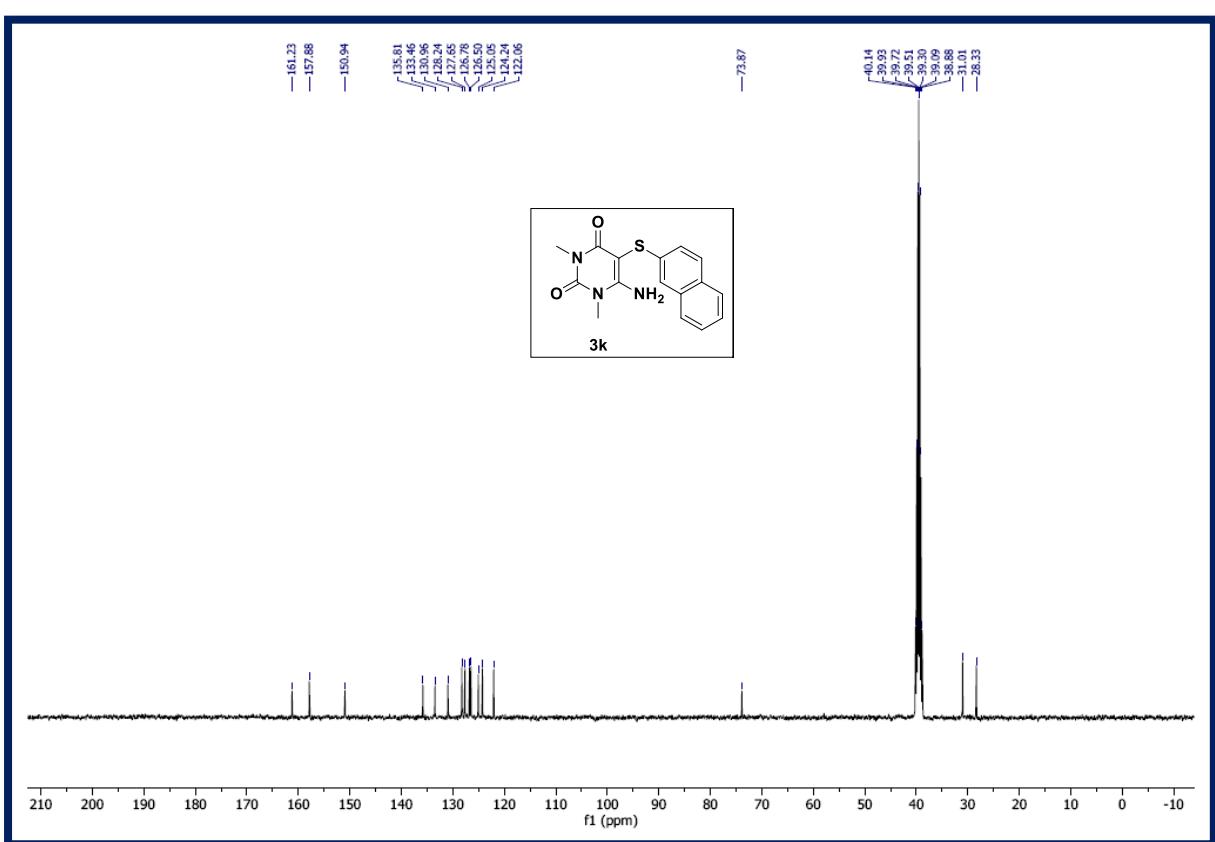
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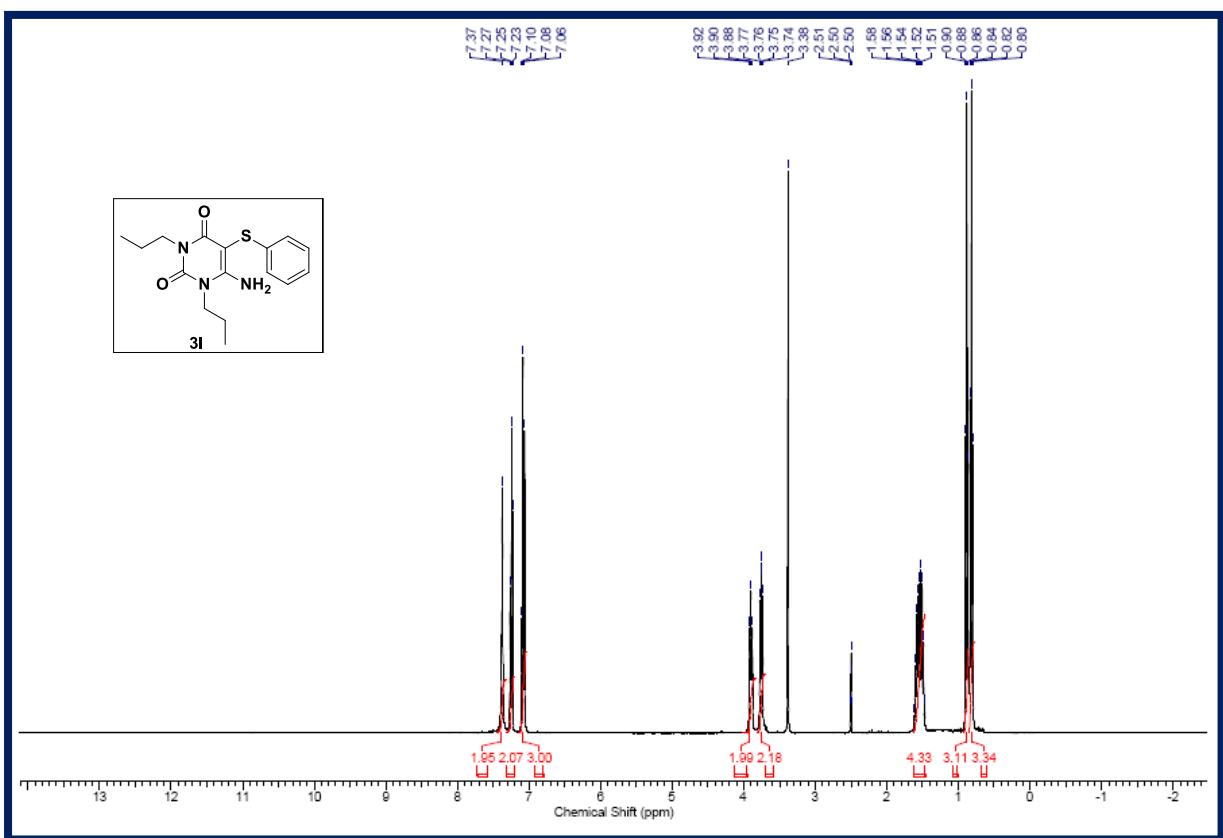
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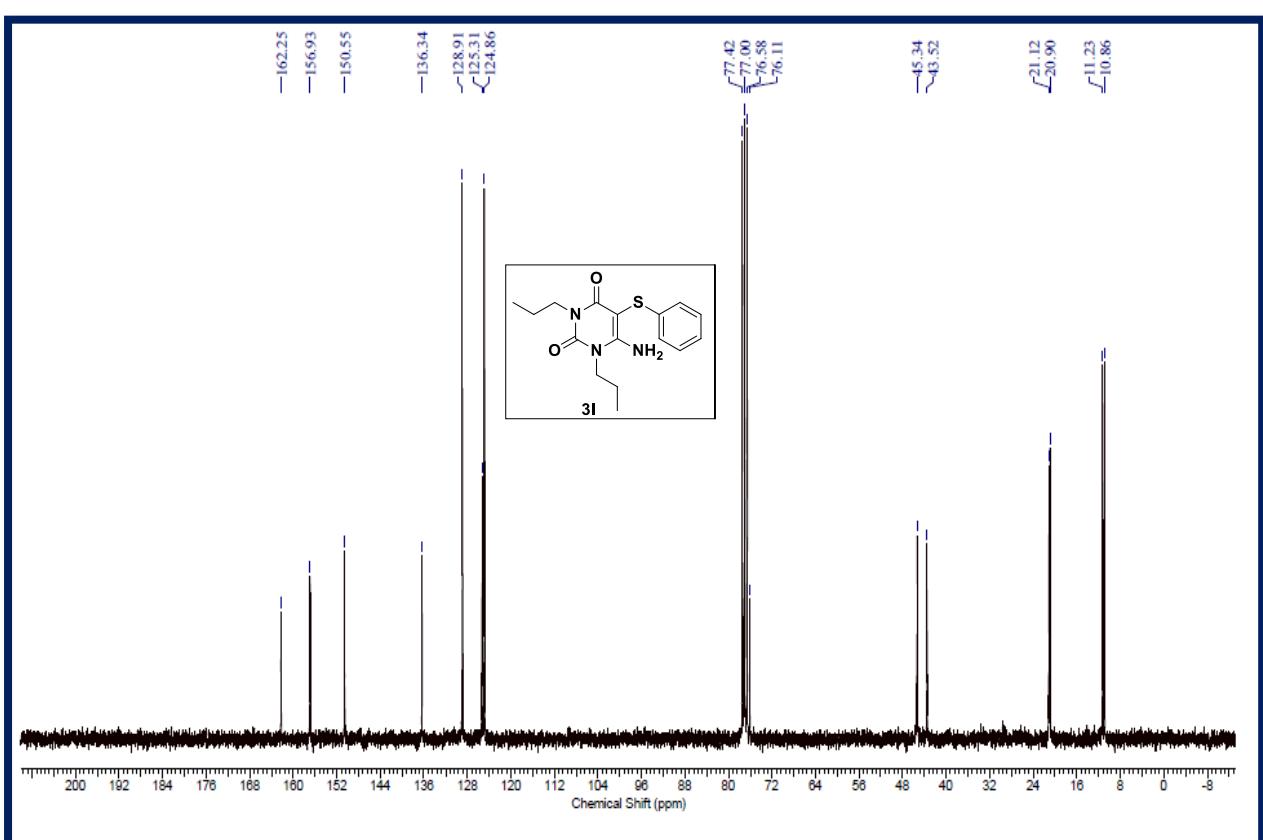
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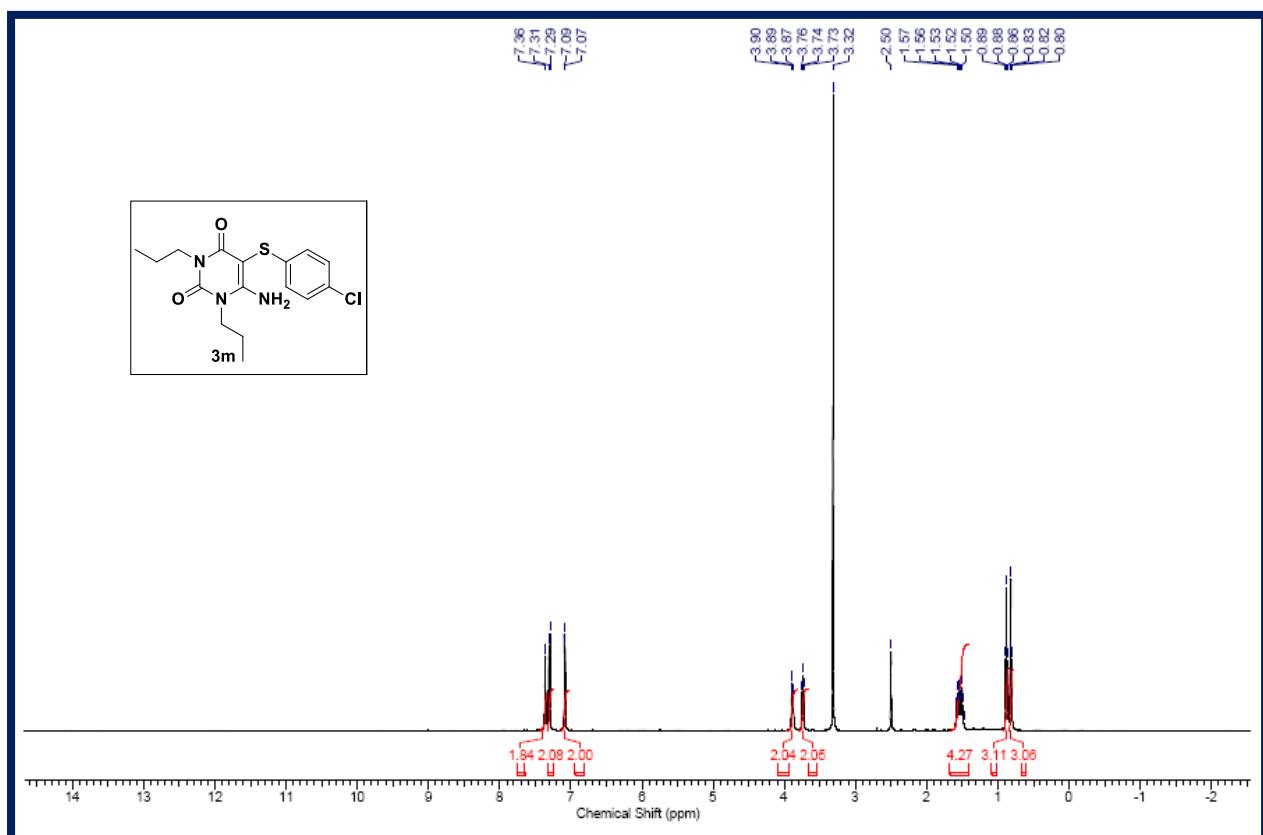
¹H NMR 3l



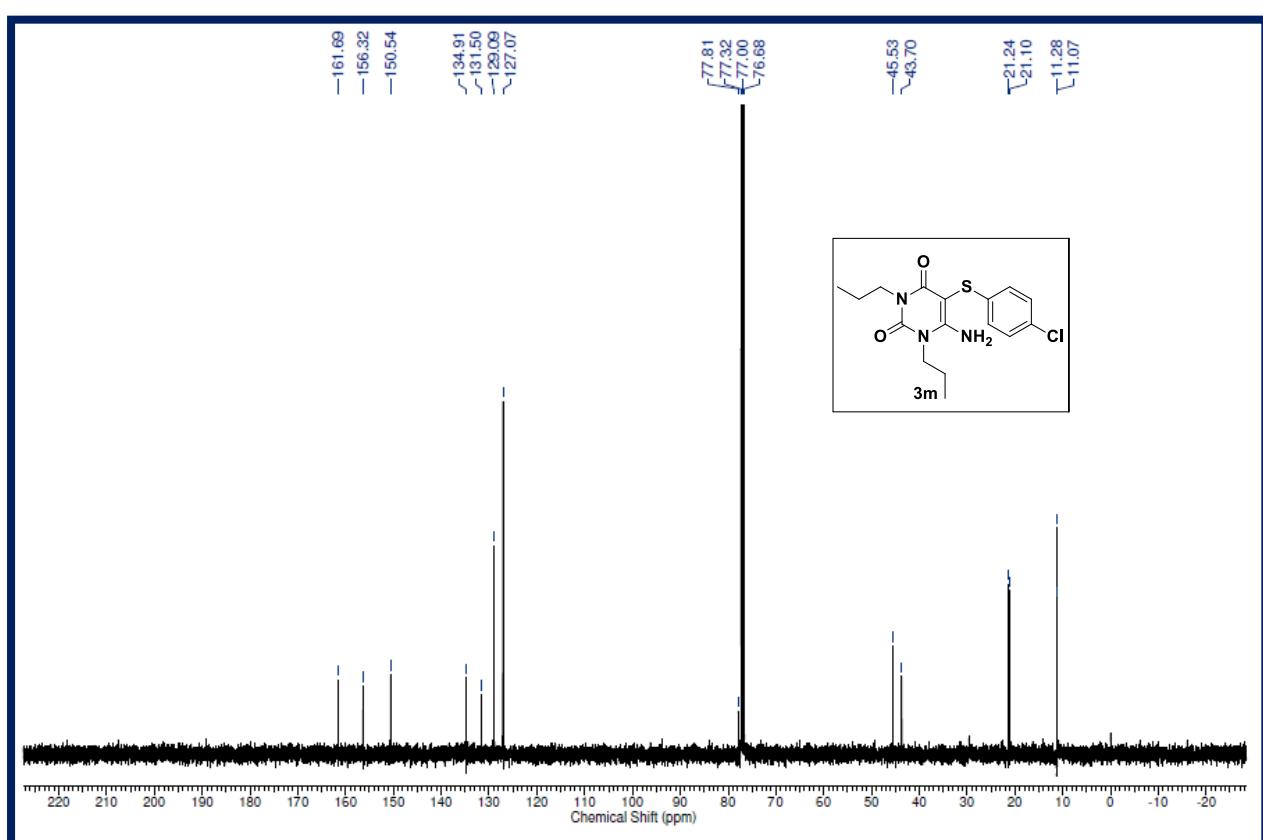
¹³C NMR 3l



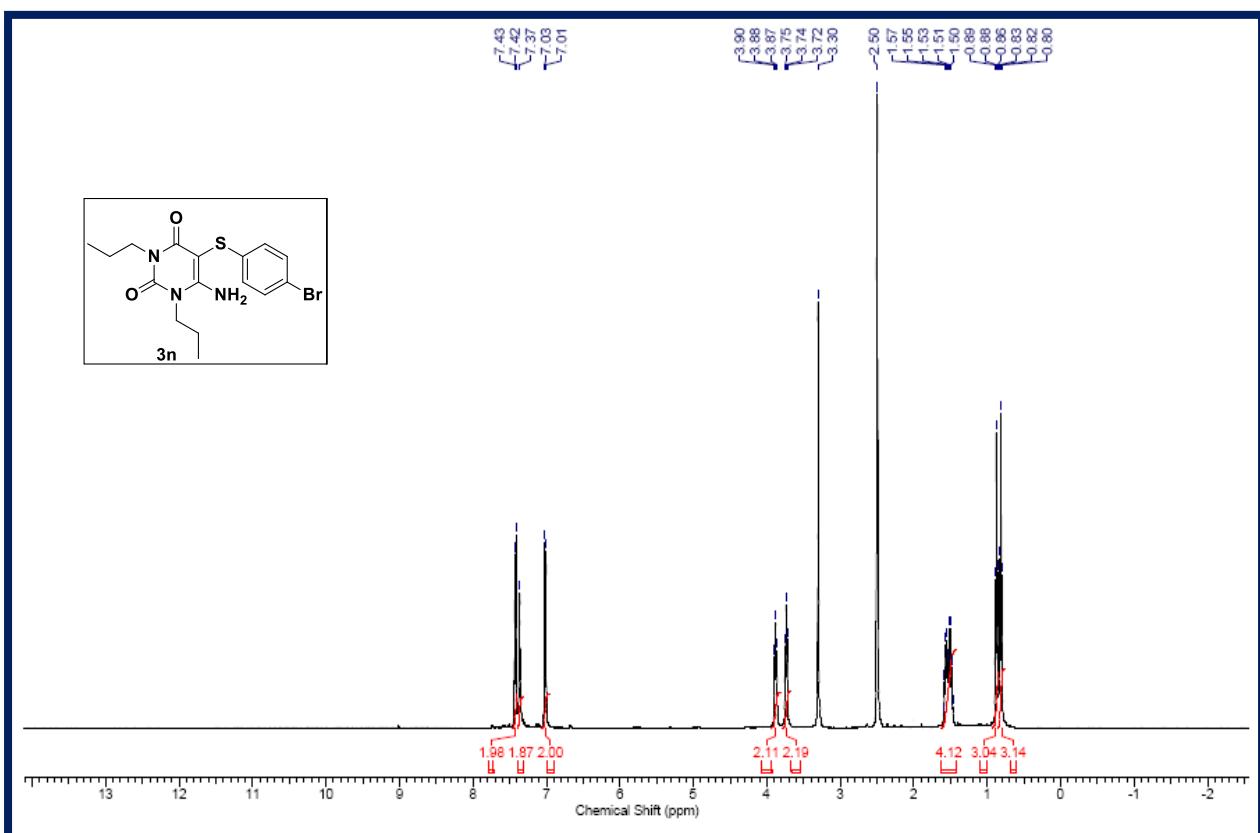
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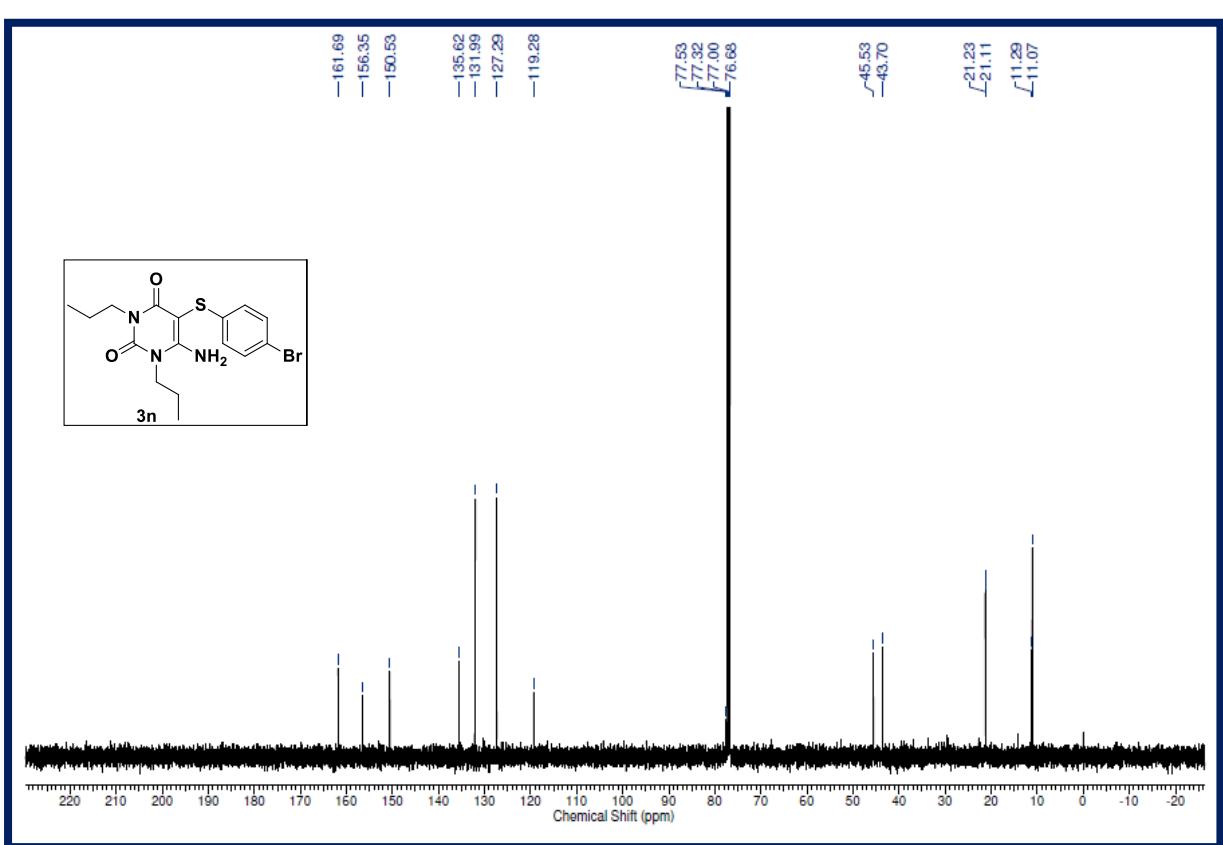
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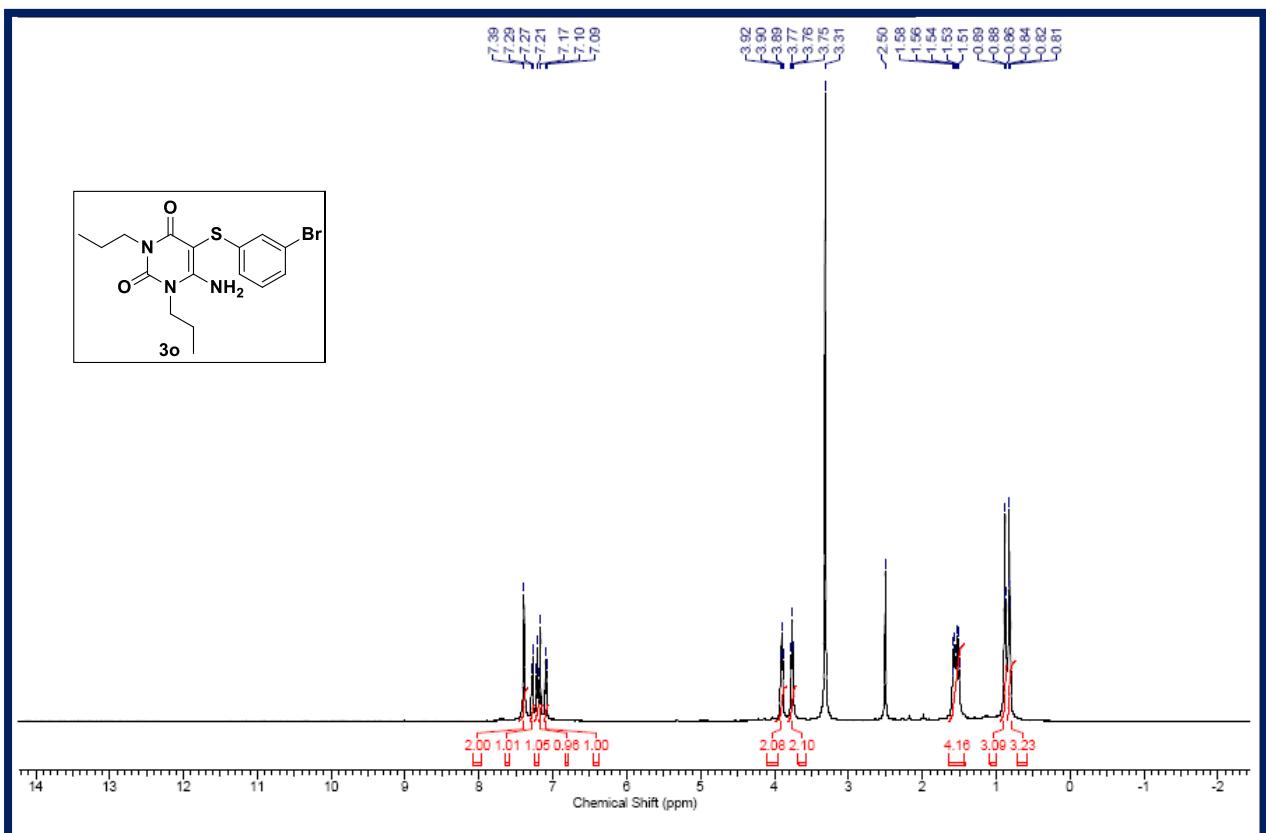
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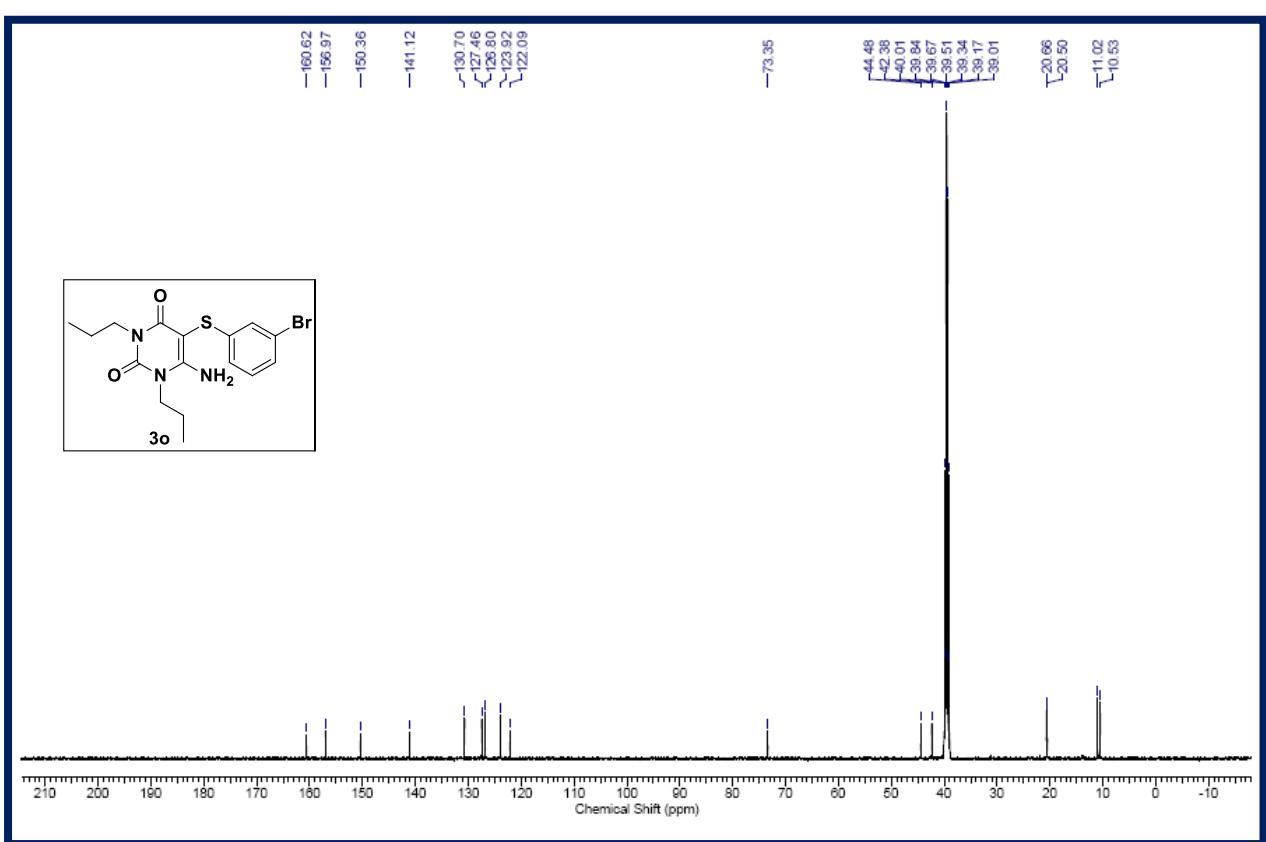
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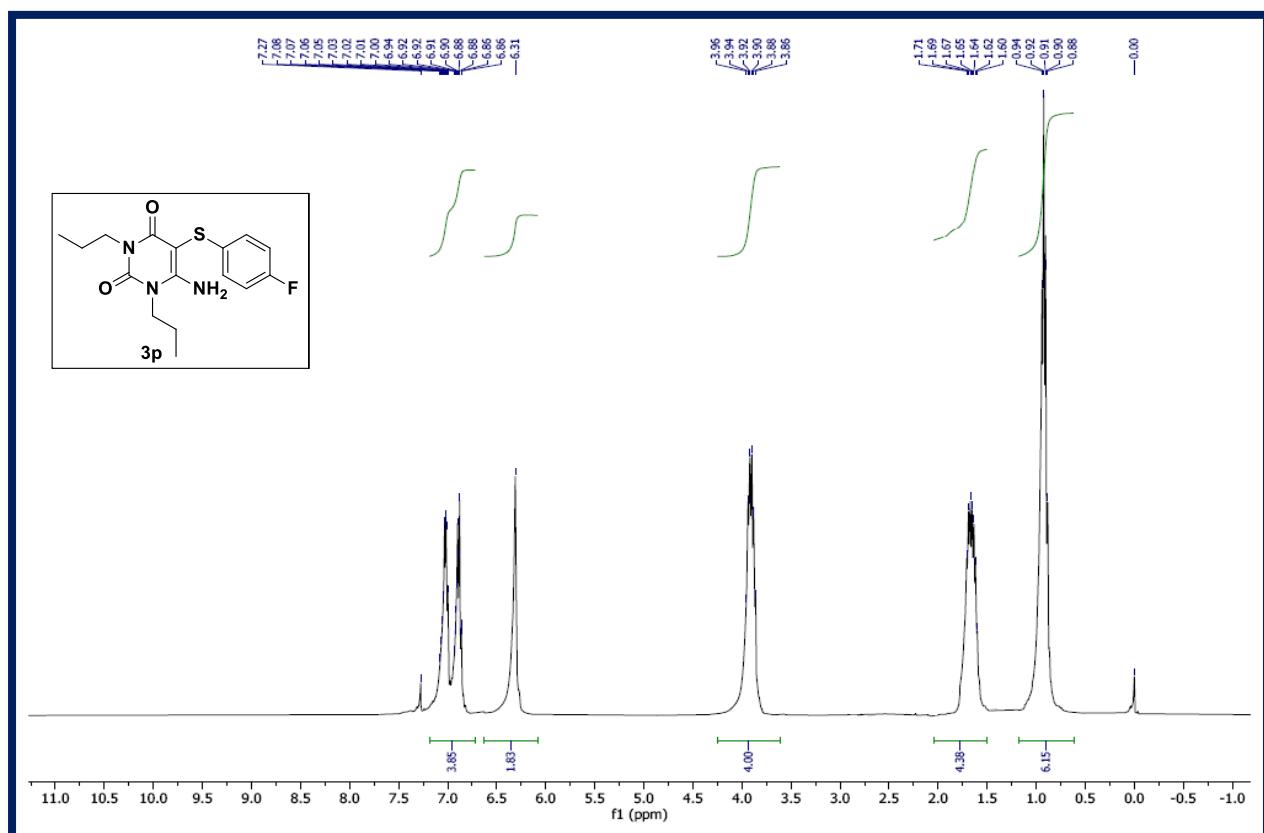
¹H NMR 3o



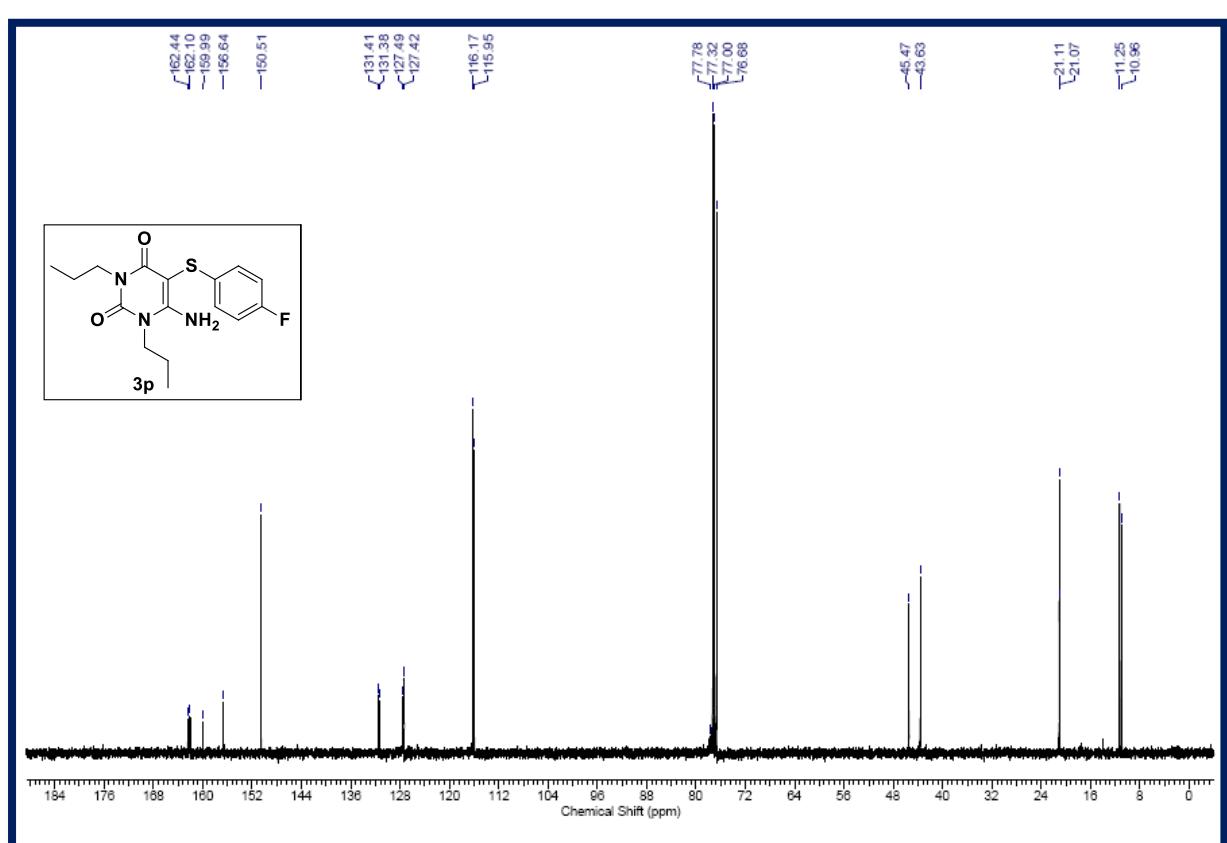
¹³C NMR 3o



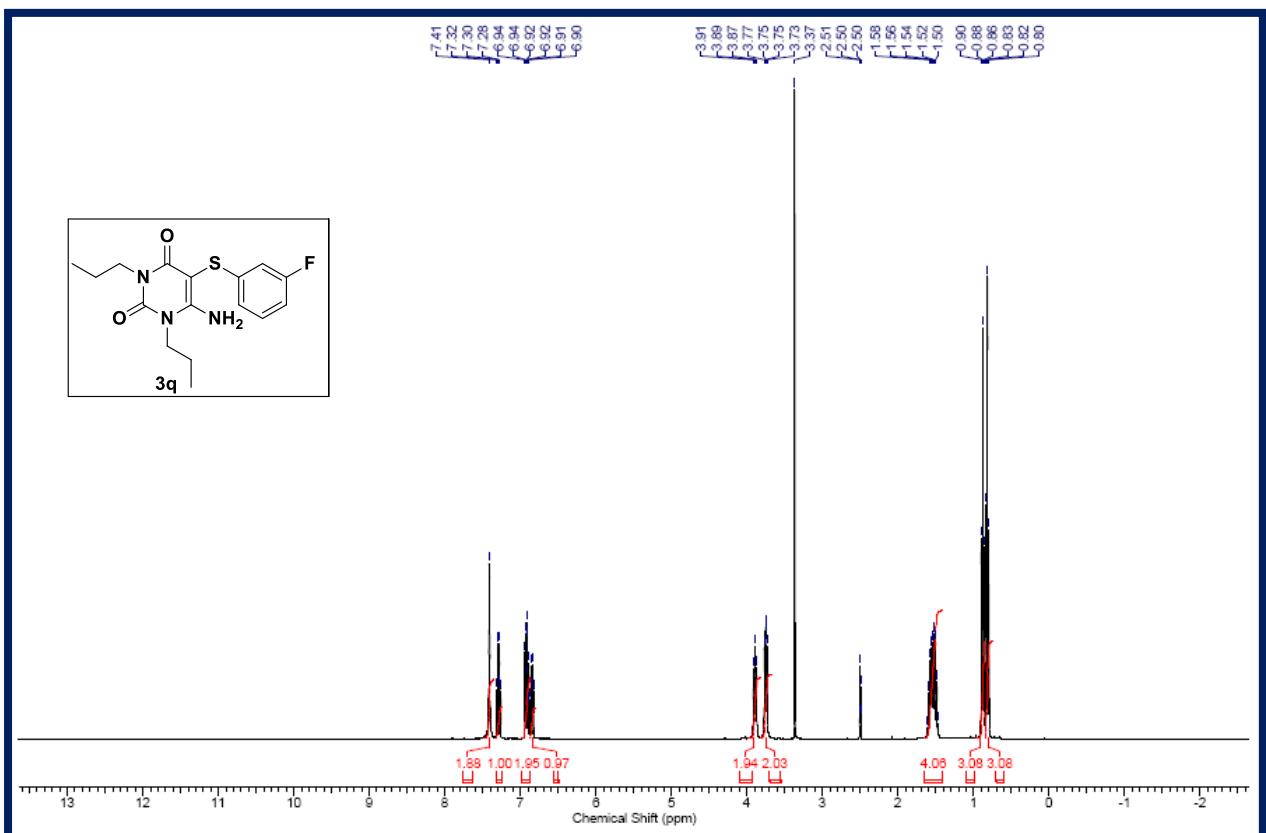
¹H NMR 3p



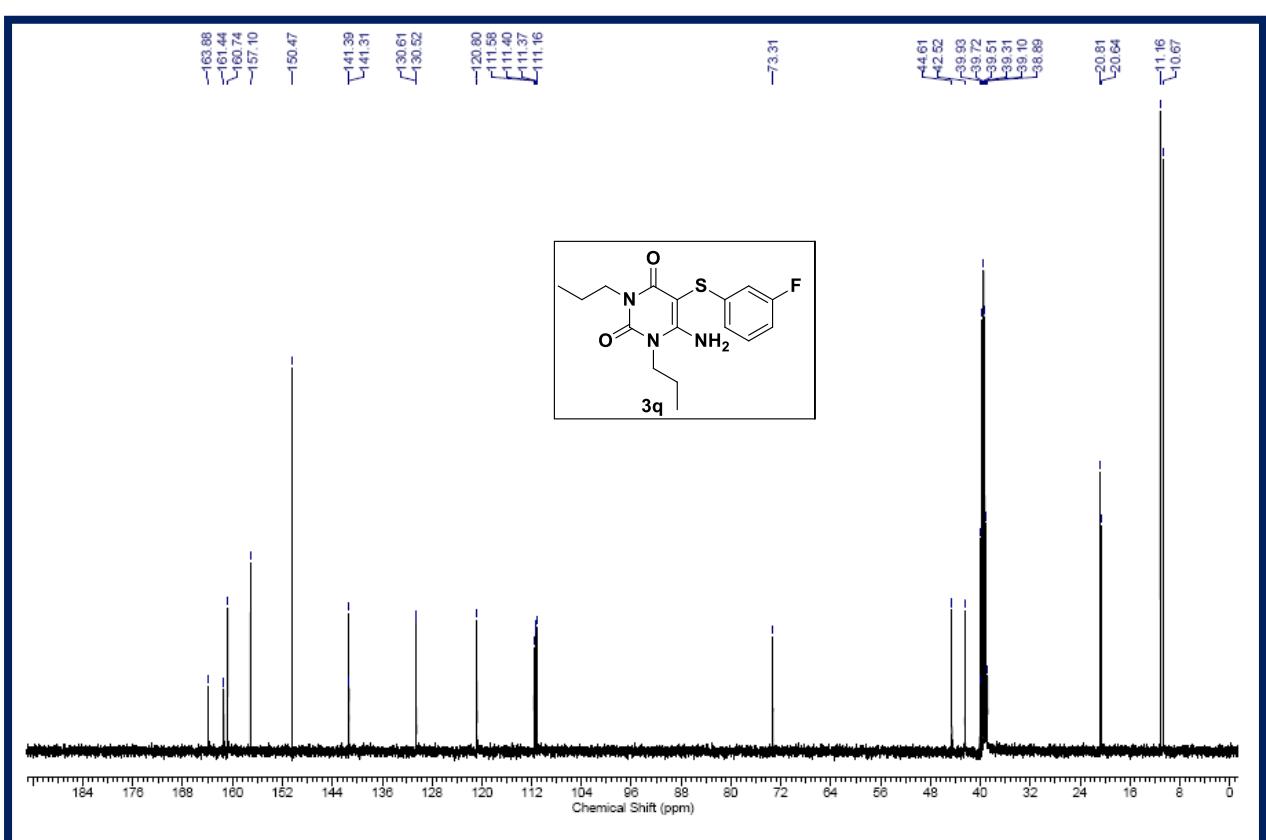
¹³C NMR 3p



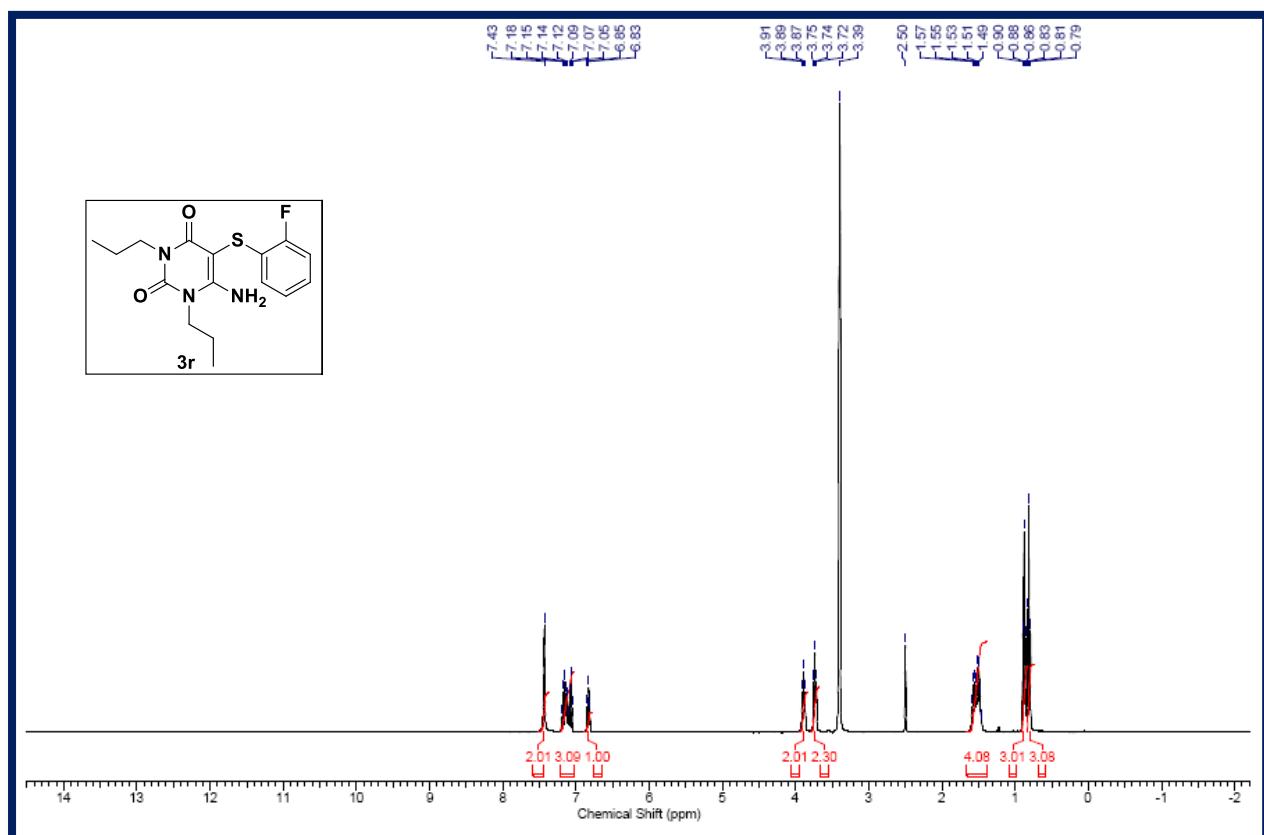
¹H NMR 3q



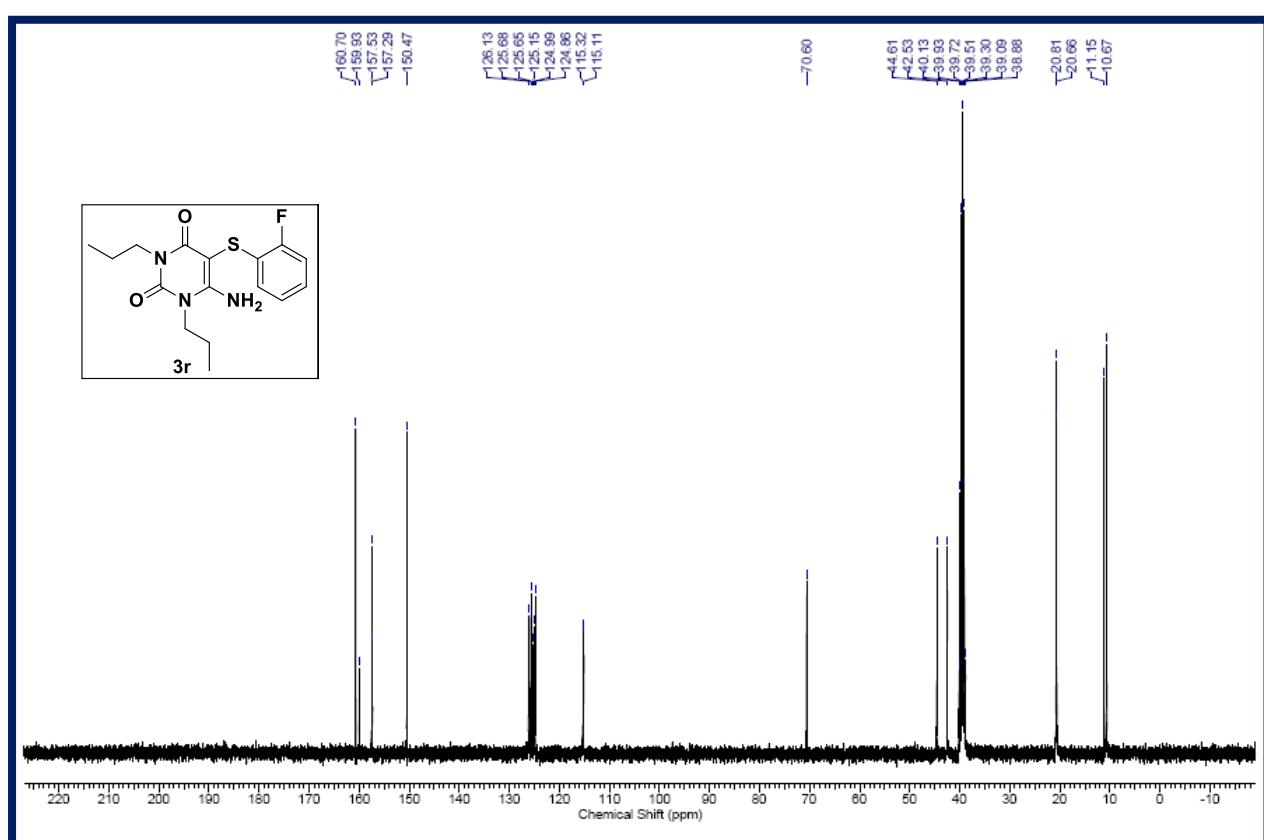
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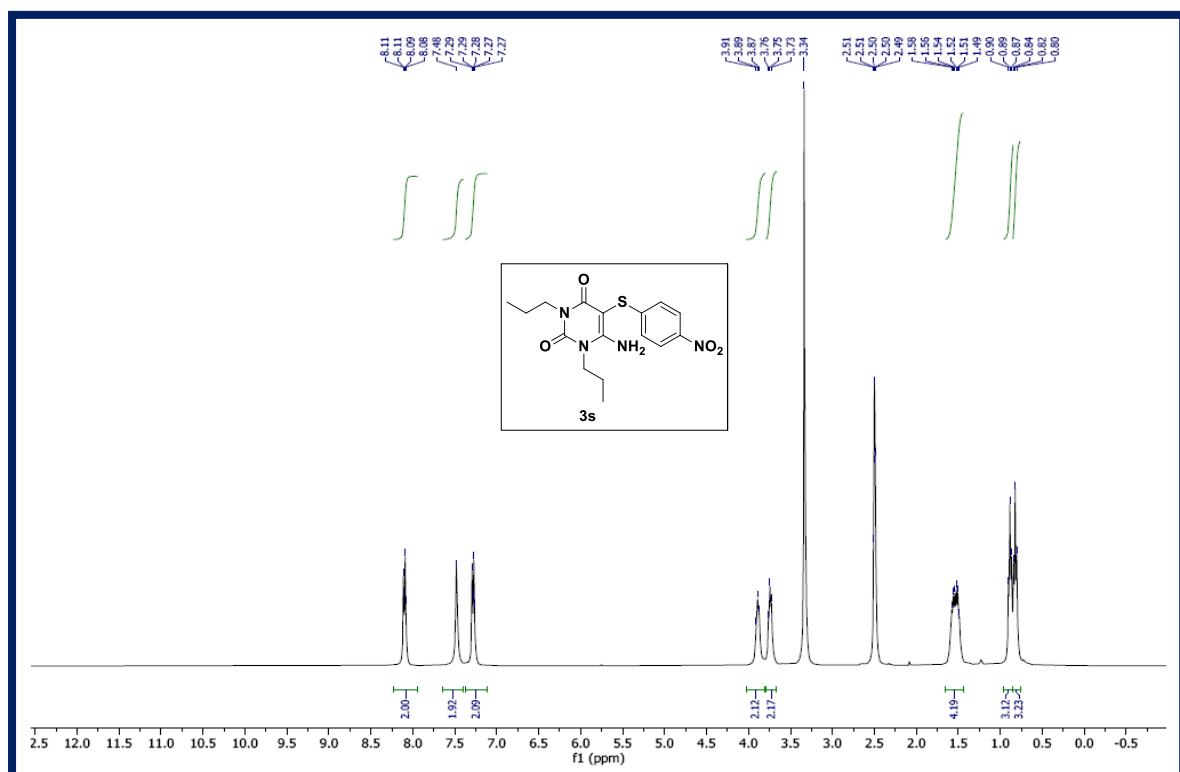
¹H NMR 3r



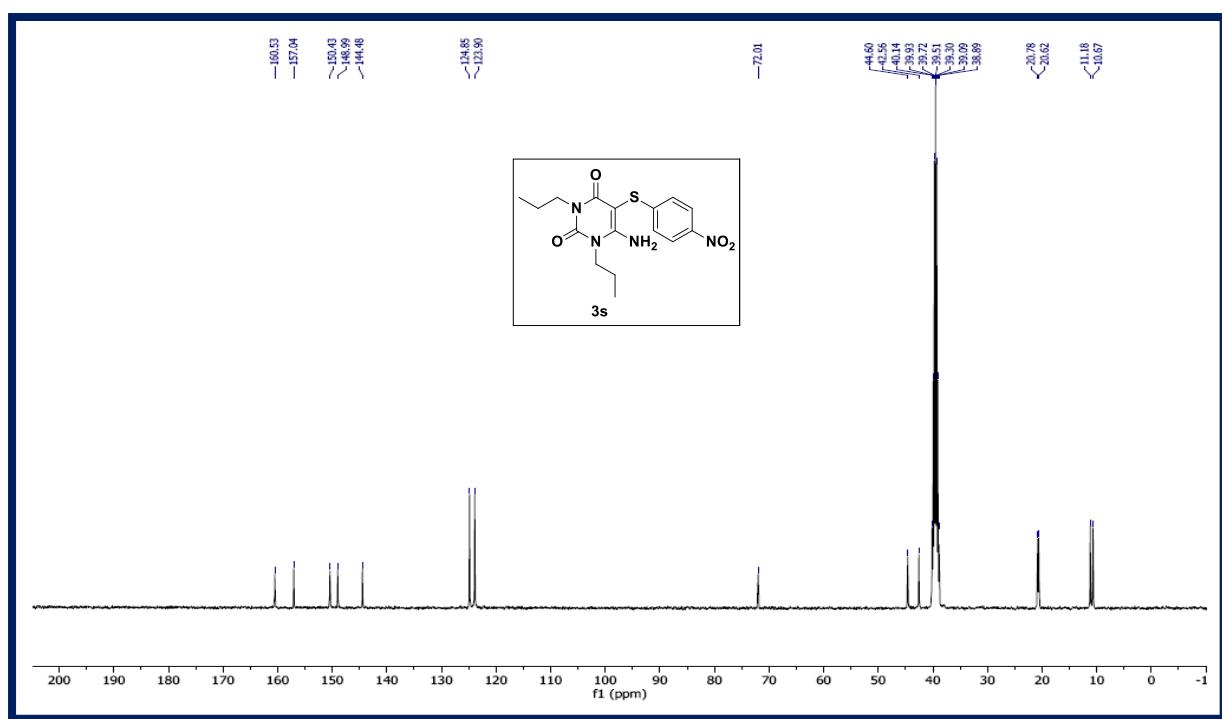
¹³C NMR 3r



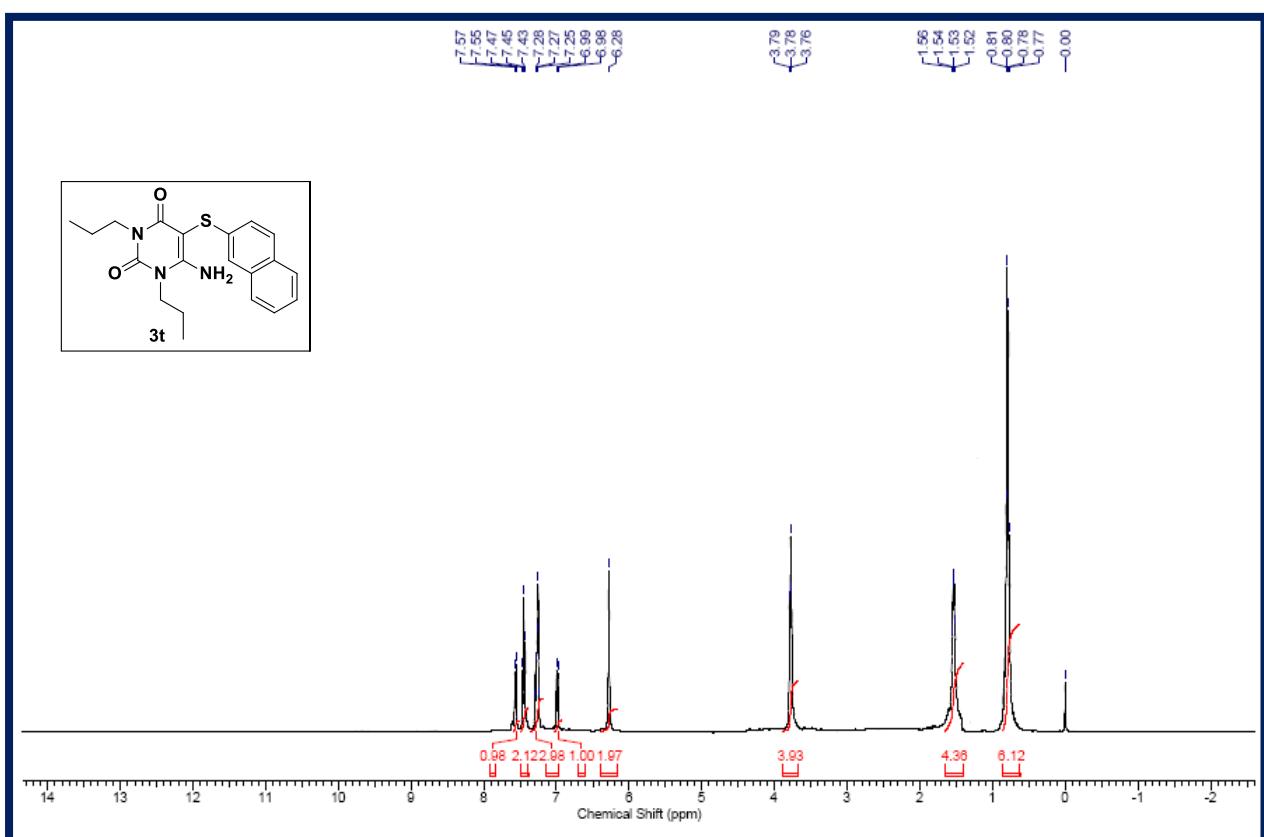
¹H NMR 3s



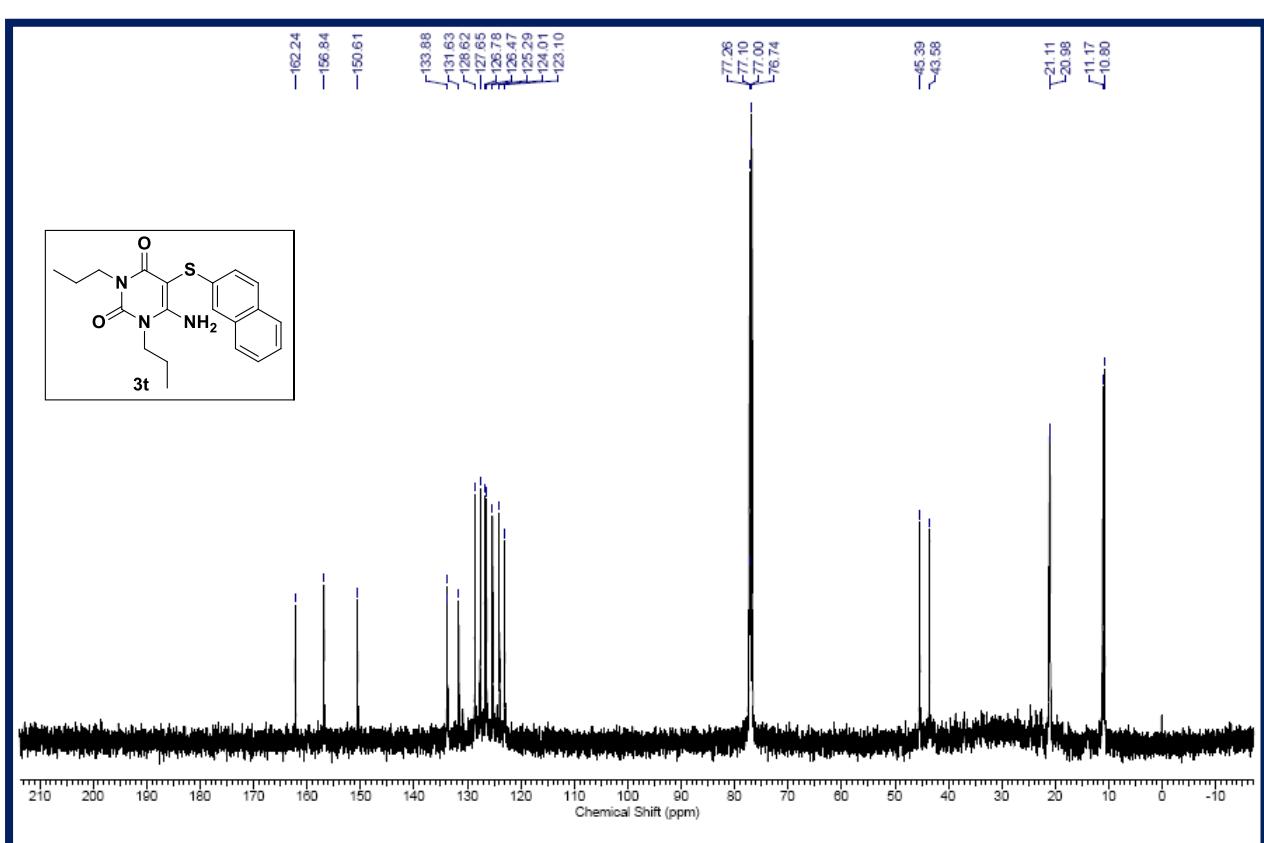
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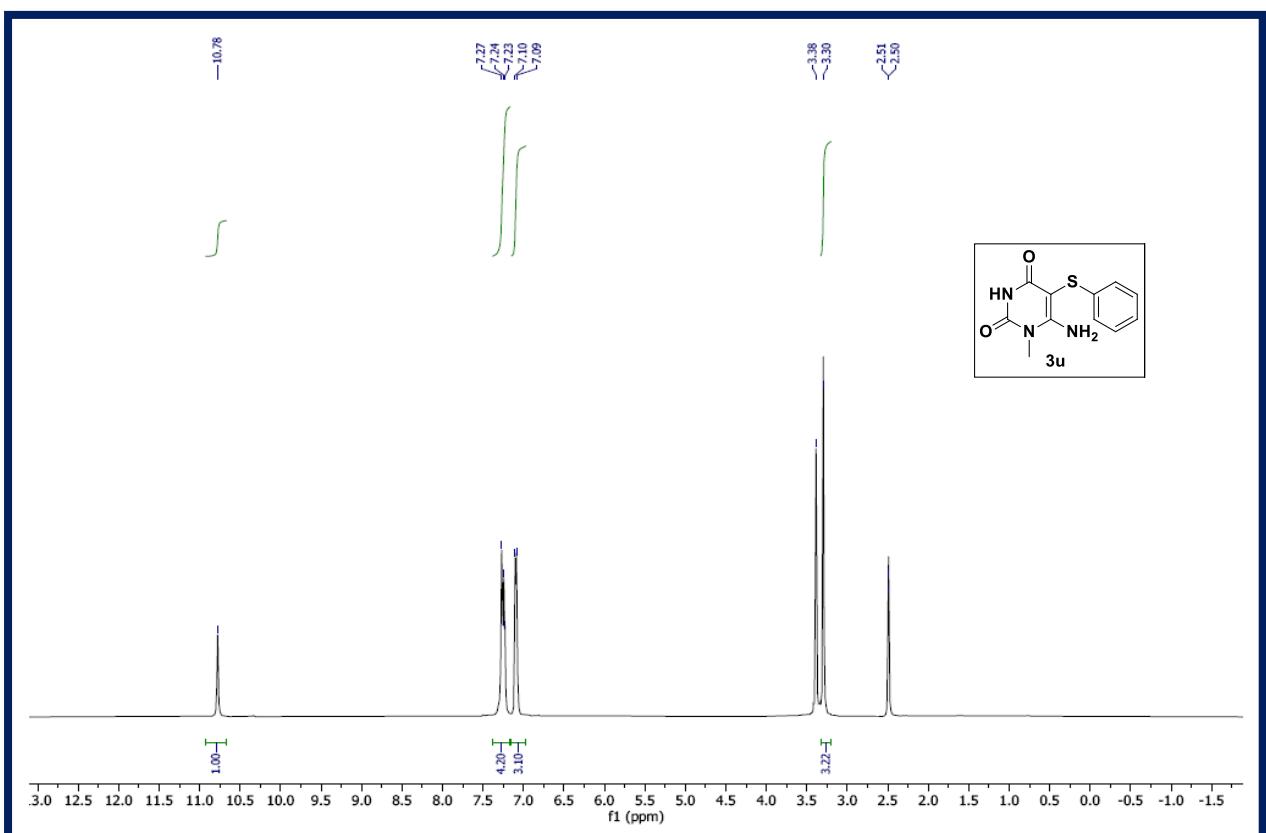
¹H NMR 3t



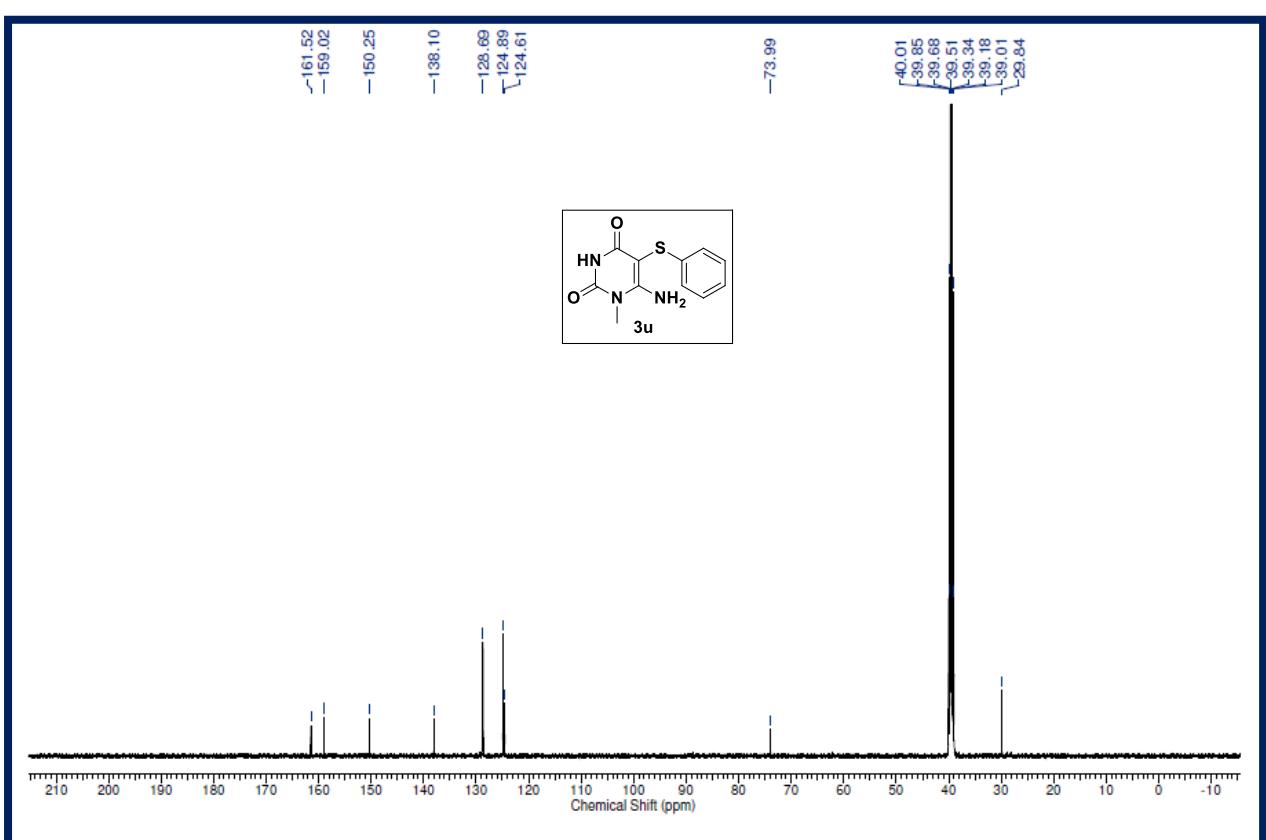
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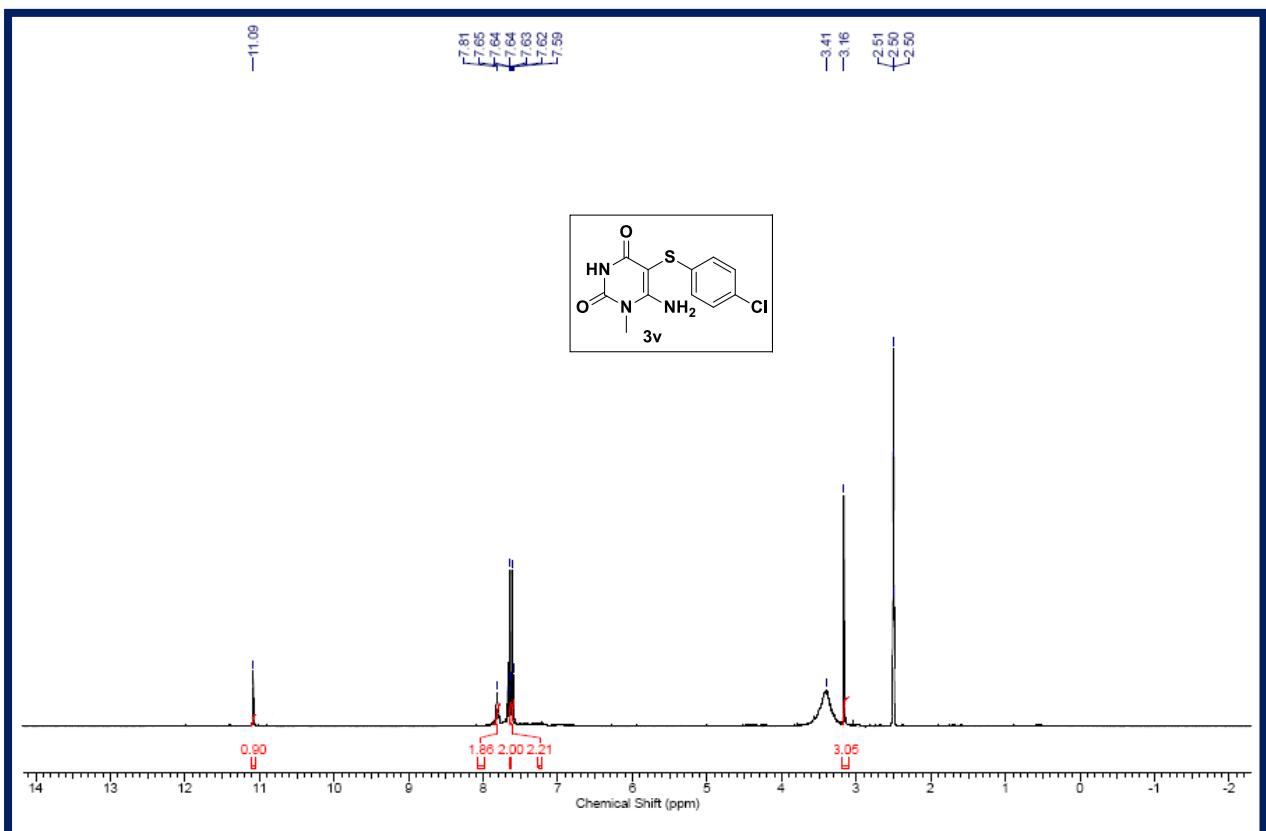
¹H NMR 3u



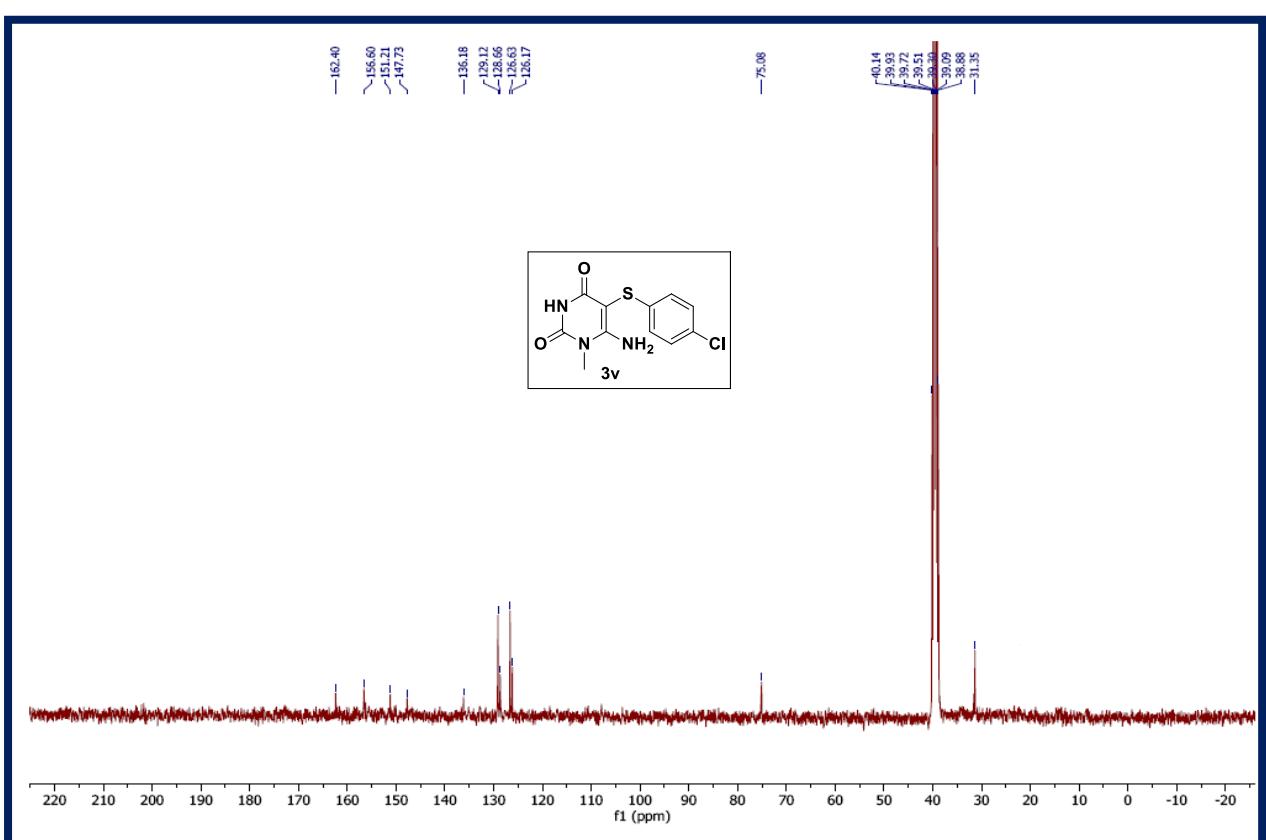
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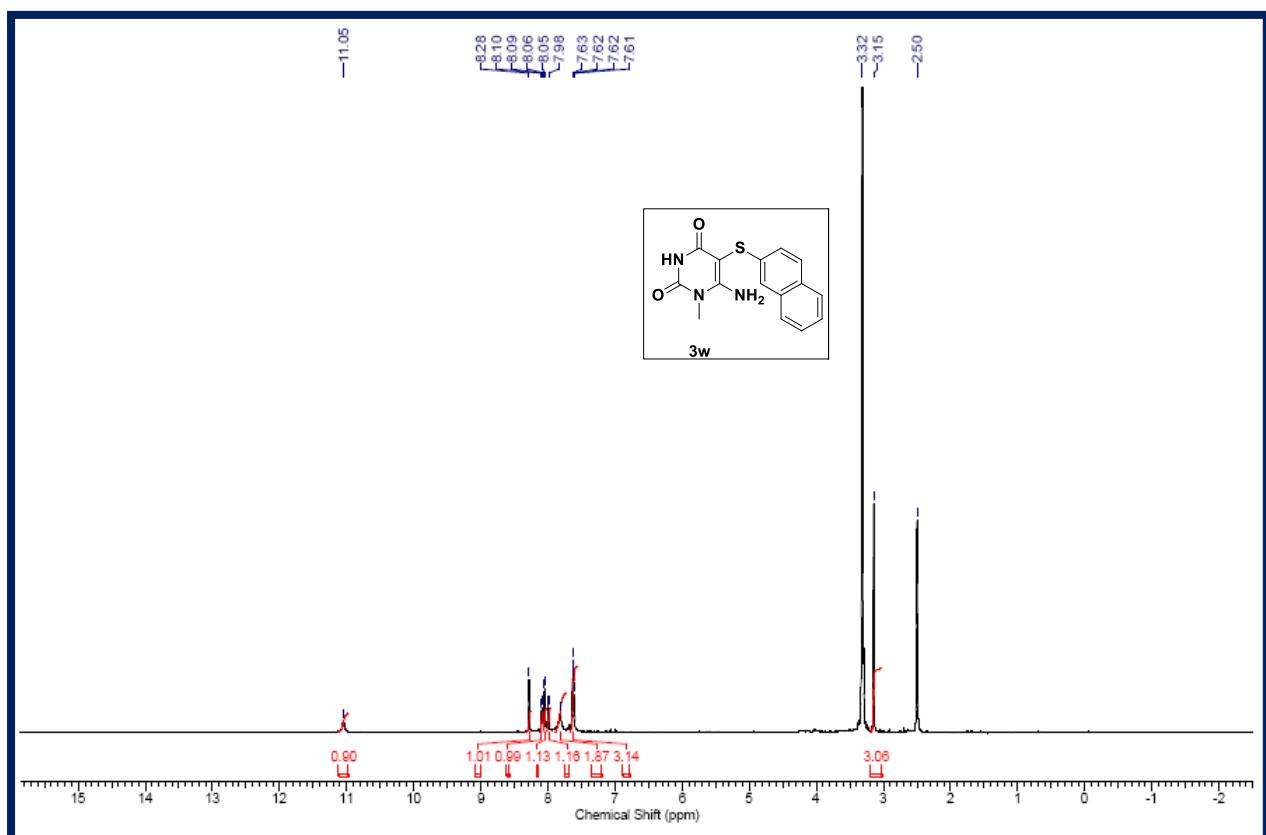
¹H NMR 3v



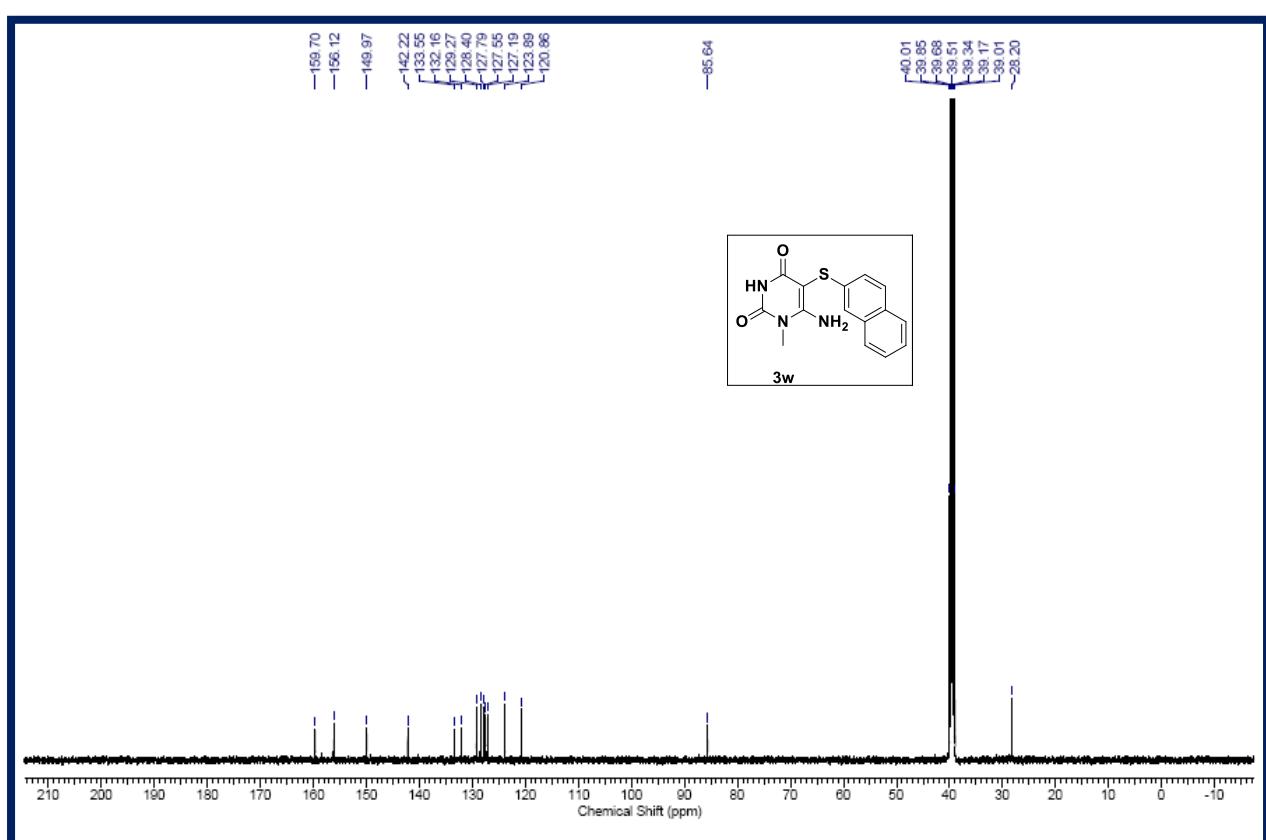
¹³C NMR 3v



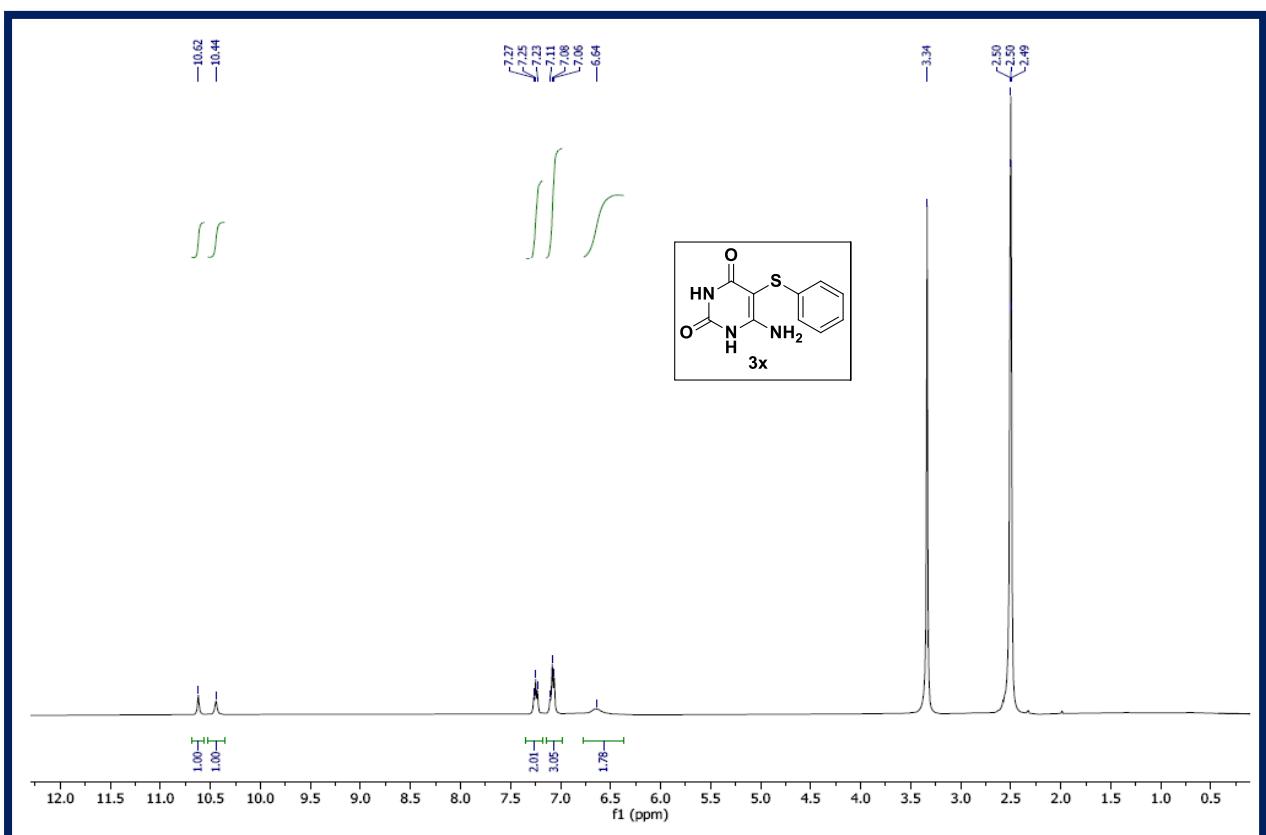
¹H NMR 3w



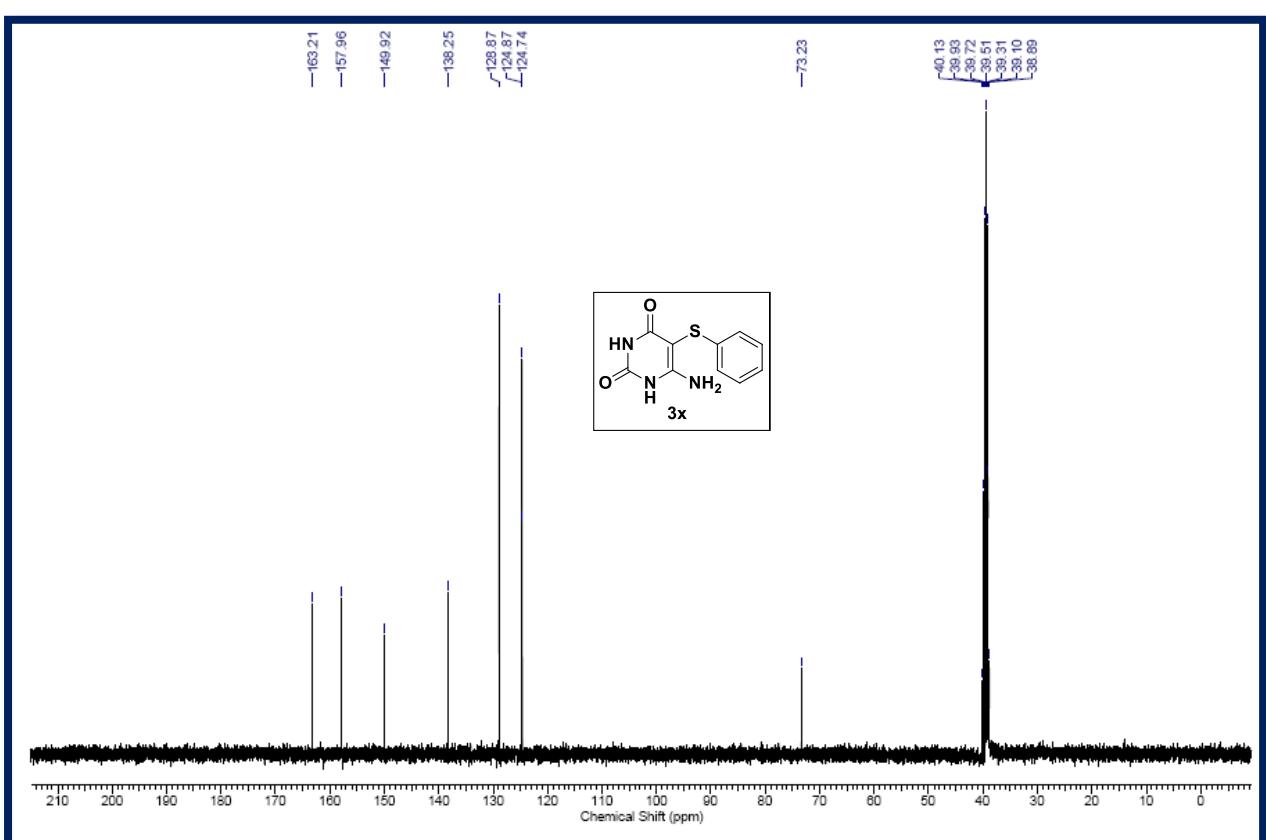
¹³C NMR 3w



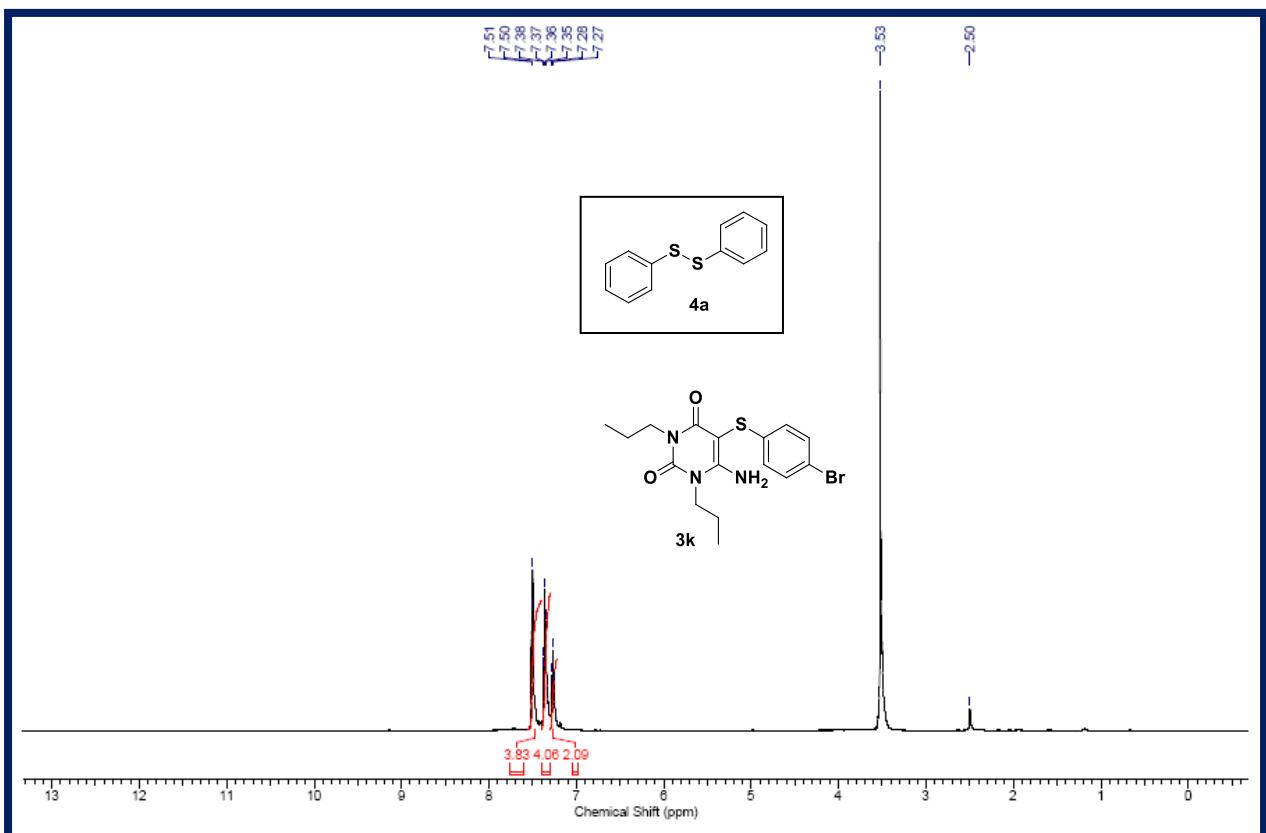
¹H NMR 3x



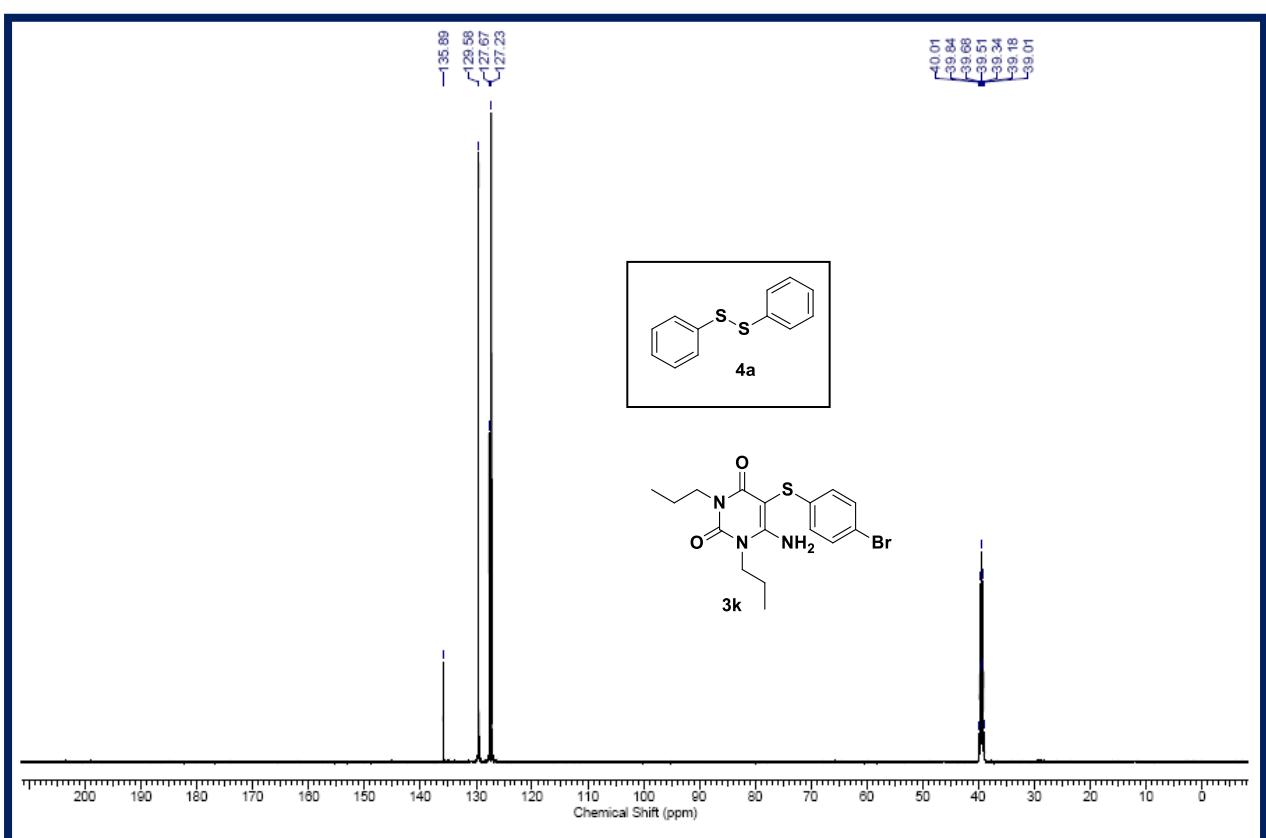
¹³C NMR 3x



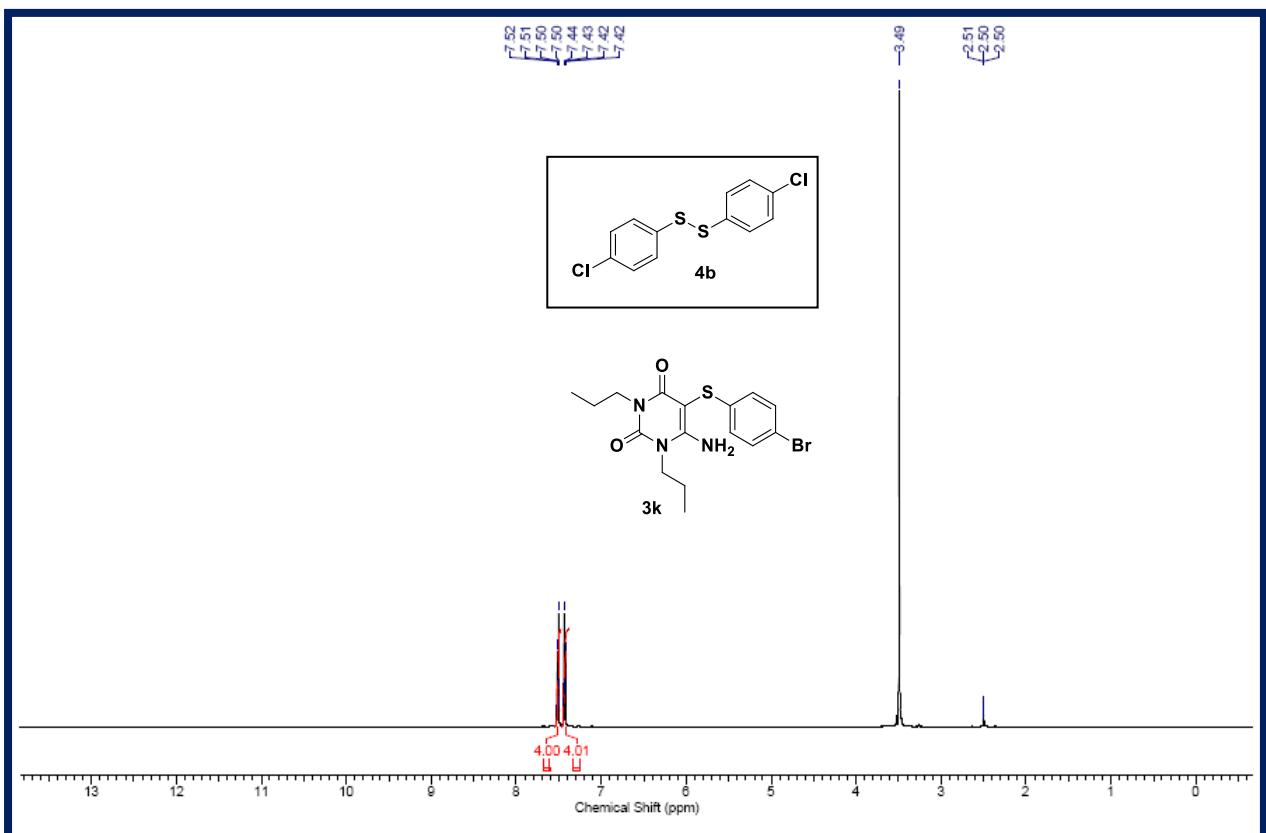
¹H NMR 4a



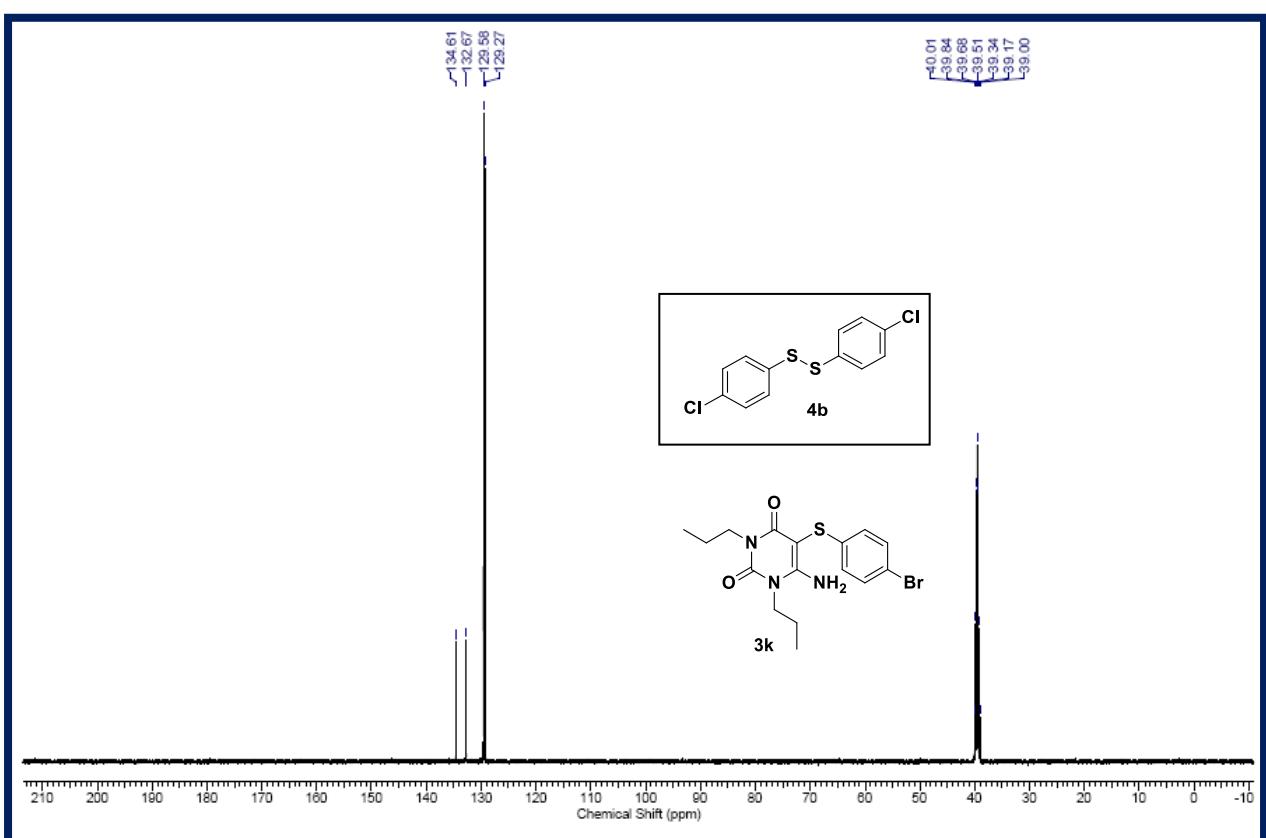
¹³C NMR 4a



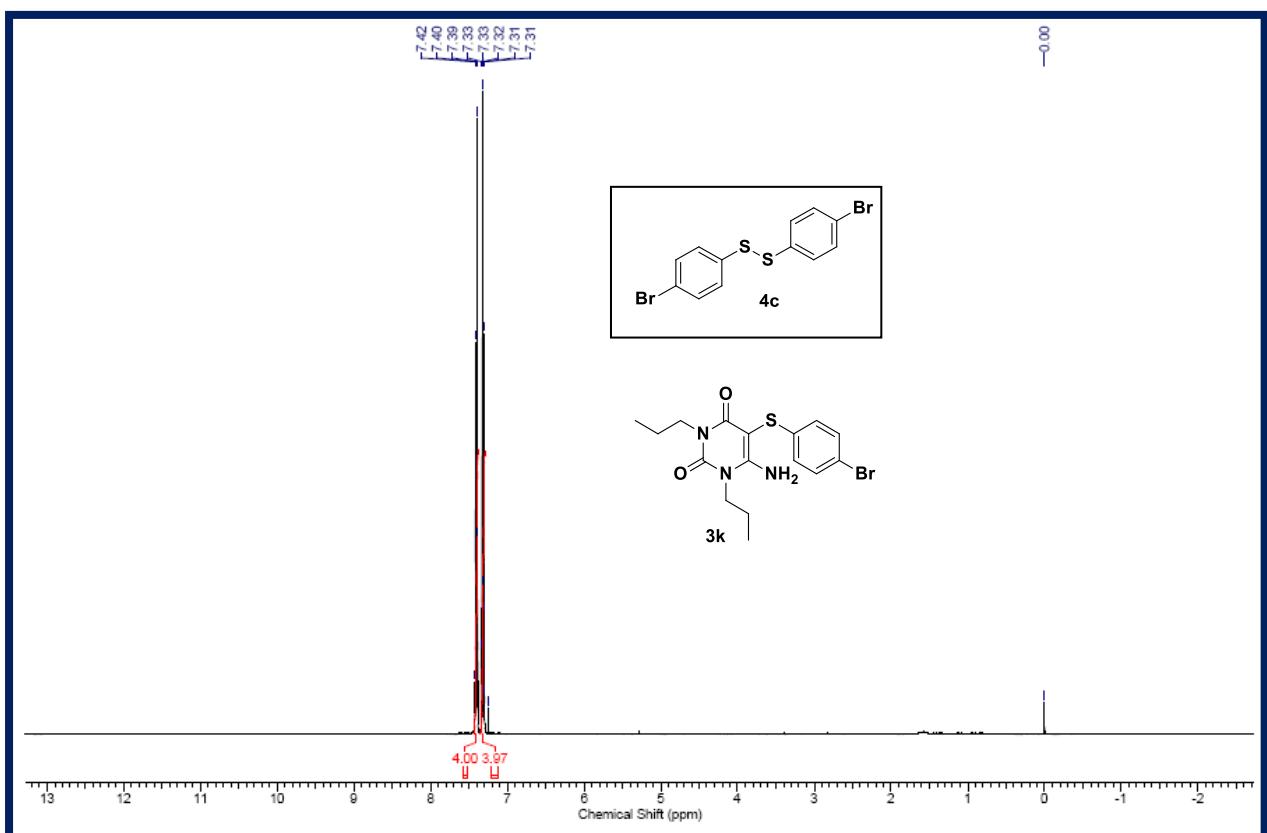
¹H NMR 4b



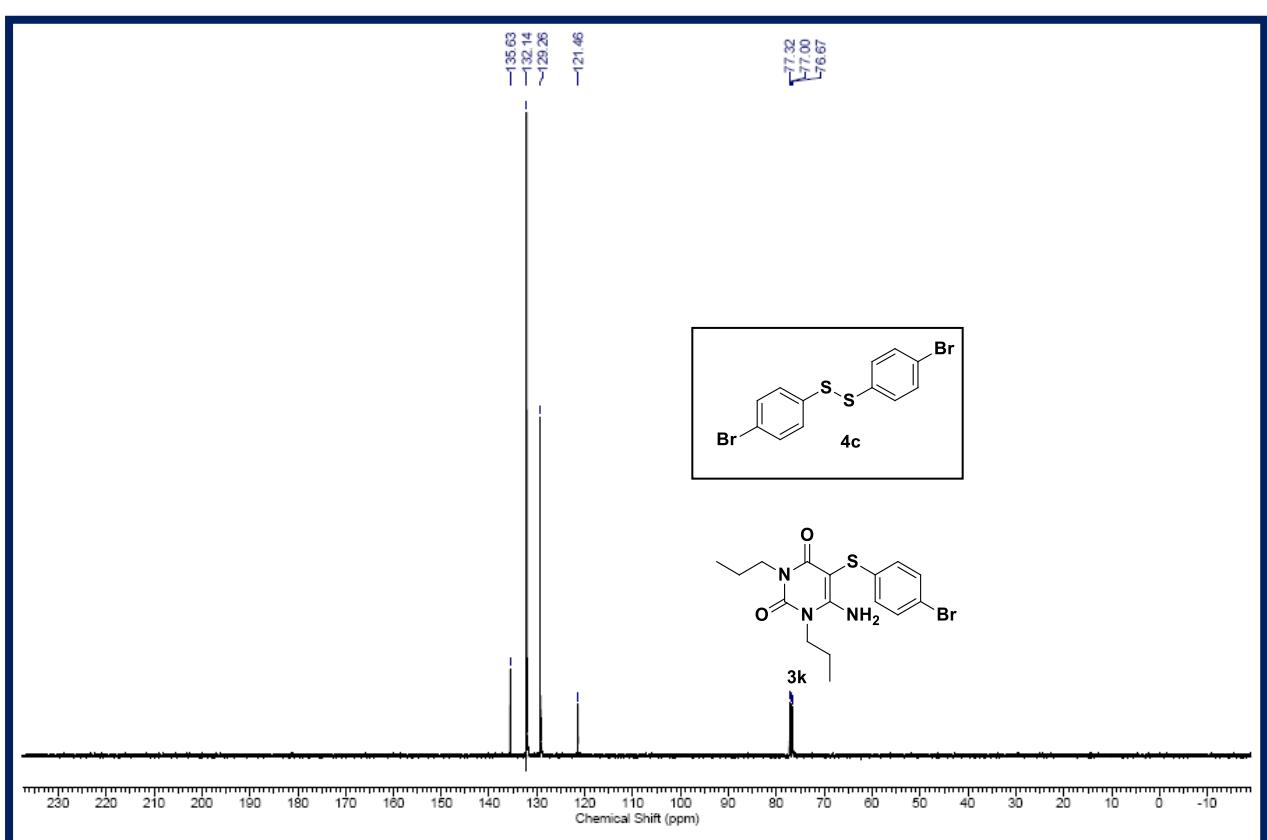
¹³C NMR 4b



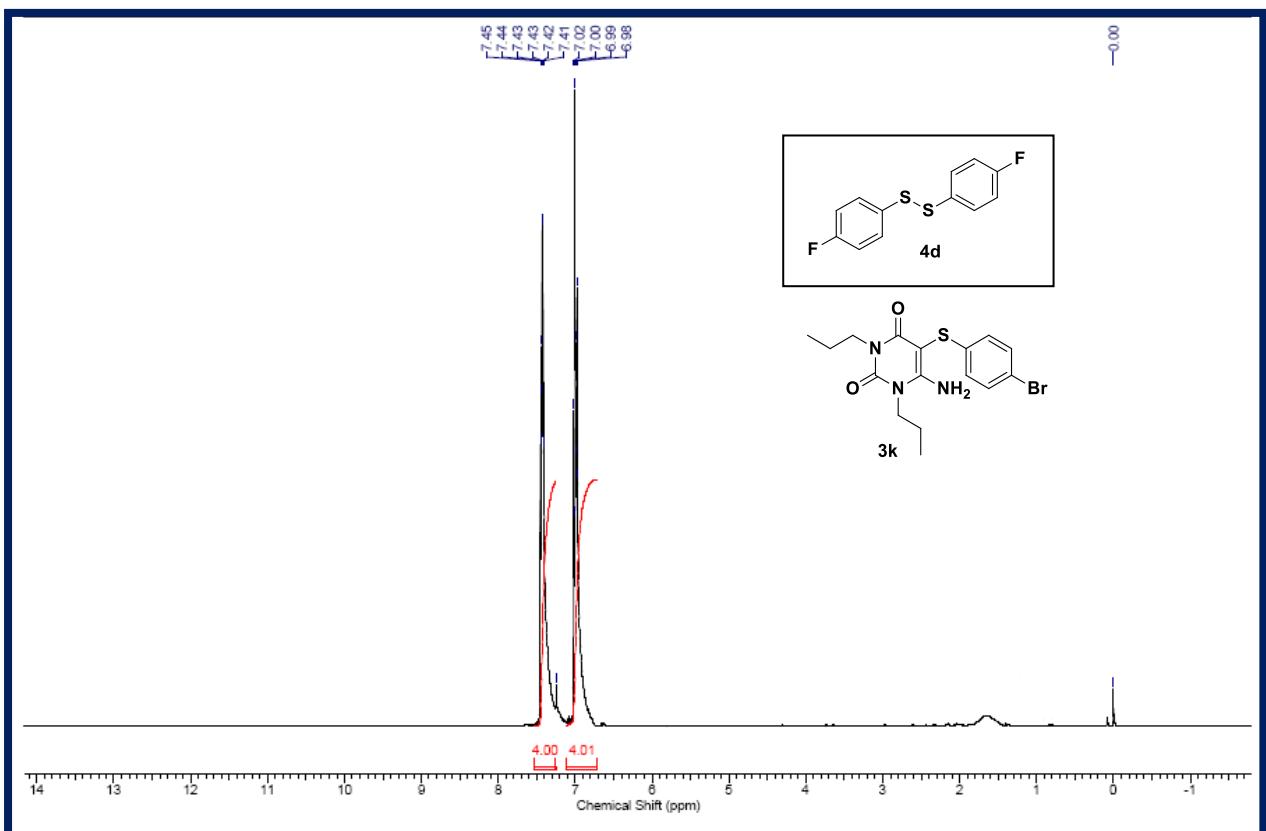
¹H NMR 4c



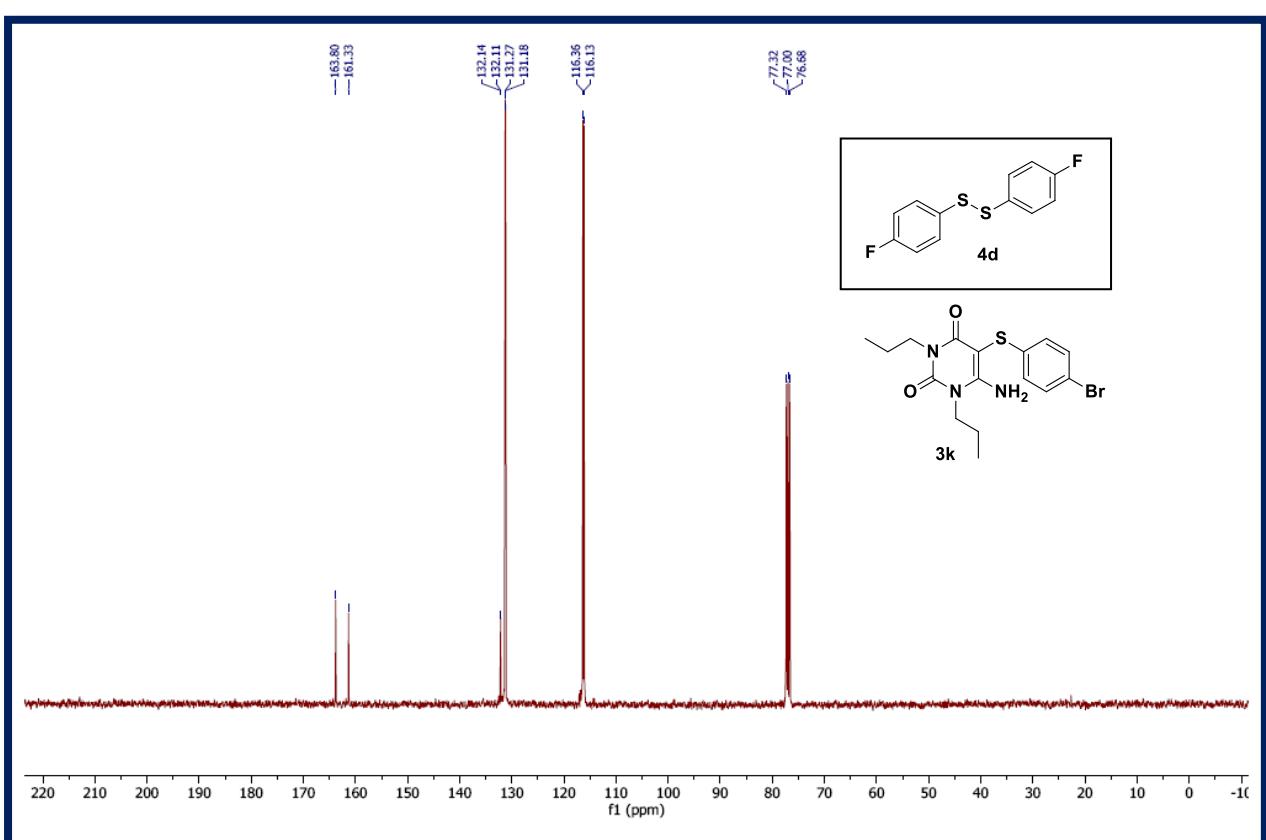
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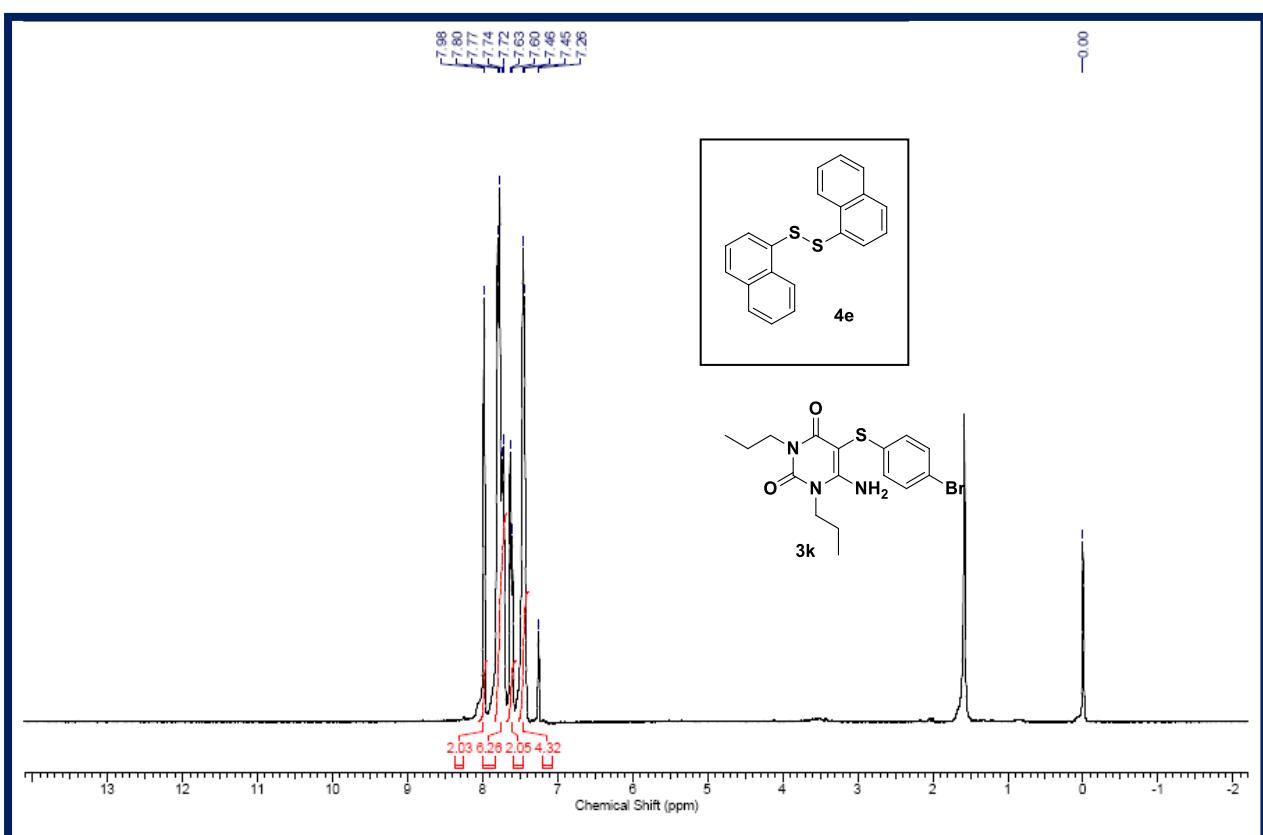
¹H NMR 4d



¹³C NMR 4d



¹H NMR 4e



¹³C NMR 4e

