

## Supplementary Information

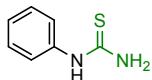
### Green and Efficient Synthesis of Thioureas, Ureas, Primary O-Thiocarbamates, and Carbamates in Deep Eutectic Solvent/ Catalyst Systems Using Thiourea and Urea

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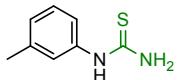
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## Physical data



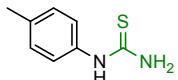
### 1-Phenylthiourea (**3a**)

White solid (0.114 gr, 75% yield), m.p. 146 °C (Literature report 148-151 °C); <sup>1</sup>H NMR (DMSO-*d*<sub>6</sub>, 300 MHz, 25 °C): δ (ppm) 9.70 (s, NH), 8.09-7.17 (brNH<sub>2</sub> and m, 3H, aromatic ring), 7.12 (t, *J* = 9 Hz, 1H, aromatic ring); <sup>13</sup>C{H} NMR (DMSO-*d*<sub>6</sub>, 75 MHz, 25 °C): δ (ppm) 181.38, 139.51, 129.17, 124.85, 123.48; FT-IR (KBr, cm<sup>-1</sup>): 3425 (vs), 3278 (s), 3181 (vs), 3001 (m), 1611 (vs), 1590 (s), 1519 (vs), 1461 (m), 1446 (s), 1296-1231 (m) 1061 (m), 810 (w), 750 (s), 693 (s), 638 (w), 605 (w), 499 (s), 481 (s), 463 (m), 416 (vw); Anal. Calcd for C<sub>7</sub>H<sub>8</sub>N<sub>2</sub>S, C, 55.24; H, 5.30; N, 18.40; S, 21.06%; Found C, 55.20; H, 5.25; N, 18.46; S, 21.09 %.



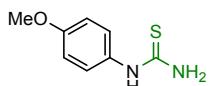
### 1-(*m*-Tolyl) thiourea (**3b**)

White solid (0.130 gr, 78% yield), m.p. 92-94 °C (Literature report 109-111 °C); <sup>2</sup><sup>1</sup>H NMR (DMSO-*d*<sub>6</sub>, 300 MHz, 25 °C): δ (ppm) 9.64 (s, NH), 7.69-7.06 (brNH<sub>2</sub> and m, 3H aromatic ring), 6.94 (d, *J* = 6.9 Hz, 1H, aromatic ring), 2.28 (s, 3H, aromatic CH<sub>3</sub>); <sup>13</sup>C{H} NMR (DMSO-*d*<sub>6</sub>, 75 MHz, 25 °C): δ 181.29, 139.32, 138.50, 129.03, 125.63, 124.05, 120.67, 21.50; FT-IR (KBr, cm<sup>-1</sup>): 3421 (s), 3292 (vs), 3184 (vs), 3015 (m), 1610 (vs), 1527 (vs), 1491 (s), 1457 (m), 1298 (m), 1258 (m), 1165 (w), 1064 (m), 876 (vw), 831 (w), 783 (m), 748 (w), 692 (m), 620 (m), 570 (m), 463 (w); Anal. Calcd for C<sub>8</sub>H<sub>10</sub>N<sub>2</sub>S, C, 57.80; H, 6.06; N, 16.85; S, 19.29%; Found C, 57.76; H, 6.03; N, 16.90; S, 19.31 %.



### 1-(*p*-Tolyl) thiourea (**3c**)

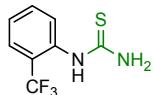
White solid (0.138 gr, 83% yield), m.p. 180 °C (Literature report 185-186 °C); <sup>3</sup><sup>1</sup>H NMR (DMSO-*d*<sub>6</sub>, 300 MHz, 25 °C): δ (ppm) 9.68 (s, NH), 7.02-7.74 (brNH<sub>2</sub> and m, 4H aromatic ring), 2.26 (s, 3H, aromatic CH<sub>3</sub>); <sup>13</sup>C{H} NMR (DMSO-*d*<sub>6</sub>, 75 MHz, 25 °C): δ (ppm) 181.35, 136.91, 134.08, 129.60, 123.71, 20.95; FT-IR (KBr, cm<sup>-1</sup>): 3425 (vs), 3284 (s), 3183 (vs), 2962 (w), 2928 (w), 2856 (w), 1611 (vs), 1594 (m), 1557 (m), 1520 (vs), 1489 (m), 1447 (m), 1396 (m), 1315-1232 (m), 1062 (m), 810 (w), 750 (m), 693 (m), 667 (v), 499 (m); Anal. Calcd for C<sub>8</sub>H<sub>10</sub>N<sub>2</sub>S, C, 57.80; H, 6.06; N, 16.85; S, 19.29%; Found C, 57.74; H, 6.02; N, 16.91; S, 19.32%.



### 1-(4-Methoxyphenyl) thiourea (**3d**)

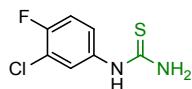
White solid (0.155 gr, 85% yield), m.p. 171 °C (Literature report 172.4 - 173.2 °C); <sup>4</sup><sup>1</sup>H NMR (DMSO-*d*<sub>6</sub>, 400 MHz, 25 °C): δ (ppm) 9.68 (s, NH), 8.00-7.13 (brNH<sub>2</sub>), 7.40 (d, 2H, *J* = 6 Hz, aromatic ring), 7.31 (d, 2H, *J* = 6 Hz, aromatic ring), 4.13 (s, 3H, OCH<sub>3</sub>); <sup>13</sup>C{H} NMR (101 MHz, DMSO-*d*<sub>6</sub>, 25 °C) δ (ppm) 180.95, 139.00, 128.62, 124.35, 122.99, 48.51; FT-IR (KBr, cm<sup>-1</sup>): 3416 (s), 3270 (s), 3134 (vs), 3019 (m), 2831 (vw), 2784 (vw), 1620 (s), 1592 (m), 1514 (s), 1472 (m), 1431 (m),

1392 (m), 1310-1236 (m), 1064 (m), 810 (w), 753 (m), 696 (m), 631 (m), 609 (m), 497 (m); Anal. Calcd for C<sub>8</sub>H<sub>10</sub>N<sub>2</sub>OS, C, 52.73; H, 5.53; N, 15.37; O, 8.78; S, 17.59%; Found C, 52.70; H, 5.49; N, 15.40; S, 17.61 %.



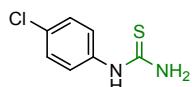
#### 1-(2-(Trifluoromethyl phenyl) thiourea (**3e**)

White solid (0.152 gr, 69% yield), m.p. 159 °C (Literature report 170 °C); <sup>1</sup>H NMR (DMSO-d<sub>6</sub>, 300 MHz, 25 °C): δ (ppm) 9.30 (s, NH), 8.06-7.28 (brNH<sub>2</sub>) 7.64-7.72 (m, 2H, aromatic ring), 7.54 (d, J = 9 Hz, 1H, aromatic ring), 7.45 (t, J = 7.5 Hz, 1H, aromatic ring); <sup>13</sup>C{H} NMR (DMSO-d<sub>6</sub>, 75 MHz, 25 °C): δ (ppm) 183.49, 135.54 (q, J = 315.75 Hz), 132.39, 127.48, 126.49 (q, J = 5.25 Hz), 125.92 (q, J = 29.25 Hz), 125.81, 122.19; FT-IR (KBr, cm<sup>-1</sup>): 3431 (vs), 3250 (m), 3140 (s), 3068 (m), 2982 (w), 1620 (vs), 1513 (s), 1454 (m), 1319 (vs), 1294 (s), 1270 (m), 1230 (w), 1179 (s), 1157 (s), 1131 (vs), 1116 (vs), 1065- 1035 (m), 828 (w), 785 (s), 762 (m), 640 (w), 614 (m), 524 (w), 481 (m), 467 (m); Anal. Calcd for C<sub>8</sub>H<sub>7</sub>F<sub>3</sub>N<sub>2</sub>S, C, 43.63; H, 3.20; N, 12.72; S, 14.56%; Found C, 43.60; H, 3.18; N, 12.75, 14.57%.



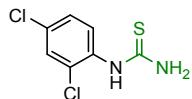
#### 1-(3-Chloro-4-fluorophenyl) thiourea (**3f**)

White solid (0.119 gr, 58% yield), m.p. 187-188 °C (Literature report 236 °C); <sup>1</sup>H NMR (DMSO-d<sub>6</sub>, 300 MHz, 25 °C): δ (ppm) 9.79 (s, NH), 7.76 (d, J = 6 Hz, 1H, aromatic ring), 7.39-7.28 (m, 4H, brNH<sub>2</sub> and 2H, aromatic ring); <sup>13</sup>C{H} NMR (DMSO-d<sub>6</sub>, 75 MHz, 25 °C): δ(ppm) 181.85, 156.11-152.88 (d, J = 242.25 Hz), 136.93-136.89 (d, J = 3 Hz), 125.46, 124.29-124.20 (d, J = 6.75 Hz), 119.43-119.19 (d, J = 18 Hz), 117.21-116.92 (d, J = 21.75 Hz); FT-IR (KBr, cm<sup>-1</sup>): 3422 (vs), 3270 (s), 3160 (vs), 3090 (s), 2992 (m), 1625 (s), 1590 (m), 1522-1471 (s), 1406 (m), 1396 (m), 1266-1275 (s), 1116 (w), 1054 (s), 884 (w), 843 (w), 816 (m), 704 (s), 634 (m), 591 (m), 546 (m), 497 (m). Anal. Calcd for C<sub>7</sub>H<sub>6</sub>Cl<sub>1</sub>F<sub>1</sub>N<sub>2</sub>S, C, 41.08; H, 2.96; N, 13.69; S, 15.67%; Found C, 41.04; H, 2.92; N, 13.72; S, 15.69%.



#### 1-(4-Chlorophenyl) thiourea (**3g**)

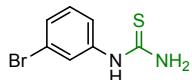
White solid (0.118 gr, 63% yield), m.p. 178 °C (Literature report 174.7-178.9 °C); <sup>1</sup>H NMR (DMSO-d<sub>6</sub>, 250 MHz, 25 °C): δ(ppm) 9.72(s, NH), 7.23-7.75 (brNH<sub>2</sub>), 7.44 (d, J = 10 Hz, 2H, aromatic ring), 7.34 (d, J = 10 Hz, 2H, aromatic ring); <sup>13</sup>C{H} NMR (DMSO-d<sub>6</sub>, 101 MHz, 25 °C) δ(ppm) 181.12, 138.17, 128.45, 128.06, 124.54; FT-IR (KBr, cm<sup>-1</sup>): 3435(vs), 3284 (m), 3187 (s), 3089 (w), 3006 (w), 1622 (vs), 1586 (m), 1529 (vs), 1487 (s), 1401 (m), 1308-1233 (m), 1160 (m), 1088 (m), 1057 (m), 1012 (m), 886 (w), 810 (m), 703 (m), 615 (m), 576 (w), 485 (s), 418 (w); Anal. Calcd for C<sub>7</sub>H<sub>6</sub>ClN<sub>2</sub>S, C, 45.04; H, 3.78; N, 15.01; S, 17.18%; Found C, 45.01; H, 3.74; N, 15.04; S, 17.20%.



#### 1-(2, 4-Dichlorophenyl) thiourea (**3h**)

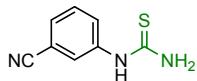
White solid (0.124 gr, 56% yield), m.p. 160 °C (Literature report 160 °C); <sup>1</sup>H NMR (DMSO-d<sub>6</sub>, 300 MHz, 25 °C): δ(ppm) 9.75 (s, NH), 8.13-7.30 (brNH<sub>2</sub>), 7.70 (s, 1H, aromatic ring), 7.65 (d, J = 9 Hz, 1H, aromatic ring), 7.43 (d, J = 9 Hz, 1H, aromatic ring); <sup>13</sup>C{H} NMR (DMSO-d<sub>6</sub>, 75 MHz, 25 °C): δ (ppm) 181.88, 135.82, 131.67, 131.55, 130.88, 129.44, 127.92. FT-IR (KBr, cm<sup>-1</sup>): 3698 (vw), 3453 (s), 3398 (s), 3189 (s), 3081 (vw), 2986 (m), 1583 (s), 1523 (vs), 1474 (s), 1382 (w),

1326 (s), 1306 (vs), 1234 (m), 1227 (m), 1136 (w), 1100 (s), 1060 (m), 867 (s), 845-722 (m), 678 (s), 556 (w), 462 (w); Anal. Calcd for  $C_7H_6Cl_2N_2S$ , C, 38.03; H, 2.74; N, 12.67; S, 14.50%; Found C, 37.98; H, 2.70; N, 12.71; S, 14.53%.



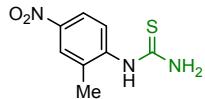
### 1-(3-Bromophenyl) thiourea (**3i**)

White solid (0.148 gr, 64% yield), m.p. 150-151 °C (Literature report 151-152 °C);  $^2H$  NMR (DMSO- $d_6$ , 300 MHz, 25 °C): δ(ppm) 9.83 (s, NH), 7.82 (s, 1H, aromatic ring), 8.27-7.26 (brNH<sub>2</sub> and m, 3H, aromatic ring);  $^{13}C\{H\}$  NMR (DMSO- $d_6$ , 75 MHz, 25 °C): δ(ppm) 181.56, 141.39, 130.94, 127.14, 125.42, 121.94, 121.58; FT-IR (KBr, cm<sup>-1</sup>): 3422 (vs), 3256 (m), 3158 (vs), 3081 (w), 2993 (vw), 1620 (vs), 1590 (m), 1578 (s), 1522 (vs), 1467 (s), 1405 (w), 1302 (s), 1256 (w), 1222 (w), 1066 (s), 889-830 (vw), 790 (m), 689-609 (w), 492 (m); Anal. Calcd for  $C_7H_7BrN_2S$ , C, 36.38; H, 3.05; N, 12.12; S, 13.87%; Found C, 36.35; H, 3.01; N, 12.14; S, 13.90 %.



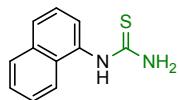
### 1-(3-Cyanophenyl) thiourea (**3j**)

White solid (0.106 gr, 60% yield), m.p. 159-161 °C (Literature report 158-160 °C);  $^2H$  NMR (DMSO- $d_6$ , 300 MHz, 25 °C): δ (ppm) 10.18 (s, NH), 8.07 (s, 1H, aromatic ring), 7.80-7.17 (brNH<sub>2</sub> and m, 3H, aromatic ring);  $^{13}C\{H\}$  NMR (DMSO- $d_6$ , 75 MHz, 25 °C): δ(ppm) 181.77, 140.81, 130.32, 127.87, 127.69, 125.79, 119.15, 111.59; FT-IR (KBr, cm<sup>-1</sup>): 3364(m), 3187 (s), 3144 (s), 3042 (s), 2804 (vw), 2053 (w), 1672 9vs, 1623 (vs), 1572 (s), 1530 (vs), 1502 (s), 1449 (s), 1407 (s), 1357 (m), 1334 (w), 1265 (m), 1231 (w), 1201(s), 1158 (w), 1139 (w), 936 (vw), 879 (vw), 761(vw), 714-572 (vw); Anal. Calcd for  $C_8H_7N_3S$ , C, 54.22; H, 3.98; N, 23.71; S, 18.09%; Found C, 54.17; H, 3.95; N, 23.76; S, 18.12%.



### 1-(2-Methyl-4-nitrophenyl) thiourea (**3k**)

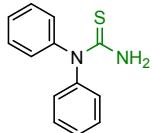
Pale yellow solid (0.110 gr, 52% yield), m.p. 162 °C (Literature report 188.5 °C);  $^9H$  NMR (DMSO- $d_6$ , 300 MHz, 25 °C): δ(ppm) 9.43 (s, NH), 8.28 (s, 1H), 8.18-7.26 (brNH<sub>2</sub>), 7.99 (d,  $J$  = 6 Hz, 1H, aromatic ring, 7.52 (d,  $J$  = 9Hz, 1H, aromatic ring), 2.31 (s, 3H, aromatic CH<sub>3</sub>);  $^{13}C\{H\}$  NMR (DMSO- $d_6$ , 75 MHz, 25 °C): δ(ppm) 182.50, 146.07, 142.58, 138.91, 131.85, 122.42, 120.92, 18.40; FT-IR (KBr, cm<sup>-1</sup>): 3442 (m), 3431 (m), 3246 (m), 3138 (s), 2962 (m), 2760 (vw), 2666 (vw), 1615 (s), 1540 (vs), 1516 (m), 1491 (m), 1348 (vs), 1308-1257 (m), 1188 (w), 1133 (w), 1092 (w), 1056 (m), 835 (m), 742 (m), 703 (w), 649 (w), 604 (w), 528 (w); Anal. Calcd for  $C_8H_9N_3O_2S$ , C, 45.49; H, 4.29; N, 19.89; S, 15.18%; Found C, 45.45; H, 4.26; N, 21.89; S, 15.21 %.



### 1-Naphthalen-1-yl thiourea (**3l**)

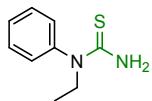
White solid (0.115 gr, 57% yield), m.p. 197 °C (Literature report 193-197 °C);  $^{10}H$  NMR (DMSO- $d_6$ , 300 MHz, 25 °C): δ (ppm) 9.77 (s, NH), 7.97 (d,  $J$  = 6 Hz, 1H, aromatic ring), 7.88 (t,  $J$  = 7.5 Hz, 1H, aromatic ring), 7.46-7.60 (brNH<sub>2</sub> and m, 4H, aromatic ring);  $^{13}C\{H\}$  NMR (DMSO- $d_6$ , 75 MHz, 25 °C): δ(ppm) 182.68, 134.82, 134.41, 130.11, 128.58, 127.34, 126.73, 126.68, 126.25, 125.49, 123.30; FT-IR (KBr, cm<sup>-1</sup>): 3415 (vs), 3267 (m), 3167 (s), 2993-2928 (vw), 1618 (s), 1522

(s), 1408 (w), 1288 (m), 1097 (m), 1059 (m), 1040 (m), 792 (m), 772 (m), 716 (w), 638 (w), 493 (m); Anal. Calcd for C<sub>11</sub>H<sub>10</sub>N<sub>2</sub>S, C, 65.32; H, 4.98; N, 13.85; S, 15.85%; Found C, 65.27; H, 4.95; N, 13.89; S, 15.89 %.



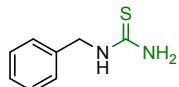
#### 1,1-Diphenylthiourea (**3m**)

White solid (0.114 gr, 50% yield), m.p. 210 °C (Literature report 212 °C); <sup>11</sup><sup>1</sup>H NMR (DMSO-d<sub>6</sub>, 300 MHz, 25 °C): δ (ppm) 8.17-7.01 (brNH<sub>2</sub> and m, 10H, aromatic ring); <sup>13</sup>C{H} NMR (DMSO-d<sub>6</sub>, 75 MHz, 25 °C): δ(ppm) 183.22, 145.13, 129.83, 128.87, 127.53; FT-IR (KBr, cm<sup>-1</sup>): 3452 (vs), 3277 (s), 3144 (w), 3032 (vw), 1594 (vs), 1491 (s), 1437 (vs), 1352 (vs), 1339 (vs), 1261 (w), 1075 (w), 1020 (vw), 1002 (vw), 819 (m), 769 (w), 756 (w), 705 (s), 693 (m), 625 (w), 561 (m), 543 (m), 491 (w); Anal. Calcd for C<sub>13</sub>H<sub>12</sub>N<sub>2</sub>S, C, 68.39; H, 5.30; N, 12.27; S, 14.04 %; Found C, 68.34; H, 5.27; N, 12.30; S, 14.08 %.



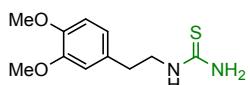
#### 1-Ethyl-1-phenylthiourea (**3n**)

White solid (0.112 gr, 65% yield), m.p. 108 °C (Literature report 110 °C); <sup>12</sup><sup>1</sup>H NMR (DMSO-d<sub>6</sub>, 300 MHz, 25 °C): δ (ppm) 7.20-7.49 (brNH<sub>2</sub> and m, 5H, aromatic ring), 3.98-4.05 (q, J = 15 Hz, 2H, CH<sub>2</sub>), 1.04 (t, J = 7.5 Hz, 3H, CH<sub>3</sub>); <sup>13</sup>C{H} NMR (DMSO-d<sub>6</sub>, 75 MHz, 25 °C): δ(ppm) 181.49, 142.33, 130.37, 128.36, 128.32, 49.47, 13.14; FT-IR (KBr, cm<sup>-1</sup>): 3390 (vs), 3284 (vs), 3226 (s), 3175 (vs), 2976 (w), 2933 (w), 2871 (vw), 1626 (vs), 1597 (vs), 1489 (s), 1470 (vs), 1450 (s), 1422 (m), 1388 (vs), 1374 (s), 1353 (m), 1294 (m), 1186 (w), 1116 (m), 1073 (vw), 1026-997 (w), 938 (w), 817 (s), 772 (s), 709 (m), 700 (s), 677 (m), 640 (w), 615 (vw), 581 (vw), 544 (m), 496 (s); Anal. Calcd for C<sub>9</sub>H<sub>12</sub>N<sub>2</sub>S, C, 59.97; H, 6.71; N, 15.54; S, 17.78 %; Found C, 59.94; H, 6.68; N, 15.58; S, 17.80 %.



#### 1-Benzylthiourea (**3o**)

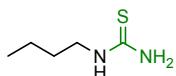
White solid (0.145 gr, 87% yield), m.p. 163-164 °C (Literature report 165 °C); <sup>13</sup><sup>1</sup>H NMR (DMSO-d<sub>6</sub>, 300 MHz, 25 °C): δ(ppm) 8.22 (s, NH), 7.42 –7.14 (brNH<sub>2</sub> and m, 5H, aromatic ring), 4.61 (s, 2H, CH<sub>2</sub>); <sup>13</sup>C{H} NMR (DMSO-d<sub>6</sub>, 75 MHz, 25 °C): δ(ppm) 183.97, 139.79, 128.71, 127.78, 127.65, 127.31, 47.81; FT-IR (KBr, cm<sup>-1</sup>): 3412 (s), 3248 (s), 3191 (s), 3025 (w), 2917 (vw), 2853 (vw), 1628 (vs), 1557 (vs), 1467 (m), 1317 (m), 1233-961 (m), 699 (m), 644 (m), 605 (w), 517 (w), 453 (w); Anal. Calcd for C<sub>8</sub>H<sub>10</sub>N<sub>2</sub>S, C, 57.80; H, 6.06; N, 16.85; S, 19.29%; Found C, 57.74; H, 6.01; N, 16.90, 19.35%.



#### 1-(3,4-Dimethoxyphenethyl)thiourea (**3p**)

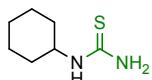
White solid (0.219 gr, 91% yield), m.p. 161-163 °C (Literature report 165 °C); <sup>14</sup><sup>1</sup>H NMR (DMSO-d<sub>6</sub>, 300 MHz, 25 °C): δ(ppm) 7.40 (s, NH), 6.86 (d, J = 9 Hz, 1H, aromatic ring ), 6.81 (s, 1H, aromatic ring), 6.72 (d, J = 9 Hz ,1H, aromatic ring), 3.73 (s. OMe), 3.71 (s, OMe), 3.57 (brs, 2H, CH<sub>2</sub>), 2.71 (t, J = 9 Hz, 2H, CH<sub>2</sub>); <sup>13</sup>C{H} NMR (DMSO-d<sub>6</sub>, 101 MHz, 25 °C): δ(ppm) 183.91, 148.56, 146.97, 132.82, 120.34, 112.37, 111.65, 55.33, 55.21, 43.83, 39.44; FT-IR (KBr, cm<sup>-1</sup>): 3324 (s), 3310 (s), 2999 (s), 2916 (s), 2837 (s), 1608 (s), 1590 (s), 1518 (vs), 1464 (vs), 1452 (vs), 1418 (s), 1382 (s), 1323 (s), 1292

(m), 1262 (vs), 1237 (vs), 1185 (m), 1155 (s), 1141 (s), 1060 (vw), 1026 (s), 934 (vw), 848 (w), 805 (m), 766 (w), 740 (w), 643 (vw), 594 (vw), 553 (vw); Anal. Calcd for C<sub>11</sub>H<sub>16</sub>N<sub>2</sub>O<sub>2</sub>S, C, 54.98; H, 6.71; N, 11.66; S, 13.34%; Found C, 54.92; H, 6.67; N, 11.70; S, 13.37%.



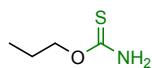
### 1-Butylthiourea (**3q**)

White solid (0.127 gr, 96% yield), m.p. 78-80 °C (Literature report 81 °C); <sup>15</sup><sup>1</sup>H NMR (DMSO-d<sub>6</sub>, 300 MHz, 25 °C): δ (ppm) 7.54 (s, NH), 6.88 (brNH<sub>2</sub>), 3.33 (m, 2H, CH<sub>2</sub>), 1.23-1.5 (m, 4H, CH<sub>2</sub>), 0.87 (q, J = 6 Hz, 3H, CH<sub>3</sub>); <sup>13</sup>C{H} NMR (DMSO-d<sub>6</sub>, 75 MHz, 25 °C): δ(ppm) 183.48, 44.02, 31.37, 19.97, 14.14. FT-IR (KBr, cm<sup>-1</sup>): 3362 (s), 3280 (s), 3235 (s), 3180 (s), 3083-2875 (m), 1618 (s), 1560 (m), 1458 (m), 1432 (m), 1357 (m), 1320 (w), 1163 (m), 1122 (m), 1024 (m), 900 (w), 727(m), 600-502 (m); Anal. Calcd for C<sub>6</sub>H<sub>14</sub>N<sub>2</sub>S, C, 45.42; H, 9.15; N, 21.19; S, 24.25%; Found C, 45.38; H, 9.12; N, 21.21, S, 24.28 %.



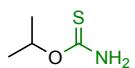
### 1-Cyclohexylthiourea (**3r**)

White solid (0.141 gr, 89% yield), m.p. 160 °C (Literature report 164-165 °C); <sup>3</sup><sup>1</sup>H NMR (DMSO-d<sub>6</sub>, 300 MHz, 25 °C): δ(ppm) 7.51(s, NH), 6.80 (brNH<sub>2</sub>), 1.11-1.85 (m, 11H, CH<sub>2</sub> and NCH); <sup>13</sup>C{H} NMR (DMSO-d<sub>6</sub>, 101 MHz, 25 °C): δ(ppm) 188.94, 55.18, 41.57, 30.70, 29.93; FT-IR (KBr, cm<sup>-1</sup>): 3289 (vs), 3159 (vs), 3101 (vs), 2930 (s), 2853 (m), 2681 (w), 1615 (vs), 1487-1402 (s), 1338 (m), 1259 (w), 1237 (w), 1157 (w), 1089 (m), 1030 (w), 820 (vw), 720 (s), 652 (w), 478 (vw); Anal. Calcd for C<sub>7</sub>H<sub>14</sub>N<sub>2</sub>S C, 53.12; H, 8.92; N, 17.70; S, 20.26%; Found C, 53.09; H, 8.88; N, 17.73; S, 20.30 %.



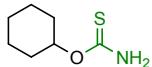
### O-Propyl thiocarbamate (**5a**)

Yellow solid (0.107 gr, 90% yield), m.p. 34-35 °C (Literature report: 35 °C); <sup>16</sup><sup>1</sup>H NMR (DMSO-d<sub>6</sub>, 250 MHz, 25 °C): δ (ppm) 8.57 (s, NH), 8.24 (s, NH), 4.19 (t, J = 7.5 Hz, 2H, OCH<sub>2</sub>), 1.59 (m, J = 7.5 Hz, 2H, CH<sub>2</sub>), 0.85 (t, J = 7.5 Hz, 3H, CH<sub>3</sub>); <sup>13</sup>C{H} NMR (DMSO-d<sub>6</sub>, 63 MHz, 25 °C): δ (ppm) 191.36, 71.07, 21.58, 10.05; FT-IR (KBr, cm<sup>-1</sup>): 3339 (vs), 3277 (s), 3175 (s), 2979 (m), 2927 (m), 1617 (s), 1438 (vs), 1376 (s), 1364 (vw), 1309 (s), 1264 (s), 1185 (m), 1146 (m), 1084 (vs), 917 (s), 859 (m), 694 (m), 670 (vw), 640 (w), 589 (s); Anal. Calcd for C<sub>4</sub>H<sub>9</sub>NOS: C, 40.31; H, 7.61; N, 11.75, S, 26.90%. Found: C, 40.26; H, 7.57; N, 11.78, S, 26.94%.



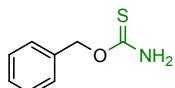
### O-2-Propyl thiocarbamate (**5b**)

Yellow solid (0.098 gr, 82% yield), m.p. 80-81 °C (Literature report: 79-80 °C); <sup>17</sup><sup>1</sup>H NMR (DMSO-d<sub>6</sub>, 250 MHz, 25 °C): δ(ppm) 8.58 (s, NH), 8.23 (s, NH), 5.30 (sept, J = 6.25 Hz, 1H, OCH), 1.21 (d, J = 6.25 Hz, 6H, CH<sub>3</sub>); <sup>13</sup>C NMR (DMSO-d<sub>6</sub>, 63 MHz, 25 °C): δ(ppm) 190.43, 72.78, 21.56; FT-IR (KBr, cm<sup>-1</sup>): 3338 (vs), 3276 (s), 3171 (s), 2978 (m), 2929 (w), 1618 (vs), 1438 (s), 1376 (s), 1364 (s), 1310 (s), 1264 (s), 1185 (s), 1147 (m), 1083 (vs), 917 (s), 858 (s), 694 (m), 670 (vw), 640 (w), 589 (m), 493 (vw); Anal. Calcd for C<sub>4</sub>H<sub>9</sub>NOS: C, 40.31; H, 7.61; N, 11.75, S, 26.90%. Found: C, 40.28; H, 7.57; N, 11.78, S, 26.93%.



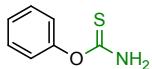
*O*-Cyclohexyl thiocarbamate (**5c**)

Yellow solid (0.127 gr, 80% yield), m.p. 73-75 °C (Literature report 72.5-73.5 °C);  $^{18}\text{H}$  NMR (DMSO- $d_6$ , 250 MHz, 25 °C):  $\delta$ (ppm) 8.57 (s, NH), 8.24 (s, NH), 5.04 (m, 1H, OCH), 1.15-1.88 (m, 10H,  $\text{CH}_2$ );  $^{13}\text{C}\{\text{H}\}$  NMR (DMSO- $d_6$ , 63 MHz, 25 °C):  $\delta$ (ppm) 190.32, 77.40, 31.16, 24.81, 23.34; FT-IR (KBr, cm $^{-1}$ ): 3400 (s), 3330 (s), 3272 (vs), 3172 (vs), 2933 (vs), 2858 (vs), 1612 (vs), 1430 (s), 1376 (s), 1353 (s), 1321 (s), 1300 (s), 1154 (m), 1076 (vs), 1008 (s), 942 (s), 932 (s), 892 (m), 845 (m), 807 (w), 789 (w), 736 (vs), 670 (w), 644 (s), 586 (s), 548 (s), 520 (m); Anal. Calcd for  $\text{C}_7\text{H}_{13}\text{NOS}$ : C, 52.80; H, 8.23; N, 8.80, S, 20.13%. Found: C, 52.77; H, 8.79; N, 8.85, S, 20.12%.



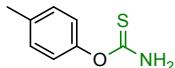
*O*-Benzyl thiocarbamate (**5d**)

Yellow solid (0.137 gr, 82% yield), m.p. 63-65 °C (Literature report 61-62 °C);  $^{19}\text{H}$  NMR (DMSO- $d_6$ , 250 MHz, 25 °C):  $\delta$ (ppm) 8.09 (s, NH), 7.73 (s, NH), 7.41-7.32 (m, 2H, aromatic ring), 7.20 (t,  $J$  = 7.5 Hz, 1H, aromatic ring), 7.07 (d,  $J$  = 7.5 Hz, 1H, aromatic ring), 6.70 (d,  $J$  = 7.5 Hz, 1H, aromatic ring), 5.71 (s, 2H,  $\text{CH}_2$ );  $^{13}\text{C}\{\text{H}\}$  NMR (DMSO- $d_6$ , 101 MHz, 25 °C):  $\delta$ (ppm) 183.99, 142.49, 128.05, 126.65, 126.47, 63.08; FT-IR (KBr, cm $^{-1}$ ): 3408 (s), 3277 (s), 3161 (s), 2925 (w), 1606 (vs), 1490 (w), 1458 (vw), 1426 (s), 1290 (m), 1227 (m), 1178 (vs), 1156 (w), 1105 (w), 1113 (s), 1040 (w), 1017 (s), 864 (s), 832 (w), 816 (m), 782 (m), 712 (s), 643 (vw), 630 (w), 592 (w), 541 (s), 496 (m), 448 (vw); Anal. Calcd for  $\text{C}_8\text{H}_9\text{NOS}$ : C, 57.46; H, 5.42; N, 8.38; S, 19.17%. Found: C, 57.40; H, 5.38; N, 8.42; S, 19.20%.



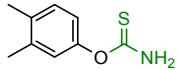
*O*-Phenyl thiocarbamate (**5e**)

Yellow solid (0.106 gr, 69% yield), m.p. 133-134 °C (Literature report 136-137 °C);  $^{20}\text{H}$  NMR (DMSO- $d_6$ , 250 MHz, 25 °C):  $\delta$ (ppm) 9.22 (s, NH), 9.02 (s, NH), 7.37 (t,  $J$  = 7.5 Hz, 2H, aromatic ring), 7.21 (t,  $J$  = 7.5 Hz, 1H, aromatic ring), 7.04 (d,  $J$  = 10 Hz, 2H, aromatic ring);  $^{13}\text{C}\{\text{H}\}$  NMR (DMSO- $d_6$ , 63 MHz, 25 °C):  $\delta$ (ppm) 190.36, 153.16, 129.02, 125.54, 122.78; FT-IR (KBr, cm $^{-1}$ ): 3448 (vw), 3410 (vs), 3268 (s), 3160 (s), 1601 (s), 1458 (w), 1430 (s), 1289 (m), 1222 (w), 1199 (vs), 1162 (w), 1110 (vw), 1066 (w), 1024 (s), 1000 (s), 910 (w), 852 (s), 772 (s), 710 (m), 691 (s), 670 (vw), 622 (w), 563 (m), 503 (vw); Anal. Calcd for  $\text{C}_7\text{H}_7\text{NOS}$ : C, 54.88; H, 4.61; N, 9.14; S, 20.93%. Found: C, 54.83; H, 4.58; N, 9.17; 20.96%.



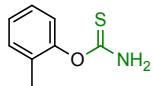
*O*-(*p*-Tolyl) thiocarbamate (**5f**)

Yellow solid (0.125 gr, 75% yield), m.p. 148-149 °C (Literature report 151-152 °C);  $^{21}\text{H}$  NMR (DMSO- $d_6$ , 250 MHz, 25 °C):  $\delta$ (ppm) 9.18 (s, NH), 8.98 (s, NH), 7.15 (d,  $J$  = 8.5 Hz, 2H, aromatic ring), 6.91 (d,  $J$  = 8.5 Hz, 2H, aromatic ring), 2.28 (s, 3H, aromatic  $\text{CH}_3$ );  $^{13}\text{C}\{\text{H}\}$  NMR (DMSO- $d_6$ , 63 MHz, 25 °C):  $\delta$ (ppm) 190.56, 150.99, 134.63, 129.40, 122.44, 20.36; FT-IR (KBr, cm $^{-1}$ ): 3448 (w), 3413 (vs), 3270 (s), 3156 (s), 2956 (w), 2923 (w), 1602 (vs), 1501 (m), 1420 (vs), 1291 (s), 1220 (s), 1198 (vs), 1162 (s), 1104 (m), 1017 (vs), 864 (s), 832 (w), 816 (m), 782 (w), 712 (m), 643 (vw), 630 (vw), 592 (w), 541 (s), 596 (m), 448 (vw); Anal. Calcd for  $\text{C}_8\text{H}_9\text{NOS}$ : C, 57.46; H, 4.42; N, 8.38, S, 19.17%. Found: C, 57.41; H, 4.38; N, 8.43, S, 19.21%.



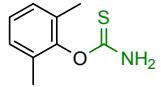
*O*-(3,4-Dimethylphenyl) thiocarbamate (**5g**)

Yellow solid (0.140 gr, 77% yield), m.p. 140–141 °C;  $^1\text{H}$  NMR (DMSO- $d_6$ , 250 MHz, 25 °C):  $\delta$ (ppm) 9.13 (s, NH), 8.93 (s, NH), 7.10 (d,  $J$  = 8.0 Hz, 2H, aromatic ring), 6.81 (s, 1H, aromatic ring), 6.74 (d,  $J$  = 7.5, 1H, aromatic ring), 2.19 (s, 3H, aromatic CH<sub>3</sub>), 2.18 (s, 3H, aromatic CH<sub>3</sub>);  $^{13}\text{C}$  NMR (DMSO- $d_6$ , 63 MHz, 25 °C):  $\delta$ (ppm) 190.64, 151.13, 136.99, 133.36, 129.70, 123.38, 119.77, 19.31; FT-IR (KBr, cm<sup>-1</sup>): 3409 (s), 3282 (s), 3182 (s), 2957 (w), 2924 (w), 1612 (vs), 1474 (m), 1466 (w), 1459 (w), 1421 (vs), 1376 (w), 1302 (s), 1242 (m), 1169 (vs), 1092 (s), 1025 (s), 961 (w), 857 (s), 782 (s), 748 (w), 739 (w), 656 (vw), 628 (m), 590 (vw), 529 (vs); Anal. Calcd for C<sub>9</sub>H<sub>11</sub>NOS: C, 59.64; H, 6.12; N, 7.73, S, 17.69%. Found: C, 59.61; H, 6.10; N, 7.76, S, 17.65%.



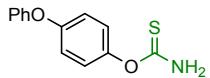
*O*-(*o*-Tolyl) thiocarbamate (**5h**)

Yellow solid (0.120 gr, 72% yield), m.p. 134–135 °C (Literature report 132–134 °C);  $^{22}\text{H}$  NMR (DMSO- $d_6$ , 250 MHz, 25 °C):  $\delta$ (ppm) 9.22 (s, NH), 9.03 (s, NH), 7.17 (m, 3H, aromatic ring), 6.96 (m, 1H, aromatic ring), 2.13 (s, 3H, aromatic CH<sub>3</sub>);  $^{13}\text{C}\{\text{H}\}$  NMR (DMSO- $d_6$ , 63 MHz, 25 °C):  $\delta$ (ppm) 195.13, 156.89, 135.92, 132.50, 131.90, 130.94, 128.28, 20.98; FT-IR (KBr, cm<sup>-1</sup>): 3402 (vs), 3274 (vs), 3170 (vs), 2934 (w), 1606 (vs), 1490 (w), 1474 (w), 1458 (m), 1426 (vs), 1290 (s), 1227 (s), 1178 (vs), 1156 (m), 1113 (s), 1040 (m), 1017 (s), 865 (w), 856 (s), 787 (w), 774 (m), 719 (s), 664 (w), 640 (w), 623 (w), 569 (s), 473 (vw); Anal. Calcd for C<sub>8</sub>H<sub>9</sub>NOS: C, 57.46; H, 4.42; N, 8.38, S, 19.17%. Found: C, 57.42; H, 4.37; N, 8.44, S, 19.20%.



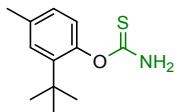
*O*-(2,6-Dimethylphenyl) thiocarbamate (**5i**)

Yellow solid (0.120 gr, 66% yield), m.p. 168–169 °C (Literature report 171–172 °C);  $^{19}\text{H}$  NMR (DMSO- $d_6$ , 250 MHz, 25 °C):  $\delta$ (ppm) 9.17 (s, NH), 8.98 (s, NH), 7.03 (m, 3H, aromatic ring), 2.09 (s, 6H, aromatic CH<sub>3</sub>);  $^{13}\text{C}\{\text{H}\}$  NMR (DMSO- $d_6$ , 63 MHz, DMSO- $d_6$ ):  $\delta$ (ppm) 189.18, 150.18, 130.63, 128.21, 125.37, 15.93; FT-IR (KBr, cm<sup>-1</sup>): 3410 (s), 3292 (s), 3181 (s), 2956 (vw), 2923 (w), 1611 (vs), 1466 (w), 1459 (w), 1420 (vs), 1376 (w), 1302 (s), 1383 (s), 1267 (m), 1239 (w), 1174 (vs), 1167 (vs), 1092 (s), 1025 (s), 898 (w), 857 (s), 782 (s), 748 (w), 739 (w), 656 (w), 628 (w), 590 (vw), 529 (vs); Anal. Calcd for C<sub>9</sub>H<sub>11</sub>NOS: C, 59.64; H, 6.12; N, 7.73, S, 17.69%. Found: C, 59.60; H, 6.10; N, 7.76, S, 17.68%.



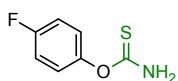
*O*-(4-Phenoxyphenyl) thiocarbamate (**5j**)

Yellow solid (0.191 gr, 78% yield), m.p. 139–141 °C;  $^1\text{H}$  NMR (DMSO- $d_6$ , 250 MHz, 25 °C):  $\delta$ (ppm) 9.25 (s, NH), 9.04 (s, NH), 7.40 (m, 2H, aromatic ring), 7.05 (m, 7H, aromatic ring);  $^{13}\text{C}$  NMR (DMSO- $d_6$ , 63 MHz, 25 °C):  $\delta$ (ppm) 190.53, 156.60, 153.96, 148.74, 130.05, 124.24, 123.51, 118.86, 118.54; FT-IR (KBr, cm<sup>-1</sup>): 3418 (s), 3276 (m), 3172 (w), 2998 (vw), 1602 (s), 1504 (s), 1438 (w), 1358 (w), 1344 (w), 1294 (w), 1211 (vs), 1094 (w), 1074 (w), 1010 (w), 978 (m), 875 (s), 850 (vs), 815 (s), 752 (vs), 691 (m), 629 (w), 549 (w), 501 (s); Anal. Calcd for C<sub>13</sub>H<sub>11</sub>NO<sub>2</sub>S: C, 63.65; H, 4.52; N, 5.71, S, 13.07%. Found: C, 63.61; H, 4.48; N, 5.73, S, 13.10%.



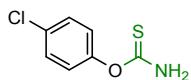
*O*-(2-(*tert*-Butyl)-4-methylphenyl) thiocarbamate (**5k**)

Yellow solid (0.116 gr, 52% yield), m.p. 164-165 °C; <sup>1</sup>H NMR (DMSO-*d*<sub>6</sub>, 250 MHz, 25 °C): δ(ppm) 9.15 (s, NH), 8.96 (s, NH), 7.10 (s, 1H, aromatic ring), 6.98 (d, *J* = 7.5 Hz, 1H, aromatic ring), 6.83 (d, *J* = 7.5 Hz, 1H, aromatic ring), 2.07 (s, 3H, aromatic CH<sub>3</sub>), 1.28(s, 9H, C(CH<sub>3</sub>)<sub>3</sub>); <sup>13</sup>C{H} NMR (DMSO-*d*<sub>6</sub>, 63 MHz, 25 °C): δ(ppm) 190.44, 149.32, 140.34, 134.07, 127.15, 126.60, 125.21, 34.01, 30.17, 20.70; FT-IR (KBr, cm<sup>-1</sup>): 3382 (s), 3283 (vs), 3178 (vs), 3033 (w), 3009 (w), 2961 (s), 2922 (m), 2869 (w), 1611 (s), 1420 (vs), 1340 (w), 1288 (m), 1274 (s), 1261 (w), 1204 (vs), 1141 (w) 1092 (s), 1021 (s), 937 (w), 913 (vw), 878 (w), 846 (s), 815 (w), 748 (vw), 682 (vw), 670 (vw), 663 (vw), 631 (w), 614 (w), 526 (s), 450 (vw); Anal. Calcd for C<sub>11</sub>H<sub>16</sub>FNOS: C, 49.11; H, 3.53; N, 8.18, S, 18.73%. Found: C, 49.07; H, 3.51; N, 8.20, S, 18.75%.



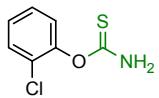
*O*-(4-Fluorophenyl) thiocarbamate (**5l**)

Yellow solid (0.087 gr, 51% yield), m.p. 157-158 °C; <sup>1</sup>H NMR (DMSO-*d*<sub>6</sub>, 250 MHz, 25 °C): δ(ppm) 9.25 (s, NH), 9.04 (s, NH), 7.18 (d,d, *J* = 9.0 Hz, *J* = 4.5 Hz, 2H, aromatic ring), 7.07 (d,d, *J* = 9.0 Hz, *J* = 4.5 Hz, 2H, aromatic ring); <sup>13</sup>C{H} NMR (DMSO-*d*<sub>6</sub>, 63 MHz, 25 °C): δ(ppm) 190.40, 157.56-161.39 (d, *J* = 241.3 Hz), 147.29-147.31 (d, *J* = 1.3 Hz), 124.50-124.64 (d, *J* = 8.2 Hz), 115.38-115.75 (d, *J* = 23.3 Hz); FT-IR (KBr, cm<sup>-1</sup>): 3344 (s), 3265 (s), 3160 (s), 3043 (w), 1612 (s), 1508 (s), 1501 (s), 1474 (w), 1466 (w), 1458 (w), 1430 (vs), 1294 (m), 1254 (m), 1201 (vs), 1087 (m), 1038 (vs), 1016 (m), 878 (m), 859 (m), 841 (m), 803 (s), 718 (w), 670 (vw), 644 (vw), 634 (vw), 592 (m), 510 (vw), 463 (vw); Anal. Calcd for C<sub>7</sub>H<sub>6</sub>FNOS: C, 49.11; H, 3.53; N, 8.18, S, 18.73%. Found: C, 49.07; H, 3.50; N, 8.20, S, 18.71%.



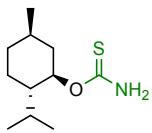
*O*-(4-Chlorophenyl) thiocarbamate (**5m**)

Yellow solid (0.101 gr, 54% yield), m.p. 152-153 °C (Literature report 155-157 °C); <sup>16</sup><sup>1</sup>H NMR (DMSO-*d*<sub>6</sub>, 250 MHz, 25 °C): δ (ppm) 9.32 (s, NH), 9.10 (s, NH), 7.43 (d, *J* = 8.75 Hz, 2H, aromatic ring), 7.09 (d, *J* = 8.75 Hz, 2H, aromatic ring); <sup>13</sup>C{H} NMR (DMSO-*d*<sub>6</sub>, 63 MHz, 25 °C): δ (ppm) 190.02, 151.9, 129.76, 128.94, 124.78; FT-IR (KBr, cm<sup>-1</sup>): 3370 (s), 3270 (vs), 3173 (vs), 2925 (w), 1603 (vs), 1478 (s), 1465 (s), 1458 (s), 1438 (vs), 1283 (m), 1214 (vs), 1084 (vs), 1012 (vs), 864 (s), 842 (m), 822 (w), 738 (s), 714 (w), 631 (w), 622 (m), 548 (m), 522 (s), 469 (m), 439 (w); Anal. Calcd for C<sub>7</sub>H<sub>6</sub>CINOS: C, 44.81; H, 3.22; N, 7.46, S, 17.09%. Found: C, 44.78; H, 3.18; N, 7.51, S, 17.03%.



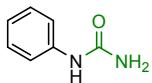
*O*-(2-Chlorophenyl) thiocarbamate (**5n**)

Yellow solid (0.086 gr, 46% yield), m.p. 158-160 °C; <sup>1</sup>H NMR (DMSO-*d*<sub>6</sub>, 250 MHz, 25 °C): δ(ppm) 9.35 (s, NH), 9.18 (s, NH), 7.50 (d, *J* = 7.5 Hz, 1H, aromatic ring), 7.35 (t, *J* = 7.5 Hz, 1H, aromatic ring), 7.22 (m, 2H, aromatic ring); <sup>13</sup>C NMR (DMSO-*d*<sub>6</sub>, 63 MHz, 25 °C): δ(ppm) 189.18, 148.90, 129.81, 127.90, 127.20, 126.81, 125.48; FT-IR (KBr, cm<sup>-1</sup>): 3368 (s), 3277 (vs), 3176 (vs), 1618 (vs), 1474 (s), 1458 (s), 1442 (s), 1285 (m), 1260 (s), 1223 (vs), 1064 (s), 1034 (m), 1017 (s), 871 (m), 856 (m), 767 (m), 728 (m), 717 (m), 670 (w), 633 (w), 617 (w), 560 (m), 494 (w), 426 (vw); Anal. Calcd for C<sub>7</sub>H<sub>6</sub>CINOS: C, 44.81; H, 3.22; N, 7.46, S, 17.09%. Found: C, 44.78; H, 3.16; N, 7.52, S, 17.04%.



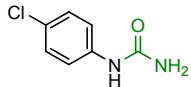
*L*-(-)-Menthyl thiocarbamate (**5o**)

Yellow solid (0.151 gr, 70% yield), m.p. 126-127 °C (Literature report 124 °C);  $^{23}[\alpha]_{D}^{18}=-125$  (*c* 0.05, CHCl<sub>3</sub>); HPLC (254 nm, H<sub>2</sub>O/CH<sub>3</sub>CN (30: 70%)): retention time, 3.06 min; <sup>1</sup>H NMR (DMSO-*d*<sub>6</sub>, 250 MHz, 25 °C): δ (ppm) 8.58 (s, NH), 8.28 (s, NH), 5.02 (m, 1H, OCH), 1.90 (m, 2H, CH<sub>2</sub>), 1.63 (m, 2H, CH<sub>2</sub>), 1.56 (m, 1H, CH), 1.40 (m, 1H, CH), 1.26 (m, 1H, CH), 1.06 (m, 2H, CH<sub>2</sub>), 0.89 (m, 6H, CH<sub>3</sub>), 0.84 (d, *J* = 8.5 Hz, 3H, CH<sub>3</sub>); <sup>13</sup>C{H} NMR (DMSO-*d*<sub>6</sub>, 63 MHz, 25 °C): δ (ppm) 190.73, 79.16, 46.70, 33.70, 30.75, 25.82, 23.08, 21.88, 20.38, 16.65; FT-IR (KBr, cm<sup>-1</sup>): 3401 (vs), 3292 (vs), 3180 (vs), 2964 (vs), 2928 (vs), 2873 (s), 1608 (vs), 1422 (s), 1303 (s), 1179 (s), 1073 (vs), 1055 (s), 981 (s), 954 (s), 917 (s), 878 (s), 848 (s), 808 (vw), 775 (vw), 738 (vs), 706 (w), 626 (m), 556 (s); Anal. Calcd for C<sub>11</sub>H<sub>21</sub>NOS: C, 62.83; H, 10.11; N, 6.11, S, 13.98%. Found: C, 62.80; H, 10.08; N, 6.14, S, 14.00%.



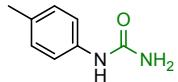
1-Phenylurea (**7a**)

white solid (0.113 gr, 83% yield), m. p. 146-147 °C (Literature report: 143-145 °C); <sup>24</sup> <sup>1</sup>H NMR (DMSO-*d*<sub>6</sub>, 250 MHz, 25 °C): δ (ppm) 8.48 (s, NH), 7.36 (d, *J* = 7.5 Hz, 2H, aromatic ring), 7.19 (t, *J* = 7.5 Hz, 2H, aromatic ring), 6.86 (t, *J* = 7.5 Hz, 1H, aromatic ring), 5.81 (s, NH<sub>2</sub>); <sup>13</sup>C{H} NMR (DMSO-*d*<sub>6</sub>, 63 MHz, 25 °C): δ (ppm) 155.94, 140.46, 128.52, 120.98, 117.64; FT-IR (KBr, cm<sup>-1</sup>): 3428 (vs), 3312 (vs), 3038 (m), 1652 (vs), 1616 (vs), 1594 (vs), 1558 (vs), 1500 (m), 1357 (s), 1291 (w), 1257 (m), 1194 (w), 1117 (w), 1075 (vw), 1034 (w), 904 (w), 860 (w), 774 (w), 752 (vs), 697 (vs), 621 (w), 588 (s), 496 (m), 443 (vw), 438 (vw); Anal. Calcd for C<sub>7</sub>H<sub>8</sub>N<sub>2</sub>O: C, 61.75; H, 5.92; N, 20.58%. Found: C, 60.83; H, 5.83; N, 21.04%.



1-(4-Chlorophenyl) urea (**7b**)

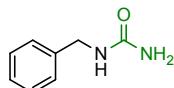
White solid (0.123 gr, 72% yield), m.p. 210 °C (Literature report: 208-210 °C); <sup>24</sup> <sup>1</sup>H NMR (DMSO-*d*<sub>6</sub>, 250 MHz, 25 °C): δ (ppm) 8.65 (s, NH), 7.41 (d, *J* = 8.0 Hz, 2H, aromatic ring), 7.22 (d, *J* = 8.0 Hz, 2H, aromatic ring), 5.90 (s, NH<sub>2</sub>); <sup>13</sup>C{H} NMR (DMSO-*d*<sub>6</sub>, 63 MHz, 25 °C): δ (ppm) 155.85, 139.43, 128.33, 124.51, 119.16; FT-IR (KBr, cm<sup>-1</sup>): 3420 (vs), 3289 (vs), 2874 (w), 1652 (vs), 1614 (vs), 1591 (vs), 1548 (vs), 1492 (m), 1401 (m), 1357 (s), 1296 (w), 1275 (m), 1251 (m), 1112 (vw), 1092 (s), 1014 (m), 870 (s), 820 (vs), 773 (m), 731 (s), 686 (w), 622 (w), 504 (s), 491 (s), 452 (vw); Anal. Calcd for C<sub>7</sub>H<sub>7</sub>ClN<sub>2</sub>O: C, 49.28; H, 4.14; N, 16.42 %. Found: C, 49.21; H, 4.16; N, 16.44 %.



1-(*p*-Tolyl) urea (**7c**)

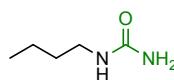
White solid (0.132 gr, 88% yield), m. p. 180-182 °C (Literature report: 182-184 °C); <sup>24</sup> <sup>1</sup>H NMR (DMSO-*d*<sub>6</sub>, 250 MHz, 25 °C): δ (ppm) 8.37 (s, NH), 7.25 (d, *J* = 8.5 Hz, 2H, aromatic ring), 6.99 (d, *J* = 8.5 Hz, 2H, aromatic ring), 5.76 (s, NH<sub>2</sub>), 2.18 (s, 3H, aromatic CH<sub>3</sub>); <sup>13</sup>C{H} NMR (DMSO-*d*<sub>6</sub>, 63 MHz, 25 °C): δ (ppm) 156.03, 137.89, 129.70, 128.92, 117.78, 20.22; FT-IR (KBr, cm<sup>-1</sup>): 3428 (vs), 3311 (vs), 3042 (m), 2919 (m), 1652 (vs), 1600 (vs), 1553 (vs), 1408 (m), 1357 (s), 1304 (w),

1280 (w), 1257 (m), 1110 (m), 1024 (m), 931 (vw), 871 (w), 825 (vw), 812 (vs), 779 (s), 708 (vw), 638 (vw), 551 (m), 502 (m), 484 (vw), 416 (vw); Anal. Calcd for C<sub>8</sub>H<sub>10</sub>N<sub>2</sub>O: C, 63.98; H, 6.71; N, 18.65 %. Found: C, 63.86; H, 6.75; N, 18.64 %.



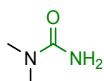
### 1-Benzylurea (**7d**)

White solid (0.134 gr, 89% yield), m. p. 143-146 °C (Literature report: 144-145 °C); <sup>25</sup> <sup>1</sup>H NMR (DMSO-d<sub>6</sub>, 250 MHz, 25 °C): δ(ppm) 7.28 (m, 5H, aromatic ring), 6.44 (s, NH), 4.17 (s, 2H, CH<sub>2</sub>), 5.56 (s, NH<sub>2</sub>); <sup>13</sup>C{H} NMR (DMSO-d<sub>6</sub>, 63 MHz, 25 °C): δ(ppm) 158.72, 140.82, 128.13, 126.43, 126.47, 42.75; FT-IR (KBr, cm<sup>-1</sup>): 3429 (s), 3321 (s), 3031 (m), 2932 (m), 2881 (m), 1652 (s), 1594 (s), 1565 (s), 1468 (m), 1457 (m), 1383 (m), 1328 (v), 1312 (m), 1208 (vw), 1143 (m), 1110 (w), 1026 (w), 912 (vw), 750 (m), 696 (s), 585 (s), 547 (vw), 464 (m); Anal. Calcd for C<sub>8</sub>H<sub>10</sub>N<sub>2</sub>O: C, 43.98; H, 6.71; N, 18.65 %. Found: C, 43.83; H, 6.63; N, 18.74 %.



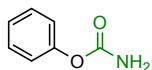
### 1-Butylurea (**7e**)

White solid (0.114 gr, 98% yield), m.p. 97°C (Literature report: 95-96.5 °C); <sup>26</sup> <sup>1</sup>H NMR (DMSO-d<sub>6</sub>, 250 MHz, 25 °C): δ(ppm) 6.57 (s, NH), 6.00 (s, NH<sub>2</sub>), 3.51 (t, J = 6.2 Hz, 2H, CH<sub>2</sub>), 1.89 (m, 4H, CH<sub>2</sub>), 1.45 (t, J = 7.5 Hz, 3H, CH<sub>3</sub>); <sup>13</sup>C{H} NMR (DMSO-d<sub>6</sub>, 63 MHz, 25 °C): δ(ppm) 158.84, 38.75, 32.00, 19.44, 13.61; FT-IR (KBr, cm<sup>-1</sup>): 3430 (vs), 3355 (vs), 2960 (vs), 2936 (vs), 2872 (vs), 1649 (vs), 1602 (vs), 1566 (vs), 1478 (m), 1461 (m), 1389 (w), 1362 (s), 1328 (s), 1265 (m), 1158 (s), 1125 (w), 1060 (vw), 996 (vw), 780 (m), 744 (m), 660 (vw), 653 (vs), 434 (m); Anal. Calcd for C<sub>5</sub>H<sub>12</sub>N<sub>2</sub>O: C, 51.70; H, 10.41; N, 24.12 %. Found: C, 51.61; H, 10.45; N, 24.04 %.



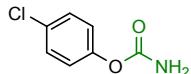
### 1,1-Dimethylurea (**7f**)

White solid (0.079 gr, 90% yield), m. p. 178-181 °C (Literature report: 181.05 °C); <sup>27</sup> <sup>1</sup>H NMR (DMSO-d<sub>6</sub>, 250 MHz, 25 °C), δ(ppm) 5.74 (s, NH<sub>2</sub>), 2.73 (s, 6H, CH<sub>3</sub>); <sup>13</sup>C{H} NMR (DMSO-d<sub>6</sub>, 63 MHz, 25 °C): δ(ppm) 159.01, 35.85; FT-IR (KBr, cm<sup>-1</sup>): 3407 (s), 3204 (s), 2934 (m), 2871 (vw), 1650 (s), 1611 (s), 1513 (s), 1407 (s), 1277 (m), 1180 (w), 1102 (m), 1072 (m), 1027 (m), 876 (vw), 775 (s), 722 (m), 606 (s), 556 (s); Anal. Calcd for C<sub>3</sub>H<sub>8</sub>N<sub>2</sub>O: C, 40.90; H, 9.15; N, 31.79 %. Found: C, 40.83; H, 9.19; N, 31.84 %.



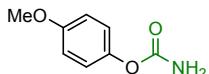
### Phenyl carbamate (**7g**)

White solid (0.110 gr, 80% yield), m. p. 143-144°C (Literature report: 145.1 °C); <sup>28</sup> <sup>1</sup>H NMR (DMSO-d<sub>6</sub>, 250 MHz, 25 °C): δ(ppm) 7.35 (t, J = 7.5 Hz, 2H, aromatic ring), 7.17 (t, J = 7.5 Hz, 1H, aromatic ring), 7.07 (d, J = 7.5 Hz, 2H, aromatic ring), 6.89 (s, NH<sub>2</sub>); <sup>13</sup>C{H} NMR (DMSO-d<sub>6</sub>, 63 MHz, 25 °C): δ(ppm) 154.76, 151.02, 129.11, 124.77, 121.88; FT-IR (KBr, cm<sup>-1</sup>): 3410 (s), 3335 (s), 3271 (s), 3189 (s), 3071 (s), 1707 (vs), 1614 (s), 1486 (s), 1372 (vs), 1201 (vs), 1162 (s), 1069 (m), 1003 (m), 1022 (m), 973 (vs), 915 (w), 838 (m), 762 (s), 696 (vs), 585 (s); Anal. Calcd for C<sub>7</sub>H<sub>7</sub>NO<sub>2</sub>: C, 61.31; H, 5.14; N, 10.21%. Found: C, 61.28; H, 5.16; N, 10.32%.



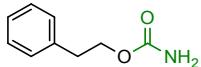
**4-Chlorophenyl carbamate (7h)**

White solid (0.113 gr, 66% yield), m. p. 167-168 °C (Literature report: 165-166 °C); <sup>29</sup> <sup>1</sup>H NMR (DMSO-d<sub>6</sub>, 250 MHz, 25 °C): δ(ppm) 6.99 (brs, NH), 7.33 (d, J = 7.5 Hz, 2H, aromatic ring), 7.26 (brs, NH), 7.42 (d, J = 7.5 Hz, 2H, aromatic ring); <sup>13</sup>C{H} NMR (DMSO-d<sub>6</sub>, 63 MHz, 25 °C): δ(ppm) 154.38, 149.82, 129.00, 128.84, 123.75; FT-IR (KBr, cm<sup>-1</sup>): 3413 (m), 3334 (w), 3272 (m), 3192 (w), 1706 (vs), 1595 (s), 1478 (s), 1365 (s), 1225 (vs), 1097 (s), 1013 (m), 970 (vs), 859 (m), 827 (s), 763 (m), 736 (m), 501 (m); Anal. Calcd for C<sub>7</sub>H<sub>6</sub>ClNO<sub>2</sub>: C, 49.00; H, 3.52; N, 8.16%. Found: C, 48.67; H, 3.58; N, 8.21%.



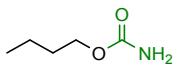
**4-Methoxyphenyl carbamate (7i)**

White solid (0.140 gr, 84% yield), m. p. 129-130 °C (Literature report: 127.3-129 °C); <sup>30</sup> <sup>1</sup>H NMR (DMSO-d<sub>6</sub>, 250 MHz, 25 °C): δ(ppm) 7.08 (brs, NH), 6.99 (d, J = 9 Hz, 2H, aromatic ring), 6.87 (brs, NH and d, J = 9 Hz, 2H, aromatic ring), 3.71 (s, 3H, OCH<sub>3</sub>); <sup>13</sup>C{H} NMR (DMSO-d<sub>6</sub>, 63 MHz, 25 °C): δ(ppm) 156.18, 155.16, 144.45, 122.72, 114.05, 55.27; FT-IR (KBr, cm<sup>-1</sup>): 3410 (s), 3340 (s), 3270 (s), 3208 (s), 3065 (vw), 3016 (vw), 2974 (m), 2938 (m), 2844 (m), 1764 (w), 1714 (vs), 1624 (w), 1595 (vw), 1507 (s), 1459 (m), 1445 (w), 1376 (vs), 1298 (m), 1246 (s), 1206 (vs), 1186 (s), 1127 (w), 1102 (m), 1030 (s), 1008 (m), 978 (s), 954 (m), 933 (vw), 848 (s), 821 (s), 784 (m), 765 (m), 726 (w), 705 (w), 670 (w), 564 (s), 524 (s), 419 (w); Anal. Calcd for C<sub>8</sub>H<sub>9</sub>NO<sub>3</sub>: C, 57.48; H, 5.43; N, 8.38%. Found: C, 57.41; H, 5.47; N, 8.45%.



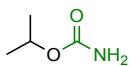
**Phenethyl carbamate (7j)**

White solid (0.142 gr, 86% yield), m. p. 94-97 °C (Literature report: 93-95 °C); <sup>31</sup> <sup>1</sup>H NMR (DMSO-d<sub>6</sub>, 250 MHz, 25 °C): δ(ppm) 7.16-7.32 (m, 5H, aromatic ring), 6.46 (brs, NH<sub>2</sub>), 4.10 (t, J = 7.5 Hz, 2H, CH<sub>2</sub>), 2.83 (t, J = 7.5 Hz, 2H, CH<sub>2</sub>); <sup>13</sup>C{H} NMR (DMSO-d<sub>6</sub>, 63 MHz, 25 °C): δ(ppm) 134.87, 123.89, 126.17, 128.25, 128.77, 138.26, 156.65; FT-IR (KBr, cm<sup>-1</sup>): 3429 (s), 2965(m), 1694 (vs), 1412(vs), 1343 (vs), 1240 (m), 1079(s), 1046 (m), 752 (s), 702 (vs), 645 (m), 570 (m), 497 (m); Anal. Calcd for C<sub>9</sub>H<sub>11</sub>NO<sub>2</sub>: C, 65.44; H, 6.71; N, 8.48%. Found: C, 65.40; H, 6.68; N, 8.48%.



**n-Butyl carbamate (7k)**

White solid (0.011 gr, 93% yield), m. p. 54-55 °C (Literature report: 55 °C); <sup>32</sup> <sup>1</sup>H NMR (DMSO-d<sub>6</sub>, 250 MHz, 25 °C): δ(ppm) 6.38 (brs, NH<sub>2</sub>), 3.87 (t, J = 5.0 Hz, 2H, CH<sub>2</sub>), 1.50 (m, 2H, CH<sub>2</sub>), 1.28 (m, 2H, CH<sub>2</sub>), 0.86 (t, J = 7.5 Hz, 3H, CH<sub>3</sub>); <sup>13</sup>C{H} NMR (DMSO-d<sub>6</sub>, 63 MHz, 25 °C): δ(ppm) 156.83, 62.91, 30.69, 18.54, 13.51; FT-IR (KBr, cm<sup>-1</sup>): 3417 (m), 3200 (m), 2962 (m), 2874 (m), 1696 (s), 1435 (m), 1339 (m), 1254 (m), 1121 (w), 1080 (vs), 943 (m), 887 (vw), 788 (m), 740 (w), 638 (s), 556 (s), 438 (m); Anal. Calcd for C<sub>5</sub>H<sub>11</sub>NO<sub>2</sub>: C, 51.26; H, 9.46; N, 11.96%. Found: C, 51.18; H, 9.48; N, 12.05%.



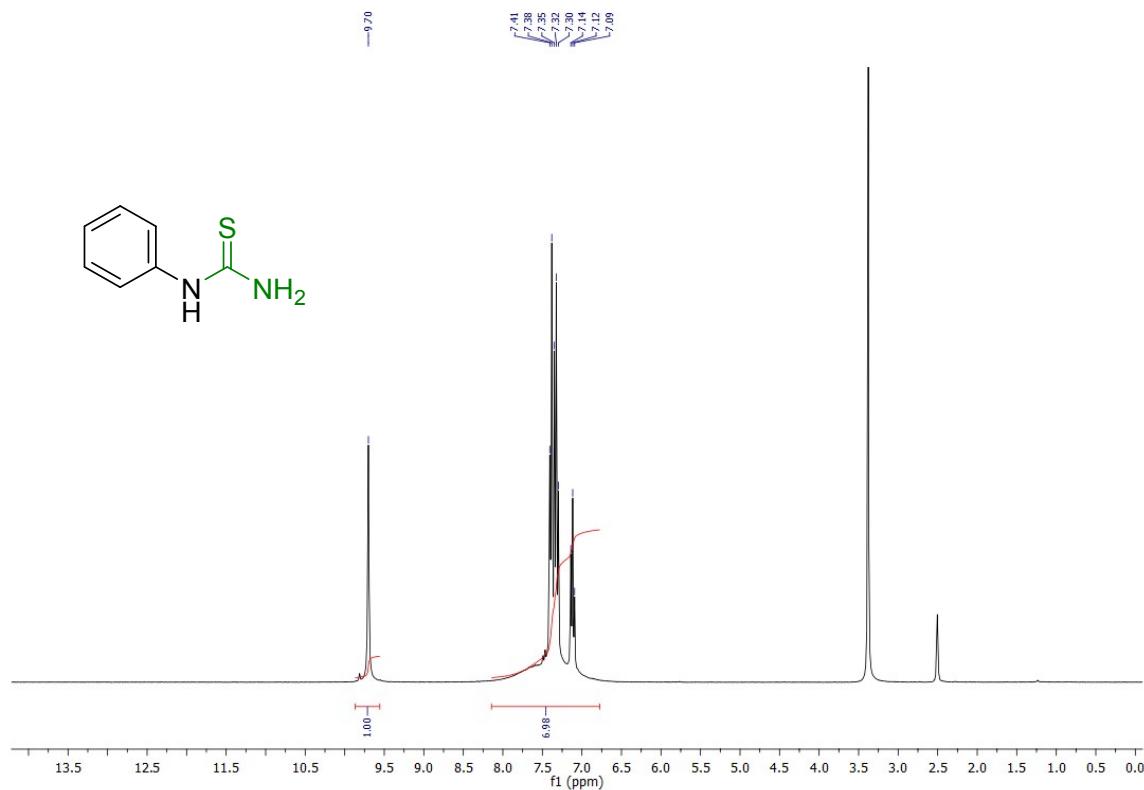
**Isopropyl carbamate (7l)**

White solid (0.086 gr, 83% yield), m. p. 89–90 °C (Literature report: 89–93 °C);  $^{33}\text{H}$  NMR (DMSO- $d_6$ , 250 MHz, 25 °C):  $\delta$ (ppm) 6.33 (brs, NH<sub>2</sub>), 4.67 (h,  $J$  = 6.3 Hz, 1H, CH), 1.12 (d,  $J$  = 6.3 Hz, 6H, CH<sub>3</sub>);  $^{13}\text{C}\{\text{H}\}$  NMR (DMSO- $d_6$ , 63 MHz, 25 °C):  $\delta$ (ppm): 156.7, 66.0, 22.0; FT-IR (KBr, cm<sup>-1</sup>): 3429 (vw), 3218 (v), 2985 (m), 2985 (s), 1684 (s), 1417 (s), 1320 (s), 1108 (s), 1046 (s), 901 (m), 825 (m), 793 (s), 600 (vs), 460 (m), 412 (m); Anal. Calcd for C<sub>4</sub>H<sub>9</sub>NO<sub>2</sub>: C, 46.59; H, 8.80; N, 13.58%. Found: C, 46.58; H, 8.78; N, 13.65%.

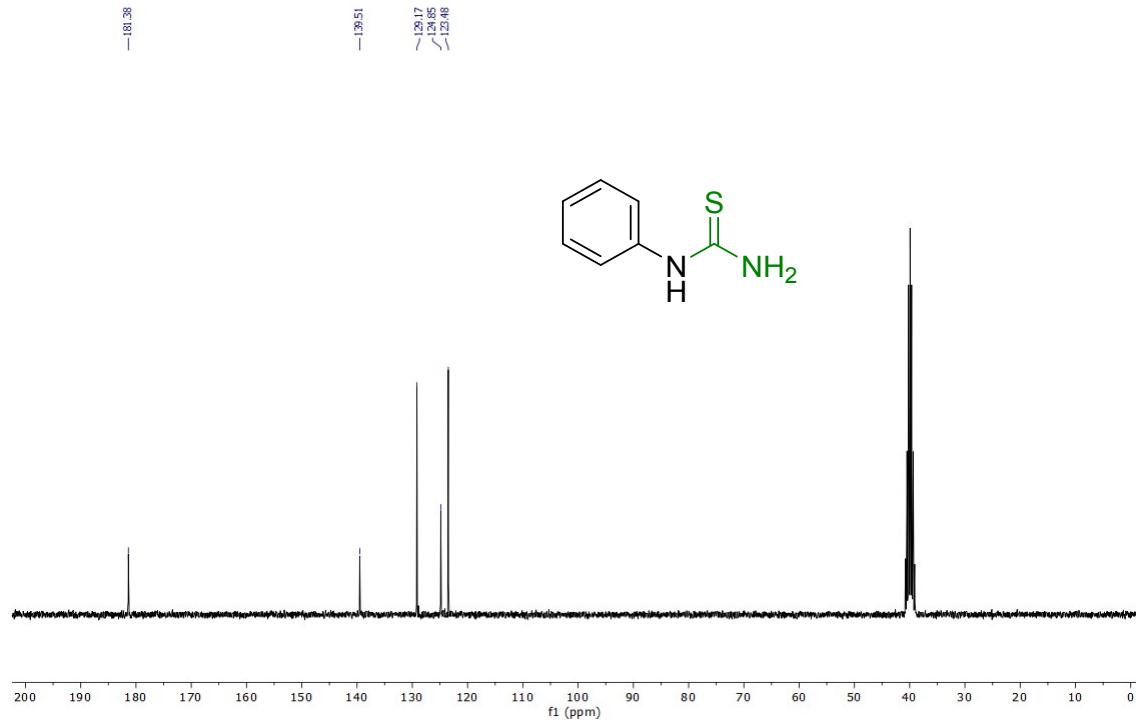
## References

1. C.D. Pawar, S. L. Chavan, U. D. Pawar, D. N. Pansare, S. V. Deshmukh and D. B. Shinde, *J. Chin. Chem. Soc.*, 2019, **66**, 257–264.
2. M. P. Hay, S. Turcotte, J. U. Flanagan, M. Bonnet, D. A. Chan, P. D. Sutphin, p. Nguyen, A. J. Giaccia and W. A. J. Denny, *Med. Chem.*, 2010, **53**, 787–797.
3. S. Grosjean, S. Trikib, J.C. Meslina, K. Julienne, D. Deniaud. *Tetrahedron*, 2010, **66**, 9912–9924.
4. Z. Ngaini, W. S. H. Wan Zulkiplee and A. N. Abd Halim, *J. Chem.*, 2017.
5. A. K. Verma, A. Bishnoi, S. Fatma, H. Parveen and V. Singh. *Drug Res.*, 2018, **68**, 222–231.
6. P. D. Zalavadiya, R. M. Ghetiya, B. L. Dodiya, P. B. Vekariya and H. S. Joshi, *J. Heterocycl. Chem.*, 2013, **50**, 973–978.
7. J. L. Woodring, S. H. Lu, L. Krasnova, S. C. Wan, J. B. Chen, C. C. Chou, Y. C. Huan, T. J. Rachel Cheng, Y. T. Wu, Y. H. Chen, J. M. Fang, M. D. Tsai and C. H. Wong, *J. Med. Chem.*, 2020, **63**, 205–215.
8. G. M. Dyson, H. J. George and R. F. Hunter, *J. Chem. Soc.*, 1926, **129**, 3041–3044.
9. G. M. Dyson and H. J. George, *J. Chem. Soc., Trans.* 1924, **125**, 1702–1708.
10. R. V. Patel, S. W. Park, *Eur. J. Med. Chem.*, 2014, **71**, 24–30.
11. R. G. Neville and J. J. McGee, *Can. J. Chem.*, 1963, **41**, 2123–2129.
12. G. V. Nair, *Indian J. Chem.*, 1966, **4**, 516–520
13. M. M. Aly, A. M. Fahmy and A. M. Gaber, 1990, **53**, 253–258.
14. J. Kosary, E. Kasztreiner, F. Andras, *Die Pharmazie*, 1989, **44**, 191–193.
15. J. Fatimi, J. F. Lagorce, J. L. Duroux, M. L. Chabernaud, J. Buxeraud and C. Raby, *Chem. Pharm. Bull.*, 1994, **42**, 698–701.
16. M. Delepine and P. Schving, *Bull. Soc. Chim.*, 1910, **7**, 895–901.
17. M. Battegay and E. Hegazi, *Helv. Chim. Acta*, 1933, **16**, 999–1008.
18. C. Weibull, *Biochim. Biophys. Acta*, 1948, **2**, 351–361.
19. K. A. Jensen, A. Holm and J. Wolffjen, *Acta Chem. Scand.*, 1969, **23**, 1567.
20. O. Ponomarov, Z. Padalkova and J. Hanusek, *J. Heterocycl. Chem.*, 2011, **48**, 1225–1228.
21. W. Walter and K. D. Bode, *Justus Liebigs Ann. Chem.*, 1965, **681**, 64–84.
22. A. R. Modarresi-Alam, I. D. Inaloo and E. Kleinpeter, *J. Mol. Struct.*, 2012, **1024**, 156–162.
23. F. Peudru, F. Le Cavelier, J. F. Lohier, M. Gulea and V. Reboul, *Org. Lett.*, 2013, **15**, 5710–5715.
24. D. Habibi, S. Heydari, A. Faraji, H. Keypour and M. Mahmoudabadi, *Polyhedron*, 2018, **151**, 520–529.
25. C. H. Wang, T. H. C. C. Hiseh, C. C. Lin, W. H. Yeh, C. A. Lin and T. C. Chien, *Synlett*, 2015, **26**, 1823–1826.
26. D. M. Manidhar, K. Uma Maheswara Rao, C. Suresh Reddy, Ch. Syamasunder, K. Adeppa and K. Misra, *Res. Chem. Intermed.*, 2012, **9**, 2479–2489.
27. D. Ferro, G. Barone, G. Della Gatta and V. Piacente, *J. Chem. Thermodyn.*, 1987, **19**, 915–923.
28. S. Wishkerman and J. Bernstein, *Chem. Erup. J.*, 2008, **14**, 197–203.
29. E. Grigat and R. Puetter, *Chem. Ber.*, 1966, **99**, 958–975.
30. Y. Liang, M.L. Tang, Z. Huo, C. Zhang and X. Sun, *Molecules*, 2020, **25**, 1138.
31. N. Hen, M. Bialer and B. Yagen, *J. Med. Chem.*, 2012, **55**, 2835–2845.
32. P. Wang, Sh. Liu, L. Lu, X. Ma, Y. He and Y. Deng, *RSC Adv.*, 2015, **5**, 62110–62115.
33. V. Valenta, M. Bartosova and M. Protiva, *Collect. Czech. Chem. Commun.*, 1980, **45**, 517–528.

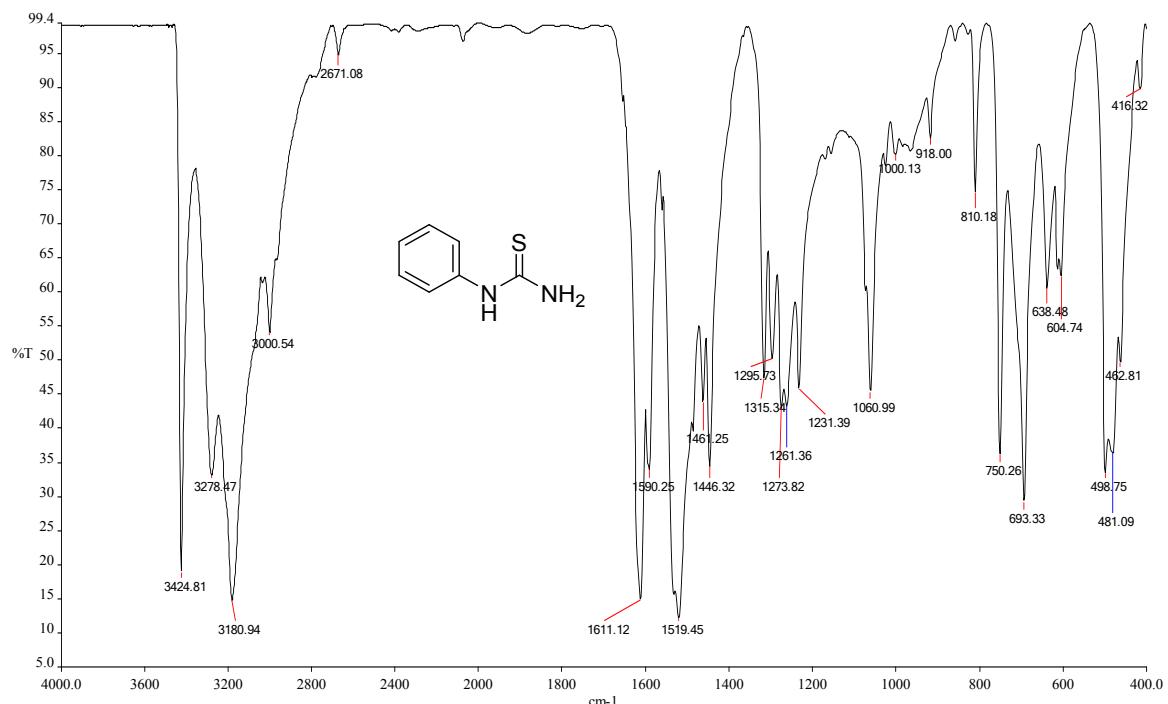
## Spectral of Products



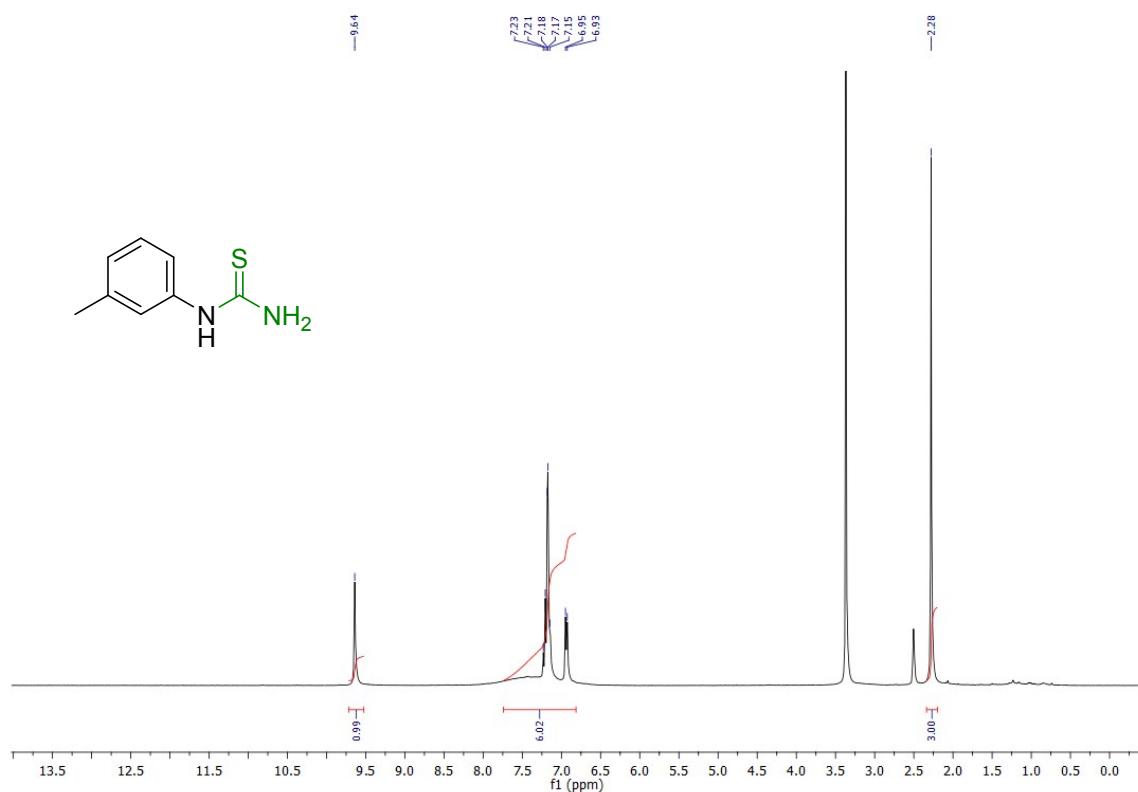
**Figure S1.**  $^1\text{H}$  NMR spectrum of 3a in  $\text{DMSO}-d_6$  (300MHz)

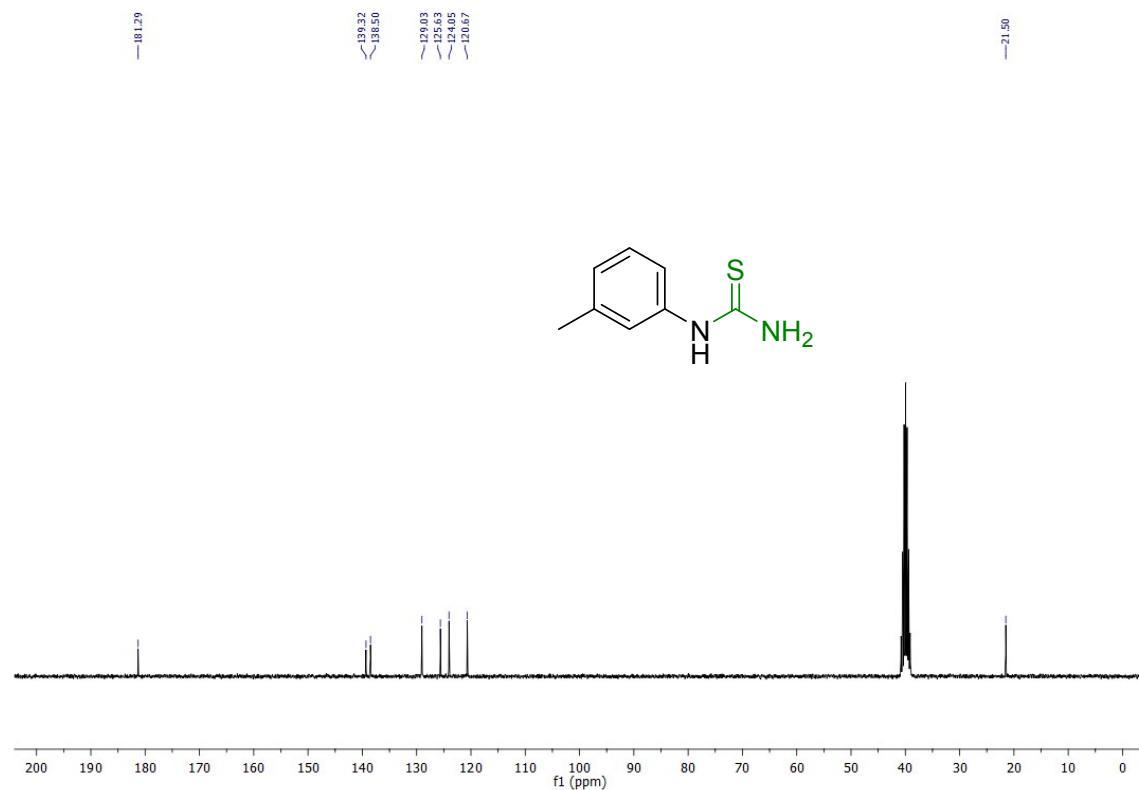


**Figure S2.**  $^{13}\text{C}$  NMR spectrum of 3a in  $\text{DMSO}-d_6$  (75MHz)

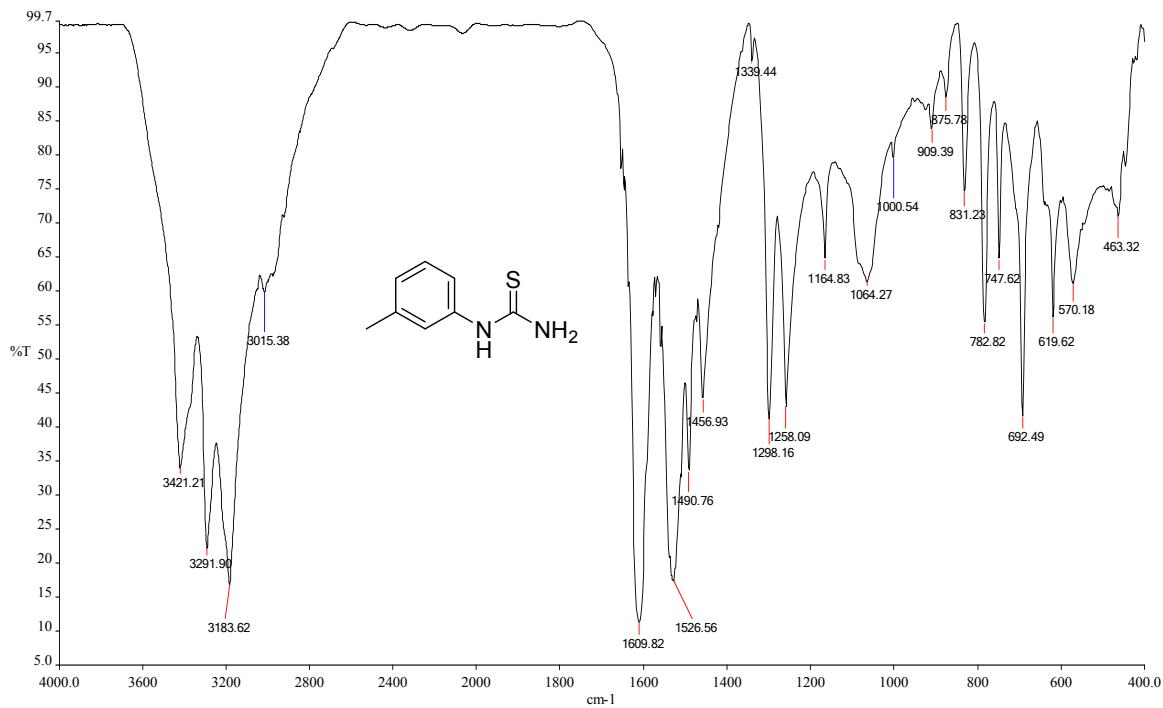


**Figure S3.** FT-IR spectrum of 3a in KBr

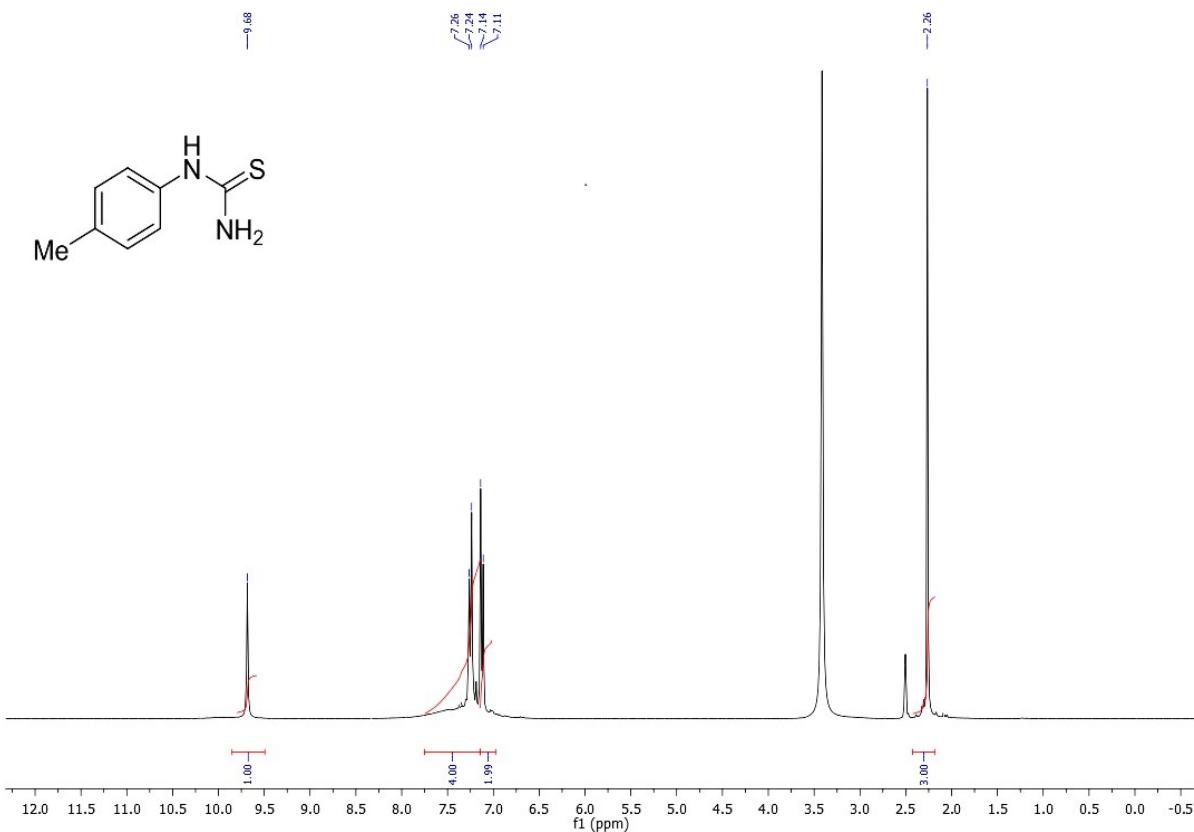




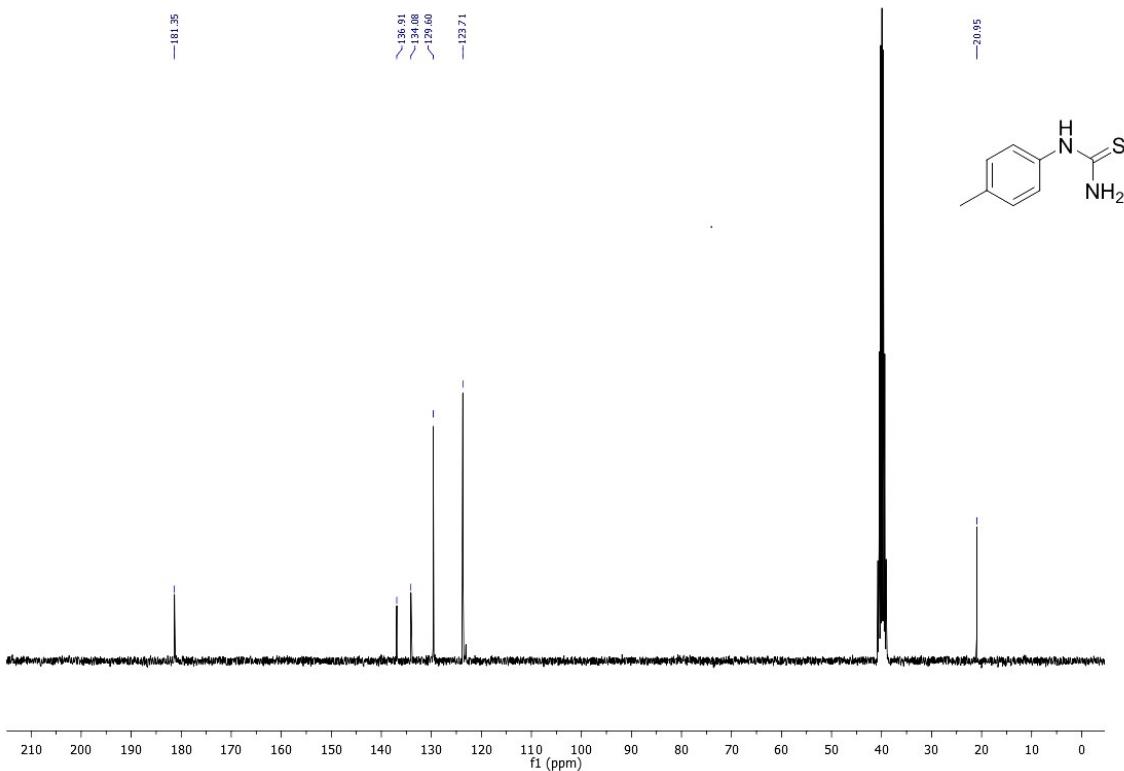
**Figure S5.**  $^{13}\text{C}$  NMR spectrum of **3b** in  $\text{DMSO}-d_6$  (75MHz)



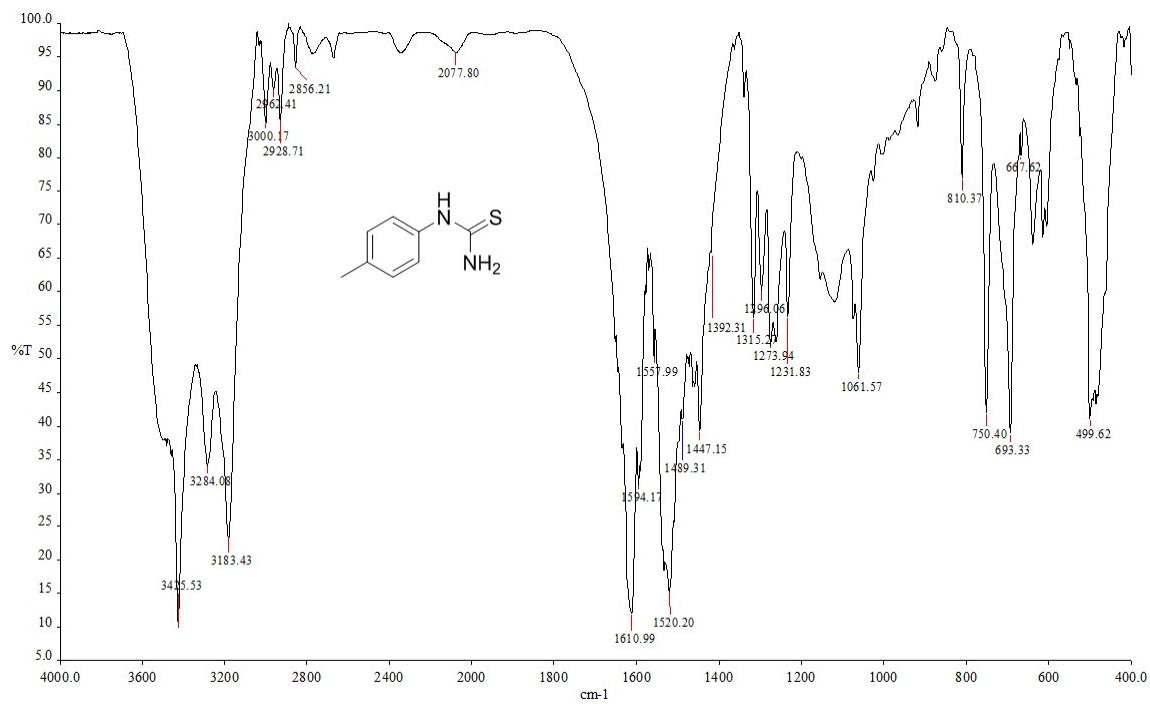
**Figure S6.** FT-IR spectrum of **3b** in KBr



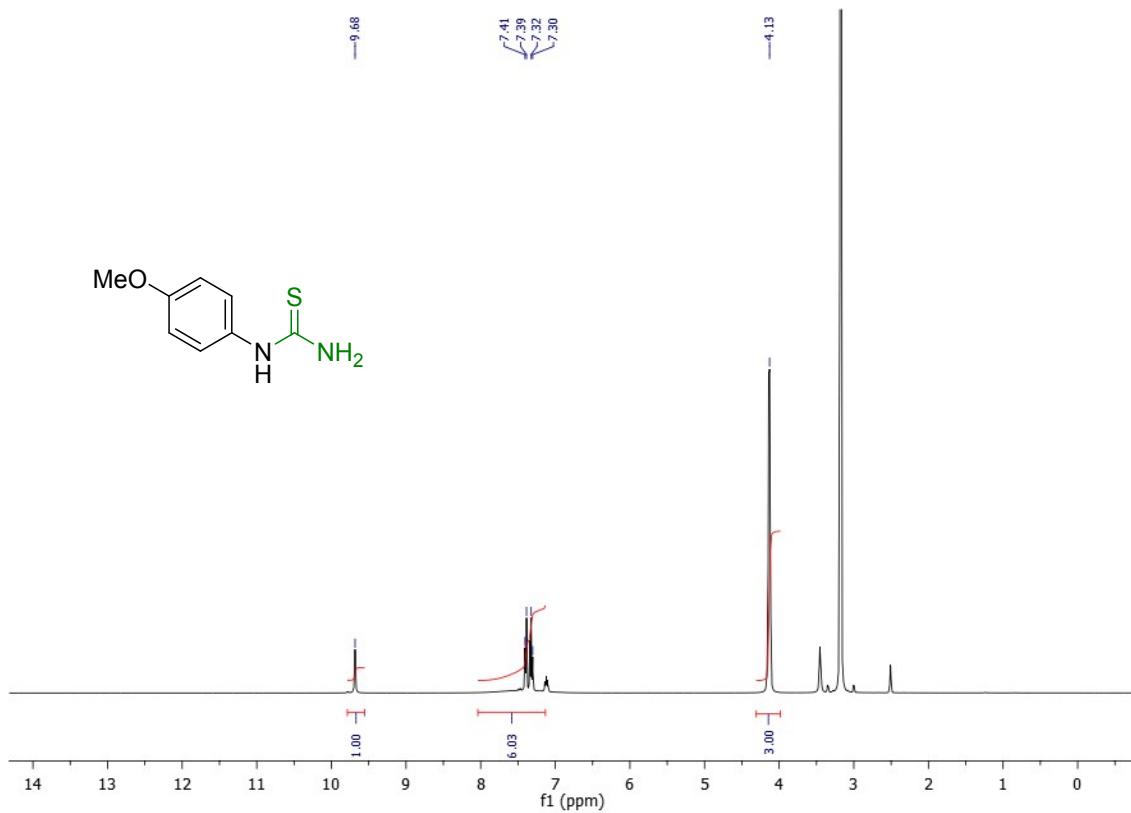
**Figure S7.** <sup>1</sup>H NMR spectrum of 3c in DMSO-*d*<sub>6</sub> (300MHz)



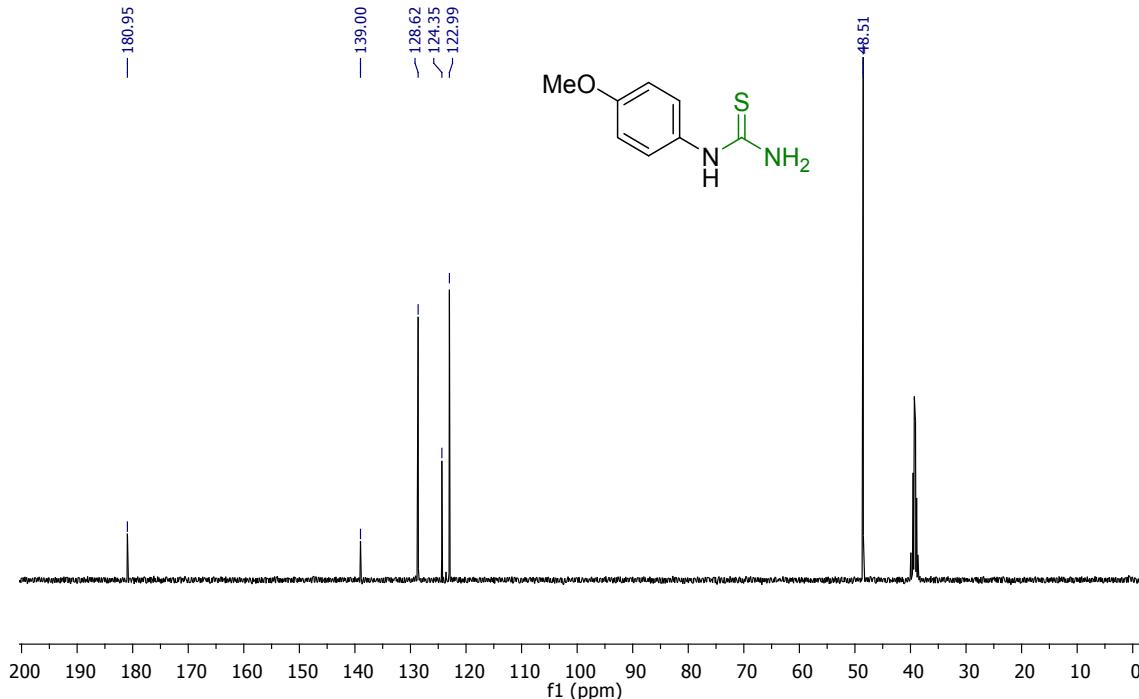
**Figure S8.** <sup>13</sup>C NMR spectrum of 3C in DMSO-*d*<sub>6</sub> (75MHz)



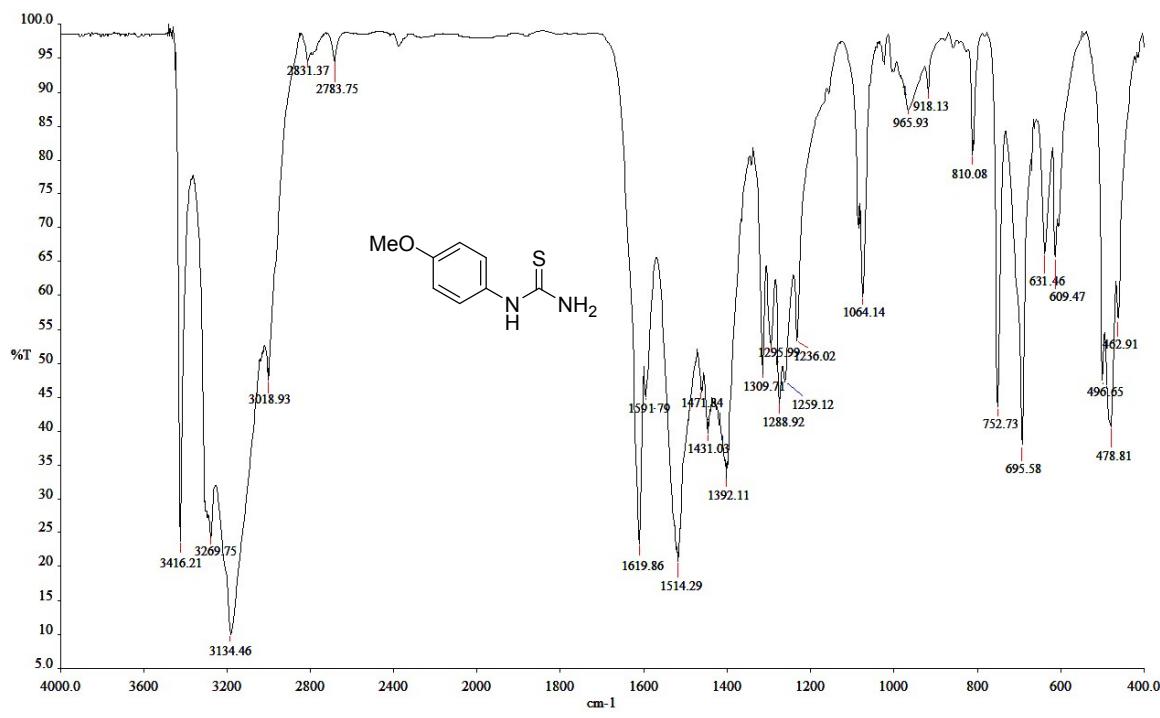
**Figure S9.** FT-IR spectrum of 3C in KBr



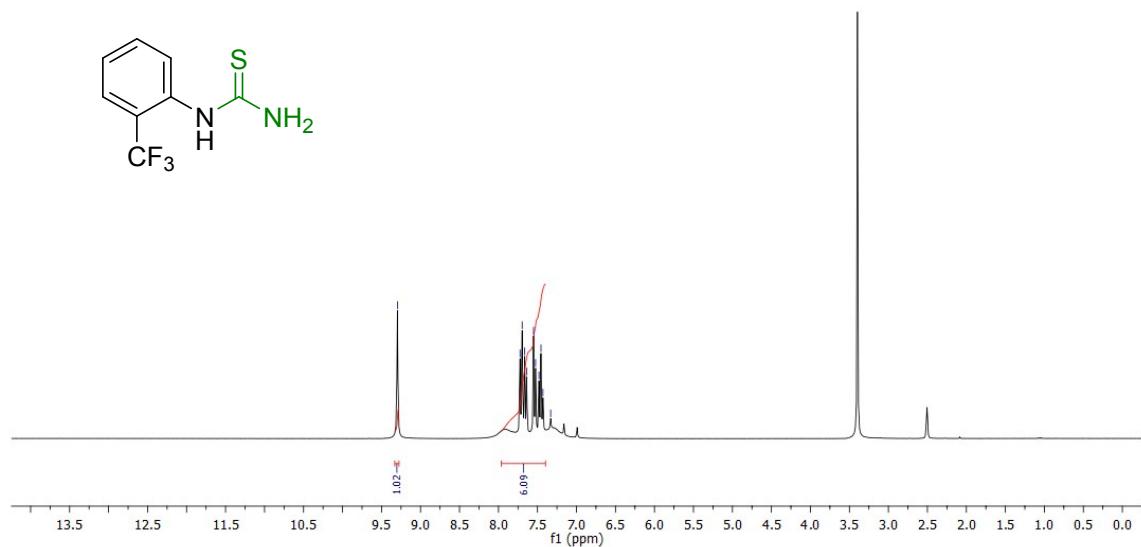
**Figure S10.**  $^1\text{H}$  NMR spectrum of 3d in  $\text{DMSO}-d_6$  (400MHz)



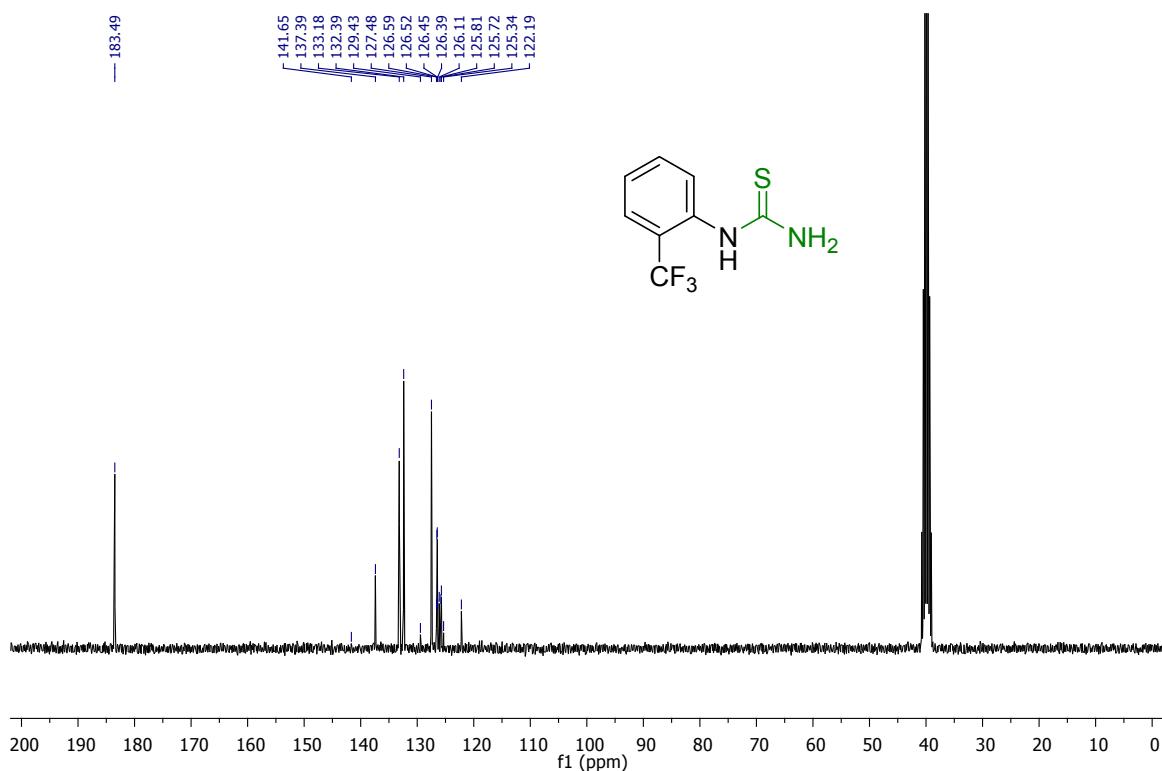
**Figure S11.**  $^{13}\text{C}$  NMR spectrum of 3d in  $\text{DMSO}-d_6$  (101MHz)



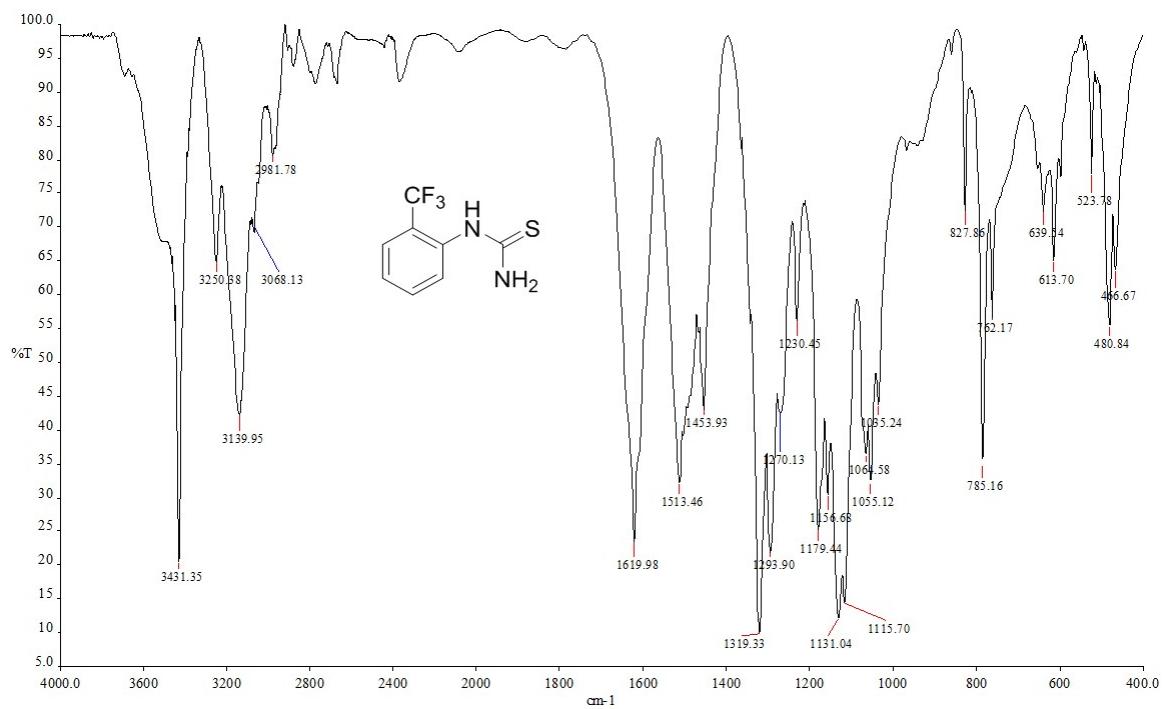
**Figure S12.** FT-IR spectrum of 3d in KBr



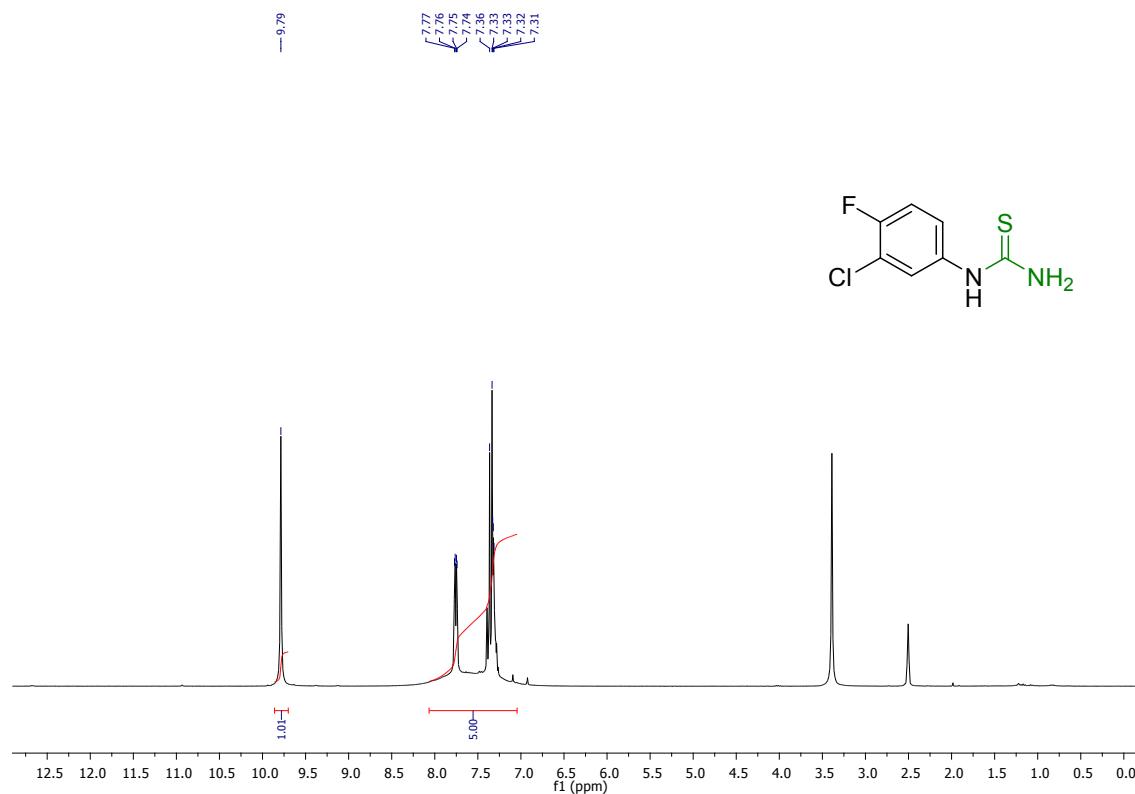
**Figure S13.**  $^1\text{H}$  NMR spectrum of 3e in  $\text{DMSO}-d_6$  (300MHz)



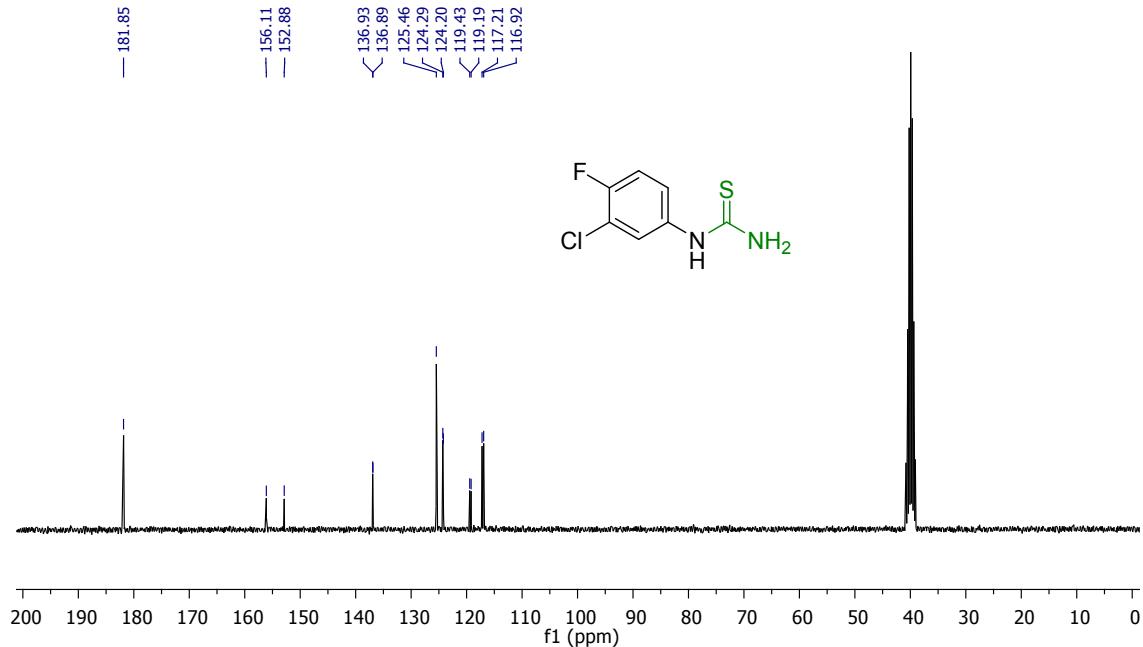
**Figure S14.**  $^{13}\text{C}$  NMR spectrum of 3e in  $\text{DMSO}-d_6$  (75MHz)



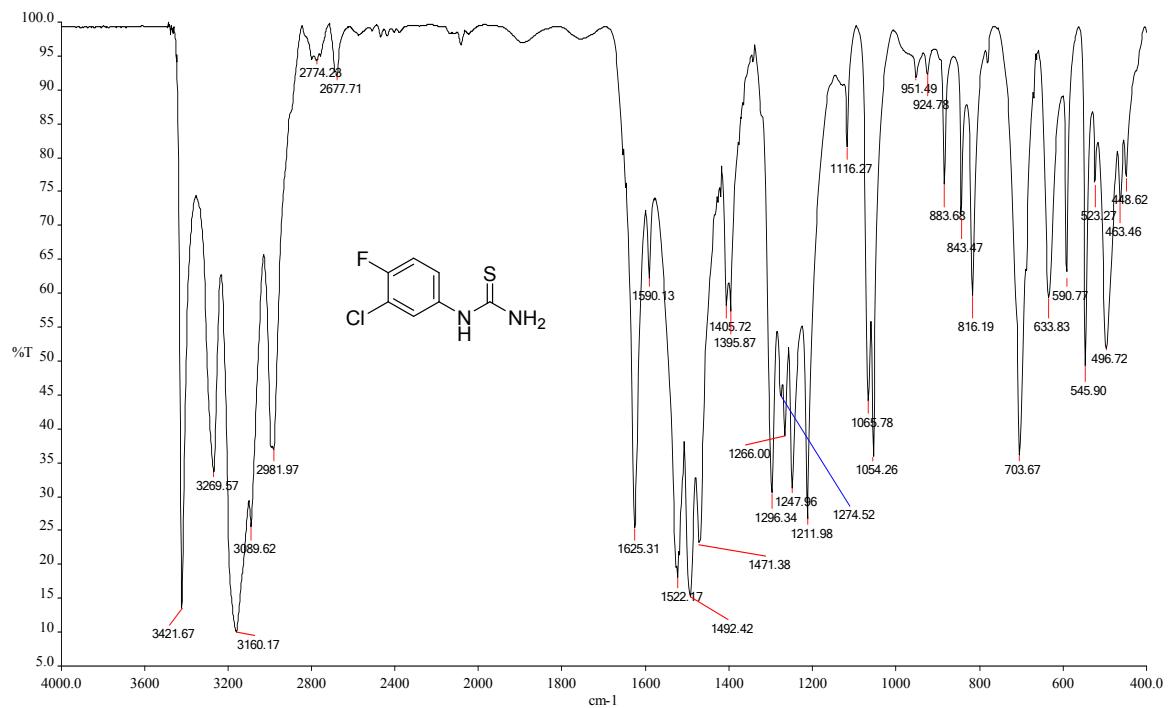
**Figure S15.** FT-IR spectrum of **3e** in KBr



**Figure S16.**  $^1\text{H}$  NMR spectrum of **3f** in  $\text{DMSO}-d_6$  (300MHz)



**Figure S17.** <sup>13</sup>C NMR spectrum of 3f in DMSO-d<sub>6</sub> (75MHz)



**Figure S18.** FT-IR spectrum of 3f in KBr

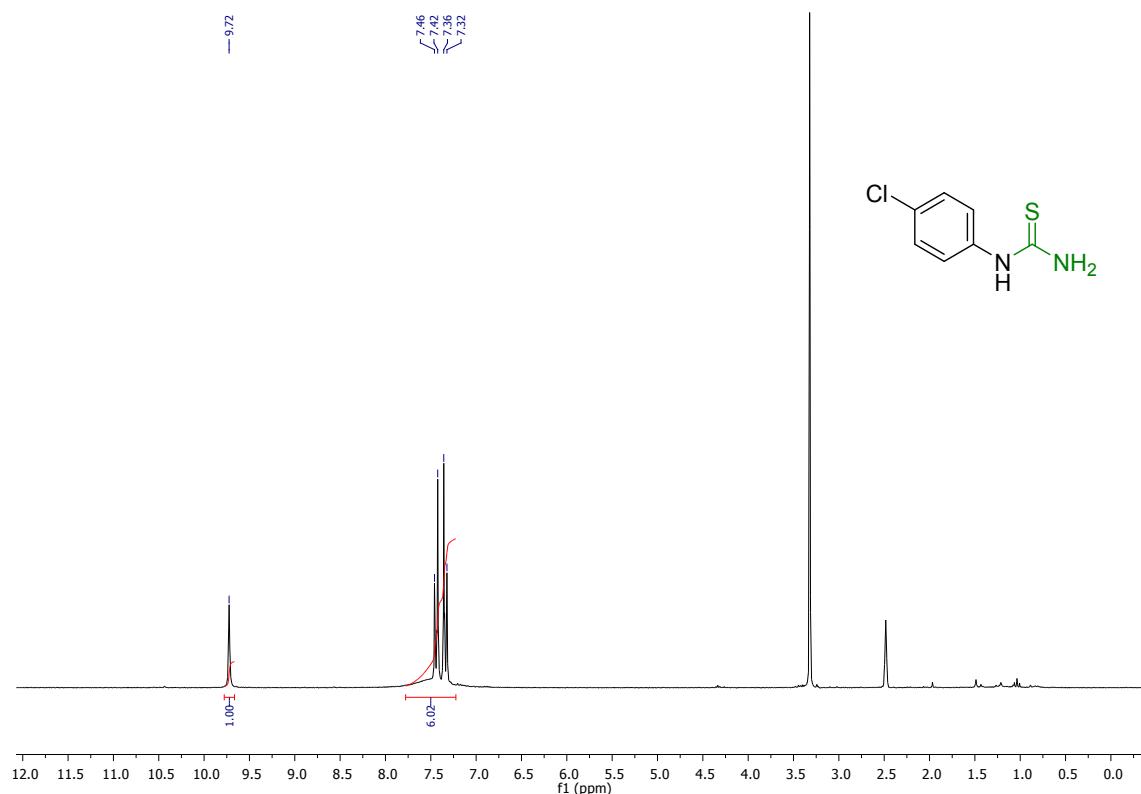


Figure S19. <sup>1</sup>H NMR spectrum of 3g in DMSO-*d*<sub>6</sub> (250MHz)

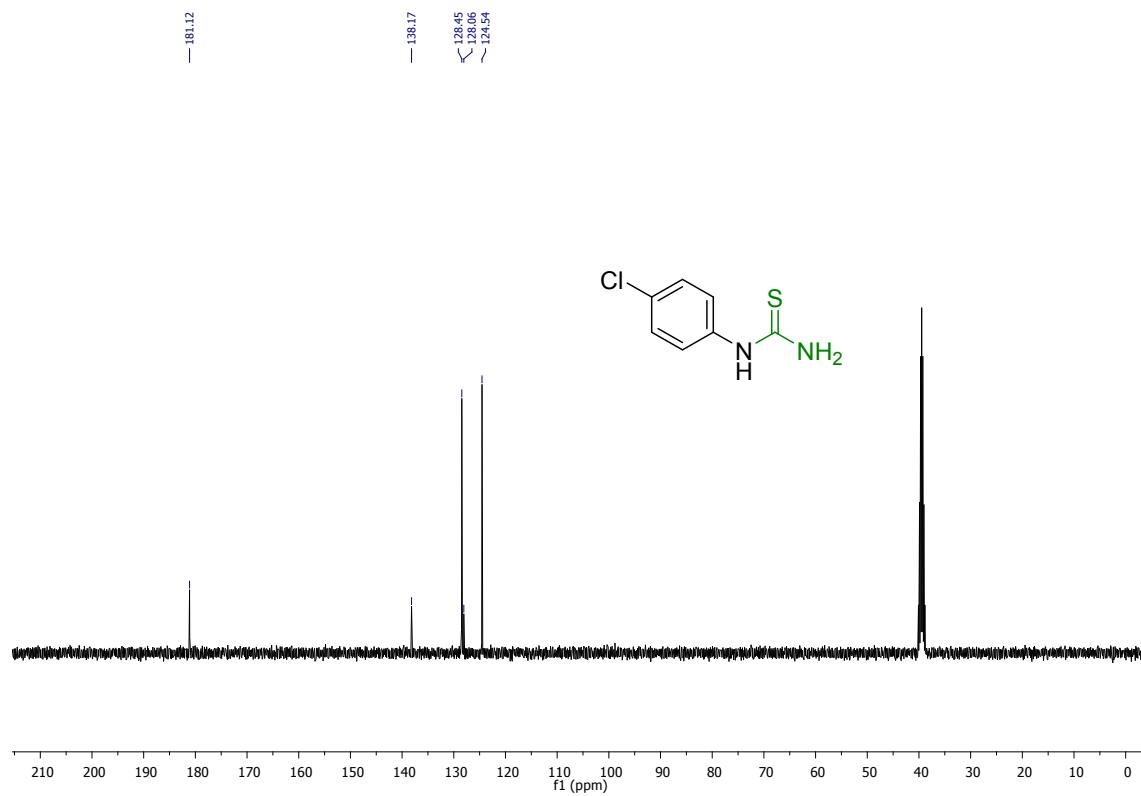
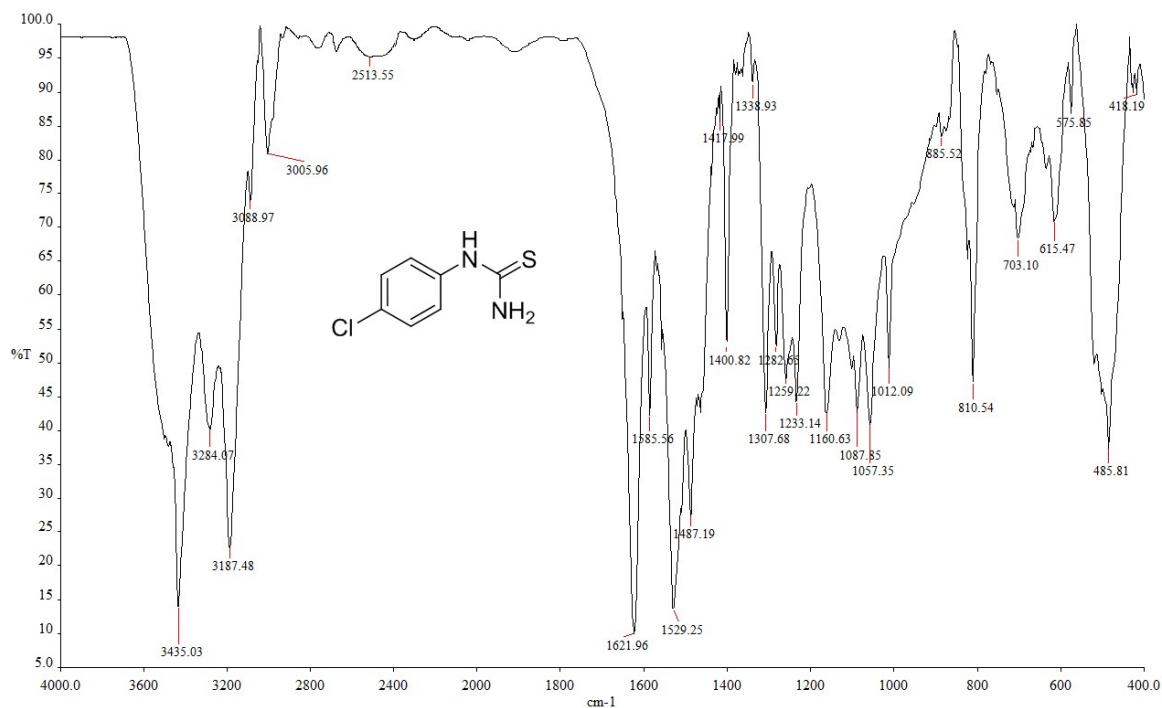
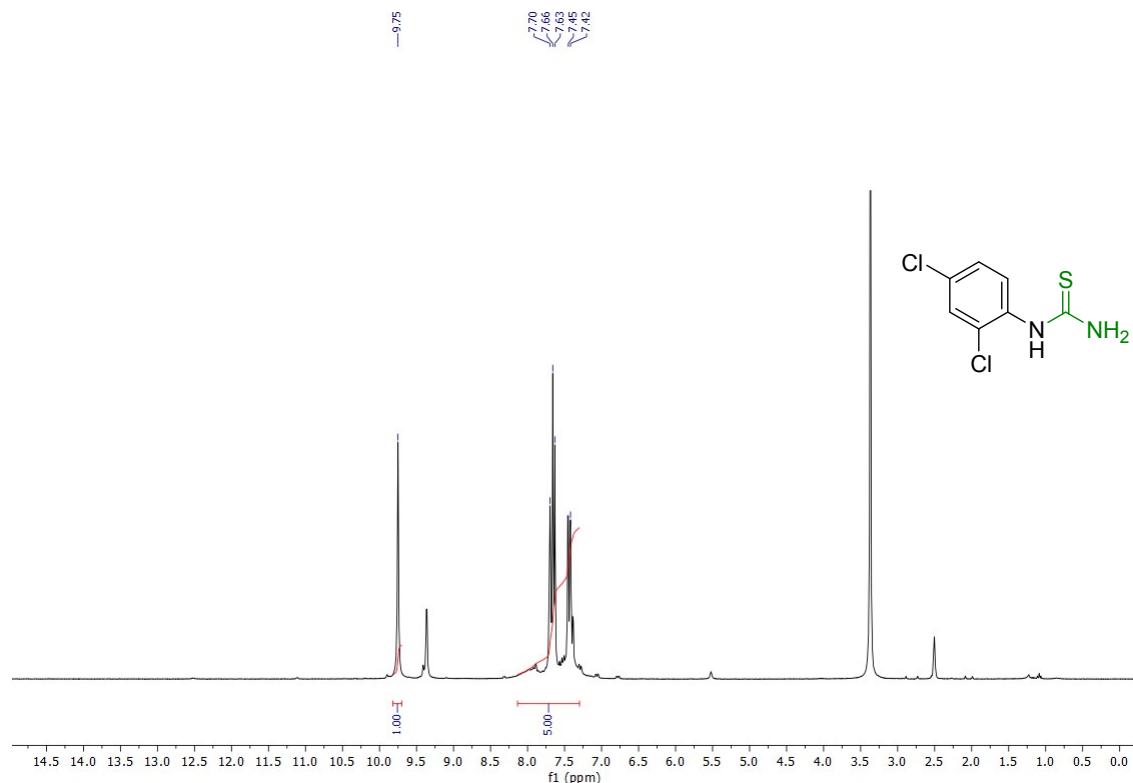


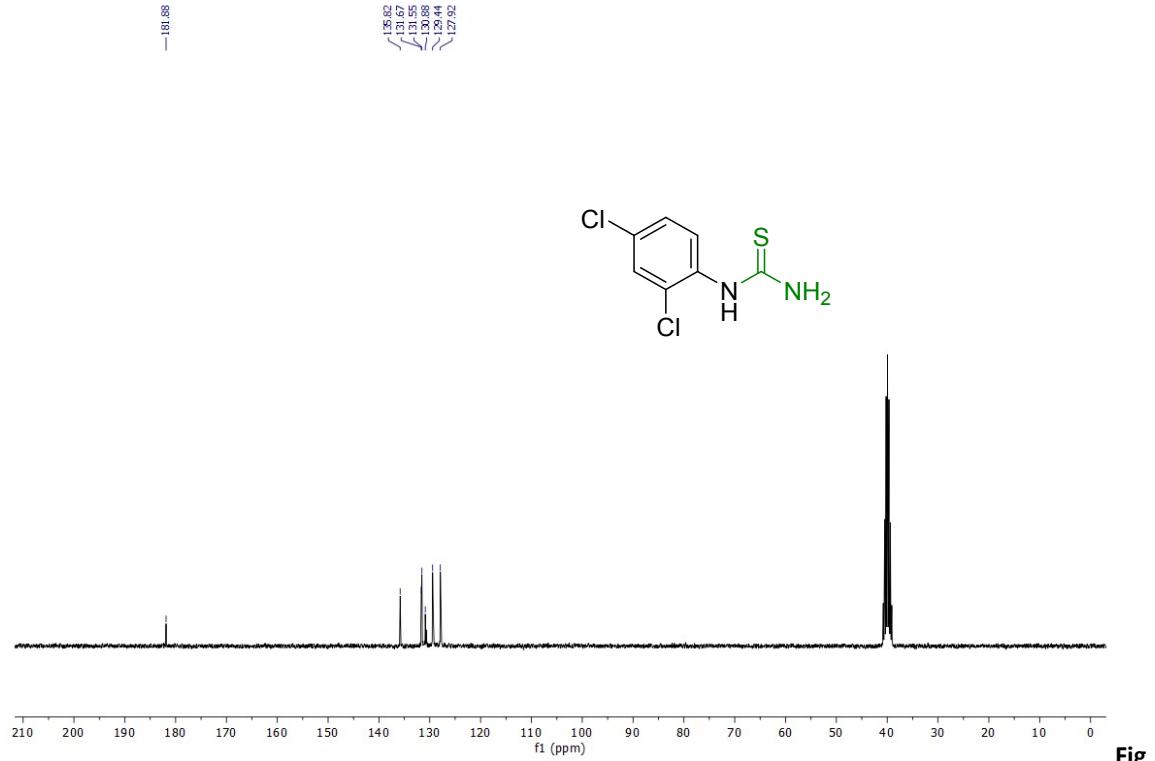
Figure S20. <sup>13</sup>C NMR spectrum of 3g in DMSO-*d*<sub>6</sub> (101MHz)



**Figure S21.** FT-IR spectrum of **3g** in KBr

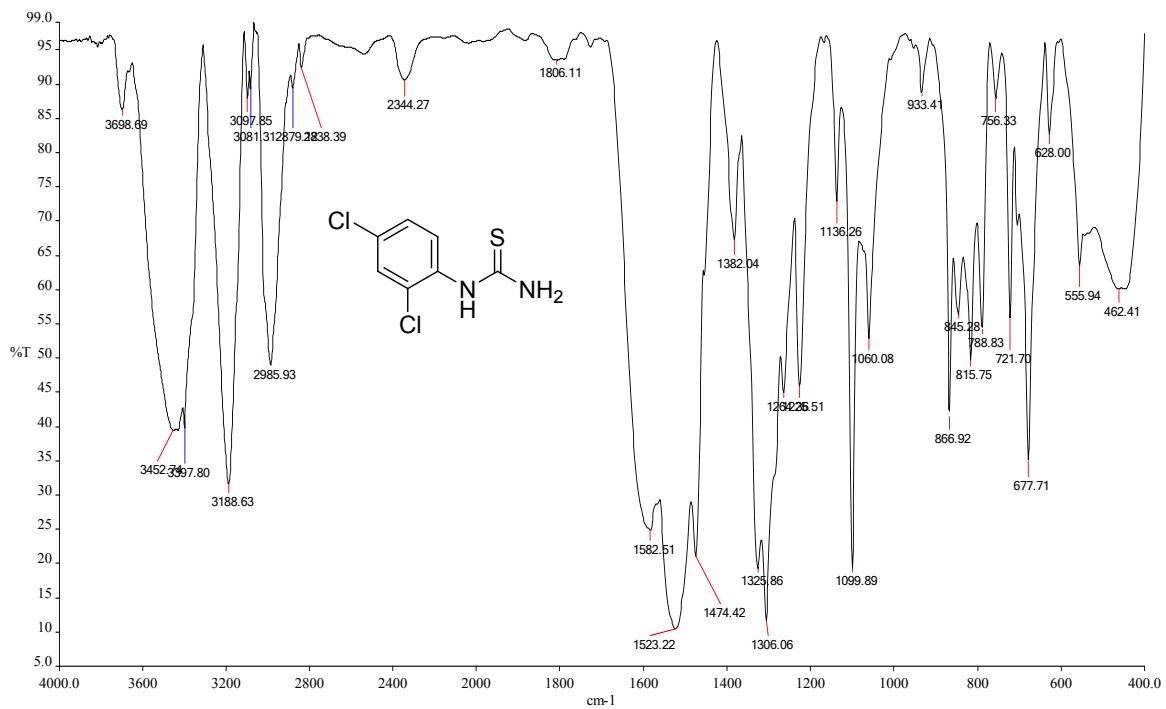


**Figure S22.**  $^1\text{H}$  NMR spectrum of **3h** in  $\text{DMSO}-d_6$  (300MHz)

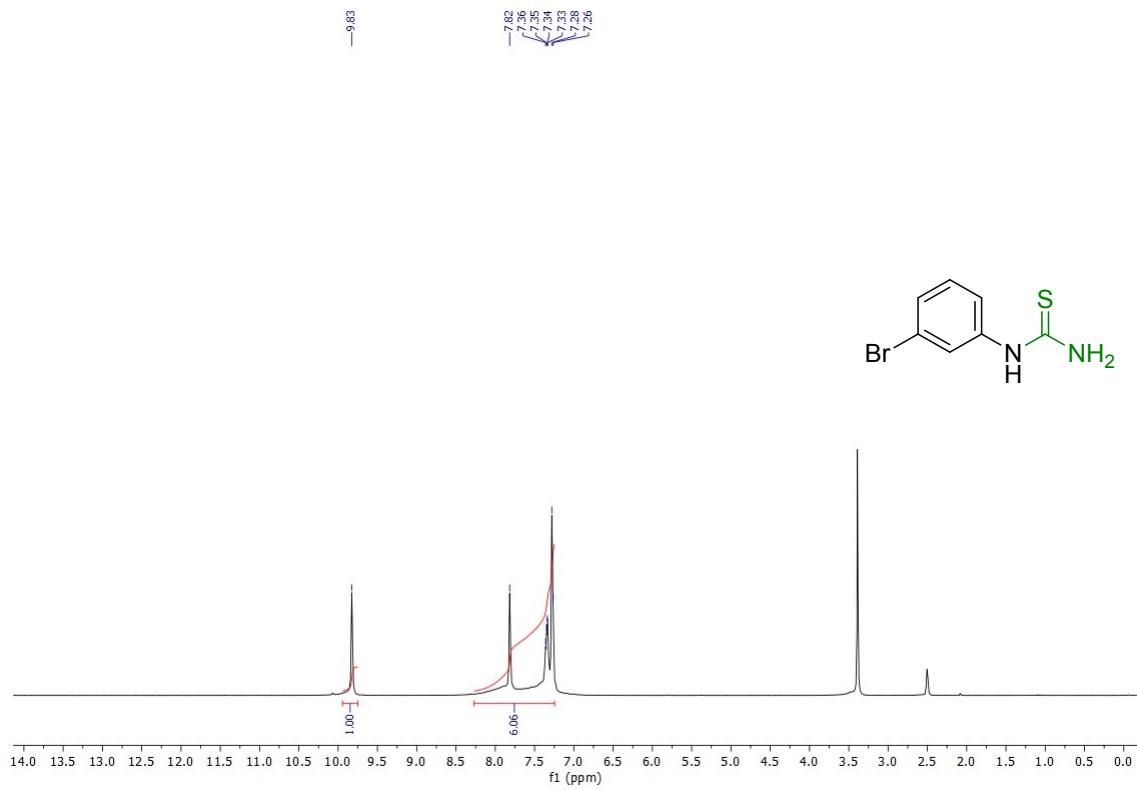


**Fig**

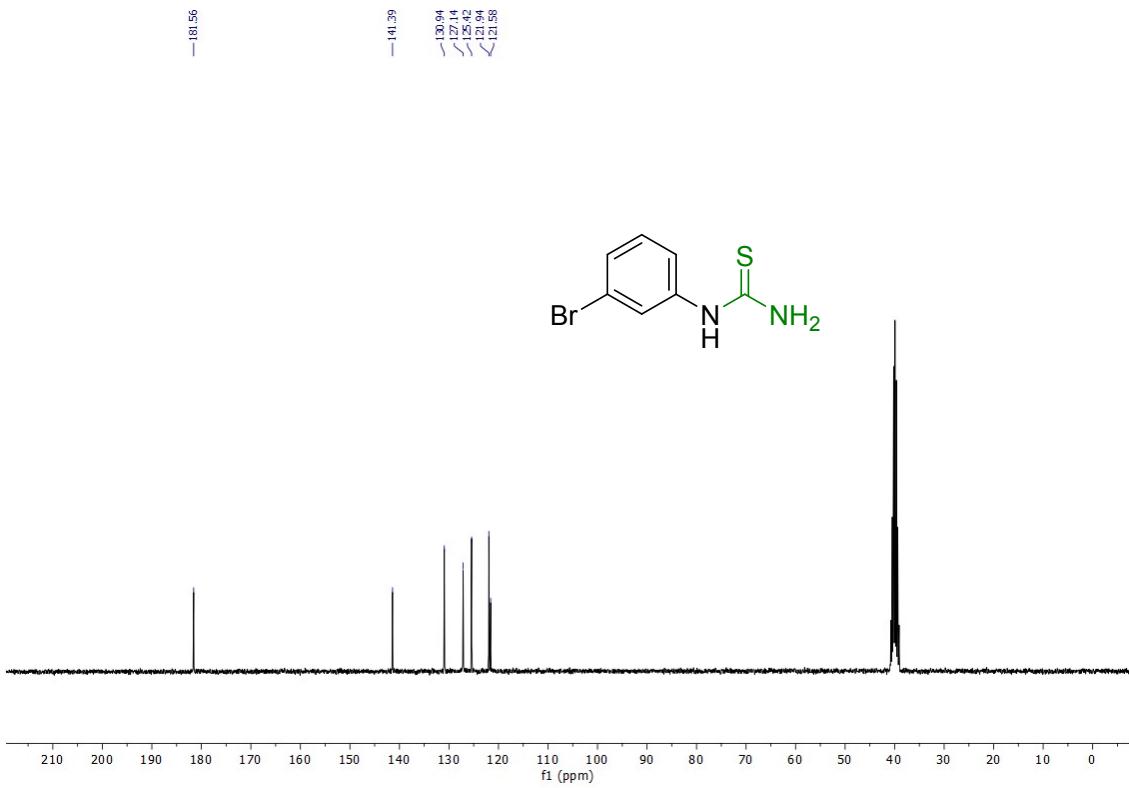
**ure S23. <sup>13</sup>C NMR spectrum of 3h in DMSO-d<sub>6</sub> (75MHz)**



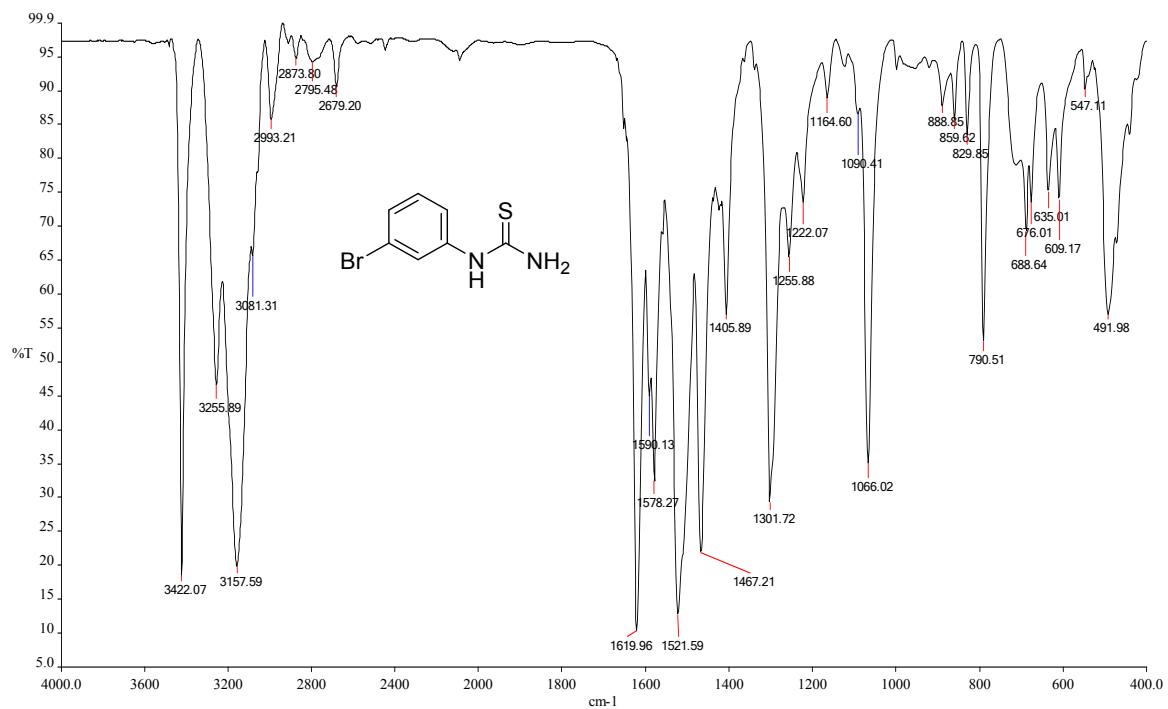
**Figure S24. FT-IR spectrum of 3h in KBr**



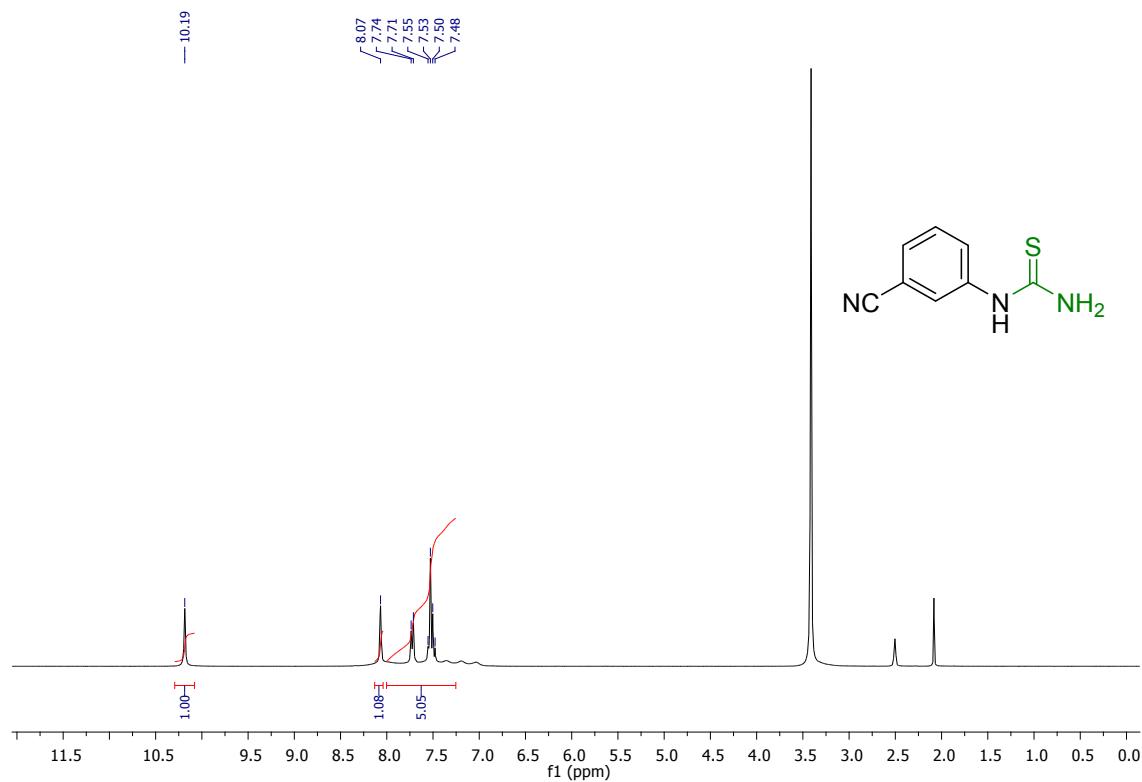
**Figure S25.** <sup>1</sup>H NMR spectrum of 3i in DMSO-d<sub>6</sub> (300MHz)



**Figure S26.** <sup>13</sup>C NMR spectrum of 3i in DMSO-d<sub>6</sub> (75MHz)



**Figure S27.** FT-IR spectrum of **3i** in KBr



**Figure S28.**  $^1\text{H}$  NMR spectrum of **3j** in  $\text{DMSO}-d_6$  (300MHz)

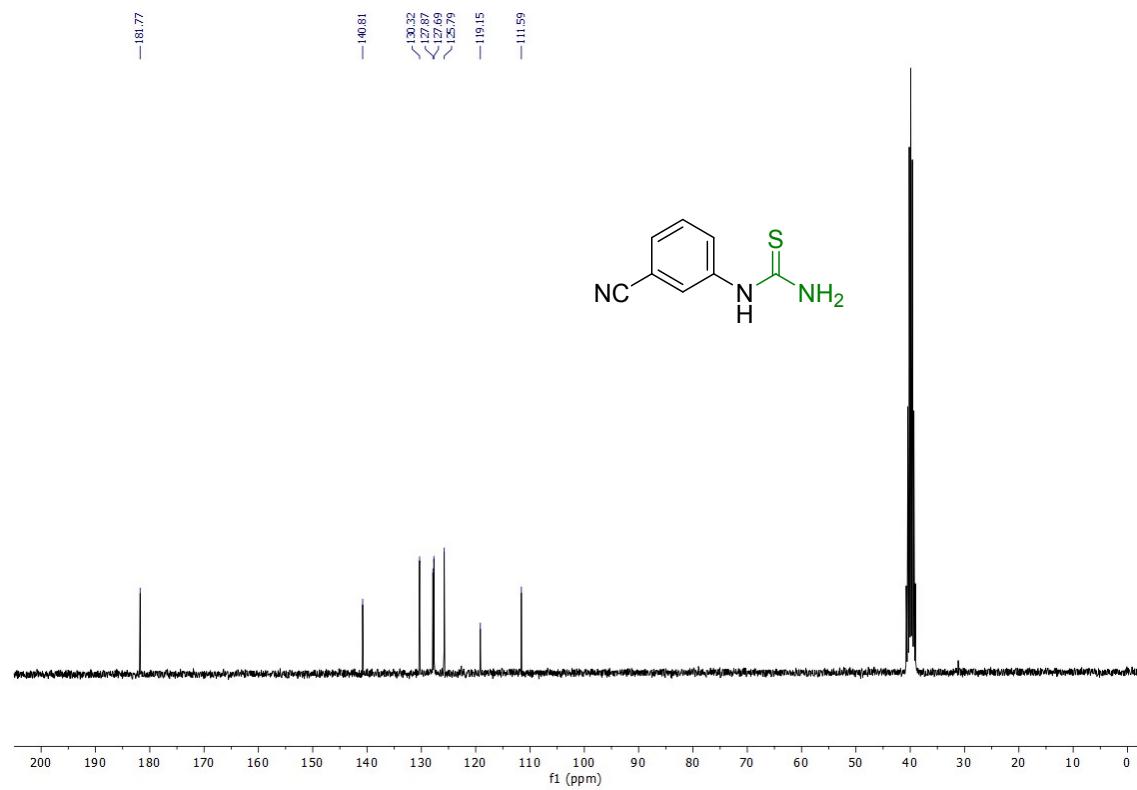


Figure S29. <sup>13</sup>C NMR spectrum of 3j in DMSO-*d*<sub>6</sub> (75MHz)

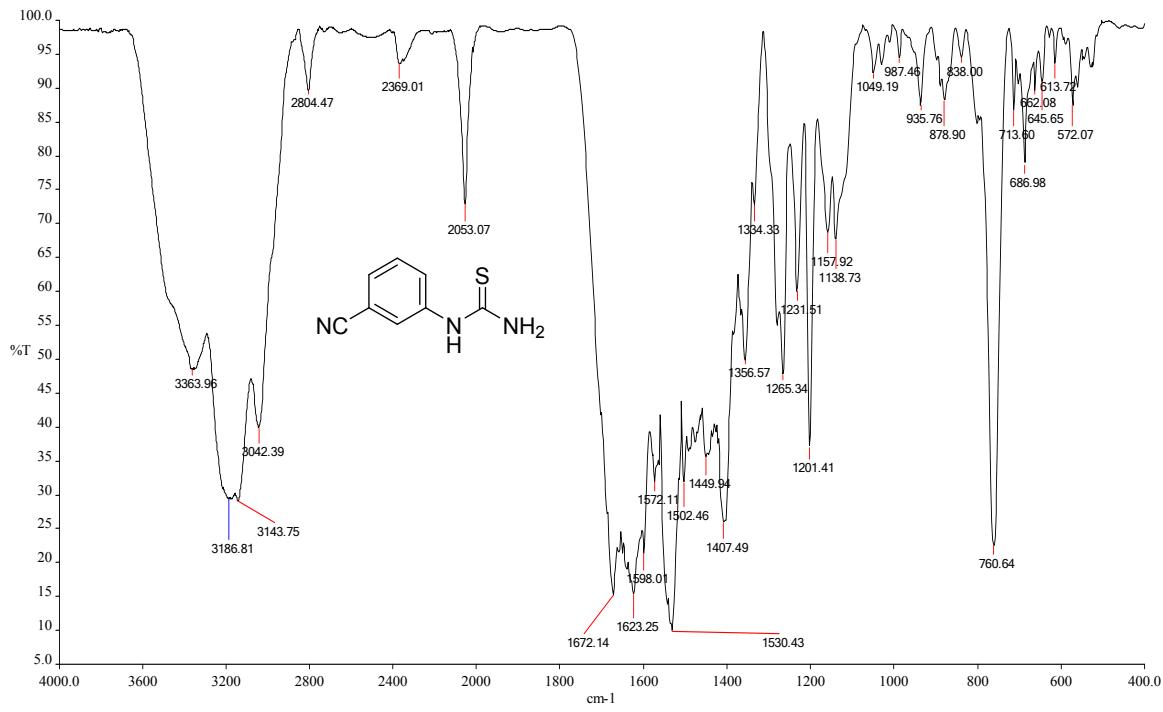
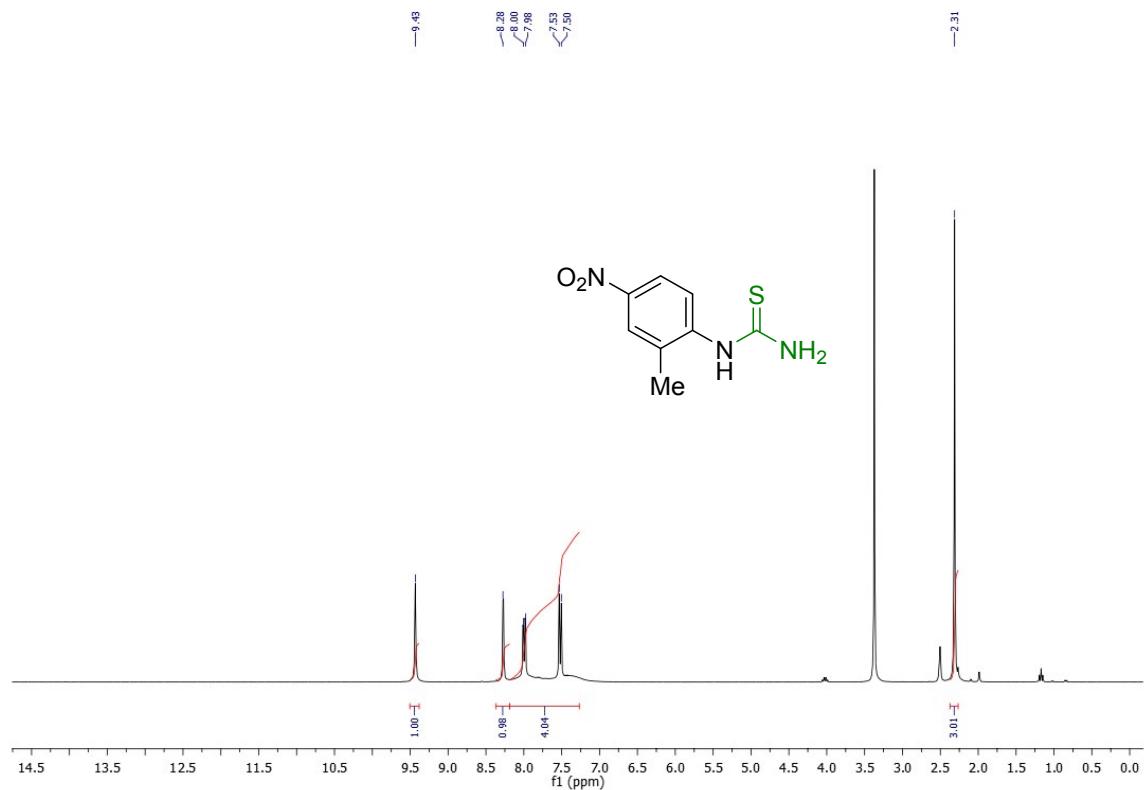
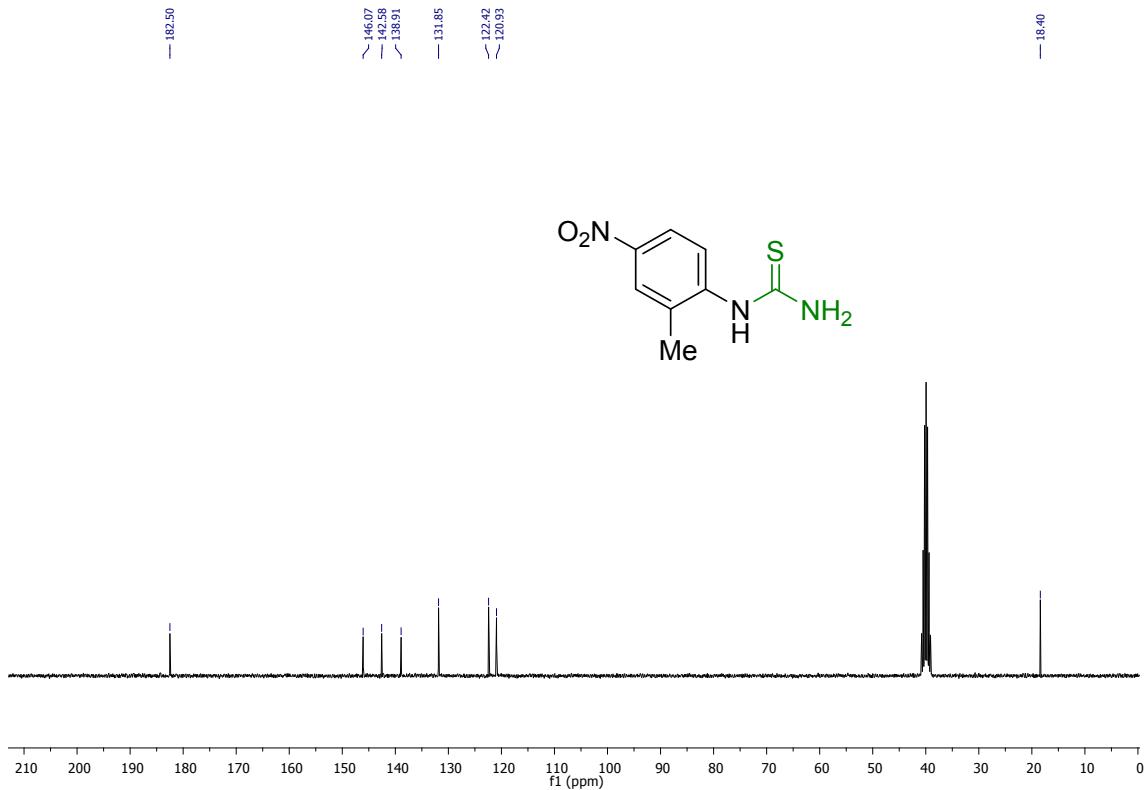


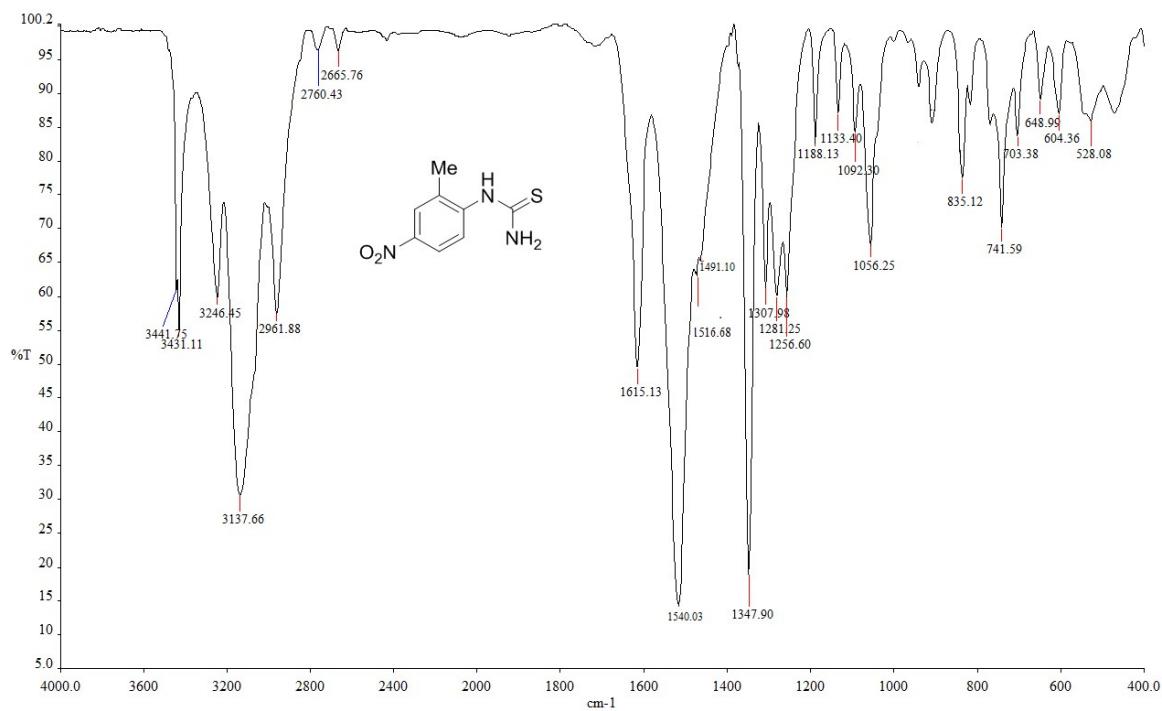
Figure S30. FT-IR spectrum of 3j in KBr



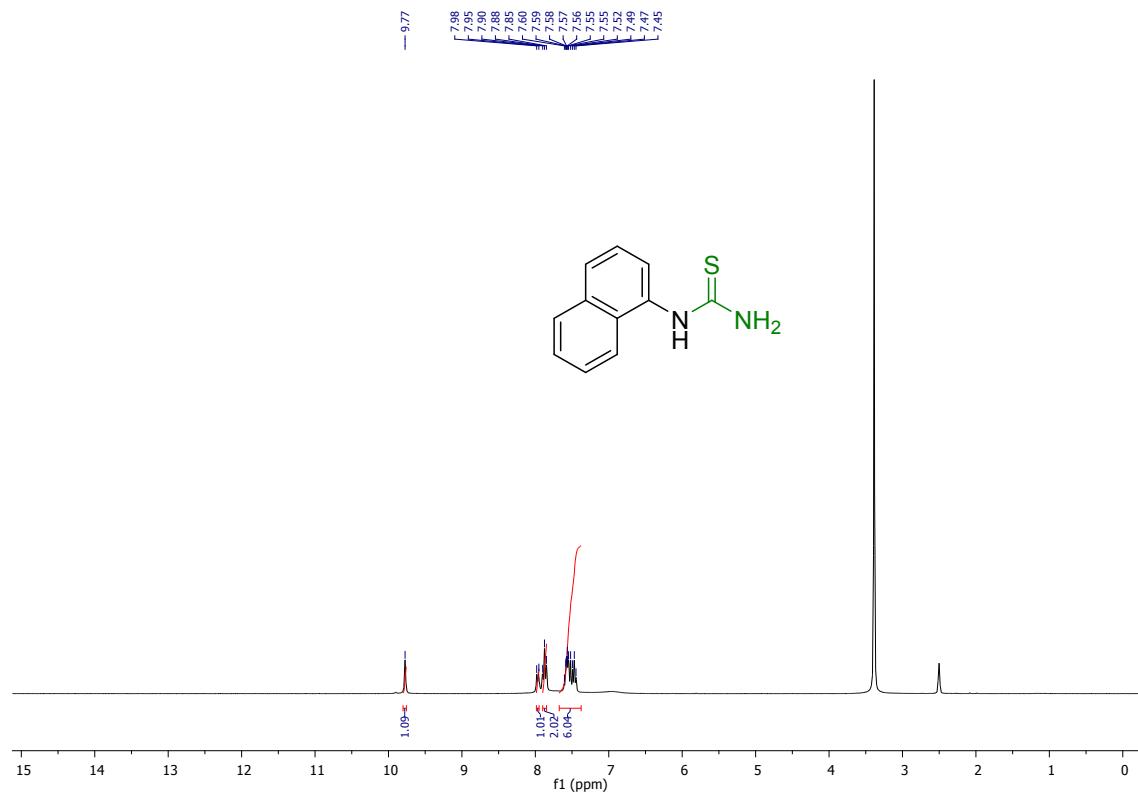
**Figure S31.**  $^1\text{H}$  NMR spectrum of **3k** in  $\text{DMSO}-d_6$  (300MHz)



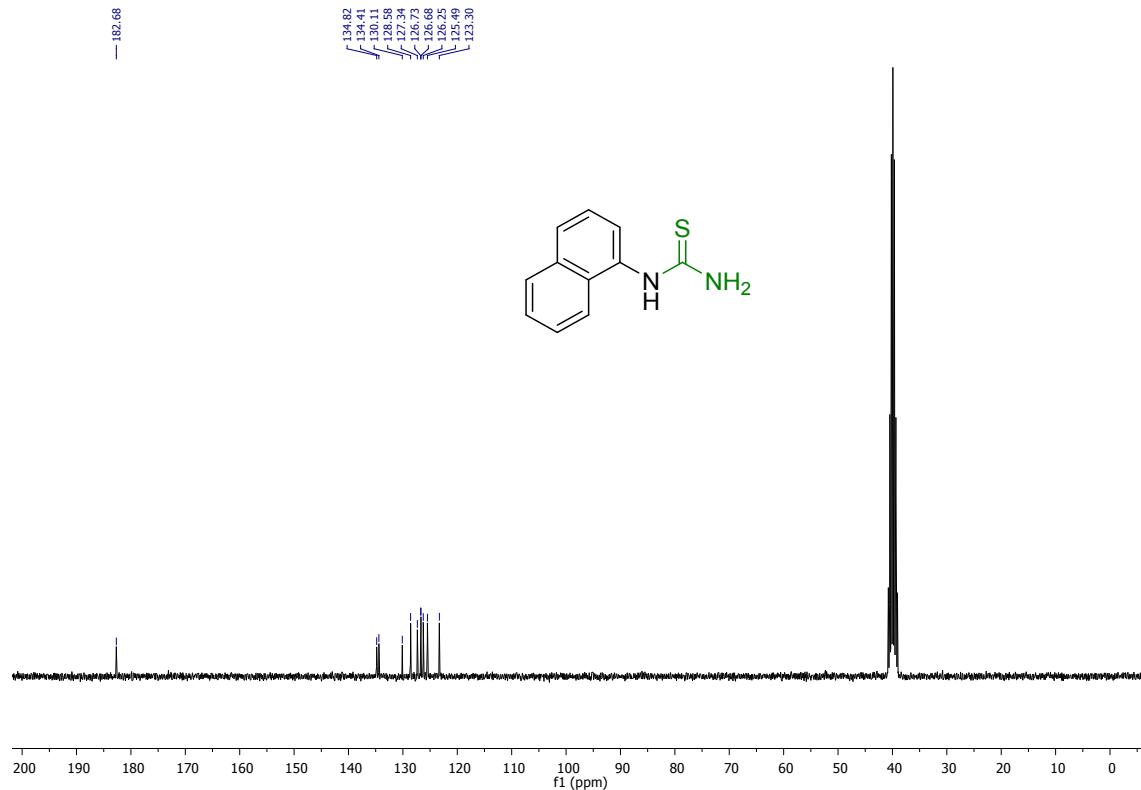
**Figure S32.**  $^{13}\text{C}$  NMR spectrum of **3k** in  $\text{DMSO}-d_6$  (75MHz).



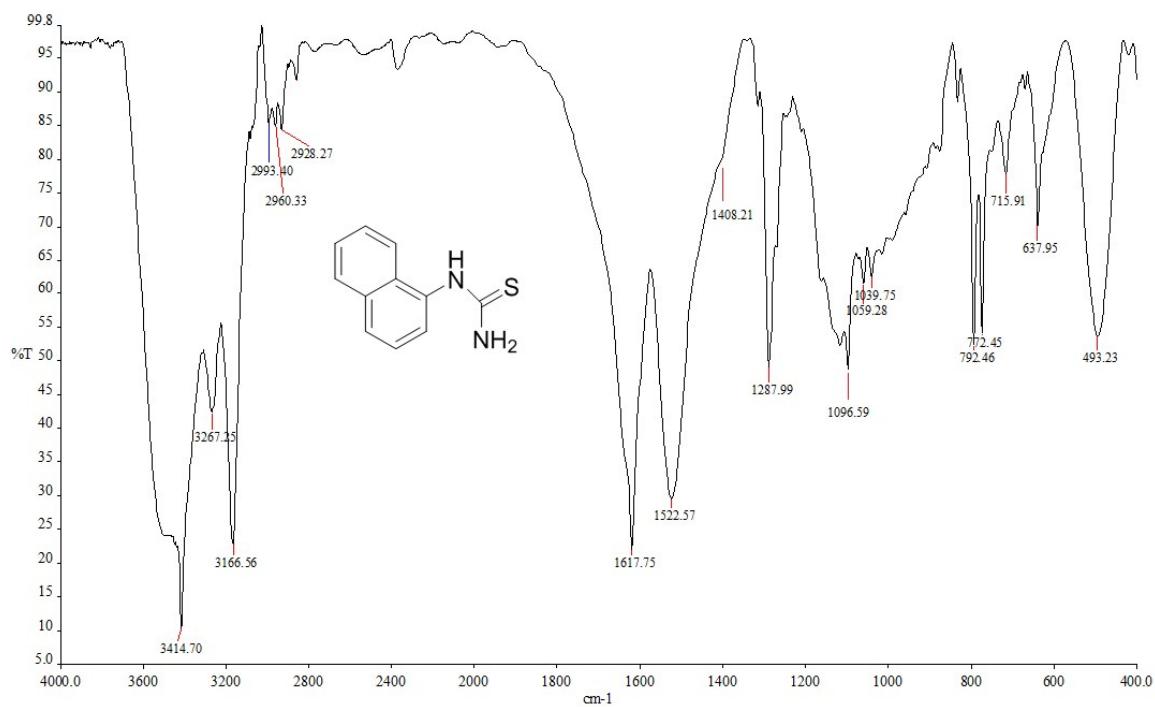
**Figure S33.** FT-IR spectrum of **3k** in KBr



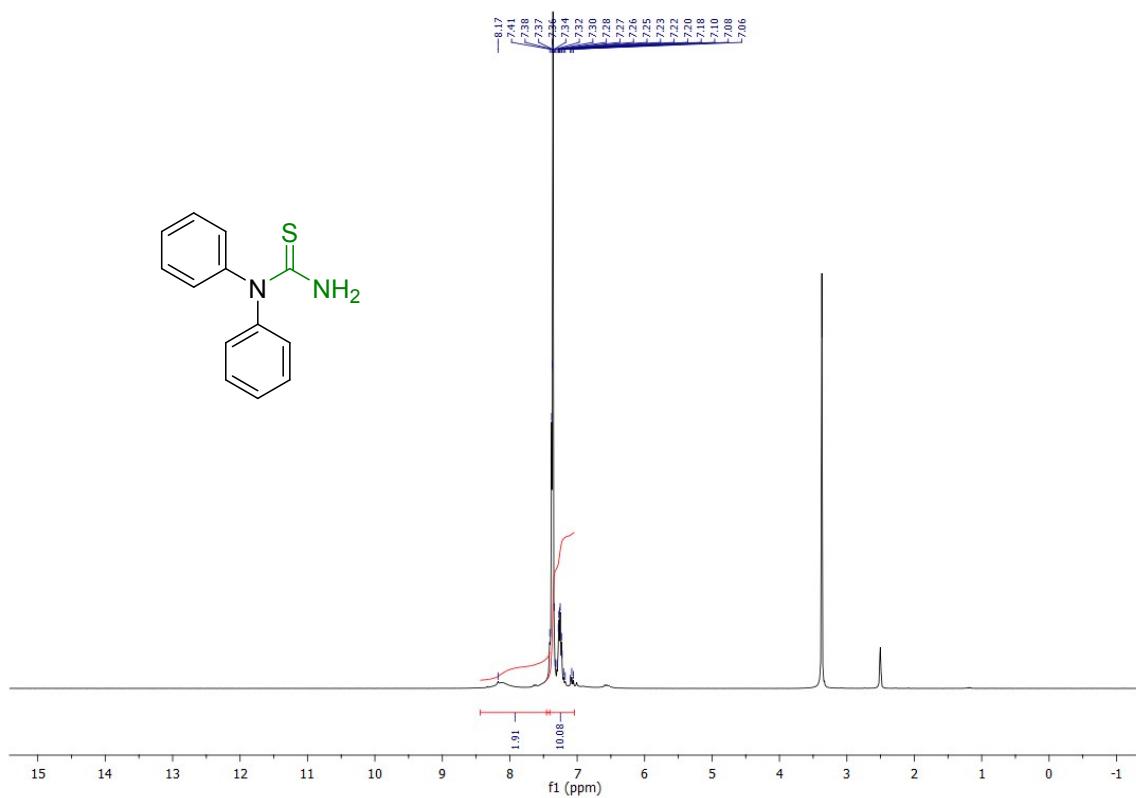
**Figure S34.**  $^1\text{H}$  NMR spectrum of **3l** in  $\text{DMSO}-d_6$  (300MHz)



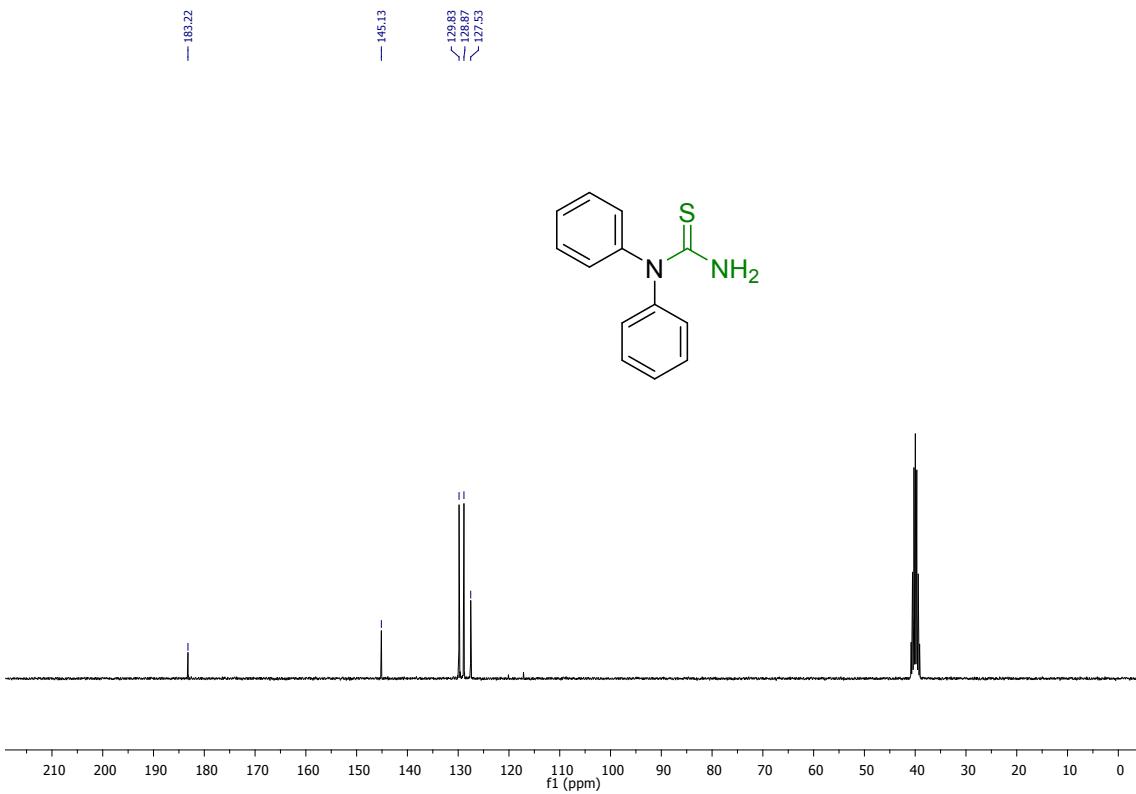
**Figure S35.**  $^{13}\text{C}$  NMR spectrum of 3l in  $\text{DMSO}-d_6$  (75MHz).



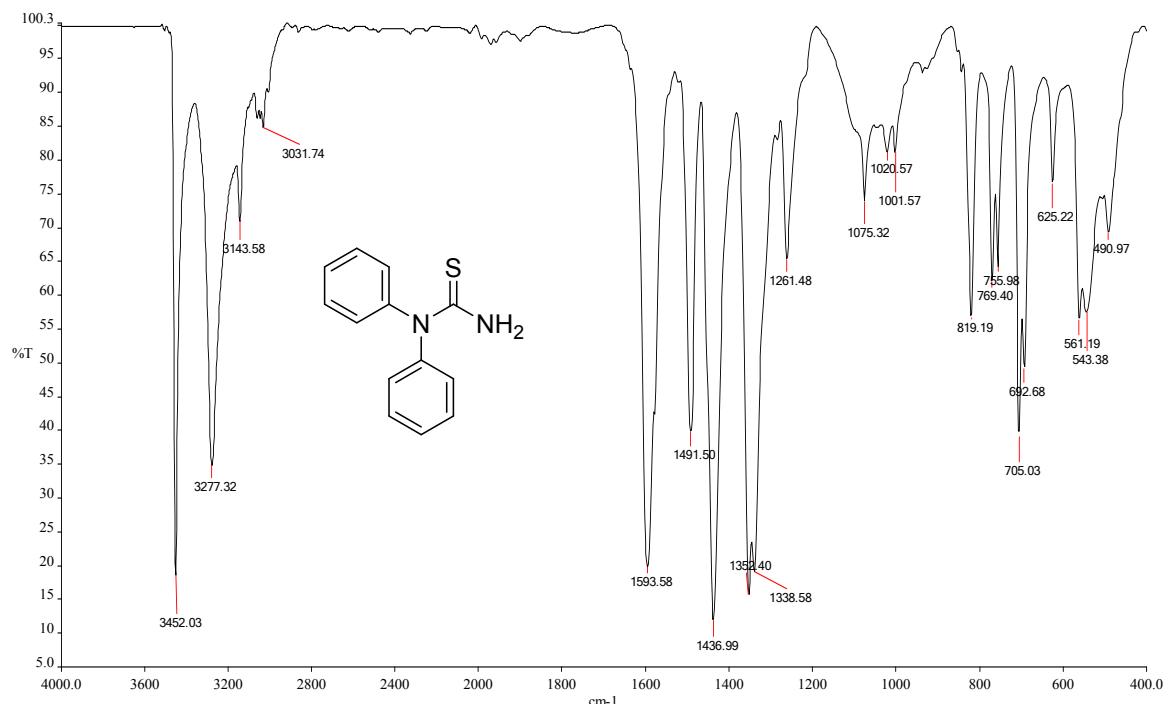
**Figure S36.** FT-IR spectrum of 3l in KBr



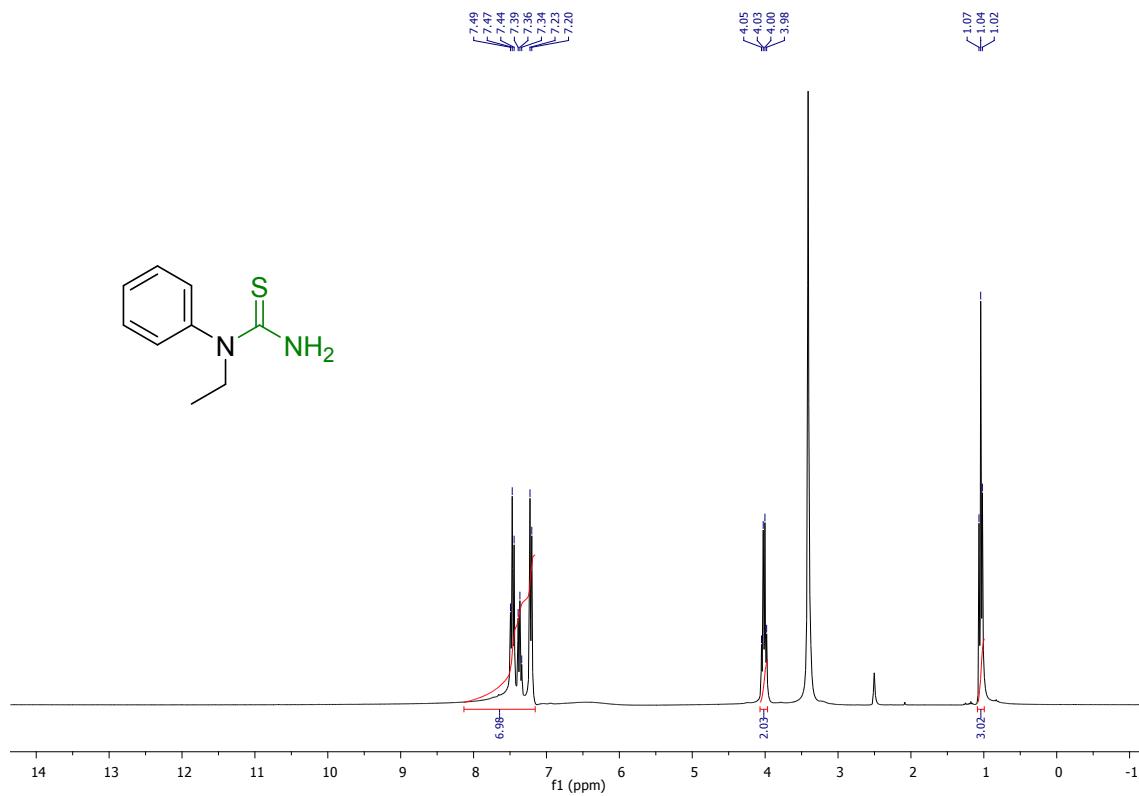
**Figure S37.**  $^1\text{H}$  NMR spectrum of 3m in  $\text{DMSO}-d_6$  (300MHz).



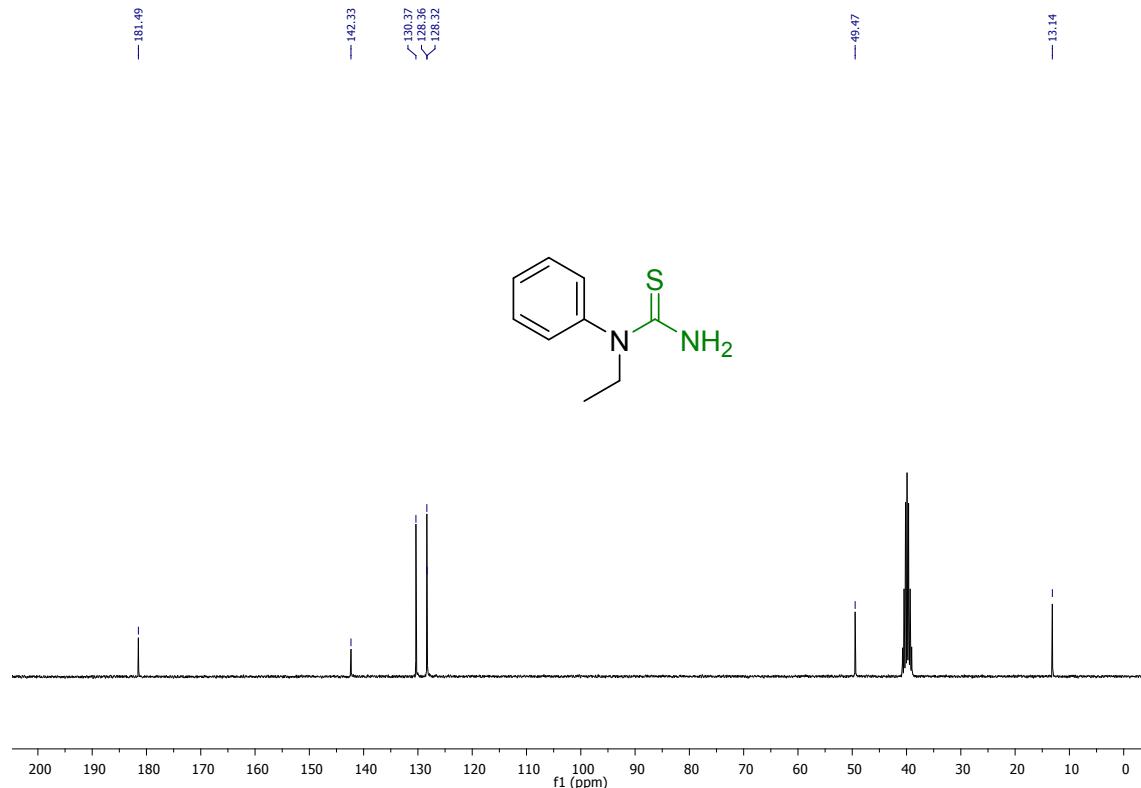
**Figure S38.**  $^{13}\text{C}$  NMR spectrum of 3m in  $\text{DMSO}-d_6$  (75 MHz)



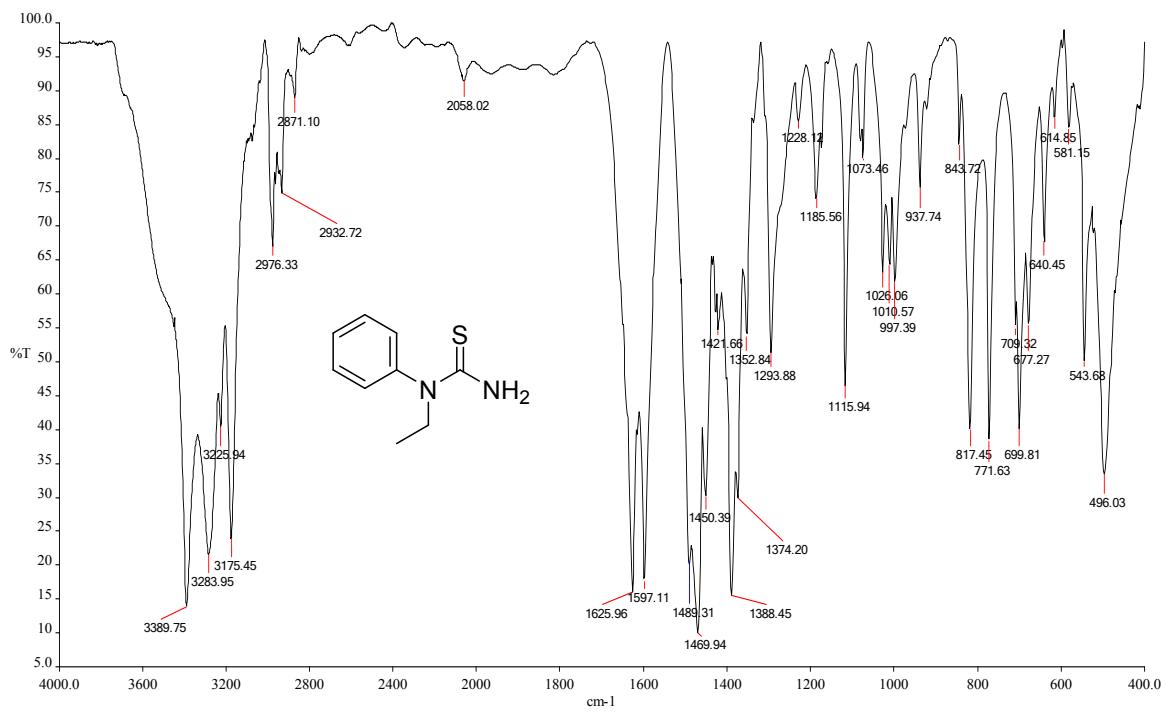
**Figure S39.** FT-IR spectrum of 3m in KBr



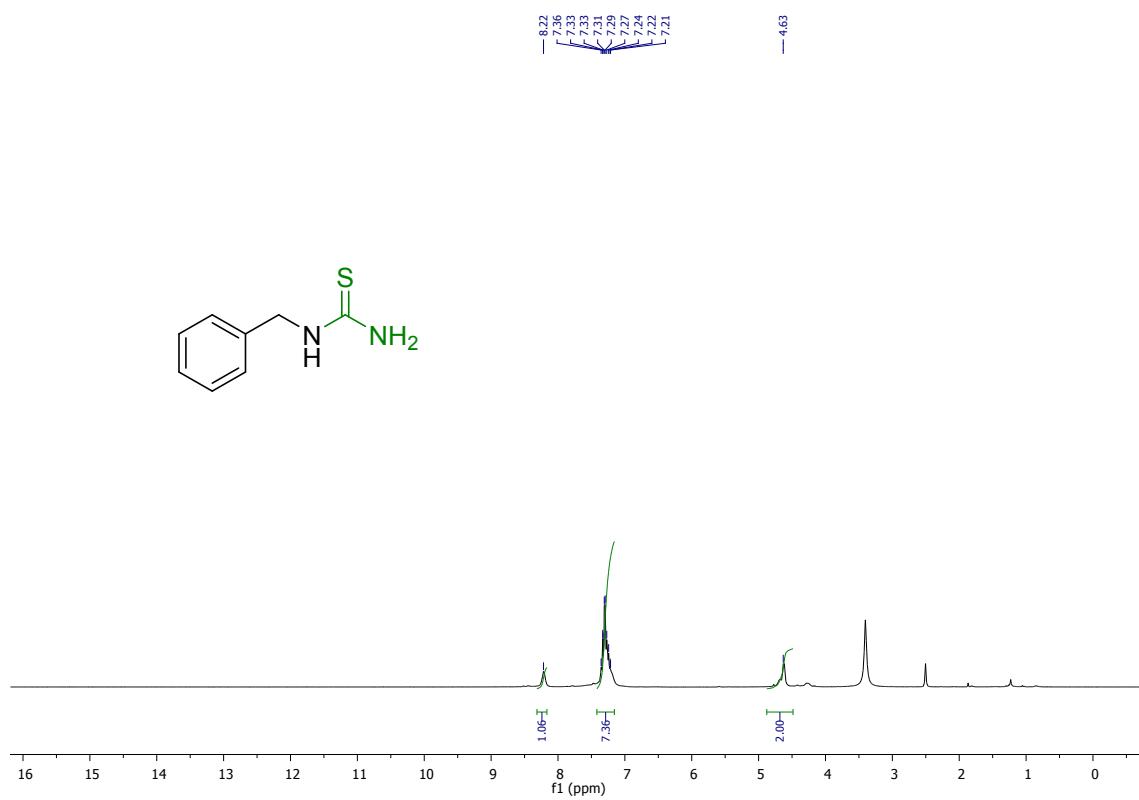
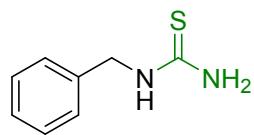
**Figure S40.**  $^1\text{H}$  NMR spectrum of 3n in  $\text{DMSO}-d_6$  (300MHz)



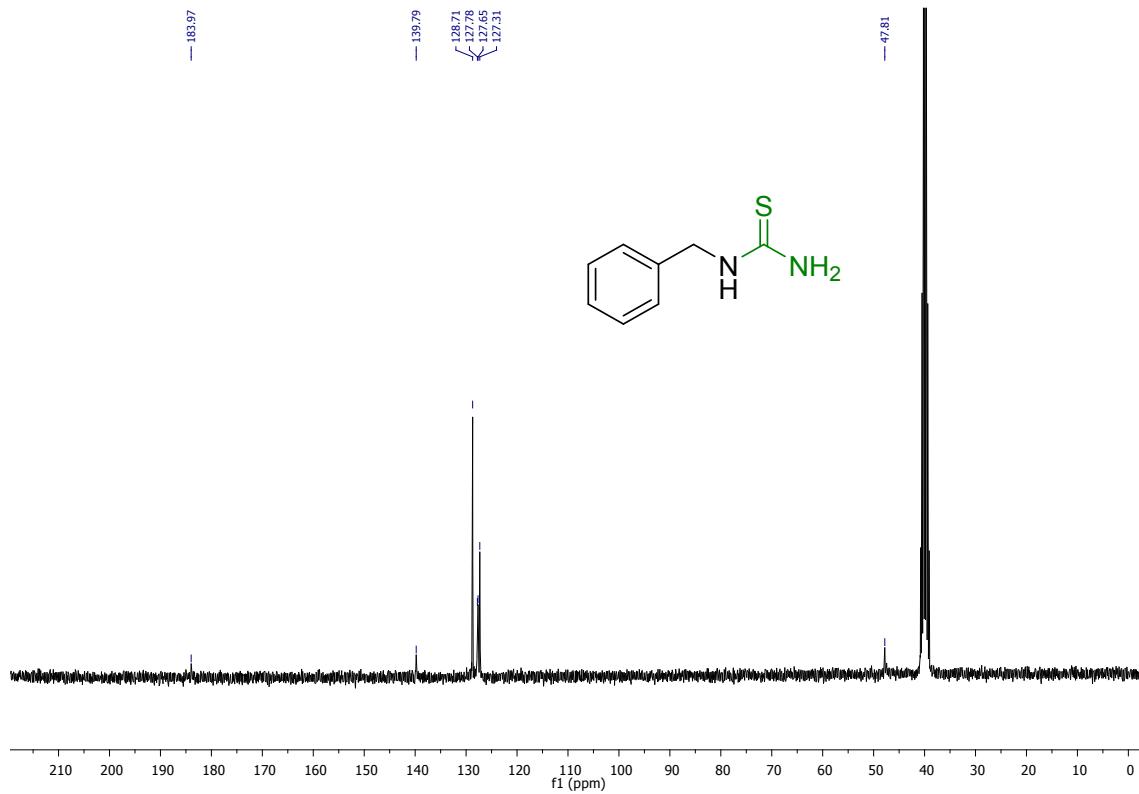
**Figure S41.**  $^{13}\text{C}$  NMR spectrum of **3n** in  $\text{DMSO}-d_6$  (75MHz)



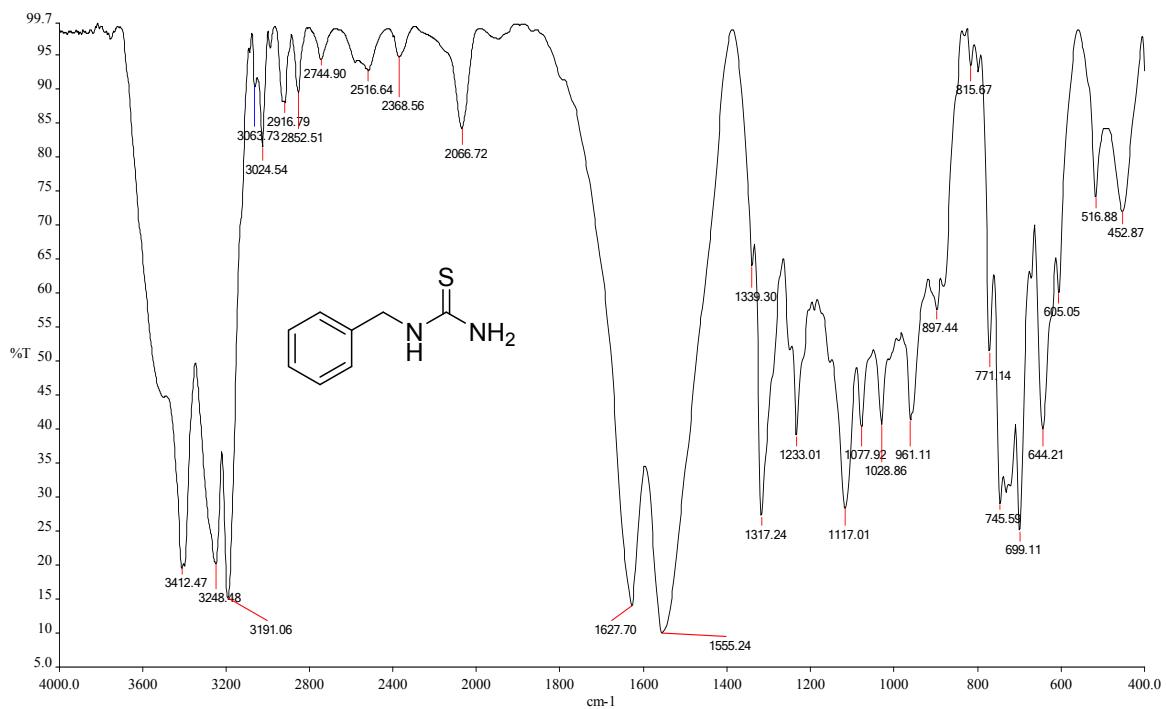
**Figure S42.** FT-IR spectrum of **3n** in KBr



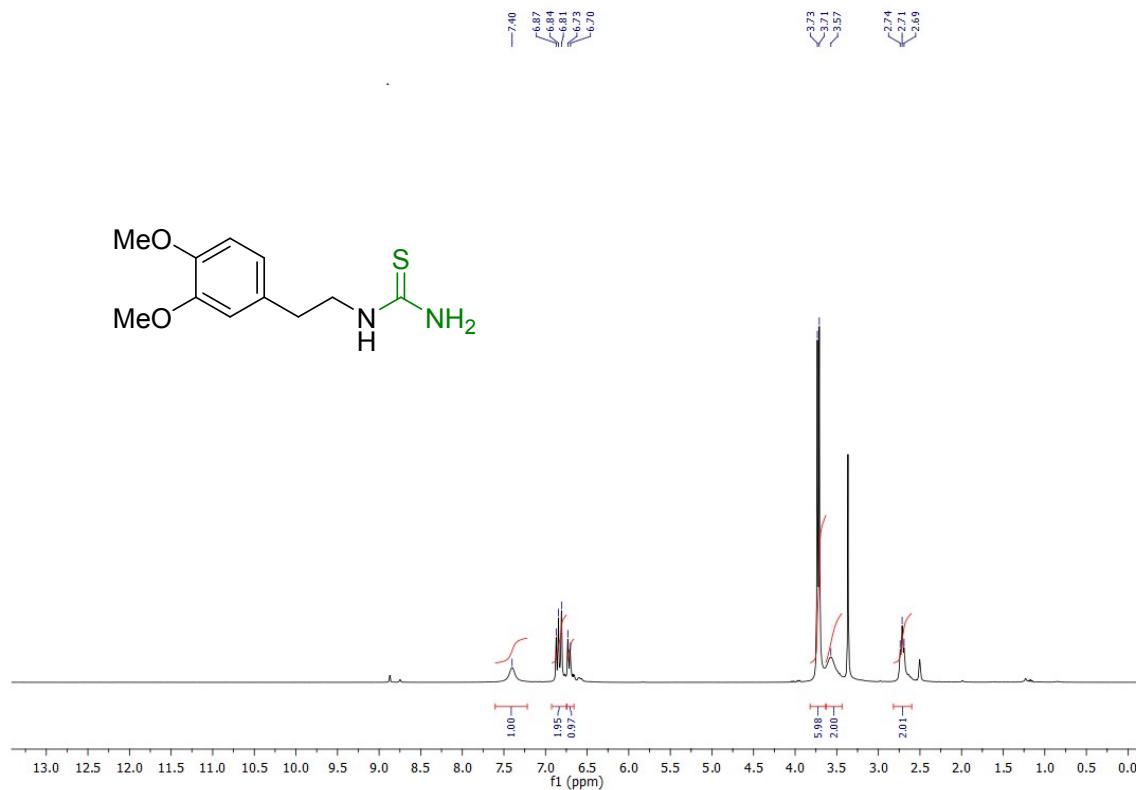
**Figure S43.**  $^1\text{H}$  NMR spectrum of 3o in  $\text{DMSO}-d_6$  (300MHz)



**Figure S44.**  $^{13}\text{C}$  NMR spectrum of 3o in  $\text{DMSO}-d_6$  (75MHz)



**Figure S45.** FT-IR spectrum of 3o in KBr



**Figure S46.**  $^1\text{H}$  NMR spectrum of 3p in  $\text{DMSO}-d_6$  (300MHz)

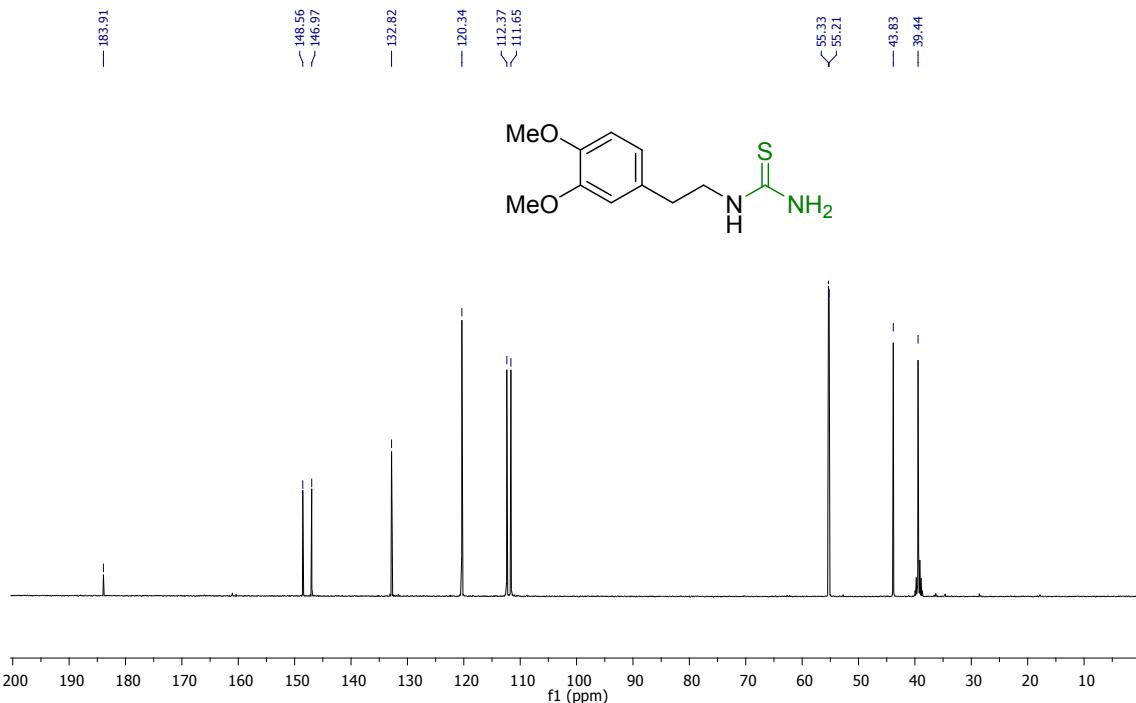


Figure S47.  $^{13}\text{C}$  NMR spectrum of 3p in  $\text{DMSO}-d_6$  (101MHz)

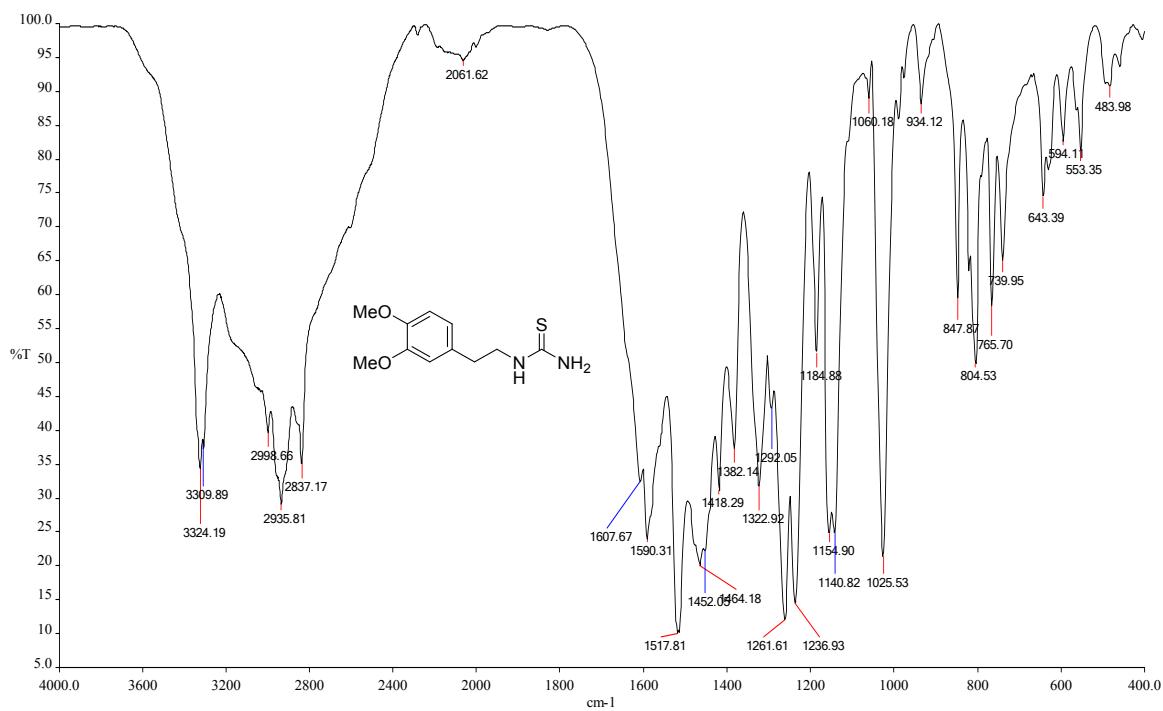
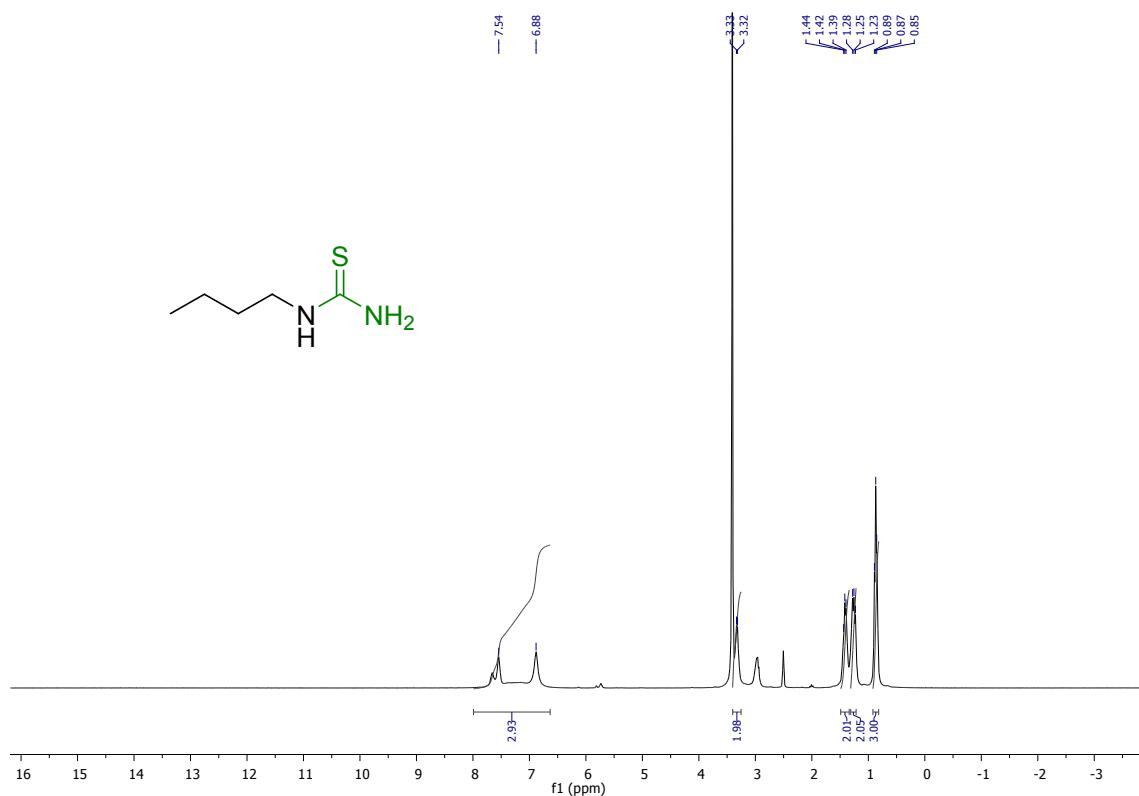
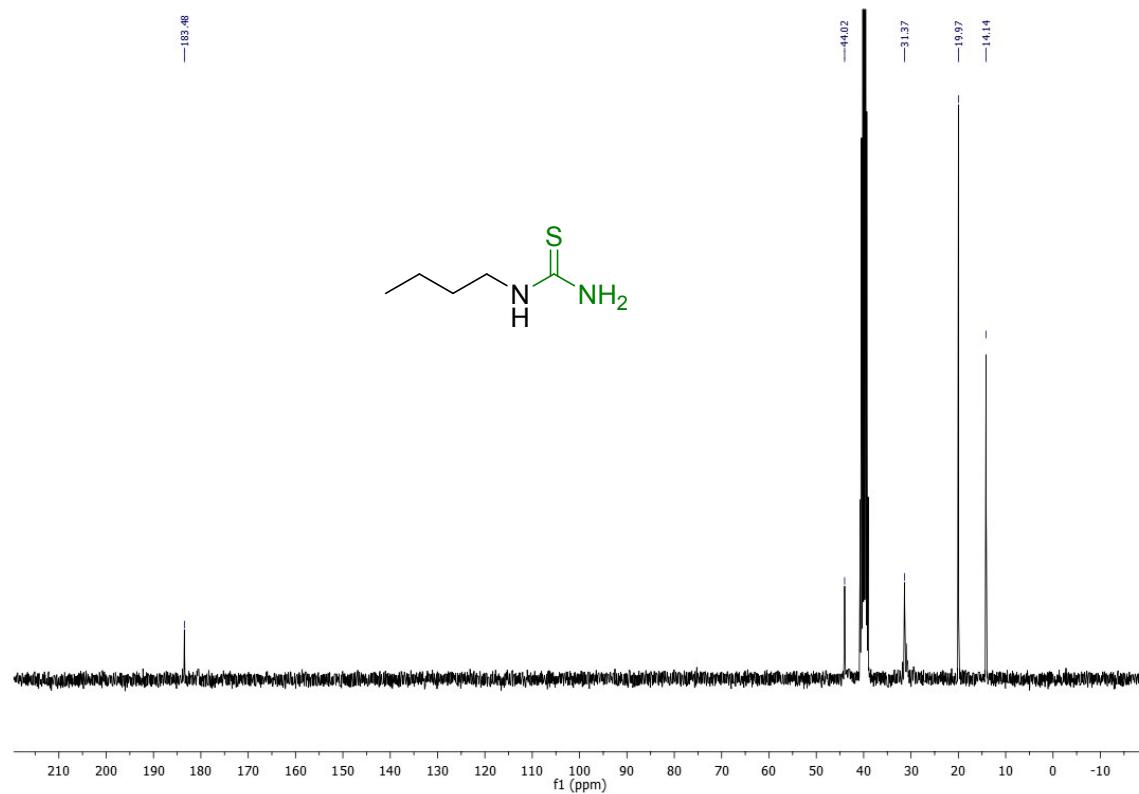


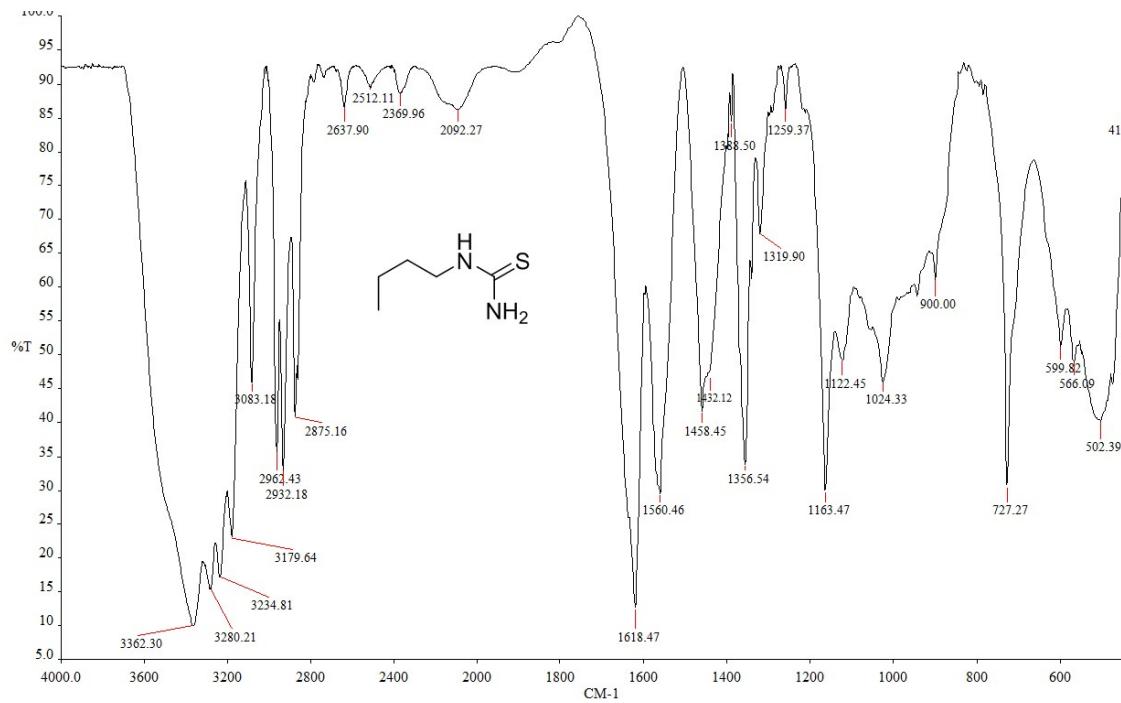
Figure S48. FT-IR spectrum of 3p in KBr



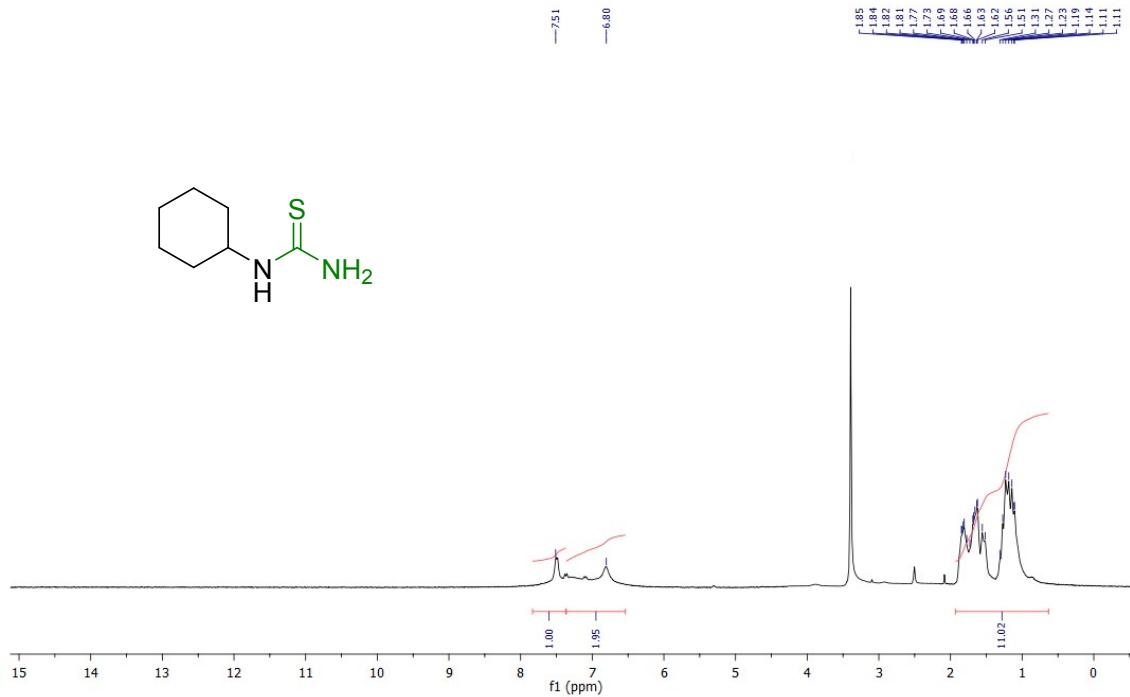
**Figure S49.**  $^1\text{H}$  NMR spectrum of 3q in  $\text{DMSO}-d_6$  (300MHz)



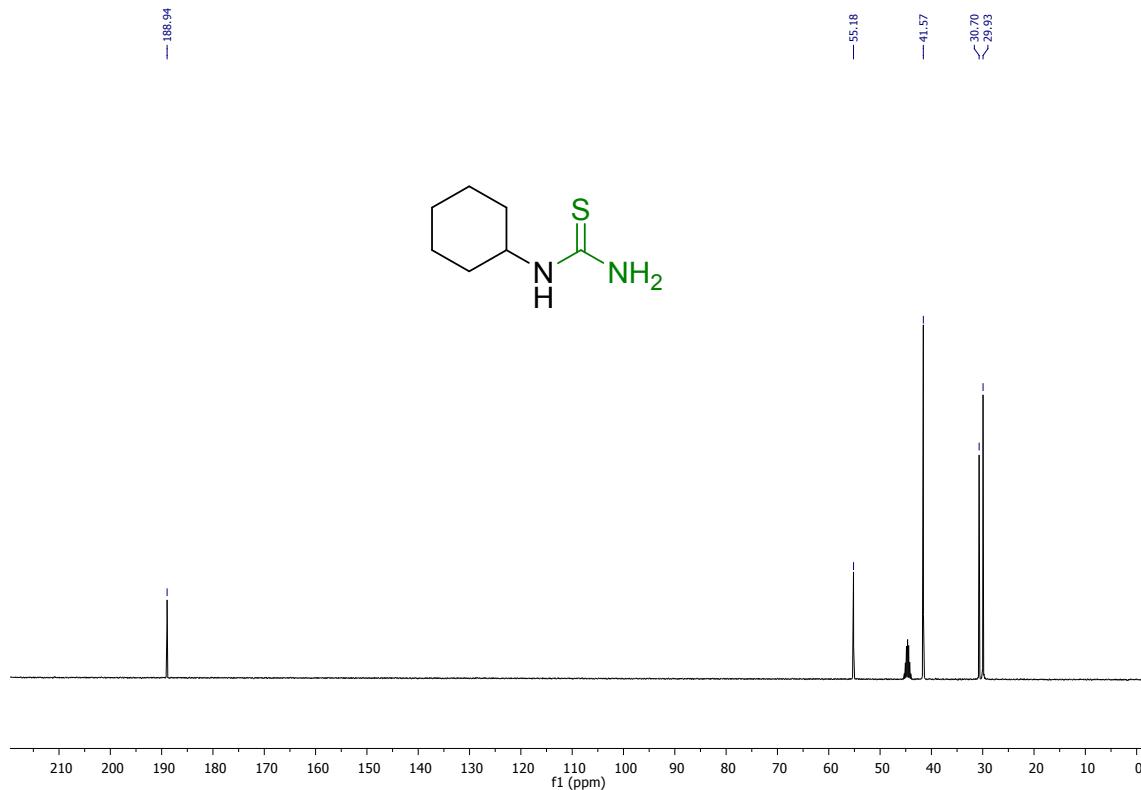
**Figure S50.**  $^{13}\text{C}$  NMR spectrum of 3q in  $\text{DMSO}-d_6$  (75MHz)



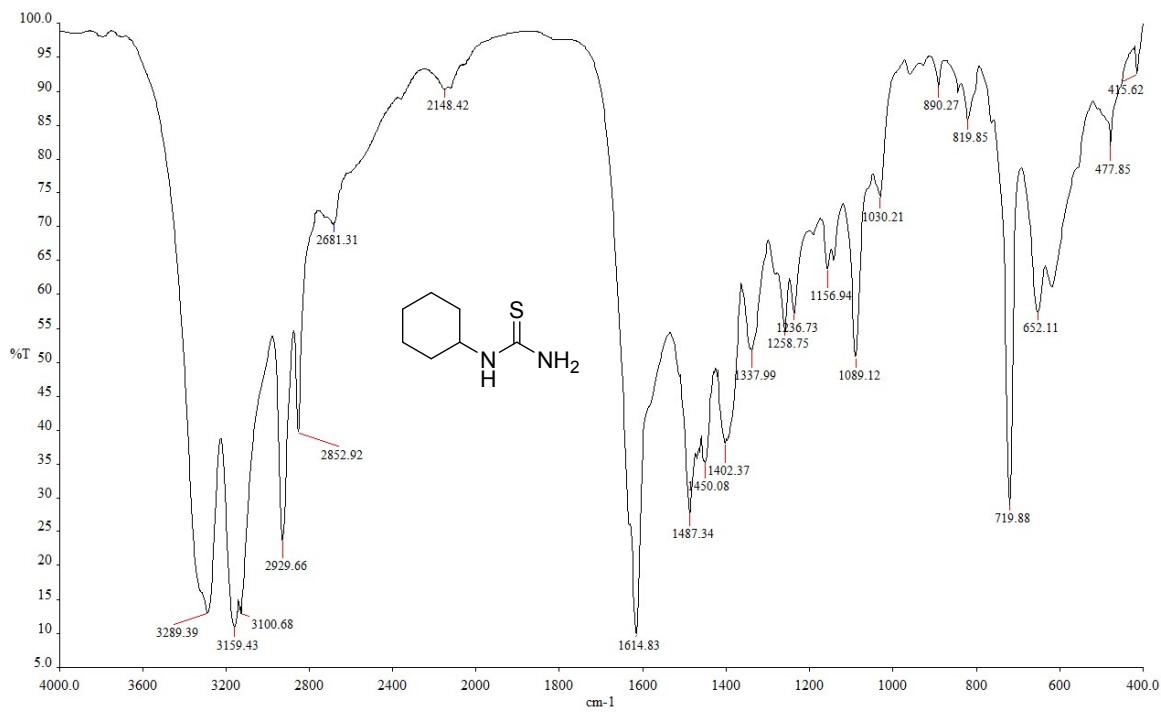
**Figure S51.** FT-IR spectrum of **3p** in KBr



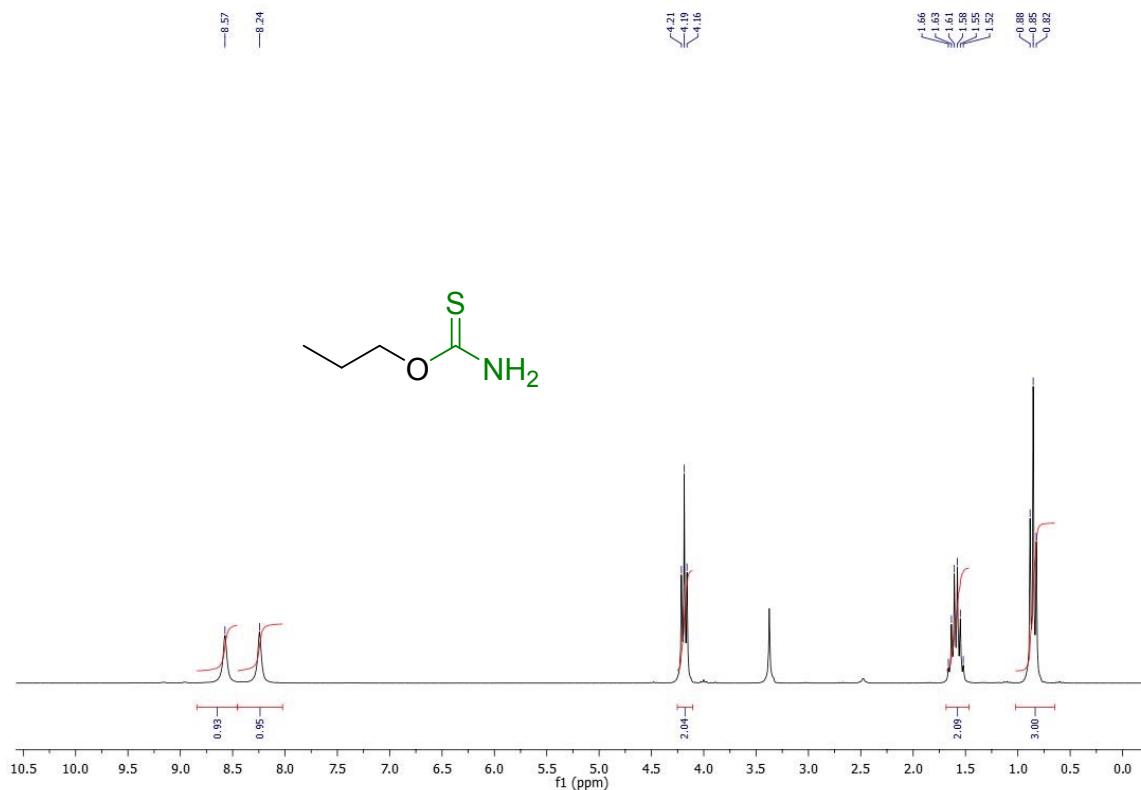
**Figure S52.**  $^1\text{H}$  NMR spectrum of **3r** in  $\text{DMSO}-d_6$  (300MHz)



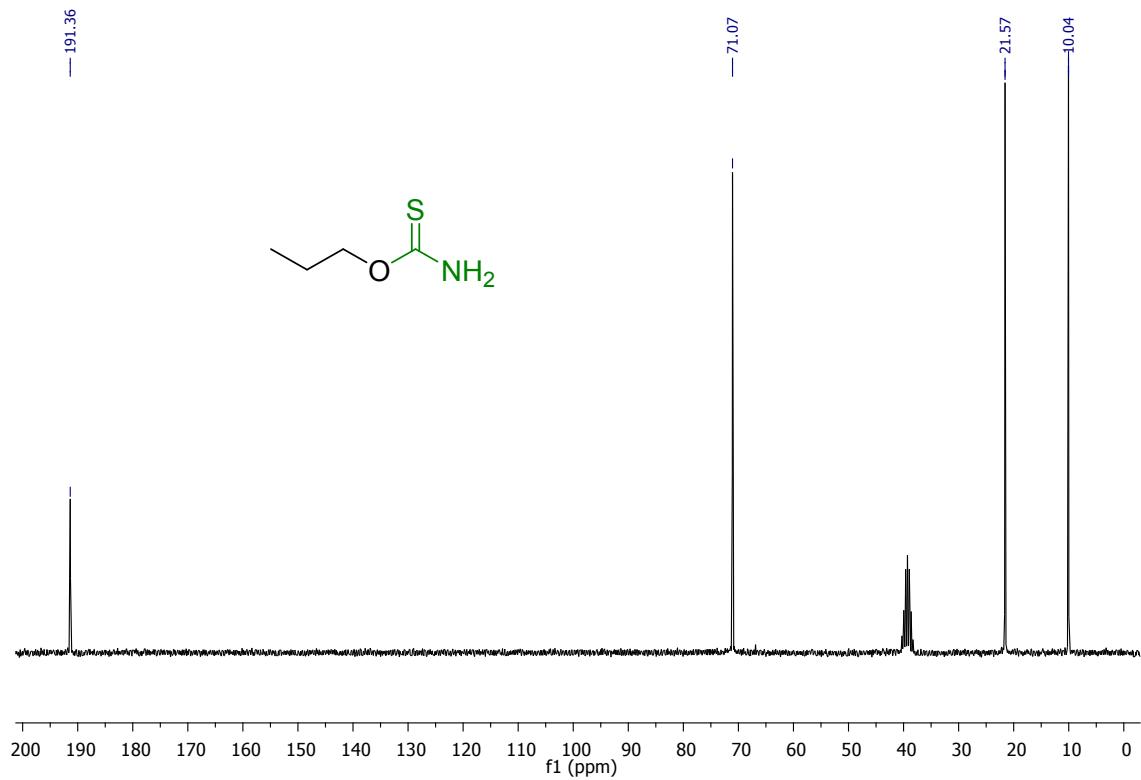
**Figure S53.**  $^{13}\text{C}$  NMR spectrum of **3r** in  $\text{DMSO}-d_6$  (101MHz)



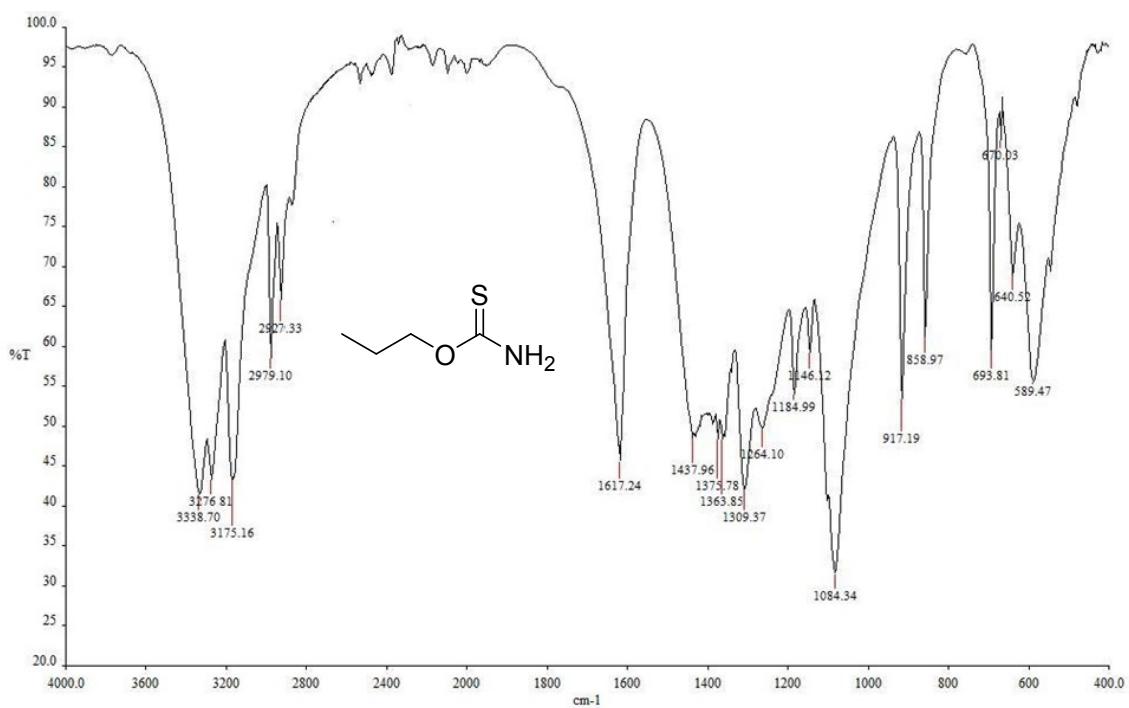
**Figure S54.** FT-IR spectrum of **3r** in KBr



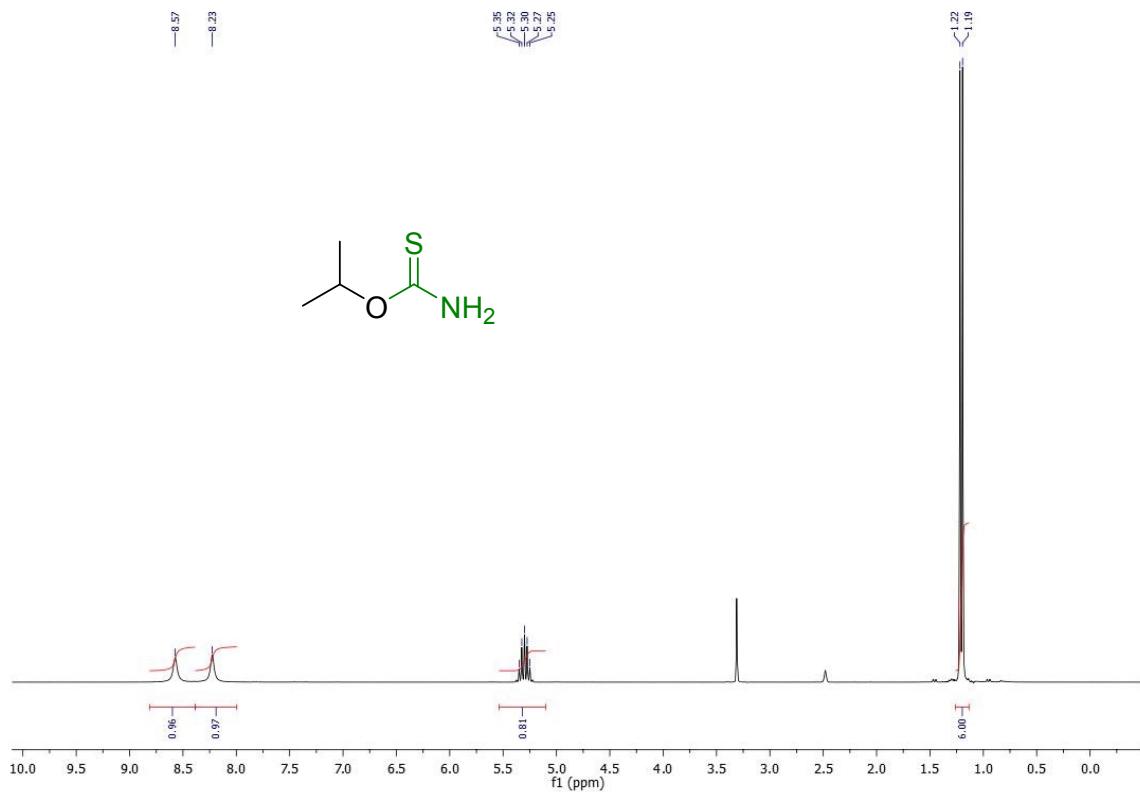
**Figure S55.**  $^1\text{H}$  NMR spectrum of 5a in  $\text{DMSO}-d_6$  (250MHz).



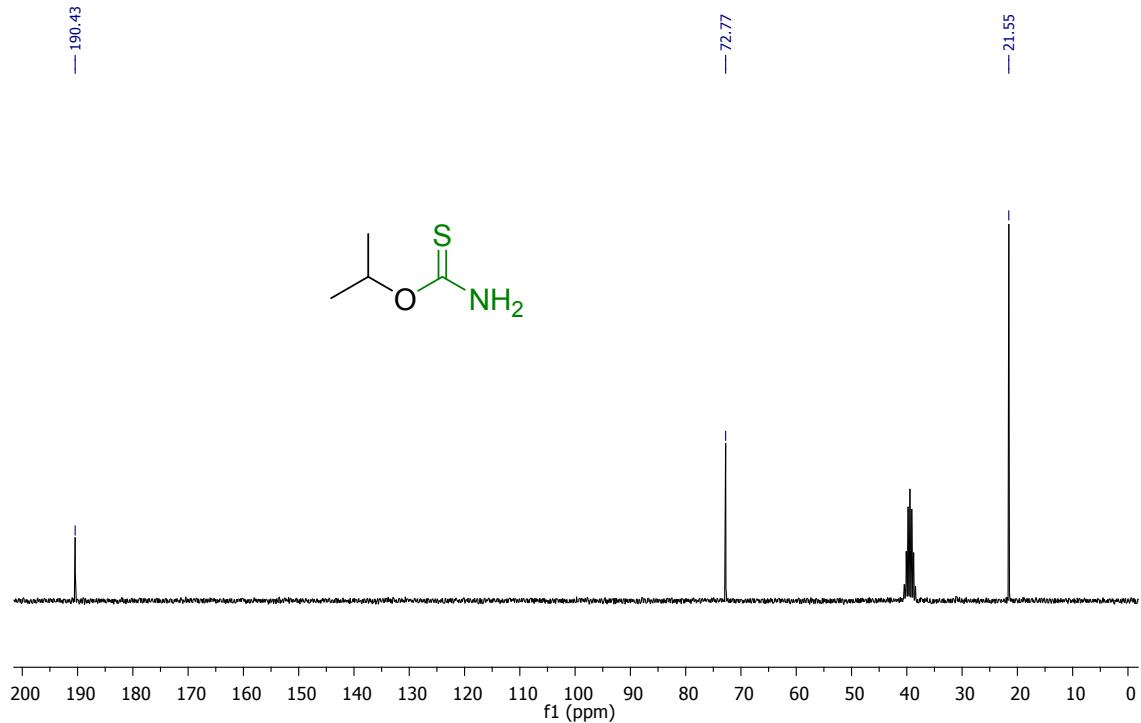
**Figure S56.**  $^{13}\text{C}$  NMR spectrum of 5a in  $\text{DMSO}-d_6$  (63MHz)



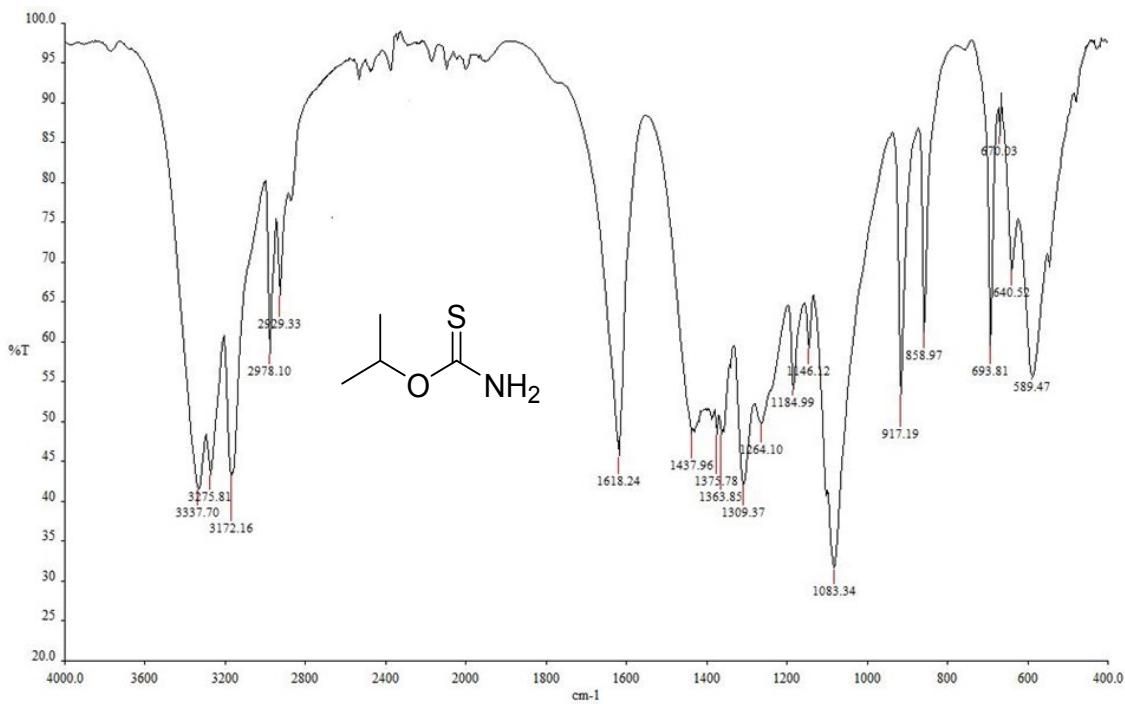
**Figure S57.** FT-IR spectrum of 5a in KBr



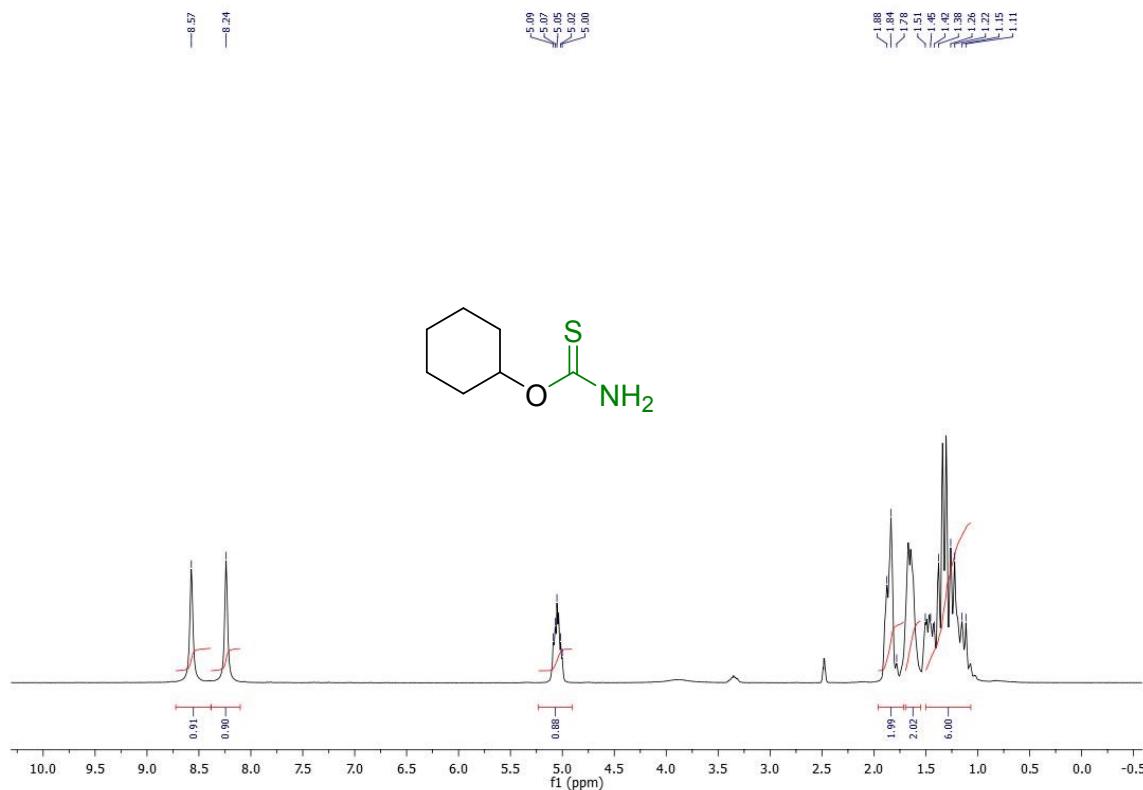
**Figure S58.**  $^1\text{H}$  NMR spectrum of 5b in  $\text{DMSO}-d_6$  (250MHz)



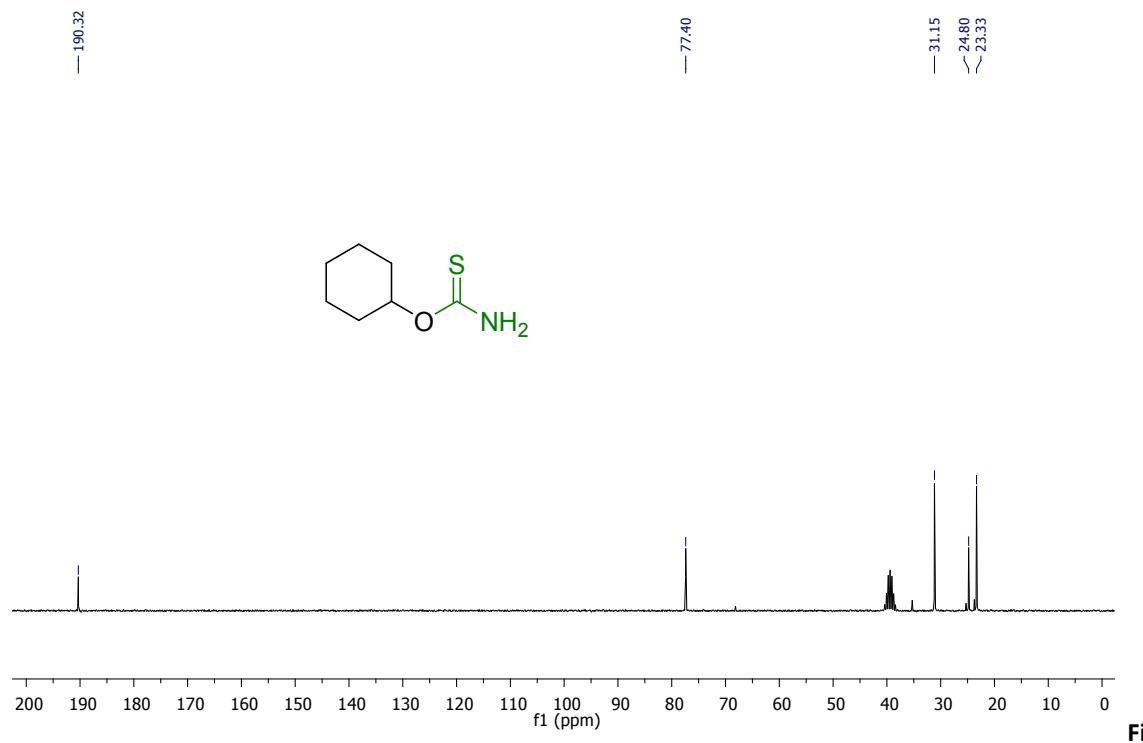
**Figure S59.**  $^{13}\text{C}$  NMR spectrum of 5b in  $\text{DMSO}-d_6$  (63MHz)



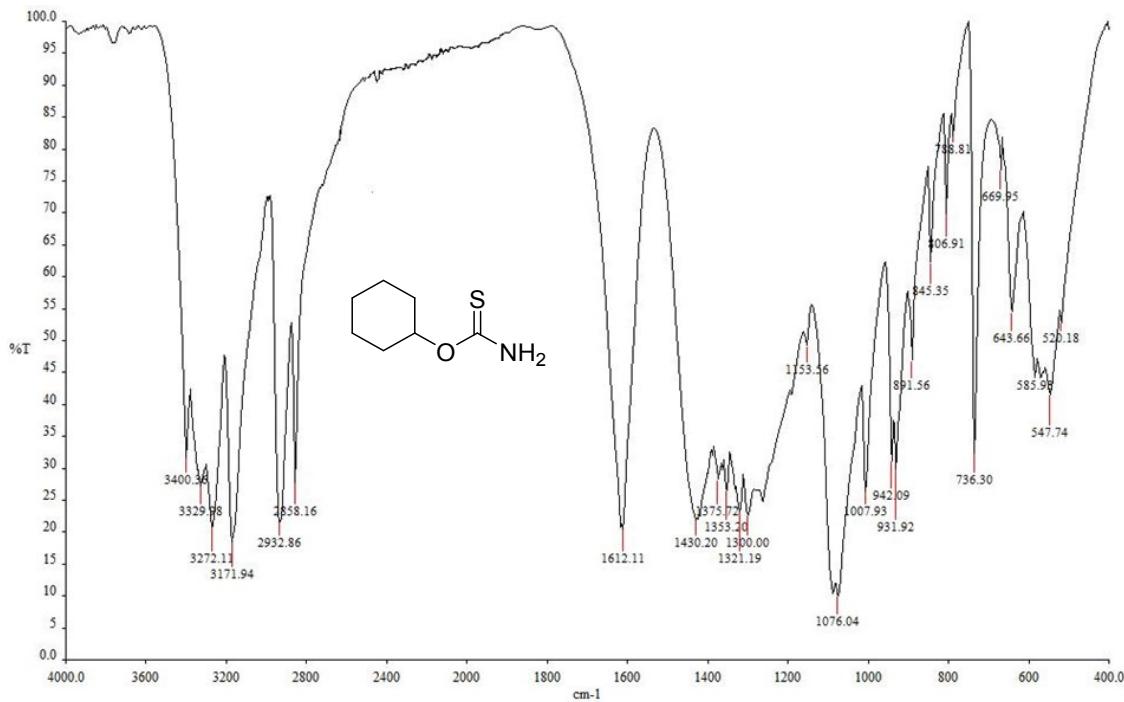
**Figure S60.** FT-IR spectrum of 5b in KBr



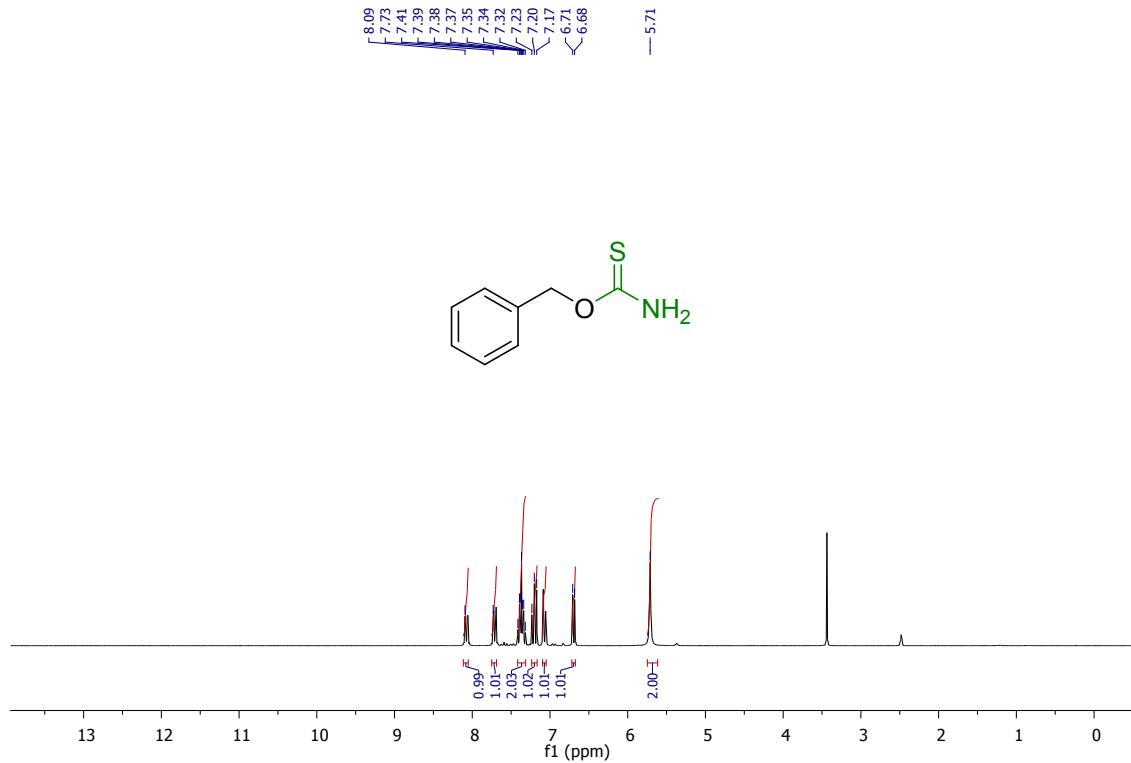
**Figure S61.** <sup>1</sup>H NMR spectrum of 5c in DMSO-d<sub>6</sub> (250MHz)



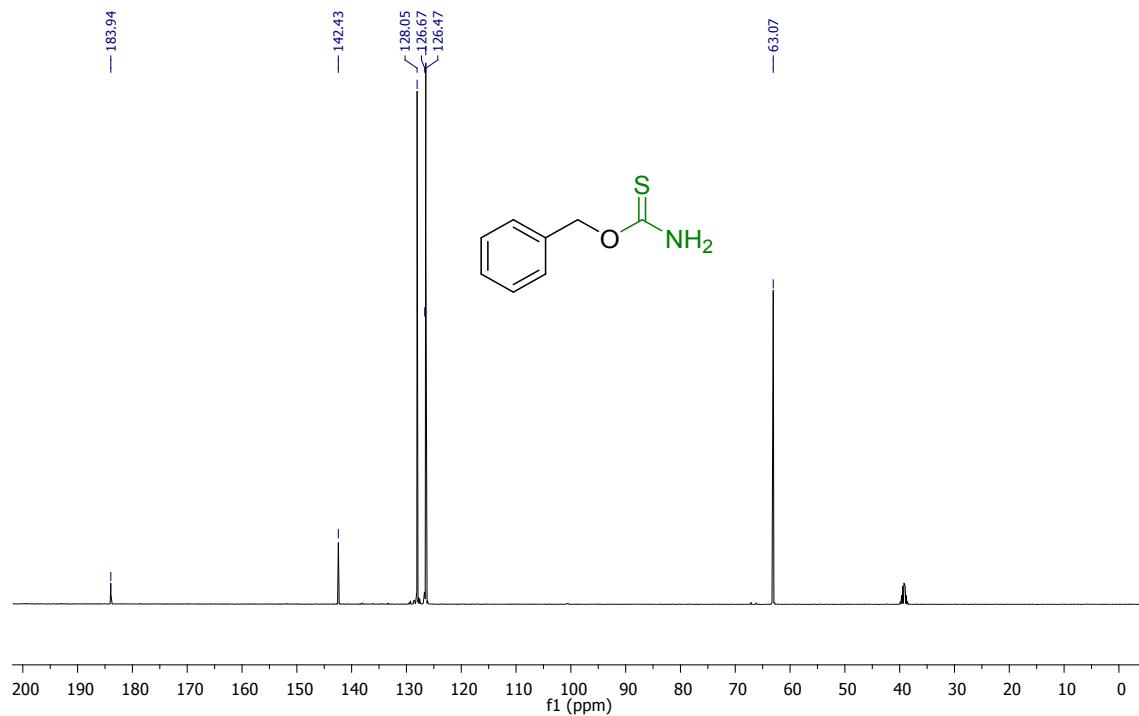
**Figure S62.** <sup>13</sup>C NMR spectrum of 5c in DMSO-d<sub>6</sub> (63MHz)



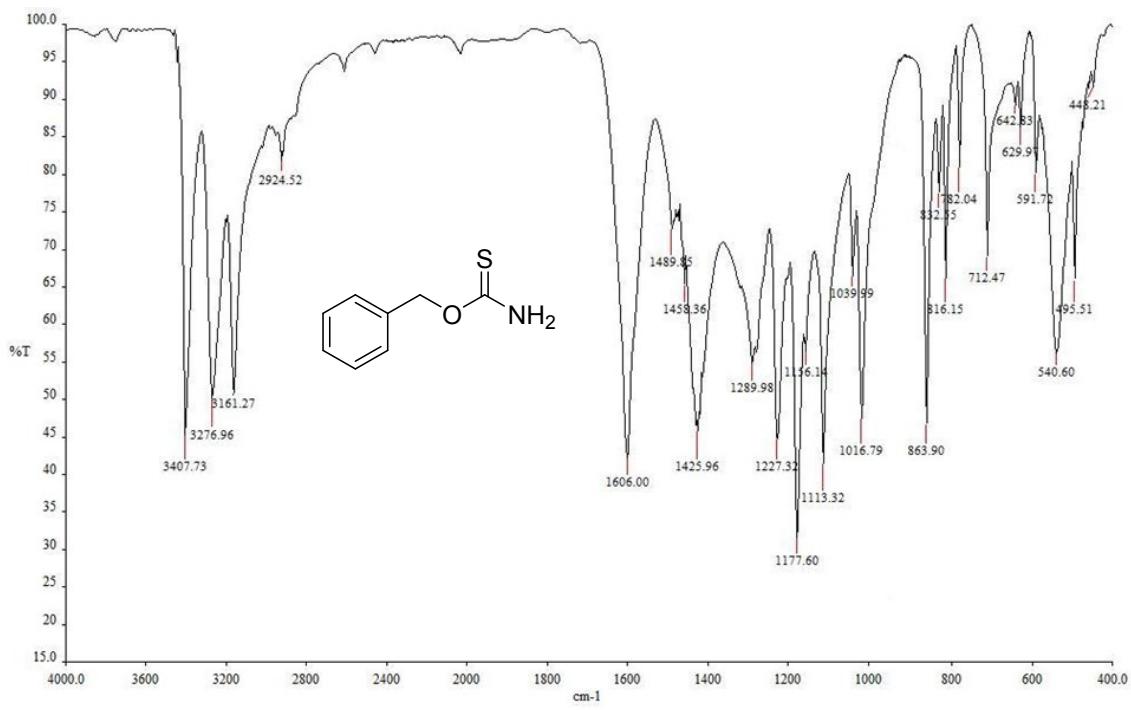
**Figure S63. FT-IR spectrum of 5c in KBr**



**Figure S64.  $^1\text{H}$  NMR spectrum of 5d in  $\text{DMSO}-d_6$  (250MHz)**



**Figure S65.**  $^{13}\text{C}$  NMR spectrum of **5d** in DMSO (101MHz)



**Figure S66.** FT-IR spectrum of **5d** in KBr

— 0.22  
— 0.02

7.40  
7.37  
7.34  
7.23  
7.21  
7.18  
7.06  
7.02

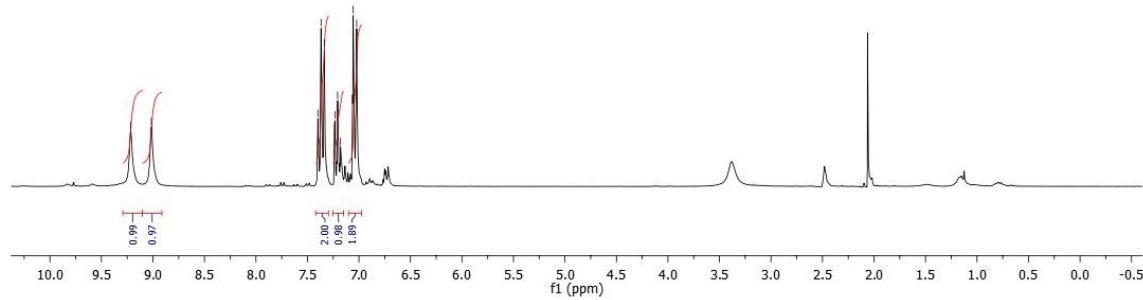
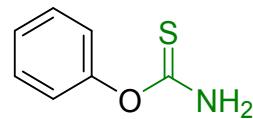


Figure S67.  $^1\text{H}$  NMR spectrum of 5e in  $\text{DMSO}-d_6$  (250MHz).

— 190.36

— 153.16

— 129.02  
— 125.53  
— 122.77

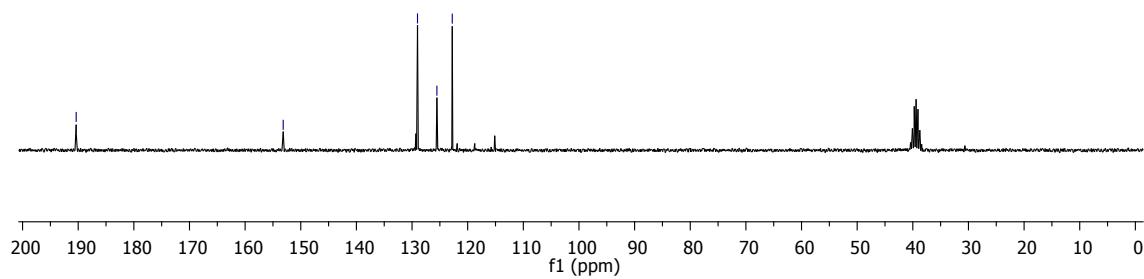
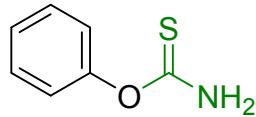
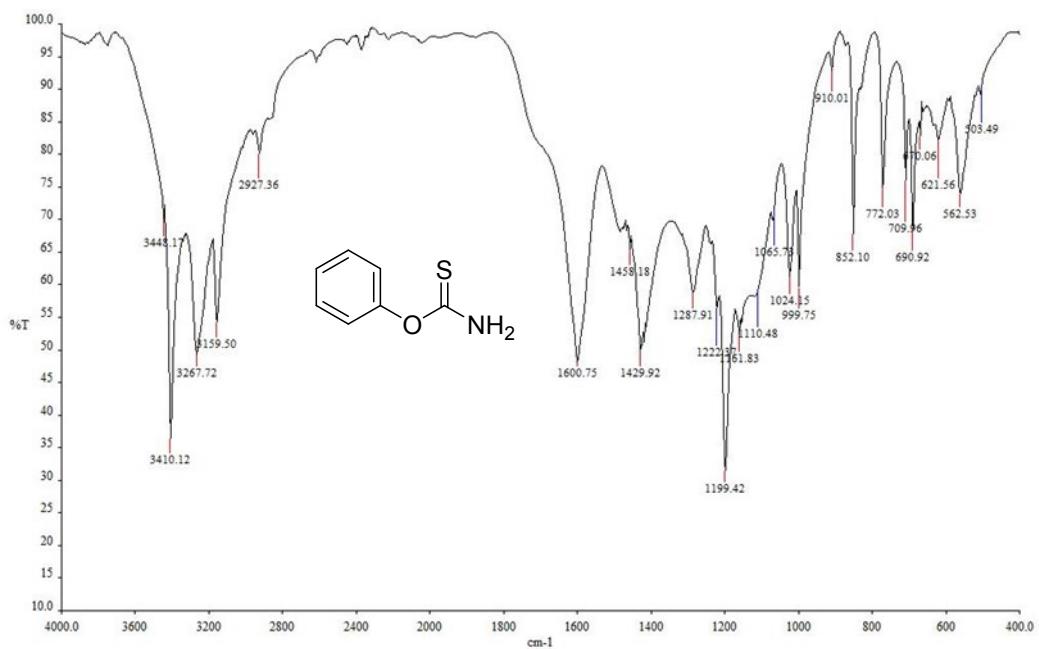
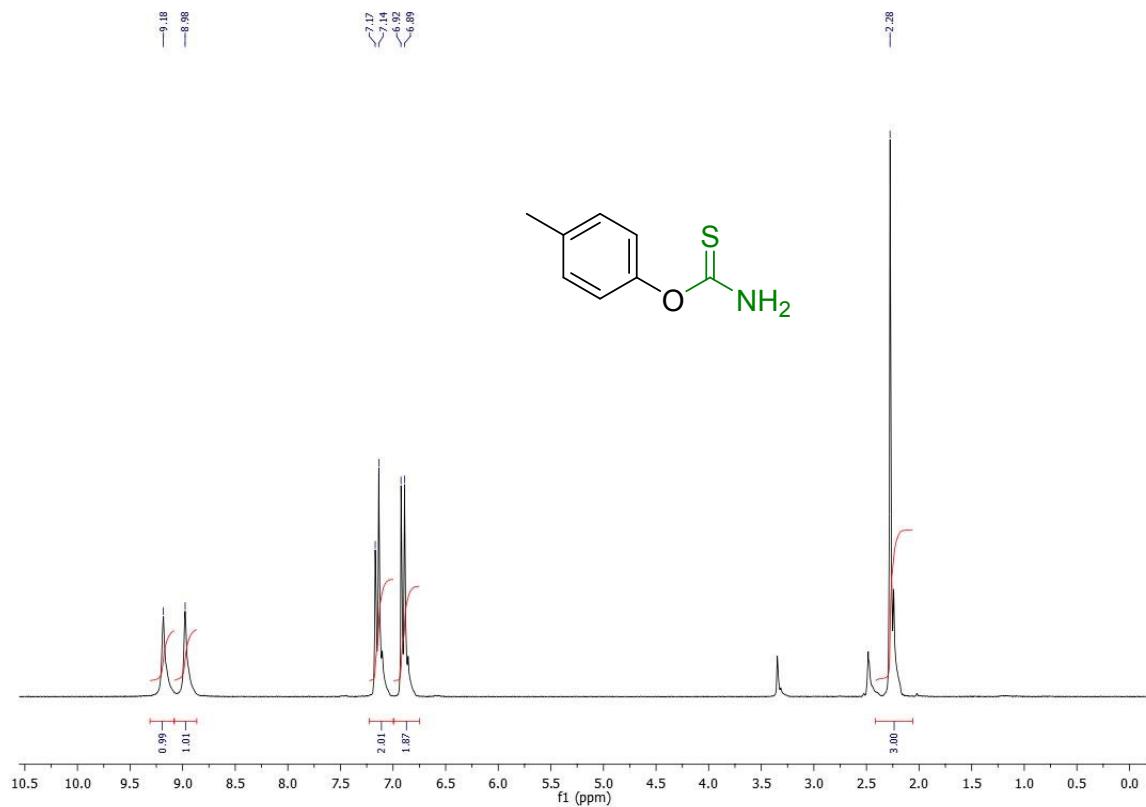


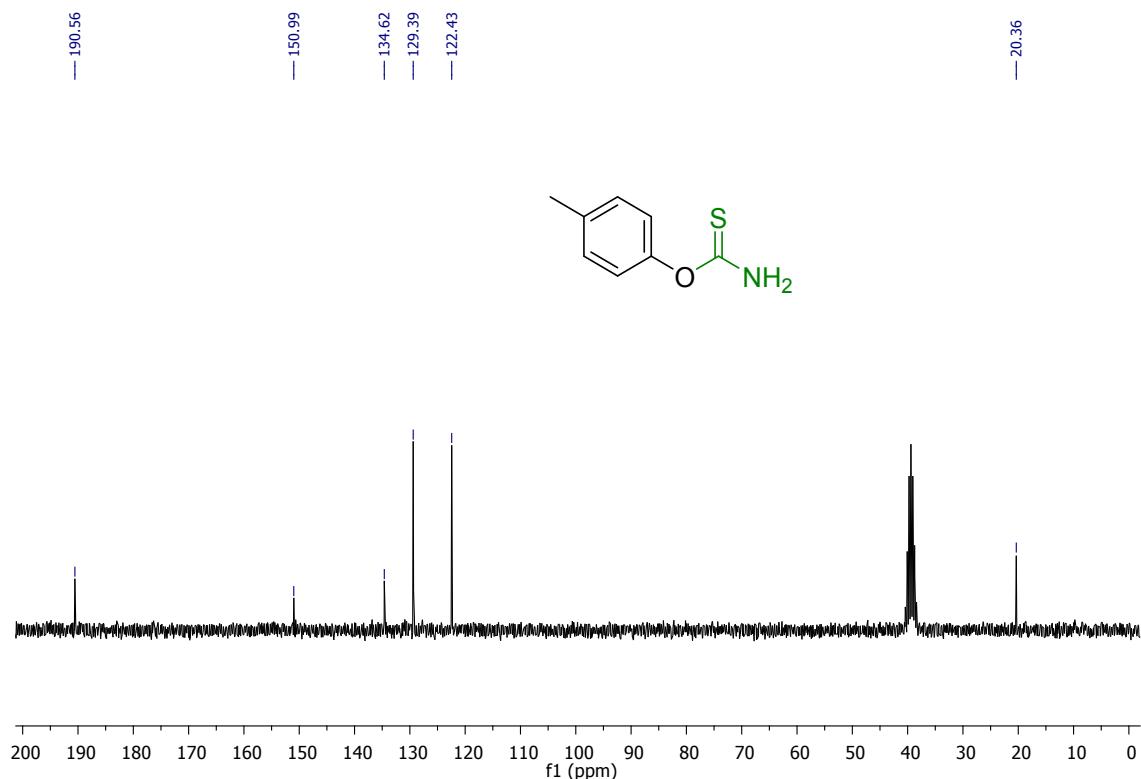
Figure S68.  $^{13}\text{C}$  NMR spectrum of 5e in  $\text{DMSO}-d_6$  (63MHz)



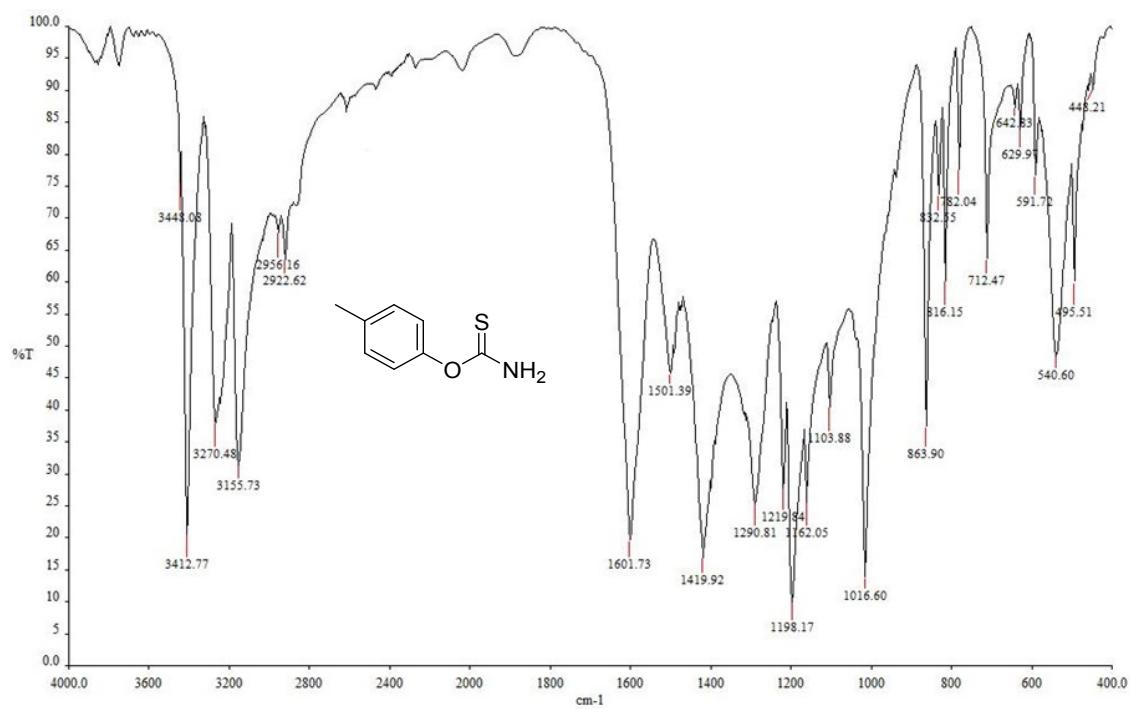
**Figure S69.** FT-IR spectrum of **5e** in kBr



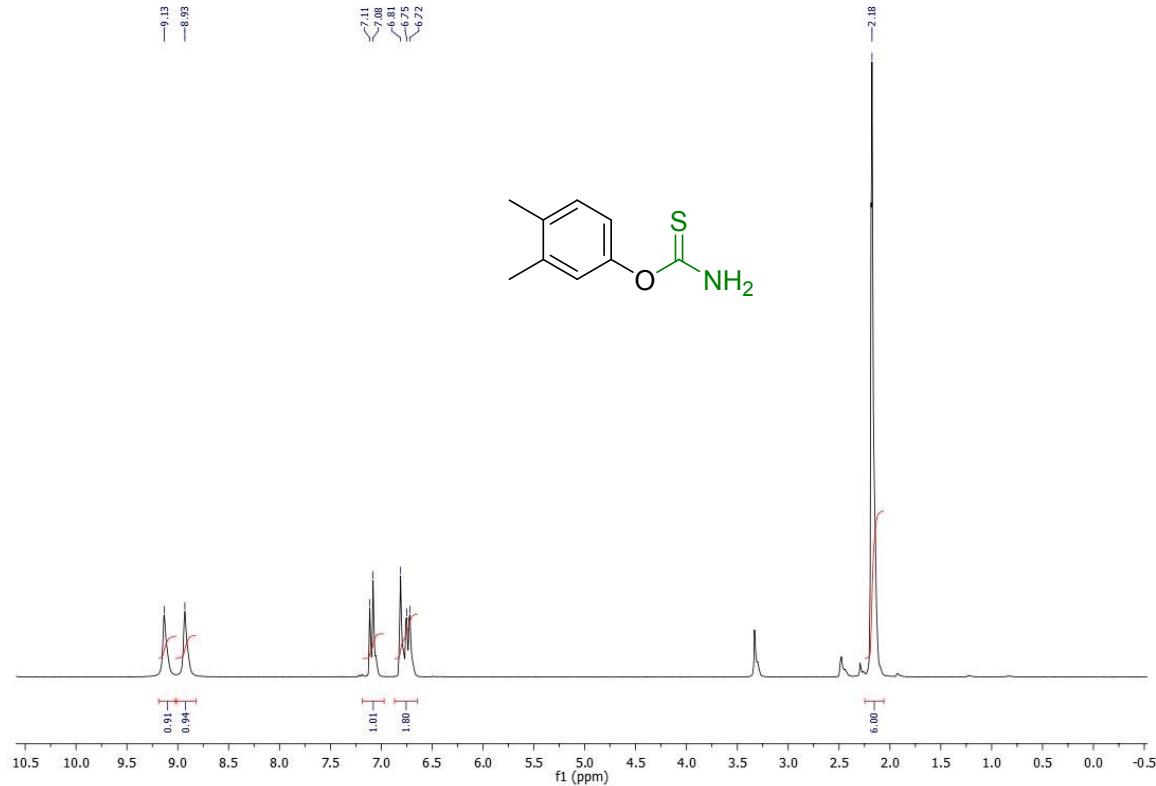
**Figure S70.**  $^1\text{H}$  NMR spectrum of **5f** in  $\text{DMSO}-d_6$  (250MHz)



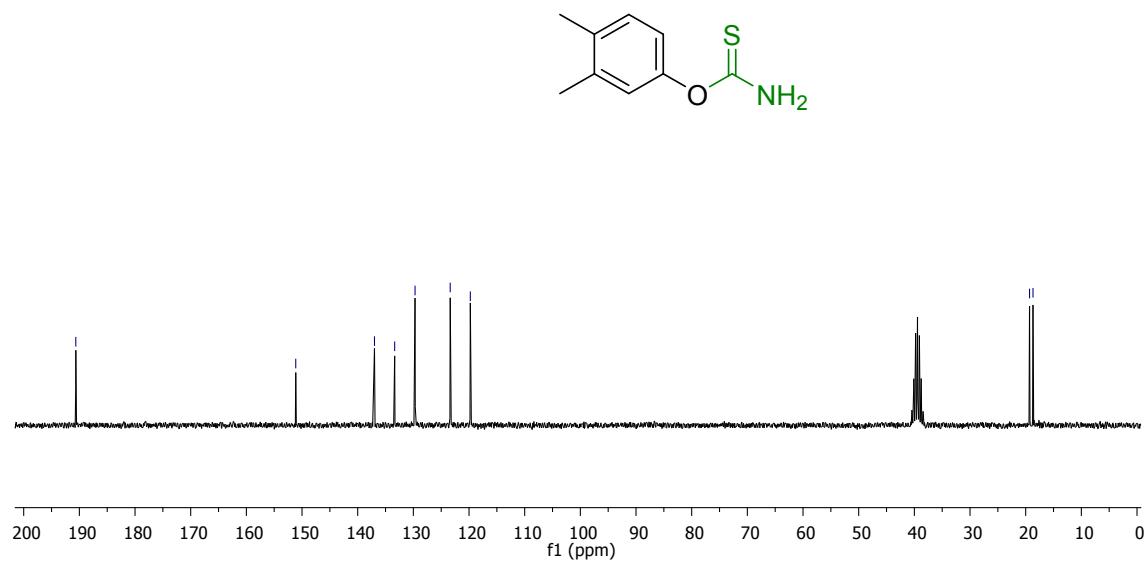
**Figure S71.**  $^{13}\text{C}$  NMR spectrum of 5f in  $\text{DMSO}-d_6$  (63MHz)



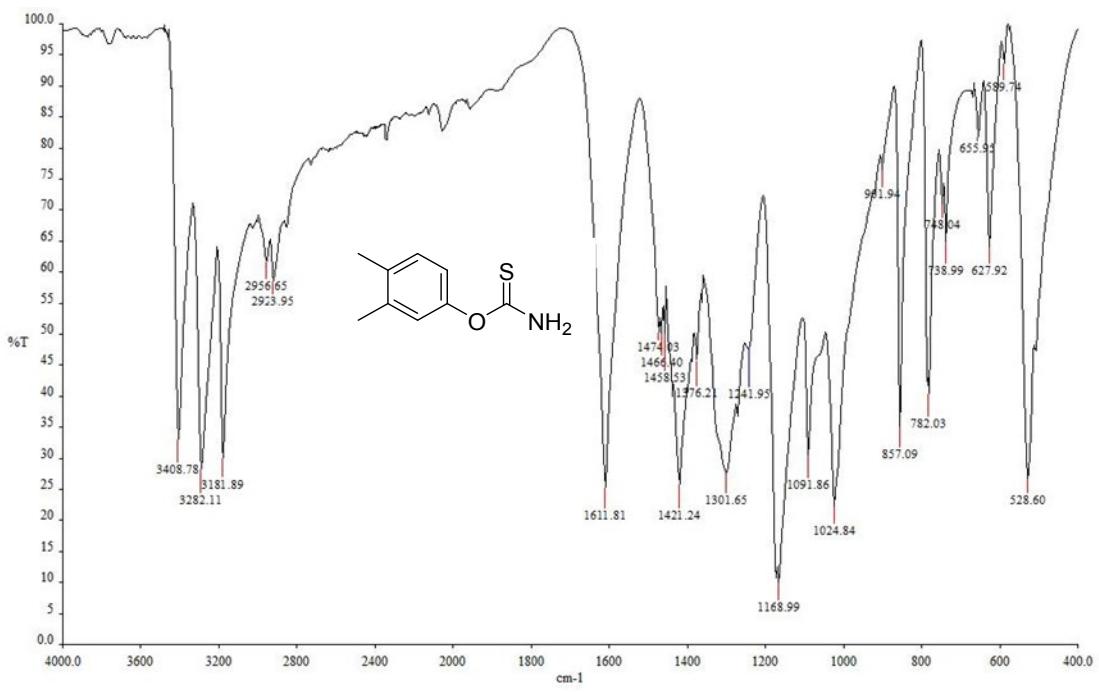
**Figure S72.** FT-IR spectrum of 5f in KBr



**Figure S73.** <sup>1</sup>H NMR spectrum of 5g in DMSO-d<sub>6</sub> (250MHz)

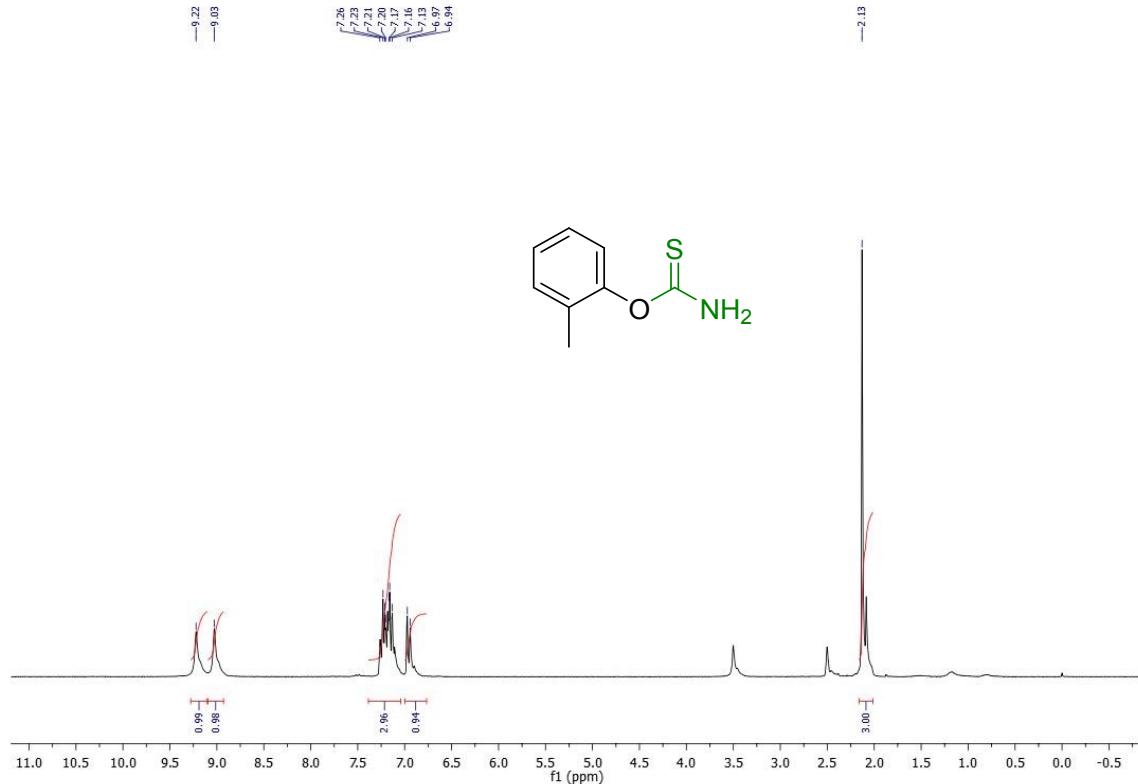


**Figure S74.** <sup>13</sup>C NMR spectrum of 5g in DMSO-d<sub>6</sub> (63MHz)

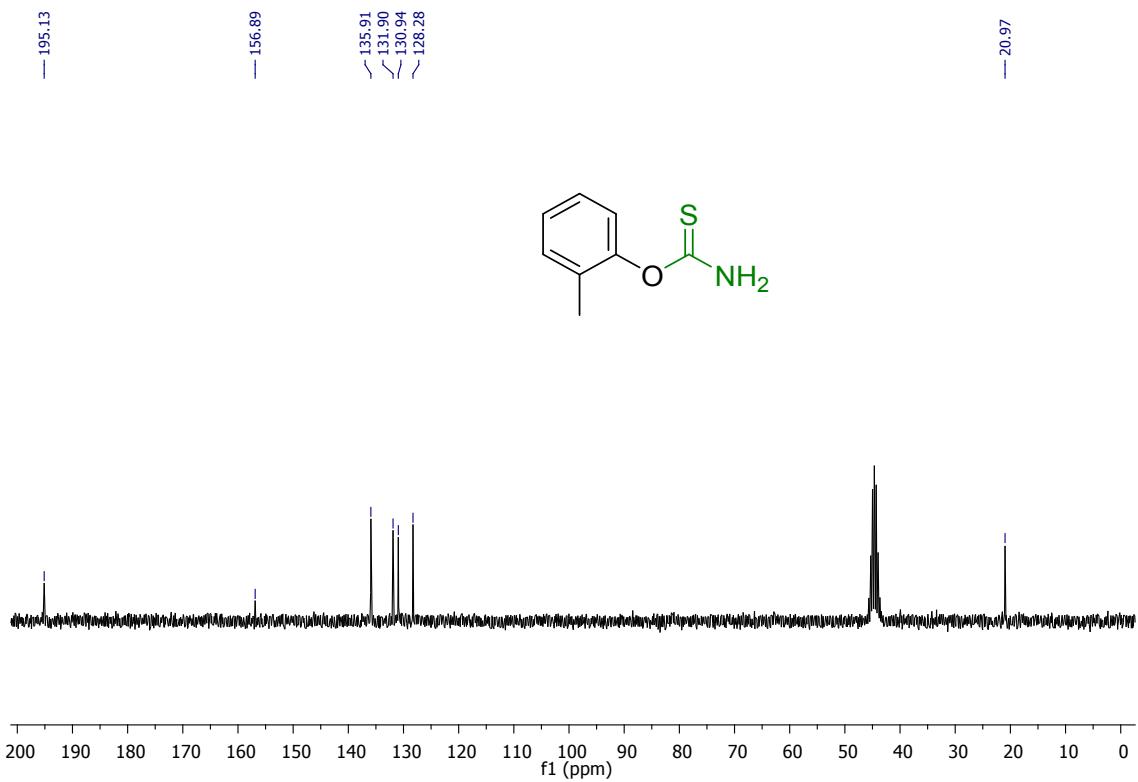


ure S75. FT-IR spectrum of 5g in KBr

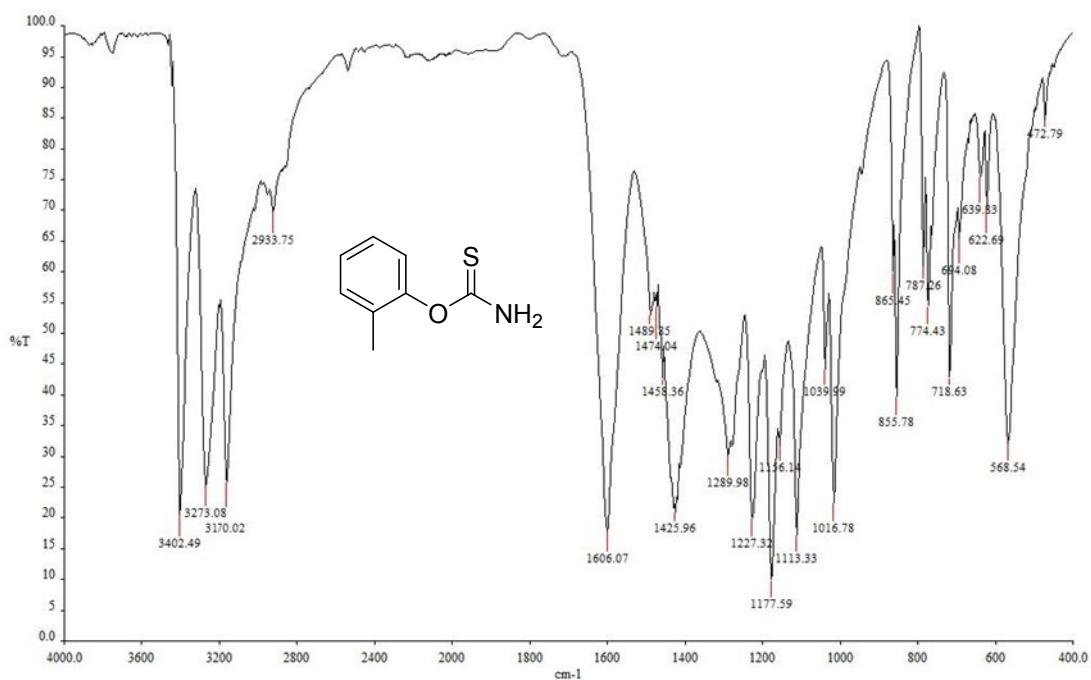
Fig



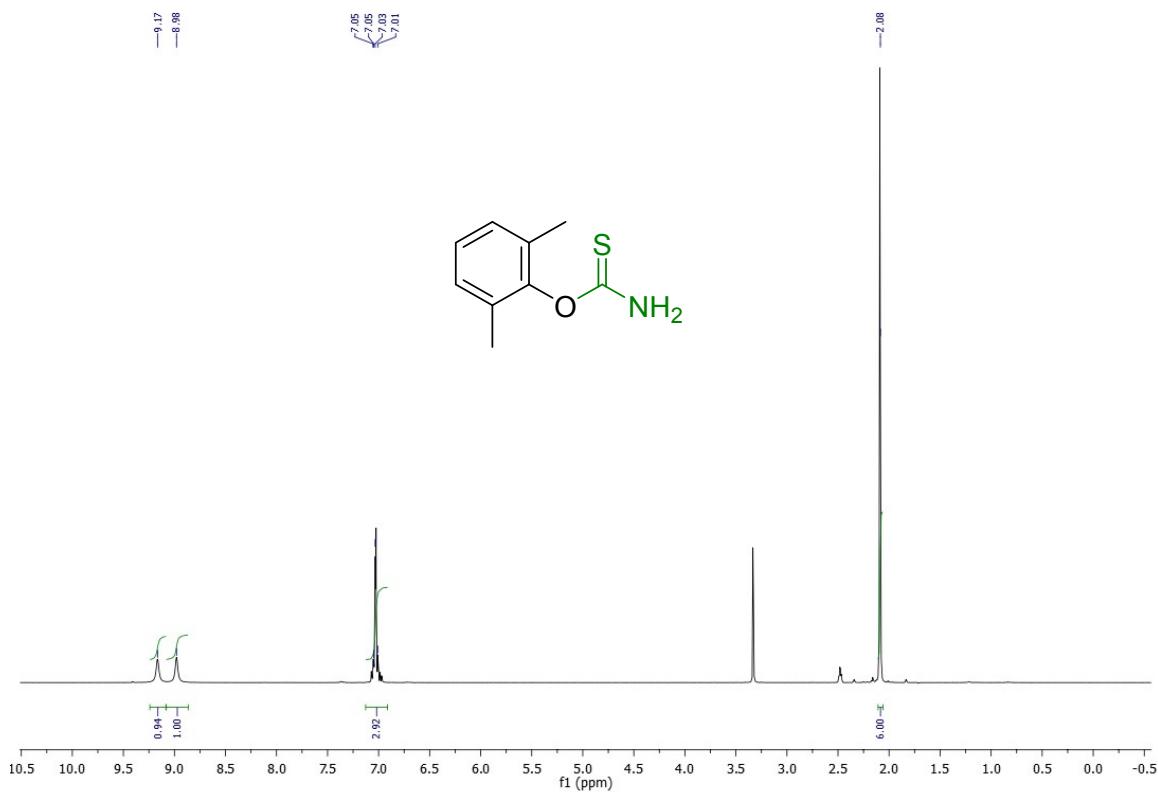
**Figure S76.**  $^1\text{H}$  NMR spectrum of 5h in  $\text{DMSO}-d_6$  (250MHz)



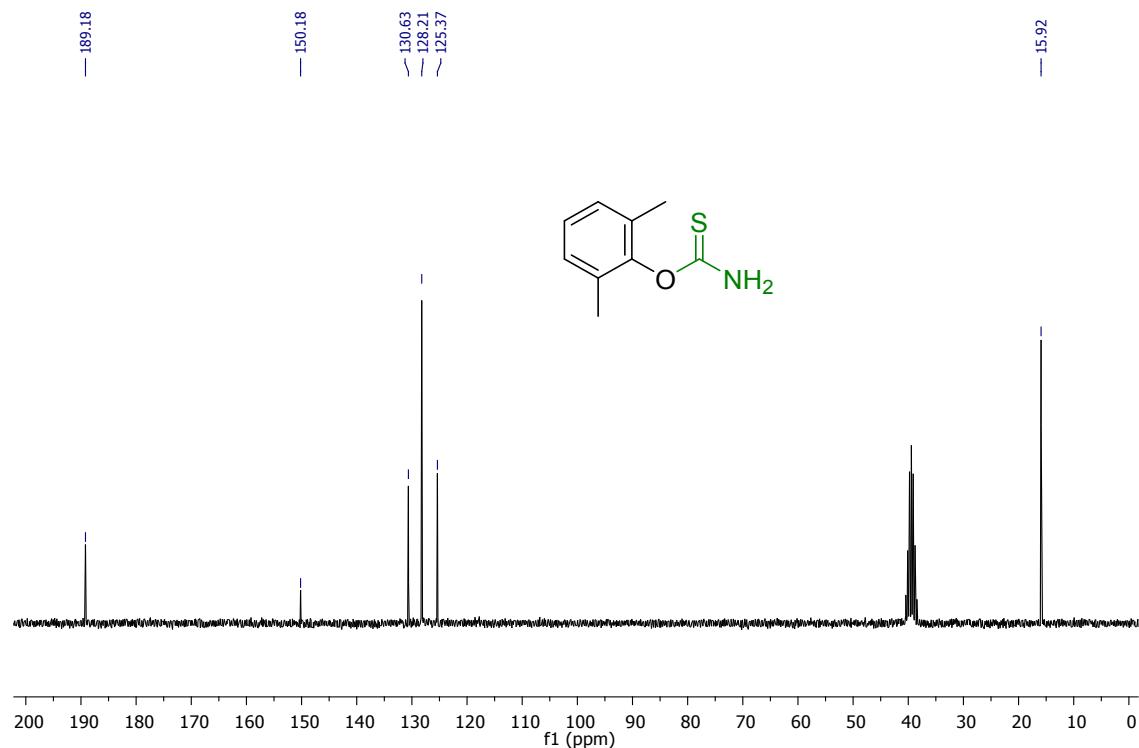
**Figure S77.**  $^{13}\text{C}$  NMR spectrum of 5h in  $\text{DMSO}-d_6$  (63MHz)



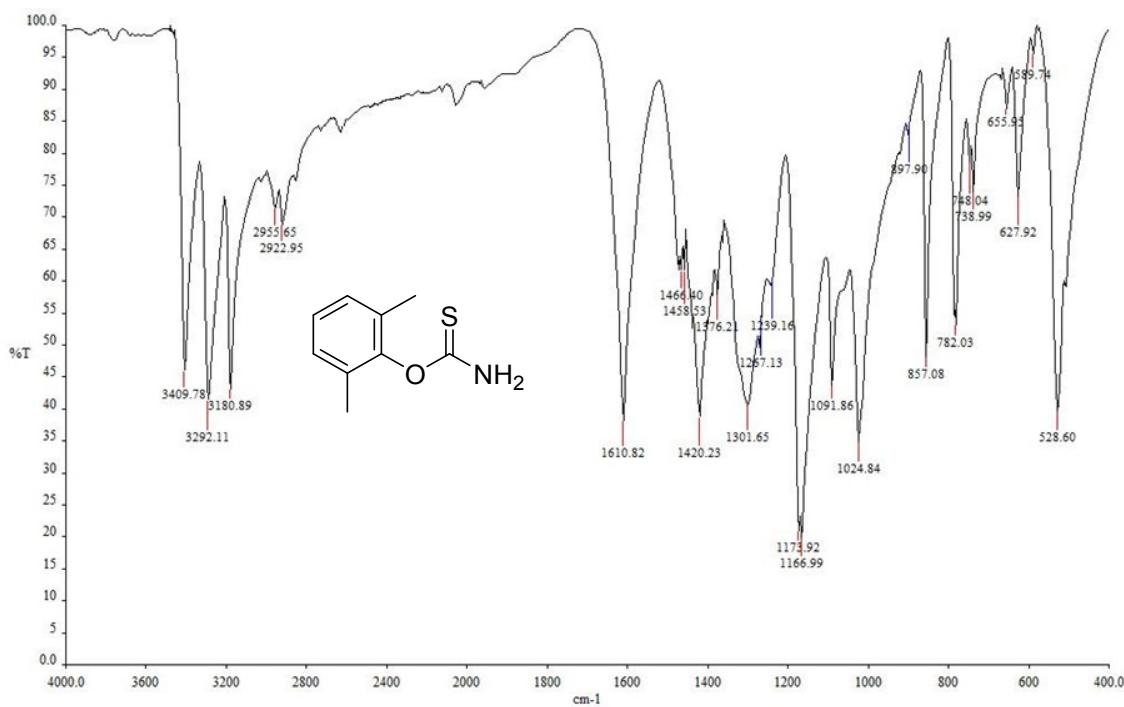
**Figure S78. FT-IR spectrum of 5h in KBr**



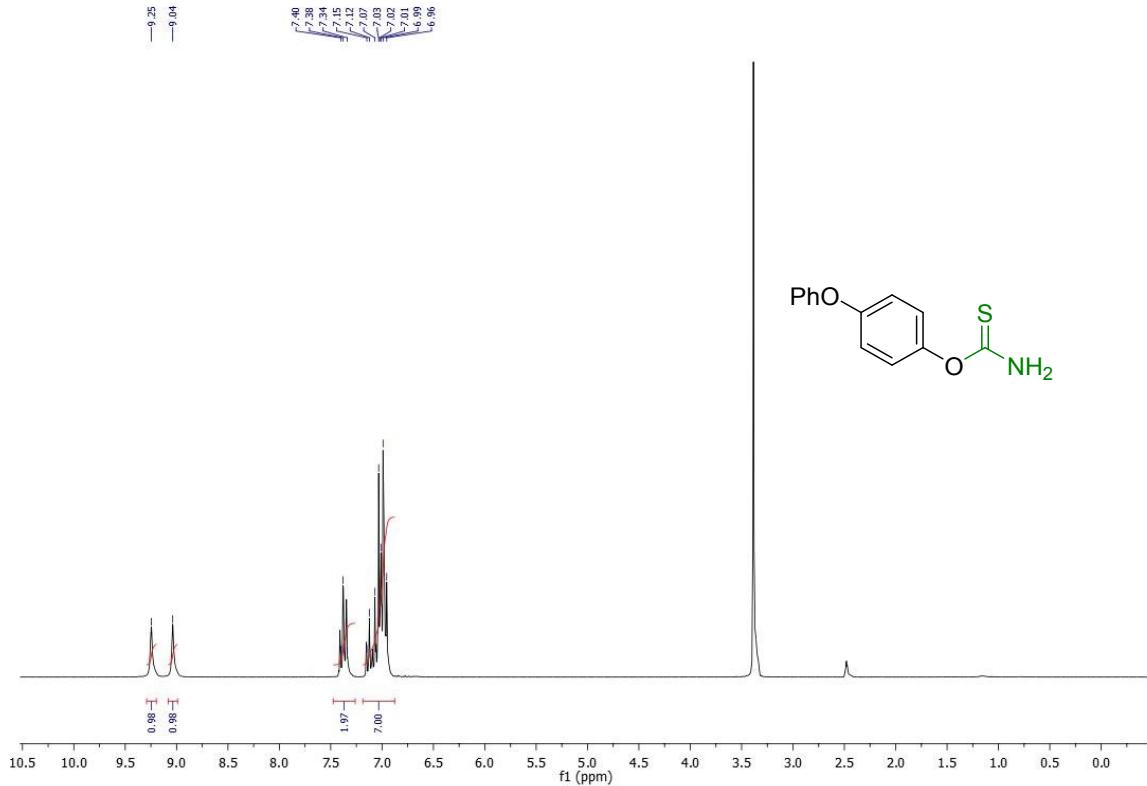
**Figure S79.  $^1\text{H}$  NMR spectrum of 5i in  $\text{DMSO}-d_6$  (250MHz)**



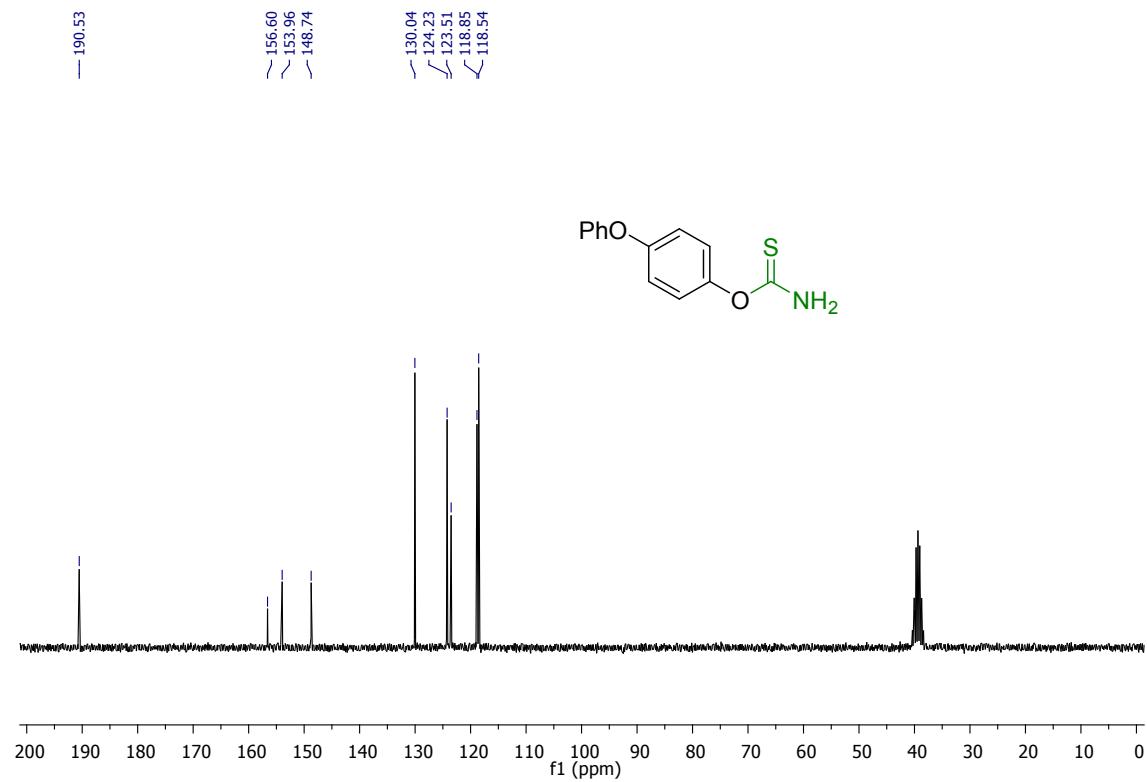
**Figure S80.**  $^{13}\text{C}$  NMR spectrum of 5i in  $\text{DMSO}-d_6$  (63MHz)



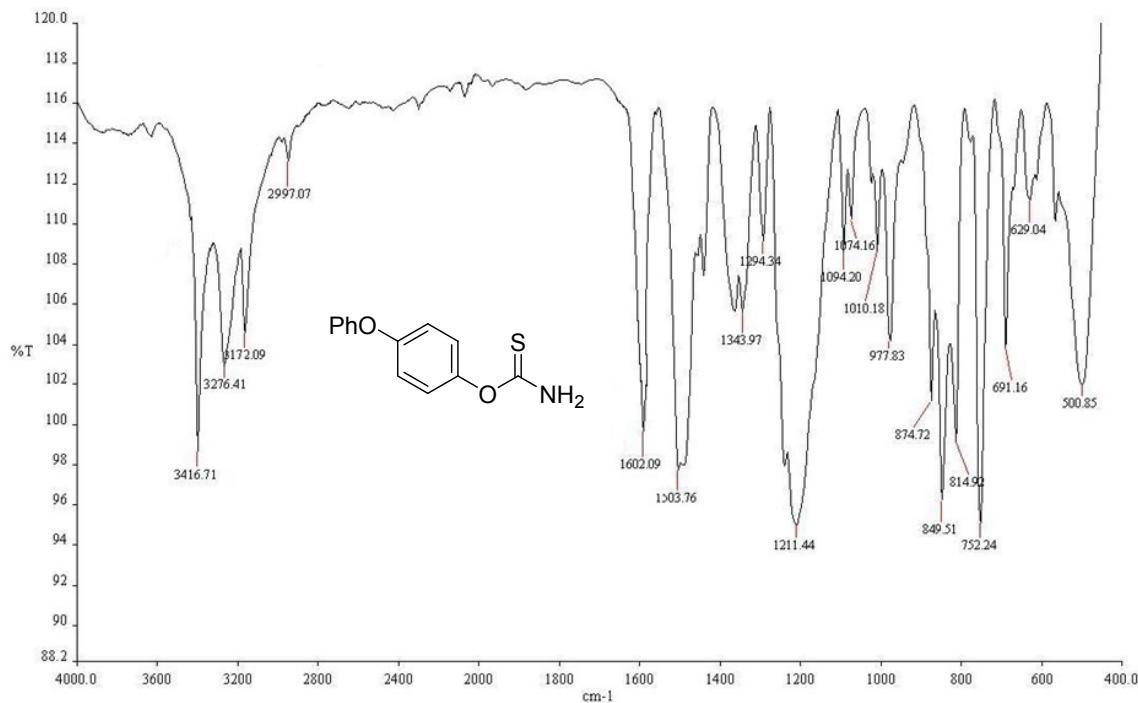
**Figure S81.** FT-IR spectrum of 5i in KBr



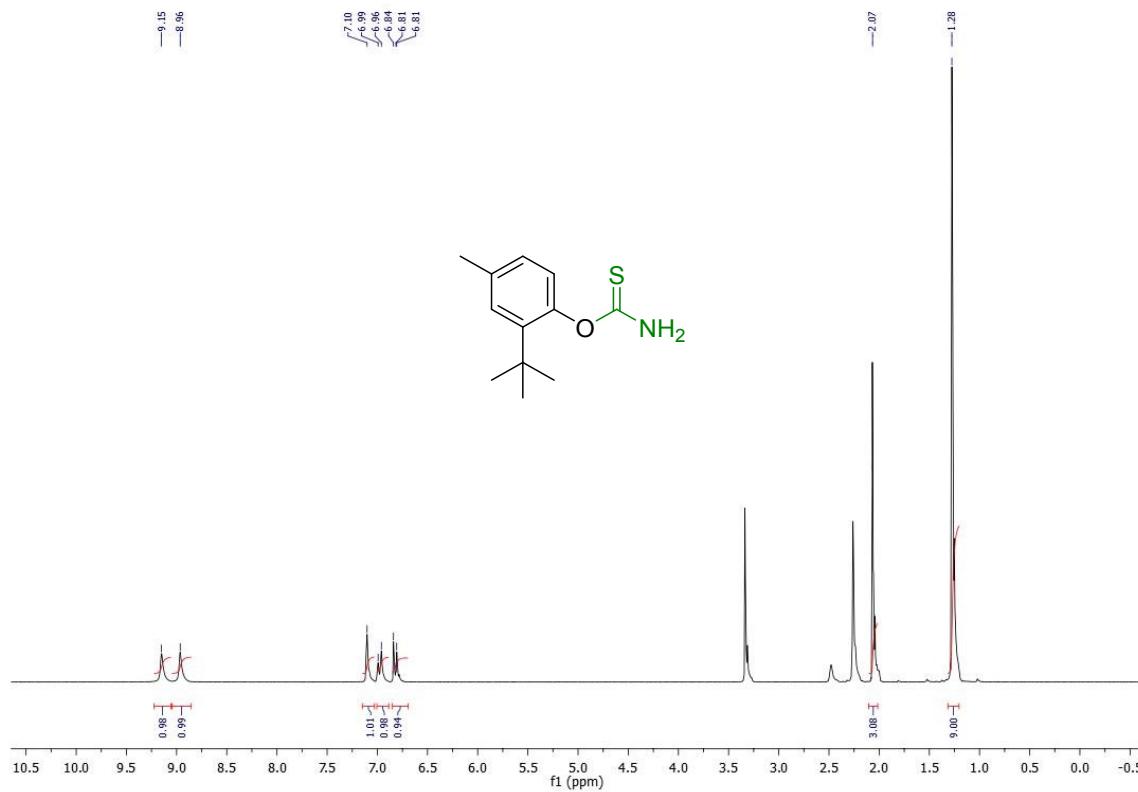
**Figure S82.**  $^1\text{H}$  NMR spectrum of **5j** in  $\text{DMSO}-d_6$  (250MHz)



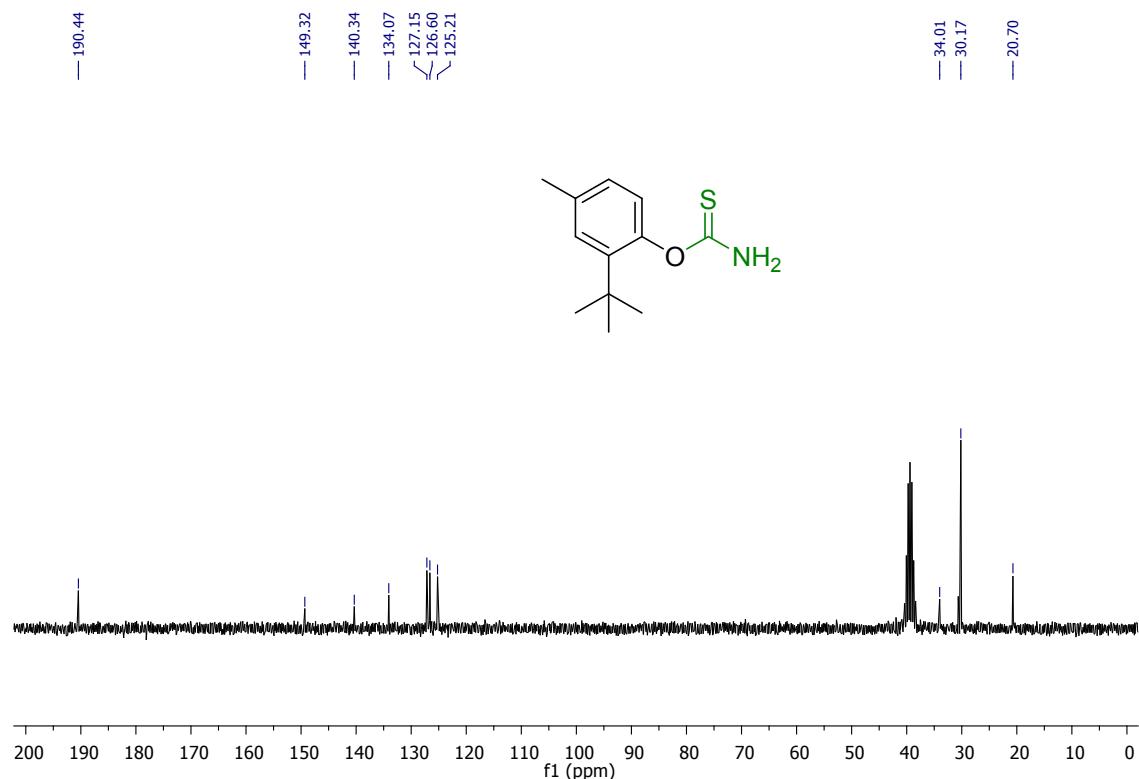
**Figure S83.**  $^{13}\text{C}$  NMR spectrum of **5j** in  $\text{DMSO}-d_6$  (63MHz)



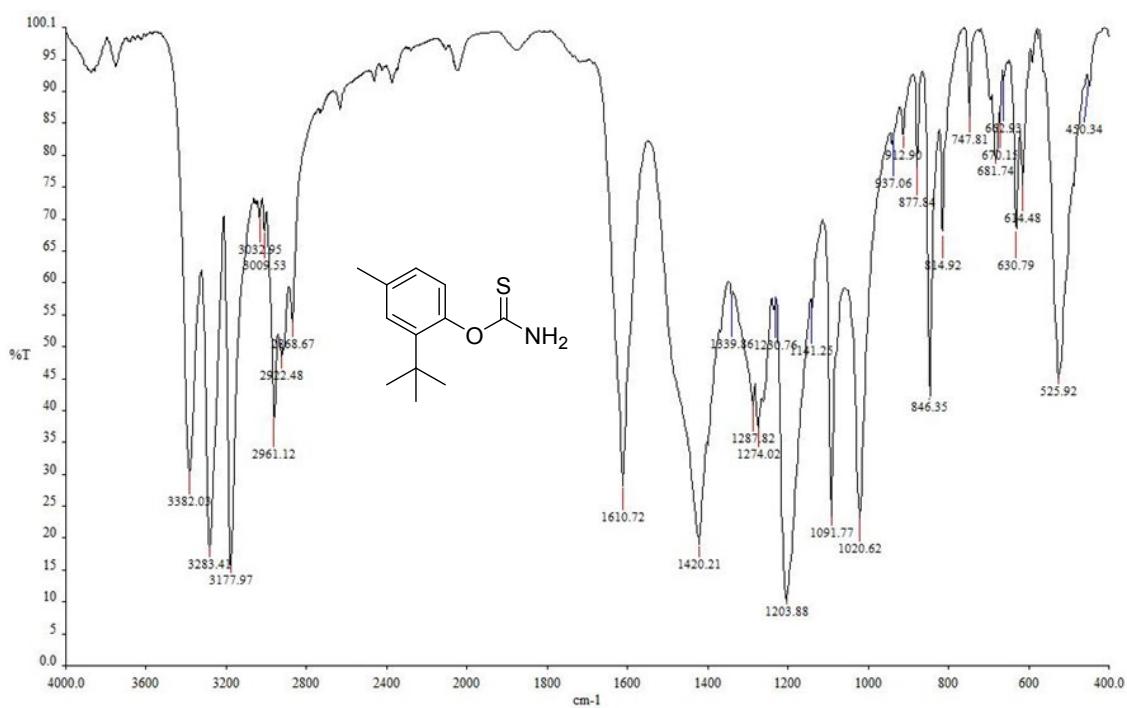
**Figure S84.** FT-IR spectrum of 5j in KBr



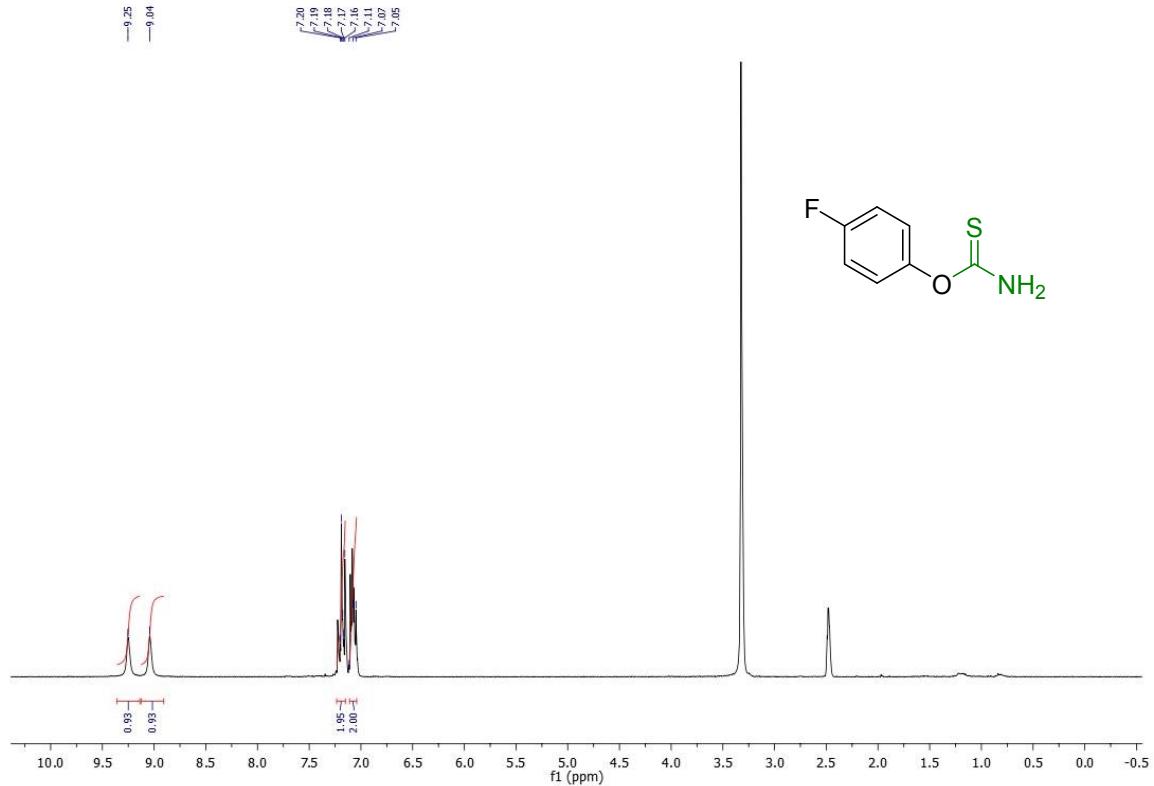
**Figure S85.**  $^1\text{H}$  NMR spectrum of 5k in  $\text{DMSO}-d_6$  (250MHz)



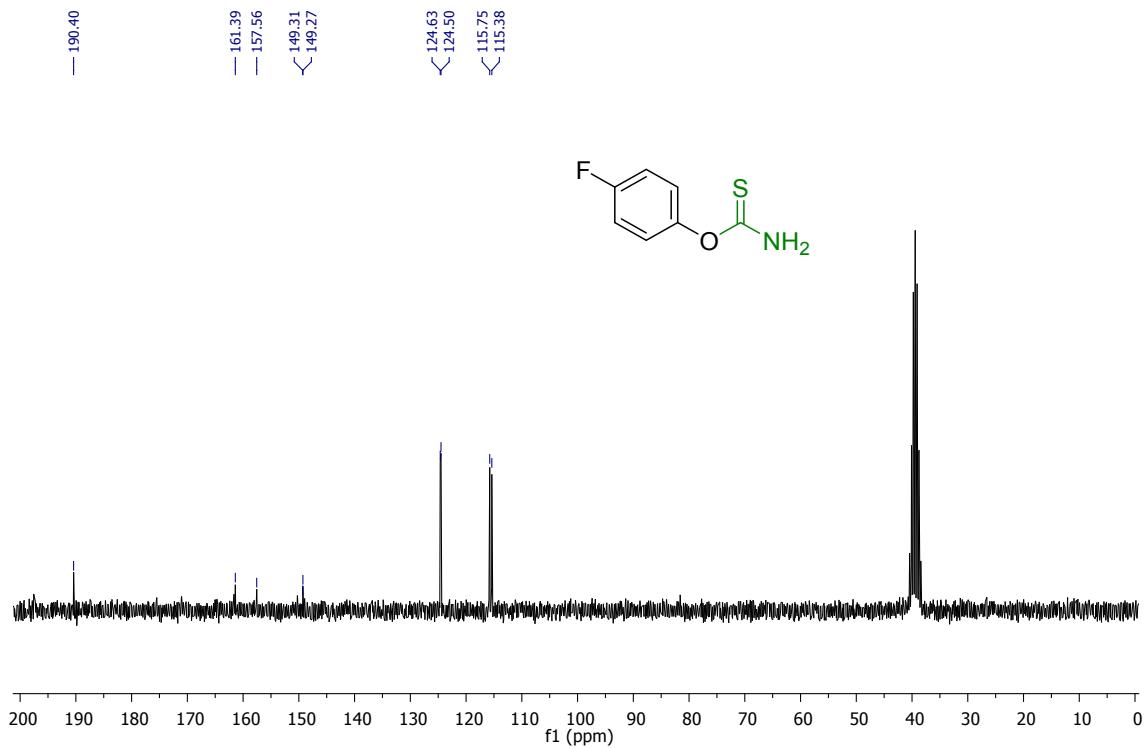
**Figure S86.** <sup>13</sup>C NMR spectrum of 5k in DMSO-d<sub>6</sub> (63MHz)



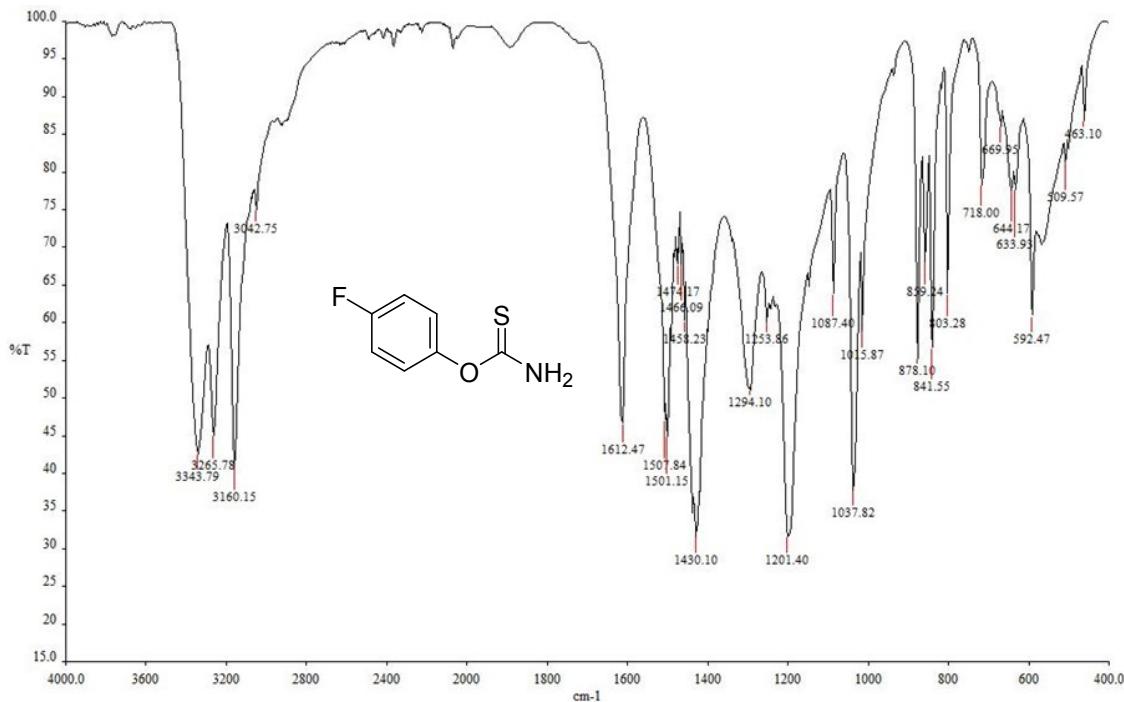
**Figure S87.** FT-IR spectrum of 5k in KBr



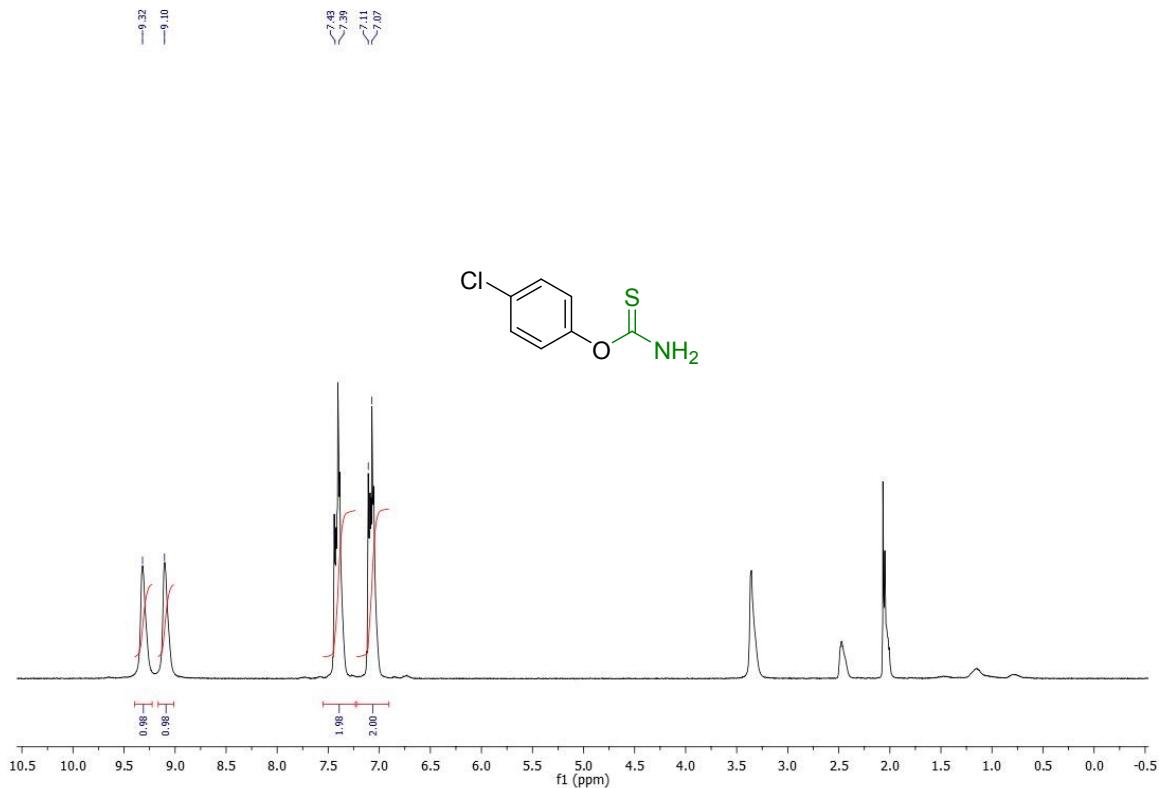
**Figure S88.**  $^1\text{H}$  NMR spectrum of 5l in  $\text{DMSO}-d_6$  (250MHz)



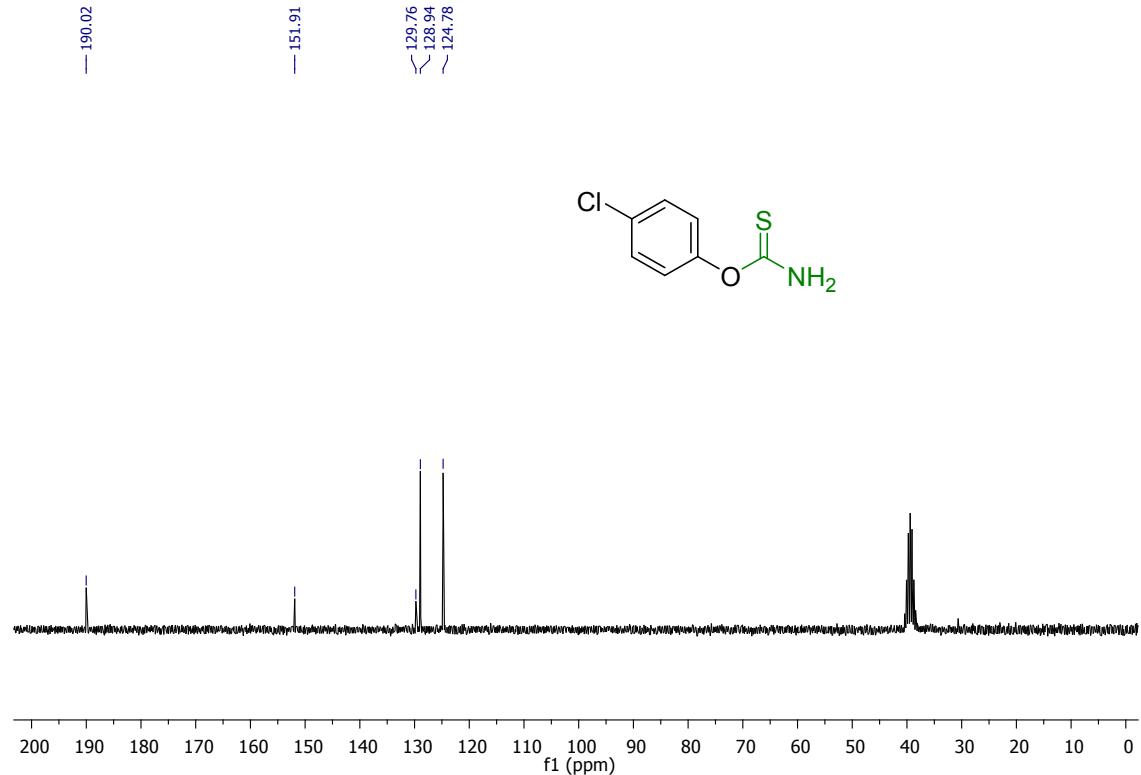
**Figure S89.**  $^{13}\text{C}$  NMR spectrum of 5l in  $\text{DMSO}-d_6$  (63MHz)



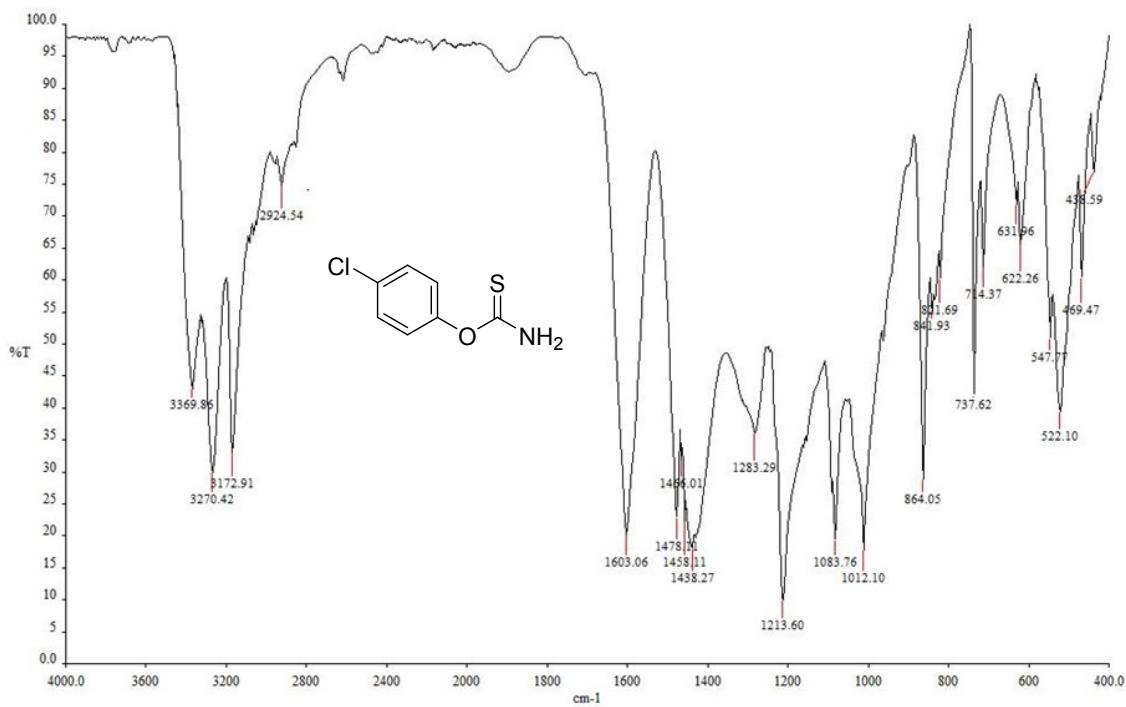
**Figure S90. FT-IR spectrum of 5l in KBr**



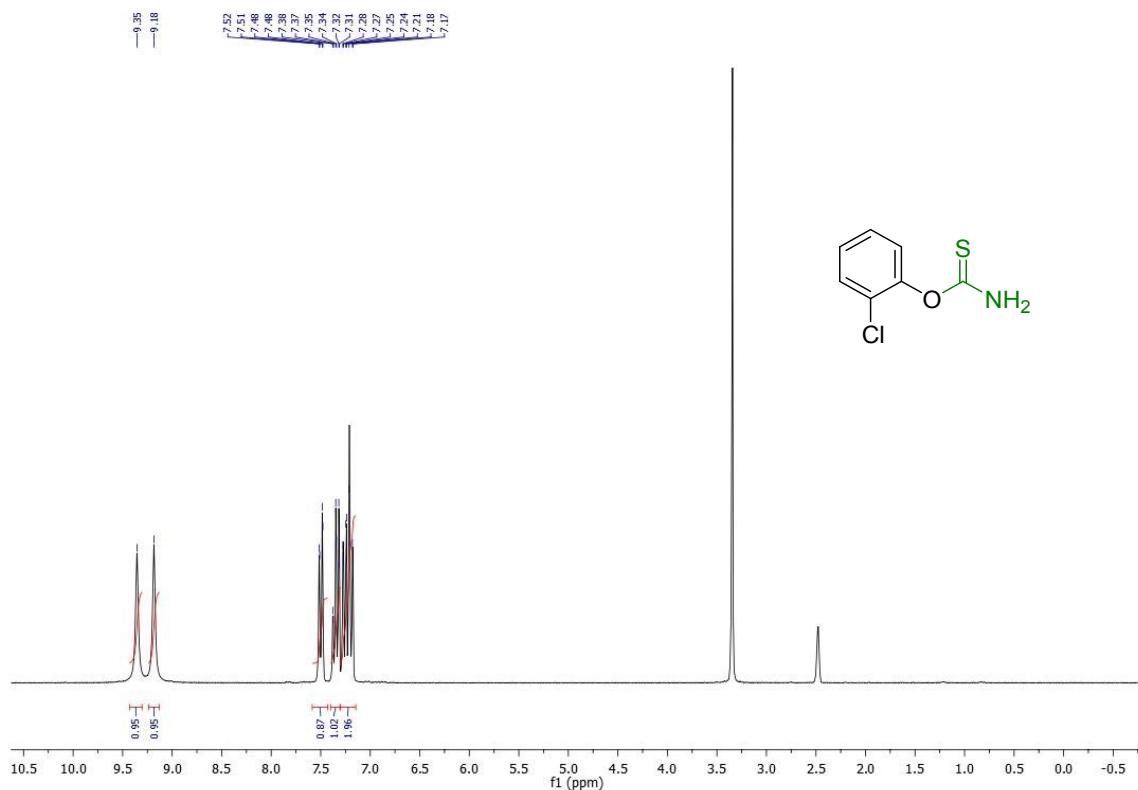
**Figure S91.  $^1\text{H}$  NMR spectrum of 5m in  $\text{DMSO}-d_6$  (250MHz)**



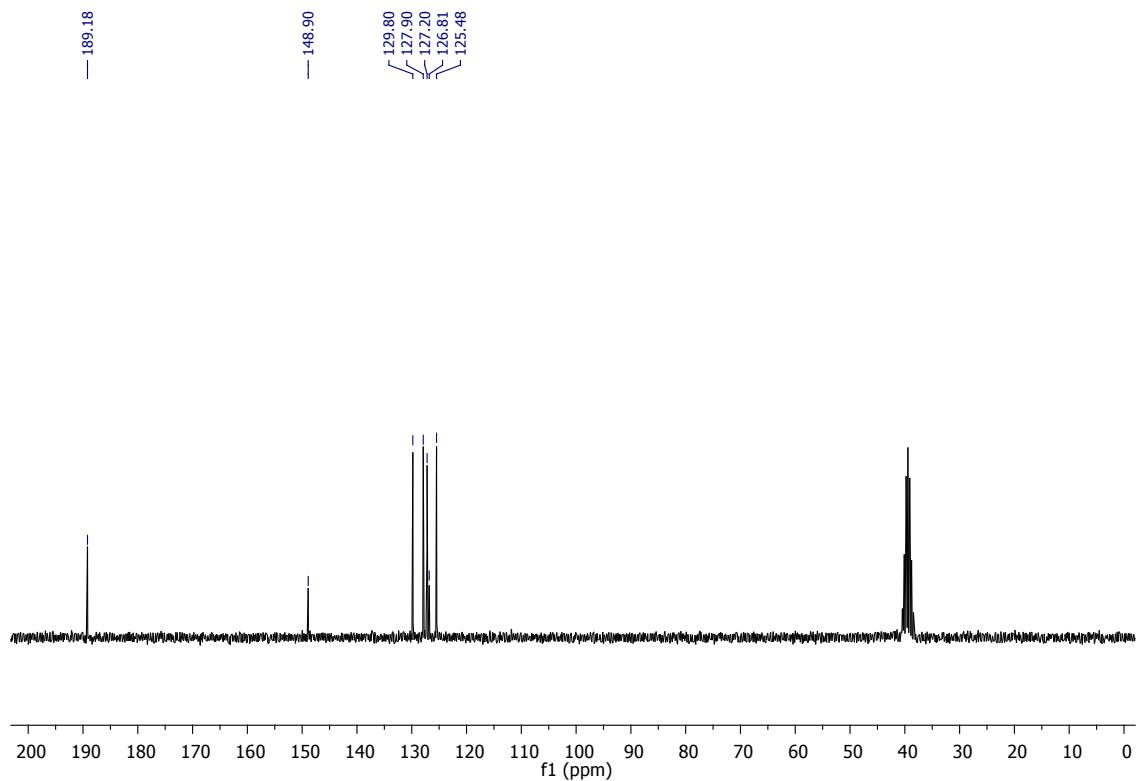
**Figure S92.**  $^{13}\text{C}$  NMR spectrum of 5m in  $\text{DMSO}-d_6$  (63MHz)



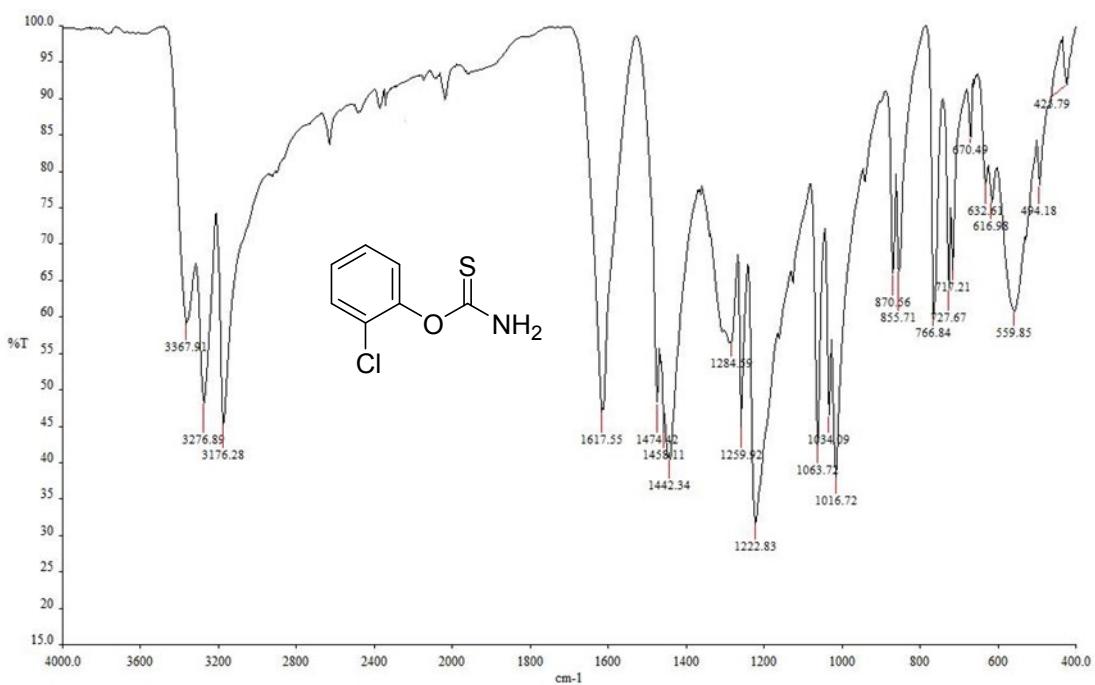
**Figure S93.** FT-IR spectrum of 5m in KBr



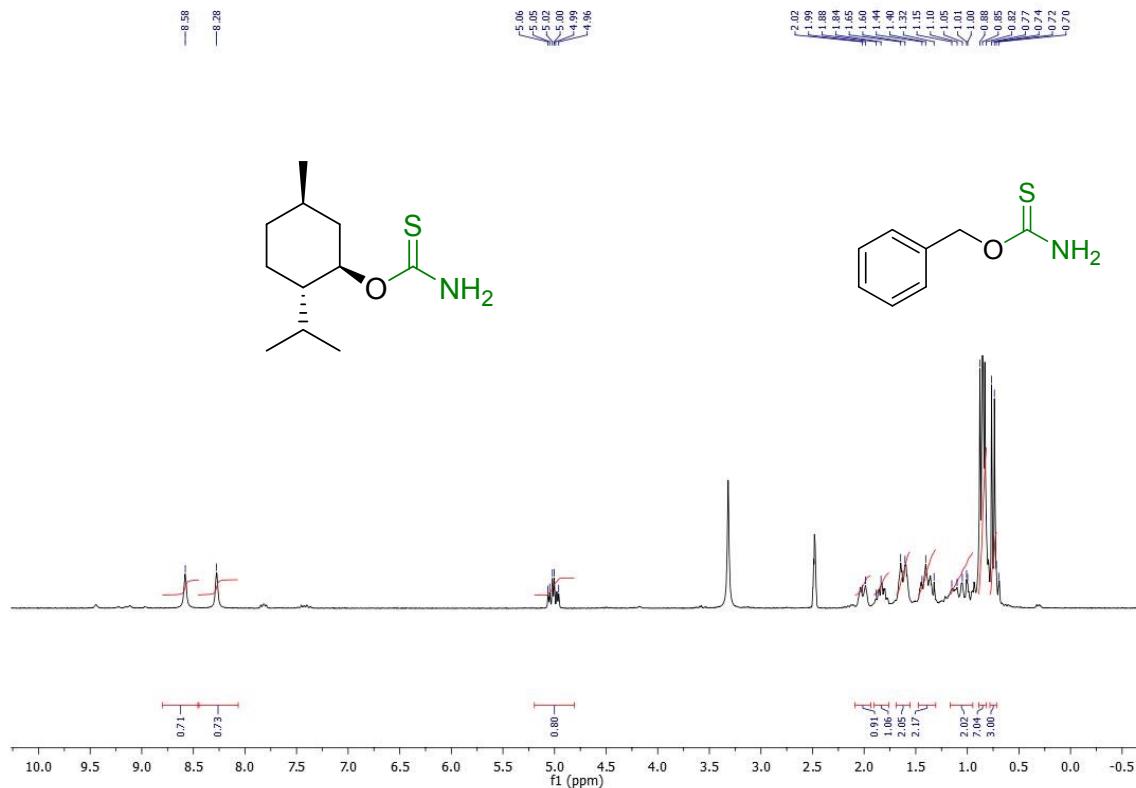
**Figure S94.**  $^1\text{H}$  NMR spectrum of **5n** in  $\text{DMSO}-d_6$  (250MHz)



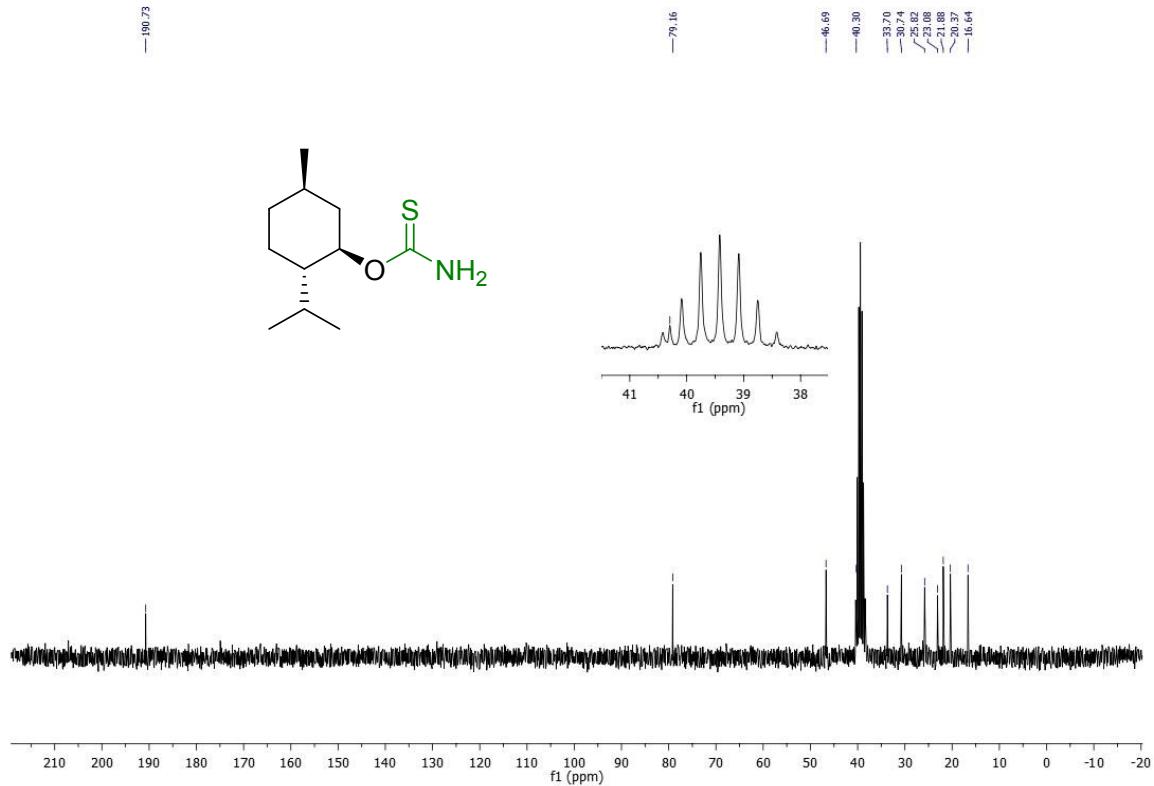
**Figure S95.**  $^{13}\text{C}$  NMR spectrum of **5n** in  $\text{DMSO}-d_6$  (63MHz)



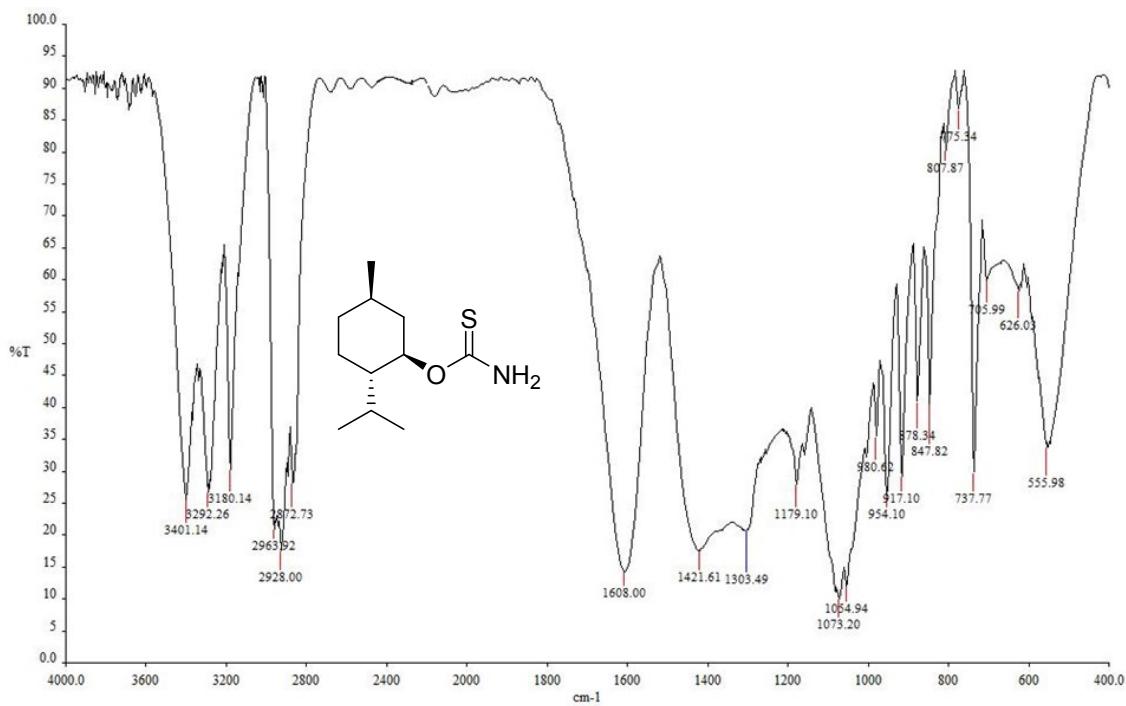
**Figure S96. FT-IR spectrum of 5n in KBr**



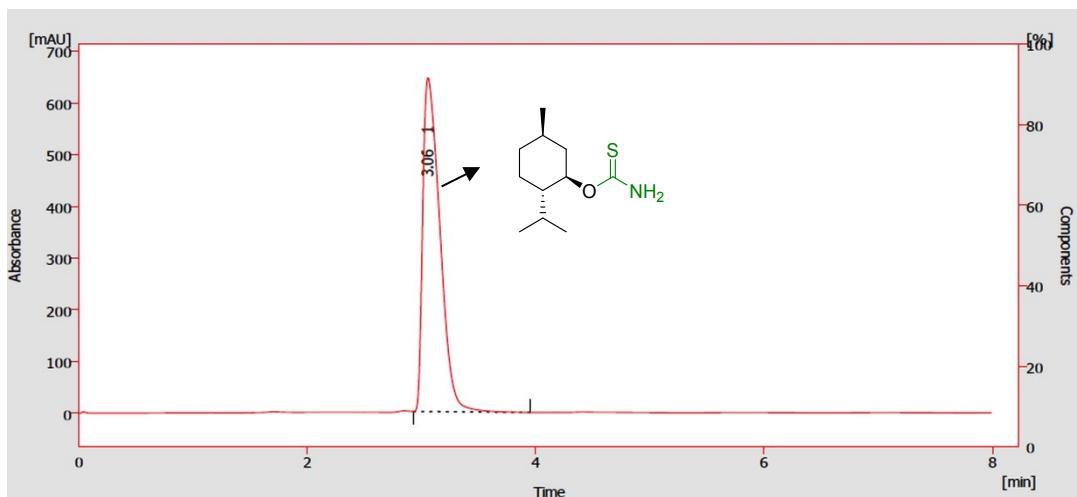
**Figure S97.  $^1\text{H}$  NMR spectrum of 5o in  $\text{DMSO}-d_6$  (250MHz)**



**Figure S98.**  $^{13}\text{C}$  NMR spectrum of 5o in  $\text{DMSO}-d_6$  (63MHz)



**Figure S99.** FT-IR spectrum of 5o in KBr



	Reten. Time [min]	Start Time [min]	End Time [min]	Start Value [mAU]	End Value [mAU]	Area [mAU.s]	Height [mAU]	Area [%]	Height [%]	W05 [min]
1	3.057	2.930	3.953	3.345	1.486	6735.375	645.649	100.0	100.0	0.17
Total						6735.375	645.649	100.0	100.0	

Figure S100. HPLC spectrum of 5o in  $\text{H}_2\text{O}:\text{CH}_3\text{CN}$  (30:70%) at 254 (nm).

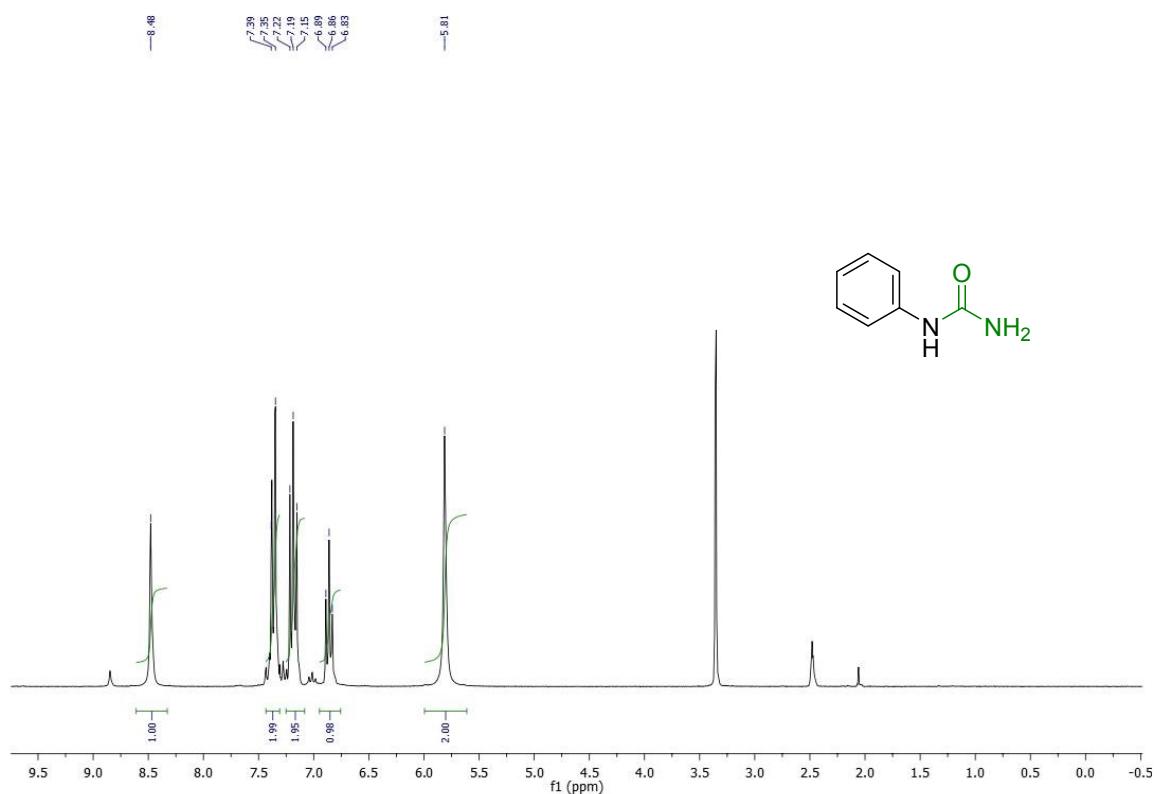
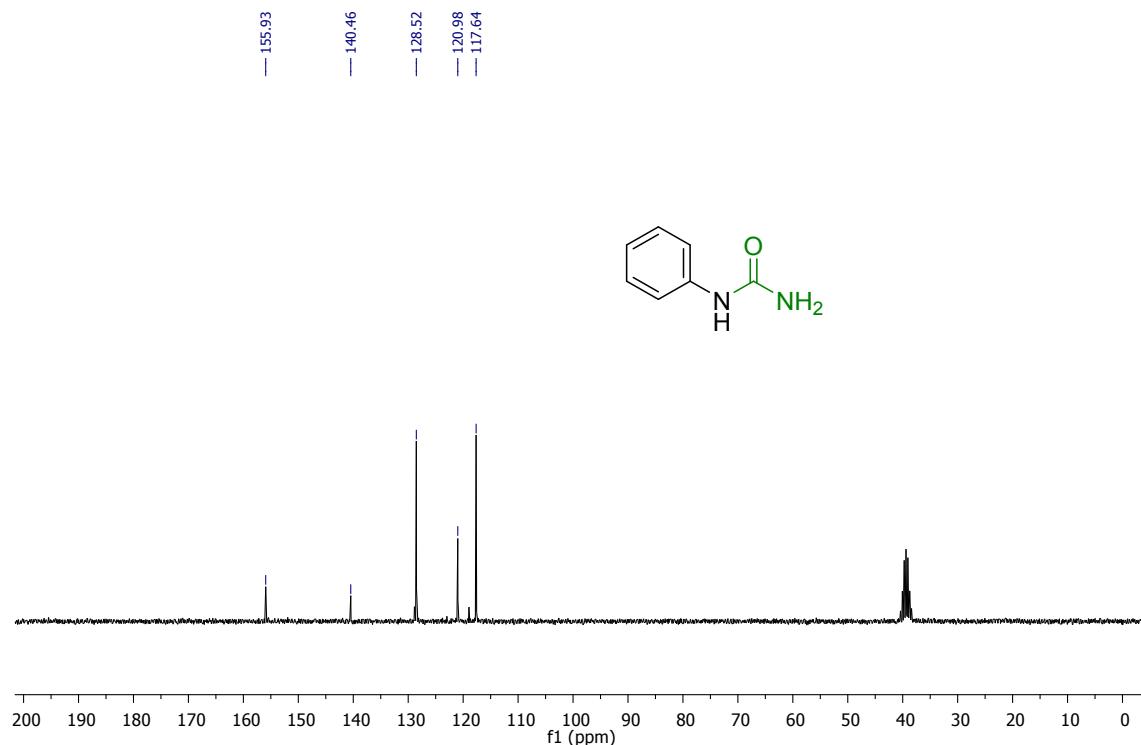
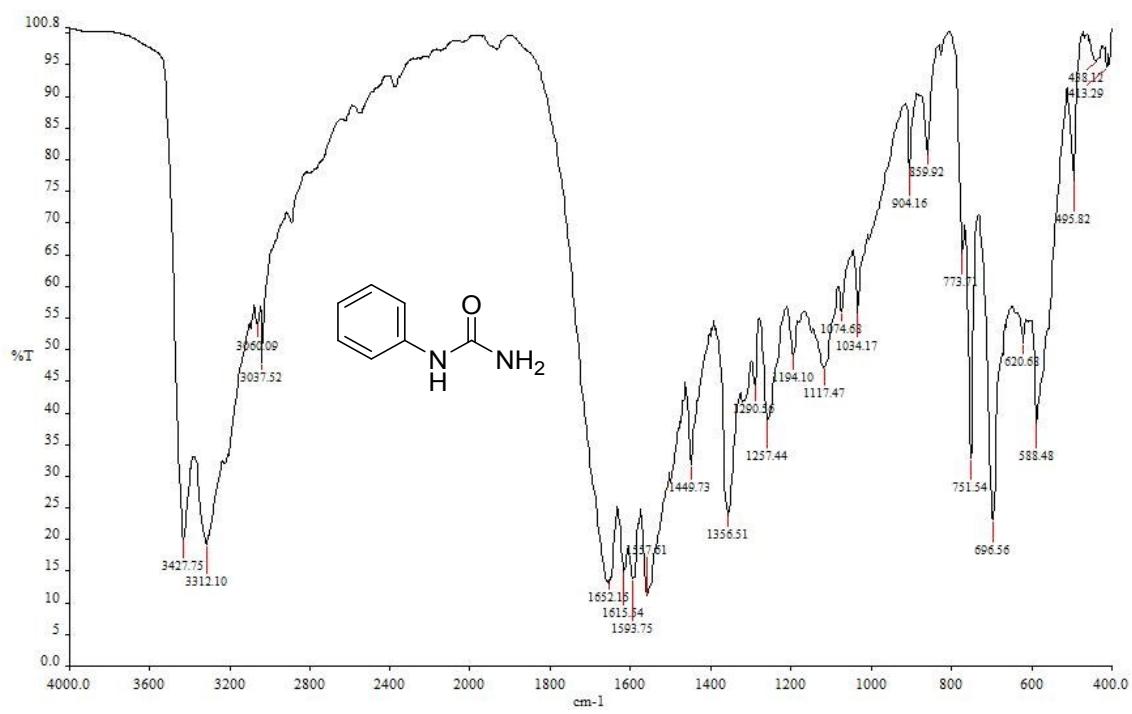


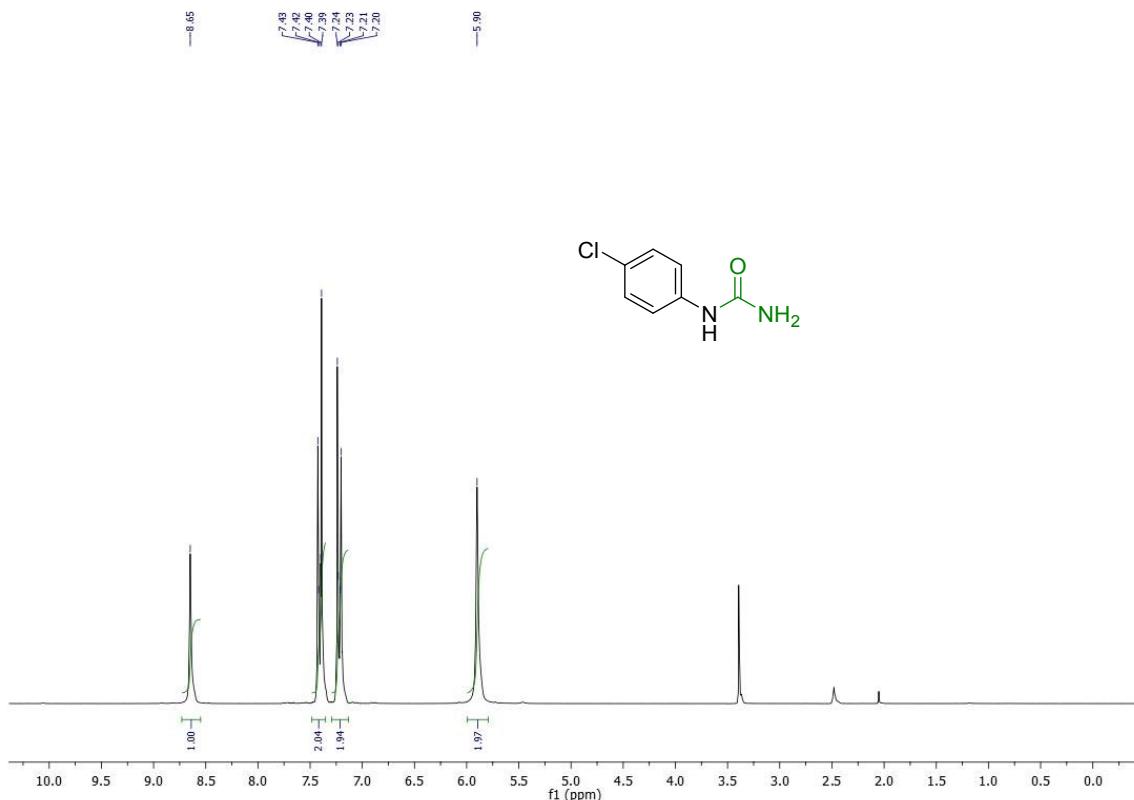
Figure S101.  $^1\text{H}$  NMR spectrum of 7a in  $\text{DMSO}-d_6$  (250MHz)



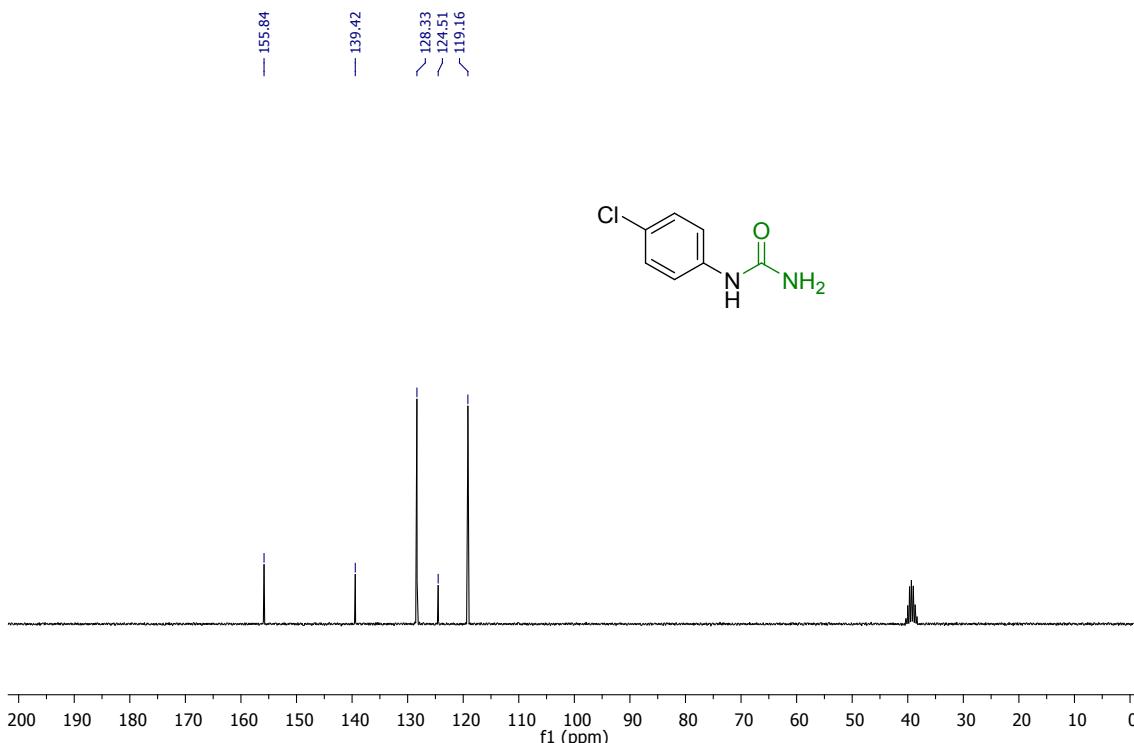
**Figure S102.** <sup>13</sup>C NMR spectrum of 7a in DMSO-d<sub>6</sub> (63MHz)



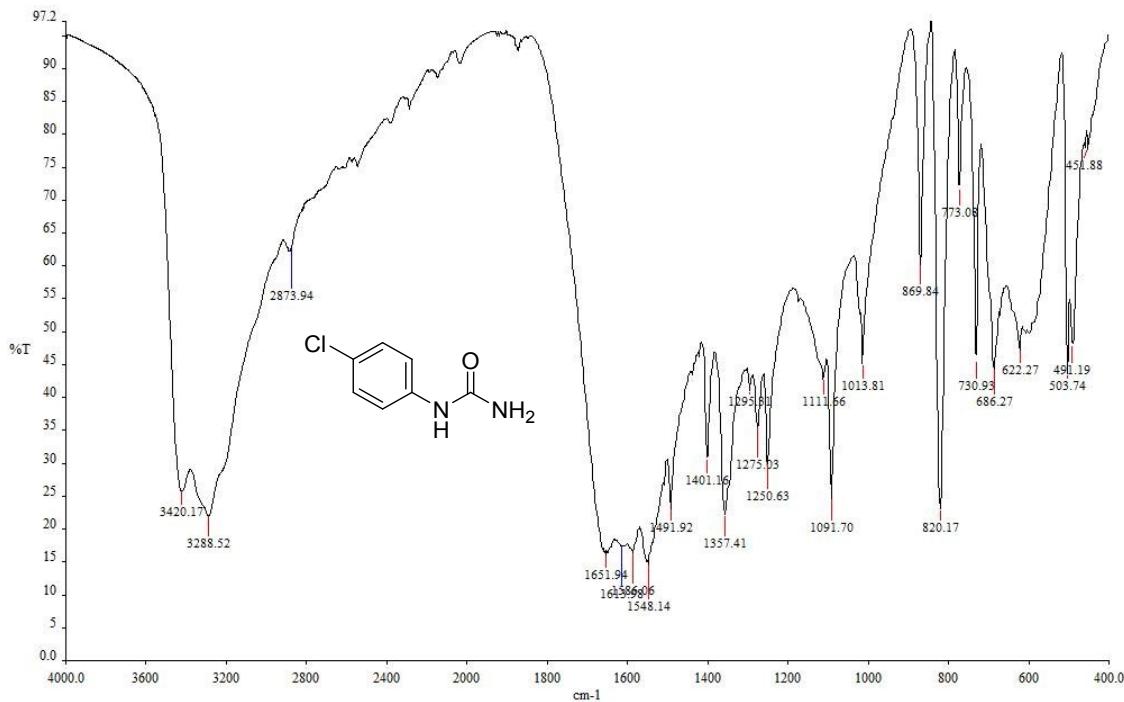
**Figure S103.** FT-IR spectrum of 7a in KBr



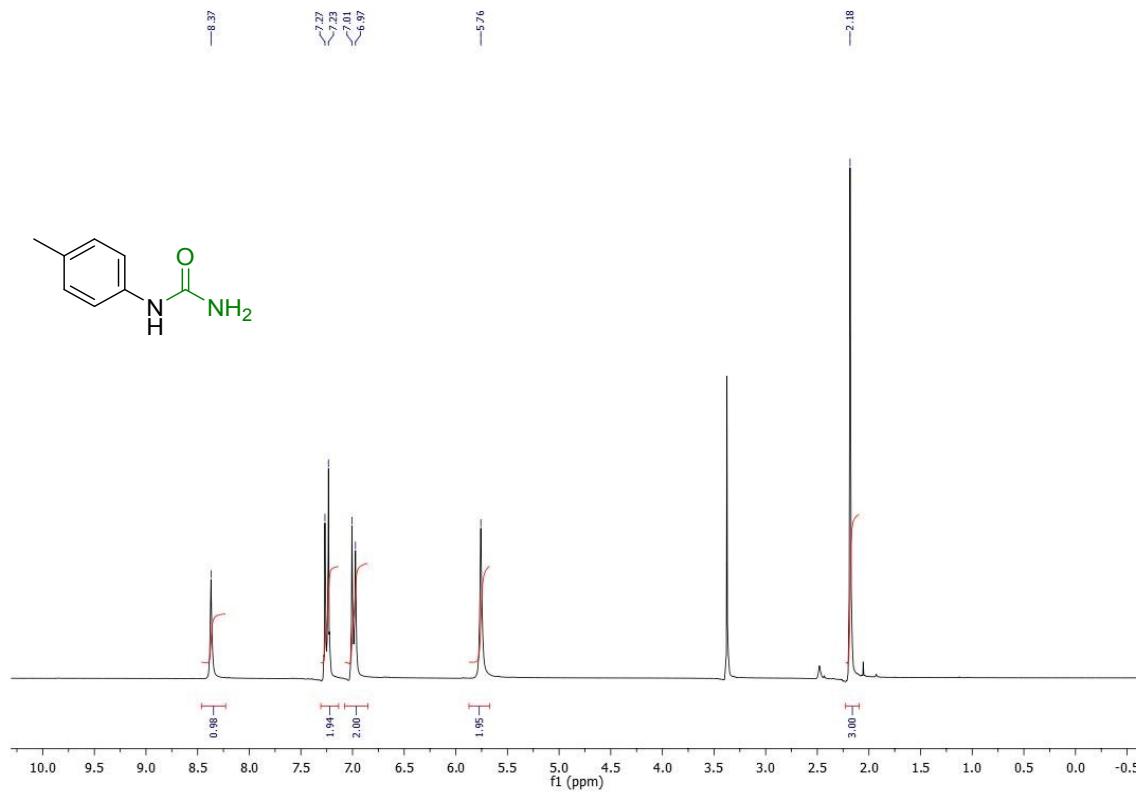
**Figure S104.**  $^1\text{H}$  NMR spectrum of **7b** in  $\text{DMSO}-d_6$  (250MHz)



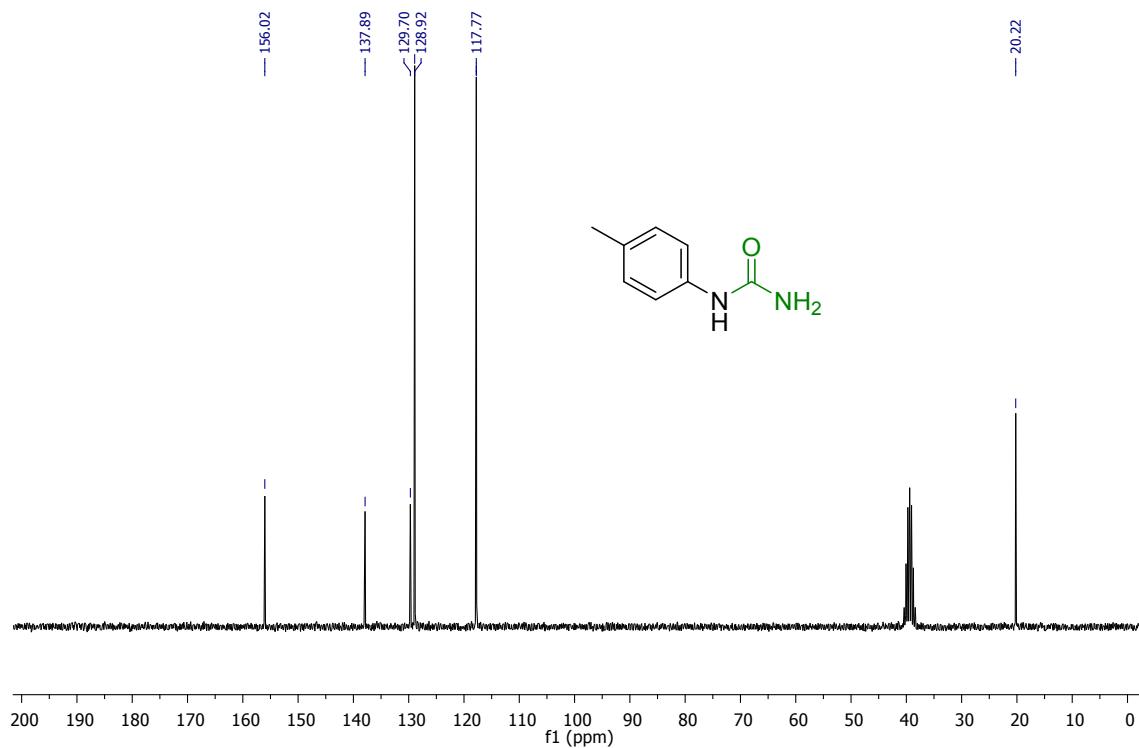
**Figure S105.**  $^{13}\text{C}$  NMR spectrum of **7b** in  $\text{DMSO}-d_6$  (63MHz)



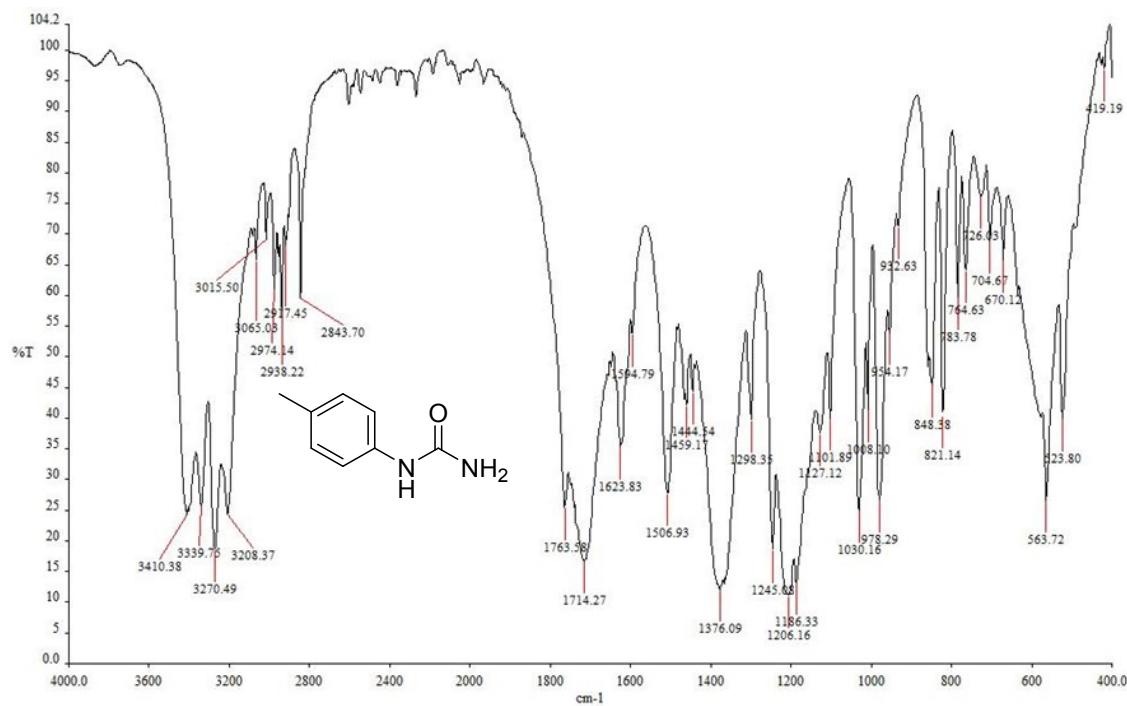
**Figure S106.** FT-IR spectrum of **7b** in KBr



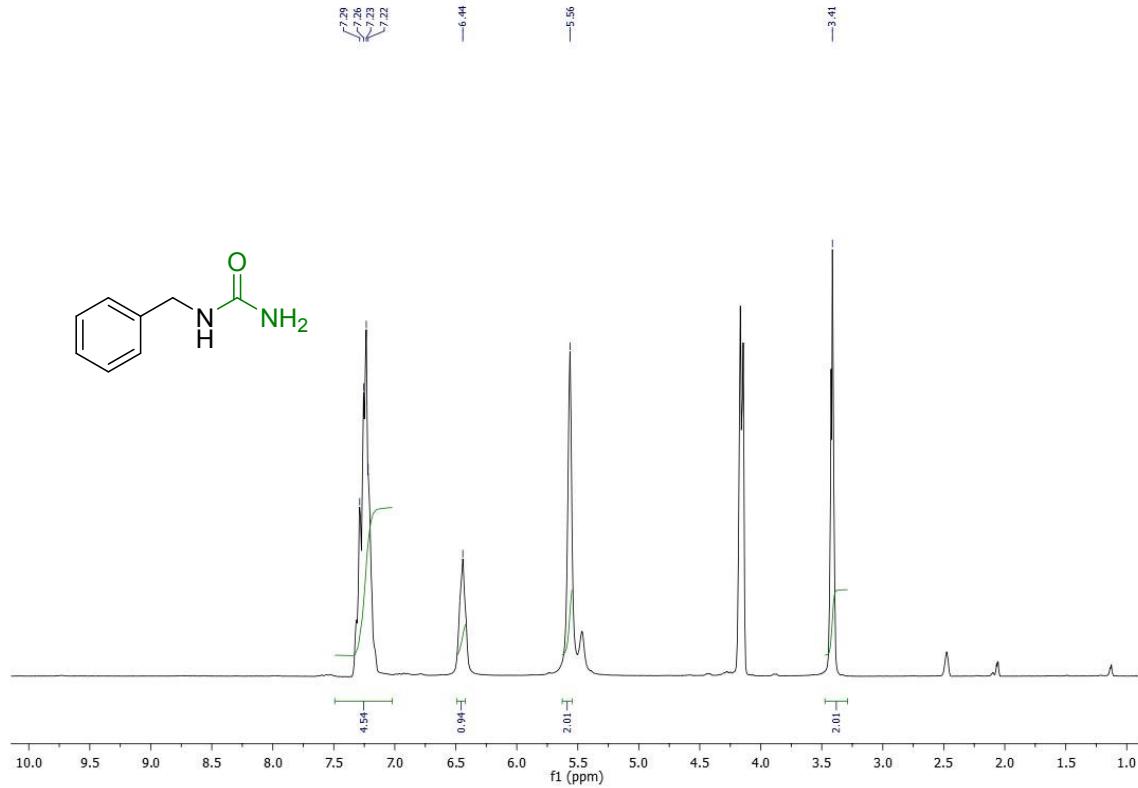
**Figure S107.**  $^1\text{H}$  NMR spectrum of **7c** in  $\text{DMSO}-d_6$  (250MHz)



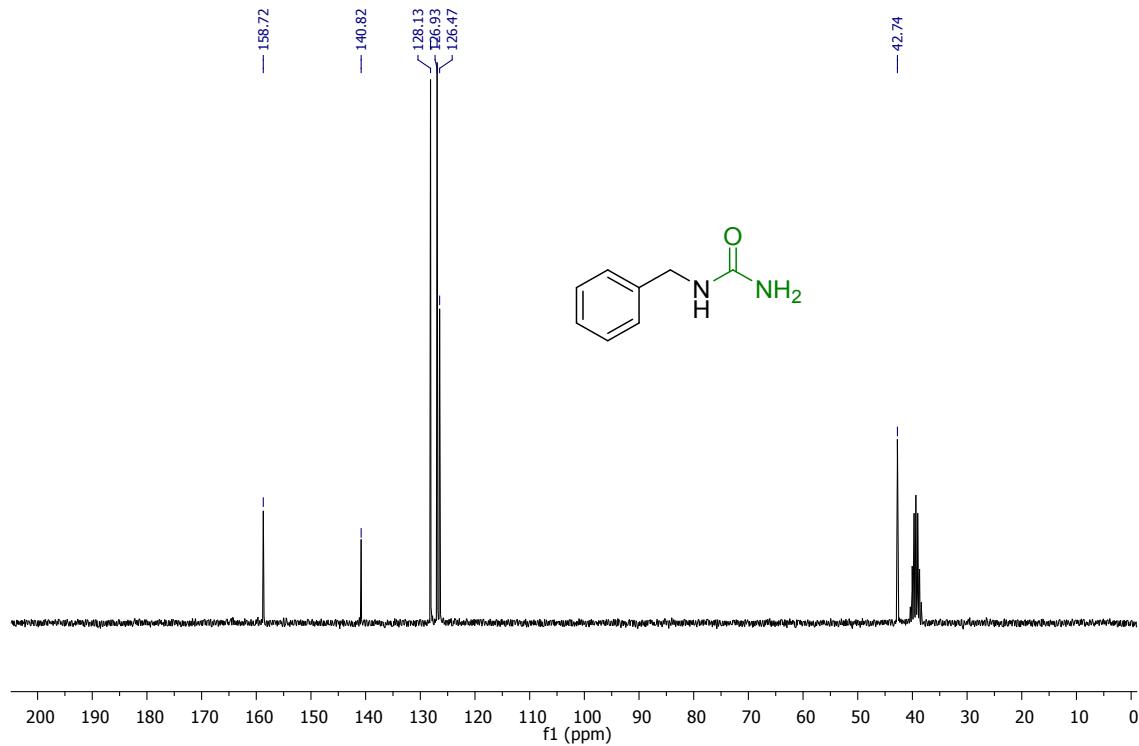
**Figure S108.**  $^{13}\text{C}$  NMR spectrum of **7c** in  $\text{DMSO}-d_6$  (63MHz)



**Figure S109.** FT-IR spectrum of **7c** in KBr



**Figure S110.**  $^1\text{H}$  NMR spectrum of **7d** in  $\text{DMSO}-d_6$  (250MHz)



**Figure S111.**  $^{13}\text{C}$  NMR spectrum of **7d** in  $\text{DMSO}-d_6$  (63MHz)

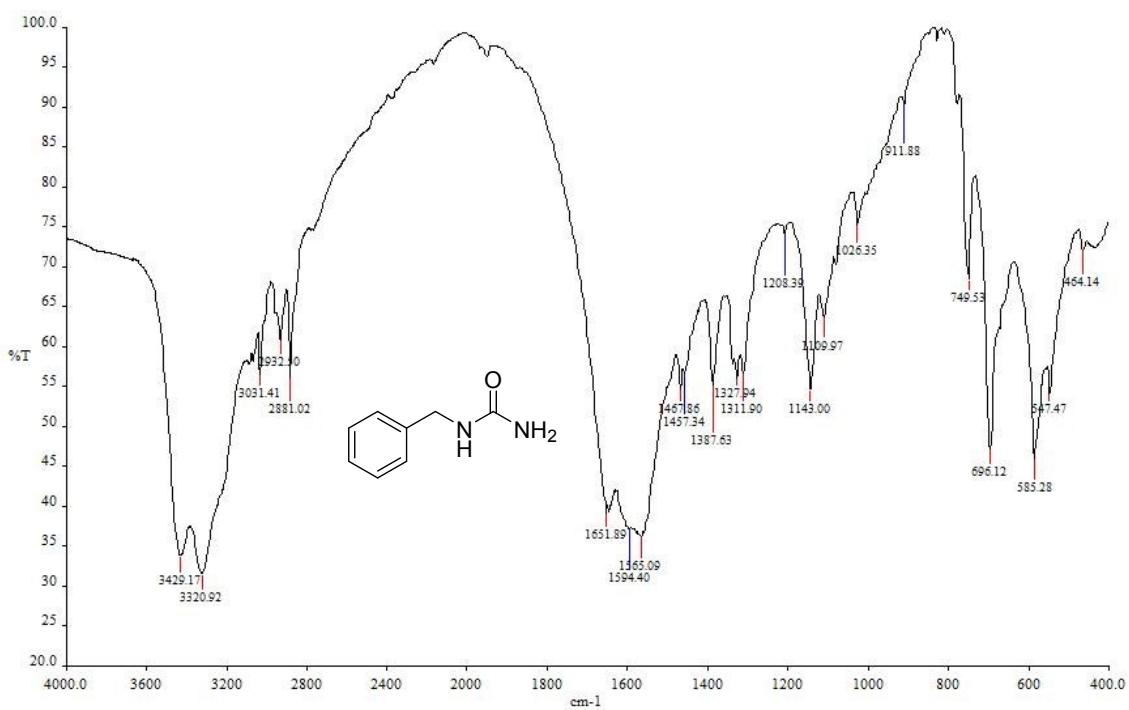


Figure S112. FT-IR spectrum of 7d in KBr

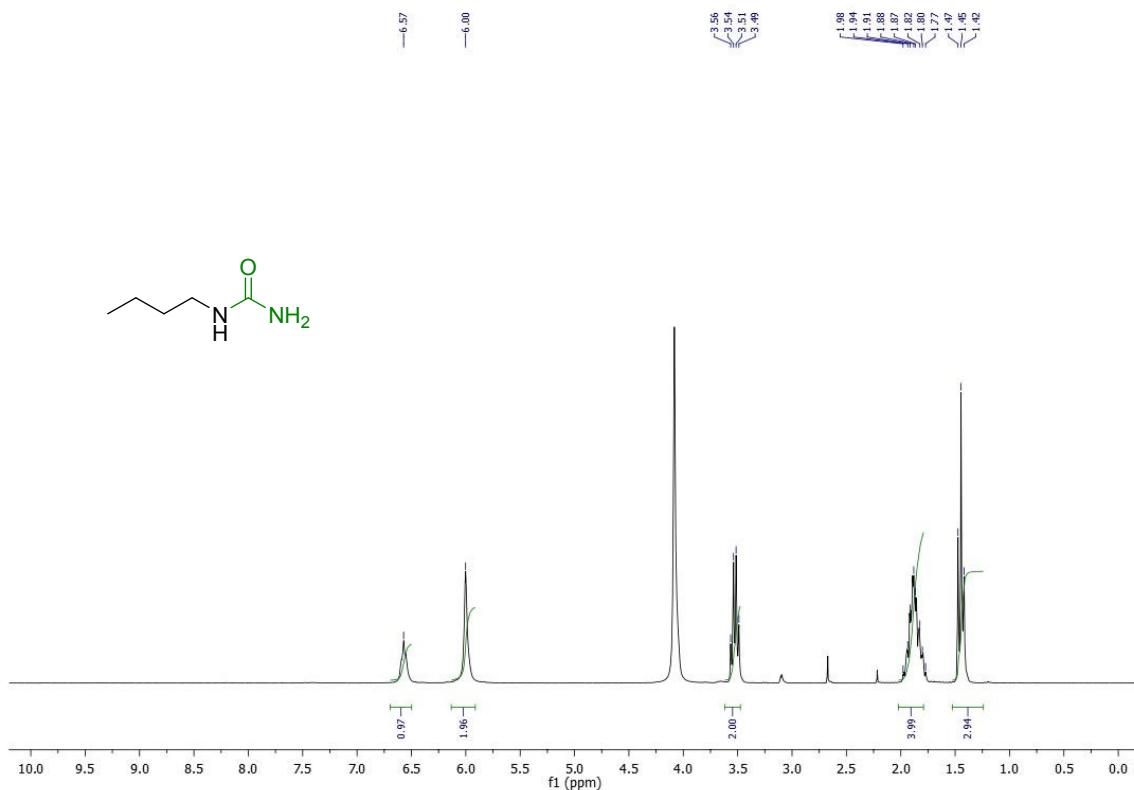


Figure S113. <sup>1</sup>H NMR spectrum of 7e in DMSO-d<sub>6</sub> (250MHz)

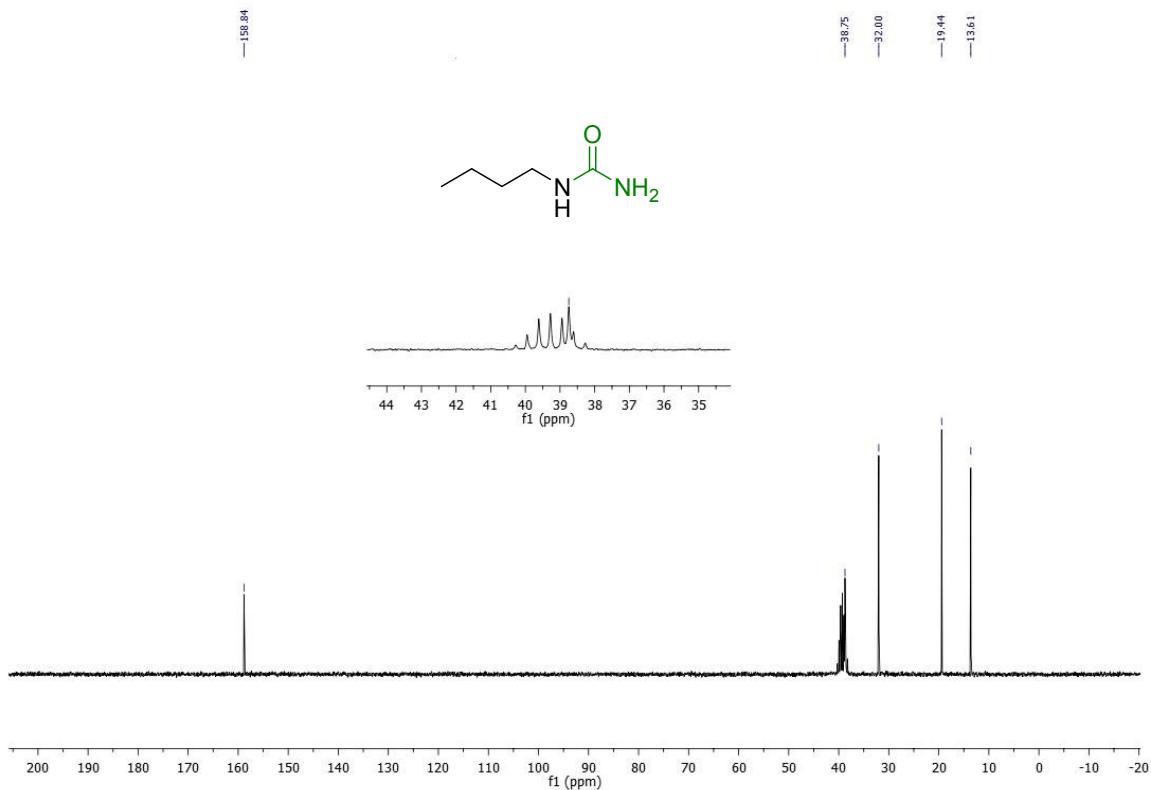
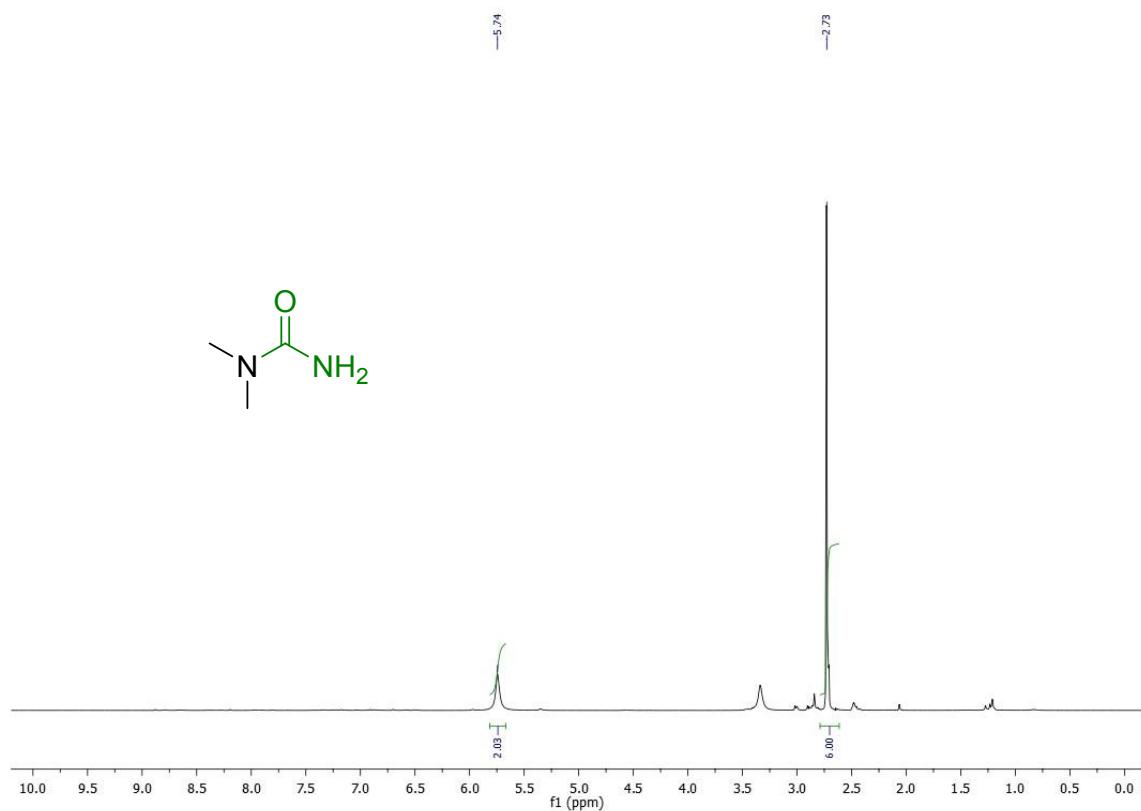
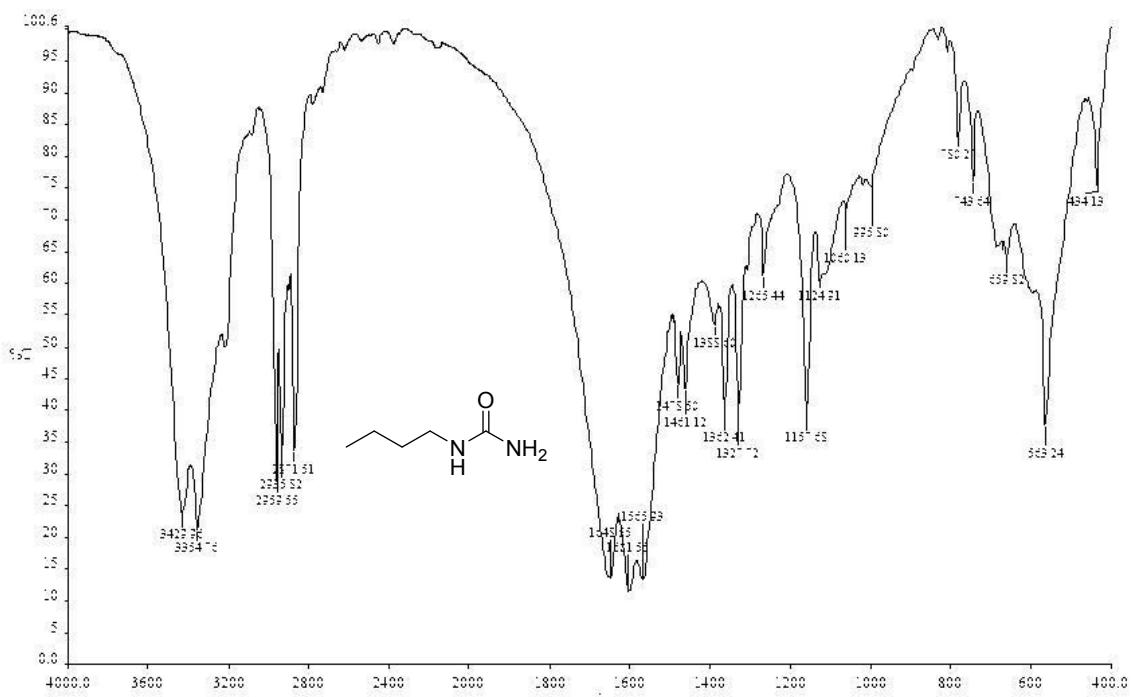
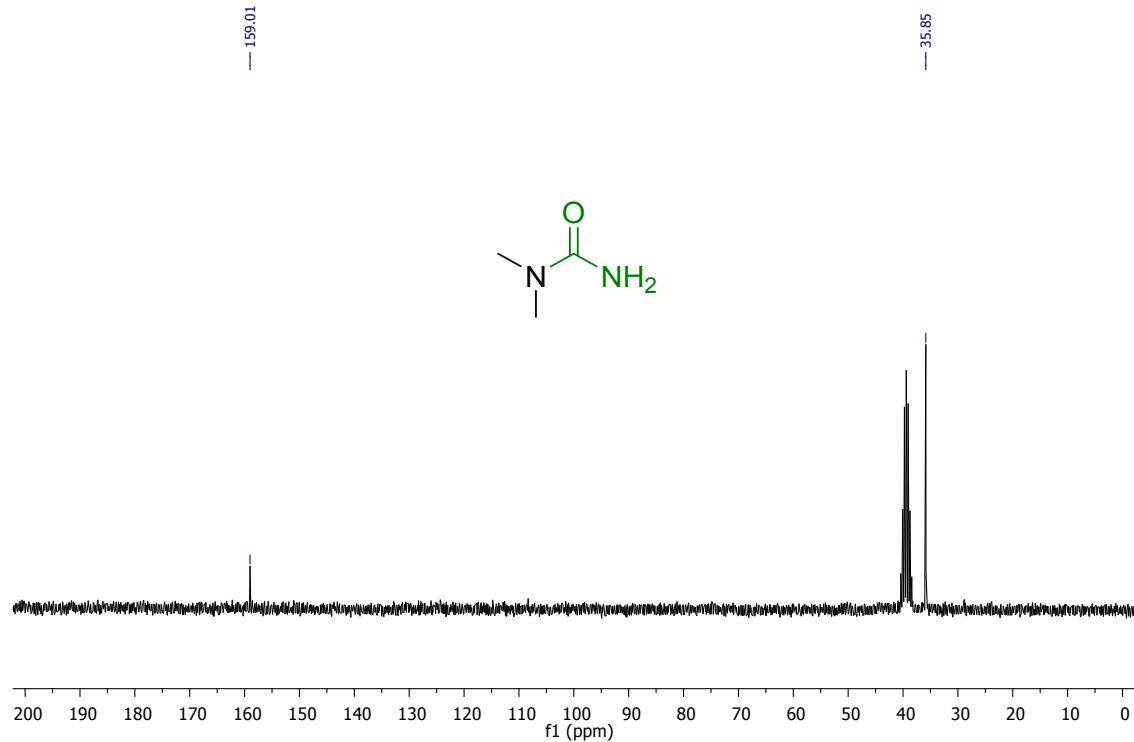
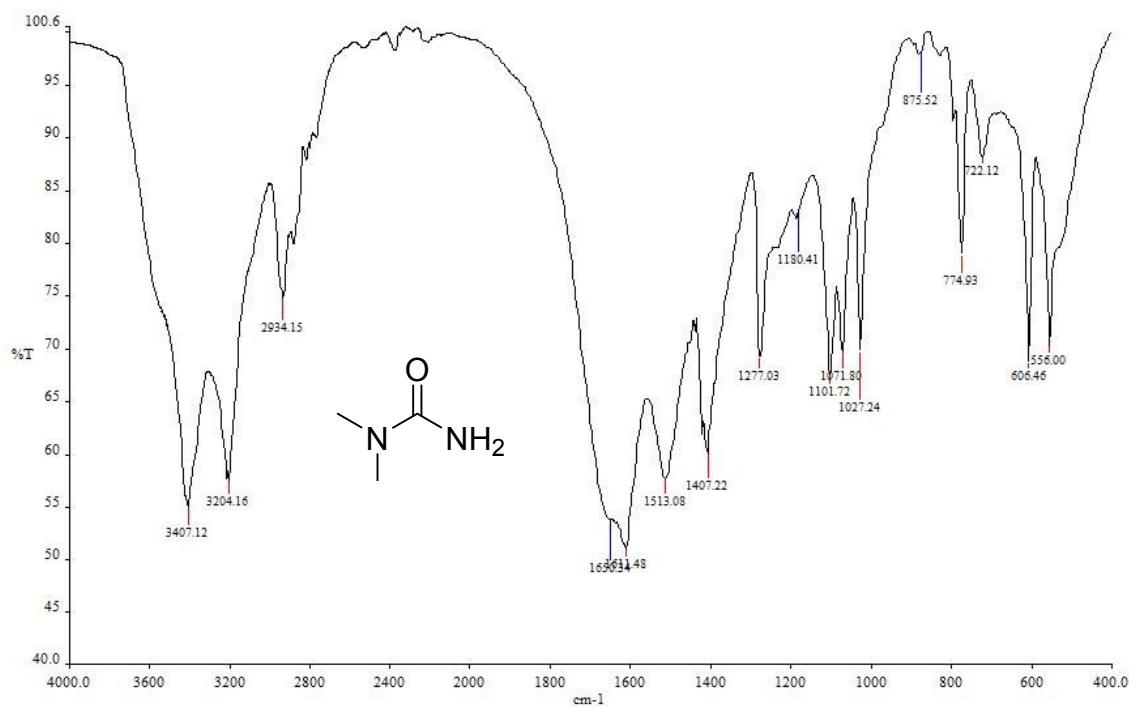


Figure S114. <sup>13</sup>C NMR spectrum of 7e in DMSO-d<sub>6</sub> (63MHz)

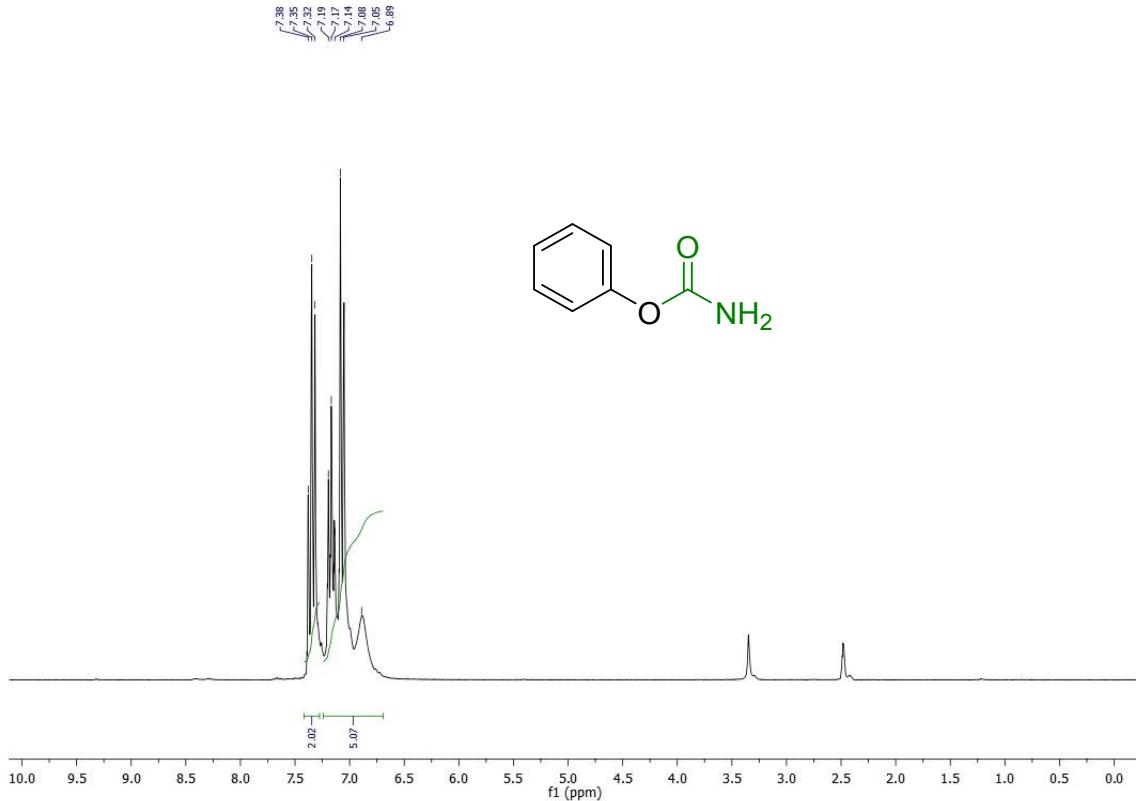




**Figure S117.**  $^{13}\text{C}$  NMR spectrum of 7f in  $\text{DMSO}-d_6$  (63MHz)

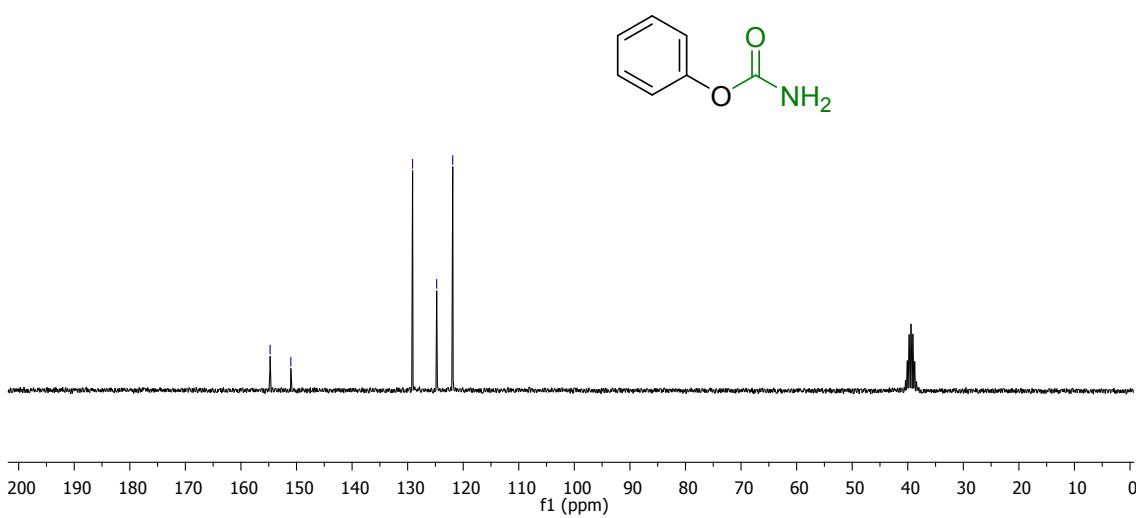


**Figure S118.** FT-IR spectrum of 7f in KBr

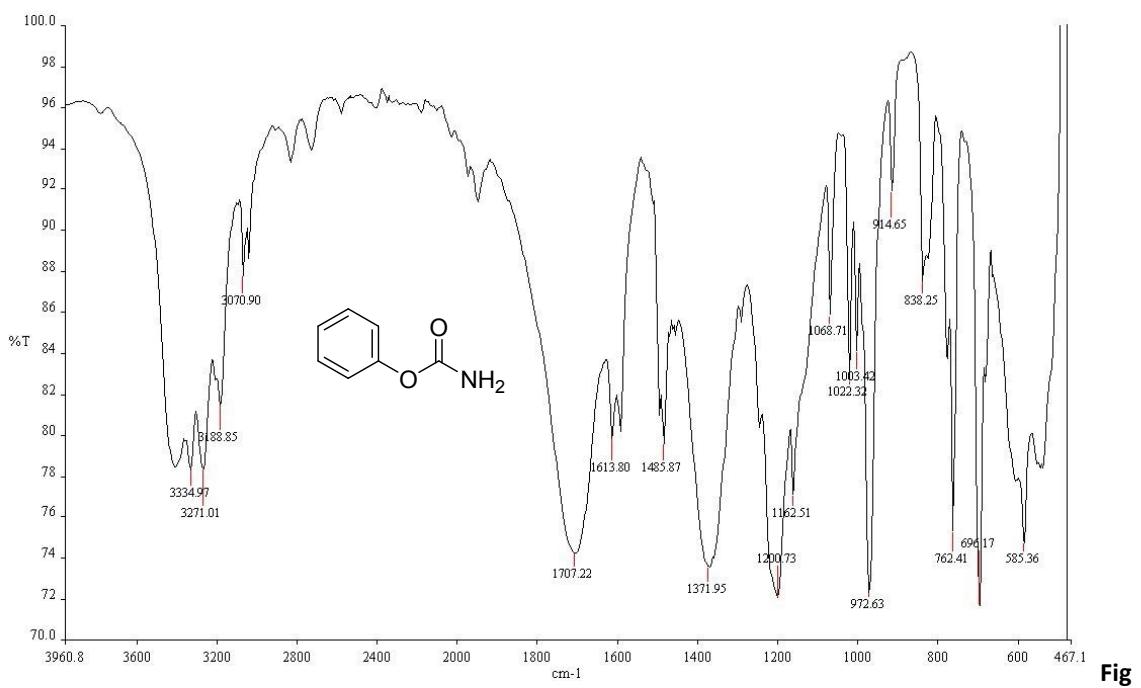


**Figure S119.**  $^1\text{H}$  NMR spectrum of **7g** in  $\text{DMSO}-d_6$  (250MHz)

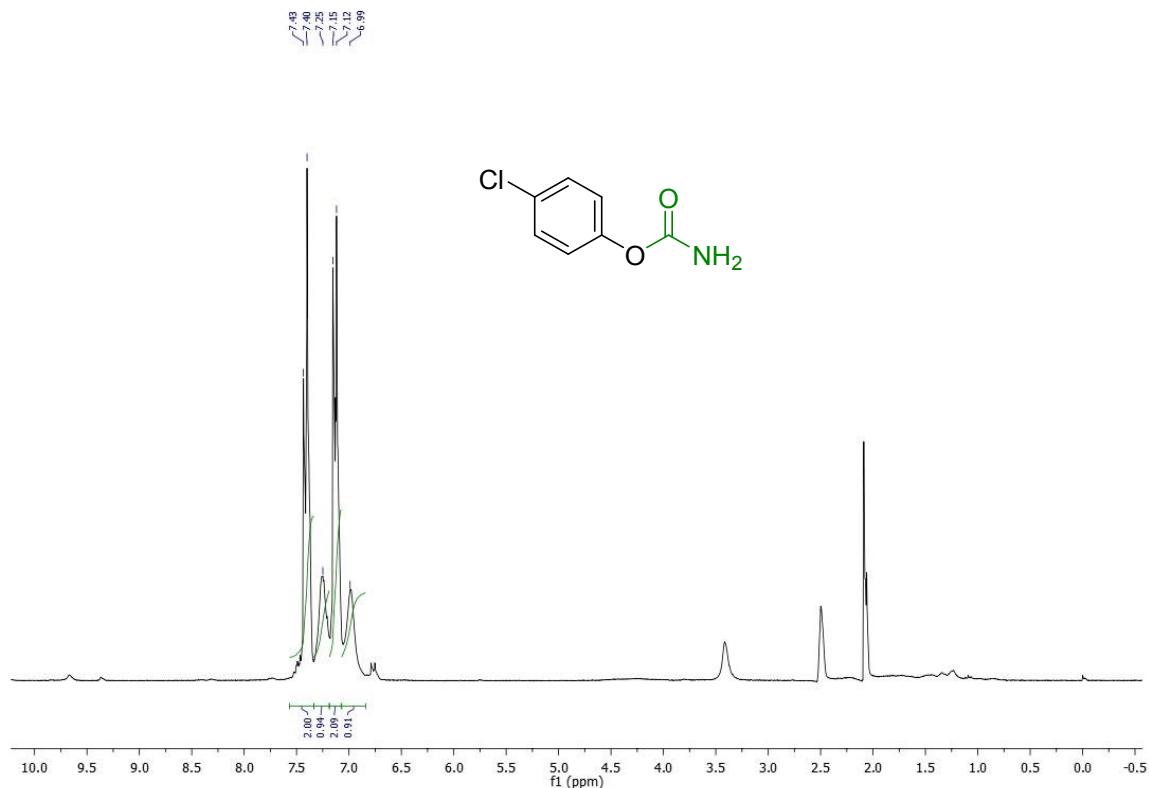
— 154.76  
— 151.02  
/ \ 129.11  
/ \ 124.77  
/ \ 121.88



**Figure S120.**  $^{13}\text{C}$  NMR spectrum of **7g** in  $\text{DMSO}-d_6$  (63MHz)



**ure S121. FT-IR spectrum of 7g in KBr**



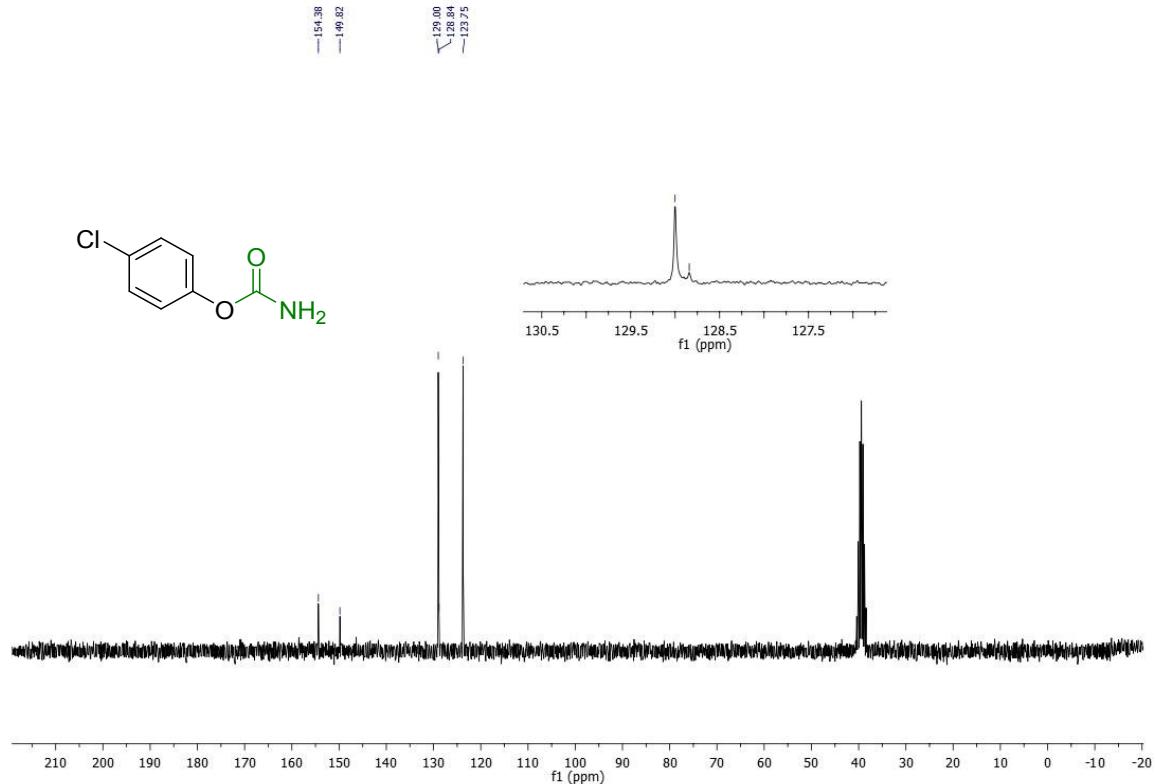
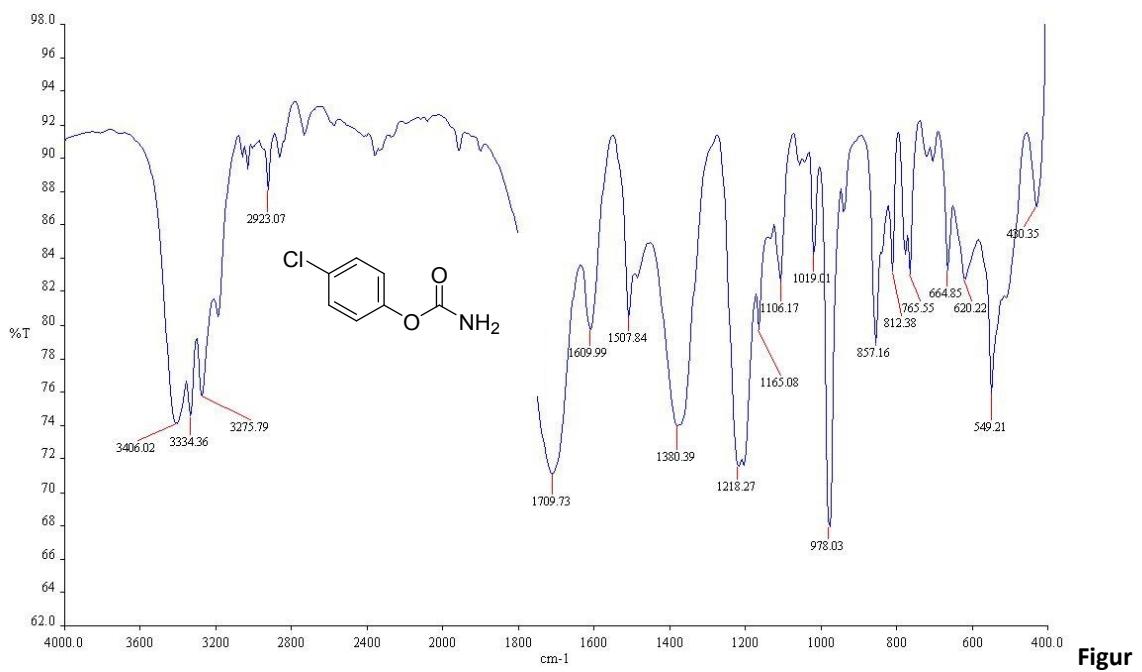
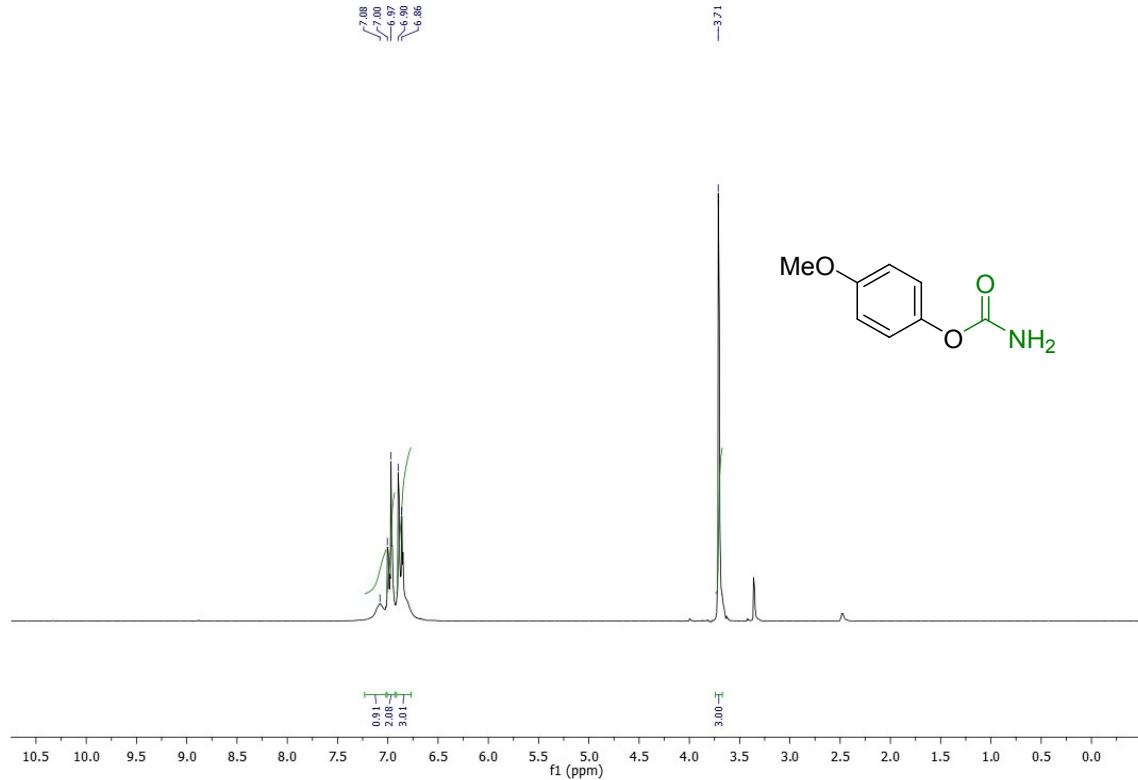


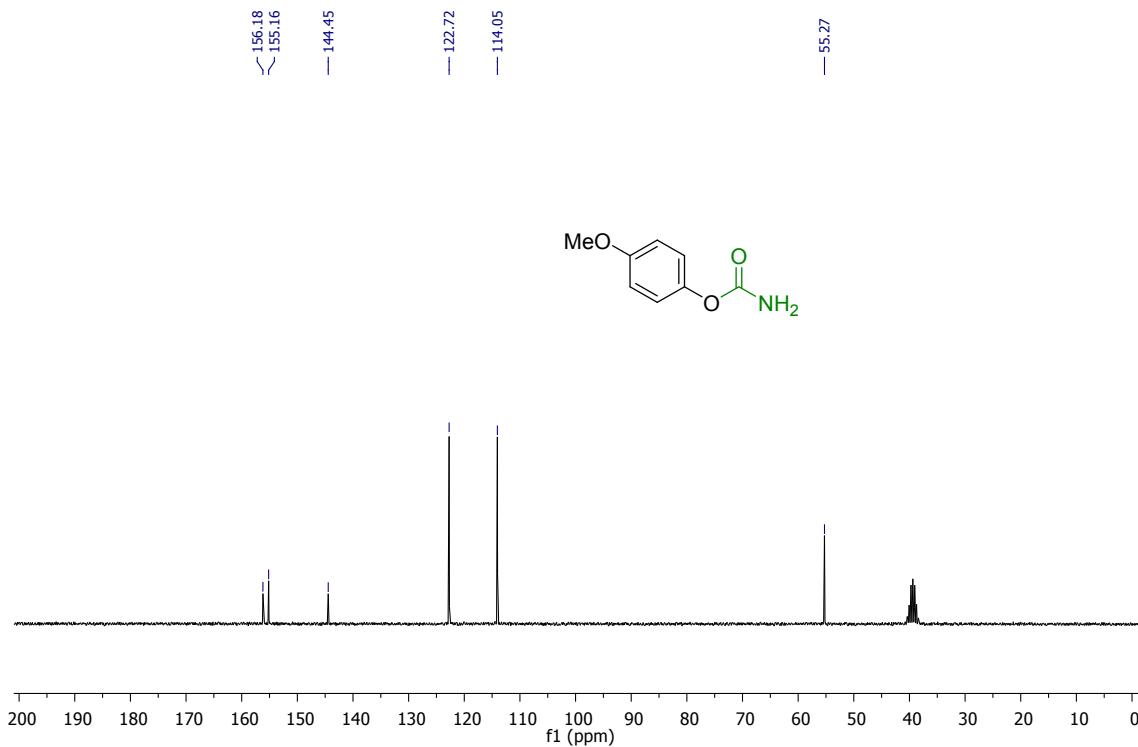
Figure S123. <sup>13</sup>C NMR spectrum of 7h in DMSO-*d*<sub>6</sub> (63MHz)



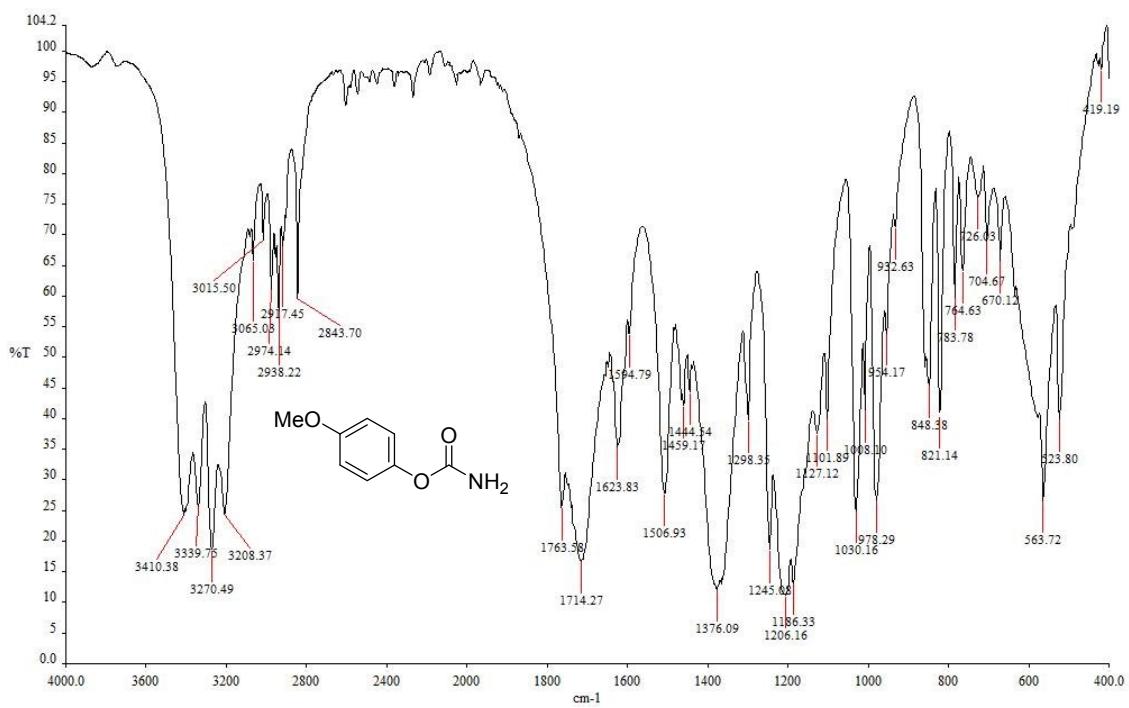
e S124. FT-IR spectrum of 7h in KBr



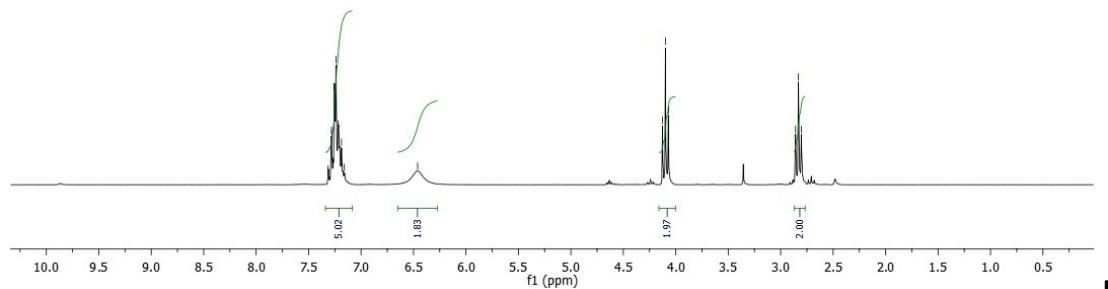
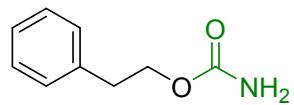
**Figure S125.**  $^1\text{H}$  NMR spectrum of **7i** in  $\text{DMSO}-d_6$  (250MHz)



**Figure S126.**  $^{13}\text{C}$  NMR spectrum of **7i** in  $\text{DMSO}-d_6$  (63MHz)

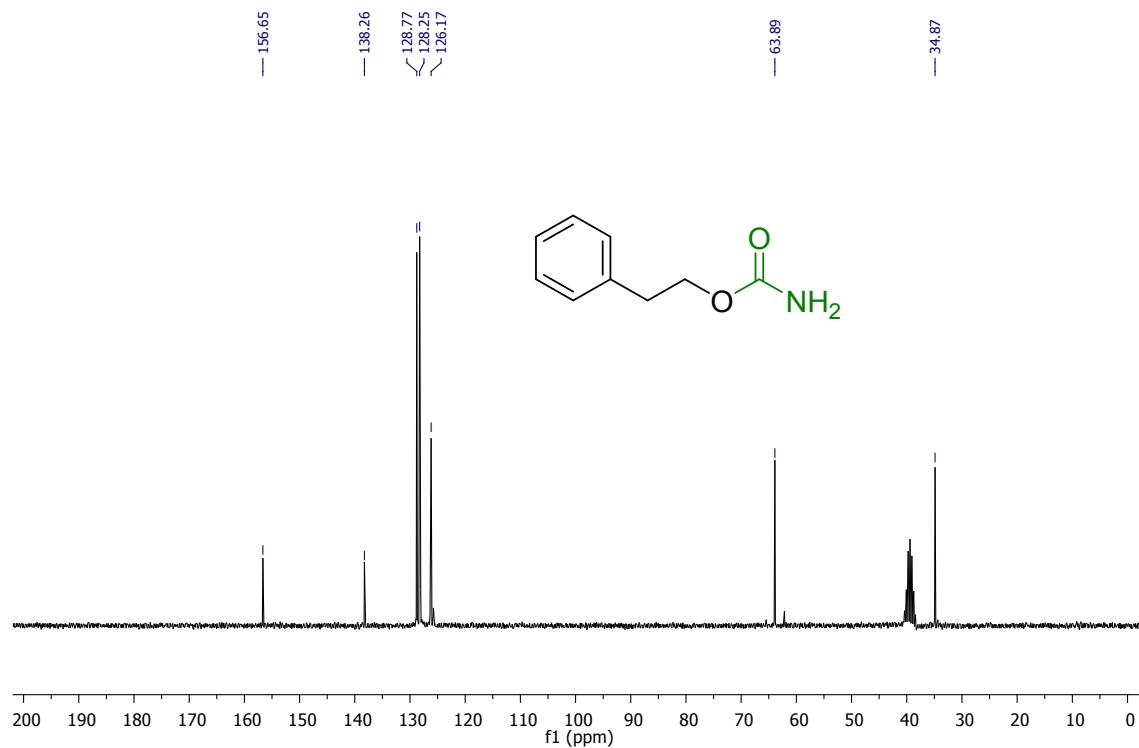


**Figure S127. FT-IR spectrum of 7i in KBr**

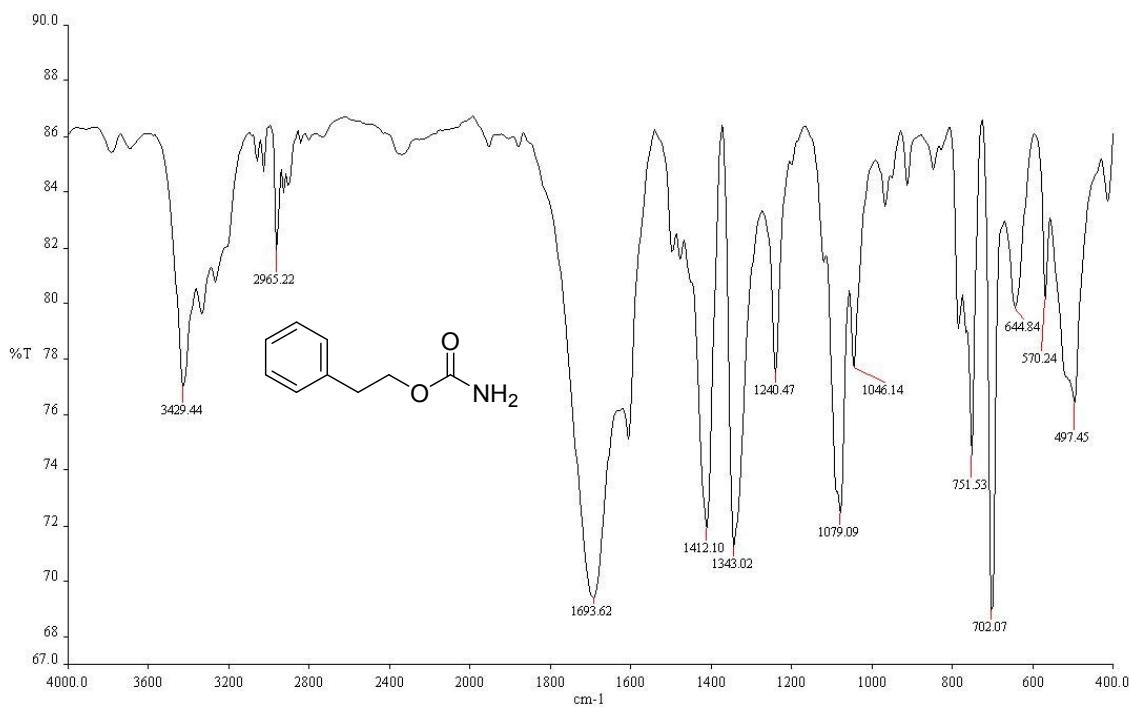


**Figure S128.  $^1\text{H}$  NMR spectrum of 7j in  $\text{DMSO}-d_6$  (250MHz)**

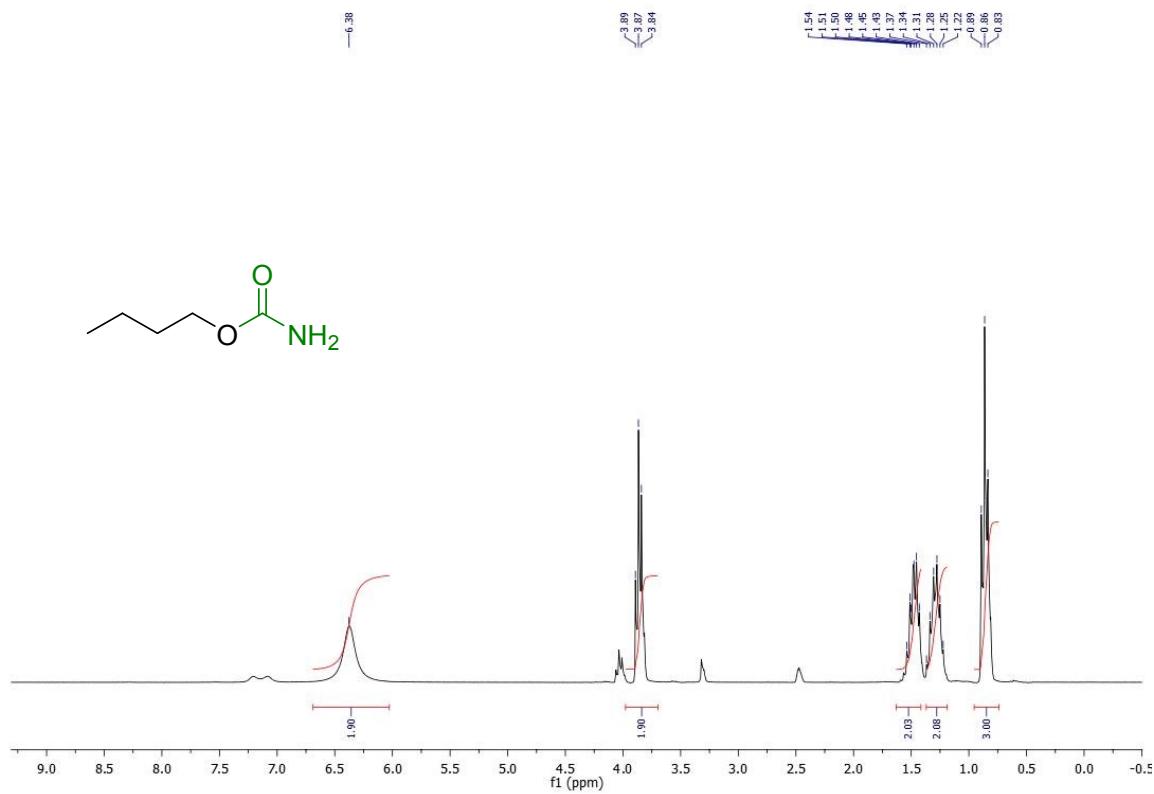
**Fig**



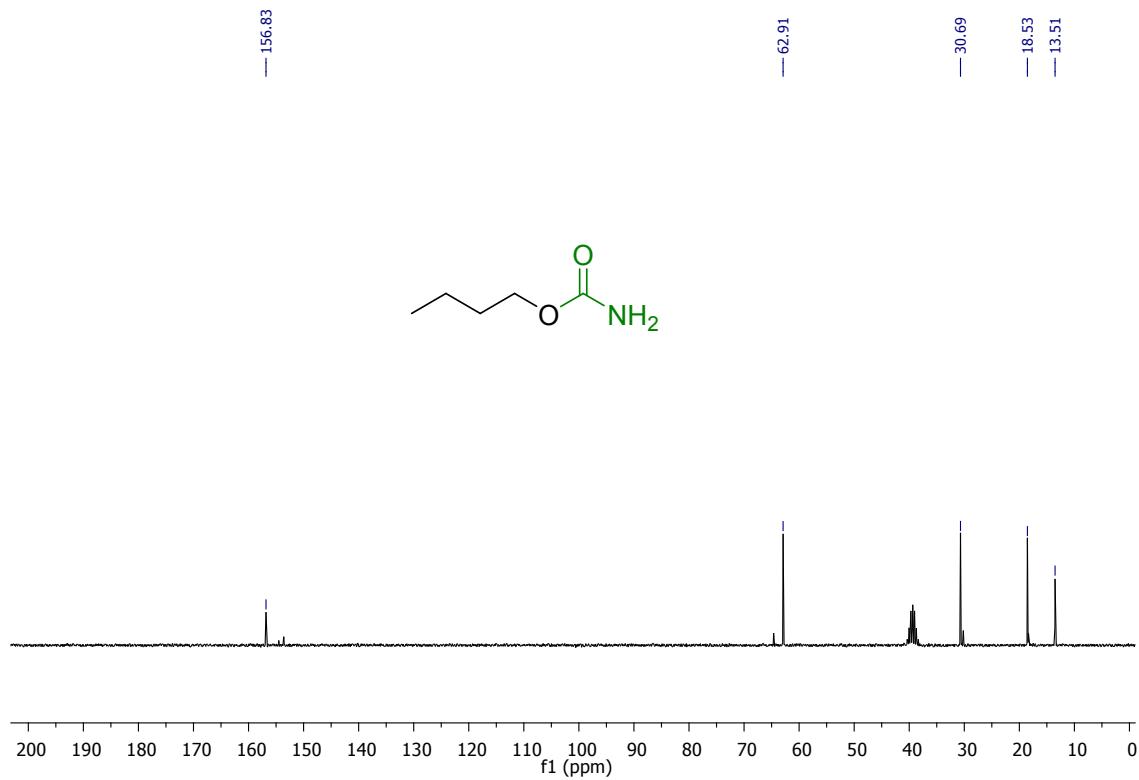
**Figure S129.**  $^{13}\text{C}$  NMR spectrum of **7j** in  $\text{DMSO}-d_6$  (63MHz)



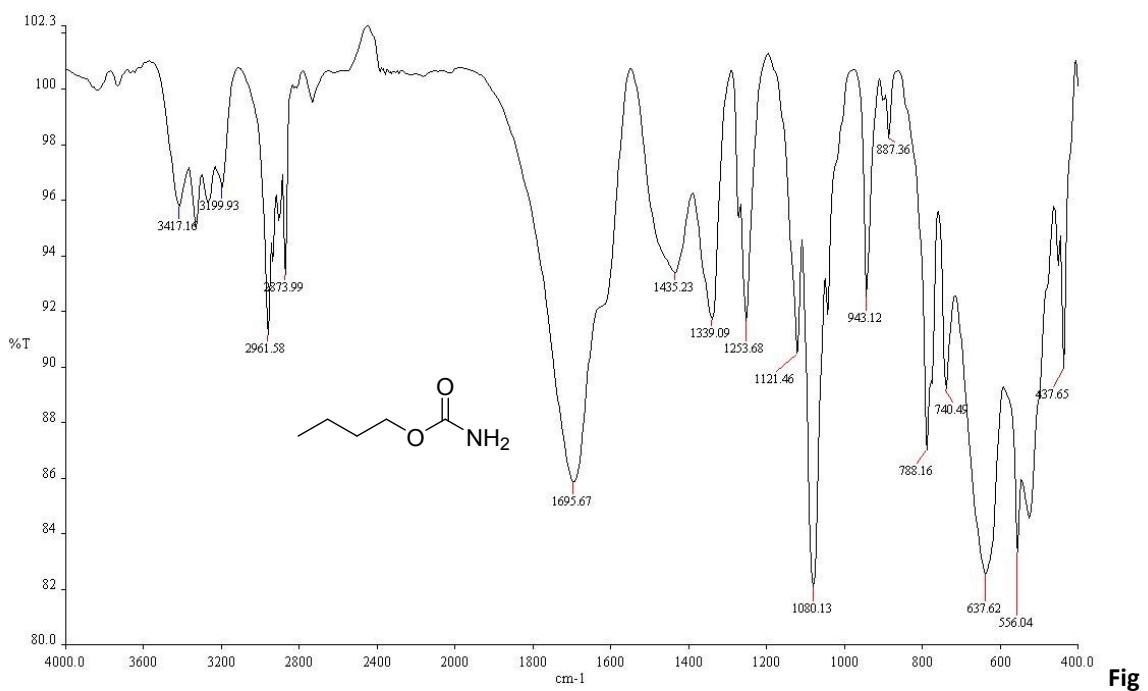
**Figure S130.** FT.IR spectrum of **7j** in KBr



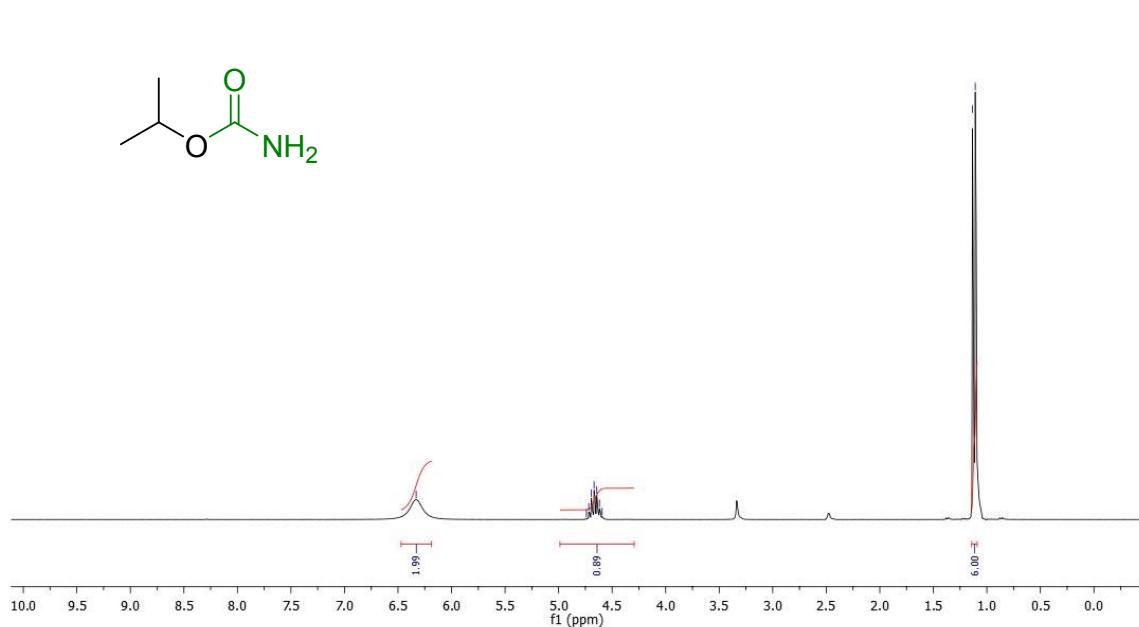
**Figure S131.** <sup>1</sup>H NMR spectrum of 7k in DMSO-d<sub>6</sub> (250MHz)

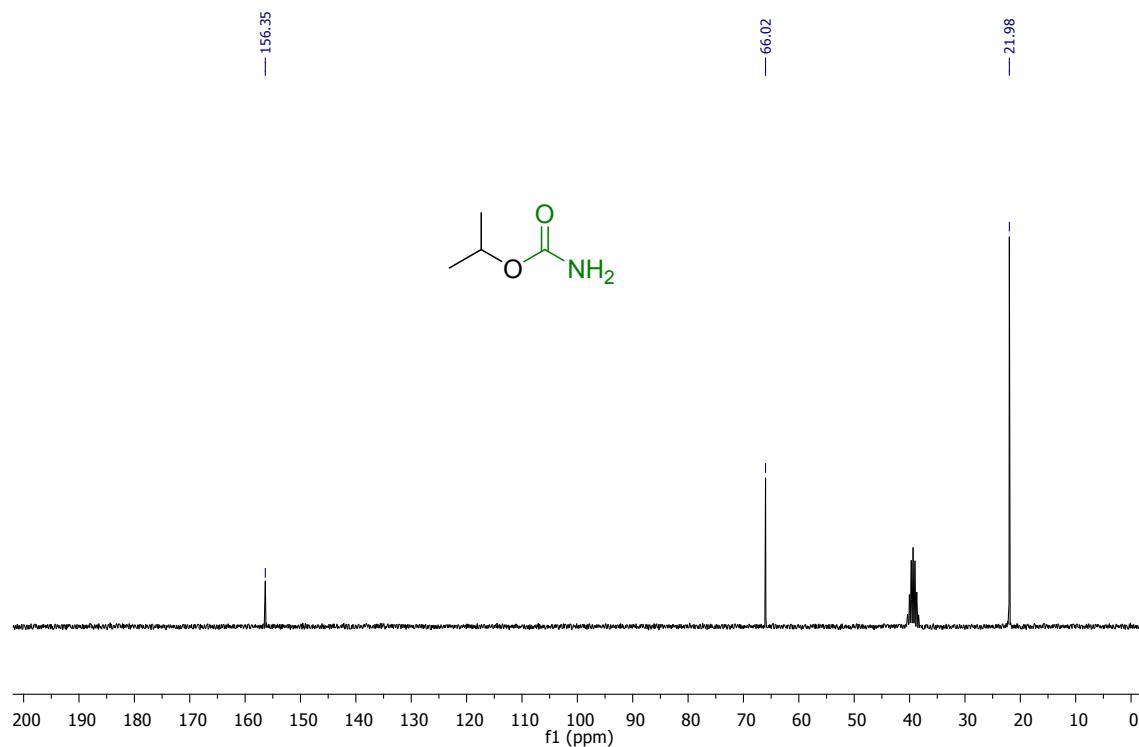


**Figure S132.** <sup>13</sup>C NMR spectrum of 7k in DMSO-d<sub>6</sub> (63MHz)

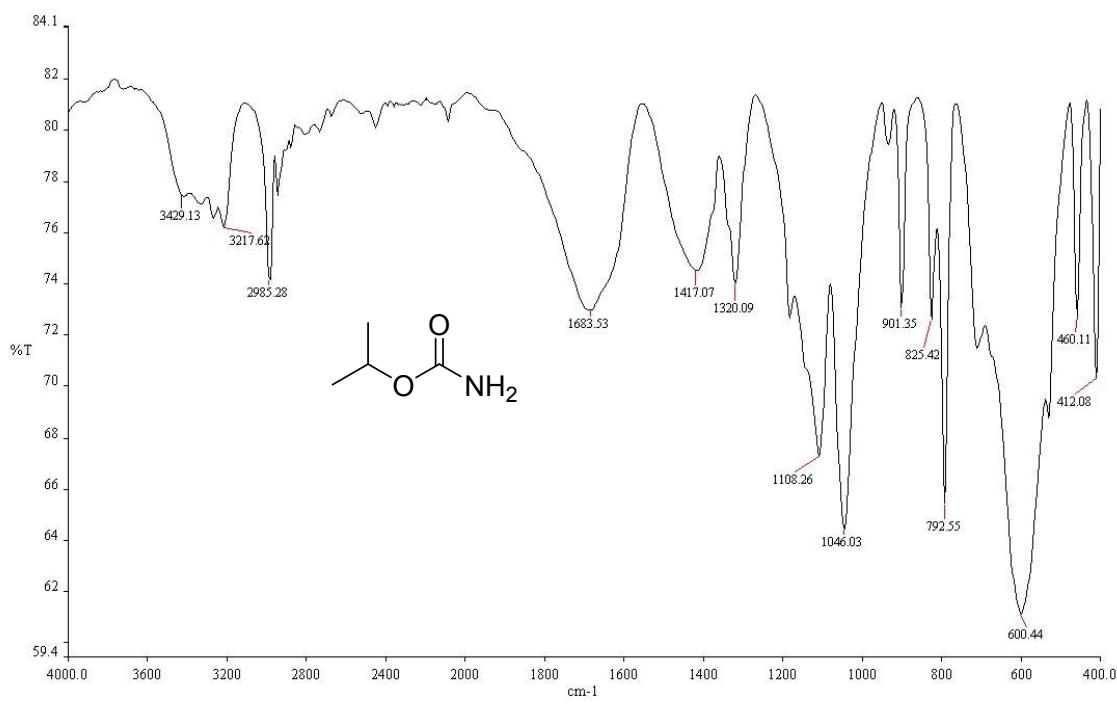


**Fig**





**Figure S135.**  $^{13}\text{C}$  NMR spectrum of 7I in  $\text{DMSO}-d_6$  (63MHz)



**Figure S136.** FT-IR spectrum of 7I in KBr

