

**An oxidative cleavage of *anti*-Hugerschoff product: A mild  
environmentally benign and one pot synthesis of ureas from  
isothiocyanates**

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## Experimental:

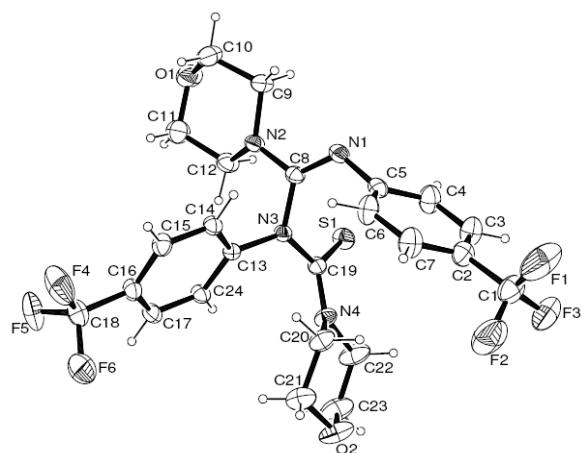
### General remarks

Unless otherwise stated, all reagents were purchased from commercial sources and used without further purification. Reaction progress was monitored by TLC using Merck silica gel 60 F<sub>254</sub> (0.25mm) with detection by UV or iodine. Chromatography was performed using Merck silica gel (60-120) mesh size with freshly distilled solvents. Columns were typically packed as slurry and equilibrated with the appropriate solvent system prior to use.<sup>1</sup>H NMR (400 MHz) and <sup>13</sup>C NMR (100 MHz) spectra were recorded on a Varian FT-400 MHz instrument using TMS as an internal standard. Data are presented as follows: chemical shift (ppm), multiplicity (s = singlet, d = doublet, t = triplet, m = multiplet, b = broad, brs = broad singlet, brm= broad multiplet, coupling constant *J* (Hz). Elemental analyses were carried out on a Perkin–Elmer 2400 automatic carbon, hydrogen, nitrogen and sulfur analyser. Melting points were recorded on Buchi B-545 melting point apparatus and are uncorrected. IR spectra were recorded in KBr or neat on a Nicolet Impact 410 spectrophotometer. Mass data were obtained with a WATERS MS system, Q-tof premier and data analyzed using Mass Lynx4.1.

**Crystallographic Analysis:** Crystal data were collected with Bruker Smart Apex-II CCD diffractometer using graphite by using graphite-monochromated Mo-*K*<sub>α</sub> radiation ( $\lambda = 0.71073 \text{ \AA}$ ) at 298 K. Cell parameters were retrieved using SMART <sup>1</sup>USA, 1995 software and refined with SAINT<sup>1</sup> for all observed reflections. Data reduction was performed with the SAINT software and corrected for Lorentzian and polarization effects. Absorption corrections were applied with the SADABS program.<sup>2</sup> The structures were solved by direct methods implemented in the SHELX-97<sup>3</sup> program and refined by full-matrix least-squares methods on  $F^2$ . All non-hydrogen atom positions were located in difference Fourier maps and refined anisotropically. The hydrogen atoms were placed in their geometrically generated positions. The crystals were isolated in rectangular shape from ethyl acetate and hexane mixture at room temperature.

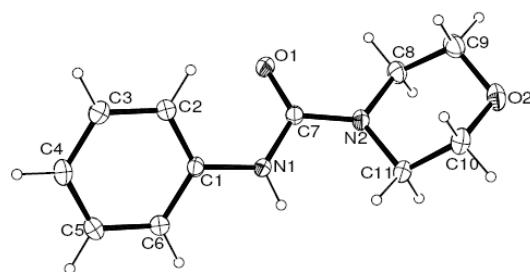
## References

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- 2 G. M. Sheldrick, *SADABS: Empirical Absorption and Correction Software*, University of Gottingen, Institut fur Anorganische Chemie der Universitat, Tammanstrasse 4, D-3400 Gottingen, Germany, 1999–2003.
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**Fig. 2** ORTEP views of *N*-(*E*)-(4-(Trifluoromethyl)phenylimino)(morpholino)methyl)-*N*-(4-(trifluoromethyl)phenyl) morpholine-4-carbothioamide (**11**)

**Crystallographic description of *N*-(*E*)-(4-(Trifluoromethyl)phenylimino)(morpholino)methyl)-*N*-(4-(trifluoromethyl)phenyl)morpholine-4-carbothioamide (**11**):**  $C_{24}H_{24}F_6N_4O_2S$ , crystal dimensions  $0.42 \times 0.35 \times 0.26$  mm,  $M_r = 546.54$ , monoclinic, space group  $P21/c$ ,  $a = 14.9136(7)$ ,  $b = 9.0945(5)$ ,  $c = 18.6160(9)$  Å,  $\alpha = 90.00^\circ$ ,  $\beta = 98.612(2)^\circ$ ,  $\gamma = 90.00^\circ$ ,  $V = 2496.5(2)$  Å<sup>3</sup>,  $Z = 4$ ,  $\rho_{\text{calcd}} = 1.454$  mg/m<sup>3</sup>,  $\mu = 0.204$  mm<sup>-1</sup>,  $F(000) = 1128$ , reflection collected / unique = 4516 / 3803, refinement method = full-matrix least-squares on  $F^2$ , final  $R$  indices [ $I > 2\sigma(I)$ ]:  $R_1 = 0.0709$ ,  $wR_2 = 0.2116$ ,  $R$  indices (all data):  $R_1 = 0.0916$ ,  $wR_2 = 0.2336$ , goodness of fit = 1.056. CCDC-815734 (for *N*-(4-Morpholinylthioxomethyl)-*N,N'*-bis[(4-trifluoromethyl)phenyl]) contains the supplementary crystallographic data for this paper. These data can be obtained free of charge from The Cambridge Crystallographic Data Centre via [www.ccdc.cam.ac.uk/data\\_request/cif](http://www.ccdc.cam.ac.uk/data_request/cif).



**Fig. 3** ORTEP views of Morpholine-4-carboxylic acid phenylamide **14a**.

**Crystallographic description of Morpholine-4-carboxylic acid phenylamide (**14a**):**  $C_{11}H_{14}N_2O_2$ , crystal dimensions  $0.40 \times 0.32 \times 0.24$  mm,  $M_r = 206.24$ , monoclinic, space group  $P\bar{1}1/c$ ,  $a = 8.0909(3)$ ,  $b = 15.7683(6)$ ,  $c = 8.4586(3)$  Å,  $\alpha = 90.00^\circ$ ,  $\beta = 104.174(2)^\circ$ ,  $\gamma = 90.00^\circ$ ,  $V = 1046.29(7)$  Å<sup>3</sup>,  $Z = 4$ ,  $\rho_{\text{calcd}} = 1.309$  mg/m<sup>3</sup>,  $\mu = 0.092$  mm<sup>-1</sup>,  $F(000) = 440$ , reflection collected / unique = 2616 / 1643, refinement method = full-matrix least-squares on  $F^2$ , final  $R$  indices [ $I > 2\sigma(I)$ ]:  $R_1 = 0.0381$ ,  $wR_2 = 0.0946$ ,  $R$  indices (all data):  $R_1 = 0.0603$ ,  $wR_2 = 0.1018$ , goodness of fit = 0.992. CCDC-815735 (for **14a**) contains the supplementary crystallographic data for this paper. These data can be obtained free of charge from The Cambridge Crystallographic Data Centre via [www.ccdc.cam.ac.uk/data\\_request/cif](http://www.ccdc.cam.ac.uk/data_request/cif).

## Spectral Data

**Morpholine-4-carboxylic acid phenylamide (14a):** Brown solid; M.p. 154-156 °C; <sup>1</sup>H NMR (CDCl<sub>3</sub>, 400 MHz) δ (ppm) 3.44 (t, 4H, *J* = 5.2 Hz), 3.69 (t, 4H, *J* = 4.8 Hz), 6.53 (s, 1H), 7.04 (t, 1H, *J* = 6 Hz), 7.27 (t, 2H, *J* = 7.6 Hz), 7.33 (d, 2H, *J* = 8.4 Hz); <sup>13</sup>C NMR (CDCl<sub>3</sub>, 100 MHz) δ (ppm) 44.4, 66.6, 120.6, 123.5, 128.9, 139.0, 155.5; IR (KBr) 3269 (s), 3125 (m), 3053 (m), 2953 (m), 2858 (m), 1633 (s), 1538 (s), 1443 (s), 1416 (s), 1302 (s), 1244 (s), 1113 (s), 992 (m), 873 (m), 859 (m), 746 (s) cm<sup>-1</sup>; Elemental analysis for C<sub>11</sub>H<sub>14</sub>N<sub>2</sub>O<sub>2</sub> (206.24): calcd. C 64.06, H 6.84, N 13.58; found C 64.27, H 6.81, N 13.53.

**Piperidine-1-carboxylic acid phenylamide (14b):** White solid; M.p. 142-144 °C; <sup>1</sup>H NMR (CDCl<sub>3</sub> + DMSO-*d*<sub>6</sub>, 400 MHz) δ (ppm) 1.51-1.58 (m, 6H), 3.41-3.46 (m, 4H), 6.85-6.9 (m, 1H), 7.13-7.18 (m, 2H), 7.39-7.42 (m, 2H), 8.05 (s, 1H); <sup>13</sup>C NMR (CDCl<sub>3</sub> + DMSO-*d*<sub>6</sub>, 100 MHz) δ (ppm) 23.4, 24.8, 44.1, 119.1, 120.9, 127.2, 139.5, 154.5; IR (KBr) 3288 (m), 3129 (w), 2925 (m), 2855 (m), 1628 (s), 1599 (s), 1538 (s), 1448 (s), 1302 (m), 1242 (s), 1028 (m), 905 (w), 872 (w), 754 (s) cm<sup>-1</sup>; C<sub>12</sub>H<sub>16</sub>N<sub>2</sub>O (204.26): calcd. C 70.56, H 7.90, N 13.71; found C 70.34, H 7.88, N 13.68.

**4-Hydroxy-piperidine-1-carboxylic acid phenylamide (14c):** White solid; M.p. 173-175 °C; <sup>1</sup>H NMR (CDCl<sub>3</sub>, 400 MHz) δ (ppm) 1.45-1.53 (m, 2H), 1.83-1.86 (m, 2H), 3.10-3.14 (m, 2H), 3.76-3.79 (m, 1H), 3.94-3.90 (m, 2H), 4.47 (s, 1H), 6.94 (t, 1H, *J* = 7.2 Hz), 7.21 (t, 2H, *J* = 8 Hz), 7.43 (d, 2H, *J* = 8 Hz), 8.02 (s, 1H); <sup>13</sup>C NMR (CDCl<sub>3</sub>, 100 MHz) δ (ppm) 33.6, 41.2, 65.98, 119.5, 121.5, 127.7, 139.6, 154.9; IR (KBr) 3409 (s), 2927 (w), 2256 (w), 2129 (w), 1644 (m), 1537 (w), 1446 (w), 1240 (w), 1048 (s), 1025 (s), 1003 (s), 826 (w), 763 (w) cm<sup>-1</sup>; C<sub>12</sub>H<sub>16</sub>N<sub>2</sub>O<sub>2</sub> (220.26): calcd. C 65.43, H 7.32, N 12.72; found C 65.57, H 7.33, N 12.70.

**1,1-Diethyl-3-phenyl-urea (14d):** Brown solid; M.p. 87 °C; <sup>1</sup>H NMR (CDCl<sub>3</sub>, 400 MHz) δ (ppm) 1.2 (t, 6H, *J* = 6.8 Hz), 3.53 (q, 4H, *J* = 7.2 Hz), 6.40 (s, 1H), 7.0 (t, 1H, *J* = 7.2 Hz), 7.26 (t, 2H, *J* = 8 Hz), 7.39 (d, 2H, *J* = 8.8 Hz); <sup>13</sup>C NMR (CDCl<sub>3</sub>, 100 MHz) δ (ppm) 14.06, 41.7, 120.0, 122.9, 128.9, 139.5, 154.8; IR (KBr) 3307 (m), 2972 (m), 2932 (m), 1637 (s), 1534 (s), 1445 (s), 1405 (m), 1302 (s), 1242 (s), 1167

(m), 1084 (w), 976 (w), 751 (s), 694 (m)  $\text{cm}^{-1}$ ;  $\text{C}_{11}\text{H}_{16}\text{N}_2\text{O}$  (192.25): calcd. C 68.72, H 8.39, N 14.57; found C 68.93, H 8.35, N 14.54.

**1,1-Diisopropyl-3-phenyl-urea (14e):** Brown solid; M.p. 112-115°C;  $^1\text{H}$  NMR ( $\text{CDCl}_3$ , 400 MHz)  $\delta$  (ppm) 1.32 (d, 12H,  $J$  = 6.8 Hz), 3.97 (septet, 2H,  $J$  = 6.8 Hz), 6.24 (s, 1H), 6.97-7.01 (m, 1H), 7.23-7.29 (m, 2H), 7.36 (d, 2H,  $J$  = 7.6 Hz);  $^{13}\text{C}$  NMR (100 MHz,  $\text{CDCl}_3$ )  $\delta$  21.6, 45.6, 119.9, 121.3, 122.7, 128.9, 139.5, 150.2; IR (KBr) 3281 (m), 2969 (m), 2928 (m) 1626 (s), 1588 (s), 1447 (m), 1336 (m), 1230 (m), 1148 (m), 896 (w), 746 (m);  $\text{C}_{13}\text{H}_{20}\text{N}_2\text{O}$  (220.31): calcd. C 70.87, H 9.15, N 12.72; found C 70.65, H 9.12, N 12.67.

**Morpholine-4-carboxylic acid (2-bromo-phenyl)-amide (15a):** Brown Solid; M.p. 126 °C;  $^1\text{H}$  NMR ( $\text{CDCl}_3$ , 400 MHz)  $\delta$  (ppm) 3.52 (t, 4H,  $J$  = 4.8 Hz), 3.76 (t, 4H,  $J$  = 4.8 Hz), 6.91 (t, 1H,  $J$  = 8 Hz), 7.02 (s, 1H), 7.29 (t, 1H,  $J$  = 8.4 Hz), 7.41 (d, 1H,  $J$  = 8 Hz), 8.18 (d, 1H,  $J_1$  = 8.4 Hz);  $^{13}\text{C}$  NMR ( $\text{CDCl}_3$ , 100 MHz)  $\delta$  (ppm) 44.3, 66.6, 94.6, 121.3, 124.0, 128.6, 132.4, 154.4; IR (KBr) 3333 (m), 2915 (w), 2894 (w) 2854 (w), 1636 (s), 1574 (w), 1530 (s), 1402 (m), 1256 (m) 1110 (m), 1026 (w), 993 (w), 752 (m)  $\text{cm}^{-1}$ ;  $\text{C}_{11}\text{H}_{13}\text{BrN}_2\text{O}_2$  (285.13): calcd. C 46.33, H 4.60, N 9.82; found C 46.55, H 4.63, N 9.80.

**Piperidine-1-carboxylic acid (2-bromo-phenyl)-amide (15b):** Brown solid; M.p. 102 °C;  $^1\text{H}$  NMR ( $\text{CDCl}_3$ , 400 MHz)  $\delta$  (ppm) 1.65 (s, 6H), 3.49 (s, 4H), 6.87 (t, 1H,  $J$  = 7.6 Hz), 7.05 (s, 1H), 7.27 (t, 1H,  $J$  = 8 Hz), 7.48 (d, 1H,  $J$  = 8 Hz), 8.19 (d, 1H,  $J$  = 8.4 Hz);  $^{13}\text{C}$  NMR ( $\text{CDCl}_3$ , 100 MHz)  $\delta$  (ppm) 24.5, 25.8, 45.5, 113.3, 121.3, 123.6, 128.5, 132.0, 137.3, 154.3; IR (KBr) 3258 (m), 2936 (m), 2847 (w), 1634 (s), 1506 (s), 1469 (m), 1434 (m), 1271 (m), 1243 (m), 1232 (m), 1024 (w), 749 (m)  $\text{cm}^{-1}$ ;  $\text{C}_{12}\text{H}_{15}\text{BrN}_2\text{O}$  (283.16): calcd. C 50.90, H 5.34, N 9.89; found C 50.71, H 5.30, N 9.82.

**4-Hydroxy-peperidine-1-carboxylic acid (2-bromo-phenyl)-amide (15c):** Gummy;  $^1\text{H}$  NMR ( $\text{CDCl}_3$ , 400 MHz)  $\delta$  (ppm) 1.55-1.63 (m, 2H), 1.92-1.95 (m, 2H), 2.38 (s, 1H), 3.22-3.28 (m, 2H), 3.84-3.94 (m, 3H), 6.89 (t, 1H,  $J$  = 8 Hz), 7.05 (s, 1H), 7.28 (t, 1H,  $J$  = 7.2 Hz), 7.49 (d, 1H,  $J$  = 8 Hz), 8.12 (d, 1H,  $J$  = 8 Hz);  $^{13}\text{C}$  NMR ( $\text{CDCl}_3$ , 100 MHz)  $\delta$  (ppm) 34.0, 41.9, 67.0, 113.5, 121.5, 123.9, 128.5, 132.1, 137.0, 154.3; IR (KBr) 3419 (s), 3318 (s), 2927 (m), 2856 (m), 1644 (s), 1519 (s), 1436 (s), 1298

(s), 1265 (s), 1223 (s), 1065 (s), 1025 (m), 751 (s)  $\text{cm}^{-1}$ ;  $\text{C}_{12}\text{H}_{15}\text{BrN}_2\text{O}_2$  (299.16): calcd. C 48.18, H 5.05, N 9.36; found C 48.29, H 5.01, N 9.31.

**3-(2-bromo-phenyl)-1,1-diethyl-urea (15d):** Gummy;  $^1\text{H}$  NMR ( $\text{CDCl}_3$ , 400 MHz)  $\delta$  (ppm) 1.27 (t, 6H,  $J = 7.2$  Hz), 3.41 (q, 4H,  $J = 7.2$ ), 6.86 (t, 1H,  $J = 7.6$  Hz), 7.01 (s, 1H), 7.27 (t, 1H,  $J = 7.2$ ), 7.48 (d, 1H,  $J = 6.4$ ), 8.26 (d, 1H,  $J = 6.8$  Hz);  $^{13}\text{C}$  NMR ( $\text{CDCl}_3$ , 100 MHz)  $\delta$  (ppm) 13.9, 41.9, 113.0, 121.0, 123.4, 128.4, 131.9, 137.3, 154.0; IR (KBr) 3192 (m), 2961 (m), 1621 (s), 1519 (m), 1475 (s), 1438 (s), 1335 (s), 1149 (s), 1047 (m), 1027 (m), 908 (w), 738 (s)  $\text{cm}^{-1}$ ;  $\text{C}_{11}\text{H}_{15}\text{BrN}_2\text{O}$  (271.15): calcd. C 48.72, H 5.58, N 10.33; found C 48.93, H 5.63, N 10.28.

**Morpholine-4-carboxylic acid (2-methoxy-phenyl)-amide (16a):** White solid; M.p. 108-110 °C;  $^1\text{H}$  NMR ( $\text{CDCl}_3$ , 400 MHz)  $\delta$  (ppm) 3.45 (t, 4H,  $J = 5.2$  Hz), 3.70 (t, 4H,  $J = 5.2$  Hz), 3.84 (s, 3H), 6.84 (d, 1H,  $J = 7.2$  Hz), 6.92-6.95 (m, 2H), 7.09 (s, 1H), 8.12 (d, 1H,  $J = 6.8$  Hz);  $^{13}\text{C}$  NMR ( $\text{CDCl}_3$ , 100 MHz)  $\delta$  (ppm) 44.0, 55.7, 66.4, 109.8, 119.1, 121.06, 122.3, 128.5, 147.7, 154.8; IR (KBr) 3192 (s), 2961 (s), 1621 (s), 1519 (s), 1475 (s), 1438 (s), 1335 (s), 1249 (w), 1149 (s), 1047 (m), 1027 (m), 908 (w), 738 (s)  $\text{cm}^{-1}$ ;  $\text{C}_{12}\text{H}_{16}\text{N}_2\text{O}_3$  (236.26): calcd. C 61.00, H 6.83, N 11.86; found C 61.18, H 6.91, N 11.81;

**Piperidine-1-carboxylic acid (2-methoxy-phenyl)-amide (16b):** Oily liquid;  $^1\text{H}$  NMR ( $\text{CDCl}_3$ , 400 MHz)  $\delta$  (ppm) 1.59 (s, 6H), 3.43 (s, 4H), 3.84 (s, 3H), 6.79-6.82 (m, 1H), 6.88-6.91 (m, 1H), 7.09 (s, 1H), 8.11-8.13 (m, 1H);  $^{13}\text{C}$  NMR ( $\text{CDCl}_3$ , 100 MHz)  $\delta$  (ppm) 24.5, 25.8, 45.2, 55.8, 109.8, 119.0, 121.3, 121.9, 129.2, 147.7, 154.8; IR (KBr) 3449 (m), 2936 (s), 2853 (m), 1661 (s), 1601 (s), 1524 (s), 1458 (s), 1434 (s), 1392 (m), 1247 (s), 1175 (m), 1116 (m), 1026 (s), 984 (w), 748 (s)  $\text{cm}^{-1}$ ;  $\text{C}_{13}\text{H}_{18}\text{N}_2\text{O}_2$  (234.29): calcd. C 66.64, H 7.74, N 11.86; found C 66.45, H 7.80, N 11.81.

**4-Hydroxy-piperidine-1-carboxylic acid (2-methoxy-phenyl)-amide (16c):** Gummy;  $^1\text{H}$  NMR ( $\text{CDCl}_3$ , 400 MHz)  $\delta$  (ppm) 1.49-1.56 (m, 2H), 1.85-1.87 (m, 2H), 3.09-3.15 (m, 2H), 3.81-3.83 (m, 7H), 6.81-6.83 (m, 1H), 6.90-6.94 (m, 2H), 7.12 (s, 1H), 8.03 (d, 1H,  $J = 7.2$  Hz);  $^{13}\text{C}$  NMR ( $\text{CDCl}_3$ , 100 MHz)  $\delta$  (ppm) 33.7, 41.6, 55.7, 66.7, 109.8, 119.2, 120.9, 122.2, 128.5, 147.8, 154.7; IR (KBr) 3437 (m), 2926 (m), 2853 (m), 1644 (s), 1600 (s), 1524 (s), 1460 (s), 1435 (s), 1249 (s), 1218 (s), 1176

(m), 1115 (m), 1026 (m), 972 (w), 750 (m)  $\text{cm}^{-1}$ ;  $\text{C}_{13}\text{H}_{18}\text{N}_2\text{O}_3$  (250.29): calcd. C 62.38, H 7.25, N 11.19; found C 62.59, H 7.32, N 11.13.

**1,1-Diethyl-3-(2-methoxy-phenyl)-urea (16d):** Brown solid; M.p. 61-64 °C;  $^1\text{H}$  NMR ( $\text{CDCl}_3$ , 400 MHz)  $\delta$  (ppm) 1.22 (t, 6H,  $J = 7.2$  Hz), 3.37 (q, 4H,  $J = 7.2$  Hz), 3.85 (s, 3H), 6.81-6.84 (m, 1H), 6.90-6.95 (m, 2H), 7.08 (s, 1H), 8.16-8.19 (m, 1H);  $^{13}\text{C}$  NMR ( $\text{CDCl}_3$ , 100 MHz)  $\delta$  (ppm) 13.9, 41.8, 55.9, 109.7, 119.0, 121.2, 121.7, 129.3, 147.6, 154.5; IR (KBr) 3429 (m), 2973 (m), 2932 (m), 1673 (s), 1590 (s), 1578 (m), 1520 (s), 1435 (s), 1299 (s), 1263 (s), 1158 (s), 1022 (m), 751 (s)  $\text{cm}^{-1}$ ;  $\text{C}_{12}\text{H}_{18}\text{N}_2\text{O}_2$  (222.28): calcd. C 64.84, H 8.16, N 12.60; found C 65.02, H 8.11, N 12.55.

**1,1-Diisopropyl-3-(2-methoxy-phenyl)-urea (16e):** Gummy;  $^1\text{H}$  NMR ( $\text{CDCl}_3$ , 400 MHz)  $\delta$  (ppm) 1.31 (d, 12H,  $J = 6.8$  Hz), 3.87 (s, 3H), 4.32 (septet, 2H,  $J = 6.8$  Hz), 6.86 (s, 1H,  $J = 6.8$  Hz), 6.88 (m, 2H), 6.88-6.95 (m, 2H), 7.09 (s, 1H), 8.21 (d, 1H,  $J = 8$  Hz);  $^{13}\text{C}$  NMR ( $\text{CDCl}_3$ , 100 MHz)  $\delta$  (ppm) 21.5, 45.1, 55.9, 109.8, 118.9, 121.4, 125.6, 128.4, 147.6, 154.7; IR (KBr) 3464 (w), 2971 (m), 2039 (w), 1662 (s), 1600 (m), 1522 (s), 1488 (m), 1458 (s), 1436 (m), 1372 (w), 1337 (m), 1274 (m), 1246 (s), 1149 (m), 1027 (m), 930 (w), 747 (m)  $\text{cm}^{-1}$ ;  $\text{C}_{14}\text{H}_{22}\text{N}_2\text{O}_2$  (250.33): calcd. C 67.17, H 8.86, N 11.19; found C 67.36, H 8.80, N 11.13.

**Morpholine-4-carboxylic acid benzylamide (17a):** Brown solid; M.p. 116-118 °C;  $^1\text{H}$  NMR ( $\text{CDCl}_3$ , 400 MHz)  $\delta$  (ppm) 3.32 (t, 4H,  $J = 4.8$  Hz), 3.63 (t, 4H,  $J = 4.4$  Hz), 4.38-4.40 (m, 2H), 5.05 (s, 1H), 7.25-7.32 (m, 5H);  $^{13}\text{C}$  NMR ( $\text{CDCl}_3$ , 100 MHz)  $\delta$  (ppm) 44.2, 45.0, 66.6, 127.5, 127.8, 128.8, 139.4, 157.9; IR (KBr) 3335 (m), 2921 (m), 2855 (m), 1626 (s), 1539 (s), 1267 (s), 1114 (s), 1067 (m), 1017 (m), 957 (m), 851 (w), 730 (m)  $\text{cm}^{-1}$ ;  $\text{C}_{12}\text{H}_{16}\text{N}_2\text{O}_2$  (220.12): calcd. C 65.43, H 7.32, N 12.72; found C 65.58, H 7.37, N 12.66.

**Piperidine-4-carboxylic acid benzylamide (17b):** White Solid; M.p. 102-104 °C;  $^1\text{H}$  NMR ( $\text{CDCl}_3$ , 400 MHz)  $\delta$  (ppm) 1.52 (m, 6H), 3.31 (m, 4H), 4.36 (s, 2H), 4.8 (brs, 1H), 7.22-7.27 (m, 5H);  $^{13}\text{C}$  NMR ( $\text{CDCl}_3$ , 100 MHz)  $\delta$  (ppm) 24.4, 25.6, 44.8, 44.9, 127.05, 127.59, 128.5, 139.9, 157.8; IR (KBr) 3345 (s), 2933 (m), 2852 (w), 1625 (s), 1538 (s), 1271 (s), 1021 (w), 717 (w)  $\text{cm}^{-1}$ ;  $\text{C}_{13}\text{H}_{18}\text{N}_2\text{O}$  (218.29): calcd. C 71.53, H 8.31, N 12.83; found C 71.66, H 8.38, N 12.78.

**3-Benzyl-1,1-diisopropyl-urea (17e):** White Solid; M.p. 80-82 °C;  $^1\text{H}$  NMR ( $\text{CDCl}_3$ , 400 MHz)  $\delta$  (ppm) 1.24 (d, 12H,  $J$  = 6.8), 3.88 (septet, 2H,  $J$  = 6.8 Hz), 4.44 (d, 1H,  $J$  = 5.2 Hz), 4.56 (s, 1H), 7.22–7.35 (m, 5H);  $^{13}\text{C}$  NMR ( $\text{CDCl}_3$ , 100 MHz)  $\delta$  (ppm) 21.5, 44.8, 45.2, 127.1, 127.6, 128.6, 140.0, 157.2; IR (KBr) 3365 (s), 2964 (w), 2923 (w) 1620 (s), 1543 (m), 1334 (m), 1162 (w), 702 (w)  $\text{cm}^{-1}$ ;  $\text{C}_{14}\text{H}_{22}\text{N}_2\text{O}$  (234.33): calcd. C 71.76, H 9.46, N 11.95; found C 71.88, H 9.52, N 11.89.

**Morpholine-4-carboxylic acid (2-chloro-phenyl)-amide (18a):** Brown Solid; M.p. 127-129 °C;  $^1\text{H}$  NMR ( $\text{CDCl}_3$ , 400 MHz)  $\delta$  (ppm) 3.50 (t, 4H,  $J$  = 4.8), 3.74 (t, 4H,  $J$  = 8.8 Hz), 6.93–6.99 (m, 2H), 7.21–7.26 (m, 1H), 7.32 (d, 1H,  $J$  = 8 Hz), 8.16 (d, 1H,  $J$  = 8.4 Hz);  $^{13}\text{C}$  NMR ( $\text{CDCl}_3$ , 100 MHz)  $\delta$  (ppm) 44.3, 66.6, 121.2, 122.6, 123.5, 127.9, 128.9, 135.7, 154.4; IR (KBr) 3218 (m), 2963 (m), 2887 (w), 2853 (m), 1632 (s), 1513 (s), 1381 (m), 1270 (m), 1251 (s), 1119 (s), 1000 (m), 752 (m)  $\text{cm}^{-1}$ ;  $\text{C}_{11}\text{H}_{13}\text{ClN}_2\text{O}_2$  (240.69): calcd. C 54.89, H 5.44, N 11.64; found C 55.04, H 5.51, N 11.58.

**Morpholine-4-carboxylic acid (4-cyano-phenyl)-amide (19a):** White Solid; M.p. 170-172 °C;  $^1\text{H}$  NMR ( $\text{CDCl}_3$ , 400 MHz)  $\delta$  (ppm) 3.51 (t, 4H,  $J$  = 4.8 Hz), 3.74 (t, 4H,  $J$  = 4.4 Hz), 6.95 (s, 1H), 7.50–7.56 (m, 4H);  $^{13}\text{C}$  NMR ( $\text{CDCl}_3$ , 100 MHz)  $\delta$  (ppm) 44.5, 66.5, 105.5, 117.2, 119.5, 133.3, 143.7, 154.4; IR (KBr) 3438 (s), 3280 (s), 2983 (w), 2225 (w), 1639 (s), 1504 (m), 1425 (m), 1248 (m), 1112 (m), 841 (m)  $\text{cm}^{-1}$ ;  $\text{C}_{12}\text{H}_{13}\text{N}_3\text{O}_2$  (231.25): calcd. C 62.33, H 5.67, N 18.17; found C 62.46, H 5.76, N 18.11.

**Morpholine-4-carboxylic acid (4-trifluoromethyl-phenyl)-amide (20a):** Oily;  $^1\text{H}$  NMR ( $\text{CDCl}_3$ , 400 MHz)  $\delta$  (ppm) 3.51 (t, 4H,  $J$  = 4.8 Hz), 3.73 (t, 4H,  $J$  = 4.4 Hz), 7.0 (s, 1H), 7.55 (d, 2H,  $J$  = 8.4 Hz), 6.65 (d, 2H,  $J$  = 8.8 Hz);  $^{13}\text{C}$  NMR ( $\text{CDCl}_3$ , 100 MHz)  $\delta$  (ppm) 44.4, 66.5, 118.1, 119.8, 126.3, 126.5, 140.0, 159.7; IR (KBr) 3283 (m), 3131 (w), 2925 (w), 2864 (w), 1682 (m) 1614 (m), 1538 (m), 1415 (m), 1326 (s), 1250 (m), 1164 (m), 1112 (s), 1067 (s), 1016 (w), 839 (m)  $\text{cm}^{-1}$ ;  $\text{C}_{12}\text{H}_{13}\text{F}_3\text{N}_2\text{O}_2$  (274.23): calcd. C 52.56, H 4.78, N 10.21; found C 52.69, H 4.82, N 10.16

**Morpholine-4-carboxylic acid (2,4-dimethyl-phenyl)-amide (21a):** White Solid; M.p. 160-162 °C;  $^1\text{H}$  NMR ( $\text{CDCl}_3$ , 400 MHz)  $\delta$  (ppm) 2.13 (s, 3H), 2.27 (s, 3H), 3.30 (t, 4H  $J$  = 4.4 Hz), 3.58 (t, 4H,  $J$  = 4.4 Hz), 6.45 (s, 1H), 6.92–6.95 (m, 2H), 7.19

(d, 1H,  $J = 8$  Hz);  $^{13}\text{C}$  NMR ( $\text{CDCl}_3$ , 100 MHz)  $\delta$  (ppm) 17.8, 20.9, 44.3, 66.6, 124.8, 127.1, 131.1, 131.4, 134.2, 134.6, 156.2; IR (KBr) 3262 (s), 2966 (m), 2892 (m), 2853 (s), 1638 (s), 1505 (s), 1385 (m), 1254 (s), 1118 (s), 1000 (w), 810 (m)  $\text{cm}^{-1}$ ;  $\text{C}_{13}\text{H}_{18}\text{N}_2\text{O}_2$  (234.13): calcd. C 66.64, H 7.74, N 11.96; found C 66.79, H 7.80, N 11.90.

**Morpholine-4-carboxylic acid cyclohexylamide (22a):** White Solid; M.p. 178-180 °C;  $^1\text{H}$  NMR ( $\text{CDCl}_3$ , 400 MHz)  $\delta$  (ppm) 1.12 (t, 3H,  $J = 11.6$ ), 1.35 (q, 3H,  $J = 12.4$  Hz), 1.62 (d, 1H,  $J = 12.4$  Hz), 1.69–1.72 (m, 2H), 1.93 (d, 2H,  $J = 11.2$  Hz), 3.33 (t, 3H,  $J = 4.8$  Hz), 3.68 (t, 5H,  $J = 4.4$  Hz), 4.5 (brs, 1H);  $^{13}\text{C}$  NMR ( $\text{CDCl}_3$ , 100 MHz)  $\delta$  (ppm) 25.1, 25.7, 33.9, 49.5, 66.5, 157.3; IR (KBr) 3310 (s), 2930 (s), 2855 (s), 1615 (s), 1542 (s), 1414 (m), 1275 (s), 1253 (m), 1109 (s), 1076 (m), 999 (w)  $\text{cm}^{-1}$ ;  $\text{C}_{11}\text{H}_{20}\text{N}_2\text{O}_2$  (212.28): calcd. C 62.23, H 9.50, N 13.20; found C 62.38, H 9.54, N 13.15.

**Morpholine-4-carboxylic acid butylamide (23a):** Gummy;  $^1\text{H}$  NMR ( $\text{CDCl}_3$ , 400 MHz)  $\delta$  (ppm) 0.83-0.87 (m, 2H), 1.18 (s, 2H), 1.24-1.34 (m, 2H), 1.38-1.45 (m, 1H), 3.13-3.18 (m, 2H), 3.27 (t, 4H,  $J = 5.2$  Hz), 3.61 (t, 4H,  $J = 4.8$  Hz);  $^{13}\text{C}$  NMR ( $\text{CDCl}_3$ , 100 MHz)  $\delta$  (ppm) 13.9, 20.2, 32.4, 40.8, 44.1, 66.6, 158.2; IR (KBr) 3350 (m), 2958 (s), 2926 (s), 2856 (s), 1629 (s), 1542 (s), 1456 (m), 1302 (m), 1266 (s), 1119 (s), 1071 (w), 994 (w), 860 (w)  $\text{cm}^{-1}$ ;  $\text{C}_9\text{H}_{18}\text{N}_2\text{O}_2$  (186.25): calcd. C 58.04, H 9.74, N 15.04; found C 58.18, H 9.70, N 15.01.

**Morpholine-4-carboxylic acid naphthalen-2-ylamide (24a):** Brown Solid; M.p. 198-200 °C;  $^1\text{H}$  NMR ( $\text{CDCl}_3$ , 400 MHz)  $\delta$  (ppm) 3.26 (s, 4H), 3.48 (s, 4H), 6.87 (s, 1H), 7.35-7.37 (m, 1H), 7.43-7.44 (m, 3H), 7.63 (d, 1H,  $J = 7.6$ ), 7.75-7.77 (m, 1H), 7.80-7.82 (m, 1H);  $^{13}\text{C}$  NMR ( $\text{CDCl}_3$ , 100 MHz)  $\delta$  (ppm) 44.4, 66.5, 121.8, 122.1, 125.8, 126.1, 126.2, 128.7, 128.9, 134.1, 134.4, 154.5; IR (KBr) 3439 (m), 3296 (m), 2856 (m), 1633 (s), 1520 (m), 1501 (m), 1377 (w), 1255 (m), 1119 (m), 792 (w)  $\text{cm}^{-1}$ ;  $\text{C}_{15}\text{H}_{16}\text{N}_2\text{O}_2$  (256.29): calcd. C 70.29, H 6.29, N 10.93; found C 70.08, H 6.21, N 10.88.

**Piperidine-1-carboxylic acid (2-chloro-phenyl)-amide (18b):** Brown Solid; M.p. 103-105 °C;  $^1\text{H}$  NMR ( $\text{CDCl}_3$ , 400 MHz)  $\delta$  (ppm) 1.6 (s, 6H), 3.44 (s, 4H), 6.88 (t, 1H,  $J = 7.6$  Hz), 6.99 (s, 1H), 7.18 (t, 1H,  $J = 8$  Hz), 7.27 (d, 1H,  $J = 8$  Hz), 8.15 (d,

1H,  $J = 8.4$  Hz);  $^{13}\text{C}$  NMR ( $\text{CDCl}_3$ , 100 MHz)  $\delta$  (ppm) 24.4, 25.7, 45.3, 121.0, 122.3, 122.9, 127.7, 128.8, 136.2, 154.2; IR (KBr) 3218 (m), 2961 (w), 2286 (w), 2853 (m), 1632 (s), 1513 (s), 1381 (m), 1251 (s), 1119 (s), 1000 (m), 752 (m)  $\text{cm}^{-1}$ ;  $\text{C}_{12}\text{H}_{15}\text{ClN}_2\text{O}$  (238.71): calcd. C 60.38, H 6.33, N 11.74; found C 60.20, H 6.25, N 11.68.

**Piperidine-4-carboxylic acid (3-nitro-phenyl)-amide (25b):** White Solid; M.p. 127-129 °C;  $^1\text{H}$  NMR ( $\text{CDCl}_3$ , 400 MHz)  $\delta$  (ppm) 1.58–1.63 (m, 6H), 3.44–3.49 (m, 4H), 7.33 (t, 1H,  $J = 8$  Hz), 7.45–7.49 (d, 1H), 7.76 (t, 2H,  $J = 9.6$  Hz), 8.22 (s, 1H);  $^{13}\text{C}$  NMR ( $\text{CDCl}_3$ , 100 MHz)  $\delta$  (ppm) 24.4, 25.8, 45.4, 114.7, 117.2, 125.9, 129.4, 141.1, 148.4, 154.8; IR (KBr) 3312 (m), 3097 (w), 2936 (m), 2856 (m), 1644 (s), 1594 (w), 1530 (s) 1485 (s), 1434 (s), 1349 (s), 1278 (w), 1242 (s), 1145 (w), 1024 (w), 799 (w), 736 (m)  $\text{cm}^{-1}$ ;  $\text{C}_{12}\text{H}_{15}\text{N}_3\text{O}_3$  (249.26): calcd. C 57.82, H 6.07, N 16.86; found C 57.96, H 6.15, N 16.81.

**Piperidine-1-carboxylic acid (2,4-dimethyl-phenyl)-amide (21b):** White Solid; M.p. 130-132 °C;  $^1\text{H}$  NMR ( $\text{CDCl}_3$ , 400 MHz)  $\delta$  (ppm) 1.58–1.61 (m, 6H), 2.18 (s, 3H), 2.27 (s, 3H), 3.39–3.42 (m, 4H), 6.11 (s, 1H), 6.95–6.96 (m, 2H), 7.40 (s, 1H);  $^{13}\text{C}$  NMR ( $\text{CDCl}_3$ , 100 MHz)  $\delta$  (ppm) 17.8, 20.8, 24.5, 25.7, 45.3, 123.8, 127.1, 129.9, 130.9, 133.6, 134.7, 155.8; IR (KBr) 3297 (s), 2940 (s), 2919 (s), 2852 (s), 1633 (s), 1501 (s), 1453 (m), 1244 (s), 1031 (w), 808 (m)  $\text{cm}^{-1}$ ;  $\text{C}_{14}\text{H}_{20}\text{N}_2\text{O}$  (232.32): calcd. C 72.38, H 8.68, N 12.06; found C 72.55, H 8.61, N 12.01.

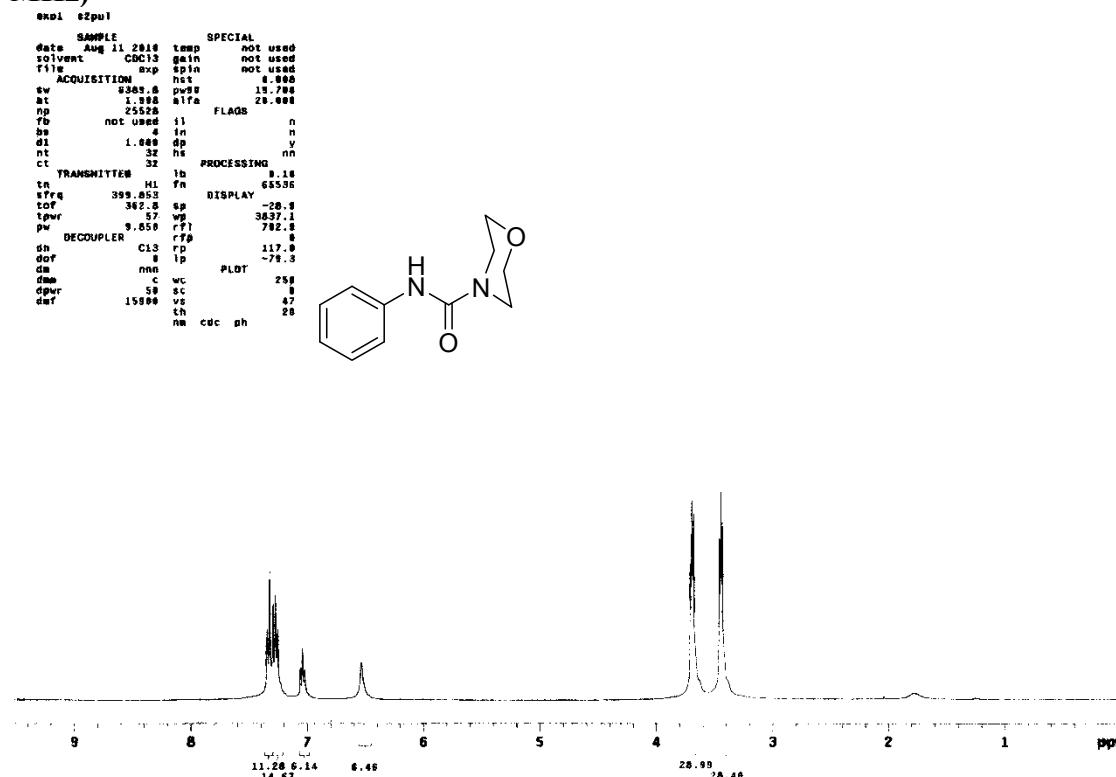
**Piperidine-1-carboxylic acid (3,4-dimethyl-phenyl)-amide (26b):** White Solid; M.p. 103-105 °C;  $^1\text{H}$  NMR ( $\text{CDCl}_3$ , 400 MHz)  $\delta$  (ppm) 1.55–1.56 (m, 6H), 2.15 (s, 3H), 2.17 (s, 3H), 3.38–3.39 (m, 4H), 6.51 (s, 1H), 6.96 (d, 1H,  $J = 8$  Hz), 7.02 (d, 1H,  $J = 7.6$  Hz), 7.13 (s, 1H);  $^{13}\text{C}$  NMR ( $\text{CDCl}_3$ , 100 MHz)  $\delta$  (ppm) 19.1, 19.9, 24.5, 25.8, 45.3, 117.8, 121.8, 129.8, 131.0, 136.9, 137.1, 155.5; IR (KBr) 3296 (m), 2932 (s), 2853 (s), 1633 (s), 1597 (s), 1531 (s), 1417 (s), 1303 (m), 1250 (s), 1232 (s), 1024 (m), 814 (m)  $\text{cm}^{-1}$ ;  $\text{C}_{14}\text{H}_{20}\text{N}_2\text{O}$  (232.32): calcd. C 72.38, H 8.68, N 12.06; found C 72.19, H 8.61, N 12.01.

***N*-(*E*)-(4-(Trifluoromethyl)phenylimino)(morpholino)methyl-*N*-(4-(trifluoromethyl) phenyl) morpholine-4-carbothioamide (11):** White Solid; M.p. 147 °C;  $^1\text{H}$  NMR ( $\text{CDCl}_3$ , 400 MHz)  $\delta$  (ppm) 2.92–3.95 (m, 16H), 6.90 (brs, 2H),

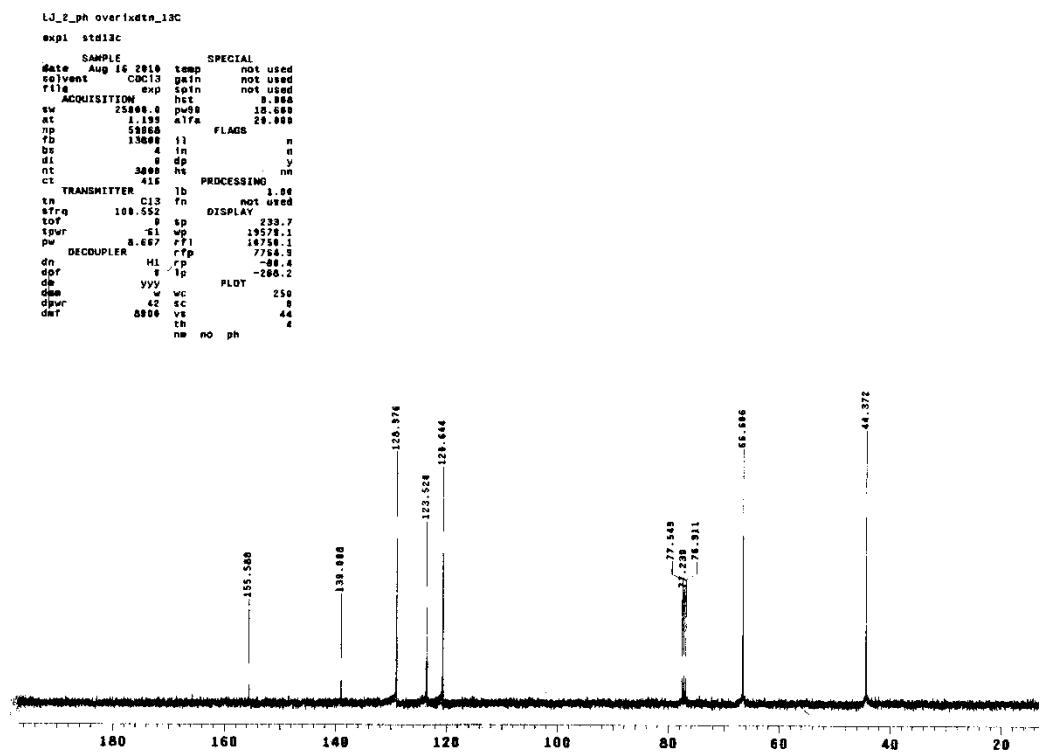
7.07 (d, 2H,  $J = 8.0$  Hz), 7.47 (d, 2H,  $J = 8.0$  Hz), 7.62 (brs, 2H);  $^{13}\text{C}$  NMR ( $\text{CDCl}_3$ , 100 MHz)  $\delta$  (ppm) 46.9, 51.0, 65.4, 66.2, 120.5, 121.1, 122.3, 122.4, 124.9, 125.2, 125.3, 125.9, 126.1, 126.5, 126.8, 127.2, 145.4, 148.9, 152.5, 184.5; IR (KBr): 3395 (w), 2966 (w), 2906 (w), 2858 (w), 1639 (0, 1602 (s), 1424 (m), 1323 (s), 1292 (s), 1236 (s), 1159 (s), 1114 (s), 1064 (s), 1013 (m), 999 (m), 844 (m)  $\text{cm}^{-1}$ ;  $\text{C}_{24}\text{H}_{24}\text{F}_6\text{N}_4\text{O}_2\text{S}$  (546.53): calcd C 52.74, H 4.43, N 10.25, S 5.87; found: C 52.83, H 4.46, N 10.21, S 5.82.

## SPECTRA

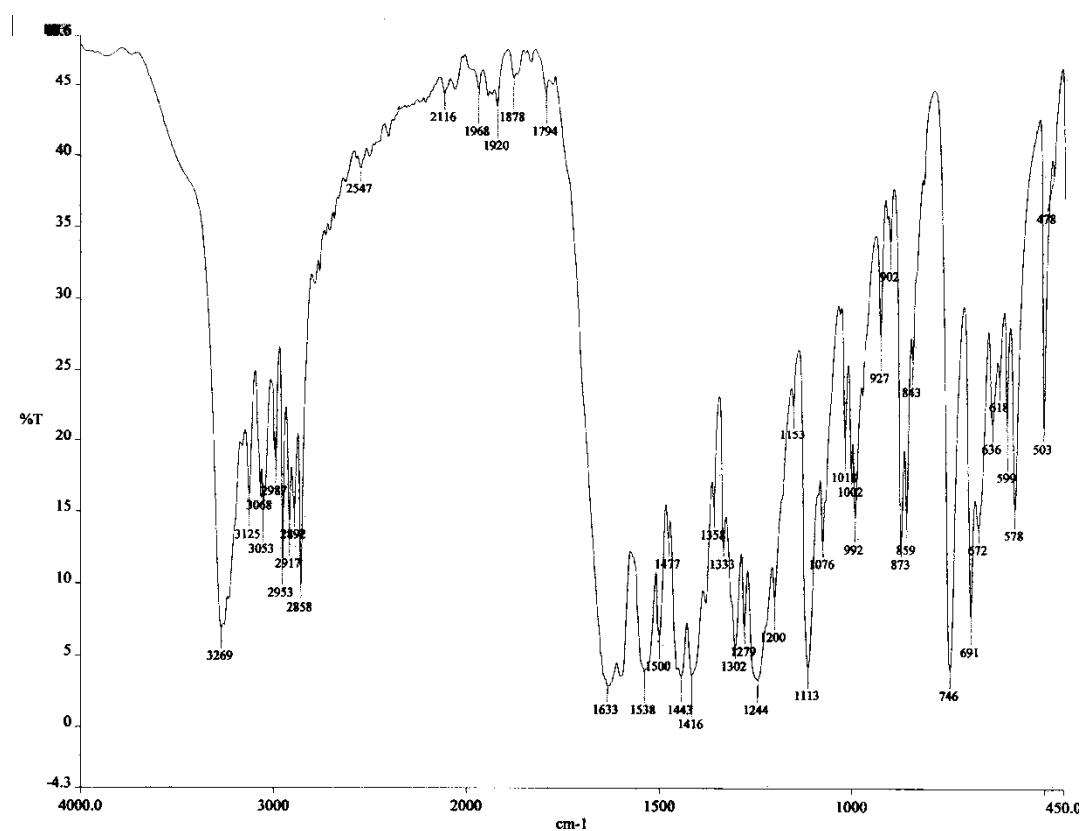
Morpholine-4-carboxylic acid phenylamide (14a):  $^1\text{H}$  NMR ( $\text{CDCl}_3$ , 400 MHz)



Morpholine-4-carboxylic acid phenylamide (14a):  $^{13}\text{C}$  NMR ( $\text{CDCl}_3$ , 100 MHz)

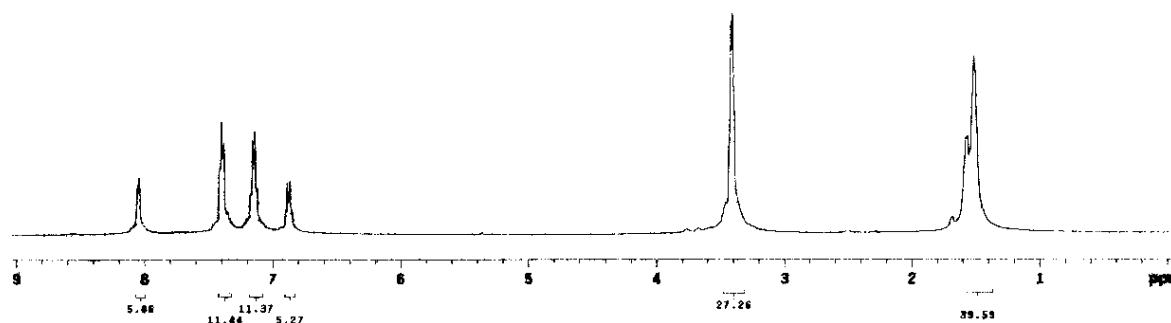
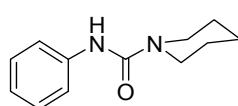


**Morpholine-4-carboxylic acid phenylamide (14a): IR (KBr)**

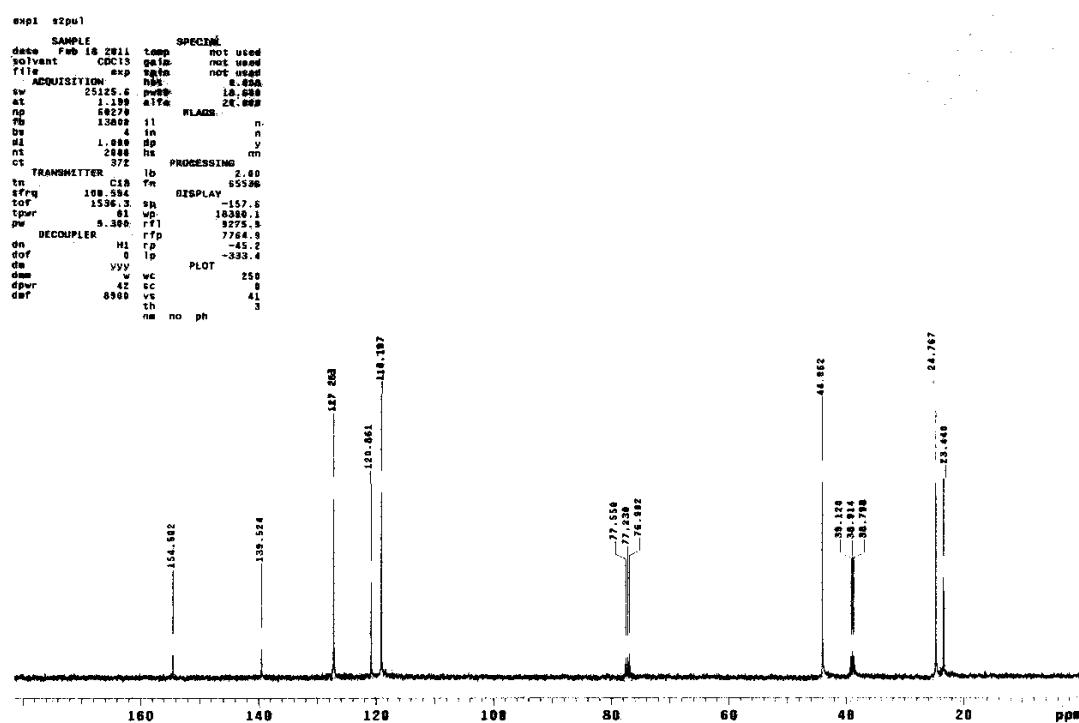


**Piperidine-1-carboxylic acid phenylamide (14b):  $^1\text{H}$  NMR ( $\text{CDCl}_3 + \text{DMSO}-d_6$ , 400 MHz)**

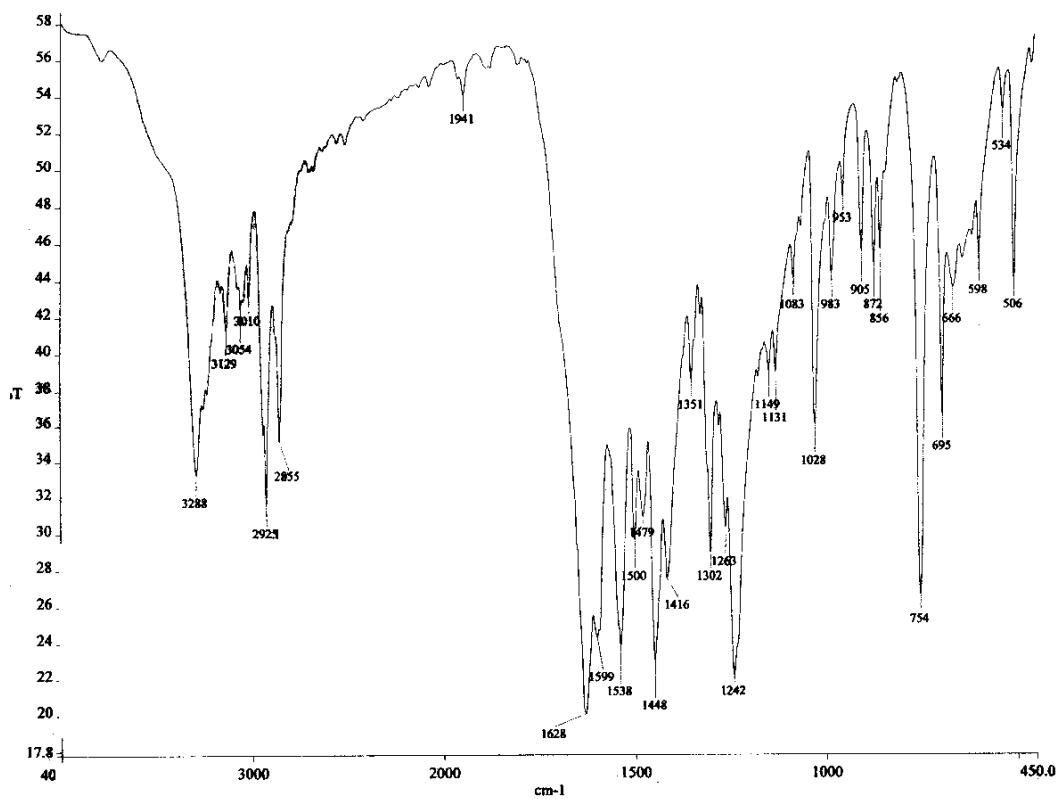
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dm YYY PLOT 256  
dmm W WC 6  
dpw 42 SC 43  
dft 8900 VS 3  
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Piperidine-1-carboxylic acid phenylamide (14b):  $^{13}\text{C}$  NMR ( $\text{CDCl}_3 + \text{DMSO}-d_6$ , 100 MHz)

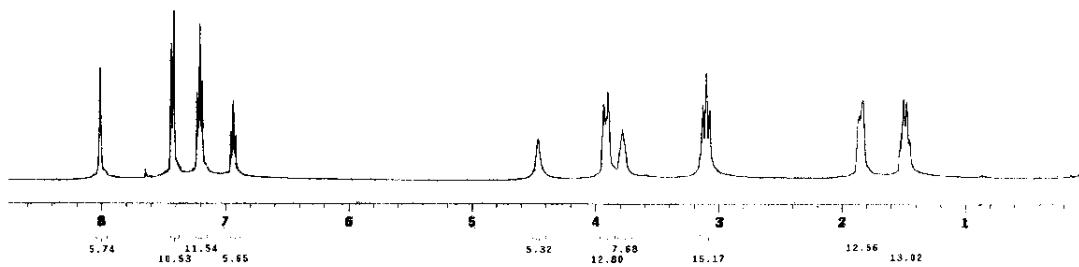
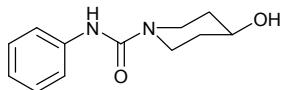


Piperidine-1-carboxylic acid phenylamide (14b): IR (KBr)



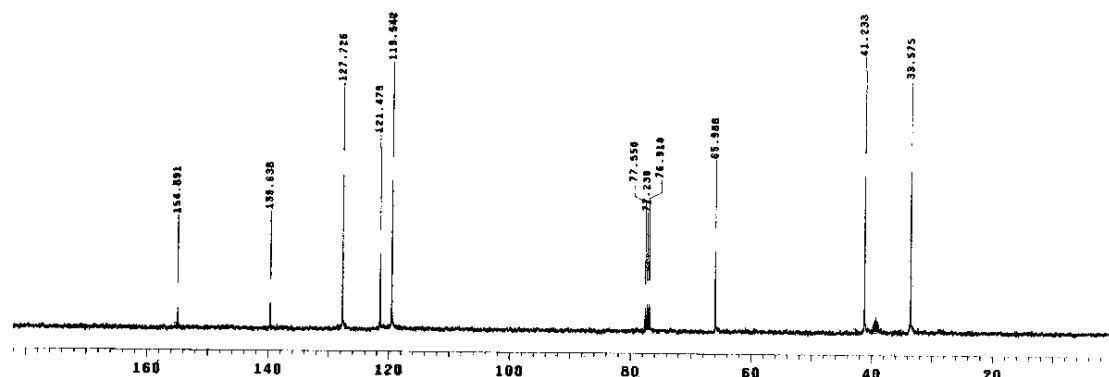
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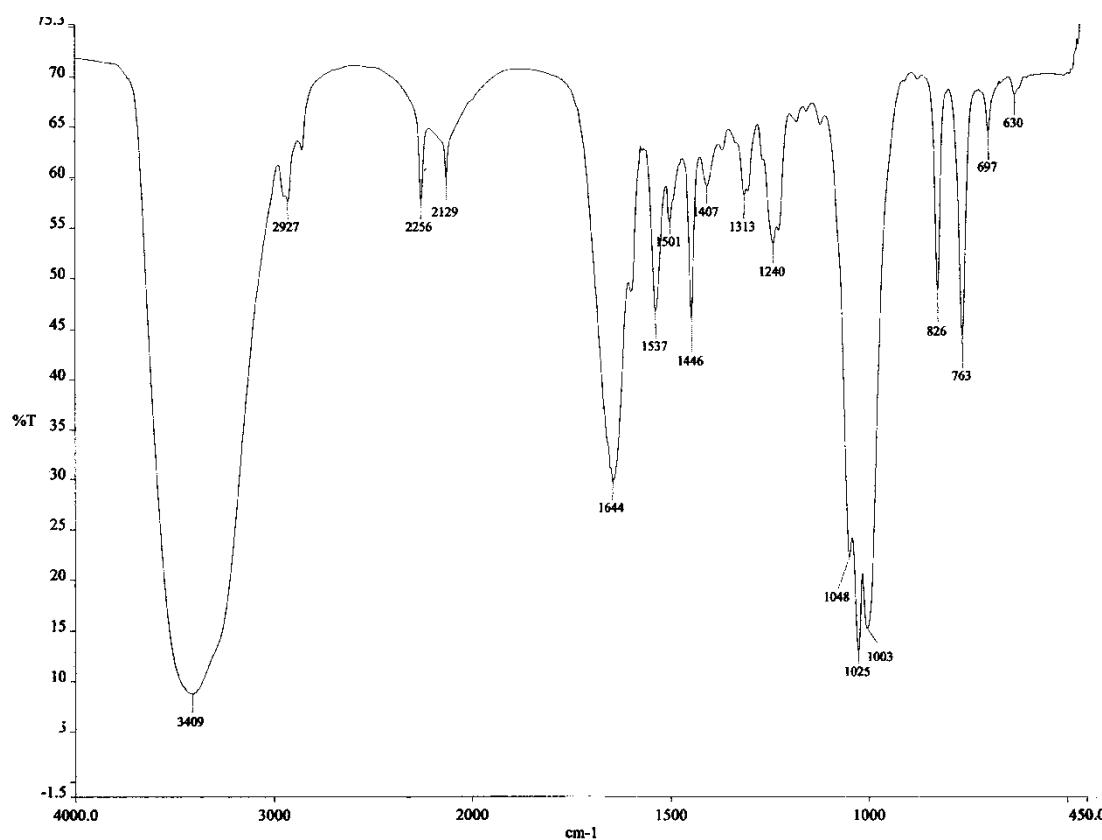


**4-Hydroxy-piperidine-1-carboxylic acid phenylamide (14c):  $^{13}\text{C}$  NMR ( $\text{CDCl}_3$ , 100 MHz)**

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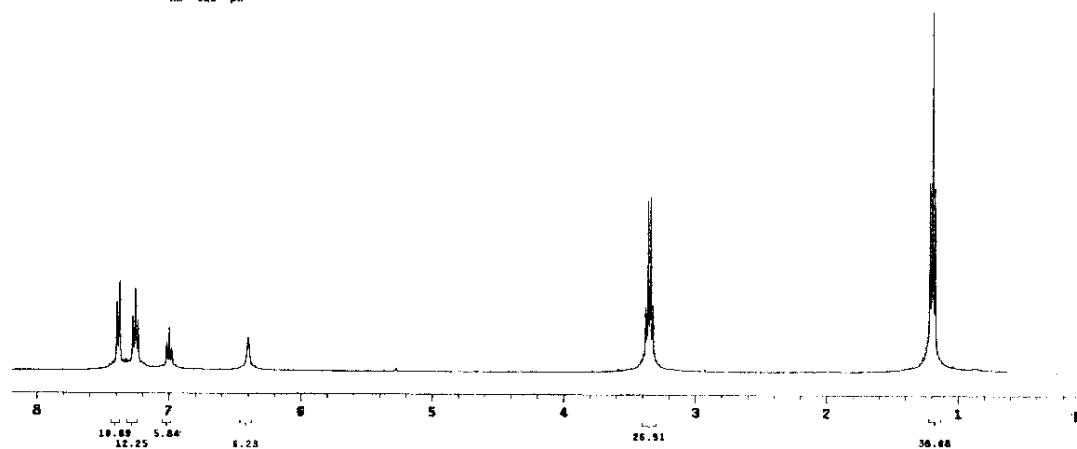
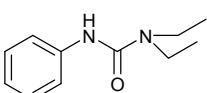


**4-Hydroxy-piperidine-1-carboxylic acid phenylamide (14c): IR (KBr)**

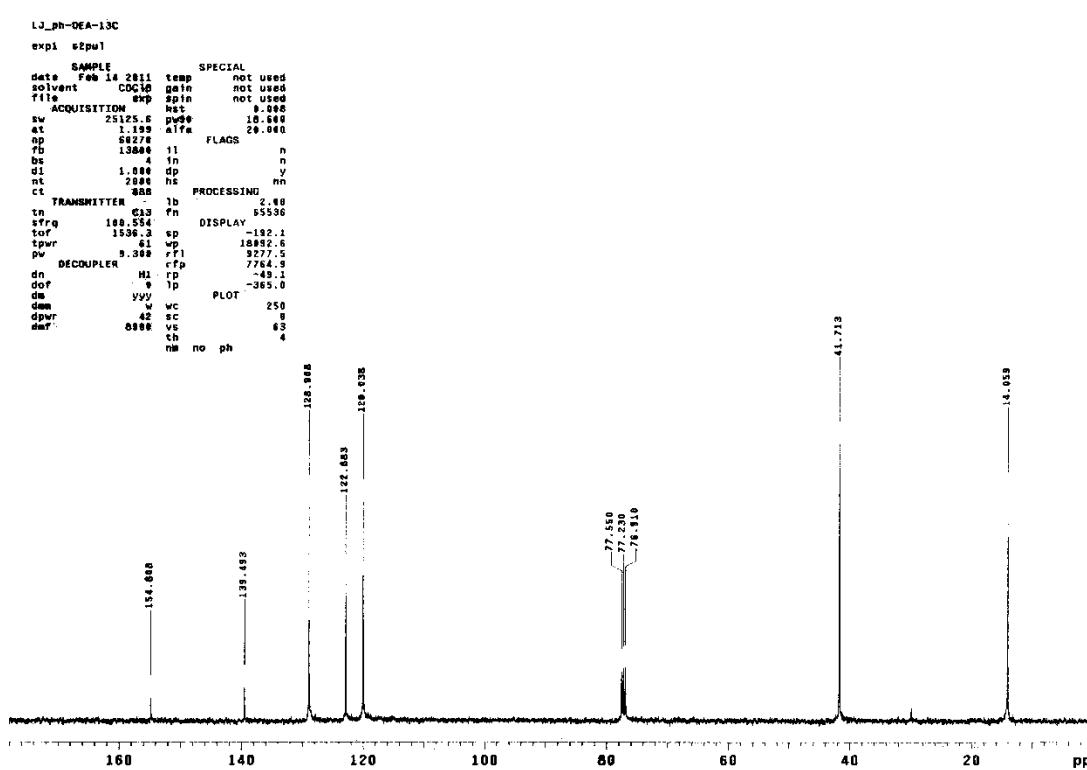


**1,1-Diethyl-3-phenyl-urea (14d): <sup>1</sup>H NMR (CDCl<sub>3</sub>, 400 MHz)**

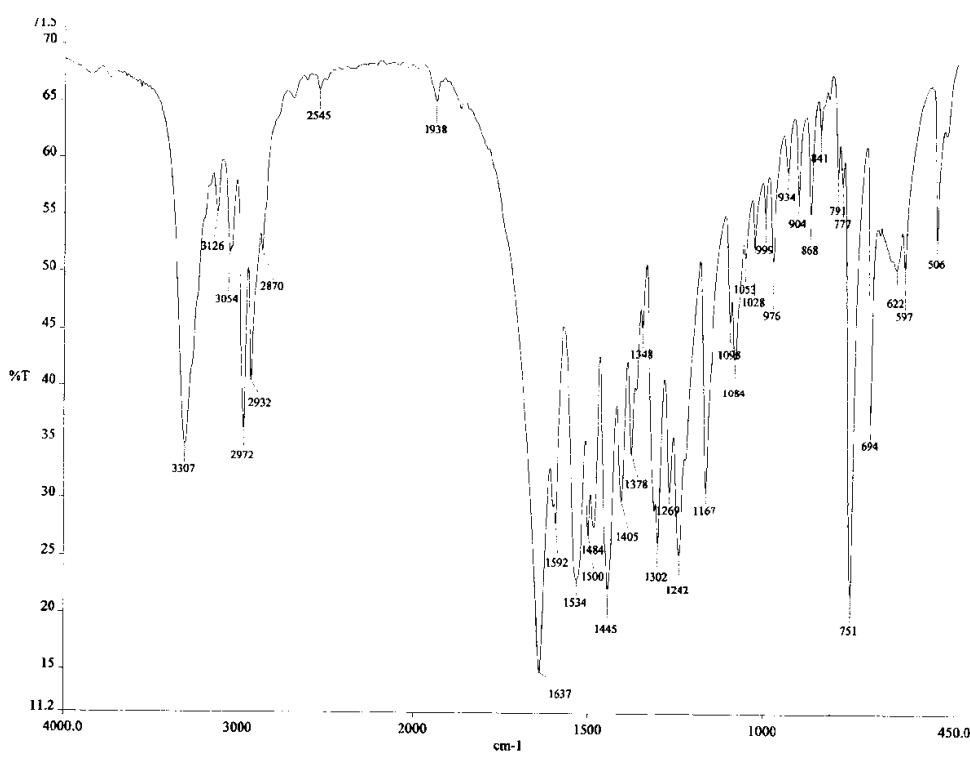
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dof 1p
dpp -99.3
dm ann
plot
dpp c 250
dpp g ec 8
dpp t th 20
nm cdc ph
```



1,1-Diethyl-3-phenyl-urea (14d):  $^{13}\text{C}$  NMR ( $\text{CDCl}_3$ , 100 MHz)

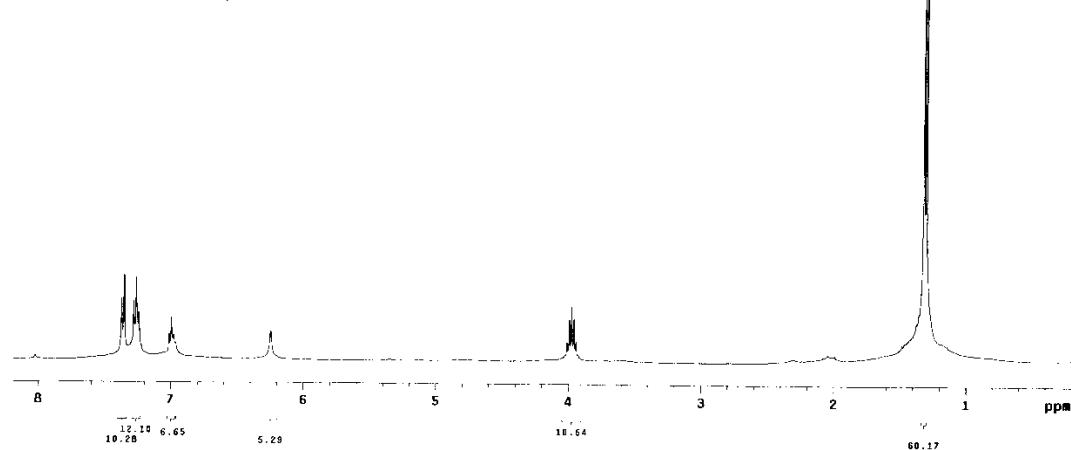
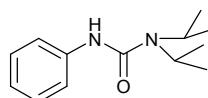


1,1-Diethyl-3-phenyl-urea (14d): IR (KBr)



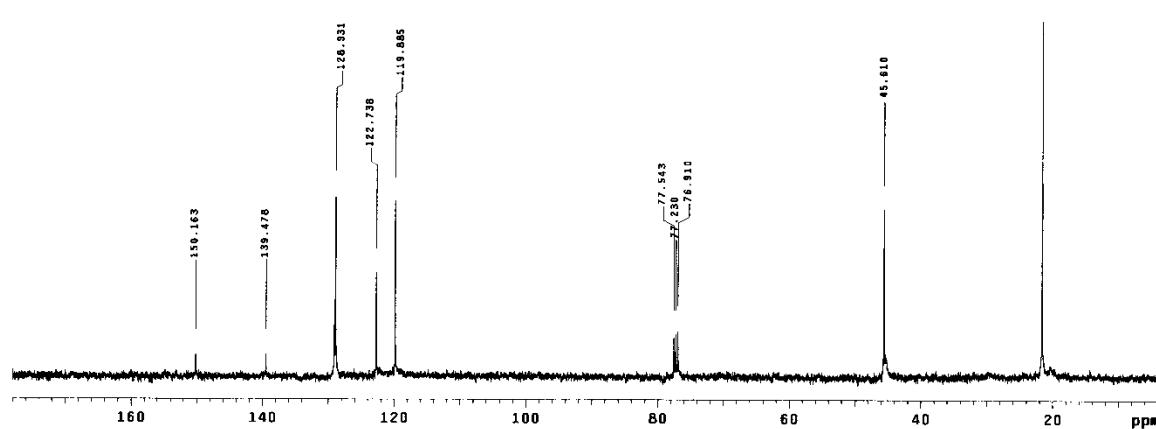
**1,1-Diisopropyl-3-phenyl-urea (14e):  $^1\text{H}$  NMR ( $\text{CDCl}_3$ , 400 MHz)**

```
expt s2pul
SAMPLE          SPECIAL
date Mar 19 2011 temp not used
solvent   CDCl3 gain not used
file    exp spin not used
ACQUISITION hst n 0.000
sw      339.8 pw90 19.708
at      1.938 alfa 20.303
nt      256   flags
rfb     not used 11  n
bs      4   in  n
d1      1.000 dp  y
mt      32   hs  nn
ct      32   PROCESSING nn
TRANSMITTER 1b  0.10
tn      HI fn 65538
sfrq    339.853 DISPLAY
t0f     362.8 sp 66.3
tpwr    57 w1 3207.5
pw      8.850 r1 796.4
DECOUPLER   rfp 0
dn      C13 rp 119.3
dof     0 1p -100.5
de      nm  PLOT
dm      c wc 256
dpwr    50 sc 0
def     15900 ts 161
th      nm cdc ph 16
```

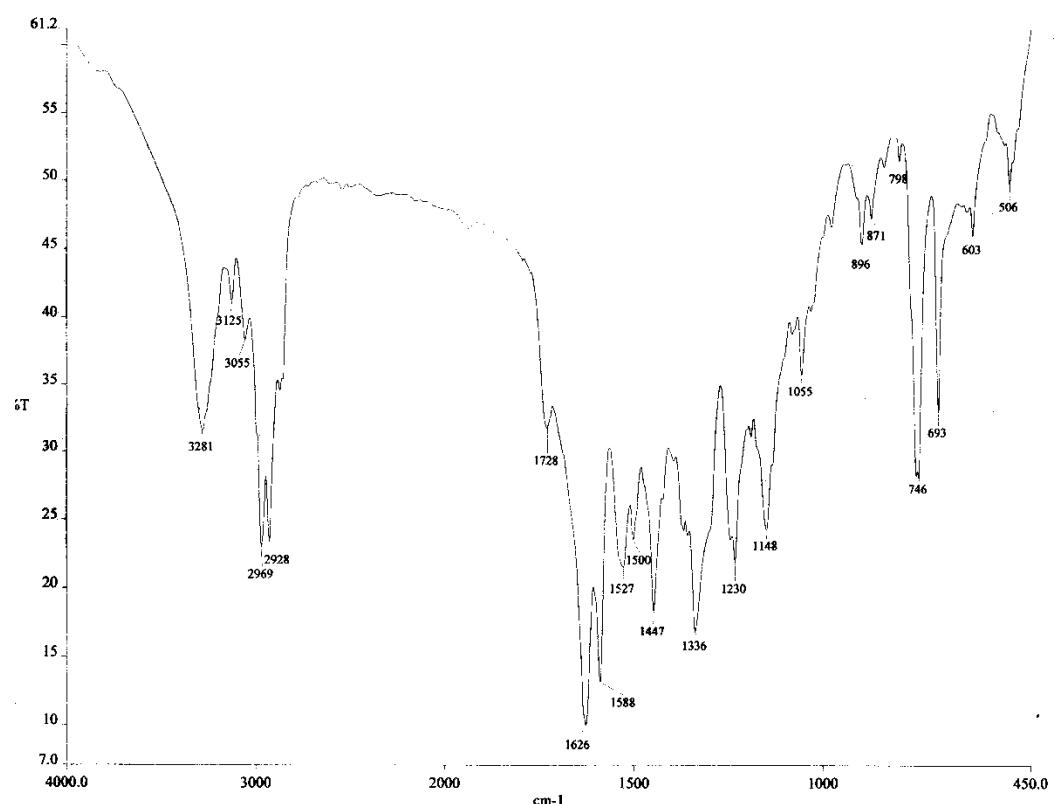


**1,1-Diisopropyl-3-phenyl-urea (14e):  $^{13}\text{C}$  NMR ( $\text{CDCl}_3$ , 100 MHz)**

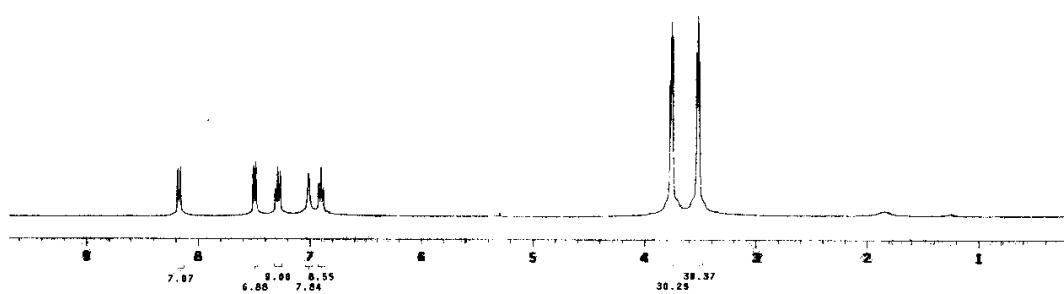
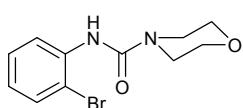
```
expt s2pul
SAMPLE          SPECIAL
date Mar 19 2011 temp not used
solvent   CDCl3 gain not used
file    exp spin not used
ACQUISITION hst n 0.000
sw      2025.6 pw16 16.000
at      1.155 alfa 20.000
nt      60270 flags
rfb     13800 t1  n
bs      1.000 in  n
d1      1.000 dp  y
mt      1000 hs  nn
ct      328 PROCESSING nn
TRANSMITTER 1b  2.00
tn      C13 fn 65538
sfrq    100.554 DISPLAY
t0f     1536.3 sp 300.9
tpwr    61 w1 17557.2
pw      5.300 r1 9279.8
DECOUPLER   rfp 77.0
dn      HI rp 77.0
dof     0 1p -344.3
de      vvv PLOT
dm      v wc 256
dpwr    42 sc 0
def     8500 ts 77
th      nm no ph 4
```



#### **1,1-Diisopropyl-3-phenyl-urea (14e): IR (KBr)**

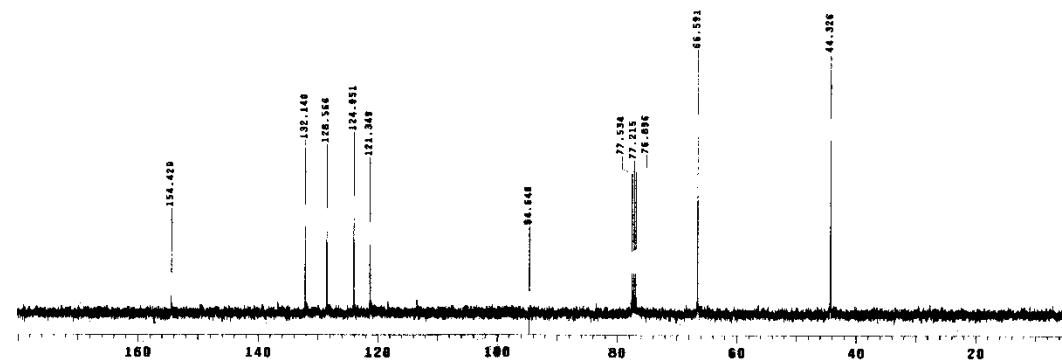


**Morpholine-4-carboxylic acid (2-bromo-phenyl)-amide (15a):  $^1\text{H}$  NMR ( $\text{CDCl}_3$ , 400 MHz)**

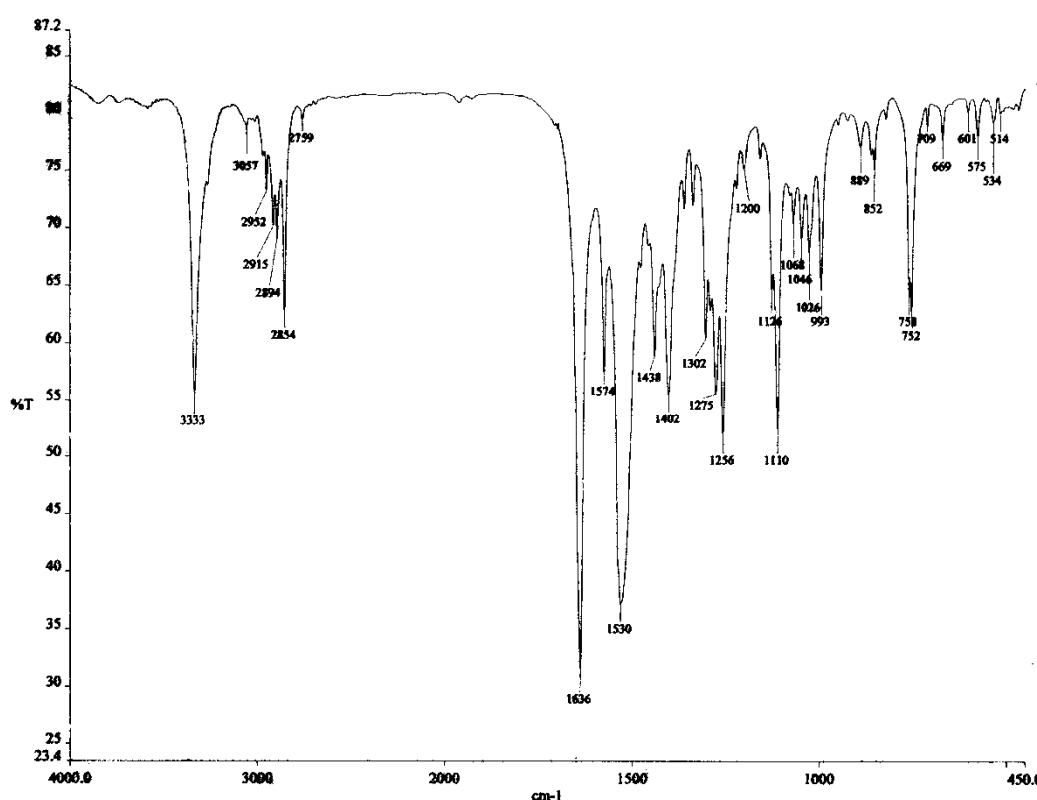


**Morpholine-4-carboxylic acid (2-bromo-phenyl)-amide (15a):  $^{13}\text{C}$  NMR ( $\text{CDCl}_3$ , 100 MHz)**

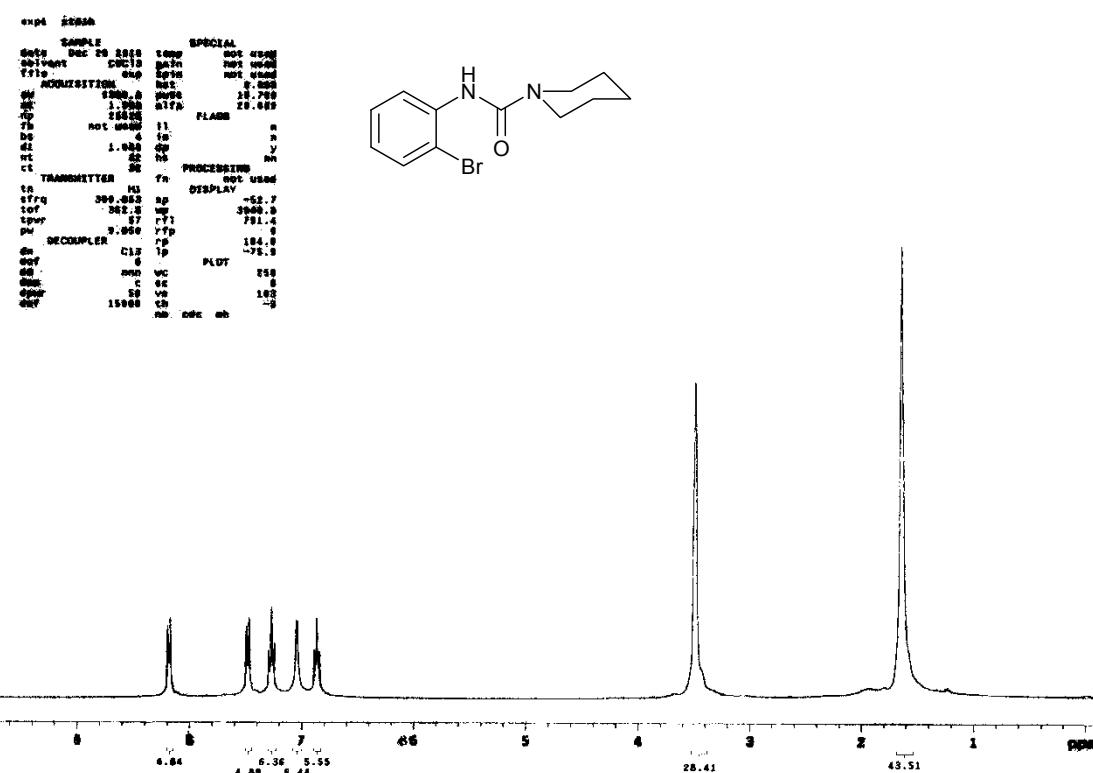
```
expt std13c
SAMPLE
date Dec 24 2010 temp not used
solvent CDCl3 gain not used
t1flip exp spin not used
ACQUISITION n1 8.00
sw 2500.0 p100 11.700
at 1.199 alfa 28.000
np 59984 flags
td 13000 11 n
bs 4 in n
dt 64 dp y
rt 60.00 ns nn
ct 432 tb PROCESSING
TRANSMITTER C13 fn 1.00
tr C13 fn not used
stfrq 100.532 DISPLAY
tof 0 sp -125.7
tpwr 61 wp 18232.5
pw 8.687 r17 18748.6
DECOUPLER H1 r17 7748.6
dn HI rp -35.8
dof s ip -493.5
ds vyy w PLOT
doe 250
dpwr 42 ec 0
def 8898 vs 41
d1 4 no ph
```



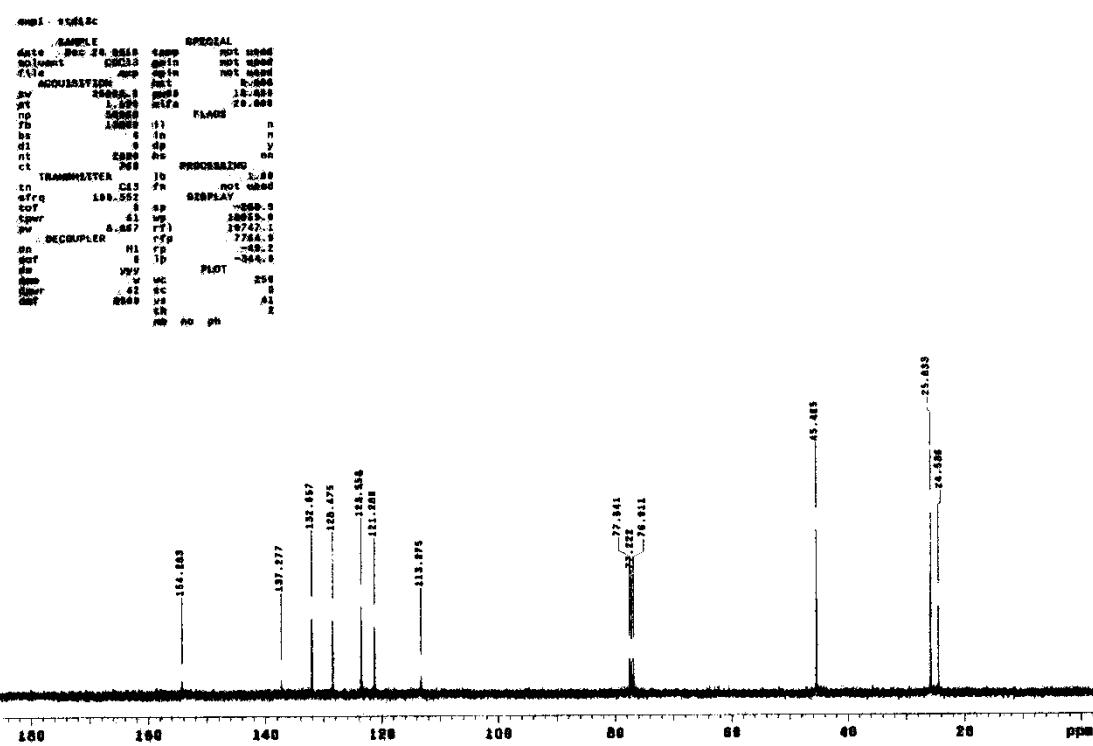
**Morpholine-4-carboxylic acid (2-bromo-phenyl)-amide (15a): IR (KBr)**



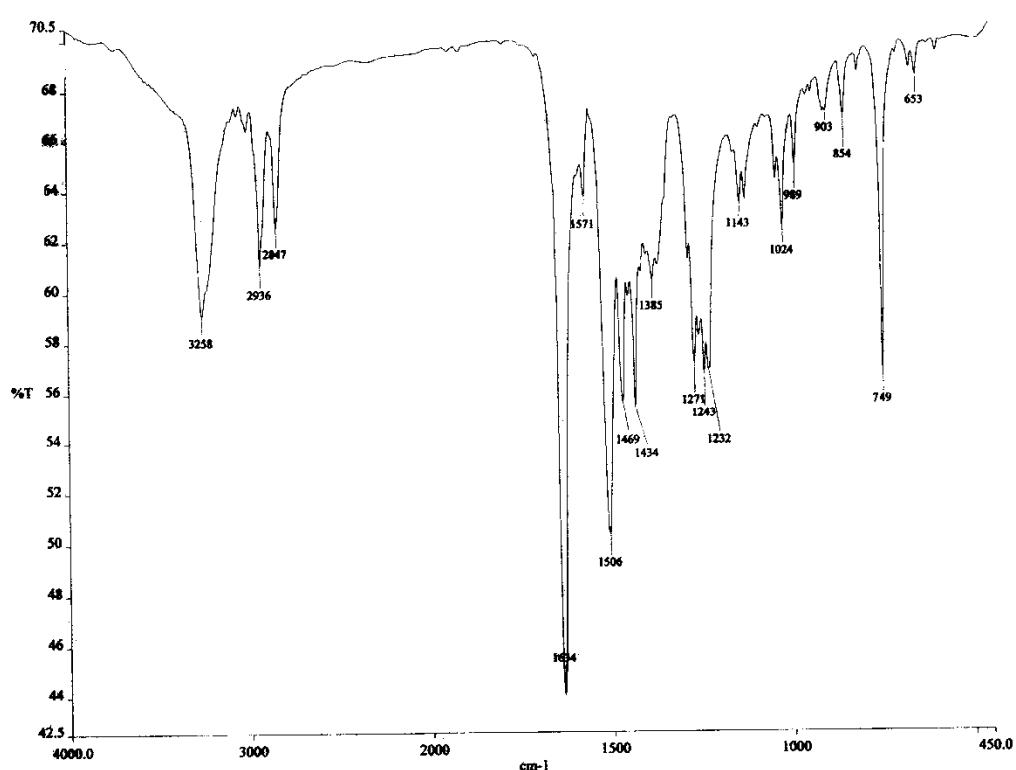
Piperidine-1-carboxylic acid (2-bromo-phenyl)-amide (15b):  $^1\text{H}$  NMR ( $\text{CDCl}_3$ , 400 MHz)



Piperidine-1-carboxylic acid (2-bromo-phenyl)-amide (15b):  $^{13}\text{C}$  NMR ( $\text{CDCl}_3$ , 100 MHz)



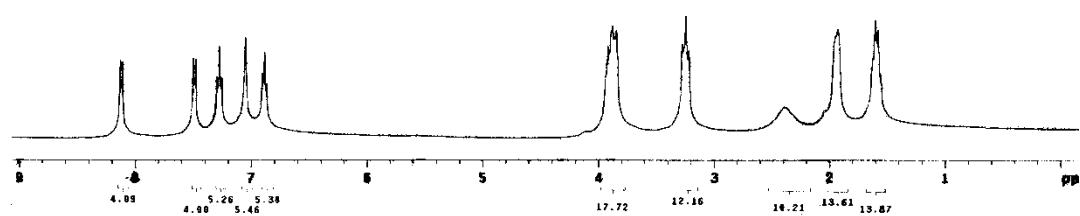
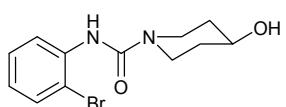
Piperidine-1-carboxylic acid (2-bromo-phenyl)-amide (15b): IR (KBr)



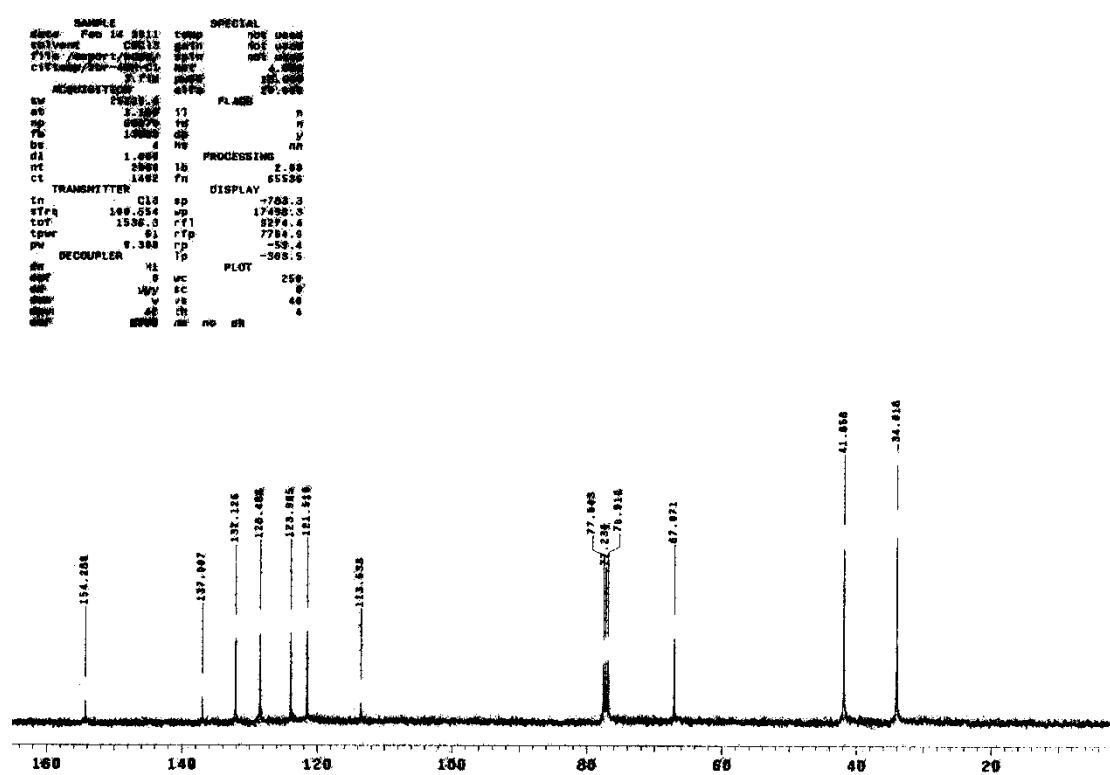
4-Hydroxy-piperidine-1-carboxylic acid (2-bromo-phenyl)-amide (15c):  $^1\text{H}$  NMR ( $\text{CDCl}_3$ , 400 MHz)

```
expt1 (ppm)
SAMPLE          SPCLN
date   Feb 14 2014 temp    RT
solvent  CDCl3   pres   1.00
file   exp      freq   400.00
ACQUISITION     hz    8.00
sw      4200.0   pw00  30.000
st      1.000   nfft  32768
sp      256.0   fwhm  10.000
rf0    not need   t1      a
bs      4      in      p
d1      1.000   de      90
rt      0.02   sv      16
ct      32   PROCESSING
TRANSMITTER    1b      0.10
th      31      n1      85538
tfrq   399.853   DISPLAY
t0f      362.0   sp      -111.7
tprf      57      w1      3738.3
pw      9.859   t1      791.7
DECOUPLER      C13   rfp
dc      0      ip      164.1
d0f      0      p0      -95.4
de      0      nc      150
dms      60      vc      150
dmw      10000   sc      100
dmt      10000   st      100
dt      0      cpm     0.00

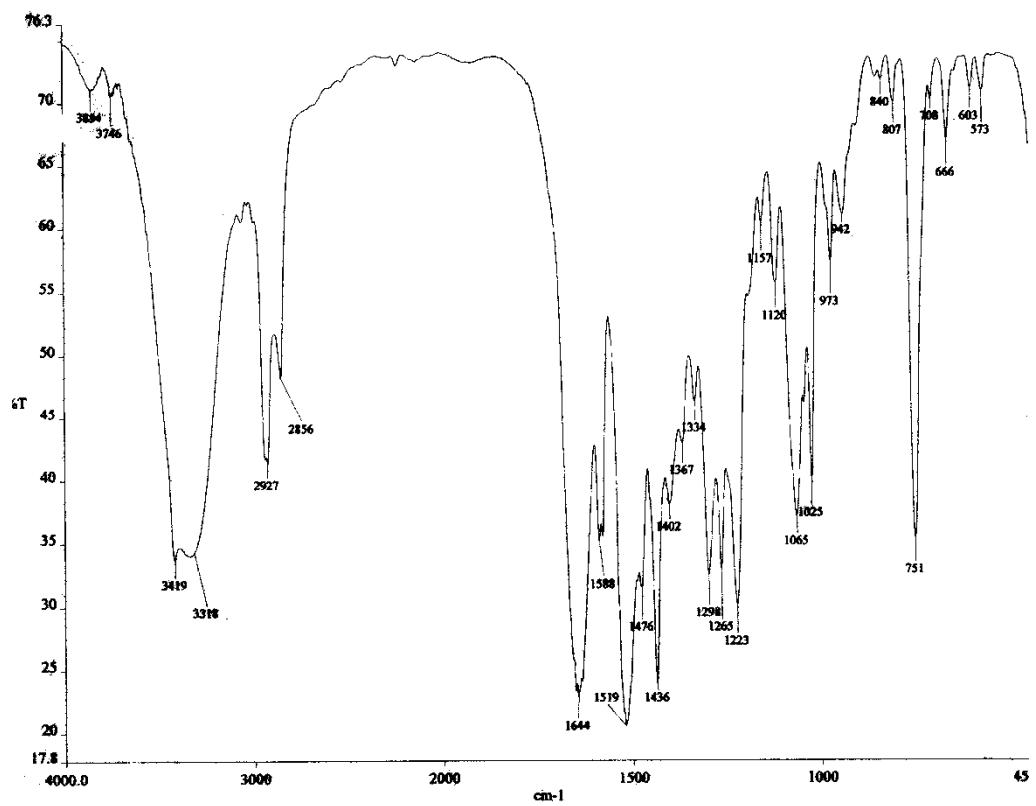
```



**4-Hydroxy-peridine-1-carboxylic acid (2-bromo-phenyl)-amide (15c):  $^{13}\text{C}$  NMR ( $\text{CDCl}_3$ , 100 MHz)**

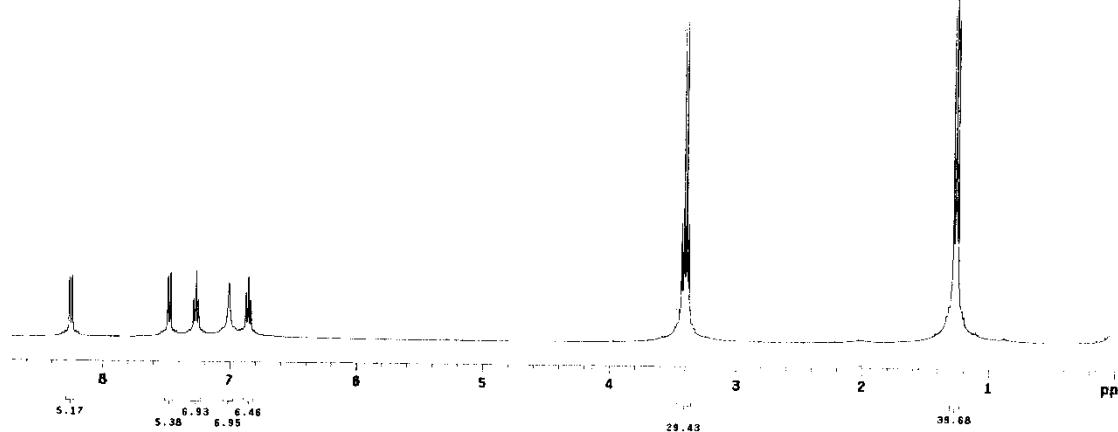
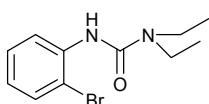


#### 4-Hydroxy-peridine-1-carboxylic acid (2-bromo-phenyl)-amide (15c): IR (KBr)



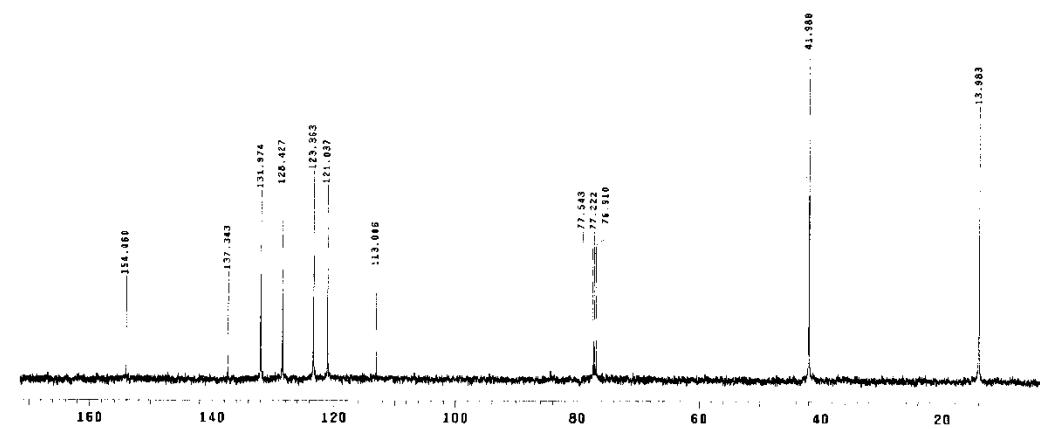
**3-(2-Bromo-phenyl)-1,1-diethyl-urea (15d):  $^1\text{H}$  NMR ( $\text{CDCl}_3$ , 400 MHz)**

```
expt1 *2pu1
SAMPLE          SPECIAL
date  Feb 14 2011 temp  not used
solvent   CDCl3  gain  not used
file    exp  spin  not used
ACQUISITION hct  0.008
sw     6000.0  pw90  19.788
at     1.998  alfa  29.008
np    25528  FLAGS
tb      not used 11  n
bs      4  t1n  n
d1    1.000  dp  y
nt     32  ns  nn
ct      32  PROCESSING
TRANSMITTER Tb  4.18
tn      HI  fn  65536
sfrq   399.63  DISPLAY
t0f    362.8  sp  -57.7
tpwr   9.650  w1  3565.9
pw     9.650  r1  790.0
DECOUPLER C13  rfp  0
d1f    9  1p  114.1
dof    9  1p  -92.5
da    nm  PLOT
dss   C  wc  250
dpwr   50  sc  0
dsf    15900  vs  145
th     22  th  22
ns  cdc  ph
```

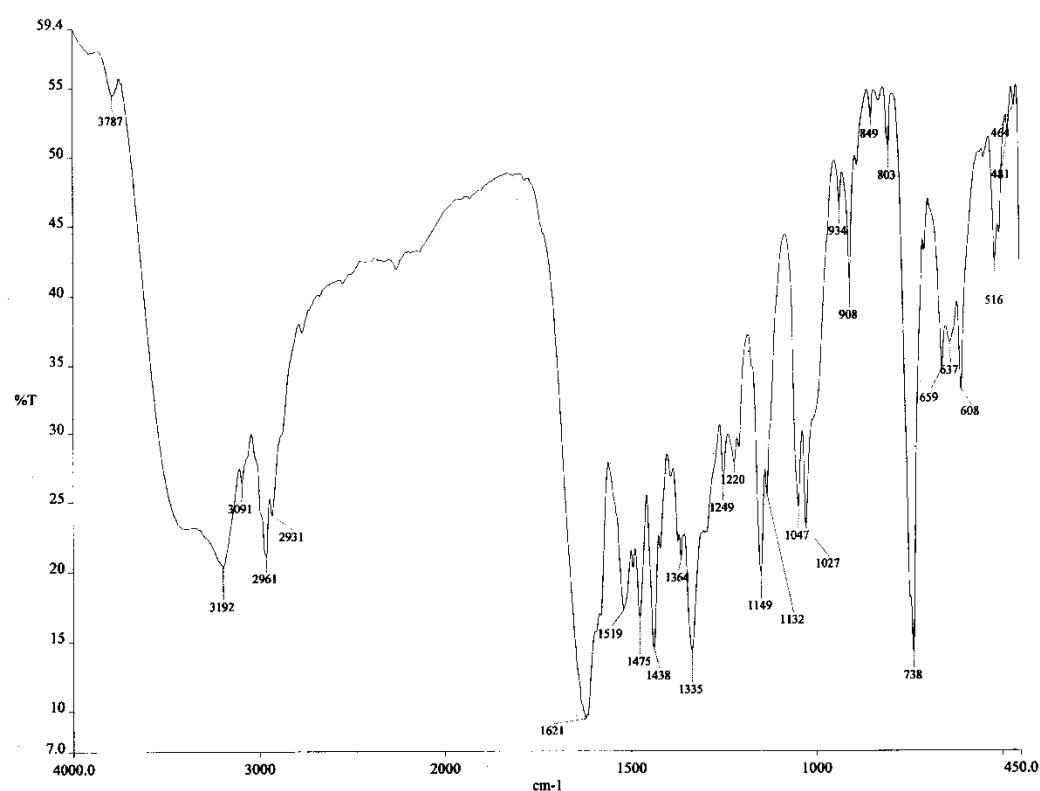


**3-(2-bromo-phenyl)-1,1-diethyl-urea (15d):  $^{13}\text{C}$  NMR (100 MHz,  $\text{CDCl}_3$ )**

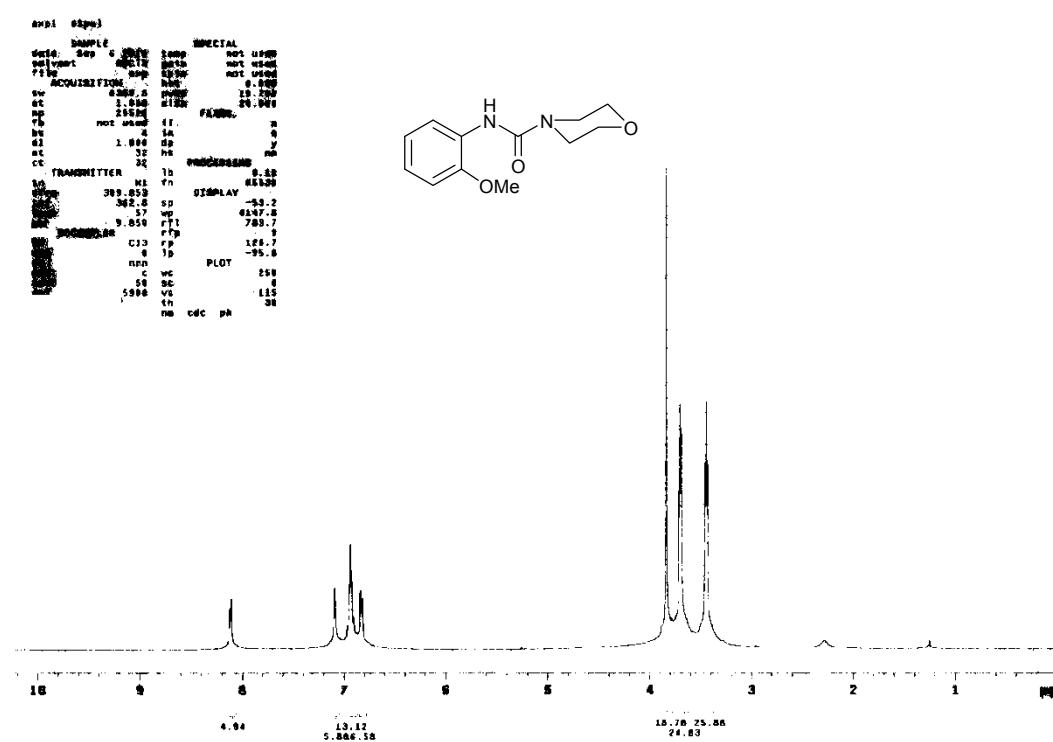
```
expt1 *2pu1
SAMPLE          SPECIAL
date  Feb 14 2011 temp  not used
solvent   CDCl3  gain  not used
file    exp  spin  not used
ACQUISITION hct  0.008
sw     2000.0  pw90  18.608
at     1.193  pw1c  23.008
np    60270  FLAGS
tb      13890  11  n
bs      4  t1n  n
d1    1.000  dp  y
nt     3010  ns  nn
ct      224  PROCESSING
TRANSMITTER Tb  2.68
tn      HI  fn  65536
sfrq   100.531  DISPLAY
t0f    1536.3  sp  -192.9
tpwr   9.380  w1  17432.4
pw     9.380  r1f  9278.2
DECOUPLER C13  rfp  0
d1f    9  1p  114.3
dof    9  1p  -345.9
da    vVY  PLOT
dss   C  wc  250
dpwr   42  sc  0
dsf    6890  vs  58
th     4  th  4
ns  no  ph
```



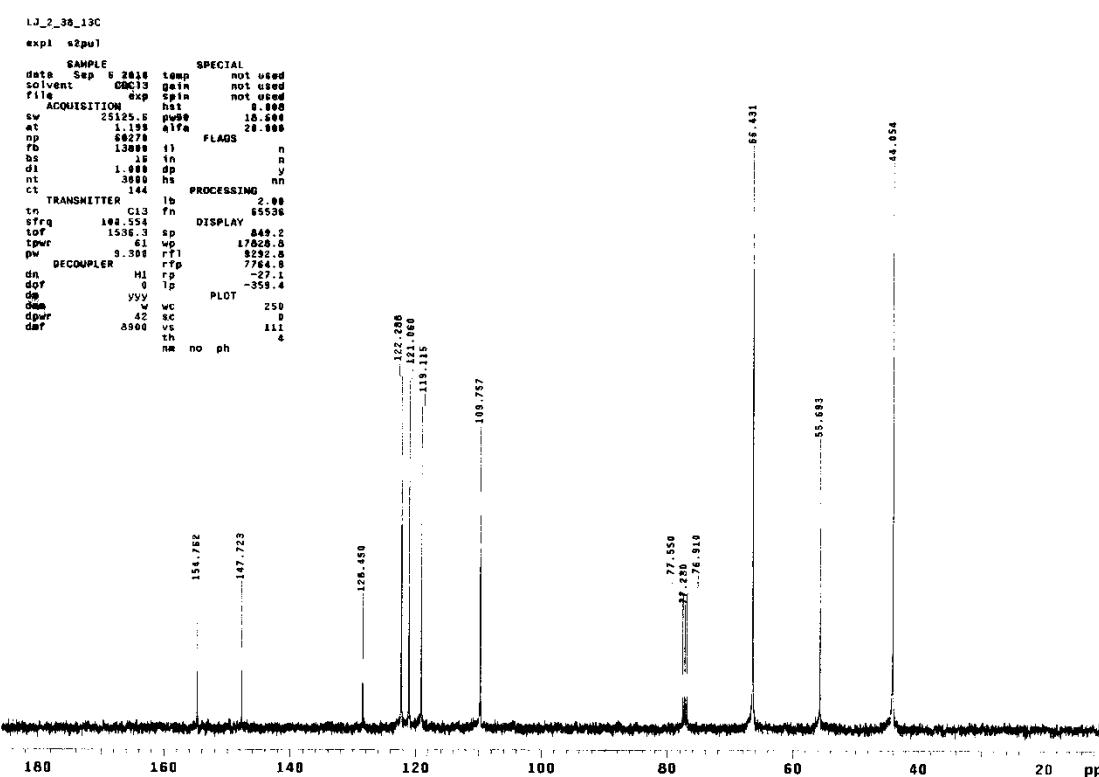
**3-(2-Bromo-phenyl)-1,1-diethyl-urea (15d): IR (KBr)**



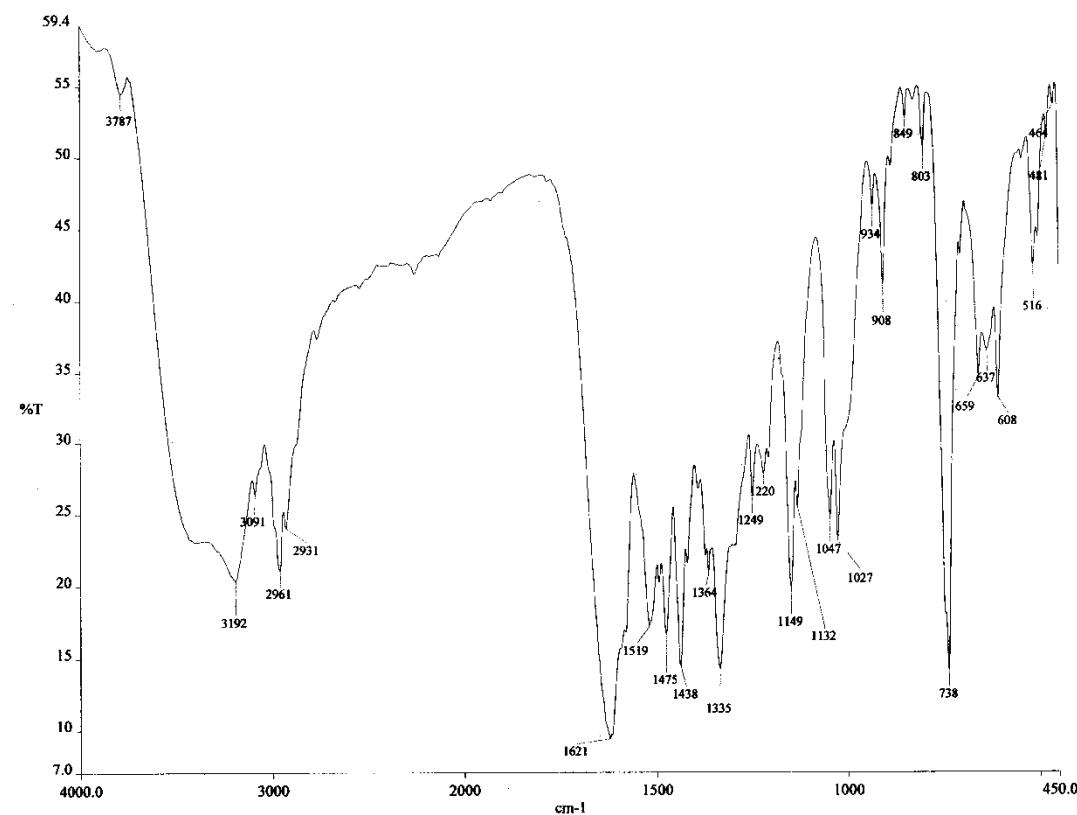
**Morpholine-4-carboxylic acid (2-methoxy-phenyl)-amide (16a) <sup>1</sup>H NMR (CDCl<sub>3</sub>, 400 MHz)**



**Morpholine-4-carboxylic acid (2-methoxy-phenyl)-amide (16a):  $^{13}\text{C}$  NMR ( $\text{CDCl}_3$ , 100 MHz)**

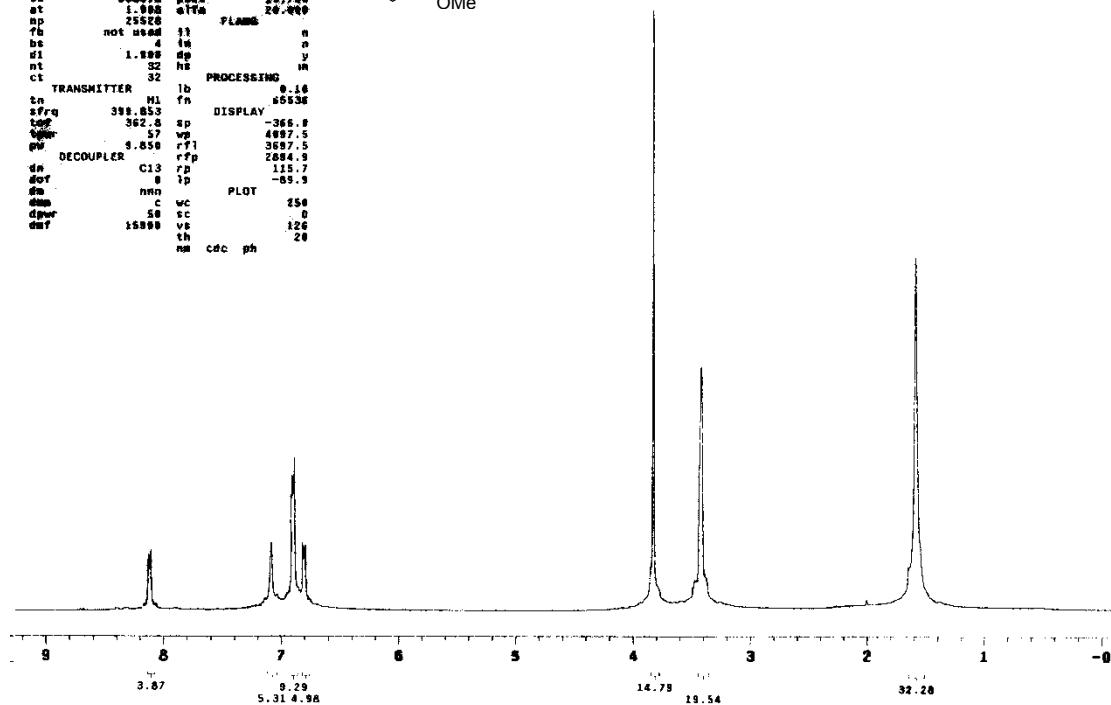
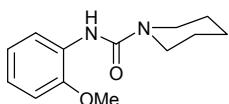


**Morpholine-4-carboxylic acid (2-methoxy-phenyl)-amide (16a): IR (KBr)**



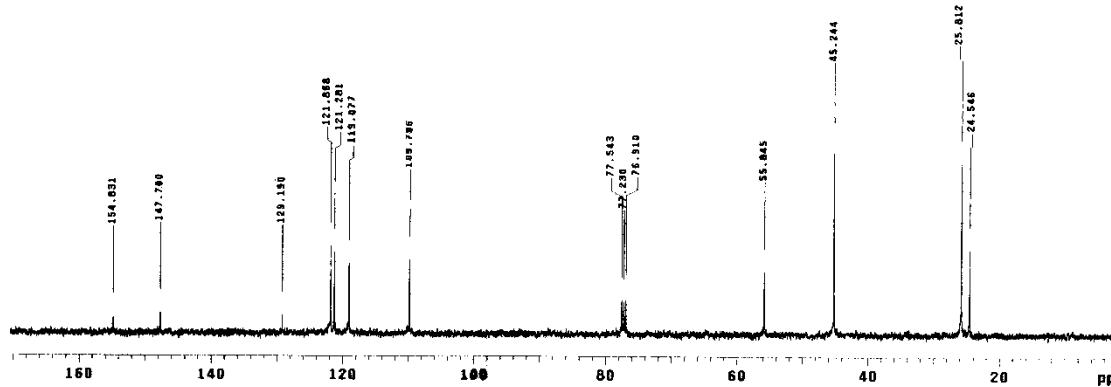
Piperidine-1-carboxylic acid (2-methoxy-phenyl)-amide (16b):  $^1\text{H}$  NMR ( $\text{CDCl}_3$ , 400 MHz)

exp1 s2pu1  
SAMPLE: 16b  
date: Feb 14 2011 temp: not used  
solvent: CDCl<sub>3</sub> gain: not used  
file: not used pw1: not used  
ACQUISITION: not used tdec: 1.468  
sw: 4000.0 Hz pdec: 16.748  
et: 1.998 sIT: 26.499  
sp: 255280 PLANS: 1  
fb: not used 11 n  
bs: 4 in  
dl: 1.000 np: y  
nt: 32 hs IR  
ct: 32 PROCESSING:  
TRANSMITTER: 1b 0.16  
tn: H1 fb: 65536  
sfrq: 398.853 DISPLAY: -365.8  
tgr: 362.8 sp: -365.8  
tgrw: 57 vp: 4897.5  
pw: 9.859 r1: 3697.5  
DECOUPLER: rfp 2884.9  
dn: C13 rp: 115.7  
dof: 4 ip: -69.3  
de: mnm PLOT: 256  
dme: c wc: 256  
dpw: 50 sc: 0  
dmt: 15898 vs: 126  
dt: th: 20  
ns: cdc ph:

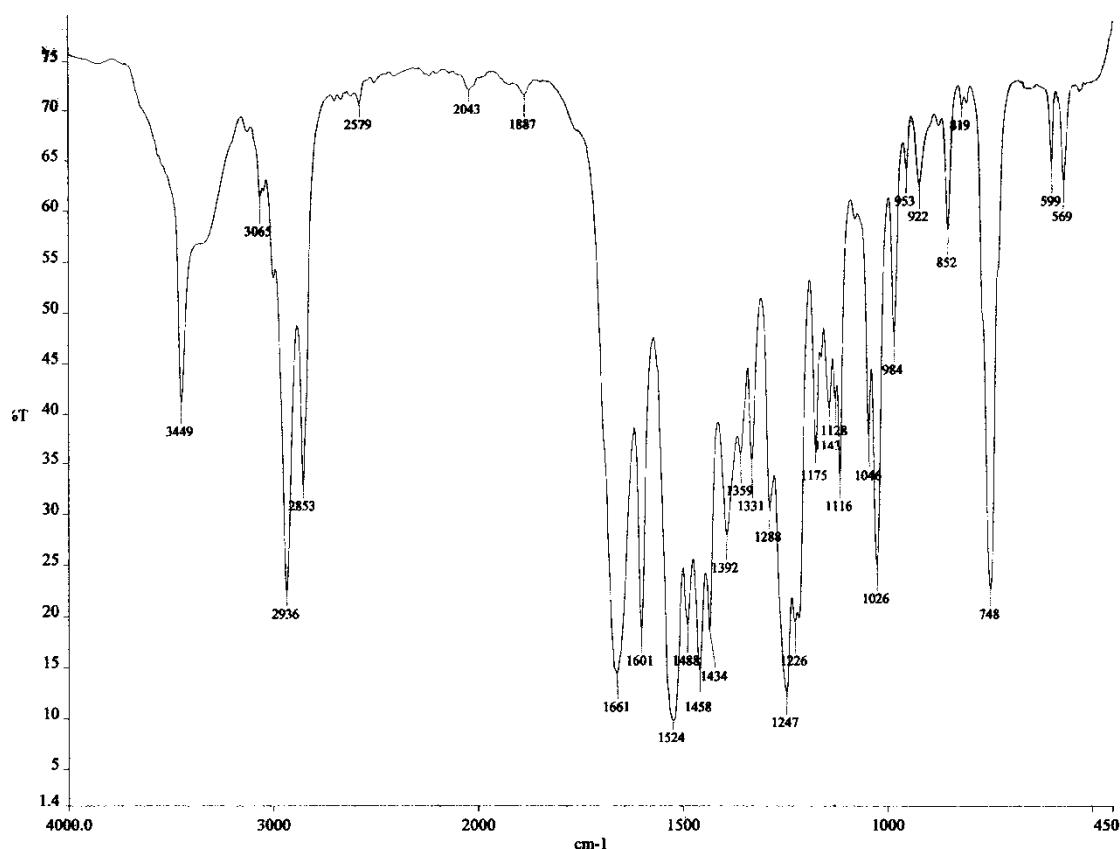


Piperidine-1-carboxylic acid (2-methoxy-phenyl)-amide (16b):  $^{13}\text{C}$  NMR ( $\text{CDCl}_3$ , 100 MHz)

20Me-piper-13C  
exp1 s2pu1  
SAMPLE: 16b  
date: Feb 14 2011 temp: AL not used  
solvent: CDCl<sub>3</sub> gain: not used  
file: not used pw1: not used  
ACQUISITION: not used tdec: 1.468  
sw: 25128.6 Hz pdec: 16.748  
et: 1.193 sIT: 26.499  
sp: 60270 PLANS: 1  
fb: 136000 11 n  
bs: 4 in  
dl: 1.000 dp: v  
nt: 2880 ht: nn  
ct: 210 PROCESSING:  
TRANSMITTER: 1b 2.48  
tn: C13 fb: 65536  
sfrq: 100.554 DISPLAY: 4.4  
tgr: 1536.1 sp: 4.4  
tgrw: 51 vp: 17235.0  
pw: 8.308 r1: 5276.2  
DECOUPLER: rfp 7764.9  
dn: H1 rp: -44.8  
dof: 4 ip: -323.6  
de: vvv PLOT: 256  
dme: c wc: 256  
dpw: 42 sc: 0  
dmt: 8898 vs: 48  
dt: th: 4  
ns: no ph:

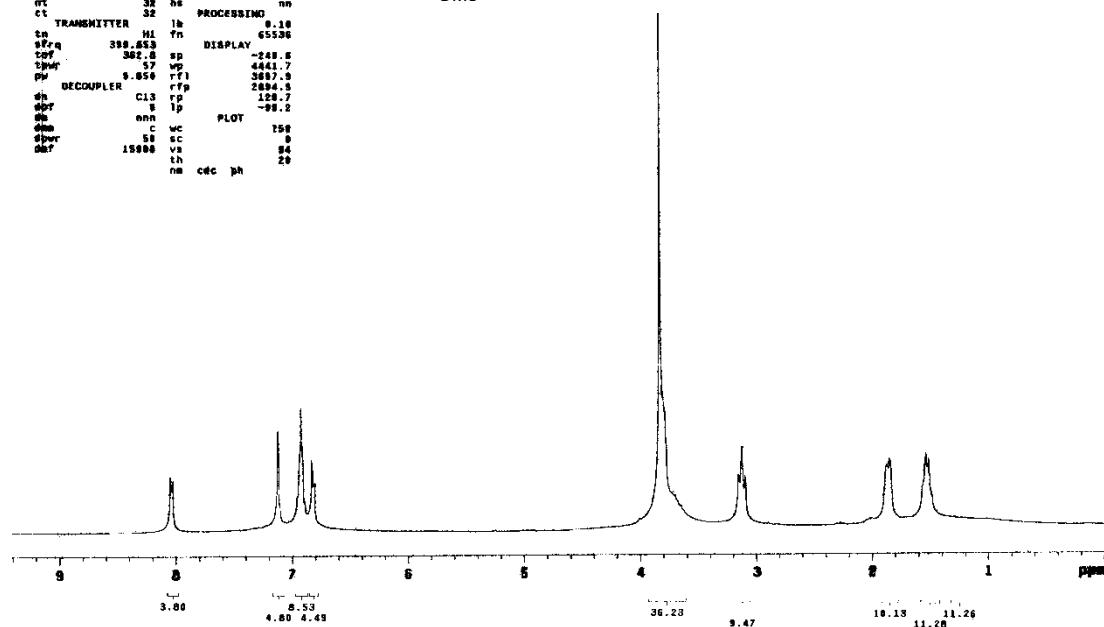
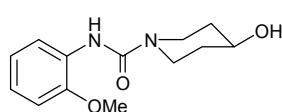


Piperidine-1-carboxylic acid (2-methoxy-phenyl)-amide (16b): IR (KBr)

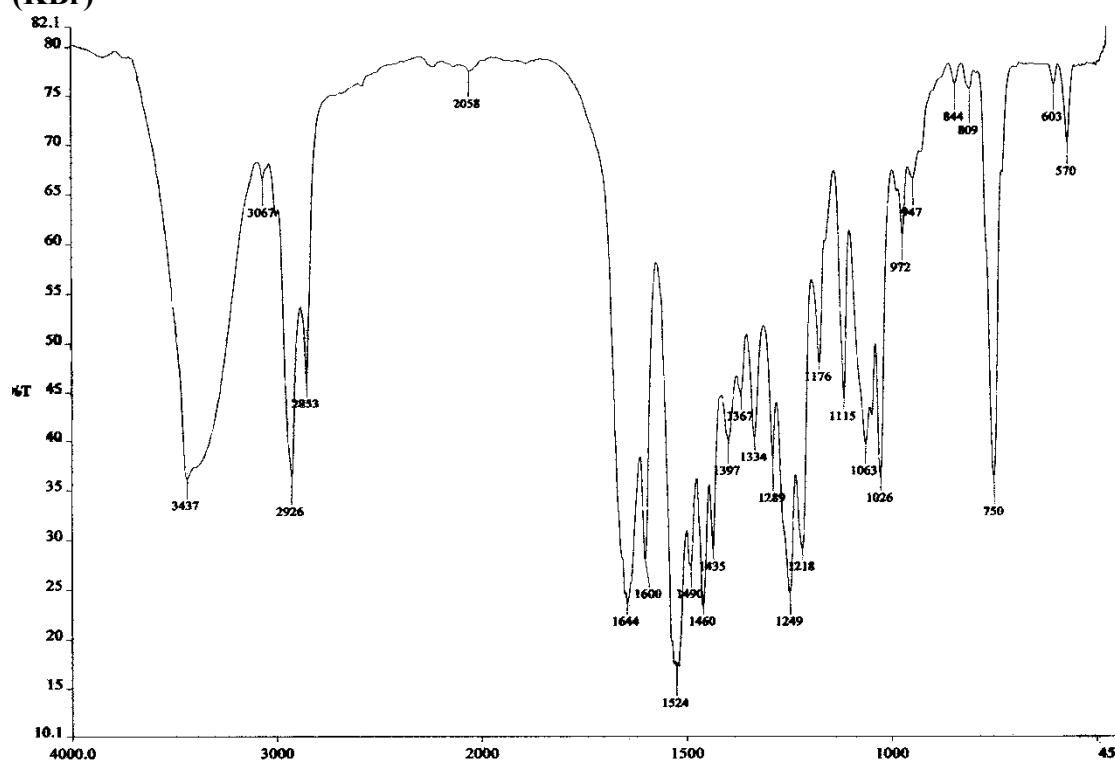
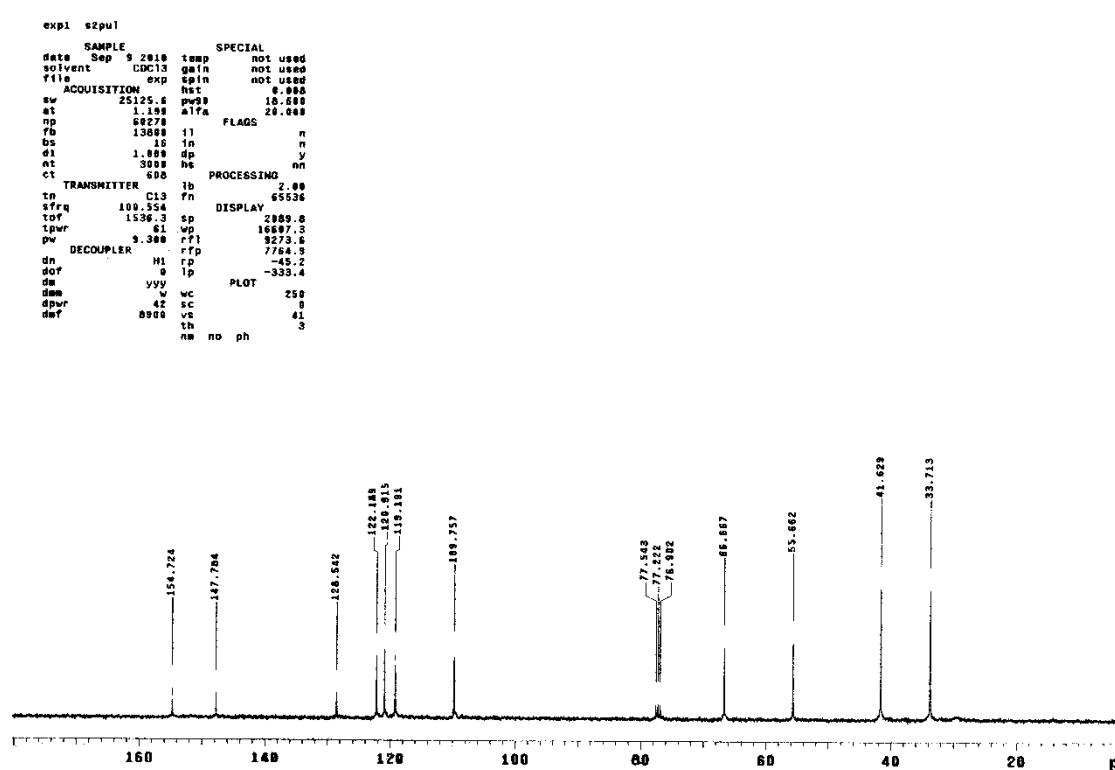


4-Hydroxy-piperidine-1-carboxylic acid (2-methoxy-phenyl)-amide (16c): <sup>1</sup>H NMR ( $\text{CDCl}_3$ , 400 MHz)

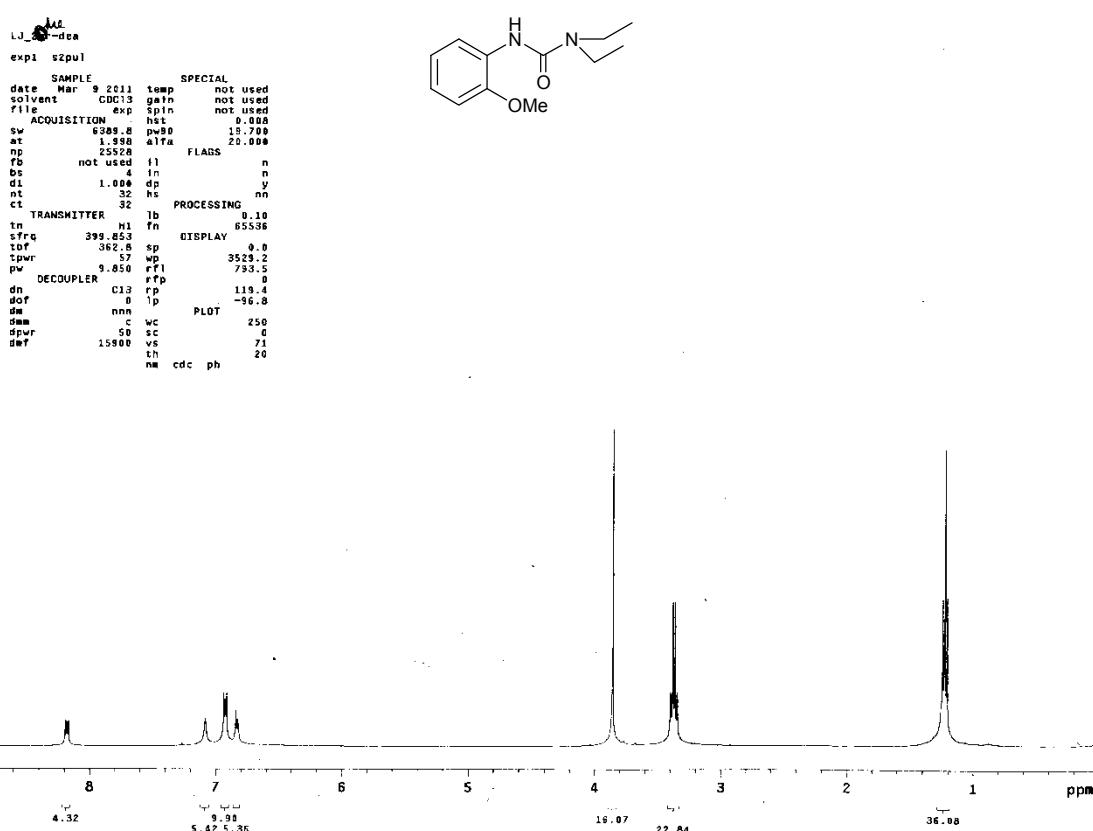
```
expt 52pm1
SAMPLE          SPECIAL
date Oct 2 2010 temp    not used
solvent   CDCl3 gain   not used
file      exp    not used
        nmt   not used
        nmt   not used
sw       8369.8 psize 19.798
at      1.000 a1f8 28.888
rt      25528   FLAOS
rf      not used fl  n
dd      4      in  n
d1      1.000 dp  y
rt      32      he  nn
ct      32      PROCESSING nn
        TRANSMITTER 1b  0.10
        HI  fm  65536
        399.650  DISPLAY 65536
        399.650  sp  -248.5
        361.8  sp  -248.5
        57  wp  4441.7
        9.850 rrf1 3887.9
        DECOUPLER  C13  rf  2888.5
        13  rf  128.7
        dpr  1p  -98.2
        dd  ann  PLOT
        dm  C  wc  750
        dver  S  sc  9
        dmt  15000  vs  94
        th  cdc  ph  29
        nm  cdc  ph
```



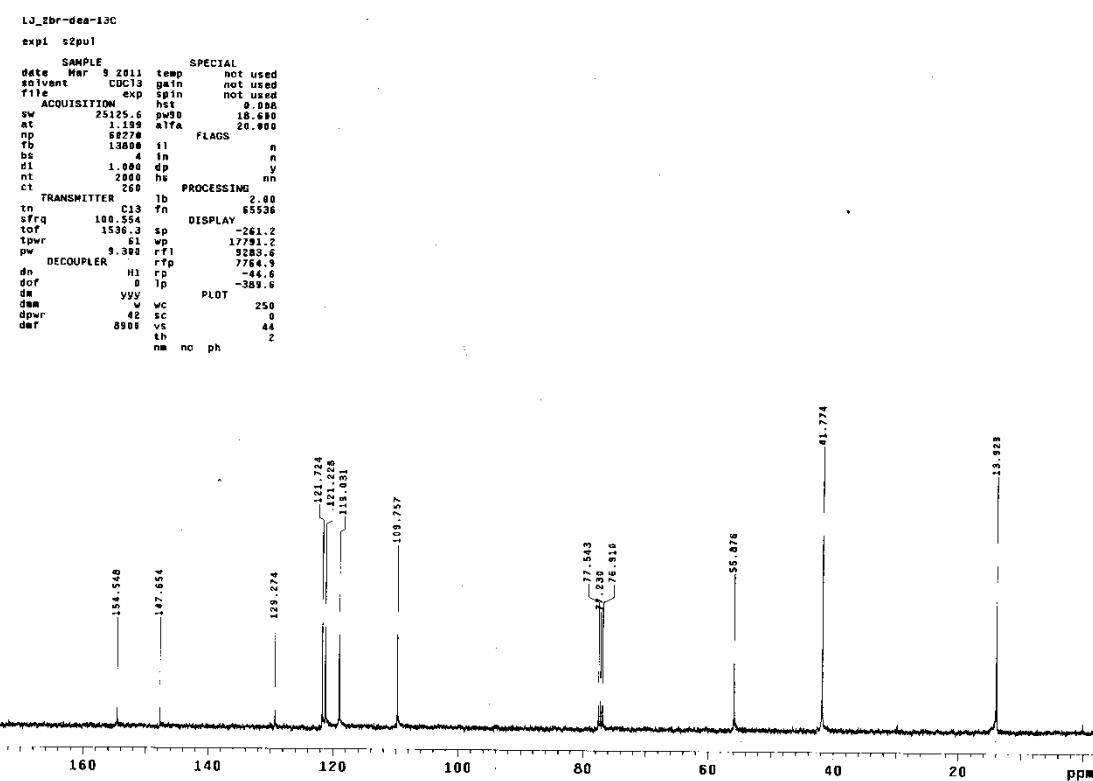
**4-Hydroxy-piperidine-1-carboxylic acid (2-methoxy-phenyl)-amide (16c):  $^{13}\text{C}$  NMR ( $\text{CDCl}_3$ , 100 MHz)**



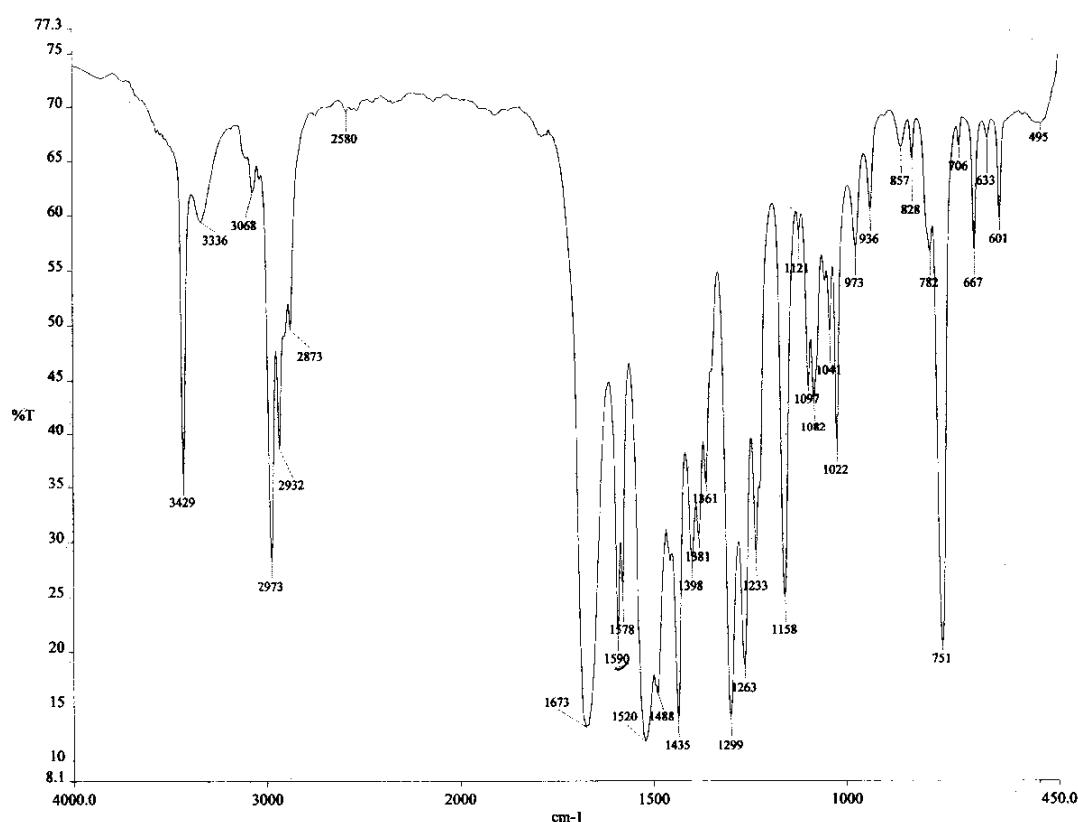
**1,1-Diethyl-3-(2-methoxy-phenyl)-urea (16d):  $^1\text{H}$  NMR ( $\text{CDCl}_3$ , 400 MHz)**



**1,1-Diisopropyl-3-(2-methoxy-phenyl)-urea (16e):  $^{13}\text{C}$  NMR ( $\text{CDCl}_3$ , 100 MHz)**

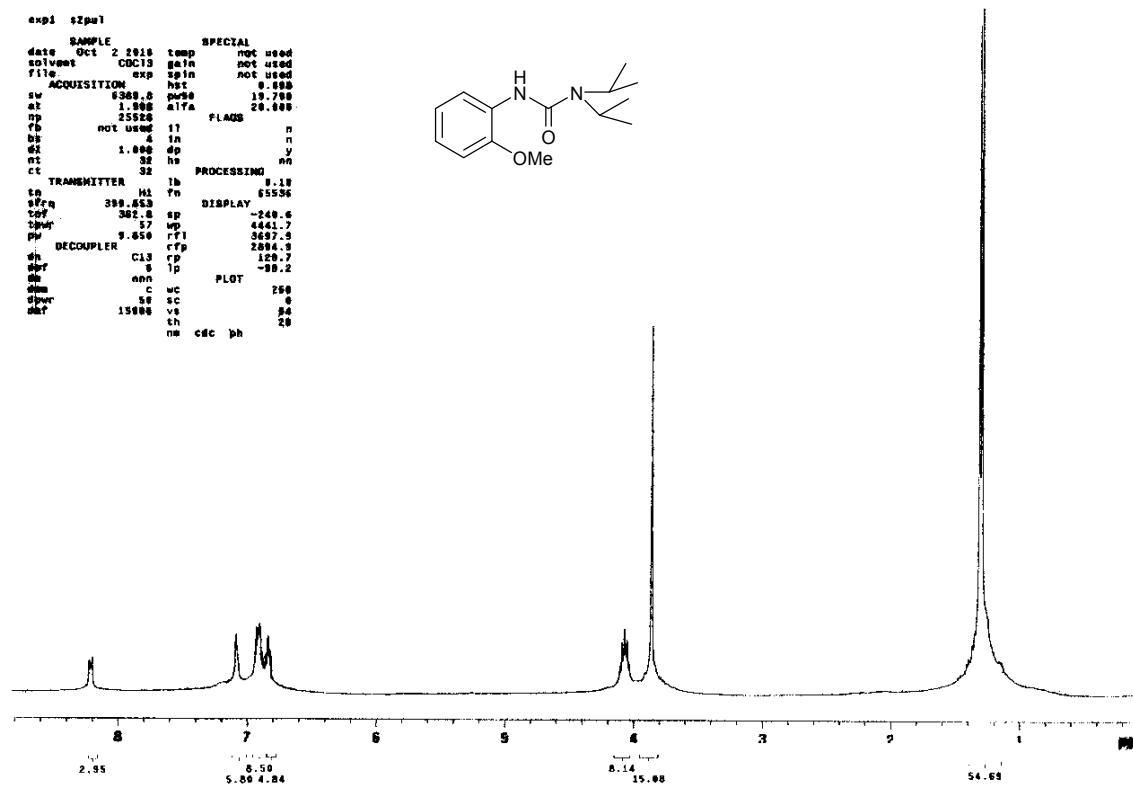
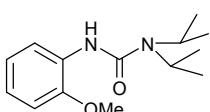


**1,1-Diethyl-3-(2-methoxy-phenyl)-urea (16d): IR (KBr)**

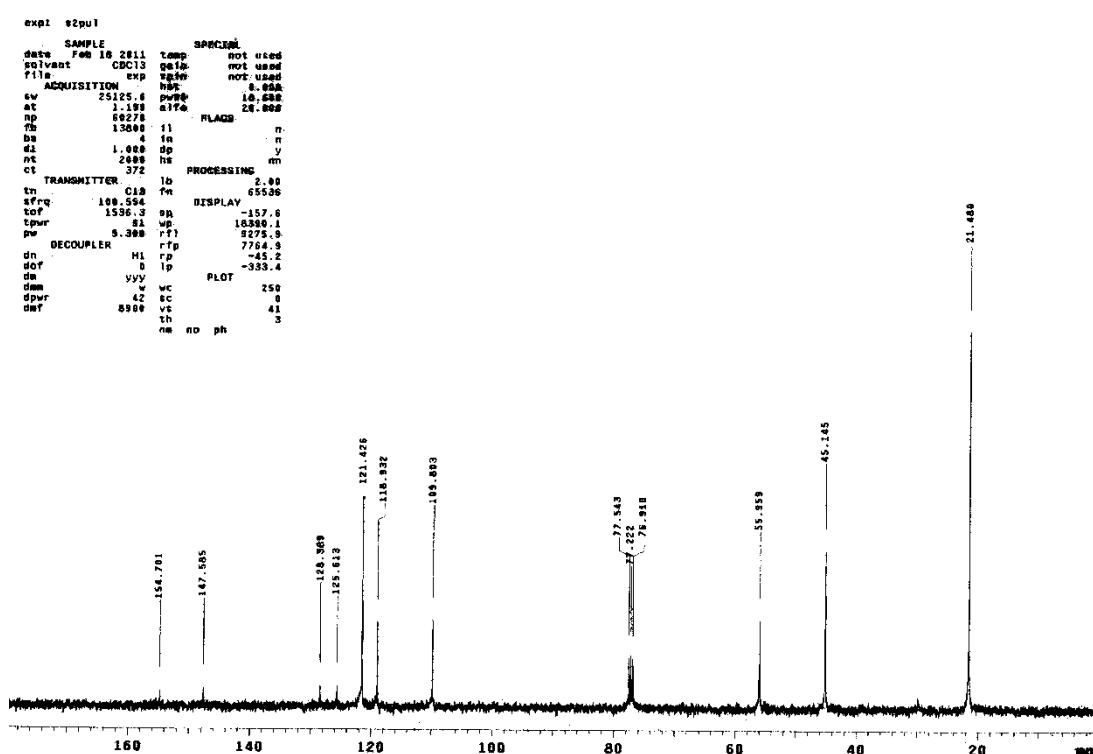


**1,1-Diisopropyl-3-(2-methoxy-phenyl)-urea (16e): <sup>1</sup>H NMR (CDCl<sub>3</sub>, 400 MHz)**

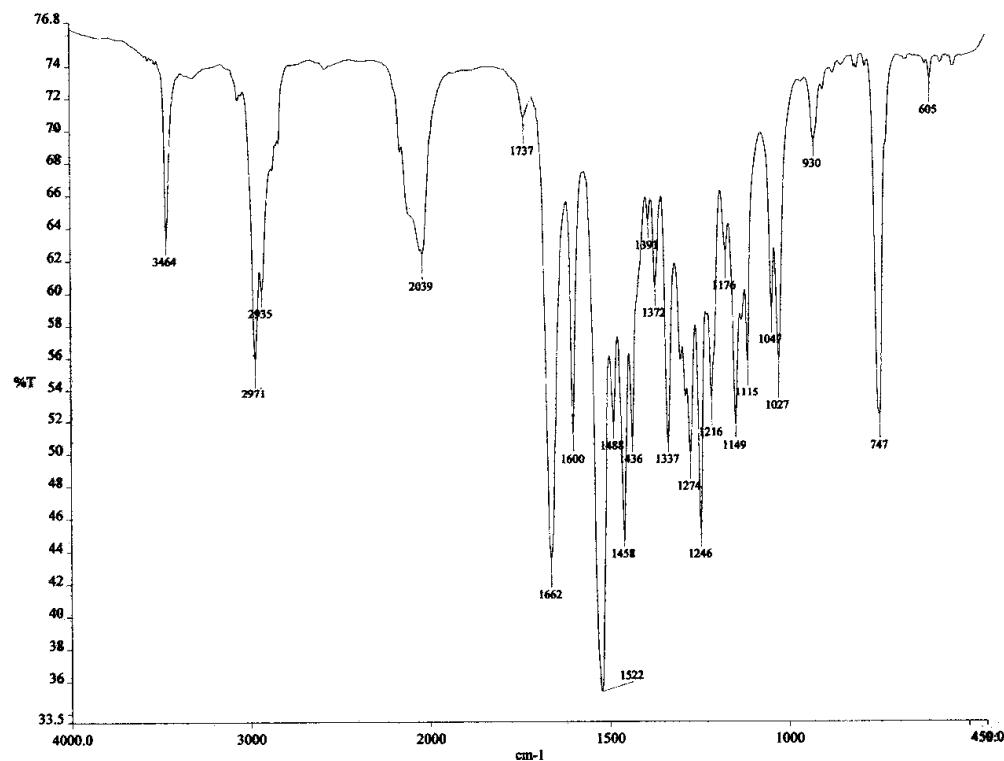
```
exp1 #2pm1
SAMPLE          SPECIAL
date Oct 2 2011 temp not used
solvent   CDCl3 spin not used
file      exp spin not used
ACQUISITION hst    spin 0.100
sv       6300.0 pw06 19.700
et       1.000   a17a 20.000
sp       1.000   flags
fb       not used 11   n
bb       4   in   n
de      1.000   dp   y
rt       10   in   an
ct       32   PROCESSING
TRANSMITTER 1b   8.10
tr       H1   tn   65536
swfq    399.450   DISPLAY 65536
tcf     300.8   sp   -240.6
tphf    57   mp   4441.7
pw     9.650   rrf  3697.9
DECOUPLER C13   rrf  2881.9
dp     1.0   tp   129.7
drf    1.0   tp   -89.2
dd     ann   wc   260
dppr   56   sc   0
dchf   15000  vs   54
dchf   15000  th   20
nm   cdc  ph
```



**1,1-Diethyl-3-(2-methoxy-phenyl)-urea (16d):  $^{13}\text{C}$  NMR ( $\text{CDCl}_3$ , 100 MHz)**

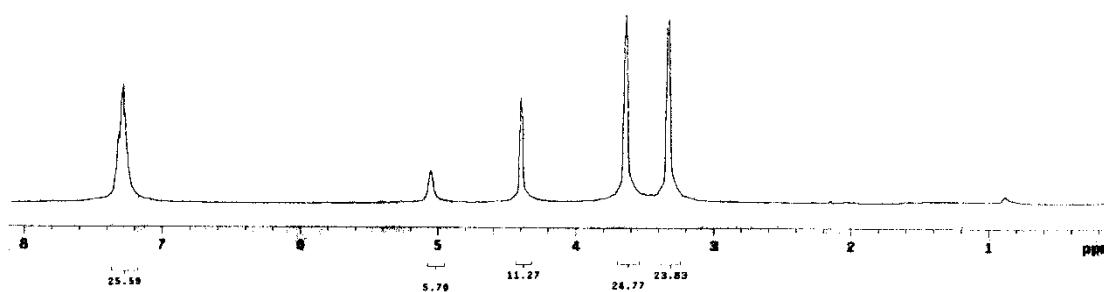
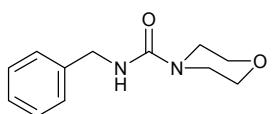


**1,1-Diisopropyl-3-(2-methoxy-phenyl)-urea (16e): IR (KBr)**



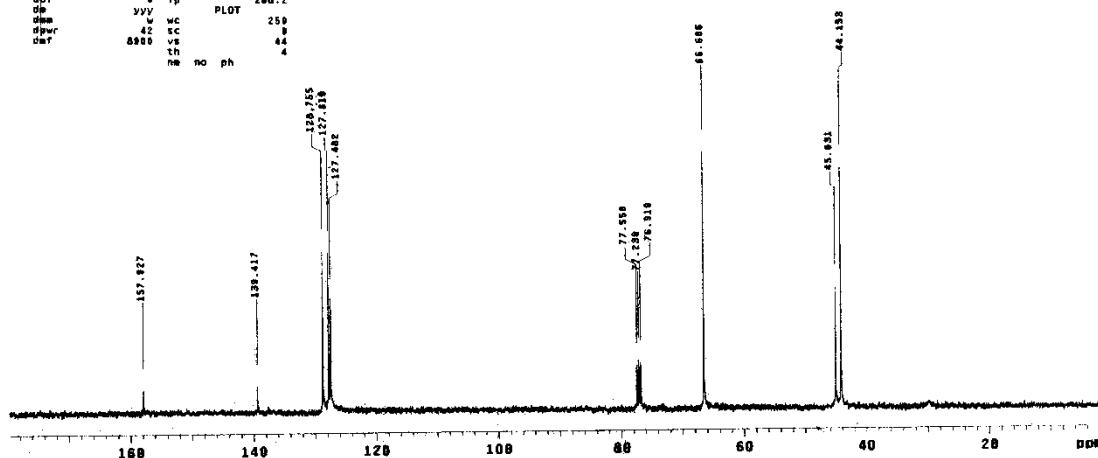
Morpholine-4-carboxylic acid benzylamide (17a):  $^1\text{H}$  NMR ( $\text{CDCl}_3$ , 400 MHz)

```
exptl s2pu1
SAMPLE           SPECIAL
date  Aug 16 2010 temp  not used
solvent   CDCl3 gain  not used
file    exp spin  not used
ACQUISITION hst pw90  90.786
sw   6389.8  pw90  127.786
at   1.998  alfa  29.001
np   25520  FLAGS
rfb  not used  l1  n
bs   1.000  in  n
di   1.000  dp  y
nt   32  hs  nn
ct   32  PROCESSING
TRANSMITTER 1b  8.18
tn   H1  fn  $5536
sfrq  399.853  DISPLAY
t0f   362.8  sp  -28.4
tpwr  57  wp  3837.1
pw   9.850  rfp  782.8
DECOUPLER  rfp  9
dn   C13  rfp  117.8
dof  8  ip  -79.3
dm   0nn  PLOT
dme   c  wc  259
dpwr  59  sc  9
dmt   15900  vs  47
th   20
RM  cdc  ph
```

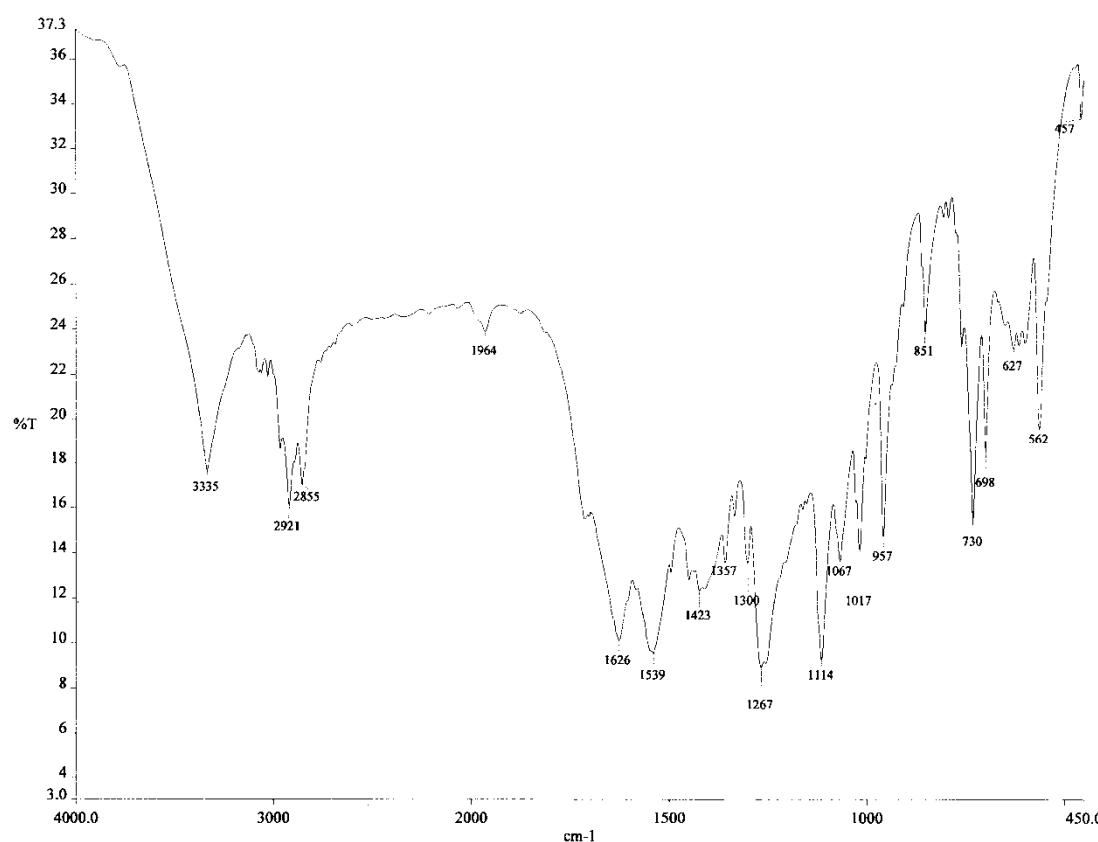


Morpholine-4-carboxylic acid benzylamide (17a):  $^{13}\text{C}$  NMR ( $\text{CDCl}_3$ , 100 MHz)

```
exptl std13c
SAMPLE           SPECIAL
date  Aug 16 2010 temp  not used
solvent   CDCl3 gain  not used
file    exp spin  not used
ACQUISITION hst pw90  10.600
sw   25000.0  pw90  20.000
at   1.998  alfa  29.000
np   34880  FLAGS
fb   13880  l1  n
bs   4  in  n
di   1  dp  y
nt   34880  HS  nn
ct   410  PROCESSING
TRANSMITTER 1b  1.68
tn   C13  fn  not used
sfrq  109.332  DISPLAY
t0f   61  sp  233.7
tpwr  8.667  rfp  19579.1
pw   9.667  rfp  19771.1
DECOUPLER  rfp  7764.9
dn   H1  rfp  -88.4
dpf  8  ip  -266.2
dm   39V  PLOT
dme   v  wc  259
dpwr  42  sc  9
dmt   8980  vs  44
th   4
RM  no  ph
```

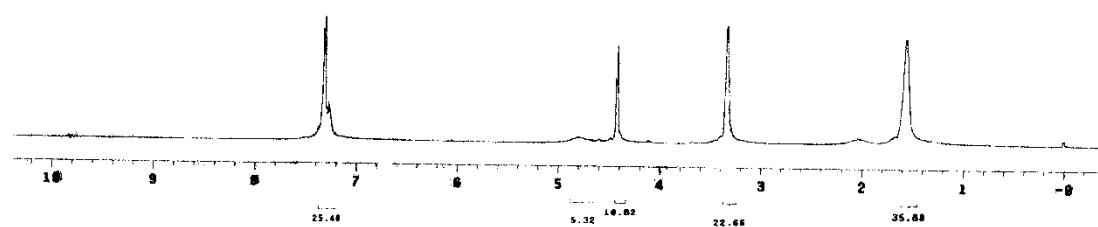
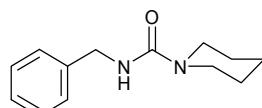


**Morpholine-4-carboxylic acid benzylamide (17a): IR (KBr)**

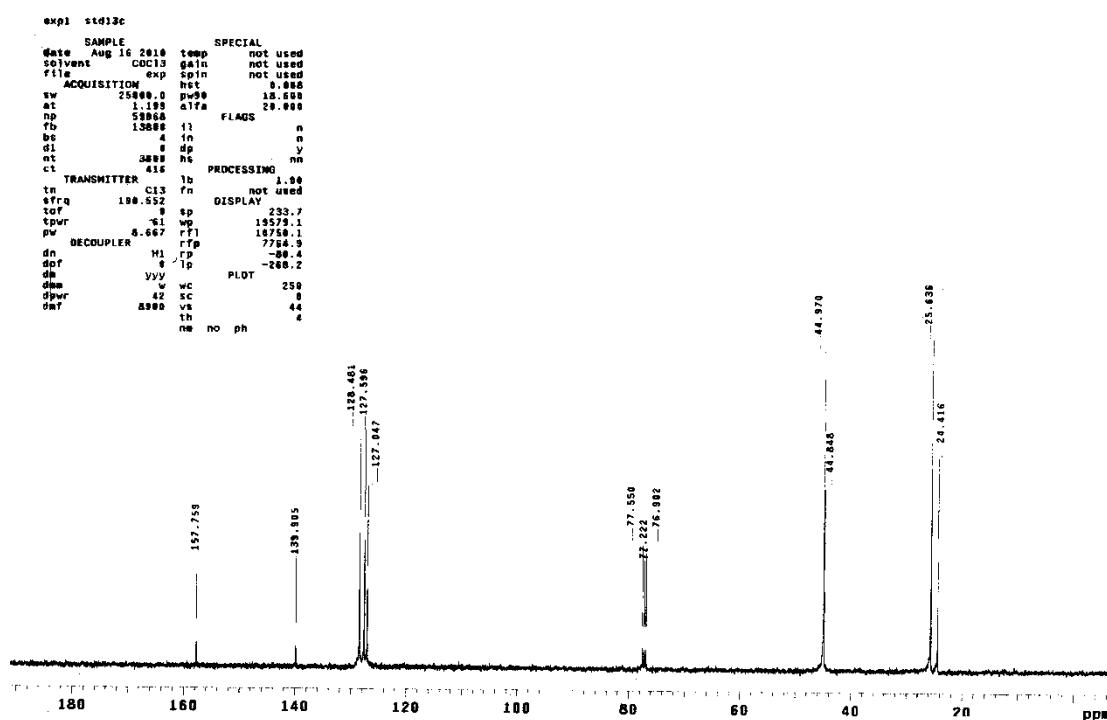


**Piperidine-4-carboxylic acid benzylamide (17b): <sup>1</sup>H NMR (CDCl<sub>3</sub>, 400 MHz)**

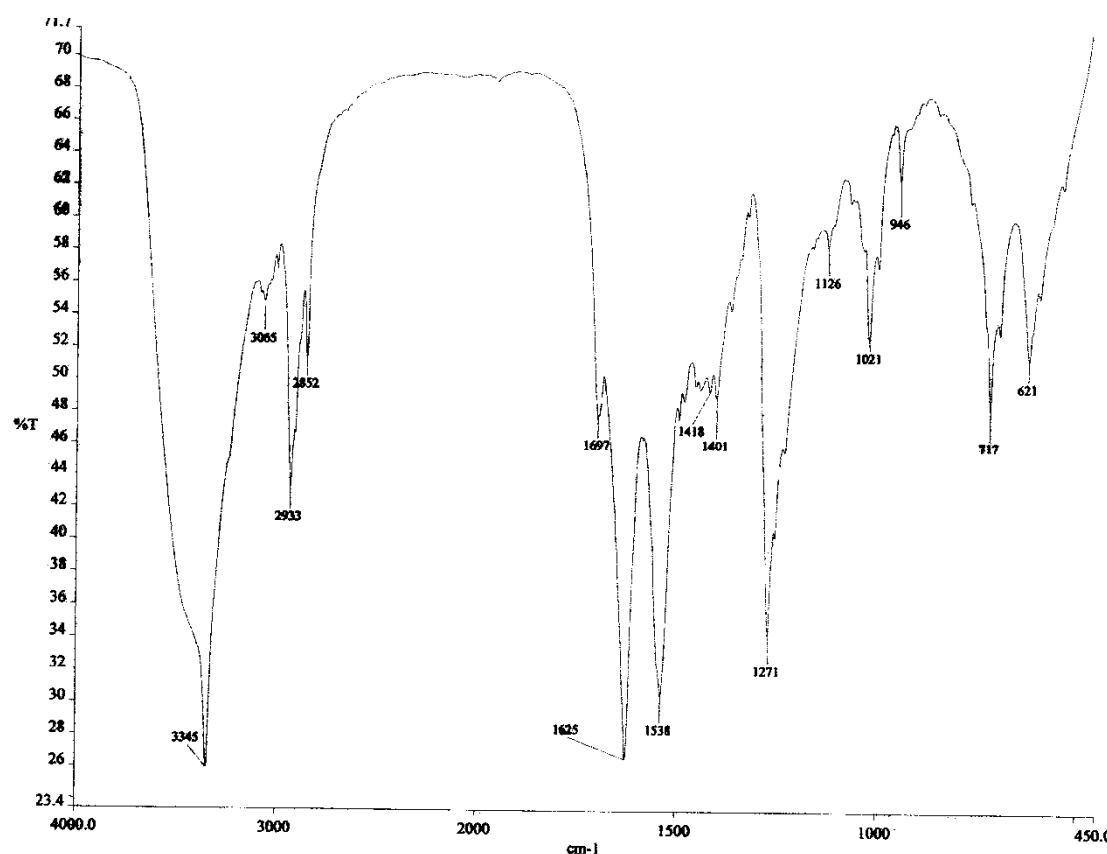
```
encl :2pu1
SAMPLE          SPECIAL
date Aug 11 2010 temp not used
solvent CDCl3  gain not used
time exp  spin not used
ACQUISITION nst 9.000
sw 6389.6   freq 117.988
at 1.998   w1w 20.099
np 25528   flags
tr not used 1)
bs 4      in    n
di 1.048   dp    y
nt 32     ns    nn
ct 32     PROCESSING
TH TRANSMITTER 1b   8.19
TH H1 FID 65536
SFRE 359.053  DISPLAY
TCF 362.569  SP  -28.9
TPW 5.57    WP  3857.1
PW 9.050   RF1  792.5
DECOPPLER C13  RF2  0
D1H C13  RP  117.9
D1F 8      IP  -79.3
D2H MNN  IP  PLOT
D2F C  VC  258
DPWR 50  SC  9
D1F 15900  VR  67
D2F TH  CD  20
RM cdc ph
```



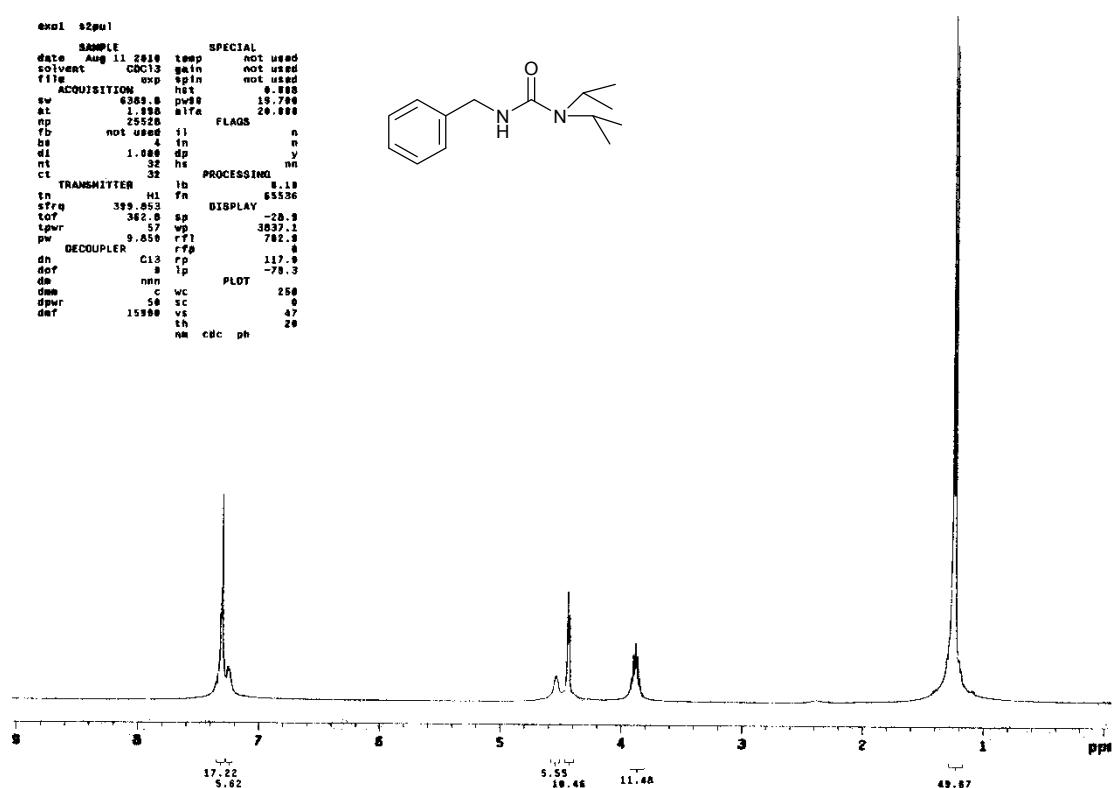
Piperidine-4-carboxylic acid benzylamide (17b):  $^{13}\text{C}$  NMR ( $\text{CDCl}_3$ , 100 MHz)



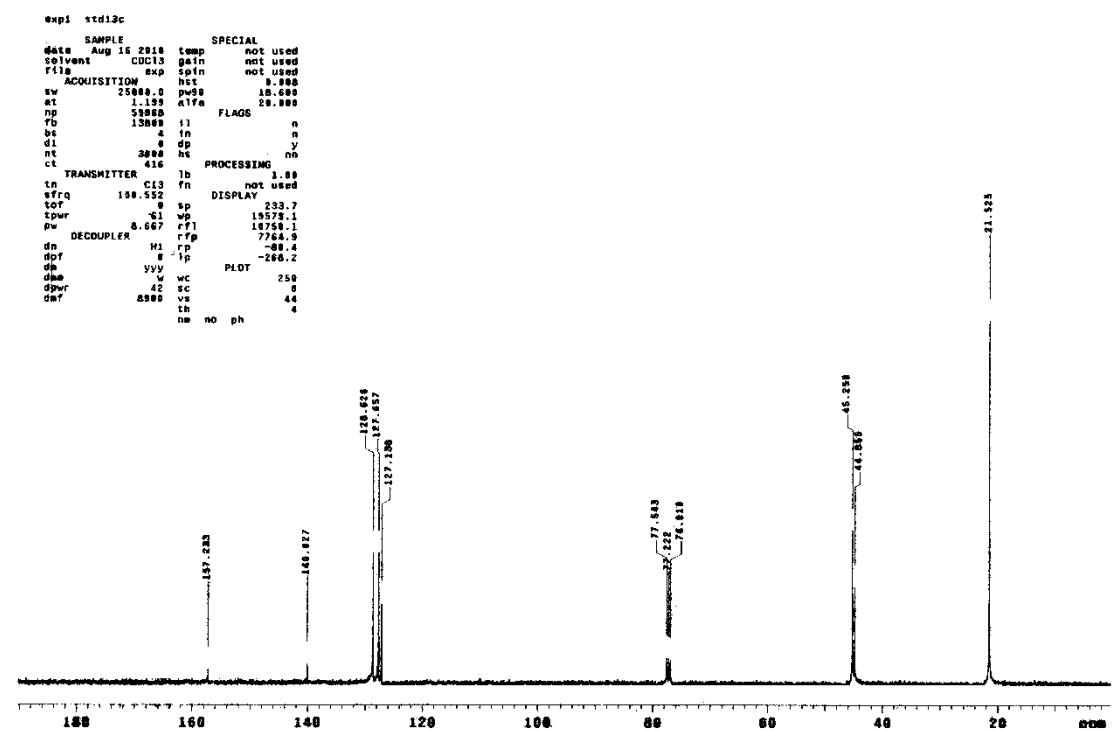
Piperidine-4-carboxylic acid benzylamide (17b): IR (KBr)



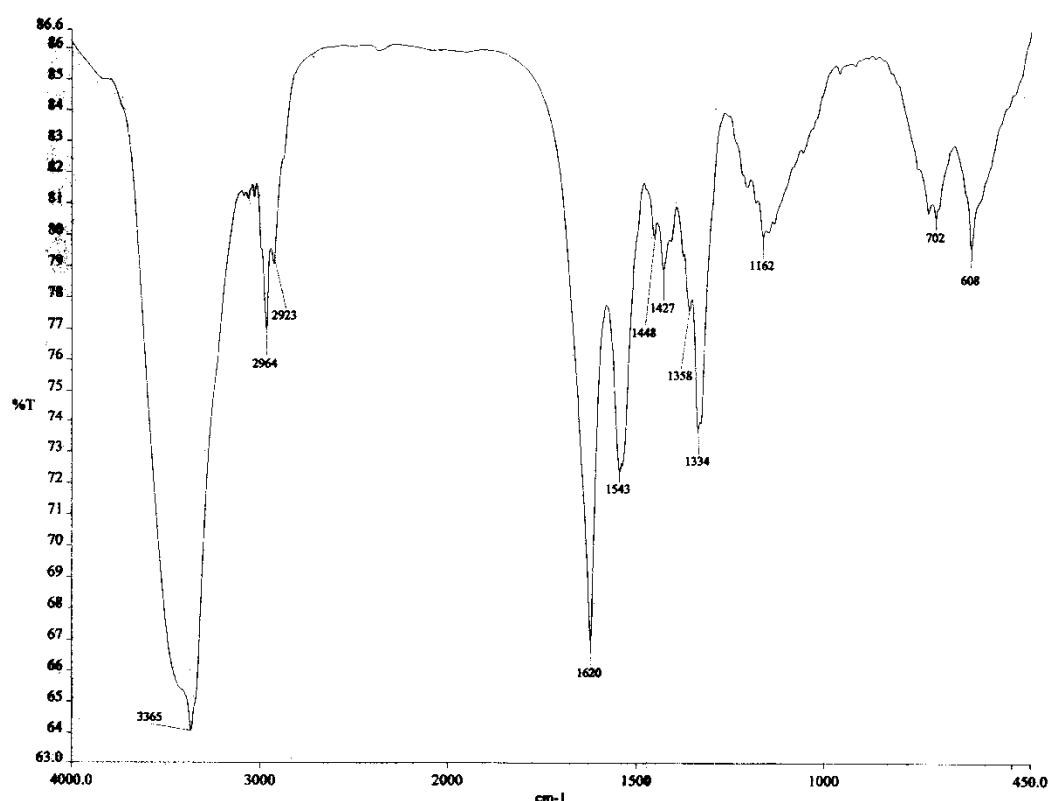
**3-Benzyl-1,1-diisopropyl-urea (17e):  $^1\text{H}$  NMR ( $\text{CDCl}_3$ , 400 MHz)**



**3-Benzyl-1,1-diisopropyl-urea (17e):  $^{13}\text{C}$  NMR ( $\text{CDCl}_3$ , 100 MHz)**

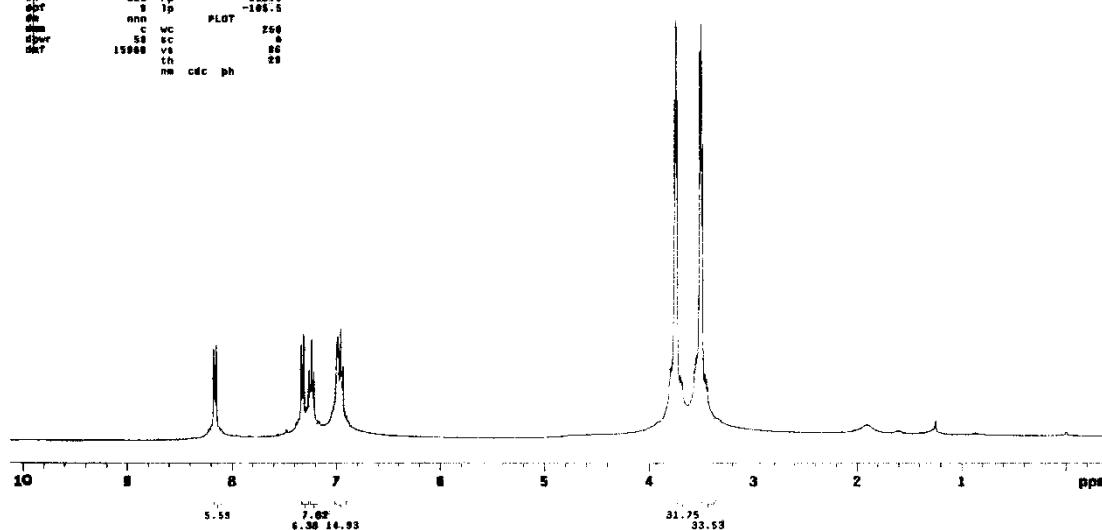
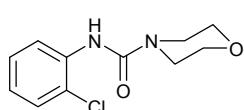


**3-Benzyl-1,1-diisopropyl-urea (17e): IR (KBr)**



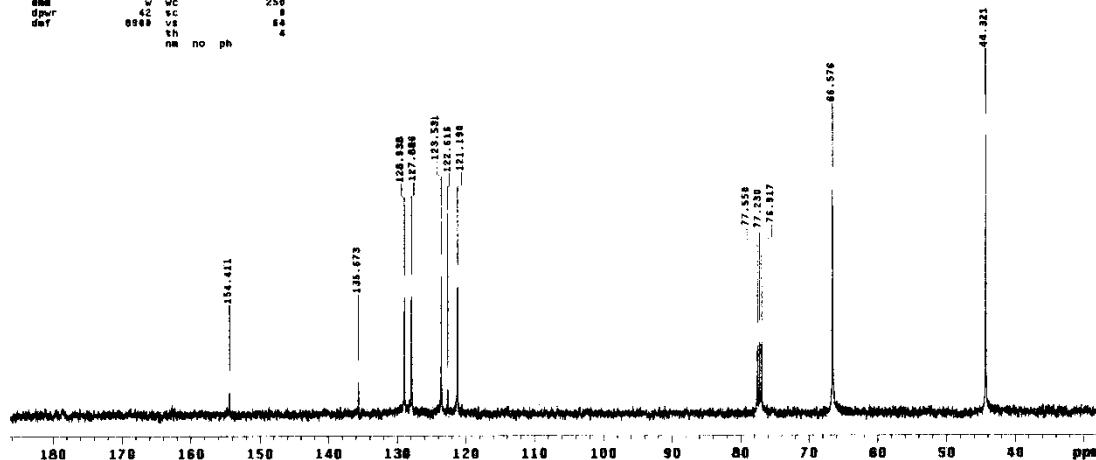
**Morpholine-4-carboxylic acid (2-chloro-phenyl)-amide (18a):  $^1\text{H}$  NMR ( $\text{CDCl}_3$ , 400 MHz)**

```
exp1_s2pm1
SAMPLE          SPECIAL
date Sep 2 2010 temp    not used
solvent   CDCl3 spin    not used
file#    exp1    spin    not used
ACQUISITION hst      8.000
sw      4300.0 pw06    10.700
ns      32760   pw12    20.000
dp      25598   flags
rf      not used 11   n
dd      4      in
et      1.000   ap
et      0.000   ap
ct      32      na
cc      32      PROCESSING
TRANSMITTER 1b      8.18
ta      HI fn    05536
trfq   399.653   DISPLAY
t0f    362.8   sp    -171.9
t1pr   57      w1    4531.0
dp      8.000   r1p   1500.0
DECOUPLER   C13   r1p   2000.0
ph      9      1p    115.0
dpt    9      1p    -105.5
dm      ame   PLOT
dim     c      wc    250
solv   50      sc    0
datf   15988   vs    80
        th    20
        nm cdc ph
```

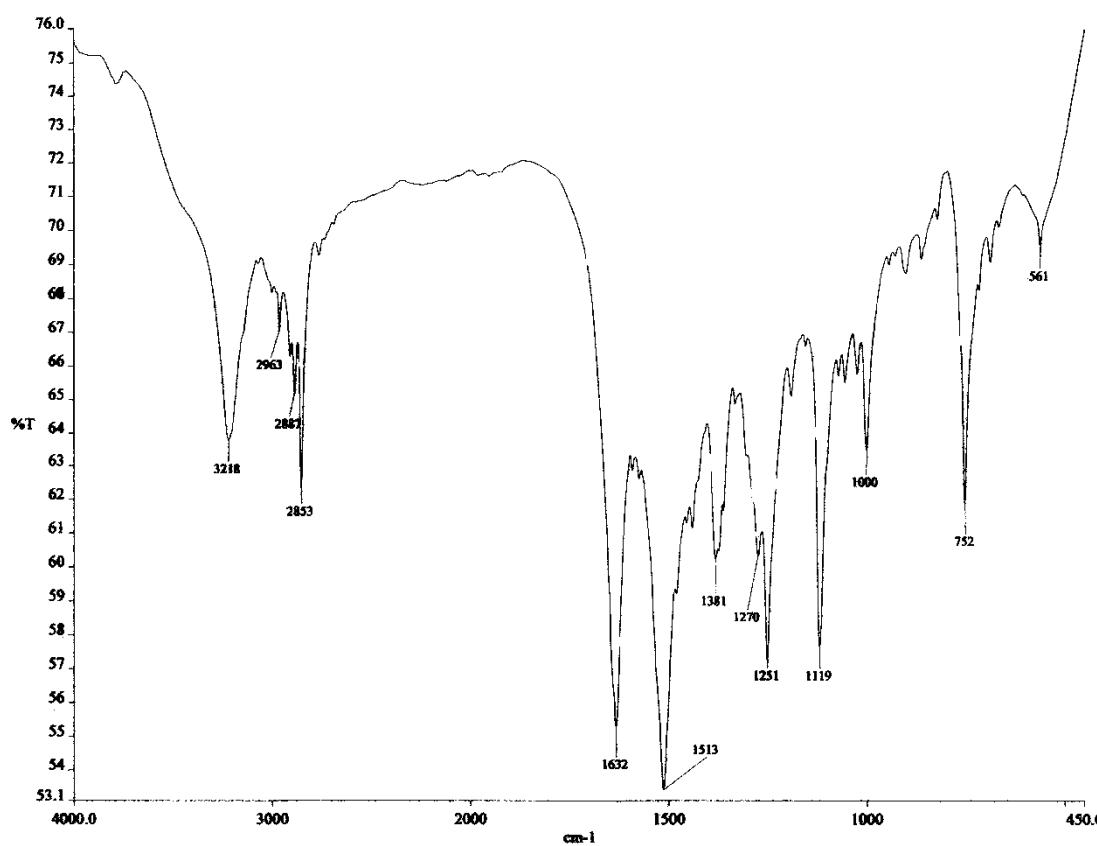


**Morpholine-4-carboxylic acid (2-chloro-phenyl)-amide (18a):  $^{13}\text{C}$  NMR ( $\text{CDCl}_3$ , 100 MHz)**

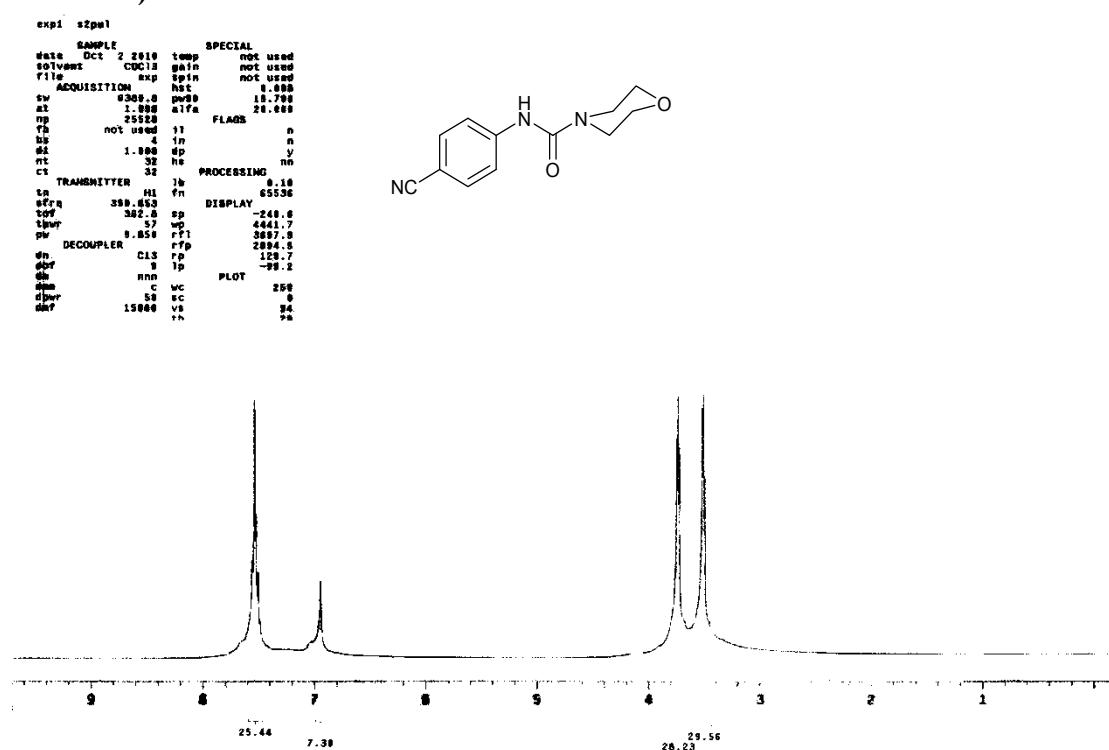
expt s2pu1  
sample 18a  
date Sep 16 2010 temp not used  
solvent CDCl<sub>3</sub> spin not used  
rtic 25.000 sp 18.000  
ACQUISITION nst 0.000  
sw 25125.6 pw88 18.000  
at 60.000 alfa 20.000  
np 60278 FIDABBR  
rb 138000 11 n  
bs 15 t1 1  
sl 1.000 d9 y  
nt 50000 ns nn  
ct 1024  
TRANSMITTER Cl3 1b 2.00  
tn 100.554 fm 65516  
sfra 100.554 DISPLAY  
tfr 1538.3 tp 2714.9  
tpwr 81 w 180.00  
pw 9.348 rfp 7764.9  
DECOUPLER H1 rfp 7764.9  
dn H1 rp -54.1  
dof 1p -386.2  
de vvv PLOT  
dm w vc 250  
dpwr 42 vc 8  
def 8888 vs 8  
d1 th 4  
nm no ph 4



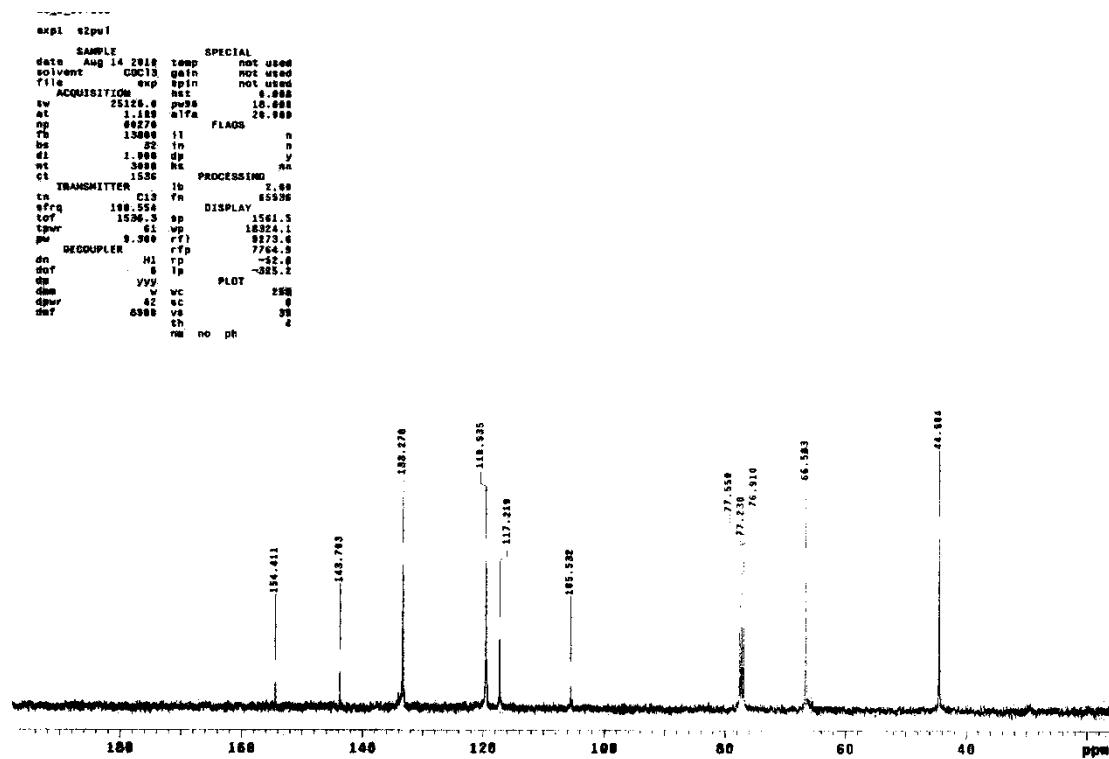
**Morpholine-4-carboxylic acid (2-chloro-phenyl)-amide (18a): IR (KBr)**



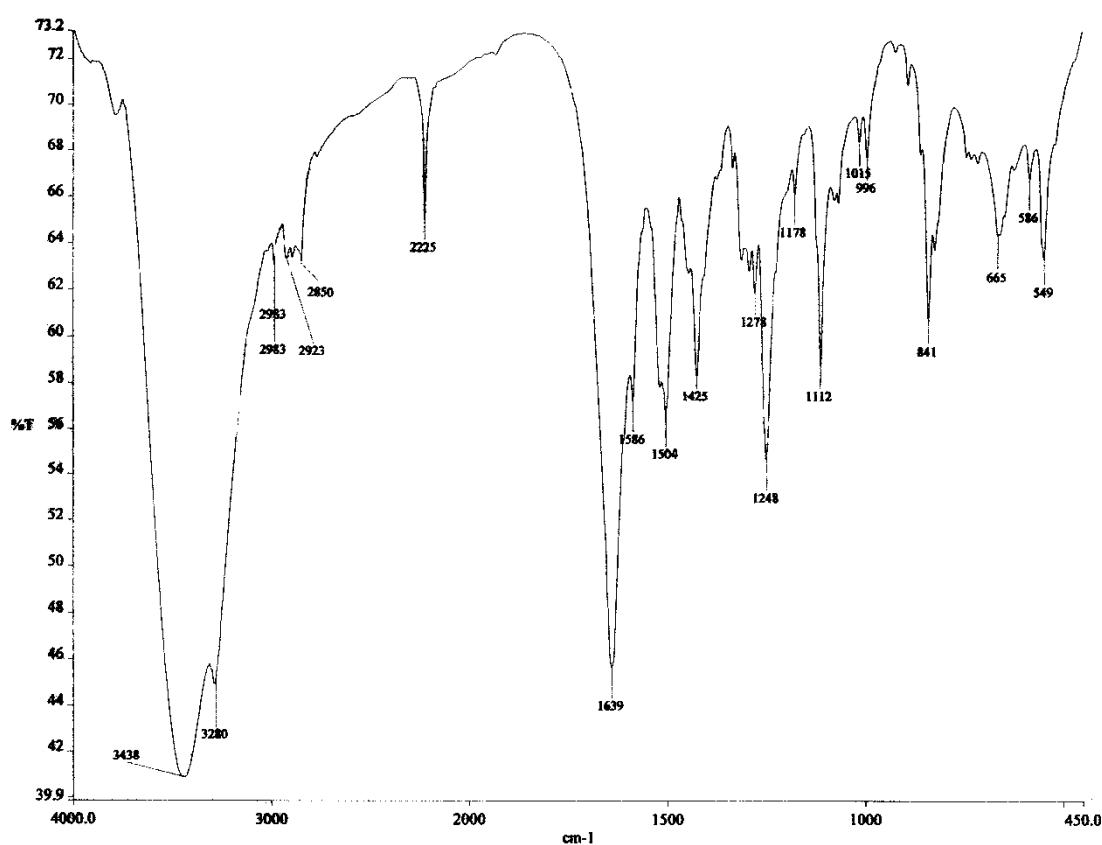
**Morpholine-4-carboxylic acid (4-cyano-phenyl)-amide (19a):  $^1\text{H}$  NMR ( $\text{CDCl}_3$ , 400 MHz)**



**Morpholine-4-carboxylic acid (4-cyano-phenyl)-amide (19a):  $^{13}\text{C}$  NMR ( $\text{CDCl}_3$ , 100 MHz)**

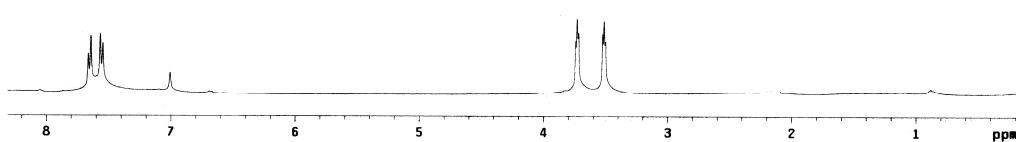
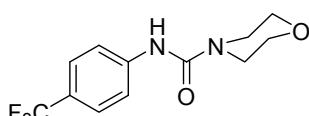


**Morpholine-4-carboxylic acid (4-cyano-phenyl)-amide (19a): IR (KBr)**

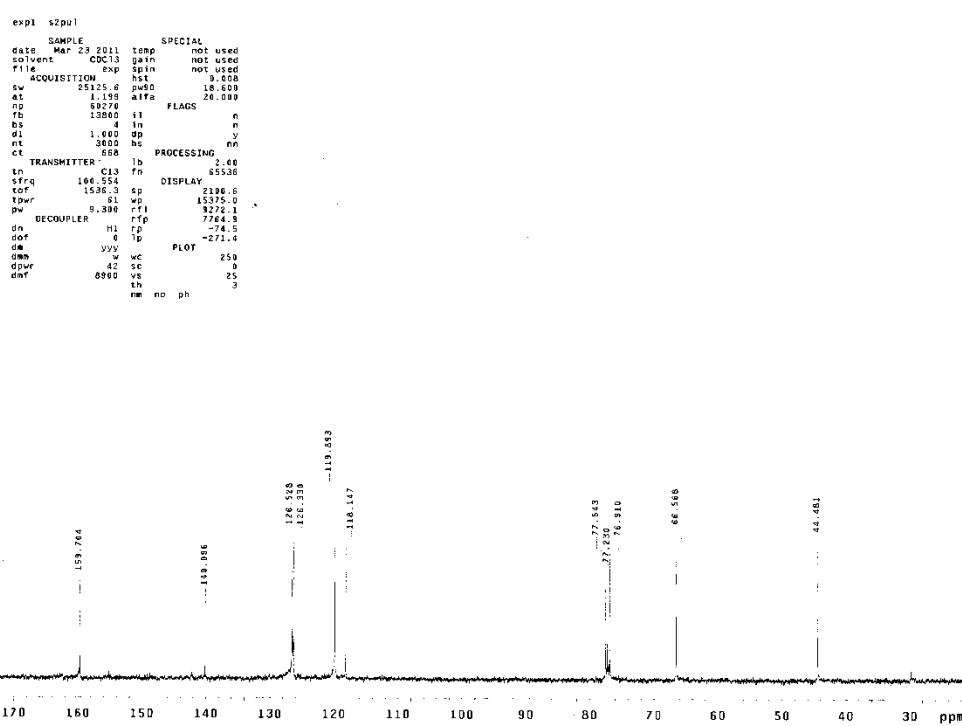


**Morpholine-4-carboxylic acid (4-trifluoromethyl-phenyl)-amide (20a):  $^1\text{H}$  NMR ( $\text{CDCl}_3$ , 400 MHz)**

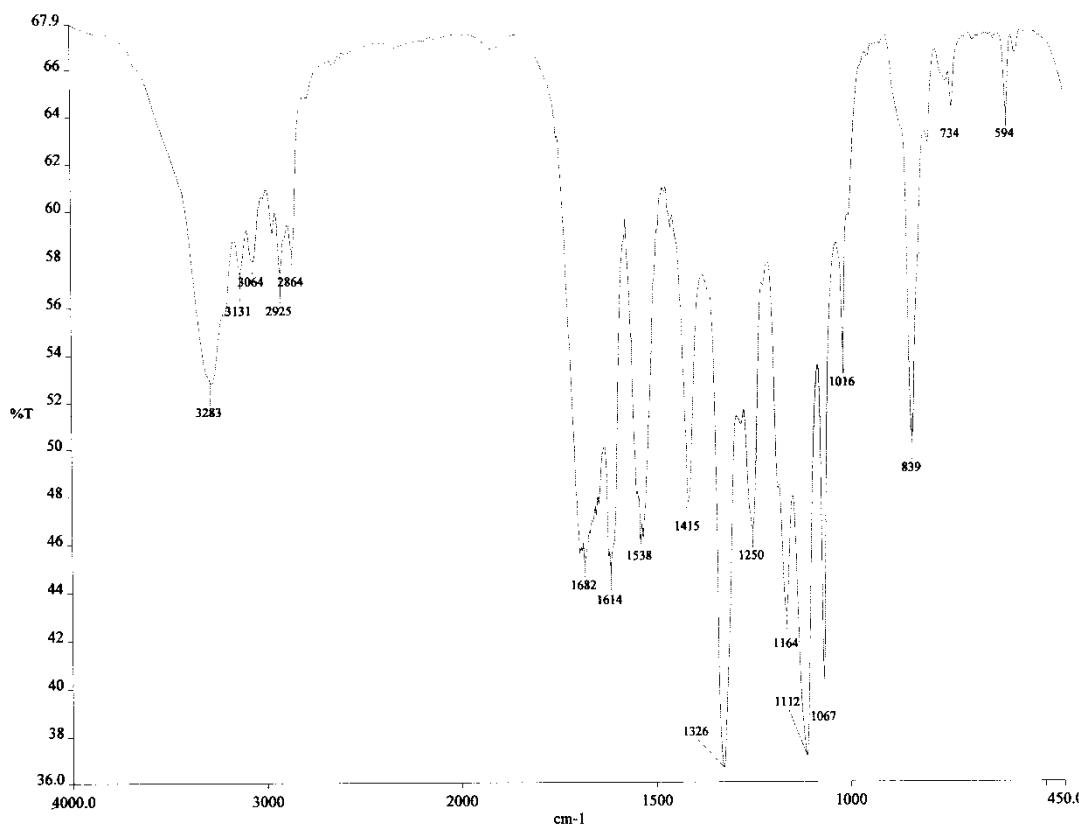
```
expt s2pul
date Mar 23 2011 temp not used
solvent CDCl3 gain not used
rf1 399.88 exp spin not used
ACQUISITION pw0.0 19.700
sw 6389.8 pw0.0 20.000
at 1.998 alra
np 25568
nb not used j1 n
bs 4 in n
dt 1.00 n
ct 32 ns nn
ct 32 PROCESSING
TRANSMITTER 1b 8.10
tn H1 f1 65536
sfrq 399.853 DISPLAY
t0f 362.8 sp 59.3
tpw 7.0 rfp 324.8
pw 9.850 rfp 789.4
DECOUPLER 8.50 rfp 0
dn c13 rfp 128.2
dof 0 ip -31.6
dm nnn PLOT
dss c sc 250
dpgr 15900 vs 39
dmf th cdc ph 82
nm cdc ph
```



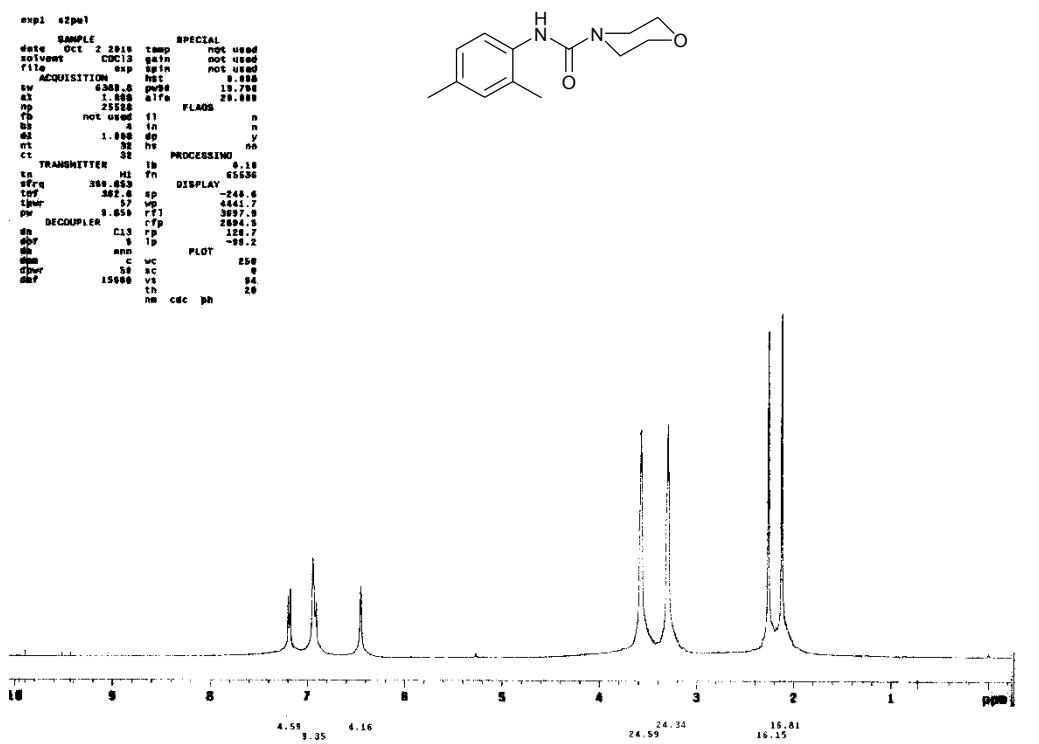
**Morpholine-4-carboxylic acid (4-trifluoromethyl-phenyl)-amide (20a)  $^{13}\text{C}$  NMR ( $\text{CDCl}_3$ , 100 MHz)**



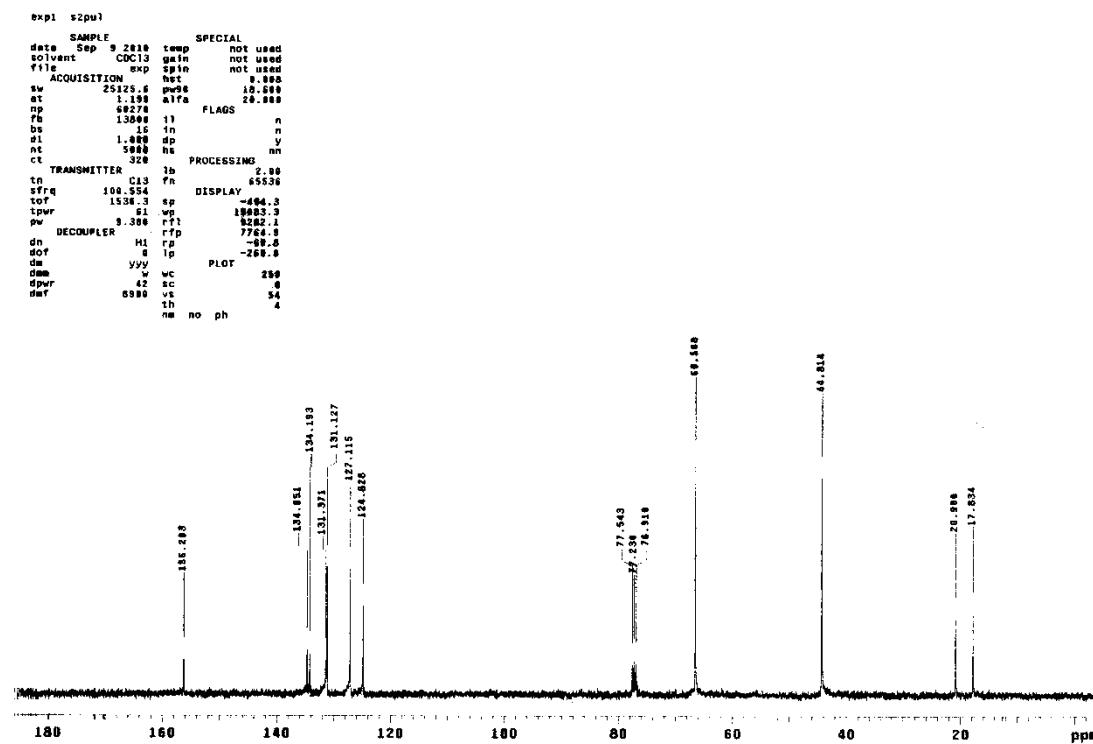
**Morpholine-4-carboxylic acid (4-trifluoromethyl-phenyl)-amide (20a): IR (KBr)**



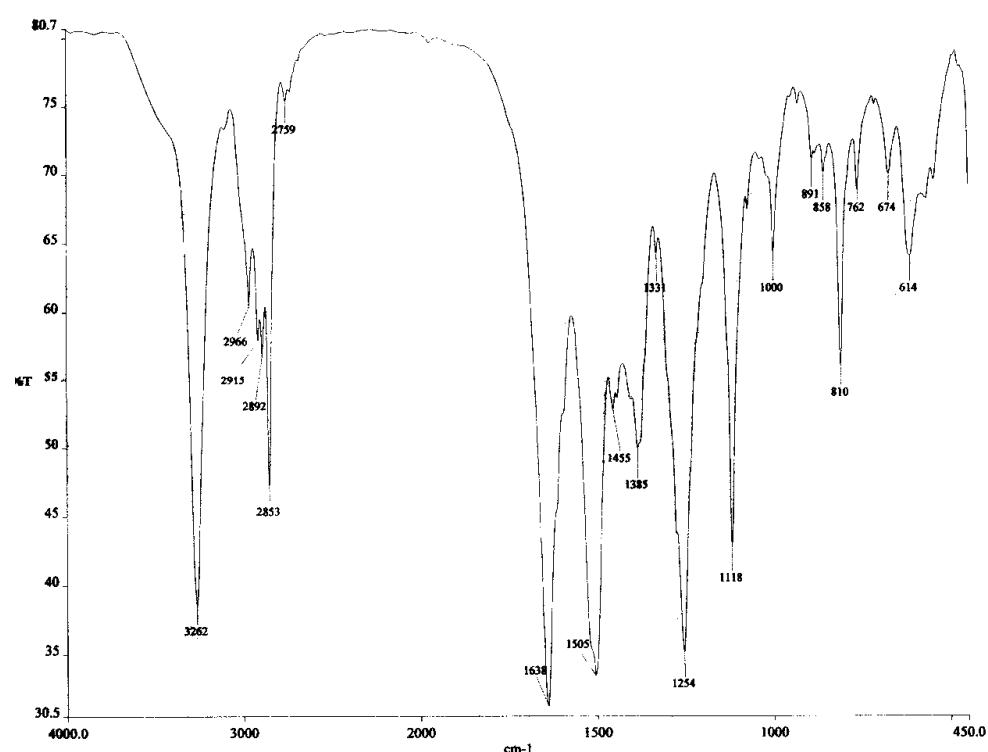
**Morpholine-4-carboxylic acid (2,4-dimethyl-phenyl)-amide (21a):  $^1\text{H}$  NMR ( $\text{CDCl}_3$ , 400 MHz)**



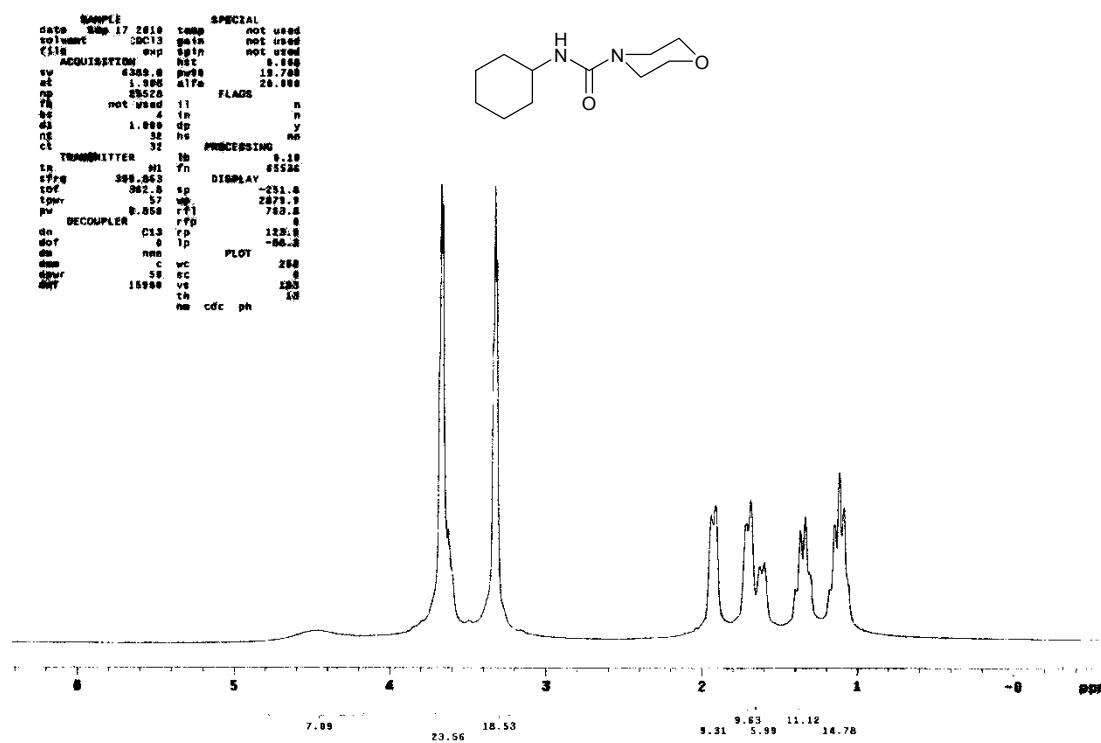
**Morpholine-4-carboxylic acid (2,4-dimethyl-phenyl)-amide (21a):  $^{13}\text{C}$  NMR ( $\text{CDCl}_3$ , 100 MHz)**



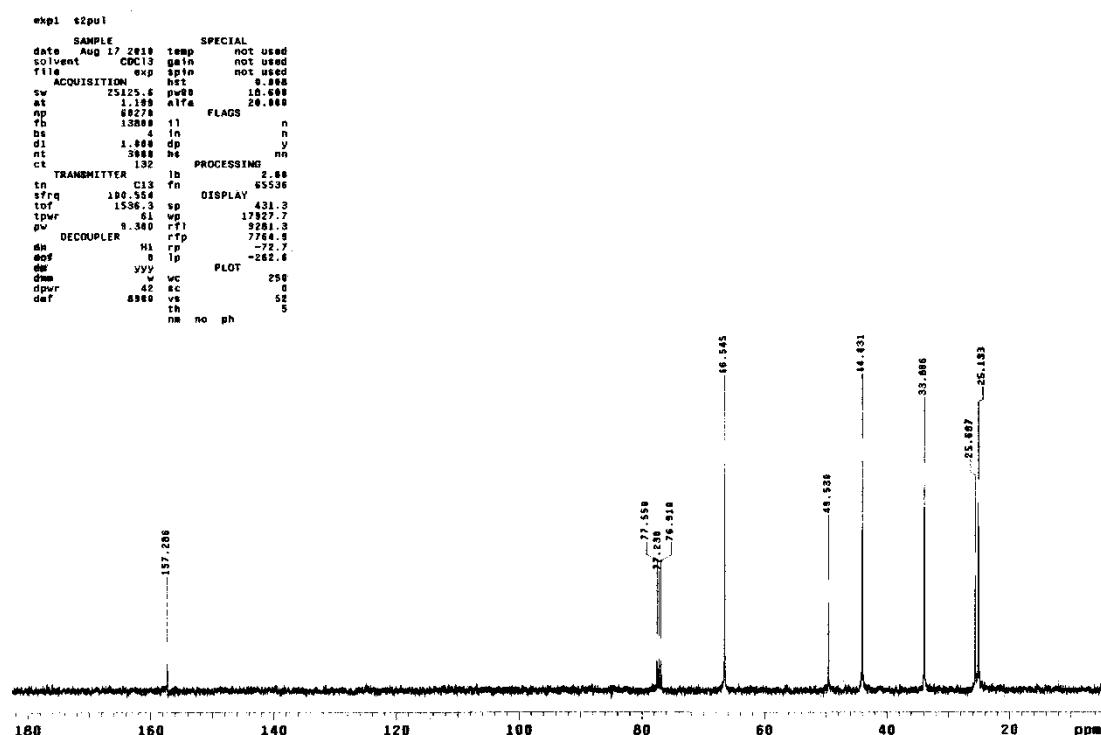
**Morpholine-4-carboxylic acid (2,4-dimethyl-phenyl)-amide (21a): IR(KBr)**



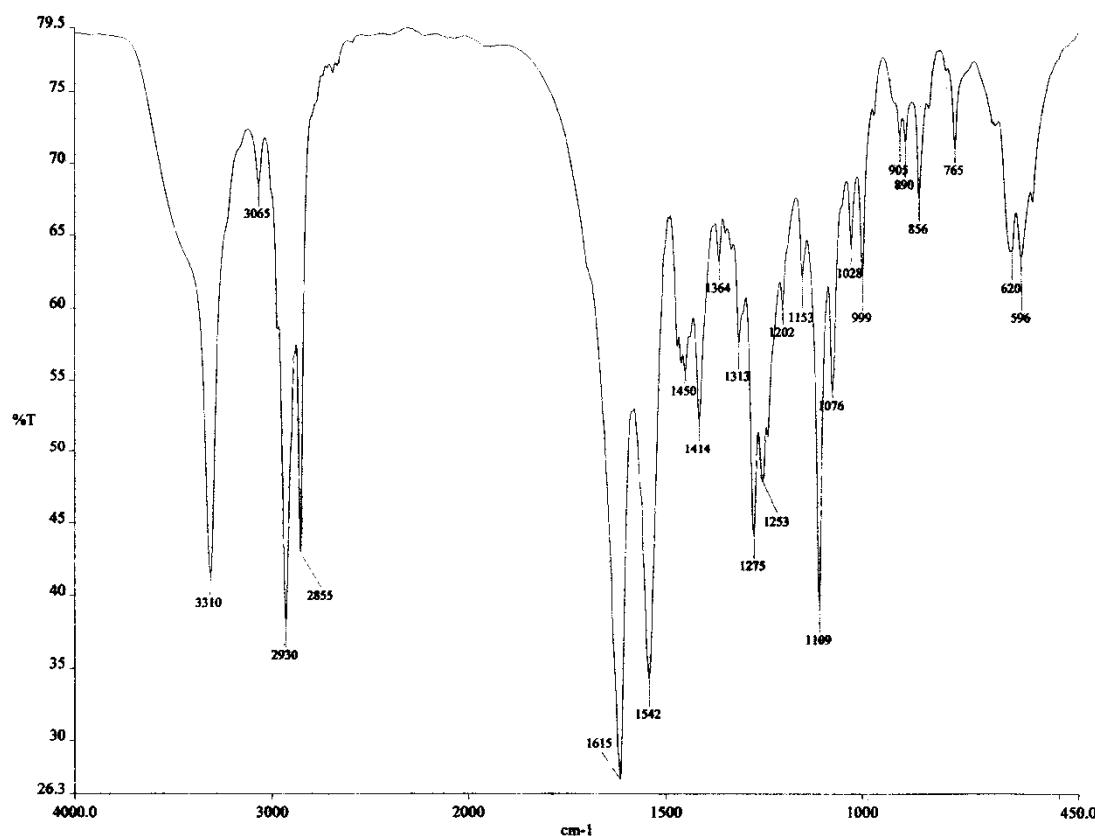
**Morpholine-4-carboxylic acid cyclohexylamide (22a): <sup>1</sup>H NMR (CDCl<sub>3</sub>, 400 MHz)**



**Morpholine-4-carboxylic acid cyclohexylamide (22a):  $^{13}\text{C}$  NMR ( $\text{CDCl}_3$ , 100 MHz)**

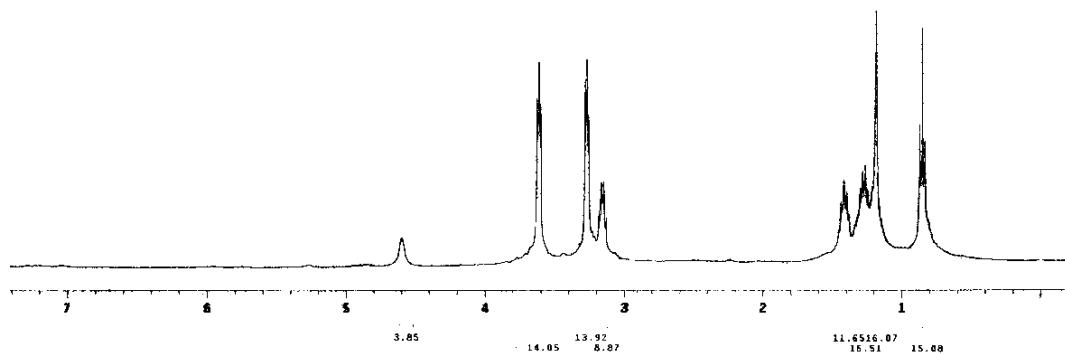
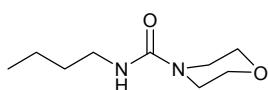


**Morpholine-4-carboxylic acid cyclohexylamide (22a): IR (KBr)**



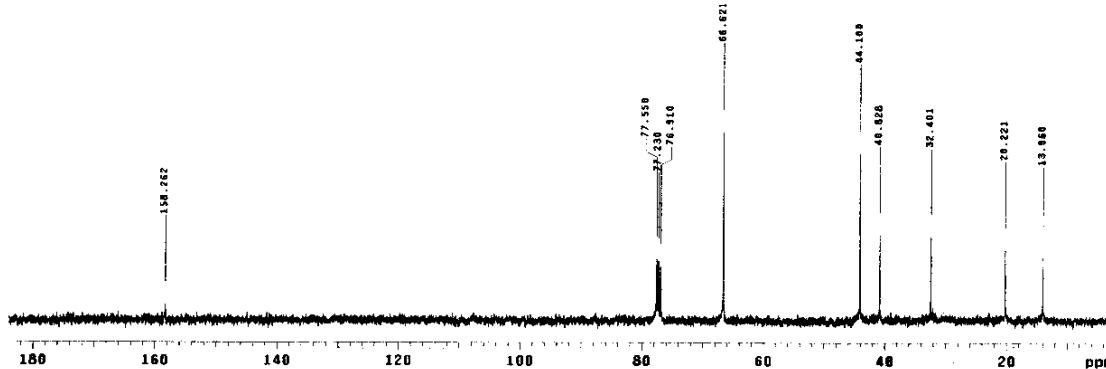
Morpholine-4-carboxylic acid butylamide (23a):  $^1\text{H}$  NMR ( $\text{CDCl}_3$ , 400 MHz)

```
expt s2pul
SAMPLE          SPECIAL
date Oct 2 2010 temp    not used
solvent   CDCl3 gain    not used
file      exp spin    not used
ACQUISITION nct    6.700
sw       8340.0   10.700
at       1.000   617a   20.000
rp       25520   flags
rfb      not used 11   n
ds       4   in   n
dt       1.000   dp   y
nt       32   nc   nn
ct       32   PROCESSING
TRANSMITTER H1 fn   0.10
ta       65536
stftr  338.613   DISPLAY
t0f    342.3   sp   249.6
tprtr  57   wp   4441.7
pw     0.850   r11   3697.0
DECOUPLER C13 rfp   200.0
dn      C13 rfp   129.7
dof     8   1p   -99.2
dm      v   wc   250
dpwr   89   sc   8
dmt    15948   v6   84
th     20   nc   20
nm   cdc ph
```

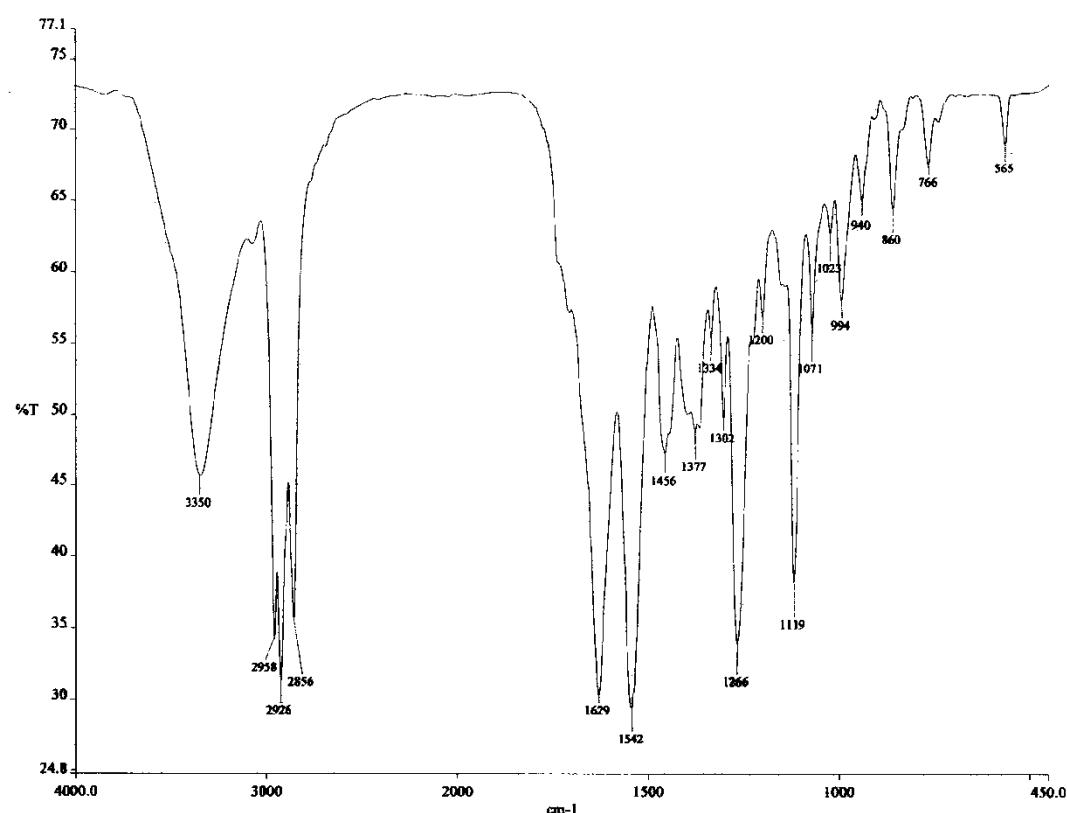


Morpholine-4-carboxylic acid butylamide (23a):  $^{13}\text{C}$  NMR ( $\text{CDCl}_3$ , 100 MHz)

```
expt s2pul
SAMPLE          SPECIAL
date Jan 28 2011 temp    not used
solvent   CDCl3 gain    not used
file      exp spin    not used
ACQUISITION nct    9.800
sw       2525.6   10.800
at       1.195   617a   20.000
rp       68270   flags
rfb      13890   11   n
ds       4   in   n
dt       1.000   dp   y
nt       3000   nc   nn
ct       532   PROCESSING
TRANSMITTER C13 fn   2.00
ta       65536
stftr  100.554   DISPLAY
t0f    1536.3   sp   172.6
tprtr  51   wp   1000.1
pw     9.300   r11   9275.2
DECOUPLER C13 rfp   7764.9
dn      H1 rfp   -67.5
dof     8   1p   -279.7
dm      v   wc   250
dpwr   89   sc   8
dmt    8940   v6   43
th     20   nc   20
nm   no ph
```

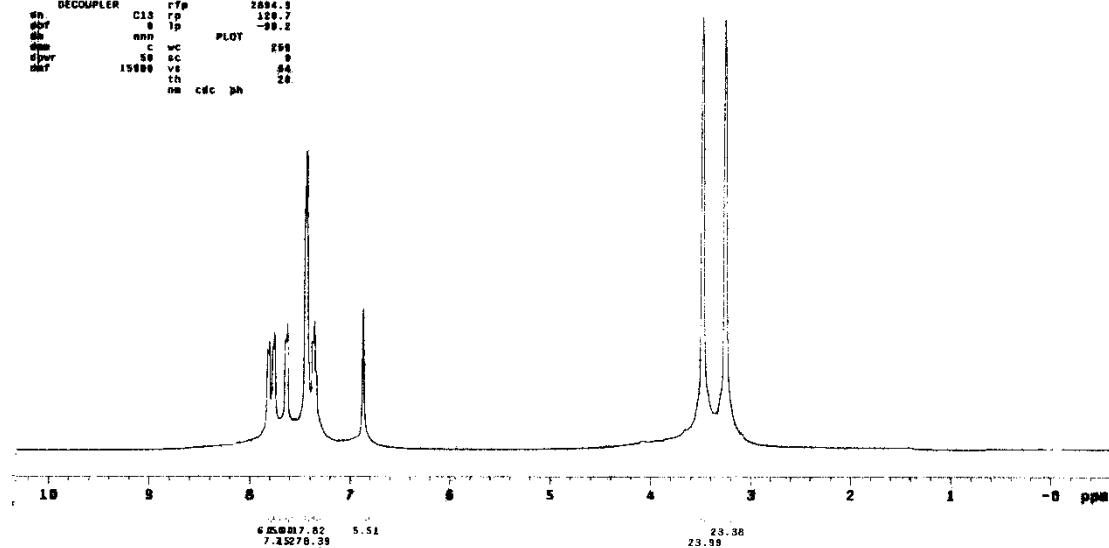
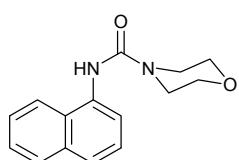


**Morpholine-4-carboxylic acid butylamide (23a): IR (KBr)**

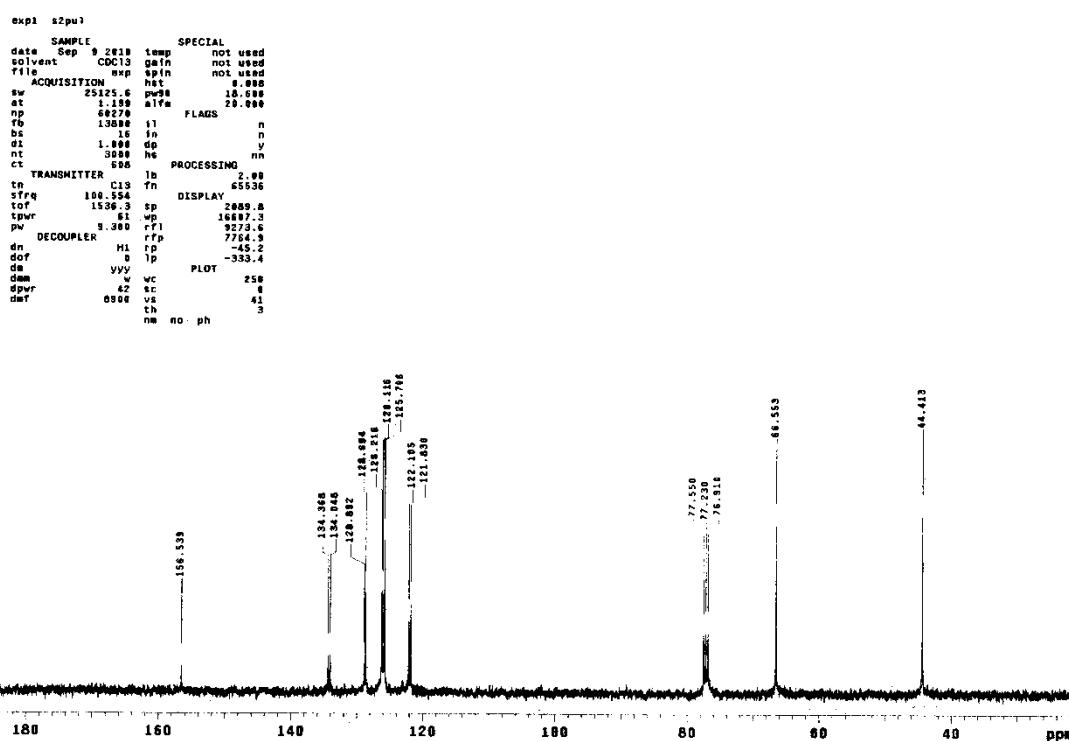


**Morpholine-4-carboxylic acid naphthalen-2-ylamide (24a):  $^1\text{H}$  NMR ( $\text{CDCl}_3$ , 400 MHz)**

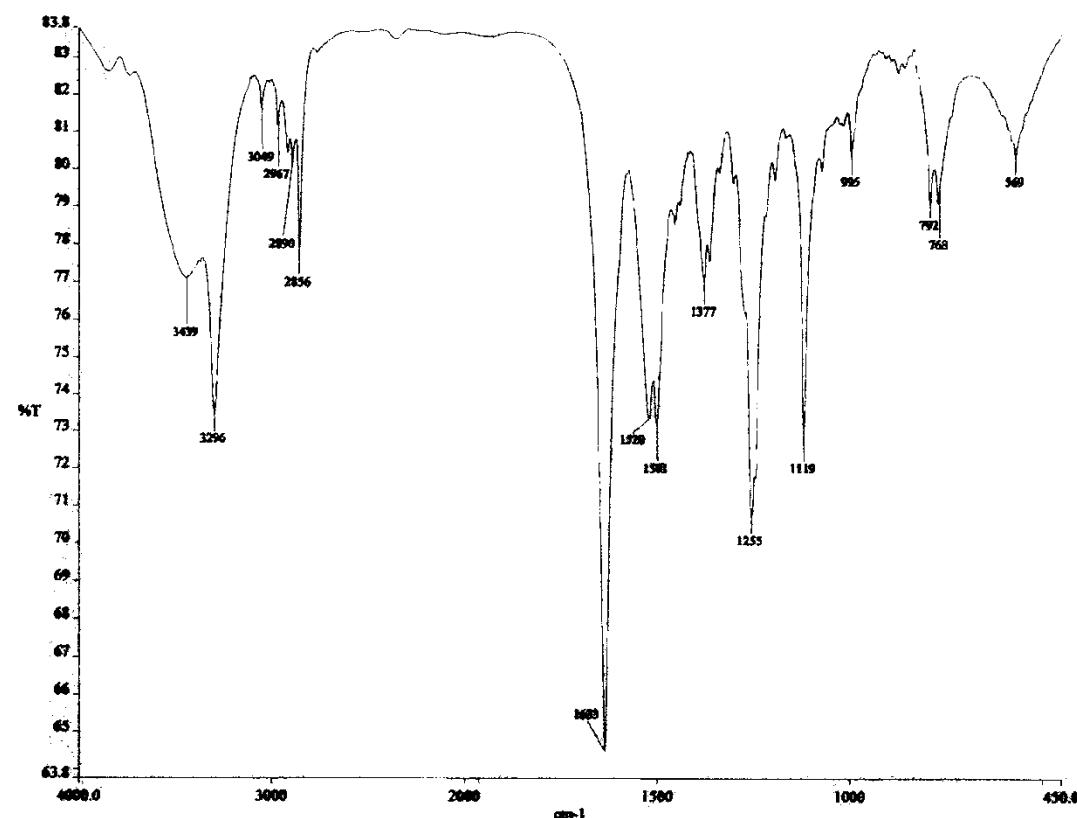
exp1 s2pu1  
SAMPLE  
date Oct 2 2010 temp not used  
solvent CDCl<sub>3</sub> spin not used  
file exp spin not used  
ACQUISITION hat 0.005  
sw 6308.0 pw0 10.700  
et 28.000 a1fa 28.000  
ng 25528  
rf not used t1 n  
ns 4 in  
dt 1.000 dp y  
nt 32 hs na  
ct 32 PROCESSING  
tn H1 fn 0.10  
sfreq 399.653 DISPLAY  
tpp 362.8 sp -240.6  
tthr 57 w0 4441.7  
pp 0.050 rfp 2880.3  
decoupler C13 rfp 2880.3  
sn 0 ip 129.7  
gbr 0 ip -99.2  
dme mmr PLOT  
dmr 0 sc 250  
dmr 59 sc 9  
dmr 15000 vs 84  
dmr th 20  
nm cdc ph



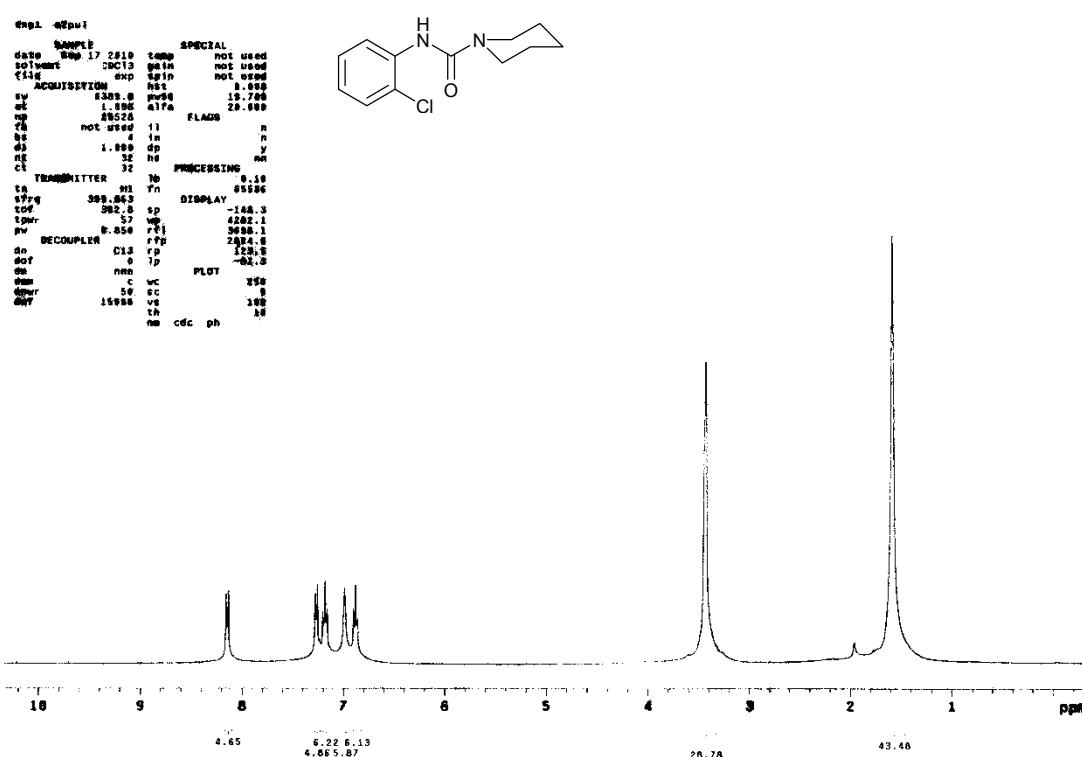
**Morpholine-4-carboxylic acid naphthalen-2-ylamide (24a):  $^{13}\text{C}$  NMR ( $\text{CDCl}_3$ , 100 MHz)**



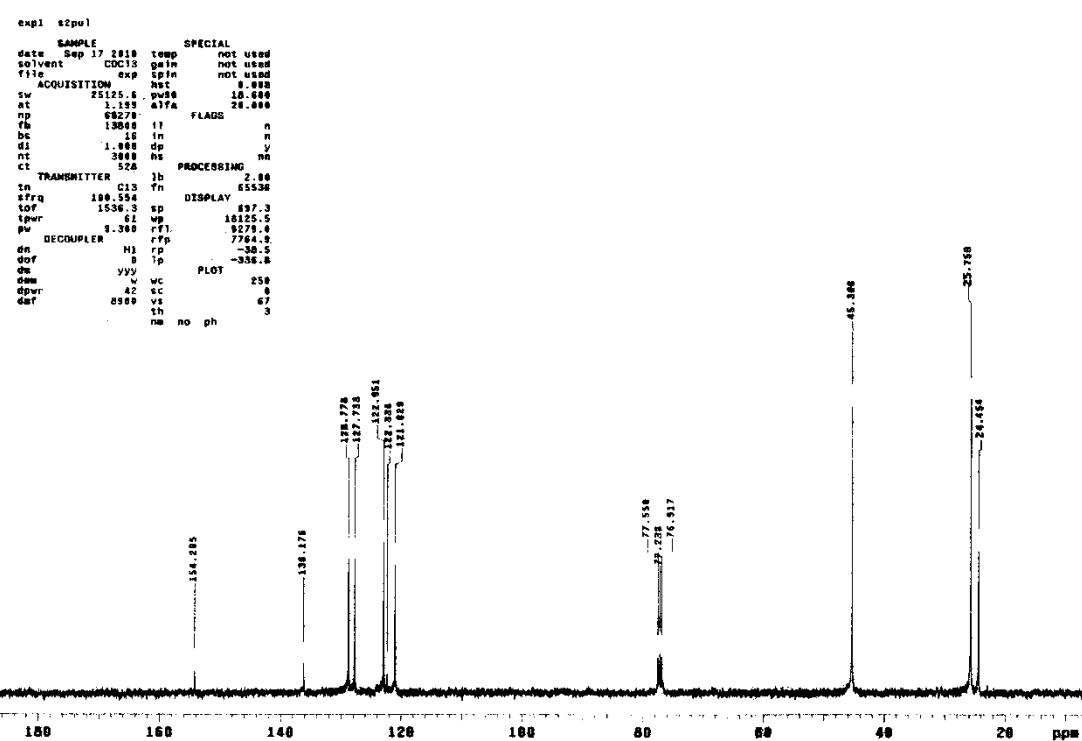
**Morpholine-4-carboxylic acid naphthalen-2-ylamide (24a): IR (KBr)**



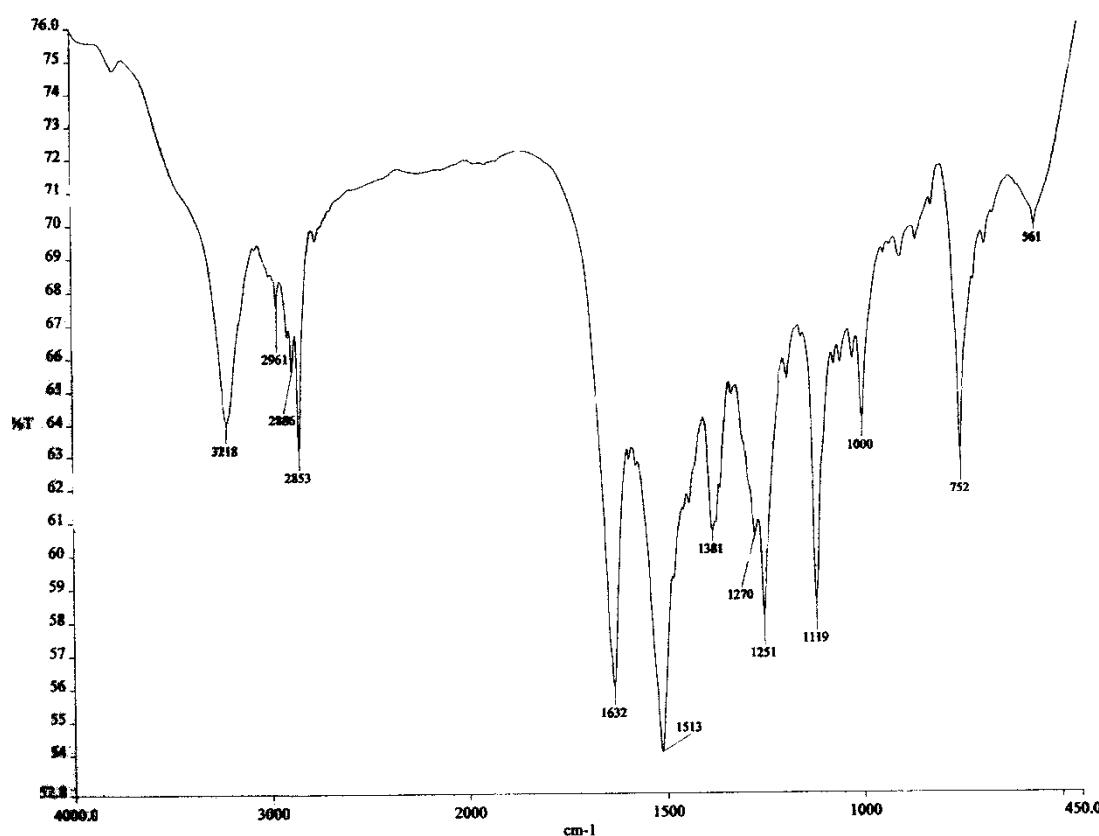
Piperidine-1-carboxylic acid (2-chloro-phenyl)-amide (18b):  $^1\text{H}$  NMR ( $\text{CDCl}_3$ , 400 MHz)



Piperidine-1-carboxylic acid (2-chloro-phenyl)-amide (18b):  $^{13}\text{C}$  NMR ( $\text{CDCl}_3$ , 100 MHz)

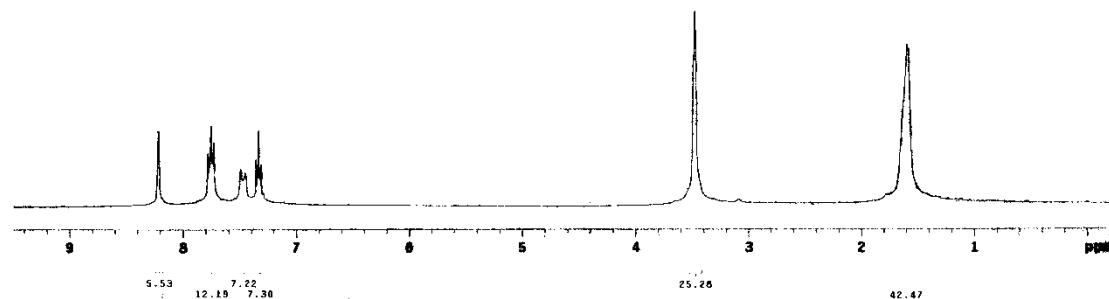
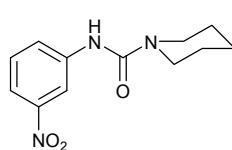


Piperidine-1-carboxylic acid (2-chloro-phenyl)-amide (18b): IR (KBr)



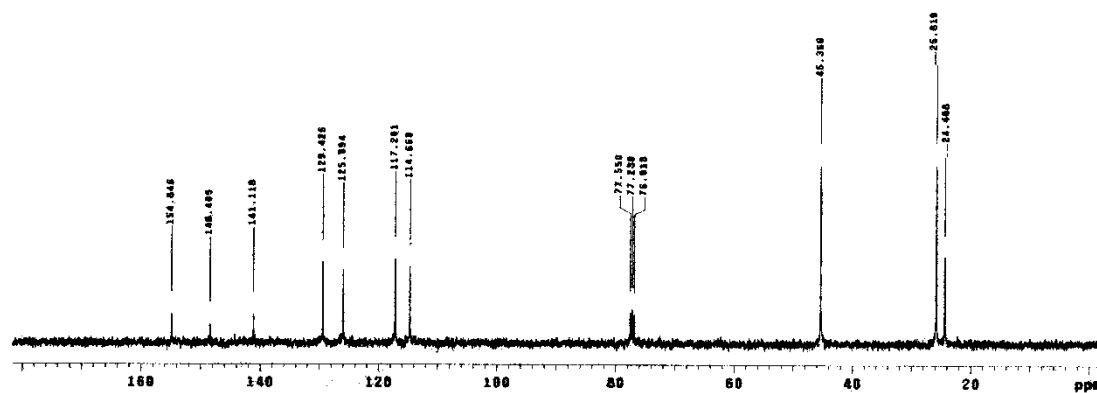
Piperidine-4-carboxylic acid (3-nitro-phenyl)-amide (25b): <sup>1</sup>H NMR (CDCl<sub>3</sub>, 400 MHz)

SAMPLE: Jan 26 2011 temp: not used  
solvent: CDCl<sub>3</sub> pres: not used  
Pulse: exp: spin: not used  
SW: FIDWID: pds: 12768  
ET: 1.000 atten: 28.994  
OB: 25528 PLADS:  
TS: not used: 11: n  
DS: 4: in: n  
DT: 1.000: dp: y  
RT: 32: ns: nn  
CT: 32: PROCEESSING:  
TRANSMITTER: 1b: 6.19  
TR: 300.000: H1: 65536  
SFRE: 300.000: DISPLAY:  
DPP: 300.000: SP: 100.0  
TDR: 1.000: WP: 3000.0  
PW: 9.850: RTF: 760.0  
DECOPPLER: C13: RTF:  
DE: C13: TP: 100.0  
DD: 8: TP: -10.0  
DPP: 15000: TC: 150  
DPP: 15000: VTE: 44  
DPP: 15000: TH: 20  
NM: cdc ph:

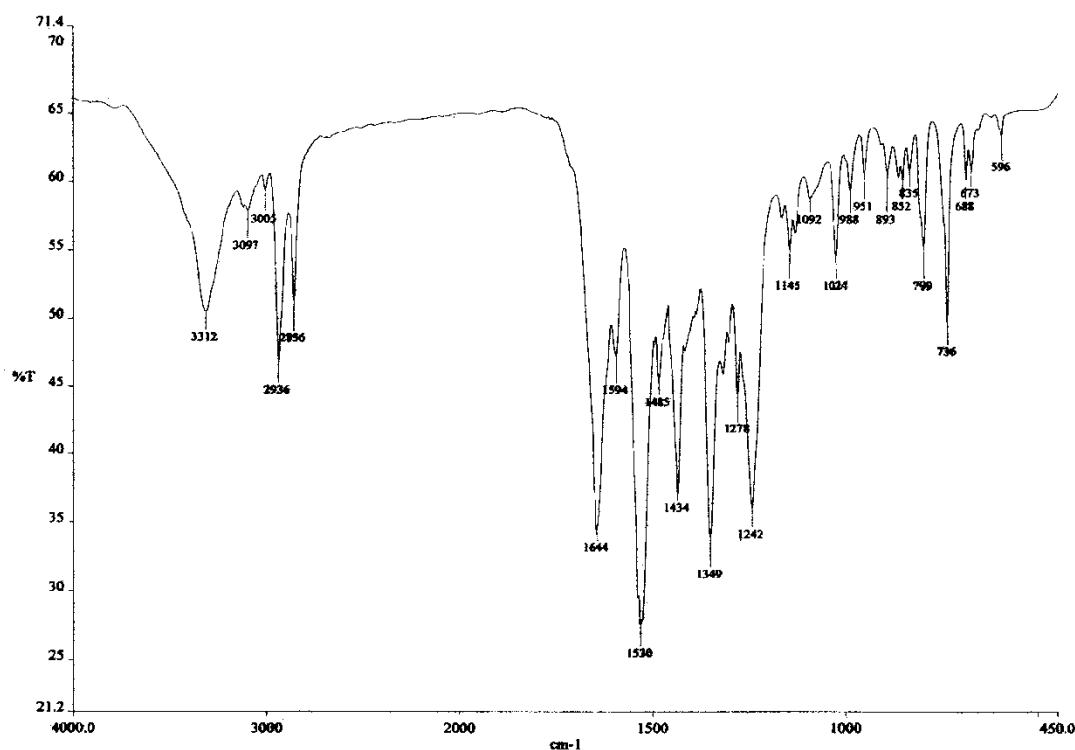


**Morpholine-4-carboxylic acid (3-nitro-phenyl)-amide (25b):  $^{13}\text{C}$  NMR ( $\text{CDCl}_3$ , 100 MHz)**

data 1 Jan 28 2011 sample SPECIAL  
solvent CDCl<sub>3</sub> gain not used  
file bpx gain not used  
ACQUISITION pw90 16.000  
sw 25125.4 pw90 16.000  
at 1.300 atra 28.000  
rt 68279 flags n  
fb 13000 t1 n  
bs 13000 te n  
di 1.000 dp v  
nt 30000 hs nr  
ct 300 PROCESSING  
TRANSMITTER 1b 2.00  
tr C13 fm 65536  
afrq 180.554 DISPLAY  
t0f 1532.3 sp -282.5  
tpwr 81 vp 16554.9  
pw 9.389 r1 9279.0  
DECOUPLER H1 fp 7774.5  
dn 1H fp -32.5  
def 3 s PLOT  
dec vvv wc 256  
dppr 42 sc 0  
det 8980 vs 41  
nm no ph

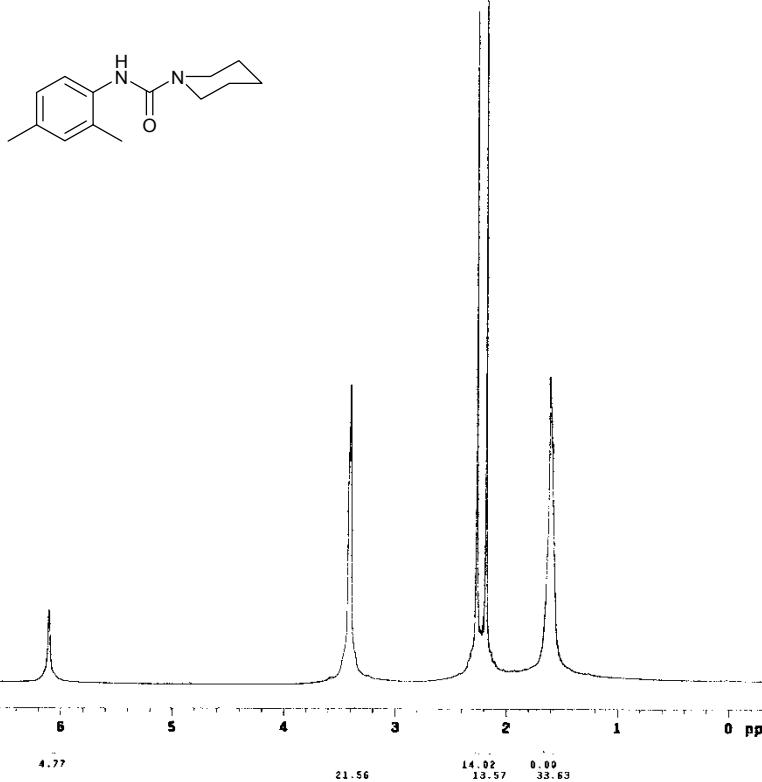


**Morpholine-4-carboxylic acid (3-nitro-phenyl)-amide (25b): IR (KBr)**



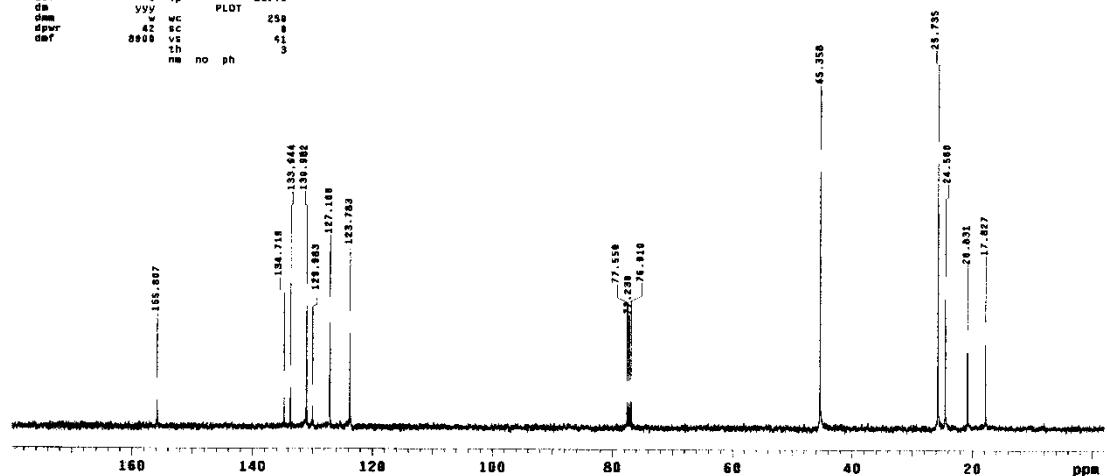
Piperidine-1-carboxylic acid (2,4-dimethyl-phenyl)-amide (21b):  $^1\text{H}$  NMR ( $\text{CDCl}_3$ , 400 MHz)

```
expt1 s2pu1
SAMPLE          SPECIAL
date Sep 2 2010 temp not used
solvent CDCl3 gain not used
file   exp spin not used
ACQUISITION      int
sw   8388.0 pswd 15.798
et   7.380 alfa 20.000
np   25520   FLAGS
fb   not used 11 n
bs   16   in n
di   1.000   dp y
nt   32   hs nn
ct   32   PROCESSING
tn   TRANSMITTER 1b 0.10
tr   H1 fm 65536
trfq  500.653   DISPLAY
tppr  382.8 sp -176.7
tppw  57    wp 4887.5
pw   9.854 r71 798.5
decoupler       C13 r7p
dn   C13 r7p 121.3
dof   0   1p -99.3
dm   vyy   PLOT
dmw  50   sc 0
dpwr  15000 vs 152
dmtf  15000 th 16
nm cdc ph
```

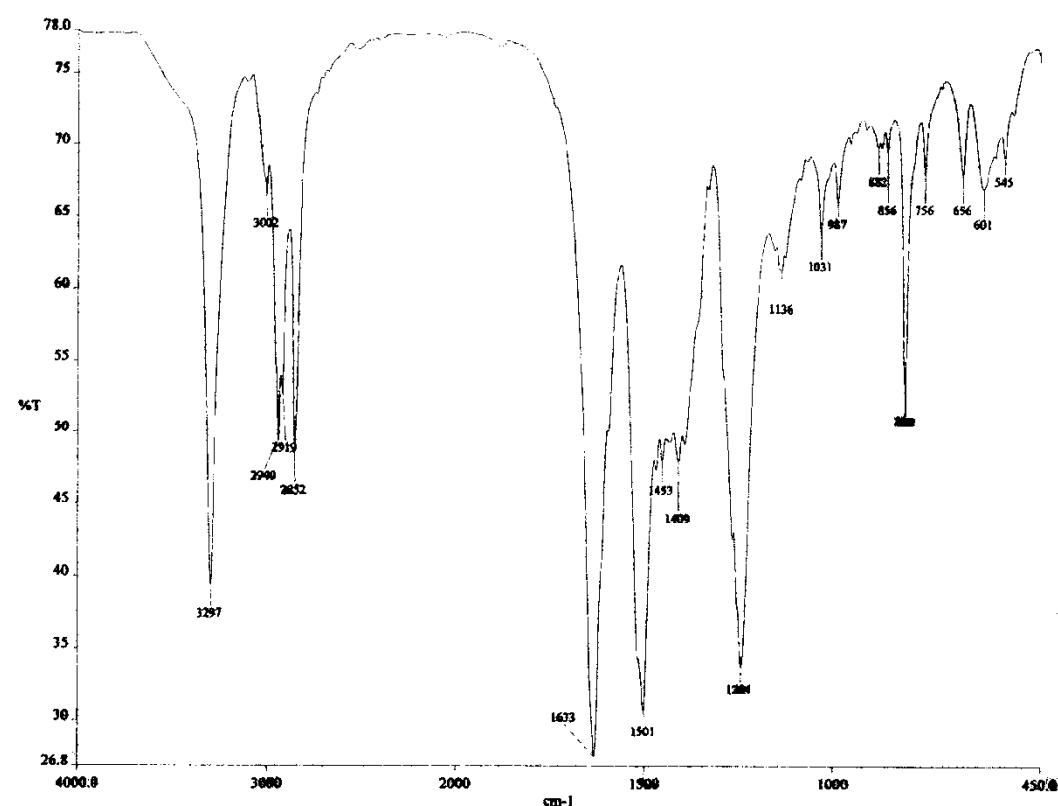


Piperidine-1-carboxylic acid (2,4-dimethyl-phenyl)-amide (21b):  $^{13}\text{C}$  NMR ( $\text{CDCl}_3$ , 100 MHz)

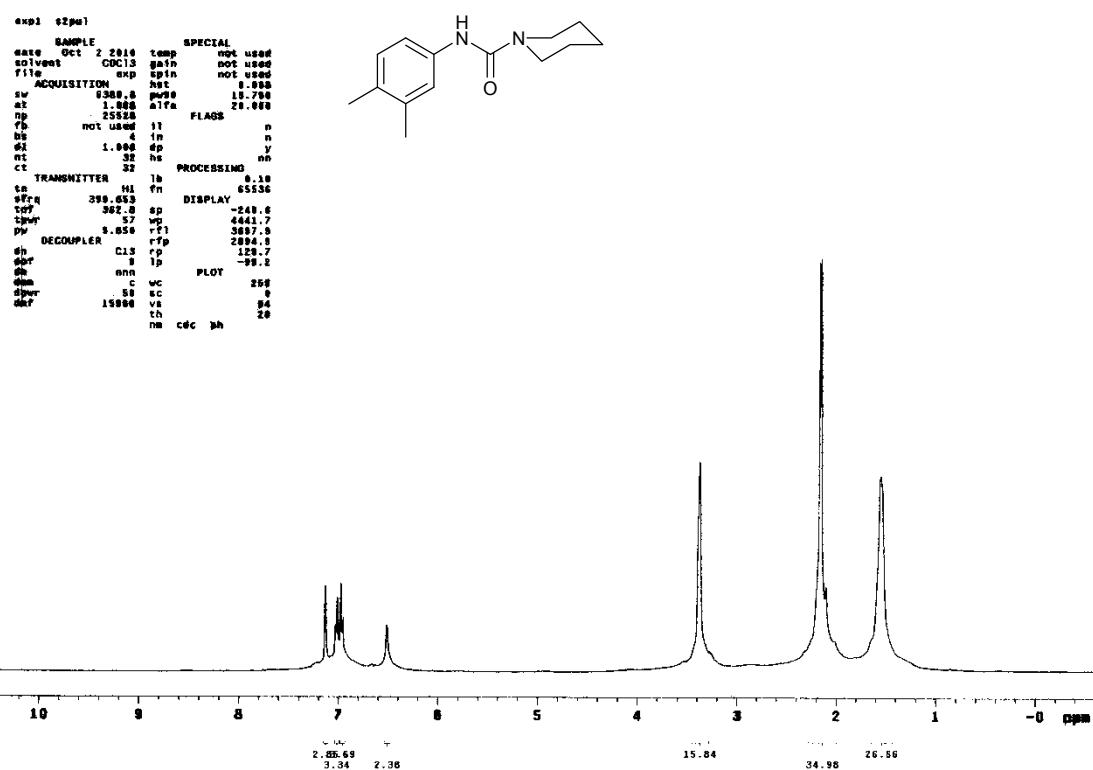
```
expt1 s2pu1
SAMPLE          SPECIAL
date Sep 1 2010 temp not used
solvent CDCl3 gain not used
file   exp spin not used
ACQUISITION      int
sw   25125.6 pswd 16.688
et   1.180 alfa 20.000
np   68270   FLAGS
fb   13000   11 n
bs   16   in n
di   1.000   dp y
nt   2880   hs nn
ct   288   PROCESSING
tn   TRANSMITTER 1b 2.00
tr   C13 fm 65536
trfq  100.653   DISPLAY
tppr  61    wp -285.3
tppw  9.380 r71 10281.2
pw   9.380 r71 9286.7
decoupler       r7p 7704.9
dn   H1 r7p -176.3
dof   0   1p -257.1
dm   vyy   PLOT
dmw  42   sc 258
dpwr  8800 vs 61
dmtf  8800 th 3
nm no ph
```



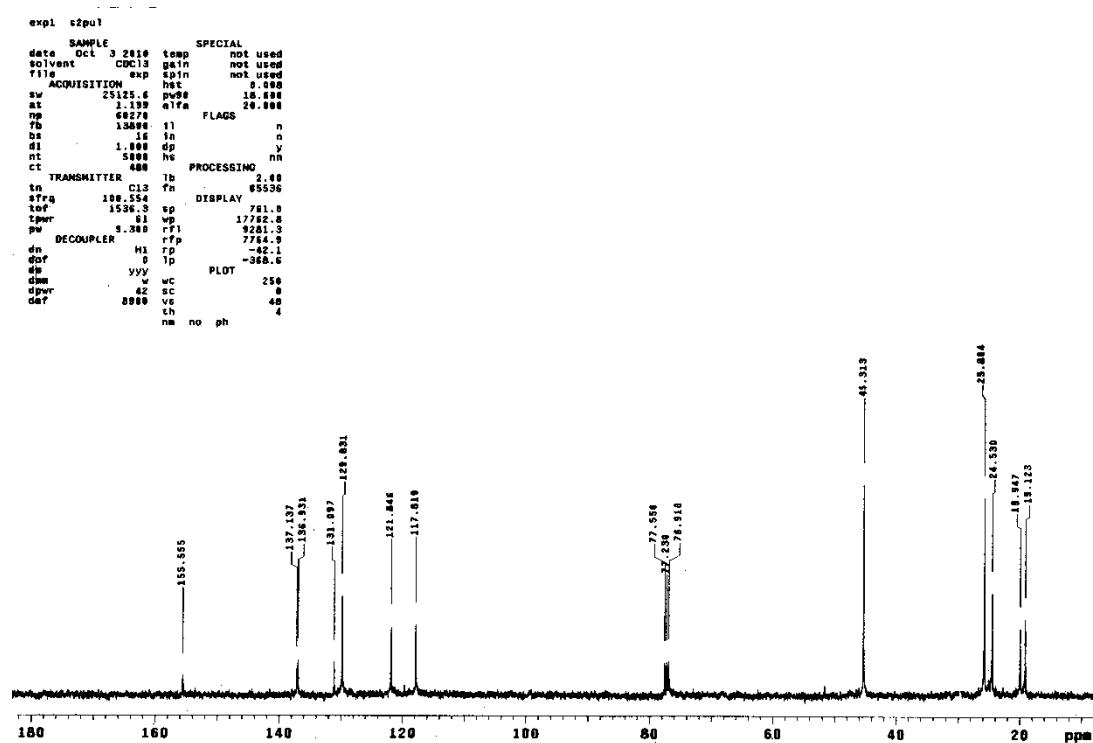
Piperidine-1-carboxylic acid (2,4-dimethyl-phenyl)-amide (21b): IR(KBr)



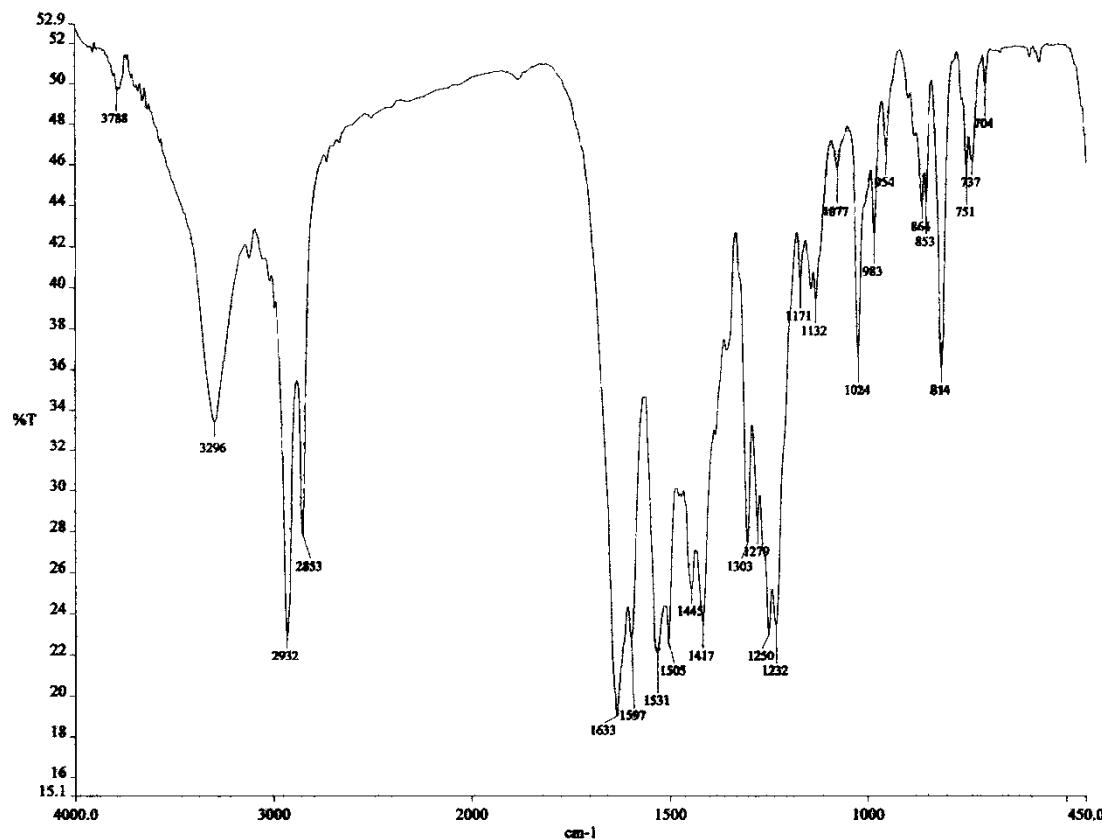
Piperidine-1-carboxylic acid (3,4-dimethyl-phenyl)-amide (26b): <sup>1</sup>H NMR (CDCl<sub>3</sub>, 400 MHz)



Piperidine-1-carboxylic acid (3,4-dimethyl-phenyl)-amide (26b):  $^{13}\text{C}$  NMR ( $\text{CDCl}_3$ , 100 MHz)

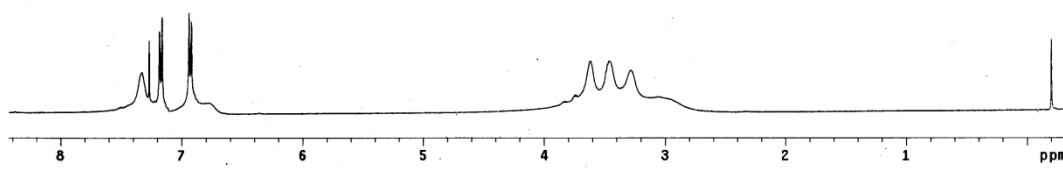
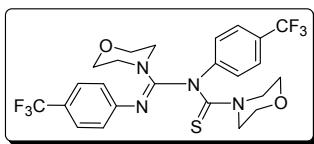


Piperidine-1-carboxylic acid (3,4-dimethyl-phenyl)-amide (26b): IR (KBr)



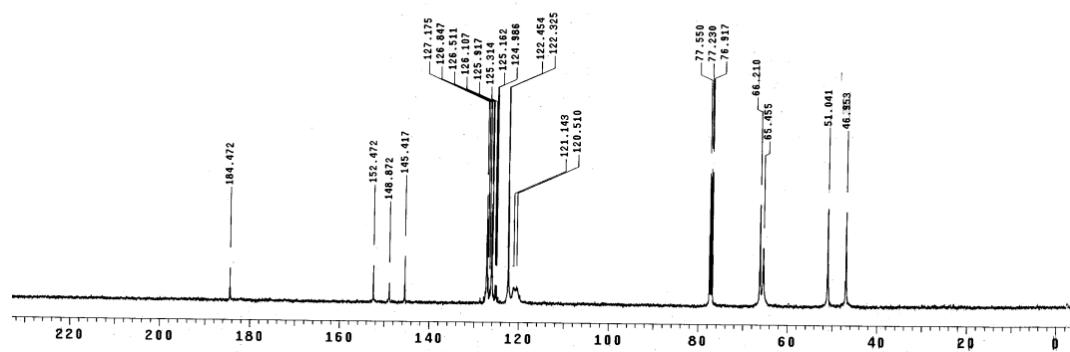
***N-((E)-(4-(Trifluoromethyl)phenylimino)(morpholino)methyl)-N-(4-(trifluoromethyl)phenyl) morpholine-4-carbothioamide (11):  $^1\text{H}$  NMR ( $\text{CDCl}_3$ , 400 MHz):***

```
RY_2_94
exp1 s2pul
SAMPLE SPECIAL
date Apr 30 2009 temp not used
solvent CDCl3 gain not used
file /export/home/~ spin not used
pmtl/RY_2_200_rfid hst 0.008
sw 6389.8 pw0 19.700
et 1.998 alfa 20.000
np 256 dpp 11
fb not used n FLAG0
bs 4 ln n
d1 1.000 dp y
nt 32 hs nn
ct 32 PROCESSING
TRANSMITTER H1 fb 0.10
tn H1 fn 65536
sfrq 399.853 DISPLAY
tof 362.8 sp -146.6
tpwr 27.0 rf 359.0
pw 8.800 rfp 793.3
DECOUPLER C13 rfp 0
dn C13 rp 137.5
dof mnm lp -86.5
dm VVY SC 250
dpwr 50 sc 0
dmt 15900 vs 24
drt th 10
nm cdc ph
```



***N-((E)-(4-(Trifluoromethyl)phenylimino)(morpholino)methyl)-N-(4-(trifluoromethyl)phenyl)morpholine-4-carbothioamide (11):  $^{13}\text{C}$  NMR ( $\text{CDCl}_3$ , 100 MHz):***

```
RY_2_200_13C
exp1 s2pul
SAMPLE SPECIAL
date Aug 12 2009 temp not used
solvent CDCl3 gain not used
file /export/home/~ spin not used
pmtl/RY_2_200_rfid hst 0.008
sw 25125.6 pw0 18.800
ACQUISITION alfa 20.000
et 1.198 b 2.00
np 60270 ln n
fb 13900 dp y
bs 32 hs nn
d1 1.000
dt 1.0000 b 2.00
ct 140000 fn 65536
TRANSMITTER H1 DISPLAY
sfrq 100.554 sp -1503.8
tof 1536.3 rf1 25125.6
tpwr 61 rfp 1504.6
pw 9.300 dp -27.5
DECOUPLER C13 rf 355.4
dn VVY SC 250
dm VVY SC 0
dpwr 42 th 33
dmt 8900 nm no ph
```



***N-((E)-(4-(Trifluoromethyl)phenylimino)(morpholino)methyl)-N-(4-(trifluoromethyl)phenyl)morpholine-4-carbothioamide (11): IR (KBr)***

