

An oxidative cleavage of *anti*-Hugerschhoff 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 F₂₅₄ (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. ¹H NMR (400 MHz) and ¹³C 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*_α radiation ($\lambda = 0.71073 \text{ \AA}$) at 298 K. Cell parameters were retrieved using SMART ¹USA, 1995 software and refined with SAINT¹ 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.² The structures were solved by direct methods implemented in the SHELX-97³ 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

- 1 SMART, SAINT and XPREP, Siemens Analytical X-ray Instruments Inc., Madison, Wisconsin.
- 2 G. M. Sheldrick, *SADABS: Empirical Absorption and Correction Software*, University of Gottingen, Institut für Anorganische Chemie der Universität, Tammanstrasse 4, D-3400 Gottingen, Germany, 1999–2003.
- 3 G. M. Sheldrick, SHELXS-97, University of Gottingen, Germany, 1997.

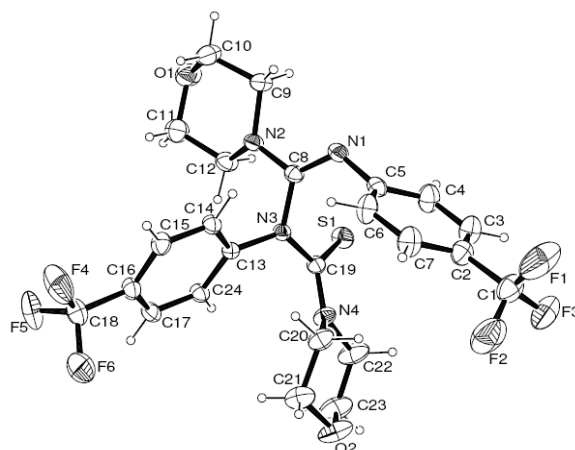


Fig. 2 ORTEP views of *N-((E)-(4-(Trifluoromethyl)phenylimino)(morpholino)methyl)-N-(4-(trifluoromethyl)phenyl)morpholine-4-carbothioamide (11)*

Crystallographic description of *N-((E)-(4-(Trifluoromethyl)phenylimino)(morpholino)methyl)-N-(4-(trifluoromethyl)phenyl)morpholine-4-carbothioamide (11)*

carbothioamide (11): $C_{24}H_{24}F_6N_4O_2S$, crystal dimensions 0.42 x 0.35 x 0.26 mm, $M_r = 546.54$, monoclinic, space group $P2_1/c$, $a = 14.9136(7)$, $b = 9.0945(5)$, $c = 18.6160(9)$ Å, $\alpha = 90.00^\circ$, $\beta = 98.612(2)^\circ$, $\gamma = 90.00^\circ$, $V = 2496.5(2)$ Å³, $Z = 4$, $\rho_{\text{calcd}} = 1.454$ mg/m³, $\mu = 0.204$ mm⁻¹, $F(000) = 1128$, 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.

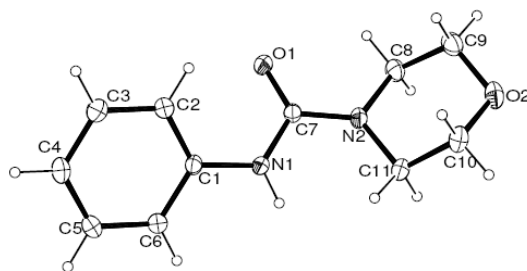


Fig. 3 ORTEP views of Morpholine-4-carboxylic acid phenylamide **14a**.

Crystallographic description of Morpholine-4-carboxylic acid phenylamide (14a): $C_{11}H_{14}N_2O_2$, crystal dimensions 0.40 x 0.32 x 0.24 mm, $M_r = 206.24$, monoclinic, space group P 21/c, $a = 8.0909(3)$, $b = 15.7683(6)$, $c = 8.4586(3)$ Å, $\alpha = 90.00^\circ$, $\beta = 104.174(2)^\circ$, $\gamma = 90.00^\circ$, $V = 1046.29(7)$ Å³, $Z = 4$, $\rho_{\text{calcd}} = 1.309$ mg/m³, $\mu = 0.092$ mm⁻¹, $F(000) = 440$, 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.

Spectral Data

Morpholine-4-carboxylic acid phenylamide (14a): Brown solid; M.p. 154-156 °C; ^1H NMR (CDCl_3 , 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); ^{13}C NMR (CDCl_3 , 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^{-1} ; Elemental analysis for $\text{C}_{11}\text{H}_{14}\text{N}_2\text{O}_2$ (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; ^1H NMR ($\text{CDCl}_3 + \text{DMSO}-d_6$, 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); ^{13}C NMR ($\text{CDCl}_3 + \text{DMSO}-d_6$, 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^{-1} ; $\text{C}_{12}\text{H}_{16}\text{N}_2\text{O}$ (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; ^1H NMR (CDCl_3 , 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); ^{13}C NMR (CDCl_3 , 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^{-1} ; $\text{C}_{12}\text{H}_{16}\text{N}_2\text{O}_2$ (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; ^1H NMR (CDCl_3 , 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); ^{13}C NMR (CDCl_3 , 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) 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; ^1H NMR (CDCl_3 , 400 MHz) δ (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}C NMR (100 MHz, CDCl_3) δ 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; ^1H NMR (CDCl_3 , 400 MHz) δ (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, $J = 8.4$ Hz); ^{13}C NMR (CDCl_3 , 100 MHz) δ (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) 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; ^1H NMR (CDCl_3 , 400 MHz) δ (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}C NMR (CDCl_3 , 100 MHz) δ (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) 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; ^1H NMR (CDCl_3 , 400 MHz) δ (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}C NMR (CDCl_3 , 100 MHz) δ (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) 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; ^1H NMR (CDCl_3 , 400 MHz) δ (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}C NMR (CDCl_3 , 100 MHz) δ (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) 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 $^\circ\text{C}$; ^1H NMR (CDCl_3 , 400 MHz) δ (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}C NMR (CDCl_3 , 100 MHz) δ (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) 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; ^1H NMR (CDCl_3 , 400 MHz) δ (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}C NMR (CDCl_3 , 100 MHz) δ (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) 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; ^1H NMR (CDCl_3 , 400 MHz) δ (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}C NMR (CDCl_3 , 100 MHz) δ (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) 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; ^1H NMR (CDCl_3 , 400 MHz) δ (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}C NMR (CDCl_3 , 100 MHz) δ (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) 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; ^1H NMR (CDCl_3 , 400 MHz) δ (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}C NMR (CDCl_3 , 100 MHz) δ (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) 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; ^1H NMR (CDCl_3 , 400 MHz) δ (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}C NMR (CDCl_3 , 100 MHz) δ (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) 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; ^1H NMR (CDCl_3 , 400 MHz) δ (ppm) 1.52 (m, 6H), 3.31 (m, 4H), 4.36 (s, 2H), 4.8 (brs, 1H), 7.22-7.27 (m, 5H); ^{13}C NMR (CDCl_3 , 100 MHz) δ (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) 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; ^1H NMR (CDCl_3 , 400 MHz) δ (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}C NMR (CDCl_3 , 100 MHz) δ (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) 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; ^1H NMR (CDCl_3 , 400 MHz) δ (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}C NMR (CDCl_3 , 100 MHz) δ (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) 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; ^1H NMR (CDCl_3 , 400 MHz) δ (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}C NMR (CDCl_3 , 100 MHz) δ (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) 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; ^1H NMR (CDCl_3 , 400 MHz) δ (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}C NMR (CDCl_3 , 100 MHz) δ (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) 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; ^1H NMR (CDCl_3 , 400 MHz) δ (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}C NMR (CDCl_3 , 100 MHz) δ (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) 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; ^1H NMR (CDCl_3 , 400 MHz) δ (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}C NMR (CDCl_3 , 100 MHz) δ (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) 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; ^1H NMR (CDCl_3 , 400 MHz) δ (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}C NMR (CDCl_3 , 100 MHz) δ (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) 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; ^1H NMR (CDCl_3 , 400 MHz) δ (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}C NMR (CDCl_3 , 100 MHz) δ (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) 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; ^1H NMR (CDCl_3 , 400 MHz) δ (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}C NMR (CDCl_3 , 100 MHz) δ (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) 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; ^1H NMR (CDCl_3 , 400 MHz) δ (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}C NMR (CDCl_3 , 100 MHz) δ (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) 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; ^1H NMR (CDCl_3 , 400 MHz) δ (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}C NMR (CDCl_3 , 100 MHz) δ (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) 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.

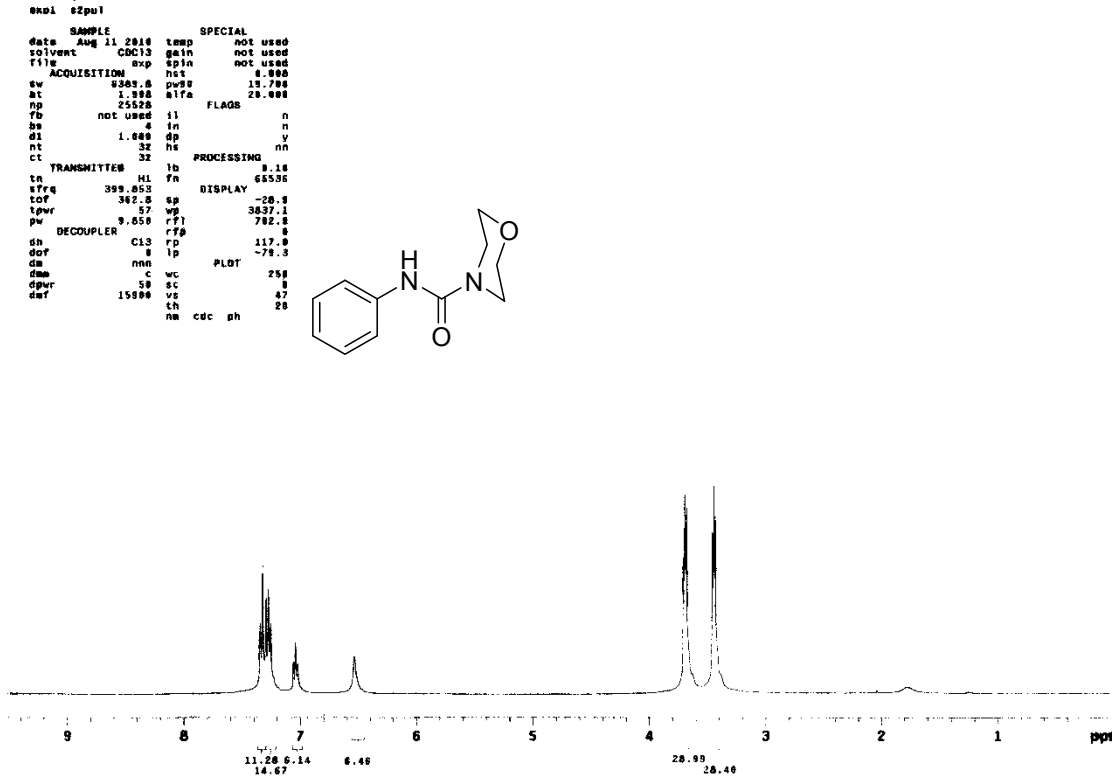
Piperidine-1-carboxylic acid (3,4-dimethyl-phenyl)-amide (26b): White Solid; M.p. 103-105 °C; ^1H NMR (CDCl_3 , 400 MHz) δ (ppm) 1.55-1.56 (m, 6H), 2.15 (s, 3H), 2.17 (s, 3H), 3.38-3.39 (m, 4H), 6.51 (s, 1H), 6.96 (d, 1H, $J = 8$ Hz), 7.02 (d, 1H, $J = 7.6$ Hz), 7.13 (s, 1H); ^{13}C NMR (CDCl_3 , 100 MHz) δ (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) 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; ^1H NMR (CDCl_3 , 400 MHz) δ (ppm) 2.92–3.95 (m, 16H), 6.90 (brs, 2H),

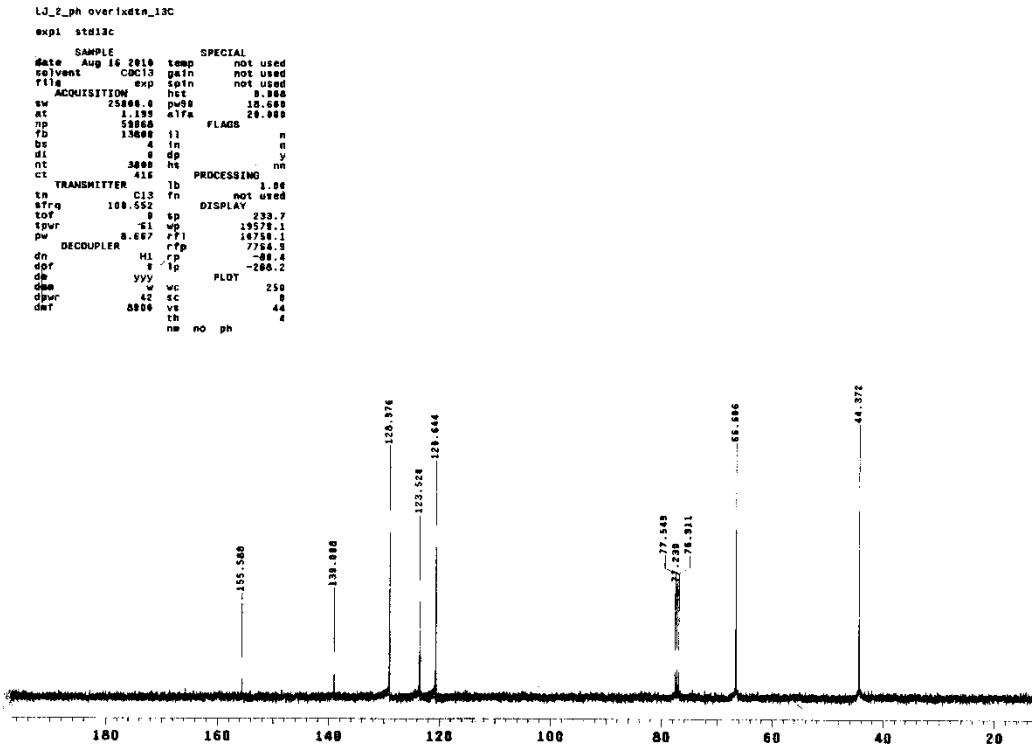
7.07 (d, 2H, $J = 8.0$ Hz), 7.47 (d, 2H, $J = 8.0$ Hz), 7.62 (brs, 2H); ^{13}C NMR (CDCl_3 , 100 MHz) δ (ppm) 46.9, 51.0, 65.4, 66.2, 120.5, 121.1, 122.3, 122.4, 124.9, 125.2, 125.3, 125.9, 126.1, 126.5, 126.8, 127.2, 145.4, 148.9, 152.5, 184.5; IR (KBr): 3395 (w), 2966 (w), 2906 (w), 2858 (w), 1639 (0, 1602 (s), 1424 (m), 1323 (s), 1292 (s), 1236 (s), 1159 (s), 1114 (s), 1064 (s), 1013 (m), 999 (m), 844 (m) 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

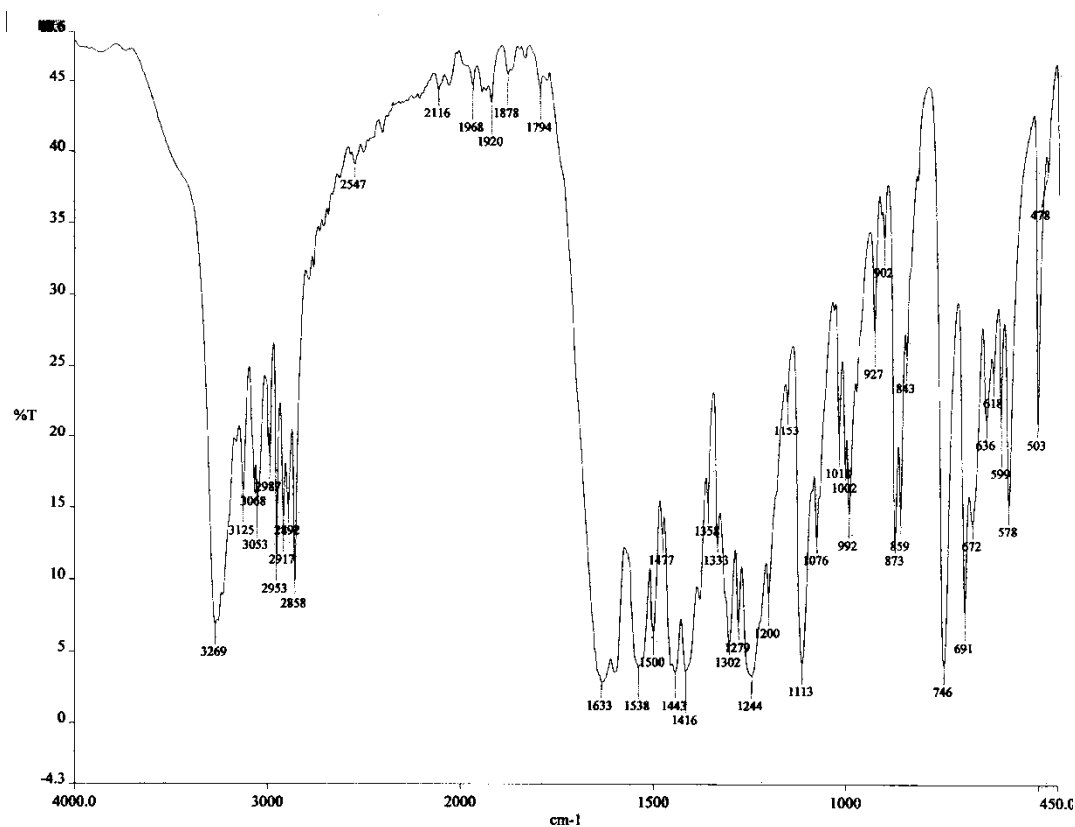
Morpholine-4-carboxylic acid phenylamide (14a): ¹H NMR (CDCl₃, 400 MHz)



Morpholine-4-carboxylic acid phenylamide (14a): ¹³C NMR (CDCl₃, 100 MHz)



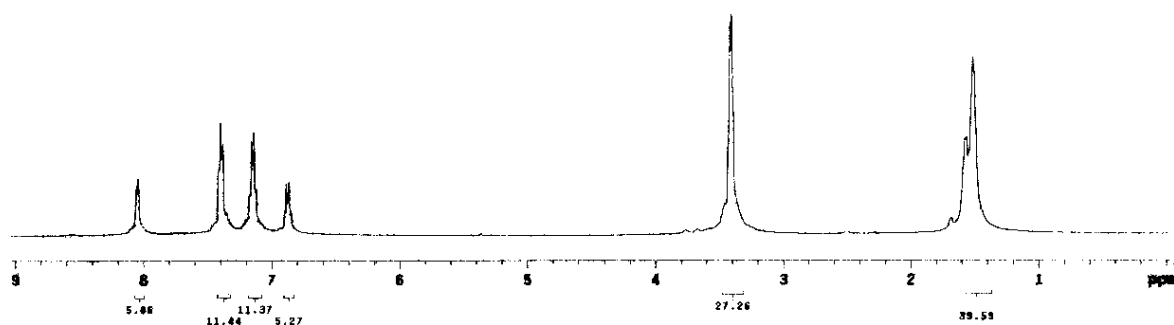
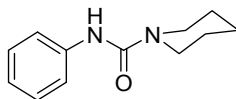
Morpholine-4-carboxylic acid phenylamide (14a): IR (KBr)



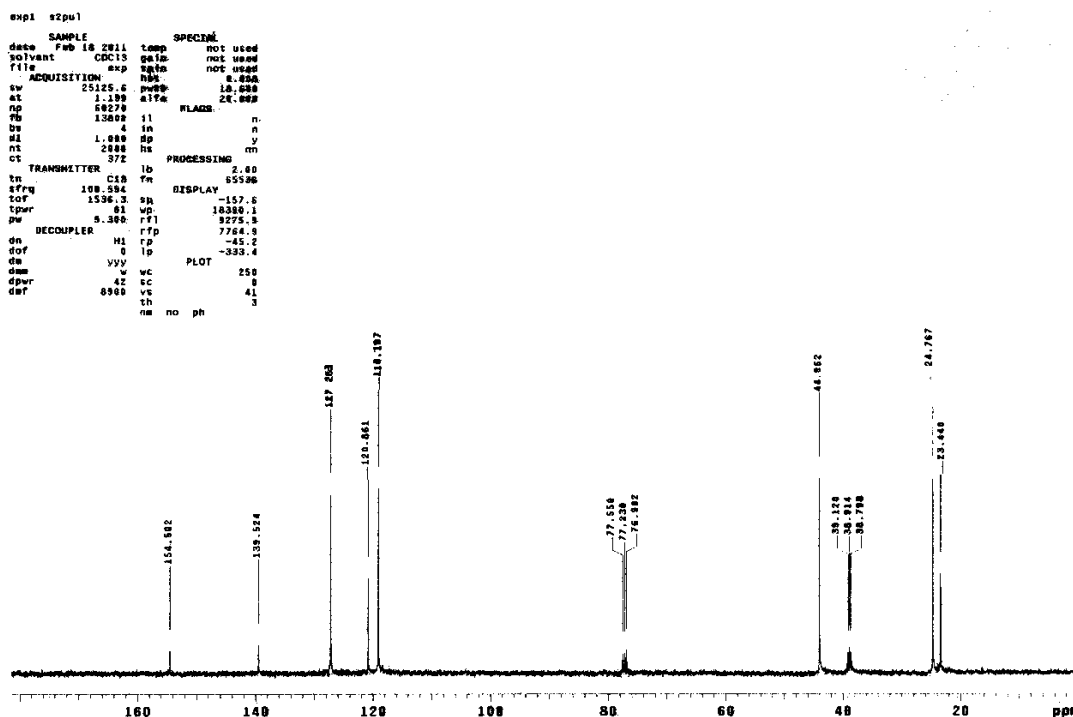
Piperidine-1-carboxylic acid phenylamide (14b): ^1H NMR ($\text{CDCl}_3 + \text{DMSO}-d_6$, 400 MHz)

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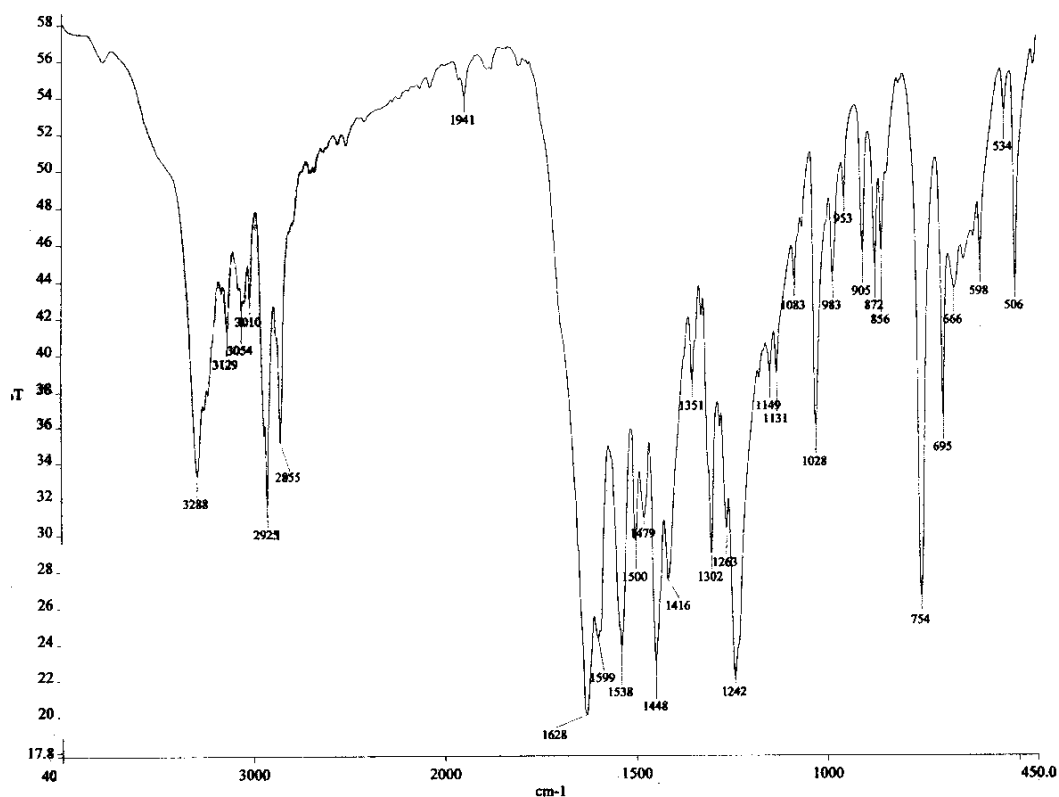
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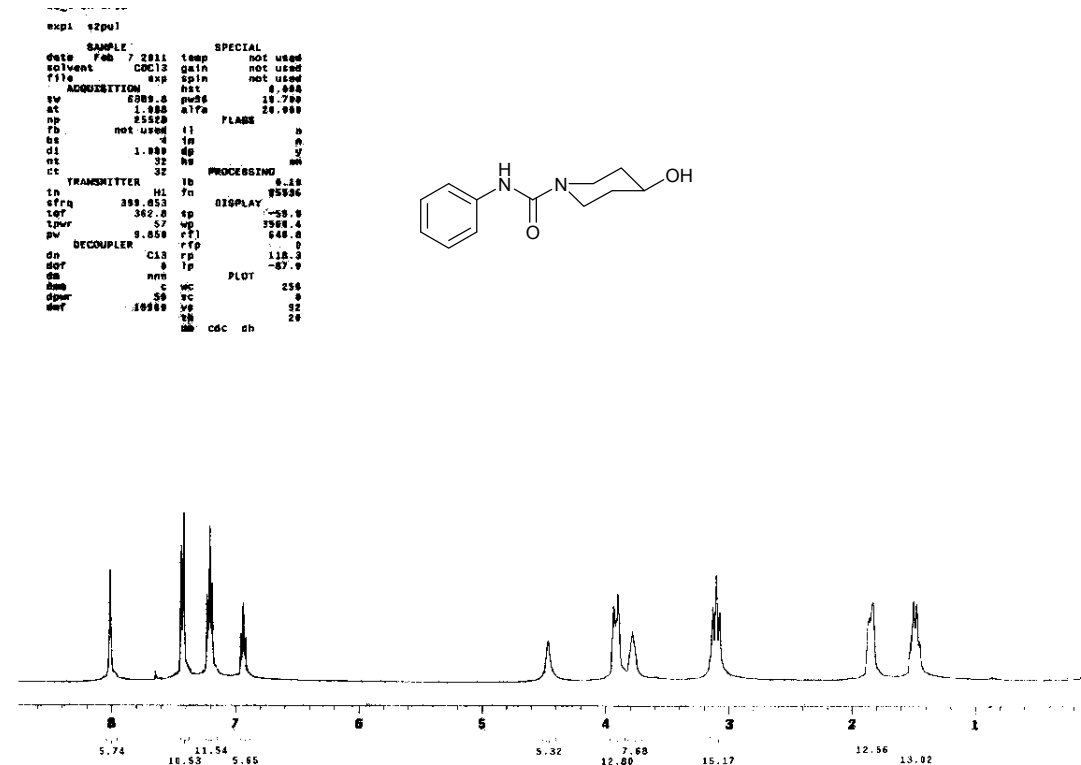
Piperidine-1-carboxylic acid phenylamide (14b): ^{13}C NMR ($\text{CDCl}_3 + \text{DMSO}-d_6$, 100 MHz)



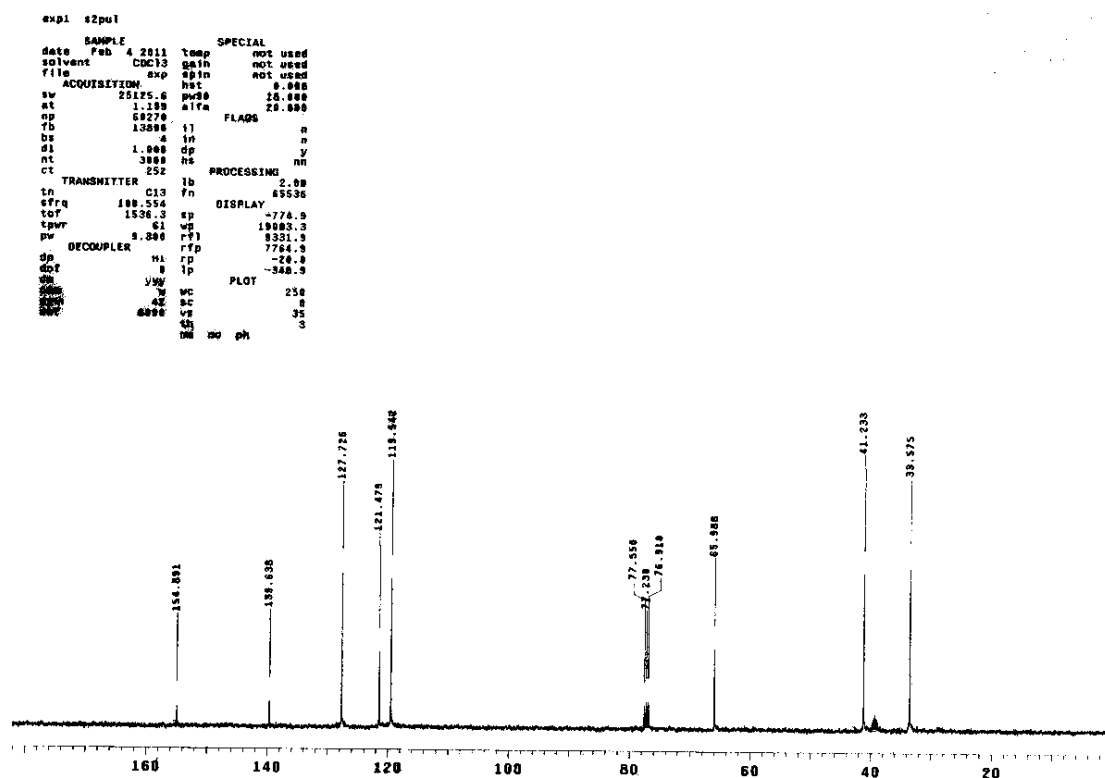
Piperidine-1-carboxylic acid phenylamide (14b): IR (KBr)



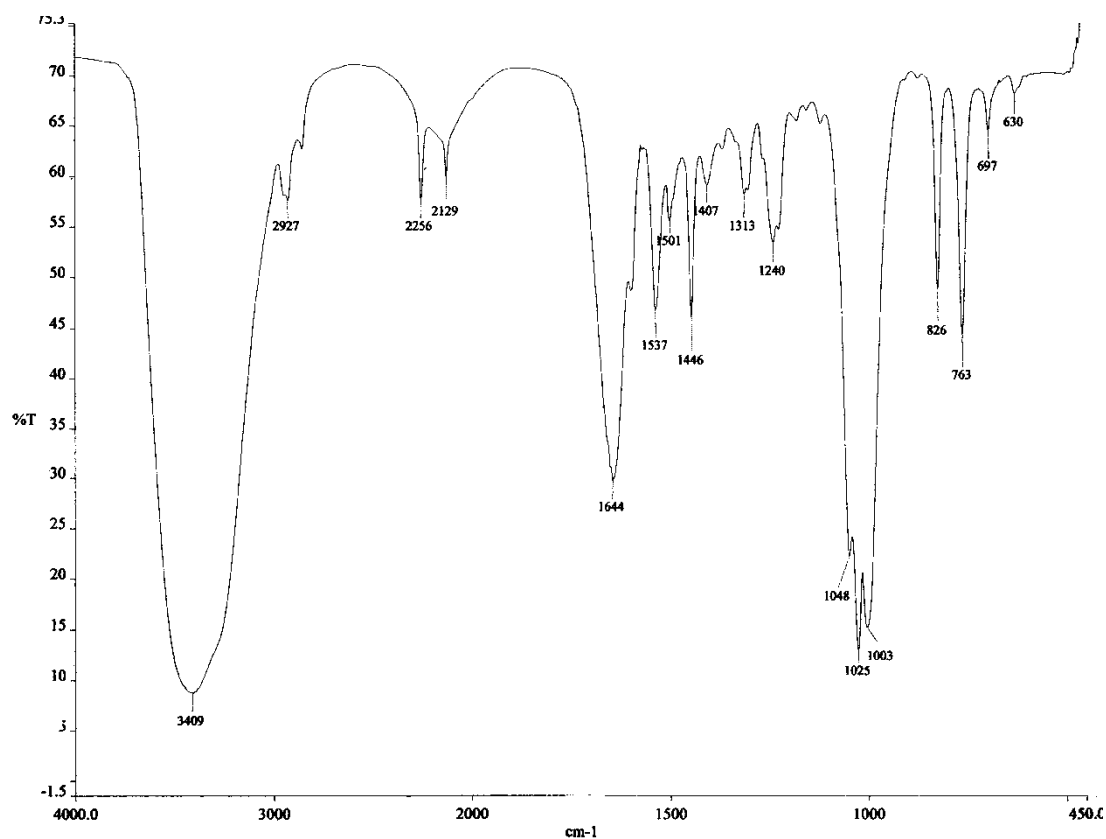
4-Hydroxy-piperidine-1-carboxylic acid phenylamide (14c): ^1H NMR (CDCl_3 , 400 MHz)



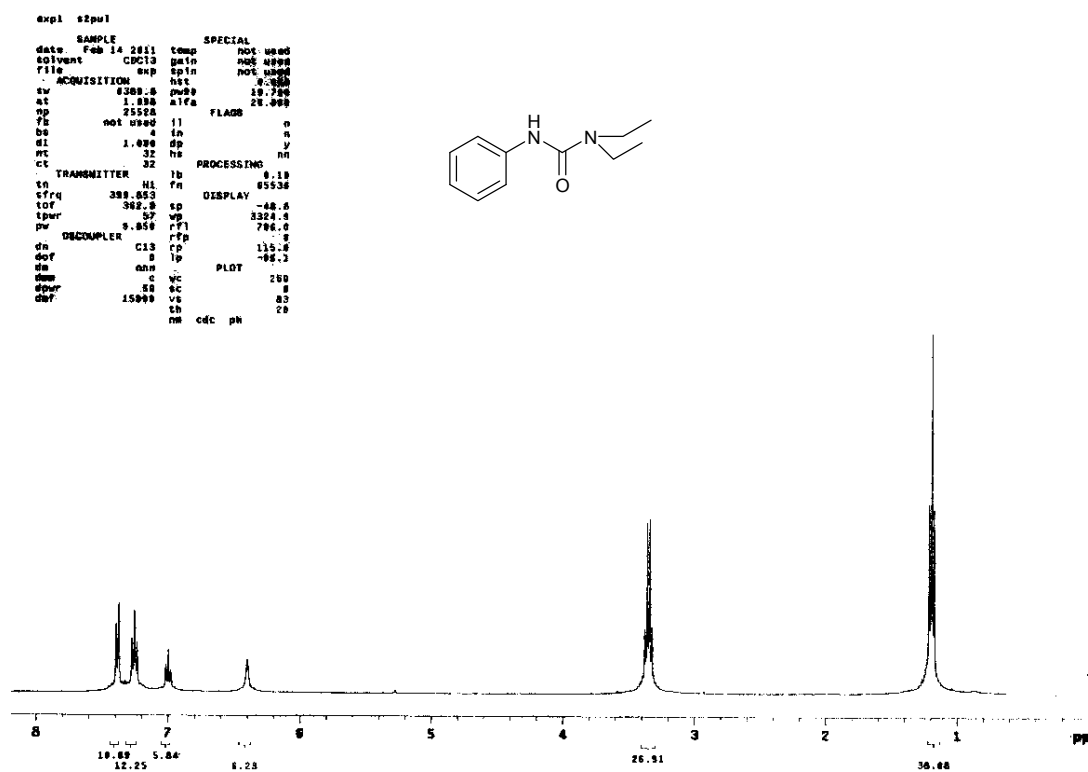
4-Hydroxy-piperidine-1-carboxylic acid phenylamide (14c): ^{13}C NMR (CDCl_3 , 100 MHz)



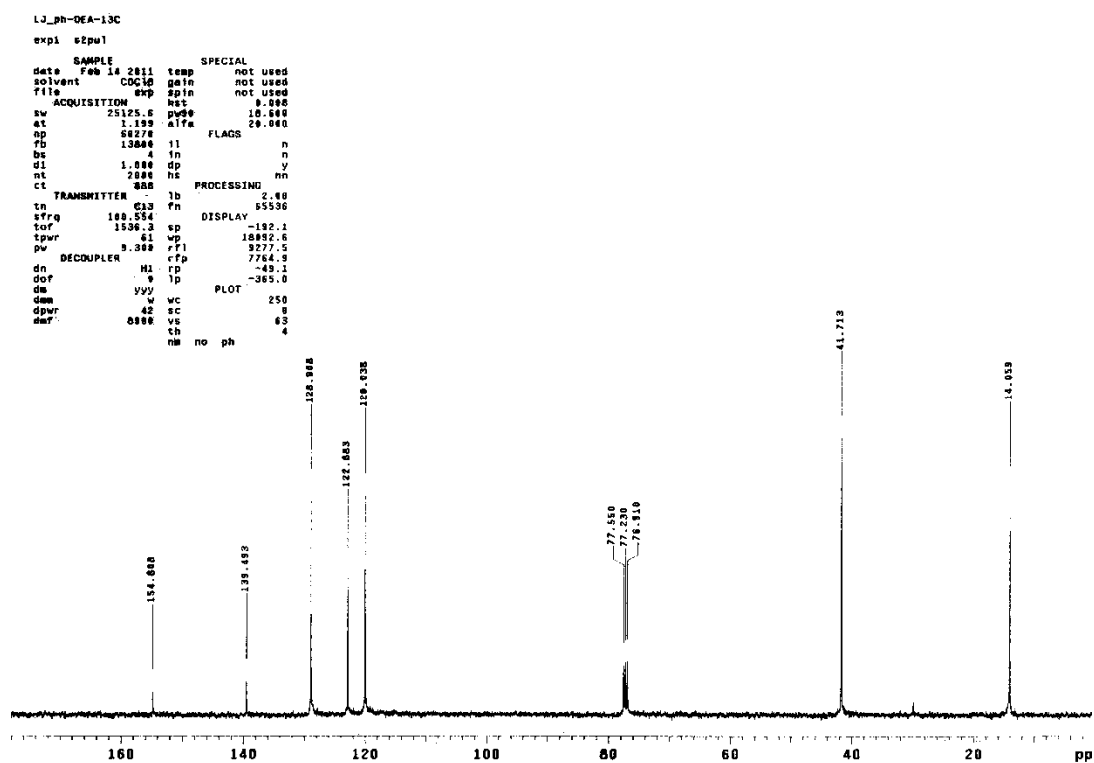
4-Hydroxy-piperidine-1-carboxylic acid phenylamide (14c): IR (KBr)



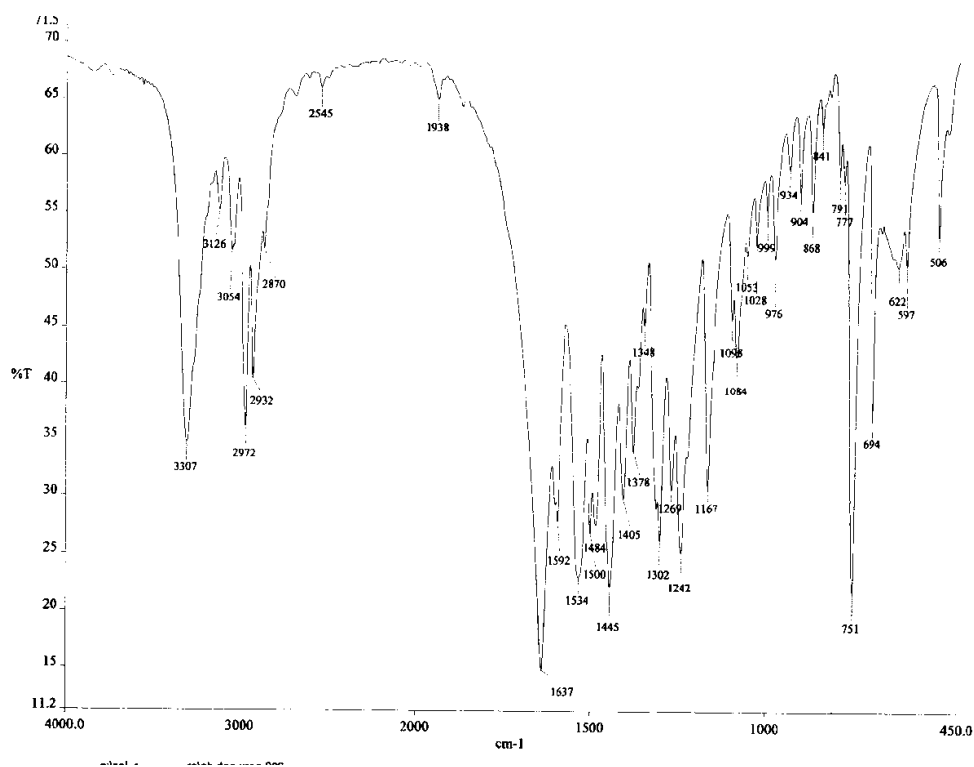
1,1-Diethyl-3-phenyl-urea (14d): ¹H NMR (CDCl₃, 400 MHz)



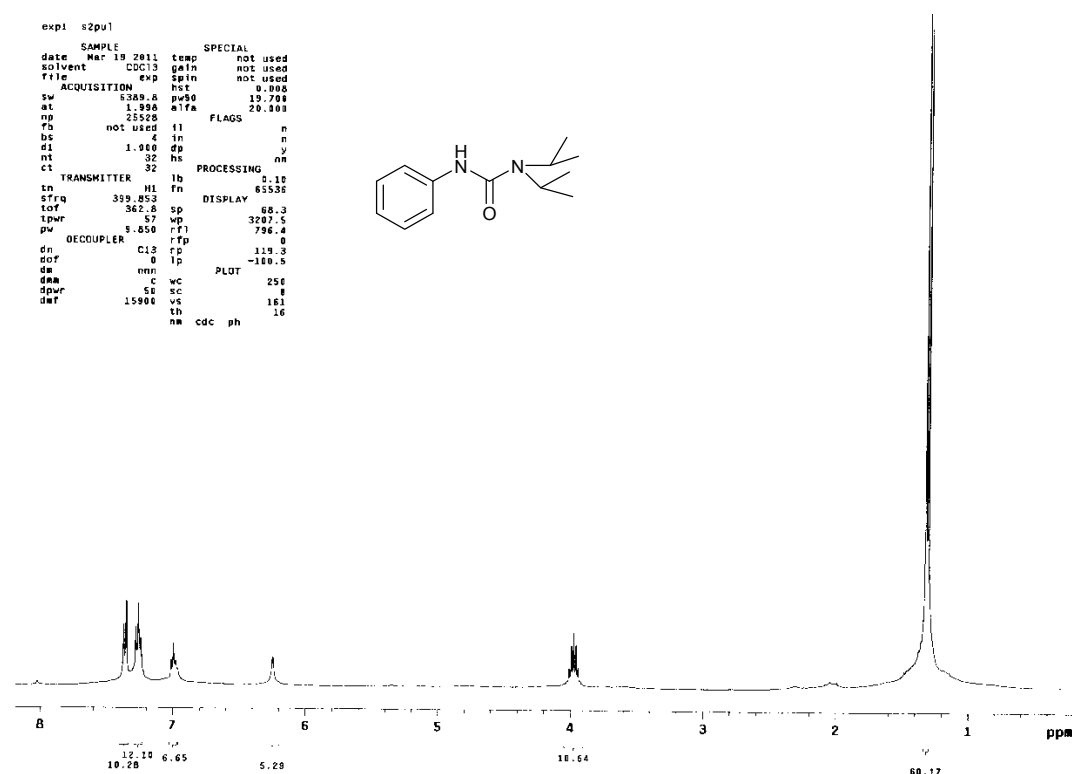
1,1-Diethyl-3-phenyl-urea (14d): ^{13}C NMR (CDCl_3 , 100 MHz)



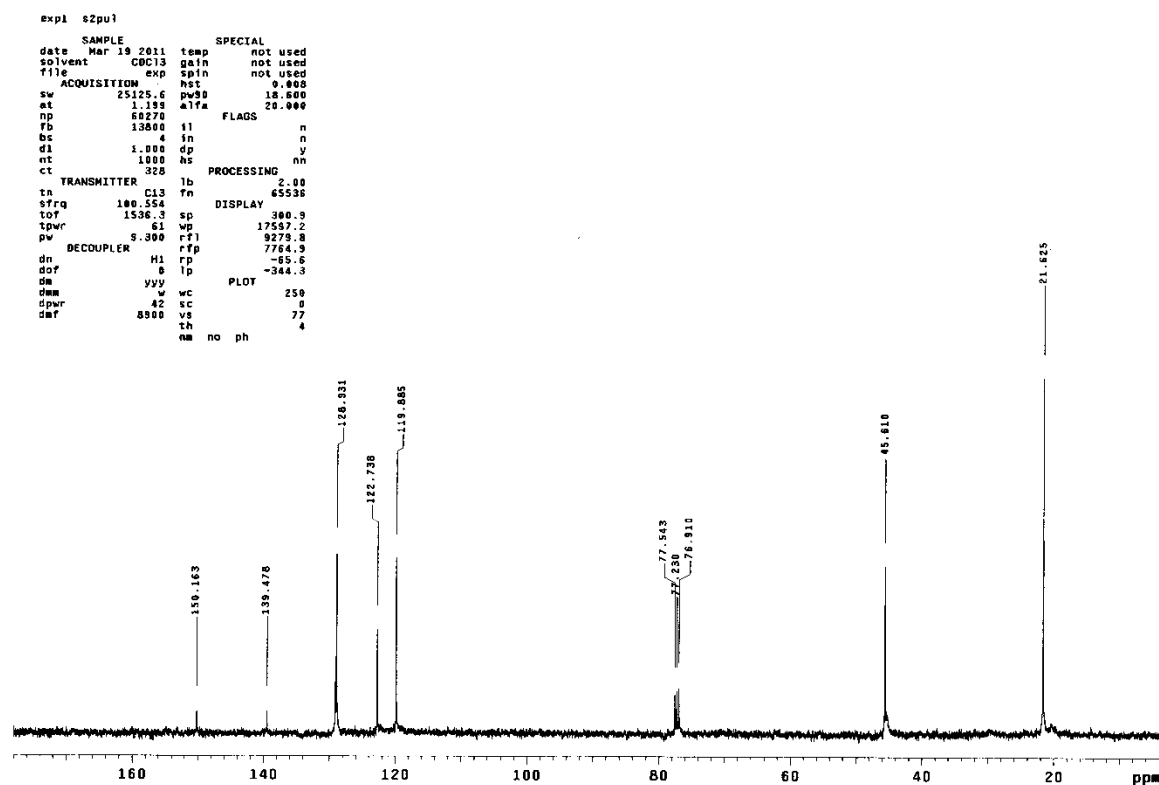
1,1-Diethyl-3-phenyl-urea (14d): IR (KBr)



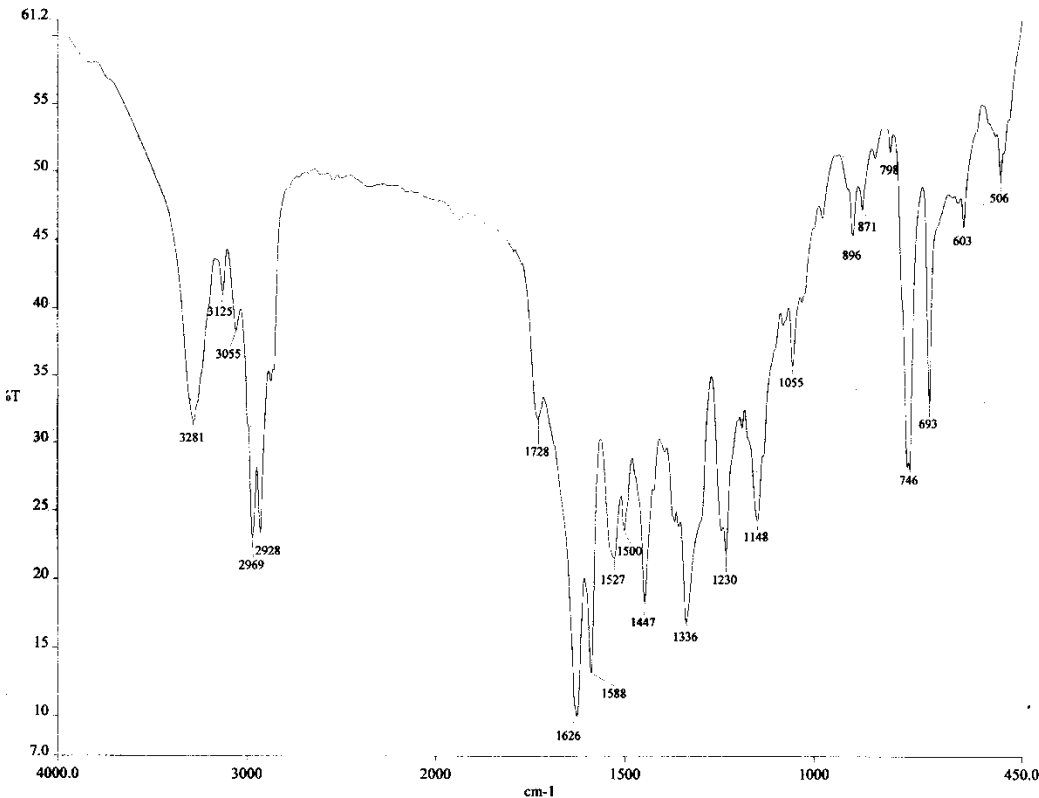
1,1-Diisopropyl-3-phenyl-urea (14e): ¹H NMR (CDCl₃, 400 MHz)



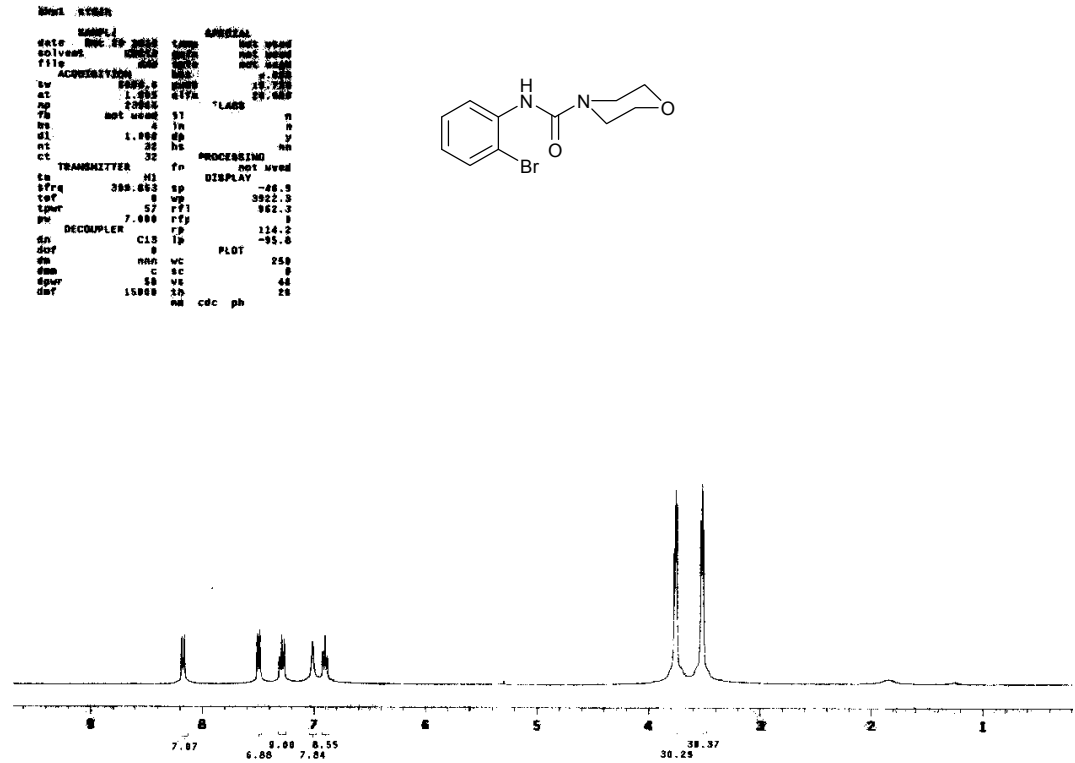
1,1-Diisopropyl-3-phenyl-urea (14e): ¹³C NMR (CDCl₃, 100 MHz)



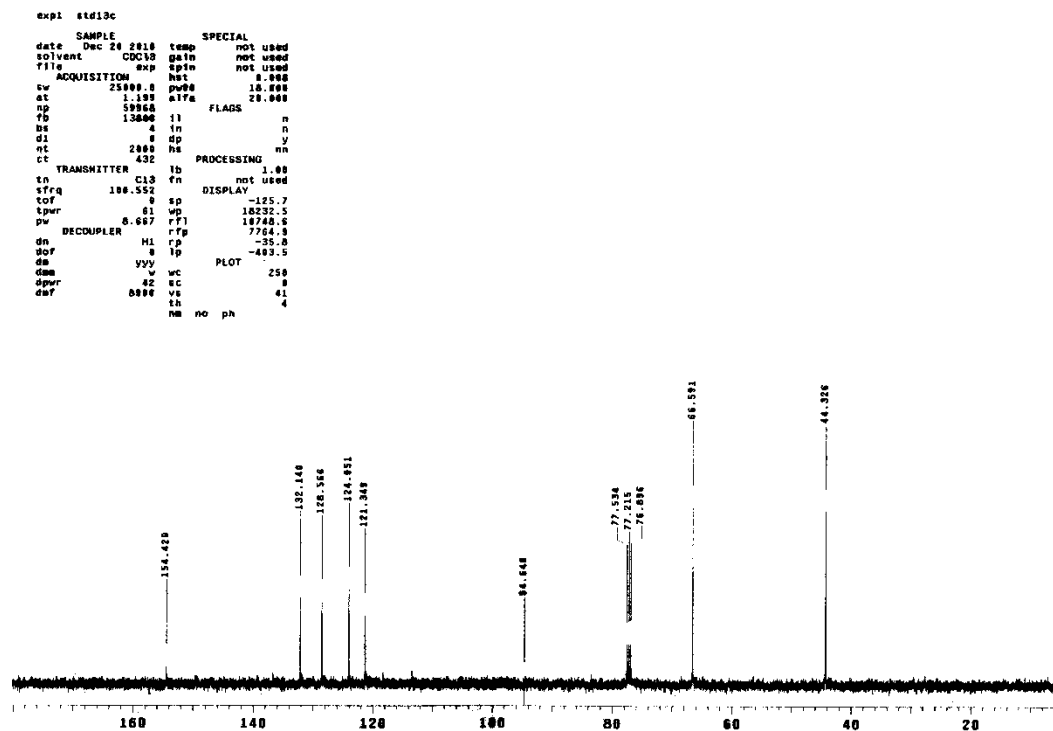
1,1-Diisopropyl-3-phenyl-urea (14e): IR (KBr)



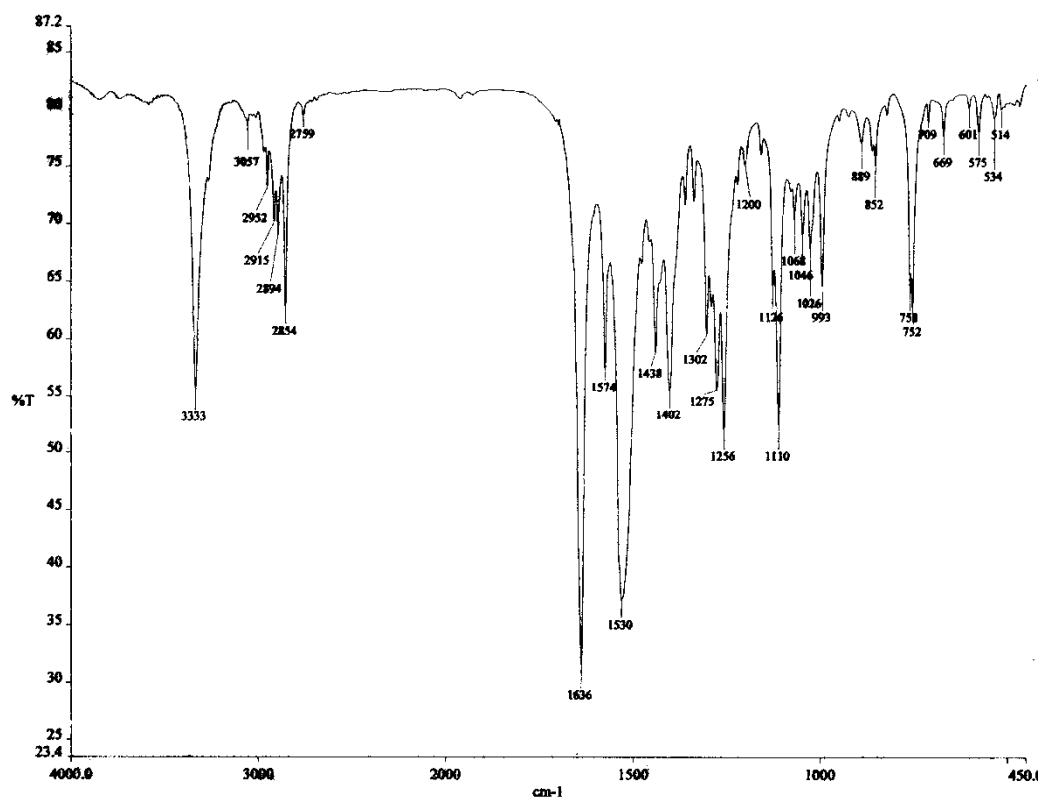
Morpholine-4-carboxylic acid (2-bromo-phenyl)-amide (15a): ¹H NMR (CDCl₃, 400 MHz)



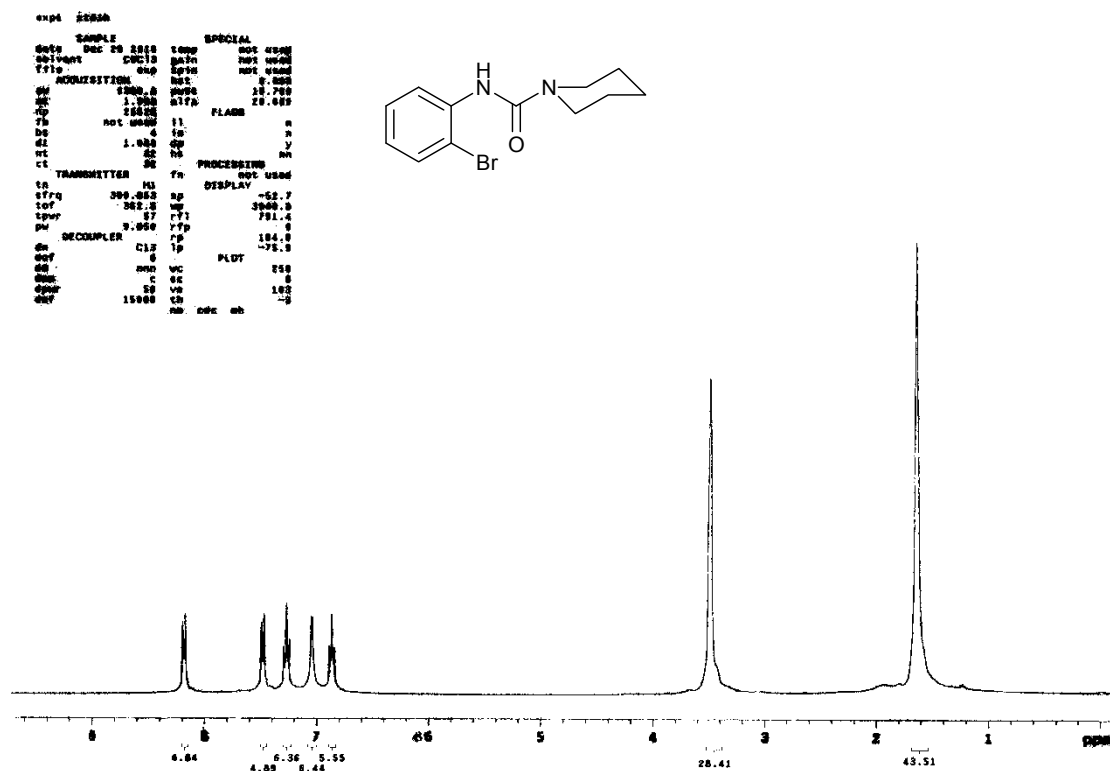
Morpholine-4-carboxylic acid (2-bromo-phenyl)-amide (15a): ^{13}C NMR (CDCl_3 , 100 MHz)



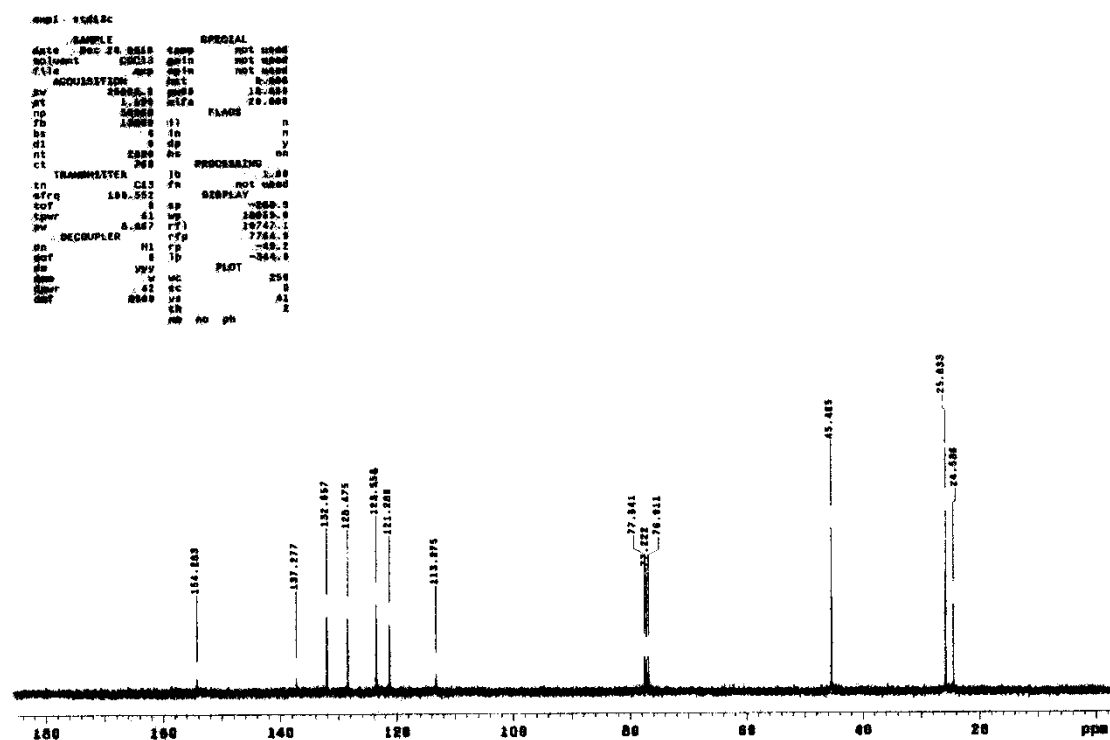
Morpholine-4-carboxylic acid (2-bromo-phenyl)-amide (15a): IR (KBr)



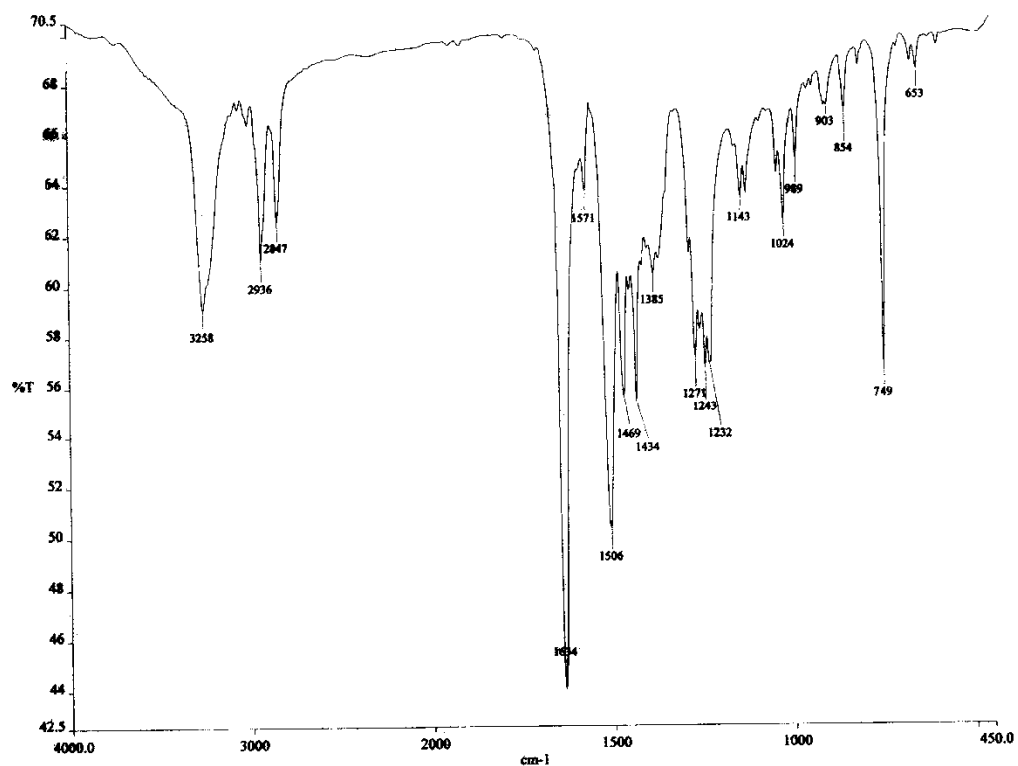
Piperidine-1-carboxylic acid (2-bromo-phenyl)-amide (15b): ^1H NMR (CDCl_3 , 400 MHz)



Piperidine-1-carboxylic acid (2-bromo-phenyl)-amide (15b): ^{13}C NMR (CDCl_3 , 100 MHz)



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

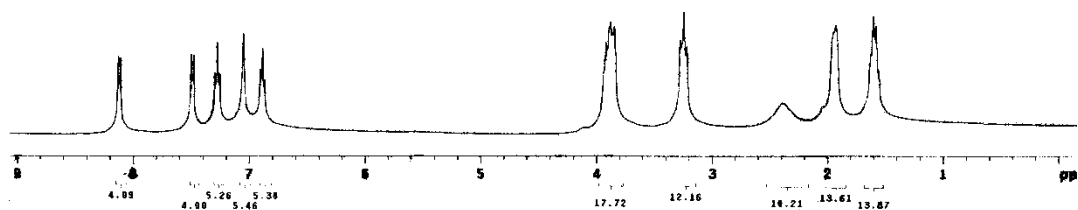
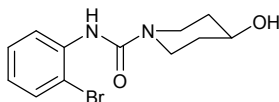


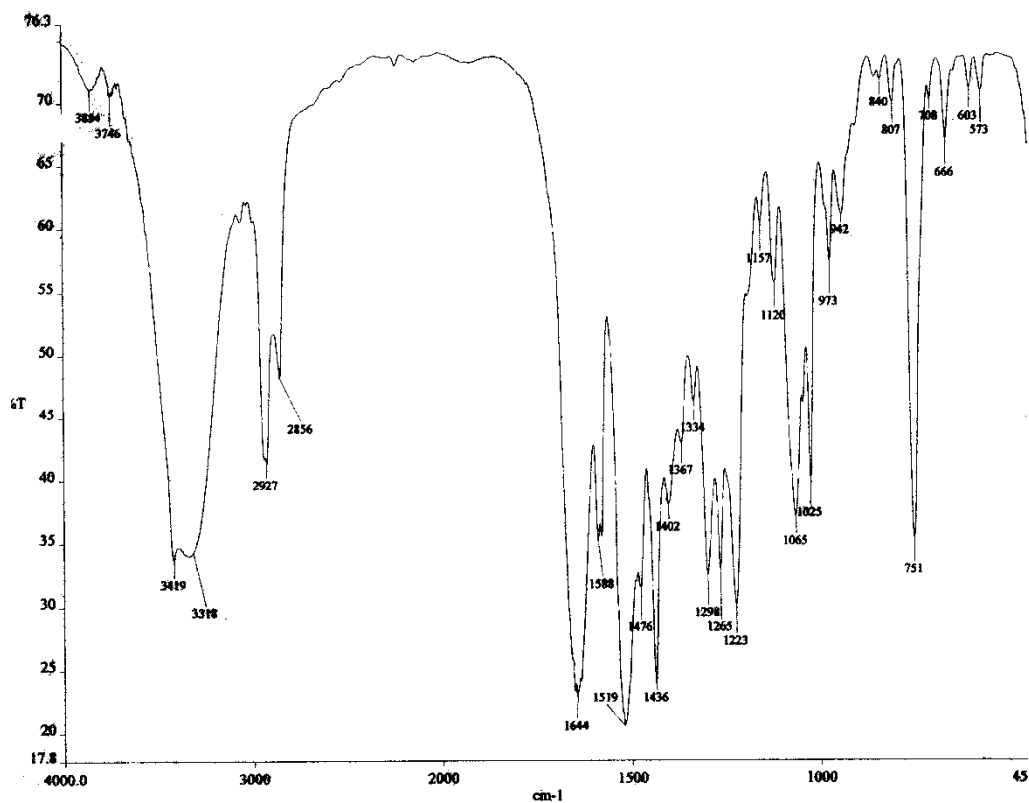
4-Hydroxy-piperidine-1-carboxylic acid (2-bromo-phenyl)-amide (15c): ¹H NMR (CDCl₃, 400 MHz)

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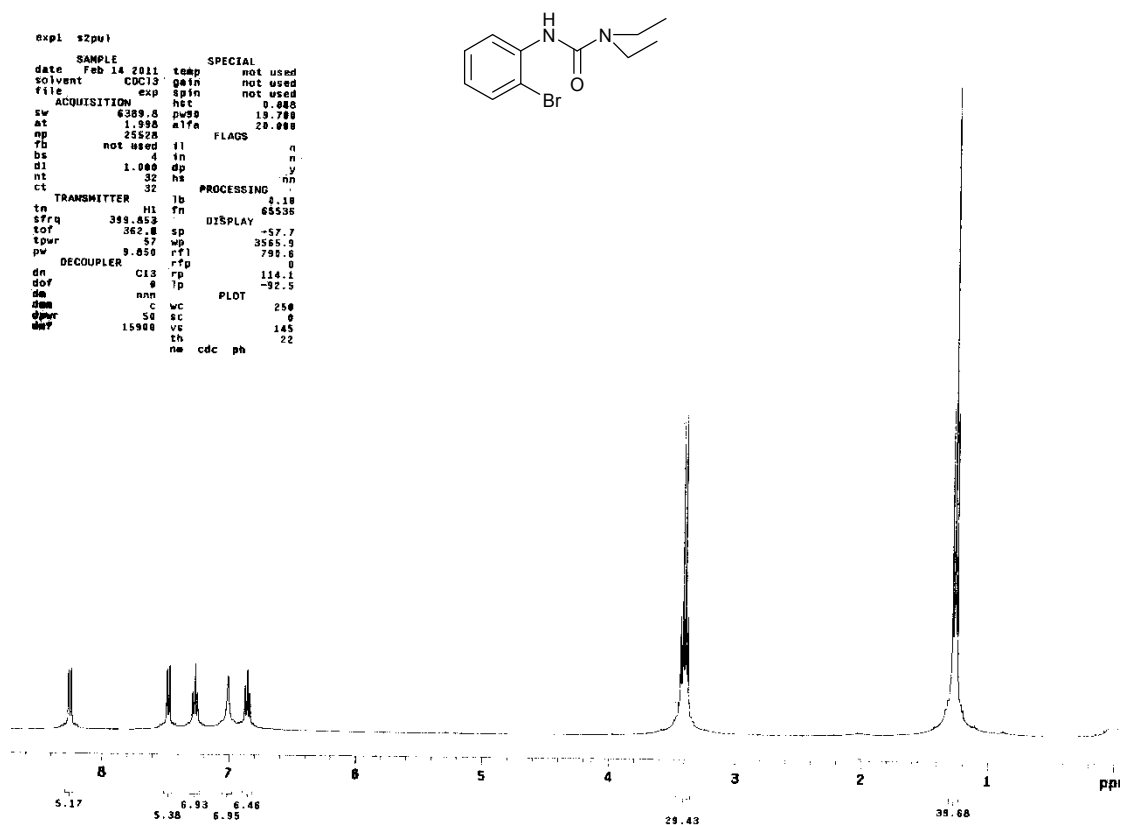
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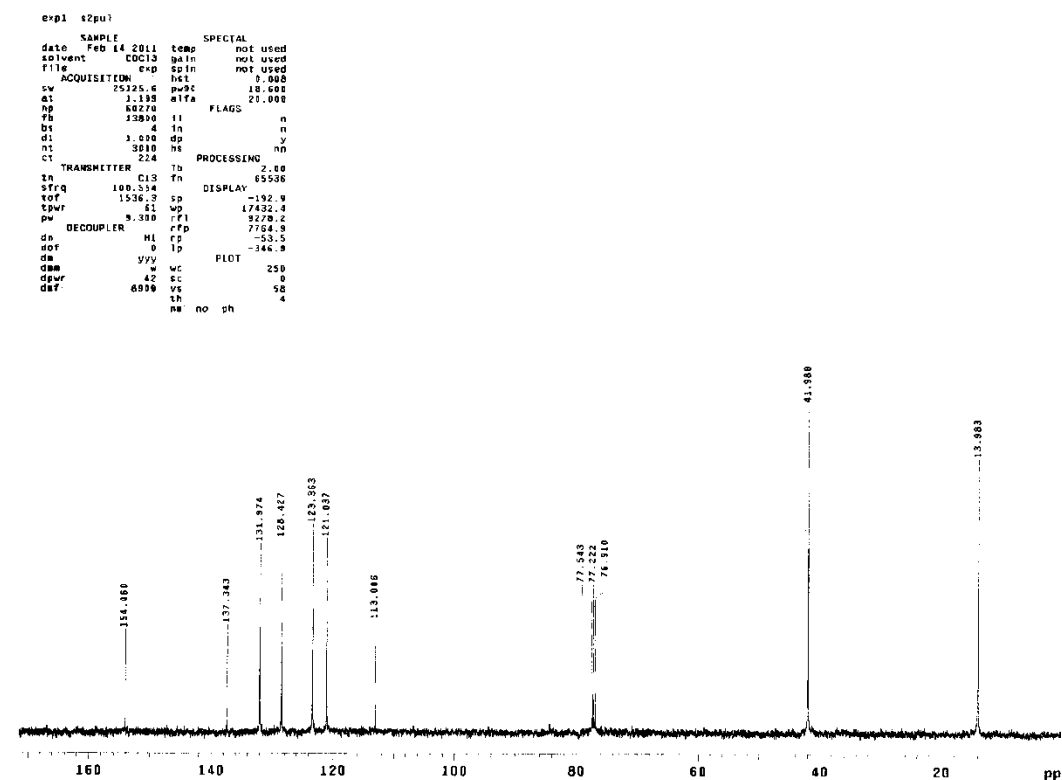


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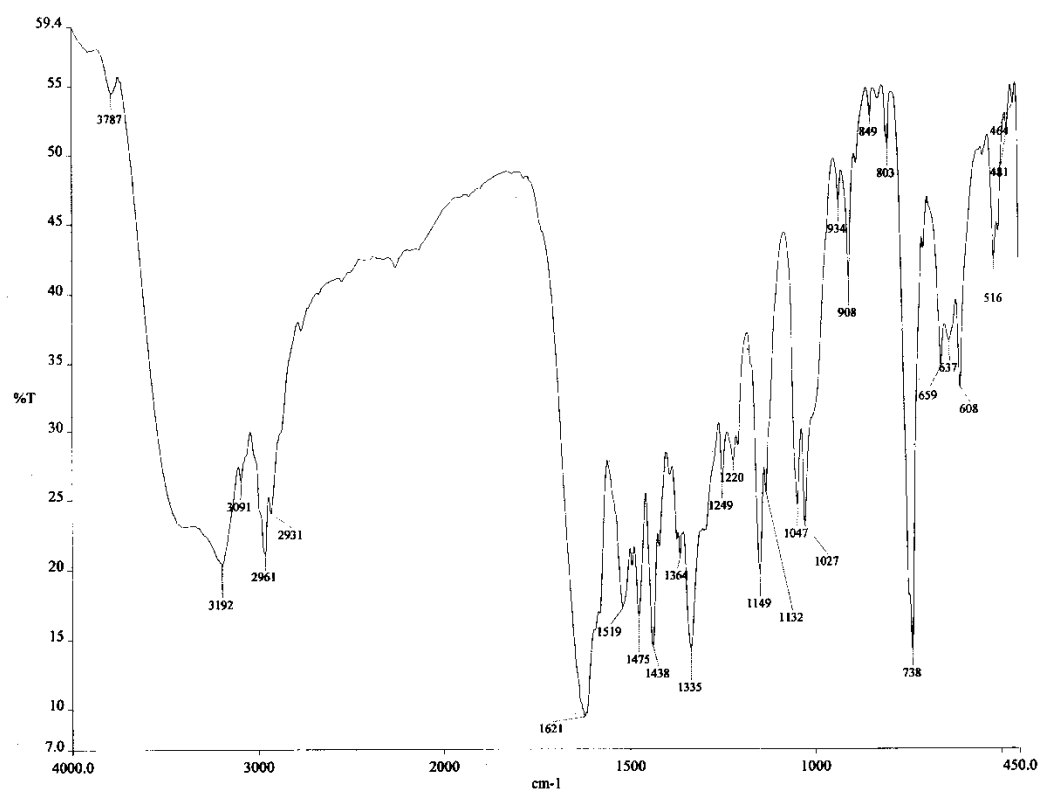
3-(2-Bromo-phenyl)-1,1-diethyl-urea (15d): ^1H NMR (CDCl_3 , 400 MHz)



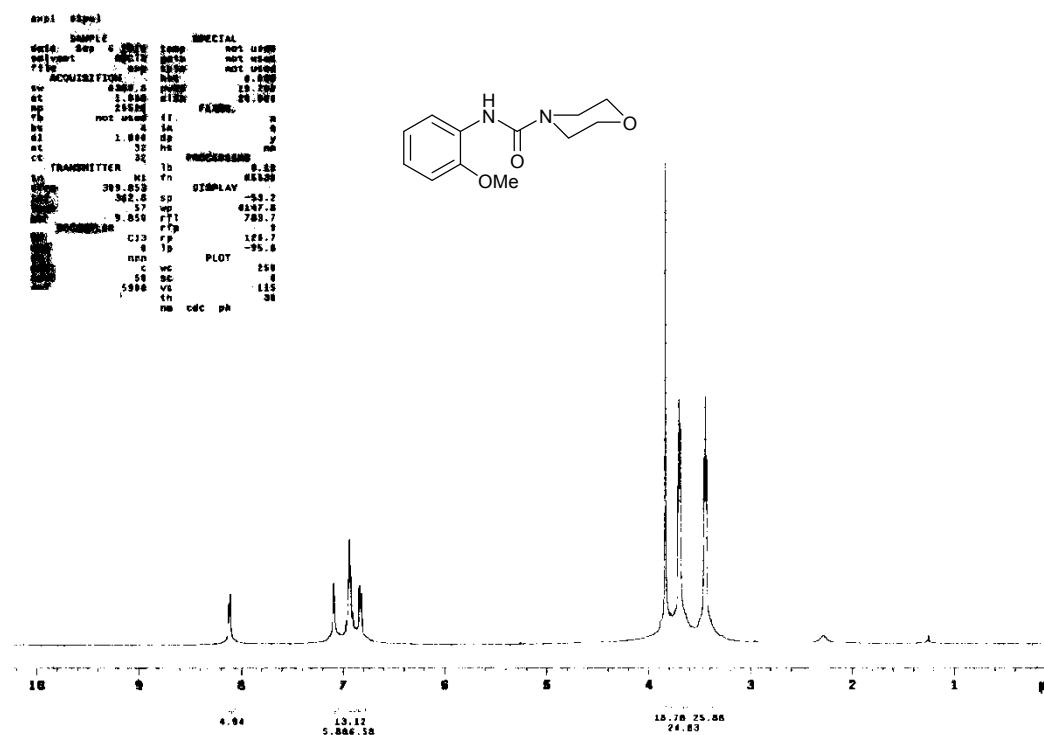
3-(2-bromo-phenyl)-1,1-diethyl-urea (15d): ^{13}C NMR (100 MHz, CDCl_3)



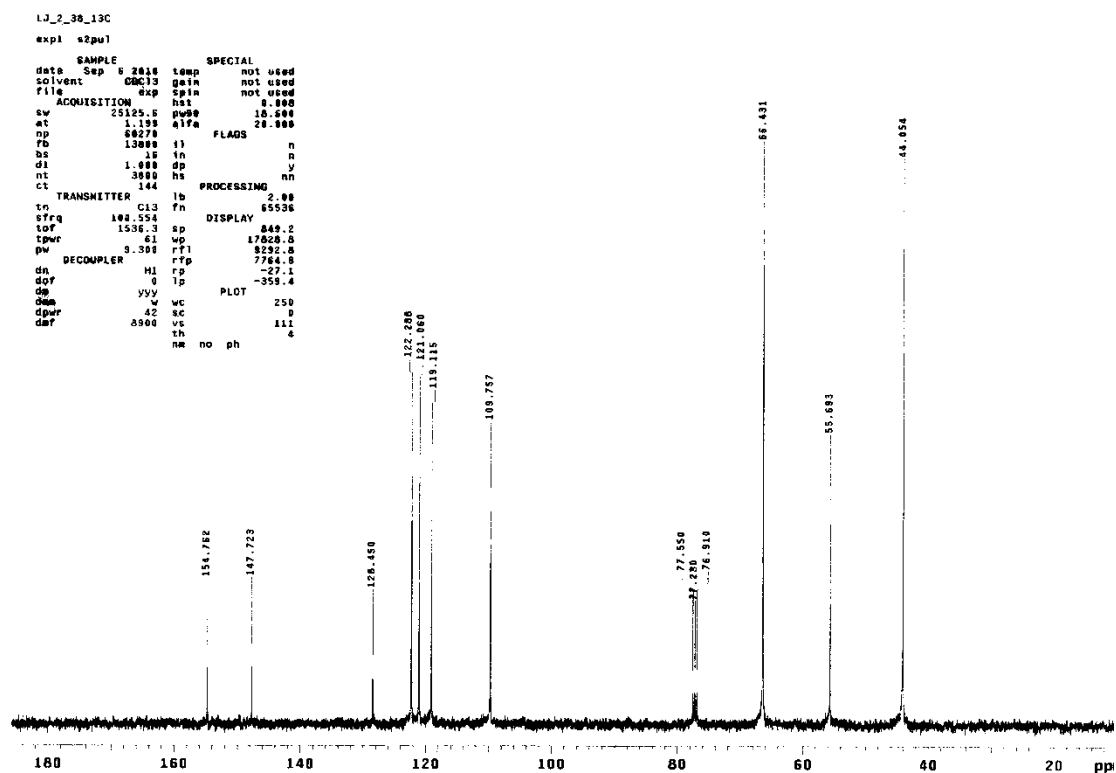
3-(2-Bromo-phenyl)-1,1-diethyl-urea (15d): IR (KBr)



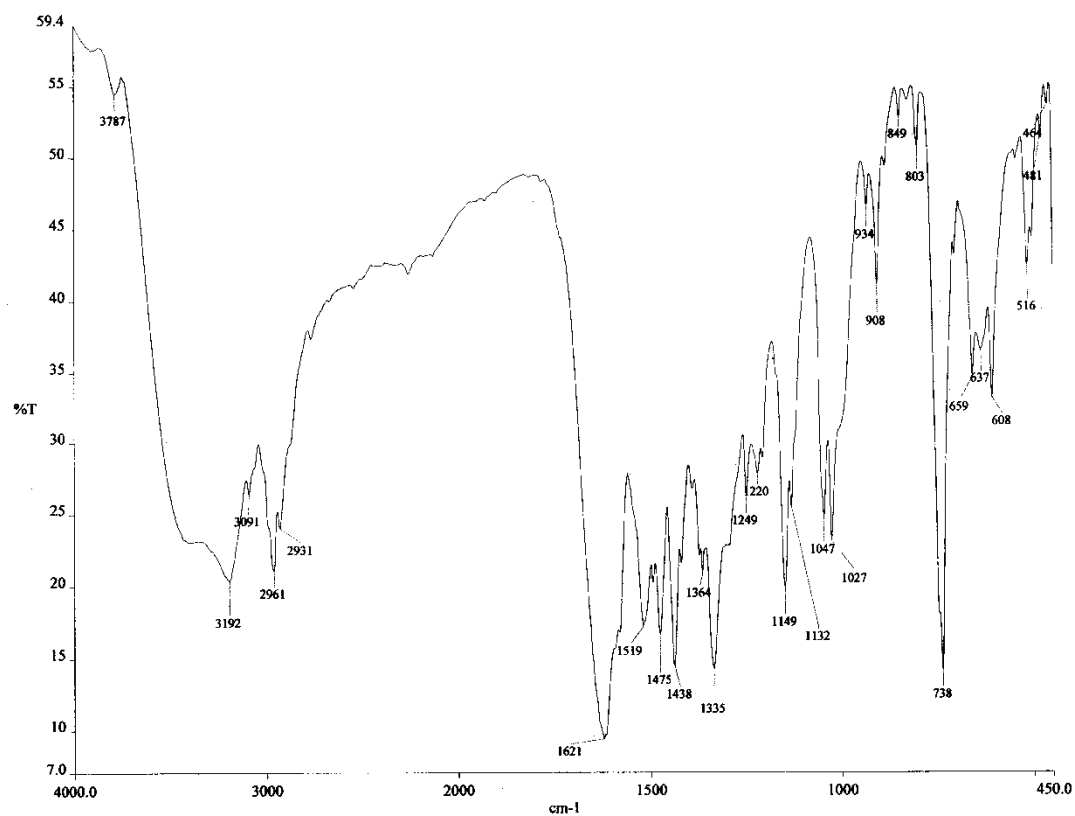
Morpholine-4-carboxylic acid (2-methoxy-phenyl)-amide (16a) ¹H NMR (CDCl₃, 400 MHz)



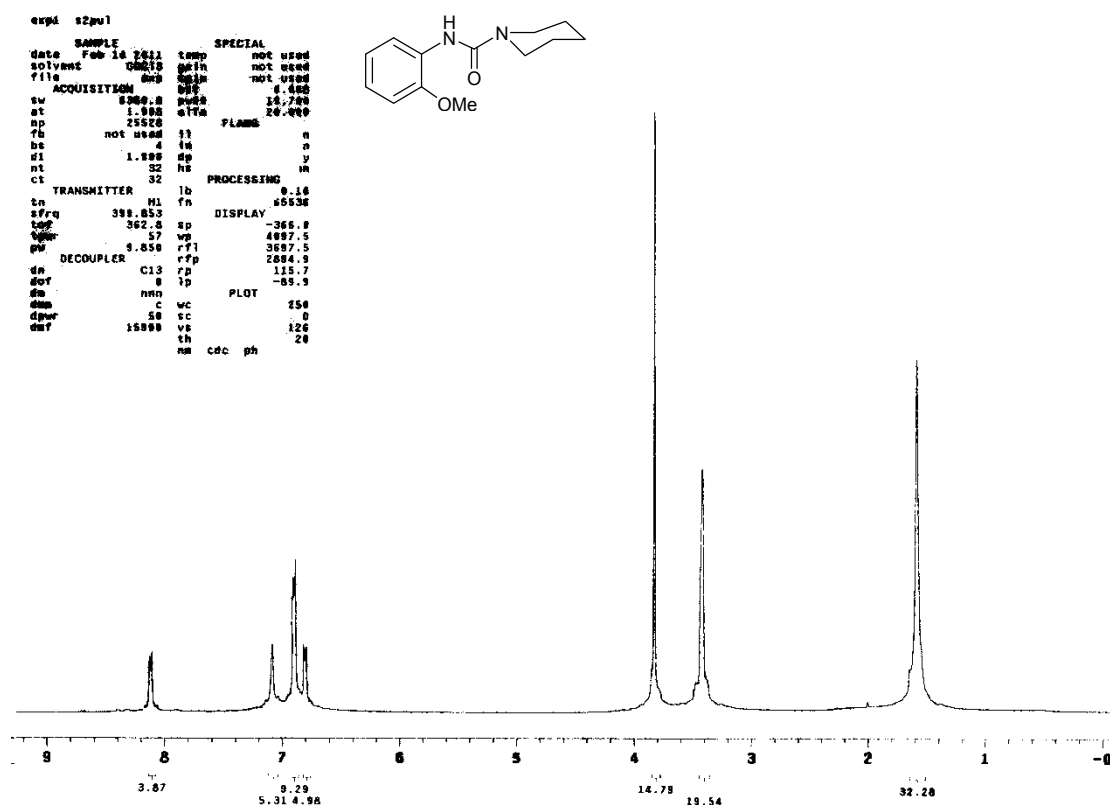
Morpholine-4-carboxylic acid (2-methoxy-phenyl)-amide (16a): ^{13}C NMR
(CDCl_3 , 100 MHz)



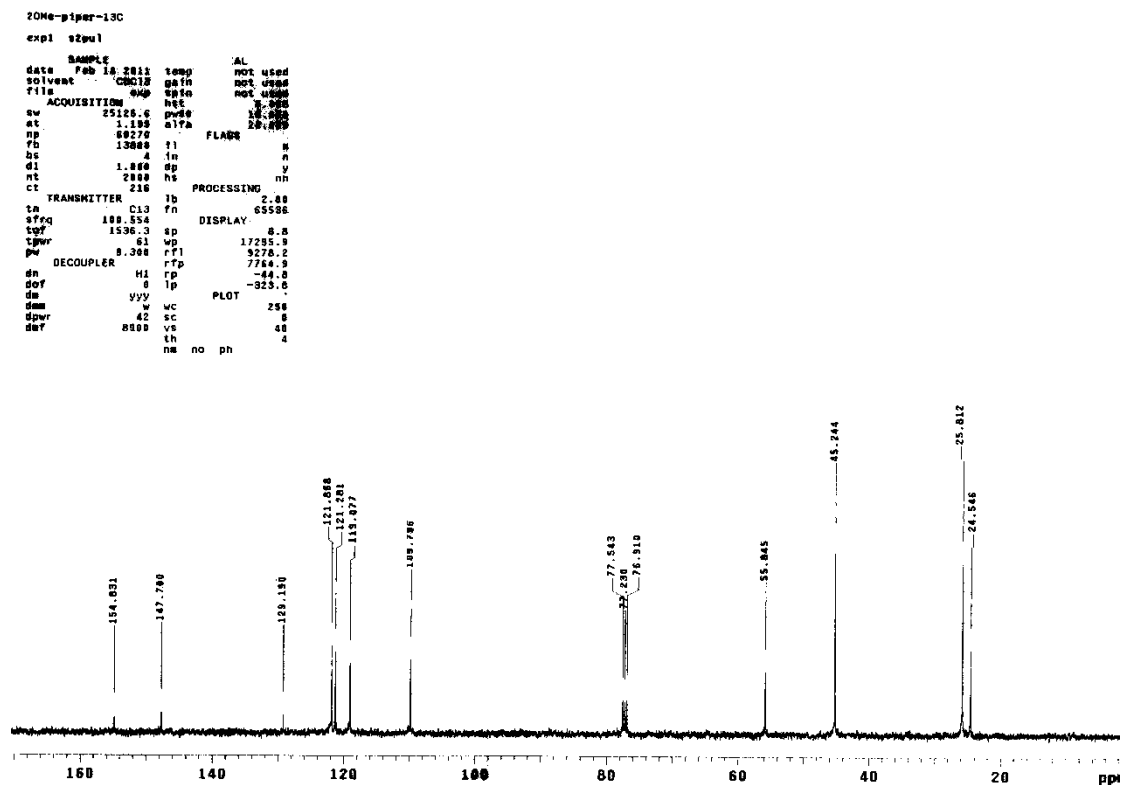
Morpholine-4-carboxylic acid (2-methoxy-phenyl)-amide (16a): IR (KBr)



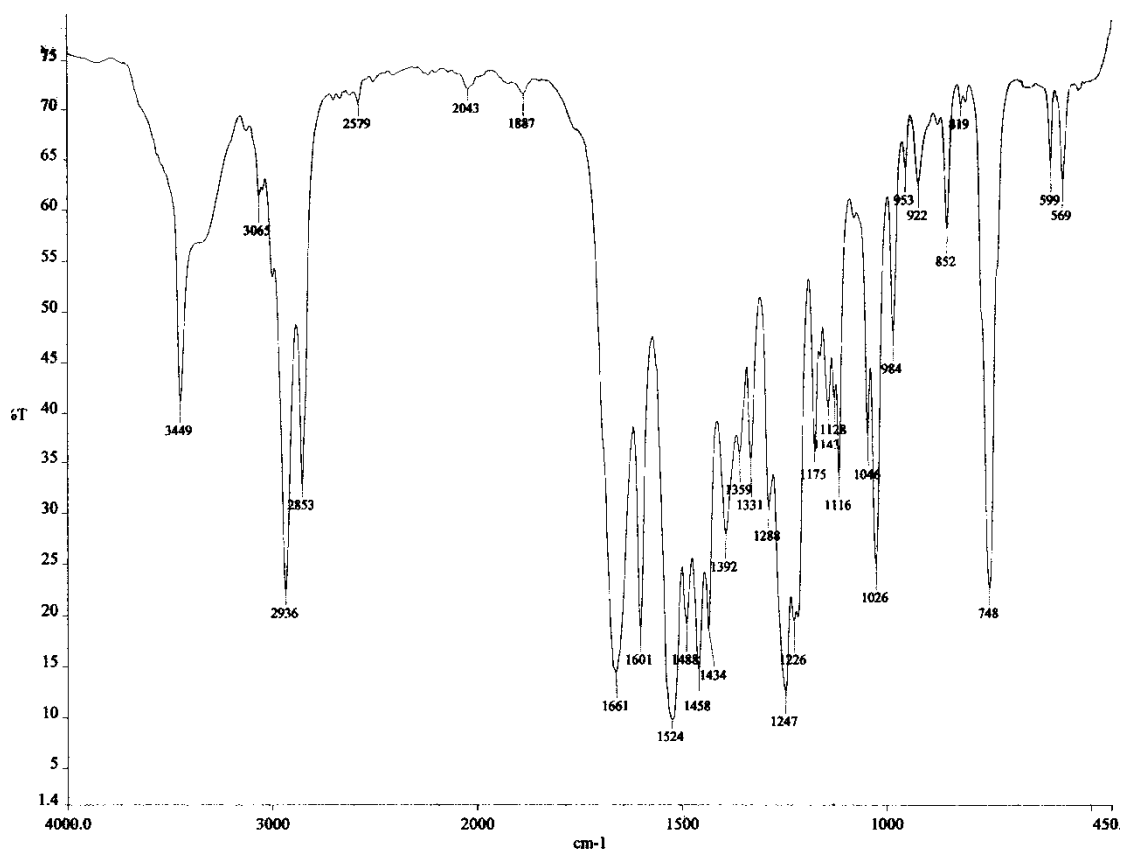
Piperidine-1-carboxylic acid (2-methoxy-phenyl)-amide (16b): ^1H NMR (CDCl_3 , 400 MHz)



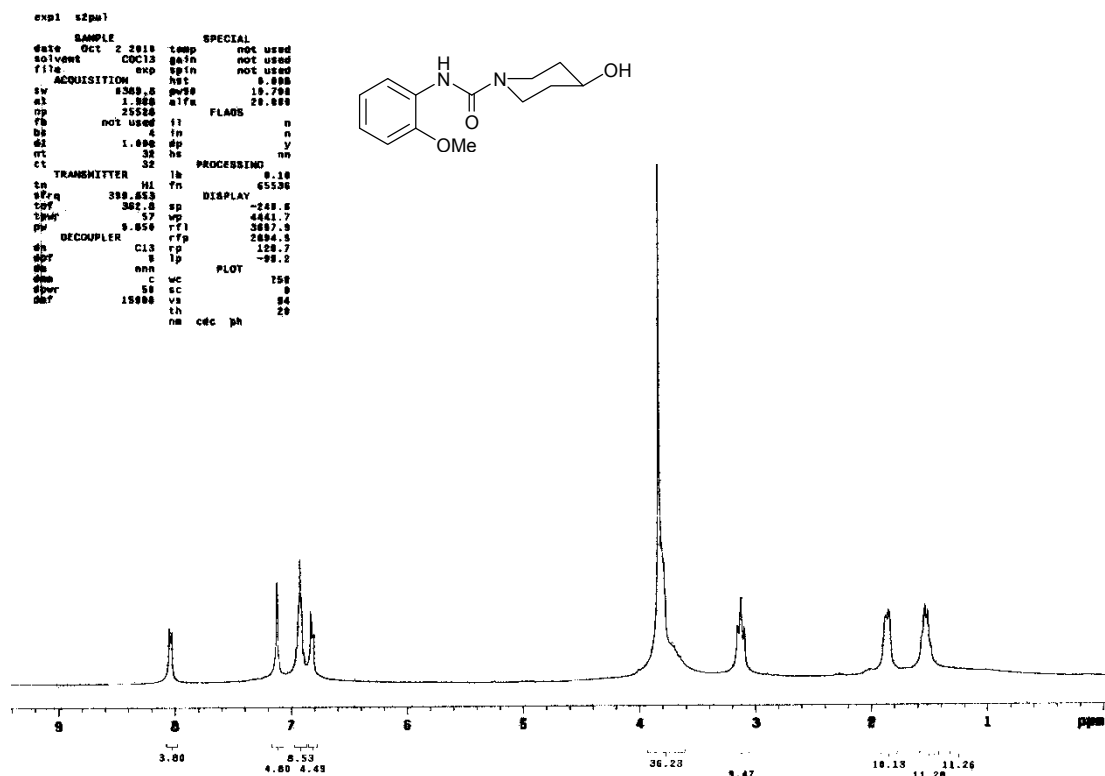
Piperidine-1-carboxylic acid (2-methoxy-phenyl)-amide (16b): ^{13}C NMR (CDCl_3 , 100 MHz)



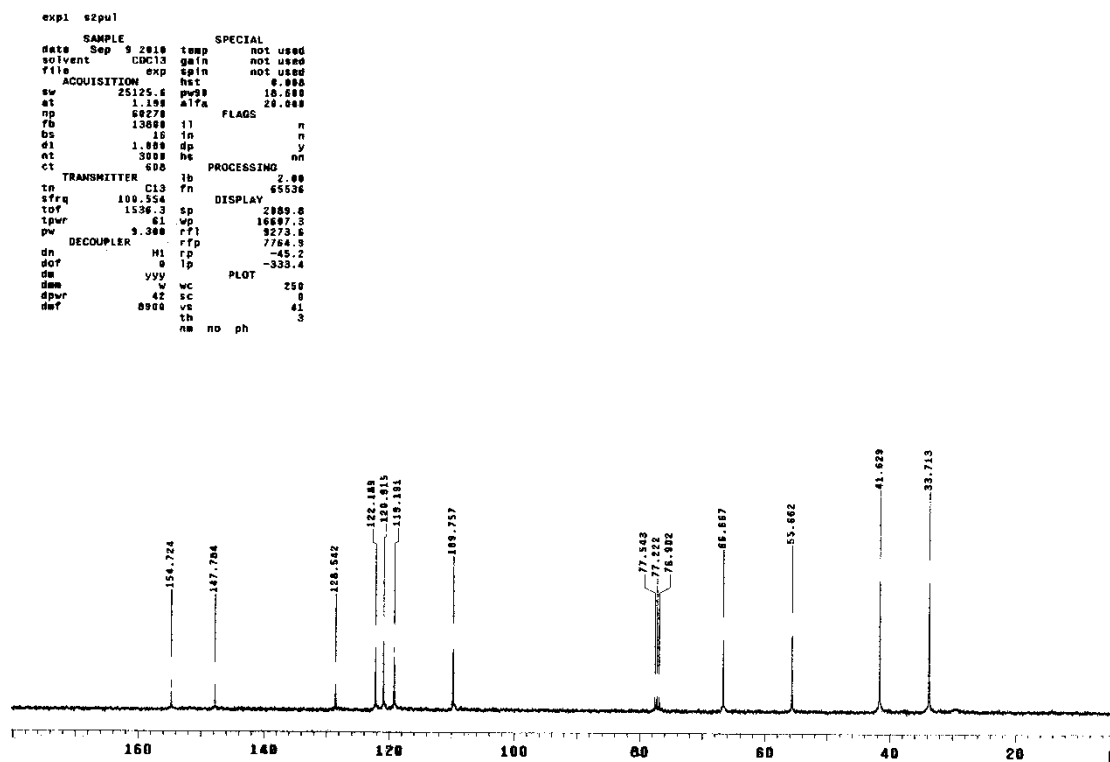
Piperidine-1-carboxylic acid (2-methoxy-phenyl)-amide (16b): IR (KBr)



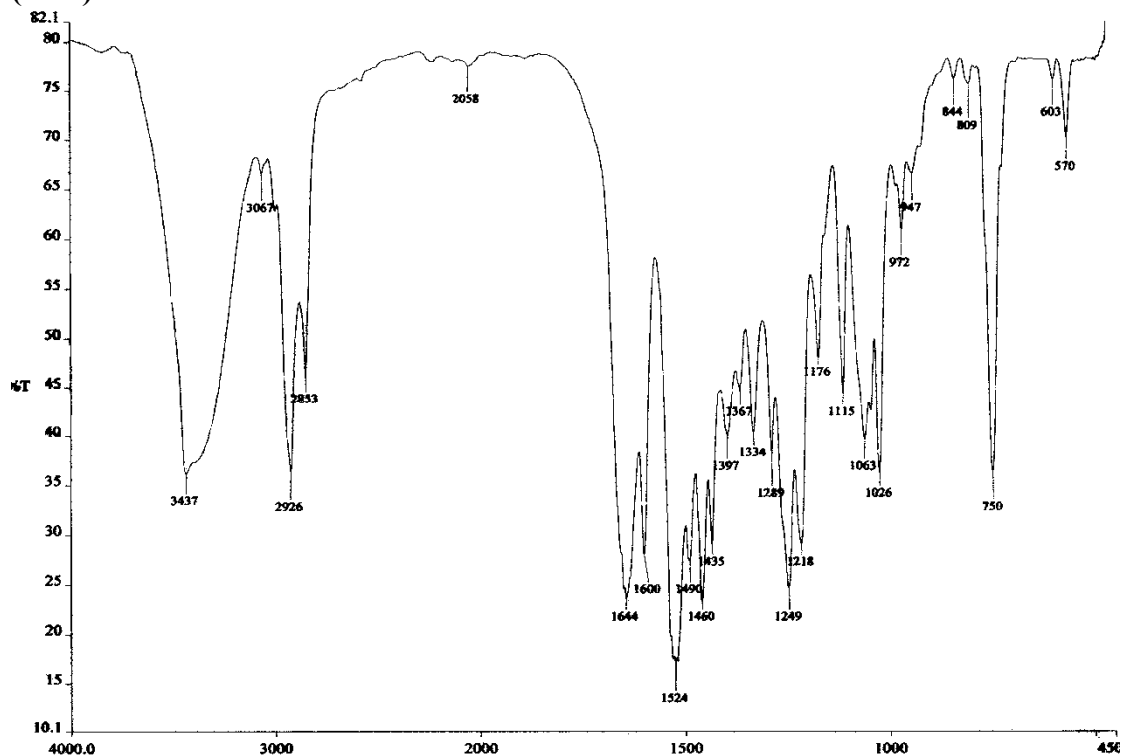
4-Hydroxy-piperidine-1-carboxylic acid (2-methoxy-phenyl)-amide (16c): ¹H NMR (CDCl₃, 400 MHz)



4-Hydroxy-piperidine-1-carboxylic acid (2-methoxy-phenyl)-amide (16c): ^{13}C NMR (CDCl_3 , 100 MHz)



4-Hydroxy-piperidine-1-carboxylic acid (2-methoxy-phenyl)-amide (16c): IR (KBr)

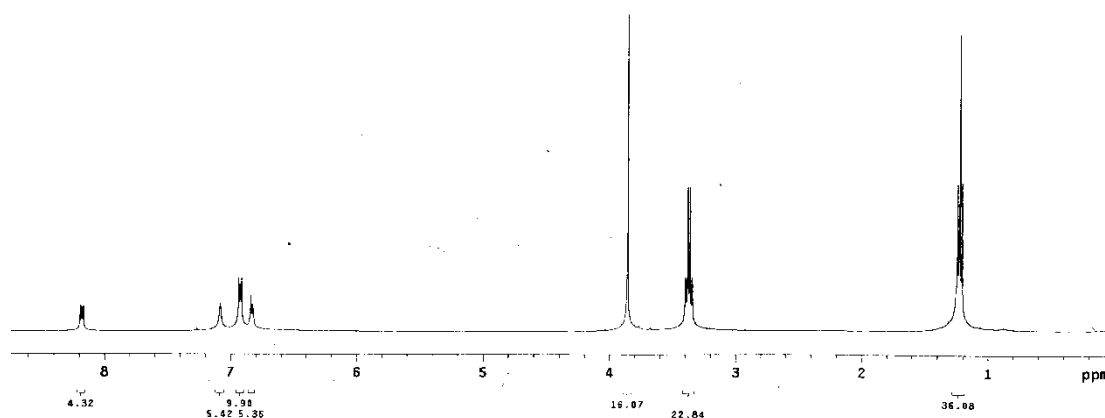
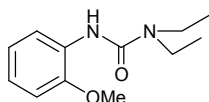


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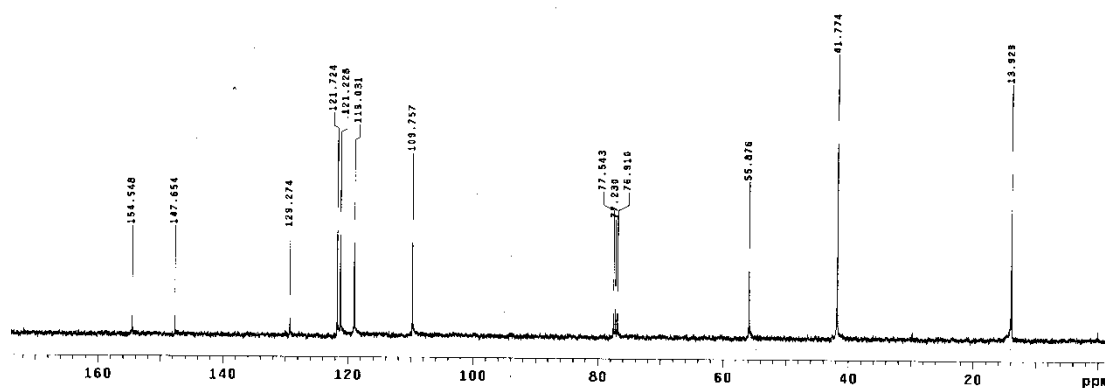
LJ_idea
expl szpul

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ft 1.998 pw0.0 alpha 20.000
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ct 32 hs nn
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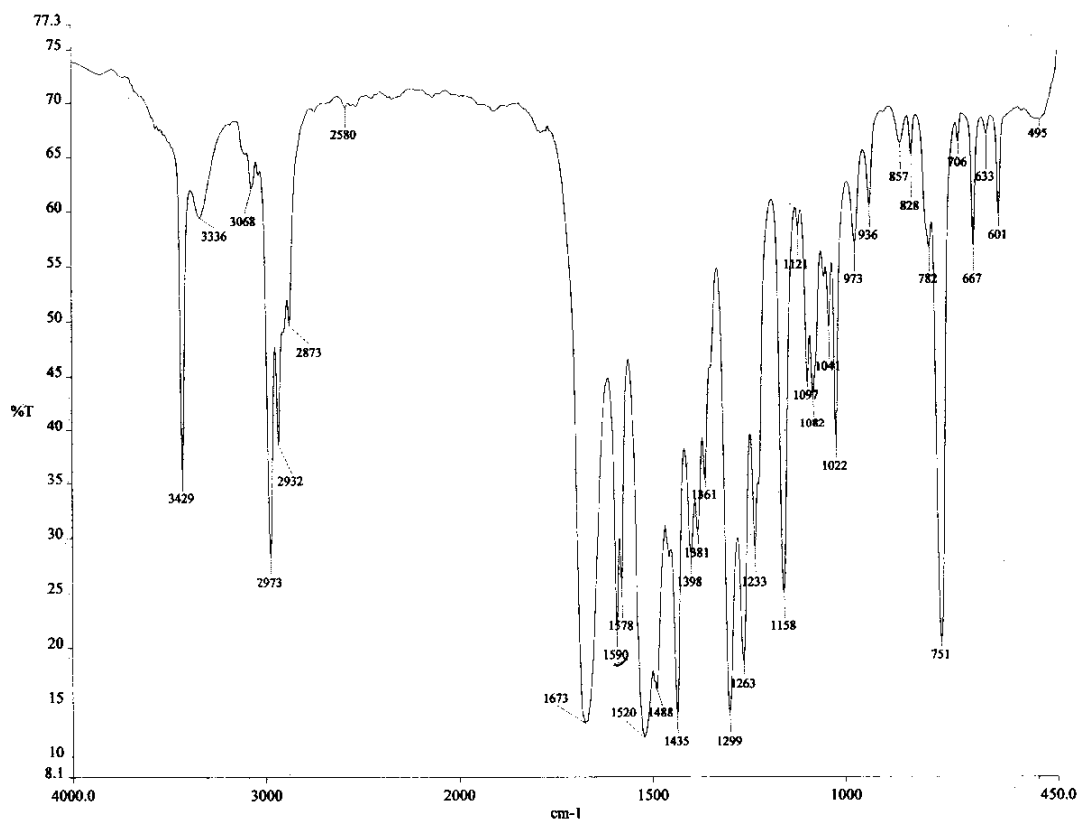
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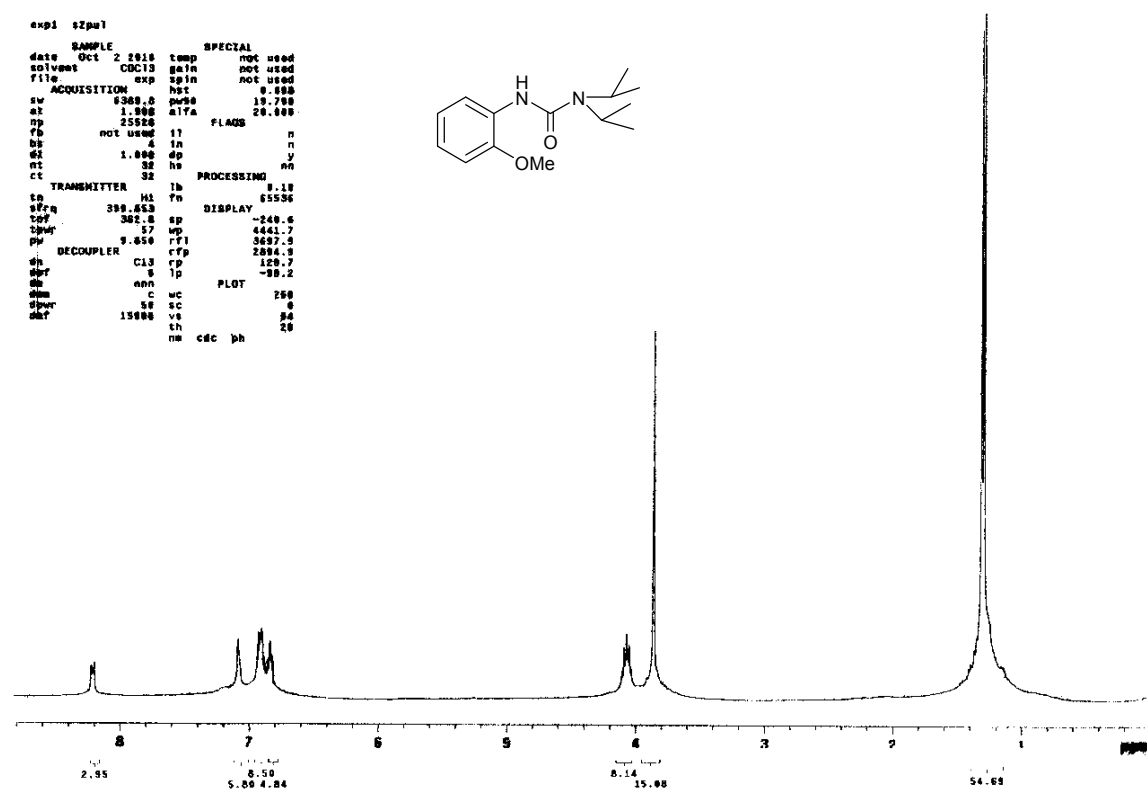
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date SAMPLE				SPECIAL			
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np	86778		FLAGS				
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dl	1.000	dp	n				
ct	2000	hs	y				
ct	260	PROCESSING					
transmitter	1b	1b	2.800				
rf	C13	1b	\$2.800				
sfreq	100.554	DISPLAY					
nu	1536.3	1b	-261.2				
tpwr	5.380	1b	17791.2				
decoupler	4	rf	17265.8				
pn	H1	rf	-44.6				
dcof	1p	rf	-359.9				
dm	yv	PLOT					
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dmc	vs	sc	44				
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		nm	na ph				



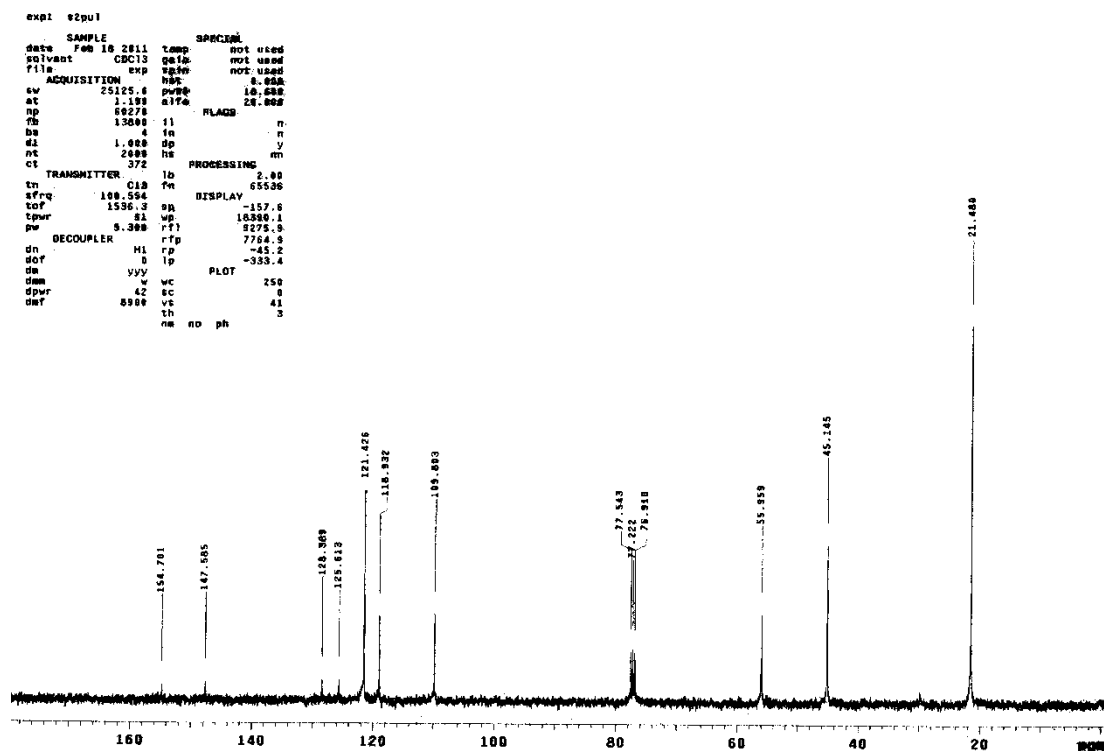
1,1-Diethyl-3-(2-methoxy-phenyl)-urea (16d): IR (KBr)



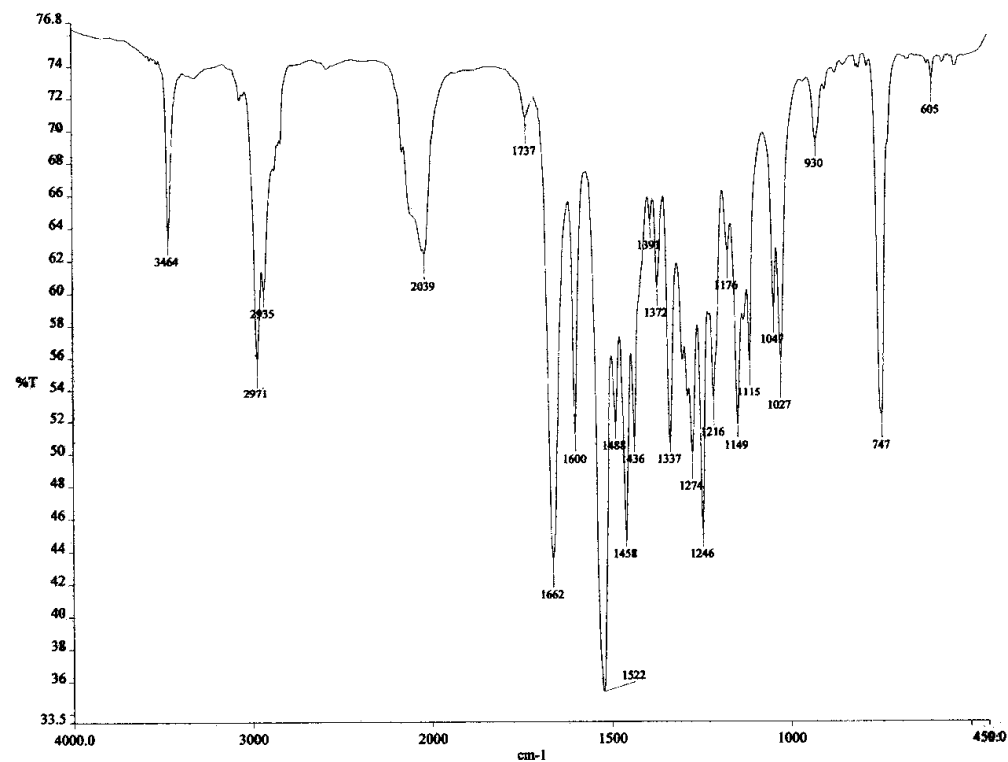
1,1-Diisopropyl-3-(2-methoxy-phenyl)-urea (16e): ¹H NMR (CDCl₃, 400 MHz)



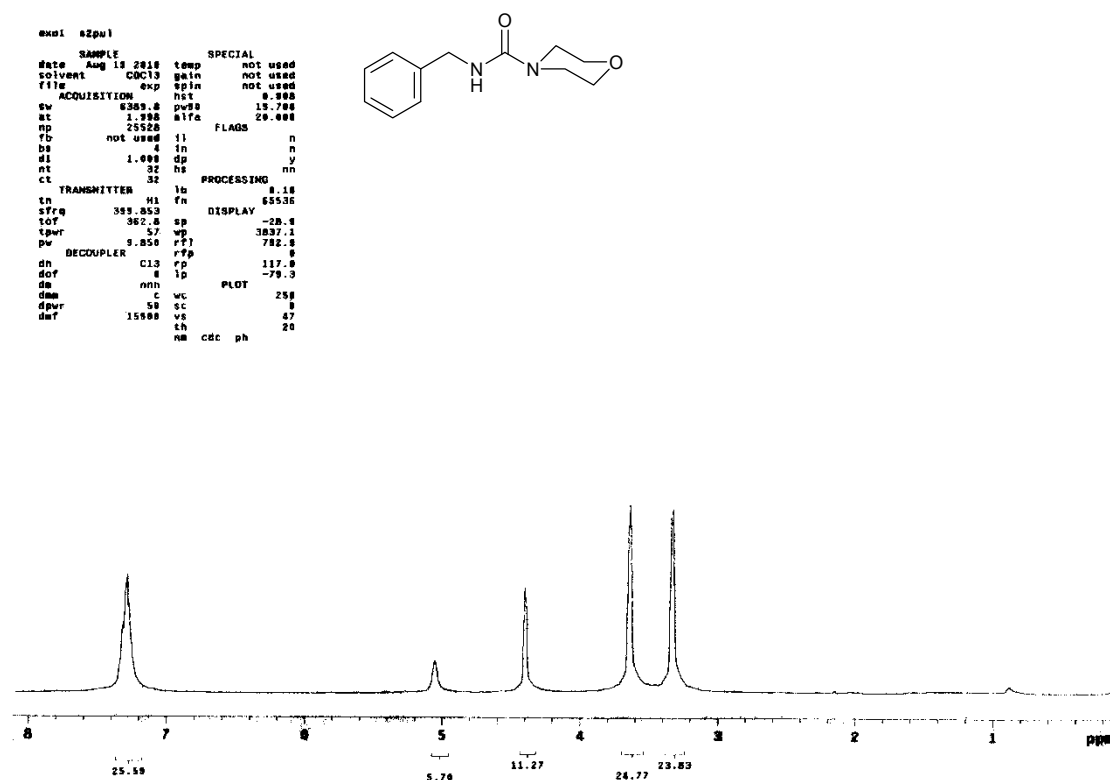
1,1-Diethyl-3-(2-methoxy-phenyl)-urea (16d): ^{13}C NMR (CDCl_3 , 100 MHz)



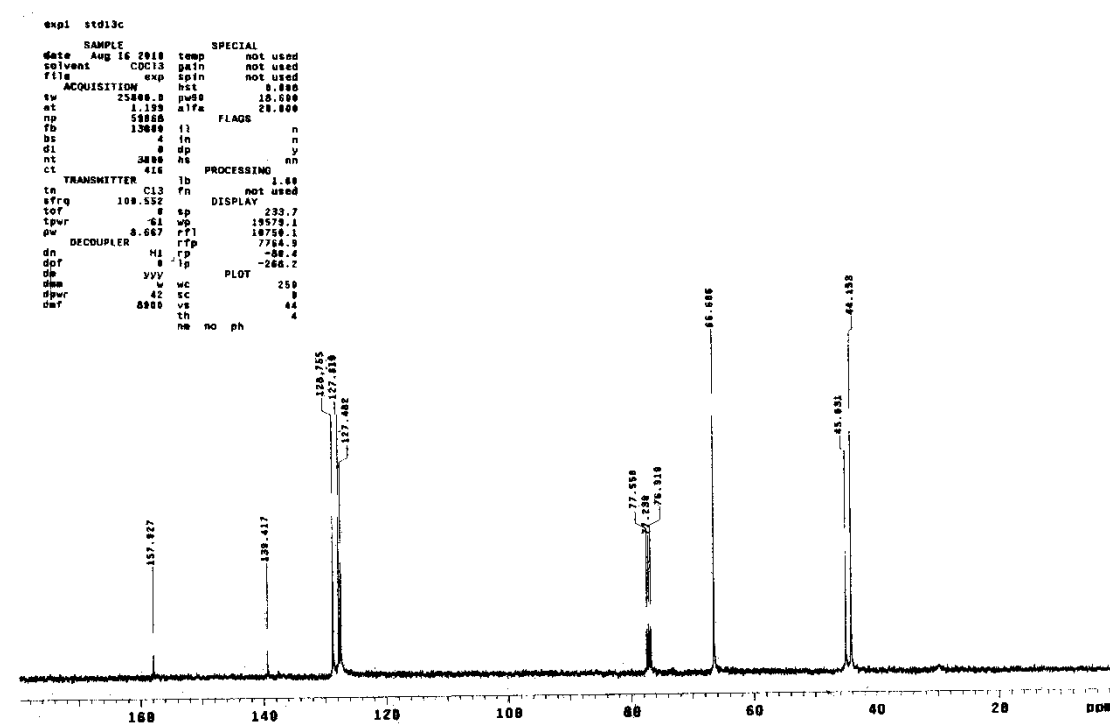
1,1-Diisopropyl-3-(2-methoxy-phenyl)-urea (16e): IR (KBr)



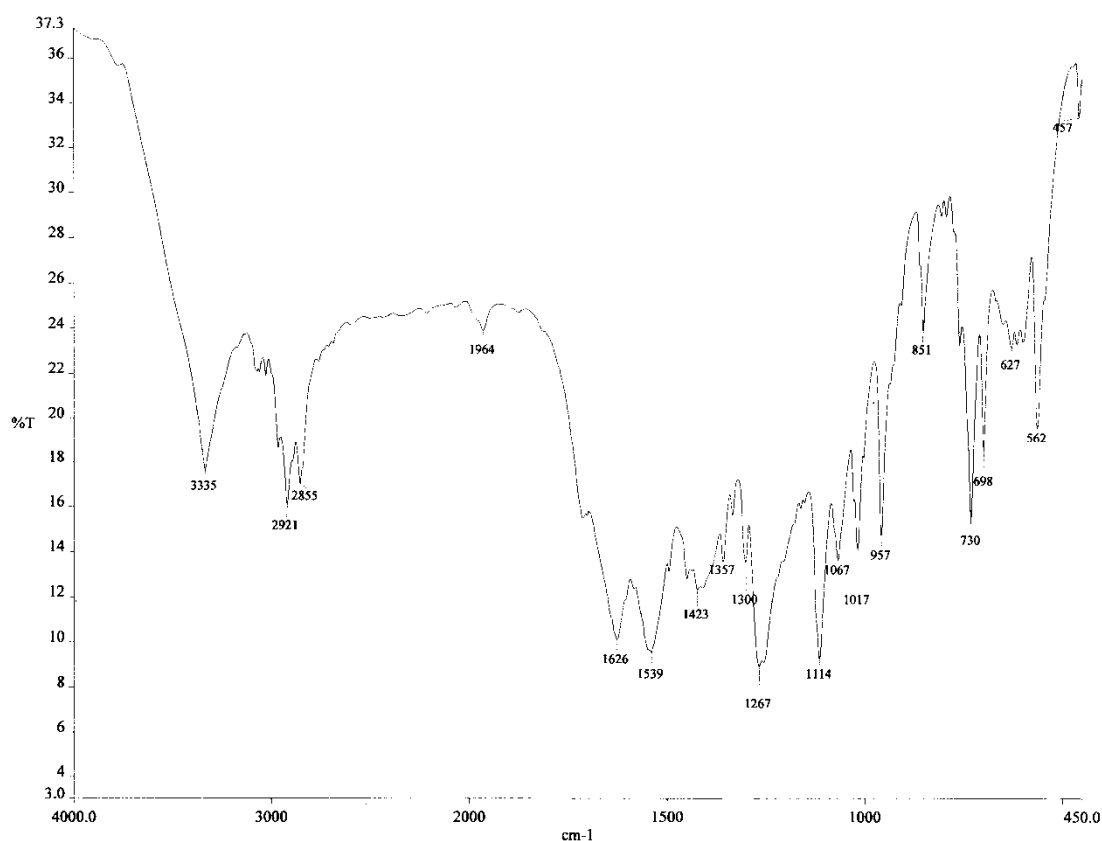
Morpholine-4-carboxylic acid benzylamide (17a): ^1H NMR (CDCl_3 , 400 MHz)



Morpholine-4-carboxylic acid benzylamide (17a): ^{13}C NMR (CDCl_3 , 100 MHz)



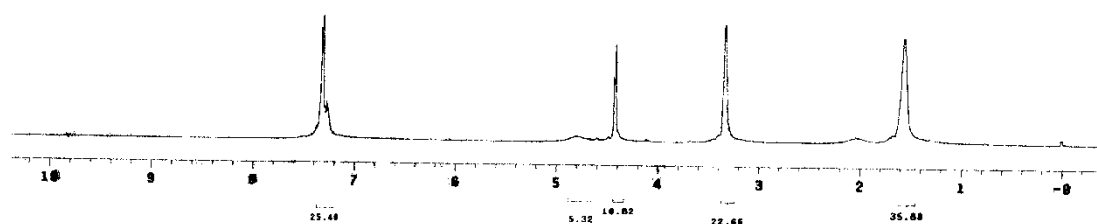
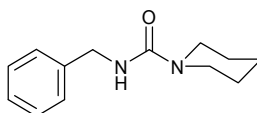
Morpholine-4-carboxylic acid benzylamide (17a): IR (KBr)



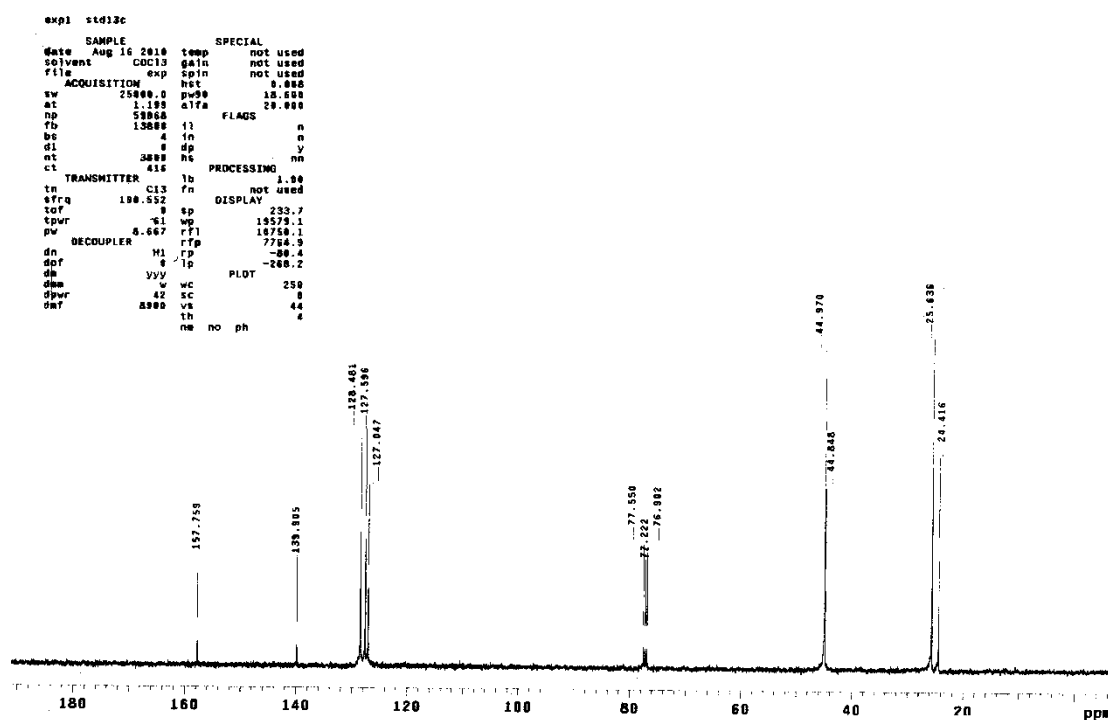
Piperidine-4-carboxylic acid benzylamide (17b): ^1H NMR (CDCl_3 , 400 MHz)

expt1 s2pul

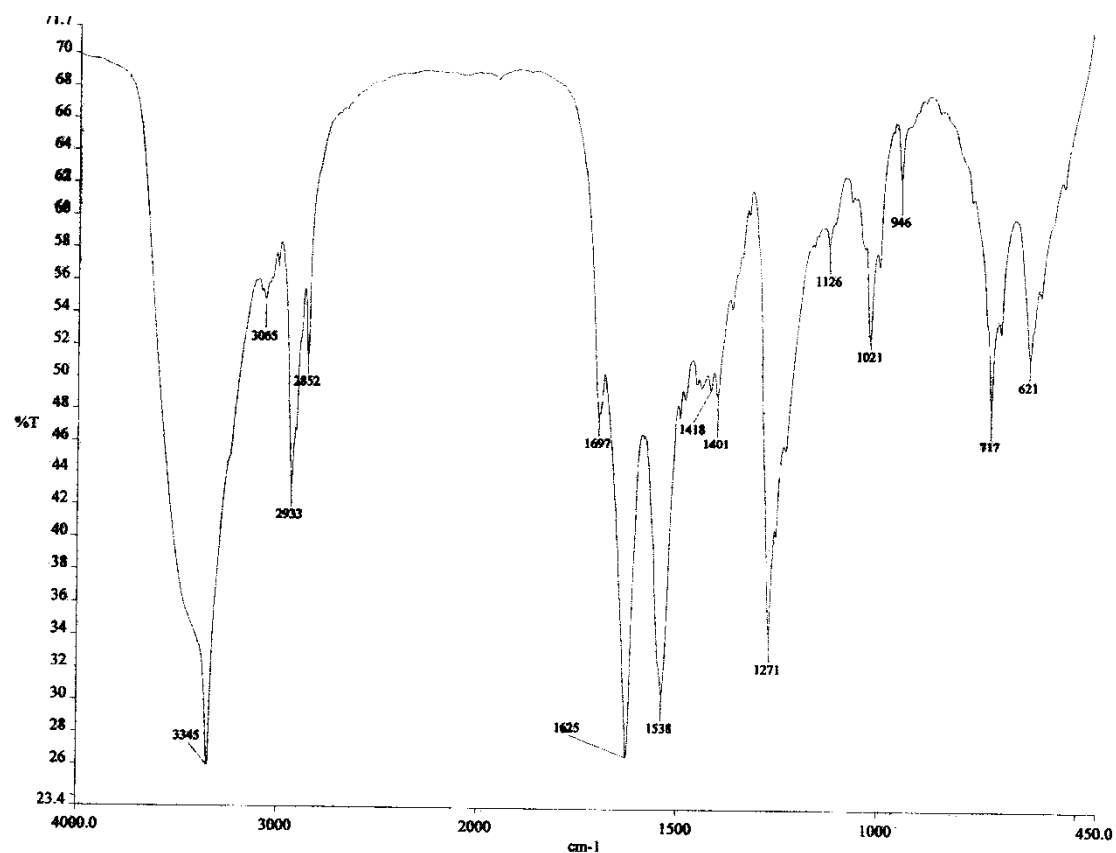
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np	25528	FLAGS	
fb	not used	il	n
bs	4	in	n
dl	1.840	dp	y
nt	32	ht	nn
ct	32	PROCESSING	
TRANSMITTER		lb	8.19
tr	H1	fn	65536
sfrq	399.853	DISPLAY	
tof	362.0	sp	-28.9
tpwr	57	vp	3837.1
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DECOUPLER		rfl	0
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	na	cdc	ph



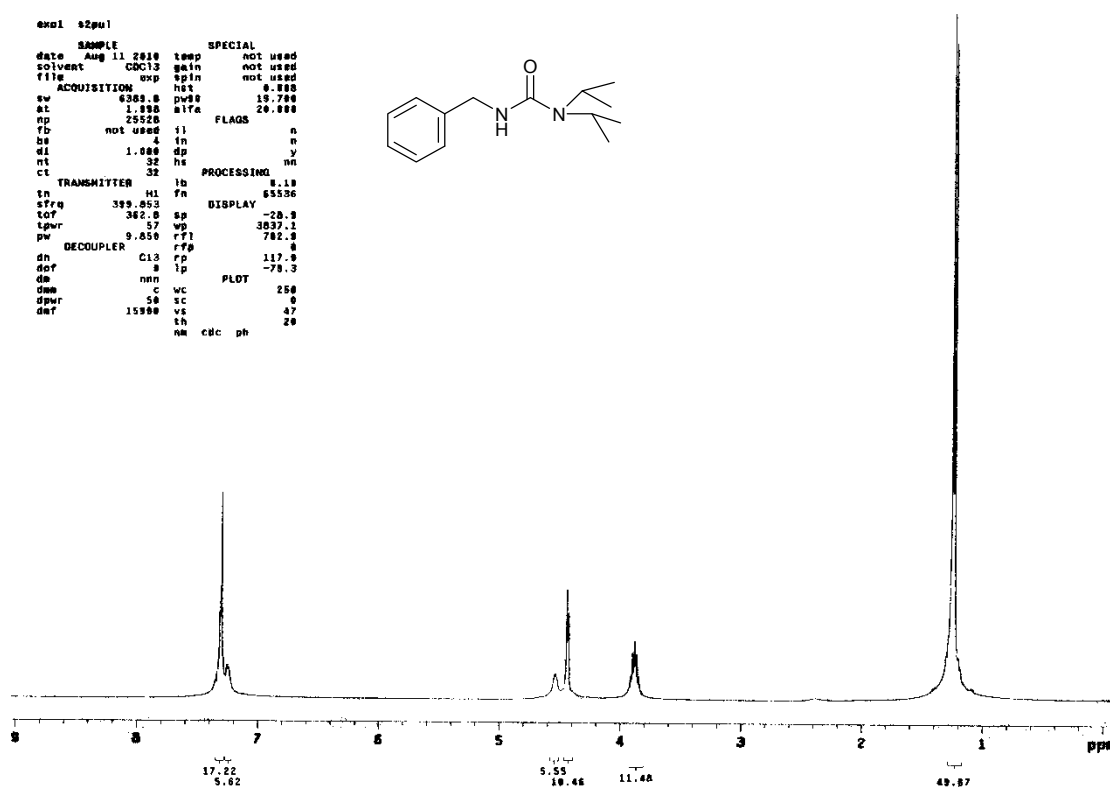
Piperidine-4-carboxylic acid benzylamide (17b): ^{13}C NMR (CDCl_3 , 100 MHz)



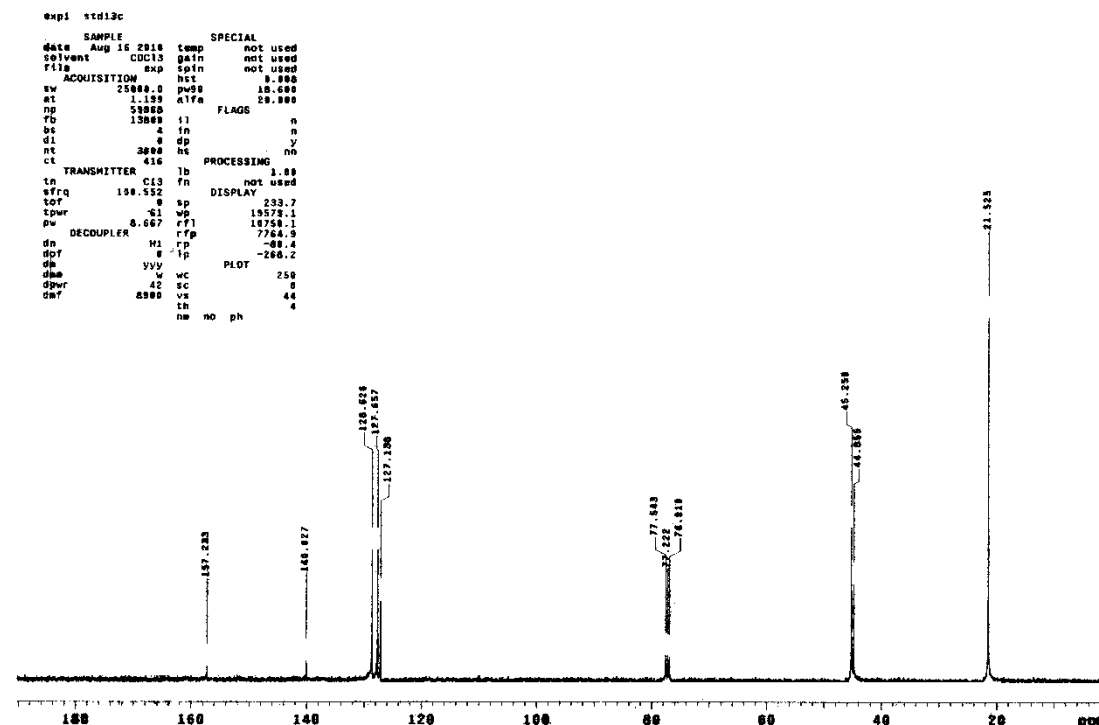
Piperidine-4-carboxylic acid benzylamide (17b): IR (KBr)



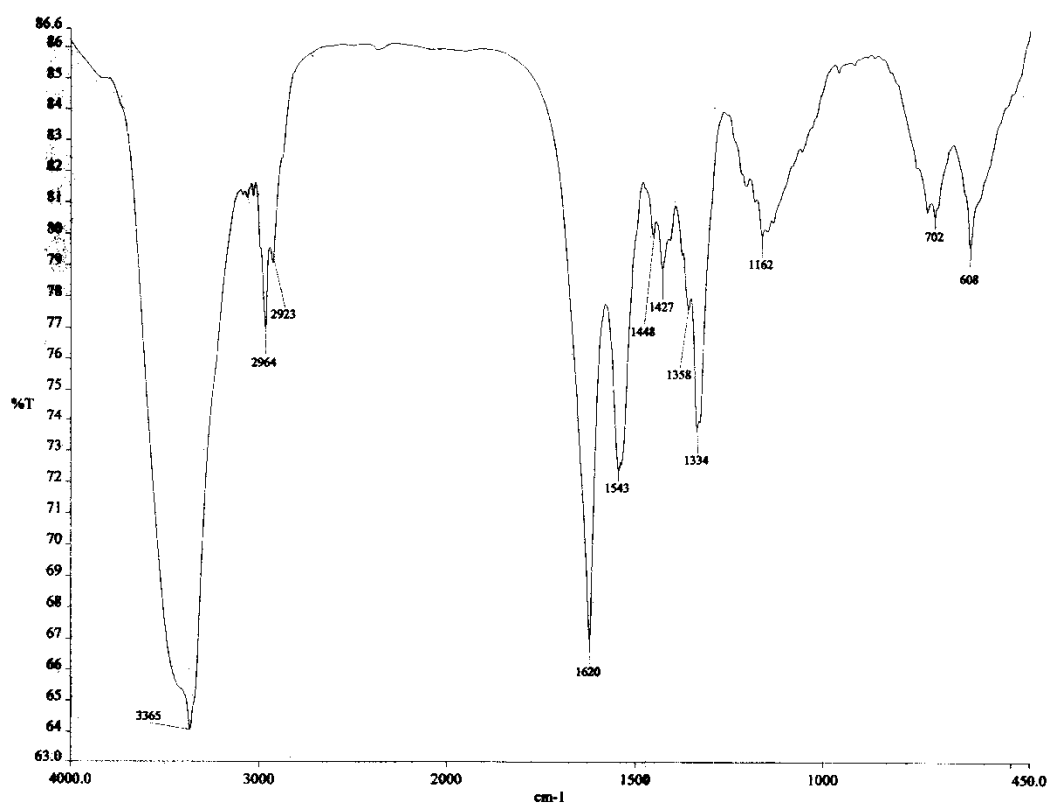
3-Benzyl-1,1-diisopropyl-urea (17e): ¹H NMR (CDCl₃, 400 MHz)



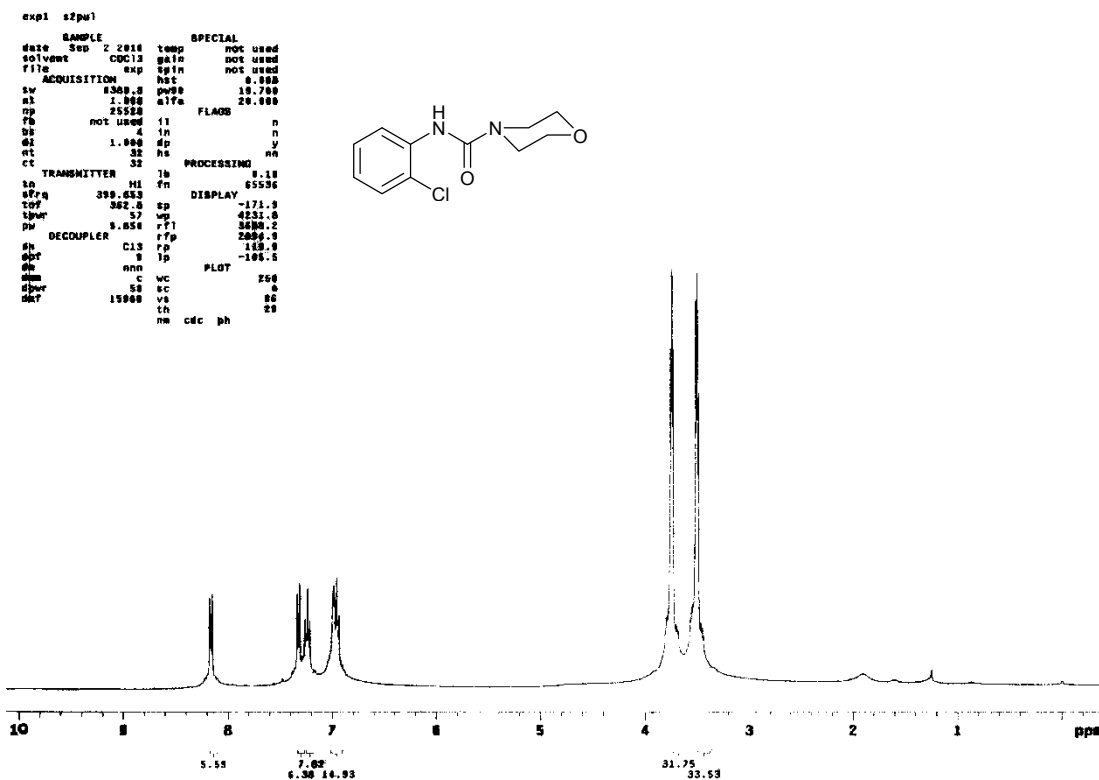
3-Benzyl-1,1-diisopropyl-urea (17e): ¹³C NMR (CDCl₃, 100 MHz)



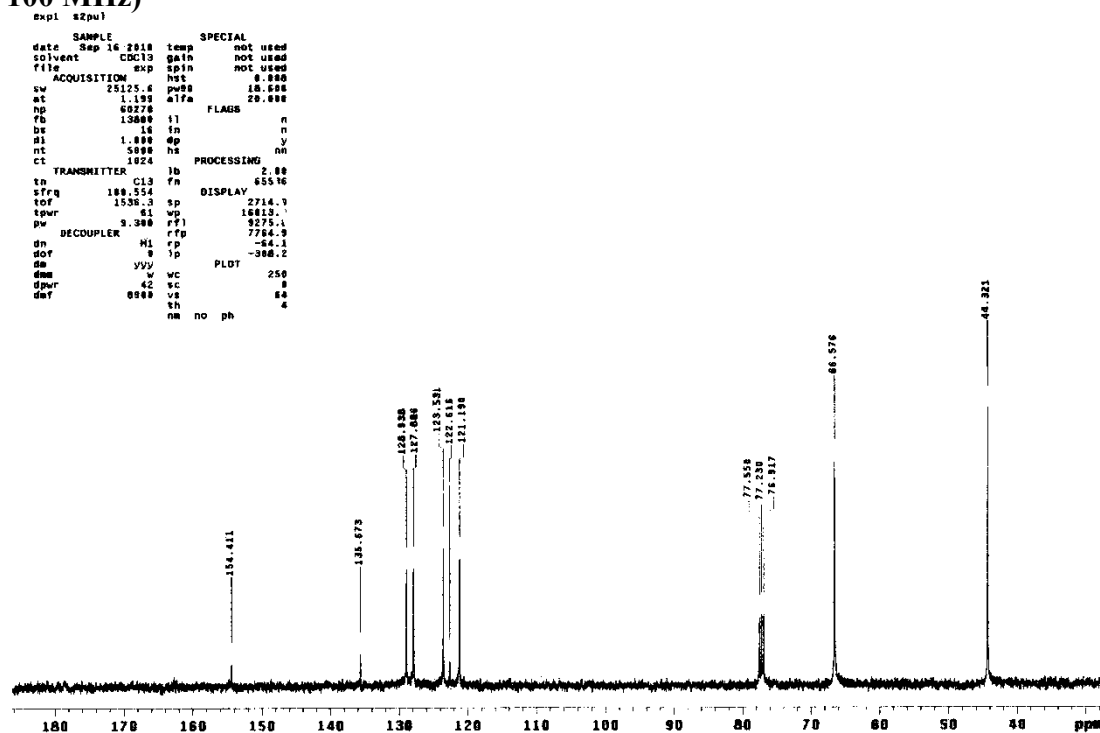
3-Benzyl-1,1-diisopropyl-urea (17e): IR (KBr)



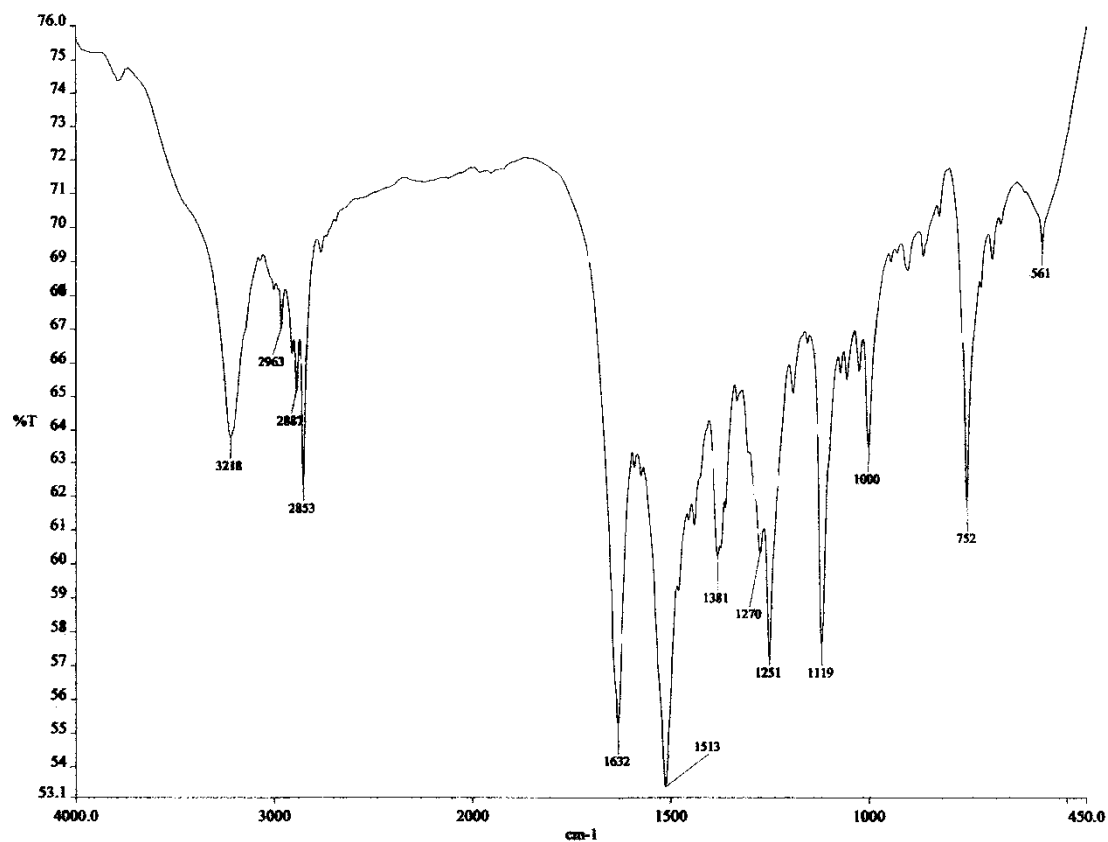
Morpholine-4-carboxylic acid (2-chloro-phenyl)-amide (18a): ¹H NMR (CDCl₃, 400 MHz)



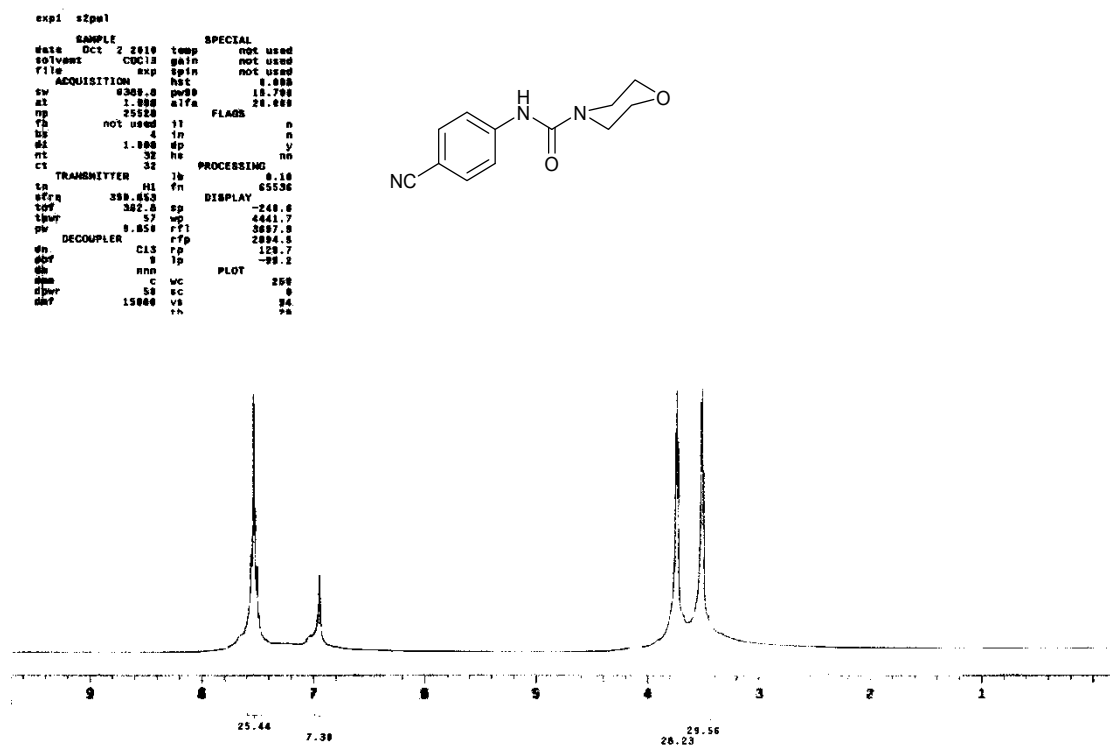
Morpholine-4-carboxylic acid (2-chloro-phenyl)-amide (18a): ^{13}C NMR (CDCl_3 , 100 MHz)



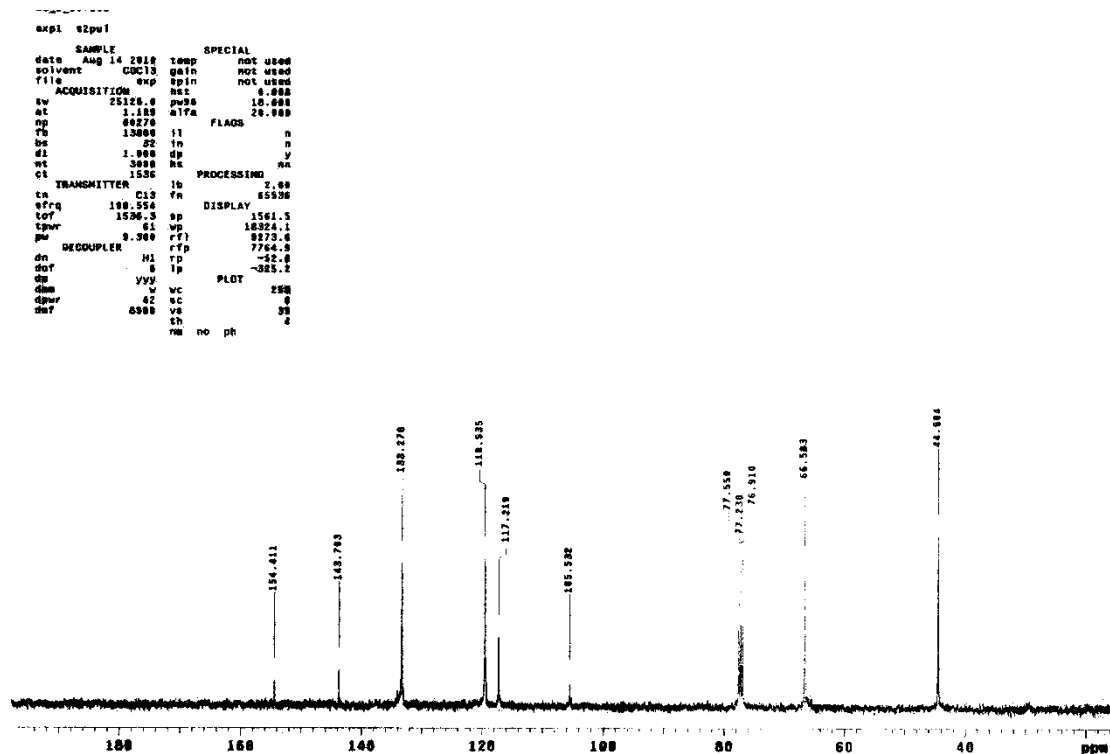
Morpholine-4-carboxylic acid (2-chloro-phenyl)-amide (18a): IR (KBr)



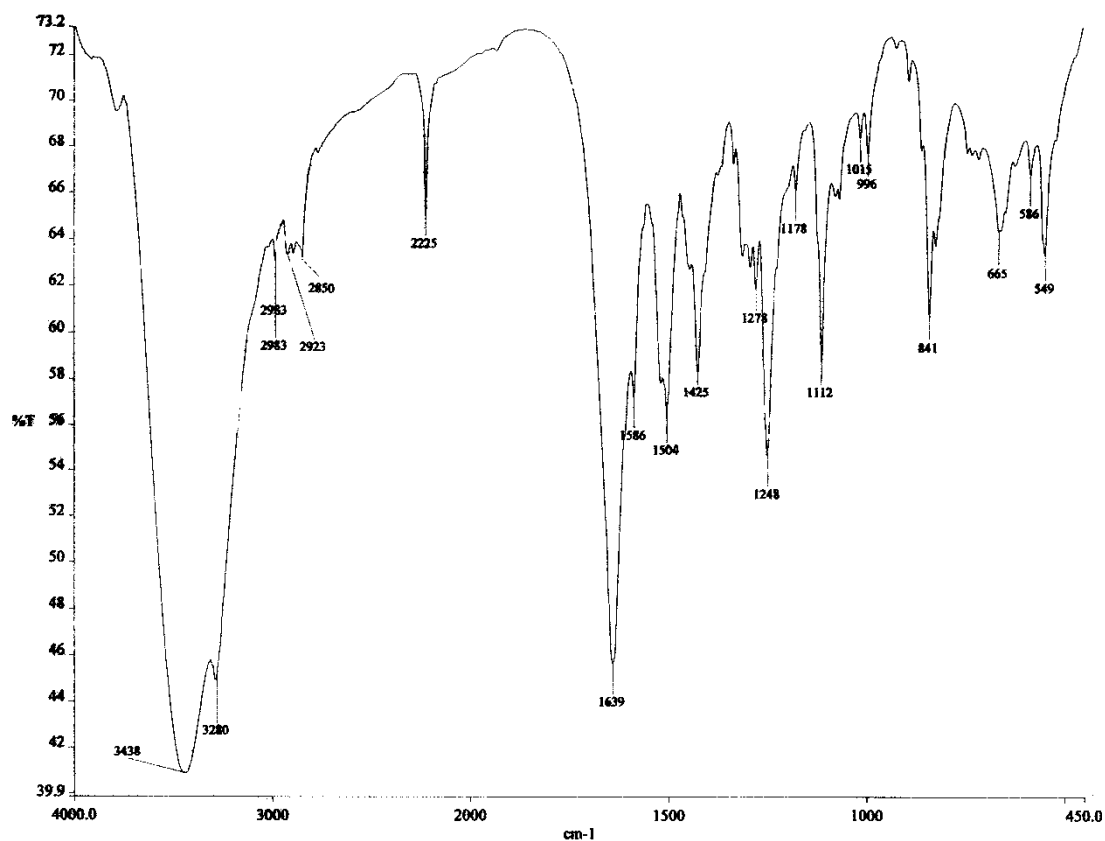
Morpholine-4-carboxylic acid (4-cyano-phenyl)-amide (19a): ^1H NMR (CDCl_3 , 400 MHz)



Morpholine-4-carboxylic acid (4-cyano-phenyl)-amide (19a): ^{13}C NMR (CDCl_3 , 100 MHz)



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

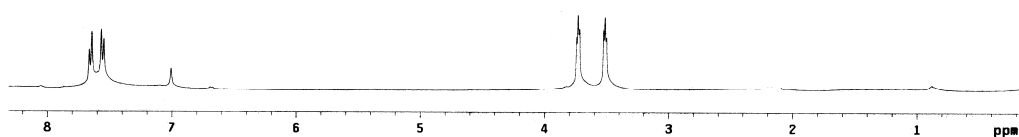
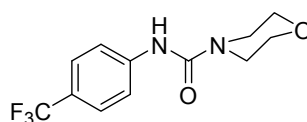


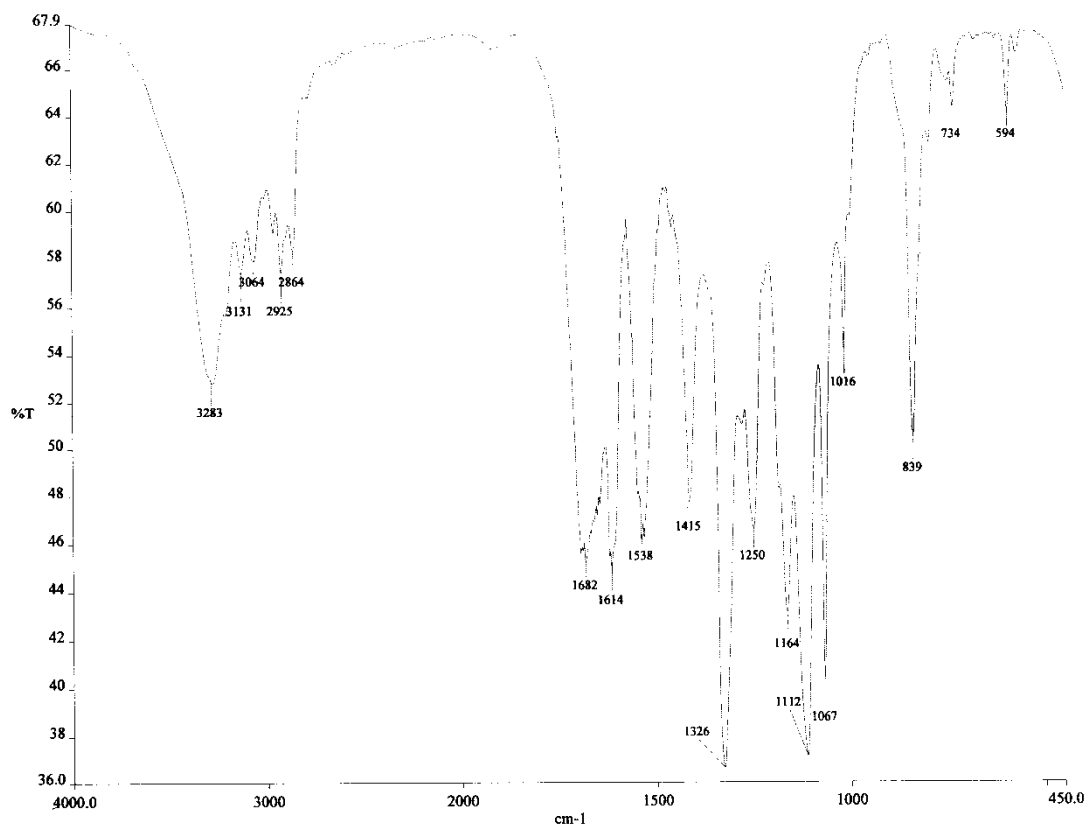
Morpholine-4-carboxylic acid (4-trifluoromethyl-phenyl)-amide (20a): ¹H NMR (CDCl₃, 400 MHz)

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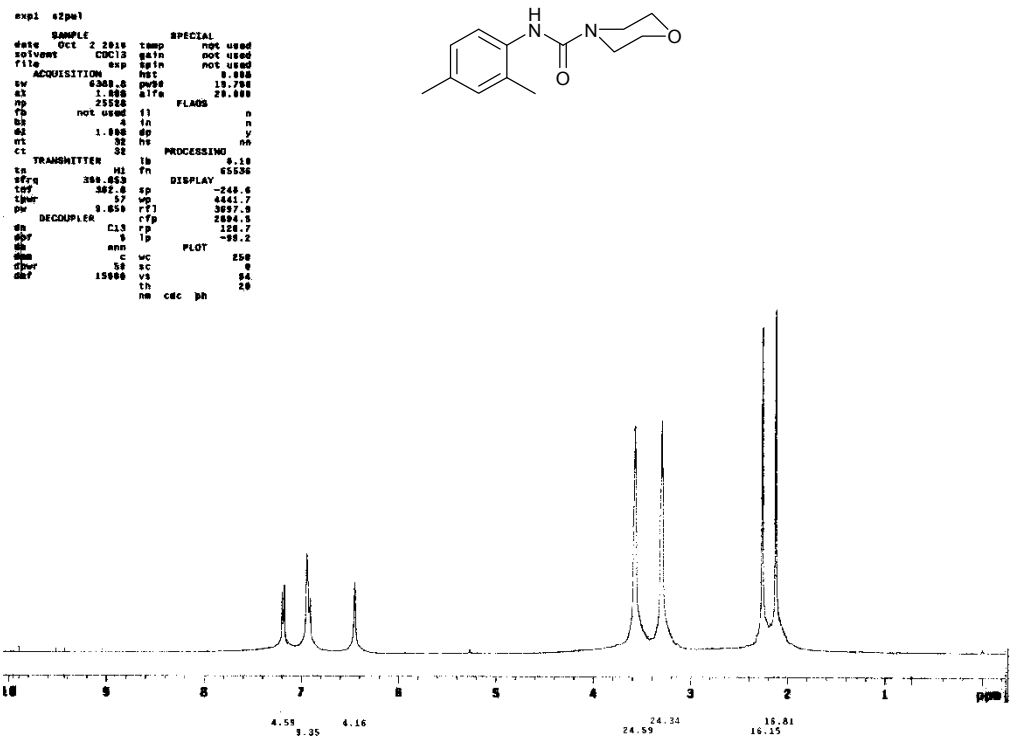
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file                                         spin    not used
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ct      32
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tprc     57        vp      3264.6
pw      9.650       rfp     789.4
=====
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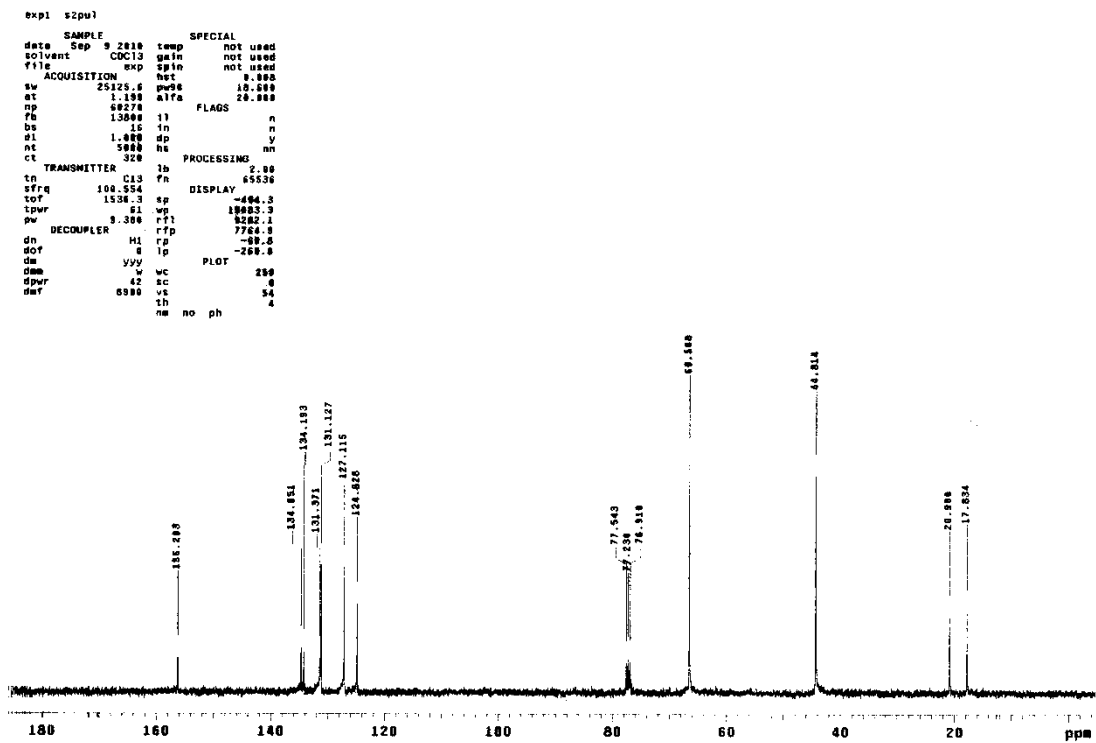


[illegible]

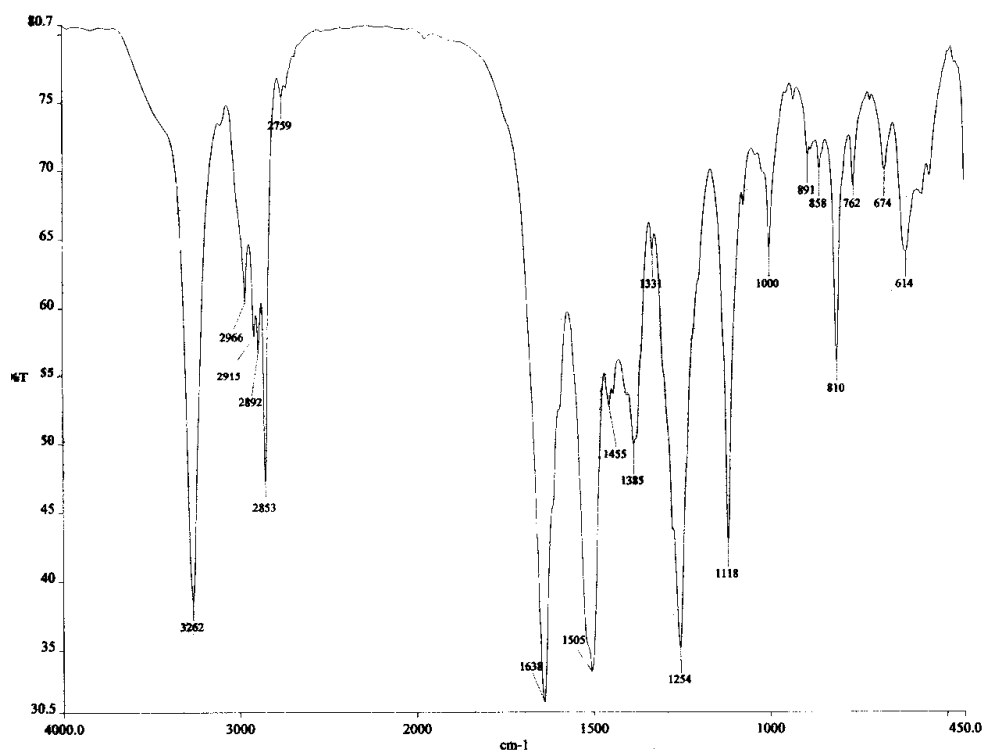
Morpholine-4-carboxylic acid (2,4-dimethyl-phenyl)-amide (21a): ¹H NMR (CDCl₃, 400 MHz)



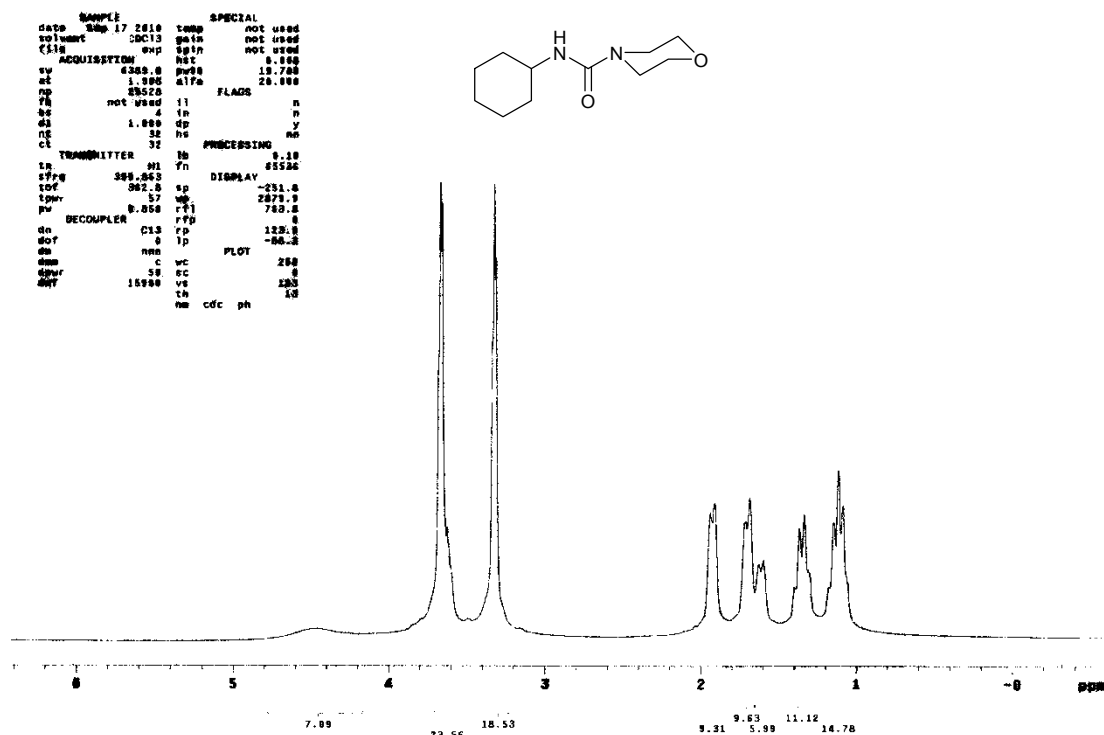
Morpholine-4-carboxylic acid (2,4-dimethyl-phenyl)-amide (21a): ¹³C NMR (CDCl₃, 100 MHz)



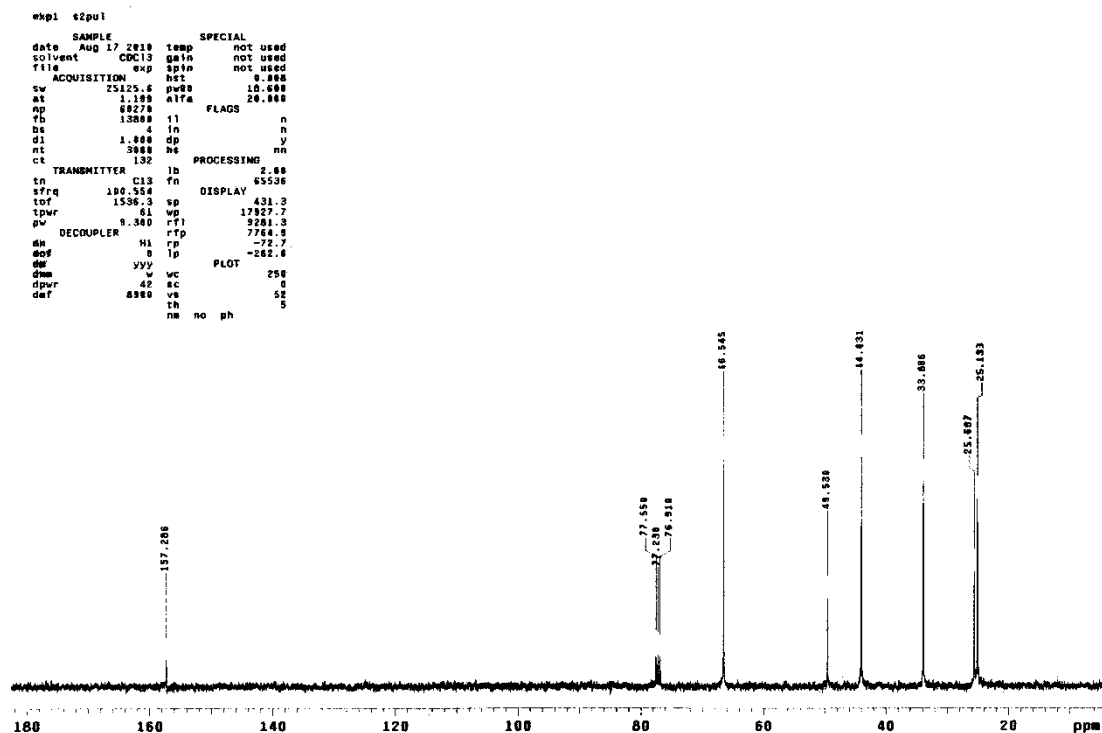
Morpholine-4-carboxylic acid (2,4-dimethyl-phenyl)-amide (21a): IR(KBr)



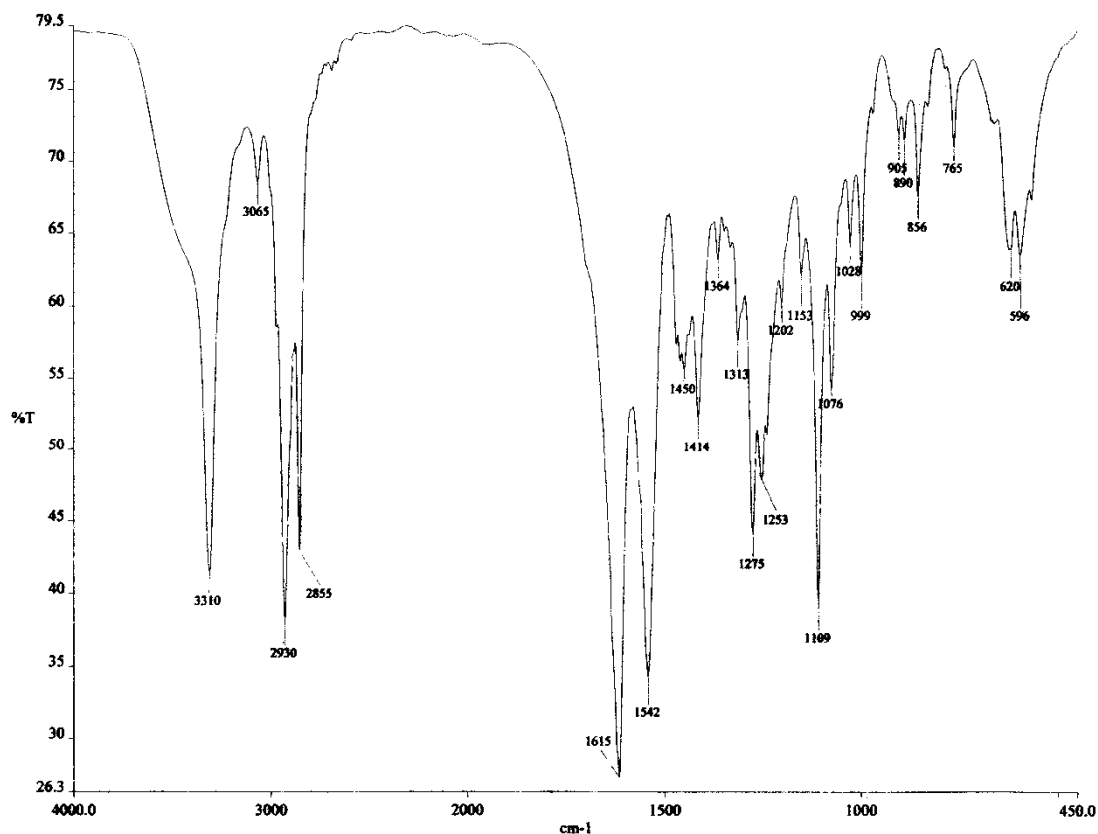
Morpholine-4-carboxylic acid cyclohexylamide (22a): ¹H NMR (CDCl₃, 400 MHz)



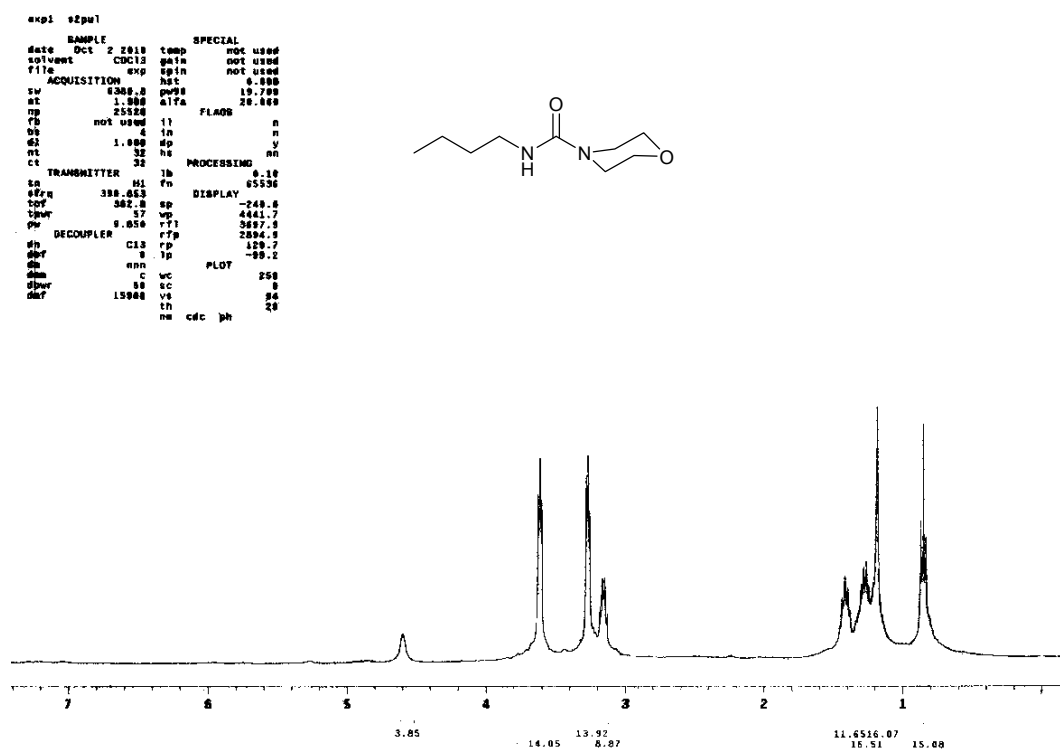
Morpholine-4-carboxylic acid cyclohexylamide (22a): ^{13}C NMR (CDCl_3 , 100 MHz)



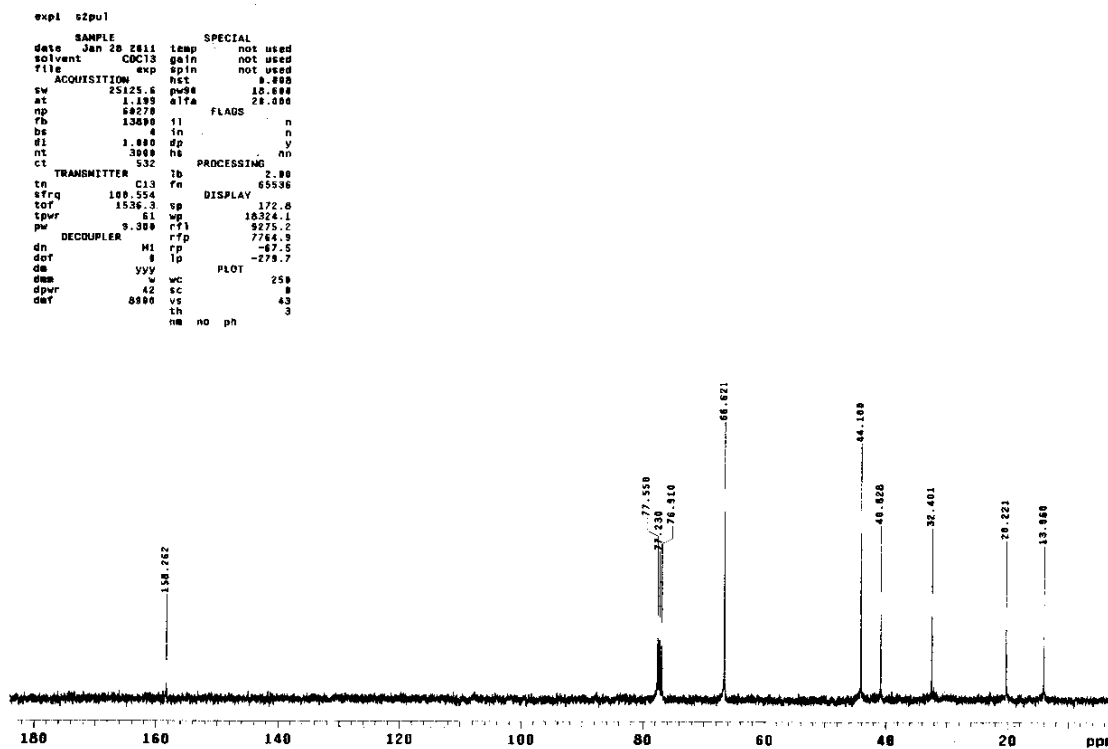
Morpholine-4-carboxylic acid cyclohexylamide (22a): IR (KBr)



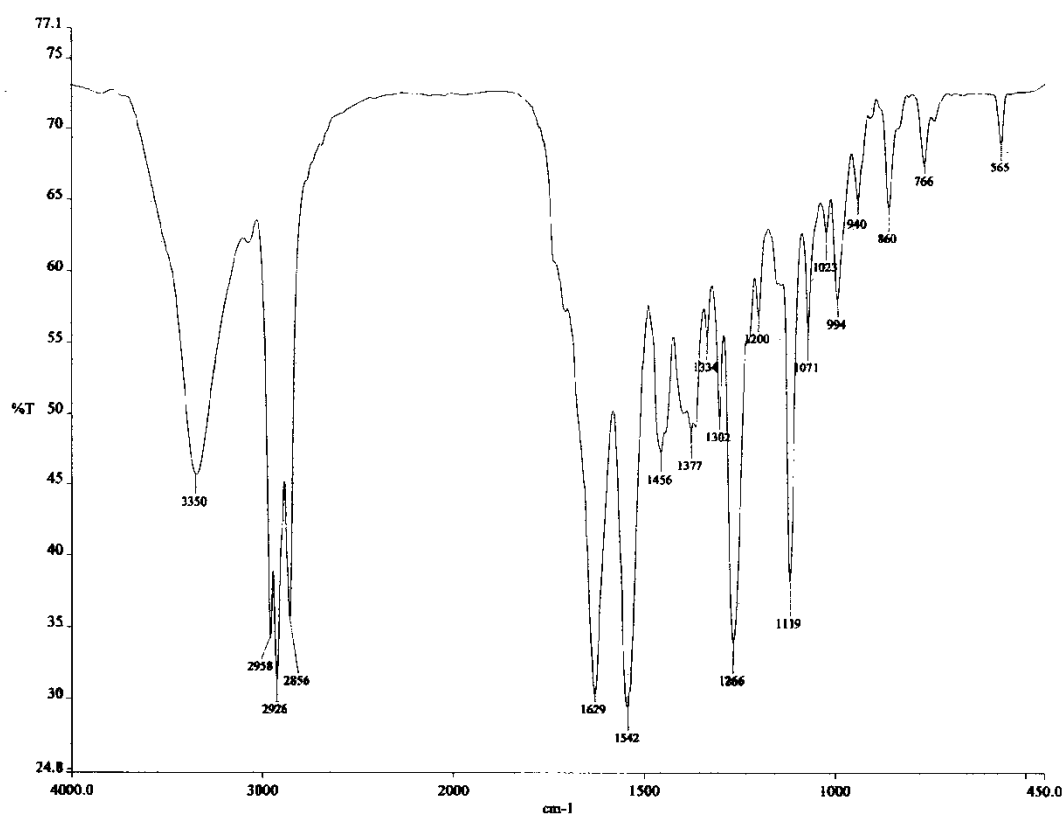
Morpholine-4-carboxylic acid butylamide (23a): ¹H NMR (CDCl₃, 400 MHz)



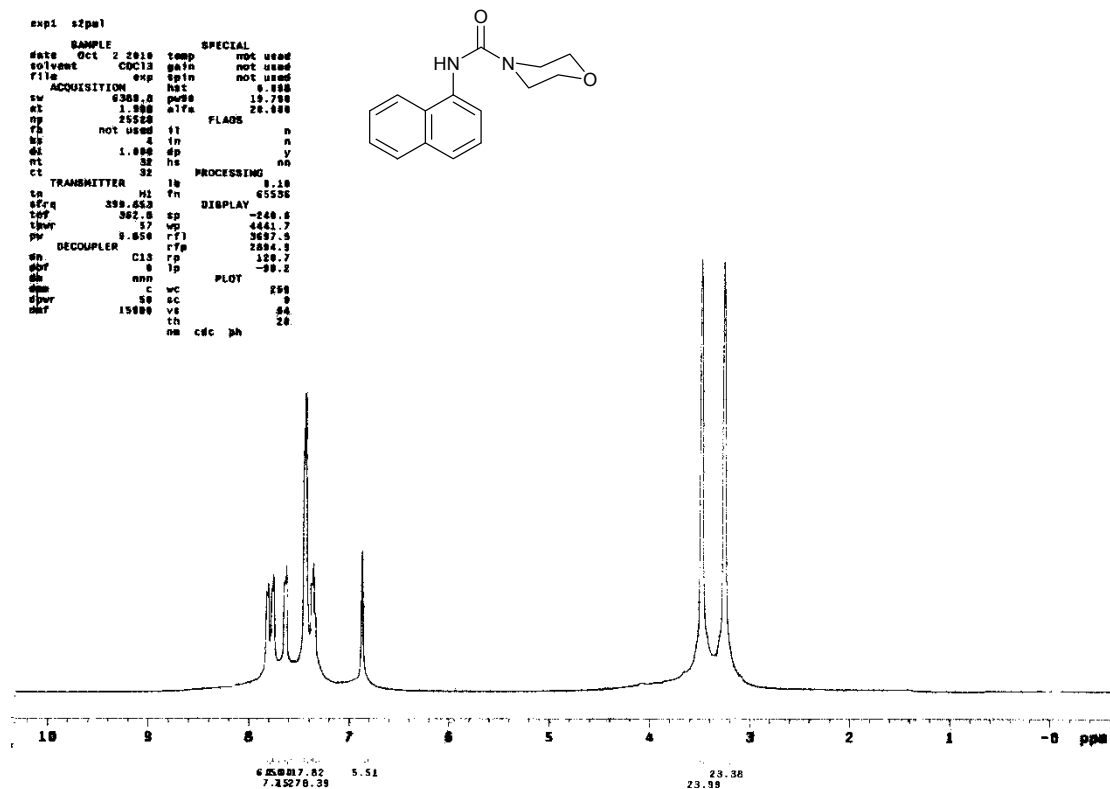
Morpholine-4-carboxylic acid butylamide (23a): ¹³C NMR (CDCl₃, 100 MHz)



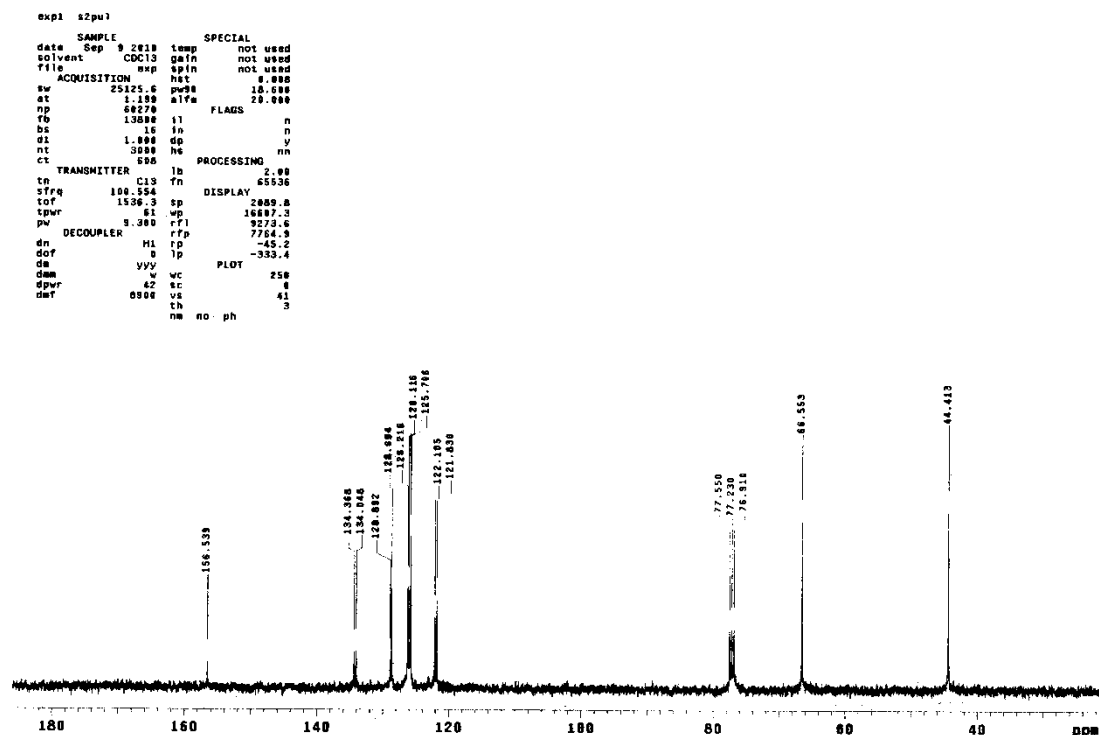
Morpholine-4-carboxylic acid butylamide (23a): IR (KBr)



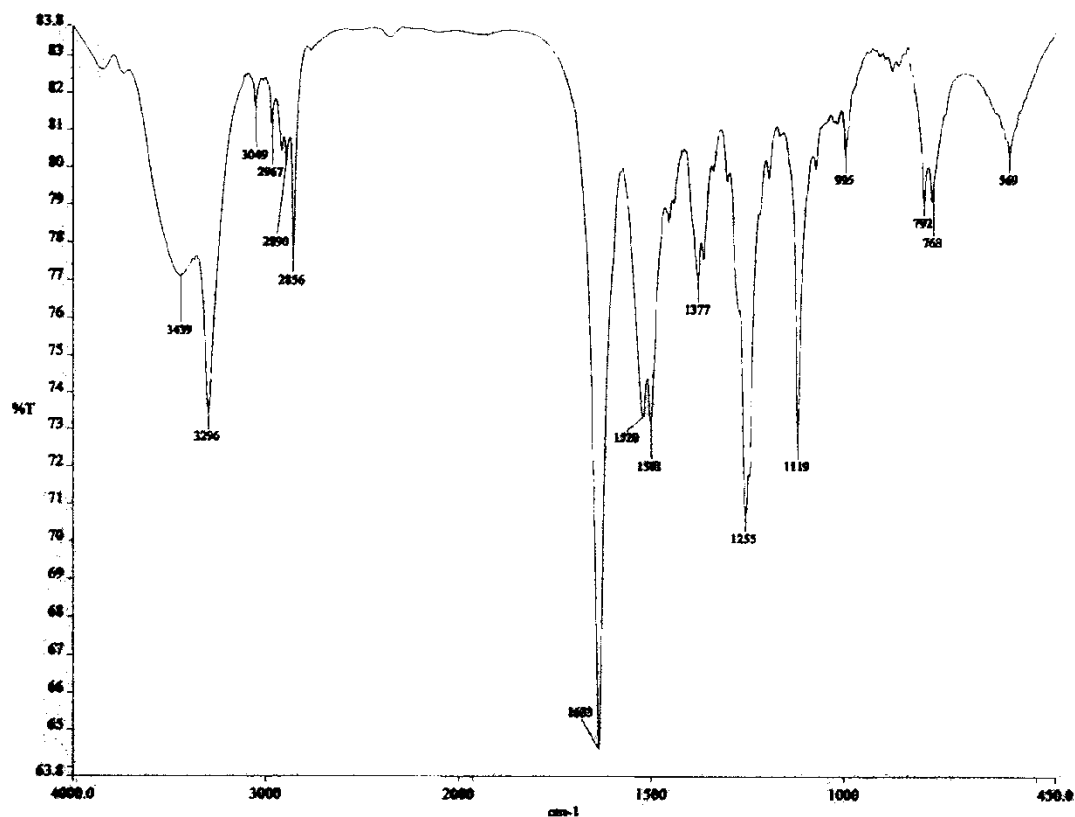
Morpholine-4-carboxylic acid naphthalen-2-ylamide (24a): ¹H NMR (CDCl₃, 400 MHz)



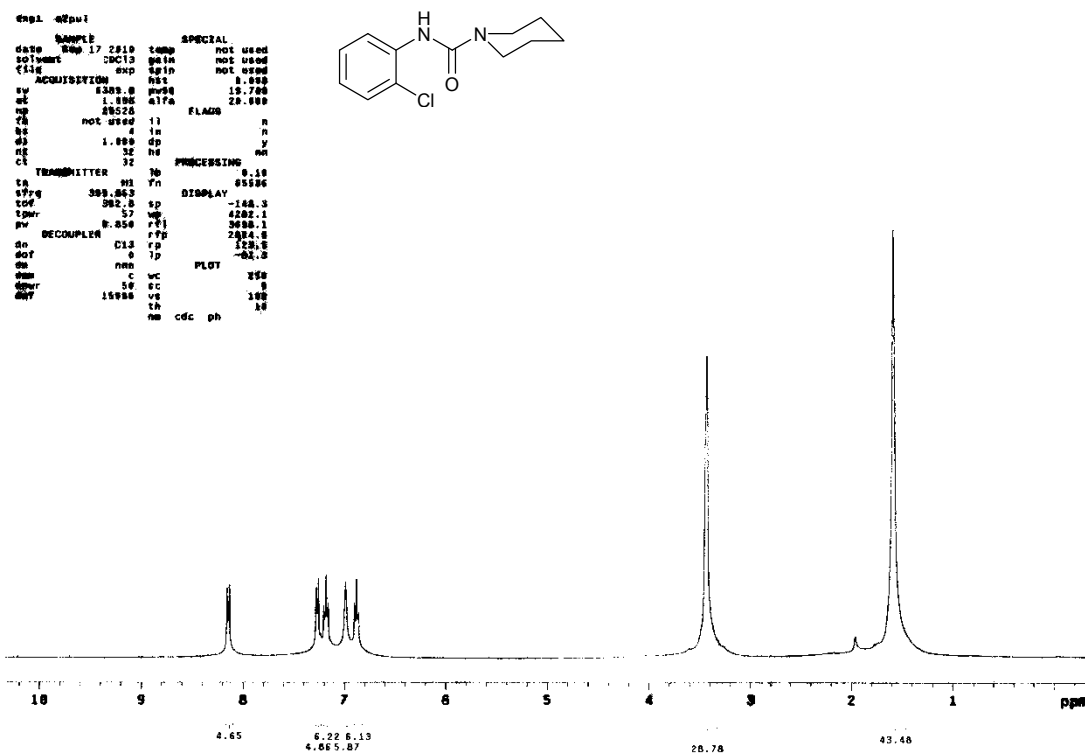
Morpholine-4-carboxylic acid naphthalen-2-ylamide (24a): ^{13}C NMR (CDCl_3 , 100 MHz)



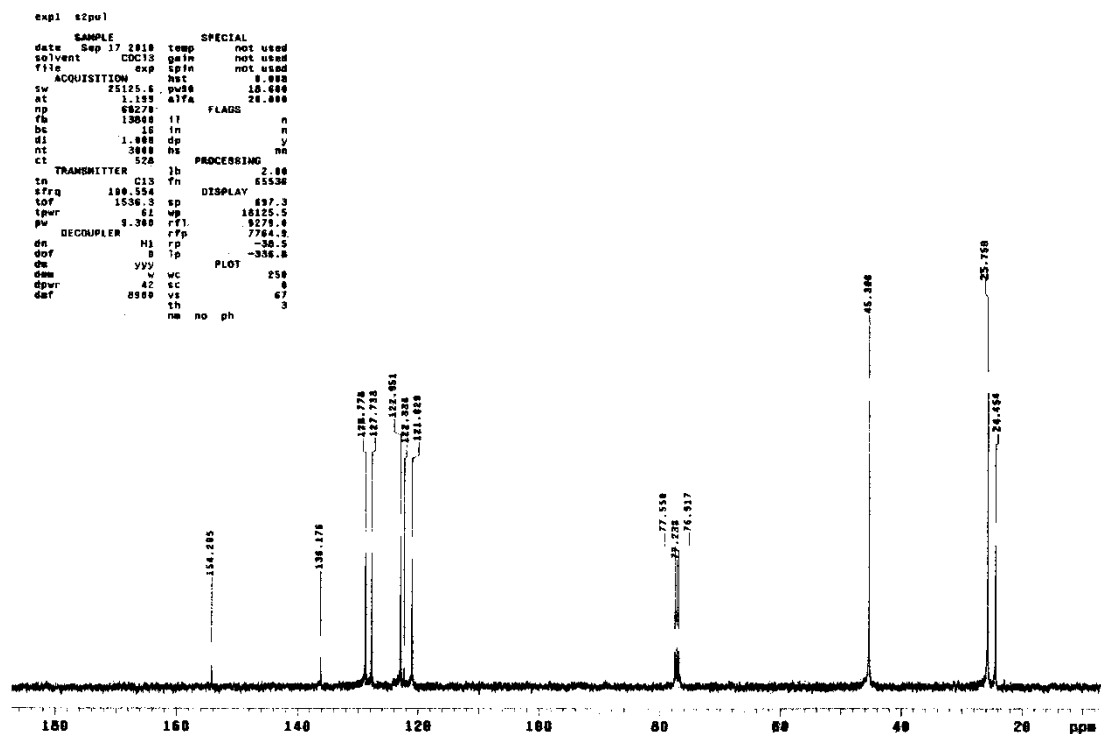
Morpholine-4-carboxylic acid naphthalen-2-ylamide (24a): IR (KBr)



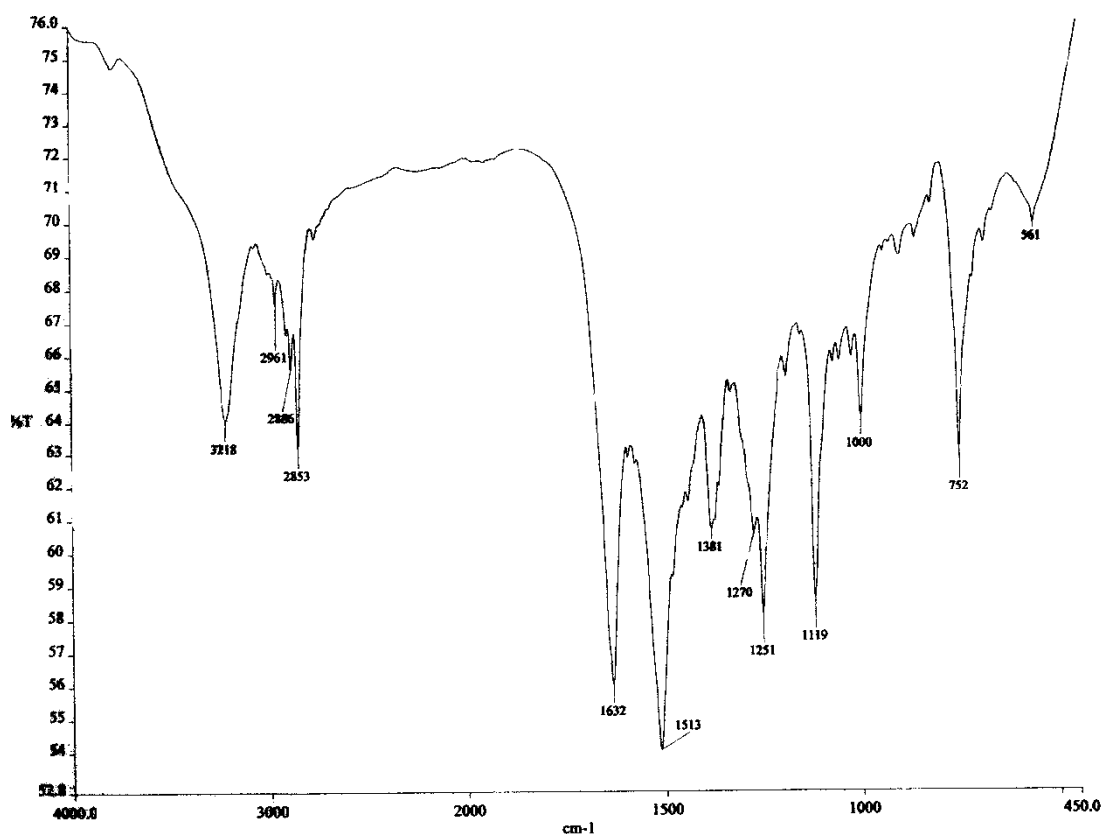
Piperidine-1-carboxylic acid (2-chloro-phenyl)-amide (18b): ^1H NMR (CDCl_3 , 400 MHz)



Piperidine-1-carboxylic acid (2-chloro-phenyl)-amide (18b): ^{13}C NMR (CDCl_3 , 100 MHz)



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

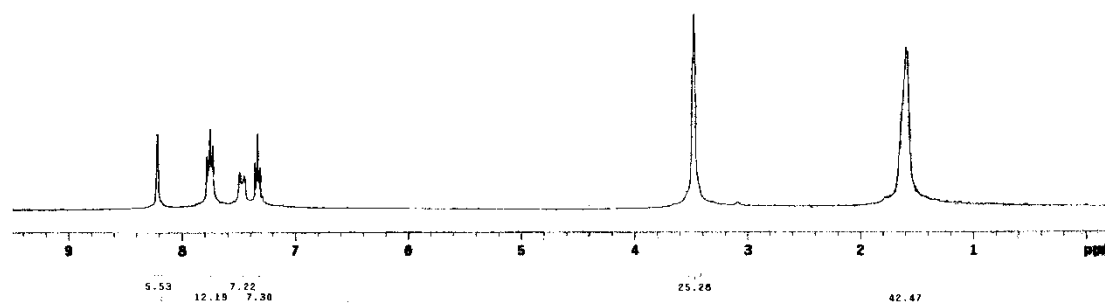
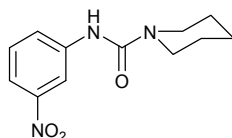


Piperidine-4-carboxylic acid (3-nitro-phenyl)-amide (25b): ¹H NMR (CDCl₃, 400 MHz)

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nt 32 iv
cs 32
TRANSMITTER 1h
sc 389.053 fN
str 362.0 sp
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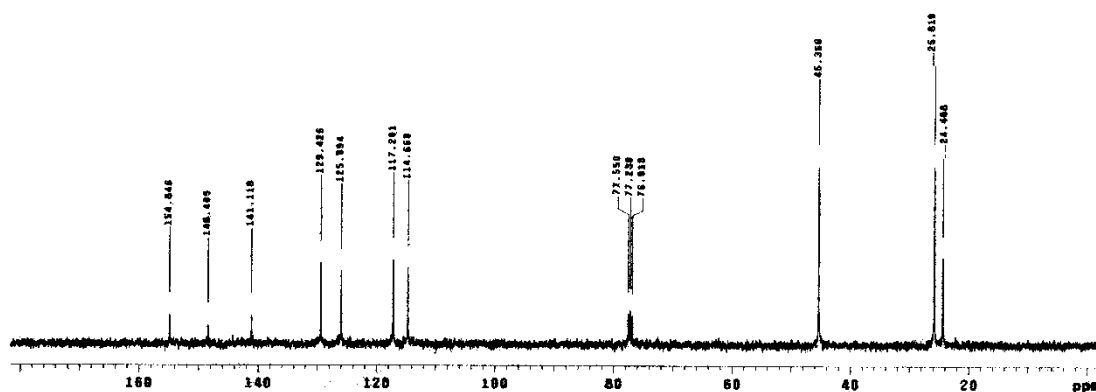


Morpholine-4-carboxylic acid (3-nitro-phenyl)-amide (25b): ^{13}C NMR (CDCl_3 , 100 MHz)

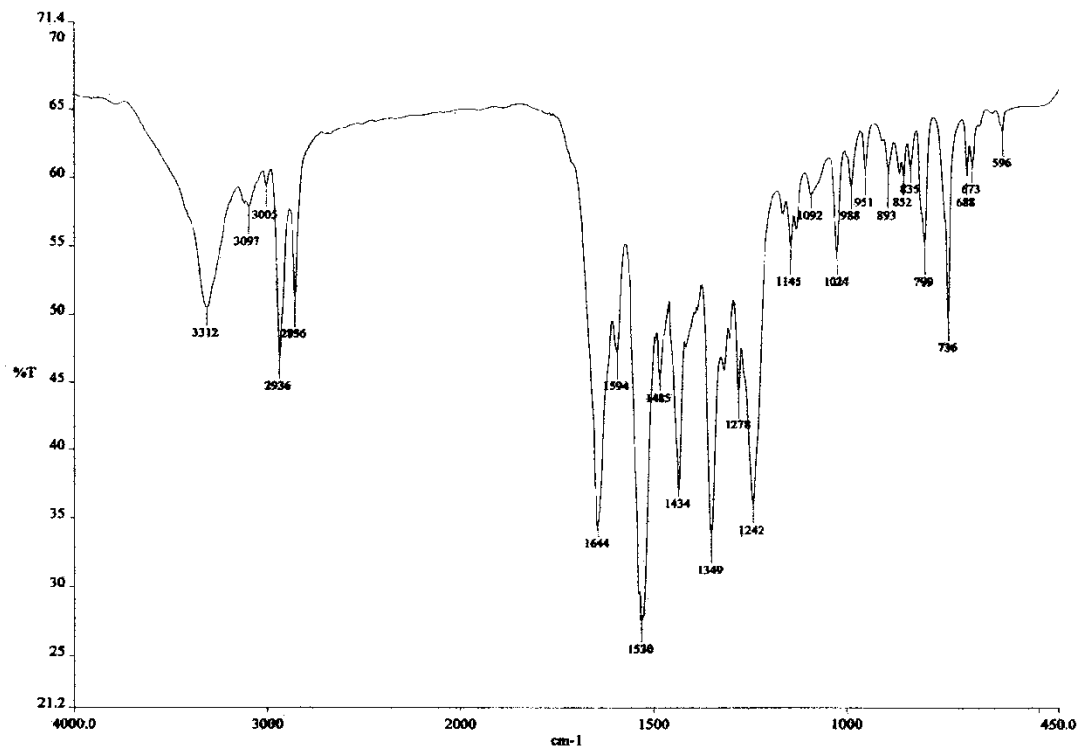
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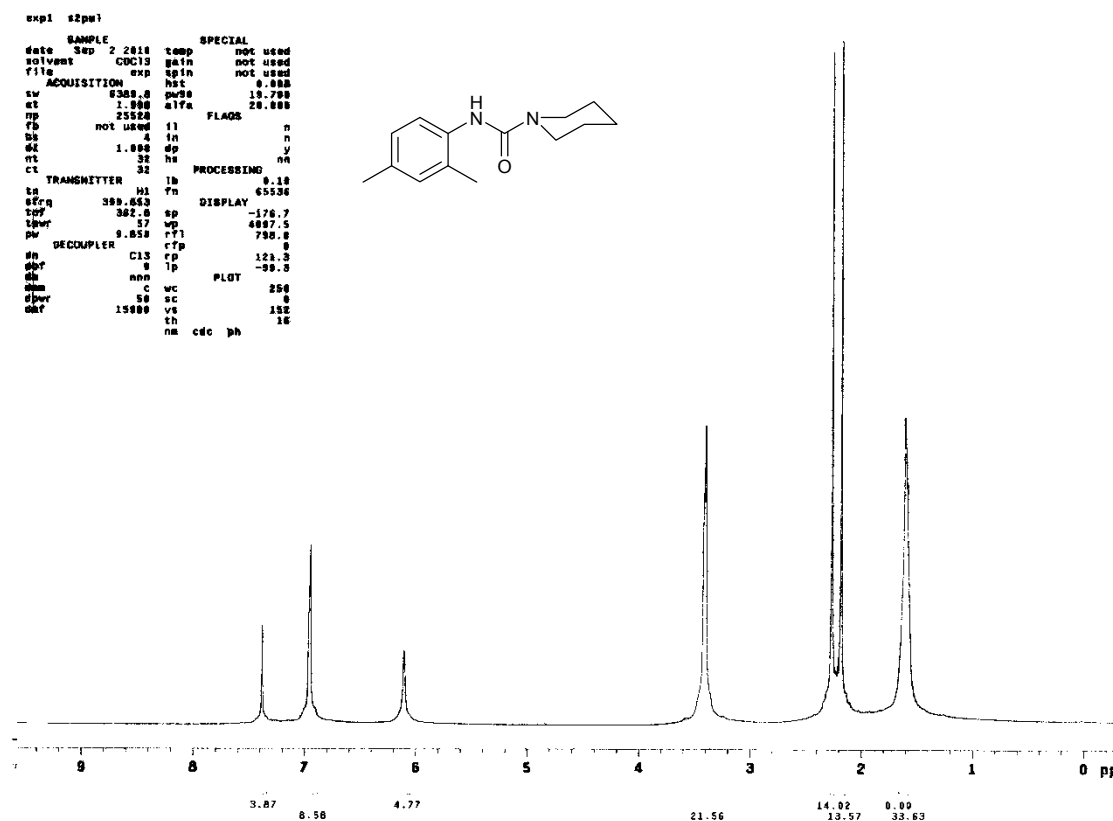
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nm no ph
    
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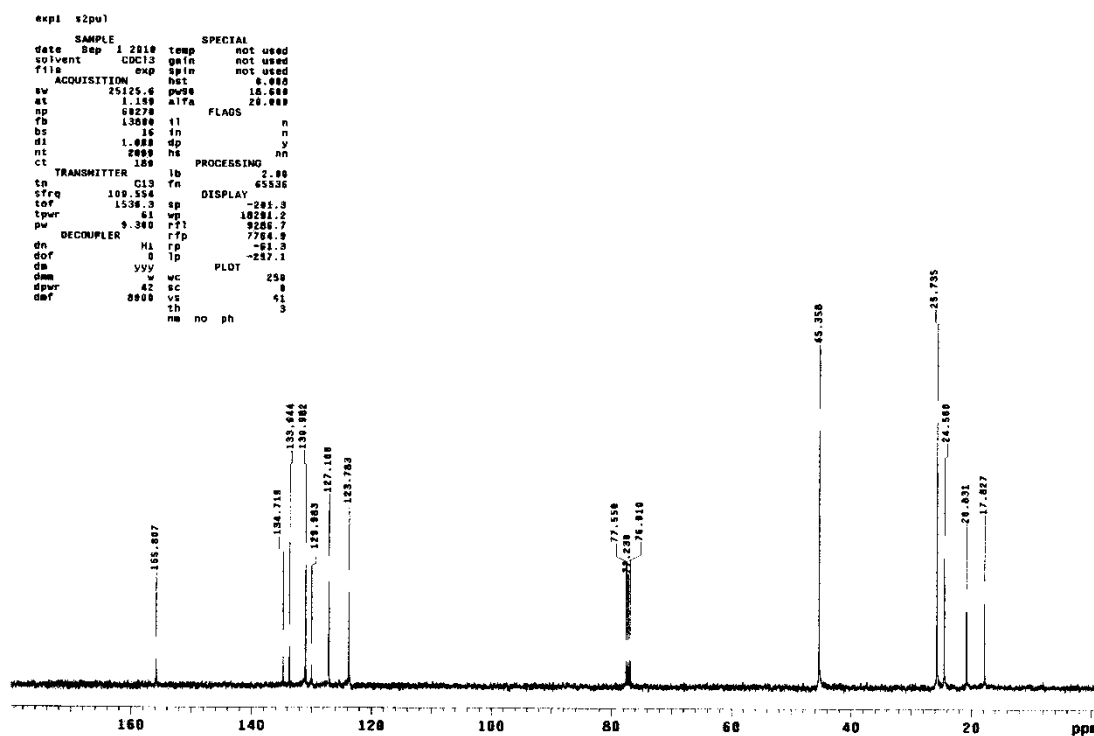
Morpholine-4-carboxylic acid (3-nitro-phenyl)-amide (25b): IR (KBr)



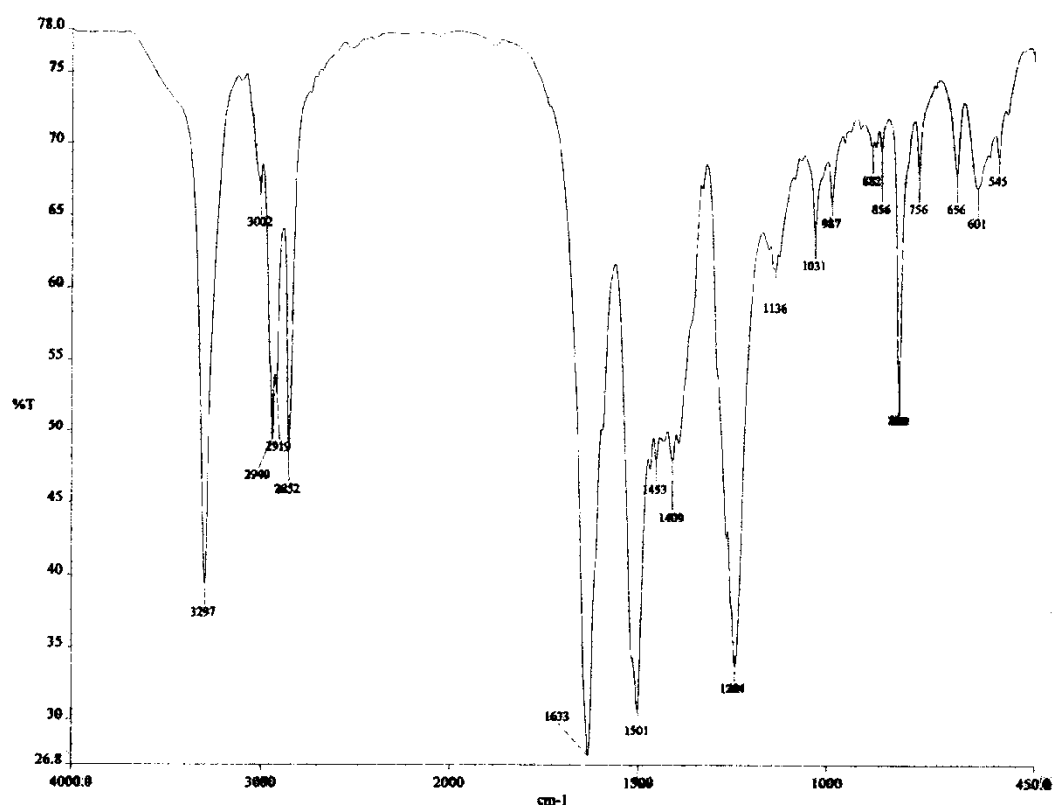
Piperidine-1-carboxylic acid (2,4-dimethyl-phenyl)-amide (21b): ^1H NMR
(CDCl_3 , 400 MHz)



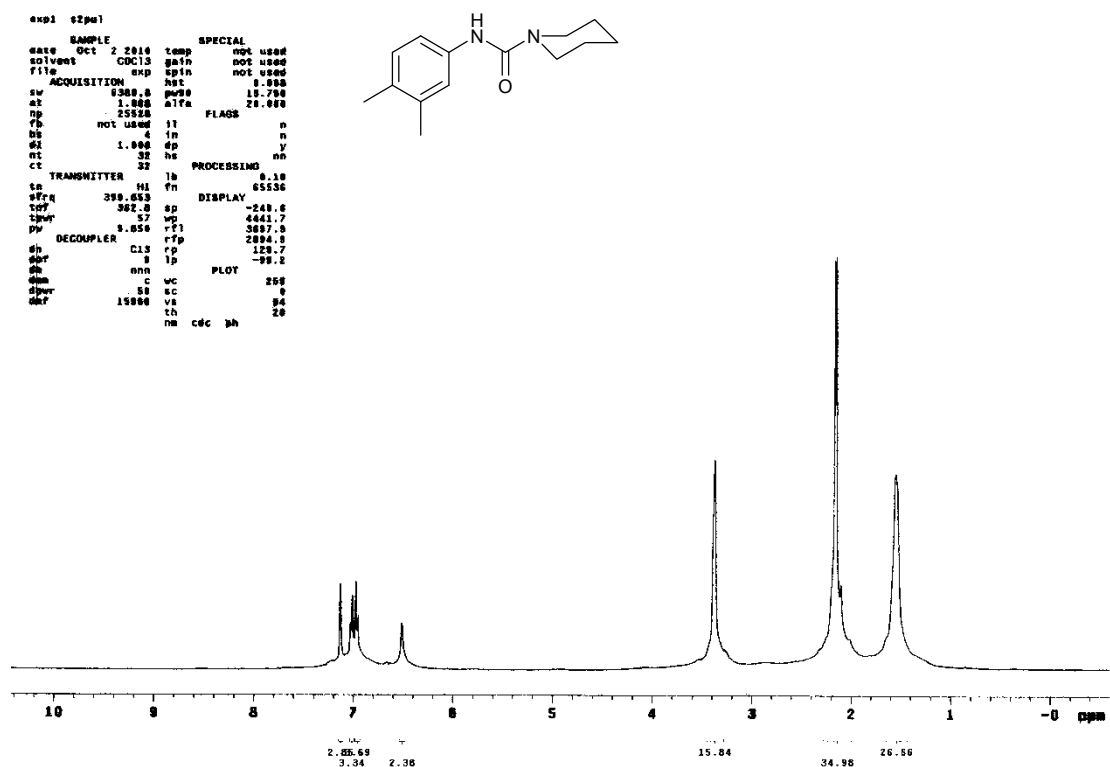
Piperidine-1-carboxylic acid (2,4-dimethyl-phenyl)-amide (21b): ^{13}C NMR
(CDCl_3 , 100 MHz)



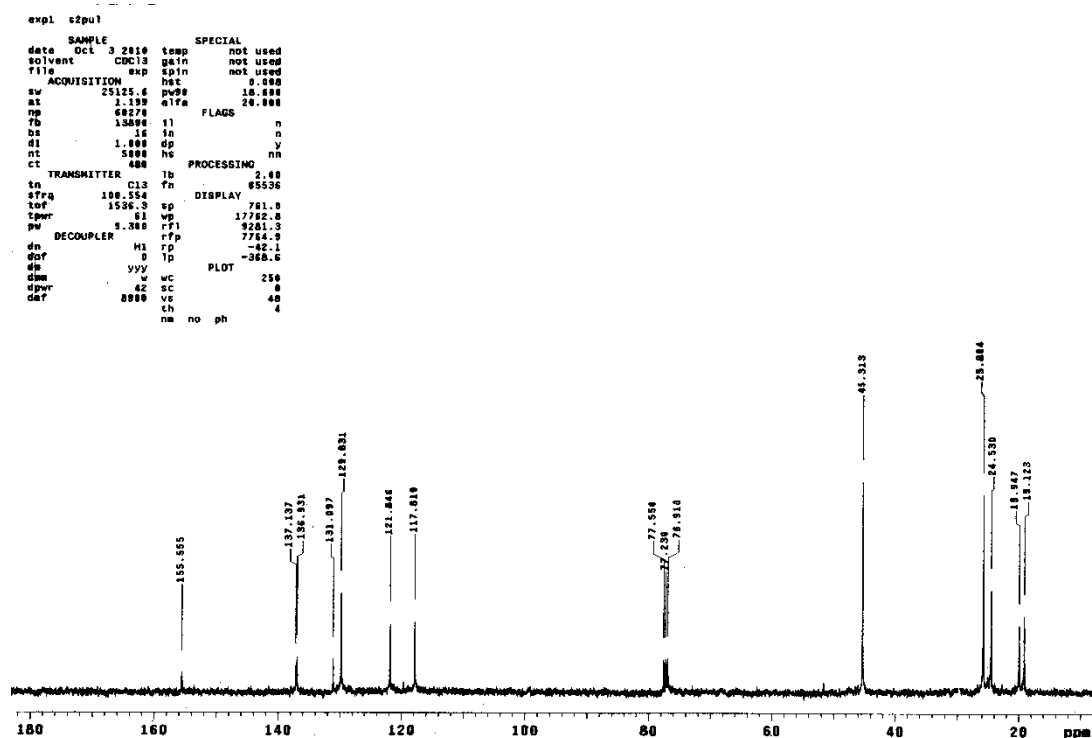
Piperidine-1-carboxylic acid (2,4-dimethyl-phenyl)-amide (21b): IR(KBr)



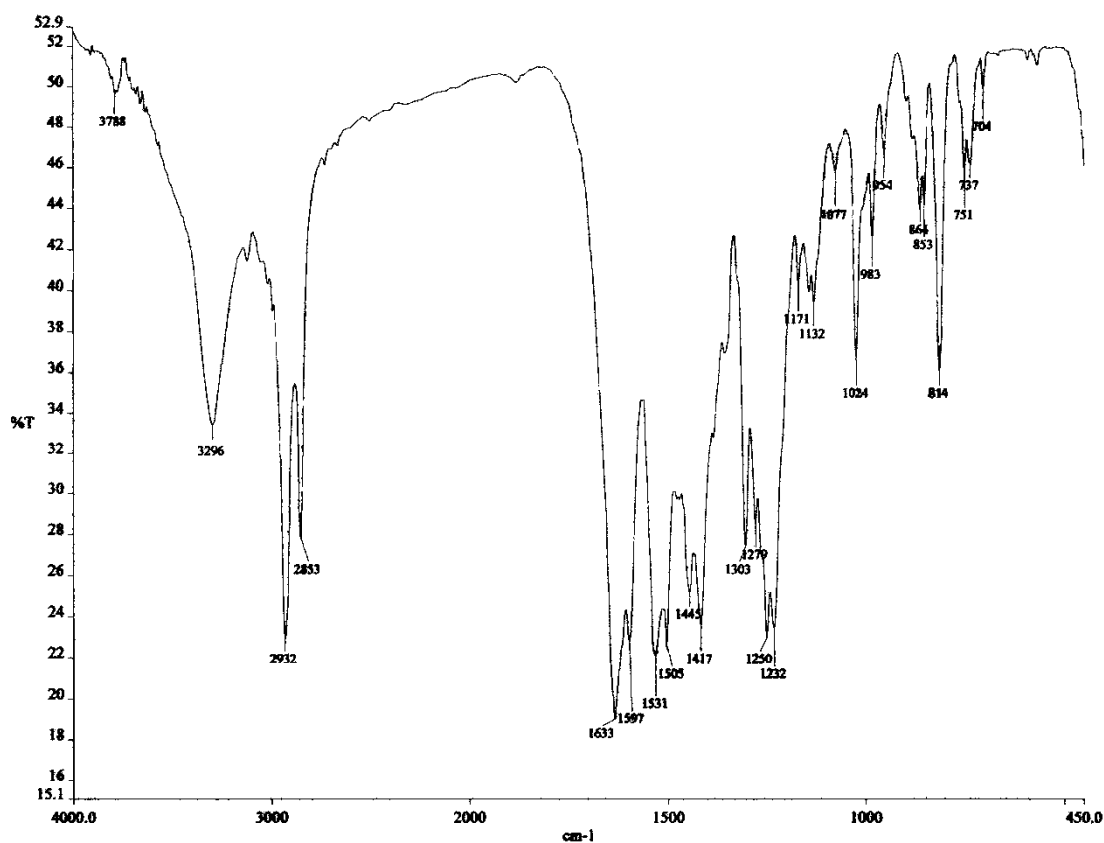
Piperidine-1-carboxylic acid (3,4-dimethyl-phenyl)-amide (26b): ^1H NMR (CDCl₃, 400 MHz)



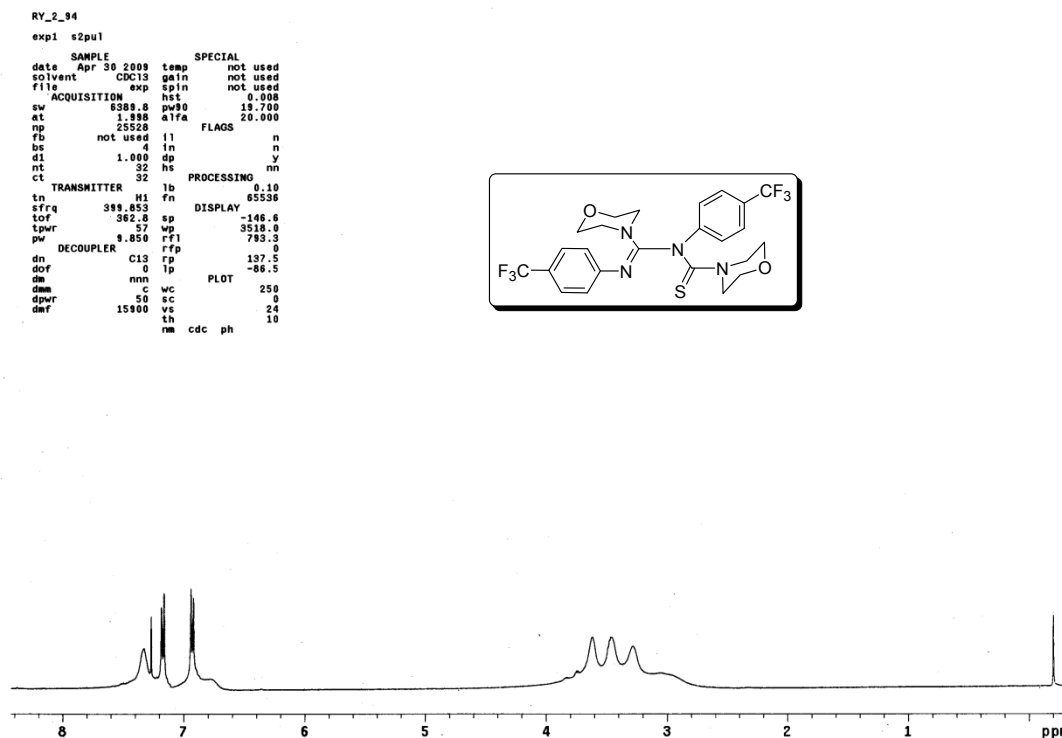
Piperidine-1-carboxylic acid (3,4-dimethyl-phenyl)-amide (26b): ^{13}C NMR
(CDCl_3 , 100 MHz)



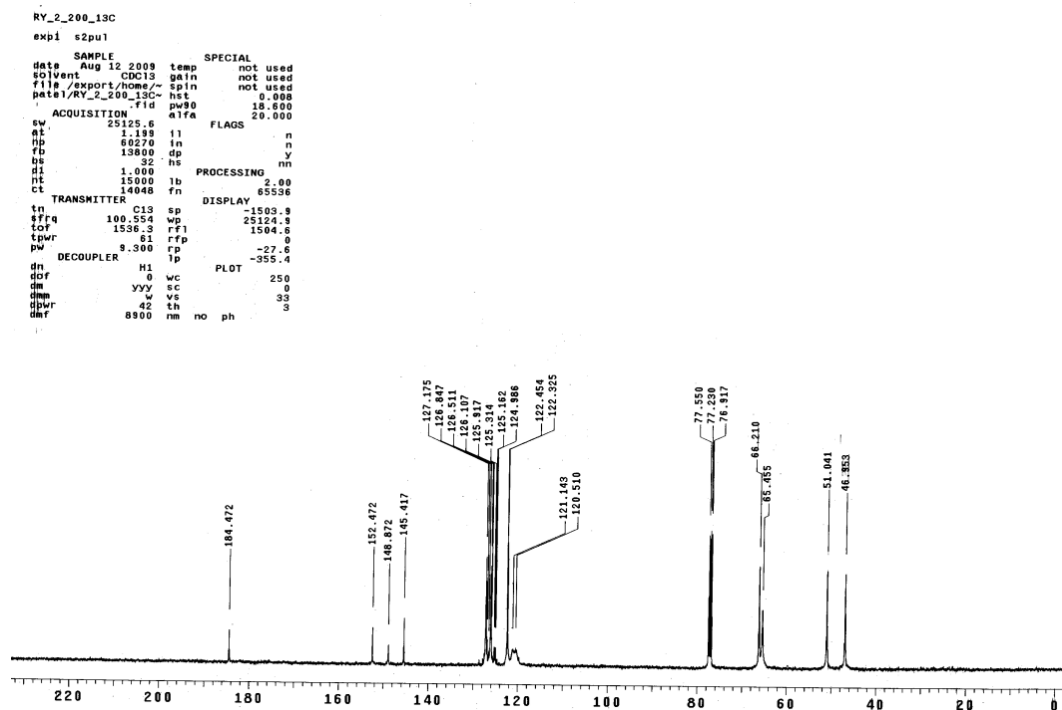
Piperidine-1-carboxylic acid (3,4-dimethyl-phenyl)-amide (26b): IR (KBr)



***N*-((*E*)-(4-(Trifluoromethyl)phenylimino)(morpholino)methyl)-*N*-(4-(trifluoromethyl)phenyl) morpholine-4-carbothioamide (11): ¹H NMR (CDCl₃, 400 MHz):**



***N*-((*E*)-(4-(Trifluoromethyl)phenylimino)(morpholino)methyl)-*N*-(4-(trifluoromethyl)phenyl)morpholine-4-carbothioamide (11): ¹³C NMR (CDCl₃, 100 MHz):**



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

