

Supporting Information for

Synthesis of Secondary Arylamines through Copper-Mediated Intermolecular Aryl Amination

Kentaro Okano, Hidetoshi Tokuyama, and Tohru Fukuyama*

*Graduate School of Pharmaceutical Sciences, The University of Tokyo
7-3-1 Hongo, Bunkyo-ku, Tokyo 113-0033, Japan*

General Remarks: All reactions were carried out in oven-dried glassware under a slight positive pressure of argon unless otherwise noted. $^1\text{H-NMR}$ (400 Hz), and $^{13}\text{C-NMR}$ (100 Hz) spectra were determined on a JEOL JNM-LA400. Chemical shifts for $^1\text{H-NMR}$ are reported in parts per million (ppm) downfield from tetramethylsilane as the internal standard and coupling constants are in Hertz (Hz). The following abbreviations are used for spin multiplicity: s = singlet, d = doublet, t = triplet, q = quartet, m = multiplet, and br = broad. Chemical shifts for $^{13}\text{C-NMR}$ are reported in ppm, relative to the central line of a triplet at 77.0 ppm for deuteriochloroform. Infrared (IR) spectra were recorded on a JASCO FT/IR-410 Fourier Transform Infrared Spectrometer and are reported in wavenumbers (cm^{-1}). Where noted “neat”, the sample was loaded as a thin film on zinc-selenium plate. Optical rotations were measured on a JASCO DIP-1000 digital polarimeter with a sodium lamp. Melting points (mp) were measured on a YANACO MP-500V. High-resolution mass spectra (HRMS) were obtained on a JEOL JMS-700 in positive FAB ionization method, using Kanto Chemical’s PEG 400 or 200 matrix as the internal standard. Analytical thin layer chromatography (TLC) separations were performed on Merck precoated analytical plates, 0.25 mm thick, silica gel 60 F₂₅₄. Preparative TLC separations were performed on Merck analytical plates (0.25 or 0.50 mm thick) precoated with silica gel 60 F₂₅₄. Flash column chromatography separations were performed on Kanto Chemical Silica Gel 60 (40 – 100 mesh). DMF and DMSO was distilled from calcium hydride and was kept over molecular sieves 4Å, which was degassed through three to five cycles of freeze-pump-thaw prior to use. Use of completely degassed solvent and elimination of water from reaction system ensure satisfactory yields. Cesium acetate was weighed under argon atmosphere¹ to prevent absorption of moisture.

Materials: Copper iodide (99.5% purity unless otherwise noted) and cesium acetate were purchased from Wako Pure Chemical Industries, Ltd. and were used as supplied. Substrates were prepared according to the procedures outlined below. Spectroscopic data for all compounds are also listed below.

General Procedures of Amination of Aryl Iodides: An oven-dried pyrex screw tube was charged with CsOAc (219.3 mg, 1.14 mmol, 2.5 eq), CuI (86.8 mg, 0.456 mmol, 1.0 eq) and a small amount of dry benzene (ca. 0.3 mL). The tube was evacuated and backfilled with argon. To the mixture were added degassed DMF (0.46 mL), iodobenzene (50.8 μ L, 0.456 mmol, 1.0 eq), butylamine (90.1 μ L, 0.912 mmol, 2.0 eq). The reaction mixture was heated (color of the solution tuned to be deep blue), then stirred at 90 °C for 12 h. After cooling to room temperature, to the resulting mixture were added ethyl acetate and ammoniacal aqueous NaCl. The mixture was shaken vigorously to dissolve the precipitate, then extracted with ethyl acetate 3 times. The combined organic extracts were dried over Na₂SO₄, filtered, and concentrated *in vacuo*. The crude material was purified by column chromatography (hexane : dichloromethane = 4 : 1 to 1 : 1, gradient) to afford **1a** (63.3 mg, 0.424 mmol, 93% yield).

A Large-Scale Reaction: An oven-dried pyrex screw tube was charged with CsOAc (8.71 g, 45.4 mmol, 2.5 eq), CuI (3.46 g, 18.2 mmol, 1.0 eq, 95% purity). The tube was evacuated and backfilled with argon. To the mixture were added non-degassed dried DMSO (18 mL), iodobenzene (2.03 mL, 18.2 mmol, 1.0 eq), butylamine (3.60 mL, 36.4 mmol, 2.0 eq). The reaction mixture was stirred at 90 °C for 24 h (color of the solution tuned to be deep blue). After cooling to room temperature, to the resulting mixture were added ethyl acetate and ammoniacal aqueous NaCl. The mixture was separated, and the aqueous layer was extracted with ethyl acetate 3 times. The combined organic extracts were washed with brine, dried over Na₂SO₄, filtered, and concentrated *in vacuo*. The crude material (ca. 2.8 g) was purified by column chromatography (hexane : dichloromethane = 4 : 1 to 1 : 1, gradient) to afford **1a** (2.59g, 17.4 mmol, 96% yield).

Butylphenylamine² (1a). Colorless viscous liquid. ¹H NMR (400 MHz, CDCl₃): δ 7.17 (t, 2H, $J = 7.6$ Hz), 6.68 (t, 1H, $J = 7.6$ Hz), 6.60 (d, 2H, $J = 7.6$ Hz), 3.57 (br, 1H), 3.11 (t, 2H, $J = 7.1$

Hz), 1.60 (tt, 2H, $J = 7.1, 7.1$ Hz), 1.43 (tq, 2H, $J = 7.3, 7.1$ Hz), 0.96 (t, 3H, $J = 7.3$ Hz). ^{13}C NMR (100 MHz, CDCl_3): δ 148.5, 129.2, 117.0, 112.7, 43.6, 31.7, 20.3, 13.9. IR (neat, cm^{-1}): 3410, 3052, 3020, 2957, 2930, 2871, 1604, 1506, 1478, 1430, 1321, 1264, 1179, 1153, 992, 867, 748, 692. HRMS-FAB calcd. for $\text{C}_{10}\text{H}_{16}\text{N}$ (M^+H), 150.1282; found 150.1283.

Butyl-(4-methoxy-phenyl)amine (1b). Colorless viscous liquid. ^1H NMR (400 MHz, CDCl_3): δ 6.78 (dd, 2H, $J = 6.6, 2.4$ Hz), 6.58 (dd, 2H, $J = 6.6, 2.4$ Hz), 3.74 (s, 3H), 3.32 (br s, 1H), 3.06 (t, 2H, $J = 7.1$ Hz), 1.59 (tt, 2H, $J = 7.1, 7.1$ Hz), 1.42 (tq, 2H, $J = 7.3, 7.1$ Hz), 0.95 (t, 3H, $J = 7.3$ Hz). ^{13}C NMR (100 MHz, CDCl_3): 151.9, 142.9, 114.9, 114.0, 55.8, 44.7, 31.8, 20.3, 13.9. IR (neat, cm^{-1}): 3392, 2957, 2932, 2872, 2831, 1515, 1464, 1297, 1235, 1180, 1142, 1040, 819. HRMS-FAB calcd. for $\text{C}_{11}\text{H}_{18}\text{NO}$ (M^+H), 180.1388; found 180.1384.

Butyl-*p*-tolylamine (1c). Colorless viscous liquid. ^1H NMR (400 MHz, CDCl_3): δ 6.98 (d, 2H, $J = 8.5$ Hz), 6.53 (d, 2H, $J = 8.5$ Hz), 3.42 (brs, 1H), 3.08 (t, 2H, $J = 7.1$ Hz), 2.23 (s, 3H), 1.58 (tt, 2H, $J = 7.1, 7.1$ Hz), 1.41 (tq, 2H, $J = 7.2, 7.1$ Hz), 0.95 (t, 3H, $J = 7.2$ Hz). ^{13}C NMR (100 MHz, CDCl_3): 146.3, 129.7, 126.2, 112.9, 44.0, 31.7, 20.33, 20.28, 13.9. IR (neat, cm^{-1}): 3408, 2957, 2929, 2871, 1619, 1522, 1480, 1318, 1301, 1260, 1182, 807. HRMS-FAB calcd. for $\text{C}_{11}\text{H}_{18}\text{N}$ (M^+H), 164.1439; found 164.1441.

Butyl-(4-nitro-phenyl)amine³ (1d). Yellow crystals; mp 54-56 °C (dichloromethane-hexane). ^1H NMR (400 MHz, CDCl_3): δ 8.07 (d, 2H, $J = 9.3$ Hz), 6.52 (d, 2H, $J = 9.3$ Hz), 4.65 (brs, 1H), 3.21 (t, 2H, $J = 7.1$ Hz), 1.64 (tt, 2H, $J = 7.1, 7.1$ Hz), 1.43 (tq, 2H, $J = 7.3, 7.1$), 0.97 (t, 3H, $J = 7.3$ Hz). ^{13}C NMR (100 MHz, CDCl_3): 153.5, 137.5, 126.4, 110.8, 43.0, 31.1, 20.1, 13.7. IR (neat, cm^{-1}): 3364, 2960, 2932, 2871, 1615, 1538, 1500, 1475, 1329, 1191, 1141, 1116, 909, 834, 754, 737, 661. HRMS-FAB calcd. for $\text{C}_{10}\text{H}_{15}\text{N}_2\text{O}_2$ (M^+H), 195.1133; found 195.1125.

(4-Bromo-phenyl)butylamine (1e). Colorless viscous liquid. ^1H NMR (400 MHz, CDCl_3): δ 7.22 (dd, 2H, $J = 6.8, 1.7$ Hz), 6.45 (dd, 2H, $J = 6.8, 1.7$ Hz), 3.60 (brs, 1H), 3.04 (t, 2H, $J = 7.1$ Hz), 1.57 (tt, 2H, $J = 7.1, 7.1$ Hz), 1.40 (tq, 2H, $J = 7.3, 7.1$ Hz), 0.94 (t, 3H, $J = 7.3$ Hz). ^{13}C NMR (100 MHz, CDCl_3): δ 147.4, 131.8, 114.1, 108.4, 43.6, 31.4, 20.2, 13.8. IR (neat, cm^{-1}): 3414, 2957, 2930, 2871, 1596, 1499, 1318, 1293, 1262, 1177, 1072, 999, 812. HRMS-FAB

calcd. for $C_{10}H_{15}NBr$ ($M^+ + H$), 228.0388; found 228.0394.

(4-Iodo-phenyl)butylamine (1f). Colorless viscous liquid. 1H NMR (400 MHz, $CDCl_3$): δ 7.40 (d, 2H, $J = 6.8$ Hz), 6.38 (d, 2H, $J = 6.8$ Hz), 3.63 (br s, 1H), 3.07 (t, 2H, $J = 7.1$ Hz), 1.58 (tt, 2H, $J = 7.1, 7.1$ Hz), 1.41 (tq, 2H, $J = 7.3, 7.1$ Hz), 0.95 (t, 2H, $J = 7.3$ Hz). ^{13}C NMR (100 MHz, $CDCl_3$): δ 148.0, 137.7, 129.0, 114.9, 43.5, 31.5, 20.2, 13.9. IR (neat, cm^{-1}): 3412, 2956, 2929, 2871, 1592, 1496, 1318, 1293, 1181, 996, 809. HRMS-FAB calcd. for $C_{10}H_{14}NI$ ($M^+ + H$), 276.0249; found 276.0253.

Butyl-(3-methoxy-phenyl)amine (1g). Colorless viscous liquid. 1H NMR (400 MHz, $CDCl_3$): δ 7.06 (t, 1H, $J = 8.1$ Hz), 6.25 (dd, 1H, $J = 8.1, 2.2$ Hz), 6.21 (dd, 1H, $J = 8.1, 2.2$ Hz), 6.15 (dd, 1H, $J = 2.2, 2.2$ Hz), 3.76 (brs, 1H), 3.08 (t, 2H, $J = 7.1$ Hz), 1.58 (tt, 2H, $J = 7.1, 7.1$ Hz), 1.41 (tq, 2H, $J = 7.1, 7.3$ Hz), 0.94 (t, 3H, $J = 7.3$ Hz). ^{13}C NMR (100 MHz, $CDCl_3$): δ 160.8, 149.9, 129.8, 105.8, 102.0, 98.5, 55.0, 43.6, 31.6, 20.2, 13.8. IR (neat, cm^{-1}): 3404, 2957, 2931, 2871, 1616, 1512, 1497, 1464, 1342, 1301, 1211, 1162, 1051, 828, 756, 688. HRMS-FAB calcd. for $C_{11}H_{18}NO$ ($M^+ + H$), 180.1388; found 180.1385.

Butyl-(3-methyl-phenyl)-amine (1h). Colorless viscous liquid. 1H NMR (400 MHz, $CDCl_3$): δ 7.05 (td, 1H, $J = 7.1, 1.2$ Hz), 6.51 (d, 1H, $J = 7.3$ Hz), 6.43-6.38 (m, 2H), 3.52 (brs, 1H), 3.09 (t, 2H, $J = 7.1$ Hz), 2.22 (s, 3H), 1.59 (tt, 2H, $J = 7.1, 7.1$ Hz), 1.44 (tq, 2H, $J = 7.1, 7.3$ Hz), 0.95 (t, 3H, $J = 7.3$ Hz). ^{13}C NMR (100 MHz, $CDCl_3$): δ 148.6, 138.9, 129.0, 118.0, 113.4, 109.8, 43.6, 31.7, 21.6, 20.3, 13.9. IR (neat, cm^{-1}): 3408, 2957, 2929, 2871, 1606, 1589, 1511, 1491, 1328, 1304, 1180, 992, 843, 768, 692. HRMS-FAB calcd. for $C_{11}H_{18}N$ ($M^+ + H$), 164.1439; found 164.1436.

Butyl-(3-iodo-phenyl)-amine (1i). Colorless viscous liquid. 1H NMR (400 MHz, $CDCl_3$): δ 6.99 (dd, 1H, $J = 7.6, 0.7$ Hz), 6.94-6.92 (m, 1H), 6.85 (dd, 1H, $J = 8.3, 7.6$ Hz), 6.53 (dd, 1H, $J = 8.3, 2.2$ Hz), 3.61 (br s, 1H), 3.06 (t, 2H, $J = 7.1$ Hz), 1.58 (tt, 2H, $J = 7.1, 7.1$ Hz), 1.42 (tq, 2H, $J = 7.1, 7.3$ Hz), 0.96 (t, 3H, $J = 7.3$ Hz). ^{13}C NMR (100 MHz, $CDCl_3$): δ 149.7, 130.6, 125.8, 121.0, 112.0, 95.3, 43.4, 31.4, 20.2, 13.8. IR (neat, cm^{-1}): 3411, 2956, 2928, 2871, 1592, 1568, 1498, 1478, 1327, 983, 839, 762, 682. HRMS-FAB calcd. for $C_{10}H_{15}NI$ ($M^+ + H$), 276.0249;

found 276.0252

Butyl-(2-methoxy-phenyl)amine (1j). Colorless liquid. ^1H NMR (400 MHz, CDCl_3): δ 6.87 (td, 1H, $J = 7.6, 1.2$ Hz), 6.76 (dd, 1H, $J = 7.6, 1.2$ Hz), 6.65 (td, 1H, $J = 7.6, 1.2$ Hz), 6.61 (dd, 1H, $J = 7.6, 1.2$ Hz), 4.15 (brs, 1H), 3.84 (s, 3H), 3.12 (t, 2H, $J = 7.1$ Hz), 1.64 (tt, 2H, $J = 7.1, 7.1$ Hz), 1.44 (tq, 2H, $J = 7.1, 7.3$ Hz), 0.96 (t, 3H, $J = 7.3$ Hz). ^{13}C NMR (100 MHz, CDCl_3): δ 146.7, 138.5, 121.3, 116.0, 109.7, 109.3, 55.4, 43.4, 31.6, 20.4, 13.9. IR (neat, cm^{-1}): 3425, 2958, 1603, 1521, 1456, 1429, 1342, 1244, 1222, 1177, 1143, 1109, 1031, 733. HRMS-FAB calcd. for $\text{C}_{11}\text{H}_{18}\text{NO}$ ($\text{M}^+\text{+H}$), 180.1388; found 180.1381.

Butyl-(2-methyl-phenyl)amine⁴ (1k). Colorless viscous liquid. ^1H NMR (400 MHz, CDCl_3): δ 7.12 (dd, 1H, $J = 7.8, 7.8$ Hz), 7.04 (d, 1H, $J = 7.3$ Hz), 6.66-6.59 (m, 2H), 3.43 (br s, 1H), 3.15 (t, 2H, $J = 7.1$ Hz), 2.13 (s, 3H), 1.65 (tt, 2H, $J = 7.1, 7.1$ Hz), 1.45 (tq, 2H, $J = 7.3, 7.1$ Hz), 0.97 (t, 3H, $J = 7.3$ Hz). ^{13}C NMR (100 MHz, CDCl_3): δ 146.4, 130.0, 127.1, 121.6, 116.6, 109.6, 43.6, 31.7, 20.4, 17.4, 13.9. IR (neat, cm^{-1}): 3432, 2957, 2930, 2872, 1607, 1515, 1473, 1317, 1264, 745. HRMS-FAB calcd. for $\text{C}_{11}\text{H}_{18}\text{N}$ ($\text{M}^+\text{+H}$), 164.1439; found 164.1433.

Butyl-(2-nitro-phenyl)amine (1l). Yellow liquid. ^1H NMR (400 MHz, CDCl_3): δ 8.17 (dd, 1H, $J = 8.5, 1.4$ Hz), 8.06 (br s, 1H), 7.43 (t, 1H, $J = 7.8$ Hz), 6.85 (d, 1H, $J = 8.5$ Hz), 6.29 (td, 1H, $J = 7.8, 1.4$ Hz), 3.31 (dt, 2H, $J = 12.3, 7.1$ Hz), 1.72 (tt, 2H, $J = 7.1, 7.1$ Hz), 1.49 (tq, 2H, $J = 7.1, 7.3$ Hz), 0.99 (t, 3H, $J = 7.3$ Hz). ^{13}C NMR (100 MHz, CDCl_3): δ 145.6, 136.2, 131.7, 126.9, 115.0, 113.8, 42.7, 31.0, 20.2, 13.8. IR (neat, cm^{-1}): 3384, 2958, 2874, 1620, 1575, 1514, 1419, 1355, 1262, 1232, 1159, 1038, 742. HRMS-FAB calcd. for $\text{C}_{10}\text{H}_{15}\text{N}_2\text{O}_2$ ($\text{M}^+\text{+H}$), 195.1133; found 195.1130.

Butyl-(2-fluoro-phenyl)amine (1m). Colorless viscous liquid. ^1H NMR (400 MHz, CDCl_3): δ 7.02-6.92 (m, 2H), 6.69 (dd, 1H, $J = 8.4, 8.4$ Hz), 6.63-6.56 (m, 1H), 3.84 (br s, 1H), 3.14 (t, 2H, $J = 7.1$ Hz), 1.63 (tt, 2H, $J = 7.1, 7.1$ Hz), 1.44 (tq, 2H, $J = 7.3, 7.1$ Hz), 0.96 (t, 3H, $J = 7.3$ Hz). ^{13}C NMR (100 MHz, CDCl_3): δ 151.5 ($^1J_{\text{C-F}} = 237$ Hz), 137.0 ($J_{\text{C-F}} = 12$ Hz), 124.5 ($J_{\text{C-F}} = 3$ Hz), 116.1 ($J_{\text{C-F}} = 7$ Hz), 114.2 ($J_{\text{C-F}} = 19$ Hz), 111.9 ($J_{\text{C-F}} = 4$ Hz), 43.3, 31.5, 20.2, 13.9. IR (neat, cm^{-1}): 3432, 3069, 2959, 2931, 2873, 1621, 1524, 1469, 1448, 1337, 1246, 1189, 739.

HRMS-FAB calcd. for $C_{10}H_{14}FN$ ($M^+ + H$), 168.1188; found 168.1185.

***N*¹-(4-methoxyphenethyl)-*N*³-butylbenzene-1,3-diamine (2).** Pale yellow liquid. ¹H NMR (400 MHz, CDCl₃): δ 7.14 (d, 2H, *J* = 8.7 Hz), 6.97 (t, 1H, *J* = 7.9 Hz), 6.85 (d, 2H, *J* = 8.7 Hz), 6.02-5.97 (m, 2H), 5.86 (dd, 1H, *J* = 2.2, 2.2 Hz), 3.79 (s, 3H), 3.54 (br s, 1H), 3.33 (t, 2H, *J* = 7.1 Hz), 3.07 (t, 2H, *J* = 7.1 Hz), 2.84 (t, 2H, *J* = 7.1 Hz), 1.58 (tt, 2H, *J* = 7.1, 7.1 Hz), 1.41 (tq, 2H, *J* = 7.1, 7.3 Hz), 0.95 (t, 3H, *J* = 7.3 Hz). ¹³C NMR (100 MHz, CDCl₃): 158.2, 149.7, 149.2, 131.4, 129.9, 129.7, 114.0, 103.0, 102.8, 97.2, 55.2, 45.3, 43.7, 34.7, 31.7, 20.3, 13.9. IR (neat, cm⁻¹): 3400, 2955, 2930, 2858, 2834, 1612, 1512, 1464, 1246, 1177, 1166, 1035, 822, 755, 689. HRMS-FAB calcd. for $C_{19}H_{27}N_2O$ ($M^+ + H$), 299.2123; found 299.2130.

1-(4-Nitro-phenyl)-pyrrolidine⁵ (3a). Yellow crystals; mp 163-165 °C (dichloromethane-hexane). ¹H NMR (400 MHz, CDCl₃): δ 8.12 (d, 2H, *J* = 9.3 Hz), 6.47 (d, 2H, *J* = 9.3 Hz), 3.40 (t, 4H, *J* = 6.6 Hz), 2.12-2.04 (m, 4H). ¹³C NMR (100 MHz, CDCl₃): δ 151.8, 136.5, 126.3, 110.4, 47.9, 25.4. IR (neat, cm⁻¹): 2976, 2859, 1610, 1572, 1475, 1466, 1438, 1407, 1322, 1196, 1180, 1158, 1113, 822, 754. HRMS-FAB calcd. for $C_{12}H_{13}N_2O_2$ ($M^+ + H$), 193.0977; found 193.0972.

1-(4-Nitro-phenyl)-1*H*-indole⁶ (3b). Yellow crystals; mp 132-133 °C (dichloromethane-hexane). ¹H NMR (400 MHz, CDCl₃): δ 8.34 (d, 2H, *J* = 7.3 Hz), 7.69 (d, 1H, *J* = 7.8 Hz), 7.67-7.60 (m, 3H), 7.35 (d, 1H, *J* = 3.6 Hz), 7.28 (t, 1H, *J* = 7.8 Hz), 7.22 (t, 1H, *J* = 7.8 Hz), 6.76 (d, 1H, *J* = 3.6 Hz). ¹³C NMR (100 MHz, CDCl₃): δ 145.1, 144.9, 135.1, 130.0, 127.0, 125.4, 123.3, 123.2, 121.6, 121.5, 110.4, 106.1. IR (neat, cm⁻¹): 3083, 1595, 1518, 1502, 1455, 1335, 1237, 1209, 1135, 1114, 852, 749. HRMS-FAB calcd. for $C_{14}H_{11}N_2O_2$ ($M^+ + H$), 239.0820; found 239.0826.

Diphenylamine⁷ (3c). Colorless crystals; mp 51-52 °C (dichloromethane-hexane). ¹H NMR (400 MHz, CDCl₃): δ 7.25 (t, 4H, *J* = 8.1 Hz), 7.06 (dd, 4H, *J* = 8.1, 1.0 Hz), 6.92 (t, 2H, *J* = 8.1 Hz), 5.67 (br s, 1H). ¹³C NMR (100 MHz, CDCl₃): 143.1, 129.3, 120.1, 117.9. IR (neat, cm⁻¹): 3384, 3043, 3016, 1600, 1506, 1459, 1417, 1312, 1244, 1218, 1174, 1028, 877, 747, 690, 667. HRMS-FAB calcd. for $C_{12}H_{12}N$ ($M^+ + H$), 170.0969; found 170.0968.

Allyl-(4-nitro-phenyl)-amine⁷ (3d). Yellow crystals; mp 75-77 °C (dichloromethane-hexane). ¹H NMR (400 MHz, CDCl₃): δ 8.08 (dd, 2H, *J* = 9.3, 1.0 Hz), 6.55 (d, 2H, *J* = 9.3 Hz), 5.97-5.84 (m, 1H), 5.29 (d, 1H, *J* = 17.1 Hz), 5.23 (dt, 1H, *J* = 9.0, 1.2 Hz), 4.76 (br s, 1H), 3.88 (dd, 2H, *J* = 5.9, 5.1 Hz). ¹³C NMR (100 MHz, CDCl₃): δ 153.2, 138.0, 133.3, 126.3, 117.2, 111.2, 45.7. IR (neat, cm⁻¹): 3360, 1605, 1533, 1308, 1182, 1112, 830, 753. HRMS-FAB calcd. for C₉H₁₁N₂O₂ (M⁺+H), 179.0820; found 179.0822.

Furan-2-ylmethyl-(4-nitro-phenyl)-amine (3e). Yellow crystals; mp 104-105 °C (dichloromethane-hexane). ¹H NMR (400 MHz, CDCl₃): δ 8.08 (dd, 2H, *J* = 9.0, 1.7 Hz), 7.38 (d, 1H, *J* = 0.7 Hz), 6.61 (d, 2H, *J* = 9.0 Hz), 6.37-6.31 (m, 1H), 6.28 (d, 1H, *J* = 3.2 Hz), 4.96 (br s, 1H), 4.41 (d, 2H, *J* = 5.9 Hz). ¹³C NMR (100 MHz, CDCl₃): δ 152.7, 150.7, 142.4, 138.4, 126.3, 111.4, 110.5, 107.8, 40.6. IR (neat, cm⁻¹): 3359, 1599, 1544, 1282, 1187, 1105, 1011, 830, 790, 751, 694. HRMS-FAB calcd. for C₁₁H₁₁N₂O₃ (M⁺+H), 219.0769; found 219.0769.

Butyl-pyridin-2-yl-amine⁸ (3f). Pale beige needles; mp 34-35 °C (dichloromethane-hexane). ¹H NMR (400 MHz, CDCl₃): δ 8.07 (d, 1H, *J* = 5.1 Hz), 7.41 (dd, 1H, *J* = 8.4, 8.4 Hz), 6.54 (dd, 1H, *J* = 6.1, 6.1 Hz), 6.36 (d, 1H, *J* = 8.4 Hz), 4.54 (br s, 1H), 3.24 (dt, 2H, *J* = 6.9, 6.4 Hz), 1.60 (tt, 2H, *J* = 7.1, 7.1 Hz), 1.43 (tq, 2H, *J* = 7.1, 7.3 Hz), 0.95 (t, 3H, *J* = 7.3 Hz). ¹³C NMR (100 MHz, CDCl₃): δ 158.9, 148.1, 137.3, 112.5, 106.2, 41.9, 31.6, 20.2, 13.8. IR (neat, cm⁻¹): 3271, 2958, 2931, 2872, 1604, 1571, 1515, 1451, 1418, 1329, 1288, 1152, 770, 735. HRMS-FAB calcd. for C₉H₁₅N₂ (M⁺+H), 151.1235; found 151.1234.

General Procedure of Introduction of NsNH₂ into Aryl Iodides. An oven-dried pyrex screw tube was charged with Cs₂CO₃ (407 mg, 1.25 mmol, 2.5 eq), CuI (95.2 mg, 0.500 mmol, 1.0 eq), and NsNH₂ (101 mg, 0.500 mmol, 1.0 eq). The tube was evacuated and backfilled with argon. Degassed DMSO (0.50 mL), iodobenzene (111 μL, 1.00 mmol, 2.0 eq) were added to the mixture. Then, the reaction mixture was heated (color of the solution tuned to be brown) to 90 °C and stirred for 18 h. After cooling to room temperature, to the resulting mixture was added dichloromethane and 1 M HCl until pH = 1 (off-white precipitate was formed). The mixture was extracted with dichloromethane 3 times. The combined organic extracts was dried over

Na₂SO₄, filtered and concentrated *in vacuo*. The crude material was purified by column chromatography (hexane : dichloromethane = 2 : 3 to dichloromethane, gradient) to afford **4** (97.4 mg, 0.352 mmol, 70% yield) as a white solid with recovery of NsNH₂ (25.0 mg, 0.124 mmol, 25%)..

N-Phenyl-2-nitrobenzenesulfonamide (4). Colorless crystals; mp 109-110 °C (dichloromethane-hexane). ¹H NMR (400 MHz, CDCl₃): δ 7.85 (dd, 1H, *J* = 8.1, 1.4 Hz), 7.82 (dd, 1H, *J* = 7.9, 1.1 Hz), 7.69 (td, 1H, *J* = 8.1, 1.4 Hz), 7.57 (td, 1H, *J* = 7.9, 1.1 Hz), 7.31-7.23 (m, 3H), 7.22-7.13 (m, 3H). ¹³C NMR (100 MHz, CDCl₃): 148.2, 135.5, 133.9, 132.5, 132.2, 131.8, 129.4, 126.6, 125.3, 123.3. IR (neat, cm⁻¹): 3319, 3100, 2892, 1538, 1496, 1403, 1358, 1168, 1126, 921, 854, 782, 741, 695, 656. HRMS-FAB calcd. for C₁₂H₁₁N₂O₄S (M⁺+H), 279.0439; found 279.0445.

N-(4-Methoxycarbonylphenyl)-2-nitrobenzenesulfonamide (5). Prepared as the above procedure, pale yellow crystals, mp 194-196 °C (dichloromethane-hexane). ¹H NMR (400 MHz, CDCl₃): δ 7.96 (d, 2H, *J* = 8.5 Hz), 7.92 (dd, 1H, *J* = 7.8, 1.2 Hz), 7.87 (dd, 1H, *J* = 7.8, 0.7 Hz), 7.71 (ddd, 1H, *J* = 7.8, 7.8, 1.2 Hz), 7.61 (ddd, 1H, *J* = 7.8, 7.8, 0.7 Hz), 7.45 (br s, 1H), 7.29 (d, 2H, *J* = 8.5 Hz), 3.88 (s, 3H). ¹³C NMR (100 MHz, CDCl₃): 166.2, 139.8, 134.3, 133.4, 132.7, 131.9, 131.8, 131.1, 127.7, 125.5, 121.3, 52.2. IR (neat, cm⁻¹): 3216, 1689, 1607, 1546, 1510, 1430, 1400, 1364, 1294, 1168, 1117, 921, 767. HRMS-FAB calcd. for C₁₄H₁₃N₂O₆S (M⁺+H), 337.0494; found 337.0483.

Methyl 4-(3-phenylpropylamino)benzoate (6). To a mixture of **4** (168 mg, 0.500 mmol), potassium carbonate (207 mg, 1.50 mmol, 3.0 eq), and tetrabutylammonium iodide (18.5 mg, 0.050 mmol, 10 mol%) were added DMF and 3-phenylpropyl bromide (114 μL, 0.750 mmol, 1.5 eq). The reaction mixture was heated to 80 °C for 6 h, then cooled to room temperature. PhSH (1.28 mL, 1.25 mmol, 2.5 eq) was added dropwise to the mixture at room temperature and stirred for 2 h. The resulting mixture was diluted with dichloromethane followed by water, then extracted with dichloromethane 3 times. Combined organic layers were washed with brine, dried over Na₂SO₄. After removal of solvent *in vacuo*, the residual crude oil was purified by column chromatography (hexane : dichloromethane = 4 : 1 to 1 : 4, gradient) to afford **5** (114.8

mg, 85%) as a colorless crystals; mp 87-88 °C (dichloromethane-hexane). ¹H NMR (400 MHz, CDCl₃): δ 7.84 (d, 2H, *J* = 8.8 Hz), 7.31-7.27 (m, 2H), 7.25-7.16 (m, 3H), 6.49 (d, 2H, *J* = 8.8 Hz), 4.12 (br s, 1H), 3.83 (s, 3H), 3.17 (t, 2H, *J* = 7.1 Hz), 2.72 (t, 2H, *J* = 7.1 Hz), 1.95 (quintet, 2H, *J* = 7.1 Hz). ¹³C NMR (100 MHz, CDCl₃): 167.3, 151.9, 141.2, 131.5, 128.5, 128.3, 126.0, 118.1, 111.3, 51.4, 42.7, 33.2, 30.7. IR (neat, cm⁻¹): 3359, 2944, 1684, 1601, 1530, 1477, 1435, 1342, 1279, 1179, 1113, 749, 696. HRMS-FAB calcd. for C₁₇H₂₀NO₂ (M⁺+H), 270.1494; found 270.1506.

(+)-*N*-Nosyl-*N*-((*R*)-2-(*tert*-butyloxycarbonylamino)-2-phenylethyl)benzenamine (7). To a stirred mixture of **4** (111 mg, 0.400 mmol), *N*-Boc-(*R*)-α-phenylglycinol (199 mg, 0.800 mmol, 2.0 eq), triphenylphosphine (210 mg, 0.800 mmol, 2.0 eq), and toluene (5.0 mL), was added DEAD (40% in toluene, 0.35 mL, 0.80 mmol, 2.0 eq) dropwise. Then the reaction mixture was heated at 80 °C for 2 h. After cooled to room temperature, toluene was removed under reduced pressure. The residue was purified by column chromatography (dichloromethane) to afford **6** (190 mg, 95%) as a pale yellow liquid. ¹H NMR (400 MHz, CDCl₃): δ 7.66-7.58 (m, 2H), 7.49-7.40 (m, 2H), 7.36-7.20 (m, 10H), 5.35 (br s, 1H), 4.70 (br s, 1H), 4.22 (m, 1H), 3.77 (dd, 1H, *J* = 14.6, 4.4 Hz), 1.45 (s, 9H). ¹³C NMR (100 MHz, CDCl₃): δ 155.3, 147.9, 139.5, 137.4, 133.6, 132.1, 131.1, 129.8, 129.6, 128.9, 128.7, 127.8, 126.6, 123.8, 79.6, 57.0, 53.1, 28.4 (one carbon is lacking due to overlapping). IR (neat, cm⁻¹): 3423, 3067, 2979, 1713, 1544, 1506, 1495, 1373, 1247, 1169, 1127, 1060, 1025, 912, 852, 779, 733, 697, 653. [α]_D²⁶ = +65 (*c* 1.04, CHCl₃). HRMS-FAB calcd. for C₂₅H₂₈N₃O₆S (M⁺+H), 498.1699; found 498.1682.

(-)-*N*-((*R*)-2-(*tert*-butyloxycarbonylamino)-2-phenylethyl)benzenamine (8). *The reaction should be conducted in a well-ventilated hood.* To a stirred suspension of **7** (91.9 mg, 0.185 mmol), cesium carbonate (121 mg, 0.370 mmol, 2.0 eq), and acetonitrile (2.0 mL) was added PhSH (38 μL, 0.37 mmol, 2.0 eq) at room temperature. The reaction mixture was heated at 50 °C for 2 h. The resulting suspension was filtered through a glass filter, then evaporated to dryness *in vacuo*. The crude material was purified by column chromatography (hexane : dichloromethane = 1 : 1 to dichloromethane, gradient) to give **8** (49.0 mg, 85%) as colorless viscous liquid. ¹H NMR (400 MHz, CDCl₃): δ 7.41-7.34 (m, 2H), 7.33-7.28 (m, 3H), 7.17 (t, 2H, *J* = 7.6 Hz), 6.71 (t, 1H, *J* = 7.6 Hz), 6.61 (d, 2H, *J* = 7.6 Hz), 5.08 (br s, 1H), 4.94 (br s, 1H),

3.82 (br s, 1H), 3.45 (d, 2H, $J = 5.6$ Hz), 1.43 (s, 9H). ^{13}C NMR (100 MHz, CDCl_3): 155.6, 147.8, 140.3, 129.3, 128.9, 127.8, 126.4, 117.9, 113.1, 79.9, 54.2, 49.7, 28.3. IR (neat, cm^{-1}): 3403, 2977, 1700, 1603, 1507, 1392, 1366, 1253, 1169, 750, 700. $[\alpha]_{\text{D}}^{25} = -4.0$ (c 1.44, CHCl_3). HRMS-FAB calcd. for $\text{C}_{19}\text{H}_{25}\text{N}_2\text{O}_2$ ($\text{M}^+\text{+H}$), 313.1916; found 313.1925.

- (1) Transferred in a plastic bag filled with argon.
- (2) Kim, S.; Oh, C. H.; Ko, J. S.; Ahn, K. H.; Kim, Y. J. *J. Org. Chem.* **1985**, *50*, 1927.
- (3) Ibata, T.; Isogami, Y.; Toyoda, J. *Chem. Lett.* **1987**, *6*, 1187.
- (4) Hamann, B. C.; Hartwig, J. F. *J. Am. Chem. Soc.* **1998**, *120*, 3694.
- (5) Hashimoto, S.; Otani, S.; Okamoto, T.; Matsumoto, K. *Heterocycles* **1988**, *27*, 319.
- (6) Abramovitch, R. A.; Miyashita, K. *J. Chem. Soc. Perkin Trans. 1* **1975**, *23*, 2413.
- (7) Ibata, T.; Isogami, Y.; Toyoda, J. *Bull. Chem. Soc. Jpn.* **1991**, *64*, 42.
- (8) Khan, M. A.; Rocha, E. K. *Chem. Pharm. Bull.* **1977**, *25*, 3110.

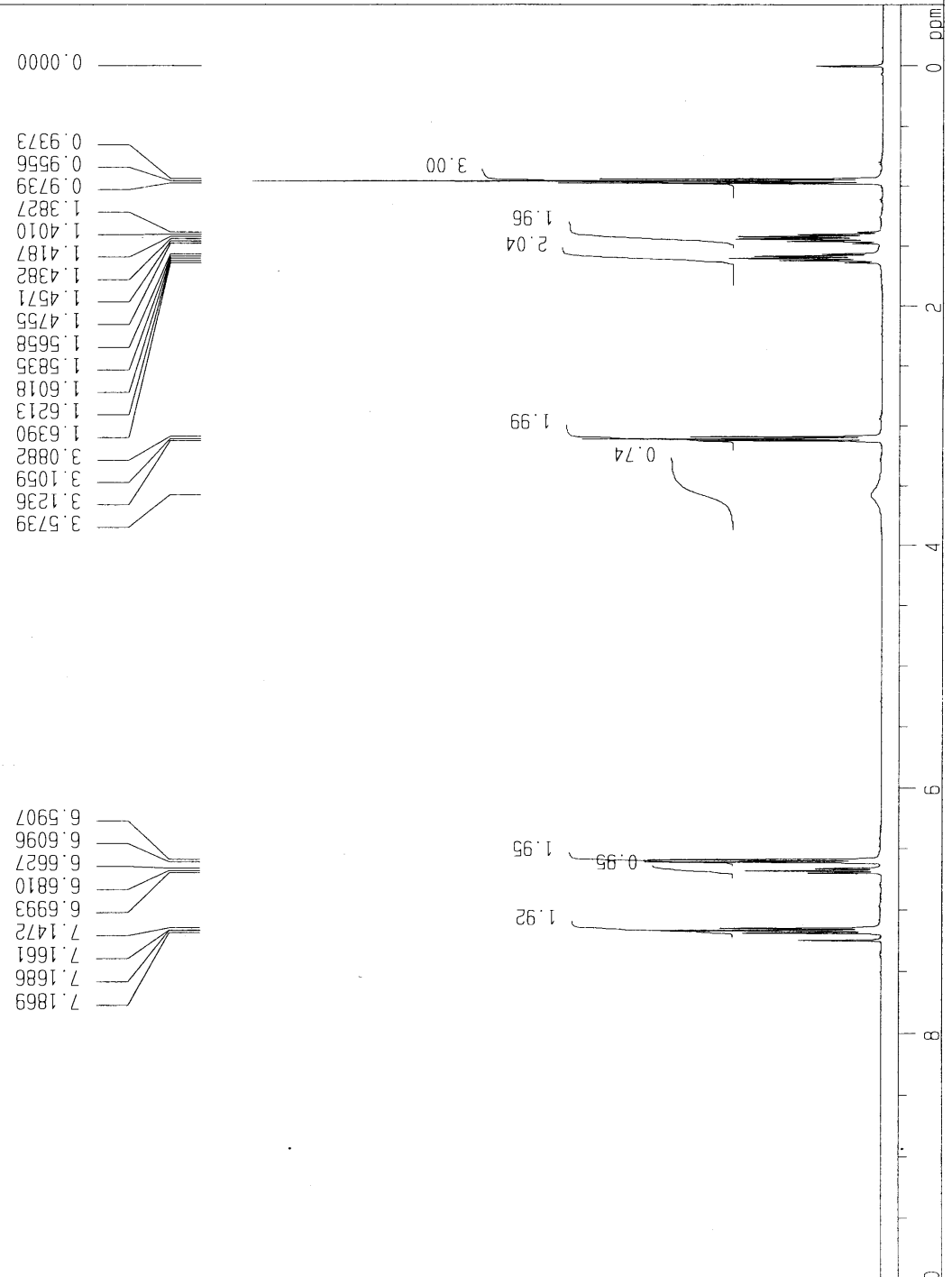
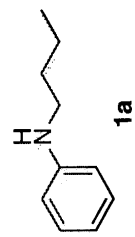
ko01072a'

Date : Mon Aug 4 15:28:01 2003

FileName : ko01072a'.nmdata
Comment : ko01072a'
SliceHistory : non
EXMODE :

POINT : 32768 points
SAMPO : 32768 points
FREQU : 7993.6 Hz
FILTR : 4000 Hz
DELAY : 50.0 usec
DEADT : 72.1 usec
INVL : 125.1 usec
TIMES : 32 times
DUMMY : 0 times
PD : 2.9007 sec
ACQTM : 4099.2769 msec
PREDL : 10.00000 msec
INIWT : 0.5000 msec
RESOL : 0.24 Hz
PW1 : 5.75 usec
OBNUC : ¹H
OBFRQ : 399.65 MHz
OBSET : 134300.00 Hz
RGAIN : 18

SCANS : 32 times
SLVNT : COCL3
SPINNING : 12 Hz
TEMP : 22.4 C



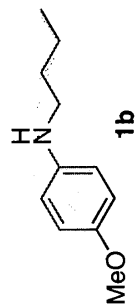
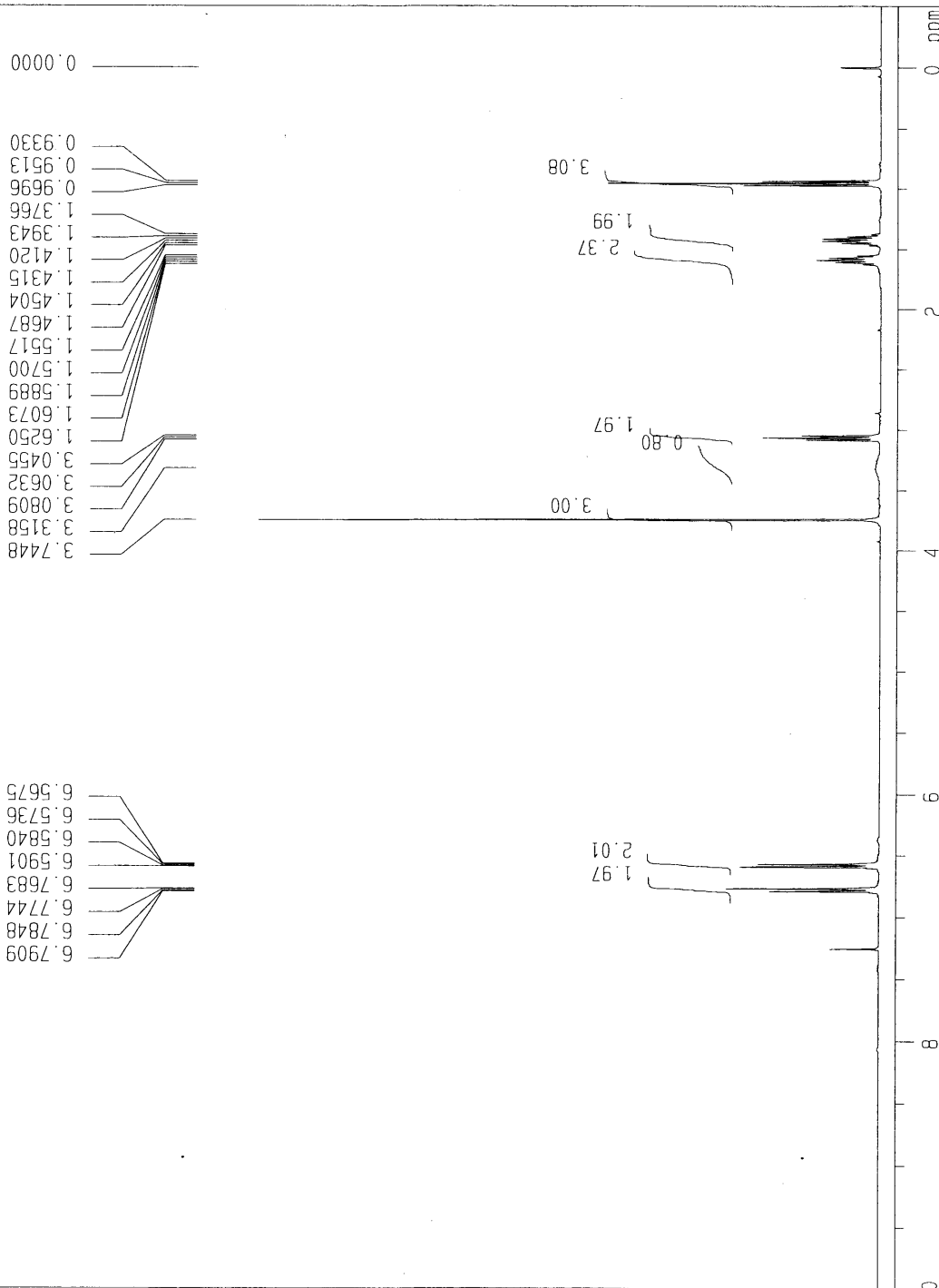
ko01112a''

Date : Tue Aug 5 14: 35: 28 2003

FileName : ko01112a''.nmdata
Comment : ko01112a''
SliceHistory :
EXMODE : non

POINT : 32768 points
SAMP0 : 32768 points
FREGU : 7993.6 Hz
FILTR : 4000 Hz
DELAY : 50.0 usec
DEADT : 72.1 usec
INTVL : 125.1 usec
TIMES : 32 times
DUMMY : 0 times
PD : 2.9007 sec
ACQTM : 4099.2769 msec
PREDL : 10.0000 msec
INIWT : 0.5000 msec
RESOL : 0.24 Hz
PWI : 5.75 usec
OBNUC : 1H
OBFRQ : 399.65 MHz
OBSET : 134300.00 Hz
RGAIN : 18

SCANS : 32 times
SLVNT : CDCL3
SPINNING : 14 Hz
TEMP : 22.3 C

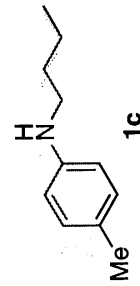


ko01134a'

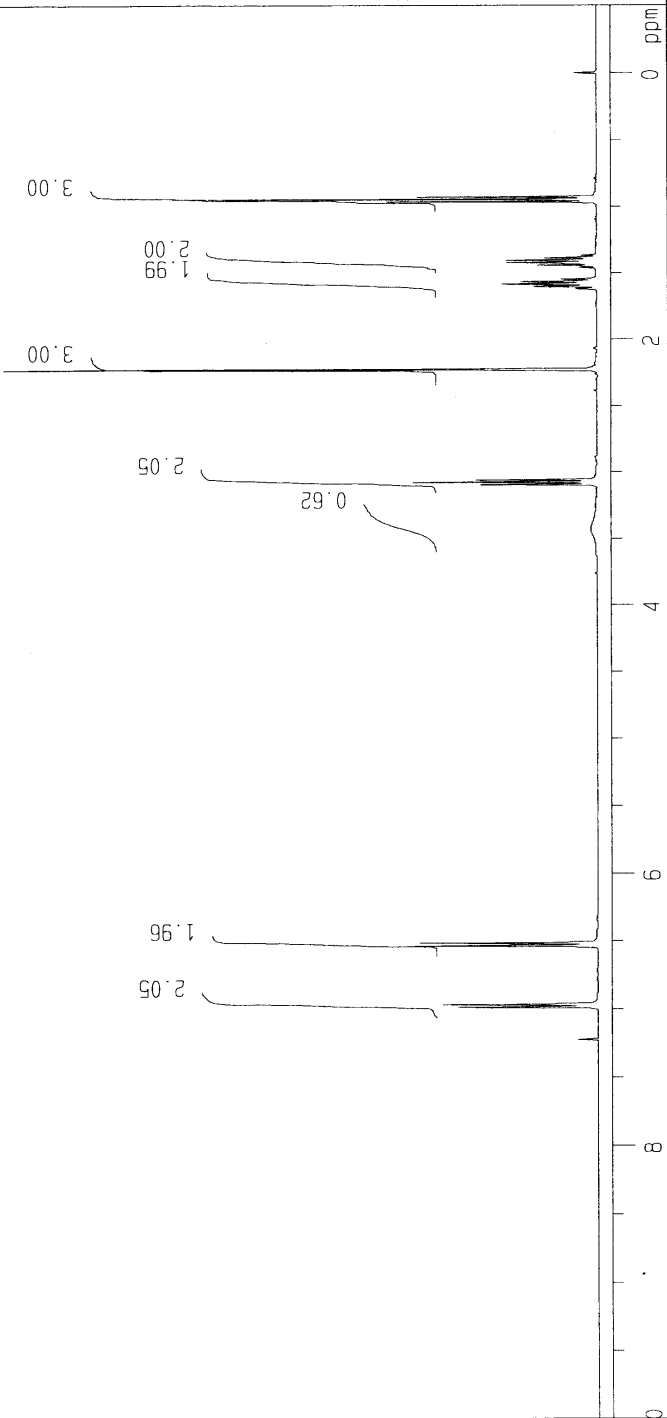
Date : Sat Aug 2 18:46:22 2003

FileName : ko01134a'.nmdata
Comment : ko01134a'
SliceHistory :
EXMODE : non

POINT 32768 points
SAMP0 32768 points
FREQ0 7993.6 Hz
FILTR 4000 Hz
DELAY 50.0 usec
DEADT 72.1 usec
INTVL 125.1 usec
TIMES 32 times
DUMMY 0 times
PD 2.9007 sec
ACQTM 4099.2769 msec
PREDL 10.00000 msec
ININT 0.5000 msec
RESOL 0.24 Hz
PW1 5.75 usec
1H
OBNUC 399.65 MHz
OBFRQ 134300.00 Hz
OBSET 14
RGAIN 14
SCANS 32 times
SLVNT CDCL3
SPINNING 14 Hz
TEMP 21.3 C



3.4201
3.0937
3.0754
3.0577
2.2290
1.6176
1.5999
1.5816
1.5621
1.5444
1.4602
1.4419
1.4224
1.4034
1.3858
1.3681
0.9635
0.9452
0.9269
0.0000



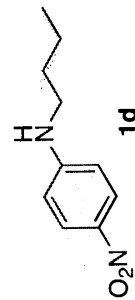
ko01128a-data

Date : Tue Jul 15 02:52:36 2003

File Name : ko01128a-data.nmdata
Comment : ko01128a-data
SliceHistory :
EXMODE : non

POINT 32768 points
SAMP0 32768 points
FREQ0 7993.6 Hz
FILTR 4000 Hz
DELAY 50.0 usec
DEADT 72.1 usec
INIVL 125.1 usec
TIMES 256 times
DUMMY 0 times
PD 2.9007 sec
ACQTM 4099.2769 msec
PREDL 10.00000 msec
INIWT 0.5000 msec
RESOL 0.24 Hz
PW1 5.75 usec
OBNUC 1H
OBFRQ 399.65 MHz
OBSET 134300.00 Hz
RGAIN 15

SCANS 256 times
SLVNT CDCL3
SPINNING 10 Hz
TEMP 22.8 C



3.2322
3.2163
3.1986
3.1809
1.6780
1.6597
1.6414
1.6231
1.6048
1.4852
1.4681
1.4492
1.4303
1.4114
1.3925
1.9873
0.9684
0.9501

4.6497

6.5285
6.5053

8.0784
8.0552

2.93

1.97
1.94

2.00

0.94

1.94

1.94



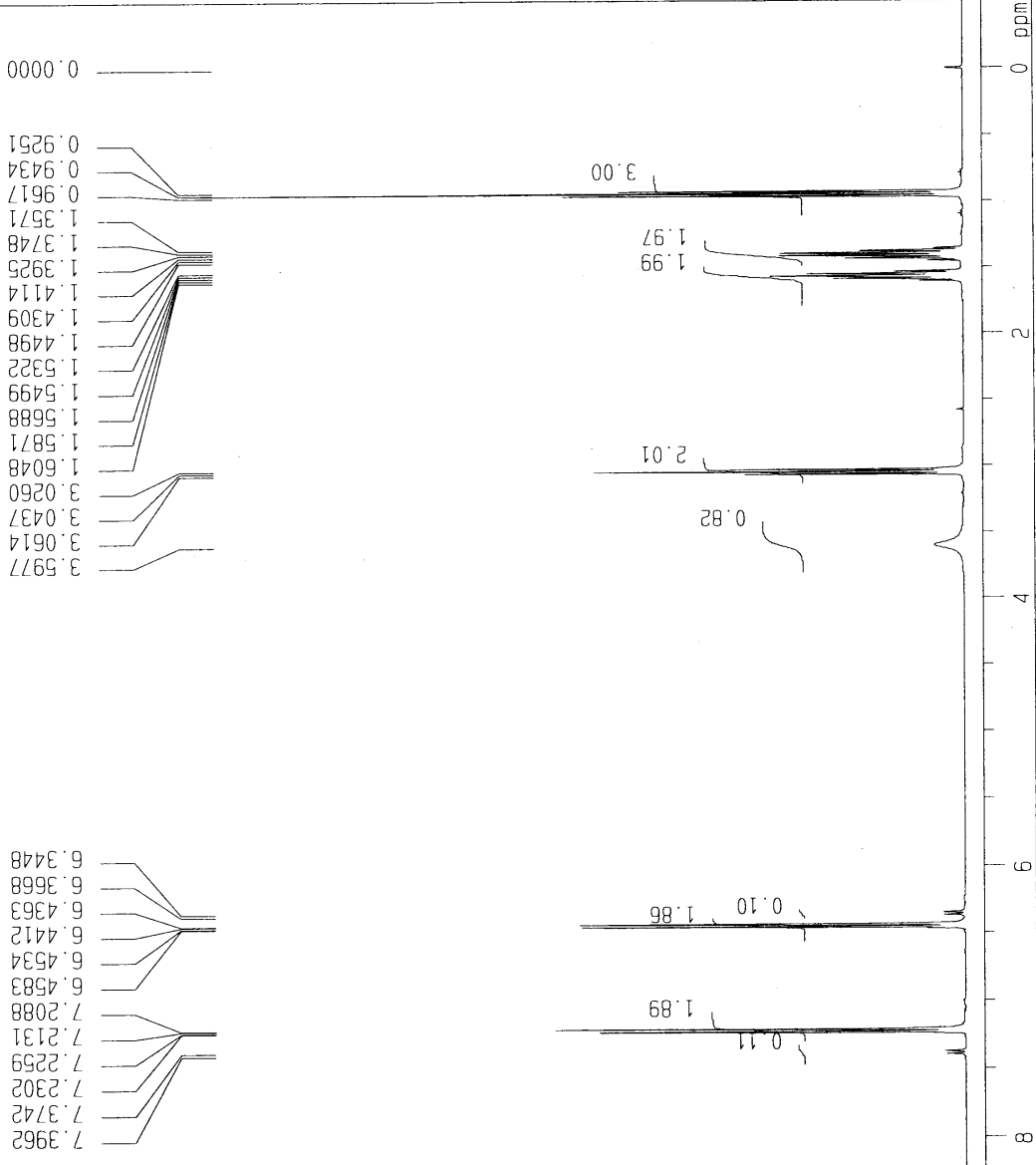
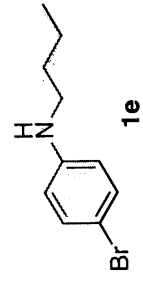
ko01135a''

Date : Tue Aug 5 15: 34: 39 2003

FileName : ko01135a''.nmdata
Comment : ko01135a''
SliceHistory :
EXMODE : non

POINT 32768 points
SAMPO 32768 points
FREQU 7993.6 Hz
FILTR 4000 Hz
DELAY 50.0 usec
DEADT 72.1 usec
INTVL 125.1 usec
TIMES 32 times
DUMMY 0 times
PD 2.9007 sec
ACQTM 4099.2769 msec
PREDL 10.00000 msec
INWIT 0.5000 msec
RESOL 0.24 Hz
PWI 5.75 usec
1H
OBNUC
OBFRQ 399.65 MHz
OBSET 134300.00 Hz
RGAIN 13

SCANS 32 times
SLVNT CDCL3
SPINNING 11 Hz
TEMP 22.5 C



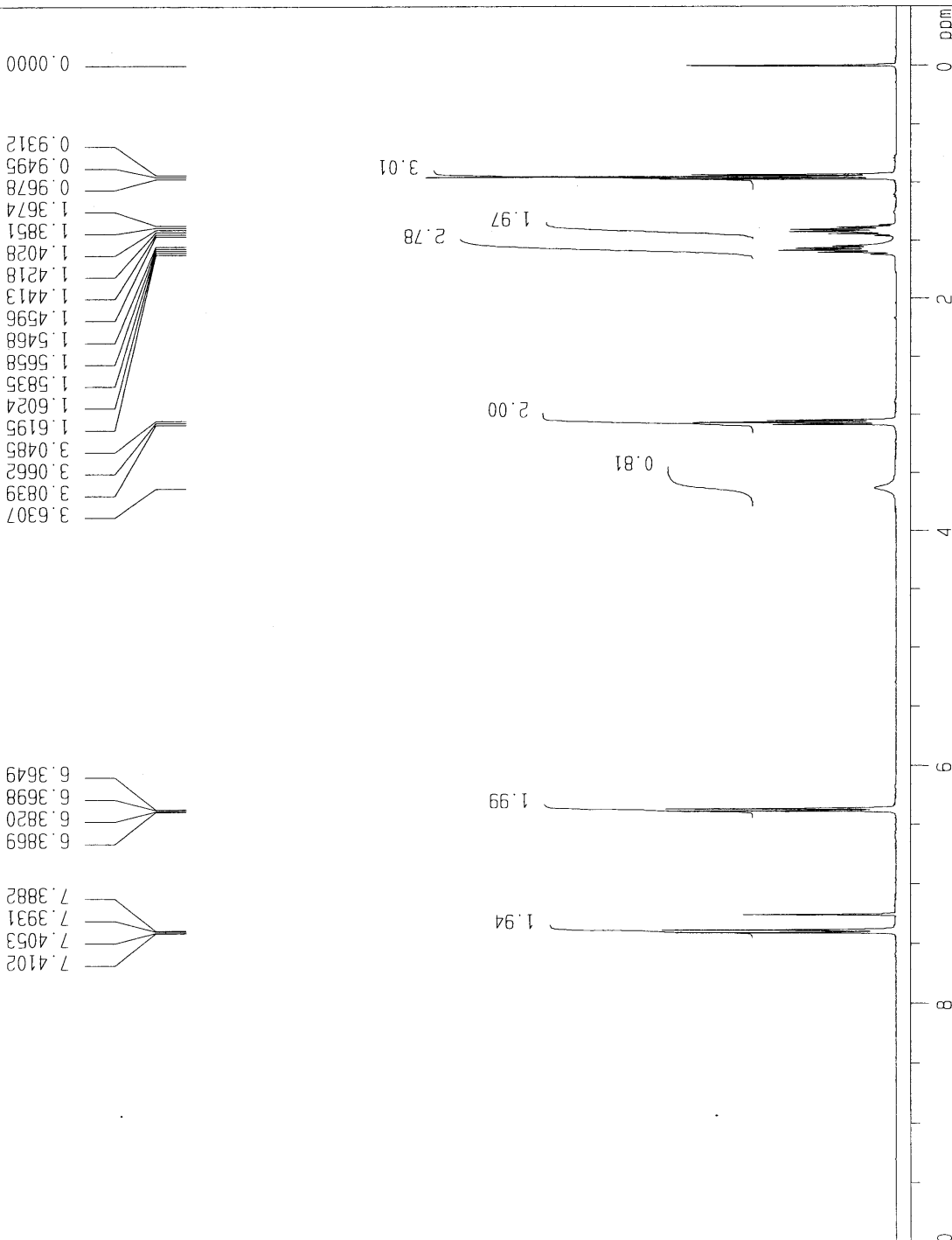
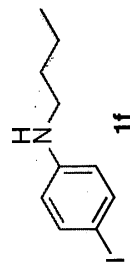
k002087a'

Date : Fri Sep 12 07:58:13 2003

FileName : k002087a'.nmdata
Comment : k002087a'
SliceHistory :
EXMODE : non

POINT 32768 points
SAMP0 32768 points
FREQU 7993.6 Hz
FILTR 4000 Hz
DELAY 50.0 usec
DEADT 72.1 usec
INTVL 125.1 usec
TIMES 64 times
DUMMY 0 times
PD 2.9007 sec
ACQTM 4099.2769 msec
PREDL 10.00000 msec
INIWT 0.5000 msec
RESOL 0.24 Hz
PWI 5.75 usec
OBNUC 1H
OBFRG 399.65 MHz
OBSET 134300.00 Hz
RGAIN 22

SCANS 64 times
SLVNT CDCL3
SPINNING 14 Hz
TEMP 25.3 C

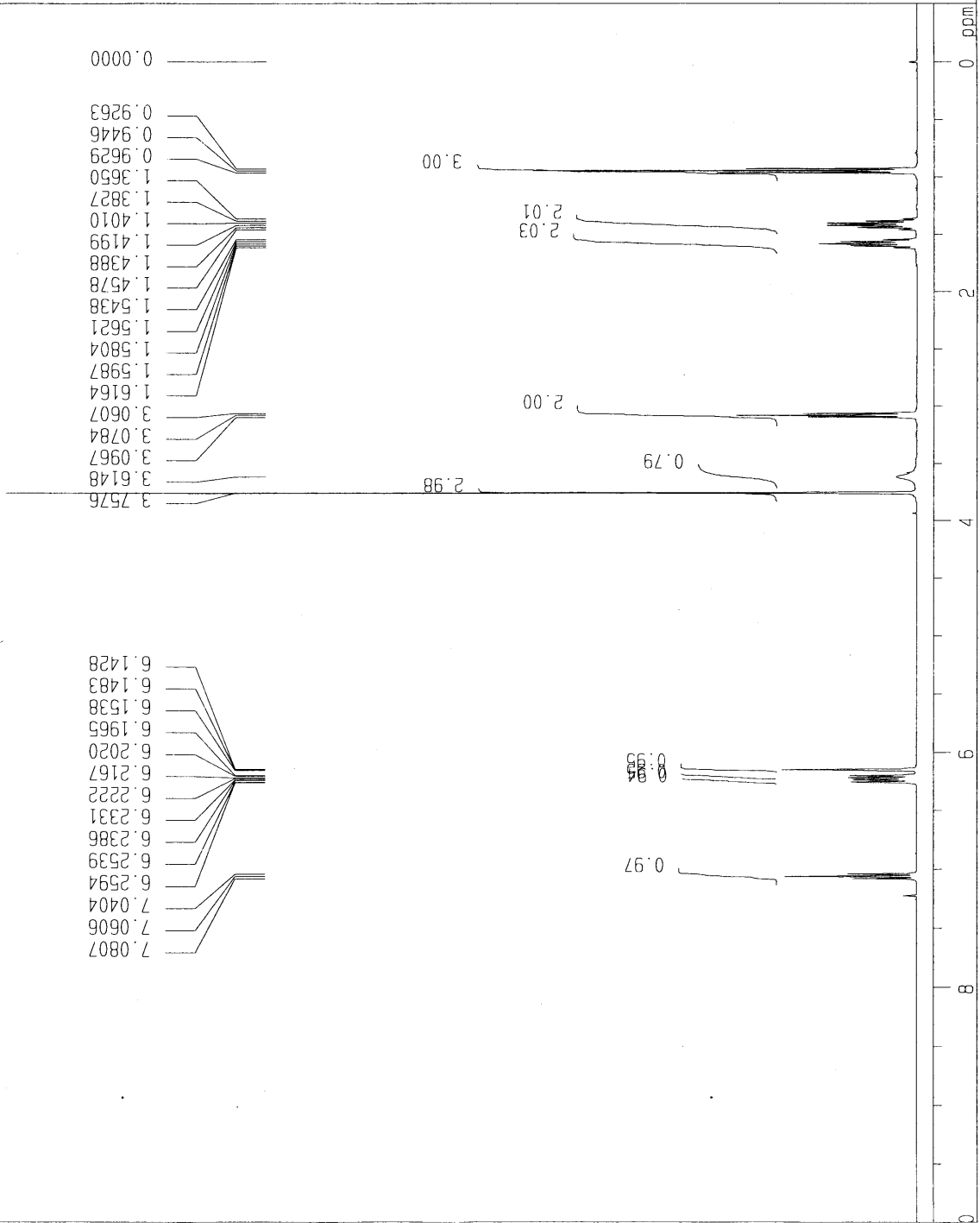
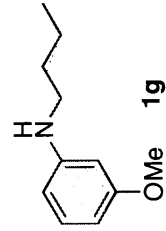


k002018a

Date : Sat Aug 2 07:41:21 2003

FileName : k002018a.nmdata
Comment : k002018a
SliceHistory :
EXMODE : non

POINT : 32768 points
SAMP0 : 32768 points
FREQ0 : 7993.6 Hz
FILTR : 4000 Hz
DELAY : 50.0 usec
DEADT : 72.1 usec
INTVL : 125.1 usec
TIMES : 64 times
DUMMY : 0 times
PD : 2.9007 sec
ACQTM : 4099.2769 msec
PREDL : 10.0000 msec
INIWT : 0.5000 msec
RESOL : 0.24 Hz
PW1 : 5.75 usec
OBNUC : 1H
OBFRQ : 399.65 MHz
OBSET : 134300.00 Hz
RGAIN : 13
SCANS : 64 times
SLVNT : COCL3
SPINNING : 14 Hz
TEMP : 20.4 C



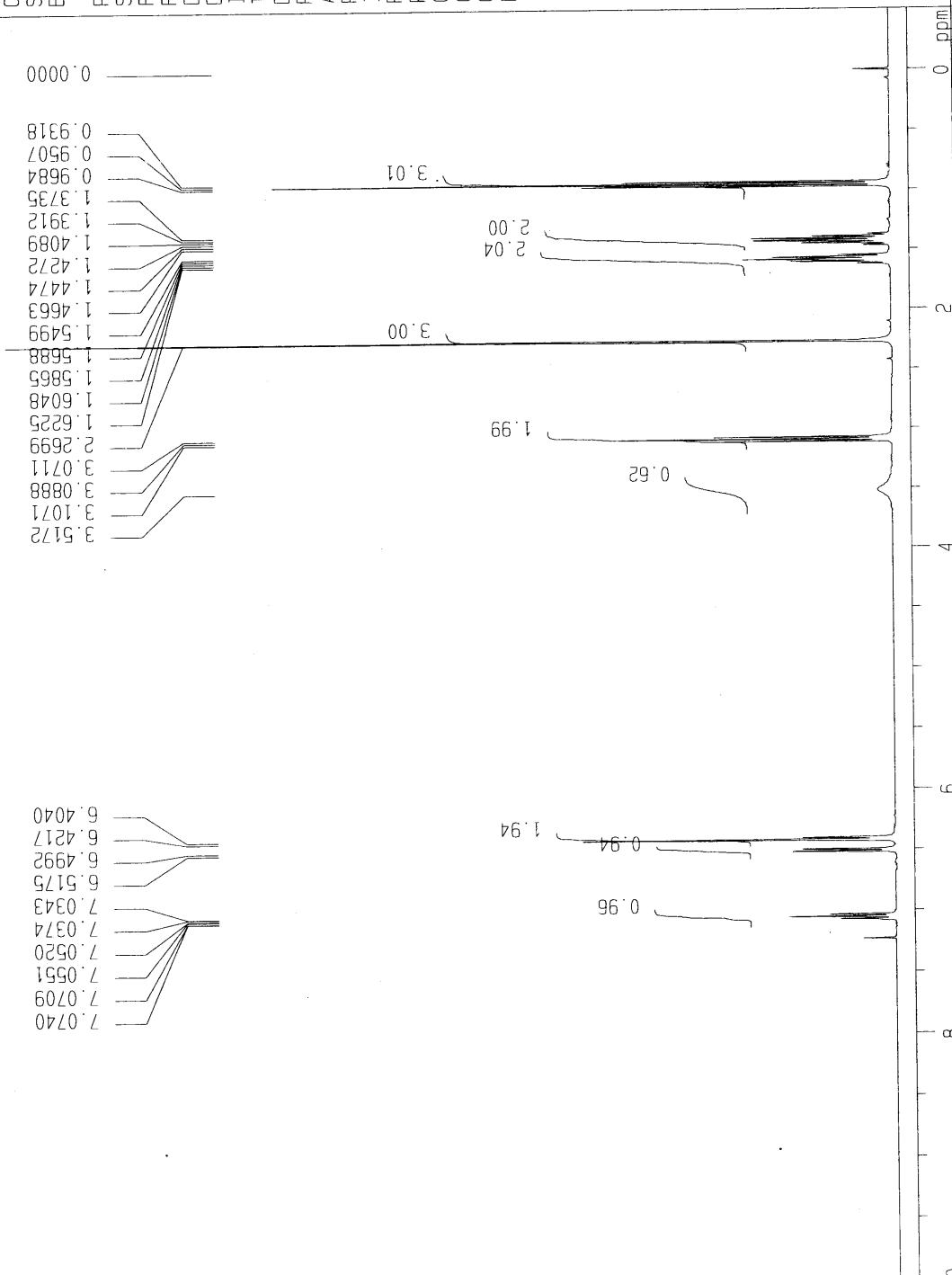
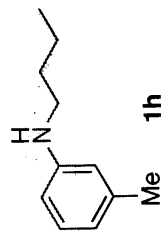
kc02017a

Date : Sat Aug 2 05:43:38 2003

FileName : kc02017a.nmdata
Comment : kc02017a
SliceHistory : non
EXMODE :

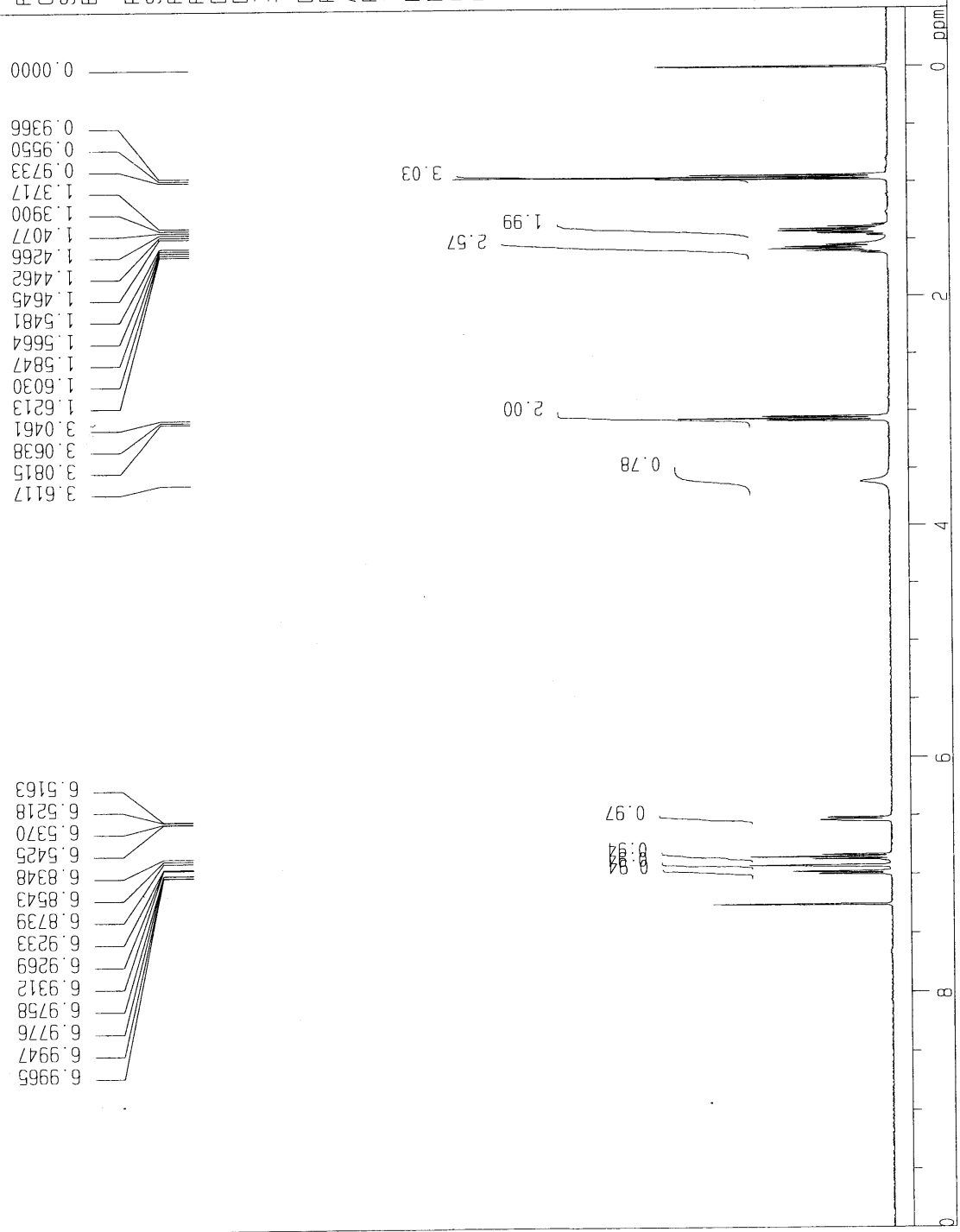
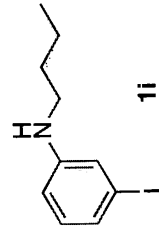
POINT 32768 points
SAMP0 32768 points
FREQU 7993.6 Hz
FILTR 4000 Hz
DELAY 50.0 usec
DEADT 72.1 usec
INTVL 125.1 usec
TIMES 64 times
DUMMY 0 times
PD 2.9007 sec
ACQTM 4099.2769 msec
PREDL 10.00000 msec
INIWT 0.5000 msec
RESOL 0.24 Hz
PW1 5.75 usec
OBNUC 1H
OBFRQ 399.65 MHz
OBSET 134300.00 Hz
RGAIN 15

SCANS 64 times
SLVNT CDCL3
SPINNING 7 Hz
TEMP 20.6 C



k002085a'

Date : Wed Sep 10 12:51:46 2003
FileName : k002085a', nmdata
Comment : k002085a
SliceHistory : non
EXMODE : non
POINT : 32768 points
SAMPO : 32768 points
FREQU : 7993.6 Hz
FILTR : 4000 Hz
DELAY : 50.0 usec
DEADT : 72.1 usec
INTVL : 125.1 usec
TIMES : 32 times
DUMMY : 0 times
PD : 2.9007 sec
ACQTM : 4099.2769 msec
PREDL : 10.0000 msec
INIWT : 0.5000 msec
RESOL : 0.24 Hz
PW1 : 5.75 usec
1H : 399.65 MHz
134300.00 Hz
OBSET : 22
SCANS : 32 times
SLVNT : CDCL3
SPINNING : 13 Hz
TEMP : 24.6 C

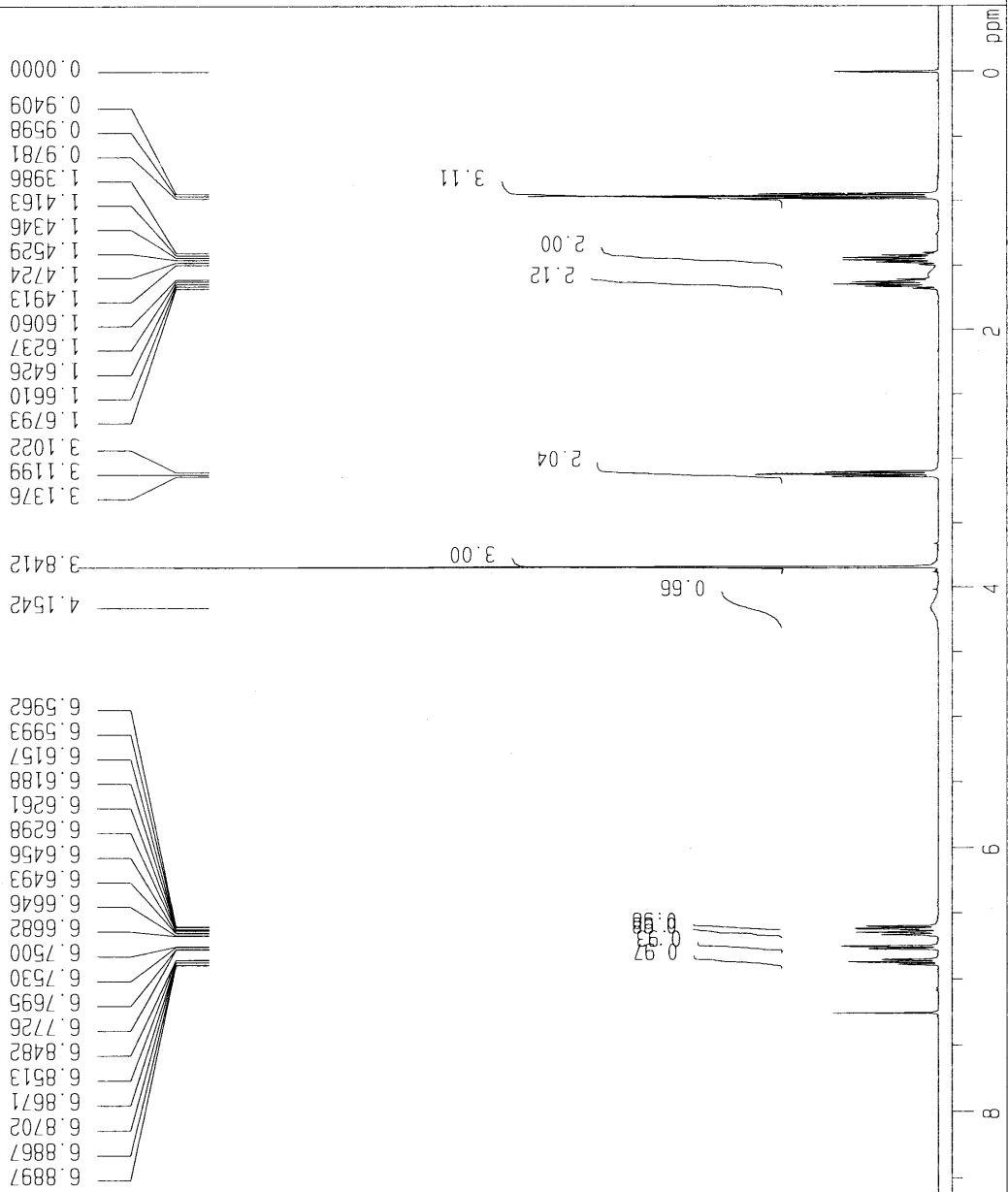
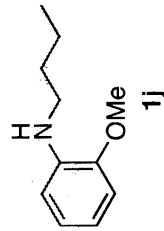


ko01178a'

Date : Sat Aug 2 19: 34: 52 2003

FileName : ko01178a'.nmdata
Comment : ko01178a'
SliceHistory :
EXMODE : non

POINT 32768 points
SAMP0 32768 points
FREQU 7993.6 Hz
FILTR 4000 Hz
DELAY 50.0 usec
DEADT 72.1 usec
INVL 125.1 usec
TIMES 64 times
DUMMY 0 times
PD 2.9007 sec
ACQTM 4099.2769 msec
PREDL 10.00000 msec
INIWT 0.5000 msec
RESOL 0.24 Hz
PW1 5.75 usec
1H
OBNUC 399.65 MHz
OBFRQ 134300.00 Hz
OBSET 19
RGAIN 64 times
SCANS 64 times
SLVNT CDCL3
SPINNING 12 Hz
TEMP 20.7 C



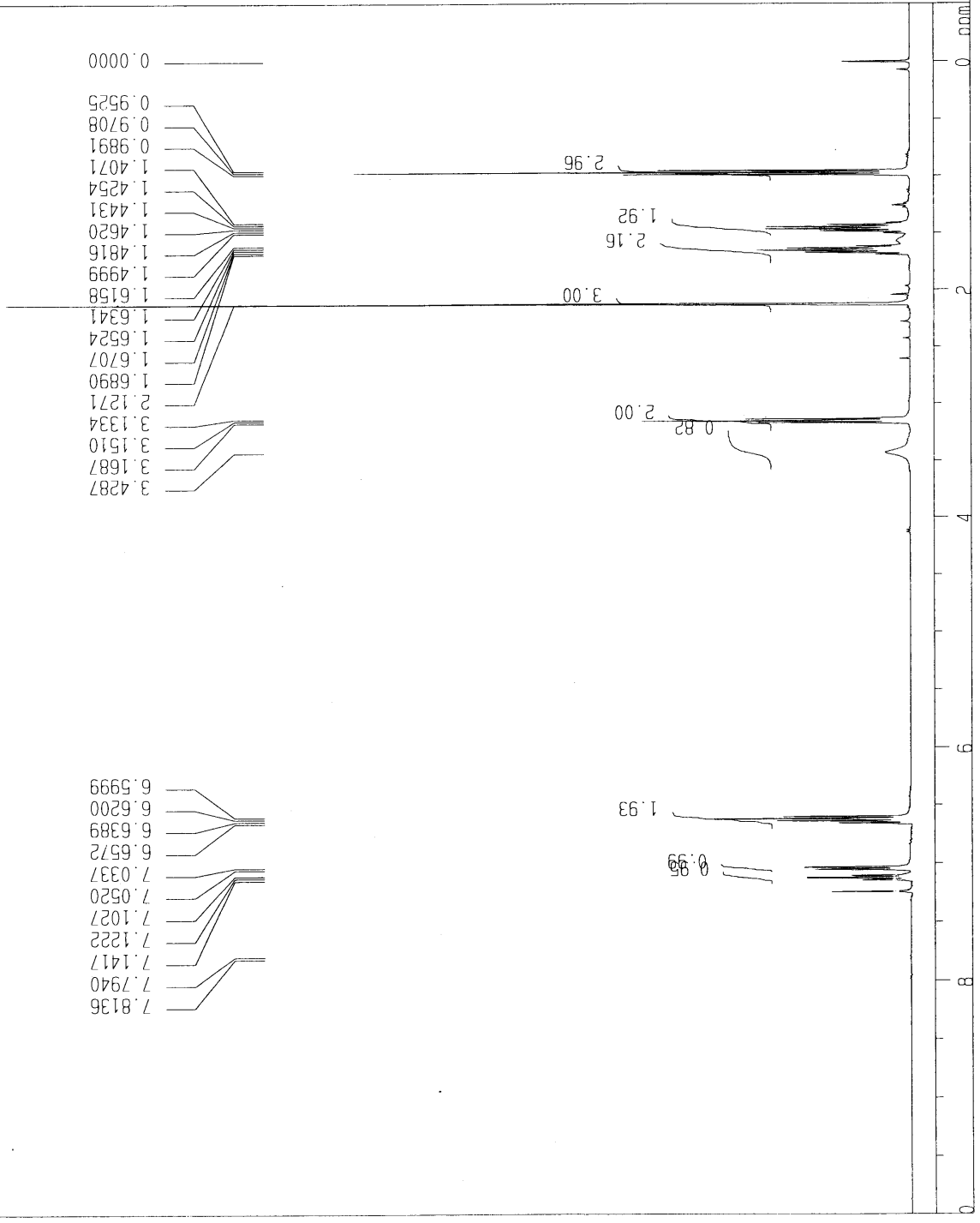
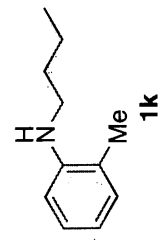
k002105a'

Date : Fri Sep 25 01:42:42 2003

FileName : k002105a'.nmdata
Comment : k002105a'
SliceHistory : non
EXMODE :

POINT 32768 points
SAMP0 32768 points
FREQU 7993.6 Hz
FILTR 4000 Hz
DELAY 50.0 usec
DEADT 72.1 usec
INTVL 125.1 usec
TIMES 64 times
DUMMY 0 times
PD 2.9007 sec
ACQTM 4099.2769 msec
PREDL 10.00000 msec
IN1WT 0.5000 msec
RESOL 0.24 Hz
PW1 5.75 usec
OBNUC 1H
OBFRQ 399.65 MHz
OBSET 134300.00 Hz
RGAIN 17

SCANS 64 times
SLVNT CDCL3
SPINNING 11 Hz
TEMP 20.6 C



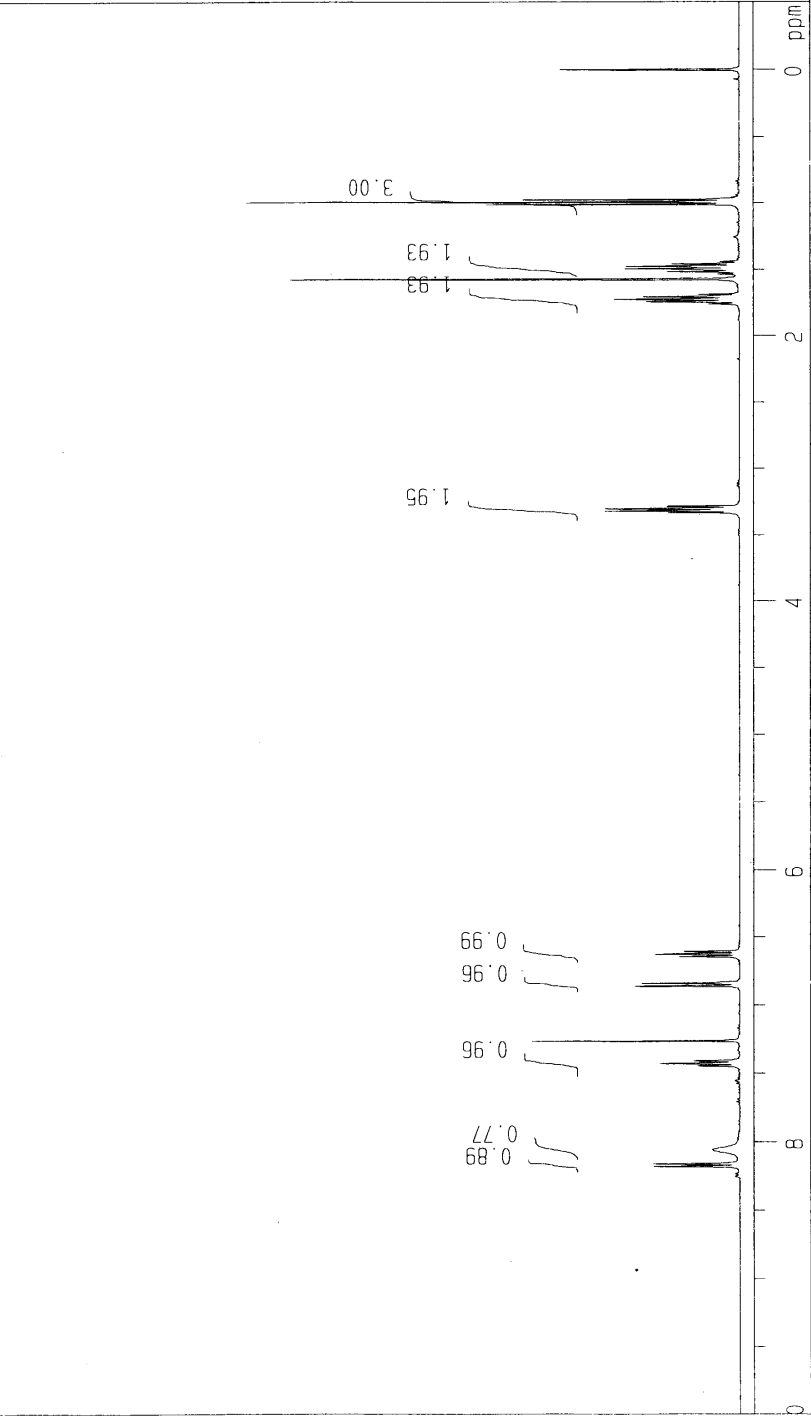
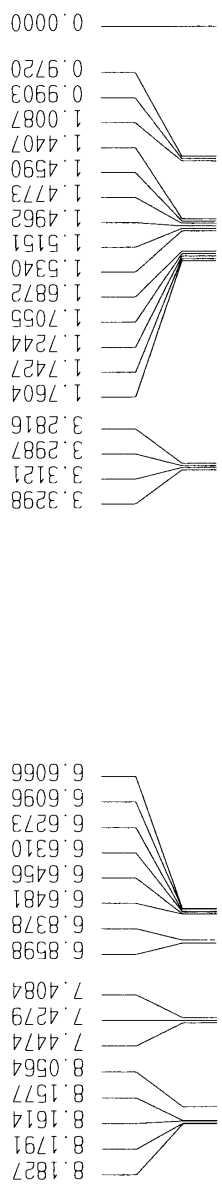
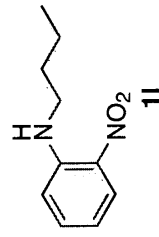
kc01179a'

Date : Sat Aug 2 20:14:01 2003

FileName : kc01179a'.nmdata
 Comment : kc01179a'
 SliceHistory : non
 EXMODE :

POINT 32768 points
 SAMPO 32768 points
 FREQU 7993.6 Hz
 FILTR 4000 Hz
 DELAY 50.0 usec
 DEADT 72.1 usec
 INI VL 125.1 usec
 TIMES 64 times
 DUMMY 0 times
 PD 2.9007 sec
 ACQTM 4099.2769 msec
 PREDL 10.00000 msec
 INI WT 0.5000 msec
 RESOL 0.24 Hz
 PW1 5.75 usec
 OBNUC 1H
 OBFREQ 399.65 MHz
 OBTSET 134300.00 Hz
 RGAIN 21
 SCANS 64 times

SOLVENT : CDCL3
 SPINNING : 18 Hz
 TEMP : 21.3 C



ko02106a'

Date : Fri Sep 26 05:22:23 2003

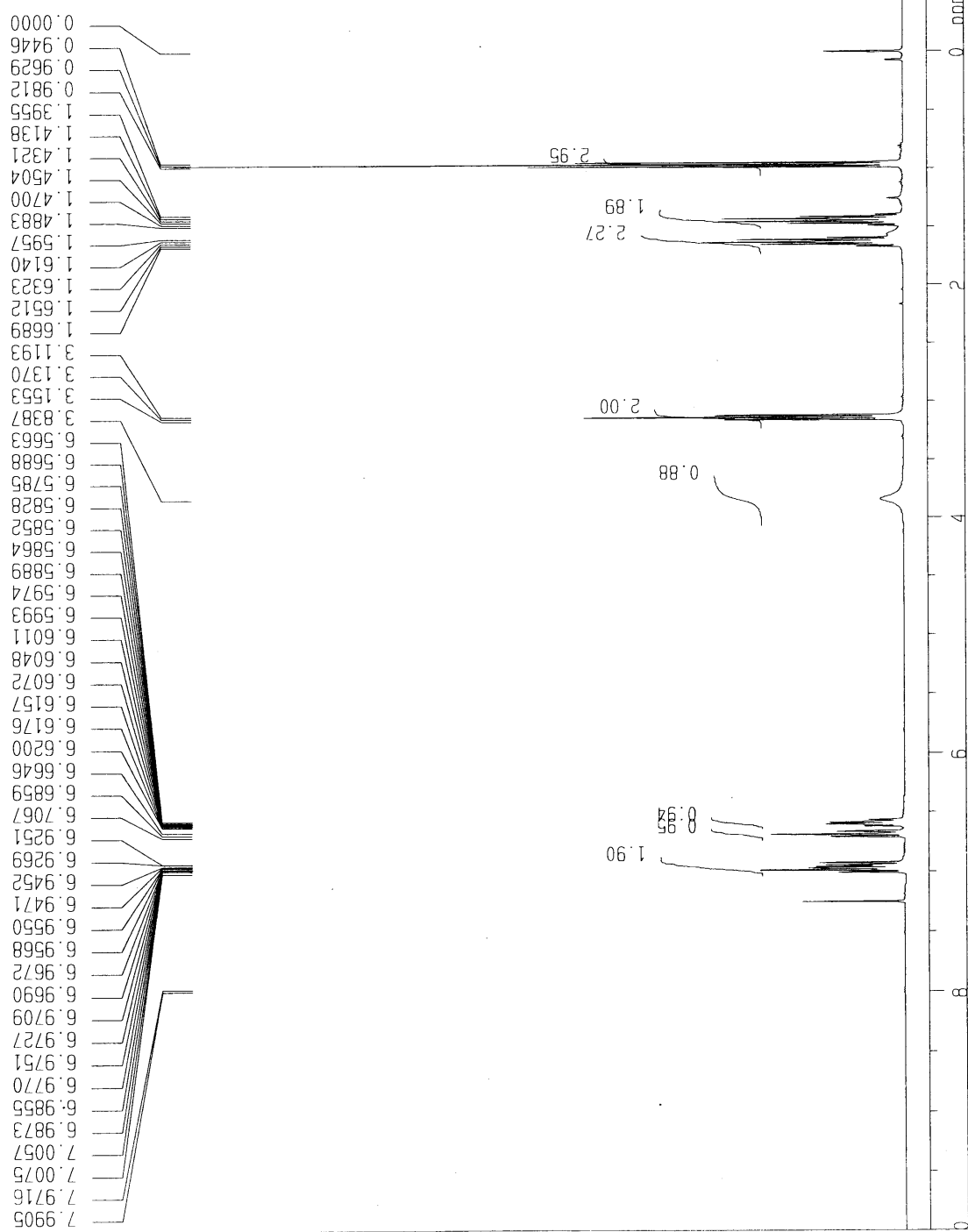
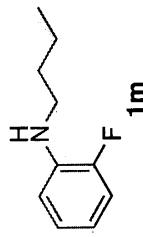
FileName : ko02106a'.nmdata
Comment : ko02106a'
SiichHistory : non
EXMODE :

POINT 32768 points
SAMPO 32768 points
FREGU 7993.6 Hz
FILTR 4000 Hz
DELAY 50.0 usec
DEADT 72.1 usec
INTVL 125.1 usec
TIMES 64 times
DUMMY 0 times

PD 2.9007 sec
ACGTM 4099.2769 msec
PREDL 10.00000 msec
INIWT 0.5000 msec
RESOL 0.24 Hz
PWI 5.75 usec

OBNUC 1H
OBFRQ 399.65 MHz
OBSET 134300.00 Hz
RGAIN 19

SCANS 64 times
SLVNT CDCL3
SPINNING 13 Hz
TEMP 20.9 C



ko02072a'

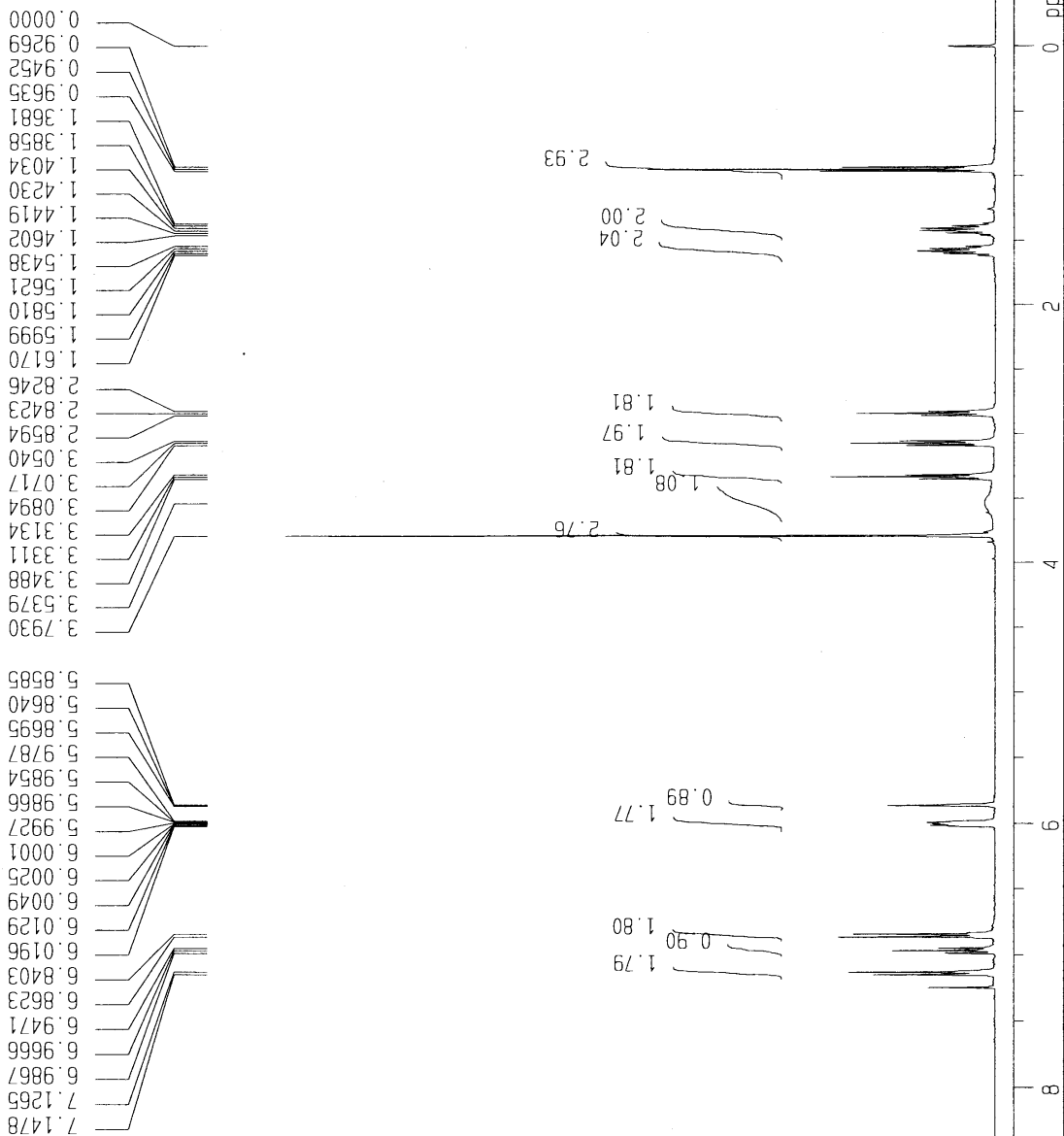
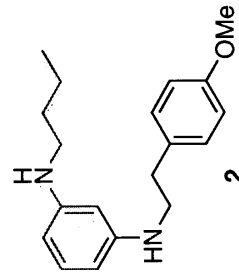
Date : Thu Sep 4 15:46:03 2003

FileName : ko02072a'.nmdata
Comment : ko02072a'

SliceHistory :
EXMODE : non

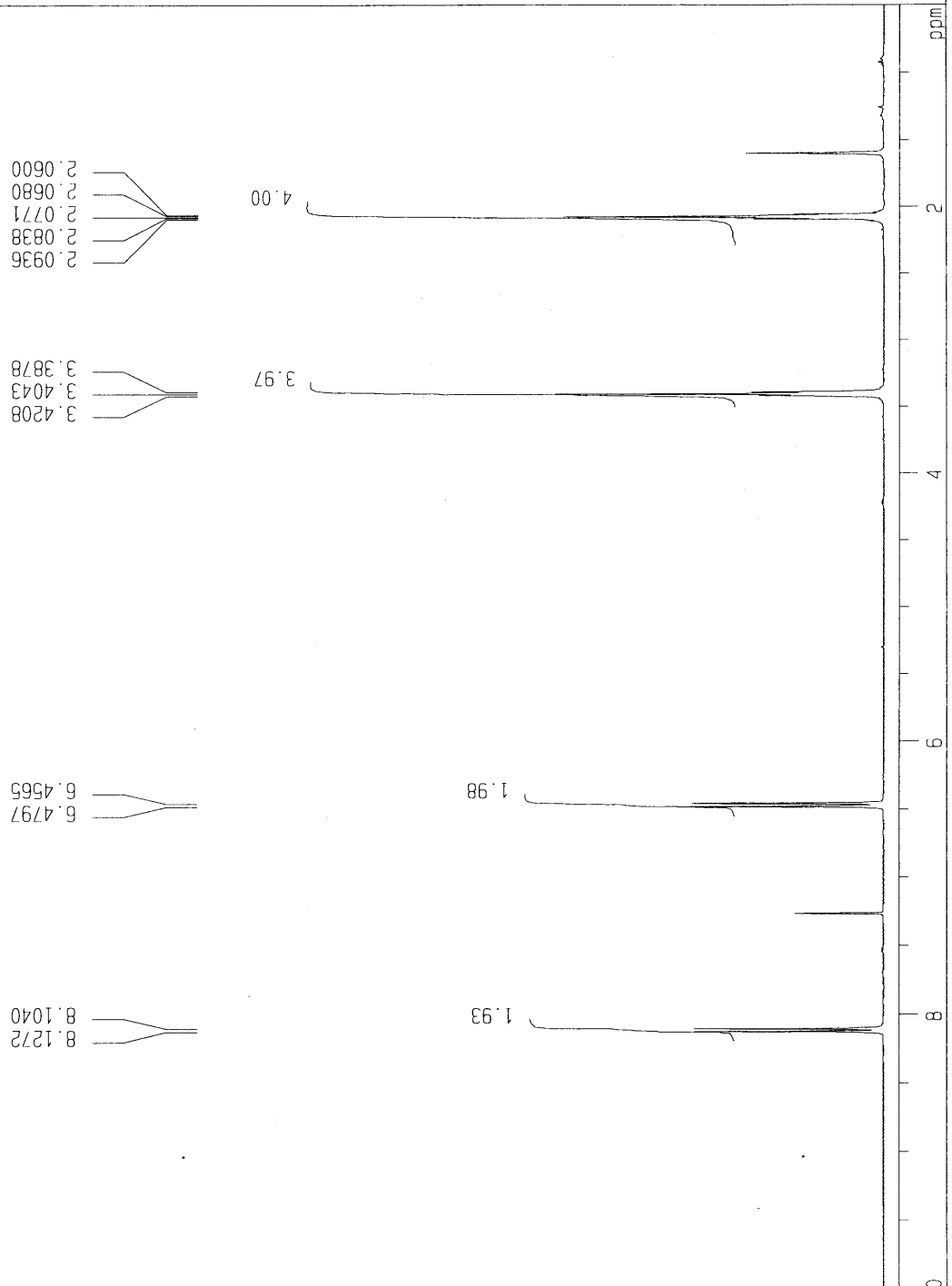
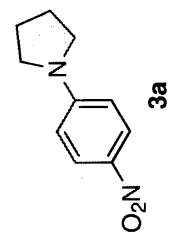
POINT : 32768 points
SAMPO : 32768 points
FREQU : 7993.6 Hz
FILT : 4000 Hz
DELAY : 50.0 usec
DEADT : 72.1 usec
INIVL : 125.1 usec
TIMES : 32 times
DUMMY : 0 times
PD : 2.9007 sec
ACGTM : 4099.2769 msec
PREDL : 10.00000 msec
INIWT : 0.5000 msec
RESOL : 0.24 Hz
PW1 : 5.75 usec
OBNUC : ¹H
OBFRQ : 399.65 MHz
OBSET : 134300.00 Hz
RGAIN : 18

SCANS : 32 times
SLVNT : CDCL3
SPINNING : 11 Hz
TEMP : 24.3 C



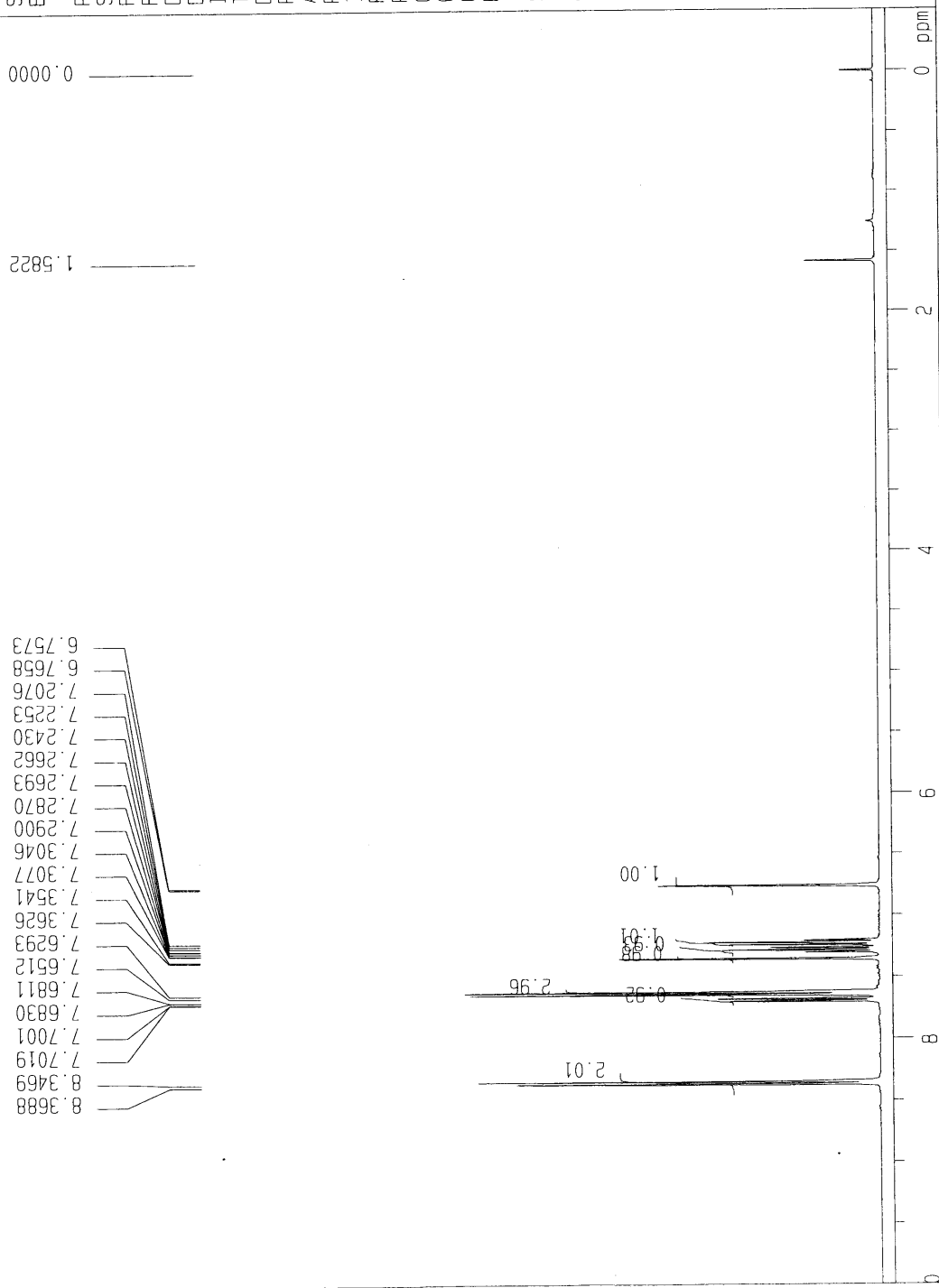
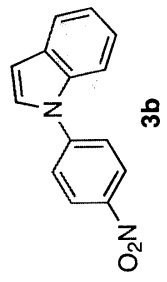
ko01137a''

Date : Tue Aug 5 14:44:14 2003
FileName : ko01137a''.nmdata
Comment : ko01137a''
SliceHistory :
EXMODE : non
POINT : 32768 points
SAMPO : 32768 points
FREQU : 7993.6 Hz
FILTR : 4000 Hz
DELAY : 50.0 usec
DEADT : 72.1 usec
INTVL : 125.1 usec
TIMES : 32 times
DUMMY : 0 times
PD : 2.9007 sec
ACQTM : 4099.2769 msec
PREDL : 10.00000 msec
INIWT : 0.5000 msec
RESOL : 0.24 Hz
PW1 : 5.75 usec
OBNUC : ¹H
OBFRQ : 399.65 MHz
OBSET : 134300.00 Hz
RGAIN : 20
SCANS : 32 times
SLVNT : CDCL3
SPINNING : 12 Hz
TEMP : 22.2 C



k001136a''

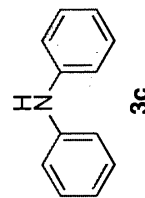
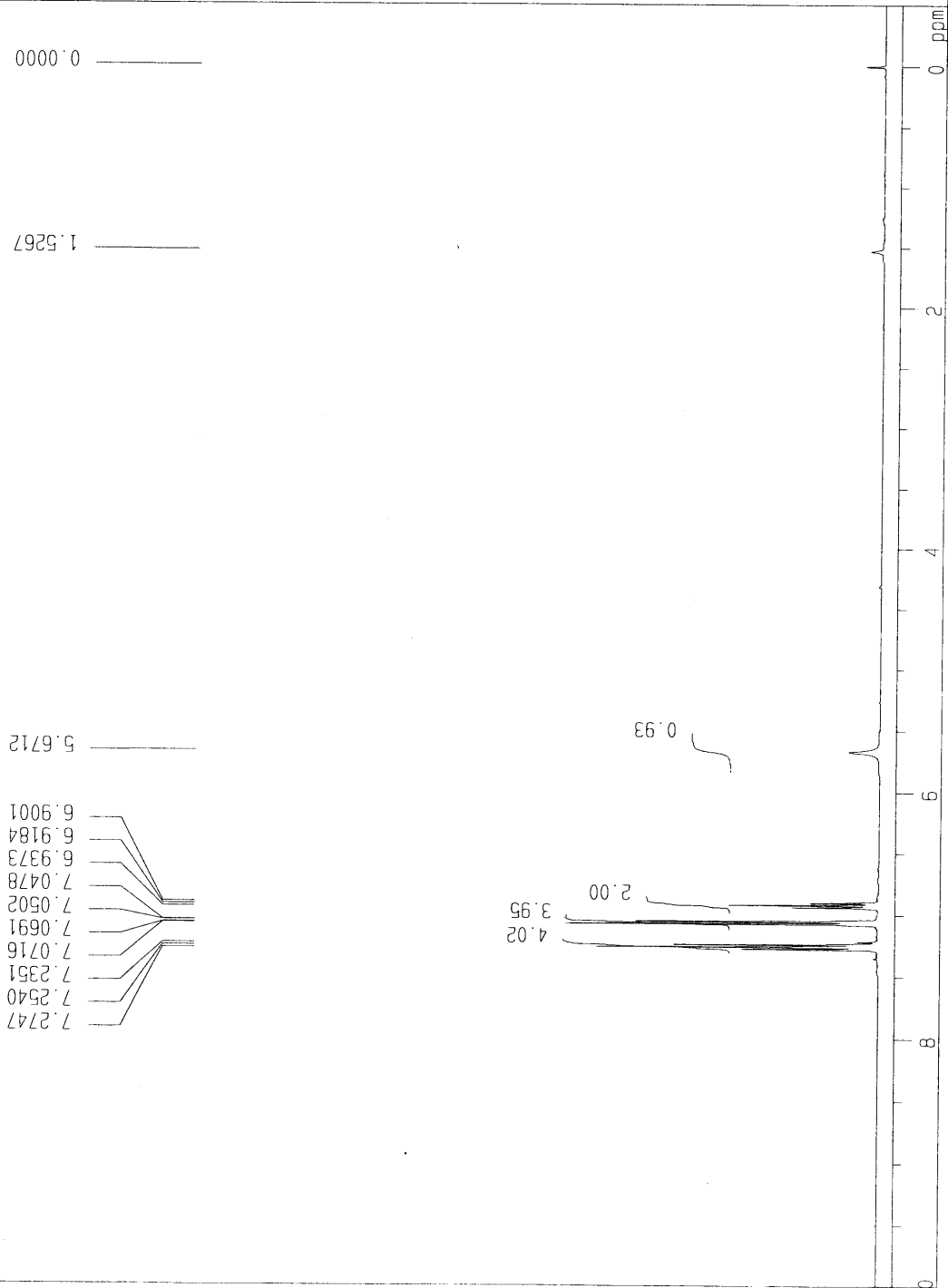
Date : Sun Aug 3 11:18:58 2003
FileName : k001136a''.nmData
Comment : k001136a''
SliceHistory : non
EXMODE : non
POINT 32768 points
SAMPO 32768 points
FREQU 7993.6 Hz
FILTR 4000 Hz
DELAY 50.0 usec
DEADT 72.1 usec
INTVL 125.1 usec
TIMES 64 times
DUMMY 0 times
PD 2.9007 sec
ACQTM 4099.2769 msec
PREDL 10.00000 msec
INIWT 0.5000 msec
RESOL 0.24 Hz
PWI 5.75 usec
OBNUC 1H
OBFRQ 399.65 MHz
OBSET 134300.00 Hz
RGAIN 15
SCANS 64 times
SLVNT CDCL3
SPINNING 14 Hz
TEMP 20.5 C



ko01088a'

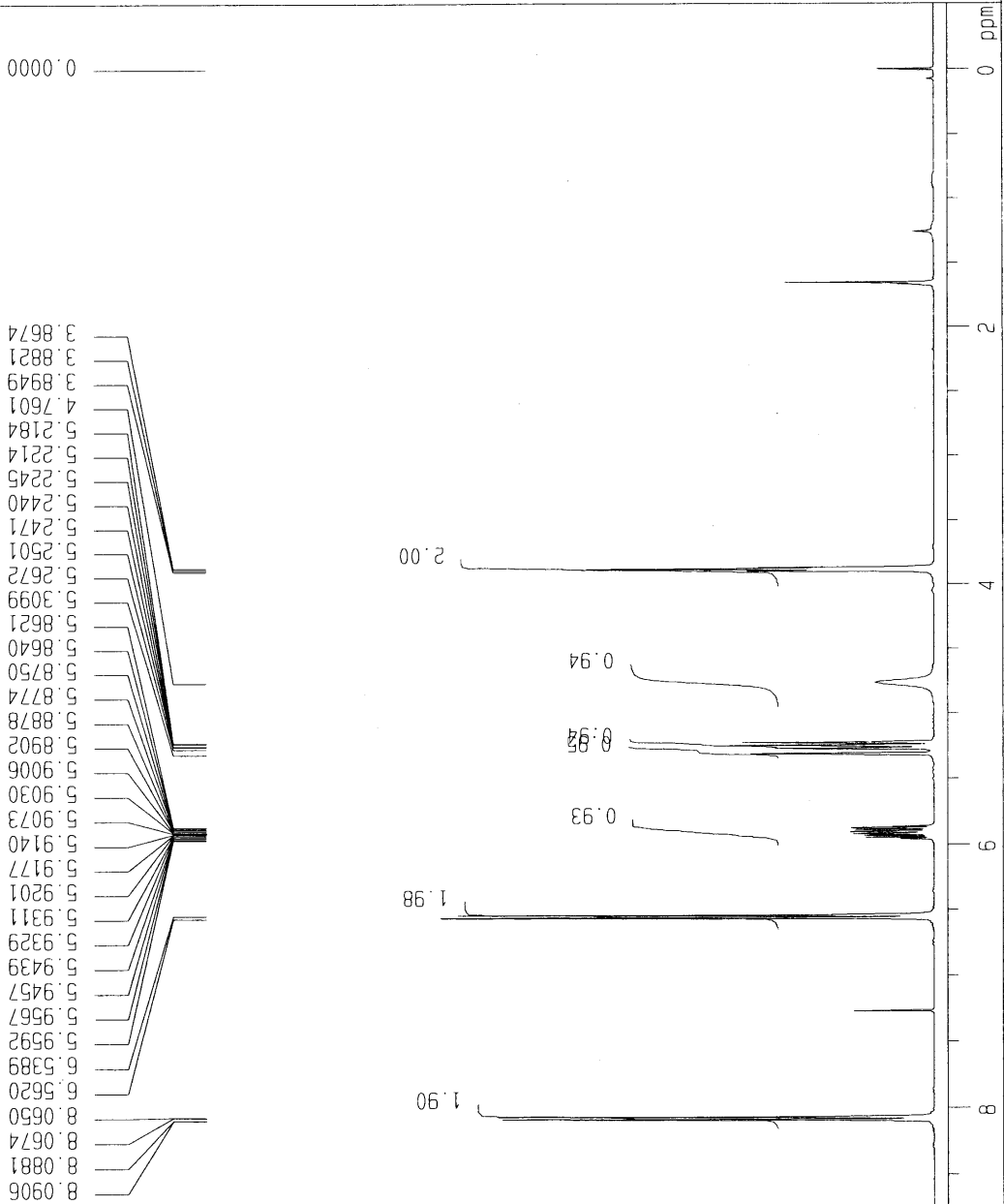
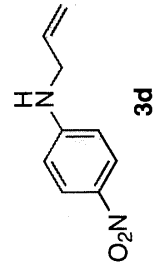
Date : Fri Aug 1 15:58:34 2003

FileName : ko01088a'.nmdata
Comment : ko01088a'
SliceHistory : non
EXMODE : non
POINT : 32768 points
SAMPO : 32768 points
FREQU : 7993.6 Hz
FILTR : 4000 Hz
DELAY : 50.0 usec
DEADT : 72.1 usec
INTVL : 125.1 usec
TIMES : 64 times
DUMMY : 0 times
PD : 2.9007 sec
ACQTM : 4099.2769 msec
PREDL : 10.00000 msec
INIWT : 0.5000 msec
RESOL : 0.24 Hz
PW1 : 5.75 usec
OBNUC : ¹H
OBFRQ : 399.65 MHz
OBSET : 134300.00 Hz
RGAIN : 15
SCANS : 64 times
SLVNT : CDCL3
SPINNING : 14 Hz
TEMP : 20.8 C



ko01139a''

Date : Mon Aug 4 00:55:32 2003
FileName : ko01139a''.nmdata
Comment : ko01139a''
SliceHistory : non
EXMODE :
POINT : 32768 points
SAMPO : 32768 points
FREQU : 7993.6 Hz
FILTR : 4000 Hz
DELAY : 50.0 usec
DEADT : 72.1 usec
INVL : 125.1 usec
TIMES : 64 times
DUMMY : 0 times
PD : 2.907 sec
ACQTM : 4099.2769 msec
PREDL : 10.00000 msec
INIWT : 0.5000 msec
RESOL : 0.24 Hz
PW1 : 5.75 usec
OBNUC : ¹H
OBFRQ : 399.65 MHz
OBSET : 134300.00 Hz
RGAIN : 17
SCANS : 64 times
SLVNT : CDCL3
SPINNING : 11 Hz
TEMP : 21.3 C



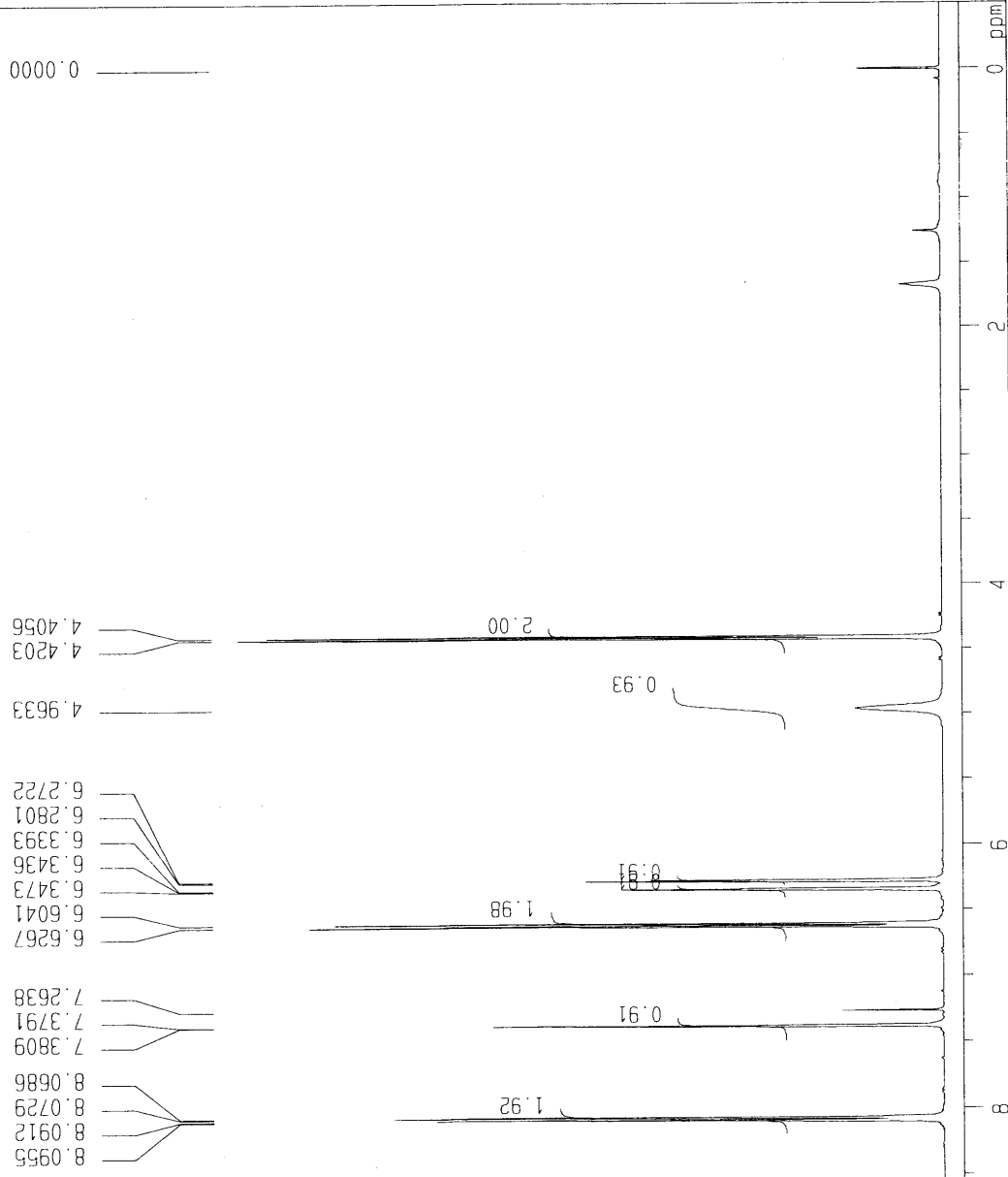
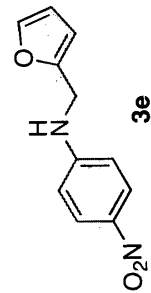
kc01140a'

Date : Sat Aug 2 03:44:19 2003

FileName : kc01140a'.nmdata
Comment : kc01140a'
SliceHistory : non
EXMODE :

POINT 32768 points
SAMPO 32768 points
FREQU 7993.6 Hz
FILTR 4000 Hz
DELAY 50.0 usec
DEADT 72.1 usec
INTVL 125.1 usec
TIMES 64 times
DUMMY 0 times
PD 2.9007 sec
ACQTM 4099.2769 msec
PREDL 10.00000 msec
IN1WT 0.5000 msec
RESOL 0.24 Hz
PW1 5.75 usec
OBNUC 1H
OBFRQ 399.65 MHz
OBSET 134300.00 Hz
RGAIN 16
SCANS 64 times

SOLVENT : CDCL3
SPINNING : 14 Hz
TEMP : 20.5 C



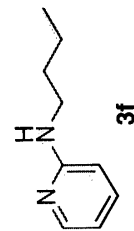
k001156a'

Date : Mon Aug 4 15:50:16 2003

File Name : k001156a'.nmdata
Comment : k001156a'
Slice History :
EXMODE : non

POINT 32768 points
SAMP0 32768 points
FREQU 7993.6 Hz
FILTR 4000 Hz
DELAY 50.0 usec
DEADT 72.1 usec
INTVL 125.1 usec
TIMES 32 times
DUMMY 0 times
PD 2.9007 sec
ACQTM 4099.2769 msec
PREDL 10.00000 msec
INIWT 0.5000 msec
RESOL 0.24 Hz
PW1 5.75 usec
OBNUC 1H
OBFRQ 399.65 MHz
OBSET 134300.00 Hz
RGAIN 16

SCANS 32 times
SLVNT CDCL3
SPINNING 12 Hz
TEMP 22.4 C



8.0729
8.0601
7.4261
7.4053
7.3846
6.5578
6.5425
6.5273
6.3753
6.3540
3.2682
3.2517
3.2346
3.2188
1.6408
1.6237
1.6048
1.5865
1.5676
1.4748
1.4565
1.4370
1.4187
1.4004
1.3827
0.9702
0.9519
0.9342
0.0000

3:00

1.98

1.98

2:00

0.92

0.98

0.96

0.95

0.95

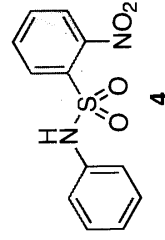
0 2 4 6 8 0 ppm

ko01186b

Date : Mon Jul 7 22:29:15 2003

FileName : ko01186b.nmdata
Comment : ko01186b
SliceHistory : non
EXMODE : non
POINT 32768 points
SAMPO 32768 points
FREQU 7993.6 Hz
FILTR 4000 Hz
DELAY 50.0 usec
DEADT 72.1 usec
INTVL 125.1 usec
TIMES 64 times
DUMMY 0 times
PD 2.9007 sec
ACQTM 4099.2769 msec
PREDL 10.00000 msec
INIWT 0.5000 msec
RESOL 0.24 Hz
PW1 5.75 usec
OBNUC 1H
OBFRQ 399.65 MHz
OBSET 134300.00 Hz
RGAIN 20

SCANS : 64 times
SLVNT : COCL3
SPINNING : 10 Hz
TEMP : 22.7 C



0.0000

1.5859

7.8648
7.8618
7.8447
7.8416
7.8325
7.8288
7.8123
7.8093
7.7086
7.7049
7.6897
7.6860
7.6708
7.6695
7.6659
7.5902
7.5872
7.5707
7.5676
7.5518
7.5487
7.2943
7.2894
7.2747
7.2619
7.2558
7.2064
7.2003
7.1851
7.1796
7.1619

3.18
2.13
1.04
1.00

0 ppm

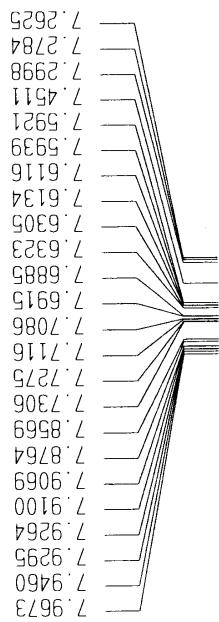
ko02039a''

Date : Tue Sep 9 11:36:45 2003

FileName : ko02039a''.mdata
Comment : ko02039a''
SliceHistory :
EXMODE : non

POINT : 32768 points
SAMPO : 32768 points
FREQ : 7993.6 Hz
FILTR : 4000 Hz
DELAY : 50.0 usec
DEADT : 72.1 usec
INTVL : 125.1 usec
TIMES : 32 times
DUMMY : 0 times
PO : 2.9007 sec
ACQTM : 4099.2769 msec
PREDL : 10.00000 msec
INIWT : 0.5000 msec
RESOL : 0.24 Hz
PW1 : 5.75 usec
1H :
OBNUC :
OBFRQ : 399.65 MHz
OBSET : 134300.00 Hz
RGAIN : 25
SCANS : 32 times
SLVNT : CDCL3
SPINNING : 12 Hz
TEMP : 22.9 C

0.0000



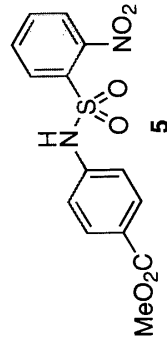
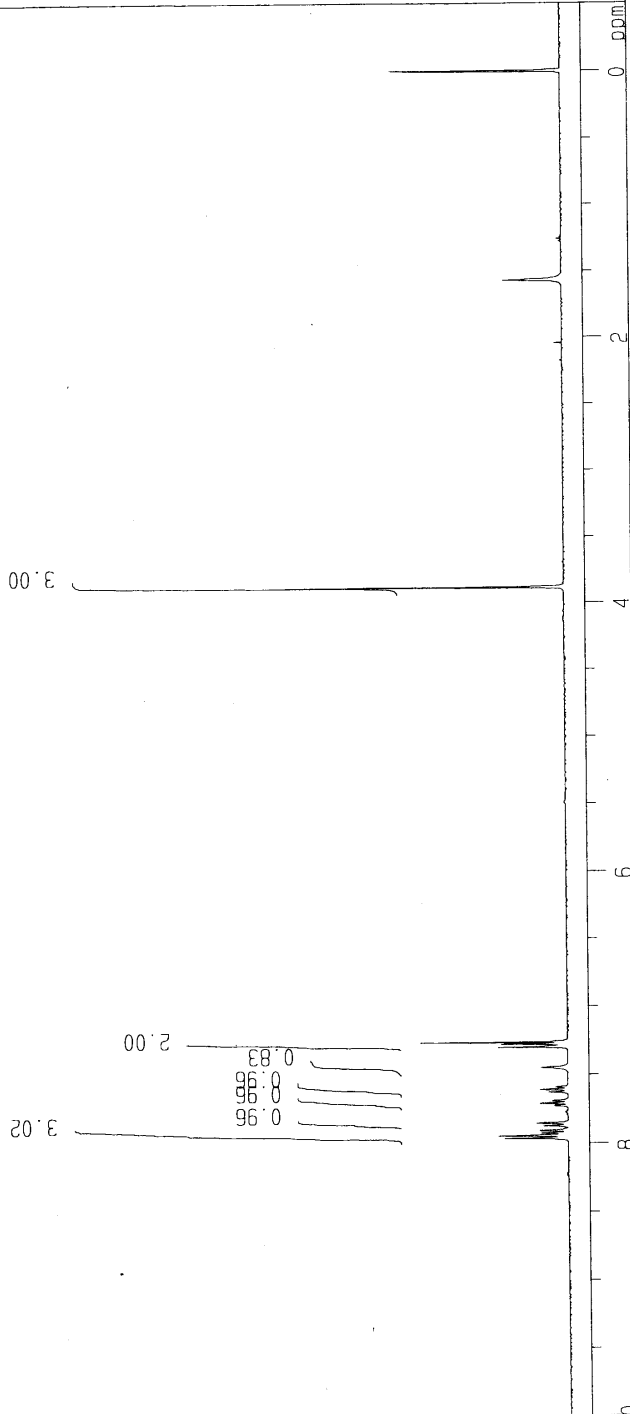
3.8833

3.00

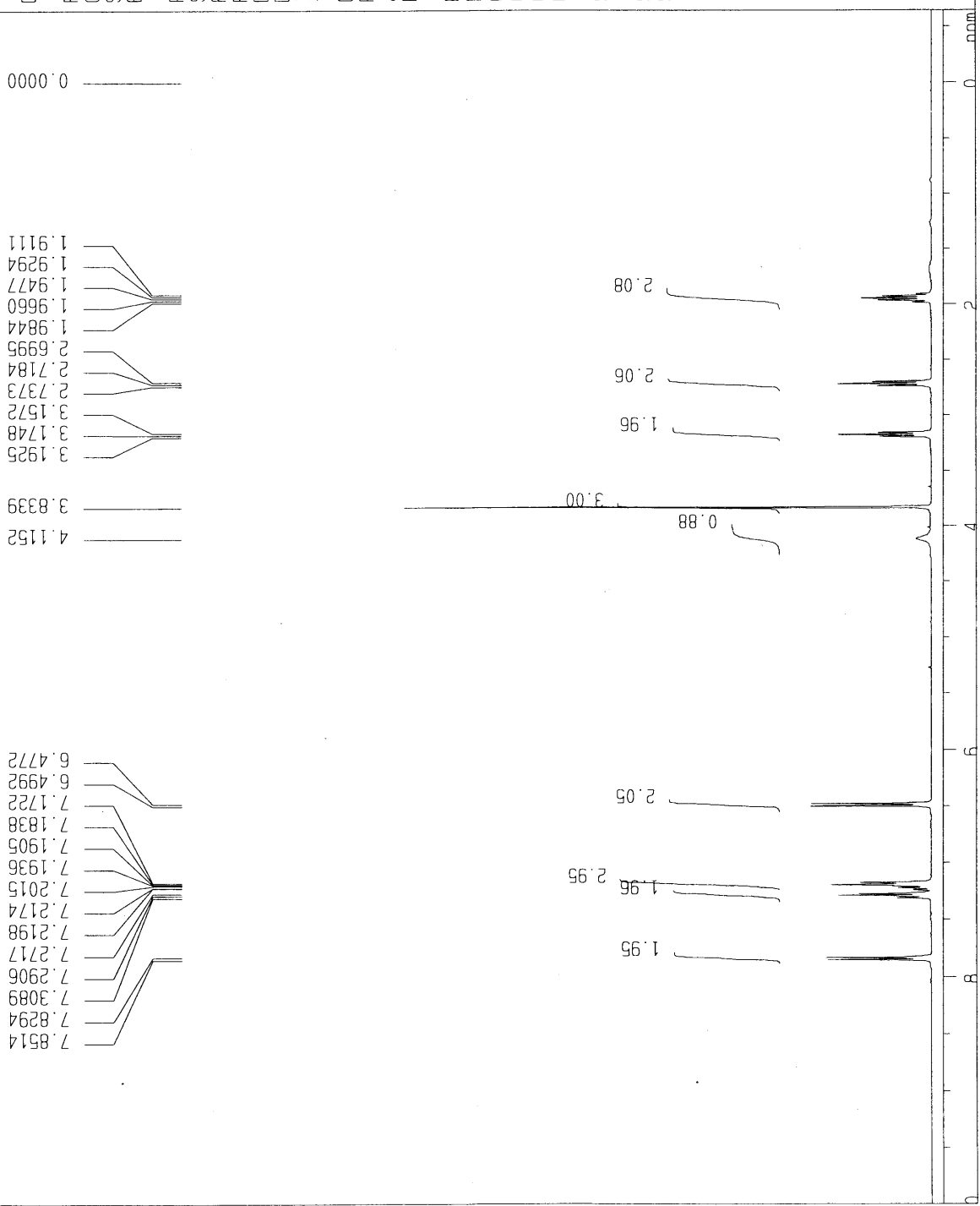
3.02

2.00

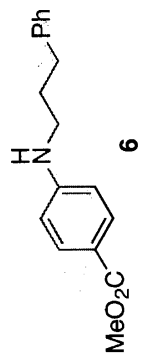
0.96
0.96
0.83



ko02052b'



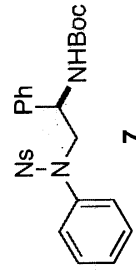
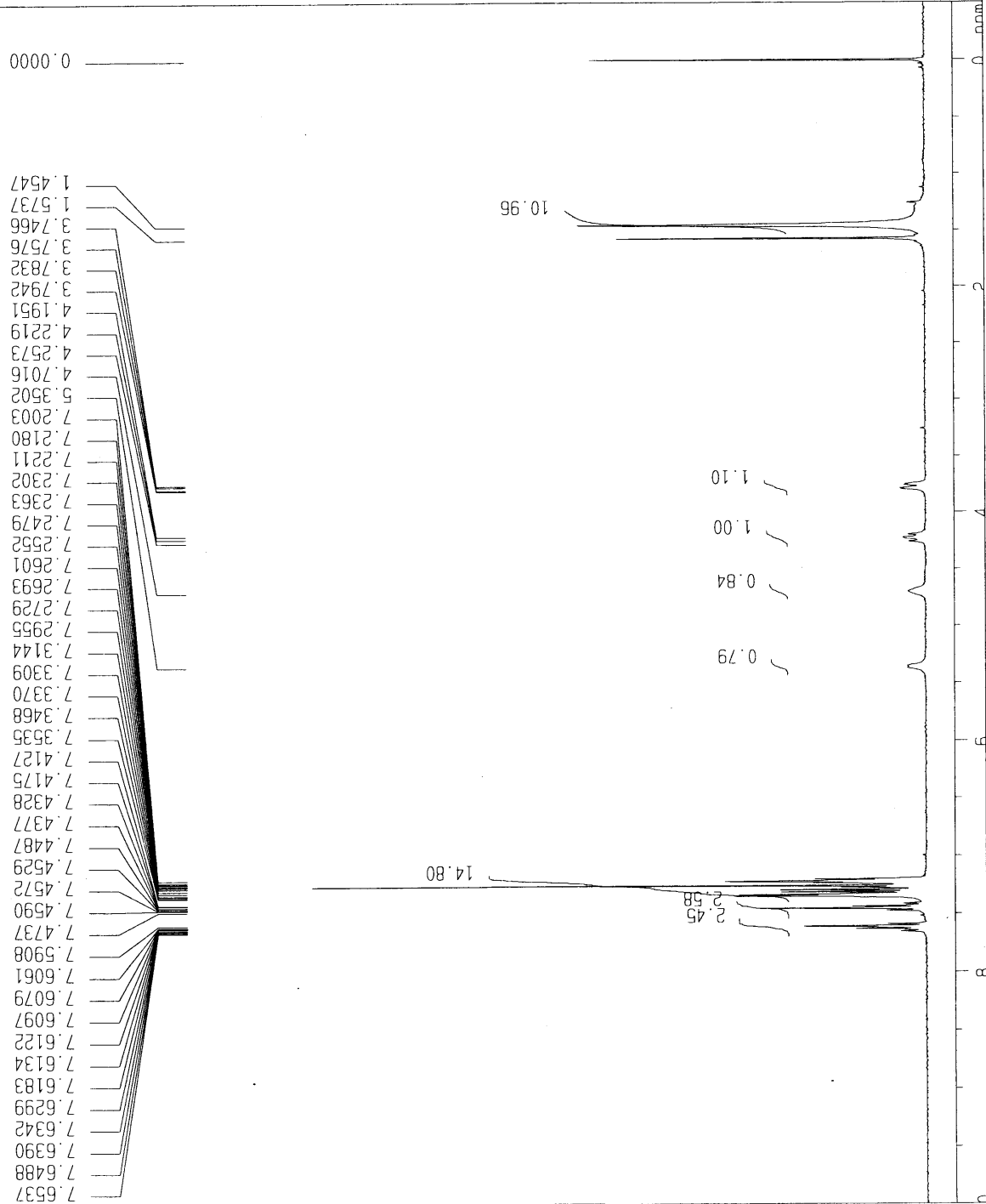
Date : Fri Aug 29 01:41:09 2003
FileName : ko02052b'.nmdata
Comment : ko02052b'
SliceHistory : non
EXMODE : non
POINT : 32768 points
SAMP0 : 32768 points
FREQU : 7993.6 Hz
FILTR : 4000 Hz
DELAY : 50.0 usec
DEADT : 72.1 usec
INTVL : 125.1 usec
TIMES : 64 times
DUMMY : 0 times
PD : 2.9007 sec
ACQTM : 4099.2769 msec
PREDL : 10.00000 msec
INIWT : 0.5000 msec
RESOL : 0.24 Hz
PW1 : 5.75 usec
OBNUC : 1H
OBFRQ : 399.65 MHz
OBSET : 134300.00 Hz
RGAIN : 15
SCANS : 64 times
SLVNT : COCL3
SPINNING : 10 Hz
TEMP : 23.4 C



k002079a'

7.6537
7.6488
7.6390
7.6342
7.6299
7.6183
7.6134
7.6122
7.6097
7.6079
7.6061
7.5908
7.4737
7.4590
7.4572
7.4529
7.4487
7.4377
7.4328
7.4175
7.4127
7.3535
7.3468
7.3370
7.3309
7.3144
7.2955
7.2729
7.2693
7.2601
7.2552
7.2479
7.2363
7.2302
7.2211
7.2180
7.2003
5.3502
4.7016
4.2573
4.2219
4.1951
3.7942
3.7832
3.7576
3.7466
1.5737
1.4547
0.0000

Date : Sat Sep 20 19:50:10 2003
FileName : k002079a'.nmdata
Comment : k002079a'
SliceHistory : non
EXMODE : non
POINT : 32768 points
SAMPO : 32768 points
FREQU : 7993.6 Hz
FILTR : 4000 Hz
DELAY : 50.0 usec
DEAT : 72.1 usec
INTVL : 125.1 usec
TIMES : 8 times
DUMMY : 0 times
PD : 2.9007 sec
ACQTM : 4099.2769 msec
PREDL : 10.0000 msec
INIWT : 0.5000 msec
RESOL : 0.24 Hz
PW1 : 5.75 usec
1H : 399.65 MHz
OBFRQ : 134300.00 Hz
OBSET : 20
RGAIN : 8 times
SCANS : 8 times
SLVNT : CDCL3
SPINNING : 13 Hz
TEMP : 24.1 C



k002071a'

Date : Wed Sep 3 01:46:28 2003

FileName : k002071a'.nmdata
Comment : k002071a'
SliceHistory :
EXMODE : non

POINT : 32768 points
SAMP0 : 32768 points
FREQU : 7993.6 Hz
FILTR : 4000 Hz
DELAY : 50.0 usec
DEADT : 72.1 usec
INVL : 125.1 usec
TIMES : 64 times
DUMMY : 0 times
PD : 2.9007 sec
ACQTM : 4099.2769 msec
PREDL : 10.00000 msec
IN1WT : 0.5000 msec
RESOL : 0.24 Hz
PW1 : 5.75 usec
OBNUC : ¹H
OBFRQ : 399.65 MHz
OBSET : 134300.00 Hz
RGAIN : 20

SCANS : 64 times
SLVNT : CDCL3
SPINNING : 10 Hz
TEMP : 23.9 C

