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"Nitrogen-philic" Cyclization of Acyl Radicals onto N=C Bond. New Synthesis of 2-Pyrrolidinones by Radical Carbonylation/Annulation Method

Supporting Information

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Experimental Procedure and Spectral Data for Products 2a-2j

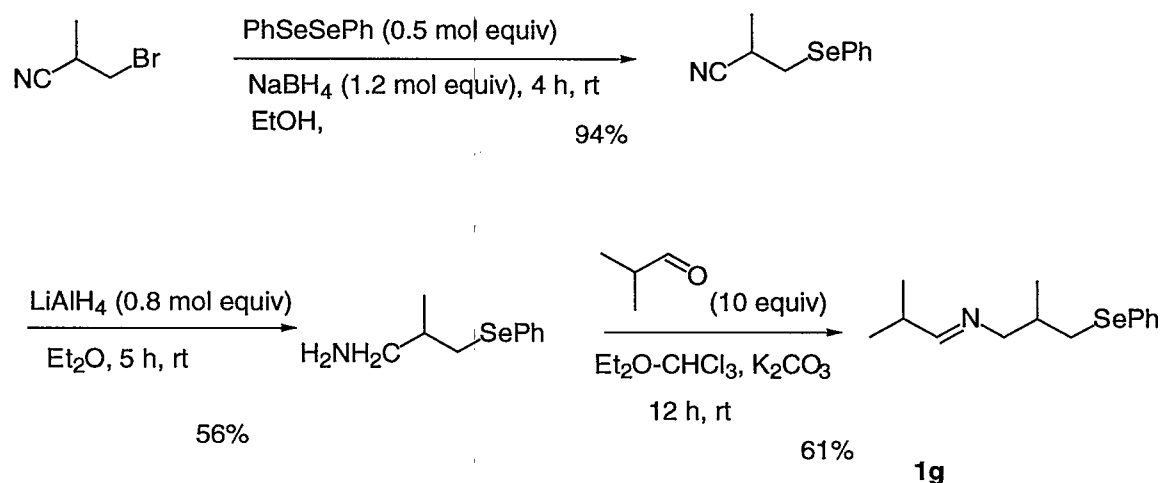
General Methods. ^1H NMR spectra were recorded with a JEOL JNM-GX67S (270 MHz) spectrometer and a Bruker AM600 (600 MHz) spectrometer. Chemical shifts are reported in parts per million (δ) downfield from internal TMS. ^{13}C NMR spectra were recorded with a JEOL JNM-GX67S (68MHz) spectrometer. Infrared spectra were recorded with a HITACHI 270-30 Infrared Spectrometer. Both conventional and high resolution mass spectra were recorded with a JEOL JMS-DX303HF spectrometer. The ratios of compounds were assayed with a Shimadzu GC-17A gas chromatography equipped with Supelco fused silica capillary column DB-1. The products were purified by flash chromatography on silica gel (Fuji Silysia BW-300) and, if necessary, were further purified by recycling preparative HPLC (JAI, JAIGEL-1H) equipped with a GPC column.

Preparation of 3-Bromo-N-cyclohexylmethylidenepropylamine (1a). HBr salt of 3-bromo-1-propylamine (29.46 g, 135 mmol) was treated with 1N NaOH (50 mL) and the mixture was extracted into benzene (50 mL x 5). To the combined benzene solutions was added a 100 mL benzene solution of cyclohexanecarboxaldehyde (17 mL, 140 mmol) dropwise. Then the solution was heated to reflux for two hours and during the reflux water was trapped by a Dean-Stark tube. The benzene solution was concentrated in vacuo and the vacuum distillation of the resulting residue gave 19.7 g (63%) of **1a** (72-74°C, 0.15 mmHg): ^1H -NMR (CDCl_3 , 270 MHz) δ 1.21-1.33 (m, 3H), 1.68-1.81 (m, 3H), 2.11-2.16 (m, 3H), 3.38-3.50 (m, 4H), 7.57 (d, 1H, $J = 5.0$ Hz); ^{13}C -NMR (CDCl_3 , 68 MHz) δ 25.42 (t), 25.98 (t), 29.70 (t), 31.54 (t), 34.00 (t), 43.46 (d), 58.51 (t), 170.51 (d); HRMS calcd for $\text{C}_{10}\text{H}_{18}\text{NBr}$ m/z 232.0623, found: 232.0686.

Bromoalkyl imines **1b**, **1c**, **1d**, and **1j** were prepared by a similar procedure in the yields of 65-72% and **1e** was prepared according to the Warkentin's report (reference 4c). Selected Spectral Data: **1b**: ^1H -NMR (CDCl_3 , 270 MHz) δ 1.48-1.62 (m, 2H), 1.85-1.91 (m, 2H), 2.15 (tt, 4H, $J = 6.3, 6.3$ Hz), 2.45 (bs, 1H), 3.41 (t, 2H, $J = 6.3$ Hz), 3.52 (t, 2H, $J = 6.3$ Hz), 5.70 (m,

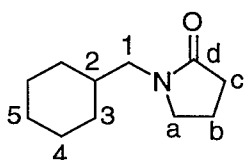
2H), 7.67 (d, 1H, $J = 5.0$ Hz); ^{13}C -NMR (CDCl_3 , 68 MHz) δ 25.13 (t), 25.62 (t), 27.91 (t), 31.50 (t), 32.94 (t), 39.19 (d), 58.47 (t), 125.34 (d), 126.99 (d), 169.64 (d); HRMS calcd for $\text{C}_{10}\text{H}_{16}\text{NBr}$ m/z 229.0466, found: 229.0466. **1c**: ^1H -NMR (CDCl_3 , 270 MHz) δ 2.25 (tt, 2H, $J = 6.3, 6.3$ Hz), 3.45 (t, 2H, $J = 6.3$ Hz), 3.75 (t, 2H, $J = 6.3$ Hz), 7.40-7.43 (m, 3H), 7.71-7.74 (m, 2H), ^{13}C -NMR (CDCl_3 , 68 MHz) δ 31.56 (t), 33.17 (t), 58.65 (t), 127.92 (d), 128.43 (d), 130.55 (d), 135.90 (s), 161.81 (d); HRMS calcd for $\text{C}_{10}\text{H}_{18}\text{NBr}$ m/z 231.0623, found: 232.0686 ($M + 1$). **1d**: ^1H -NMR (CDCl_3 , 270 MHz) δ 0.86-0.92 (m, 4H), 1.27-1.50 (m, 8H), 2.15 (tt, 4H, $J = 6.3, 6.3$ Hz), 3.43 (t, 2H, $J = 6.3$ Hz), 3.53 (t, 2H, $J = 6.3$ Hz), 7.46 (d, 1H, $J = 6.9$ Hz); ^{13}C -NMR (CDCl_3 , 68 MHz) δ 11.29 (q), 13.66 (q), 22.41 (t), 25.02 (t), 29.02 (t), 31.16 (t), 31.50 (t), 32.62 (t), 46.35 (d), 58.08 (t), 170.35 (d); HRMS calcd for $\text{C}_{11}\text{H}_{22}\text{NBr}$ m/z 247.0936, found: 247.0950. **1i**: ^1H -NMR (CDCl_3 , 270 MHz) δ 1.04-1.10 (m, 3H), 1.30-1.59 (m, 2H), 1.68 (s, 3H), 1.72 (s, 3H), 1.95-2.03 (m, 2H), 2.09-2.19 (m, 2H), 2.27-2.37 (m, 1H), 3.41 (td, 2H, $J = 6.3, 1.7$ Hz), 3.49 (t, 2H, $J = 6.3$ Hz), 5.07-5.12 (m, 1H), 7.55 (dd, 2H, $J = 6.3, 1.7$ Hz); ^{13}C -NMR (CDCl_3 , 68 MHz) δ 17.37 (q), 17.68 (q), 25.62 (t), 25.71 (q), 31.55 (t), 33.01 (t), 34.18 (t), 39.08 (d), 58.49 (t), 124.02 (d), 131.82 (s), 170.89 (d); HRMS calcd for $\text{C}_{12}\text{H}_{22}\text{NBr}$ m/z 259.0936, found: 260.1026.

Phenylselenenylimines **1f**, **1g**, and **1h** were prepared from cyanobromides via three steps according to the following Scheme depicted for the case of **1g** (see reference 5b).

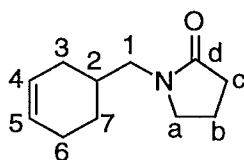


Scheme. Preparation of Phenylseleno Imine **1g**

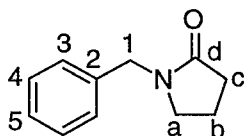
Typical Procedure: *N*-Cyclohexylmethyl-2-pyrrolidinone (2a). A magnetic stirring bar, AIBN (8.2 mg, 0.05 mmol), benzene (50 mL), Bu_3SnH (274.9 mg, 0.94 mmol), and imine **1a** (178.7 mg, 0.77 mmol) were placed in a 100-mL stainless steel autoclave lined with glass liner. The autoclave was closed, purged twice with carbon monoxide, pressurized with 80 atm of CO and then heated at 80 °C for 2 h. Excess CO was discharged at room temperature. The solvent was removed under reduced pressure, and the residue was dissolved in Et_2O (30 mL). The ethereal solution was treated with aqueous KF (10 mL) for 2 hours, and the resultant precipitate of Bu_3SnF was removed by vacuum filtration. The two liquid layers were separated, and aqueous layer was extracted with Et_2O (5 x 20 mL). The combined organic layers were dried (MgSO_4) and concentrated. The residue was purified by flash chromatography on silica gel (hexane/ EtOAc = 2/8 eluant). The major fraction (R_f = 0.23) eluted from the column contained 113.3 mg (81 %) of product **2a**: $^1\text{H-NMR}$ (CDCl_3 , 270 MHz) δ 0.90-0.97 (m, 2 H, H-3 or H-4), 1.16-1.38 (m, 3 H, , H-5, H-3 or H-4), 1.61-1.71 (m, 6 H, H-2, H-5, H-3 or H-4), 1.96-2.07 (tt, 2 H, J = 8.0, 7.0 Hz, H-b), 2.40 (t, 2 H, J = 8.0 Hz, H-c), 3.11 (d, 2 H, J = 7.0 Hz, H-1), 3.37 (t, 2 H, J = 7.0 Hz, H-a); $^{13}\text{C-NMR}$ (CDCl_3 , 68 MHz) δ 18.10 (t, C-b), 25.79 (t), 26.40 (t), 30.75 (t), 31.11 (t, C-c), 35.92 (d, C-2), 47.94 (t, C-a), 48.93 (t, C-1), 175.20 (s, C-d); EIMS (relative intensity) m/z 181 (M^+ , 13), 99 (57), 98 (100), 86 (52), 70 (31), 42 (18), 41 (48); IR(neat) 2928, 2856, 1692, 1450, 1286 cm^{-1} ; HRMS calcd for $\text{C}_{11}\text{H}_{19}\text{NO}$ m/z 181.1467, found: 181.1459.



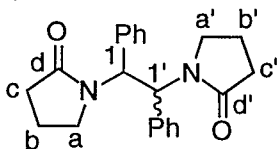
***N*-(Cyclohexen-4-ylmethyl)-2-pyrrolidinone (2b):** $^1\text{H-NMR}$ (CDCl_3 , 270 MHz) δ 1.18-1.35 (m, 1 H, H-3, 6 or 7), 1.69-2.08 (m, 8 H, H-2, H-3, H-6, H-7, H-b), 2.41 (t, 2 H, J = 7.3 Hz, H-c), 3.20 (d, 2 H, J = 7.3 Hz, H-1), 3.39 (t, 2 H, J = 7.3 Hz, H-a), 5.66 (m, 2 H, H-5); $^{13}\text{C-NMR}$ (CDCl_3 , 68 MHz) δ 18.10 (t, C-b), 24.64 (t), 26.29 (t), 26.40 (t), 31.07 (t, C-c), 32.04 (d, C-2), 47.85 (t, C-a), 48.16 (t, C-1), 125.59 (d, C-4 or C-5), 127.08 (d, C-4 or C-5), 175.25 (s, C-d); EIMS (relative intensity) m/z 179 (M^+ , 29), 99 (87), 98 (100), 78 (21), 70 (24), 41 (47); IR(neat) 2920, 1688, 1442, 1288, 652 cm^{-1} ; HRMS calcd for $\text{C}_{11}\text{H}_{17}\text{NO}$ m/z 179.1310, found: 179.1318.



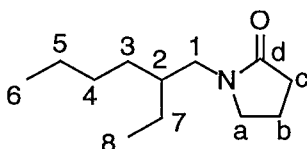
***N*-Benzyl-2-pyrrolidinone (2c):** $^1\text{H-NMR}$ (CDCl_3 , 270 MHz) δ 1.99 (quint, 2 H, $J = 7.6$ Hz, H-b), 2.45 (t, 2 H, $J = 7.6$ Hz, H-c), 3.27 (t, 2 H, $J = 7.6$ Hz, H-a), 4.45 (s, 2 H, H-1), 7.23-7.26 (m, 5 H, H-3, H-4, H-5); $^{13}\text{C-NMR}$ (CDCl_3 , 68 MHz) δ 17.67 (t, C-b), 30.89 (t, C-c), 46.56 (t, C-a, C-1), 127.48 (d), 128.05 (d), 128.61 (d), 136.51 (s, C-2), 174.91 (s, C-d); EIMS (relative intensity) m/z 175 (M^+ , 56), 146 (36), 104 (37), 91 (100), 84 (35), 65 (38), 41 (44); IR(neat) 1686, 1426, 1290, 1266, 702 cm^{-1} ; HRMS calcd for $\text{C}_{11}\text{H}_{13}\text{NO}$ m/z 175.0997, found: 175.0994; Anal. Calcd for $\text{C}_{11}\text{H}_{13}\text{NO}$: C, 75.40; H, 7.48; N, 8.00. Found: C, 75.62; H, 7.48; N, 7.70.



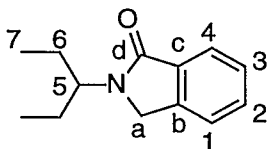
1, 1'-Bis-(*N*-phenylmethyl-2-pyrrolidinone) (2c'): Hexane/EtOAc (7:3), MeOH was used as the eluant to give each product as a colorless solid: major isomer (more polar): $^1\text{H-NMR}$ (CDCl_3 , 270 MHz) δ 1.79-1.94 (m, 4 H, H-b, H-b'), 2.33-2.39 (m, 4 H, H-c, H-c'), 2.85-2.94 (m, 2 H, H-a, H-a'), 3.67-3.76 (m, 2 H, H-a, H-a'), 6.08 (s, 2 H, H-1, H-1'), 7.13-7.31 (m, 10 H, Ph-H); $^{13}\text{C-NMR}$ (CDCl_3 , 68 MHz) δ 18.08 (t, C-b, C-b'), 31.48 (t, C-c, C-c'), 43.25 (t, C-a, C-a'), 53.06 (d, C-1, C-1'), 127.87 (d, Ph), 128.61 (d, Ph), 128.89 (d, Ph), 135.72 (s, Ph), 175.06 (s, C-d, C-d'); EIMS (relative intensity) m/z 175 (13), 174 (100), 131 (16), 91 (12), 69 (18), 41 (32); IR (KBr) 1684, 1426, 1288, 1270, 702 cm^{-1} ; HRMS calcd for $\text{C}_{22}\text{H}_{24}\text{N}_2\text{O}_2$ m/z 348.1837, found: 348.1910; Anal. Calcd for $\text{C}_{22}\text{H}_{24}\text{N}_2\text{O}_2$: C, 75.83; H, 6.94; N, 8.04. Found: C, 75.43; H, 6.97; N, 7.99., minor isomer (less polar): $^1\text{H-NMR}$ (CDCl_3 , 270 MHz) δ 1.48-1.78 (m, 4 H, H-b, H-b'), 1.95-2.21 (m, 4 H, H-c, H-c'), 3.10-3.22 (m, 4 H, H-a, H-a'), 6.08 (s, 2 H, H-1, H-1'), 7.27-7.56 (m, 10 H, Ph-H); $^{13}\text{C-NMR}$ (CDCl_3 , 68 MHz) δ 17.79 (t, C-b, C-b'), 30.74 (t, C-c, C-c'), 42.19 (t, C-a, C-a'), 52.94 (d, C-1, C-1'), 128.12 (d, Ph), 128.28 (d, Ph), 128.75 (d, Ph), 135.89 (s, Ph), 174.52 (s, C-d, C-d'); EIMS (relative intensity) m/z 175 (12), 174 (100), 131 (13), 91 (13), 69 (16), 41 (33); IR(KBr) 1668, 1426, 1422, 1280, 1182, 696 cm^{-1} ; HRMS calcd for $\text{C}_{22}\text{H}_{24}\text{N}_2\text{O}_2$ m/z 348.1837, found: 349.1923.



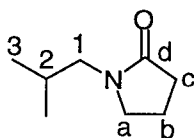
***N*-(2-Ethylhexyl)-2-pyrrolidinone (2d):** $^1\text{H-NMR}$ (CDCl_3 , 270 MHz) δ 0.86-0.94 (m, 6 H, H-6, H-8), 1.29-1.33 (m, 8 H, H-3, H-4, H-5, H-7), 1.61 (m, 1 H, H-2), 2.05 (q, 2 H, $J = 7.3$ Hz, H-b), 2.42 (t, 2 H, $J = 7.3$ Hz, H-c), 3.13-3.28 (m, 2 H, H-1), 3.38 (t, 2 H, $J = 7.3$ Hz, H-a); $^{13}\text{C-NMR}$ (CDCl_3 , 68 MHz) δ 10.58 (q, C-6 or C-8), 14.09 (q, C-6 or C-8), 18.08 (t, C-b), 23.05 (t, C-5), 23.86 (t, C-7), 28.70 (t, C-4), 30.58 (t, C-3), 31.16 (t, C-c), 37.12 (d, C-2), 46.36 (t, C-1), 47.56 (t, C-a), 175.20 (s, C-d); EIMS (relative intensity) m/z 197 (M^+ , 7), 99 (38), 98 (100), 86 (15), 70 (18), 43 (10), 42 (12), 41 (35); IR(neat) 2964, 2932, 1692 cm^{-1} ; HRMS calcd for $\text{C}_{12}\text{H}_{23}\text{NO}$ m/z 197.1780, found: 198.1861; Anal. Calcd for $\text{C}_{12}\text{H}_{23}\text{NO}$: C, 73.00; H, 11.70; N, 7.10. Found: C, 72.92; H, 11.82; N, 7.23.



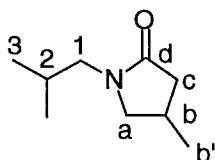
***N*-(1-Ethylpropyl)-2-phthalimidine (2e):** $^1\text{H-NMR}$ (CDCl_3 , 270 MHz) δ 0.87 (t, 6 H, $J = 7.3$ Hz, H-7), 1.52-1.75 (m, 4 H, H-6), 4.20-4.23 (m, 1 H, H-5), 4.25 (s, 2 H, H-a), 7.43-7.56 (m, 3 H, H-2, H-3, H-1 or H-4), 7.85-7.88 (m, 1 H, H-1 or H-4); $^{13}\text{C-NMR}$ (CDCl_3 , 68 MHz) δ 11.31 (q, C-7), 26.88 (t, C-6), 45.32 (t, C-a), 54.83 (d, C-5), 123.11 (d), 124.13 (d), 128.34 (d), 131.43 (d), 133.51 (s, C-b or C-c), 141.56 (s, C-b or C-c), 169.58 (s, C-d); EIMS (relative intensity) m/z 203 (M^+ , 4), 175 (16), 174 (100), 134 (7), 132 (6), 119 (5), 91 (8); IR(neat) 1676, 1452, 1410, 1214, 730 cm^{-1} ; HRMS calcd for $\text{C}_{13}\text{H}_{17}\text{NO}$ m/z 203.1310, found: 203.1319.



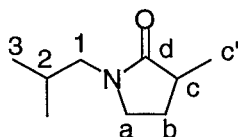
***N*-(2-Methylpropyl)-2-pyrrolidinone (2f):** $^1\text{H-NMR}$ (CDCl_3 , 270 MHz) δ 0.89 (d, 6 H, $J = 6.6$ Hz, H-3), 1.91 (t of septet, 1 H, $J = 6.6, 7.3$ Hz, H-2), 2.01 (tt, 2 H, $J = 7.3, 7.9$ Hz, H-b), 2.40 (t, 2 H, $J = 7.9$ Hz, H-c), 3.08 (d, 2 H, $J = 7.3$ Hz, H-1), 3.37 (t, 2 H, $J = 7.3$ Hz, H-a); $^{13}\text{C-NMR}$ (CDCl_3 , 68 MHz) δ 17.92 (t, C-b), 19.86 (q, C-3), 26.45 (d, C-2), 30.93 (t, C-c), 47.55 (t, C-a), 49.96 (t, C-1), 175.06 (t, C-d); EIMS (relative intensity) m/z 141 (M^+ , 28), 126 (11), 98 (100), 70 (31), 69 (11), 41 (12); IR(neat) 1674, 1462, 1274 cm^{-1} ; HRMS calcd for $\text{C}_8\text{H}_{15}\text{NO}$ m/z 141.1154, found: 141.1142.



***N*-(2-Methylpropyl)-4-methyl-2-pyrrolidinone (2g):** $^1\text{H-NMR}$ (CDCl_3 , 270 MHz) δ 0.89 (d, 6 H, $J = 6.6$ Hz, H-3), 1.13 (d, 3 H, $J = 6.6$ Hz, H-b'), 1.90 (t of septet, 1 H, $J = 6.9$, 6.6 Hz, H-2), 2.04 (dd, 1 H, $J = 16.0$, 6.3 Hz, H-c), 2.43 (ddqdd, 1 H, $J = 8.6$, 7.6, 6.6, 6.3, 5.6 Hz, H-b), 2.56 (dd, 1 H, $J = 16.0$, 8.6 Hz, H-c), 2.95 (dd, 1 H, $J = 9.6$, 5.6 Hz, H-a), 3.07 (d, 2 H, $J = 6.9$ Hz, H-1), 3.48 (dd, 1 H, $J = 9.6$, 7.6 Hz, H-a); $^{13}\text{C-NMR}$ (CDCl_3 , 68 MHz) δ 20.11 (q, C-3), 20.16 (q, C-3'), 20.22 (q, C-b'), 26.65 (d, C-2), 26.78 (d, C-b), 39.68 (t, C-c), 50.12 (t, C-1), 55.22 (t, C-a), 174.84 (s, C-d); EIMS (relative intensity) m/z 155 (M^+ , 25), 113 (11), 112 (100), 84 (25), 83 (14); IR(neat) 2924, 1650, 1572, 1452, 1264 cm^{-1} ; HRMS calcd for $\text{C}_9\text{H}_{17}\text{NO}$ m/z 155.1310, found: 155.1316.

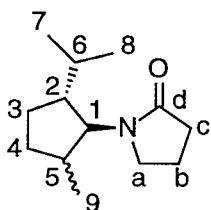


***N*-(2-Methylpropyl)-3-methyl-2-pyrrolidinone (2h):** $^1\text{H-NMR}$ (CDCl_3 , 600 MHz) δ 0.89 (dd, 6 H, $J = 6.6$, 0.6 Hz, H-3), 1.22 (d, 3 H, $J = 7.2$ Hz, H-c'), 1.63 (dq, 1 H, $J = 12.6$, 8.4 Hz, H-b), 1.86-1.95 (9-lines-m, 1 H, $J = 6.6 \sim 7.8$ Hz, H-2), 2.21-2.27 (m, 1 H, H-b), 2.50 (tq, 1 H, $J = 8.4$, 7.2 Hz, H-c), 3.08 (dd, 1 H, $J = 13.2$, 7.8 Hz, H-1), 3.09 (dd, 1 H, $J = 13.2$, 7.8 Hz, H-1), 3.26-3.33 (m, 2 H H-a); $^{13}\text{C-NMR}$ (CDCl_3 , 68 MHz) δ 16.91 (q, C-c'), 20.45 (q, C-3), 27.06 (d, C-2), 27.82 (t, C-b), 37.29 (d, C-c), 46.15 (t, C-a), 50.69 (t, C-1), 178.02 (s, C-d); EIMS (relative intensity) m/z 155 (M^+ , 40), 140 (12), 113 (13), 112(100), 100 (11), 84 (77), 55 (11), 42 (13); IR(neat) 2932, 2876, 1680, 1456, 1432 cm^{-1} ; HRMS calcd for $\text{C}_9\text{H}_{17}\text{NO}$ m/z 155.1310, found: 155.1313.

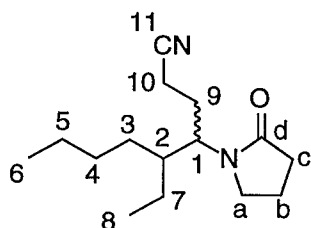


***N*-(2-Isopropyl-5-methylcyclopentyl)-2-pyrrolidinone (2i):** Hexane/EtOAc (5:5) was used as the eluant to give a colorless oil as a mixture of two diastereoisomers (major:minor = 78:22 based on $^1\text{H-NMR}$). $^1\text{H-NMR}$ (C_6D_6 , 270 MHz) (signals for minor isomer are in square brackets) δ 0.84-0.98 (m, 10 H, H-7, H-8, H-9, H-3 or H-4), 1.05-1.36 (m, 3 H, H-b, H-3 or H-4),

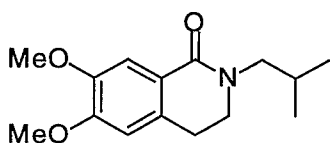
1.44-1.80 (m, 4 H, H-2, H-3, H-4, H-5), 1.91-2.09 (m, 2 H, H-c), 2.62-2.77 (m, 2 H, H-a), 4.40 (dd, 1 H, $J = 2.3, 7.4$ Hz, H-1) [3.95 (dd, 1 H, $J = 9.6, 9.6$ Hz, H-1)]; ^{13}C -NMR (C_6D_6 , 68 MHz) (signals for minor isomer are in square brackets) δ 18.54 (t, C-b) [17.52 (t, C-b)], 21.73 (q, C-9) [19.14 (q, C-9)], 22.23 (q, C-7 or C-8) [20.77 (q, C-7 or C-8)], 22.57 (q, C-7 or C-8) [24.62 (q, C-7 or C-8)], 28.81 (d, C-6) [30.31 (d, C-6)], 31.11 (t, C-c) [30.39 (t, C-c)], 31.16 (t, C-3 or C-4) [31.90 (t, C-3 or C-4)], 33.14 (t, C-3 or C-4) [36.60 (t, C-3 or C-4)], 37.92 (d, C-5) [42.59 (d, C-5)], 46.47 (t, C-a) [46.04 (t, C-a)], 50.73 (d, C-2) [50.73 (d, C-2)], 60.05 (d, C-1) [61.67 (d, C-1)], 174.98 (s, C-d) [175.24 (s, C-d)]; EIMS (relative intensity) major isomer: m/z 209 (M^+ , 7), 98 (22), 86 (100), 81 (14), 55 (12), 43 (11), 41 (40), minor isomer: m/z 209 (M^+ , 10), 166 (13), 98 (60), 86 (100), 81 (45), 55 (33), 41 (70); IR(neat) a mixture of two isomers: 3308, 2960, 1702, 1428 cm^{-1} ; HRMS (signals for minor isomer are in square brackets) calcd for $\text{C}_{13}\text{H}_{23}\text{NO}$ m/z 209.1780, found: 209.1781 [209.1795].



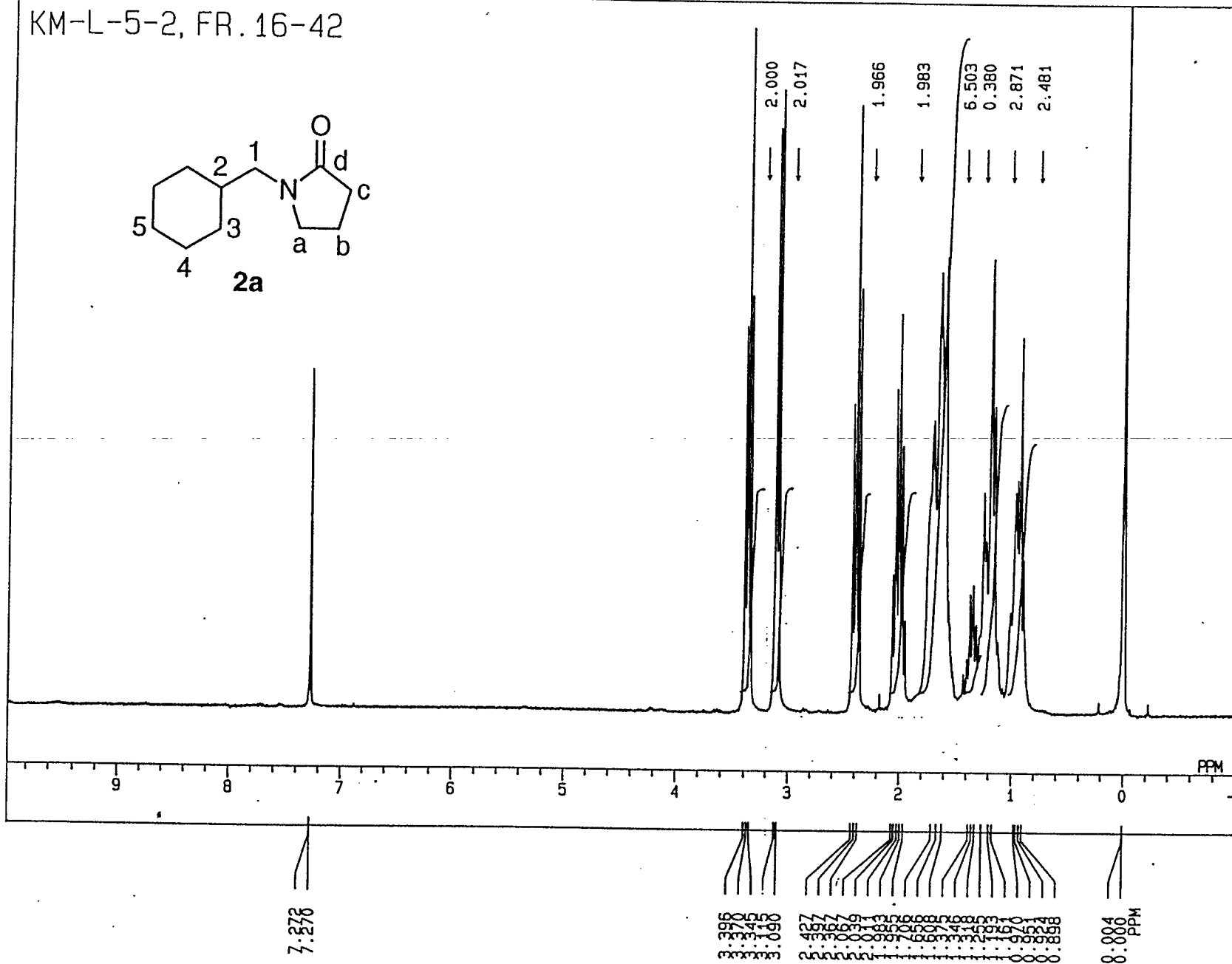
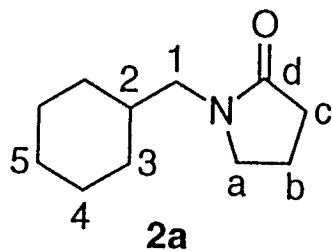
***N*-[1-(2-Cyanoethyl)-2-ethylhexyl]-2-pyrrolidinone (2j):** Hexane/EtOAc (8:2) was used as the eluant to give a colorless oil as a mixture of two diastereoisomers (major:minor = 53:47 based on GC). ^1H -NMR (CDCl_3 , 270 MHz) δ 0.84-0.91 (m, 6 H, H-6, H-8), 1.14-1.53 (m, 9 H, H-2, H-3, H-4, H-5, H-7), 1.77-1.82 (m, 1 H, H-9), 1.97-2.45 (m, 7 H, H-b, H-c, H-9, H-10), 3.23-3.32 (m, 2 H, H-a), 3.93-3.97 (m, 1 H, H-1); ^{13}C -NMR (C_6D_6 , 68 MHz) (signals for minor isomer are in square brackets) δ 18.54 (t, C-b) [17.52 (t, C-b)], 21.73 (q, C-9) [19.14 (q, C-9)], 22.23 (q, C-7 or C-8) [20.77 (q, C-7 or C-8)], 22.57 (q, C-7 or C-8) [24.62 (q, C-7 or C-8)], 28.81 (d, C-6) [30.31 (d, C-6)], 31.11 (t, C-c) [30.39 (t, C-c)], 31.16 (t, C-3 or C-4) [31.90 (t, C-3 or C-4)], 33.14 (t, C-3 or C-4) [36.60 (t, C-3 or C-4)], 37.92 (d, C-5) [42.59 (d, C-5)], 46.47 (t, C-a) [46.04 (t, C-a)], 50.73 (d, C-2) [50.73 (d, C-2)], 60.05 (d, C-1) [61.67 (d, C-1)], 174.98 (s, C-d) [175.24 (s, C-d)]; EIMS (relative intensity) major isomer: m/z 250 (M^+ , 1), 151 (100), 124 (41), 86 (13), 69 (17), 41 (57) minor isomer: m/z 250 (M^+ , 1), 151 (100), 124 (43), 86 (15), 69 (17), 41 (64); IR(neat): 2948, 1672, 1422, 1282 cm^{-1} ; HRMS (signals for minor isomer are in square brackets) calcd for $\text{C}_{15}\text{H}_{26}\text{N}_2\text{O}$ m/z 250.2045, found: 250.2047 [250.2041].



***N*-(2-Methylpropyl)-3,4-dihydro-6,7-dimethoxy-1(2H)-isoquinolinone:** $^1\text{H-NMR}$ (CDCl_3 , 270 MHz) δ 0.94 (d, 6 H, $J = 7$ Hz), 2.02 (m, 1H), 2.91 (t, 2H, $J = 6.7$ Hz), 3.36 (d, 2H, $J = 7.6$ Hz), 3.52 (t, 2H, $J = 6.7$ Hz), 3.90 (s, 3H), 3.91 (s, 3H), 6.64 (s, 1H), 7.60 (s, 3H); $^{13}\text{C-NMR}$ (CDCl_3 , 68 MHz) δ 20.04, 27.15, 27.62, 46.70, 54.56, 55.83, 109.04, 110.41, 122.10, 131.38, 147.73, 151.41, 164.37; EIMS (relative intensity) m/z 263 (M^+ , 20), 220 (100), 207 (28), 105 (19), 77 (16); IR(neat) 2936, 1635, 1600, 1456, 1338, 1272, 1092, 1012, 740 cm^{-1} .

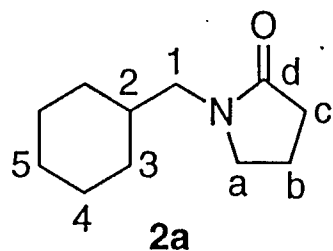


KM-L-5-2, FR. 16-42

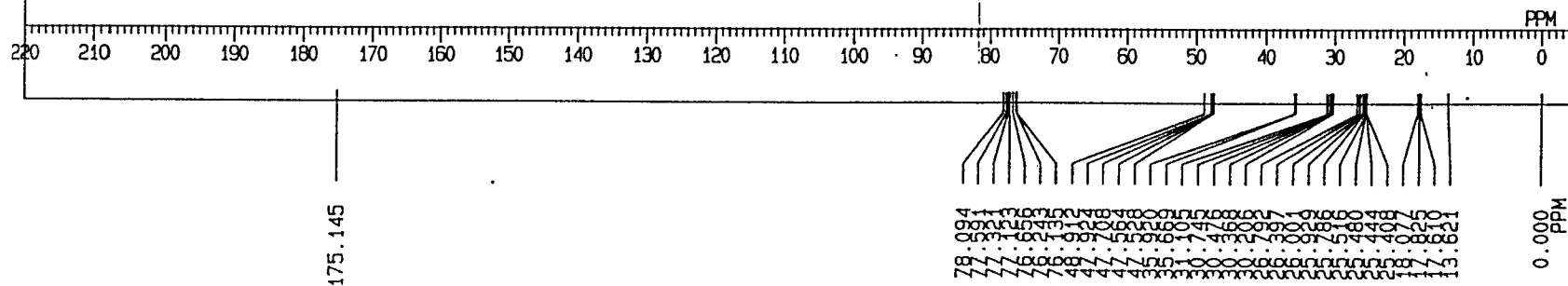


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 FREQU 5405.4 Hz
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 PD 10.000 sec
 PW1 4.7 us
 IRNUC 1H
 CTEMP 20.7 c
 SLVNT CDCL3
 EXREF 0.00 ppm
 BF 0.10 Hz
 RGAIN 18
 OPERATOR :

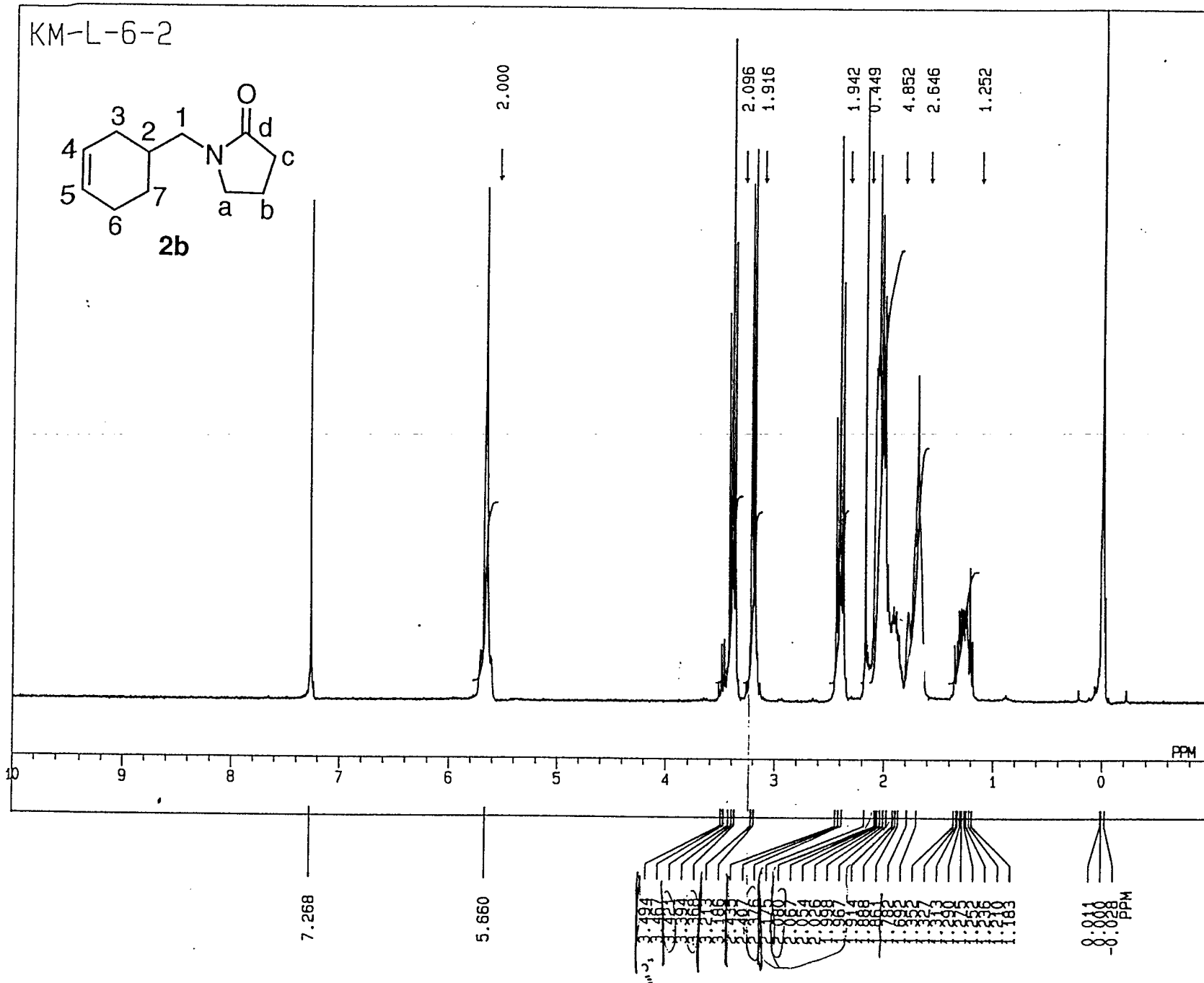
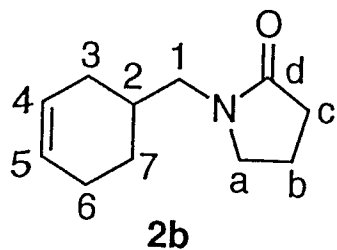
KM-L-5 (GPC)



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FREQU 20000.0 Hz
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CTEMP 21.7 c
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BF 1.50 Hz
RGAIN 32
OPERATOR: _____



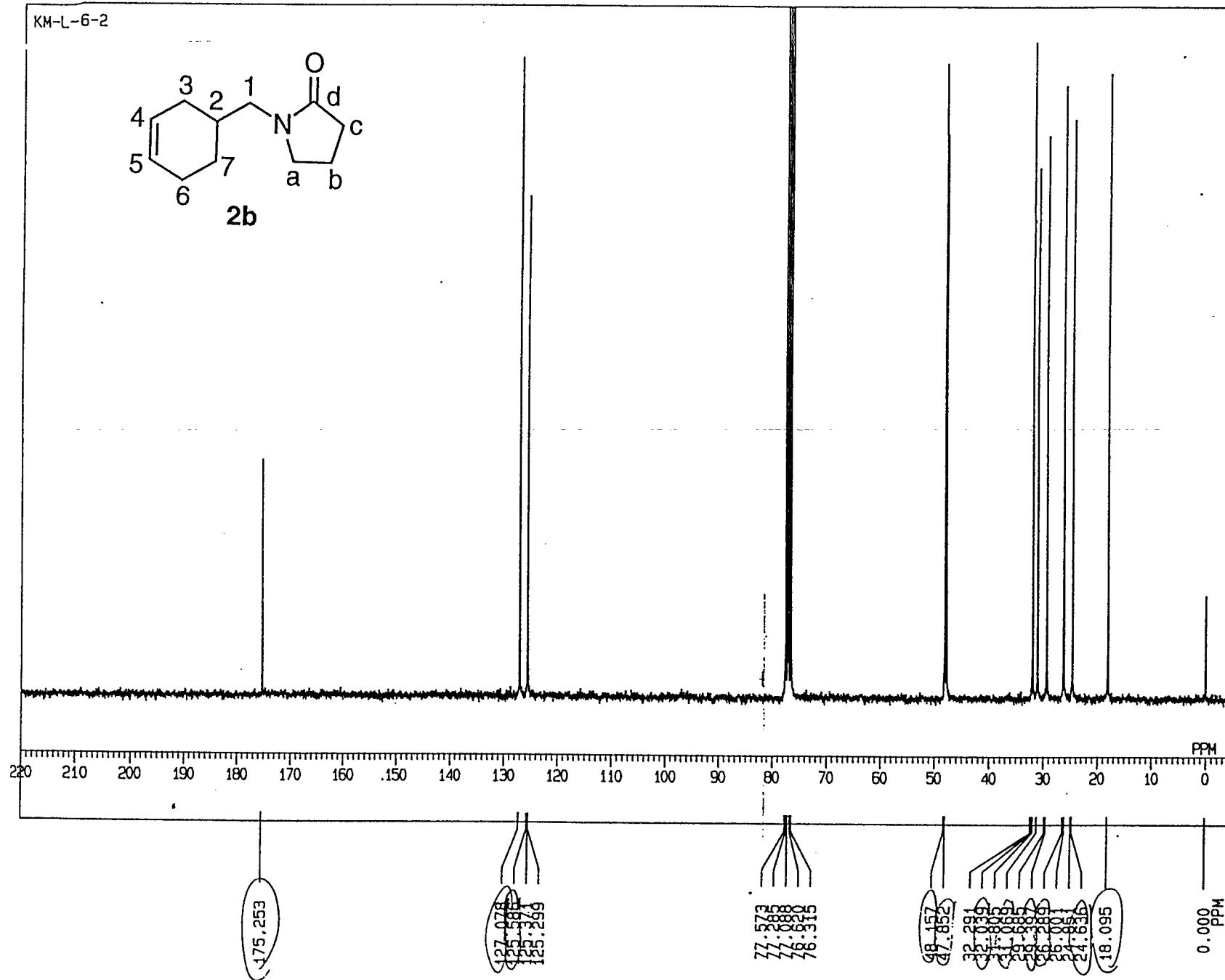
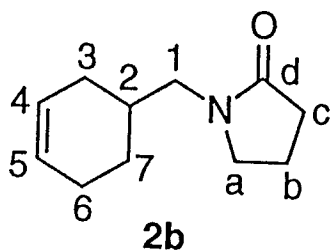
KM-L-6-2



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 POINT 32768
 FREQU 5405.4 Hz
 SCANS 32
 ACQTM 3.031 sec
 PD 10.000 sec
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 IRNUC 1H
 CTEMP 21.1 c
 SLVNT COCL3
 EXREF 0.00 ppm
 BF 0.10 Hz
 RGAIN 19
 OPERATOR :

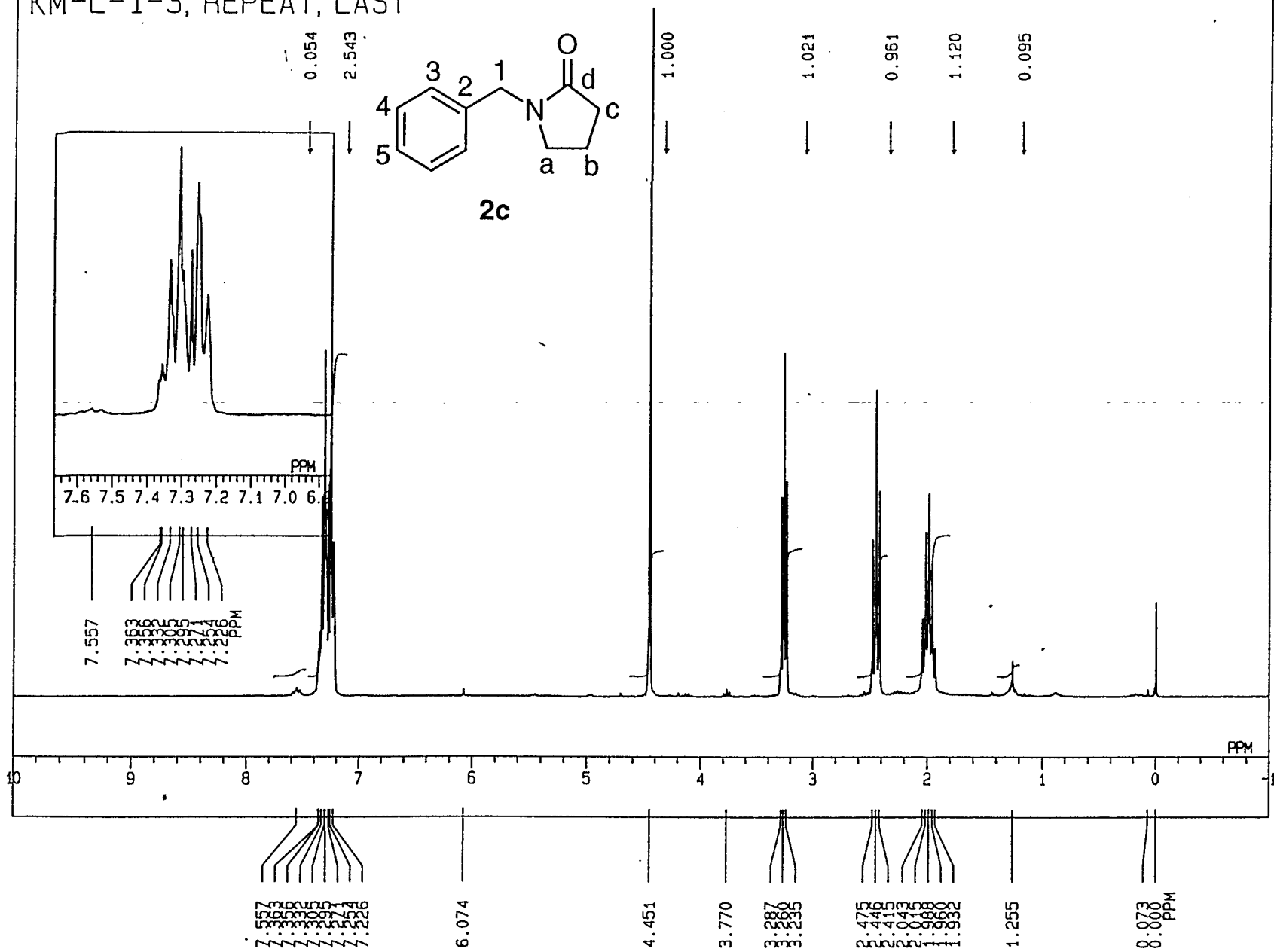
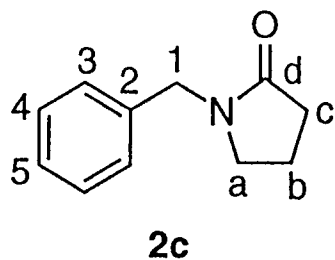
S11

KM-L-6-2



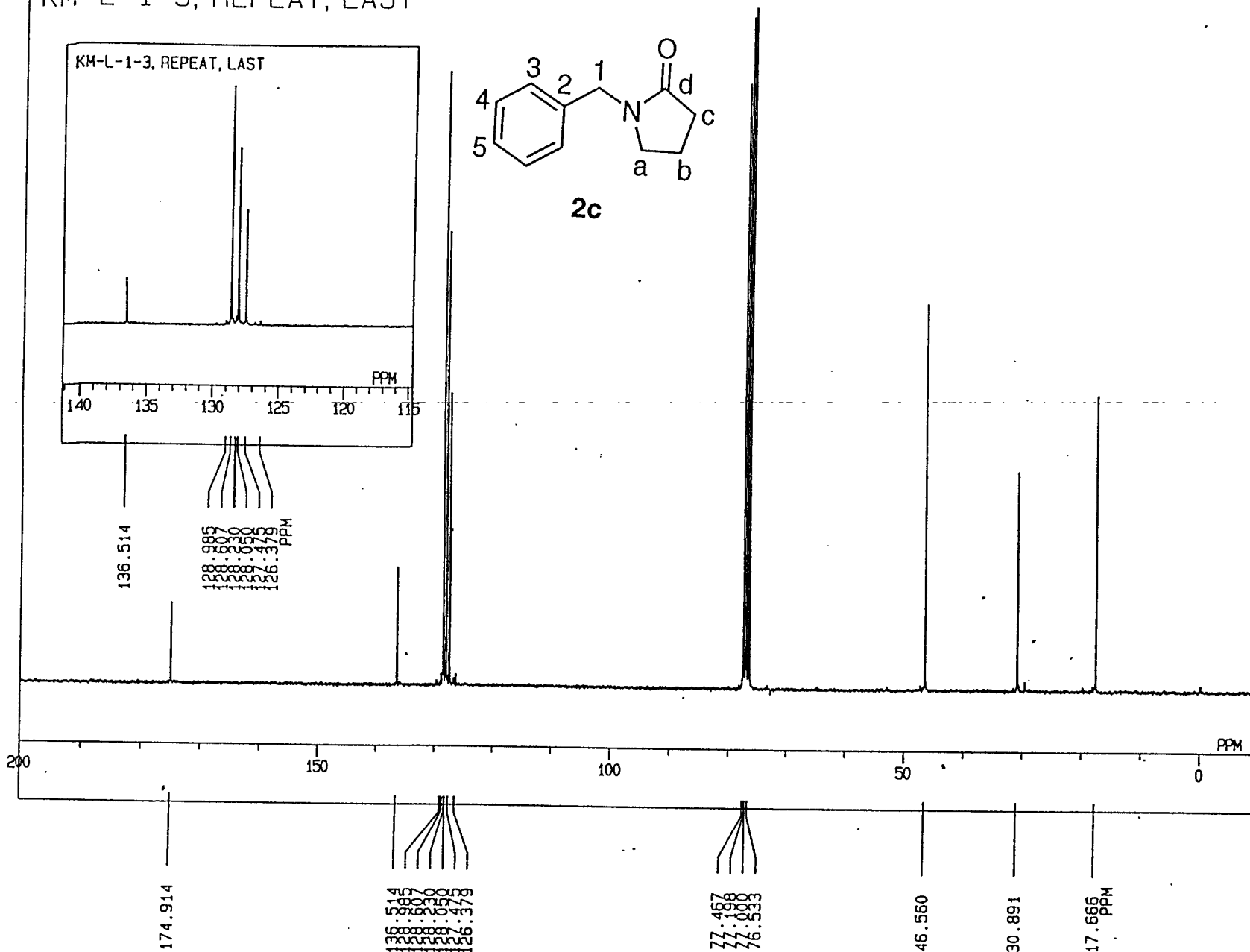
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 OBSET 135.00 kHz
 OBFIN 5200.0 Hz
 POINT 32768
 FREQU 20000.0 Hz
 SCANS 3685
 ACQTM 0.819 sec
 PD 2.181 sec
 PW1 4.0 us
 IRNUC 1H
 CTEMP 21.5 c
 SLVNT CDCL3
 EXREF 0.00 ppm
 BF 1.50 Hz
 RGAIN 32
 OPERATOR : _____

KM-L-1-3, REPEAT, LAST



04-OCT-95 17: 01: 23
 DFILE SAVING
 OBNUC 1H
 EXMOD NON
 OFR 270.05 MHz
 OBSET 112.00 kHz
 OBFIN 5800.0 Hz
 POINT 32768
 FREQU 5405.4 Hz
 SCANS 16
 ACQTM 3.031 sec
 PD 3.969 sec
 PW1 4.7 us
 IRNUC 1H
 CTEMP 23.7 c
 SLVNT CDCL3
 EXREF 0.00 ppm
 BF 0.10 Hz
 RGAIN 16
 OPERATOR :

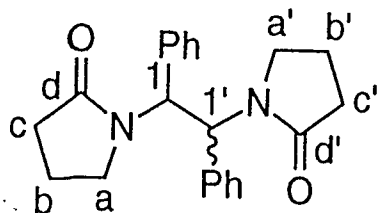
KM-L-1-3, REPEAT, LAST



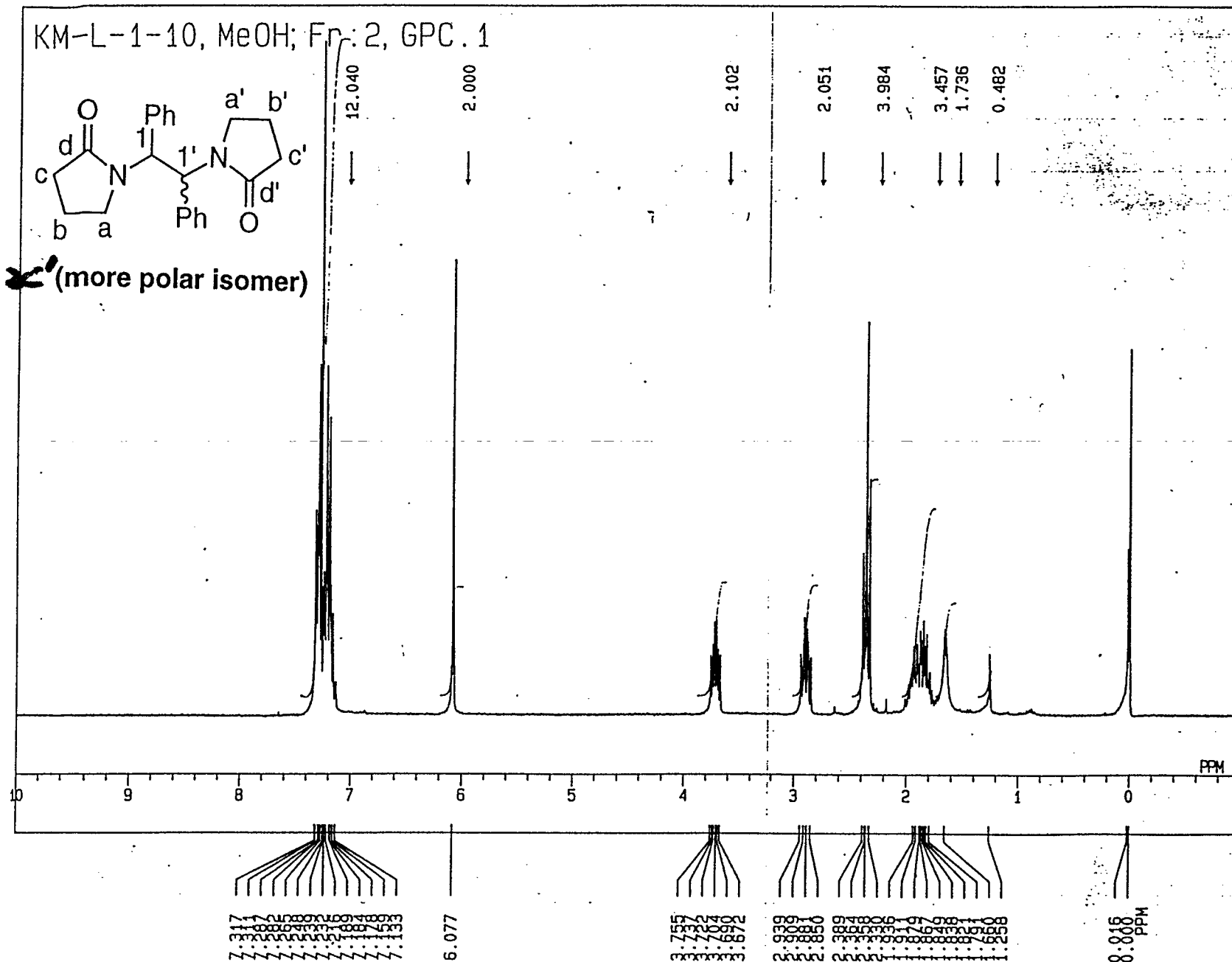
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 OBFIN 5200.0 Hz
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 FREQU 20000.0 Hz
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 PW1 4.0 us
 IRNUC 1H
 CTEMP 0.0 c
 SLVNT CDCL3
 EXREF 77.00 ppm
 BF 1.22 Hz
 RGAIN 18
 OPERATOR: _____

S14

KM-L-1-10, MeOH; Fr: 2, GPC .1



~~2~~' (more polar isomer)



13-NOV-95 21: 40: 54

DFILE SAVING

OBNUC 1H

EXMOD NON

OFR 270.05 MHz

OBSET 112.00 kHz

OBFIN 5800.0 Hz

POINT 32768

FREQU 5405.4 Hz

SCANS 16

ACQTM 3.031 sec

PD 3.969 sec

PW1 4.7 us

IRNUC 1H

CTEMP 19.9 c

SLVNT CDCL3

EXREF 0.00 ppm

BF 0.10 Hz

RGAIN 21

OPERATOR :

S15

The chemical structure is N,N'-diphenylmaleimide. It consists of two maleimide rings connected by a diphenylmethane bridge. The bridgehead carbons are labeled 1 and 1'. The protons on the rings are labeled as follows: the maleimide ring on the left has protons labeled a, b, c, and d; the maleimide ring on the right has protons labeled a', b', c', and d'. The carbonyl groups are labeled O and O'.

KM-L-1-10, MeOH; Fr :2, GPC:1

The chemical structure shows a central chiral carbon atom bonded to two phenyl groups (Ph) and two pyrrolidin-2-one rings. The left pyrrolidinone ring has protons labeled a, b, c, and d. The right pyrrolidinone ring has protons labeled a', b', c', and d'. The central carbon is also labeled 1 and 1'.

2c' (more polar isomer)

The ¹H NMR spectrum shows several peaks in the aromatic region (6.5-7.5 ppm), a large solvent peak for MeOH at approximately 3.3 ppm, and several peaks in the aliphatic region (1.5-2.5 ppm). The x-axis is labeled PPM and ranges from 0 to 200.

175.055

135.721

2000

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77.501

44:23-24

76:548

53.063

43.252

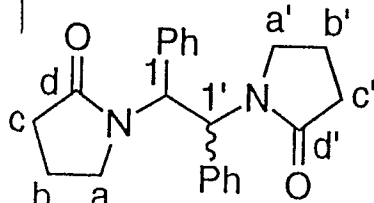
31.482

18.077

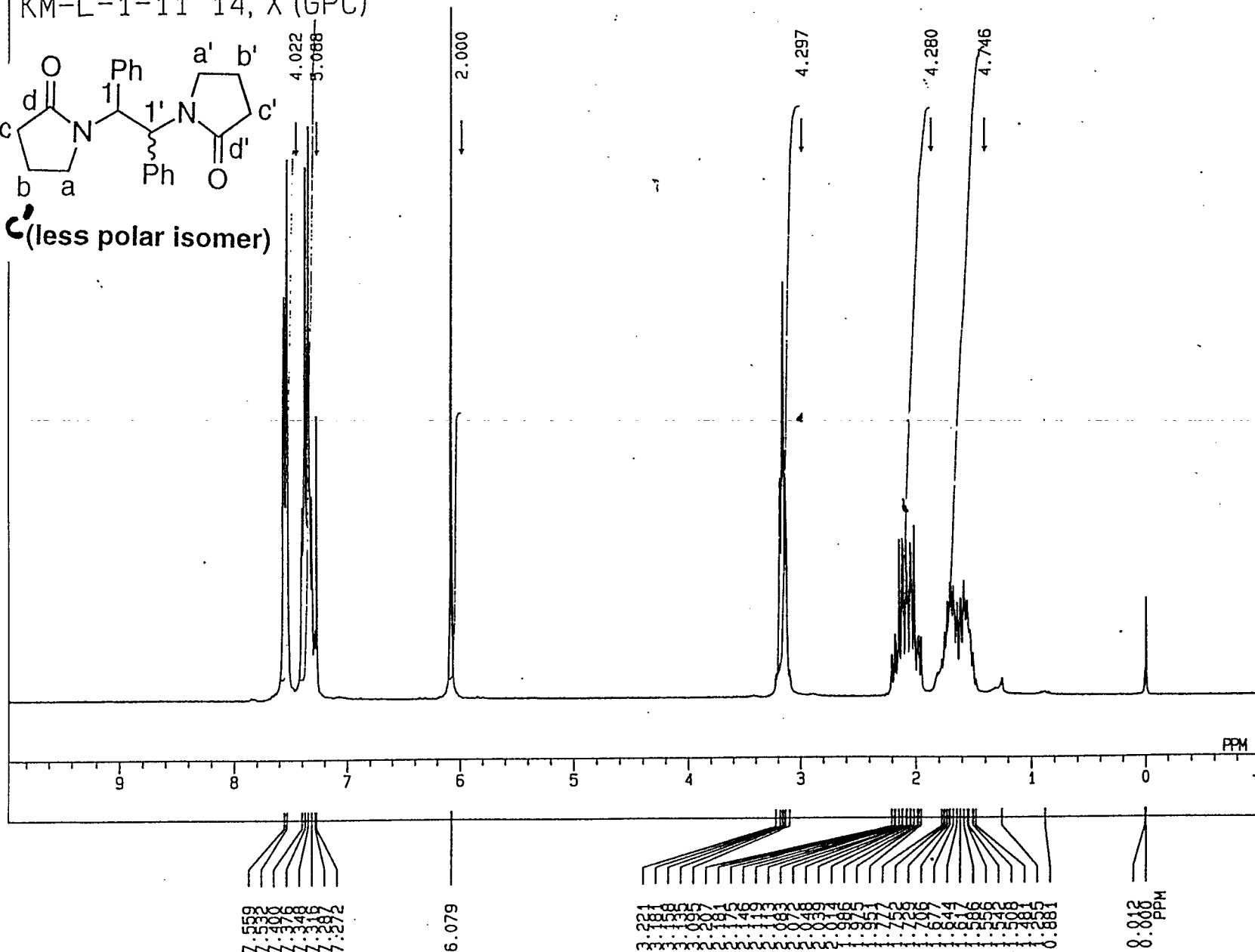
0.000 PPM

Fr. 30 ~,

KM-L-1-11~14, X (GPC)



2c' (less polar isomer)



25-NOV-95 02:03:47

DFILE SAVING

OBNUC 1H

EXMOD' NON

OFR 270.05 MHz

OBSET 112.00 kHz

OBFIN 5800.0 Hz

POINT 32768

FREQU 5405.4 Hz

SCANS 32

ACQTM 3.031 sec

PD 10.000 sec

PW1 4.7 us

IRNUC 1H

CTEMP 18.9 c

SLVNT' CDCL3

EXREF 0.00 ppm

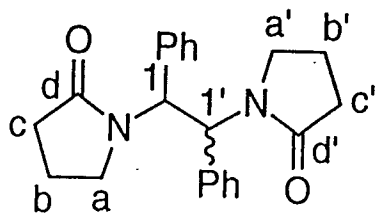
BF 0.10 Hz

RGAIN 17

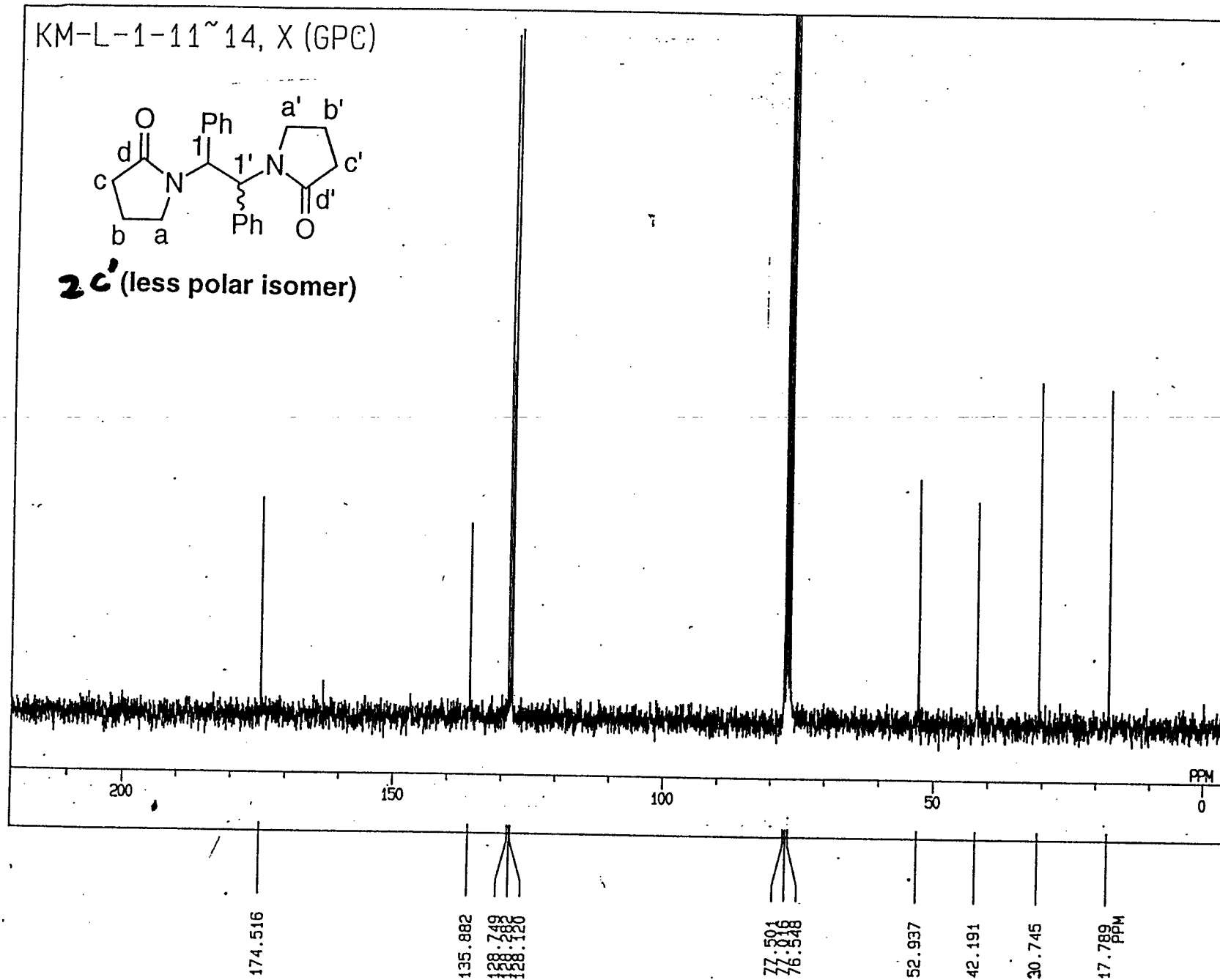
OPERATOR :

S17

KM-L-1-11~14, X (GPC)

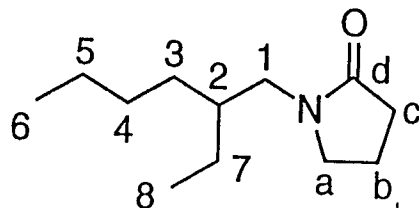


2c' (less polar isomer)

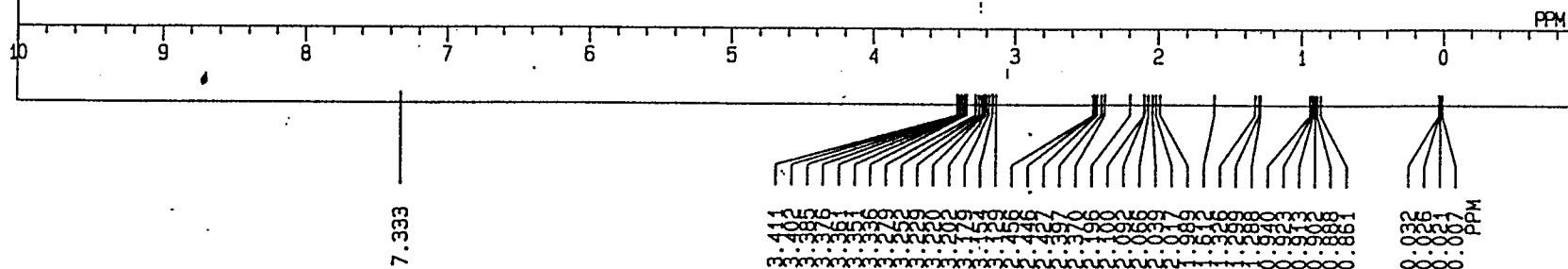
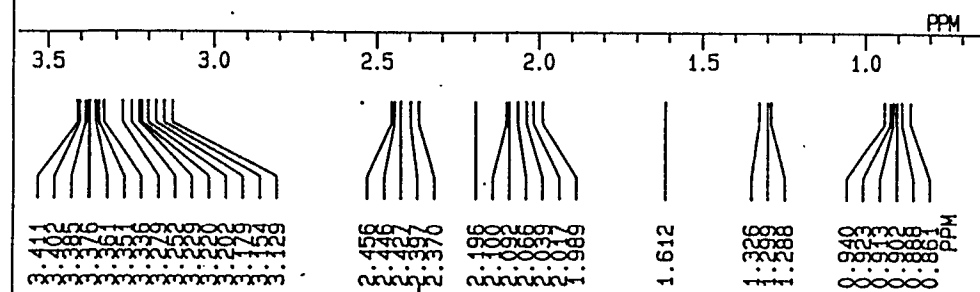


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 SLVNT CDCL3
 EXREF 0.00 ppm
 BF 1.22 Hz
 RGAIN 18
 OPERATOR : _____

KM-L-3



2d

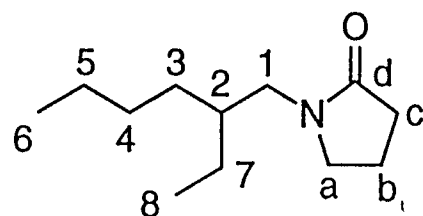


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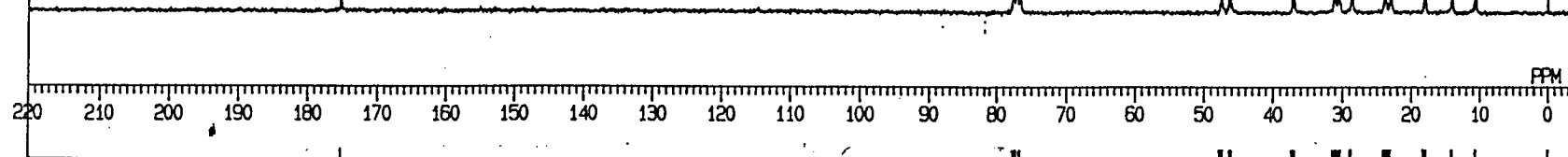
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BF 0.10 Hz
RGAIN 12
OPERATOR:

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KM-L-3



2d



O 175.199

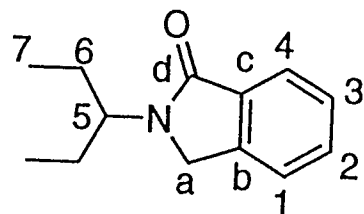
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0.000 PPM

P3-JAN-96 04: 48: 39
 DFILE Q13C
 OBNUC 13C
 EXMOD BCM
 OFR 67.80 MHz
 OBSET 135.00 kHz
 OBFIN 5200.0 Hz
 POINT 32768
 FREGU 20000.0 Hz
 SCANS 4290
 ACQTM 0.819 sec
 PD 2.181 sec
 PW1 4.0 us
 IRNUC 1H
 CTEMP 20.6 c
 SLVNT CDCL3
 EXREF 0.00 ppm
 BF 1.50 Hz
 RGAIN 32
 OPERATOR :

S20

KM-L-10-12, Fr. 1-2, GPC3



2e

0.939

2.995

3.000

0.330

4.191

0.381

5.956

31-OCT-97 02:04:29

DFILE SAVING

OBNUC 1H

EXMOD NON

QFR 270.05 MHz

OBSET 112.00 kHz

OBFIN 5800.0 Hz

POINT 32768

FREQU 5405.4 Hz

SCANS 100

ACQTM 3.031 sec

PD 3.969 sec

PW1 4.7 us

IRNUC 1H

CTEMP 23.3 c

SLVNT CDCL3

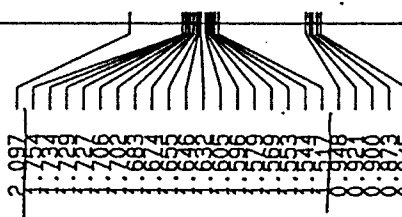
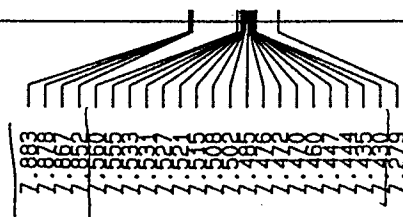
EXREF 0.00 ppm

BF 0.10 Hz

RGAIN 15

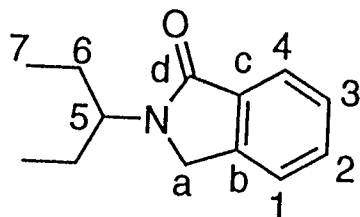
OPERATOR :

10 9 8 7 6 5 4 3 2 1 0 PPM

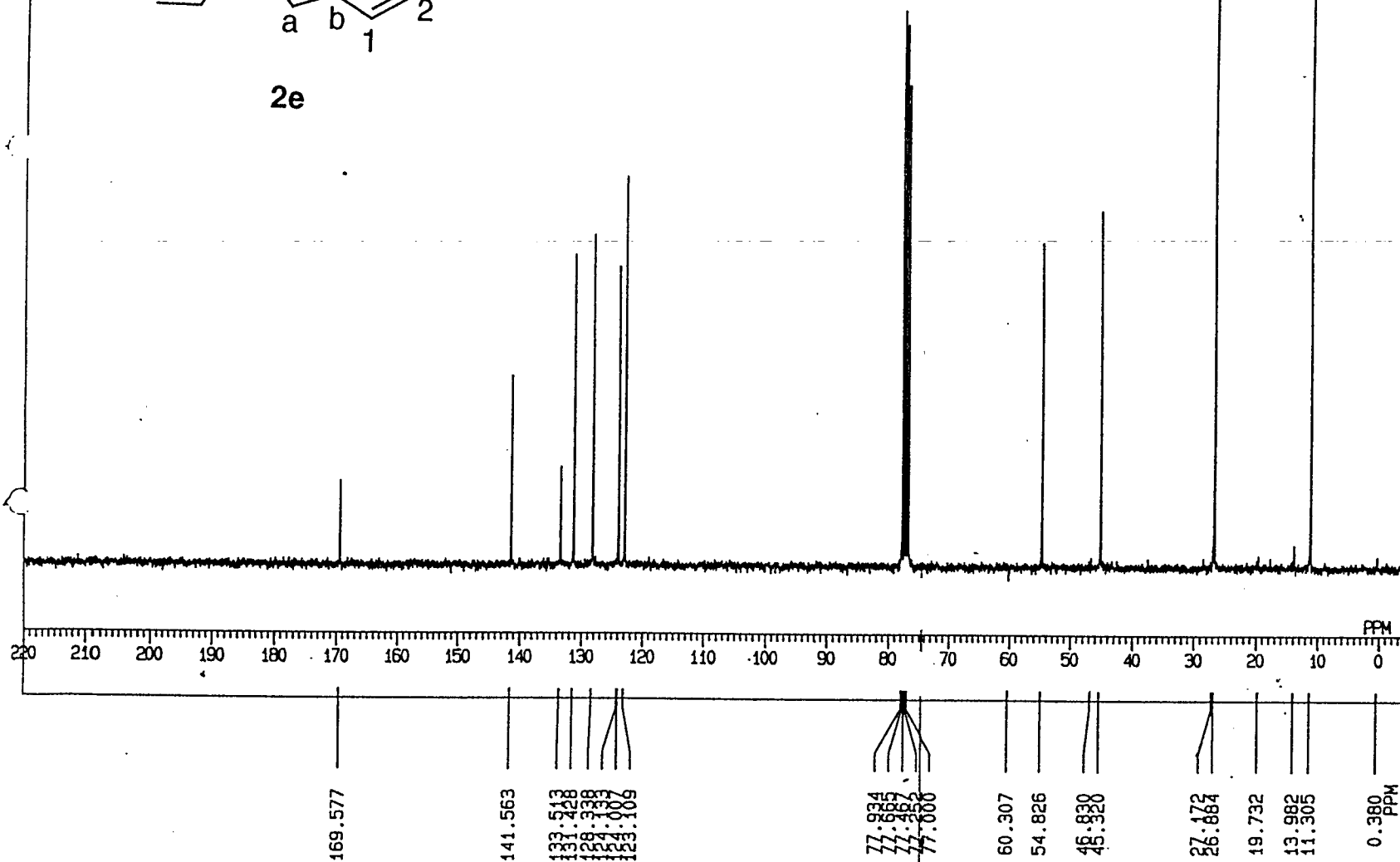


0.000 PPM

KM-L-10-12, Fr. 1-2, GPC3



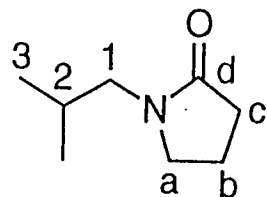
2e



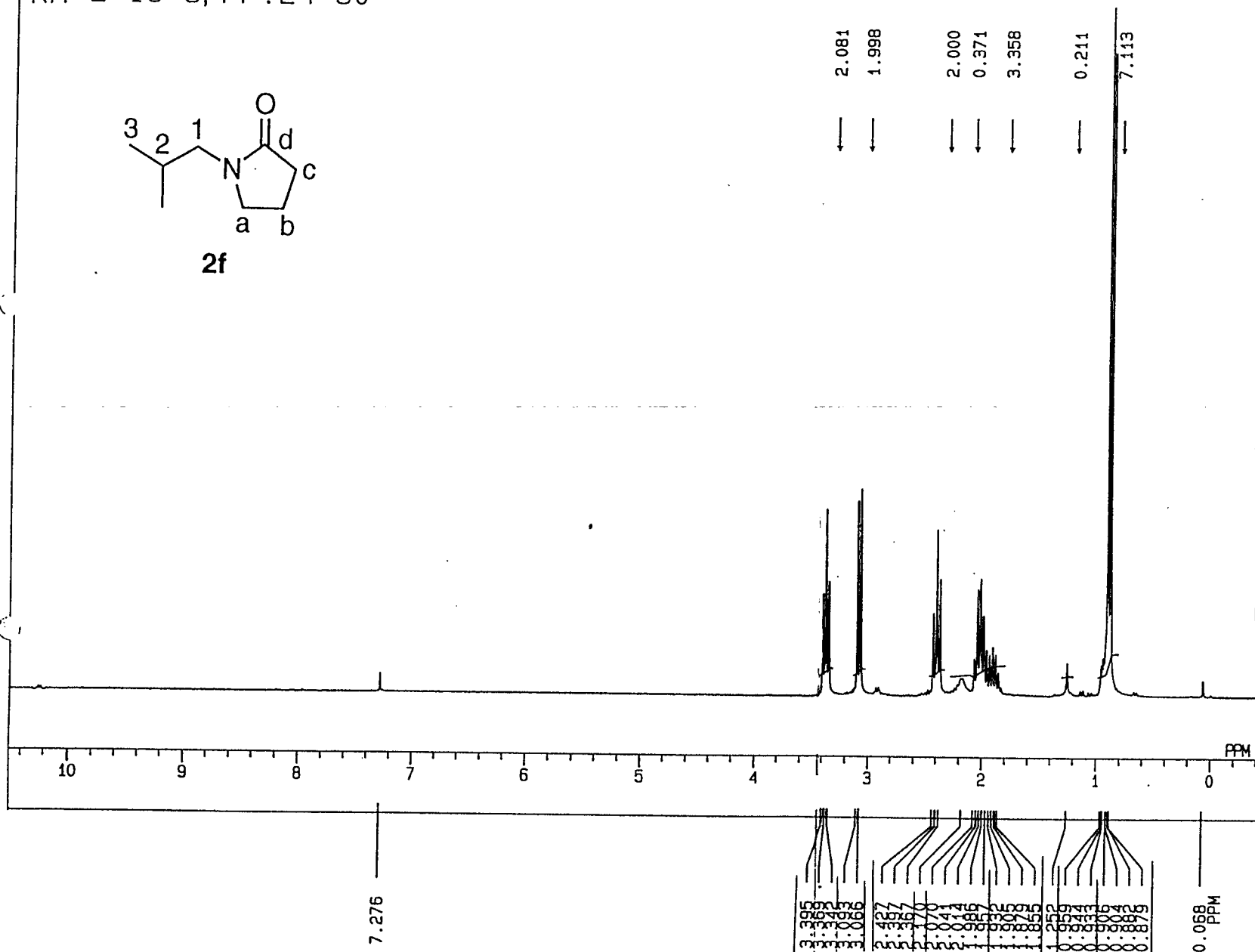
31-OCT-97 03:32:54
DFILE Q13C
OBNUC 13C
EXMOD BCM
OFR 67.80 MHz
OBSET 135.00 kHz
OBFIN 5200.0 Hz
POINT 32768
FREQU 20000.0 Hz
SCANS 1184
ACQTM 0.819 sec
PD 2.181 sec
PW1 4.0 us
IRNUC 1H
CTEMP 23.9 c
SLVNT CDCL3
EXREF 77.00 ppm
BF 1.50 Hz
RGAIN 32
OPERATOR :

S22

KM-L-16-5, Fr. 24-30



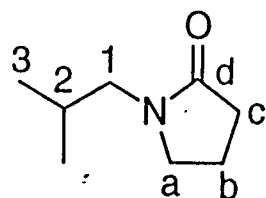
2f



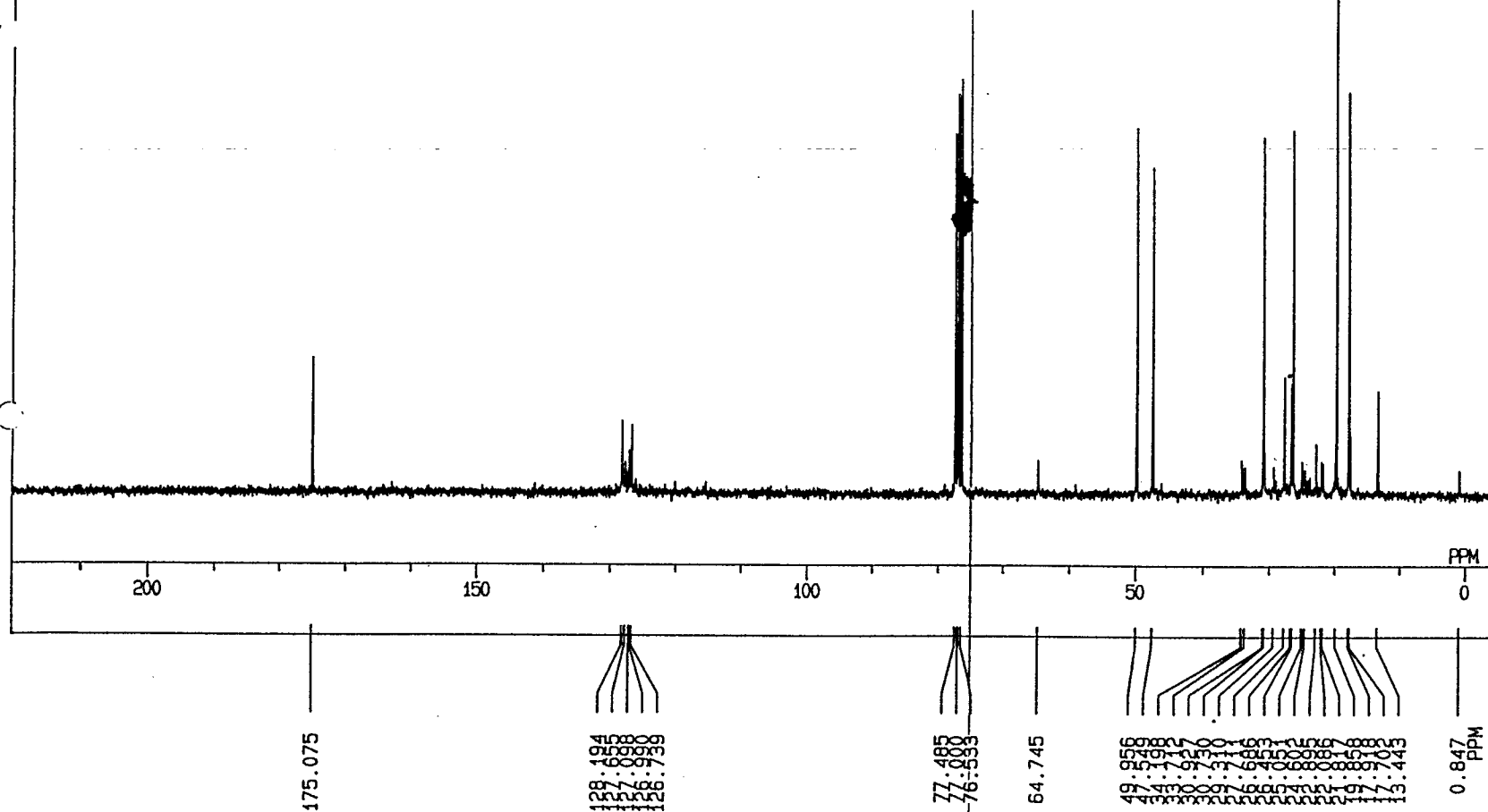
30-JUN-97 15:32:55
DFILE SAVING
OBNUC 1H
EXMOD NON
QFR 270.05 MHz
OBSET 112.00 kHz
OBFIN 5800.0 Hz
POINT 32768
FREQU 5405.4 Hz
SCANS 16
ACQTM 3.031 sec
PD 3.969 sec
PW1 4.7 us
IRNUC 1H
CTEMP 25.6 c
SLVNT CDCL3
EXREF 0.00 ppm
BF 0.10 Hz
RGAIN 15
OPERATOR : _____

S23

KM-L-16-6, Fr. 2



2f



04-JUL-97 02:19:59
 DFILE SAVING
 OBNUC 13C
 EXMOD BCM
 OFR 67.80 MHz
 OBSET 135.00 kHz
 OBFIN 5200.0 Hz
 POINT 32768
 FREQU 20000.0 Hz
 SCANS 936
 ACQTM 0.819 sec
 PD 4.000 sec
 PW1 4.0 us
 IRNUC 1H
 CTEMP 0.0 c
 SLVNT CDCL3
 EXREF 77.00 ppm
 BF 1.22 Hz
 RGAIN 18
 OPERATOR : _____

S24

KM-L-17-5, FR. 4

CC(C)CN1CC(=O)CC1

2g

7.289

1.000
1.963
1.129
2.121
1.448
0.867
2.920
6.233

0.000 PPM

M-3

POOH QUALITY 1 UNIT=1000000

KM-L-17-5, FR.3-14

10-JUL-97 06:07:32

DFILE Q13C

OBNUC 13C

EXMOD BCM

QFR 67.80 MHz

OBSET 135.00 kHz

OBFIN 5200.0 Hz

POINT 32768

FREQU 20000.0 Hz

SCANS 164

ACQTM 0.819 sec

PD 2.181 sec

PW1 4.0 us

IRNUC 1H

CTEMP 25.5 c

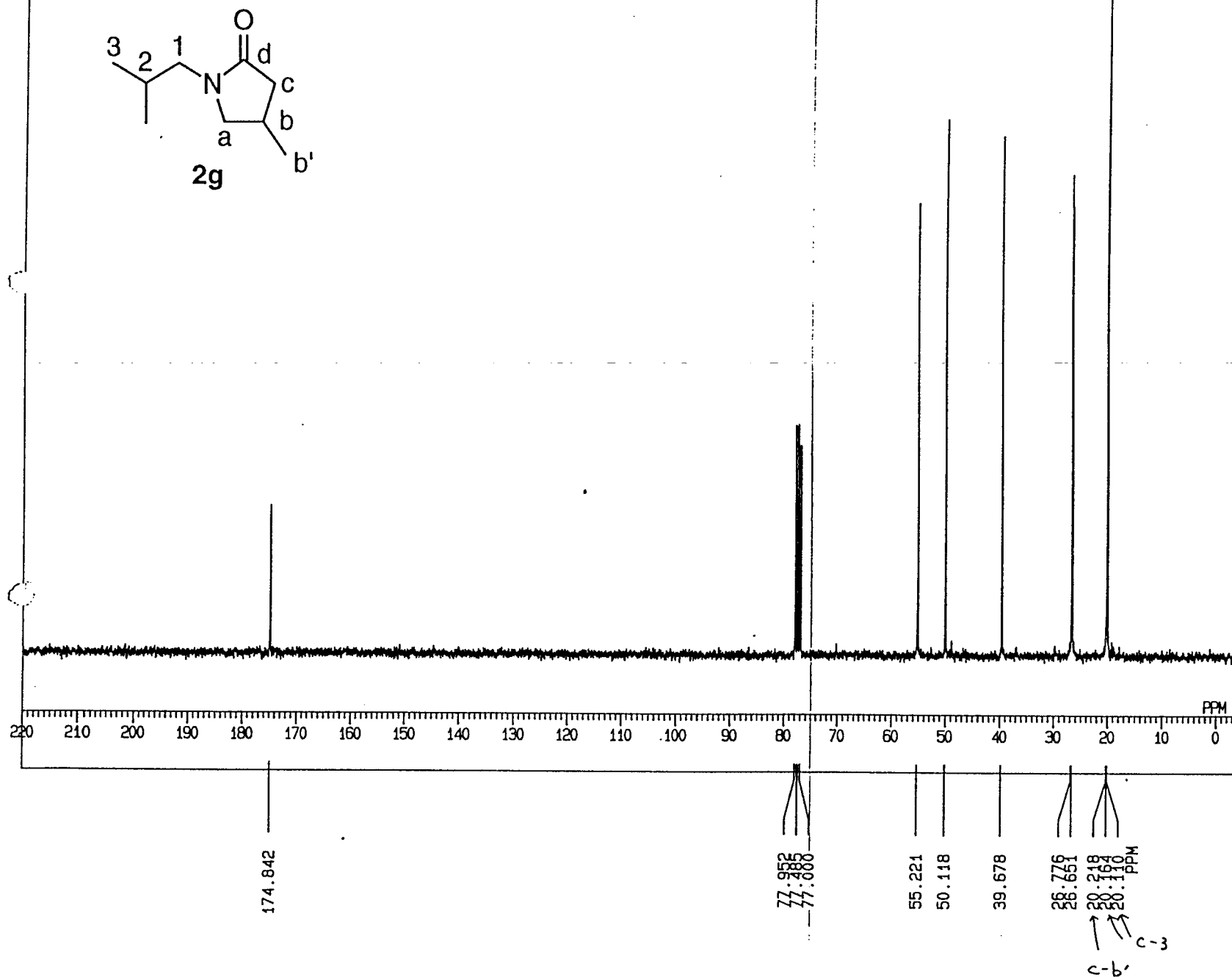
SLVNT CDCL3

EXREF 77.00 ppm

BF 1.50 Hz

RGAIN 32

OPERATOR :

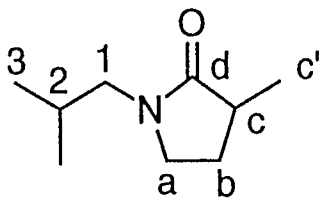


S26

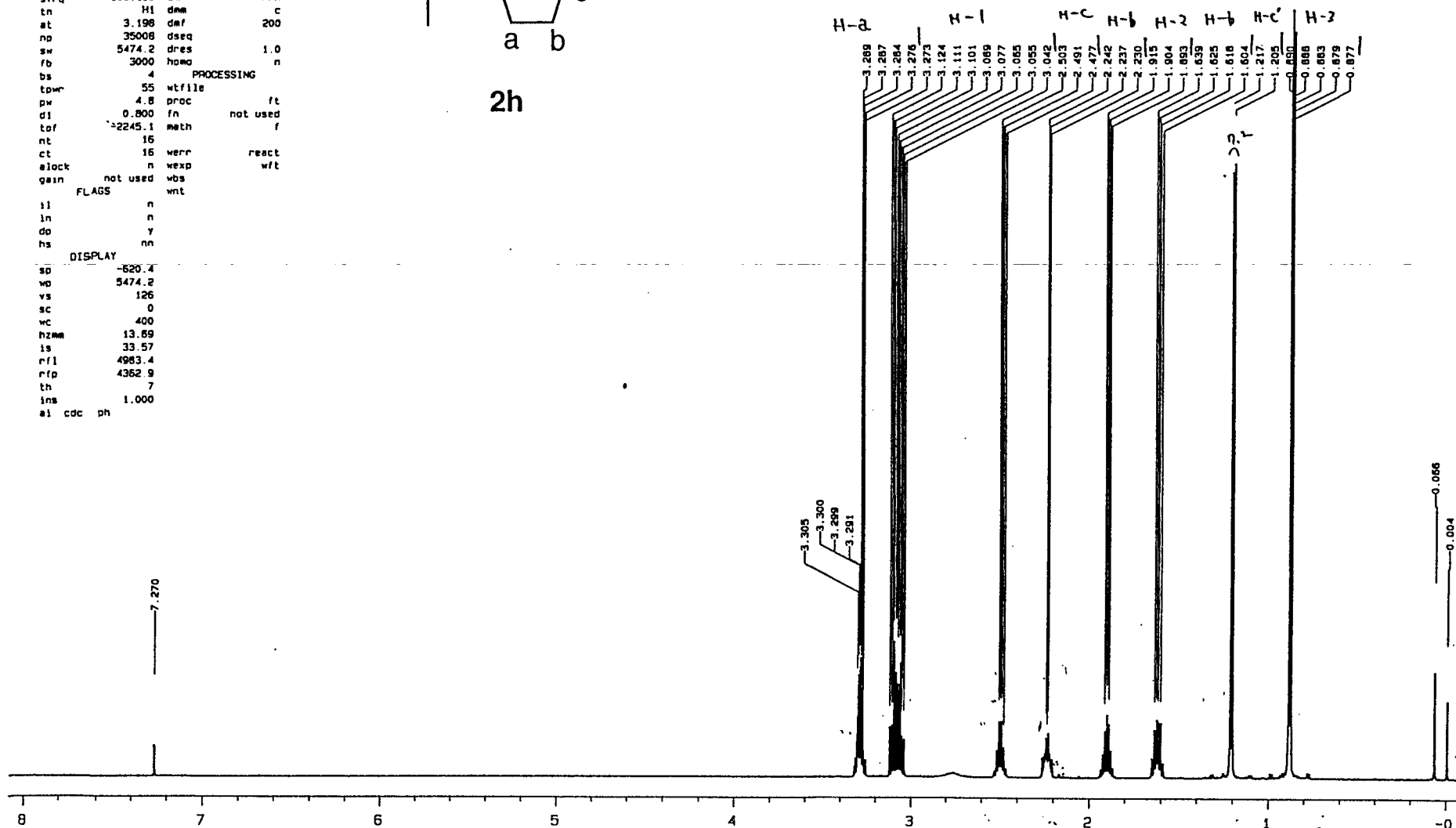
KM-L-31-4, Fr. 11

exp3 s2pu1

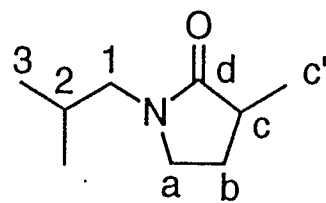
SAMPLE		DEC. & VT	
date	Dec 20 97	dfrq	500.133
solvent	CDCl3	dn	H1
file	exp	dpr	18
ACQUISITION		dof	0
sfrq	500.130	dm	nnn
tn	H1	dsm	c
at	3.198	daf	200
np	35008	dseq	
sw	5474.2	dres	1.0
fb	3000	homo	n
bs		PROCESSING	
tpwr	55	wtfile	
pw	4.8	proc	ft
d1	0.800	fn	not used
tof	2245.1	meth	f
nt	16		
ct	16	werr	react
elock	n	wexp	wft
gain	not used	wbs	
FLAGS		wnt	
il	n		
in	n		
dp	y		
hs	nn		
DISPLAY			
sp	-520.4		
wd	5474.2		
vs	126		
sc	0		
wc	400		
hzmm	13.59		
is	33.57		
rfl	4983.4		
rfo	4352.9		
th	7		
ins	1.000		
al	cdc ph		



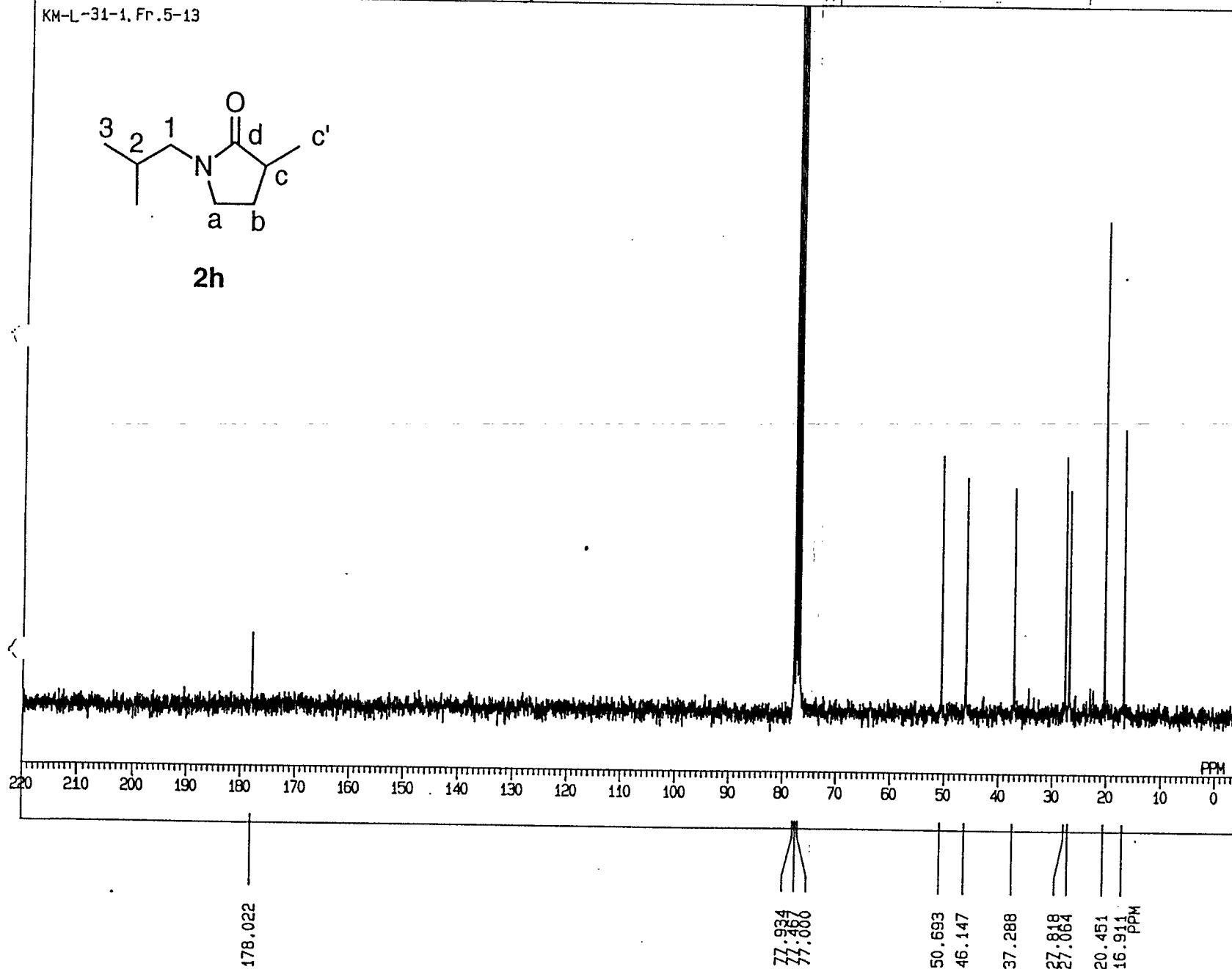
2h



KM-L-31-1, Fr. 5-13

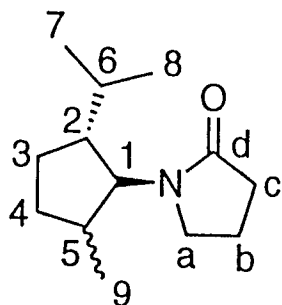


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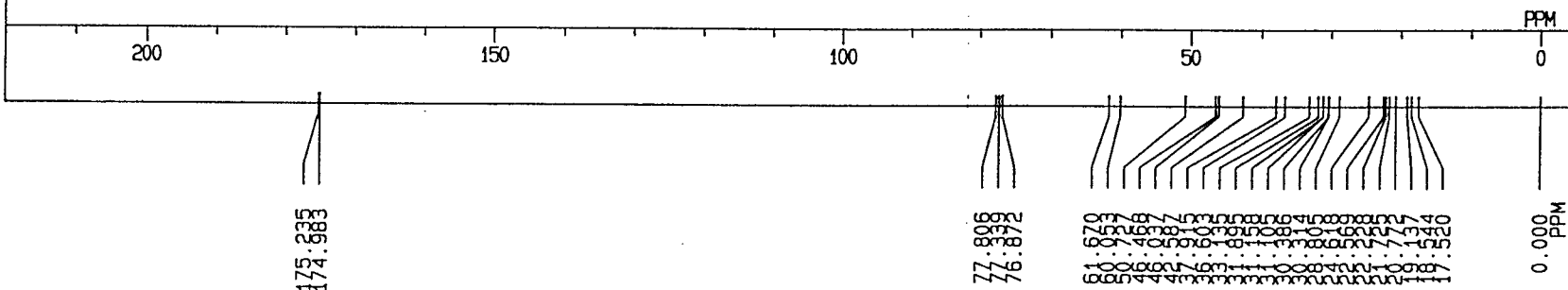
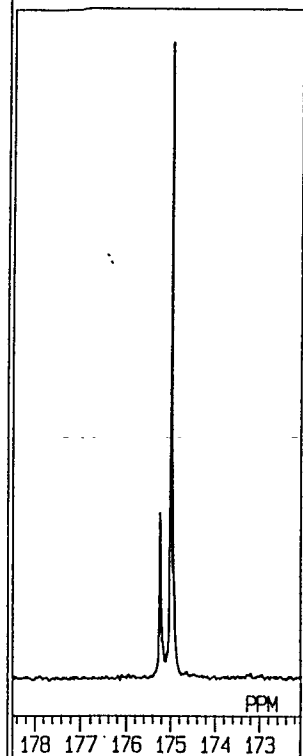


02-SEP-97 12:56:50
DFILE Q13C
OBNUC 13C
EXMOD BCM
QFR 67.80 MHz
OBSET 135.00 kHz
OBFIN 5200.0 Hz
POINT 32768
FREQU 20000.0 Hz
SCANS 1348
ACQTM 0.819 sec
PD 2.181 sec
PW1 4.0 us
IRNUC 1H
CTEMP 26.4 c
SLVNT CDCL3
EXREF 77.00 ppm
BF 1.50 Hz
RGAIN 32
OPERATOR : _____

KM-L-4-1

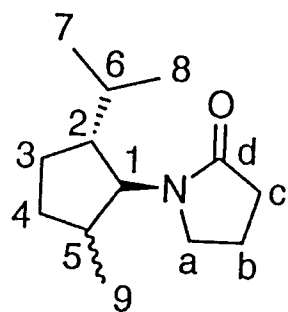


2i



07-FEB-96 23: 12: 35
 OFILE SAVING
 OBNUC 13C
 EXMOD BCM
 OFR 67.80 MHz
 OBSET 135.00 kHz
 OBFIN 5200.0 Hz
 POINT 32768
 FREQU 20000.0 Hz
 SCANS 7330
 ACQTM 0.819 sec
 PD 4.000 sec
 PW1 4.0 us
 IRNUC 1H
 CTEMP 0.0 c
 SLVNT CDCL3
 EXREF 0.00 ppm
 BF 1.22 Hz
 RGAIN 18
 OPERATOR : _____

KM-L-4-2



2i

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0.012
0.217

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1.000

↓ ↓

9.216

↓

95.434

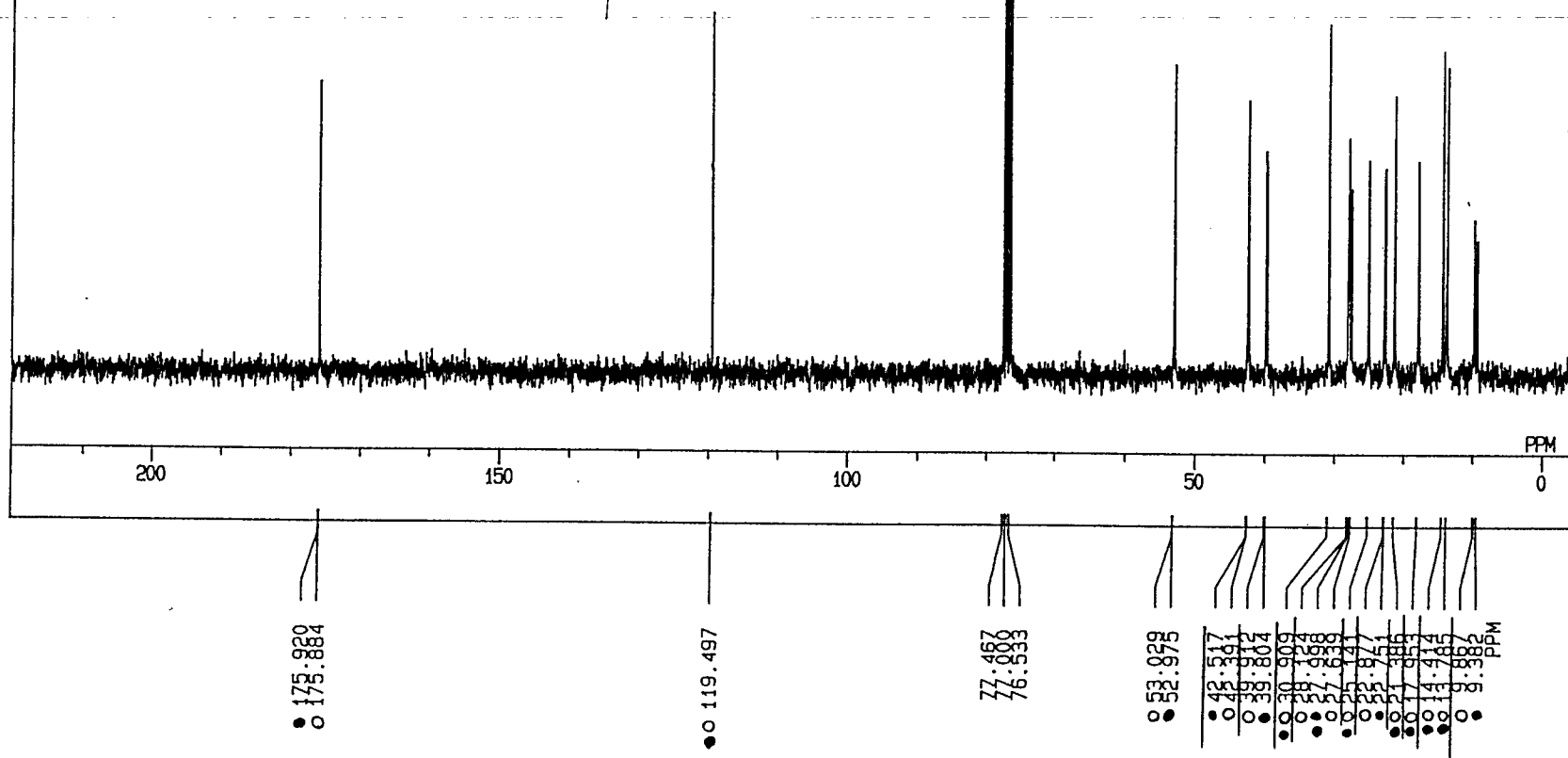
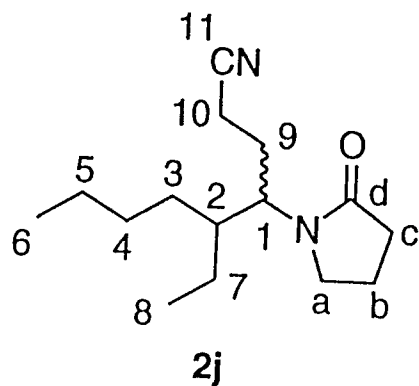
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10 9 8 7 6 5 4 3 2 1 0 PPM

08-MAR-96 12:56:01
DFILE Q1H
COMNT KM-L-4-2
EXMOD SGNON
OBNUC 1H
OBFIN 10492.9 Hz
POINT 32768
FREQU 12019.2 Hz
SCANS 16
ACQTH 1.363 sec
PD 3.637 sec
PW1 7.7 us
IRFIN 10300.0 Hz
IRATN 15
IRRPW 60 us
TEMP. 27.0 c
SLVNT C606
EXREF 0.00 ppm
BF 0.37 Hz
RGAIN 6
XE 4249.5640 Hz
XS -81.3550 Hz

S30

KM-L-35-3, Fr. EtOAc 100%

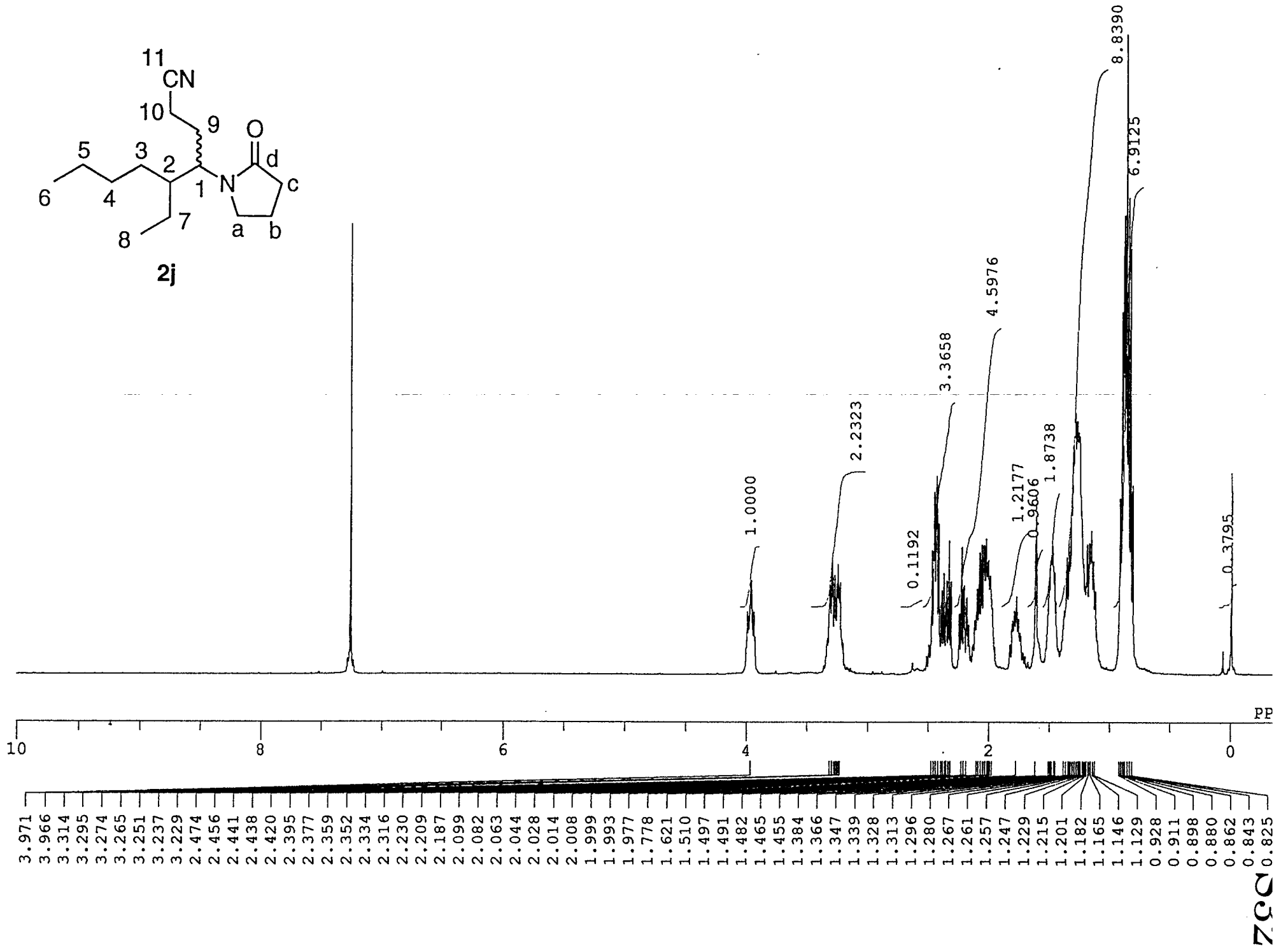


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03-FEB-98 08:24:01
DFILE SAVING
OBNUC 13C
EXMOD BCM
OFR                67.80 MHz
OBSET              135.00 kHz
OBFIN              5200.0 Hz
POINT              32768
FREQU              20000.0 Hz
SCANS              161
ACQTM              0.819 sec
PD                 4.000 sec
PW1                4.0 us
IRNUC 1H
CTEMP              0.0 c
SLVNT CDCL3
EXREF              77.00 ppm
BF                 1.22 Hz
GAIN 18
OPERATOR :

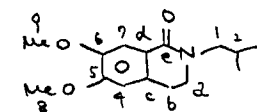
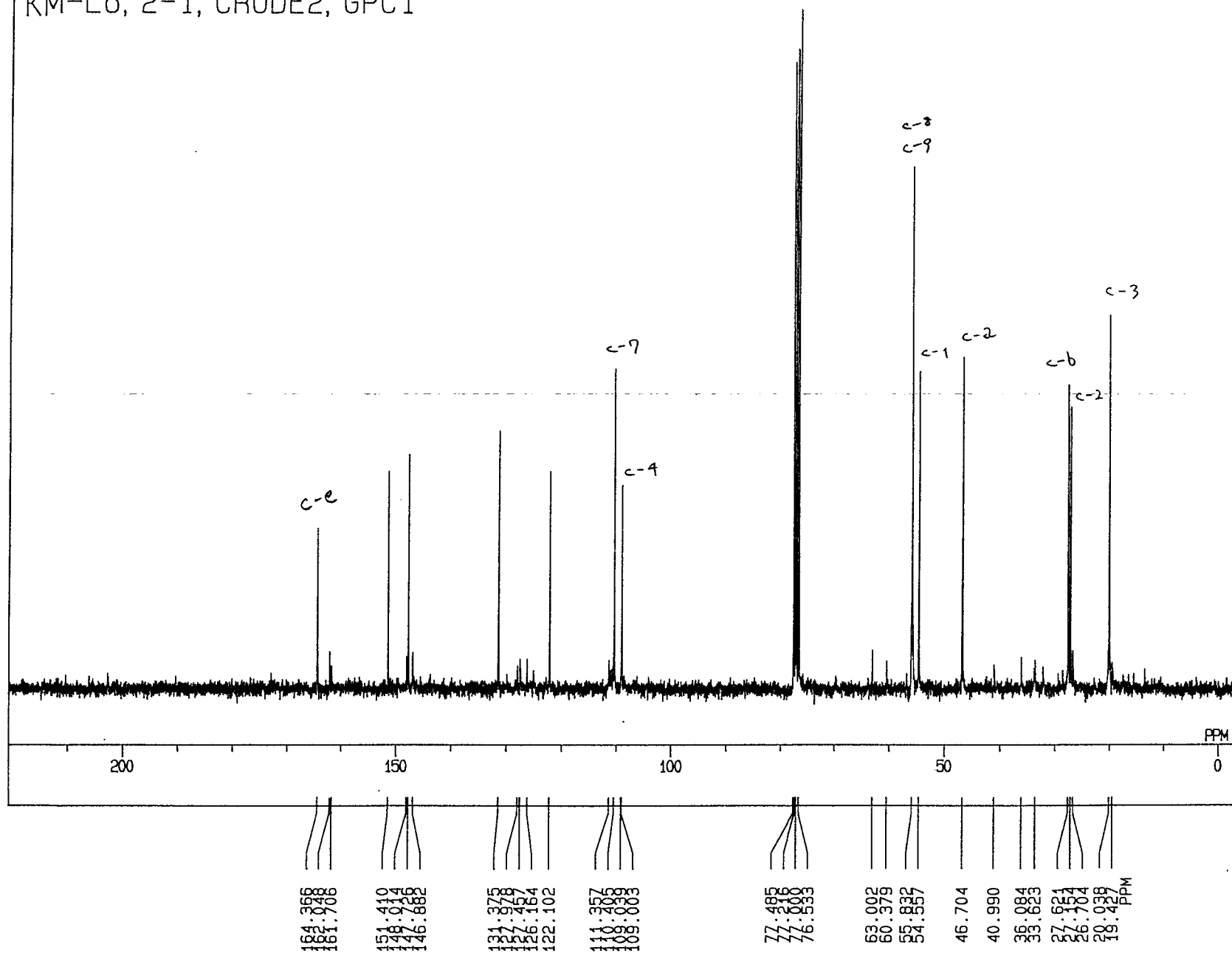
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132



KM-L6, 2-1, CRUDE2, GPC1

13-FEB-98 01:08:38
 OFILE SAVING
 OBNUC 13C
 EXMOD BCM
 OFR 67.80 MHz
 OBSET 135.00 kHz
 OBFIN 5200.0 Hz
 POINT 32768
 FREQU 20000.0 Hz
 SCANS 358
 ACQTM 0.819 sec
 PD 4.000 sec
 PW1 4.0 us
 IRNUC 1H
 CTEMP 24.0 c
 SLVNT CDCL3
 EXREF 77.00 ppm
 BF 1.22 Hz
 RGAIN 32
 OPERATOR : _____



S33