Synthesis of Carbocyclic and Heterocyclic Fused-Quinolines by Cascade Radical Annulations of Unsaturated *N*-Aryl Thiocarbamates, Thioamides and Thioureas

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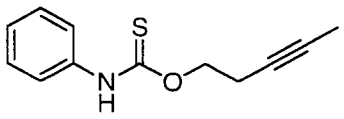
Supporting Information

General Procedure A: Synthesis of Thiocarbamates:

Homopropargyl alcohol (5 mmol) and phenyl isothiocyanate (745 mg, 5.5 mmol) were dissolved in anhydrous THF (10 mL), and this mixture was cooled to 0 °C. Then NaH (220 mg, 60% suspension on mineral oil, 5.5 mmol) was slowly added. The reaction mixture was stirred at room temperature overnight, then poured into water and extracted with EtOAc. The organic layer was washed with brine, dried and evaporated to give the crude thiocarbamate, which was purified by column chromatography (5% EtOAc in hexane) to give the desired thiocarbamate as solids or sticky oils in 80-90% yield.

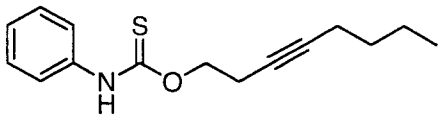
Phenylthiocarbamic acid *O*-pent-3-ynyl ester (8a).



Using general procedure A, the title compound was prepared in 92% yield as white solid. IR: 3197, 3116, 3036, 1594, 1549, 1497, 1450, 1408, 1353, 1280, 1220, 1201, 1083, 1051, 753, 691; ^{1}H NMR (300 MHz, $CDCl_3$) δ 1.81 (t, 3H J = 2.5 Hz), 2.64 (m, 2H), 4.64 (br, 2H), 7.19-

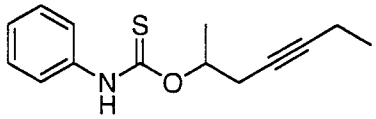
7.35 (br, 5H); ^{13}C NMR ($CDCl_3$, 75 MHz) δ 3.4, 19.0, 70.5, 74.6, 77.6, 121.4, 123.4, 128.9, 137.0, 187.9; HRMS *m/z* calcd for $C_{12}H_{13}NOS$ 219.0718, found 219.0715.

Phenylthiocarbamic acid *O*-oct-3-ynyl ester (8b).



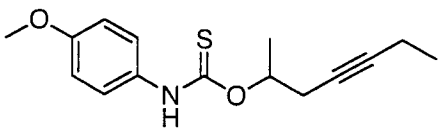
Using the general procedure A, the title compound was prepared in 84% yield. IR: 3232, 3036, 2958, 2932, 2871, 1597, 1542, 1497, 1447, 1403, 1338, 1221, 1186, 1163, 1047, 904, 750, 692; ^{1}H NMR (300 MHz, $CDCl_3$) δ 0.90 (t, 3H *J* = 7.1 Hz), 1.34-1.52 (m, 4H), 2.17 (m, 2H), 2.66 (m, 2H), 4.64 (br, 2H), 7.18-7.35 (br m, 5H), 8.27 (br, 1H); ^{13}C NMR ($CDCl_3$, 75 MHz) δ 13.4, 18.3, 19.1, 21.8, 30.9, 70.6, 75.3, 82.3, 121.4, 125.2, 128.9, 137.0, 188.0; HRMS *m/z* calcd for $C_{15}H_{19}NOS$ 261.1187, found 261.1177.

Phenylthiocarbamic acid *O*-(1-methylhex-3-ynyl) ester (8c).



Using the general procedure A, the title compound was prepared in 76% yield. IR: 3226, 3036, 2977, 2935, 1597, 1542, 1499, 1447, 1383, 1328, 1293, 1216, 1188, 1126, 1056, 981, 904, 752, 691; ^{1}H NMR (300 MHz, $CDCl_3$) δ 1.13 (t, 3H *J* = 7.5 Hz), 1.49 (d, 3H, *J* = 6.3 Hz), 2.19 (qt, 2H, *J* = 7.5, 2.4 Hz), 2.61 (br m, 2H), 5.63 (sextet, *J* = 5.8 Hz), 7.17-7.34 (br m, 5H) 8.28 (br,s, 1H); ^{13}C NMR ($CDCl_3$, 75 MHz) δ 12.3, 14.0, 18.7, 25.5, 74.2, 77.9, 84.3, 121.4, 125.0, 128.8, 137.1, 187.3; HRMS *m/z* calcd for $C_{14}H_{17}NOS$ 247.1031, found 247.1027.

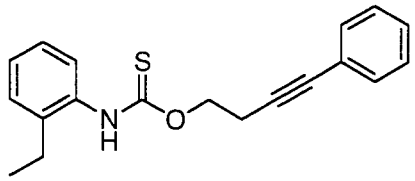
(4-Methoxyphenyl)-thiocarbamic acid *O*-(1-methylhex-3-ynyl) ester (8d).



Using the general procedure 6, the title compound was prepared in 76% yield.

IR: 3218, 2975, 2934, 2836, 1596, 1515, 1382, 1298, 1250, 1179, 1125, 1058, 1036, 983, 829; ^{1}H NMR (300 MHz, $CDCl_3$) δ 1.12 (t, 3H J = 7.5 Hz), 1.45 (d, 3H, J = 6.3 Hz), 2.18 (qt, 2H, J = 7.5, 2.3 Hz), 2.59 (m, 2H), 3.80 (s, 3H), 5.61 (sextet, J = 6.0 Hz), 6.85 (d, 2H, J = 8.8 Hz), 7.26 (d, 2H, J = 8.8 Hz), 8.89 (br s, 1H); ^{13}C NMR ($CDCl_3$, 75 MHz) δ 12.3, 14.1, 18.7, 25.5, 55.3, 74.3, 77.6, 84.3, 114.0, 123.3, 130.2, 156.9, 187.0; HRMS *m/z* calcd for $C_{15}H_{19}NO_2S$ 277.1137, found 277.1125.

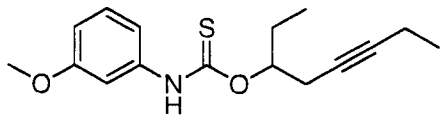
(2-Ethylphenyl)thiocarbamic acid *O*-(4-phenylbut-3-ynyl) ester (8e).



Using the general procedure A, the title compound was prepared in 85% yield.

IR: 3199, 2967, 1638, 1599, 1583, 1509, 1492, 1449, 1398, 1341, 1292, 1226, 1156, 1069, 1048, 758, 693; ^{1}H NMR (300 MHz, $CDCl_3$) δ 1.22 (t, 3H J = 7.6 Hz), 2.64 (q, 2H, J = 7.6 Hz), 4.72 (t, 2H, J = 6.6 Hz), 7.04-7.40 (br m, 9H); ^{13}C NMR ($CDCl_3$, 75 MHz) δ 14.2, 19.8, 24.4, 69.7, 82.1, 85.3, 123.2, 126.1, 126.5, 127.2, 127.9, 128.2, 128.8, 131.6, 134.4, 137.6, 189.1; HRMS *m/z* calcd for $C_{19}H_{19}NOS$ 309.1187, found 309.1184.

(3-Methoxyphenyl)thiocarbamic acid *O*-(1-ethylhex-3-ynyl) ester (8f).



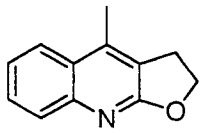
Using the general procedure A, the title compound was prepared in 90% yield.

IR: 3233, 2974, 2938, 1608, 1537, 1495, 1457, 1391, 1318, 1275, 1186, 1046, 1025, 942, 850, 775,754, 685; ^{1}H NMR (300 MHz, $CDCl_3$) δ 0.98 (t, 3H, J = 7.4 Hz), 1.11 (t, 3H J = 7.5 Hz), 1.90 (p, 2H, J = 6.9 Hz), 2.17 (q, 2H, J =7.5 Hz), 2.64 (br m, 2H), 3.81 (s, 3H), 5.51 (p, 1H, J = 5.7 Hz), 6.71 –7.27 (m, 4H), 8.28 (br, 1H); ^{13}C NMR ($CDCl_3$, 75 MHz) δ 9.43, 12.3, 14.0, 21.0, 22.5, 23.1, 25.5, 55.1, 74.1, 82.5, 84.2, 107.4, 110.5, 113.5, 129.5, 138.2, 159.9, 187.6; HRMS *m/z* calcd for $C_{16}H_{21}NO_2S$ 291.1293, found 291.1307.

General Procedure B: Radical Cyclization of Thiocarbamate, Thioamide and Thiourea under Photolytic Conditions.

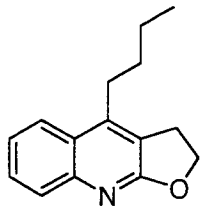
A pyrex test tube was charged with the reaction substrate (0.2 mmol) and AIBN (66 mg, 0.2 mmol) and placed under Ar. Then degassed anhydrous benzene (4 mL) followed by $(TMS)_3SiH$ (280 μL, 0.8 mmol) were added. This solution was irradiated with medium pressure Hg lamp for 20 h. TLC showed the starting substrate disappeared. The reaction mixture was evaporated to dryness and applied to silica gel column. Flash chromatography (5-10% EtOAc in hexane) gave the desired quinoline compounds as a solid in 40-80% yield.

4-Methyl-2,3-dihydrofuro[2,3-*b*]quinoline (9a).



Using the general procedure B, the title compound was prepared in 48% yield.
IR: 3066, 2976, 2916, 1635, 1420, 1400, 1319, 1262, 1230, 1173, 1004, 953, 764; ^{1}H NMR (300 MHz, $CDCl_3$) δ 2.54 (s, 3H), 3.33 (t, 2H, J = 8.2 Hz), 4.70 (t, 2H, J = 8.2 Hz), 7.38 (t, 1H, J = 8.1 Hz), 7.57 (t, J = 8.2 Hz), 7.83 (d, 1H, J = 8.3 Hz); ^{13}C NMR ($CDCl_3$, 75 MHz) δ 15.4, 27.1, 68.8, 119.8, 123.2, 123.7, 125.2, 127.9, 128.8, 140.7, 146.9, 167.0; HRMS *m/z* calcd for $C_{12}H_{11}NO$ 185.0841, found 185.0836.

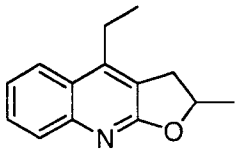
4-Butyl-2,3-dihydrofuro[2,3-*b*]quinoline (9b).



Using the general procedure B, the title compound was prepared in 44% yield.

IR: 3067, 2957, 2930, 2871, 1699, 1630, 1593, 1410, 1401, 1317, 1256, 1230, 1176, 1103, 1044, 1008, 965, 850, 759; ^{1}H NMR (300 MHz, $CDCl_3$) δ 0.98 (t, 3H, J = 7.3 Hz), 1.49 (m, 2H), 1.66 (m, 2H), 2.96 (dd, 2H, J = 7.9, 7.7 Hz), 3.34 (t, 2H, J = 8.2 Hz), 4.71 (t, 2H, 8.1 Hz), 7.38 (ddd, 1H, J = 8.3, 6.9, 1.2 Hz), 7.57 (ddd, 1H, J = 8.2, 7.0, 1.2 Hz), 7.85 (ddd, 2H, J = 8.3, 6.6, 1.2 Hz); ^{13}C NMR ($CDCl_3$, 75 MHz) δ 13.9, 23.0, 27.0, 29.5, 31.6, 68.8, 119.3, 123.2, 123.6, 124.4, 128.1, 128.7, 145.5, 147.3, 167.1; HRMS *m/z* calcd for $C_{15}H_{17}NO$ 227.1310, found 227.1308.

4-Ethyl-2-methyl-2,3-dihydrofuro[2,3-*b*]quinoline (9c).

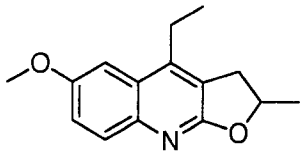


Using the general procedure B, the title compound was prepared in 44% yield.

IR: 3069, 2973, 2935, 2876, 1632, 1591, 1463, 1413, 1397, 1357, 1310, 1260, 1225, 1177, 1088, 1027, 951, 924, 761; ^{1}H NMR (300 MHz, $CDCl_3$) δ 1.30 (t, 3H, J = 7.7 Hz), 1.56 (d, 3H, J = 6.3 Hz), 2.90 (dd, 1H, J = 16.4, 6.5 Hz), 2.97 (q, 2H, J = 7.7 Hz), 3.46 (dd, 1H, J = 16.3, 8.5 Hz), 5.06 (m, 1H), 7.37 (ddd, 1H, J = 8.2, 7.3, 1.4 Hz), 7.56 (ddd, 1H, J = 8.3, 7.0, 1.4 hz), 7.84 (d,

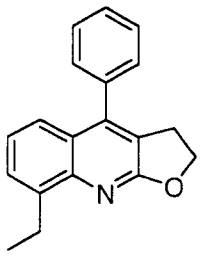
1H, J = 7,8 Hz), 7.87 (d, 1H, J = 8.4 Hz); ^{13}C NMR ($CDCl_3$, 75 MHz) δ 13.7, 22.2, 22.8, 34.2, 77.6, 119.5, 122.8, 123.0, 124.1, 128.1, 128.7, 146.7, 147.3, 166.6; HRMS *m/z* calcd for $C_{14}H_{15}NO$ 213.1154, found 213.1156.

4-Ethyl-6-methoxy-2-methyl-2,3-dihydrofuro[2,3-*b*]quinoline (9d).



Using the general procedure B, the title compound was prepared in 67% yield. IR: 2971, 2936, 1627, 1593, 1522, 1456, 1422, 1360, 1314, 1230, 1175, 1105, 1088, 1028, 936, 830, 795, 723; ^{1}H NMR (300 MHz, $CDCl_3$) δ 1.37 (t, 3H, J = 7.7 Hz), 1.62 (d, 3H, J = 6.3 Hz), 2.95 (dd, 1H, J = 16.4, 6.7 Hz), 3.00 (q, 2H, J = 7.7 Hz), 3.51 (dd, 1H, J = 16.3, 8.5 Hz), 3.99 (s, 3H), 5.06 (m, 1H), 7.27-7.38 (m, 2H), 7.84 (d, 1H, J = 9.1 Hz); ^{13}C NMR ($CDCl_3$, 75 MHz) δ 13.3, 22.2, 23.0, 34.4, 55.5, 77.3, 103.5, 119.0, 119.7, 124.7, 129.3, 142.6, 145.4, 155.9, 165.4; HRMS *m/z* calcd for $C_{15}H_{17}NO_2$ 243.1259, found 243.1258.

8-Ethyl-4-phenyl-2,3-dihydrofuro[2,3-*b*]quinoline (9e).



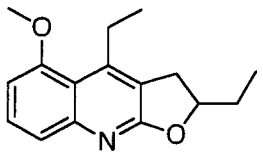
Using the general procedure B, the title compound was prepared in 88 % yield.

IR: 3059, 2966, 2929, 1628, 1596, 1523, 1444, 1406, 1323, 1222, 1127, 1004, 847, 762, 704; ^{1}H NMR (300 MHz, $CDCl_3$) δ 1.46 (t, 3H, J = 7.4 Hz), 3.30 (t, 2H, J = 8.2 Hz), 3.32 (q, 2H, J = 7.5 Hz), 4.76 (t, 2H, J = 8.2 Hz), 7.25-7.64 (m, 8H); ^{13}C NMR ($CDCl_3$, 75 MHz) δ 15.1, 23.4, 27.6, 68.8, 119.1, 121.4, 123.47, 123.54, 124.5, 127.8, 128.2, 128.6, 128.9, 136.5, 141.2, 145.6, 145.7, 166.2; HRMS *m/z* calcd for $C_{19}H_{17}NO$ 275.1310, found 275.1306.

Compounds 9f and 9g.

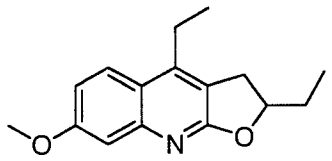
Using the general procedure 6B, the two title compounds were prepared in 1.3:1 ratio and 67% combined yield. The two title compounds were separated by careful flash chromatography on silica gel.

2,4-diethyl-5-methoxy-2,3-dihydrofuro[2,3-*b*]quinoline (9f).



IR: 2964, 2936, 1624, 1610, 1593, 1464, 1408, 1381, 1308, 1264, 1228, 1172, 1138, 1075, 1038, 983, 936, 812, 761, 712; ^{1}H NMR (300 MHz, $CDCl_3$) δ 1.09 (t, 3H, J = 7.4 Hz), 1.26 (t, 3H, J = 7.4 Hz), 1.97-1.74 (m, 2H), 2.92 (dd, 1H, J = 16.3, 6.7 Hz), 3.14 (q, 2H, J = 7.4 Hz), 3.40 (dd, 1H, J = 16.3, 8.7 Hz), 3.94 (s, 3H), 4.82 (m, 1H), 6.73 (dd, 1H, J = 5.5, 3.5 Hz), 7.428 (d, 1H, J = 5.5 Hz), 7.431 (d, 1H, J = 3.5 Hz); ^{13}C NMR ($CDCl_3$, 75 MHz) δ 9.4, 14.3, 27.0, 29.5, 32.5, 55.4, 82.2, 103.9, 116.2, 119.8, 121.1, 128.3, 148.0, 149.9, 157.3, 166.6; HRMS *m/z* calcd for $C_{16}H_{19}NO_2$ 257.1416, found 257.1411.

2,4-Diethyl-7-methoxy-2,3-dihydrofuro[2,3-*b*]quinoline (9f').

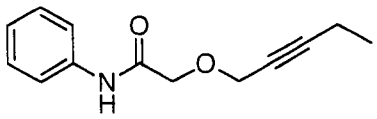


IR: 2967, 2935, 1619, 1597, 1518, 1468, 1437, 1406, 1272, 1226, 1159, 1032, 924; ^{1}H NMR (300 MHz, $CDCl_3$) δ 1.09 (t, 3H, J = 7.4 Hz), 1.28 (t, 3H, J = 7.4 Hz), 1.906-1.74 (m, 2H), 2.91 (dd, 1H, J = 16.1, 6.7 Hz), 2.92 (q, 2H, J = 7.4 Hz), 3.37 (dd, 1H, J = 16.1, 8.7 Hz), 3.91 (s, 3H), 4.84 (m, 1H), 7.00 (dd, 1H, J = 9.2, 2.7 Hz), 7.23 (d, 1H, J = 2.6 Hz), 7.74 (d, 1H, J = 9.1 Hz); ^{13}C NMR ($CDCl_3$, 75 MHz) δ 9.4, 13.8, 22.9, 29.4, 32.1, 55.3, 82.5, 107.7, 115.1, 116.8, 118.7, 124.1, 146.5, 149.4, 160.2, 167.4; HRMS m/z calcd for $C_{16}H_{19}NO_2$ 257.1416, found 257.1424.

General Procedure C: Synthesis of Aryl Amides.

Carboxylic acid (2 mmol) and aniline (2 mmol) were dissolved in anhydrous MeCN (5 mL). Then EDCI (593 mg, 3 mmol) was added. The reaction mixture was stirred at room temperature for 24 h, then it was diluted with EtOAc and washed with 5% HCl, saturated $NaHCO_3$ and brine. The organic layer was dried and evaporated to give a crude product. Flash chromatography gave the desired amide in 80- 90% yield.

2-Pent-2-ynyloxy-*N*-phenyl acetamide.

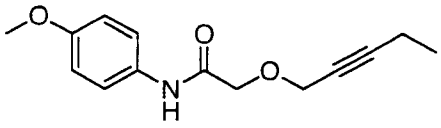


Using general procedure C, the title compound was prepared in 93% yield.

IR: 3388, 3312, 2979, 2940, 2918, 2853, 1684, 1603, 1534, 1501, 1446, 1319, 1248, 1140, 1101, 1026, 972, 757, 696; ^{1}H NMR (300 MHz, $CDCl_3$) δ 1.14 (t, 3H, J = 7.5 Hz), 2.23 (qt, 2H, J = 7.5, 2.1 Hz), 4.16 (s, 2H), 4.30 (t, 2H, J = 2.1 Hz), 7.13 (t, 1H, J = 7.3 Hz), 7.35 (t, 2H, J = 7.8

Hz), 7.58 (d, 2H, J = 8.3 Hz); ^{13}C NMR ($CDCl_3$, 75 MHz) δ 12.3, 13.6, 59.4, 68.9, 73.6, 90.2, 119.7, 124.4, 129.0, 137.1, 167.4; HRMS m/z calcd for $C_{13}H_{15}NO_2$ 217.1103, found 217.1109.

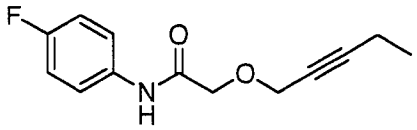
N-(4-Methoxyphenyl)-2-pent-2-ynyloxy acetamide.



Using general procedure C, the title compound was prepared in 94% yield.

IR: 3313, 2936, 2855, 1688, 1601, 1514, 1456, 1416, 1306, 1248, 1184, 1149, 1102, 1033, 975, 836, 783; ^{1}H NMR (300 MHz, $CDCl_3$) δ 1.15 (t, 3H, J = 7.5 Hz), 2.24 (qt, 2H, J = 7.5, 2.2 Hz), 3.81 (s, 3H), 4.15 (s, 2H), 4.28 (t, 2H, J = 2.1 Hz), 6.89 (d, 2H, J = 9.0 Hz), 7.49 (d, 2H, J = 9.0 Hz), 8.14 (br s, 1H); ^{13}C NMR ($CDCl_3$, 75 MHz) δ 12.2, 13.5, 55.3, 59.2, 68.8, 73.6, 90.0, 113.9, 121.4, 130.1, 156.3, 167.1; HRMS m/z calcd for $C_{14}H_{17}NO_3$ 247.1208, found 247.1214.

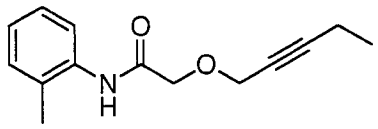
N-(4-Fluorophenyl)-2-pent-2-ynyloxy acetamide.



Using general procedure C, the title compound was prepared in 84% yield.

IR: 3391, 3302, 2986, 1687, 1533, 1511, 1411, 1212, 1099, 835, 798; ^{1}H NMR (300 MHz, $CDCl_3$) δ 1.14 (t, 3H, J = 7.6 Hz), 2.24 (qt, 2H, J = 7.5, 2.0 Hz), 4.16 (s, 2H), 4.30 (t, 2H, J = 2.1 Hz), 7.04 (t, 2H, J = 8.7 Hz), 7.55 (dd, 2H, J = 9.1, 4.8 Hz), 8.23 (br s, 1H); ^{13}C NMR ($CDCl_3$, 75 MHz) δ 12.2, 13.4, 59.2, 68.6, 73.5, 90.1, 115.5 (d, J = 89.1 Hz), 121.4 (d, J = 30.6 Hz), 133.1, 157.6, 160.8, 167.3; HRMS m/z calcd for $C_{13}H_{14}NO_2F$ 235.1009, found 235.1014.

2-Pent-2-ynyloxy-*N*-*o*-tolyl acetamide.

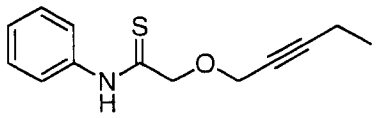


Using general procedure C, the title compound was prepared in 84% yield. IR: 3398, 2978, 2916, 1697, 1593, 1531, 1457, 1309, 1253, 1139, 1099, 752.99; ^{1}H NMR (300 MHz, $CDCl_3$) δ 1.14 (t, 3H, J = 7.6 Hz), 2.24 (qt, 2H, J = 7.5, 2.1 Hz), 2.30 (s, 3H), 4.20 (s, 2H), 4.32 (t, 2H, J = 2.1 Hz), 7.08 (dd, 1H, J = 8.4, &.2 Hz), 7.19-7.26 (m, 2H), 8.00 (d, 1H, J = 7.7 Hz), 8.24 (br s, 1H); ^{13}C NMR ($CDCl_3$, 75 MHz) δ 12.2, 13.4, 17.3, 59.3, 68.9, 73.6, 89.9, 121.8, 124.7, 126.6, 128.1, 130.2, 134.9, 167.2; HRMS *m/z* calcd for $C_{14}H_{17}NO_2$ 231.1259, found 231.1258.

General Procedure D: Synthesis of Thioamide 10.

The corresponding amide (1 mmol) was dissolved in toluene (10 mL). Then Lawesson's reagent (0.55 mmol) was added. The reaction mixture was heated at 80 °C for 4 h. TLC showed the starting amide disappeared. Toluene was removed under reduced pressure and the residue was purified by flash chromatography to give the desired thioamide.

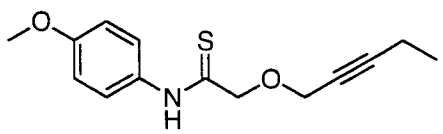
2-Pent-2-ynyloxy-*N*-phenyl thioacetamide (10c).



Using general procedure D, the title compound was prepared in 82% yield.

IR: 3307, 2976, 2917, 2855, 1687, 1598, 1531, 1445, 1398, 1319, 1142, 1071, 753, 690; ^{1}H NMR (300 MHz, $CDCl_3$) δ 1.13 (t, 3H, J = 7.5 Hz), 2.22 (q, 2H, J = 7.5 Hz), 3.81 (s, 3H), 4.32 (s, 2H), 4.52 (s, 2H), 7.28 (t, 1H, J = 7.4 Hz), 7.42 (t, 2H, J = 7.7 Hz), 7.86 (d, 2H, J = 7.6 Hz), 9.83 (br s, 1H); ^{13}C NMR ($CDCl_3$, 75 MHz) δ 12.3, 13.5, 59.2, 73.6, 76.1, 90.3, 122.8, 126.8, 128.8, 137.6, 195.9; HRMS *m/z* calcd for $C_{13}H_{15}NOS$ 233.0874, found 233.0876.

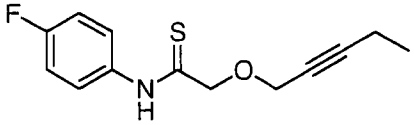
_N_-(4-Methoxyphenyl)-2-pent-2-ynyloxy thioacetamide (10d).



Using general procedure D, the title compound was prepared in 80% yield.

IR: 3306, 2982, 2941, 2858, 1713, 1619, 1595, 1513, 1454, 1401, 1253, 1182, 1147, 1076, 1041, 834, 799, 752; ^{1}H NMR (300 MHz, $CDCl_3$) δ 1.13 (t, 3H, J = 7.5 Hz), 2.22 (qt, 2H, J = 7.5, 2.1 Hz), 3.81 (s, 3H), 4.3 (t, 2H, J = 2.1 Hz), 4.51 (s, 2H), 6.93 (d, 2H, J = 9.0 Hz), 7.72 (d, 2H, J = 9.0 Hz), 9.73 (br s, 1H); ^{13}C NMR ($CDCl_3$, 75 MHz) δ 12.3, 13.5, 55.4, 59.2, 73.7, 76.0, 90.2, 113.9, 124.6, 130.6, 158.0, 195.4; HRMS *m/z* calcd for $C_{14}H_{17}NO_2S$ 263.0980, found 263.0989.

_N_-(4-Fluorophenyl)-2-pent-2-ynyloxy acetamide (10e).

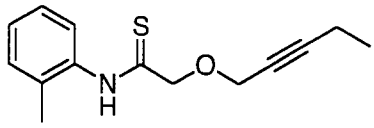


Using general procedure D, the title compound was prepared in 97% yield.

IR: 3310, 2979, 2939, 2858, 1686, 1609, 1508, 1400, 1320, 1231, 1143, 1073, 1025, 887, 837, 813, 750; ^{1}H NMR (300 MHz, $CDCl_3$) δ 1.13 (t, 3H, J = 7.5 Hz), 2.23 (qt, 2H, J = 7.5, 2.0 Hz),

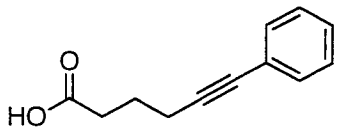
4.33 (t, 2H, J = 1.9 Hz), 4.53 (s, 2H), 7.12 (t, 2H, J = 8.6 Hz), 7.81 (dd, 2H, J = 8.9, 4.1 Hz), 9.78 (br s, 1H); ^{13}C NMR ($CDCl_3$, 75 MHz) δ 12.2, 13.4, 59.1, 73.5, 75.9, 90.2, 115.5 (d, J = 90.8 Hz), 124.9 (d, J = 32.4 Hz), 133.5, 158.9, 162.2, 196.2; HRMS m/z calcd for $C_{13}H_{14}NOFS$ 251.0780, found 251.0782.

2-Pent-2-ynyloxy-*N*-*o*-tolyl thioacetamide (10f).



Using general procedure D, the title compound was prepared in 88% yield. IR: 3316, 2978, 2921, 1695, 1588, 1509, 1459, 1400, 1319, 1258, 1143, 1068, 889, 739; ^{1}H NMR (300 MHz, $CDCl_3$) δ 1.15 (t, 3H, J = 7.5 Hz), 2.26 (qt, 2H, J = 7.4, 2.0 Hz), 2.31 (s, 3H), 4.34 (t, 2H, J = 1.9 Hz), 4.59 (s, 2H), 7.27-7.30 (m, 3H), 7.84 (d, 1H, J = 7.5 Hz), 9.69 (br s, 1H); ^{13}C NMR ($CDCl_3$, 75 MHz) δ 12.2, 13.4, 17.4, 59.0, 73.6, 75.7, 89.9, 125.2, 126.3, 127.5, 130.6, 132.5, 135.8, 197.1; HRMS m/z calcd for $C_{14}H_{17}NOS$ 247.1031, found 247.1024.

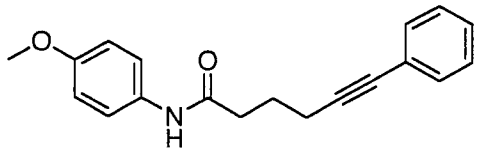
6-Phenyl-hex-5-ynoic acid.



Iodobenzene (0.56 mL, 5 mmol) was dissolved in toluene (12 mL) and $Pd(PhCN)_2Cl_2$ (95 mg, 0.25 mmol), Ph_3P (132 mg, 0.5 mmol) and CuI (144 mg, 0.75 mmol) were introduced successively. To this suspension was then added diisopropylamine (2.1 mL, 30 mmol) and hex-5-ynoic acid (0.54 mL, 5 mmol). The reaction mixture was stirred at room temperature

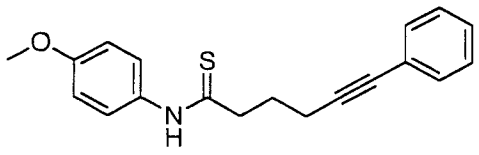
overnight, and then diluted with EtOAc, washed with 0.5 N HCl and brine. The organic layer was dried and evaporated to give a crude residue, which was further purified by flash chromatography to give 510 mg of the title compound as colorless oil in 54% yield. IR: 3068 (br), 2943, 2669, 1710, 1597, 1491, 1433, 1320, 1282, 1247, 1206, 1158, 916, 756, 692; ^{1}H NMR (300 MHz, $CDCl_3$) δ 1.96 (quint, 2H, J = 7.1 Hz), 2.53 (t, 2H, J = 6.9 Hz), 2.58 (t, 2H, J = 7.4 Hz), 7.27-7.44 (m, 5H); ^{13}C NMR ($CDCl_3$, 75 MHz) δ 18.7, 23.5, 32.8, 81.6, 88.5, 123.6, 128.2, 131.5, 179.8; HRMS *m/z* calcd for $C_{12}H_{12}O_2$ 188.0837, found 188.0844.

6-Phenylhex-5-ynoic acid (4-methoxyphenyl) amide.



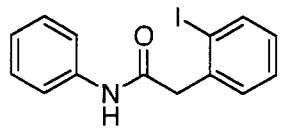
Using the general procedure C, the title compound was prepared in 91% yield. IR: 3238, 3125, 3058, 2959, 2836, 1649, 1603, 1539, 1513, 1455, 1413, 1302, 1244, 1183, 1150, 1035, 832, 757, 692; ^{1}H NMR (300 MHz, $CDCl_3$) δ 2.04 (quint, 2H, J = 7.0 Hz), 2.55 (t, 4H, J = 6.7 Hz), 3.80 (s, 3H), 6.86 (d, 2H, J = 8.9 Hz), 7.11-7.51 (m, 7H); ^{13}C NMR ($CDCl_3$, 75 MHz) δ 18.8, 24.3, 36.0, 55.4, 81.6, 89.0, 114.0, 121.8, 123.6, 127.7, 128.2, 130.9, 131.5, 156.3, 170.6; HRMS *m/z* calcd for $C_{19}H_{19}NO_2$ 293.1416, found 293.1425.

6-Phenyl-hex-5-ynethioic acid (4-methoxyphenyl) amide (10a).



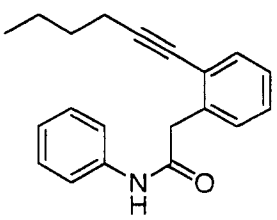
Using the general procedure D, the title compound was prepared in 91% yield. IR: 3194, 3004, 2933, 2836, 1611, 1511, 1442, 1398, 1300, 1251, 1178, 1129, 1033, 831, 758, 693; ^{1}H NMR (300 MHz, $CDCl_3$) δ 2.2 (quint, 2H, J = 7.0 Hz), [2.1 (quint, J = 8.0 Hz)], 2.59 (t, 2H, J = 6.7 Hz), [2.46 (t, J = 6.7 Hz)], 3.00 (t, 2H, J = 7.2 Hz), [2.76 (t, J = 7.5 Hz)], 3.82 (s, 2H), [3.73 (s)], 6.93 (1H, d, J = 8.9 Hz), [6.84, (d, J = 8.8 Hz)], 7.11-7.56 (m, 7H), 8.80 (br, s, 1H), [8.28 (br s)]; ^{13}C NMR ($CDCl_3$, 75 MHz) δ 18.2, 27.9, 46.3, 55.3, 81.8, 88.9, 113.8, 114.5, 123.3, 125.6, 127.0, 127.7, 128.2, 131.4, 158.0; HRMS m/z calcd for $C_{19}H_{19}NOS$ 309.1187, found 309.1196.

2-(2-Iodophenyl)-*N*-phenyl acetamide.



Using the general procedure C, the title compound was prepared in 93% yield. IR: 3280, 3057, 2930, 1662, 1599, 1540, 1499, 1444, 1346, 1311, 1252, 1015, 752; ^{1}H NMR (300 MHz, $CDCl_3$) δ 3.80 (s, 2H), 7.02 (td, 1H, J = 7.8, 2.1 Hz), 7.11 (t, 1H, J = 7.4 Hz), 7.27-7.48 (m, 6H), 7.90 (dd, 1H, J = 8.0, 0.8 Hz); ^{13}C NMR ($CDCl_3$, 75 MHz) δ 49.4, 101.1, 120.1, 124.5, 128.9, 129.0, 129.3, 131.0, 137.5, 138.0, 139.9, 167.7; HRMS m/z calcd for $C_{14}H_{12}NOI$ 336.9964, found 336.9962.

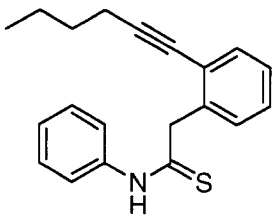
2-(2-Hex-1-ynyl-phenyl)-*N*-phenyl acetamide.



Using procedure similar to 6-phenyl-hex-5-ynoic acid, the title compound was prepared in 71% yield.

IR: 3300, 3062, 2956, 2931, 2872, 1663, 1601, 1544, 1500, 1444, 1351, 1310, 1252, 1175, 756, 691; ^{1}H NMR (300 MHz, $CDCl_3$) δ 0.92 (t, 3H, J =7.2 Hz), 1.48 (m, 2H), 1.60 (m, 2H), 2.49 (t, 2H, J = 7.0 Hz), 3.91 (s, 2H), 7.08 (t, 1H, J = 7.4 Hz), 7.24-7.75 (m, 8H), 7.60 (br s, 1H); ^{13}C NMR ($CDCl_3$, 75 MHz) δ 13.5, 19.2, 22.0, 30.7, 43.8, 78.9, 95.9, 119.7, 123.8, 124.1, 127.5, 128.4, 128.8, 129.9, 132.6, 136.3, 137.9, 168.7; HRMS *m/z* calcd for $C_{20}H_{21}NO$ 291.1623, found 291.1629.

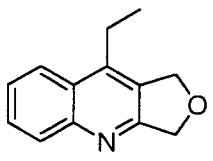
2-(2-Hex-1-ynyl-phenyl)-*N*-phenyl thioacetamide (10b).



Using the general procedure D, the title compound was prepared in 72% yield.

IR: 3326, 3062, 2957, 2931, 2871, 1674, 1598, 1535, 1496, 1447, 1394, 1316, 1121, 756, 716, 690; ^{1}H NMR (300 MHz, $CDCl_3$) δ 0.93 (t, 3H, J =7.2 Hz), 1.48 (m, 2H), 1.63 (m, 2H), 2.51 (t, 2H, J = 7.0 Hz), 4.43 (s, 2H), 7.21-7.39 (m, 5H), 7.48 (d, 1H, J = 7.1 Hz), 7.51 (d, 1H, J = 6.0 Hz), 7.62 (d, 2H, J = 8.0 Hz), 9.10 (brs, 1H); ^{13}C NMR ($CDCl_3$, 75 MHz) δ 13.6, 19.3, 22.1, 30.7, 53.8, 79.3, 96.1, 123.3, 123.5, 126.7, 127.9, 128.6, 128.8, 130.1, 132.7, 137.1, 138.8, 201.0; HRMS *m/z* calcd for $C_{20}H_{21}NS$ 307.1395, found 307.1426.

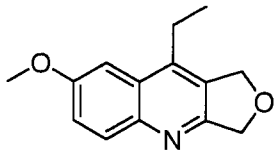
9-Ehtyl-1,3-dihydrofuro[3,4-*b*]quinoline (11c).



Using general procedure B, the title compound was prepared in 67% yield.

IR: 3068, 2972, 2937, 2874, 1766, 1621, 1585, 1512, 1458, 1351, 1183, 1056, 907, 766; ^{1}H NMR (300 MHz, $CDCl_3$) δ 1.32 (t, 3H, J = 7.7 Hz), 2.99 (q, 2H, J = 7.7 Hz), 5.19 (s, 2H), 5.31 (s, 2H), 7.56 (t, 1H, J = 8.1 Hz), 7.70 (t, 1H, J = 8.1 Hz), 8.06 (t, 2H, J = 8.0 Hz); ^{13}C NMR ($CDCl_3$, 75 MHz) δ 14.0, 22.9, 71.2, 73.0, 123.2, 126.0, 126.1, 128.4, 128.9, 129.7, 142.7, 148.6, 162.1; HRMS *m/z* calcd for $C_{13}H_{13}NO$ 199.0997, found 199.1002.

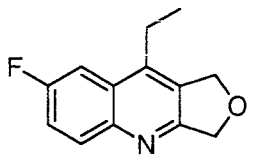
9-Ehtyl-7-methoxy-1,3-dihydrofuro[3,4-*b*]quinoline (11d).



Using general procedure B, the title compound was prepared in 50% yield.

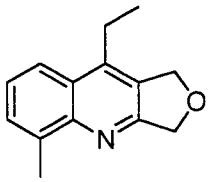
IR: 2970, 2941, 2883, 2854, 1630, 1513, 1473, 1357, 1229, 1078, 1055, 1032, 910, 835; ^{1}H NMR (300 MHz, $CDCl_3$) δ 1.32 (t, 3H, J = 7.7 Hz), 2.95 (q, 2H, J = 7.7 Hz), 3.96 (s, 3H), 5.18 (s, 2H), 5.29 (s, 2H), 7.28 (d, 1H, J = 3.0 Hz), 7.37 (dd, 1H, J = 9.2, 2.7 Hz), 8.01 (d, 1H, J = 9.2 Hz); ^{13}C NMR ($CDCl_3$, 75 MHz) δ 13.5, 23.1, 31.6, 55.5, 71.3, 72.8, 102.0, 120.9, 127.0, 128.9, 130.5, 141.9, 143.8, 157.7, 159.2; HRMS *m/z* calcd for $C_{14}H_{15}NO_2$ 229.1103, found 229.1101.

9-Ehtyl-7-fluoro-1,3-dihydrofuro[3,4-*b*]quinoline (11e).



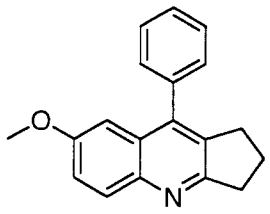
Using general procedure B, the title compound was prepared in 53% yield. IR: 2973, 2938, 2876, 1633, 1517, 1457, 1353, 1216, 1182, 1069, 1056, 970, 907, 833, 799, 729; ^{1}H NMR (300 MHz, $CDCl_3$) δ 1.31 (t, 3H, J = 7.7 Hz), 2.93 (q, 2H, J = 7.7 Hz), 5.17 (s, 2H), 5.30 (s, 2H), 7.46 (ts, 1H, J = 10.5, 2.5 Hz), 7.63 (dd, 1H, J = 10.2, 2.7 Hz), 8.05 (dd, 1H, J = 9.2, 5.6 Hz); ^{13}C NMR ($CDCl_3$, 75 MHz) δ 13.7, 23.1, 71.2, 72.8, 77.2, 107.0 (d, J = 100.2 Hz), 118.9 (d, J = 101.7 Hz), 126.9 (d, J = 36.3 Hz), 129.3, 131.8 (d, J = 36.3 Hz), 142.2 (d, J = 21.6 Hz), 145.6, 158.9, 161.6, 162.1; HRMS *m/z* calcd for $C_{13}H_{12}NOF$ 217.0903, found 217.0902.

9-Ehtyl-5-methyl-1,3-dihydrofuro[3,4-*b*]quinoline (11f).



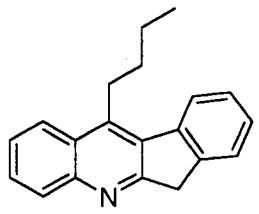
Using general procedure B, the title compound was prepared in 62% yield. IR: 2969, 2935, 2878, 2855, 1629, 1589, 1514, 1474, 1457, 1354, 1073, 1056, 970, 764; ^{1}H NMR (300 MHz, $CDCl_3$) δ 1.31 (t, 3H, J = 7.7 Hz), 2.80 (s, 3H), 2.98 (q, 2H, J = 7.6 Hz), 5.22 (s, 2H), 5.31 (s, 2H), 7.44 (dd, 1H, J = 8.1, 7.2 Hz), 7.54 (d, 1H, J = 6.9 Hz), 7.90 (d, 1H, J = 8.3 Hz); ^{13}C NMR ($CDCl_3$, 75 MHz) δ 14.1, 18.7, 23.1, 71.3, 73.3, 121.2, 125.6, 125.8, 127.9, 129.1, 137.4, 142.7, 147.8, 160.9; HRMS *m/z* calcd for $C_{14}H_{15}NO$ 213.1154, found 213.1164.

7-Methoxy-9-phenyl-2,3-dihydro-1*H*-cyclopenta[*b*]quinoline (11a).



Using the general procedure B, the title compound was prepared in 87% yield. IR: 3271, 2987, 2940, 1620, 1511, 1463, 1383, 1366, 1246, 1229, 1201, 1177, 1032, 849; ^{1}H NMR (300 MHz, $CDCl_3$) δ 1.46 (qint, 2H, J = 7.5 Hz), 2.96 (t, 2H, J = 7.4 Hz), 3.28 (t, 2H, J = 7.6 Hz), 3.81 (s, 3H), 7.01 (d, 1H, J = 2.7 Hz), 7.37 (dd, J = 8.5, 2.7 Hz), 7.44-7.63 (m, 5H), 8.05 (d, 1H, J = 9.2 Hz); ^{13}C NMR ($CDCl_3$, 75 MHz) δ 23.5, 30.4, 34.8, 55.3, 104.3, 119.8, 127.0, 128.2, 128.5, 129.1, 130.1, 133.9, 136.9, 141.6, 143.8, 157.1, 164.9; HRMS *m/z* calcd for $C_{19}H_{17}NO$ 275.1310, found 275.1315.

11-Butyl-6*H*-indeno[2,1-*b*]quinoline (11b).

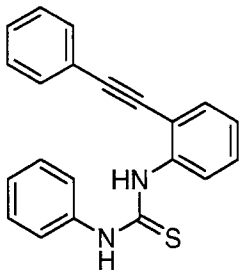


Using the general procedure B, the title compound was prepared in 52% yield. IR: 3068, 2957, 2929, 2873, 1607, 1574, 1508, 1466, 1390, 1332, 1033, 950, 841, 762, 727; ^{1}H NMR (300 MHz, $CDCl_3$) δ 1.07 (t, 3H, J =7.2 Hz), 1.68 (m, 2H), 1.83 (m, 2H), 3.50 (m, 2H), 4.18 (s, 2H), 7.40-7.73 (m, 5H), 7.99 (d, 1H, J = 7.7 Hz), 8.12 (d, 1H, J = 8.3 Hz), 8.19 (d, 1H, J = 8.4 Hz); ^{13}C NMR ($CDCl_3$, 75 MHz) δ 14.0, 23.4, 27.9, 31.3, 38.9, 123.6, 123.9, 125.5, 125.8, 126.9, 127.4, 127.6, 128.5, 129.4, 130.5, 139.7, 141.9, 142.2, 146.9, 165.5; HRMS *m/z* calcd for $C_{20}H_{19}N$ 273.1518, found 273.1523.

General Procedure E: Synthesis of Aryl Thioureas.

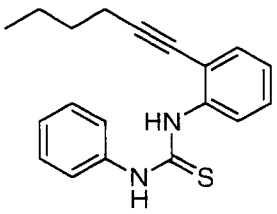
The corresponding aniline (0.34 mmol) was mixed with phenyl isothiocyanate (3 equiv) and the mixture was allowed to stand overnight. The reaction mixture gradually solidified to give a wax-like material. TLC showed disappearance of starting aniline. Purification by chromatography gave the desired thiourea in high yield.

1-Phenyl-3-(2-phenylethynyl-phenyl) thiourea (12b).



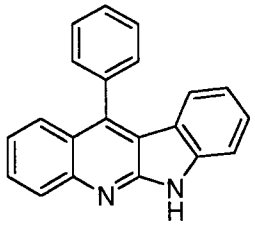
Using the general procedure E, the title compound was prepared in 94% yield. IR: 3326, 3168, 1580, 1539, 1495, 1446, 1356, 1257, 1181, 757, 691; ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.14-7.42 (m, 12H), 7.51 (dd, 1H, J = 7.7, 1.2 Hz), 8.43 (d, 1H, J = 8.9 Hz), 8.46 (br s, 1H), 8.50 (br s, 1H); ^{13}C NMR ($CDCl_3$, 75 MHz) δ 13.5, 19.0, 22.0, 30.5, 75.5, 98.2, 116.5, 122.5, 124.8, 125.4, 127.3, 128.0, 129.9, 132.0, 136.4, 138.9, 178.4; HRMS *m/z* calcd for $C_{21}H_{16}N_2S$ 328.1034, found 328.1036.

1-(2-Hex-1-ynyl-phenyl)-3-phenyl thioureas (12c).



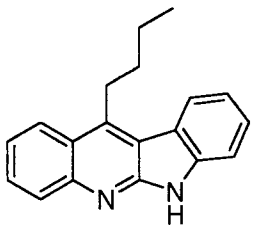
Using the general procedure E, the title compound was prepared in 87% yield. IR: 3304, 3175, 2955, 2931, 2871, 1580, 1539, 1497, 1445, 1354, 1314, 1245, 1175, 751, 694; ^{1}H NMR (300 MHz, $CDCl_3$) δ 0.90 (t, 3H, J = 7.0 Hz), 1.36 (m, 4H), 2.15 (t, 2H, J = 6.9 Hz), 7.09 (td, 1H, J = 7.6, 1.1 Hz), 7.29-7.49 (m, 7H), 7.97 (br, s, 1H), 8.34 (br m, 1H), 8.46 (d, 1H, J = 8.2 Hz); ^{13}C NMR ($CDCl_3$, 75 MHz) δ 13.5, 19.0, 22.0, 30.5, 75.5, 98.2, 116.5, 122.5, 124.8, 125.4, 127.3, 128.0, 129.9, 132.0, 136.4, 138.9, 178.4; HRMS *m/z* calcd for $C_{19}H_{20}N_2S$ 308.1347, found 308.1344.

11-Phenyl-6-*H*-indolo[2,3-*b*]quinoline (13b).

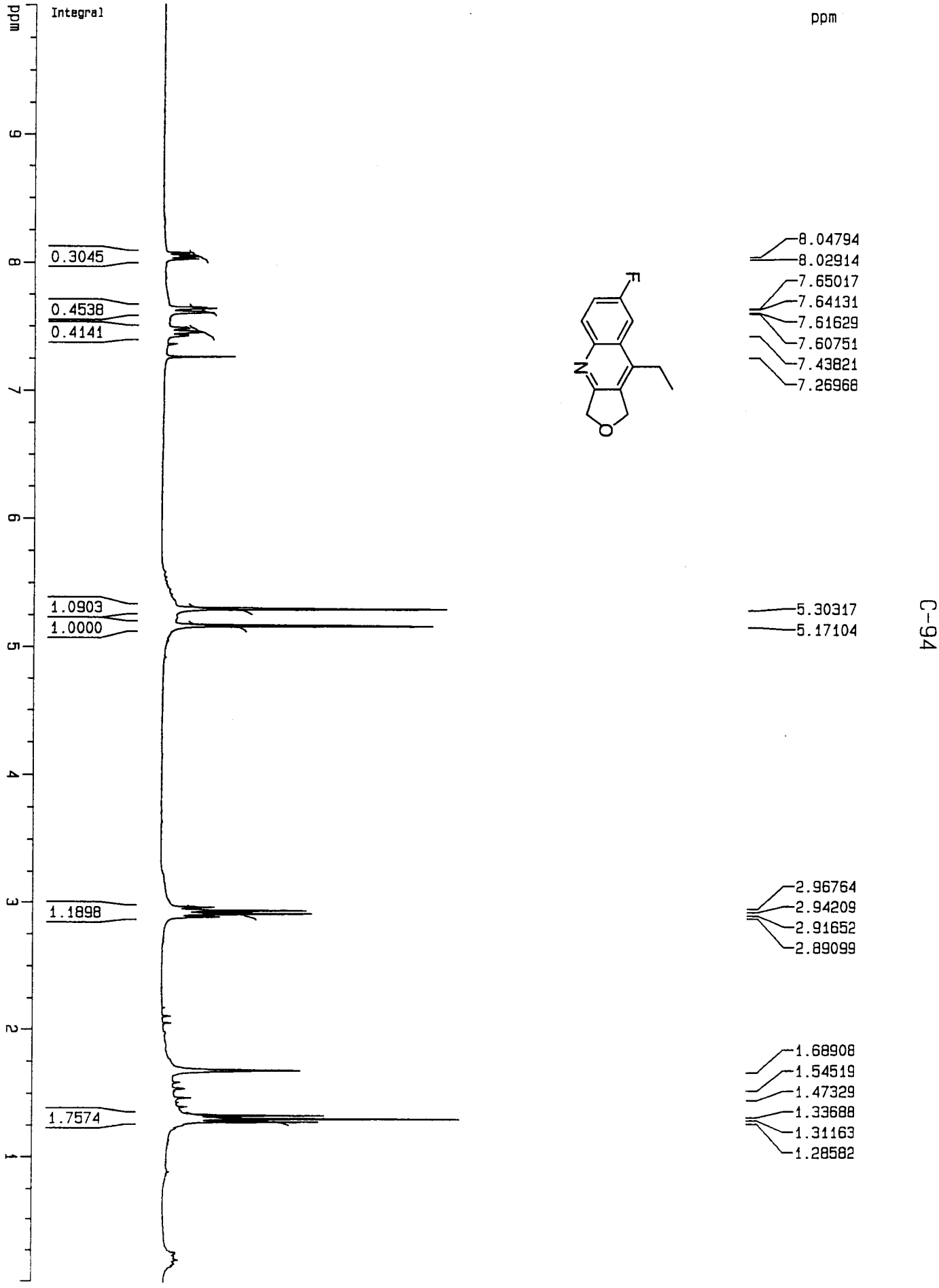


Using the general procedure B, the title compound was prepared in 64% yield. IR: 3063, 1612, 1601, 1459, 1401, 1356, 1251, 1232, 745; ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.01 (td, 1H, J = 8.0, 1.0 hz), 7.09 (d, 7.9 Hz), 7.41 (dd, 1H, J = 7.2, 1.2 Hz), 7.47 (dd, J = 8.2, 1.3 hz), 7.54 (7.82 (m, 8H), 8.27 (d, J = 8.3 Hz), 10.95 (br s, 1H); ^{13}C NMR ($CDCl_3$, 75 MHz) δ 110.8, 116.7, 119.8, 121.1, 122.9, 123.1, 123.8, 126.5, 126.6, 127.9, 128.6, 129.0, 129.3, 136.4, 141.4, 142.9, 146.2, 153.3; HRMS *m/z* calcd for $C_{21}H_{14}N_2$ 294.1157, found 294.1144.

11-*n*-Butyl-6-*H*-indolo[2,3-*b*]quinoline (13c).



Using the general procedure B, the title compound was prepared in 47% yield. IR: 2953, 2922, 2855, 1611, 1468, 1397, 1353, 1253, 1229, 1128, 741; ^{1}H NMR (300 MHz, $CDCl_3$) δ 1.08 (t, 3H, *J* = 7.2 Hz), 1.71 (m, 2H), 1.93 (m, 2H), 3.70 (t, 2H, *J* = 7.9 hz), 7.33 (t, 1H, *J* = 7.3 Hz), 7.52-7.60 (m, 3H), 7.79 (t, 1H, *J* 7.1 Hz), 8.19-8.23 (m, 2H), 8.31 (d, 1H, *J* = 8.2 Hz), 11.4 (br s, 1H); ^{13}C NMR ($CDCl_3$, 75 MHz) δ 14.1, 23.4, 28.9, 31.7, 110,9, 116.5, 120.1, 121.4, 122.8, 123.4, 124.1, 127.2, 127.5, 128.8, 141.2, 144.6, 146.3, 153.3; HRMS *m/z* calcd for $C_{19}H_{18}N_2$ 274.1470, found 274.1466.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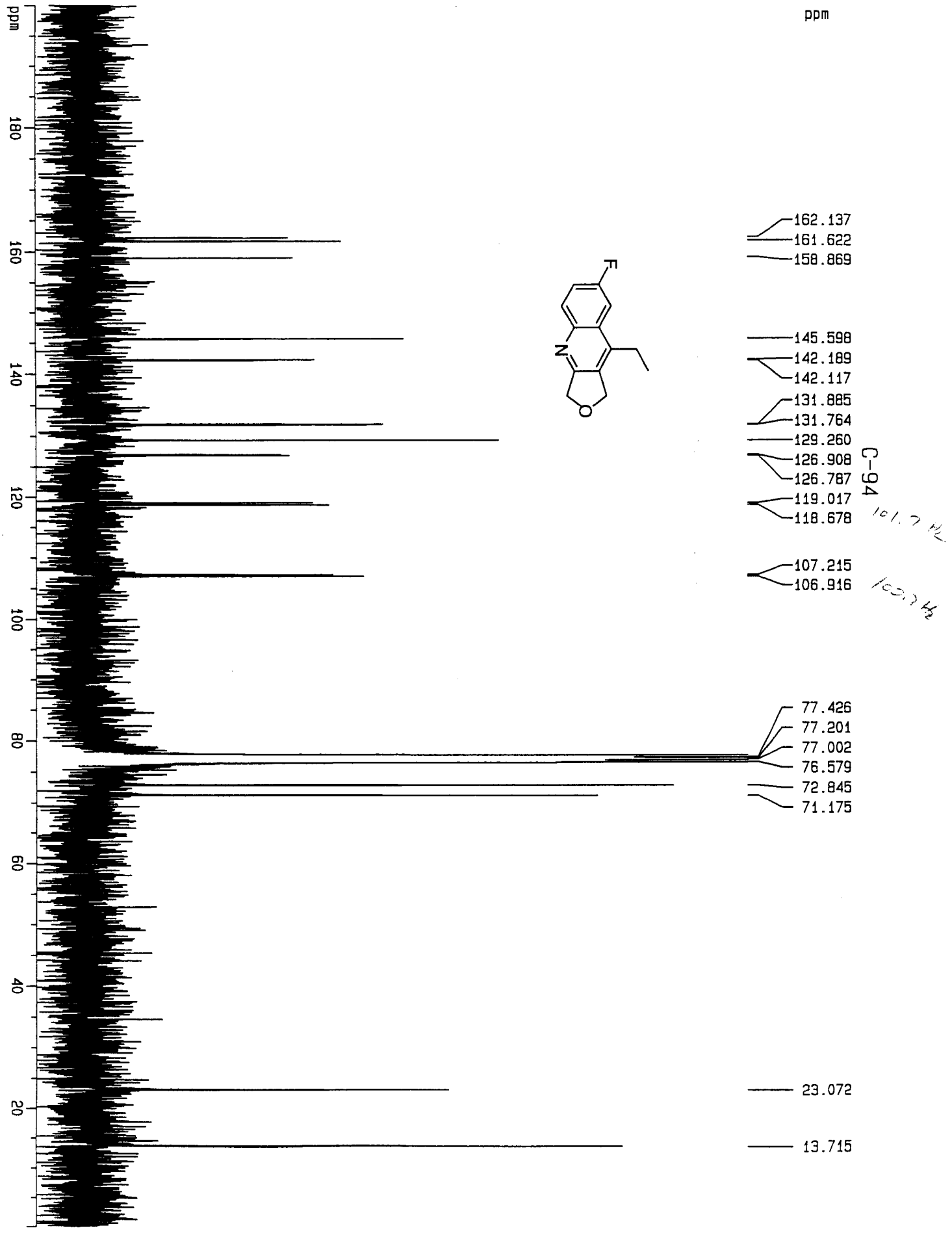
O

C-94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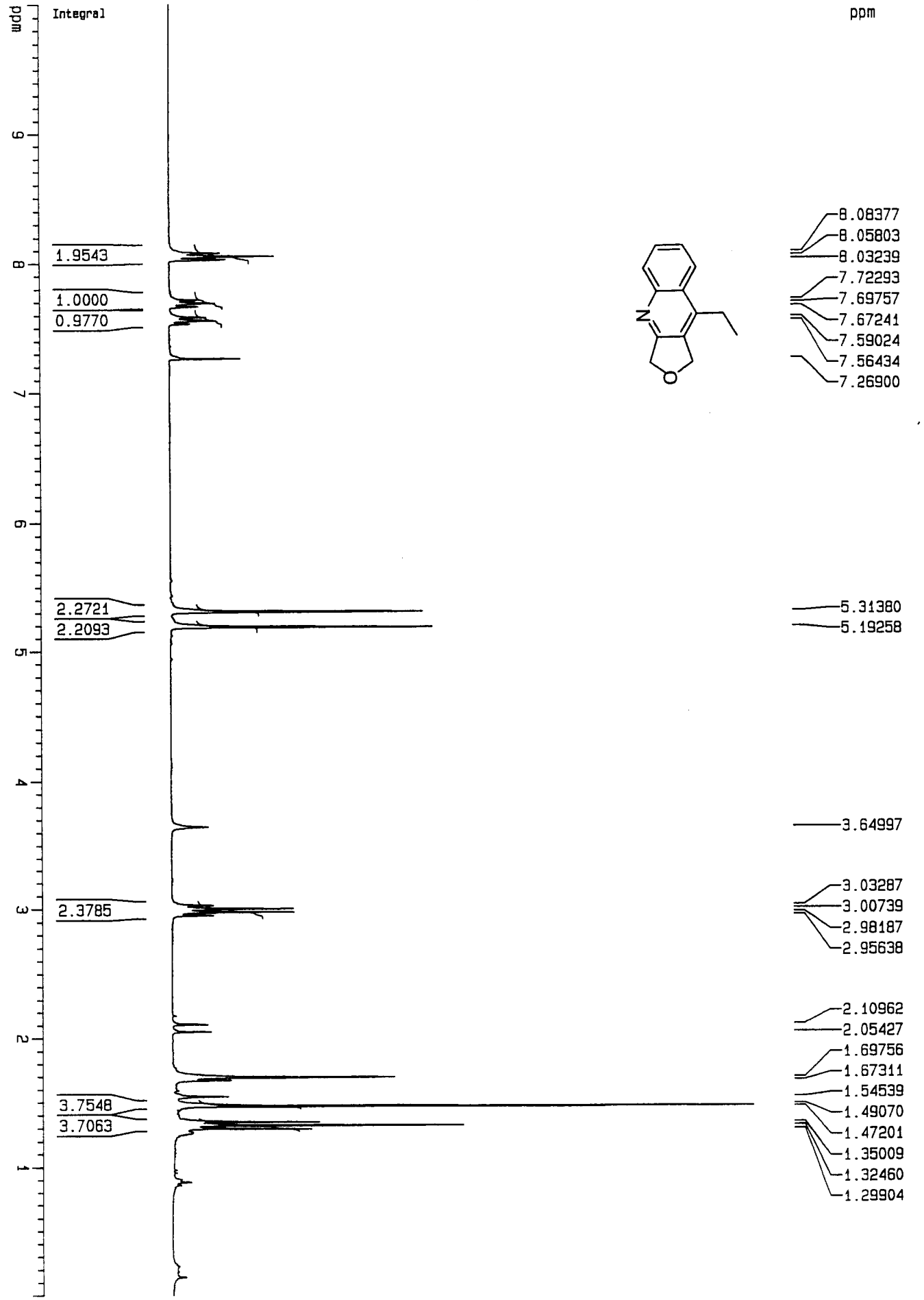
C-94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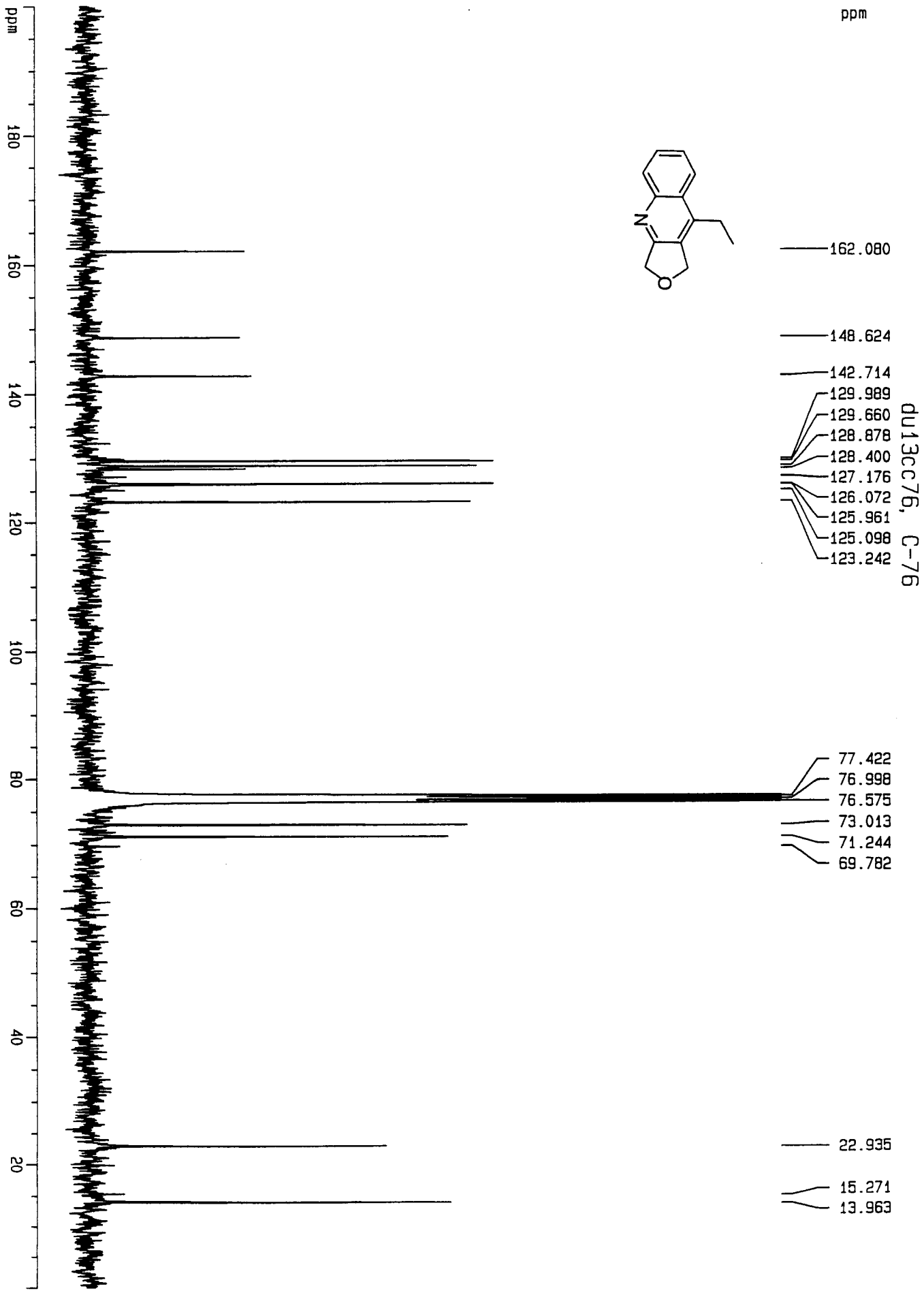
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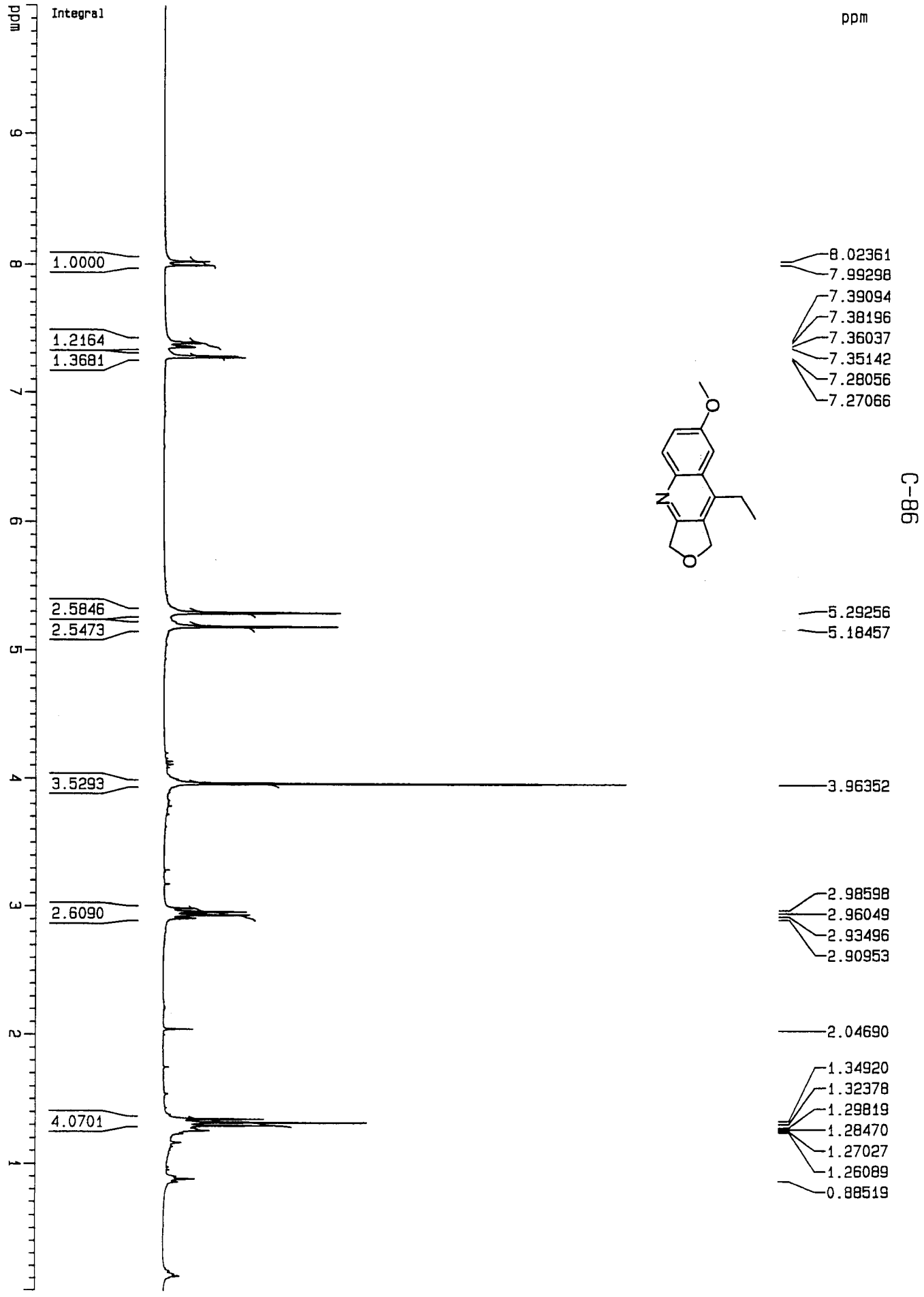
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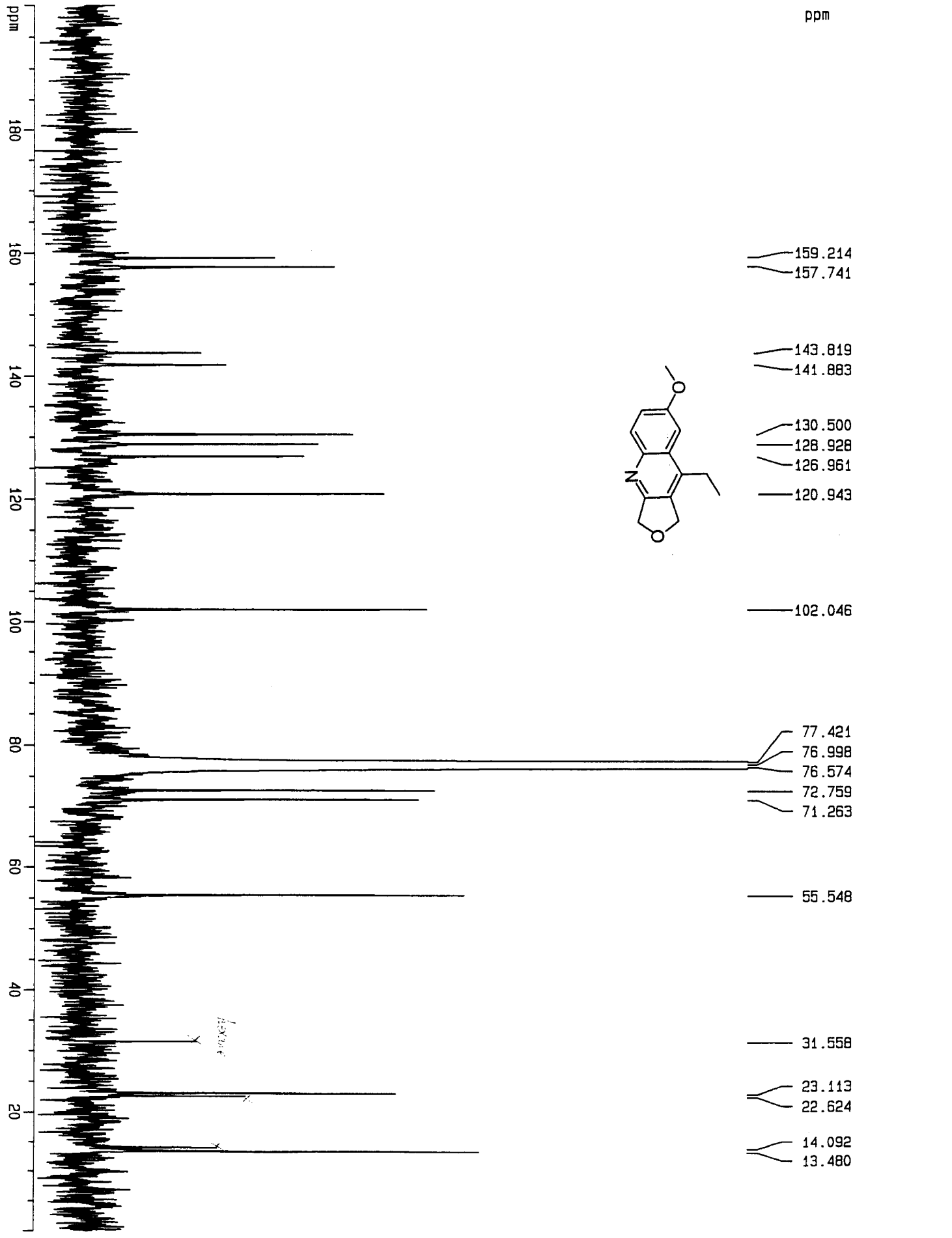
C-86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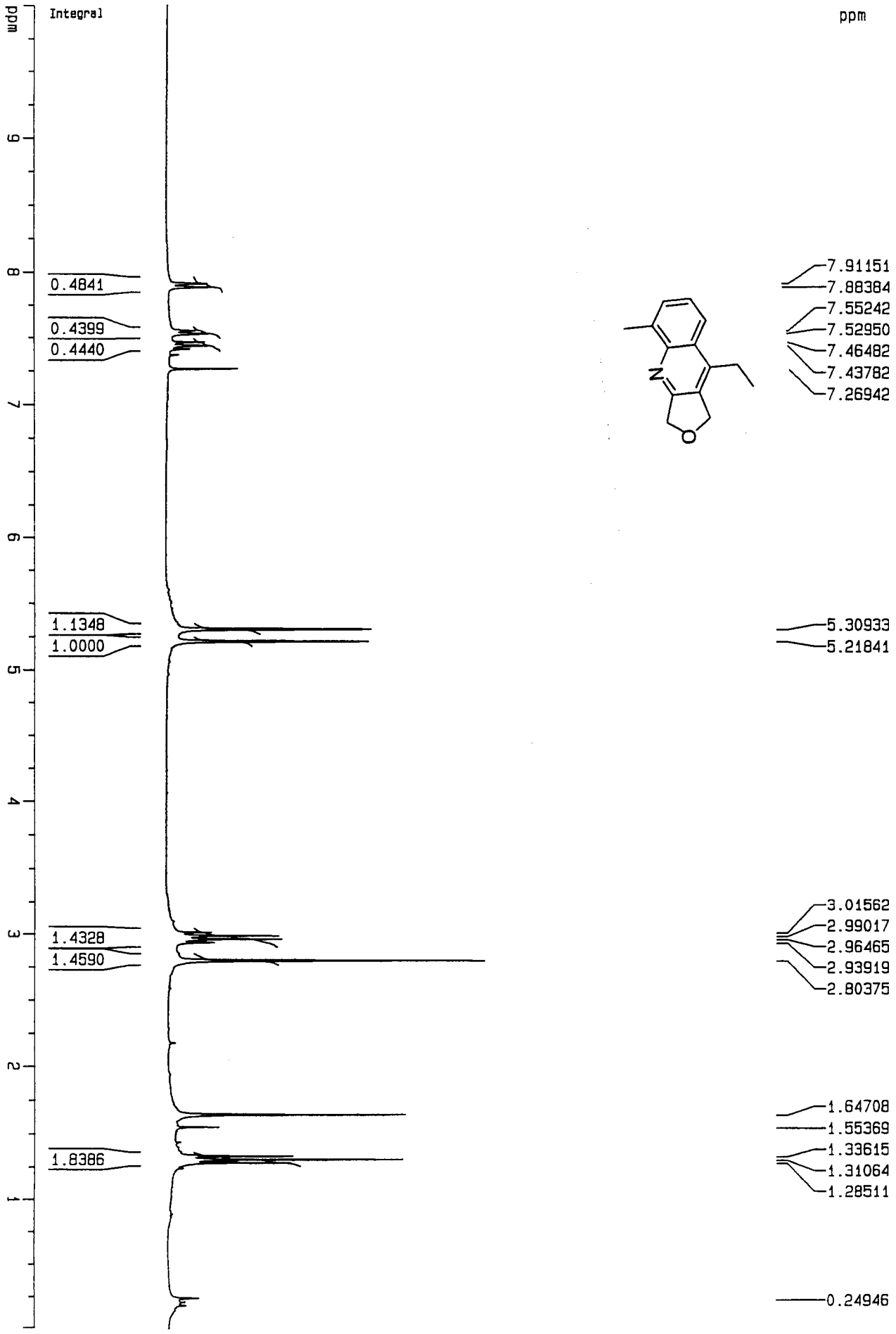
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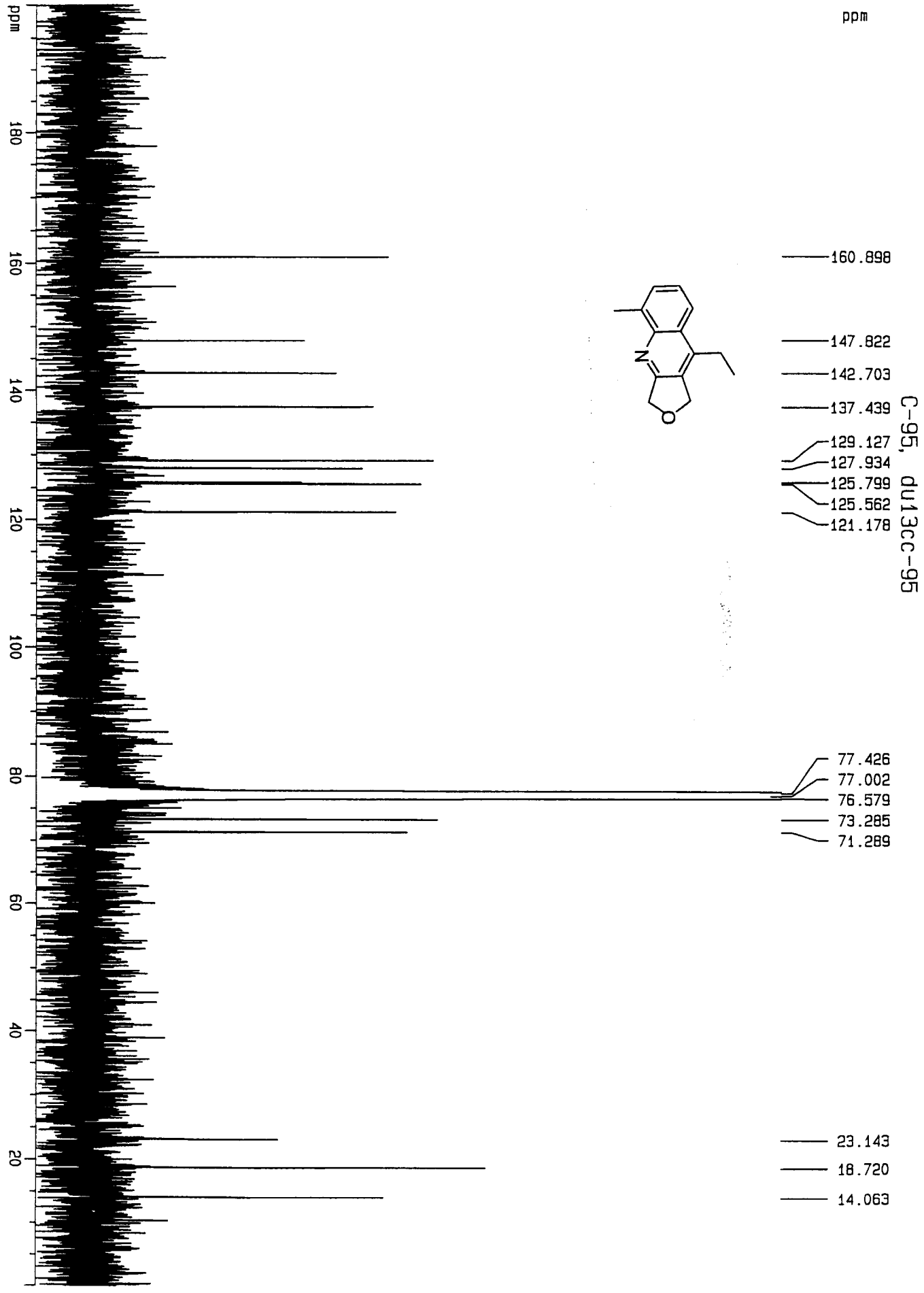
C-95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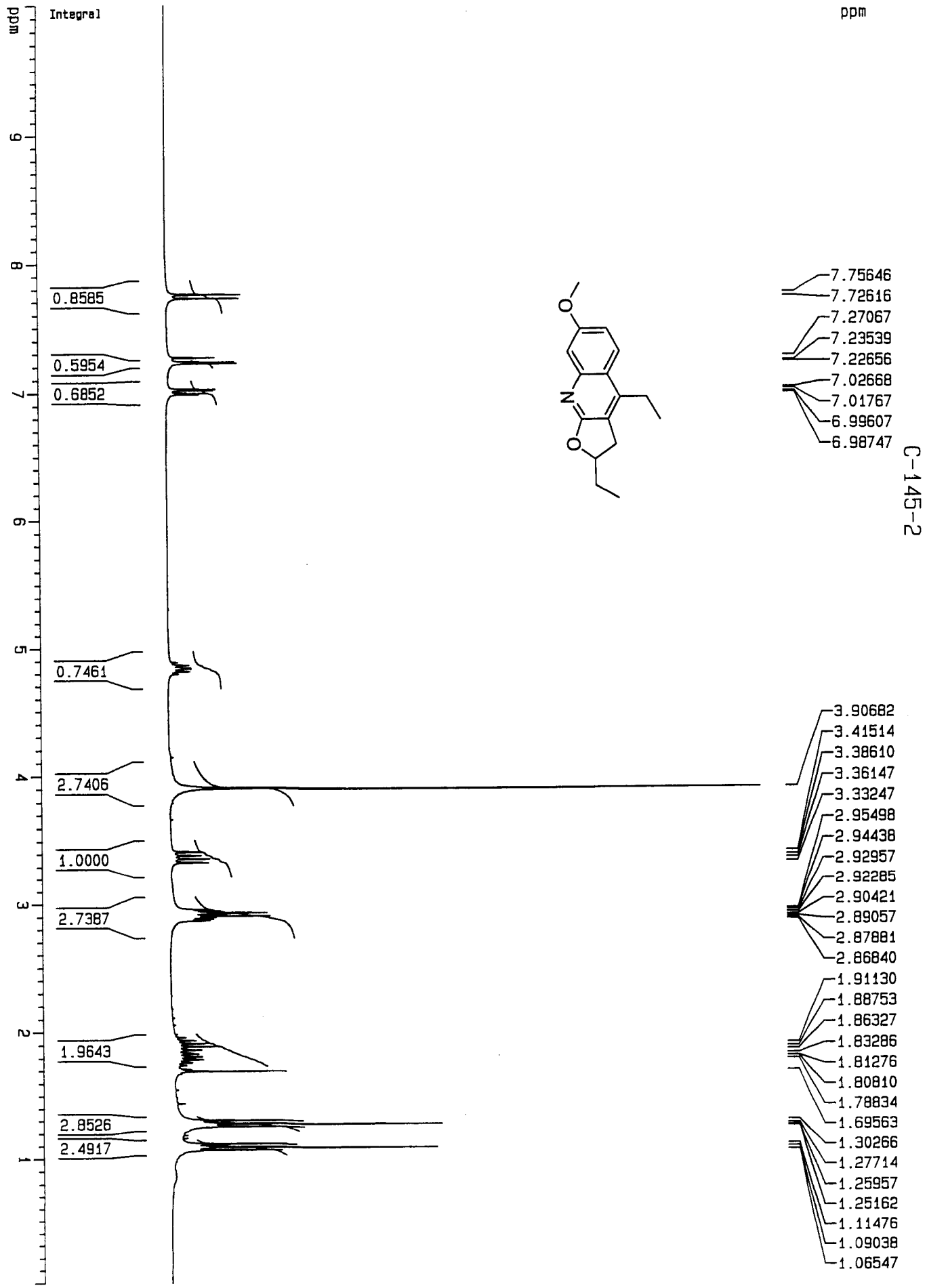
C-95, du13cc-95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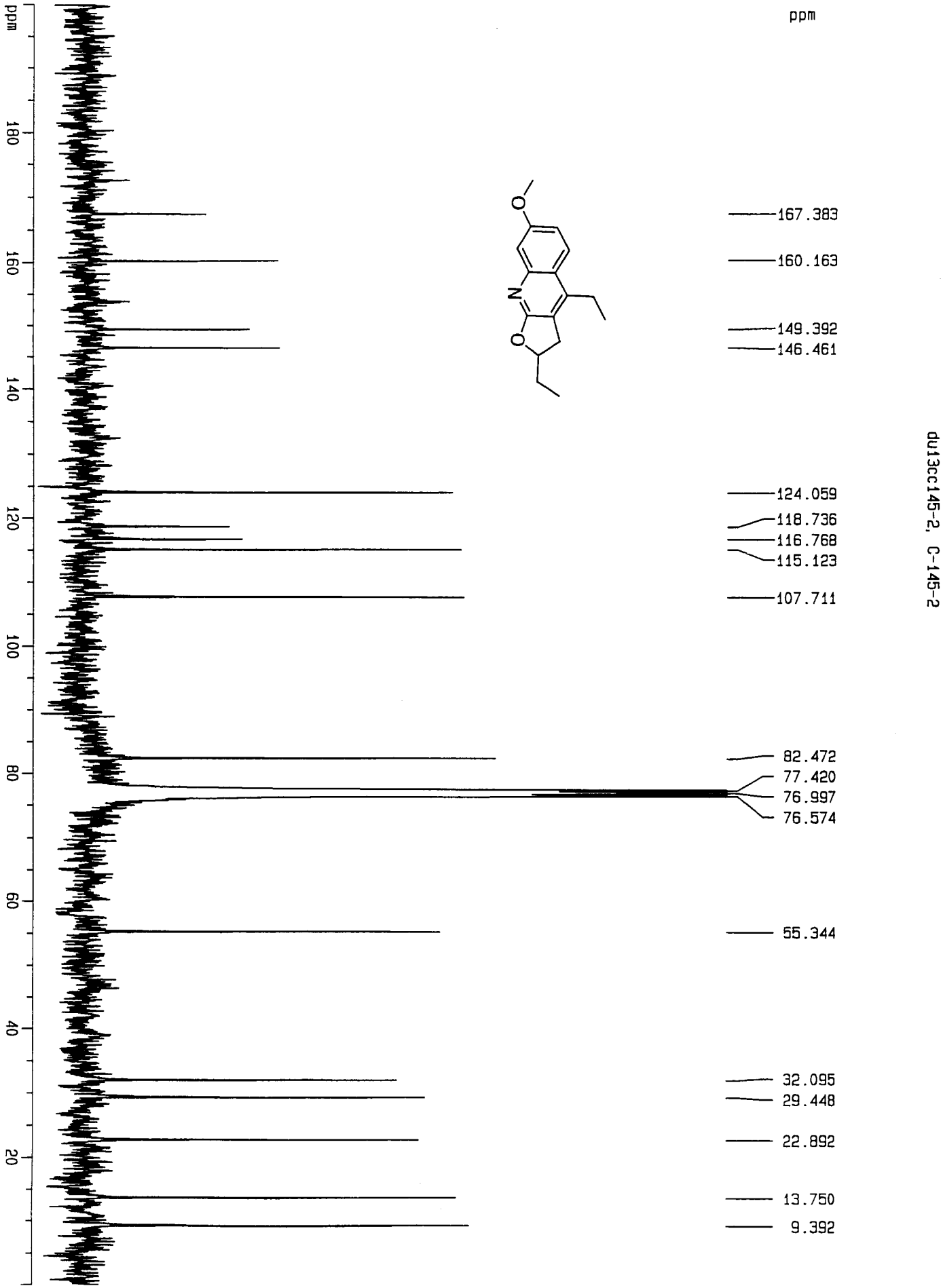
C-145-2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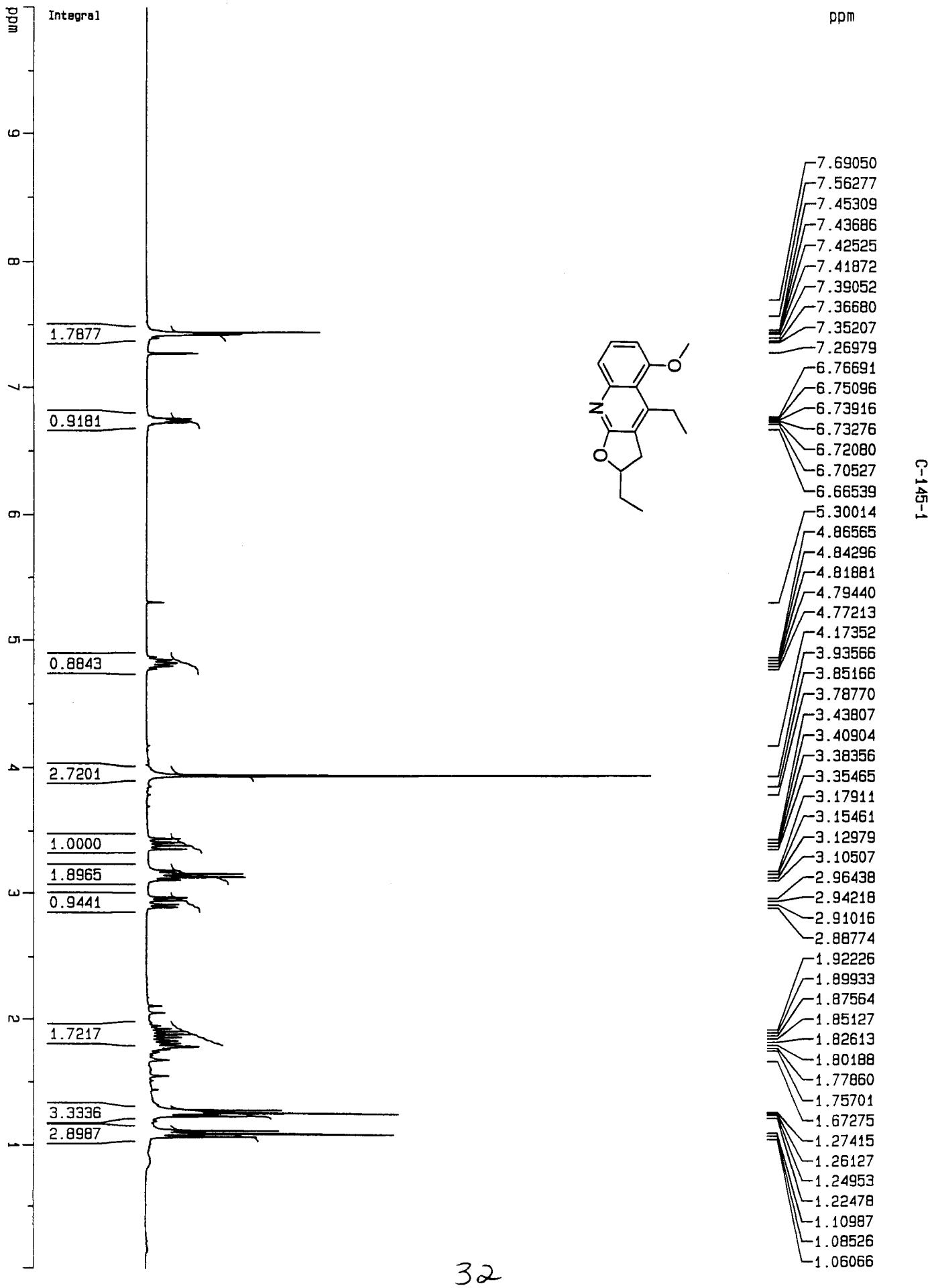
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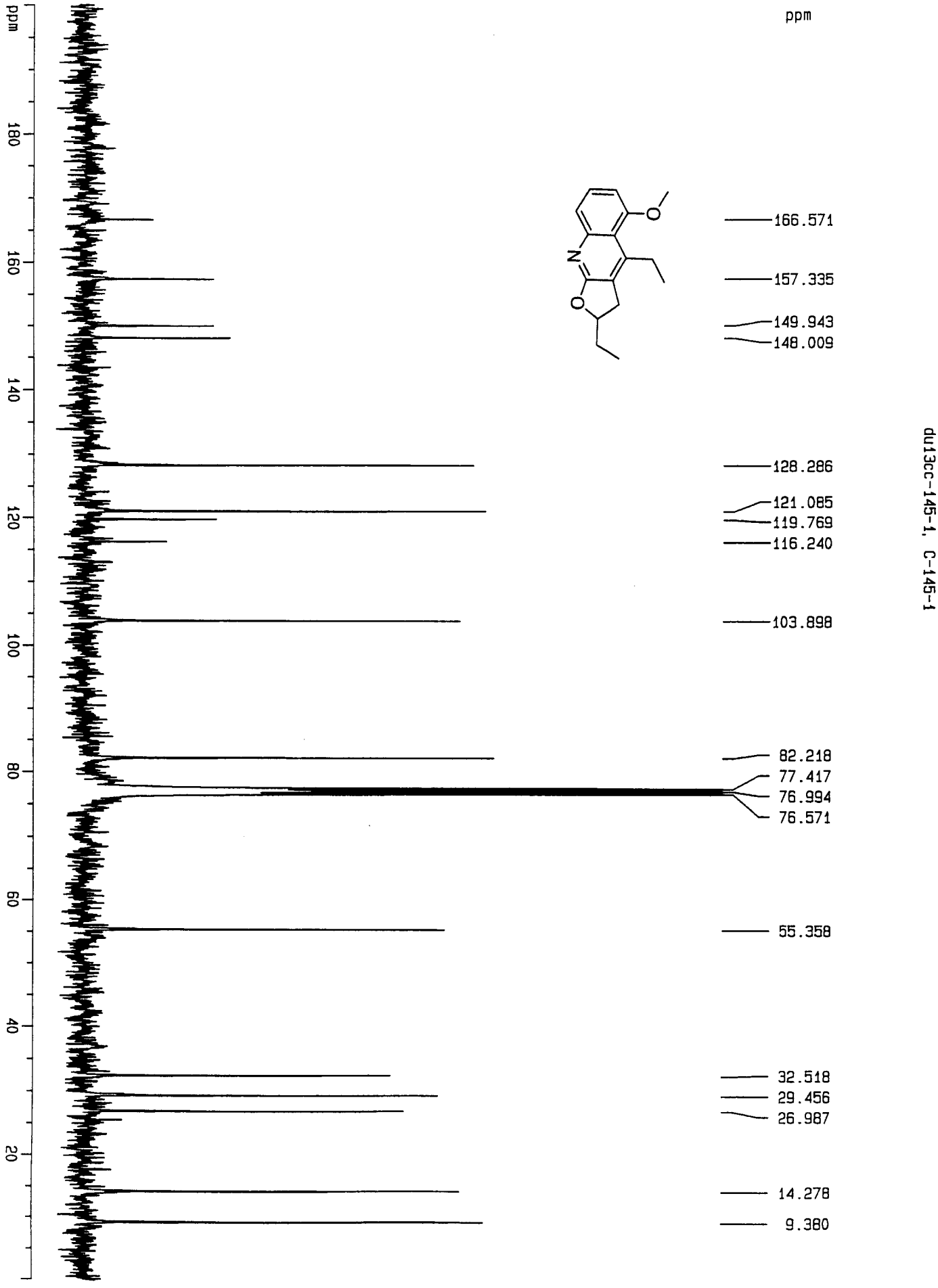
C-145-1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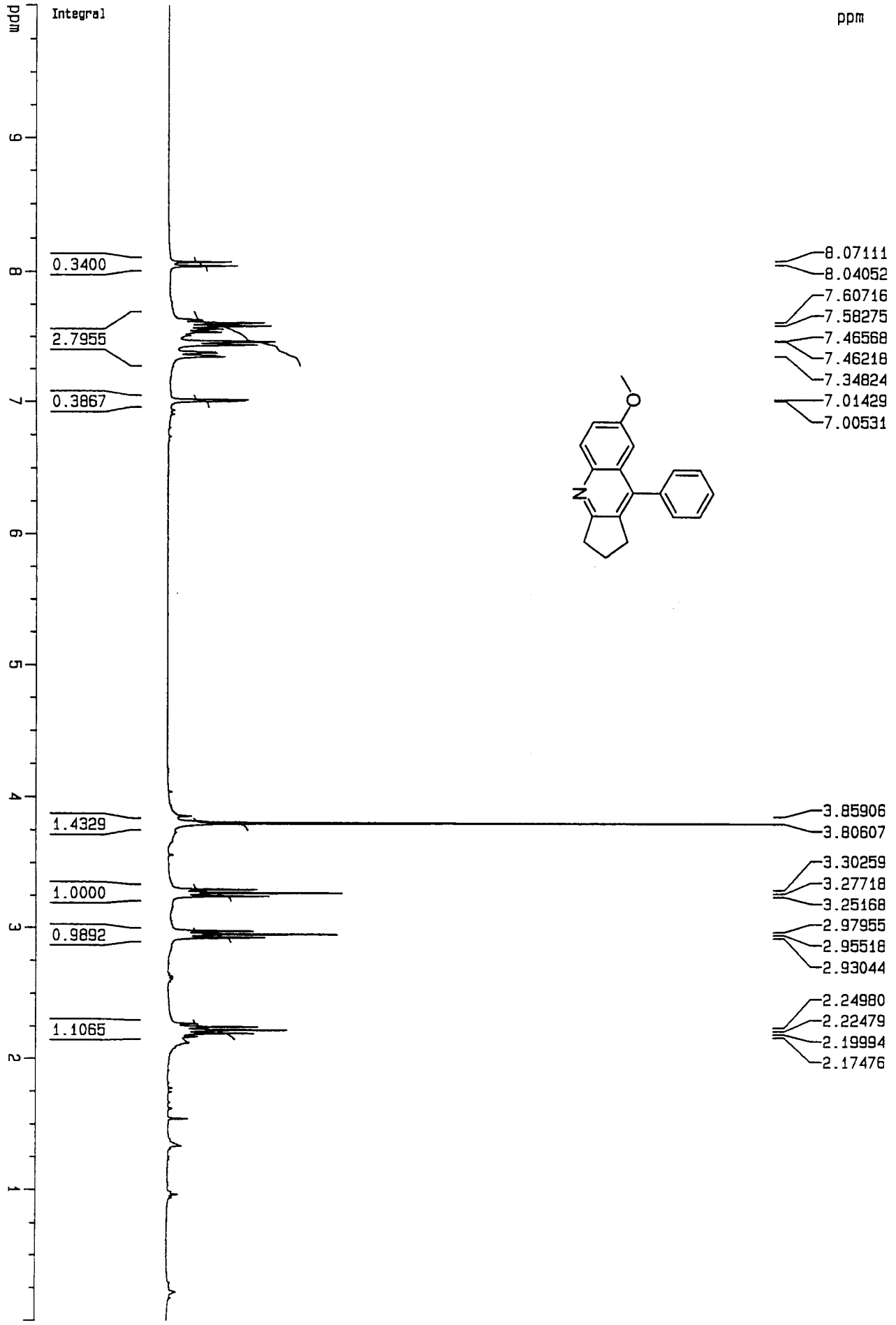
du13cc-145-1, C-145-1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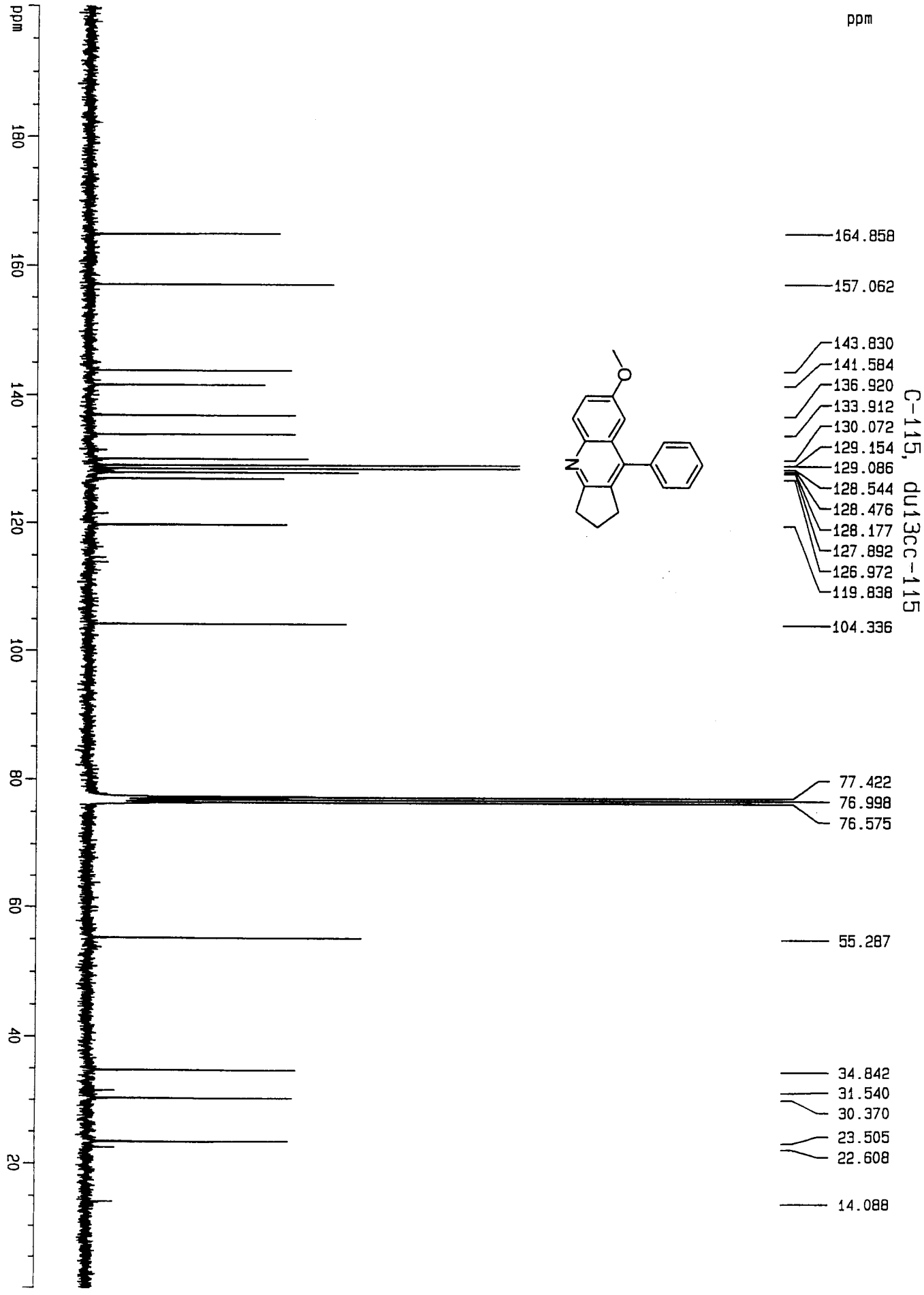
O
N

C-115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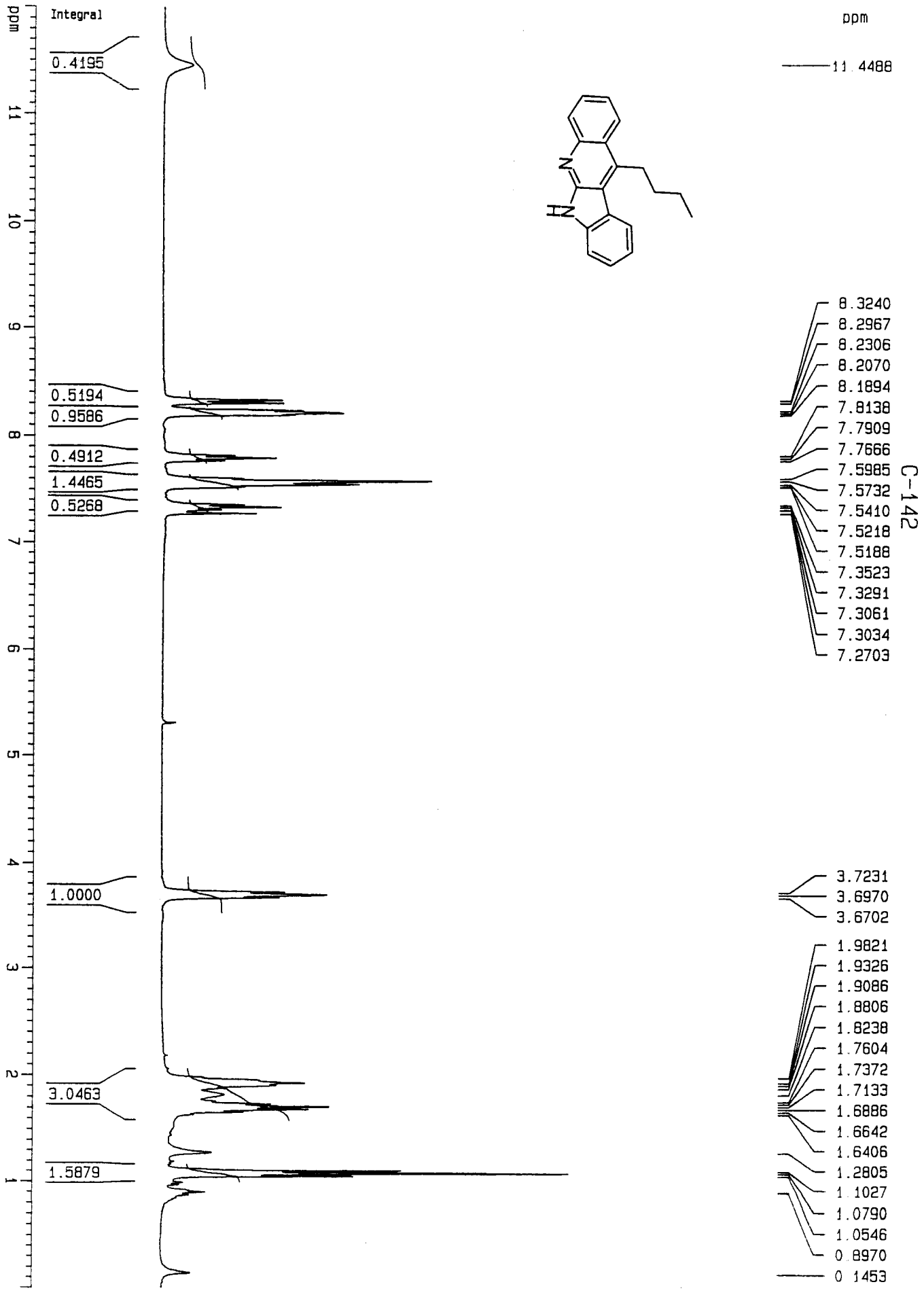
C-115, du13cc-115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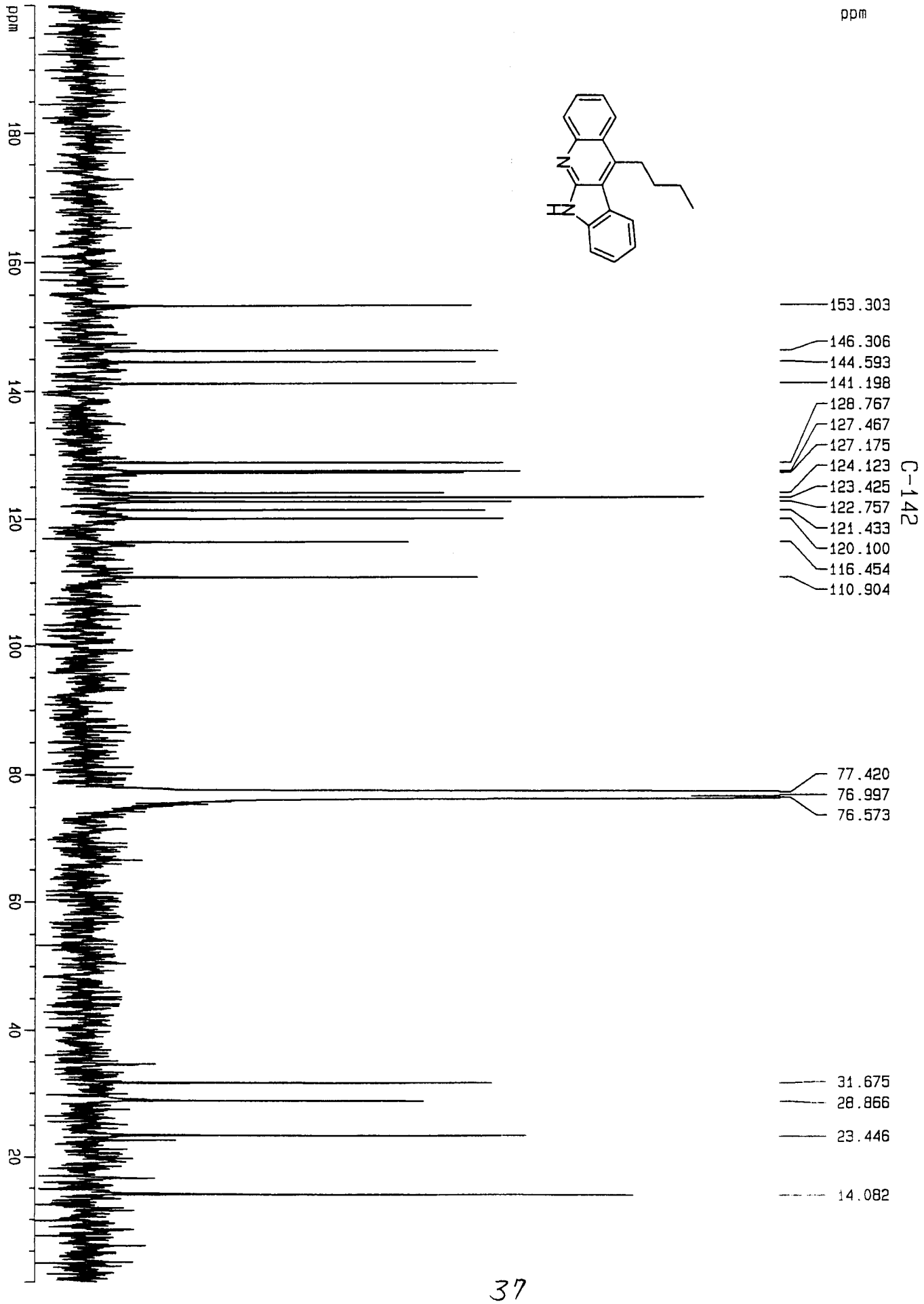
C-142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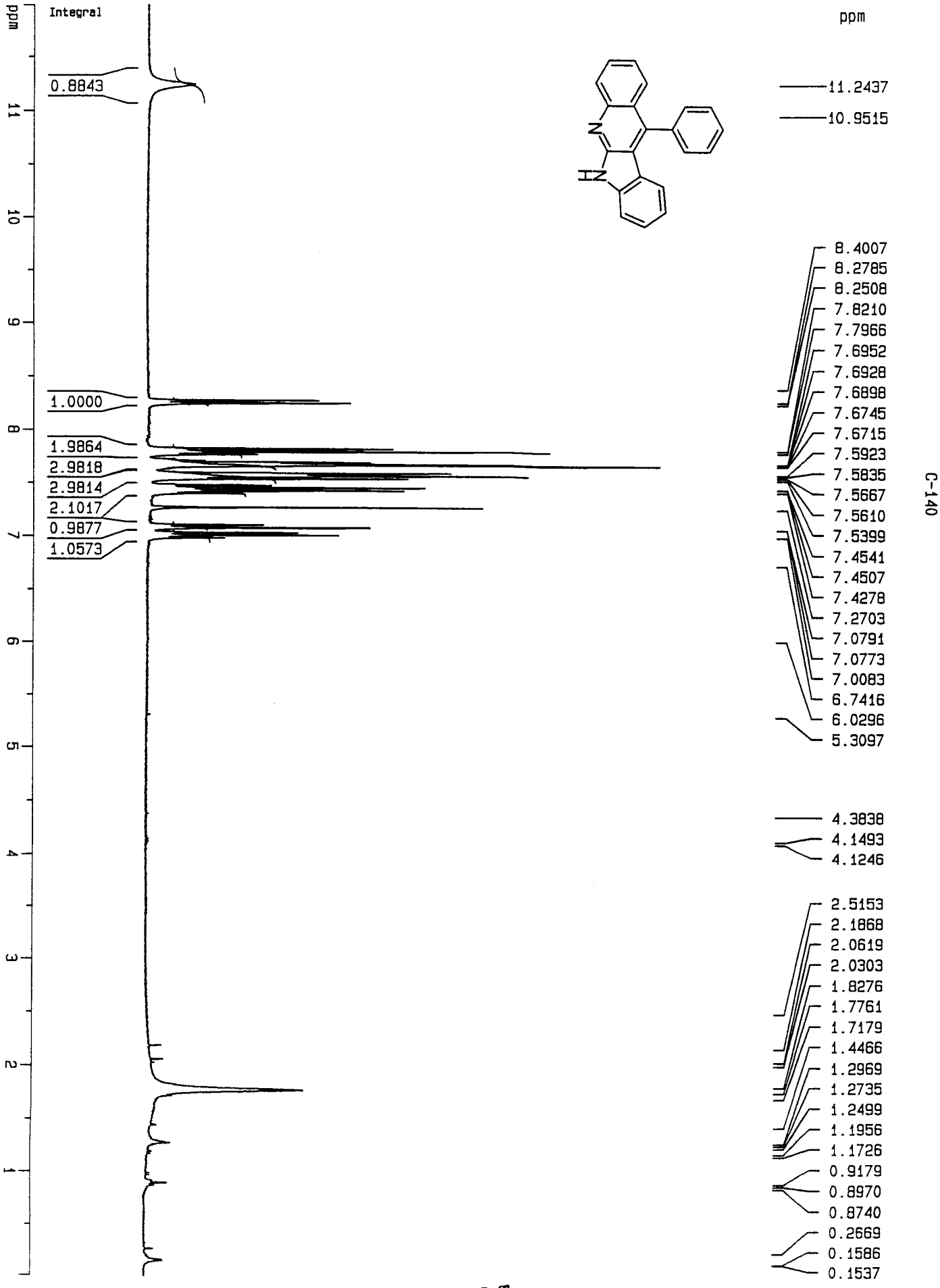
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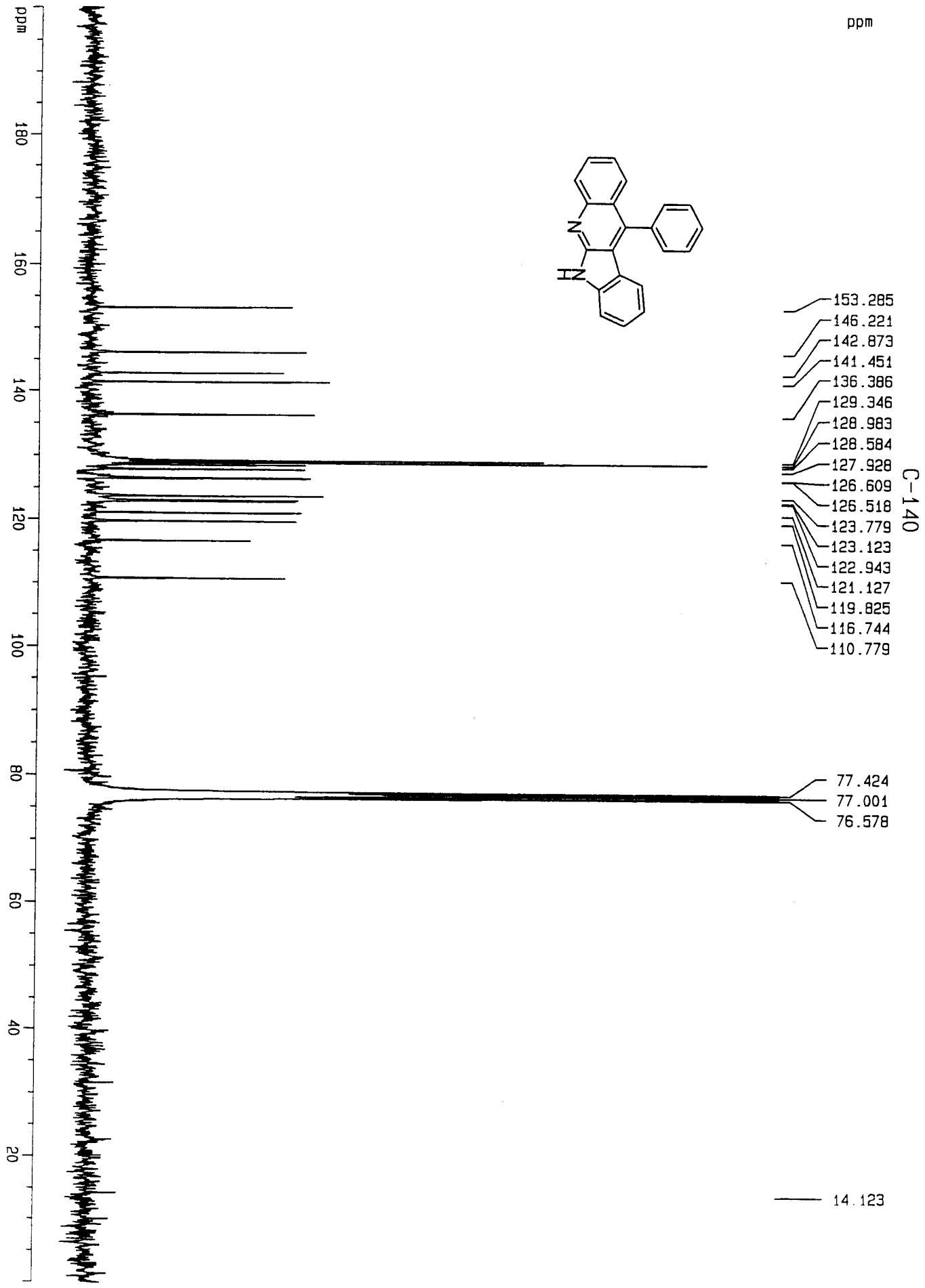
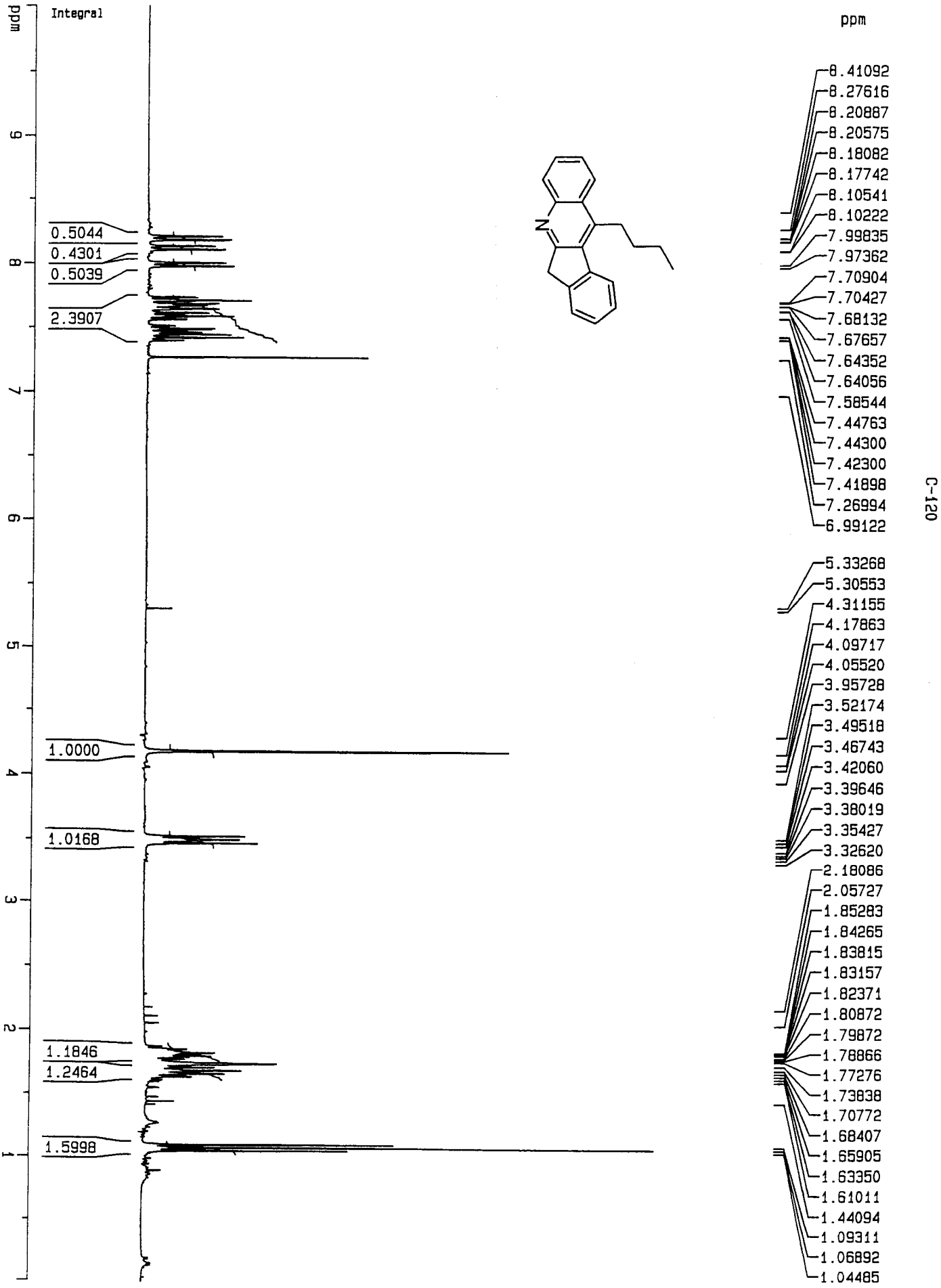
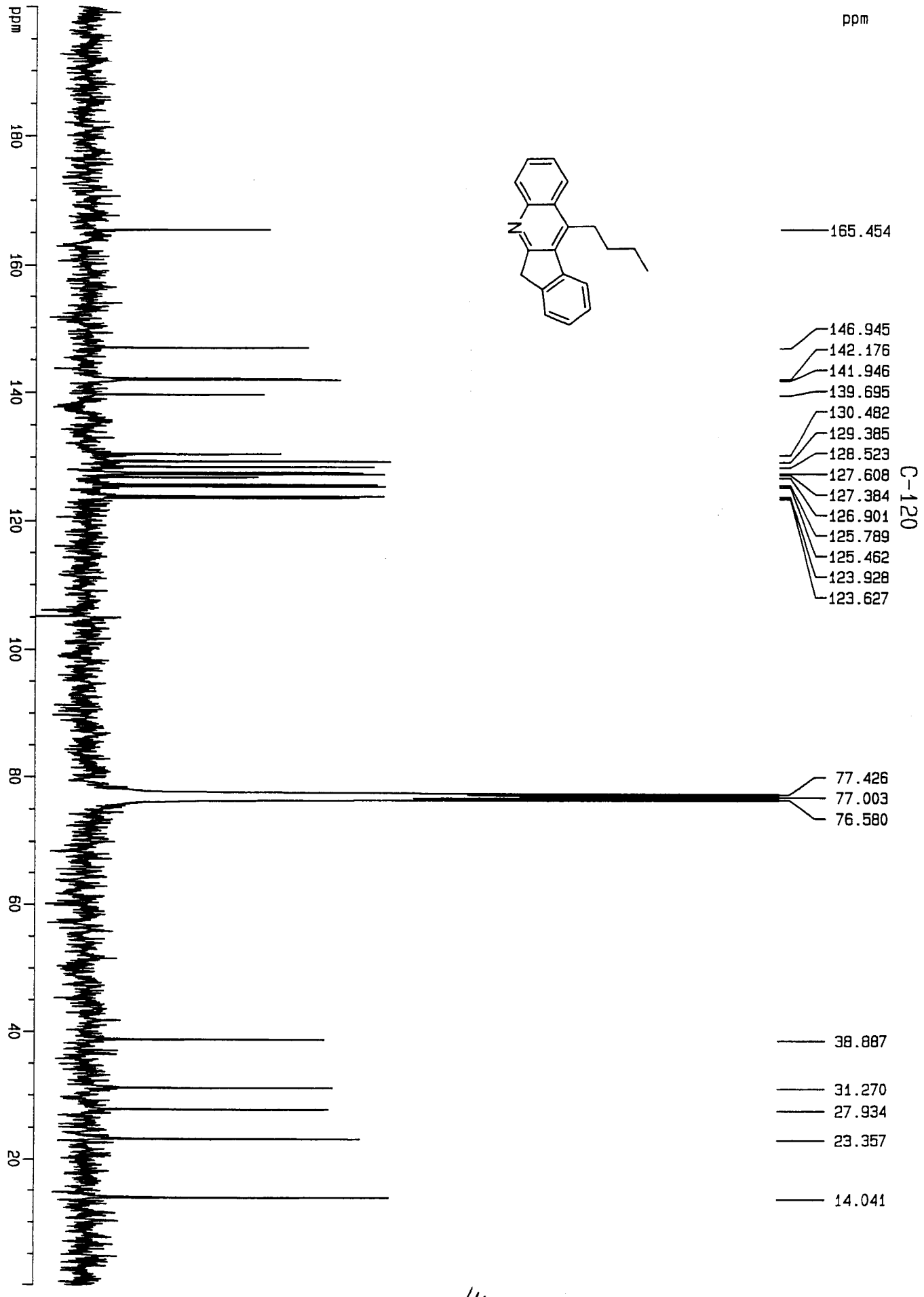
0.8970
0.8740
0.2669
0.1586
0.1537

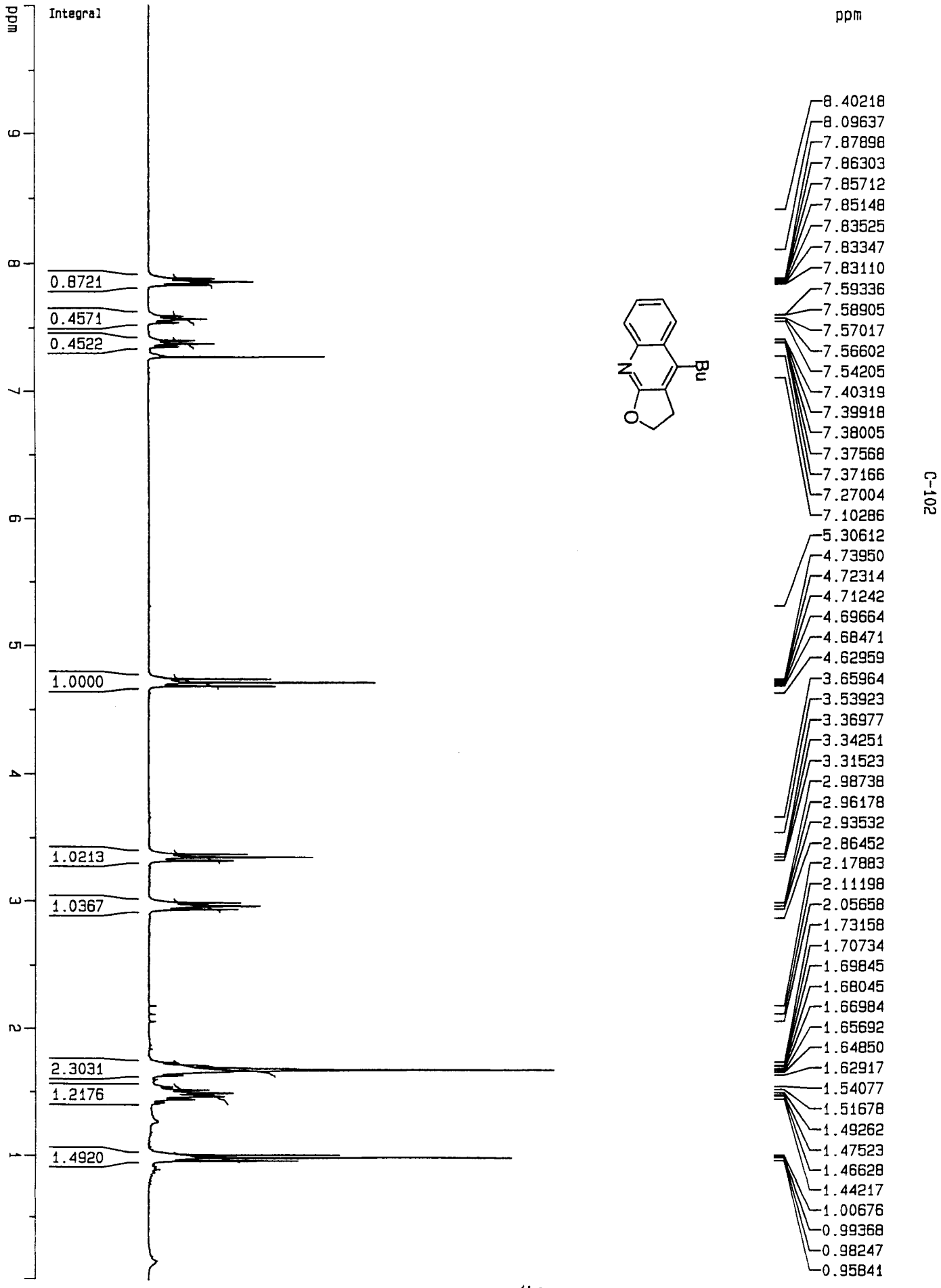
C-140


ppm
180
160
140
120
100
80
60
40
20
ppm
153.285
146.221
142.873
141.451
136.386
129.346
128.983
128.584
127.928
126.609
126.518
123.779
123.123
122.943
121.127
119.825
116.744
110.779
77.424
77.001
76.578
14.123
C-140

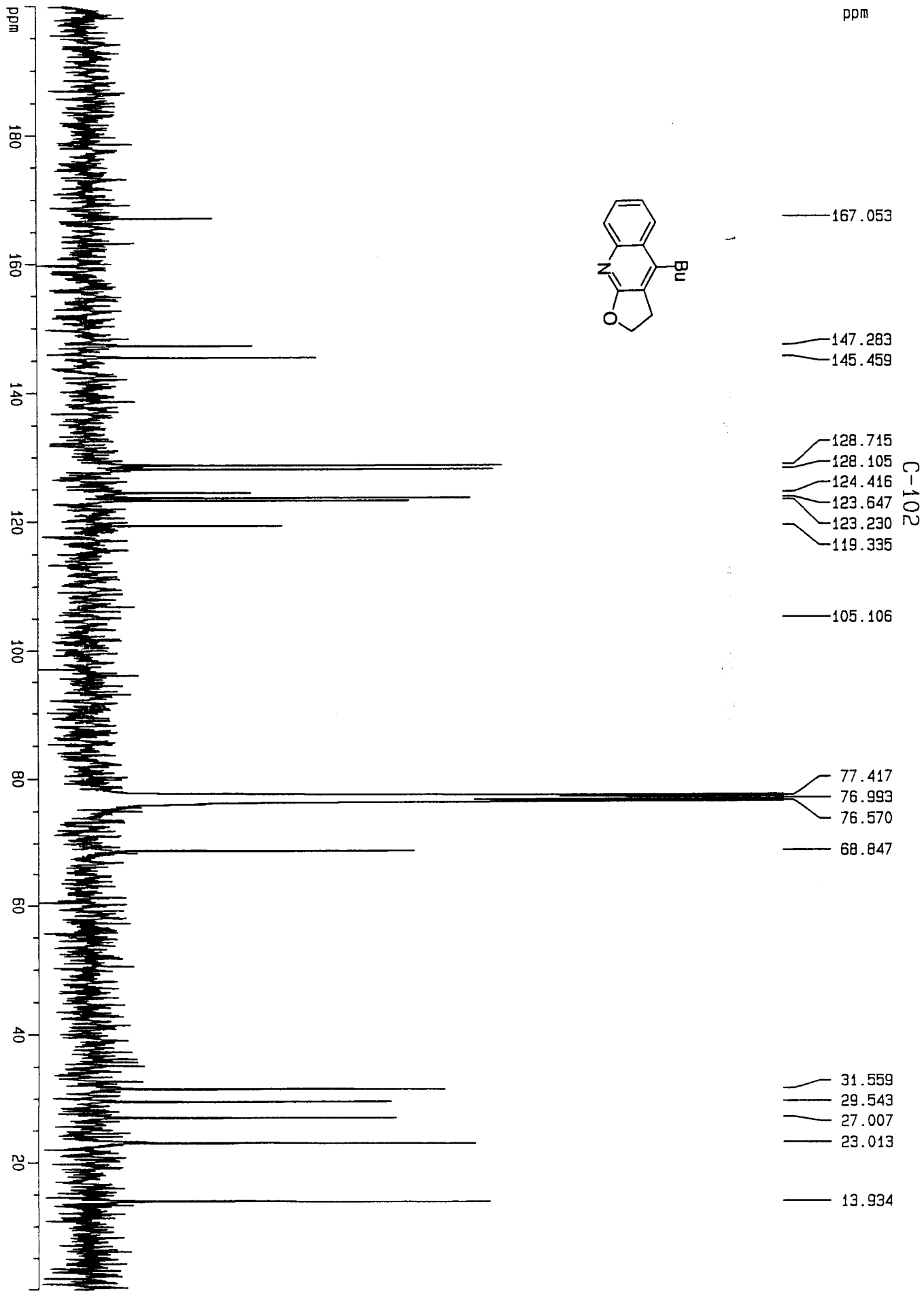

ppm
Integral
0.5044
0.4301
0.5039
2.3907
1.0000
1.0168
1.1846
1.2464
1.5998
ppm
8.41092
8.27616
8.20887
8.20575
8.18082
8.17742
8.10541
8.10222
7.99835
7.97362
7.70904
7.70427
7.68132
7.67657
7.64352
7.64056
7.58544
7.44763
7.44300
7.42300
7.41898
7.26994
6.99122
5.33268
5.30553
4.31155
4.17863
4.09717
4.05520
3.95728
3.52174
3.49518
3.46743
3.42060
3.39646
3.38019
3.35427
3.32620
2.18086
2.05727
1.85283
1.84265
1.83815
1.83157
1.82371
1.80872
1.79872
1.78866
1.77276
1.73838
1.70772
1.68407
1.65905
1.63350
1.61011
1.44094
1.09311
1.06892
1.04485

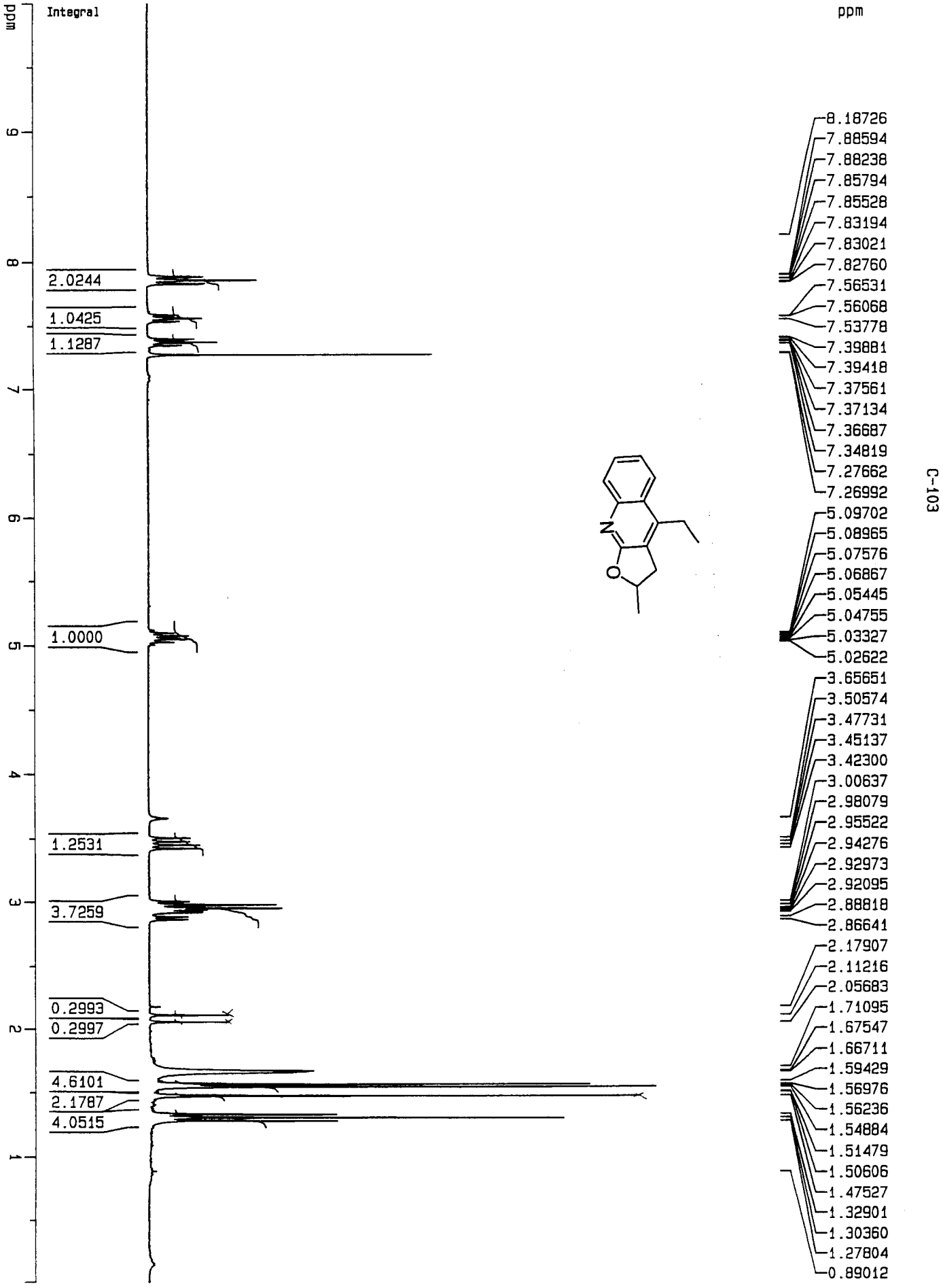
C-120


ppm
180
160
140
120
100
80
60
40
20
N
165.454
146.945
142.176
141.946
139.695
130.482
129.385
128.523
127.608
127.384
126.901
125.789
125.462
123.928
123.627
77.426
77.003
76.580
38.887
31.270
27.934
23.357
14.041
C-120

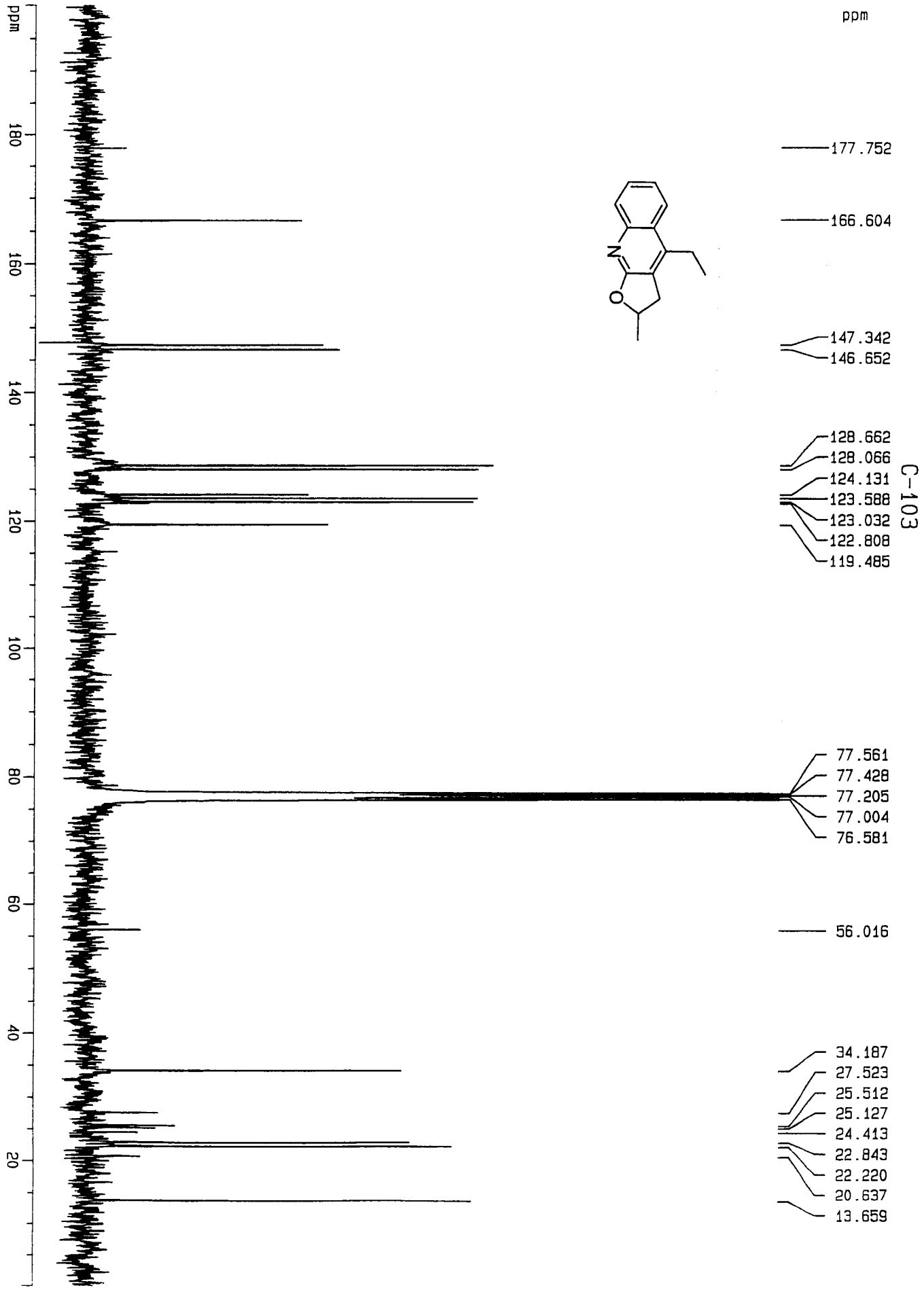

ppm
Integral
0.8721
0.4571
0.4522
1.0000
1.0213
1.0367
2.3031
1.2176
1.4920
Bu
N
O
ppm
8.40218
8.09637
7.87898
7.86303
7.85712
7.85148
7.83525
7.83347
7.83110
7.59336
7.58905
7.57017
7.56602
7.54205
7.40319
7.39918
7.38005
7.37568
7.37166
7.27004
7.10286
5.30612
4.73950
4.72314
4.71242
4.69664
4.68471
4.62959
3.65964
3.53923
3.36977
3.34251
3.31523
2.98738
2.96178
2.93532
2.86452
2.17883
2.11198
2.05658
1.73158
1.70734
1.69845
1.68045
1.66984
1.65692
1.64850
1.62917
1.54077
1.51678
1.49262
1.47523
1.46628
1.44217
1.00676
0.99368
0.98247
0.95841

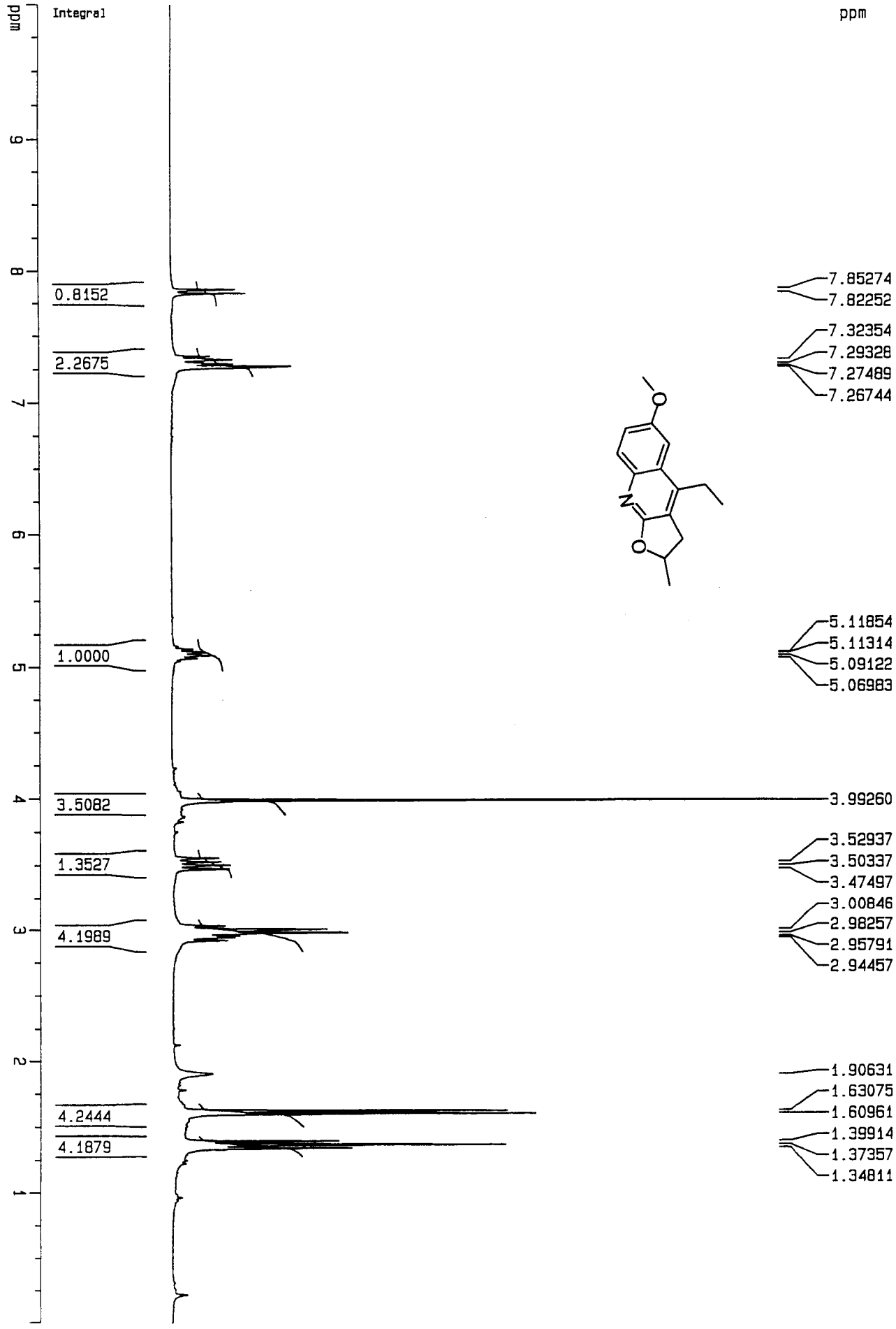
C-102


ppm
180
160
140
120
100
80
60
40
20
N
O
Bu
167.053
147.283
145.459
128.715
128.105
124.416
123.647
123.230
119.335
105.106
77.417
76.993
76.570
68.847
31.559
29.543
27.007
23.013
13.934
C-102

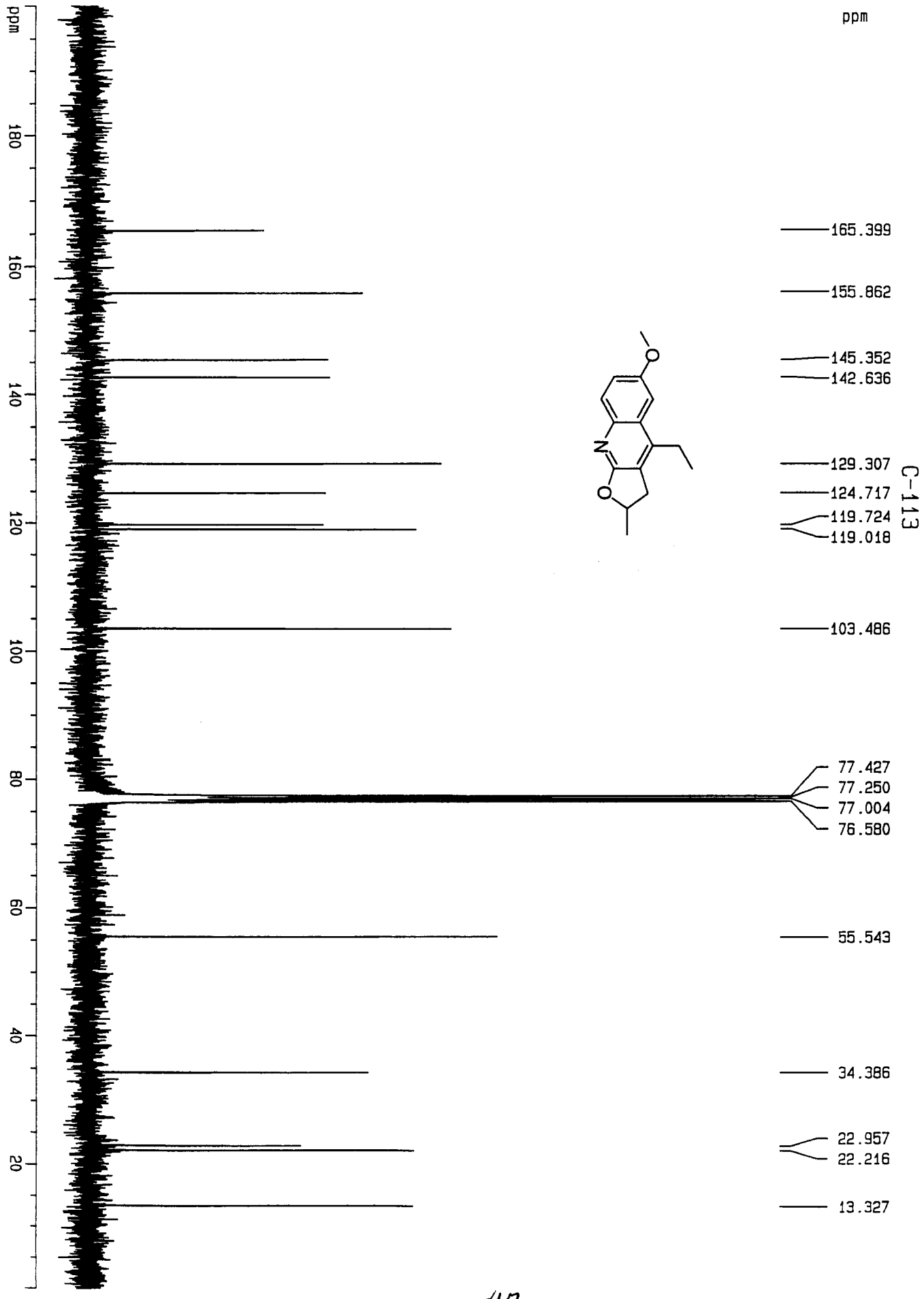

ppm
Integral
2.0244
1.0425
1.1287
1.0000
1.2531
3.7259
0.2993
0.2997
4.6101
2.1787
4.0515
ppm
8.18726
7.88594
7.88238
7.85794
7.85528
7.83194
7.83021
7.82760
7.56531
7.56068
7.53778
7.39881
7.39418
7.37561
7.37134
7.36687
7.34819
7.27662
7.26992
5.09702
5.08965
5.07576
5.06867
5.05445
5.04755
5.03327
5.02622
3.65651
3.50574
3.47731
3.45137
3.42300
3.00637
2.98079
2.95522
2.94276
2.92973
2.92095
2.88818
2.86641
2.17907
2.11216
2.05683
1.71095
1.67547
1.66711
1.59429
1.56976
1.56236
1.54884
1.51479
1.50606
1.47527
1.32901
1.30360
1.27804
0.89012
N
O

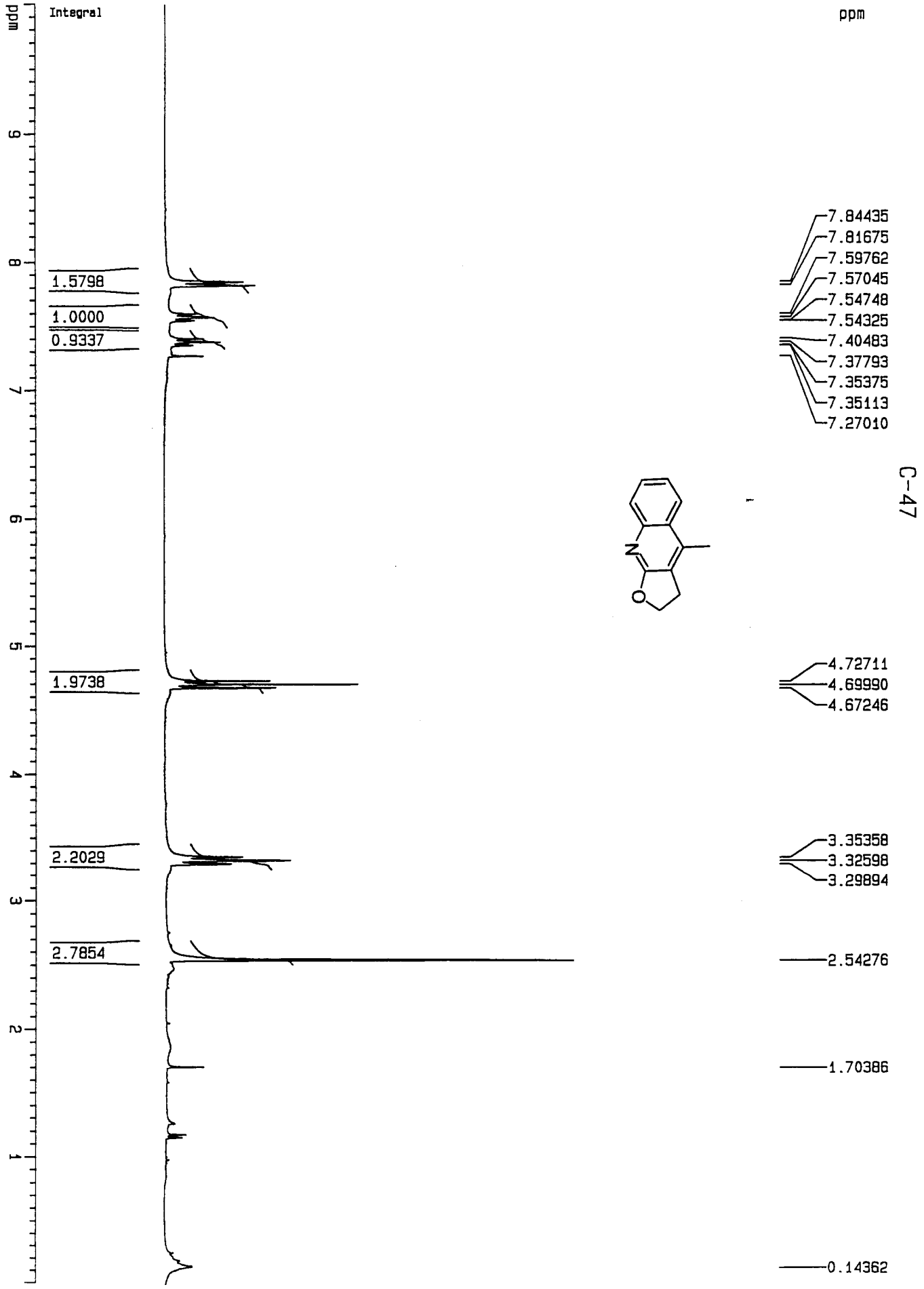
C-103


ppm
180
160
140
120
100
80
60
40
20
ppm
177.752
166.604
147.342
146.652
128.662
128.066
124.131
123.588
123.032
122.808
119.485
77.561
77.428
77.205
77.004
76.581
56.016
34.187
27.523
25.512
25.127
24.413
22.843
22.220
20.637
13.659
N
O
C-103

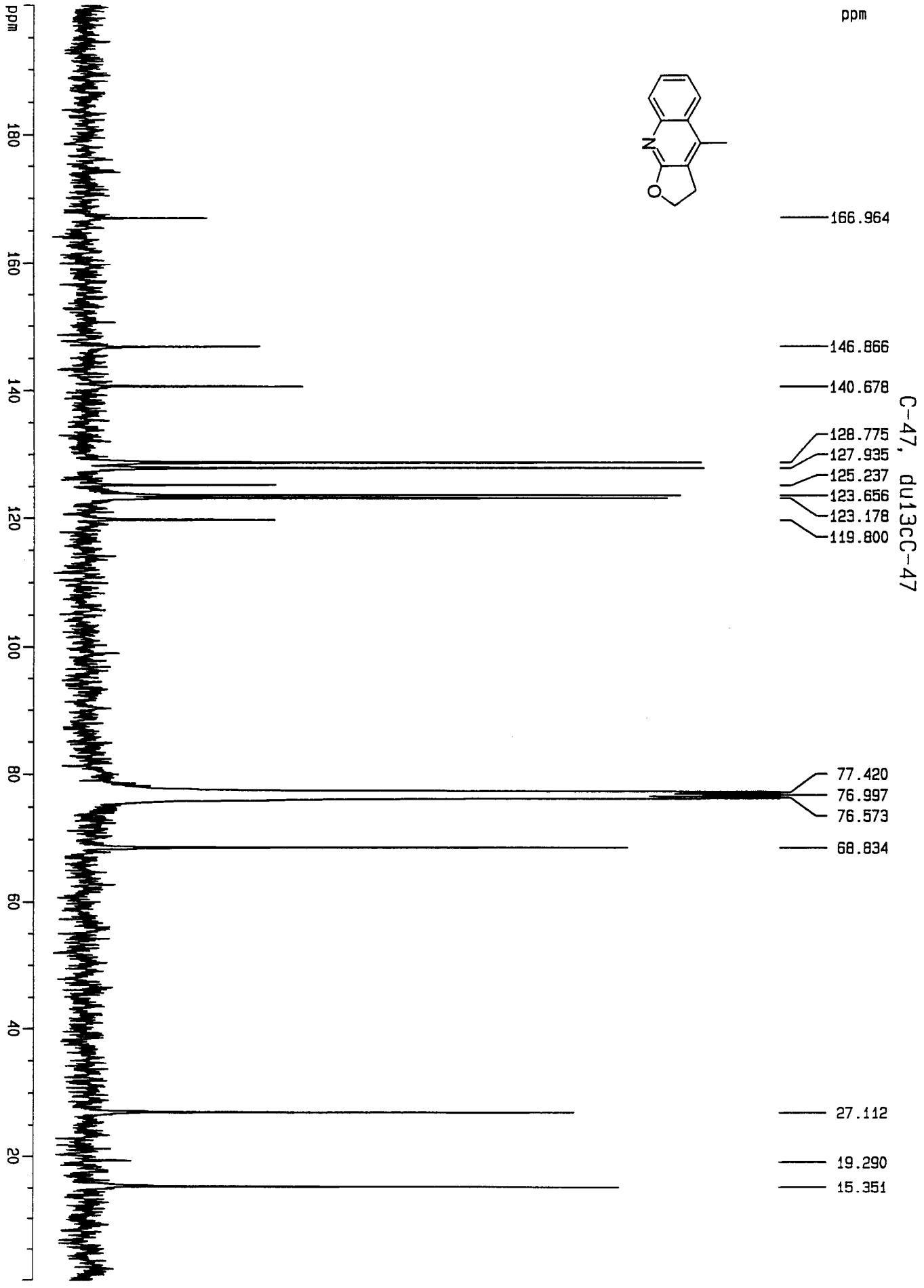

Integral
ppm
0.8152
2.2675
1.0000
3.5082
1.3527
4.1989
4.2444
4.1879
7.85274
7.82252
7.32354
7.29328
7.27489
7.26744
5.11854
5.11314
5.09122
5.06983
3.99260
3.52937
3.50337
3.47497
3.00846
2.98257
2.95791
2.94457
1.90631
1.63075
1.60961
1.39914
1.37357
1.34811

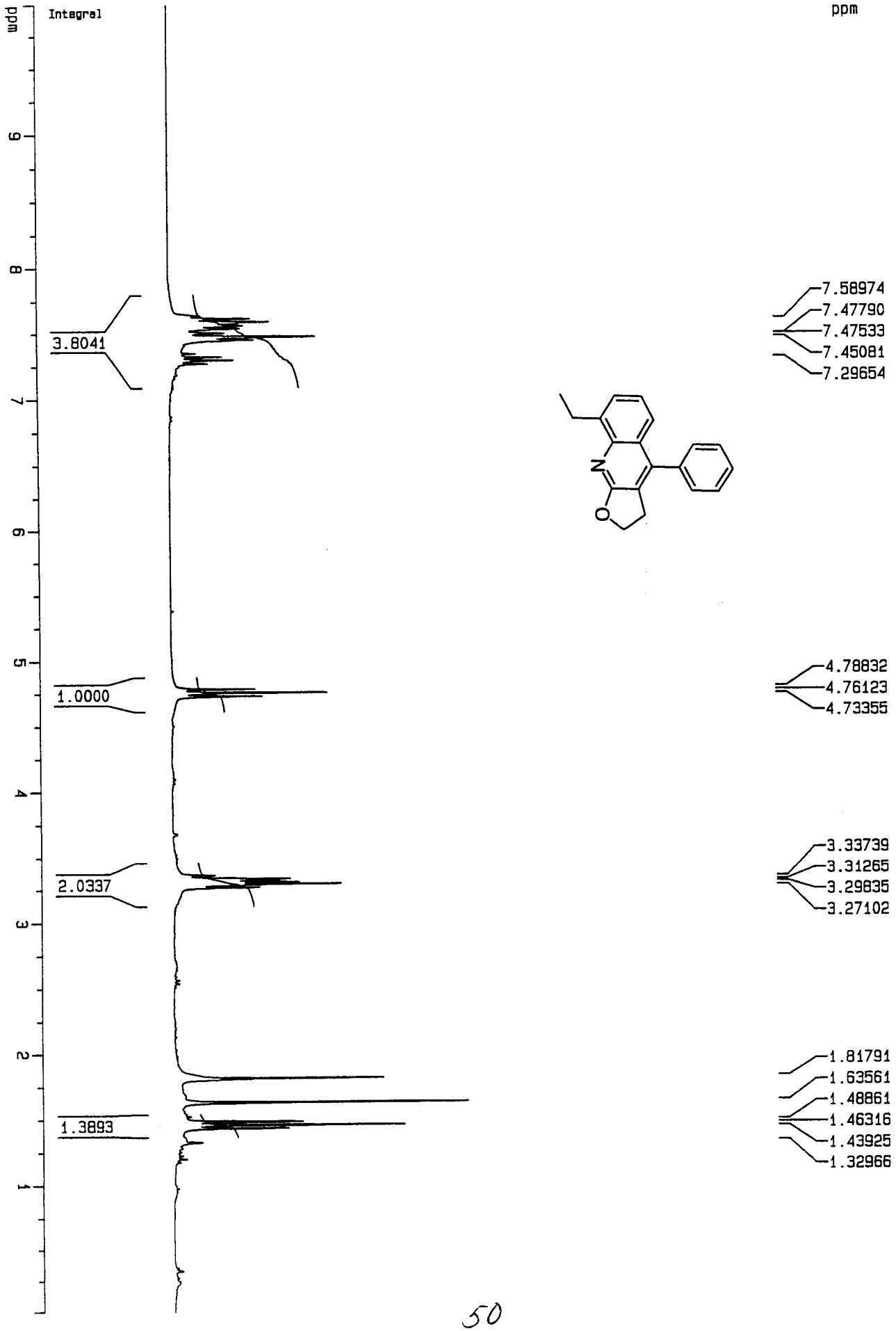
C-113


ppm
180
160
140
120
100
80
60
40
20
ppm
165.399
155.862
145.352
142.636
129.307
124.717
119.724
119.018
103.486
77.427
77.250
77.004
76.580
55.543
34.386
22.957
22.216
13.327
O
N
O
C-113

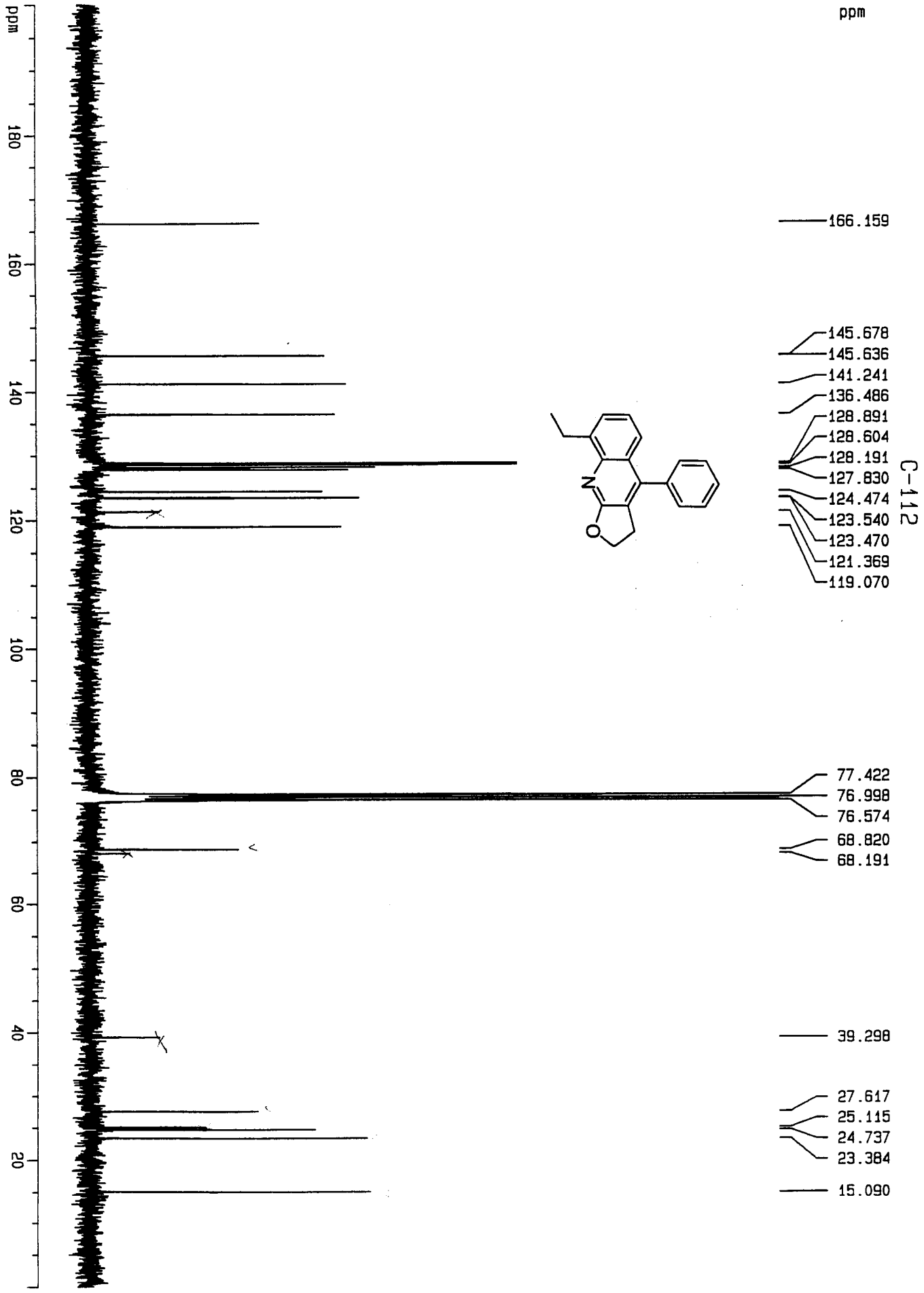

Integral
ppm
1.5798
1.0000
0.9337
1.9738
2.2029
2.7854
7.84435
7.81675
7.59762
7.57045
7.54748
7.54325
7.40483
7.37793
7.35375
7.35113
7.27010
4.72711
4.69990
4.67246
3.35358
3.32598
3.29894
2.54276
1.70386
0.14362
N
O

C-47


ppm
180
160
140
120
100
80
60
40
20
ppm
166.964
146.866
140.678
128.775
127.935
125.237
123.656
123.178
119.800
77.420
76.997
76.573
68.834
27.112
19.290
15.351
C-47, du13cC-47


Integral
ppm
3.8041
1.0000
2.0337
1.3893
7.58974
7.47790
7.47533
7.45081
7.29654
4.78832
4.76123
4.73355
3.33739
3.31265
3.29835
3.27102
1.81791
1.63561
1.48861
1.46316
1.43925
1.32966
N
O

C-112


ppm
180
160
140
120
100
80
60
40
20
ppm
166.159
145.678
145.636
141.241
136.486
128.891
128.604
128.191
127.830
124.474
123.540
123.470
121.369
119.070
77.422
76.998
76.574
68.820
68.191
39.298
27.617
25.115
24.737
23.384
15.090
C-112