

Supporting Information

Synthesis of Substituted Quinolines via Allylic Amination and Intramolecular Heck-Coupling

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General information:

All the reagents were commercial grade and purified according to the established procedures. Organic extracts were dried over anhydrous magnesium sulphate. Solvents were removed in a rotary evaporator under reduced pressure. Silica gel (60-120 mesh size) was used for the column chromatography. Reactions were monitored by TLC on silica gel 60 F₂₅₄ (0.25mm). NMR spectra were recorded in CDCl₃ with tetramethylsilane (TMS) as the internal standard for ¹H NMR (400 MHz) and for ¹³C NMR (100 MHz). IR spectra were recorded on JASCO 480-plus instrument. GC-MS analysis carried out on an Agilent GC-MS (7890A – 5975C VL MSD) system.

General Procedure for the Preparation of Aza Baylis-Hillman Adducts and Allyl Amines: To the solution of $\text{FeCl}_2 \cdot 4\text{H}_2\text{O}$ (10 mg, 0.05 mmol), Olefin (1.50 mmol) in dioxane (5 mL), was added the phenylhydroxylamine (0.5 mmol) solution slowly in dioxane (5 mL) via syringe pump over 4 hours at 40 °C (70 °C for styrenes). Reactions were allowed to continue for two more hours to get complete consumption of phenylhydroxylamine. After that the mixture was filtered through celite and the filtrate was concentrated to dryness. The crude product was purified over a short column of silica gel (hexane/ethylacetate eluents) to give the corresponding ABH adduct/Allylamine which was then directly analyzed by GC-MS, NMR and IR analysis.

General Procedure for the Intramolecular Heck-coupling of Aza Baylis-Hillman Adducts and Allyl Amines: ABH adduct/Allylamine (0.25 mmol), tetrabutyl ammoniumiodide (TBAI) (0.25 mmol) and $\text{Pd}(\text{OAc})_2$ (0.025 mmol) were combined in a round-bottom flask. DMF (4 mL) was added to the same flask and kept for heating on a sand bath at 90 °C for 6-12 hours while stirring. Once the reaction is completed (monitored by TLC and GC-MS), the mixture was filtered through celite and the filtrate was concentrated to dryness. The crude product was purified over a short column of silica gel (hexane/ethylacetate eluents) to isolate substituted quinolines which were then directly analyzed by GC-MS, NMR and IR analysis.

Spectral data:

TABLE 1:

Methyl 2-((2-iodophenylamino)methyl)acrylate (1a): ^1H NMR (400 MHz, CDCl_3): δ 3.79 (s, 3H), 4.09 (d, 2H, $J = 6.4$ Hz), 4.58 (brs, 1H), 5.72 (s, 1H), 6.28 (s, 1H), 6.41-6.47 (m, 2H), 7.15 (t, 1H, $J = 7.6$ Hz), 7.68 (d, 1H, $J = 7.6$ Hz); ^{13}C NMR (100 MHz, CDCl_3): δ 44.9, 52.2, 85.7, 111.2, 119.2, 126.1, 129.6, 136.7, 139.3, 146.6, 166.8; IR (KBr): 3399, 3065, 2996, 2949, 1716, 1636, 1590, 1507, 1453, 1321, 1267, 1154, 1084, 1004, 949, 816, 744 cm^{-1} .

Ethyl 3-(2-iodophenylamino)-2-methylenebutanoate (1b): Confirmed the products by comparing our previous data (Reported in our previous publication: S. Murru, A. A. Gallo, R. S. Srivastava, *J. Org. Chem.* 2012, **77**, 7119)

Benzyl 3-(2-iodophenylamino)-2-methylenebutanoate (1c): ^1H NMR (400 MHz, CDCl_3): δ 1.47 (d, 3H, $J = 6.4$ Hz), 4.45 (brs, 1H), 4.49 (t, 1H, $J = 6.4$ Hz), 5.25 (s, 2H), 5.73 (s, 1H), 6.24 (s, 1H), 6.37-6.44 (m, 2H), 7.12 (t, 1H, $J = 7.6$ Hz), 7.33-7.38 (m, 5H), 7.64 (d,

1H, $J = 8.0$ Hz; ^{13}C NMR (100 MHz, CDCl_3): δ 22.2, 50.3, 66.8, 85.7, 112.0, 119.1, 125.1, 128.3, 128.4, 128.8, 129.5, 135.9, 139.1, 141.9, 145.7, 166.3; IR (KBr): 3394, 3065, 2932, 1726, 1634, 1589, 1505, 1318, 1176, 1081, 819, 744 cm^{-1} .

Methyl 3-(2-iodophenylamino)-2-methylenepentanoate (1d): ^1H NMR (400 MHz, CDCl_3): δ 1.03 (t, 3H, $J = 7.2$ Hz), 1.65-1.71 (m, 1H), 1.86-1.91 (m, 1H), 3.80 (s, 3H), 4.29 (t, 1H, $J = 7.2$ Hz), 4.50 (d, 1H, $J = 7.2$ Hz), 5.68 (s, 1H), 6.22 (s, 1H), 6.37 – 6.43 (m, 2H), 7.13 (t, 1H, $J = 8.0$ Hz), 7.64 (d, 1H, $J = 7.6$ Hz); ^{13}C NMR (100 MHz, CDCl_3): δ 10.8, 28.7, 52.1, 56.2, 85.8, 111.8, 118.9, 125.6, 129.5, 139.1, 140.5, 146.0, 167.1; IR (KBr): 3398, 3063, 2992, 1718, 1635, 1592, 1437, 1370, 1266, 1004, 951, 849, 744 cm^{-1} .

4-(2-iodophenylamino)-3-methylenepentan-2-one (1e): ^1H NMR (400 MHz, CDCl_3): δ 2.03 (d, 3H, $J = 7.2$ Hz), 2.34 (s, 3H), 4.02 (s, 2H), 4.35 (brs, 1H), 6.43 (t, 1H, $J = 8.0$ Hz), 6.94 (t, 1H, $J = 7.6$ Hz), 7.20 (t, 1H, $J = 8.8$ Hz), 7.64 (d, 1H, $J = 7.6$ Hz); ^{13}C NMR (100 MHz, CDCl_3): δ 15.1, 25.6, 38.8, 85.8, 111.0, 118.9, 129.3, 139.1, 139.7, 142.3, 147.2, 199.0; IR (KBr): 3403, 3068, 2992, 1692, 1632, 1588, 1456, 1267, 1152, 1089, 952, 817, 742 cm^{-1} .

2-Iodo-N-(2-phenylallyl)benzenamine (1f): ^1H NMR (400 MHz, CDCl_3): δ 4.21 (d, 2H, $J = 4.8$ Hz), 4.51 (brs, 1H), 5.30 (s, 1H), 5.48 (s, 1H), 6.45 (t, 1H, $J = 7.6$ Hz), 6.57 (d, 1H, $J = 8.0$ Hz), 7.19 (t, 1H, $J = 7.2$ Hz), 7.31-7.38 (m, 3H), 7.44 (d, 2H, $J = 8.0$ Hz), 7.66 (d, 1H, $J = 8.0$ Hz); ^{13}C NMR (100 MHz, CDCl_3): δ 48.2, 85.5, 111.1, 113.6, 119.0, 126.3, 128.1, 128.7, 129.6, 139.1, 139.4, 144.1, 147.1; IR (KBr): 3400, 3055, 1590, 1506, 1453, 1321, 1004, 905, 778, 742, 705 cm^{-1} .

2-Iodo-N-(2-p-tolylallyl)benzenamine (1g): ^1H NMR (400 MHz, CDCl_3): δ 2.36 (s, 3H), 4.18 (d, 2H, $J = 5.6$ Hz), 4.50 (brs, 1H), 5.24 (s, 1H), 5.45 (s, 1H), 6.44 (t, 1H, $J = 8.0$ Hz), 6.55 (d, 1H, $J = 8.0$ Hz), 7.17 (m, 3H), 7.34 (d, 2H, $J = 8.0$ Hz), 7.65 (d, 1H, $J = 8.0$ Hz); ^{13}C NMR (100 MHz, CDCl_3): δ 21.3, 48.2, 85.5, 111.1, 112.8, 118.9, 126.1, 129.4, 129.6, 136.5, 138.0, 139.1, 143.8, 147.1; IR (KBr): 3399, 2918, 1590, 1506, 1453, 1321, 1073, 1004, 901, 822, 741 cm^{-1} .

N-(2-(4-chlorophenyl)allyl)-2-iodobenzenamine (1h): ^1H NMR (400 MHz, CDCl_3): δ 4.16 (d, 2H, $J = 6.0$ Hz), 4.45 (brs, 1H), 5.31 (s, 1H), 5.46 (s, 1H), 6.45 (t, 1H, $J = 7.6$ Hz), 6.54 (d, 1H, $J = 8.4$ Hz), 7.18 (t, 1H, $J = 6.8$ Hz), 7.31 (d, 2H, $J = 8.8$ Hz), 7.35 (d, 2H, $J = 8.8$

Hz), 7.64 (d, 1H, $J = 8.0$ Hz); ^{13}C NMR (100 MHz, CDCl_3): δ 48.2, 85.6, 111.1, 114.3, 119.2, 127.6, 128.9, 129.6, 134.0, 137.8, 139.2, 143.1, 146.9; IR (KBr): 3400, 3065, 2917, 1632, 1590, 1492, 1321, 1093, 1005, 833, 741 cm^{-1} .

N-(2-(4-fluorophenyl)allyl)-2-iodobenzenamine (1i): ^1H NMR (400 MHz, CDCl_3): δ 4.15 (d, 2H, $J = 5.6$ Hz), 4.46 (brs, 1H), 5.28 (s, 1H), 5.42 (s, 1H), 6.45 (t, 1H, $J = 8.0$ Hz), 6.55 (d, 1H, $J = 8.0$ Hz), 7.01-7.05 (m, 2H), 7.18 (t, 1H, $J = 8.0$ Hz), 7.37 – 7.41 (m, 2H), 7.64 (d, 1H, $J = 8.0$ Hz); ^{13}C NMR (100 MHz, CDCl_3): δ 48.4, 85.6, 111.1, 113.7, 115.5, 115.7, 119.1, 127.9, 128.0, 129.6, 135.3, 135.4, 139.2, 143.1, 146.9, 161.5, 163.9; IR (KBr): 3401, 3065, 2921, 2856, 1631, 1590, 1507, 1453, 1321, 1233, 1161, 1005, 906, 837, 742, 647 cm^{-1} .

2-Iodo-N-(4-methyl-2-methylenepent-3-enyl)benzenamine (1j): ^1H NMR (400 MHz, CDCl_3): δ 1.78 (s, 3H), 1.81 (s, 3H), 3.76 (d, 2H, $J = 5.6$ Hz), 4.40 (brs, 1H), 4.94 (s, 1H), 5.19 (s, 1H), 5.63 (s, 1H), 6.42 (t, 1H, $J = 7.6$ Hz), 6.50 (d, 1H, $J = 8.4$ Hz), 7.17 (t, 1H, $J = 7.2$ Hz), 7.64 (d, 1H, $J = 7.6$ Hz); ^{13}C NMR (100 MHz, CDCl_3): δ 19.8, 26.9, 49.9, 85.5, 111.2, 113.8, 118.8, 123.6, 129.5, 137.7, 139.1, 142.4, 147.3; IR (KBr): 3399, 3066, 2967, 2926, 1590, 1505, 1453, 1318, 1004, 900, 741 cm^{-1} .

2-iodo-N-((E)-7-methyl-3-methyleneocta-4,6-dien-2-yl)benzenamine (1k): ^1H NMR (400 MHz, CDCl_3): δ 1.49 (d, 3H, $J = 6.8$ Hz), 1.80 (s, 3H), 1.83 (s, 3H), 4.21 (brs, 1H), 4.35 (brs, 1H), 5.03 (s, 1H), 5.09 (s, 1H), 5.88 (d, 1H, $J = 10.8$ Hz), 6.15 (d, 1H, $J = 15.6$ Hz), 6.34 – 6.41 (m, 2H), 6.59 (dd, 1H, $J_1 = 15.6$ Hz, $J_2 = 10.8$ Hz), 7.10 (t, 1H, $J = 7.2$ Hz), 7.62 (d, 1H, $J = 7.6$ Hz).; ^{13}C NMR (100 MHz, CDCl_3): δ 18.8, 22.6, 26.4, 50.9, 85.4, 111.9, 113.1, 118.7, 125.3, 125.7, 129.4, 129.9, 137.0, 139.0, 146.3, 147.6; IR (KBr): 2988, 2956, 2931, 1586, 1507, 1460, 1343, 1011 cm^{-1} .

TABLE 2:

Ethyl 3-(2-bromophenylamino)-2-methylenebutanoate (2b): Confirmed the products by comparing our previous data (Reported in our previous publication: S. Murru, A. A. Gallo, R. S. Srivastava, *J. Org. Chem.* 2012, **77**, 7119)

Ethyl 3-(2,5-dibromophenylamino)-2-methylenebutanoate (3b): ^1H NMR (400 MHz, CDCl_3): δ 1.34 (t, 3H, $J = 7.6$ Hz), 1.46 (d, 3H, $J = 6.8$ Hz), 4.27 (q, 2H, $J = 7.6$ Hz), 4.41 (t, 1H, $J = 6.8$ Hz), 4.66 (d, 1H, $J = 7.2$ Hz), 5.69 (s, 1H), 6.22 (s, 1H), 6.56 (s, 1H), 6.67 (d,

1H, $J = 8.4$ Hz), 7.17 (d, 1H, $J = 8.4$ Hz); ^{13}C NMR (100 MHz, CDCl_3): δ 14.4, 22.1, 50.2, 61.2, 108.4, 115.2, 120.9, 122.4, 124.8, 133.5, 141.6, 144.7, 166.3; IR (KBr): 3415, 3073, 2982, 1712, 1598, 1505, 1285, 1106, 743 cm^{-1} .

Ethyl 3-(2-bromo-5-(trifluoromethyl)phenylamino)-2-methylenebutanoate (4b): ^1H NMR (400 MHz, CDCl_3): δ 1.12 (t, 3H, $J = 7.2$ Hz), 1.40 (d, 3H, $J = 6.8$ Hz), 3.94 (q, 2H, $J = 7.2$ Hz), 4.20 (t, 1H, $J = 6.4$ Hz), 4.75 (q, 1H, $J = 7.2$ Hz), 5.77 (s, 1H), 6.40 (s, 1H), 7.16 (d, 1H, $J = 8.4$ Hz), 7.59 (d, 1H, $J = 8.4$ Hz), 7.65 (s, 1H); ^{13}C NMR (100 MHz, CDCl_3): δ 14.1, 23.3, 59.3, 61.1, 119.7, 122.5, 128.6, 129.0, 131.1, 133.7, 138.5, 150.5, 167.1; IR (KBr): 3408, 3070, 2981, 2933, 1715, 1594, 1323, 1282, 1107, 741 cm^{-1} .

Ethyl 3-(2-iodo-4-methylphenylamino)-2-methylenebutanoate (5b): ^1H NMR (400 MHz, CDCl_3): δ 1.31 (t, 3H, $J = 6.8$ Hz), 1.43 (d, 3H, $J = 6.8$ Hz), 2.16 (s, 3H), 4.24 (q, 2H, $J = 7.2$ Hz), 4.29 (brs, 1H), 4.42 (t, 1H, $J = 6.8$ Hz), 5.66 (s, 1H), 6.16 (s, 1H), 6.28 (d, 1H, $J = 8.8$ Hz), 6.93 (dd, 1H, $J_1 = 8.4$ Hz, $J_2 = 1.6$ Hz), 7.47 (t, 1H, $J = 1.6$ Hz); ^{13}C NMR (100 MHz, CDCl_3): δ 14.4, 19.9, 22.2, 50.5, 61.0, 85.7, 111.9, 124.4, 128.4, 130.1, 139.4, 142.3, 143.7, 166.7; IR (KBr): 3389, 3062, 2968, 1715, 1589, 1506, 1266, 1099, 1003, 742 cm^{-1} .

Ethyl 3-(2-iodo-3,5-dimethylphenylamino)-2-methylenebutanoate (6b): ^1H NMR (400 MHz, CDCl_3): δ 1.31 (t, 3H, $J = 7.2$ Hz), 1.44 (d, 3H, $J = 6.4$ Hz), 2.17 (s, 3H), 2.36 (s, 3H), 4.25 (q, 2H, $J = 7.2$ Hz), 4.44 (t, 1H, $J = 6.0$ Hz), 4.56 (d, 1H, $J = 6.8$ Hz), 5.67 (s, 1H), 6.01 (s, 1H), 6.17 (s, 1H), 6.45 (s, 1H); ^{13}C NMR (100 MHz, CDCl_3): δ 14.4, 21.4, 22.3, 29.6, 50.5, 60.9, 89.1, 110.0, 120.2, 124.3, 138.6, 141.9, 142.3, 145.8, 166.6; IR (KBr): 3386, 3060, 2968, 1715, 1586, 1265, 1098, 743 cm^{-1} .

Ethyl 3-(2-bromopyridin-3-ylamino)-2-methylenebutanoate (7b): ^1H NMR (400 MHz, CDCl_3): δ 1.32 (m, 3H), 1.50 (d, 3H, $J = 6.4$ Hz), 4.27 (q, 2H, $J = 7.2$ Hz), 4.42 (t, 1H, $J = 6.8$ Hz), 4.71 (d, 1H, $J = 6.4$ Hz), 5.70 (s, 1H), 6.22 (s, 1H), 6.67 (d, 1H, $J = 8.0$ Hz), 7.05 (dd, 1H, $J^1 = 8.0$ Hz, $J^2 = 4.4$ Hz), 7.68 (d, 1H, $J = 4.4$ Hz); ^{13}C NMR (100 MHz, CDCl_3): δ 14.2, 21.9, 49.7, 61.0, 118.5, 123.6, 124.5, 130.3, 137.4, 140.7, 141.2, 166.0; IR (KBr): 3408, 3061, 2980, 2934, 1713, 1579, 1487, 1378, 1284, 1264, 1181, 1133, 1106, 1042, 956, 789 cm^{-1} .

TABLE 3:

Methyl quinoline-3-carboxylate (1a'): ^1H NMR (400 MHz, CDCl_3): δ 4.02 (s, 3H), 7.63 (t, 1H, $J = 7.2$ Hz), 7.83 (t, 1H, $J = 7.2$ Hz), 7.94 (d, 1H, $J = 8.0$ Hz), 8.16 (d, 1H, $J = 8.0$ Hz), 8.86 (s, 1H), 9.45 (s, 1H); ^{13}C NMR (100 MHz, CDCl_3): δ 52.5, 123.0, 126.8, 127.5, 129.1, 129.5, 131.9, 138.8, 149.8, 150.0, 165.9; IR (KBr): 2954, 2915, 1726, 1621, 1564, 1440, 1272, 1128, 1054, 805, 762 cm^{-1} .

Ethyl 2-methylquinoline-3-carboxylate (1b', 2b'): ^1H NMR (400 MHz, CDCl_3): δ 1.45 (t, 3H, $J = 7.2$ Hz), 3.00 (s, 3H), 4.44 (q, 2H, $J = 7.2$ Hz), 7.26 (s, 1H), 7.54 (t, 1H, $J = 6.8$ Hz), 7.76 – 7.80 (m, 1H), 7.87 (d, 1H, $J = 8.0$ Hz), 8.04 (d, 1H, $J = 8.0$ Hz), 8.74 (s, 1H); ^{13}C NMR (100 MHz, CDCl_3): δ 14.3, 25.7, 61.4, 123.9, 125.7, 126.5, 128.4, 128.5, 131.7, 139.9, 148.6, 158.5, 166.5; IR (KBr): 3062, 2963, 2926, 2852, 1725, 1620, 1562, 1423, 1258, 1199, 1062 cm^{-1} .

Benzyl 2-methylquinoline-3-carboxylate (1c'): ^1H NMR (400 MHz, CDCl_3): δ 2.98 (s, 3H), 5.40 (s, 2H), 7.38 – 7.45 (m, 3H), 7.49 – 7.55 (m, 3H), 7.78 (t, 1H, $J_1 = 7.6$ Hz, $J_2 = 1.6$ Hz), 7.85 (d, 1H, $J = 8.4$ Hz), 8.04 (d, 1H, $J = 8.4$ Hz), 8.77 (s, 1H); ^{13}C NMR (100 MHz, CDCl_3): δ 26.0, 67.4, 123.7, 125.9, 126.7, 128.6, 128.7, 128.75, 128.9, 131.2, 132.0, 135.9, 140.3, 148.9, 158.8, 166.5; IR (KBr): 3062, 2972, 2928, 1724, 1619, 1563, 1455, 1422, 1240, 1126, 1055, 788, 750 cm^{-1} .

Methyl 2-ethylquinoline-3-carboxylate (1d'): ^1H NMR (400 MHz, CDCl_3): δ 1.37 (t, 3H, $J = 7.2$ Hz), 3.34 (q, 2H, $J = 7.2$ Hz), 3.97 (s, 3H), 7.52 (t, 1H, $J = 7.6$ Hz), 7.77 (t, 1H, $J = 7.6$ Hz), 7.82 (d, 1H, $J = 7.6$ Hz), 8.00 (d, 1H, $J = 8.4$ Hz), 8.70 (s, 1H); ^{13}C NMR (100 MHz, CDCl_3): δ 14.1, 31.2, 52.6, 123.5, 125.8, 126.7, 128.6, 128.9, 131.8, 140.2, 148.9, 163.2, 167.2; IR (KBr): 2951, 2919, 2874, 1725, 1620, 1563, 1437, 1273, 1252, 1201, 1127, 1069, 803, 754 cm^{-1} .

1-(2-Methylquinolin-3-yl)ethanone (1e'): ^1H NMR (400 MHz, CDCl_3): δ 2.69 (s, 3H), 2.88 (s, 3H), 7.52 (t, 1H, $J = 7.6$ Hz), 7.73 – 7.78 (m, 1H), 7.82 (d, 1H, $J = 8.0$ Hz), 8.00 (d, 1H, $J = 8.4$ Hz), 8.44 (s, 1H); ^{13}C NMR (100 MHz, CDCl_3): δ 25.9, 29.4, 125.8, 126.9, 128.5, 128.8, 131.3, 131.9, 138.4, 148.5, 157.8, 200.1; IR (KBr): 2965, 2920, 1680, 1618, 1561, 1418, 1253, 1196, 1029, 926, 862, 781, 750, 691 cm^{-1} .

3-Phenylquinoline (1f'): ^1H NMR (400 MHz, CDCl_3): δ 7.44 (t, 1H, $J = 7.2$ Hz), 7.51 – 7.61 (m, 3H), 7.71 – 7.74 (m, 3H), 7.89 (d, 1H, $J = 8.0$ Hz), 8.15 (d, 1H, $J = 8.8$ Hz), 8.31 (d, 1H, $J = 2.0$ Hz), 9.19 (d, 1H, $J = 2.0$ Hz); ^{13}C NMR (100 MHz, CDCl_3): δ 126.4, 127.2, 127.7, 128.2, 128.3, 129.0, 129.4, 129.45, 129.6, 131.1, 133.5, 138.1, 150.2; IR (KBr): 2963, 2926, 1725, 1493, 1459, 1262, 1074, 1025, 902, 787, 761, 697 cm^{-1} .

3-p-Tolylquinoline (1g'): ^1H NMR (400 MHz, CDCl_3): δ 2.43 (s, 3H), 7.04 (d, 1H, $J = 8.0$ Hz), 7.08 (d, 1H, $J = 8.0$ Hz), 7.25 (s, 1H), 7.32 (d, 2H, $J = 7.6$ Hz), 7.56 (t, 1H, $J = 7.6$ Hz), 7.61 (d, 2H, $J = 7.6$ Hz), 7.70 (t, 1H, $J = 7.6$ Hz), 7.87 (d, 1H, $J = 8.4$ Hz), 8.11 (d, 1H, $J = 6.8$ Hz), 8.28 (s, 1H); ^{13}C NMR (100 MHz, CDCl_3): δ 21.4, 121.6, 122.8, 126.3, 126.8, 127.1, 127.5, 128.1, 129.4, 129.7, 130.1, 133.0, 138.3, 150.2; IR (KBr): 3015, 2969, 2921, 1667, 1540, 1456, 1261, 815, 751 cm^{-1} .

3-(4-Chlorophenyl)quinoline (1h'): ^1H NMR (400 MHz, CDCl_3): δ 7.49 (d, 2H, $J = 8.8$ Hz), 7.58 (t, 1H, $J = 7.6$ Hz), 7.62 (d, 2H, $J = 8.8$ Hz), 7.70 – 7.75 (m, 1H), 7.87 (d, 1H, $J = 8.4$ Hz), 8.13 (d, 1H, $J = 8.4$ Hz), 8.27 (s, 1H), 9.12 (s, 1H); ^{13}C NMR (100 MHz, CDCl_3): δ 127.5, 128.1, 128.2, 128.9, 129.4, 129.6, 129.9, 132.9, 133.5, 134.6, 136.5, 147.5, 149.6; IR (KBr): 3052, 2925, 1604, 1512, 1231, 1161, 833, 752 cm^{-1} .

3-(4-Fluorophenyl)quinoline (1i'): ^1H NMR (400 MHz, CDCl_3): δ 7.20 – 7.26 (m, 2H), 7.59 (t, 1H, $J = 7.6$ Hz), 7.66 – 7.69 (m, 2H), 7.33 (t, 1H, $J = 7.6$ Hz), 7.88 (d, 1H, $J = 8.0$ Hz), 8.14 (d, 1H, $J = 8.0$ Hz), 8.27 (s, 1H), 9.14 (s, 1H); ^{13}C NMR (100 MHz, CDCl_3): δ 116.3, 116.5, 127.3, 128.0, 128.1, 128.2, 128.3, 128.4, 128.5, 128.7, 134.3, 147.5, 149.9, 161.9, 164.4; IR (KBr): 3048, 2925, 1604, 1512, 1341, 1231, 1161, 954, 910, 833, 785, 752 cm^{-1} .

1,2-dihydro-3-(2-methylprop-1-enyl)quinoline (1J'): ^1H NMR (400 MHz, CDCl_3): δ 1.75 (s, 3H), 1.77 (s, 3H), 3.46 (d, 2H, $J = 7.2$ Hz), 5.46 (t, 1H, $J = 2.0$ Hz), 6.96 (s, 1H), 7.12 (t, 1H, $J = 6.8$ Hz), 7.18 (t, 1H, $J = 6.8$ Hz), 7.35 (d, 1H, $J = 8.0$ Hz), 7.59 (d, 1H, $J = 8.0$ Hz), 7.92 (brs, 1H); ^{13}C NMR (100 MHz, CDCl_3): δ 24.1, 25.7, 68.1, 111.0, 116.2, 119.0, 119.1, 121.1, 121.9, 123.0, 128.8, 131.9, 136.5; IR (KBr): 3414, 2962, 2925, 1716, 1455, 1262, 1079, 789, 740 cm^{-1} .

2-methyl-3-((E)-4-methylpenta-1,3-dienyl)quinoline (1k'): ^1H NMR (400 MHz, CDCl_3): δ 1.89 (s, 3H), 1.92 (s, 3H), 2.76 (s, 3H), 6.15 (d, 1H, $J = 8.4$ Hz), 6.68 (d, 1H, $J = 15.2$ Hz), 7.02 (d, 1H, $J_1 = 15.2$ Hz, $J_2 = 11.2$ Hz), 7.46 (t, 1H, $J = 7.6$ Hz), 7.61 (t, 1H, $J = 7.6$ Hz), 7.77 (d, 1H, $J = 7.6$ Hz), 7.97 (d, 1H, $J = 8.4$ Hz), 8.15 (s, 1H); ^{13}C NMR (100 MHz, CDCl_3): δ 24.1, 26.5, 29.9, 125.5, 125.7, 126.1, 127.6, 127.65, 128.5, 129.0, 129.4, 130.9, 131.5, 138.5, 146.9, 157.6; IR (KBr): 2961, 2924, 2854, 1725, 1685, 1617, 1490, 1461, 1377, 1261, 1101, 799, 752 cm^{-1} .

Ethyl 7-bromo-2-methylquinoline-3-carboxylate (3b'): ^1H NMR (400 MHz, CDCl_3): δ 1.46 (t, 3H, $J = 7.2$ Hz), 2.98 (s, 3H), 4.45 (q, 2H, $J = 7.2$ Hz), 7.64 (d, 1H, $J = 8.8$ Hz), 7.73 (d, 1H, $J = 8.8$ Hz), 8.24 (s, 1H), 8.70 (s, 1H); ^{13}C NMR (100 MHz, CDCl_3): δ 14.5, 25.9, 61.7, 124.5, 124.6, 126.3, 129.8, 130.4, 131.3, 139.8, 149.3, 159.9, 166.5; IR (KBr): 2963, 2916, 1727, 1614, 1477, 1262, 1177, 1062, 803 cm^{-1} .

Ethyl 7-(trifluoromethyl)-2-methylquinoline-3-carboxylate (4b'): ^1H NMR (400 MHz, CDCl_3): δ 1.45 (t, 3H, $J = 7.2$ Hz), 2.98 (s, 3H), 4.44 (q, 2H, $J = 7.2$ Hz), 7.67 (d, 1H, $J = 8.4$ Hz), 7.95 (d, 1H, $J = 8.4$ Hz), 8.32 (s, 1H), 8.72 (s, 1H); ^{13}C NMR (100 MHz, CDCl_3): δ 14.4, 25.8, 61.9, 122.2, 122.3, 122.35, 126.0, 126.6, 126.65, 127.4, 129.7, 139.5, 147.7, 160.1, 166.2; IR (KBr): 2982, 2934, 2875, 1730, 1603, 1563, 1447, 1431, 1347, 1324, 1283, 1191, 1130, 1058, 934, 819 cm^{-1} .

Ethyl 2,6-dimethylquinoline-3-carboxylate (5b'): ^1H NMR (400 MHz, CDCl_3): δ 1.42 (t, 3H, $J = 7.2$ Hz), 2.51 (s, 3H), 2.94 (s, 3H), 4.41 (q, 2H, $J = 7.2$ Hz), 7.58 (d, 1H, $J = 8.0$ Hz), 7.59 (s, 1H), 7.90 (d, 1H, $J = 8.4$ Hz), 8.62 (s, 1H); ^{13}C NMR (100 MHz, CDCl_3): δ 14.5, 21.7, 25.8, 61.5, 126.0, 127.4, 128.3, 129.5, 134.1, 136.6, 139.5, 147.4, 157.7, 166.9; IR (KBr): 2977, 2915, 1720, 1594, 1566, 1277, 1239, 1215, 1069, 828, 778 cm^{-1} .

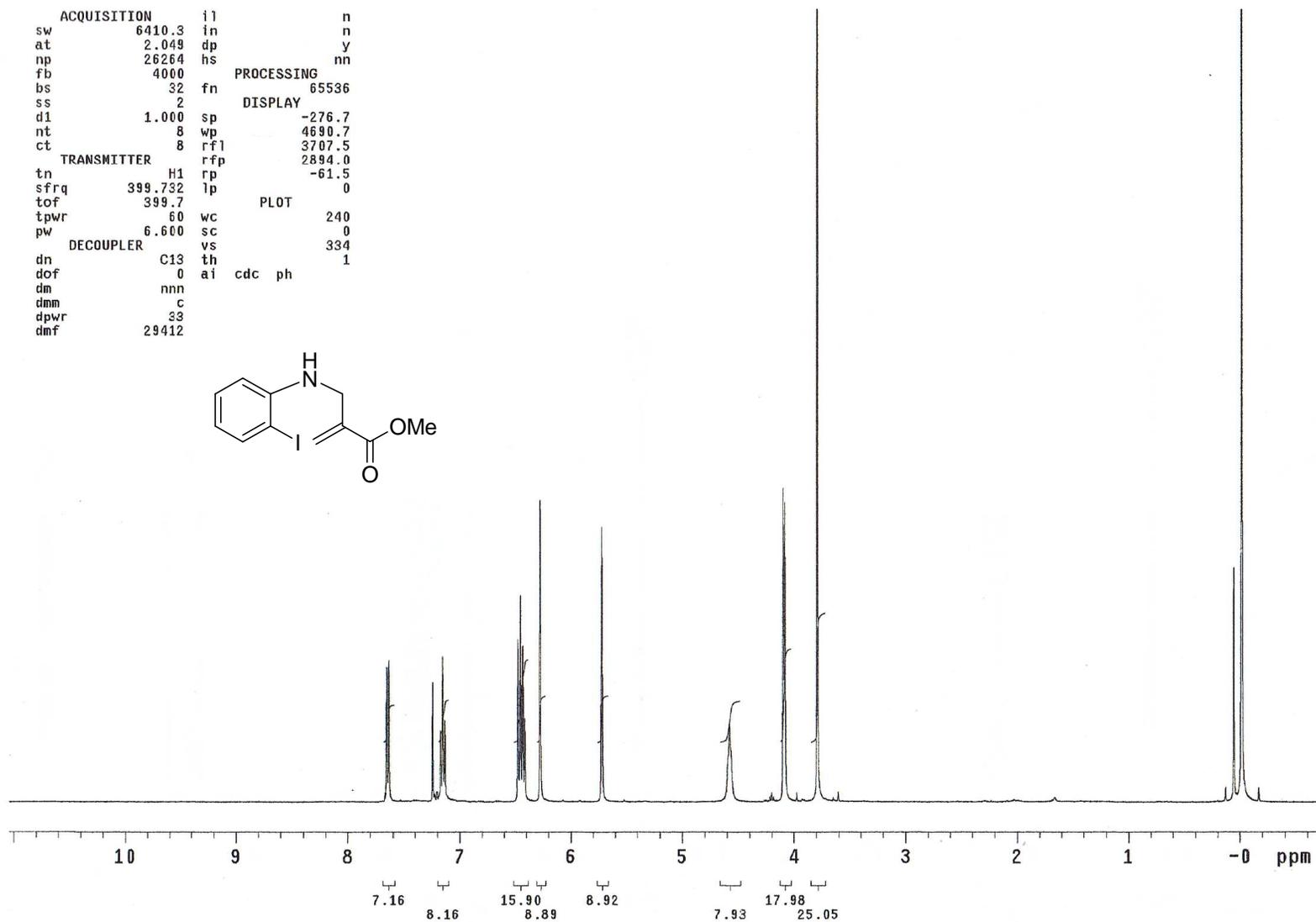
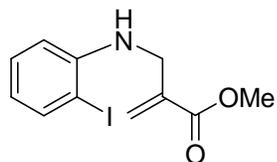
Ethyl 2,5,7-trimethylquinoline-3-carboxylate (6b'): ^1H NMR (400 MHz, CDCl_3): δ 1.45 (t, 3H, $J = 7.2$ Hz), 2.50 (s, 3H), 2.67 (s, 3H), 2.96 (s, 3H), 4.43 (q, 2H, $J = 7.2$ Hz), 7.18 (s, 1H), 7.65 (s, 1H), 8.82 (s, 1H); ^{13}C NMR (100 MHz, CDCl_3): δ 14.6, 18.6, 22.2, 25.9, 61.5, 122.7, 123.5, 126.0, 129.5, 135.4, 136.4, 142.2, 149.5, 158.1, 167.2; IR (KBr): 2978, 2927, 1720, 1620, 1600, 1566, 1444, 1380, 1269, 1226, 1086, 1056, 857, 778 cm^{-1} .

Ethyl 2-methyl-1,5-naphthyridine-3-carboxylate (7b'): ^1H NMR (400 MHz, CDCl_3): δ 1.41 (t, 3H, $J = 7.2$ Hz), 2.98 (s, 3H), 4.42 (q, 2H, $J = 7.2$ Hz), 7.64 (dd, 1H, $J^1 = 8.4$ Hz, $J^2 = 4.0$ Hz), 8.29 (d, 1H, $J = 8.4$ Hz), 8.89 (s, 1H), 8.95 (d, 1H, $J = 3.6$ Hz); ^{13}C NMR (100 MHz, CDCl_3): δ 14.4, 25.6, 61.9, 126.1, 127.4, 136.5, 140.6, 142.1, 144.6, 151.5, 159.7, 166.2; IR (KBr): 2982, 2928, 1725, 1588, 1472, 1263, 1204, 1125, 1065, 934, 832, 784 cm^{-1} .

Spectra

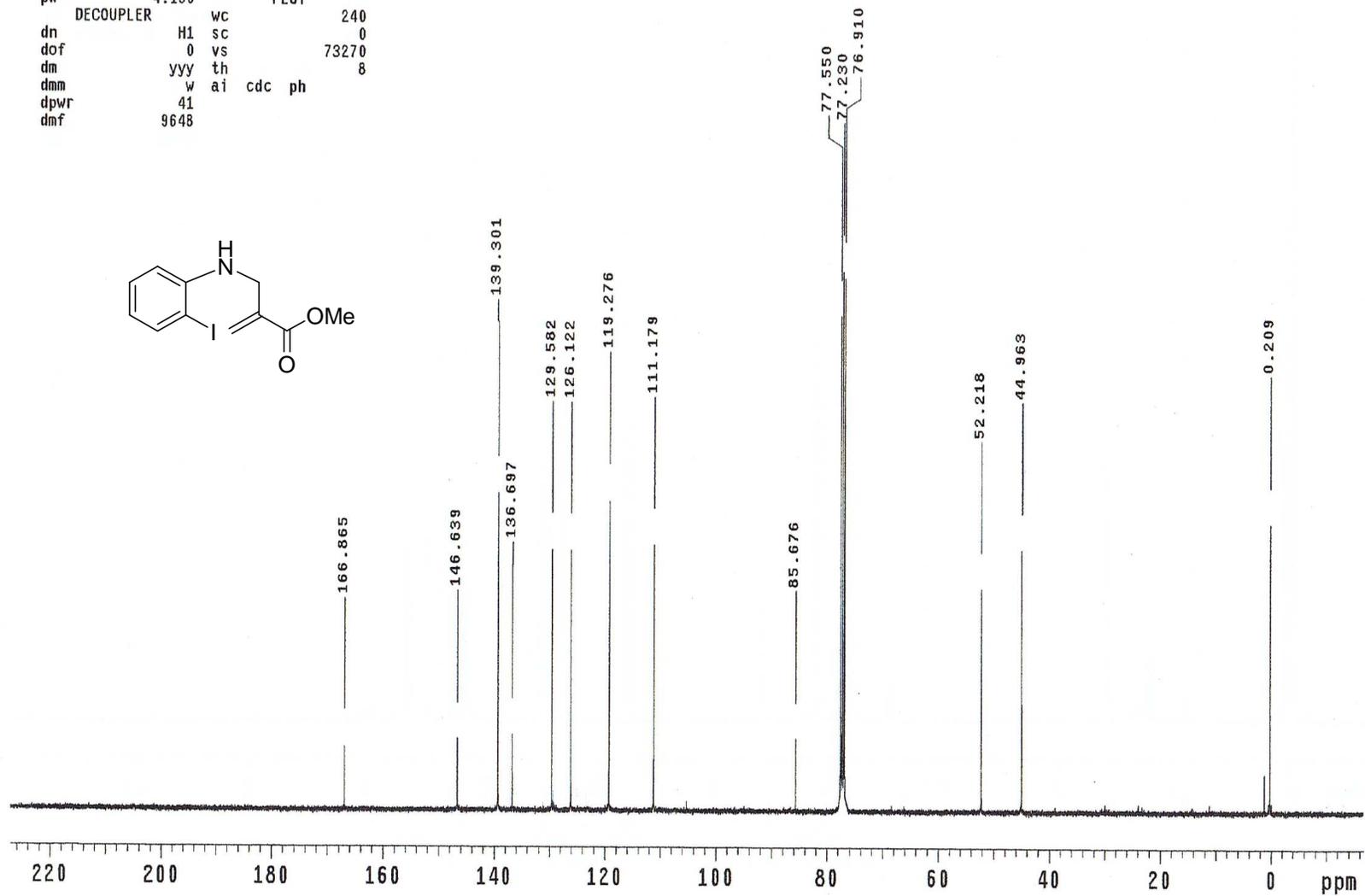
Methyl 2-((2-iodophenylamino)methyl)acrylate (1a): ^1H NMR (400 MHz, CDCl_3)

```
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sw      6410.3   in          n
at      2.049   dp          y
np      26264   hs          nn
fb      4000
bs      32      fn          65536
ss      2
d1      1.000   sp          -276.7
nt      8       wp          4690.7
ct      8       rfl         3707.5
          rfp         2894.0
          rfp         -61.5
          lp          0
TRANSMITTER      H1
tn      399.732  sfrq         399.7
          399.7
tpwr    60      wc          240
pw      6.600   sc          0
          vs          334
DECOUPLER      C13   th          1
          ai   cdc ph
dn      0
dof     nnn
dm      c
dmm     33
dpwr    29412
dmf
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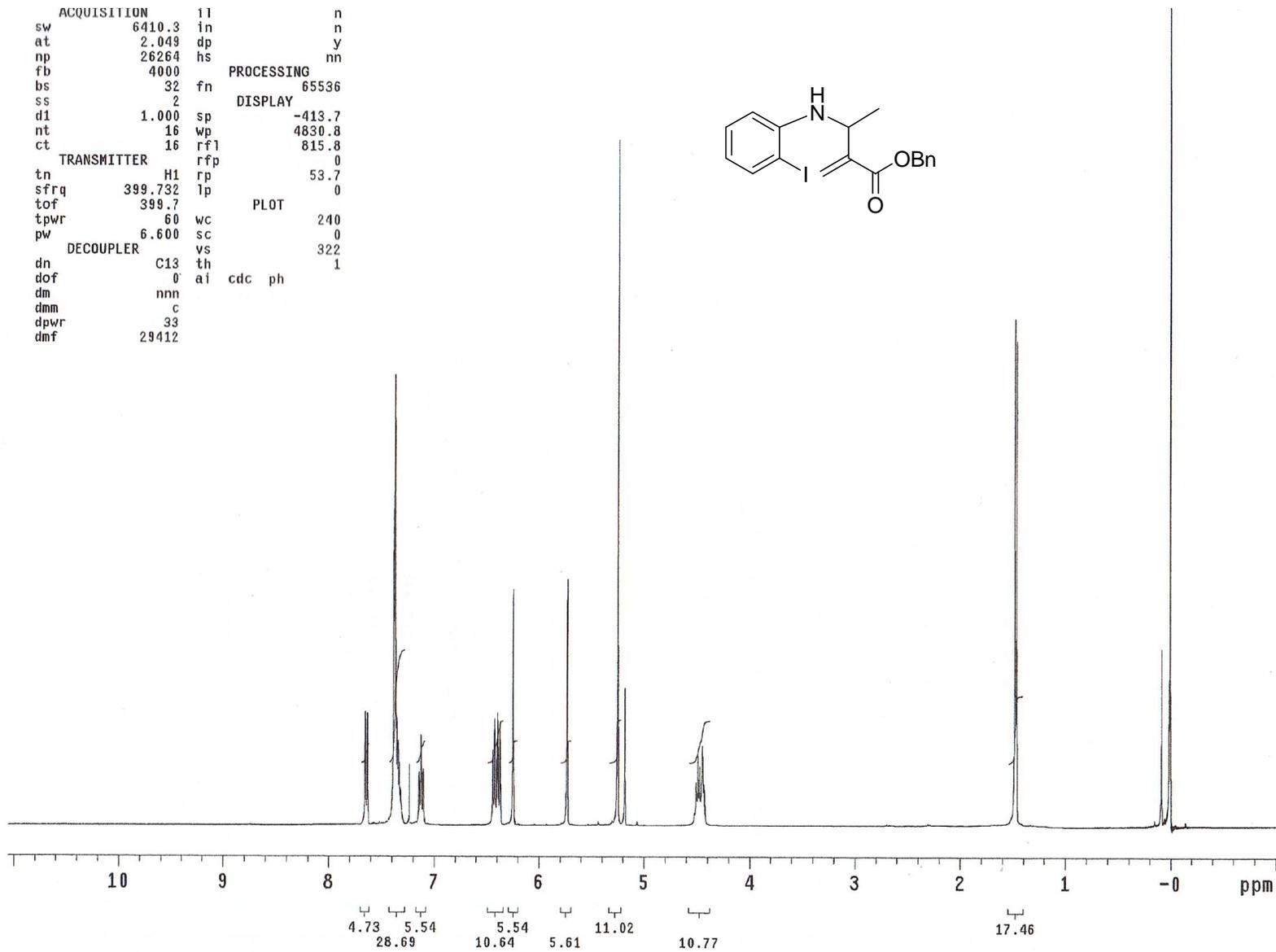
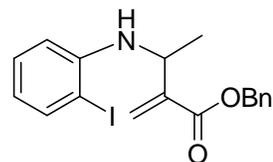
Methyl 2-((2-iodophenylamino)methyl)acrylate (1a): ^1H NMR (400 MHz, CDCl_3)

| | | |
|-------------|---------|------------|
| TRANSMITTER | wp | 24509.1 |
| tn | C13 | rf1 9442.5 |
| sfrq | 100.523 | rff 7762.6 |
| tof | 1027.9 | rp 82.4 |
| tpwr | 55 | lp 0 |
| pw | 4.150 | PLOT |
| DECOUPLER | wc | 240 |
| dn | H1 | sc 0 |
| dof | 0 | vs 73270 |
| dm | yyy | th 8 |
| dmm | w | ai cdc ph |
| dpwr | 41 | |
| dmf | 9648 | |

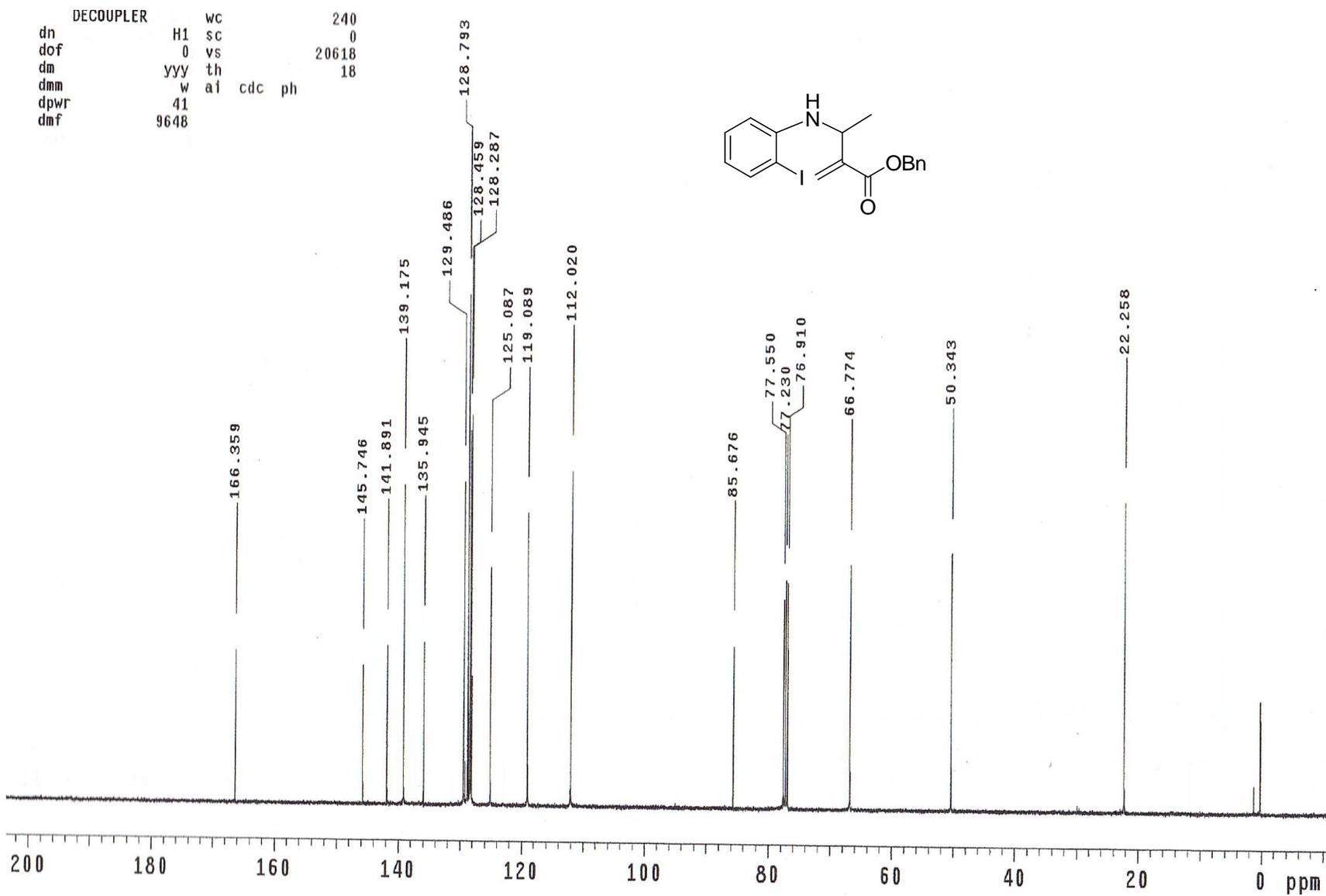


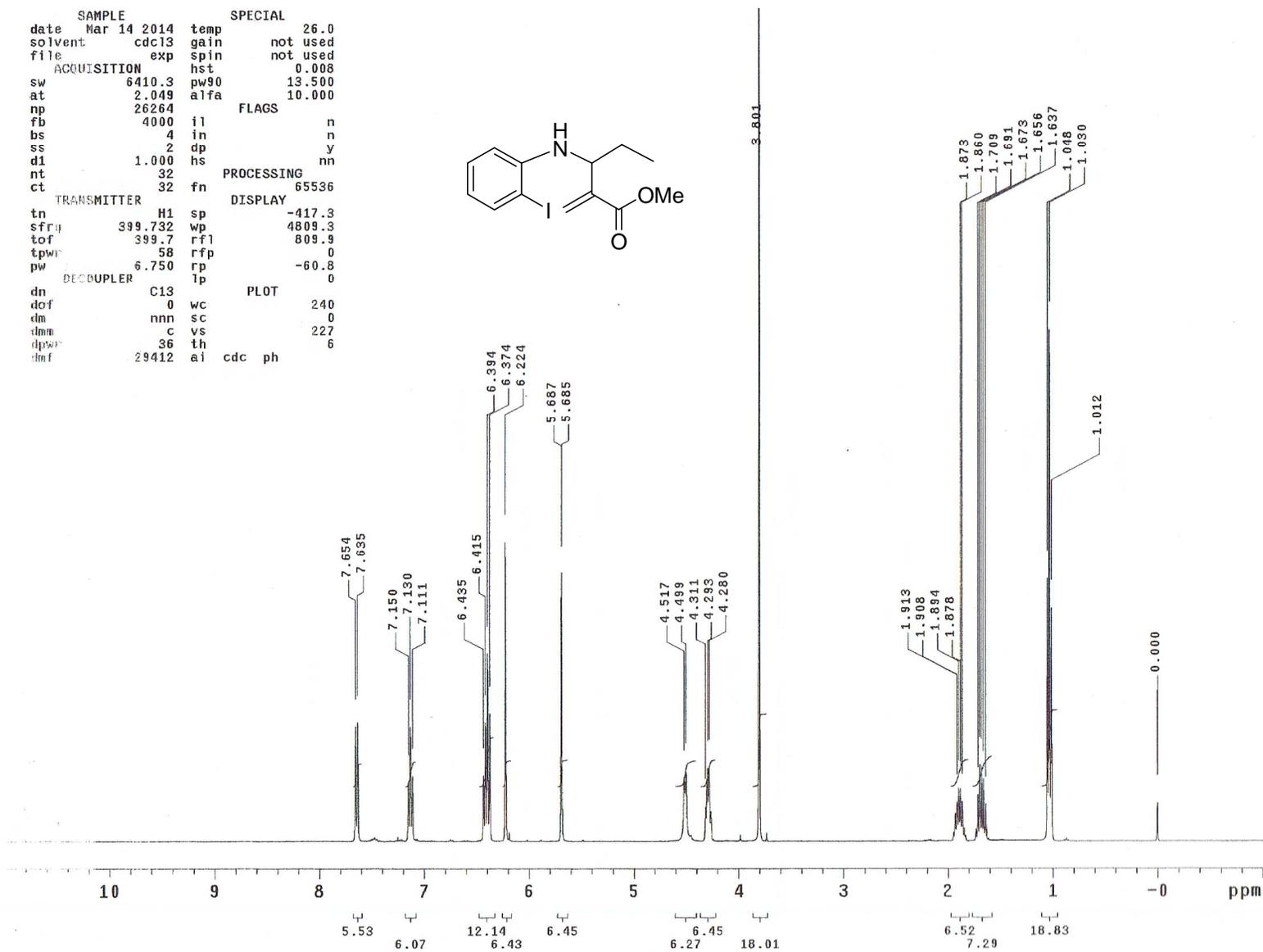
Benzyl 3-(2-iodophenylamino)-2-methylenebutanoate (1c): ^1H NMR (400 MHz, CDCl_3)

```
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sw 6410.3 in n
at 2.049 dp y
np 26264 hs nn
fb 4000
bs 32 fn PROCESSING 65536
ss 2 DISPLAY
d1 1.000 sp -413.7
nt 16 wp 4830.8
ct 16 rfl 815.8
TRANSMITTER rfp 0
tn H1 rp 53.7
sfrq 399.732 lp 0
tof 399.7 PLOT
tpwr 60 wc 240
pw 6.600 sc 0
DECOUPLER vs 322
dn C13 th 1
dof 0 ai cdc ph
dm nnn
dmm c
dpwr 33
dmf 29412
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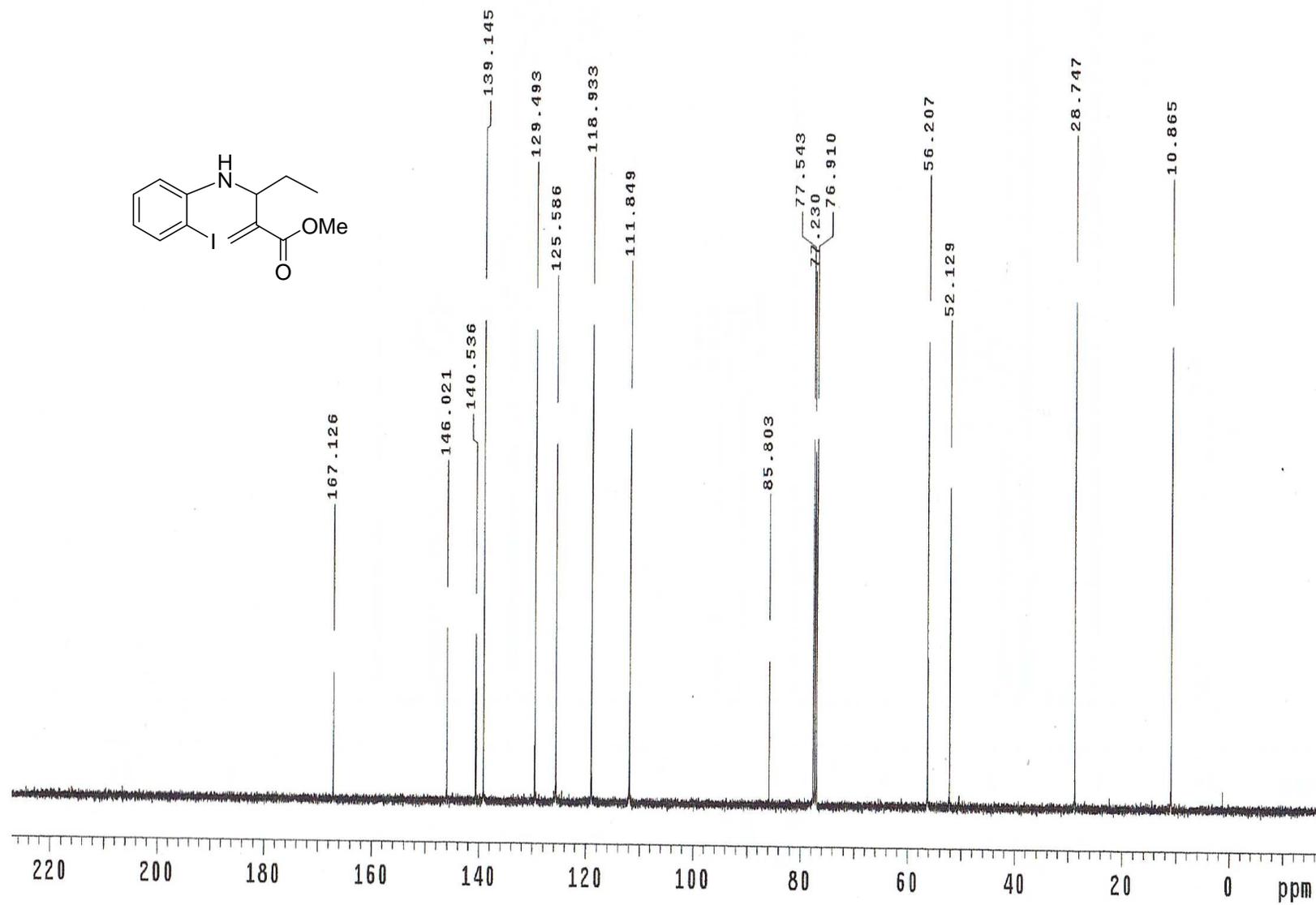
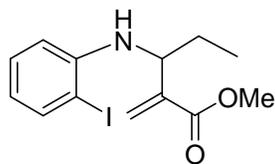
Benzyl 3-(2-iodophenylamino)-2-methylenebutanoate (1c): ^{13}C NMR (100 MHz, CDCl_3)



Methyl 3-(2-iodophenylamino)-2-methylenepentanoate (1d): ^1H NMR (400 MHz, CDCl_3)

Methyl 3-(2-iodophenylamino)-2-methylenepentanoate (1d): ^{13}C NMR (100 MHz, CDCl_3)

at cdc ph

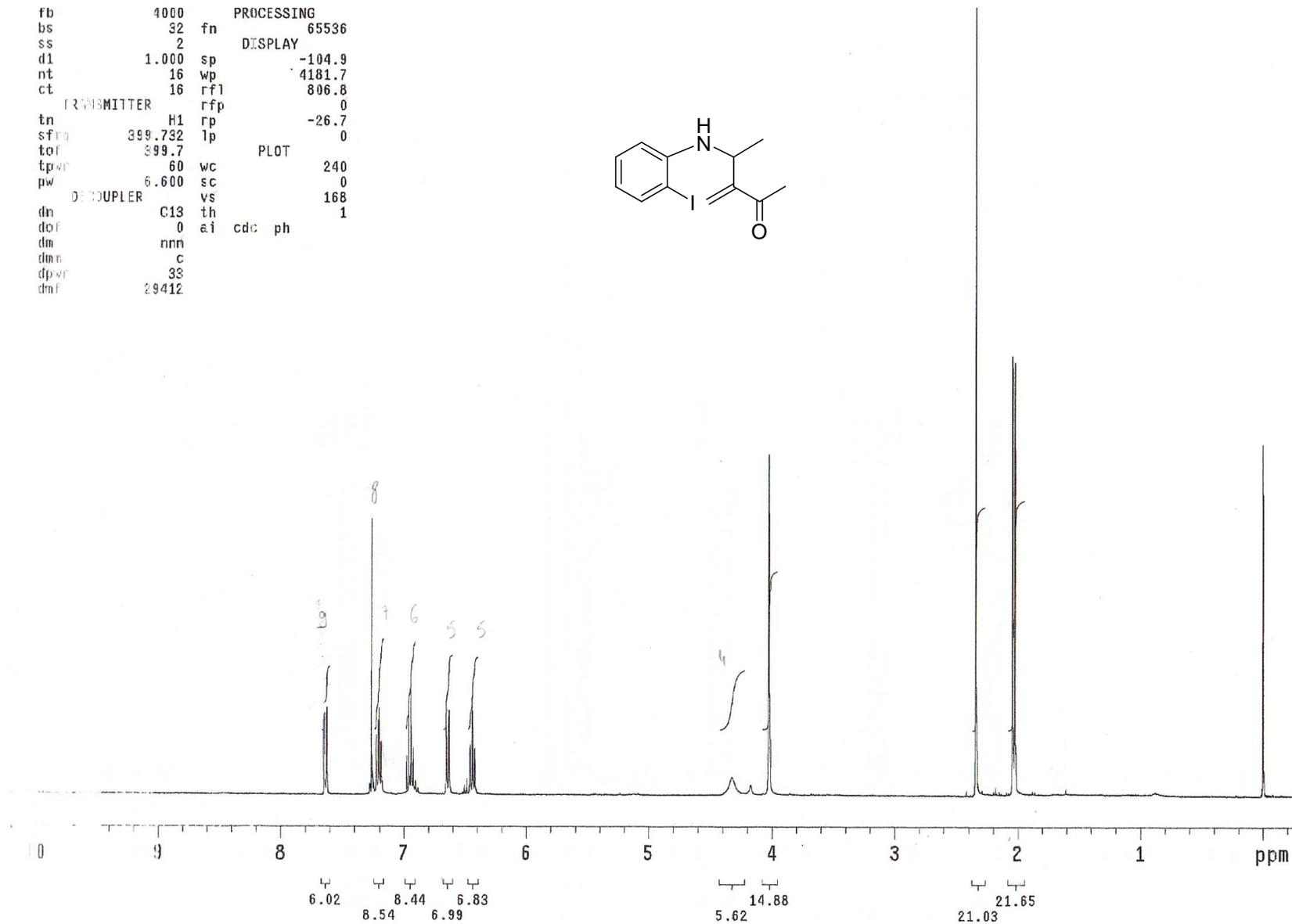
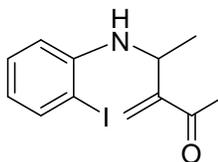


4-(2-iodophenylamino)-3-methylenepentan-2-one (1e): ^1H NMR (400 MHz, CDCl_3)

```

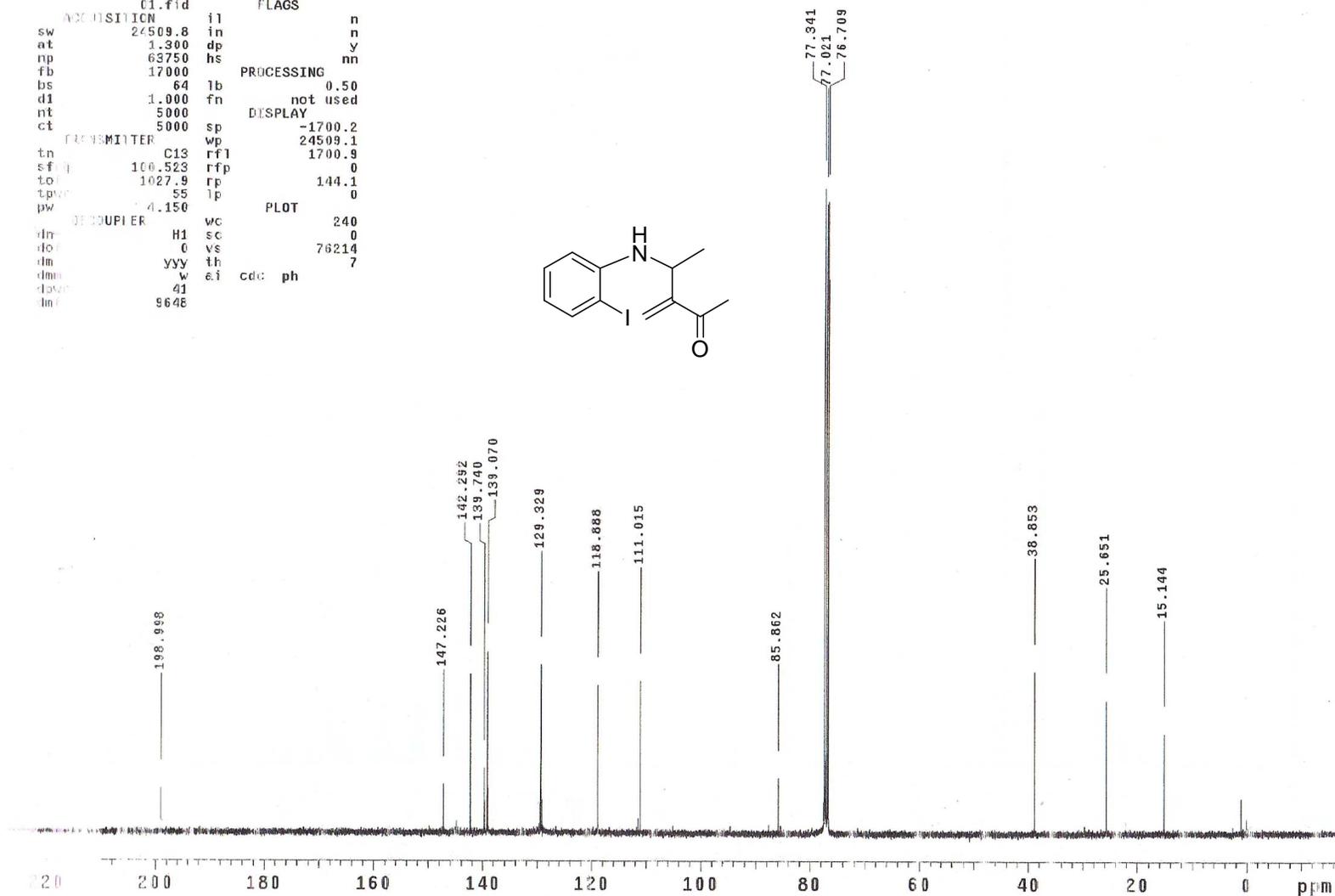
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ss      2      DISPLAY
d1      1.000    sp      -104.9
nt      16      wp      4181.7
ct      16      rfl     806.8
          TRANSMITTER rfp     0
tn      H1      rp     -26.7
sfreq   399.732 lp     0
tof     399.7      PLOT
tprf    60      wc     240
pw      6.600    sc     0
          DECOUPLER vs     168
dn      C13     th     1
dof     0      ai cdc ph
dm      nnn
dnn     c
dprf    33
dnf     29412

```



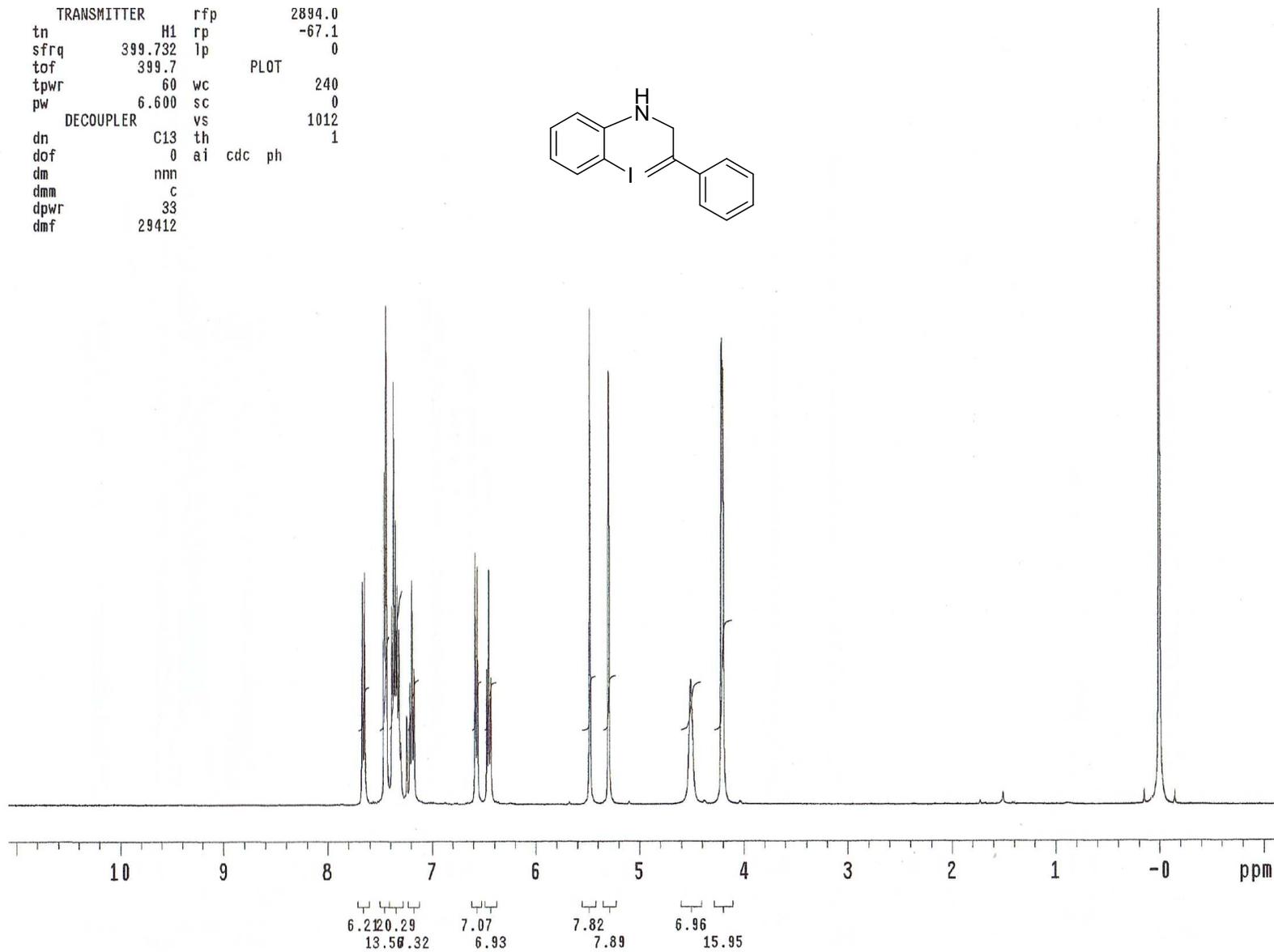
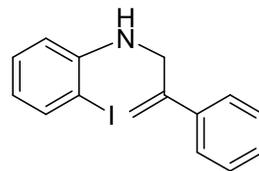
4-(2-iodophenylamino)-3-methylenepentan-2-one (1e): ^{13}C NMR (100 MHz, CDCl_3)

| | | | |
|-------------|-----------------|------------|----------|
| SAMPLE | | SPECIAL | |
| date | Mar 19 2012 | temp | not used |
| solvent | cdc13 | gain | 30 |
| file | home_gallo/v | spin | not used |
| nmr | sysdate/auto_2 | hst | 0.008 |
| 01 | 08 08/1 201203 | pw90 | 8.300 |
| 19 | MS-1802_Carbon_ | alfa | 10.000 |
| | 01.fid | FLAGS | |
| ACQUISITION | il | n | |
| sw | 24509.8 | in | n |
| at | 1.300 | dp | y |
| np | 63750 | hs | nn |
| fb | 17000 | PROCESSING | |
| bs | 64 | lb | 0.50 |
| d1 | 1.000 | fn | not used |
| nt | 5000 | DISPLAY | |
| ct | 5000 | sp | -1700.2 |
| PRERMITTER | | wp | 24509.1 |
| tn | C13 | rfl | 1700.9 |
| sf | 100.523 | rfp | 0 |
| to | 1027.9 | rp | 144.1 |
| tpw | 55 | lp | 0 |
| pw | 4.150 | PLOT | |
| DECOUPLER | | wc | 240 |
| dm | H1 | sc | 0 |
| dof | 0 | vs | 76214 |
| dm | yy | th | 7 |
| dmm | w | ai | cdc: ph |
| dpc | 41 | | |
| dmp | 9648 | | |



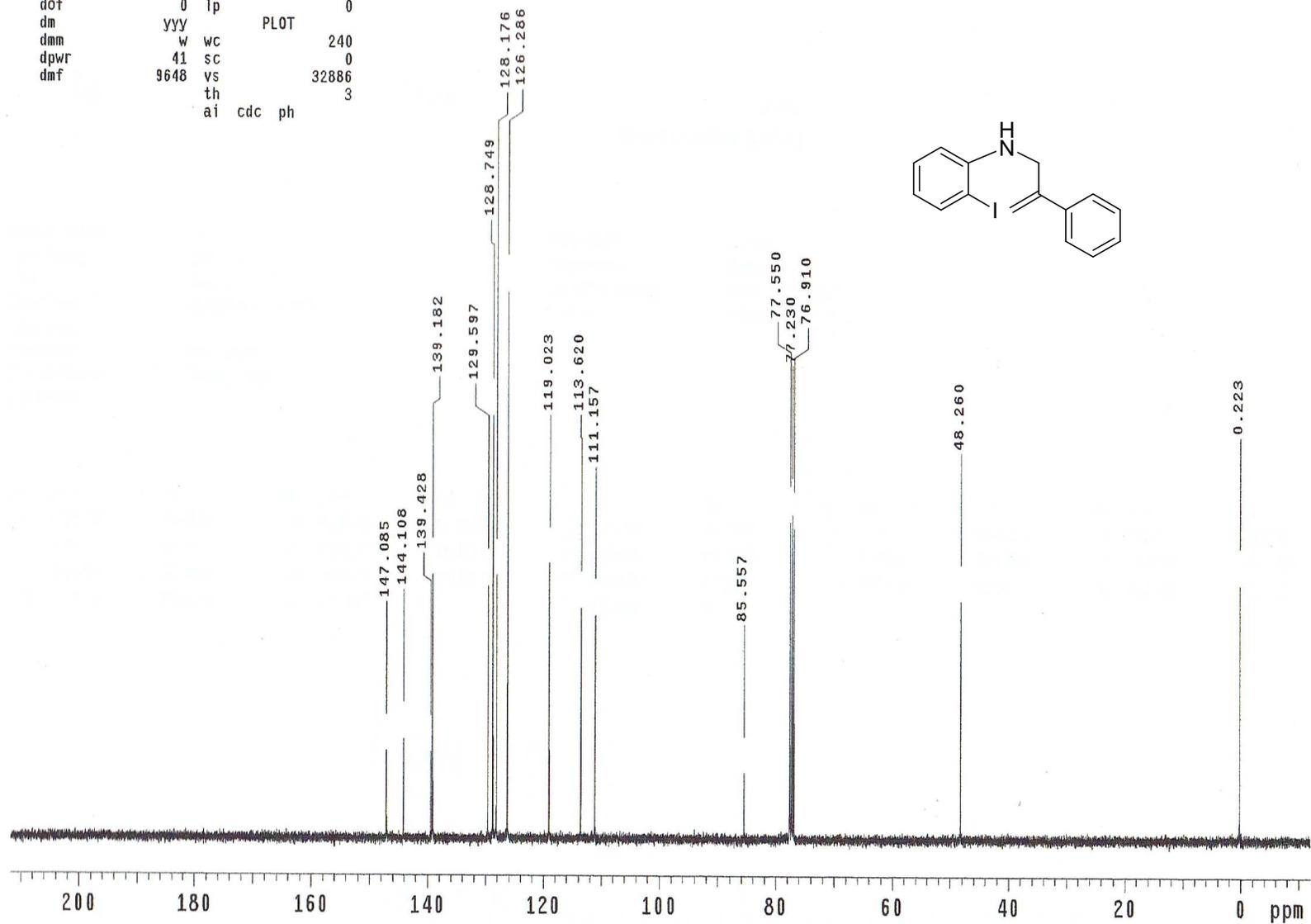
2-Iodo-N-(2-phenylallyl)benzenamine (1f): ^1H NMR (400 MHz, CDCl_3)

| | | | |
|-------------|---------|--------|--------|
| TRANSMITTER | rfp | 2894.0 | |
| tn | H1 | rp | -67.1 |
| sfrq | 399.732 | lp | 0 |
| tof | 399.7 | PLOT | |
| tpwr | 60 | wc | 240 |
| pw | 6.600 | sc | 0 |
| DECOUPLER | C13 | vs | 1012 |
| dn | 0 | th | 1 |
| dof | 0 | ai | cdc ph |
| dm | nnn | | |
| dmm | c | | |
| dpwr | 33 | | |
| dmf | 29412 | | |



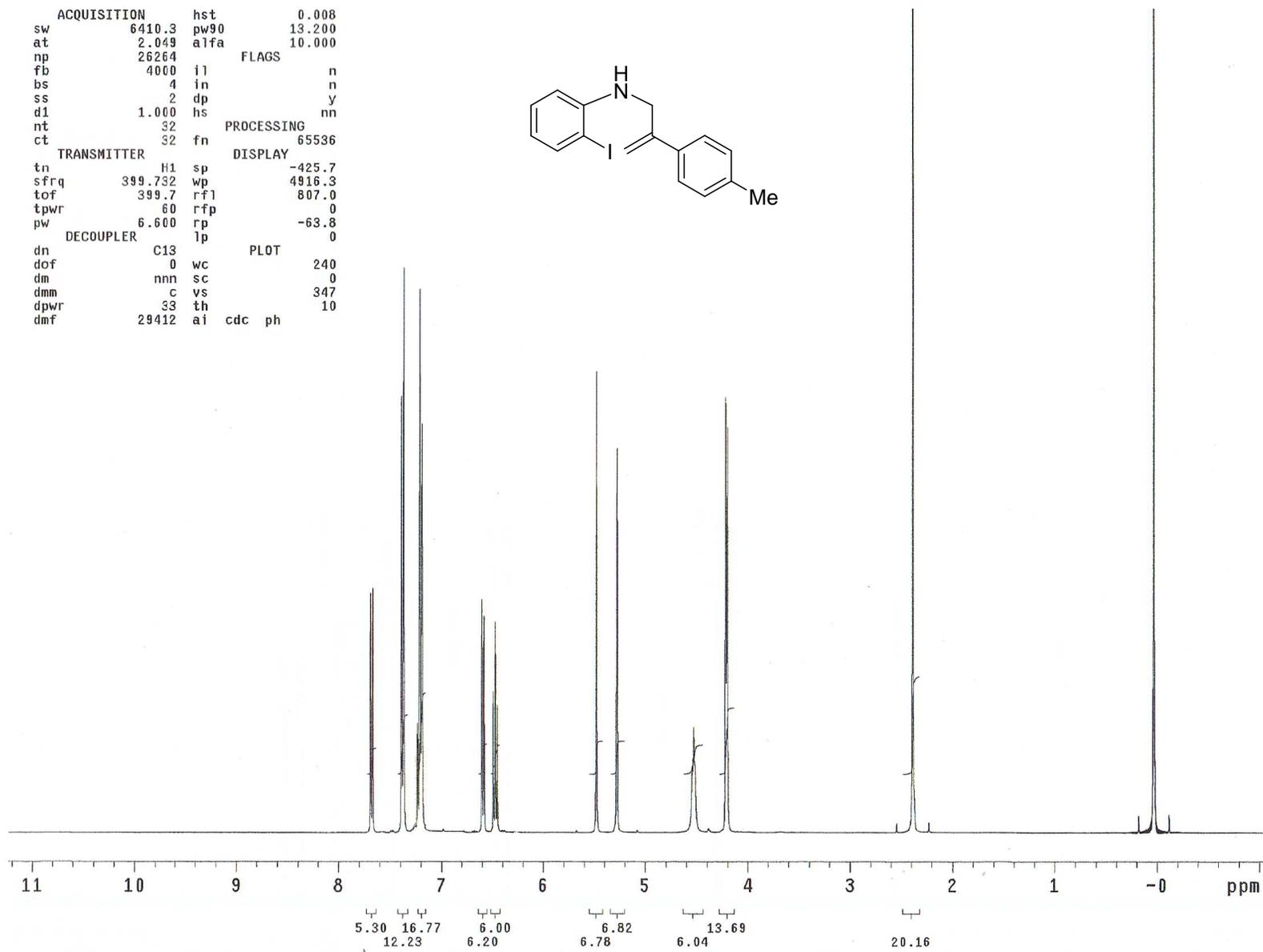
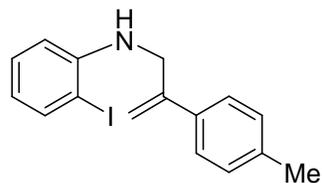
2-Iodo-N-(2-phenylallyl)benzenamine (1f): ^{13}C NMR (100 MHz, CDCl_3)

| | | | |
|-----------|------|------|--------|
| DECOUPLER | | rfp | 7762.6 |
| dn | H1 | rp | 87.2 |
| dof | 0 | lp | 0 |
| dm | yyy | PLOT | |
| dmm | w | wc | 240 |
| dpwr | 41 | sc | 0 |
| dmf | 9648 | vs | 32886 |
| | | th | 3 |
| | ai | cdc | ph |



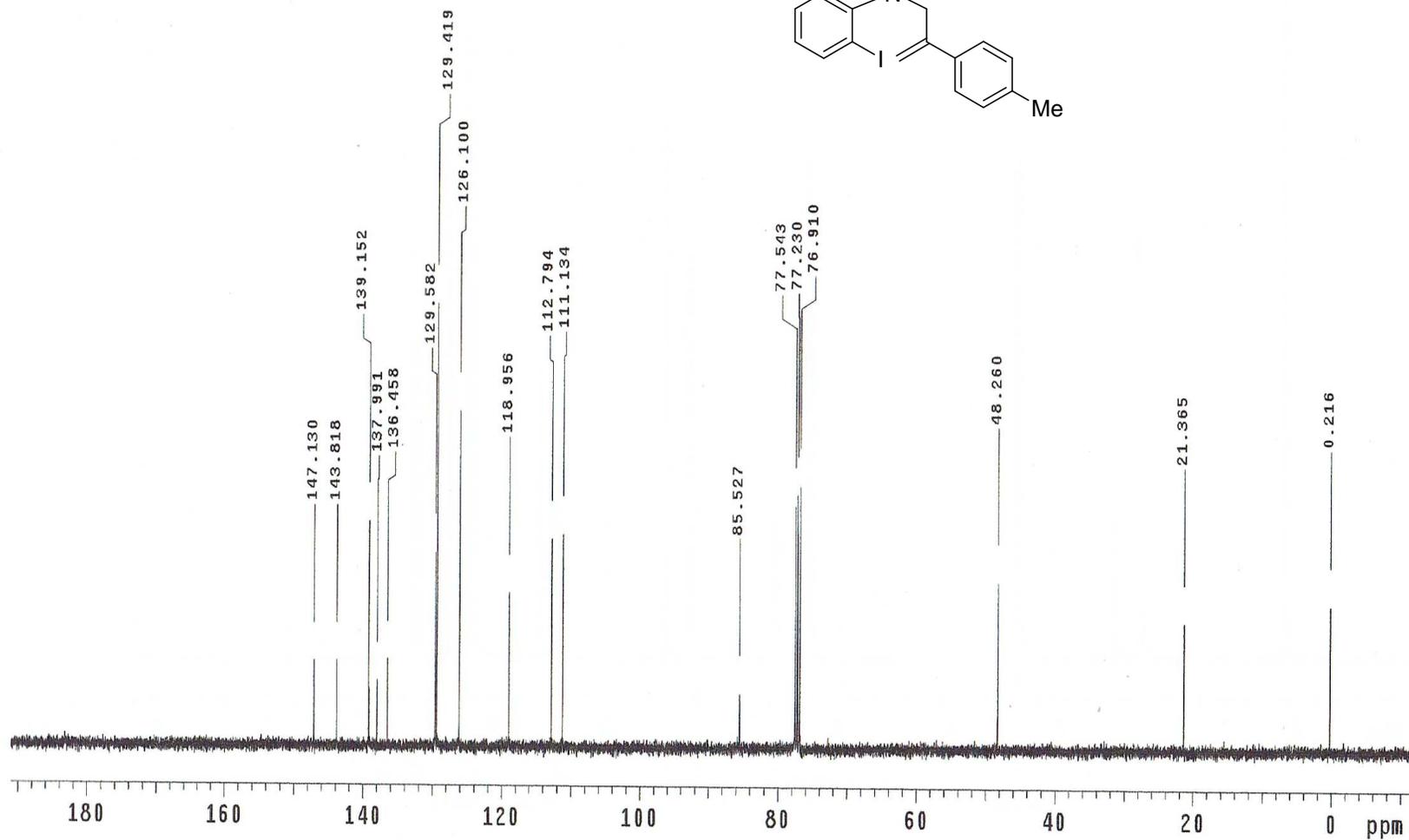
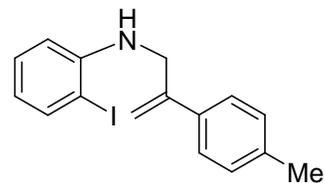
2-Iodo-N-(2-p-tolylallyl)benzenamine (1g): ^1H NMR (400 MHz, CDCl_3)

| | | |
|-------------|---------|-------------|
| ACQUISITION | hst | 0.008 |
| sw | 6410.3 | pw90 13.200 |
| at | 2.049 | alfa 10.000 |
| np | 26264 | FLAGS |
| fb | 4000 | il n |
| bs | 4 | in n |
| ss | 2 | dp y |
| d1 | 1.000 | hs nn |
| nt | 32 | PROCESSING |
| ct | 32 | fn 65536 |
| TRANSMITTER | | DISPLAY |
| tn | H1 | sp -425.7 |
| sfrq | 399.732 | wp 4916.3 |
| tof | 399.7 | rfl 807.0 |
| tpwr | 60 | rfp 0 |
| pw | 6.600 | rp -63.8 |
| DECOUPLER | | lp 0 |
| dn | C13 | PLOT |
| dof | 0 | wc 240 |
| dm | nnn | sc 0 |
| dmm | c | vs 347 |
| dpwr | 33 | th 10 |
| dmf | 29412 | ai cdc ph |



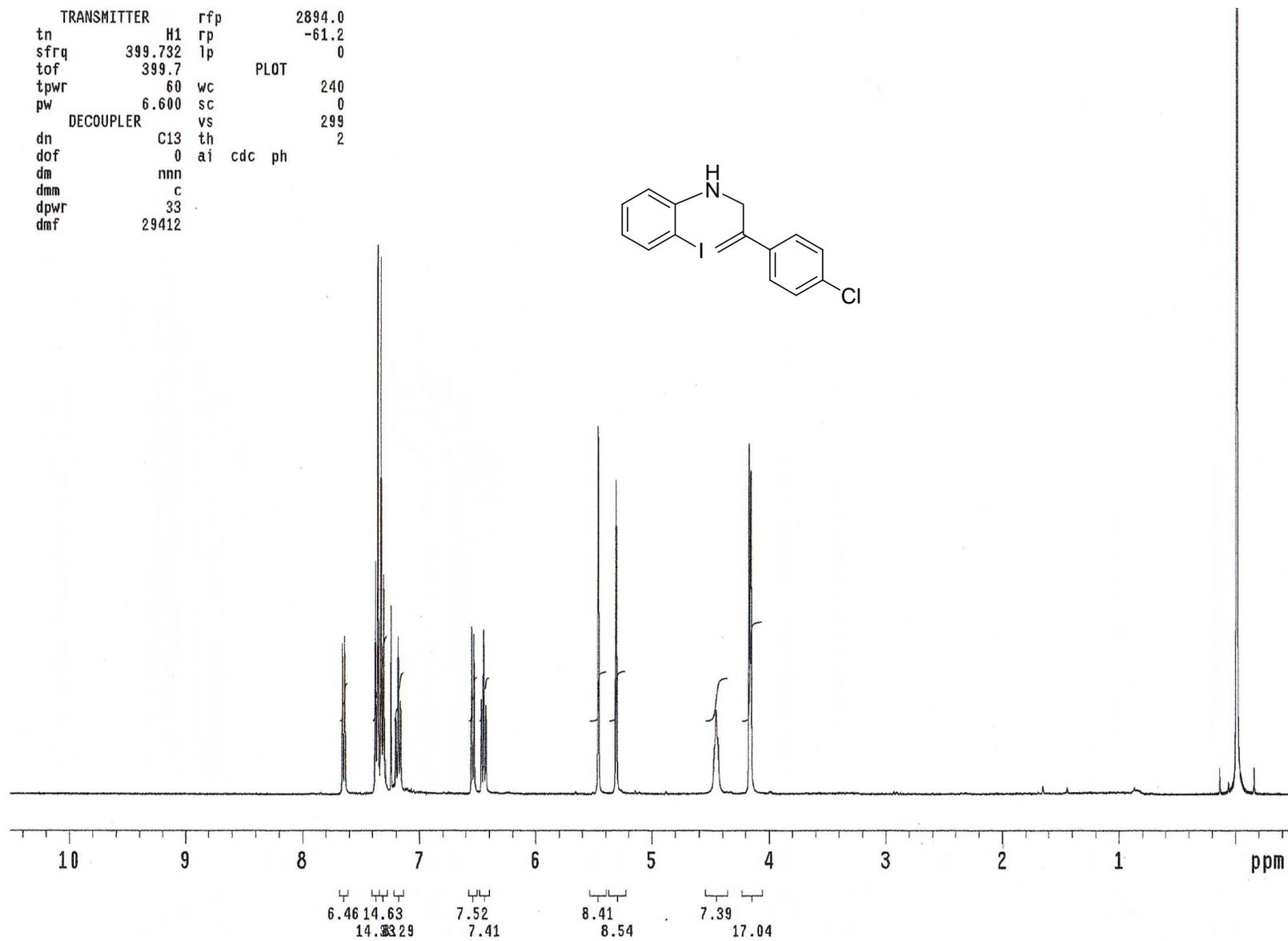
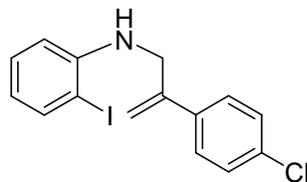
2-Iodo-N-(2-p-tolylallyl)benzenamine (1g): ^{13}C NMR (100 MHz, CDCl_3)

| | | | |
|-----------|------|--------|-------|
| DECOUPLER | rfp | 7762.6 | |
| dn | H1 | rp | 93.5 |
| dof | 0 | lp | 0 |
| dm | yyy | PLOT | |
| dmm | w | wc | 240 |
| dpwr | 41 | sc | 0 |
| dmf | 9648 | vs | 16671 |
| | | th | 4 |
| | ai | cdc | ph |



N-(2-(4-chlorophenyl)allyl)-2-iodobenzamide (1h): ^1H NMR (400 MHz, CDCl_3)

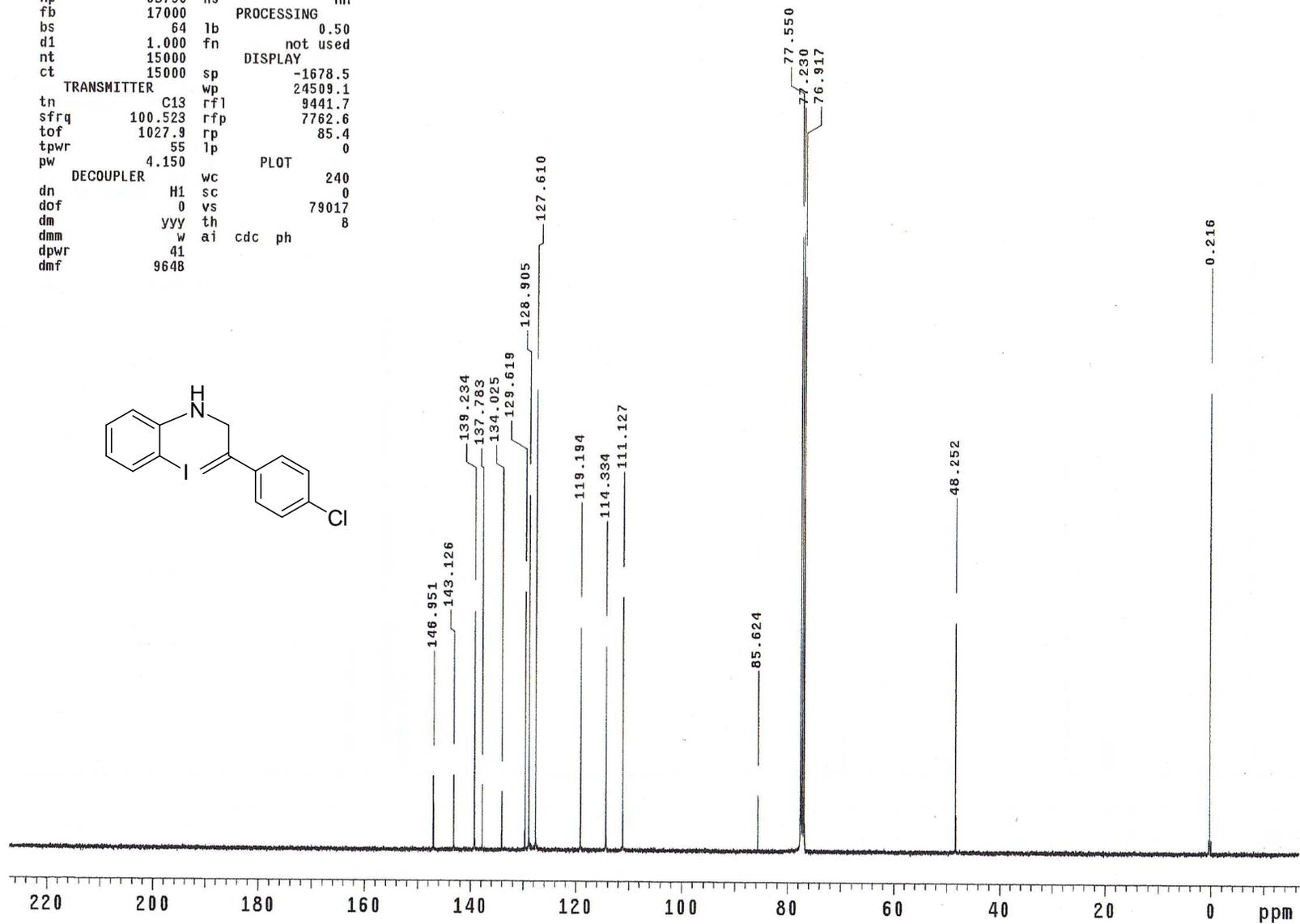
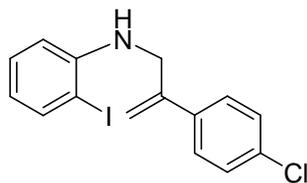
| | | |
|-------------|---------|-----------|
| TRANSMITTER | rfp | 2894.0 |
| tn | H1 | rp -61.2 |
| sfrq | 399.732 | lp 0 |
| tof | 399.7 | PLOT |
| tpwr | 60 | wc 240 |
| pw | 6.600 | sc 0 |
| DECOUPLER | vs | 299 |
| dn | C13 | th 2 |
| dof | 0 | ai cdc ph |
| dm | nnn | |
| dmm | c | |
| dpwr | 33 | |
| dmf | 29412 | |



N-(2-(4-chlorophenyl)allyl)-2-iodobenzamide (1h): ^{13}C NMR (100 MHz, CDCl_3)

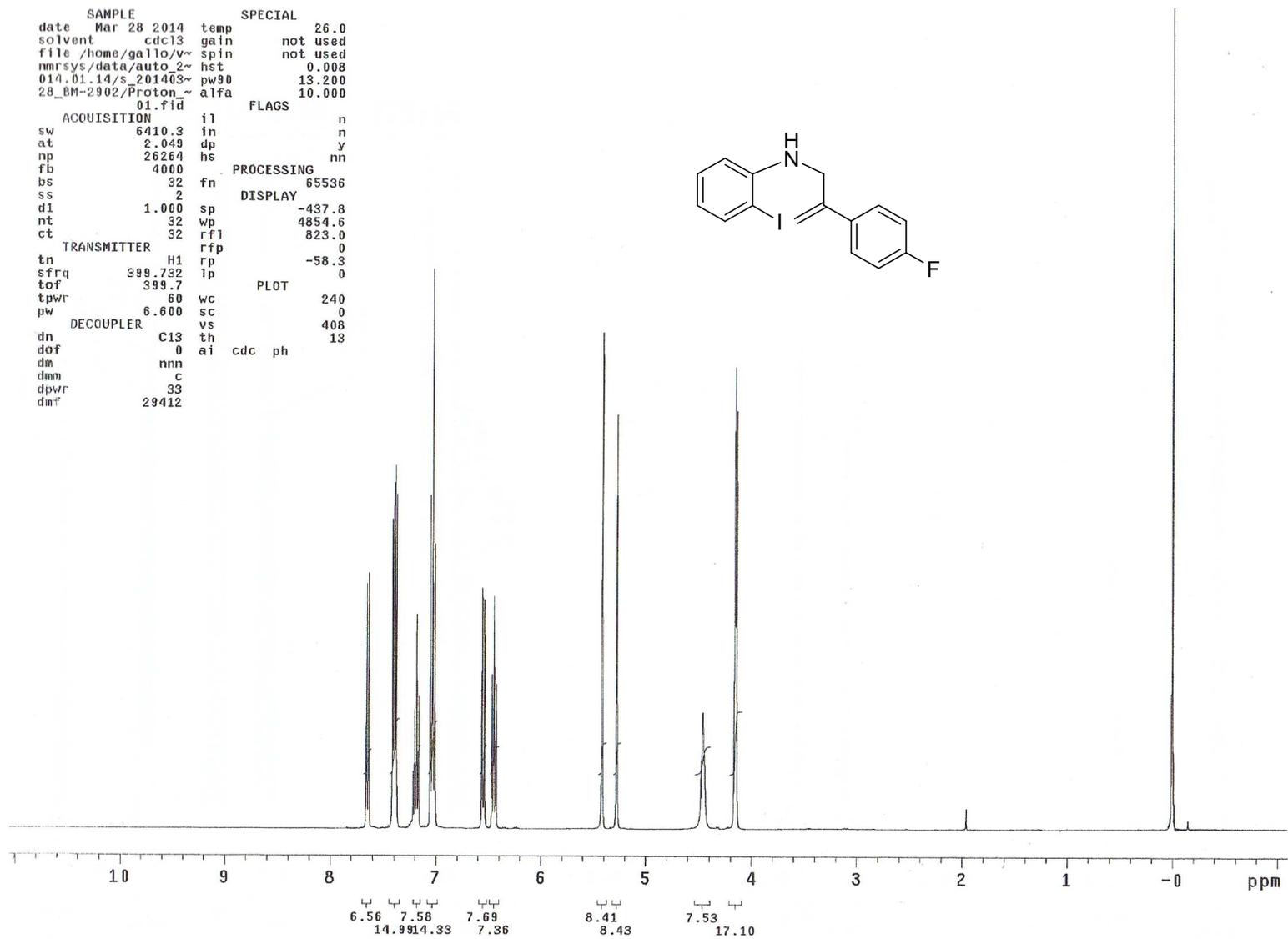
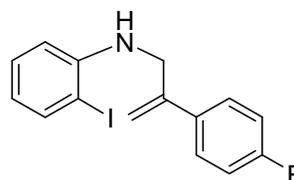
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d1      1.000  fn      not used
nt      15000  DISPLAY
ct      15000  sp      -1678.5
          TRANSMITTER  wp      24509.1
tn      C13   rfl     9441.7
sfrq    100.523 rfp    7762.6
tof     1027.9 rp     85.4
tpwr    55    lp     0
pw      4.150  PLOT
          DECOUPLER   wc     240
dn      H1    sc     0
dof     0    vs    79017
dm      yyy  th     8
dmm     w    ai    cdc ph
dpwr    41
dmf     9648
  
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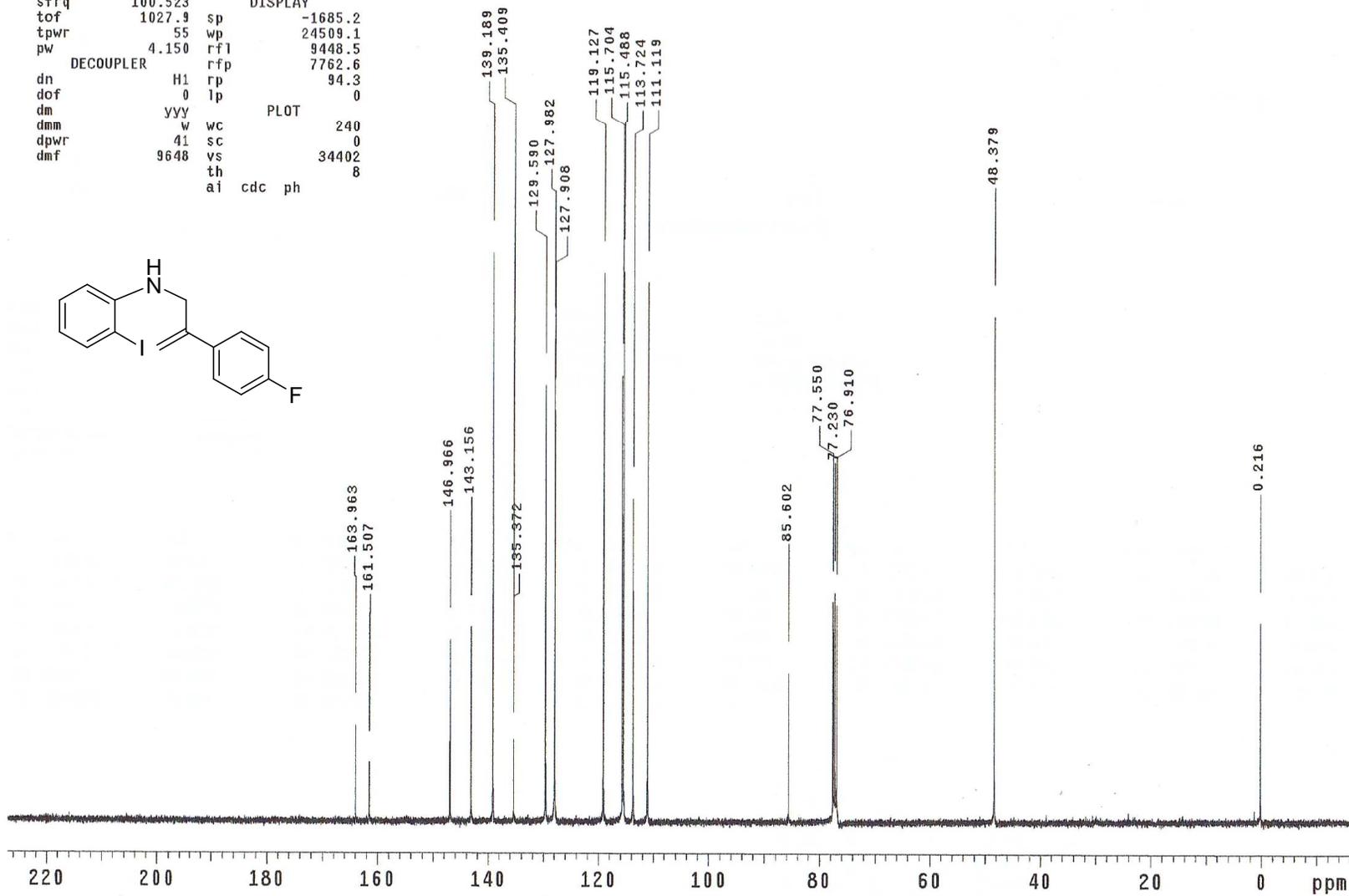
N-(2-(4-fluorophenyl)allyl)-2-iodobenzenamine (1i): ^1H NMR (400 MHz, CDCl_3)

| | | | |
|---------------------|------------------|------------|----------|
| SAMPLE | | SPECIAL | |
| date | Mar 28 2014 | temp | 26.0 |
| solvent | cdc13 | gain | not used |
| file | /home/gallo/v~ | spin | not used |
| nmr | sys/data/auto_2~ | hst | 0.008 |
| 014.01.14/s_201403~ | | pw90 | 13.200 |
| 28_BM-2902/Proton~ | | alfa | 10.000 |
| 01.fid | | FLAGS | |
| ACQUISITION | | il | n |
| sw | 6410.3 | in | n |
| at | 2.049 | dp | y |
| np | 26264 | hs | nn |
| fb | 4000 | PROCESSING | |
| bs | 32 | fn | 65536 |
| ss | 2 | DISPLAY | |
| d1 | 1.000 | sp | -437.8 |
| nt | 32 | wp | 4854.6 |
| ct | 32 | rfl | 823.0 |
| TRANSMITTER | | rffp | 0 |
| tn | H1 | rp | -58.3 |
| sfrq | 399.732 | lp | 0 |
| tof | 399.7 | PLOT | |
| tpwr | 60 | wc | 240 |
| pw | 6.600 | sc | 0 |
| DECOUPLER | | vs | 408 |
| dn | C13 | th | 13 |
| dof | 0 | ai | cdc ph |
| dm | nnn | | |
| dmm | c | | |
| dpwr | 33 | | |
| dmr | 29412 | | |



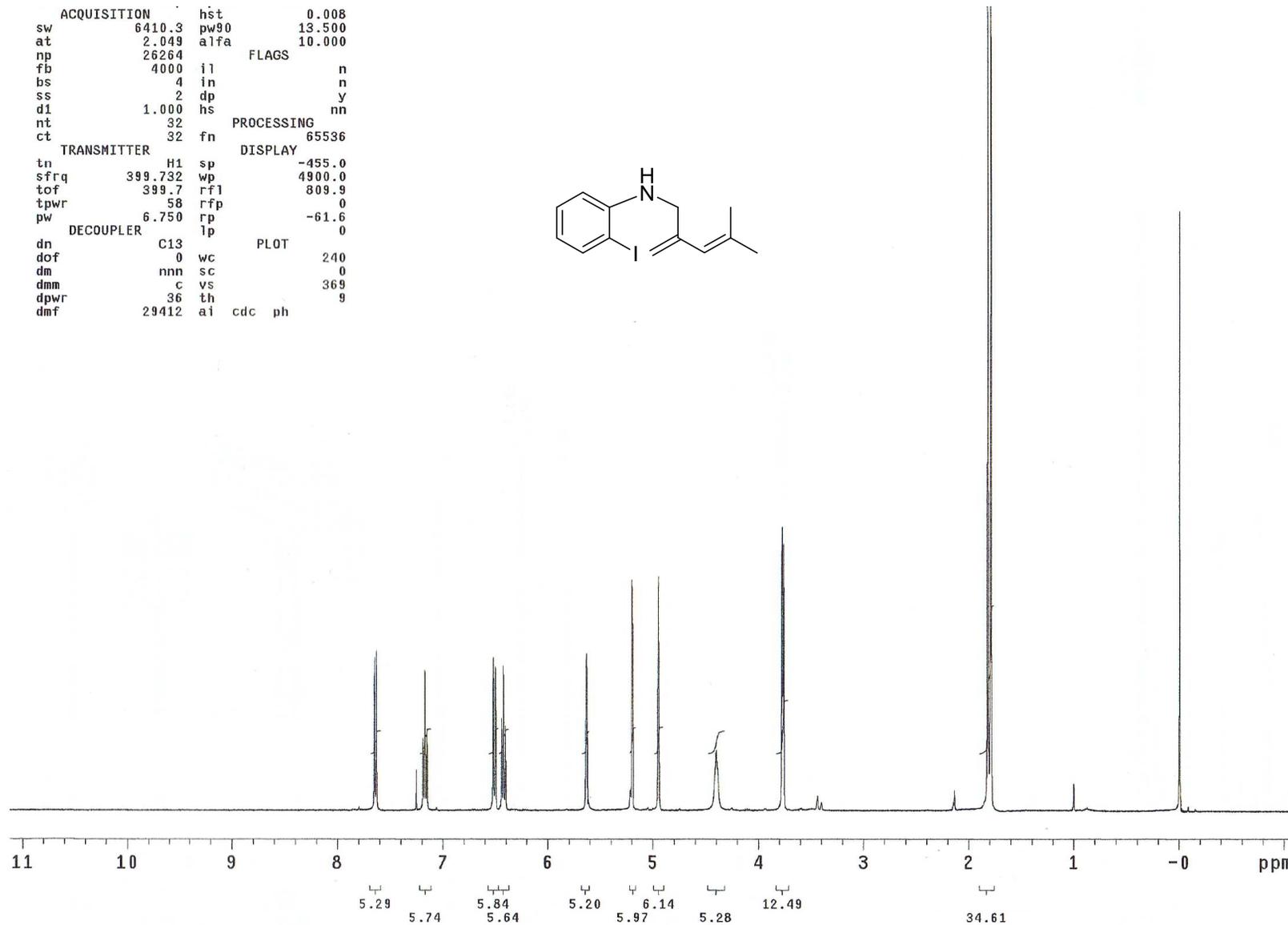
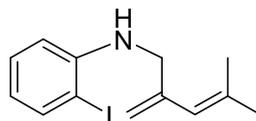
N-(2-(4-fluorophenyl)allyl)-2-iodobenzenamine (1i): ^{13}C NMR (100 MHz, CDCl_3)

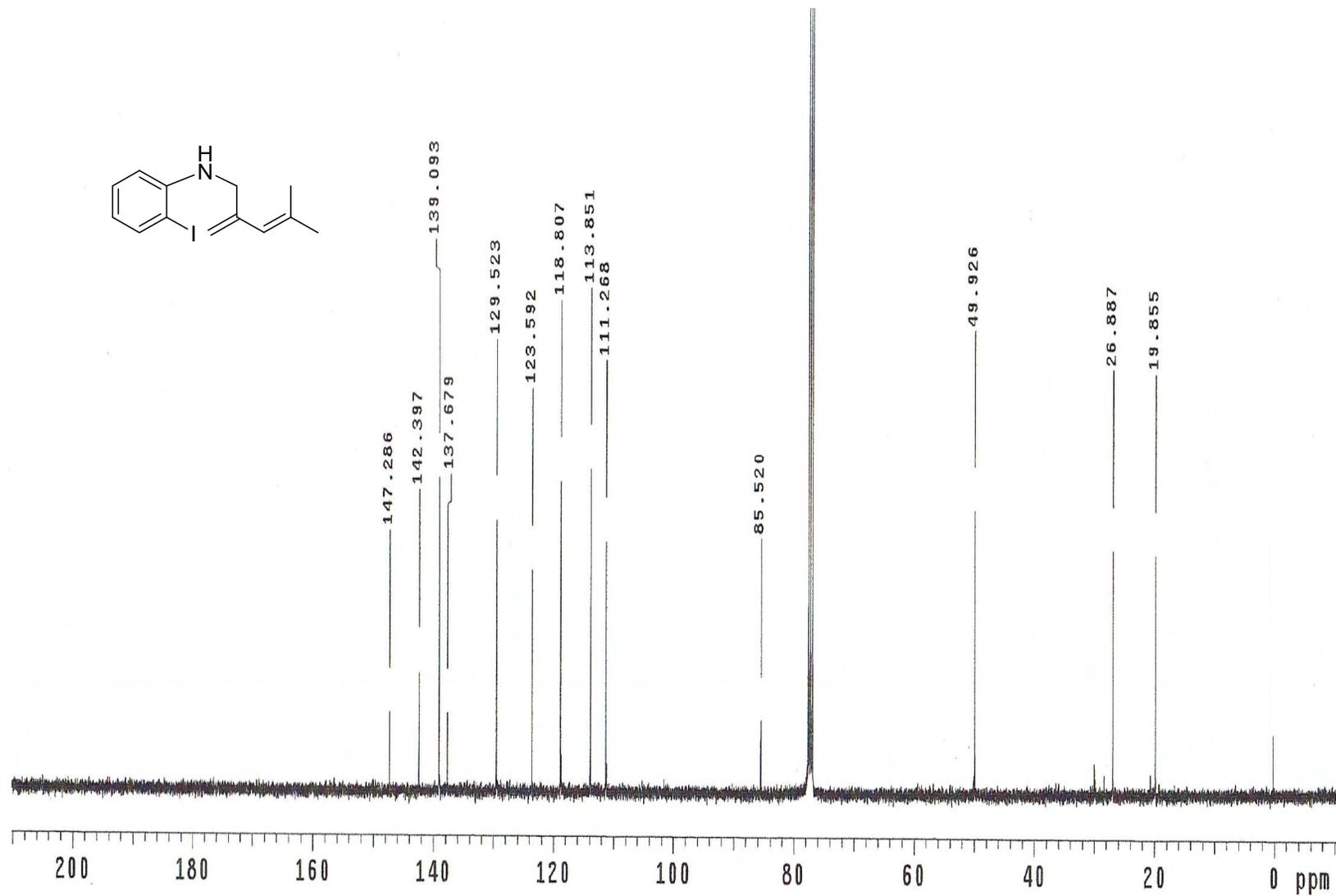
| | | |
|-------------|---------|-------------|
| TRANSMITTER | lb | 0.50 |
| tn | C13 | fn not used |
| sfrq | 100.523 | DISPLAY |
| tof | 1027.9 | sp -1685.2 |
| tpwr | 55 | wp 24509.1 |
| pw | 4.150 | rfl 9448.5 |
| DECOUPLER | H1 | rfl 7762.6 |
| dn | 0 | rp 94.3 |
| dof | 0 | lp 0 |
| dm | yyy | PLOT |
| dmm | w | wc 240 |
| dpwr | 41 | sc 0 |
| dmf | 9648 | vs 34402 |
| | th | 8 |
| | ai | cdc ph |



2-Iodo-N-(4-methyl-2-methylenepent-3-enyl)benzenamine (1j): ^1H NMR (400 MHz, CDCl_3)

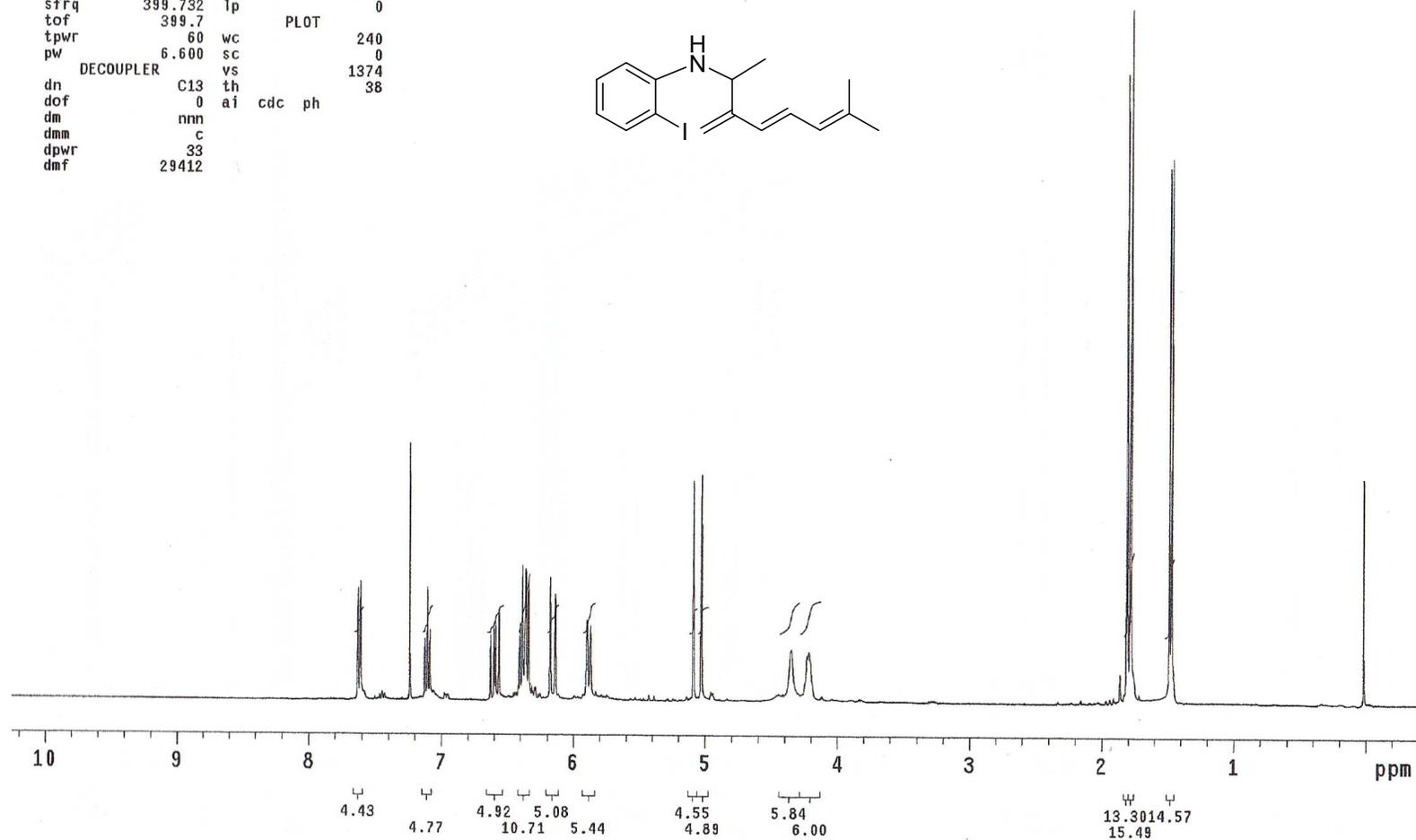
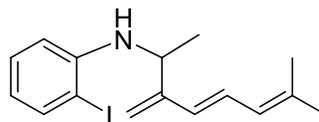
| | | |
|-------------|---------|-------------|
| ACQUISITION | hst | 0.008 |
| sw | 6410.3 | pw90 13.500 |
| at | 2.049 | alfa 10.000 |
| np | 26264 | FLAGS |
| fb | 4000 | il n |
| bs | 4 | in n |
| ss | 2 | dp y |
| d1 | 1.000 | hs nn |
| nt | 32 | PROCESSING |
| ct | 32 | fn 65536 |
| TRANSMITTER | | DISPLAY |
| tn | H1 | sp -455.0 |
| sfrq | 399.732 | wp 4900.0 |
| tof | 399.7 | rfl 809.9 |
| tpwr | 58 | rfp 0 |
| pw | 6.750 | rp -61.6 |
| DECOUPLER | | lp 0 |
| dn | C13 | PLOT |
| dof | 0 | wc 240 |
| dm | nnn | sc 0 |
| dmm | c | vs 369 |
| dpwr | 36 | th 9 |
| dmf | 29412 | ai cdc ph |



2-Iodo-N-(4-methyl-2-methylenepent-3-enyl)benzenamine (1j): ^{13}C NMR (100 MHz, CDCl_3)

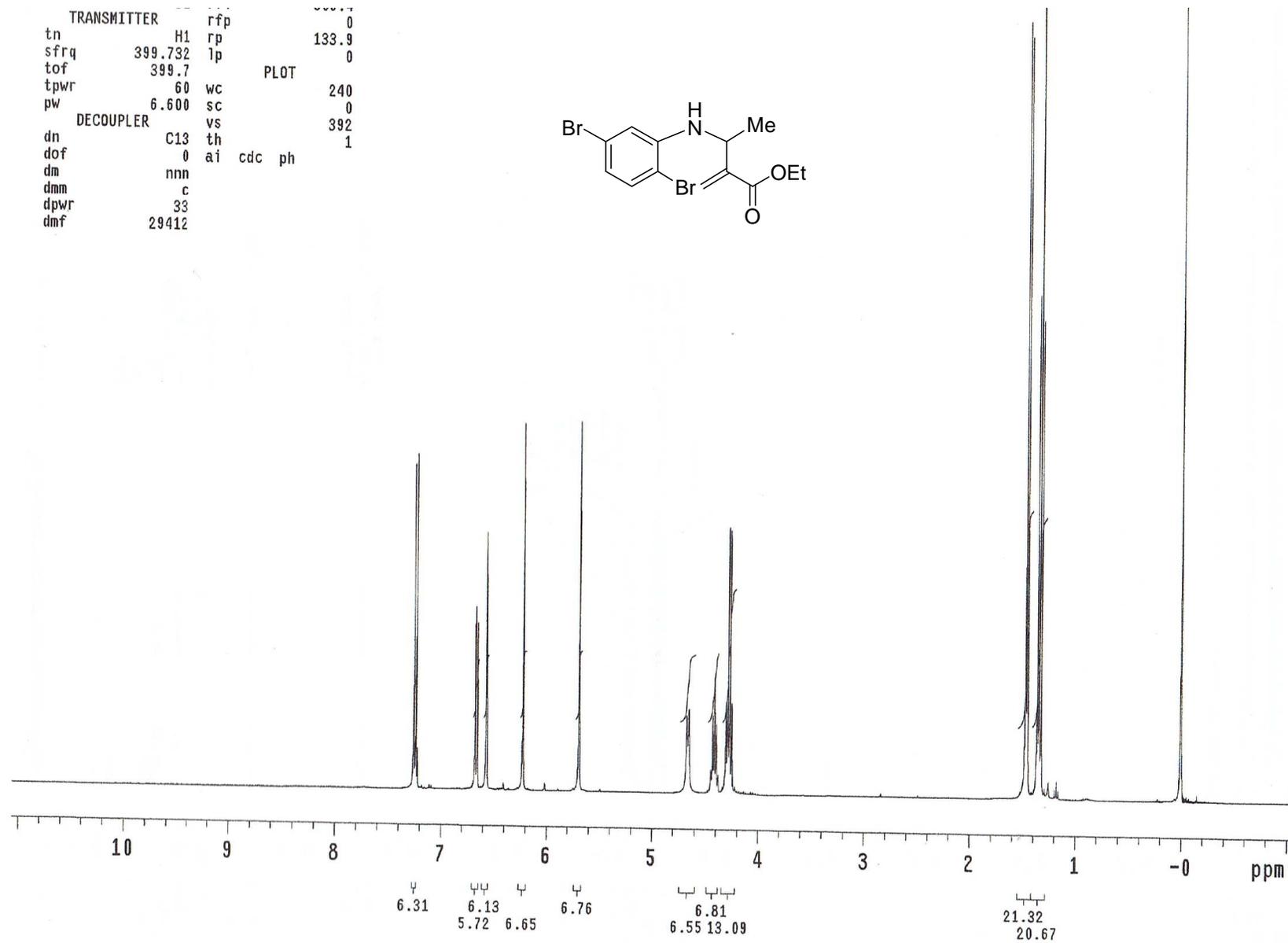
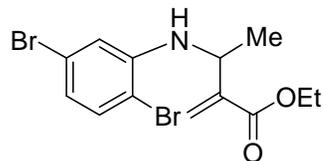
2-Iodo-N-((E)-7-methyl-3-methyleneocta-4,6-dien-2-yl)benzenamine (1k): ^1H NMR (400 MHz, CDCl_3)

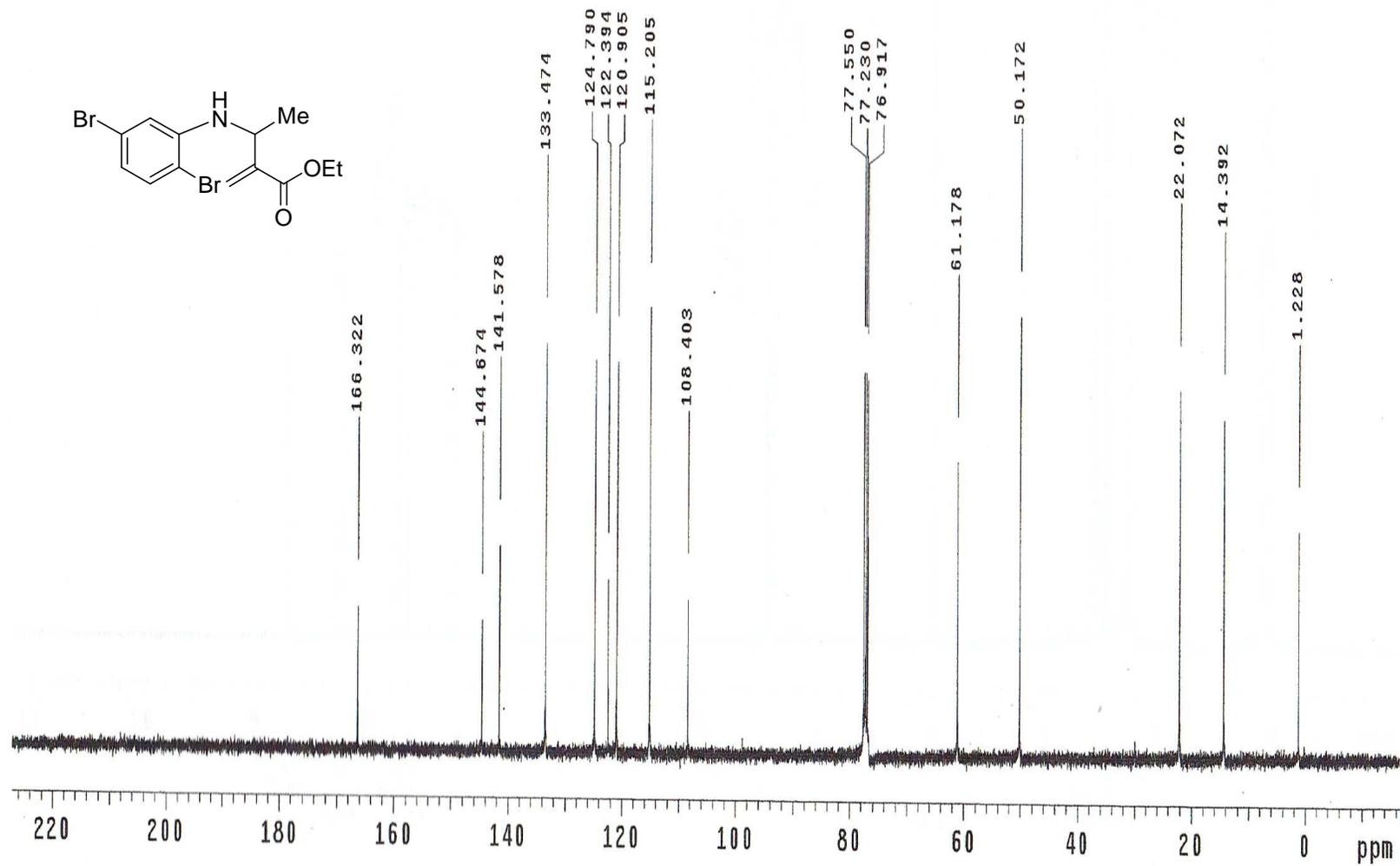
| | | | |
|-------------|---------|------------|--------|
| ACQUISITION | | il | n |
| sw | 6410.3 | in | n |
| at | 2.049 | dp | y |
| np | 26264 | hs | nn |
| fb | 4000 | PROCESSING | |
| bs | 32 | fn | 65536 |
| ss | 2 | DISPLAY | |
| d1 | 1.000 | sp | -161.0 |
| nt | 32 | wp | 4259.0 |
| ct | 32 | rfl | 3709.8 |
| TRANSMITTER | | rfl | 2894.0 |
| tn | H1 | rp | -44.9 |
| sfrq | 399.732 | lp | 0 |
| tof | 399.7 | PLOT | |
| tpwr | 60 | wc | 240 |
| pw | 6.600 | sc | 0 |
| DECOUPLER | | vs | 1374 |
| dn | C13 | th | 38 |
| dof | 0 | ai | cdc ph |
| dm | nnn | | |
| dmm | c | | |
| dpwr | 33 | | |
| dmf | 29412 | | |



Ethyl 3-(2,5-dibromophenylamino)-2-methylenebutanoate (3b): ^1H NMR (400 MHz, CDCl_3)

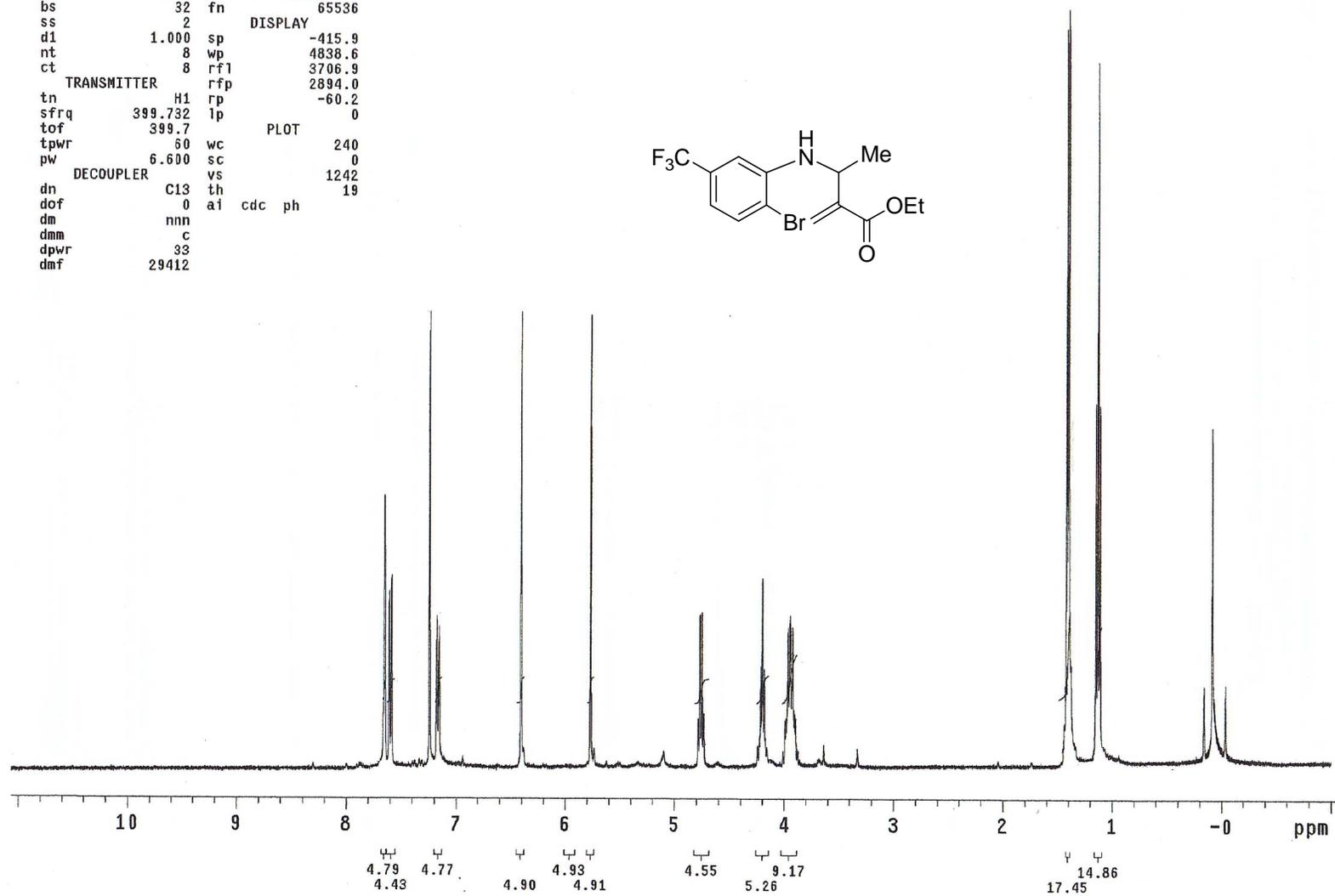
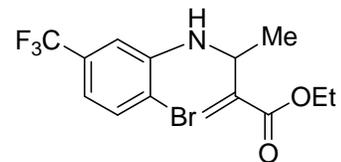
| | | | |
|-------------|---------|-----|--------|
| TRANSMITTER | H1 | rfp | 0 |
| tn | | rp | 133.9 |
| sfrq | 399.732 | lp | 0 |
| tof | 399.7 | | |
| tpwr | 60 | wc | 240 |
| pw | 6.600 | sc | 0 |
| DECOUPLER | C13 | vs | 392 |
| dn | 0 | th | 1 |
| dof | | ai | cdc ph |
| dm | nnn | | |
| dmm | c | | |
| dpwr | 33 | | |
| dmf | 29412 | | |



Ethyl 3-(2,5-dibromophenylamino)-2-methylenebutanoate (3b): ^{13}C NMR (100 MHz, CDCl_3)

Ethyl 3-(2-bromo-5-(trifluoromethyl)phenylamino)-2-methylenebutanoate (4b): ^1H NMR (400 MHz, CDCl_3)

| | | | | |
|-------------|---------|------------|-------|--------|
| ACQUISITION | | il | ----- | n |
| sw | 6410.3 | in | | n |
| at | 2.049 | dp | | y |
| np | 26264 | hs | | nn |
| fb | 4000 | PROCESSING | | |
| bs | 32 | fn | | 65536 |
| ss | 2 | DISPLAY | | |
| d1 | 1.000 | sp | | -415.9 |
| nt | 8 | wp | | 4836.6 |
| ct | 8 | rfl | | 3706.9 |
| TRANSMITTER | | rfp | | 2894.0 |
| tn | H1 | rp | | -60.2 |
| sfrq | 399.732 | lp | | 0 |
| tof | 399.7 | PLOT | | |
| tpwr | 60 | wc | | 240 |
| pw | 6.600 | sc | | 0 |
| DECOUPLER | | vs | | 1242 |
| dn | C13 | th | | 19 |
| dof | 0 | ai | cdc | ph |
| dm | nnn | | | |
| dmm | c | | | |
| dpwr | 33 | | | |
| dmf | 29412 | | | |

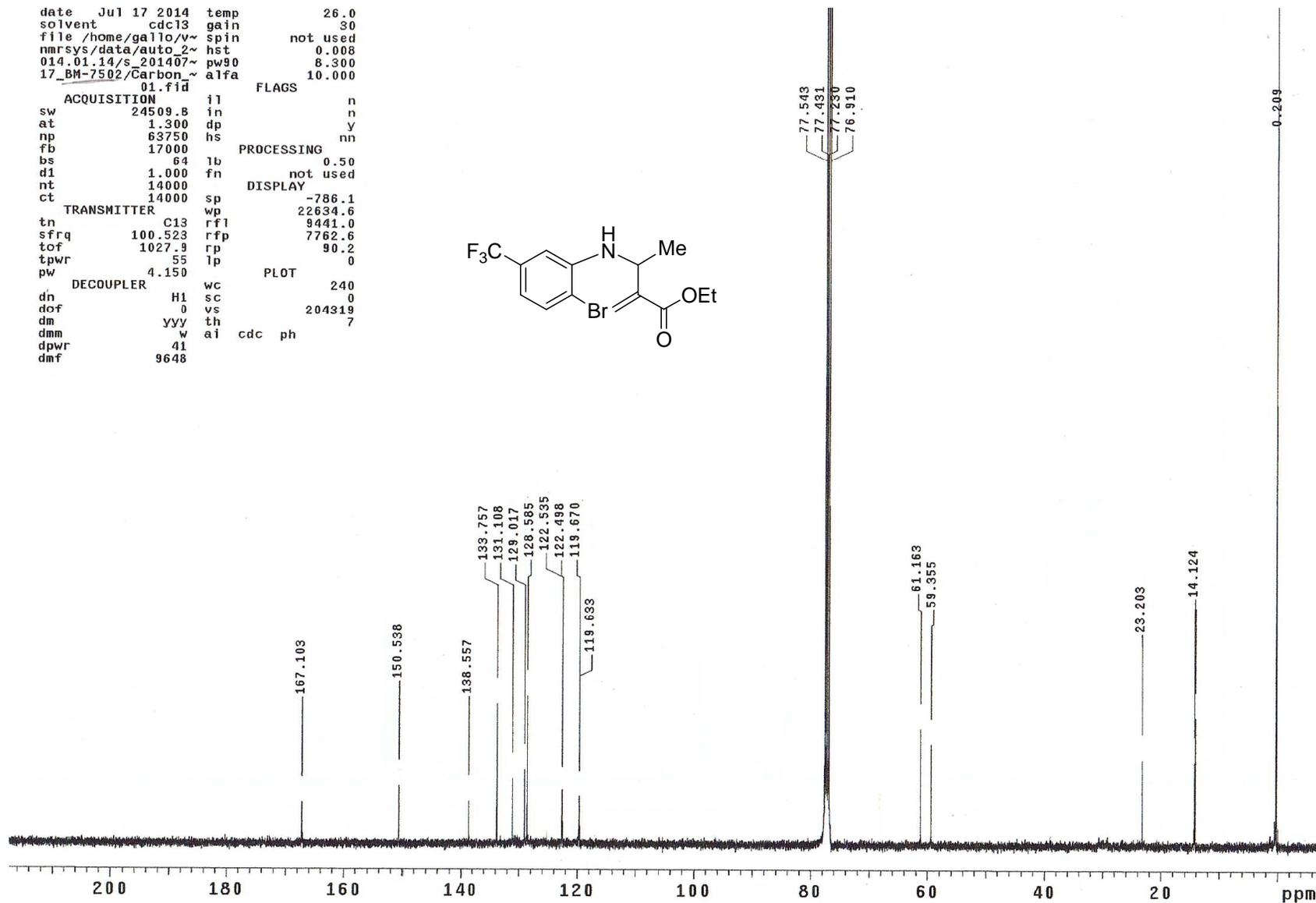
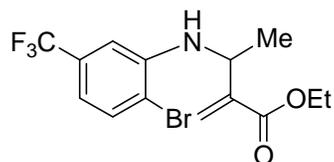


Ethyl 3-(2-bromo-5-(trifluoromethyl)phenylamino)-2-methylenebutanoate (4b): ^{13}C NMR (100 MHz, CDCl_3)

```

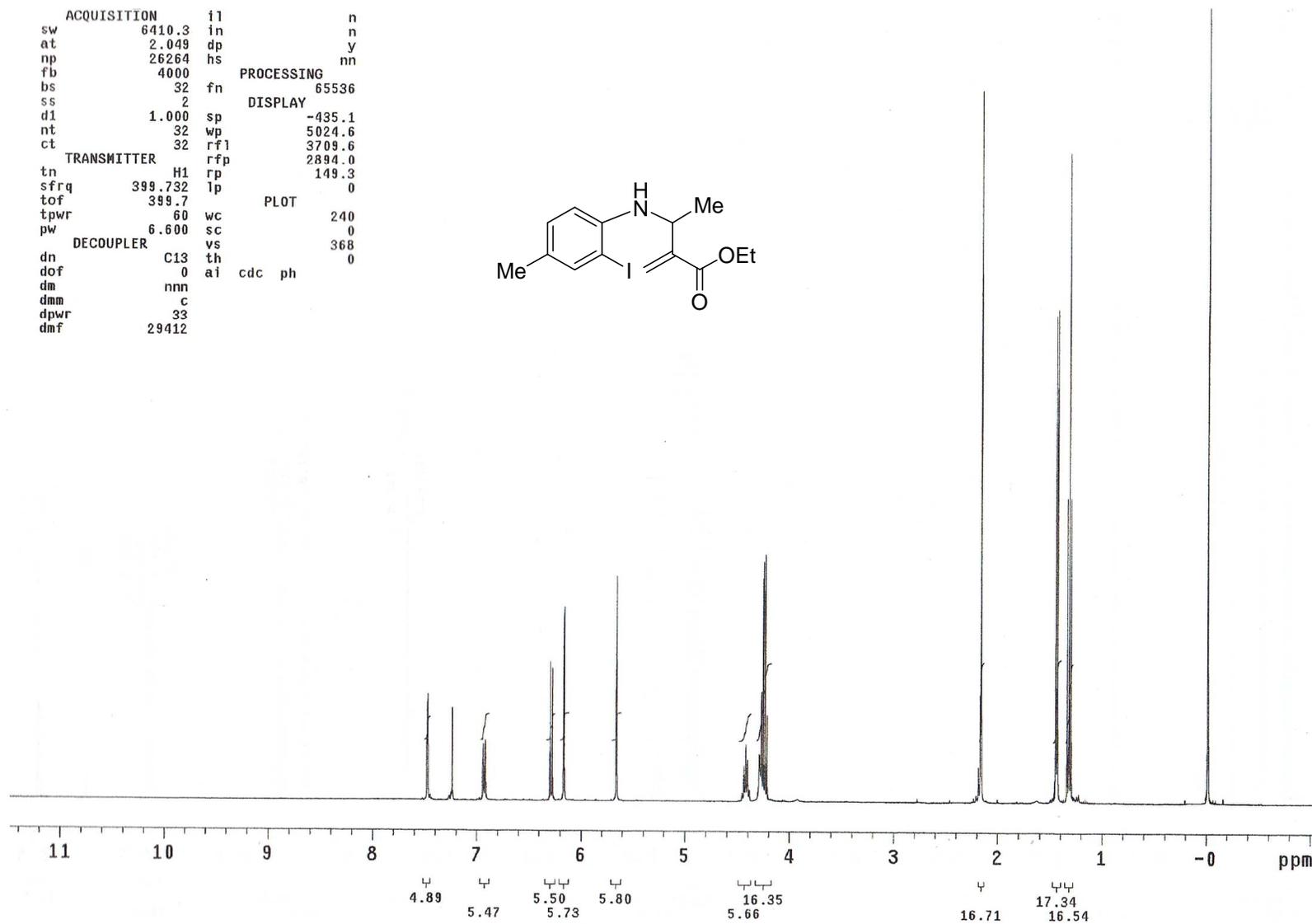
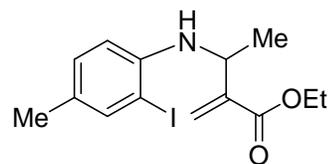
date Jul 17 2014 temp 26.0
solvent cdc13 gain 30
file /home/gallo/v~ spin not used
nmrSYS/data/auto_2~ hst 0.008
014.01.14/s_201407~ pw90 8.300
17_BM-7502/Carbon~ alfa 10.000
01.fid
ACQUISITION il n
sw 24509.8 in n
at 1.300 dp Y
np 63750 hs nn
fb 17000
bs 64 lb PROCESSING 0.50
d1 1.000 fn not used
nt 14000 DISPLAY
ct 14000 sp -786.1
TRANSMITTER wp 22634.6
tn C13 rfl 9441.0
sfrq 100.523 rfp 7762.6
tof 1027.9 rp 90.2
tpwr 55 lp 0
pw 4.150 PLOT
DECOUPLER H1 wc 240
dn 0 sc 0
dof 0 vs 204319
dm YYY th 7
dmm w ai cdc ph
dpwr 41
dmf 9648

```



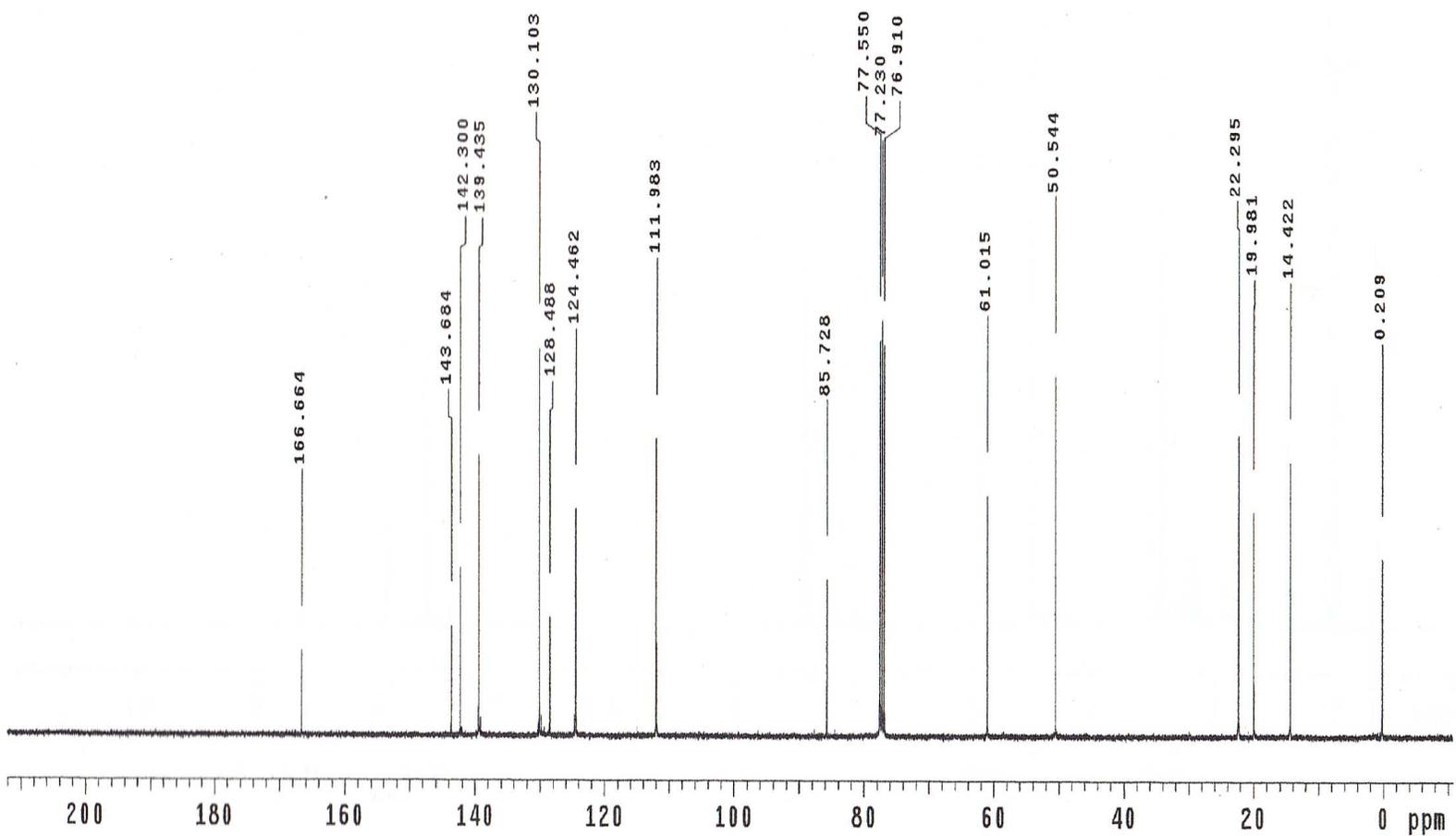
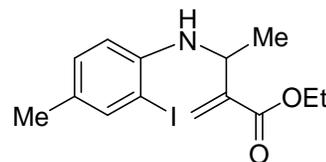
Ethyl 3-(2-iodo-4-methylphenylamino)-2-methylenebutanoate (**5b**): ^1H NMR (400 MHz, CDCl_3)

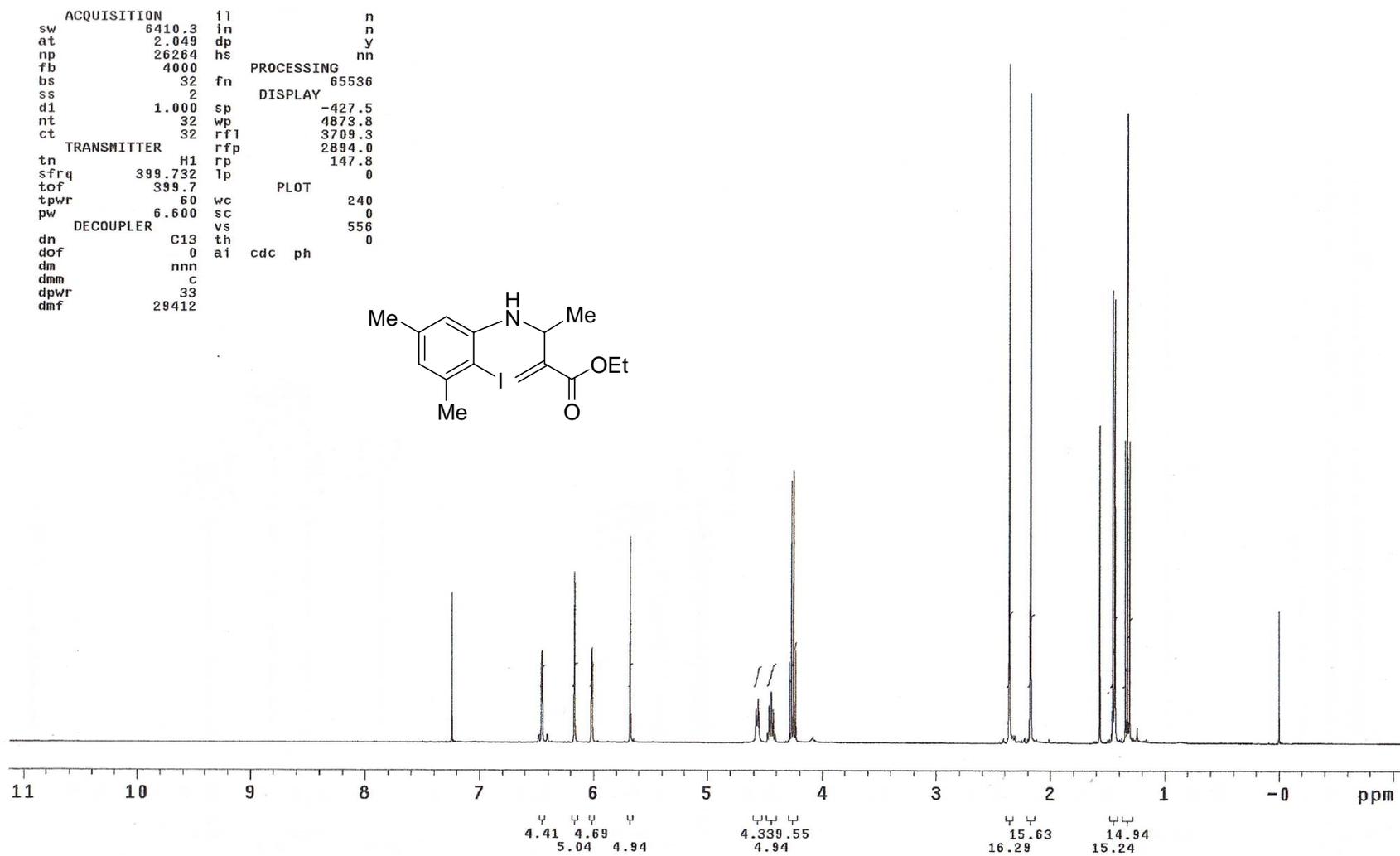
```
ACQUISITION      i1          n
sw      6410.3    in          n
at      2.049     dp          y
np      26264     hs          nn
fb      4000
bs      32       fn          65536
ss      2        DISPLAY
d1      1.000    sp          -435.1
nt      32      wp          5024.6
ct      32      rfl         3709.6
          TRANSMITTER rfp         2894.0
tn      H1      rp          149.3
sfrq    399.732 lp          0
tof     399.7   PLOT
tpwr    60      wc          240
pw     6.600   sc          0
          DECOUPLER C13      vs          368
dn      0      th          0
dof     0      ai      cdc  ph
dm      nnn
dmm     c
dpwr    33
dmf     29412
```



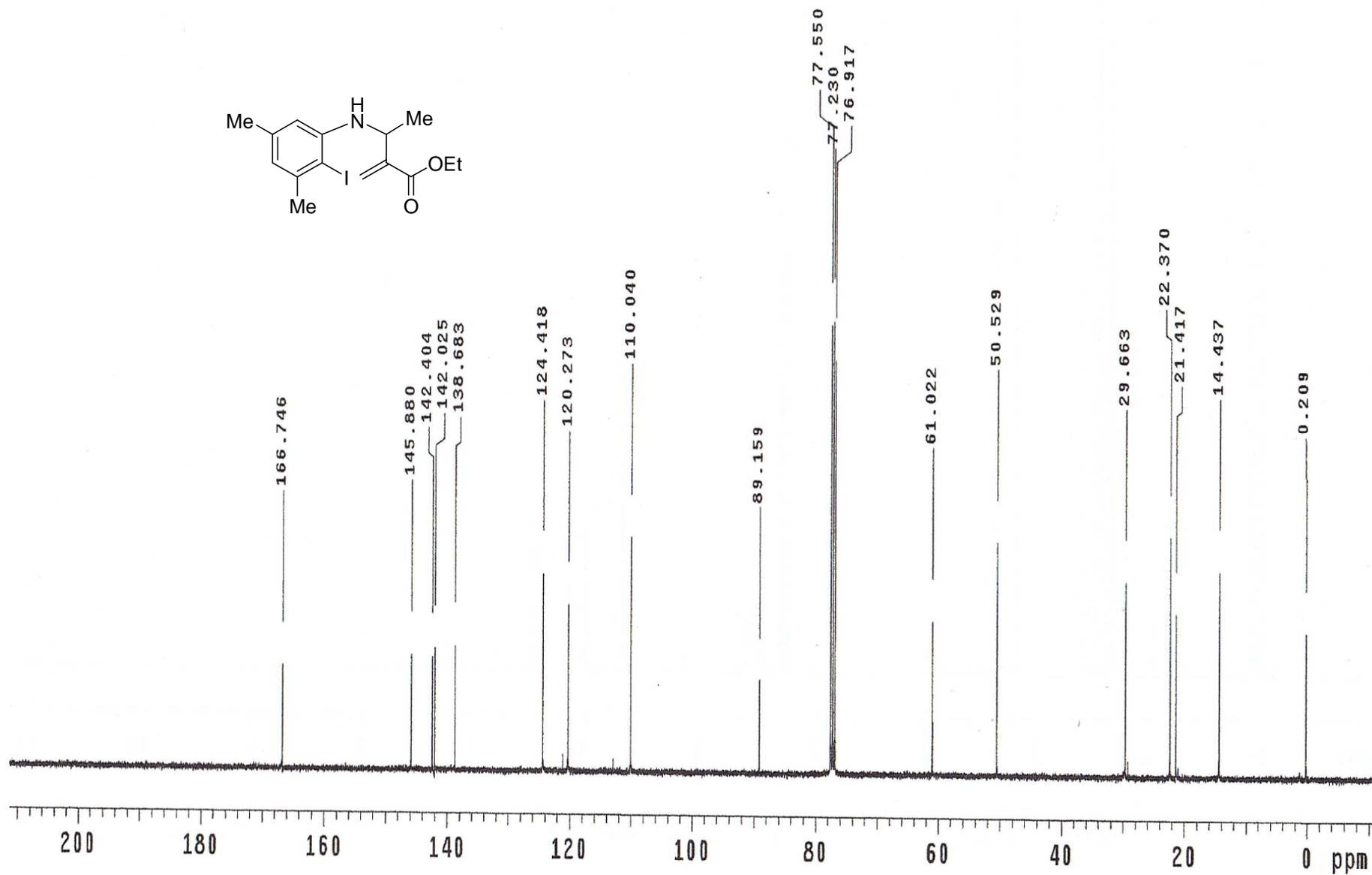
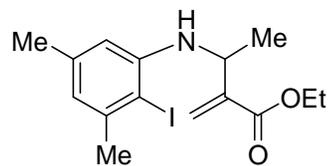
Ethyl 3-(2-iodo-4-methylphenylamino)-2-methylenebutanoate (5b): ^{13}C NMR (100 MHz, CDCl_3)

| | | |
|-----------|-------------|-------|
| DECOUPLER | wc | 240 |
| dn | H1 sc | 0 |
| dof | 0 vs | 31563 |
| dm | yyy th | 7 |
| dmm | w ai cdc ph | |
| dpwr | 41 | |
| dmf | 9648 | |



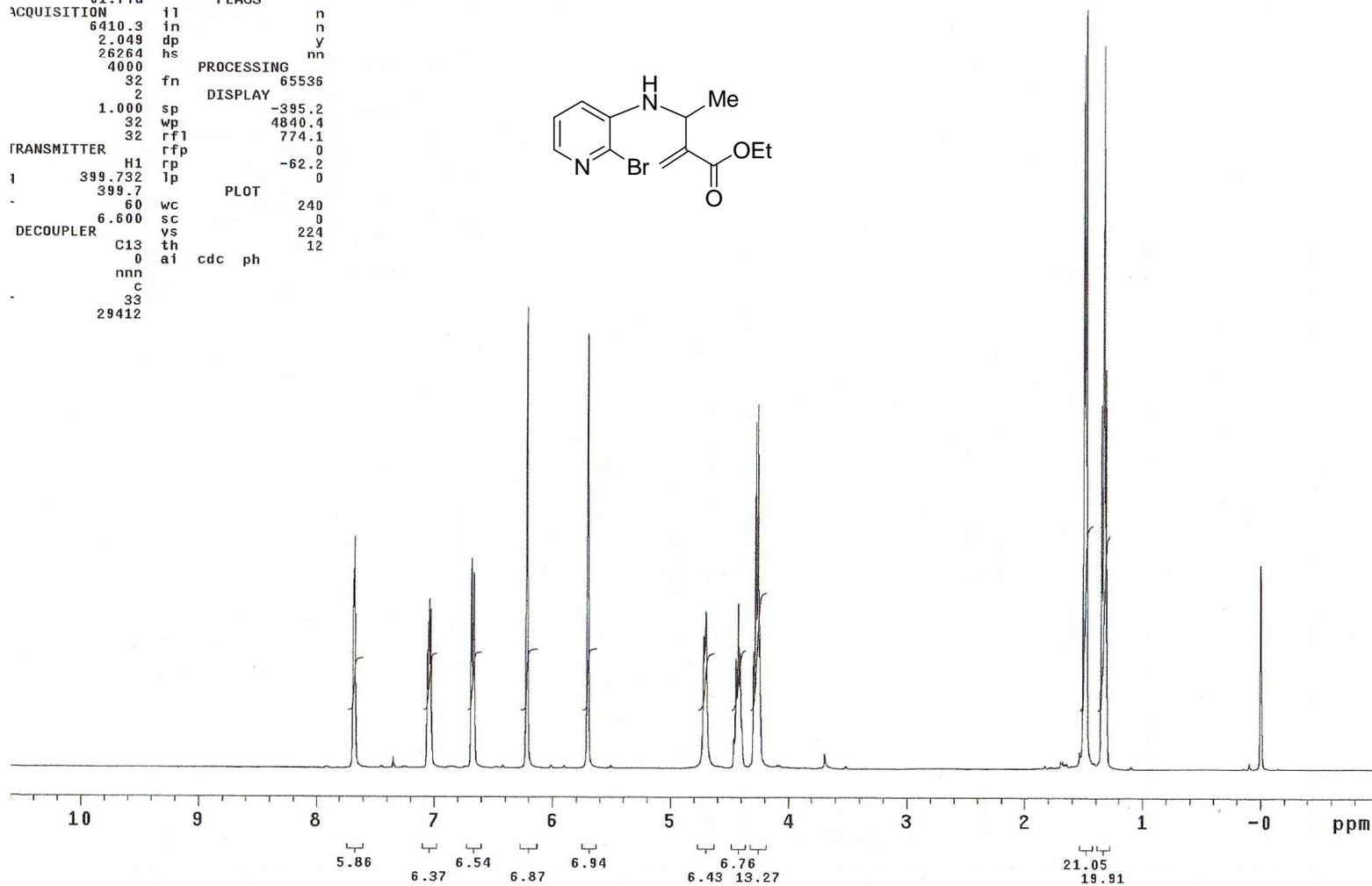
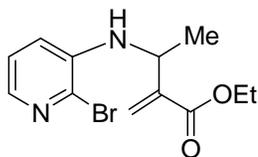
Ethyl 3-(2-iodo-3,5-dimethylphenylamino)-2-methylenebutanoate (6b): ^1H NMR (400 MHz, CDCl_3)

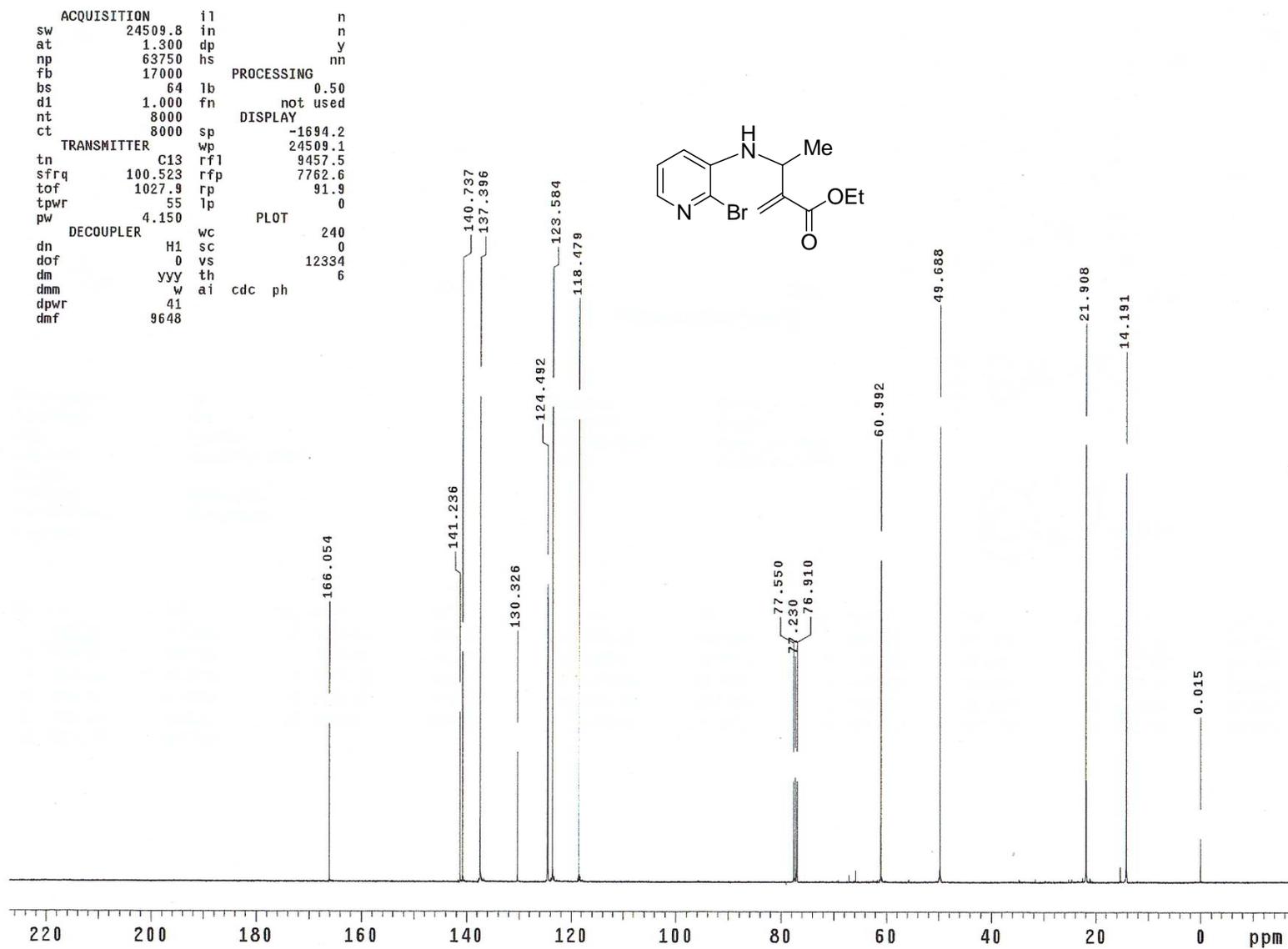
Ethyl 3-(2-iodo-3,5-dimethylphenylamino)-2-methylenebutanoate (6b): ^{13}C NMR (100 MHz, CDCl_3)

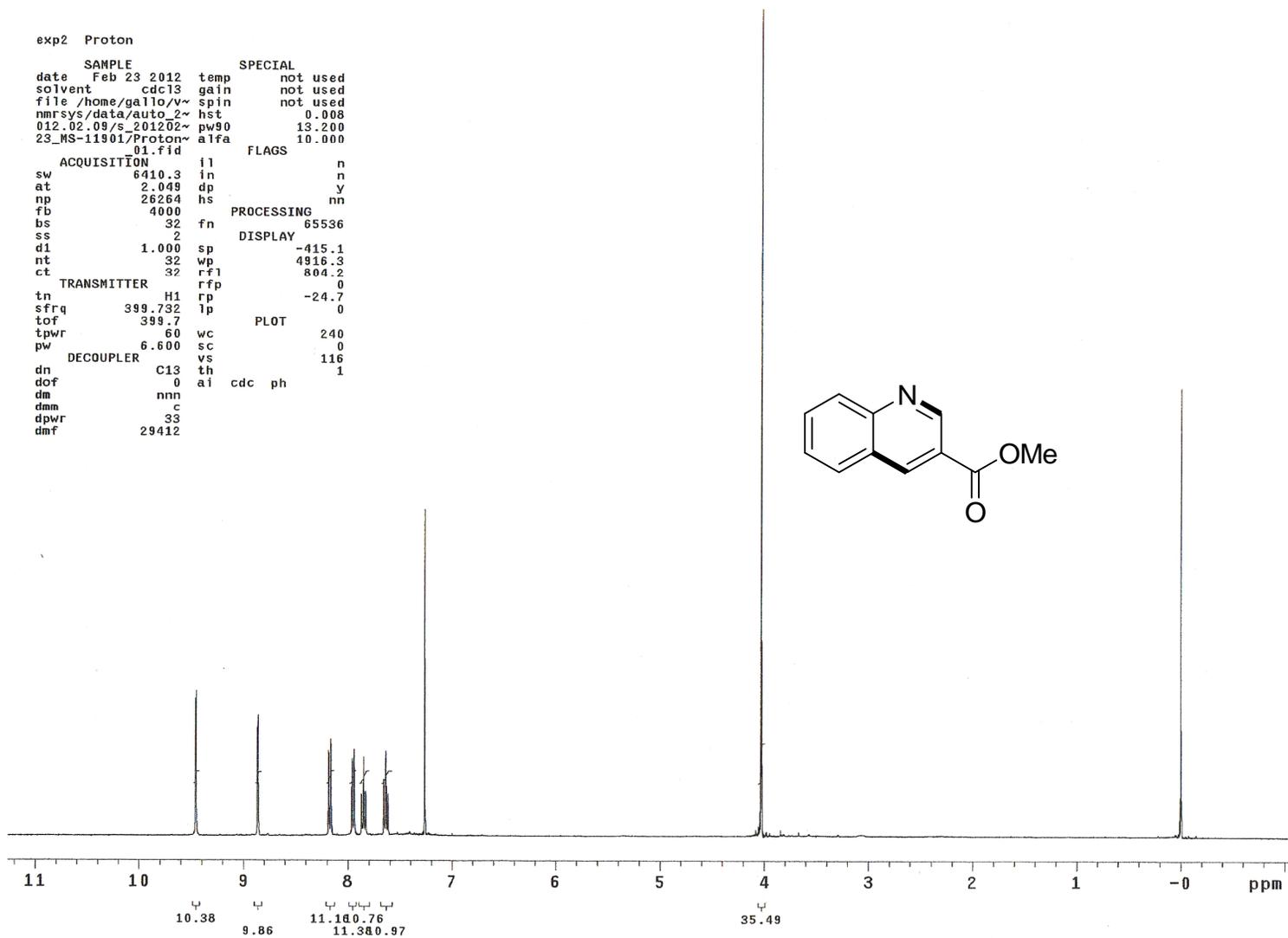


Ethyl 3-(2-bromopyridin-3-ylamino)-2-methylenebutanoate (7b): ^1H NMR (400 MHz, CDCl_3)

| | | | |
|------------------|------------|----------|--|
| SAMPLE | | SPECIAL | |
| 8 Sep 2 2014 | temp | 26.0 | |
| vent cdc13 | gain | not used | |
| s /home/gallo/v~ | spin | not used | |
| sys/data/auto_2~ | hst | 0.008 | |
| .01.14/s_201409~ | pw90 | 13.200 | |
| ?BM-801/Proton~ | alfa | 10.000 | |
| 01.fid | FLAGS | | |
| ACQUISITION | il | n | |
| 6410.3 | in | n | |
| 2.049 | dp | y | |
| 26264 | hs | nn | |
| 4000 | PROCESSING | | |
| 32 | fn | 65536 | |
| 2 | DISPLAY | | |
| 1.000 | sp | -395.2 | |
| 32 | wp | 4840.4 | |
| 32 | rf1 | 774.1 | |
| TRANSMITTER | rfl | 0 | |
| H1 | rpf | -62.2 | |
| 399.732 | lp | 0 | |
| 399.7 | PLOT | | |
| 60 | wc | 240 | |
| 6.600 | sc | 0 | |
| DECOUPLER | vs | 224 | |
| C13 | th | 12 | |
| 0 | ai | cdc ph | |
| nnn | | | |
| c | | | |
| 33 | | | |
| 29412 | | | |

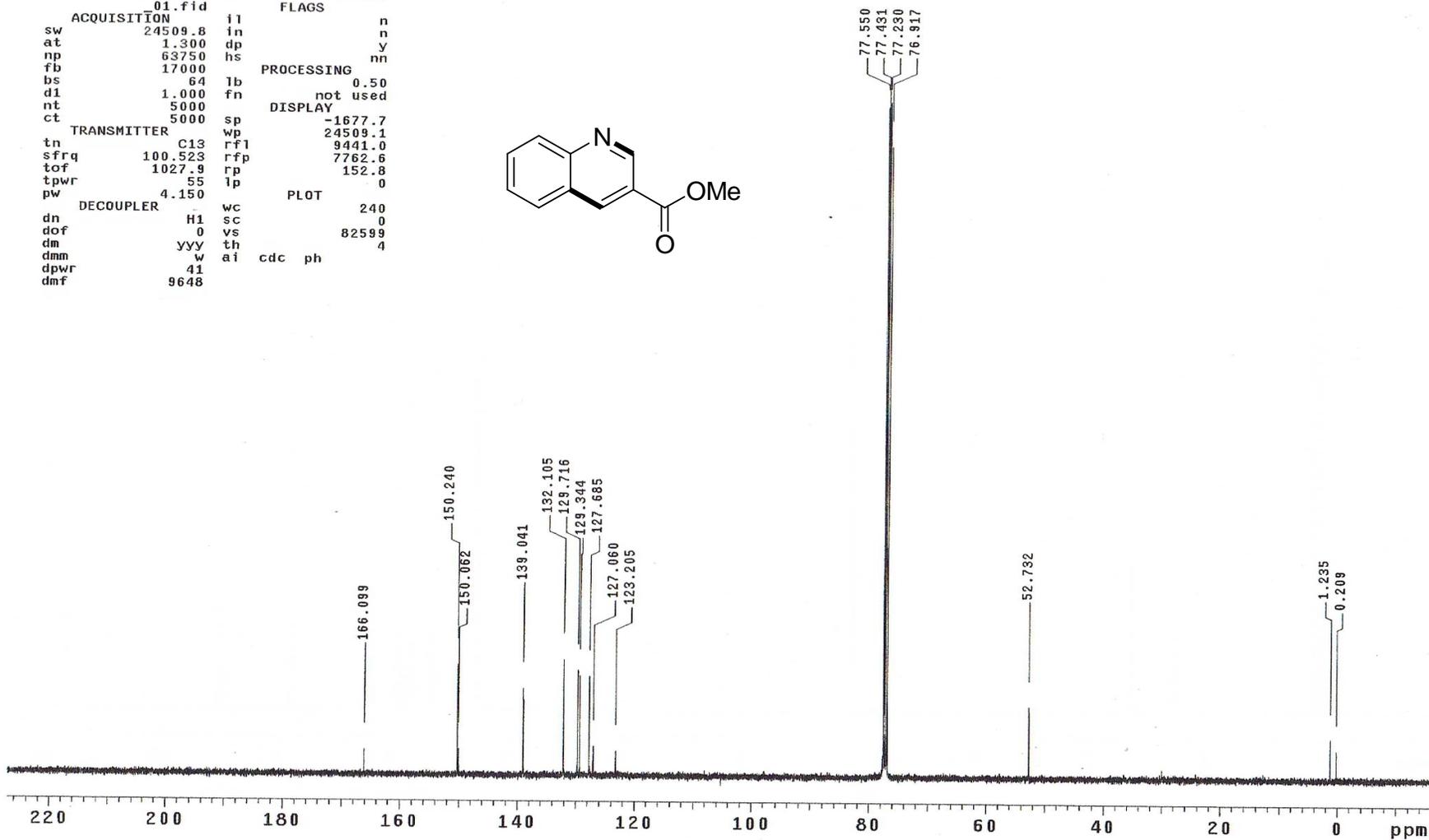
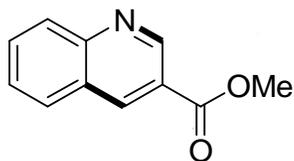


Ethyl 3-(2-bromopyridin-3-ylamino)-2-methylenebutanoate (**7b**): ^{13}C NMR (100 MHz, CDCl_3)

Methyl quinoline-3-carboxylate (1a'): ^1H NMR (400 MHz, CDCl_3)

Methyl quinoline-3-carboxylate (1a'): ^{13}C NMR (100 MHz, CDCl_3)

| | | | |
|---------------------|------------------|------------|----------|
| SAMPLE | | SPECIAL | |
| date | Feb 23 2012 | temp | not used |
| solvent | cdc13 | gain | 30 |
| file | /home/gallo/v~ | spin | not used |
| nmr | sys/data/auto_2~ | hst | 0.008 |
| 012_02_09/s_201202~ | | pw90 | 8.300 |
| 23_MS-11902/Carbon~ | alfa | 10.000 | |
| 01.fid | | FLAGS | |
| ACQUISITION | | il | n |
| sw | 24509.8 | in | n |
| at | 1.300 | dp | y |
| np | 63750 | hs | nn |
| fb | 17000 | PROCESSING | |
| bs | 64 | lb | 0.50 |
| d1 | 1.000 | fn | not used |
| nt | 5000 | DISPLAY | |
| ct | 5000 | sp | -1677.7 |
| TRANSMITTER | | wp | 24509.1 |
| tn | C13 | rfl | 9441.0 |
| sfrq | 100.523 | rfl | 7762.6 |
| tof | 1027.9 | rp | 152.8 |
| tpwr | 55 | lp | 0 |
| pw | 4.150 | PLOT | |
| DECOUPLER | | wc | 240 |
| dn | H1 | sc | 0 |
| dof | 0 | vs | 82599 |
| dm | yyy | th | 4 |
| dmm | w | ai | cdc ph |
| dpwr | 41 | | |
| dmf | 9648 | | |

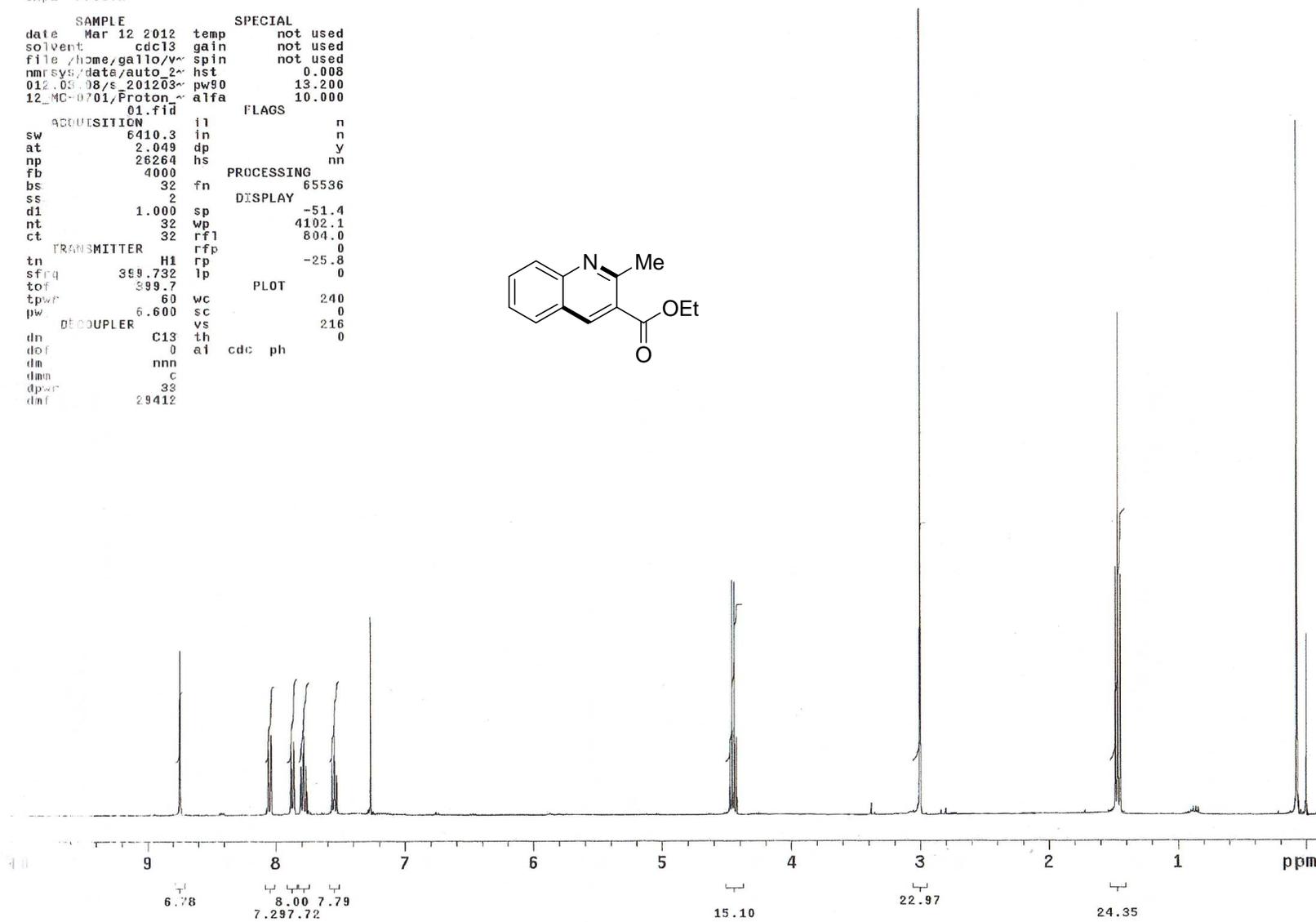
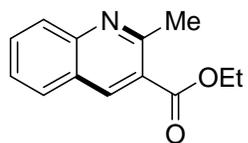


Ethyl 2-methylquinoline-3-carboxylate (1b', 2b'): ¹H NMR (400 MHz, CDCl₃)

```

exp1 Proton
SAMPLE
date Mar 12 2012 temp not used
solvent cdc13 gain not used
file /home/gallo/v spin not used
nmr sys /data/auto_2 ist 0.008
012_09_08/s 201203 pw90 13.200
12_MC-0701/Proton~ alfa 10.000
01.fid FLAGS
ACQUISITION i1 n
sw 6410.3 in n
at 2.049 dp y
np 26264 hs nn
fb 4000 PROCESSING
bs 32 fn 65536
ss 2 DISPLAY
d1 1.000 sp -51.4
nt 32 wp 4102.1
ct 32 rfl 804.0
TRANSMITTER H1 rfp 0
tn sfreq 389.732 rp -25.8
tof 399.7 lp 0
tpwr 60 wc 240
pw 6.600 sc 0
DECOUPLER C13 th 216
dn 0 al cdc ph
dof nnn
dm c
dmp 33
dmf 29412

```

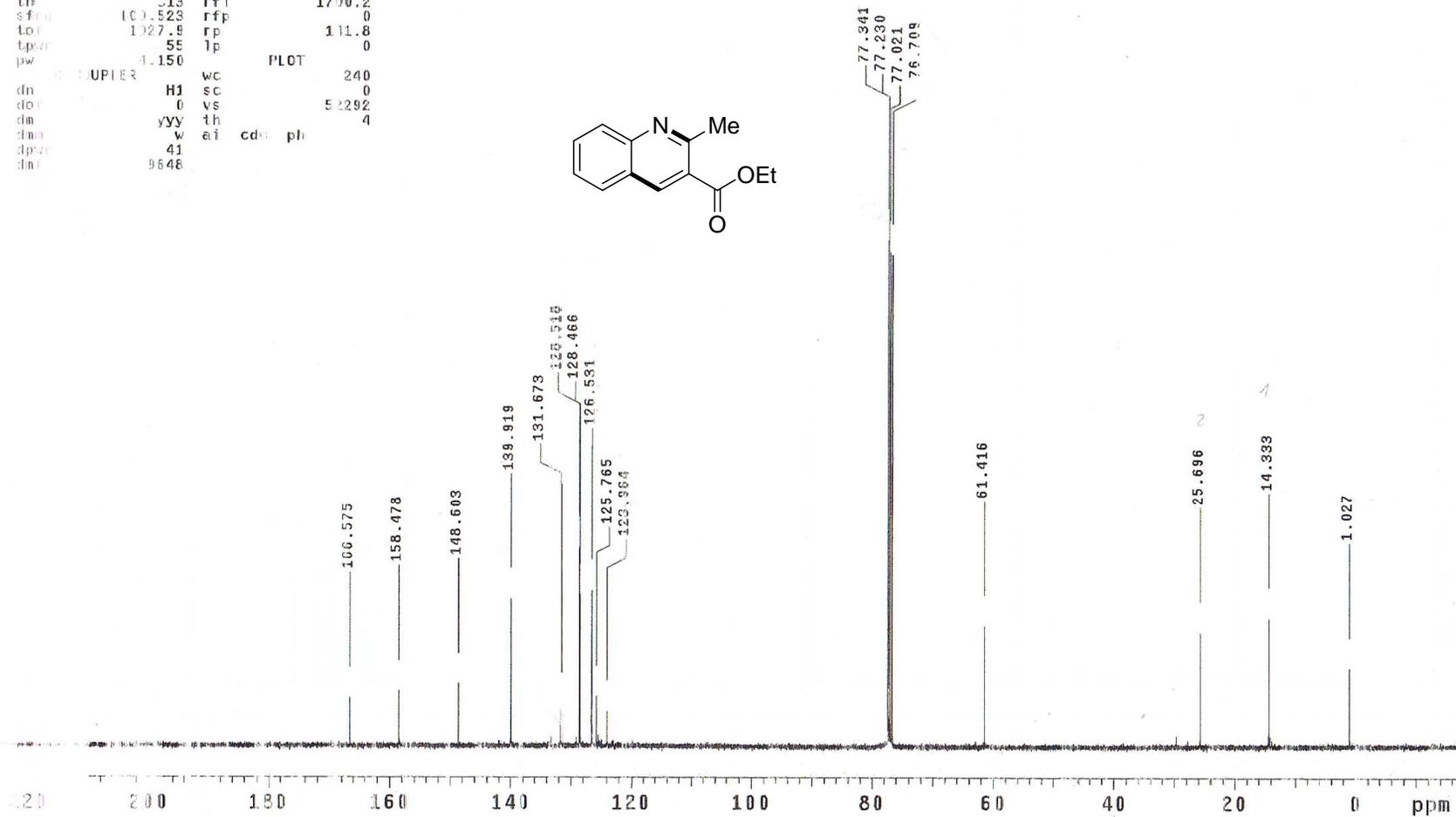


Ethyl 2-methylquinoline-3-carboxylate (1b', 2b'): ^{13}C NMR (100 MHz, CDCl_3)

```

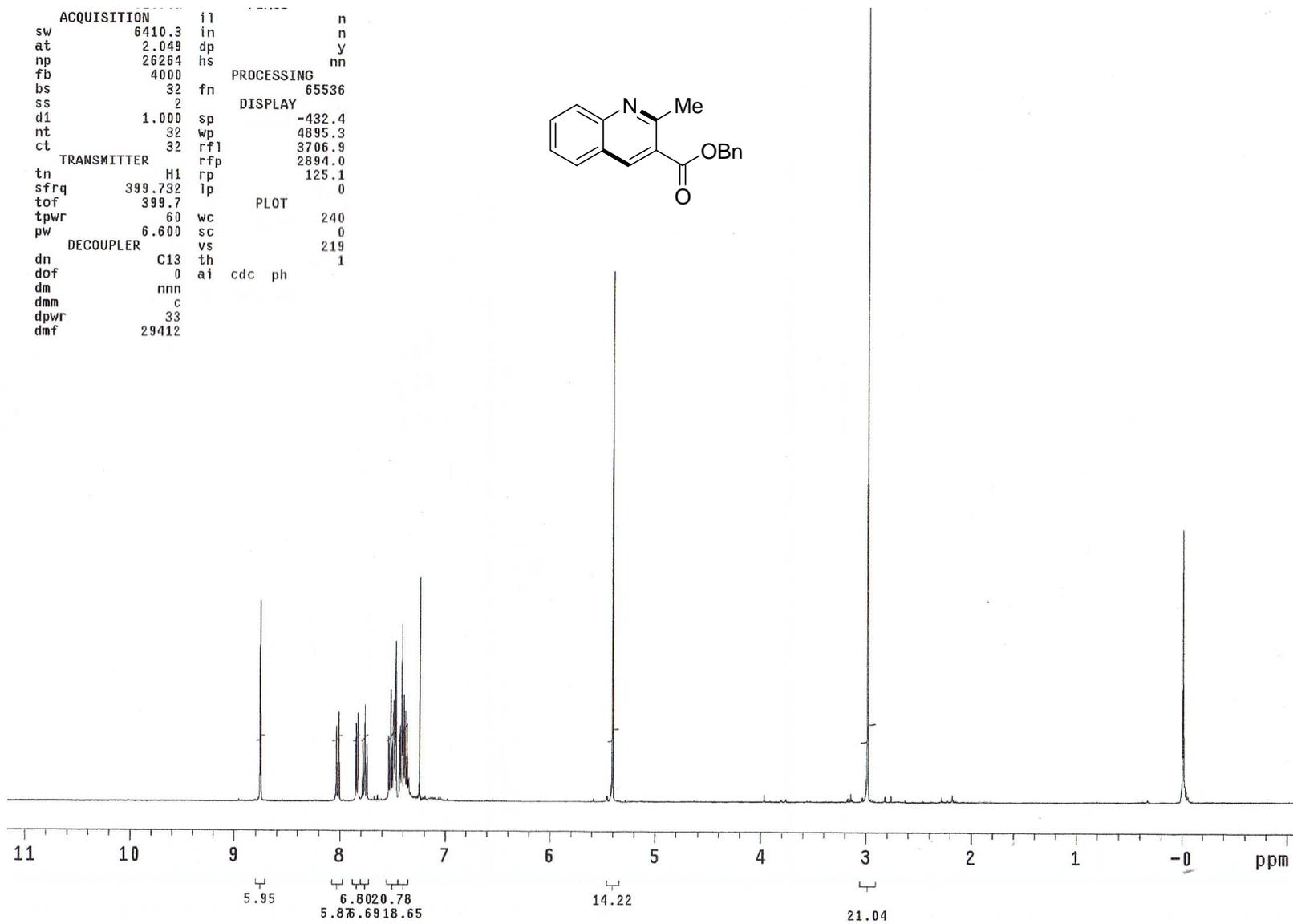
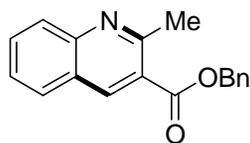
NAME POSITION      fl      n
sw          24509.8 in      n
at          1.300  dp      y
np          63750  hs      nn
fb          17000  PRCESSING
bs          64    lb      0.50
dl          1.000  fn      not used
nt          5000  DISPLAY
ct          5000  sp      -1699.4
          (P) M I T T I E R
tr          313   wp      24509.1
sfreq      100.523 rfp     1700.2
tol        1027.9 rp      111.8
tproc      55    lp      0
pw          4.150  PLOT
          (D) U P L I E R
dn          0     wc      240
dof         0     sc      0
dm          yy   vs      52292
dms         w    ai      4
dps         41   cd:    ph
dmf         9648

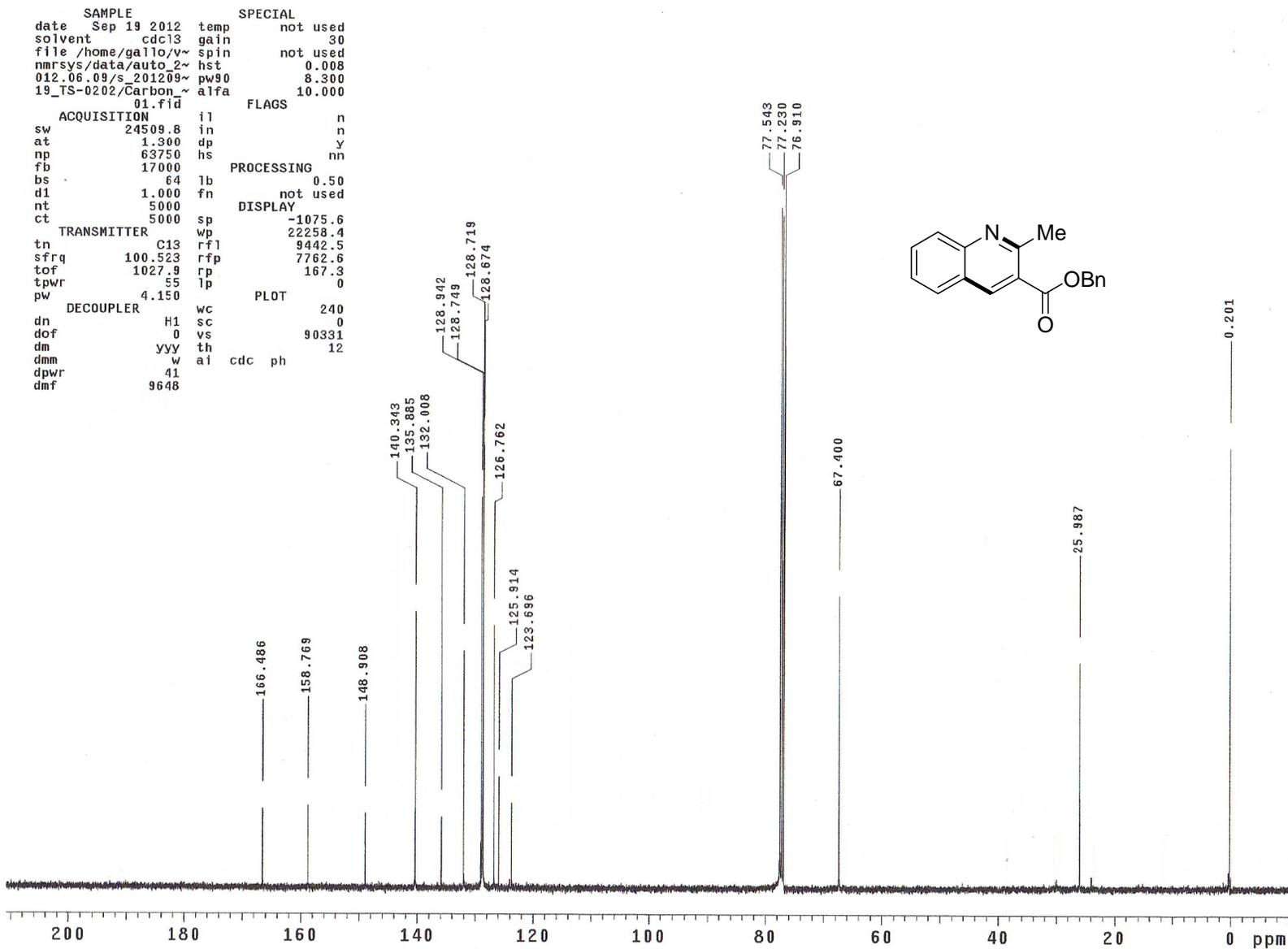
```

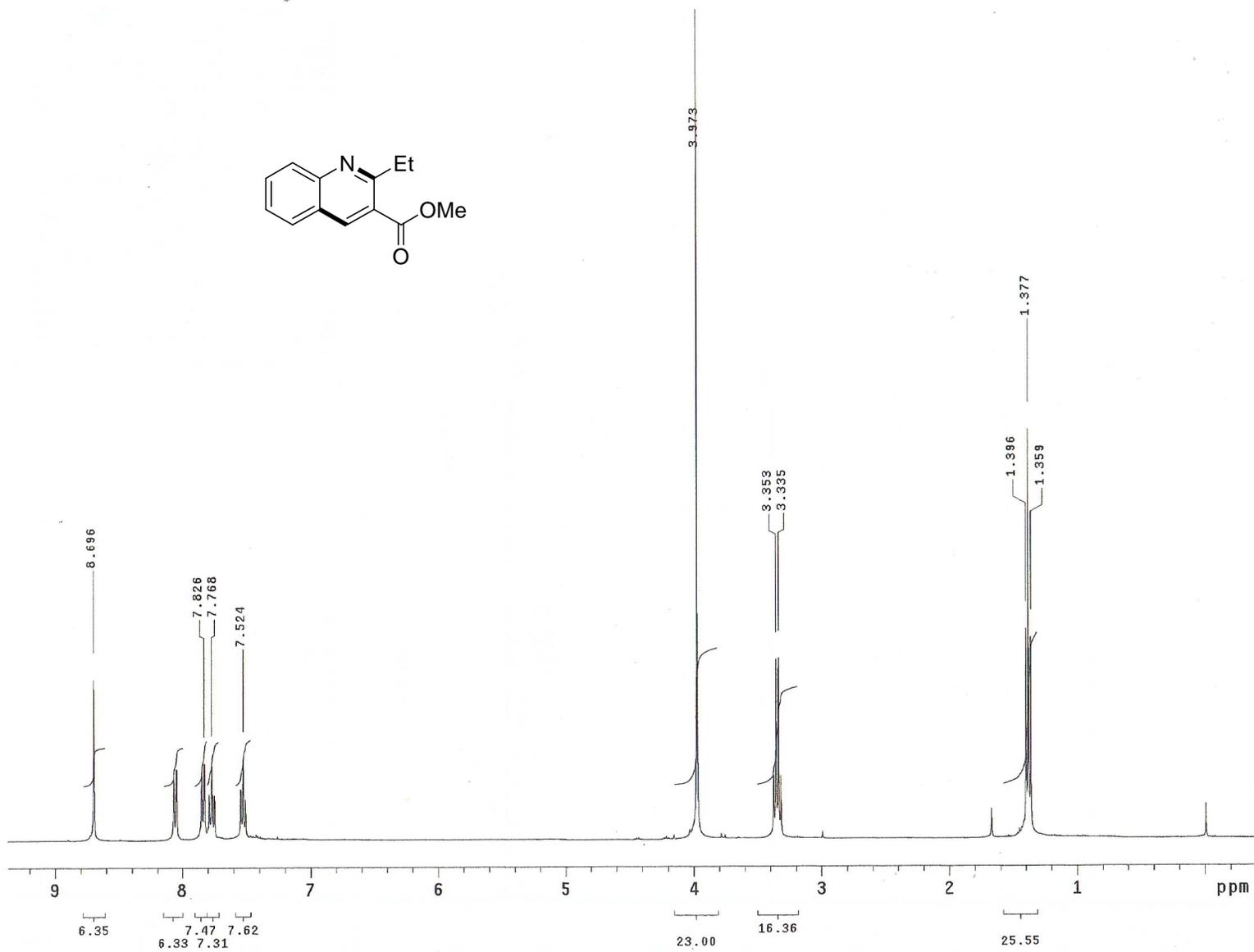


Benzyl 2-methylquinoline-3-carboxylate (1c'): ^1H NMR (400 MHz, CDCl_3)

```
ACQUISITION      il      n
sw      6410.3    in      n
at      2.049    dp      y
np      26264    hs      nn
fb      4000
bs      32      fn      65536
ss      2
d1      1.000    sp      -432.4
nt      32      wp      4895.3
ct      32      rfl     3706.9
TRANSMITTER      rfp     2894.0
tn      H1      rp     125.1
sfrq     399.732 lp      0
tof     399.7
tpwr     60      wc     240
pw     6.600    sc      0
DECOUPLER      vs     219
dn      C13    th      1
dof      0      ai    cdc ph
dm      nnn
dmm      c
dpwr     33
dmf     29412
```

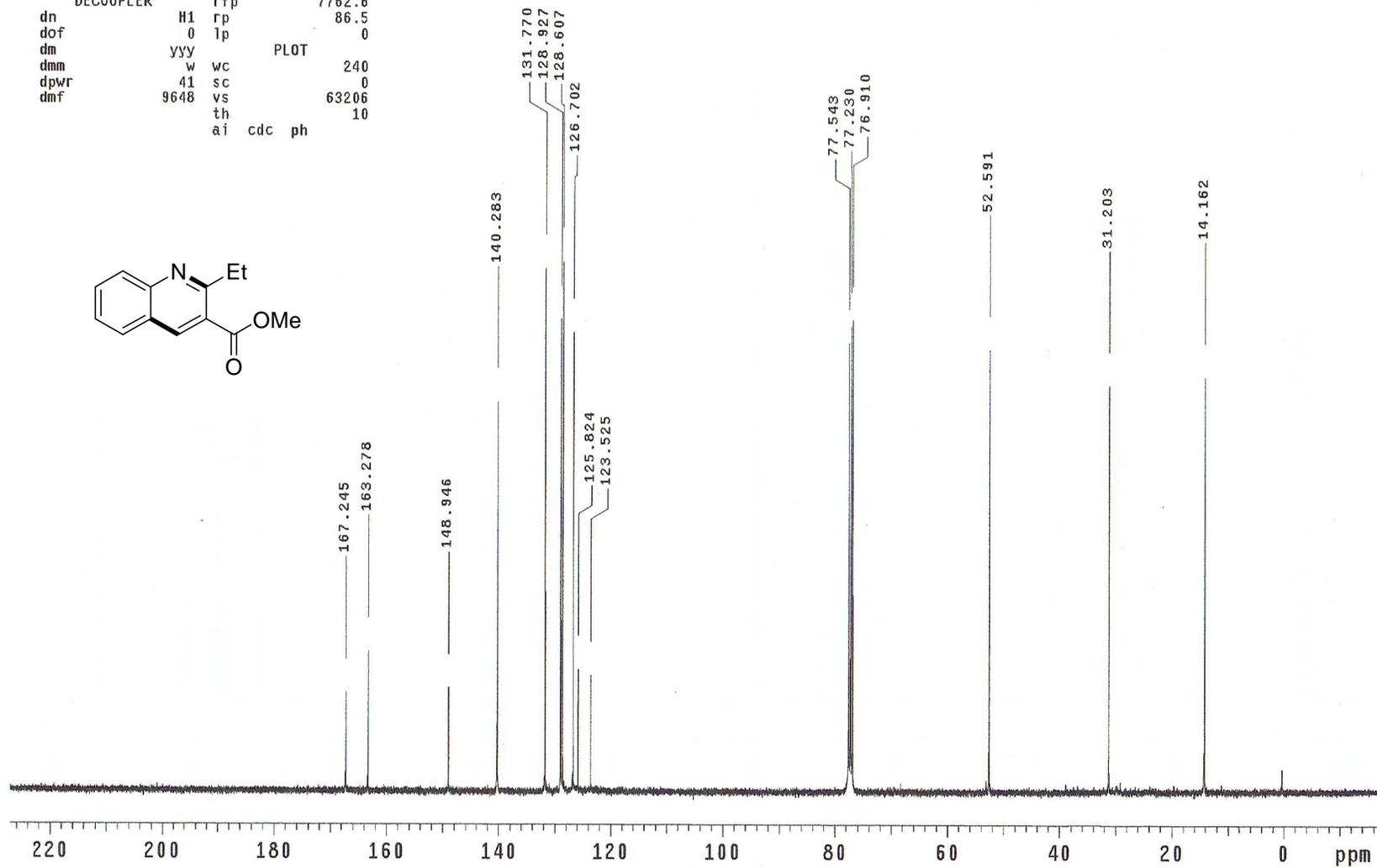
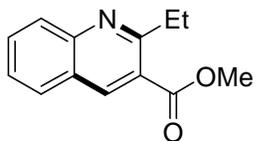


Benzyl 2-methylquinoline-3-carboxylate (1c'): ^{13}C NMR (100 MHz, CDCl_3)

Methyl 2-ethylquinoline-3-carboxylate (1d'): ^1H NMR (400 MHz, CDCl_3)

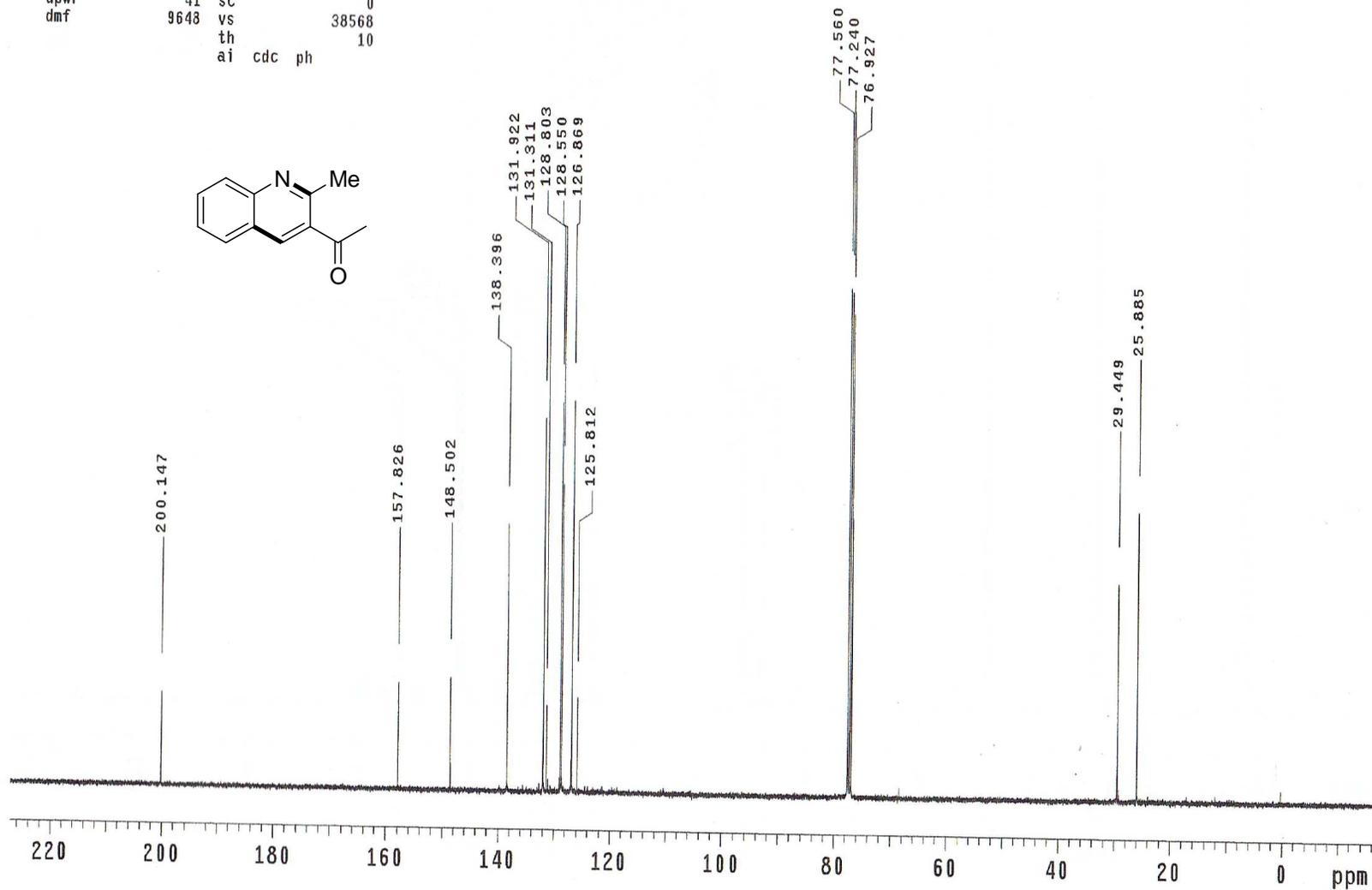
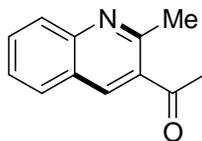
Methyl 2-ethylquinoline-3-carboxylate (1d'): ^{13}C NMR (100 MHz, CDCl_3)

| | | |
|-------------|---------|-------------|
| TRANSMITTER | lb | 0.50 |
| tn | C13 | fn not used |
| sfrq | 100.523 | DISPLAY |
| tof | 1027.9 | sp -1680.7 |
| tpwr | 55 | wp 24509.1 |
| pw | 4.150 | rfl 9444.0 |
| DECOUPLER | rffp | 7762.6 |
| dn | H1 | rp 86.5 |
| dof | 0 | lp 0 |
| dm | YYY | PLOT |
| dmm | w | wc 240 |
| dpwr | 41 | sc 0 |
| dmf | 9648 | vs 63206 |
| | th | 10 |
| | ai | cdc ph |



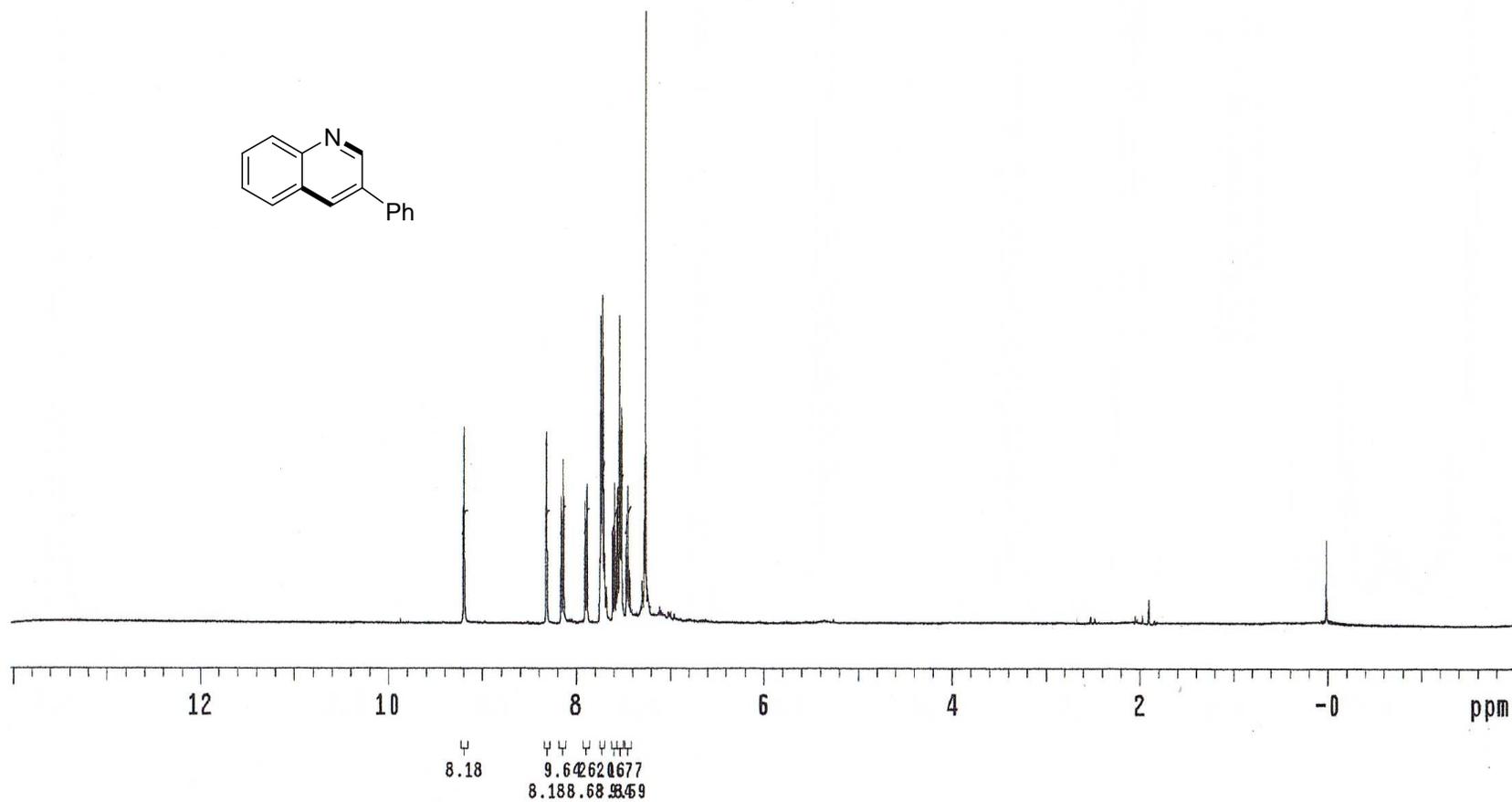
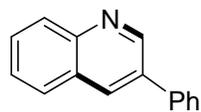
1-(2-methylquinolin-3-yl)ethanone (1e'): ^{13}C NMR (100 MHz, CDCl_3)

| | | | |
|-----------|------|--------|--------|
| DECOUPLER | | rfp | 7763.6 |
| dn | H1 | rp | 96.4 |
| dOf | 0 | lp | 0 |
| dm | YYY | PL0T | |
| dmm | w | WC | 240 |
| dpwr | 41 | sc | 0 |
| dmf | 9648 | vs | 38568 |
| | | th | 10 |
| | ai | cdc ph | |



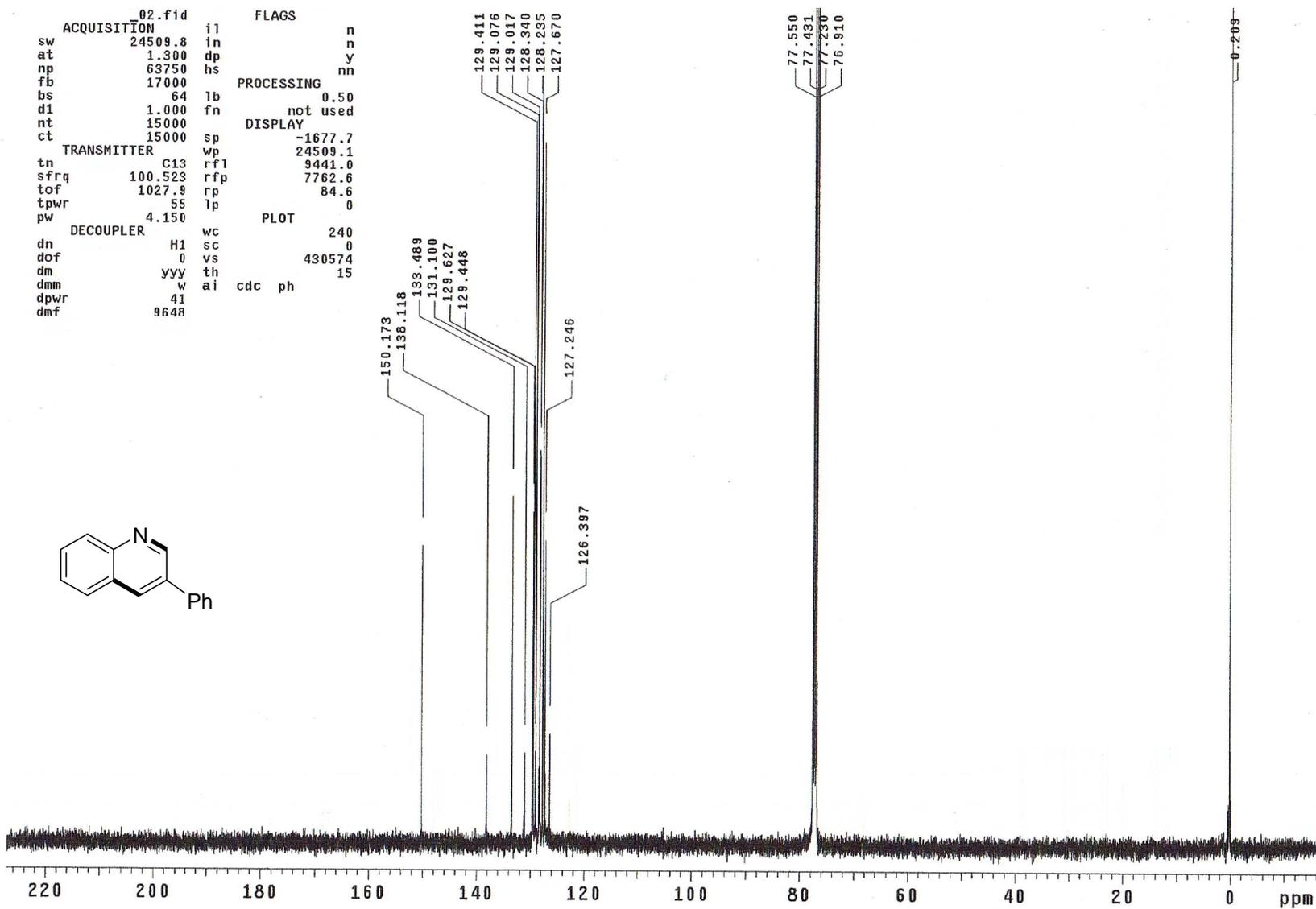
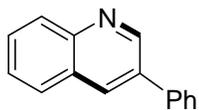
3-Phenylquinoline (1f'): ^1H NMR (400 MHz, CDCl_3)

```
DECOUPLER vs 529  
dn C13 th 8  
dof 0 ai cdc ph  
dm nnn  
dmm c  
dpwr 33  
dmf 29412
```



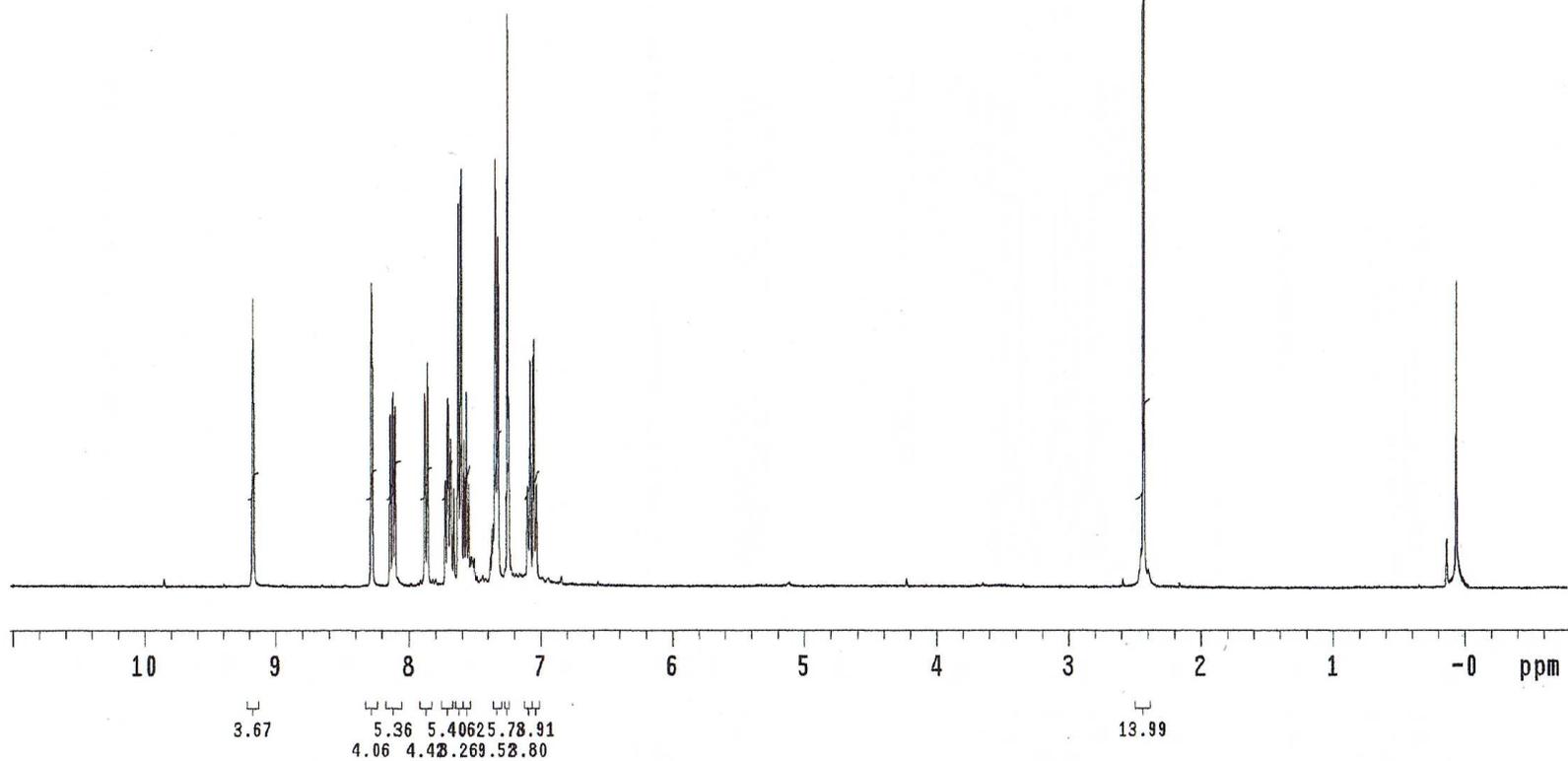
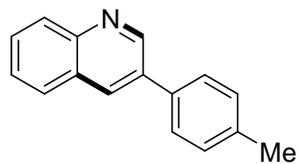
3-Phenylquinoline (1f'): ^{13}C NMR (100 MHz, CDCl_3)

| | | | |
|-------------|---------|---------|----------|
| _02.fid | | FLAGS | |
| ACQUISITION | i1 | n | |
| sw | 24509.8 | in | n |
| at | 1.300 | dp | y |
| np | 63750 | hs | nn |
| fb | 17000 | | |
| bs | 64 | lb | 0.50 |
| d1 | 1.000 | fn | not used |
| nt | 15000 | DISPLAY | |
| ct | 15000 | sp | -1677.7 |
| TRANSMITTER | wp | 24509.1 | |
| tn | C13 | rfl | 9441.0 |
| sfrq | 100.523 | rfp | 7762.6 |
| tof | 1027.9 | rp | 84.6 |
| tpwr | 55 | lp | 0 |
| pw | 4.150 | PLOT | |
| DECOUPLER | wc | 240 | |
| dn | H1 | sc | 0 |
| dof | 0 | vs | 430574 |
| dm | YY | th | 15 |
| dmm | w | ai | cdc ph |
| dpwr | 41 | | |
| dmf | 9648 | | |



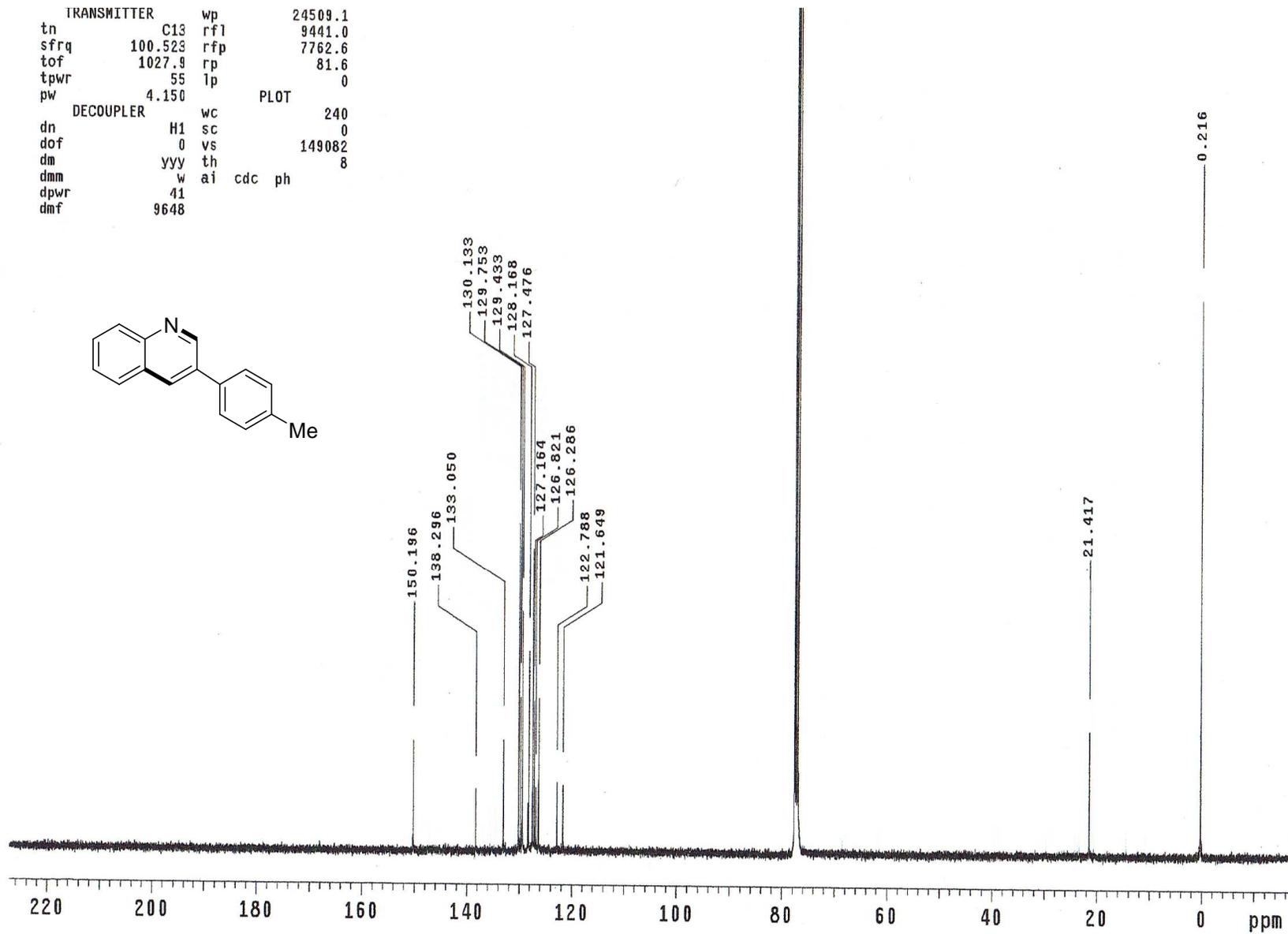
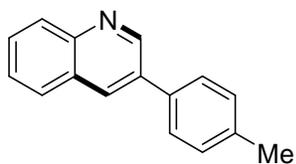
3-p-Tolylquinoline (1g'): ^1H NMR (400 MHz, CDCl_3)

| | | |
|-------------|---------|--------|
| TRANSMITTER | rfp | 2894.0 |
| tn | H1 | -60.7 |
| sfrq | 399.732 | lp |
| tof | 399.7 | PLOT |
| tpwr | 60 | wc |
| pw | 6.600 | sc |
| DECOUPLER | C13 | vs |
| dn | 0 | th |
| dof | 0 | ai |
| dm | nnn | cdc ph |
| dmm | c | |
| dpwr | 33 | |
| dmi | 29412 | |



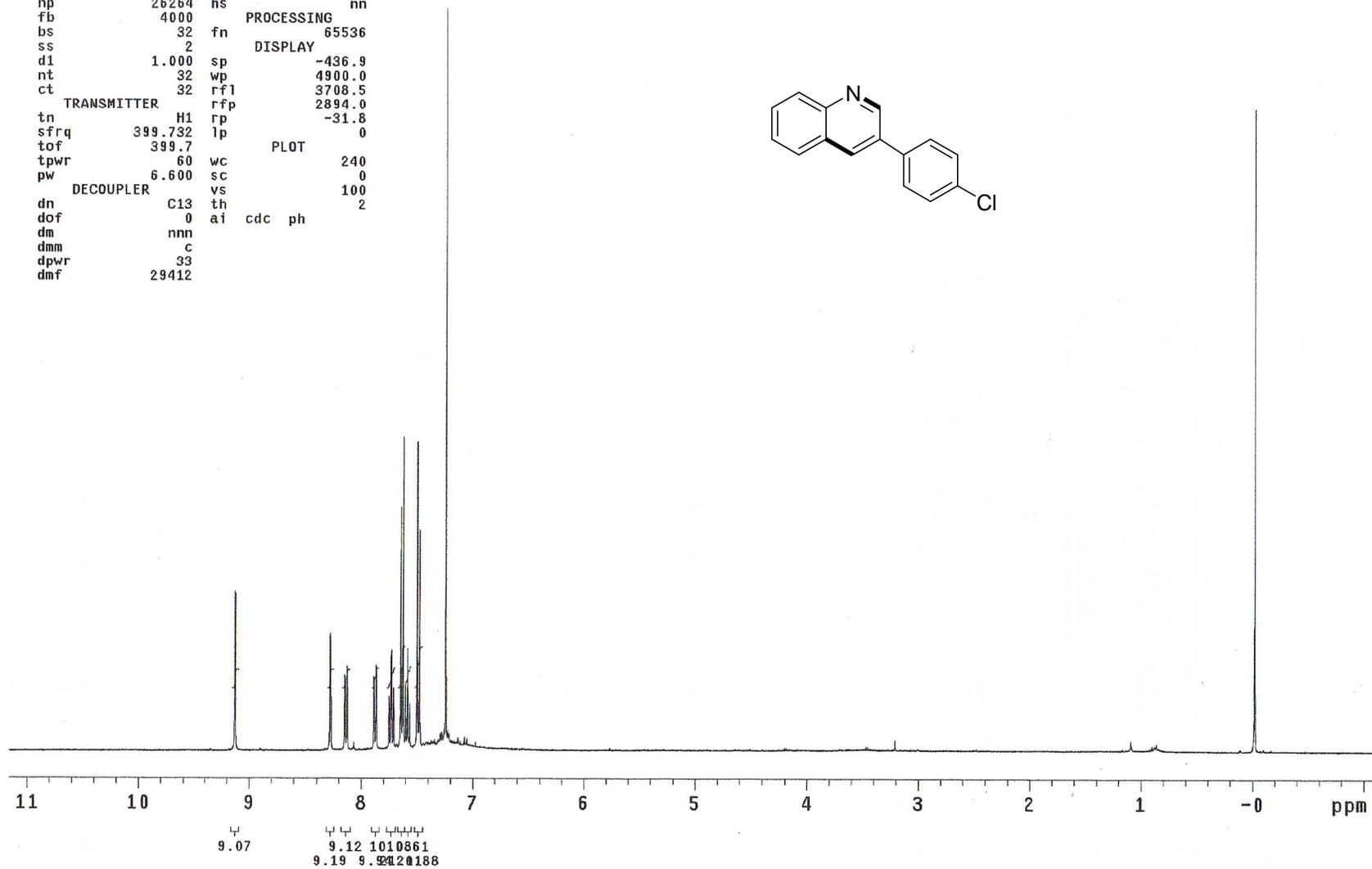
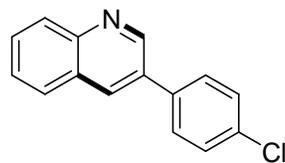
3-p-Tolylquinoline (1g'): ^{13}C NMR (100 MHz, CDCl_3)

| | | |
|-------------|---------|------------|
| TRANSMITTER | wp | 24509.1 |
| tn | C13 | rf1 9441.0 |
| sfrq | 100.523 | rfl 7762.6 |
| tof | 1027.9 | rp 81.6 |
| tpwr | 55 | lp 0 |
| pw | 4.150 | PLOT |
| DECOUPLER | wc | 240 |
| dn | H1 | sc 0 |
| dof | 0 | vs 149082 |
| dm | yyy | th 8 |
| dmm | w | ai cdc ph |
| dpwr | 41 | |
| dmf | 9648 | |



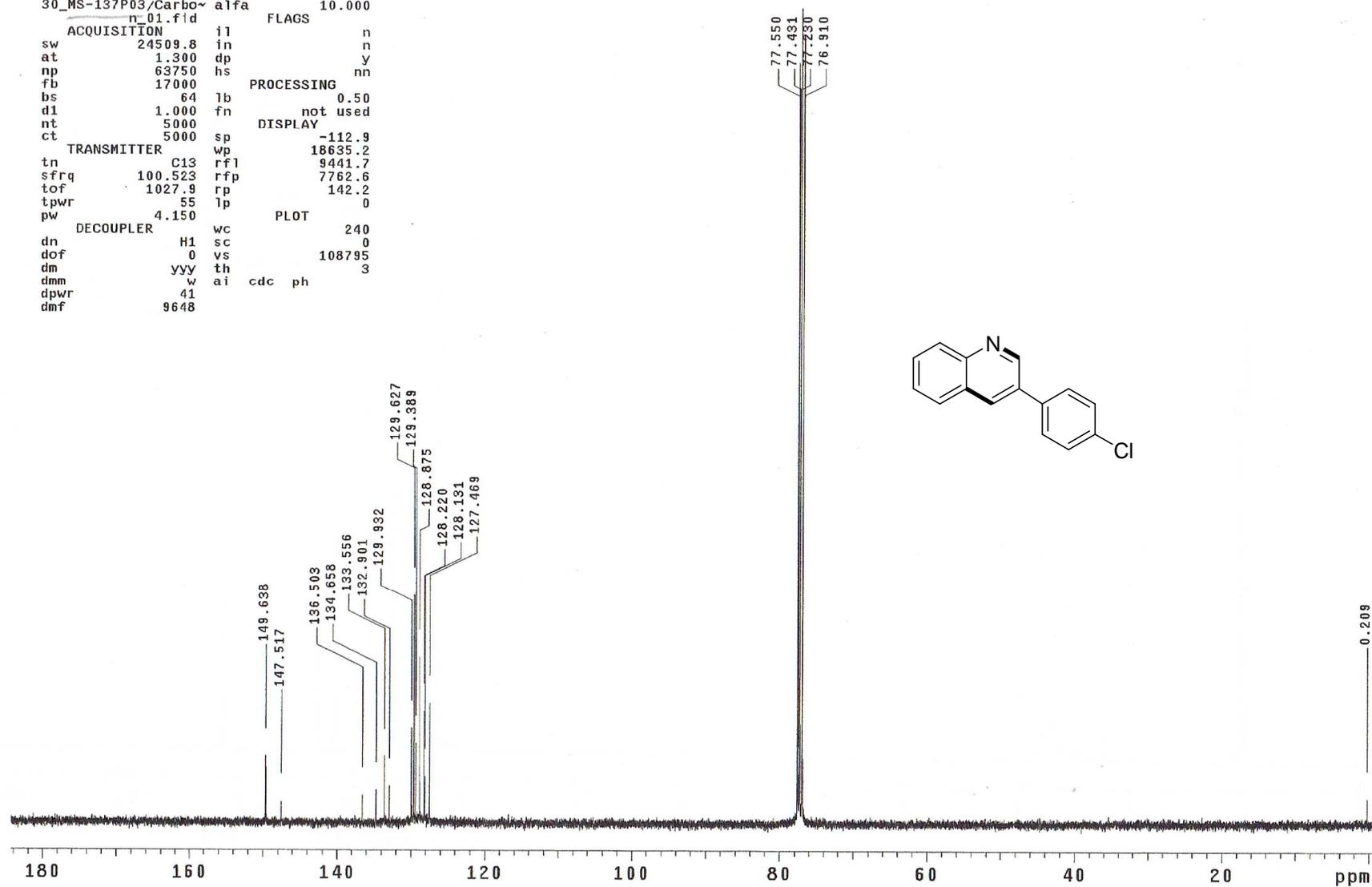
3-(4-chlorophenyl)quinoline (1h'): ^1H NMR (400 MHz, CDCl_3)

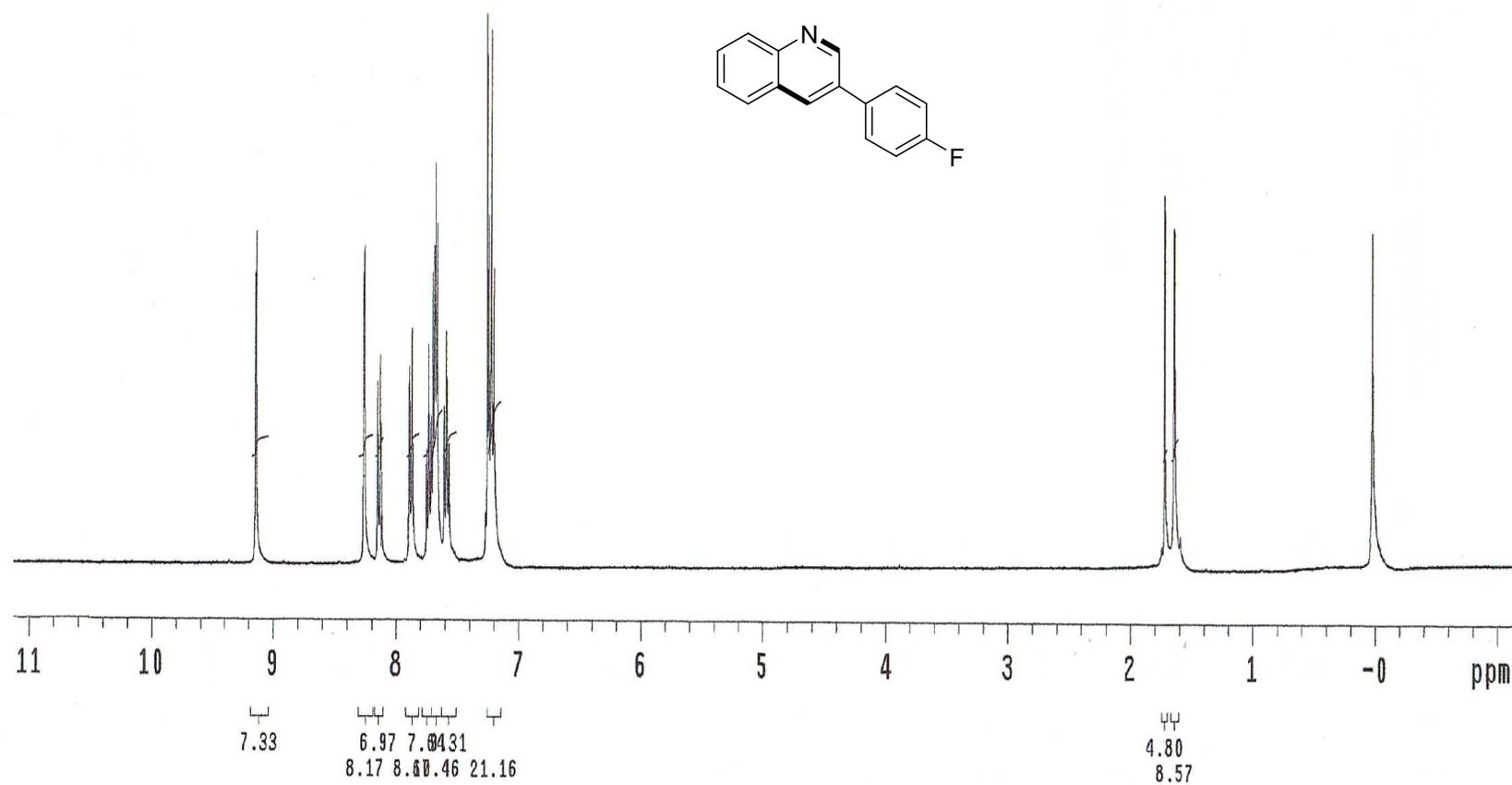
| | | |
|-------------|-----------|------------|
| ACQUISITION | i1 | n |
| sw | 6410.3 | in |
| at | 2.049 | dp |
| np | 26264 | hs |
| fb | 4000 | nn |
| bs | 32 | fn |
| ss | 2 | PROCESSING |
| d1 | 1.000 | sp |
| nt | 32 | wp |
| ct | 32 | rfl |
| TRANSMITTER | H1 | rff |
| tn | 399.732 | rp |
| sfrq | 399.7 | lp |
| tof | 60 | PLOT |
| tpwr | 6.600 | wc |
| pw | DECOUPLER | sc |
| dn | C13 | vs |
| dof | 0 | th |
| dm | nnn | ai |
| dmm | c | cdc |
| dpwr | 33 | ph |
| dmf | 29412 | |



3-(4-chlorophenyl)quinoline (1h'): ^{13}C NMR (100 MHz, CDCl_3)

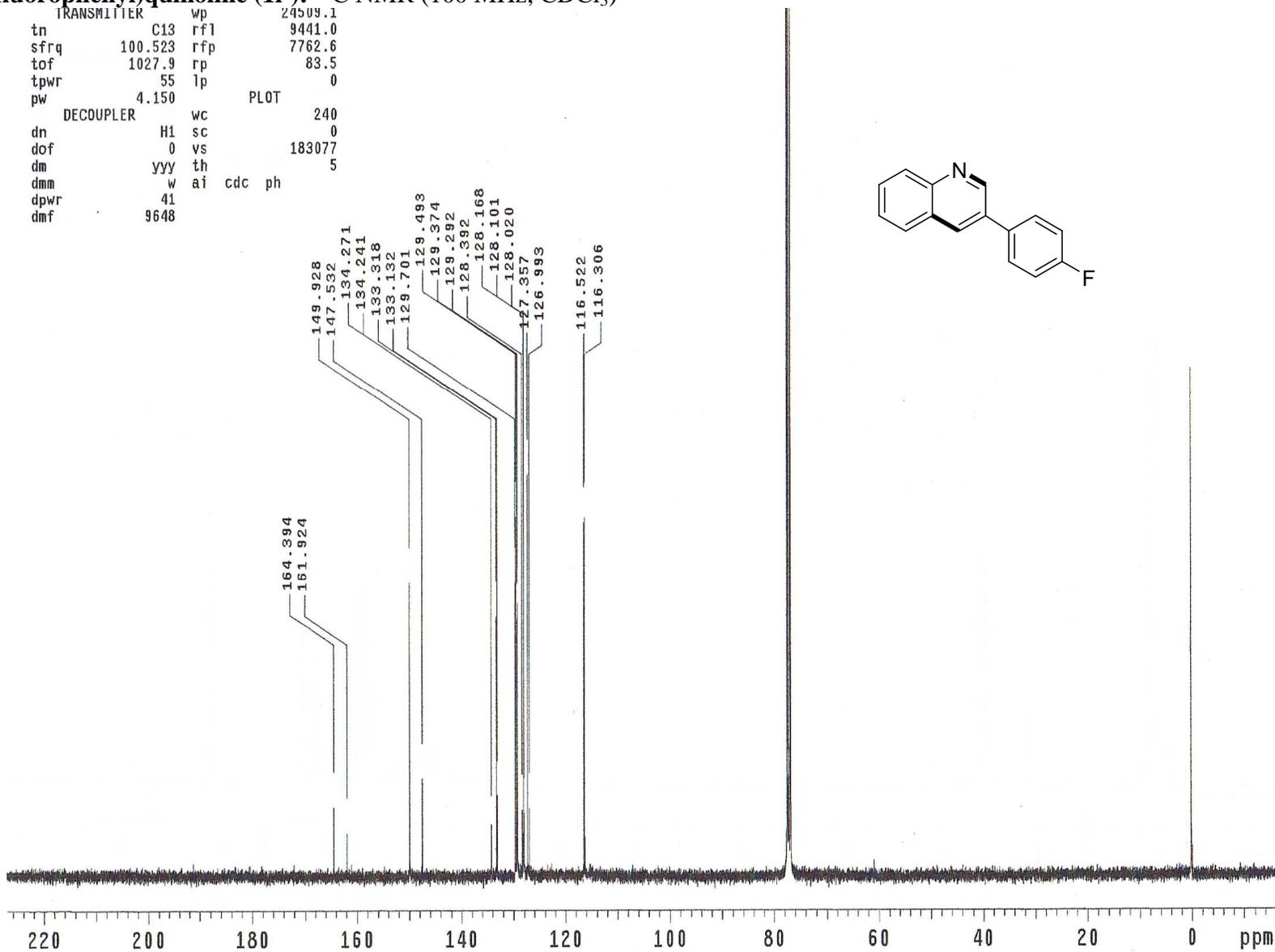
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nmrSYS/data/auto_2~ hst      0.008
012.04.30/s_201204~ pw90    8.300
30_MS-137P03/Carbo~ alfa    10.000
      n_01.fid      FLAGS
ACQUISITION      il      n
sw      24509.8    in      y
at      1.300     dp      y
np      63750    hs      nn
fb      17000
bs      64      lb      0.50
dl      1.000     fn      not used
nt      5000     DISPLAY
ct      5000     sp      -112.9
      TRANSMITTER      wp      18635.2
tn      C13      rfl      9441.7
sfrq    100.523   rfp      7762.6
tof     1027.9   rp      142.2
tpwr    55      lp      0
pw      4.150
      DECOUPLER      wc      240
dn      H1      sc      0
dof     0      vs      108795
dm      YVY    th      3
dmm     w      ai      cdc ph
dpwr    41
dmf     9648
```



3-(4-fluorophenyl)quinoline (1i'): ^1H NMR (400 MHz, CDCl_3)

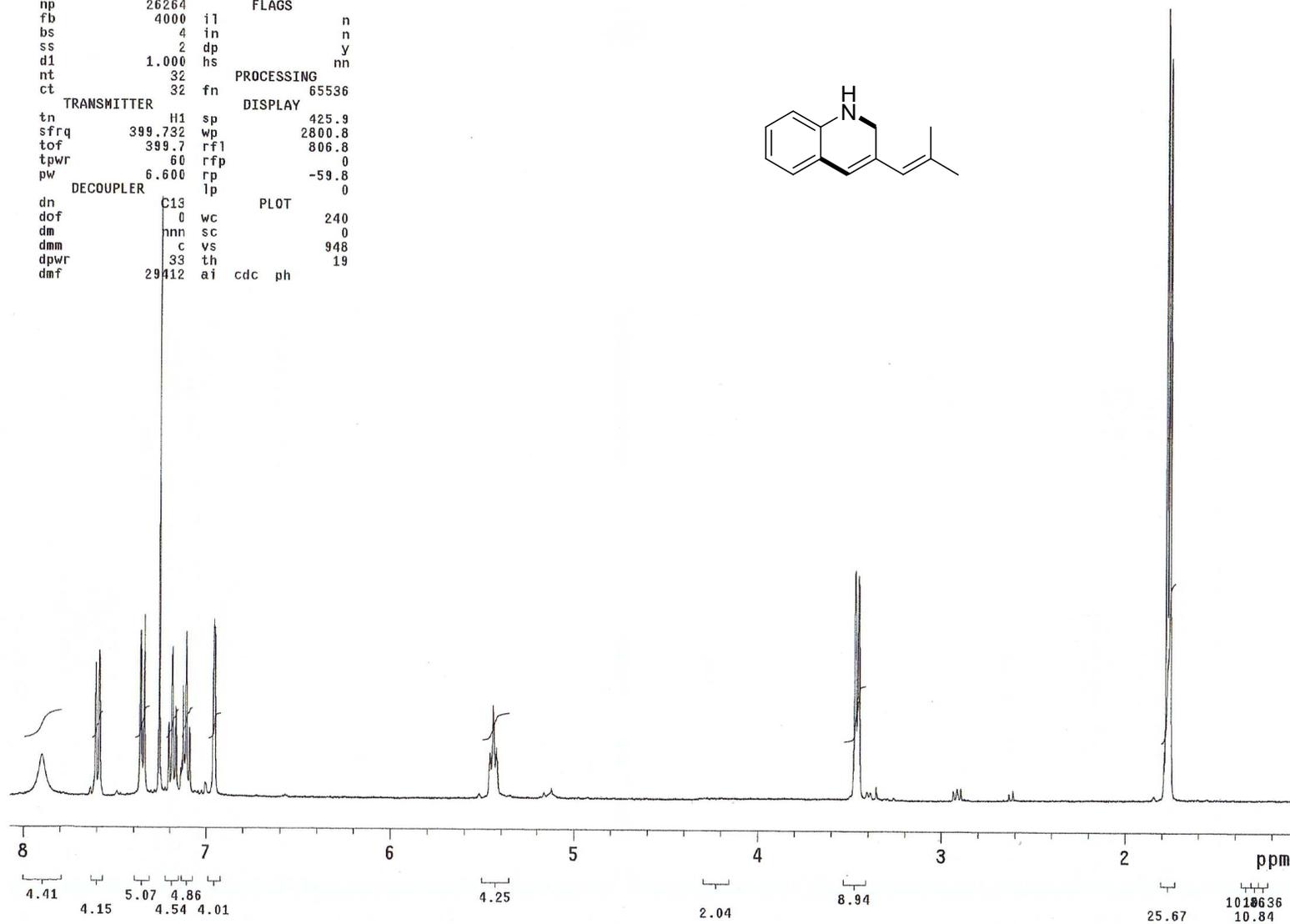
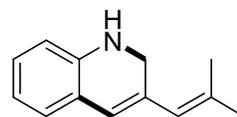
3-(4-fluorophenyl)quinoline (1i'): ^{13}C NMR (100 MHz, CDCl_3)

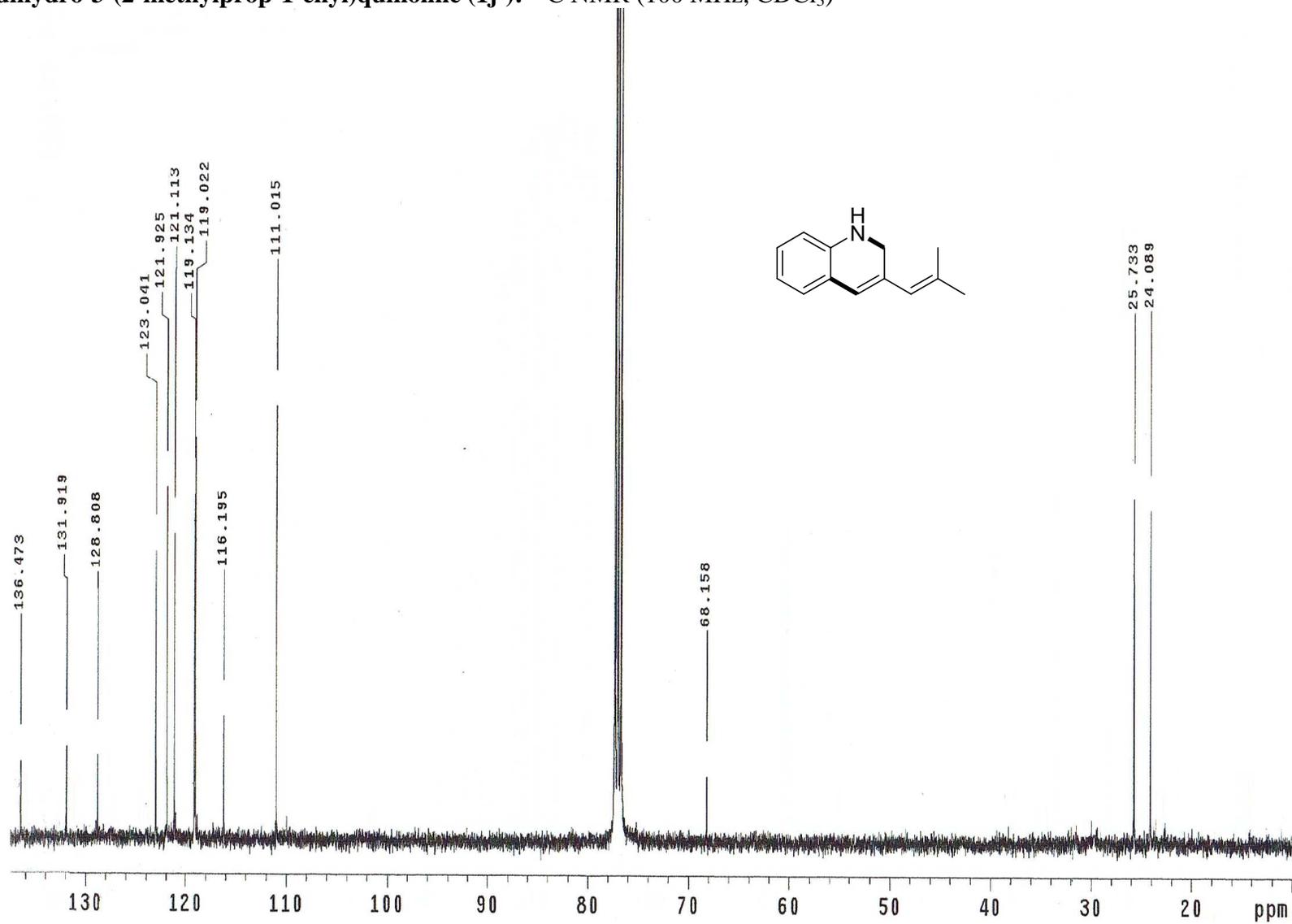
| | | | | |
|-------------|---------|---------|--------|----|
| TRANSMITTER | wp | 24509.1 | | |
| tn | C13 | rfl | 9441.0 | |
| sfrq | 100.523 | rfp | 7762.6 | |
| tof | 1027.9 | rp | 83.5 | |
| tpwr | 55 | lp | 0 | |
| pw | 4.150 | PLOT | | |
| DECOUPLER | wc | 240 | | |
| dn | H1 | sc | 0 | |
| dof | 0 | vs | 183077 | |
| dm | YYY | th | 5 | |
| dmm | w | ai | cdc | ph |
| dpwr | 41 | | | |
| dmf | 9648 | | | |

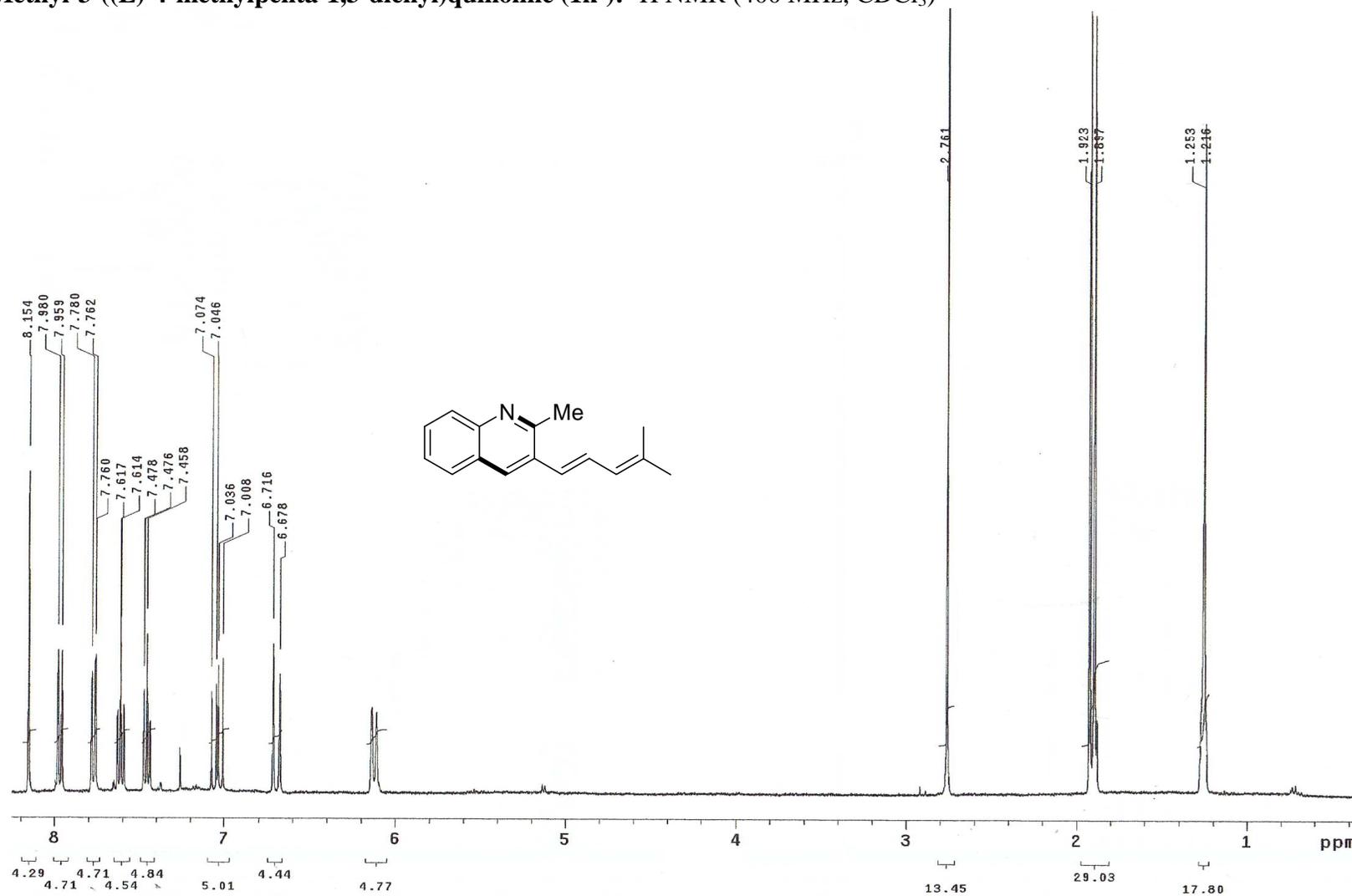


1,2-dihydro-3-(2-methylprop-1-enyl)quinoline (1j'): ^1H NMR (400 MHz, CDCl_3)

| | | | |
|-------------|---------|------------|--------|
| ACQUISITION | | hst | 0.008 |
| sw | 6410.3 | pw90 | 13.200 |
| at | 2.049 | alfa | 10.000 |
| np | 26264 | FLAGS | |
| fb | 4000 | il | n |
| bs | 4 | in | n |
| ss | 2 | dp | y |
| d1 | 1.000 | hs | nn |
| nt | 32 | PROCESSING | |
| ct | 32 | fn | 65536 |
| TRANSMITTER | | DISPLAY | |
| tn | H1 | sp | 425.9 |
| sfrq | 399.732 | wp | 2800.8 |
| tof | 399.7 | rfl | 806.8 |
| tpwr | 60 | rfp | 0 |
| pw | 6.600 | rp | -59.8 |
| DECOUPLER | | lp | 0 |
| dn | C13 | PLOT | |
| dof | 0 | wc | 240 |
| dm | nnn | sc | 0 |
| dmm | c | vs | 948 |
| dpwr | 33 | th | 19 |
| dmf | 29412 | ai | cdc ph |

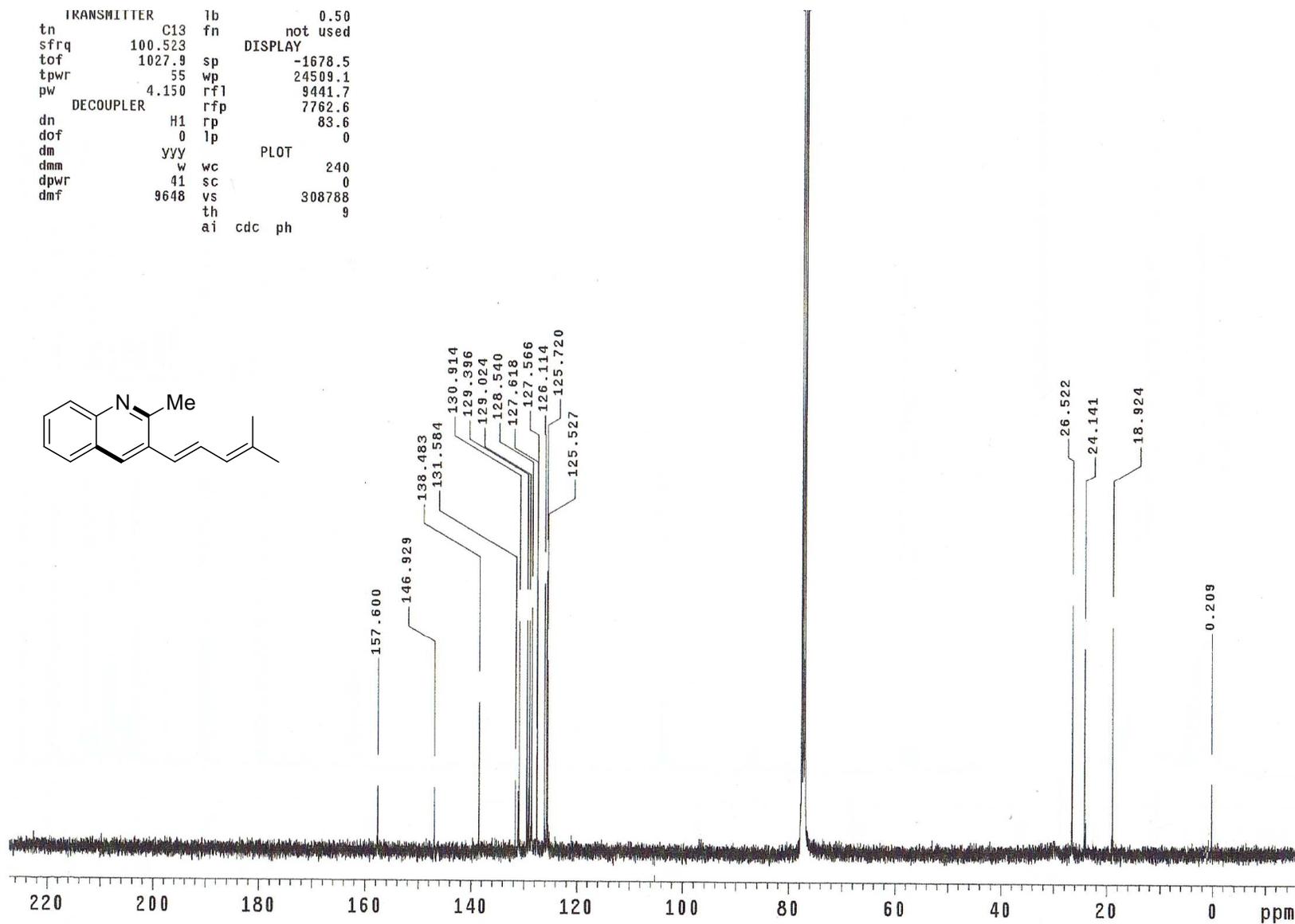


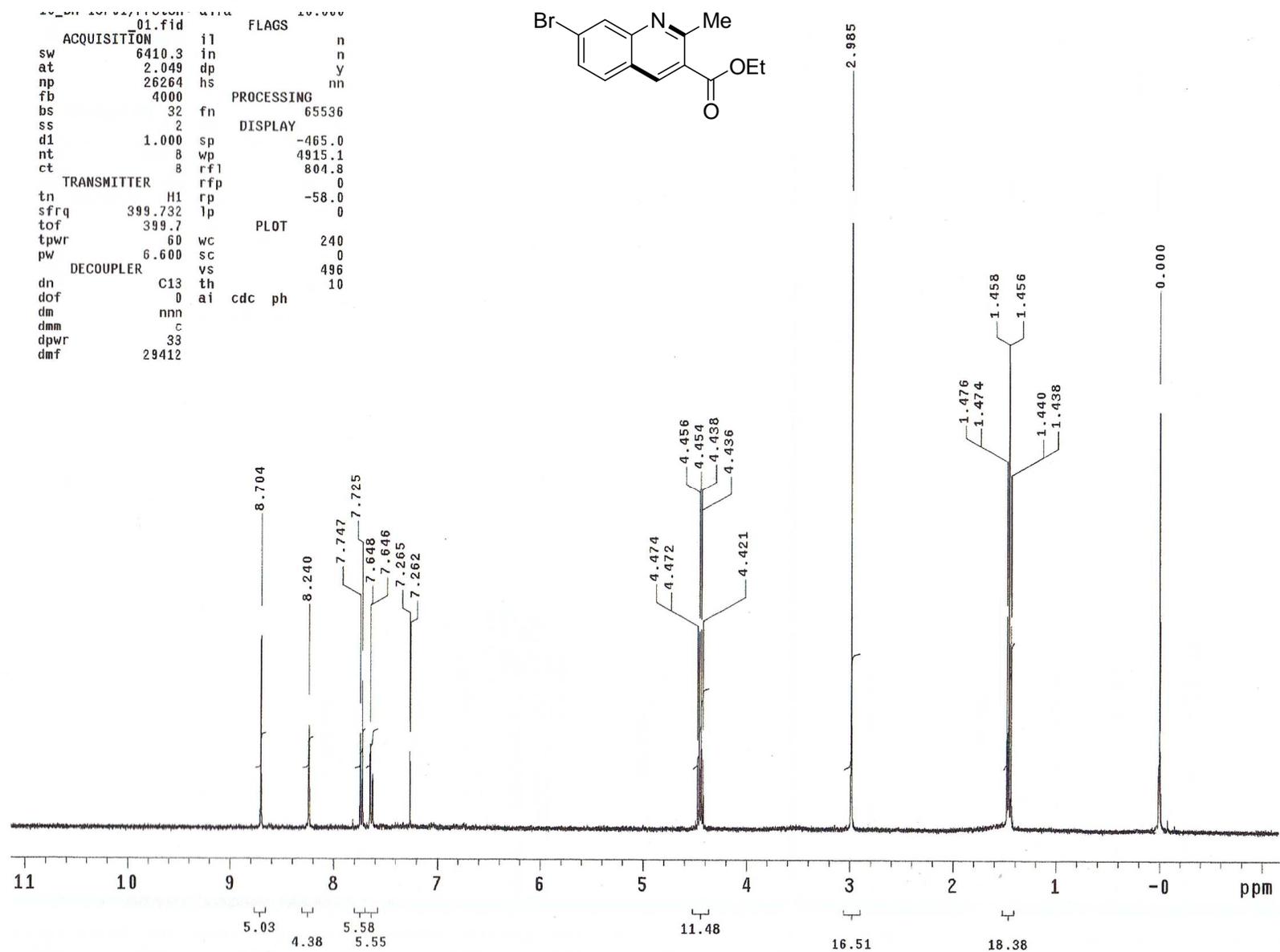
1,2-dihydro-3-(2-methylprop-1-enyl)quinoline (1j'): ^{13}C NMR (100 MHz, CDCl_3)

2-Methyl-3-((E)-4-methylpenta-1,3-dienyl)quinoline (1k'): ^1H NMR (400 MHz, CDCl_3)

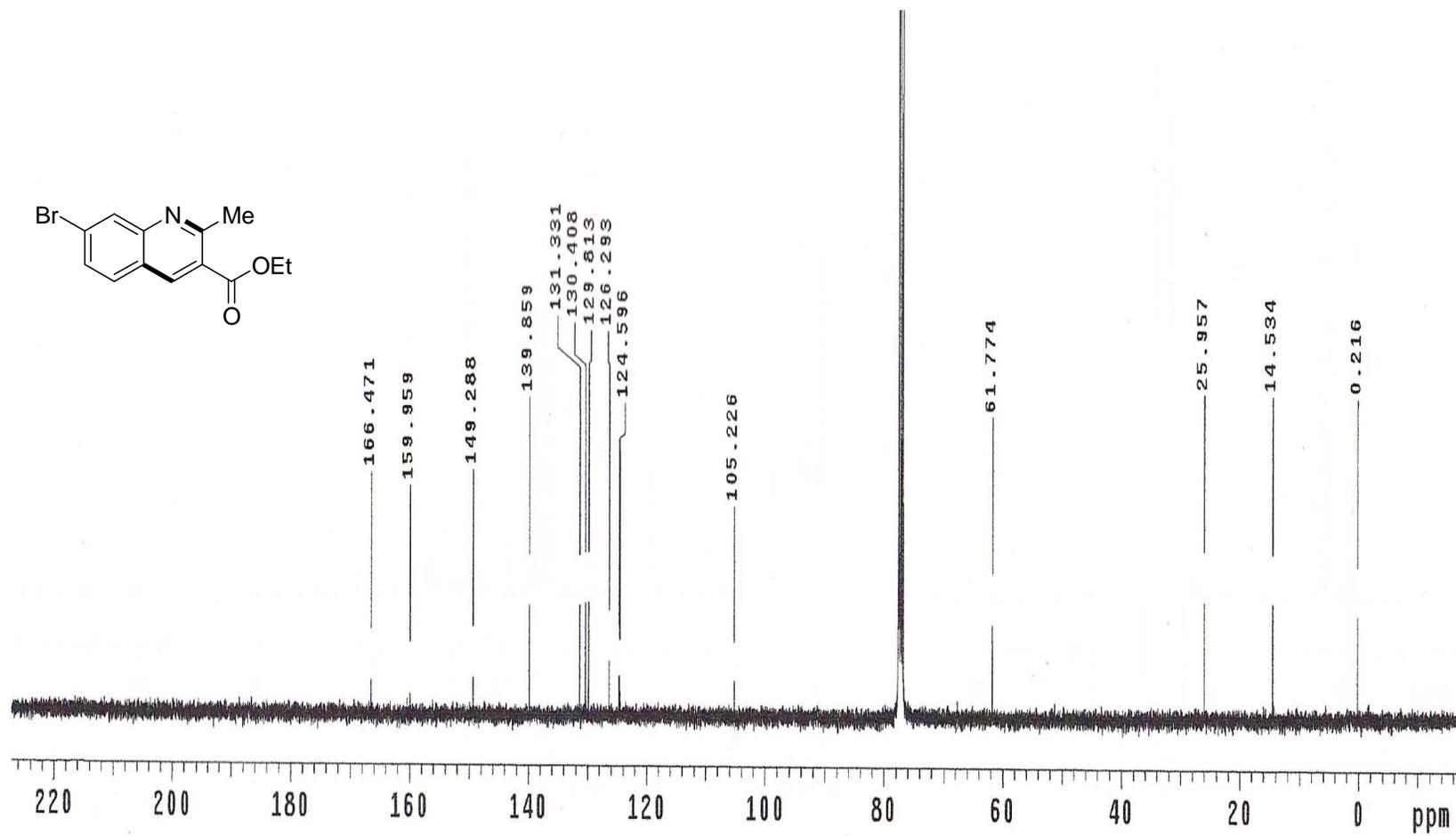
2-Methyl-3-((E)-4-methylpenta-1,3-dienyl)quinoline (1k'): ^{13}C NMR (100 MHz, CDCl_3)

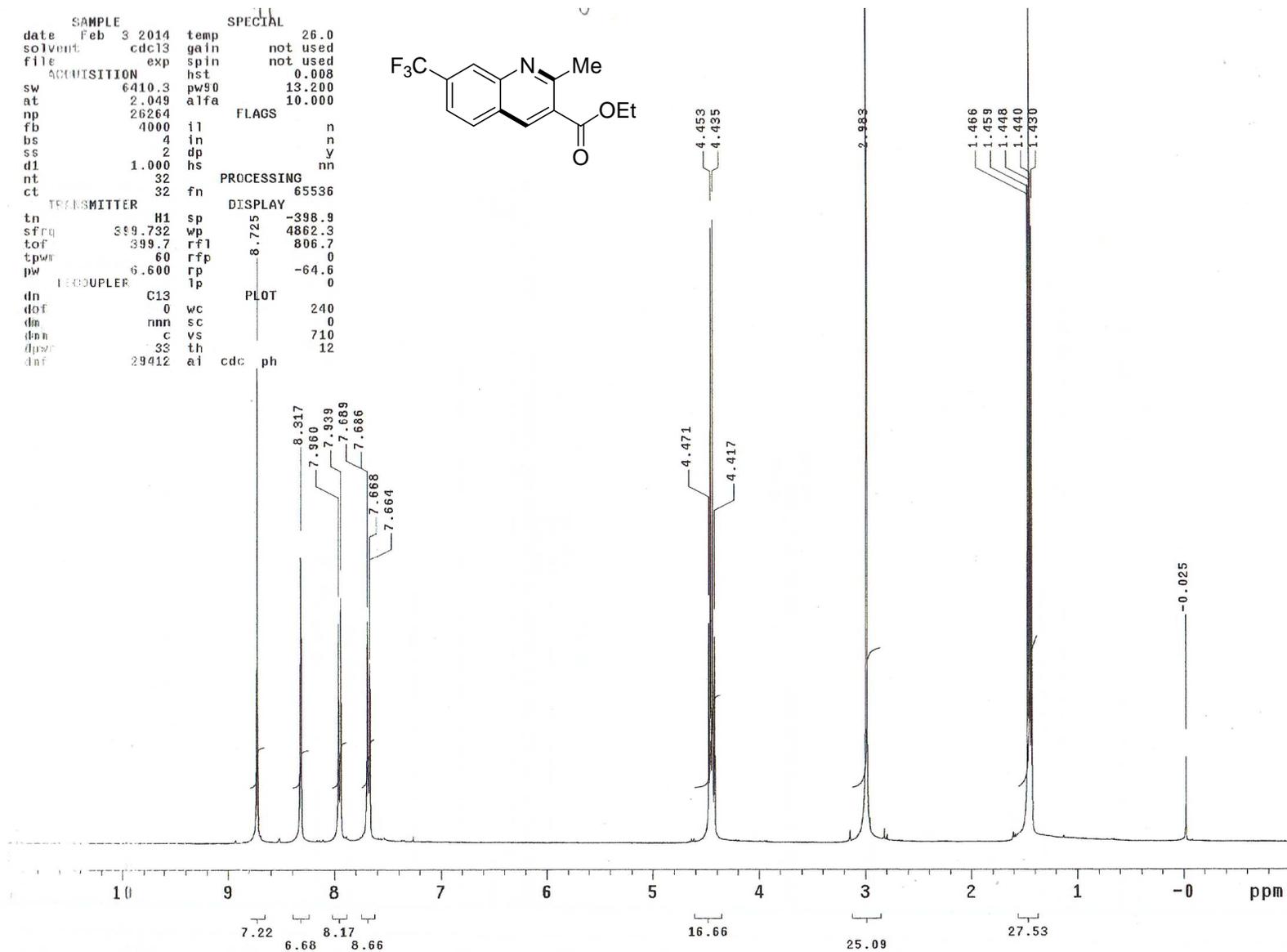
| | | |
|-------------|---------|-------------|
| TRANSMITTER | lb | 0.50 |
| tn | C13 | fn not used |
| sfrq | 100.523 | DISPLAY |
| tof | 1027.9 | sp -1678.5 |
| tpwr | 55 | wp 24509.1 |
| pw | 4.150 | rfl 9441.7 |
| DECOUPLER | rfl | rff 7762.6 |
| dn | H1 | rp 83.6 |
| dof | 0 | lp 0 |
| dm | YYY | PLOT |
| dmm | w | wc 240 |
| dpwr | 41 | sc 0 |
| dmf | 9648 | vs 308788 |
| | th | 9 |
| | ai | cdc ph |

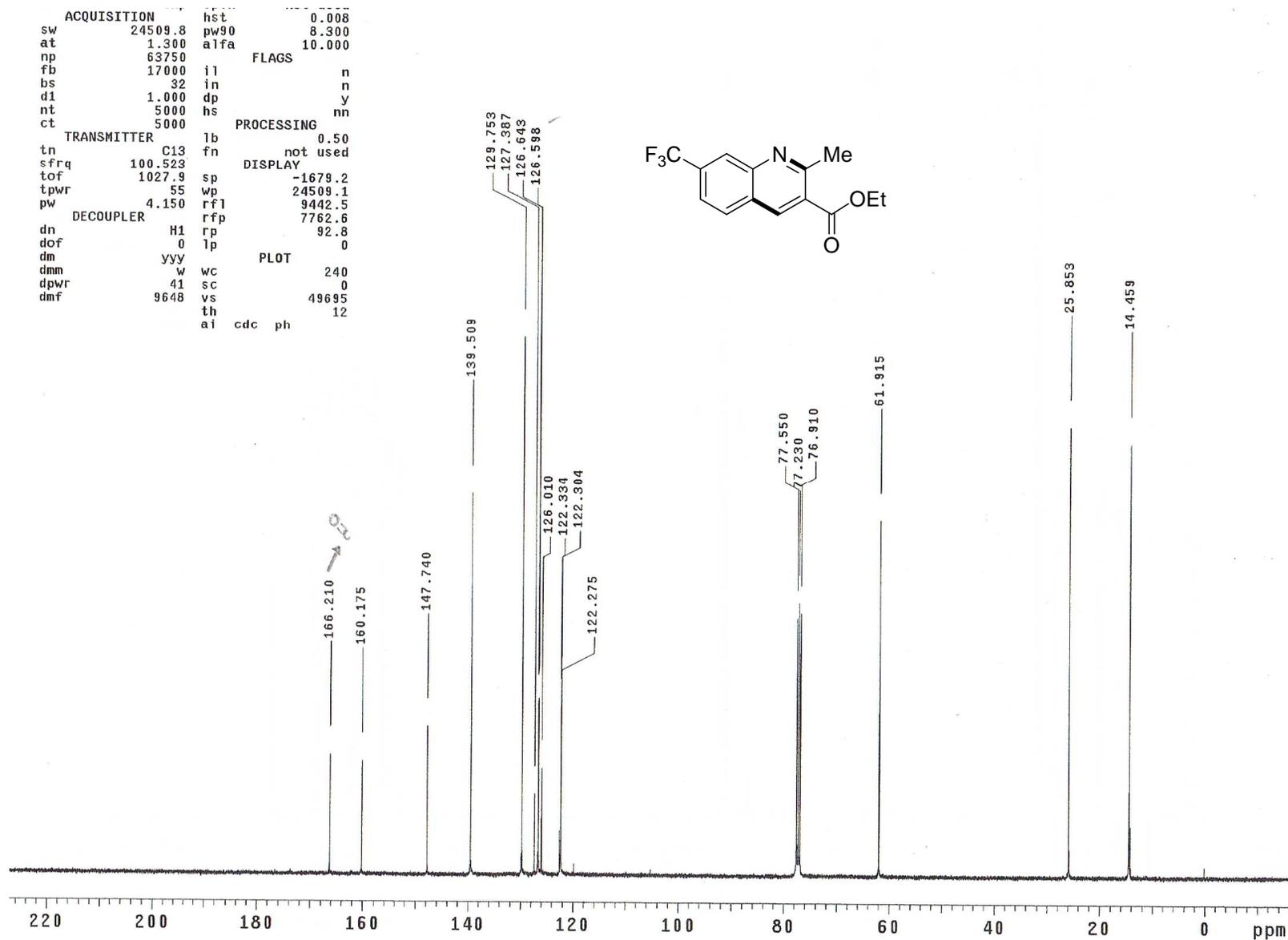


Ethyl 7-bromo-2-methylquinoline-3-carboxylate (3b'): ^1H NMR (400 MHz, CDCl_3)

Ethyl 7-bromo-2-methylquinoline-3-carboxylate (**3b'**): ^{13}C NMR (100 MHz, CDCl_3)

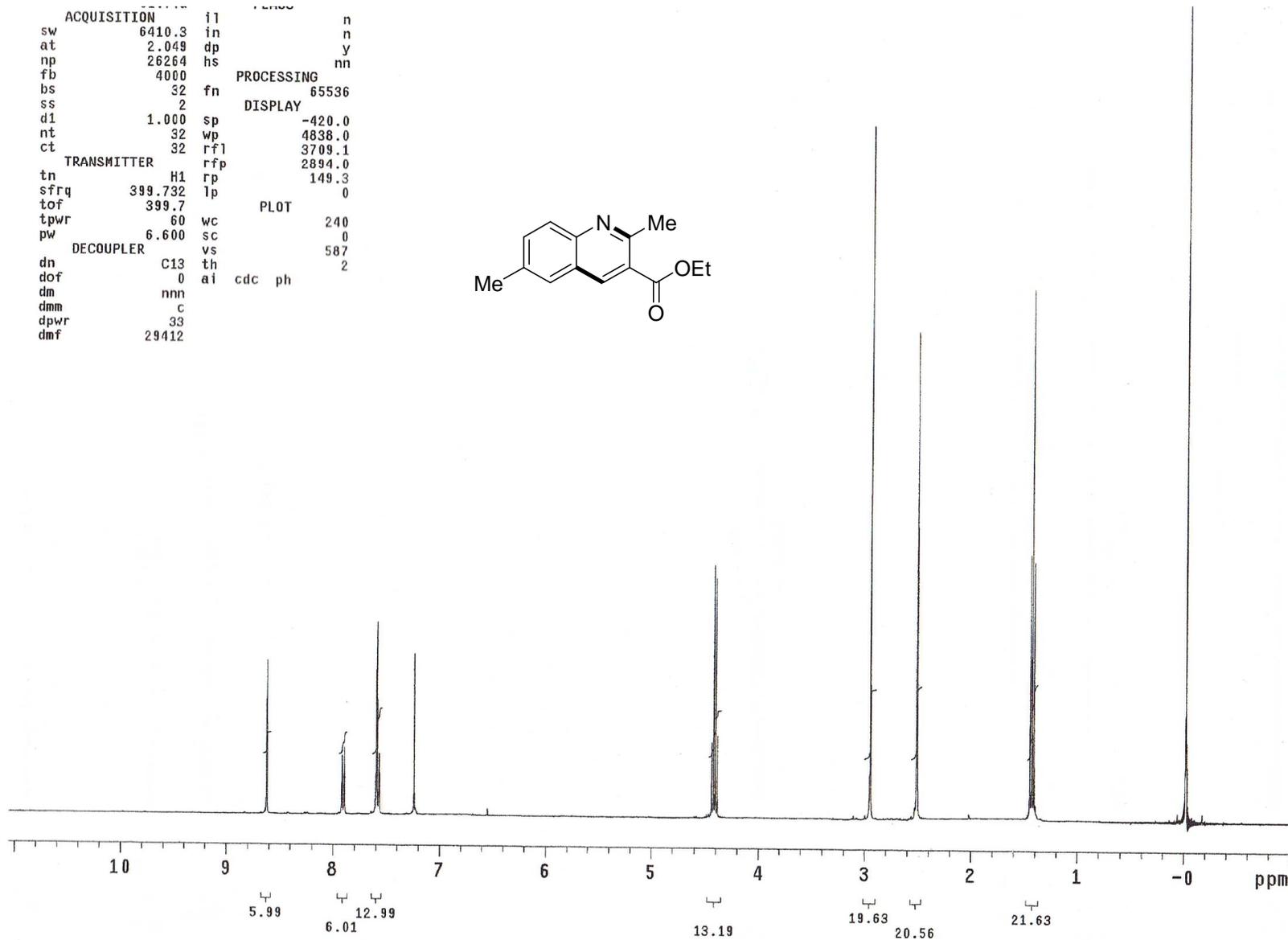
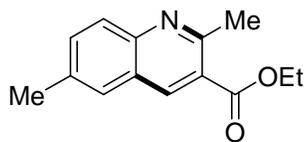


Ethyl 7-(trifluoromethyl)-2-methylquinoline-3-carboxylate (4b'): ^1H NMR (400 MHz, CDCl_3)

Ethyl 7-(trifluoromethyl)-2-methylquinoline-3-carboxylate (4b'): ^{13}C NMR (100 MHz, CDCl_3)

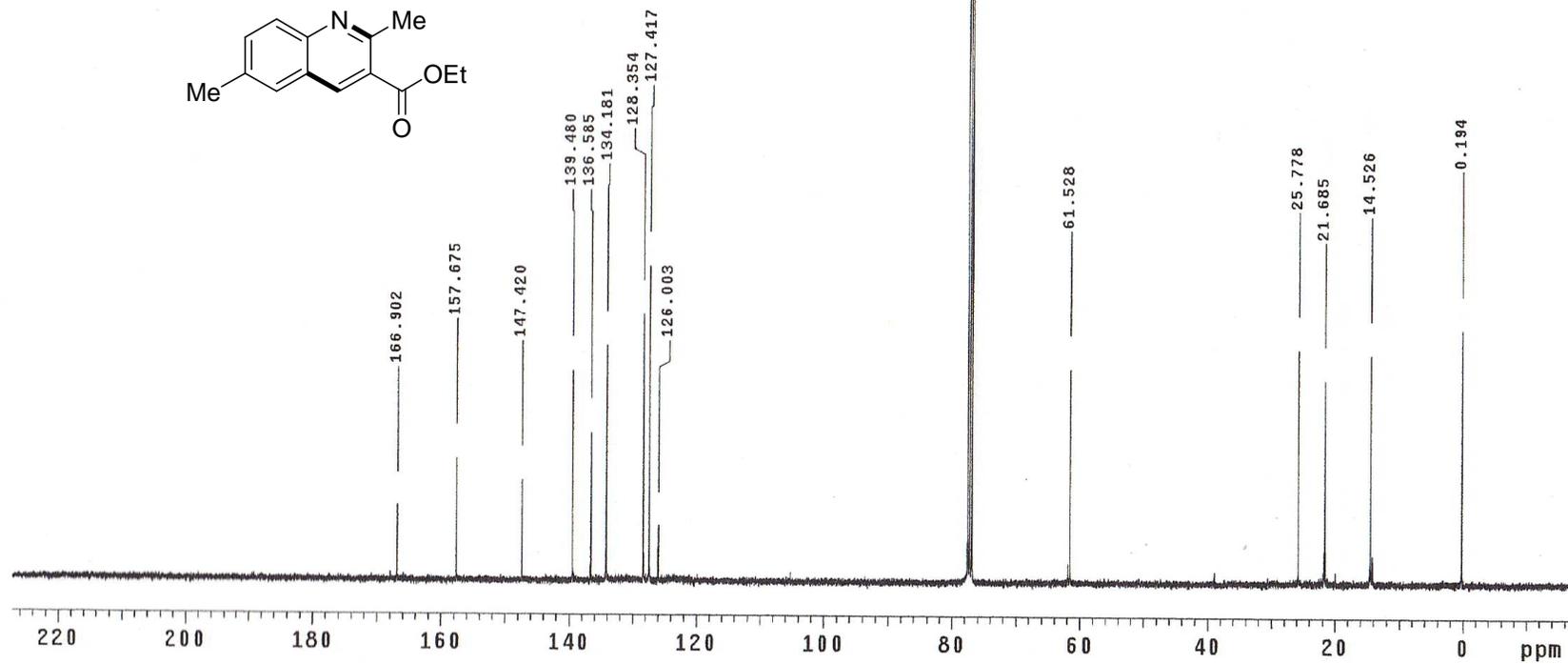
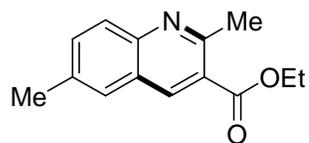
Ethyl 2,6-dimethylquinoline-3-carboxylate (5b'): ^1H NMR (400 MHz, CDCl_3)

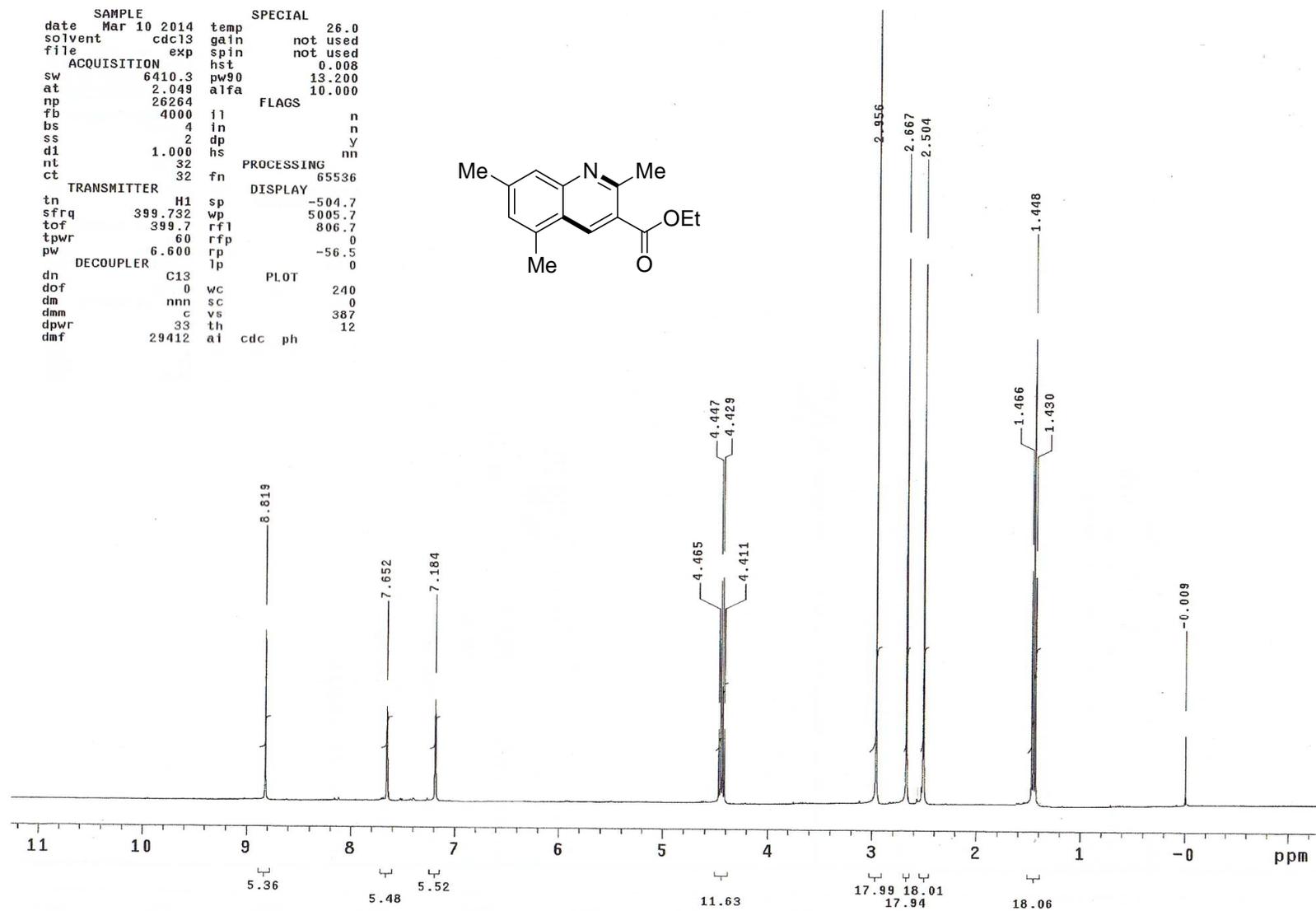
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ACQUISITION 11 n
sw 6410.3 in n
at 2.049 dp y
np 26264 hs nn
fb 4000
bs 32 fn PROCESSING 65536
ss 2 DISPLAY
d1 1.000 sp -420.0
nt 32 wp 4838.0
ct 32 rfl 3709.1
TRANSMITTER rfp 2894.0
tn H1 rp 149.3
sfrq 399.732 lp 0
tof 399.7 PLOT
tpwr 60 wc 240
pw 6.600 sc 0
DECOUPLER C13 vs 587
dn 0 ai cdc ph 2
dm nnn
dmm c
dpwr 33
dmf 29412
```

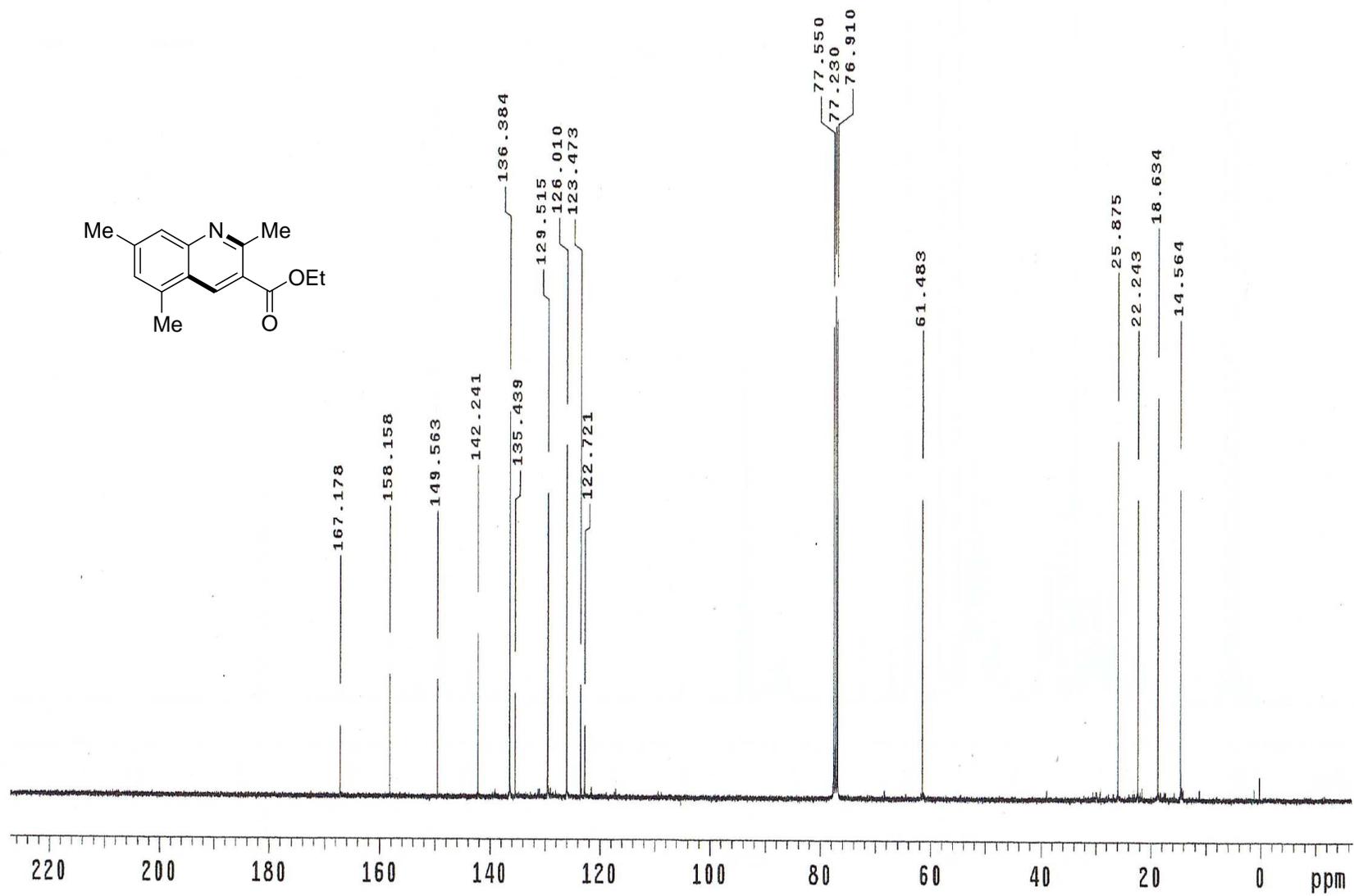


Ethyl 2,6-dimethylquinoline-3-carboxylate (5b'): ^{13}C NMR (100 MHz, CDCl_3)

| | | | |
|-------------|---------|------------|----------|
| ACQUISITION | | il | n |
| sw | 24509.8 | in | n |
| at | 1.300 | dp | y |
| np | 63750 | hs | nn |
| fb | 17000 | PROCESSING | |
| bs | 64 | lb | 0.50 |
| d1 | 1.000 | fn | not used |
| nt | 5000 | DISPLAY | |
| ct | 5000 | sp | -1679.9 |
| TRANSMITTER | | wp | 24509.1 |
| tn | C13 | rfl | 9443.2 |
| sfrq | 100.523 | rfp | 7762.6 |
| tof | 1027.9 | rp | 167.1 |
| tpwr | 55 | lp | 0 |
| pw | 4.150 | PLOT | |
| DECOUPLER | | wc | 240 |
| dn | H1 | sc | 0 |
| dof | 0 | vs | 66565 |
| dm | yyy | th | 7 |
| dmm | w | ai | cdc ph |
| dpwr | 41 | | |
| dmf | 9648 | | |

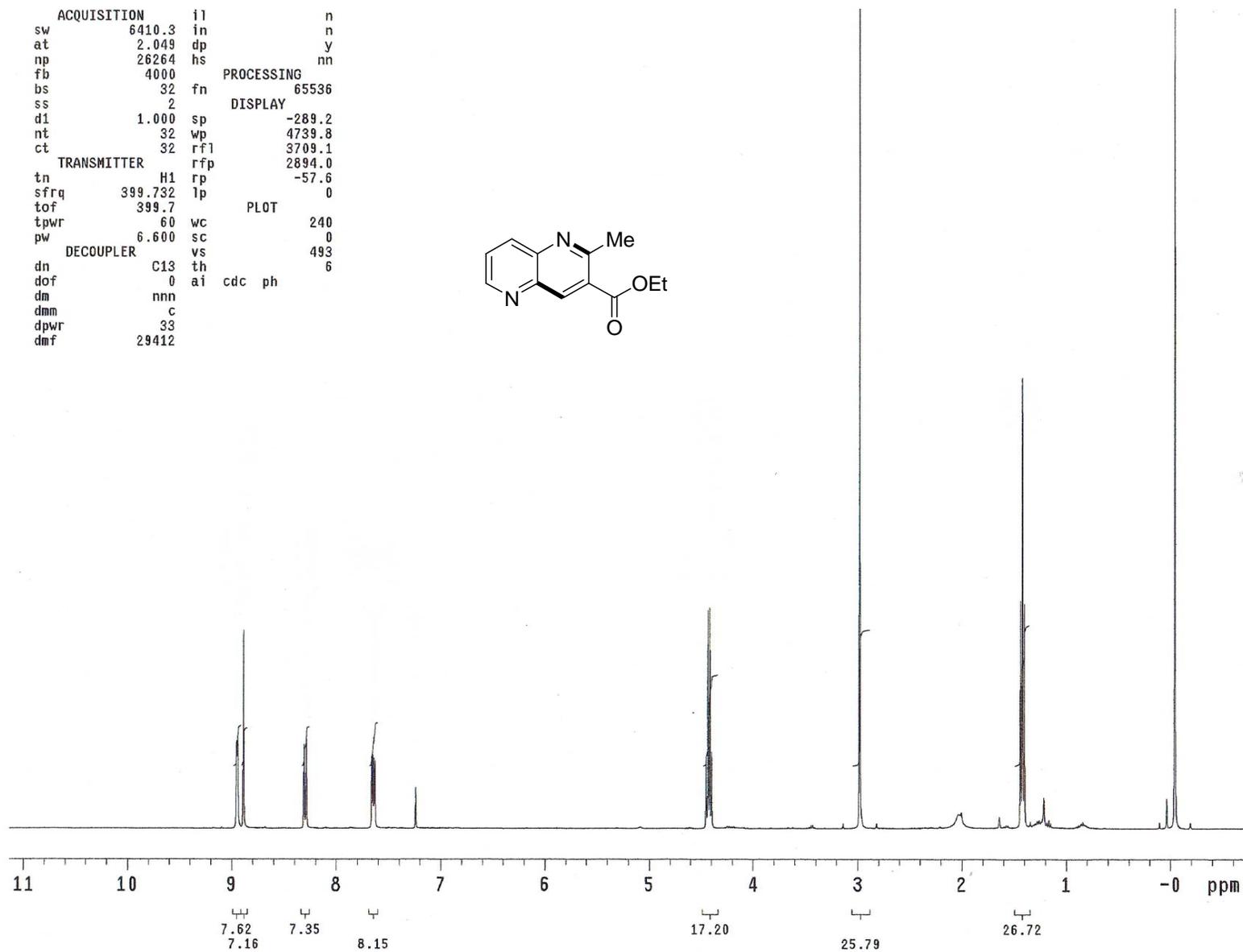
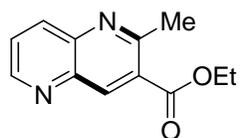


Ethyl 2,5,7-trimethylquinoline-3-carboxylate (6b'): ^1H NMR (400 MHz, CDCl_3)

Ethyl 2,5,7-trimethylquinoline-3-carboxylate (6b'): ^{13}C NMR (100 MHz, CDCl_3)

Ethyl 2-methyl-1,5-naphthyridine-3-carboxylate (7b'): ^1H NMR (400 MHz, CDCl_3)

| | | | |
|-------------|---------|------------|--------|
| ACQUISITION | il | n | |
| sw | 6410.3 | in | n |
| at | 2.049 | dp | y |
| np | 26264 | hs | nn |
| fb | 4000 | PROCESSING | |
| bs | 32 | fn | 65536 |
| ss | 2 | DISPLAY | |
| d1 | 1.000 | sp | -289.2 |
| nt | 32 | wp | 4739.8 |
| ct | 32 | rfl | 3709.1 |
| TRANSMITTER | | rffp | 2894.0 |
| tn | H1 | rp | -57.6 |
| sfrq | 399.732 | lp | 0 |
| tof | 399.7 | PLOT | |
| tpwr | 60 | wc | 240 |
| pw | 6.600 | sc | 0 |
| DECOUPLER | | vs | 493 |
| dn | C13 | th | 6 |
| dof | 0 | ai | cdc ph |
| dm | nnn | | |
| dmm | c | | |
| dpwr | 33 | | |
| dmf | 29412 | | |



Ethyl 2-methyl-1,5-naphthyridine-3-carboxylate (7b'): ^{13}C NMR (100 MHz, CDCl_3)

| | | | |
|-------------|---------|---------|----------|
| ACQUISITION | il | n | |
| sw | 24509.8 | in | n |
| at | 1.300 | dp | y |
| np | 63750 | hs | nn |
| fb | 17000 | | |
| bs | 64 | lb | 0.50 |
| d1 | 1.000 | fn | not used |
| nt | 5000 | DISPLAY | |
| ct | 5000 | sp | -1678.5 |
| TRANSMITTER | | wp | 24509.1 |
| tn | C13 | rfl | 9441.7 |
| sfrq | 100.523 | rfp | 7762.6 |
| tof | 1027.9 | rp | 78.7 |
| tpwr | 55 | lp | 0 |
| pw | 4.150 | PLOT | |
| DECOUPLER | | wc | 240 |
| dn | H1 | sc | 0 |
| dof | 0 | vs | 42452 |
| dm | yyy | th | 7 |
| dmm | w | ai | cdc ph |
| dpwr | 41 | | |
| dmf | 9648 | | |

