SUPPORTING INFORMATION

Synthesis of β-keto esters in-flow and rapid access to substituted pyrimidines

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General Experimental

Starting materials were obtained from commercial suppliers and used without further purification. Reactions were monitored by TLC using silica 60 gel aluminium-backed plates, which were visualised by exposure to UV light, followed by staining with basic potassium permanganate solution. Flash chromatography was carried out using silica gel 60, 35-70 μ, as the stationary phase and the solvents were of analytical purity.

Melting points are uncorrected. Infra-red spectra were recorded as a dilute solution in chloroform, or as a solid. NMR spectra were recorded at 298 K, at the frequency stated and run as a dilute solution in $CDCl_3$ or CD_3OD. Chemical shifts are expressed in ppm downfield with the solvent residual peak ($CDCl_3$ δ_H 7.26, δ_C 77.1, CD_3OD δ_H 3.31, δ_C 49.0) as the internal standard. All coupling constants are reported in Hertz (Hz) and multiplicity of each signal is designated by the following abbreviations: s, singlet; d, doublet; t, triplet; q, quartet; m, multiplet; app, apparent; br, broad or some combinations thereof. The ^{13}C NMR spectra were assigned with the benefit of DEPT experiments.

Catalyst screening under batch conditions: formation of ethyl-5-phenyl-3-oxopentanoate, 3

Ethyl diazoacetate **2** (0.505 mmol) in CH_2Cl_2 (2 mL) was added dropwise to a solution of hydrocinnamaldehyde **1** (0.500 mmol) and Lewis acid (mol%) in CH_2Cl_2 (2 mL). The reaction was stirred at room temperature until TLC indicated complete consumption of the starting material. The reaction mixture was then diluted (brine), the organic layer separated and the aqueous layer extracted with CH_2Cl_2 (2 x 10 mL). The combined organic extracts were dried ($MgSO_4$), filtered and concentrated *in vacuo* to give the crude β-keto ester. Purification by

column chromatography (2% EtOAc/petrol) gave the pure ethyl-5-phenyl-3-oxopentanoate, **3** as a colourless oil. R_f 0.3 (10% EtOAc/petrol); $\nu_{max}(CHCl_3)/cm^{-1}$ 3010, 1745, 1716, 1497, 1454, 1409, 1369, 1317, 1242, 1184 and 1031; The ^1H NMR spectrum showed that the product exists as a 14:1 mixture of the keto and enol forms. δ_H (400 MHz; $CDCl_3$) Data for the major keto form: 7.30□7.26 (2H, m), 7.22□7.17 (3H, m), 4.18 (2H, q, *J* 7.6), 3.42 (2H, s), 2.96□2.87 (4H, m, CCH_2), 2.51 (3H, t, *J* 7.6); δ_C (100 MHz; $CDCl_3$) 202.0 (C), 167.2 (C), 140.6 (C), 128.6 (CH), 128.4 (CH), 126.3 (CH), 61.5 (CH_2), 49.6 (CH_2), 44.6 (CH_2), 29.5 (CH_2) and 14.2 (CH_3); *m/z* (ESI) found 221.1177 (M+H, $C_{13}H_{17}O_3$ requires 221.1172). Found 243.0994 (M+Na, $C_{13}H_{16}NaO_3$ requires 243.0992). Data consistent with that previously reported.[1]

General Procedures

Flow reaction and heterocycle formation: Procedure A

The flow reaction was controlled using Vapourtec Flowcommander software.

A solution of aldehyde (0.500 mmol) and $BF_3{\cdot}OEt_2$ (1 mol%) in CH_2Cl_2 (2 mL) was injected into one of the 2 mL sample loops of the R2+ unit. The other 2 mL sample loop was loaded with a solution of ethyl diazoacetate (0.505 mmol) in CH_2Cl_2 (2 mL). The valves of the loop were set to load and the reagents pumped through the system using CH_2Cl_2 as a system solvent at a flow rate of 0.139 mL/min. The reagents mixed in a T-piece before entering a 2 mL coil reactor (PFA, 2.9 m of 1 mm ID tubing), maintained at 30 °C (R4 unit). Successive back pressure regulators (250, 100 and 40 Psi) were added in line after the reactor to ensure the nitrogen produced during the reaction stayed in solution. The output (8 mL total volume) was collected directly into a round-bottomed flask.

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To the "output" from the flow reaction (crude β-ketoester in 8 mL CH_2Cl_2) was added EtOH (8 mL), the desired amidine hydrochloride (1.10 eq.) and DBU (2.00 eq.) in a modification of a literature procedure.[2] The resultant solution was stirred under Ar at rt for 48 h (formamidine-, acetamidine- and cyclopropylcarbamidine- hydrochlorides) or heated to reflux for 18 h (benzamidine hydrochloride). The solvent was then removed *in vacuo* and the crude material purified by column chromatography.

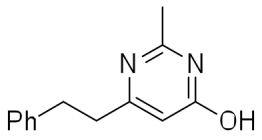
Flow reaction and heterocycle formation: Procedure B

As **procedure A** for the flow reaction, but the crude β-ketoester was treated with amidine hydrochloride (2.20 eq.) and DBU (4.00 eq.) in the heterocycle formation step.

Flow reaction and heterocycle formation: Procedure C

As **procedure A** for the flow reaction, except a solution of aldehyde (0.500 mmol) and ethyl diazoacetate (0.505 mmol) in CH_2Cl_2 (2 mL) was injected into one of the 2 mL sample loops of the R2+ unit, and the other 2 mL sample loop was loaded with a solution of $BF_3{\cdot}OEt_2$ (10 mol%) in CH_2Cl_2 (2 mL). **Procedure B** was followed for the heterocycle formation step.

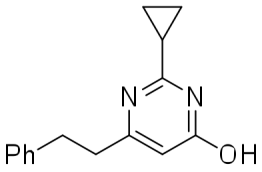
2-Methyl-6-(2-phenylethyl)-pyrimidin-4-ol, 5a



Following **procedure A**, hydrocinnamaldehyde gave, after purification by column chromatography (2% MeOH/CH_2Cl_2) the pyrimidinol **5a** (74 mg, 70%) as colorless needles, mp 126-127 °C (lit.,[3] 125-127 °C). (Found: C, 72.7; H, 6.6; N, 13.1. $C_{13}H_{14}N_2O$ requires C, 72.9; H, 6.6; N, 13.1); R_f 0.6 (10% MeOH/CH_2Cl_2);

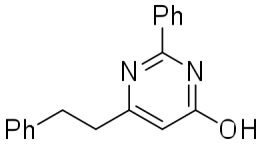
$\nu_{max}(CHCl_3)/cm^{-1}$ 3011, 2865, 2760, 1663, 1606, 1562, 1497, 1455, 1383, 1304, 1184, 967 and 856; δ_H (400 MHz; $CDCl_3$) 13.13 (1H, br s), 7.31□7.26 (2H, m), 7.21□7.18 (3H, m), 6.12 (1H, s), 3.02□2.96 (2H, m), 2.86□2.81 (2H, m), 2.48 (3H, s); δ_C (100 MHz; $CDCl_3$) 169.5 (C), 166.0 (C), 158.7 (C), 140.8 (C), 128.5 (CH), 128.4 (CH), 126.3 (CH), 109.6 (CH), 39.4 (CH_2), 34.1 (CH_2) and 21.7 (CH_3); *m/z* (ESI) found 237.1001. (M+Na $C_{13}H_{14}N_2NaO$ requires 237.0998). Data consistent with that previously reported.[4]

2-Cyclopropyl-6-(2-phenylethyl)-pyrimidin-4-ol, 5b



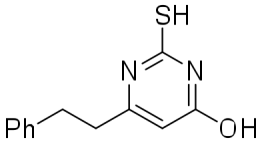
Following **procedure A**, hydrocinnamaldehyde gave, after purification by column chromatography (2% MeOH/CH_2Cl_2) the pyrimidinol **5b** (88 mg, 67%) as colorless needles, mp 143-144 °C. R_f 0.6 (10% MeOH/CH_2Cl_2); $\nu_{max}(CHCl_3)/cm^{-1}$ 3085, 3011, 2836, 1658, 1597, 1561, 1497, 1447, 1403, 1261, 1184, 966 and 850; δ_H (400 MHz; $CDCl_3$) 13.62 (1H, br s), 7.30□7.25 (2H, m), 7.22□7.16 (3H, m), 6.05 (1H, s), 2.99□2.94 (2H, m), 2.81□2.76 (2H, m), 1.97□1.90 (1H, m), 1.25□1.21 (2H, m), 1.11□1.06 (2H, m); δ_C (100 MHz; $CDCl_3$) 169.8 (C), 166.0 (C), 163.9 (C), 141.1 (C), 128.6 (CH), 128.5 (CH), 126.2 (CH), 108.7 (CH), 39.4 (CH_2), 34.0 (CH_2), 14.4 (CH) and 10.6 (CH_2); *m/z* (ESI) found 241.1333. (M+H, $C_{15}H_{17}N_2O$ requires 241.1335). Found 263.1145. (M+Na $C_{15}H_{16}N_2NaO$ requires 263.1155).

6-(2-Phenylethyl)-2-phenylpyrimidin-4-ol, 5c



Following **procedure A**, hydrocinnamaldehyde gave, after purification by column chromatography (2% MeOH/CH_2Cl_2) the pyrimidinol **5c** (46 mg, 33%) as colorless needles, mp 189-191 °C. R_f 0.5 (10% MeOH/CH_2Cl_2); ν_{max}($CHCl_3$)/cm^{-1} 3220, 3149, 3066, 3011, 2958, 2863, 2771, 1656, 1604, 1597, 1597, 1570, 1545, 1500, 1454, 1443, 1400, 1310, 1191, 979 and 854; δ_H (400 MHz; $CDCl_3$) 13.22 (1H, br s), 8.28□8.25 (2H, m), 7.56□7.55 (3H, m), 7.34□7.29 (2H, m), 7.27□7.19 (3H, m), 6.28 (1H, s), 3.16□3.08 (2H, m), 3.00□2.93 (2H, m); δ_C (100 MHz; $CDCl_3$) 169.0 (C), 165.6 (C), 156.7 (C), 141.1 (C), 132.3 (C), 132.1 (CH), 129.1 (CH), 128.6 (CH), 128.5 (CH), 128.0 (CH), 126.3 (CH), 110.8 (CH), 39.5 (CH_2) and 34.2 (CH_2); *m/z* (ESI) found 277.1325. (M+H, $C_{18}H_{17}N_2O$ requires 277.1335). Found 299.1145. (M+Na $C_{18}H_{16}N_2NaO$ requires 299.1155).

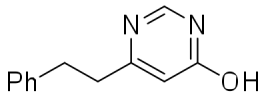
2-Mercapto-6-(2-phenylethyl)-pyrimidin-4-ol, 5d



Procedure A was followed for the formation of the β-ketoester (in flow) from hydrocinnamaldehyde, but the "output" from the flow reaction was then concentrated *in vacuo* to give the crude β-ketoester (0.5 mmol). This crude material was then treated with NaOEt and thiourea according to the literature procedure[5] to yield the pyrimidinol **5d** (73 mg, 63%) as colorless needles, mp 220-223 °C (lit.,[6] 215-220 °C). (Found: C, 62.0; H, 5.2; N, 11.9. $C_{12}H_{12}N_2OS$ requires C, 62.0; H,5.2; N, 12.1); ν_{max}(solid)/cm^{-1} 3142, 2967, 1676, 1657, 1633, 1568, 1494, 1440, 1266, 1242, 1197, 1167, 957, 855 and 819; δ_H (400 MHz; CD_3OD) 7.31□7.26 (2H, m), 7.24□7.17 (3H, m), 5.61 (1H, s), 2.96□2.89 (2H, m), 2.73□2.68 (2H, m), OH, SH exchange; δ_C (100 MHz; CD_3OD) 178.0 (C), 164.2 (C), 158.2 (C), 141.0 (C),

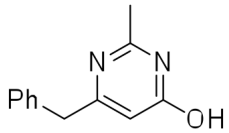
129.6 (CH), 129.5 (CH), 127.6 (CH), 104.0 (CH), 35.0 (CH_2) and 34.7 (CH_2); *m/z* (ESI) found 255.0560. (M+Na, $C_{12}H_{12}N_2ONaS$ requires 255.0563). Data consistent with that previously reported.[6]

6-(2-phenylethyl)-pyrimidin-4-ol, 5e



Following **procedure A**, hydrocinnamaldehyde gave, after purification by column chromatography (2% MeOH/CH_2Cl_2) the pyrimidinol **5e** (30 mg, 30%) as a pale tan solid, mp 217-219 °C decomp. R_f 0.5 (10% MeOH/CH_2Cl_2); ν_{max}(solid)/cm^{-1} 2919, 1667s, 1651s, 1605m, 1531, 1497, 1455, 1411, 1344, 1166, 974 and 860; δ_H (400 MHz; CD_3OD) 8.16 (1H, s), 7.28□7.23 (2H, m), 7.21□7.14 (3H, m), 6.18 (1H, s), 3.00□2.94 (2H, m), 2.86□2.81 (2H, m), OH exchanges; δ_C (100 MHz; CD_3OD) 168.8 (C), 165.1 (C), 150.6 (CH), 141.9 (C), 129.5 (CH), 129.4 (CH), 127.3 (CH), 113.9 (CH), 39.7 (CH_2) and 35.1 (CH_2); *m/z* (ESI) found 223.0847 (M+Na $C_{12}H_{12}N_2NaO$ requires 223.0842).

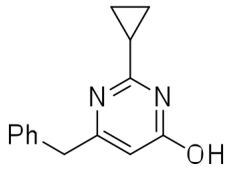
6-Benzyl-2-methylpyrimidin-4-ol, 5f



Following **procedure B**, phenylacetaldehyde gave, after purification by column chromatography (2% MeOH/CH_2Cl_2) the pyrimidinol **5f** (71 mg, 54%) as colorless needles, mp 219-221 °C (lit.,[7] 222-224 °C). (Found: C, 71.6; H, 6.0; N, 13.8. $C_{12}H_{12}N_2O$ requires C, 72.0; H, 6.0; N, 14.0); R_f 0.4 (10% MeOH/CH_2Cl_2); ν_{max}($CHCl_3$)/cm^{-1} 3067, 3010, 2844, 2759, 1663, 1608, 1564, 1496, 1454, 1421, 1384, 1182, 967 and 859; δ_H (400 MHz; $CDCl_3$) 13.45 (1H, br s), 7.35□7.27 (2H, m), 7.26□7.22 (3H, m), 6.02 (1H, s), 3.86 (2H,

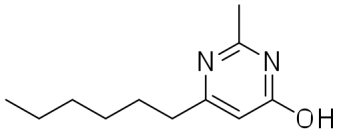
s), 2.44 (3H, s); δ_C (100 MHz; $CDCl_3$) 169.6 (C), 166.1 (C), 158.7 (C), 137.0 (C), 129.5 (CH), 128.8 (CH), 127.0 (CH), 110.0 (CH), 44.1 (CH_2) and 21.7 (CH_3); *m/z* (ESI) found 223.0852 (M+Na, $C_{12}H_{12}N_2NaO$ requires 223.0842). Data consistent with that previously reported.[7]

6-Benzyl-2-cyclopropylpyrimidin-4-ol, 5g



Following **procedure A**, phenylacetaldehyde gave, after purification by column chromatography (2% MeOH/CH_2Cl_2) the pyrimidinol **5g** (54 mg, 54%) as colorless needles, mp 182-183 °C. (Found: C, 74.0; H, 6.2; N, 12.3. $C_{14}H_{14}N_2O$ requires C, 74.3; H, 6.2; N, 12.4); R_f 0.5 (10% MeOH/CH_2Cl_2); ν_{max}($CHCl_3$)/cm^{-1} 3084, 3011, 2898, 2836, 1657, 1596, 1562, 1495, 1472, 1447, 1403, 1263, 1184, 1015, 963, 867 and 853; δ_H (400 MHz; $CDCl_3$) 13.62 (1H, br s), 7.33□7.26 (5H, m), 5.95 (1H, s), 3.77 (2H, s), 1.91□1.85 (1H, m), 1.22□1.18 (2H, m), 1.07□1.03 (2H, m); δ_C (100 MHz; $CDCl_3$) 169.8 (C), 166.2 (C), 163.9 (C), 137.4 (C), 129.5 (CH), 128.7 (CH), 126.8 (CH), 108.8 (CH), 44.2 (CH_2), 14.3 (CH) and 10.6 (CH_2); *m/z* (ESI) found 227.1185 (M+H requires $C_{14}H_{15}N_2O$ 227.1179). Found 249.0997 (M+Na, $C_{14}H_{14}N_2NaO$ requires 249.0998).

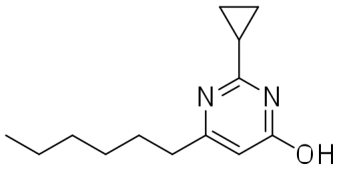
6-Hexyl-2-methylpyrimidin-4-ol, 5h



Following **procedure A**, heptaldehyde gave, after purification by column chromatography (2% MeOH/CH_2Cl_2) the pyrimidinol **5h** (60 mg, 62%) as colorless needles, mp 80-82 °C. (Found: C, 67.8; H, 9.3; N, 14.3. $C_{11}H_{18}N_2O$ requires C, 68.0; H, 9.3; N, 14.4); R_f 0.4 (10% MeOH/CH_2Cl_2);

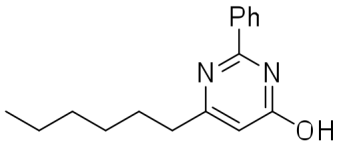
$\nu_{max}(CHCl_3)/cm^{-1}$ 2959, 2931, 2860, 1663, 1607, 1562, 1467, 1383, 1305, 1186, 968 and 853; δ_H (400 MHz; $CDCl_3$) 13.41 (1H, br s), 6.15 (1H, s), 2.50 (2H, t, *J* 7.6), 2.46 (3H, s), 1.63 (2H, *app* pent, *J* 7.6), 1.37□1.27 (6H, m), 0.87 (3H, t *J* 6.8); δ_C (100 MHz; $CDCl_3$) 170.9 (C), 166.1 (C), 158.5 (C), 109.2 (CH), 37.9 (CH_2), 31.7 (CH_2), 29.0 (CH_2), 28.1 (CH_2), 22.7 (CH_2), 21.8 (CH_3) and 14.2 (CH_3); *m/z* (ESI) found 195.1508. (M+H, $C_{11}H_{19}N_2O$ requires 195.1492). Found 217.1327 (M+Na, $C_{11}H_{18}N_2NaO$ requires 217.1311).

2-Cyclopropyl-6-hexylpyrimidin-4-ol, 5i



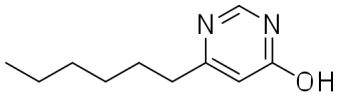
Following **procedure B**, heptaldehyde gave, after purification by column chromatography (2% MeOH/CH_2Cl_2) the pyrimidinol **5i** (65 mg, 59%) as a pale yellow solid, mp 95-96 °C. (Found: C, 70.8; H, 9.2; N, 12.5. $C_{13}H_{20}N_2O$ requires C, 70.9; H, 9.2; N, 12.7); R_f 0.3 (10% MeOH/CH_2Cl_2); $\nu_{max}(CHCl_3)/cm^{-1}$ 2958, 2931, 2859, 1657, 1595, 1560, 1447, 1402, 1262, 1185, 967 and 865; δ_H (400 MHz; $CDCl_3$) 13.50 (1H, br s), 6.10 (1H, s), 2.43 (2H, t, *J* 7.6), 1.94□1.89 (1H, m), 1.59 (2H, *app* pent, *J* 7.6), 1.33□1.27 (6H, m), 1.22□1.l7 (2H, m), 1.08□1.03 (2H, m), 0.89□0.85 (3H, m); δ_C (100 MHz; $CDCl_3$) 171.2 (C), 166.1 (C), 163.7 (C), 108.2 (CH), 37.9 (CH_2), 31.8 (CH_2), 28.9 (CH_2), 27.9 (CH_2), 22.7 (CH_2), 14.4 (CH), 14.2 (CH_3) and 10.5 (CH_2); *m/z* (ESI) found 221.1658. (M+H, $C_{13}H_{21}N_2O$ requires 221.1648). Found 243.1471 (M+Na, $C_{13}H_{20}N_2NaO$ requires 243.1468).

6-Hexyl-2-phenylpyrimidin-4-ol, 5j



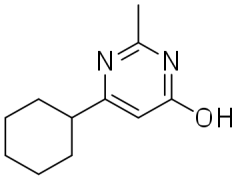
Following **procedure B**, heptaldehyde gave, after purification by column chromatography (2% MeOH/CH_2Cl_2) the pyrimidinol **5j** (65 mg, 51%) as an off-white solid, mp 104-106 °C. R_f 0.5 (10% MeOH/CH_2Cl_2); $\nu_{max}(CHCl_3)/cm^{-1}$ 3067, 3011, 2958, 2931, 2860, 1656, 1596, 1571, 1545, 1500, 1443, 1400, 1311, 1191, 980 and 851; δ_H (400 MHz; $CDCl_3$) 13.1 (1H, br s), 8.25□8.20 (2H, m), 7.56□7.53 (3H, m), 6.30 (1H, s), 2.62 (2H, t, *J* 7.6), 1.74 (2H, *app* pent, *J* 7.6), 1.43□1.36 (6H, m), 0.93□0.87 (3H, m); δ_C (100 MHz; $CDCl_3$) 170.4 (C), 165.7 (C), 156.6 (C), 132.5 (C), 132.0 (CH), 129.0 (CH), 128.0 (CH), 110.3 (CH), 37.9 (CH_2), 31.8 (CH_2), 29.0 (CH_2), 28.0 (CH_2), 22.7 (CH_2) and 14.2 (CH_3); *m/z* (ESI) found 257.1646. (M+H, $C_{16}H_{21}N_2O$ requires 257.1648). Found 279.1457 (M+Na, $C_{16}H_{20}N_2NaO$ requires 279.1468).

6-Hexylpyrimidin-4-ol, 5k



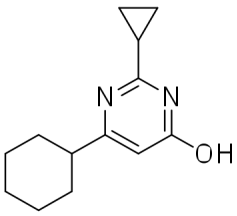
Following **procedure B**, heptaldehyde gave, after purification by column chromatography (2% MeOH/CH_2Cl_2) the pyrimidinol **5k** (30 mg, 33%) as a bright yellow solid, mp 115-117 °C. R_f 0.5 (10% MeOH/CH_2Cl_2); $\nu_{max}(CHCl_3)/cm^{-1}$ 3011, 2959, 2931, 2859, 1665, 1611, 1545, 1420, 1340, 1240, 1162, 978 and 862; δ_H (400 MHz; $CDCl_3$) 13.13 (1H, br s), 8.10 (1H, s) 6.30 (1H, s), 2.56 (2H, t, *J* 7.6), 1.67 (2H, t, *J* 7.6), 1.28□1.37 (6H, m), 0.88 (3H, t *J* 6.8); δ_C (100 MHz; $CDCl_3$) 170.1 (C), 164.8 (C), 148.0 (CH), 112.8 (CH), 37.8 (CH_2), 31.7 (CH_2), 28.9 (CH_2), 28.0 (CH_2), 22.7 (CH_2) and 14.2 (CH_3); *m/z* (ESI) found 181.1344. (M+H, $C_{10}H_{17}N_2O$ requires 181.1335). Found 203.1168 (M+Na, $C_{10}H_{16}N_2NaO$ requires 203.1155).

6-Cyclohexyl-2-methyllpyrimidin-4-ol, 5l



Following **procedure A**, cyclohexanecarboxaldehyde gave, after purification by column chromatography (2% MeOH/CH_2Cl_2) the pyrimidinol **5l** (75 mg, 78%) as colorless needles, mp 175-177 °C. (Found: C, 68.3; H, 8.4; N, 14.4. $C_{11}H_{16}N_2O$ requires C, 68.7; H, 8.4; N, 14.6); R_f 0.4 (10% MeOH/CH_2Cl_2); $\nu_{max}(CHCl_3)/cm^{-1}$ 2993, 2932, 2856, 2759, 1661, 1607, 1559, 1450, 1406, 1384, 1357, 1307, 1240, 1186, 964 and 853; δ_H (400 MHz; $CDCl_3$) 13.41 (1H, br s), 6.14 (1H, s), 2.45 (3H, s), 2.41□2.37 (1H, m), 1.95□1.87 (2H, m), 1.86□1.69 (2H, m), 1.75□1.69 (1H, m), 1.40□1.31 (4H, m), 1.28□1.23 (1H, m); δ_C (100 MHz; $CDCl_3$) 174.9 (C), 166.5 (C), 158.3 (C), 107.3 (CH), 45.8 (CH), 31.7 (CH_2), 26.3 (CH_2), 26.1 (CH_2) and 21.8 (CH_3); *m/z* (ESI) found 193.1348. (M+H, $C_{11}H_{17}N_2O$ requires 193.1335). Found 215.1166 (M+Na, $C_{11}H_{16}N_2NaO$ requires 215.1155).

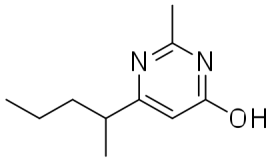
6-Cyclohexyl-2-cyclopropylpyrimidin-4-ol, 5m



Following **procedure A**, cyclohexanecarboxaldehyde gave, after purification by column chromatography (2% MeOH/CH_2Cl_2) the pyrimidinol **5m** (65 mg, 60%) as colorless needles, mp 165-166 °C. R_f 0.4 (10% MeOH/CH_2Cl_2); $\nu_{max}(CHCl_3)/cm^{-1}$ 3078, 2932, 2855, 1655, 1596, 1559, 1448, 1402, 1261, 1182, 1006, 953 and 847; δ_H (400 MHz; $CDCl_3$) 13.42 (1H, br s), 6.06 (1H, s), 2.36□2.28 (1H, m), 1.93□1.77 (5H, m), 1.69□1.67 (1H, m), 1.40□1.30 (4H, m), 1.22□1.19 (3H, m), 1.08□1.03 (2H, m); δ_C (100 MHz; $CDCl_3$) 175.0 (C), 166.5 (C), 163.5 (C), 106.4 (CH), 45.8 (CH), 31.5 (CH_2), 26.3 (CH_2), 26.1 (CH_2), 14.4 (CH) and 10.5 (CH_2); *m/z* (ESI)

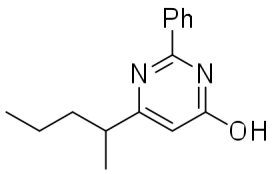
found 219.1498. (M+H, $C_{13}H_{19}N_2O$ requires 219.1492). Found 241.1312 (M+Na, $C_{13}H_{18}N_2NaO$ requires 241.1311).

2-Methyl-6-(pent-2-yl)pyrimidin-4-ol, 5n



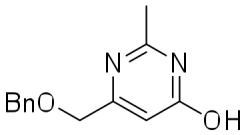
Following **procedure B**, methylvaleraldehyde gave, after purification by column chromatography (2% MeOH/CH_2Cl_2) the desired pyrimidinol **5n** (45 mg, 50%) as a colourless oil. R_f 0.3 (10% MeOH/CH_2Cl_2); $\nu_{max}(CHCl_3)/cm^{-1}$ 2964, 2933, 2874, 1661, 1604, 1563, 1467, 1382, 1242, 1186, 964, 925 and 855; The ^1H NMR spectrum showed that the product exists as a 4:1 mixture of the enol and amide forms. δ_H (400 MHz; $CDCl_3$): Data for the enol form: 13.34 (1H, br s), 6.14 (1H, s), 2.59 (1H, *app* sext, *J* 7.2), 2.45 (3H, s), 1.71□1.60 (1H, m), 1.53□1.38 (1H, m), 1.35□1.23 (2H, m), 1.21□1.17 (3H, m), 0.92□0.86 (3H, m). Data for the amide form, where different from the enol form: 7.75 (1H, s), 2.89 (1H, *app* sext, *J* 7.2), 2.44 (3H, s); δ_C (100 MHz; $CDCl_3$) 175.2 (C), 166.3 (C), 165.1 (C), 158.5 (C), 156.7 (C), 151.2 (CH), 130.3 (C), 108.0 (CH), 41.3 (CH), 37.8 (CH_2), 31.2 (CH), 21.8 (CH_3), 21.5 (CH_3), 20.8 (CH_2), 20.6 (CH_2), 19.6 (CH_3), 19.5 (CH_3) and 14.2 (CH_3). More peaks than expected in ^{13}C NMR due to the presence of enol and amide forms. *m/z* (ESI) found 181.1339 (M+H requires $C_{10}H_{17}N_2O$ 181.1335). Found 203.1158 (M+Na, $C_{10}H_{16}N_2NaO$ requires 203.1155).

6-(Pent-2-yl)-2-phenylpyrimidin-4-ol, 5o



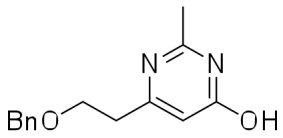
Following **procedure B**, 2-methylvaleraldehyde gave, after purification by column chromatography (2% MeOH/CH_2Cl_2) the pyrimidinol **5o** (75 mg, 62%) as colorless needles, mp 99-101°C. (Found: C, 74.0; H, 7.4; N, 11.5. $C_{15}H_{18}N_2O$ requires C, 74.4; H, 7.5; N, 11.6); R_f 0.4 (10% MeOH/CH_2Cl_2); $\nu_{max}(CHCl_3)/cm^{-1}$ 3069, 3008, 2962, 2933, 2874, 1655, 1596, 1570, 1546, 1501, 1458, 1443, 1401, 1380, 1311, 1184, 987 and 855; δ_H (400 MHz; $CDCl_3$) 13.05 (1H, br s), 8.27□8.24 (2H, m), 7.56□7.53 (3H, m), 6.28 (1H, s), 2.71 (1H, *app* sext, *J* 6.8), 1.82□1.73 (1H, m), 1.57□1.48 (1H, m), 1.40□1.29 (2H, m), 1.27 (3H, d, *J* 6.8), 0.92 (3H, t, *J* 7.4); δ_C (100 MHz; $CDCl_3$) 174.5 (C), 166.0 (C), 156.5 (C), 132.6 (C), 131.9 (CH), 129.0 (CH), 128.0 (CH), 109.4 (CH), 41.3 (CH), 37.9 (CH_2), 20.7 (CH_2), 19.6 (CH_3) and 14.3 (CH_3); *m/z* (ESI) found 265.1306 (M+Na, $C_{15}H_{18}N_2NaO$ requires 265.1311).

6-(Benzyloxymethyl)-2-methylpyrimidin-4-ol, 5p



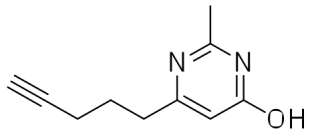
The required aldehyde, 2-(benzyloxy)acetaldehyde was prepared from 2-(benzyloxy)ethanol following the literature procedure.[8] Following **procedure B**, 2-(benzyloxy)acetaldehyde gave, after purification by column chromatography (3% MeOH/CH_2Cl_2) the pyrimidinol **5p** (81 mg, 70%) as colorless needles, mp 169-171°C. R_f 0.2 (10% MeOH/CH_2Cl_2); $\nu_{max}(CHCl_3)/cm^{-1}$ 3001, 2864, 2760, 1668, 1608, 1572, 1497, 1454, 1385, 1329, 1240, 1184, 1114, 1029, 970, 909 and 860; δ_H (400 MHz; $CDCl_3$) 13.41 (1H, br s), 7.28□7.40 (5H, m), 6.56 (1H, s), 4.65 (2H, s), 4.41 (2H, s), 2.47 (3H, s); δ_C (100 MHz; $CDCl_3$) 167.0 (C), 166.1 (C), 159.0 (C), 137.6 (C), 128.6 (CH), 128.0 (CH), 127.8 (CH), 107.8 (CH), 73.3 (CH_2), 71.3 (CH_2) and 21.7 (CH_3); *m/z* (ESI) found 231.1124 (M+H, $C_{13}H_{15}N_2O_2$ requires 231.1128). Found 253.0943 (M+Na, $C_{13}H_{14}N_2NaO_2$ requires 253.0947.

6-(2-(Benzyloxy)ethyl)-2-methylpyrimidin-4-ol, 5q



The required aldehyde, 3-(benzyloxy)propanal was prepared from propane-1,3-diol following the literature procedure.[9] Following a modification of **procedure B** in which 10 mol% $BF_3{\cdot}OEt_2$ was employed for the flow process, 3-(benzyloxy)propanal gave, after purification by column chromatography (3% MeOH/CH_2Cl_2) the pyrimidinol **5q** (64 mg, 52%) as colorless needles, mp 100-102°C. R_f 0.3 (10% MeOH/CH_2Cl_2); ν_{max}($CHCl_3$)/cm^{-1}3008, 2862, 1664, 1608, 1455, 1182, 1100, 968 and 857; The ^{1}H NMR spectrum showed that the product exists as a 7:1 mixture of the enol and amide forms. δ_H (400 MHz; $CDCl_3$): Data for the enol form: 13.31 (1H, br s), 7.35□7.25 (5H, m), 6.26 (1H, s), 4.52 (2H, s), 3.80 (2H, t, *J* 6.4), 2.82 (2H, t, *J* 6.4), 2.45 (3H, s). Data for the amide form, where different from enol form: 7.89 (1H, s), 3.70 (2H, t, *J* 6.4), 2.77 (2H, t, *J* 6.4); δ_C (100 MHz; $CDCl_3$) 167.4 (C), 165.8 (C), 165.2 (C), 158.6 (C), 157.5 (C), 153.7 (CH), 138.4 (C), 138.2 (C), 128.5 (CH), 128.4 (CH), 127.8 (CH), 127.7 (CH), 127.6 (CH), 127.5 (CH), 122.5 (C), 110.5 (CH), 73.1 (CH_2), 73.0 (CH_2), 68.0 (CH_2), 67.8 (CH_2), 38.1 (CH_2), 27.8 (CH_2), 21.7 (CH_3) and 21.5 (CH_3). More peaks than expected in ^{13}C NMR due to the presence of enol and amide forms. *m/z* (ESI) found 245.1282 (M+H, $C_{14}H_{17}N_2O_2$ requires 245.1285). Found 267.1101 (M+Na, $C_{14}H_{16}N_2NaO_2$ requires 267.1104.

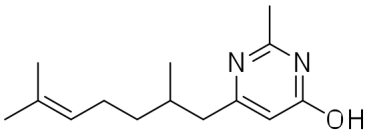
2-Methyl-6-(pent-4-ynyl)pyrimidin-4-ol, 5r



The required aldehyde, hex-5-ynal was prepared from hex-5-yn-1-ol following the literature procedure.[10] Following **procedure B**, hex-5-ynal gave, after purification by column chromatography (3%

MeOH/CH_2Cl_2) the pyrimidinol **5r** (55 mg, 63%) as colorless needles, mp 122-124°C. R_f 0.3 (10% MeOH/CH_2Cl_2); ν_{max}($CHCl_3$)/cm^{-1} 3385, 3308, 3010, 1664, 1606, 1563, 1443, 1383, 1304, 1240, 1186, 967 and 860. δ_H (400 MHz; $CDCl_3$) 13.29 (1H, br s), 6.19 (1H, s), 2.64 (2H, t, *J* 7.6), 2.46 (3H, s), 2.25 (2H, dt, *J* 2.4 and 6.8), 1.98 (1H, t, *J* 2.4), 1.86□1.94 (2H, m); δ_C (100 MHz; $CDCl_3$) 169.5 (C), 165.9 (C), 158.7 (C), 109.7 (CH), 83.6 (C), 69.3 (CH), 36.5 (CH_2), 26.7 (CH_2), 21.8 (CH_3) and 18.0 (CH_2); *m/z* (ESI) found 177.1031 (M+H, $C_{10}H_{13}N_2O$ requires 177.1022). Found 199.0844 (M+Na, $C_{10}H_{12}N_2NaO$ requires 199.0842.

6-(2,6-Dimethylhept-5-enyl)-2-methylpyrimidin-4-ol, 5s



Following **procedure C**, (±)-citronellal gave, after purification by column chromatography (3% MeOH/CH_2Cl_2) the pyrimidinol **5s** (41 mg, 35%) as colorless needles, mp 70-71°C. (Found: C, 71.3; H, 9.4; N, 11.7. $C_{14}H_{22}N_2O$ requires C, 71.8; H, 9.5; N, 12.0); R_f 0.4 (10% MeOH/CH_2Cl_2); ν_{max}($CHCl_3$)/cm^{-1} 3136, 2959, 2930, 2853, 2759, 1661, 1607, 1562, 1456, 1402, 1382, 1346, 1305, 1185, 1110, 1081, 1041, 1008, 969, 889 and 865. δ_H (400 MHz; $CDCl_3$) 13.12 (1H, br s), 6.14 (1H, s), 5.11□5.07 (1H, m), 2.53 (1H, dd, *J* 13.4 and 6.0), 2.47 (3H, s), 2.29 (1H, dd, *J* 13.4 and 8.4), 2.09□1.92 (3H, m), 1.70 (3H, s), 1.64 (3H, s), 1.43□1.34 (1H, m), 1.25□1.16 (1H, m), 0.90 (3H, d, *J* 6.8); δ_C (100 MHz; $CDCl_3$) 170.0 (C), 166.0 (C), 158.5 (C), 131.5 (C), 124.5 (CH), 110.3 (CH), 45.5 (CH_2), 36.9 (CH_2), 32.0 (CH), 25.8 (CH_3), 25.5 (CH_2), 21.7 (CH_3), 19.3 (CH_3) and 17.8 (CH_3); *m/z* (ESI) found 235.1804 (M+H, $C_{14}H_{23}N_2O$ requires 235.1805). Found 257.1618 (M+Na, $C_{14}H_{22}N_2NaO$ requires 257.1624.

References

1) Balaji, B. S.; Chanda, B. M. *Tetrahedron* **1998**, *54*, 13237-13252

2) Alker, D.; Campbell, S. F.; Cross, P. E.; Burges, R. A.; Carter, A. J.; Gardiner, D. G. *J. Med. Chem.* **1989**, *32*, 2381-2388

3) Murray, T. P.; Hay, J. V.; Portlock, D. E.; Wolfe, J. F. *J. Org. Chem.* **1974**, *39*, 595-600

4) Sakamoto, T.; Yoshizawa, H.; Yamanaka, H. *Chem. Pharm. Bull*. **1984**, *32*, 2005-2010

5) Parry, R. J.; Yan, L.; Gomez, E. E. *J. Am. Chem. Soc.* **1992**, *114*, 5946-5959

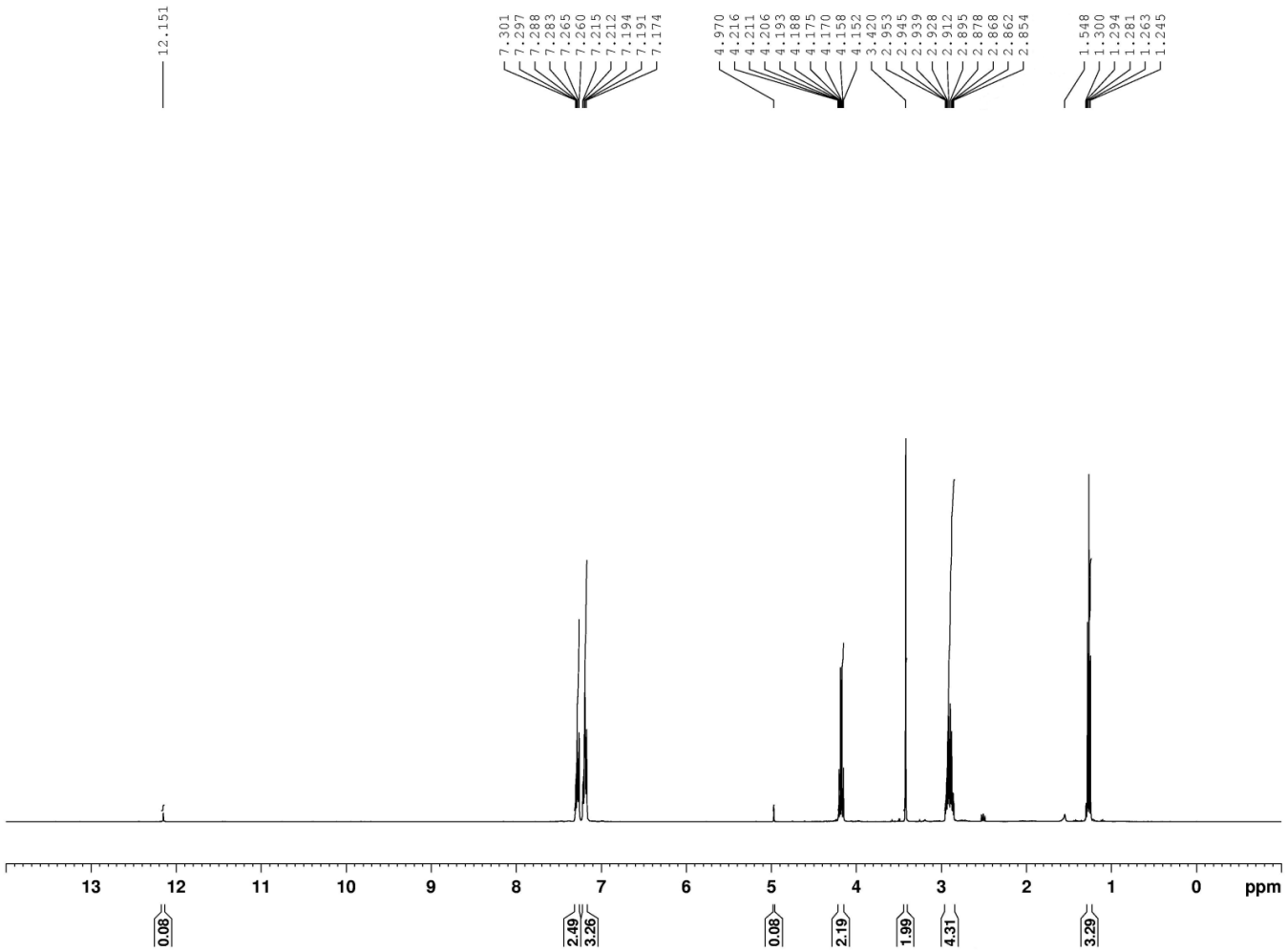
6) Rodriguez, A. L.; Tamrazi, A.; Collins, M. L.; Katzenellenbogen, J, A. *J. Med. Chem.* **2004**, *47*, 600-611

7) Sato, M.; Ogasawara, H.; Komatsu, S.; Kato, T. *Chem. Pharm. Bull.* **1984**, *32*, 3848-3856

8) Takano, D.; Nagamitsu, T.; Ui, H.; Shiomi, K.; Yamaguchi, Y.; Masuma, R.; Kuwajima, I.; Õmura, S. *Tetrahedron Lett.* **2001**, *42*, 3017-3020

9) Schomaker, J. M.; Pulgam, V. R.; Borhan, B. *J. Am. Chem. Soc.* **2004**, *126*, 13600-13601

10) Böttcher, T.; Sieber, S, A. *Angew. Chem. Int. Ed.* **2008** *47*, 4600-4603

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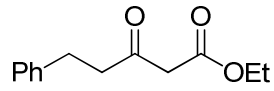

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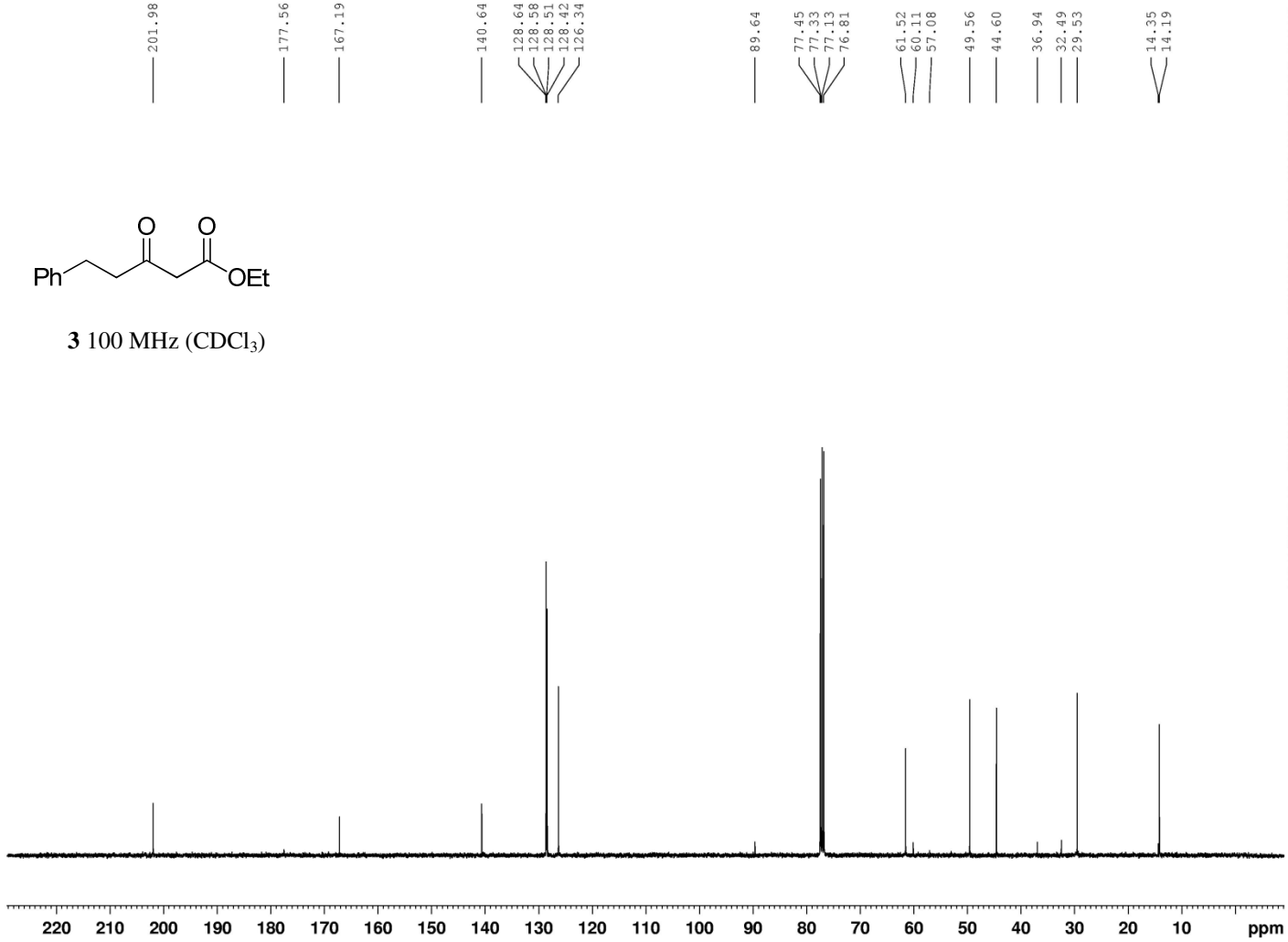

3 100 MHz ($CDCl_3$)

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PL2                -2.00 dB
PL12               17.00 dB
PL13               19.30 dB
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PL12W        0.20147727 W
PL13W        0.11863863 W
SFO2       400.1316005 MHz

F2 - Processing parameters
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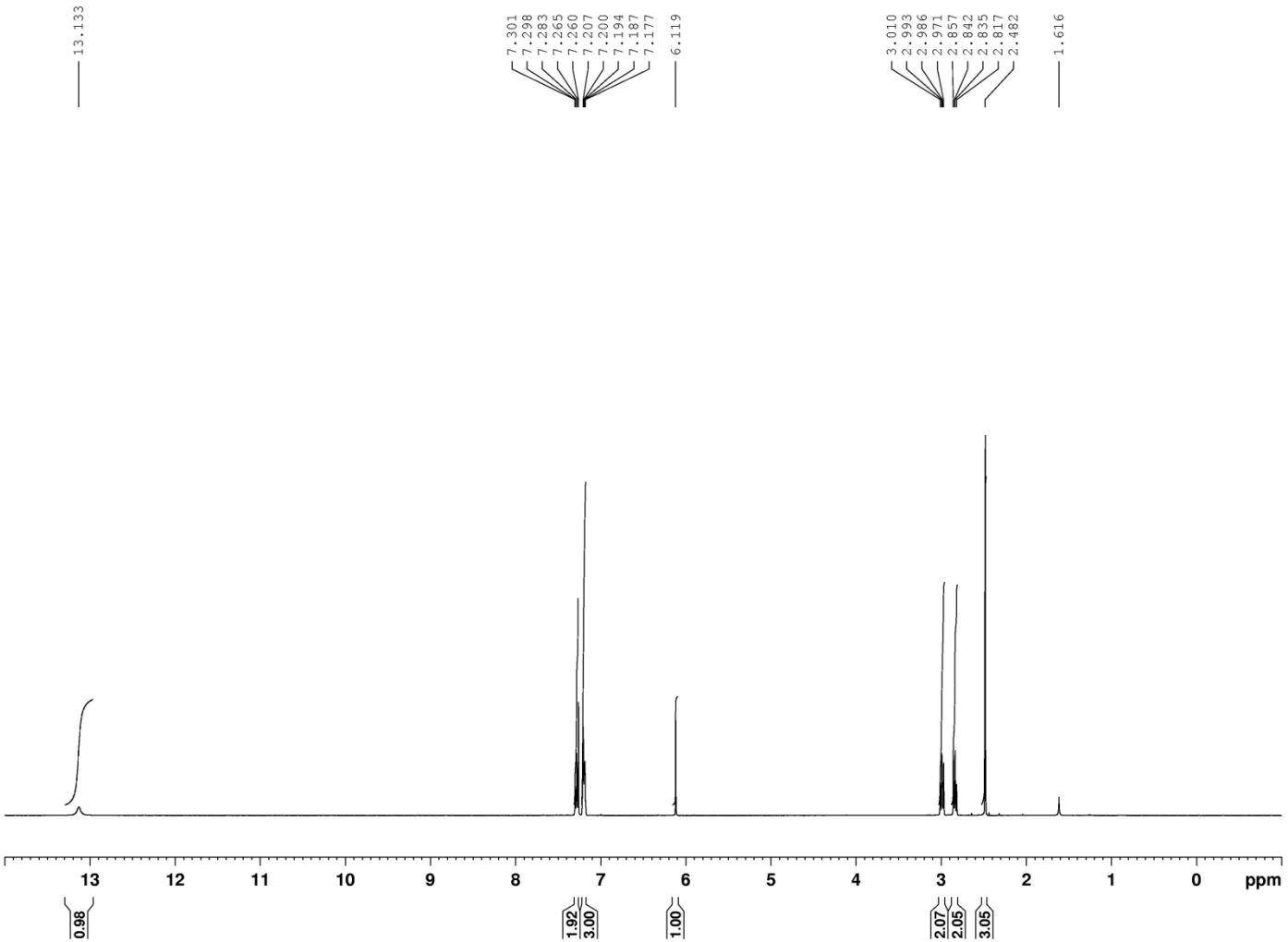


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DE                 6.50 usec
TE                298.0 K
D1             1.00000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                  1H
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PL1                -0.90 dB
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SFO1         400.0724706 MHz

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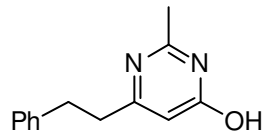
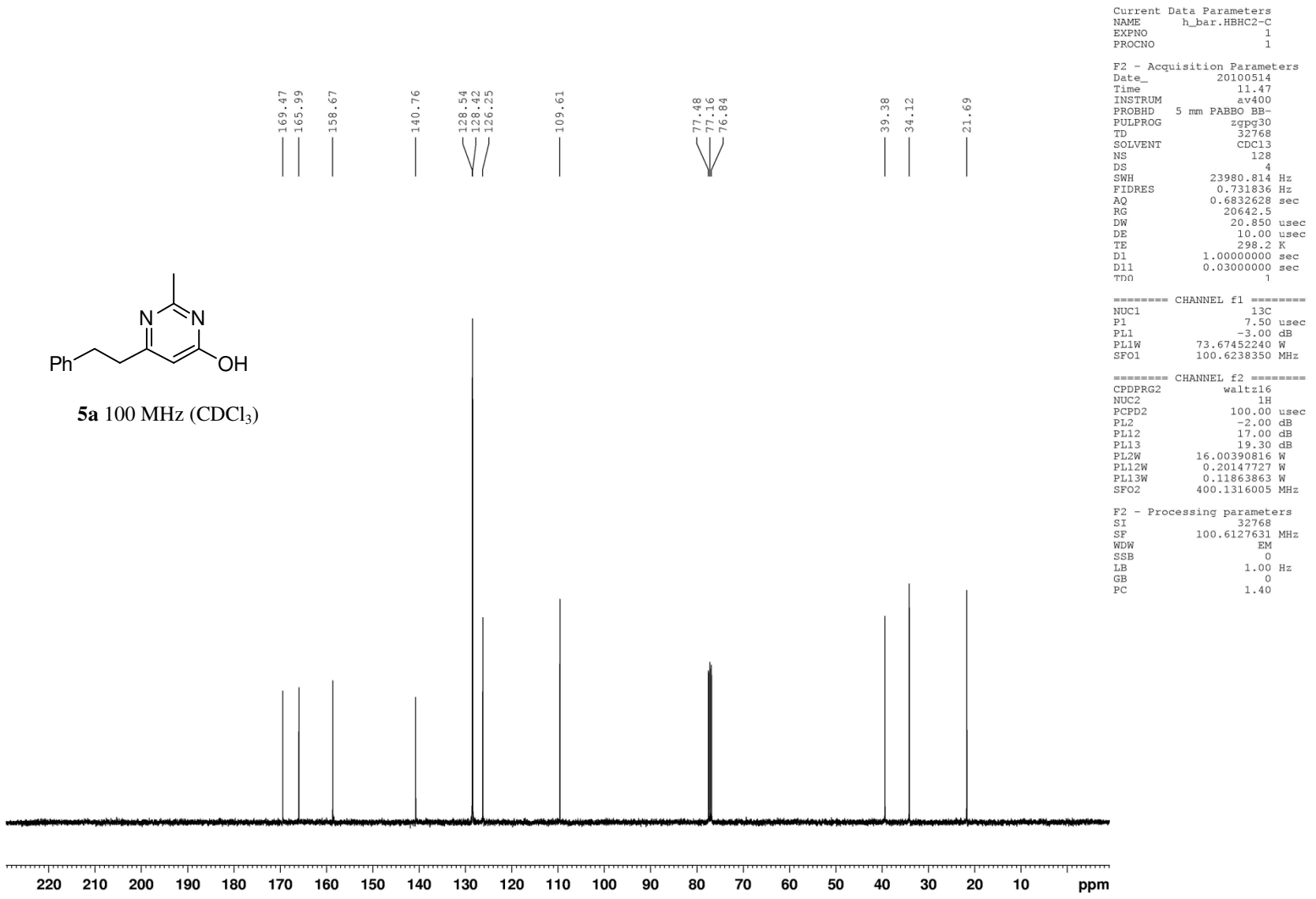


5a 400 MHz ($CDCl_3$)



5a 100 MHz ($CDCl_3$)

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PROCNO                 1

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DE               10.00 usec
TE               298.2 K
D1           1.00000000 sec
D11          0.03000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                 13C
P1                  7.50 usec
PL1                -3.00 dB
PL1W        73.67452240 W
SFO1       100.6238350 MHz

======== CHANNEL f2 ========
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PCPD2            100.00 usec
PL2               -2.00 dB
PL12              17.00 dB
PL13              19.30 dB
PL2W        16.00390816 W
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SFO2       400.1316005 MHz

F2 - Processing parameters
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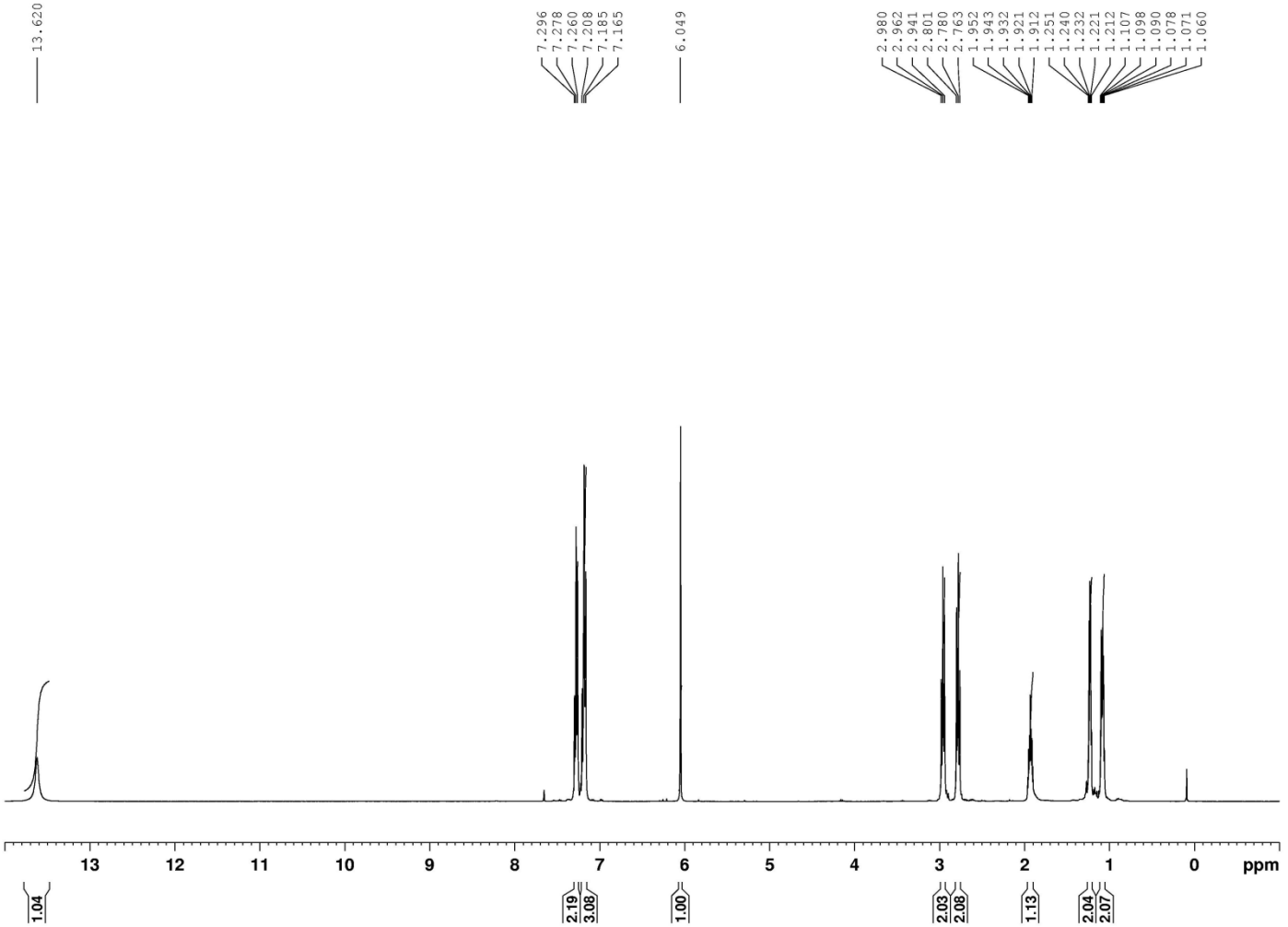


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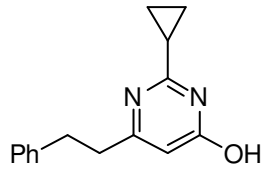
EXPNO                  4
PROCNO                 1

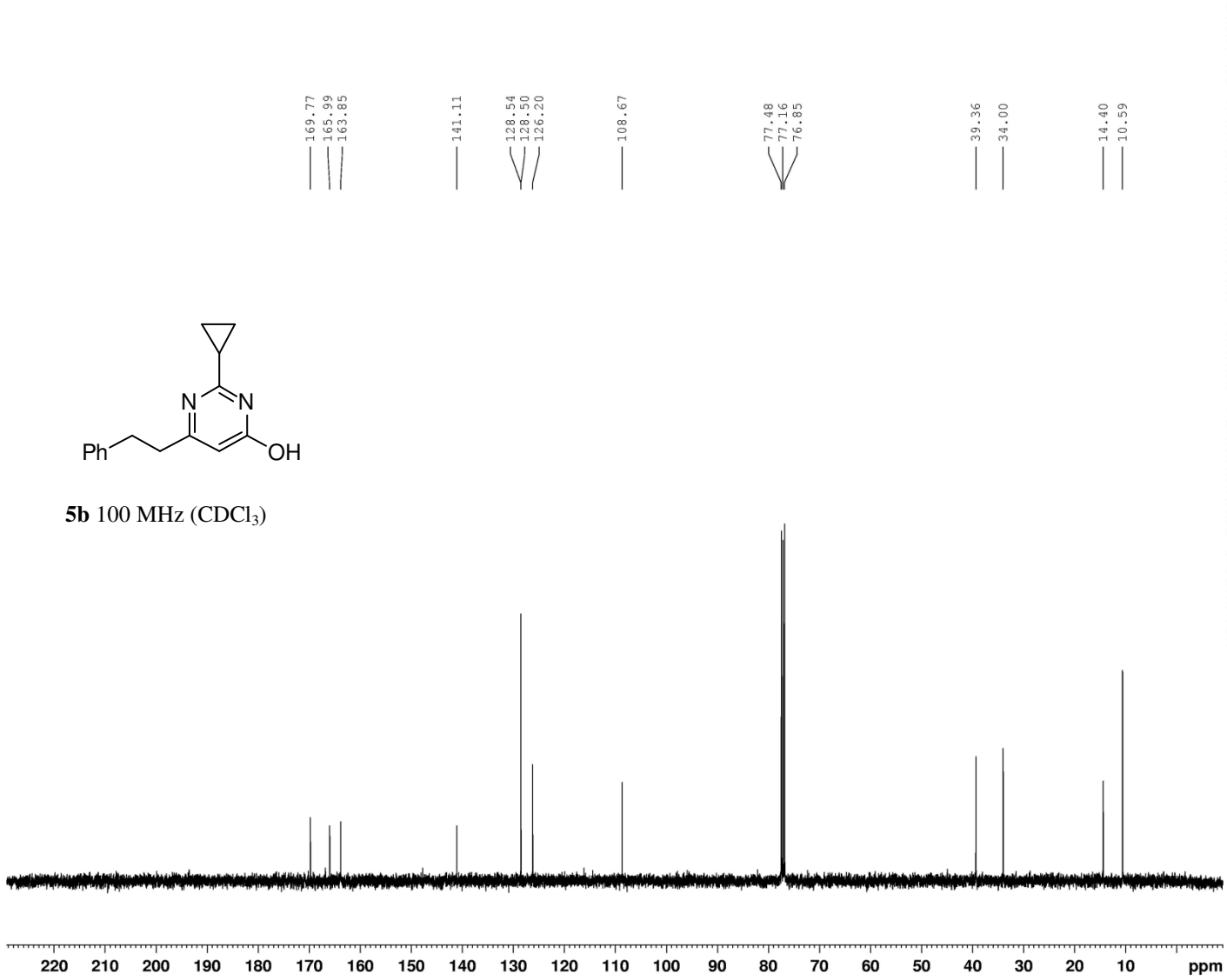
F2 - Acquisition Parameters
Date_           20100629
Time               15.40
INSTRUM          av3400
PROBHD   5 mm PABBI 1H/
PULPROG            zg30
TD                65536
SOLVENT           CDCl3
NS                   16
DS                    2
SWH            8223.685 Hz
FIDRES        0.125483 Hz
AQ            3.9846387 sec
RG                 45.2
DW               60.800 usec
DE                6.50 usec
TE               298.0 K
D1            1.00000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                  1H
P1                 7.30 usec
PL1               -0.90 dB
PL1W        11.52680206 W
SFO1       400.0724706 MHz

F2 - Processing parameters
SI                 65536
SF         400.0700097 MHz
WDW                   EM
SSB                    0
LB                0.30 Hz
GB                     0
PC                1.00
```



5b 400 MHz ($CDCl_3$)



5b 100 MHz ($CDCl_3$)

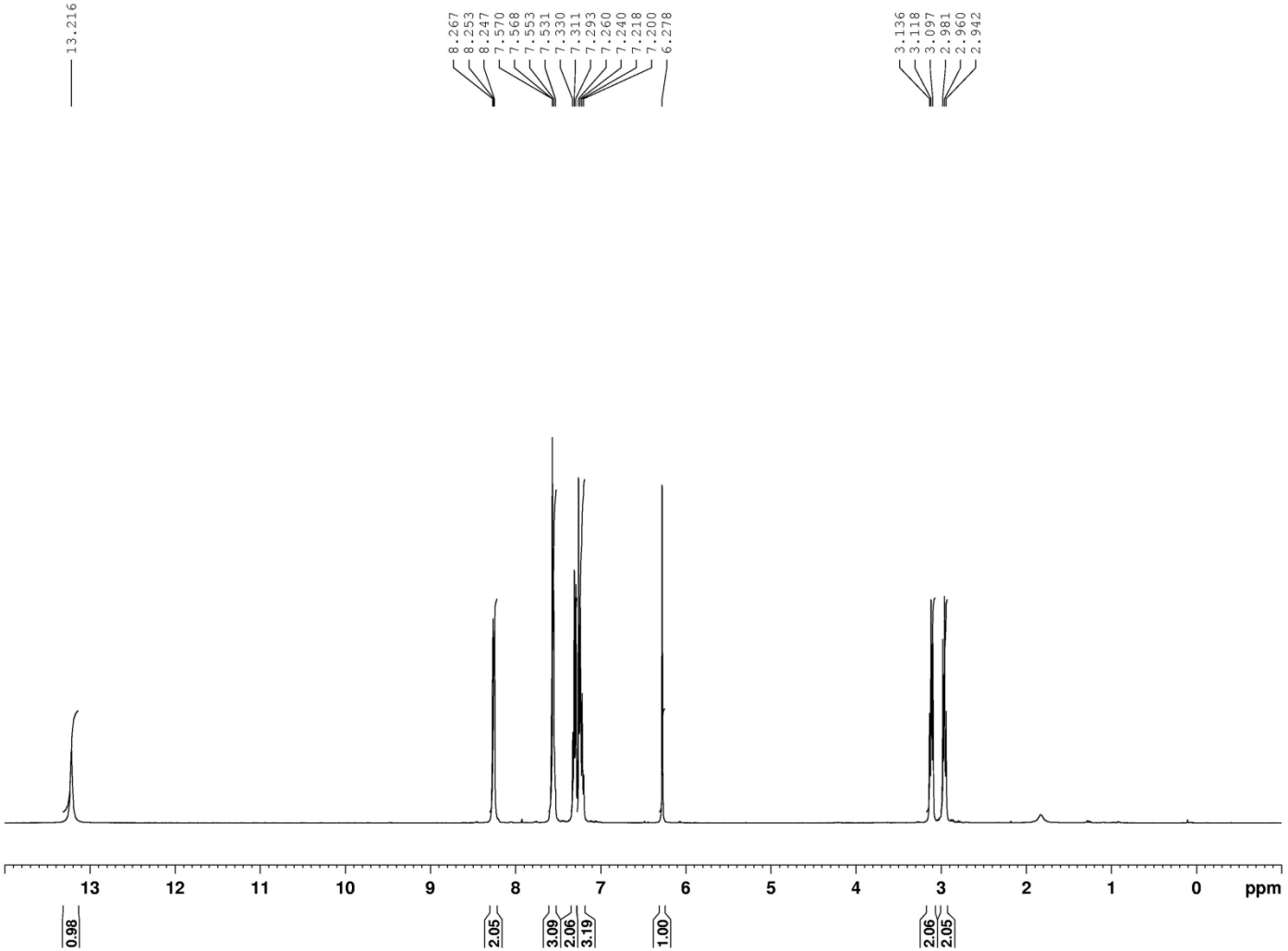
```
Current Data Parameters
NAME         h_bar.HBHC3
EXPNO                  1
PROCNO                 1

F2 - Acquisition Parameters
Date_           20100519
Time                8.06
INSTRUM           av400
PROBHD   5 mm PABBO BB-
PULPROG          zgpg30
TD                32768
SOLVENT           CDCl3
NS                  128
DS                    4
SWH           23980.814 Hz
FIDRES         0.731836 Hz
AQ            0.6832628 sec
RG              14596.5
DW               20.850 usec
DE               10.00 usec
TE               298.2 K
D1          1.00000000 sec
D11         0.03000000 sec
TD0                   1

======== CHANNEL f1 ========
NUC1                 13C
P1                 7.50 usec
PL1               -3.00 dB
PL1W        73.67452240 W
SFO1       100.6238350 MHz

======== CHANNEL f2 ========
CPDPRG2          waltz16
NUC2                 1H
PCPD2            100.00 usec
PL2               -2.00 dB
PL12              17.00 dB
PL13              19.30 dB
PL2W        16.00390816 W
PL12W        0.20147727 W
PL13W        0.11863863 W
SFO2       400.1316005 MHz

F2 - Processing parameters
SI                32768
SF          100.6127557 MHz
WDW                  EM
SSB                   0
LB                 1.00 Hz
GB                    0
PC                 1.40
```

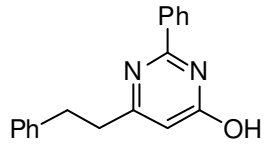



```
Current Data Parameters
NAME         h_bar.HBHC4
EXPNO                  3
PROCNO                 1

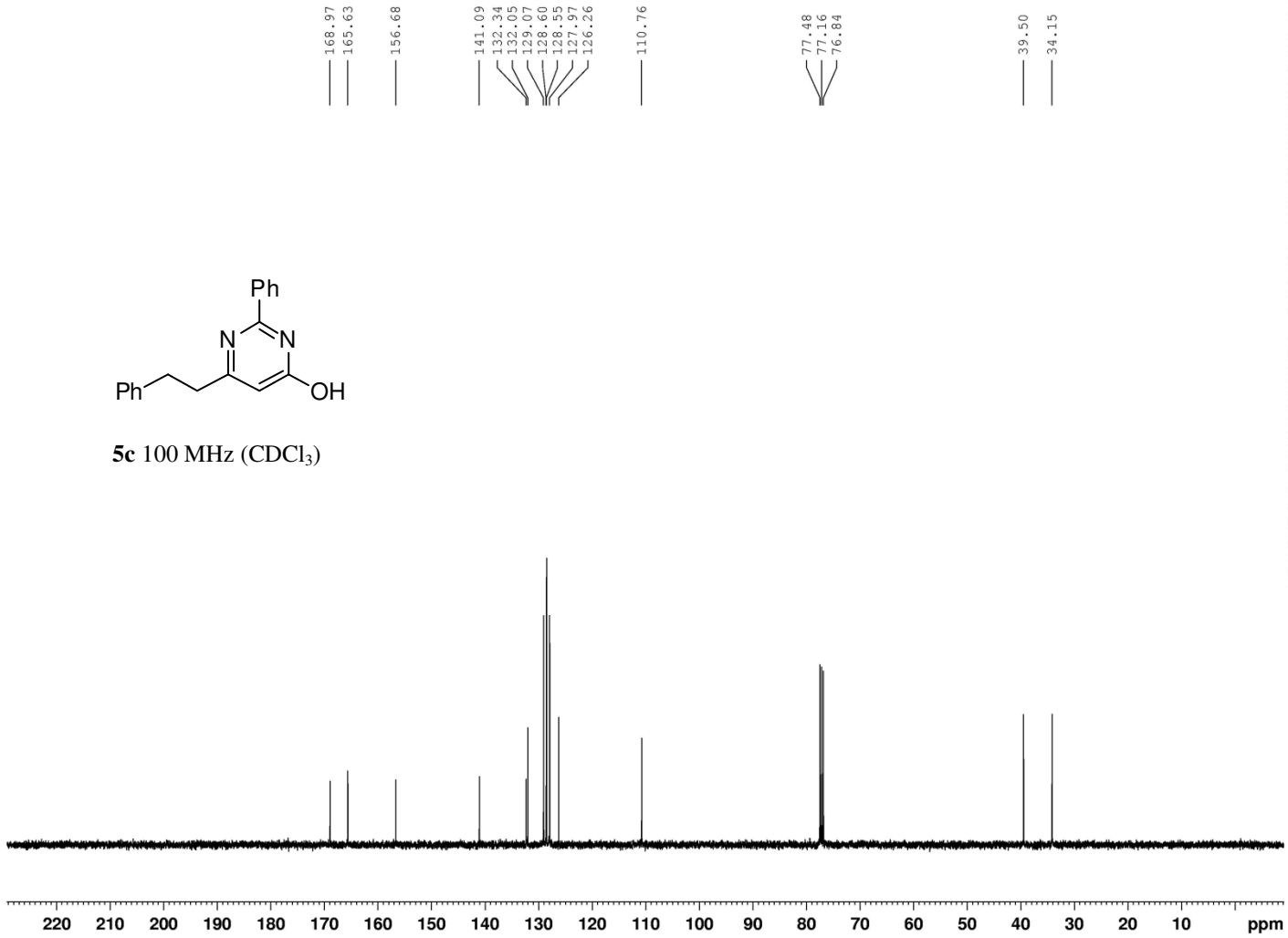
F2 - Acquisition Parameters
Date_           20100520
Time               12.22
INSTRUM          av3400
PROBHD   5 mm PABBI 1H/
PULPROG             zg30
TD                 65536
SOLVENT            CDCl3
NS                    16
DS                     2
SWH             8223.685 Hz
FIDRES          0.125483 Hz
AQ             3.9846387 sec
RG                  50.8
DW                60.800 usec
DE                 6.50 usec
TE                298.0 K
D1           1.00000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                  1H
P1                  7.30 usec
PL1                -0.90 dB
PL1W        11.52680206 W
SFO1        400.0724706 MHz

F2 - Processing parameters
SI                 65536
SF          400.0700106 MHz
WDW                   EM
SSB                    0
LB                  0.30 Hz
GB                     0
PC                  1.00
```



5c 400 MHz ($CDCl_3$)



5c 100 MHz ($CDCl_3$)

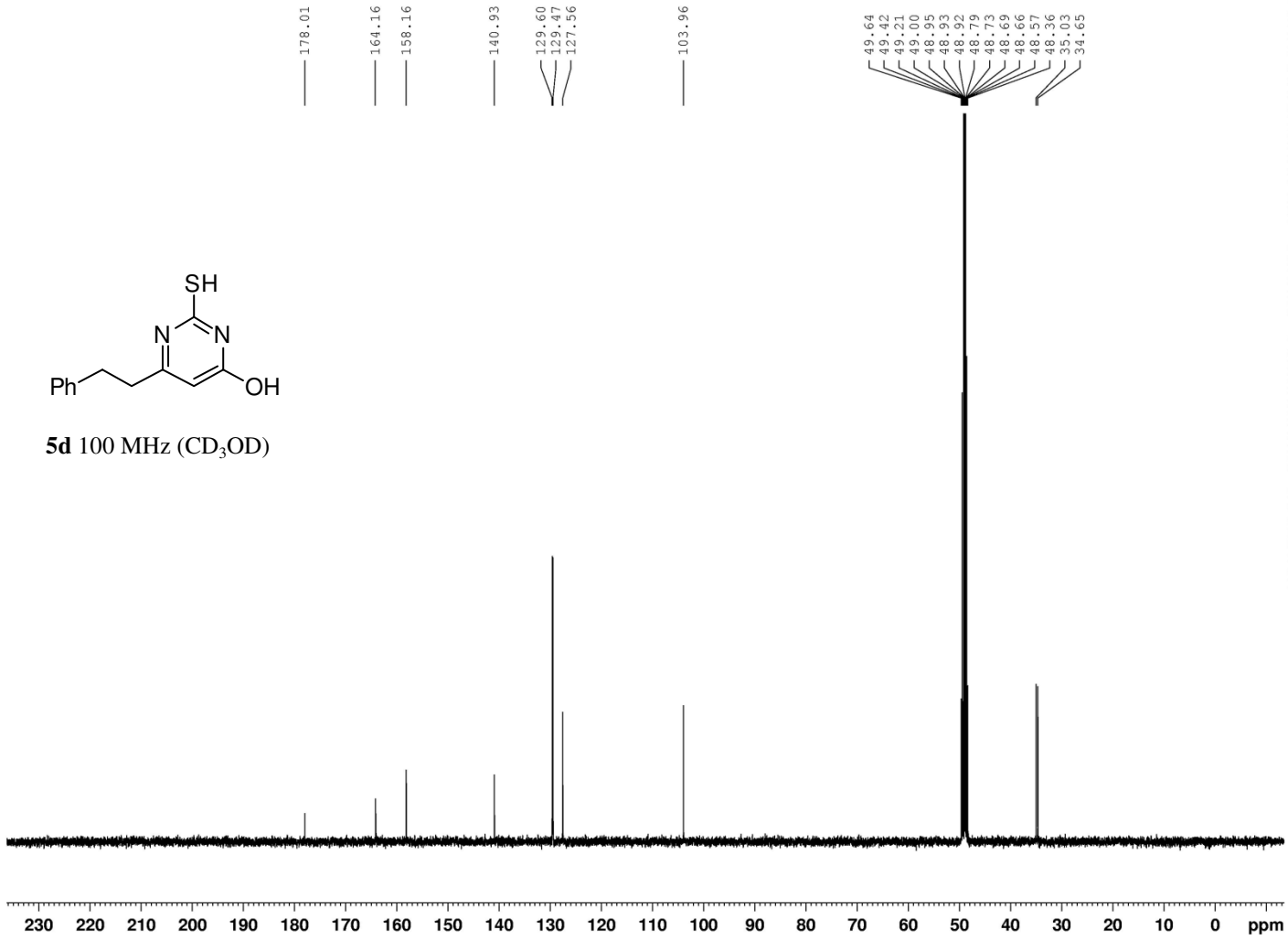
```
Current Data Parameters
NAME       h_bar.HBHC4-c
EXPNO                  1
PROCNO                 1

F2 - Acquisition Parameters
Date_           20100520
Time               14.50
INSTRUM            av400
PROBHD   5 mm PABBO BB-
PULPROG          zgpg30
TD                 32768
SOLVENT            CDCl3
NS                   128
DS                     4
SWH            23980.814 Hz
FIDRES          0.731836 Hz
AQ             0.6832628 sec
RG                 18390.4
DW                20.850 usec
DE                10.00 usec
TE                298.2 K
D1           1.00000000 sec
D11          0.03000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                 13C
P1                  7.50 usec
PL1                -3.00 dB
PL1W        73.67452240 W
SFO1       100.6238350 MHz

======== CHANNEL f2 ========
CPDPRG2          waltz16
NUC2                 1H
PCPD2             100.00 usec
PL2                -2.00 dB
PL12               17.00 dB
PL13               19.30 dB
PL2W        16.00390816 W
PL12W        0.20147727 W
PL13W        0.11863863 W
SFO2       400.1316005 MHz

F2 - Processing parameters
SI                 32768
SF         100.6127594 MHz
WDW                   EM
SSB                    0
LB                  1.00 Hz
GB                     0
PC                  1.40
```



5d 100 MHz (CD_3OD)

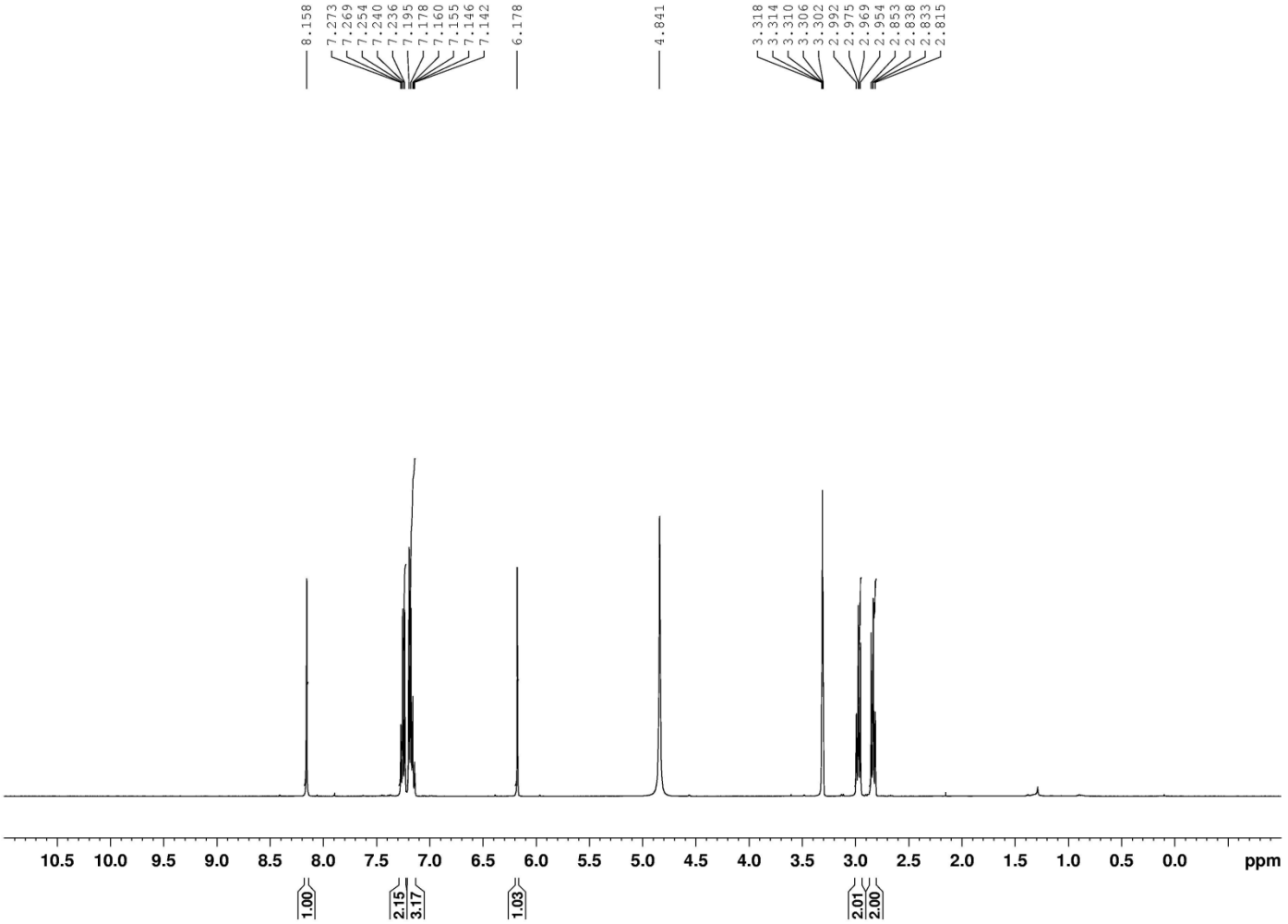
```
Current Data Parameters
NAME         h_bar.HBHC5
EXPNO                  4
PROCNO                 1

F2 - Acquisition Parameters
Date_           20100522
Time               11.10
INSTRUM            av400
PROBHD   5 mm PABBO BB-
PULPROG           zgpg30
TD                 32768
SOLVENT             MeOD
NS                   512
DS                     2
SWH            25125.629 Hz
FIDRES          0.766773 Hz
AQ              0.6521332 sec
RG                20642.5
DW                19.900 usec
DE                10.00 usec
TE                298.2 K
D1             1.00000000 sec
D11            0.03000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                  13C
P1                   7.50 usec
PL1                 -3.00 dB
PL1W         73.67452240 W
SFO1        100.6238350 MHz

======== CHANNEL f2 ========
CPDPRG2           waltz16
NUC2                  1H
PCPD2              100.00 usec
PL2                 -2.00 dB
PL12                17.00 dB
PL13                19.30 dB
PL2W         16.00390816 W
PL12W         0.20147727 W
PL13W         0.11863863 W
SFO2        400.1316005 MHz

F2 - Processing parameters
SI                 32768
SF          100.6126289 MHz
WDW                   EM
SSB                    0
LB                  1.00 Hz
GB                     0
PC                  1.40
```

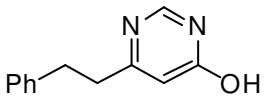



```
Current Data Parameters
NAME         h_bar.HBHC1
EXPNO                  3
PROCNO                 1

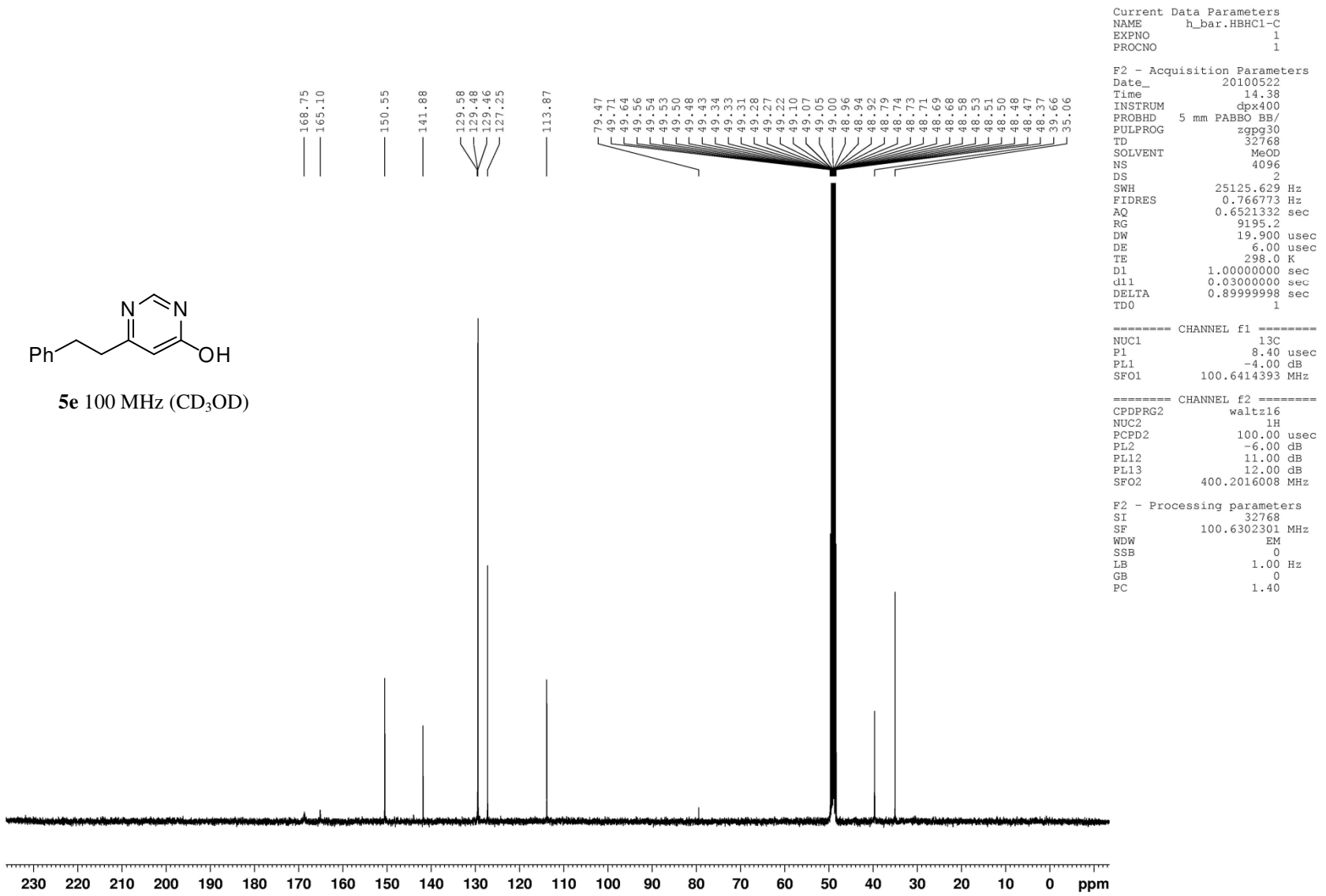
F2 - Acquisition Parameters
Date_           20100520
Time               12.45
INSTRUM          av3400
PROBHD   5 mm PABBI 1H/
PULPROG            zg30
TD                65536
SOLVENT            MeOD
NS                   16
DS                    2
SWH            8223.685 Hz
FIDRES        0.125483 Hz
AQ            3.9846387 sec
RG                  287
DW               60.800 usec
DE                6.50 usec
TE                298.0 K
D1           1.00000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                  1H
P1                 7.30 usec
PL1               -0.90 dB
PL1W       11.52680206 W
SFO1       400.0724706 MHz

F2 - Processing parameters
SI                 65536
SF         400.0700107 MHz
WDW                   EM
SSB                    0
LB                0.30 Hz
GB                     0
PC                1.00
```



5e 400 MHz (CD_3OD)



5e 100 MHz (CD_3OD)

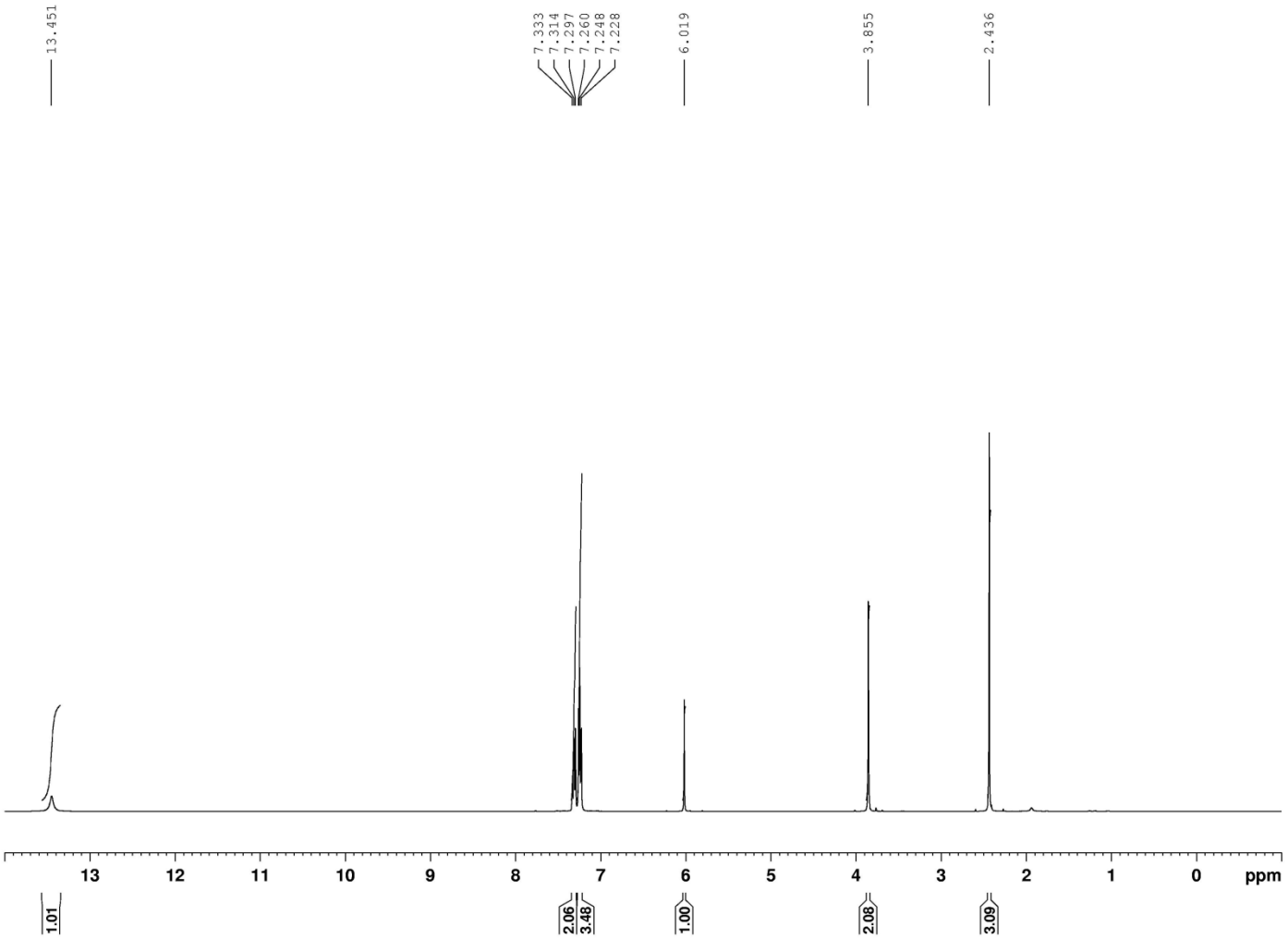
```
Current Data Parameters
NAME       h_bar.HBHC1-C
EXPNO                  1
PROCNO                 1

F2 - Acquisition Parameters
Date_           20100522
Time               14.38
INSTRUM          dpx400
PROBHD   5 mm PABBO BB/
PULPROG          zgpg30
TD                32768
SOLVENT            MeOD
NS                 4096
DS                    2
SWH           25125.629 Hz
FIDRES         0.766773 Hz
AQ             0.6521332 sec
RG                9195.2
DW                19.900 usec
DE                 6.00 usec
TE                298.0 K
D1           1.00000000 sec
d11          0.03000000 sec
DELTA        0.89999998 sec
TD0                   1

======== CHANNEL f1 ========
NUC1                13C
P1                 8.40 usec
PL1               -4.00 dB
SFO1        100.6414393 MHz

======== CHANNEL f2 ========
CPDPRG2          waltz16
NUC2                1H
PCPD2             100.00 usec
PL2                -6.00 dB
PL12               11.00 dB
PL13               12.00 dB
SFO2        400.2016008 MHz

F2 - Processing parameters
SI                 32768
SF          100.6302301 MHz
WDW                   EM
SSB                    0
LB                 1.00 Hz
GB                     0
PC                 1.40
```

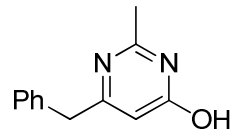



```
Current Data Parameters
NAME        h_bar.HBHC12
EXPNO                  1
PROCNO                 1

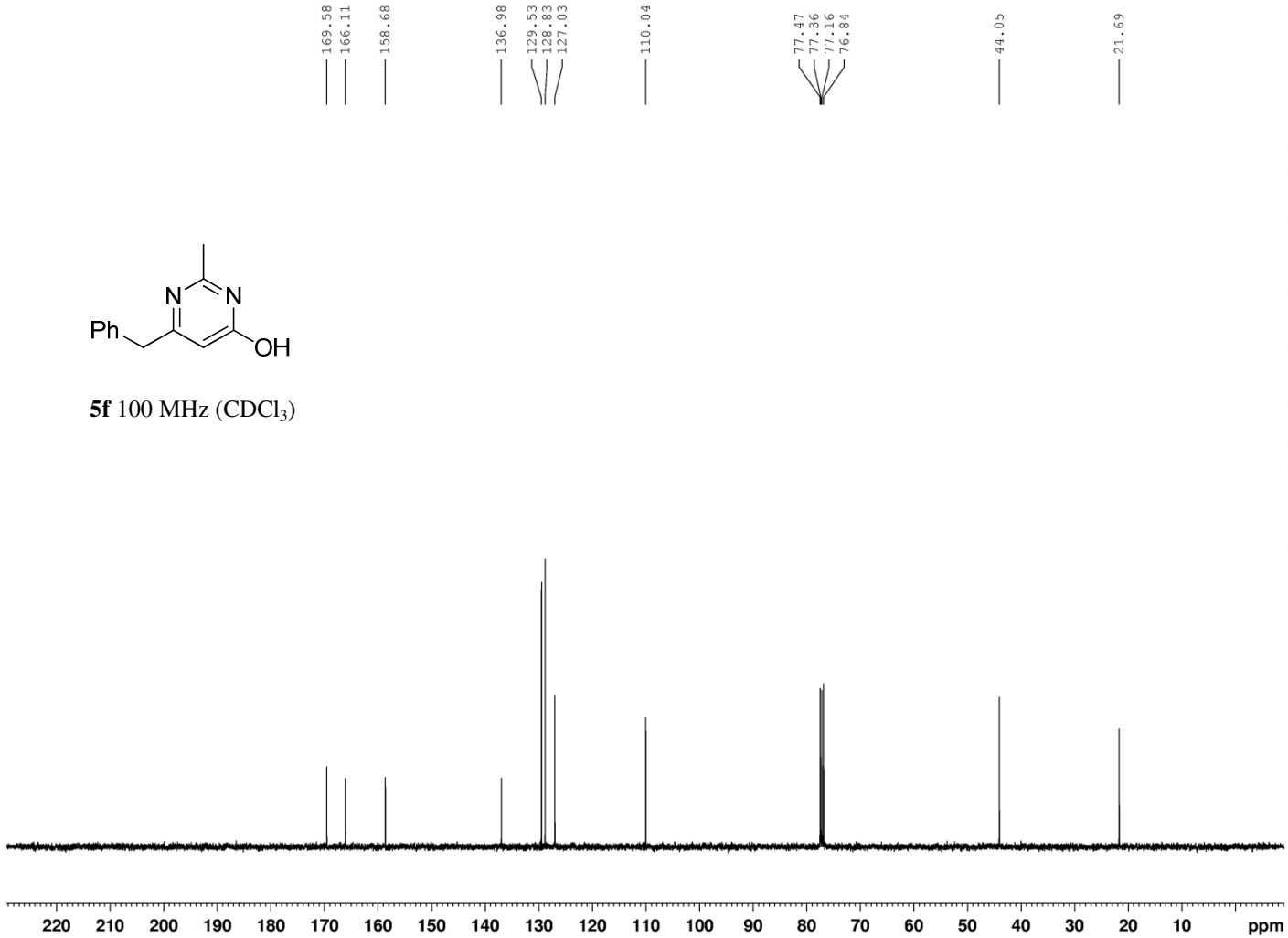
F2 - Acquisition Parameters
Date_           20100522
Time              11.42
INSTRUM           av3400
PROBHD   5 mm PABBI 1H/
PULPROG             zg30
TD                65536
SOLVENT           CDCl3
NS                   16
DS                    2
SWH            8223.685 Hz
FIDRES         0.125483 Hz
AQ             3.9846387 sec
RG                 90.5
DW                60.800 usec
DE                 6.50 usec
TE                298.0 K
D1            1.00000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                  1H
P1                 7.30 usec
PL1               -0.90 dB
PL1W        11.52680206 W
SFO1       400.0724706 MHz

F2 - Processing parameters
SI                 65536
SF         400.0700122 MHz
WDW                   EM
SSB                    0
LB                  0.30 Hz
GB                     0
PC                  1.00
```



5f 400 MHz ($CDCl_3$)



5f 100 MHz ($CDCl_3$)

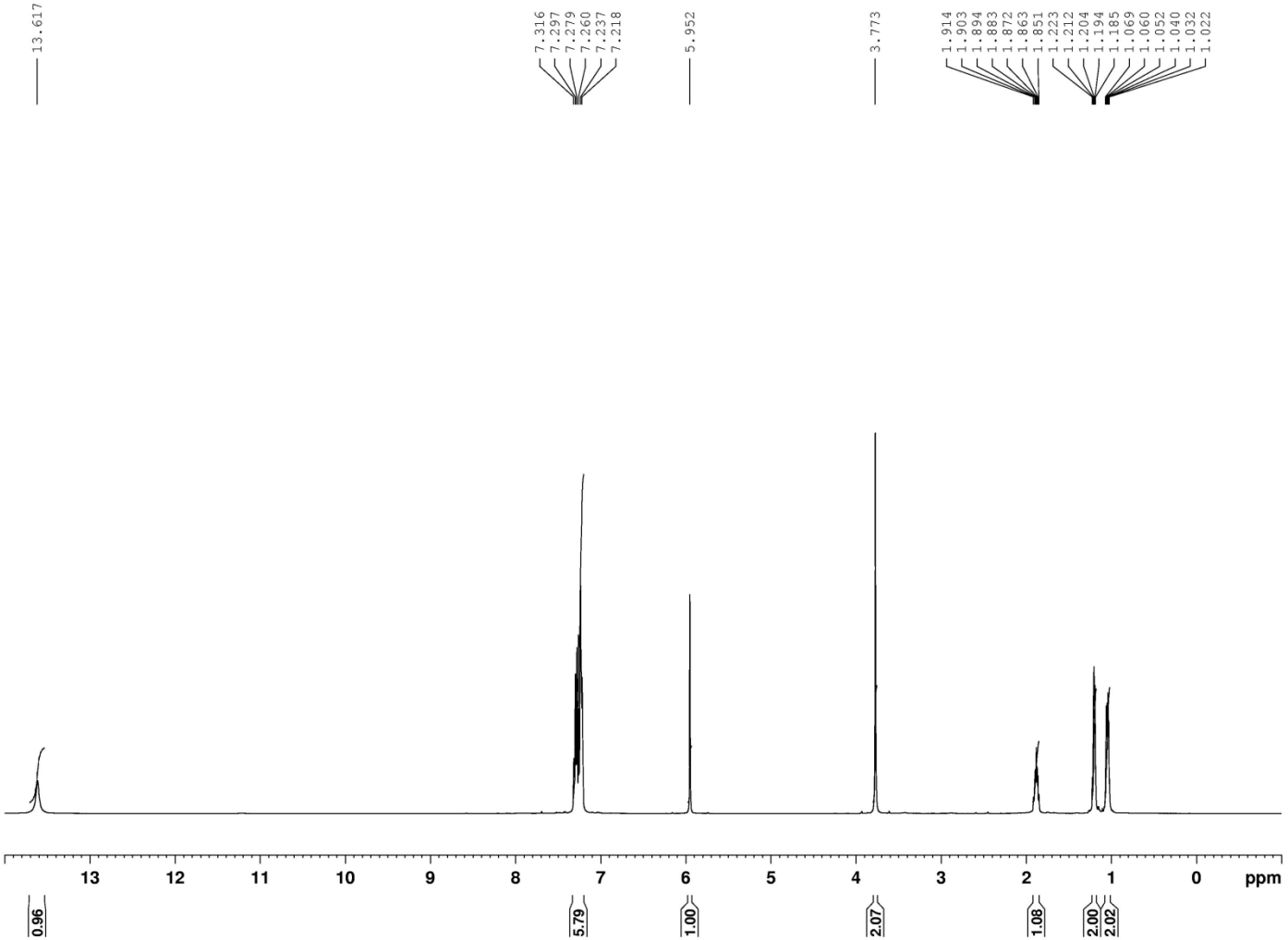
```
Current Data Parameters
NAME      h_bar.HBHC12-c
EXPNO                  1
PROCNO                 1

F2 - Acquisition Parameters
Date_           20100522
Time               12.23
INSTRUM            av400
PROBHD   5 mm PABBO BB-
PULPROG          zgpg30
TD                 32768
SOLVENT            CDCl3
NS                   128
DS                     4
SWH            23980.814 Hz
FIDRES         0.731836 Hz
AQ             0.6832628 sec
RG                 23170.5
DW                20.850 usec
DE                10.00 usec
TE                298.2 K
D1           1.00000000 sec
D11          0.03000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                 13C
P1                  7.50 usec
PL1                -3.00 dB
PL1W        73.67452240 W
SFO1       100.6238350 MHz

======== CHANNEL f2 ========
CPDPRG2          waltz16
NUC2                 1H
PCPD2             100.00 usec
PL2                -2.00 dB
PL12               17.00 dB
PL13               19.30 dB
PL2W        16.00390816 W
PL12W        0.20147727 W
PL13W        0.11863863 W
SFO2       400.1316005 MHz

F2 - Processing parameters
SI                 32768
SF            100.6127587 MHz
WDW                   EM
SSB                    0
LB                  1.00 Hz
GB                     0
PC                  1.40
```

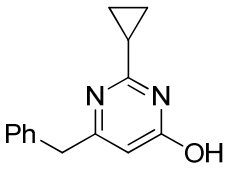



```
Current Data Parameters
NAME      h_bar.HBHC13-h
EXPNO                  1
PROCNO                 1

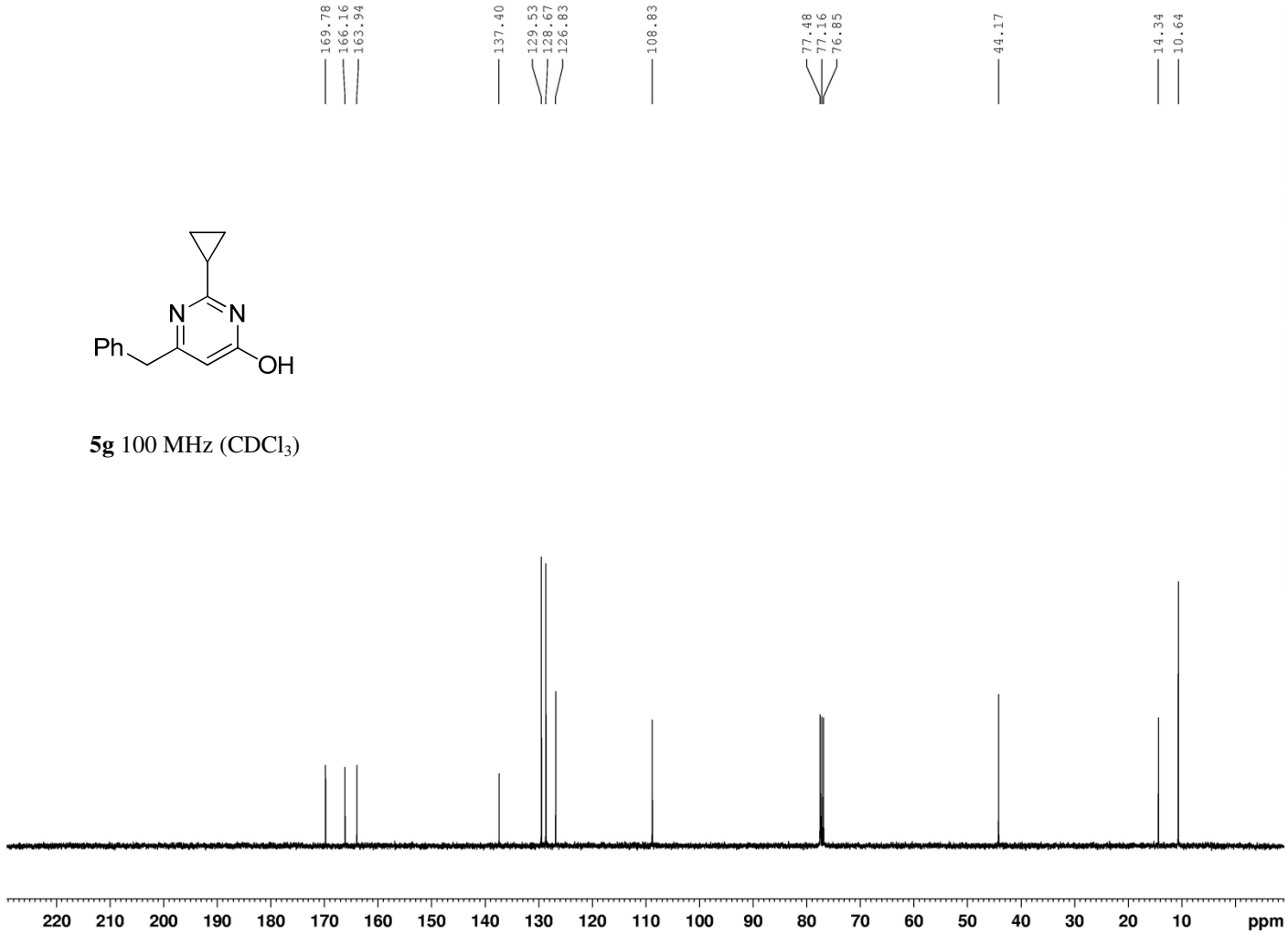
F2 - Acquisition Parameters
Date_           20100522
Time              11.48
INSTRUM           av3400
PROBHD   5 mm PABBI 1H/
PULPROG             zg30
TD                65536
SOLVENT           CDCl3
NS                   16
DS                    2
SWH            8223.685 Hz
FIDRES        0.125483 Hz
AQ            3.9846387 sec
RG                 45.2
DW               60.800 usec
DE                6.50 usec
TE               298.0 K
D1           1.00000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                  1H
P1                 7.30 usec
PL1               -0.90 dB
PL1W        11.52680206 W
SFO1       400.0724706 MHz

F2 - Processing parameters
SI                 65536
SF          400.0700122 MHz
WDW                   EM
SSB                    0
LB                0.30 Hz
GB                     0
PC                1.00
```



5g 400 MHz ($CDCl_3$)



5g 100 MHz ($CDCl_3$)

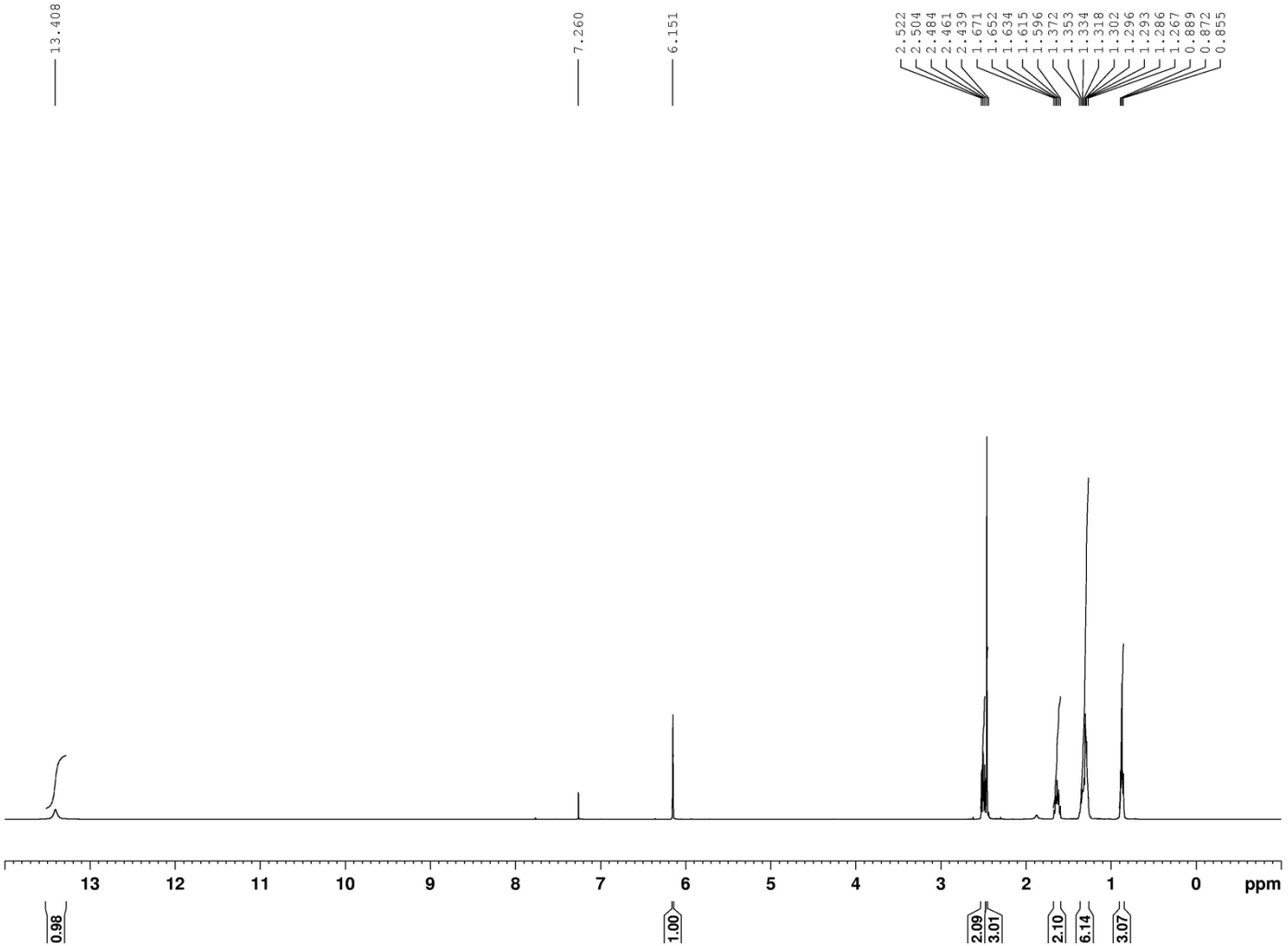
```
Current Data Parameters
NAME        h_bar.HBHC13
EXPNO                  1
PROCNO                 1

F2 - Acquisition Parameters
Date_           20100522
Time               12.39
INSTRUM            av400
PROBHD   5 mm PABBO BB-
PULPROG          zgpg30
TD                32768
SOLVENT           CDC13
NS                  128
DS                    4
SWH           23980.814 Hz
FIDRES         0.731836 Hz
AQ            0.6832628 sec
RG              18390.4
DW               20.850 usec
DE               10.00 usec
TE               298.2 K
D1           1.00000000 sec
D11          0.03000000 sec
TD0                   1

======== CHANNEL f1 ========
NUC1                 13C
P1                  7.50 usec
PL1                -3.00 dB
PL1W        73.67452240 W
SFO1       100.6238350 MHz

======== CHANNEL f2 ========
CPDPRG2          waltz16
NUC2                 1H
PCPD2             100.00 usec
PL2                -2.00 dB
PL12               17.00 dB
PL13               19.30 dB
PL2W        16.00390816 W
PL12W        0.20147727 W
PL13W        0.11863863 W
SFO2       400.1316005 MHz

F2 - Processing parameters
SI                32768
SF         100.6127594 MHz
WDW                  EM
SSB                   0
LB                 1.00 Hz
GB                    0
PC                 1.40
```

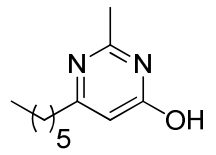



```
Current Data Parameters
NAME       h_bar.HBHC7-h
EXPNO                  1
PROCNO                 1

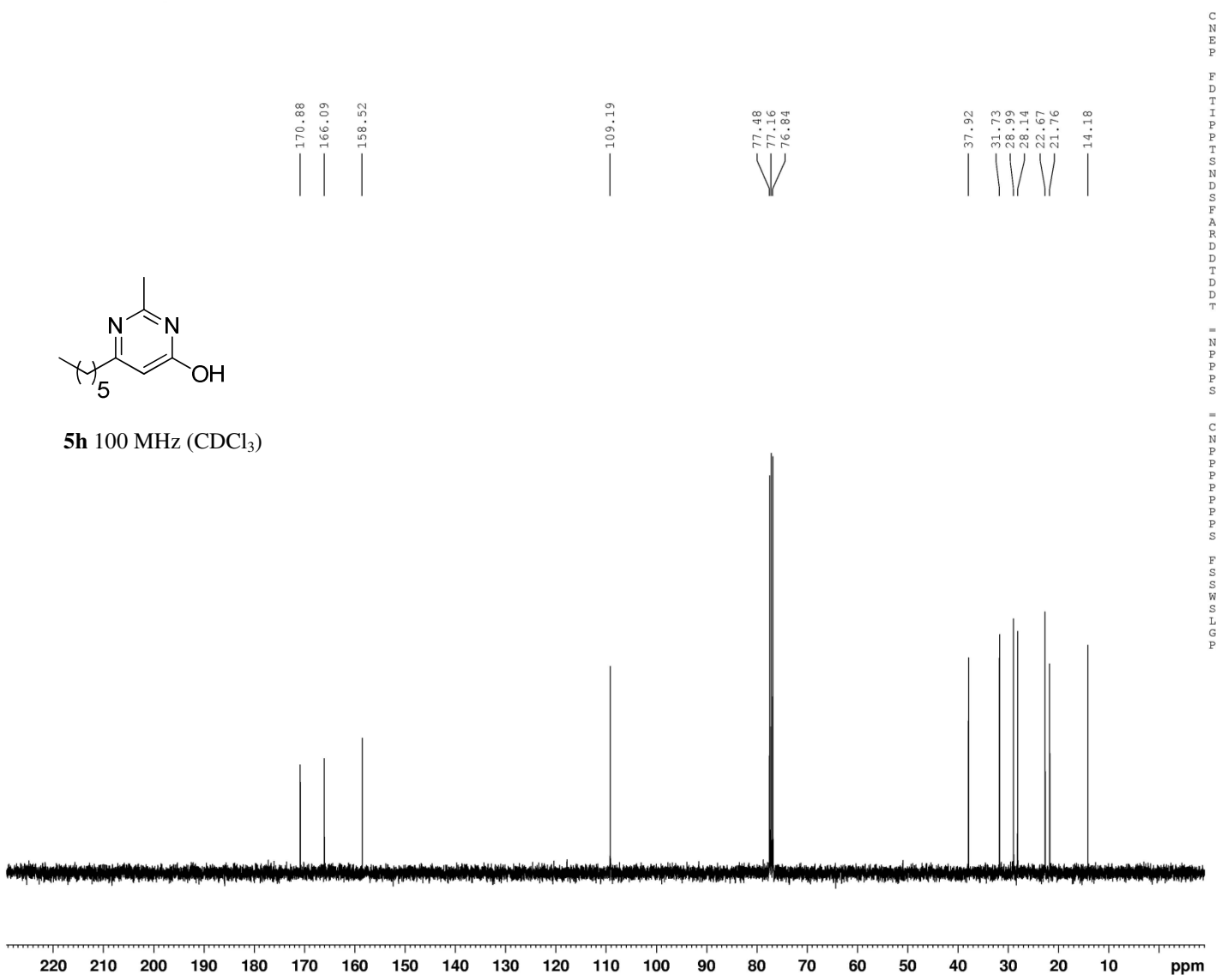
F2 - Acquisition Parameters
Date_           20100521
Time              16.34
INSTRUM           av3400
PROBHD   5 mm PABBI 1H/
PULPROG            zg30
TD                65536
SOLVENT           CDCl3
NS                   16
DS                    2
SWH            8223.685 Hz
FIDRES        0.125483 Hz
AQ            3.9846387 sec
RG                  101
DW               60.800 usec
DE                6.50 usec
TE                298.0 K
D1           1.00000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                  1H
P1                 7.30 usec
PL1                -0.90 dB
PL1W        11.52680206 W
SFO1        400.0724706 MHz

F2 - Processing parameters
SI                 65536
SF          400.0700122 MHz
WDW                   EM
SSB                    0
LB                  0.30 Hz
GB                     0
PC                  1.00
```



5h 400 MHz ($CDCl_3$)



5h 100 MHz ($CDCl_3$)

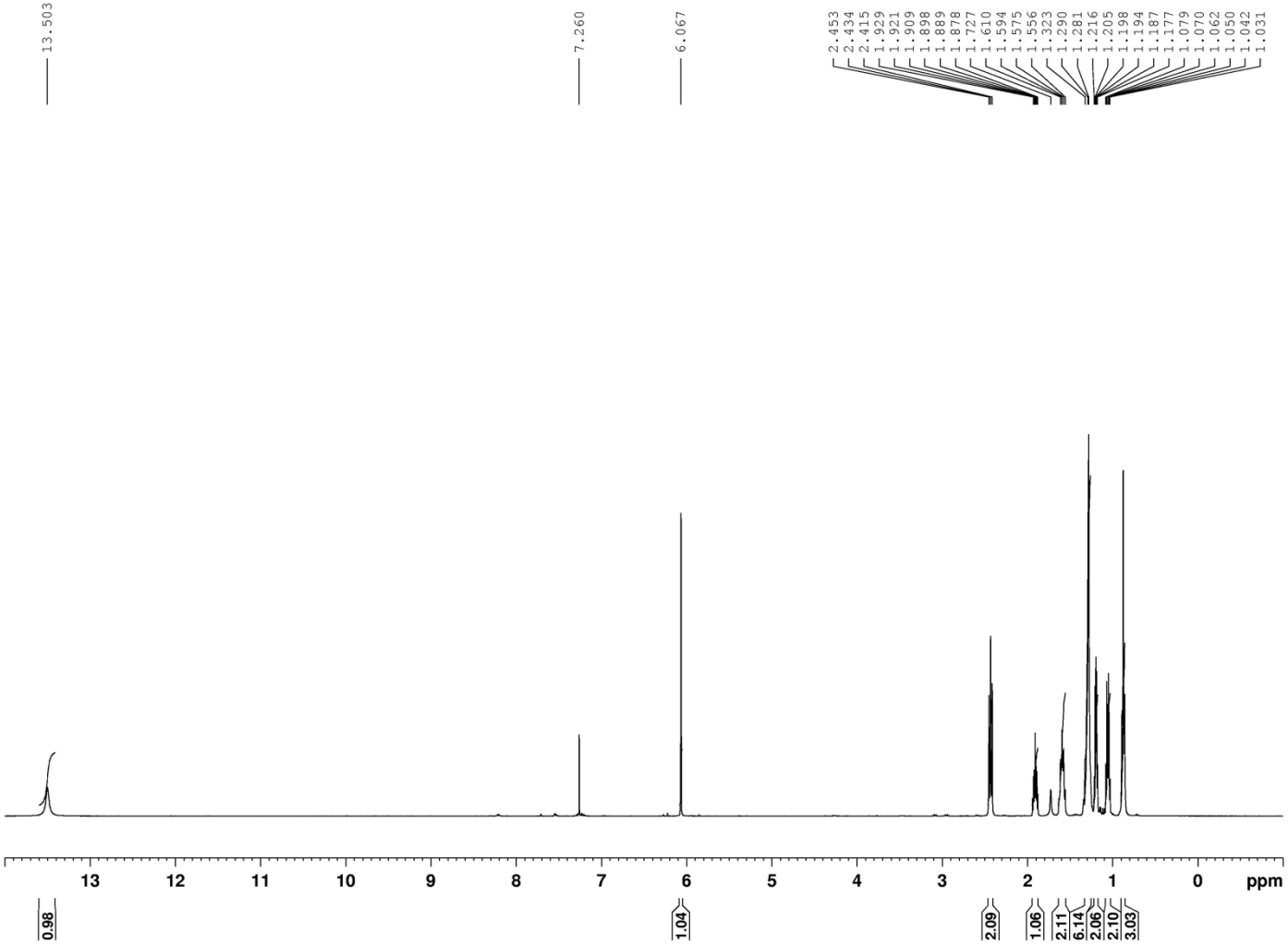
```
Current Data Parameters
NAME          h_bar.HBHC7
EXPNO                   1
PROCNO                  1

F2 - Acquisition Parameters
Date_            20100521
Time                17.17
INSTRUM             av400
PROBHD   5 mm PABBO BB-
PULPROG           zgpg30
TD                 32768
SOLVENT             CDCl3
NS                    128
DS                      4
SWH             23980.814 Hz
FIDRES          0.731836 Hz
AQ              0.6832628 sec
RG                20642.5
DW                20.850 usec
DE                10.00 usec
TE                298.2 K
D1           1.00000000 sec
D11          0.03000000 sec
TD0                     1

======== CHANNEL f1 ========
NUC1                  13C
P1                 7.50 usec
PL1               -3.00 dB
PL1W         73.67452240 W
SFO1        100.6238350 MHz

======== CHANNEL f2 ========
CPDPRG2           waltz16
NUC2                  1H
PCPD2            100.00 usec
PL2               -2.00 dB
PL12              17.00 dB
PL13              19.30 dB
PL2W         16.00390816 W
PL12W         0.20147727 W
PL13W         0.11863863 W
SFO2        400.1316005 MHz

F2 - Processing parameters
SI                 32768
SF            100.6127557 MHz
WDW                   EM
SSB                    0
LB                  1.00 Hz
GB                     0
PC                  1.40
```

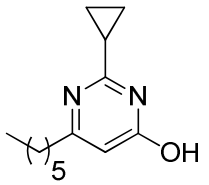



```
Current Data Parameters
NAME         h_bar.HBHC8
EXPNO                  1
PROCNO                 1

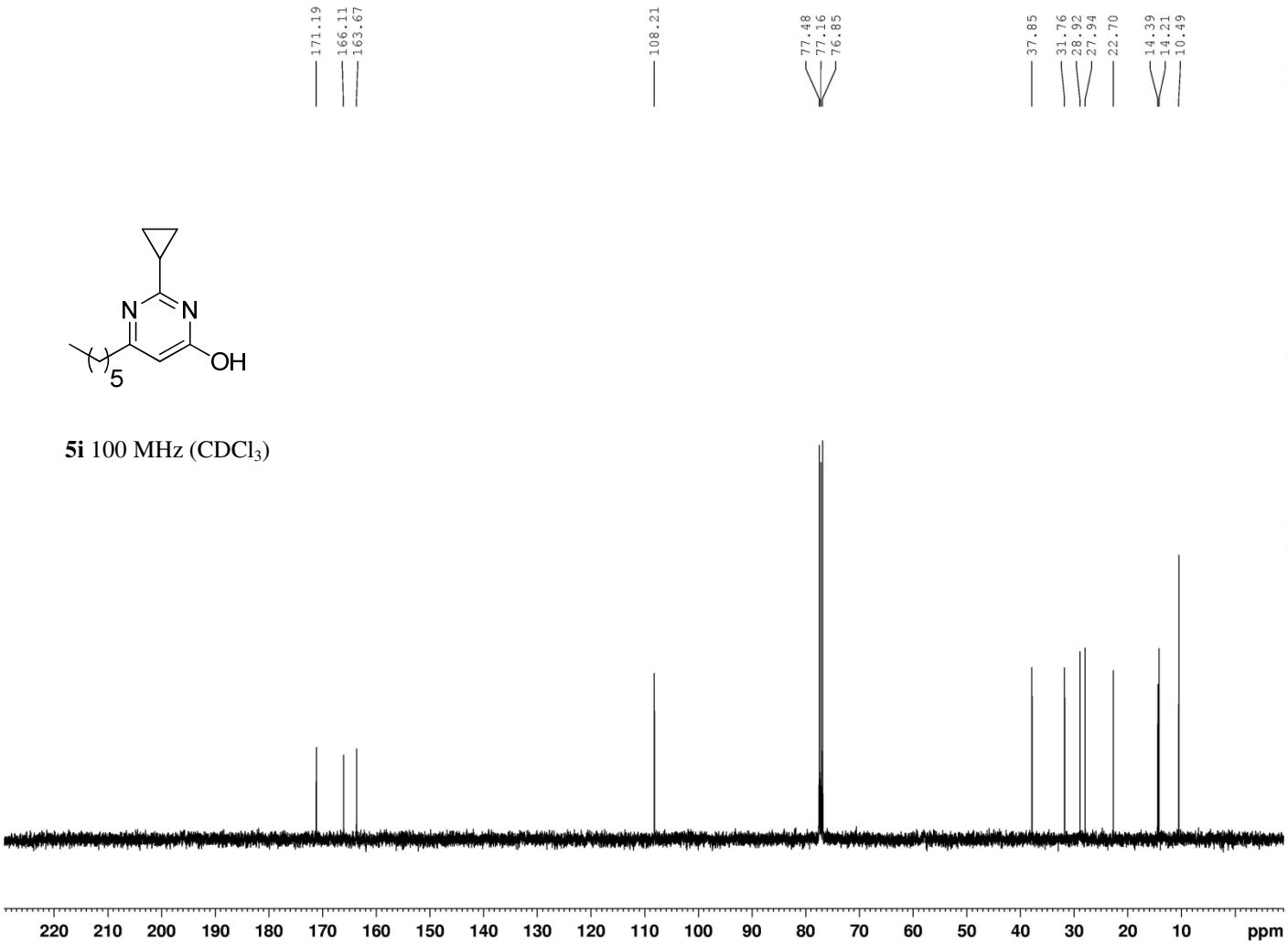
F2 - Acquisition Parameters
Date_           20100521
Time               13.43
INSTRUM            av3400
PROBHD   5 mm PABBI 1H/
PULPROG             zg30
TD                 65536
SOLVENT            CDCl3
NS                    16
DS                     2
SWH             8223.685 Hz
FIDRES          0.125483 Hz
AQ              3.9846387 sec
RG                   114
DW                60.800 usec
DE                 6.50 usec
TE                298.0 K
D1             1.00000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                  1H
P1                  7.30 usec
PL1                -0.90 dB
PL1W          11.52680206 W
SFO1          400.0724706 MHz

F2 - Processing parameters
SI                 65536
SF            400.0700122 MHz
WDW                   EM
SSB                    0
LB                  0.30 Hz
GB                     0
PC                  1.00
```



5i 400 MHz ($CDCl_3$)



5i 100 MHz ($CDCl_3$)

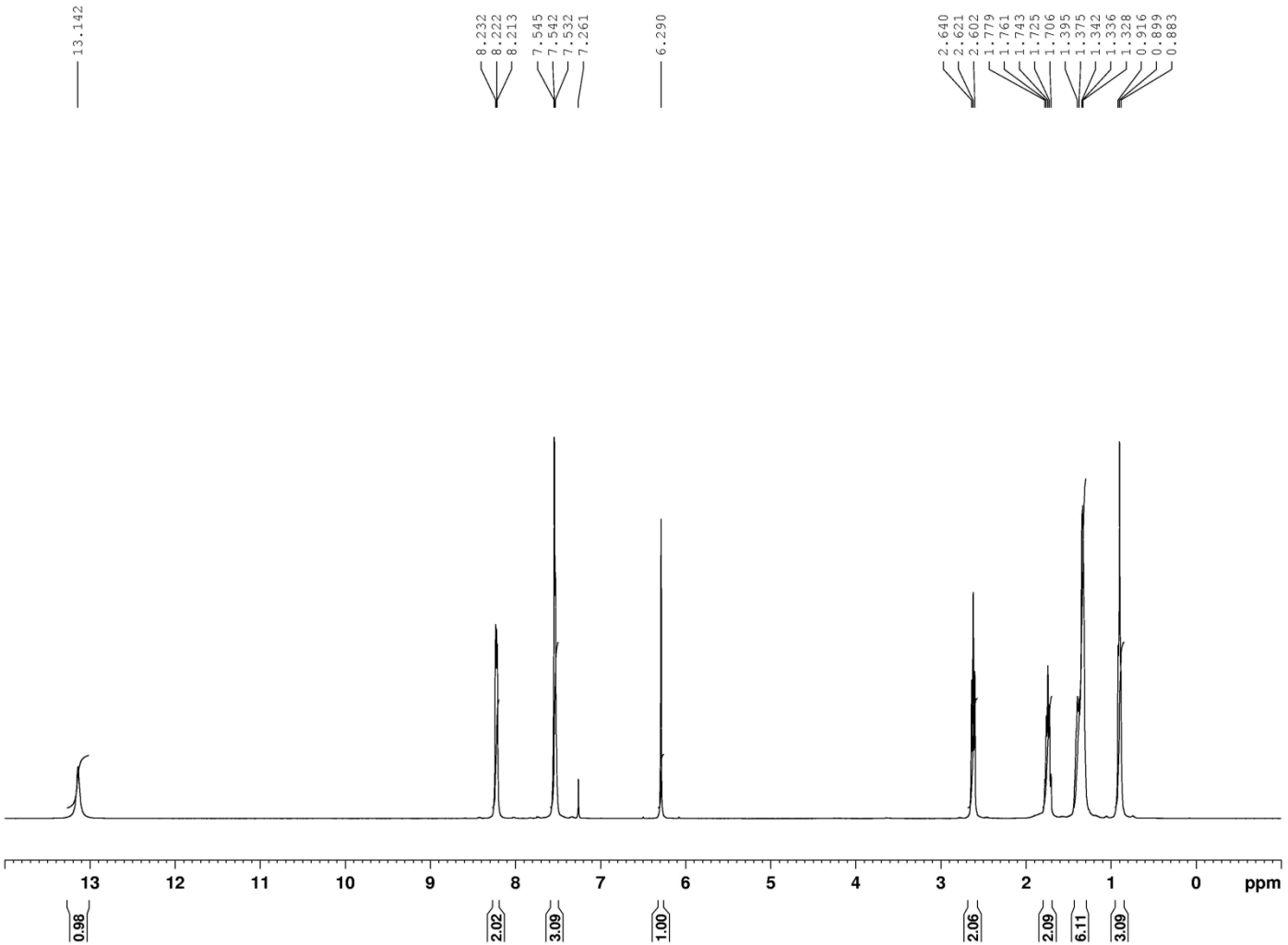
```
Current Data Parameters
NAME       h_bar.HBHC8-c
EXPNO                  1
PROCNO                 1

F2 - Acquisition Parameters
Date_           20100521
Time               14.48
INSTRUM            av400
PROBHD   5 mm PABBO BB-
PULPROG          zgpg30
TD                32768
SOLVENT           CDCl3
NS                  128
DS                    4
SWH           23980.814 Hz
FIDRES         0.731836 Hz
AQ            0.6832628 sec
RG              20642.5
DW               20.850 usec
DE               10.00 usec
TE               298.2 K
D1           1.00000000 sec
D11          0.03000000 sec
TD0                   1

======== CHANNEL f1 ========
NUC1                 13C
P1                  7.50 usec
PL1                -3.00 dB
PL1W        73.67452240 W
SFO1       100.6238350 MHz

======== CHANNEL f2 ========
CPDPRG2          waltz16
NUC2                 1H
PCPD2             100.00 usec
PL2                -2.00 dB
PL12               17.00 dB
PL13               19.30 dB
PL2W        16.00390816 W
PL12W        0.20147727 W
PL13W        0.11863863 W
SFO2       400.1316005 MHz

F2 - Processing parameters
SI                32768
SF           100.6127550 MHz
WDW                  EM
SSB                   0
LB                 1.00 Hz
GB                    0
PC                 1.40
```

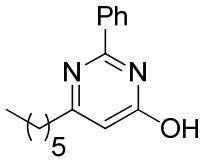



```
Current Data Parameters
NAME         h_bar.HBHC9
EXPNO                  1
PROCNO                 1

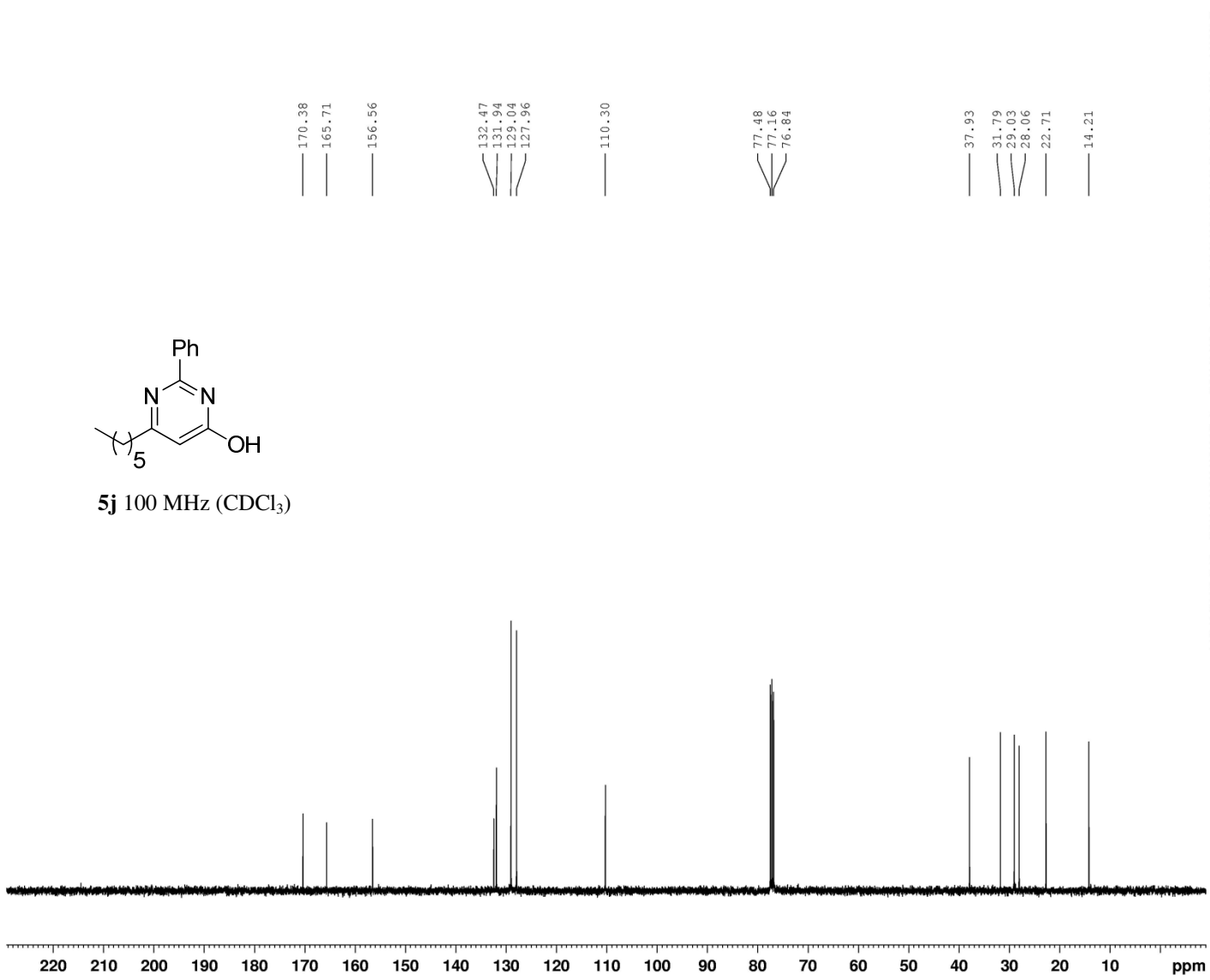
F2 - Acquisition Parameters
Date_           20100521
Time               13.49
INSTRUM          av3400
PROBHD   5 mm PABBI 1H/
PULPROG            zg30
TD                65536
SOLVENT           CDCl3
NS                   16
DS                    2
SWH            8223.685 Hz
FIDRES         0.125483 Hz
AQ             3.9846387 sec
RG                 50.8
DW               60.800 usec
DE                6.50 usec
TE               298.0 K
D1            1.00000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                  1H
P1                 7.30 usec
PL1                -0.90 dB
PL1W         11.52680206 W
SFO1         400.0724706 MHz

F2 - Processing parameters
SI                 65536
SF           400.0700121 MHz
WDW                   EM
SSB                    0
LB                 0.30 Hz
GB                     0
PC                 1.00
```



5j 400 MHz ($CDCl_3$)



5j 100 MHz ($CDCl_3$)

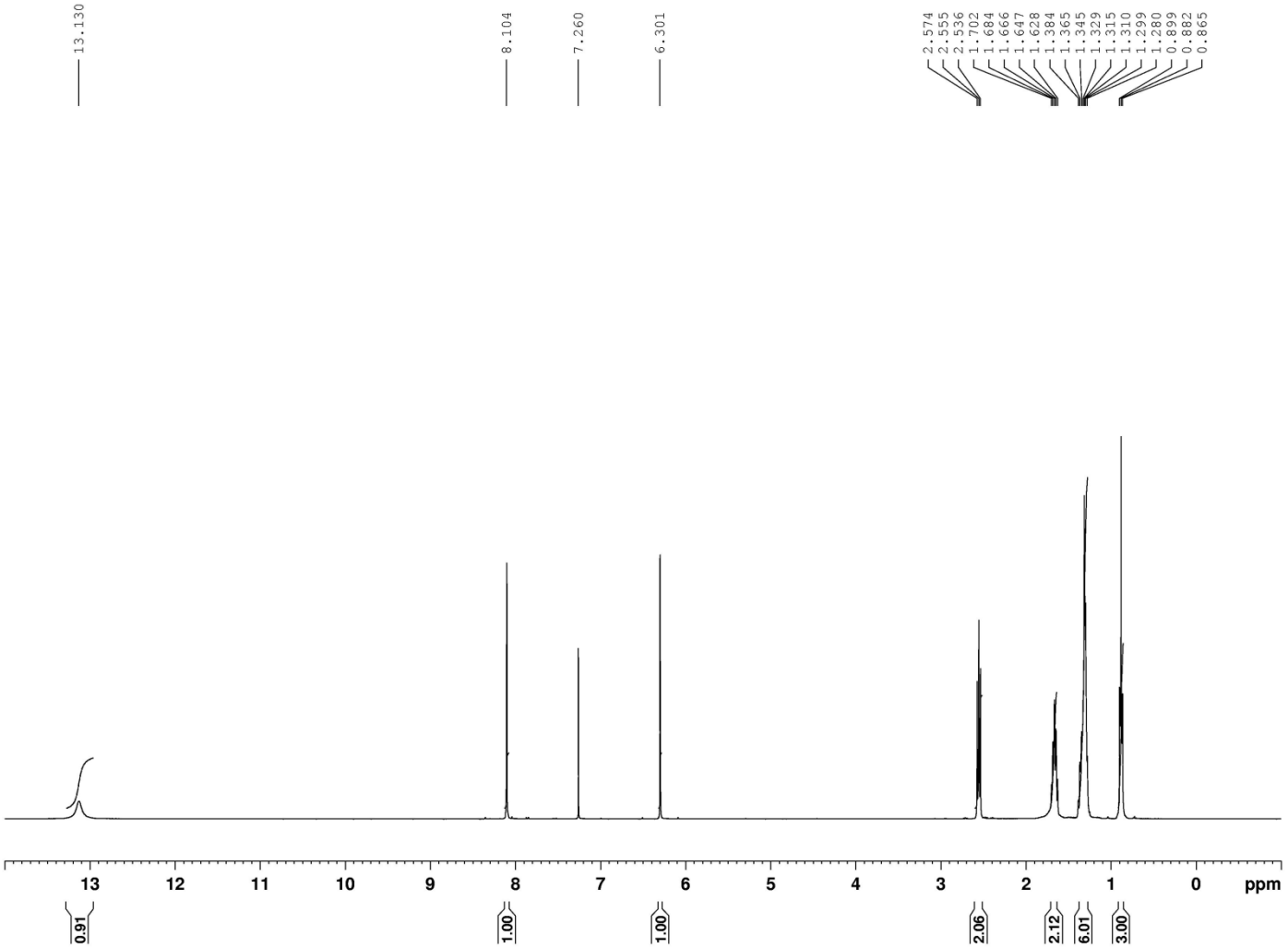
```
Current Data Parameters
NAME       h_bar.HBHC9-c
EXPNO                  1
PROCNO                 1

F2 - Acquisition Parameters
Date_           20100521
Time               14.09
INSTRUM            av400
PROBHD   5 mm PABBO BB-
PULPROG          zgpg30
TD                32768
SOLVENT           CDCl3
NS                  128
DS                    4
SWH           23980.814 Hz
FIDRES         0.731836 Hz
AQ             0.6832628 sec
RG                18390.4
DW                20.850 usec
DE                10.00 usec
TE                298.2 K
D1           1.00000000 sec
D11          0.03000000 sec
TD0                   1

======== CHANNEL f1 ========
NUC1                 13C
P1                  7.50 usec
PL1                -3.00 dB
PL1W        73.67452240 W
SFO1       100.6238350 MHz

======== CHANNEL f2 ========
CPDPRG2          waltz16
NUC2                 1H
PCPD2             100.00 usec
PL2                -2.00 dB
PL12               17.00 dB
PL13               19.30 dB
PL2W        16.00390816 W
PL12W        0.20147727 W
PL13W        0.11863863 W
SFO2       400.1316005 MHz

F2 - Processing parameters
SI                32768
SF           100.6127572 MHz
WDW                  EM
SSB                   0
LB                 1.00 Hz
GB                    0
PC                 1.40
```

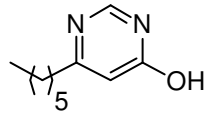



```
Current Data Parameters
NAME          h_bar.HBHC6
EXPNO                   1
PROCNO                  1

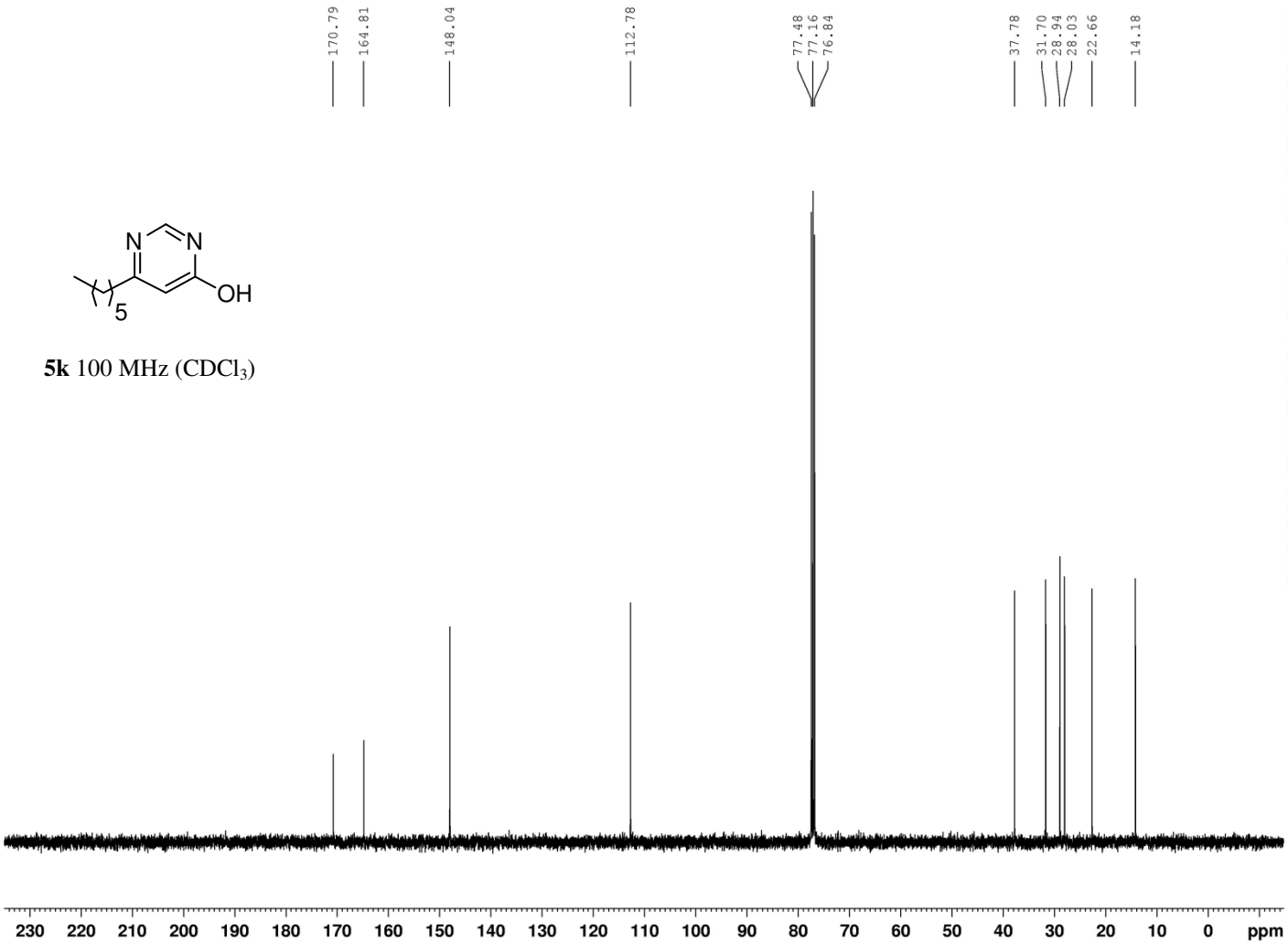
F2 - Acquisition Parameters
Date_            20100521
Time                16.28
INSTRUM           av3400
PROBHD   5 mm PABBI 1H/
PULPROG             zg30
TD                 65536
SOLVENT            CDCl3
NS                    16
DS                     2
SWH             8223.685 Hz
FIDRES          0.125483 Hz
AQ              3.9846387 sec
RG                   228
DW                60.800 usec
DE                 6.50 usec
TE                298.0 K
D1             1.00000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                  1H
P1                  7.30 usec
PL1                -0.90 dB
PL1W         11.52680206 W
SFO1         400.0724706 MHz

F2 - Processing parameters
SI                 65536
SF          400.0700122 MHz
WDW                   EM
SSB                    0
LB                  0.30 Hz
GB                     0
PC                  1.00
```



5k 400 MHz ($CDCl_3$)



5k 100 MHz ($CDCl_3$)

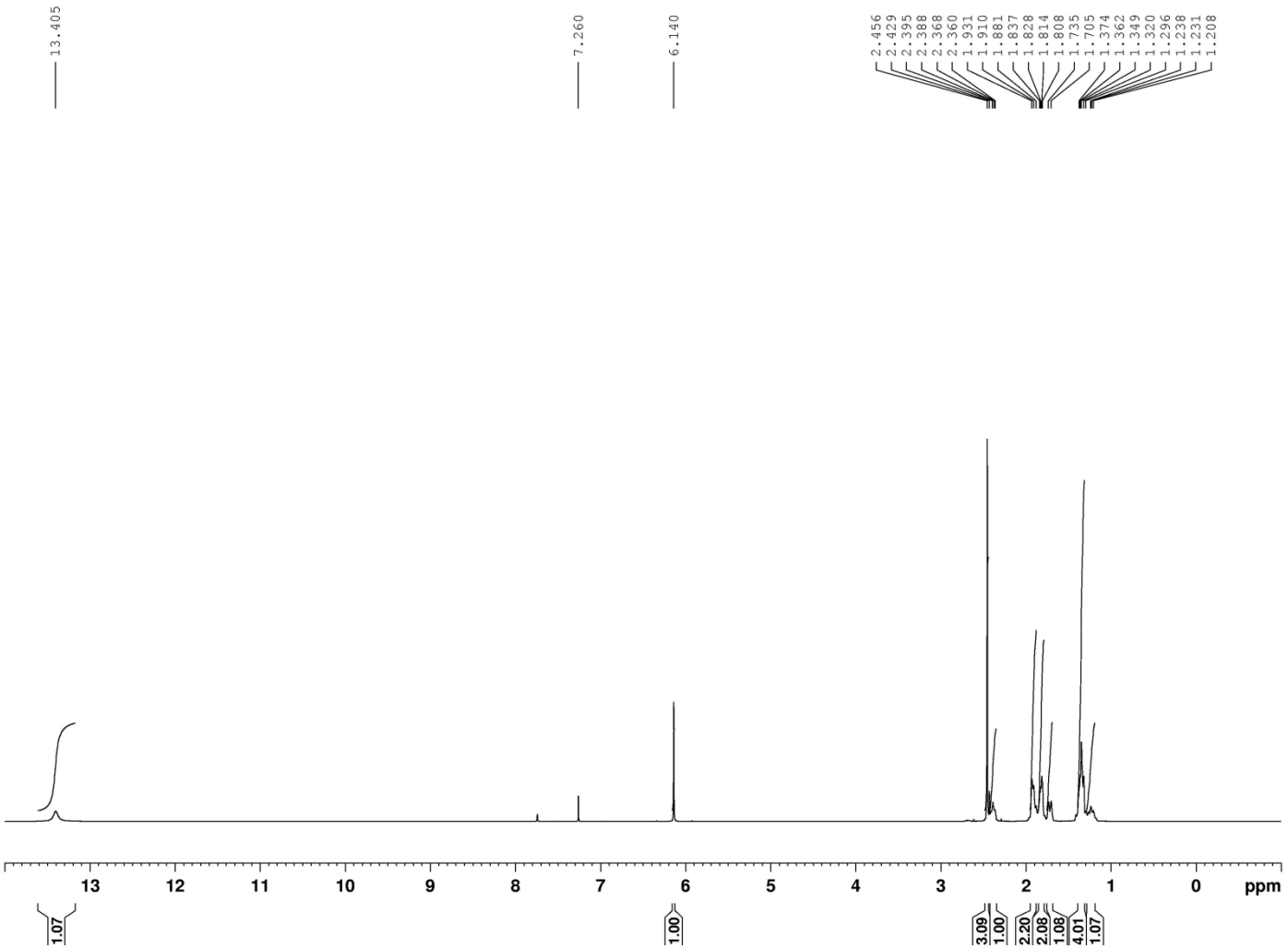
```
Current Data Parameters
NAME       h_bar.HBHC6-c
EXPNO                  1
PROCNO                 1

F2 - Acquisition Parameters
Date_           20100522
Time               11.38
INSTRUM            av400
PROBHD   5 mm PABBO BB-
PULPROG          zgpg30
TD                32768
SOLVENT           CDCl3
NS                  512
DS                    2
SWH           25125.629 Hz
FIDRES         0.766773 Hz
AQ             0.6521332 sec
RG               20642.5
DW                19.900 usec
DE                10.00 usec
TE                298.2 K
D1           1.00000000 sec
D11          0.03000000 sec
TD0                   1

======== CHANNEL f1 ========
NUC1                 13C
P1                  7.50 usec
PL1                -3.00 dB
PL1W        73.67452240 W
SFO1       100.6238350 MHz

======== CHANNEL f2 ========
CPDPRG2          waltz16
NUC2                 1H
PCPD2             100.00 usec
PL2                -2.00 dB
PL12               17.00 dB
PL13               19.30 dB
PL2W        16.00390816 W
PL12W        0.20147727 W
PL13W        0.11863863 W
SFO2       400.1316005 MHz

F2 - Processing parameters
SI                32768
SF           100.6127554 MHz
WDW                  EM
SSB                   0
LB                 1.00 Hz
GB                    0
PC                 1.40
```

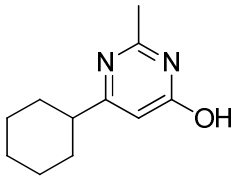



```
Current Data Parameters
NAME      h_bar.HBHC11-h
EXPNO                  1
PROCNO                 1

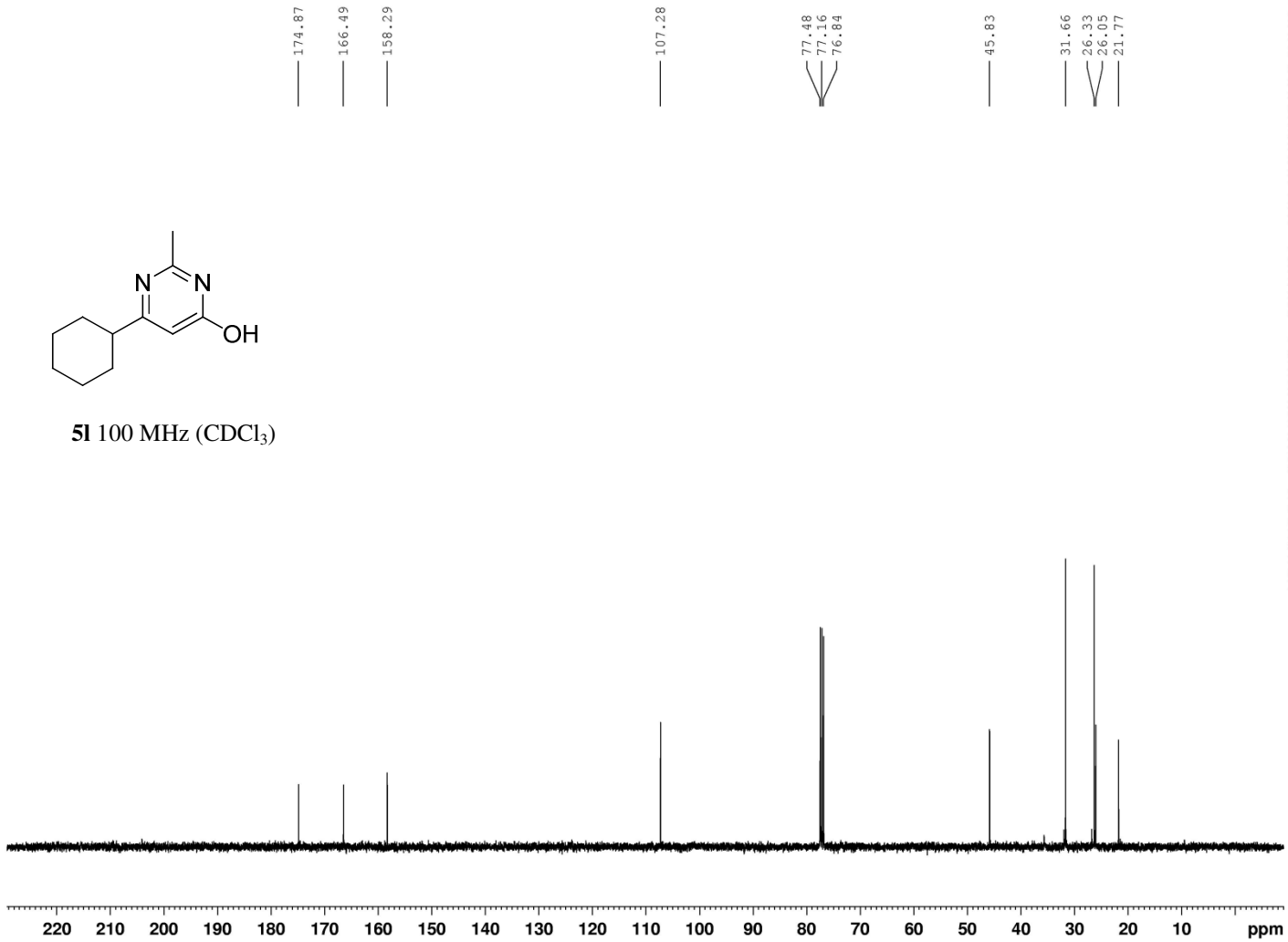
F2 - Acquisition Parameters
Date_           20100524
Time               13.47
INSTRUM          av3400
PROBHD   5 mm PABBI 1H/
PULPROG            zg30
TD                65536
SOLVENT           CDCl3
NS                   16
DS                    2
SWH            8223.685 Hz
FIDRES        0.125483 Hz
AQ            3.9846387 sec
RG                  101
DW               60.800 usec
DE                6.50 usec
TE                298.0 K
D1           1.00000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                  1H
P1                 7.30 usec
PL1                -0.90 dB
PL1W        11.52680206 W
SFO1        400.0724706 MHz

F2 - Processing parameters
SI                 65536
SF          400.0700123 MHz
WDW                   EM
SSB                    0
LB                  0.30 Hz
GB                     0
PC                  1.00
```



5l 400 MHz ($CDCl_3$)



5l 100 MHz ($CDCl_3$)

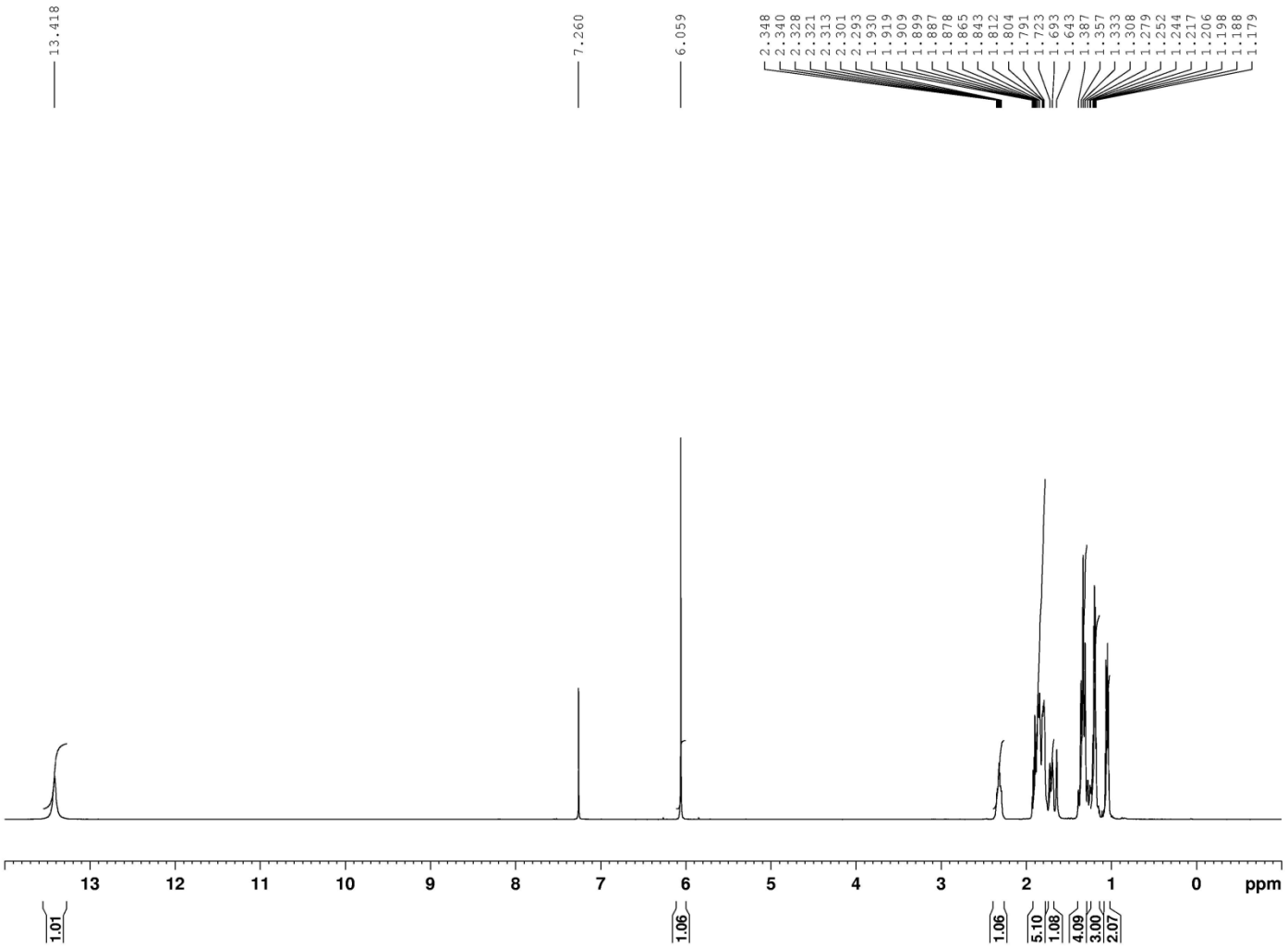
```
Current Data Parameters
NAME        h_bar.HBHC11
EXPNO                  1
PROCNO                 1

F2 - Acquisition Parameters
Date_           20100524
Time               15.32
INSTRUM            av400
PROBHD   5 mm PABBO BB-
PULPROG           zgpg30
TD                 32768
SOLVENT            CDCl3
NS                   128
DS                     4
SWH            23980.814 Hz
FIDRES         0.731836 Hz
AQ             0.6832628 sec
RG                 20642.5
DW                20.850 usec
DE                10.00 usec
TE                298.2 K
D1            1.00000000 sec
D11           0.03000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                  13C
P1                   7.50 usec
PL1                 -3.00 dB
PL1W         73.67452240 W
SFO1        100.6238350 MHz

======== CHANNEL f2 ========
CPDPRG2           waltz16
NUC2                   1H
PCPD2              100.00 usec
PL2                 -2.00 dB
PL12                17.00 dB
PL13                19.30 dB
PL2W         16.00390816 W
PL12W         0.20147727 W
PL13W         0.11863863 W
SFO2        400.1316005 MHz

F2 - Processing parameters
SI                 32768
SF            100.6127557 MHz
WDW                   EM
SSB                    0
LB                  1.00 Hz
GB                     0
PC                  1.40
```

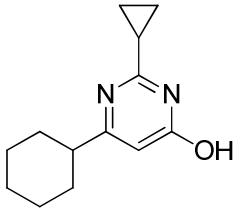



```
Current Data Parameters
NAME      h_bar.HBHC11-h
EXPNO                  2
PROCNO                 1

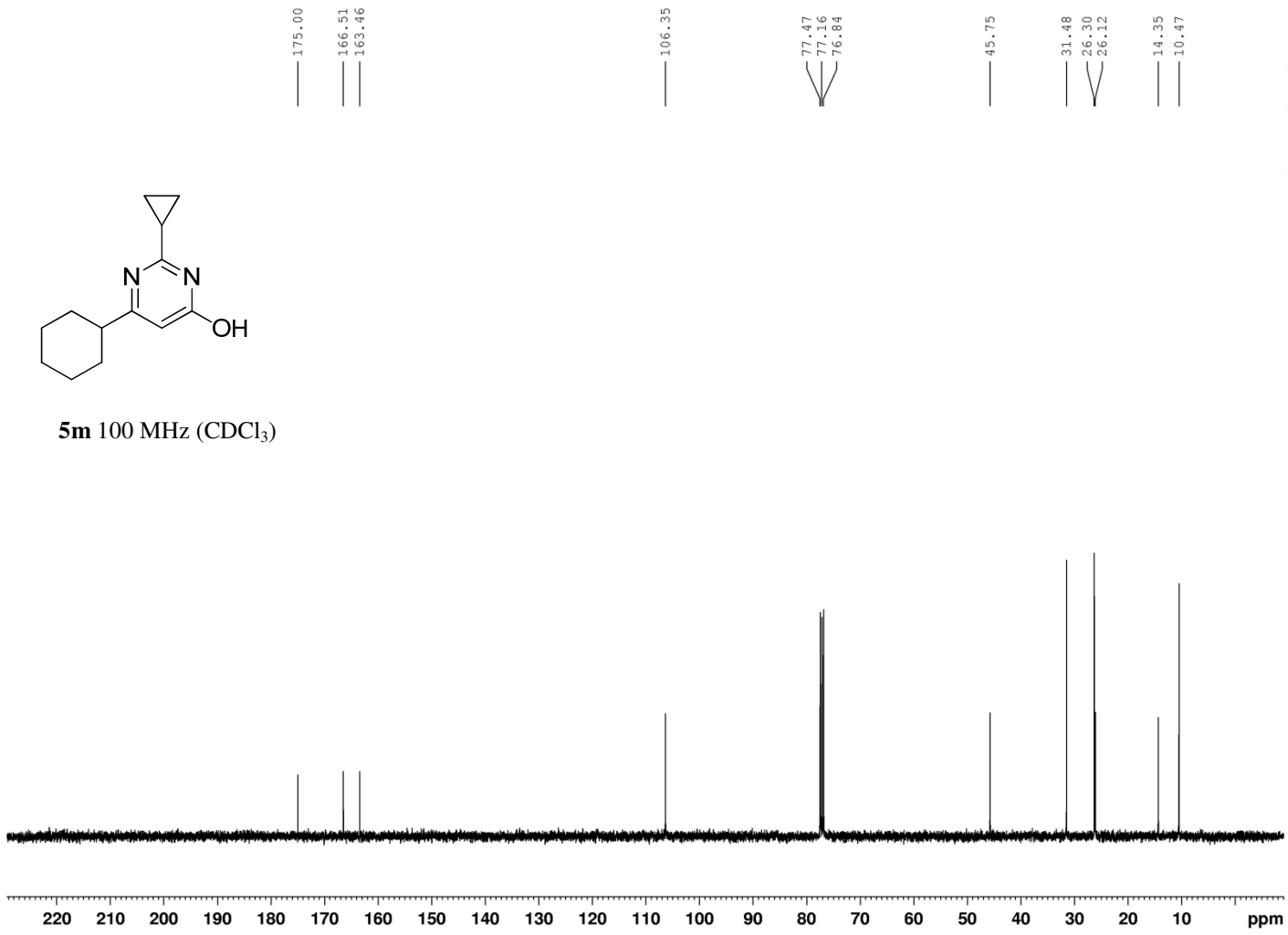
F2 - Acquisition Parameters
Date_           20100525
Time               10.44
INSTRUM          av3400
PROBHD   5 mm PABBI 1H/
PULPROG             zg30
TD                 65536
SOLVENT            CDCl3
NS                    16
DS                     2
SWH             8223.685 Hz
FIDRES          0.125483 Hz
AQ              3.9846387 sec
RG                   181
DW                60.800 usec
DE                 6.50 usec
TE                298.0 K
D1           1.00000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                  1H
P1                  7.30 usec
PL1                -0.90 dB
PL1W         11.52680206 W
SFO1         400.0724706 MHz

F2 - Processing parameters
SI                 65536
SF          400.0700122 MHz
WDW                   EM
SSB                    0
LB                  0.30 Hz
GB                     0
PC                  1.00
```



5m 400 MHz ($CDCl_3$)



5m 100 MHz ($CDCl_3$)

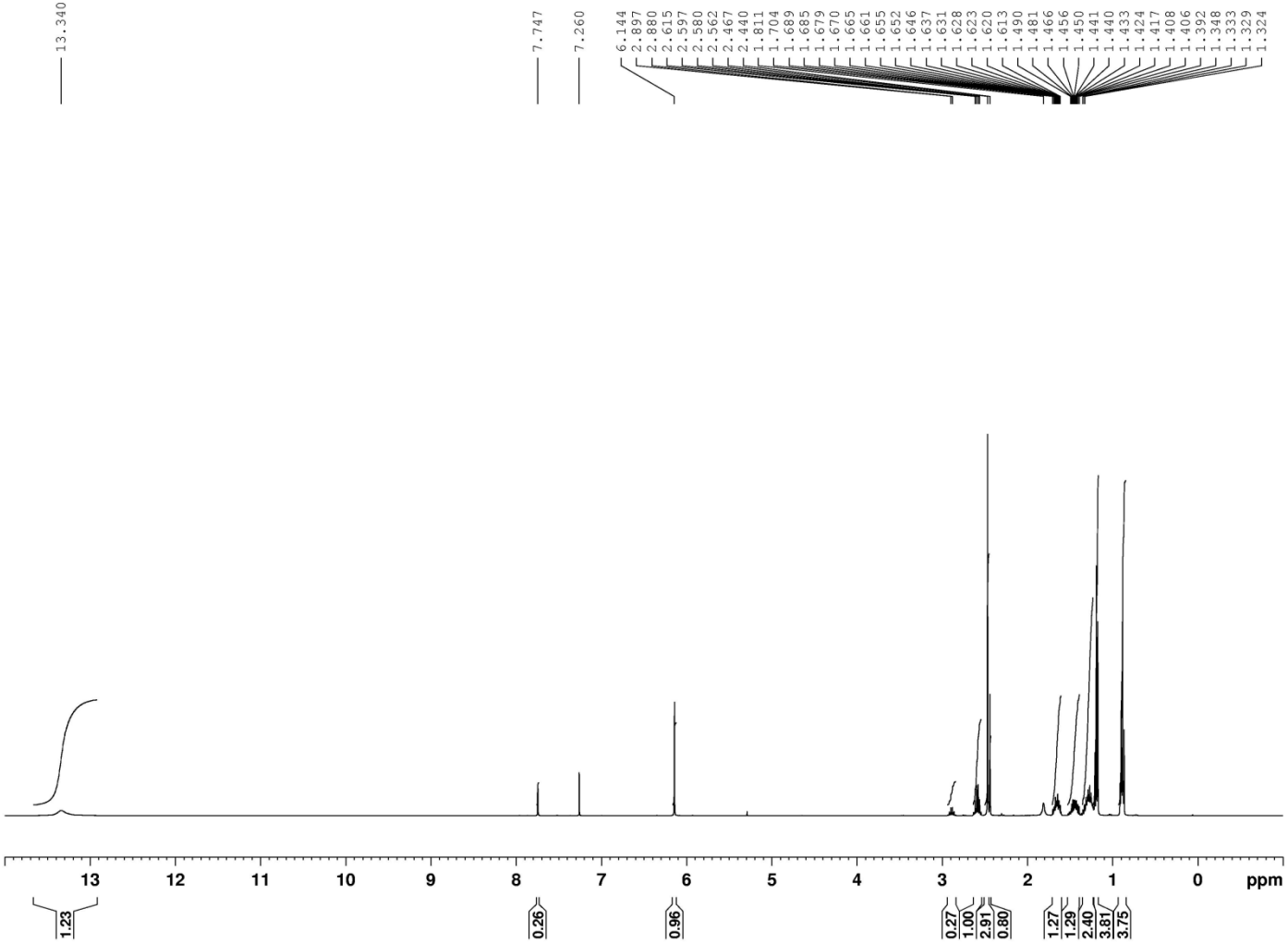
```
Current Data Parameters
NAME        h_bar.HBHC11
EXPNO                  4
PROCNO                 1

F2 - Acquisition Parameters
Date_           20100525
Time               12.06
INSTRUM            av400
PROBHD   5 mm PABBO BB-
PULPROG          zgpg30
TD                32768
SOLVENT           CDCl3
NS                  128
DS                    4
SWH           23980.814 Hz
FIDRES         0.731836 Hz
AQ            0.6832628 sec
RG              18390.4
DW               20.850 usec
DE               10.00 usec
TE               298.2 K
D1           1.00000000 sec
D11          0.03000000 sec
TD0                   1

======== CHANNEL f1 ========
NUC1                 13C
P1                  7.50 usec
PL1                -3.00 dB
PL1W        73.67452240 W
SFO1       100.6238350 MHz

======== CHANNEL f2 ========
CPDPRG2          waltz16
NUC2                 1H
PCPD2            100.00 usec
PL2               -2.00 dB
PL12              17.00 dB
PL13              19.30 dB
PL2W        16.00390816 W
PL12W        0.20147727 W
PL13W        0.11863863 W
SFO2       400.1316005 MHz

F2 - Processing parameters
SI                32768
SF          100.6127565 MHz
WDW                  EM
SSB                   0
LB                 1.00 Hz
GB                    0
PC                 1.40
```

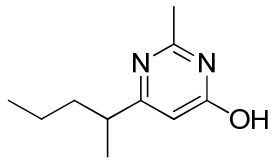



```
Current Data Parameters
NAME      h_bar.HBHC14-3
EXPNO                  3
PROCNO                 1

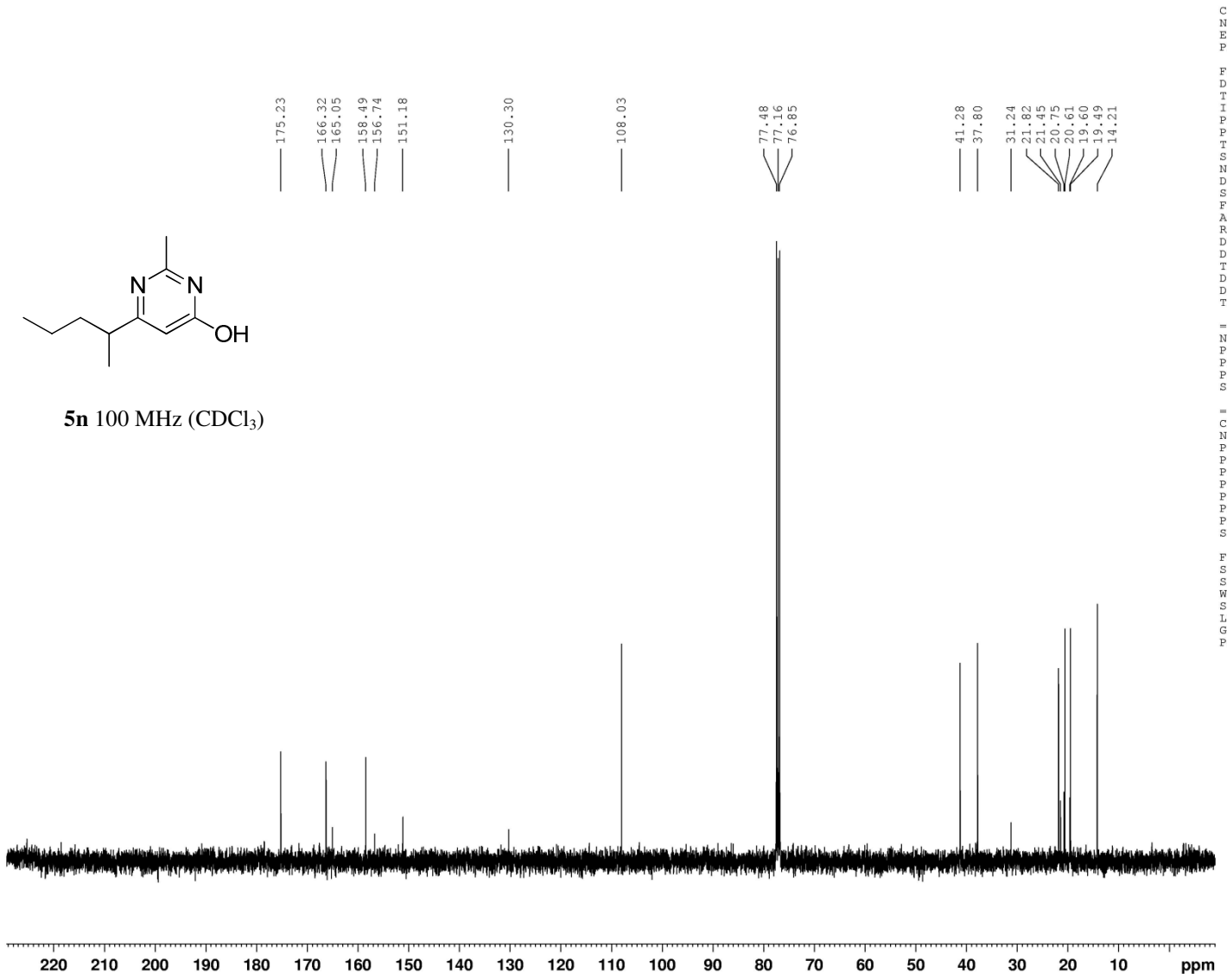
F2 - Acquisition Parameters
Date_           20100630
Time               15.28
INSTRUM            av3400
PROBHD   5 mm PABBI 1H/
PULPROG             zg30
TD                 65536
SOLVENT            CDCl3
NS                    16
DS                     2
SWH             8223.685 Hz
FIDRES          0.125483 Hz
AQ              3.9846387 sec
RG                   128
DW                60.800 usec
DE                 6.50 usec
TE                298.0 K
D1           1.00000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                  1H
P1                  7.30 usec
PL1                -0.90 dB
PL1W         11.52680206 W
SFO1         400.0724706 MHz

F2 - Processing parameters
SI                 65536
SF          400.0700122 MHz
WDW                   EM
SSB                    0
LB                  0.30 Hz
GB                     0
PC                  1.00
```



5n 400 MHz ($CDCl_3$)



5n 100 MHz ($CDCl_3$)

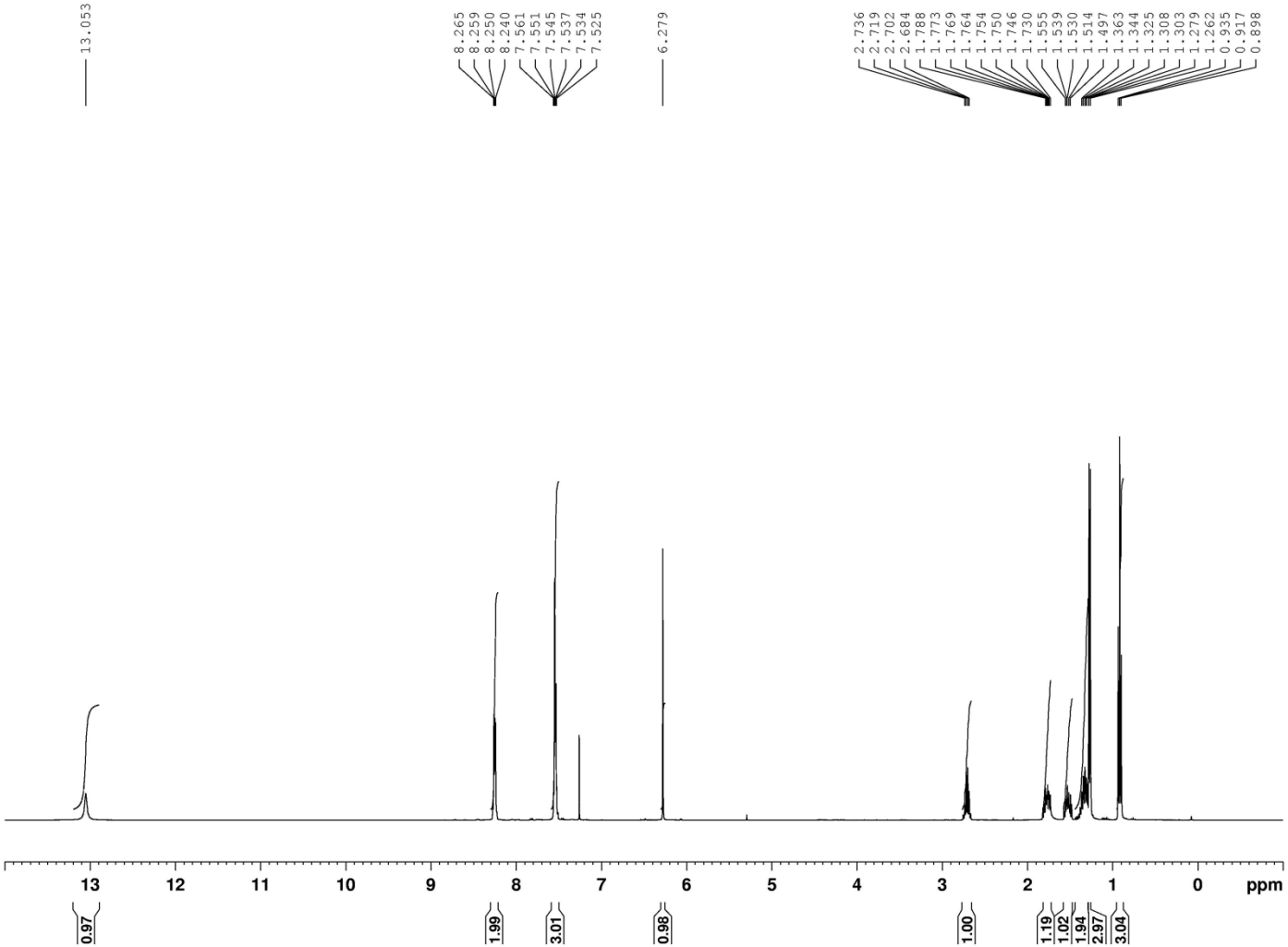
```
Current Data Parameters
NAME        h_bar.HBHC14
EXPNO                  4
PROCNO                 1

F2 - Acquisition Parameters
Date_           20100630
Time               15.37
INSTRUM            av400
PROBHD   5 mm PABBO BB-
PULPROG          zgpg30
TD                32768
SOLVENT             CDC13
NS                   128
DS                     4
SWH            23980.814 Hz
FIDRES          0.731836 Hz
AQ              0.6832628 sec
RG                18390.4
DW                 20.850 usec
DE                 10.00 usec
TE                 298.2 K
D1           1.00000000 sec
D11          0.03000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                 13C
P1                  7.50 usec
PL1                -3.00 dB
PL1W         73.67452240 W
SFO1        100.6238350 MHz

======== CHANNEL f2 ========
CPDPRG2          waltz16
NUC2                1H
PCPD2             100.00 usec
PL2                -2.00 dB
PL12               17.00 dB
PL13               19.30 dB
PL2W         16.00390816 W
PL12W         0.20147727 W
PL13W         0.11863863 W
SFO2        400.1316005 MHz

F2 - Processing parameters
SI                 32768
SF          100.6127550 MHz
WDW                   EM
SSB                    0
LB                  1.00 Hz
GB                     0
PC                  1.40
```

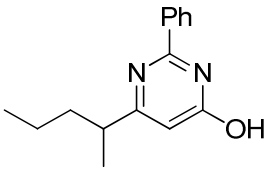



```
Current Data Parameters
NAME        h_bar.HBHC15
EXPNO                  2
PROCNO                 1

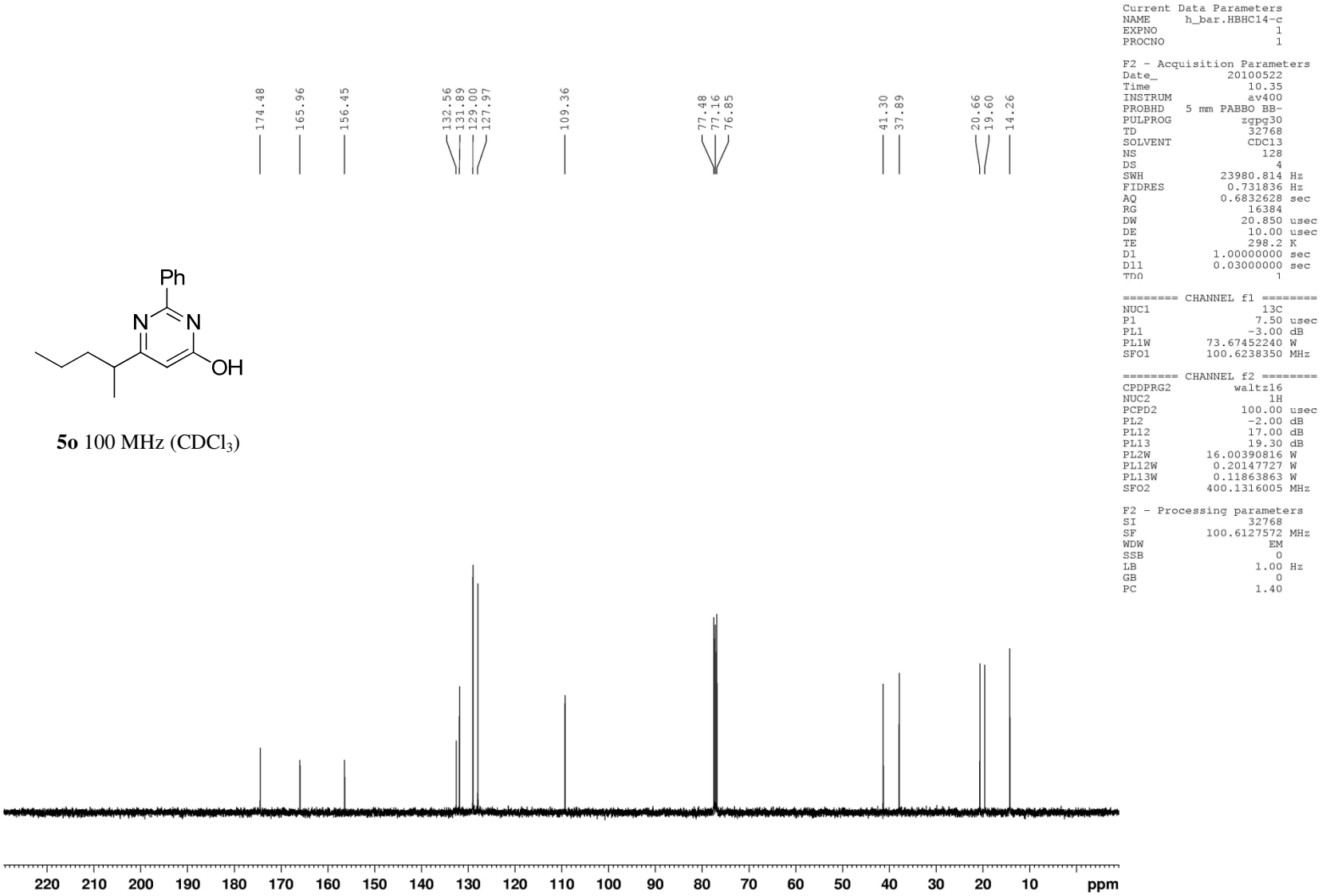
F2 - Acquisition Parameters
Date_           20100629
Time              16.25
INSTRUM           av3400
PROBHD   5 mm PABBI 1H/
PULPROG             zg30
TD                65536
SOLVENT           CDCl3
NS                   16
DS                    2
SWH            8223.685 Hz
FIDRES         0.125483 Hz
AQ             3.9846387 sec
RG                  114
DW               60.800 usec
DE                6.50 usec
TE                298.0 K
D1           1.00000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                  1H
P1                 7.30 usec
PL1                -0.90 dB
PL1W        11.52680206 W
SFO1        400.0724706 MHz

F2 - Processing parameters
SI                 65536
SF          400.0700122 MHz
WDW                   EM
SSB                    0
LB                0.30 Hz
GB                     0
PC                1.00
```



5o 400 MHz ($CDCl_3$)



5o 100 MHz ($CDCl_3$)

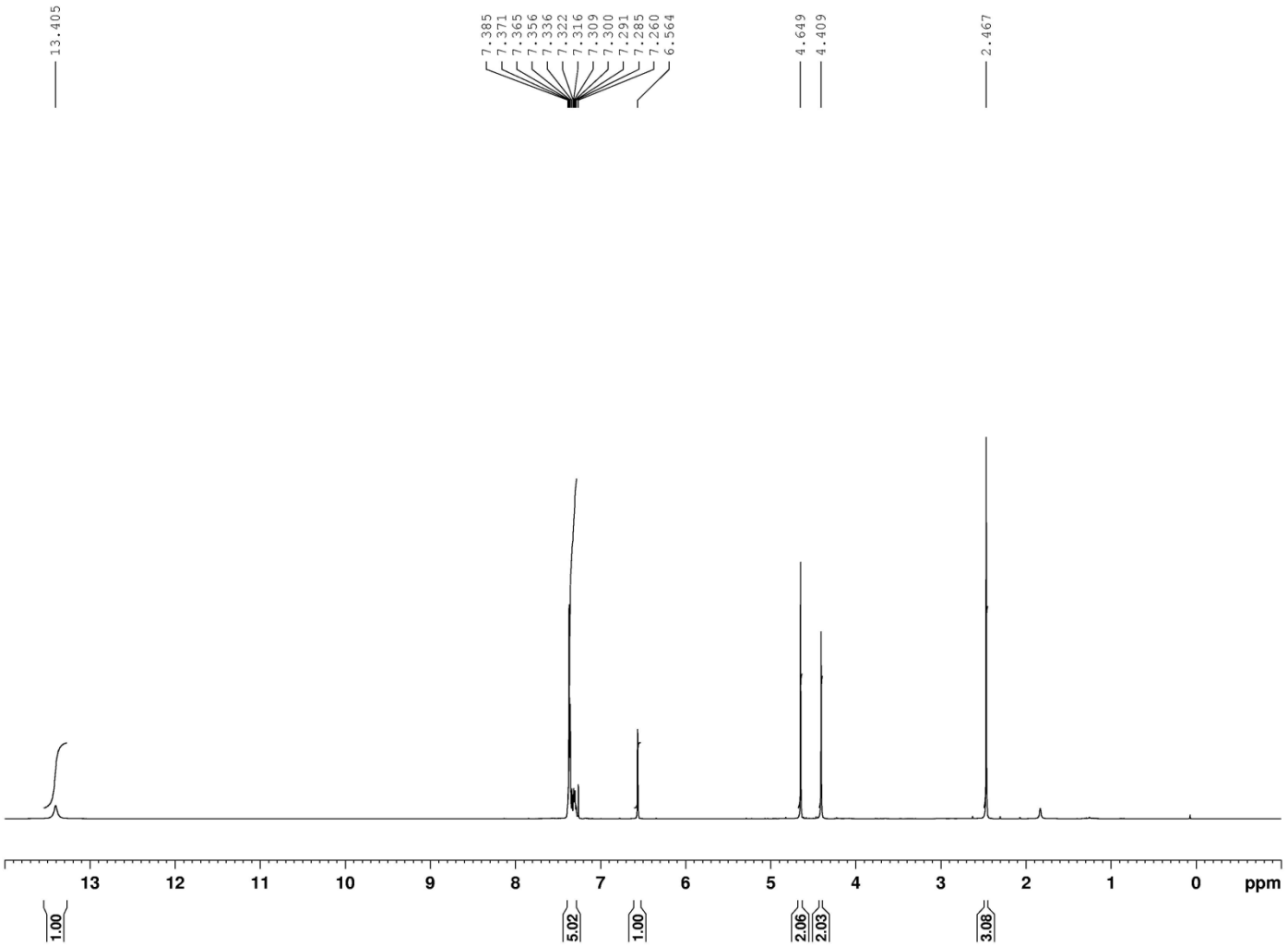
```
Current Data Parameters
NAME      h_bar.HBHC14-c
EXPNO                  1
PROCNO                 1

F2 - Acquisition Parameters
Date_           20100522
Time               10.35
INSTRUM            av400
PROBHD   5 mm PABBO BB-
PULPROG          zgpg30
TD                32768
SOLVENT           CDCl3
NS                  128
DS                    4
SWH           23980.814 Hz
FIDRES         0.731836 Hz
AQ            0.6832628 sec
RG                16384
DW               20.850 usec
DE               10.00 usec
TE               298.2 K
D1           1.00000000 sec
D11          0.03000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                 13C
P1                 7.50 usec
PL1               -3.00 dB
PL1W        73.67452240 W
SFO1       100.6238350 MHz

======== CHANNEL f2 ========
CPDPRG2          waltz16
NUC2                 1H
PCPD2            100.00 usec
PL2               -2.00 dB
PL12              17.00 dB
PL13              19.30 dB
PL2W        16.00390816 W
PL12W        0.20147727 W
PL13W        0.11863863 W
SFO2       400.1316005 MHz

F2 - Processing parameters
SI                32768
SF          100.6127572 MHz
WDW                  EM
SSB                   0
LB                 1.00 Hz
GB                    0
PC                 1.40
```

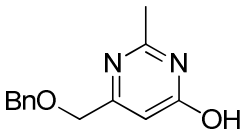



```
Current Data Parameters
NAME        h_bar.HBHC16
EXPNO                  3
PROCNO                 1

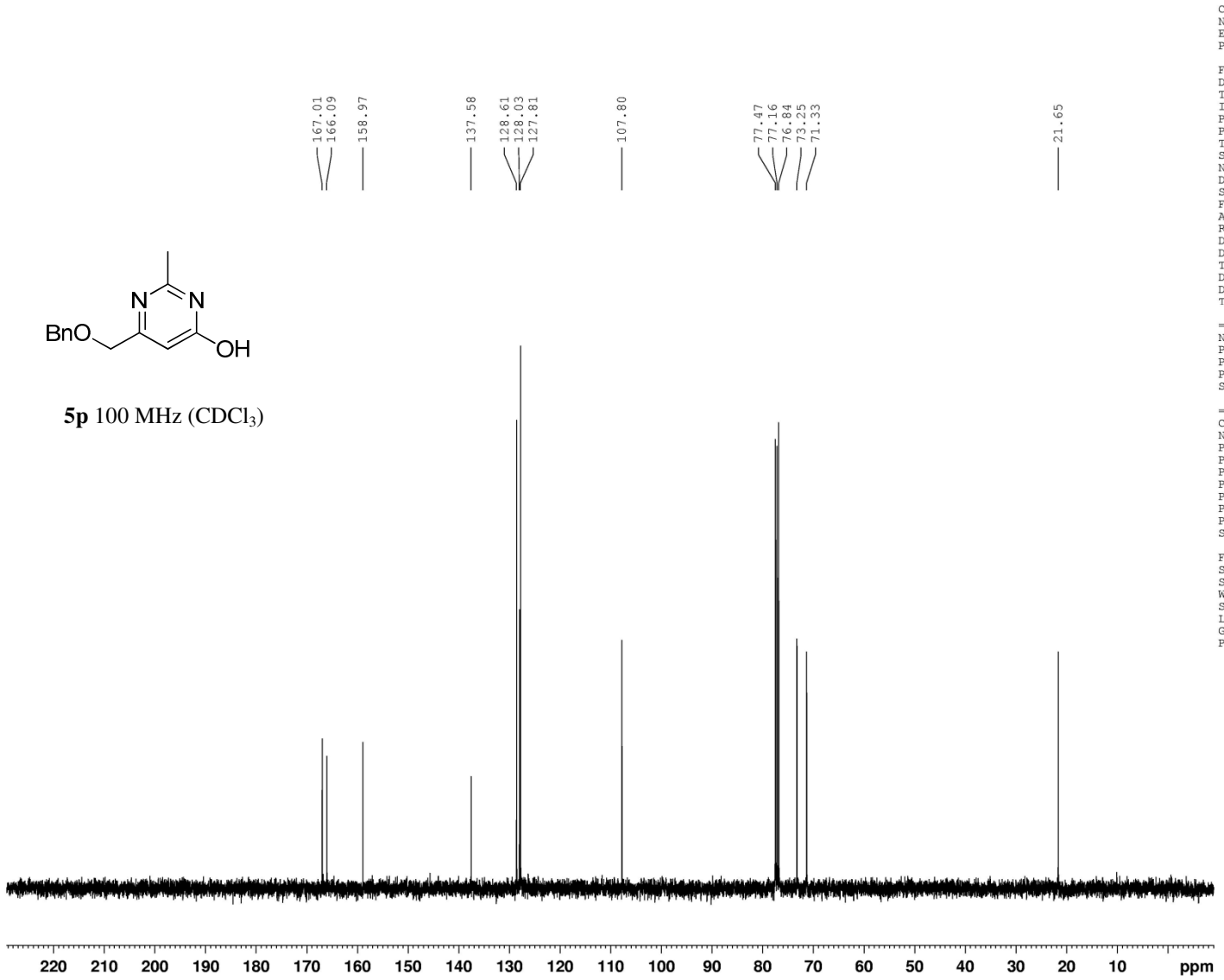
F2 - Acquisition Parameters
Date_           20100630
Time               12.20
INSTRUM          av3400
PROBHD   5 mm PABBI 1H/
PULPROG            zg30
TD                65536
SOLVENT           CDCl3
NS                   16
DS                    2
SWH            8223.685 Hz
FIDRES        0.125483 Hz
AQ            3.9846387 sec
RG                  114
DW               60.800 usec
DE                6.50 usec
TE                298.0 K
D1           1.00000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                  1H
P1                 7.30 usec
PL1                -0.90 dB
PL1W        11.52680206 W
SFO1        400.0724706 MHz

F2 - Processing parameters
SI                 65536
SF          400.0700119 MHz
WDW                   EM
SSB                    0
LB                  0.30 Hz
GB                     0
PC                  1.00
```



5p 400 MHz ($CDCl_3$)



5p 100 MHz ($CDCl_3$)

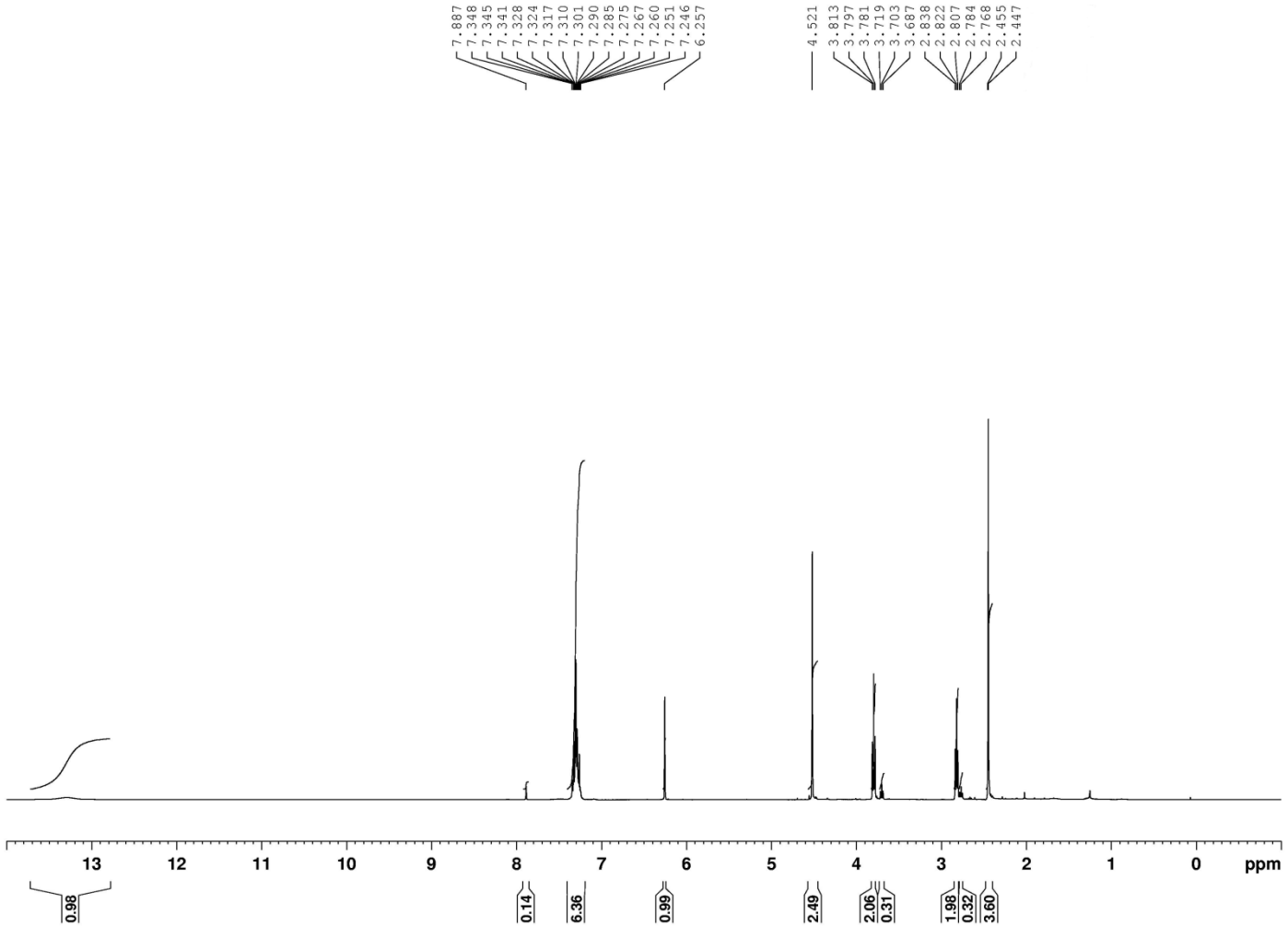
```
Current Data Parameters
NAME        h_bar.HBHC16
EXPNO                  1
PROCNO                 1

F2 - Acquisition Parameters
Date_           20100630
Time               12.14
INSTRUM            av400
PROBHD   5 mm PABBO BB-
PULPROG          zgpg30
TD                32768
SOLVENT           CDCl3
NS                  128
DS                    4
SWH           23980.814 Hz
FIDRES         0.731836 Hz
AQ            0.6832628 sec
RG                16384
DW               20.850 usec
DE               10.00 usec
TE               298.2 K
D1           1.00000000 sec
D11          0.03000000 sec
TD0                   1

======== CHANNEL f1 ========
NUC1                 13C
P1                 7.50 usec
PL1                -3.00 dB
PL1W        73.67452240 W
SFO1       100.6238350 MHz

======== CHANNEL f2 ========
CPDPRG2          waltz16
NUC2                 1H
PCPD2            100.00 usec
PL2                -2.00 dB
PL12               17.00 dB
PL13               19.30 dB
PL2W        16.00390816 W
PL12W        0.20147727 W
PL13W        0.11863863 W
SFO2       400.1316005 MHz

F2 - Processing parameters
SI                32768
SF           100.6127579 MHz
WDW                  EM
SSB                   0
LB                 1.00 Hz
GB                    0
PC                 1.40
```

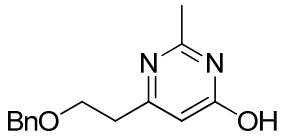



```
Current Data Parameters
NAME      h_bar.HB0386-h
EXPNO                  1
PROCNO                 1

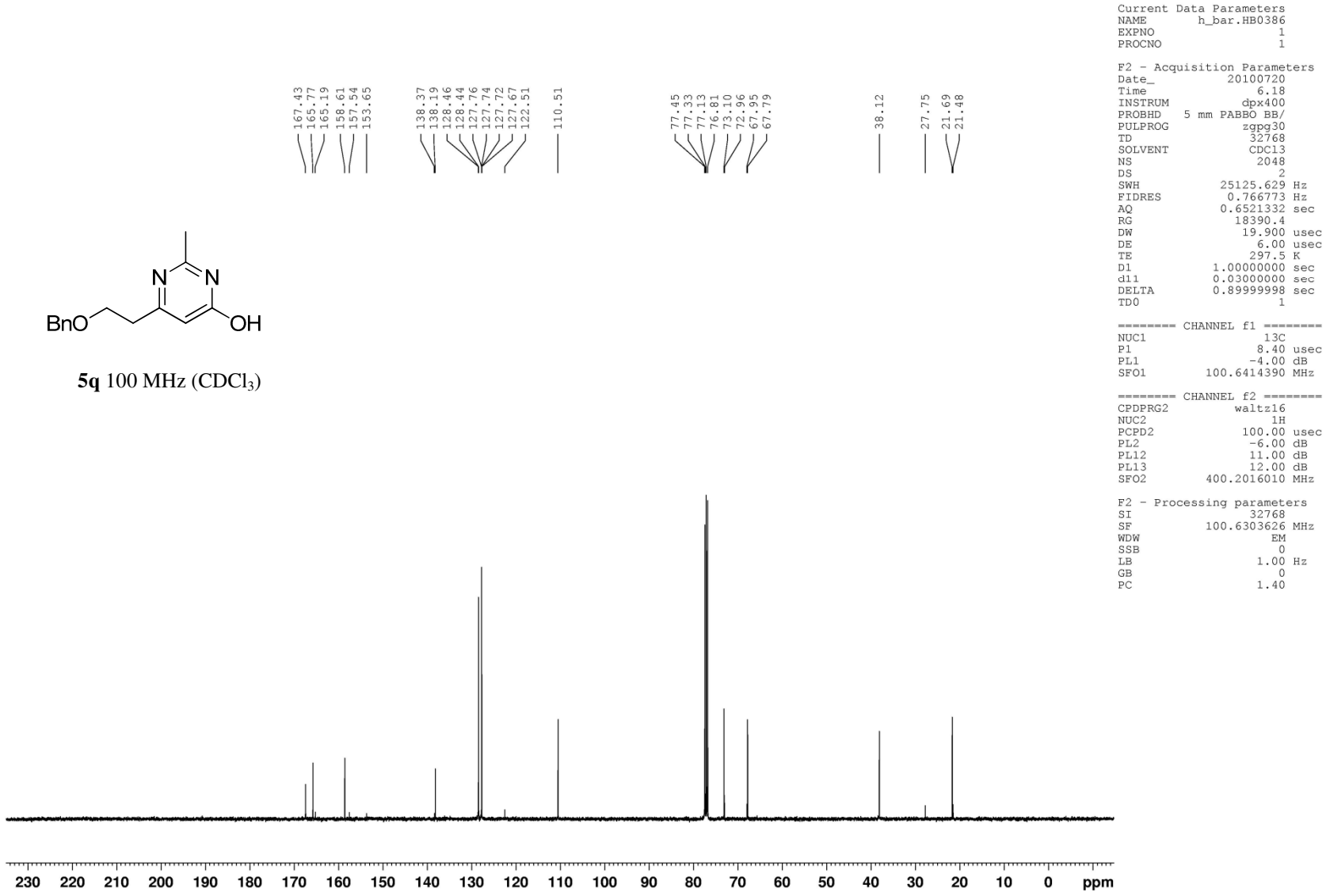
F2 - Acquisition Parameters
Date_           20100719
Time               14.32
INSTRUM          av3400
PROBHD   5 mm PABBI 1H/
PULPROG            zg30
TD                65536
SOLVENT           CDCl3
NS                   16
DS                    2
SWH            8223.685 Hz
FIDRES         0.125483 Hz
AQ             3.9846387 sec
RG                  144
DW                60.800 usec
DE                 6.50 usec
TE                298.0 K
D1           1.00000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                  1H
P1                  7.30 usec
PL1                -0.90 dB
PL1W        11.52680206 W
SFO1        400.0724706 MHz

F2 - Processing parameters
SI                 65536
SF          400.0700121 MHz
WDW                   EM
SSB                    0
LB                  0.30 Hz
GB                     0
PC                  1.00
```



5q 400 MHz ($CDCl_3$)



5q 100 MHz ($CDCl_3$)

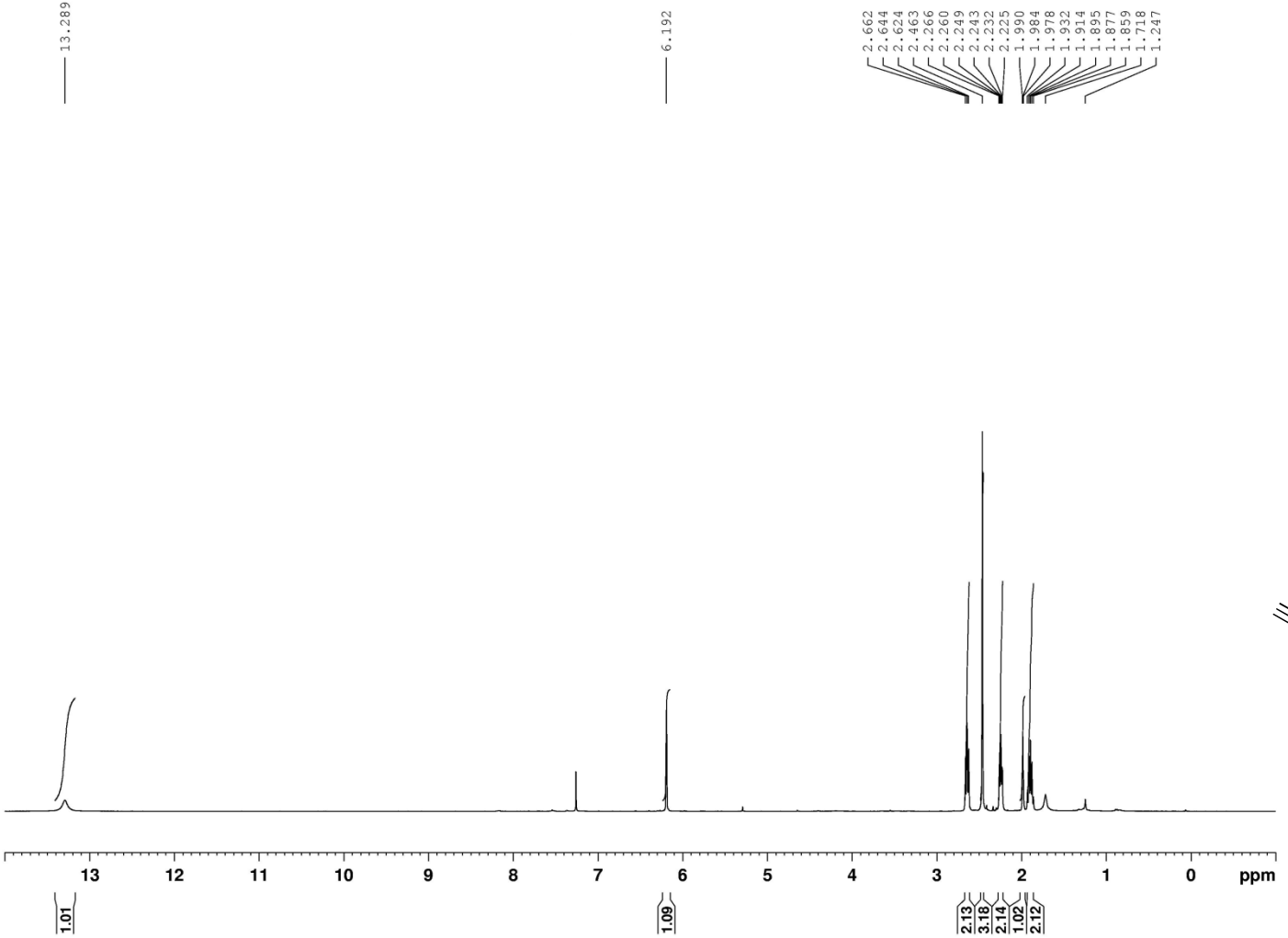
```
Current Data Parameters
NAME        h_bar.HB0386
EXPNO                  1
PROCNO                 1

F2 - Acquisition Parameters
Date_           20100720
Time                6.18
INSTRUM           dpx400
PROBHD   5 mm PABBO BB/
PULPROG           zgpg30
TD                32768
SOLVENT            CDC13
NS                 2048
DS                    2
SWH           25125.629 Hz
FIDRES         0.766773 Hz
AQ             0.6521332 sec
RG               18390.4
DW                19.900 usec
DE                 6.00 usec
TE                297.5 K
D1           1.00000000 sec
d11          0.03000000 sec
DELTA        0.89999998 sec
TD0                   1

======== CHANNEL f1 ========
NUC1                13C
P1                 8.40 usec
PL1               -4.00 dB
SFO1        100.6414390 MHz

======== CHANNEL f2 ========
CPDPRG2          waltz16
NUC2                1H
PCPD2             100.00 usec
PL2                -6.00 dB
PL12               11.00 dB
PL13               12.00 dB
SFO2        400.2016010 MHz

F2 - Processing parameters
SI                 32768
SF          100.6303626 MHz
WDW                  EM
SSB                   0
LB                 1.00 Hz
GB                    0
PC                 1.40
```

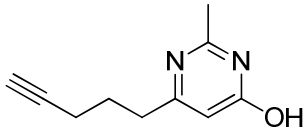



```
Current Data Parameters
NAME        h_bar.HBHC17
EXPNO                  3
PROCNO                 1

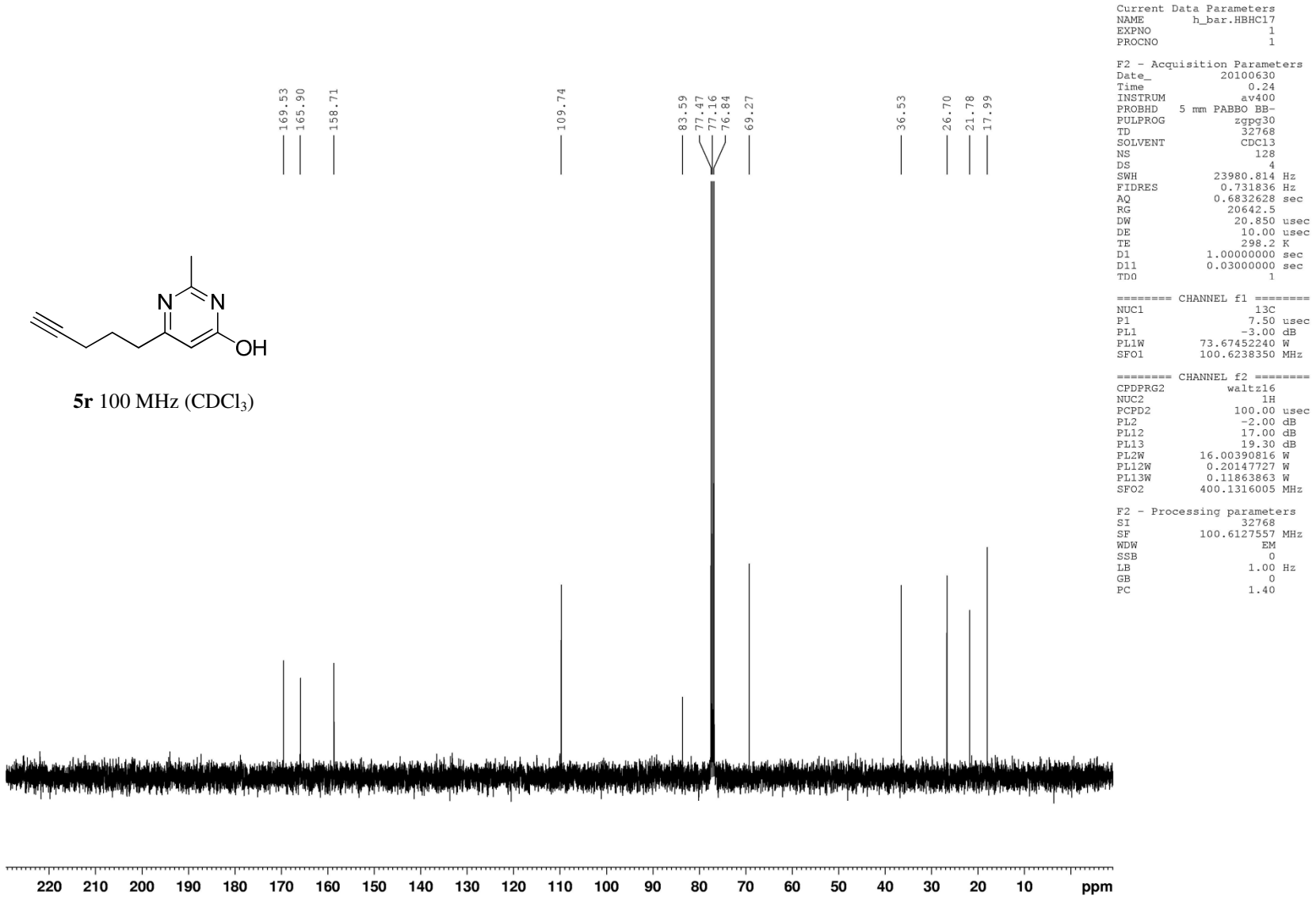
F2 - Acquisition Parameters
Date_           20100629
Time               18.37
INSTRUM          av3400
PROBHD   5 mm PABBI 1H/
PULPROG            zg30
TD                65536
SOLVENT           CDCl3
NS                   16
DS                    2
SWH            8223.685 Hz
FIDRES         0.125483 Hz
AQ             3.9846387 sec
RG                  228
DW                60.800 usec
DE                 6.50 usec
TE                298.0 K
D1           1.00000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                  1H
P1                 7.30 usec
PL1                -0.90 dB
PL1W        11.52680206 W
SFO1        400.0724706 MHz

F2 - Processing parameters
SI                 65536
SF          400.0700123 MHz
WDW                   EM
SSB                    0
LB                  0.30 Hz
GB                     0
PC                  1.00
```



5r 400 MHz ($CDCl_3$)



5r 100 MHz ($CDCl_3$)

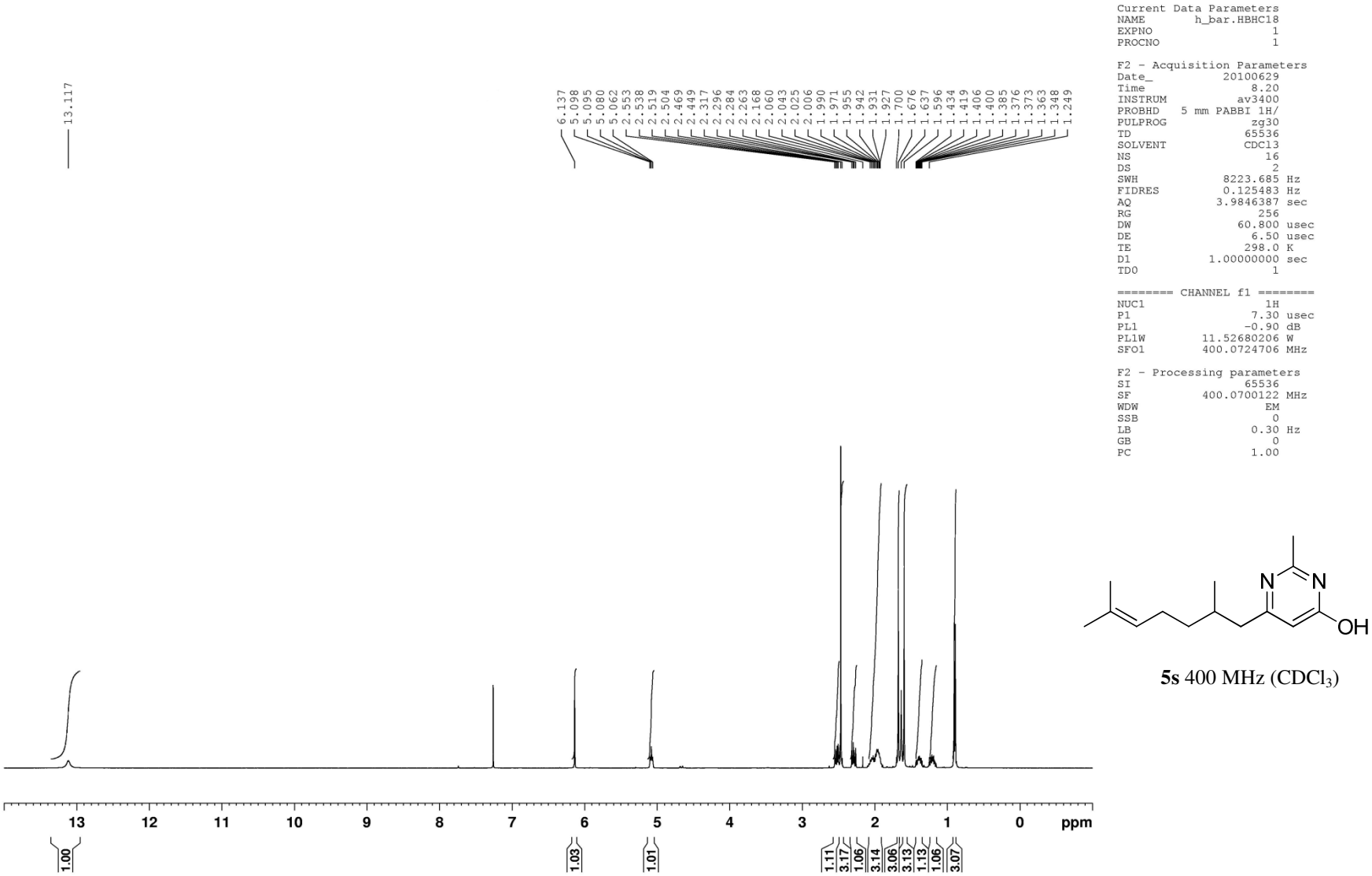
```
Current Data Parameters
NAME        h_bar.HBHC17
EXPNO                  1
PROCNO                 1

F2 - Acquisition Parameters
Date_           20100630
Time                0.24
INSTRUM          av400
PROBHD   5 mm PABBO BB-
PULPROG          zgpg30
TD                32768
SOLVENT           CDCl3
NS                  128
DS                    4
SWH           23980.814 Hz
FIDRES         0.731836 Hz
AQ            0.6832628 sec
RG              20642.5
DW               20.850 usec
DE                10.00 usec
TE                298.2 K
D1           1.00000000 sec
D11          0.03000000 sec
TD0                   1

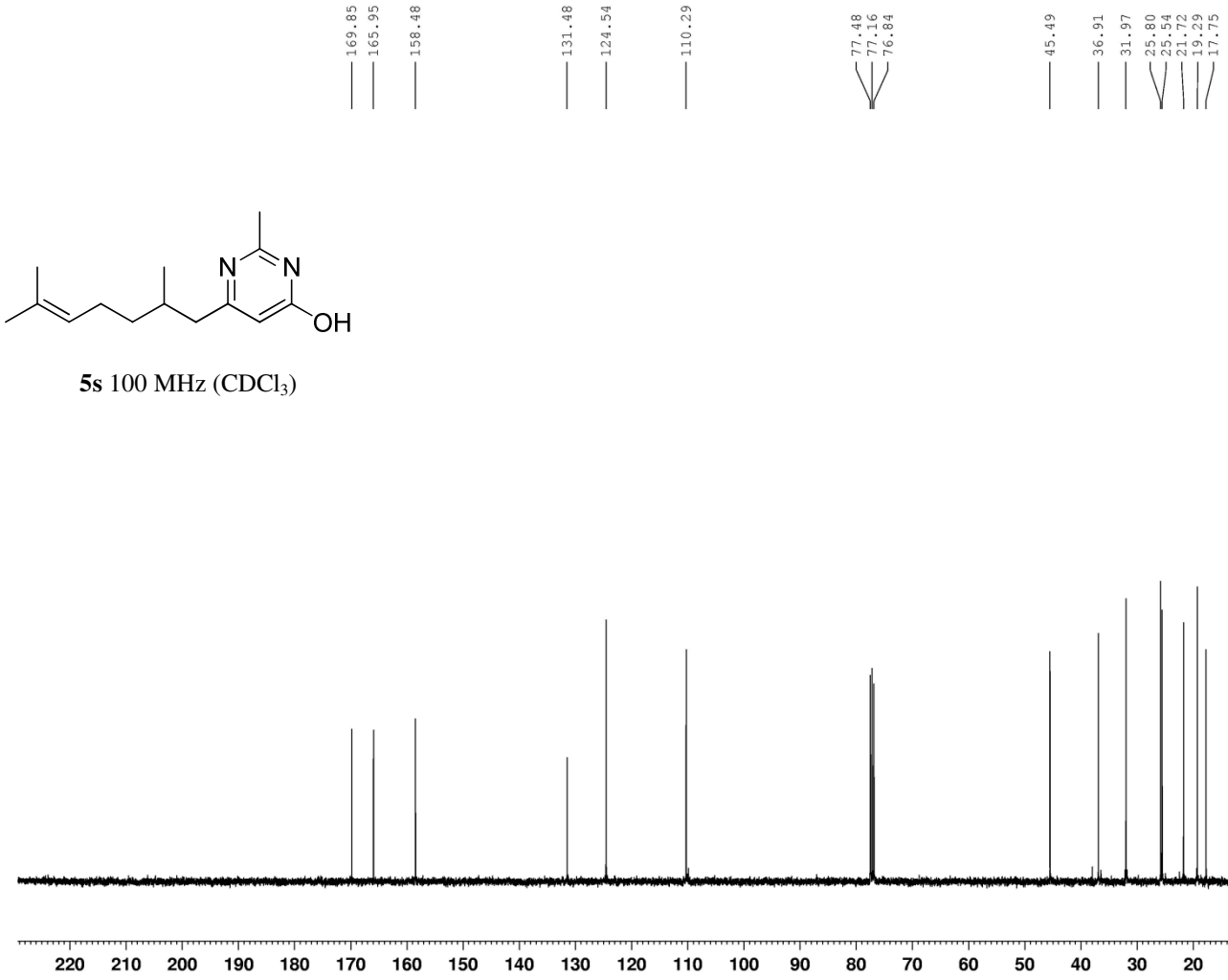
======== CHANNEL f1 ========
NUC1                 13C
P1                  7.50 usec
PL1                -3.00 dB
PL1W        73.67452240 W
SFO1       100.6238350 MHz

======== CHANNEL f2 ========
CPDPRG2          waltz16
NUC2                 1H
PCPD2             100.00 usec
PL2                -2.00 dB
PL12               17.00 dB
PL13               19.30 dB
PL2W        16.00390816 W
PL12W        0.20147727 W
PL13W        0.11863863 W
SFO2       400.1316005 MHz

F2 - Processing parameters
SI                32768
SF           100.6127557 MHz
WDW                  EM
SSB                   0
LB                 1.00 Hz
GB                    0
PC                 1.40
```



5s 400 MHz ($CDCl_3$)



5s 100 MHz ($CDCl_3$)

```
Current Data Parameters
NAME      h_bar.HBHC18-C
EXPNO                  1
PROCNO                 1

F2 - Acquisition Parameters
Date_           20100630
Time                1.30
INSTRUM           av400
PROBHD   5 mm PABBO BB-
PULPROG          zgpg30
TD                32768
SOLVENT           CDC13
NS                  128
DS                    4
SWH           23980.814 Hz
FIDRES         0.731836 Hz
AQ            0.6832628 sec
RG              18390.4
DW                20.850 usec
DE                10.00 usec
TE                298.2 K
D1           1.00000000 sec
D11          0.03000000 sec
TD0                   1

======== CHANNEL f1 ========
NUC1                 13C
P1                  7.50 usec
PL1                -3.00 dB
PL1W        73.67452240 W
SFO1       100.6238350 MHz

======== CHANNEL f2 ========
CPDPRG2          waltz16
NUC2                 1H
PCPD2             100.00 usec
PL2                -2.00 dB
PL12               17.00 dB
PL13               19.30 dB
PL2W        16.00390816 W
PL12W        0.20147727 W
PL13W        0.11863863 W
SFO2       400.1316005 MHz

F2 - Processing parameters
SI                32768
SF          100.6127579 MHz
WDW                  EM
SSB                   0
LB                 1.00 Hz
GB                    0
PC                 1.40
```