Alkyl–Alkyl Suzuki Cross-Couplings of Unactivated Secondary Alkyl Halides at Room Temperature

Bunnai Saito and Gregory C. Fu*
Department of Chemistry, Massachusetts Institute of Technology, Cambridge, Massachusetts 02139

Supporting Information

I. General

The following reagents were purchased and used as received: 9-borabicyclo[3.3.1]nonane dimer (Aldrich), $NiCl_2$·glyme (Strem), *rac-trans-N,N'*-dimethyl-1,2-cyclohexanediamine (Aldrich), KO*t*-Bu (Acros), *i*-BuOH (anhydrous, Aldrich), 1,4-dioxane (anhydrous, Fluka), bromocyclohexane (Alfa), bromocyclopentane (Alfa), bromocycloheptane (Alfa), (±)-*exo*-2-bromonorbornane (Aldrich), 2-bromo-1-phenylpropane (Acros), benzyl 4-bromo-1-piperidinecarboxylate (Aldrich), iodocyclohexane (Alfa), *N*-(4-bromobutyl)phthalimide (Alfa), 1-iodo-3-methylbutane (Aldrich), allylbenzene (Alfa), 4-pentene-1-ol (TCI), methyl 3,3-dimethyl-4-pentenoate (TCI), and 1-allyl-3,4-dimethoxybenzene (TCI). All other compounds were prepared via published procedures.

II. Suzuki Cross-Couplings

Generation of the *B*-alkyl-9-BBN reagent: General procedure. In a glove box, the olefin (37.5 mmol) was added to a suspension of 9-BBN dimer (18.8 mmol) in dioxane (10 mL), and the resulting reaction mixture was stirred at 60 °C for 1 h. The mixture was then allowed to cool to room temperature, and dioxane was added to produce a total volume of 25 mL (~1.5 M).

Generation of the activated *B*-alkyl-9-BBN reagent: General procedure. In a glove box, the solution of the *B*-alkyl-9-BBN reagent (1.5 M; 1.44 mL, 2.16 mmol) was added to a mixture of KO*t*-Bu (162 mg, 1.44 mmol) and *i*-BuOH (222 μL, 2.4 mmol) in a vial. The resulting mixture was stirred vigorously at room temperature for at least 30 min prior to use in the cross-coupling reaction.

Suzuki cross-coupling reactions: General procedure (Table 2). In a glove box, a solution of the activated *B*-alkyl-9-BBN reagent (1.8 mmol) and then the alkyl bromide (1.0 mmol) were added to a mixture of $NiCl_2$·glyme (13.2 mg, 0.060 mmol) and *rac-trans-N,N'*-dimethylcyclohexane-1,2-diamine (12.6 μL, 0.080 mmol) in dioxane (0.80 mL) in a 4-mL vial. The resulting solution was stirred vigorously at room temperature for 24 h, and then it was passed through a short pad of silica gel (washed with hexane/Et_2O 1:1). The solvent was removed on a rotary evaporator, and the residue was purified by flash chromatography.

Note: If the cross-coupling is set up without the use of a glove box, the yield for the process illustrated in Table 1 is 71% (vs. 83% when a glovebox is employed: entry 1).

(3-Cyclohexylpropyl)benzene [170661-44-6] (Table 2, entry 1). Cyclohexyl bromide (123 μL, 1.0 mmol) and a solution of the boron reagent prepared by hydroboration of allylbenzene with 9-BBN (1.5 M solution in dioxane; 1.2 mL, 1.8 mmol) were used. Solvent for chromatography: hexane (100%). Colorless oil. First run: 146 mg (72%). Second run: 158 mg (78%).

^{1}H NMR ($CDCl_3$, 400 MHz) δ 7.27-7.24 (m, 2H), 7.17-7.13 (m, 3H), 2.46 (t, J = 7.8 Hz, 2H), 1.69-1.56 (m, 7H), 1.23-1.08 (m, 6H), 0.88-0.79 (m, 2H).

(3-Cyclopentylpropyl)benzene [2883-12-7] (Table 2, entry 2). Cyclopentyl bromide (107 μL, 1.0 mmol) and a solution of the boron reagent prepared by hydroboration of allylbenzene with 9-BBN (1.5 M solution in dioxane; 1.2 mL, 1.8 mmol) were used. Solvent for chromatography: hexane (100%). Colorless oil. First run: 137 mg (73%). Second run: 152 mg (81%).

^{1}H NMR ($CDCl_3$, 400 MHz) δ 7.28-7.24 (m, 2H), 7.17-7.14 (m, 3H), 2.58 (t, J = 7.9 Hz, 2H), 1.77-1.54 (m, 3H), 1.54-1.45 (m, 6H), 1.35-1.30 (m, 2H), 1.09-1.00 (m, 2H).

(3-Cycloheptylpropyl)benzene (Table 2, entry 3). Cycloheptyl bromide (136 μL, 1.0 mmol) and a solution of the boron reagent prepared by hydroboration of allylbenzene with 9-BBN (1.5 M solution in dioxane; 1.2 mL, 1.8 mmol) were used. Solvent for chromatography: hexane (100%). Colorless oil. First run: 161 mg (80%). Second run: 160 mg (79%).

^{1}H NMR ($CDCl_3$, 400 MHz) δ 7.27-7.24 (m, 2H), 7.17-7.13 (m, 3H), 2.56 (t, J = 7.8 Hz, 2H), 1.69-1.33 (m, 13H), 1.27-1.21 (m, 2H), 1.17-1.09 (m, 2H).

^{13}C NMR ($CDCl_3$, 100 MHz) δ 143.2, 128.6, 128.4, 125.8, 39.4, 38.1, 36.6, 34.8, 29.7, 28.8, 26.8.

IR (neat) 3063, 3026, 2922, 2852, 1605, 1496, 1460, 1030, 747, 698 cm^{-1}.

MS (EI) m/z (M^+) calcd for $C_{16}H_{24}$: 216, found: 216.

2-(3-Phenylpropyl)bicyclo[2.2.1]heptane (Table 2, entry 4). (±)-*exo*-2-Bromonorbornane (128 μL, 1.0 mmol) and a solution of the boron reagent prepared by hydroboration of allylbenzene with 9-BBN (1.5 M solution in dioxane; 1.2 mL, 1.8 mmol) were used. Solvent for chromatography: hexane (100%). Colorless oil. First run: 156 mg (73%). Second run: 164 mg (77%). The stereochemistry of the product is tentatively assigned as exo, on the basis of analogy with prior work.[1]

^{1}H NMR ($CDCl_3$, 400 MHz) δ 7.27-7.24 (m, 2H), 7.16-7.13 (m, 3H), 2.56 (t, J = 7.7 Hz, 2H), 2.13 (br s, 1H), 1.91 (br s, 1H), 1.60-1.52 (m, 2H), 1.49-1.23 (m, 6H), 1.13-0.91 (m, 5H).

^{13}C NMR ($CDCl_3$, 100 MHz) δ 143.2, 128.6, 128.4, 125.8, 42.4, 41.3, 38.4, 36.9, 36.8, 36.4, 35.5, 30.4, 30.1, 29.1.

IR (neat) 3063, 3026, 2948, 2868, 1604, 1496, 1453, 1030, 745, 698 cm^{-1}.

MS (EI) *m*/*z* (M^+) calcd for $C_{16}H_{22}$: 214, found: 214.

2-(3-Phenylpropyl)bicyclo[2.2.1]heptane (Table 2, entry 5). (±)-*endo*-2-Bromonorbornane (175 mg, 1.0 mmol) and a solution of the boron reagent prepared by hydroboration of allylbenzene with 9-BBN (1.5 M solution in dioxane; 1.2 mL, 1.8 mmol) were used. Solvent for chromatography: hexane (100%). Colorless oil. First run: 136 mg (63%). Second run: 143 mg (67%). The stereochemistry of the product is tentatively assigned as exo, on the basis of analogy with prior work.[1]

The ^{1}H NMR and ^{13}C NMR spectra of the product were identical with those of Table 2, entry 4.

2-Methyl-1,5-diphenylpentane [31444-36-7] (Table 2, entry 6). 2-Bromo-1-phenylpropane (154 μL, 1.0 mmol) and a solution of the boron reagent prepared by hydroboration of allylbenzene with 9-BBN (1.5 M solution in dioxane; 1.2 mL, 1.8 mmol) were used. Solvent for chromatography: hexane/EtOAc (50:1). Colorless oil. First run: 195 mg (82%). Second run: 196 mg (82%).

^{1}H NMR ($CDCl_3$, 400 MHz) δ 7.31-7.26 (m, 4H), 7.21-7.13 (m, 6H), 2.67-2.53 (m, 3H), 2.37 (dd, *J* = 13.4, 8.2 Hz, 1H), 1.79-1.57 (m, 3H), 1.47-1.35 (m, 1H), 1.28-1.18 (m, 1H), 0.86 (d, *J* = 6.6 Hz, 3H).

(3-(4-Chlorocyclohexyl)propyl)benzene (Table 2, entry 7). *cis*-1-Bromo-4-chlorocyclohexane (198 mg, 1.0 mmol) and a solution of the boron reagent prepared by hydroboration of allylbenzene with 9-BBN (1.5 M solution in dioxane; 1.2 mL, 1.8 mmol) were used. Solvent for chromatography: hexane/EtOAc (50:1). Colorless oil. First run: 198 mg (83%). Second run: 192 mg (81%). We were unable to separate the diastereomeric products (2:1 trans:cis).

^{1}H NMR ($CDCl_3$, 400 MHz) δ 7.29-7.26 (m, 2H), 7.20-7.16 (m, 3H), 4.41 (t, *J* = 3.5 Hz, 0.35H), 4.41 (tt, *J* = 4.2, 11.6 Hz, 0.65H), 2.62-2.56 (m, 2H), 2.20-2.16 (m, 1.3H), 1.98-1.94 (m, 0.7H), 1.83-1.71 (m, 2H), 1.68-1.44 (m, 5H), 1.33-1.20 (m, 3H), 1.03-0.92 (m, 1H).

^{13}C NMR ($CDCl_3$, 100 MHz) δ 142.8, 128.6, 128.54, 128.46, 125.9, 125.8, 60.4, 60.3, 37.3, 36.4, 36.33, 36.29, 33.9, 33.0, 29.1, 29.0, 27.2.

IR (neat) 3061, 3025, 2931, 2857, 1603, 1496, 1453, 1113, 1087, 1018, 958, 924, 893, 749, 700 cm^{-1}.

MS (EI) *m*/*z* (M^+) calcd for $C_{15}H_{21}Cl$: 236, found: 236.

***tert*-Butyldimethyl(5-(tetrahydro-2*H*-pyran-4-yl)pentyloxy)silane (Table 2, entry 8)**. 4-Bromotetrahydropyran (125 mg, 1.0 mmol) and a solution of the boron reagent prepared by hydroboration of *tert*-butyldimethyl(4-pentenyloxy)silane with 9-BBN (1.5 M solution in dioxane; 1.2 mL, 1.8 mmol) were used. Solvent for chromatography: hexane/EtOAc (20:1). Colorless oil. First run: 249 mg (87%). Second run: 248 mg (86%).

^{1}H NMR ($CDCl_3$, 400 MHz) δ 3.94-3.90 (m, 2H), 3.58 (t, *J* = 6.6 Hz, 2H), 3.34 (td, *J* = 11.8, 2.0 Hz, 2H), 1.58-1.38 (m, 5H), 1.30-1.17 (m, 8H), 0.87 (s, 9H), 0.03 (s, 6H).

^{13}C NMR ($CDCl_3$, 100 MHz) δ 68.4, 63.4, 37.1, 35.1, 33.4, 33.0, 26.3, 26.2, 26.1, 18.6, -5.1.

IR (neat) 2929, 2856, 1472, 1464, 1443, 1387, 1361, 1255, 1102, 1007, 981, 836, 775, 662 cm^{-1}.

MS (EI) *m*/*z* (M^+) calcd for $C_{16}H_{34}O_2Si$: 286, found: 229 (M-*t*-Bu).

Benzyl 4-(5-methoxy-3,3-dimethyl-5-oxopentyl)piperidine-1-carboxylate (Table 2, entry 9). Benzyl 4-bromo-1-piperidinecarboxylate (217 μL, 1.0 mmol) and a solution of the boron reagent prepared by hydroboration of methyl 3,3-dimethyl-4-pentenoate with 9-BBN (1.5 M solution in dioxane; 1.2 mL, 1.8 mmol) were used. Solvent for chromatography: CH_2Cl_2/EtOAc (20:1). Colorless oil. First run: 243 mg (67%). Second run: 218 mg (60%).

^{1}H NMR ($CDCl_3$, 400 MHz) δ 7.34-7.24 (m, 5H), 5.10 (s, 2H), 4.14 (br s, 2H), 3.62 (s, 3H), 2.72 (br s, 2H), 2.17 (s, 2H), 1.67-1.64 (m, 2H), 1.31-1.07 (m, 7H), 0.95 (s, 6H).

^{13}C NMR ($CDCl_3$, 100 MHz) δ 172.9, 155.5, 137.2, 128.6, 128.1, 128.0, 67.1, 51.3, 45.8, 44.4, 39.1, 36.7, 33.2, 32.3, 30.9, 27.5.

IR (neat) 3065, 3032, 2931, 2856, 1734, 1701, 1434, 1364, 1320, 1276, 1228, 1164, 1085, 1019, 963, 761, 738, 699 cm^{-1}.

MS (ESI) *m*/*z* ($M+H^+$) calcd for $C_{21}H_{32}NO_4$: 362, found: 362.

4-(3-(3,4-Dimethoxyphenyl)propyl)tetrahydro-2*H*-pyran (Table 2, entry 10). 4-Bromotetrahydoropyran (125 mg, 1.0 mmol) and a solution of the boron reagent prepared by hydroboration of 1-allyl-3,4-dimethoxybenzene with 9-BBN (1.5 M solution in dioxane; 1.2 mL, 1.8 mmol) were used. Solvent for chromatography: hexane/EtOAc (4:1). Colorless oil. First run: 210 mg (80%). Second run: 205 mg (78%).

^{1}H NMR ($CDCl_3$, 400 MHz) δ 6.78-6.76 (m, 1H), 6.70-6.68 (m, 2H), 3.93 (dd, *J* = 11.1, 4.4 Hz, 2H), 3.86 (s, 3H), 3.84 (s, 3H), 3.34 (td, *J* = 11.1, 1.9 Hz, 2H), 2.52 (t, *J* = 7.8 Hz, 2H), 1.67-1.41 (m, 5H), 1.30-1.19 (m, 4H).

^{13}C NMR ($CDCl_3$, 100 MHz) δ 148.8, 147.2, 135.4, 120.2, 111.8, 111.2, 68.3, 56.0, 55.9, 36.7, 35.8, 35.1, 33.3, 28.6.

IR (neat) 2996, 2929, 2839, 1607, 1591, 1516, 1464, 1417, 1387, 1330, 1262, 1237, 1156, 1141, 1096, 1031, 980, 873, 854, 806, 765, 611 cm^{-1}.

MS (EI) *m*/*z* (M^+) calcd for $C_{16}H_{24}O_3$: 264, found: 264.

***tert*-Butyl(5-cyclohexylpentyloxy)dimethylsilane (Table 2, entry 11)**. Cyclohexyl bromide (123 mL, 1.0 mmol) and a solution of the boron reagent prepared by hydroboration of *tert*-butyldimethyl(4-pentenyloxy)silane with 9-BBN (1.5 M solution in dioxane; 1.2 mL, 1.8 mmol) were used. Solvent for chromatography: hexane/EtOAc (50:1). Colorless oil. First run: 228 mg (80%). Second run: 233 mg (82%).

^{1}H NMR (C_6D_6, 400 MHz) δ 3.58 (t, *J* = 6.4 Hz, 2H), 1.71-1.64 (m, 5H), 1.59-1.52 (m, 2H), 1.39-1.13 (m, 10H), 1.00 (s, 9H), 0.90-0.82 (m, 2H), 0.09 (s, 6H).

^{13}C NMR (C_6D_6, 100 MHz) δ 63.7, 38.4, 38.3, 34.1, 33.7, 27.5, 27.4, 27.2, 27.0, 26.5, 18.9, -4.8.

IR (neat) 2927, 2855, 1472, 1463, 1449, 1388, 1361, 1255, 1101, 1006, 835, 775, 662 cm^{-1}.
MS (EI) *m*/*z* (M^+) calcd for $C_{17}H_{36}OSi$: 284, found: 227 (M-*t*-Bu).

Methyl 5-cyclohexyl-3,3-dimethylpentanoate (Table 2, entry 12). Cyclohexyl bromide (123 μL, 1.0 mmol) and a solution of the boron reagent prepared by hydroboration of methyl 3,3-dimethyl-4-pentenoate with 9-BBN (1.5 M solution in dioxane; 1.2 mL, 1.8 mmol) were used. Solvent for chromatography: hexane/EtOAc (50:1). Colorless oil. First run: 211 mg (93%). Second run: 206 mg (91%).

^{1}H NMR ($CDCl_3$, 400 MHz) δ 3.62 (s, 3H), 2.17 (s, 2H), 1.69-1.60 (m, 5H), 1.29-1.09 (m, 8H), 0.94 (s, 6H), 0.85-0.83 (m, 2H).

^{13}C NMR ($CDCl_3$, 100 MHz) δ 173.0, 51.3, 46.0, 39.7, 38.5, 33.7, 33.3, 31.8, 27.5, 26.9, 26.6.

IR (neat) 2923, 2852, 1740, 1448, 1389, 1368, 1332, 1230, 1133, 1023, 886 cm^{-1}.

HRMS (ESI) *m*/*z* ($M+H^+$) calcd for $C_{14}H_{27}O_2$: 227.2009, found: 227.2008.

Methyl 3,3,6-trimethyl-7-phenylheptanoate (Table 2, entry 13). 2-Bromo-1-phenylpropane (154 mg, 1.0 mmol) and a solution of the boron reagent prepared by hydroboration of methyl 3,3-dimethyl-4-pentenoate with 9-BBN (1.5 M solution in dioxane; 1.2 mL, 1.8 mmol) were used. Solvent for chromatography: hexane/EtOAc (25:1). Colorless oil. First run: 249 mg (95%). Second run: 240 mg (91%).

^{1}H NMR ($CDCl_3$, 400 MHz) δ 7.27-7.23 (m, 2H), 7.17-7.11 (m, 3H), 3.62 (s, 3H), 2.65 (dd, *J* = 13.4, 6.1 Hz, 1H), 2.32 (dd, *J* = 13.4, 8.4 Hz, 1H), 2.17 (s, 2H), 1.69-1.58 (m, 1H), 1.40-1.22 (m, 3H), 1.16-1.06 (m, 1H), 0.96 (s, 6H), 0.82 (d, *J*= 6.6 Hz, 3H).

^{13}C NMR ($CDCl_3$, 100 MHz) δ 173.0, 141.6, 129.3, 128.2, 125.8, 51.2, 45.9, 43.7, 39.6, 35.7, 33.3, 31.0, 27.50, 27.47, 19.6.

IR (neat) 3063, 3027, 2958, 2924, 2872, 1737, 1603, 1495, 1454, 1368, 1330, 1227, 1131, 1021, 737, 700 cm^{-1}.

MS (EI) *m*/*z* (M^+) calcd for $C_{17}H_{26}O_2$: 262, found: 262.

(3-Cyclohexylpropyl)benzene [170661-44-6] (Table 3, entry 1). Cyclohexyl iodide (129 μL, 1.0 mmol) and a solution of the boron reagent prepared by hydroboration of allylbenzene with 9-BBN (1.5 M solution in dioxane; 1.2 mL, 1.8 mmol) were used. Solvent for chromatography: hexane (100%). Colorless oil. First run: 134 mg (66%). Second run: 142 mg (70%).

The ^{1}H NMR spectrum of the product was identical with that of Table 2, entry 1.

2-(7-Phenylheptyl)isoindoline-1,3-dione [151921-82-3] (Table 3, entry 2). *N*-(4-Bromobutyl)phthalimide (282 μL, 1.0 mmol) and a solution of the boron reagent prepared by hydroboration of allylbenzene with 9-BBN (1.5 M solution in dioxane; 1.2 mL, 1.8 mmol) were used. Solvent for chromatography: hexane/EtOAc (9:1). Colorless oil. First run: 255 mg (79%). Second run: 239 mg (74%).

^{1}H NMR ($CDCl_3$, 400 MHz) δ 7.84-7.80 (m, 2H), 7.71-7.66 (m, 2H), 7.26-7.23 (m, 2H), 7.16-7.13 (m, 3H), 3.65 (t, *J* = 7.3 Hz, 2H), 2.46 (t, *J* = 7.7 Hz, 2H), 1.68-1.54 (m, 4H), 1.35-1.27 (m, 6H).

***tert*-Butyldimethyl(8-methylnonyloxy)silane (Table 3, entry 3)**. 1-Iodo-3-methylbutane (132 μL, 1.0 mmol) and a solution of the boron reagent prepared by hydroboration of *tert*-butyldimethyl(4-pentenyloxy)silane with 9-BBN (1.5 M solution in dioxane; 1.2 mL, 1.8 mmol) were used. Solvent for chromatography: hexane/EtOAc (20:1). Colorless oil. First run: 260 mg (96%). Second run: 250 mg (92%).

^{1}H NMR ($CDCl_3$, 400 MHz) δ 3.58 (d, *J* = 6.6 Hz, 2H), 1.54-1.44 (m, 3H), 1.31-1.24 (m, 8H), 1.15-1.10 (m, 2H), 0.88 (s, 9H), 0.84 (d, *J* = 6.6 Hz, 6H), 0.03 (s, 6H).

^{13}C NMR ($CDCl_3$, 100 MHz) δ 63.6, 39.3, 33.1, 30.2, 29.7, 28.2, 27.6, 26.2, 26.1 22.9, 18.6, -5.0.

IR (neat) 2929, 2857, 1471, 1385, 1362, 1255, 1103, 1006, 939, 834, 813, 775, 662 cm^{-1}.

MS (EI) *m*/*z* (M^+) calcd for $C_{16}H_{36}OSi$: 272, found: 215 (M-*t*-Bu).

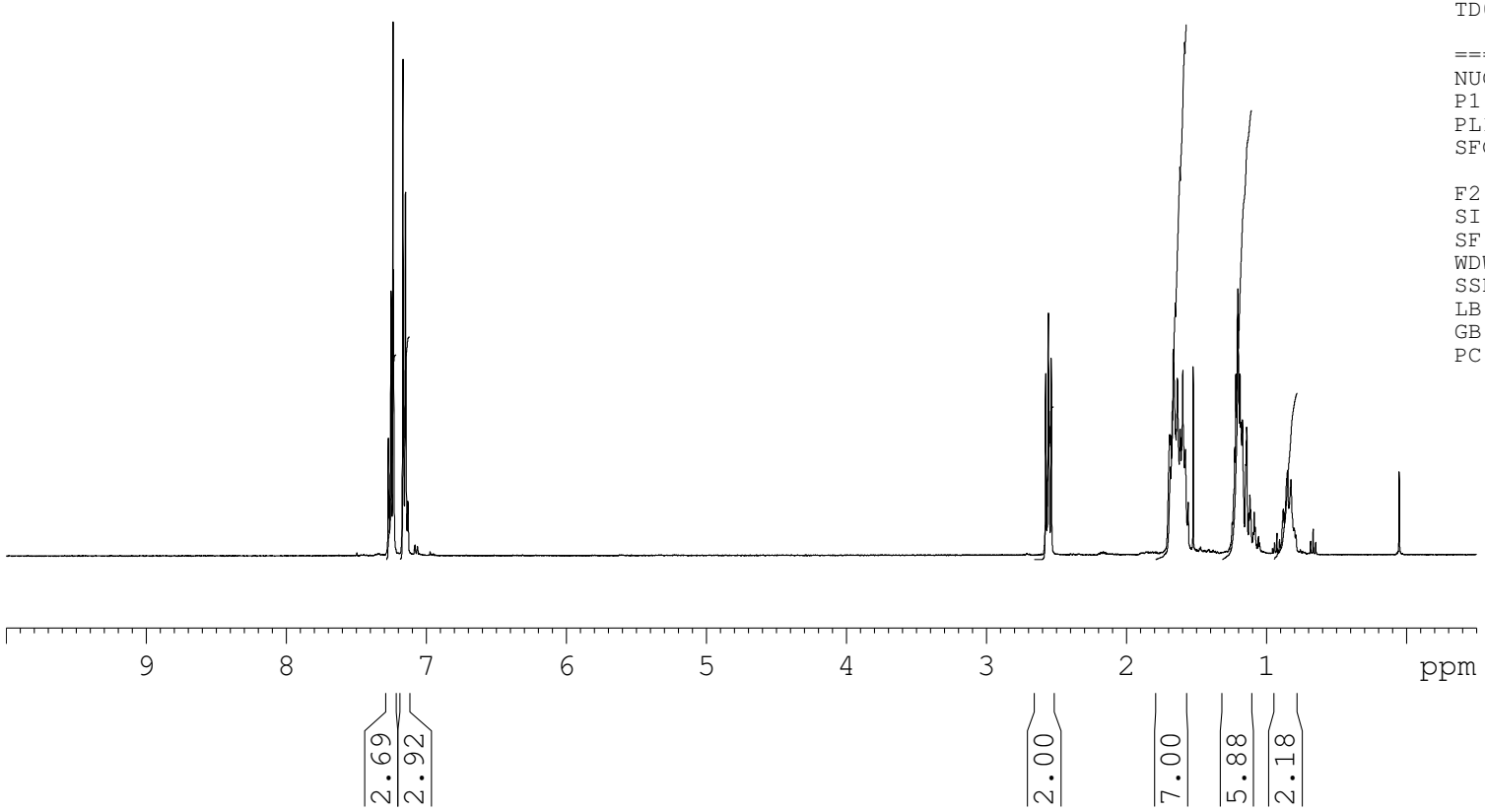
III. Equation 3

i-BuOH (138 μL, 1.5 mmol) and KO*t*-Bu (168 mg, 1.5 mmol) were added to a solution of $PhCH_2CH_2CH_2$(9-BBN) (1.5 mmol) in dioxane (1.0 mL). The mixture was stirred for 1 h at room temperature, and then a ^{11}B NMR spectrum was obtained: a broad resonance was present at δ –0.1, and the resonance due to $PhCH_2CH_2CH_2$(9-BBN) (δ 86) was absent.

IV. References

(1) (a) Zhou, J.; Fu, G. C. *J. Am. Chem. Soc.* **2004**, *126*, 1340–1341. (b) González-Bobes, F.; Fu, G. C. *J. Am. Chem. Soc.* **2006**, *128*, 5360–5361.

Table 2, Entry 1

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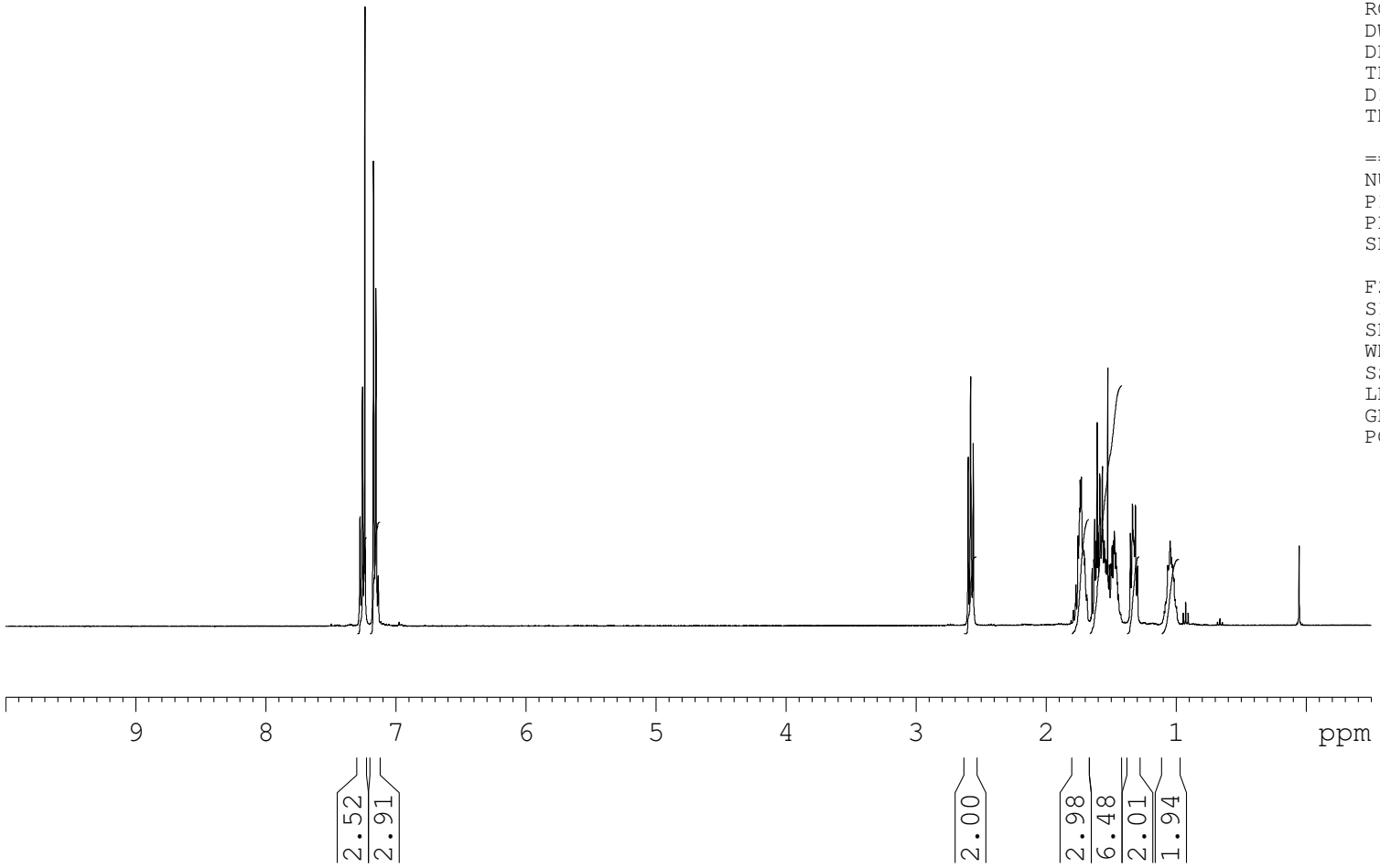


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DS                     2
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TE                293.2 K
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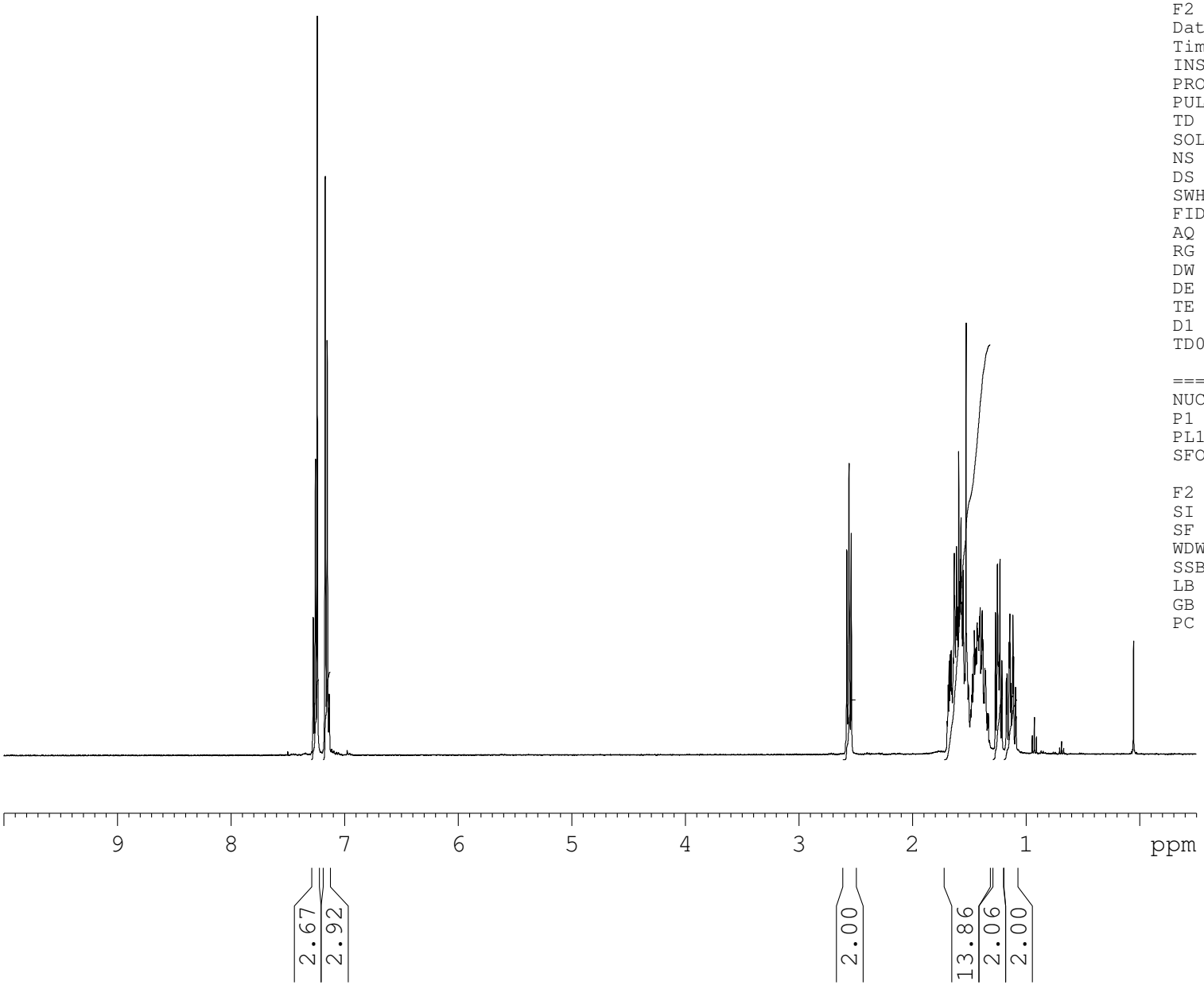


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TE                292.2 K
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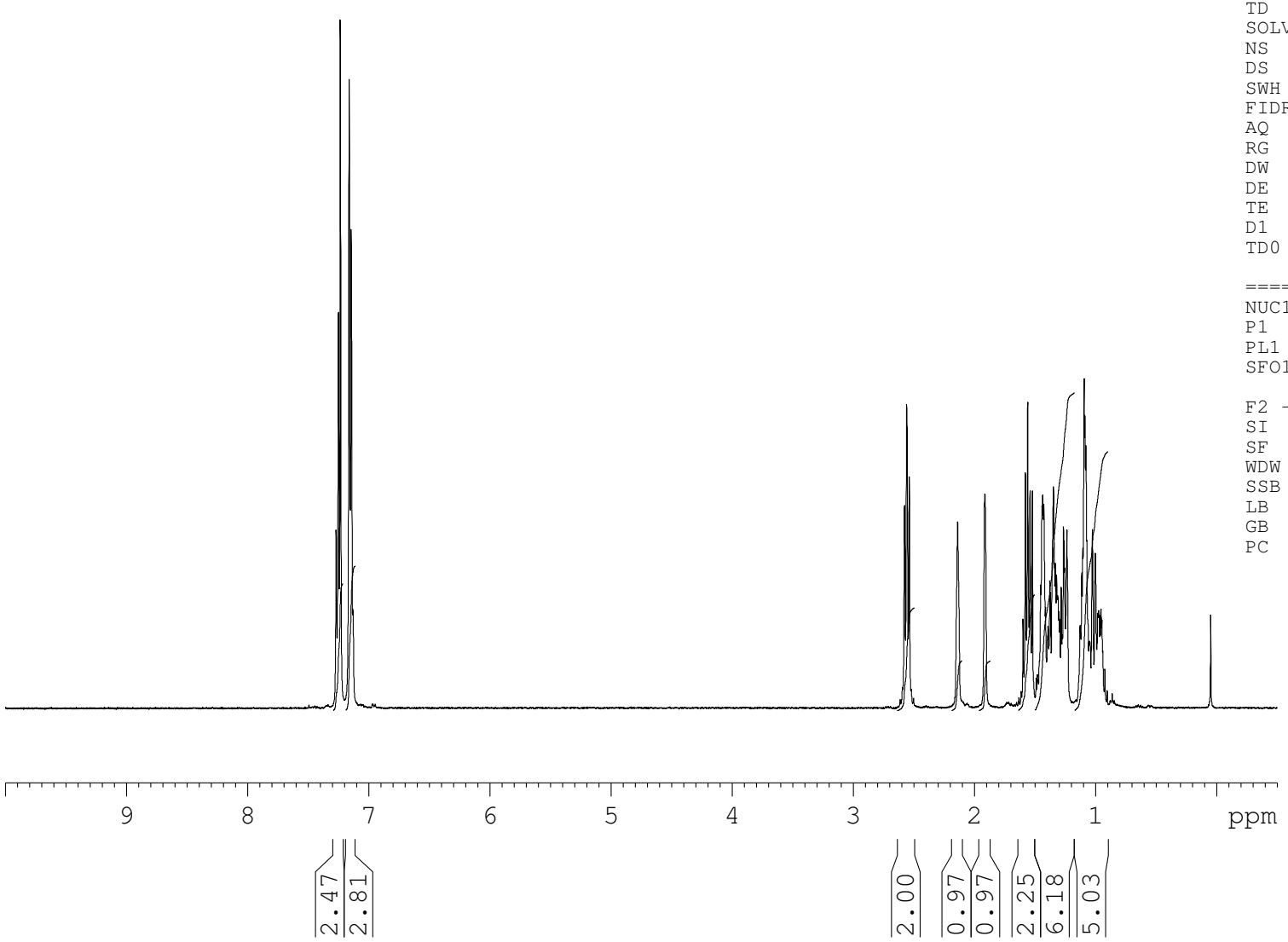
Table 2, Entry 4

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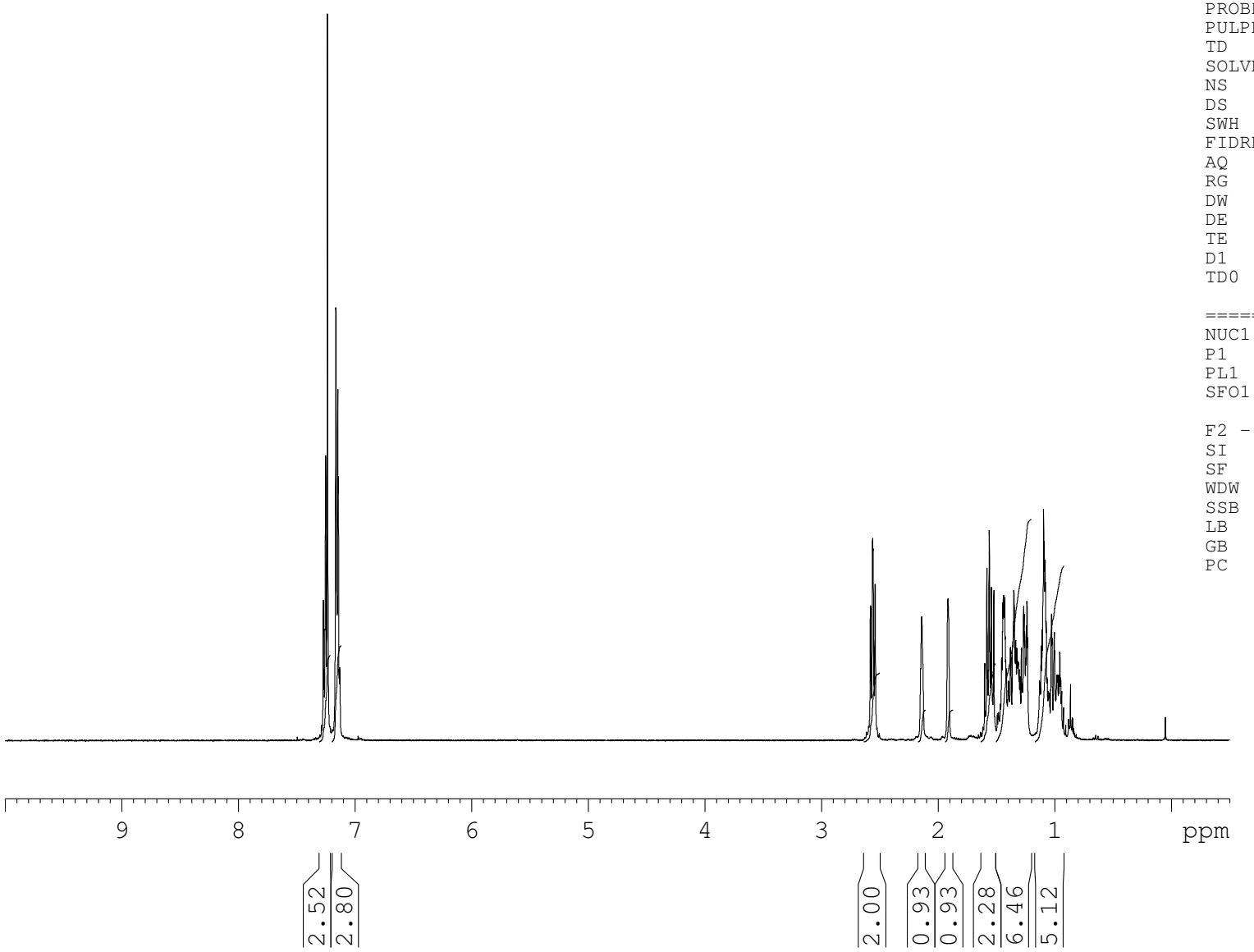
SOLVENT            CDCl3
NS                    16
DS                     2
SWH             8278.146 Hz
FIDRES          0.126314 Hz
AQ             3.9584243 sec
RG                 287.4
DW                60.400 usec
DE                 6.00 usec
TE                293.2 K
D1           1.00000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                  1H
P1                15.07 usec
PL1                0.00 dB
SFO1         400.1324710 MHz

F2 - Processing parameters
SI                 65536
SF          400.1300190 MHz
WDW                   EM
SSB                    0
LB                  0.30 Hz
GB                     0
PC                  1.00
```

Table 2, Entry 5



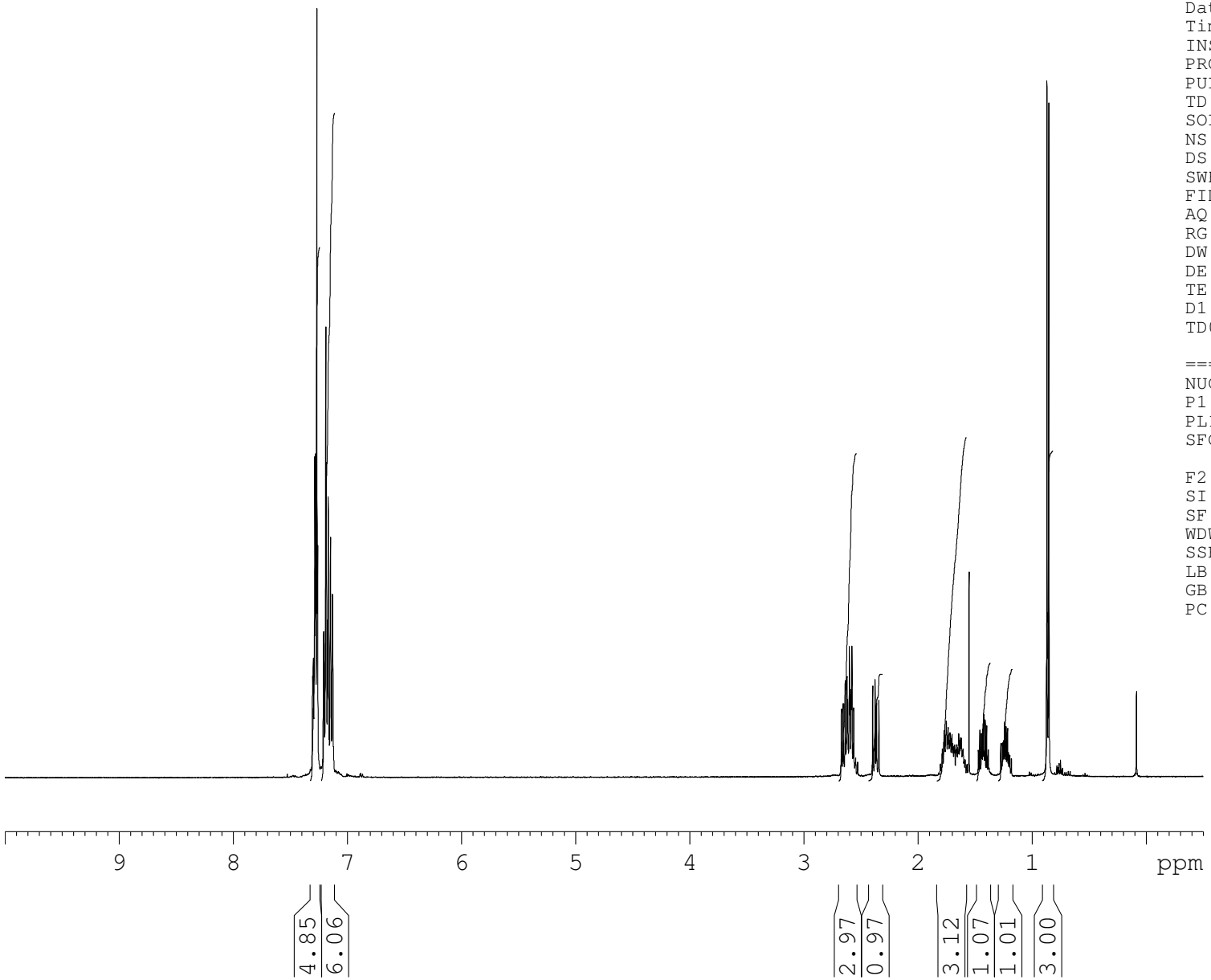

```
Current Data Parameters
NAME         Bun-2258-C1
EXPNO                  1
PROCNO                 1

F2 - Acquisition Parameters
Date_           20070327
Time               20.08
INSTRUM            spect
PROBHD   5 mm BBO BB-1H
PULPROG             zg30
TD                 65536
SOLVENT            CDCl3
NS                    16
DS                     2
SWH             8278.146 Hz
FIDRES          0.126314 Hz
AQ              3.9584243 sec
RG                 287.4
DW                60.400 usec
DE                 6.00 usec
TE                293.2 K
D1           1.00000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                  1H
P1                 15.07 usec
PL1                 0.00 dB
SFO1         400.1324710 MHz

F2 - Processing parameters
SI                 65536
SF           400.1300181 MHz
WDW                   EM
SSB                    0
LB                  0.30 Hz
GB                     0
PC                  1.00
```

Table 2, Entry 6



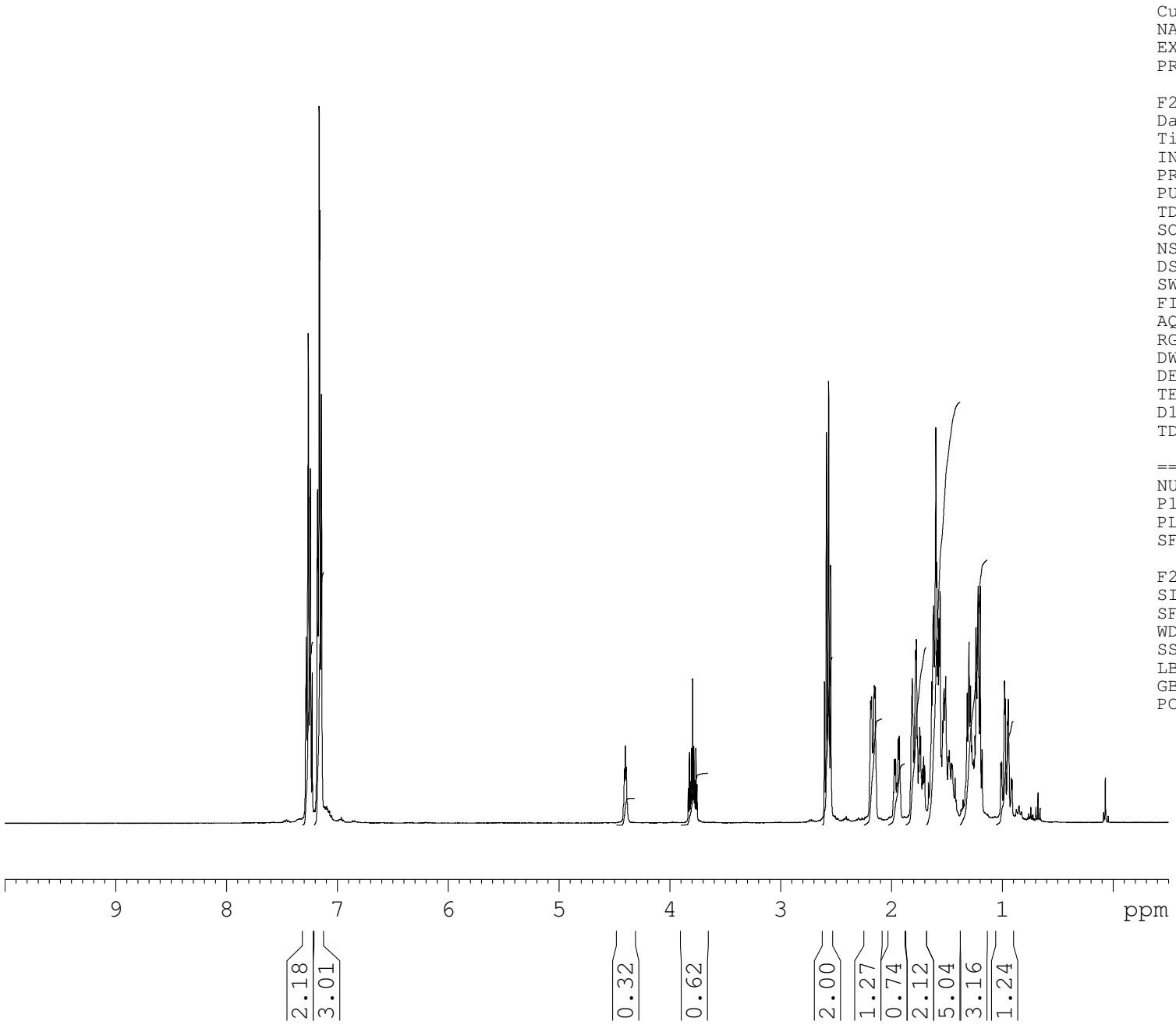

```
Current Data Parameters
NAME         Bun-2230-G1
EXPNO                100
PROCNO                 1

F2 - Acquisition Parameters
Date_           20070312
Time               20.08
INSTRUM            spect
PROBHD   5 mm QNP 1H/13
PULPROG             zg30
TD                 65536
SOLVENT            CDCl3
NS                    16
DS                     2
SWH             8278.146 Hz
FIDRES          0.126314 Hz
AQ             3.9584243 sec
RG                 287.4
DW                60.400 usec
DE                 6.00 usec
TE                293.2 K
D1           1.00000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                  1H
P1                14.00 usec
PL1                0.00 dB
SFO1         400.1324710 MHz

F2 - Processing parameters
SI                 65536
SF          400.1300058 MHz
WDW                   EM
SSB                    0
LB                 0.30 Hz
GB                     0
PC                 1.00
```

Table 2, Entry 7



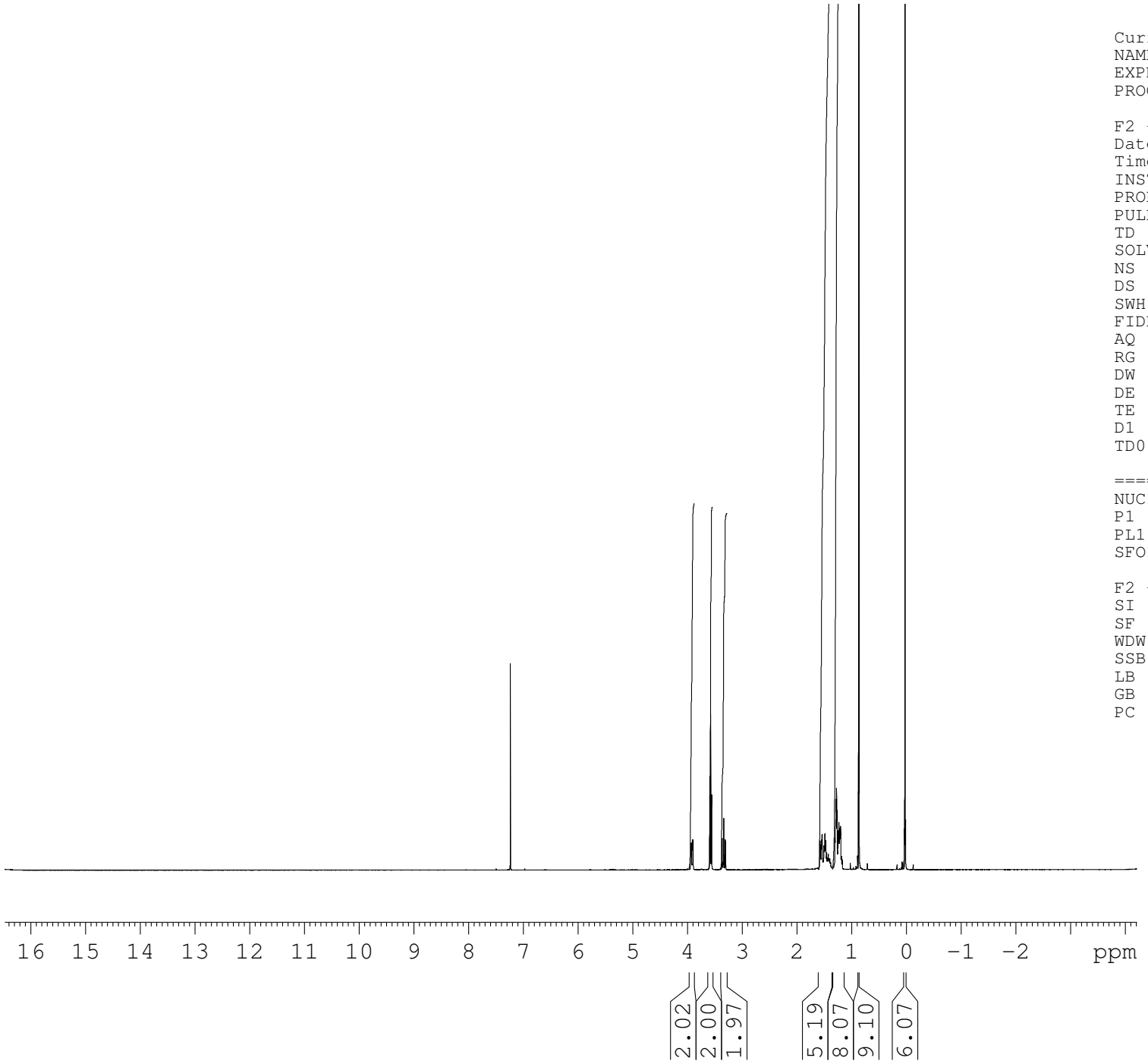

```
Current Data Parameters
NAME         Bun-2294-B2
EXPNO                100
PROCNO                 1

F2 - Acquisition Parameters
Date_           20070510
Time               15.23
INSTRUM            spect
PROBHD   5 mm QNP 1H/13
PULPROG             zg30
TD                 65536
SOLVENT            CDCl3
NS                    16
DS                     2
SWH             8278.146 Hz
FIDRES          0.126314 Hz
AQ              3.9584243 sec
RG                  90.5
DW                60.400 usec
DE                 6.00 usec
TE                294.2 K
D1             1.00000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                  1H
P1                14.00 usec
PL1                0.00 dB
SFO1         400.1324710 MHz

F2 - Processing parameters
SI                 65536
SF            400.1300220 MHz
WDW                   EM
SSB                    0
LB                 0.30 Hz
GB                     0
PC                 1.00
```

Table 2, Entry 8



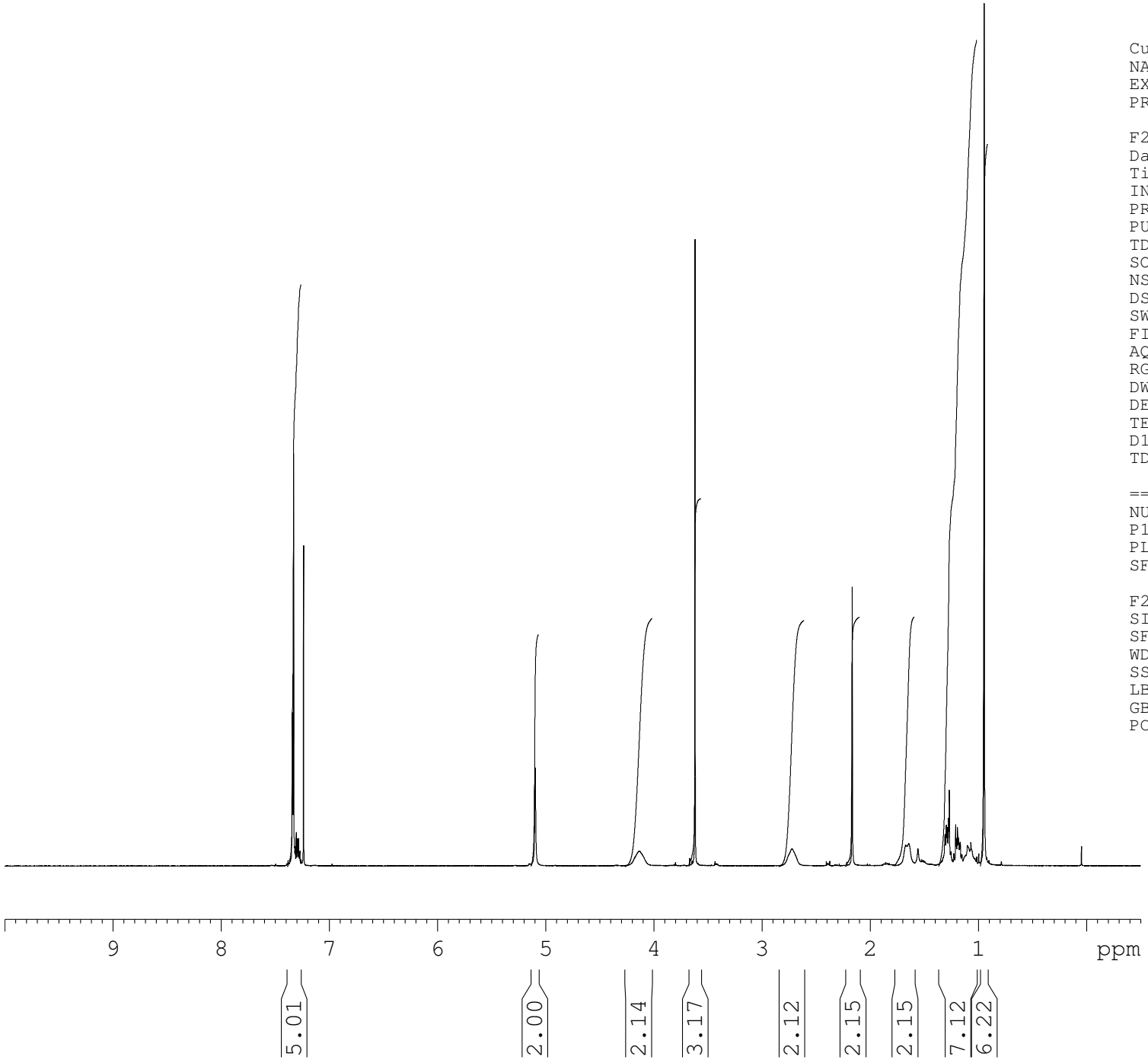

```
Current Data Parameters
NAME         Bun-2256-A1
EXPNO                100
PROCNO                 1

F2 - Acquisition Parameters
Date_           20070326
Time               21.14
INSTRUM            spect
PROBHD   5 mm QNP 1H/13
PULPROG             zg30
TD                 65536
SOLVENT            CDCl3
NS                    16
DS                     2
SWH             8278.146 Hz
FIDRES          0.126314 Hz
AQ             3.9584243 sec
RG                 322.5
DW                60.400 usec
DE                 6.00 usec
TE                292.2 K
D1           1.00000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                  1H
P1                 14.00 usec
PL1                 0.00 dB
SFO1         400.1324710 MHz

F2 - Processing parameters
SI                 65536
SF           400.1300177 MHz
WDW                   EM
SSB                    0
LB                  0.30 Hz
GB                     0
PC                  1.00
```

Table 2, Entry 9



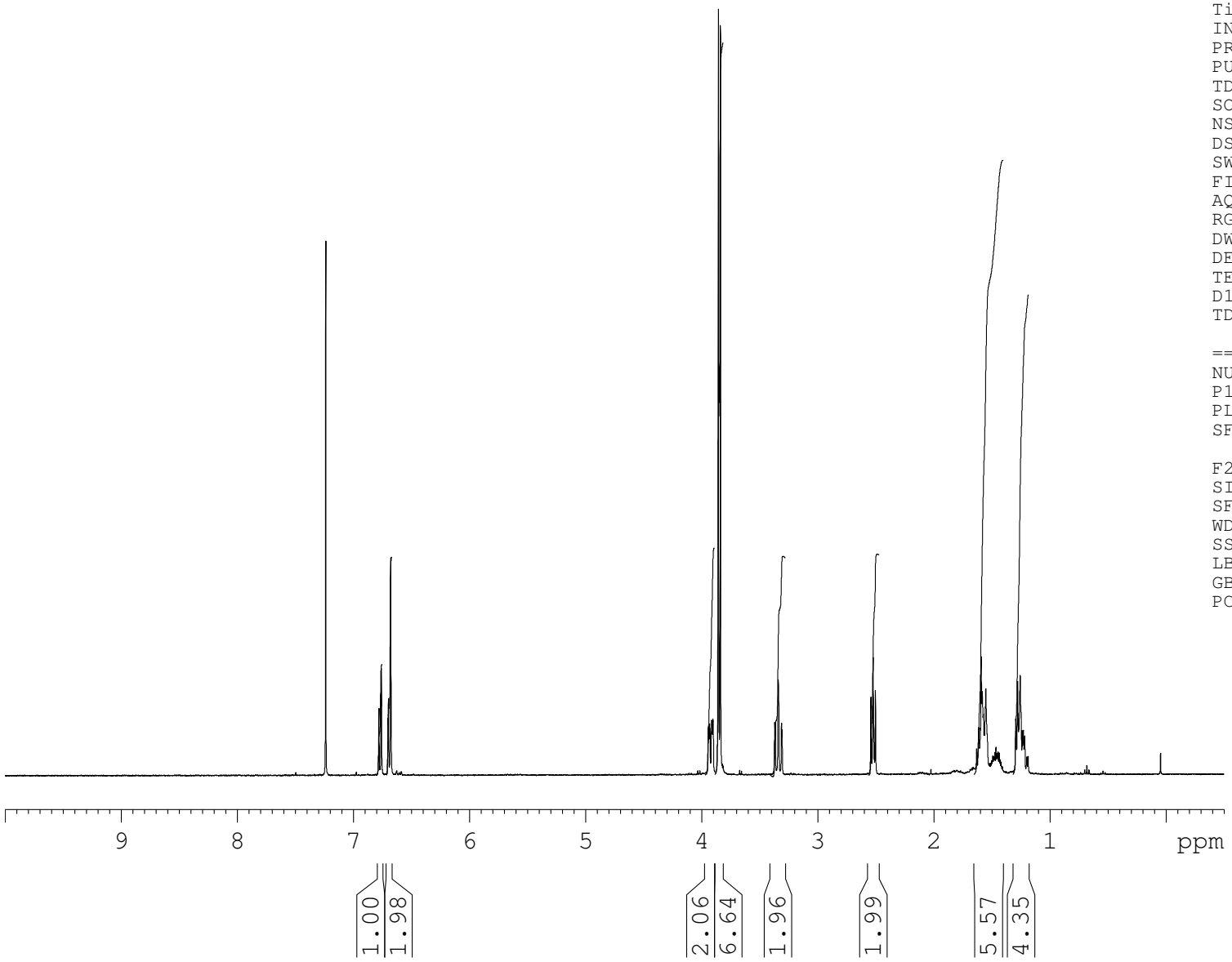

```
Current Data Parameters
NAME         Bun-2236-F1
EXPNO                  2
PROCNO                 1

F2 - Acquisition Parameters
Date_           20070404
Time               21.13
INSTRUM            spect
PROBHD   5 mm BBO BB-1H
PULPROG             zg30
TD                 65536
SOLVENT            CDCl3
NS                    16
DS                     2
SWH             8278.146 Hz
FIDRES          0.126314 Hz
AQ              3.9584243 sec
RG                   362
DW                60.400 usec
DE                 6.00 usec
TE                293.2 K
D1           1.00000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                  1H
P1                15.07 usec
PL1                0.00 dB
SFO1         400.1324710 MHz

F2 - Processing parameters
SI                 65536
SF           400.1300176 MHz
WDW                   EM
SSB                    0
LB                 0.30 Hz
GB                     0
PC                  1.00
```

Table 2, Entry 10



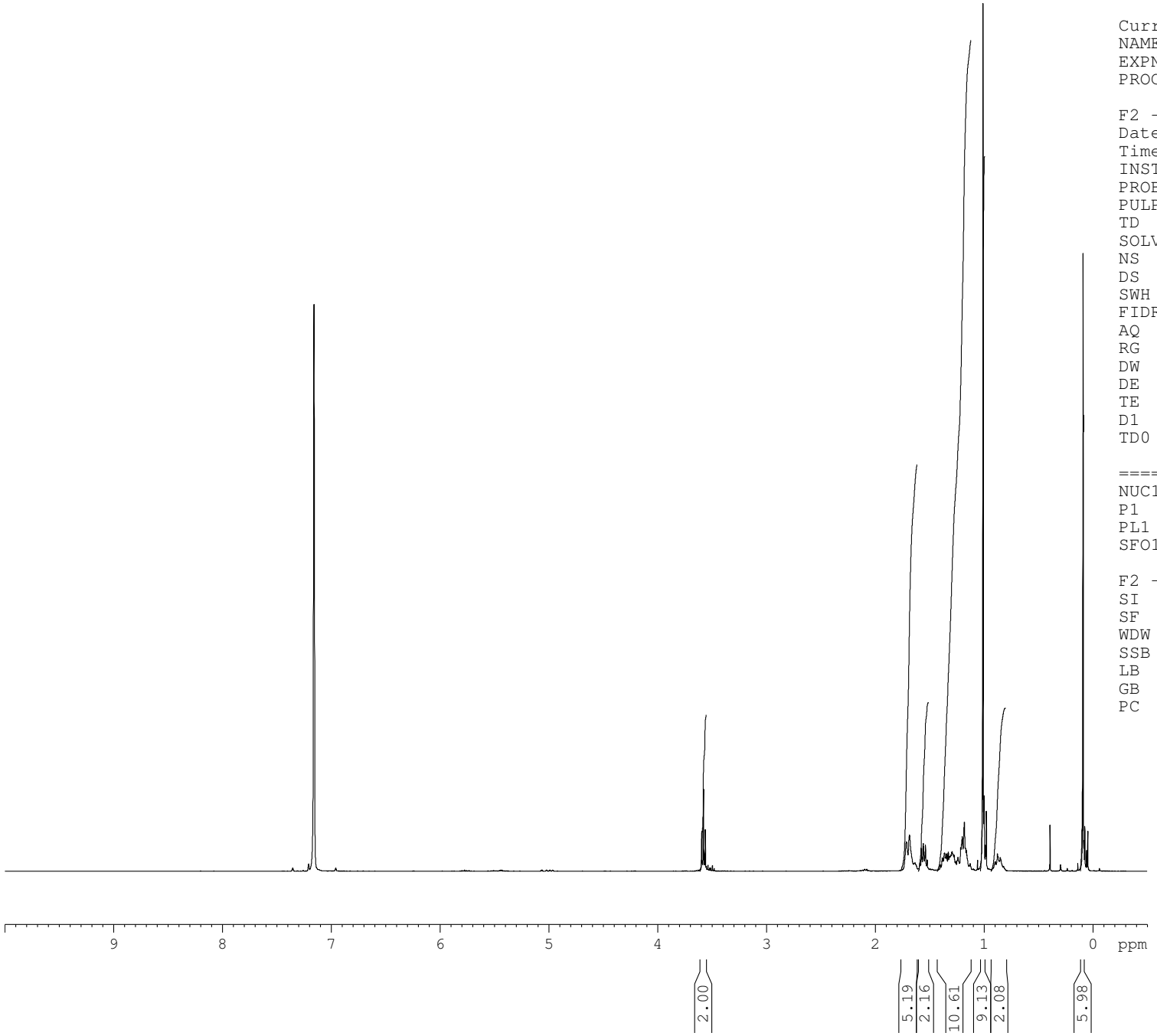

```
Current Data Parameters
NAME         Bun-2250-A3
EXPNO                100
PROCNO                 1

F2 - Acquisition Parameters
Date_           20070324
Time               22.16
INSTRUM            spect
PROBHD   5 mm QNP 1H/13
PULPROG             zg30
TD                 65536
SOLVENT            CDCl3
NS                    16
DS                     2
SWH             8278.146 Hz
FIDRES          0.126314 Hz
AQ              3.9584243 sec
RG                 287.4
DW                60.400 usec
DE                 6.00 usec
TE                 293.2 K
D1             1.00000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                  1H
P1                14.00 usec
PL1                0.00 dB
SFO1         400.1324710 MHz

F2 - Processing parameters
SI                 65536
SF           400.1300175 MHz
WDW                   EM
SSB                    0
LB                 0.30 Hz
GB                     0
PC                 1.00
```

Table 2, Entry 11



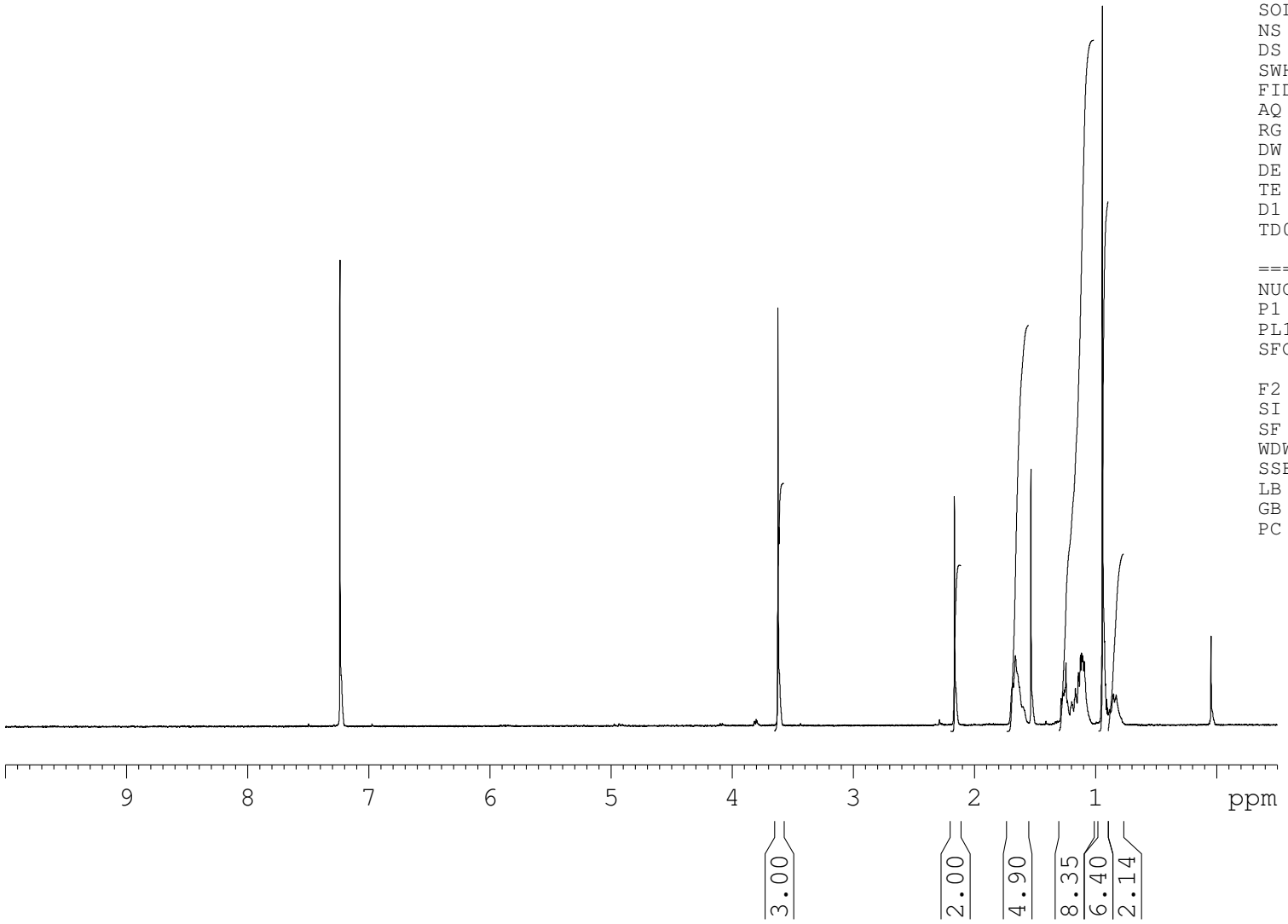

```
Current Data Parameters
NAME      Bun-2244-B-1H
EXPNO                1
PROCNO               1

F2 - Acquisition Parameters
Date_          20070418
Time              17.35
INSTRUM           spect
PROBHD   5 mm BBO BB-1H
PULPROG            zg30
TD                65536
SOLVENT            C6D6
NS                   16
DS                    2
SWH            8278.146 Hz
FIDRES         0.126314 Hz
AQ            3.9584243 sec
RG                  128
DW                60.400 usec
DE                 6.00 usec
TE                294.2 K
D1           1.00000000 sec
TD0                  1

======== CHANNEL f1 ========
NUC1                1H
P1                15.07 usec
PL1                0.00 dB
SFO1        400.1324710 MHz

F2 - Processing parameters
SI                65536
SF          400.1300446 MHz
WDW                 EM
SSB                  0
LB                0.30 Hz
GB                   0
PC                1.00
```

Table 2, Entry 12



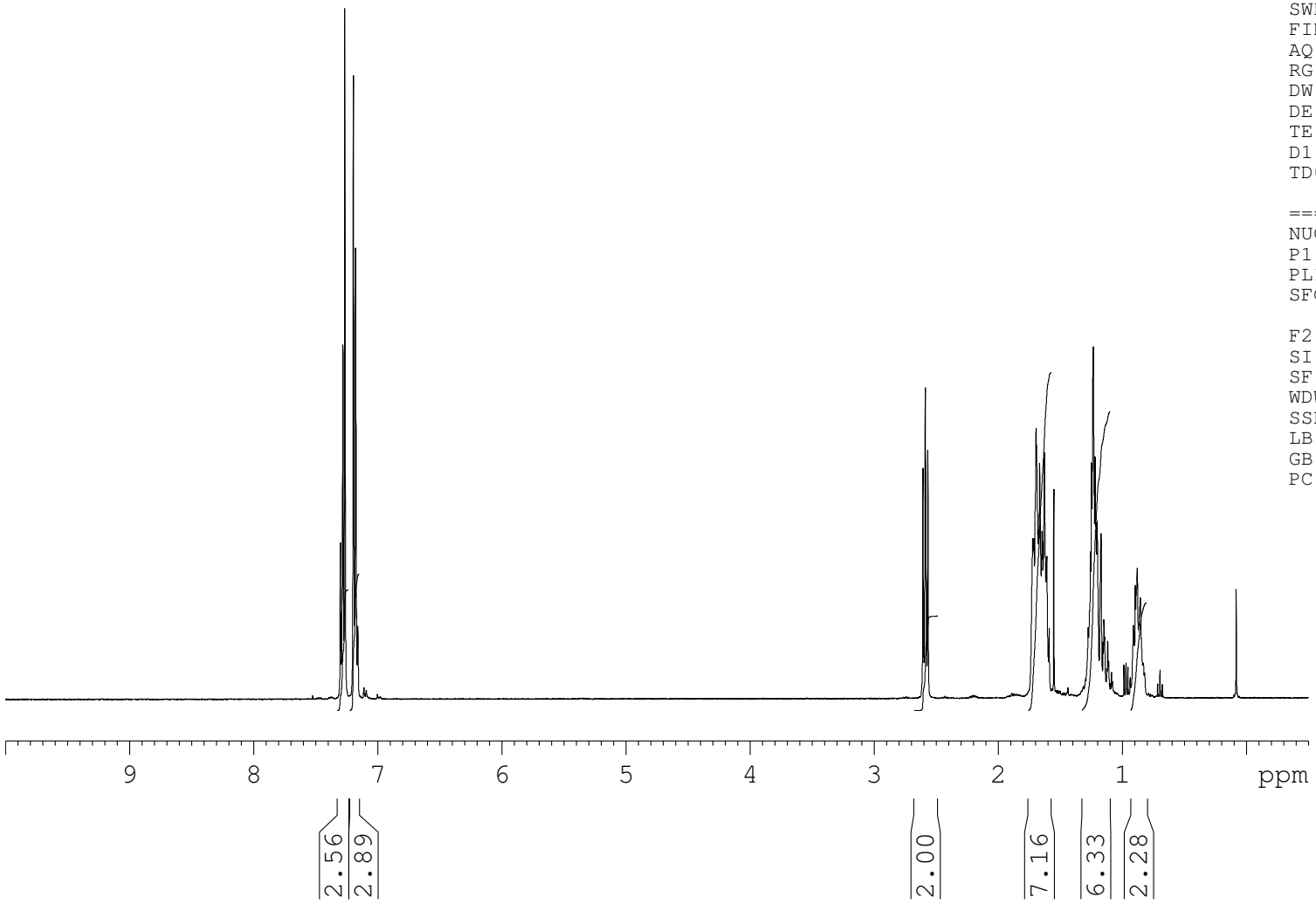

```
Current Data Parameters
NAME         Bun-2236-A1
EXPNO                100
PROCNO                 1

F2 - Acquisition Parameters
Date_           20070314
Time               20.51
INSTRUM            spect
PROBHD   5 mm QNP 1H/13
PULPROG             zg30
TD                 65536
SOLVENT            CDCl3
NS                    16
DS                     2
SWH             8278.146 Hz
FIDRES          0.126314 Hz
AQ             3.9584243 sec
RG                 287.4
DW                60.400 usec
DE                 6.00 usec
TE                292.2 K
D1           1.00000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                  1H
P1                14.00 usec
PL1                0.00 dB
SFO1         400.1324710 MHz

F2 - Processing parameters
SI                 65536
SF           400.1300182 MHz
WDW                   EM
SSB                    0
LB                 0.30 Hz
GB                     0
PC                  1.00
```

Table 3, Entry 1



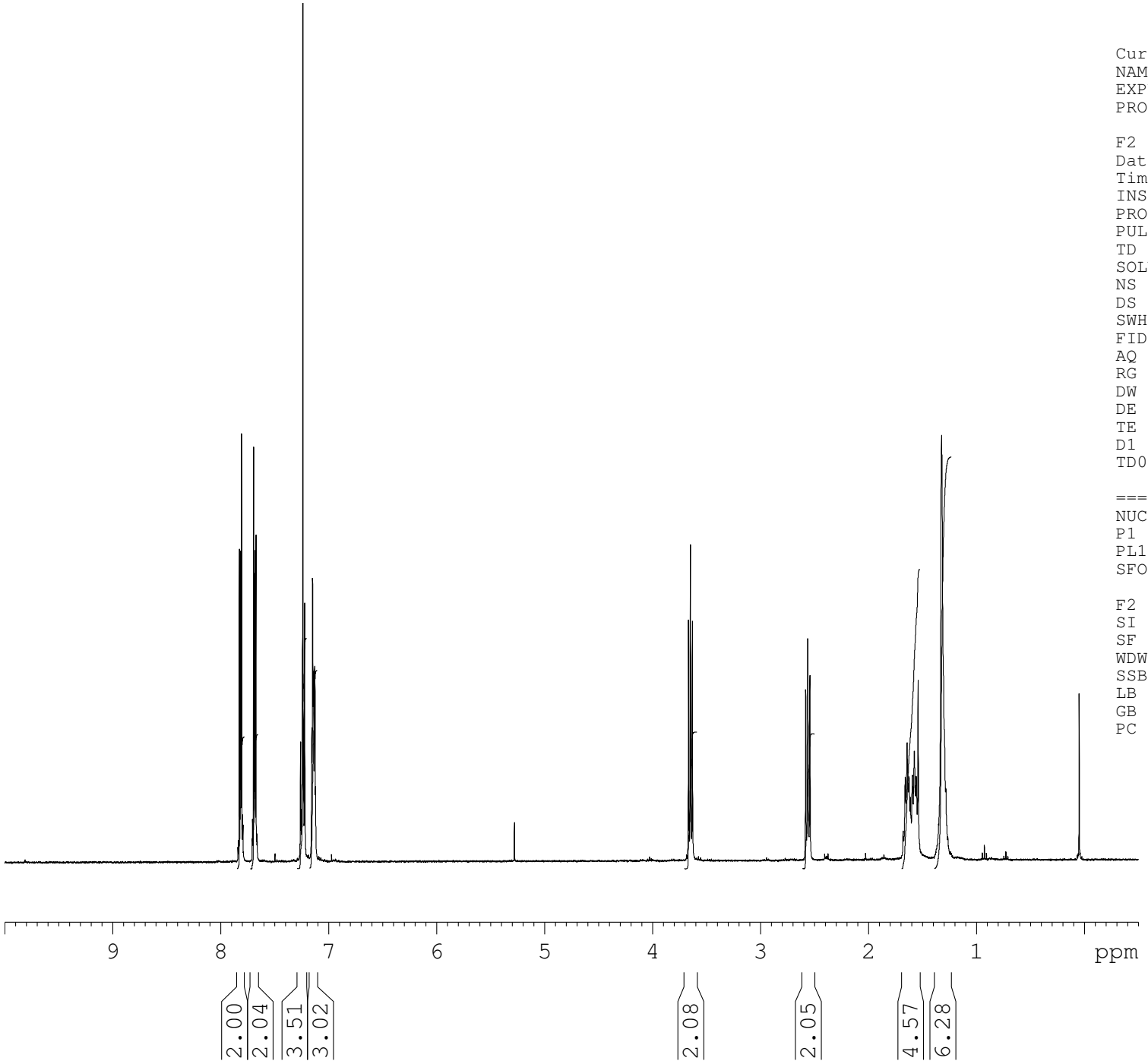

```
Current Data Parameters
NAME         Bun-2230-O1
EXPNO                100
PROCNO                 1

F2 - Acquisition Parameters
Date_           20070312
Time               19.54
INSTRUM            spect
PROBHD   5 mm QNP 1H/13
PULPROG             zg30
TD                 65536
SOLVENT            CDCl3
NS                    16
DS                     2
SWH             8278.146 Hz
FIDRES          0.126314 Hz
AQ             3.9584243 sec
RG                 322.5
DW                60.400 usec
DE                 6.00 usec
TE                293.2 K
D1           1.00000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                  1H
P1                 14.00 usec
PL1                 0.00 dB
SFO1         400.1324710 MHz

F2 - Processing parameters
SI                 65536
SF           400.1300063 MHz
WDW                   EM
SSB                    0
LB                  0.30 Hz
GB                     0
PC                  1.00
```

Table 3, Entry 2




```
Current Data Parameters
NAME         Bun-2274-A1
EXPNO                100
PROCNO                 1

F2 - Acquisition Parameters
Date_           20070410
Time               15.39
INSTRUM            spect
PROBHD   5 mm QNP 1H/13
PULPROG             zg30
TD                 65536
SOLVENT            CDCl3
NS                    16
DS                     2
SWH             8278.146 Hz
FIDRES          0.126314 Hz
AQ              3.9584243 sec
RG                 228.1
DW                60.400 usec
DE                 6.00 usec
TE                293.2 K
D1           1.00000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                  1H
P1                14.00 usec
PL1                0.00 dB
SFO1        400.1324710 MHz

F2 - Processing parameters
SI                 65536
SF          400.1300176 MHz
WDW                   EM
SSB                    0
LB                 0.30 Hz
GB                     0
PC                 1.00
```

Table 3, Entry 3