A Concise Approach to the Core Structures of Pinnaic Acid and Halichlorine

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Supporting Information:

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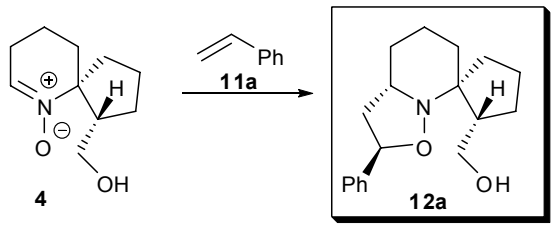
General. All reactions were performed under a nitrogen atmosphere using oven-dried glassware. All solvents were dried by distillation from calcium hydride (CH_2Cl_2, DMF, toluene) or sodium-benzophenone (THF and diethyl ether). Flash chromatography was performed using Scharlau 60 (230-400 mesh ASTM) silica gel Thin layer chromatography was performed on Merck silica gel 60 F_{254} plates. Melting points were measured by a Reicher-Kofler block and are uncorrected. IR spectra were recorded using a Perkin-Elmer Spectrum 1000 Fourier-Transform IR spectrometer. NMR spectra were recorded using a Bruker Avance 300 Spectrometer or a Bruker DRX 400 Spectrometer. ^{1}H NMR chemical shifts are reported in parts per million (ppm) relative to the tetramethylsilane peak (δ0.00 ppm). ^{1}H NMR values are reported as chemical shift δ, relative integral, multiplicity, (s, singlet; d, doublet; t, triplet; q, quartet; quintet; m, multiplet), coupling constant (J)$_2$ and assignment. Coupling constants were taken directly from the spectra. Assignments were made with the aid of DEPT, COSY, HSQC, HMBC and NOESY experiments. The ^{1}H and ^{13}C NMR spectra of compounds **32** and **34** are complicated by the presence of a mixture of diastereoisomers. Resonances for individual diastereomers are denoted by asterisks. Low resolution and accurate mass data were recorded on a VG70SE spectrometer operating at a nominal accelerating voltage of 70 eV. Ionisation was effected using electron impact (EI^+), chemical ionisation (CI^+) using ammonia as a carrier gas, or fast atom bombardment (FAB^+) using 3-nitrobenzylalcohol as the matrix. Major and significant fragments are quoted in the form x (y), where x is the mass to charge ratio (m/z) and y is the percentage abundance relative to the base peak (100%).

Experimental Procedures



(1*S,2*S**,2'*R**,3a'*S**)-2-hydroxymethyl-2'-phenylhexahydrospiro[cyclopentane-1,7'-isoxazolo[2,3-*a*]pyridine] 12a.**

A solution of styrene **11a** (2.60 g, 25.3 mmol) in toluene (30 mL) was added to a stirred solution of nitrone **4** (1.10 g, 7.94 mmol) in toluene (45 mL) at 0 °C. The reaction mixture was stirred under reflux for 13 h then cooled to room temperature and concentrated. The crude residue was purified by column chromatography with 30% ethyl acetate-hexane as eluent to afford isoxazolidine **12a** (1.40 g, 64%) as a colourless solid. Recrystallisation of **12a** from diethyl ether and hexane yielded crystals suitable for X-ray crystallography.

Mp 69.5–73.2 °C.

ν_{max} (solid)/cm^{-1} 3415, 2939, 2875, 1643, 1449.

δ_H (300 MHz; $CDCl_3$; Me_4Si) 1.47-1.70 (8H, m, H-3$_a$, H-4', H-5$_a$, H-5', H-6'), 1.70-1.90 (3H, m, H-3$_b$, H-4$_a$, H-5$_b$), 2.14-2.25 (2H, m, H-2, H-4$_b$), 2.25-2.37 (1H, m, H-3'$_a$), 2.37-2.47 (1H, m, 3'$_b$-H), 3.54-3.64 (1H, m, H-3a'), 3.66-3.72 (2H, m, C*H*$_2$OH), 5.09 (1H, dd, *J* = 8.4, 6.6 Hz, H-2'), 7.19-7.37 (5H, m, Ar).

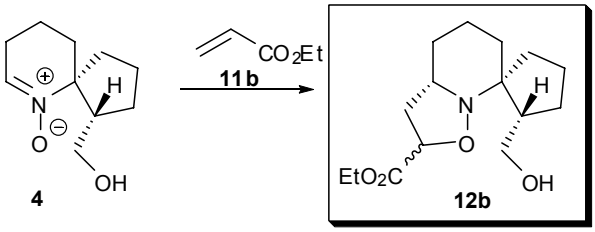
δ_c (75 MHz; $CDCl_3$; Me_4Si) 19.1 (CH_2), 21.3 (CH_2), 26.5 (CH_2), 27.7 (CH_2), 29.6 (CH_2), 37.8 (CH_2), 44.4 (CH_2), 45.5 (CH), 58.4 (CH), 65.4 (CH_2), 69.7 (C), 76.4 (CH), 125.3 (CH,), 127.0 (CH), 128.4 (CH), 144 (C).

m/z (EI) 287.1885 (M^+ $C_{18}H_{25}NO_2$ requires 287.1884). 287 (M^+, 49%). 270 (30), 228 (79), 215 (100), 143 (21), 130 (19), 77 (24), 41 (34).

Crystal structure determination of isoxazolidine 12a

Crystal data. $C_{18}H_{25}NO_2$, *M* = 287.39, monoclinic, *a* = 7.0366(1), *b* = 20.6330(3), *c* = 10.9160(1) Å, β = 102.955(1)º, *U* = 1543.85(3), *T* = 84 K, space group $P2_1$, *Z* = 4, 7756 reflections measured, 4206 unique (R_{int} = 0.017) which were used in all calculations. The final *R* was 0.0872 (all data).

(1*S,2*S**,2'*S**,3*a*'*S**)-2-(hydroxymethyl)-2'-(ethoxycarbonyl)hexahydrospiro[cyclopentane-1,7'-isoxazolo[2,3-*a*]pyridine] 12b**



A solution of ethyl acrylate **11b** (0.89 mL, 8.18 mmol) in dichloromethane (1.0 mL) was added to a stirred solution of nitrone **4** (0.1 g, 0.55 mmol) in

dichloromethane (10 mL) at room temperature and the reaction mixture stirred at this temperature for 48 h. The reaction mixture was then concentrated and the crude residue purified by column chromatography using 15% ethyl acetate-hexane as eluent to afford isoxazolidine **12b** (0.15 g, 94%) as a white solid.

Mp 62.5–63.7°C.

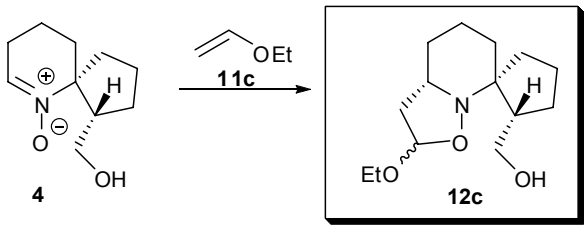
υ_{max} (solid)/cm^{-1}: 3442, 2936, 2876, 1731, 1444, 1193.

δ_H (300 MHz; $CDCl_3$; Me_4Si) 1.28 (3 H, t, *J* = 7.1 Hz, OCH_2CH_3), 1.35-1.70 (8H, m, H-3$_a$, H-4$_a$, H-4', H-5', H-6'), 1.70-1.98 (3H, m, H-3$_b$, H-4$_b$, H-5$_a$), 2.06-2.17 (1H, m, H-2), 2.17-2.29 (2H, m, H-3'$_a$, H-5$_b$), 2.42-2.55 (1 H, m, H-3'$_b$), 3.39-3.52 (1H, m, H-3a'), 3.52-3.70 (2H, m, CH_2OH), 4.22 (2H, q, *J* = 7.1 Hz, OCH_2CH_3), 4.45 (1H, dd, *J* = 9.6, 4.9, Hz, H-2').

δ_C (75 MHz; $CDCl_3$; Me_4Si) 14.0 (CH_3), 19.4 (CH_2), 21.4 (CH_2), 26.6 (CH_2), 28.3 (CH_2), 30.3 (CH_2), 38.6 (CH_2), 40.0 (CH_2), 44.9 (CH), 56.9 (CH), 61.1 (CH_2), 65.4 (CH_2), 69.1 (C), 72.4 (CH), 172.9 (C).

m/z (FAB) found MH^+ 284.1869, ($C_{15}H_{26}NO_4$ requires MH 284.1861). 284 (MH+, 74%), 211 (14), 154 (100), 136 (67), 107 (23), 89 (22), 77 (21).

(1*S,2*S**,3a'*S**)-2'-ethoxy-2-hydroxymethylhexahydrospiro[cyclopentane-1,7'-isoxazolo[2,3-*a*]pyridine] 12c**



A solution of ethyl vinyl ether **11c** (4.65 g, 64.5 mmol) in ethanol (10 mL) was added to a stirred solution of nitrone **4** (0.68 g, 3.69 mmol) in ethanol (25 mL) at 0 °C. The reaction mixture was stirred under reflux for 55 h then cooled to room temperature and concentrated. The crude residue was purified by column chromatography with 60% ethyl acetate-hexane as eluent to give isoxazolidine **12c** (0.75 g, 80%) as a colourless oil.

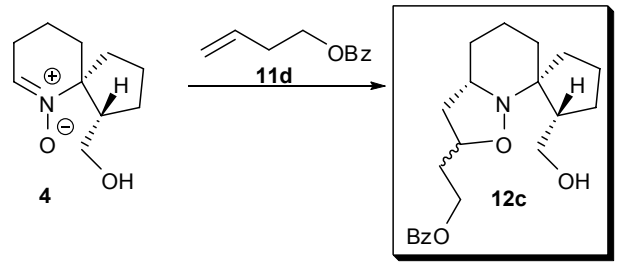
ν_{max} (film)/cm^{-1}: 3429, 2939, 2876, 1642, 1443.

δ_H (300 MHz; $CDCl_3$; Me_4Si) 1.20 (3H, t, *J* = 7.1 Hz, $CH_3CH_aH_bO$) 1.35-1.65 (8H, m, H-3$_a$, H-4$_a$, H-4', H-5', H-6'), 1.65-1.95 (3H, m, H-3$_b$, H-4$_b$, H-5$_a$), 2.0-2.20 (3H, m, H-3'$_a$, H-2, H-5$_b$), 2.20-2.30 (1H, m, H-3'$_b$), 3.35-3.55 (2H, m, H-3a', $CH_3CH_aH_bO$), 3.55-3.75 (3H, m, $CH_3CH_aH_bO$, CH_2OH), 5.05 (1H, dd, *J* = 2.7, 6.2 Hz, H-2').

δ_c (75 MHz; $CDCl_3$; Me_4Si) 15.2 (CH_3), 19.1 (CH_2), 21.5 (CH_2), 26.7 (CH_2), 28.1 (CH_2), 30.3 (CH_2), 38.6 (CH_2), 43.3 (CH_2), 45.2 (CH), 56.9 (CH), 63.0 (CH_2), 65.4 (CH_2), 69.0 (C), 99.5 (CH).

m/z (EI) 255.1835 (M^+ $C_{14}H_{25}NO_3$ requires M 255.1834), 255 (M^+, 35%), 238 (42), 210 (32), 196 (100), 183 (67), 166 (22), 111 (52), 98 (44), 96 (42), 83 (24), 55 (31), 41 (46).

(1*S,2*S**,3a'*S**)-2'-(2''-benzoyloxyethyl)-2-(hydroxymethyl)hexahydrospiro[cyclopentane-1,7'-isoxazolo[2,3-*a*]pyridine] 12d**



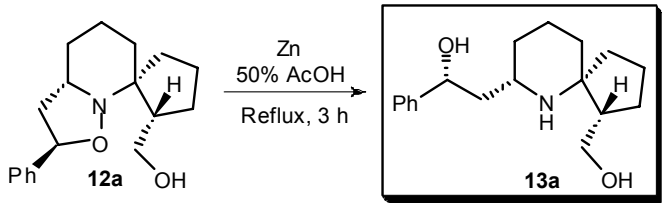
A solution of alkene **11d** (1.15g, 6.55 mmol) in toluene (2 mL) was added to a stirred solution of nitrone **4** (0.40 g, 2.18 mmol) in toluene (4 mL) at 0 °C. The reaction mixture was stirred under reflux for 8 h then cooled to room temperature and concentrated. The crude residue was purified by column chromatography with 50% diethyl ether-hexane as eluent to give the isoxazolidine **12d** (0.55g, 70%) as a colourless oil.

ν_{max} (film)/cm-1 3421, 2941, 1718, 1601, 1451, 1314, 1275.; δH (400 MHz; CDCl3; Me4Si) 1.31-2.20 (17 H, m, H-2, H- 3, H-3', H-1'', H-4, H-4', H-5, H-5', H-6'), 3.34- 3.51 (1 H, br s, H-3a'), 3.64 (2 H, d, J = 5.7 Hz, H-2''), 4.16- 4.29 (1 H, m, H-2'), 4.41 (2 H, t, J = 6.0 Hz, CH_2OH), 7.38- 7.47 (2 H, m, H-Ar), 7.50-7.58 (1 H, m, H-Ar), 8.00-8.07 (2 H, m, H-Ar).

δ_c (100 MHz; $CDCl_3$; Me_4Si) 19.2 (CH_2), 20.9 (CH_2), 26.6 (CH_2), 27.6 (CH_2), 30.8 (CH_2), 35.3 (CH_2), 37.8 (CH_2), 41.3 (CH_2), 45.1 (CH), 57.2 (CH), 62.2 (CH_2), 65.1 (CH_2), 69.2 (C), 71.7 (CH), 128.1, 129.3 (CH), 130.1 (C), 132.6 (CH), 166.3 (C).

m/z (EI) 359.2091 (M^+ $C_{21}H_{29}NO_4$ requires 359.2097). 369 (M^+, 41%), 342 (26), 300 (78), 287 (100), 105 (50), 77 (29), 41 (22) 55 (50), 41 (60).

(1*S,5*S**,7*S**)-1-hydroxymethyl-7-((1'*R**)-1'-hydroxy-1'-phenylethyl)-6-azaspiro[4.5]decane 13a.**



Zinc dust (0.29 g, 4.40 mmol) was added to a stirred solution of cycloadduct **12a** (0.12g, 0.4 mmol) in 50% aqueous acetic acid (2 mL) at room temperature. The reaction mixture was stirred under reflux for 3 h then a saturated aqueous solution of sodium bicarbonate was added. The aqueous phase was extracted with dichloromethane and the combined organic phases dried ($MgSO_4$) and concentrated. The crude residue was purified by column chromatography with 90% dichloromethane-methanol as eluent afforded azaspiro[4.5]decane **13a** (0.09g, 76%) as a white solid.

Mp 57.7-58.6 °C

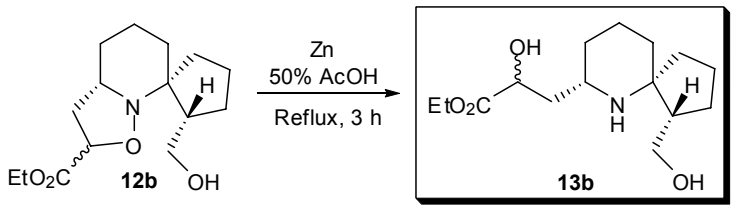
ν_{max} (film)/cm^{-1}: 3412, 2931, 1452.

δ_H (400 MHz; C_6D_6; Me_4Si) 0.70-0.85 (2H, m, H-8$_a$, H-10$_a$), 1.15-1.25 (1H, m, H-4$_a$), 1.25-1.46 (5H, m, H-2'$_a$, H-3, H-8$_b$, H-10$_b$), 1.46-1.67 (5H, m, H-2, H-4$_b$, H-9, 1.68-1.78 (1H, m, H-2'$_b$), 1.84-1.93 (1H, m, H-1), 3.00-3.12 (1H, m, H-7), 3.55-3.67 (2H, m, CH_2OH), 5.04 (1H, dd, J = 10.2, 2.2 Hz, H-1'), 7.00-7.30 (5H, m, H-Ar).

δ_c (75 MHz; $CDCl_3$; Me_4Si) 20.6 (CH_2), 21.4 (CH_2), 28.4 (CH_2), 33.3 (CH_2), 37.3 (CH_2), 41.7 (CH_2), 42.6 (CH), 47.3 (CH_2), 50.3 (CH), 64.0 (C), 65.3 (CH_2), 70.0 (CH), 126.1 (CH), 127.0 (CH), 128.4 (CH), 146.6 (C).

m/z (EI) 289.2039 (M^+ $C_{18}H_{27}NO_2$ requires 289.2041). 289 (M^+, 29%), 272 (16), 230 (48), 217 (58), 169 (100), 108 (66), 96 (82), 79 (44), 96 (42), 77 (30), 55 (32), 41 (26).

(1*S,5*S**,7S*)-1-hydroxymethyl-7-(2'-ethyloxycarbonyl-2'-hydroxyethyl)-6-azaspiro[4.5]decane (13b)**



Zinc dust (0.041 g, 1.76 mmol) was added to a stirred solution of cycloadduct **12a** (0.05g, 0.16 mmol) in 50% aqueous acetic acid (0.9 mL) at room temperature. The reaction mixture was stirred under reflux for 3 h then a saturated aqueous solution of sodium bicarbonate was added. The aqueous phase was extracted with dichloromethane and the combined organic phases dried ($MgSO_4$) and concentrated. The crude residue was purified by column chromatography with 90% dichloromethane-methanol as eluent afforded azaspiro[4.5]decane **13a** (0.03g, 70%) as a yellow oil.

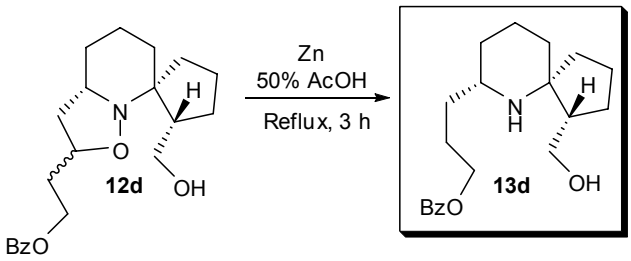
υ_{max} (film)/cm^{-1}: 3368, 2933, 1732, 1443, 1210.

δ_H (300 MHz; $CDCl_3$; Me_4Si) 1.29 (3H, t, J = 7.1 Hz, CH_3CH$_2$O), 1.29-1.31 (2H, m, H-8$_a$, H-10$_a$), 1.55-1.97 (12H, m, H-1', H-2, H-3, H-4, H-8$_b$, H-9, H-10$_b$), 2.15-2.26 (1H, br s, H-1), 3.20-3.27 (1H, m, H-7), 3.67-3.78 (2H, m, CH_2OH), 4.23 (2H, q, J = 7.1 Hz, CH$_3$CH_2O), 4.48 (1H, dd, J = 7.4, 3.9 Hz, H-2').

δ_C (75 MHz; $CDCl_3$; Me_4Si) 14.1 (CH_3), 20.0 (CH_2), 20.5 (CH_2), 27.8 (CH_2), 31.8 (CH_2), 35.9 (CH_2), 39.2 (CH_2), 40.2 (CH_2), 42.1 (CH), 51.4 (CH), 61.3 (CH_2), 64.4 (CH_2), 64.9 (C), 68.5 (CH), 174.5 (C).

m/z (EI) 285.1943 (M^+ $C_{15}H_{27}NO_4$ requires 285.1940). 285 (M^+, 22%), 268 (37), 226 (89), 213 (53), 150 (90), 96 (100), 41 (44).

(1*S,5*S**,7S*)-1-Hydroxymethyl-7-(2'-hydroxybenzoyloxybutyl)-6-azaspiro[4.5]decane (13d)**



Zinc dust (0.16 g 2.42 mmol) was added to a stirred solution of cycloadduct **12a** (0.08g, 0.22 mmol) in 50% aqueous acetic acid (1.1 mL) at room temperature. The reaction mixture was stirred under reflux for 3 h then a saturated aqueous solution of sodium bicarbonate was added. The aqueous phase was extracted with dichloromethane and the combined organic phases dried ($MgSO_4$) and concentrated. The crude residue was purified by column chromatography with 90% dichloromethane-methanol as eluent afforded azaspiro[4.5]decane **13a** (0.065g, 81%) as a yellow oil.

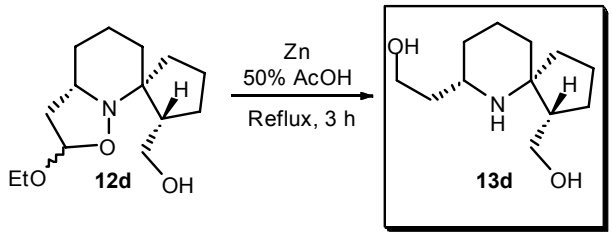
υ_{max} (film)/cm^{-1}: 3418, 2934, 1714, 1648, 1451, 1277.

δ_H (300 MHz; $CDCl_3$; Me_4Si) 1.08-1.23 (2H, m, H-8_a, H-10_a), 1.38-1.60 (7H, m, H-1', H-2_a, H-4_a, H-8_b, H-9), 1.67-1.95 (7H, m, $BzOCH_2CH_2$, H-2_b, H-3, H-4_b, H-10_b), 2.10-2.27 (1H, m, H-1), 3.08-3.25 (1H, m H-7), 3.69 (2H, d, J = 6 Hz, CH_2OH), 4.01-4.15 (1H, m, H-2'), 4.46 (2H, t, J = 6.5 Hz, $BzOCH_2CH_2$), 7.30-7.48 (2H, m, H-Ar), 7.48-7.62 (1H, m, H-Ar), 7.92-8.10 (2H, m, H-Ar).

δ_C (75 MHz; $CDCl_3$; Me_4Si) 19.7 (CH_2), 20.7 (CH_2), 27.5 (CH_2), 32.1 (CH_2), 36.1 (CH_2), 36.4 (CH_2), 40.2 (CH_2), 41.6 (CH), 43.2 (CH_2), 50.4 (CH), 62.2 (CH_2), 64.4 (C), 64.7 (CH_2), 64.7 (CH), 128.2 (CH), 129.4 (CH), 130.2 (C), 132.7 (CH), 166.6 (C).

m/z (EI) 361.2251 (M^+ $C_{21}H_{31}NO_4$ requires 361.2253). 361 (M^+, 30%), 344 (29), 302 (77), 289 (70), 169 (65), 105 (79), 96 (100), 77 (46), 57 (58), 41 (48).

(1*S,5*S**,7*S**)-1-hydroxymethyl-7-(1'-hydroxyethyl)-6-azaspiro[4.5] decane 14.**



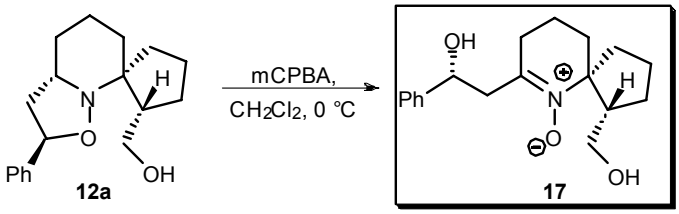
Palladium hydroxide (0.04 g, 0.06 mmol) was added to a stirred solution of compound **12c** (0.08 g, 0.31 mmol) in methanol (30 mL) at room temperature. The suspension was stirred under hydrogen for 48 h, filtered through a pad of Celite® and the filtrate concentrated. The crude residue was purified by column chromatography with 30% ethanol-dichloromethane as eluent to afford azaspiro[4.5]decane **14** (0.31 g, 44%) as an orange oil.

ν_{max} (film)/cm^{-1}: 3304, 2930, 2873, 1573, 1441, 1332; δ_H (300 MHz; C_6D_6; Me_4Si) 1.05-1.20 (2H, m, H-8_a, H-10_a), 1.44-1.57 (4H, m, H- 2'$_a$, H-4_a, H-9), 1.57-1.88 (8H, m, H-2, H-2'$_b$, H-3, H-4_b, H-8_b, H-10_b), 2.12-2.29 (1H, m, H-1), 2.95-3.12

(1H, m, H-7), 3.57-3.66 (1H, m, CH_aH$_b$OH), 3.66-3.86 (3H, m, CH$_a$$H_b$OH, H-1'). δ_c (75 MHz; $CDCl_3$; Me_4Si) 19.9 (CH_2), 20.9 (CH_2), 21.3 (CH_2) 32.8 (CH_2), 36.5 (CH_2), 38.6 (CH_2), 40.4 (CH_2), 42.0 (CH), 52.6 (CH), 60.6 (CH_2), 64.0 (C), 64.5 (CH_2).

m/z (EI) 213.1729 (M^+ $C_{12}H_{23}NO_2$ requires 213.1728), 213 (M^+, 17%), 196 (38), 168 (23), 15 (100), 141 (60), 110 (36), 96 (61), 67 (14), 55 (21), 41 (27), 39 (9).

(1*S,5*S**)-7-((1'*S**)-1'-hydroxy-1'-phenylethyl)-1-(hydroxymethyl)-6-azaspiro[4.5]dec-6-ene-6-oxide 17**



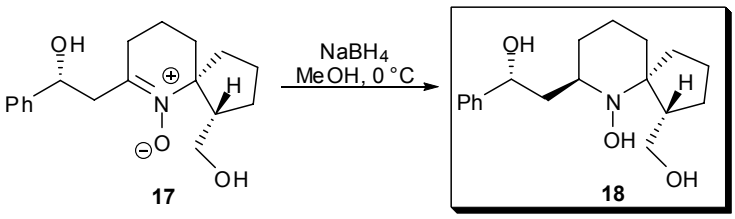
A solution of *m*-CPBA (0.09 g, 0.05 mmol) in dichloromethane (4 mL) was added dropwise over 1 h to a stirred solution of isoxazolidine **12a** (0.1 g, 0.34 mmol) in dichloromethane (2 mL) at 0 ºC. The reaction mixture was stirred for a further 10 min at the same temperature and a mixture of saturated aqueous sodium thiosulfate:sodium hydrogen carbonate (1:1, 20 mL) was added. The aqueous phase was extracted with dichloromethane (3 x 10 mL) and the combined organic extracts dried ($MgSO_4$) and concentrated. The crude residue was purified by column chromatography with 5% methanol-dichloromethane as eluent to afford nitrone **17** (0.09 g, 85%) as a colourless oil.
υ_{max} (film)/cm^{-1}: 3254, 2938, 2872, 1713, 1602, 1449, 1133, 1116, 925.

δ_H (300 MHz; $CDCl_3$; Me_4Si) 1.55-1.62 (1H, m, H-3$_a$), 1.62-1.71 (2H, m, H-9), 1.71-1.87 (3H, m, H-2$_a$, H-4$_a$, H-10$_a$), 1.87-2.00 (2H, m, H-1, H-10$_b$), 2.00-2.08 (1H, m, H-3$_b$), 2.14-2.25 (1H, m, H-2$_b$), 2.32-2.44 (2H, m, H-8), 2.68-2.77 (1H, m, H-4$_b$), 2.81 (1H, dd, J = 12.7, 3.4 Hz, H-2'$_a$), 3.07 (1H, dd, J = 12.7, 9.1 Hz, H-2'$_b$), 3.67-3.82 (2H, m, CH_2OH), 5.22 (1H, dd, J = 9.1, 3.4 Hz, H-1'), 7.22-7.28 (1H, m, H-Ar), 7.31-7.34 (2H, m, H-Ar), 7.39-7.41 (2H, m, H-Ar).

δ_C (75 MHz; $CDCl_3$; Me_4Si) 16.2 (CH_2), 24.1 (CH_2), 28.7 (CH_2), 31.9 (CH_2), 37.3 (CH_2), 39.0 (CH_2), 43.6 (CH_2), 53.2 (CH), 60.9 (CH_2), 72.7 (CH), 77.3 (C), 125.2 (CH), 127.3 (CH), 128.3 (CH), 144.5 (C), 154.0 (C).

m/z (FAB) 304.1910 (MH^+ $C_{18}H_{26}NO_3$ requires 304.1912). 304 (MH^+, 18%), 289 (12), 154 (100), 136 (66), 107 (22), 89 (20), 77 (21).

(1*S,5*S**,7*R**)-7-((1'*S**)-1'-hydroxy-1'-phenylethyl)-1-(hydroxymethyl)-6-azaspiro[4.5] decan-6-ol 18**



Sodium borohydride (0.08 g, 2.24 mmol) was added in one portion to a stirred solution of nitrone **17** (0.1 g, 0.35 mmol) in methanol (5 mL) at 0 ºC. The reaction mixture was then stirred at room temperature for a further 15 min and concentrated. The residue was diluted with ethyl acetate (20 mL) and brine (20 mL) and the aqueous phase was further extracted with ethyl acetate (3 x 10 mL). The combined organic extracts were dried ($MgSO_4$) and concentrated and the crude residue purified by column chromatography with 5 % methanol-dichloromethane as eluent to give *N*-hydroxyazaspiro[4.5]decane **18** (0.09 g, 90%) as a white solid. Recrystallisation of the **18** from diethyl ether and hexane yielded crystals suitable for X-ray crystallography.

Mp 129.8-131.7 ºC.

υ_{max} (film)/cm^{-1}: 3444, 2959, 2930, 2860, 1460, 1274, 1123.

δ_H (300 MHz; $CDCl_3$; Me_4Si) 1.07-1.21 (2H, m, H-2_a, H-8_a), 1.21-1.28 (1H, m, H-10_a), 1.35 (1H, t, *J* =14.3, 1.8 Hz, H-$2'_a$), 1.47-1.73 (5H, m, 3-H, H-4_a, H-9), 1.73-1.89 (2H, m, H-2_b, H-8_b), 1.89-2.05 (3H, m, H-1, H-4_b, H-10_b), 2.26 (1H, dd, *J* = 14.3, 11.0 Hz, H-$2'_b$), 3.06 (1H, dd, *J* = 10.4, 2.7 Hz, C*H*$_a$$CH_b$OH), 3.04-3.18 (1H, m, H-7), 3.67 (1H, d, *J* = 10.4 Hz, CH_aC*H*$_b$OH), 4.90 (1H, dd, *J* = 11.0, 1.8, H-1'), 7.17-7.26 (1H, m, H-Ar), 7.26-7.36 (2H, m, H-Ar), 7.36-7.45 (2H, m, H-Ar).

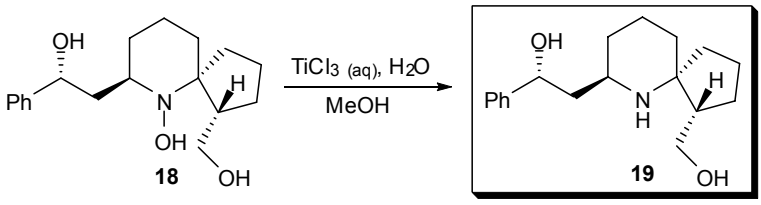
δ_C (75 MHz; $CDCl_3$; Me_4Si) 20.3 (CH_2), 20.9 (CH_2), 23.9 (CH_2), 26.3 (CH_2), 26.7 (CH_2), 29.6 (CH_2), 39.5 (CH_2), 50.0 (CH), 61.6 (CH), 64.5 (CH_2), 71.3 (C), 76.7 (CH), 125.7 (CH), 127.0 (CH), 128.2 (CH), 144.4 (C).

m/z (FAB) 306.2067 (MH $C_{18}H_{28}NO_3$ requires 306.2069). 306 (MH^+, 27%), 289 (13), 154 (100), 136 (67), 107 (23), 89 (20), 77 (20).

Crystal structure determination of azaspirodecane 18

Crystal data. $C_{18}H_{27}NO_3$, *M* = 305.41, triclinic, *a* = 6.2552(1), *b* = 10.5282(2), *c* = 13.4329(2) Å, α = 104.454(1)º, β = 101.796(1)º, γ = 97.250(1)º, *U* = 823.76(2), *T* = 84 K, space group P-1, *Z* = 2, 7744 reflections measured, 3258 unique (R_{int} = 0.034) which were used in all calculations. The final *R* was 0.074 (all data).

(1*S,5*S**,7*R**)-7-((1'*S**)-1'-hydroxy-1'-phenylethyl)-1-(hydroxymethyl)-6-hydroxyazaspiro[4.5] decane 19**



A solution of titanium trichloride (0.03 g, 0.37 mmol) in water (0.15 mL) was added dropwise to a stirred solution of hydroxylamine **18** (0.04 g, 0.12 mmol) in methanol (4 mL) at room temperature. The reaction mixture was stirred at room temperature for 3 h, dried ($MgSO_4$) and concentrated. The residue was purified by column chromatography with 5 % methanol-dichloromethane as eluent to afford azaspiro[4.5]decane **19** (0.03 g, 92%) as a yellow oil.

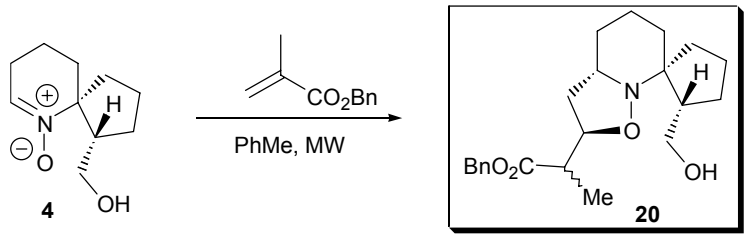
υ_{max} (film)/cm^{-1}: 3444, 2959, 2831, 2873, 1459, 1274, 1123, 1073.

δ_H (300 MHz; $CDCl_3$; Me_4Si) 1.47-1.63 (3H, m, H-3_a, H-4), 1.63-1.91 (7H, m, H-2, H-$2'_a$, H-3_b, H-8_a, H-9), 1.91-2.12 (4H, m, H-1, H-8_b, H-10), 2.52-2.65 (1H, m, H-$2'_b$), 3.32-3.45 (1H, m, H-7), 3.64 (1H, dd, *J* = 11.5, 6.3, Hz, C$H_a$$H_b$OH), 4.03 (1H, dd, *J* = 11.5, 3.1 Hz, $CH_a$$H_b$OH), 4.84 (1H, dd, 11.1, 2.6, Hz, H-1'), 7.18-7.42 (5H, m, H-Ar).

δ_C (75 MHz; $CDCl_3$; Me_4Si) 20.3 (CH_2), 22.1 (CH_2), 26.6 (CH_2), 29.0 (CH_2), 32.9 (CH_2), 33.1 (CH_2), 41.0 (CH_2), 49.5 (CH), 54.9 (CH), 60.7 (CH_2), 67.4 (C), 73.1 (CH), 125.8 (CH), 127.6 (CH), 128.4 (CH), 143.4 (C).

m/z (EI) 289.2042 (M, $C_{18}H_{27}NO_2$ requires 289.2041). 289 (M^+, 56%), 272 (25), 254 (37), 247 (38), 230 (74), 217 (88), 169 (100), 108 (88), 96 (80), 79 (68), 55 (60), 41 (55), 36 (43).

(1*S,2*S**,2'*S**,3*a*'*S**)-2-(hydroxymethyl)-2'-(1''-benzyloxycarbonylpropyl)hexahydrospiro[cyclopentane-1,7'-isoxazolo[2,3-*a*]pyridine] 20**



A 10 mL microwave reaction vial was charged with nitrone **4** (0.10 g, 0.54 mmol), dipolarophile **8** (0.21 g, 1.09 mmol) and toluene (3 mL). The vial was sealed with a cap containing a silicon septum, loaded into the cavity of a focussed microwave reactor (Discover® CEM, 300W) and heated at 165 °C for 50 min. (The following microwave reactor conditions were used: Power: 250 KW, ramp time: 5 min 30 sec). The reaction mixture was cooled to room temperature, concentrated under reduced pressure and the crude residue purified by column chromatography with 15% ethyl acetate-hexane as eluent to afford isoxazolidine **20** (0.17 g, 83%) as a yellow oil and as a separable 1:1 diastereomeric mixture.

Data for less polar diastereomer:

υ_{max} (film)/cm^{-1}: 3443, 2958, 2931, 2873, 1728, 1599, 1579, 1461, 1274.

δ_H (300 MHz; $CDCl_3$; Me_4Si) 1.25 (3H, d, *J* = 6.9 Hz, H-2''), 1.30-1.63 (8H, m, H-3$_a$, H-4$_a$, H-4', H-5', H-6'), 1.63-1.93 (3H, m, H-3$_b$, H-4$_b$, H-5$_a$), 1.93-2.08 (2H, m, H-3'$_a$, H-5$_b$), 2.08-2.12 (2H, m, H-2, H-3'$_b$), 2.66 (1H, dq, *J* = 7.6, 6.9, H-1''), 3.35-3.48 (1H, br d, *J* = 5.8 Hz, H-3*a*'), 3.56-3.65 (2H, m, C*H*$_2$OH), 4.08-4.15 (1H, dt, *J* = 7.6, 5.9 Hz H-2'), 5.11 (2H, d, *J* = 7.3 Hz, C*H*$_2$Ph), 7.30-7.38 (5H, m, H-Ar).

δ_C (75 MHz; $CDCl_3$; Me_4Si) 14.5 (CH_3), 19.2 (CH_2), 21.2 (CH_2), 26.4 (CH_2), 27.8 (CH_2), 30.2 (CH_2), 38.2 (CH_2), 40.0 (CH_2,), 45.3 (CH) (2C), 57.8 (CH), 65.4 (CH_2), 66.1 (CH_2), 69.2 (C), 76.0 (CH), 128.0 (CH), 128.1 (CH), 128.5 (CH), 135.9 (C), 174.3 (C).

m/z (EI) 373.2253 ($C_{22}H_{31}NO_4$ requires M^+ 373.2253). 373 (M^+, 31%), 356 (22), 314 (56), 301 (32), 91 (100).

Data for more polar diastereomer:

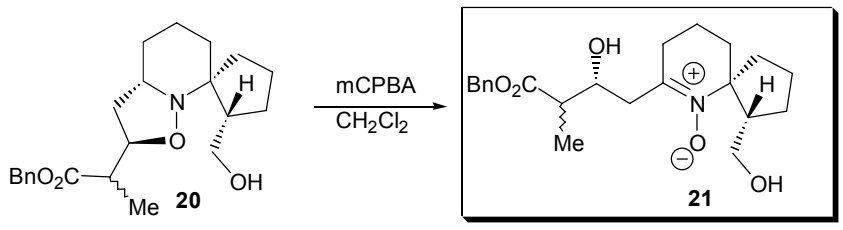
υ_{max} (film)/cm^{-1}: 3448, 2958, 2931, 2873, 1727, 1578, 1459, 1273.

δ_H (300 MHz; $CDCl_3$; Me_4Si) 1.10 (3H, d, *J* = 7.0 Hz, H-2''), 1.23-1.68 (10H, m, H-3$_a$, H-4, H-4', H-5$_a$, H-5', H-6'), 1.68-1.96 (2H, m, H-3$_b$, H-5$_b$), 1.96-2.08 (2H, m, H-3'), 2.08-2.15 (1H, m, H-2), 2.70 (1H, dq, *J* = 8.8, 7.0 Hz, H-1''), 3.40-3.51 (1H, br d, *J* = 6.3 Hz, H-3*a*'), 3.54-3.64 (2H, m, C*H*$_2$OH), 4.20 (1H, dt, *J* = 8.8, 6.0 Hz, H-2'), 5.14 (2H, d, *J* = 4.9 Hz, C*H*$_2$Ph), 7.29-7.36 (5H, m, H-Ar).

δ_C (75 MHz; $CDCl_3$; Me_4Si) 12.7 (CH_3), 19.2 (CH_2), 21.1 (CH_2), 26.4 (CH_2), 27.7 (CH_2), 29.6 (CH_2), 38.0 (CH_2), 39.1 (CH_2), 45.2 (CH), 45.4 (CH), 57.9 (CH), 65.4 (CH_2), 66.0 (CH_2), 69.1 (C), 76.3 (CH), 127.9 (CH), 128.0 (CH), 128.4 (CH), 136.1 (C), 174.5 (C).

m/z (EI) 373.2256 ($C_{22}H_{31}NO_4$ requires M^+ 373.2253). 373 (M^+, 38%), 356 (28), 314 (64), 301 (64), 91 (100).

(1*S,2'*S**,5*S**)-7-((2'*S**)-3'-(benzyloxycarbonyl)-2'-hydroxybutyl)-1-(hydroxymethyl)-6-azaspiro[4.5]dec-6-ene 6-oxide 21**



A solution of *m*-CPBA (1.86 g, 10.8 mmol) in dichloromethane (94 mL) was added dropwise over 1 h to a stirred solution of isoxazolidine **20** (2.02 g, 5.41 mmol) in dichloromethane (86 mL) at 0 ºC and the reaction mixture stirred for 10 min at this temperature. A mixture of saturated aqueous solutions of sodium thiosulfate:sodium hydrogen carbonate (1:1, 150 mL) was added and the aqueous phase extracted with dichloromethane (3 x 30 mL). The combined organic phases were dried ($MgSO_4$) and concentrated. under reduced pressure and the crude residue purified by column chromatography with 5% methanol-dichloromethane as eluent to afford the nitrone **21** (1.92 g, 91%) as a yellow oil and as a separable 1:1 diastereomeric mixture.

Data for less polar diastereomer:

υ_{max} (film)/cm^{-1}: 3367, 2942, 2872, 1730, 1655.

δ_H (300 MHz; $CDCl_3$; Me_4Si) 1.27 (3H, d, *J* = 7.0 Hz, C*H*$_3$), 1.42-1.98 (9H, m, H-1, H-2$_a$, H-3, H-4$_a$, H-9, H-10), 2.19-2.36 (2H, m, H-2$_b$, H-1'$_a$), 2.36-2.72 (4H, m, H-4$_b$, H-3', H-8), 3.04 (1H, dd, *J* = 12.6, 10.2 Hz, H-1'$_b$), 3.68 (2H, d, *J* = 3.6 Hz, C*H*$_2$OH), 4.22 (1H, ddd, *J* = 10.2, 6.7, 2.5 Hz, H-2'), 5.13 (2H, d, *J* = 6.6 Hz, C*H*$_2$Ph), 7.29-7.36 (5H, m, H-Ar).

δ_C (75 MHz; $CDCl_3$; Me_4Si) 12.4 (CH_3), 16.0 (CH_2), 24.0 (CH_2), 28.6 (CH_2), 31.1 (CH_2), 37.0 (CH_2), 38.2 (CH_2), 38.8 (CH_2), 46.4 (CH), 53.1 (CH), 60.2 (CH_2), 65.9 (CH_2), 71.9 (CH), 76.9 (C), 127.9 (CH) (2C), 128.2 (CH), 135.6 (C), 154.3 (C), 174.2 (C).

m/z (EI) 389.2194 ($C_{22}H_{31}NO_5$ requires M^+ 389.2202). 389 (M^+, 5%), 372 (17), 298 (14), 180 (23), 107 (19), 91 (100), 79 (13).

Data for more polar diastereomer:

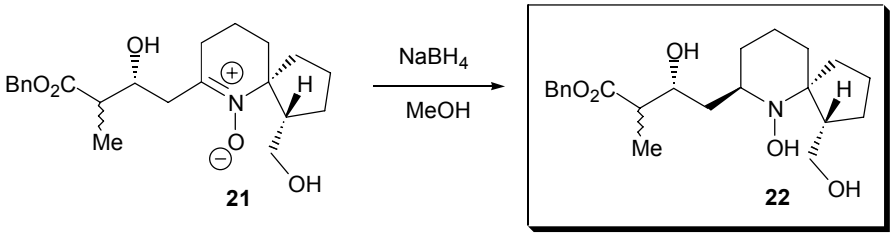
υ_{max} (film)/cm^{-1}: 3394, 2941, 2872, 1731, 1656.

δ_H (300 MHz; $CDCl_3$; Me_4Si) 1.21 (3H, d, *J* = 7.1 Hz, C*H*$_3$), 1.51-1.68 (2H, m, H-3$_a$, H-9$_a$), 1.68-1.84 (4H, m, H-2$_a$, H-4$_a$, H-9$_b$, H-10$_a$), 1.84-2.01 (3H, m, H-1, H-3$_b$, H-10$_b$), 2.07-2.18 (1H, m, H-2$_b$), 2.39-2.49 (3H, m, H-1'$_a$, H-8), 2.57-2.75 (2H, m, H-4$_b$, H-3'), 2.92 (1H, dd, *J* = 12.6, 10.5 Hz, H-1'$_b$), 3.63-3.74 (2H, m, C*H*$_2$OH), 4.27 (1H, ddd, *J* = 10.5, 6.4, 2.3 Hz, H-2'), 5.14 (2H, d, *J* = 5.2, C*H*$_2$Ph), 7.27-7.36 (5H, m, H-Ar).

δ_C (75 MHz; $CDCl_3$; Me_4Si) 11.9 (CH_3), 15.9 (CH_2), 23.9 (CH_2), 28.6 (CH_2), 31.1 (CH_2), 36.8 (CH_2), 37.1 (CH_2), 38.6 (CH_2), 46.2 (CH), 52.9 (CH), 60.2 (CH_2), 65.6 (CH_2), 71.6 (CH), 76.6 (C), 127.6 (CH), (2C), 128.0 (CH), 135.5 (C), 154.1 (C), 173.9 (C).

m/z (EI) 389.2207 ($C_{22}H_{31}NO_5$ requires M^+ 389.2202). 389 (M+, 4%), 372 (11), 298 (11), 180 (21), 107 (19), 91 (100), 79 (13).

(1*S,5*S**,7*R**)-7-((2'*S**)-3'-(benzyloxycarbonyl)-2'-hydroxybutyl)-1-(hydroxymethyl)-6-hydroxyazaspiro[4.5]decan-6-ol 22**



Sodium borohydride (0.75 g, 19.8 mmol) was added to a stirred solution of nitrone **21** (2.60 g, 6.62 mmol) in methanol (165 mL) at 0 ºC. The reaction mixture was then warmed to room temperature and stirred for a further 30 min. The reaction mixture was concentrated under reduced pressure and the residue diluted with ethyl acetate (80 mL) and aqueous brine (120 mL) added. The layers were separated and the aqueous phase further extracted with ethyl acetate (3 x 50 mL). The combined organic phases were dried ($MgSO_4$) and concentrated and the crude residue purified by column chromatography with 35% ethyl acetate-hexane as eluent to afford *N*-hydroxyazaspiro[4.5]decane **22** (1.76 g, 68%) as a yellow oil and as a seperable 1:1 diastereomeric mixture.

Data for less polar diastereomer:

υ_{max} (film)/cm^{-1}: 3320, 2940, 2878, 1732, 1647.

δ_H (300 MHz; $CDCl_3$; Me_4Si) 0.96-1.12 (2H, m, H-1'$_a$, H-8$_a$), 1.12-1.27 (1H, m, H-10$_a$), 1.23 (3H, d, J = 6.9 Hz, CH_3), 1.38-1.68 (5H, m, H-3, H-4$_a$, H-9), 1.68-2.08 (7H, m, H-1, H-2, H-1'$_b$, H-4$_b$, H-8$_b$, H-10$_b$), 2.48 (1H, dq, J = 7.3, 6.9, Hz, H-3'), 2.91 (1H, br t, J =11.3 Hz, H-7), 3.37 (1H, dd, J = 10.3, 2.3, CH_aCH$_b$OH), 3.70 (1H, d, J = 10.3, CH$_a$CH_bOH), 4.04 (1H, dd, J = 9.4, 7.3, H-2'), 5.11 (2H, s, CH_2Ph), 7.25-7.40 (5H, m, H-Ar).

δ_C (75 MHz; $CDCl_3$; Me_4Si) 12.6 (CH_3), 20.2 (CH_2), 20.8 (CH_2), 23.6 (CH_2), 26.2 (CH_2), 26.6 (CH_2), 29.5 (CH_2), 34.8 (CH_2), 46.1 (CH), 50.2 (CH), 61.2 (CH), 64.4 (CH_2), 65.8 (CH_2), 71.1 (C), 74.8 (CH), 127.9 (CH) (2C), 128.2 (CH), 135.9 (C), 174.6 (C).

m/z (EI) 391.2358 ($C_{22}H_{33}NO_5$ requires M^+ 391.2358). 391 (M^+, 26%), 374 (10), 332 (9), 319 (10), 300 (44), 91 (100), 79 (14), 67 (14), 55 (20), 41 (22).

Data for more polar diastereomer:

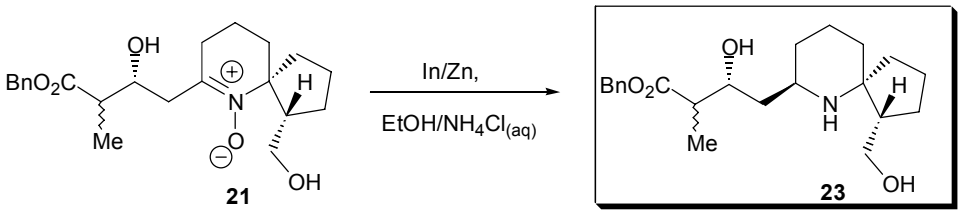
υ_{max} (film)/cm^{-1}: 3389, 2939, 2878, 1732, 1644.

δ_H (300 MHz; $CDCl_3$; Me_4Si) 0.98-1.09 (1H, m, H-8$_a$), 1.12 (3H, d, J = 7.1 Hz, CH_3), 1.14-1.27 (2H, m, H-1'$_a$, H-10$_a$), 1.36-1.67 (5H, m, H-3, H-4$_a$, H-9), 1.67-2.18 (7H, m, H-1, H-2, H-1'$_b$, H-4$_b$, H-8$_b$, H-10$_b$), 2.58 (1H, q, J = 7.1 Hz, H-3'), 2.92 (1H, br t, J =11.1 Hz, H-7), 3.37 (1H, dd, J = 10.1, 2.1 Hz, CH_aH$_b$OH), 3.68 (1H, d, J = 10.1 Hz, CH$_a$CH_bOH), 4.10 (1H, dd, J = 9.4, 7.1 Hz, H-2'), 5.14 (2H, d, J = 5.4, CH_2Ph), 7.24-7.44 (5H, m, H-Ar).

δ_C (75 MHz; $CDCl_3$; Me_4Si) 12.5 (CH_3), 20.2 (CH_2), 20.8 (CH_2), 23.8 (CH_2), 26.2 (CH_2), 26.6 (CH_2), 29.5 (CH_2), 33.1 (CH_2), 45.9 (CH), 50.1 (CH), 61.2 (CH), 64.3 (CH_2), 65.8 (CH_2), 71.1 (C), 75.0 (CH), 127.8 (CH) (2C), 128.2 (CH), 136.0 (C), 174.8 (C).

m/z (EI) 391.2355 ($C_{22}H_{33}NO_5$ requires M^+ 391.2358). 391 (M^+, 23%), 374 (9), 332 (7), 319 (9), 300 (35), 91 (100), 79 (15), 67 (15), 55 (23), 41 (27).

(1*S,5*S**,7*R**)-7-((2'*S**)-3'-(benzyloxycarbonyl)-2'-hydroxybutyl)-1-(hydroxymethyl)-6-azaspiro[4.5]decane 23**



A solution of hydroxylamine **22** (1.76 g, 4.49 mmol) in ethanol (85 mL) was transferred *via* cannula to a flask containing indium powder (0.025 g, 0.22 mmol) and zinc powder (0.73 g, 11.2 mmol). Saturated aqueous ammonium chloride (8.9 mL) was added and the reaction mixture was stirred under reflux for 4 h. The reaction mixture was cooled to room temperature, filtered though a pad of Celite® and the filtrate concentrated. Ethyl acetate (50 mL) and saturated aqueous sodium bicarbonate (50 mL) were added to the residue and the layers separated. The aqueous phase was further extracted with ethyl acetate (3 x 30 mL) and the combined organic phases dried ($MgSO_4$) and concentrated. The crude residue was purified by column chromatography with 5% methanol-dichloromethane as eluent

to afford azaspiro[4.5]decane **23** (1.68 g, 100%) as a yellow oil and as a separable 1:1 diastereomeric mixture.

Data for less polar diastereomer:

υ_{max} (film)/cm^{-1}: 3394, 2933, 2864, 1731, 1648.

δ_H (300 MHz; $CDCl_3$; Me_4Si) 0.92-1.06 (1H, m, H-8_a), 1.22 (3H, d, *J* = 7.0 Hz, C*H*$_3$), 1.25-1.61 (9H, m, H-2_a, H-1', H-3_a, H-8_b, H-9, H-10), 1.61-1.84 (5H, m, H-1, H-2_b, H-3_b, H-4), 2.48 (1H, dq, *J* = 7.0, 6.7 Hz, H-3'), 2.77-2.89 (1H, m, H-7), 3.35-3.82 (2H, m, C*H*$_2$OH), 4.01 (1H, ddd, *J* = 9.6, 6.7, 3.0 Hz, H-2'), 5.13 (2H, s, C*H*$_2$Ph), 7.27-7.36 (5H, m, H-Ar).

δ_C (75 MHz; $CDCl_3$; Me_4Si) 12.9 (CH_3), 22.1 (CH_2), 22.7 (CH_2), 27.2 (CH_2), 33.1 (CH_2), 35.5 (CH_2), 36.7 (CH_2), 40.1 (CH_2), 46.1 (CH), 51.0 (CH), 52.5 (CH), 62.7 (CH_2), 63.1 (C), 66.0 (CH_2), 73.4 (CH), 128.0 (CH) (2C), 128.4 (CH), 135.9 (C), 175.0 (C).

m/z (FAB) 376.2487 ($C_{22}H_{34}NO_4$ requires MH^+ 376.2487). 376 (MH^+, 73%), 154 (100), 136 (71), 107 (25), 91 (30), 77 (24):

Data for more polar diastereomer:

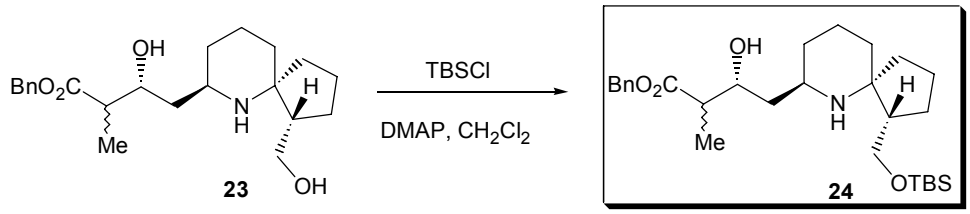
υ_{max} (film)/cm^{-1}: 3400, 2934, 2864, 1734, 1647.

δ_H (300 MHz; $CDCl_3$; Me_4Si) 0.96-1.10 (1H, m, H-8_a), 1.13 (3H, d, *J* = 7.1 Hz, C*H*$_3$), 1.18-1.32 (1H, m, H-$1'_a$), 1.32-1.55 (4H, m, H-2_a, H-$1'_b$, H-3_a, H-10_a), 1.55-1.84 (9H, m, H-1, H-2_b, H-3_b, H-4, H-8_b, H-9, H-10_b), 2.54 (1H, dq, *J* = 7.1, 7.0 Hz, H-3'), 2.78-2.86 (1H, m, H-7), 3.59-3.72 (2H, m, C*H*$_2$OH), 3.99 (1H, ddd, 10.6, 7.0, 2.1 Hz, H-2'), 5.14 (2H, s, C*H*$_2$Ph), 7.26-7.35 (5H, m, H-Ar).

δ_C (75 MHz; $CDCl_3$; Me_4Si) 12.9 (CH_3), 22.1 (CH_2), 22.7 (CH_2), 27.2 (CH_2), 33.2 (CH_2), 35.5 (CH_2), 36.7 (CH_2), 39.3 (CH_2), 46.3 (CH), 51.1 (CH), 52.3 (CH), 62.6 (CH_2), 63.0 (C), 65.9 (CH_2), 74.0 (CH), 127.9 (CH) (2C), 128.3 (CH), 135.9 (C), 174.9 (C).

m/z (FAB) 376.2489 ($C_{22}H_{34}NO_4$ requires MH^+ 376.2487). 376 (MH^+, 100%), 154 (35), 136 (26), 107 (10), 91 (44), 77 (11).

(1*S,5*S**,7*R**)-7-((2'*S**)-3'-(benzyloxycarbonyl)-2'-hydroxybutyl)-1-(*tert*-butyldimethylsilyloxymethyl)-6-azaspiro[4.5]decane 24**



tert-Butyldimethylsilyl chloride (0.64 g, 4.29 mmol) was added to a stirred solution of alcohol **23** (1.34 g, 3.57 mmol), triethylamine (2.97 mL, 21.4 mmol) and DMAP (0.04 g, 0.36 mmol) in dichloromethane (30 mL) at 0 °C. The reaction mixture was warmed to room temperature and stirred for 24 h. Water (40 mL) was added and the aqueous phase extracted with dichloromethane (3 x 20 mL). The combined organic phases were dried ($MgSO_4$) and concentrated and the crude residue purified by column chromatography with 1% methanol-dichloromethane as eluent to afford the silyl ether **24** (1.55 g, 89%) as a yellow oil and as a separable 1:1 diastereomeric mixture.

Data for less polar diastereomer:

υ_{max} (film)/cm^{-1}: 3444, 2930, 2858, 1732 , 1460, 1257, 1100, 1074.

δ_H (300 MHz; $CDCl_3$; Me_4Si) 0.06 (3H, s, $(CH_3)_2Si$), 0.07 (3H, s, $(CH_3)_2Si$), 0.90 (9H, s, $C(CH_3)_3Si$), 1.23 (3H, d, J = 7.0 Hz, CH_3), 1.29-1.40 (1H, m, H-2_a), 1.40-1.52 (4H, m, H-1', H-3_a, H-10_a), 1.52-1.59 (2H, m, H-8), 1.59-1.82 (8H, m, H-1, H-2_b, H-3_b, H-4, H-9, H-10_b), 2.46 (1H, dq, J =7.0, 6.7 Hz, H-3'), 2.88 (1H, br s, H-7), 3.62-3.81 (2H, m, CH_2OTBS), 3.98-4.11 (1H, m, H-2'), 5.13 (2H, s, CH_2Ph), 7.28-7.43 (5H, m, H-Ar).

δ_C (75 MHz; $CDCl_3$; Me_4Si) -5.6 (CH_3), 12.4 (CH_3), 18.0 (C), 22.0 (CH_2), 23.5 (CH_2), 25.8 (CH_3), 27.7 (CH_2), 33.4 (CH_2), 36.0 (CH_2), 37.2 (CH_2), 39.6 (CH_2), 46.5 (CH), 51.4 (CH), 53.2 (CH), 62.7 (C), 63.3 (CH_2), 65.9 (CH_2), 73.7 (CH), 128.0 (CH), 128.4 (CH) (2C), 136.1 (C), 174.9 (C).

m/z (EI) 489.3271 ($C_{28}H_{47}NO_4Si$ requires M^+ 489.3274). (489 (M^+, 54%), 474 (8), 446 (33), 432 (26), 326 (57), 316 (54), 303 (31), 283 (38), 169 (25), 91 (100), 73 (37).

Data for more polar diastereomer:

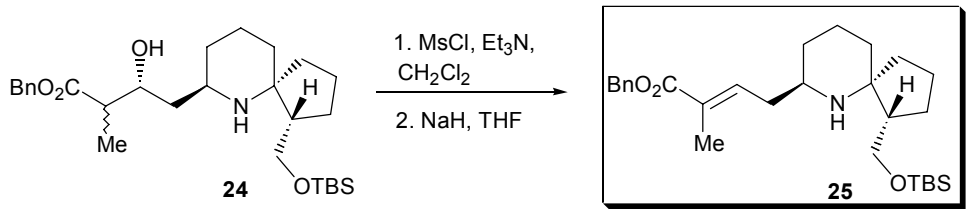
υ_{max} (film)/cm^{-1}: 3272, 2928, 2857, 1734, 1461, 1252, 1170, 1070.

δ_H (400 MHz; $CDCl_3$; Me_4Si) 0.06 (6H, s, $(CH_3)_2Si$), 0.91 (9H, s, $C(CH_3)_3Si$), 0.91-1.01 (1H, m, H-8_a), 1.12 (3H, d, J =7.0, CH_3), 1.18-1.33 (2H, m, H-2_a, H-$1'_a$), 1.33-1.45 (2H, m, H-3_a, H-10_a), 1.45-1.73 (8H, m, H-2_b, H-$1'_b$, H-3_b, H-4_a, H-8_b, H-9, H-10_b), 1.73-1.82 (2H, m, H-1, H-4_b), 2.58 (1H, d, J = 7.0, 6.9 Hz, H-3'), 2.82 (1H, br s, H-7), 3.68 (2H, br d, J =8.0 Hz, CH_2OTBS), 4.11 (1H, ddd, J = 10.4, 6.9, 1.5 Hz, H-2'), 5.14 (2H, s, CH_2Ph), 7.21-7.43 (5H, m, H-Ar).

δ_C (100 MHz; $CDCl_3$; Me_4Si) -5.7 (CH_3), 11.9 (CH_3), 17.7 (C), 22.2 (CH_2), 23.4 (CH_2), 25.6 (CH_3), 27.6 (CH_2), 33.7 (CH_2), 36.1 (CH_2), 37.3 (CH_2), 38.1 (CH_2), 46.2 (CH), 51.4 (CH), 52.8 (CH), 62.1 (C), 63.1 (CH_2), 65.6 (CH_2), 74.1 (CH), 127.6 (CH), 127.8 (CH), 128.1 (CH), 136.0 (C), 174.5 (C).

m/z (EI) 489.3282 ($C_{28}H_{47}NO_4Si$ requires M^+ 489.3274) 489 (M^+, 31%), 474 (4), 446 (25), 432 (11), 326 (22), 316 (27), 303 (20), 282 (19), 169 (20), 91 (100), 73 (30).

(1*S,5*S**,7*R**)-7-((*E*)-3'-(benzyloxycarbonyl)but-2'-enyl)-1-(*tert*-butyldimethylsilyloxymethyl)-6-azaspiro[4.5]decane 25**



Mesyl chloride (0.15 mL, 1.95 mmol) was added dropwise to a stirred solution of alcohol **24** (0.32 g, 0.65 mmol) and triethylamine (0.45 mL, 3.24 mmol) in dichloromethane (20 mL) at 0 °C. The reaction mixture was stirred for 15 min at this temperature then warmed to room temperature and stirred for a further 3 h. Water (20 mL) was added and the aqueous phase extracted with dichloromethane (3 x 10 mL). The combined organic phases were dried ($MgSO_4$) and concentrated under reduced pressure to give an orange oil. The crude residue was dissolved in tetrahydrofuran (23 mL) and added to a flask containing a 60% dispersion of sodium hydride in mineral oil (0.11 g, 4.71 mmol) at 0 °C. The reaction mixture

was warmed to room temperature and stirred for 3 days. Water (25 mL) was added and the aqueous phase extracted with ethyl acetate (3 x 10 mL). The combined organic phases were dried ($MgSO_4$) and concentrated and the crude residue purified by column chromatography with 1% methanol-dichloromethane (1:99) as eluent to afford (*E*)-alkene **25** (0.26 g, 85% over two steps) as a yellow oil.

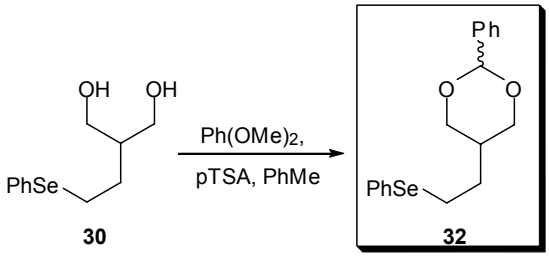
υ_{max} (film)/cm^{-1}: 3426, 3034, 2928, 2857, 1713, 1650, 1462, 1255, 1074.

δ_H (400 MHz; $CDCl_3$; Me_4Si) 0.14 (6H, s, $(CH_3)_2Si$), 0.99 (9H, s, $C(CH_3)_3Si$), 1.04-1.18 (1H, m, H-8_a), 1.41-1.68 (6H, m, H-2_a, H-3_a, H-4_a, H-9_a, H-10), 1.68-1.90 (6H, m, H-1, H-2_b, H-3_b, H-4_b, H-8_b, H-9_b), 1.98 (3H, s, CH_3), 2.23-2.53 (2H, m, H-1'), 2.89 (1H, br s, H-7), 3.78-3.94 (2H, m, CH_2OTBS), 5.28 (2H, s, CH_2Ph), 6.93 (1H, t, J =7.3 Hz, H-2'), 7.38-7.52 (5H, m, H-Ar).

δ_C (100 MHz; $CDCl_3$; Me_4Si) -5.5 (CH_3), 12.6 (CH_3), 18.0 (C), 22.0 (CH_2), 22.6 (CH_2), 25.8 (CH_3), 27.7 (CH_2), 32.4 (CH_2), 35.5 (CH_2), 36.5 (CH_2), 36.8 (CH_2), 51.6 (CH) (2C), 63.4 (CH_2), 64.2 (C), 66.1 (CH_2), 127.8 (CH), 127.9 (CH), 128.4 (CH), 129.0 (C), 136.3 (C), 139.7 (CH), 167.6 (C).

m/z (EI) 471.3168 ($C_{28}H_{45}NO_3Si$ requires M^+ 471.3168). 471 (M^+, 2%), 456 (2), 428 (4), 414 (3), 282 (100), 224 (6), 150 (12), 91 (31), 73 (13).

2-phenyl-5-[1'-(phenylseleno)ethyl]-1,3-dioxane 32



A solution of benzaldehyde dimethyl acetal (1.67 mL, 11.2 mmol) in toluene (20 mL) was added to a stirred solution of diol **30** (2.62 g, 10.1 mmol) and *para*-toluene sulfonic acid (0.09 g, 0.5 mmol). The reaction mixture was heated under reflux using a Dean-Stark trap for 6 h then cooled to room temperature. Potassium carbonate (1.5 g) was added and the mixture stirred for a further 24 h then filtered through a pad of Celite®. The filtrate was concentrated and the crude residue purified by column chromatography with 3% ethyl acetate- hexane as eluent to afford acetal **32** (3.51 g, 100%) as a yellow oil and a diastereomeric mixture.

υ_{max} (film)/cm^{-1}: 3067, 2960, 2929, 2844, 1578, 1386, 1136, 1073.

δ_H (300 MHz; $CDCl_3$; Me_4Si) δ_H (300 MHz; $CDCl_3$; Me_4Si) 1.39-1.51 (1H, m, $CH_aH_bCH_2$SePh), 1.54-1.63 (0.5H, m, H-5), 2.13-2.34 (1.5H, m, H-5*, $CH_aH_bCH_2$SePh), 2.78-2.87 (1H, m, H-CH_aH_bSePh), 3.02 (1H, t, J = 7.4 Hz, CH_aH_bSePh), 3.48 (1H, t, J = 11.6 Hz, H-4_a), 3.99-4.04 (2H, m, H-4*), 4.19 (1H, dd, J = 11.6, 4.6 Hz, H-4_b), 5.35 (0.5H, s, H-2), 5.45 (0.5H, s, H-2*), 7.18-7.56 (10H, m, H-Ar).

δ_C (75 MHz; $CDCl_3$; Me_4Si) 24.2 (CH_2), 25.6 (CH_2*), 28.7 (CH_2), 29.4 (CH_2*), 33.8 (CH_2), 34.3 (CH_2*), 70.0 (CH_2) (2C), 71.7 (CH_2*) (2C), 101.3 (CH*), 101.8 (CH), 125.9 (CH), 126.7 (CH), 126.9 (CH), 128.1 (CH), 128.7 (CH) (2C), 128.9 (CH), 129.0 (CH), 129.5 (C), 130.0 (C), 132.4 (CH), 132.7 (CH), 138.2 (C), 138.4 (C).

m/z (EI) 348.0629 (M^+ $C_{18}H_{20}O_2{}^{80}Se$ requires 348.0628). (M^+, 67%), 191 (68), 105 (100), 91 (68), 77 (53), 55 (95).

A Concise Approach to the Core Structures of Pinnaic Acid and Halichlorine

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Supporting Information:

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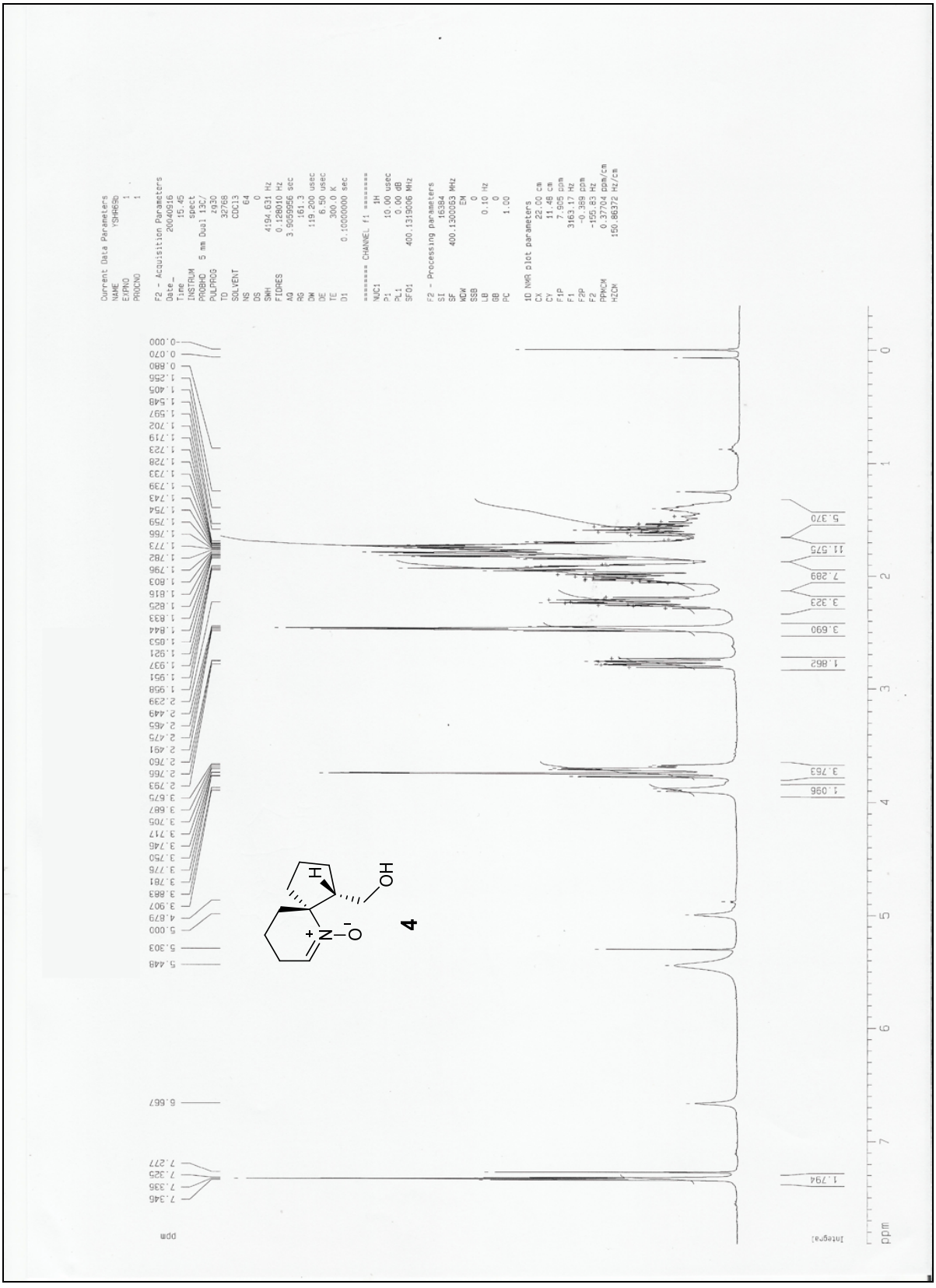
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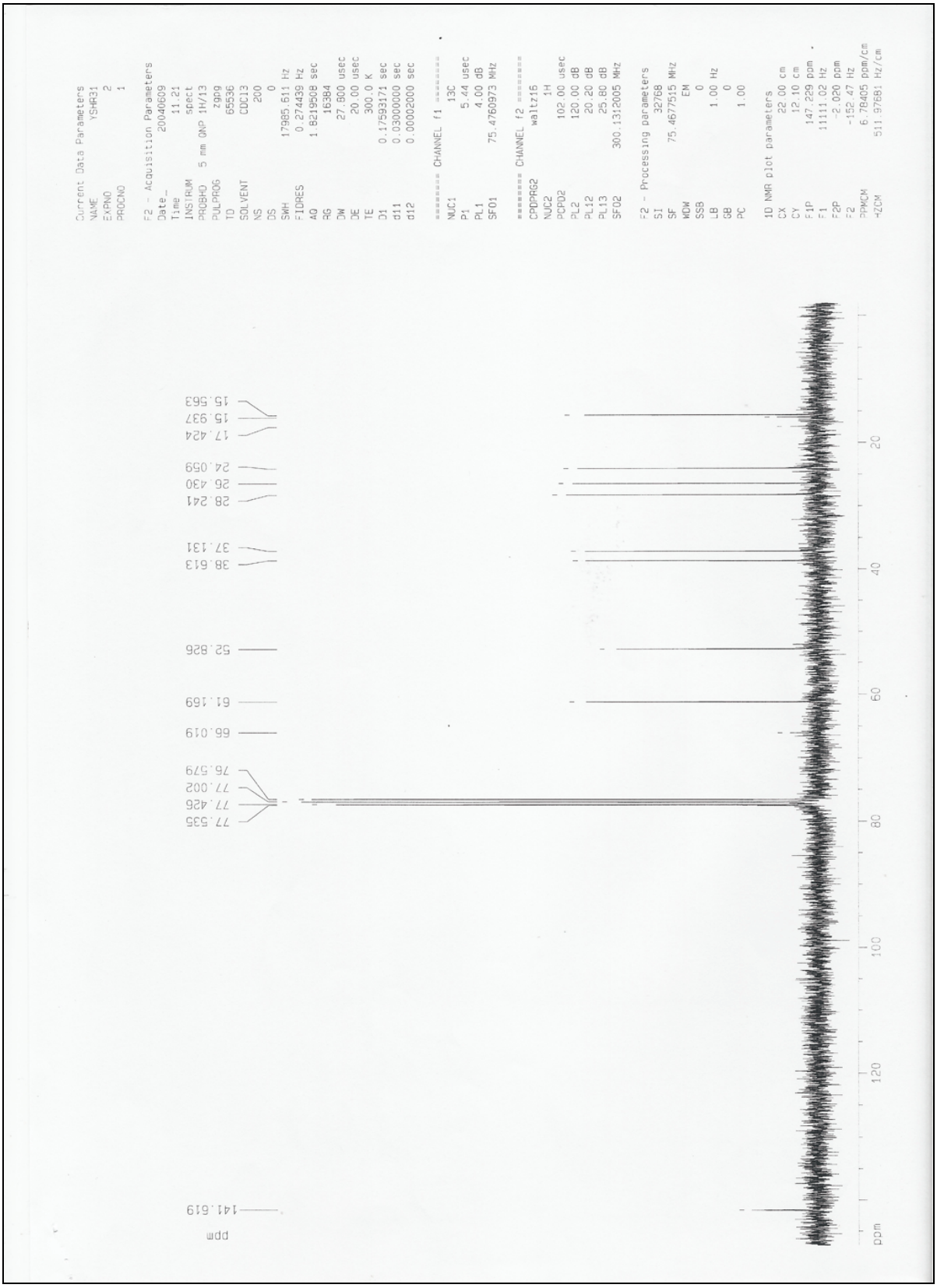
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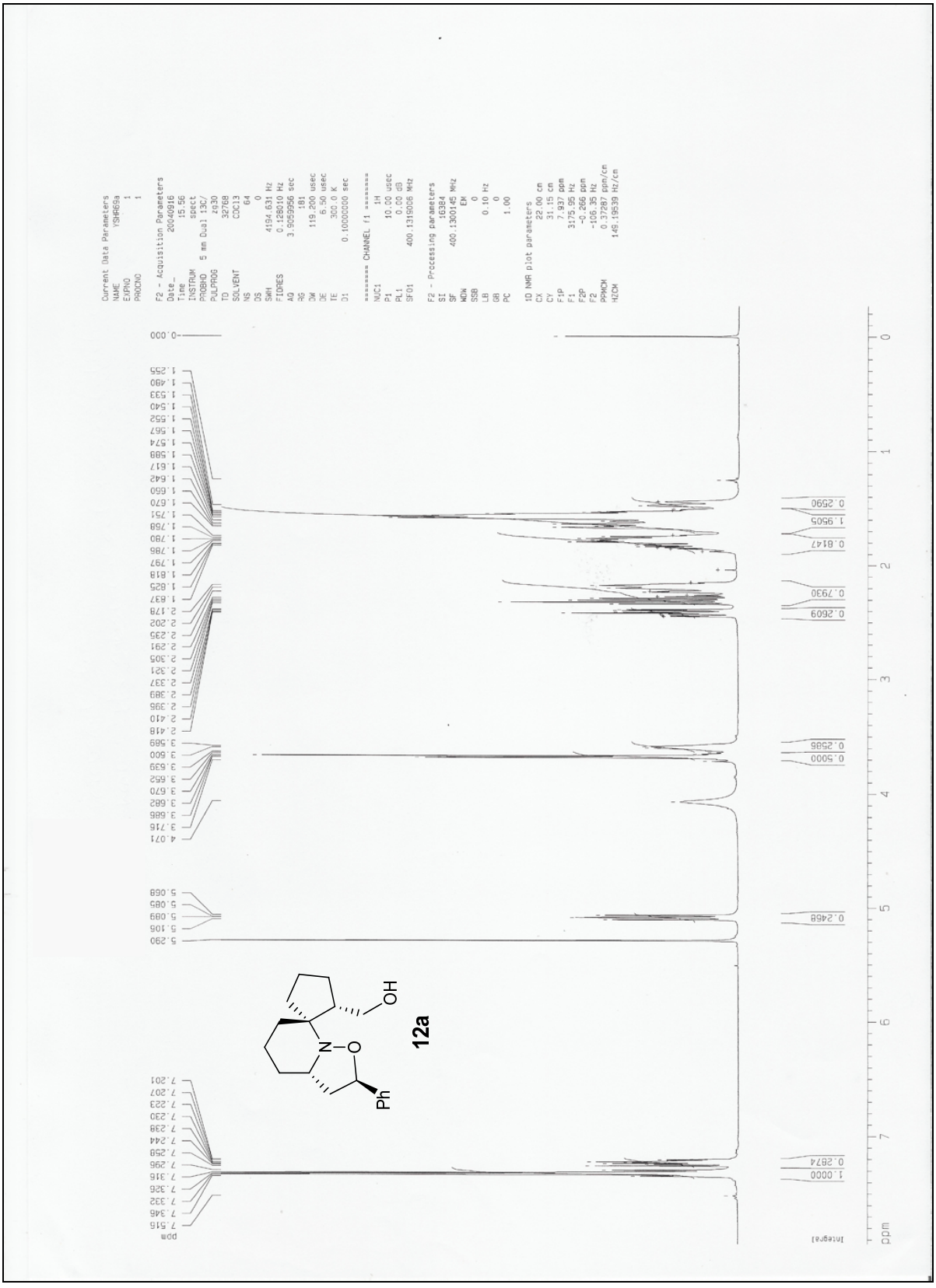
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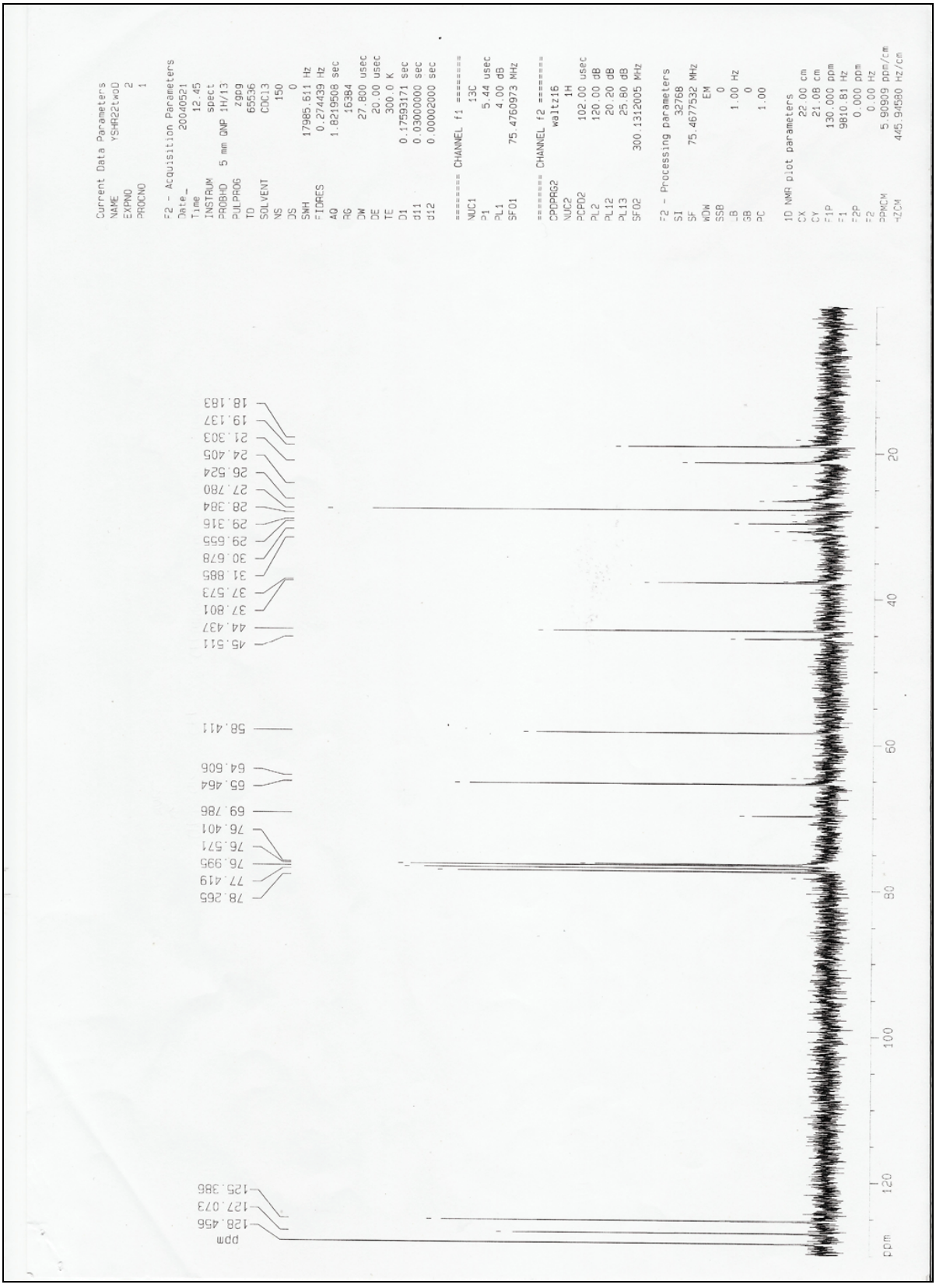
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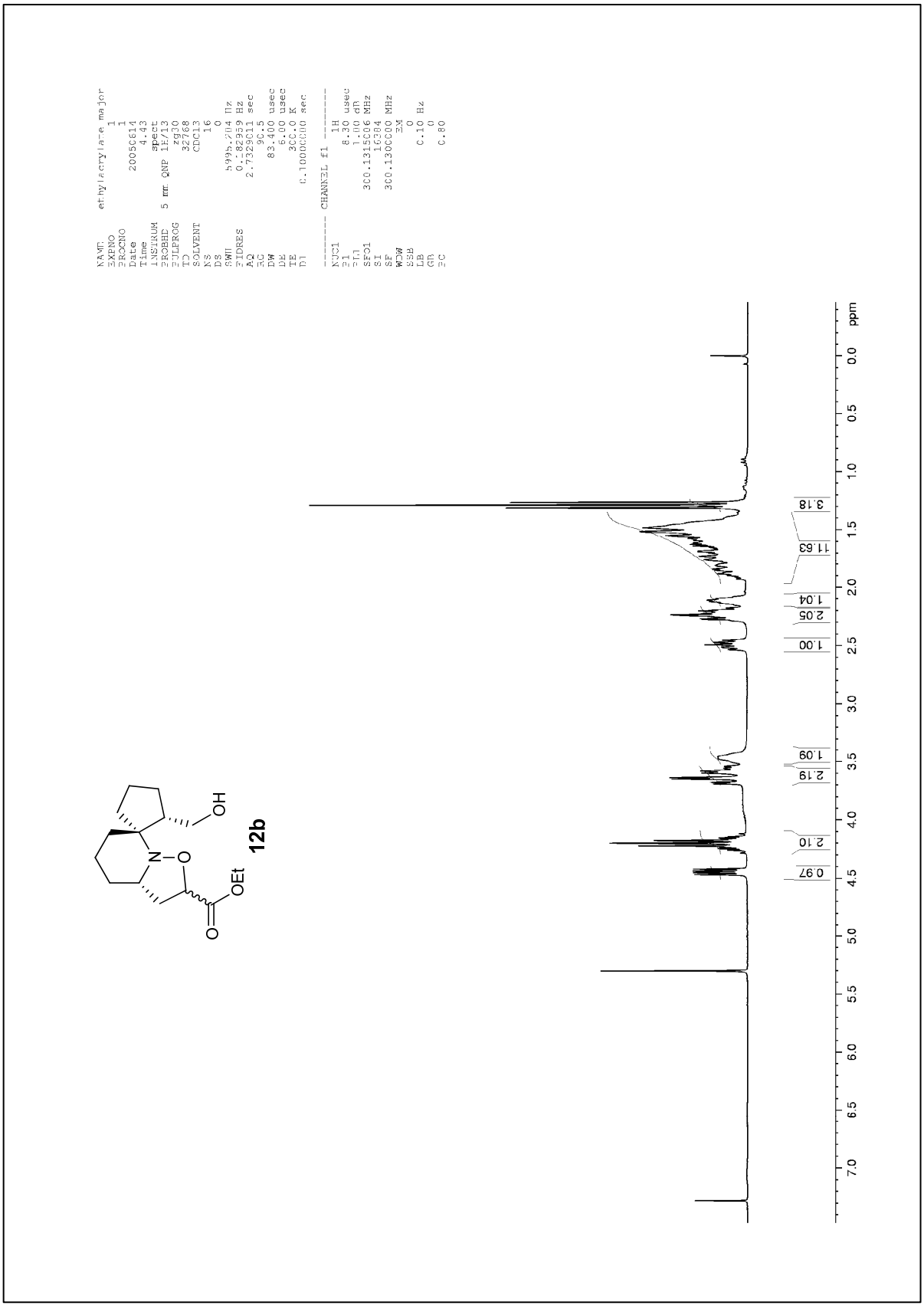
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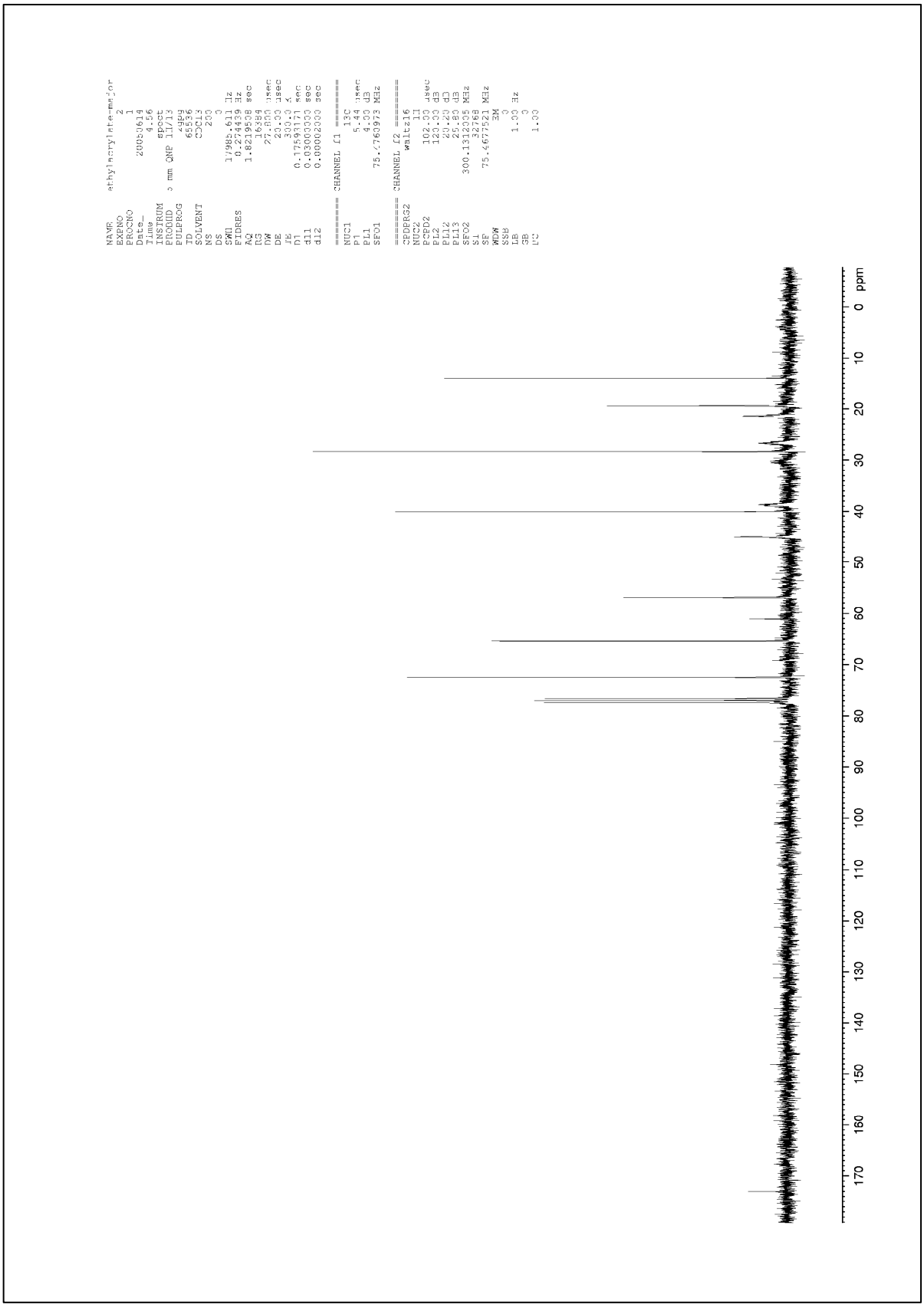
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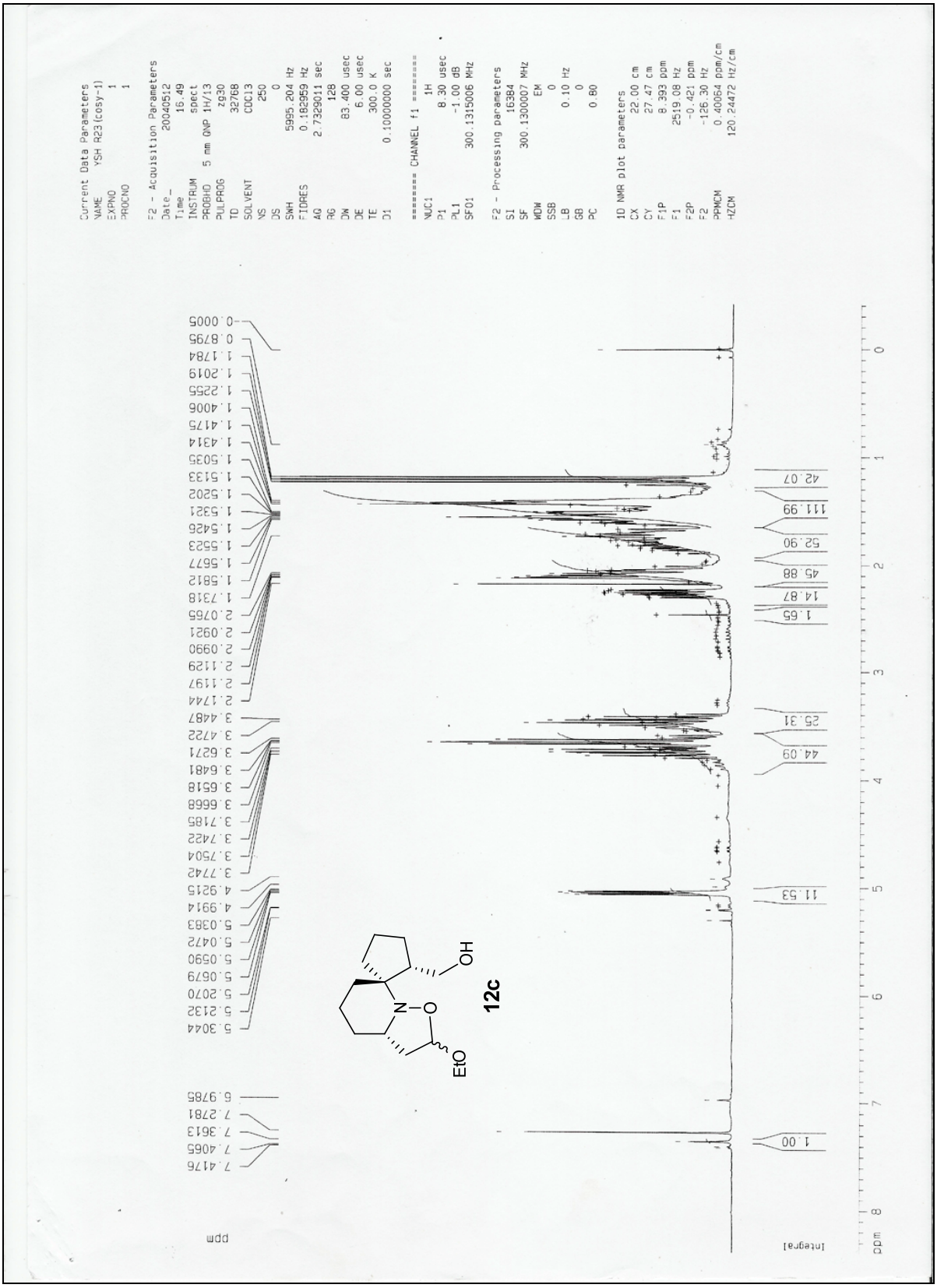
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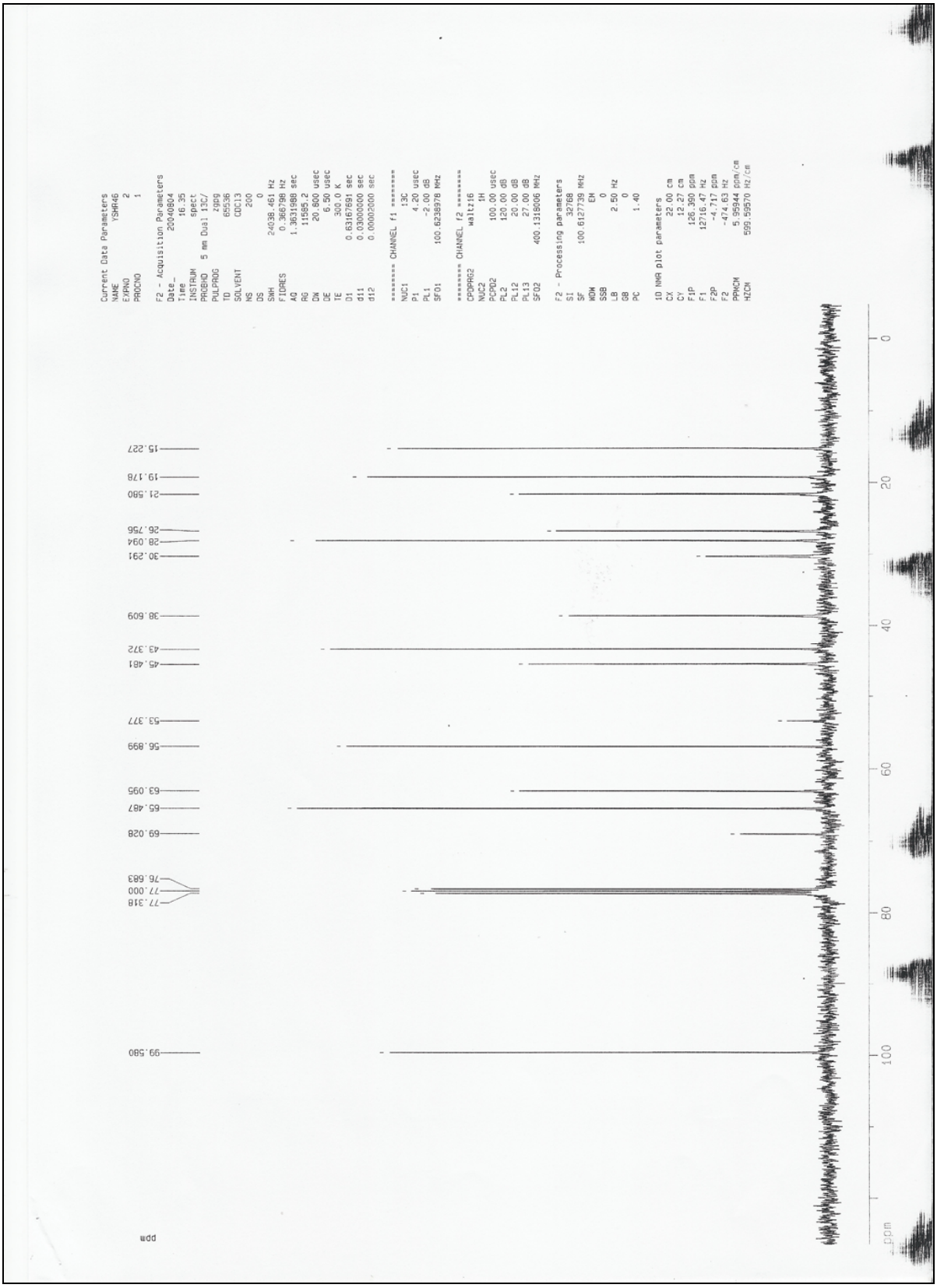
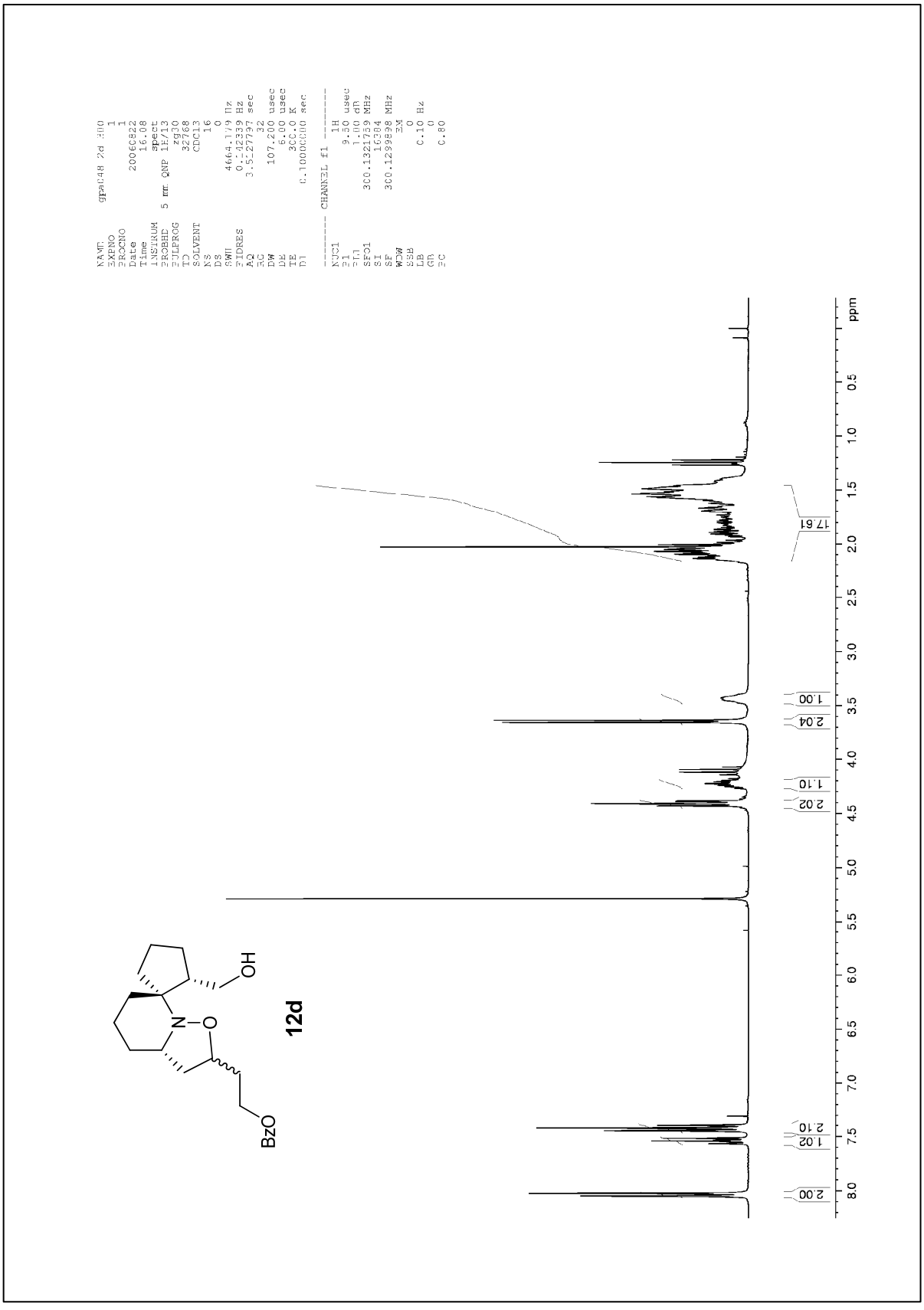
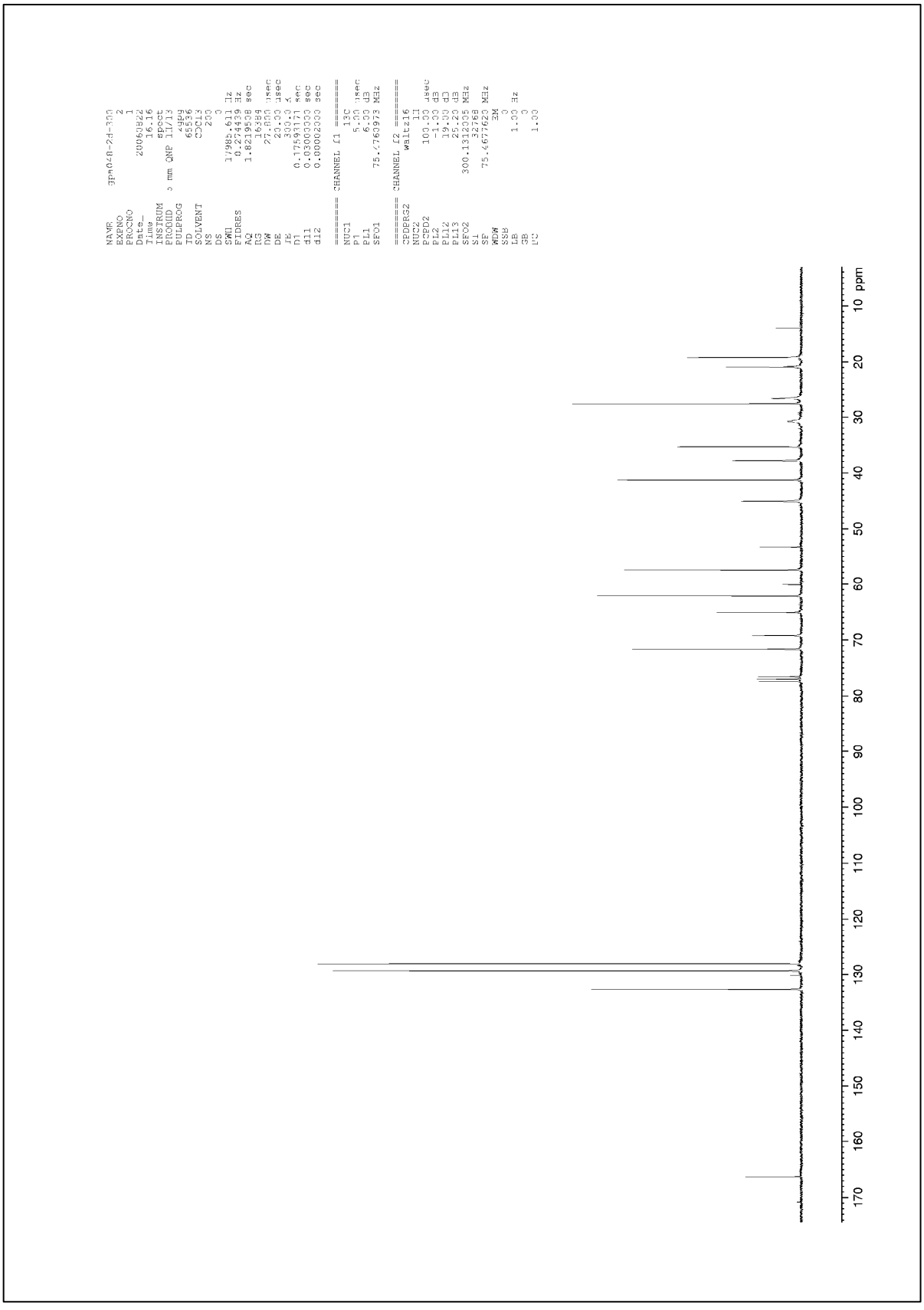
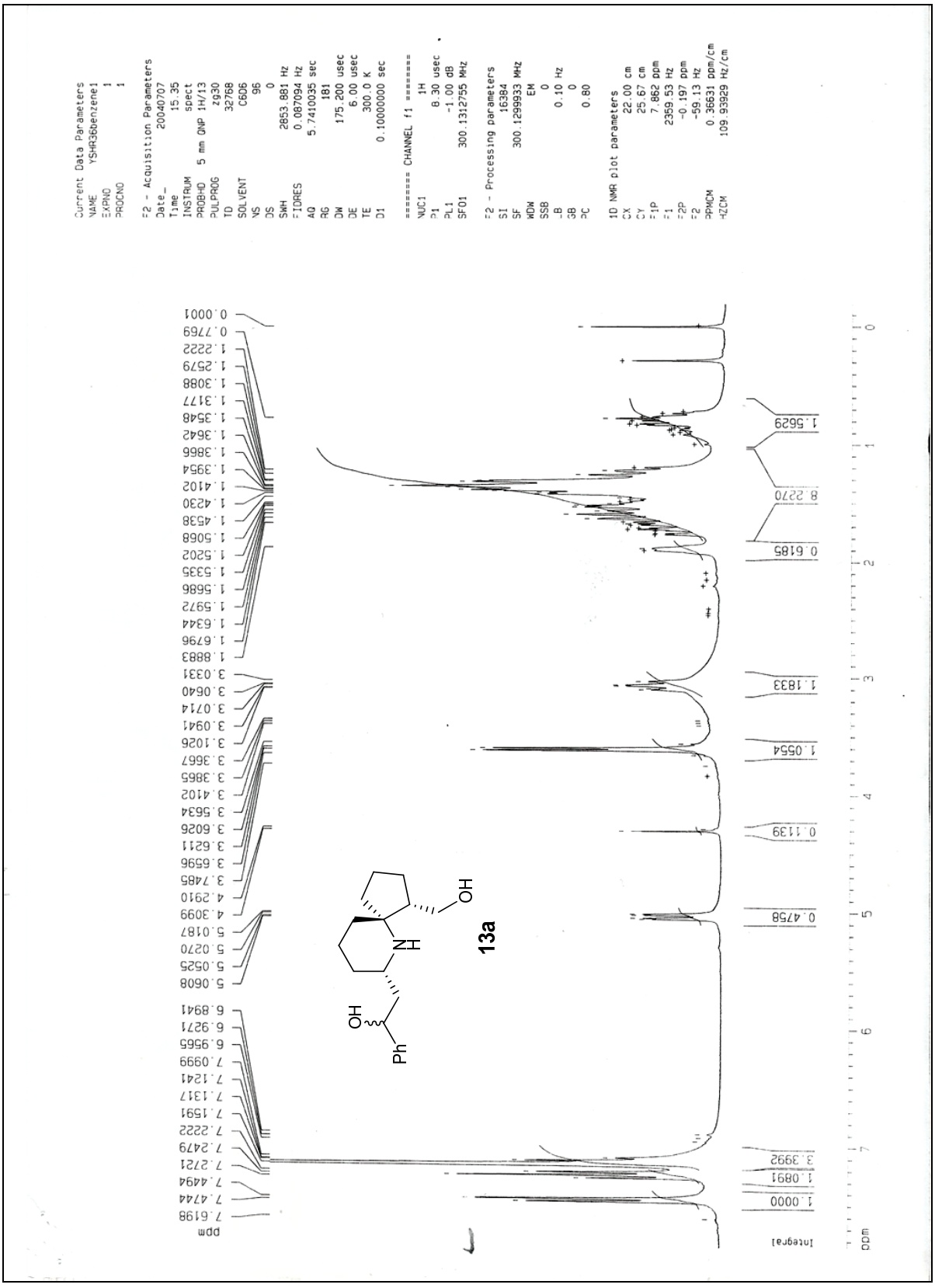
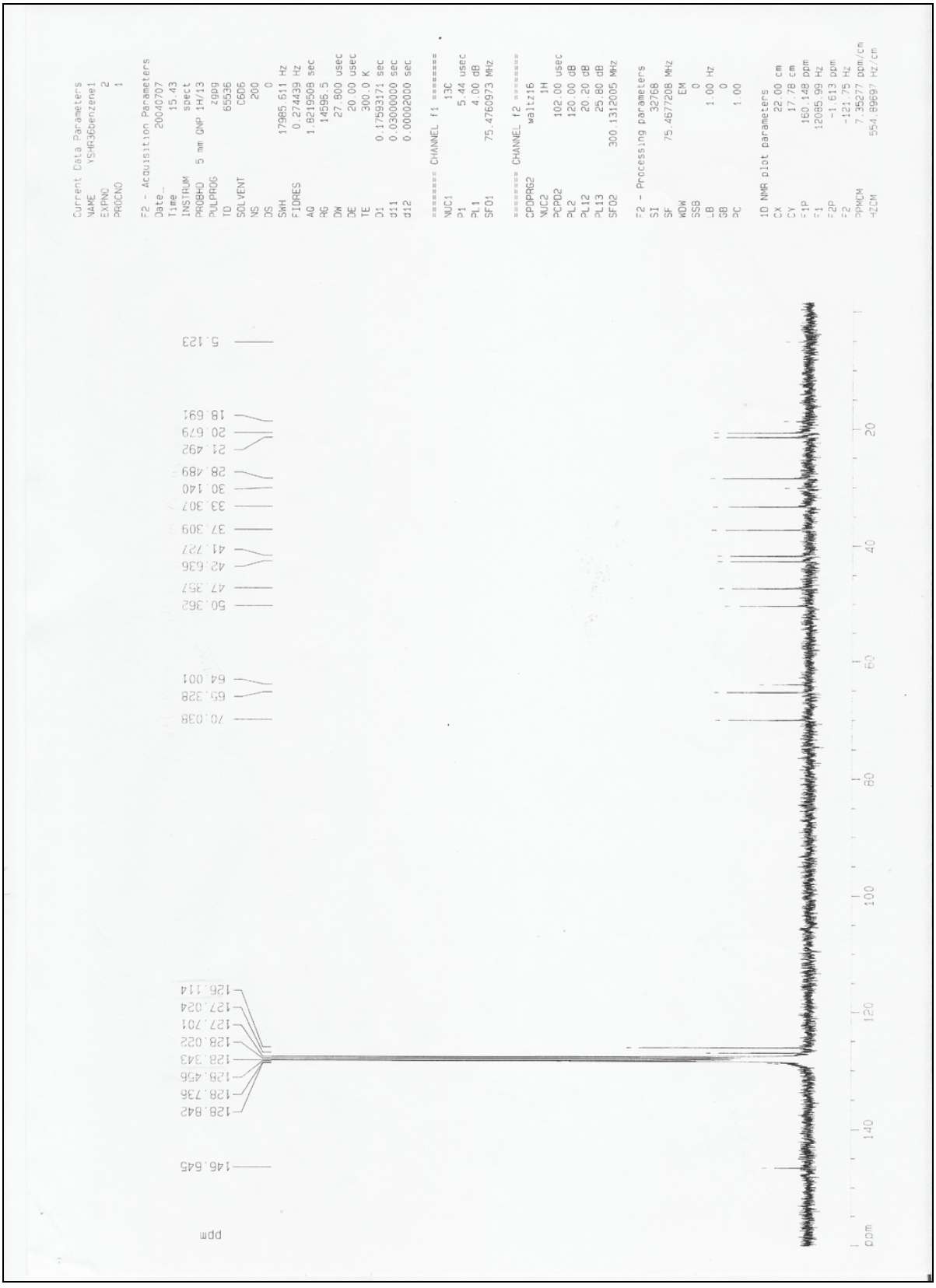
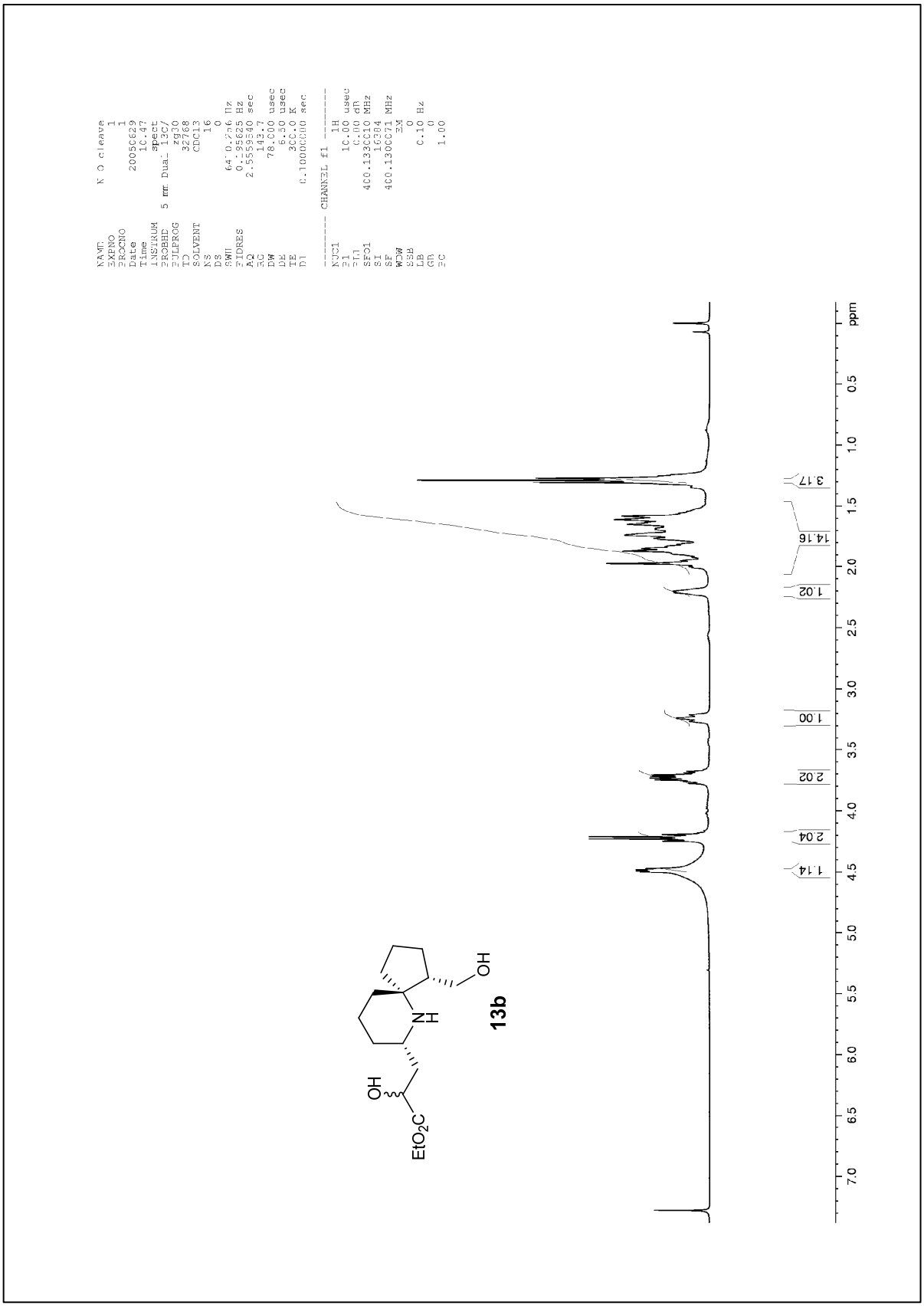

Current Data Parameters
NAME YSHR46
EXPNO 2
PROCNO 1
F2 - Acquisition Parameters
Date_ 20040804
Time 16.35
INSTRUM spect
PROBHD 5 mm Dual 13C/
PULPROG zgpg
TD 65536
SOLVENT CDCl3
NS 200
DS 0
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3631988 sec
RG 11585.2
DW 20.800 usec
DE 6.50 usec
TE 300.0 K
D1 0.63167691 sec
d11 0.03000000 sec
d12 0.00002000 sec
======== CHANNEL f1 ========
NUC1 13C
P1 4.20 usec
PL1 -2.00 dB
SFO1 100.6238978 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 100.00 usec
PL2 120.00 dB
PL12 27.00 dB
PL13 27.00 dB
SFO2 400.1316006 MHz
F2 - Processing parameters
SI 32768
SF 100.6127339 MHz
WDW EM
SSB 0
LB 2.50 Hz
GB 0
PC 1.40
1D NMR plot parameters
CX 22.00 cm
CY 12.27 cm
F1P 126.950 ppm
F1 12716.47 Hz
F2P -4.717 ppm
F2 -474.63 Hz
PPMCM 5.95944 ppm/cm
HZCM 599.59570 Hz/cm
15.227
19.178
21.580
26.756
28.094
30.291
38.609
43.372
45.481
53.377
56.899
63.095
65.487
69.028
76.683
77.000
77.318
99.580
ppm
0
20
40
60
80
100
ppm

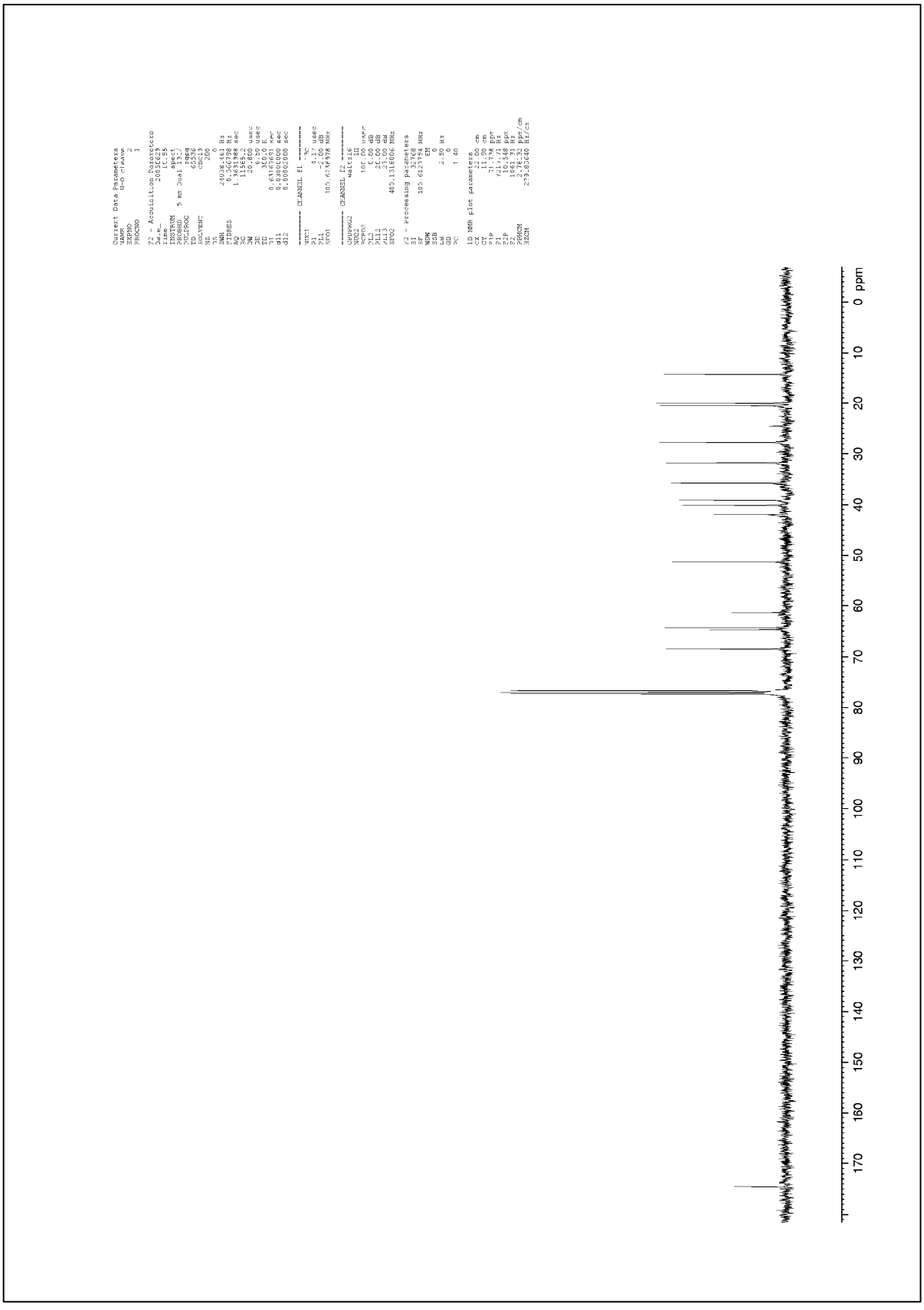

12d
OH
N—O
BzO
ppm
17.61
1.00
2.04
1.10
2.02
1.02
2.10
2.00

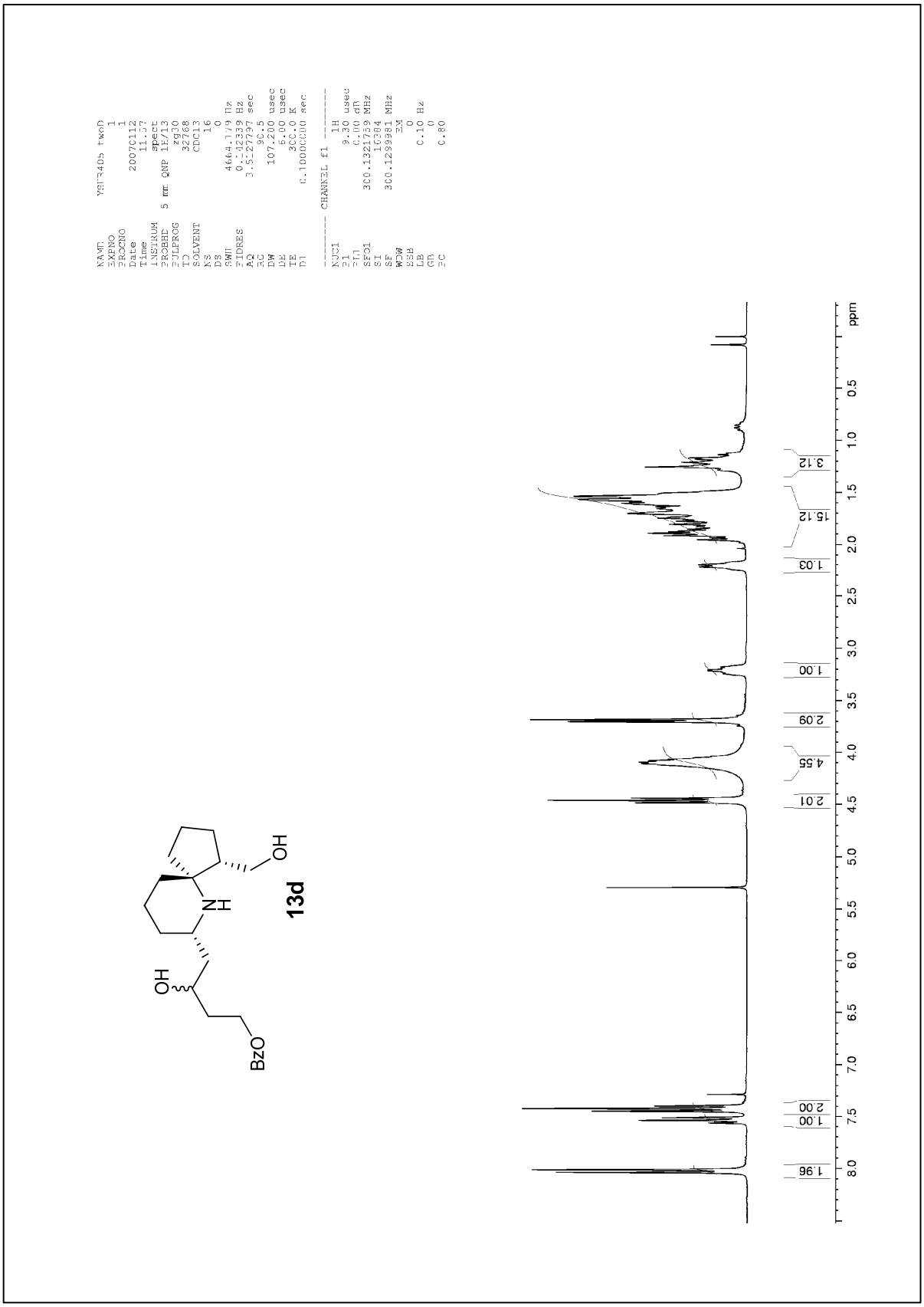

NAME gpn048-24-300
EXPNO 2
PROCNO 1
Date_ 20060822
Time 16.16
INSTRUM spect
PROBHD 5 mm QNP 1H/13
PULPROG zgpg
TD 65536
SOLVENT CDCl3
NS 200
DS 0
SWH 19980.414 Hz
FIDRES 0.304810 Hz
AQ 1.8219958 sec
RG 16384
DW 27.800 usec
DE 20.00 usec
TE 300.0 K
D1 0.17593171 sec
d11 0.03000000 sec
d12 0.00002000 sec
======== CHANNEL f1 ========
NUC1 13C
P1 5.00 usec
PL1 6.00 dB
SFO1 75.4769973 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 100.00 usec
PL2 -1.00 dB
PL12 19.00 dB
PL13 25.20 dB
SFO2 300.1312005 MHz
SI 32768
SF 75.4677622 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.00
170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 ppm


Current Data Parameters
NAME YSHR36benzene1
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20040707
Time 15.35
INSTRUM spect
PROBHD 5 mm QNP 1H/13
PULPROG zg30
TD 32768
SOLVENT C606
NS 96
DS 0
SWH 2853.881 Hz
FIDRES 0.087094 Hz
AQ 5.7410035 sec
RG 181
DW 175.200 usec
DE 6.00 usec
TE 300.0 K
D1 0.10000000 sec
======== CHANNEL f1 ========
NUC1 1H
P1 8.30 usec
PL1 -1.00 dB
SFO1 300.1312755 MHz
F2 - Processing parameters
SI 16384
SF 300.1299933 MHz
WDW EM
SSB 0
LB 0.10 Hz
GB 0
PC 0.80
1D NMR plot parameters
CX 22.00 cm
CY 25.67 cm
F1P 7.862 ppm
F1 2359.53 Hz
F2P -0.197 ppm
F2 -59.13 Hz
PPMCM 0.36631 ppm/cm
HZCM 109.93929 Hz/cm
13a
Integral
ppm

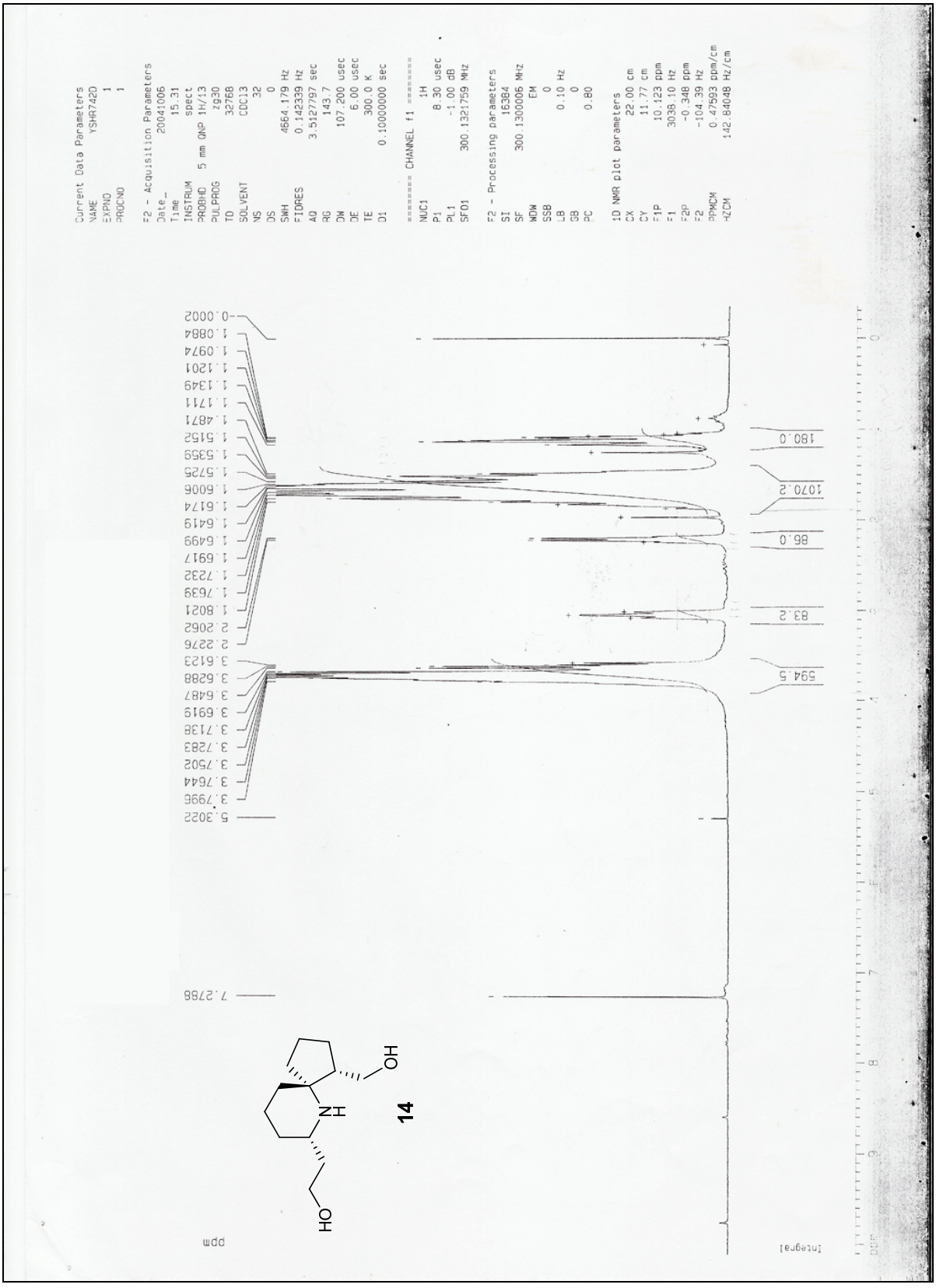

Current Data Parameters
NAME YSHA360enzene1
EXPNO 2
PROCNO 1
F2 - Acquisition Parameters
Date_ 20040707
Time 15.43
INSTRUM spect
PROBHD 5 mm QNP 1H/13
PULPROG zpgg
TD 65536
SOLVENT C6D6
NS 200
DS 0
SWH 17985.611 Hz
FIDRES 0.27439 Hz
AQ 1.8219508 sec
RG 14596.5
DW 27.800 usec
DE 20.00 usec
TE 300.0 K
D1 0.17593171 sec
d11 0.03000000 sec
d12 0.00002000 sec
======= CHANNEL f1 =======
NUC1 13C
P1 5.44 usec
PL1 4.00 dB
SFO1 75.4760973 MHz
======= CHANNEL f2 =======
CPDPRG2 waltz16
NUC2 1H
PCPD2 102.00 usec
PL2 120.00 dB
PL12 20.20 dB
PL13 25.80 dB
SFO2 300.1312005 MHz
F2 - Processing parameters
SI 32768
SF 75.4677208 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.00
1D NMR plot parameters
CX 22.00 cm
CY 17.78 cm
F1P 160.148 ppm
F1 12085.99 Hz
F2P -1.613 ppm
F2 -121.75 Hz
PPMCM 7.35277 ppm/cm
HZCM 554.89697 Hz/cm
146.645
128.842
128.736
128.456
128.343
128.022
127.701
127.024
126.114
70.038
65.328
64.001
50.362
47.357
42.636
41.727
37.309
33.307
30.140
28.489
21.492
20.679
18.691
5.123
ppm
140
120
100
80
60
40
20

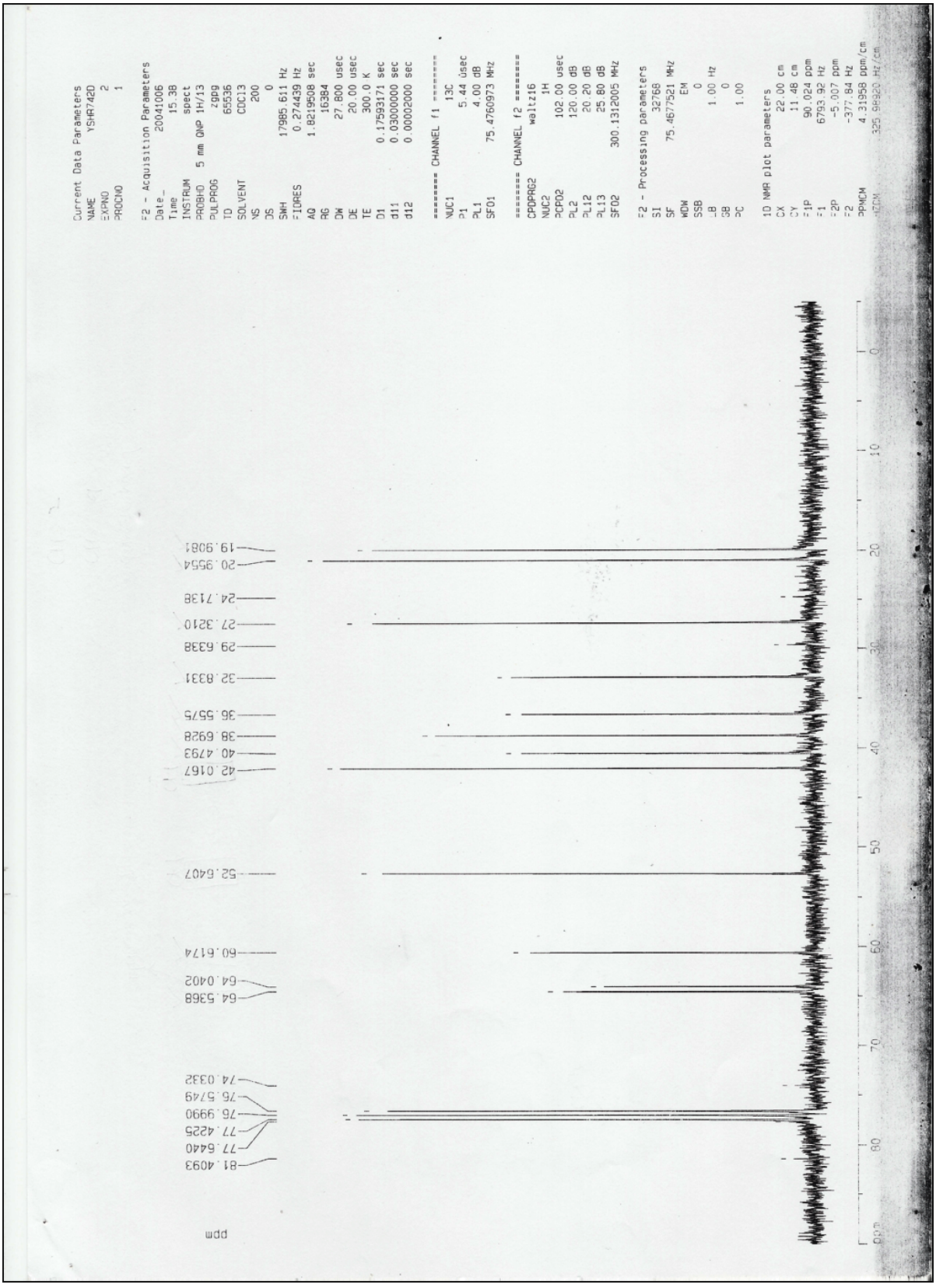

NAME N O cleave
EXPNO 1
PROCNO 1
Date 20050629
Time 10.47
INSTRUM spect
PROBHD 5 mm Dua. 13C/
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 16
DS 0
SWH 64102.56 Hz
FIDRES 1.955625 Hz
AQ 2.5559540 sec
RG 143.7
DW 78.000 usec
DE 6.50 usec
TE 300.0 K
D1 0.10000000 sec
CHANNEL f1
NUC1 1H
P1 10.00 usec
PL1 0.00 dB
SFO1 400.1330110 MHz
SI 16384
SF 400.1300071 MHz
WDW EM
SSB 0
LB 0.10 Hz
GB 0
PC 1.00
OH
NH
OH
EtO2C
13b
3.17
14.16
1.02
1.00
2.02
2.04
1.14
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
ppm

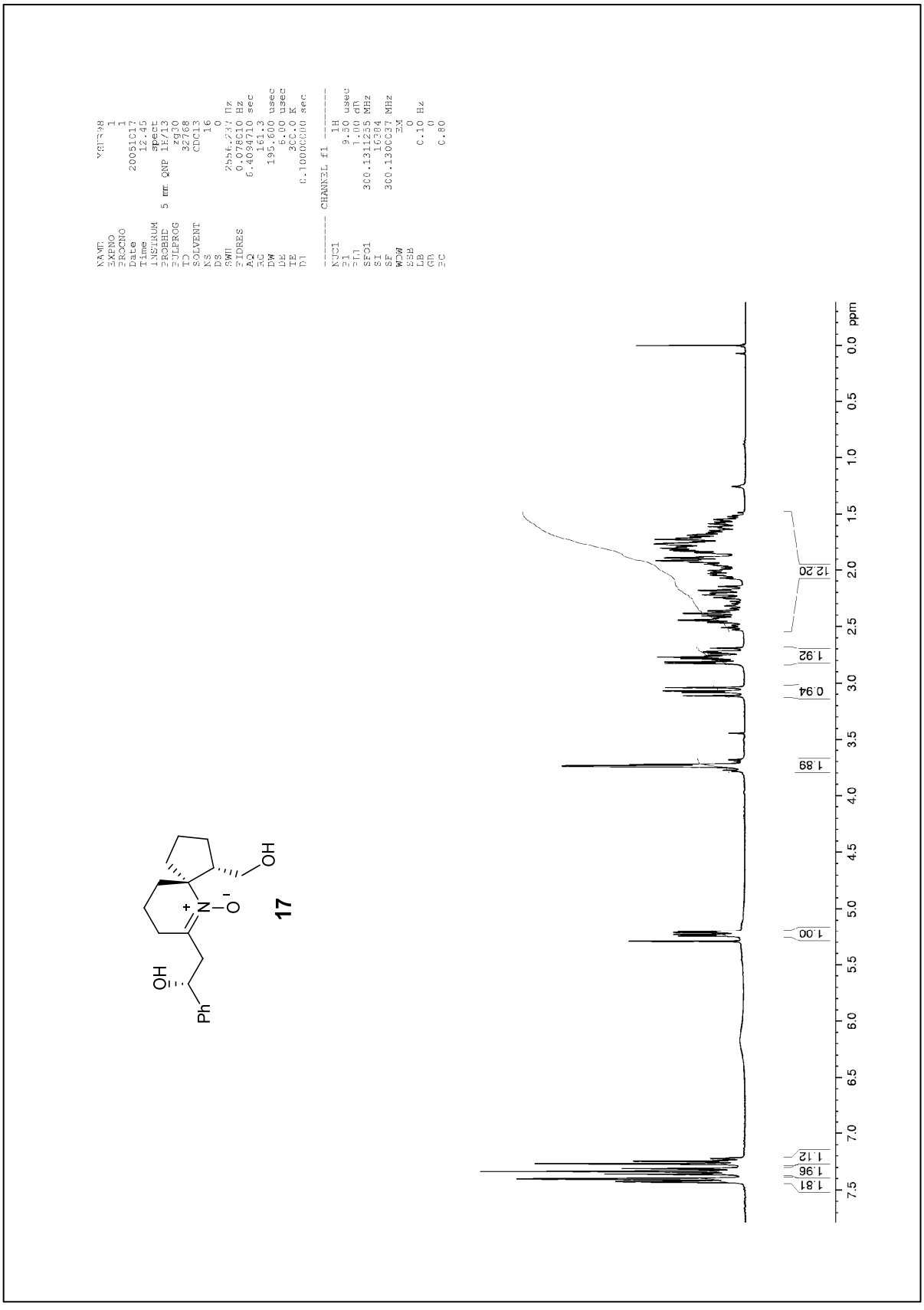

Current Data Parameters
NAME N-D cleavage
EXPNO 2
PROCNO 1
F2 - Acquisition Parameters
Date_ 20050629
Time 16:55
INSTRUM spect
PROBHD 5 mm Dual 13C/
PULPROG zgdc
TD 65536
SOLVENT CDCl3
NS 200
DS 0
SWH 24038.461 Hz
FIDRES 0.366998 Hz
AQ 1.3631968 sec
RG 11585.2
DW 20.800 usec
DE 6.00 usec
TE 300.0 K
D1 0.03000000 sec
D11 0.03000000 sec
D12 0.00002000 sec
======== CHANNEL f1 ========
NUC1 13C
P1 7.40 usec
PL1 -2.00 dB
SFO1 100.6378978 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 100.00 usec
PL2 0.00 dB
PL12 22.00 dB
PL13 22.00 dB
SFO2 400.1316006 MHz
F2 - Processing parameters
SI 32768
SF 100.6127754 MHz
WDW EM
SSB 0
LB 2.50 Hz
GB 0
PC 1.40
1D NMR plot parameters
CX 22.00 cm
CY 11.00 cm
F1P 71.798 ppm
F1 7221.54 Hz
F2P 0.000 ppm
F2 1061.31 Hz
PPMCM 3.76232 ppm/cm
HZCM 378.62640 Hz/cm
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0 ppm

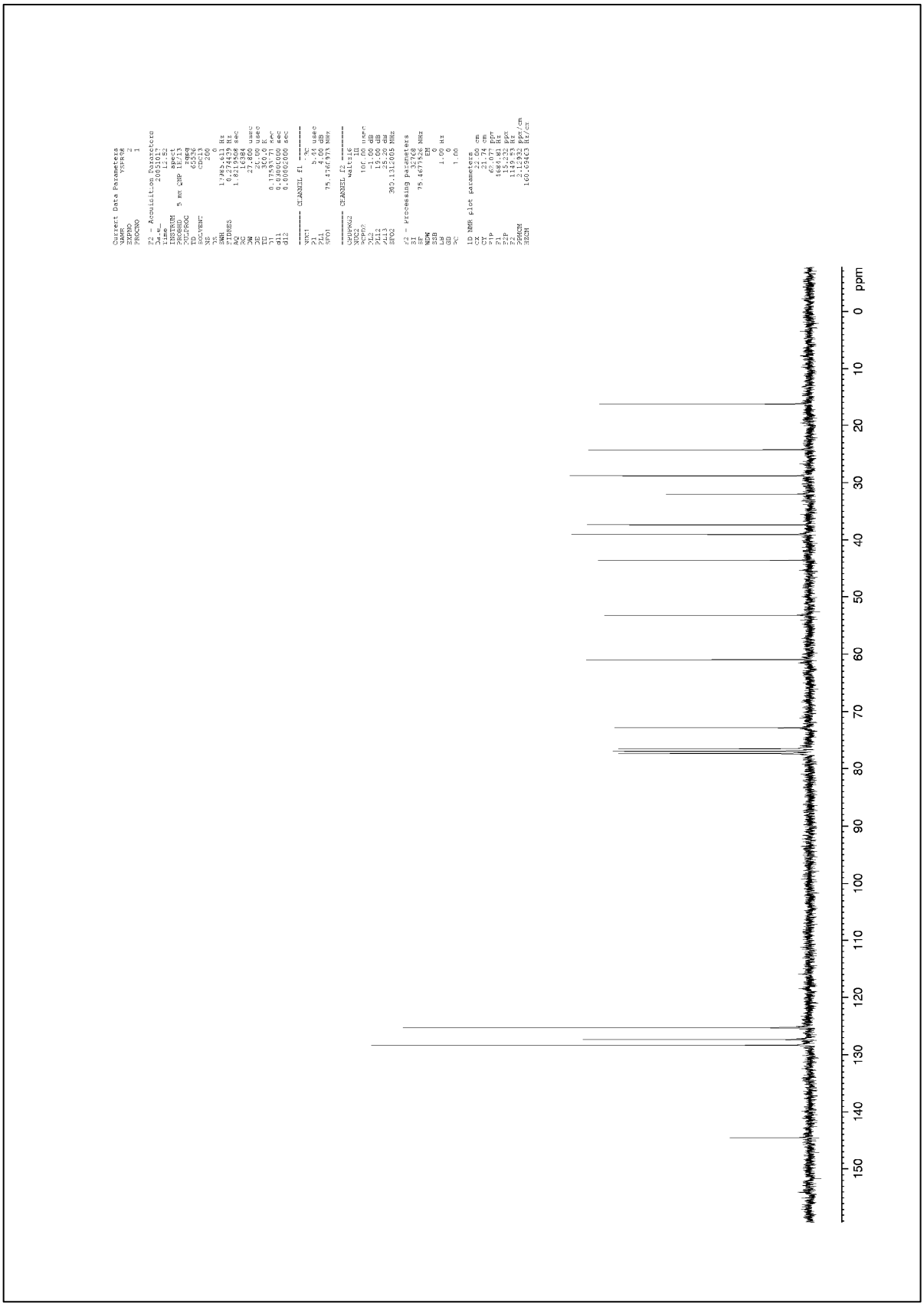

NAME YSH7405 hwnD
EXPNO 1
PROCNO 1
Date 20070112
Time 11.57
INSTRUM spect
PROBHD 5 mm QNP 1H/13
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 16
DS 0
SWH 4664.179 Hz
FIDRES 0.142339 Hz
AQ 3.5127797 sec
RG 90.5
DW 107.200 usec
DE 6.00 usec
TE 300.0 K
D1 0.10000000 sec
CHANNEL f1
NUC1 1H
P1 9.30 usec
PL1 0.00 dB
SFO1 300.1321739 MHz
SI 16384
SF 300.1299981 MHz
WDW EM
SSB 0
LB 0.10 Hz
GB 0
PC 0.80
OH
NH
OH
BzO
13d
1.96
1.00
2.00
4.55
2.09
1.00
1.03
15.12
3.12
ppm

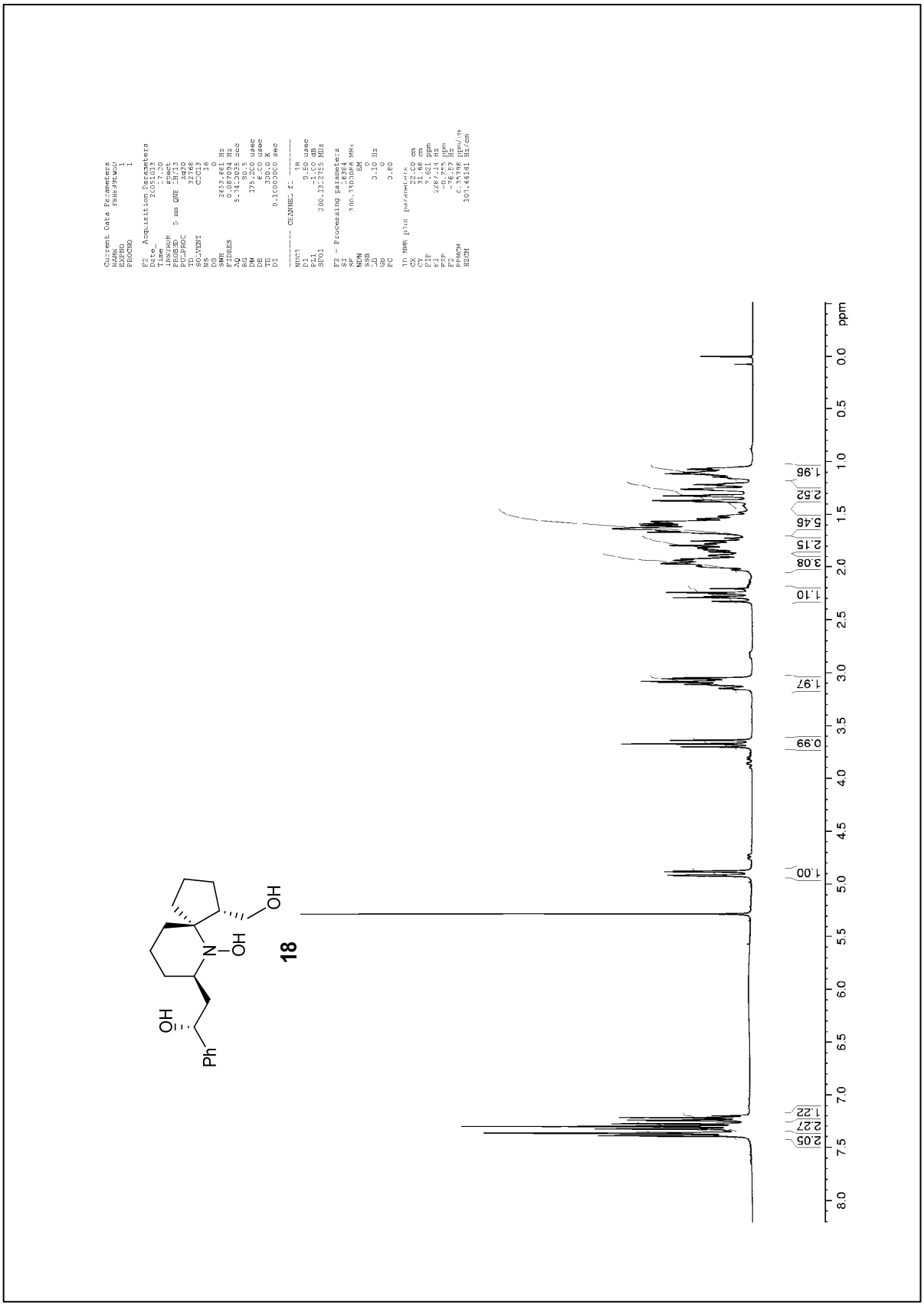

NAME YSHR405 two2
EXPNO 2
PROCNO 1
Date_ 20070112
Time 12.05
INSTRUM spect
PROBHD 5 mm QNP 1H/13
PULPROG zgpg
TD 65536
SOLVENT CDCl3
NS 200
DS 0
SWH 19980.414 Hz
FIDRES 0.304810 Hz
AQ 1.8219528 sec
RG 16384
DW 27.800 usec
DE 20.00 usec
TE 300.0 K
D1 0.17593171 sec
D11 0.03000000 sec
D12 0.00002000 sec
======== CHANNEL f1 ========
NUC1 13C
P1 5.44 usec
PL1 4.03 dB
SFO1 75.4769973 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 100.00 usec
PL2 0.00 dB
PL12 20.80 dB
PL13 26.40 dB
SFO2 300.1312005 MHz
SI 32768
SF 75.4677562 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.00
ppm

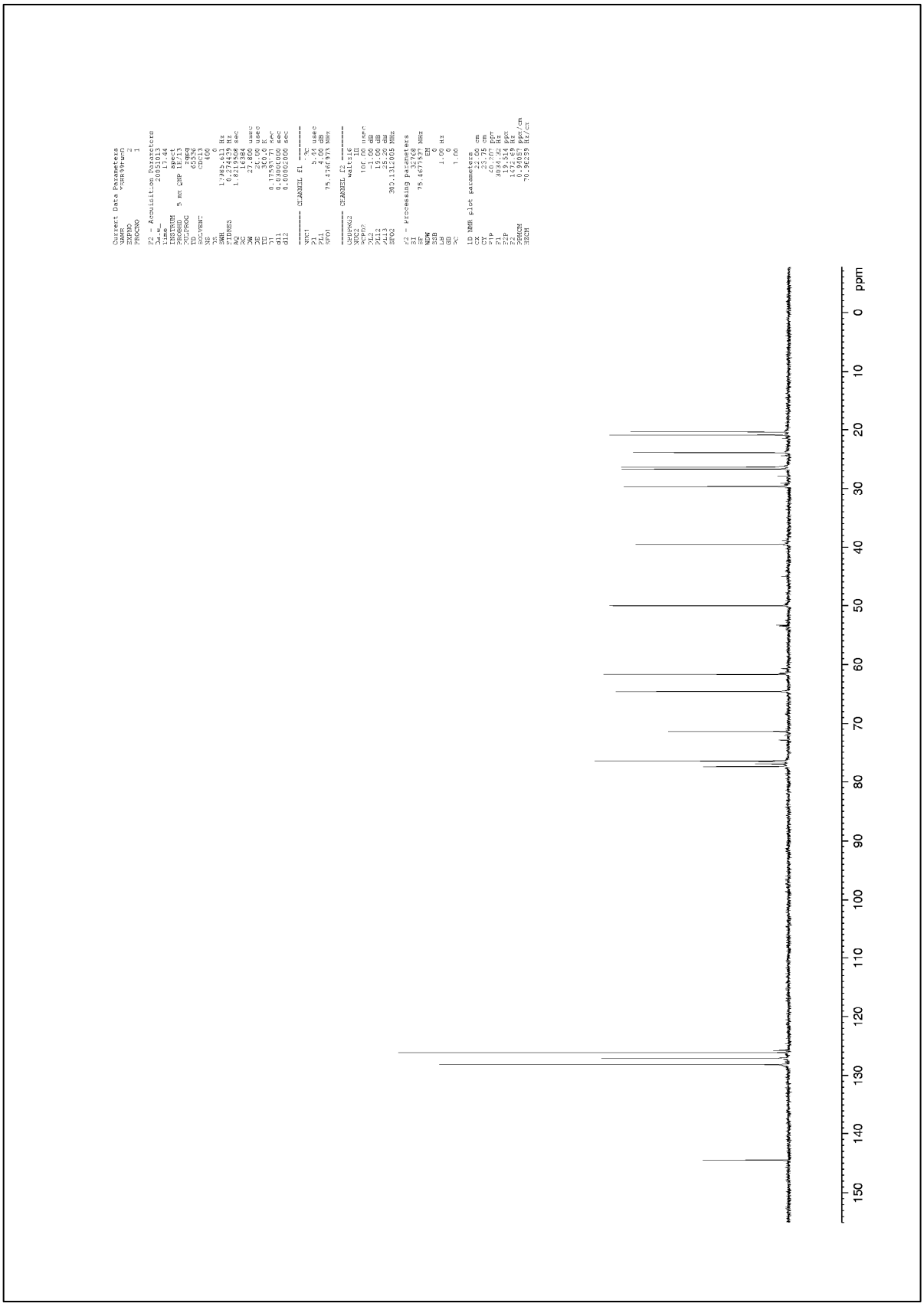

Current Data Parameters
NAME YSHR742D
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20041006
Time 15.31
INSTRUM spect
PROBHD 5 mm QNP 1H/13
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 32
DS 0
SWH 4564.179 Hz
FIDRES 0.142339 Hz
AQ 3.5127797 sec
RG 143.7
DW 107.200 usec
DE 6.00 usec
TE 300.0 K
D1 0.10000000 sec
======== CHANNEL f1 ========
NUC1 1H
P1 8.30 usec
PL1 -1.00 dB
SFO1 300.1321759 MHz
F2 - Processing parameters
SI 16384
SF 300.1300006 MHz
WDW EM
SSB 0
LB 0.10 Hz
GB 0
PC 0.80
1D NMR plot parameters
CX 22.00 cm
CY 11.77 cm
F1P 10.123 ppm
F1 3038.10 Hz
F2P -0.348 ppm
F2 -104.39 Hz
PPMCM 0.47593 ppm/cm
HZCM 142.84048 Hz/cm
7.2788
5.3022
3.7996
3.7644
3.7502
3.7283
3.7138
3.6919
3.6487
3.6288
3.6123
2.2276
2.2062
1.8021
1.7639
1.7232
1.6917
1.6499
1.6419
1.6174
1.6006
1.5725
1.5359
1.5152
1.4871
1.1711
1.1349
1.1201
1.0974
1.0884
1.0002
180.0
1070.2
86.0
83.2
594.5
OH
N
H
HO
14
ppm
Integral

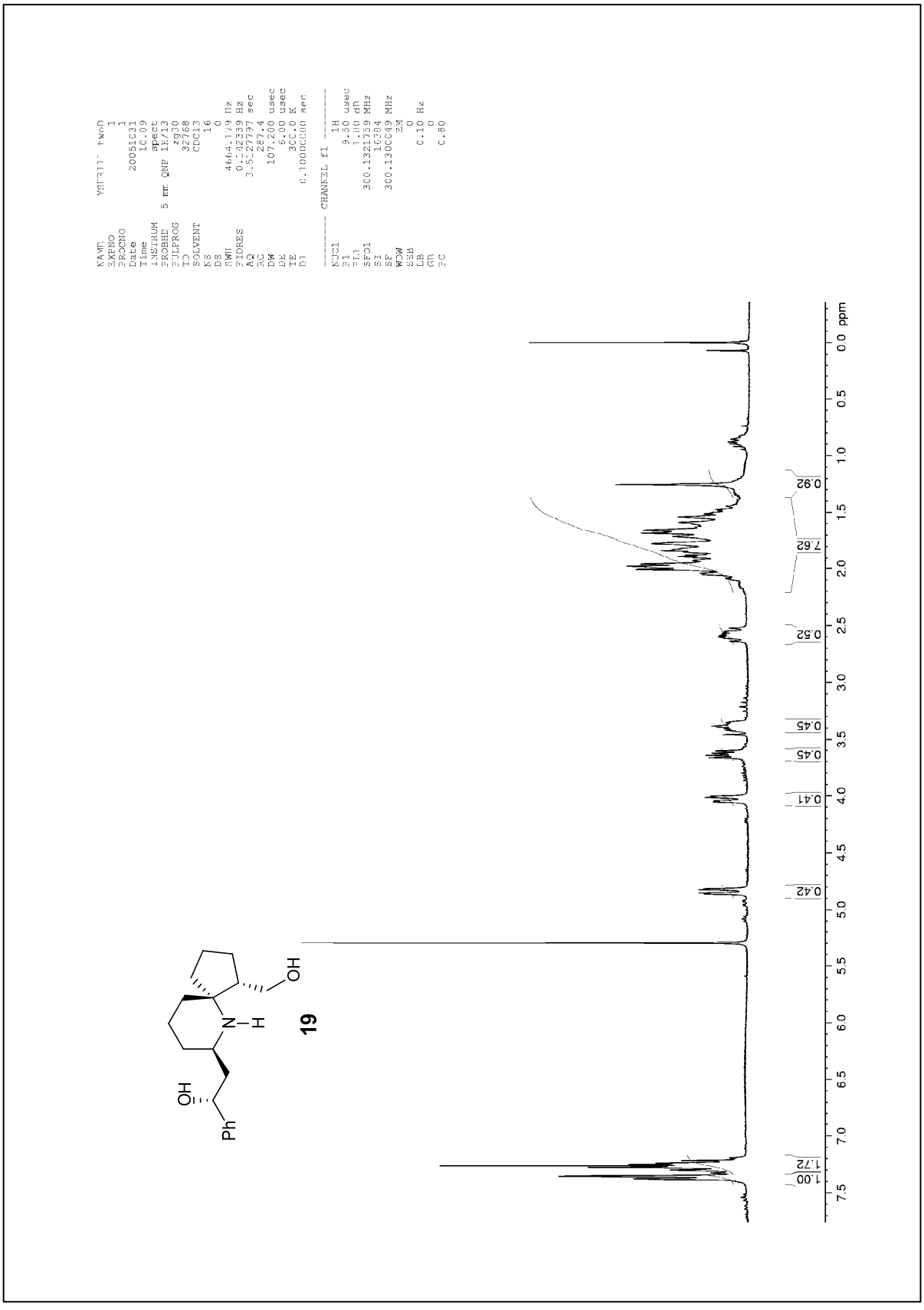

Current Data Parameters
NAME YSMR742D
EXPNO 2
PROCNO 1
F2 - Acquisition Parameters
Date_ 20041006
Time 15.38
INSTRUM spect
PROBHD 5 mm QNP 1H/13
PULPROG zgpg
TD 65536
SOLVENT CDCl3
NS 200
DS 0
SWH 17985.611 Hz
FIDRES 0.274439 Hz
AQ 1.8219508 sec
RG 16384
DW 27.800 usec
DE 20.00 usec
TE 300.0 K
D1 0.17593171 sec
d11 0.03000000 sec
d12 0.00002000 sec
======== CHANNEL f1 ========
NUC1 13C
P1 5.44 usec
PL1 4.00 dB
SFO1 75.4760973 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 102.00 usec
PL2 120.00 dB
PL12 20.20 dB
PL13 25.80 dB
SFO2 300.1312005 MHz
F2 - Processing parameters
SI 32768
SF 75.4677521 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.00
1D NMR plot parameters
CX 22.00 cm
CY 11.48 cm
F1P 90.024 ppm
F1 6793.92 Hz
F2P -5.007 ppm
F2 -377.84 Hz
PPMCM 4.31958 ppm/cm
HZCM -325.88920 Hz/cm
19.9081
20.9554
24.7138
27.3210
29.6338
32.8331
36.5575
38.6928
40.4793
42.0167
52.6407
60.6174
64.0402
64.5368
74.0332
76.5749
76.9990
77.4225
77.6440
81.4093
ppm

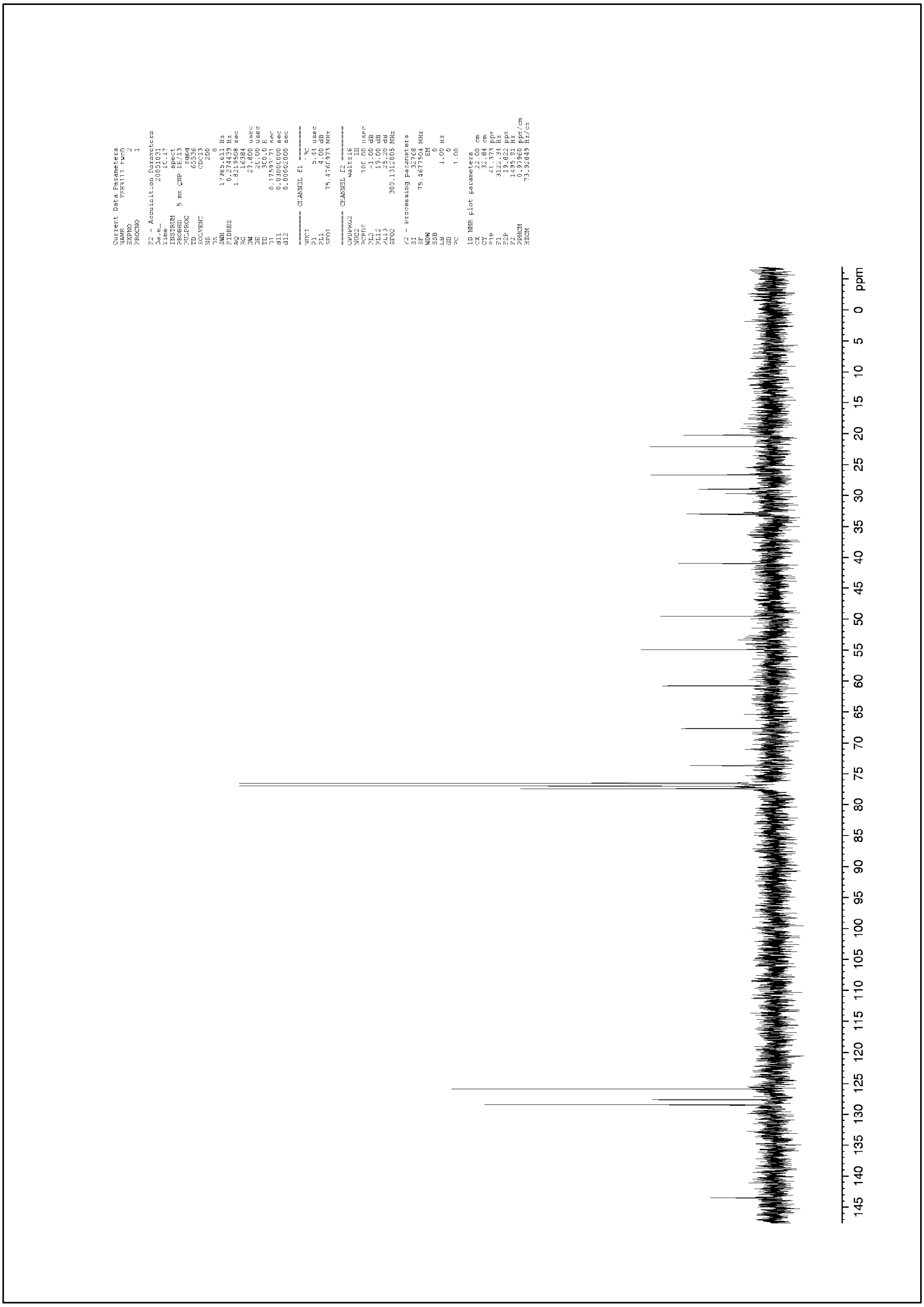

17
OH
OH
Ph
1.81
1.96
1.12
1.00
1.89
0.94
1.92
12.20
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0 ppm

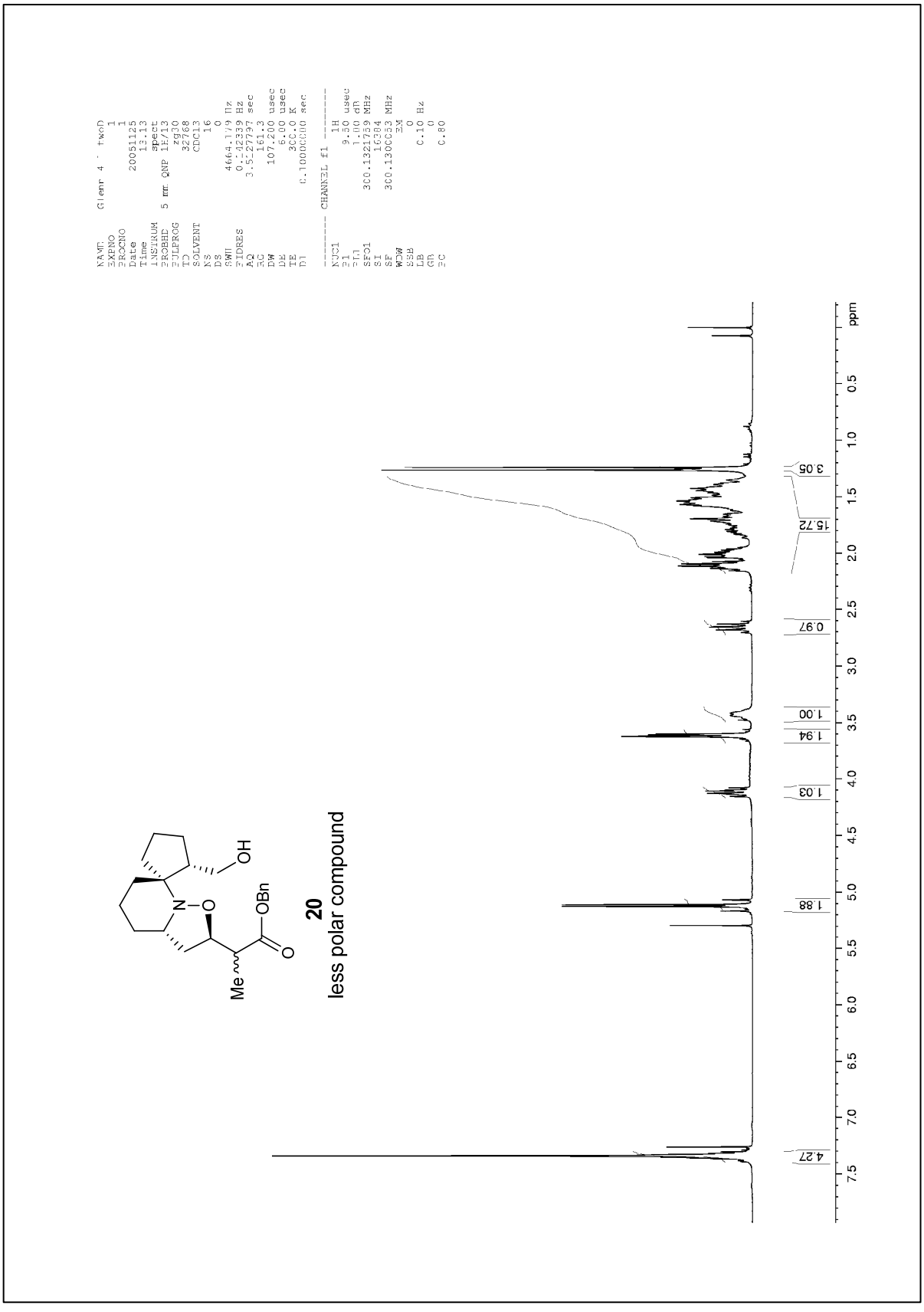

Current Data Parameters
NAME YSFR38
EXPNO 2
PROCNO 1
F2 - Acquisition Parameters
Date_ 20051017
Time 12:52
INSTRUM spect
PROBHD 5 mm QNP 1H/13
PULPROG zgdc
TD 65536
SOLVENT CDCl3
NS 200
DS 0
SWH 17985.611 Hz
FIDRES 0.274459 Hz
AQ 1.8217600 sec
RG 16384
DW 27.800 usec
DE 6.00 usec
TE 300.0 K
D1 0.1700000 sec
d11 0.0300000 sec
d12 0.0000200 sec
CHANNEL f1
NUC1 13C
P1 4.00 usec
PL1 4.00 dB
SFO1 75.4767973 MHz
CHANNEL f2
CPDPRG2 waltz16
NUC2 1H
PCPD2 100.00 usec
PL2 -1.00 dB
PL12 16.50 dB
PL13 25.20 dB
SFO2 300.1312005 MHz
F2 - Processing parameters
SI 32768
SF 75.4677956 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.00
1D NMR plot parameters
CX 22.00 cm
CY 21.14 cm
F1P 6.077 ppm
F1 458.91 Hz
F2P 150.221 ppm
F2 11429.53 Hz
PPMCM 2.11923 ppm/cm
HZCM 317.51415 Hz/cm
ppm

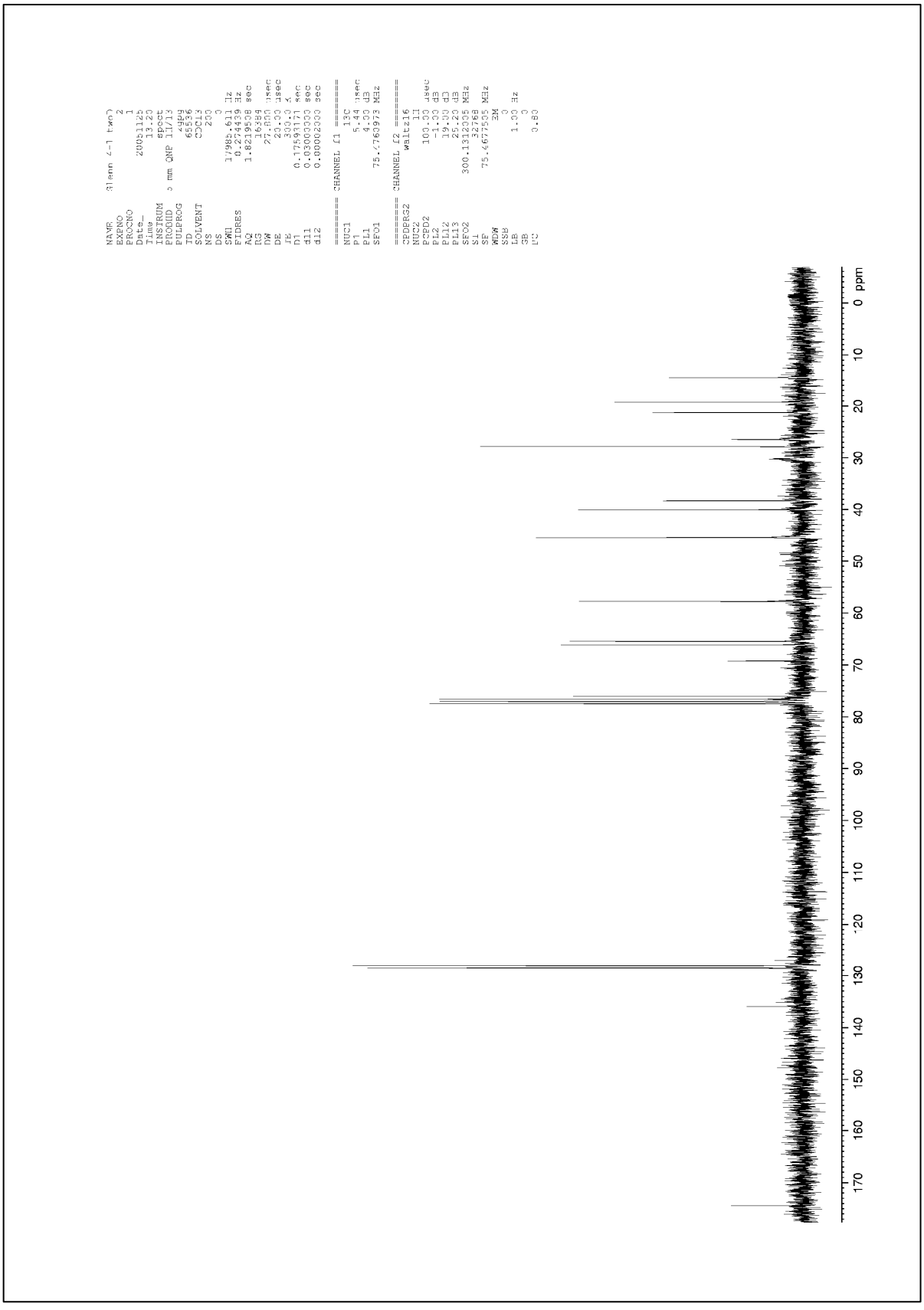

18
OH
N
OH
OH
Ph
1.00
0.99
1.97
1.10
3.08
2.15
5.46
2.52
1.96
2.05
2.27
1.22
ppm

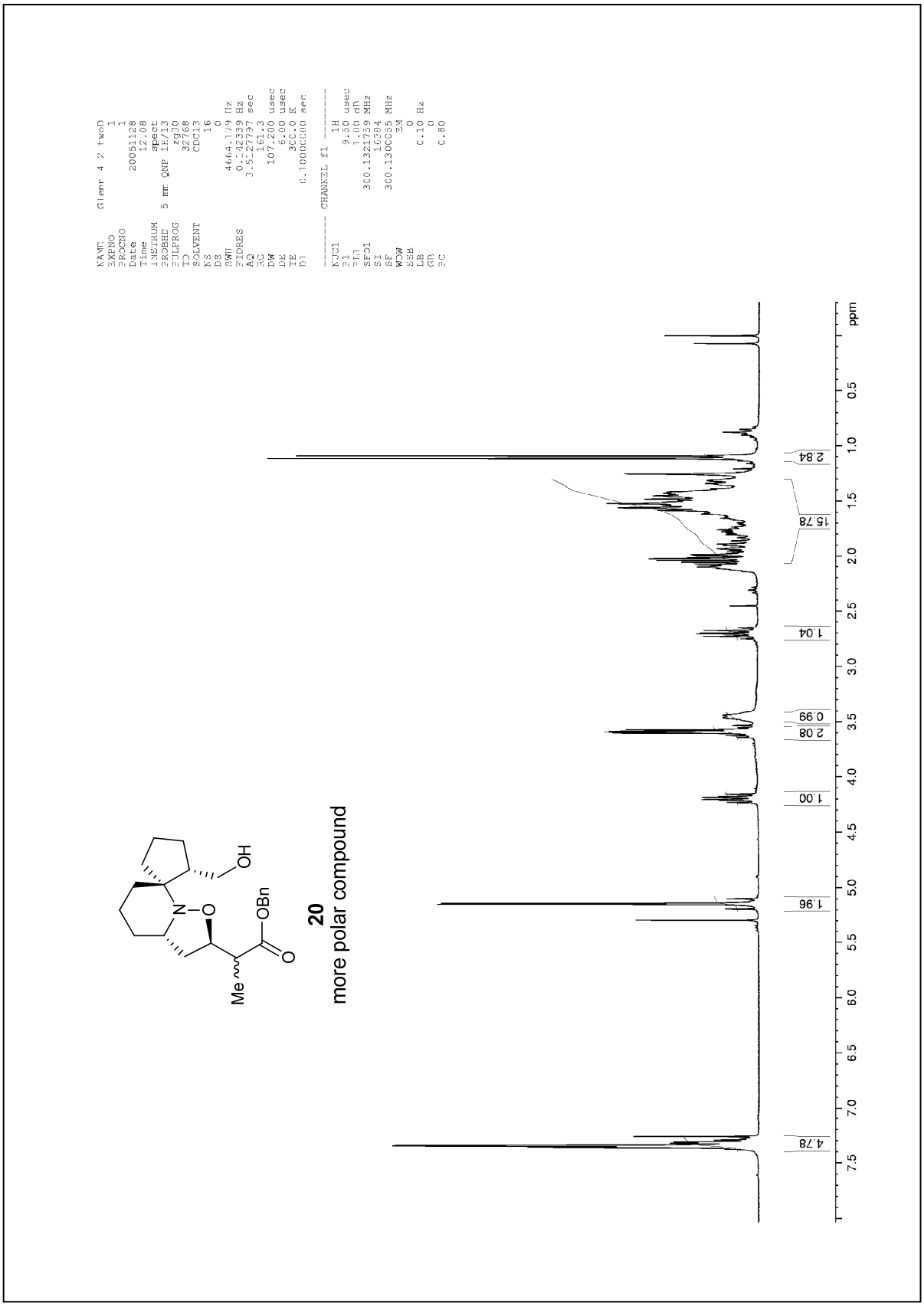

Current Data Parameters
NAME ysnk9yrun
EXPNO 2
PROCNO 1
F2 - Acquisition Parameters
Date_ 20051013
Time 17:44
INSTRUM spect
PROBHD 5 mm QNP 1H/13
PULPROG zgdc
TD 65536
SOLVENT CDCl3
NS 400
DS 0
SWH 17985.611 Hz
FIDRES 0.274509 Hz
AQ 1.8217960 sec
RG 16384
DW 27.800 usec
DE 6.00 usec
TE 300.0 K
D1 0.1700000 sec
d11 0.0300000 sec
d12 0.0000200 sec
CHANNEL f1
NUC1 13C
P1 9.00 usec
PL1 4.00 dB
SFO1 75.4767973 MHz
CHANNEL f2
CPDPRG2 waltz16
NUC2 1H
PCPD2 100.00 usec
PL2 -1.00 dB
PL12 15.50 dB
PL13 25.20 dB
SFO2 300.1312005 MHz
F2 - Processing parameters
SI 32768
SF 75.4677957 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.00
1D NMR plot parameters
CX 22.00 cm
CY 23.35 cm
F1P 40.207 ppm
F1 3034.34 Hz
F2P 0.394 ppm
F2 177.26 Hz
PPMCM 0.94057 ppm/cm
HZCM 70.91239 Hz/cm
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
ppm

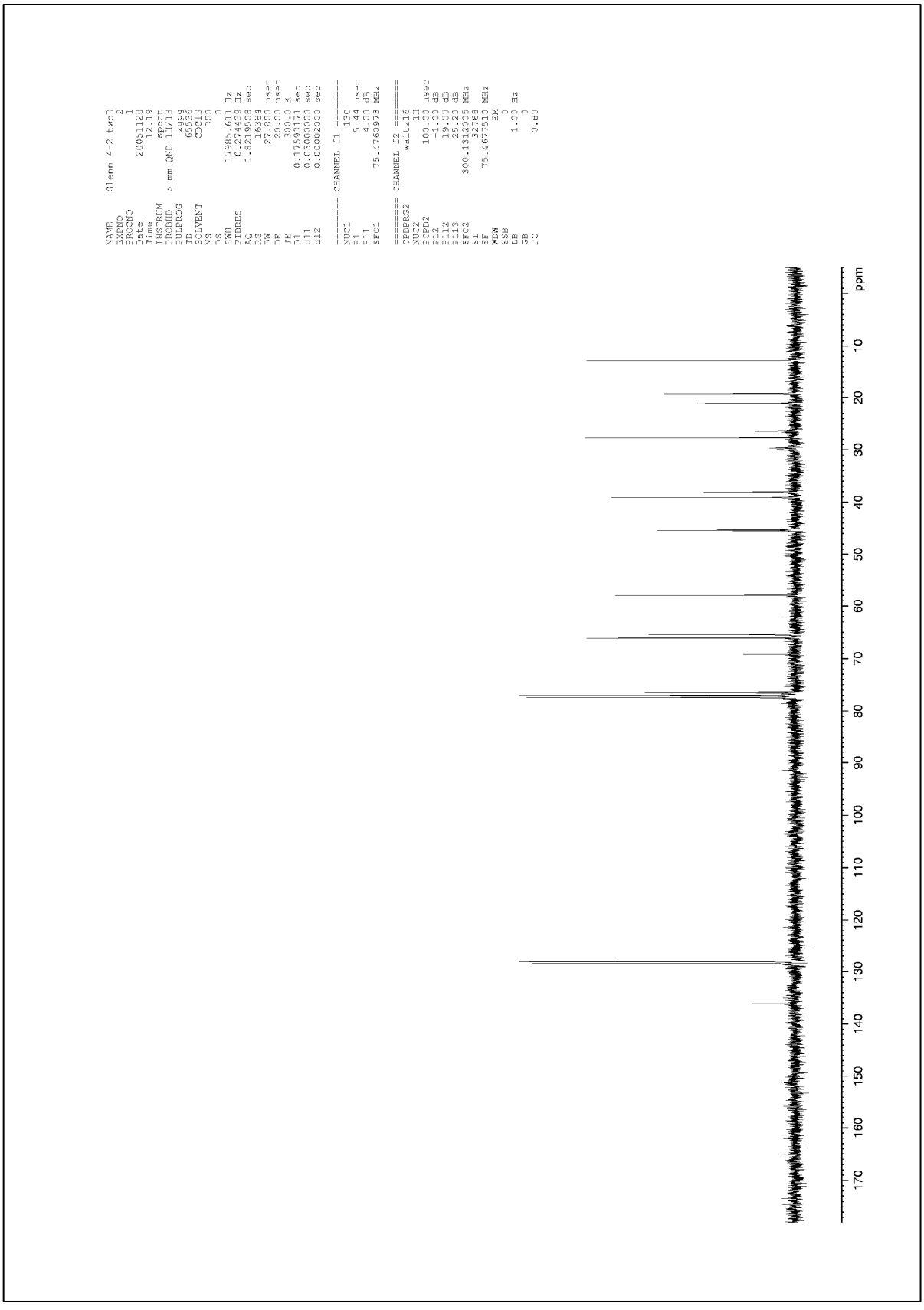

19
OH
N
H
OH
Ph
1.00
1.72
0.42
0.41
0.45
0.45
0.52
7.62
0.92
0.0 ppm

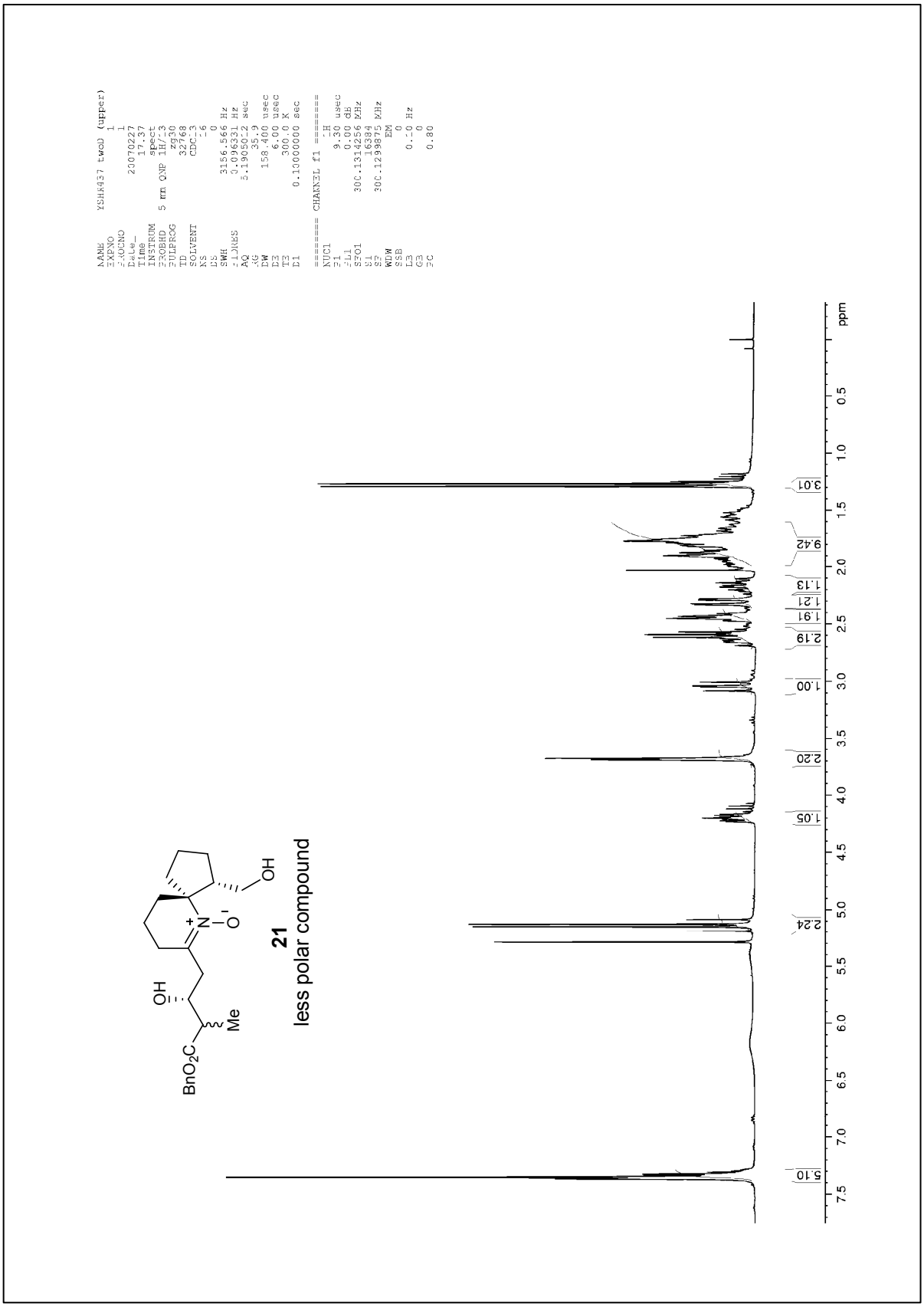

ppm
0 5 10 15 20 25 30 35 40 45 50 55 60 65 70 75 80 85 90 95 100 105 110 115 120 125 130 135 140 145

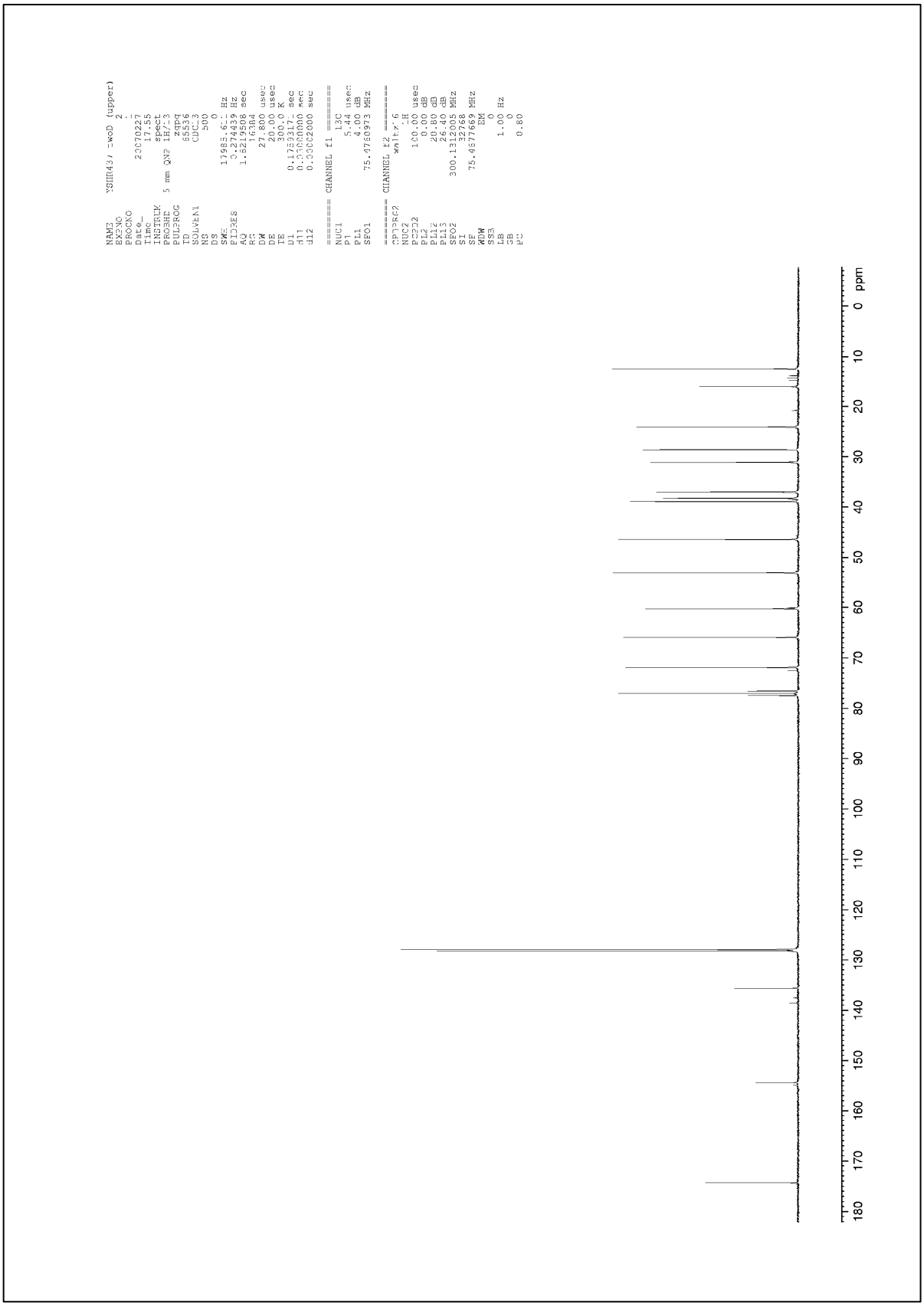

NAME Glenn 4 - tword
EXPNO 1
PROCNO 1
Date 20051125
Time 13.13
INSTRUM spect
PROBHD 5 mm QNP 1E/13
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 16
DS 0
SWH 4664.179 Hz
FIDRES 0.142339 Hz
AQ 3.5127797 sec
RG 161.3
DW 107.200 usec
DE 6.00 usec
TE 300.0 K
D1 0.10000000 sec
CHANNEL f1
NUC1 1H
P1 9.30 usec
PL1 1.10 dB
SFO1 300.1321753 MHz
SI 16384
SF 300.1300033 MHz
WDW EM
SSB 0
LB 0.10 Hz
GB 0
PC 0.80
OH
OBn
N
O
O
Me
20
less polar compound
4.27
1.88
1.03
1.94
1.00
0.97
15.72
3.05
ppm

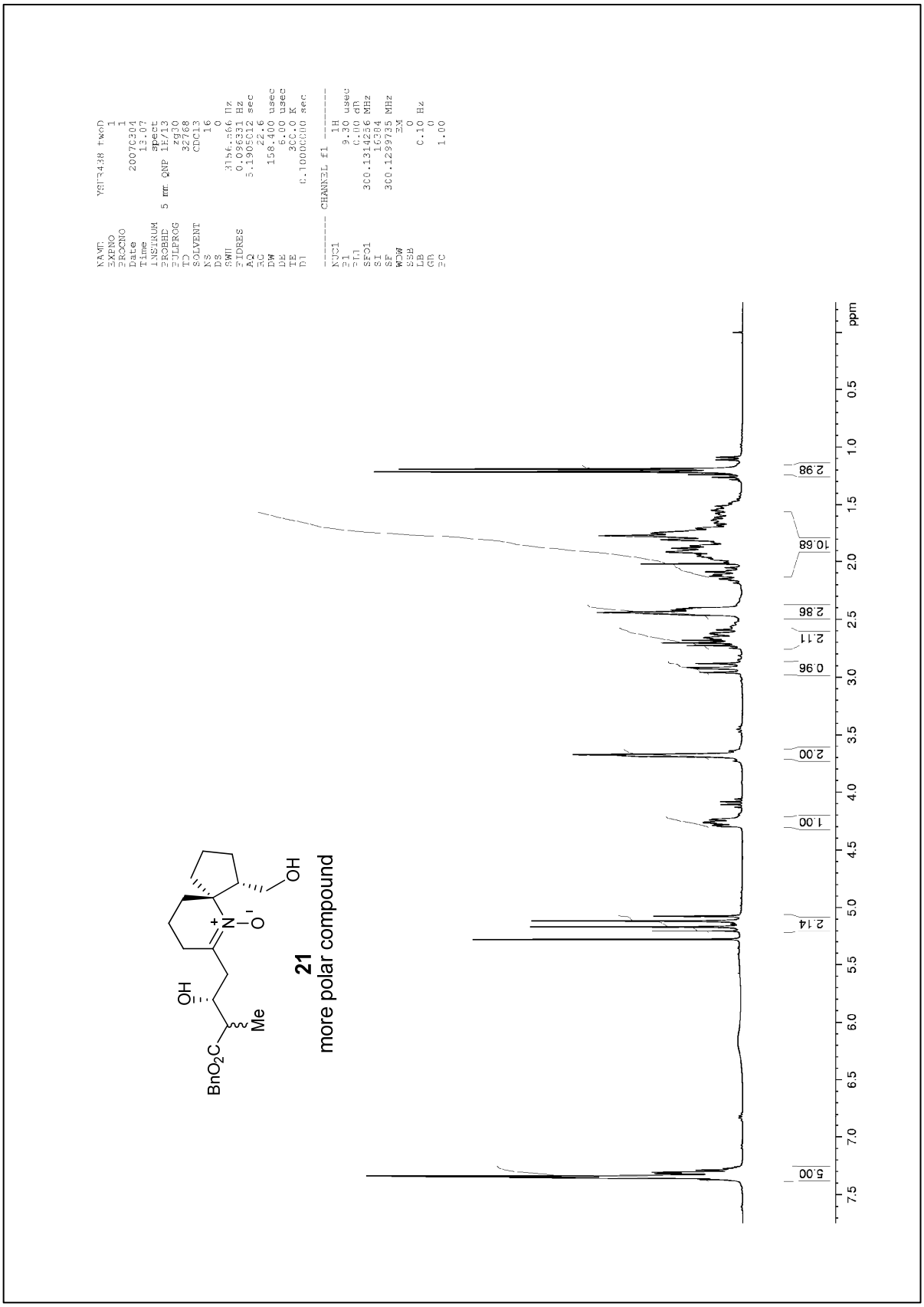

NAME Eilenn 4-1 two2
EXPNO 2
PROCNO 1
Date_ 20081125
Time 13:20
INSTRUM spect
PROBHD 5 mm QNP 1H/13
PULPROG zgpg
TD 65536
SOLVENT CDCl3
NS 200
DS 0
SWH 19980.414 Hz
FIDRES 0.304938 Hz
AQ 1.8219456 sec
RG 16384
DW 27.800 usec
DE 20.00 usec
TE 300.0 K
D1 0.17593171 sec
d11 0.03000000 sec
d12 0.00002000 sec
======== CHANNEL f1 ========
NUC1 13C
P1 5.44 usec
PL1 4.03 dB
SFO1 75.4769973 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 100.00 usec
PL2 -1.00 dB
PL12 19.00 dB
PL13 25.20 dB
SFO2 300.1312005 MHz
SI 32768
SF 75.4677555 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 0.80
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0 ppm


NAME Glenn 4 2 two0
EXPNO 1
PROCNO 1
Date 20051128
Time 12.08
INSTRUM spect
PROBHD 5 mm QNP 1H/13
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 16
DS 0
SWH 4664.179 Hz
FIDRES 0.142339 Hz
AQ 3.5127797 sec
RG 161.3
DW 107.200 usec
DE 6.00 usec
TE 300.0 K
D1 0.10000000 sec
CHANNEL f1
NUC1 1H
P1 9.30 usec
PL1 1.10 dB
SFO1 300.1321753 MHz
SI 16384
SF 300.1300035 MHz
WDW EM
SSB 0
LB 0.10 Hz
GB 0
PC 0.80
OH
OBn
N
O
O
Me
20
more polar compound
4.78
1.96
1.00
2.08
0.99
1.04
15.78
2.84
ppm

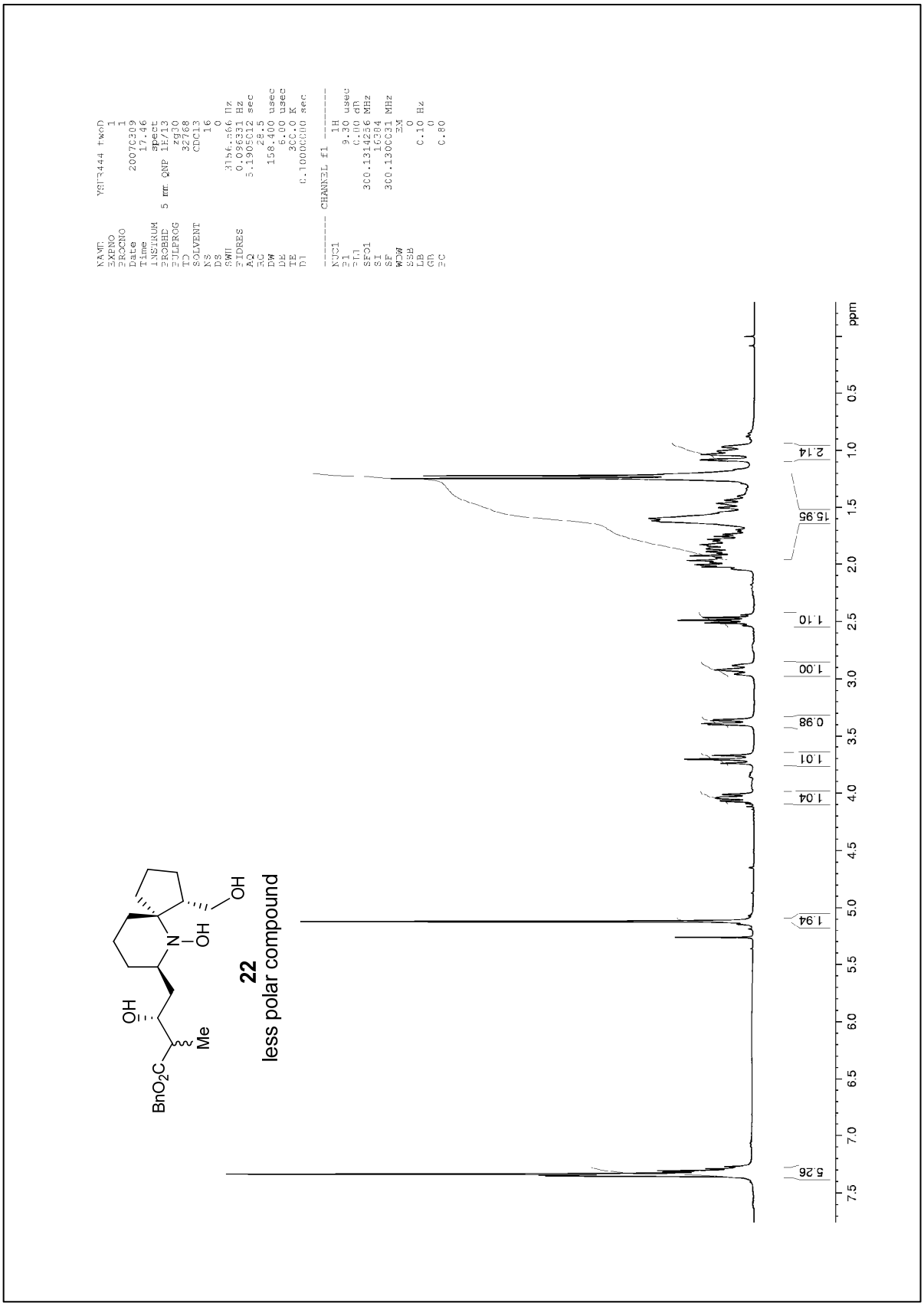

NAME Eilenn 4-2 two2
EXPNO 2
PROCNO 1
Date_ 20081128
Time 12.19
INSTRUM spect
PROBHD 5 mm QNP 1H/13
PULPROG zgpg
TD 65536
SOLVENT CDCl3
NS 300
DS 0
SWH 19980.414 Hz
FIDRES 0.304898 Hz
AQ 1.8219456 sec
RG 16384
DW 27.800 usec
DE 20.00 usec
TE 300.0 K
D1 0.17593171 sec
D11 0.03000000 sec
D12 0.00002000 sec
CHANNEL f1
NUC1 13C
P1 5.44 usec
PL1 4.03 dB
SFO1 75.4769973 MHz
CHANNEL f2
CPDPRG2 waltz16
NUC2 1H
PCPD2 100.00 usec
PL2 -1.00 dB
PL12 19.00 dB
PL13 25.20 dB
SFO2 300.1312005 MHz
SI 32768
SF 75.4677510 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 0.80
ppm


NAME YSHK497 two0 (upper)
EXPNO 1
PROCNO 1
Date_ 20070227
Time 17.37
INSTRUM spect
PROBHD 5 mm QNP 1H/13
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 8
DS 0
SWH 3156.566 Hz
FIDRES 0.096331 Hz
AQ 5.190502 sec
RG 35.9
DW 158.400 usec
DE 6.00 usec
TE 300.0 K
D1 0.10000000 sec
======== CHANNEL f1 ========
NUC1 1H
P1 9.30 usec
PL1 0.00 dB
SFO1 300.1314256 MHz
SI 16384
SF 300.1299875 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 0.80
OH
N
O
OH
Me
BnO2C
21
less polar compound
5.10
2.24
1.05
2.20
1.00
2.19
1.91
1.21
1.13
9.42
3.01
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
ppm

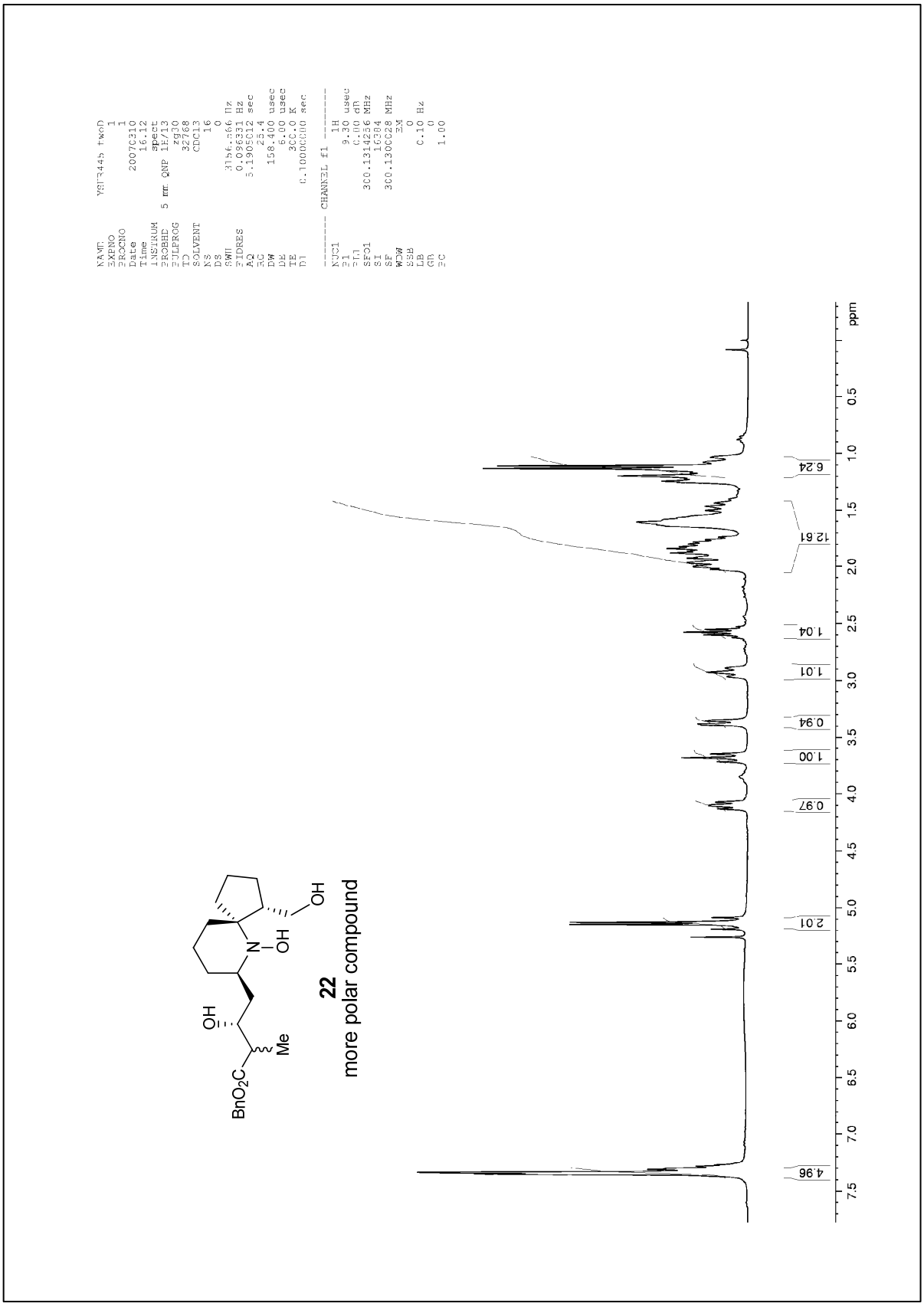

NAME YSHH431-new5 (upper)
EXPNO 2
PROCNO 1
Date_ 20070227
Time 17.55
INSTRUM spect
PROBHD 5 mm QNP 1H/13
PULPROG zgpg
TD 65536
SOLVENT CDCl3
NS 500
DS 0
SWH 17985.6 Hz
FIDRES 0.274439 Hz
AQ 1.8219508 sec
RG 16384
DW 27.800 usec
DE 20.00 usec
TE 300.0 K
D1 0.17533317 sec
D11 0.03000000 sec
D12 0.00002000 sec
======== CHANNEL f1 ========
NUC1 13C
P1 5.44 usec
PL1 4.00 dB
SFO1 75.4780973 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 100.00 usec
PL2 0.00 dB
PL12 20.80 dB
PL13 26.40 dB
SFO2 300.1320005 MHz
SI 32768
SF 75.4677669 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 0.80
180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

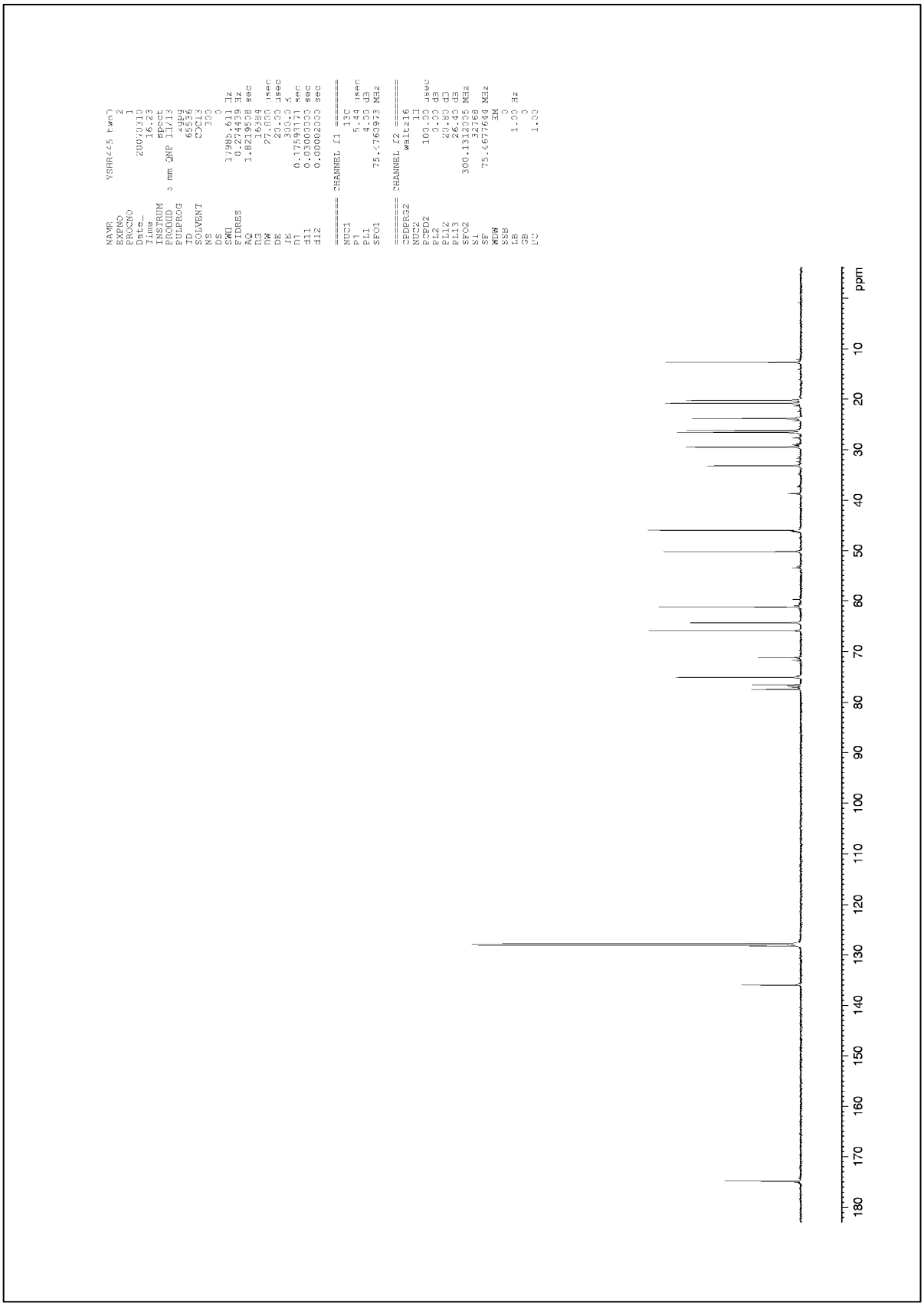

21
more polar compound
OH
OH
Me
BnO2C
2.98
10.68
2.86
2.11
0.96
2.00
1.00
2.14
5.00
ppm

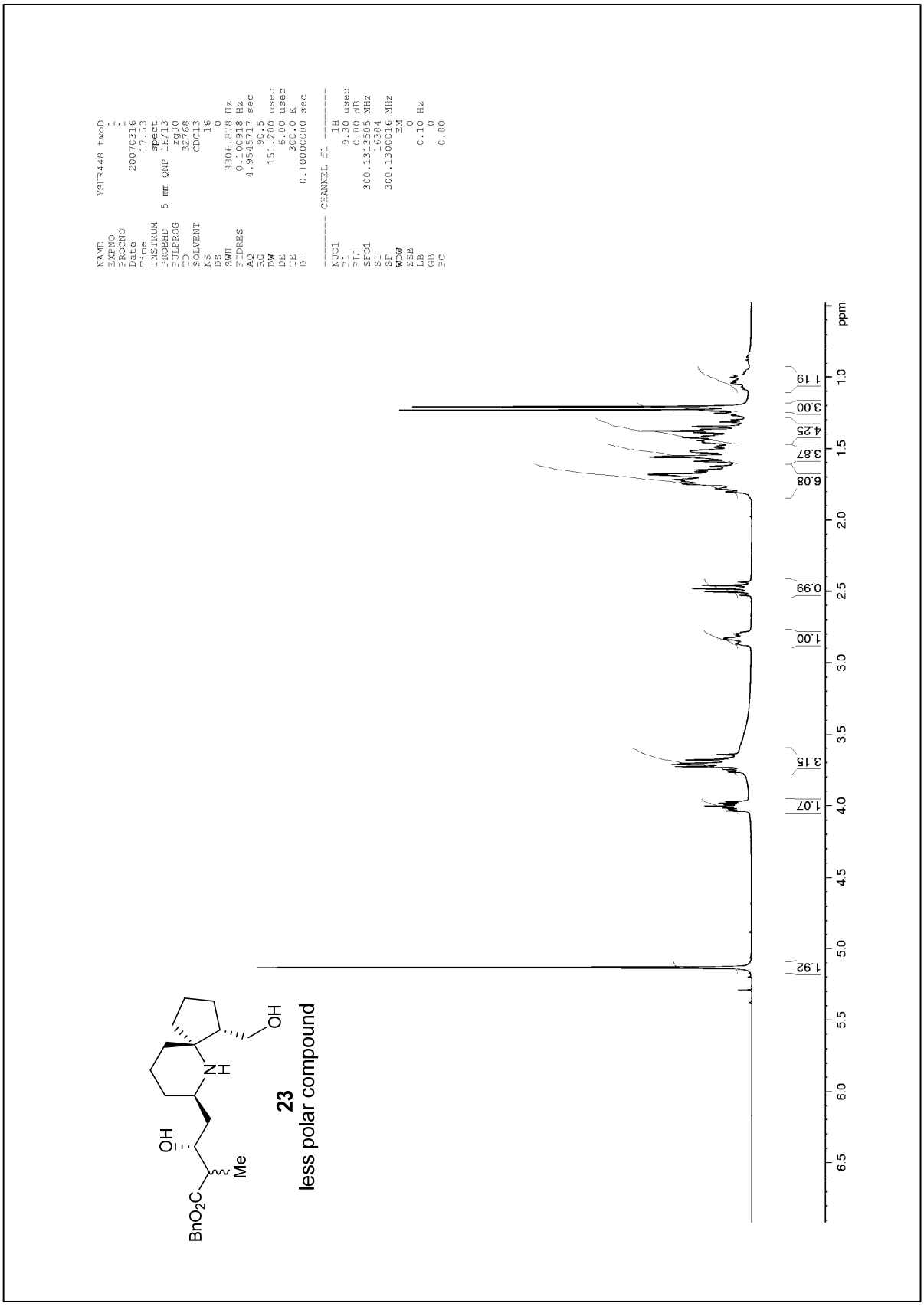

NAME YSHR438 two2
EXPNO 2
PROCNO 1
Date_ 20070304
Time 13:21
INSTRUM spect
PROBHD 5 mm QNP 1H/13
PULPROG zgpg
TD 65536
SOLVENT CDCl3
NS 400
DS 0
SWH 11990.411 Hz
FIDRES 0.182948 Hz
AQ 1.8219456 sec
RG 16384
DW 27.800 usec
DE 20.00 usec
TE 300.0 K
D1 0.17593171 sec
D11 0.03000000 sec
D12 0.00002000 sec
======== CHANNEL f1 ========
NUC1 13C
P1 5.44 usec
PL1 4.03 dB
SFO1 75.4769973 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 100.00 usec
PL2 0.00 dB
PL12 20.80 dB
PL13 26.40 dB
SFO2 300.1312005 MHz
SI 32768
SF 75.4677782 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 0.80
ppm


22
less polar compound
BnO2C
Me
OH
OH
N
OH
5.26
1.94
1.04
1.01
0.98
1.00
1.10
15.95
2.14
ppm

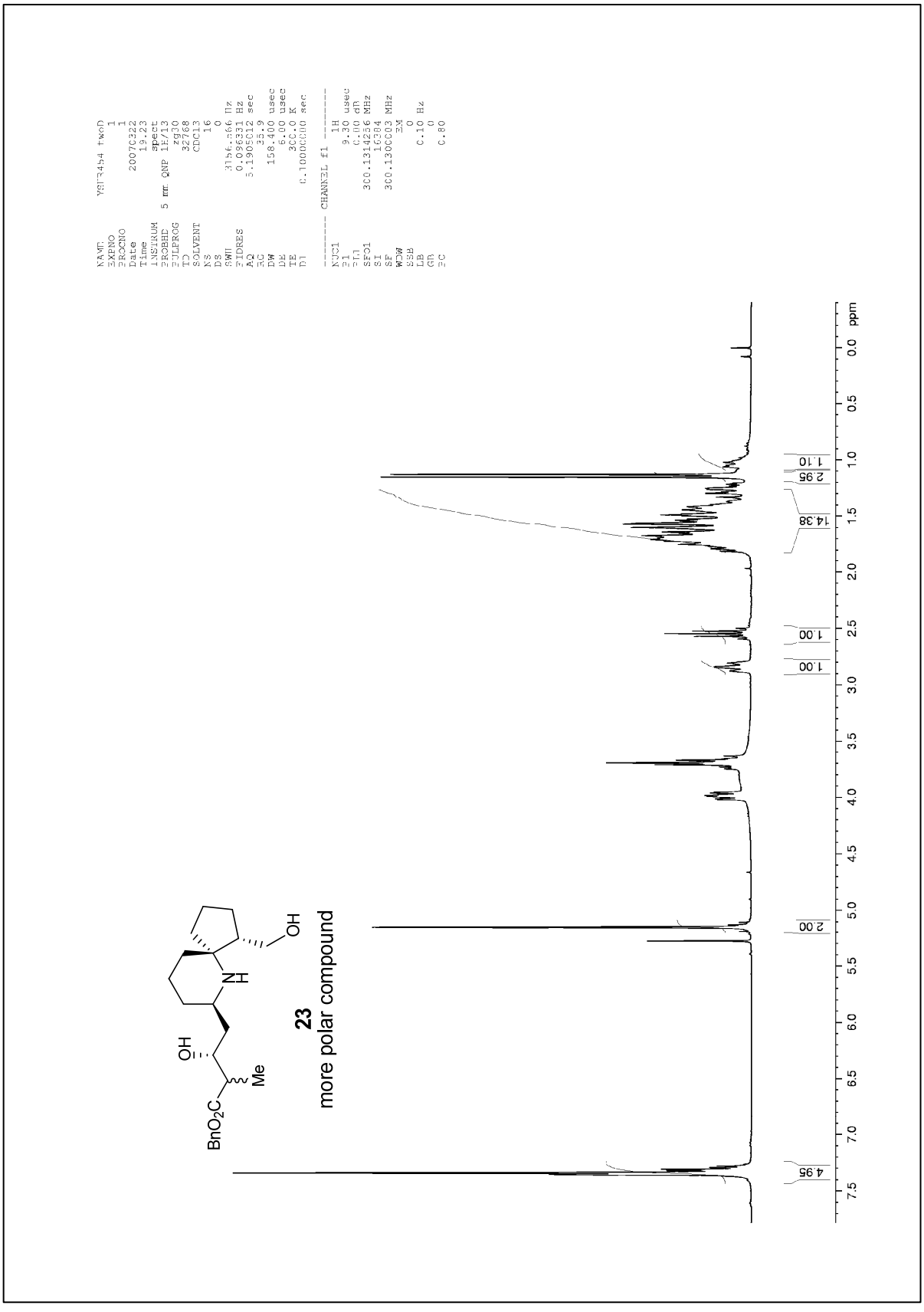

NAME YSHR477 two2
EXPNO 2
PROCNO 1
Date_ 20070309
Time 17.57
INSTRUM spect
PROBHD 5 mm QNP 1H/13
PULPROG zgpg
TD 65536
SOLVENT CDCl3
NS 300
DS 0
SWH 11990.419 Hz
FIDRES 0.182948 Hz
AQ 1.8219456 sec
RG 16384
DW 27.800 usec
DE 20.00 usec
TE 300.0 K
D1 0.17593171 sec
D11 0.03000000 sec
D12 0.00002000 sec
======== CHANNEL f1 ========
NUC1 13C
P1 5.44 usec
PL1 4.03 dB
SFO1 75.4769973 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 100.00 usec
PL2 0.00 dB
PL12 20.80 dB
PL13 26.40 dB
SFO2 300.1312005 MHz
SI 32768
SF 75.4677629 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 0.80
ppm

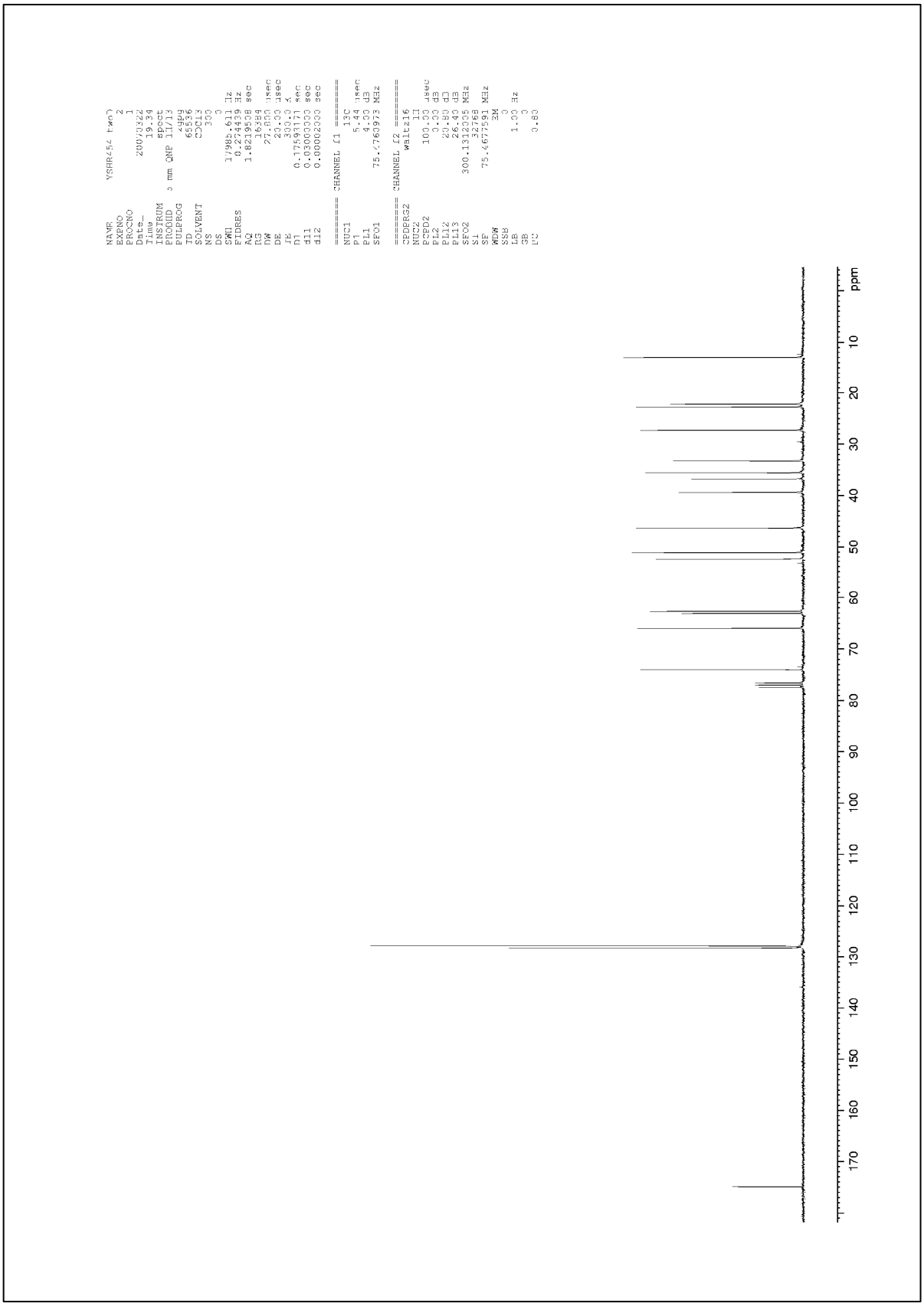

NAME YSH7445 fwoD
EXPNO 1
PROCNO 1
Date 20070310
Time 16.12
INSTRUM spect
PROBHD 5 mm QNP 1H/13
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 16
DS 0
SWH 3756.566 Hz
FIDRES 0.09831 Hz
AQ 5.1905012 sec
RG 2.14
DW 158.400 usec
DE 6.00 usec
TE 300.0 K
D1 0.10000000 sec
CHANNEL f1
NUC1 1H
P1 9.30 usec
PL1 0.00 dB
SFO1 300.1314228 MHz
SI 16384
SF 300.1300028 MHz
WDW EM
SSB 0
LB 0.10 Hz
GB 0
PC 1.00
6.24
12.61
1.04
1.01
0.94
1.00
0.97
2.01
4.96
ppm
22
more polar compound
BnO2C
Me
OH
N
OH
OH

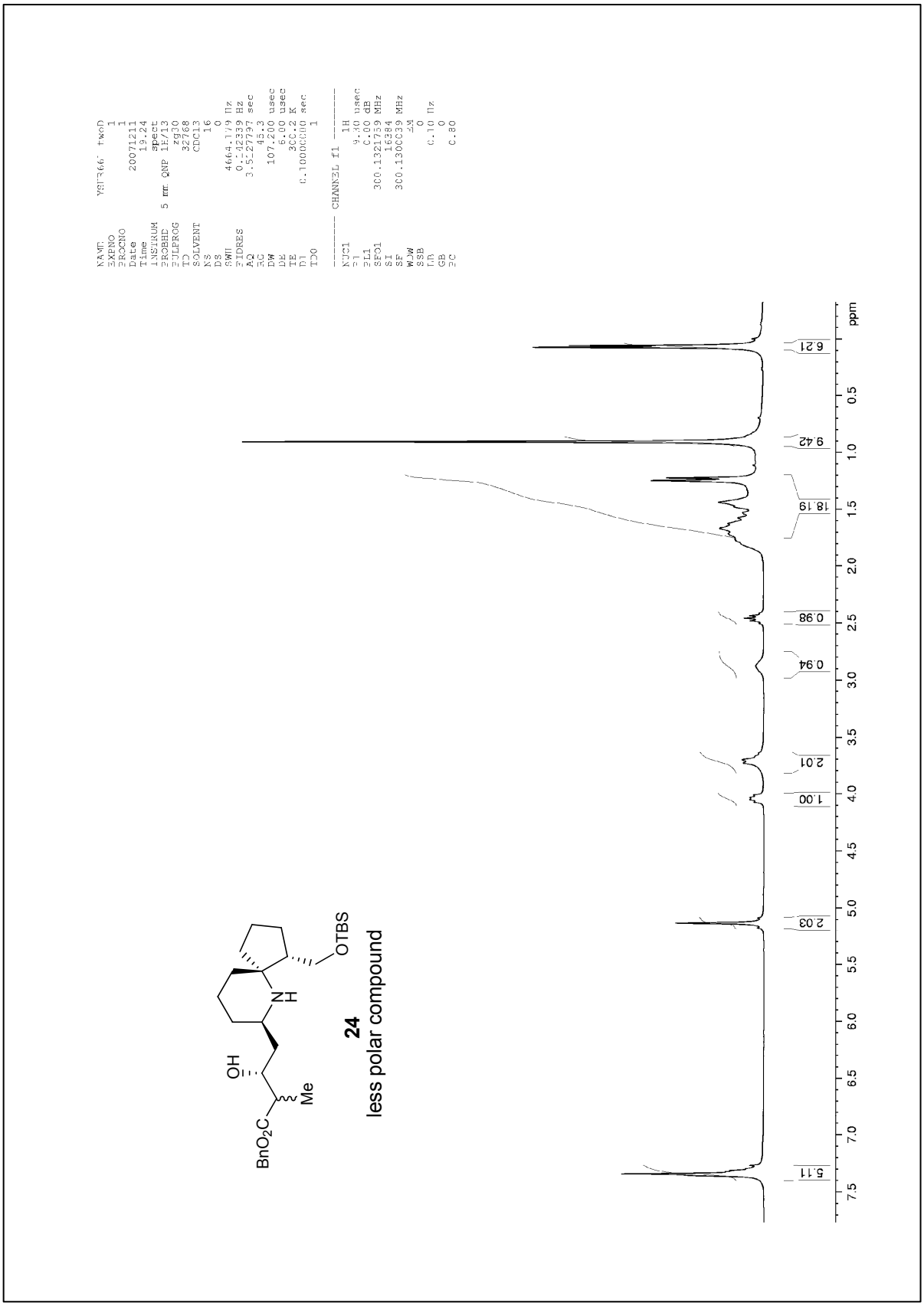

NAME YSHR475 two2
EXPNO 2
PROCNO 1
Date_ 20070310
Time 16.23
INSTRUM spect
PROBHD 5 mm QNP 1H/13
PULPROG zgpg
TD 65536
SOLVENT CDCl3
NS 300
DS 0
SWH 19980.414 Hz
FIDRES 0.304868 Hz
AQ 1.8219456 sec
RG 16384
DW 27.800 usec
DE 20.00 usec
TE 300.0 K
D1 0.17593171 sec
D11 0.03000000 sec
D12 0.00002000 sec
======== CHANNEL f1 ========
NUC1 13C
P1 5.44 usec
PL1 4.03 dB
SFO1 75.4769973 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 100.00 usec
PL2 0.00 dB
PL12 20.80 dB
PL13 26.40 dB
SFO2 300.1312005 MHz
SI 32768
SF 75.4677644 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.00
ppm

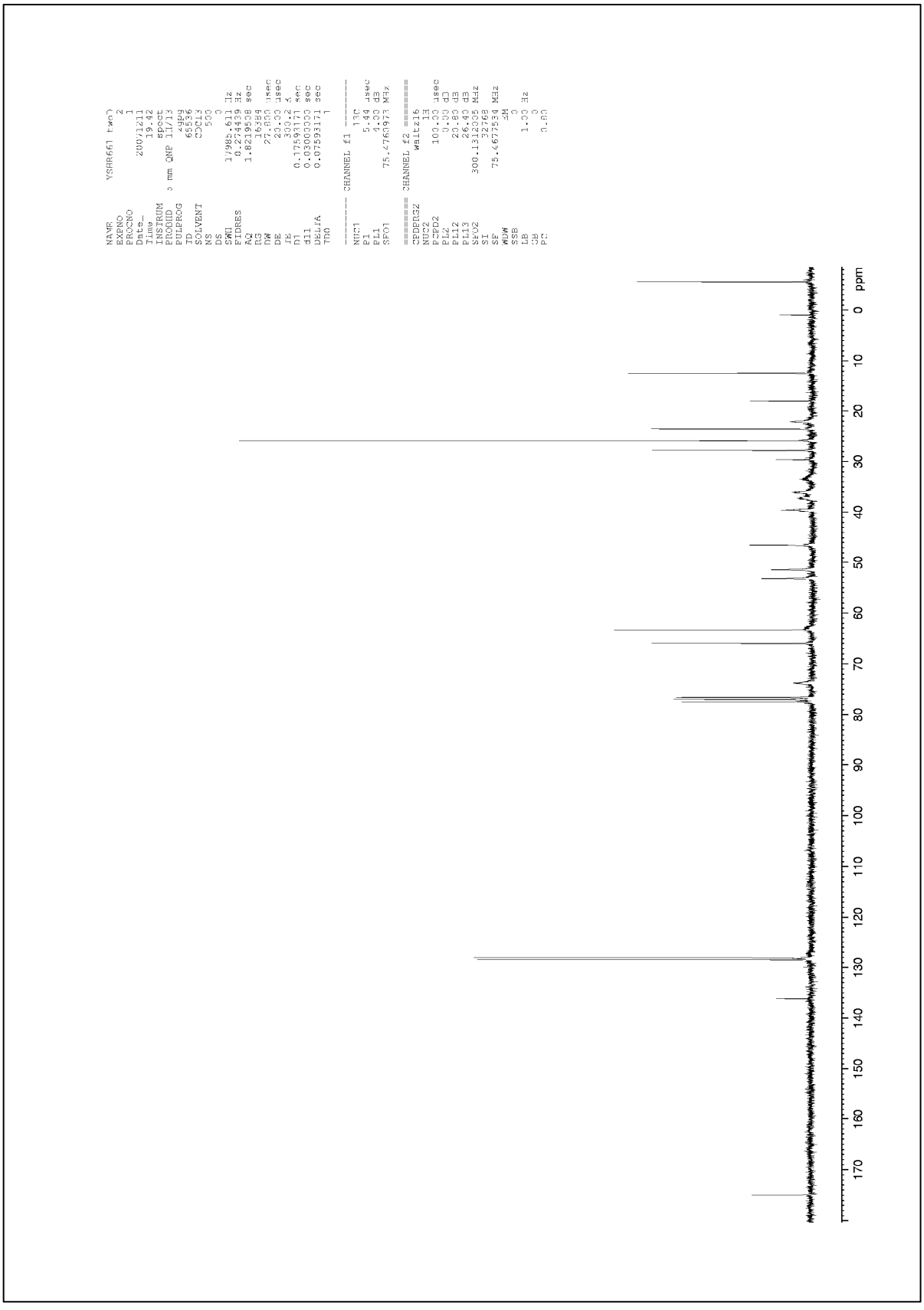

NAME YBT3448 fwod
EXPNO 1
PROCNO 1
Date 20070316
Time 17.53
INSTRUM spect
PROBHD 5 mm QNP 1H/13
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 16
DS 0
SWH 3306.878 Hz
FIDRES 0.100918 Hz
AQ 4.9545717 sec
RG 90.5
DW 151.200 usec
DE 6.00 usec
TE 300.0 K
D1 0.10000000 sec
CHANNEL f1
NUC1 1H
P1 9.30 usec
PL1 0.00 dB
SFO1 300.1315005 MHz
SI 16384
SF 300.1300016 MHz
WDW EM
SSB 0
LB 0.10 Hz
GB 0
PC 0.80
1.92
3.15
1.07
1.00
0.99
6.08
3.87
4.25
3.00
1.19
ppm
OH
N
H
OH
Me
BnO2C
23
less polar compound

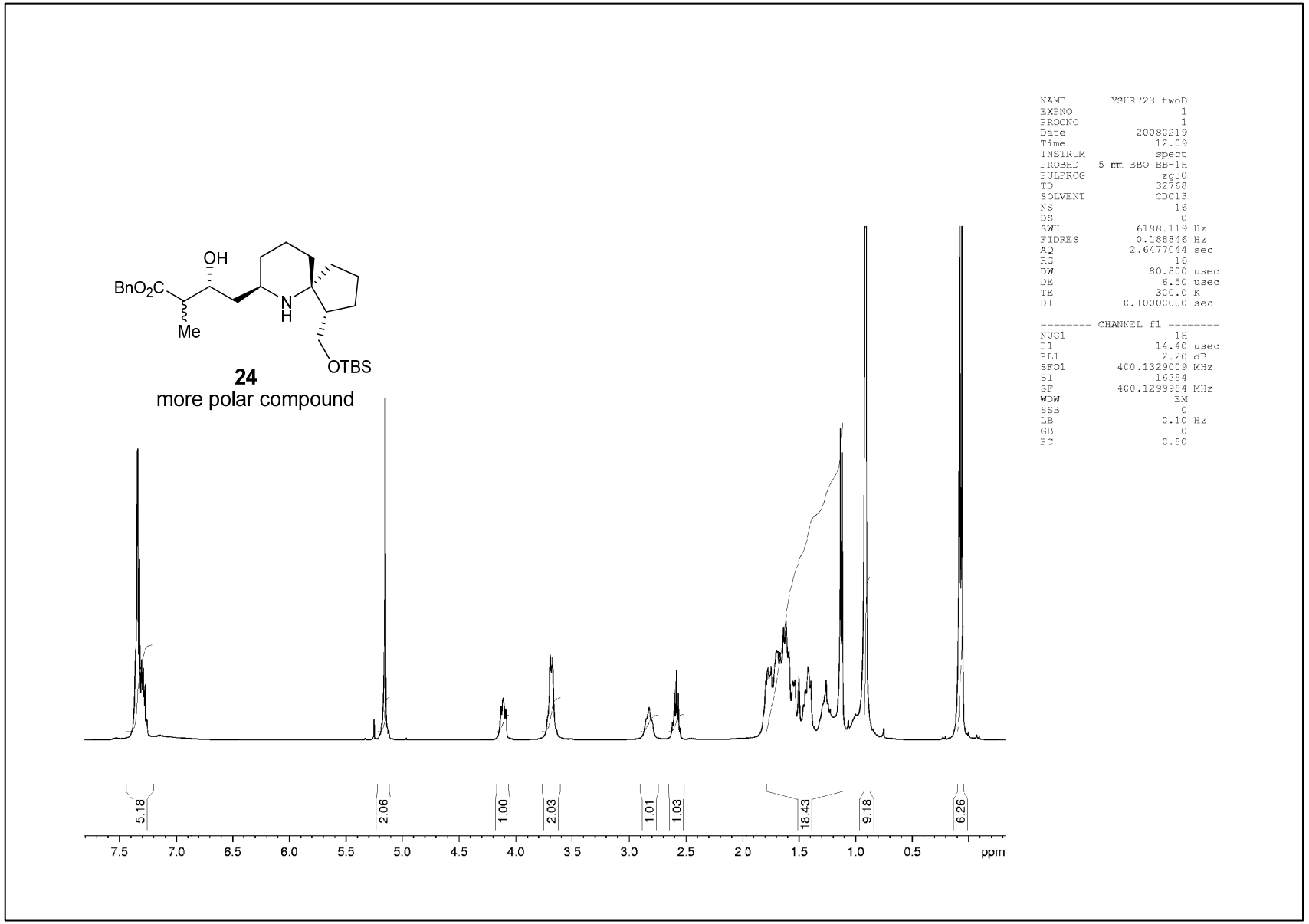

NAME YSHR478 two2
EXPNO 2
PROCNO 1
Date_ 20070316
Time 18.07
INSTRUM spect
PROBHD 5 mm QNP 1H/13
PULPROG zgpg
TD 65536
SOLVENT CDCl3
NS 400
DS 0
SWH 11990.419 Hz
FIDRES 0.182948 Hz
AQ 1.8219456 sec
RG 16384
DW 27.800 usec
DE 20.00 usec
TE 300.0 K
D1 0.17593171 sec
D11 0.03000000 sec
D12 0.00002000 sec
======== CHANNEL f1 ========
NUC1 13C
P1 5.44 usec
PL1 4.03 dB
SFO1 75.4769973 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 100.00 usec
PL2 0.00 dB
PL12 20.80 dB
PL13 26.40 dB
SFO2 300.1312005 MHz
SI 32768
SF 75.4677559 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.00
ppm

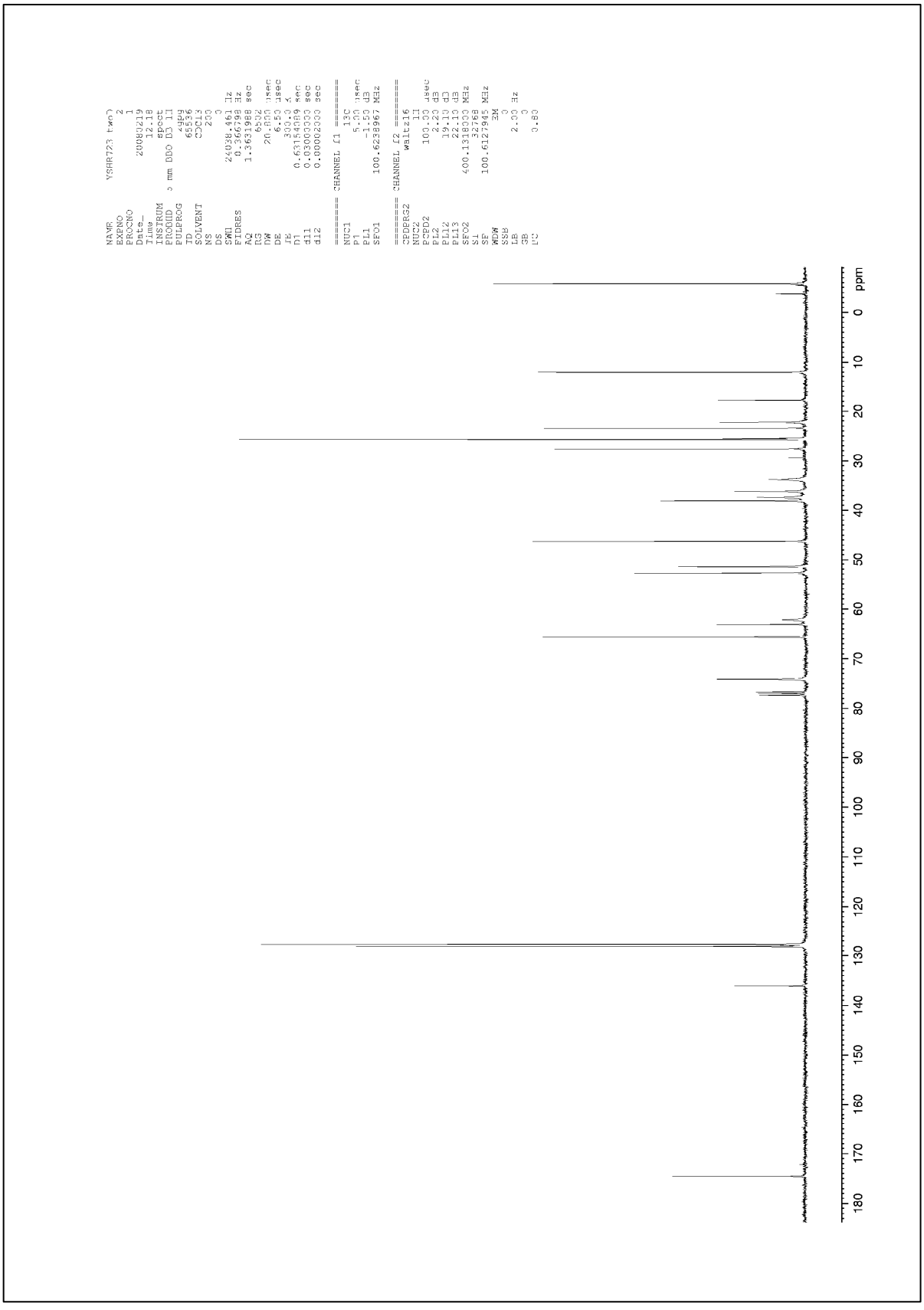

NAME YBT3454 tmod
EXPNO 1
PROCNO 1
Date 20070322
Time 19.23
INSTRUM spect
PROBHD 5 mm QNP 1H/13
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 16
DS 0
SWH 3756.566 Hz
FIDRES 0.096331 Hz
AQ 5.1905012 sec
RG 25.9
DW 158.400 usec
DE 6.00 usec
TE 300.0 K
D1 0.10000000 sec
CHANNEL f1
NUC1 1H
P1 9.30 usec
PL1 0.00 dB
SFO1 300.1314228 MHz
SI 16384
SF 300.1300000 MHz
WDW EM
SSB 0
LB 0.10 Hz
GB 0
PC 0.80
OH
N
H
OH
Me
BnO2C
23
more polar compound
4.95
2.00
1.00
1.00
14.38
2.95
1.10
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
ppm

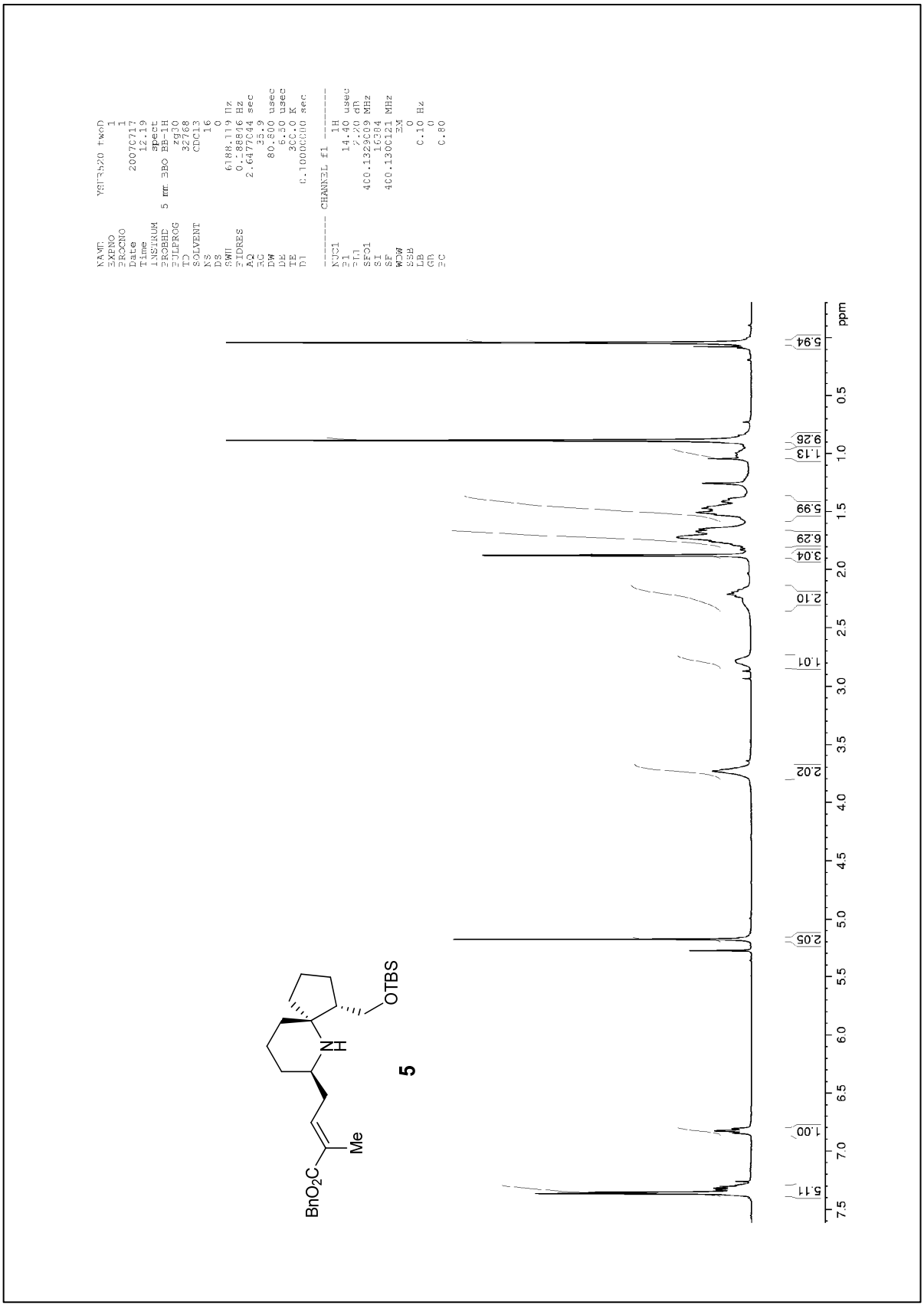

NAME YSHR454 two2
EXPNO 2
PROCNO 1
Date_ 20070322
Time 19.34
INSTRUM spect
PROBHD 5 mm QNP 1H/13
PULPROG zgpg
TD 65536
SOLVENT CDCl3
NS 300
DS 0
SWH 17985.611 Hz
FIDRES 0.274498 Hz
AQ 1.8219958 sec
RG 16384
DW 27.800 usec
DE 20.00 usec
TE 300.0 K
D1 0.17593171 sec
d11 0.03000000 sec
d12 0.00002000 sec
======== CHANNEL f1 ========
NUC1 13C
P1 5.44 usec
PL1 4.03 dB
SFO1 75.4769973 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 100.00 usec
PL2 0.00 dB
PL12 20.80 dB
PL13 26.40 dB
SFO2 300.1312005 MHz
SI 32768
SF 75.4677591 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 0.80
ppm


OTBS
N
H
OH
Me
BnO2C
24
less polar compound
6.21
9.42
18.19
0.98
0.94
2.01
1.00
2.03
5.11
ppm

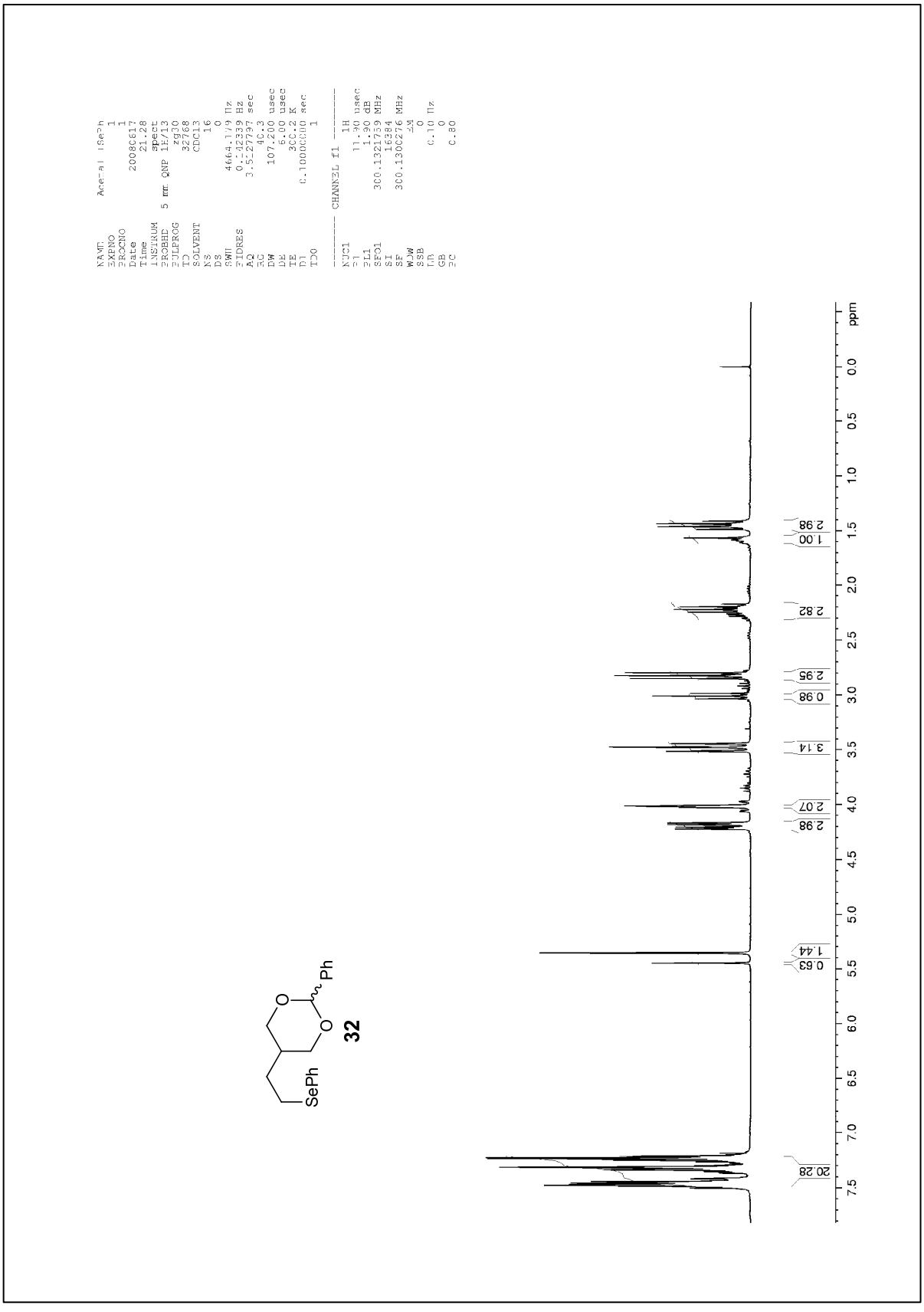

NAME YSHR661 two2
EXPNO 2
PROCNO 1
Date_ 20071211
Time 19.42
INSTRUM spect
PROBHD 5 mm QNP 1H/13
PULPROG zgpg
TD 65536
SOLVENT CDCl3
NS 500
DS 0
SWH 17985.611 Hz
FIDRES 0.274488 Hz
AQ 1.821955 sec
RG 16384
DW 27.800 usec
DE 20.00 usec
TE 300.2 K
D1 0.17593171 sec
d11 0.03000000 sec
DELTA 0.07593171 sec
TD0 1
CHANNEL f1
NUC1 13C
P1 5.40 usec
PL1 -1.00 dB
SFO1 75.4760973 MHz
CHANNEL f2
CPDPRG2 waltz16
NUC2 1H
PCPD2 100.00 usec
PL2 0.00 dB
PL12 20.80 dB
PL13 26.40 dB
SFO2 300.1312005 MHz
SI 32768
SF 75.4677594 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.80
0 ppm

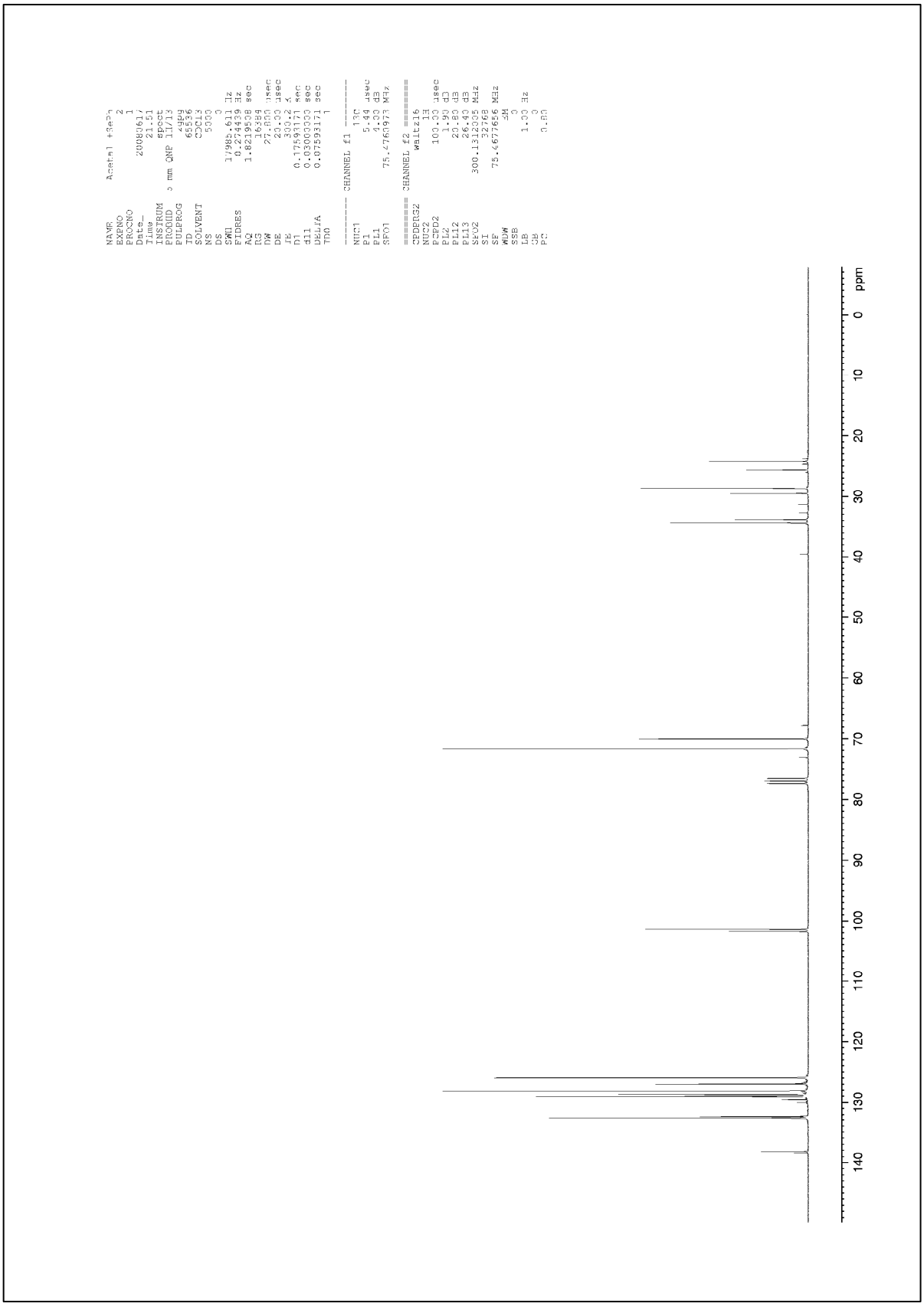

OH
BnO2C
Me
N
H
OTBS
24
more polar compound
NAME YSTR723 twoD
EXPNO 1
PROCNO 1
Date 20080219
Time 12.09
INSTRUM spect
PROBHD 5 mm BBO BB-1H
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 16
DS 0
SWH 6188.119 Hz
FIDRES 0.188816 Hz
AQ 2.6477044 sec
RG 16
DW 80.800 usec
DE 6.50 usec
TE 300.0 K
D1 0.10000000 sec
CHANNEL f1
NUC1 1H
P1 14.40 usec
PL1 2.20 dB
SFO1 400.1329009 MHz
SI 16384
SF 400.1299984 MHz
WDW EM
SSB 0
LB 0.10 Hz
GB 0
PC 0.80
5.18
2.06
1.00
2.03
1.01
1.03
18.43
9.18
6.26
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
ppm

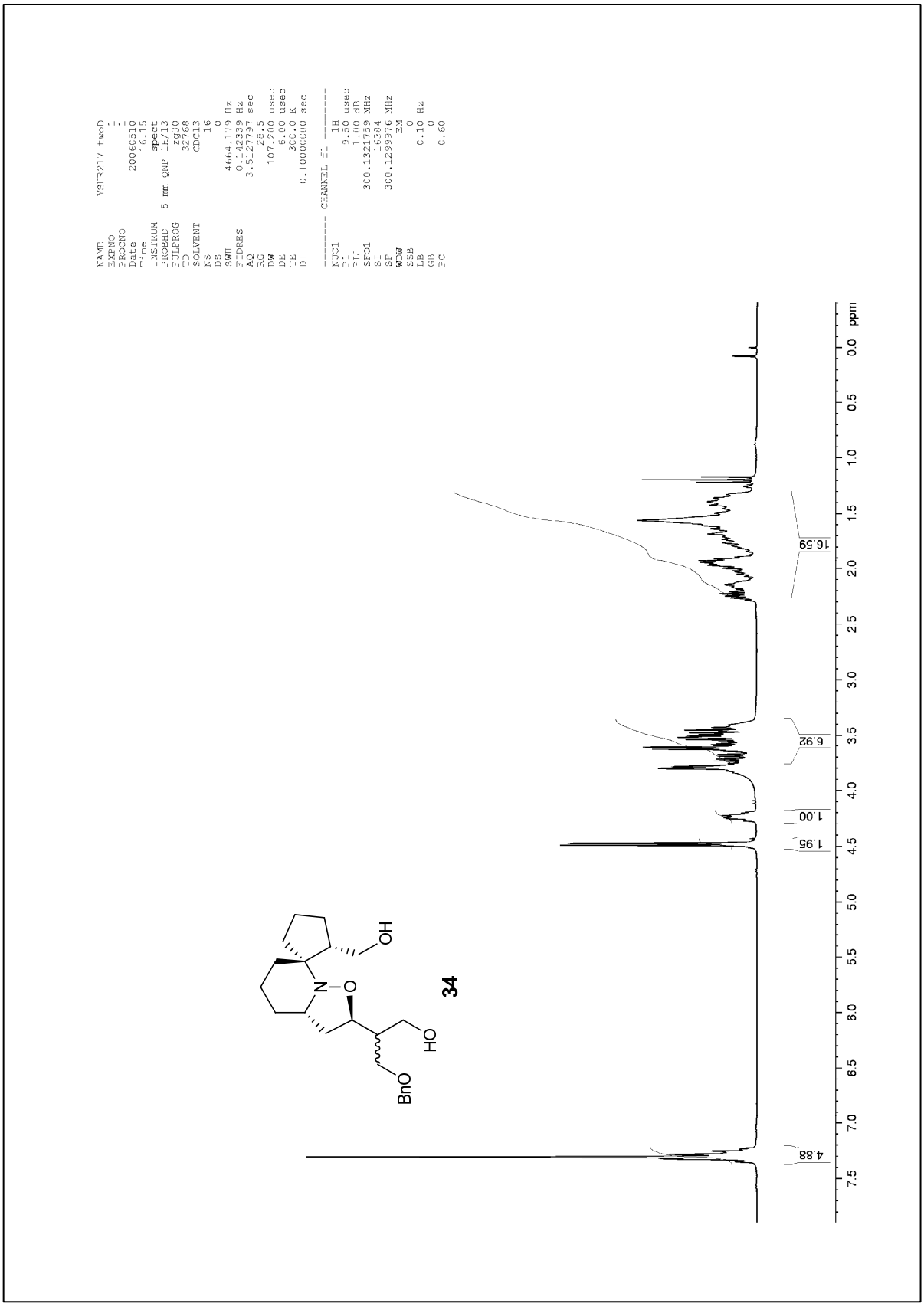

NAME YSHR723 two2
EXPNO 2
PROCNO 1
Date_ 20080219
Time 12.18
INSTRUM spect
PROBHD 5 mm DDO DS 1H
PULPROG zgpg
TD 65536
SOLVENT CDCl3
NS 200
DS 0
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3631988 sec
RG 4532
DW 20.800 usec
DE 6.50 usec
TE 300.0 K
D1 0.63154089 sec
d11 0.03000000 sec
d12 0.00002000 sec
======== CHANNEL f1 ========
NUC1 13C
P1 5.00 usec
PL1 -5.03 dB
SFO1 100.6239967 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 100.00 usec
PL2 2.20 dB
PL12 19.10 dB
PL13 22.10 dB
SFO2 400.1318033 MHz
SI 32768
SF 100.6127945 MHz
WDW EM
SSB 0
LB 2.00 Hz
GB 0
PC 0.80
ppm

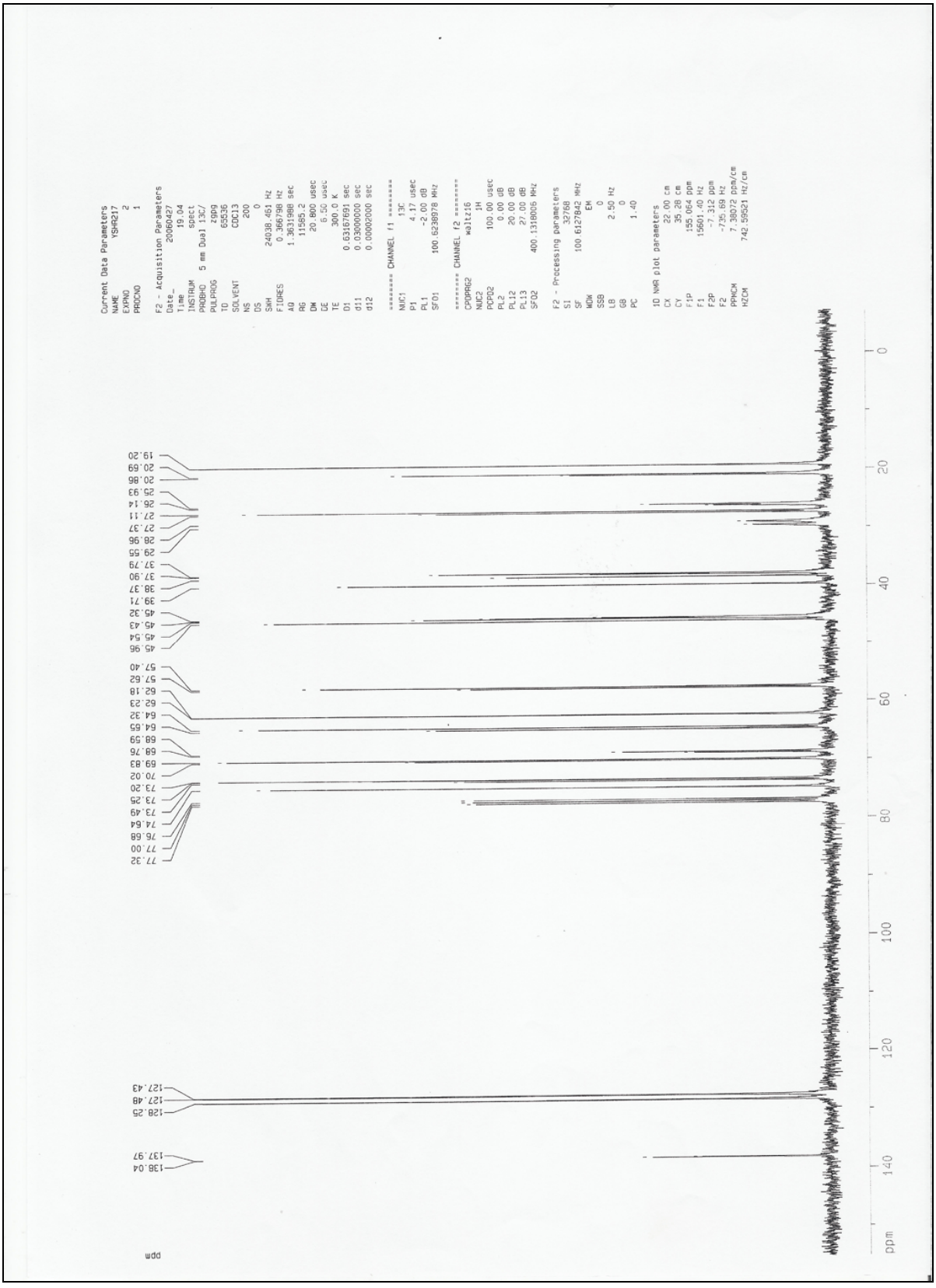

OTBS
N
H
Me
BnO2C
5
ppm
5.94
1.13
9.25
5.99
6.29
3.04
2.10
1.01
2.02
2.05
1.00
5.11

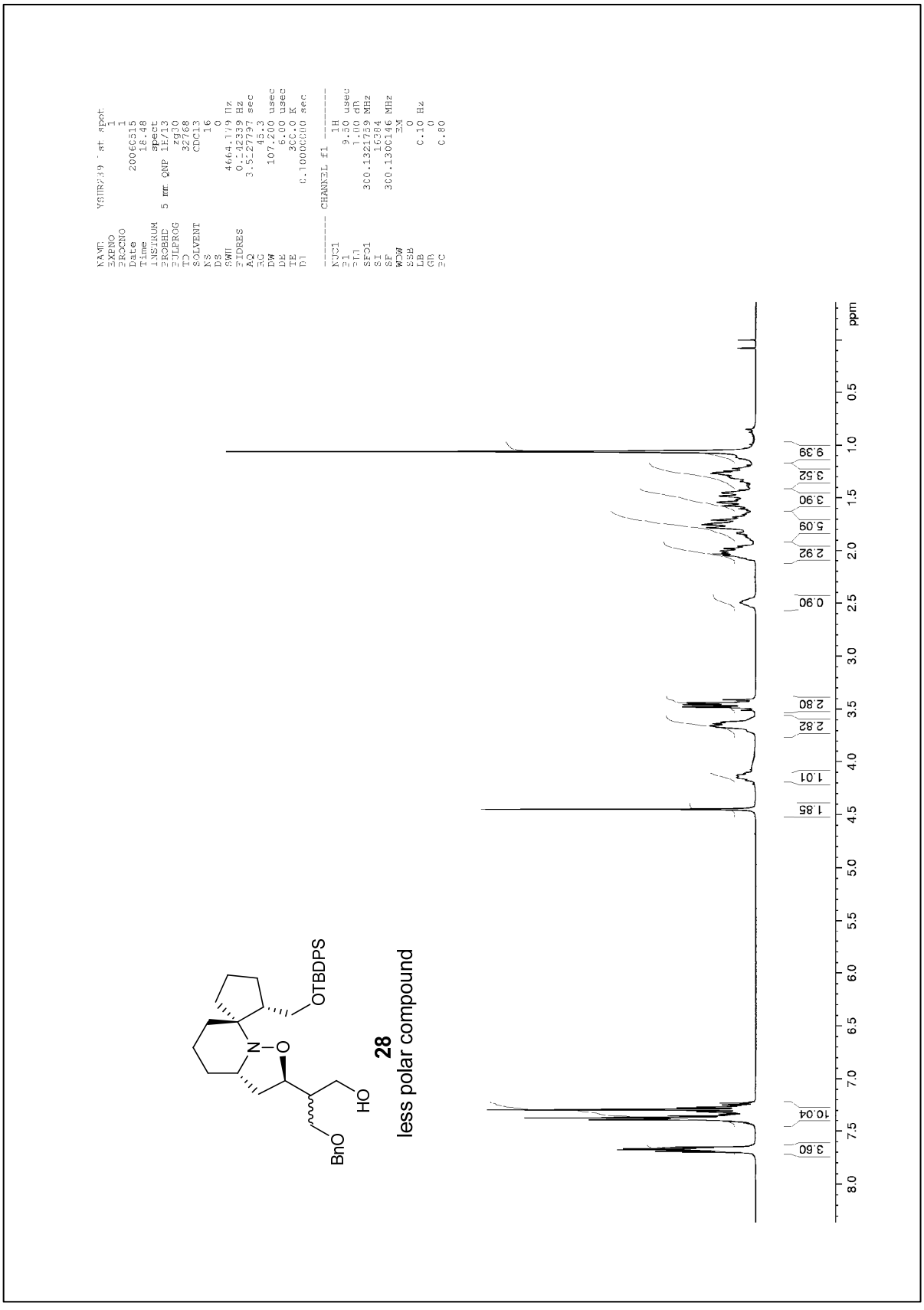

NAME YSHR520 two2
EXPNO 2
PROCNO 1
Date_ 20070717
Time 12.36
INSTRUM spect
PROBHD 5 mm DUO D3 11
PULPROG zgpg
TD 65536
SOLVENT CDCl3
NS 400
DS 0
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3631988 sec
RG 91958.7
DW 20.800 usec
DE 6.50 usec
TE 300.0 K
D1 0.63154089 sec
d11 0.03000000 sec
d12 0.00002000 sec
======== CHANNEL f1 ========
NUC1 13C
P1 5.00 usec
PL1 -5.13 dB
SFO1 100.6239967 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 100.00 usec
PL2 2.20 dB
PL12 19.10 dB
PL13 22.10 dB
SFO2 400.1318000 MHz
SI 32768
SF 100.6127787 MHz
WDW EM
SSB 0
LB 2.00 Hz
GB 0
PC 0.80
170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

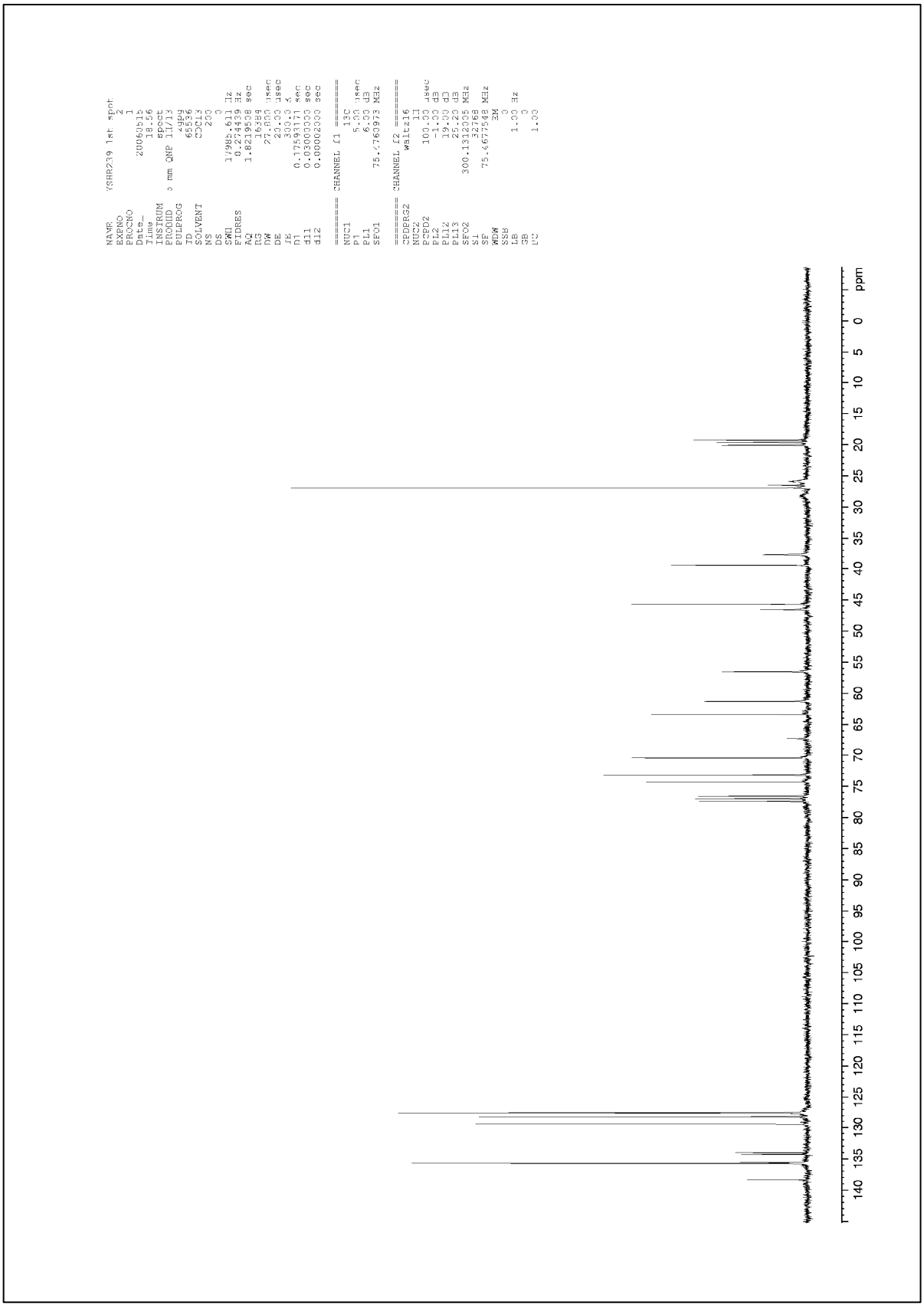

NAME Acetal_SePh
EXPNO 1
PROCNO 1
Date 20080617
Time 21.28
INSTRUM spect
PROBHD 5 mm QNP 1H/13
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 16
DS 0
SWH 4664.179 Hz
FIDRES 0.142339 Hz
AQ 3.5127797 sec
RG 40.3
DW 107.200 usec
DE 6.00 usec
TE 300.2 K
D1 0.10000000 sec
TD0 1
CHANNEL f1 1H
NUC1 1H
P1 11.30 usec
PL1 -1.39 dB
SFO1 300.1321739 MHz
SI 16384
SF 300.1300276 MHz
WDW EM
SSB 0
LB 0.10 Hz
GB 0
PC 0.80
SePh
Ph
32
20.28
1.44
0.83
2.07
2.98
3.14
0.98
2.95
2.82
1.00
2.98
ppm

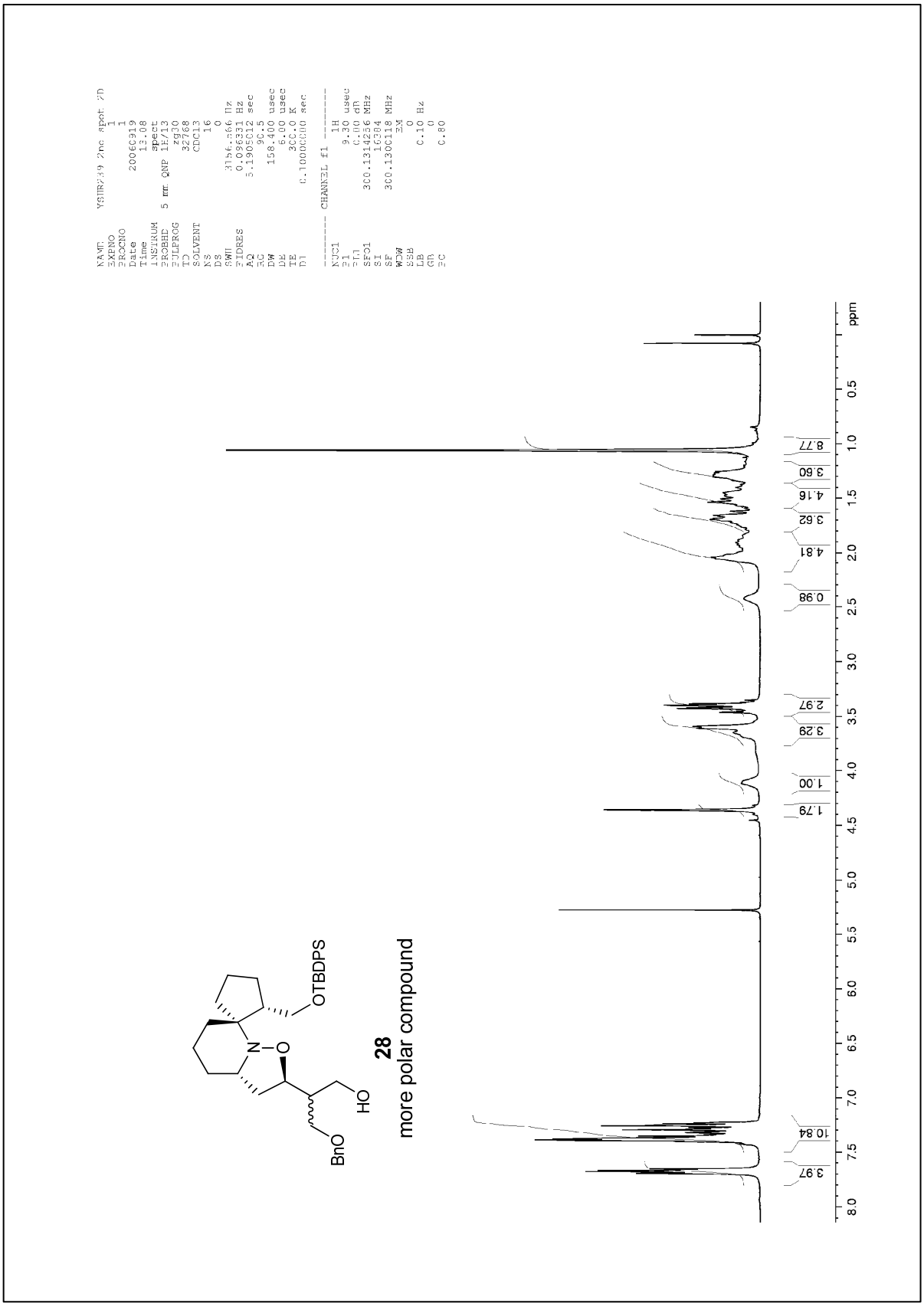

ppm
0
10
20
30
40
50
60
70
80
90
100
110
120
130
140

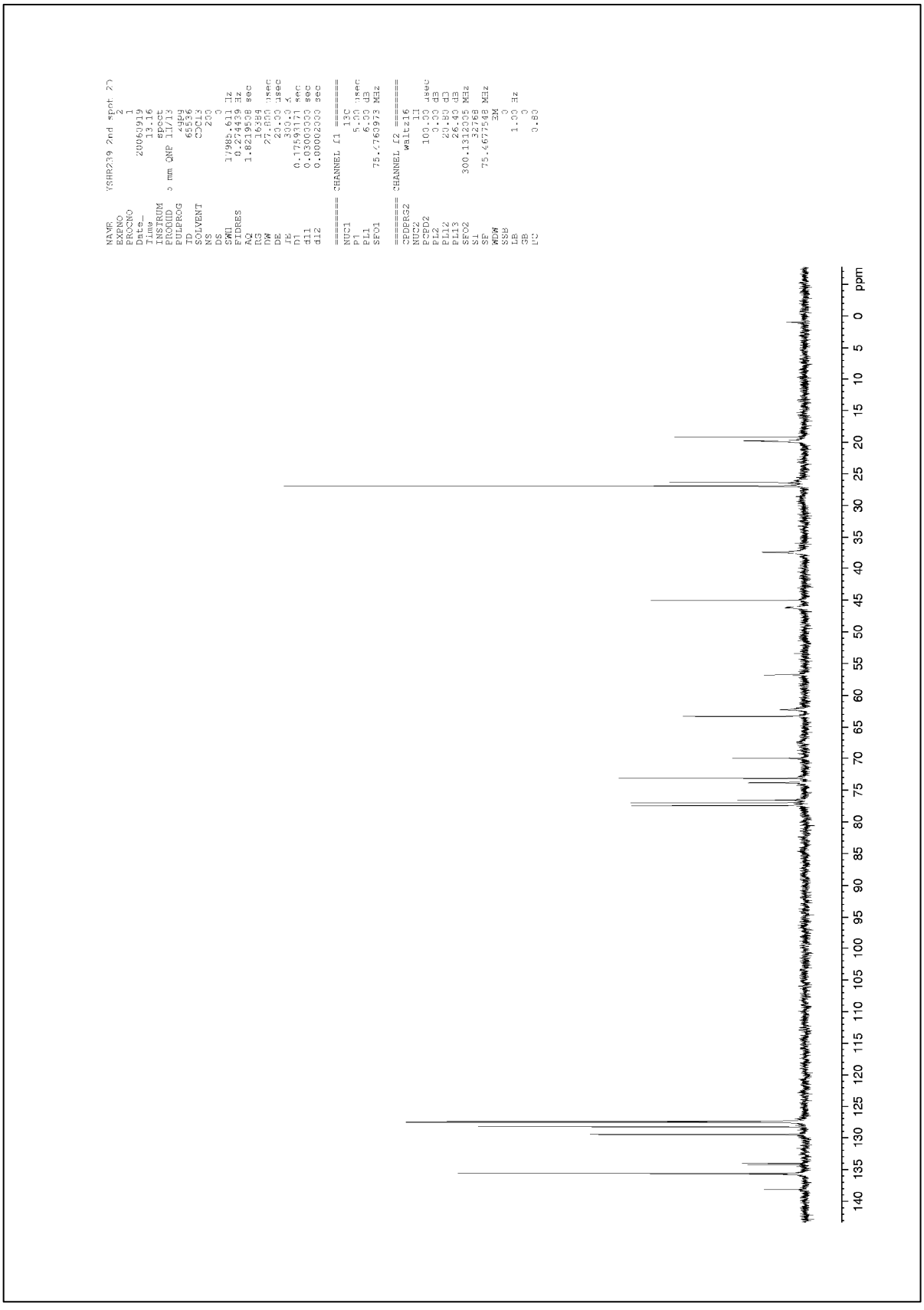

34
OH
HO
BnO
N
O
4.88
1.95
1.00
6.92
16.59
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
ppm

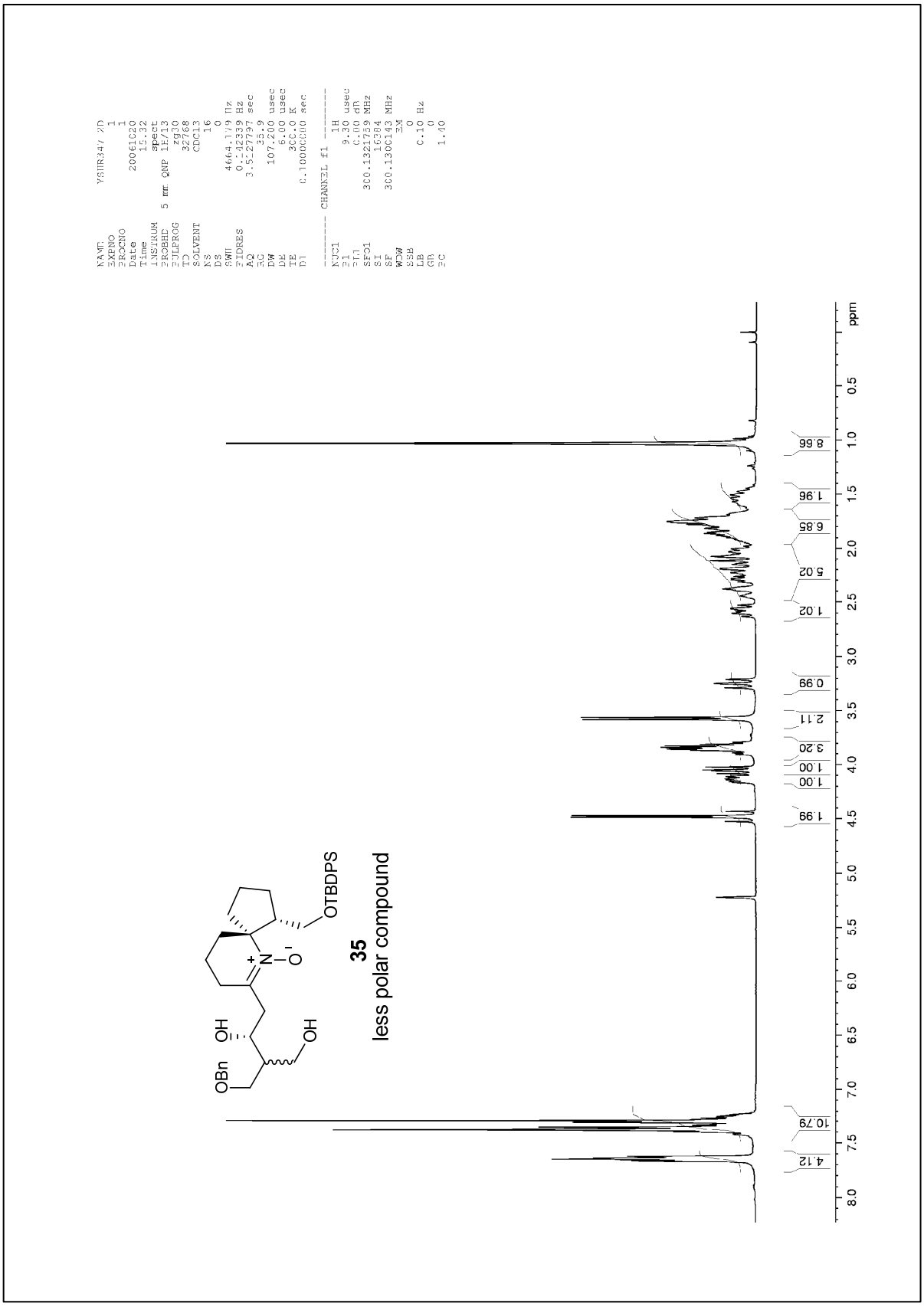

Current Data Parameters
NAME YSHR217
EXPNO 2
PROCNO 1
F2 - Acquisition Parameters
Date_ 20060427
Time 19.04
INSTRUM spect
PROBHD 5 mm Dual 13C/
PULPROG zgpg
TD 65536
SOLVENT CDCl3
NS 200
DS 0
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3631988 sec
RG 11585.2
DW 20.800 usec
DE 6.50 usec
TE 300.0 K
D1 0.63167691 sec
d11 0.03000000 sec
d12 0.00002000 sec
======== CHANNEL f1 ========
NUC1 13C
P1 4.17 usec
PL1 -2.00 dB
SFO1 100.6238978 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 100.00 usec
PL2 0.00 dB
PL12 20.00 dB
PL13 27.00 dB
SFO2 400.1318005 MHz
F2 - Processing parameters
SI 32768
SF 100.6127842 MHz
WDW EM
SSB 0
LB 2.50 Hz
GB 0
PC 1.40
1D NMR plot parameters
CX 22.00 cm
CY 35.20 cm
F1P 155.064 ppm
F1 15601.40 Hz
F2P -7.312 ppm
F2 -735.69 Hz
PPMCM 7.38072 ppm/cm
HZCM 742.59521 Hz/cm
19.20
20.69
20.86
25.93
26.14
27.11
27.37
28.96
29.55
37.79
37.90
38.37
39.71
45.32
45.43
45.54
45.96
57.40
57.62
62.18
62.23
64.32
64.65
68.59
68.76
69.83
70.02
73.20
73.25
73.49
74.64
76.68
77.00
77.32
127.43
127.48
128.25
137.97
138.04
ppm
0
20
40
60
80
100
120
140
ppm


OTBDPS
HO
BnO
28
less polar compound
10.04
3.60
1.85
1.01
2.82
2.80
0.90
2.92
5.09
3.90
3.52
9.39
ppm

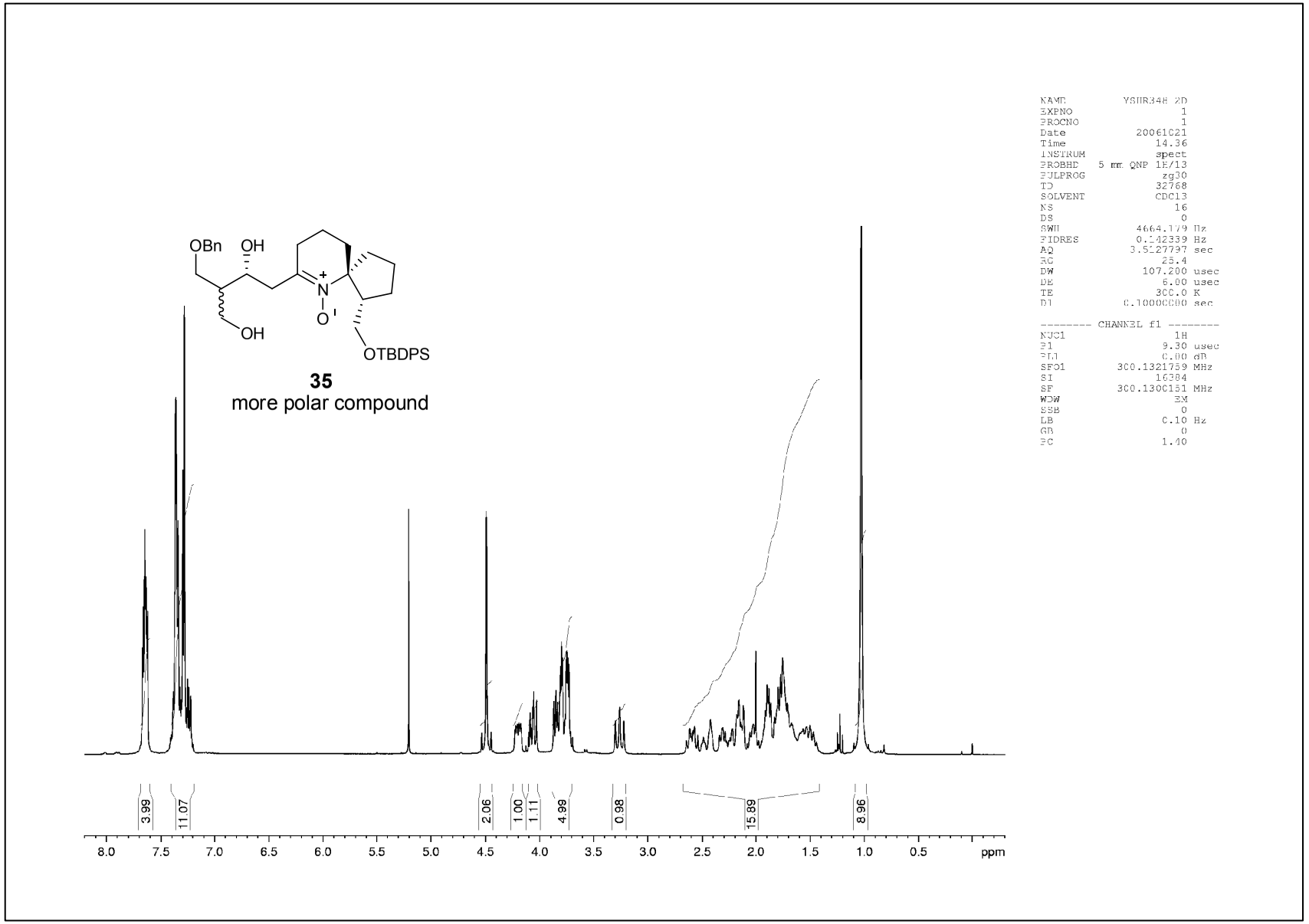

ppm


OTBDPS
N–O
HO
BnO
28
more polar compound
ppm

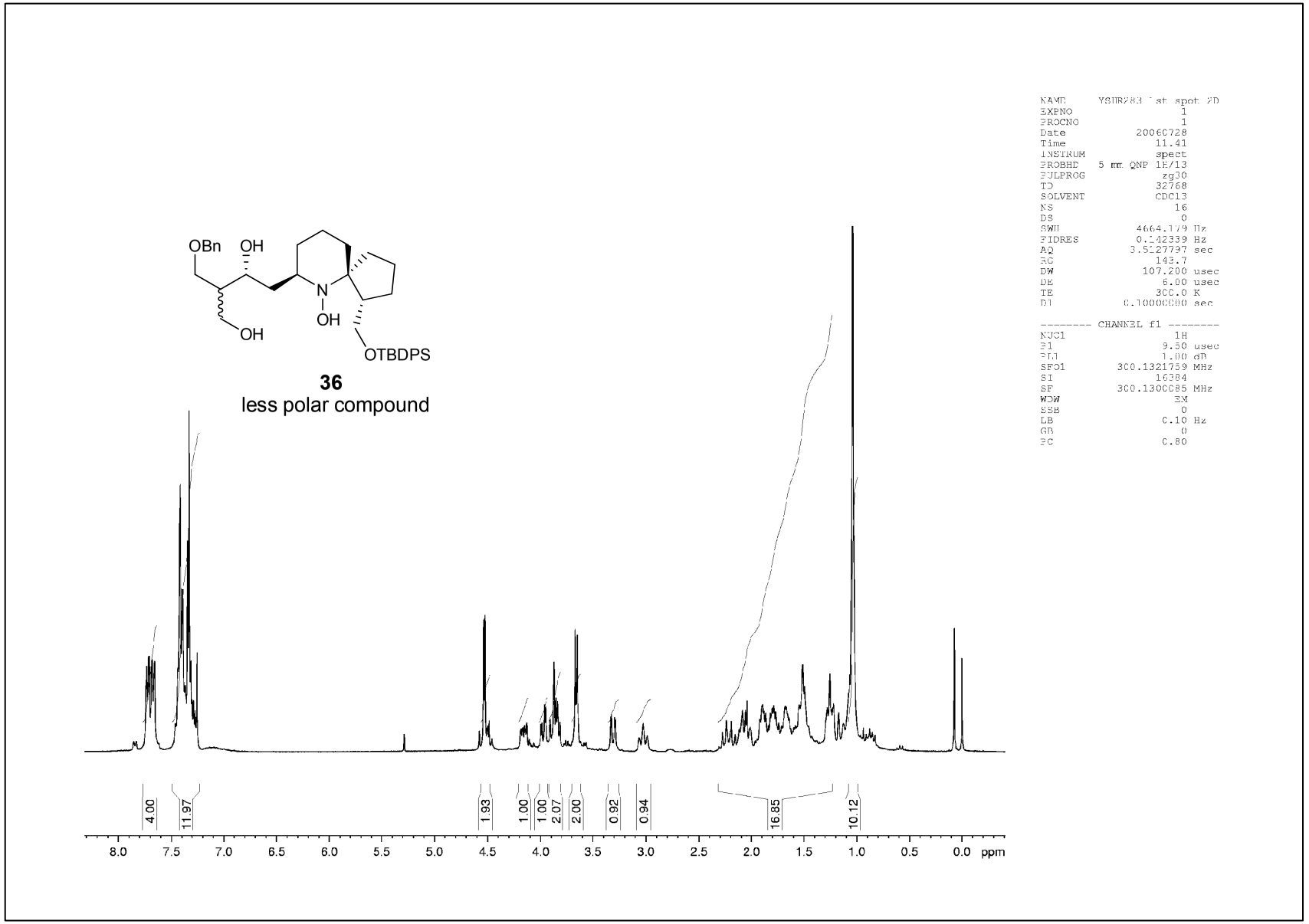

NAME YSHR239 2nd spot 2)
EXPNO 2
PROCNO 1
Date_ 20060919
Time 13.16
INSTRUM spect
PROBHD 5 mm QNP 1H/13
PULPROG zgpg
TD 65536
SOLVENT CDCl3
NS 200
DS 0
SWH 19980.414 Hz
FIDRES 0.304810 Hz
AQ 1.8219456 sec
RG 16384
DW 27.800 usec
DE 20.00 usec
TE 300.0 K
D1 0.17593171 sec
D11 0.03000000 sec
D12 0.00002000 sec
======== CHANNEL f1 ========
NUC1 13C
P1 5.00 usec
PL1 6.00 dB
SFO1 75.4760973 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 100.00 usec
PL2 0.00 dB
PL12 20.80 dB
PL13 26.40 dB
SFO2 300.1312005 MHz
SI 32768
SF 75.4677548 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 0.80
ppm


OTBDPS
OBn
OH
OH
35
less polar compound
ppm


NAME YSHR347 22
EXPNO 2
PROCNO 1
Date_ 20061020
Time 15.39
INSTRUM spect
PROBHD 5 mm QNP 1H/13
PULPROG zgpg
TD 65536
SOLVENT CDCl3
NS 200
DS 0
SWH 11990.419 Hz
FIDRES 0.182948 Hz
AQ 1.8219456 sec
RG 16384
DW 27.800 usec
DE 20.00 usec
TE 300.0 K
D1 0.17593171 sec
D11 0.03000000 sec
D12 0.00002000 sec
======== CHANNEL f1 ========
NUC1 13C
P1 5.00 usec
PL1 6.03 dB
SFO1 75.4769973 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 100.00 usec
PL2 0.00 dB
PL12 20.80 dB
PL13 26.40 dB
SFO2 300.1312005 MHz
SI 32768
SF 75.4677494 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 0.80
ppm

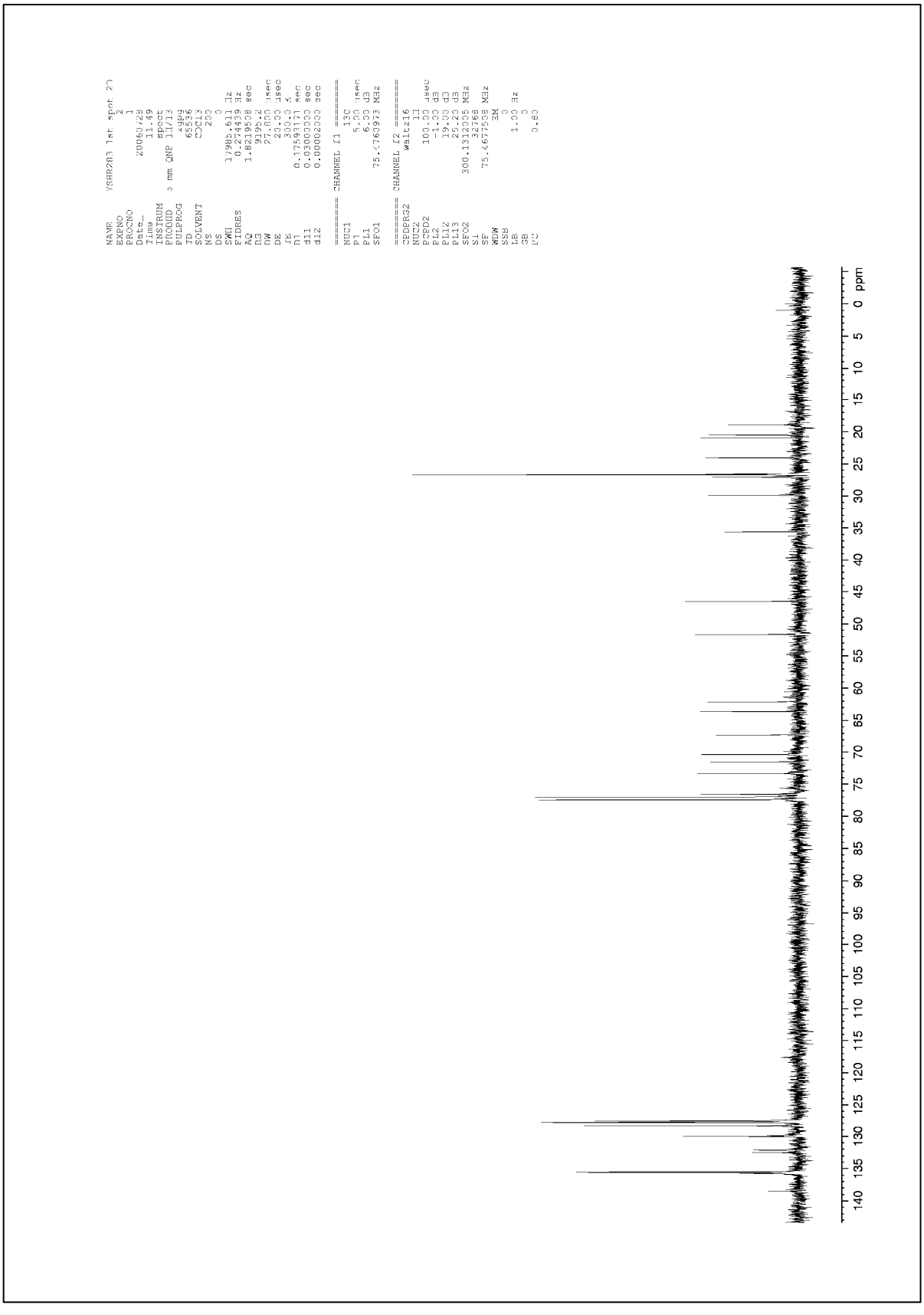

OBn
OH
OH
N
O
OTBDPS
35
more polar compound
3.99
11.07
2.06
1.00
1.11
4.99
0.98
15.89
8.96
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
ppm
NAME YSUR348 2D
EXPNO 1
PROCNO 1
Date 20061021
Time 14.36
INSTRUM spect
PROBHD 5 mm QNP 1H/13
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 16
DS 0
SWH 4664.179 Hz
FIDRES 0.142339 Hz
AQ 3.5227797 sec
RG 25.4
DW 107.200 usec
DE 6.00 usec
TE 300.0 K
D1 0.10000000 sec
-------- CHANNEL f1 --------
NUC1 1H
P1 9.30 usec
PL1 0.00 dB
SFO1 300.1321759 MHz
SI 16384
SF 300.1300151 MHz
WDW EM
SSB 0
LB 0.10 Hz
GB 0
PC 1.40

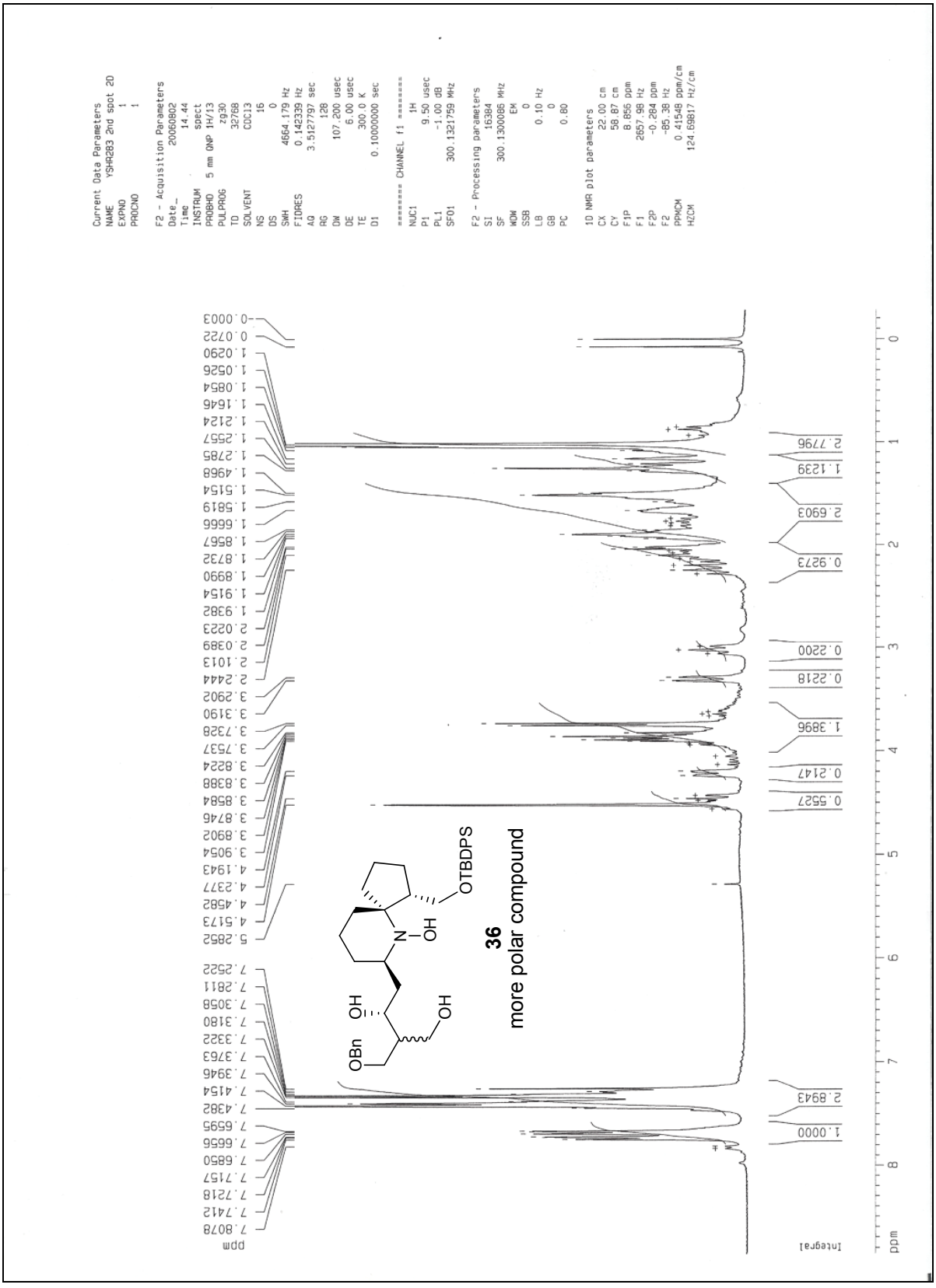

NAME YSHR348 22
EXPNO 2
PROCNO 1
Date_ 20061021
Time 14.44
INSTRUM spect
PROBHD 5 mm QNP 1H/13
PULPROG zgpg
TD 65536
SOLVENT CDCl3
NS 200
DS 0
SWH 11990.419 Hz
FIDRES 0.182948 Hz
AQ 1.8219456 sec
RG 16384
DW 27.800 usec
DE 20.00 usec
TE 300.0 K
D1 0.17593171 sec
D11 0.03000000 sec
D12 0.00002000 sec
======== CHANNEL f1 ========
NUC1 13C
P1 5.00 usec
PL1 6.03 dB
SFO1 75.4760973 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 100.00 usec
PL2 0.00 dB
PL12 20.80 dB
PL13 26.40 dB
SFO2 300.1312005 MHz
SI 32768
SF 75.4677494 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 0.80
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
ppm

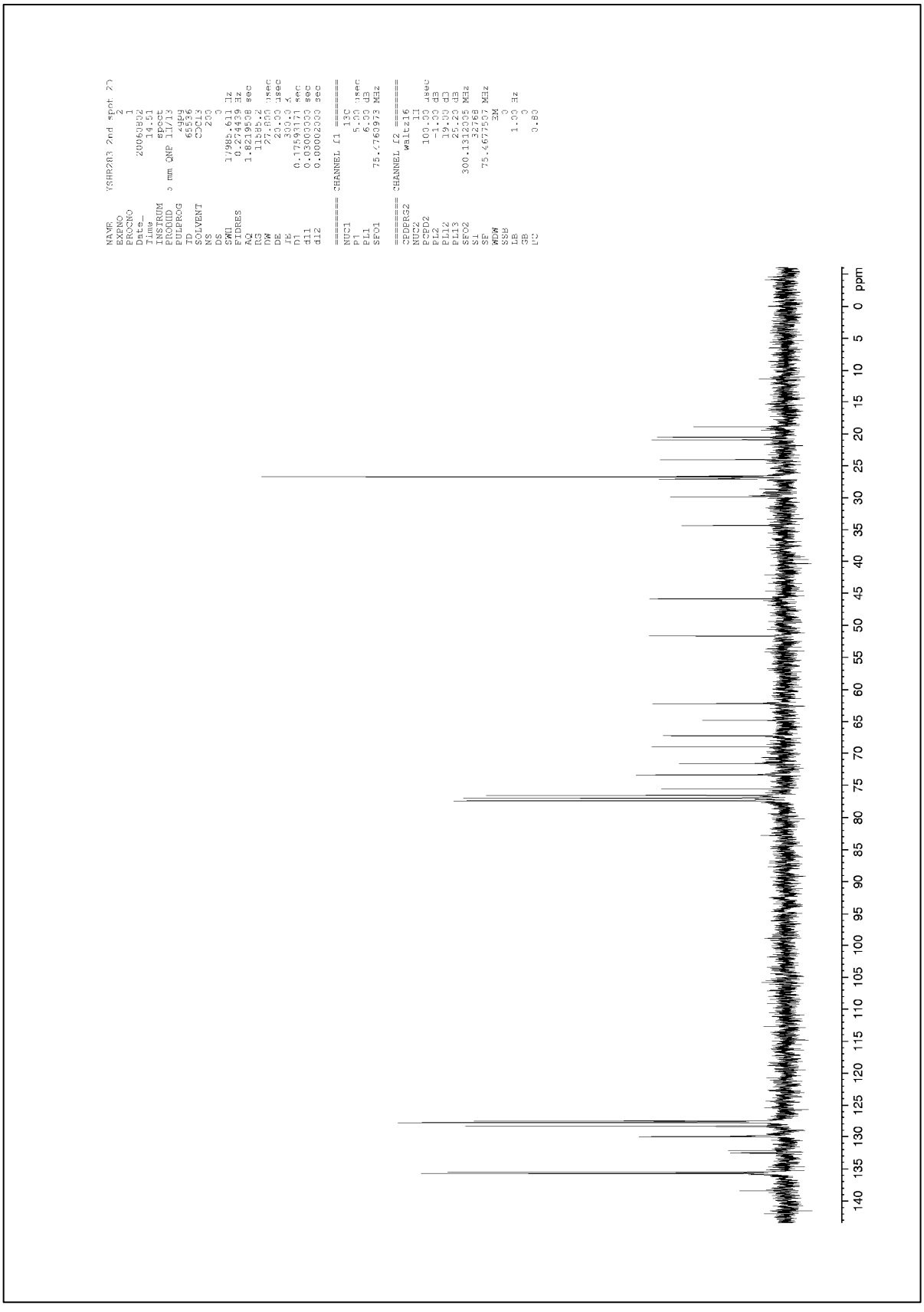


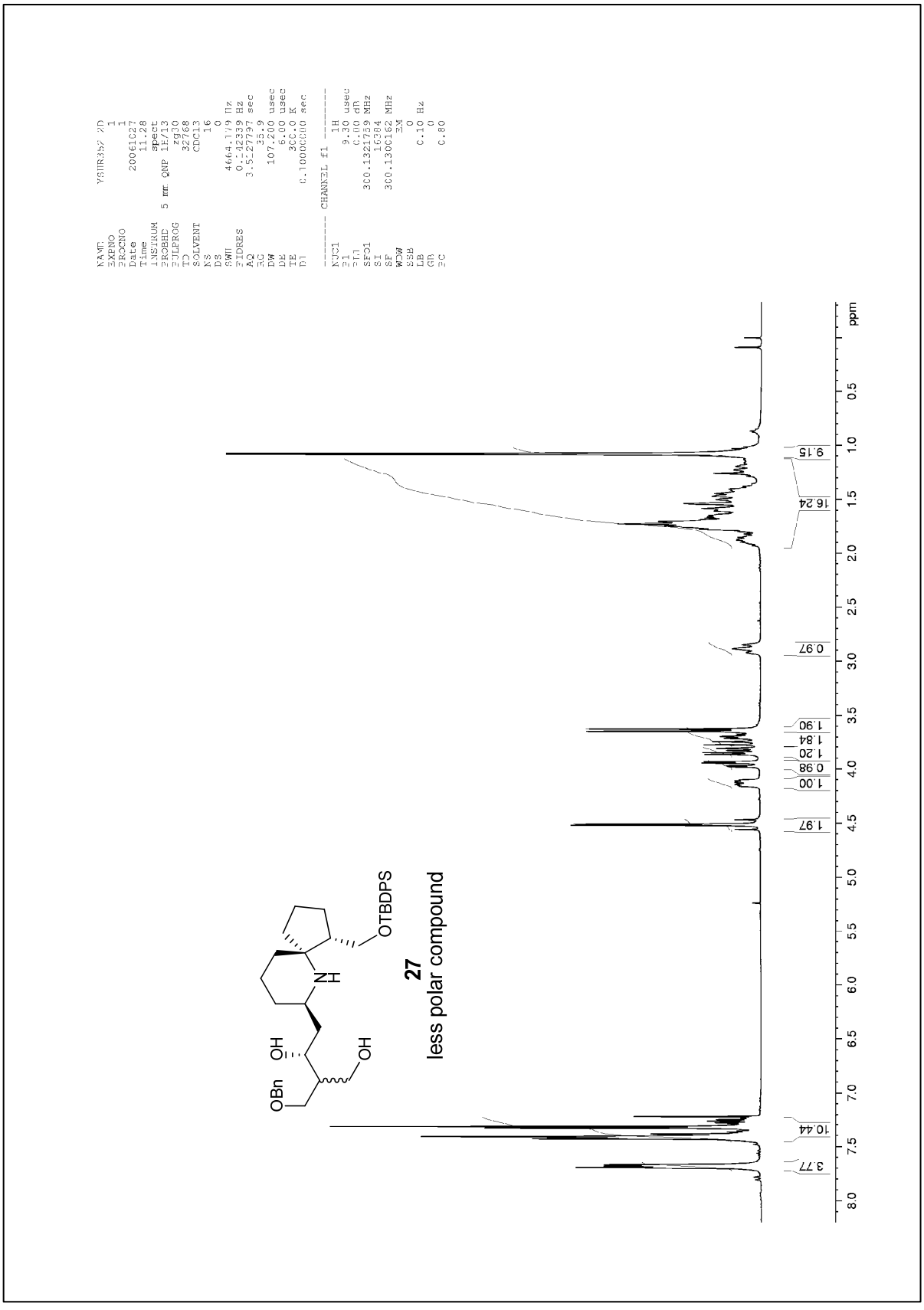

```
NAME      YSUR283 1st spot 2D
EXPNO                1
PROCNO               1
Date          20060728
Time             11.41
INSTRUM          spect
PROBHD   5 mm QNP 1H/13
PULPROG          zg30
TD              32768
SOLVENT          CDCl3
NS                  16
DS                   0
SWH           4664.179 Hz
FIDRES        0.142339 Hz
AQ            3.5127797 sec
RG                143.7
DW               107.200 usec
DE                 6.00 usec
TE                300.0 K
D1             0.10000000 sec

-------- CHANNEL f1 --------
NUC1                 1H
P1                 9.50 usec
PL1                1.00 dB
SFO1        300.1321759 MHz
SI                 16384
SF          300.1300085 MHz
WDW                  EM
SSB                  0
LB                0.10 Hz
GB                   0
PC                0.80
```

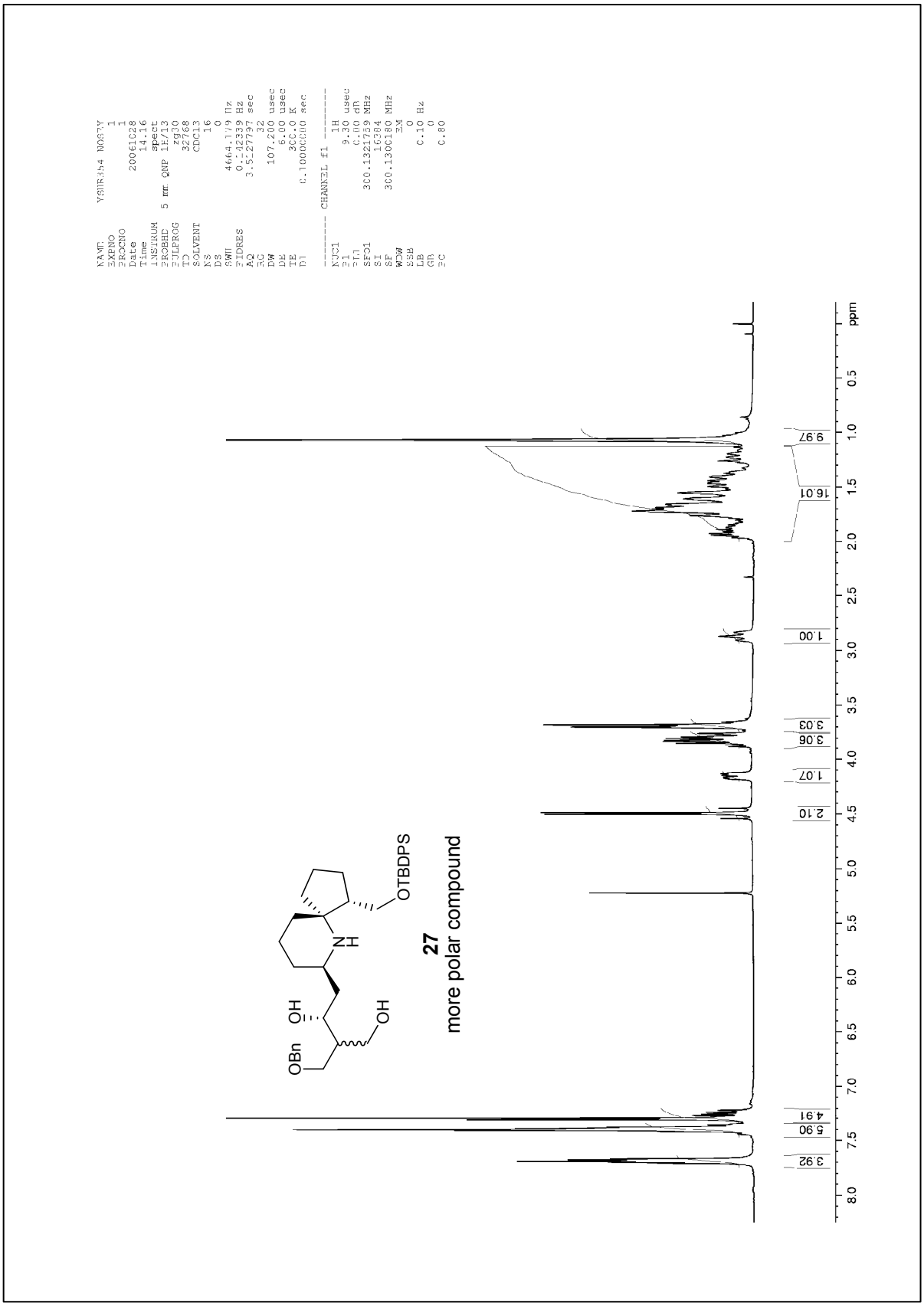

NAME YSHA203 1st spot 20
EXPNO 2
PROCNO 1
Date_ 20060728
Time 11.49
INSTRUM spect
PROBHD 5 mm QNP 1H/13
PULPROG zgpg
TD 65536
SOLVENT CDCl3
NS 200
DS 0
SWH 19980.414 Hz
FIDRES 0.304810 Hz
AQ 1.8219456 sec
RG 91912
DW 27.800 usec
DE 20.00 usec
TE 300.0 K
D1 0.17593171 sec
d11 0.03000000 sec
d12 0.00002000 sec
======== CHANNEL f1 ========
NUC1 13C
P1 5.00 usec
PL1 6.00 dB
SFO1 75.4760973 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 100.00 usec
PL2 -1.00 dB
PL12 19.00 dB
PL13 25.20 dB
SFO2 300.1312005 MHz
SI 32768
SF 75.4677519 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 0.80
140 135 130 125 120 115 110 105 100 95 90 85 80 75 70 65 60 55 50 45 40 35 30 25 20 15 10 5 0 ppm


Current Data Parameters
NAME YSHR283 2nd spot 2D
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20060802
Time 14.44
INSTRUM spect
PROBHD 5 mm QNP 1H/13
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 16
DS 0
SWH 4664.179 Hz
FIDRES 0.142339 Hz
AQ 3.5127797 sec
RG 128
DW 107.200 usec
DE 6.00 usec
TE 300.0 K
D1 0.10000000 sec
======== CHANNEL f1 ========
NUC1 1H
P1 9.50 usec
PL1 -1.00 dB
SFO1 300.1321759 MHz
F2 - Processing parameters
SI 16384
SF 300.1300086 MHz
WDW EM
SSB 0
LB 0.10 Hz
GB 0
PC 0.80
1D NMR plot parameters
CX 22.00 cm
CY 58.87 cm
F1P 8.856 ppm
F1 2657.98 Hz
F2P -0.264 ppm
F2 -85.38 Hz
PPMCM 0.41548 ppm/cm
HZCM 124.69817 Hz/cm
OTBDPS
OBn
OH
OH
OH
N
36
more polar compound
ppm
Integral

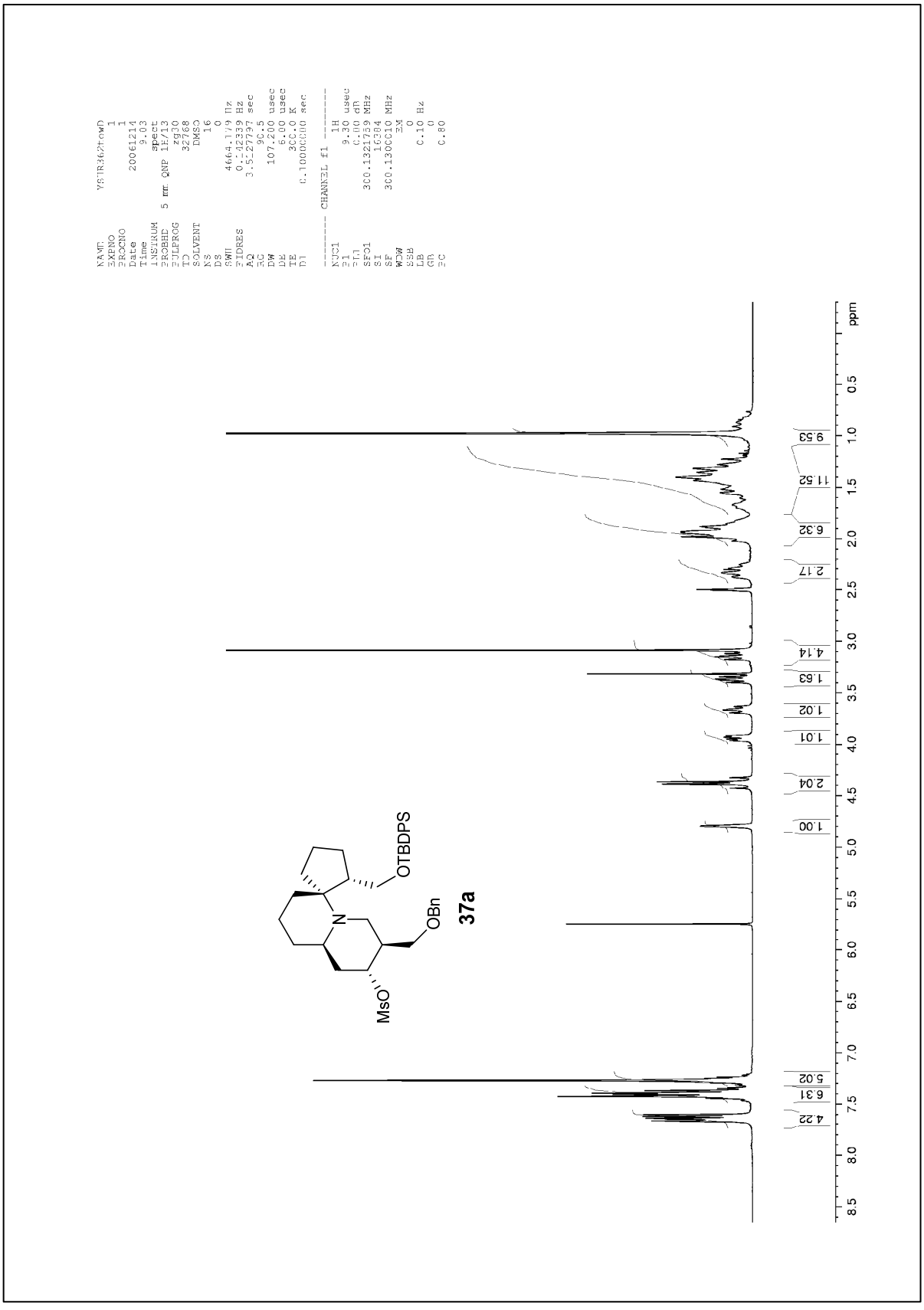

NAME YSHAR203 2nd spot 20
EXPNO 2
PROCNO 1
Date_ 20060802
Time 14.51
INSTRUM spect
PROBHD 5 mm QNP 1H/13
PULPROG zgpg
TD 65536
SOLVENT CDCl3
NS 200
DS 0
SWH 19980.414 Hz
FIDRES 0.304810 Hz
AQ 1.6214998 sec
RG 11585.2
DW 25.000 usec
DE 20.00 usec
TE 300.0 K
D1 0.17593171 sec
d11 0.03000000 sec
d12 0.00002000 sec
======== CHANNEL f1 ========
NUC1 13C
P1 5.00 usec
PL1 6.03 dB
SFO1 75.4760973 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 100.00 usec
PL2 -1.00 dB
PL12 19.00 dB
PL13 25.20 dB
SFO2 300.1312005 MHz
SI 32768
SF 75.4677557 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 0.80
140 135 130 125 120 115 110 105 100 95 90 85 80 75 70 65 60 55 50 45 40 35 30 25 20 15 10 5 0 ppm

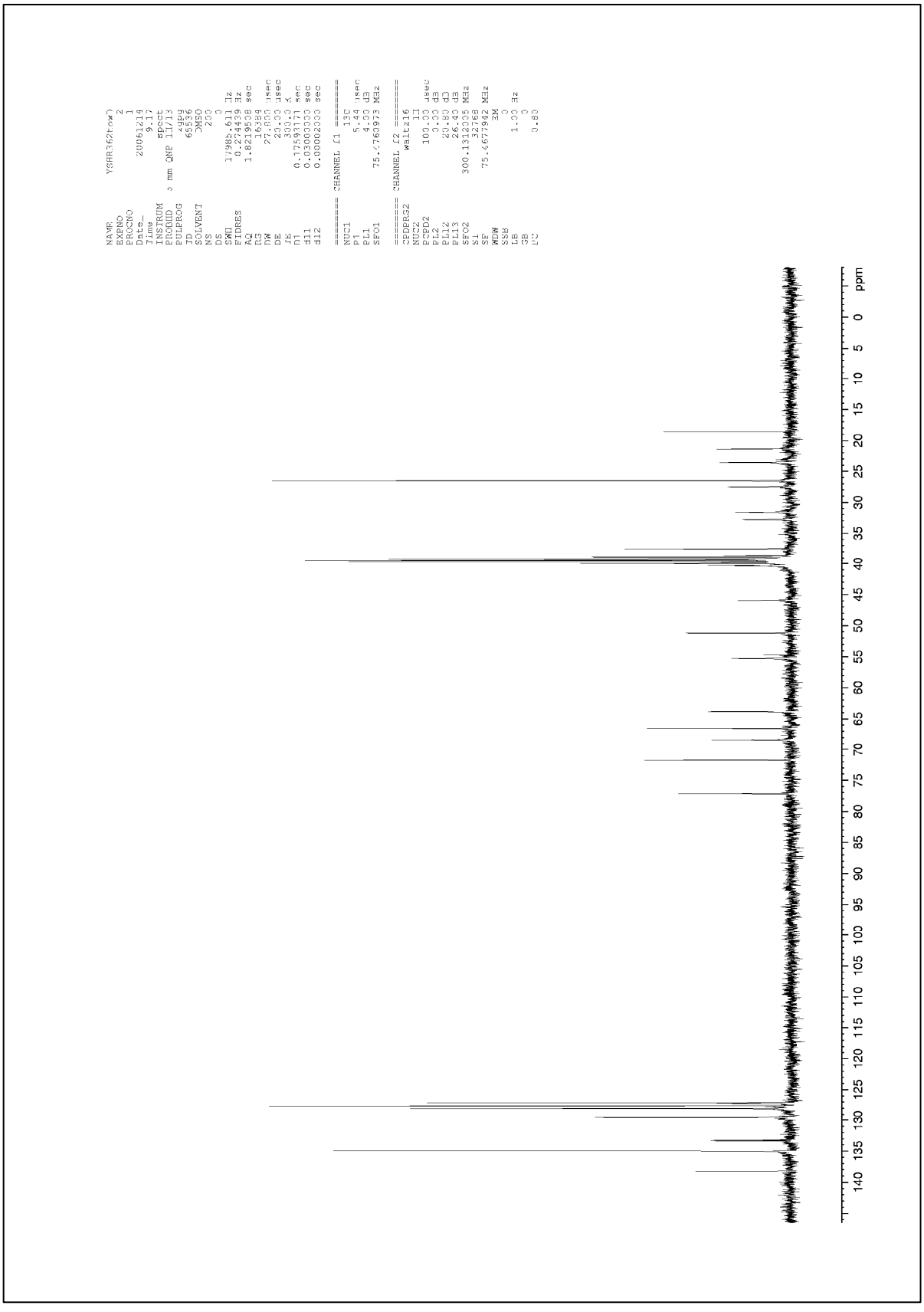

OBn
OH
OH
OTBDPS
N
H
27
less polar compound
9.15
16.24
0.97
1.90
1.84
1.20
0.98
1.00
1.97
10.44
3.77
ppm

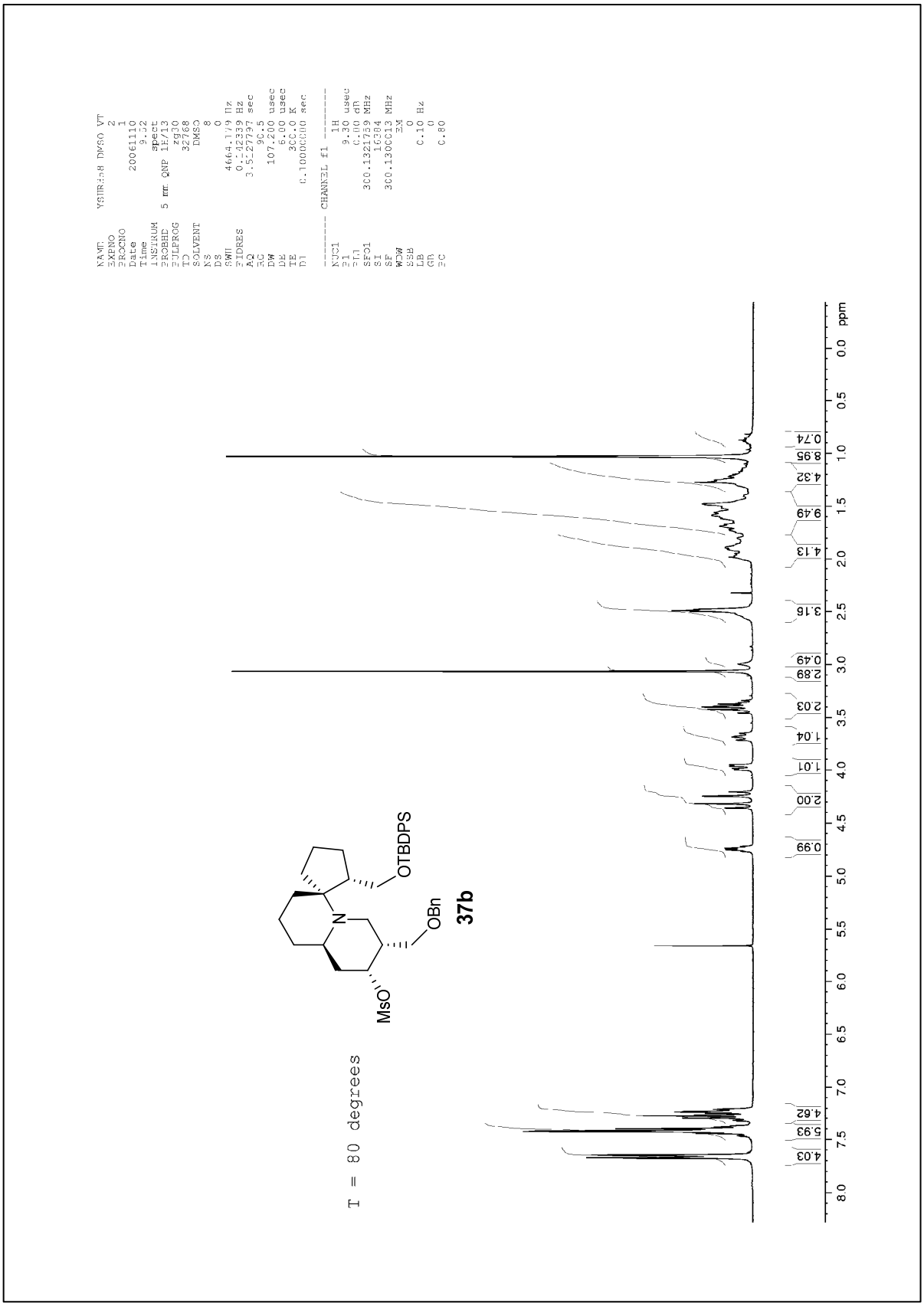

NAME YSHR352 22
EXPNO 2
PROCNO 1
Date_ 20061027
Time 11.35
INSTRUM spect
PROBHD 5 mm QNP 1H/13
PULPROG zgpg
TD 65536
SOLVENT CDCl3
NS 200
DS 0
SWH 11990.411 Hz
FIDRES 0.182948 Hz
AQ 1.8219456 sec
RG 16384
DW 27.800 usec
DE 20.00 usec
TE 300.0 K
D1 0.17593171 sec
D11 0.03000000 sec
D12 0.00002000 sec
======== CHANNEL f1 ========
NUC1 13C
P1 6.00 usec
PL1 6.03 dB
SFO1 75.4760973 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 100.00 usec
PL2 0.00 dB
PL12 20.80 dB
PL13 26.40 dB
SFO2 300.1312005 MHz
SI 32768
SF 75.4677590 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 0.80
ppm

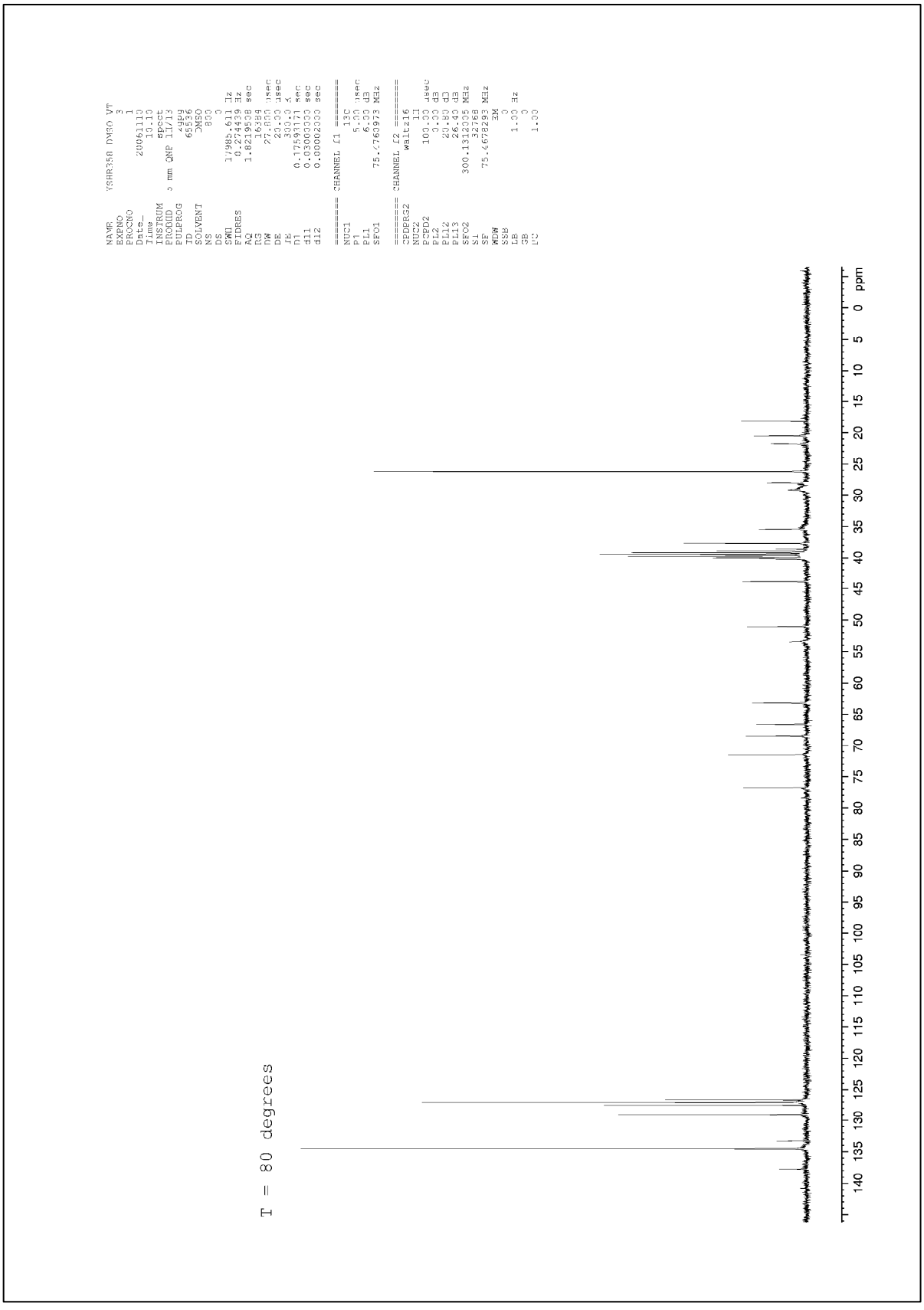

NAME YSHR354 NOESY
EXPNO 1
PROCNO 1
Date 20061028
Time 14.16
INSTRUM spect
PROBHD 5 mm QNP 1H/13
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 16
DS 0
SWH 4664.179 Hz
FIDRES 0.142339 Hz
AQ 3.5127797 sec
RG 32
DW 107.200 usec
DE 6.00 usec
TE 300.0 K
D1 0.10000000 sec
CHANNEL f1
NUC1 1H
P1 9.30 usec
PL1 0.00 dB
SFO1 300.1321739 MHz
SI 16384
SF 300.1300149 MHz
WDW EM
SSB 0
LB 0.10 Hz
GB 0
PC 0.80
OTBDPS
OBn
OH
OH
27
more polar compound
9.97
16.01
1.00
3.03
3.06
1.07
2.10
4.91
5.90
3.92
ppm

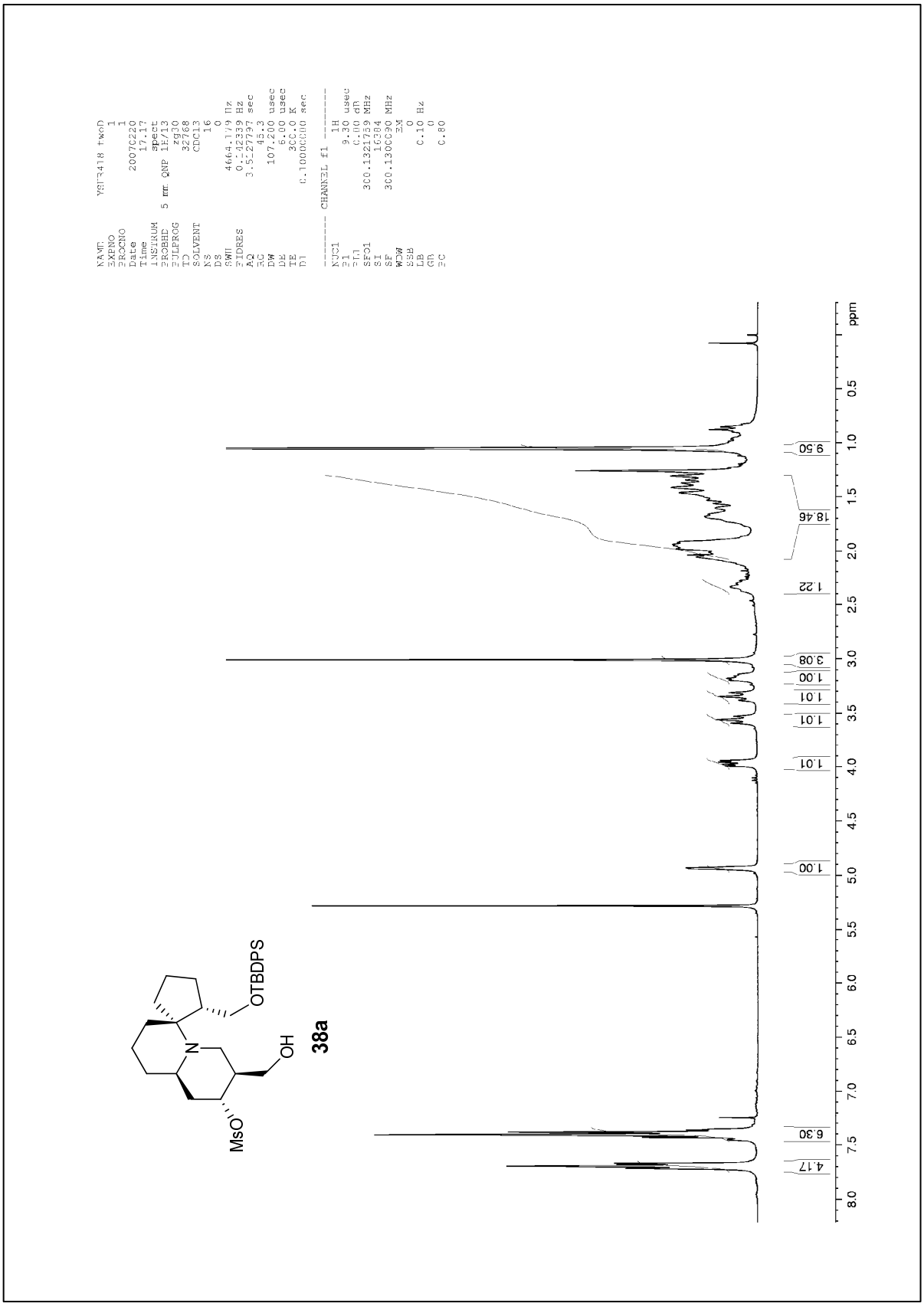

NAME YSHR354 NOESY
EXPNO 2
PROCNO 1
Date_ 20061028
Time 14.24
INSTRUM spect
PROBHD 5 mm QNP 1H/13
PULPROG 4gpy
TD 65536
SOLVENT CDCl3
NS 200
DS 0
SWH 11990.414 Hz
FIDRES 0.182948 Hz
AQ 1.821958 sec
RG 16384
DW 27.800 usec
DE 20.00 usec
TE 300.0 K
D1 0.17593171 sec
d11 0.03000000 sec
d12 0.00002000 sec
======== CHANNEL f1 ========
NUC1 13C
P1 5.00 usec
PL1 6.03 dB
SFO1 75.4760973 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 100.00 usec
PL2 0.00 dB
PL12 20.80 dB
PL13 26.40 dB
SFO2 300.1312005 MHz
SI 32768
SF 75.4677632 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 0.80
ppm

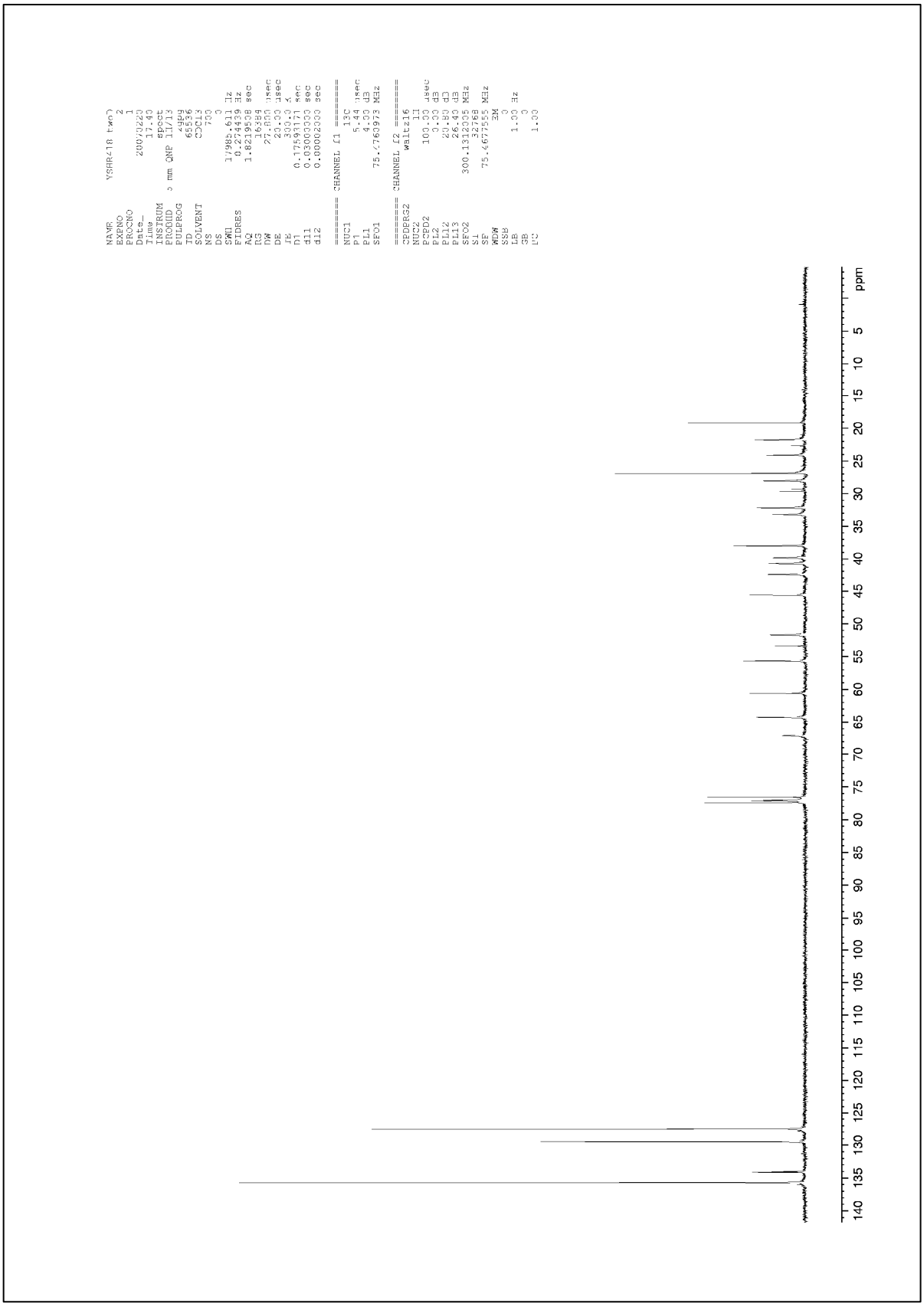

OTBDPS
OBn
MsO
37a
9.53
11.52
6.32
2.17
4.14
1.63
1.02
1.01
2.04
1.00
5.02
6.31
4.22
ppm

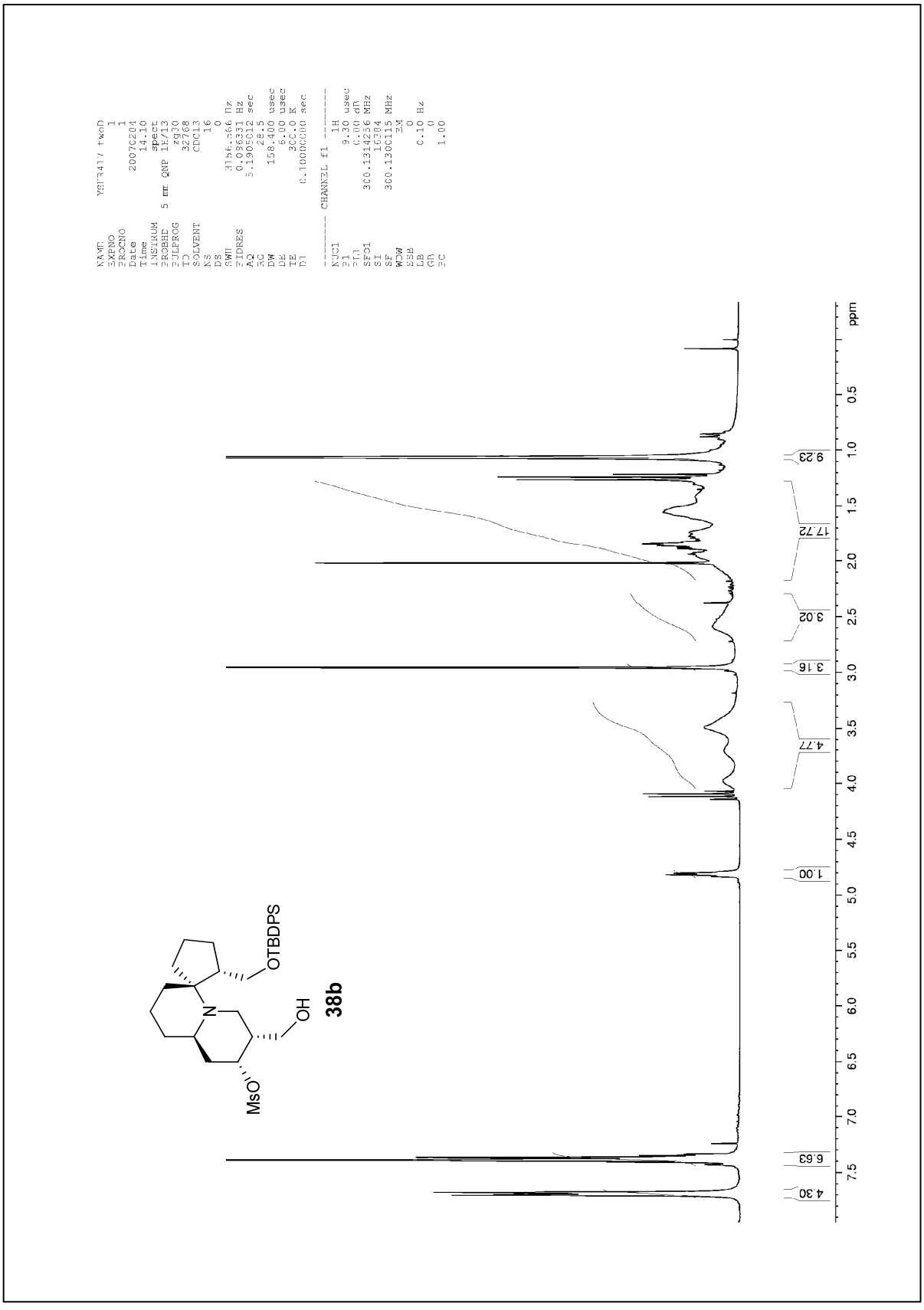

NAME YSHR362(new)
EXPNO 2
PROCNO 1
Date_ 20061214
Time 9.17
INSTRUM spect
PROBHD 5 mm QNP 1H/13
PULPROG zgpg
TD 65536
SOLVENT DMSO
NS 200
DS 0
SWH 19980.414 Hz
FIDRES 0.304810 Hz
AQ 1.8219520 sec
RG 16384
DW 27.800 usec
DE 20.00 usec
TE 300.0 K
D1 0.17593171 sec
D11 0.03000000 sec
D12 0.00002000 sec
CHANNEL f1
NUC1 13C
P1 5.44 usec
PL1 0.00 dB
SFO1 75.4769973 MHz
CHANNEL f2
CPDPRG2 waltz16
NUC2 1H
PCPD2 100.00 usec
PL2 0.00 dB
PL12 20.80 dB
PL13 26.40 dB
SFO2 300.1312005 MHz
SI 32768
SF 75.4677942 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 0.80
ppm

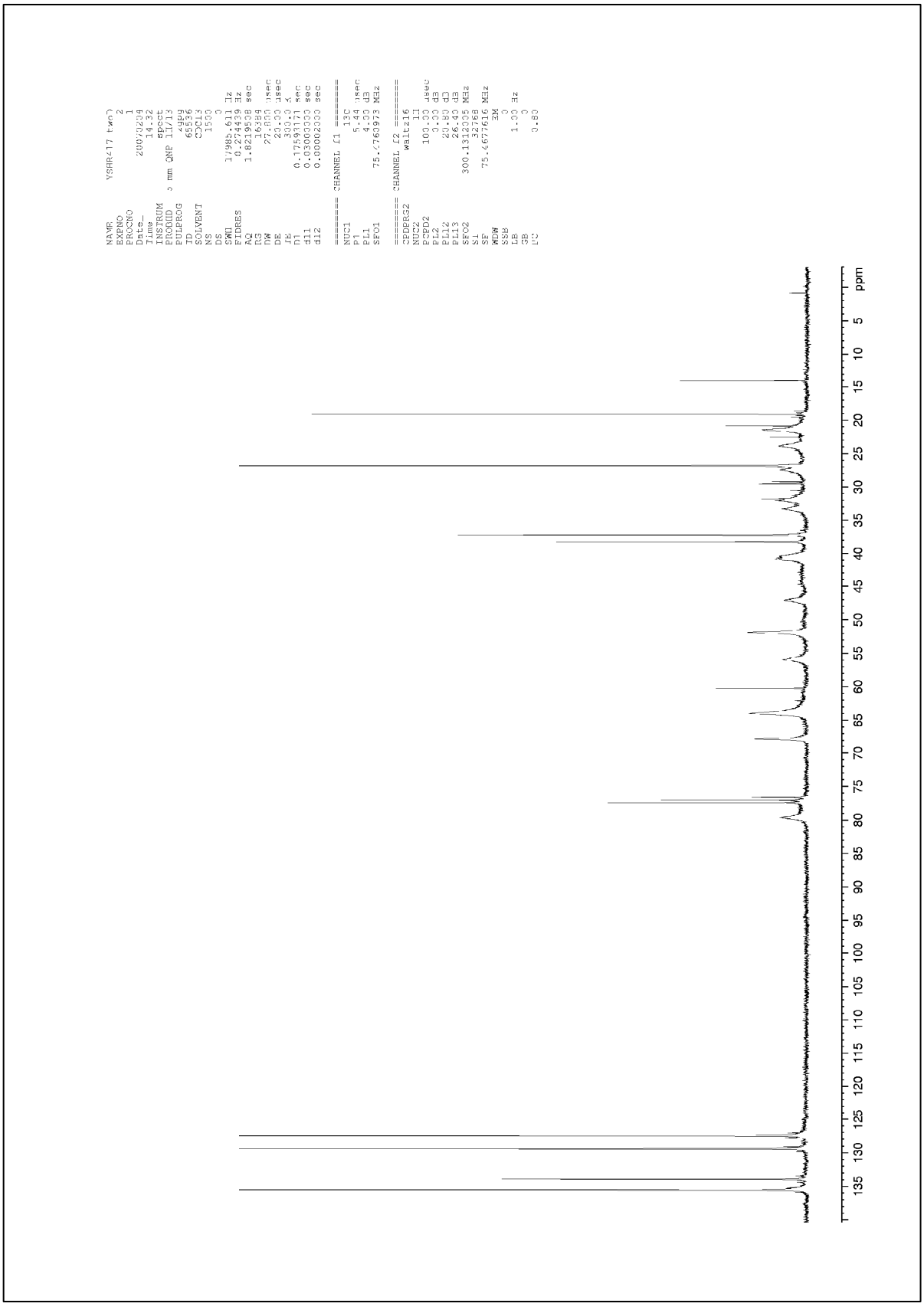

NAME YSTR::8 DMSO VT
EXPNO 2
PROCNO 1
Date 20061110
Time 9.52
INSTRUM spect
PROBHD 5 mm QNP 1H/13
PULPROG zg30
TD 32768
SOLVENT DMSO
NS 8
DS 0
SWH 4664.179 Hz
FIDRES 0.142339 Hz
AQ 3.5127797 sec
RG 90.5
DW 107.200 usec
DE 6.00 usec
TE 300.0 K
D1 0.10000000 sec
CHANNEL f1
NUC1 1H
P1 9.30 usec
PL1 0.00 dB
SFO1 300.1321739 MHz
SI 16384
SF 300.1300013 MHz
WDW EM
SSB 0
LB 0.10 Hz
GB 0
PC 0.80
T = 80 degrees
MsO
OBn
OTBDPS
37b
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
ppm
4.03
5.93
4.62
0.99
2.00
1.01
1.04
2.03
2.89
0.49
3.16
4.13
9.49
4.32
8.95
0.74

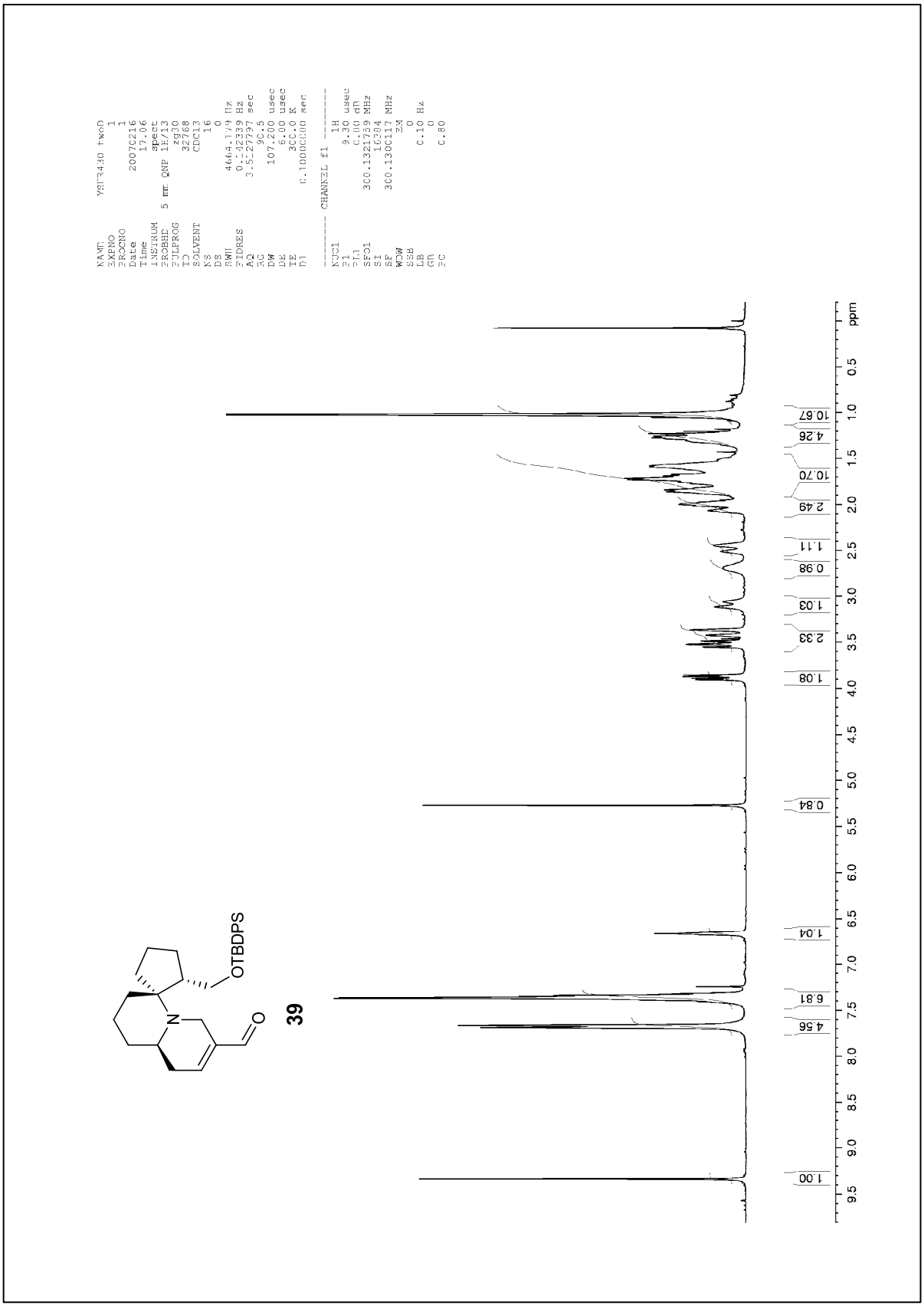

T = 80 degrees
ppm


38a
OTBDPS
OH
MsO
9.50
18.46
1.22
3.08
1.00
1.01
1.01
1.01
1.00
6.30
4.17
ppm

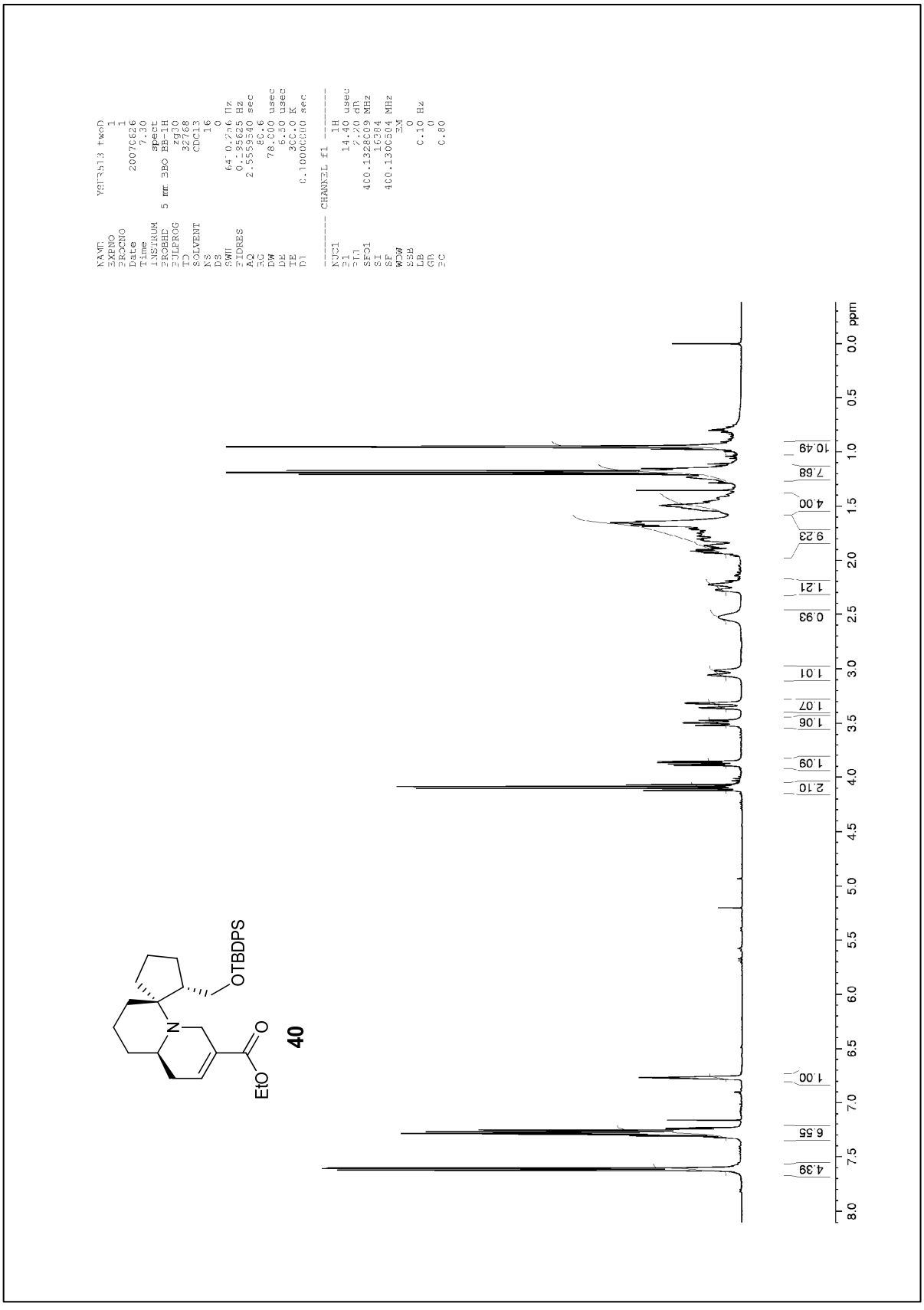

NAME YSHR418 two2
EXPNO 2
PROCNO 1
Date_ 20070220
Time 17.40
INSTRUM spect
PROBHD 5 mm QNP 1H/13
PULPROG zgpg
TD 65536
SOLVENT CDCl3
NS 700
DS 0
SWH 17985.611 Hz
FIDRES 0.274498 Hz
AQ 1.821955 sec
RG 16384
DW 27.800 usec
DE 20.00 usec
TE 300.0 K
D1 0.17593171 sec
D11 0.03000000 sec
D12 0.00002000 sec
======== CHANNEL f1 ========
NUC1 13C
P1 5.44 usec
PL1 0.00 dB
SFO1 75.4769973 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 100.00 usec
PL2 0.00 dB
PL12 20.80 dB
PL13 26.40 dB
SFO2 300.1312005 MHz
SI 32768
SF 75.4677555 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.00
ppm

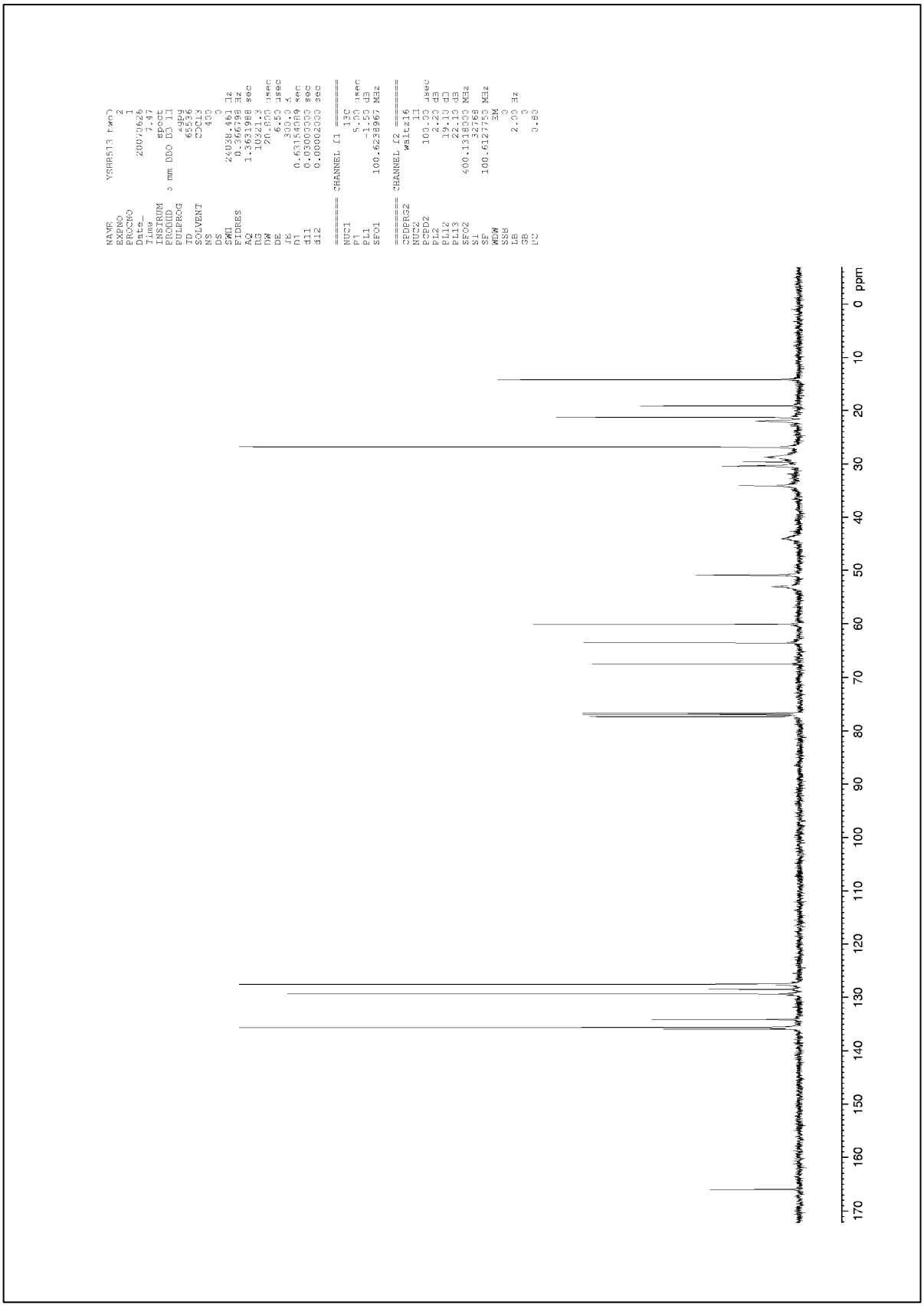

38b
OTBDPS
OH
MsO
9.23
17.72
3.02
3.16
4.77
1.00
6.63
4.30
ppm

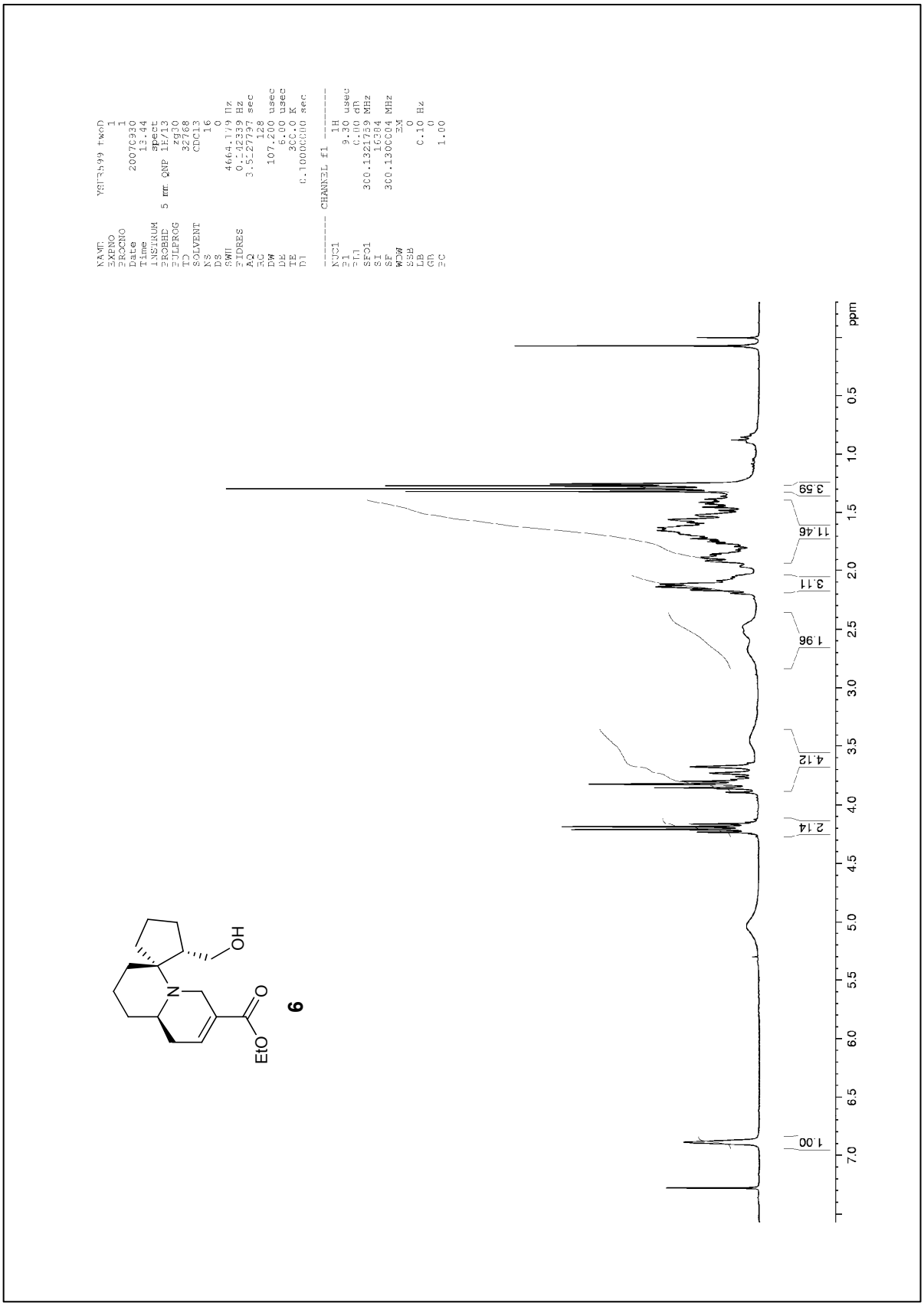

NAME YSHR417 two2
EXPNO 2
PROCNO 1
Date_ 20070204
Time 14.32
INSTRUM spect
PROBHD 5 mm QNP 1H/13
PULPROG zgpg
TD 65536
SOLVENT CDCl3
NS 1500
DS 0
SWH 11990.411 Hz
FIDRES 0.182948 Hz
AQ 1.8219456 sec
RG 16384
DW 27.800 usec
DE 20.00 usec
TE 300.0 K
D1 0.17593171 sec
d11 0.03000000 sec
d12 0.00002000 sec
======== CHANNEL f1 ========
NUC1 13C
P1 5.44 usec
PL1 4.03 dB
SFO1 75.4769973 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 100.00 usec
PL2 0.00 dB
PL12 20.80 dB
PL13 26.40 dB
SFO2 300.1312005 MHz
SI 32768
SF 75.4677616 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 0.80
ppm

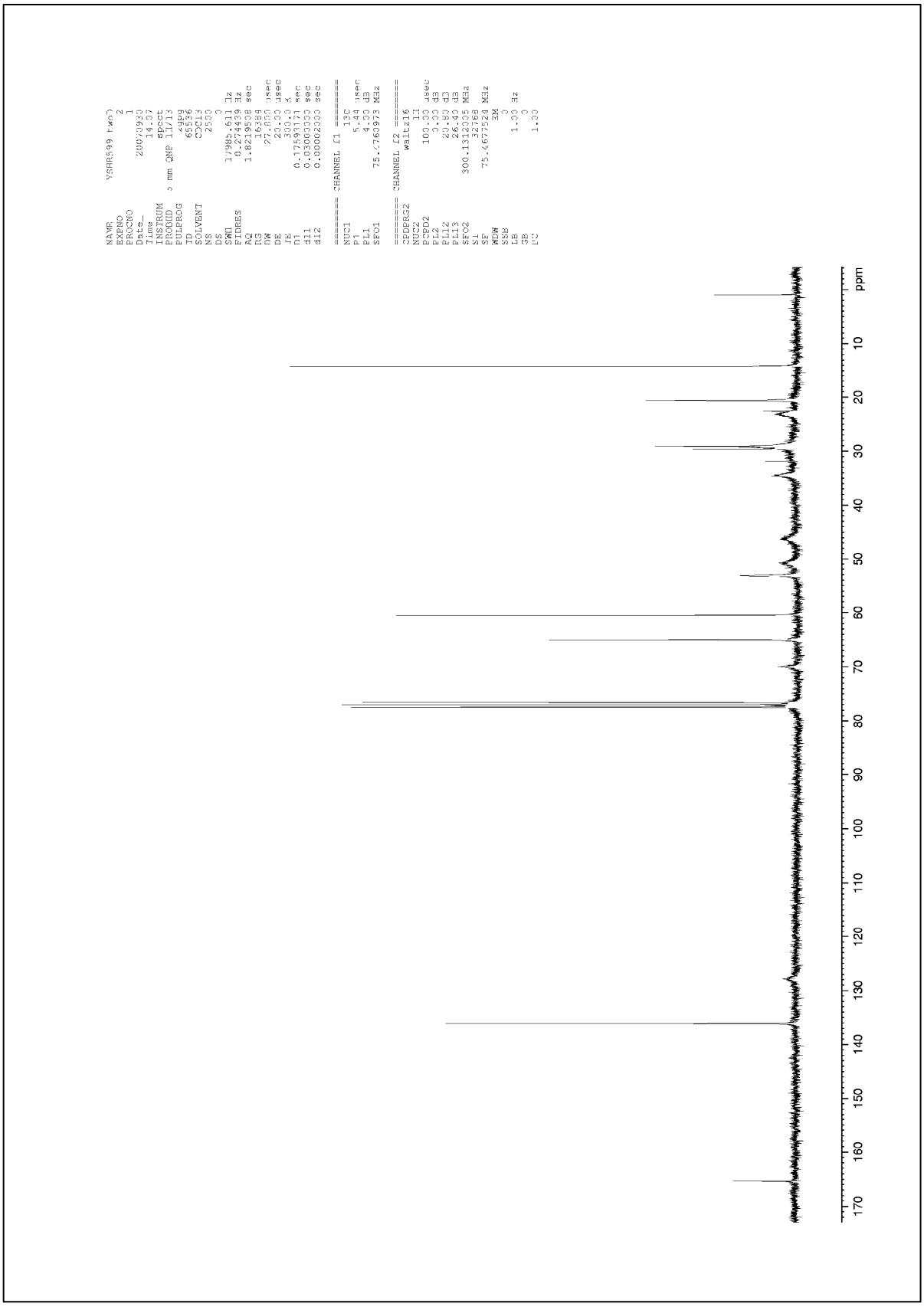

OTBDPS
N
O
39
1.00
6.81
4.56
1.04
0.84
1.08
2.33
1.03
0.98
1.11
2.49
10.70
4.26
10.67
ppm


NAME YSHR430 two2
EXPNO 2
PROCNO 1
Date_ 20070216
Time 17:29
INSTRUM spect
PROBHD 5 mm QNP 1H/13
PULPROG zgpg
TD 65536
SOLVENT CDCl3
NS 700
DS 0
SWH 19980.414 Hz
FIDRES 0.304749 Hz
AQ 1.8219599 sec
RG 16384
DW 27.800 usec
DE 20.00 usec
TE 300.0 K
D1 0.17593171 sec
D11 0.03000000 sec
D12 0.00002000 sec
======== CHANNEL f1 ========
NUC1 13C
P1 5.44 usec
PL1 0.00 dB
SFO1 75.4769973 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 100.00 usec
PL2 0.00 dB
PL12 20.80 dB
PL13 26.40 dB
SFO2 300.1312005 MHz
SI 32768
SF 75.4677550 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.00
ppm


OTBDPS
N
EtO
O
O
40
4.39
6.55
1.00
2.10
1.09
1.06
1.07
1.01
0.93
1.21
9.23
4.00
7.68
10.49
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0 ppm


NAME YSHR513 two2
EXPNO 2
PROCNO 1
Date_ 20070626
Time 7.47
INSTRUM spect
PROBHD 5 mm DDO DS 1H
PULPROG zgpg
TD 65536
SOLVENT CDCl3
NS 400
DS 0
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3631988 sec
RG 10321.7
DW 20.800 usec
DE 6.50 usec
TE 300.0 K
D1 0.63154089 sec
D11 0.03000000 sec
D12 0.00002000 sec
CHANNEL f1
NUC1 13C
P1 5.00 usec
PL1 -5.13 dB
SFO1 100.6239967 MHz
CHANNEL f2
CPDPRG2 waltz16
NUC2 1H
PCPD2 100.00 usec
PL2 2.20 dB
PL12 19.10 dB
PL13 22.10 dB
SFO2 400.1318000 MHz
SI 32768
SF 100.6127750 MHz
WDW EM
SSB 0
LB 2.00 Hz
GB 0
PC 0.80
0 ppm


NAME YBT35499 fwod
EXPNO 1
PROCNO 1
Date 20070930
Time 13.44
INSTRUM spect
PROBHD 5 mm QNP 1H/13
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 16
DS 0
SWH 4664.179 Hz
FIDRES 0.142339 Hz
AQ 3.5127739 sec
RG 128
DW 107.200 usec
DE 6.00 usec
TE 300.0 K
D1 0.10000000 sec
CHANNEL f1
NUC1 1H
P1 9.30 usec
PL1 0.00 dB
SFO1 300.1321739 MHz
SI 16384
SF 300.1300004 MHz
WDW EM
SSB 0
LB 0.10 Hz
GB 0
PC 1.00
OH
N
O
EtO
6
1.00
3.59
11.46
3.11
1.96
4.12
2.14
ppm


NAME YSHR599 two2
EXPNO 2
PROCNO 1
Date_ 20070930
Time 14.07
INSTRUM spect
PROBHD 5 mm QNP 1H/13
PULPROG zgpg
TD 65536
SOLVENT CDCl3
NS 2500
DS 0
SWH 19980.414 Hz
FIDRES 0.304810 Hz
AQ 1.8219508 sec
RG 16384
DW 27.800 usec
DE 20.00 usec
TE 300.0 K
D1 0.17593171 sec
D11 0.03000000 sec
D12 0.00002000 sec
======== CHANNEL f1 ========
NUC1 13C
P1 5.44 usec
PL1 4.03 dB
SFO1 75.4769973 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 100.00 usec
PL2 0.00 dB
PL12 20.80 dB
PL13 26.40 dB
SFO2 300.1312005 MHz
SI 32768
SF 75.4677524 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.00
ppm