Supporting Information for:

Stereocontrolled Synthesis of 2,3-Anhydro-β-D-lyxofuranosyl Glycosides

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General. Solvents were distilled from the appropriate drying agents before use. Unless stated otherwise, all reactions were carried out under a positive pressure of argon and were monitored by TLC on silica gel 60 F_{254} (0.25 mm, E. Merck). Spots were detected under UV light or by charring with 10% H_2SO_4 in ethanol. Solvents were evaporated under reduced pressure and below 40 ˚C (bath). Organic solutions of crude products were dried over anhydrous Na_2SO_4. Column chromatography was performed on silica gel 60 (40-60 μM). The ratio between silica gel and crude product ranged from 100 to 50:1 (w/w). Optical rotations were measured at 21±2 ˚C. Melting points are uncorrected. ^{1}H NMR spectra were recorded at 400 or 500 MHz, and chemical shifts are referenced to either TMS (0.0, $CDCl_3$) or external dioxane (3.75, D_2O). ^{13}C NMR spectra were recorded at 125 MHz, and ^{13}C chemical shifts are referenced to $CDCl_3$ (77.00, $CDCl_3$) or external dioxane (68.11, D_2O). Elemental analyses were performed by Atlantic Microlab Inc., Norcross, GA. Melting points are uncorrected. Electrospray mass spectra were recorded on samples suspended in THF or CH_3OH. Acceptors **8**–**13**, and **15** were prepared as previously reported;[1] acceptors **14**, and **16** as described below. Product yields from glycosylation reactions are given in Table 1.

***p*-Tolyl 2,3-anhydro-5-*O*-benzoyl-1-thio-α-D-lyxofuranoside** (**2**). Compound **4** (2.0 g, 7.8 mmol), triphenylphosphine (5.2 g, 20 mmol), and benzoic acid (1.42 g, 12 mmol) were dissolved in tetrahydrofuran (50 mL) and the solution was cooled to 0 °C. Diisopropylazodicarboxylate (3.86 mL, 19.5 mmol) was added dropwise over a period of 10 min. After complete addition, the reaction mixture was allowed to warm to room temperature and was stirred for 45 min. The solution was subsequently concentrated to yield a crude oil which upon trituration with cold diethyl ether precipitated triphenylphosphine oxide. The solid was filtered off and the filtrate was concentrated. The resulting oil was purified by chromatography (hexanes/EtOAc, 5:1) to obtain **2** (2.10 g, 82%) as a white crystalline solid: R_f 0.69 (hexanes/EtOAc, 3:1); $[\alpha]_D$ +125.4° (*c* 1.1, $CHCl_3$); mp 60–61 °C; ^{1}H NMR (500 MHz, $CDCl_3$, δ) 8.07 (dd, 2 H, *J* = 7.1, 0.9 Hz), 7.60 (dd, 1 H, *J* = 7.4, 7.3 Hz), 7.47–7.43 (m, 4 H), 7.13 (d, *J* = 8.0 Hz, 2 H), 5.52 (s, 1 H), 4.56 (dd, 1 H, *J* = 11.3, 5.7 Hz), 4.52 (dd, 1 H, *J* = 11.3, 5.7 Hz), 4.29 (dd, 1 H, *J* = 5.8 Hz), 3.95 (d, 1 H, *J* = 2.8 Hz), 3.84 (d, 1 H, *J* = 2.8 Hz), 2.34 (s, 3 H); ^{13}C NMR (125.7 MHz, $CDCl_3$, δ) 166.0, 138.2, 133.2, 133.1, 129.8, 129.6, 129.5, 128.4, 128.3, 87.0, 74.0, 62.2, 57.5, 55.5, 21.0. Anal. Calcd for $C_{19}H_{18}O_4S$: C, 66.66, 5.26. Found: C, 66.48; H, 5.34.

2,3-anhydro 5-*O*-benzoyl-α-D-lyxofuranosyl *p*-tolyl (R/S) sulfoxide (**3**). To a solution of **2** (1.0 g, 2.92 mmol) in CH_2Cl_2 (20 mL) at -78 °C was added *m*-chloroperbenzoic acid (0.55 g, 3.21 mmol). After stirring for 2 h the reaction mixture was warmed to room temperature and stirred for 30 min. The reaction mixture was washed with a saturated solution of $NaHCO_3$ and then water. The organic layer was dried, filtered, and concentrated to yield a crude oil which was purified by chromatography (hexanes/EtOAc, 2:1) to provide the title compounds **3 Fast** (0.400 g, 40%) and **3 Slow** (0.452 g, 38%) as white crystalline solids.

(**3 Fast**) R_f 0.36 (hexanes/EtOAc, 1:1); $[\alpha]_D$ -188.8° (*c* 1.5, $CHCl_3$); mp 71–72 °C; ^{1}H NMR (500 MHz, $CDCl_3$, δ) 8.11 (dd, 2 H, *J* = 7.4, 7.1 Hz), 7.61 (dd, 1 H, *J* = 7.4, 7.3 Hz), 7.63 (d, 2 H, *J* = 8.2 Hz), 7.50 (dd, 2 H, *J* = 7.4 Hz, 7.1 Hz), 7.40 (d, 2 H, *J* = 8.2 Hz), 4.79 (s, 1 H), 4.74 (dd, 1 H, *J* = 5.9, 5.9 Hz), 4.54 (dd, 2 H. *J* = 5.7, 1.7 Hz), 4.07–4.05 (m, 2 H), 2.48 (s, 3 H). ^{13}C NMR (125.7 MHz, $CDCl_3$, δ) 166.9, 142.2, 136.5, 133.3, 130.2, 129.7, 129.6, 128.4, 124.2, 96.1, 77.7, 62.4, 56.1, 55.2, 21.4. Anal. Calcd for $C_{19}H_{18}O_5S$: C, 63.68, 5.02. Found: C, 63.44; H, 5.06.

(**3 Slow** R_f 0.31 (hexanes/EtOAc, 1:1); $[\alpha]_D$ +196.0° (*c* 3.0, $CHCl_3$); mp 65–66 °C; ^{1}H NMR (500 MHz, $CDCl_3$, δ) 8.06 (dd, 2 H, *J* = 7.4, 7.1 Hz), 7.63–7.61 (m, 3 H), 7.49 (d, 2 H, *J* = 7.4, 7.1 Hz), 7.37 (d, 2 H, *J* = 8.2 Hz), 4.88 (s, 1 H), 4.64 (dd, 1 H, *J* = 6.1, 6.0 Hz), 4.49–4.42 (m, 2 H), 4.26 (d, 1 H, *J* = 2.8 Hz), 4.00 (dd, 1 H, *J* = 2.8, 0.8 Hz), 2.46 (s, 3 H); ^{13}C NMR (125.7 MHz, $CDCl_3$, δ) 166.0, 142.5, 136.2, 133.2, 129.9, 129.7, 129.6, 128.4, 125.2, 94.7, 78.7, 62.3, 56.5, 56.3, 21.5. Anal. Calcd for $C_{19}H_{18}O_5S$: C, 63.68, 5.02. Found: C, 63.49; H, 5.05.

General Procedures for Glycosylations

Method A: To a mixture of the alcohol (0.5 mmol, vacuum dried overnight), donor **2** (0.6 mmol) and 4 Å molecular sieves (0.1 g) was added CH_2Cl_2 (10 mL). The mixture was cooled to -40 °C and then *N*-iodosuccinimide (0.6 mmol) and silver triflate (0.15 mmol) were added. After stirring for 15–30 min at this temperature, the reaction mixture turned dark red/brown and then triethylamine was added. The reaction mixture was then diluted with CH_2Cl_2 and filtered through Celite. The filtrate was concentrated to give a crude residue which was purified by chromatography to obtain the product.

Method B: Donor **3** (0.5 mmol), 2,6-di-*tert*-butyl-4-methyl pyridine (2.0 mmol), 4 Å molecular sieves (0.1 g) were dried overnight under vacuum in the presence of P_2O_5. To this mixture was added CH_2Cl_2 (10 mL) and the reaction mixture was cooled to -78 °C. Triflic anhydride (0.6 mmol) was added and the mixture was allowed to stir for 10 min. A solution of

the vacuum dried alcohol (0.6 mmol) in CH_2Cl_2 (1.0 mL) was added via syringe dropwise over 5 min. After 15 min, the reaction mixture turned dark brown/green and a saturated solution of $NaHCO_3$ was added and then the solution was allowed to warm to room temperature. The resulting solution was filtered through Celite, dried, filtered, and concentrated to yield a crude oil which was purified by chromatography to obtain the product.

Methyl 5-*O*-(2,3-anhydro-β-D-lyxofuranosyl)-2,3-anhydro-α-D-lyxofuranoside (**14**). The compound was isolated after Zemplen deacylation[2] of methyl 5-*O*-(5-*O*-benzoyl-2,3-anhydro-α-D-lyxofuranosyl)-2,3-anhydro-α-D-lyxofuranoside[3] Chromatography (hexanes/EtOAc, 3:1) yielded the product as a white solid: R_f 0.15 (hexanes/EtOAc, 2:1); $[\alpha]_D$ +46.9 ° (*c* 0.6, $CHCl_3$); mp 113–114 °C; ^{1}H NMR (500 MHz, $CDCl_3$, δ) 5.30 (s, 1 H), 5.14 (s, 1 H), 4.19–4.15 (m, 2 H), 3.93–3.86 (m, 3 H), 3.76 (d, 1 H, *J* = 2.8 Hz), 3.74 (d, 1 H, *J* = 2.8 Hz), 3.72–3.66 (m, 3 H), 3.42 (s, 3 H); ^{13}C NMR (125.7 MHz, $CDCl_3$, δ) 102.6, 102.0, 76.8, 75.0, 66.9, 62.1, 56.6, 56.0, 54.6, 54.5. HRMS (ESI) calcd for (M+Na) $C_{11}H_{16}O_7$: 283.0794, found 283.0796.

Methyl 3,5-di-*O*-benzyl-α-D-arabinofuranoside (**16**). To a solution of methyl 5-*O*-benzyl-2,3-anhydro-α-D-lyxofuranoside[4] (1.2 g, 0.5 mmol) dissolved in dry DMF (5 mL) was added 1M sodium benzylate in benzyl alcohol (1.0 mL, 1.0 mmol) The reaction mixture was stirred at 100 °C for 2.5 h and then cooled and neutralized with acetic acid. The excess benzyl alcohol was removed by vacuum distillation and the crude oil was purified by chromatography (hexanes/EtOAc, 2:1) to yield the **16** (1.5 g, 84%) as a colorless oil: R_f 0.36 (hexanes/EtOAc, 2:1); $[\alpha]_D$ +125.4 ° (*c* 1.2, $CHCl_3$); ^{1}H NMR (500 MHz, $CDCl_3$, δ) 7.33–7.24 (m, 10 H), 4.89 (s, 1 H), 4.68 (d, 1 H, *J* = 12.3 Hz), 4.60 (d, 1 H, *J* = 11.8 Hz), 4.52 (d, 1 H, *J* = 12.3 Hz), 4.45 (d, 1 H, *J* = 11.8 Hz), 4.25 (dd, 1 H, *J* = 5.2, 2.5 Hz), 4.11 (d, 1 H, *J* = 10.6 Hz), 3.83 (d, 1 H, *J* = 2.8 Hz), 3.64 (dd, 1 H, *J* = 10.3, 2.3 Hz), 3.43 (dd, 1 H, *J* = 10.4, 2.4 Hz), 3.41 (s, 3 H), 3.29 (d, 1 H, *J* = 10.9 Hz); ^{13}C NMR (125.7 MHz, $CDCl_3$, δ) 138.1, 137.4, 128.9, 128.8, 128.4, 128.3, 128.2, 110.9, 85.3, 83.9, 78.3, 74.1, 72.5, 70.1, 55.6. HRMS (ESI) calcd for (M+Na) $C_{20}H_{24}O_5$: 367.1521, found 367.1530.

***n*-Octyl 2,3-anhydro-5-*O*-benzoyl-β-D-lyxofuranoside** (**17**). The compound was isolated after chromatography (hexanes/EtOAc, 6:1) as a colorless oil: R_f 0.46 (hexanes/EtOAc 3:1); $[\alpha]_D$ -34.8° (*c* 0.9, $CHCl_3$); ^{1}H NMR (500 MHz, $CDCl_3$, δ) 8.08 (d, 2 H, *J* = 7.1 Hz), 7.57 (dd, 1 H, *J* = 7.4, 7.3 Hz), 7.43 (dd, 2 H, *J* = 7.9, 7.6 Hz), 5.13 (s, 1 H), 4.55 (d, 1 H, *J* = 6.2 Hz),

4.20 (ddd, 1 H, *J* = 6.2, 6.1, 0.8 Hz), 3.82–3.78 (m, 2 H) 3.75 (d, 1 H, *J* = 2.9 Hz), 3.60–3.57 (m, 1 H) 1.65–1.26 (m, 12 H), 0.87 (t, 3 H, *J* = 6.8 Hz); ^{13}C NMR (125.7 MHz, $CDCl_3$, δ) 166.2, 133.1, 129.8, 129.7, 128.4, 101.6, 73.9, 69.8, 63.1, 55.8, 54.8, 31.8, 29.7, 29.3, 29.2, 25.9, 22.6, 14.1, $J_{C1\text{-}H1}$ = 164.1 Hz. HRMS (ESI) calcd for (M+Na) $C_{20}H_{28}O_5$ 371.1829, found 371.1798.

Cyclohexyl 2,3-anhydro-5-*O*-benzoyl-β-D-lyxofuranoside (**18**). The compound was isolated after chromatography (hexanes/EtOAc, 6:1) as white solid. R_f 0.42 (hexanes/EtOAc, 3:1); $[\alpha]_D$ -57.6° (*c* 1.4, $CHCl_3$); mp 74–75 °C; ^{1}H NMR (500 MHz, $CDCl_3$, δ) 8.12 (d, 2 H, *J* = 7.1 Hz), 7.61 (dd, 1 H, *J* = 7.4, 7.3 Hz), 7.45 (dd, 2 H, *J* = 7.9 Hz, 7.6 Hz), 5.26 (s, 1 H), 4.59 (dd, 2 H, *J* = 6.0, 3.0 Hz), 4.22 (ddd, 1 H, *J* = 6.3, 6.2, 0.9 Hz), 3.80 (dd, 1 H, *J* = 2.9, 0.9 Hz), 3.75 (d, 1 H, *J* = 2.9 Hz), 3.75–3.65 (m, 1 H), 1.95–1.18 (m, 10 H); ^{13}C NMR (125.7 MHz, $CDCl_3$, δ) 166.2, 133.1, 129.9, 129.7, 128.4, 100.3, 76.7, 73.9, 63.1, 56.4, 54.7, 33.4, 32.4, 25.5, 24.2, $J_{C1\text{-}H1}$ = 163.1 Hz. Anal. Calcd for $C_{18}H_{22}O_5$: C, 67.91, 6.97. Found: C, 67.89; H, 7.01.

***t*-Butyl 2,3-anhydro-5-*O*-benzoyl-β-D-lyxofuranoside** (**19**). The compound was isolated after chromatography (hexanes/EtOAc, 8:1) as an oil: R_f 0.52 (hexanes/EtOAc, 4:1); $[\alpha]_D$ -61.8° (*c* 1.2, $CHCl_3$); ^{1}H NMR (500 MHz, $CDCl_3$, δ) 8.10 (d, 2 H, *J* = 7.1 Hz), 7.60 (dd, 1 H, *J* = 7.4, 7.3 Hz), 7.48 (dd, 2 H, *J* = 7.9, 7.6 Hz), 5.32 (s, 1 H), 4.62–4.56 (m, 2 H), 4.19 (dd, 1 H, *J* = 6.9, 0.7 Hz), 3.78 (d, 1 H, *J* = 2.8 Hz), 3.70 (d, 1 H, *J* = 2.8 Hz), 1.33 (s, 9 H); ^{13}C NMR (125.7 MHz, $CDCl_3$, δ) 166.6, 133.5, 130.3, 130.1, 128.8, 96.9, 76.1, 74.3, 63.5, 57.6, 54.7, 28.9, $J_{C1\text{-}H1}$ = 164.5 Hz. HRMS (ESI) calcd for (M+Na) $C_{16}H_{20}O_5$ 315.1208, found 315.1206.

Also obtained was the corresponding α-glycoside as an oil: R_f 0.81 (hexanes/EtOAc, 2:1); $[\alpha]_D$ +7.1° (*c* 0.7, $CHCl_3$); ^{1}H NMR (500 MHz, $CDCl_3$, δ) 8.07 (d, 2 H, *J* = 7.1 Hz), 7.56 (dd, 1 H, *J* = 7.4, 7.3 Hz), 7.45 (dd, 2 H, *J* = 7.9, 7.6 Hz), 5.38 (s, 1 H), 4.55–4.44 (m, 2 H), 4.34 (dd, 1 H, *J* = 6.0, 5.8 Hz), 3.81 (d, 1 H, *J* = 2.8 Hz), 3.58 (d, 1 H, *J* = 2.8 Hz), 1.28 (s, 9 H). ^{13}C NMR (125.7 MHz, $CDCl_3$, δ) 166.1, 141.9, 133.7, 129.9, 129.7, 129.4, 95.9, 75.3, 73.3, 62.8, 54.5, 29.7, $J_{C1\text{-}H1}$ = 174.1 Hz. HRMS (ESI) calcd for (M+Na) $C_{16}H_{20}O_5$ 315.1208, found 315.1204.

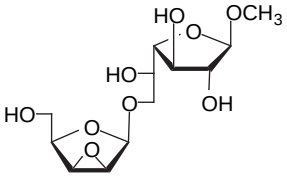
Methyl 5-*O*-(2,3-anhydro-5-*O*-benzoyl-β-D-lyxofuranosyl)-2,3-anhydro-α-D-lyxofuranoside (**20**). The compound was isolated after chromatography (hexanes/EtOAc, 3:1) as a white solid: R_f 0.29 (hexanes/EtOAc, 1:1); mp 94–95 °C; $[\alpha]_D$ -5.4° (*c* 1.5, $CHCl_3$); ^{1}H NMR (500 MHz, $CDCl_3$, δ) 8.06 (dd, 2 H, *J* = 8.5, 0.9 Hz), 7.56 (ddd, 1 H, *J* = 8.6, 8.6, 1.2 Hz, 1H), 7.45 (dd, 2 H, *J* = 7.8, 7.6 Hz), 5.23 (s, 1 H), 4.97 (s, 1 H), 4.57 (dd, 2 H, *J* = 6.4, 0.7 Hz),

4.25 (dd, 2 H, *J* = 10.4, 5.2 Hz), 4.02 (dd, 1 H, *J* = 10.4, 5.5 Hz), 3.86–3.77 (m, 4 H), 3.64 (d, 1 H, *J* = 2.9 Hz), 3.42 (s, 3 H); ^{13}C NMR (500 MHz, $CDCl_3$) δ 166.7, 133.6, 130.2, 128.9, 102.8, 102.2, 75.7, 74.7, 68.2, 63.5, 56.5, 56.2, 56.0, 55.4, 54.6, $J_{C1\text{-}H1}$ = 165.9, 172.8 Hz. Anal. Calcd for $C_{18}H_{20}O_8$: C, 59.34, 5.49. Found: C, 59.07; H, 5.52.

***n*-Octyl 5-*O*-(2,3-anhydro-5-*O*-benzoyl-β-D-lyxofuranosyl)-2,3-di-*O*-benzoyl-α-D-arabinofuranoside** (**21**). The compound was isolated after chromatography (hexanes/EtOAc, 4:1) as a clear oil: R_f 0.61 (hexanes/EtOAc, 2:1); $[\alpha]_D$ +96.8° (*c* 1.6, $CHCl_3$); ^{1}H NMR (500 MHz, $CDCl_3$, δ) 8.12–8.09 (m, 6 H), 7.61–7.46 (m, 9 H), 5.52 (d, 1 H, *J* = 1.3 Hz), 5.46 (d, 1 H, *J* = 4.7 Hz), 5.35 (s, 1 H), 5.30 (s, 1 H), 4.59 (d, 2 H, *J* = 11.0 Hz), 4.54–4.51 (m, 1 H), 4.32–4.26 (m, 2 H), 4.08 (dd, 1 H, *J* = 11.1, 6.3 Hz), 3.84 (m, 2 H), 3.57–3.55 (m, 1 H), 1.69–1.29 (m, 12 H), 0.92 (t, 3 H, *J* = 3.6 Hz); ^{13}C NMR (125.7 MHz, $CDCl_3$, δ) 166.2, 165.7, 165.4, 133.4, 133.3, 133.1, 130.1, 129.9, 129.8, 129.7, 129.4, 129.3, 128.5, 128.4, 128.3, 105.,7, 101.5, 82.0, 81.6, 77.6, 74.1, 68.2, 67.5, 63.0, 55.7, 54.8, 31.8, 29.6, 29.4, 29.3, 26.2, 22.6, 14.1, $J_{C1\text{-}H1}$ = 166.0 Hz. HRMS (ESI) calcd for (M+Na) $C_{39}H_{44}O_{11}$ 711.2776, found 711.2832.

Also obtained was the corresponding α-glycoside as an oil: R_f 0.76 (hexanes/EtOAc, 2:1); $[\alpha]_D$ +31.1° (*c* 0.6, $CHCl_3$); ^{1}H NMR (500 MHz, $CDCl_3$, δ) 8.11–8.08 (m, 6 H), 7.64–7.58 (m, 3 H), 7.50–7.45 (m, 6 H), 5.54 (d, 1 H, *J* = 4.8 Hz), 5.50 (d, 1 H, *J* = 1.2 Hz), 5.31 (s, 1 H), 5.28 (s, 1 H), 4.56–4.50 (m, 2 H), 4.44–4.40 (m, 2 H), 4.19 (d, 1 H, *J* = 11.2, 4.3 Hz), 3.98 (dd, 1 H, *J* = 11.2, 3.4 Hz), 3.84–3.79 (m, 1 H), 3.72 (d, 1 H, *J* = 2.8 Hz), 3.67 (d, 1 H, *J* = 2.8 Hz), 3.59–3.55 (m, 1 H), 1.72–129 (m, 12 H), 0.91 (t, 3 H, *J* = 3.5 Hz). ^{13}C NMR (125.7 MHz, $CDCl_3$, δ) 166.6, 166.1, 165.7, 133.9, 133.8, 133.5, 130.3, 130.2, 130.1, 129.9, 129.7, 128.9, 128.8, 128.7, 106.0, 101.9, 82.4, 82.3, 77.1, 74.6, 67.9, 67.4, 63.2, 56.6, 54.4, 32.2, 29.9, 29.8, 29.6, 26.5, 23.0, 14.5, $J_{C1\text{-}H1}$ = 173.7 Hz. HRMS (ESI) calcd for (M+Na) $C_{39}H_{44}O_{11}$ 711.2781, found 711.2781.

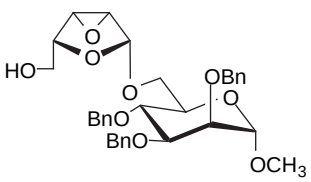
Methyl 5-*O*-(2,3-anhydro-β-D-lyxofuranosyl)-β-D-galactofuranoside (**22, de-*O*-benzoylated**). In order to purify the disaccharide from other organic impurities, the benzoyl esters were removed by standard Zemplén deacylation.[2] The compound was isolated after chromatography ($CHCl_3$/CH_3OH, 10:1) as a colorless oil: R_f 0.15 ($CHCl_3$/CH_3OH, 10:1);



$[\alpha]_D$ -63.2° (*c* 0.9, CH_3OH); ^{1}H NMR (400 MHz, D_2O, δ) 5.24 (s, 1 H), 4.88 (s, 1 H), 4.15–3.81 (m, 8 H), 3.81–3.63 (m, 3 H), 3.43 (s, 3 H); ^{13}C NMR (125.7 MHz, D_2O, δ) 108.7, 103.0, 102.9,

83.9, 81.7, 81.2, 77.6, 77.1, 76.8, 74.8, 71.7, 71.6, 70.9, 69.9, 60.8, 56.4, 56.1, 55.4, $J_{C1\text{-}H1}$ = 165.3 Hz.. HRMS (ESI) calcd for (M+Na) $C_{12}H_{20}O_9$: 331.0999, found 331.0978.

Methyl 6-*O*-(2,3-anhydro-β-D-lyxofuranosyl)-2,3,4-tri-*O*-benzyl-α-D-mannopyranoside (**23, de-*O*-benzoylated**). In order to purify the disaccharide from other organic impurities, the benzoyl esters were removed by standard Zemplén deacylation.[2] The compound was isolated after chromatography (hexanes/EtOAc, 4:1) as a colorless oil: R_f 0.55 (hexanes/EtOAc, 2:1); $[\alpha]_D$ +26.2° (*c* 2.9, $CHCl_3$); ^{1}H NMR (500 MHz, $CDCl_3$, δ) 7.40–7.26 (m, 15 H), 5.23 (s, 1 H), 4.94 (d, 1 H, *J* = 11 Hz), 4.76–4.75 (m, 3 H), 4.67 (d, 1 H, *J* = 11.0 Hz), 4.61 (s, 1 H), 4.10 (dd, 1 H, *J* =11, 1.4 Hz), 3.97–3.93 (m, 4 H), 3.67 (dd, 1 H, *J* = 10.1, 2.9 Hz), 3.34 (s, 3 H), 2.20 (br s, 1 H); ^{13}C NMR (125.7 MHz, $CDCl_3$, δ) 138.5, 138.4, 138.1, 128.3, 127.8, 127.6, 127.6, 127.5, 127.4, 101.6, 98.9, 80.7, 76.3, 74.8, 74.7, 74.3, 72.5, 72.0, 71.4, 67.8, 61.7, 55.1, 54.6, 54.4, $J_{C1\text{-}H1}$ = 166.0, 166.2. HRMS (ESI) calcd for (M+Na) $C_{33}H_{38}O_9$: 601.2414, found 601.2432.



Methyl 6-*O*-(2,3-anhydro-5-*O*-benzoyl-β-D-lyxofuranosyl)-2,3,4-tri-*O*-benzyl-α-D-galactopyranoside (**24**). The compound was isolated after chromatography (hexanes/EtOAc, 4:1) as a colorless oil: R_f 0.43 (hexanes/EtOAc, 2:1); $[\alpha]_D$ +9.1° (*c* 1.0, $CHCl_3$); ^{1}H NMR (500 MHz, $CDCl_3$, δ) 8.10 (dd, 2 H, *J* = 7.4, 7.3 Hz, 2 H), 7.60 (dd, 1 H, *J* = 7.1, 7.0 Hz), 7.49–7.30 (m, 17 H), 5.17 (s, 1 H), 5.00 (d, 1 H, *J* = 11.2 Hz), 4.89 (dd, 2 H, *J* = 11.7, 2.2 Hz), 4.78–4.68 (m, 4 H), 4.60–4.53 (m, 2 H), 4.25 (ddd, 1 H, *J* = 6.7, 6.7, 6.0 Hz), 4.09–4.07 (m, 1 H), 4.00–3.98 (m, 3 H), 3.89 (dd, 1 H, *J* = 10.3, 5.5, Hz), 3.81–3.78 (m, 2 H), 3.75 (d, 1 H, *J* = 3.0 Hz), 3.44 (s, 3 H); ^{13}C NMR (125.7 MHz, $CDCl_3$, δ) 166.1, 138.7, 138.6, 138.5, 122.1, 129.7, 129.6, 128.3, 128.1, 127.6, 127.5, 127.4, 102.0, 98.8, 79.0, 76.3, 75.3, 74.7, 74.2, 73.5, 73.3, 69.6, 68.9, 63.1, 55.7, 55.3, 54.8; $J_{C1\text{-}H1}$ = 166.3, 172.1. HRMS (ESI) calcd for (M+Na) $C_{40}H_{42}O_{10}$: 705.2676, found 705.2673.

Methyl 3-*O*-(2,3-anhydro-5-*O*-benzoyl-β-D-lyxofuranosyl)-3-*O*-benzyl-4,6-*O*-benzylidene-α-D-glucopyranoside (**25**). The compound was isolated after chromatography (hexanes/EtOAc, 4:1) as a white solid: R_f 0.36 (hexanes/EtOAc, 2:1); mp 94–95 °C; $[\alpha]_D$ -75.0° (*c* 1.3, $CHCl_3$); ^{1}H NMR (500 MHz, $CDCl_3$, δ) 8.09 (dd, 2 H, *J* = 7.4, 7.3 Hz), 7.57–7.30 (m, 13 H), 5.59 (s, 1 H), 5.40 (s, 1 H), 4.91 (d, 1 H, *J* = 12.1 Hz), 4.79 (d, 1 H, *J* = 12.1 Hz), 4.66–4.60 (m, 3 H), 4.33–4.18 (m, 3 H), 3.87–3.50 (m, 7 H), 3.40 (s, 3 H); ^{13}C NMR (125.7 MHz, $CDCl_3$,

δ) 166.1, 137.9, 137.3, 133.0, 129.7, 129.6, 128.6, 128.5, 128.3, 128.1, 128.0, 127.9, 125.8, 103.7, 100.9, 98.9, 80.1, 78.8, 78.7, 74.2, 73.7, 68.8, 63.0, 62.4, 56.2, 55.3; $J_{C1\text{-}H1}$ = 168.0, 163.5 Hz. HRMS (ESI) calcd for (M+Na) $C_{34}H_{36}O_{10}$: 613.2050, found 613.2048.

Also obtained was the corresponding α-glycoside as a white solid. The ^{1}H and ^{13}C NMR spectra were identical to those previously reported for this compound.[3]

Methyl 5-*O*-[5-*O*-(2,3-anhydro-5-*O*-benzoyl-β-D-lyxofuranosyl)-2,3-anhydro-α-D-lyxofuranosyl]-2,3-anhydro-α-D-lyxofuranoside (**26**). The compound was isolated after chromatography (hexanes/EtOAc, 2:1) as white solid. R_f 0.18 (hexanes/EtOAc, 1:1); $[\alpha]_D$ +11.8° (*c* 0.9, $CHCl_3$); mp 123–124 °C; ^{1}H NMR (500 MHz, $CDCl_3$, δ) 8.10 (dd, 2 H, *J* = 7.4, 7.3 Hz), 7.61 (d, 1 H, *J* = 7.3 Hz), 7.48 (dd, 2 H, *J* = 7.8, 7.6 Hz), 5.26 (s, 1 H), 5.17 (s, 1 H), 4.98 (s, 1 H), 4.60 (d, 2 H, *J* = 6.1 Hz), 4.33–4.27 (m, 2 H), 4.20 (dd, 1 H, *J* = 7.1, 5.6 Hz), 4.04 (dd, 1 H, *J* = 10.5, 5.6 Hz), 3.94 (dd, 1 H, *J* = 10.5, 5.6 Hz), 3.85 (dd, 1 H, *J* = 10.5, 6.6 Hz), 3.83 (br s, 3 H), 3.78 (d, 1 H, *J* = 2.9 Hz), 3.74–3.70 (m, 3 H); ^{13}C NMR (125.7 MHz, $CDCl_3$, δ) 166.2, 133.1, 129.8, 129.7, 128.8, 102.2, 101.8, 101.7, 75.5, 74.6, 74.3, 67.6, 66.4, 63.0, 56.3, 56.1, 55.8, 55.6, 54.9, 54.2, 54.1, $J_{C1\text{-}H1}$ = 166.9, 174.3, 173.7 Hz. HRMS (ESI) calcd for (M+Na) $C_{23}H_{26}O_{11}$: 501.1373, found 501.1372.

Methyl 3-*O*-(2,3-anhydro-5-*O*-benzoyl-β-D-lyxofuranosyl)-2,4,6-tri-*O*-benzyl-α-D-gulopyranoside (**27**). The compound was isolated after chromatography in (hexanes/EtOAc, 1:1) as a colorless oil: R_f 0.26 (hexanes/EtOAc, 2:1); $[\alpha]_D$ +26.1° (*c* 1.2, $CHCl_3$); ^{1}H NMR (500 MHz, $CDCl_3$, δ) 8.10 (d, 2 H, *J* = 8.0 Hz), 7.61 (dd, 1 H, *J* = 7.3, 7.3 Hz), 7.48–7.26 (m, 17 H), 5.45 (s, 1 H), 4.94 (dd, 1 H, *J* = 3.7, 3.7 Hz), 4.80–4.77 (m, 2 H), 4.73 (d, 1 H, *J* = 3.9 Hz), 4.70 (d, 1 H, *J* = 3.4 Hz), 4.60 (d, 2 H, *J* = 6.0 Hz), 4.47 (d, 1H, *J* = 12.0 Hz), 4.45 (d, 1 H, *J* = 12.0 Hz), 4.35 (dd, 1 H, *J* = 6.7, 5.7 Hz), 4.22 (dd, 1 H, *J* = 6.0, 5.8 Hz), 3.88–3.83 (m, 2 H), 3.81–3.79 (m, 2 H), 3.60 (dd, 1 H, *J* = 9.9, 2.9 Hz), 3.48 (s, 3 H); ^{13}C NMR (125.7 MHz, $CDCl_3$, δ) 166.6, 138.8, 138.5, 133.6, 130.1, 128.9, 128.8, 128.7, 128.4, 128.3, 128.2, 128.0, 127.8, 101.0, 98.6, 77.3, 73.7, 72.8, 72.2, 71.9, 69.9, 68.6, 65.5, 63.3, 56.2, 55.7, 54.0, $J_{C1\text{-}H1}$ = 169.9, 164.9 Hz HRMS (ESI) calcd for (M+Na) $C_{40}H_{42}O_{10}$: 705.2676, found 705.2654.

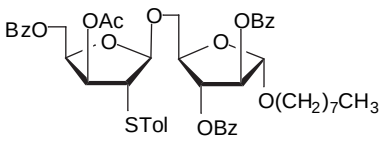
Also obtained was the corresponding α-glycoside as an oil: R_f 0.40 (hexanes/EtOAc, 2:1); ^{1}H NMR (500 MHz, $CDCl_3$, δ) 8.10 (d, 2 H, *J* = 8.0 Hz), 7.61 (dd, 1 H, *J* = 7.3, 7.3 Hz), 7.48–7.26 (m, 17 H), 5.37 (s, 1 H), 4.77 (d, 1 H, *J* = 3.9 Hz), 4.68–4.60 (m, 2 H), 4.57–4.45 (m, 4 H), 4.26–4.23 (m, 2 H), 4.07 (dd, 1 H, *J* = 3.3, 3.3 Hz), 3.92 (d, 1H, 2.8 Hz), 3.83–3.81 (m, 2

H), 3.67 (d, 1 H, *J* = 2.5 Hz), 3.62–3.54 (m, 2H), 3.45 (s, 3 H). ^{13}C NMR (125.7 MHz, $CDCl_3$, δ) 166.5, 138.6, 138.1, 133.6, 130.2, 130.1, 128.9, 128.8, 128.7, 128.6, 128.5, 128.5, 128.0, 127.9, 103.1, 98.7, 76.6, 74.3, 73.8, 73.2, 72.9, 72.1, 72.0, 69.5, 65.6, 63.2, 56.8, 56.3, 54.6. HRMS (ESI) calcd for (M+Na) $C_{40}H_{42}O_{10}$: 705.2676, found 705.2651.

Methyl 2-*O*-(2,3-anhydro-5-*O*-benzoyl-β-D-lyxofuranosyl)-3,5-di-*O*-benzyl-α-D-arabinofuranoside (**28**). The compound was isolated after chromatography in (hexanes/EtOAc, 2:1) as a colorless oil: R_f 0.22 (hexanes/EtOAc, 2:1); $[\alpha]_D$ +19.6 ° (*c* 1.9, $CHCl_3$); ^{1}H NMR (500 MHz, $CDCl_3$, δ) 8.10 (d, 2 H, *J* = 8.0 Hz), 7.58–7.28 (m, 13 H), 5.28 (s, 1 H), 5.03 (s, 1 H), 4.77 (d, 1 H, *J* = 11.9 Hz), 4.62–4.55 (m , 5 H), 4.43 (d, 1 H, *J* = 2.8 Hz), 4.28 (dd, 1 H, *J* = 6.0, 6.0 Hz), 4.25–4.23 (m, 1 H), 4.05 (dd, 1 H, *J* = 6.6, 3.3 Hz), 3.84 (d, 1 H, *J* = 2.8 Hz), 3.77 (d, 1 H, *J* = 2.8 Hz), 3.69–3.61 (m, 2 H), 3.45 (s, 3 H); ^{13}C NMR (125.7 MHz, $CDCl_3$, δ) 166.6, 138.5, 138.3, 133.6, 130.1, 128.8, 128.7, 128.4, 128.1, 128.0, 127.9, 108.2, 101.6, 87.6, 83.7, 81.3, 75.0, 73.8, 72.5, 70.2, 63.5, 56.5, 55.4, 55.3, $J_{C1\text{-}H1}$ = 172.5, 164.2 Hz. HRMS (ESI) calcd for (M+Na) $C_{32}H_{34}O_9$: 585.2101, found 585.2095.

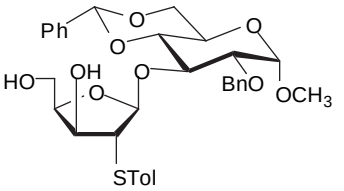
Also obtained was the corresponding α-glycoside as an oil: R_f 0.38 (hexanes/EtOAc, 2:1); $[\alpha]_D$ +34.0° (*c* 0.5, $CHCl_3$); ^{1}H NMR (500 MHz, $CDCl_3$, δ) 8.11 (d, 2 H, *J* = 8.0 Hz), 7.55 (dd, 1 H, *J* = 7.8, 7.5 Hz), 7.5–7.3 (m, 12 H), 5.04 (s, 1 H), 4.97 (s, 1 H), 4.63 (d, 1 H, *J* = 2.9 Hz), 4.60 (d, 1 H, *J* = 7.8 Hz), 4.55 (dd, 1 H, *J* = 6.0, 1.4 Hz), 4.35 (dd, 1 H, *J* = 5.9, 5.5 Hz), 4.23–4.21 (m, 1 H), 4.17 (dd, 1 H, *J* = 2.4, 0.5 Hz), 3.88 (dd, 1 H, *J* = 6.3, 2.4 Hz), 3.80 (d, 1 H, *J* = 2.8 Hz), 3.68 –3.61 (m, 2 H), 3.56 (d, 1 H, *J* = 2.8 Hz), 3.52 (d, 1 H, *J* = 2.7 Hz), 3.40 (s, 3 H). ^{13}C NMR (125.7 MHz, $CDCl_3$, δ) 166.6, 138.4, 138.1, 133.6, 130.2, 130.1, 128.9, 128.8, 128.7, 128.6, 128.3, 128.2, 128.1, 108.4, 101.3, 87.5, 83.8, 81.1, 74.6, 73.8, 72.8, 69.8, 63.2, 56.5, 55.5, 54.4, $J_{C1\text{-}H1}$ = 172.5, 174.1 Hz. HRMS (ESI) calcd for (M+Na) $C_{32}H_{34}O_9$: 585.2101, found 585.2098.

***n*-Octyl 5-*O*-(3-*O*-acetyl-5-*O*-benzoyl-2-deoxy-2-*p*-thiocresyl-β-D-xylofuranosyl)-2,3-di-*O*-benzoyl-α-D-arabinofuranoside** (**29, 3´-*O*-Acetate**) This compound and the α-isomer of **21** had the same R_f. Separation required acetylation (Ac_2O, pyridine) of the mixture followed by chromatography (hexanes/EtOAc, 4:1) which provided the compound as a colorless oil: R_f 0.21 (hexanes/EtOAc, 3:1); $[\alpha]_D$ –21.6° (*c* 0.7, $CHCl_3$); ^{1}H NMR (500 MHz, $CDCl_3$, δ) 8.12–8.04 (m, 6 H), 7.65–7.55 (m, 3 H), 7.56–7.38 (m, 6 H), 7.37 (d, 2 H,

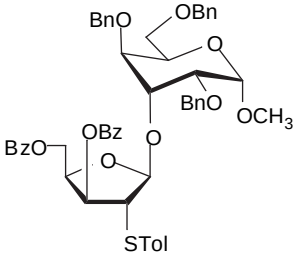


J = 7.9 Hz), 7.13 (d, 2 H, *J* = 7.9 Hz), 5.49 (d, 1 H, *J* = 1.2 Hz), 5.41 (d, 1 H, *J* = 4.7 Hz), 4.38 (dd, 1 H, *J* = 5.8, 3.4 Hz), 5.27 (s, 2 H), 4.78 (dd, 1 H, *J* = 12.0, 5.9 Hz), 4.64–4.50 (m, 2 H), 4.46–4.43 (m, 1 H), 4.19 (d, 1 H, *J* = 10.9, 3.6 Hz), 3.85 (dd, 1 H, *J* = 10.9, 6.1 Hz), 3.80 (dd, 1 H, *J* = 3.3, 1.7 Hz), 3.79–3.72 (m, 1 H), 3.54–3.49 (m, 1 H), 2.34 (s, 3 H), 1.98 (s, 3 H), 1.69–1.62 (m, 2 H), 1.44–1.29 (m, 10 H), 0.91 (t, 3 H, *J* = 3.6 Hz); ^{13}C NMR (125.7 MHz, $CDCl_3$, δ) 170.4, 166.5, 166.1, 165.7, 138.4, 133.8, 133.7, 132.9, 130.3, 130.2, 130.1, 130.1, 130.0, 130.0, 129.8, 129.7, 129.4, 128.9, 128.8, 128.7, 108.3, 106.1, 96.2, 82.4, 82.2, 78.3, 78.2, 76.9, 76.2, 68.2, 67.9, 63.9, 56.5, 32.2, 30.0, 29.9, 29.7, 26.6, 23.1, 21.5, 21.1, 14.5. HRMS (ESI) calcd for (M+Na) $C_{48}H_{54}O_{12}S$: 877.3234, found 877.3234.

Methyl 3-*O*-(2-deoxy-2-*p*-thiocresyl-β-D-xylofuranosyl)-2-*O*-benzyl-4,6-*O*-benzylidene-α-D-glucopyranoside (30, de-*O*-benzoylated). This compound and the α-isomer of **25** had the same R_f. Separation required debenzoylation[2] of the mixture followed by chromatography (hexanes/EtOAc, 3:1), which provided the compound as a white solid: R_f 0.20 (hexanes/EtOAc, 2:1); $[\alpha]_D$ -29.5° (*c* 1.0, $CHCl_3$); mp 159–160 °C, ^{1}H NMR (500 MHz, $CDCl_3$, δ) 7.54 (d, 2 H, *J*= 7.8 Hz), 7.42–7.31 (m, 10 H), 7.11 (d, 2 H, *J* = 7.7 Hz), 5.61 (s, 1 H), 5.52 (s, 1 H), 4.63 (d, 1 H, *J* = 12.2 Hz), 4.58 (d, 1 H, *J* = 3.6 Hz), 4.51 (d, 1 H, *J* = 2.1 Hz), 4.39–4.24 (m, 6 H), 3.89 (ddd, 1 H, *J* = 10.0, 4.9, 4.8 Hz), 3.76–3.69 (m, 2 H), 3.61–3.47 (m, 4 H), 3.42 (s, 3 H), 2.31 (s, 3 H); ^{13}C NMR (125.7 MHz, $CDCl_3$, δ) 138.0, 137.9, 136.9, 132.3, 131.3, 130.4, 130.3, 129.9, 129.3, 128.9, 128.8, 128.6, 128.5, 126.9, 108.4, 102.9, 99.1, 82.7, 80.4, 80.2, 75.2, 73.5, 69.4, 61.9, 61.6, 59.9, 55.9, 22.9. HRMS (ESI) calcd for (M+Na) $C_{33}H_{38}O_9S$: 633.2134, found 633.2136.

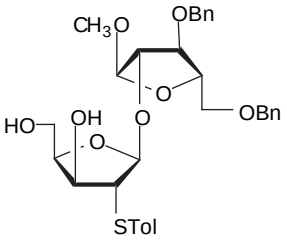


Methyl 3-*O*-(2-deoxy-2-*p*-thiocresyl-3,5-di-*O*-benzoyl-β-D-xylofuranosyl)-2,4,6-tri-*O*-benzyl-α-D-gulopyranoside (31, 3´-*O*-benzoate). This compound and the α-isomer of **27** had the same R_f. Separation required benzoylation (BzCl, pyridine) of the mixture followed by chromatography (hexanes/EtOAc, 4:1), which provided the product as a colorless oil: R_f 0.50 (hexanes/EtOAc, 2:1); ^{1}H NMR (500 MHz, $CDCl_3$, δ) 8.09–8.07 (m, 4 H), 7.45–7.29 (m, 23 H), 7.06 (d, 2 H, *J* = 8.2 Hz), 5.18 (s, 1 H), 5.11 (d, 1 H, *J* = 3.2 Hz), 4.88 (dd, 1 H, *J* = 11.9, 3.4 Hz), 4.73–4.53 (m, 6 H), 4.33 (dd, 1 H, *J* = 3.4, 3.4 Hz), 4.21–4.19 (m, 3 H), 3.94 (dd, 1H, *J* = 4.2 3.3 Hz), 3.83 (dd, 1 H, *J* = 3.8, 3.8 Hz), 3.75 (d, 1 H, *J* = 2.8 Hz), 3.69



(d, 1 H, J = 2.8 Hz), 3.63–3.52 (m, 3 H), 3.43 (s, 3 H), 2.27 (s, 3 H); ^{13}C NMR (125.7 MHz, $CDCl_3$, δ) 166.6, 166.5, 138.2, 138.1, 137.9, 137.8, 134.0, 133.5, 132.0, 130.6, 130.3, 130.2, 130.1, 128.9, 128.8, 128.7, 128.6, 128.5, 128.4, 128.3, 128.0, 127.9, 100.6, 98.7, 80.8, 79.3, 74.9, 73.7, 73.1, 70.2, 69.6, 65.1, 63.4, 56.2, 56.0, 55.9, 55.3, 21.5. HRMS (ESI) calcd for (M+Na) $C_{54}H_{54}O_{11}S$: 933.3285, found 933.3281.

Methyl 2-*O*-(2-deoxy-2-*p*-thiocresyl-β-D-xylofuranosyl)-3,5-di-*O*-benzyl-α-D-arabinofuranoside (32, de-*O*-benzoylated). This compound and the α-isomer of **28** had the same R_f. Separation required debenzoylation[2] of the mixture followed by chromatography (hexanes/EtOAc, 3:2), which provided the compound as a colorless oil: R_f 0.40 (hexanes/EtOAc, 3:2); $[\alpha]_D$ +12.3° (*c* 0.6, $CHCl_3$); ^{1}H NMR (500 MHz, $CDCl_3$, δ) 7.43–7.32 (m, 12 H), 7.18 (d, 2 H, J = 7.9 Hz), 5.19 (s, 1 H), 4.83 (s, 1 H), 4.69 (d, 1 H, J = 11.8 Hz), 4.62 (d, 1 H, J = 12.1 Hz), 4.55 (d, 2 H, J = 12.2 Hz), 4.41 (dd, 1 H, J = 4.9, 3.3 Hz), 4.29–42.6 (m, 2 H), 4.19–4.18 (m, 1 H), 4.12 (dd, 1 H, J = 6.1, 5.0 Hz), 3.86 (m, 2 H), 3.72 (s, 1 H), 3.66–3.55 (m, 4 H), 3.43 (dd, 1 H, J = 11.1, 8.0 Hz), 3.39 (s, 3 H), 2.36 (s, 3 H); ^{13}C NMR (125.7 MHz, $CDCl_3$, δ) 138.3, 138.1, 138.0, 132.4, 130.5, 129.9, 128.8, 128.7, 128.3, 128.2, 128.1, 126.7, 107.8, 107.7, 86.7, 83.3, 82.6, 81.6, 77.2, 73.9, 72.5, 69.6, 62.3, 59.3, 55.4, 21.5. HRMS (ESI) calcd for (M+Na) $C_{32}H_{38}O_8S$: 605.2185, found 605.2183.



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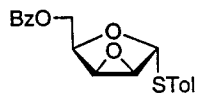

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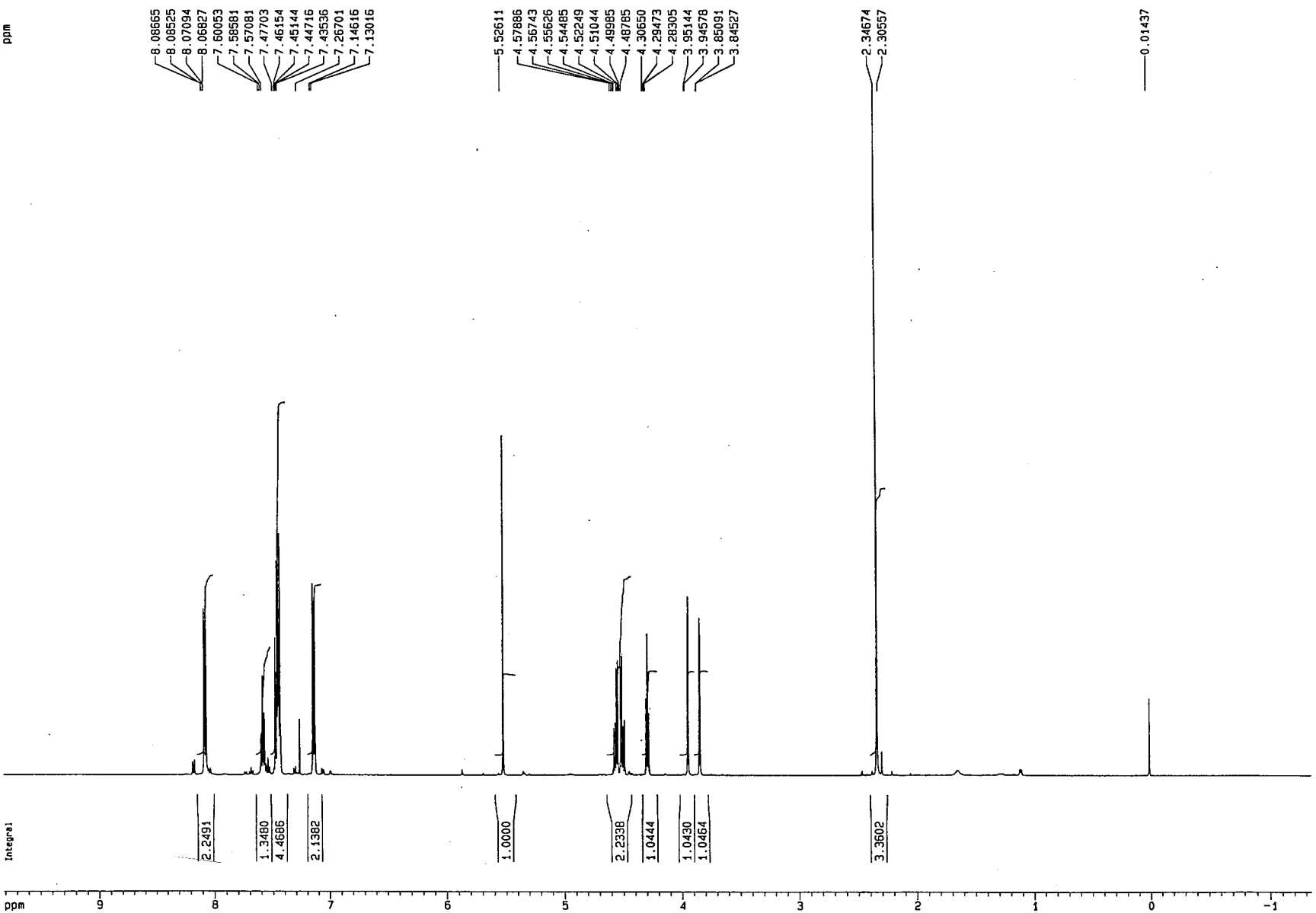


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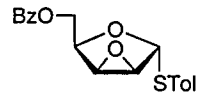
Date_           20000726
Time                9.08
INSTRUM            spect
PROBHD     5 mm BBO BB-1
PULPROG             zg30
TD                 65536
SOLVENT            CDCl3
NS                     5
DS                     2
SWH           10330.578 Hz
FIDRES        0.157632 Hz
AQ            3.1719923 sec
RG                   128
DW                48.400 usec
DE                 6.00 usec
TE                300.0 K
D1            1.00000000 sec

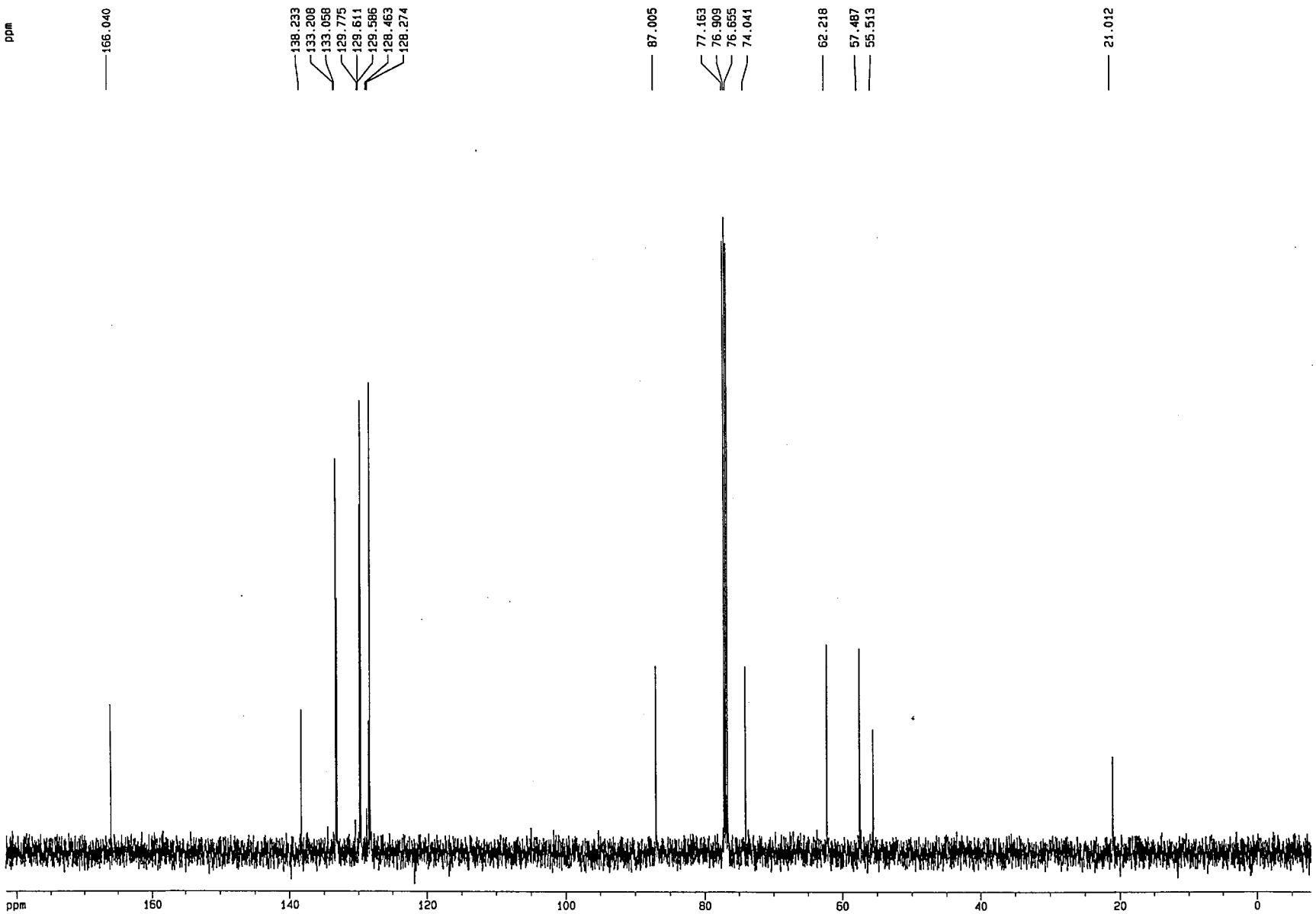
============ CHANNEL f1 ============
NUC1                1H
P1                13.70 usec
PL1               -1.00 dB
SFO1         500.1330885 MHz

F2 - Processing parameters
SI                 32768
SF           500.1300200 MHz
WDW                   EM
SSB                    0
LB                0.30 Hz
GB                     0
PC                1.00

1D NMR plot parameters
CX                34.00 cm
F1P               9.812 ppm
F1              4907.15 Hz
F2P              -1.342 ppm
F2              -671.13 Hz
PPMCM          0.32805 ppm/cm
HZCM          164.05723 Hz/cm
```



2




```
Current Data Parameters
NAME          CoupleFinal
EXPNO                 101
PROCNO                  1

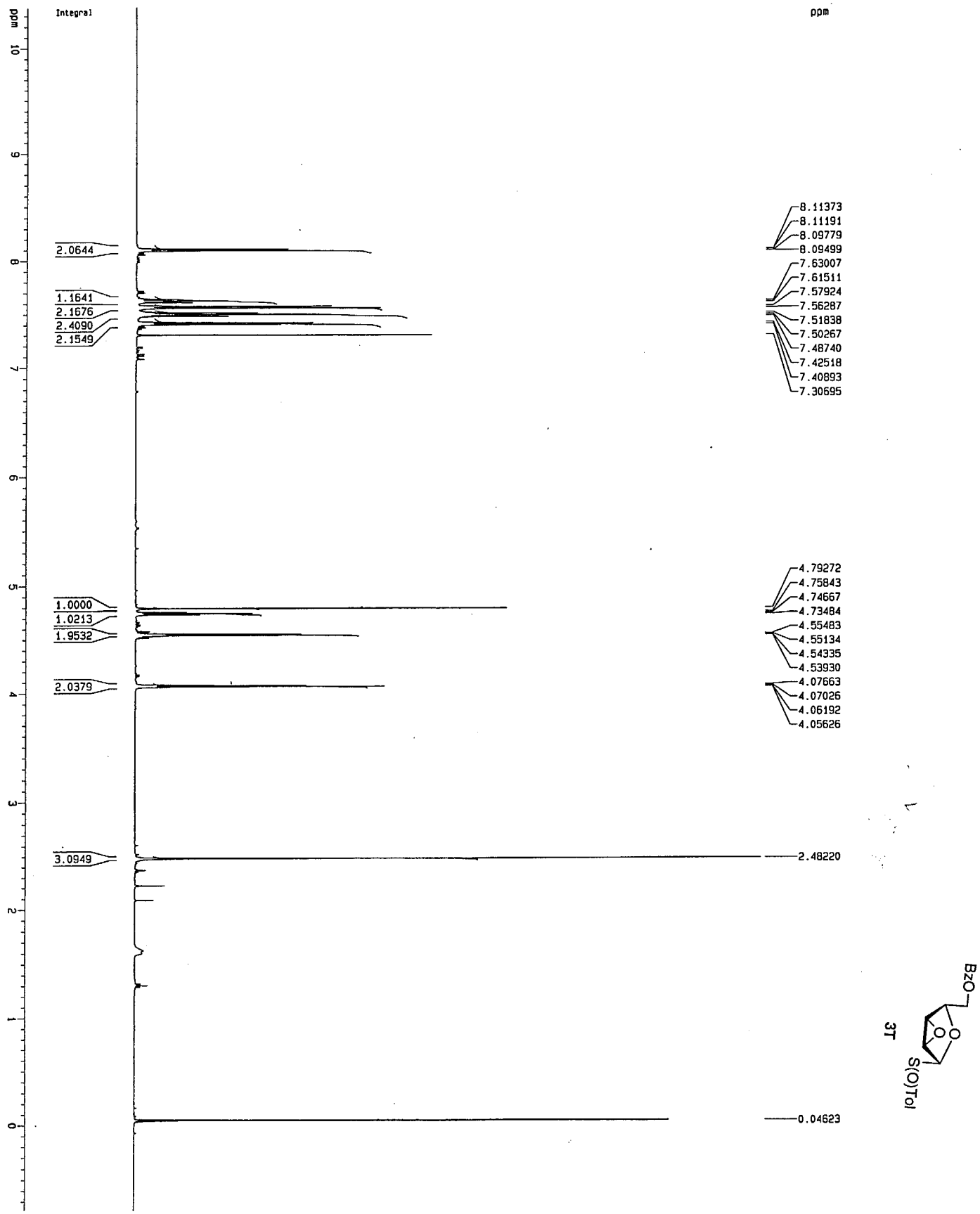
F2 - Acquisition Parameters
Date_          20000726
Time               9.14
INSTRUM           spect
PROBHD    5 mm BBO BB-1
PULPROG         zgpg30
TD               65536
SOLVENT          CDCl3
NS                  23
DS                   4
SWH          31446.541 Hz
FIDRES         0.479836 Hz
AQ             1.0420724 sec
RG                8192
DW              15.900 usec
DE               6.00 usec
TE              300.0 K
D1           2.00000000 sec
D11          0.03000000 sec
D12          0.00002000 sec

========== CHANNEL f1 ==========
NUC1               13C
P1              8.00 usec
PL1             3.00 dB
SFO1       125.7715719 MHz

========== CHANNEL f2 ==========
CPDPRG2         waltz16
NUC2                1H
PCPD2          100.00 usec
PL2             -1.00 dB
PL12            18.80 dB
PL13            18.80 dB
SFO2       500.1320005 MHz

F2 - Processing parameters
SI                32768
SF          125.7578137 MHz
WDW                  EM
SSB                   0
LB                 1.00 Hz
GB                    0
PC                 1.40

1D NMR plot parameters
CX                34.00 cm
F1P              181.570 ppm
F1             22833.88 Hz
F2P               -7.684 ppm
F2              -956.30 Hz
PPMCM             5.56630 ppm/cm
HZCM            700.00513 Hz/cm
```



3T

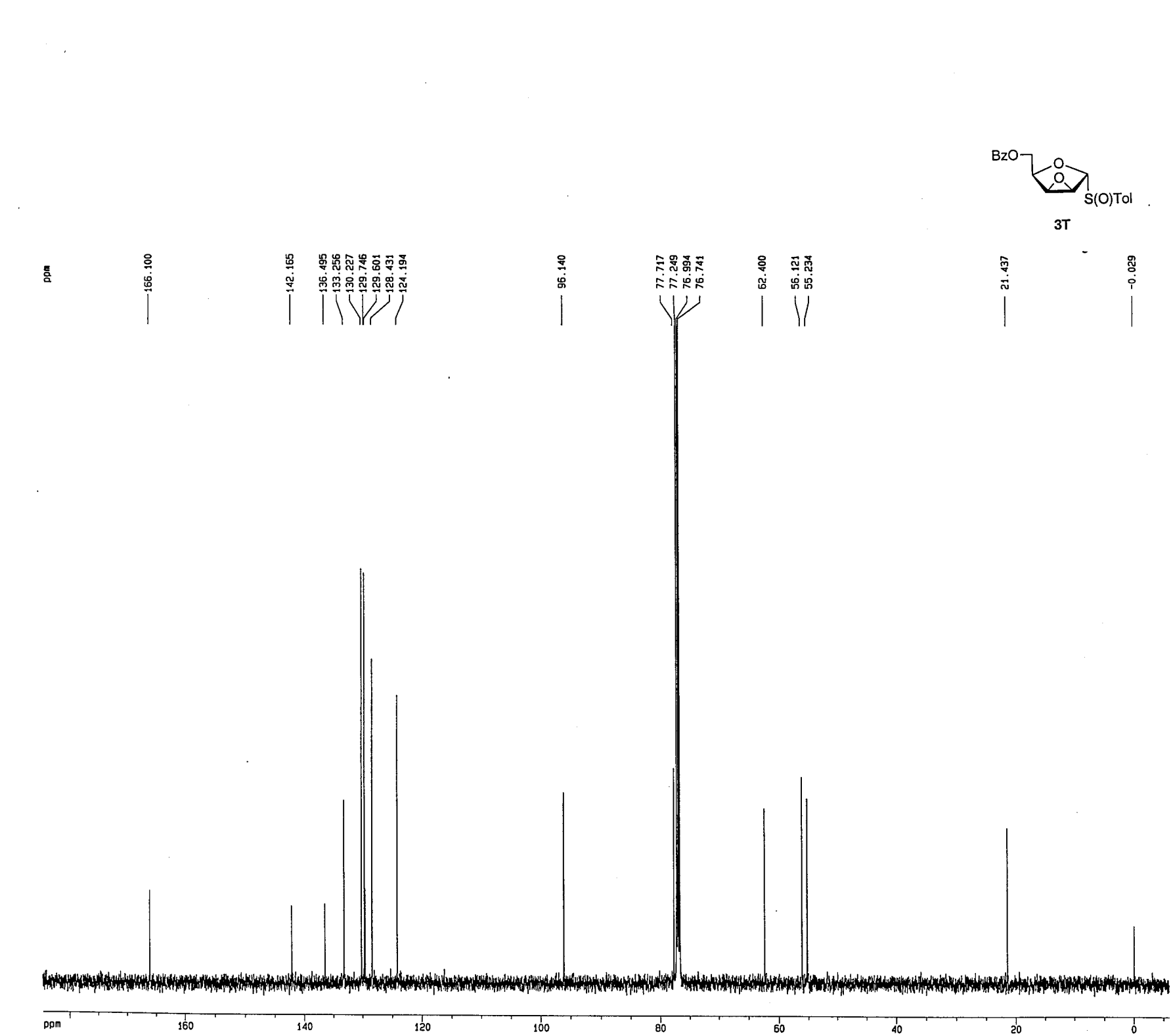
```
Current Data Parameters
NAME                  CSC-9-7-00
EXPNO                         10
PROCNO                         1

F2 - Acquisition Parameters
Date_              20000907
Time                       8.13
INSTRUM                    spect
PROBHD     5 mm BBO BB-1
PULPROG                    zg30
TD                        65536
SOLVENT                   CDCl3
NS                           65
DS                            2
SWH                10330.578 Hz
FIDRES              0.157632 Hz
AQ                  3.1719323 sec
RG                          362
DW                       48.400 usec
DE                        6.00 usec
TE                       300.0 K
D1                  1.00000000 sec

========== CHANNEL f1 ==========
NUC1                        1H
P1                       13.70 usec
PL1                      -1.00 dB
SFO1          500.1330885 MHz

F2 - Processing parameters
SI                        32768
SF          500.1300000 MHz
WDW                        EM
SSB                         0
LB                       0.30 Hz
GB                          0
PC                       1.00

1D NMR plot parameters
CX                       34.00 cm
F1P                   10.372 ppm
F1                  5187.44 Hz
F2P                   -0.781 ppm
F2                   -390.85 Hz
PPMCM                 0.32805 ppm/cm
HZCM                164.06723 Hz/cm
```



Current Data Parameters
NAME CSC-9-7-00
EXPNO 11
PROCNO 1

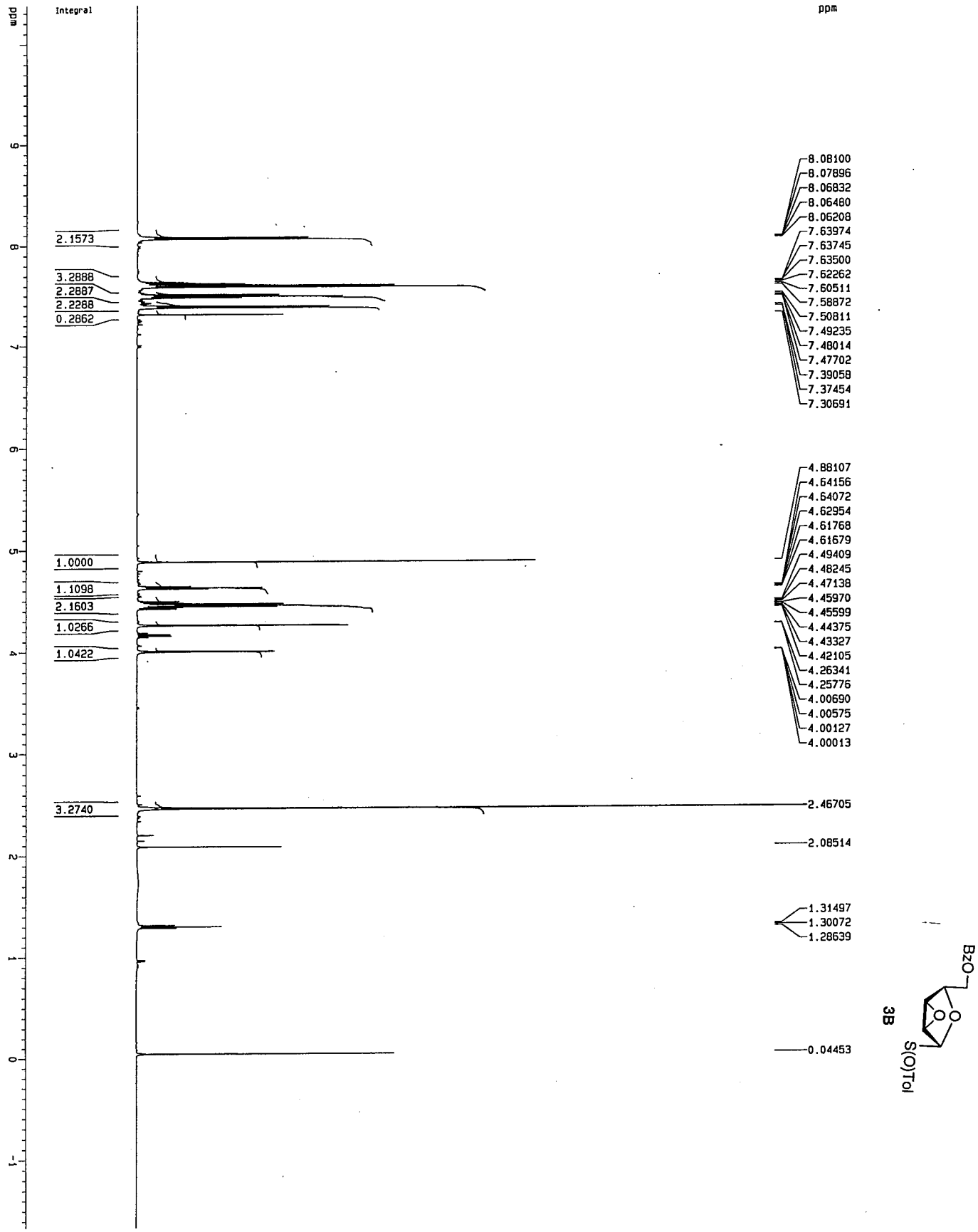
F2 - Acquisition Parameters
Date_ 20000907
Time 8.23
INSTRUM spect
PROBHD 5 mm BBO BB-1
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 850
DS 4
SWH 31446.541 Hz
FIDRES 0.479836 Hz
AQ 1.0420724 sec
RG 16384
DW 15.900 usec
DE 6.00 usec
TE 300.0 K
D1 2.00000000 sec
D11 0.03000000 sec
D12 0.00002000 sec

---------- CHANNEL f1 ----------
NUC1 13C
P1 8.00 usec
PL1 3.00 dB
SFO1 125.7715719 MHz

---------- CHANNEL f2 ----------
CPDPRG2 waltz16
NUC2 1H
PCPD2 100.00 usec
PL2 -1.00 dB
PL12 18.80 dB
PL13 18.80 dB
SFO2 500.1320005 MHz

F2 - Processing parameters
SI 32768
SF 125.7577946 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

1D NMR plot parameters
CX 34.00 cm
F1P 184.452 ppm
F1 23197.49 Hz
F2P -5.888 ppm
F2 -740.45 Hz
PPMCM 5.59852 ppm/cm
HZCM 704.05731 Hz/cm



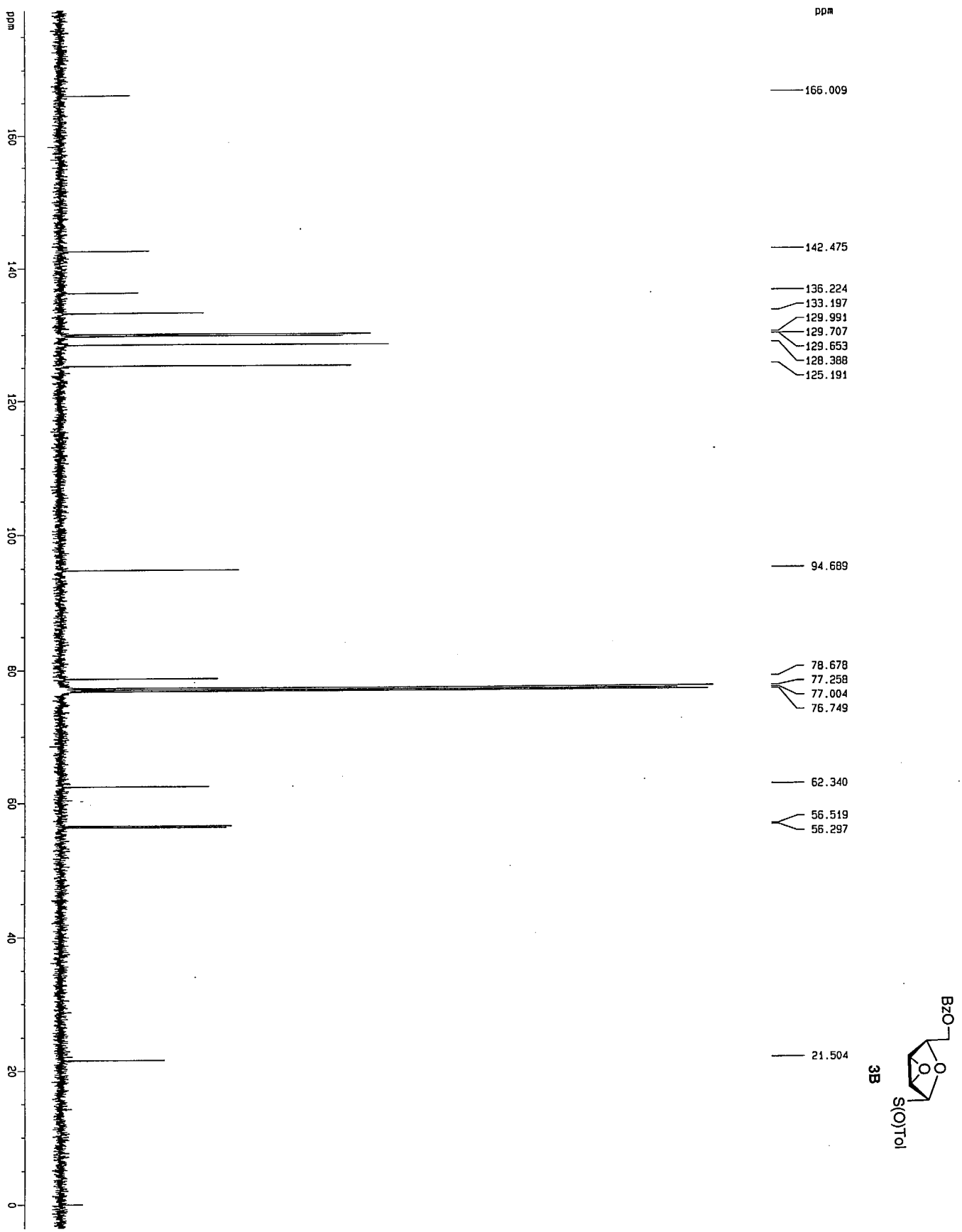

```
Current Data Parameters
NAME                  CSC-9-7-00
EXPNO                         20
PROCNO                         1

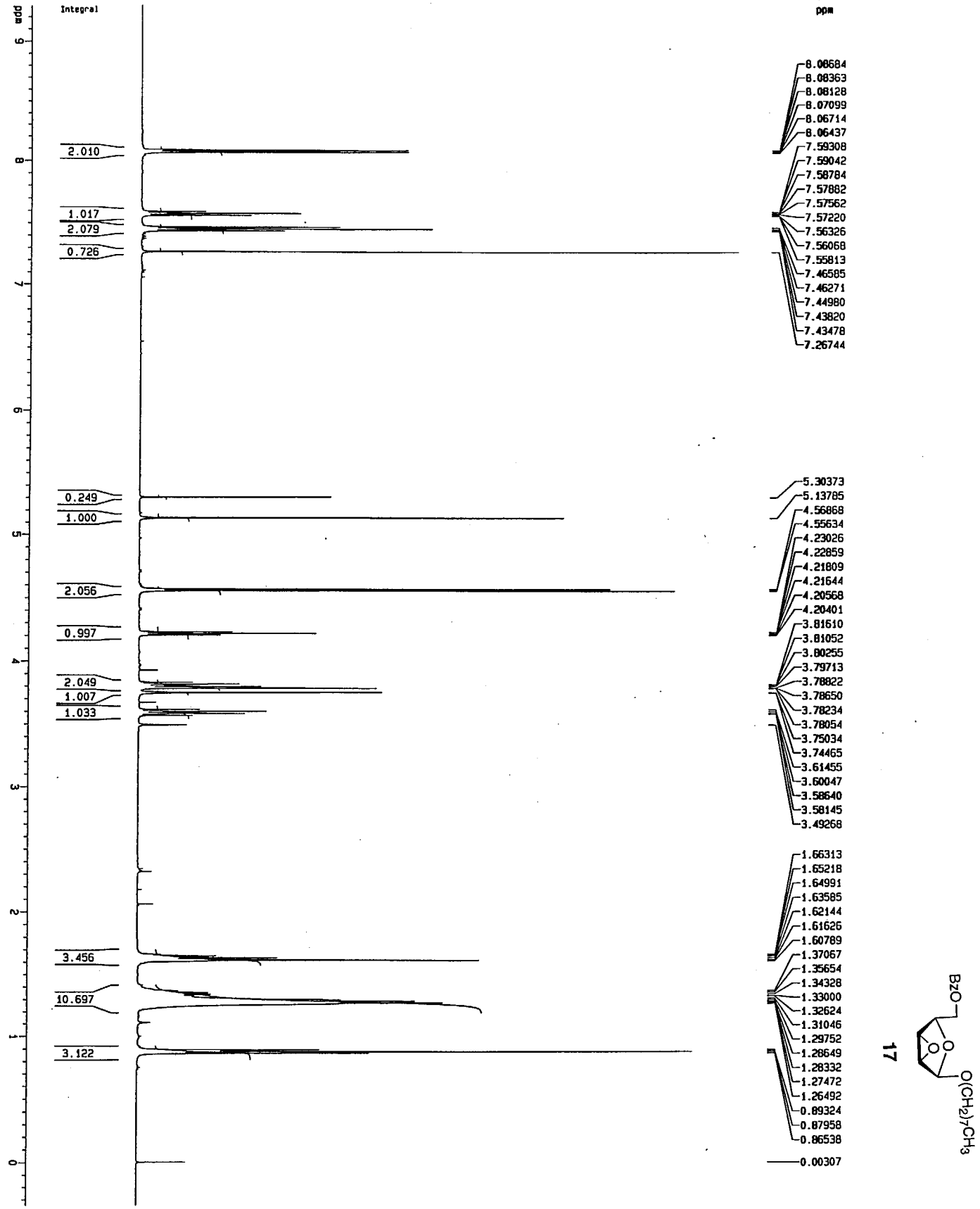
F2 - Acquisition Parameters
Date_                   20000907
Time                        9.36
INSTRUM                     spect
PROBHD     5 mm BBO BB-1
PULPROG                     zg30
TD                         65536
SOLVENT                    CDCl3
NS                             65
DS                              2
SWH                    10330.578 Hz
FIDRES                 0.157632 Hz
AQ                     3.1719923 sec
RG                            181
DW                         48.400 usec
DE                          6.00 usec
TE                         300.0 K
D1                     1.00000000 sec

========== CHANNEL f1 ==========
NUC1                          1H
P1                         13.70 usec
PL1                        -1.00 dB
SFO1                 500.1330885 MHz

F2 - Processing parameters
SI                         32768
SF                 500.1300000 MHz
WDW                           EM
SSB                            0
LB                          0.30 Hz
GB                             0
PC                          1.00

1D NMR plot parameters
CX                         34.00 cm
F1P                        10.372 ppm
F1                       5187.44 Hz
F2P                        -1.664 ppm
F2                       -832.14 Hz
PPMCM                   0.35400 ppm/cm
HZCM                  177.04617 Hz/cm
```


ppm
166.009
142.475
136.224
133.197
129.991
129.707
129.653
128.388
125.191
94.689
78.678
77.258
77.004
76.749
62.340
56.519
56.297
21.504
BzO
O
O
O
S(O)Tol
3B
Current Data Parameters
NAME CSC-9-7-00
EXPNO 21
PROCNO 1
F2 - Acquisition Parameters
Date_ 20000907
Time 9.42
INSTRUM spect
PROBHD 5 mm BBD BB-1
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 172
DS 4
SWH 31446.541 Hz
FIDRES 0.479836 Hz
AQ 1.0420724 sec
RG 8192
DW 15.900 usec
DE 6.00 usec
TE 300.0 K
D1 2.00000000 sec
D11 0.03000000 sec
D12 0.00002000 sec
CHANNEL f1
NUC1 13C
P1 8.00 usec
PL1 3.00 dB
SFO1 125.7715719 MHz
CHANNEL f2
CPDPRG2 waltz16
NUC2 1H
PCPD2 100.00 usec
PL2 -1.00 dB
PL12 18.90 dB
PL13 18.90 dB
SFO2 500.1320005 MHz
F2 - Processing parameters
SI 32768
SF 125.7577955 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
1D NMR plot parameters
CX 34.00 cm
F1P 178.878 ppm
F1 22495.31 Hz
F2P -3.529 ppm
F2 -443.79 Hz
PPMCM 5.36491 ppm/cm
HZCM 674.67963 Hz/cm



17

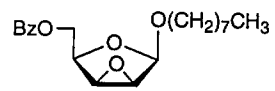
Current Data Parameters
NAME Couplefinal
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20000720
Time 11.04
INSTRUM spect
PROBHD 5 mm BBO BB-1
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 25
DS 2
SWH 10330.578 Hz
FIDRES 0.157632 Hz
AQ 3.1719923 sec
RG 181
DW 48.400 usec
DE 6.00 usec
TE 300.0 K
D1 1.00000000 sec

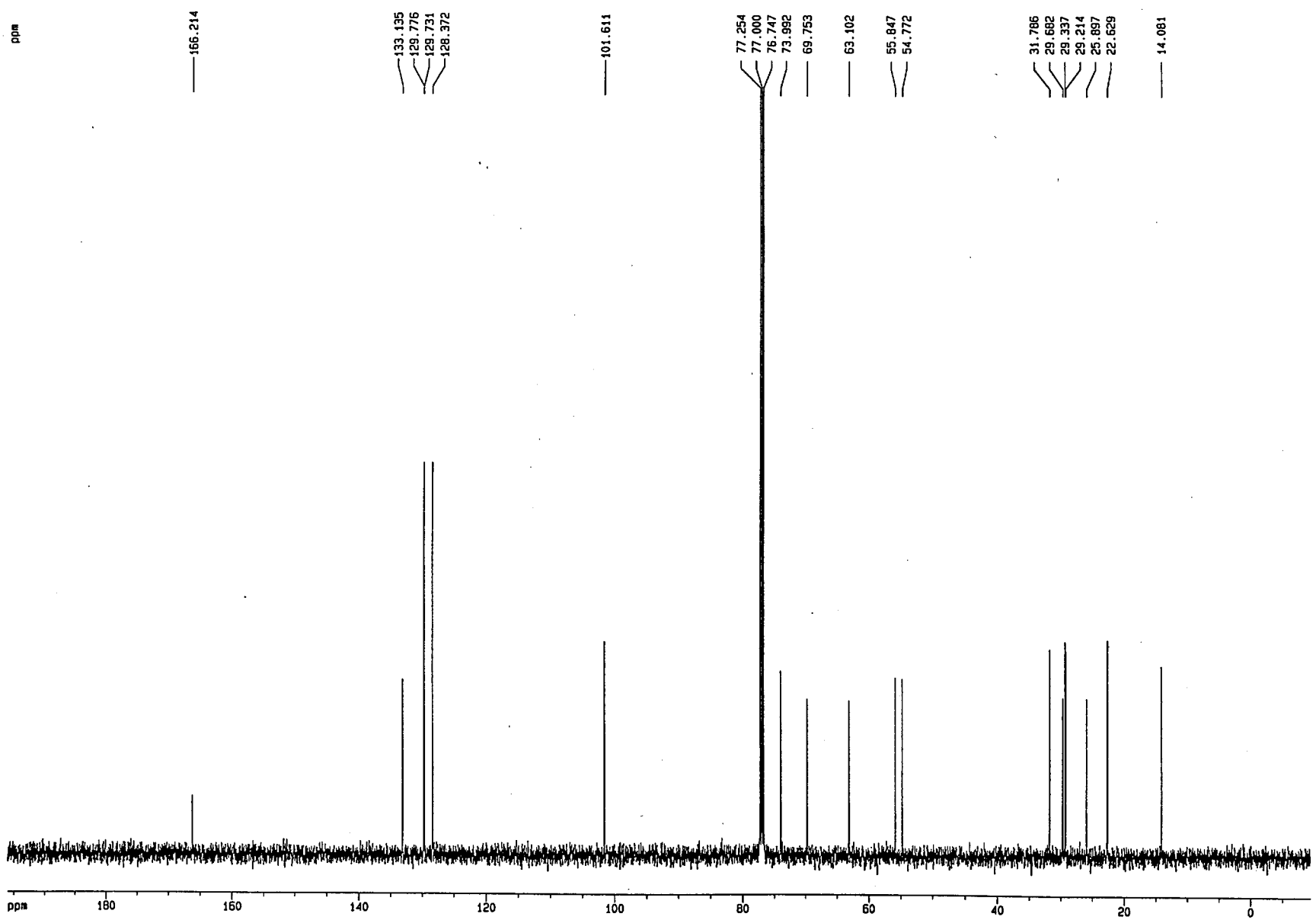
======== CHANNEL f1 ========
NUC1 1H
P1 13.70 usec
PL1 -1.00 dB
SFO1 500.1330885 MHz

F2 - Processing parameters
SI 32768
SF 500.1300200 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

1D NMR plot parameters
CX 34.00 cm
F1P 9.291 ppm
F1 4645.91 Hz
F2P -0.345 ppm
F2 -173.28 Hz
PPMCM 0.28347 ppm/cm
HZCM 141.77007 Hz/cm



17




```
Current Data Parameters
NAME          CoupleFinal
EXPNO                   2
PROCNO                  1

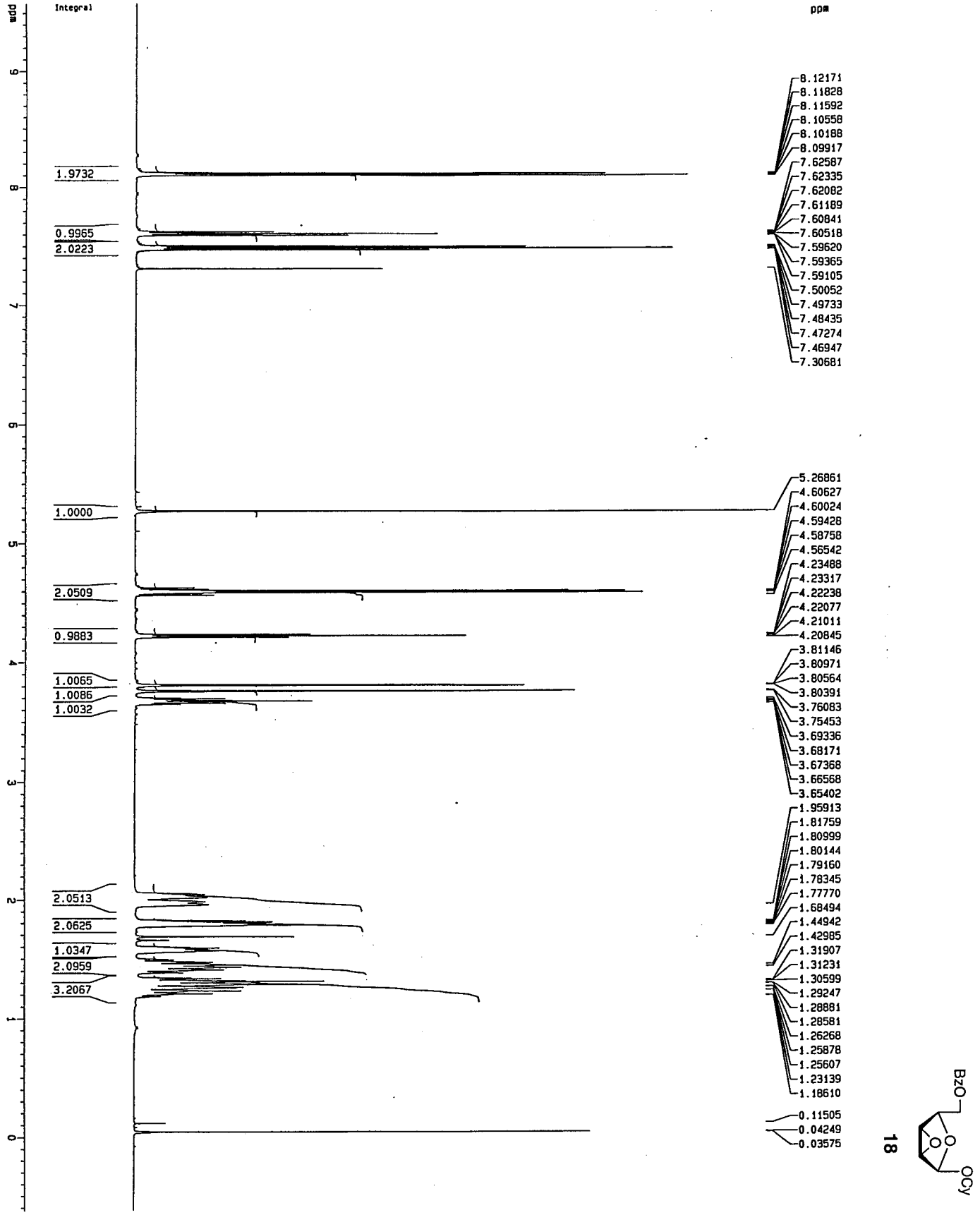
F2 - Acquisition Parameters
Date_          20000720
Time              11.08
INSTRUM           spect
PROBHD    5 mm BBO BB-1
PULPROG         zgpg30
TD                65536
SOLVENT           CDCl3
NS                  171
DS                    4
SWH           31446.541 Hz
FIDRES        0.479836 Hz
AQ            1.0420724 sec
RG                 4096
DW               15.900 usec
DE                6.00 usec
TE               300.0 K
D1            2.00000000 sec
D11           0.03000000 sec
D12           0.00002000 sec

============ CHANNEL f1 ============
NUC1               13C
P1                8.00 usec
PL1               3.00 dB
SFO1        125.7715719 MHz

============ CHANNEL f2 ============
CPDPRG2         waltz16
NUC2               1H
PCPD2           100.00 usec
PL2              -1.00 dB
PL12             18.80 dB
PL13             18.80 dB
SFO2        500.1320005 MHz

F2 - Processing parameters
SI                32768
SF          125.7577965 MHz
WDW                EM
SSB                0
LB                1.00 Hz
GB                 0
PC                1.40

1D NMR plot parameters
CX               34.00 cm
F1P             195.950 ppm
F1            24642.19 Hz
F2P             -9.464 ppm
F2            -1190.12 Hz
PPMCM          6.04157 ppm/cm
HZCM          759.77405 Hz/cm
```



18

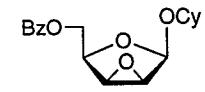
Current Data Parameters
NAME Coupling2
EXPNO 20
PROCNO 1

F2 - Acquisition Parameters
Date_ 20001019
Time 9.37
INSTRUM spect
PROBHD 5 mm BBO BB-1
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 17
DS 2
SWH 10330.578 Hz
FIDRES 0.157632 Hz
AQ 3.1719923 sec
RG 161.3
DW 48.400 usec
DE 6.00 usec
TE 300.0 K
D1 1.00000000 sec

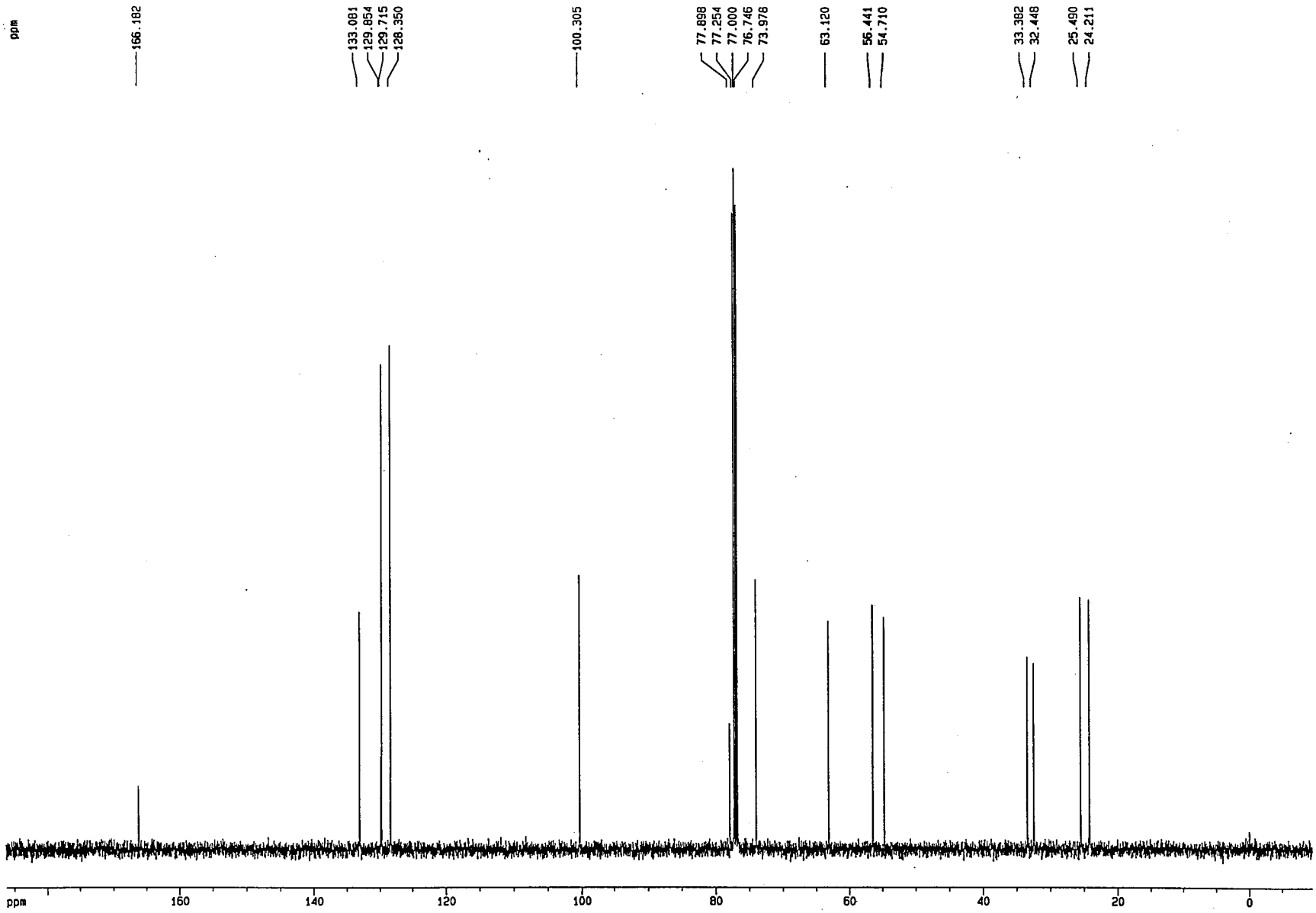
========== CHANNEL f1 ==========
NUC1 1H
P1 13.70 usec
PL1 -1.00 dB
SFO1 500.1330885 MHz

F2 - Processing parameters
SI 32768
SF 500.1300000 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

1D NMR plot parameters
CX 34.00 cm
F1P 9.580 ppm
F1 4791.41 Hz
F2P -0.623 ppm
F2 -311.65 Hz
PPMCM 0.30010 ppm/cm
HZCM 150.08989 Hz/cm



18




```
Current Data Parameters
NAME            Coupling2
EXPNO                  21
PROCNO                  1

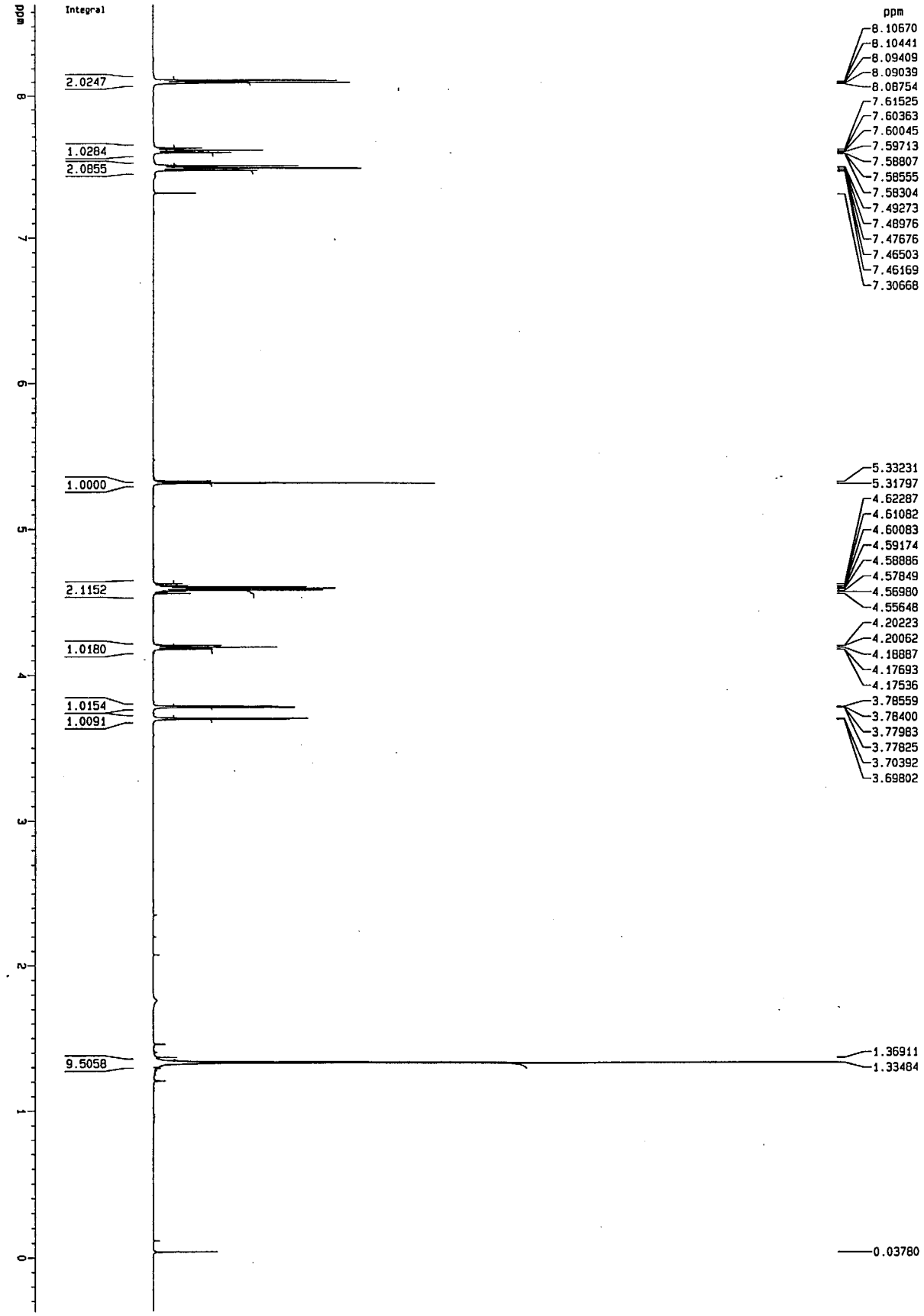
F2 - Acquisition Parameters
Date_          20001019
Time               9.40
INSTRUM           spect
PROBHD    5 mm BBO BB-1
PULPROG         zgpg30
TD               65536
SOLVENT          CDCl3
NS                  93
DS                   4
SWH          31446.541 Hz
FIDRES        0.479836 Hz
AQ            1.0420724 sec
RG               9195.2
DW              15.900 usec
DE               6.00 usec
TE              300.0 K
D1           2.00000000 sec
D11          0.03000000 sec
D12          0.00002000 sec

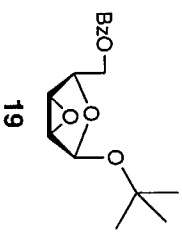
------------ CHANNEL f1 ------------
NUC1                13C
P1                 8.00 usec
PL1                3.00 dB
SFO1        125.7715719 MHz

------------ CHANNEL f2 ------------
CPDPRG2         waltz16
NUC2                1H
PCPD2          100.00 usec
PL2              -1.00 dB
PL12              18.80 dB
PL13              18.80 dB
SFO2        500.1320005 MHz

F2 - Processing parameters
SI                32768
SF          125.7577965 MHz
WDW                EM
SSB                 0
LB                1.00 Hz
GB                  0
PC                1.40

1D NMR plot parameters
CX                34.00 cm
F1P             186.273 ppm
F1            23425.28 Hz
F2P              -9.554 ppm
F2            -1201.54 Hz
PPMCM          5.75963 ppm/cm
HZCM         724.31824 Hz/cm
```





19

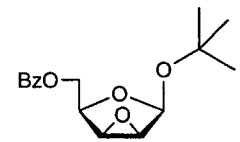
Current Data Parameters
NAME FinalEPOX
EXPNO 30
PROCNO 1

F2 - Acquisition Parameters
Date_ 20001115
Time 10.07
INSTRUM spect
PROBHD 5 mm BBO BB-1
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 2
DS 2
SWH 10330.578 Hz
FIDRES 0.157632 Hz
AQ 3.1719823 sec
RG 90.5
DW 48.400 usec
DE 6.00 usec
TE 300.0 K
D1 1.00000000 sec

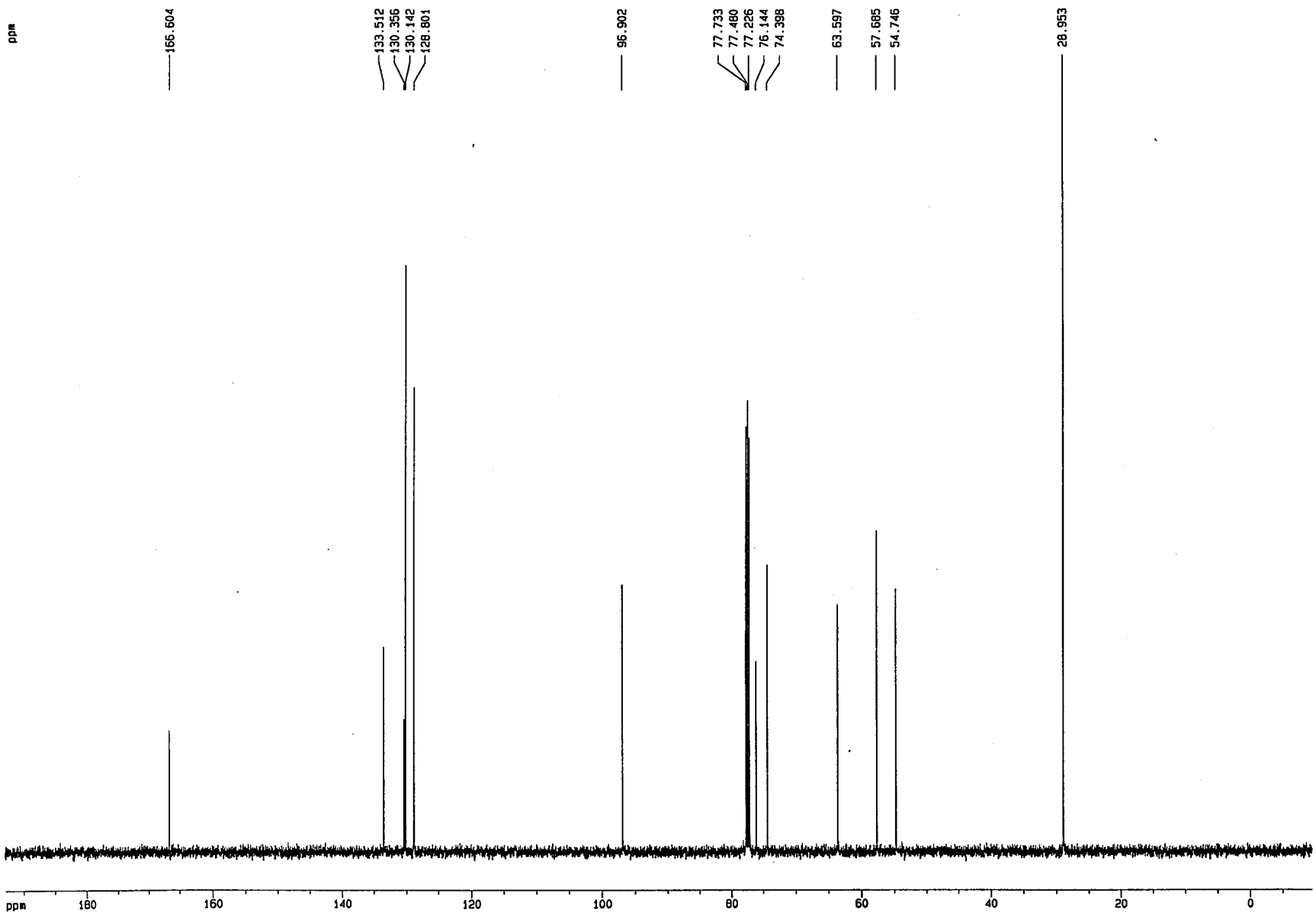
========== CHANNEL f1 ==========
NUC1 1H
P1 13.70 usec
PL1 -1.00 dB
SFO1 500.1330885 MHz

F2 - Processing parameters
SI 32768
SF 500.1300000 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

1D NMR plot parameters
CX 34.00 cm
F1P 8.653 ppm
F1 4327.50 Hz
F2P -0.374 ppm
F2 -187.18 Hz
PPMCM 0.25550 ppm/cm
HZCM 132.78464 Hz/cm



19




```
Current Data Parameters
NAME            FinalEPOX
EXPNO                  31
PROCNO                  1

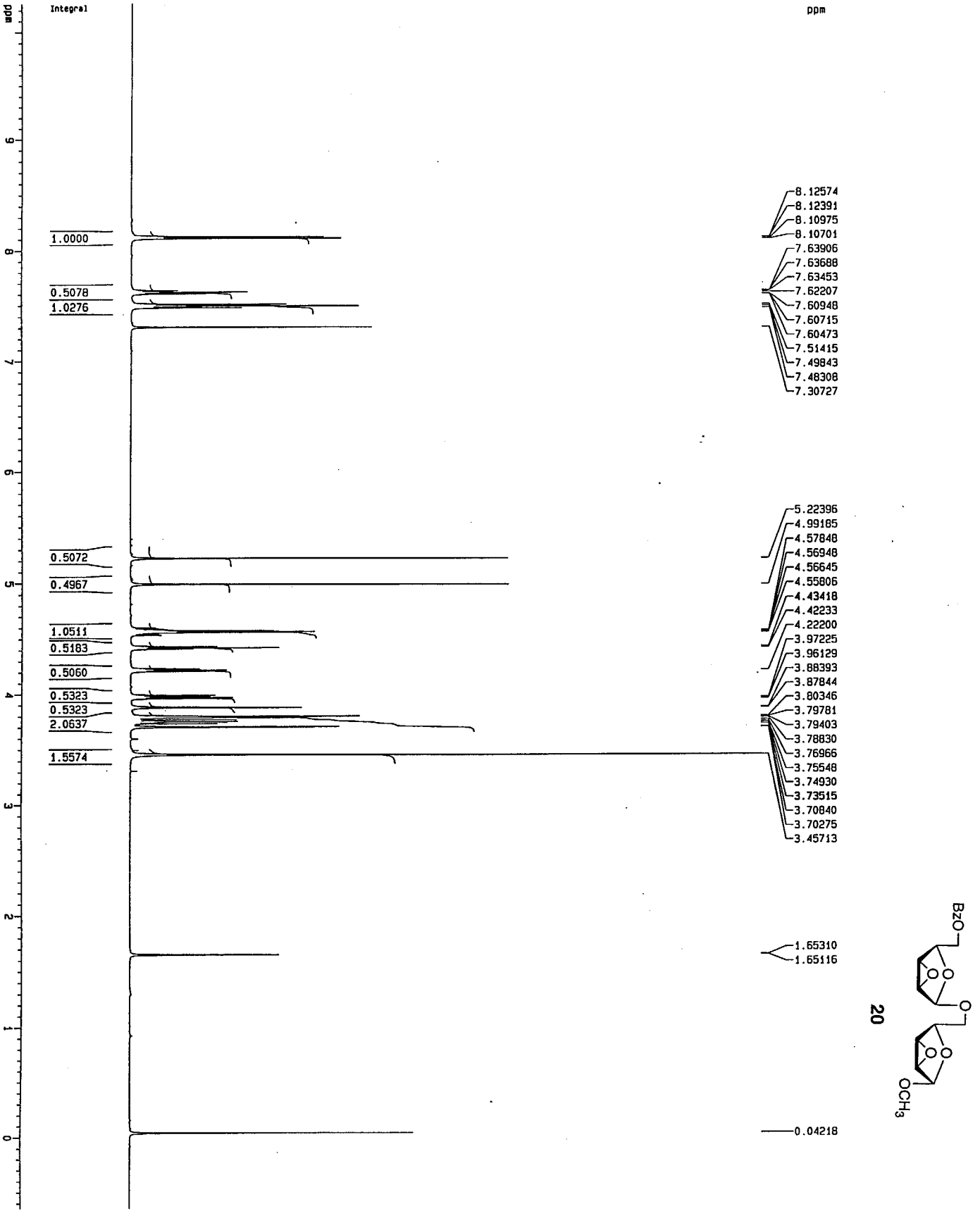
F2 - Acquisition Parameters
Date_          20001115
Time              10.15
INSTRUM           spect
PROBHD     5 mm BBO BB-1
PULPROG         zgpg30
TD               65536
SOLVENT          CDCl3
NS                  76
DS                   4
SWH           31446.541 Hz
FIDRES        0.479836 Hz
AQ            1.0420724 sec
RG                8192
DW              15.900 usec
DE               6.00 usec
TE              300.0 K
D1            2.00000000 sec
D11           0.03000000 sec
D12           0.00002000 sec

============ CHANNEL f1 ===========
NUC1               13C
P1                8.00 usec
PL1               3.00 dB
SF01        125.7715719 MHz

============ CHANNEL f2 ===========
CPDPRG2         waltz16
NUC2               1H
PCPD2           100.00 usec
PL2              -1.00 dB
PL12             18.80 dB
PL13             18.80 dB
SF02        500.1320005 MHz

F2 - Processing parameters
SI                32768
SF          125.7577390 MHz
WDW                EM
SSB                 0
LB               1.00 Hz
GB                  0
PC               1.40

1D NMR plot parameters
CX               34.00 cm
F1P             192.846 ppm
F1             24251.90 Hz
F2P              -9.554 ppm
F2             -1201.54 Hz
PPMCM            5.95296 ppm/cm
HZCM           748.63068 Hz/cm
```



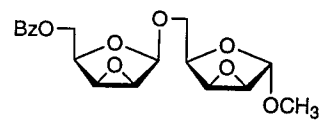
Current Data Parameters
NAME C13CDPLE
EXPNO 20
PROCNO 1

F2 - Acquisition Parameters
Date_ 20000717
Time 11.28
INSTRUM spect
PROBHD 5 mm BBO BB-1
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 28
DS 2
SWH 10330.578 Hz
FIDRES 0.157632 Hz
AQ 3.1719923 sec
RG 203.2
DW 48.400 usec
DE 6.00 usec
TE 300.0 K
D1 1.00000000 sec

========== CHANNEL f1 ==========
NUC1 1H
P1 13.70 usec
PL1 -1.00 dB
SFO1 500.1330885 MHz

F2 - Processing parameters
SI 32768
SF 500.1300000 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

1D NMR plot parameters
CX 34.00 cm
F1P 10.259 ppm
F1 5130.86 Hz
F2P -0.646 ppm
F2 -322.96 Hz
PPMCM 0.32073 ppm/cm
HZCM 160.40651 Hz/cm



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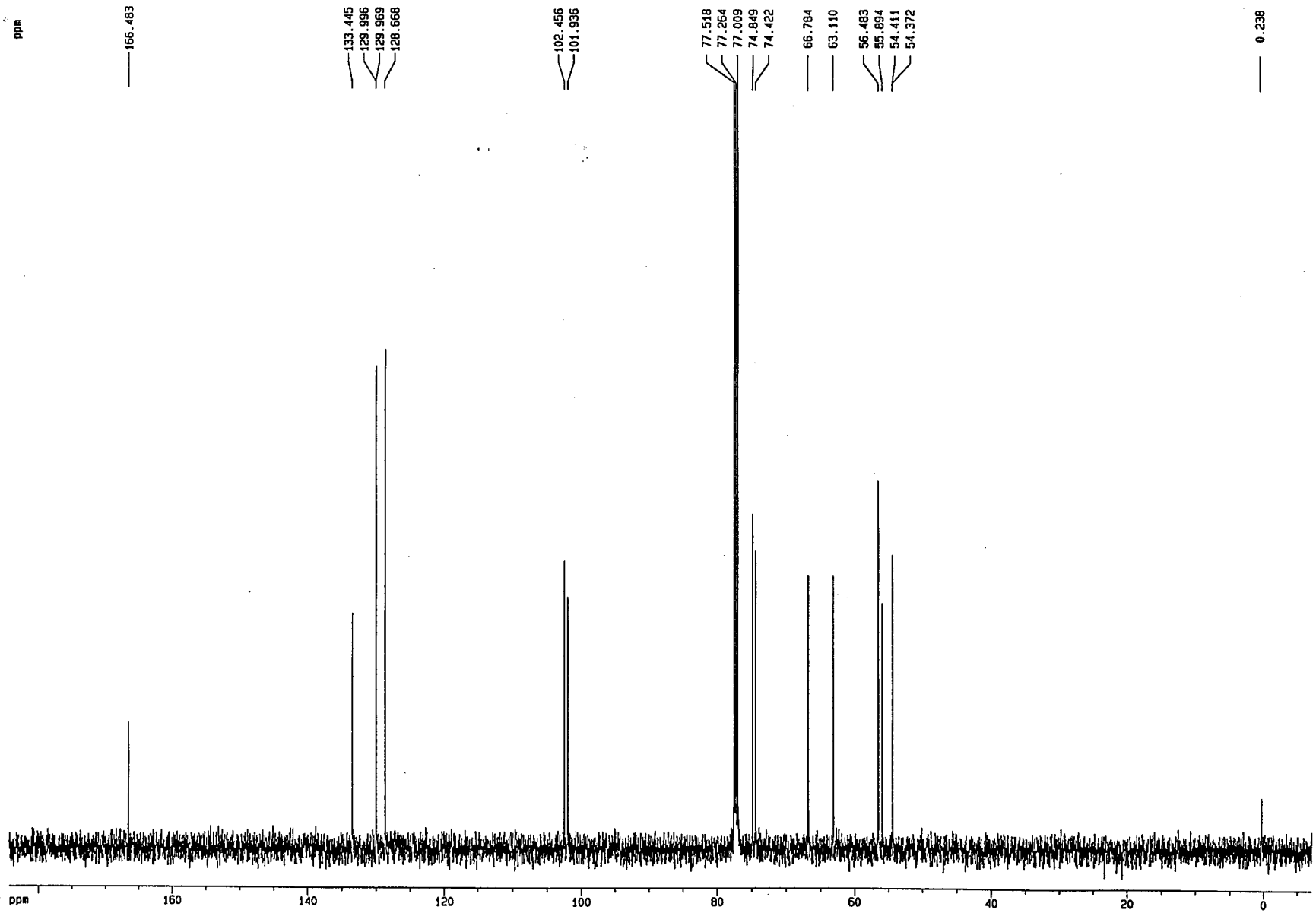



```
Current Data Parameters
NAME            C13COUPLE
EXPNO                  21
PROCNO                  1

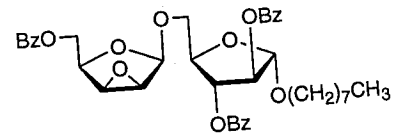
F2 - Acquisition Parameters
Date_            20000717
Time                11.33
INSTRUM             spect
PROBHD    5 mm BBO BB-1
PULPROG           zgpg30
TD                 65536
SOLVENT            CDCl3
NS                   181
DS                     4
SWH            31446.541 Hz
FIDRES          0.479836 Hz
AQ              1.0420724 sec
RG                 13004
DW                15.900 usec
DE                 6.00 usec
TE                300.0 K
D1             2.00000000 sec
D11            0.03000000 sec
D12            0.00002000 sec

============ CHANNEL f1 ============
NUC1                13C
P1                 8.40 usec
PL1                3.00 dB
SFO1         125.7715719 MHz

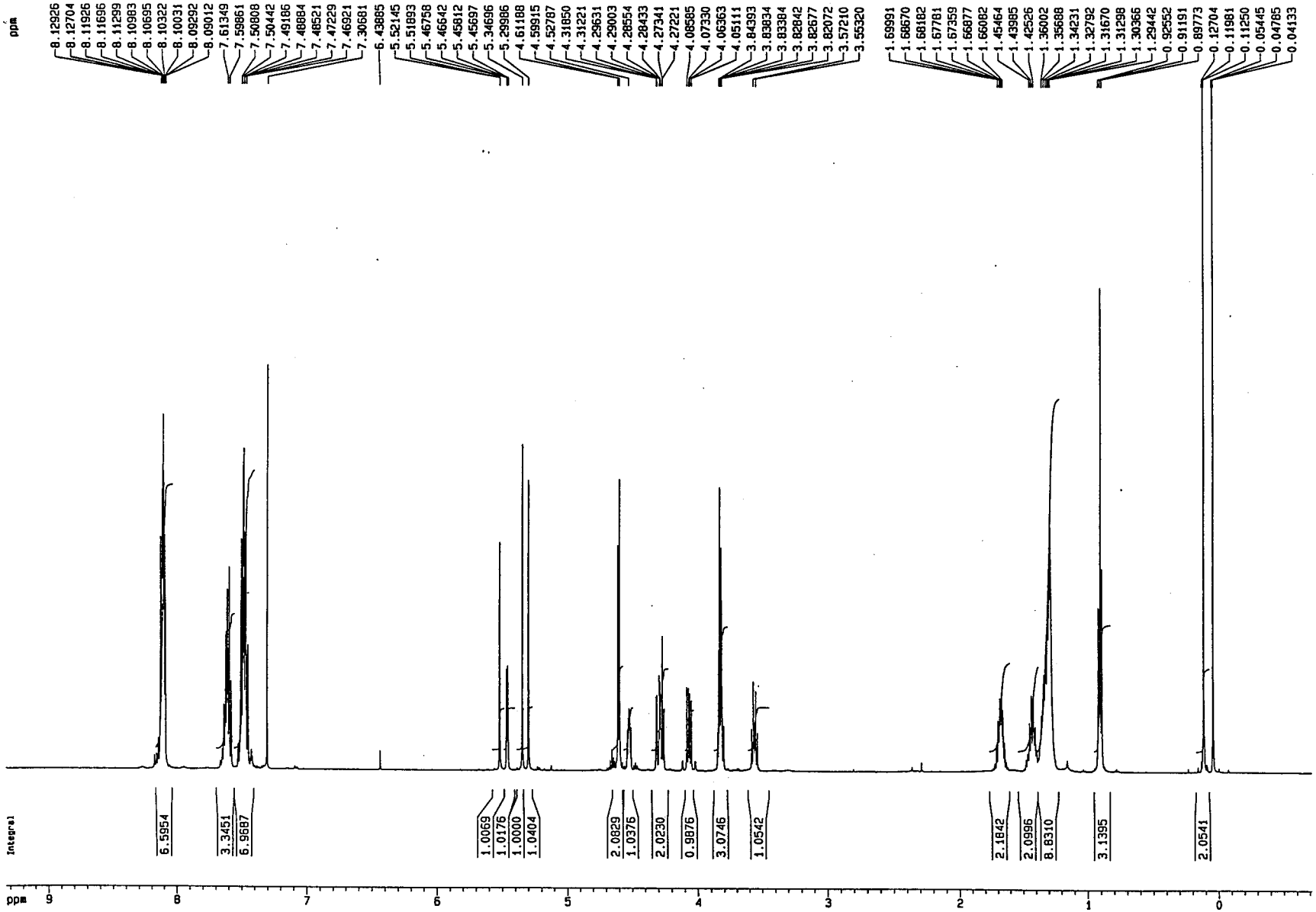
============ CHANNEL f2 ============
CPDPRG2         waltz16
NUC2                1H
PCPD2            100.00 usec
PL2               -1.00 dB
PL12              18.80 dB
PL13              18.80 dB
SFO2         500.1320005 MHz

F2 - Processing parameters
SI                 32768
SF           125.7577638 MHz
WDW                   EM
SSB                    0
LB                  1.00 Hz
GB                     0
PC                  1.40

1D NMR plot parameters
CX                 34.00 cm
F1P              184.367 ppm
F1             23185.64 Hz
F2P               -7.100 ppm
F2              -892.82 Hz
PPMCM            5.63138 ppm/cm
HZCM           708.19006 Hz/cm
```



21



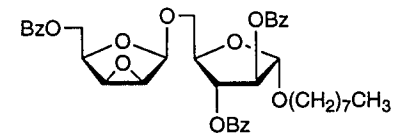
Current Data Parameters
NAME CompleFinal
EXPNO 190
PROCNO 1

F2 - Acquisition Parameters
Date_ 20001104
Time 15.44
INSTRUM spect
PROBHD 5 mm BBO BB-1
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 54
DS 2
SWH 10330.578 Hz
FIDRES 0.157632 Hz
AQ 3.1719923 sec
RG 256
DW 48.400 usec
DE 6.00 usec
TE 300.0 K
D1 1.00000000 sec

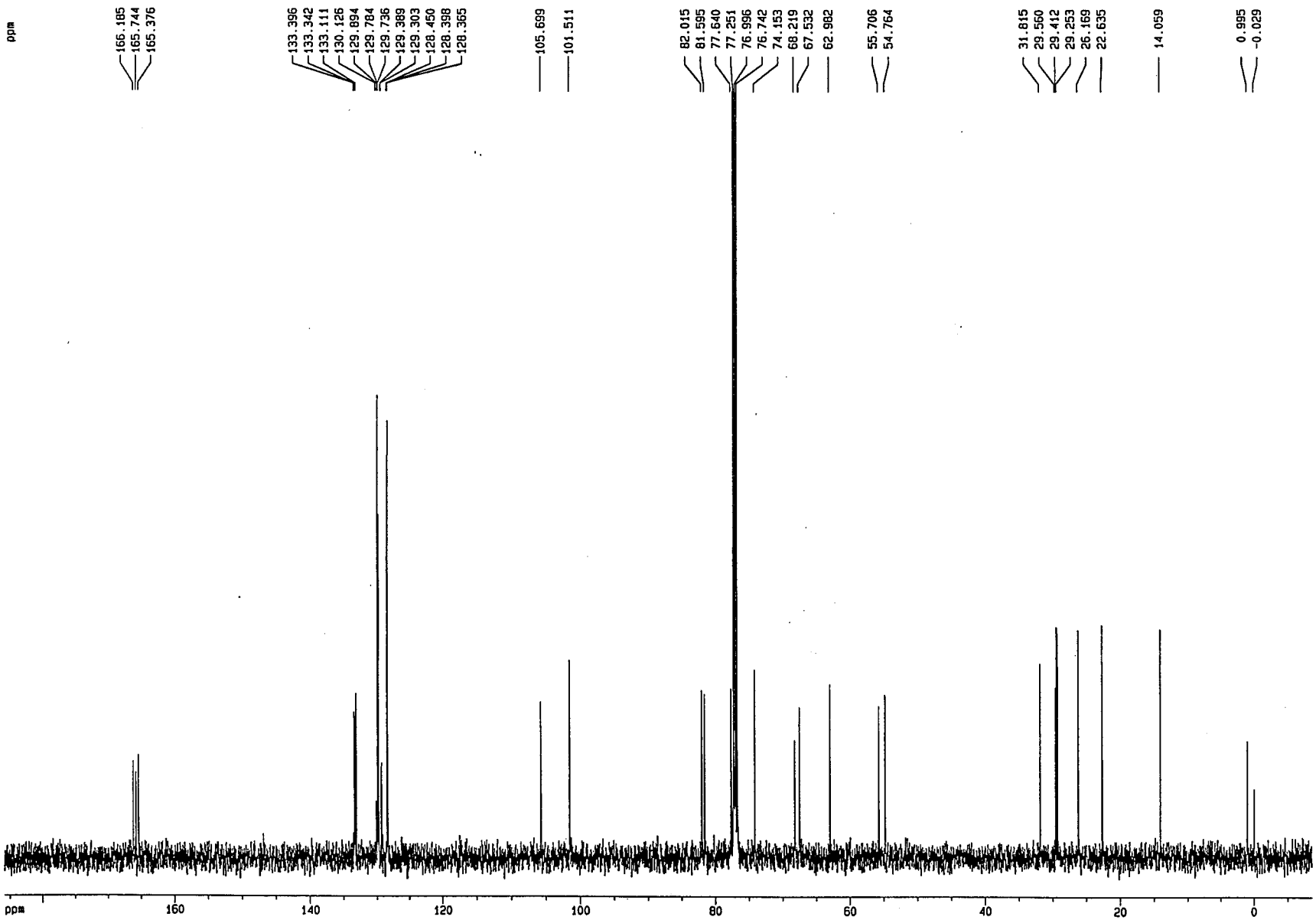
---------- CHANNEL f1 ----------
NUC1 1H
P1 13.70 usec
PL1 -1.00 dB
SFO1 500.1330885 MHz

F2 - Processing parameters
SI 32768
SF 500.1300000 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

1D NMR plot parameters
CX 34.00 cm
F1P 9.331 ppm
F1 4666.95 Hz
F2P -0.714 ppm
F2 -356.91 Hz
PPMCM 0.29544 ppm/cm
HZCM 147.76033 Hz/cm



21




```
Current Data Parameters
NAME          CoupleFinal
EXPNO                 191
PROCNO                  1

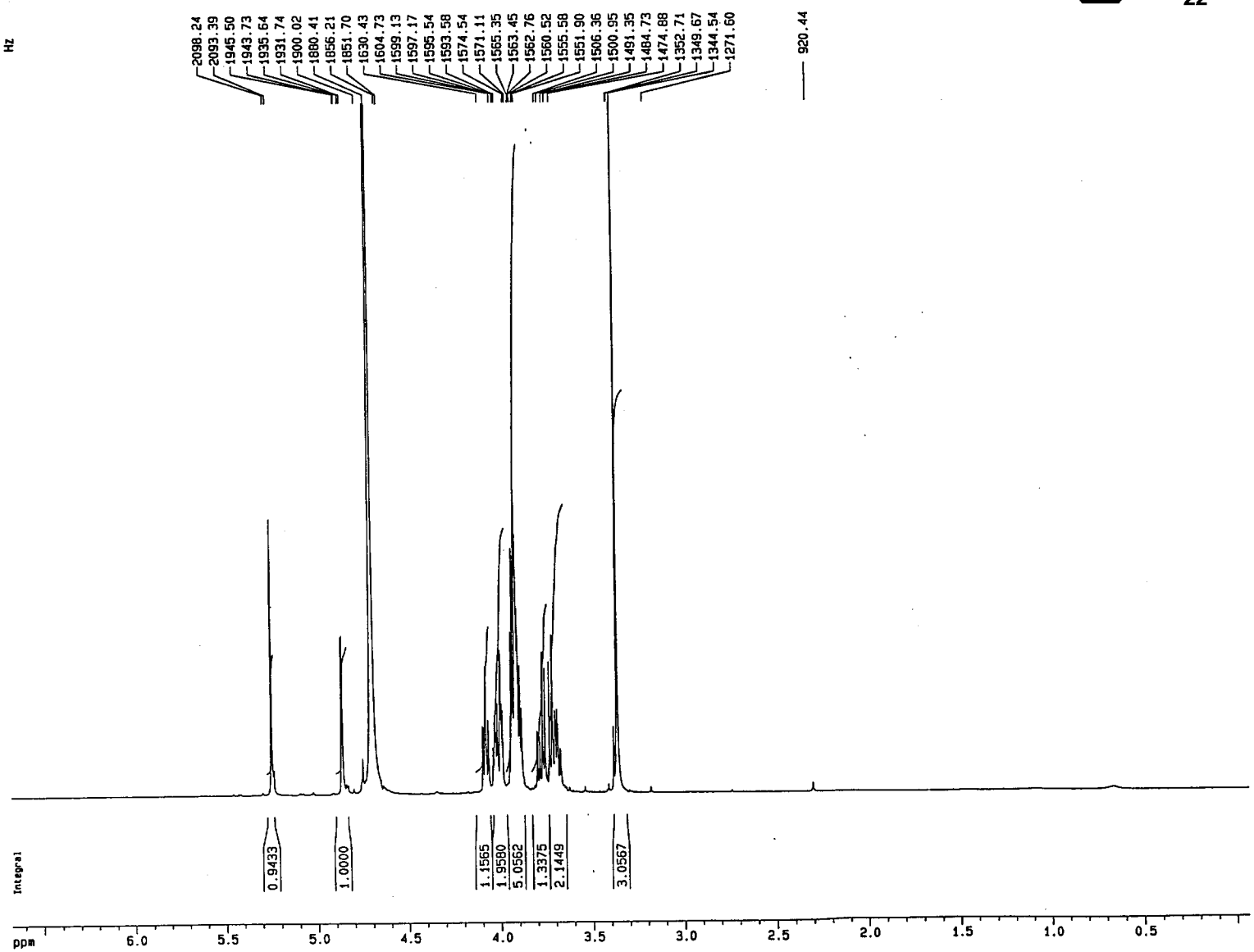
F2 - Acquisition Parameters
Date_           20001104
Time               15.47
INSTRUM             spect
PROBHD   5 mm BBO BB-1
PULPROG           zgpg30
TD                 65536
SOLVENT            CDCl3
NS                   469
DS                     4
SWH           31446.541 Hz
FIDRES        0.479836 Hz
AQ            1.0420724 sec
RG               10321.3
DW               15.900 usec
DE                6.00 usec
TE               300.0 K
D1            2.00000000 sec
D11           0.03000000 sec
D12           0.00002000 sec

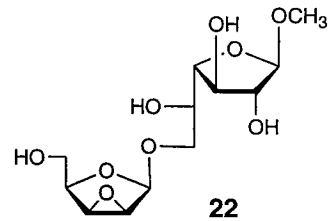
========== CHANNEL f1 ==========
NUC1                13C
P1                7.50 usec
PL1               3.00 dB
SFO1        125.7715719 MHz

========== CHANNEL f2 ==========
CPDPRG2         waltz16
NUC2                1H
PCPD2           100.00 usec
PL2              -1.00 dB
PL12             18.80 dB
PL13             18.80 dB
SFO2        500.1320005 MHz

F2 - Processing parameters
SI                 32768
SF          125.7577946 MHz
WDW                   EM
SSB                    0
LB                1.00 Hz
GB                     0
PC                  1.40

1D NMR plot parameters
CX                34.00 cm
F1P             185.831 ppm
F1              23369.71 Hz
F2P              -8.627 ppm
F2              -1084.89 Hz
PPMCM            5.71935 ppm/cm
HZCM           719.25275 Hz/cm
```





22

Current Data Parameters

NAME	May20-2000-rgad
EXPNO	20
PROCNO	1

F2 - Acquisition Parameters

Date_	20000520
Time	14.56
INSTRUM	spect
PROBHD	5 mm BBO BB-1
PULPROG	zg30
TD	32768
SOLVENT	D20
NS	16
DS	2
SWH	8278.146 Hz
FIDRES	0.252629 Hz
AQ	1.9792372 sec
RG	90.5
DW	60.400 usec
DE	6.00 usec
TE	300.0 K
D1	1.00000000 sec

========== CHANNEL f1 ==========

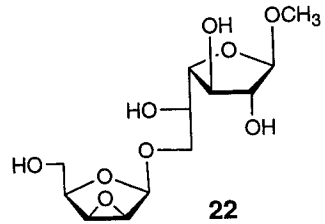
NUC1	1H
P1	7.50 usec
PL1	-6.00 dB
SFO1	400.1324710 MHz

F2 - Processing parameters

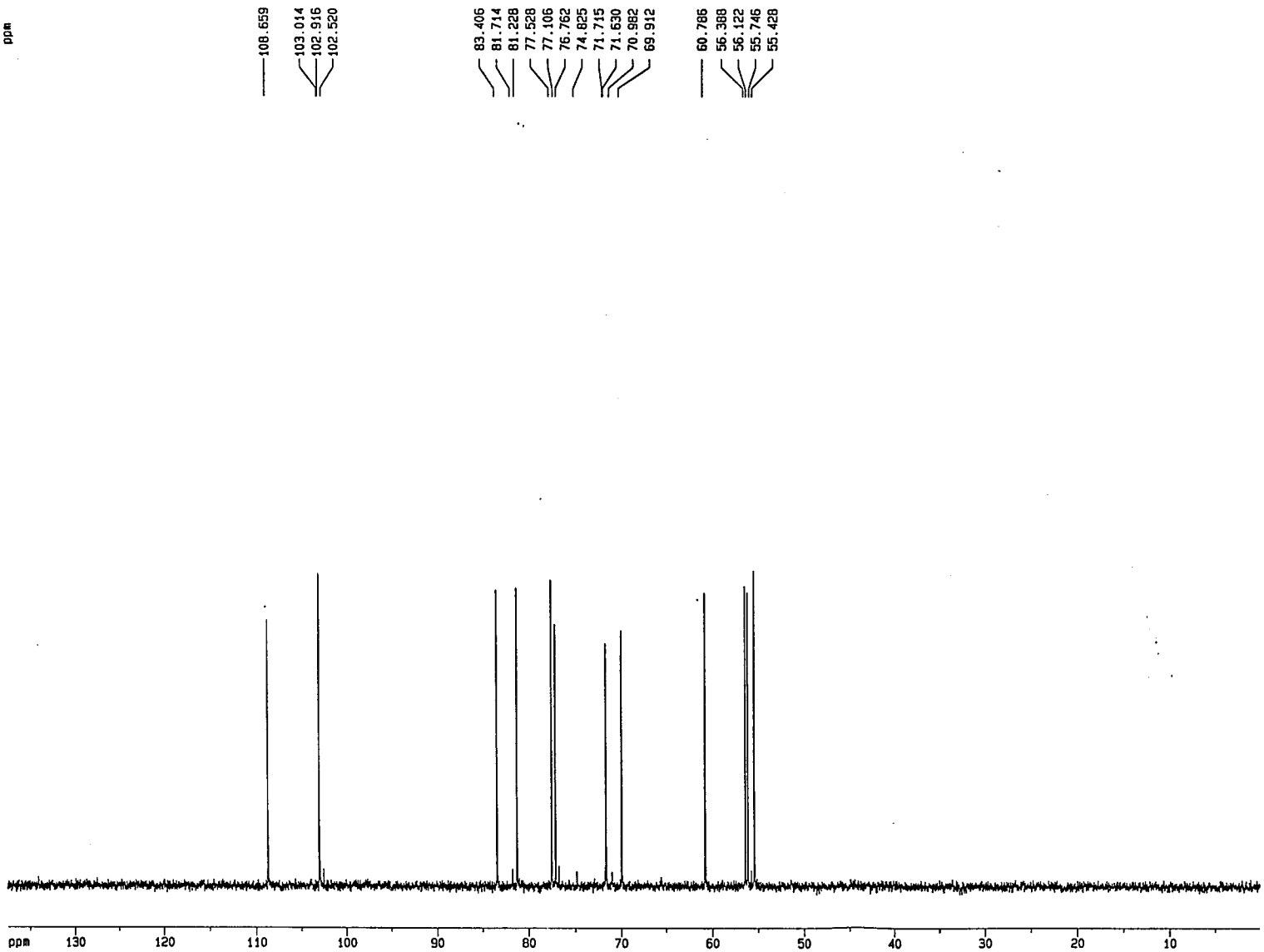
SI	32768
SF	400.1300000 MHz
WDW	EM
SSB	0
LB	0.30 Hz
GB	0
PC	1.00

1D NMR plot parameters

CX	30.00 cm
F1P	6.674 ppm
F1	2670.35 Hz
F2P	-0.067 ppm
F2	-26.81 Hz
PPMCM	0.22469 ppm/cm
HZCM	89.90549 Hz/cm



22




```
Current Data Parameters
NAME      May20-2000-rgad
EXPNO                 21
PROCNO                 1

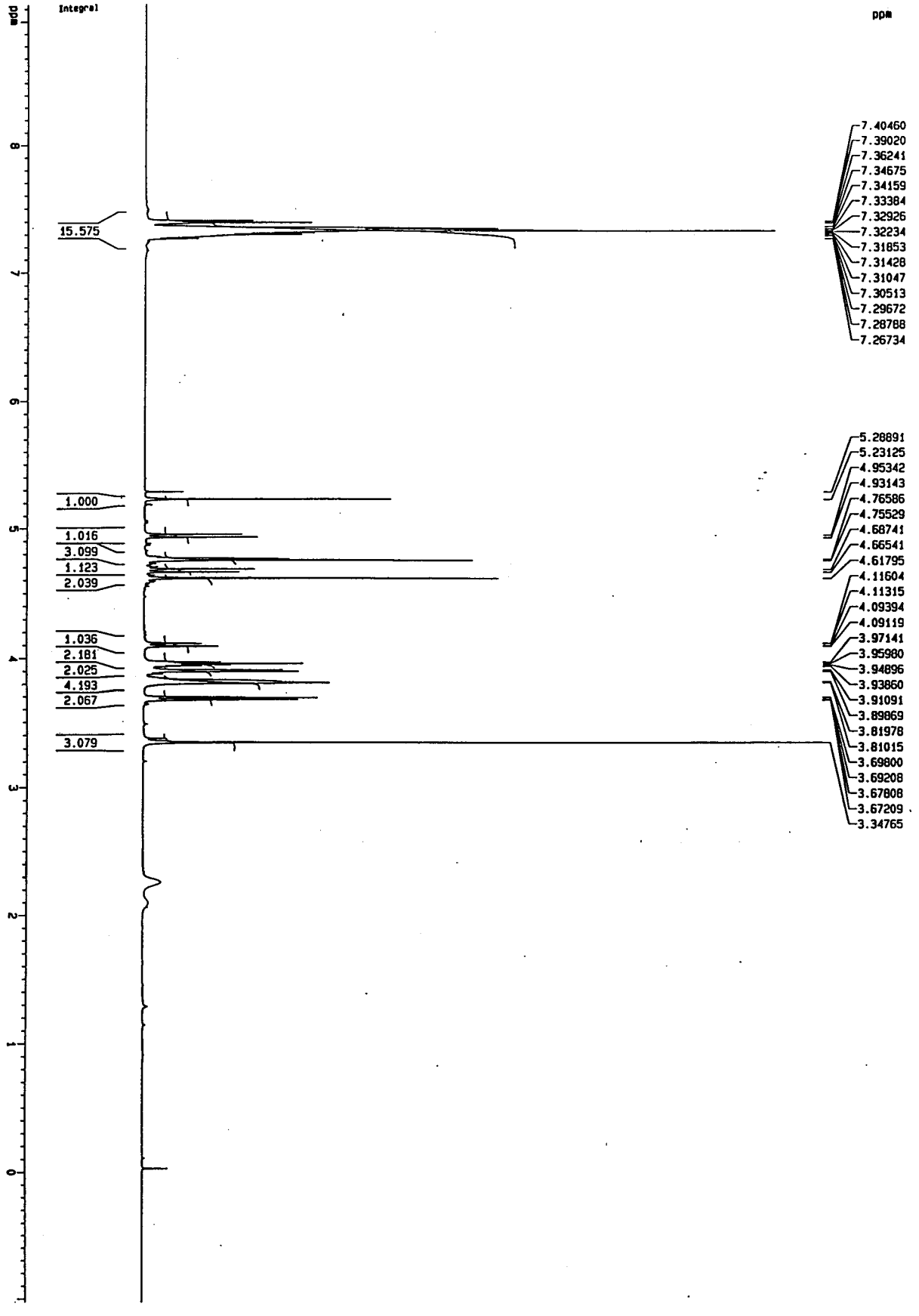
F2 - Acquisition Parameters
Date_           20000520
Time               15.01
INSTRUM            spect
PROBHD     5 mm BBO BB-1
PULPROG          zgpg30
TD                65536
SOLVENT             D2O
NS                  974
DS                    4
SWH           25125.629 Hz
FIDRES        0.383387 Hz
AQ            1.3042164 sec
RG                 4096
DW               19.900 usec
DE                6.00 usec
TE               300.0 K
D1            2.00000000 sec
d11           0.03000000 sec
d12           0.00002000 sec

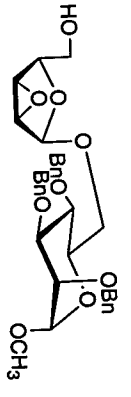
========== CHANNEL f1 ==========
NUC1                13C
P1                 5.90 usec
PL1               -6.00 dB
SF01        100.6237959 MHz

========== CHANNEL f2 ==========
CPDPRG2         waltz16
NUC2                1H
PCPD2             80.00 usec
PL2               -6.00 dB
PL12              15.80 dB
PL13              15.80 dB
SF02        400.1316005 MHz

F2 - Processing parameters
SI                 32768
SF          100.6127176 MHz
WDW                  EM
SSB                  0
LB                1.00 Hz
GB                   0
PC                1.40

1D NMR plot parameters
CX                30.00 cm
F1P              137.506 ppm
F1             13834.86 Hz
F2P               0.054 ppm
F2                5.39 Hz
PPMCM          4.58175 ppm/cm
HZCM         460.98221 Hz/cm
```





23

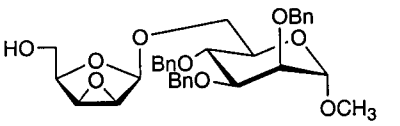
```
Current Data Parameters
NAME                  Coupling1
EXPNO                       60
PROCNO                       1

F2 - Acquisition Parameters
Date_              20000722
Time               9.04
INSTRUM            spect
PROBHD     5 mm BBO BB-1
PULPROG            zg30
TD                 65536
SOLVENT            CDCl3
NS                 32
DS                 2
SWH          10330.578 Hz
FIDRES        0.157632 Hz
AQ            3.1719823 sec
RG                 40.3
DW            48.400 usec
DE             6.00 usec
TE               300.0 K
D1           1.00000000 sec

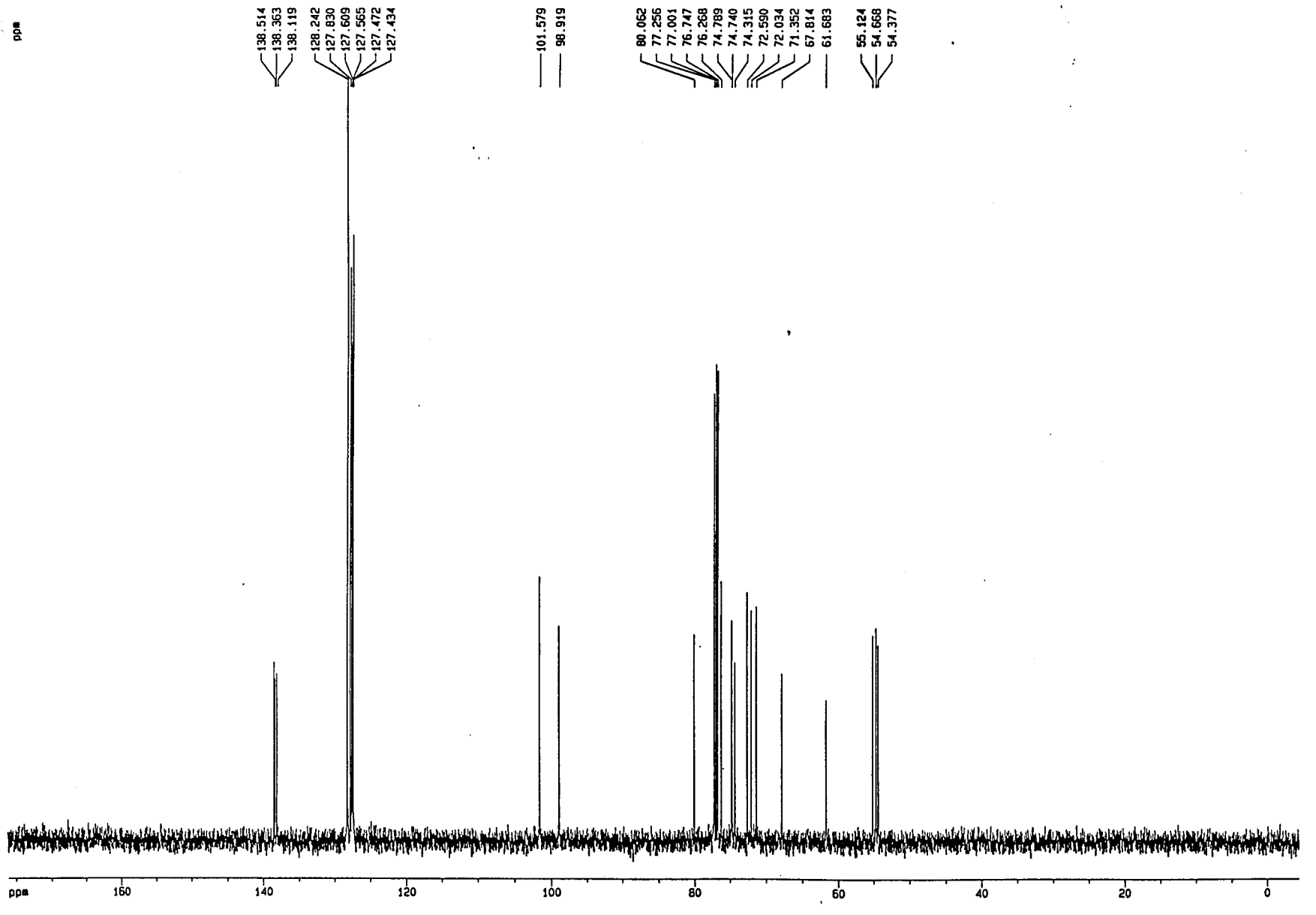
======== CHANNEL f1 ========
NUC1               1H
P1                13.70 usec
PL1               -1.00 dB
SFO1         500.1330885 MHz

F2 - Processing parameters
SI                32768
SF           500.1300200 MHz
WDW                EM
SSB                 0
LB               0.30 Hz
GB                  0
PC                1.00

1D NMR plot parameters
CX                34.00 cm
F1P               9.133 ppm
F1                4567.70 Hz
F2P              -1.025 ppm
F2             -512.73 Hz
PPMCM          0.26877 ppm/cm
HZCM         149.42430 Hz/cm
```



23




```
Current Data Parameters
NAME          CoupleFinal
EXPNO                  61
PROCNO                  1

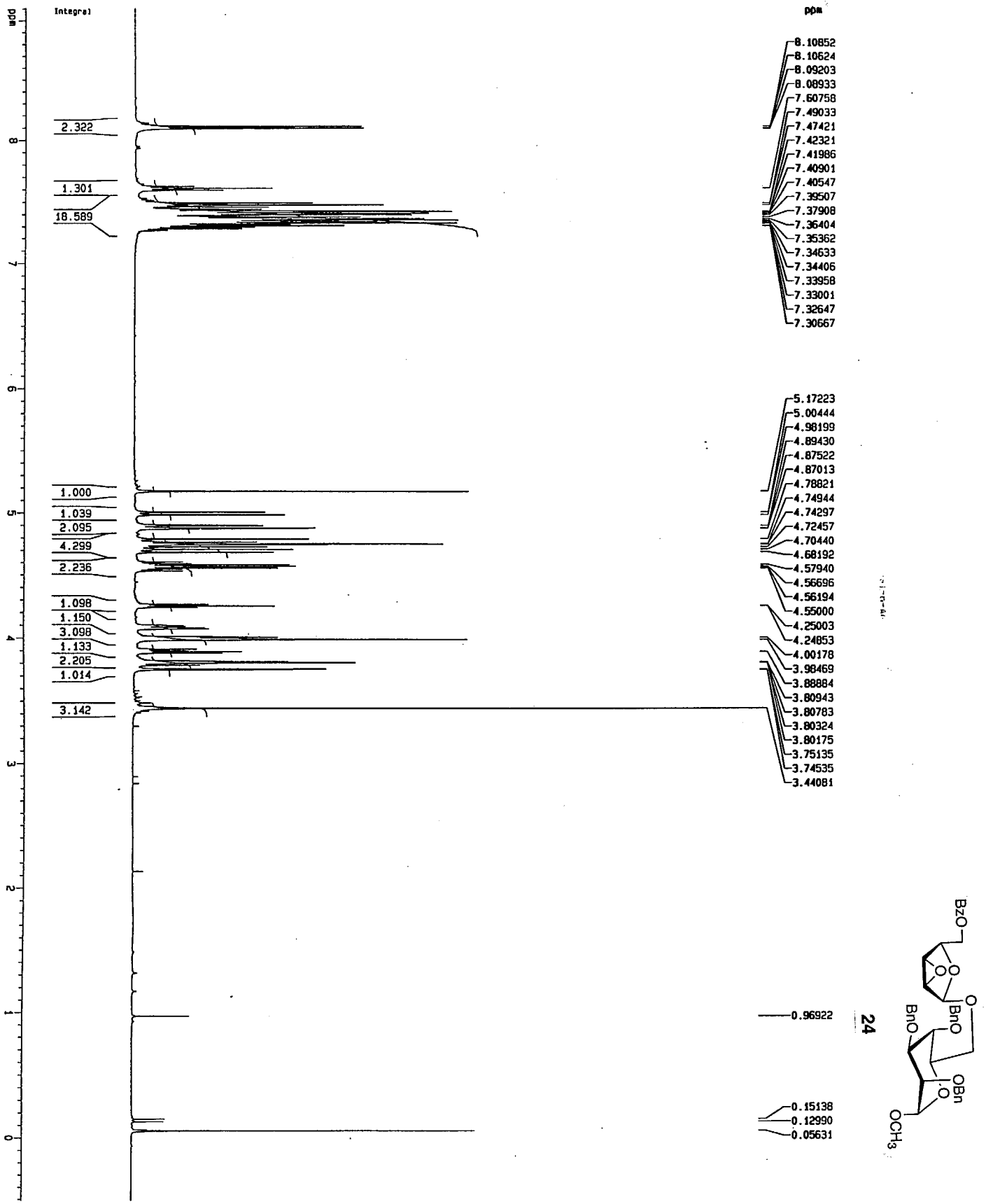
F2 - Acquisition Parameters
Date_           20000722
Time                9.07
INSTRUM            spect
PROBHD    5 mm BBO BB-1
PULPROG          zgpg30
TD                65536
SOLVENT           CDCl3
NS                   23
DS                    4
SWH           31446.541 Hz
FIDRES        0.479836 Hz
AQ            1.0420724 sec
RG                 8502
DW               15.900 usec
DE                6.00 usec
TE               300.0 K
D1            2.00000000 sec
D11           0.03000000 sec
D12           0.00002000 sec

============ CHANNEL f1 ============
NUC1                13C
P1                8.00 usec
PL1               3.00 dB
SF01        125.7715719 MHz

============ CHANNEL f2 ============
CPDPRG2         waltz16
NUC2                1H
PCPD2          100.00 usec
PL2              -1.00 dB
PL12             18.80 dB
PL13             18.80 dB
SF02        500.1320005 MHz

F2 - Processing parameters
SI                 32768
SF          125.7578109 MHz
WDW                   EM
SSB                    0
LB                1.00 Hz
GB                     0
PC                1.40

1D NMR plot parameters
CX                34.00 cm
F1P             176.115 ppm
F1             22147.89 Hz
F2P              -4.374 ppm
F2              -550.10 Hz
PPMCM           5.30852 ppm/cm
HZCM          667.58813 Hz/cm
```



24

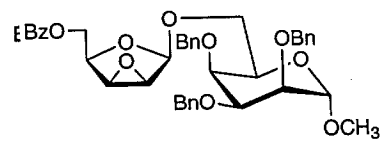
Current Data Parameters
NAME Couplefinal
EXPNO 200
PROCNO 1

F2 - Acquisition Parameters
Date_ 20001104
Time 18.01
INSTRUM spect
PROBHD 5 mm BBO BB-1
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 29
DS 2
SWH 10330.578 Hz
FIDRES 0.157632 Hz
AQ 3.1719923 sec
RG 128
DW 48.400 usec
DE 6.00 usec
TE 300.0 K
D1 1.00000000 sec

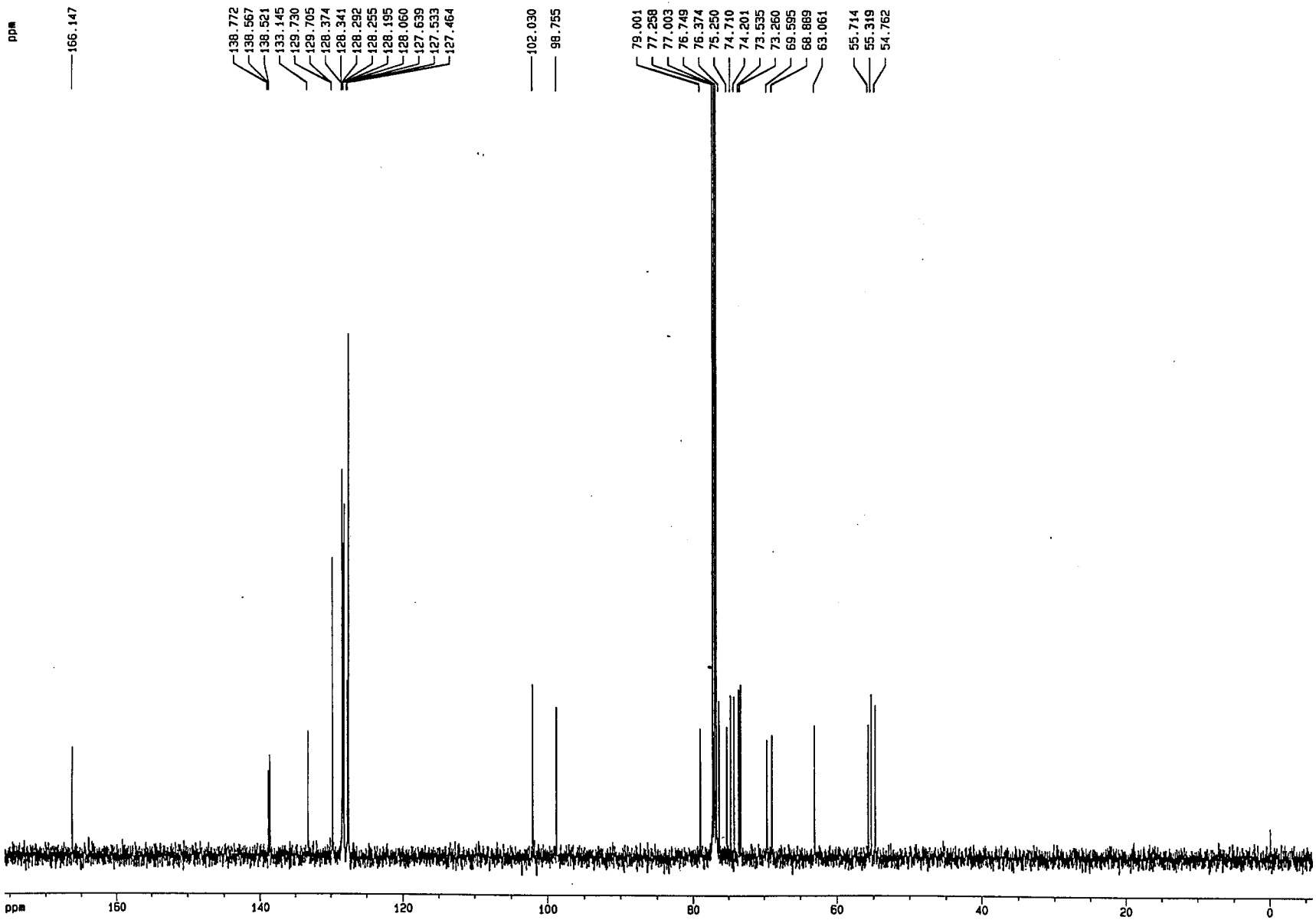
========== CHANNEL f1 ==========
NUC1 1H
P1 13.70 usec
PL1 -1.00 dB
SFO1 500.1330885 MHz

F2 - Processing parameters
SI 32768
SF 500.1300000 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

1D NMR plot parameters
CX 34.00 cm
F1P 9.105 ppm
F1 4553.80 Hz
F2P -0.510 ppm
F2 -255.07 Hz
PPMCM 0.28260 ppm/cm
HZCM 141.43727 Hz/cm



24




```
Current Data Parameters
NAME        CoupleFinal
EXPNO              201
PROCNO               1

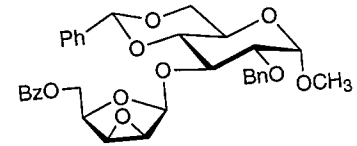
F2 - Acquisition Parameters
Date_         20001104
Time             18.11
INSTRUM          spect
PROBHD    5 mm BBO BB-1
PULPROG         zgpg30
TD               65536
SOLVENT          CDCl3
NS                 156
DS                   4
SWH          31446.541 Hz
FIDRES        0.479836 Hz
AQ            1.0420724 sec
RG               4597.6
DW              15.900 usec
DE               6.00 usec
TE              300.0 K
D1           2.00000000 sec
D11          0.03000000 sec
D12          0.00002000 sec

============ CHANNEL f1 ============
NUC1               13C
P1              7.50 usec
PL1             3.00 dB
SF01       125.7715719 MHz

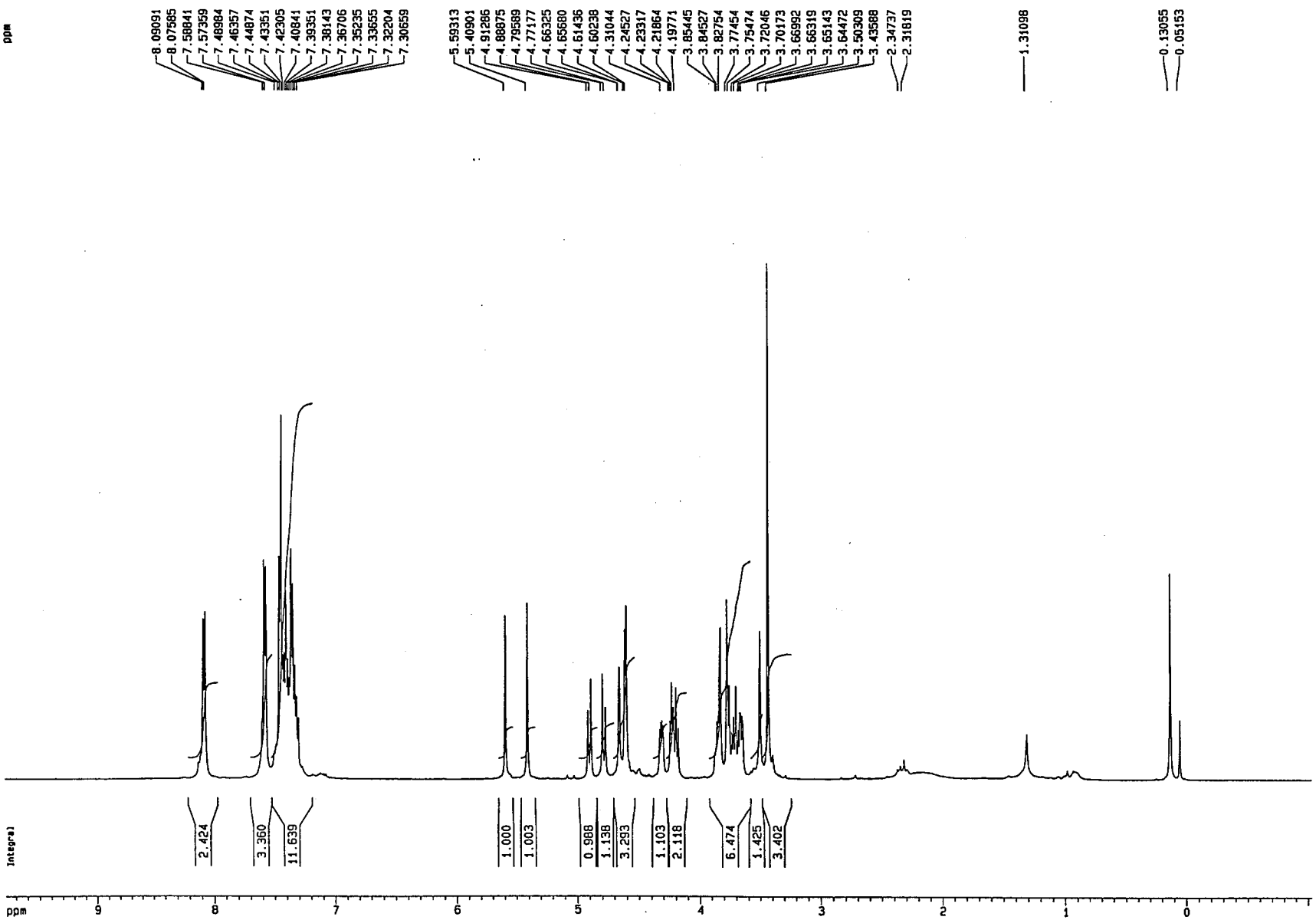
============ CHANNEL f2 ============
CPDPRG2        waltz16
NUC2               1H
PCPD2          100.00 usec
PL2            -1.00 dB
PL12           18.80 dB
PL13           18.80 dB
SF02       500.1320005 MHz

F2 - Processing parameters
SI                32768
SF         125.7577974 MHz
WDW                EM
SSB                0
LB              1.00 Hz
GB                 0
PC              1.40

1D NMR plot parameters
CX              34.00 cm
F1P            175.674 ppm
F1           22092.43 Hz
F2P             -5.911 ppm
F2            -743.33 Hz
PPMCM          5.34074 ppm/cm
HZCM         671.64020 Hz/cm
```



25



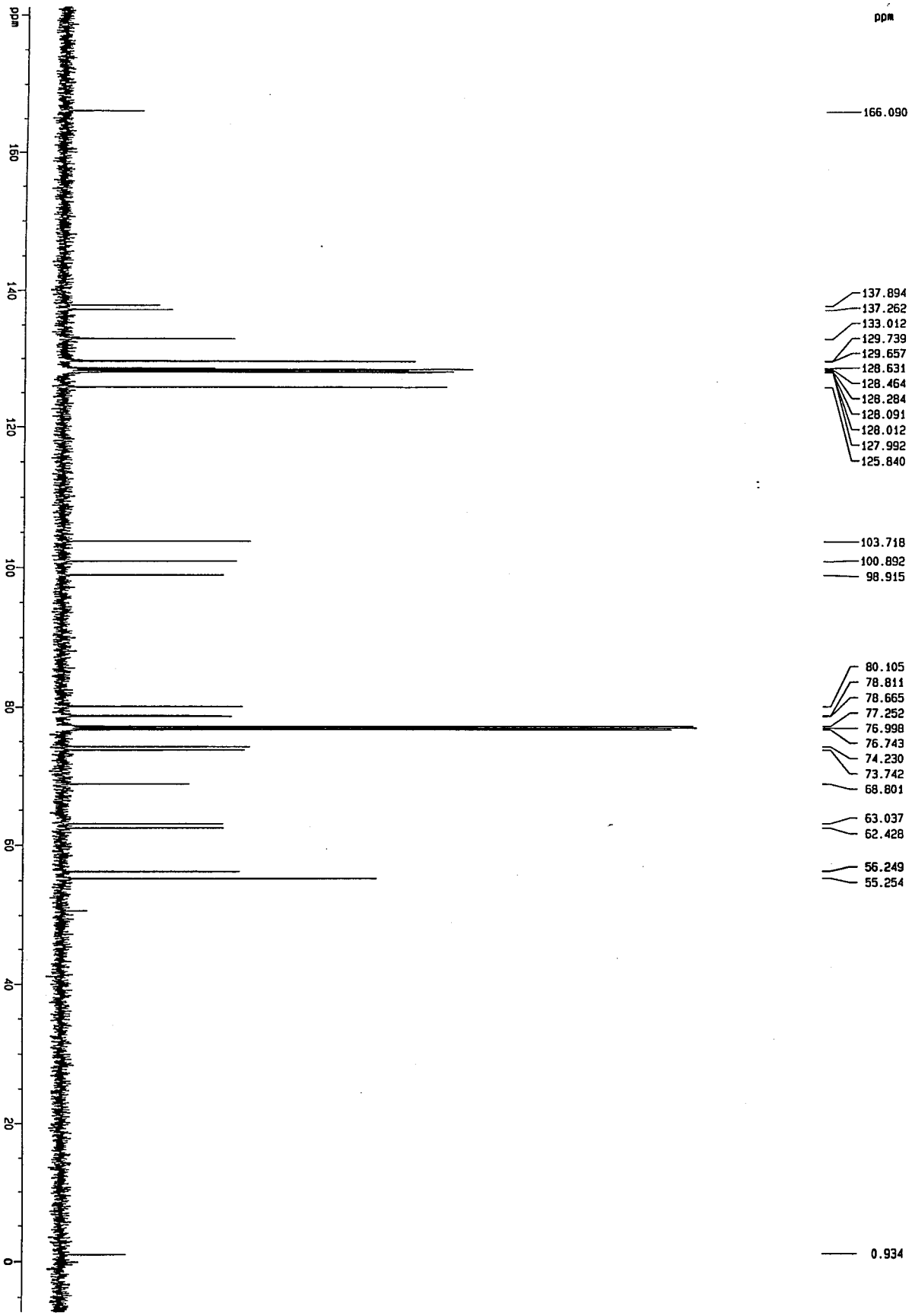
Current Data Parameters
NAME CoupleFinal
EXPNO 180
PROCNO 1

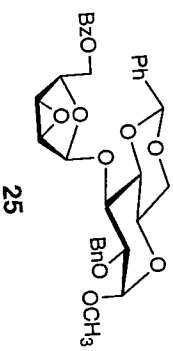
F2 - Acquisition Parameters
Date_ 20001104
Time 16.04
INSTRUM spect
PROBHD 5 mm BBO BB-1
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 32
DS 2
SWH 10330.578 Hz
FIDRES 0.157632 Hz
AQ 3.1719923 sec
RG 64
DW 48.400 usec
DE 6.00 usec
TE 300.0 K
D1 1.00000000 sec

CHANNEL f1
NUC1 1H
P1 13.70 usec
PL1 -1.00 dB
SFO1 500.1330885 MHz

F2 - Processing parameters
SI 32768
SF 500.1300000 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

1D NMR plot parameters
CX 34.00 cm
F1P 9.784 ppm
F1 4893.25 Hz
F2P -1.030 ppm
F2 -515.32 Hz
PPMCM 0.31807 ppm/cm
HZCM 159.07532 Hz/cm





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Current Data Parameters
NAME Couplefinal
EXPNO 181
PROCNO 1

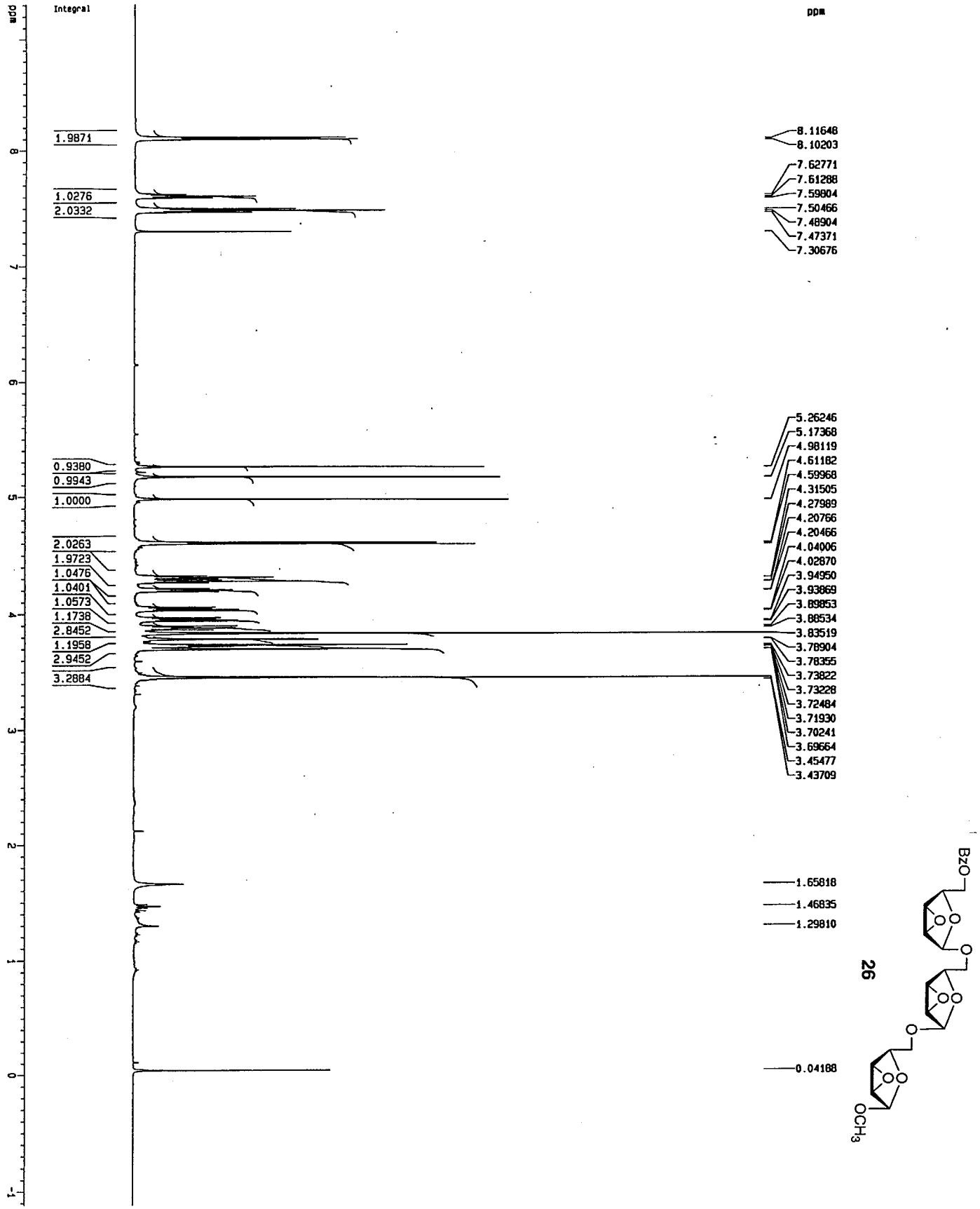
F2 - Acquisition Parameters
Date_ 20001104
Time 16.11
INSTRUM spect
PROBHD 5 mm BBO BB-1
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 97
DS 4
SWH 31446.541 Hz
FIDRES 0.479836 Hz
AQ 1.042072 sec
RG 5792.6
DW 15.900 usec
DE 6.00 usec
TE 300.0 K
D1 2.00000000 sec
D11 0.03000000 sec
D12 0.00002000 sec

========== CHANNEL f1 ==========
NUC1 13C
P1 7.50 usec
PL1 3.00 dB
SFO1 125.771519 MHz

========== CHANNEL f2 ==========
CPDPRG2 waltz16
NUC2 1H
PCPD2 100.00 usec
PL2 -1.00 dB
PL12 18.80 dB
PL13 18.80 dB
SFO2 500.1320005 MHz

F2 - Processing parameters
SI 32768
SF 125.7570042 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

1D NMR plot parameters
CX 34.00 cm
F1P 181.059 ppm
F1 22774.56 Hz
F2P -7.060 ppm
F2 -887.82 Hz
PPMCM 5.53407 ppm/cm
HZCM 695.95300 Hz/cm



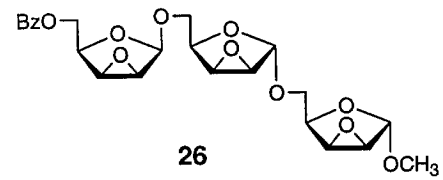
Current Data Parameters
NAME Coupleftwal
EXPNO 150
PROCNO 1

F2 - Acquisition Parameters
Date_ 20001004
Time 8.28
INSTRUM spect
PROBHD 5 mm BBO BB-1
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 41
DS 2
SWH 10330.578 Hz
FIDRES 0.157622 Hz
AQ 3.1719823 sec
RG 228.1
DW 48.400 usec
DE 6.00 usec
TE 308.0 K
D1 1.00000000 sec

======== CHANNEL f1 ========
NUC1 1H
P1 13.70 usec
PL1 -1.00 dB
SFO1 500.1330885 MHz

F2 - Processing parameters
SI 32768
SF 500.1300000 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

1D NMR plot parameters
CX 34.00 cm
F1P 9.389 ppm
F1 4655.63 Hz
F2P -1.121 ppm
F2 -560.58 Hz
PPMCM 0.30606 ppm/cm
HZCM 153.41783 Hz/cm



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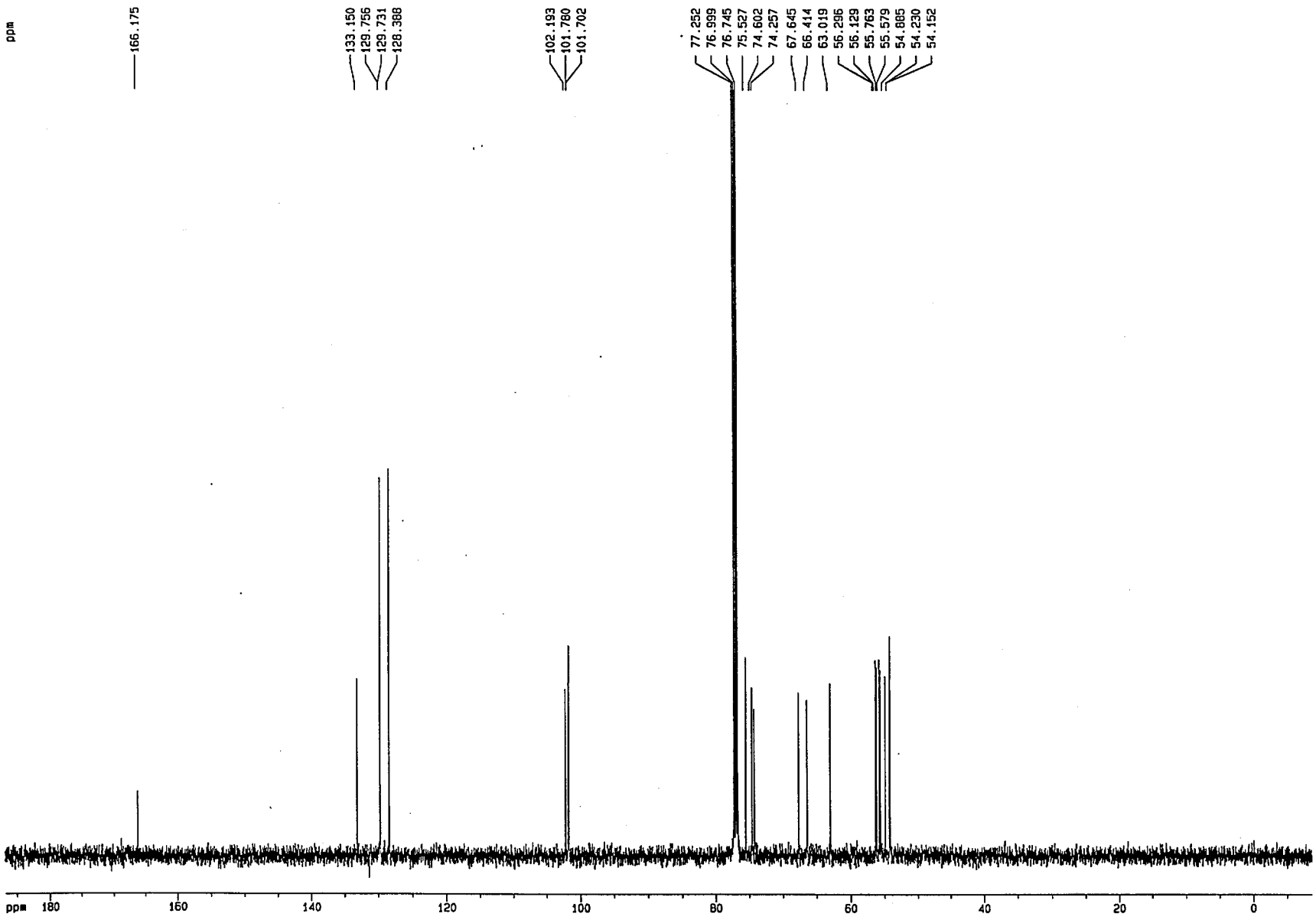



```
Current Data Parameters
NAME          CouplsFinal
EXPNO                 151
PROCNO                  1

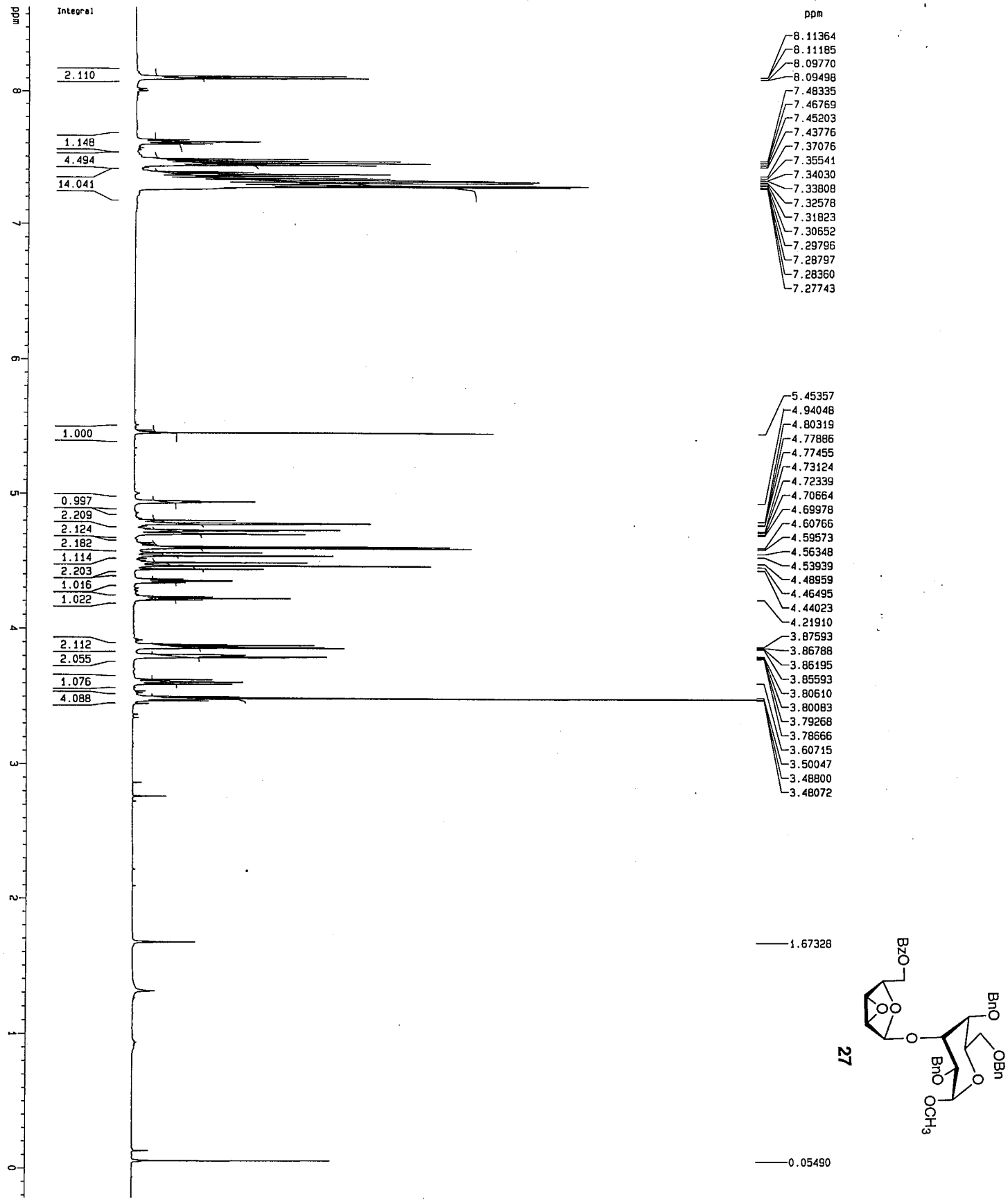
F2 - Acquisition Parameters
Date_          20001104
Time               8.35
INSTRUM           spect
PROBHD    5 mm BBO BB-1
PULPROG          zgpg30
TD                65536
SOLVENT           CDCl3
NS                  276
DS                    4
SWH           31446.541 Hz
FIDRES        0.479836 Hz
AQ            1.0420724 sec
RG               5160.6
DW              15.900 usec
DE               6.00 usec
TE              300.0 K
D1            2.00000000 sec
D11           0.03000000 sec
D12           0.00002000 sec

========== CHANNEL f1 ==========
NUC1               13C
P1              7.50 usec
PL1             3.00 dB
SF01        125.7715719 MHz

========== CHANNEL f2 ==========
CPDPRG2         waltz16
NUC2               1H
PCPD2          100.00 usec
PL2             -1.00 dB
PL12            18.80 dB
PL13            18.80 dB
SF02        500.1320005 MHz

F2 - Processing parameters
SI                32768
SF          125.7577955 MHz
WDW                EM
SSB                0
LB              1.00 Hz
GB                 0
PC              1.40

1D NMR plot parameters
CX             34.00 cm
F1P           186.645 ppm
F1           23472.08 Hz
F2P            -8.634 ppm
F2           -1085.84 Hz
PPMCM          5.74352 ppm/cm
HZCM         722.29187 Hz/cm
```



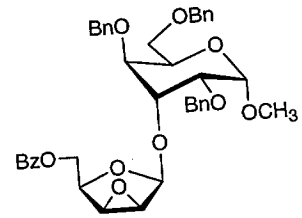
Current Data Parameters
NAME 12-7-00
EXPNO 50
PROCNO 1

F2 - Acquisition Parameters
Date_ 20001207
Time 18.39
INSTRUM spect
PROBHD 5 mm BBO BB-1
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 17
DS 2
SWH 10330.578 Hz
FIDRES 0.157632 Hz
AQ 3.1719923 sec
RG 128
DW 48.400 usec
DE 6.00 usec
TE 300.0 K
D1 1.00000000 sec

========== CHANNEL f1 ==========
NUC1 1H
P1 13.70 usec
PL1 -1.00 dB
SFO1 500.1330885 MHz

F2 - Processing parameters
SI 32768
SF 500.1300000 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

1D NMR plot parameters
CX 34.00 cm
F1P 8.653 ppm
F1 4327.50 Hz
F2P -0.216 ppm
F2 -107.98 Hz
PPMCM 0.26084 ppm/cm
HZCM 130.45508 Hz/cm



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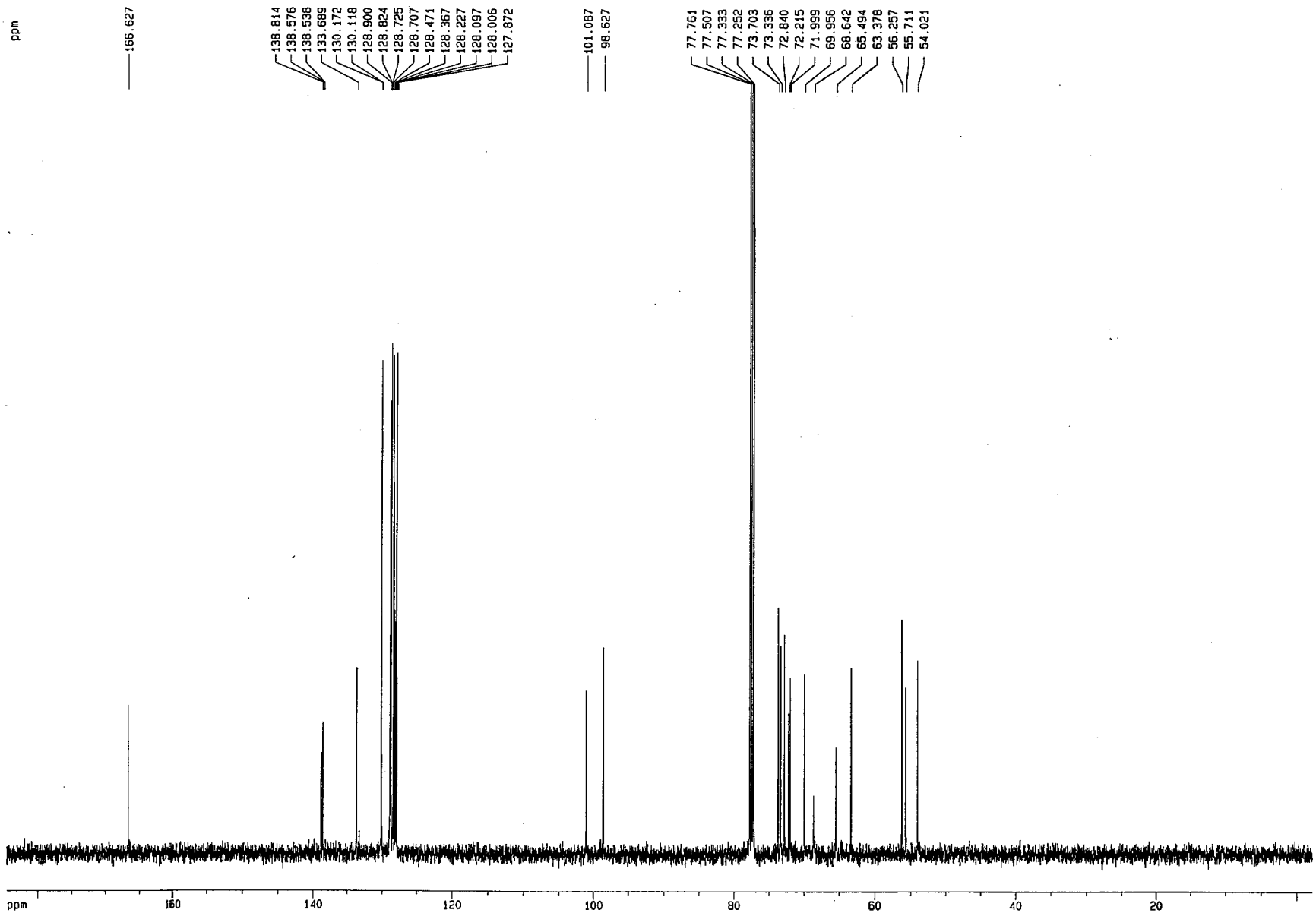



```
Current Data Parameters
NAME                 12-7-00
EXPNO                     11
PROCNO                     1

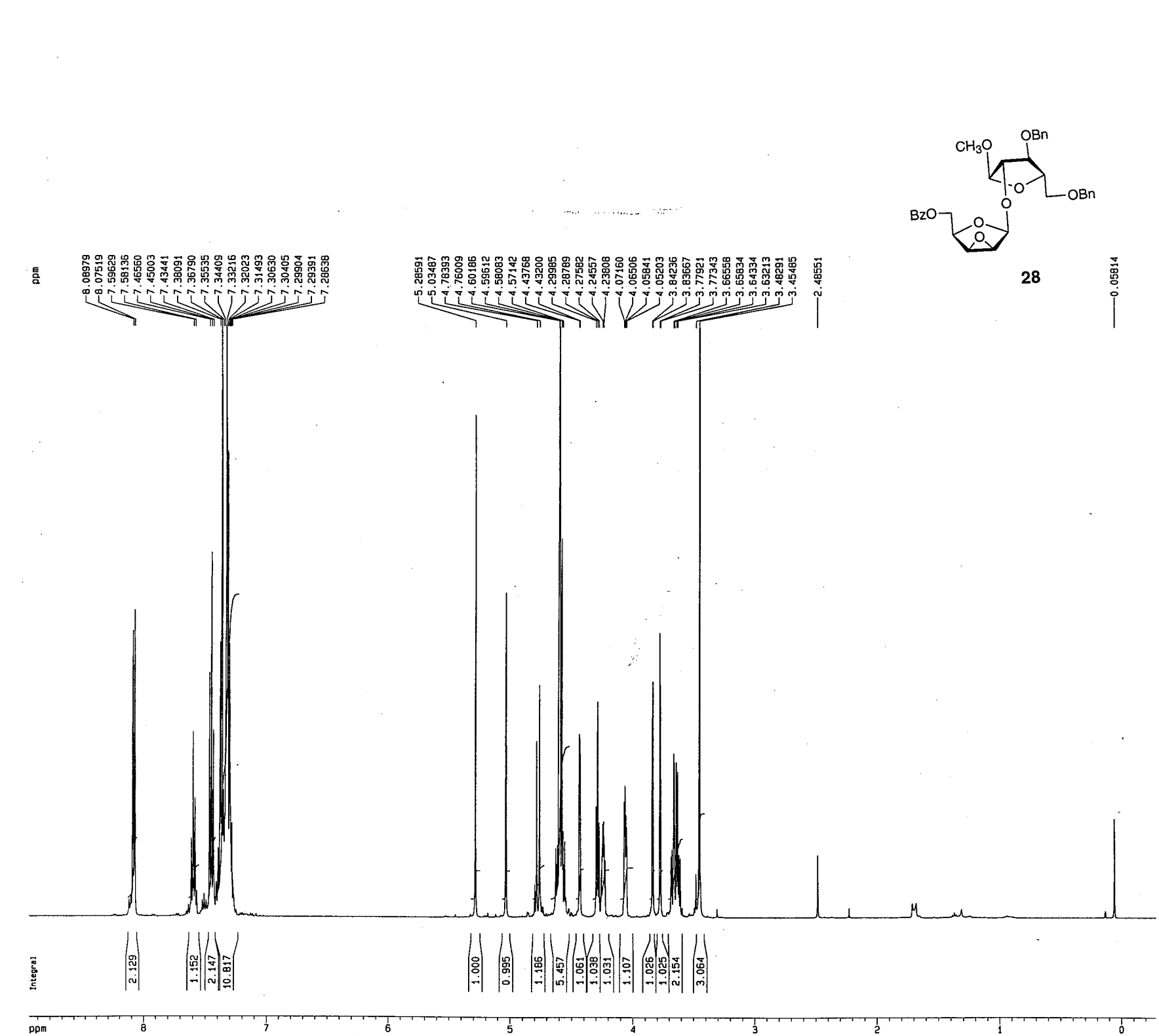
F2 - Acquisition Parameters
Date_              20001207
Time                   8.12
INSTRUM                spect
PROBHD     5 mm BBO BB-1
PULPROG              zgpg30
TD                    65536
SOLVENT               CDCl3
NS                      101
DS                        4
SWH               31446.541 Hz
FIDRES            0.479836 Hz
AQ                1.0420724 sec
RG                      4096
DW                 15.900 usec
DE                  6.00 usec
TE                  300.0 K
D1                2.00000000 sec
D11               0.03000000 sec
D12               0.00002000 sec

============ CHANNEL f1 ============
NUC1                     13C
P1                    8.00 usec
PL1                   3.00 dB
SF01             125.7715719 MHz

============ CHANNEL f2 ============
CPDPRG2              waltz16
NUC2                     1H
PCPD2               100.00 usec
PL2                  -1.00 dB
PL12                 18.80 dB
PL13                 18.80 dB
SF02             500.1320005 MHz

F2 - Processing parameters
SI                       32768
SF               125.7577390 MHz
WDW                         EM
SSB                          0
LB                    1.00 Hz
GB                           0
PC                    1.40

1D NMR plot parameters
CX                   34.00 cm
F1P                184.356 ppm
F1                 23184.16 Hz
F2P                 -2.160 ppm
F2                 -271.58 Hz
PPMCM               5.48574 ppm/cm
HZCM              689.87476 Hz/cm
```



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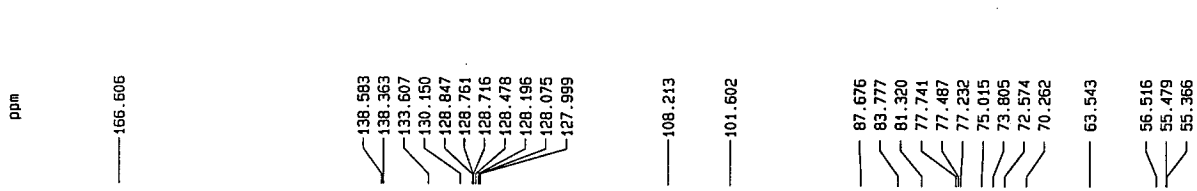
```
Current Data Parameters
NAME                  12-5-00
EXPNO                      10
PROCNO                      1

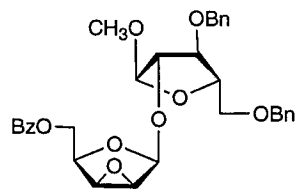
F2 - Acquisition Parameters
Date_               20001205
Time                    8.04
INSTRUM                spect
PROBHD     5 mm BBO BB-1
PULPROG                 zg30
TD                     65536
SOLVENT                CDCl3
NS                         15
DS                          2
SWH              10330.578 Hz
FIDRES           0.157632 Hz
AQ               3.1719923 sec
RG                      101.6
DW                   48.400 usec
DE                    6.00 usec
TE                   300.0 K
D1               1.00000000 sec

========== CHANNEL f1 ==========
NUC1                       1H
P1                      13.70 usec
PL1                     -1.00 dB
SF01            500.1330885 MHz

F2 - Processing parameters
SI                         32768
SF              500.1300000 MHz
WDW                           EM
SSB                            0
LB                      0.30 Hz
GB                             0
PC                      1.00

1D NMR plot parameters
CX                     34.00 cm
F1P                    8.969 ppm
F1                   4485.91 Hz
F2P                   -0.284 ppm
F2                   -141.92 Hz
PPMCM                0.27215 ppm/cm
HZCM               136.11258 Hz/cm
```





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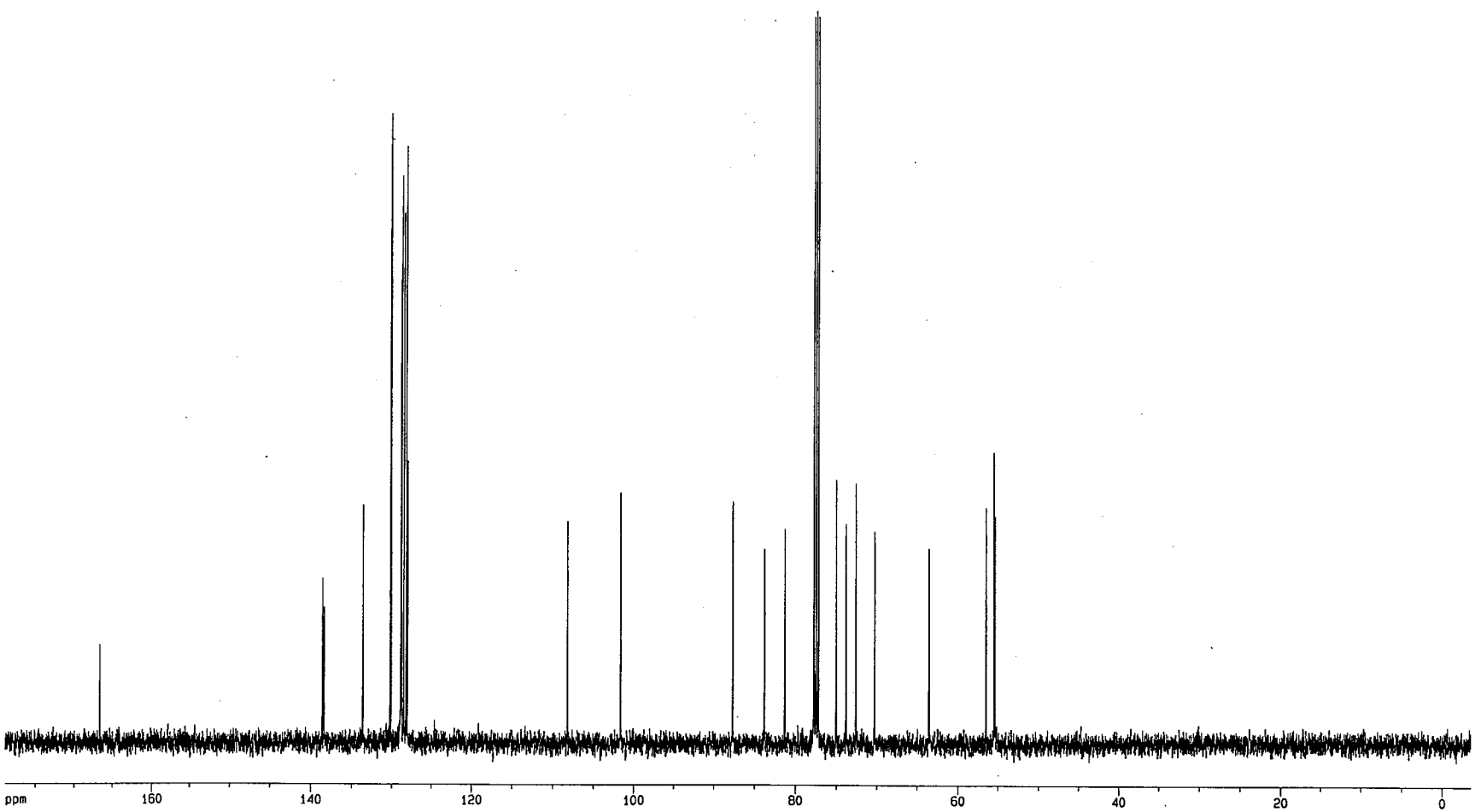



```
Current Data Parameters
NAME                  12-5-00
EXPNO                      11
PROCNO                      1

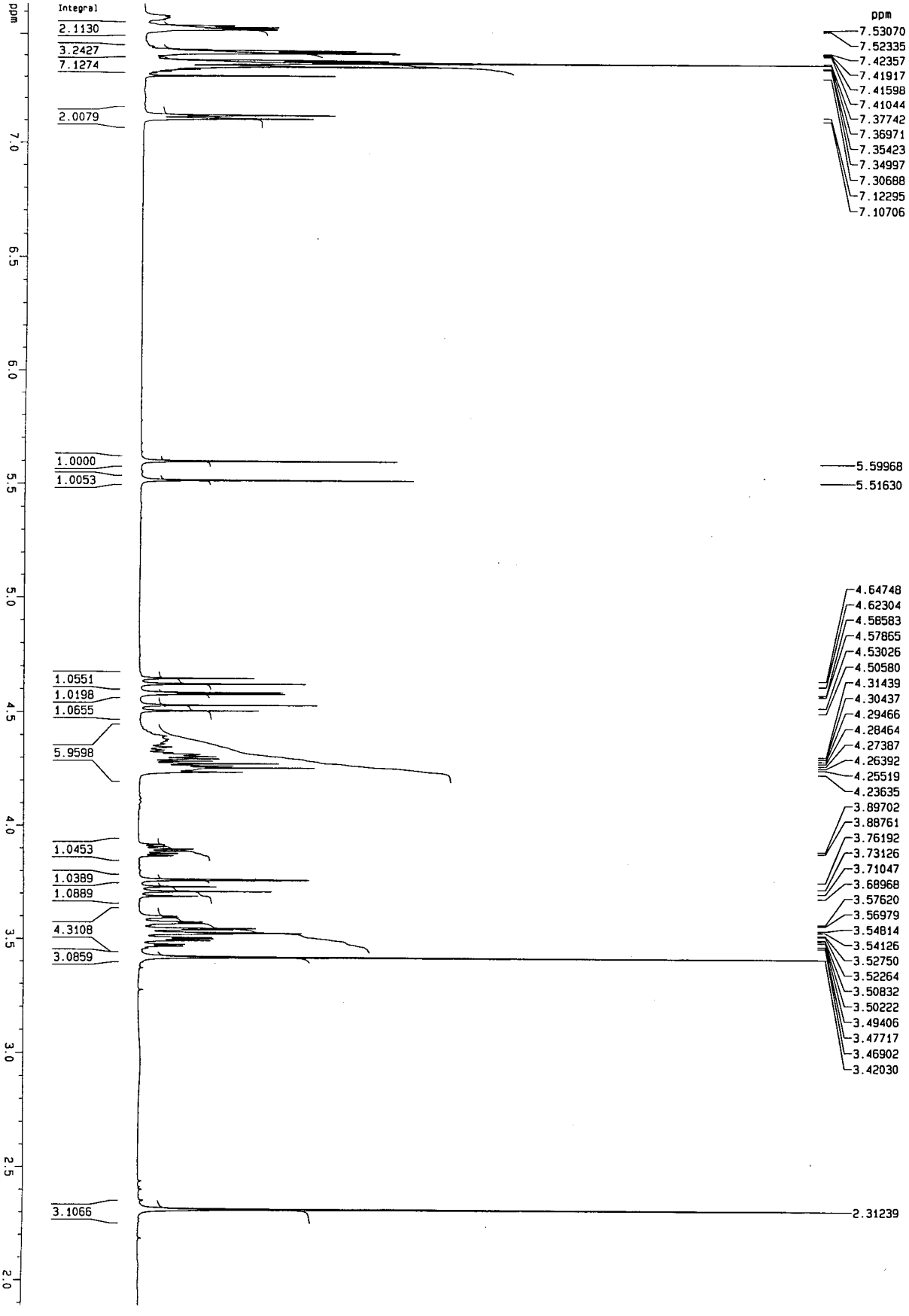
F2 - Acquisition Parameters
Date_               20001205
Time                    8.10
INSTRUM                 spect
PROBHD     5 mm BBO BB-1
PULPROG                zgpg30
TD                      65536
SOLVENT                 CDCl3
NS                         81
DS                          4
SWH                31445.541 Hz
FIDRES             0.479836 Hz
AQ                 1.0420724 sec
RG                       4096
DW                   15.900 usec
DE                    6.00 usec
TE                    300.0 K
D1                 2.00000000 sec
D11                0.03000000 sec
D12                0.00002000 sec

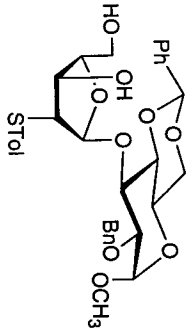
========== CHANNEL f1 ==========
NUC1                      13C
P1                     8.00 usec
PL1                    3.00 dB
SFO1              125.7715719 MHz

========== CHANNEL f2 ==========
CPDPRG2                waltz16
NUC2                       1H
PCPD2                100.00 usec
PL2                   -1.00 dB
PL12                  18.80 dB
PL13                  18.80 dB
SFO2              500.1320005 MHz

F2 - Processing parameters
SI                        32768
SF                125.7577390 MHz
WDW                          EM
SSB                           0
LB                     1.00 Hz
GB                            0
PC                         1.40

1D NMR plot parameters
CX                    34.00 cm
F1P                  178.878 ppm
F1                 22495.30 Hz
F2P                   -3.529 ppm
F2                  -443.79 Hz
PPMCM                5.36491 ppm/cm
HZCM               674.67932 Hz/cm
```





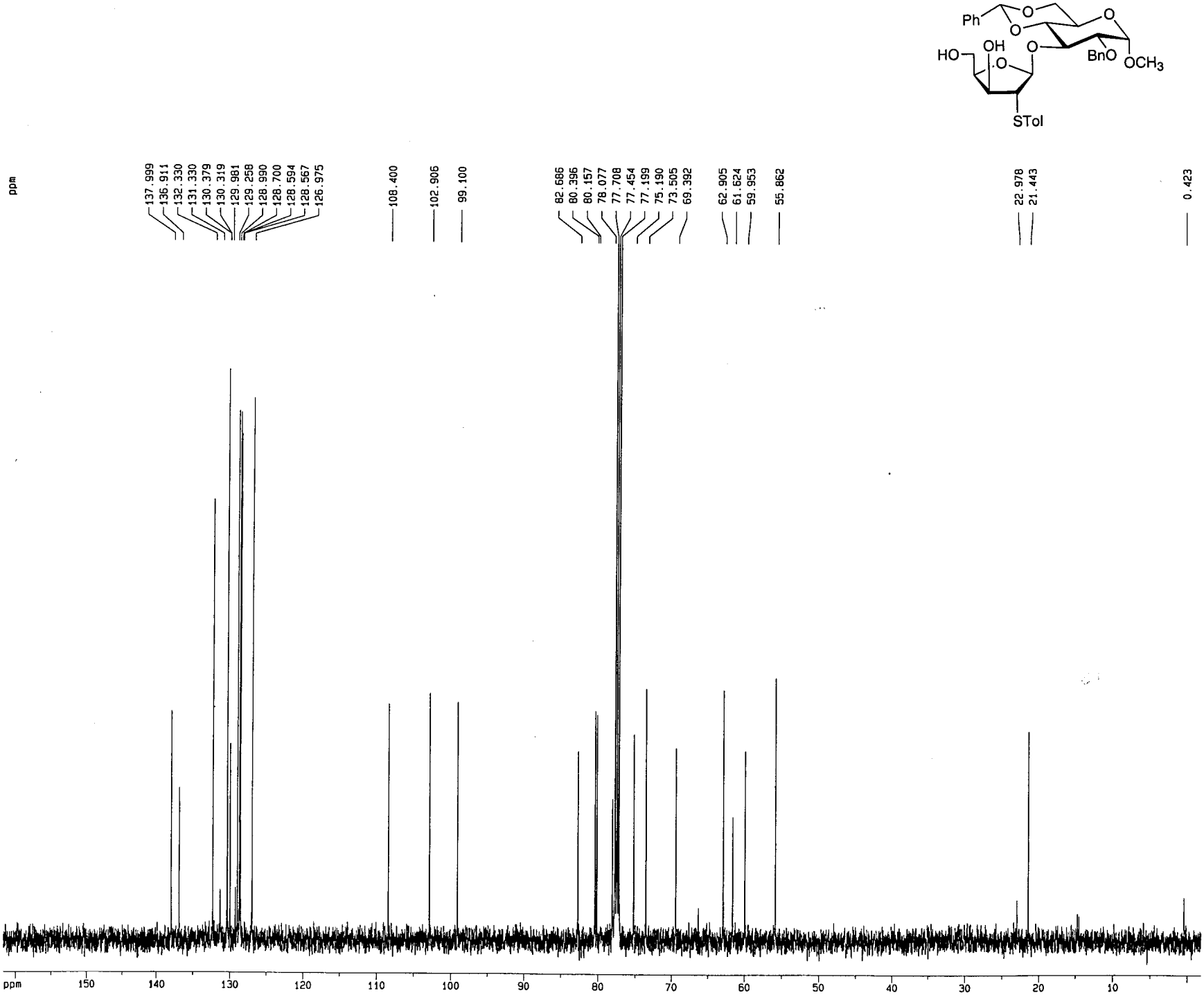

```
Current Data Parameters
NAME                  12-13-00
EXPNO                        10
PROCNO                        1

F2 - Acquisition Parameters
Date_              20001213
Time               13.05
INSTRUM            spect
PROBHD     5 mm BBO BB-1
PULPROG            zg30
TD                 65536
SOLVENT            CDCl3
NS                    53
DS                     2
SWH            10330.578 Hz
FIDRES         0.157632 Hz
AQ             3.1719923 sec
RG                 143.7
DW                 48.400 usec
DE                 6.00 usec
TE                 300.0 K
D1             1.00000000 sec

========== CHANNEL f1 ==========
NUC1                   1H
P1                 13.70 usec
PL1                -1.00 dB
SFO1          500.1330865 MHz

F2 - Processing parameters
SI                    32768
SF            500.1300000 MHz
WDW                      EM
SSB                       0
LB                     0.30 Hz
GB                        0
PC                     1.00

1D NMR plot parameters
CX                    34.00 cm
F1P                 7.635 ppm
F1                3818.32 Hz
F2P                 1.888 ppm
F2                 944.32 Hz
PPMCM             0.16902 ppm/cm
HZCM              84.52956 Hz/cm
```




```
Current Data Parameters
NAME                  12-13-00
EXPNO                       11
PROCNO                       1

F2 - Acquisition Parameters
Date_               20001213
Time                    13.15
INSTRUM                 spect
PROBHD     5 mm BBO BB-1
PULPROG                 zgpg30
TD                       65536
SOLVENT                  CDCl3
NS                         304
DS                           4
SWH                  31446.541 Hz
FIDRES               0.479836 Hz
AQ                   1.0420724 sec
RG                        8192
DW                      15.900 usec
DE                       6.00 usec
TE                      300.0 K
D1                   2.00000000 sec
D11                  0.03000000 sec
D12                  0.00002000 sec

========== CHANNEL f1 ==========
NUC1                       13C
P1                        8.00 usec
PL1                       3.00 dB
SFO1                125.7715719 MHz

========== CHANNEL f2 ==========
CPDPRG2                waltz16
NUC2                       1H
PCPD2                   100.00 usec
PL2                      -1.00 dB
PL12                     18.80 dB
PL13                     18.80 dB
SFO2                500.1320005 MHz

F2 - Processing parameters
SI                        32768
SF                  125.7577390 MHz
WDW                          EM
SSB                           0
LB                        1.00 Hz
GB                            0
PC                         1.40

1D NMR plot parameters
CX                       34.00 cm
F1P                     151.623 ppm
F1                    20325.39 Hz
F2P                      -1.886 ppm
F2                     -237.13 Hz
PPMCM                  4.80909 ppm/cm
HZCM                 604.78003 Hz/cm
```