Supplementary Information

Tunable Acid-Sensitive Ester Protecting Groups in Oligosaccharide Synthesis

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General Information. All chemicals used were reagent grade and used as supplied except where noted. All reactions were performed in oven-dried glassware under an inert atmosphere (nitrogen) unless noted otherwise. Reagent grade dichloromethane (CH_2Cl_2) and dimethylformamide (DMF) were passed through activated neutral alumina column prior to use. HPLC grade toluene (Sigma-Aldrich, cat# 34866) and trifluoroacetic acid (Sigma-Aldrich, cat#T6508) were used as received. Analytical thin layer chromatography (TLC) was performed on Merck silica gel 60 F_{254} plates (0.25mm). Compounds were visualized by UV irradiation or dipping the plate in a cerium sulfate-ammonium molybdate solution. Flash column chromatography (FC) was carried out using Biotage Isolera One Flash Purification System over Silicycle P60 (230-400 mesh) silica gel. ^{1}H and ^{13}C NMR spectra were recorded on a Bruker DRX400 (400 MHz), Bruker DRX500 (500 MHz), or a Bruker AV600 (600 MHz) spectrometer in $CDCl_3$ with chemical shifts referenced to $CDCl_3$ (7.26 ppm for ^{1}H NMR and 77.0 ppm for ^{13}C NMR). Splitting patterns are indicated as s, singlet; d, doublet; t, triplet; q, quartet; brs, broad singlet for ^{1}H NMR data. High-resolution mass spectral (HRMS) analyses were performed by the MS-service at the Department of Chemistry at University of Pittsburgh. HRMS-ESI were run on a Water® Q-TOF instrument. Optical rotations were measured using a Perkin-Elmer 241 polarimeter.

General Procedure 1 - Preparation of PMB- or NAP-modified acetic and benzonic acid analogues 3a, 3b, 4a, 4b: Lactone **3** or **4** (30.0 mmol), 2-(bromomethyl)naphthalene (NAPBr) or PMBCl (75 mmol) and KOH (120 mmol) were weighed into a 250 mL round bottle flask. Toluene (100 mL) was added and the mixture was heated to reflux for 2 d before cooling down to room temperature. The mixture was then diluted with ethyl acetate and washed with H_2O three times. The combined water phase was carefully acidified with 1 N H_2SO_4 solution and extracted with CH_2Cl_2 three times. The combined organic layer was concentrated *in vacuo* to give the target molecule in pure form.

General Procedure 2 – Protection of alcohol with PMB- or NAP-modified acetic and benzonic acid analogues 3a, 3b, 4a, 4b: Alcohol (0.36 mmol), PMB- or NAP-modified acetic and benzonic acid analogues (**3a** or **3b** or **4a** or **4b**) (0.43 mmol), *N*, *N*'-dicyclohexylcarbodiimide (DCC) (0.54 mmol) and 4-dimethylaminopyridine (DMAP) (0.07 mmol) were dissolved in dry CH_2Cl_2 (2 mL).

The mixture was stirred at room temperature for 3 h before being concentrated *in vacuo* to remove the solvent. The crude residue was purified by FC to give the target molecule in pure form.

General Procedure 3 –Removal of PMBAc, PMBBz, NAPAc, NAPBz esters by acid: Ester (0.1 mmol) was dissolved in a premixed solvent of TFA and toluene (1.5 mL, v/v=1/10 for PMBAc and PMBBz, v/v=10/1 for NAPAc, NAPBz) at 0 ºC and was stirred at room temperature for 5-60 min (for PMBAc and PMBBz) or 2-8 h (for NAPAc, NAPBz). The mixture was further diluted with toluene and concentrated *in vacuo* to remove the solvents at room temperature. The crude residue was purified by FC to give the target molecule in pure form. For water-soluble substrates, refer to work-up procedure for **12a** and **12b**.

TFA-mediated deprotection of NAP ethers as global protecting groups: Per-NAPylated dodecyl maltose **1** was obtained by treatment of commercially available dodecyl maltose **2** with excess NAPBr, NaH in DMF. Analytical data for **1:** ^{1}H NMR (500 MHz, $CDCl_3$): δ 7.80–7.20 (m, 48 H), 7.05 (d, 1 H, J = 1.5 Hz), 5.80 (d, 1 H, J = 4.0 Hz), 5.26–4.49 (m, 14 H), 4.28 (d, 1 H, J = 12.0 Hz), 4.22 (t, 1 H, J = 9.0 Hz), 4.06–3.83 (m, 3H), 3.75–3.55 (m, 6 H), 3.46 (m, 1 H). 1.70 (m, 2 H), 1.41 (m, 2 H), 1.22 (m, 18 H), 0.87 (t, 1 H, J = 9.0 Hz). ^{13}C NMR (125 MHz, $CDCl_3$): δ 136.4, 136.3, 133.3, 133.2(2C), 133.1, 132.9(2C), 132.8, 132.7, 128.1, 128.0(2C), 127.9(4C), 127.8, 127.6(4C), 127.5(2C), 126.8, 126.4, 126.3(2C), 126.2, 126.1, 126.0(2C), 125.9, 125.8(3C), 125.7, 125.6, 77.2, 31.9, 29.7, 29.6(2C), 29.5, 29.3, 26.3, 22.7, 14.1. HRMS ES+: *m/z* $C_{101}H_{102}O_{11}$ $[M+Na]^+$ calcd 1698.7213, found 1698.7258. To remove all the NAP ether protecting groups in **1**, general procedure **3** was followed and final purification was achieved by filtration through a pad of silica gel to give **2** in quantitative yield.

4-(4-methoxybenzyloxy)butanoic acid (PMBAcOH) (3a): General procedure 1 using γ-butyrolactone **3** and PMBCl, KOH gave **3a** (68%) as light yellow solid. ^{1}H NMR (500 MHz, $CDCl_3$): δ 8.04 (d, 1 H, J = 9.0 Hz), 7.26 (m, 1 H), 6.94 (d, 1 H, J = 9.0 Hz), 6.87 (m, 1 H), 4.44 (s, 2 H), 3.87 (s, 1 H), 3.79 (s, 3 H), 3.50 (t, 2 H, J = 6.0 Hz), 2.48 (t, 2 H, J = 7.5 Hz), 1.94 (m, 2 H). ^{13}C NMR (125 MHz, $CDCl_3$): δ 164.1, 159.2, 132.4, 130.3, 129.3, 113.8(2C), 72.6, 68.7, 55.5, 55.3, 31.0, 24.8. HRMS-ESI: *m/z* $C_{12}H_{15}O_4$ $[M]^-$ calcd 223.0970, found 223.1003.

4-(naphthalen-2-ylmethoxy)butanoic acid (NAPAcOH) (3b): General procedure 1 using γ-butyrolactone **3** and NAPBr, KOH gave **3b** (74%) as white solid. ^{1}H NMR (300 MHz, $CDCl_3$): δ 7.83–7.42 (m, 7 H), 4.67 (s, 2 H), 3.56 (t, 2 H, J = 6.0 Hz), 2.50 (t, 2 H, J = 7.7 Hz), 1.98 (m, 2 H). ^{13}C NMR (100 MHz, $CDCl_3$): δ 179.8, 125.7, 133.2, 132.9, 128.1, 127.8, 127.6, 126.2, 126.0, 125.8, 125.6, 72.9, 68.9, 30.9, 24.7. HRMS-ESI: *m/z* $C_{15}H_{15}O_3$ $[M]^-$ calcd 243.1021, found 243.1033.

2-((4-methoxybenzyloxy)methyl)benzoic acid (PMBBzOH) (4a): General procedure 1 using phthalide **4** and PMBCl, KOH gave **4a** (69%) as white solid. ^{1}H NMR (500 MHz, $CDCl_3$): δ 8.10 (m, 1 H), 7.75 (d, 1 H, J = 8.0 Hz), 7.60 (t, 1 H, J = 7.5 Hz), 7.40 (t, 1 H, J = 7.5 Hz), 7.35 (d, 1 H, J = 9.0 Hz), 6.91 (d, 1 H, J = 9.0 Hz), 5.00 (s, 2 H), 4.62 (s, 2 H), 3.81 (s, 3 H). ^{13}C NMR (125 MHz, $CDCl_3$): δ 159.2, 141.2, 133.1, 131.5, 130.1, 129.4, 128.1, 127.6, 127.2, 113.8, 72.5, 70.2, 55.2. HRMS ESI: m/z $C_{16}H_{15}O_4$ $[M]^-$ calcd 271.0970, found 271.0973.

2-((naphthalen-2-ylmethoxy)methyl)benzoic acid (NAPBzOH) (4b): General procedure 1 using phthalide **4** and NAPBr, KOH gave **4b** (77%) as white solid. ^{1}H NMR (500 MHz, $CDCl_3$): δ 8.09 (m, 1 H), 7.86–7.73 (m, 5 H), 7.60 (m, 1 H), 7.53–7.40 (m, 4 H), 5.03 (s, 2 H), 4.83 (s, 2 H). ^{13}C NMR (100 MHz, $CDCl_3$): δ 172.2, 141.2, 135.5, 133.3, 133.0, 131.6, 128.2, 127.9, 127.7, 127.4, 127.3, 126.4, 126.1, 125.9, 125.7, 73.0, 70.5. HRMS-ESI: m/z $C_{19}H_{15}O_3$ $[M]^-$ calcd 291.1021, found 291.1010.

Methyl 2,3,4-tri-*O*-benzoyl-6-(4-(4-methoxybenzyloxy)butanoyl)-α-D-glucopyranoside (6a): General procedure 2 using **3a** and **5**, DCC gave **6a** (93%) as colorless foam. $[\alpha]_D^{18}$ = 47.3 (*c* 0.97, $CHCl_3$). ^{1}H NMR (500 MHz, $CDCl_3$): δ 8.00–7.85 (m, 6 H), 7.51–7.22 (m, 11 H), 6.89 (m, 2 H), 6.18 (t, 1 H, J = 10.0 Hz), 5.62 (t, 1 H, J = 9.5 Hz), 5.29 (m, 2 H), 4.42 (s, 2 H), 4.30 (m, 3 H), 3.76 (s, 3 H), 3.46 (m, 5 H), 2.48 (m, 2 H), 1.94 (m, 2 H). ^{13}C NMR (125 MHz, $CDCl_3$): δ 172.9, 165.6(2C), 165.1, 159.0, 133.3(2C), 133.0, 130.4, 129.8, 129.7, 129.5, 129.1, 129.0, 128.9, 128.7, 128.3, 128.1, 113.6, 96.9, 77.2, 72.4, 71.9, 70.3, 69.1, 68.7, 67.4, 62.2, 55.5, 55.1, 30.7, 24.8. HRMS-ESI: m/z $C_{40}H_{40}O_{12}$ $[M+NH_4]^+$ calcd 730.2864, found 730.2886.

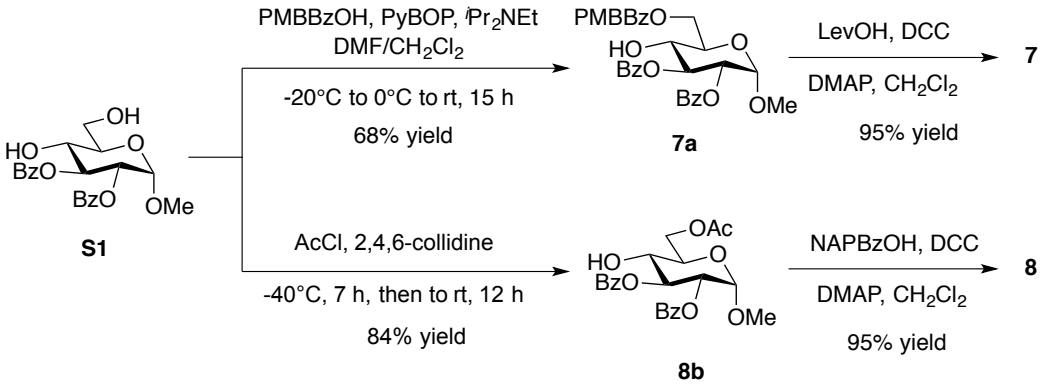
Methyl 2,3,4-tri-*O*-benzoyl-6-(2-((4-methoxybenzyloxy)methyl)benzoyl)-α-D-glucopyranoside (6b): General procedure 2 using **4a** and **5**, DCC gave **6b** (97%) as colorless foam. $[\alpha]_D^{18}$ = 59.1 (*c* 0.23, $CHCl_3$). ^{1}H NMR (500 MHz, $CDCl_3$): δ 8.06–7.42 (m, 7 H), 7.73 (m, 1 H), 7.52–7.28 (m, 13 H), 6.89 (d, 1 H, J = 10.5 Hz), 6.19 (t, 1 H, J = 9.0 Hz), 5.67 (t, 1 H, J = 9.5 Hz), 5.27 (m, 2 H), 4.96 (s, 2 H), 4.58 (s, 2 H), 4.57–4.38 (m, 3 H), 3.80 (s, 3 H), 3.48 (s, 3 H). ^{13}C NMR (125 MHz, $CDCl_3$): δ 166.5, 165.8(2C), 165.3, 159.1, 141.4, 133.4(2C), 133.1, 132.5, 130.5(2C), 130.4, 129.9(2C), 129.7, 129.2, 129.1, 129.0, 128.8, 128.4, 128.3, 127.5, 127.4, 126.9, 113.8(2C), 97.0, 72.5, 72.0, 70.3, 69.9, 69.5, 67.6, 62.9, 55.7, 55.3. HRMS-ESI: m/z $C_{44}H_{40}O_{12}$ $[M+Na]^+$ calcd 783.2417, found 783.2385.

Methyl 2,3,4-tri-*O*-benzoyl-6-(4-4-(naphthalen-2-ylmethoxy)butanoyl)-α-D-glucopyranoside (6c): General procedure 2 using **3b** and **5**, DCC gave **6c** (95%) as colorless foam. $[\alpha]_D^{18}$ = 50.5 (*c* 0.50, $CHCl_3$). ^{1}H NMR (500 MHz, $CDCl_3$): δ 8.00–7.85 (m, 6 H), 7.50–7.21 (m, 12 H), 6.89 (m, 2 H), 6.15 (t, 1 H, J = 10.0 Hz), 5.59 (t, 1 H, J = 10.0 Hz), 5.25 (m, 2 H), 4.43 (s, 2 H), 4.26 (m, 3 H),

3.79 (s, 3 H), 3.47 (m, 5 H), 2.47 (m, 2 H), 1.92 (m, 2 H). ^{13}C NMR (125 MHz, $CDCl_3$): δ 173.1, 165.8, 165.7, 165.2, 159.1, 133.4(2C), 133.1, 130.5, 129.9, 129.8, 129.6, 129.2, 129.1, 129.0, 128.8, 128.4, 128.2, 113.7, 97.0, 72.5, 72.0, 69.2, 68.8, 67.5, 62.3, 55.6, 55.2, 30.8, 24.9. HRMS-ESI: *m/z* $C_{43}H_{40}O_{11}$ $[M+NH_4]^+$ calcd 750.2914, found 750.2925.

Methyl 6-*O*-(2-((naphthalen-2-ylmethoxy)methyl)benzoyl)-2,3,4-tri-*O*-benzoyl-α-D-gluco pyranoside (6d): General procedure 2 using **4b** and **5**, DCC gave **6d** (97%) as colorless foam. $[\alpha]_D^{18}$ = 66.9 (*c* 0.97, $CHCl_3$). ^{1}H NMR (500 MHz, $CDCl_3$): δ 8.04–7.76 (m, 12 H), 7.58–7.26 (m, 14 H), 6.20 (t, 1 H, *J* = 10.0 Hz), 5.67 (t, 1 H, *J* = 10. 0 Hz), 5.29 (m, 1 H), 5.23 (m, 1 H), 5.05 (s, 2 H), 4.82 (s, 2 H), 4.56–4.37 (m, 3 H), 3.45 (s, 3 H). ^{13}C NMR (125 MHz, $CDCl_3$): δ 166.5, 165.8(2C), 165.3, 141.2, 135.9, 133.4, 133.3, 133.1, 133.0, 132.6, 130.6, 129.9(2C), 129.7, 129.2, 129.0, 128.8, 128.4, 128.3, 128.1, 127.9, 127.7, 127.5, 127.0, 126.3, 126.0, 125.8, 125.7, 97.0, 72.9, 72.0, 70.4, 70.3, 69.5, 67.6, 62.9, 55.7. HRMS-ESI: *m/z* $C_{47}H_{40}O_{11}$ $[M+NH_4]^+$ calcd 798.2914, found 798.2946.

SI-Scheme 1. Preparation of glucosides **7** and **8**.



Methyl 2,3-di-*O*-benzoyl-4-*O*-levulinyl-6-*O*-(2-((4-methoxybenzyloxy)methyl)benzoyl)-α-D-glucopyranoside (7): Compound **7a** (189 mg, 0.29 mmol) (prepared via **S1**[1] in one step, SI-Scheme 1), DCC (101 mg, 0.49 mmmol) and DMAP (4 mg, 0.3 mmol) were dissolved in dry CH_2Cl_2 (3 mL). Levulinic acid (44 uL, 0.43 mmol) was added and the mixture was stirred at room temperature for 3 h. After being diluted with ethyl acetate, the mixture was filtered, concentrated *in vacuo* and purified by FC to provide **7** (206 mg, 95%). $[\alpha]_D^{18}$ = 104.3 (*c* 1.06, $CHCl_3$). ^{1}H NMR (500 MHz, $CDCl_3$): δ 8.04–7.32 (m, 16 H), 6.90 (d, 1 H, *J* = 8.5 Hz), 6.01 (t, 1 H, *J* = 9.5 Hz), 5.42 (t, 1 H, *J* = 10.0 Hz), 5.41 (m, 2 H), 5.00 (m, 2 H), 4.60 (s, 3 H), 4.50 (m, 2 H), 4.27 (m, 1 H), 3.81 (s, 3 H), 3.43 (s, 3 H), 2.65–2.35 (m, 4 H), 2.00 (s, 3 H). ^{13}C NMR (125 MHz, $CDCl_3$): δ 205.7, 171.5, 166.4, 165.8, 159.1, 141.3, 133.4, 133.2, 132.5, 130.5(2C), 129.9, 129.8, 129.4, 129.2, 129.0, 128.4, 128.3, 127.6, 127.5, 126.9, 113.8, 113.7, 96.9, 72.5, 71.9, 70.4, 70.0, 68.9, 67.5, 62.5, 55.6, 55.2, 37.8, 29.4, 27.9. HRMS-ESI: *m/z* $C_{42}H_{42}O_{13}$ $[M+Na]^+$ calcd 777.2523, found 777.2540.

Methyl 6-O-acetyl-2,3-di-*O*-benzoyl-4-(2-((naphthalen-2-ylmethoxy)methyl)benzoyl)-α-D-glucopyranoside (8): Compound **8b** (360 mg, 0.36 mmol) (prepared via **S1**[1] in one step, SI-Scheme 1), DMAP (20 mg, 0.16 mmol), **4b** (126 mg, 0.43 mmol) and DCC (307 mg, 1.05 mmol) were dissolved in 3 mL dry CH_2Cl_2. And the mixture was stirred at rt for 3 h. Then diluted with ethyl acetate, filtered and concentrated and purified to provide **6** (553 mg, 95%). $[\alpha]_D^{18} = 65.2$ (*c* 1.26, $CHCl_3$). ^{1}H NMR (400 MHz, $CDCl_3$): δ 8.00–7.58 (m, 10 H), 7.55–7.21 (m, 12 H), 6.14 (t, 1 H, *J* = 9.6 Hz), 5.59 (t, 1 H, *J* = 10.0 Hz), 5.24 (m, 2 H), 4.90 (d, 1 H, *J* = 14.4 Hz), 4.74 (d, 1 H, *J* = 14.4 Hz), 4.63 (m, 2 H), 4.35 (m, 1 H), 4.21 (m, 2 H), 3.45 (s, 3 H), 2.05 (s, 3 H). ^{13}C NMR (100 MHz, $CDCl_3$): δ 170.6, 165.8, 165.7, 165.4, 141.3, 135.8, 133.4, 133.3, 133.2, 132.9, 132.8, 130.4, 129.9, 129.7, 129.1, 129.0, 128.4, 128.3, 128.1, 127.9, 127.7, 127.5, 127.0, 126.8, 126.2, 126.0, 125.8, 97.0, 72.7, 72.0, 70.4, 69.9, 68.8, 67.5, 62.2, 55.6, 20.7. HRMS-ESI: *m/z* $C_{42}H_{38}O_{11}$ $[M+Na]^+$ calcd 741.2312, found 741.2366.

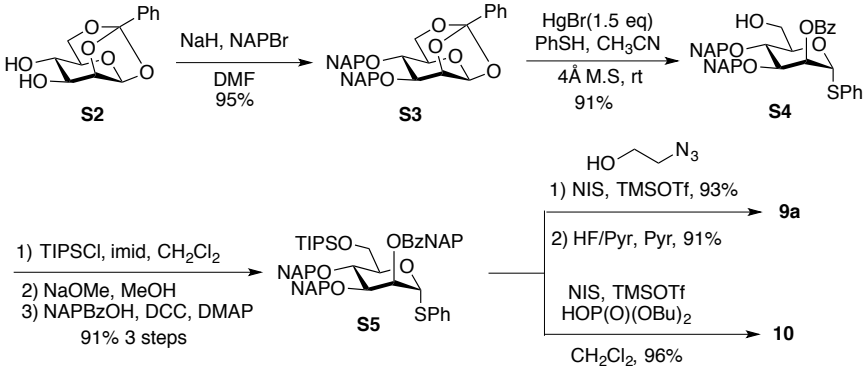
Methyl 2, 3-di-*O*-benzoyl-6-*O*-(2-((4-methoxybenzyloxy)methyl)benzoyl)-α-D-glucopyranoside (7a) via selective deprotection in 7: Compound **7** (36 mg, 0.048 mmol) was dissolved in 4.4 mL THF/MeOH (v : v = 10 : 1). Hydrazine acetate (23 mg, 0.24 mmol) was added and the mixture was stirred for 1.5 h at room temperature. Solvents were removed *in vacuo* and the crude residue was dissolved in ethyl acetate and washed with aqueous $NaHCO_3$. The organic layer was dried over Na_2SO_4 and filtered. Solvents were removed *in vacuo* and the crude residue was purified by FC on silica gel to give **7a** (30.3 mg, 96%). $[\alpha]_D^{18} = 96.0$ (*c* 1.24, $CHCl_3$). ^{1}H NMR (500 MHz, $CDCl_3$): δ 7.99 (m, 5 H), 7.70 (m, 1 H), 7.50 (m, 3 H), 7.32 (m, 8 H), 6.87 (d, 1 H, *J* = 8.5 Hz), 5.78 (t, 1 H, *J* = 10.0 Hz), 5.26 (m, 1 H), 5.13 (d, 1 H, *J* = 4.0 Hz), 4.97 (ab, 2 H, *J* = 18.5, 4.5 Hz), 4.74 (dd, 1 H, *J* = 12.0, 4.5 Hz), 4.58 (m, 3 H), 4.09 (m, 1 H), 3.88 (m, 1 H), 3.79 (s, 3 H), 3.43 (s, 3 H). ^{13}C NMR (125 MHz, $CDCl_3$): δ 167.4, 167.2, 165.9, 159.2, 141.0, 133.4, 132.5, 130.6, 130.3, 129.9, 129.8, 129.4, 129.3, 129.2, 129.1, 128.4, 128.0, 127.9, 127.1, 113.8, 113.7, 97.1, 73.8, 72.6, 72.4, 71.4, 70.0, 69.9, 69.7, 69.4, 63.5, 55.5, 55.2. HRMS-ESI: *m/z* $C_{37}H_{36}O_{11}$ $[M+Na]^+$ calcd 679.2155, found 679.2145.

Methyl 2, 3-di-*O*-benzoyl-4-*O*-levulinyl-α-D-glucopyranoside (7b): General procedure 3 using TFA/Toluene (v : v = 1 : 10) and stirred at room temperature for 5 min gave **7b** (96%) as colorless foam. $[\alpha]_D^{18} = 212.9$ (*c* 0.17, $CHCl_3$). ^{1}H NMR (500 MHz, $CDCl_3$): δ 7.95 (m, 4 H), 7.50 (m, 2 H), 7.37 (m, 4 H), 6.02 (dd, 1 H, *J* = 12.0, 10.0 Hz), 5.30 (t, 1 H, *J* = 10.0 Hz), 5.19 (m, 2 H), 3.92 (m, 1 H), 3.79 (m, 2 H), 3.44 (s, 3 H), 2.67 (m, 1 H), 2.56 (m, 2 H), 2.36 (m, 1 H), 2.08 (s, 3 H). ^{13}C NMR (125 MHz, $CDCl_3$): δ 206.1, 172.5, 165.8(2C), 133.3, 133.2, 129.9, 129.7, 128.4, 97.1, 72.1, 70.3, 69.6, 69.1, 61.0, 55.6, 37.8, 29.5, 27.9. HRMS-ESI: *m/z* $C_{26}H_{28}O_{10}$ $[M+H]^+$ calcd 501.1761, found 501.1765.

Methyl 2,3-di-*O*-benzoyl-4-(2-((naphthalen-2-ylmethoxy)methyl)benzoyl)-α-D-glucopyranoside (8a): Compound **8** (72 mg, 0.1 mmol) was dissolved in a mixture of CH_2Cl_2/MeOH (v:v=1:1) and AcCl (0.1 mL) was added at 0 ºC. After stirred at room temperature for 2 h, the mixture was concentrated *in vacuo* to directly provide **8a** in pure form (68 mg, 100%). $[\alpha]_D^{18}$ = 72.4 (*c* 1.46, $CHCl_3$). ^{1}H NMR (400 MHz, $CDCl_3$): δ 8.00–7.21 (m, 22 H), 6.21 (t, 1 H, *J* = 9.6 Hz), 5.52 (t, 1 H, *J* = 10.0 Hz), 5.25 (m, 2 H), 4.97 (d, 1 H, *J* = 14.4 Hz), 4.77 (d, 1 H, *J* = 14.0 Hz), 4.67 (ab, 2 H, *J* = 18.0, 12.0 Hz), 3.97 (d, 1 H, *J* = 11.6 Hz), 3.70 (m, 2 H), 3.42 (s, 3 H). ^{13}C NMR (100 MHz, $CDCl_3$): δ 166.5, 165.8, 141.1, 135.6, 133.3, 133.2(2C), 132.9, 130.6, 129.9, 129.6, 129.1, 129.0, 128.4, 128.3, 128.1, 128.0, 127.9, 127.6, 127.3, 126.8, 126.3, 126.0, 125.8, 125.7, 97.0, 72.7, 72.1, 70.2, 70.1, 69.6, 69.2, 60.9, 55.6. HRMS-ESI: *m/z* $C_{40}H_{36}O_{10}$ $[M+NH_4]^+$ calcd 694.2652, found 694.2708.

Methyl 6-*O*-acetyl-2,3-di-*O*-benzoyl-α-D-glucopyranoside (8b): General procedure 3 using **8** (72 mg, 0.1 mmol), 2.0 mL TFA/Toluene (v : v = 10 : 1), room temperature for 2 h to provide **8b** (43 mg, 97%) as colorless foam. $[\alpha]_D^{18}$ = 137.1 (*c* 3.42, $CHCl_3$). ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.97 (m, 4 H), 7.49 (m, 2 H), 7.35 (m, 4 H), 5.77 (t, 1 H, *J* = 10.0 Hz), 5.24 (dd, 1 H, *J* = 9.2, 3.6 Hz), 5.13 (d, 1 H, *J* = 3.6 Hz), 4.50 (dd, 1 H, *J* = 12.0, 4.8 Hz), 4.40 (dd, 1 H, *J* = 12.0, 2.0 Hz), 3.43 (m, 4 H), 2.14 (s, 3 H). ^{13}C NMR (100 MHz, $CDCl_3$): δ 171.4, 167.1, 165.9, 133.3, 129.8(2C), 129.1, 129.0, 128.3, 128.3, 97.0, 73.7, 71.3, 69.7, 69.4, 62.9, 55.3, 20.8. HRMS-ESI: *m/z* $C_{23}H_{24}O_9$ $[M+Na]^+$ calcd 467.1318, found 467.1305.

SI-Scheme 2. Preparation of mannosides **9a** and **10**.



2-Azidoethyl 2-*O*-(2-((4-methoxybenzyloxy)methyl)benzoyl)-2,3-di-*O*-(2-methylnaphthyl)-α-D-mannopyranoside (9a): Prepared from S2[2] in seven steps (SI-Scheme 2). $[\alpha]_D^{18}$ = -33.3 (*c* 0.48, $CHCl_3$). ^{1}H NMR (400 MHz, $CDCl_3$): δ 8.00 (m, 1 H), 7.78–7.30 (m, 24 H), 5.63 (m, 1 H), 5.10–4.62 (m, 9 H), 4.20 (dd, 1 H, *J* = 9.2, 3.2 Hz), 4.02 (t, 1 H, *J* = 9.2 Hz), 3.87–3.63 (m, 4 H), 3.48 (m, 1 H), 3.22 (m, 2 H). ^{13}C NMR (100 MHz, $CDCl_3$): δ 166.2, 140.8, 135.8, 135.5, 135.3, 133.2, 133.1(2C), 132.9, 132.8, 132.6, 130.7, 128.0(2C), 127.8(2C), 127.7, 127.6, 127.5, 127.1, 126.6, 126.1, 126.0, 125.9(2C), 125.8, 125.7(2C), 125.5, 97.8, 77.8, 75.1, 73.9, 72.5, 72.1, 71.6, 70.3, 69.0, 66.7, 61.9, 50.2. HRMS-ESI: *m/z* $C_{58}H_{65}N_3O_8Si$ $[M+Na]^+$ calcd 982.4439, found 982.4482.

Dibutyl 6-*O*-triisopropylsilyl-2-*O*-(2-((4-methoxybenzyloxy)methyl)benzoyl)-2,3-di-*O*-(2-methylnaphthyl)-α-D-mannopyranoside phosphate (10): Prepared from S2[2] in six steps (SI-Scheme 2). $[\alpha]_D^{18}$ = -29.7 (*c* 2.48, $CHCl_3$). ^{1}H NMR (300 MHz, $CDCl_3$) δ 8.08 (m, 1 H), 7.85–7.26 (m, 24 H), 5.80 (dd, 1 H, *J* = 6.3, 1.8 Hz), 5.69 (t, 1 H, *J* = 2.7 Hz), 5.14–4.63 (m, 8 H), 4.24–3.87 (m, 9 H), 1.61 (m, 4 H), 1.38 (m, 4 H), 1.08 (m, 21 H), 0.91 (t, 1 H, *J* = 7.2 Hz). ^{13}C NMR (125 MHz, $CDCl_3$) *δ* 165.6, 141.6, 136.0, 135.9, 135.3, 133.3, 133.2, 133.0, 132.7, 130.8, 128.0(2C), 127.9, 127.6(3C), 127.4, 126.8, 126.7, 126.4, 126.1, 126.0, 125.9, 125.8(2C), 125.7, 125.6, 74.5, 72.8, 70.4, 67.9, 67.8, 67.7, 32.2(3C), 32.1, 18.6, 18.0, 17.9, 13.5, 12.0.

2-Azidoethyl 6-*O*-triisopropylsilyl-2-*O*-(2-((4-methoxybenzyloxy)methyl)benzoyl)-2,3-di-*O*-(2-methylnaphthyl)-α-D-mannopyransyl-(1→6)-2-*O*-(2-((4-methoxybenzyloxy)methyl) benzoyl)-2, 3-di-*O*-(2-methylnaphthyl)-α-D-mannopyranoside (11a): Mannosyl dibutylphosphate **10** (39 mg, 0.036 mmol), mannoside **9** (24 mg, 0.03 mmol) and 4Å molecular sieves (100 mg) were weighed into a 10 mL round bottom flask under N_2 atmosphere. Dry CH_2Cl_2 (2 mL) was added and the mixture was stirred at room temperature for 1 h before cooled to -30 ℃ and TBSOTf (8.3 uL, 0.036 mmol) was added. The mixture was stirred between -30℃ to -20℃ for 1 h and quenched with drops of Et_3N. The mixture was diluted with CH_2Cl_2, washed with brine and the organic layer was dried with Na_2SO_4. Solvents were evaporated *in vacuo* and the crude residue was purified by FC to provide **11a** (34 mg, 97%). $[\alpha]_D^{18}$ = -13.3 (*c* 2.15, $CHCl_3$). ^{1}H NMR (400 MHz, $CDCl_3$): *δ* 8.10 (m, 2 H), 7.8–7.15 (m, 48 H), 5.74 (s, 1 H), 5.69 (s, 1 H), 5.11–4.55 (m, 18 H), 4.20–3.65 (m, 11 H), 3.40 (m, 1 H), 3.18 (m, 1 H), 0.95 (m, 21 H). ^{13}C NMR (125 MHz, $CDCl_3$): *δ* 166.3, 166.0, 141.2, 141.0, 136.2, 135.9(2C), 135.7, 135.4, 135.3, 133.2(3C), 133.1, 132.9(2C), 132.8, 130.9, 130.7, 128.0, 127.9(2C), 127.8(2C), 127.6(2C), 127.5, 127.4, 127.3, 126.9, 126.8, 126.6, 126.3, 126.1(2C), 126.0(2C), 125.9(2C), 125.8, 125.7, 125.6(3C), 125.4, 97.8(2C), 78.3, 78.0, 77.2, 75.2, 75.0, 74.2, 72.7, 72.6, 71.7, 71.3, 70.4, 68.9, 66.6, 66.0, 50.2, 17.9(2C), 11.9. HRMS-ESI: *m/z* $C_{49}H_{45}N_3O_8$ $[M+Na]^+$ calcd 826.3104, found 826.3140.

2-Azidoethyl α-D-mannopyranosyl-(1→6)-α-D-mannopyranoside (12a): Compound **11a** (22mg, 0.024 mmol) was dissolved in a mixture of TFA/toluene (3.3 mL, v/v = 10 : 1) at 0 ºC and was stirred at 0 ºC for 2 h, warmed to room temperature and stirred for additional 2 h. The mixture was then diluted with toluene (10 mL), concentrated *in vacuo* at room temperature. The crude residue was then treated with MeOH (5 mL) and sodium methoxide (1 mg) (to remove trace amount of trifluoroacetates that are present on the carbohydrate backbone) and stirred at room temperature for 2 h. Solvent was then evaporated *in vacuo*. The crude residue was dissolved with H_2O and extracted with Et_2O three times. The water phase was concentrated to provide compound **10a** (quant). $[\alpha]_D^{18}$ = 35.5 (*c* 0.48, MeOH). ^{1}H NMR (500 MHz, CD_3OD) *δ* 4.86 (d, 1 H, *J* = 1.5 Hz), 4.82 (d, 1 H, *J* = 1.5

Hz), 3.97–3.83 (m, 5 H), 3.80-3.64 (m, 9 H), 3.46 (t, 2 H, *J* = 5.0 Hz). ^{13}C NMR (100 MHz, $CDCl_3$) δ 100.5, 99.9, 72.9, 72.0, 71.2, 70.7, 70.6, 67.1, 67.0, 66.4, 66.0, 61.3, 50.4. HRMS-ESI: *m/z* $C_{14}H_{25}N_3O_{11}Na$ $[M+Na]^+$ calcd 434.1387, found 434.1387.

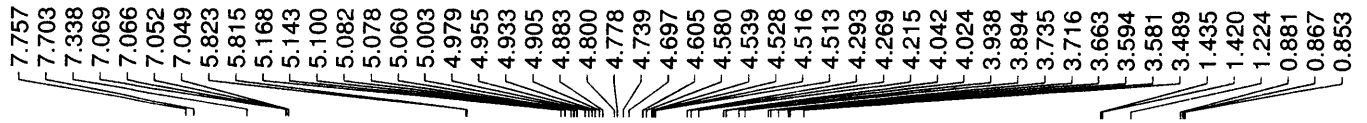
2-Azidoethyl 6-*O*-triisopropylsilyl-2-*O*-(2-((4-methoxybenzyloxy)methyl)benzoyl)-2,3-di-*O*-(2-methylnaphthyl)-α-D-mannopyransyl-(1→6)-2-*O*-(2-((4-methoxybenzyloxy)methyl)benzoyl)-2,3-di-*O*-(2-methylnaphthyl)-α-D-mannopyransyl-(1→6)-2-*O*-(2-((4-methoxybenzyloxy)methyl)benzoyl)-2,3-di-*O*-(2-methylnaphthyl)-α-D-mannopyranoside (11b): The synthesis of trimmannoside **11b** was carried our in an analogous manner as described for **11a**, except the corresponding glycosyl acceptor is dimmanoside **9b**. $[\alpha]_D^{18}$ = -1.6 (*c* 1.75, $CHCl_3$). ^{1}H NMR (500 MHz, $CDCl_3$) δ 8.37–8.09 (m, 3 H), 7.87–7.13 (m, 72 H), 5.86 (t, 1 H, *J* = 2.0 Hz), 5.81 (s, 1 H), 5.76 (t, 1 H, *J* = 1.5 Hz), 5.20–4.51 (m, 26 H), 4.17 (d, 1 H, *J* = 1.5 Hz), 4.21–3.88 (m, 10 H), 3.78 (m, 3 H), 3.66 (m, 3 H), 3.48 (m, 1 H), 3.23 (m, 2 H), 1.06–0.83 (m, 21 H). ^{13}C NMR (125 MHz, $CDCl_3$) δ 166.3, 166.1, 165.9, 141.3, 141.1(2C), 136.3, 135.9 (3C), 135.6, 135.4, 135.3, 135.1, 133.3, 133.2, 133.1(2C), 133.0, 132.9, 132.8(2C), 132.7, 132.6, 130.9, 130.8, 130.7, 128.1, 128.0(2C), 127.9, 127.8, 127.7, 127.6(2C), 127.5(3C), 127.3, 127.0, 126.8, 126.7, 126.3, 126.2, 126.1(2C), 126.0(2C), 125.9(2C), 125.8(2C), 125.7, 125.6(3C), 125.5(2C), 125.3, 98.2, 98.1, 97.9, 78.3, 78.0, 75.1, 74.1(2C), 72.7, 72.6(2C), 71.8, 71.2, 71.0, 70.5, 68.9, 68.6, 66.7, 66.2, 50.2, 25.8, 17.9(2C), 11.9. HRMS-ESI: *m/z* $C_{152}H_{149}N_4O_{22}Si$ $[M+Na]^+$ calcd 2410.0433, found 2410.0239.

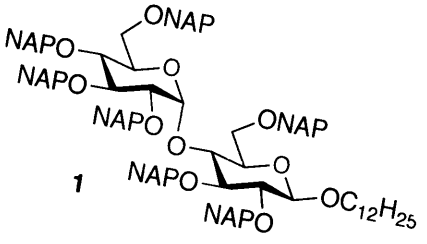
2-Azidoethyl α-D-mannopyransyl-(1→6)-α-D-mannopyransyl-(1→6)-α-D-mannopyranoside (12b): Removal of all protecting groups by TFA treatment in trimannoside **11b** (11 mg, 0.0045 mmol) was carried out in an identical fashion as described for **12a**. $[\alpha]_D^{18}$ = 18.9 (*c* 0.29, MeOH). ^{1}H NMR (500 MHz, CD_3OD) δ 4.89 (d, 1 H, *J* = 2.4 Hz), 4.84 (d, 1 H, *J* = 1.5 Hz), 4.82 (d, 1 H, *J* = 1.8 Hz), 3.96–3.61 (m, 20 H), 3.46 (t, 2 H, *J* = 5.1 Hz). ^{13}C NMR (125 MHz, $CDCl_3$) δ 110.8, 110.1, 110.9, 74.4, 73.4, 72.8, 72.6, 72.5, 72.4, 72.0(2C), 68.6(2C), 68.5, 67.8, 67.3, 67.2, 62.7, 51.8. HRMS-ESI: *m/z* $C_{20}H_{35}N_3O_{16}Na$ $[M+Na]^+$ calcd 596.1915, found 596.1921.

References:

[1] Roy B, Verma P and Mukhopadhyay B. *Carbo. Res.* **2009**, *344*, 145–148.

[2] Liu X, Wada R, Boonyarattanakalin S, Castagner B and Seeberger PH. *Chem. Commun.* **2008**, *30*, 3510–2.





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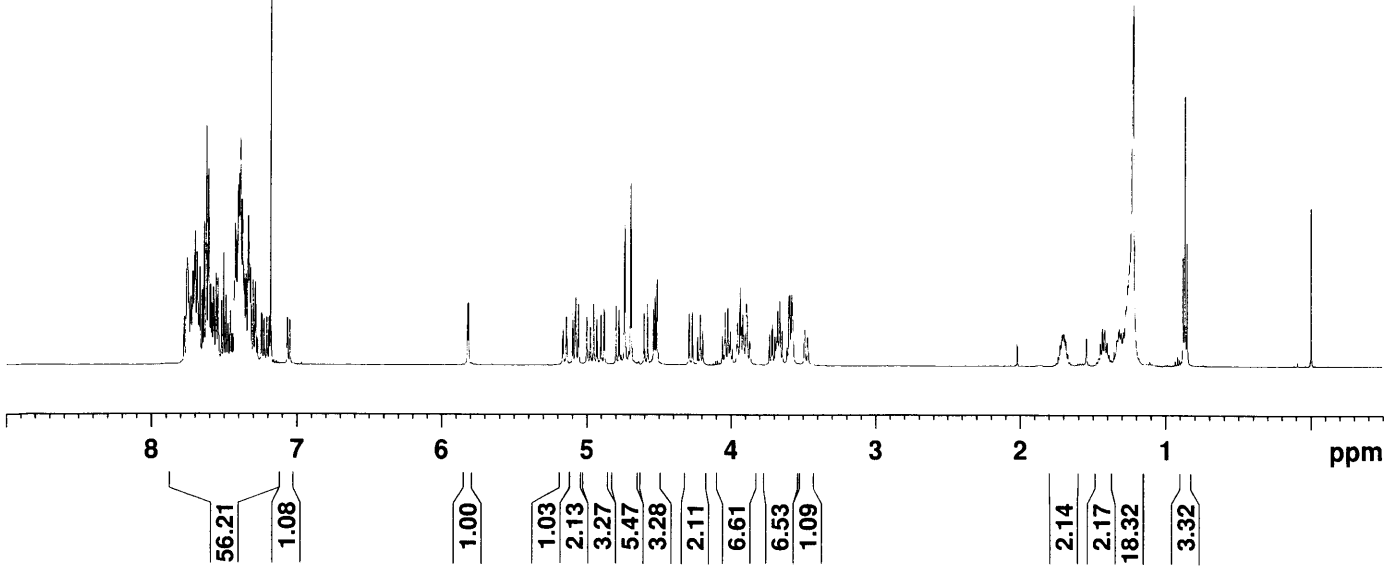


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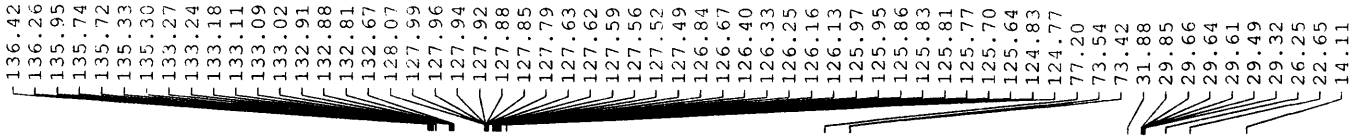

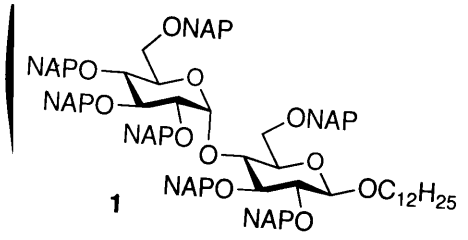

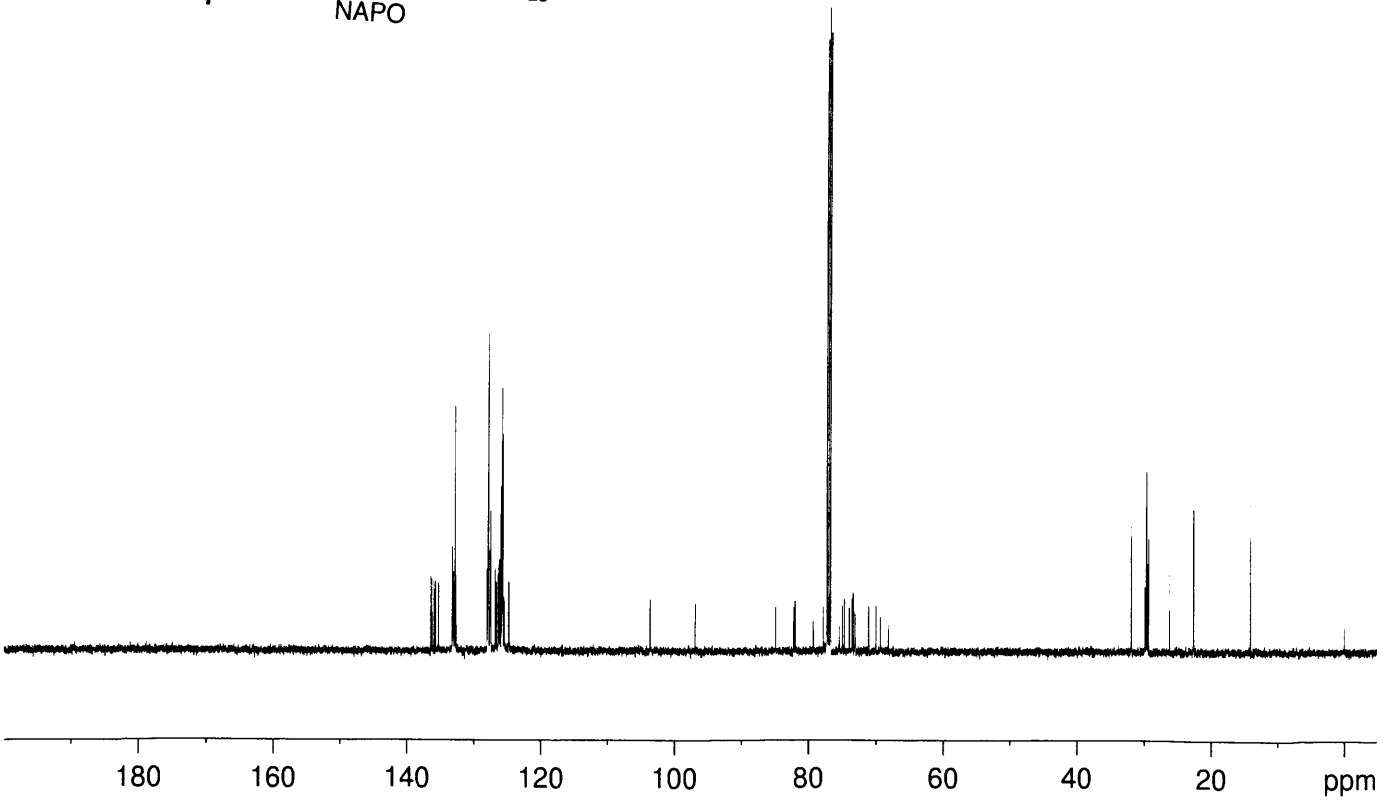

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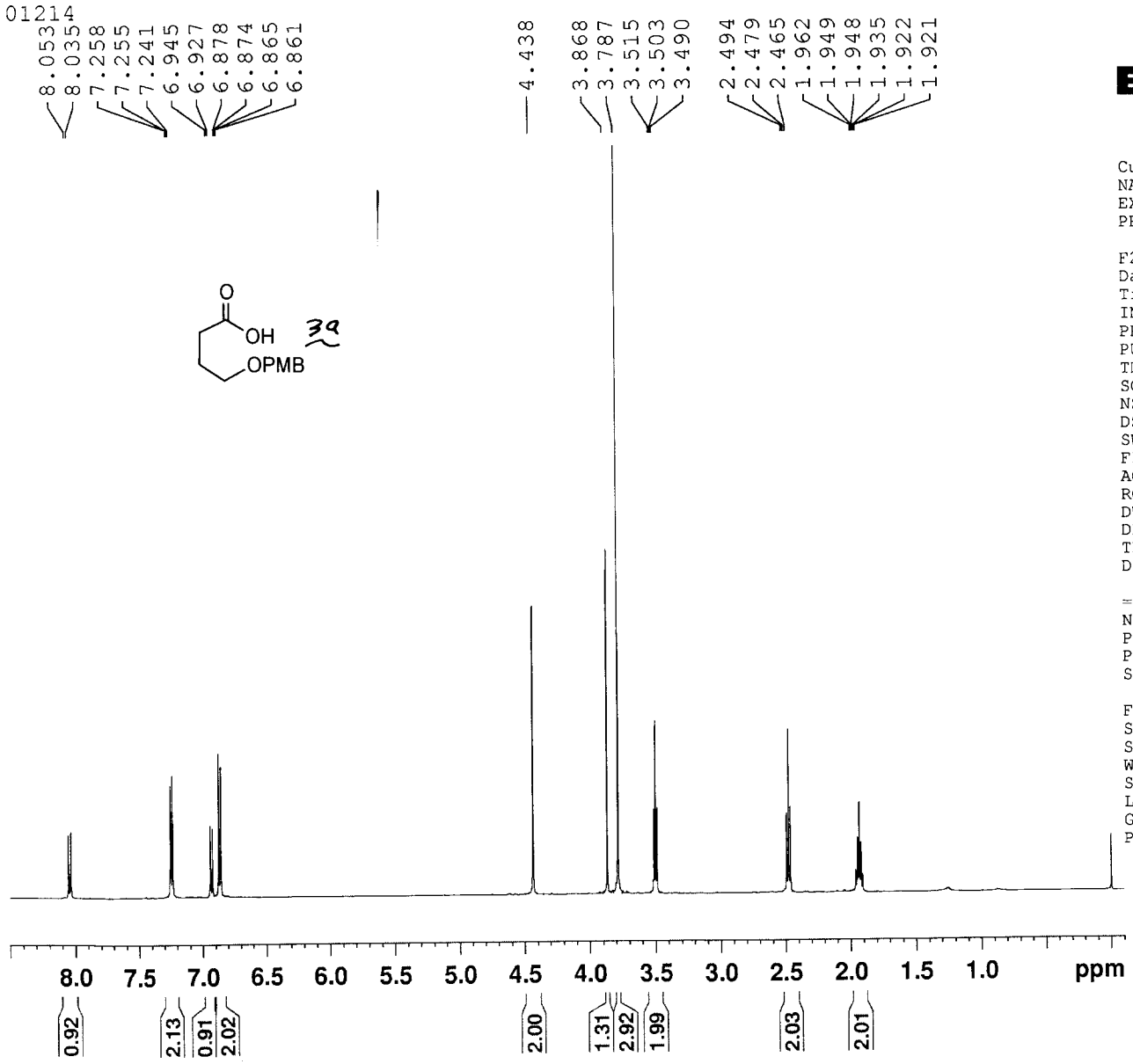

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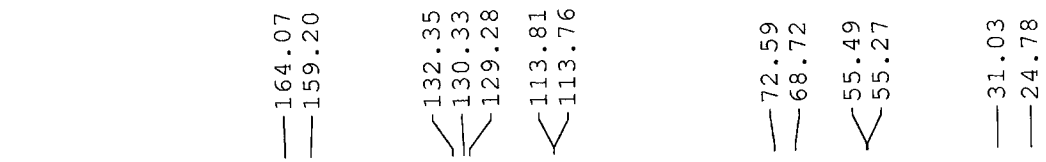

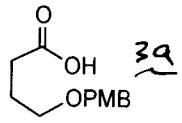

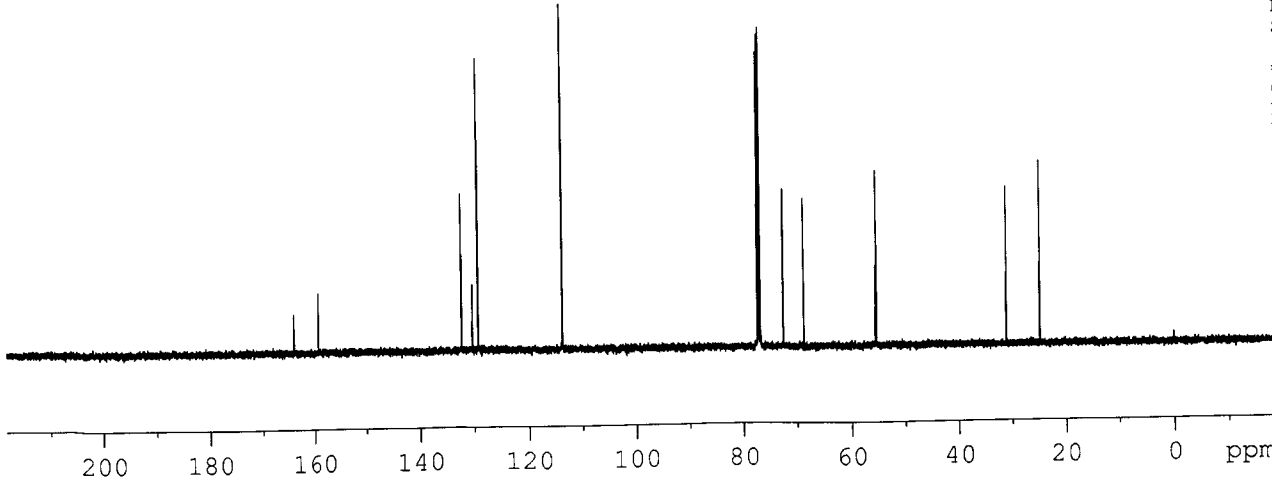

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01204-9

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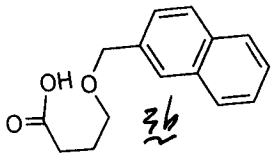

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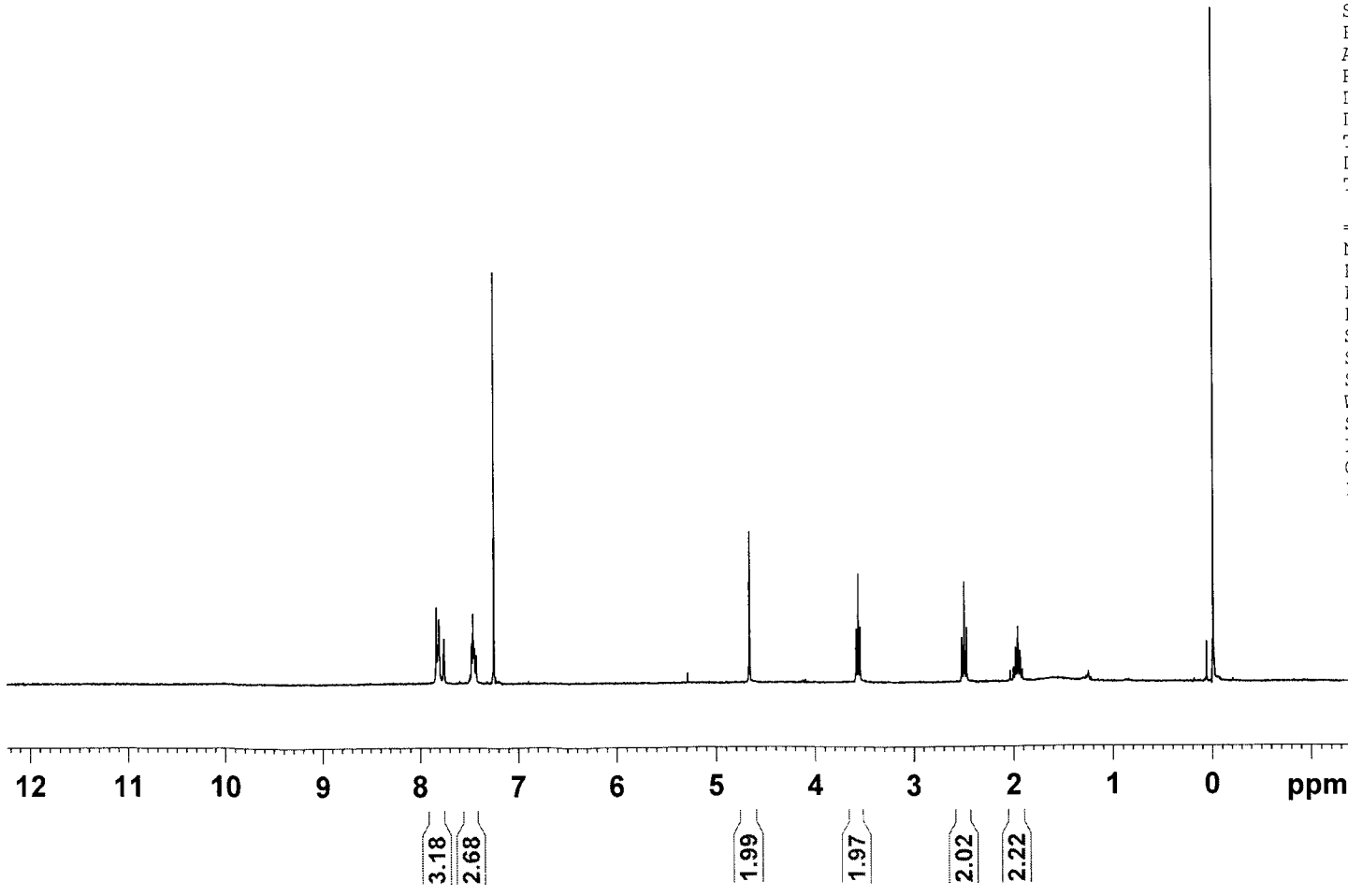

01204

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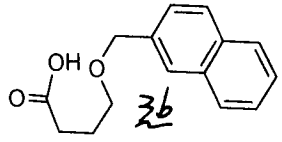

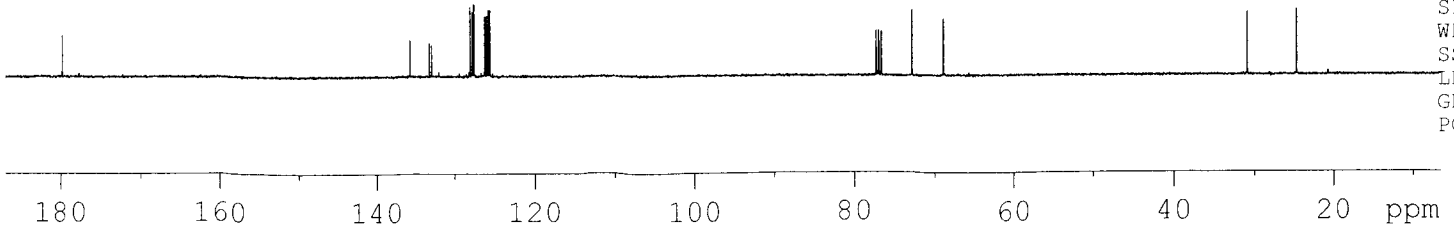

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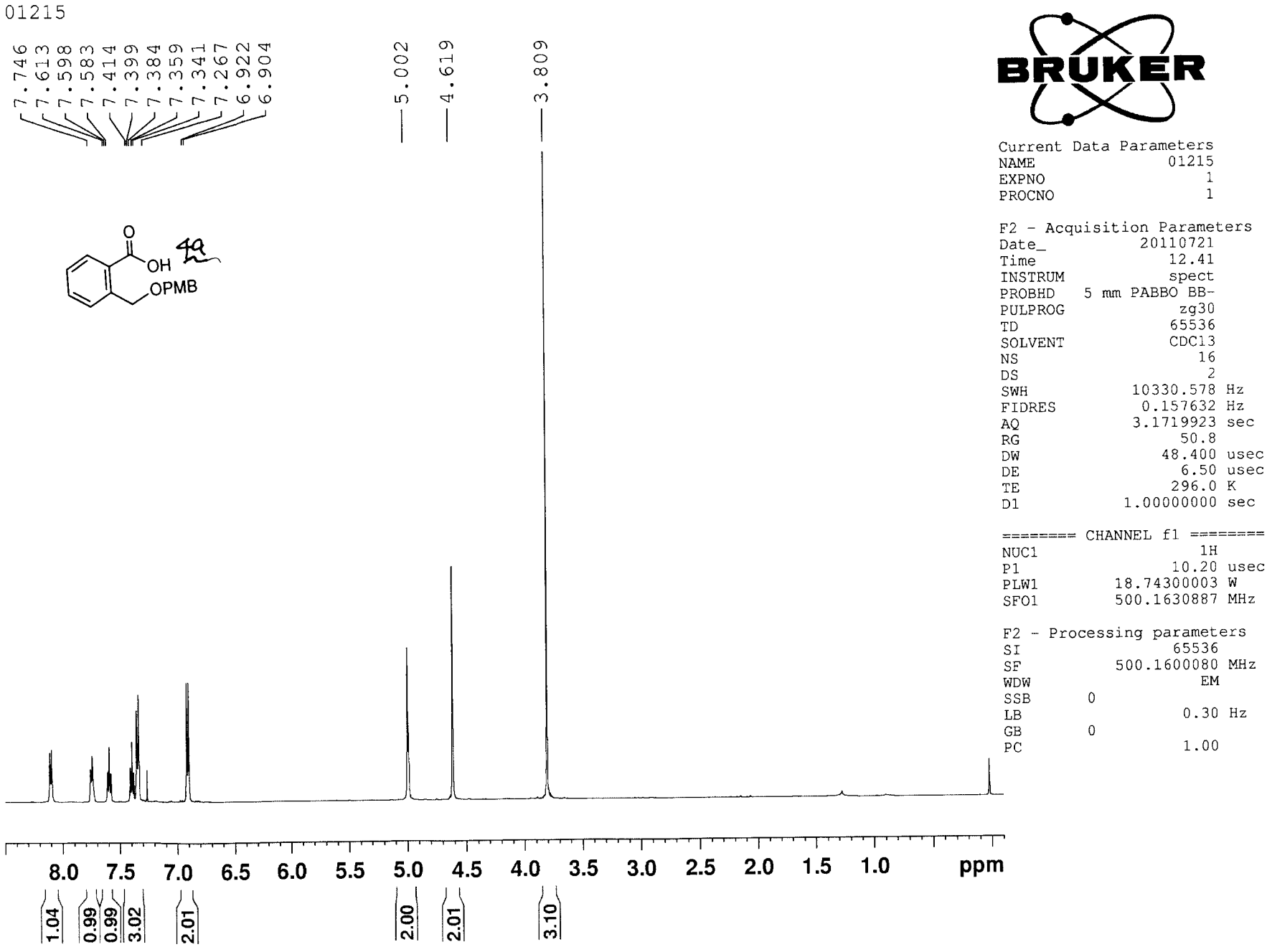

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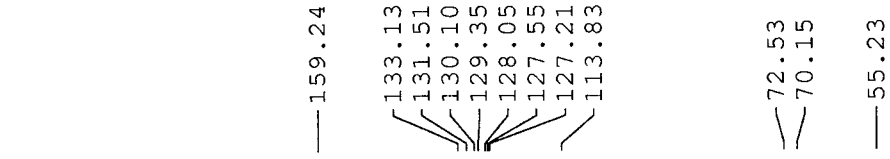

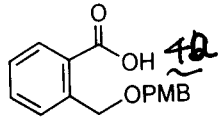

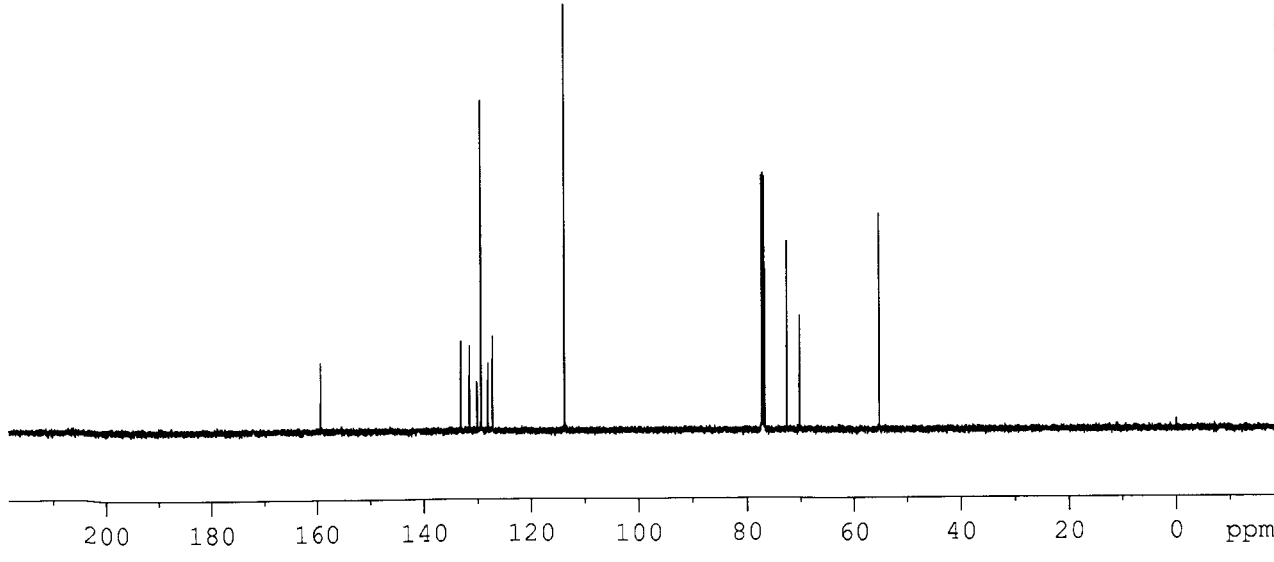

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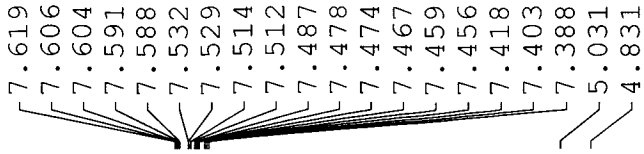
Date_              20110921
Time                  11.10
INSTRUM               spect
PROBHD    5 mm PABBO BB-
PULPROG              zgpg30
TD                    65536
SOLVENT               CDCl3
NS                      133
DS                        4
SWH               29761.904 Hz
FIDRES             0.454131 Hz
AQ                 1.1010548 sec
RG                      203
DW                   16.800 usec
DE                    6.50 usec
TE                   296.5 K
D1                2.00000000 sec
D11               0.03000000 sec

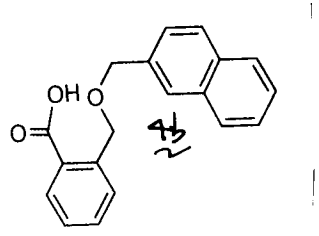
======== CHANNEL f1 ========
NUC1                    13C
P1                    8.80 usec
PLW1          112.58000183 W
SFO1           125.7779080 MHz

======== CHANNEL f2 ========
CPDPRG2            waltz16
NUC2                    1H
PCPD2                80.00 usec
PLW2          18.74300003 W
PLW12          0.36736000 W
PLW13          0.18408000 W
SFO2           500.1620006 MHz

F2 - Processing parameters
SI                      32768
SF             125.7653396 MHz
WDW                        EM
SSB       0
LB                     1.00 Hz
GB        0
PC                     1.40
```

01206





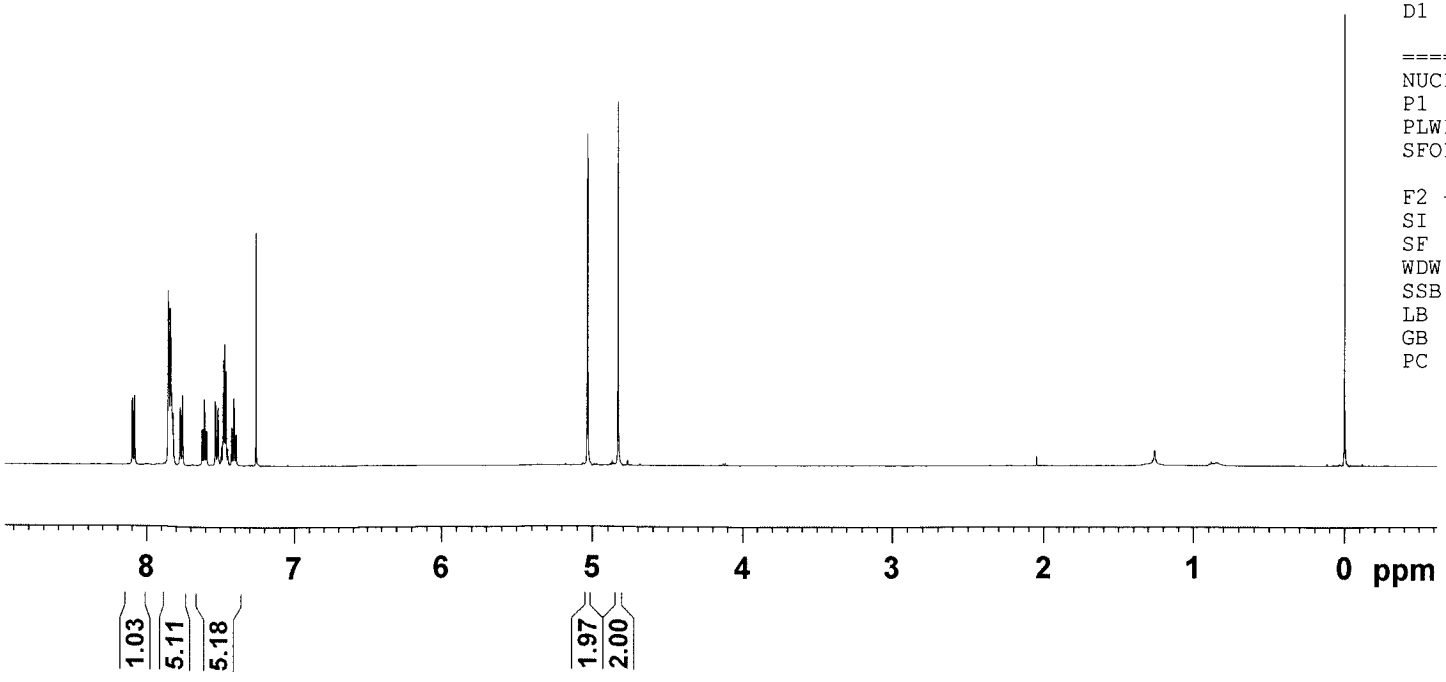
BRUKER

```
Current Data Parameters
NAME                  01206
EXPNO                     1
PROCNO                    1

F2 - Acquisition Parameters
Date_              20110711
Time                  19.36
INSTRUM               spect
PROBHD   5 mm PABBO BB-
PULPROG                zg30
TD                    65536
SOLVENT               CDCl3
NS                       16
DS                        2
SWH              10330.578 Hz
FIDRES            0.157632 Hz
AQ                3.1719923 sec
RG                      161
DW                 48.400 usec
DE                  6.50 usec
TE                  294.2 K
D1              1.00000000 sec

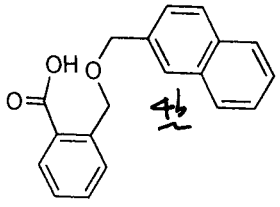
======== CHANNEL f1 ========
NUC1                    1H
P1                  10.20 usec
PLW1          18.74300003 W
SFO1          500.1630887 MHz

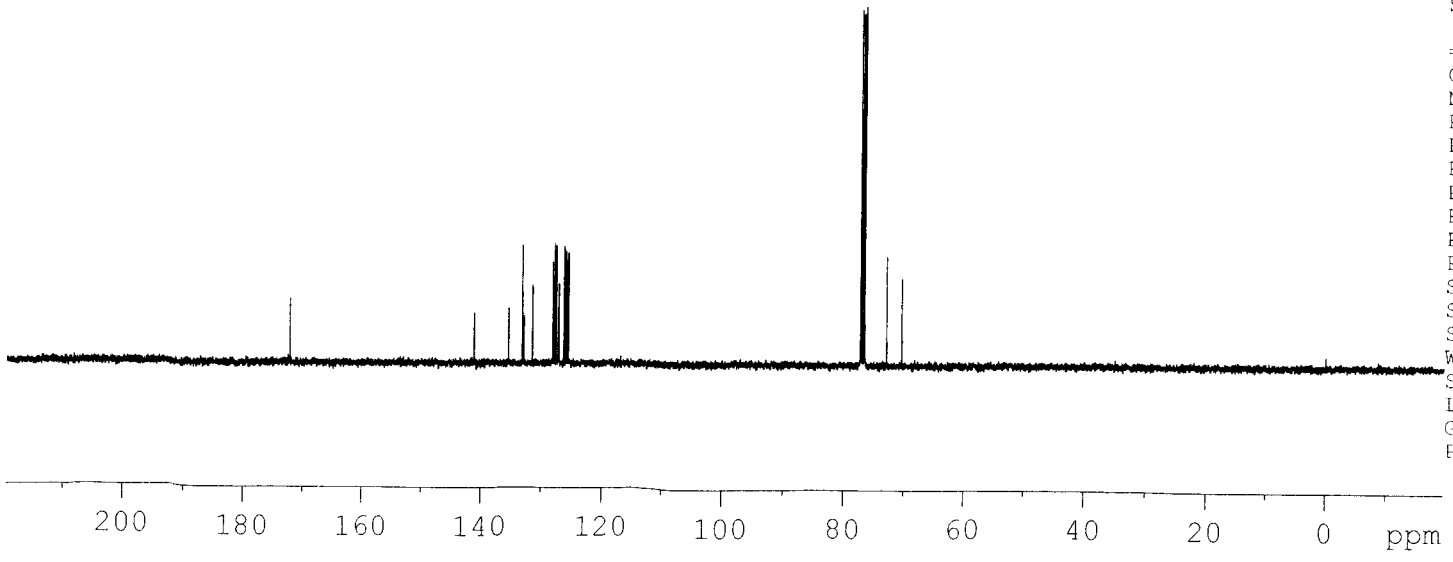
F2 - Processing parameters
SI                    65536
SF            500.1600143 MHz
WDW                     EM
SSB       0
LB                  0.30 Hz
GB        0
PC                  1.00
```



01206

172.23
141.17
135.51
133.28
133.00
131.58
128.20
128.00
127.90
127.68
127.35
127.27
126.40
126.08
125.86
125.68
72.97
70.45



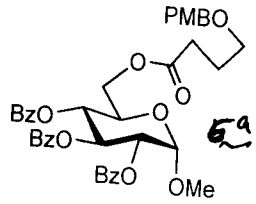


BRUKER

NAME	01206
EXPNO	1
PROCNO	1
Date_	20111017
Time	18.53
INSTRUM	spect
PROBHD	5 mm PABBO BB-
PULPROG	zgpg30
TD	65536
SOLVENT	CDCl3
NS	80
DS	4
SWH	24038.461 Hz
FIDRES	0.366798 Hz
AQ	1.3631988 sec
RG	203
DW	20.800 usec
DE	6.50 usec
TE	297.0 K
D1	3.00000000 sec
D11	0.03000000 sec
TD0	1
======== CHANNEL f1 ========	
NUC1	13C
P1	10.00 usec
PL1	-1.59 dB
PL1W	51.07626343 W
SFO1	100.6479773 MHz
======== CHANNEL f2 ========	
CPDPRG2	waltz16
NUC2	1H
PCPD2	75.00 usec
PL2	-1.00 dB
PL12	13.39 dB
PL13	20.00 dB
PL2W	11.09959412 W
PL12W	0.40393090 W
PL13W	0.08816721 W
SFO2	400.2316009 MHz
SI	32768
SF	100.6379199 MHz
WDW	EM
SSB	0
LB	1.00 Hz
GB	0
PC	1.40

01225p

7.990 7.976 7.973 7.942 7.927 7.925 7.875 7.861 7.858 7.473 7.469 7.348 7.337 7.249 6.883 6.196 6.176 6.157 5.618 5.315 5.308 5.295 5.287 5.251 5.244 4.423 4.333 4.323 4.294 4.289 3.764 3.474 3.461 3.456 2.494 2.488 2.479 2.473 2.463 2.459 1.952 1.950 1.937 1.924



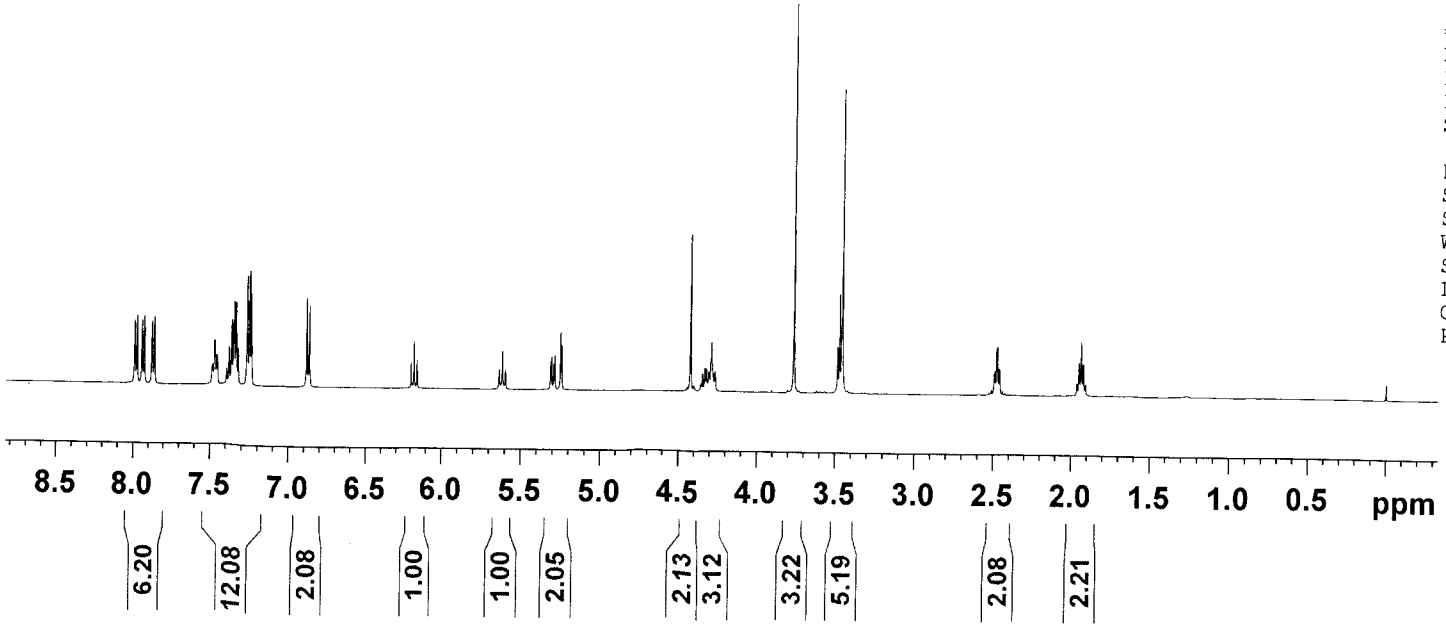
5g

Current Data Parameters
NAME 01225p
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20111019
Time 11.46
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 12
DS 2
SWH 10330.578 Hz
FIDRES 0.157632 Hz
AQ 3.1719923 sec
RG 16
DW 48.400 usec
DE 6.50 usec
TE 297.3 K
D1 1.00000000 sec

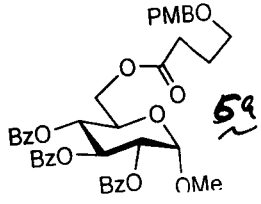
======== CHANNEL f1 ========
NUC1 1H
P1 10.20 usec
PLW1 18.74300003 W
SFO1 500.1630887 MHz

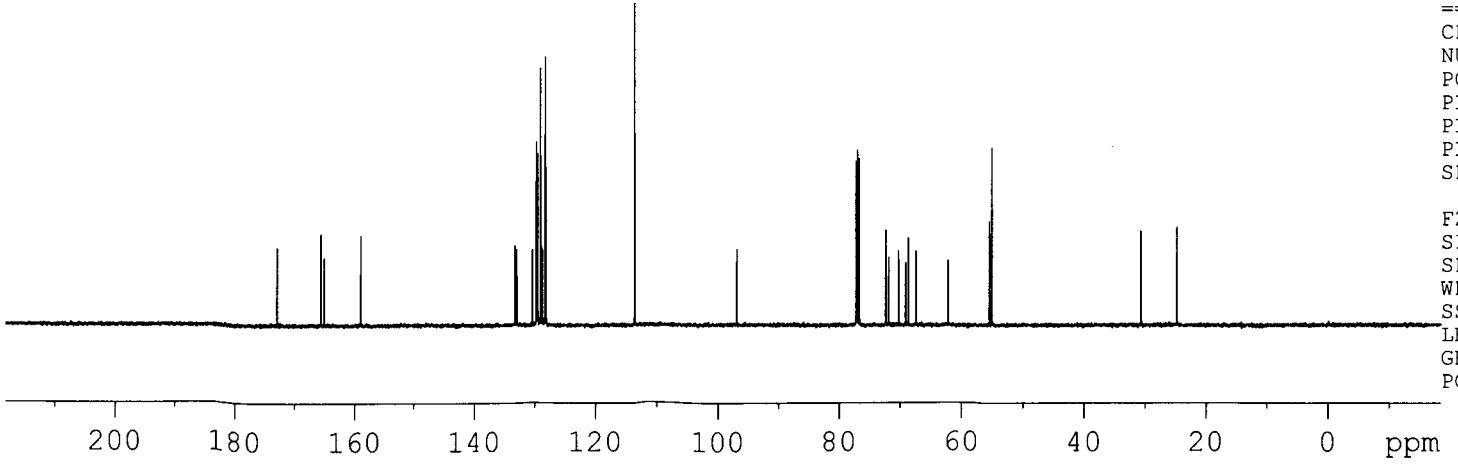
F2 - Processing parameters
SI 65536
SF 500.1600209 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00



01225p

172.91 165.63 165.60 165.07 159.00 133.28 133.25 132.97 130.38 129.76 129.65 129.49 129.06 129.01 128.86 128.74 128.28 128.13 113.62 96.90 77.20 72.37 71.86 70.25 69.12 68.67 67.41 62.18 55.47 55.06 30.71 24.82





BRUKER

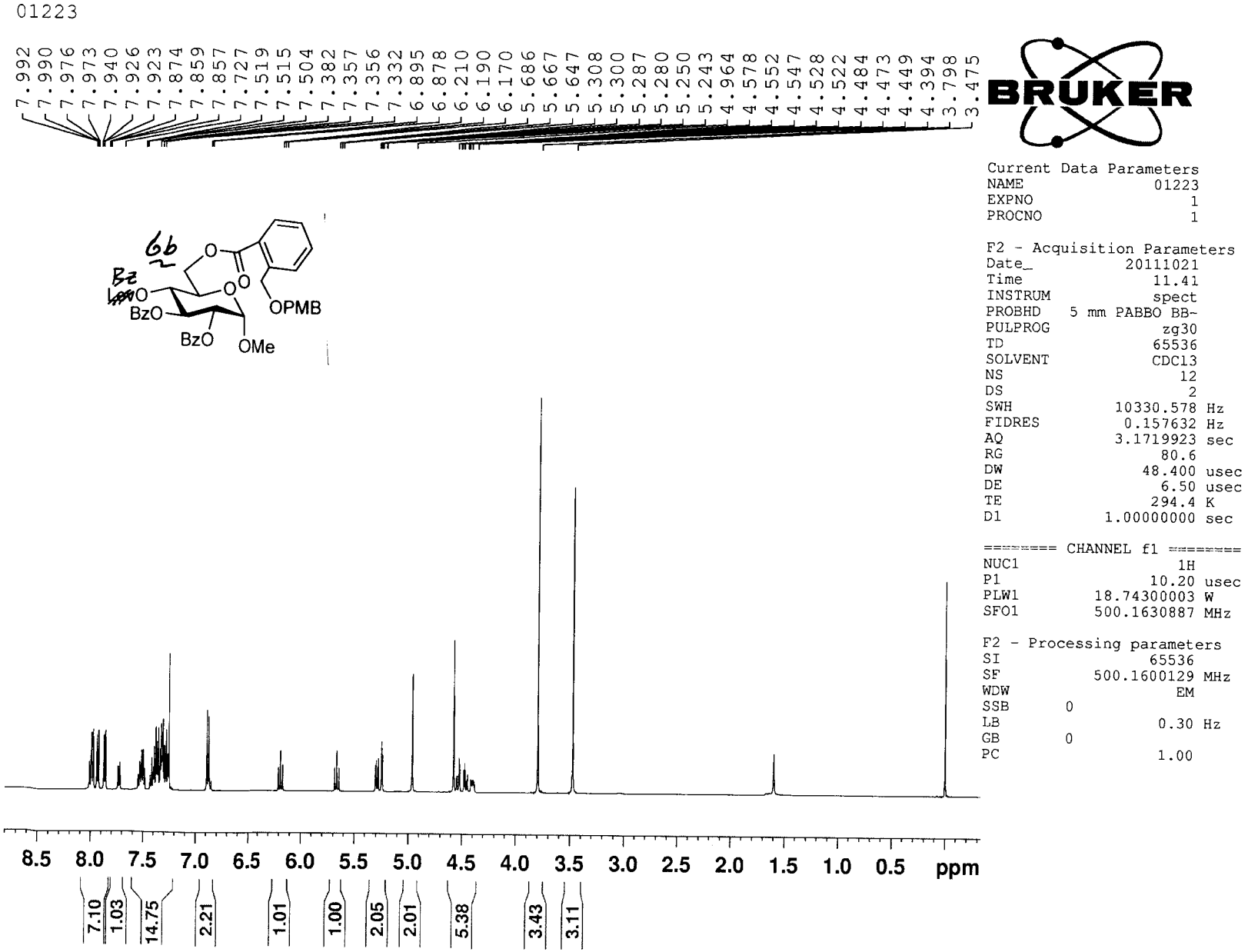
```
Current Data Parameters
NAME                  01225p
EXPNO                      2
PROCNO                     1

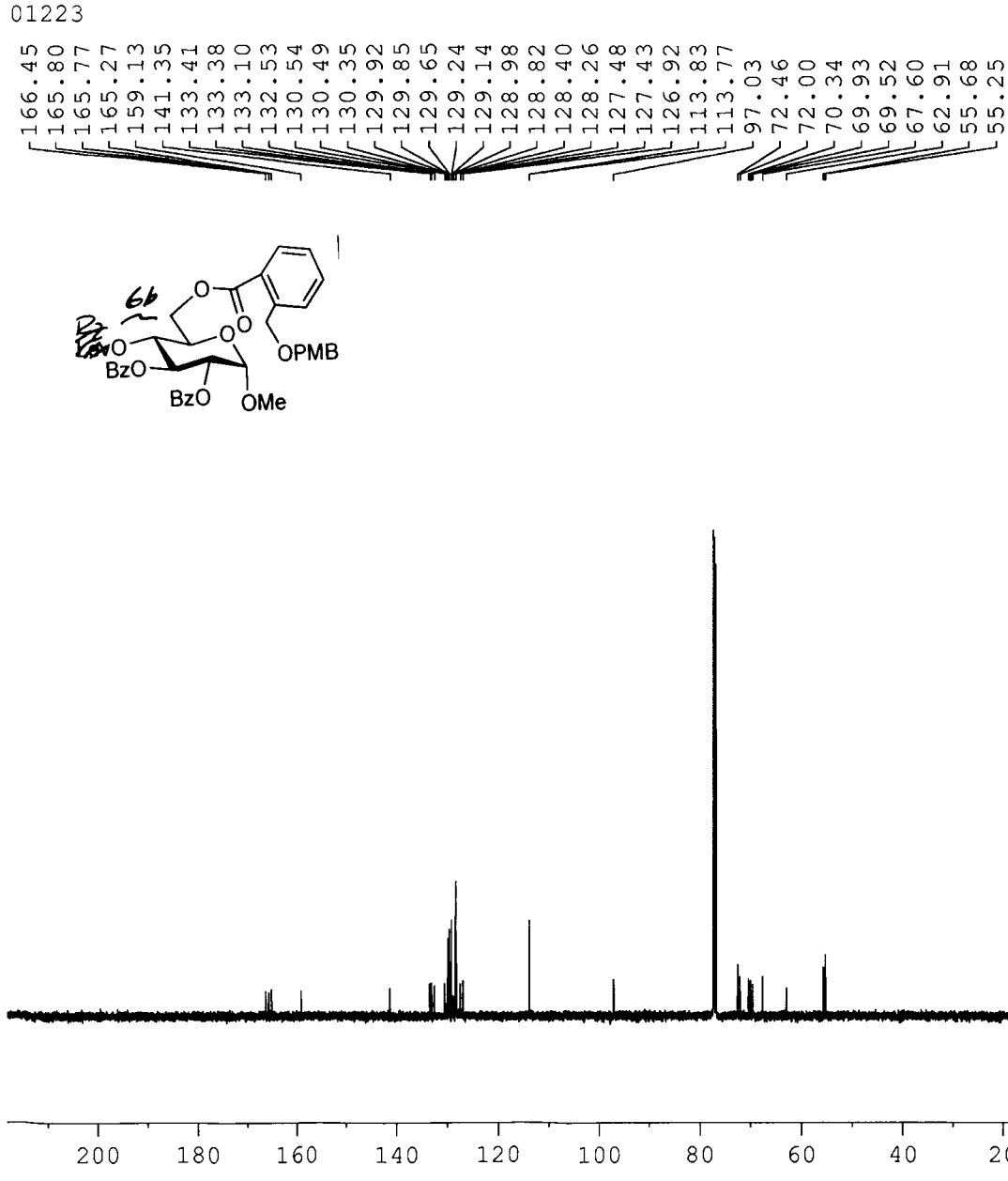
F2 - Acquisition Parameters
Date_              20111019
Time                  11.54
INSTRUM               spect
PROBHD   5 mm PABBO BB-
PULPROG              zgpg30
TD                    65536
SOLVENT               CDCl3
NS                       74
DS                        4
SWH               29761.904 Hz
FIDRES             0.454131 Hz
AQ                 1.1010548 sec
RG                      203
DW                   16.800 usec
DE                    6.50 usec
TE                   297.5 K
D1                2.00000000 sec
D11               0.03000000 sec

======== CHANNEL f1 ========
NUC1                    13C
P1                    8.80 usec
PLW1          112.58000183 W
SFO1           125.7779080 MHz

======== CHANNEL f2 ========
CPDPRG2           waltz16
NUC2                    1H
PCPD2                80.00 usec
PLW2          18.74300003 W
PLW12          0.36736000 W
PLW13     0 W
SFO2           500.1620006 MHz

F2 - Processing parameters
SI                      32768
SF             125.7653562 MHz
WDW                        EM
SSB       0
LB                    1.00 Hz
GB        0
PC                    1.40
```


01223
7.992
7.990
7.976
7.973
7.940
7.926
7.923
7.874
7.859
7.857
7.727
7.519
7.515
7.504
7.382
7.357
7.356
7.332
6.895
6.878
6.210
6.190
6.170
5.686
5.667
5.647
5.308
5.300
5.287
5.280
5.250
5.243
4.964
4.578
4.552
4.547
4.528
4.522
4.484
4.473
4.449
4.394
3.798
3.475
BRUKER
6b
Bz
BzO
BzO
OMe
OPMB
Current Data Parameters
NAME 01223
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20111021
Time 11.41
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 12
DS 2
SWH 10330.578 Hz
FIDRES 0.157632 Hz
AQ 3.1719923 sec
RG 80.6
DW 48.400 usec
DE 6.50 usec
TE 294.4 K
D1 1.00000000 sec
======== CHANNEL f1 ========
NUC1 1H
P1 10.20 usec
PLW1 18.74300003 W
SFO1 500.1630887 MHz
F2 - Processing parameters
SI 65536
SF 500.1600129 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
8.5 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
7.10
1.03
14.75
2.21
1.01
1.00
2.05
2.01
5.38
3.43
3.11




```
Current Data Parameters
NAME                  01223
EXPNO                     3
PROCNO                    1

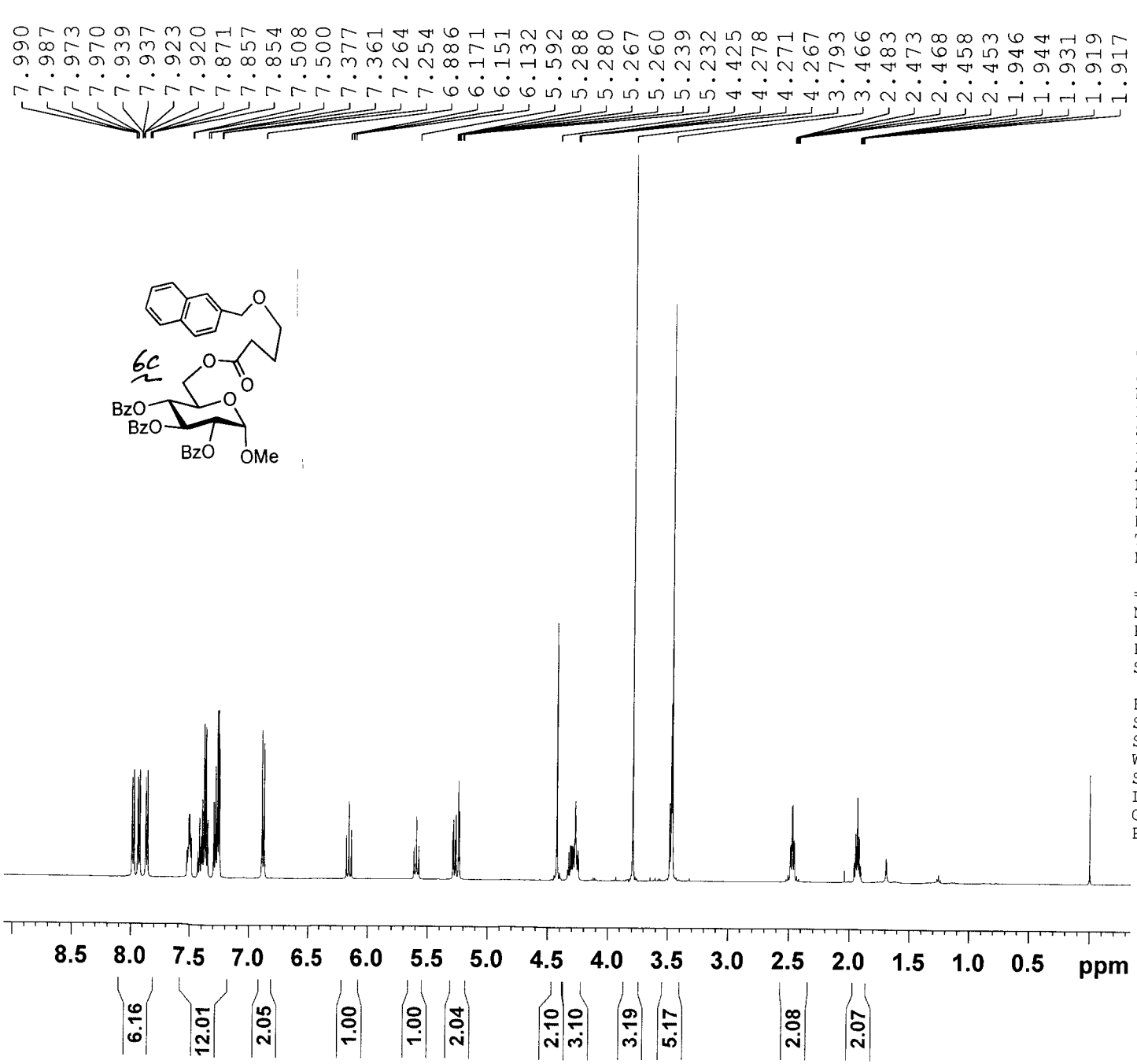
F2 - Acquisition Parameters
Date_              20110728
Time                  12.57
INSTRUM               spect
PROBHD   5 mm PABBO BB-
PULPROG              zgpg30
TD                    65536
SOLVENT               CDCl3
NS                      131
DS                        4
SWH              29761.904 Hz
FIDRES            0.454131 Hz
AQ                1.1010548 sec
RG                      203
DW                  16.800 usec
DE                   6.50 usec
TE                   294.8 K
D1               2.00000000 sec
D11              0.03000000 sec

======== CHANNEL f1 ========
NUC1                    13C
P1                   8.80 usec
PLW1          112.58000183 W
SFO1           125.7779080 MHz

======== CHANNEL f2 ========
CPDPRG2            waltz16
NUC2                    1H
PCPD2                80.00 usec
PLW2           18.74300003 W
PLW12           0.36736000 W
PLW13           0.18408000 W
SFO2           500.1620006 MHz

F2 - Processing parameters
SI                    32768
SF             125.7653370 MHz
WDW                      EM
SSB       0
LB                    1.00 Hz
GB        0
PC                    1.40
```

01224p




```
Current Data Parameters
NAME                  01224p
EXPNO                      2
PROCNO                     1

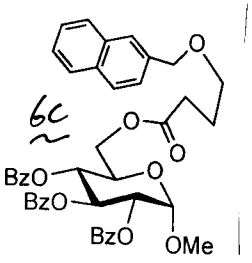
F2 - Acquisition Parameters
Date_              20110728
Time                  11.24
INSTRUM               spect
PROBHD    5 mm PABBO BB-
PULPROG                zg30
TD                    65536
SOLVENT               CDCl3
NS                        8
DS                        2
SWH              10330.578 Hz
FIDRES            0.157632 Hz
AQ                3.1719923 sec
RG                       32
DW                  48.400 usec
DE                   6.50 usec
TE                  294.5 K
D1               1.00000000 sec

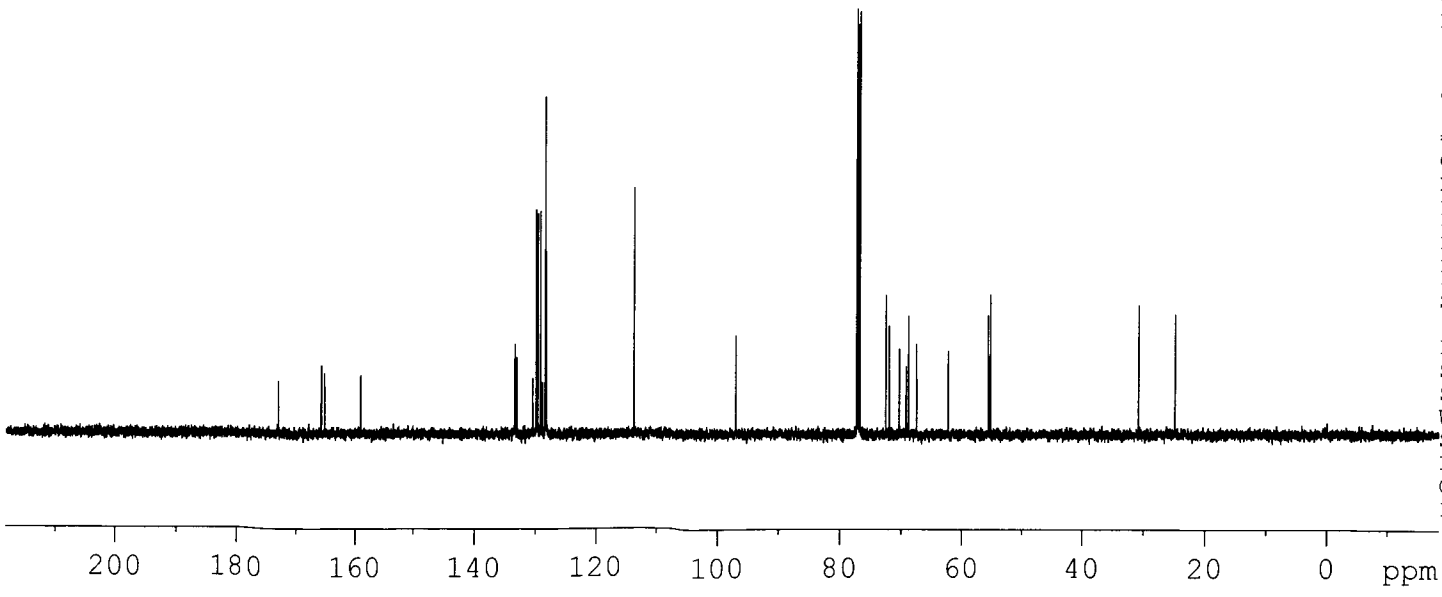
======== CHANNEL f1 ========
NUC1                     1H
P1                   10.20 usec
PLW1          18.74300003 W
SFO1          500.1630887 MHz

F2 - Processing parameters
SI                    65536
SF            500.1600143 MHz
WDW                      EM
SSB       0
LB                    0.30 Hz
GB        0
PC                    1.00
```

01224p

173.06
165.76
165.71
165.18
159.09
133.38
133.35
133.07
130.46
129.88
129.77
129.61
129.18
129.11
128.95
128.82
128.38
128.23
113.71
96.98
72.49
71.95
70.29
69.17
68.78
67.48
62.28
55.60
55.21
30.80
24.89





Current Data Parameters
NAME 01224p
EXPNO 3
PROCNO 1

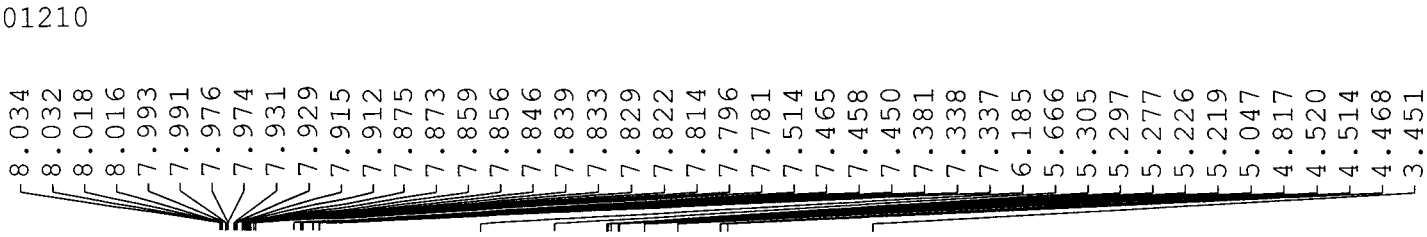
F2 - Acquisition Parameters
Date_ 20110728
Time 11.30
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 44
DS 4
SWH 29761.904 Hz
FIDRES 0.454131 Hz
AQ 1.1010548 sec
RG 203
DW 16.800 usec
DE 6.50 usec
TE 294.8 K
D1 2.00000000 sec
D11 0.03000000 sec

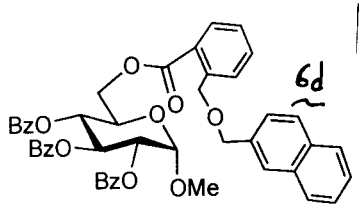
======== CHANNEL f1 ========
NUC1 13C
P1 8.80 usec
PLW1 112.58000183 W
SFO1 125.7779080 MHz

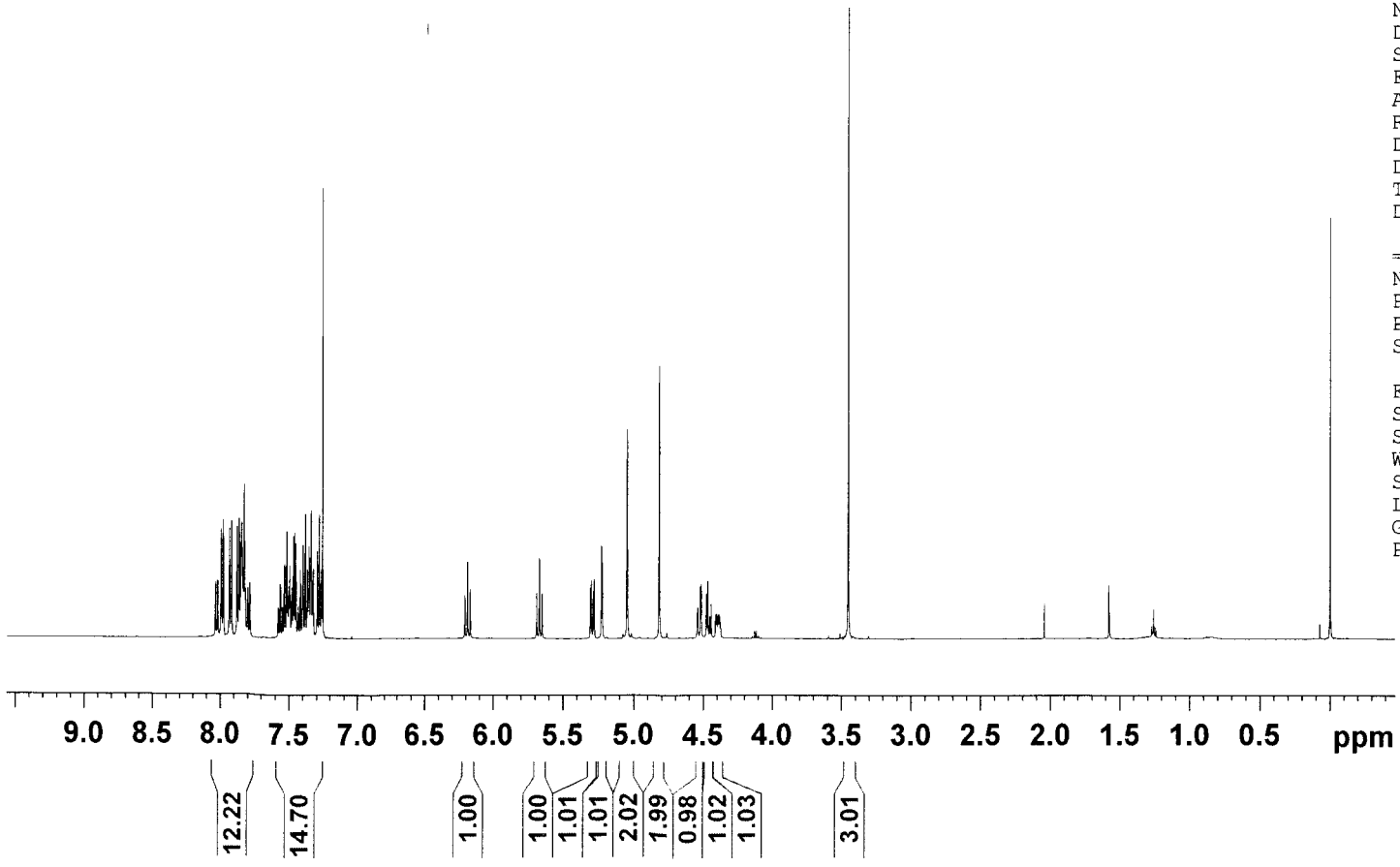
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PLW2 18.74300003 W
PLW12 0.36736000 W
PLW13 0.18408000 W
SFO2 500.1620006 MHz

F2 - Processing parameters
SI 32768
SF 125.7653413 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

01210







Current Data Parameters
NAME 01210
EXPNO 1
PROCNO 1

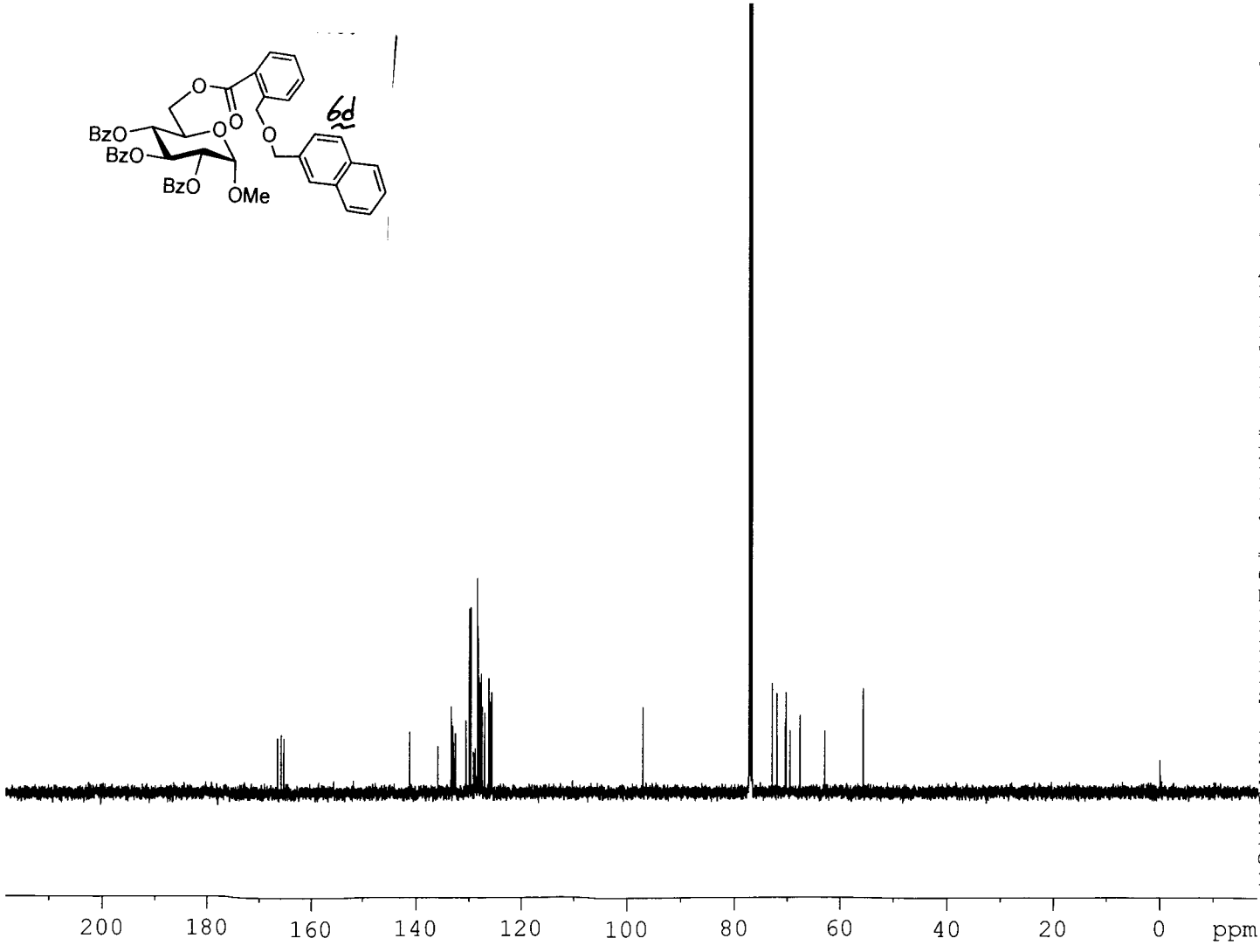
F2 - Acquisition Parameters
Date_ 20110713
Time 16.50
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 12
DS 2
SWH 10330.578 Hz
FIDRES 0.157632 Hz
AQ 3.1719923 sec
RG 80.6
DW 48.400 usec
DE 6.50 usec
TE 294.2 K
D1 1.00000000 sec

======== CHANNEL f1 ========
NUC1 1H
P1 10.20 usec
PLW1 18.74300003 W
SFO1 500.1630887 MHz

F2 - Processing parameters
SI 65536
SF 500.1600150 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

01210

166.46 165.80 165.77 165.27 141.21 135.91 133.40 133.31 133.09 132.96 132.58 130.60 129.93 129.85 129.65 129.16 129.01 128.83 128.41 128.27 128.12 127.91 127.67 127.52 127.02 126.25 126.02 125.76 125.70 97.04 72.88 71.99 70.35 70.26 69.51 67.59 62.91 55.67



BRUKER

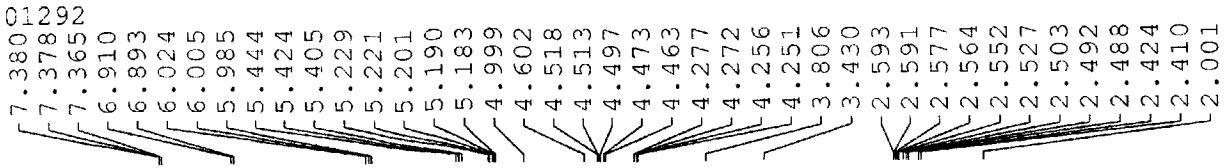
Current Data Parameters
NAME 01210
EXPNO 2
PROCNO 1

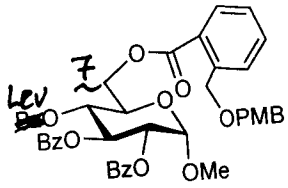
F2 - Acquisition Parameters
Date_ 20110713
Time 17.09
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 245
DS 4
SWH 29761.904 Hz
FIDRES 0.454131 Hz
AQ 1.1010548 sec
RG 203
DW 16.800 usec
DE 6.50 usec
TE 294.8 K
D1 2.00000000 sec
D11 0.03000000 sec

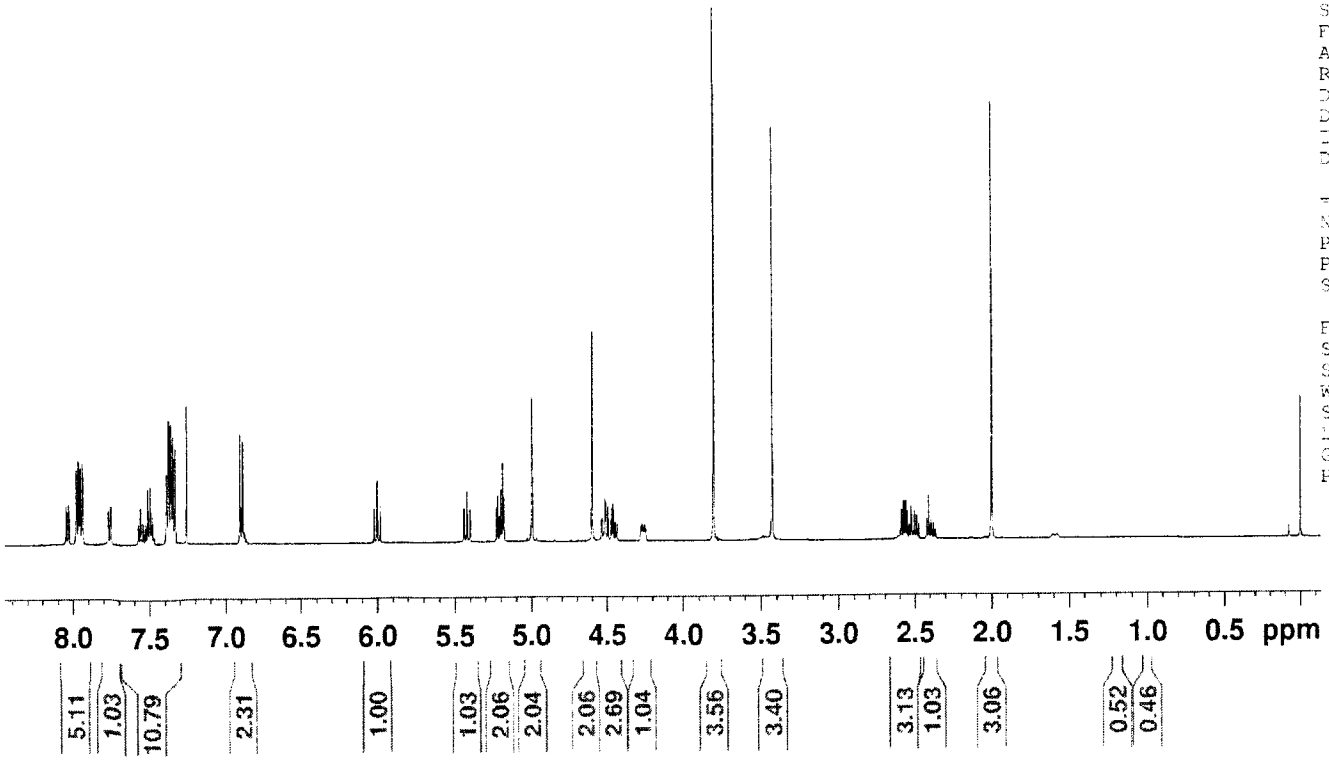
======== CHANNEL f1 ========
NUC1 13C
P1 8.80 usec
PLW1 112.58000183 W
SFO1 125.7779080 MHz

======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PLW2 18.74300003 W
PLW12 0.36736000 W
PLW13 0.18408000 W
SFO2 500.1620006 MHz

F2 - Processing parameters
SI 32768
SF 125.7653364 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40





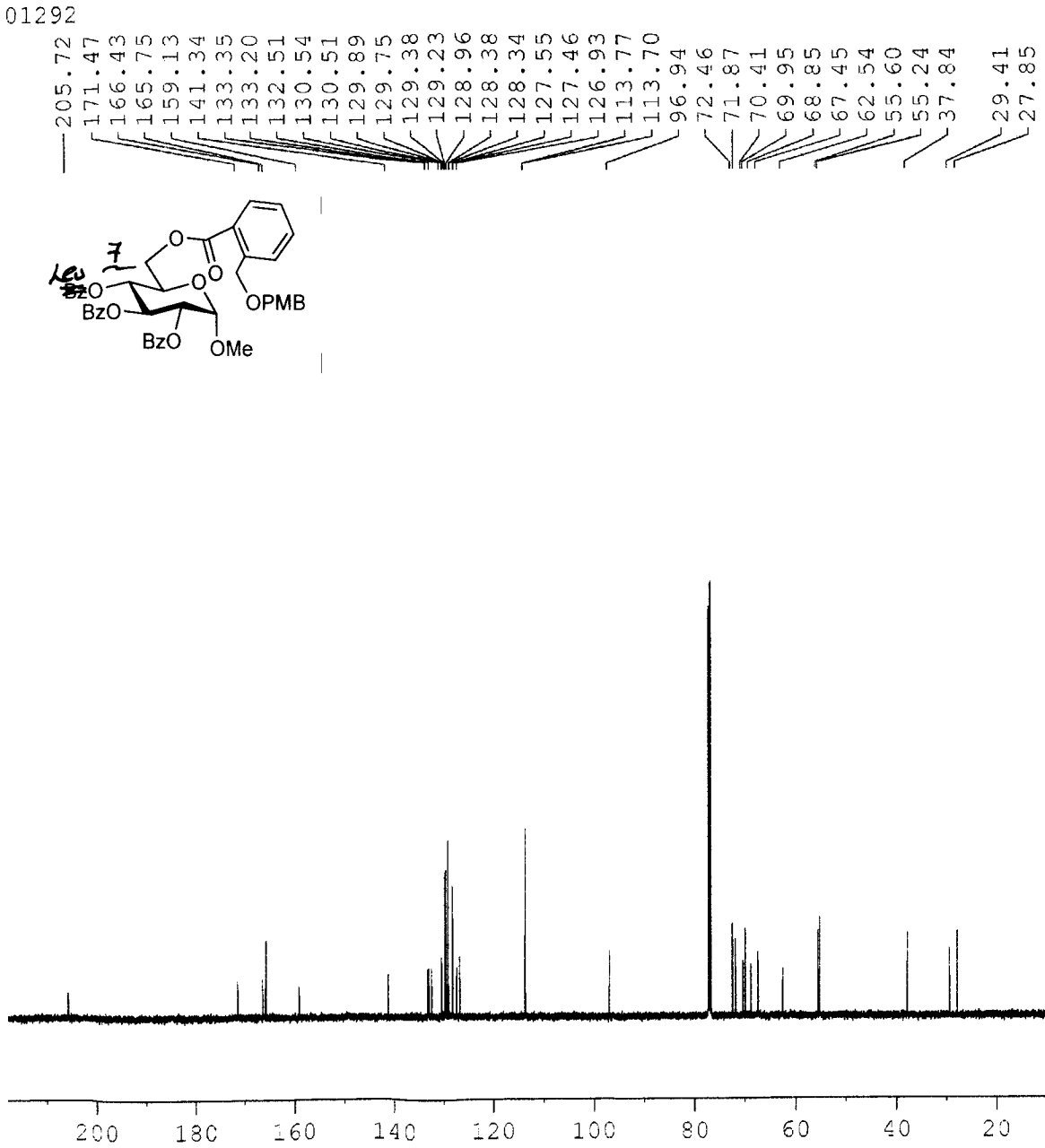


Current Data Parameters
NAME 01292
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20111015
Time 11.13
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 10330.578 Hz
FIDRES 0.157637 Hz
AQ 3.1719923 sec
RG 50.8
DW 48.400 usec
DE 6.50 usec
TE 293.7 K
D1 1.00000000 sec

======== CHANNEL f1 ========
NUC1 1H
P1 10.20 usec
PLW1 18.74300003 W
SFO1 500.1630887 MHz

F2 - Processing parameters
SI 65536
SF 500.1600118 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00



BRUKER

Current Data Parameters
NAME 01292
EXPNO 2
PROCNO 1

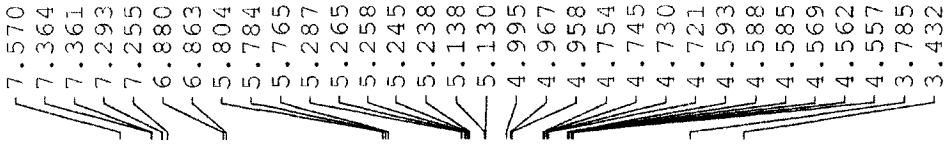
F2 - Acquisition Parameters
Date_ 20111016
Time 16.55
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 176
DS 4
SWH 29761.904 Hz
FIDRES 0.454131 Hz
AQ 1.1010548 sec
RG 203
DW 16.800 usec
DE 6.50 usec
TE 294.3 K
D1 2.00000000 sec
D11 0.03000000 sec

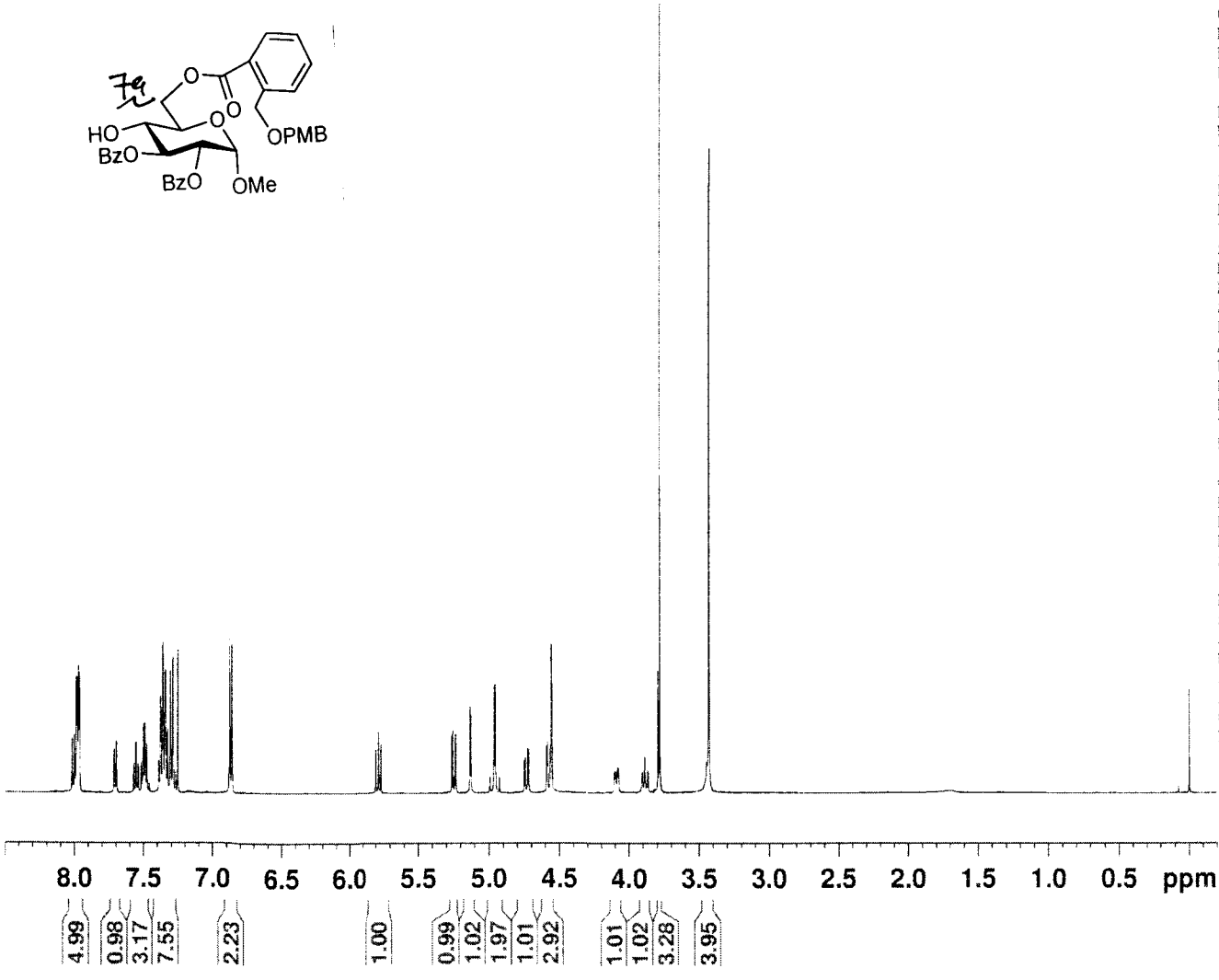
======== CHANNEL f1 ========
NUC1 13C
P1 8.80 usec
PLW1 112.58000183 W
SFO1 125.7779080 MHz

======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PLW2 18.74300003 W
PLW12 0.36736000 W
PLW13 0.18408000 W
SFO2 500.1620006 MHz

F2 - Processing parameters
SI 32768
SF 125.7653391 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

01290





Current Data Parameters
NAME 01290
EXPNO 1
PROCNO 1

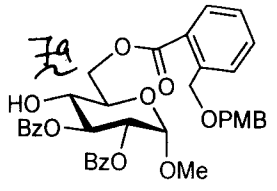
F2 - Acquisition Parameters
Date_ 20110930
Time 17.11
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 12
DS 2
SWH 10330.578 Hz
FIDRES 0.157632 Hz
AQ 3.1719923 sec
RG 32
DW 48.400 usec
DE 6.50 usec
TE 294.1 K
D1 1.00000000 sec

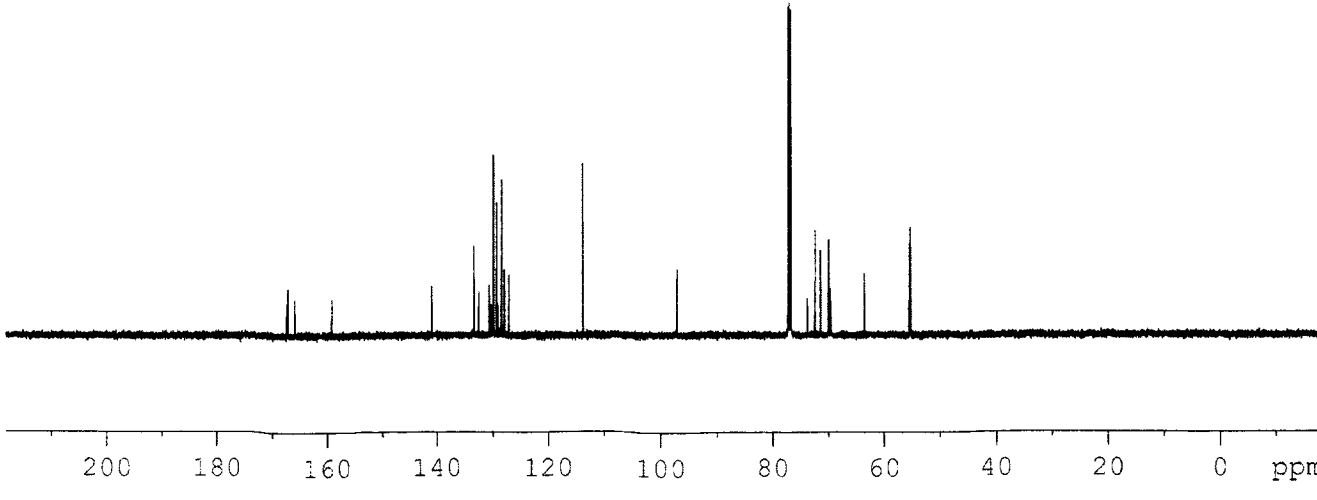
======== CHANNEL f1 ========
NUC1 1H
P1 10.20 usec
PLW1 18.74300003 W
SFO1 500.1630887 MHz

F2 - Processing parameters
SI 65536
SF 500.1600141 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

01290

167.40
167.22
165.92
159.17
140.95
133.35
132.52
130.61
130.28
129.85
129.83
129.38
129.30
129.19
129.08
128.38
128.36
127.95
127.91
127.11
113.79
113.71
97.10
73.78
72.55
72.39
71.40
70.01
69.91
69.73
69.39
63.45
55.45
55.23





BRUKER

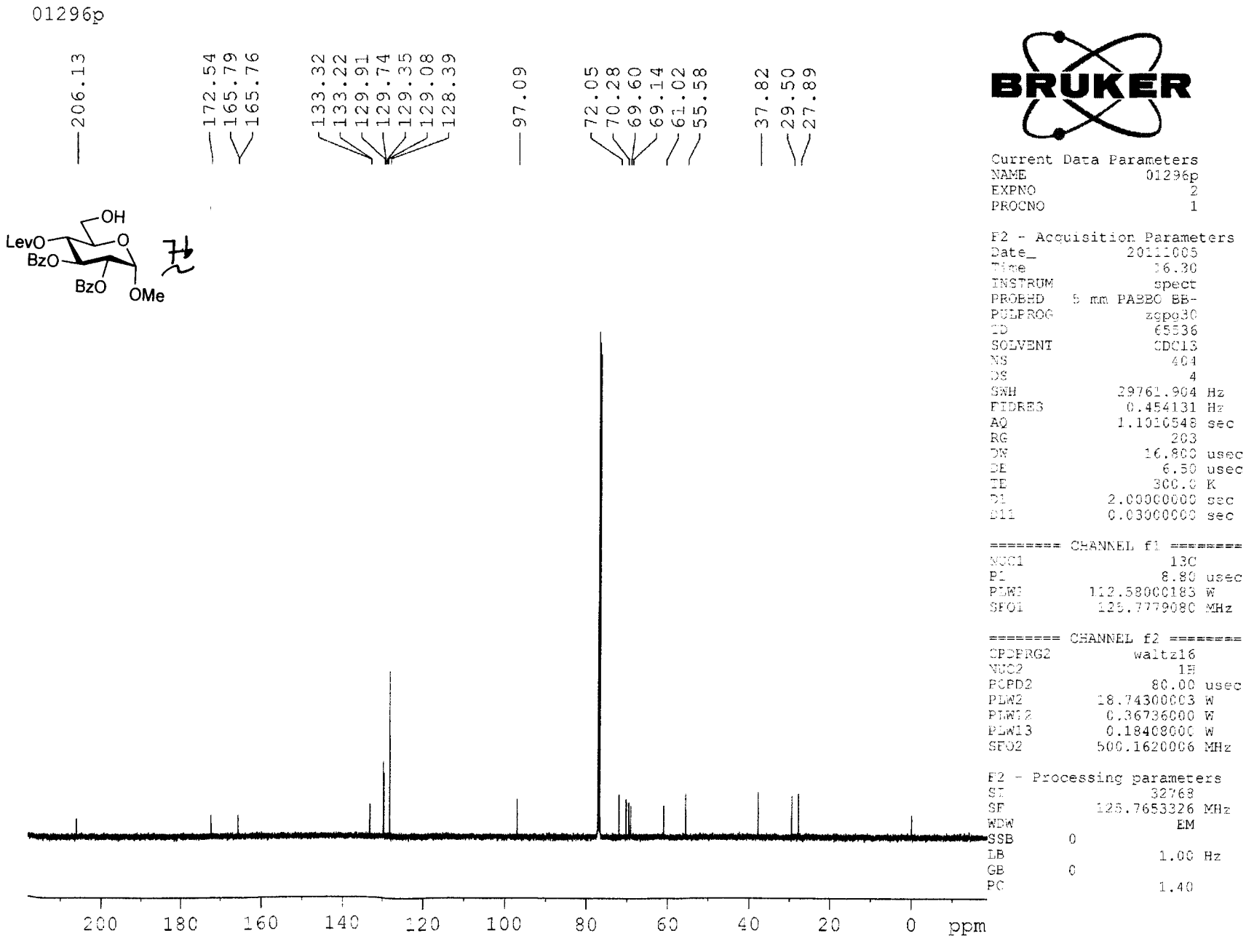
```
Current Data Parameters
NAME                  01290
EXPNO                     2
PROCNO                    1

F2 - Acquisition Parameters
Date_              20110930
Time                  17.55
INSTRUM               spect
PROBHD    5 mm PABBO BB-
PULPROG             zgpg30
TD                   65536
SOLVENT               CDCl3
NS                      156
DS                        4
SWH               29761.904 Hz
FIDRES             0.454131 Hz
AQ                 1.1010548 sec
RG                      203
DW                 16.800 usec
DE                  6.50 usec
TE                  295.2 K
D1              2.00000000 sec
D11             0.03000000 sec

======== CHANNEL f1 ========
NUC1                    13C
P1                   8.80 usec
PLW1         112.58000183 W
SFO1          125.7779080 MHz

======== CHANNEL f2 ========
CPDPRG2             waltz16
NUC2                    1H
PCPD2               80.00 usec
PLW2          18.74300003 W
PLW12          0.36736000 W
PLW13          0.18408000 W
SFO2          500.1620006 MHz

F2 - Processing parameters
SI                      32768
SF            125.7653389 MHz
WDW                        EM
SSB       0
LB                   1.00 Hz
GB        0
PC                   1.40
```



BRUKER

Current Data Parameters
NAME 01296p
EXPNO 2
PROCNO 1

F2 - Acquisition Parameters
Date_ 20111005
Time 16.30
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 404
DS 4
SWH 29761.904 Hz
FIDRES 0.454131 Hz
AQ 1.1010548 sec
RG 203
DW 16.800 usec
DE 6.50 usec
TE 300.0 K
D1 2.00000000 sec
D11 0.03000000 sec

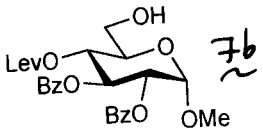
======== CHANNEL f1 ========
NUC1 13C
P1 8.80 usec
PLW1 112.58000183 W
SFO1 125.7779080 MHz

======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PLW2 18.74300003 W
PLW12 0.36736000 W
PLW13 0.18408000 W
SFO2 500.1620006 MHz

F2 - Processing parameters
SI 32768
SF 125.7653326 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

01196p

7.953 7.951 7.505 7.502 7.490 7.370 6.039 6.019 6.015 6.000 5.322 5.302 5.282 5.202 5.199 5.186 5.179 3.936 3.918 3.916 3.835 3.830 3.809 3.804 3.774 3.772 3.765 3.746 3.438 2.680 2.668 2.597 2.585 2.561 2.557 2.553 2.536 2.526 2.519 2.395 2.383 2.381 2.370 2.083



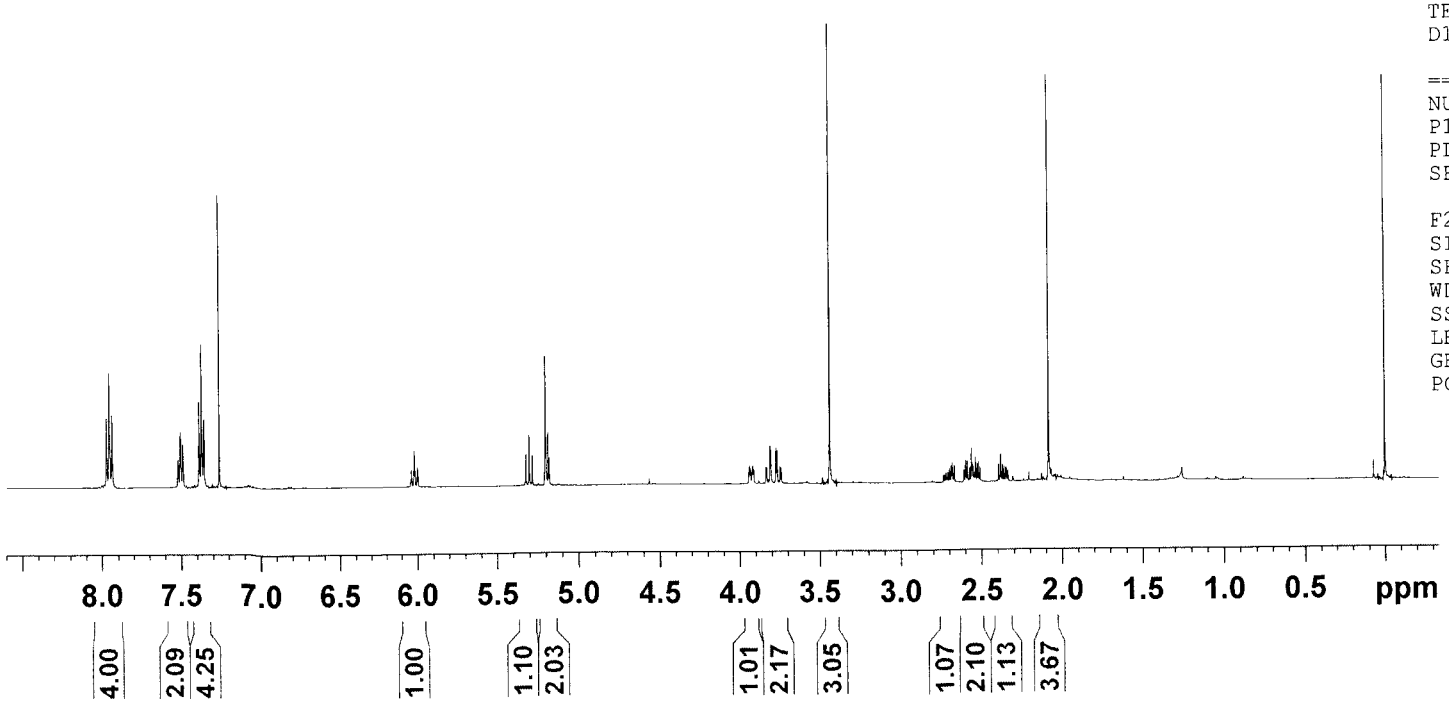
76

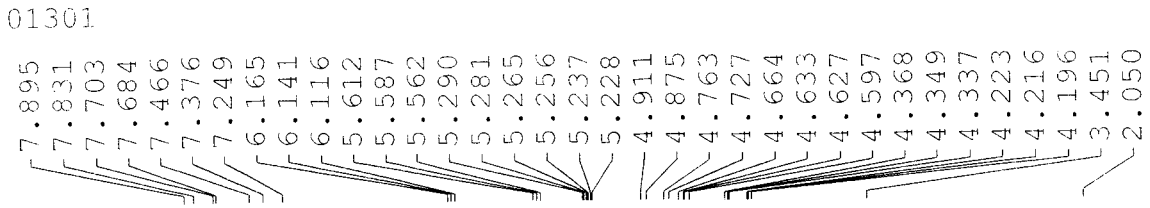
Current Data Parameters
NAME 01196p
EXPNO 1
PROCNO 1

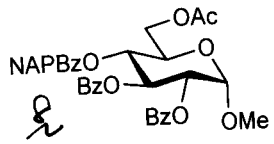
F2 - Acquisition Parameters
Date_ 20111005
Time 10.58
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 10330.578 Hz
FIDRES 0.157632 Hz
AQ 3.1719923 sec
RG 101
DW 48.400 usec
DE 6.50 usec
TE 300.0 K
D1 1.00000000 sec

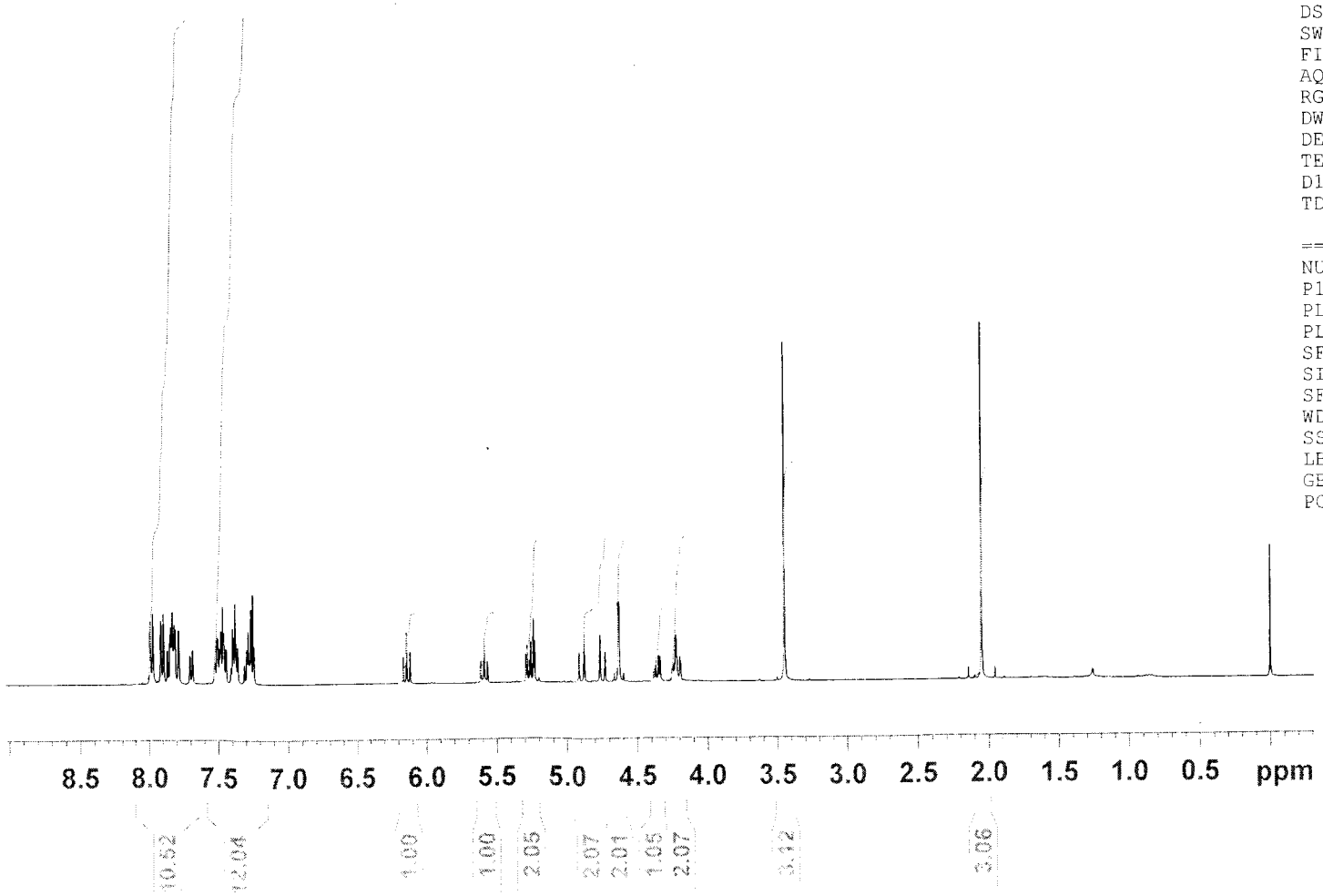
======== CHANNEL f1 ========
NUC1 1H
P1 10.20 usec
PLW1 18.74300003 W
SFO1 500.1630887 MHz

F2 - Processing parameters
SI 65536
SF 500.1600117 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00





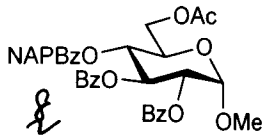


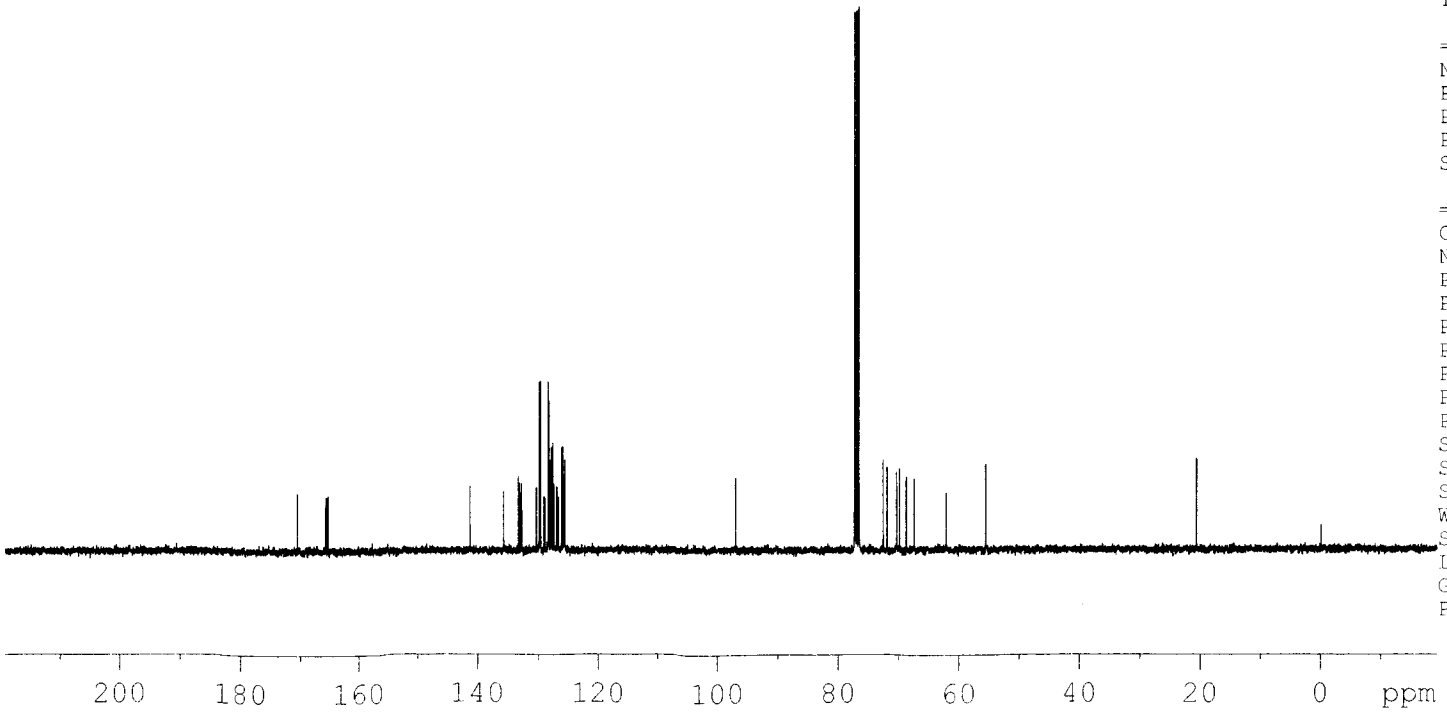


NAME	01301
EXPNO	1
PROCNO	1
Date_	20111012
Time_	17.06
INSTRUM	spect
PROBHD	5 mm PABBO BB-
PULPROG	zg30
TD	65536
SOLVENT	CDCl3
NS	16
DS	2
SWH	8223.685 Hz
FIDRES	0.125483 Hz
AQ	3.9846387 sec
RG	90.5
DW	60.800 usec
DE	6.50 usec
TE	296.1 K
D1	2.00000000 sec
TD0	1
======== CHANNEL f1 ========	
NUC1	1H
P1	14.31 usec
PL1	-1.00 dB
PL1W	11.09959412 W
SFO1	400.2324716 MHz
SI	32768
SF	400.2300169 MHz
WDW	EM
SSB	0
LB	0.30 Hz
GB	0
PC	1.00

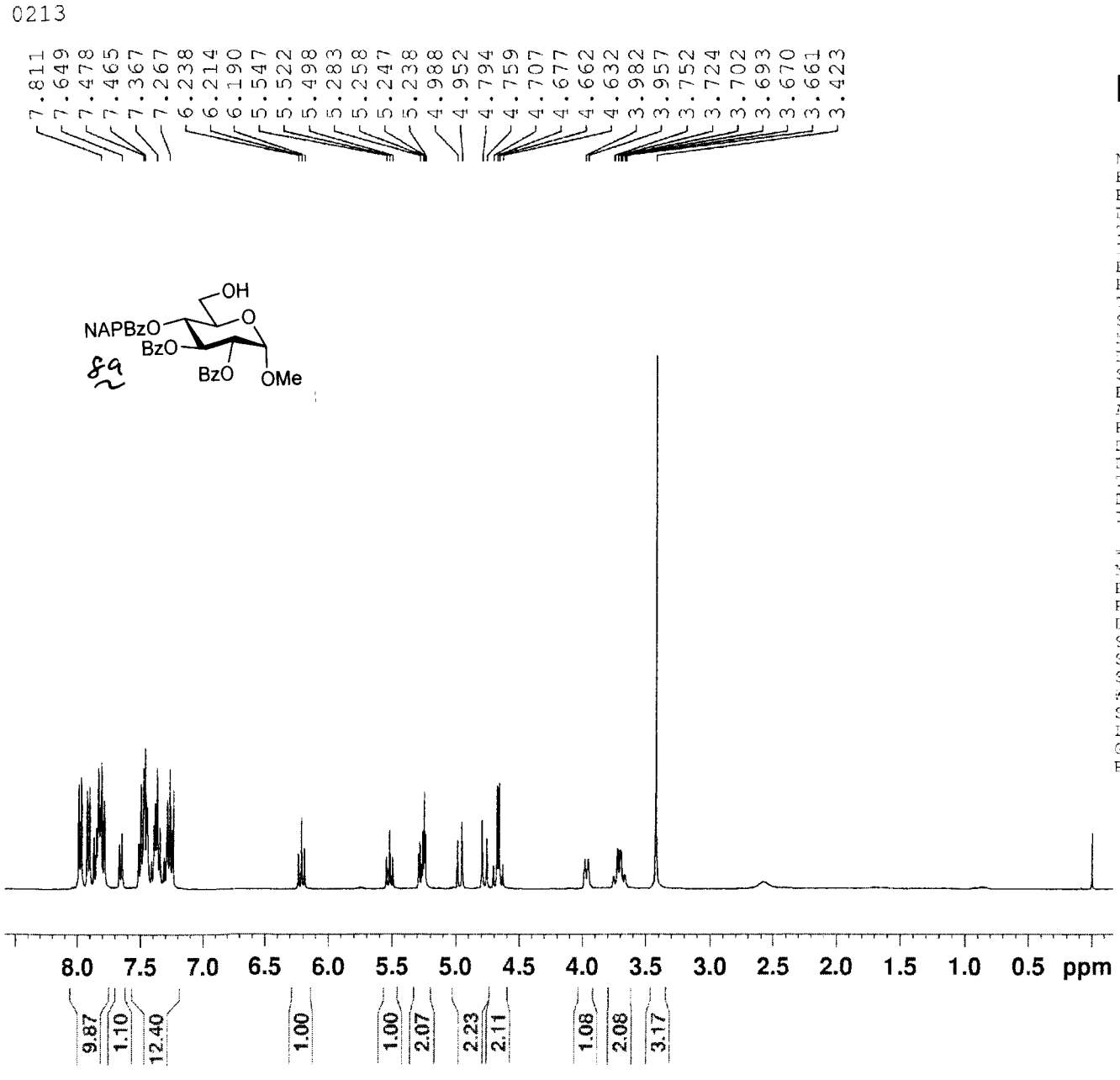
01301

170.56
165.80
165.69
165.39
141.27
135.80
133.39
133.27
133.19
132.93
132.82
130.40
129.91
129.71
129.11
128.96
128.41
128.30
128.05
127.88
127.65
127.50
127.01
126.76
126.21
126.01
125.77
125.68
97.03
72.67
71.97
70.40
69.90
68.79
67.45
62.18
55.63
20.68





NAME	01301	
EXPNO	2	
PROCNO	1	
Date_	20111012	
Time	17.27	
INSTRUM	spect	
PROBHD	5 mm PABBO BB-	
PULPROG	zgpg30	
TD	65536	
SOLVENT	CDCl3	
NS	211	
DS	4	
SWH	24038.461	Hz
FIDRES	0.366798	Hz
AQ	1.3631988	sec
RG	203	
DW	20.800	usec
DE	6.50	usec
TE	296.6	K
D1	3.00000000	sec
D11	0.03000000	sec
TD0	1	
======== CHANNEL f1 ========		
NUC1	13C	
P1	10.00	usec
PL1	-1.59	dB
PL1W	51.07626343	W
SFO1	100.6479773	MHz
======== CHANNEL f2 ========		
CPDPRG2	waltz16	
NUC2	1H	
PCPD2	75.00	usec
PL2	-1.00	dB
PL12	13.39	dB
PL13	20.00	dB
PL2W	11.09959412	W
PL12W	0.40393090	W
PL13W	0.08816721	W
SFO2	400.2316009	MHz
SI	32768	
SF	100.6379197	MHz
WDW	EM	
SSB	0	
LB	1.00	Hz
GB	0	
PC	1.40	

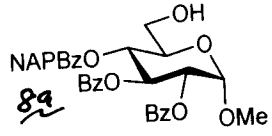


BRUKER

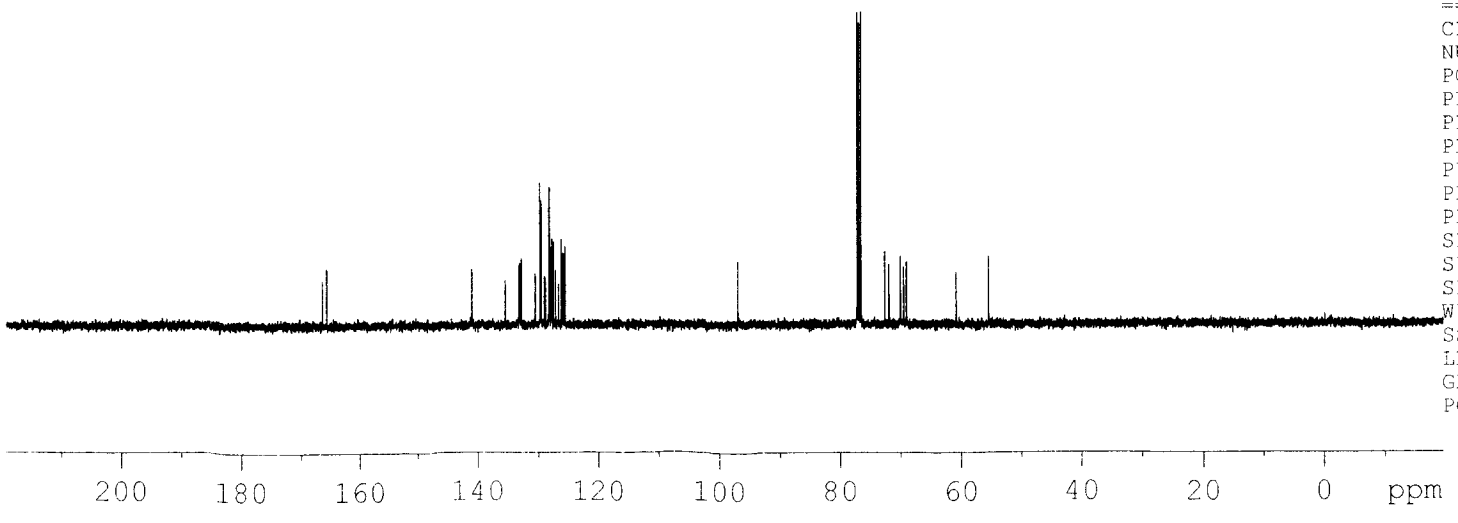
NAME	0213
EXPNO	1
PROCNO	1
Date_	20111014
Time	16.42
INSTRUM	spect
PROBHD	5 mm PABBO BB-
PULPROG	zg30
TD	65536
SOLVENT	CDCl3
NS	16
DS	2
SWH	8223.685 Hz
FIDRES	0.125483 Hz
AQ	3.9846387 sec
RG	57
DW	60.800 usec
DE	6.50 usec
TE	295.6 K
D1	2.00000000 sec
TD0	1
======== CHANNEL f1 ========	
NUC1	1H
P1	14.31 usec
PL1	-1.00 dB
PL1W	11.09959412 W
SFO1	400.2324716 MHz
SI	32768
SF	400.2300200 MHz
WDW	EM
SSB	0
LB	0.30 Hz
GB	0
PC	1.00

0213

166.51
165.77
141.11
135.59
133.33
133.21
133.18
132.92
130.62
129.86
129.64
129.12
128.95
128.37
128.29
128.07
127.98
127.85
127.63
127.26
126.77
126.33
126.04
125.81
125.70
97.02
72.74
72.05
70.16
70.10
69.59
69.15
60.93
55.55



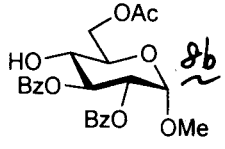
89

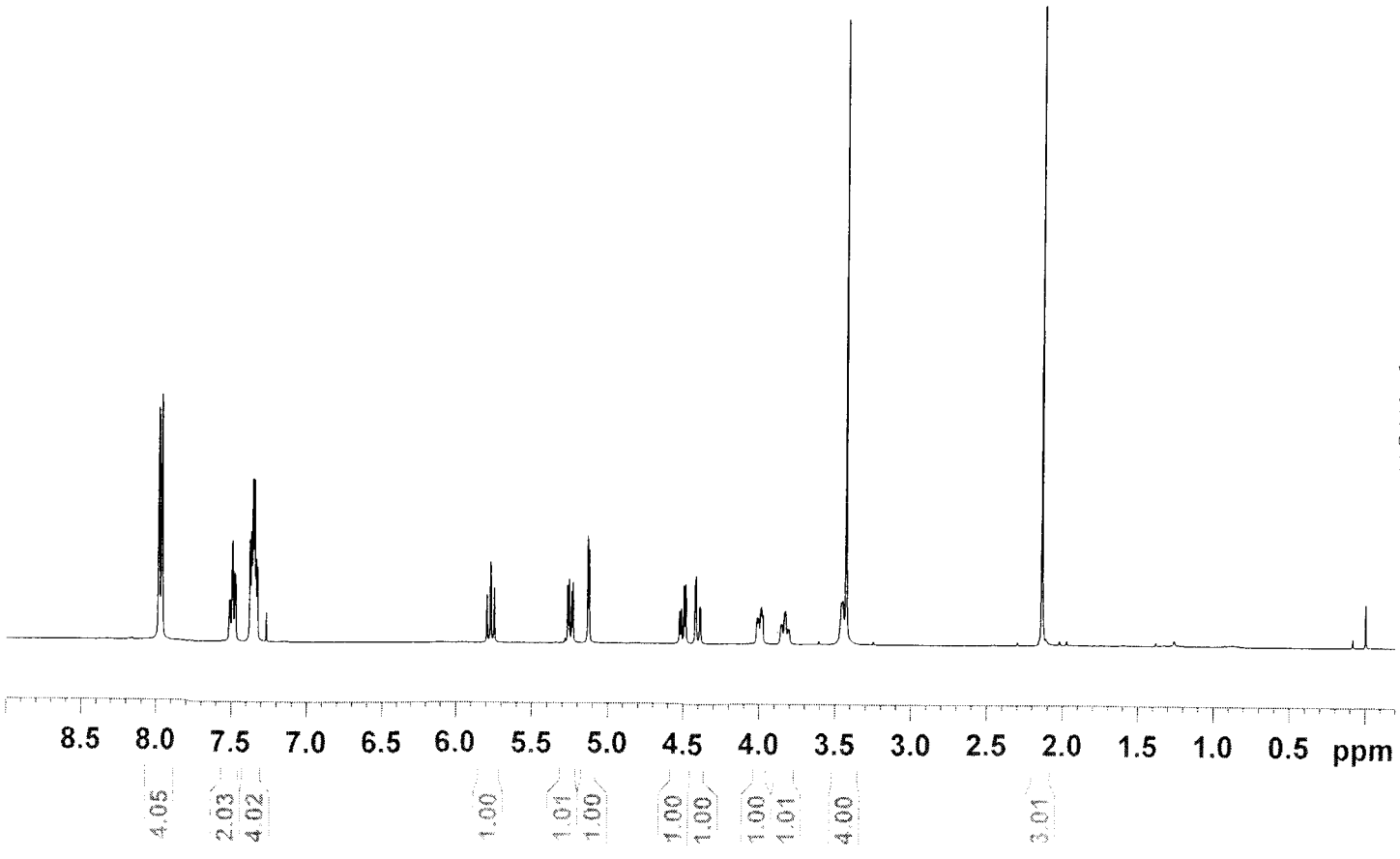


NAME	0213
EXPNO	2
PROCNO	1
Date_	20111014
Time	16.53
INSTRUM	spect
PROBHD	5 mm PABBO BB-
PULPROG	zgpg30
TD	65536
SOLVENT	CDCl3
NS	38
DS	4
SWH	24038.461 Hz
FIDRES	0.366798 Hz
AQ	1.3631988 sec
RG	203
DW	20.800 usec
DE	6.50 usec
TE	296.1 K
D1	3.00000000 sec
D11	0.03000000 sec
TD0	1
======== CHANNEL f1 ========	
NUC1	13C
P1	10.00 usec
PL1	-1.59 dB
PL1W	51.07626343 W
SFO1	100.6479773 MHz
======== CHANNEL f2 ========	
CPDPRG2	waltz16
NUC2	1H
PCPD2	75.00 usec
PL2	-1.00 dB
PL12	13.39 dB
PL13	20.00 dB
PL2W	11.09959412 W
PL12W	0.40393090 W
PL13W	0.08816721 W
SFO2	400.2316009 MHz
SI	32768
SF	100.6379243 MHz
WDW	EM
SSB	0
LB	1.00 Hz
GB	0
PC	1.40

01291

7.960 7.492 7.488 7.474 7.470 7.354 7.344 5.794 5.769 5.745 5.264 5.255 5.239 5.230 5.132 5.123 4.524 4.513 4.494 4.482 4.420 4.415 4.390 4.385 4.017 4.011 4.006 4.000 3.992 3.986 3.981 3.976 3.850 3.827 3.808 3.429 2.138

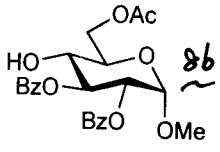


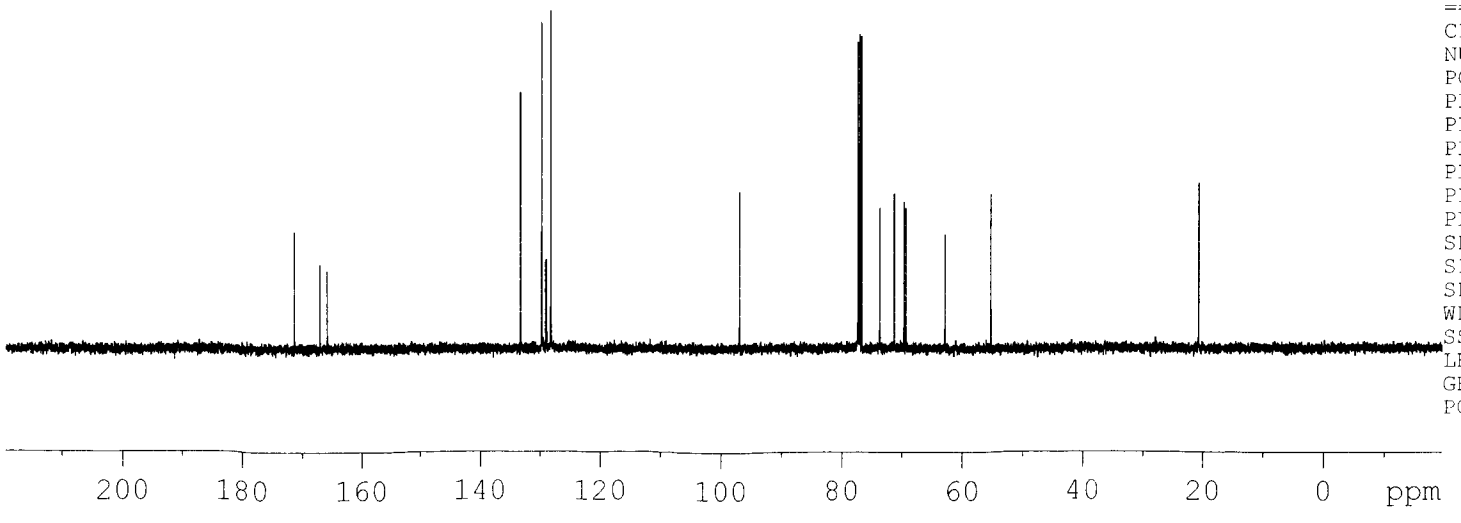


NAME	01291
EXPNO	1
PROCNO	1
Date_	20111017
Time	18.23
INSTRUM	spect
PROBHD	5 mm PABBO BB-
PULPROG	zg30
TD	65536
SOLVENT	CDCl3
NS	7
DS	2
SWH	8223.685 Hz
FIDRES	0.125483 Hz
AQ	3.9846387 sec
RG	45.2
DW	60.800 usec
DE	6.50 usec
TE	297.0 K
D1	2.00000000 sec
TD0	1
======== CHANNEL f1 ========	
NUC1	1H
P1	14.31 usec
PL1	-1.00 dB
PL1W	11.09959412 W
SFO1	400.2324716 MHz
SI	32768
SF	400.2300097 MHz
WDW	EM
SSB	0
LB	0.30 Hz
GB	0
PC	1.00

01291

171.38
167.11
165.88
133.30
129.78
129.75
129.14
128.98
128.33
128.30
97.02
73.69
71.32
69.70
69.39
62.93
55.33
20.78

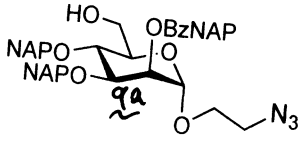




NAME	01291
EXPNO	2
PROCNO	1
Date_	20111017
Time	18.29
INSTRUM	spect
PROBHD	5 mm PABBO BB-
PULPROG	zgpg30
TD	65536
SOLVENT	CDCl3
NS	23
DS	4
SWH	24038.461 Hz
FIDRES	0.366798 Hz
AQ	1.3631988 sec
RG	203
DW	20.800 usec
DE	6.50 usec
TE	297.0 K
D1	3.00000000 sec
D11	0.03000000 sec
TD0	1
======== CHANNEL f1 ========	
NUC1	13C
P1	10.00 usec
PL1	-1.59 dB
PL1W	51.07626343 W
SFO1	100.6479773 MHz
======== CHANNEL f2 ========	
CPDPRG2	waltz16
NUC2	1H
PCPD2	75.00 usec
PL2	-1.00 dB
PL12	13.39 dB
PL13	20.00 dB
PL2W	11.09959412 W
PL12W	0.40393090 W
PL13W	0.08816721 W
SFO2	400.2316009 MHz
SI	32768
SF	100.6379250 MHz
WDW	EM
SSB	0
LB	1.00 Hz
GB	0
PC	1.40

01285

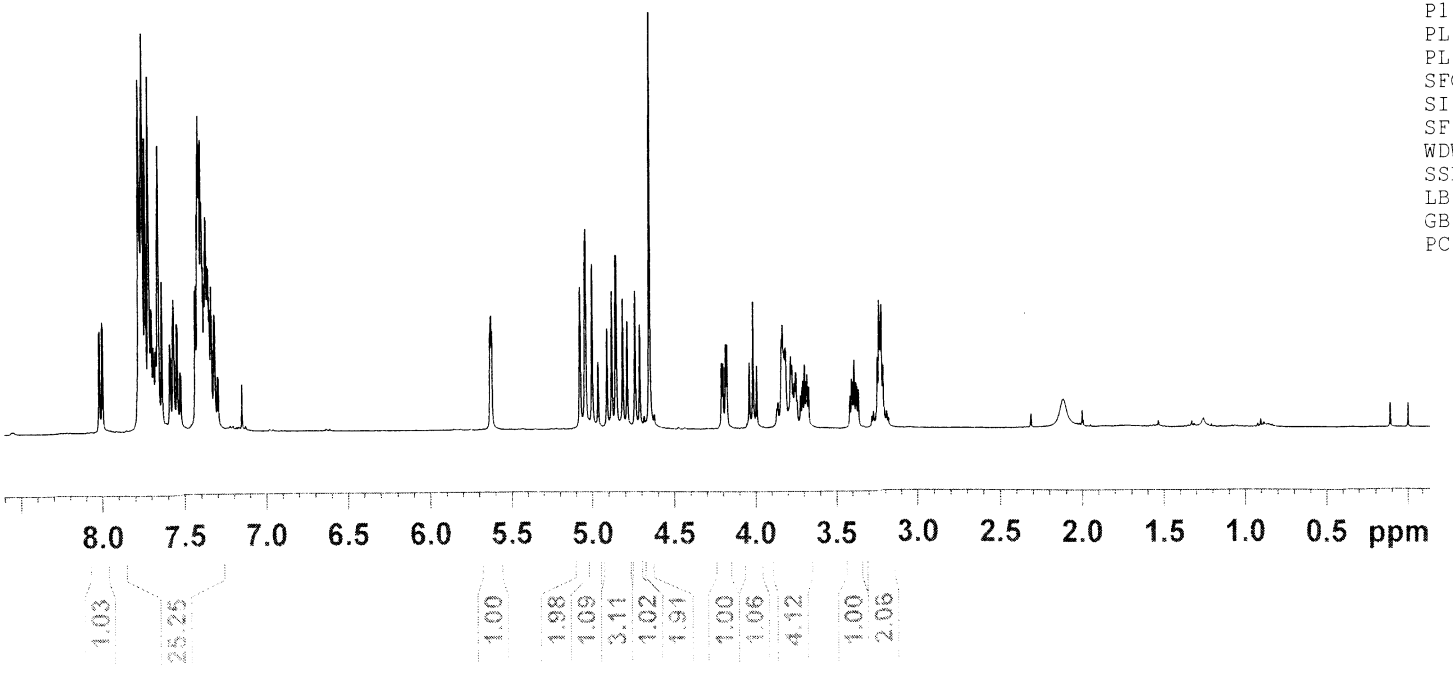
7.717 7.658 7.636 7.565 7.414 5.633 5.628 5.625 5.620 5.074 5.041 4.999 4.963 4.908 4.879 4.855 4.851 4.814 4.785 4.739 4.710 4.652 4.208 4.200 4.185 4.177 4.039 4.016 3.992 3.832 3.781 3.773 3.696 3.237 3.232 3.226 3.223



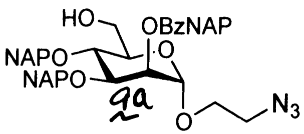
BRUKER

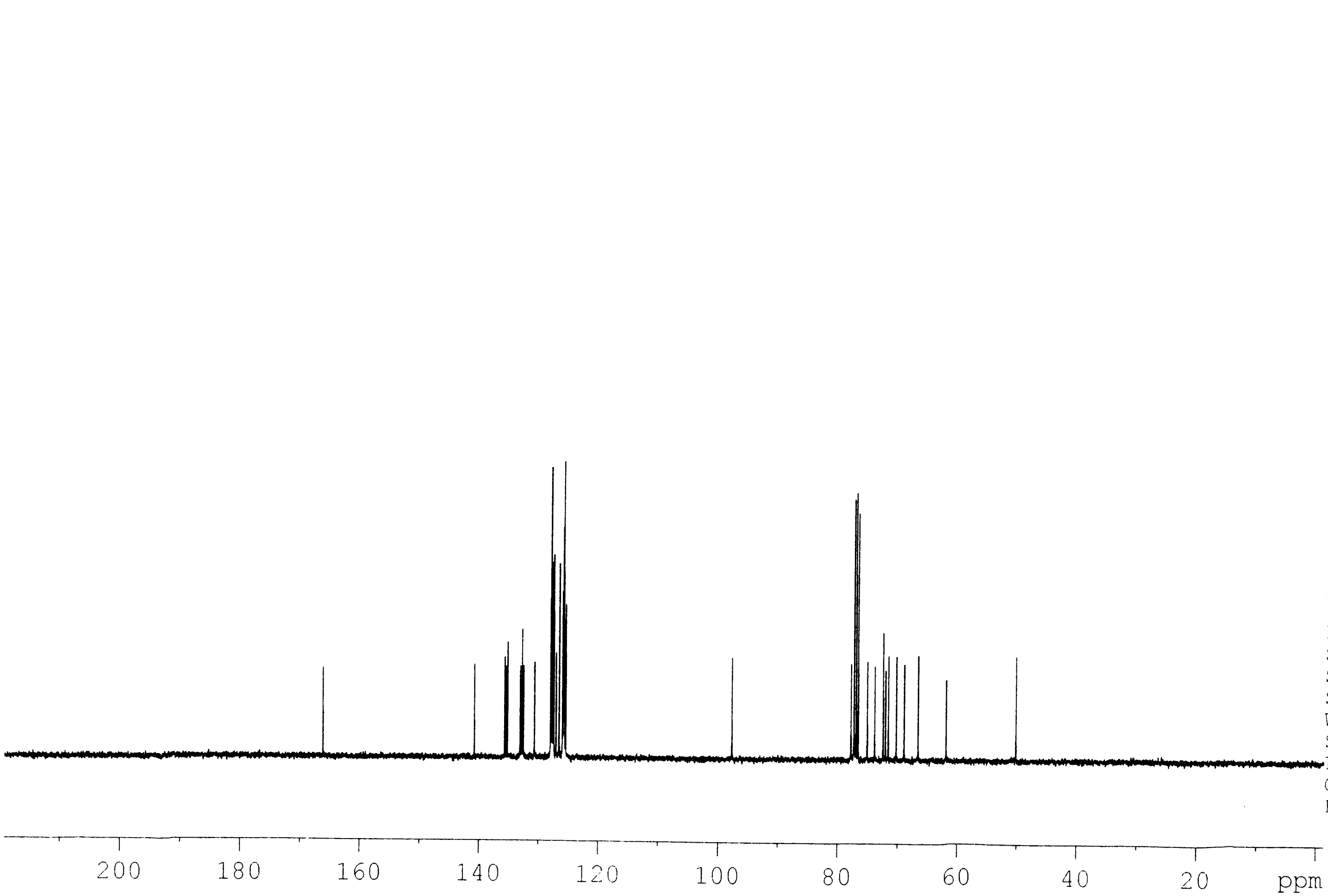
NAME	01285	
EXPNO	1	
PROCNO	1	
Date_	20110927	
Time	14.19	
INSTRUM	spect	
PROBHD	5 mm PABBO BB-	
PULPROG	zg30	
TD	65536	
SOLVENT	CDCl3	
NS	16	
DS	2	
SWH	8223.685	Hz
FIDRES	0.125483	Hz
AQ	3.9846387	sec
RG	25.4	
DW	60.800	usec
DE	6.50	usec
TE	296.7	K
D1	2.00000000	sec
TD0	1	

======== CHANNEL f1 ========		
NUC1	1H	
P1	14.31	usec
PL1	-1.00	dB
PL1W	11.09959412	W
SFO1	400.2324716	MHz
SI	32768	
SF	400.2300565	MHz
WDW	EM	
SSB	0	
LB	0.30	Hz
GB	0	
PC	1.00	



01285





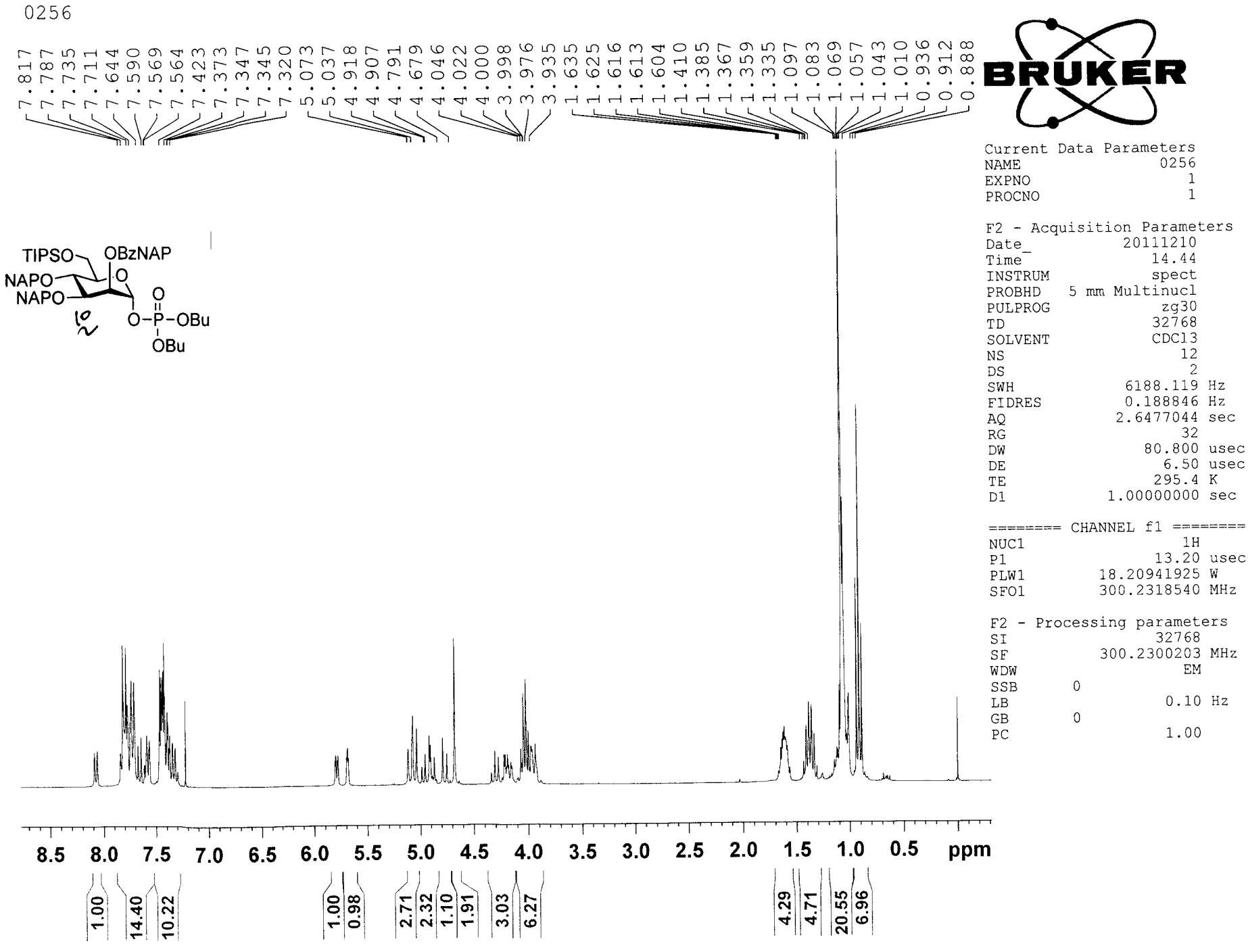
NAME	01285
EXPNO	2
PROCNO	1
Date_	20110927
Time	14.30
INSTRUM	spect
PROBHD	5 mm PABBO BB-
PULPROG	zgpg30
TD	65536
SOLVENT	CDCl3
NS	76
DS	4
SWH	24038.461 Hz
FIDRES	0.366798 Hz
AQ	1.3631988 sec
RG	203
DW	20.800 usec
DE	6.50 usec
TE	297.1 K
D1	3.00000000 sec
D11	0.03000000 sec
TD0	1

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

NUC1	13C
P1	10.00 usec
PL1	-1.59 dB
PL1W	51.07626343 W
SFO1	100.6479773 MHz

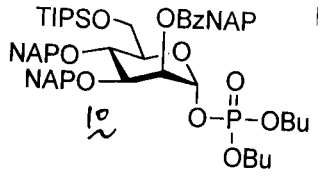
======== CHANNEL f2 ========

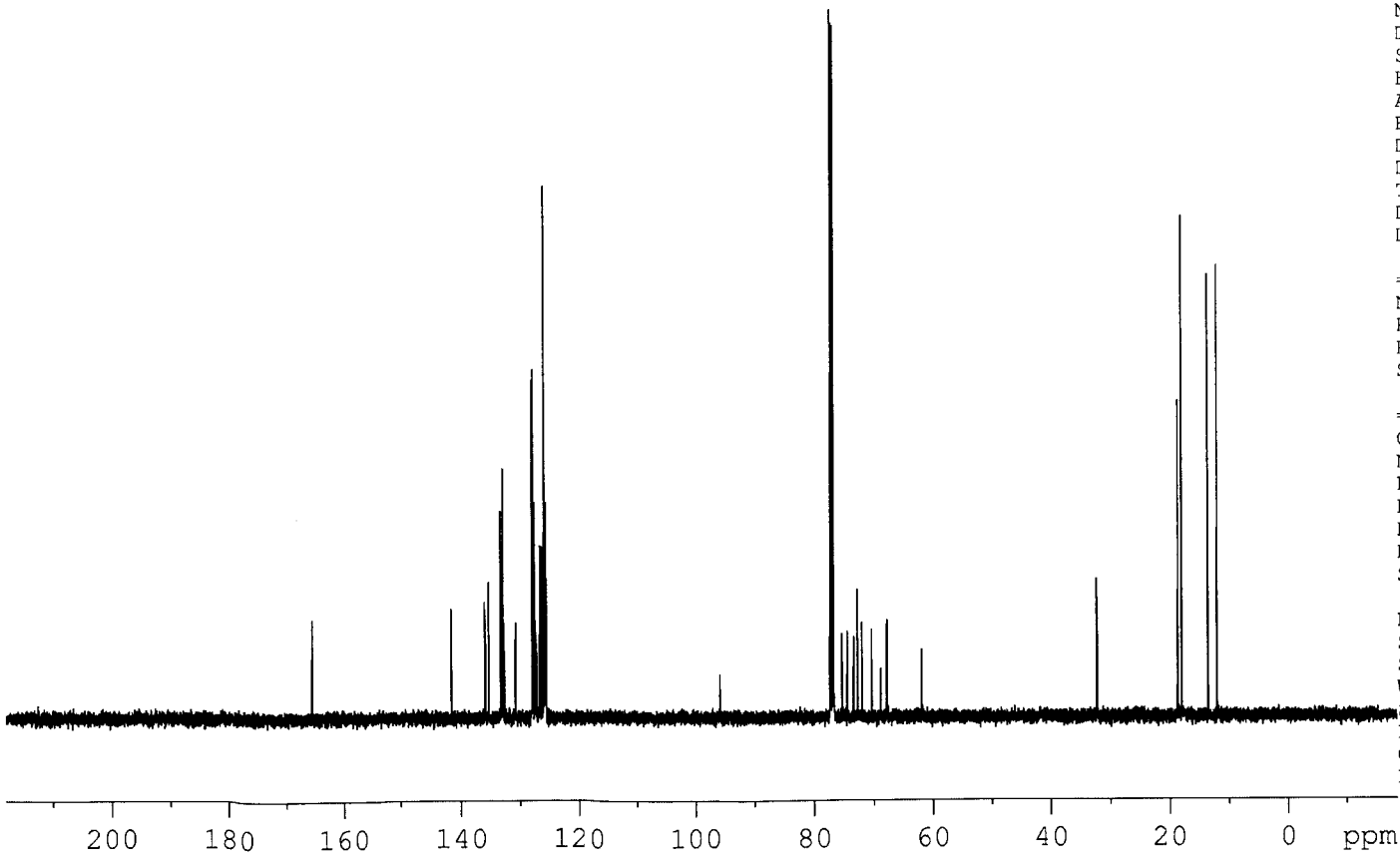
CPDPRG2	waltz16
NUC2	1H
PCPD2	75.00 usec
PL2	-1.00 dB
PL12	13.39 dB
PL13	20.00 dB
PL2W	11.09959412 W
PL12W	0.40393090 W
PL13W	0.08816721 W
SFO2	400.2316009 MHz
SI	32768
SF	100.6379382 MHz
WDW	EM
SSB	0
LB	1.00 Hz
GB	0
PC	1.40


0256
7.817
7.787
7.735
7.711
7.644
7.590
7.569
7.564
7.423
7.373
7.347
7.345
7.320
5.073
5.037
4.918
4.907
4.791
4.679
4.046
4.022
4.000
3.998
3.976
3.935
1.635
1.625
1.616
1.613
1.604
1.410
1.385
1.367
1.359
1.335
1.097
1.083
1.069
1.057
1.043
1.010
0.936
0.912
0.888
BRUKER
TIPSO
OBzNAP
NAPO
NAPO
O
O-P-OBu
OBu
Current Data Parameters
NAME 0256
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20111210
Time 14.44
INSTRUM spect
PROBHD 5 mm Multinucl
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 12
DS 2
SWH 6188.119 Hz
FIDRES 0.188846 Hz
AQ 2.6477044 sec
RG 32
DW 80.800 usec
DE 6.50 usec
TE 295.4 K
D1 1.00000000 sec
======== CHANNEL f1 ========
NUC1 1H
P1 13.20 usec
PLW1 18.20941925 W
SFO1 300.2318540 MHz
F2 - Processing parameters
SI 32768
SF 300.2300203 MHz
WDW EM
SSB 0
LB 0.10 Hz
GB 0
PC 1.00
8.5
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
1.00
14.40
10.22
1.00
0.98
2.71
2.32
1.10
1.91
3.03
6.27
4.29
4.71
20.55
6.96

0256pc

165.60 141.60 136.02 135.92 135.34 133.30 133.18 132.95 132.71 130.83 128.04 128.03 127.87 127.63 127.61 127.58 127.37 126.80 126.65 126.37 126.09 125.95 125.92 125.84 125.80 125.76 125.69 125.60 74.47 72.75 71.98 70.35 67.87 67.82 67.74 32.24 32.20 32.18 32.14 18.59 17.96 17.92 13.52 12.00





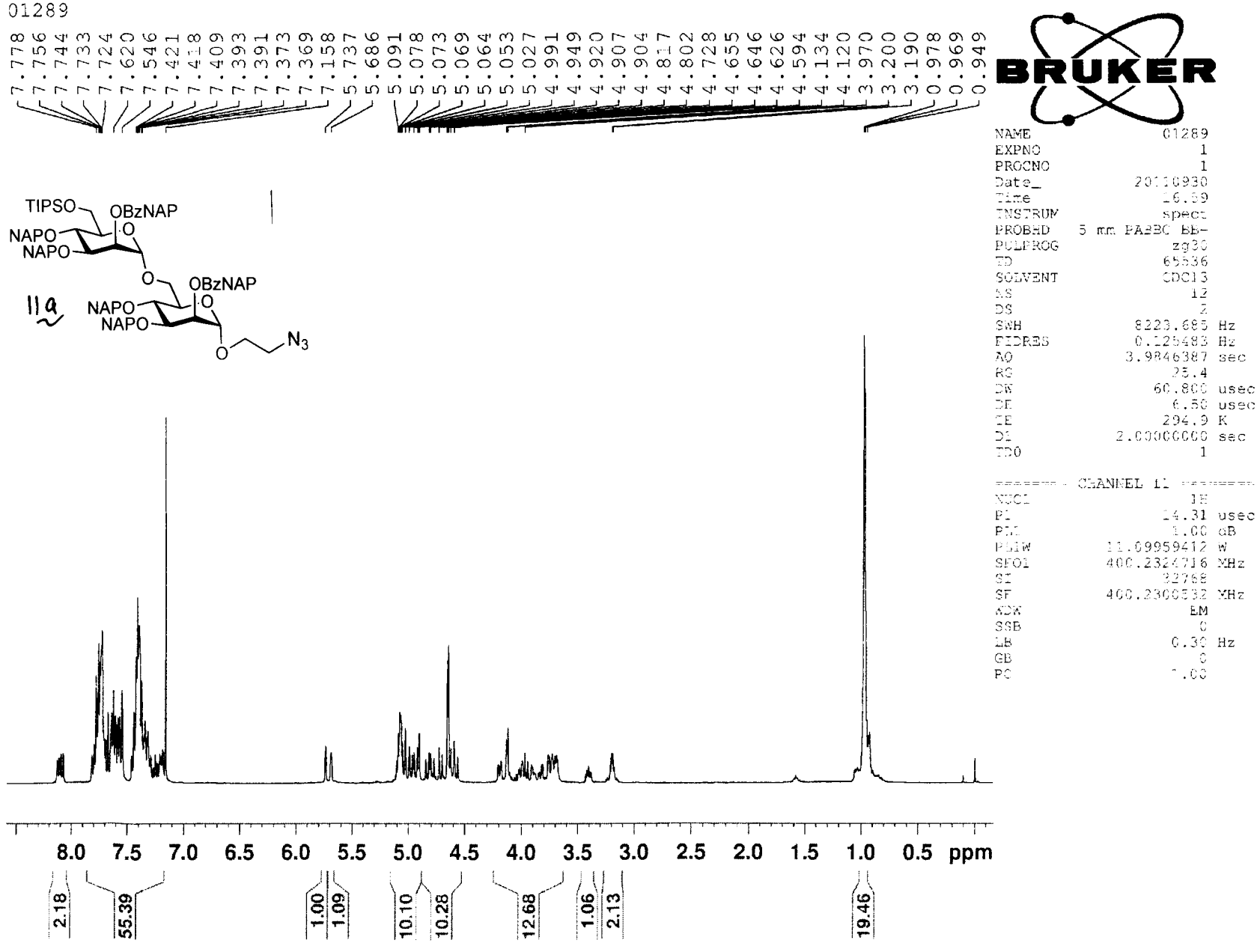
Current Data Parameters
NAME 0256pc
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20111212
Time 17.13
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 149
DS 4
SWH 29761.904 Hz
FIDRES 0.454131 Hz
AQ 1.1010548 sec
RG 203
DW 16.800 usec
DE 6.50 usec
TE 300.0 K
D1 2.00000000 sec
D11 0.03000000 sec

======== CHANNEL f1 ========
NUC1 13C
P1 8.80 usec
PLW1 112.58000183 W
SFO1 125.7779080 MHz

======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PLW2 18.74300003 W
PLW12 0.36736000 W
PLW13 0 W
SFO2 500.1620006 MHz

F2 - Processing parameters
SI 32768
SF 125.7653424 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

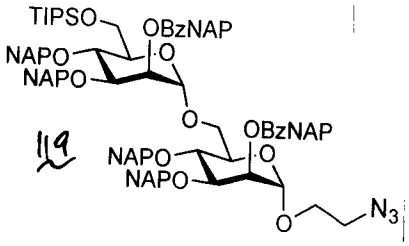


BRUKER

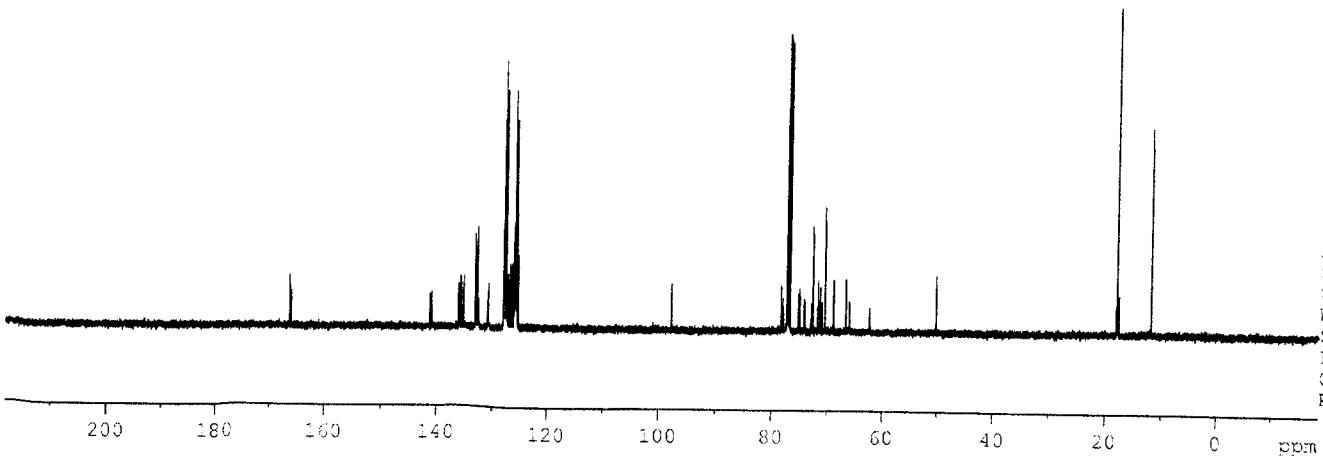
NAME	01289
EXPNO	1
PROCNO	1
Date_	20110930
Time	16.59
INSTRUM	spect
PROBHD	5 mm PABBO BB-
PULPROG	zg30
TD	65536
SOLVENT	CDCl3
NS	12
DS	2
SWH	8223.685 Hz
FIDRES	0.125483 Hz
AQ	3.9846387 sec
RG	25.4
DW	60.800 usec
DE	6.50 usec
TE	294.9 K
D1	2.00000000 sec
TD0	1
======== CHANNEL f1 ========	
NUC1	1H
P1	14.31 usec
PL1	1.00 dB
PL1W	11.09959412 W
SFO1	400.2324716 MHz
SI	32768
SF	400.2300532 MHz
ADC	EM
SSB	0
LB	0.30 Hz
GB	0
PC	1.00

01289

166.28
136.18
135.90
135.85
135.71
135.38
135.29
133.23
133.21
133.18
133.08
132.93
132.85
132.78
130.71
128.04
128.00
127.94
127.88
127.83
127.78
127.60
127.56
127.47
127.26
126.91
126.83
126.61
126.34
126.14
126.07
126.03
125.98
125.94
125.87
125.82
125.68
125.63
125.59
125.57
125.38
97.84
97.81
78.31
77.20
75.17
74.99
72.65
72.57
71.74
71.27
70.41
68.89
66.62
50.19
17.91
17.89
11.87



19



BRUKER

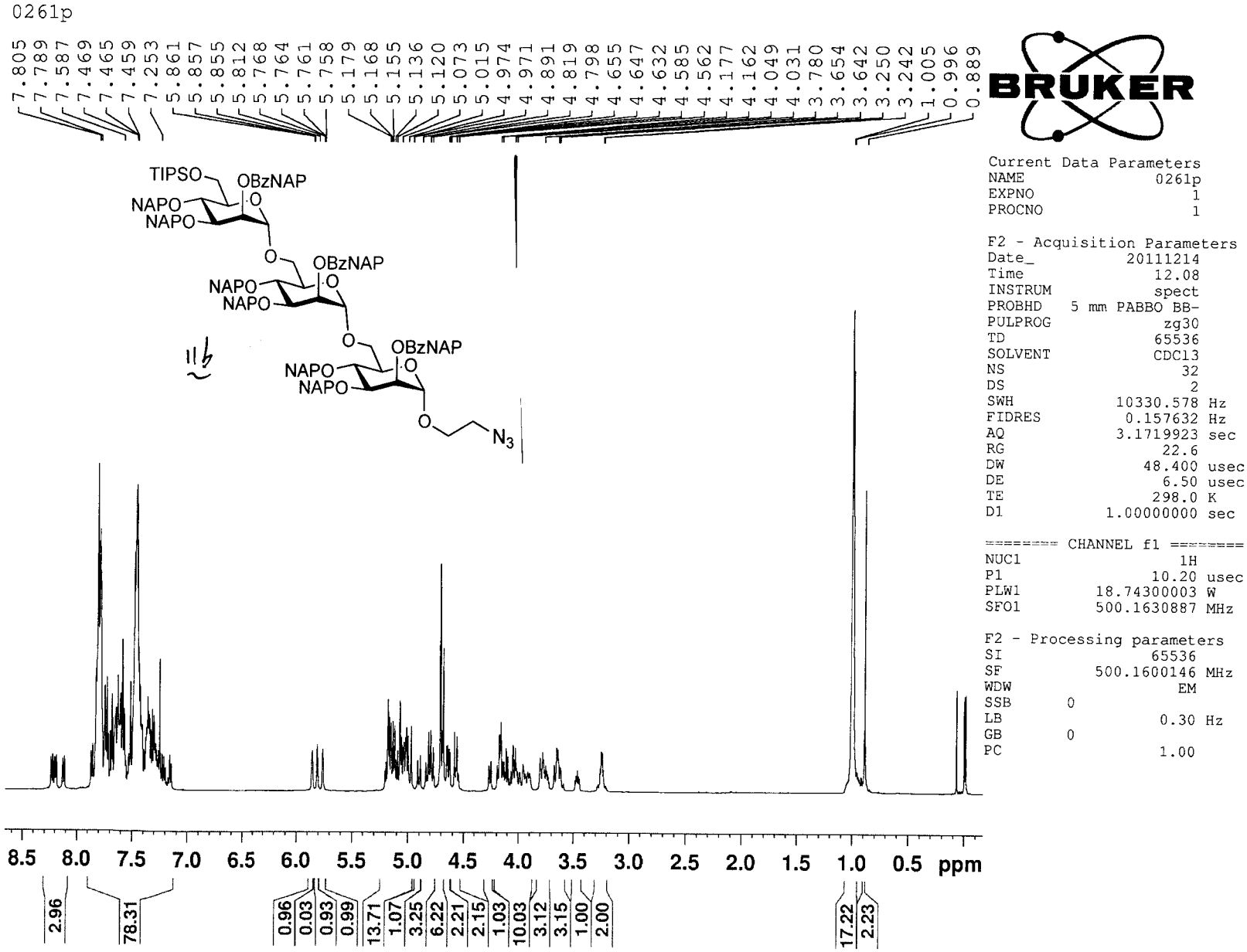
```
Current Data Parameters
NAME                  01289
EXPNO                     1
PROCNO                    1

F2 - Acquisition Parameters
Date_              20110930
Time                  17.47
INSTRUM                spect
PROBHD   5 mm PABBO BB-
PULPROG              zgpg30
TD                    65536
SOLVENT               CDCl3
NS                       91
DS                        4
SWH               29761.904 Hz
FIDRES                0.454131 Hz
AQ                1.1010548 sec
RG                      203
DW                   16.800 usec
DE                    6.50 usec
TE                   295.0 K
D1                2.00000000 sec
D11               0.03000000 sec

======== CHANNEL f1 ========
NUC1                     13C
P1                    8.80 usec
PLW1           112.58000183 W
SFO1           125.7779080 MHz

======== CHANNEL f2 ========
CPDPRG2              waltz16
NUC2                     1H
PCPD2                 80.00 usec
PLW2           18.74300003 W
PLW12           0.36736000 W
PLW13           0.18408000 W
SFO2           500.1620006 MHz

F2 - Processing parameters
SI                       32768
SF             125.7653544 MHz
WDW                         EM
SSB                  0
LB                        1.00 Hz
GB                   0
PC                        1.40
```

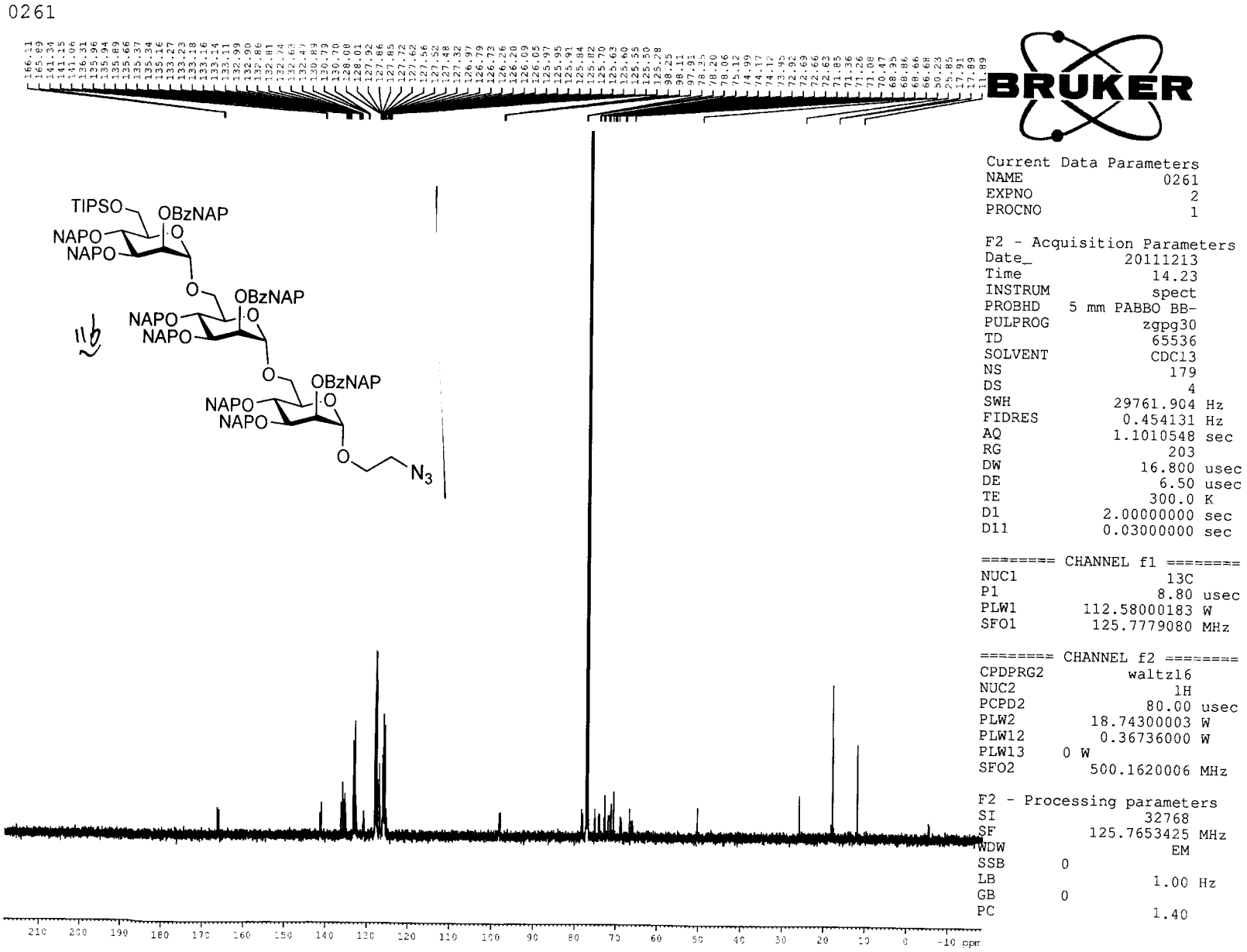


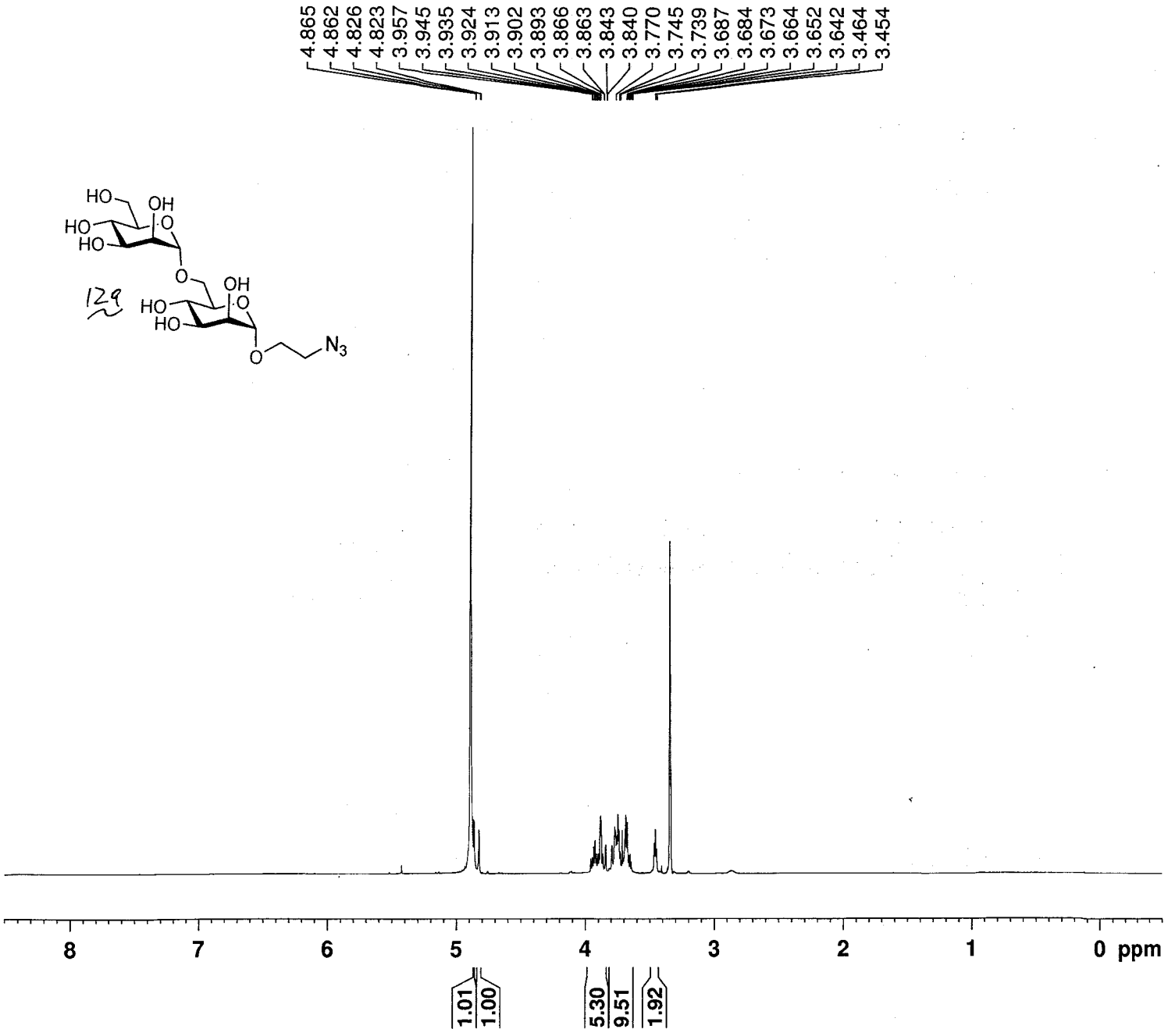

```
Current Data Parameters
NAME                  0261p
EXPNO                     1
PROCNO                    1

F2 - Acquisition Parameters
Date_              20111214
Time                 12.08
INSTRUM              spect
PROBHD    5 mm PABBO BB-
PULPROG               zg30
TD                   65536
SOLVENT              CDCl3
NS                      32
DS                       2
SWH             10330.578 Hz
FIDRES           0.157632 Hz
AQ               3.1719923 sec
RG                    22.6
DW                  48.400 usec
DE                   6.50 usec
TE                  298.0 K
D1               1.00000000 sec

========= CHANNEL f1 =========
NUC1                    1H
P1                   10.20 usec
PLW1          18.74300003 W
SFO1          500.1630887 MHz

F2 - Processing parameters
SI                    65536
SF            500.1600146 MHz
WDW                     EM
SSB       0
LB                   0.30 Hz
GB        0
PC                    1.00
```


0261
BRUKER
TIPSO
OBzNAP
NAPO
NAPO
OBzNAP
OBzNAP
N3
Current Data Parameters
NAME 0261
EXPNO 2
PROCNO 1
F2 - Acquisition Parameters
Date_ 20111213
Time 14.23
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 179
DS 4
SWH 29761.904 Hz
FIDRES 0.454131 Hz
AQ 1.1010548 sec
RG 203
DW 16.800 usec
DE 6.50 usec
TE 300.0 K
D1 2.00000000 sec
D11 0.03000000 sec
======== CHANNEL f1 ========
NUC1 13C
P1 8.80 usec
PLW1 112.58000183 W
SFO1 125.7779080 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PLW2 18.74300003 W
PLW12 0.36736000 W
PLW13 0 W
SFO2 500.1620006 MHz
F2 - Processing parameters
SI 32768
SF 125.7653425 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

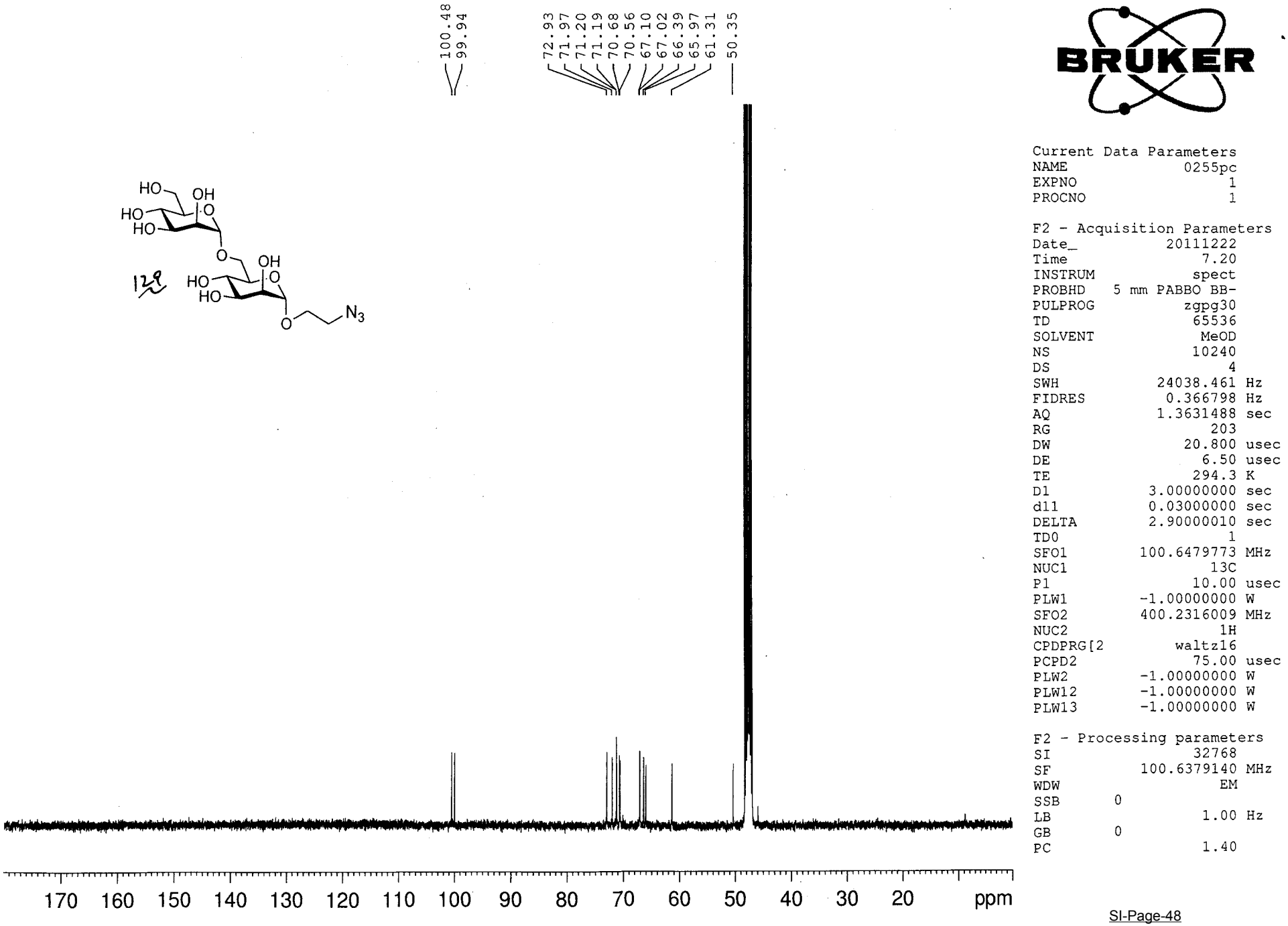


Current Data Parameters
NAME 0255
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20111210
Time 11.55
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT MeOD
NS 16
DS 2
SWH 10330.578 Hz
FIDRES 0.157632 Hz
AQ 3.1719425 sec
RG 90.5
DW 48.400 usec
DE 6.50 usec
TE 298.0 K
D1 1.00000000 sec

======== CHANNEL f1 ========
NUC1 1H
P1 10.20 usec
PLW1 18.74300003 W
SFO1 500.1630887 MHz

F2 - Processing parameters
SI 65536
SF 500.1599946 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

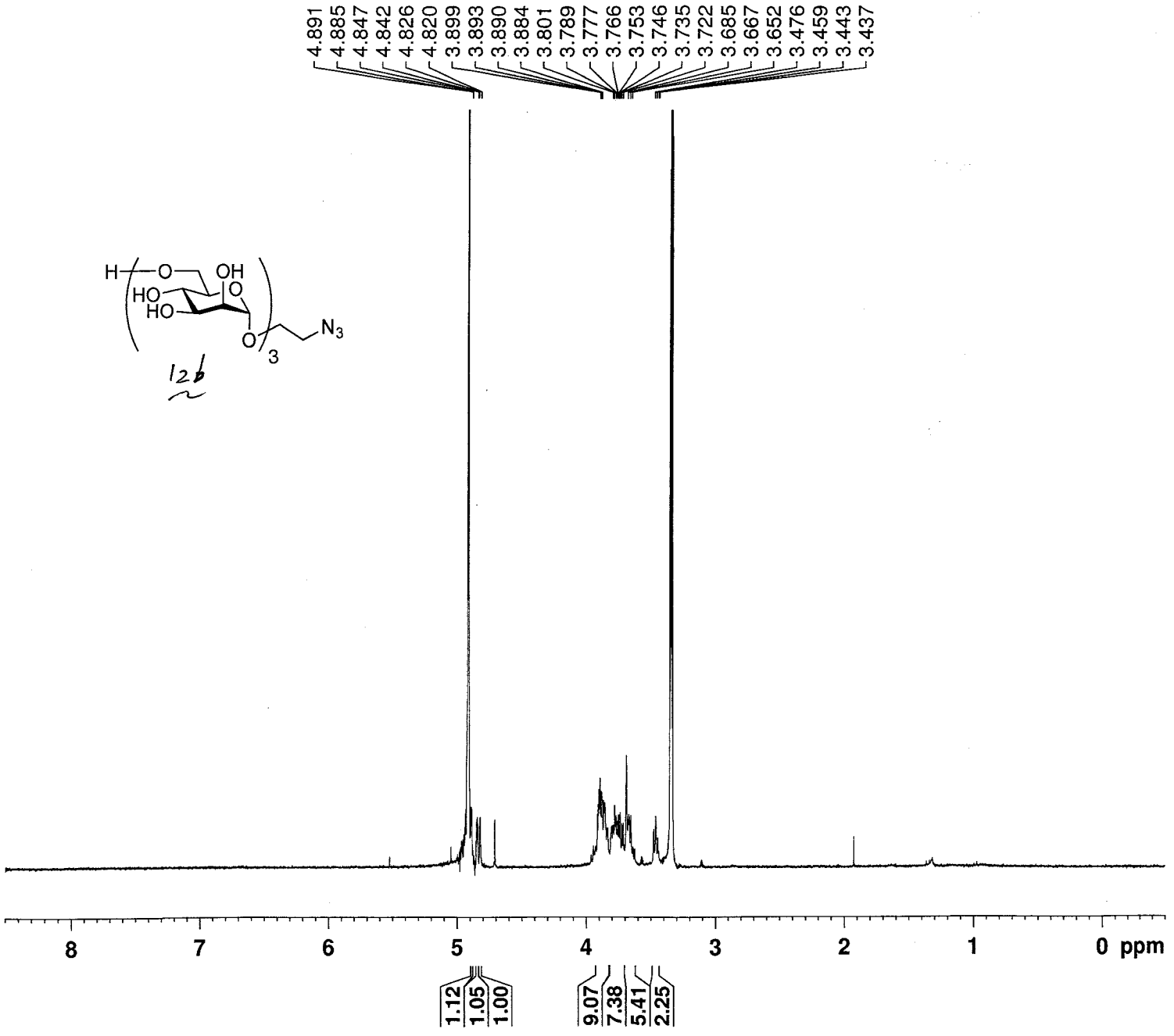


BRUKER

Current Data Parameters
NAME 0255pc
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20111222
Time 7.20
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT MeOD
NS 10240
DS 4
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3631488 sec
RG 203
DW 20.800 usec
DE 6.50 usec
TE 294.3 K
D1 3.00000000 sec
d11 0.03000000 sec
DELTA 2.90000010 sec
TD0 1
SFO1 100.6479773 MHz
NUC1 13C
P1 10.00 usec
PLW1 -1.00000000 W
SFO2 400.2316009 MHz
NUC2 1H
CPDPRG[2 waltz16
PCPD2 75.00 usec
PLW2 -1.00000000 W
PLW12 -1.00000000 W
PLW13 -1.00000000 W

F2 - Processing parameters
SI 32768
SF 100.6379140 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40



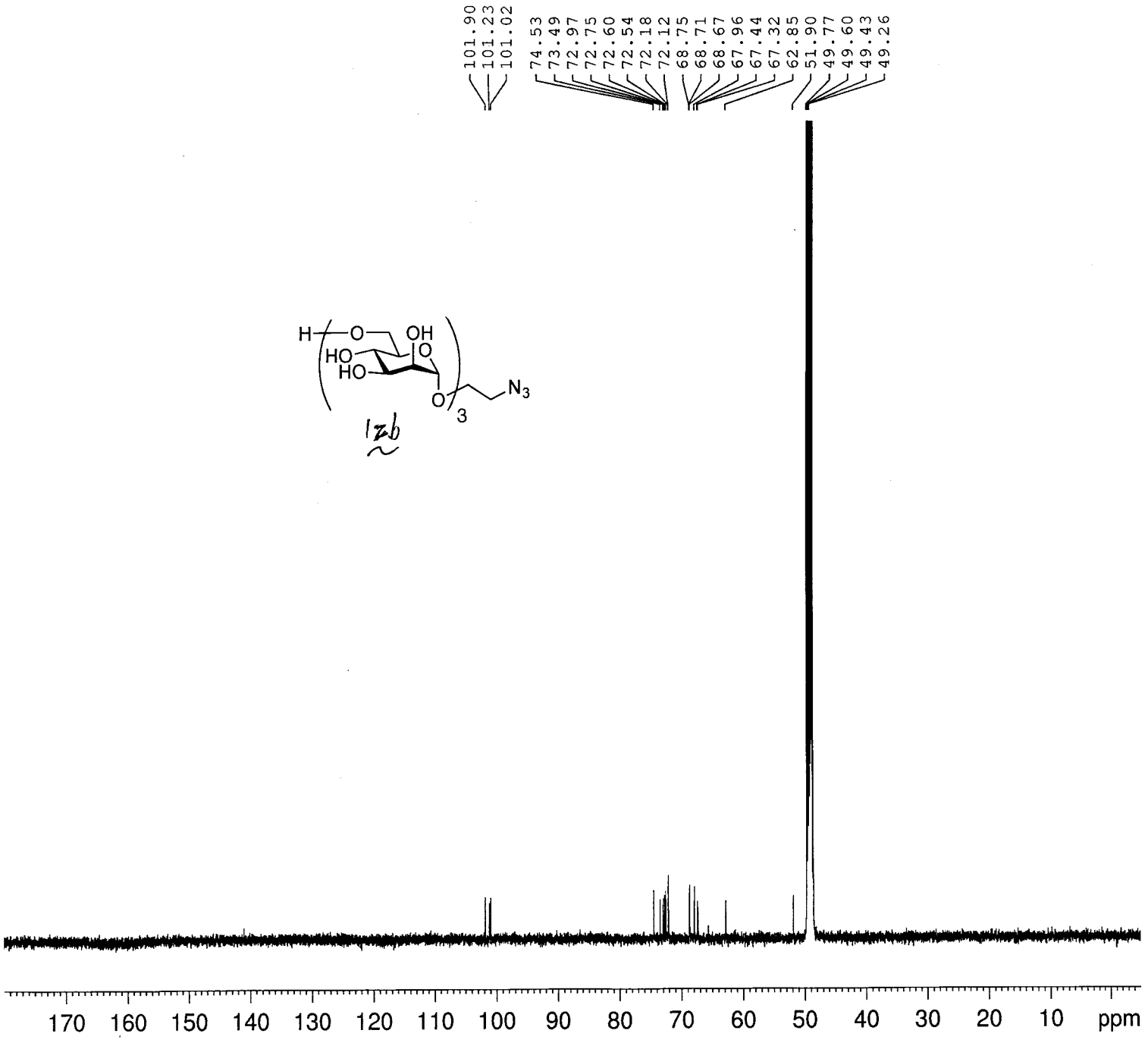
BRUKER

```
Current Data Parameters
NAME                  0266p
EXPNO                     1
PROCNO                    1

F2 - Acquisition Parameters
Date_              20111217
Time                  17.58
INSTRUM               spect
PROBHD    5 mm Multinucl
PULPROG                zg30
TD                    32768
SOLVENT                MeOD
NS                       16
DS                        2
SWH                6188.119 Hz
FIDRES            0.188846 Hz
AQ                2.6476543 sec
RG                      203
DW                  80.800 usec
DE                   6.50 usec
TE                   294.0 K
D1              1.00000000 sec

======== CHANNEL f1 ========
NUC1                      1H
P1                    13.20 usec
PLW1          18.20941925 W
SFO1          300.2318540 MHz

F2 - Processing parameters
SI                    32768
SF            300.2299980 MHz
WDW                      EM
SSB       0
LB                    0.10 Hz
GB        0
PC                    1.00
```

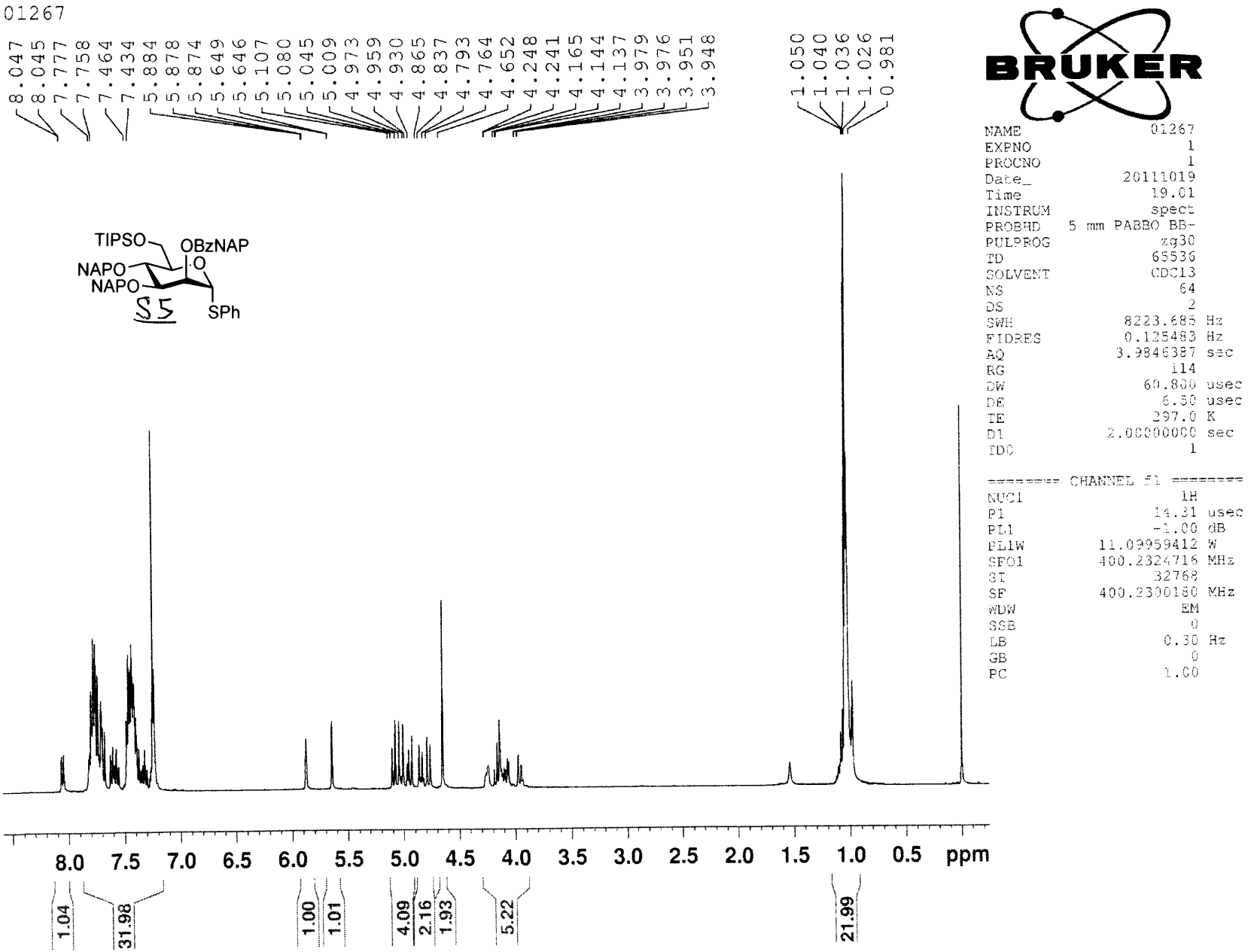


BRUKER

Current Data Parameters
NAME 0266 pc
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20120107
Time 8.22
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT MeOD
NS 15217
DS 4
SWH 29761.904 Hz
FIDRES 0.454131 Hz
AQ 1.1010048 sec
RG 203
DW 16.800 usec
DE 6.50 usec
TE 294.2 K
D1 2.00000000 sec
d11 0.03000000 sec
DELTA 1.89999998 sec
TD0 1
SFO1 125.7779080 MHz
NUC1 13C
P1 8.80 usec
PLW1 112.58000183 W
SFO2 500.1620006 MHz
NUC2 1H
CPDPRG[2 waltz16
PCPD2 80.00 usec
PLW2 18.74300003 W
PLW12 0.36736000 W
PLW13 0 W

F2 - Processing parameters
SI 32768
SF 125.7651376 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40



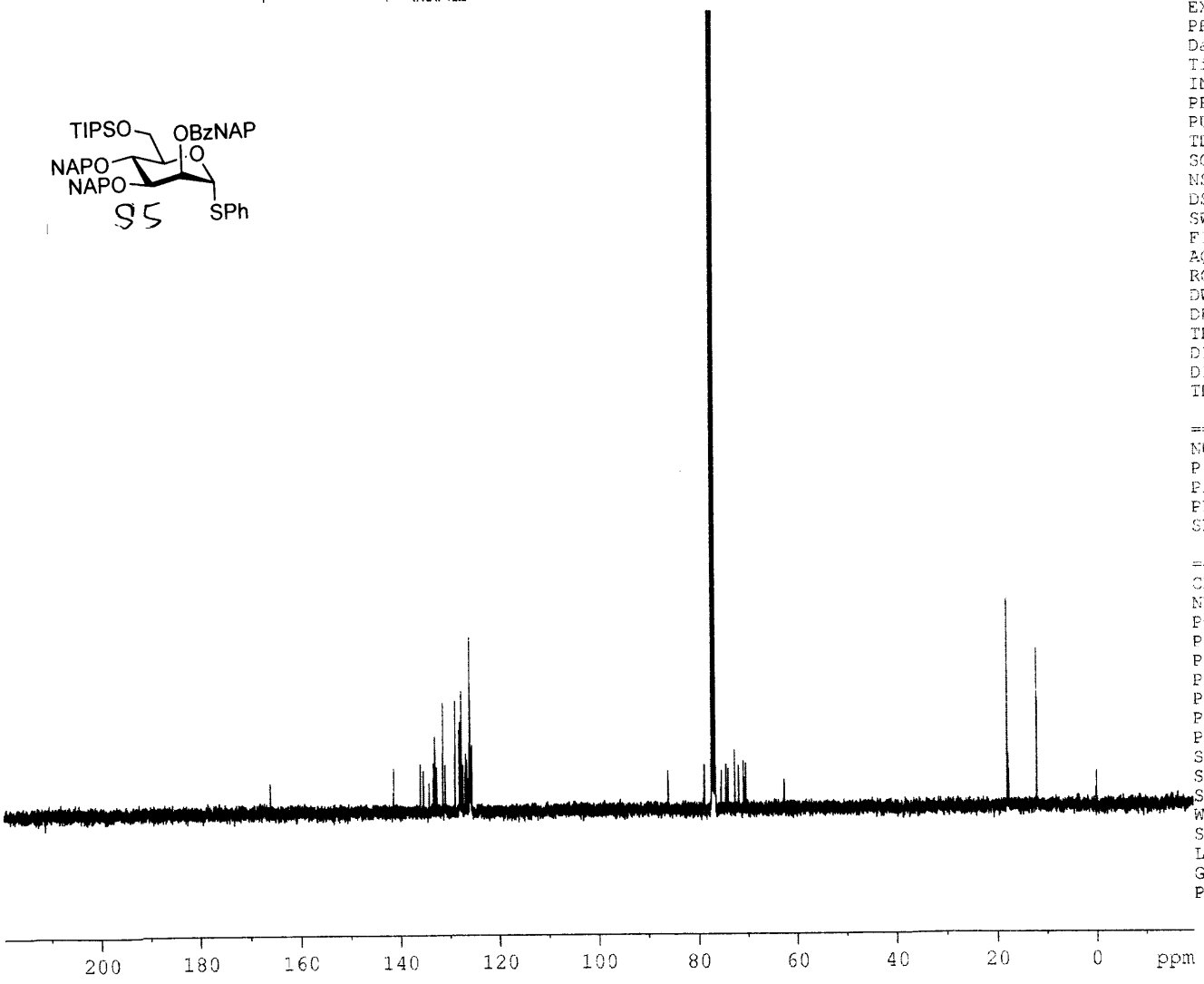
BRUKER

NAME	01267
EXPNO	1
PROCNO	1
Date_	20111019
Time	19.01
INSTRUM	spect
PROBHD	5 mm PABBO BB-
PULPROG	zg30
TD	65536
SOLVENT	CDCl3
NS	64
DS	2
SWH	8223.685 Hz
FIDRES	0.125483 Hz
AQ	3.9846387 sec
RG	114
DW	60.800 usec
DE	6.50 usec
TE	297.0 K
D1	2.00000000 sec
D0	1
======== CHANNEL f1 ========	
NUC1	1H
P1	14.31 usec
PL1	-1.00 dB
PL1W	11.09959412 W
SFO1	400.2324716 MHz
SI	32768
SF	400.2300180 MHz
WDW	EM
SSB	0
LB	0.30 Hz
GB	0
PC	1.00

01267

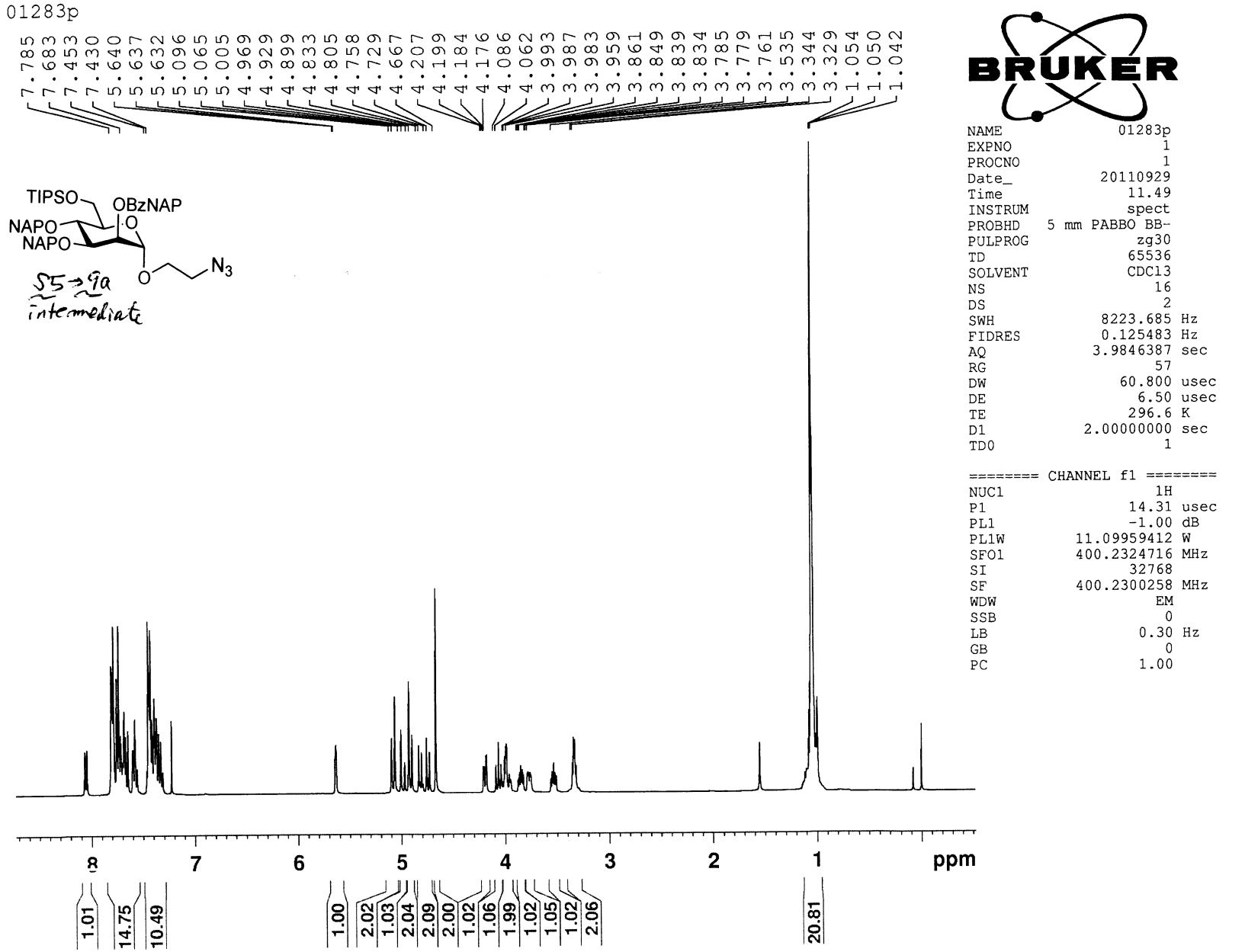
166.07 141.31 135.93 135.89 135.29 134.17 133.30 133.28 133.22 132.97 132.92 132.64 131.47 131.41 130.94 128.93 128.13 128.08 128.05 127.93 127.88 127.64 127.46 127.39 126.91 126.77 126.60 126.10 126.00 125.85 125.81 125.73 125.61 86.09 78.81 77.21 75.39 74.44 74.02 72.70 71.85 70.92 70.43 62.61 17.98 17.95 17.69 11.97

95



BRUKER

NAME	01267
EXPNO	2
PROCNO	1
Date_	20111019
Time	16.53
INSTRUM	spect
PROBHD	5 mm PABBO BB-
PULPROG	zgpg30
TD	65536
SOLVENT	CDCl3
NS	321
DS	4
SWH	24038.461 Hz
FIDRES	0.366798 Hz
AQ	1.3631988 sec
RG	203
DW	20.800 usec
DE	6.50 usec
TE	297.0 K
D1	3.00000000 sec
D11	0.03000000 sec
ID0	1
======== CHANNEL f1 ========	
NUC1	13C
P1	10.00 usec
PL1	-1.59 dB
PL1W	61.07626343 W
SFO1	100.6479773 MHz
======== CHANNEL f2 ========	
CPDPRG2	waltz16
NUC2	1H
PCPD2	75.00 usec
PL2	-1.00 dB
PL12	13.39 dB
PL13	20.00 dB
PL2W	11.09959412 W
PL12W	0.40393090 W
PL13W	0.08816721 W
SFO2	400.2316009 MHz
SI	32768
SF	100.6379177 MHz
WDW	EM
SSB	0
LB	1.00 Hz
GB	0
PC	1.40

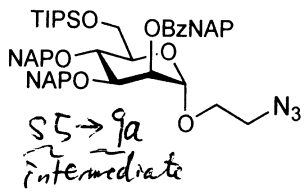


BRUKER

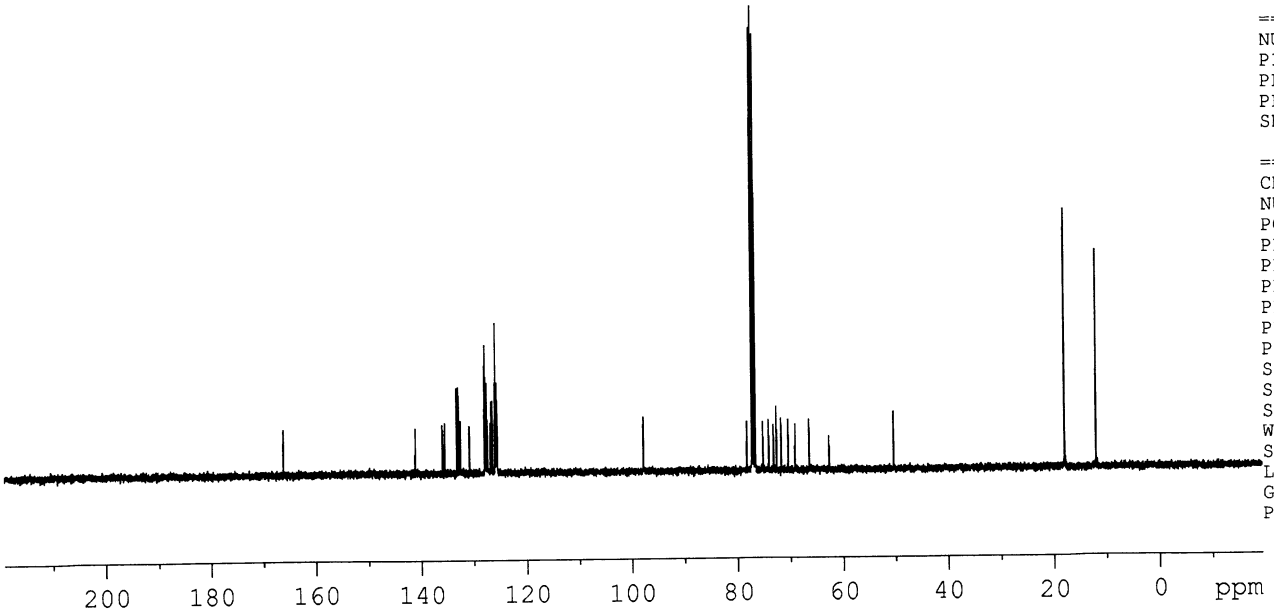
NAME	01283p
EXPNO	1
PROCNO	1
Date_	20110929
Time	11.49
INSTRUM	spect
PROBHD	5 mm PABBO BB-
PULPROG	zg30
TD	65536
SOLVENT	CDCl3
NS	16
DS	2
SWH	8223.685 Hz
FIDRES	0.125483 Hz
AQ	3.9846387 sec
RG	57
DW	60.800 usec
DE	6.50 usec
TE	296.6 K
D1	2.00000000 sec
TD0	1
======== CHANNEL f1 ========	
NUC1	1H
P1	14.31 usec
PL1	-1.00 dB
PL1W	11.09959412 W
SFO1	400.2324716 MHz
SI	32768
SF	400.2300258 MHz
WDW	EM
SSB	0
LB	0.30 Hz
GB	0
PC	1.00

01283p

166.21 141.18 135.99 135.92 135.54 133.27 133.20 132.93 132.91 132.56 130.87 128.04 128.02 128.01 127.88 127.84 127.65 127.61 127.58 127.46 126.92 126.71 126.53 126.06 125.99 125.95 125.90 125.75 125.73 125.60 97.81 78.25 75.23 74.10 73.23 72.67 71.78 70.43 69.10 66.48 50.37 17.97 17.96 11.97



S5 → 9a
intermediate



BRUKER

NAME	01283p
EXPNO	2
PROCNO	1
Date_	20110927
Time	17.10
INSTRUM	spect
PROBHD	5 mm PABBO BB-
PULPROG	zgpg30
TD	65536
SOLVENT	CDCl3
NS	161
DS	4
SWH	24038.461 Hz
FIDRES	0.366798 Hz
AQ	1.3631988 sec
RG	203
DW	20.800 usec
DE	6.50 usec
TE	297.1 K
D1	3.00000000 sec
D11	0.03000000 sec
TD0	1
======== CHANNEL f1 ========	
NUC1	13C
P1	10.00 usec
PL1	-1.59 dB
PL1W	51.07626343 W
SFO1	100.6479773 MHz
======== CHANNEL f2 ========	
CPDPRG2	waltz16
NUC2	1H
PCPD2	75.00 usec
PL2	-1.00 dB
PL12	13.39 dB
PL13	20.00 dB
PL2W	11.09959412 W
PL12W	0.40393090 W
PL13W	0.08816721 W
SFO2	400.2316009 MHz
SI	32768
SF	100.6379214 MHz
WDW	EM
SSB	0
LB	1.00 Hz
GB	0
PC	1.40