

Supporting Information

Design and Synthesis of Activity Probe for Glycosidases

Chang-Sheng Tsai,^a Yaw-Kuen Li,^b and Lee-Chiang Lo^{a,*}

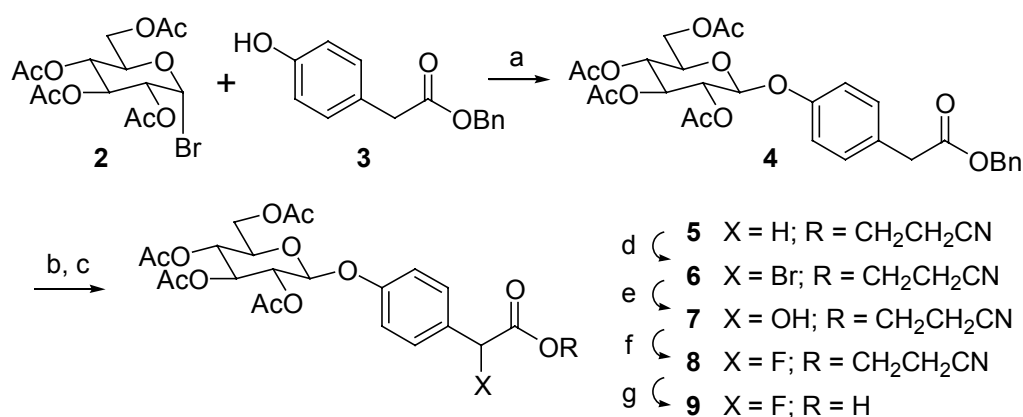
^aDepartment of Chemistry, National Taiwan University
Taipei 106, TAIWAN

^bDepartment of Applied Chemistry, The National Chiao Tung University
Hsinchu 300, TAIWAN

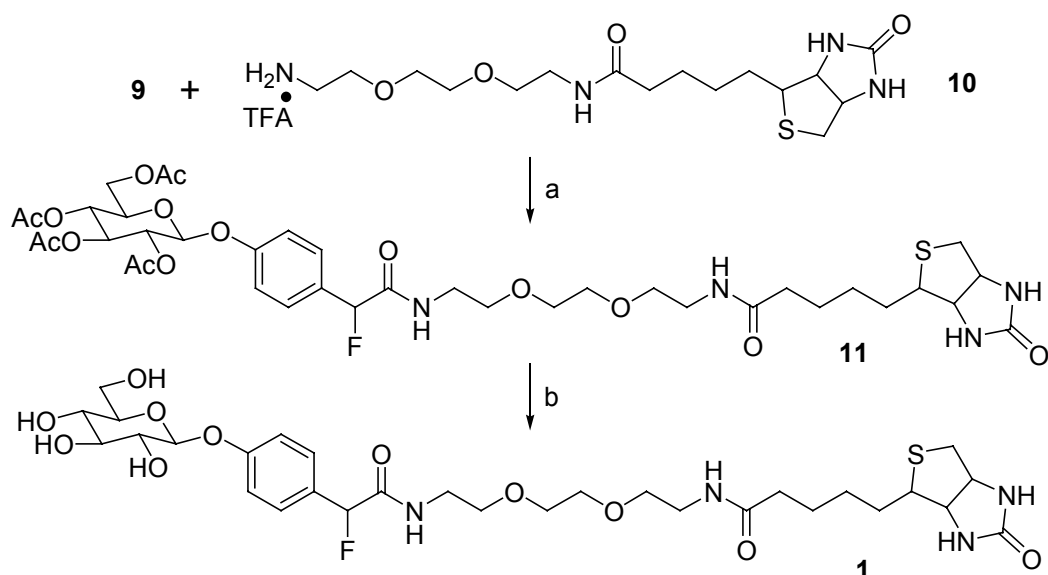
lclo@ccms.ntu.edu.tw

Experimental procedures and characterization, including copies of ¹H and ¹³C NMR, for compounds **1**, **4–9**, and **11** as well as conditions for the labeling of β-glucosidase with probe **1** are included.

Scheme 1^a



^a(a) AgOTf, CH₂Cl₂ (67%); (b) H₂, Pd/C (91%); (c) DCC, DMAP, HOCH₂CH₂CN, CH₂Cl₂ (95%); (d) NBS, CCl₄, hv (92%); (e) AgNO₃, acetone/H₂O (55%); (f) DAST, CH₂Cl₂ (85%); (g) DBU, CH₂Cl₂ (95%).

Scheme 2^a

^a(a) DCC, HOBT, TEA (83%); (b) Na₂CO₃, MeOH (92%).

Experimental:

General methods. All reagents and starting materials were obtained from commercial suppliers (Acros, Aldrich and Merck) and were used without further purification. IR spectra were recorded on a Nicolet 550 series II spectrometer. ¹H and ¹³C NMR were recorded using a Bruker AC-300 or Bruker Avance 400 spectrometer. The proton and carbon chemical shifts are given in ppm using CDCl₃ (δ_H 7.24 and 77.0) as internal standard. High resolution mass spectra were recorded with a JEOL-102A mass spectrometer. Analytical TLC (silica gel, 60F-54, Merck) and spots were visualized under UV light and/or phosphomolybdic acid-ethanol. Column chromatography was performed with Kieselgel 60 (70-230 mesh) silica gel (Merck). Melting points are reported without correction.

[4-(Tetra-*O*-acetyl-β-D-glucopyranosyloxy)-phenyl]-acetic acid benzyl ester (**4**):

Benzyl *p*-hydroxyphenylacetate **3** (265 mg, 1.1 mmol) and AgOTf (421 mg, 1.6 mmol) in the presence of 3A molecular sieves was dissolved/suspended in 10 mL of CH₂Cl₂. The mixture was cooled in an ice bath and a solution of tetra-*O*-acetyl-glucopyranosyl bromide **2** (675 mg, 1.6 mmol) in 5 mL of CH₂Cl₂ was slowly added. The reaction mixture was kept in the dark for 2 h. It was quenched by adding 1 mL of DIEA. The yellow precipitate was filtered off. The filtrate was concentrated to dryness and the residue was subjected to silica gel column chromatography eluted with *n*-hexane/EtOAc (7/3). The β-glucoside **4** (420 mg) was obtained in 67% yield. *R*_f = 0.45 (*n*-hexane/EtOAc = 1/1), mp 75–78°C. ¹H NMR (CDCl₃, 400 MHz) δ 7.34–7.25 (m, 5 H), 7.17 (d, *J* = 6.6 Hz, 2 H), 6.92 (d, *J* = 6.6 Hz, 2H), 5.29–5.21 (m, 2

H, H-2 + H-4), 5.11 (dd, $J = 11.7, 9.9$ Hz, 1H, H-3), 5.08 (s, 2H), 5.03 (d, $J = 7.6$ Hz, 1 H, H-1), 4.26 (dd, $J = 12.6, 5.3$ Hz, 1 H, H-6), 4.13 (dd, $J = 12.6, 2.4$ Hz, 1 H, H-6'), 3.83 (m, 1 H, H-5), 3.59 (s, 2 H), 1.99 (s, 3 H), 1.98 (s, 3 H), 1.97 (s, 3 H), 1.96 (s, 3 H). ^{13}C NMR (CDCl_3 , 100 MHz) δ 171.3 (C), 170.4 (C), 170.1 (C), 169.3 (C), 169.2 (C), 155.9 (C), 135.7 (C), 130.4 (CH), 128.8 (CH), 128.6 (CH), 128.5 (CH), 128.1 (C), 117.0 (CH), 99.1 (CH), 72.6 (CH), 71.9 (CH), 71.1 (CH), 68.2 (CH), 66.6 (CH₂), 61.8 (CH₂), 40.3 (CH₂), 20.6 (CH₃), 20.5 (CH₃), 20.5 (CH₃), 20.5 (CH₃). IR (KBr): 3038, 2964, 2945, 1760, 1512, 1450, 1369, 1239, 1140, 1047, 978, 922 cm^{-1} . HRMS calcd for $\text{C}_{29}\text{H}_{33}\text{O}_{12}$ ($M + 1$)⁺ 573.1972, found 573.1969.

[4-(Tetra-*O*-acetyl- β -D-glucopyranosyloxy)-phenyl]-acetic acid 2-cyanoethyl ester (5): To a solution of compound **4** (85 mg, 0.15 mmol) in 5 mL of EtOAc and a few drops of MeOH was added a spatula of 10% Pd/C. The system was evacuated and filled with H₂ three times. It was kept under H₂ atmosphere with a balloon and stirred at rt for 1 h. The Pd/C catalyst was filtered off through Celite 535, and the filtrate concentrated. The acid intermediate (65 mg, 91%) was obtained as a white foam after silica gel column chromatography eluted with MeOH/ CHCl_3 (5/95). $R_f = 0.51$ (MeOH/ $\text{CHCl}_3 = 1/9$). ^1H NMR (CDCl_3 , 400 MHz) δ 7.17 (d, $J = 6.7$ Hz, 2 H), 6.92 (d, $J = 6.7$ Hz, 2H), 5.29-5.20 (m, 2 H, H-2 + H-4), 5.13 (dd, $J = 9.7, 9.3$ Hz, 1 H, H-3), 5.03 (d, $J = 7.5$ Hz, 1 H, H-1), 4.25 (dd, $J = 12.3, 5.3$ Hz, 1 H, H-6), 4.13 (dd, $J = 12.3, 2.4$ Hz, 1 H, H-6'), 3.82 (m, 1 H), 3.56 (s, 2 H), 2.03 (s, 3 H), 2.01 (s, 3 H), 1.99 (s, 3 H), 1.98 (s, 3 H). ^{13}C NMR (CDCl_3 , 100 MHz) δ 177.1 (C), 170.6 (C), 170.2 (C), 169.4 (C), 169.3 (C), 156.0 (C), 130.5 (CH), 128.2 (C), 117.0 (CH), 99.0 (CH), 72.6 (CH), 71.9 (CH), 71.1 (CH), 68.2 (CH), 61.9 (CH₂), 40.0 (CH₂), 20.6 (CH₃), 20.5 (CH₃), 20.5 (CH₃), 20.5 (CH₃); IR (neat): 3389, 1760, 1523, 1372, 1227, 1037 cm^{-1} . HRMS calcd for $\text{C}_{22}\text{H}_{27}\text{O}_{12}$ ($M + 1$)⁺ 483.1503; found 483.1499. To a solution of the acid intermediate (575 mg, 1.19 mmol), 3-hydroxypropionitrile (98 μL , 1.43 mmol) and DMAP (15 mg, 0.11 mmol) in 50 mL of CH_2Cl_2 cooled in an ice bath was slowly injected 4 mL of DCC solution (0.45 M in CH_2Cl_2). The reaction mixture was allowed to warm to rt and stirred for 10 h. The white precipitate was filtered off and the filtrate concentrated. The oil residue was dissolved in 60 mL of EtOAc and washed with 5% citric acid ($\times 3$), 10% NaHCO_3 ($\times 3$) and brine. The organic phase was dried over anhydrous Na_2SO_4 , filtered and concentrated. The resulting oil was chromatographed on silica gel (EtOAc/ $\text{CHCl}_3 = 2/8$) to give compound **5** as an oil (606 mg, 95%). $R_f = 0.33$ (EtOAc/ $\text{CHCl}_3 = 3/7$). ^1H NMR (CDCl_3 , 400 MHz) δ 7.18 (dd, $J = 6.6, 2.1$ Hz, 2 H), 6.92 (dd, $J = 6.6, 2.1$ Hz, 2 H), 5.28-5.19 (m, 2 H, H-2 + H-4), 5.12 (dd, $J = 9.9, 9.3$ Hz, 1H, H-3), 5.03 (d, $J = 7.6$ Hz, 1 H, H-1), 4.26-4.22 (m, 3 H), 4.12 (dd, $J = 12.3, 2.5$ Hz, 1 H, H-6), 3.80 (m, 1 H,

H-5), 3.59 (s, 2 H), 2.66 (t, $J = 6.3$ Hz, 2 H), 2.03 (s, 3 H), 2.01 (s, 3H), 2.00 (s, 3 H), 1.99 (s, 3 H). ^{13}C NMR (CDCl_3 , 100 MHz) δ 170.9 (C), 170.5 (C), 170.1 (C), 169.3 (C), 169.2 (C), 156.1 (C), 130.4 (CH), 128.1 (C), 117.1 (CH), 116.6 (C), 99.0 (CH), 72.6 (CH), 71.9 (CH), 71.1 (CH), 68.2 (CH), 61.8 (CH_2), 59.0 (CH_2), 39.9 (CH_2), 20.6 (CH_3), 20.5 (CH_3), 20.5 (CH_3), 20.5 (CH_3), 17.9 (CH_2). IR (neat): 3336, 2936, 2850, 2259, 1747, 1517, 1424, 1228, 1155, 1037 cm^{-1} . HRMS calcd for $\text{C}_{25}\text{H}_{30}\text{NO}_{12}$ ($M + 1$)⁺ 536.1768; found 536.1772.

[4-(Tetra-*O*-acetyl- β -D-glucopyranosyloxy)-phenyl]-bromoacetic acid 2-cyanoethyl ester (6): To a solution of compound **5** (100 mg, 0.19 mmol) in 15 mL of CCl_4 was added NBS (36.6 mg, 0.21 mmol) and a catalytic amount of AIBN. The reaction flask was equipped with a condenser, and purged and filled with N_2 three times. It was irradiated with a 100W tungsten lamp for 1 h until white precipitate formed. The precipitate was filtered off and the filtrate concentrated. It was subjected to silica gel column chromatography eluted with EtOAc/ CHCl_3 (2/8) to give the ester product **6** (105 mg, 92%) as a white foam. $R_f = 0.33$ (EtOAc/ $\text{CHCl}_3 = 3/7$). ^1H NMR (CDCl_3 , 400 MHz) δ 7.47 (d, $J = 8.6$ Hz, 2 H), 6.95 (d, $J = 8.6$ Hz, 2 H), 5.33 (s, 1 H), 5.27-5.23 (m, 2 H, H-2 + H-4), 5.18-5.07 (m, 2 H, H-1 + H-3), 4.35-4.31 (m, 2 H), 4.24 (dd, $J = 11.8, 5.3$ Hz, 1 H, H-6), 4.15 (dd, $J = 11.8, 0.8$ Hz, 1H, H-6'), 3.84 (m, 1 H, H-5), 2.70 (t, $J = 6.3$ Hz, 2 H), 2.04 (s, 3H), 2.01 (s, 3 H), 2.00 (s, 3 H), 2.00 (s, 3 H). ^{13}C NMR (CDCl_3 , 100 MHz) δ 170.5 (C), 170.1 (C), 169.3 (C), 169.2 (C), 167.7 (C), 157.4 (C), 130.2 (CH), 129.7 (C), 117.1 (CH), 116.2 (C), 98.5 (CH), 72.5 (CH), 72.1 (CH), 71.0 (CH), 68.1 (CH), 61.8 (CH_2), 60.3 (CH_2), 45.0 (CH), 20.6 (CH_3), 20.5 (CH_3), 20.5 (CH_3), 20.5 (CH_3), 17.7 (CH_2). IR (neat): 2982, 1753, 1510, 1385, 1221, 1037 cm^{-1} . HRMS calcd for $\text{C}_{25}\text{H}_{29}^{79}\text{BrNO}_{12}$ ($M + 1$) 614.0873; found 614.0872.

[4-(Tetra-*O*-acetyl- β -D-glucopyranosyloxy)-phenyl]-hydroxyacetic acid 2-cyanoethyl ester (7): Compound **6** (150.0 mg, 0.24 mmol) was dissolved in 5 mL of acetone. A solution of AgNO_3 (83.0 mg, 0.48 mmol) and Ag_2CO_3 (33.1 mg, 0.12 mmol) in 5 mL of H_2O was added. A dark greenish precipitate was formed immediately. The reaction mixture was stirred at rt for 30 min. It was filtered and the filtrate was concentrated to dryness. The residual oil was dissolved in 50 mL of EtOAc and washed with 5% citric acid ($\times 3$), 10% NaHCO_3 ($\times 3$) and brine. The organic phase was dried over anhydrous Na_2SO_4 , filtered and concentrated. The resulting oil was chromatographed on silica gel (EtOAc/ $\text{CHCl}_3 = 4/6$) to give compound **7** as a white foam (74.0 mg, 55%). $R_f = 0.40$ (EtOAc/ $\text{CHCl}_3 = 6/4$). ^1H NMR (CDCl_3 , 400 MHz) δ 7.30 (d, $J = 8.4$ Hz, 2 H), 6.91 (d, $J = 8.4$ Hz, 2 H),

5.22-5.02 (m, 5 H), 4.28 (m, 1 H), 4.19-4.15 (m, 2 H), 4.07 (dd, $J = 12.2, 1.7$ Hz, 1 H, H-6), 3.83 (m, 1 H, H-5), 3.74 (d, $J = 3.3$ Hz, 1 H, OH), 2.61-2.54 (m, 2 H), 1.98 (s, 3 H), 1.96 (s, 3 H), 1.96 (s, 3 H), 1.94 (s, 3 H). ^{13}C NMR (CDCl_3 , 100 MHz) δ 172.6 (C), 170.4 (C), 170.0 (C), 169.3 (C), 169.2 (C), 156.8 (C), 132.5 (C), 127.8 (CH), 116.8 (CH), 116.3 (C), 98.6 (CH), 72.4 (CH), 72.1 (CH), 71.7 (CH), 70.8 (CH), 68.0 (CH), 61.7 (CH_2), 59.7 (CH_2), 20.5 (CH_3), 20.4 (CH_3), 20.4 (CH_3), 20.4 (CH_3), 17.6 (CH_2). IR (neat): 3481, 2969, 2259, 1746, 1517, 1372, 1227, 1182, 1044 cm^{-1} . HRMS calcd for $\text{C}_{25}\text{H}_{30}\text{NO}_{13}$ ($M + 1$) 552.1718; found 552.1729.

[4-(Tetra-*O*-acetyl- β -D-glucopyranosyloxy)-phenyl]-fluoroacetic acid 2-cyanoethyl ester (8): To an ice-cooled solution of compound **7** (130.0 mg, 0.23 mmol) in 10 mL of CH_2Cl_2 was added 70 μL (0.46 mmol) of DAST. The reaction mixture was allowed to warm to rt and stirred for 15 h. It was quenched by adding small amount of silica gel and 1.5 mL of MeOH. It was concentrated and the fluorinated product **8** (110.0 mg, 85%) was obtained as a white foam after silica gel column chromatography eluted with EtOAc/ CHCl_3 (2/8). $R_f = 0.33$ (EtOAc/ $\text{CHCl}_3 = 3/7$). ^1H NMR (CDCl_3 , 400 MHz) δ 7.35 (d, $J = 8.5$ Hz, 2 H), 6.97 (d, $J = 8.5$ Hz, 2 H), 5.74 (d, $J = 47.2$ Hz, 1 H, CHF), 5.27-5.18 (m, 2 H), 5.12-5.06 (m, 2 H), 4.34 (m, 1 H), 4.31-4.17 (m, 2 H), 4.10 (d, $J = 12.3$ Hz, 1 H, H-6), 3.84 (m, 1 H, H-5), 2.71-2.61 (m, 2 H), 2.00 (s, 3 H), 1.99 (s, 3 H), 1.98 (s, 3 H), 1.97 (s, 3 H). ^{13}C NMR (CDCl_3 , 100 MHz) δ 170.3 (C), 170 (C), 169.2 (C), 169.1 (C), 167.8 (d, $J = 28.8$ Hz, C), 157.7 (C), 128.3 (d, $J = 20.5$ Hz, C), 128.2 (CH), 117.1 (CH), 116.1 (C), 98.5 (CH), 88.4 (d, $J = 185.0$ Hz, CHF), 72.4 (CH), 72.0 (CH), 70.9 (CH), 68.0 (CH), 61.7 (CH_2), 59.6 (CH_2), 20.5 (CH_3), 20.4 (CH_3), 20.4 (CH_3), 20.4 (CH_3), 17.7 (CH_2). ^{19}F NMR (CDCl_3) δ -179.0 (d, $J = 49.6$ Hz), -179.3 (d, $J = 50.0$ Hz). IR (neat): 2250, 1753, 1523, 1379, 1247, 1037 cm^{-1} . HRMS calcd for $\text{C}_{25}\text{H}_{29}\text{O}_{12}\text{NF}$ ($M + 1$) 554.1674; found 554.1678.

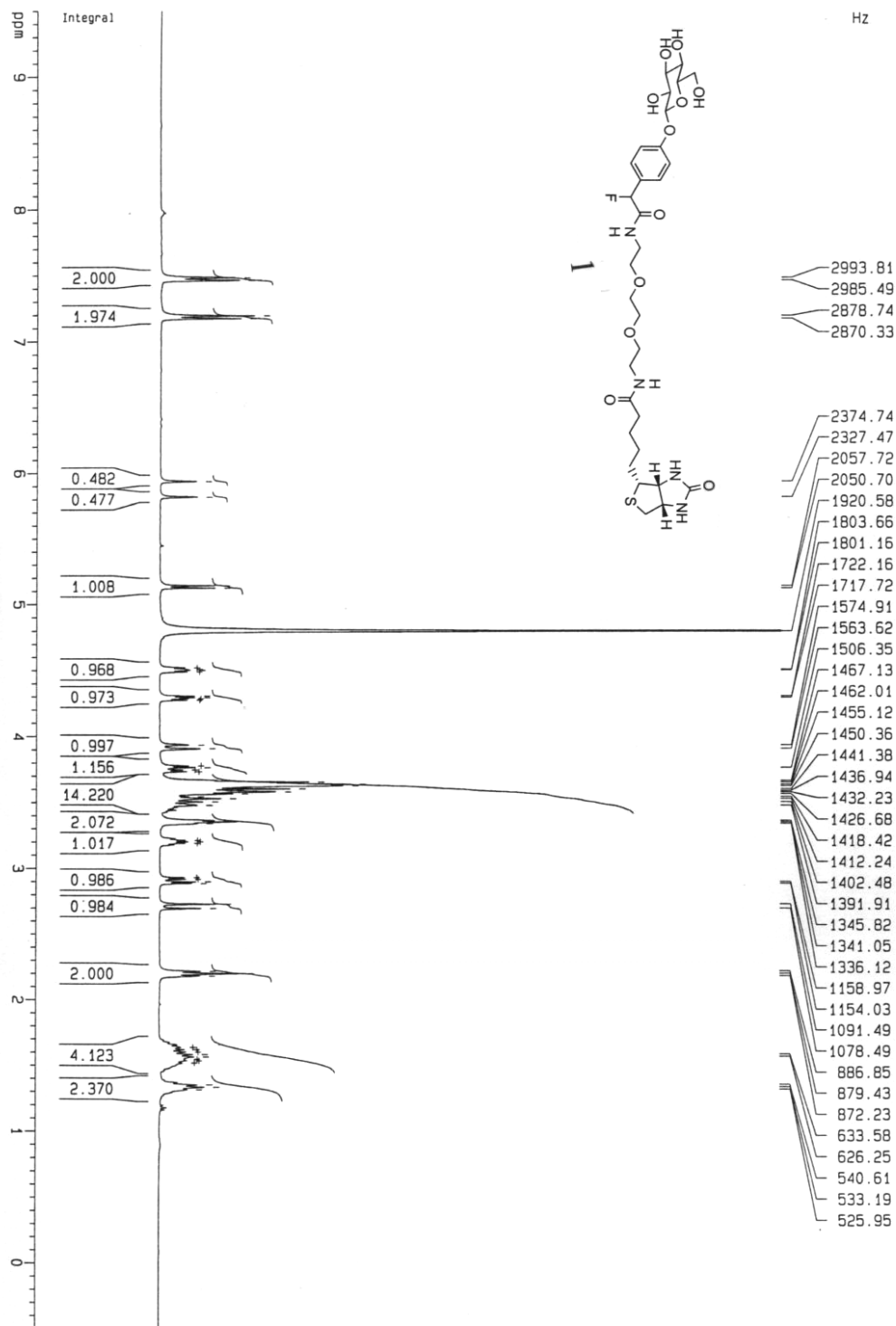
Triethylammonium [4-(Tetra-*O*-acetyl- β -D-glucopyranosyloxy)-phenyl]-fluoroacetate (9): To a solution of the fluoride **8** (130.0 mg, 0.23 mmol) in 5 mL of CH_2Cl_2 was added 50 μL (0.46 mmol) of DBU. The reaction mixture was stirred at rt for 30 min. EtOAc (50 mL) was then added, and it was washed with 5% citric acid ($\times 3$), H_2O ($\times 1$) and brine ($\times 1$). The organic phase was dried over anhydrous Na_2SO_4 and filtered. Since the α -fluoroacid was not stable, it was stored as the triethylammonium salt. This was achieved by adding 1 mL of TEA to the filtrate. The triethylammonium salt **9** was obtained as a solid (103.0 mg, 95%) after concentration. $R_f = 0.25$ (MeOH/ $\text{CHCl}_3 = 3/7$), mp 111–113°C. ^1H NMR (CDCl_3 , 400 MHz) δ 7.38 (d, $J = 8.5$ Hz, 2 H), 6.84 (d, $J = 8.5$ Hz, 2 H), 5.53 (d, $J = 50.4$ Hz,

1 H, CHF), 5.23-5.14, (m, 2 H), 5.06 (dd, $J = 11.2, 9.5$ Hz, 1 H, H-3), 4.98 (d, $J = 7.7$ Hz, 1 H, H-1), 4.97 (d, $J = 7.7$ Hz, 1 H, H-1'), 4.19 (dd, $J = 12.3, 5.5$ Hz, 1 H, H-6), 4.06 (dd, $J = 12.3, 2.4$ Hz, 1 H, H-6'), 3.78 (m, 1 H, H-5), 2.97 (q, $J = 7.5$ Hz, 6 H), 1.98 (s, 3 H), 1.96 (s, 3 H), 1.96 (s, 3 H), 1.94 (s, 3 H), 1.16 (t, $J = 7.5$ Hz, 9 H). ^{13}C NMR (CDCl_3 , 100 MHz) δ 174.2 (d, $J = 22.0$ Hz, C), 170.4 (C), 170.0 (C), 169.2 (C), 169.1 (C), 156.6 (C), 133.1 (d, $J = 20.5$ Hz, C), 128.0 (CH), 116.6 (CH), 99.0 (CH), 90.8 (d, $J = 183.6$ Hz, CHF), 72.5 (CH), 71.8 (CH), 71.0 (CH), 68.1 (CH), 61.8 (CH_2), 44.8 (CH_2), 20.5 (CH_3), 20.4 (CH_3), 20.4 (CH_3), 20.4 (CH_3), 8.2 (CH_3). ^{19}F NMR (CDCl_3) δ -170.2 (d, $J = 53.6$ Hz), -170.6 (d, $J = 53.6$ Hz). IR (neat): 3402, 1747, 1615, 1517, 1379, 1234, 1063, 1031 cm^{-1} . HRMS calcd for $\text{C}_{28}\text{H}_{41}\text{FNO}_{12}$ ($\text{M} + \text{TEA} + 1$) 602.2613; found 602.2624.

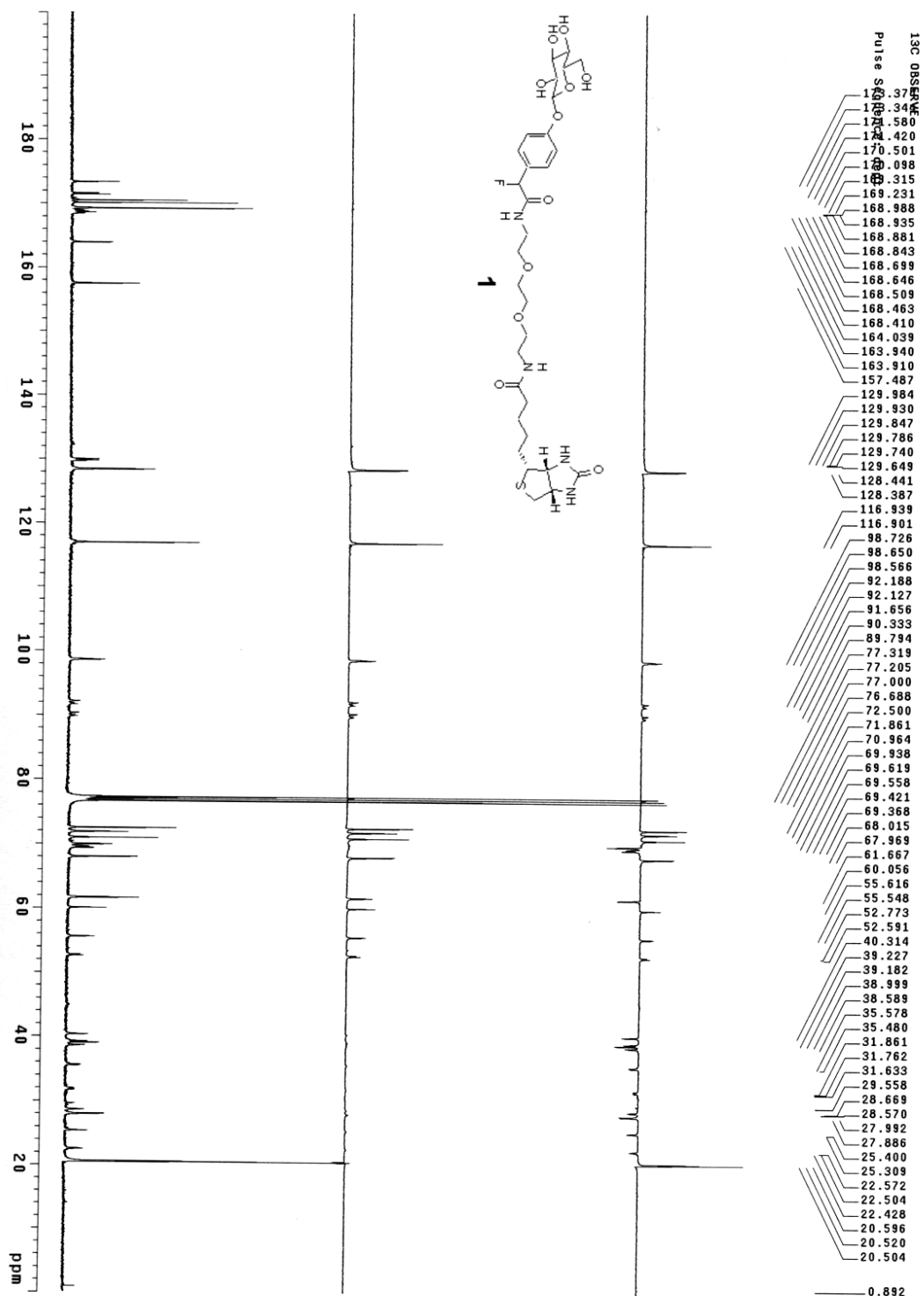
***N*-[4-(Tetra-*O*-acetyl- β -D-glucopyranosyloxy)-phenyl]-fluoroacetyl-*N'*-biotinyl-3,6-dioxaoctane-1,8-diamine (**11**):** To a solution of the triethylammonium salt of α -fluoroacid **9** (65.0 mg, 0.11 mmol) and compound **10** (61.0 mg, 0.13 mmol) in 3 mL of DMF was added HOBt (22.0 mg, 0.16 mmol), TEA (250 μL) and EDCI (31.1 mg, 0.16 mmol). The reaction mixture was stirred at rt for 24 h. DMF was then removed under high vacuum. The residual oil was dissolved in 40 mL of EtOAc. It was washed with 5% citric acid ($\times 3$), 10% NaHCO_3 ($\times 3$) and brine. The organic phase was dried over anhydrous Na_2SO_4 , filtered and concentrated. The biotinylated product **11** (77.0 mg, 83%) was obtained as an oil after silica gel column chromatography eluted with MeOH/ CHCl_3 (5/95). $R_f = 0.33$ (MeOH/ $\text{CHCl}_3 = 1/9$). ^1H NMR (CDCl_3 , 400 MHz) δ 7.34 (d, $J = 8.3$ Hz, 2 H), 7.26 (bs, 1H, NH), 6.95 (d, $J = 8.3$ Hz, 2 H), 6.72 (bs, 1 H, NH), 6.61 (bs, 1 H, NH), 5.87 (bs, 1 H, NH), 5.75 (d, $J = 48.2$ Hz, 1 H, CHF), 5.35-5.22 (m, 2 H), 5.20-5.12 (m, 2 H), 4.46 (bs, 1 H), 4.28-4.15 (m, 2 H), 4.09 (d, $J = 11.9$ Hz, 1 H, H-6), 3.92 (m, 1 H, H-5), 3.62-3.30 (m, 12 H), 3.05 (br, 1 H), 2.80 (dd, $J = 12.6, 5.0$ Hz, 1 H), 2.55 (d, $J = 12.5$ Hz, 1 H), 2.13 (dd, $J = 7.4, 4.9$ Hz, 2 H), 3.01 (s, 3 H), 1.98 (s, 3 H), 1.98 (s, 3 H), 1.97 (s, 3H), 1.68-1.48 (m, 4 H), 1.38-1.28 (m, 2 H). ^{13}C NMR (CDCl_3 , 100 MHz) δ 173.3 (C), 170.5 (C), 170.1 (C), 169.3 (C), 169.2 (C), 168.6 (d, $J = 22.1$ Hz, C), 164.2 (C), 157.5 (C), 129.8 (d, $J = 19.0$ Hz, C), 128.4 (CH), 116.8 (CH), 98.6 (CH), 91.2 (d, $J = 185.8$ Hz, CHF), 72.5 (CH), 71.9 (CH), 70.9 (CH), 70.0 (CH_2), 69.9 (CH_2), 69.8 (CH_2), 69.3 (CH_2), 68.0 (CH), 61.7 (CH_2), 61.6 (CH), 60.1 (CH), 55.6 (CH), 40.3 (CH_2), 38.9 (CH_2), 38.8 (CH_2), 35.8 (CH_2), 28.2 (CH_2), 27.9 (CH_2), 25.5 (CH_2), 20.6 (CH_3), 20.5 (CH_3), 20.5 (CH_3), 20.5 (CH_3). ^{19}F NMR (CDCl_3) δ -175.4 (d, $J = 51.2$ Hz), -175.8 (d, $J = 51.2$ Hz). IR (neat): 3350, 2929, 1753, 1694, 1661, 1569, 1550, 1517, 1379, 1228, 1050. cm^{-1} . HRMS calcd for $\text{C}_{38}\text{H}_{54}\text{FN}_4\text{O}_{15}\text{S}$ ($\text{M} + 1$) 857.3291; found 857.3297.

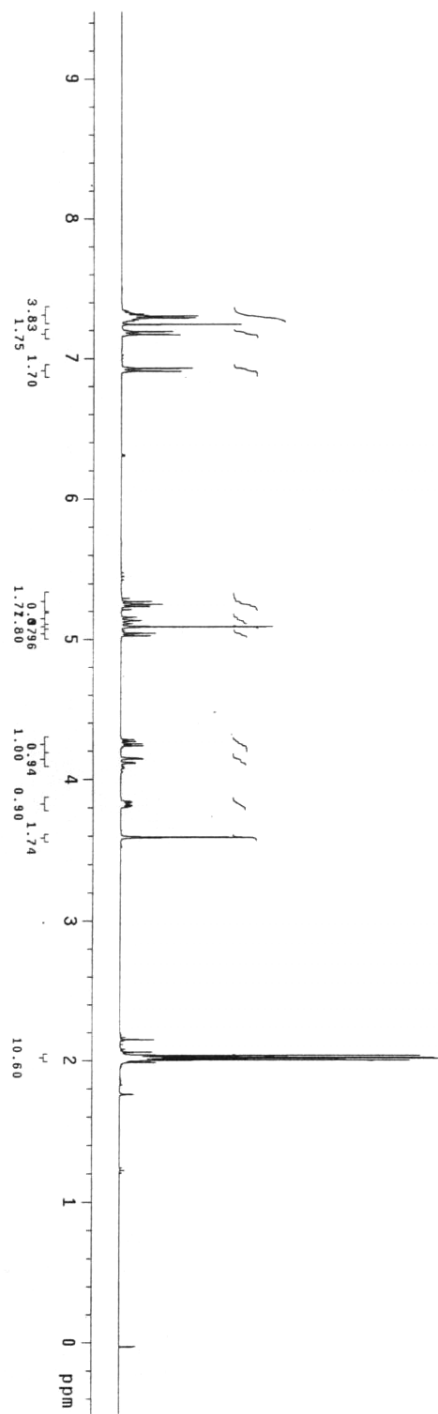
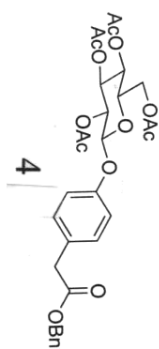
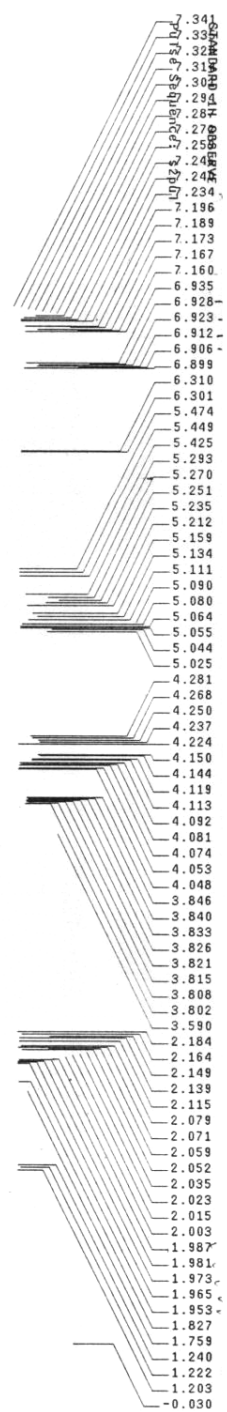
***N*-Biotinyl-*N'*-[4-(β -D-glucopyranosyloxy)-phenyl]-fluoroacetyl-3,6-dioxaoctane-1,8-diamine (**1**):** To a solution of biotinylated compound **11** (72.0 mg, 0.084 mmol) in 5 mL of MeOH was added 35.6 mg of Na₂CO₃. The mixture was stirred at rt for 1 h. It was filtered through Celite and filtrate concentrated. The target product **1** was obtained as a white foam (53.0 mg, 92%) after silica gel column chromatography eluted with MeOH/CHCl₃ (3/7). *R_f* = 0.33 (MeOH/CHCl₃ = 3/7). ¹H NMR (D₂O, 400 MHz) δ 7.47 (d, *J* = 8.4 Hz, 2 H), 7.19 (d, *J* = 8.4 Hz, 2 H), 5.88 (d, *J* = 47.3 Hz, 1 H, *CHF*), 5.13 (d, *J* = 7.0 Hz, 1 H, H-1), 4.51 (dd, *J* = 7.4, 4.9 Hz, 1 H), 4.29 (dd, *J* = 4.4, 7.8 Hz, 1 H), 3.92 (d, *J* = 11.3 Hz, 1 H), 3.67 (dd, *J* = 12.4, 5.5 Hz, 1H), 3.70-3.43 (m, 14 H), 3.35 (t, *J* = 4.8 Hz, 2 H), 3.21 (m, 1 H), 2.91 (dd, *J* = 13.0, 4.9 Hz, 1 H), 2.71 (d, *J* = 13.0 Hz, 1 H), 2.20 (t, *J* = 7.3 Hz, 2 H), 1.70-1.45 (m, 4 H), 1.38-1.27 (m, 2 H). ¹³C NMR (D₂O, 100 MHz) δ 176.7 (C), 171.3 (d, *J* = 24.0 Hz, C), 165.3 (C), 158.2 (C), 129.6 (CH), 129.3 (C), 117.1 (CH), 100.3 (CH), 91.2 (d, *J* = 183.0 Hz, *CHF*), 76.5 (CH), 75.9 (CH), 73.2 (CH), 69.8 (CH₂), 69.8 (CH₂), 69.8 (CH), 69.2 (CH₂), 69.0 (CH₂), 62.3 (CH), 60.9 (CH₂), 60.4 (CH), 55.7 (CH), 40.0 (CH₂), 39.2 (CH₂), 39.0 (CH₂), 35.7 (CH₂), 28.3 (CH₂), 28.0 (CH₂), 25.5 (CH₂). ¹⁹F NMR (D₂O) δ -169.7 (d, *J* = 49.2 Hz), -169.8 (d, *J* = 50.4 Hz). IR (KBr): 3380, 2939, 2883, 1698, 1549, 1512, 1462, 1239, 1083, 1059 cm⁻¹. HRMS calcd for C₃₀H₄₆FN₄O₁₁S (M + 1) 689.2868; found 689.2885.

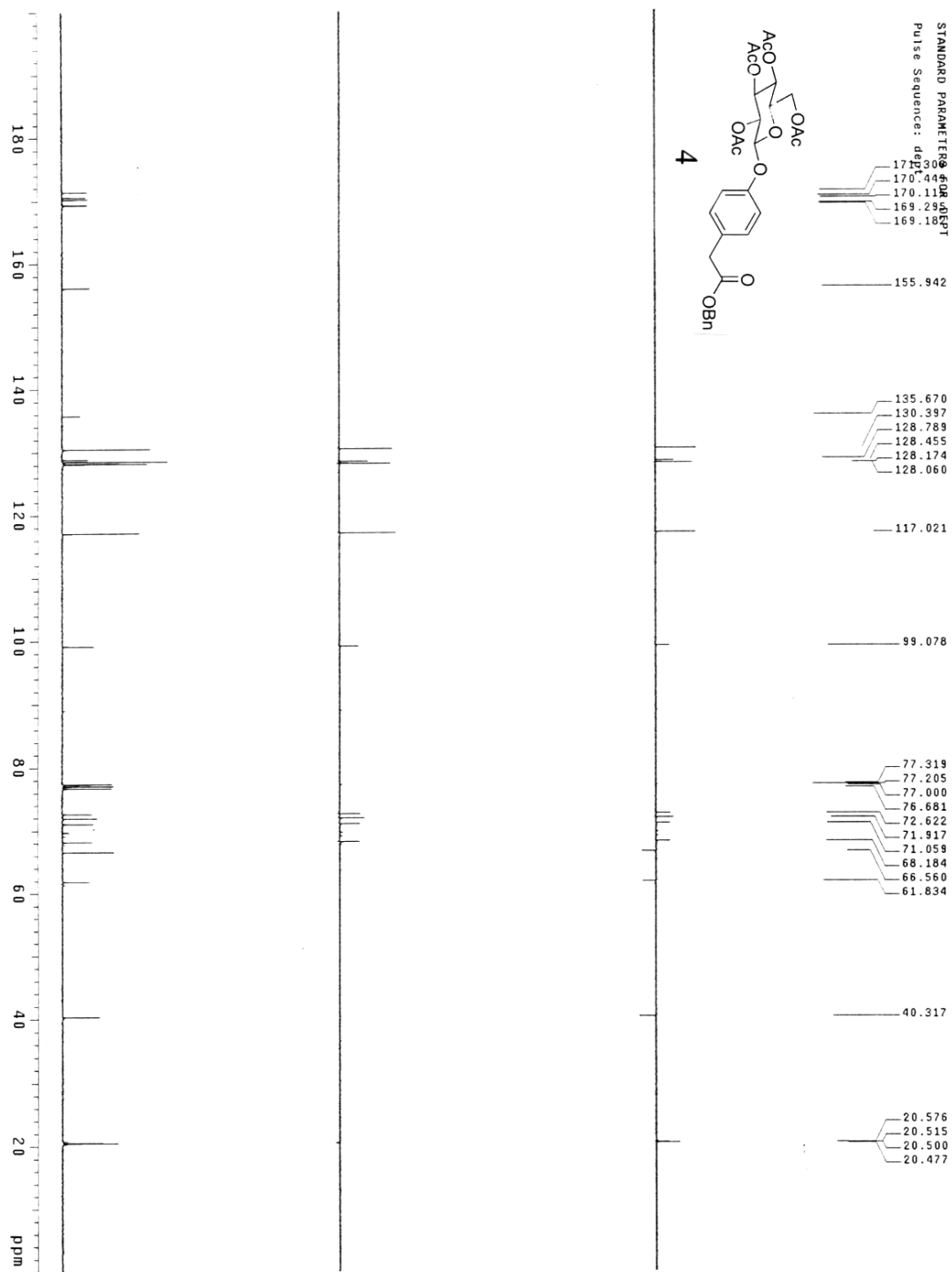
Labeling of β -glucosidase with probe **1:** Purified β -glucosidase (2.0 μ g) was incubated with 1 mM of probe **1** in 50 mM of phosphate buffer (pH 7.0) at 37°C for 30 min. The reaction products were separated by 8% SDS-polyacrylamide gel electrophoresis and transferred onto a nitrocellulose membrane. The membrane was blocked with 10% nonfat dry milk, washed with TTBS (0.05% Tween-20, 20 mM Tris pH 7.6, 137 mM NaCl), and treated with a streptavidin-horseradish peroxidase conjugate (Amersham-Pharmacia, 1:2000 dilution) in TTBS containing 1% nonfat dry milk for 1 h at 25°C. Visualization of bound streptavidin-horseradish peroxidase conjugate was achieved by treating the membrane with ECL chemiluminescence reagents (Amersham-Pharmacia) and exposed to film for 1-30 min before development.



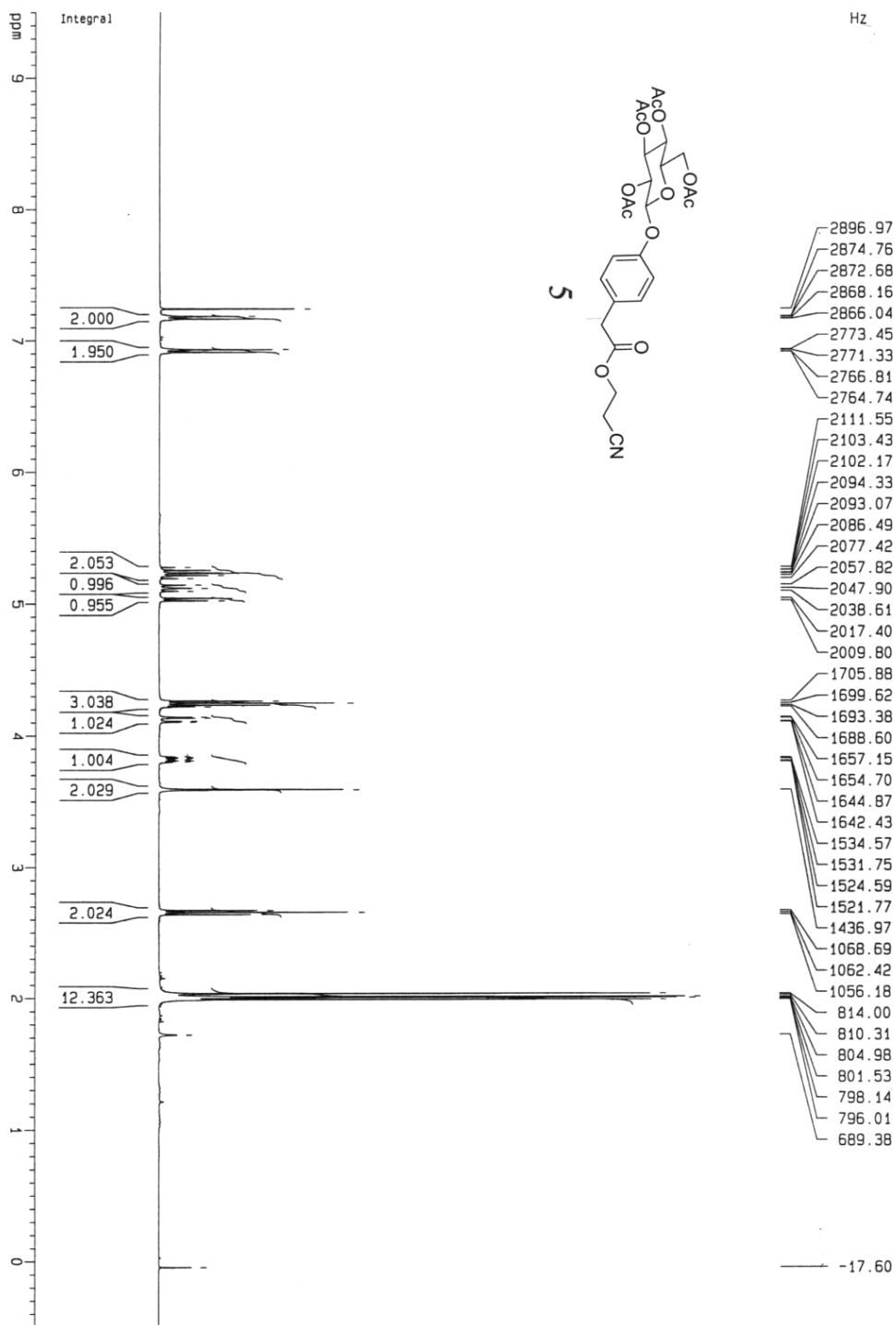
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 P1: 11.50
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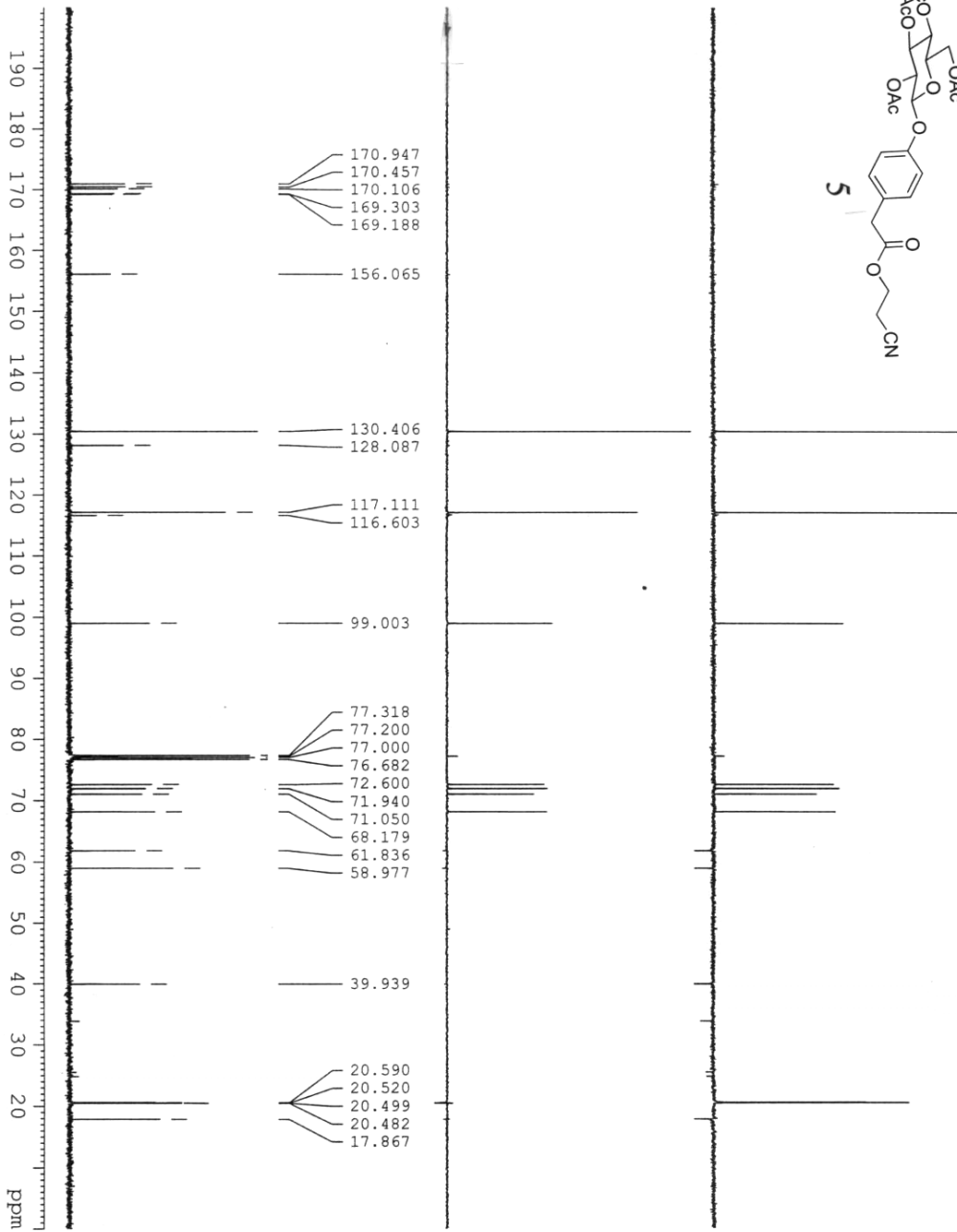
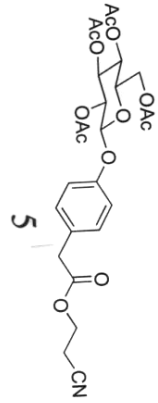




Y:038 CDCl3 011105 400MHz H1

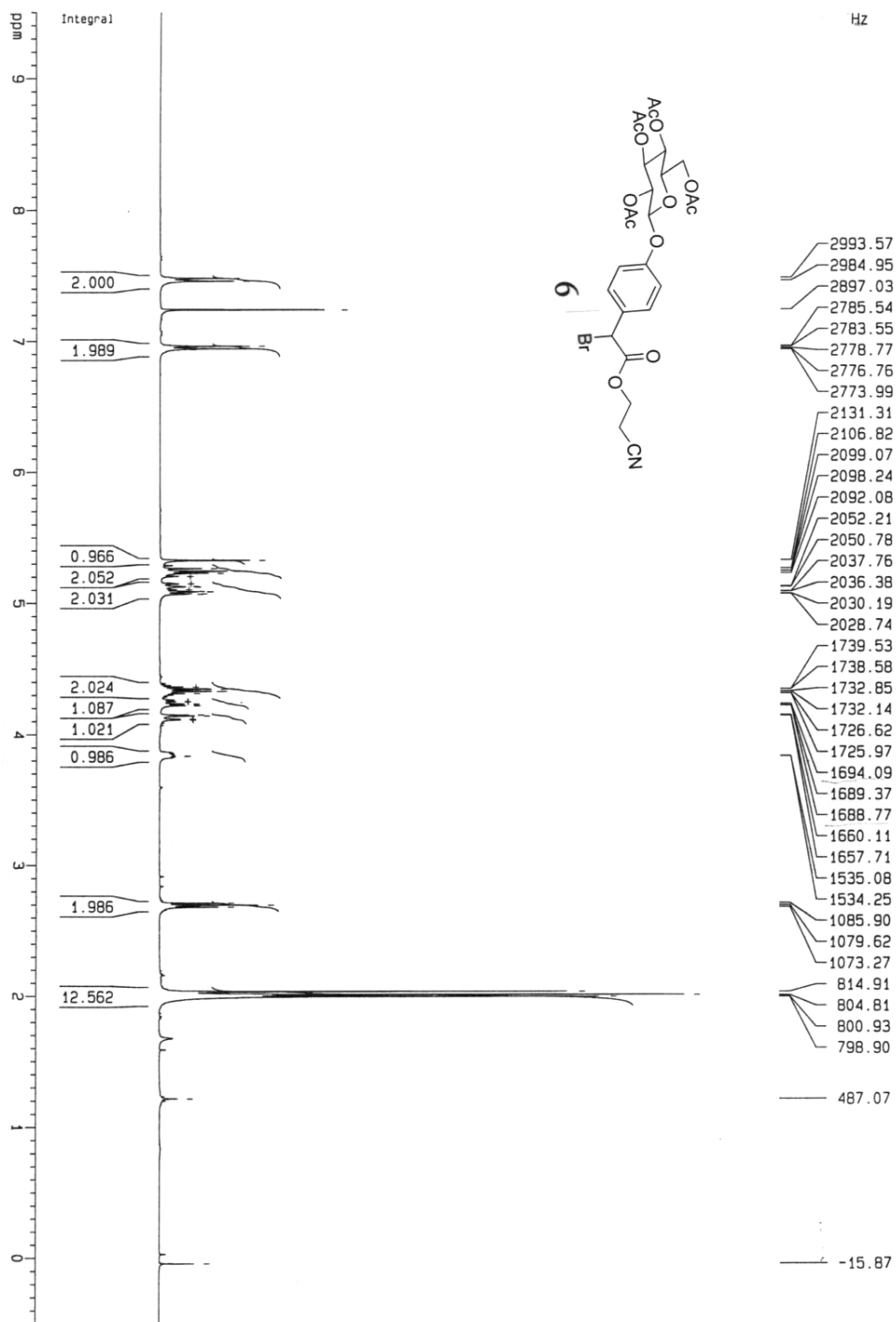


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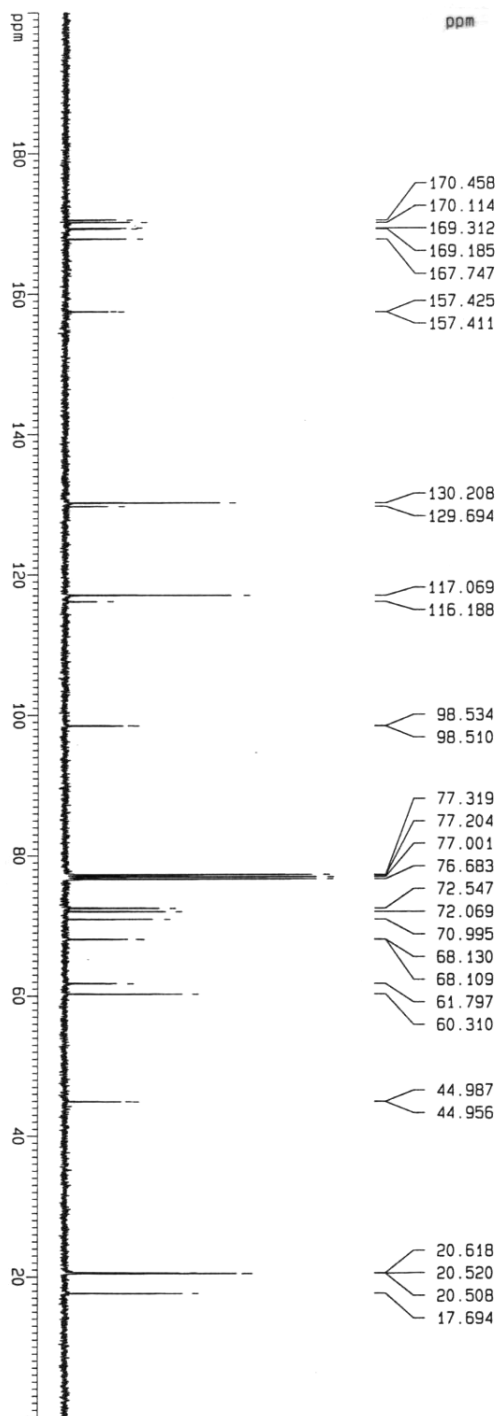
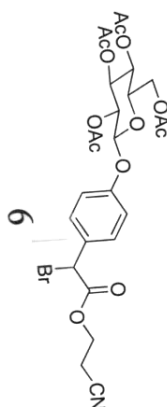
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 NS 122
 DS 4
 SWH 26178.010 Hz
 FIDRES 0.399445 Hz
 AQ 1.237768 sec
 RG 32768
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 PL1 -3.00 dB
 SFO1 100.6264025 MHz
 ===== CHANNEL f2 =====
 NUC2 1H
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 PL2 0.00 dB
 PCPDZ 88.00 usec
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Y: 039 CDC13 011105 400MHz H1



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F765 0.500
F766 0.500
F767 0.500
F768 0.500
F769 0.500
F770 0.500
F771 0.500
F772 0.500
F773 0.500
F774 0.500
F775 0.500
F776 0.500
F777 0.500
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F779 0.500
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F781 0.500
F782 0.500
F783 0.500
F784 0.500
F785 0.500
F786 0.500
F787 0.500
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F796 0.500
F797 0.500
F798 0.500
F799 0.500
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F802 0.500
F803 0.500
F804 0.500
F805 0.500
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F853 0.500
F854 0.500
F855 0.500
F856 0.500
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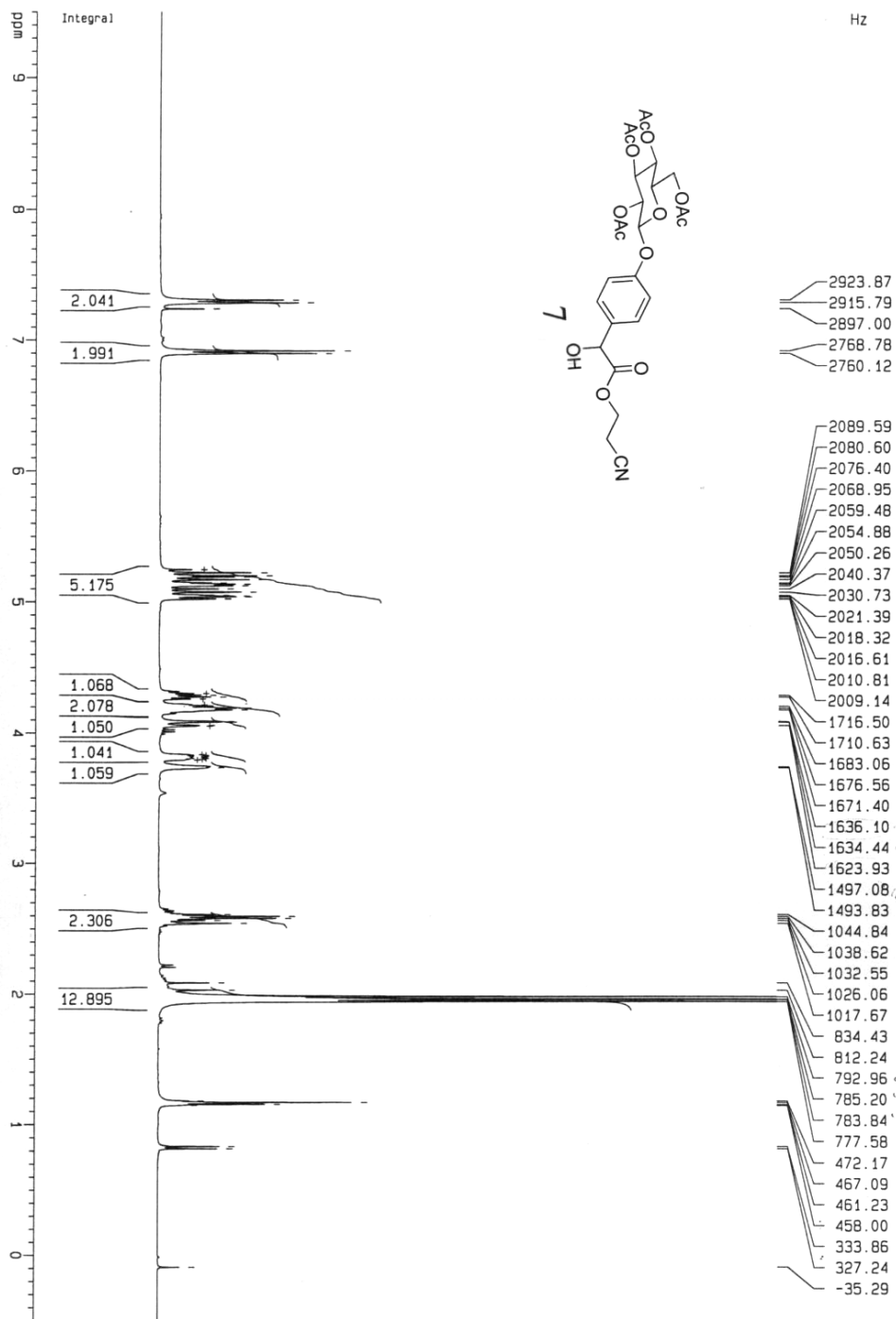
Y.039 CDCl3 011105 400MHz C13 (250p to -10p)



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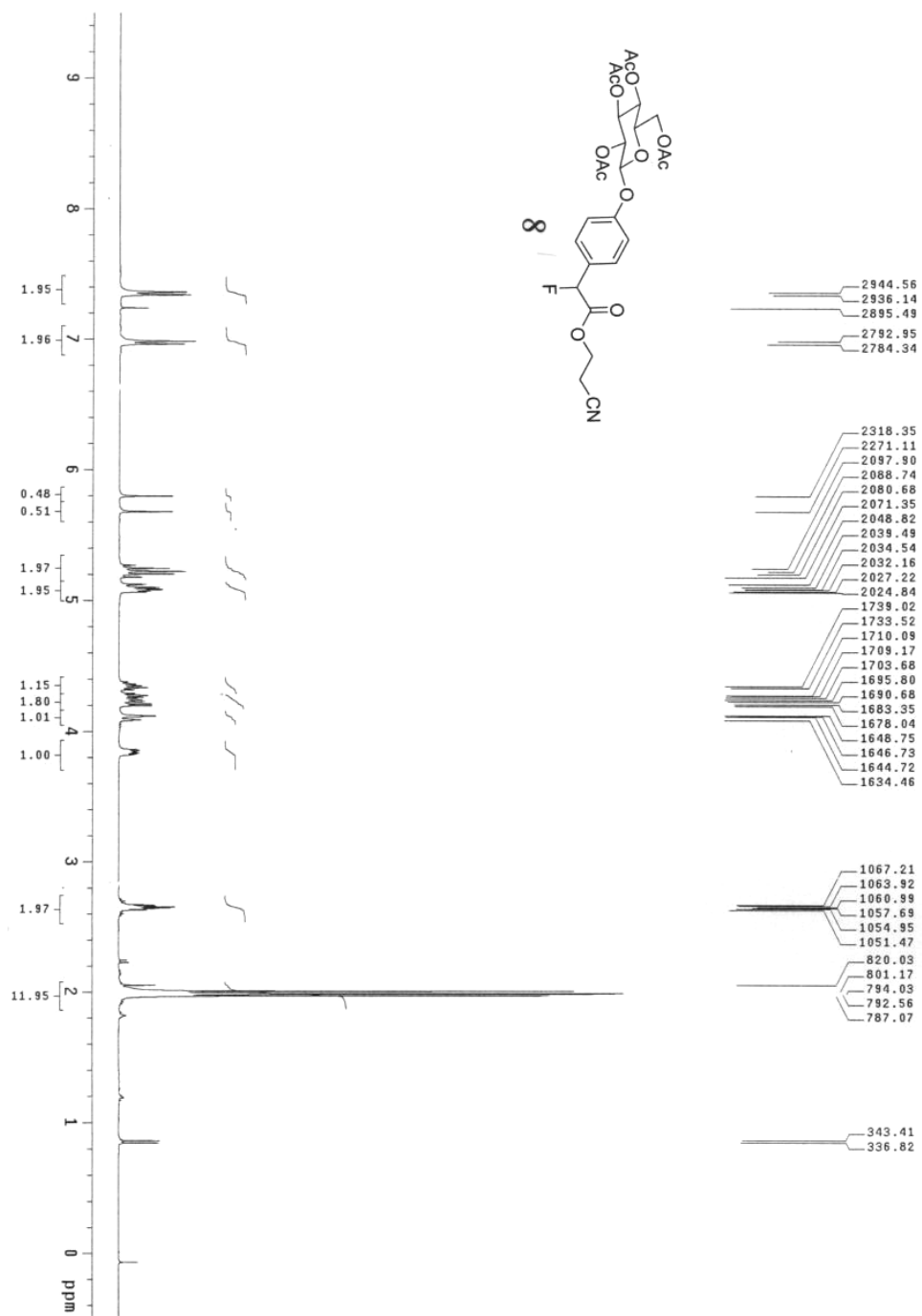
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EXPNO     1
PROCNO    1
F2 - Acquisition Parameters
Date_     20011105
Time      14.17
INSTRUM   spect
PROBHD    5 mm QNP1H
PULPROG   zgpg30
TD         65536
SOLVENT   CDCl3
NS         278
DS         2
SWH        20178.415
F2 - F1      125.76444
F2 - F2      1.2517975
AQ          32.068
RG          327.5
DE          1.00
TE          300.0
SI          2
SF          101.625000
AQ          0.03000000
AQ          0.05000000
----- CHANNEL f1 -----
NUC1       13C
P1         9.50
PC         0.00
PR         0.00
SFO1       100.626055
----- CHANNEL f2 -----
NUC2       1H
P2         1.60
PC2        0.00
PR2        0.00
SFO2       400.146401
F2 - Processing parameters
SI          32768
SF          100.626055
WDW         EM
SSB         0
LB          1.00
GB          0
PC          0.80
PC2         0.00
TO         250.000
F1P         201.26225
F2P         201.26225
RG          6.00
RFXH        8.000000
RFXH        80.418022
  
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Y:049 011226 CDCl3 400MHz H1



Current Data Parameters
 Name: Y:049011226
 ExpNO: 1
 F2 - Acquisition Parameters
 Date_: 20011226
 Time: 14.44
 Instrument: spect
 PROBHD: 5 mm QNP 1H
 NUC1: 13C
 NUC2: 13C
 PULPROG: zgpg30
 SOLVENT: Acetone
 NS: 16
 DS: 4
 SWH: 7183.508
 FIDRES: 0.218025
 AQ: 0.000125
 RG: 2.000000
 ACQ: 2.000000
 CW: 68.600
 DE: 1.00
 TE: 300.2
 D1: 1.0000000
 ===== CHANNEL f1 =====
 NUC1: 13C
 P1: 11.50
 PL1: 0.00
 SFO1: 400.130000
 F2 - Processing parameters
 SI: 32768
 SF: 400.130000
 WDM: 64
 SD: 1.00
 LR: 0.50
 GB: 0
 PC: 1.00
 1D NMR Sift parameters
 SI: 32768
 SF: 400.130000
 WDM: 64
 SD: 1.00
 LR: 0.50
 GB: 0
 PC: 1.00
 F2P: 20.50
 F1P: 102.500
 F2R: 300.134
 F1R: -0.500
 F2P: -200.127
 F1P: 0.000000
 NUC1: 13C
 NUC2: 13C





STANDARD PARAMETERS FOR ^{13}C NMR
Pulse Sequence: dept-135

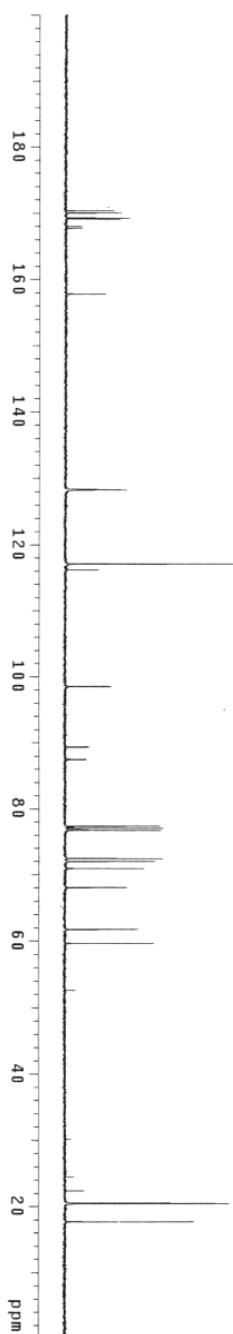
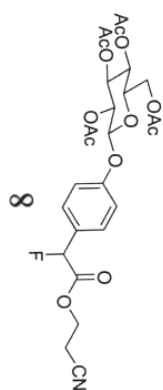
Chemical Shift (ppm)
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169.976
169.222
169.099
167.968
167.672
157.756
157.733

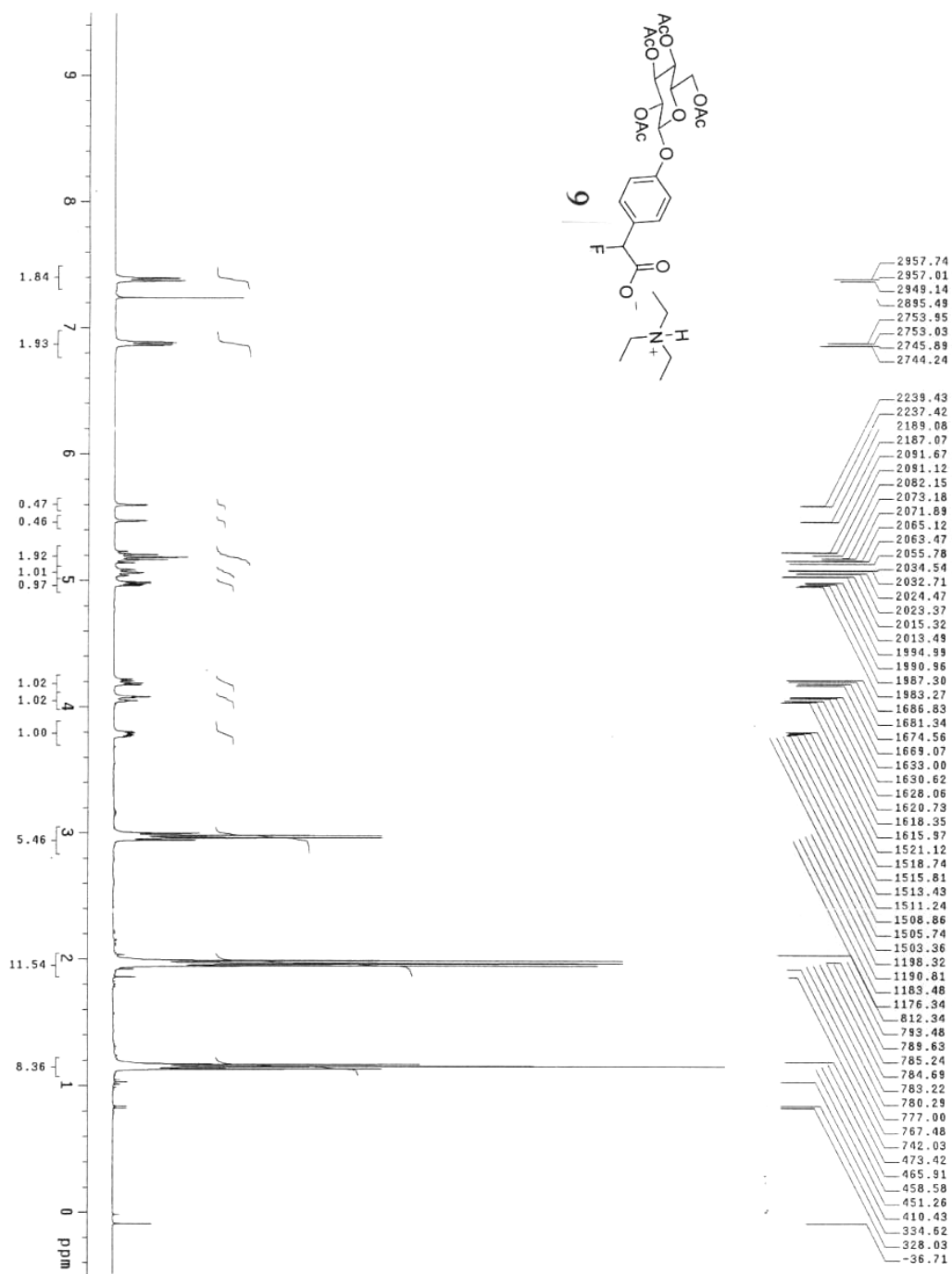
Chemical Shift (ppm)
128.364
128.303
128.273
128.242
128.212
128.159
117.067
116.141

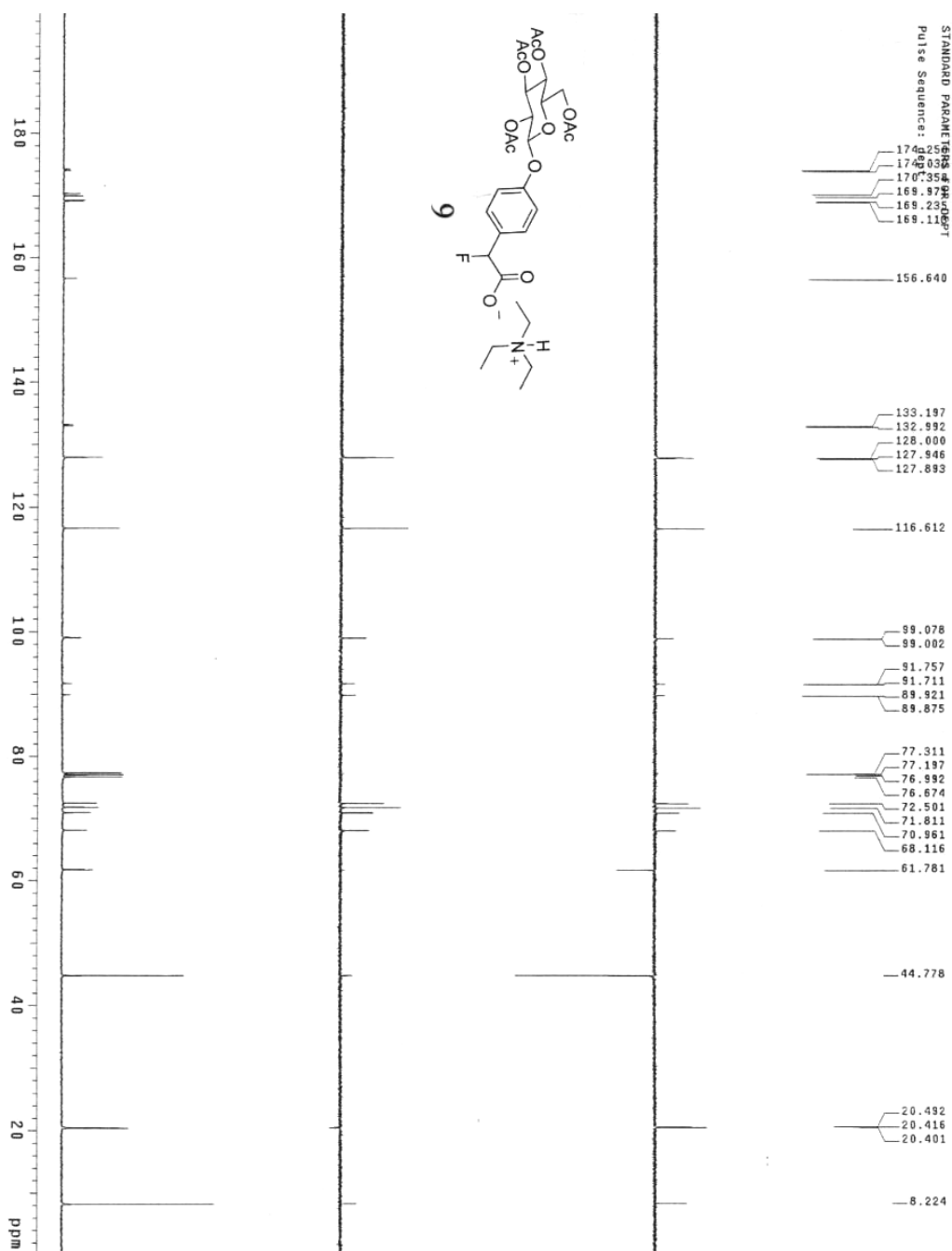
Chemical Shift (ppm)
98.494
98.464
89.336
89.276
87.485
87.424

Chemical Shift (ppm)
77.319
77.000
76.681
72.433
71.962
70.915
68.047
61.727
59.649

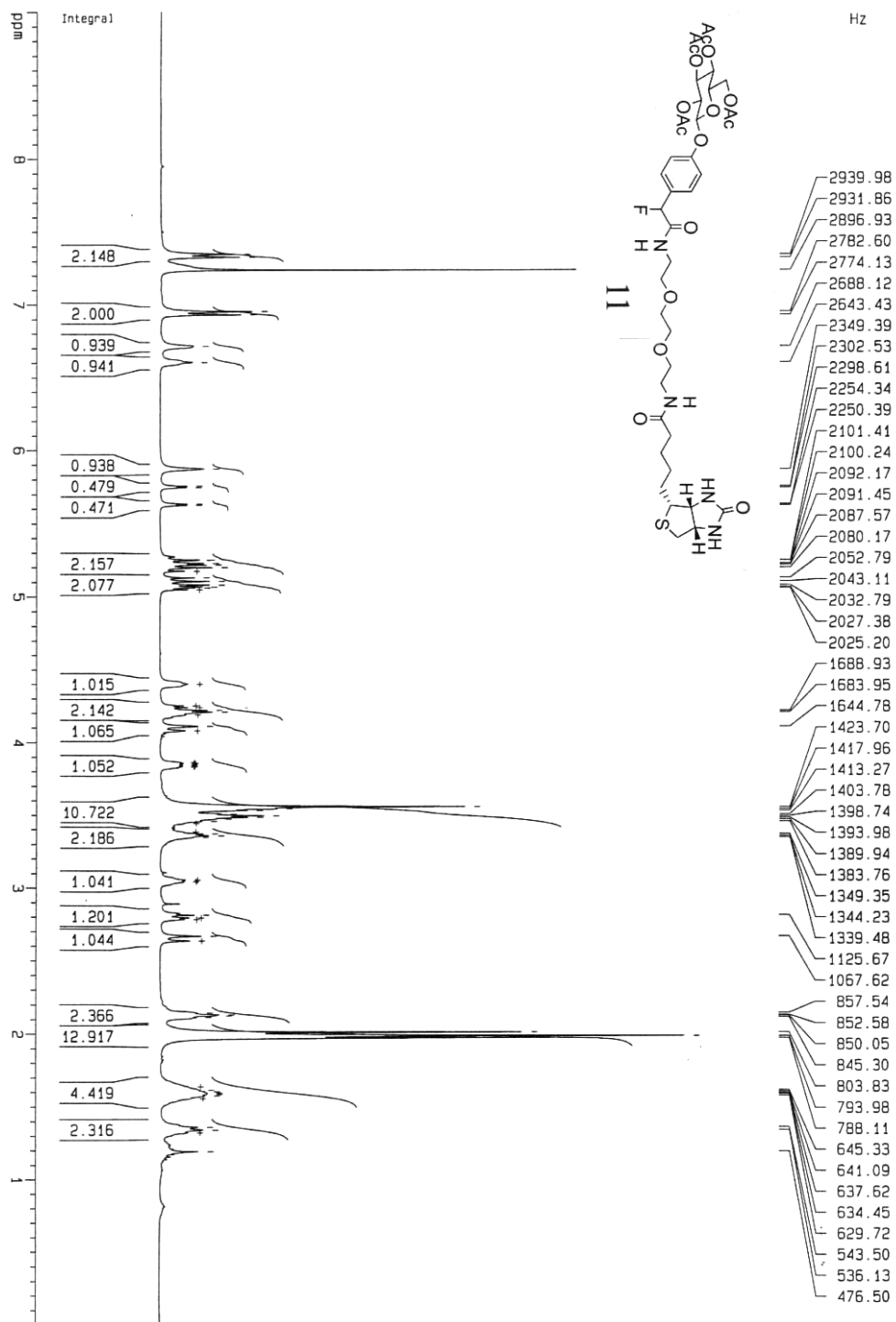
Chemical Shift (ppm)
22.351
20.492
20.416
20.401
17.677







Y.056 CDCl3 020111 400MHz H1



Current Data Parameters
 Name: Y.056020111
 ExpNO: 100
 F2 - Acquisition Parameters
 Date_: 20020111
 Time: 12.50
 Instrument: spect
 PROBHD: 5 mm QNP 1H
 PULPROG: zgpg30
 FIDRES: 0.2207028
 SFO1: 400.1260951
 AQ: 2.2807028
 DE: 63.600
 TE: 300.2
 D1: 1.00000000
 ***** CHANNEL f1 *****
 NUC1: 1H
 P1: 11.50
 PL1: 0.00
 SFO1: 400.1260951
 F2 - Processing parameters
 SI: 32768
 SF: 400.1305159
 SC: 64
 SS: 5280
 LB: 0.00
 GB: 0
 PC: 1.00
 10 MHz B1H parameters
 EX: 25.00
 FL: 1.00
 F1: 3061.17
 F2: 0.000
 PR: 0.0000
 NUCB: 144.04660

