

Supporting Information

Biomass derived xylose Guerbet surfactants: thermotropic and lyotropic properties from small-angle X-ray scattering

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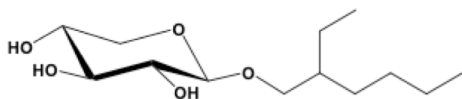
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^1H NMR and ^{13}C NMR for the synthetic Guerbet xylosides

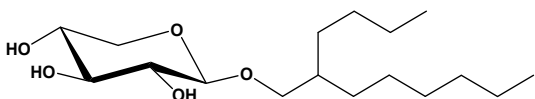
2-ethyl-hexyl- β -D-xylopyranoside, β -Xyl-C₆C₂



^1H NMR (400 MHz, CD₃OD): δ (ppm) = 0.92 (t, 6H, J = 7.16 Hz, 2 x CH₃), 1.25 -1.50 (m, 9H, CH₂ and CH), 3.22 (m, 2H, H-1'), 3.32 (m, 1H, H-3), 3.42 (m, 1H, H-2), 3.50 (m, 1H, H-4), 3.74 (m, 1H, H-5_e), 3.87 (dd, 1H, $J_{4,5a}$ = 5.28 Hz, $J_{5a,5e}$ = 11.44 Hz, H-5_a), 4.19 (d, 1H, $J_{1,2}$ = 7.48 Hz, H-1).

^{13}C NMR (400 MHz, CD₃OD): δ (ppm) = 103.97 (C-1), 76.52 (C-3), 73.53 (C-2), 72.43 (C-4), 69.86 (C-5), 38.04 (C- α), 31.67, 29.64, 29.39, 29.30, 29.28, 29.07, 26.37, 26.30, 22.33 (C -CH₂), 13.03 (CH₃).

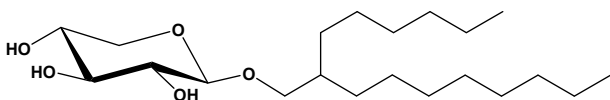
2-butyl-octyl- β -D-xylopyranoside, β -Xyl-C₈C₄



^1H NMR (400 MHz, CD₃OD): δ (ppm) = 0.92 (t, 6H, J = 4.92 Hz, 2 x CH₃), 1.25 -1.50 (m, 17H, CH₂ and CH), 3.20 (m, 2H, H-1'), 3.32 (m, 1H, H-3), 3.41 (m, 1H, H-2), 3.50 (m, 1H, H-4), 3.74 (dd, 1H, $J_{4,5e}$ = 6.06 Hz, $J_{5a,5e}$ = 9.34 Hz, H-5_e), 3.87 (dd, 1H, $J_{4,5a}$ = 5.30 Hz, $J_{5a,5e}$ = 11.42 Hz, H-5_a), 4.18 (d, 1H, $J_{1,2}$ = 7.48 Hz, H-1).

^{13}C NMR (400 MHz, CD₃OD): δ (ppm) = 103.95 (C-1), 76.48 (C-3), 73.51 (C-2), 72.36 (C-4), 69.85 (C-5), 38.12 (C- α), 31.63, 30.87, 30.85, 29.42, 28.77, 28.72, 26.44, 26.37, 22.72, 22.33, 19.66 (C -CH₂), 13.05 (CH₃).

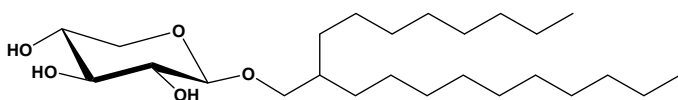
2-hexyl-decyl- β -D-xylopyranoside, β -Xyl-C₁₀C₆



^1H NMR (400 MHz, CD₃OD): δ (ppm) = 0.92 (t, 6H, J = 6.78 Hz, 2 x CH₃), 1.26 (m, 25H, CH₂ and CH), 3.19 (m, 2H, H-1'), 3.31 (m, 1H, H-3), 3.41 (m, 1H, H-2), 3.50 (m, 1H, H-4), 3.73 (dd, 1H, $J_{4,5e}$ = 6.06 Hz, $J_{5a,5e}$ = 9.42 Hz, H-5_e), 3.87 (dd, 1H, $J_{4,5a}$ = 5.31 Hz, $J_{5a,5e}$ = 11.42 Hz, H-5_a), 4.18 (d, 1H, $J_{1,2}$ = 7.52 Hz, H-1).

^{13}C NMR (400 MHz, CD₃OD): δ (ppm) = 103.95 (C-1), 76.51 (C-3), 73.51 (C-2), 72.39 (C-4), 69.85 (C-5), 38.13 (C- α), 31.70, 31.67, 30.89, 30.86, 29.76, 29.45, 29.34, 29.09, 26.47, 26.39, 22.53, 22.38, 22.37, 22.18 (C -CH₂), 19.39, 13.03 (CH₃).

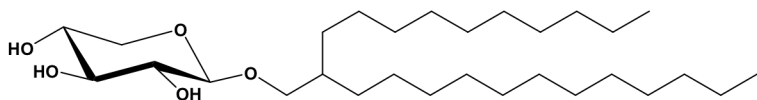
2-octyl-dodecyl- β -D-xylopyranoside, β -Xyl-C₁₂C₈



¹H NMR (400 MHz, CD₃OD): δ (ppm) = 0.93 (t, 6H, J = 6.54 Hz, 2 x CH₃), 1.32 (m, 33H, CH₂ and CH), 3.22 (m, 2H, H-1'), 3.32 (m, 1H, H-3), 3.41 (m, 1H, H-2), 3.52 (m, 1H, H-4), 3.74 (m, 1H, H-5_e), 3.87 (dd, 1H, J_{4,5a} = 7.04 Hz, J_{5a,5e} = 10.92 Hz, H-5_a), 4.19 (d, 1H, J_{1,2} = 7.48 Hz, H-1).

¹³C NMR (400 MHz, CD₃OD): δ (ppm) = 103.94 (C-1), 76.50 (C-3), 73.48 (C-2), 72.41 (C-4), 69.84 (C-5), 38.14 (C- α), 31.94, 31.77, 31.60, 30.89, 30.87, 30.70, 29.83, 29.49, 29.47, 29.45, 29.43, 29.20, 29.17, 26.54, 26.45, 22.60, 22.44, 22.26 (C -CH₂), 14.16, 13.25 (CH₃).

2-decyl-tetradecyl- β -D-xylopyranoside, β -Xyl-C₁₄C₁₀



¹H NMR (400 MHz, CD₃OD): δ (ppm) = 0.92 (t, 6H, J = 6.82 Hz, 2 x CH₃), 1.32 (m, 41H, CH₂ and CH), 3.19 (m, 2H, H-1'), 3.32 (m, 1H, H-3), 3.41 (m, 1H, H-2), 3.50 (m, 1H, H-4), 3.73 (dd, 1H, J_{4,5e} = 6.08 Hz, J_{5a,5e} = 9.40 Hz, H-5_e), 3.87 (dd, 1H, J_{4,5a} = 5.30 Hz, J_{5a,5e} = 11.42 Hz, H-5_a), 4.18 (d, 1H, J_{1,2} = 7.52 Hz, H-1).

¹³C NMR (400 MHz, CD₃OD): δ (ppm) = 103.97 (C-1), 76.51 (C-3), 73.52 (C-2), 72.43 (C-4), 69.85 (C-5), 38.06 (C- α), 31.70, 30.78, 30.76, 29.68, 29.42, 29.38, 29.35, 29.33, 29.31, 29.10, 26.41, 26.33, 22.35 (C -CH₂), 13.07 (CH₃).

FTIR for β -Xyl- C_8C_4

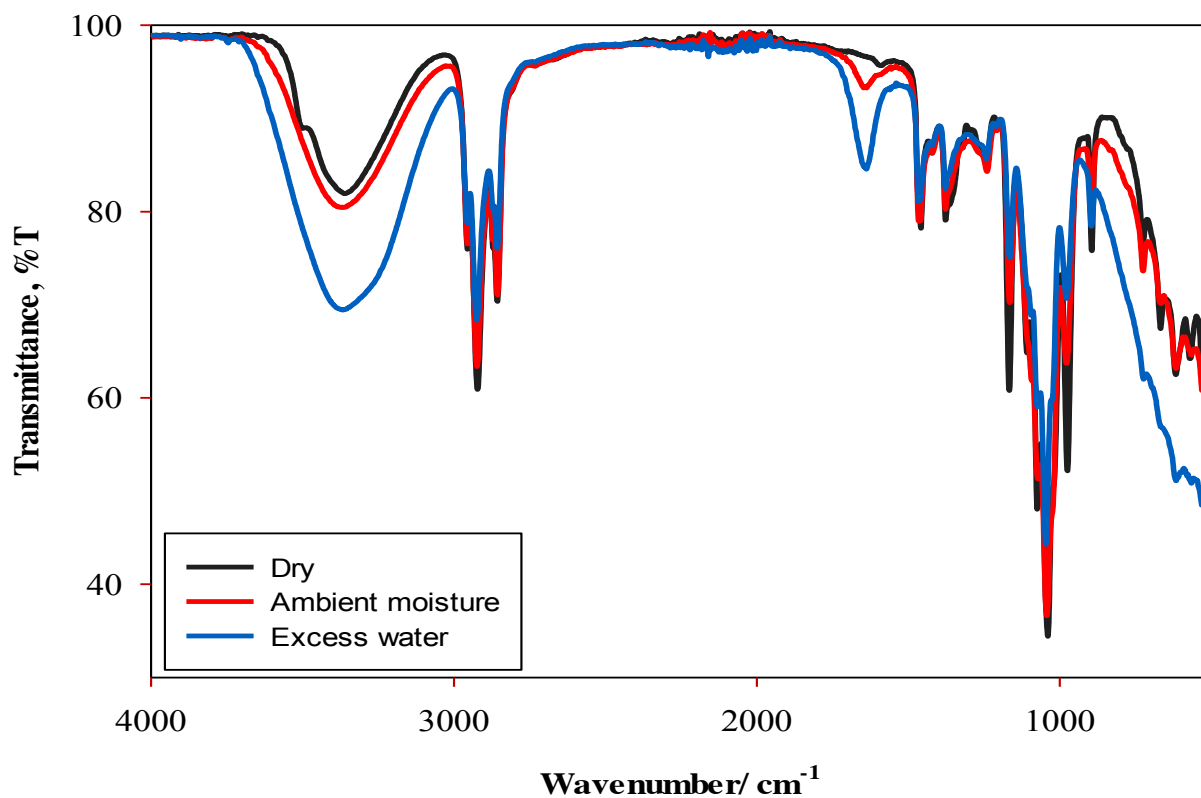


Figure S1. FTIR spectra for β -Xyl- C_8C_4 in dry (after lyophilised in freeze dryer for at least 48 hours), left in ambient moisture for 96 hours and in excess water form.

Differential Scanning Calorimetry (DSC)

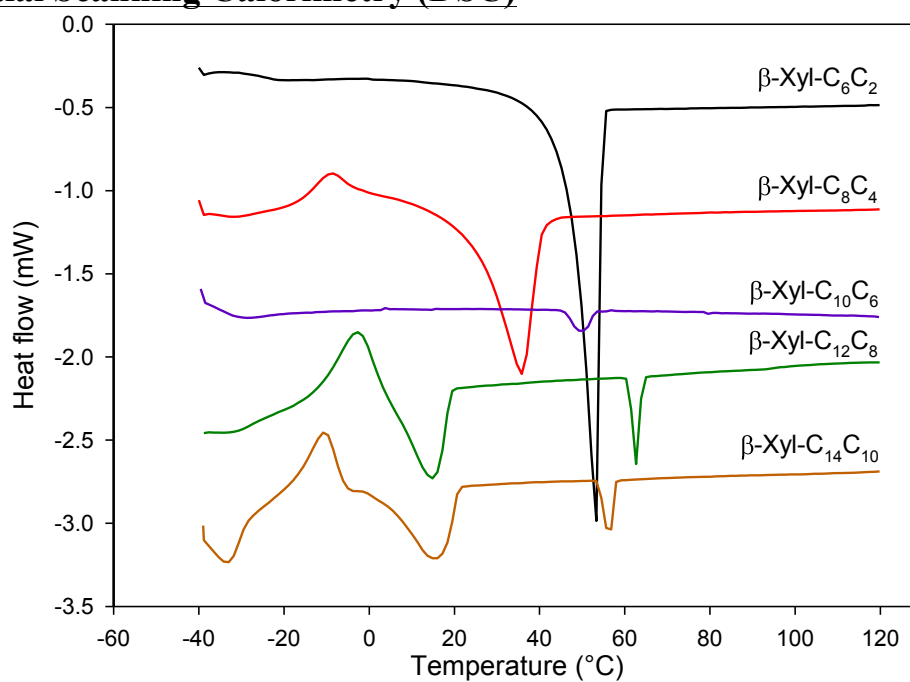


Figure S2. DSC thermograms for dry β -D-xylopyranosides.

X-Ray Scattering Patterns

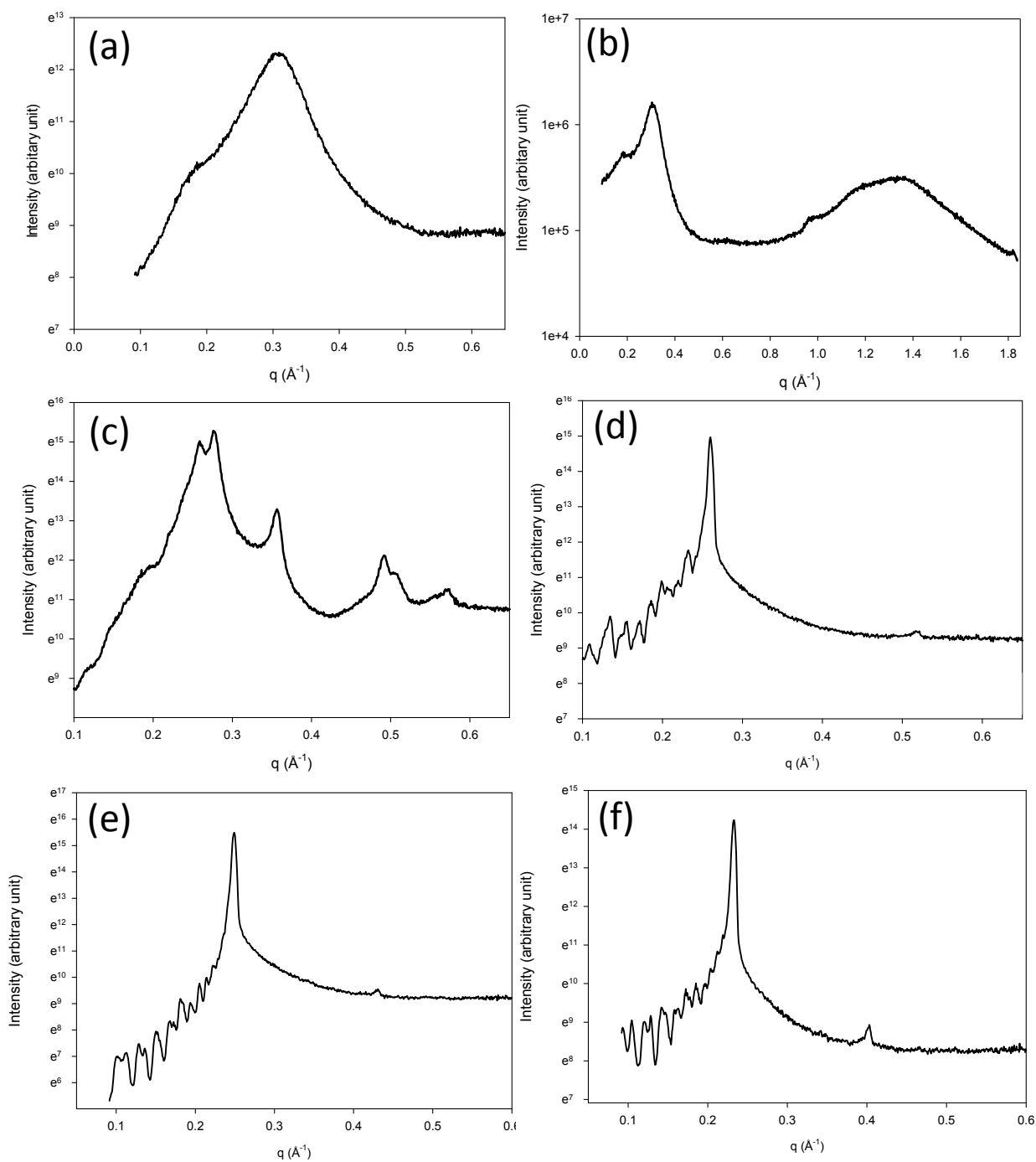
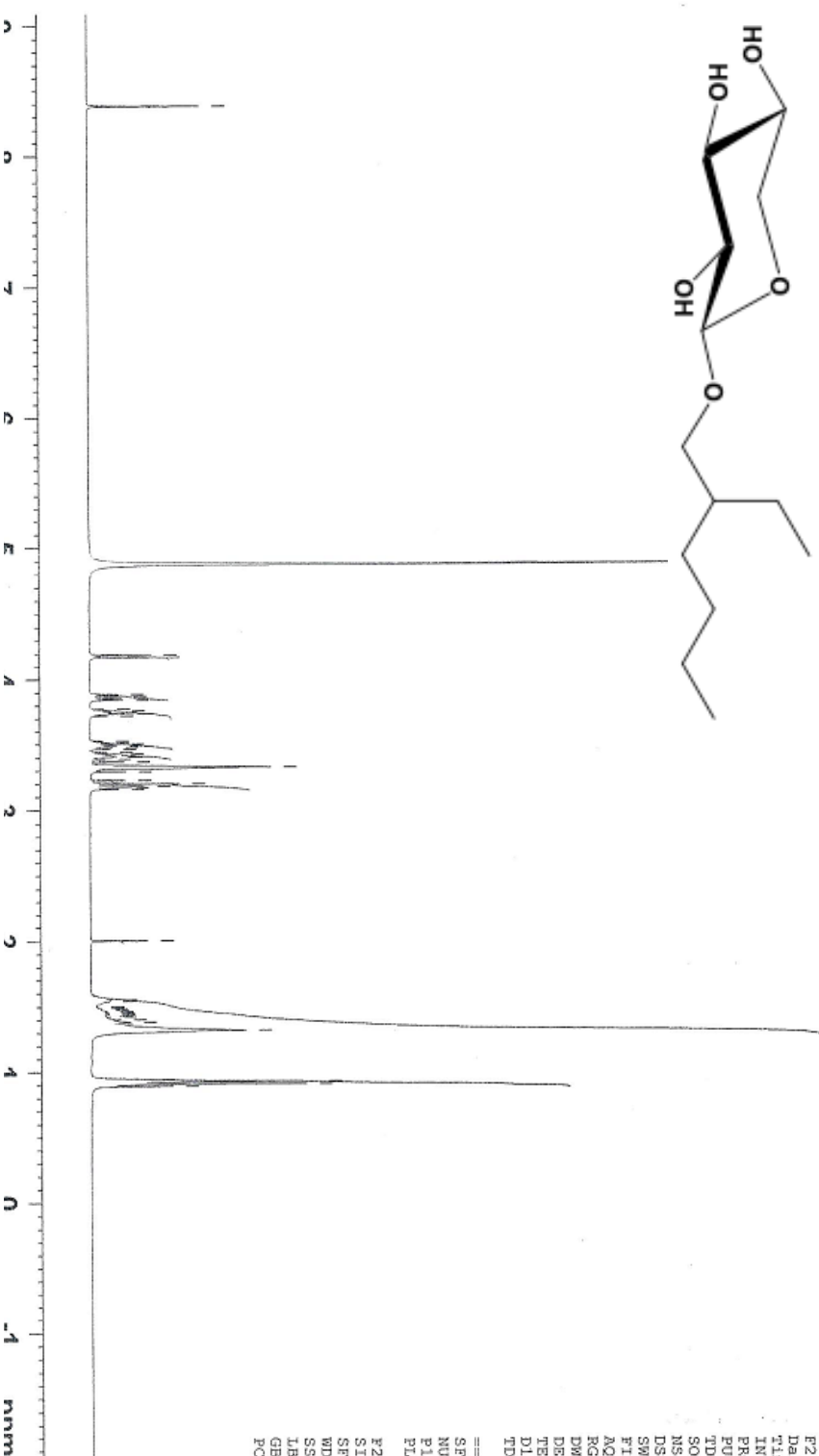


Figure S3. Scattering pattern in small angle X-ray for dry (a) β -Xyl- C_6C_2 at 25°C, (c) β -Xyl- C_8C_4 at 25°C, (d) β -Xyl- $C_{10}C_6$ at 25°C, (e) β -Xyl- $C_{12}C_8$ at 25°C, and (f) β -Xyl- $C_{14}C_{10}$ at 25°C. Scattering pattern in wide angle X-ray for (b) dry β -Xyl- C_6C_2 at 25°C.

1H-b-xyloside #1 Free



- 8.387
- 4.879
- 4.193
- 4.174
- 3.887
- 3.874
- 3.859
- 3.846
- 3.760
- 3.750
- 3.745
- 3.737
- 3.721
- 3.505
- 3.492
- 3.481
- 3.468
- 3.443
- 3.428
- 3.420
- 3.410
- 3.405
- 3.388
- 3.368
- 3.329
- 3.320
- 3.296
- 3.228
- 3.202
- 3.183
- 3.175
- 3.162
- 2.003
- 1.556
- 1.543
- 1.469
- 1.460
- 1.451
- 1.444
- 1.434
- 1.425
- 1.404
- 1.386
- 1.323
- 0.947
- 0.931
- 0.912
- 0.893

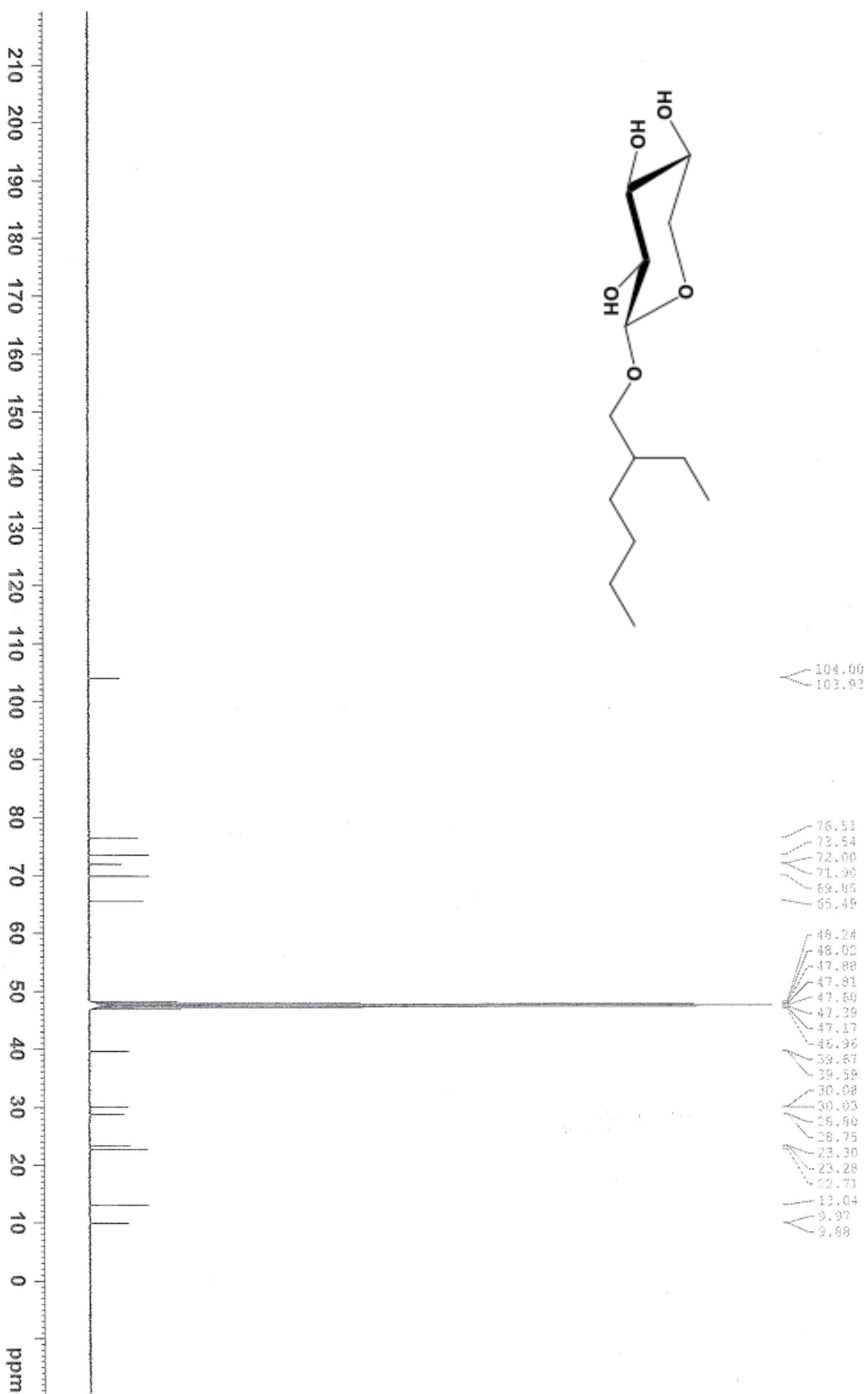
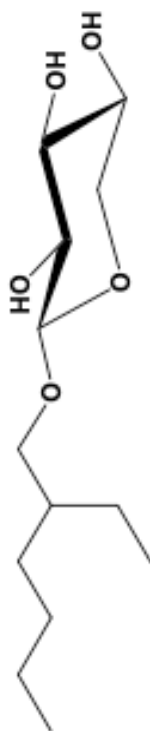


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 DE 6.50 usec
 TE 298.1 K
 D1 1.00000000 sec
 TDO 1

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 PLW1 12.00000000 w
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13C- b xyloside #1



1H- b xyloside #2

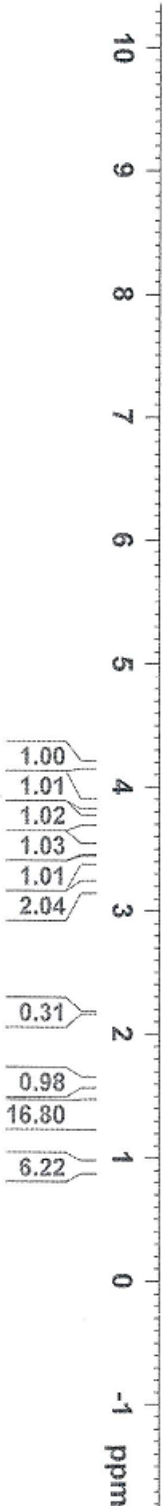
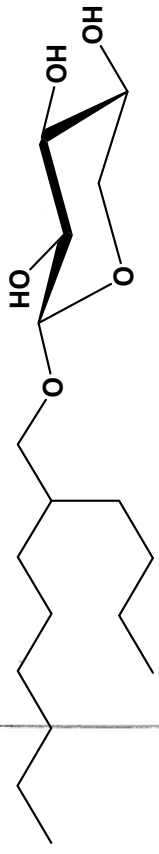
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- 4.587
- 4.190
- 4.171
- 3.887
- 3.874
- 3.858
- 3.845
- 3.755
- 3.740
- 3.732
- 3.717
- 3.529
- 3.515
- 3.505
- 3.492
- 3.482
- 3.468
- 3.429
- 3.415
- 3.406
- 3.391
- 3.329
- 3.317
- 3.294
- 3.226
- 3.200
- 3.185
- 3.172
- 3.164
- 2.176
- 1.625
- 1.613
- 1.601
- 1.418
- 1.402
- 1.316
- 0.931



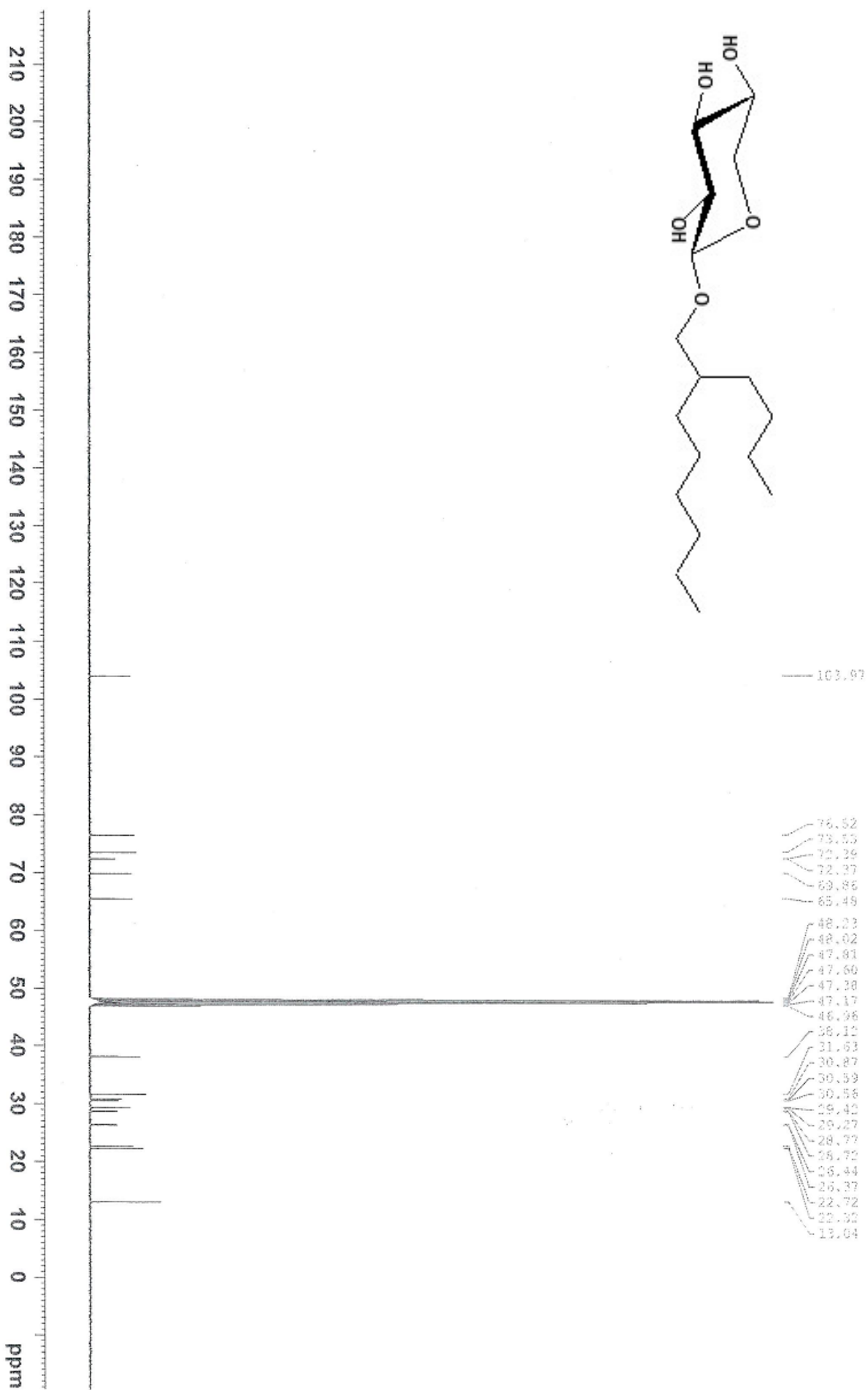
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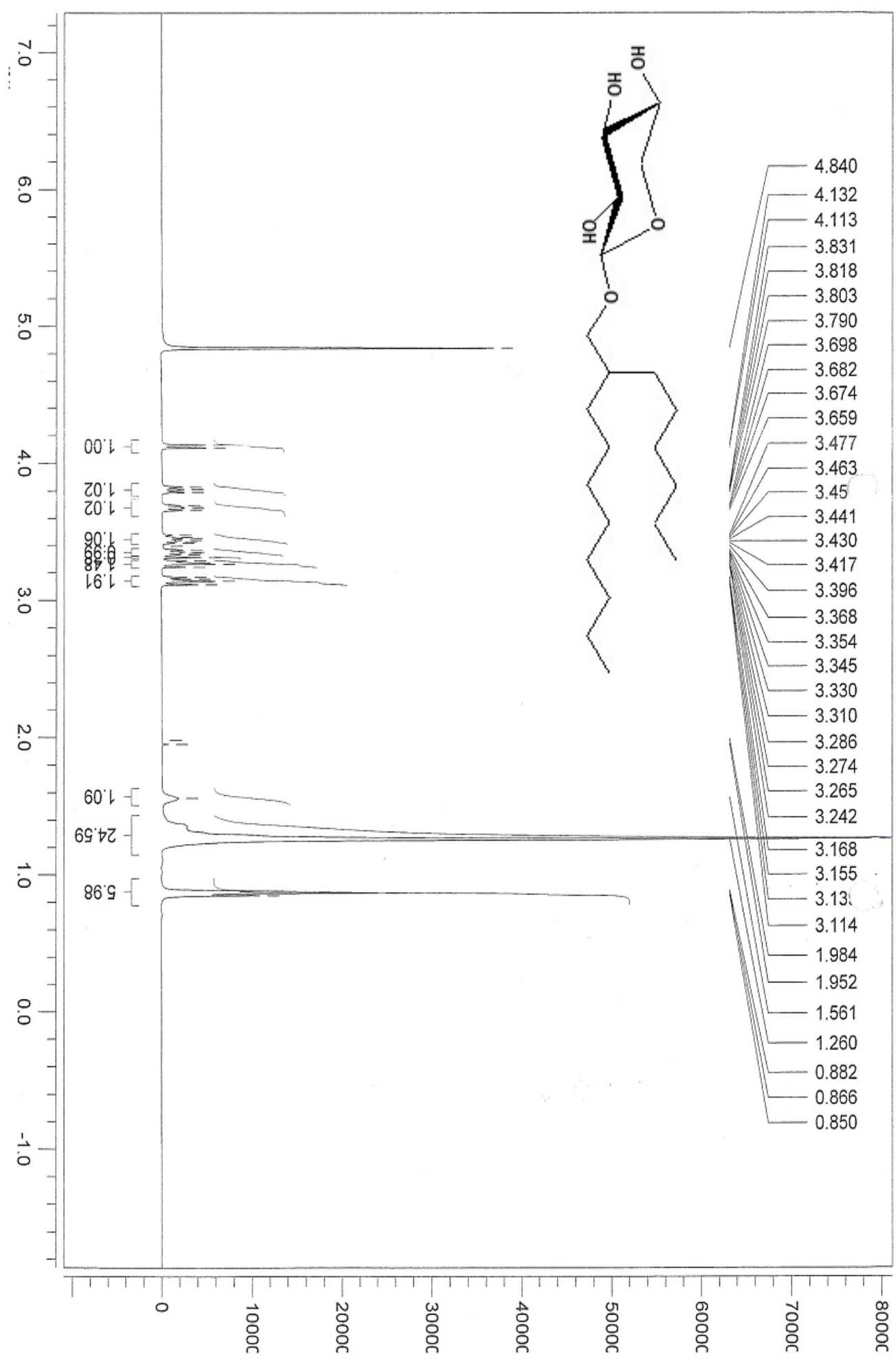
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 RG 107.78
 DM 62.400 usec
 DE 6.50 usec
 TE 298.1 K
 D1 1.00000000 sec
 TD0 1

==== CHANNEL f1 =====
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 NUC1 1H
 P1 15.50 usec
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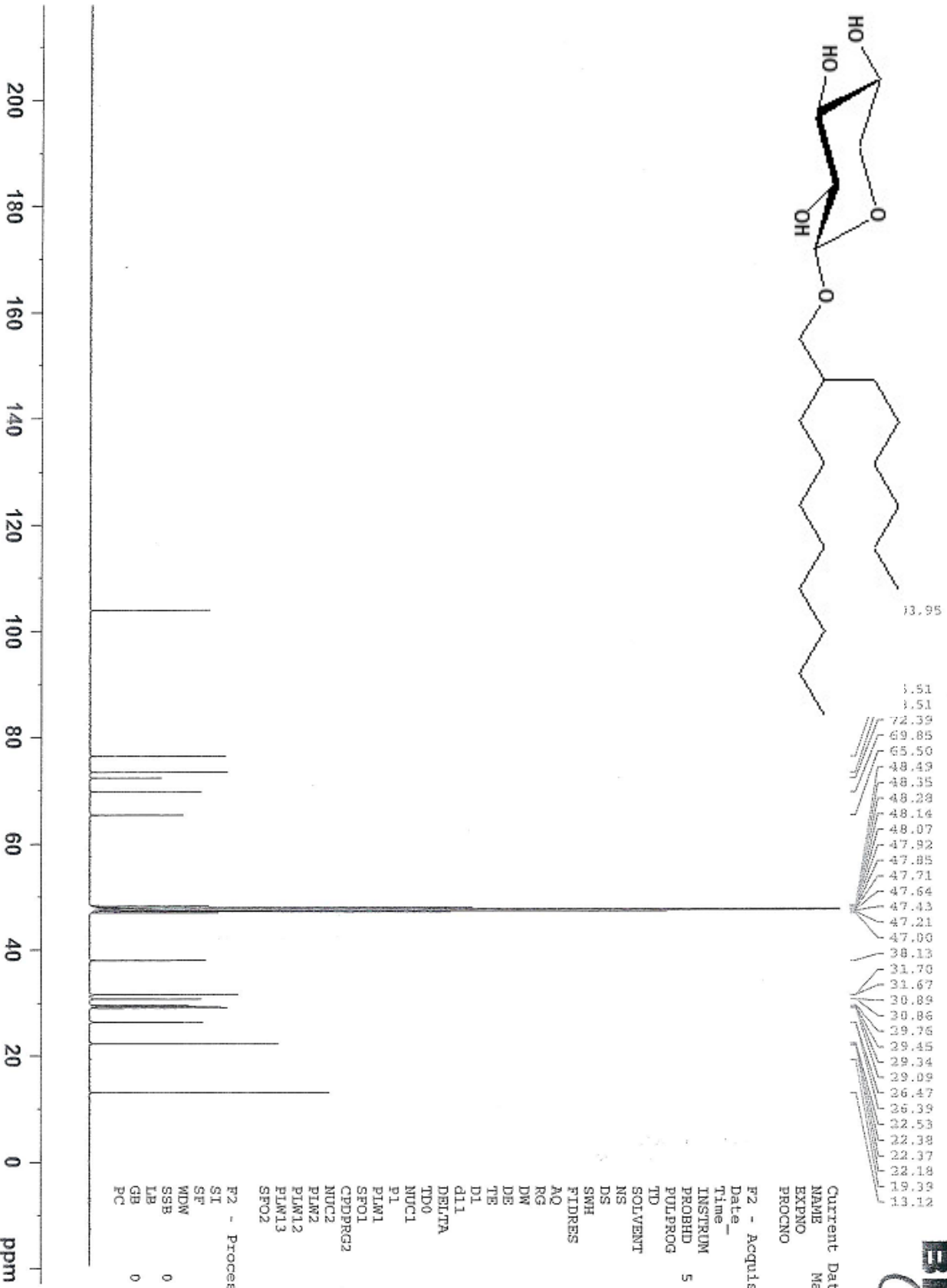
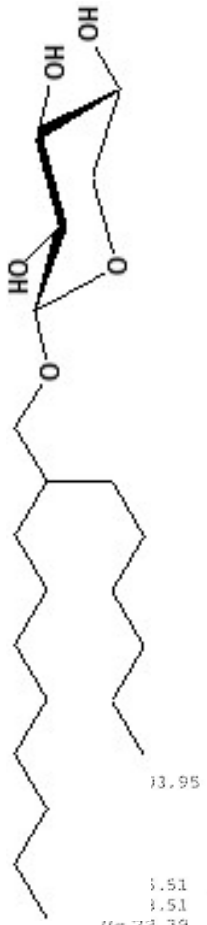


1H- b xyloside #2





13C-xylose #3 deAc



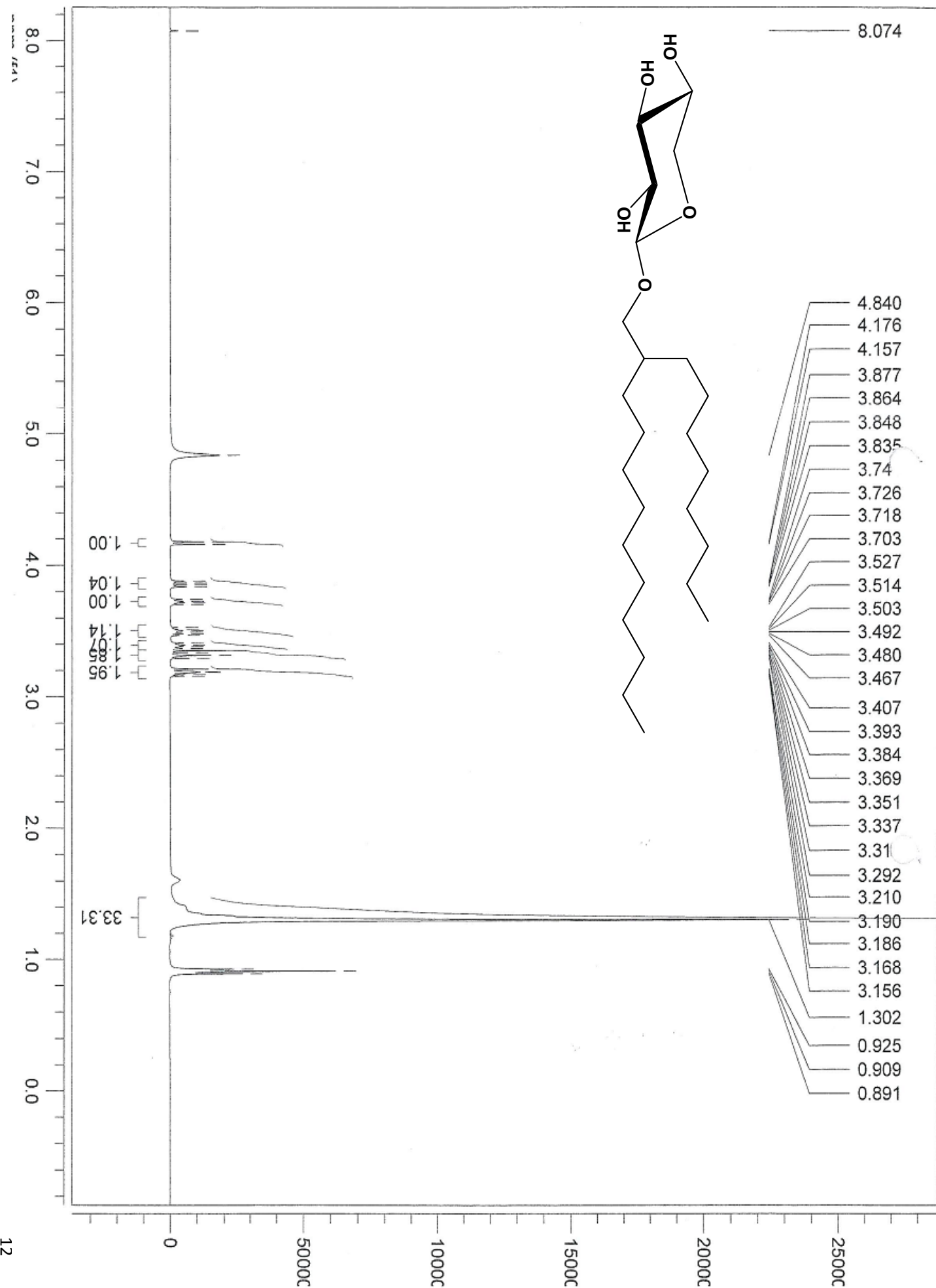
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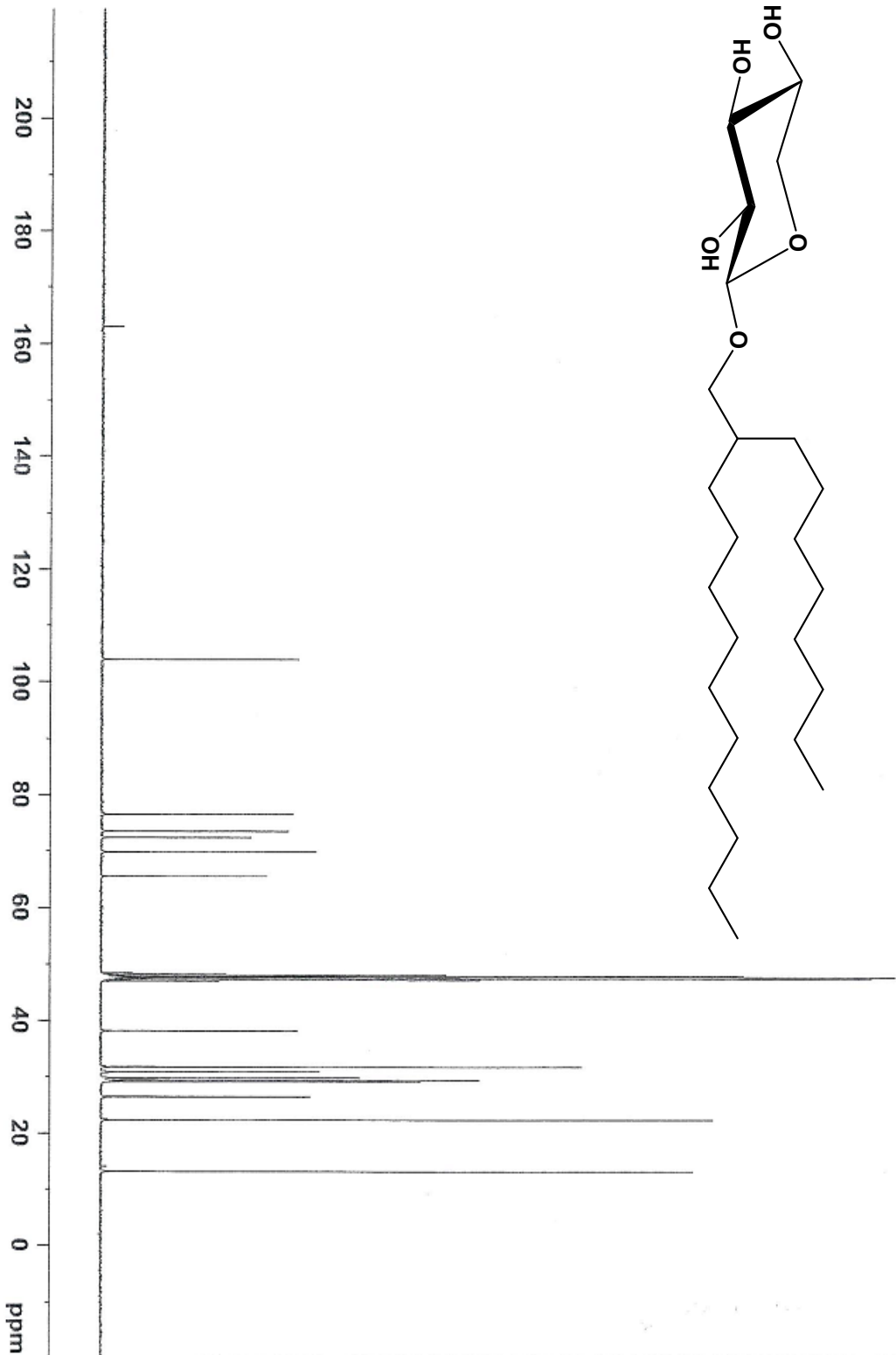
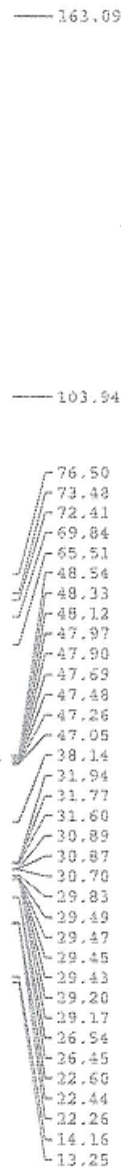
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 CPDPRG2

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 WDM EM
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 LB 1.00 Hz
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¹³C-xyloside C12C8 Free



Current Data Parameters
 NAME Mar27-2014 xylose #4
 EXPNO 11
 PROCNO 1

F2 - Acquisition Parameters

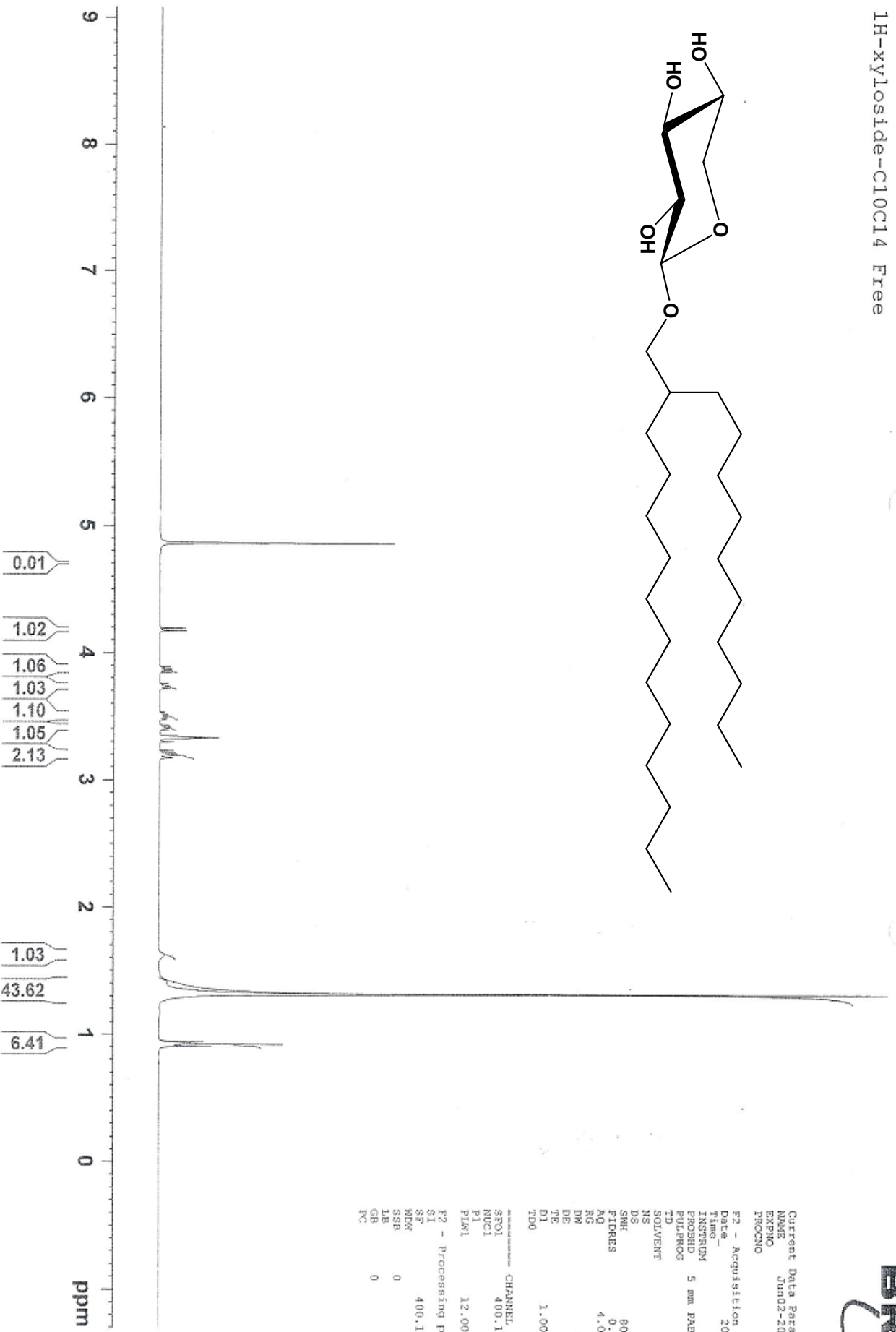
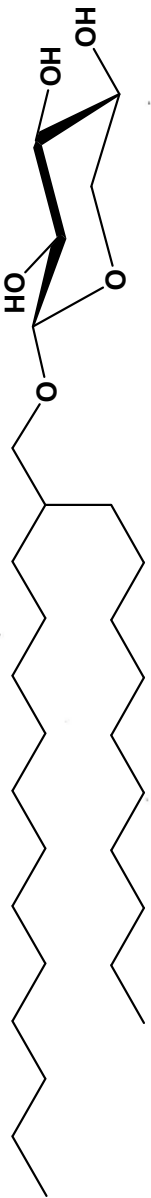
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 d11 0.03000000 sec
 DELTA 1.89999998 sec

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CPDPRG2
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 PLM13 0.28830001 W
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1H-xyloside-C10C14 Free



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 PROCNO 1

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 TD0 1

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F2 - Processing parameters
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 SFR 0.30 Hz
 ZF 0
 SFR 1.00

13C-xyloside-Cl0C14 Free

