Electronic Supplementary Material (ESI) for New Journal of Chemistry

Supporting information

Synthesis of Ag-glyconanoparticles with *C*-glycosides, their lectin binding studies and anti bacterial activity.

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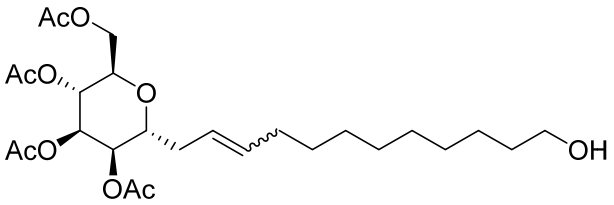
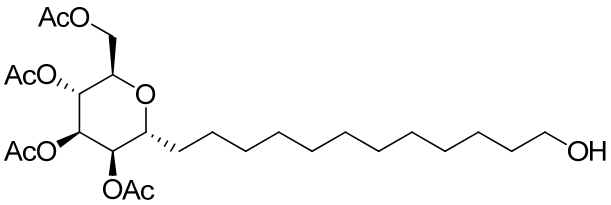


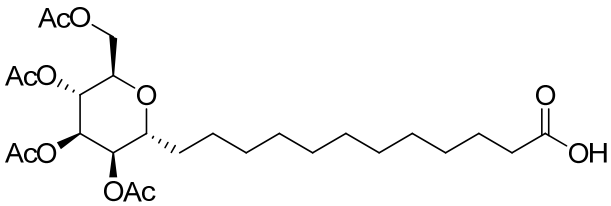
A solution of (2,3,4,6-Tetra-*O*-acetyl-α-D-mannopyranosyl)-l-propene (100 mg, 0.3 mmol), 10-undec-1-ol (228 mg, 1.3 mmol), Grubbs' Ist generation catalyst (11 mg, 0.01 mmol) was degassed with argon and heated at 40 °C (oil bath temperature) for 24 h, under inert atmosphere in dry DCM (15 mL). The reaction mixture was cooled to room temperature and concentrated under reduced pressure. The residue was purified by column chromatography (4:1 petroleum ether/ethyl acetate) to afford inseparable mixture of *E/Z* isomers **3** [2:1 ratio] (52 mg, 37%) as a colorless oil, 10-undec-1-ol dimers (51 mg, 61%) as a white amorphous solid. $[\alpha]_D^{25}$ = +6.48 (*c* 1.2, $CHCl_3$). IR ($CHCl_3$): ν 3410, 3020, 2929, 1743, 1750, 1215, 1034, 758, 668 cm^{-1}. ^{1}H NMR (200 MHz, $CDCl_3$): *δ* 1.20–1.30 (br s, 16H), 1.46–1.55 (m, 2H), 1.97 (s, 3H), 2.01 (s, 3H), 2.04 (s, 3H), 2.08 (s, 3H), 2.15–2.28 (m, 1H), 2.30–2.47 (m, 1H), 3.57 (t, *J* = 6.6 Hz, 2H), 3.80–3.87 (m, 1H), 3.90–3.97 (m, 1H), 4.02–4.08 (m, 1H), 4.26 (dd, *J* = 5.7, 12.0 Hz, 1H) 5.13–5.16 (m, 1H), 5.17–5.24 (m, 2H), 5.26–5.34 (m, 1H), 5.45–5.55 (m, 1H). ^{13}C NMR (50 MHz, $CDCl_3$): *δ* 20.4 (q, 3C), 20.6 (q), 25.5 (t), 28.8 (t), 28.9 (t), 29.1 (t, 3C), 29.2 (t, 2C), 32.4 (t), 62.5 (t, 2C), 66.6 (d), 68.6 (d), 69.8 (d), 70.1 (d), 74.7 (d), 123.3 (d), 134.4 (d), 169.4 (s), 169.7 (s), 170.0 (s), 170.4 (s) ppm. ESI-MS: (*m/z*) 537.6 (100%, $[M+Na]^+$). Anal. Calcd for $C_{26}H_{42}O_{10}$: C, 62.42; H, 8.65. Found: C, 62.39; H, 8.60.

1-(2,3,4,6-Tetra-*O*-acetyl-α-D-mannopyranosyl)-12-hydroxydodecane (5)



To a solution of alcohol **3** (200 mg, 0.4 mmol) in MeOH: EtOAc (1:2), 5% Pd-C (10 mg) was added. The solution was stirred under hydrogen atmosphere at room temperature for 2 h. Reaction mixture was filtered through a short pad of celite and washed thoroughly with methanol, concentrated and the crude residue was purified by column chromatography (4:1 petroleum ether/ethyl acetate) to furnish saturated alcohol **5** (154 mg, 80%) as a colorless liquid. $[\alpha]_D^{25}$ = +4.67 (*c* 1.2, $CHCl_3$). IR ($CHCl_3$): ν 3482, 3021, 2929, 1749, 1369, 1216, 1034, 757, 668 cm^{-1}. ^1H NMR (200 MHz, $CDCl_3$): *δ* 1.24 (br s, 18H), 1.48–1.61 (m, 4H), 2.00 (s, 3H), 2.03 (s, 3H), 2.08 (s, 3H), 2.12 (s, 3H), 3.61 (t, *J* = 6.5 Hz, 2H), 3.75–3.85 (m, 1H), 3.88–3.98 (m, 1H), 4.75 (dd, *J* = 2.6, 9.5 Hz, 1H), 4.28 (dd, *J* = 6.1, 12.1 Hz, 1H), 5.12–5.17 (m, 1H), 5.18–5.23 (m, 2H). ^{13}C NMR (50 MHz, $CDCl_3$) : *δ* 20.7 (q, 3C), 20.9 (q), 25.3 (t), 25.6 (t), 28.3 (t), 29.0 (t), 29.4 (t, 3C), 29.5 (t, 3C), 32.7 (t), 62.6 (t), 62.9 (t), 66.9 (d), 69.1 (d), 69.9 (d), 70.9 (d), 75.4 (d), 169.6 (s), 170.0 (s), 170.3 (s), 170.7 (s) ppm. ESI-MS: *m/z* 539.6 (100%, $[M+Na]^+$). Anal. Calcd for $C_{26}H_{44}O_{10}$ C, 60.45; H, 8.58. Found: C, 60.39; H, 8.54.

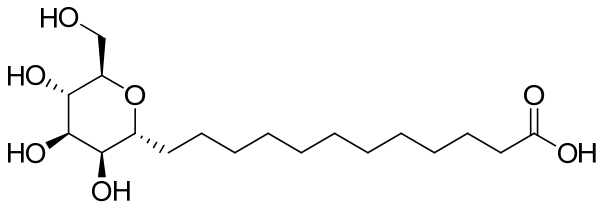
1-(2,3,4,6-Tetra-*O*-acetyl-α-D-mannopyranosyl)-12-dodecanoic acid (7):



To a solution of alcohol **5** (160 mg, 0.3 mmol) in CCl_4 (2 mL)-CH_3CN (2 mL)-H_2O (3 mL), $NaIO_4$ (266 mg, 1.2 mmol) and $RuCl_4.H_2O$ (1.6 mg, 0.006 mmol) were added. The biphasic suspension was stirred vigorously for 1 h. The color was changed from wine red to pale brown. Reaction mixture was extracted with DCM (3 x 25 mL). The combined organic layer was dried (Na_2SO_4), filtered and concentrated. Diethyl ether (20

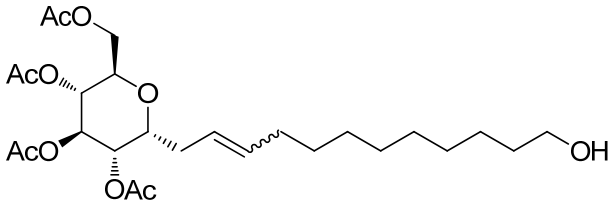
mL) was added to it, filtered through a celite pad to remove metal impurities, the filtrate was concentrated at reduced pressure and purified by column chromatography (3:1 petroleum ether/ethyl acetate) to furnish the triacetate acid **7** (146 mg, 88%) as a colorless liquid. $[\alpha]_D^{25}$ = +4.83 (*c* 1.1, $CHCl_3$). IR ($CHCl_3$): ν 3401, 2929, 1748, 1710, 1368, 1216, 1034, 756 cm^{-1}. ^1H NMR (200 MHz, $CDCl_3$): *δ* 1.26 (br s, 16H), 1.55–1.70 (m, 4H), 2.00 (s, 3H), 2.02 (s, 3H), 2.10 (s, 3H), 2.14 (s, 3H), 2.35 (t, *J* = 7.4 Hz, 2H), 3.80–3.89 (m, 1H), 3.90–4.00 (m, 1H), 4.05–4.15 (m, 2H), 4.31 (dd, *J* = 6.0, 12.0 Hz, 1H), 5.16–5.20 (m, 1H), 5.21–5.25 (m, 1H). ^{13}C NMR (50 MHz, $CDCl_3$) : *δ* 20.7 (q, 3C), 20.9 (q), 24.6 (t), 25.3 (t), 28.9 (t), 29.0 (t), 29.1 (t), 29.3 (t), 29.4 (t, 3C), 29.6 (t), 33.8 (t), 62.6 (t), 66.9 (d), 69.1 (d), 69.9 (d), 70.9 (d), 75.4 (d), 169.7 (s), 170.1 (s), 170.4 (s), 170.7 (s), 179.1 (s) ppm. ESI-MS: *m/z* 553.3 (100%, $[M+Na]^+$). Anal. Calcd for $C_{26}H_{42}O_{11}$: C, 58.85; H, 7.98. Found: C, 58.80; H, 8.02.

1-(α-D-mannopyranosyl)-12-dodecanoic acid (1)



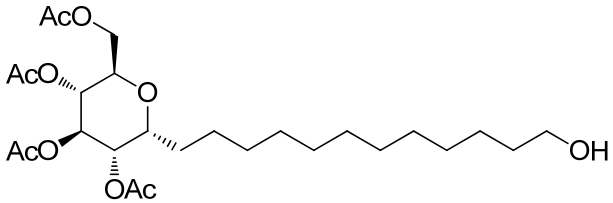
To a solution of acid **7** (100 mg, 0.2 mmol) in 5 mL of dry methanol, K_2CO_3 (78 mg, 0.6 mmol) was added. The suspension was stirred at room temperature for 1 h. Reaction mixture was filtered through a celite pad, washed with methanol and the filtrate was concentrated and purified by column chromatography (8:1:1 $CHCl_3$/MeOH/AcOH) to furnish *C*-glycosyl acid **1** (61 mg, 89 %) as a white color solid. $[\alpha]_D^{25}$ = -51.87 (*c* 4.0, MeOH). IR ($CHCl_3$): ν 3365, 2853, 1709, 1569, 1455, 1377, 1032, 721 cm^{-1}. ^1H NMR (400 MHz, MeOH-d_4): *δ* 1.28 (br s, 16H), 1.43–1.50 (m, 2H), 1.55–1.64 (m, 2H), 2.25 (t, *J* = 7.1 Hz, 2H), 3.37–3.43 (m, 1H), 3.53–3.62 (m, 1H), 3.63–3.66 (m, 1H), 3.67–3.70 (m, 1H), 3.71–3.77 (m, 2H), 3.79–3.86 (m, 1H). ^{13}C NMR (100 MHz, MeOH-d_4): *δ* 20.8 (t), 23.5 (t), 24.1 (t), 26.3 (t), 26.7 (t), 27.4 (t), 27.6 (t), 27.8 (t), 27.9 (t, 2C), 30.2 (t), 60.1 (t), 66.3 (d), 69.9 (d), 70.3 (d), 72.6 (d), 76.2 (d) ppm. ESI-MS: *m/z* 385.4 (100%, $[M+Na]^+$). Anal. Calcd for $C_{18}H_{34}O_7$: C, 59.65; H, 9.45. Found: C, 59.69; H, 9.49.

1-(2,3,4,6-Tetra-*O*-acetyl-α-D-glucopyranosyl)-12-hydroxydodec-2-ene (4)



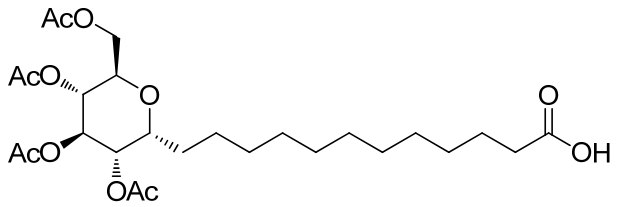
A solution of (2,3,4,6-Tetra-*O*-acetyl-α-D−mannopyranosyl)-l-propene (200 mg, 0.5 mmol), 10-undec-1-ol (456 mg, 2.7 mmol), Grubbs' I generation catalyst (22 mg, 0.03 mmol) was degassed with argon and heated at 40 °C (oil bath temperature) for 24 h, under inert atmosphere in dry DCM (15 mL). The reaction mixture was cooled to room temperature and concentrated under reduced pressure. The residue was purified by column chromatography (4:1 petroleum ether/ethyl acetate) to afford inseparable mixture of *E/Z* isomers **4** [2:1 ratio] (113 mg, 39%) as a colorless oil, 10-undec-1-ol dimer (49 mg, 60%) as a white amorphous solid. $[\alpha]_D^{25}$ = +15.23 (*c* 1.2, $CHCl_3$). IR ($CHCl_3$): ν 3410, 3020, 2929, 1743, 1750, 1215, 1034, 758, 668 cm^{-1}. ^1H NMR (200 MHz, $CDCl_3$): *δ* 1.20–1.40 (m, 12H), 1.50–1.65 (m, 4H), 2.03 (s, 3H), 2.04 (s, 3H), 2.05 (s, 3H), 2.09 (s, 3H), 2.21–2.37 (m, 1H), 2.41–2.60 (m, 1H), 3.63 (t, *J* = 6.6 Hz, 2H), 3.75–3.87 (m, 1H), 3.99–4.09 (m, 1H), 4.15–4.27 (m, 2H), 4.93–5.11 (m, 2H), 5.31 (dt, *J* = 3.4, 9.0 Hz, 2H), 5.45–5.61 (m, 1H). ^{13}C NMR (50 MHz, $CDCl_3$): *δ* 20.3 (q), 20.4 (q), 23.8 (t), 25.5 (t), 27.3 (t), 28.8 (t), 29.0 (t), 29.1 (t), 29.2 (t), 29.3 (t), 32.4 (t), 32.5 (t), 61.9 (t), 62.3 (t), 68.3 (d), 68.5 (d), 68.6 d), 70.0 (d), 70.1 (d), 72.0 (d), 72.2 (d), 123.3 (d), 123.8 (d), 132.4 (d), 133.7 (d), 169.1 (s), 169.2 (s), 169.7 (s), 169.8 (s), 170.2 (s) ppm. ESI-MS: *m/z* 537.6 (100%, $[M+Na]^+$). Anal. Calcd for $C_{26}H_{42}O_{10}$: C, 62.42; H, 8.65. Found: C, 62.39; H, 8.60.

1-(2,3,4,6-Tetra-*O*-acetyl-α-D-glucopyranosyl)-12-hydroxydodecane (6)



To a solution of alcohol **4** (300 mg, 0.6 mmol) in MeOH: EtOAc (1:2), 5% Pd-C (15 mg) was added. The solution was stirred under a hydrogen atmosphere at room temperature for 2 h. Reaction mixture was filtered through a short pad of celite and washed thoroughly with methanol, concentrated and the crude residue was purified by column chromatography (4:1 petroleum ether/ethyl acetate) to furnish saturated alcohol **6** (243 mg, 80%) as a colorless liquid. $[\alpha]_D^{25}$ = +15.2 (*c* 1.2, $CHCl_3$). IR ($CHCl_3$): ν 3482, 3021, 2929, 1749, 1369, 1216, 1034, 757, 668 cm^{-1}. ^1H NMR (200 MHz, $CDCl_3$): *δ* 1.24 (br s, 18H), 1.53–1.90 (m, 4H), 2.03 (s, 3H), 2.03 (s, 3H), 2.05 (s, 3H), 2.09 (s, 3H), 3.63 (t, *J* = 6.5 Hz, 1H), 3.79 (ddd, *J* = 2.3, 5.1, 9.2 Hz, 1H), 4.06 (dd, *J* = 2.6, 12.0 Hz, 2H), 4.22 (dd, *J* = 5.1, 12.0 Hz, 2H), 4.96 (dd, *J* = 9.1, 9.4 Hz, 1H), 5.05 (dd, *J* = 5.8, 9.6 Hz, 1H), 5.29 (dd, *J* = 9.1. 9.4 Hz, 1H). ^{13}C NMR (50 MHz, $CDCl_3$) : *δ* 20.4 (br q, 2C), 20.5 (br q, 2C), 24.8 (t), 25.0 (t), 25.6 (t), 29.1 (t), 29.3 (t), 29.4 (t, 4C), 29.4 (t), 32.6 (t), 62.2 (t), 62.6 (t), 68.3 (d), 68.8 (d), 70.4 (d), 72.4 (d), 169.3 (s), 169.3 (s), 169.9 (s), 170.3 (s) ppm. ESI-MS: *m/z* 539.6 (100%, $[M+Na]^+$). Anal. Calcd for $C_{26}H_{44}O_{10}$ C, 60.45; H, 8.58. Found: C, 60.39; H, 8.54.

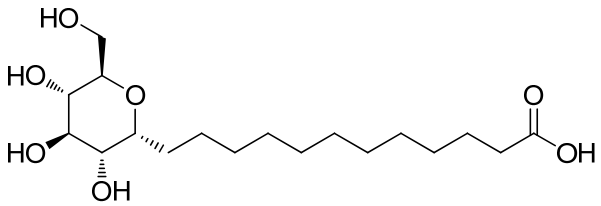
1-(2,3,4,6-Tetra-*O*-acetyl-α-D-glucopyranosyl)-12-dodecanoic acid (8)



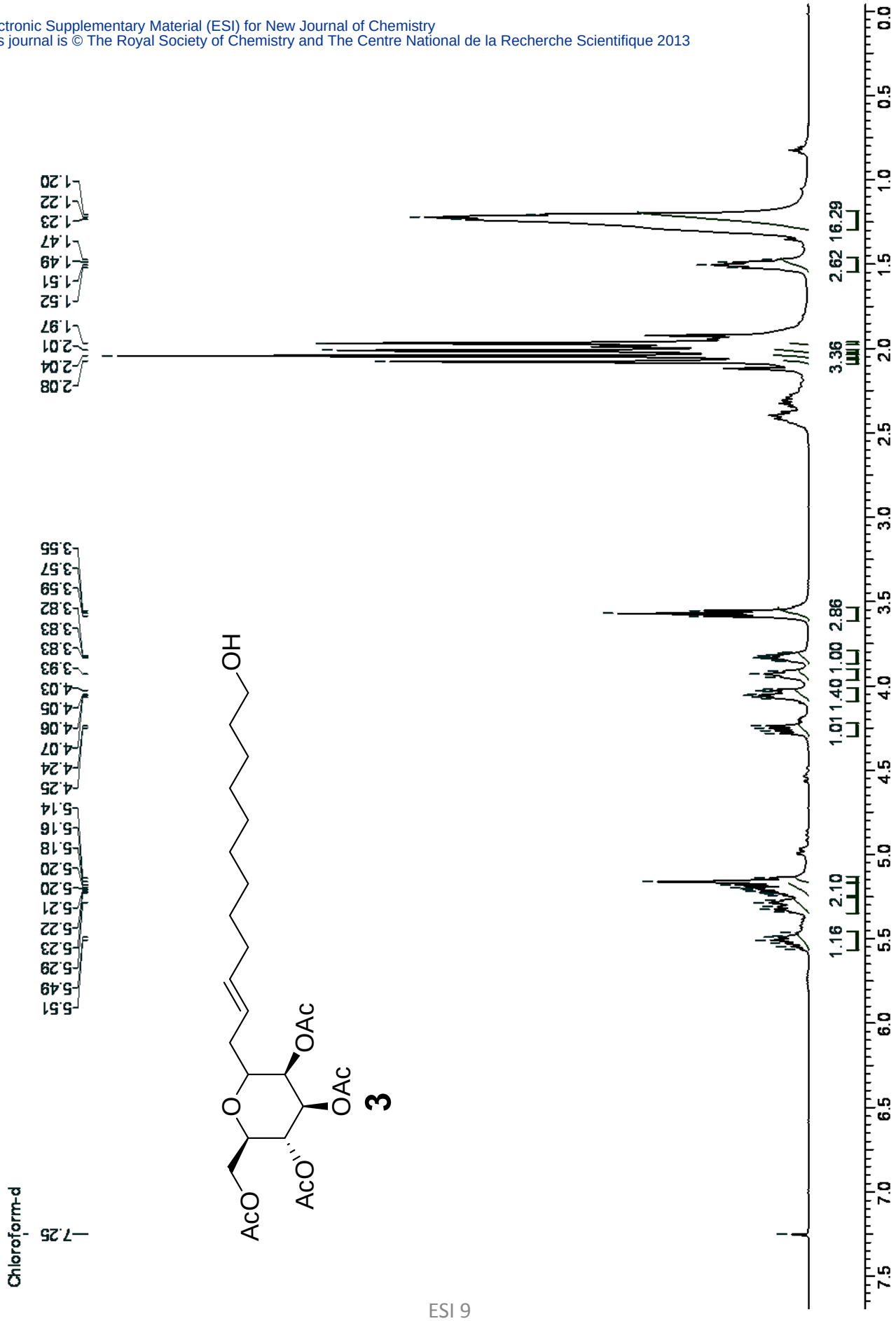
To a solution of alcohol **6** (320 mg, 0.6 mmol) in CCl_4 (2 mL)-CH_3CN (2 mL)-H_2O (3 mL), $NaIO_4$ (532 mg, 2.4 mmol) and $RuCl_4.H_2O$ (3.2 mg, 0.01 mmol) were added. The biphasic suspension was stirred vigorously for 1 h. The color was changed from wine red to pale brown. Reaction mixture was extracted with DCM (3 x 25 mL). The combined organic layer was dried (Na_2SO_4), filtered and concentrated. Diethyl ether (20 mL) was added to it, filtered through a celite pad to remove metal impurities, the filtrate was concentrated at reduced pressure and purified by column chromatography (3:1 petroleum ether/ethyl acetate) to furnish the tetraacetate acid **8** (281 mg, 86%) as a colorless liquid. $[\alpha]_D^{25}$ = +54.4 (*c* 1.1, $CHCl_3$). IR ($CHCl_3$): ν 3401, 2929, 1748, 1710,

1368, 1216, 1034, 756 cm^{-1}. ^1H NMR (400 MHz, $CDCl_3$): δ 1.27 (br s, 15H), 1.45–1.51 (m, 2H), 1.63 (qui, *J* = 7.3, 14.5 Hz, 2H), 1.73–1.81 (m, 1H), 2.03 (s, 3H), 2.04 (s, 3H), 2.05 (s, 3H), 2.09 (s, 3H), 2.35 (t, *J* = 7.5 Hz, 2H), 3.81 (ddd, *J* = 2.2, 5.3, 8.8 Hz, 1H), 4.09 (dd, *J* = 2.3, 12.0 Hz, 1H), 4.16 (ddd, *J* = 2.2, 5.3, 8.8 Hz, 1H), 4.23 (dd, *J* = 5.3, 12.0 Hz, 1H), 4.98 (dd, *J* = 9.1. 9.3 Hz, 1H), 5.07 (dd, *J* = 5.8, 9.5 Hz, 1H), 5.33 (dd, *J* = 9.1. 9.3 Hz, 1H). ^{13}C NMR (100 MHz, $CDCl_3$) : δ 20.5 (q), 20.6 (q, 3C), 24.6 (t), 24.8 (t), 25.1 (t), 28.9 (t), 29.1 (t), 29.1 (t), 29.2 (t), 29.4 (t), 29.4 (t, 2C), 29.5 (t), 62.3 (t), 68.4 (d), 68.9 (d), 70.4 (d, 2C), 72.5 (d), 169.5 (s), 169.6 (s), 170.2 (s), 170.6 (s), 179.3 (s) ppm. ESI-MS: *m/z* 553.3 (100%, $[M+Na]^+$). Anal. Calcd for $C_{26}H_{42}O_{11}$: C, 58.85; H, 7.98. Found: C, 58.80; H, 8.02.

1-(α-D-Glucopyranosyl)-12-dodecanoic acid (2)



To a solution of acid **8** (200 mg, 0.4 mmol) in 5 mL of dry methanol, K_2CO_3 (156 mg, 1.1 mmol) was added. The suspension was stirred at room temperature for 1 h. Reaction mixture was filtered through a celite pad, washed with methanol and the filtrate was concentrated and purified by column chromatography (8:1:1 $CHCl_3$/MeOH/AcOH) to furnish *C*-glycosyl acid **2** (125 mg, 90%) as a white color solid. $[\alpha]_D^{25}$ = +46.5 (*c* 4.0, MeOH). IR ($CHCl_3$): ν 3365, 2853, 1709, 1569, 1455, 1377, 1032, 721 cm^{-1}. ^1H NMR (400 MHz, MeOH-d_4): δ 1.30 (br s, 16H), 1.59–1.65 (m, 4H), 2.22 (t, *J* = 7.3 Hz, 2H), 3.25 (dd, *J* = 8.5, 9.3 Hz, 1H), 3.39 (ddd, *J* = 2.2, 5.3, 8.0 Hz, 1H), 3.52 (t, *J* = 8.8 Hz, 1H), 3.60 (dd, *J* = 5.5, 9.5 Hz, 1H), 3.63 (dd, *J* = 5.3, 11.5 Hz, 1H), 3.77 (dd, *J* = 2.5, 11.8 Hz, 1H), 3.80 (dt, *J* = 5.0, 10.0 Hz, 1H). ^{13}C NMR (100 MHz, MeOH-d_4): δ 25.4 (t), 26.7 (t), 27.0 (t), 30.6 (t), 30.6 (t), 30.7 (t), 30.7 (t, 5C), 63.1 (t), 72.4 (d), 73.1 (d), 74.3 (d), 75.3 (d), 77.3 (d) ppm. ESI-MS: *m/z* 385.4 (100%, $[M+Na]^+$). Anal. Calcd for $C_{18}H_{34}O_7$: C, 59.65; H, 9.45. Found: C, 59.69; H, 9.49.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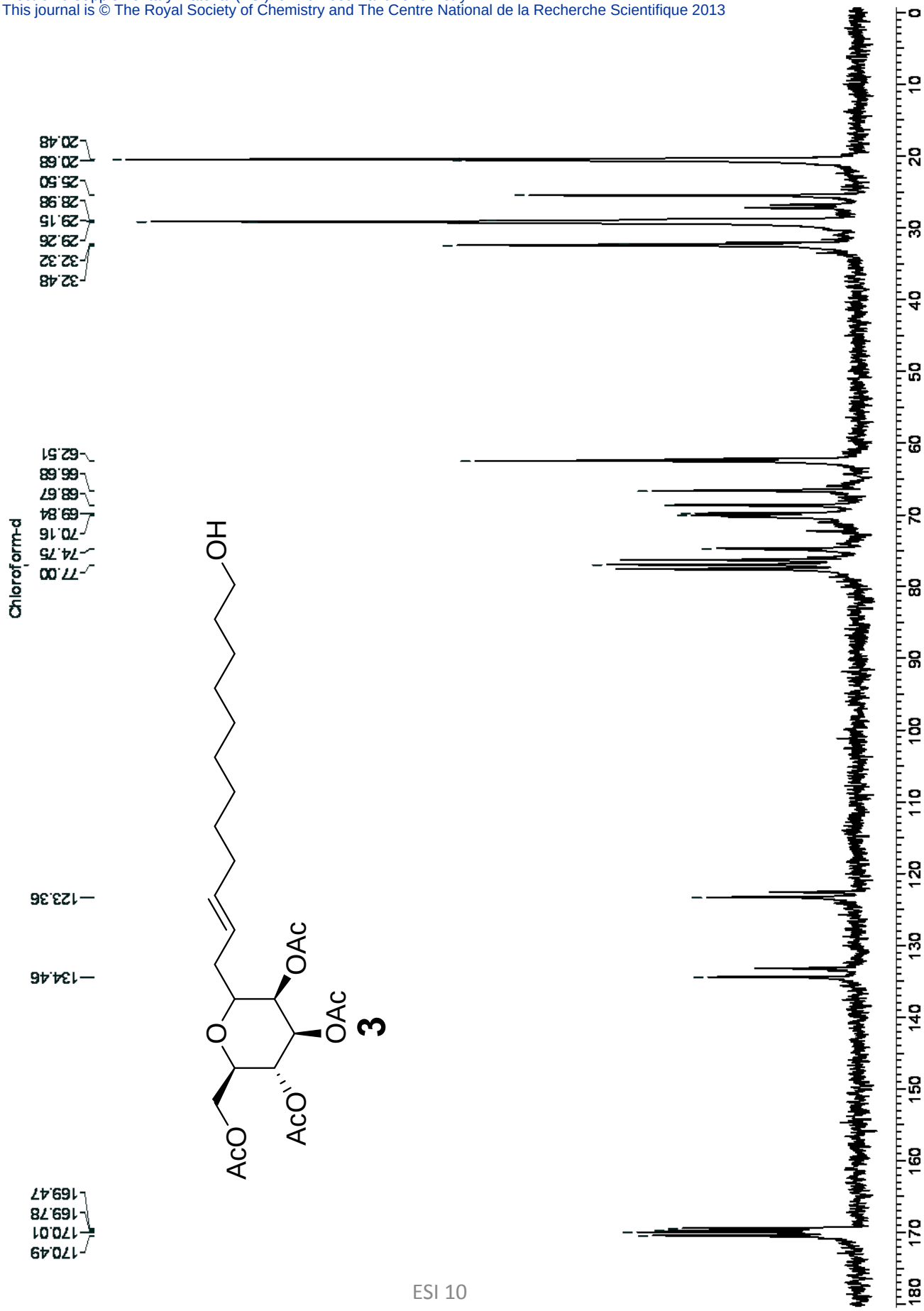
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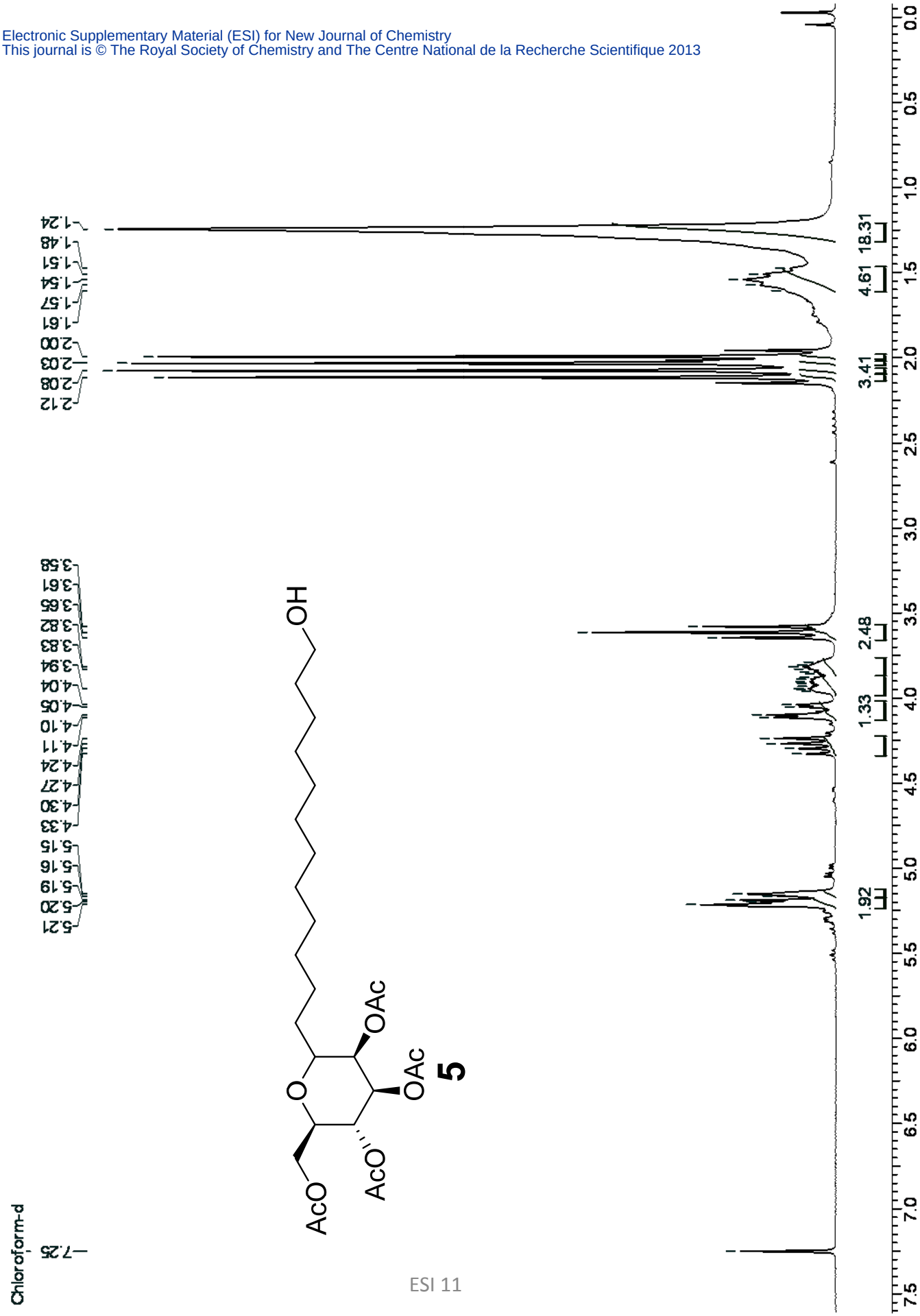
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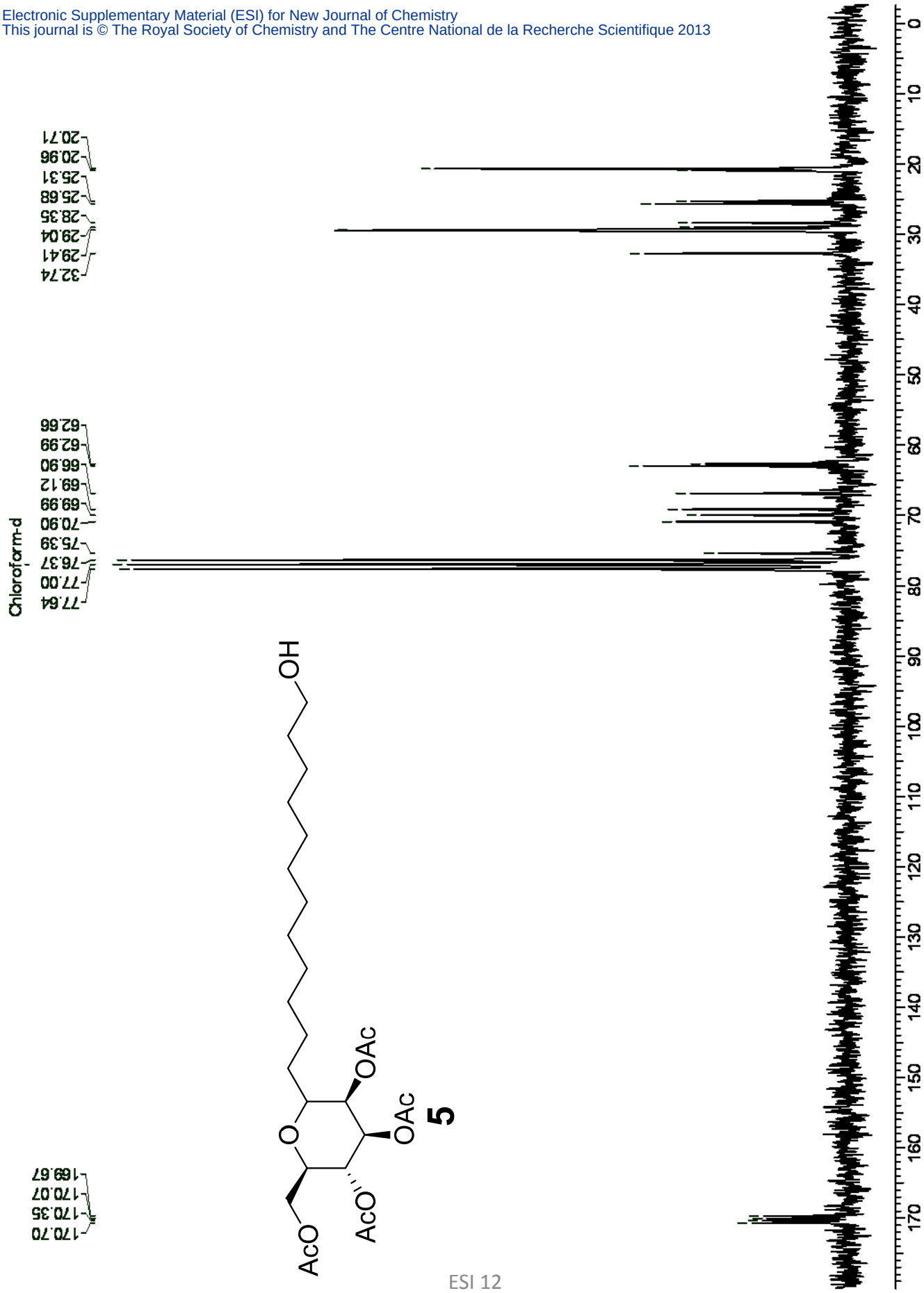
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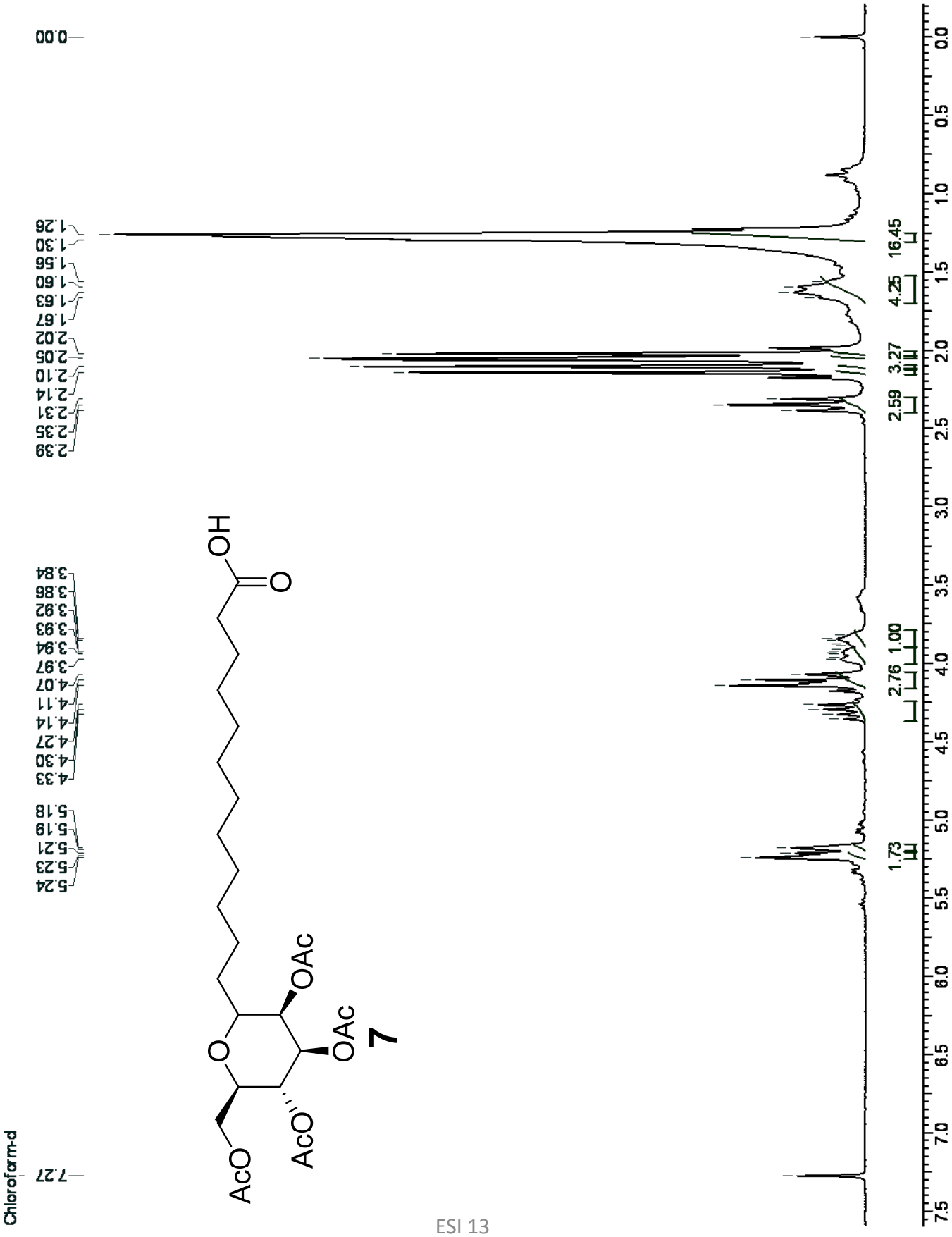
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5.19
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4.27
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4.11
4.07
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3.92
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2.05
2.02
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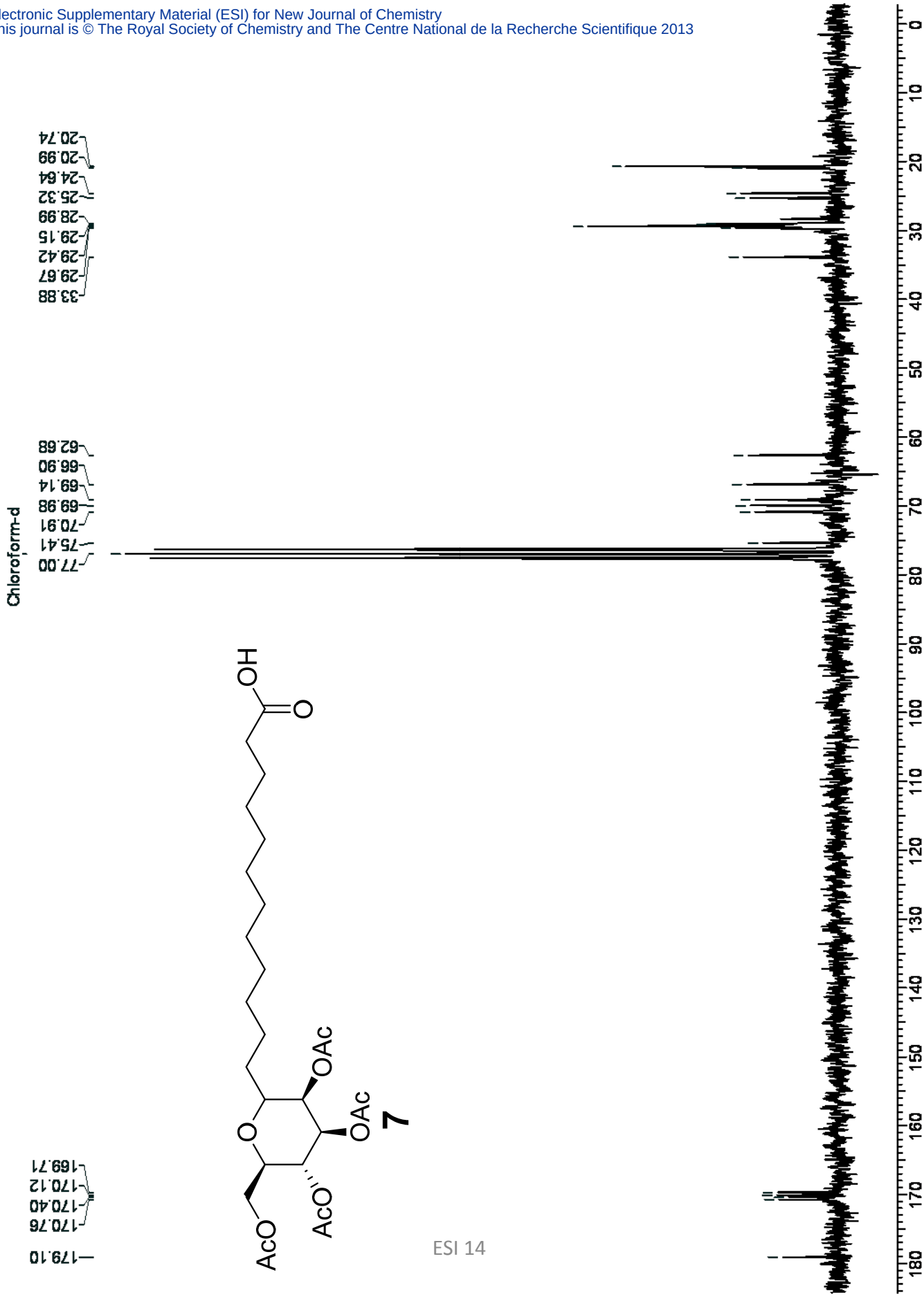
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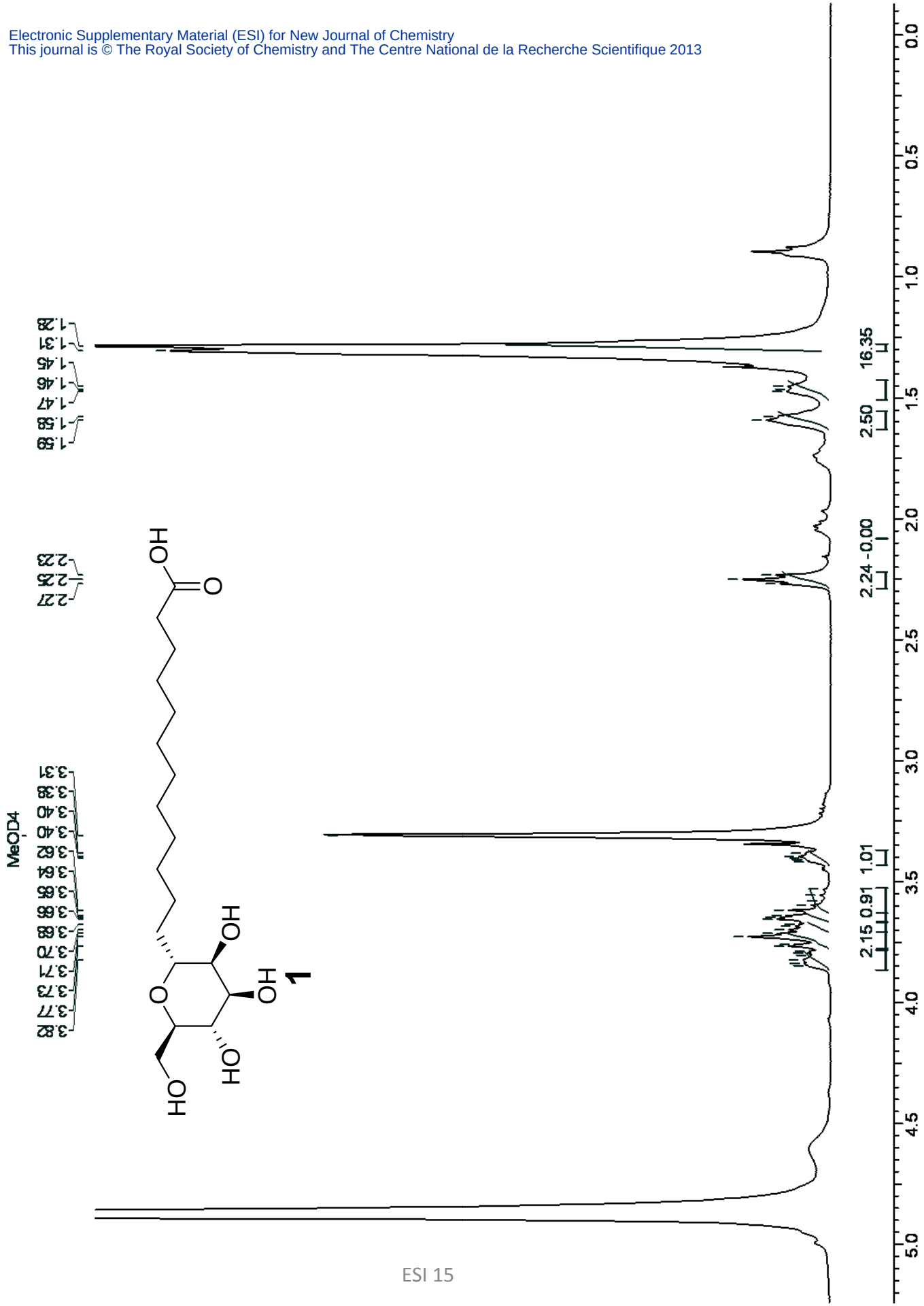
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3.38
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1.59
1.58
1.47
1.46
1.45
1.31
1.28
HO
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OH
OH
O
O
OH
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0.5
1.0
1.5
2.0
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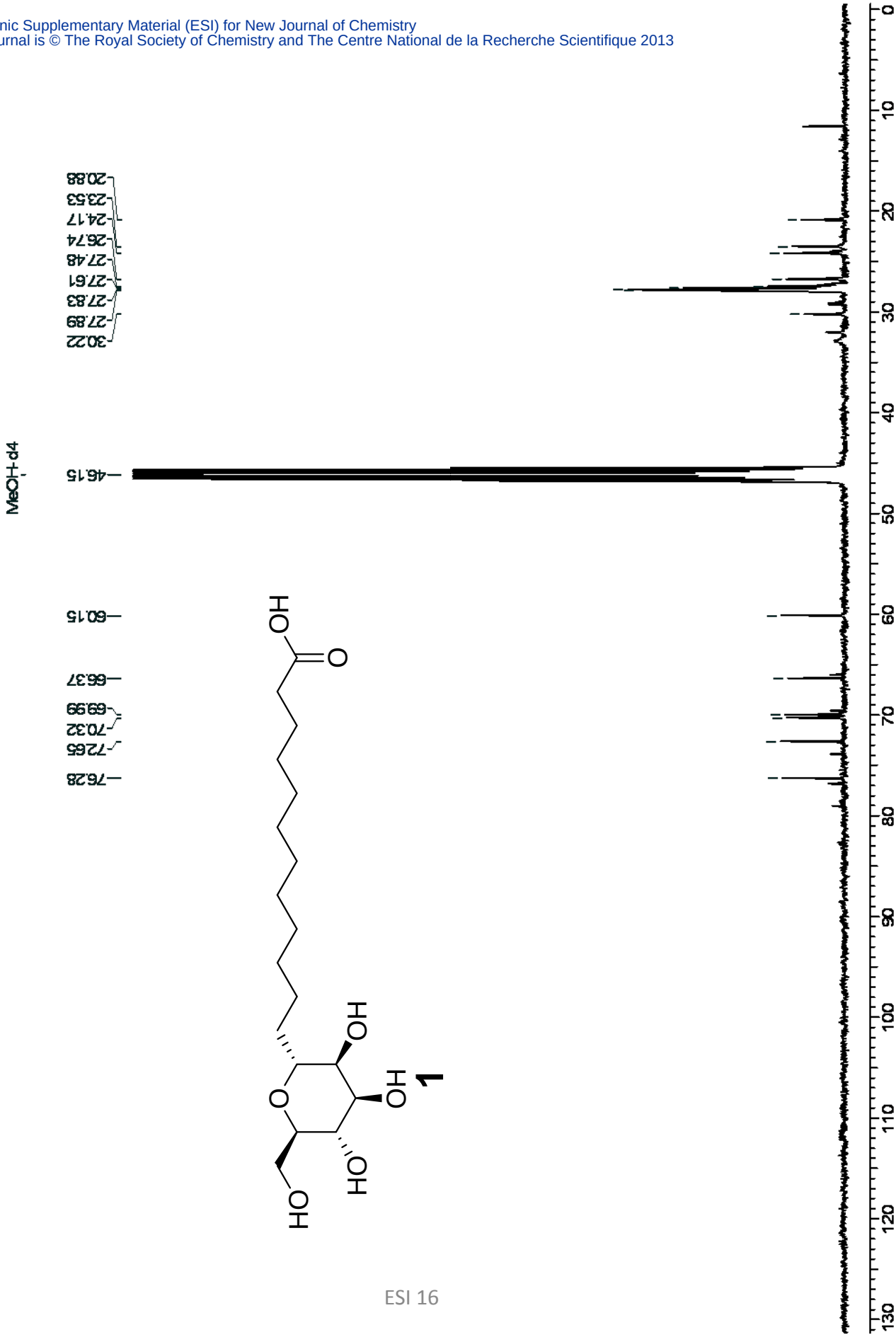
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72.65
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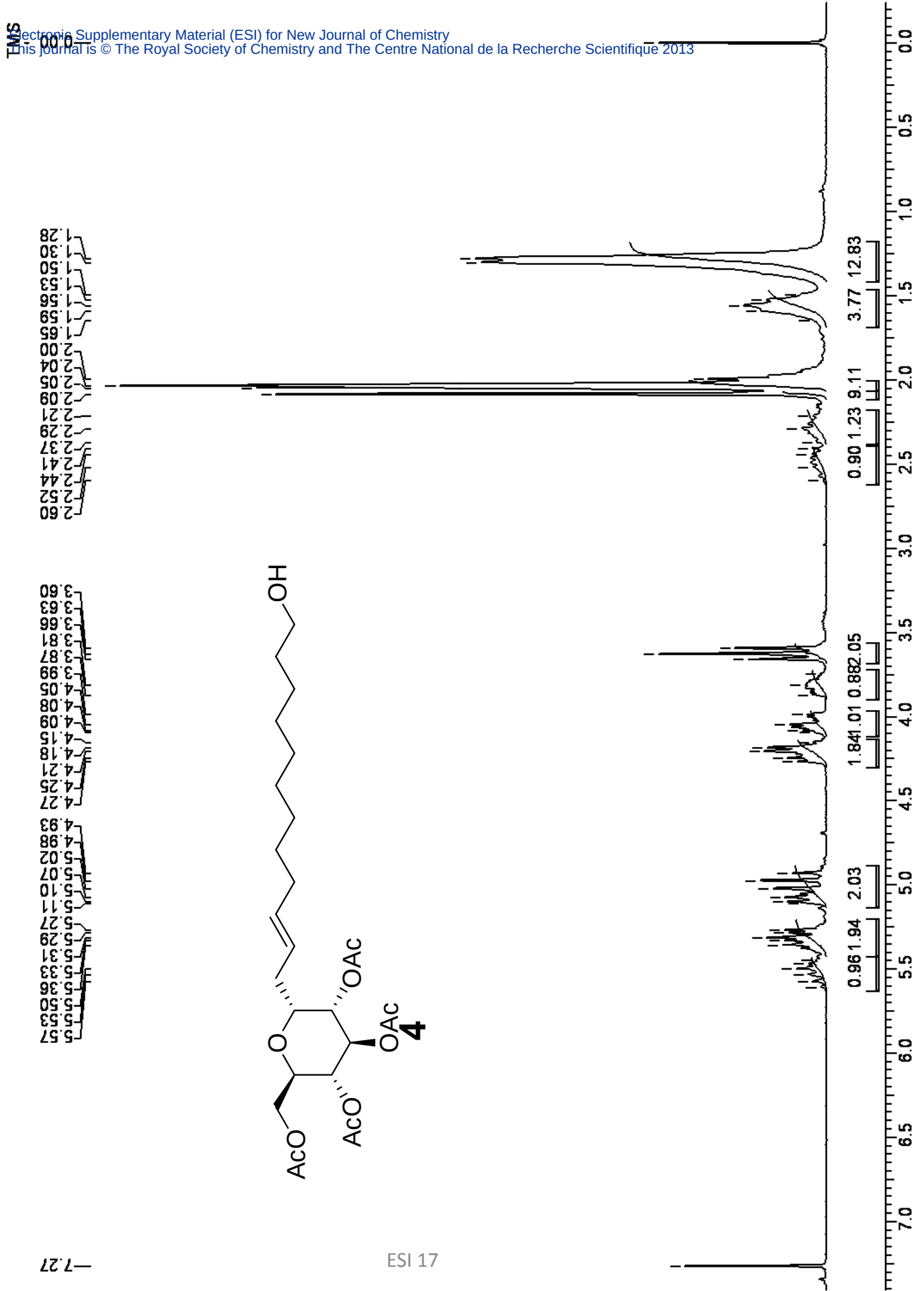
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4.18
4.15
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3.66
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1.53
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1.28
OH
O
AcO
AcO
OAc
OAc
4
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2.03
1.84 1.01
0.88 2.05
0.90 1.23
9.11
3.77
12.83
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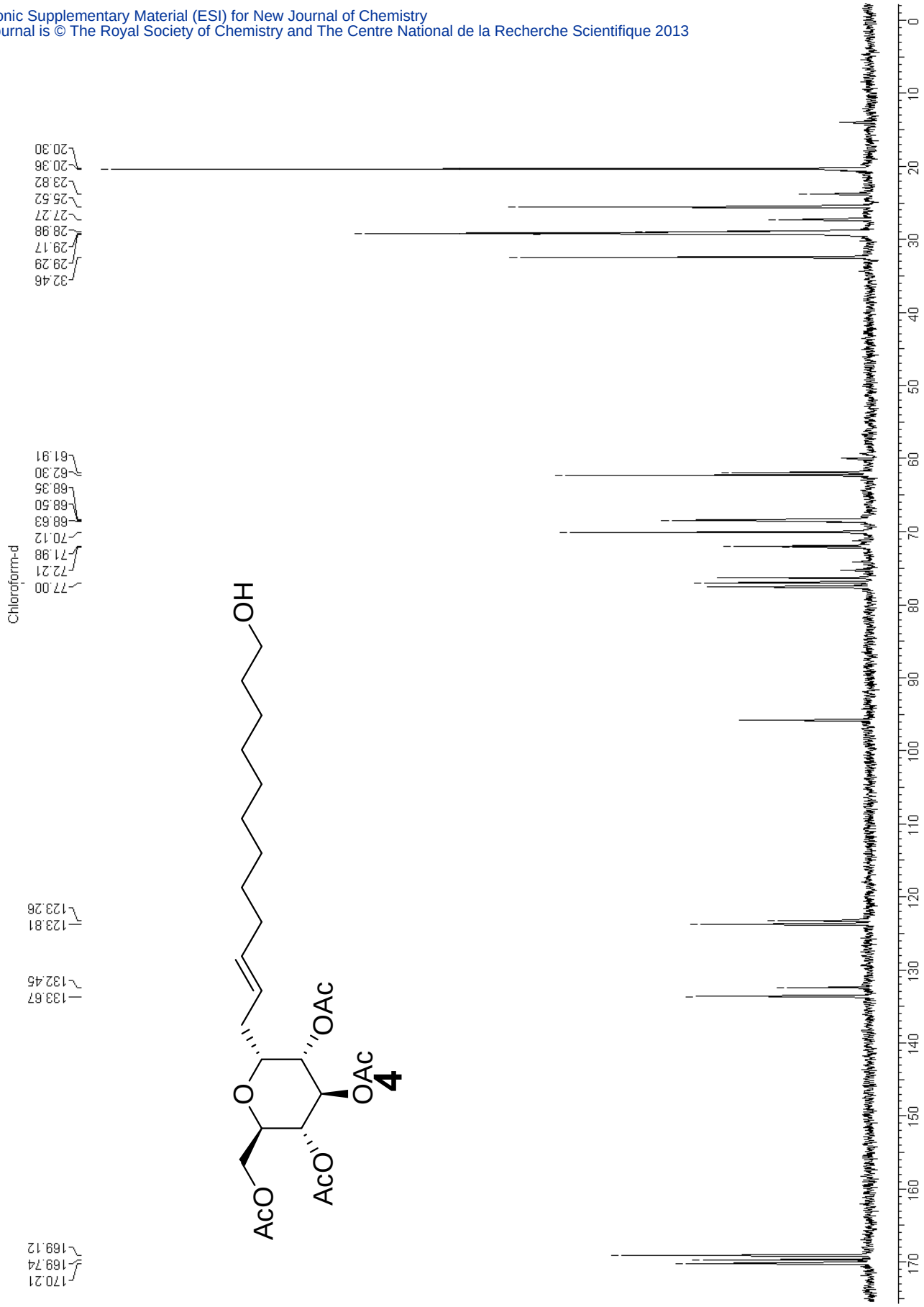
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29.29
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O
OH
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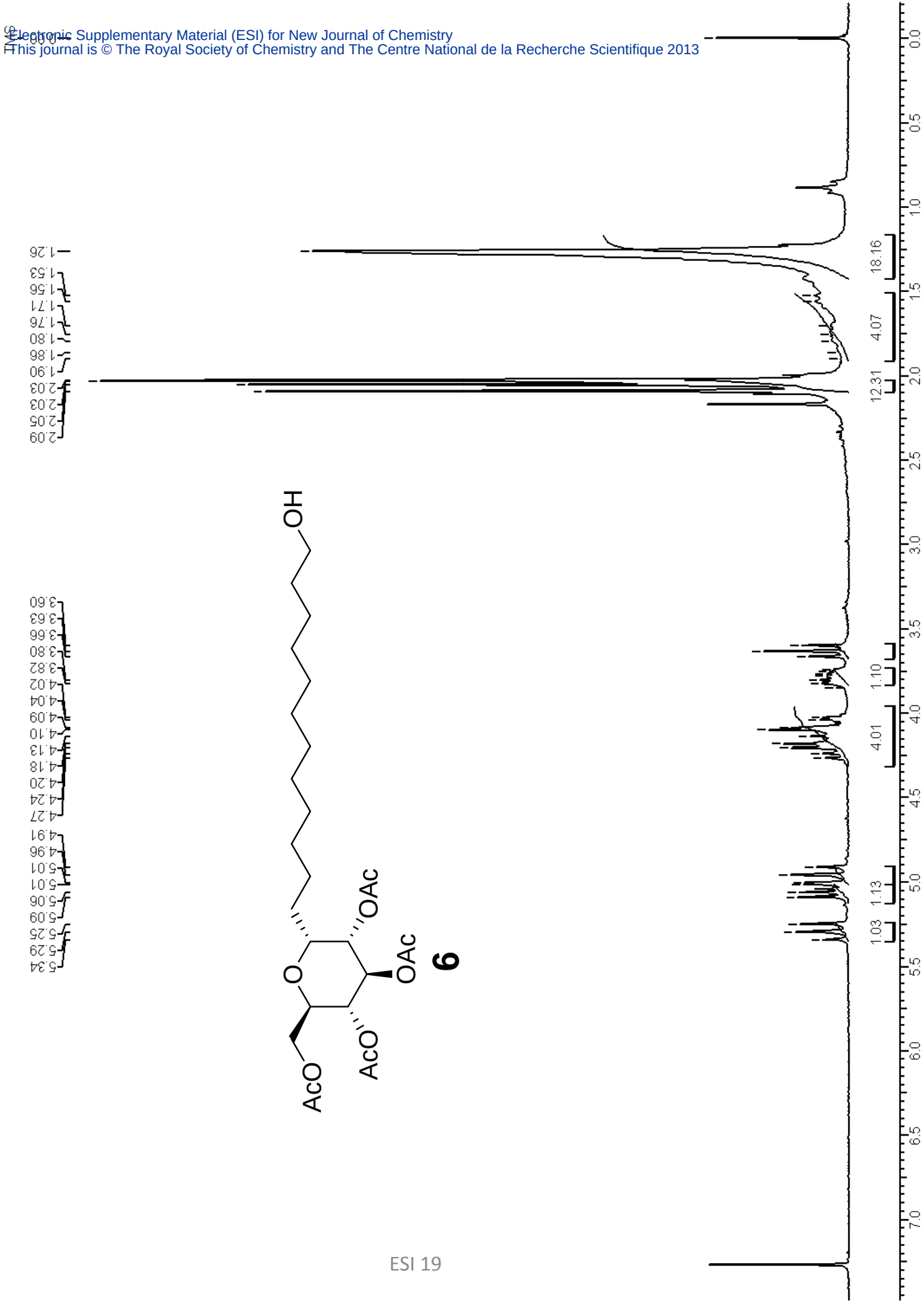

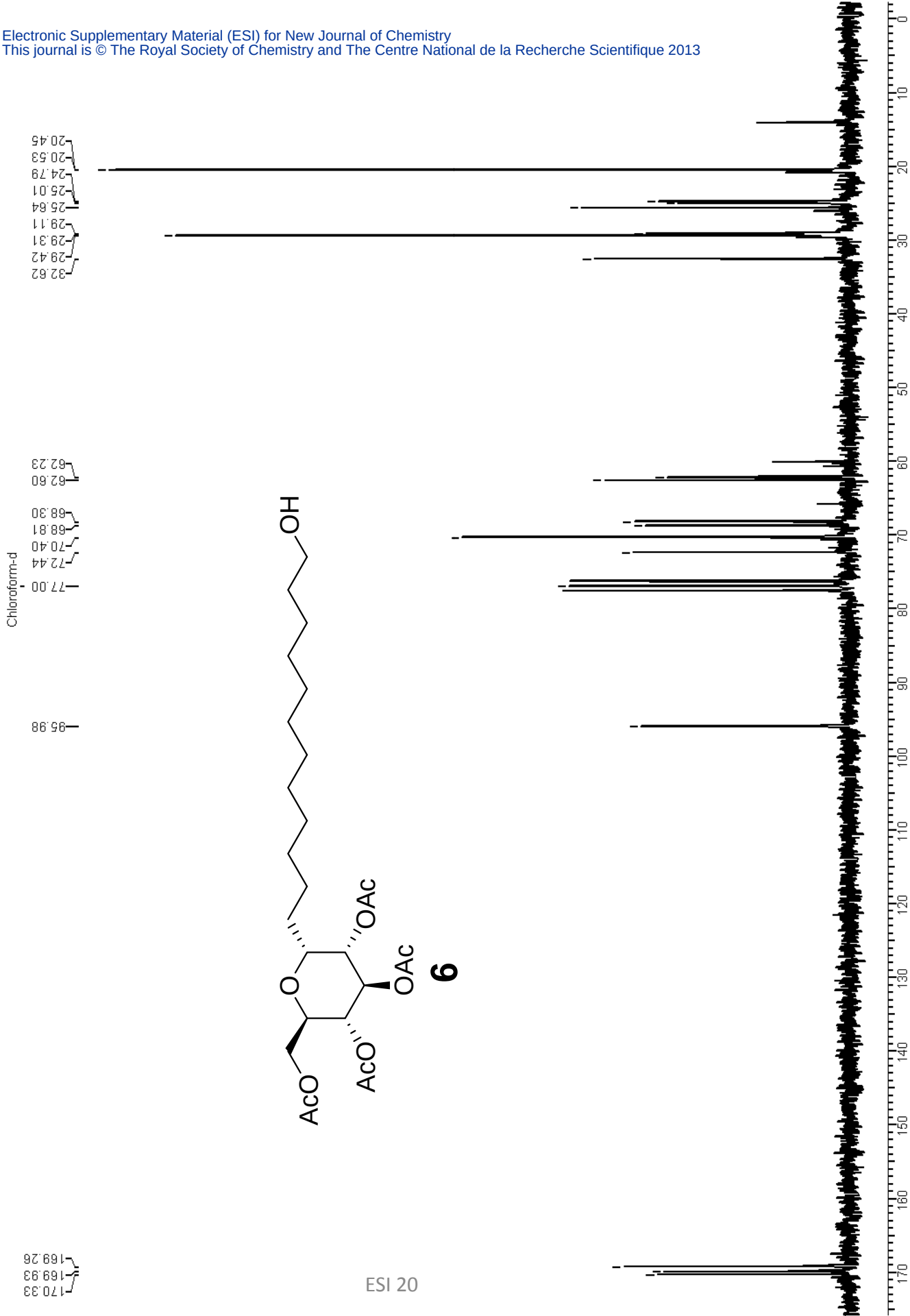
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50
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140
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OAc
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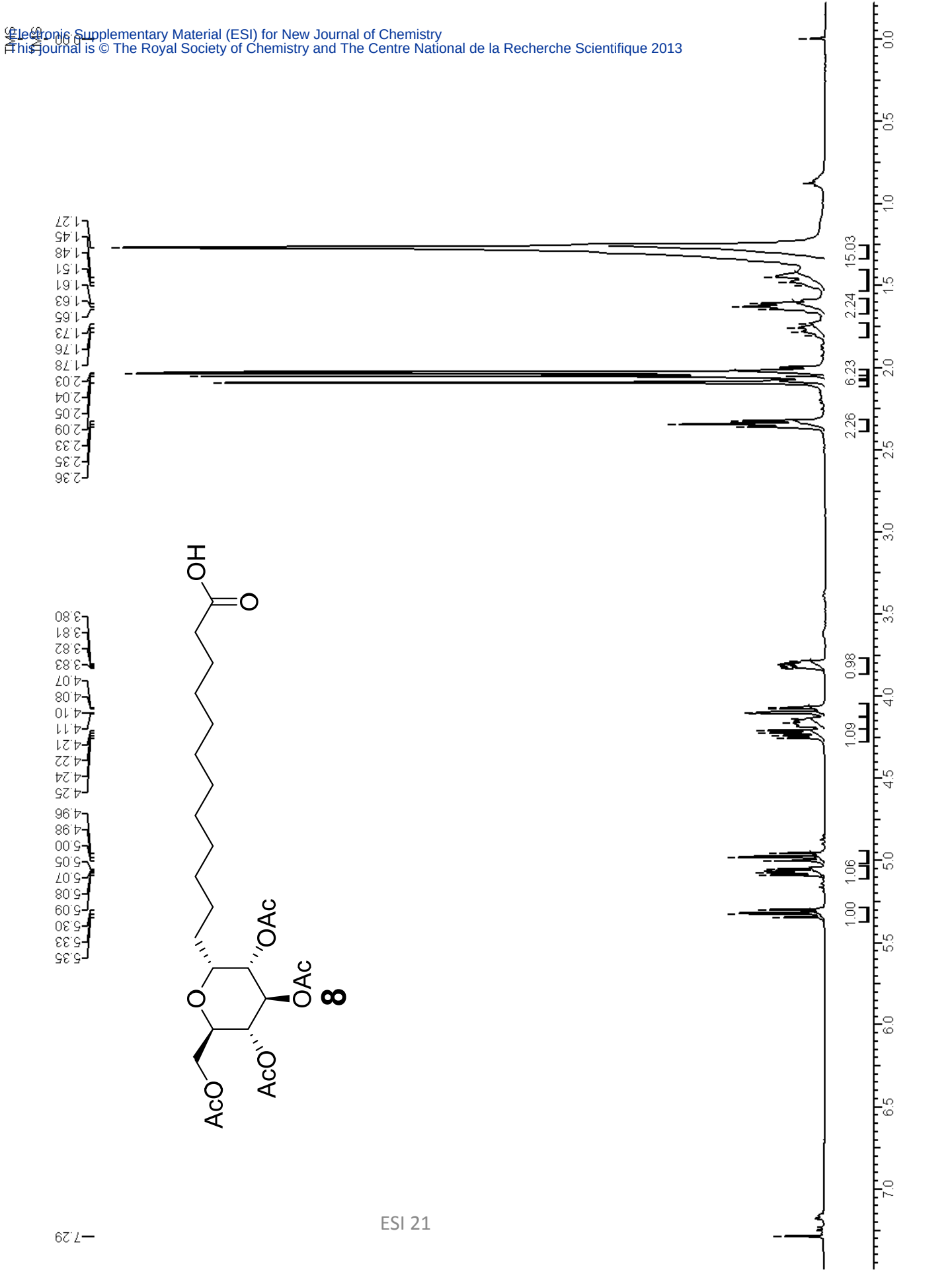

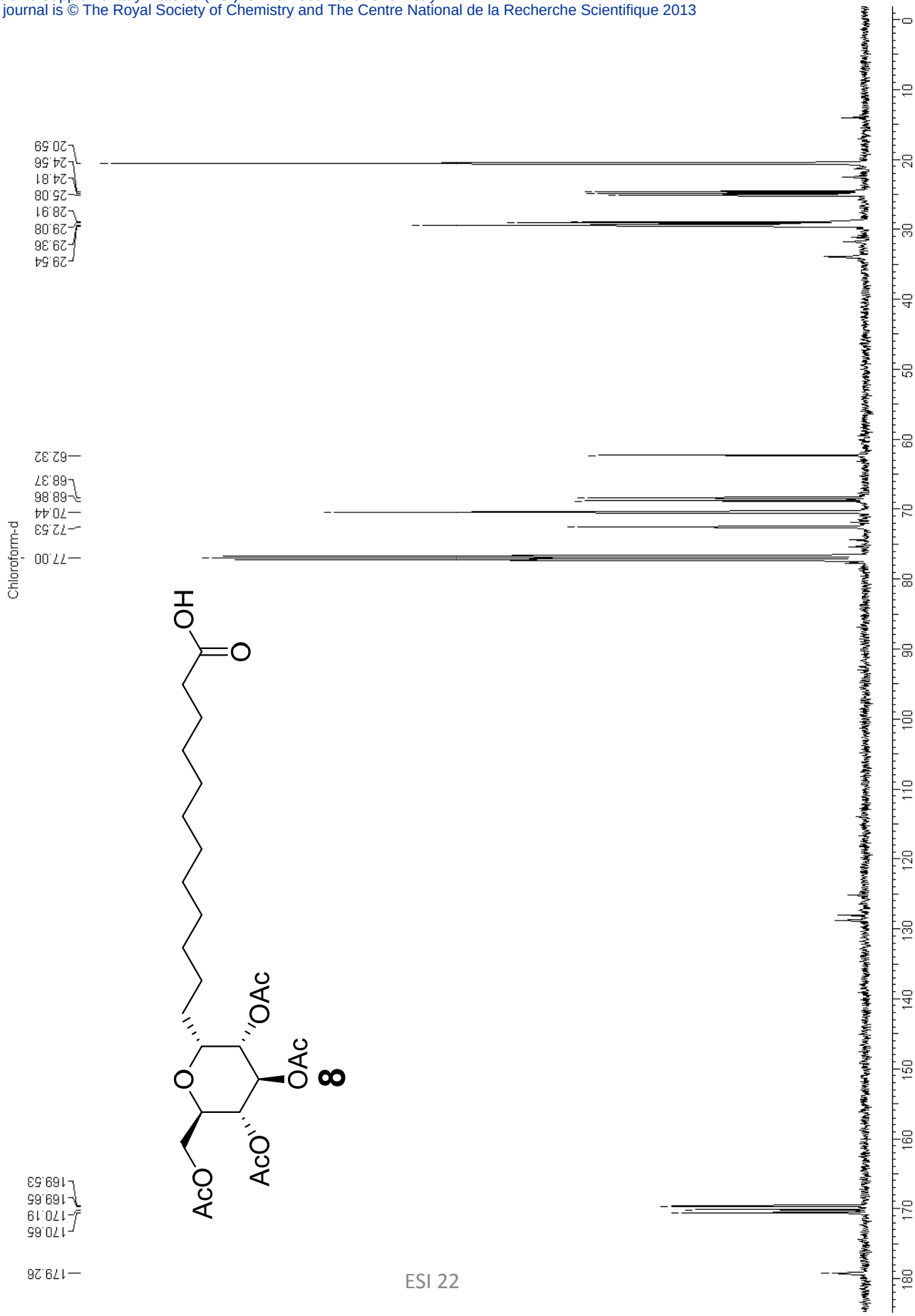
Chloroform-d
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170.65
77.00
72.53
70.44
68.86
68.37
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29.54
29.36
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25.08
24.81
24.56
20.59
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AcO
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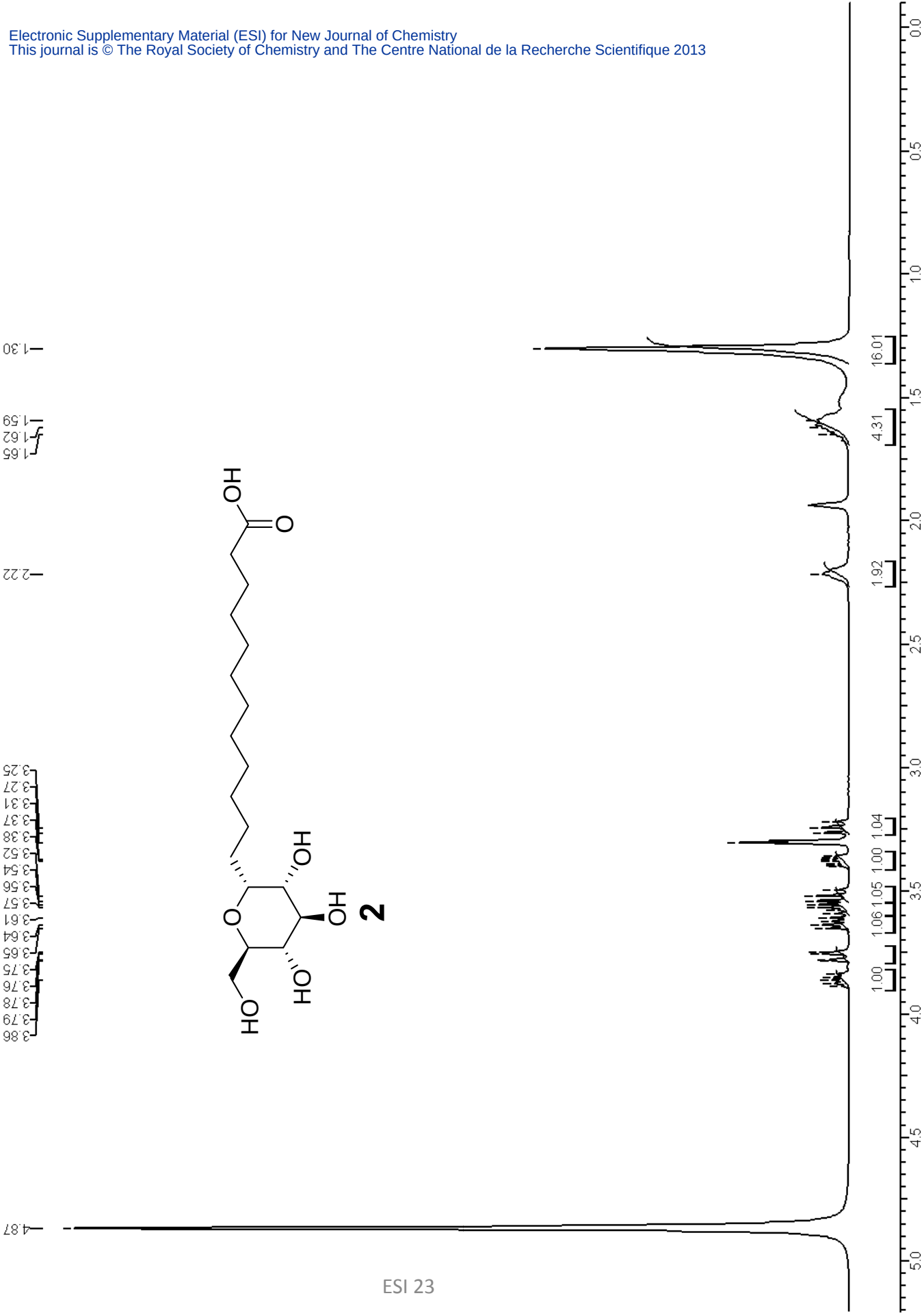
OAc
O
O
OH
8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OH
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3.64
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3.27
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1.05
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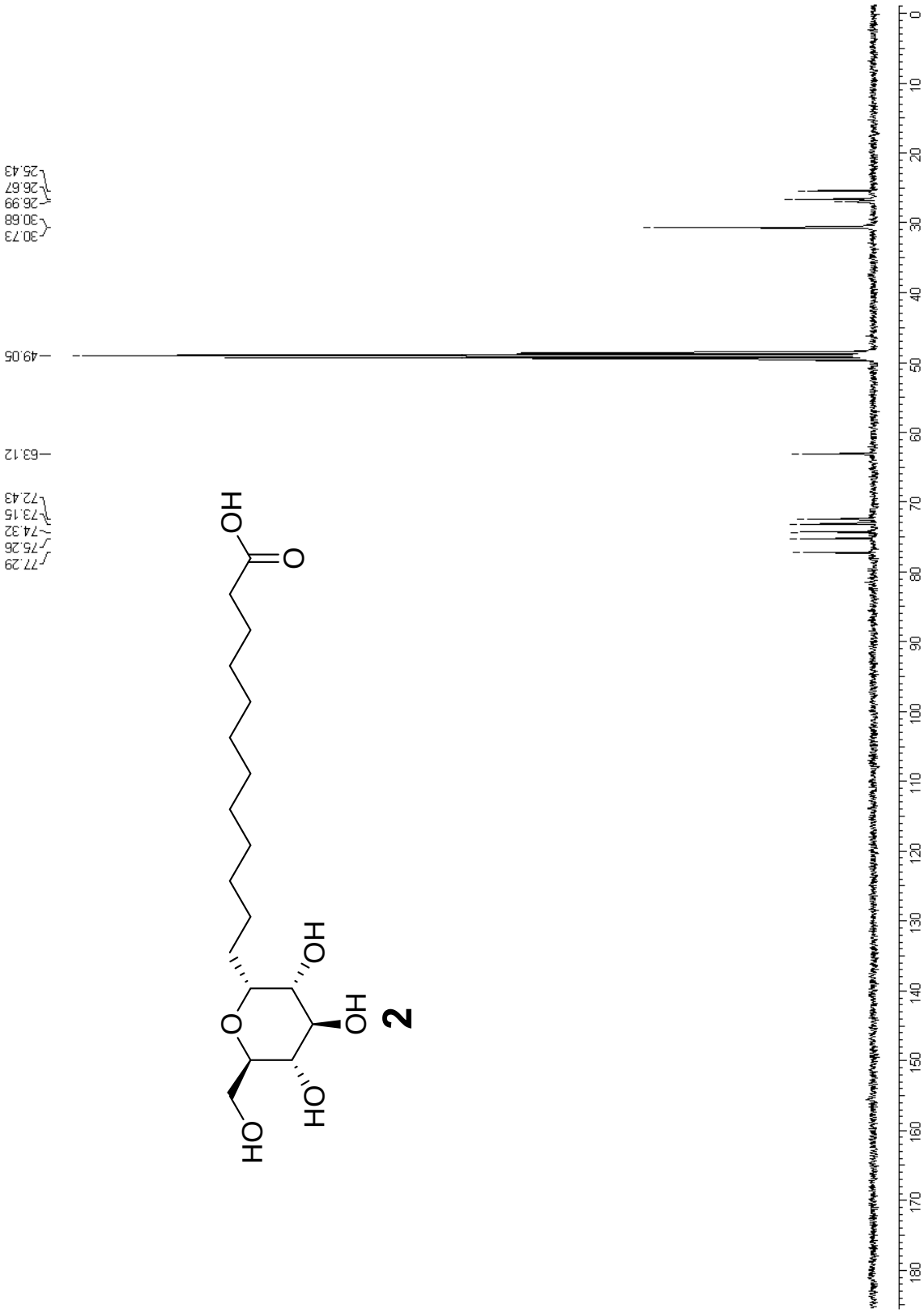
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63.12
49.05
30.73
30.68
26.99
26.67
25.43
2

Determination of amount of sugar in the nanoparticle dispersion

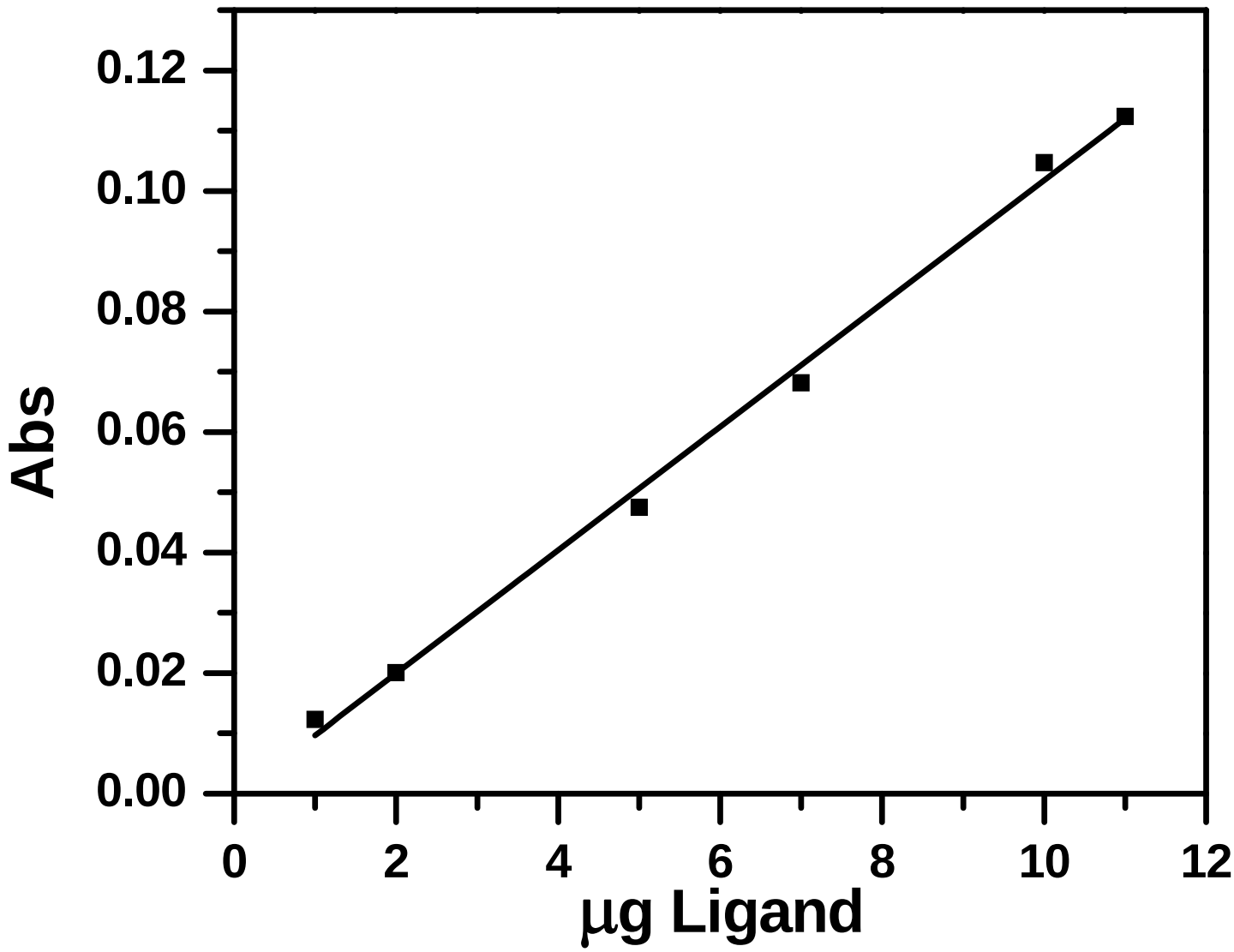

Figure S1. plot of absorbance vs free ligand concentration @620 nm

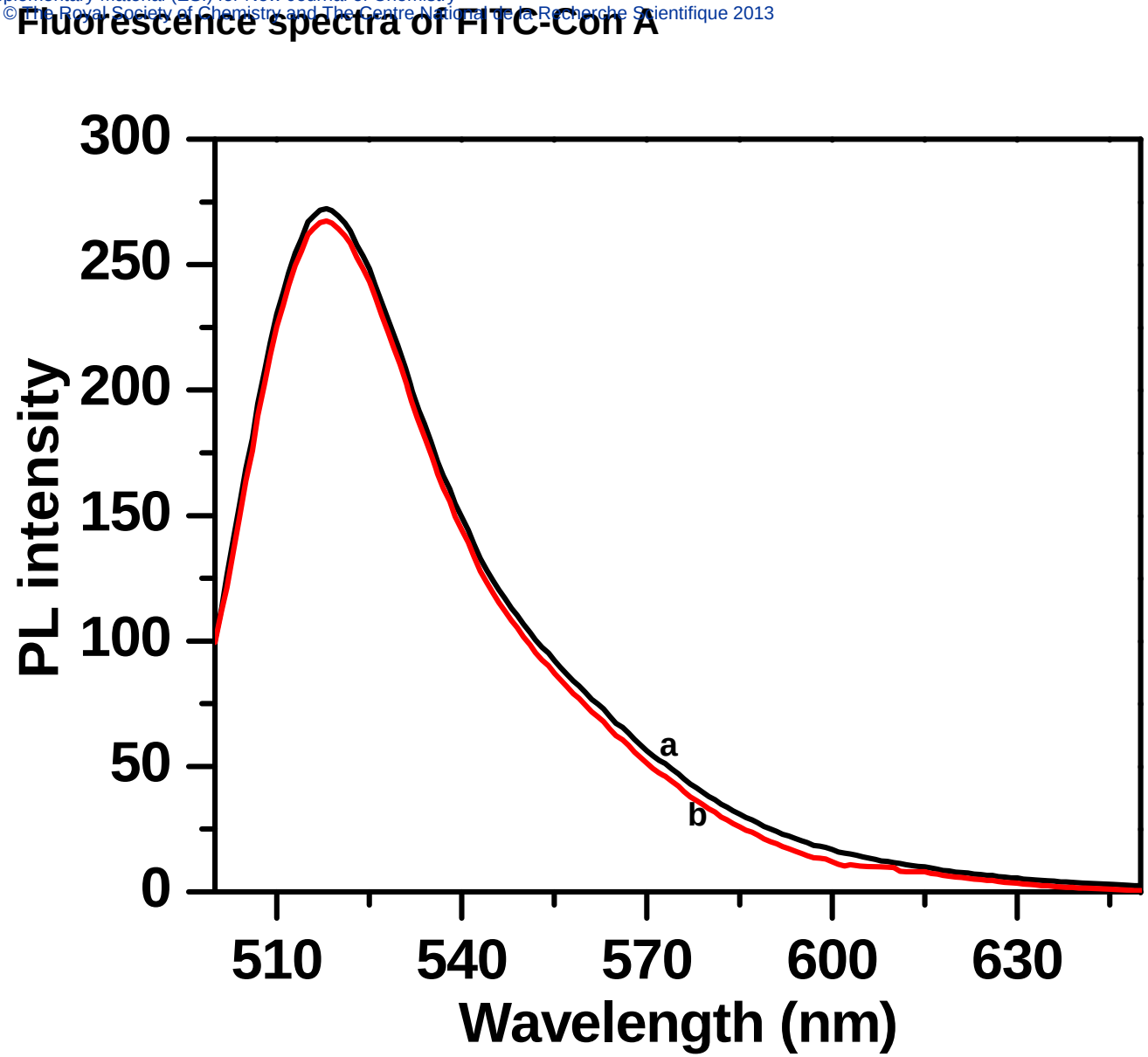

Figure S2. fluorescence intensity of FITC-Con A (10 μg/mL) (a) as prepared and (b) after 8 hrs, excited @ 490 nm