SUPPORTING INFORMATION FOR:

Short Synthesis of 3-Hydroxymethyl Xylitol and Structure Revision of the Anti-Diabetic Natural Product from *Casearia esculenta*

Ruomeng Wang, Michael N. Paddon-Row,[†] and Michael S. Sherburn*

Research School of Chemistry, Australian National University, Canberra, ACT 0200, Australia

sherburn@rsc.anu.edu.au

[†] Current address: School of Chemistry, The University of New South Wales, Sydney, NSW 2052, Australia.

CONTENTS

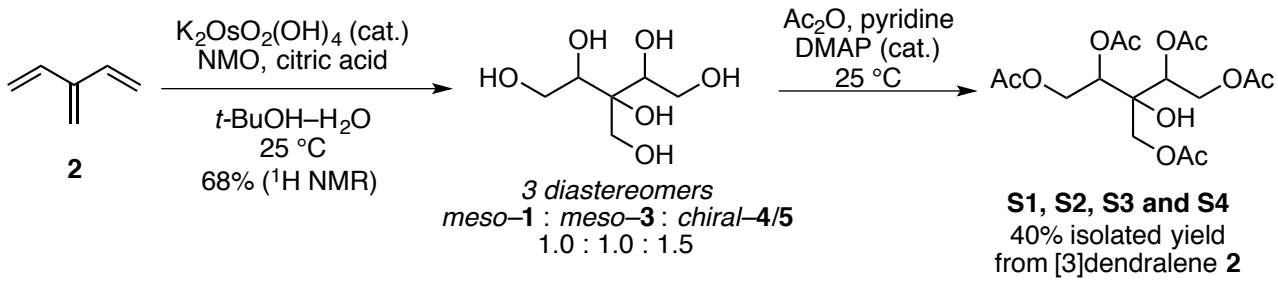
1 Experimental Section

1.1 General methods

^{1}H NMR spectra were recorded at 800 MHz and 400 MHz using a Bruker AVANCE 800 and a Varian MR400 spectrometer. Residual chloroform (δ 7.26 ppm) was used as the internal reference for ^{1}H NMR spectra recorded in this solvent. Acetonitrile (δ 2.06 ppm) was added as an internal reference for ^{1}H NMR spectra recorded in D_2O. Coupling constants (J) are quoted to the nearest 0.1 Hz. ^{13}C NMR spectra were recorded at 100 MHz using a Varian MR400 spectrometer. The central line of the deuterochloroform signal (δ 77.1 ppm) was used as an internal reference for ^{13}C NMR spectra recorded in this solvent. The acetonitrile signals (δ 1.47 and 119.68 ppm) were used as an internal reference for ^{13}C NMR spectra recorded in D_2O. Assignment of carbon signals was assisted by DEPT or HSQC experiments. IR spectra were recorded on a Perkin–Elmer Spectrum One spectrometer as thin films on sodium chloride plates for oils or as potassium bromide discs for solid products. Low resolution mass spectra were recorded on a Finnigan Polaris Q ion trap mass spectrometer using electron impact (EI+) ionization mode at 40 or 70 eV, or a VG Quattro II triple quadrupole MS for electrospray ionization (ESI). High resolution mass spectra were recorded on a VG Autospec mass spectrometer operating at 70 eV for EI, or a Bruker Apex3 4.7T FTICR-MS for ESI. Melting points were measured on a Reichert melting point stage and are uncorrected. Analytical thin layer chromatography was performed using Merck silica gel plates, pre-coated with silica gel 60 F243 (0.2 mm). Flash chromatography employed 230–400 mesh silica gel. Preparative HPLC was performed using a Waters 600E instrument. De-acetylation reactions were conducted under a positive pressure of dry argon or nitrogen in oven-dried glassware. MeOH was dried using a solvent purification system based on that described by Pangborn and co-workers.[1] Optical rotations were measured with a Perkin–Elmer 343 optical polarimeter.

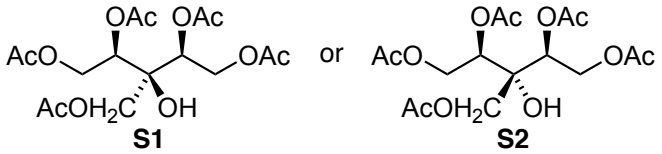
1.2 Preparation of compounds 1, 3, 4 and 5

Exhaustive dihydroxylation reaction of [3]dendralene 2 and acetylation reaction of hexa-ols



To a solution of citric acid (1.44 g, 7.49 mmol) in *t*-BuOH–water (4.7 mL, 1:1, v/v) was added [3]dendralene **2**[3] (100.0 mg, 1.248 mmol), $K_2Os(OH)_4$ (92.0 mg, 0.250 mmol) and a 50% aqueous solution of NMO (0.96 mL, 4.1 mmol). The resulting mixture was stirred in a sealed tube at 25 °C for 5 days. The reaction was quenched by the addition of saturated aqueous $Na_2S_2O_5$ solution (20 mL) and the resulting mixture was stirred for an hour. The *t*-BuOH was removed under reduced pressure and the resulting aqueous solution was freeze-dried to remove water. To the solid mixture was added acetic anhydride (15.3 mL, 152 mmol), pyridine (1.13 mL, 14.0 mmol) and DMAP (11.4 mg, 0.0936 mmol). The reaction mixture was stirred at 25 °C for 24 hours then poured into water (100 mL). The aqueous solution was extracted with dichloromethane (3 × 100 mL), the organic layer was washed with saturated $NaHCO_3$, followed by 1 M aqueous HCl (300 mL) then brine (300 mL) and dried over $MgSO_4$. The solvent was removed under reduced pressure and the resulting brown oil was purified using silica gel column chromatography, eluting with 40:60 EtOAc/CH_2Cl_2. The mixture of penta-acetoxy compounds was obtained as a colourless oil (216.0 mg, 0.5505 mmol, 44%). The colourless oil was further purified on normal phase chiral HPLC (eluting with 20:80 isopropanol:hexane on an AS-H semi-preparative column, 4 mL/min) to give three fractions: R_t = 6.9 min (one pure chiral enantiomer); R_t = 8.4 min (mixture of the other chiral enantiomer and one of the two *meso*-compounds); and R_t = 11.11 min (one pure *meso*-compound). The mixed fraction from the first, chiral HPLC separation was separated on normal phase HPLC (eluting with 5:95 isopropanol: hexane on a Phenomenex Luna 5 μm, 150 mm × 21.2 mm silica column, 20 mL/min) to give two fractions: R_t = 8.7 min (the other pure chiral enantiomer) and R_t = 10.5 min (the other pure *meso*-compound). The four stereoisomers were isolated as colourless oils. It was not possible to identify the assign absolute configurations to each of the two chiral isomers or each of the two *meso*-isomers.

Characterization data for the *meso*-isomers S1 and S2:

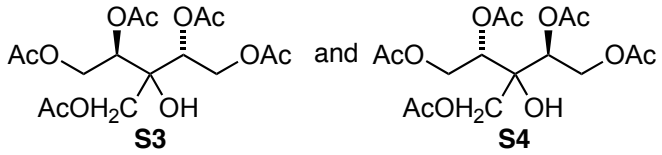


S1 **S2**

For the compound obtained pure by normal phase HPLC purification with a retention time R_t = 10.5 min; R_f = 0.47; 40:60 EtOAc/CH_2Cl_2; ^{1}H NMR (800 MHz, $CDCl_3$) δ 5.30 (dd, J = 7.7, 2.8 Hz, 2H), 4.55 (dd, *J* = 12.2, 2.8 Hz, 2H), 4.26 (s, 2H), 4.14 (dd, *J* = 12.2, 7.7 Hz, 2H), 2.96 (s, 1H), 2.13 (s, 6H), 2.12 (s, 3H), 2.03 (s, 6H) ppm; ^{13}C NMR (100 MHz, $CDCl_3$) δ 170.9 (C), 170.6 (C), 170.1 (C), 74.9 (C), 71.8 (CH), 64.3 (CH_2), 62.3 (CH_2), 20.9 (CH_3), 20.8 (CH_3), 20.7 (CH_3) ppm; IR (thin film): ν_{max} = 3465, 2916, 2849, 1743 cm^{-1}; MS (+ve, ESI): *m/z* (%): 415 (100) $[M+Na]^+$; HRMS: calcd for $C_{16}H_{24}O_{11}Na$ $[M+Na]^+$: 415.1216; found: 415.1201.

For the compound obtained pure by chiral HPLC purification with a retention time R_t = 11.1 min; R_f = 0.47; 40:60 EtOAc/CH_2Cl_2; ^{1}H NMR (800 MHz, $CDCl_3$) δ 5.35 (dd, *J* = 7.7, 3.0 Hz, 2H), 4.52 (dd, *J* = 12.2, 3.0 Hz, 2H), 4.19 (s, 2H), 4.11 (dd, *J* = 12.2, 7.8 Hz, 2H), 3.00 (s, 1H), 2.12 (s, 6H), 2.10 (s, 3H), 2.01 (s, 6H) ppm; ^{13}C NMR (100 MHz, $CDCl_3$) δ 170.6 (C), 170.5 (C), 170.3 (C), 75.1 (C), 71.8 (CH), 63.2 (CH_2), 62.1 (CH_2), 20.9 (CH_3), 20.7 (CH_3), 20.7 (CH_3) ppm; IR (thin film): ν_{max} = 3474, 2965, 2849, 1745 cm^{-1}; MS (+ve, ESI): *m/z* (%): 415 (100) $[M+Na]^+$; HRMS: calcd for $C_{16}H_{24}O_{11}Na$ $[M+Na]^+$: 415.1216; found: 415.1216.

Characterization data for the enantiomers S3 and S4:



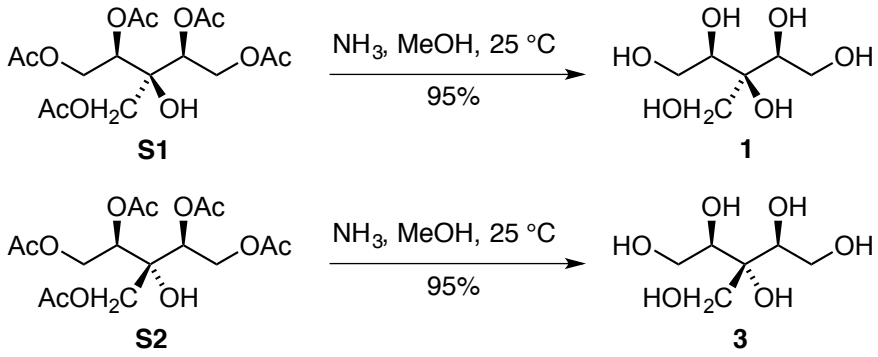
S3 **S4**

R_f = 0.47; 40:60 EtOAc/CH_2Cl_2; ^{1}H NMR (800 MHz, $CDCl_3$) δ 5.35 (dd, *J* = 7.7, 2.9 Hz, 1H), 5.25 (dd, *J* = 7.7, 3.1 Hz, 1H), 4.64–4.57 (m, 2H), 4.36 (d, *J* = 12.1 Hz, 1H), 4.21 (ddd, *J* = 12.0, 7.7, 2.9 Hz, 2H), 4.17 (d, *J* = 12.1 Hz, 1H), 2.97 (s, 1H), 2.12 (s, 3H), 2.09 (s, 3H), 2.06 (s, 3H), 2.05 (s, 3H), 2.04 (s, 3H) ppm; ^{13}C NMR (100 MHz, $CDCl_3$) δ 170.8 (C), 170.7 (C), 170.6 (C), 170.0 (C), 169.8 (C), 74.8 (C), 71.1 (CH), 71.0 (CH), 63.5 (CH_2), 62.7 (CH_2), 62.5 (CH_2), 20.8 (CH_3), 20.8 (CH_3), 20.7 (CH_3), 20.7 (CH_3), 20.7 (CH_3) ppm; IR (thin film): ν_{max} = 3374, 2917, 2849, 1744 cm^{-1}; MS (+ve, ESI): *m/z* (%): 415 (100) $[M+Na]^+$; HRMS: calcd for $C_{16}H_{24}O_{11}Na$ $[M+Na]^+$: 415.1216; found: 415.1215; for the

compound obtained pure by chiral HPLC purification with a retention time R_t = 6.87 min: $[\alpha]_D$ = +170 (*c* 0.23, CH_2Cl_2); for the compound obtained pure by normal phase HPLC purification with a retention time R_t = 8.7 min: $[\alpha]_D$ = –160 (*c* 0.23, CH_2Cl_2).

De-acetylation reactions of the penta-acetoxy compounds.

Meso-compounds 1 and 3:



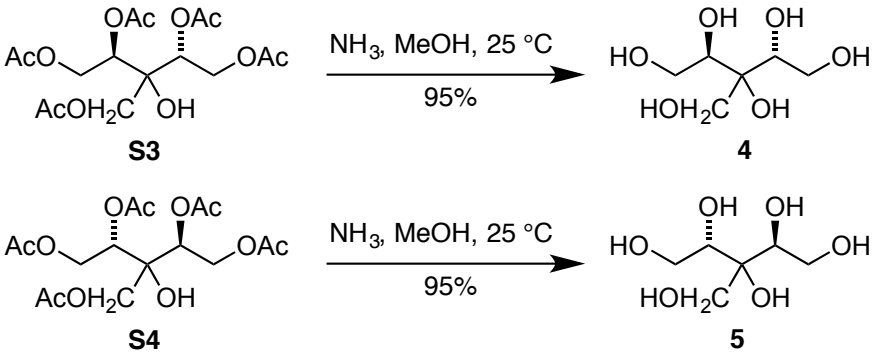
S1 **1**

S2 **3**

To penta-acetoxy compound **S1** or **S2** (for the compound obtained pure by normal phase HPLC purification with a retention time R_t = 10.5 min) (7.6 mg, 0.019 mmol) was added 8 M anhydrous NH_3 in MeOH (0.50 mL, 66 mg, 3.9 mmol). The resulting mixture was stirred in a sealed vial at 25 °C for 2 days. The solvent and acetamide byproduct were removed under reduced pressure to leave the pure deacetylated product **1** or **3** as a colourless oil (3.4 mg, 0.018 mmol, 95%): ^{1}H NMR (800 MHz, D_2O) δ 3.92–3.87 (m, 4H), 3.77 (s, 2H), 3.74 (m, 2H) ppm; ^{13}C NMR (100 MHz, D_2O) δ 76.7 (C), 73.9 (CH), 62.6 (CH_2), 62.3 (CH_2) ppm; IR (thin film): ν_{max} = 3368, 2945, 1647, 1594 cm^{-1}; MS (+ve, ESI): *m/z* (%): 205 (100) $[M+Na]^+$; HRMS: calcd for $C_6H_{14}O_6Na$ $[M+Na]^+$: 205.0688; found: 205.0689.

To penta-acetoxy compound **S1** or **S2** (for the compound obtained pure by chiral HPLC purification with a retention time R_t = 11.1 min) (20.2 mg, 0.0515 mmol) was added 8 M anhydrous NH_3 in MeOH (1.30 mL, 87.7mg, 5.15 mmol). The resulting mixture was stirred in a sealed vial at 25 °C for 2 days. The solvent and byproduct acetamide were removed under reduced pressure to leave the pure deacetylated product **1** or **3** as a colourless oil (8.9 mg, 0.049 mmol, 95%): ^{1}H NMR (800 MHz, D_2O) δ 3.95 (dd, *J* = 7.9, 3.1 Hz, 2H), 3.89 (dd, *J* = 11.9, 3.1 Hz, 2H), 3.70 (dd, *J* = 11.9, 7.9 Hz, 2H), 3.69 (s, 2H) ppm; ^{13}C NMR (100 MHz, D_2O) δ 77.1 (C), 73.5 (CH), 62.5 (CH_2), 62.0 (CH_2) ppm; IR (thin film): ν_{max} = 3368, 2945, 1647, 1595 cm^{-1}; MS (+ve, ESI): *m/z* (%): 205 (100) $[M+Na]^+$; HRMS: calcd for $C_6H_{14}O_6Na$ $[M+Na]^+$: 205.0688; found: 205.0690.

Enantiomers 4 and 5:



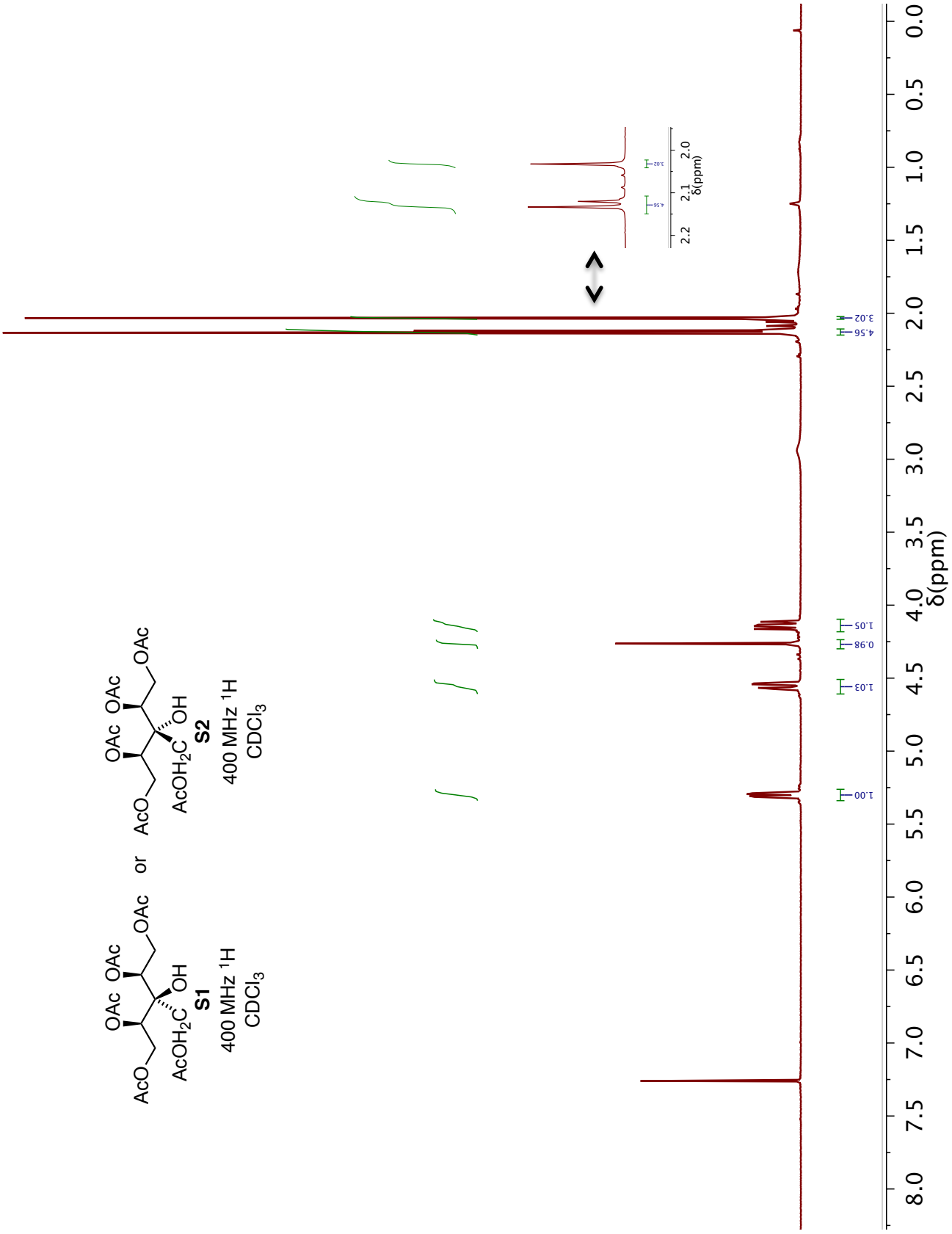
To penta-acetoxy compound **S3** or **S4 (**for the compound obtained pure by chiral HPLC purification with a retention time R_t = 6.9 min) (10.0 mg, 0.0255 mmol) was added 8 M anhydrous NH_3 in MeOH (0.64 mL, 87 mg, 5.1 mmol). The resulting mixture was stirred in a sealed vial at 25 °C for 2 days. The solvent and byproduct acetamide were removed under reduced pressure to leave the pure deacetylated product **4** or **5** as a colourless oil (4.4 mg, 0.024 mmol, 95%): ^{1}H NMR (800 MHz, D_2O) δ 3.93 (dd, J = 7.8, 3.1 Hz, 1H), 3.87 (dt, J = 8.3, 3.2 Hz, 3H), 3.77–3.70 (m, 4H) ppm; ^{13}C NMR (100 MHz, D_2O) δ 76.9 (C), 74.2 (CH), 74.2 (CH), 62.5 (CH_2), 62.5 (CH_2), 62.4 (CH_2) ppm; IR (thin film): ν_{max} = 3368, 2945, 1647, 1595 cm^{-1}; MS (+ve, ESI): *m/z* (%): 205 (100) $[M+Na]^+$; HRMS: calcd for $C_6H_{14}O_6Na$ $[M+Na]^+$: 205.0688; found: 205.0690; $[\alpha]_D$ = +290 (*c* 0.56, CH_3OH).

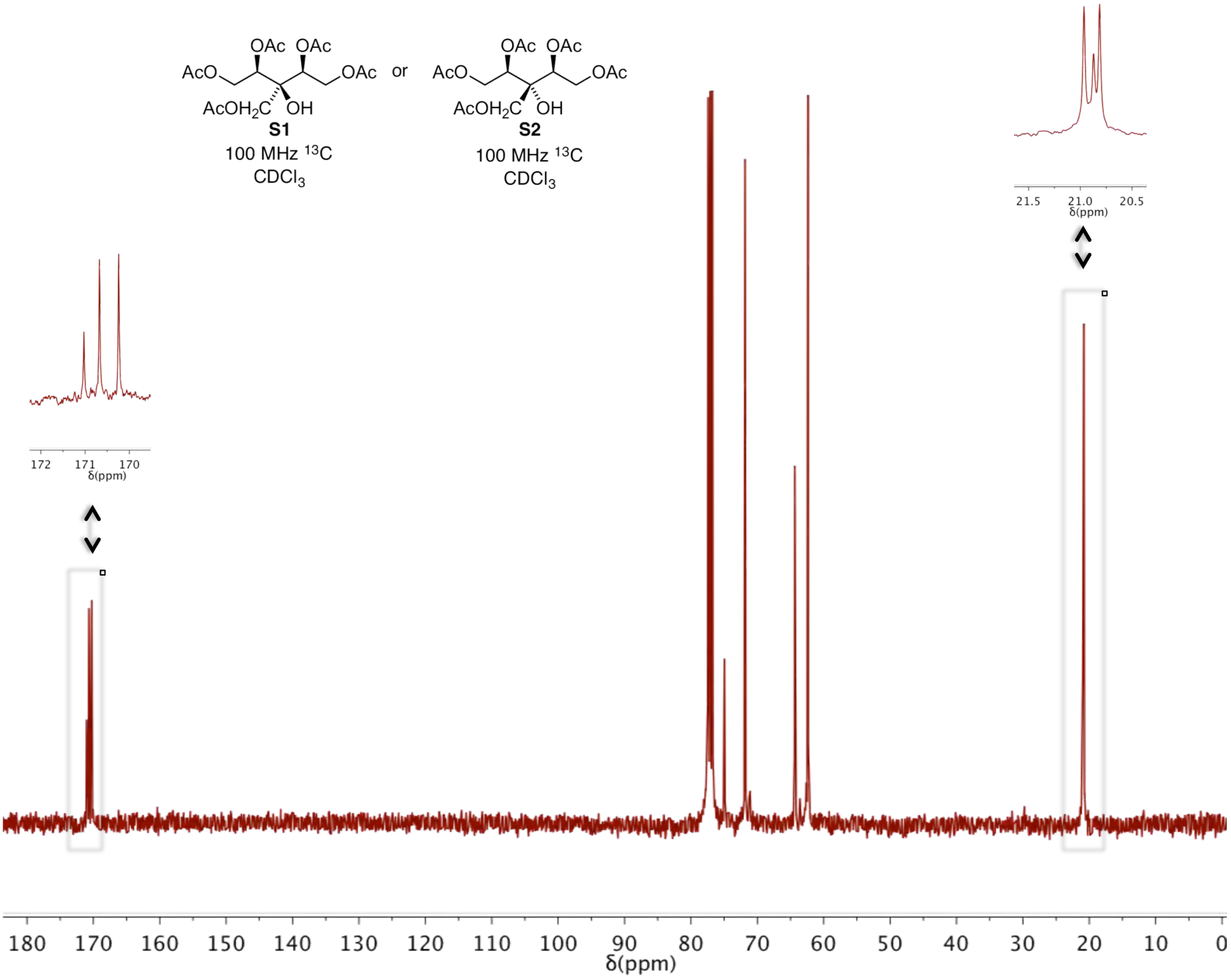
To penta-acetoxy compound **S3** or **S4** (for the compound obtained pure by normal phase HPLC purification with a retention time R_t = 8.7 min) (8.0 mg, 0.020 mmol) was added 8 M anhydrous NH_3 in MeOH (0.50 mL, 70 mg, 4.1 mmol). The resulting mixture was stirred in a sealed vial at 25 °C for 2 days. The solvent and byproduct acetamide were removed under reduced pressure to leave the pure deacetylated product **4** or **5** as a colourless oil (3.5 mg, 0.019 mmol, 95%): $[\alpha]_D$ = –300 (*c* 0.63, CH_3OH). The remaining characterization data for this sample were identical to those of the enantiomer.

1.3 References

1. Pangborn, A. B.; Giardello, M. A.; Grubbs, R. H.; Rosen, R. K.; Timmers, F. J. *Organometallics* **1996**, *15*, 1518–1520.
2. Bradford, T. A.; Payne, A. D.; Willis, A. C.; Paddon-Row, M. N.; Sherburn, M. S. J. *Org. Chem.* **2010**, *75*, 491–494.
3. Chandramohan, G.; Ignacimuthu, S.; Pugalendi, K. V. *Eur. J. Pharmacol.* **2008**, *590*, 437–443.
4. We used the Spectral Database for Organic Compounds, SDBS, a free site organized by National Institute of Advanced Industrial Science and Technology (AIST), Japan: http://sdbs.riodb.aist.go.jp/sdbs/cgi-bin/cre_index.cgi
5 Private communication with Prof. K. V. Pugalendi.

2 ^{1}H and ^{13}C NMR Spectra of Compounds (penta-acetoxy compounds S1–S4, 1 and 3–5)

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AcO
OAc
AcOH2C OH
S1
100 MHz 13C
CDCl3
or
OAc OAc
AcO
OAc
AcOH2C OH
S2
100 MHz 13C
CDCl3
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δ(ppm)
21.5 21.0 20.5
δ(ppm)
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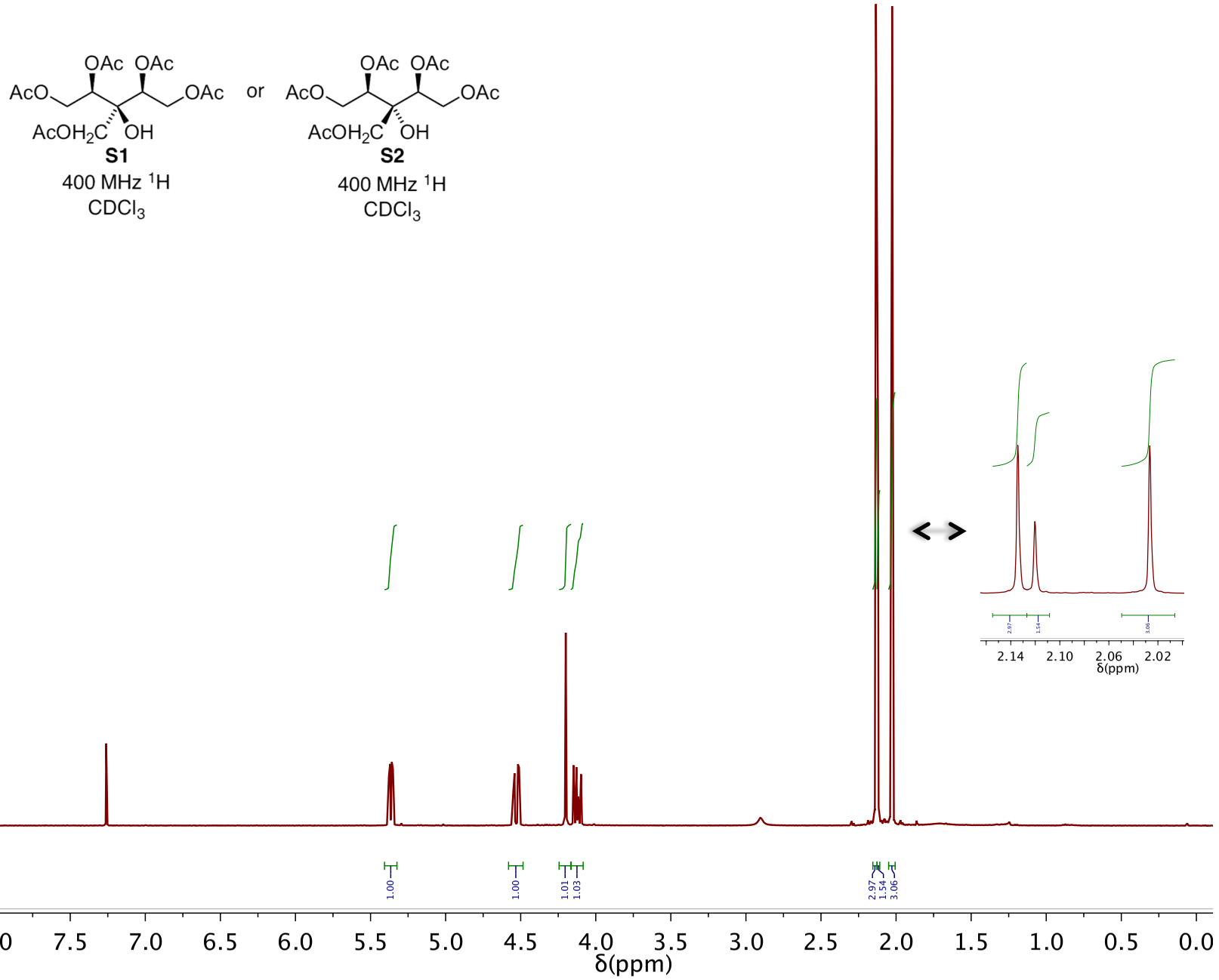
δ(ppm)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AcO
OAc
AcOH2C
OH
S1
400 MHz 1H
CDCl3
or
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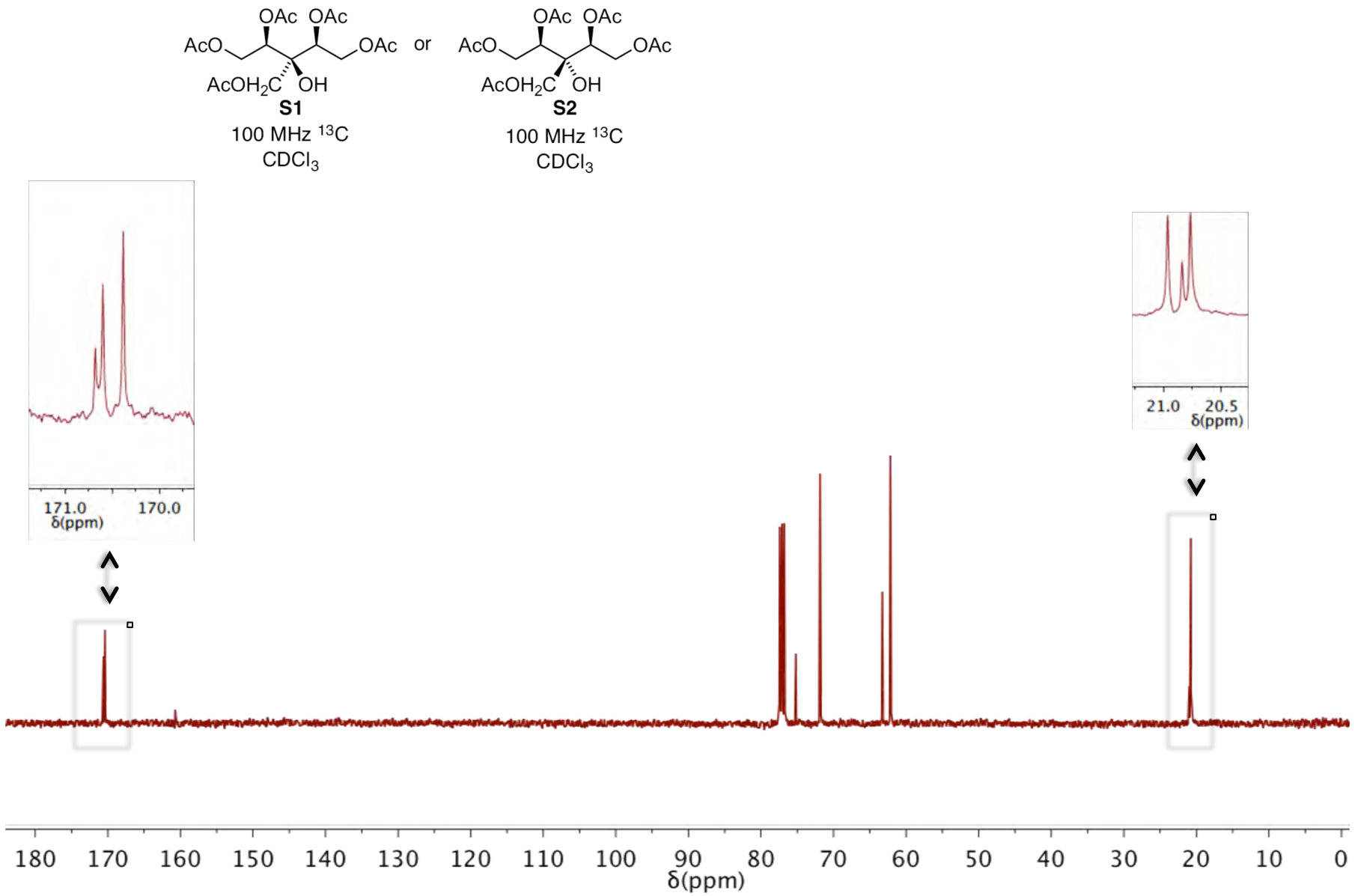
AcO
OAc
AcOH2C
OH
S2
400 MHz 1H
CDCl3
1.00
1.00
1.01
1.03
2.97
1.54
3.06
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
δ(ppm)
2.14
2.10
2.06
2.02
δ(ppm)


OAc OAc
AcO
OAc
AcOH2C
OH
S1
100 MHz 13C
CDCl3
or
OAc OAc
AcO
OAc
AcOH2C
OH
S2
100 MHz 13C
CDCl3
171.0
170.0
δ(ppm)
21.0
20.5
δ(ppm)
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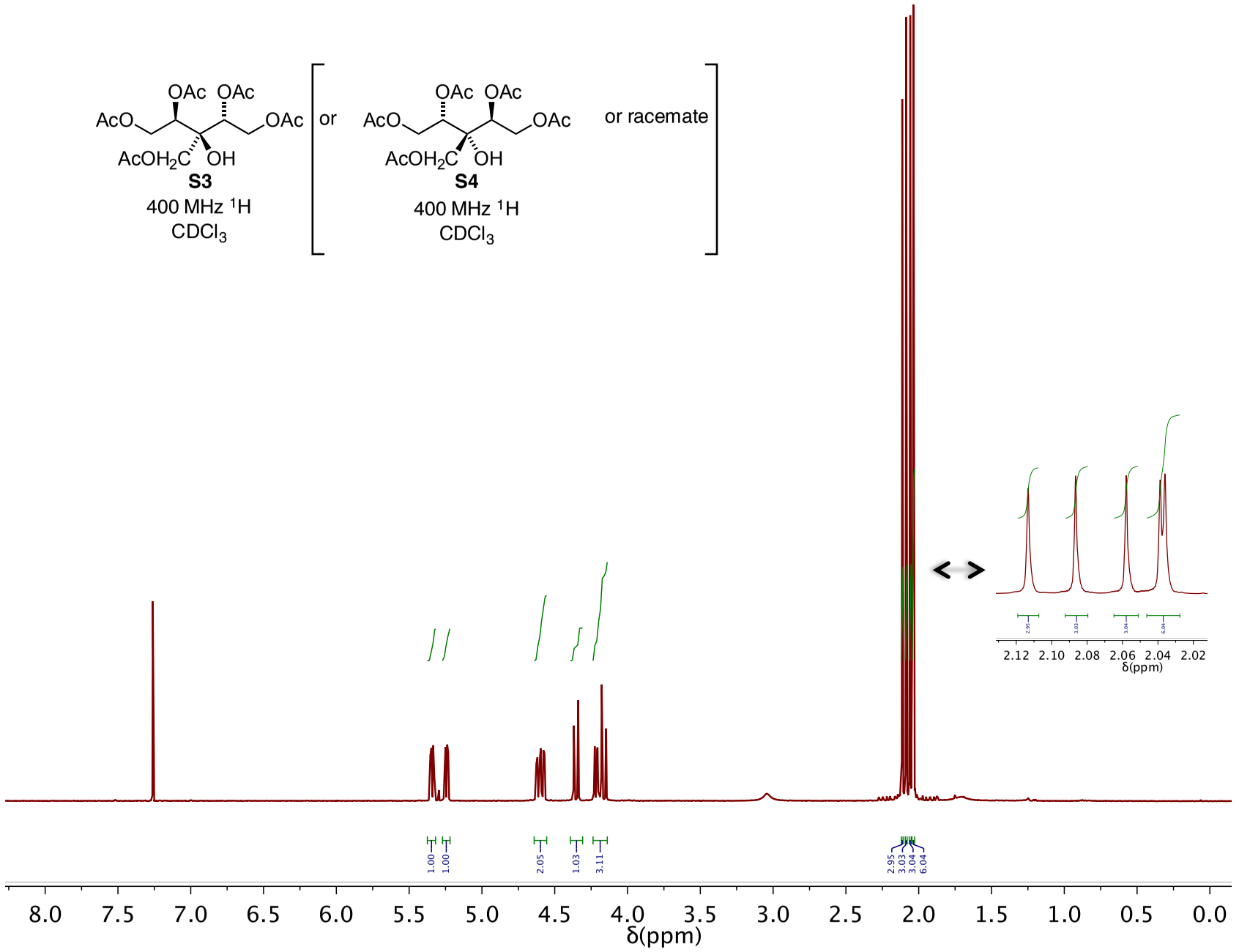
δ(ppm)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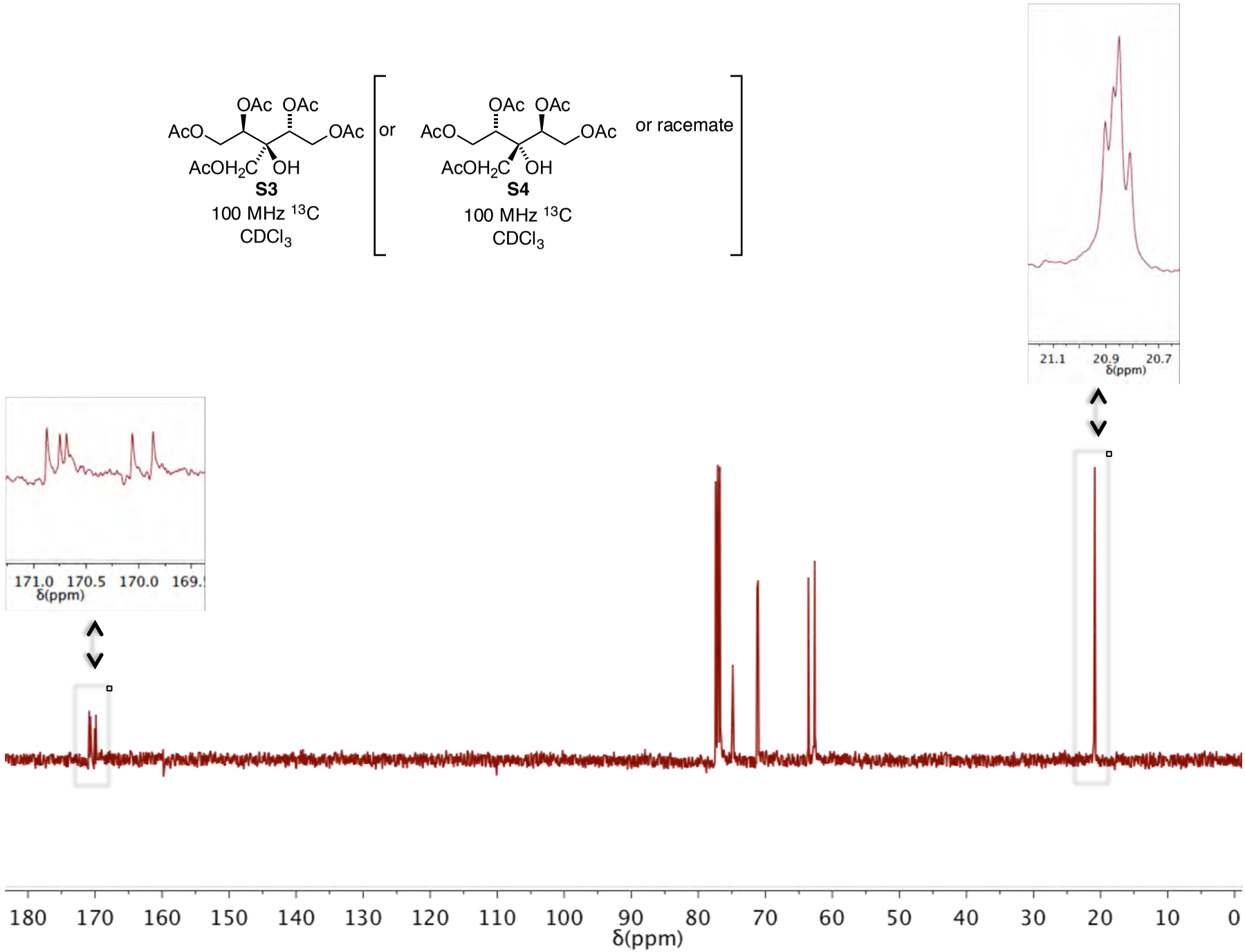
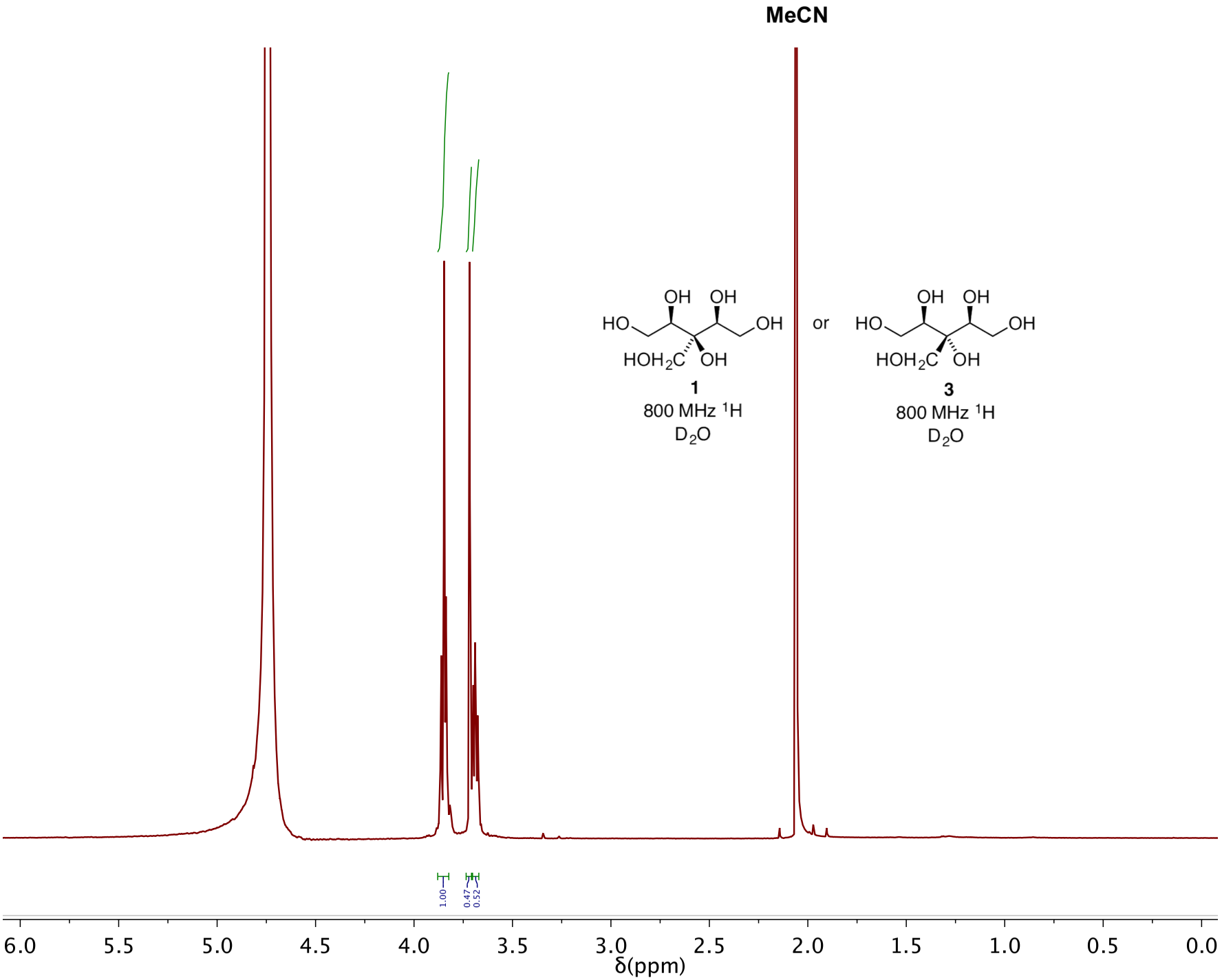
AcO
OAc
AcOH2C OH
S3
400 MHz 1H
CDCl3
or
OAc OAc
AcO
OAc
AcOH2C OH
S4
400 MHz 1H
CDCl3
or racemate
2.12 2.10 2.08 2.06 2.04 2.02
δ(ppm)
1.00 1.00 2.05 1.03 3.11 2.95 3.03 3.04 6.04
8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
δ(ppm)


OAc OAc
AcO
OAc
AcOH2C OH
S3
100 MHz 13C
CDCl3
or
OAc OAc
AcO
OAc
AcOH2C OH
S4
100 MHz 13C
CDCl3
or racemate
21.1 20.9 20.7
δ(ppm)
171.0 170.5 170.0
δ(ppm)
180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
δ(ppm)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OH
HO
OH
HOH2C
OH
1
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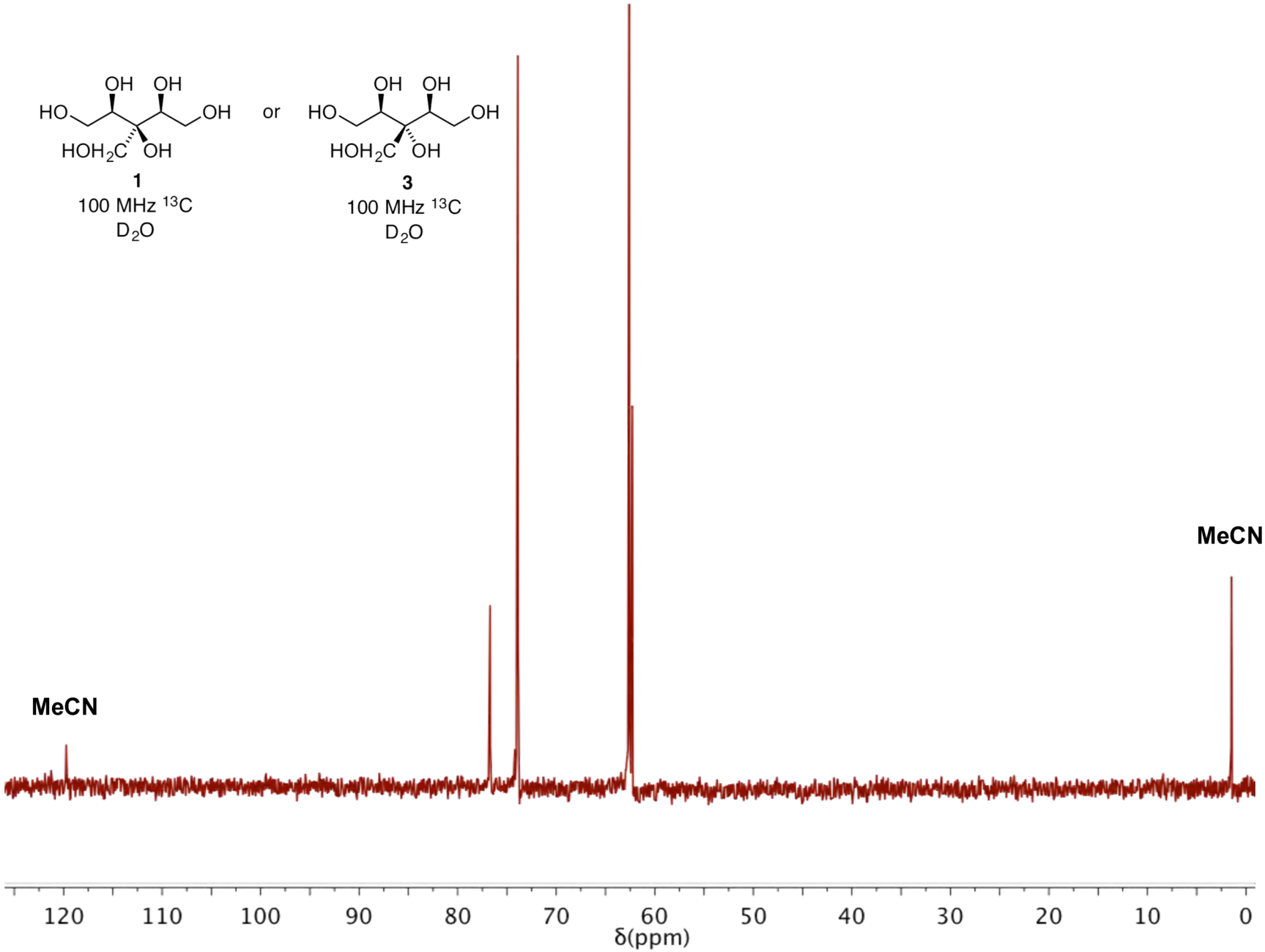
D2O
or
OH
OH
HO
OH
HOH2C
OH
3
800 MHz 1H
D2O
1.00
0.47
0.52
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
δ(ppm)


OH OH
HO OH
HOH2C OH
1
100 MHz 13C
D2O
or
OH OH
HO OH
HOH2C OH
3
100 MHz 13C
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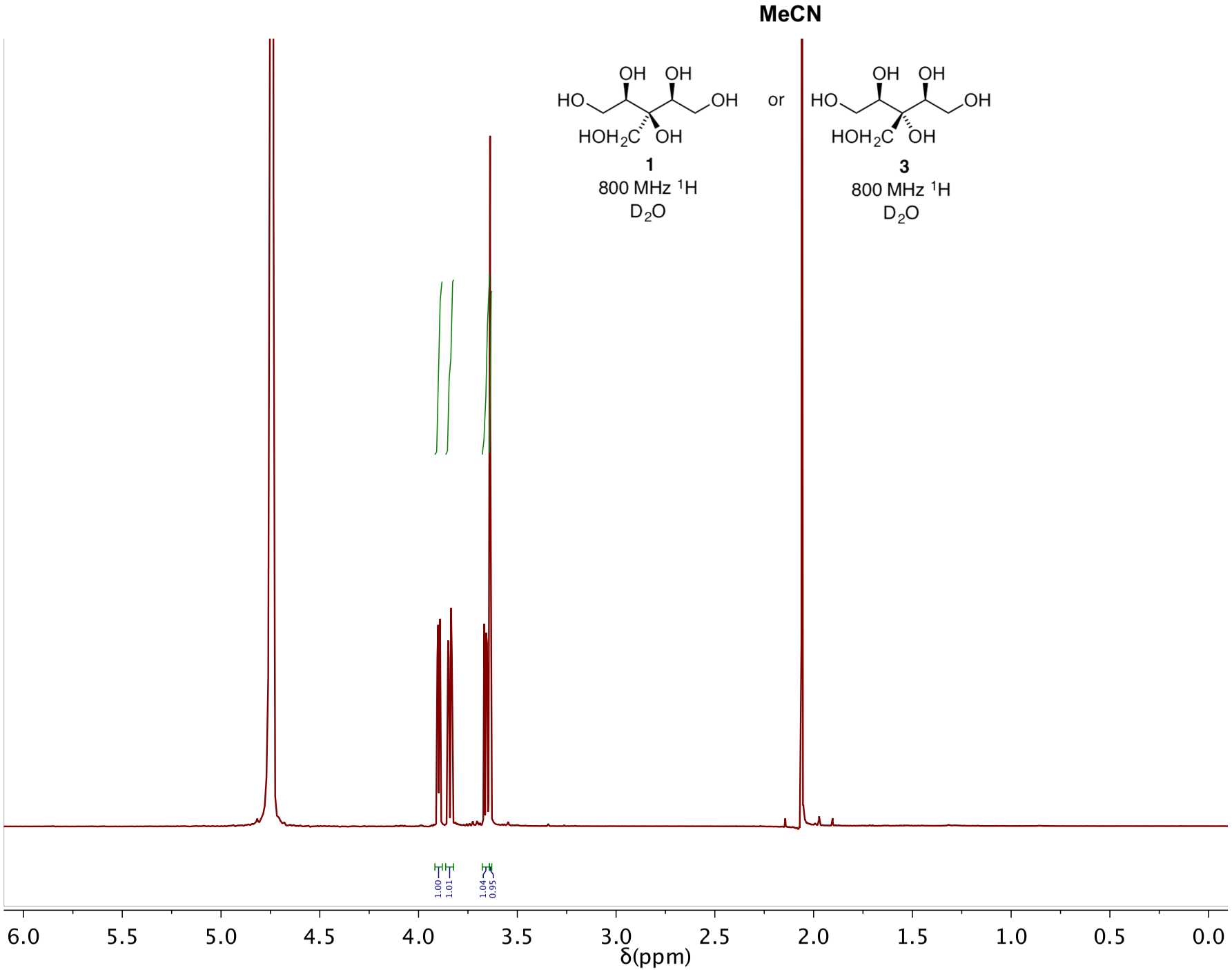
MeCN
MeCN
120 110 100 90 80 70 60 50 40 30 20 10 0
δ(ppm)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HO OH
HOH2C OH
1
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D2O
or
OH OH
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HOH2C OH
3
800 MHz 1H
D2O
1.00
1.01
1.04
0.95
6.0
5.5
5.0
4.5
4.0
3.5
3.0
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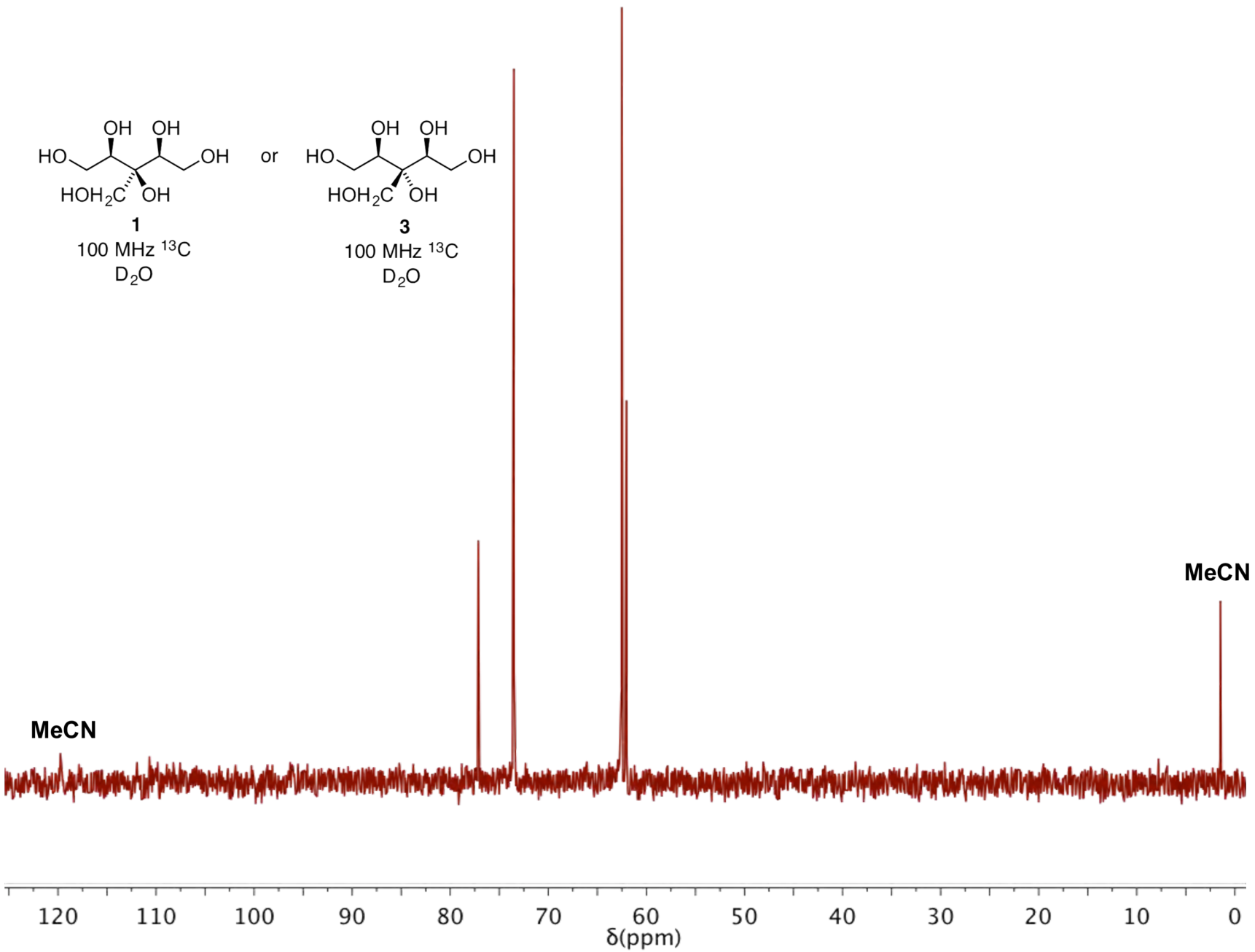
2.0
1.5
1.0
0.5
0.0
δ(ppm)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HO OH
HOH2C OH
1
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D2O
or
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HO OH
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3
100 MHz 13C
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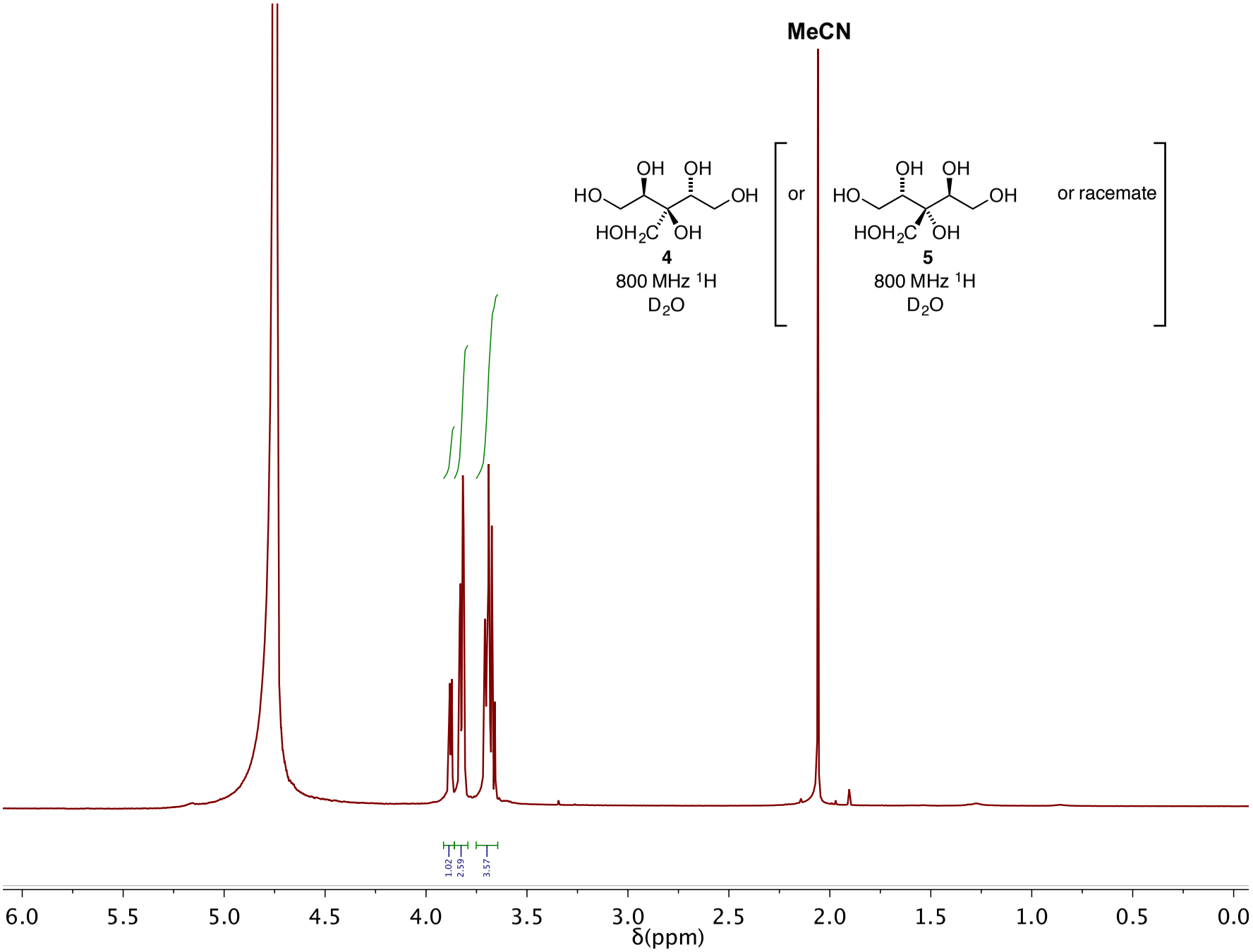
MeCN
MeCN
120 110 100 90 80 70 60 50 40 30 20 10 0
δ(ppm)


MeCN
OH
OH
HO
OH
HOH2C
OH
4
800 MHz 1H
D2O
or
OH
OH
HO
OH
HOH2C
OH
5
800 MHz 1H
D2O
or racemate
1.02
2.59
3.57
6.0
5.5
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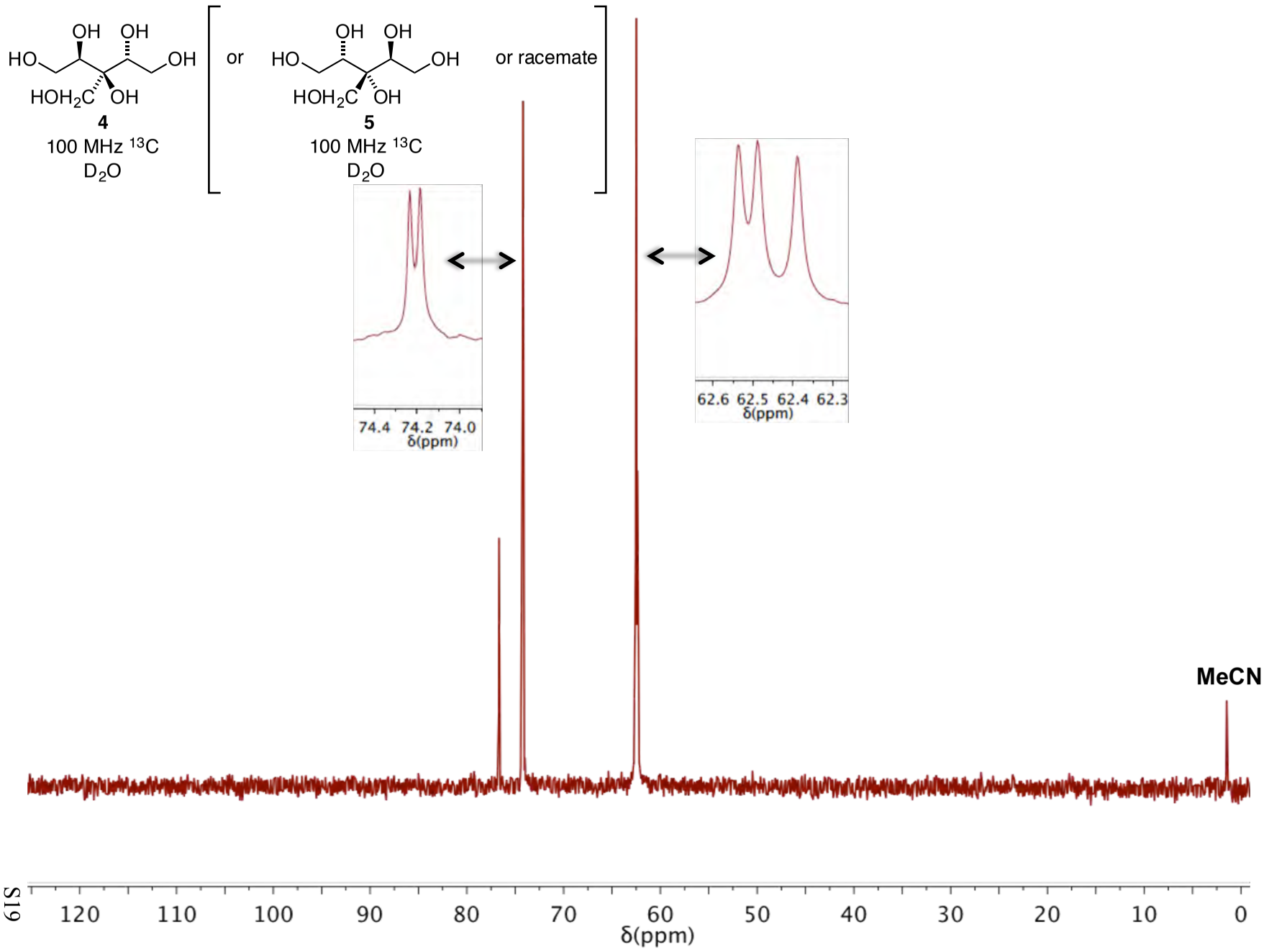
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
δ(ppm)


OH
OH
HO
OH
HOH2C
OH
4
100 MHz 13C
D2O
or
OH
OH
HO
OH
HOH2C
OH
5
100 MHz 13C
D2O
or racemate
74.4 74.2 74.0
δ(ppm)
62.6 62.5 62.4 62.3
δ(ppm)
MeCN
120 110 100 90 80 70 60 50 40 30 20 10 0
δ(ppm)

3 HPLC separations and isomer ratio calculations.

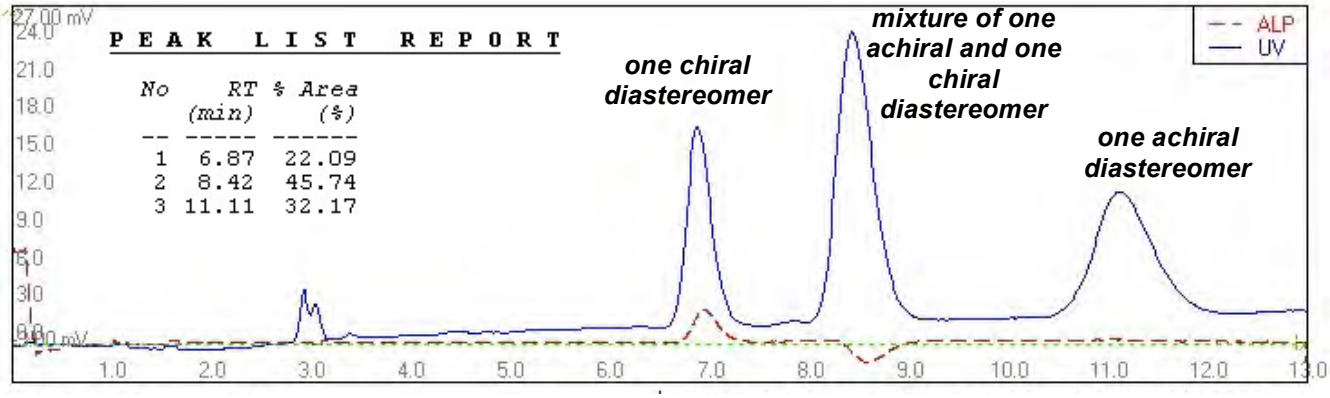
Stereoisomers were separated by HPLC and the stereoisomer ratio was estimated through ^{1}H NMR analysis of the crude product mixture from the dihydroxylation reaction. This ratio changed slightly upon acetylation and purification.

The crude mixture of penta-acetates was purified firstly on normal phase chiral HPLC (eluting with 20:80 isopropanol:hexane on an AS-H semi-preparative column, 4 mL/min) to give three fractions: R_t = 6.9 min (one pure chiral enantiomer); R_t = 8.4 min (mixture of the other chiral enantiomer and one of the two *meso*-compounds); and R_t = 11.1 min (one pure *meso*-compound). The mixed fraction from the first, chiral HPLC separation was separated on normal phase HPLC (eluting with 5:95 isopropanol: hexane on a Phenomenex Luna 5 μm, 150 mm × 21.2 mm silica column, 20 mL/min) to give two fractions: R_t = 8.7 min (the other pure chiral enantiomer) and R_t = 10.5 min (the other pure *meso*-compound). The following traces are included:

3.1 Chiral HPLC trace for the penta-acetoxy crude mixture (UV and ALP detection methods)

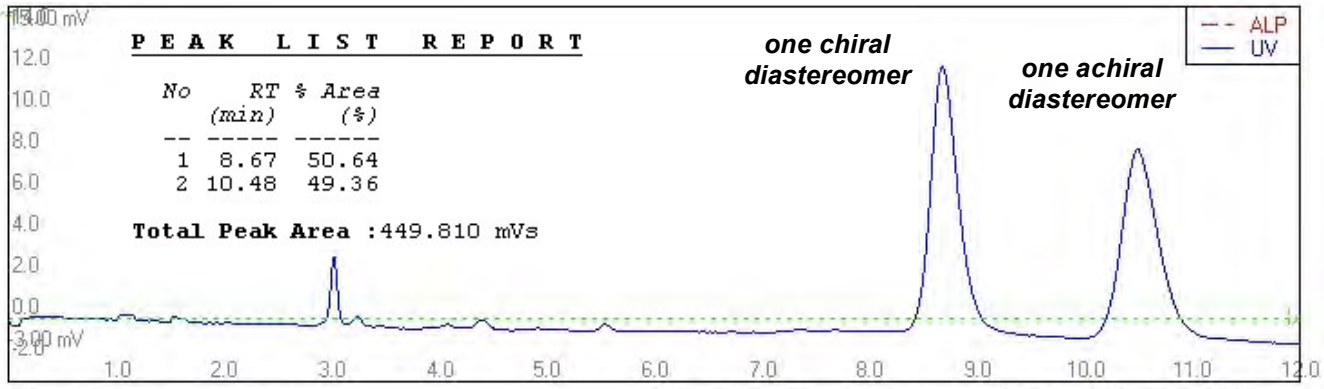
3.2 Analytical achiral HPLC trace for the mixed fraction of penta-acetoxy compounds (UV detection method)

3.1 Chiral HPLC trace for the penta-acetoxy crude mixture (UV and ALP detection methods)



The mixed fraction, comprising one chiral diastereomer and one achiral diastereomer, was re-columned on achiral HPLC (see **section 3.2**)

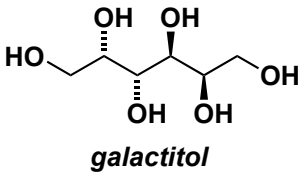
3.2 Achiral HPLC trace for the mixed fraction of penta-acetoxy compounds (UV detection method)



4 Experimental data from the published paper by Pugalendi et al[3] and comparison with data for galactitol

^{1}H NMR (400 MHz, D_2O): δ 3.8 (t, 2H, CHOH, J =6.32 Hz), 3.5 (d, –CH_2–CHOH, J=6.78 Hz) ppm; ^{13}C NMR (400 MHz, D_2O): δ 70.8 (CHOH), 70 (CHOH), 63 (CH_2OH) ppm; IR (thin film): ν_{max} = 3369, 2942, 1455 cm^{-1}; MS: m/z=182 [M]+, 183 [M+1], 133, 115, 103, 85.

A comparison of ^{1}H NMR and ^{13}C NMR spectroscopic data of (a) the natural product from Pugalendi et al;[3] (b) galactitol from the Spectral Database for Organic Compounds, SDBS database;[4] and (c) our own measurements on galactitol (≥99% purity galactitol supplied by Sigma-Aldrich).



galactitol

The three sets of ^{1}H and ^{13}C NMR spectra (all in D_2O at the same field strength) bear a strikingly close resemblance. The only discrepancy is in the chemical shifts and these are explained by different reference methods. ^{1}H and ^{13}C NMR spectra both (a) provided both by Prof K. V. Pugalendi[1] and (b) obtained from the SDBS database[2] do not specify an internal standard. We use acetonitrile as the internal reference in our spectra (δ 2.06 ppm for ^{1}H NMR spectra and δ 1.47 and 119.68 ppm for ^{13}C NMR spectra).

^{1}H NMR data (all 400 MHz, D_2O)

	(a) Data quoted in Pugalendi paper[3] (correction factor in parentheses)	(b) Spectral Database for Organic Compounds, SDBS[4]	(c) THIS WORK (referenced to MeCN, see above)
signal 1	**3.8 ppm (+ 0.17 ppm)**	**3.98 ppm**	**3.97 ppm**
signal 2	**3.5 ppm (+ 0.19 ppm)**	**3.70 ppm**	**3.69 ppm**
signal 3		**3.68 ppm**	**3.67 ppm**

In the ^{1}H NMR spectra, our data for galactitol and those in the SDBS database are essentially identical. The chemical shift differences in Prof K. V. Pugalendi's spectrum (+0.18 ppm on average) are presumably due to a different referencing method. The interpretation of the peak at δ 3.5 ppm as a doublet is in error and the 2:3 integration ratio for these signals is presumably the result of a short relaxation delay.

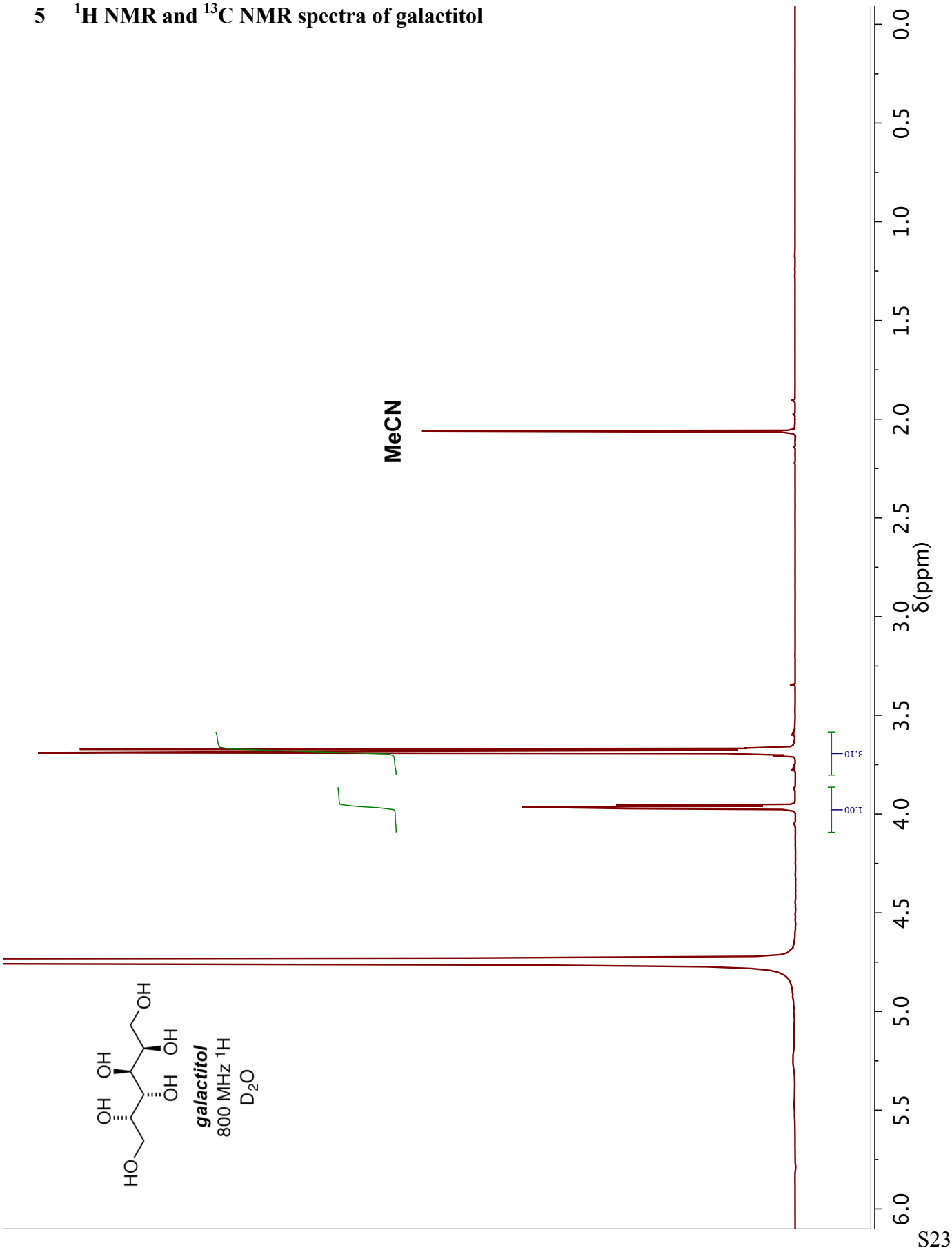
^{13}C NMR data (all 100 MHz, D_2O)

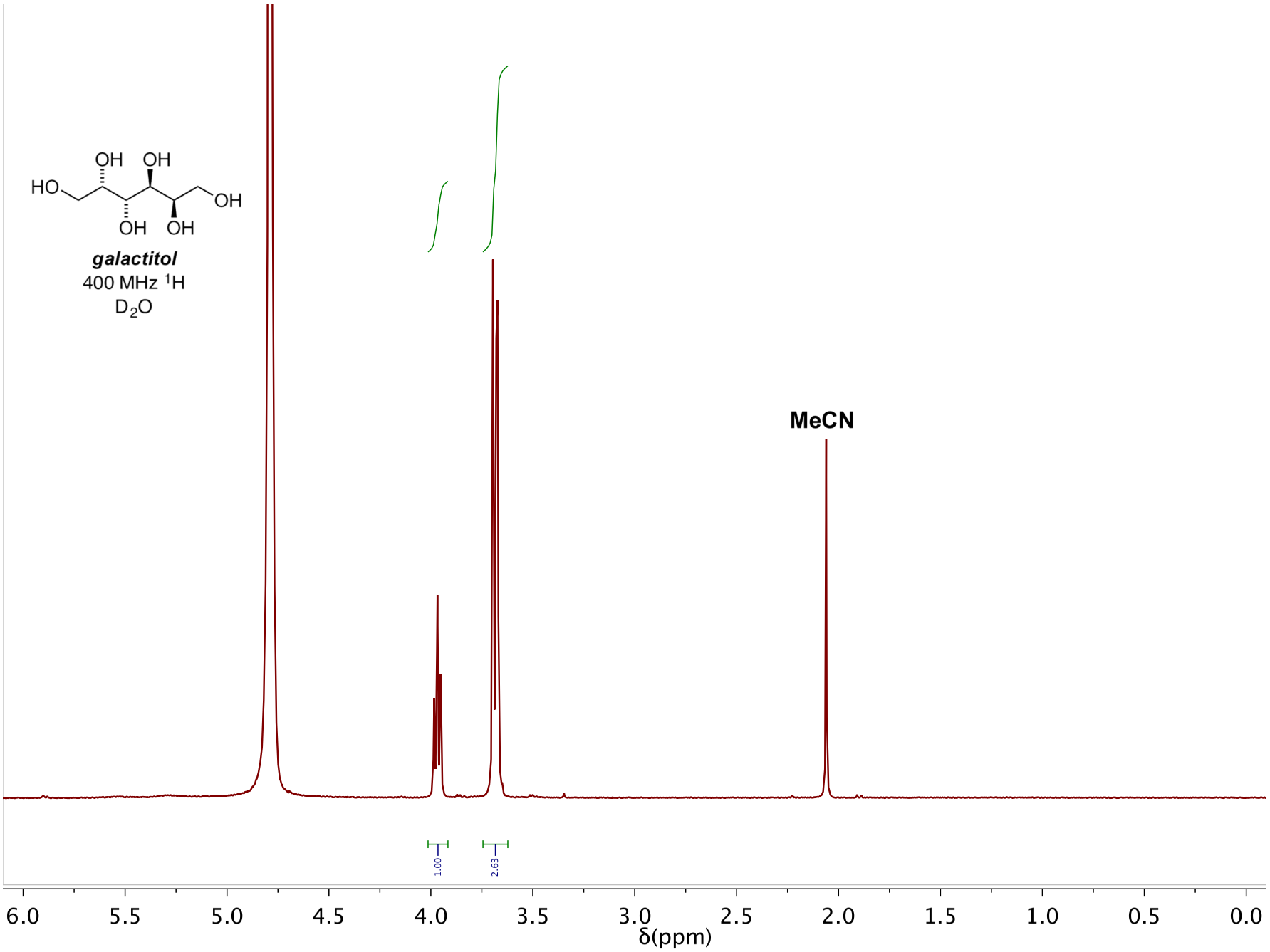
	(a) Data quoted in Pugalendi paper[3]	(b) Spectral Database for Organic Compounds, SDBS[4] (correction factor)	(c) THIS WORK (referenced to MeCN, see above)
signal 1	**70.8 ppm**	**71.35 ppm (+ 0.57 ppm)**	**70.78 ppm**
signal 2	**70 ppm**	**70.59 ppm (– 0.60 ppm)**	**69.99 ppm**
signal 3	**63 ppm**	**64.36 ppm (– 0.50 ppm)**	**63.86 ppm**

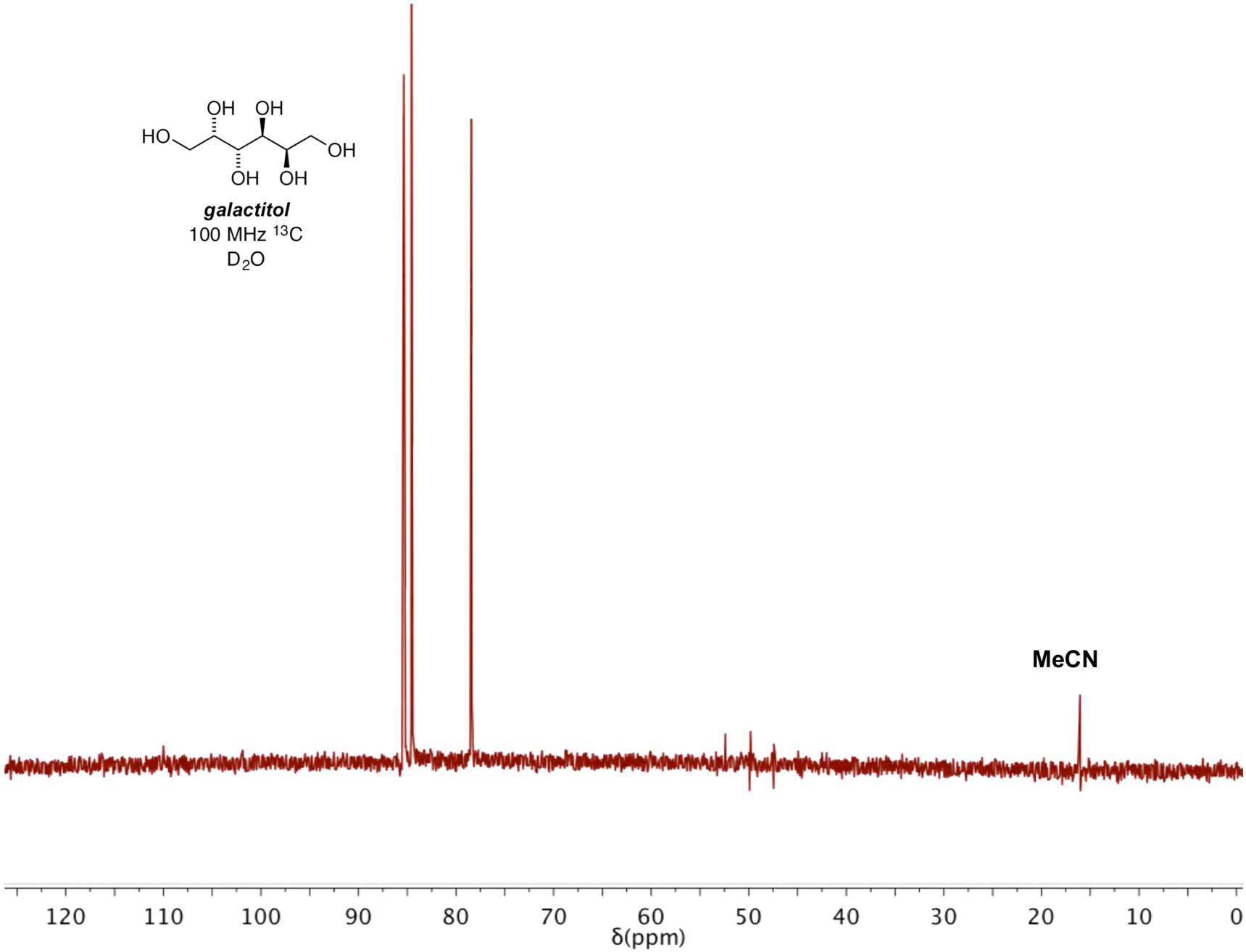
In the ^{13}C NMR spectra, Prof K. V. Pugalendi's spectrum matches that of ours very closely. The chemical shift differences with the SDBS database spectrum (+0.56 ppm on average) is presumably due to a different referencing method.

We conclude the three ^{1}H NMR spectra and the three ^{13}C NMR spectra are of the same compound, galactitol.

5 ^{1}H NMR and ^{13}C NMR spectra of galactitol




OH
OH
HO
OH
OH
OH
galactitol
400 MHz 1H
D2O
MeCN
1.00
2.63
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
δ(ppm)


OH
OH
HO
OH
OH
OH
galactitol
100 MHz 13C
D2O
MeCN
120
110
100
90
80
70
60
50
40
30
20
10
0
δ(ppm)