Electronic Supplementary Information

Synthesis of apiose-containing oligosaccharide fragments of the plant cell wall: fragments of rhamnogalacturonan-II side chains A and B, and apiogalacturonan

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¹H NMR (400 MHz) and ¹³C NMR (100 MHz) data for protected saccharide derivatives and oligosaccharides 1–4 (in D₂O)

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Crystal structure analysis of Methyl C-(4-O-acetyl-2,3-O-carbonyl-β-L-rhamnopyranosyl-oxymethyl)-(1→3’)-(2,3-O-(R)-benzylidene-β-D-erythrofuranosyl)-(1→2)-(methyl (2-O-acetyl-3,4-O-isopropylidene-α-D-galactopyranosid)uronate) (41)

Crystal data:  C_{32}H_{40}O_{17}, CH_{2}Cl, M = 781.6.  Orthorhombic, space group P2_{1}2_{1}2_{1} (no. 19), a = 11.2799(6), b = 15.1788(9), c = 21.4815(13) Å, V = 3678.0(4) Å³. Z = 4, Dc = 1.411 g cm⁻³, F(000) = 1640, T = 140(1) K, μ(Mo-Kα) = 2.5 cm⁻¹, λ(Mo-Kα) = 0.71069 Å.

Crystals are beautiful, colourless prisms. One, ca 0.52 x 0.25 x 0.24 mm, was mounted in oil on a glass fibre and fixed in the cold nitrogen stream on an Oxford Diffraction Xcalibur-3 CCD diffractometer equipped with Mo-Kα radiation and graphite monochromator. Intensity data were measured by thin-slice ω- and φ-scans. Total no. of reflections recorded, to θ_{max} = 27.5°, was 49119 of which 8410 were unique (Rint = 0.045); 7002 were 'observed' with I > 2σ(I).

Data were processed using the CrysAlis-CCD and -RED (1) programs. The structure was determined by the direct methods routines in the SHELXS program (2A) and refined by full-matrix least-squares methods, on F²'s, in SHELXL (2B). The non-hydrogen atoms were refined with anisotropic thermal parameters. Hydrogen atoms were included in idealised positions and their Uiso values were set to ride on the Ueq values of the parent carbon atoms. At the conclusion of the refinement, wR₂ = 0.093 and R₁ = 0.049 (2B) for all 8410 reflections weighted w = [σ²(F₀²) + (0.0556P)²]¹/² with P = (F₀² + 2F_c²)/3; for the 'observed' data only, R₁ = 0.037.

In the final difference map, the highest peak (ca 0.7 eÅ⁻³) was close to Cl(2).

Scattering factors for neutral atoms were taken from reference (3). Computer programs used in this analysis have been noted above, and were run through WinGX (4) on a Dell Precision 370 PC at the University of East Anglia.

References
2.  G. M. Sheldrick, SHELX-97 - Programs for crystal structure determination (SHELXS) and refinement (SHELXL), Acta Crystallogr., (2008), A64, 112-122.
Fig. 1 X-Ray crystal structure of disaccharide derivative 41.
$^1$H NMR (400 MHz) and $^{13}$C NMR (100 MHz) data for protected saccharide derivatives and oligosaccharides 1–4

Compound 8

$\text{CDCl}_3$
Compound 9a
Compound 9b

CDCl₃
Compound 12
Compound 13

CDCl₃
Compound 14

CDCl₃
Compound 16

CDCl₃

Electronic Supplementary Material (ESI) for Organic and Biomolecular Chemistry
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Compound 17
Compound 24

[Chemical structure]

CDCl₃
Compound 25

Electronic Supplementary Material (ESI) for Organic and Biomolecular Chemistry
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Compound 27

Electronic Supplementary Material (ESI) for Organic and Biomolecular Chemistry
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Compound 28

CDCl$_3$
Compound 29

Electronic Supplementary Material (ESI) for Organic and Biomolecular Chemistry
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Compound 30

CDCl₃

- S21 -
Compound 31

[Chemical structure image]

CDCl₃
Compound 33

CDCl₃
Compound 34

[Chemical structure image with CDCl₃ notation]
Compound 35
Compound 36b

CDCl₃
Compound 37a,b
Compound 38a
Compound 39
Compound 40

![NMR Spectrum of Compound 40 in CDCl₃](image-url)

- S33 -
Compound 41

CDCl$_3$
Compound 42

D$_2$O

Electronic Supplementary Material (ESI) for Organic and Biomolecular Chemistry
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Compound 1

$\text{D}_2\text{O} \ (1\text{D NOESY presaturation})$
Compound 2
Compound 3

D$_2$O (1D NOESY presaturation)
Compound 4

D$_2$O (1D NOESY presaturation)