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4-Nitrophenyl α-L-rhamnopyranoside hemihydrate

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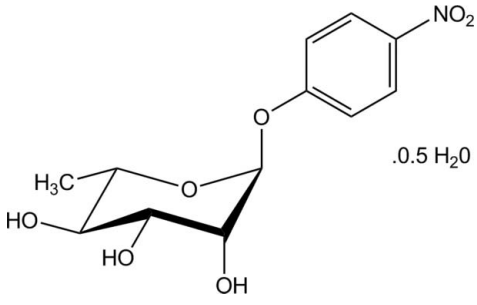
Key indicators: single-crystal X-ray study; T = 100 K; mean σ(C–C) = 0.003 Å; R factor = 0.032; wR factor = 0.081; data-to-parameter ratio = 8.4.

In the title compound, $C_{12}H_{15}NO_7\cdot0.5H_2O$, there are two independent molecules in the asymmetric unit, together with one water molecule. The pyranoside rings each have close to a $^1C_4$ chair conformation and the nitro groups are almost coplanar with the benzene rings. The water molecule links the two independent molecules through O—H···O hydrogen bonds. All the hydroxyl groups are involved in hydrogen-bond interactions, giving rise to a three-dimensional network.

Related literature

For a related structure, see Fernandez-Castaño & Foces-Foces (1996). For related literature, see: Garegg *et al.* (1978).



Experimental

Crystal data

$C_{12}H_{15}NO_7\cdot0.5H_2O$
M_r = 294.26
Monoclinic, $P2_1$
a = 10.5371 (15) Å
b = 6.8681 (8) Å
c = 19.135 (3) Å
β = 101.543 (7)°
V = 1356.8 (3) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.12 mm^{-1}
T = 100 (2) K
0.6 × 0.58 × 0.23 mm

Data collection

Bruker APEXII diffractometer
Absorption correction: multi-scan (*SADABS*; Sheldrick, 2002)
T_{min} = 0.923, T_{max} = 0.972
14281 measured reflections
3340 independent reflections
3147 reflections with $I > 2\sigma(I)$
R_{int} = 0.051

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.032
$wR(F^2)$ = 0.080
S = 1.04
3340 reflections
396 parameters
1 restraint
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.38 e Å^{-3}
$\Delta\rho_{min}$ = −0.24 e Å^{-3}

Table 1
Hydrogen-bond geometry (Å, °).

D—H···A	D—H	H···A	D···A	D—H···A
O1—H1A···O202^{i}	0.86 (3)	1.97 (7)	2.803 (7)	159 (9)
O1—H1B···O102^{ii}	0.84 (8)	2.06 (1)	2.881 (8)	162 (7)
O102—H12···O104^{iii}	0.84 (5)	1.87 (3)	2.695 (6)	163 (8)
O103—H13···O203^{iv}	0.83 (0)	2.12 (3)	2.934 (2)	165 (6)
O104—H14···O203^{iv}	0.80 (8)	1.89 (5)	2.663 (7)	158 (5)
O202—H22···O1^{iv}	0.88 (0)	1.82 (0)	2.695 (8)	173 (0)
O203—H23···O204^{v}	0.83 (7)	1.80 (6)	2.641 (8)	176 (8)
O204—H24···O103^{vi}	0.79 (7)	1.99 (1)	2.776 (5)	168 (4)

Symmetry codes: (i) $x+1, y-1, z$; (ii) $-x+1, y-\frac{3}{2}, -z+1$; (iii) $x, y+1, z$; (iv) $-x, y+\frac{1}{2}, -z+1$; (v) $-x, y-\frac{1}{2}, -z+1$; (vi) $x, y-1, z$.

Data collection: *SMART* (Bruker, 2006); cell refinement: *SAINT* (Bruker, 2006); data reduction: *SAINT*; program(s) used to solve structure: *SIR97* (Altomare *et al.*, 1995); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *ORTEP-3 for Windows* (Farrugia, 1997); software used to prepare material for publication: *WinGX* (Farrugia, 1999).

PP is grateful to the Région Bretagne for a grant.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: BV2086).

References

Altomare, A., Burla, M. C., Cascarano, G., Giacovazzo, C., Guagliardi, A., Moliterni, A. G. G. & Polidori, G. (1995). *J. Appl. Cryst.* **28**, 842–846.
Bruker (2006). *SMART*, *SAINT* and *SADABS*. Bruker AXS Inc., Madison, Wisconsin, USA.
Farrugia, L. J. (1997). *J. Appl. Cryst.* **30**, 565.
Farrugia, L. J. (1999). *J. Appl. Cryst.* **32**, 837–838.
Fernandez-Castaño, C. & Foces-Foces, C. (1996). *Acta Cryst.* C**52**, 1586–1588.
Garegg, P. J., Hultberg, C. & Iversen, T. (1978). *Carbohydr. Res.* **62**, 173–174.
Sheldrick, G. M. (1997). *SHELXL97*. University of Göttingen, Germany.
Sheldrick, G. M. (2002). *SADABS*. Bruker AXS Inc., Madison, Wisconsin, USA.

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supplementary materials

Acta Cryst. (2008). E**64**, o379 [**doi:10.1107/S1600536807068195**]

4-Nitrophenyl α-L-rhamnopyranoside hemihydrate

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Comment

α-*L*-Rhamnosidases (E.C. 3.2.1.40) catalyze the hydrolysis of *L*-rhamnose from polysaccharides and glycosides. During *in vitro* studies, the activity of these enzymes is determined most of the time using the title compound, (I), following the amount of *p*-nitrophenolate ions released by monitoring the emmision at 400 nm.

The stucture of (I) is shown in Fig. 1. The molecular packing (Fig. 2) is stabilized by hydrogen bonds between all the hydroxyl groups and the water molecules.

Experimental

The title compound is commercialy available (Sigma Chemical Company) or can be easily prepared following the described procedure of Garegg *et al.* (1978). The title compound, (I), was crystallized from methanol by slow evaporation of the solvent.

Refinement

In the absence of significant anomalous scattering effects, Friedel pairs were merged. The positional and displacement parameters for the H atoms bound to O were refined. The methyl H atoms were constrained to an ideal geometry, with C—H = 0.98Å and U_{iso}(H) = 1.5U_{eq}(C), but each group was allowed to rotate freely about its C—C bond. All other H atoms were placed in calculated positions (C—H = 0.95–1.00 Å), with U_{iso}(H) = 1.2U_{eq}(C).

Figures

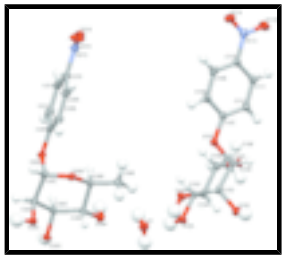


Fig. 1. The molecular structure and atom-labelling scheme for (I). Displacement ellipsoids are drawn at the 50% probability level.

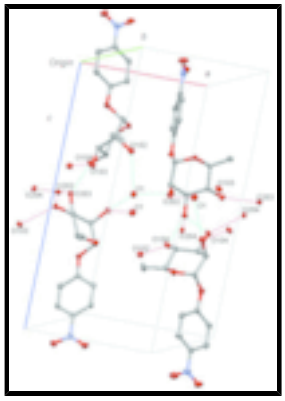


Fig. 2. The crystal packing of the structure, showing the network of hydrogen bonds (dotted lines).

4-Nitrophenyl α-*L*-rhamnopyranoside hemihydrate

Crystal data

$C_{12}H_{15}NO_7 \cdot 0.5H_2O$	$F_{000} = 620$
$M_r = 294.26$	$D_x = 1.441$ Mg m^{-3}
Monoclinic, $P2_1$	Mo $K\alpha$ radiation $\lambda = 0.71073$ Å
Hall symbol: P 2yb	Cell parameters from 8274 reflections
$a = 10.5371$ (15) Å	$\theta = 2.4$–27.4º
$b = 6.8681$ (8) Å	$\mu = 0.12$ mm^{-1}
$c = 19.135$ (3) Å	$T = 100$ (2) K
$\beta = 101.543$ (7)º	Plate, colourless
$V = 1356.8$ (3) Å^3	0.6 × 0.58 × 0.23 mm
$Z = 4$	

Data collection

Bruker APEXII diffractometer	3147 reflections with $I > 2\sigma(I)$
Monochromator: graphite	$R_{int} = 0.051$
$T = 100$(2) K	$\theta_{max} = 27.4$º
CCD rotation images, thin slices scans	$\theta_{min} = 3.5$º
Absorption correction: multi-scan (SADABS; Sheldrick, 2002)	$h = -13 \rightarrow 12$
$T_{min} = 0.923$, $T_{max} = 0.972$	$k = -8 \rightarrow 8$
14281 measured reflections	$l = -24 \rightarrow 21$
3340 independent reflections	

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)] = 0.032$	H atoms treated by a mixture of independent and constrained refinement

$wR(F^2) = 0.080$	$w = 1/[\sigma^2(F_o^2) + (0.0398P)^2 + 0.2743P]$ where $P = (F_o^2 + 2F_c^2)/3$
$S = 1.04$	$(\Delta/\sigma)_{max} = 0.001$
3340 reflections	$\Delta\rho_{max} = 0.38$ e Å^{-3}
396 parameters	$\Delta\rho_{min} = -0.24$ e Å^{-3}
1 restraint	Extinction correction: none
Primary atom site location: structure-invariant direct methods	

Special details

Geometry. All e.s.d.'s (except the e.s.d. in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell e.s.d.'s are taken into account individually in the estimation of e.s.d.'s in distances, angles and torsion angles; correlations between e.s.d.'s in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell e.s.d.'s is used for estimating e.s.d.'s involving l.s. planes.

Refinement. Refinement of F^2 against ALL reflections. The weighted *R*-factor *wR* and goodness of fit *S* are based on F^2, conventional *R*-factors *R* are based on *F*, with *F* set to zero for negative F^2. The threshold expression of $F^2 > \sigma(F^2)$ is used only for calculating *R*-factors(gt) *etc*. and is not relevant to the choice of reflections for refinement. *R*-factors based on F^2 are statistically about twice as large as those based on *F*, and *R*- factors based on ALL data will be even larger.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å^2)

	x	*y*	*z*	U_{iso}*/U_{eq}
N113	0.06897 (18)	1.3356 (3)	−0.12305 (9)	0.0208 (4)
O114	−0.04061 (15)	1.3222 (3)	−0.16072 (8)	0.0285 (4)
O115	0.16860 (16)	1.3419 (3)	−0.14777 (8)	0.0320 (4)
C110	0.0816 (2)	1.3456 (3)	−0.04511 (10)	0.0169 (4)
C109	−0.0304 (2)	1.3374 (3)	−0.01675 (11)	0.0181 (4)
H109	−0.1132	1.326	−0.0471	0.022*
C108	−0.01818 (19)	1.3462 (3)	0.05646 (11)	0.0176 (4)
H108	−0.0933	1.3403	0.0768	0.021*
C107	0.10436 (19)	1.3638 (3)	0.10079 (10)	0.0154 (4)
C112	0.21624 (19)	1.3726 (3)	0.07163 (11)	0.0184 (4)
H112	0.2992	1.3849	0.1018	0.022*
C111	0.20393 (19)	1.3632 (3)	−0.00206 (11)	0.0179 (4)
H111	0.2786	1.3688	−0.0228	0.021*
O101	0.10387 (13)	1.3698 (2)	0.17252 (7)	0.0173 (3)
C101	0.21582 (18)	1.4443 (3)	0.22070 (10)	0.0150 (4)
H101	0.2387	1.574	0.203	0.018*
C102	0.17225 (19)	1.4730 (3)	0.29214 (10)	0.0143 (4)
H102	0.0905	1.551	0.2848	0.017*
O102	0.27280 (15)	1.5708 (2)	0.34038 (8)	0.0188 (3)
H12	0.259 (3)	1.692 (6)	0.3366 (17)	0.05*
C103	0.15121 (19)	1.2737 (3)	0.32381 (10)	0.0136 (4)
H103	0.0781	1.2065	0.2914	0.016*
O103	0.11609 (15)	1.3034 (2)	0.39202 (7)	0.0180 (3)
H13	0.115 (3)	1.197 (6)	0.4126 (18)	0.05*

C104	0.2735 (2)	1.1506 (3)	0.32928 (10)	0.0136 (4)
H104	0.3475	1.2151	0.3619	0.016*
O104	0.25457 (15)	0.9597 (2)	0.35459 (8)	0.0173 (3)
H14	0.246 (3)	0.959 (6)	0.3956 (19)	0.05*
C105	0.30432 (19)	1.1288 (3)	0.25488 (11)	0.0151 (4)
H105	0.2297	1.0641	0.2228	0.018*
C106	0.4262 (2)	1.0144 (3)	0.25400 (12)	0.0232 (5)
H10A	0.4425	1.0112	0.2054	0.035*
H10B	0.4157	0.8811	0.2703	0.035*
H10C	0.4995	1.0764	0.2858	0.035*
O105	0.32371 (13)	1.3205 (2)	0.22692 (7)	0.0154 (3)
N213	−0.39091 (16)	0.4276 (3)	0.01163 (9)	0.0194 (4)
O215	−0.32520 (14)	0.3120 (3)	−0.01531 (8)	0.0235 (3)
O214	−0.46481 (17)	0.5465 (3)	−0.02365 (8)	0.0322 (4)
C210	−0.38055 (19)	0.4233 (3)	0.08925 (10)	0.0168 (4)
C209	−0.4304 (2)	0.5769 (3)	0.12271 (11)	0.0190 (4)
H209	−0.4724	0.6827	0.0955	0.023*
C208	−0.41803 (19)	0.5738 (3)	0.19635 (11)	0.0180 (4)
H208	−0.4504	0.6787	0.2201	0.022*
C207	−0.35783 (18)	0.4156 (3)	0.23528 (10)	0.0146 (4)
C212	−0.3100 (2)	0.2609 (3)	0.20071 (11)	0.0180 (4)
H212	−0.2699	0.1532	0.2276	0.022*
C211	−0.32092 (19)	0.2644 (3)	0.12718 (11)	0.0178 (4)
H211	−0.2883	0.1602	0.1033	0.021*
O201	−0.34096 (13)	0.3963 (2)	0.30809 (7)	0.0153 (3)
C201	−0.37263 (19)	0.5583 (3)	0.34905 (10)	0.0141 (4)
H201	−0.4677	0.5847	0.3353	0.017*
C202	−0.34018 (18)	0.4911 (3)	0.42743 (10)	0.0139 (4)
H202	−0.3827	0.363	0.4323	0.017*
O202	−0.38516 (14)	0.6325 (2)	0.47115 (8)	0.0168 (3)
H22	−0.464 (3)	0.597 (6)	0.4737 (17)	0.05*
C203	−0.19271 (18)	0.4703 (3)	0.45089 (10)	0.0122 (4)
H203	−0.1636	0.355	0.4263	0.015*
O203	−0.16051 (13)	0.4396 (2)	0.52635 (7)	0.0148 (3)
H23	−0.114 (3)	0.341 (6)	0.5351 (17)	0.05*
C204	−0.12210 (17)	0.6509 (3)	0.43223 (10)	0.0123 (4)
H204	−0.1453	0.7623	0.4609	0.015*
O204	0.01541 (13)	0.6233 (2)	0.45107 (7)	0.0149 (3)
H24	0.036 (3)	0.533 (6)	0.4294 (18)	0.05*
C206	−0.1076 (2)	0.8921 (3)	0.33282 (11)	0.0202 (4)
H20A	−0.1367	0.9148	0.2815	0.03*
H20B	−0.0128	0.8843	0.3443	0.03*
H20C	−0.1365	0.9997	0.3595	0.03*
C205	−0.16438 (18)	0.7030 (3)	0.35314 (10)	0.0131 (4)
H205	−0.1398	0.5948	0.3234	0.016*
O205	−0.30423 (13)	0.7267 (2)	0.33721 (7)	0.0142 (3)
O1	0.62208 (15)	0.0270 (2)	0.50941 (9)	0.0203 (3)
H1A	0.628 (3)	−0.084 (6)	0.4891 (18)	0.05*
H1B	0.664 (3)	0.021 (6)	0.5520 (19)	0.05*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
N113	0.0260 (9)	0.0160 (8)	0.0201 (9)	−0.0018 (7)	0.0036 (7)	−0.0005 (7)
O114	0.0280 (8)	0.0352 (9)	0.0194 (7)	−0.0047 (7)	−0.0025 (6)	−0.0013 (7)
O115	0.0295 (9)	0.0478 (11)	0.0201 (8)	−0.0030 (8)	0.0083 (7)	−0.0006 (8)
C110	0.0221 (10)	0.0125 (9)	0.0154 (9)	−0.0003 (8)	0.0023 (8)	0.0008 (7)
C109	0.0173 (10)	0.0138 (9)	0.0216 (10)	−0.0015 (8)	0.0003 (7)	−0.0003 (8)
C108	0.0162 (9)	0.0151 (9)	0.0222 (10)	−0.0013 (8)	0.0057 (8)	0.0005 (8)
C107	0.0174 (9)	0.0125 (9)	0.0163 (9)	0.0001 (8)	0.0030 (7)	0.0007 (7)
C112	0.0153 (9)	0.0205 (10)	0.0185 (10)	−0.0014 (8)	0.0014 (7)	0.0014 (8)
C111	0.0184 (10)	0.0166 (10)	0.0195 (10)	−0.0010 (8)	0.0056 (8)	0.0022 (8)
O101	0.0152 (7)	0.0213 (7)	0.0151 (7)	−0.0021 (6)	0.0026 (5)	0.0011 (6)
C101	0.0138 (9)	0.0137 (9)	0.0169 (9)	−0.0009 (7)	0.0019 (7)	0.0010 (7)
C102	0.0155 (9)	0.0105 (8)	0.0160 (9)	0.0034 (7)	0.0010 (7)	0.0014 (7)
O102	0.0267 (8)	0.0091 (6)	0.0192 (7)	0.0000 (6)	0.0010 (6)	−0.0003 (5)
C103	0.0157 (9)	0.0122 (8)	0.0135 (9)	−0.0005 (7)	0.0039 (7)	−0.0007 (7)
O103	0.0260 (8)	0.0131 (7)	0.0170 (7)	0.0042 (6)	0.0097 (6)	0.0012 (6)
C104	0.0168 (9)	0.0079 (8)	0.0166 (9)	0.0004 (7)	0.0045 (7)	0.0003 (7)
O104	0.0280 (8)	0.0092 (6)	0.0171 (7)	0.0004 (6)	0.0105 (6)	0.0006 (5)
C105	0.0173 (10)	0.0111 (8)	0.0183 (10)	−0.0007 (8)	0.0067 (7)	−0.0004 (7)
C106	0.0255 (11)	0.0168 (10)	0.0317 (12)	0.0061 (8)	0.0163 (9)	0.0045 (9)
O105	0.0153 (7)	0.0131 (6)	0.0189 (7)	0.0008 (5)	0.0057 (5)	0.0033 (5)
N213	0.0159 (8)	0.0243 (9)	0.0178 (8)	−0.0008 (7)	0.0029 (7)	−0.0022 (7)
O215	0.0200 (7)	0.0317 (8)	0.0196 (7)	0.0015 (7)	0.0057 (6)	−0.0045 (7)
O214	0.0348 (9)	0.0422 (10)	0.0171 (8)	0.0156 (8)	−0.0009 (6)	0.0026 (7)
C210	0.0132 (9)	0.0226 (10)	0.0139 (9)	−0.0016 (8)	0.0012 (7)	−0.0024 (8)
C209	0.0160 (10)	0.0201 (10)	0.0190 (10)	0.0038 (8)	−0.0008 (8)	0.0002 (8)
C208	0.0161 (10)	0.0176 (9)	0.0194 (10)	0.0042 (8)	0.0013 (8)	−0.0018 (8)
C207	0.0120 (9)	0.0158 (9)	0.0150 (9)	−0.0021 (7)	0.0001 (7)	−0.0034 (7)
C212	0.0174 (9)	0.0155 (9)	0.0199 (10)	0.0024 (8)	0.0006 (8)	−0.0006 (8)
C211	0.0165 (10)	0.0189 (10)	0.0179 (10)	0.0019 (8)	0.0035 (8)	−0.0046 (8)
O201	0.0176 (7)	0.0136 (7)	0.0136 (7)	0.0015 (6)	0.0008 (5)	−0.0013 (5)
C201	0.0128 (9)	0.0138 (9)	0.0152 (9)	0.0001 (8)	0.0013 (7)	−0.0033 (7)
C202	0.0127 (9)	0.0130 (9)	0.0162 (9)	0.0000 (7)	0.0031 (7)	−0.0021 (7)
O202	0.0157 (7)	0.0159 (7)	0.0197 (7)	0.0000 (6)	0.0061 (6)	−0.0038 (6)
C203	0.0143 (9)	0.0118 (8)	0.0102 (8)	0.0014 (7)	0.0014 (7)	0.0001 (7)
O203	0.0185 (7)	0.0143 (7)	0.0114 (6)	0.0026 (6)	0.0027 (5)	0.0018 (5)
C204	0.0102 (8)	0.0124 (8)	0.0141 (9)	0.0011 (7)	0.0017 (7)	−0.0005 (7)
O204	0.0110 (6)	0.0140 (7)	0.0185 (7)	0.0012 (5)	0.0005 (5)	−0.0024 (6)
C206	0.0226 (10)	0.0194 (10)	0.0169 (10)	−0.0050 (9)	0.0001 (8)	0.0052 (8)
C205	0.0123 (9)	0.0135 (8)	0.0130 (9)	0.0011 (7)	0.0016 (7)	0.0003 (7)
O205	0.0127 (7)	0.0133 (7)	0.0155 (7)	0.0011 (5)	0.0003 (5)	0.0011 (5)
O1	0.0191 (7)	0.0178 (8)	0.0238 (8)	0.0025 (6)	0.0032 (6)	−0.0006 (6)

Geometric parameters (Å, °)

N113—O115	1.236 (2)	N213—O215	1.232 (2)

N113—O114	1.236 (2)	N213—C210	1.468 (3)
N113—C110	1.472 (3)	C210—C211	1.389 (3)
C110—C111	1.389 (3)	C210—C209	1.390 (3)
C110—C109	1.395 (3)	C209—C208	1.389 (3)
C109—C108	1.382 (3)	C209—H209	0.95
C109—H109	0.95	C208—C207	1.396 (3)
C108—C107	1.401 (3)	C208—H208	0.95
C108—H108	0.95	C207—O201	1.375 (2)
C107—O101	1.374 (2)	C207—C212	1.398 (3)
C107—C112	1.403 (3)	C212—C211	1.389 (3)
C112—C111	1.391 (3)	C212—H212	0.95
C112—H112	0.95	C211—H211	0.95
C111—H111	0.95	O201—C201	1.438 (2)
O101—C101	1.438 (2)	C201—O205	1.405 (2)
C101—O105	1.406 (2)	C201—C202	1.541 (3)
C101—C102	1.539 (3)	C201—H201	1
C101—H101	1	C202—O202	1.424 (2)
C102—O102	1.426 (2)	C202—C203	1.535 (3)
C102—C103	1.531 (3)	C202—H202	1
C102—H102	1	O202—H22	0.88 (3)
O102—H12	0.85 (4)	C203—O203	1.431 (2)
C103—O103	1.440 (2)	C203—C204	1.525 (3)
C103—C104	1.527 (3)	C203—H203	1
C103—H103	1	O203—H23	0.84 (4)
O103—H13	0.83 (4)	C204—O204	1.434 (2)
C104—O104	1.426 (2)	C204—C205	1.532 (3)
C104—C105	1.530 (3)	C204—H204	1
C104—H104	1	O204—H24	0.80 (4)
O104—H14	0.81 (4)	C206—C205	1.513 (3)
C105—O105	1.450 (2)	C206—H20A	0.98
C105—C106	1.508 (3)	C206—H20B	0.98
C105—H105	1	C206—H20C	0.98
C106—H10A	0.98	C205—O205	1.453 (2)
C106—H10B	0.98	C205—H205	1
C106—H10C	0.98	O1—H1A	0.86 (4)
N213—O214	1.232 (2)	O1—H1B	0.85 (4)
O115—N113—O114	123.06 (18)	O214—N213—C210	118.61 (17)
O115—N113—C110	118.34 (17)	O215—N213—C210	118.43 (17)
O114—N113—C110	118.61 (17)	C211—C210—C209	121.90 (18)
C111—C110—C109	121.97 (19)	C211—C210—N213	118.60 (18)
C111—C110—N113	119.34 (18)	C209—C210—N213	119.49 (18)
C109—C110—N113	118.69 (17)	C208—C209—C210	119.23 (19)
C108—C109—C110	118.56 (18)	C208—C209—H209	120.4
C108—C109—H109	120.7	C210—C209—H209	120.4
C110—C109—H109	120.7	C209—C208—C207	119.57 (19)
C109—C108—C107	120.32 (18)	C209—C208—H208	120.2
C109—C108—H108	119.8	C207—C208—H208	120.2
C107—C108—H108	119.8	O201—C207—C208	124.66 (18)
O101—C107—C108	114.84 (17)	O201—C207—C212	114.85 (18)

O101—C107—C112	124.57 (17)	C208—C207—C212	120.49 (18)
C108—C107—C112	120.59 (18)	C211—C212—C207	120.10 (19)
C111—C112—C107	119.06 (18)	C211—C212—H212	119.9
C111—C112—H112	120.5	C207—C212—H212	119.9
C107—C112—H112	120.5	C210—C211—C212	118.68 (18)
C110—C111—C112	119.49 (18)	C210—C211—H211	120.7
C110—C111—H111	120.3	C212—C211—H211	120.7
C112—C111—H111	120.3	C207—O201—C201	118.71 (15)
C107—O101—C101	118.97 (15)	O205—C201—O201	111.51 (15)
O105—C101—O101	112.63 (16)	O205—C201—C202	112.49 (16)
O105—C101—C102	112.46 (15)	O201—C201—C202	105.56 (15)
O101—C101—C102	105.23 (15)	O205—C201—H201	109.1
O105—C101—H101	108.8	O201—C201—H201	109.1
O101—C101—H101	108.8	C202—C201—H201	109.1
C102—C101—H101	108.8	O202—C202—C203	109.15 (15)
O102—C102—C103	108.21 (15)	O202—C202—C201	109.41 (15)
O102—C102—C101	108.67 (16)	C203—C202—C201	109.08 (15)
C103—C102—C101	109.26 (15)	O202—C202—H202	109.7
O102—C102—H102	110.2	C203—C202—H202	109.7
C103—C102—H102	110.2	C201—C202—H202	109.7
C101—C102—H102	110.2	C202—O202—H22	106 (2)
C102—O102—H12	108 (2)	O203—C203—C204	109.13 (14)
O103—C103—C104	112.48 (15)	O203—C203—C202	109.29 (15)
O103—C103—C102	108.43 (15)	C204—C203—C202	111.63 (15)
C104—C103—C102	109.58 (16)	O203—C203—H203	108.9
O103—C103—H103	108.8	C204—C203—H203	108.9
C104—C103—H103	108.8	C202—C203—H203	108.9
C102—C103—H103	108.8	C203—O203—H23	109 (2)
C103—O103—H13	110 (2)	O204—C204—C203	110.44 (15)
O104—C104—C103	111.05 (16)	O204—C204—C205	111.02 (15)
O104—C104—C105	107.24 (15)	C203—C204—C205	111.23 (15)
C103—C104—C105	108.90 (16)	O204—C204—H204	108
O104—C104—H104	109.9	C203—C204—H204	108
C103—C104—H104	109.9	C205—C204—H204	108
C105—C104—H104	109.9	C204—O204—H24	110 (2)
C104—O104—H14	113 (3)	C205—C206—H20A	109.5
O105—C105—C106	106.61 (16)	C205—C206—H20B	109.5
O105—C105—C104	109.04 (15)	H20A—C206—H20B	109.5
C106—C105—C104	113.64 (17)	C205—C206—H20C	109.5
O105—C105—H105	109.2	H20A—C206—H20C	109.5
C106—C105—H105	109.2	H20B—C206—H20C	109.5
C104—C105—H105	109.2	O205—C205—C206	106.63 (15)
C105—C106—H10A	109.5	O205—C205—C204	108.41 (14)
C105—C106—H10B	109.5	C206—C205—C204	113.74 (16)
H10A—C106—H10B	109.5	O205—C205—H205	109.3
C105—C106—H10C	109.5	C206—C205—H205	109.3
H10A—C106—H10C	109.5	C204—C205—H205	109.3
H10B—C106—H10C	109.5	C201—O205—C205	113.88 (14)
C101—O105—C105	114.15 (14)	H1A—O1—H1B	109 (3)

O214—N213—O215	122.95 (17)		
O115—N113—C110—C111	0.4 (3)	O214—N213—C210—C211	165.9 (2)
O114—N113—C110—C111	−179.17 (19)	O215—N213—C210—C211	−14.1 (3)
O115—N113—C110—C109	−179.6 (2)	O214—N213—C210—C209	−14.0 (3)
O114—N113—C110—C109	0.8 (3)	O215—N213—C210—C209	166.02 (19)
C111—C110—C109—C108	−0.3 (3)	C211—C210—C209—C208	1.3 (3)
N113—C110—C109—C108	179.77 (18)	N213—C210—C209—C208	−178.75 (17)
C110—C109—C108—C107	0.3 (3)	C210—C209—C208—C207	−1.0 (3)
C109—C108—C107—O101	−179.59 (18)	C209—C208—C207—O201	−179.57 (18)
C109—C108—C107—C112	0.0 (3)	C209—C208—C207—C212	0.0 (3)
O101—C107—C112—C111	179.37 (18)	O201—C207—C212—C211	−179.71 (17)
C108—C107—C112—C111	−0.1 (3)	C208—C207—C212—C211	0.6 (3)
C109—C110—C111—C112	0.1 (3)	C209—C210—C211—C212	−0.7 (3)
N113—C110—C111—C112	−179.95 (18)	N213—C210—C211—C212	179.43 (17)
C107—C112—C111—C110	0.1 (3)	C207—C212—C211—C210	−0.3 (3)
C108—C107—O101—C101	−161.68 (17)	C208—C207—O201—C201	−7.9 (3)
C112—C107—O101—C101	18.8 (3)	C212—C207—O201—C201	172.50 (17)
C107—O101—C101—O105	−69.4 (2)	C207—O201—C201—O205	−56.3 (2)
C107—O101—C101—C102	167.78 (16)	C207—O201—C201—C202	−178.77 (15)
O105—C101—C102—O102	64.6 (2)	O205—C201—C202—O202	66.1 (2)
O101—C101—C102—O102	−172.44 (15)	O201—C201—C202—O202	−172.04 (14)
O105—C101—C102—C103	−53.3 (2)	O205—C201—C202—C203	−53.2 (2)
O101—C101—C102—C103	69.69 (19)	O201—C201—C202—C203	68.62 (19)
O102—C102—C103—O103	59.34 (19)	O202—C202—C203—O203	51.0 (2)
C101—C102—C103—O103	177.50 (14)	C201—C202—C203—O203	170.50 (15)
O102—C102—C103—C104	−63.78 (19)	O202—C202—C203—C204	−69.81 (19)
C101—C102—C103—C104	54.4 (2)	C201—C202—C203—C204	49.7 (2)
O103—C103—C104—O104	62.8 (2)	O203—C203—C204—O204	62.33 (19)
C102—C103—C104—O104	−176.49 (15)	C202—C203—C204—O204	−176.76 (14)
O103—C103—C104—C105	−179.32 (15)	O203—C203—C204—C205	−173.93 (14)
C102—C103—C104—C105	−58.6 (2)	C202—C203—C204—C205	−53.0 (2)
O104—C104—C105—O105	179.73 (15)	O204—C204—C205—O205	179.33 (14)
C103—C104—C105—O105	59.5 (2)	C203—C204—C205—O205	55.92 (19)
O104—C104—C105—C106	−61.5 (2)	O204—C204—C205—C206	−62.3 (2)
C103—C104—C105—C106	178.25 (17)	C203—C204—C205—C206	174.33 (16)
O101—C101—O105—C105	−61.5 (2)	O201—C201—O205—C205	−57.5 (2)
C102—C101—O105—C105	57.2 (2)	C202—C201—O205—C205	60.9 (2)
C106—C105—O105—C101	177.03 (16)	C206—C205—O205—C201	176.29 (15)
C104—C105—O105—C101	−59.9 (2)	C204—C205—O205—C201	−60.88 (19)

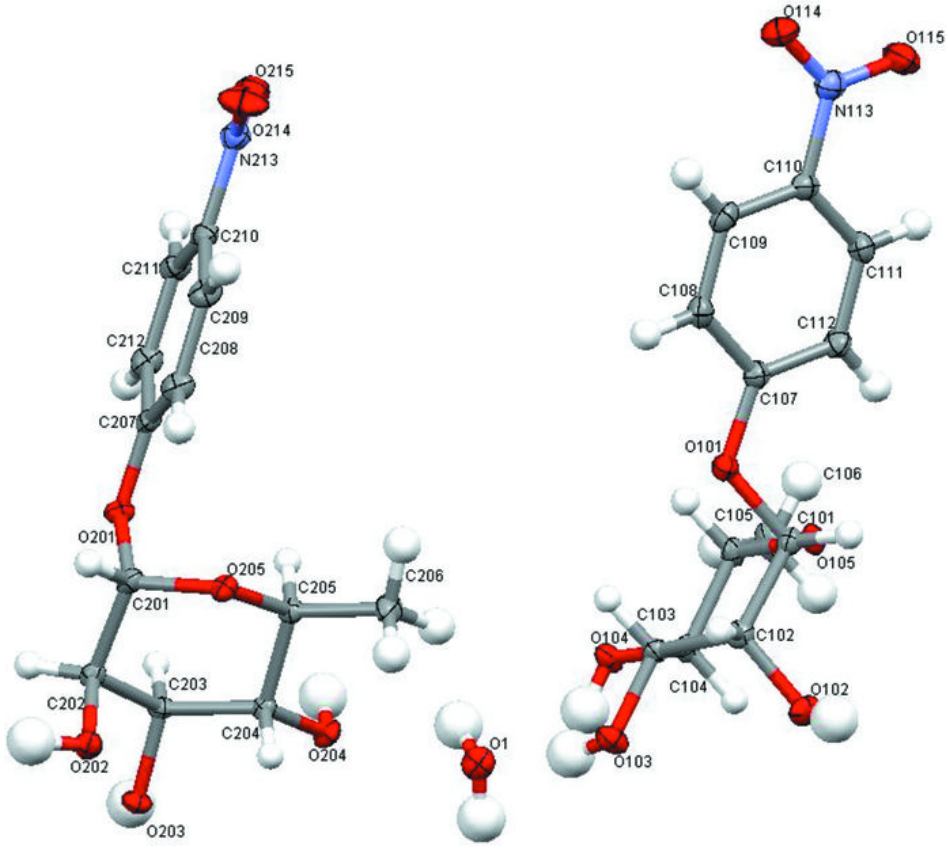
Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O1—H1A···O202^{i}	0.86 (3)	1.97 (7)	2.803 (7)	159 (9)
O1—H1B···O102^{ii}	0.84 (8)	2.06 (1)	2.881 (8)	162 (7)
O102—H12···O104^{iii}	0.84 (5)	1.87 (3)	2.695 (6)	163 (8)
O103—H13···O203^{iv}	0.83 (0)	2.12 (3)	2.934 (2)	165 (6)
O104—H14···O203^{iv}	0.80 (8)	1.89 (5)	2.663 (7)	158 (5)

O202—H22···O1^{iv}	0.88 (0)	1.82 (0)	2.695 (8)	173 (0)
O203—H23···O204^{v}	0.83 (7)	1.80 (6)	2.641 (8)	176 (8)
O204—H24···O103^{vi}	0.79 (7)	1.99 (1)	2.776 (5)	168 (4)

Symmetry codes: (i) *x*+1, *y*−1, *z*; (ii) −*x*+1, *y*−3/2, −*z*+1; (iii) *x*, *y*+1, *z*; (iv) −*x*, *y*+1/2, −*z*+1; (v) −*x*, *y*−1/2, −*z*+1; (vi) *x*, *y*−1, *z*.

Fig. 1


O114
O115
N113
C110
C109
C111
C108
C112
C107
O101
C106
C105
C101
O105
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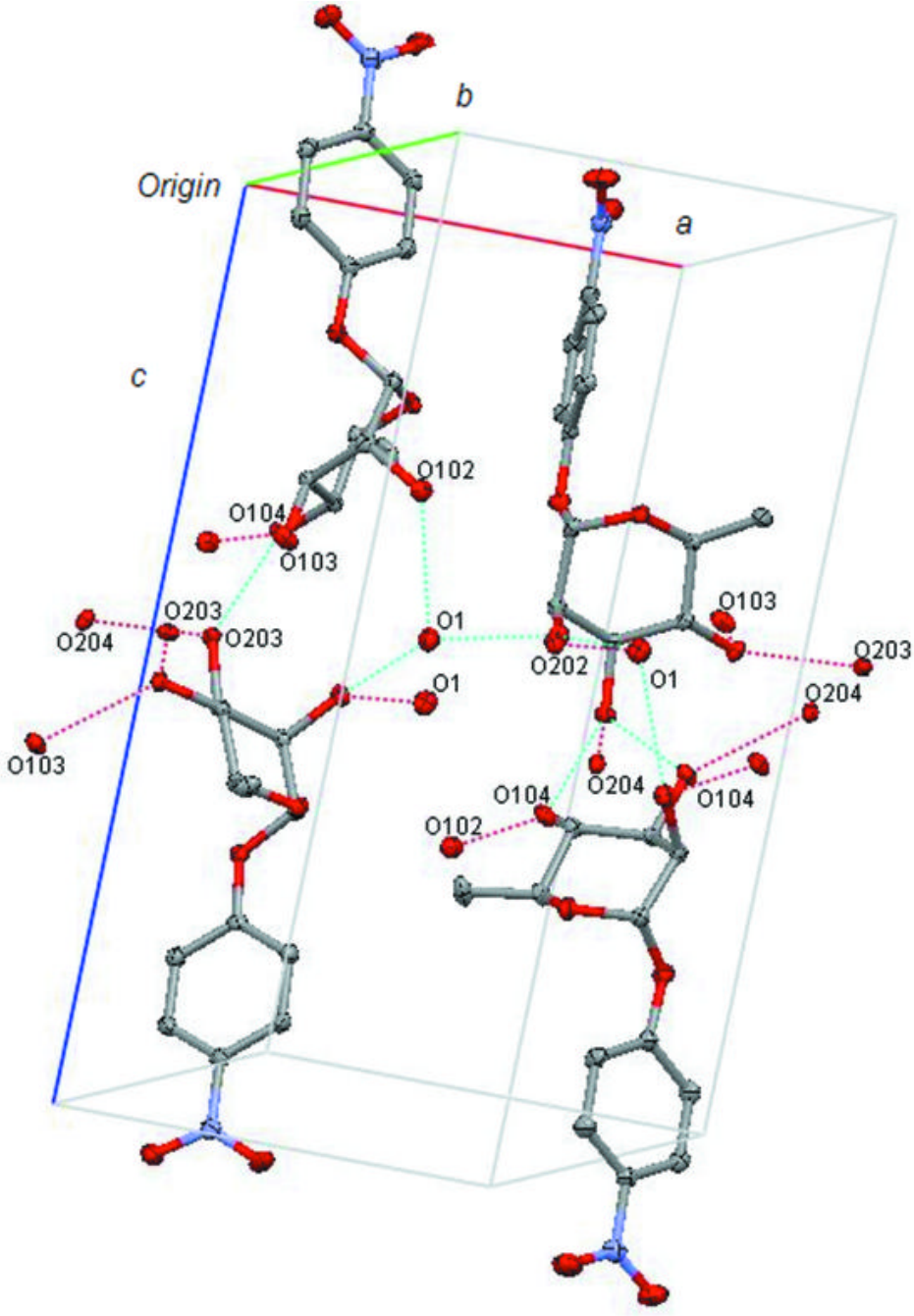
O104
C102
C104
O102
O103
O215
O214
N213
C210
C211
C209
C212
C208
C207
O201
O205
C205
C206
C201
C203
C202
C204
O204
O202
O203
O1

Fig. 2


b
Origin
a
c
O102
O104
O103
O203
O204
O203
O1
O1
O103
O202
O1
O103
O203
O204
O204
O104
O104
O102