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Benzene-1,2,4,5-tetrol

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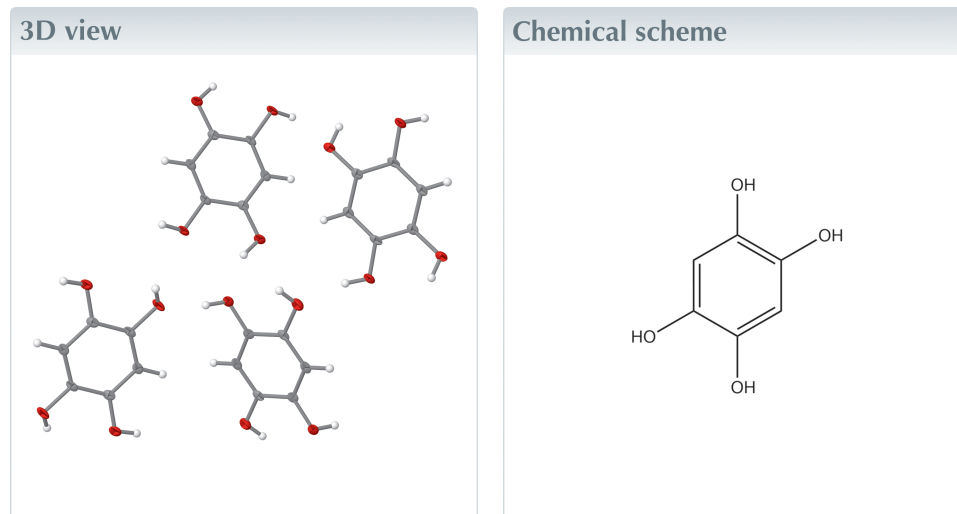
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Keywords: benzene-1,2,4,5-tetrol; crystal structure; hydrogen bonds; covalent organic framework; hydroxyl.

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Structural data: full structural data are available from iucrdata.iucr.org

The crystal structure of the title compound was determined at 120 K. It crystallizes in the triclinic space group $P\bar{1}$ with four independent molecules in the asymmetric unit. In the crystal, each symmetry-unique molecule forms π - π stacks on itself, giving four unique π - π stacking interactions. Intermolecular hydrogen bonding is observed between each pair of independent molecules, where each hydroxy group can act as a hydrogen-bond donor and acceptor.



Structure description

Benzene-1,2,4,5-tetrol, a derivative of 2,5-dihydroxy-1,4-benzoquinone, has seen extensive use as a precursor to functionalized benzenes as well as more complex molecules and ligands. It has been used to access a number of more complex organic structures, such as phosphorous-containing ligands for transition-metal complexes (Pandey *et al.*, 2019) or to bridge metal centres in complexes (Wellala *et al.*, 2018). In recent years benzene-1,2,4,5-tetrol has found a niche as a monomer for the synthesis of polymers, coordination polymers, covalent organic frameworks, and a variety of other supramolecular structures. It has seen extensive use in the synthesis of framework polymers where it acts as a linear monomer linking other structural units. Recent examples include combining benzene-1,2,4,5-tetrol with a boronic acid-containing porphyrin, a two-dimensional square-pored boronate ester covalent organic framework (COF), creating a thin film that could be integrated into a field-effect transistor (Park *et al.*, 2020), as well as the creation of hafnium- and zirconium-containing coordination polymers with water sorption properties, using benzene-1,2,4,5-tetrol as a linker (Poschmann *et al.*, 2021). Benzene-1,2,4,5-tetrol has also been used in the synthesis of a variety of other COFs (Rondelli *et al.*, 2023; Dalapati *et al.*, 2015; Ma *et al.*, 2013; Lanni *et al.*, 2011), coordination polymers (Abrahams *et al.*, 2016), supramolecular structures (Jia *et al.*, 2015; Niu *et al.*, 2006; Nakabayashi &

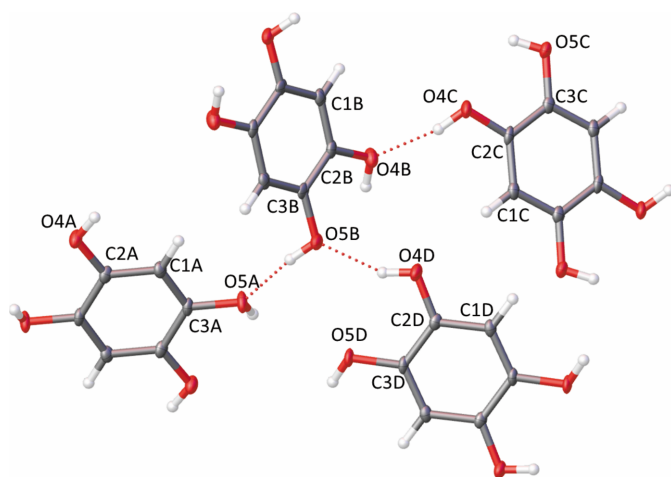


Figure 1
The asymmetric unit of the title compound showing the atom labelling with 50% probability displacement ellipsoids. Unlabelled atoms are related to labelled atoms by the symmetry operations $-x, -y + 2, -z$ for molecule *A*, $-x + 1, -y + 1, -z$ for molecule *B*, $-x + 1, -y, -z + 1$ for molecule *C* and $-x + 2, -y + 1, -z + 1$ for molecule *D*.

Ohkoshi, 2009; Yuan *et al.*, 2012), and polymers (Christinat *et al.*, 2007; Rambo & Lavigne, 2007; Nishiyabu *et al.*, 2012).

Despite of the ongoing interest in benzene-1,2,4,5-tetrol as a reagent, which stretches back at least a century (Mukerji, 1922), the crystal structure has only been solved as a water solvate and a co-crystal with 2,5-dihydroxy-1,4-benzoquinone (Jene *et al.*, 2001). A search of the Cambridge Structure Database (WebCSD, December 2023) for the molecular structure of 1,2,4,5-benzenetetrol gave three results: 1,2,4,5-tetrahydroxybenzene monohydrate (QOGMAA; Jene *et al.*, 2001); and 1,2,4,5-tetrahydroxybenzene 2,5-dihydroxy-1,4-benzoquinone (QOGMII, QOGMIII01; Jene *et al.*, 2001). Here we present the crystal structure of benzene-1,2,4,5-tetrol for

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

<i>D</i> –H··· <i>A</i>	<i>D</i> –H	H··· <i>A</i>	<i>D</i> ··· <i>A</i>	<i>D</i> –H··· <i>A</i>
O4A–H4A···O4C ⁱ	0.85 (2)	1.89 (2)	2.715 (2)	163 (2)
O4B–H4B···O4D ⁱⁱ	0.86 (2)	1.88 (2)	2.708 (2)	163 (3)
O4B–H4B···O5B	0.86 (2)	2.45 (2)	2.764 (2)	102 (2)
O4C–H4C···O4B	0.86 (2)	1.85 (2)	2.702 (2)	167 (2)
O4D–H4D···O5B	0.86 (2)	1.85 (2)	2.6425 (19)	154 (2)
O4D–H4D···O5D	0.86 (2)	2.34 (2)	2.789 (2)	113 (2)
O5A–H5A···O4A	0.83 (2)	2.40 (2)	2.711 (2)	103 (2)
O5A–H5A···O5D ⁱⁱ	0.83 (2)	1.95 (2)	2.7562 (18)	162 (2)
O5B–H5B···O5A	0.84 (2)	1.80 (2)	2.633 (2)	169 (2)
O5C–H5C···O4A ⁱⁱⁱ	0.83 (2)	2.04 (2)	2.8376 (16)	161 (2)
O5C–H5C···O4C	0.83 (2)	2.38 (2)	2.734 (2)	107 (2)
O5D–H5D···O5C ^{iv}	0.85 (2)	2.03 (2)	2.8796 (19)	175 (2)

Symmetry codes: (i) $x - 1, y + 1, z$; (ii) $x - 1, y, z$; (iii) $x, y - 1, z$; (iv) $x, y + 1, z$.

the first time, which we anticipate will be of use for the synthetic chemical community in future endeavours.

At 120 K the structure was found to crystallize in the triclinic space group $P\bar{1}$ with the asymmetric unit containing four independent molecules of benzene-1,2,4,5-tetrol labelled *A*, *B*, *C* and *D* (Figs. 1, 2*a*). Each symmetry unique molecule forms π – π stacks on itself, *i.e.* molecule *A* forms a stack consisting entirely of molecule *A* (Fig. 2*b*). This gives four unique π – π stacking interactions with centroid-to-distances of 3.7474 (11) Å, while the perpendicular centroid-to-plane distances are 3.4457 (7) Å (molecule *A*), 3.5166 (8) Å (molecule *B*), 3.5653 (8) Å (molecule *C*), and 3.5653 (8) Å (molecule *D*). Intermolecular hydrogen bonding is observed between each pair of molecules, where each hydroxy group can act as a hydrogen-bond donor and acceptor (Table 1). This creates an extended hydrogen-bond network, which can be described as a series of rings consisting of three molecules – the edges of two molecules make up the perimeter of the ring, and a single hydroxy group of a third molecule links the first two molecules into a continuous ring. There are two

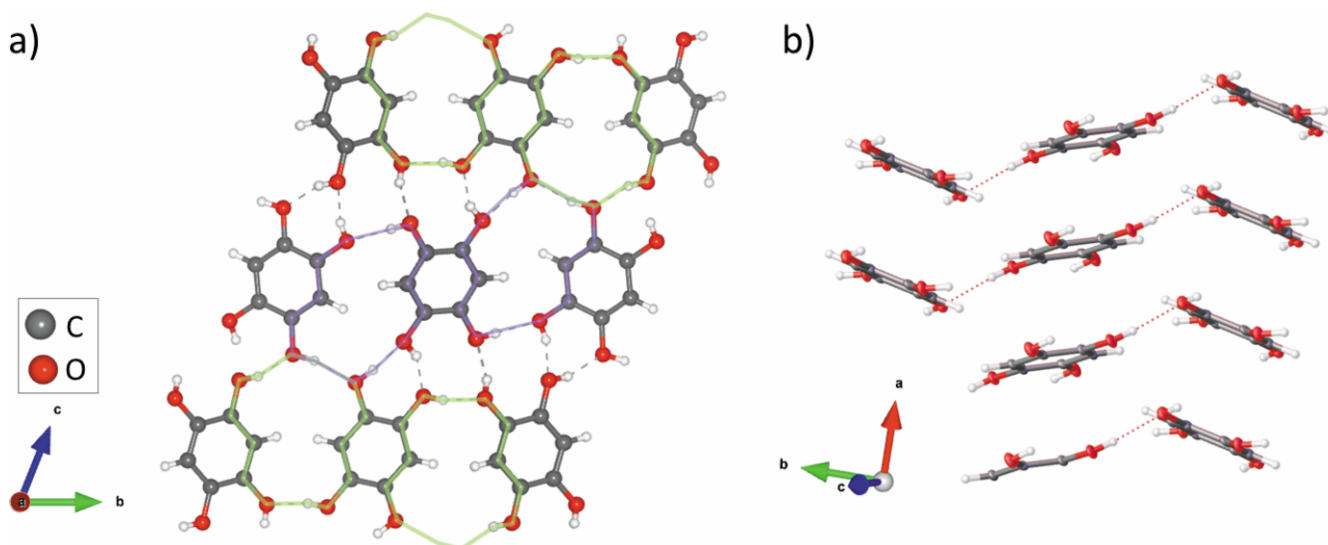


Figure 2
(*a*) View of unit cell along the crystallographic *a*-axis. Dashed lines represent hydrogen bonding between molecules. $R_2^2(14)$ rings are indicated with purple and green polygons; hydrogen bonds not lying on the indicated rings form the same class of ring with molecules not rendered in this diagram. (*b*) View approximately along the (001) axis, showing how molecules form π – π stacks. Some molecules have been removed for clarity.

Table 2

Experimental details.

Crystal data	
Chemical formula	C ₆ H ₆ O ₄
<i>M_r</i>	142.11
Crystal system, space group	Triclinic, <i>P</i> $\bar{1}$
Temperature (K)	120
<i>a</i> , <i>b</i> , <i>c</i> (Å)	3.7474 (2), 11.6254 (6), 13.7771 (8)
α , β , γ (°)	68.407 (5), 85.779 (4), 89.843 (4)
<i>V</i> (Å ³)	556.37 (6)
<i>Z</i>	4
Radiation type	Cu <i>K</i> α
μ (mm ⁻¹)	1.27
Crystal size (mm)	0.07 × 0.05 × 0.02
Data collection	
Diffractometer	XtalLAB PRO MM007, PILATUS3 R 200K
Absorption correction	Gaussian (<i>CrysAlis PRO</i> ; Rigaku OD, 2023)
<i>T_{min}</i> , <i>T_{max}</i>	0.927, 1.000
No. of measured, independent and observed [<i>I</i> > 2 σ (<i>I</i>)] reflections	8096, 2185, 1842
<i>R_{int}</i>	0.063
(<i>sin</i> θ / λ) _{max} (Å ⁻¹)	0.630
Refinement	
<i>R</i> [<i>F</i> ² > 2 σ (<i>F</i> ²)], <i>wR</i> (<i>F</i> ²), <i>S</i>	0.045, 0.133, 1.09
No. of reflections	2185
No. of parameters	205
No. of restraints	8
H-atom treatment	H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{\text{max}}$, $\Delta\rho_{\text{min}}$ (e Å ⁻³)	0.28, -0.34

Computer programs: *CrysAlis PRO* (Rigaku OD, 2023), *SHELXT2018/2* (Sheldrick, 2015a), *SHELXL2018/3* (Sheldrick, 2015b) and *OLEX2* (Dolomanov *et al.*, 2009).

unique rings comprised of molecules *A*, *B*, and *C*, and of molecules *C*, *B*, and *D*, both of which exhibit an *R*₂²(14) graph-set motif, and the remaining hydrogen-bonded rings are symmetry-related. All of the hydrogen bonds in the structure can thus be accounted for.

Synthesis and crystallization

Following a literature procedure (Weider *et al.*, 1985), 2,5-dihydroxy-1,4-benzoquinone (2.428 g, 17.3 mmol) was mixed with conc. hydrochloric acid (54 ml) under an inert atmosphere and stirred for 30 min to form a gold-coloured suspension. Addition of tin metal powder (2.1885 g, 18.4 mmol) caused vigorous effervescence and a grey suspension. The mixture was stirred for 10 min until cessation of bubbling then heated to 100° C for 1 h, during which time the mixture became dark and bubbled vigorously. The mixture was allowed to cool briefly, then hot filtered under reduced pressure to give a yellow filtrate. The filtrate was cooled on ice for 30 min to give white crystals of benzene-1,2,4,5-tetrol (0.786 g, 5.54 mmol, 32%). The crude product was dissolved in a minimum of hot tetrahydrofuran, filtered, then cooled on ice. The resulting white crystals were collected *via* filtration then washed with ice-cold THF and dried in a vacuum to give benzene-1,2,4,5-tetrol (0.735 g, 5.17 mmol, 30%). IR (ATR) ν_{max} /cm⁻¹: 3146.01 *br* (OH), 1551.54 *s* (Ar C—C), 1155.90 *w* (C—O) MS (ESI) *m/z*: 165.02 (*M*+Na). ¹H NMR (400 MHz,

DMSO-*d*₆, p.p.m., δ): 9.66 (*s*, 4H, OH), 5.94 (*s*, 2H, Ar H); ¹³C NMR (400 MHz, DMSO-*d*₆, p.p.m., δ): 138.46, 104.81. CNH analysis found: C, 50.6; H, 4.1; N, 0. Calculated for C₆H₆O₄: C, 50.7; H, 4.3; N, 0%.

Refinement

Crystal data, data collection and structure refinement details are summarized in Table 2.

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References

- Abrahams, B. F., Dharma, A. D., Dyett, B., Hudson, T. A., Maynard-Casely, H., Kingsbury, C. J., McCormick, L. J., Robson, R., Sutton, A. L. & White, K. F. (2016). *Dalton Trans.* **45**, 1339–1344.
- Christinat, N., Croisier, E., Scopelliti, R., Cascella, M., Röthlisberger, U. & Severin, K. (2007). *Eur. J. Inorg. Chem.* pp. 5177–5181.
- Dalapati, S., Addicoat, M., Jin, S., Sakurai, T., Gao, J., Xu, H., Irle, S., Seki, S. & Jiang, D. (2015). *Nat. Commun.* **6**, 7786.
- Dolomanov, O. V., Bourhis, L. J., Gildea, R. J., Howard, J. A. K. & Puschmann, H. (2009). *J. Appl. Cryst.* **42**, 339–341.
- Jene, P. G., Pernin, C. G. & Ibers, J. A. (2001). *Acta Cryst.* **C57**, 730–734.
- Jia, S.-H., Ding, X., Yu, H.-T. & Han, B.-H. (2015). *RSC Adv.* **5**, 71095–71101.
- Lanni, L. M., Tilford, R. W., Bharathy, M. & Lavigne, J. J. (2011). *J. Am. Chem. Soc.* **133**, 13975–13983.
- Ma, H., Ren, H., Meng, S., Yan, Z., Zhao, H., Sun, F. & Zhu, G. (2013). *Chem. Commun.* **49**, 9773.
- Mukerji, D. N. (1922). *J. Chem. Soc. Trans.* **121**, 545–552.
- Nakabayashi, K. & Ohkoshi, S. (2009). *Inorg. Chem.* **48**, 8647–8649.
- Nishiyabu, R., Teraoka, S., Matsushima, Y. & Kubo, Y. (2012). *ChemPlusChem* **77**, 201–209.
- Niu, W., Smith, M. D. & Lavigne, J. J. (2006). *Cryst. Growth Des.* **6**, 1274–1277.
- Pandey, M. K., Kunchur, H. S., Ananthnag, G. S., Mague, J. T. & Balakrishna, M. S. (2019). *Dalton Trans.* **48**, 3610–3624.
- Park, S. W., Liao, Z., Ibarlucea, B., Qi, H., Lin, H. H., Becker, D., Melidone, J., Zhang, T., Sahabudeen, H., Baraban, L., Baek, C. K., Zheng, Z., Zschech, E., Fery, A., Heine, T., Kaiser, U., Cuniberti, G., Dong, R. & Feng, X. (2020). *Angew. Chem. Int. Ed.* **59**, 8218–8224.
- Poschmann, M. P. M., Reinsch, H. & Stock, N. (2021). *Z. Anorg. Allg. Chem.* **647**, 436–441.
- Rambo, B. M. & Lavigne, J. J. (2007). *Chem. Mater.* **19**, 3732–3739.

- Rigaku OD (2023). *CrysAlis PRO*. Rigaku Oxford Diffraction, Yarnton, England.
- Rondelli, M., Daranas, A. H. & Martín, T. (2023). *J. Org. Chem.* **88**, 2113–2121.
- Sheldrick, G. M. (2015a). *Acta Cryst.* **A71**, 3–8.
- Sheldrick, G. M. (2015b). *Acta Cryst.* **C71**, 3–8.
- Weider, P. R., Hegedus, L. S. & Asada, H. (1985). *J. Org. Chem.* **50**, 4276–4281.
- Wellala, N. P. N., Dong, H. T., Krause, J. A. & Guan, H. (2018). *Organometallics*, **37**, 4031–4039.
- Yuan, Y., Liu, J., Ren, H., Jing, X., Wang, W., Ma, H., Sun, F. & Zhao, H. (2012). *J. Mater. Res.* **27**, 1417–1420.

full crystallographic data

IUCrData (2024). **9**, x240612 [<https://doi.org/10.1107/S2414314624006126>]

Benzene-1,2,4,5-tetrol

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Benzene-1,2,4,5-tetrol

Crystal data

$C_6H_6O_4$

$M_r = 142.11$

Triclinic, $P\bar{1}$

$a = 3.7474$ (2) Å

$b = 11.6254$ (6) Å

$c = 13.7771$ (8) Å

$\alpha = 68.407$ (5)°

$\beta = 85.779$ (4)°

$\gamma = 89.843$ (4)°

$V = 556.37$ (6) Å³

$Z = 4$

$F(000) = 296$

$D_x = 1.697$ Mg m⁻³

Cu $K\alpha$ radiation, $\lambda = 1.54184$ Å

Cell parameters from 4683 reflections

$\theta = 3.4\text{--}75.6^\circ$

$\mu = 1.26$ mm⁻¹

$T = 120$ K

Block, colourless

$0.07 \times 0.05 \times 0.02$ mm

Data collection

XtalLAB PRO MM007, PILATUS3 R 200K diffractometer

Radiation source: rotating anode, MicroMax 007 HF

Mirror monochromator

Detector resolution: 5.8140 pixels mm⁻¹

ω scans

Absorption correction: gaussian (CrysAlisPro; Rigaku OD, 2023)

$T_{\min} = 0.927$, $T_{\max} = 1.000$

8096 measured reflections

2185 independent reflections

1842 reflections with $I > 2\sigma(I)$

$R_{\text{int}} = 0.063$

$\theta_{\max} = 76.2^\circ$, $\theta_{\min} = 3.5^\circ$

$h = -4 \rightarrow 4$

$k = -14 \rightarrow 14$

$l = -17 \rightarrow 17$

Refinement

Refinement on F^2

Least-squares matrix: full

$R[F^2 > 2\sigma(F^2)] = 0.045$

$wR(F^2) = 0.133$

$S = 1.09$

2185 reflections

205 parameters

8 restraints

Primary atom site location: dual

Secondary atom site location: difference Fourier map

Hydrogen site location: mixed

H atoms treated by a mixture of independent and constrained refinement

$w = 1/[\sigma^2(F_o^2) + (0.0799P)^2 + 0.1541P]$

where $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{\max} < 0.001$

$\Delta\rho_{\max} = 0.28$ e Å⁻³

$\Delta\rho_{\min} = -0.33$ e Å⁻³

Special details

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

Refinement. All hydrogen atoms were observed in the electron difference map. All hydroxy hydrogen atoms were refined with their O-H distances restrained to a target distance of 0.84 %A (DFIX). All other hydrogen atoms were geometrically placed and refined with a riding model.

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

	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{iso}}^*/U_{\text{eq}}$
C1A	−0.1468 (5)	1.09568 (17)	0.02598 (13)	0.0161 (4)
H1A	−0.249116	1.160936	0.043654	0.019*
C2A	−0.0561 (4)	0.98695 (17)	0.10428 (13)	0.0161 (4)
C3A	0.0876 (5)	0.89114 (17)	0.07847 (13)	0.0159 (4)
O4A	−0.1011 (3)	0.96812 (13)	0.20910 (9)	0.0204 (3)
H4A	−0.224 (6)	1.024 (2)	0.2205 (19)	0.031*
O5A	0.1865 (4)	0.78369 (12)	0.15524 (10)	0.0223 (3)
H5A	0.055 (6)	0.767 (2)	0.2104 (15)	0.033*
C1B	0.3702 (5)	0.38074 (17)	0.02176 (14)	0.0176 (4)
H1B	0.280395	0.299234	0.036481	0.021*
C2B	0.3851 (5)	0.42502 (17)	0.10209 (13)	0.0164 (4)
C3B	0.5136 (5)	0.54465 (17)	0.08035 (14)	0.0167 (4)
O4B	0.2720 (4)	0.34746 (13)	0.20260 (10)	0.0231 (3)
H4B	0.192 (7)	0.388 (2)	0.2403 (18)	0.035*
O5B	0.5337 (4)	0.58394 (13)	0.16233 (10)	0.0225 (3)
H5B	0.434 (7)	0.6521 (18)	0.1517 (19)	0.034*
C1C	0.5825 (5)	0.12616 (17)	0.45145 (13)	0.0168 (4)
H1C	0.639580	0.212043	0.418210	0.020*
C2C	0.4847 (5)	0.05791 (17)	0.39276 (13)	0.0167 (4)
C3C	0.4038 (5)	−0.06764 (17)	0.44063 (13)	0.0161 (4)
O4C	0.4713 (4)	0.10954 (12)	0.28510 (9)	0.0195 (3)
H4C	0.421 (6)	0.1868 (16)	0.2668 (18)	0.029*
O5C	0.3112 (3)	−0.13754 (12)	0.38383 (9)	0.0185 (3)
H5C	0.226 (6)	−0.093 (2)	0.3286 (14)	0.028*
C1D	1.0908 (4)	0.38787 (17)	0.49271 (13)	0.0159 (4)
H1D	1.153969	0.310947	0.487689	0.019*
C2D	0.9455 (4)	0.47869 (17)	0.40886 (13)	0.0149 (4)
C3D	0.8562 (4)	0.59139 (17)	0.41683 (13)	0.0151 (4)
O4D	0.8906 (4)	0.45066 (13)	0.32286 (9)	0.0202 (3)
H4D	0.773 (6)	0.509 (2)	0.2812 (17)	0.030*
O5D	0.7198 (3)	0.68082 (12)	0.33076 (9)	0.0180 (3)
H5D	0.611 (6)	0.7358 (19)	0.3477 (18)	0.027*

Atomic displacement parameters (\AA^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1A	0.0121 (8)	0.0164 (9)	0.0193 (9)	0.0051 (7)	−0.0046 (7)	−0.0052 (7)

C2A	0.0106 (8)	0.0201 (10)	0.0156 (8)	0.0037 (7)	-0.0036 (6)	-0.0039 (7)
C3A	0.0126 (8)	0.0159 (9)	0.0153 (8)	0.0037 (7)	-0.0049 (6)	-0.0003 (7)
O4A	0.0238 (7)	0.0217 (7)	0.0146 (6)	0.0096 (6)	-0.0051 (5)	-0.0046 (5)
O5A	0.0267 (7)	0.0185 (7)	0.0153 (6)	0.0120 (6)	-0.0023 (5)	0.0012 (5)
C1B	0.0142 (8)	0.0154 (9)	0.0215 (9)	0.0067 (7)	-0.0072 (7)	-0.0036 (7)
C2B	0.0128 (8)	0.0166 (9)	0.0162 (8)	0.0056 (7)	-0.0055 (7)	-0.0009 (7)
C3B	0.0136 (8)	0.0191 (9)	0.0183 (8)	0.0092 (7)	-0.0096 (7)	-0.0065 (7)
O4B	0.0302 (8)	0.0182 (7)	0.0171 (6)	0.0073 (6)	-0.0010 (5)	-0.0022 (5)
O5B	0.0309 (8)	0.0196 (7)	0.0195 (7)	0.0136 (6)	-0.0128 (6)	-0.0083 (6)
C1C	0.0143 (8)	0.0162 (9)	0.0172 (8)	0.0063 (7)	-0.0036 (7)	-0.0024 (7)
C2C	0.0130 (8)	0.0194 (10)	0.0133 (8)	0.0072 (7)	-0.0039 (6)	-0.0005 (7)
C3C	0.0119 (8)	0.0176 (9)	0.0171 (8)	0.0058 (7)	-0.0040 (6)	-0.0041 (7)
O4C	0.0264 (7)	0.0157 (7)	0.0138 (6)	0.0074 (6)	-0.0068 (5)	-0.0013 (5)
O5C	0.0210 (7)	0.0175 (7)	0.0154 (6)	0.0051 (6)	-0.0080 (5)	-0.0032 (5)
C1D	0.0123 (8)	0.0145 (9)	0.0183 (8)	0.0043 (7)	-0.0036 (7)	-0.0026 (7)
C2D	0.0103 (8)	0.0180 (9)	0.0148 (8)	0.0026 (7)	-0.0033 (6)	-0.0037 (7)
C3D	0.0120 (8)	0.0144 (9)	0.0150 (8)	0.0041 (7)	-0.0045 (6)	0.0000 (7)
O4D	0.0237 (7)	0.0210 (7)	0.0164 (6)	0.0107 (6)	-0.0094 (5)	-0.0062 (5)
O5D	0.0194 (6)	0.0171 (7)	0.0148 (6)	0.0092 (5)	-0.0075 (5)	-0.0015 (5)

Geometric parameters (Å, °)

C1A—H1A	0.9500	C1C—H1C	0.9500
C1A—C2A	1.388 (2)	C1C—C2C	1.391 (3)
C1A—C3A ⁱ	1.391 (2)	C1C—C3C ⁱⁱⁱ	1.394 (2)
C2A—C3A	1.385 (3)	C2C—C3C	1.386 (3)
C2A—O4A	1.376 (2)	C2C—O4C	1.385 (2)
C3A—O5A	1.378 (2)	C3C—O5C	1.379 (2)
O4A—H4A	0.852 (17)	O4C—H4C	0.863 (17)
O5A—H5A	0.835 (17)	O5C—H5C	0.832 (16)
C1B—H1B	0.9500	C1D—H1D	0.9500
C1B—C2B	1.386 (2)	C1D—C2D	1.392 (2)
C1B—C3B ⁱⁱ	1.391 (3)	C1D—C3D ^{iv}	1.382 (2)
C2B—C3B	1.390 (3)	C2D—C3D	1.392 (3)
C2B—O4B	1.381 (2)	C2D—O4D	1.368 (2)
C3B—O5B	1.372 (2)	C3D—O5D	1.3881 (19)
O4B—H4B	0.861 (16)	O4D—H4D	0.858 (16)
O5B—H5B	0.843 (17)	O5D—H5D	0.851 (16)
C2A—C1A—H1A	120.1	C2C—C1C—H1C	120.2
C2A—C1A—C3A ⁱ	119.82 (17)	C2C—C1C—C3C ⁱⁱⁱ	119.53 (18)
C3A ⁱ —C1A—H1A	120.1	C3C ⁱⁱⁱ —C1C—H1C	120.2
C3A—C2A—C1A	120.03 (16)	C3C—C2C—C1C	120.55 (16)
O4A—C2A—C1A	123.07 (16)	O4C—C2C—C1C	122.61 (17)
O4A—C2A—C3A	116.90 (15)	O4C—C2C—C3C	116.82 (16)
C2A—C3A—C1A ⁱ	120.14 (16)	C2C—C3C—C1C ⁱⁱⁱ	119.92 (17)
O5A—C3A—C1A ⁱ	119.11 (16)	O5C—C3C—C1C ⁱⁱⁱ	118.51 (17)
O5A—C3A—C2A	120.70 (15)	O5C—C3C—C2C	121.57 (15)

C2A—O4A—H4A	112.3 (16)	C2C—O4C—H4C	109.5 (15)
C3A—O5A—H5A	111.3 (17)	C3C—O5C—H5C	110.6 (17)
C2B—C1B—H1B	119.9	C2D—C1D—H1D	119.7
C2B—C1B—C3B ⁱⁱ	120.17 (18)	C3D ^{iv} —C1D—H1D	119.7
C3B ⁱⁱ —C1B—H1B	119.9	C3D ^{iv} —C1D—C2D	120.58 (17)
C1B—C2B—C3B	119.88 (17)	C1D—C2D—C3D	119.20 (16)
O4B—C2B—C1B	118.40 (17)	O4D—C2D—C1D	117.50 (16)
O4B—C2B—C3B	121.71 (16)	O4D—C2D—C3D	123.28 (15)
C2B—C3B—C1B ⁱⁱ	119.95 (17)	C1D ^{iv} —C3D—C2D	120.22 (15)
O5B—C3B—C1B ⁱⁱ	121.84 (18)	C1D ^{iv} —C3D—O5D	122.17 (16)
O5B—C3B—C2B	118.17 (16)	O5D—C3D—C2D	117.60 (15)
C2B—O4B—H4B	111.8 (18)	C2D—O4D—H4D	108.3 (17)
C3B—O5B—H5B	112.5 (16)	C3D—O5D—H5D	111.5 (16)
C1A—C2A—C3A—C1A ⁱ	1.1 (3)	C1C—C2C—C3C—C1C ⁱⁱⁱ	0.5 (3)
C1A—C2A—C3A—O5A	178.53 (16)	C1C—C2C—C3C—O5C	-179.04 (15)
C3A ⁱ —C1A—C2A—C3A	-1.0 (3)	C3C ⁱⁱⁱ —C1C—C2C—C3C	-0.5 (3)
C3A ⁱ —C1A—C2A—O4A	178.86 (16)	C3C ⁱⁱⁱ —C1C—C2C—O4C	-178.89 (15)
O4A—C2A—C3A—C1A ⁱ	-178.86 (16)	O4C—C2C—C3C—C1C ⁱⁱⁱ	178.98 (15)
O4A—C2A—C3A—O5A	-1.4 (3)	O4C—C2C—C3C—O5C	-0.6 (2)
C1B—C2B—C3B—C1B ⁱⁱ	-0.5 (3)	C1D—C2D—C3D—C1D ^{iv}	-0.4 (3)
C1B—C2B—C3B—O5B	-178.19 (14)	C1D—C2D—C3D—O5D	178.51 (15)
C3B ⁱⁱ —C1B—C2B—C3B	0.5 (3)	C3D ^{iv} —C1D—C2D—C3D	0.4 (3)
C3B ⁱⁱ —C1B—C2B—O4B	-179.07 (15)	C3D ^{iv} —C1D—C2D—O4D	-178.16 (16)
O4B—C2B—C3B—C1B ⁱⁱ	179.06 (15)	O4D—C2D—C3D—C1D ^{iv}	178.07 (17)
O4B—C2B—C3B—O5B	1.4 (2)	O4D—C2D—C3D—O5D	-3.0 (3)

Symmetry codes: (i) $-x, -y+2, -z$; (ii) $-x+1, -y+1, -z$; (iii) $-x+1, -y, -z+1$; (iv) $-x+2, -y+1, -z+1$.

Hydrogen-bond geometry (\AA , $^\circ$)

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
O4A—H4A \cdots O4C ^v	0.85 (2)	1.89 (2)	2.715 (2)	163 (2)
O4B—H4B \cdots O4D ^{vi}	0.86 (2)	1.88 (2)	2.708 (2)	163 (3)
O4B—H4B \cdots O5B	0.86 (2)	2.45 (2)	2.764 (2)	102 (2)
O4C—H4C \cdots O4B	0.86 (2)	1.85 (2)	2.702 (2)	167 (2)
O4D—H4D \cdots O5B	0.86 (2)	1.85 (2)	2.6425 (19)	154 (2)
O4D—H4D \cdots O5D	0.86 (2)	2.34 (2)	2.789 (2)	113 (2)
O5A—H5A \cdots O4A	0.83 (2)	2.40 (2)	2.711 (2)	103 (2)
O5A—H5A \cdots O5D ^{vi}	0.83 (2)	1.95 (2)	2.7562 (18)	162 (2)
O5B—H5B \cdots O5A	0.84 (2)	1.80 (2)	2.633 (2)	169 (2)
O5C—H5C \cdots O4A ^{vii}	0.83 (2)	2.04 (2)	2.8376 (16)	161 (2)
O5C—H5C \cdots O4C	0.83 (2)	2.38 (2)	2.734 (2)	107 (2)
O5D—H5D \cdots O5C ^{viii}	0.85 (2)	2.03 (2)	2.8796 (19)	175 (2)

Symmetry codes: (v) $x-1, y+1, z$; (vi) $x-1, y, z$; (vii) $x, y-1, z$; (viii) $x, y+1, z$.