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Benzene-1,3,5-triol at 105 K

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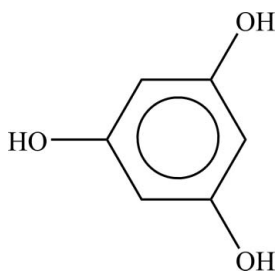
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 Key indicators: single-crystal X-ray study; $T = 105$ K; mean $\sigma(\text{C}-\text{C}) = 0.002$ Å;
 R factor = 0.026; wR factor = 0.081; data-to-parameter ratio = 8.2.

The structure of the title compound, $\text{C}_6\text{H}_6\text{O}_3$, has been redetermined at low temperature [room-temperature structure: Maartmann-Moe (1965). *Acta Cryst.* **19**, 155–157]. The molecule is planar with approximate D_{3h} point symmetry, yet it crystallizes in the chiral orthorhombic space group $P2_12_12_1$ with a three-dimensional hydrogen-bonding network containing infinite $\text{O}-\text{H}\cdots\text{O}-\text{H}\cdots\text{O}-\text{H}$ chains.

Related literature

For the structure at room temperature, see: Maartmann-Moe (1965). For the hydrate structure, see: Wallwork & Powell (1957).



Experimental

Crystal data

$\text{C}_6\text{H}_6\text{O}_3$	$V = 556.35$ (4) Å ³
$M_r = 126.11$	$Z = 4$
Orthorhombic, $P2_12_12_1$	Mo $K\alpha$ radiation
$a = 4.7778$ (2) Å	$\mu = 0.12$ mm ⁻¹
$b = 9.3581$ (4) Å	$T = 105$ (2) K
$c = 12.4433$ (6) Å	$0.20 \times 0.08 \times 0.05$ mm

Data collection

Siemens SMART CCD diffractometer	6178 measured reflections
Absorption correction: multi-scan (SADABS; Sheldrick, 1996)	743 independent reflections
$T_{\min} = 0.916$, $T_{\max} = 0.997$	728 reflections with $I > 2\sigma(I)$
	$R_{\text{int}} = 0.014$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.026$	H atoms treated by a mixture of independent and constrained refinement
$wR(F^2) = 0.081$	$\Delta\rho_{\text{max}} = 0.27$ e Å ⁻³
$S = 1.13$	$\Delta\rho_{\text{min}} = -0.20$ e Å ⁻³
743 reflections	
91 parameters	

Table 1

Hydrogen-bond geometry (Å, °).

$D-\text{H}\cdots A$	$D-\text{H}$	$\text{H}\cdots A$	$D\cdots A$	$D-\text{H}\cdots A$
$\text{O1}-\text{H1}\cdots\text{O3}^{\text{i}}$	0.83 (2)	1.94 (2)	2.7426 (13)	164 (2)
$\text{O2}-\text{H2}\cdots\text{O1}^{\text{ii}}$	0.79 (2)	1.97 (2)	2.7424 (14)	169 (2)
$\text{O3}-\text{H3}\cdots\text{O2}^{\text{iii}}$	0.86 (2)	1.85 (2)	2.7086 (16)	173.3 (17)

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

Data collection: *SMART* (Bruker, 1998); cell refinement: *SAINT-Plus* (Bruker, 2001); data reduction: *SAINT-Plus*; program(s) used to solve structure: *SHELXTL* (Sheldrick, 2008); program(s) used to refine structure: *SHELXTL*; molecular graphics: *SHELXTL*; software used to prepare material for publication: *SHELXTL*.

The purchase of the diffractometer was made possible through support from the Research Council of Norway (NFR).

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: BI2305).

References

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supporting information

Acta Cryst. (2008). E64, o2023 [doi:10.1107/S1600536808030638]

Benzene-1,3,5-triol at 105 K

Carl Henrik Görbitz, Massoud Kaboli, Matthew Lovell Read and Kristian Vestli

S1. Comment

The structure of the benzene-1,3,5-triol, commonly known as phloroglucinol, is shown in Fig. 1. The molecule is essentially planar, with D_{3h} point symmetry, having only small out-of-plane rotations for the hydroxyl groups. Rather than forming a layer-like structure, a folded molecular aggregation pattern is observed in the crystal (Fig. 2) giving a three-dimensional hydrogen-bonding pattern. The three hydrogen bonds listed in Table 1 form an infinite zigzag chain along the b axis as shown in Fig. 3. The agreement with the original structure determination (Maartmann-Moe, 1965) is generally good, but with some significant changes in the hydrogen bonding geometries.

Benzene-1,3,5-triol has also been crystallized as a dihydrate, which is divided into layers with water molecules as connectors (Wallwork & Powell, 1957).

S2. Experimental

The title compound was obtained from Fluka. Crystals were grown by diffusion of hexane into 30 μl of a solution containing 2.1 mg benzene-1,3,5-triol and 1.3 mg triazin in 3-methyl-2-butanone.

S3. Refinement

Positional parameters were refined for hydroxylic H atoms, while H atoms bonded to C were positioned with idealized geometry and C—H distance 0.95 Å. U_{iso} values were $1.5U_{\text{eq}}(\text{O})$ and $1.2U_{\text{eq}}(\text{C})$. In the absence of significant anomalous scattering effects, 1585 Friedel pairs were merged.

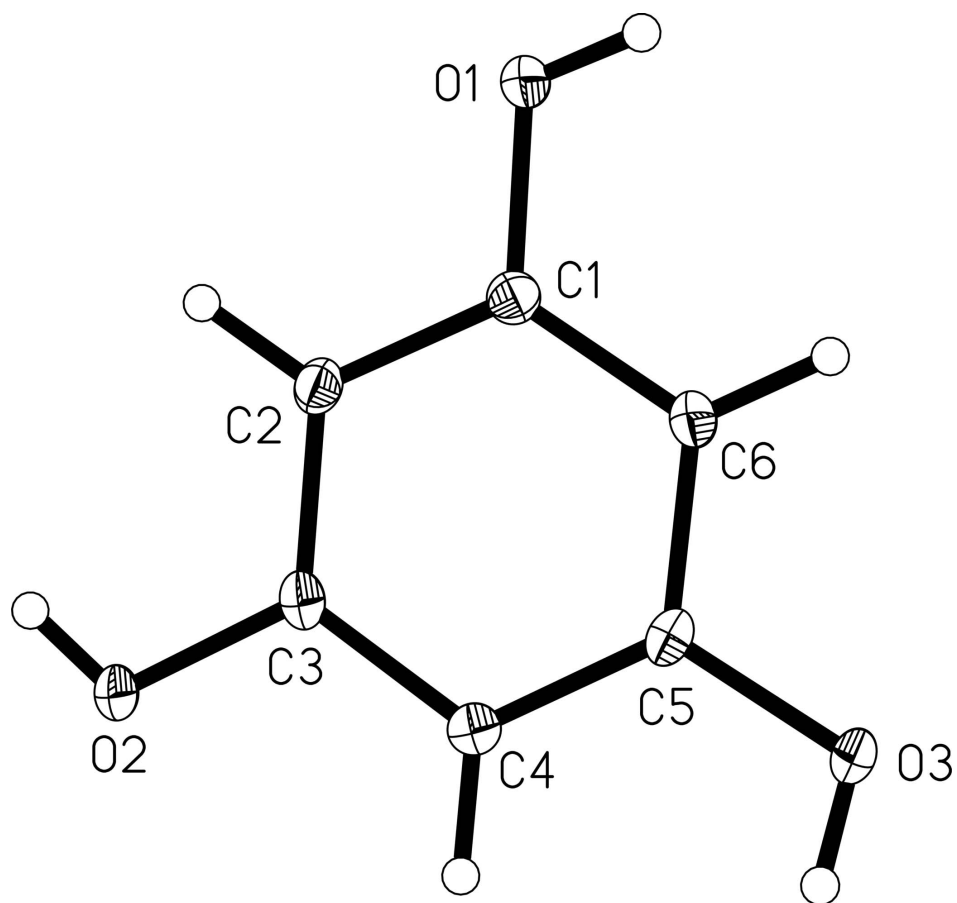


Figure 1

The molecular structure of the title compound. Displacement ellipsoids are shown at the 50% probability level and H-atoms are shown as spheres of arbitrary size.

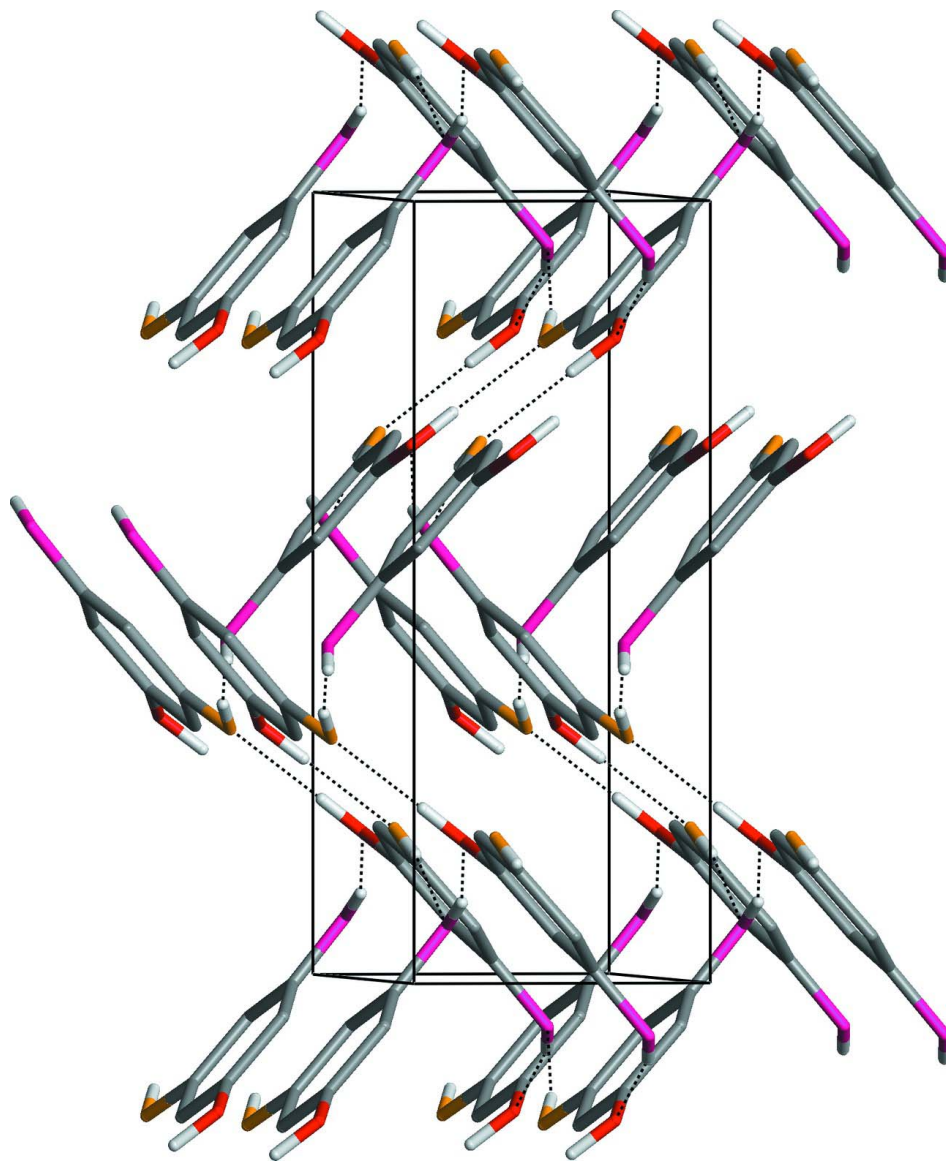
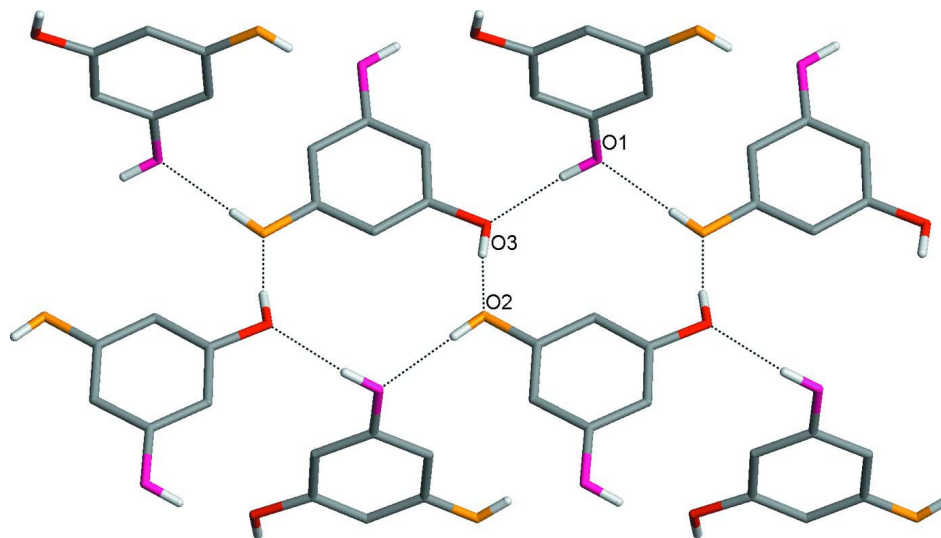


Figure 2

Molecular packing and unit cell of the title compound viewed along the *b* axis. Hydrogen bonding is indicated by dashed lines, H-atoms bonded to C have been omitted for clarity. The three different hydroxylic O atoms have been depicted in different colours.

**Figure 3**

Detail of the hydrogen bonding pattern showing infinite hydrogen-bonded chains.

benzene-1,3,5-triol

Crystal data

$C_6H_6O_3$

$M_r = 126.11$

Orthorhombic, $P2_12_12_1$

Hall symbol: P 2ac 2ab

$a = 4.7778$ (2) Å

$b = 9.3581$ (4) Å

$c = 12.4433$ (6) Å

$V = 556.35$ (4) Å³

$Z = 4$

$F(000) = 264$

$D_x = 1.506$ Mg m⁻³

Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å

Cell parameters from 4652 reflections

$\theta = 2.7$ – 27.1°

$\mu = 0.12$ mm⁻¹

$T = 105$ K

Needle, colourless

$0.20 \times 0.08 \times 0.05$ mm

Data collection

Siemens SMART CCD
diffractometer

Radiation source: fine-focus sealed tube

Graphite monochromator

ω scans

Absorption correction: multi-scan
(*SADABS*; Sheldrick, 1996)

$T_{\min} = 0.916$, $T_{\max} = 0.997$

6178 measured reflections

743 independent reflections

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

$R_{\text{int}} = 0.014$

$\theta_{\max} = 27.1^\circ$, $\theta_{\min} = 2.7^\circ$

$h = -6 \rightarrow 6$

$k = -11 \rightarrow 11$

$l = -15 \rightarrow 15$

Refinement

Refinement on F^2

Least-squares matrix: full

$R[F^2 > 2\sigma(F^2)] = 0.027$

$wR(F^2) = 0.081$

$S = 1.13$

743 reflections

91 parameters

0 restraints

Primary atom site location: structure-invariant
direct methods

Secondary atom site location: difference Fourier
map

Hydrogen site location: inferred from
neighbouring sites

H atoms treated by a mixture of independent
and constrained refinement

$$w = 1/[\sigma^2(F_o^2) + (0.054P)^2 + 0.1017P]$$

where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{\max} < 0.001$

$$\Delta\rho_{\max} = 0.27 \text{ e } \text{\AA}^{-3}$$

$$\Delta\rho_{\min} = -0.20 \text{ e } \text{\AA}^{-3}$$

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. Data were collected by measuring three sets of exposures with the detector set at $2\theta = 29^\circ$, crystal-to-detector distance 5.00 cm. Refinement of F^2 against ALL reflections.

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

	x	y	z	$U_{\text{iso}}^*/U_{\text{eq}}$
O1	0.2140 (2)	0.48267 (10)	0.56722 (8)	0.0160 (3)
H1	0.189 (5)	0.560 (2)	0.5979 (15)	0.024*
O2	0.8083 (3)	0.25657 (10)	0.31075 (8)	0.0167 (3)
H2	0.760 (5)	0.189 (2)	0.3432 (14)	0.025*
O3	0.7415 (2)	0.76483 (10)	0.32139 (8)	0.0160 (3)
H3	0.884 (5)	0.7549 (19)	0.2792 (16)	0.024*
C1	0.3967 (3)	0.49437 (14)	0.48167 (11)	0.0135 (3)
C2	0.5004 (3)	0.36808 (13)	0.43819 (11)	0.0141 (3)
H21	0.4423	0.2779	0.4652	0.017*
C3	0.6913 (3)	0.37730 (13)	0.35413 (10)	0.0135 (3)
C4	0.7758 (3)	0.50800 (15)	0.31202 (11)	0.0150 (3)
H41	0.9054	0.5126	0.2542	0.018*
C5	0.6651 (3)	0.63152 (13)	0.35689 (10)	0.0134 (3)
C6	0.4732 (3)	0.62700 (13)	0.44129 (11)	0.0137 (3)
H61	0.3970	0.7124	0.4704	0.016*

Atomic displacement parameters (\AA^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.0199 (5)	0.0107 (5)	0.0173 (5)	-0.0003 (4)	0.0057 (4)	-0.0006 (3)
O2	0.0217 (5)	0.0090 (5)	0.0194 (5)	0.0017 (4)	0.0058 (4)	0.0000 (4)
O3	0.0209 (6)	0.0093 (5)	0.0177 (5)	-0.0015 (4)	0.0054 (5)	0.0009 (3)
C1	0.0127 (6)	0.0142 (6)	0.0136 (6)	-0.0006 (6)	-0.0006 (5)	-0.0004 (5)
C2	0.0152 (6)	0.0109 (6)	0.0161 (6)	-0.0009 (5)	-0.0003 (6)	0.0017 (5)
C3	0.0142 (6)	0.0108 (6)	0.0154 (6)	0.0014 (6)	-0.0013 (6)	-0.0012 (5)
C4	0.0158 (6)	0.0141 (6)	0.0150 (6)	0.0000 (5)	0.0032 (5)	0.0003 (5)
C5	0.0152 (7)	0.0104 (6)	0.0145 (6)	-0.0017 (6)	-0.0014 (6)	0.0012 (5)
C6	0.0151 (6)	0.0110 (6)	0.0152 (6)	0.0005 (5)	0.0008 (6)	-0.0020 (5)

Geometric parameters (\AA , $^\circ$)

O1—C1	1.3808 (17)	C2—C3	1.3905 (19)
O1—H1	0.83 (2)	C2—H21	0.9500
O2—C3	1.3712 (16)	C3—C4	1.3905 (18)

O2—H2	0.79 (2)	C4—C5	1.3884 (19)
O3—C5	1.3730 (15)	C4—H41	0.9500
O3—H3	0.86 (2)	C5—C6	1.3945 (19)
C1—C6	1.3881 (17)	C6—H61	0.9500
C1—C2	1.3910 (18)		
C1—O1—H1	112.2 (14)	C2—C3—C4	121.90 (12)
C3—O2—H2	109.9 (14)	C5—C4—C3	118.05 (12)
C5—O3—H3	107.9 (12)	C5—C4—H41	121.0
O1—C1—C6	121.10 (11)	C3—C4—H41	121.0
O1—C1—C2	117.24 (11)	O3—C5—C4	121.72 (12)
C6—C1—C2	121.67 (12)	O3—C5—C6	116.41 (11)
C3—C2—C1	118.27 (12)	C4—C5—C6	121.87 (12)
C3—C2—H21	120.9	C1—C6—C5	118.22 (12)
C1—C2—H21	120.9	C1—C6—H61	120.9
O2—C3—C2	120.83 (12)	C5—C6—H61	120.9
O2—C3—C4	117.26 (12)		
O1—C1—C2—C3	-178.06 (12)	O1—C1—C6—C5	178.09 (12)
C6—C1—C2—C3	1.8 (2)	C2—C1—C6—C5	-1.8 (2)
C1—C2—C3—O2	177.59 (12)	O3—C5—C6—C1	-178.14 (12)
C1—C2—C3—C4	-1.1 (2)	C4—C5—C6—C1	1.0 (2)
O2—C3—C4—C5	-178.32 (13)	H1—O1—C1—C6	-13.0 (16)
C2—C3—C4—C5	0.4 (2)	H2—O2—C3—C2	-4.3 (16)
C3—C4—C5—O3	178.75 (13)	H3—O3—C5—C4	-10.8 (14)
C3—C4—C5—C6	-0.4 (2)		

Hydrogen-bond geometry (Å, °)

<i>D—H...A</i>	<i>D—H</i>	<i>H...A</i>	<i>D...A</i>	<i>D—H...A</i>
O1—H1...O3 ⁱ	0.83 (2)	1.94 (2)	2.7426 (13)	164 (2)
O2—H2...O1 ⁱⁱ	0.79 (2)	1.97 (2)	2.7424 (14)	169 (2)
O3—H3...O2 ⁱⁱⁱ	0.86 (2)	1.85 (2)	2.7086 (16)	173.3 (17)

Symmetry codes: (i) $x-1/2, -y+3/2, -z+1$; (ii) $x+1/2, -y+1/2, -z+1$; (iii) $-x+2, y+1/2, -z+1/2$.