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2,4,6-Trihydroxyanisole

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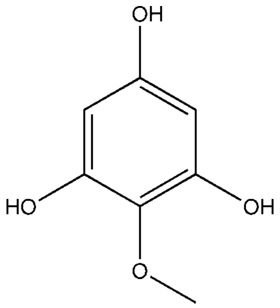
Key indicators: single-crystal X-ray study; T = 298 K; mean σ(C–C) = 0.004 Å; R factor = 0.047; wR factor = 0.125; data-to-parameter ratio = 7.7.

The title compound, $C_7H_8O_4$, crystallizes with three intermolecular O—H$\cdots$O hydrogen bonds, yielding a structure with a tightly knit pattern of hydrogen bonding.

Related literature

For related literature, see: Baker *et al.* (1970); Damschroder & Shriner (1937); De Laire & Tiemann (1893).



Experimental

Crystal data

$C_7H_8O_4$
M_r = 156.13
Orthorhombic, $P2_12_12_1$
a = 5.2310 (10) Å
b = 9.4010 (19) Å
c = 15.095 (3) Å
V = 742.3 (3) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.12 mm^{-1}
T = 298 (2) K
0.30 × 0.20 × 0.20 mm

Data collection

Bruker SMART APEX diffractometer
Absorption correction: none
881 measured reflections
861 independent reflections
736 reflections with $I > 2\sigma(I)$
$R_{\rm int}$ = 0.041

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.047
$wR(F^2)$ = 0.125
S = 1.08
861 reflections
114 parameters
1 restraint
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{\rm max}$ = 0.20 e Å^{-3}
$\Delta\rho_{\rm min}$ = −0.25 e Å^{-3}

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

D—H$\cdots A$	D—H	H$\cdots A$	$D\cdots A$	D—H$\cdots A$
O4—H4$A\cdots$O1$^{\rm i}$	0.89 (4)	1.93 (4)	2.823 (3)	177 (4)
O3—H3$\cdots$O4$^{\rm ii}$	0.92 (4)	1.83 (4)	2.743 (3)	172 (4)
O1—H1$\cdots$O3$^{\rm iii}$	0.892 (18)	1.889 (19)	2.778 (3)	174 (5)

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

Data collection: *SMART* (Bruker, 1998); cell refinement: *SMART*; data reduction: *SAINT* (Bruker, 1998); program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997*a*); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997*a*); molecular graphics: *SHELXTL* (Sheldrick, 1997*b*); software used to prepare material for publication: *SHELXL97*.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: OM2124).

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supplementary materials

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2,4,6-Trihydroxyanisole

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Comment

The title compound, (I), 2,4,6-trihydroxyanisole, was first isolated by De Laire & Tiemann (1893) as a degradation product of a glucoside obtained from Iris Jorentina. Although it is a well known intermediate for synthesis of several natural isoflavones such as tectorigenin, irigenin and caviunin (Baker *et al.*, 1970), its crystal structure has not been reported. In this article, the title comound was prepared by the method of Damschroder & Shriner (1937) with appropriate modifications.

The molecular structure is shown in Fig. 1. A l l the hydrogen atoms of the hydroxyl groups contribute to the formation of intermolecular O—H···O hydrogen bonds (Table 1), and every molecule is involved in hydrogen bond interactions with six other molecules, which leads to the formation of a three-dimensional network (Fig. 2). To our surprise, O2 was not involved in any hydrogen bond interactions. This may be due to steric hindrance around the oxygen atom. The methyl group (C7) is 1.084 (5) Å out of the plane of the benzene ring which prevents close stacking of the molecules (Fig. 3). Face to face distances between benzene rings are 4.482 (3) Å).

Experimental

A solution of 3.7 g of 2,4,6-trinitroanisole in 70 ml of absolute ethanol was reduced with hydrogen at a pressure of 3 atm in the presence of platinum oxide (0.1 g). After ten minutes, the reduction mixture was filtered quickly, and the solvent removed with a water pump, at the same time slowly flowing $N_2(g)$ through the solution. The mixture was then cooled in an ice bath, the product removed by filtration and washed with small amounts of cold absolute ethanol. 2,4,6-Triaminoanisole (1.77 g, 76%) was obtained by recrystallization from hot absolute ethanol. A solution of 1.5 g of the crude triaminoanisole in 60 ml of air-free water containing 3 ml of concentrated hydrochloric acid was refluxed for 20 h in an atmosphere of carbon dioxide. After filtration, the water was removed by vacuum distillation, passing a stream of carbon dioxide. The dry residue was then thoroughly extracted with three 60 ml portions of ether. The combined ether extracts were dried over magnesium sulfate, and the ether was removed by distillation. 2,4,6-trihydroxyanisole was obtained in 65% yield. The purified products were dissolved in ethyl acetate, and petroleum ether was added carefully. The crystals suitable for X-ray analysis were obtained after 3 days by volatilization of the solvents.

Refinement

H atoms bonded to C were placed in geometrically idealized positions and constrained to ride on their parent atoms, with $U_{iso}(H) = 1.2U_{eq}(C)$ and with C—H = 0.93 Å for the aromatic H atoms, C—H = 0.96 Å for the aliphatic H atoms. H atoms bonded to O were located from adifference electron density map and refined with a restrained O—H distance of 0.92 (2) Å.

Figures

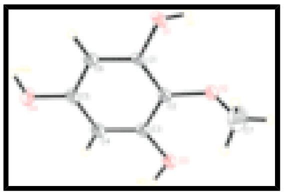

Fig. 1. The structure of 2,4,6-trihydroxyanisole, showing 30% probability displacement ellipsoids and the atom-numbering scheme.

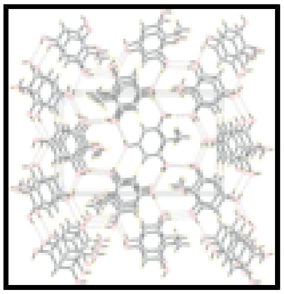

Fig. 2. The crystal packing of 2,4,6-trihydroxyanisole, viewed along the *a* axis. Dashed lines indicate O—H···O hydrogen bonds.

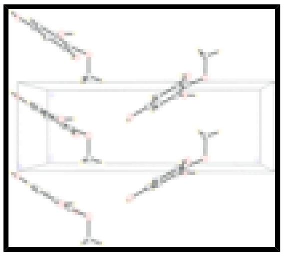

Fig. 3. The crystal packing of 2,4,6-trihydroxyanisole, viewed along the *b* axis.

2,4,6-Trihydroxyanisole

Crystal data

$C_7H_8O_4$	F_{000} = 328
M_r = 156.13	D_x = 1.397 Mg m^{-3}
Orthorhombic, $P2_12_12_1$	Mo *K*α radiation λ = 0.71073 Å
Hall symbol: P 2ac 2ab	Cell parameters from 558 reflections
a = 5.2310 (10) Å	θ = 2.8–26.2°
b = 9.4010 (19) Å	μ = 0.12 mm^{-1}
c = 15.095 (3) Å	*T* = 298 (2) K
V = 742.3 (3) Å^3	Block, colourless
Z = 4	0.30 × 0.20 × 0.20 mm

Data collection

Bruker SMART APEX diffractometer	736 reflections with $I > 2\sigma(I)$
Radiation source: fine-focus sealed tube	R_{int} = 0.041
Monochromator: graphite	θ_{max} = 26.0°
T = 298(2) K	θ_{min} = 2.6°
φ and ω scans	*h* = 0→6

Absorption correction: none	$k = 0\rightarrow11$
881 measured reflections	$l = 0\rightarrow18$
861 independent reflections	

Refinement

Refinement on F^2	H atoms treated by a mixture of independent and constrained refinement
Least-squares matrix: full	$w = 1/[\sigma^2(F_o^2) + (0.077P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$R[F^2 > 2\sigma(F^2)] = 0.047$	$(\Delta/\sigma)_{max} < 0.001$
$wR(F^2) = 0.125$	$\Delta\rho_{max} = 0.20$ e Å^{-3}
$S = 1.08$	$\Delta\rho_{min} = -0.25$ e Å^{-3}
861 reflections	Extinction correction: SHELXL97 (Sheldrick, 1997a), $Fc^* = kFc[1+0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$
114 parameters	Extinction coefficient: 0.056 (11)
1 restraint	Absolute structure: indeterminate
Primary atom site location: structure-invariant direct methods	Flack parameter: ?
Secondary atom site location: difference Fourier map	Rogers parameter: ?
Hydrogen site location: inferred from neighbouring sites	

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 $F^2 > 2\text{sigma}(F^2)$ 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}
O3	1.0972 (6)	0.7137 (2)	0.66584 (13)	0.0401 (7)
O2	1.1104 (4)	0.4545 (2)	0.74132 (13)	0.0318 (6)
O1	0.9108 (6)	0.2206 (2)	0.65022 (13)	0.0378 (7)
O4	0.6225 (6)	0.5306 (2)	0.42132 (14)	0.0474 (8)
C6	0.7719 (7)	0.3719 (3)	0.53482 (19)	0.0328 (8)
H6	0.7001	0.2941	0.5061	0.039*
C4	0.8600 (7)	0.6234 (3)	0.5401 (2)	0.0337 (8)
H4	0.8463	0.7134	0.5150	0.040*
C1	0.8988 (7)	0.3553 (3)	0.61421 (18)	0.0276 (7)
C3	0.9875 (7)	0.6045 (3)	0.6196 (2)	0.0293 (7)

C2	1.0050 (6)	0.4702 (3)	0.65761 (18)	0.0267 (7)
C5	0.7536 (8)	0.5070 (3)	0.4988 (2)	0.0326 (7)
C7	1.3791 (9)	0.4384 (6)	0.7426 (3)	0.0679 (13)
H7A	1.4238	0.3467	0.7193	0.102*
H7B	1.4398	0.4464	0.8024	0.102*
H7C	1.4562	0.5113	0.7069	0.102*
H3	1.094 (10)	0.796 (5)	0.633 (2)	0.071 (14)*
H4A	0.560 (8)	0.449 (4)	0.400 (2)	0.062 (13)*
H1	0.902 (10)	0.224 (5)	0.7092 (12)	0.070 (14)*

Atomic displacement parameters ($Å^2$)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O3	0.0739 (17)	0.0161 (10)	0.0303 (11)	−0.0086 (13)	−0.0086 (14)	0.0010 (9)
O2	0.0438 (12)	0.0274 (11)	0.0242 (10)	−0.0019 (11)	−0.0028 (11)	0.0025 (9)
O1	0.0757 (17)	0.0119 (9)	0.0256 (10)	0.0040 (12)	−0.0020 (14)	0.0028 (8)
O4	0.085 (2)	0.0186 (10)	0.0383 (13)	−0.0033 (14)	−0.0263 (15)	0.0025 (10)
C6	0.055 (2)	0.0145 (13)	0.0291 (15)	−0.0028 (15)	−0.0009 (17)	−0.0018 (12)
C4	0.055 (2)	0.0133 (12)	0.0329 (15)	0.0004 (16)	−0.0041 (16)	0.0030 (12)
C1	0.0449 (17)	0.0113 (12)	0.0267 (13)	0.0045 (14)	0.0025 (15)	0.0035 (11)
C3	0.0455 (17)	0.0142 (13)	0.0280 (15)	−0.0026 (14)	0.0008 (15)	−0.0023 (11)
C2	0.0399 (16)	0.0186 (14)	0.0215 (13)	0.0010 (14)	0.0034 (14)	−0.0008 (12)
C5	0.0491 (18)	0.0220 (14)	0.0266 (14)	0.0010 (13)	−0.0073 (16)	0.0015 (11)
C7	0.053 (2)	0.089 (3)	0.061 (3)	0.010 (3)	−0.009 (3)	0.003 (3)

Geometric parameters (Å, °)

O3—C3	1.368 (4)	C6—H6	0.9300
O3—H3	0.92 (4)	C4—C5	1.377 (4)
O2—C2	1.386 (3)	C4—C3	1.384 (4)
O2—C7	1.414 (5)	C4—H4	0.9300
O1—C1	1.379 (3)	C1—C2	1.381 (4)
O1—H1	0.892 (18)	C3—C2	1.390 (4)
O4—C5	1.374 (4)	C7—H7A	0.9600
O4—H4A	0.89 (4)	C7—H7B	0.9600
C6—C1	1.379 (4)	C7—H7C	0.9600
C6—C5	1.384 (4)		

C3—O3—H3	110 (3)	O3—C3—C2	116.3 (3)
C2—O2—C7	114.8 (3)	C4—C3—C2	120.5 (3)
C1—O1—H1	111 (3)	C1—C2—O2	120.6 (2)
C5—O4—H4A	111 (2)	C1—C2—C3	119.2 (3)
C1—C6—C5	118.6 (3)	O2—C2—C3	120.0 (3)
C1—C6—H6	120.7	O4—C5—C4	117.3 (3)
C5—C6—H6	120.7	O4—C5—C6	121.2 (3)
C5—C4—C3	119.0 (3)	C4—C5—C6	121.5 (3)
C5—C4—H4	120.5	O2—C7—H7A	109.5
C3—C4—H4	120.5	O2—C7—H7B	109.5
C6—C1—O1	117.9 (3)	H7A—C7—H7B	109.5

C6—C1—C2	121.1 (2)	O2—C7—H7C	109.5
O1—C1—C2	120.9 (3)	H7A—C7—H7C	109.5
O3—C3—C4	123.2 (2)	H7B—C7—H7C	109.5

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O4—H4A···O1^{i}	0.89 (4)	1.93 (4)	2.823 (3)	177 (4)
O3—H3···O4^{ii}	0.92 (4)	1.83 (4)	2.743 (3)	172 (4)
O1—H1···O3^{iii}	0.892 (18)	1.889 (19)	2.778 (3)	174 (5)

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

Fig. 1

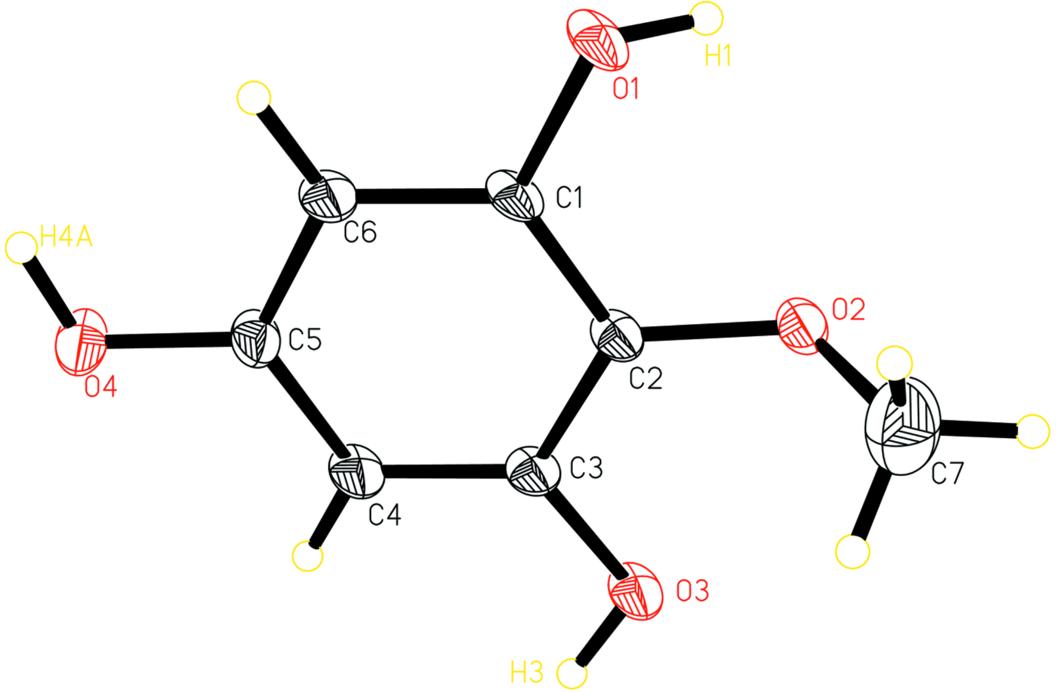

Fig. 2

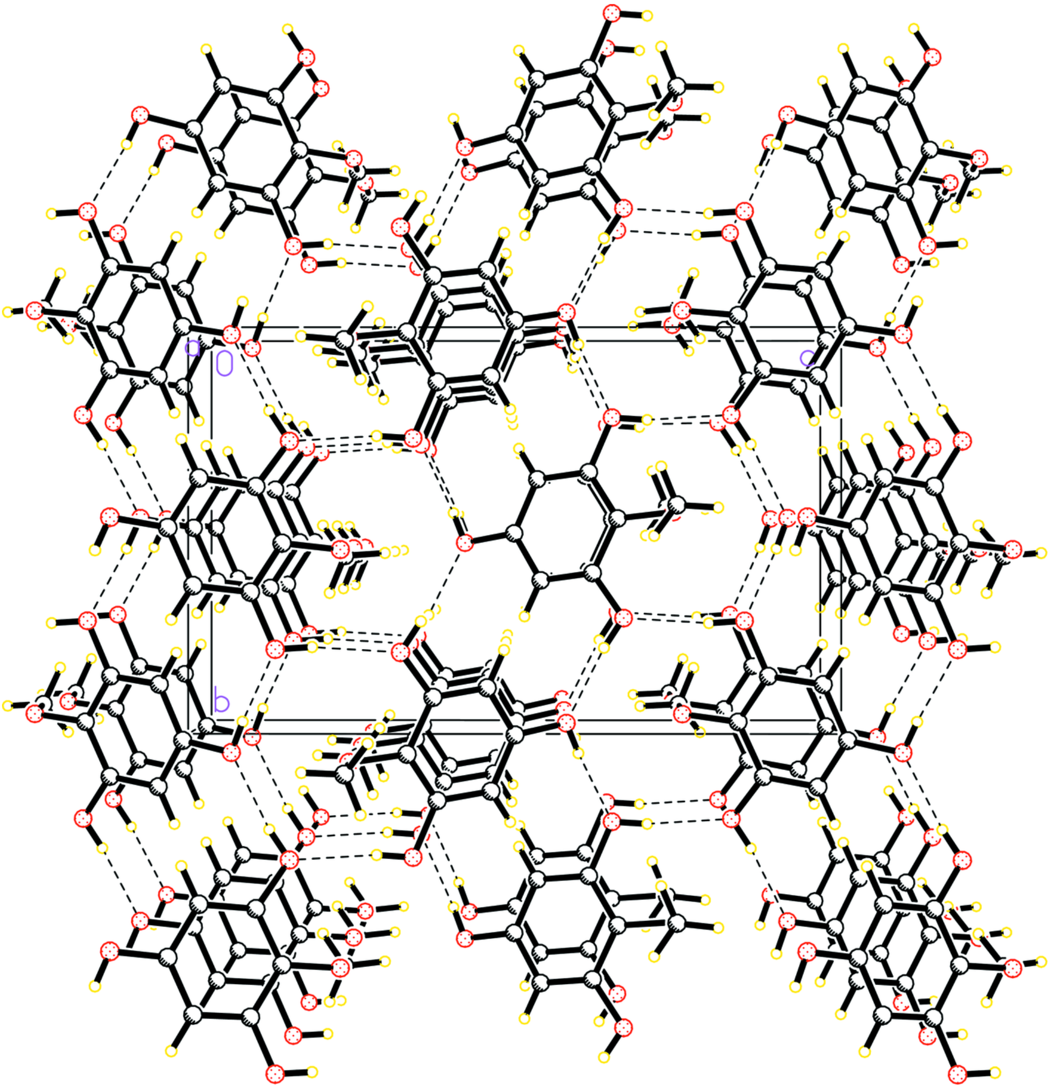

Fig. 3