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Redetermination of 4-hydroxybenzaldehyde

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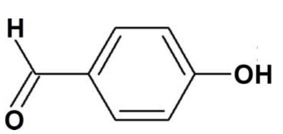
Key indicators: single-crystal X-ray study; T = 296 K; mean σ(C–C) = 0.002 Å; R factor = 0.038; wR factor = 0.117; data-to-parameter ratio = 13.6.

This is a redetermination of the structure of the title compound, $C_7H_6O_2$, which was first reported by Iwasaki [*Acta Cryst.* (1977), B**33**, 1646–1648]. The results are obtained with greater precision in the present study. Crystal packing is stabilized by intermolecular O—H⋯O interactions between the hydroxyl and aldehyde groups which link the molecules into chains in a zigzag pattern along the [110] plane of the unit cell.

Related literature

For the previous structure determination, see: Iwasaki (1977). Forrelated structures, see: Matos Beja *et al.* (1997, 2000); Paixão *et al.* (2000); Silva *et al.* (2004). For related literature, see: Antonucci (1978); Bigi *et al.* (1999); Dean (1963); Samal *et al.* (1999).



Experimental

Crystal data

$C_7H_6O_2$
M_r = 122.12
Monoclinic, $P2_1/c$
a = 6.6992 (8) Å
b = 13.5550 (12) Å
c = 7.1441 (11) Å
β = 112.871 (16)°
V = 597.74 (15) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.10 mm^{-1}
T = 296 (2) K
0.49 × 0.37 × 0.24 mm

Data collection

Oxford Diffraction Gemini R CCD diffractometer
Absorption correction: multi-scan (*CrysAlis RED*; Oxford Diffraction, 2007)
T_{min} = 0.949, T_{max} = 0.970
3559 measured reflections
1170 independent reflections
841 reflections with $I > 2\sigma(I)$
R_{int} = 0.022

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.038
$wR(F^2)$ = 0.117
S = 1.06
1170 reflections
86 parameters
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.13 e Å^{-3}
$\Delta\rho_{min}$ = −0.18 e Å^{-3}

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

D—H⋯A	D—H	H⋯A	D⋯A	D—H⋯A
O1—H1⋯O2^{i}	0.82 (3)	1.92 (3)	2.731 (2)	171 (2)

Symmetry code: (i) $-x, y+\frac{1}{2}, -z+\frac{1}{2}$.

Data collection: *CrysAlisPro* (Oxford Diffraction, 2007); cell refinement: *CrysAlisPro*; data reduction: *CrysAlisPro*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *SHELXTL* (Bruker, 2000); software used to prepare material for publication: *SHELXTL*.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: LX2042).

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supplementary materials

Acta Cryst. (2008). E**64**, o187 [**doi:10.1107/S1600536807063659**]

Redetermination of 4-hydroxybenzaldehyde

J. P. Jasinski, R. J. Butcher, B. Narayana, M. T. Swamy and H. S. Yathirajan

Comment

The title compound, 4-hydroxy benzaldehyde (Fig. 1) is used in the preparation of aldehyde methacrylates and finds application in the manufacture of dental materials which can form strong and durable bonds with dentin (Antonucci, 1978). It is used in the preparation of benzopyrans and have wide applications in the perfume, cosmetic and pharmaceutical industry (Dean, 1963; Bigi *et al.*, 1999). They are also used in the preparation of chelating resins (Samal *et al.*, 1999).

The crystal structures of *p*-hydroxybenzaldehyde (Iwasaki, 1977), 2-bromo-5-hydroxybenzaldehyde (Matos Beja *et al.*, 2000), a new polymorph of 2-bromo-5-hydroxybenzaldehyde (Silva *et al.*, 2004), 3-hydroxybenzaldehyde (Paixão *et al.*, 2000) and 2,4-dibromo-5-hydroxybenzaldehyde, (Matos Beja *et al.*, 1997) have been reported. In view of the importance of the title compound in the pharmaceutical industry, this paper reports a redetermination of the crystal structure with greater precision and accuracy. Crystal packing is stabilized by intermolecular O—H···O interactions between the hydroxyl and aldlehyde groups which link the molecules into chains in a zigzag pattern along the [110] plane of the unit cell (Fig. 2).

Experimental

A sample of 4-hydroxybenzaldehyde was obtained from Sigma–Aldrich and was recrystallized from ethylacetate by slow evaporation to obtain good quality crystals (m.p.: 385–387 K).

Refinement

The hydroxyl H was located in a difference Fourier map and all parameters were freely refined. All other H atoms were placed in their calculated places and refined using a riding model with C—H = 0.93 Å, and with $U_{iso}(H) = 1.21U_{eq}(C)$.

Figures

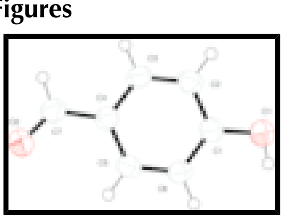

Fig. 1. *ORTEP* view of the title compound, showing the atom numbering scheme and 50% probability displacement ellipsoids.

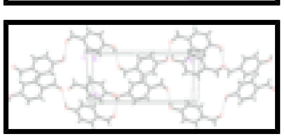

Fig. 2. The molecular packing for the title compound viewed down the *c* axis. Dashed lines indicate C—H···O intermolecular hydrogen bonds.

4-Hydroxybenzaldehyde

Crystal data

$C_7H_6O_2$	$F_{000} = 256$
$M_r = 122.12$	$D_x = 1.357$ Mg m^{-3}
Monoclinic, $P2_1/c$	Melting point: 385-387 K
Hall symbol: -P 2ybc	Mo $K\alpha$ radiation $\lambda = 0.71073$ Å
$a = 6.6992$ (8) Å	Cell parameters from 1669 reflections
$b = 13.5550$ (12) Å	$\theta = 5.3–29.0$º
$c = 7.1441$ (11) Å	$\mu = 0.10$ mm^{-1}
$\beta = 112.871$ (16)º	$T = 296$ (2) K
$V = 597.74$ (15) Å^3	Chunk, colourless
$Z = 4$	0.49 × 0.37 × 0.24 mm

Data collection

Oxford Diffraction Gemini R CCD diffractometer	1170 independent reflections
Radiation source: fine-focus sealed tube	841 reflections with $I > 2\sigma(I)$
Monochromator: graphite	$R_{int} = 0.022$
Detector resolution: 10.5081 pixels mm^{-1}	$\theta_{max} = 26.0$º
$T = 296(2)$ K	$\theta_{min} = 5.5$º
φ and ω scans	$h = -8 \rightarrow 8$
Absorption correction: multi-scan (CrysAlis RED; Oxford Diffraction, 2007)	$k = -15 \rightarrow 16$
$T_{min} = 0.949$, $T_{max} = 0.970$	$l = -8 \rightarrow 8$
3559 measured reflections	

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)] = 0.038$	H atoms treated by a mixture of independent and constrained refinement
$wR(F^2) = 0.117$	$w = 1/[\sigma^2(F_o^2) + (0.068P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$S = 1.06$	$(\Delta/\sigma)_{max} < 0.001$
1170 reflections	$\Delta\rho_{max} = 0.13$ e Å^{-3}
86 parameters	$\Delta\rho_{min} = -0.18$ e Å^{-3}
Primary atom site location: structure-invariant direct methods	Extinction correction: none

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}
O1	0.1345 (2)	0.72809 (8)	0.1335 (2)	0.0652 (4)
H1	0.016 (4)	0.7359 (17)	0.140 (3)	0.092 (8)*
O2	0.2691 (2)	0.26982 (8)	0.3786 (2)	0.0633 (4)
C1	0.1834 (2)	0.63218 (11)	0.1834 (2)	0.0458 (4)
C2	0.3718 (2)	0.59530 (11)	0.1700 (2)	0.0499 (4)
H2	0.4587	0.6359	0.1284	0.060*
C3	0.4277 (2)	0.49829 (11)	0.2187 (2)	0.0465 (4)
H3	0.5542	0.4740	0.2112	0.056*
C4	0.2990 (2)	0.43542 (11)	0.2793 (2)	0.0417 (4)
C5	0.1100 (2)	0.47379 (11)	0.2921 (2)	0.0452 (4)
H5	0.0218	0.4330	0.3317	0.054*
C6	0.0543 (2)	0.57089 (11)	0.2467 (2)	0.0470 (4)
H6	−0.0698	0.5959	0.2581	0.056*
C7	0.3667 (3)	0.33405 (12)	0.3294 (2)	0.0509 (4)
H7	0.4981	0.3161	0.3235	0.061*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.0635 (8)	0.0422 (7)	0.1018 (10)	0.0034 (6)	0.0451 (7)	0.0091 (6)
O2	0.0586 (7)	0.0434 (7)	0.0917 (9)	−0.0019 (5)	0.0332 (7)	0.0049 (6)
C1	0.0463 (8)	0.0385 (8)	0.0547 (9)	−0.0030 (6)	0.0219 (7)	−0.0039 (7)
C2	0.0472 (9)	0.0463 (9)	0.0636 (10)	−0.0078 (7)	0.0297 (8)	−0.0039 (7)
C3	0.0372 (7)	0.0483 (9)	0.0582 (9)	−0.0026 (6)	0.0231 (7)	−0.0089 (7)
C4	0.0396 (8)	0.0404 (8)	0.0447 (8)	−0.0013 (6)	0.0157 (6)	−0.0058 (6)
C5	0.0417 (8)	0.0450 (9)	0.0536 (9)	−0.0050 (7)	0.0235 (7)	−0.0012 (7)
C6	0.0404 (8)	0.0466 (9)	0.0593 (9)	0.0020 (7)	0.0252 (7)	−0.0018 (7)
C7	0.0430 (8)	0.0455 (9)	0.0648 (10)	−0.0012 (7)	0.0217 (8)	−0.0055 (7)

Geometric parameters (Å, °)

O1—C1	1.354 (2)	C3—H3	0.9300
O1—H1	0.82 (3)	C4—C5	1.404 (2)

O2—C7	1.219 (2)	C4—C7	1.448 (2)
C1—C2	1.395 (2)	C5—C6	1.372 (2)
C1—C6	1.395 (2)	C5—H5	0.9300
C2—C3	1.374 (2)	C6—H6	0.9300
C2—H2	0.9300	C7—H7	0.9300
C3—C4	1.395 (2)		
C1—O1—H1	104.4 (16)	C3—C4—C7	118.98 (13)
O1—C1—C2	117.30 (14)	C5—C4—C7	122.62 (13)
O1—C1—C6	122.68 (14)	C6—C5—C4	120.61 (13)
C2—C1—C6	120.02 (14)	C6—C5—H5	119.7
C3—C2—C1	119.35 (14)	C4—C5—H5	119.7
C3—C2—H2	120.3	C5—C6—C1	120.12 (14)
C1—C2—H2	120.3	C5—C6—H6	119.9
C2—C3—C4	121.50 (14)	C1—C6—H6	119.9
C2—C3—H3	119.3	O2—C7—C4	126.70 (15)
C4—C3—H3	119.3	O2—C7—H7	116.7
C3—C4—C5	118.39 (13)	C4—C7—H7	116.7
O1—C1—C2—C3	−179.85 (14)	C7—C4—C5—C6	178.77 (14)
C6—C1—C2—C3	0.3 (2)	C4—C5—C6—C1	1.2 (2)
C1—C2—C3—C4	0.7 (2)	O1—C1—C6—C5	178.90 (14)
C2—C3—C4—C5	−0.8 (2)	C2—C1—C6—C5	−1.2 (2)
C2—C3—C4—C7	−179.78 (14)	C3—C4—C7—O2	−177.67 (15)
C3—C4—C5—C6	−0.2 (2)	C5—C4—C7—O2	3.4 (3)

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O1—H1···O2^{i}	0.82 (3)	1.92 (3)	2.731 (2)	171 (2)

Symmetry codes: (i) −*x*, *y*+1/2, −*z*+1/2.

Fig. 1

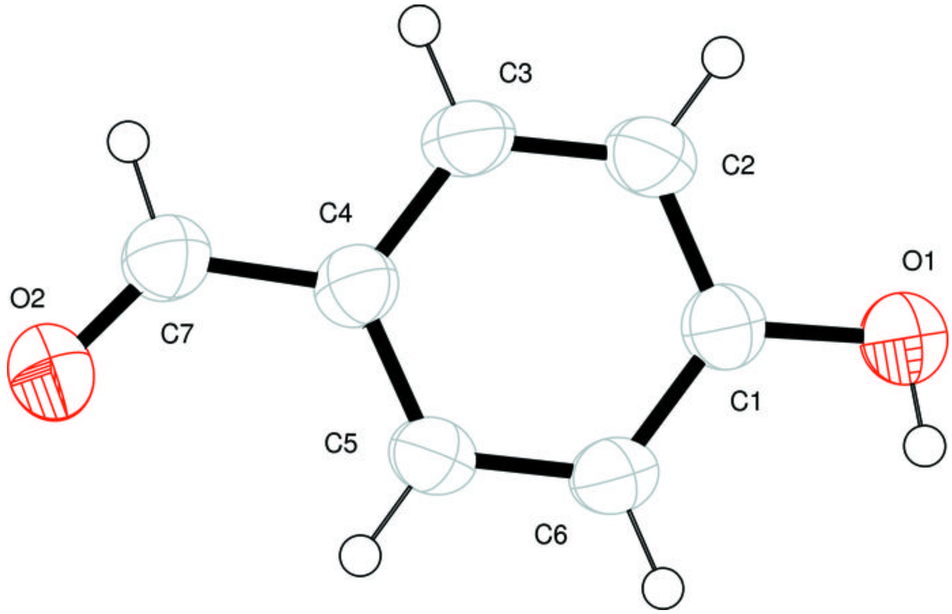

Fig. 2