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4-Acetylresorcinol

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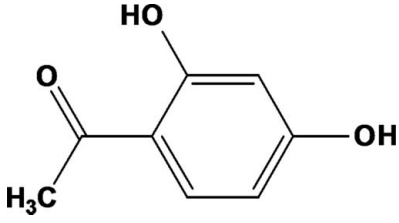
Key indicators: single-crystal X-ray study; T = 296 K; mean σ(C–C) = 0.002 Å; R factor = 0.041; wR factor = 0.115; data-to-parameter ratio = 13.5.

The molecule of the title compound [1-(2,4-dihydroxyphenyl)-ethanone], $C_8H_8O_3$, is planar except for the methyl H atoms. One intramolecular O—H···O hydrogen bond and a second intermolecular O—H···O interaction link the molecules into chains as seen down the 010 face. The distance between the sheets is 3.049 Å at its closest and 3.262 Å at its farthest.

Related literature

For related literature, see: Fronczek *et al.* (1987); Kokila *et al.* (1992); Liebich (1979); Li *et al.* (2005); van Rooyen & Breytenbach (1988).



Experimental

Crystal data

$C_8H_8O_3$
M_r = 152.14
Monoclinic, $P2_1/c$
a = 7.1325 (3) Å
b = 13.7067 (5) Å
c = 7.2998 (3) Å
β = 92.369 (2)°
V = 713.04 (5) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.11 mm^{-1}
T = 296 (2) K
0.50 × 0.32 × 0.24 mm

Data collection

Bruker APEXII CCD area-detector diffractometer
Absorption correction: numerical (*SADABS*; Bruker, 2006)
T_{min} = 0.948, T_{max} = 0.974
11606 measured reflections
1394 independent reflections
1112 reflections with $I > 2\sigma(I)$
R_{int} = 0.059

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.041
$wR(F^2)$ = 0.115
S = 1.06
1394 reflections
103 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.34 e Å^{-3}
$\Delta\rho_{min}$ = −0.21 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
O2—H2A···O1	0.82	1.84	2.56 (4)	146
O3—H1···O2^{i}	0.82	1.92	2.65 (4)	148

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

Data collection: *APEX2* (Bruker, 2006); cell refinement: *APEX2*; data reduction: *SAINT* (Bruker, 2006); program(s) used to solve structure: *SHELXTL* (Bruker, 2006); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *ORTEP-3* (Farrugia, 1997); 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: FL2166).

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supplementary materials

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4-Acetylresorcinol

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Comment

The title compound, (I, Fig. 1), was formed in a reaction mixture containing resorcinol, 3,4-pyridine dicarboxlyic acid anhydride, acetic acid and sulfuric acid where the original intent was to prepare a fluoroscein derivative containing pyridine for use as a chelating ligand which would allow for detection of metal ions at very low concentrations. The title compound is currently being used as a starting material for synthesis of new chelating ligands. There have been several derivatives of resorcinol published in the literature (Fronczek *et al.*, 1987; Kokila *et al.*, 1992; Liebich, 1979; Li *et al.*, 2005; van Rooyen & Breytenbach, 1988). The compound most similiar to (I) (Kokila *et al.*, 1992) has two acetyl groups found in the 4 and 6 positions. However, it differs from (I) in that it has two intramolecular O—H···O hydrogen bonds and no intermolecular interactions. In (I), in addition to the O3 ··· O4 intramolecular H-bond there is an intermolecular H-bod between O2 and O3 that links the molecules inttoo sheets which can be seen by viewing the unit cell down the 010 face. The distance between the sheets is 3.049 Å at its closest and 3.262 Å at its fartherest.

Experimental

The title compound was synthesized by refluxing 0.11 g (0.75 mmol) of 3,4-Pyridinedicarboxylic anhydride and 0.19 g (1.72 mmol) of resorcinol in 15:1 Glacial Acetic Acid:Water for 30 minutes. Added 250.7 µ*L* of con. H_2SO_4 and 10 ml of water. Continued relux overnight. Allowed the solution to cool. Suitable crystals of the title compound were found on the bottom.

Refinement

All non-hydrogen atoms were refined using anisotropic thermal parameters. All hydrogen atoms were included at idealized positions and not refined.

Figures

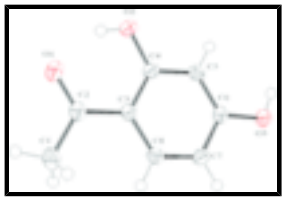

Fig. 1. *ORTEP* drawing with 50% elipsoids.

1-(2,4-dihydroxyphenyl)ethanone

Crystal data

$C_8H_8O_3$	F_{000} = 320
M_r = 152.14	D_x = 1.417 Mg m^{-3}

Monoclinic, $P2_1/c$	Mo $K\alpha$ radiation $\lambda = 0.71073$ Å
Hall symbol: -P 2ybc	Cell parameters from 3391 reflections
$a = 7.1325$ (3) Å	$\theta = 2.9$–$26.7°$
$b = 13.7067$ (5) Å	$\mu = 0.11$ mm^{-1}
$c = 7.2998$ (3) Å	$T = 296$ (2) K
$\beta = 92.369$ (2)°	Block, red
$V = 713.04$ (5) Å^3	0.50 × 0.32 × 0.24 mm
$Z = 4$	

Data collection

Bruker APEXII CCD area-detector diffractometer	1394 independent reflections
Radiation source: fine-focus sealed tube	1112 reflections with $I > 2\sigma(I)$
Monochromator: graphite	$R_{int} = 0.059$
$T = 296(2)$ K	$\theta_{max} = 26.0°$
φ and ω scans	$\theta_{min} = 2.9°$
Absorption correction: numerical (SADABS; Bruker, 2006)	$h = -8 \rightarrow 8$
$T_{min} = 0.948$, $T_{max} = 0.974$	$k = -16 \rightarrow 16$
11606 measured reflections	$l = -8 \rightarrow 8$

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)] = 0.041$	H-atom parameters constrained
$wR(F^2) = 0.115$	$w = 1/[\sigma^2(F_o^2) + (0.0585P)^2 + 0.1744P]$ where $P = (F_o^2 + 2F_c^2)/3$
$S = 1.06$	$(\Delta/\sigma)_{max} < 0.001$
1394 reflections	$\Delta\rho_{max} = 0.34$ e Å^{-3}
103 parameters	$\Delta\rho_{min} = -0.21$ 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}
C8	1.3101 (2)	0.43113 (11)	0.6235 (2)	0.0320 (4)
H8A	1.4239	0.4138	0.5657	0.048*
H8B	1.3391	0.4725	0.7270	0.048*
H8C	1.2285	0.4652	0.5374	0.048*
C7	1.2146 (2)	0.34057 (10)	0.68663 (18)	0.0249 (3)
C4	1.03441 (18)	0.34595 (9)	0.77170 (17)	0.0212 (3)
C3	0.94218 (19)	0.26014 (9)	0.82921 (17)	0.0214 (3)
C2	0.77058 (19)	0.26558 (10)	0.91087 (17)	0.0226 (3)
H2	0.7107	0.2090	0.9473	0.027*
C1	0.68871 (19)	0.35572 (9)	0.93794 (18)	0.0235 (3)
C6	0.7743 (2)	0.44146 (10)	0.87870 (19)	0.0259 (3)
H6	0.7165	0.5016	0.8941	0.031*
C5	0.94371 (19)	0.43583 (9)	0.79787 (17)	0.0239 (3)
H5	1.0005	0.4929	0.7592	0.029*
O3	1.29189 (14)	0.26013 (7)	0.66509 (14)	0.0320 (3)
O2	1.01829 (14)	0.17102 (7)	0.80826 (13)	0.0280 (3)
H2A	1.1171	0.1762	0.7555	0.042*
O1	0.52510 (14)	0.36696 (7)	1.02242 (15)	0.0320 (3)
H1	0.4859	0.3134	1.0531	0.048*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C8	0.0231 (8)	0.0356 (8)	0.0378 (8)	−0.0037 (6)	0.0075 (6)	0.0033 (6)
C7	0.0216 (8)	0.0291 (8)	0.0240 (7)	0.0015 (6)	0.0018 (5)	−0.0012 (5)
C4	0.0196 (8)	0.0216 (7)	0.0224 (7)	−0.0007 (5)	0.0018 (5)	0.0002 (5)
C3	0.0234 (7)	0.0196 (7)	0.0212 (6)	0.0027 (5)	0.0003 (5)	−0.0014 (5)
C2	0.0238 (8)	0.0201 (7)	0.0242 (7)	−0.0032 (5)	0.0025 (5)	0.0014 (5)
C1	0.0202 (7)	0.0269 (7)	0.0236 (7)	0.0009 (5)	0.0034 (5)	−0.0011 (5)
C6	0.0248 (8)	0.0201 (7)	0.0330 (7)	0.0037 (5)	0.0035 (6)	−0.0005 (6)
C5	0.0246 (8)	0.0192 (7)	0.0280 (7)	−0.0017 (5)	0.0020 (5)	0.0010 (5)
O3	0.0270 (6)	0.0319 (6)	0.0377 (6)	0.0052 (4)	0.0101 (4)	−0.0016 (4)
O2	0.0295 (6)	0.0200 (5)	0.0352 (6)	0.0038 (4)	0.0091 (4)	−0.0004 (4)
O1	0.0258 (6)	0.0273 (6)	0.0440 (6)	0.0021 (4)	0.0158 (5)	0.0019 (5)

Geometric parameters (Å, °)

C8—C7	1.4976 (19)	C2—C1	1.3842 (18)
C8—H8A	0.9600	C2—H2	0.9300
C8—H8B	0.9600	C1—O1	1.3510 (17)
C8—H8C	0.9600	C1—C6	1.4008 (19)
C7—O3	1.2455 (17)	C6—C5	1.369 (2)
C7—C4	1.4522 (19)	C6—H6	0.9300
C4—C5	1.4081 (18)	C5—H5	0.9300
C4—C3	1.4196 (18)	O2—H2A	0.8200

C3—O2	1.3480 (15)	O1—H1	0.8200
C3—C2	1.3853 (19)		
C7—C8—H8A	109.5	C1—C2—C3	119.66 (12)
C7—C8—H8B	109.5	C1—C2—H2	120.2
H8A—C8—H8B	109.5	C3—C2—H2	120.2
C7—C8—H8C	109.5	O1—C1—C2	122.98 (12)
H8A—C8—H8C	109.5	O1—C1—C6	116.15 (11)
H8B—C8—H8C	109.5	C2—C1—C6	120.87 (13)
O3—C7—C4	120.21 (12)	C5—C6—C1	119.30 (12)
O3—C7—C8	119.04 (13)	C5—C6—H6	120.4
C4—C7—C8	120.75 (12)	C1—C6—H6	120.4
C5—C4—C3	117.62 (12)	C6—C5—C4	121.77 (12)
C5—C4—C7	121.47 (12)	C6—C5—H5	119.1
C3—C4—C7	120.90 (12)	C4—C5—H5	119.1
O2—C3—C2	117.69 (12)	C3—O2—H2A	109.5
O2—C3—C4	121.57 (12)	C1—O1—H1	109.5
C2—C3—C4	120.74 (12)		

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O2—H2A···O1	0.82	1.84	2.56 (4)	146
O3—H1···O2^{i}	0.82	1.92	2.65 (4)	148

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

Fig. 1