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## Structure Reports

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# Dimethyl 2,6-dihydroxybenzene-1,4-dicarboxylate

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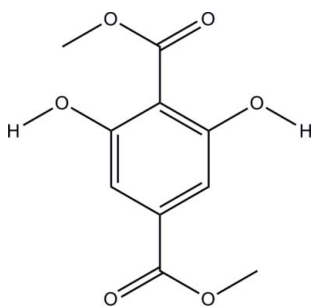
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 Key indicators: single-crystal X-ray study;  $T = 293$  K; mean  $\sigma(\text{C}-\text{C}) = 0.004$  Å;  $R$  factor = 0.049;  $wR$  factor = 0.178; data-to-parameter ratio = 14.0.

The title compound,  $\text{C}_{10}\text{H}_{10}\text{O}_6$ , was obtained from an esterification reaction of 2,6-dihydroxyterephthalic acid and methanol. In the molecular structure, all of the C atoms are nearly coplanar. The two hydroxy groups have  $C_2$  symmetry. Intramolecular  $\text{O}-\text{H}\cdots\text{O}$  hydrogen bonds are observed. In the crystal, weak  $\text{O}-\text{H}\cdots\text{O}$  interactions link the molecules.

## Related literature

For general background to terephthalate derivatives, see: Brunner (1928); Teruhiko *et al.* (1998). For a related structure, see: Dai *et al.* (2005).



## Experimental

### Crystal data

 $\text{C}_{10}\text{H}_{10}\text{O}_6$   
 $M_r = 226.18$ 

 Monoclinic,  $P2_1/c$   
 $a = 11.6462$  (8) Å

 $b = 7.0925$  (3) Å  
 $c = 13.5745$  (10) Å  
 $\beta = 114.327$  (9)°  
 $V = 1021.70$  (11) Å<sup>3</sup>  
 $Z = 4$ 

 Mo  $K\alpha$  radiation  
 $\mu = 0.12$  mm<sup>-1</sup>  
 $T = 293$  K  
 $0.34 \times 0.26 \times 0.20$  mm

### Data collection

 Oxford Diffraction Xcalibur Eos  
 Gemini ultra diffractometer  
 Absorption correction: multi-scan  
 (*CrysAlis PRO*; Oxford  
 Diffraction, 2009)  
 $T_{\min} = 0.865$ ,  $T_{\max} = 1.000$ 

 4572 measured reflections  
 2083 independent reflections  
 1161 reflections with  $I > 2\sigma(I)$   
 $R_{\text{int}} = 0.023$ 

### Refinement

 $R[F^2 > 2\sigma(F^2)] = 0.049$   
 $wR(F^2) = 0.178$   
 $S = 1.09$   
 2083 reflections

 149 parameters  
 H-atom parameters constrained  
 $\Delta\rho_{\text{max}} = 0.26$  e Å<sup>-3</sup>  
 $\Delta\rho_{\text{min}} = -0.21$  e Å<sup>-3</sup>
**Table 1**

Hydrogen-bond geometry (Å, °).

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
$\text{O3}-\text{H3}\cdots\text{O2}$	0.82	1.84	2.567 (3)	147
$\text{O4}-\text{H4}\cdots\text{O1}$	0.82	1.89	2.593 (3)	144
$\text{O4}-\text{H4}\cdots\text{O3}^i$	0.82	2.58	3.099 (3)	123

 Symmetry code: (i)  $x, y + 1, z$ .

Data collection: *CrysAlis PRO* (Oxford Diffraction, 2009); cell refinement: *CrysAlis PRO*; data reduction: *CrysAlis PRO*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *OLEX2* (Dolomanov *et al.*, 2009); software used to prepare material for publication: *OLEX2*.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: GW2078).

## References

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## supporting information

*Acta Cryst.* (2010). E66, o981 [doi:10.1107/S1600536810011074]

## Dimethyl 2,6-dihydroxybenzene-1,4-dicarboxylate

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### S1. Comment

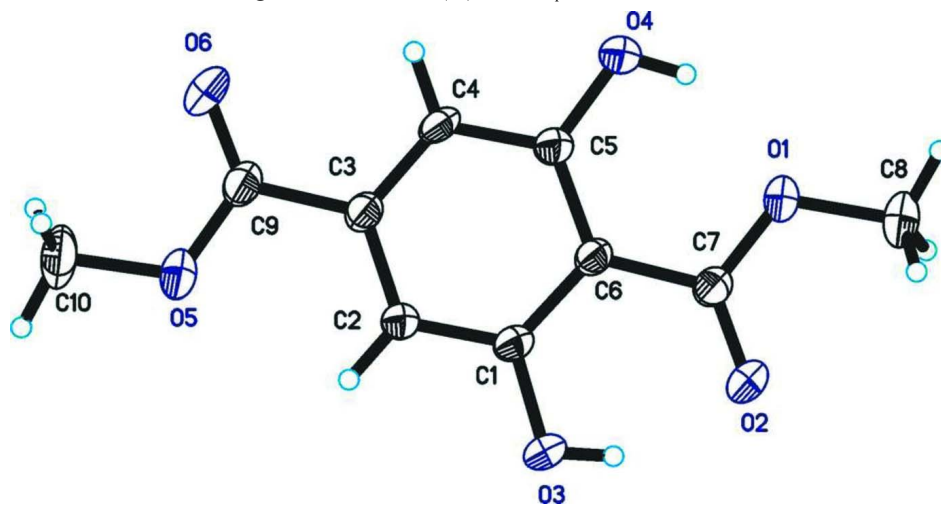
The title compound as one of terephthalate derivatives, is an important pharmacological and material intermediate (Brunner, 1928; Teruhiko *et al.*, 1998). There is almost no report about crystal structure. As part of our ongoing studies, we now describe the synthesis and the crystal structure of the title compound. In this paper, the crystal structure of it was determined (Fig. 1). The molecule contains benzene ring, dihydroxy group and dimethyl group. All of atoms except the hydrogen atoms are nearly coplanar. The terminal dimethyl group are centrosymmetric. The dihydroxy group are axisymmetric. In the crystal structure, intramolecular O—H...O hydrogen bonds are observed.

### S2. Experimental

2,6-dihydroxyterephthalic acid (15 mmol) was dissolved in methanol (45 ml), thionyl dichloride (3 ml) was slowly added to the methanol solution afterward, and the mixture was stirred at reflux temperature for 72 hours (monitored by TLC). Then the solvent was distilled under vacuum, and the residue was poured into water (50 ml). The pH of the solution was adjusted with sodium bicarbonate to pH = 7.0. The resulting white solid was filtered off, washed with water. The obtained solid was dissolved in methanol. Single crystals were obtained by slow evaporation of a methanol solution.

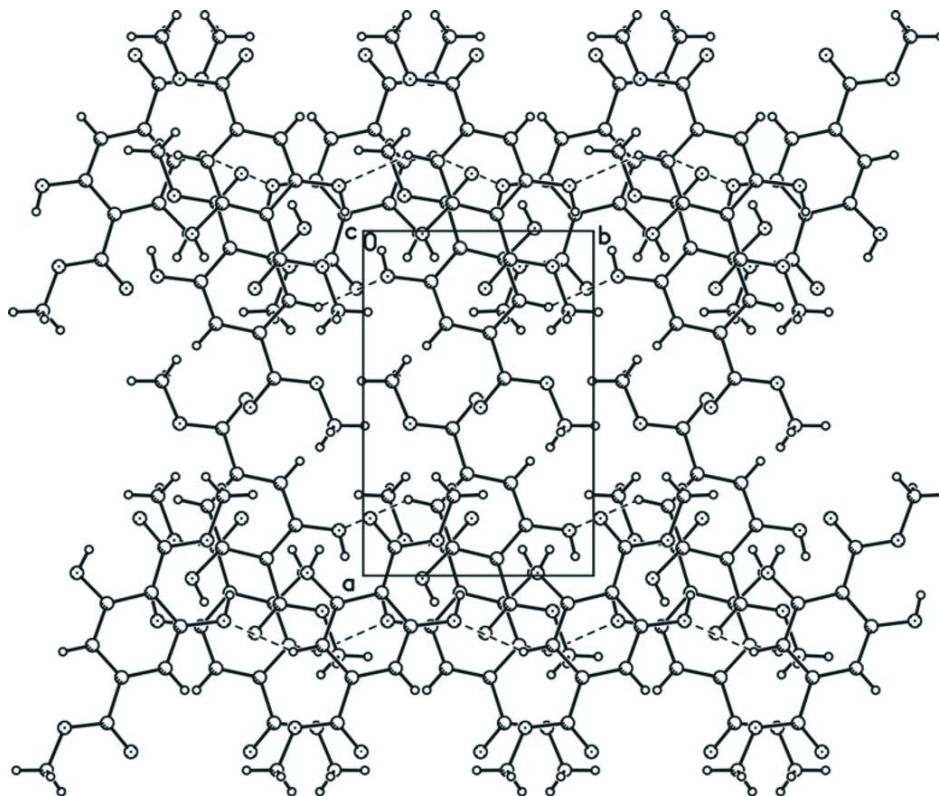
### S3. Refinement

H atoms were placed in calculated position with C—H=0.96 (1) Å(sp<sup>3</sup>), C—H=0.93 Å(aromatic). All H atoms included in the final cycles of refinement as riding mode, with  $U_{\text{iso}}(\text{H})=1.2U_{\text{eq}}$  of the carrier atoms.



**Figure 1**

The asymmetric unit of the structure of the title compound, with the atomic labeling scheme. Displacement ellipsoids are drawn at the 50% probability level.

**Figure 2**

The molecule packing of the title compound showing O—H...O interactions.

### Dimethyl 2,6-dihydroxybenzene-1,4-dicarboxylate

#### Crystal data

$C_{10}H_{10}O_6$

$M_r = 226.18$

Monoclinic,  $P2_1/c$

Hall symbol:  $-P\ 2_1/c$

$a = 11.6462\ (8)\ \text{\AA}$

$b = 7.0925\ (3)\ \text{\AA}$

$c = 13.5745\ (10)\ \text{\AA}$

$\beta = 114.327\ (9)^\circ$

$V = 1021.70\ (11)\ \text{\AA}^3$

$Z = 4$

$F(000) = 472$

$D_x = 1.470\ \text{Mg m}^{-3}$

Mo  $K\alpha$  radiation,  $\lambda = 0.71073\ \text{\AA}$

Cell parameters from 1855 reflections

$\theta = 2.9\text{--}29.2^\circ$

$\mu = 0.12\ \text{mm}^{-1}$

$T = 293\ \text{K}$

Block, white

$0.34 \times 0.26 \times 0.20\ \text{mm}$

#### Data collection

Oxford Diffraction Xcalibur Eos Gemini ultra diffractometer

Radiation source: fine-focus sealed tube

Graphite monochromator

Detector resolution:  $16.0395\ \text{pixels mm}^{-1}$

$\omega$  scans

Absorption correction: multi-scan

(*CrysAlis PRO*; Oxford Diffraction, 2009)

$T_{\min} = 0.865$ ,  $T_{\max} = 1.000$

4572 measured reflections

2083 independent reflections

1161 reflections with  $I > 2\sigma(I)$

$R_{\text{int}} = 0.023$

$\theta_{\max} = 26.4^\circ$ ,  $\theta_{\min} = 3.3^\circ$

$h = -14 \rightarrow 10$

$k = -8 \rightarrow 8$

$l = -16 \rightarrow 15$

Refinement

Refinement on  $F^2$   
 Least-squares matrix: full  
 $R[F^2 > 2\sigma(F^2)] = 0.049$   
 $wR(F^2) = 0.178$   
 $S = 1.09$   
 2083 reflections  
 149 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-atom parameters constrained  
 $w = 1/[\sigma^2(F_o^2) + (0.0906P)^2 + 0.0929P]$   
 where  $P = (F_o^2 + 2F_c^2)/3$   
 $(\Delta/\sigma)_{\max} = 0.002$   
 $\Delta\rho_{\max} = 0.26 \text{ e } \text{\AA}^{-3}$   
 $\Delta\rho_{\min} = -0.21 \text{ e } \text{\AA}^{-3}$

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.** 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  $F^2 > \sigma(F^2)$  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 ( $\text{\AA}^2$ )

	$x$	$y$	$z$	$U_{\text{iso}}^*/U_{\text{eq}}$
O1	1.10292 (17)	0.3212 (3)	0.65789 (15)	0.0531 (6)
O2	1.16658 (17)	0.0296 (3)	0.71430 (17)	0.0594 (6)
O3	1.00830 (17)	-0.2437 (3)	0.65185 (18)	0.0588 (6)
H3	1.0760	-0.1914	0.6860	0.088*
O4	0.8686 (2)	0.3940 (3)	0.5366 (2)	0.0694 (7)
H4	0.9419	0.4218	0.5747	0.104*
O5	0.48877 (19)	-0.0097 (4)	0.36441 (19)	0.0809 (8)
O6	0.55844 (19)	-0.2972 (3)	0.42310 (17)	0.0622 (6)
C1	1.2310 (3)	0.3910 (5)	0.7143 (3)	0.0605 (9)
H1A	1.2712	0.3957	0.6652	0.091*
H1C	1.2773	0.3082	0.7733	0.091*
H1B	1.2290	0.5152	0.7417	0.091*
C2	1.0826 (2)	0.1371 (4)	0.6627 (2)	0.0418 (6)
C3	0.9508 (2)	0.0794 (3)	0.6008 (2)	0.0370 (6)
C4	0.9197 (2)	-0.1122 (3)	0.5979 (2)	0.0393 (6)
C5	0.7983 (2)	-0.1768 (4)	0.5401 (2)	0.0395 (6)
H5	0.7798	-0.3044	0.5397	0.047*
C6	0.7048 (2)	-0.0498 (4)	0.4829 (2)	0.0391 (6)
C7	0.7314 (2)	0.1397 (4)	0.4830 (2)	0.0449 (7)
H7	0.6675	0.2237	0.4438	0.054*
C8	0.8531 (2)	0.2059 (3)	0.5412 (2)	0.0430 (7)
C9	0.5725 (3)	-0.1122 (4)	0.4170 (2)	0.0476 (7)
C10	0.4355 (3)	-0.3758 (5)	0.3582 (3)	0.0769 (11)
H10A	0.4105	-0.3402	0.2840	0.115*
H10B	0.3752	-0.3286	0.3835	0.115*

H10C	0.4392	-0.5108	0.3641	0.115*
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*Atomic displacement parameters (Å<sup>2</sup>)*

	$U^{11}$	$U^{22}$	$U^{33}$	$U^{12}$	$U^{13}$	$U^{23}$
O1	0.0335 (11)	0.0493 (12)	0.0635 (13)	-0.0079 (9)	0.0067 (10)	-0.0032 (10)
O2	0.0326 (10)	0.0563 (12)	0.0718 (14)	0.0062 (9)	0.0039 (10)	0.0043 (11)
O3	0.0360 (11)	0.0445 (11)	0.0775 (14)	0.0103 (9)	0.0048 (10)	0.0115 (11)
O4	0.0480 (13)	0.0347 (11)	0.0953 (18)	0.0029 (9)	-0.0010 (12)	0.0016 (11)
O5	0.0340 (11)	0.0803 (17)	0.0952 (19)	0.0052 (12)	-0.0068 (12)	0.0146 (14)
O6	0.0367 (11)	0.0582 (14)	0.0749 (15)	-0.0086 (10)	0.0062 (10)	-0.0071 (11)
C1	0.0382 (16)	0.070 (2)	0.0634 (19)	-0.0148 (15)	0.0108 (15)	-0.0065 (17)
C2	0.0343 (14)	0.0434 (16)	0.0428 (15)	0.0023 (12)	0.0111 (12)	-0.0003 (13)
C3	0.0298 (13)	0.0377 (14)	0.0389 (14)	0.0039 (11)	0.0094 (11)	-0.0029 (11)
C4	0.0333 (14)	0.0376 (15)	0.0430 (15)	0.0079 (11)	0.0116 (12)	0.0029 (12)
C5	0.0341 (15)	0.0331 (13)	0.0464 (15)	0.0031 (11)	0.0116 (13)	-0.0024 (12)
C6	0.0312 (14)	0.0421 (15)	0.0390 (14)	0.0021 (11)	0.0096 (12)	-0.0025 (11)
C7	0.0298 (14)	0.0459 (17)	0.0460 (16)	0.0130 (12)	0.0026 (12)	0.0021 (13)
C8	0.0366 (15)	0.0346 (15)	0.0498 (16)	0.0029 (11)	0.0097 (13)	-0.0027 (12)
C9	0.0322 (15)	0.0552 (19)	0.0487 (17)	0.0008 (14)	0.0098 (13)	-0.0040 (14)
C10	0.0425 (19)	0.090 (3)	0.084 (2)	-0.0231 (18)	0.0110 (18)	-0.024 (2)

*Geometric parameters (Å, °)*

O1—C2	1.334 (3)	C2—C3	1.472 (4)
O1—C1	1.454 (3)	C3—C4	1.403 (3)
O2—C2	1.210 (3)	C3—C8	1.413 (3)
O3—C4	1.360 (3)	C4—C5	1.382 (3)
O3—H3	0.8200	C5—C6	1.379 (3)
O4—C8	1.351 (3)	C5—H5	0.9300
O4—H4	0.8200	C6—C7	1.379 (4)
O5—C9	1.190 (3)	C6—C9	1.495 (4)
O6—C9	1.329 (3)	C7—C8	1.389 (4)
O6—C10	1.449 (3)	C7—H7	0.9300
C1—H1A	0.9600	C10—H10A	0.9600
C1—H1C	0.9600	C10—H10B	0.9600
C1—H1B	0.9600	C10—H10C	0.9600
C2—O1—C1	118.1 (2)	C6—C5—H5	120.4
C4—O3—H3	109.5	C4—C5—H5	120.4
C8—O4—H4	109.5	C7—C6—C5	120.8 (2)
C9—O6—C10	117.3 (3)	C7—C6—C9	117.7 (2)
O1—C1—H1A	109.5	C5—C6—C9	121.6 (2)
O1—C1—H1C	109.5	C6—C7—C8	120.4 (2)
H1A—C1—H1C	109.5	C6—C7—H7	119.8
O1—C1—H1B	109.5	C8—C7—H7	119.8
H1A—C1—H1B	109.5	O4—C8—C7	115.6 (2)
H1C—C1—H1B	109.5	O4—C8—C3	124.2 (2)

O2—C2—O1	121.8 (2)	C7—C8—C3	120.3 (2)
O2—C2—C3	124.1 (2)	O5—C9—O6	123.3 (3)
O1—C2—C3	114.1 (2)	O5—C9—C6	124.5 (3)
C4—C3—C8	117.4 (2)	O6—C9—C6	112.2 (2)
C4—C3—C2	118.8 (2)	O6—C10—H10A	109.5
C8—C3—C2	123.8 (2)	O6—C10—H10B	109.5
O3—C4—C5	116.8 (2)	H10A—C10—H10B	109.5
O3—C4—C3	121.3 (2)	O6—C10—H10C	109.5
C5—C4—C3	121.9 (2)	H10A—C10—H10C	109.5
C6—C5—C4	119.3 (2)	H10B—C10—H10C	109.5

*Hydrogen-bond geometry (Å, °)*

<i>D</i> —H $\cdots$ <i>A</i>	<i>D</i> —H	H $\cdots$ <i>A</i>	<i>D</i> $\cdots$ <i>A</i>	<i>D</i> —H $\cdots$ <i>A</i>
O3—H3 $\cdots$ O2	0.82	1.84	2.567 (3)	147
O4—H4 $\cdots$ O1	0.82	1.89	2.593 (3)	144
O4—H4 $\cdots$ O3 <sup>i</sup>	0.82	2.58	3.099 (3)	123

Symmetry code: (i) *x*, *y*+1, *z*.