Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

Dimethyl 2,3-dihydroxyterephthalate

Jian-Ying Huang* and Hong-Ze Liang

Faculty of Materials Science and Chemical Engineering, Ningbo University, Ningbo, Zhejiang 315211, People's Republic of China

Correspondence e-mail: huangjianying@nbu.edu.cn

Key indicators

Single-crystal X-ray study
T = 298 K
Mean σ(C–C) = 0.002 Å
R factor = 0.042
wR factor = 0.112
Data-to-parameter ratio = 15.6

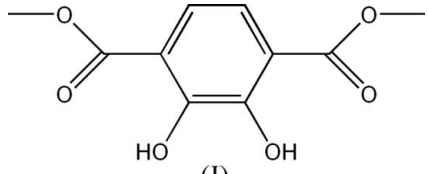
For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

The title compound, $C_{10}H_{10}O_6$, shows strong intramolecular O—H···O hydrogen bonds. Short intermolecular O···O weak interactions and weak intermolecular O—H···O hydrogen bonds link molecules into a supramolecular dimer.

Received 26 January 2007
Accepted 9 May 2007

Comment

Dimethyl 2,3-dihydroxyterephthalate is an important intermediate widely used in the synthesis of 1*H*,4*H*-benzo[2,1-*b*:3,4-*b*′]bisthiete and 2*H*,3*H*-benzo[1,2-*b*:4,3-*b*′]bisthiete, which are very versatile reagents for the generation of polycyclic sulfur-containing compounds (Meier & Rumpf, 1998; Meier & Mayer, 1994) and macrobicyclic Fe^{III} sequestering agents (Garrett *et al.*,1991). The title compound, (I), of which we report the crystal structure here, can also be used directly to synthesize enterobactin analogues (Weitl *et al.*, 1981).



(I)

The molecular structure of (I) is shown in Fig. 1, and all bond distances are normal. However, there are strong intramolecular O—H···O hydrogen bonds (Table 1), and the short intermolecular O···O weak interaction [2.693 (18) Å] and weak intermolecular O—H···O hydrogen bonds link two molecules into a supramolecular dimer (Table 2 and Fig. 2).

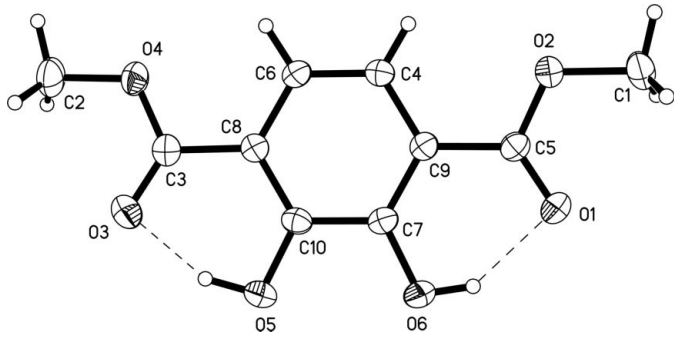


Figure 1
The molecular structure of (I), with displacement ellipsoids drawn at the 30% probability level and H atoms shown as small spheres of arbitrary radius. Intramolecular hydrogen bonds are indicated by dashed lines.

Experimental

To 2,5-dihydroxy-*p*-benzenediacetic acid (4.50 g, 22.7 mmol) was added 50 ml of methanol. The mixture was stirred until most of the diacid was dissolved. After the reaction mixture was cooled to 273 K, thionyl chloride (24.3 g, 20.4 mmol) was added over a period of 30 min. The mixture was stirred at 273 K for 2 h and then allowed to warm to room temperature. After stirring for 12 h at room temperature, the solvent was removed by distillation under reduced pressure. After filtration, the solid was recrystallized from methanol to give the product as a white solid (4.59 g, 89.5%).

Crystal data

$C_{10}H_{10}O_6$
M_r = 226.18
Monoclinic, $P2_1/n$
a = 4.8375 (10) Å
b = 10.691 (2) Å
c = 19.179 (4) Å
β = 92.39 (3)°
V = 991.1 (3) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.13 mm^{-1}
T = 298 (2) K
0.43 × 0.35 × 0.10 mm

Data collection

Bruker SMART APEX II CCD diffractometer
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
T_{min} = 0.947, T_{max} = 0.987
9475 measured reflections
2267 independent reflections
1461 reflections with $I > 2\sigma(I)$
R_{int} = 0.036

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.042
$wR(F^2)$ = 0.112
S = 1.06
2267 reflections
145 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.17 e Å^{-3}
$\Delta\rho_{min}$ = −0.14 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
O5—H5A···O3	0.84	1.88	2.6097 (19)	145
O6—H6B···O1	0.84	1.91	2.6355 (17)	143
O6—H6B···O1^{i}	0.84	2.32	2.9994 (18)	139

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

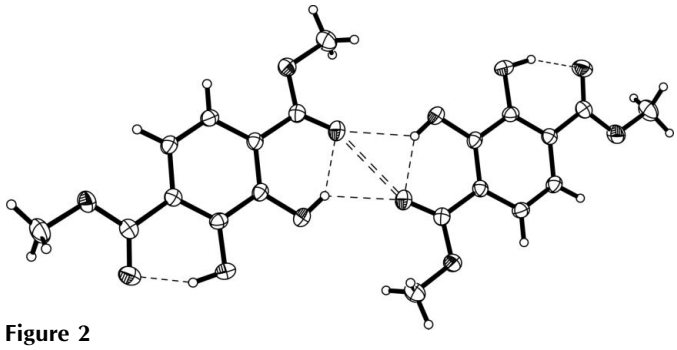


Figure 2
Fig. 2 A view of the supramolecular dimer. Hydrogen bonds are indicated by single dashed lines and the intermolecular O···O weak interaction is indicated by a double dashed line.

All H atoms were positioned geometrically and allowed to ride on their respective parent atoms [C—H = 0.95–0.98 Å, O—H = 0.84 Å and U_{iso}(H) = xU_{eq}(C), with x = 1.2 for aromatic and x = 1.5 for methyl and OH H atoms].

Data collection: *SMART* (Bruker, 2003); cell refinement: *SAINT* (Bruker, 2003); data reduction: *SAINT*; 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: *SHELXTL*.

This work was supported by the Doctoral Foundation of Ningbo University (grant No. 019–012039).

References

Bruker (2003). *SAINT* and *SMART*. Bruker AXS Inc., Madison, Wisconsin, USA.
Garrett, T. M., McMurry, T. J., Hosseini, M. W., Reyes, Z. E., Hahn, E. F. & Raymond, K. N. (1991). *J. Am. Chem. Soc.* **113**, 2965–2977.
Meier, H. & Mayer, A. (1994). *Angew. Chem. Int. Ed. Engl.* **33**, 465–467.
Meier, H. & Rumpf, N. (1998). *Tetrahedron Lett.* **39**, 9639–9642.
Sheldrick, G. M. (1996). *SADABS*. University of Göttingen, Germany.
Sheldrick, G. M. (1997*a*). *SHELXS97 and SHELXL97*. University of Göttingen, Germany.
Sheldrick, G. M. (1997*b*). *SHELXTL*. Bruker AXS Inc., Madison, Wisconsin, USA.
Weitl, F. L., Raymond, K. N. & Durbin, P. W. (1981). *J. Med. Chem.* **24**, 203–206.