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threo-3,4-Dihydroxyhexane-2,5-dione

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Key indicators

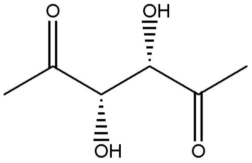
Single-crystal X-ray study
T = 298 K
Mean σ(C–C) = 0.004 Å
R factor = 0.048
wR factor = 0.128
Data-to-parameter ratio = 13.6

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

In the title compound, $C_6H_{10}O_4$, the molecule sits on a twofold axis such that there is one half-molecule in the asymmetric unit. In the crystal structure, there are one intra- and one intermolecular O—H···O hydrogen bonds, resulting in an infinite zigzag chain.

Comment

The natural product 2,5-dimethy-4-hydroxyfuran-3(2*H*)-one (furaneol) has become important in the flavor industry (Zabetakis *et al.*, 1999). The title compound, *threo*-3,4-dihydroxyhexane-2,5-dione, (I), is a key intermediate in the synthesis of furaneol. Both the *threo* and *erythro* forms can lead to the formation of furaneol (Büchi *et al.*, 1973). Assignment of the *threo* and *erythro* forms of 3,4-dihydroxyhexane-2,5-dione was originally based on ^{1}H NMR spectra (Büchi *et al.*, 1973), using comparisons with classical methods for assigning the structures of diols (Carroll, 1966). Assignment using these methods was undoubtedly justified, but we considered that confirmation by an independent method such as an X-ray crystal structure determination was desirable.



(I)

The molecular structure of (I), which has two chiral centers, sits on a twofold axis, as shown in Fig. 1.The molecule is a racemic mixture of *RR* and *SS* enantiomers, confirming the conclusion that it is in the *threo* form. The molecule has an intramolecular hydrogen bond (O2—H2···O1), with an O···O distance of 2.632 (2) Å. Furthermore, an intermolecular hydrogen-bond interaction [O2—H2···O1$^{\rm i}$; symmetry code: (i) $-x+1$, $-y+1$, $-z$] links the molecules into an infinite zigzag chain (Table 2 and Fig. 2).

Experimental

Methylglyoxal (20 g) and acetic acid (20 ml) were stirred under nitrogen while zinc (8 g) dust was added in portions over a period of 1 h. The temperature was kept at 313 K. Three extractions with ethyl acetate gave, after concentration, crude material (7 g). Two recrystallizations from ethyl acetate–petroleum ether gave the title compound. Single crystals of the title compound were obtained by slow evaporation of an ethyl acetate solution.

Crystal data

$C_6H_{10}O_4$
$M_r = 146.14$
Monoclinic, $C2/c$
$a = 17.041\ (4)$ Å
$b = 4.8657\ (10)$ Å
$c = 11.656\ (3)$ Å
$\beta = 130.910\ (3)°$
$V = 730.4\ (3)$ Å^3
$Z = 4$

$D_x = 1.329$ Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 791 reflections
$\theta = 3.2$–$24.5°$
$\mu = 0.11$ mm^{-1}
$T = 298\ (2)$ K
Block, colorless
$0.26 \times 0.25 \times 0.22$ mm

Data collection

Bruker SMART APEX area-detector diffractometer
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Bruker, 2002)
$T_{\rm min} = 0.964$, $T_{\rm max} = 0.976$
1780 measured reflections

652 independent reflections
580 reflections with $I > 2\sigma(I)$
$R_{\rm int} = 0.022$
$\theta_{\rm max} = 25.2°$
$h = -19 \rightarrow 20$
$k = -5 \rightarrow 5$
$l = -13 \rightarrow 10$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.048$
$wR(F^2) = 0.128$
$S = 1.05$
652 reflections
48 parameters
H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.0655P)^2 + 0.5476P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\rm max} < 0.001$
$\Delta\rho_{\rm max} = 0.18$ e Å^{-3}
$\Delta\rho_{\rm min} = -0.16$ e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

O1—C2	1.205 (2)	C1—C2	1.483 (3)
O2—C3	1.402 (2)	C2—C3	1.516 (3)
		C3—C3$^{\rm i}$	1.526 (3)
O1—C2—C1	122.58 (18)	O2—C3—C2	111.88 (16)
O1—C2—C3	118.61 (17)	O2—C3—C3$^{\rm i}$	109.36 (13)
C1—C2—C3	118.78 (17)	C2—C3—C3$^{\rm i}$	111.71 (17)

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

Table 2
Hydrogen-bonding geometry (Å, °).

D—H$\cdots A$	D—H	H$\cdots A$	$D\cdots A$	D—H$\cdots A$
O2—H2$\cdots$O1	0.82	2.18	2.632 (2)	115
O2—H2$\cdots$O1$^{\rm ii}$	0.82	2.24	2.987 (2)	151

Symmetry code: (ii) $1 - x, 1 - y, -z$.

H atoms were included in the refinement at calculated positions in the riding-model approximation [C—H = 0.93 Å and $U_{\rm iso}$(H) = $1.2U_{\rm eq}$(C); O—H = 0.82 Å and $U_{\rm iso}$(H) = $1.5U_{\rm eq}$(O)].

Data collection: *SMART* (Bruker, 2002); cell refinement: *SAINT* (Bruker, 2002); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *XP* in *SHELXTL* ((Bruker, 2002); software used to prepare material for publication: *SHELXL97*.

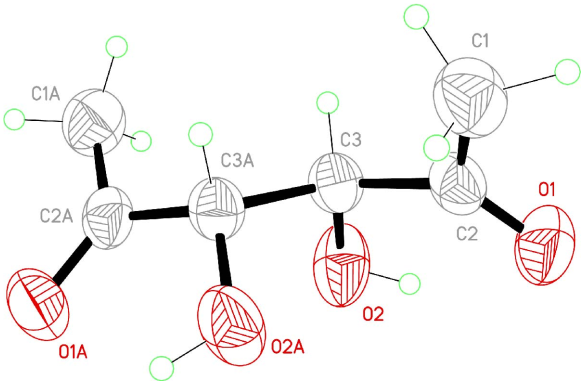


Figure 1
The structure (I), showing the atomic numbering scheme and displacement ellipsoids at the 30% probability level. The suffix A indicates the symmetry operator $1 - x, y, \frac{1}{2} - z$.

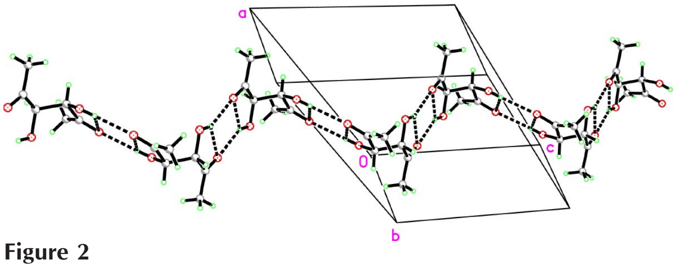


Figure 2
A view of the zigzag chain in (I). Dashed lines indicate hydrogen bonds.

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