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Low-temperature redetermination of *trans*-cyclohexane-1,2-dicarboxylic acid

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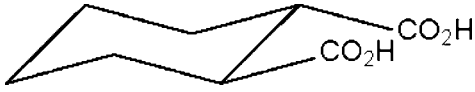
Key indicators: single-crystal X-ray study; T = 100 K; mean σ(C–C) = 0.002 Å; R factor = 0.042; wR factor = 0.115; data-to-parameter ratio = 15.0.

The molecule of the title compound, $C_8H_{12}O_4$, lies on a twofold rotation axis that passes through the mid-points of two opposite C—C bonds of the ring. Carboxyl groups of adjacent molecules are linked by pairs of hydrogen bonds around a centre of inversion; this interaction gives rise to a chain that runs along [101].

Related literature

Studies on the metal derivatives of *trans*-1,2-cyclohexanedicarboxylic acid refer to the room-temperature structure of Benedetti *et al.* (1969). The absence of a preferred orientation (either axial or equatorial) of the carboxyl groups in cyclohexanedicarboxylic acids is discussed in the case of 1,3-cyclohexanedicarboxylic acid by van Koningsveld (1984). For the crystal structure of 1,4-cyclohexanedicarboxylic acid, see: Luger *et al.* (1972).



Experimental

Crystal data

$C_8H_{12}O_4$
M_r = 172.18
Monoclinic, $C2/c$
a = 5.585 (1) Å
b = 13.840 (3) Å
c = 10.035 (2) Å
β = 96.114 (3)°
V = 771.3 (3) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.12 mm^{-1}
T = 100 (2) K
0.38 × 0.06 × 0.04 mm

Data collection

Bruker SMART APEX diffractometer
Absorption correction: none
2320 measured reflections
883 independent reflections
715 reflections with $I > 2\sigma(I)$
R_{int} = 0.035

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.042
$wR(F^2)$ = 0.115
S = 1.07
883 reflections
59 parameters
1 restraint
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.31 e Å^{-3}
$\Delta\rho_{min}$ = −0.28 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
O1—H1o···O2^{i}	0.85 (1)	1.81 (1)	2.662 (2)	178 (2)

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

Data collection: *APEX2* (Bruker, 2007); cell refinement: *SAINT* (Bruker, 2007); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *X-SEED* (Barbour, 2001); software used to prepare material for publication: *publCIF* (Westrip, 2008).

We thank the University of Malaya for the purchase of the diffractometer.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: CV2404).

References

Barbour, L. J. (2001). *J. Supramol. Chem.* **1**, 189–191.
Benedetti, E., Corradini, P., Perrone, C. & Post, B. (1969). *J. Am. Chem. Soc.* **91**, 4072–4074.
Bruker (2007). *APEX2* and *SAINT*. Bruker AXS Inc., Madison, Wisconsin, USA.
Koningsveld, H. van (1984). *Acta Cryst.* C**40**, 1857–1863.
Luger, P., Plieth, K. & Ruban, G. (1972). *Acta Cryst.* B**28**, 706–710.
Sheldrick, G. M. (2008). *Acta Cryst.* A**64**, 112–122.
Westrip, S. P. (2008). *publCIF*. In preparation.

supporting information

Acta Cryst. (2008). E**64**, o992 [doi:10.1107/S1600536808012592]

Low-temperature redetermination of *trans*-cyclohexane-1,2-dicarboxylic acid

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S1. Comment

Crystallographic studies of the metal derivatives of *trans*-1,2-cyclohexanedicarboxylic acid occasionally refer to the room-temperature crystal structure of the dicarboxylic acid, which was reported in 1969. The report (Benedetti *et al.*, 1969) contains typographical errors that have since been corrected in the Cambridge Structural Database (Version 5.29, Nov. 2007). The reported monoclinic cell dimensions can be transformed to 5.65 (1), *b* 13.34 (3), *c* 10.22 (3) Å; *β* 97.2 (2)°.

Whereas the low-temperature unit cell has a slightly larger volume compared with the room-temperature cell, the low-temperature cell has a much longer *b*-axis [13.840 (3) Å]. The bond distances and angles of room-temperature structure are normal; those of the present study are not significantly different despite the longer axis. Possibly, the expansion of this axis is a genuine observation. Moreover, the present study is able to establish the hydrogen bonding scheme of the compound (Scheme I, Fig. 1). Adjacent molecules are linked by a linear O–H···O hydrogen bond [2.662 (2) Å] into a chain (Fig. 2).

The crystal structures of 1,3- and 1,4-cyclohexanedicarboxylic acids have already been reported (van Koningsveld, 1984; Luger *et al.*, 1972).

S2. Experimental

The commercially available acid was recrystallized from ethanol.

S3. Refinement

Carbon-bound H-atoms were placed in calculated positions (C—H 0.99 to 1.00 Å) and were included in the refinement in the riding model approximation, with *U*(H) set to 1.2 *U*(C). The acid H-atom was located in a difference Fourier map, and was isotropically refined with a distance restraint of O–H 0.85 (1) Å.

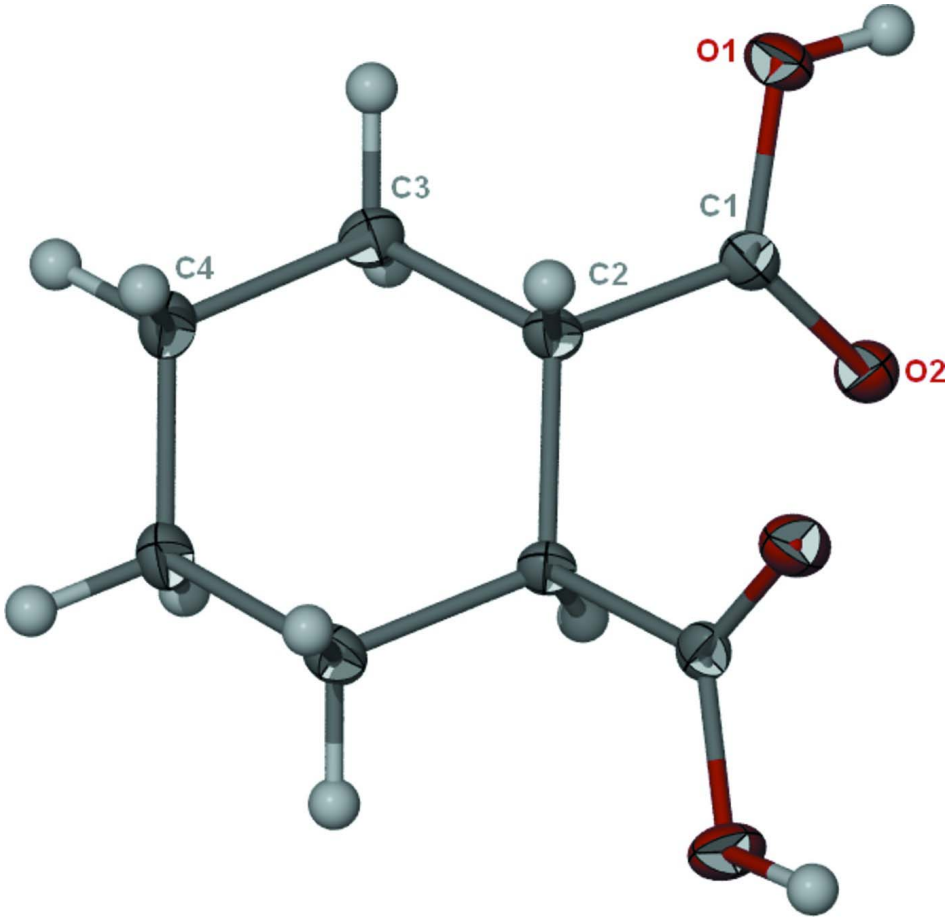

Figure 1

The molecular structure of the title compound with atomic numbering and 70% probability displacement ellipsoids. Hydrogen atoms are drawn as spheres of arbitrary radiius. The unlabeled atoms are related to the labeled ones by 1 - *x*, *y*, 1/2 - *z*.

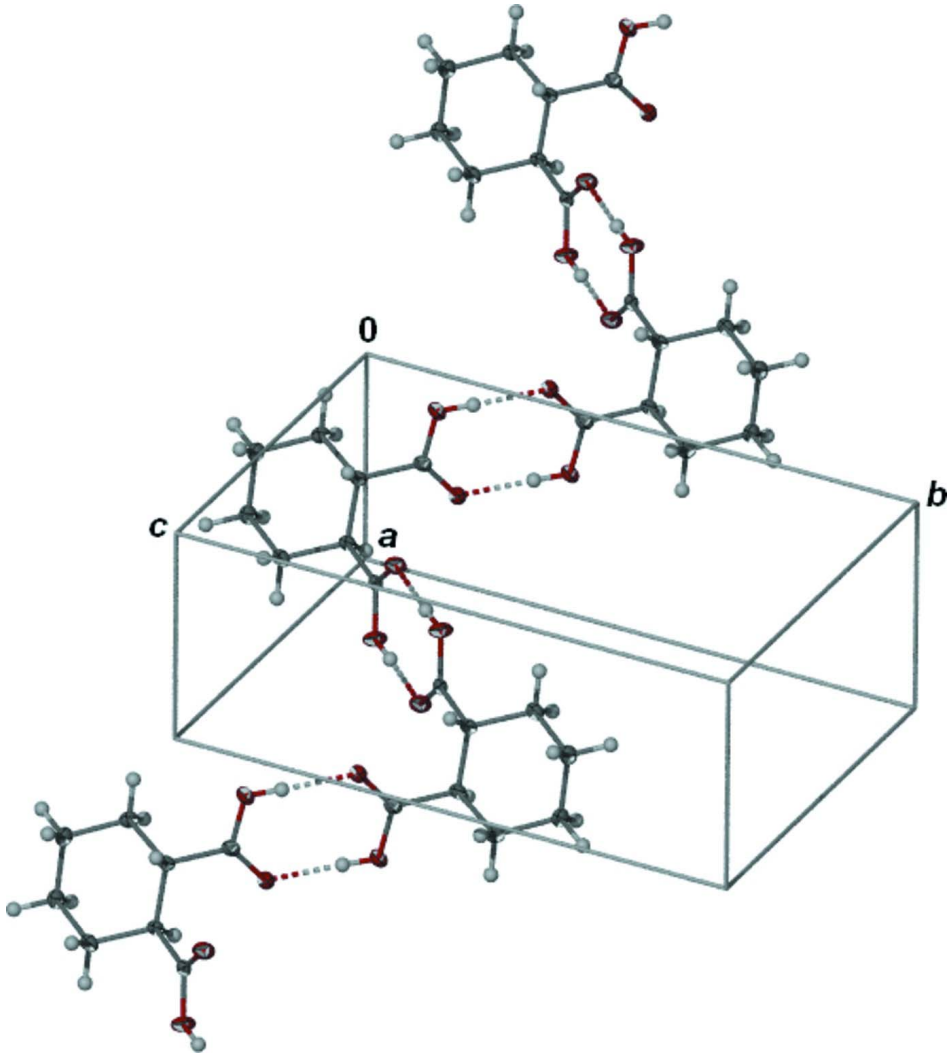

Figure 2

A portion of the crystal packing showing the hydrogen-bonded (dashed lines) chain.

trans-cyclohexane-1,2-dicarboxylic acid

Crystal data

$C_8H_{12}O_4$
$M_r = 172.18$
Monoclinic, $C2/c$
Hall symbol: -C 2yc
$a = 5.585$ (1) Å
$b = 13.840$ (3) Å
$c = 10.035$ (2) Å
$\beta = 96.114$ (3)°
$V = 771.3$ (3) Å^3
$Z = 4$
$F(000) = 368$
$D_x = 1.483$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 739 reflections
$\theta = 3.6$–28.2°
$\mu = 0.12$ mm^{-1}
$T = 100$ K
Strip, colourless
0.38 × 0.06 × 0.04 mm

Data collection

Bruker SMART APEX diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
ω scans
2320 measured reflections
883 independent reflections
715 reflections with $I > 2\sigma(I)$
$R_{int} = 0.035$
$\theta_{max} = 27.5°$, $\theta_{min} = 2.9°$
$h = -7 \rightarrow 7$
$k = -17 \rightarrow 17$
$l = -13 \rightarrow 8$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.042$
$wR(F^2) = 0.115$
$S = 1.07$
883 reflections
59 parameters
1 restraint
Primary atom site location: structure-invariant direct methods
Secondary atom site location: difference Fourier map
Hydrogen site location: inferred from neighbouring sites
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0635P)^2 + 0.2009P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.001$
$\Delta\rho_{max} = 0.31$ e Å^{-3}
$\Delta\rho_{min} = -0.28$ e Å^{-3}

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²)

	x	*y*	*z*	U_{iso}*/U_{eq}
O1	0.0840 (2)	0.1596 (1)	0.0902 (1)	0.0173 (3)
O2	0.4725 (2)	0.1968 (1)	0.0869 (1)	0.0166 (3)
C1	0.3157 (3)	0.1471 (1)	0.1279 (2)	0.0122 (3)
C2	0.3668 (2)	0.0626 (1)	0.2220 (2)	0.0122 (4)
C3	0.2893 (3)	−0.0314 (1)	0.1475 (2)	0.0139 (4)
C4	0.3647 (3)	−0.1208 (1)	0.2303 (2)	0.0159 (4)
H1o	0.068 (4)	0.206 (1)	0.035 (2)	0.036 (6)*
H2	0.2676	0.0704	0.2986	0.015*
H3a	0.1121	−0.0315	0.1261	0.017*
H3b	0.3626	−0.0339	0.0620	0.017*
H4a	0.2799	−0.1218	0.3120	0.019*
H4b	0.3184	−0.1796	0.1776	0.019*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.0124 (6)	0.0179 (6)	0.0205 (7)	0.0008 (4)	−0.0027 (5)	0.0074 (5)
O2	0.0149 (6)	0.0153 (6)	0.0186 (6)	−0.0017 (4)	−0.0028 (4)	0.0048 (4)
C1	0.0140 (7)	0.0112 (7)	0.0107 (8)	0.0012 (5)	−0.0025 (6)	−0.0034 (6)
C2	0.0115 (7)	0.0118 (7)	0.0126 (8)	0.0003 (5)	−0.0025 (6)	−0.0004 (6)
C3	0.0138 (7)	0.0141 (7)	0.0134 (8)	−0.0013 (5)	−0.0012 (6)	−0.0014 (6)
C4	0.0170 (8)	0.0109 (7)	0.0190 (9)	−0.0011 (5)	−0.0012 (6)	0.0003 (6)

Geometric parameters (Å, º)

O1—C1	1.321 (2)	O1—H1o	0.85 (1)
O2—C1	1.220 (2)	C2—H2	1.0000
C1—C2	1.511 (2)	C3—H3a	0.9900
C2—C2^{i}	1.533 (3)	C3—H3b	0.9900
C2—C3	1.5397 (19)	C4—H4a	0.9900
C3—C4	1.523 (2)	C4—H4b	0.9900
C4—C4^{i}	1.521 (3)		
O2—C1—O1	123.1 (1)	C3—C2—H2	108.3
O2—C1—C2	123.6 (1)	C4—C3—H3a	109.2
O1—C1—C2	113.3 (1)	C2—C3—H3a	109.2
C1—C2—C2^{i}	109.9 (1)	C4—C3—H3b	109.2
C1—C2—C3	109.0 (1)	C2—C3—H3b	109.2
C2^{i}—C2—C3	112.9 (1)	H3a—C3—H3b	107.9
C4—C3—C2	112.0 (1)	C4^{i}—C4—H4a	109.5
C4^{i}—C4—C3	110.5 (1)	C3—C4—H4a	109.5
C1—O1—H1o	109 (1)	C4^{i}—C4—H4b	109.5
C1—C2—H2	108.3	C3—C4—H4b	109.5
C2^{i}—C2—H2	108.3	H4a—C4—H4b	108.1
O2—C1—C2—C2^{i}	11.2 (2)	C1—C2—C3—C4	172.7 (1)
O1—C1—C2—C2^{i}	−171.2 (1)	C2^{i}—C2—C3—C4	50.2 (2)
O2—C1—C2—C3	−113.0 (2)	C2—C3—C4—C4^{i}	−56.6 (2)
O1—C1—C2—C3	64.6 (2)		

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

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O1—H1*o*···O2^{ii}	0.85 (1)	1.81 (1)	2.662 (2)	178 (2)

Symmetry code: (ii) −*x*+1/2, −*y*+1/2, −*z*.