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Redetermination of *cis*-4-cyclohexene-1,2-dicarboxylic acid at 173 K

Michael Bolte* and Alexander Degen

Institut für Organische Chemie, J. W. Goethe-Universität Frankfurt, Marie-Curie-Strasse 11, 60439 Frankfurt/Main, Germany
Correspondence e-mail: bolte@chemie.uni-frankfurt.de

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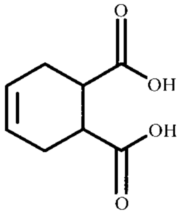
The structure of the title compound, $C_8H_{10}O_4$, previously determined by Küppers & Kim [*Acta Cryst.* (1993), C**49**, 1218–1220] has been redetermined at 173 K. The cyclohexene ring exhibits a half-chair conformation. The molecular geometry and the crystal packing, which is stabilized by two hydrogen bonds, agree well with the room-temperature structure determination.

Comment

The crystal structure of the title compound, (I), was determined in order to establish unambiguously the nature of a reaction product. The molecular geometry and the crystal structure of the title compound agree well with the structure determined at room temperature (Küppers & Kim, 1993). A least-squares fit of all non-H atoms shows an r.m.s. deviation of 0.014 Å. The cyclohexene ring exhibits a half-chair conformation, with atoms C3, C4, C5 and C6 in a common plane (σ = 0.003 Å), and C1 0.326 (5) Å above and C2 0.400 (5) Å below that plane. The cell axes are a little shorter at 173 K, and the anisotropic displacement parameters are, as expected, smaller. However, there are no significant differences between the structures at different temperatures. The crystal packing is stabilized by two hydrogen bonds (Table 1).



(I)

Experimental

The title compound was synthesized according to the method of Hünig *et al.* (1979) in two steps. A solution of freshly sublimed maleic anhydride (19.6 g, 200 mmol) in dried benzene (50 ml) was heated to approximately 350 K. Butadiene was then passed into the solution for 30 min with continuous stirring. The reaction product, 4-cyclohexene-1,2-dicarboxylic acid anhydride, was washed with petroleum ether and recrystallized from diethyl ether. The second step comprised the hydrolysis of the anhydride. 4-Cyclohexene-1,2-dicarboxylic acid anhydride (1 g) was dissolved in water (10 ml) and refluxed under a take-off condenser for 2 h. When the solution was cooled down to room temperature the product precipitated. Recrystallization from ethyl acetate yielded crystals suitable for an X-ray structure analysis.

Crystal data

$C_8H_{10}O_4$
M_r = 170.16
Triclinic, $P\bar{1}$
a = 6.308 (4) Å
b = 6.892 (4) Å
c = 10.875 (7) Å
α = 74.57 (1)°
β = 73.78 (1)°
γ = 64.41 (1)°
V = 403.6 (4) Å^3
Z = 2
D_x = 1.400 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 2610 reflections
θ = 0–25°
μ = 0.113 mm^{-1}
T = 173 K
Block, colourless
0.25 × 0.15 × 0.15 mm

Data collection

Siemens CCD three-circle diffractometer
ω scans
Absorption correction: empirical (*SADABS*; Sheldrick, 1996)
$T_{\rm min}$ = 0.972, $T_{\rm max}$ = 0.983
4809 measured reflections
1645 independent reflections
1151 reflections with $I > 2\sigma(I)$
$R_{\rm int}$ = 0.043
$\theta_{\rm max}$ = 26.37°
$h = -7 \rightarrow 7$
$k = -8 \rightarrow 8$
$l = -13 \rightarrow 13$
503 standard reflections
frequency: 1100 min
intensity decay: none

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.052
$wR(F^2)$ = 0.127
S = 1.078
1645 reflections
112 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0414P)^2 + 0.3350P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\rm max}$ < 0.001
$\Delta\rho_{\rm max}$ = 0.29 e Å^{-3}
$\Delta\rho_{\rm min}$ = −0.23 e Å^{-3}
Extinction correction: *SHELXL*97
Extinction coefficient: 0.068 (11)

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

D—H···A	D—H	H···A	D···A	D—H···A
O12—H12···O11$^{\rm i}$	0.84	1.83	2.667 (3)	172
O22—H22···O21$^{\rm ii}$	0.84	1.81	2.645 (3)	176

Symmetry codes: (i) $1-x, 1-y, -z$; (ii) $1-x, 1-y, 1-z$.

All H atoms were located by difference Fourier synthesis and refined with U(H) = 1.2$U_{\rm eq}$(C) or U(H) = 1.2$U_{\rm eq}$(O) using a riding model with Csp^2—H = 0.95, C—H(secondary) = 0.99, C—H(tertiary) = 1.00 and O—H = 0.84 Å. The OH groups were allowed to rotate about the C—O axis.

Data collection: *SMART* (Siemens, 1995); cell refinement: *SMART*; data reduction: *SAINT* (Siemens, 1995); program(s) used to solve structure: *SHELXS*97 (Sheldrick, 1990); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997).

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