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Oxacyclohexane-2,6-dione (glutaric anhydride)

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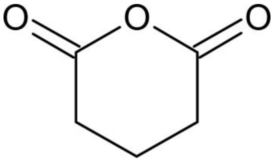
Key indicators: single-crystal X-ray study; T = 240 K; mean σ(C–C) = 0.003 Å; R factor = 0.041; wR factor = 0.116; data-to-parameter ratio = 10.6.

In the title compound, $C_5H_6O_3$, the ring adopts a slightly distorted envelope conformation. Adjacent molecules are linked *via* weak C—H···O contacts. Two phase transitions at temperatures of 154/183 and 172/192 K are observed for the title compound in a differential scanning calorimetry experiment; these occur below the structure determination temperature of 240 K.

Related literature

For related literature, see: Bertolasi *et al.* (1997); Bocelli & Grenier-Loustalot (1982); Cremer & Pople (1975); Freer *et al.* (1988); Koer *et al.* (1972); Qian *et al.* (2006); Stefanowicz (2006); Takahashi *et al.* (2004).



Experimental

Crystal data

$C_5H_6O_3$
M_r = 114.10
Orthorhombic, $P2_12_12_1$
a = 5.410 (4) Å
b = 7.520 (6) Å
c = 13.180 (8) Å
V = 536.2 (7) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.12 mm^{-1}
T = 240 (2) K
0.53 × 0.15 × 0.15 mm

Data collection

Kuma KM-4 CCD κ-geometry diffractometer
Absorption correction: none
3600 measured reflections
774 independent reflections
668 reflections with $I > 2\sigma(I)$
R_{int} = 0.034

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.041
$wR(F^2)$ = 0.116
S = 1.14
774 reflections
73 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.22 e Å^{-3}
$\Delta\rho_{min}$ = −0.11 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
C3—H3B···O2^i	0.98	2.53	3.353 (4)	142

Symmetry code: (i) $-x+1, y-\frac{1}{2}, -z+\frac{3}{2}$.

Data collection: *CrysAlis CCD* (Oxford Diffraction, 2003); cell refinement: *CrysAlis RED* (Oxford Diffraction, 2003); data reduction: *CrysAlis RED*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *PLATON* (Spek, 2003) and *XP* (Bruker, 1998); software used to prepare material for publication: *SHELXL97*.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: BX2098).

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supplementary materials

Acta Cryst. (2007). E**63**, o3431 [**doi:10.1107/S1600536807032977**]

Oxacyclohexane-2,6-dione (glutaric anhydride)

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Comment

The title compound (I) (Fig. 1) and some of its derivatives are commonly used as reagents in the organic synthesis (Freer et al., 1988; Takahashi et al., 2004). Several papers concerning the crystal state studies of glutaric anhydride derivatives, as 3-phenyl-1-oxacyclohexane-2,6-dione or β-chloroglutaric acid anhydride are available in the literature (Koer et al., 1972; Bocelli & Grenier-Loustalot, 1982), but surprisingly the crystal structure of the glutaric anhydride has not been determined yet.

This paper reports the crystal state studies of the glutaric anhydride. Compound I crystallizes in the i>$P2_12_12_1$ space group with one independent molecule in the asymmetric unit.

The C1=O3 and C5=O2 bond lengths of 1.197 (3) Å and 1.188 (3) Å, respectively, remain in good agreement with those C=O bond lengths observed in related compounds crystal structures (Koer et al., 1972; Bocelli & Grenier-Loustalot, 1982; Qian et al., 2006). The differences in the C=O bonds lengths existing between discussed structures are similar to those observed for two independent molecules of β-chloroglutaric acid anhydride (Koer et al., 1972) and can be justified by the different intermolecular interactions, present in the crystal network. The C1—O1—C3 angle of 124.5 (2)° remains also in agreement with the numerous literature data (Koer et al., 1972; Bocelli & Grenier-Loustalot, 1982; Bertolasi et al., 1997; Qian et al., 2006). The six-membered ring of glutaric anhydride molecule adopts nearly envelope conformation, what is confirmed by the values of puckering parameters: q_2 = 0.375 (2) Å, q_3 = 0.251 (2) Å and ψ_2= 174.5 (4)° (Cremer & Pople, 1975) for the O1/C1/C2/C3/C4/C5 ring atom sequence. The O1, C1, C2, C4 and C5 atoms are coplanar (r.m.s. deviation = 0.0188), and C3 carbon is deviated from the plane defined by above atoms by −0.625 (4) Å.

In glutaric anhydride crystal structure no hydrogen bonds are observed. Only very weak interactions as C—H···O contacts between adjacent molecules can be recognized (Table 2). Molecular packing (Fig. 2), which exists in the glutaric anhydride crystals can explain the low value of the melting point.

Experimental

The title compound was obtained (Stefanowicz, 2006) by the dehydratation of the glutaric acid in the acetic anhydride solution. The glutaric acid (30 g; 0,227 mol) was added to the 25 ml of the acetic anhydride. Obtained mixture was heated slowly until the boiling point was reached, and then refluxed for 15 minutes. Later the solvent was removed in a vacuo. Treatment of the obtained oil residue with naphthyl ether (200 ml) and later washing with the n-hexane resulted in the single crystals suitable for X-ray measurements. Glutaric anhydride undergoes the phase transitions, what was confirmed by the DSC technique. DSC studies on (I) disclosed two closely laying phase transitions at 154/183 K (cooling-heating) and 172/192 K. These phase transitions characterized by a significant temperature hysteresis may be classified as discontinuous ones. They are accompanied by a relatively small entropy effects: ΔS = 0.36 J/(mol K) and 0.20 J/(mol K).

Refinement

The structure was solved by direct methods with using *SHELXS97*. The H atoms were placed in the idealized positions as riding on their parent atoms with distances of 0.98 Å with U_{iso}(H) values of $1.2U_{\mathrm{eq}}$(C). Although the molecule is achiral, the structure possesses a polar axis. The absence of atoms, which possess the atomic number higher than silicon causes that no anomoulus dispersion is observed. The Flack [(1983). *Acta Cryst.* A**39**, 876–881] parameter is meanigless in this case. The absolute direction of the polar axis was assigned arbitrarily and the Friedel pairs were merged before the final refinement.

Figures

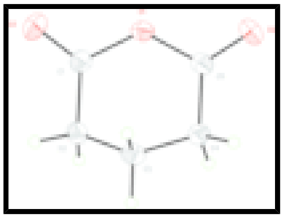

Fig. 1. *ORTEP* atom numbering scheme of I. The thermal ellipsoides were drawn at 20% probability.

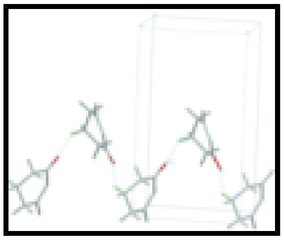

Fig. 2. The view of the packing of I, viewed along the *a* axis. Atom O2 is at ($1 - x$,$-1/2 + y$,$3/2 - z$).

Oxacyclohexane-2,6-dione

Crystal data

$C_5H_6O_3$
$M_r = 114.10$
Orthorhombic, $P2_12_12_1$
Hall symbol: P 2ac 2ab
$a = 5.410$ (4) Å
$b = 7.520$ (6) Å
$c = 13.180$ (8) Å
$V = 536.2$ (7) Å^3
$Z = 4$

$F_{000} = 240$
$D_x = 1.413$ Mg m^{-3}
Mo $K\alpha$ radiation
$\lambda = 0.71073$ Å
Cell parameters from 668 reflections
$\theta = 3.1$–$28.5°$
$\mu = 0.12$ mm^{-1}
$T = 240$ (2) K
Needle, colourless
0.53 × 0.15 × 0.15 mm

Data collection

KUMA KM-4 CCD κ-geometry diffractometer
Radiation source: fine-focus sealed tube
Monochromator: graphite

668 reflections with $I > 2\sigma(I)$
$R_{\mathrm{int}} = 0.034$
$\theta_{\max} = 28.5°$

$T = 240(2)$ K	$\theta_{\min} = 3.1°$
ω and φ scans	$h = -7 \to 5$
Absorption correction: none	$k = -9 \to 10$
3600 measured reflections	$l = -17 \to 16$
774 independent reflections	

Refinement

Refinement on F^2	H-atom parameters constrained
Least-squares matrix: full	$w = 1/[\sigma^2(F_o^2) + (0.0681P)^2 + 0.0184P]$ where $P = (F_o^2 + 2F_c^2)/3$
$R[F^2 > 2\sigma(F^2)] = 0.041$	$(\Delta/\sigma)_{\max} < 0.001$
$wR(F^2) = 0.116$	$\Delta\rho_{\max} = 0.22$ e Å^{-3}
$S = 1.14$	$\Delta\rho_{\min} = -0.10$ e Å^{-3}
774 reflections	Extinction correction: none
73 parameters	
Primary atom site location: structure-invariant direct methods	
Secondary atom site location: difference Fourier map	
Hydrogen site location: inferred from neighbouring sites	

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å^2)

	x	y	z	$U_{\rm iso}$*/$U_{\rm eq}$
O1	0.5092 (2)	0.6331 (2)	0.60650 (11)	0.0495 (4)
C1	0.5835 (4)	0.5529 (3)	0.51710 (16)	0.0471 (5)
O3	0.4401 (3)	0.5620 (2)	0.44814 (12)	0.0712 (6)
C2	0.8311 (4)	0.4690 (3)	0.51408 (16)	0.0504 (5)
H2A	0.9512	0.5552	0.4880	0.060*
H2B	0.8270	0.3681	0.4671	0.060*
C5	0.6538 (4)	0.6438 (3)	0.69384 (16)	0.0517 (5)
O2	0.5715 (4)	0.7309 (3)	0.76100 (13)	0.0813 (7)
C4	0.8970 (4)	0.5521 (3)	0.69368 (16)	0.0530 (5)
H4A	0.9273	0.5026	0.7613	0.064*
H4B	1.0269	0.6398	0.6799	0.064*
C3	0.9151 (4)	0.4048 (3)	0.61660 (17)	0.0545 (6)
H3A	1.0865	0.3634	0.6122	0.065*
H3B	0.8121	0.3044	0.6381	0.065*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.0396 (7)	0.0576 (9)	0.0512 (8)	0.0085 (7)	0.0013 (6)	−0.0010 (7)
C1	0.0472 (10)	0.0467 (10)	0.0475 (10)	−0.0027 (10)	−0.0023 (9)	0.0008 (9)
O3	0.0670 (11)	0.0888 (13)	0.0577 (10)	0.0057 (11)	−0.0200 (9)	−0.0027 (9)
C2	0.0518 (12)	0.0504 (11)	0.0489 (11)	0.0006 (10)	0.0030 (10)	−0.0099 (10)

C5	0.0465 (10)	0.0605 (13)	0.0479 (11)	0.0054 (10)	0.0058 (10)	−0.0034 (11)
O2	0.0701 (12)	0.1111 (15)	0.0627 (11)	0.0210 (12)	0.0087 (10)	−0.0317 (11)
C4	0.0471 (11)	0.0664 (13)	0.0455 (10)	0.0088 (11)	−0.0034 (9)	0.0005 (11)
C3	0.0493 (11)	0.0535 (12)	0.0606 (12)	0.0103 (10)	0.0033 (11)	0.0022 (10)

Geometric parameters (Å, °)

O1—C1	1.383 (3)	C5—O2	1.188 (3)
O1—C5	1.394 (3)	C5—C4	1.485 (3)
C1—O3	1.197 (3)	C4—C3	1.506 (3)
C1—C2	1.481 (3)	C4—H4A	0.9800
C2—C3	1.505 (3)	C4—H4B	0.9800
C2—H2A	0.9800	C3—H3A	0.9800
C2—H2B	0.9800	C3—H3B	0.9800
C1—O1—C5	124.43 (15)	C5—C4—C3	113.55 (18)
O3—C1—O1	115.69 (19)	C5—C4—H4A	108.9
O3—C1—C2	126.2 (2)	C3—C4—H4A	108.9
O1—C1—C2	118.13 (17)	C5—C4—H4B	108.9
C1—C2—C3	112.67 (19)	C3—C4—H4B	108.9
C1—C2—H2A	109.1	H4A—C4—H4B	107.7
C3—C2—H2A	109.1	C2—C3—C4	110.50 (18)
C1—C2—H2B	109.1	C2—C3—H3A	109.6
C3—C2—H2B	109.1	C4—C3—H3A	109.6
H2A—C2—H2B	107.8	C2—C3—H3B	109.6
O2—C5—O1	115.9 (2)	C4—C3—H3B	109.6
O2—C5—C4	126.1 (2)	H3A—C3—H3B	108.1
O1—C5—C4	117.99 (18)		

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C3—H3B···O2$^{\mathrm{i}}$	0.98	2.53	3.353 (4)	142

Symmetry codes: (i) $-x+1, y-1/2, -z+3/2$.

Fig. 1

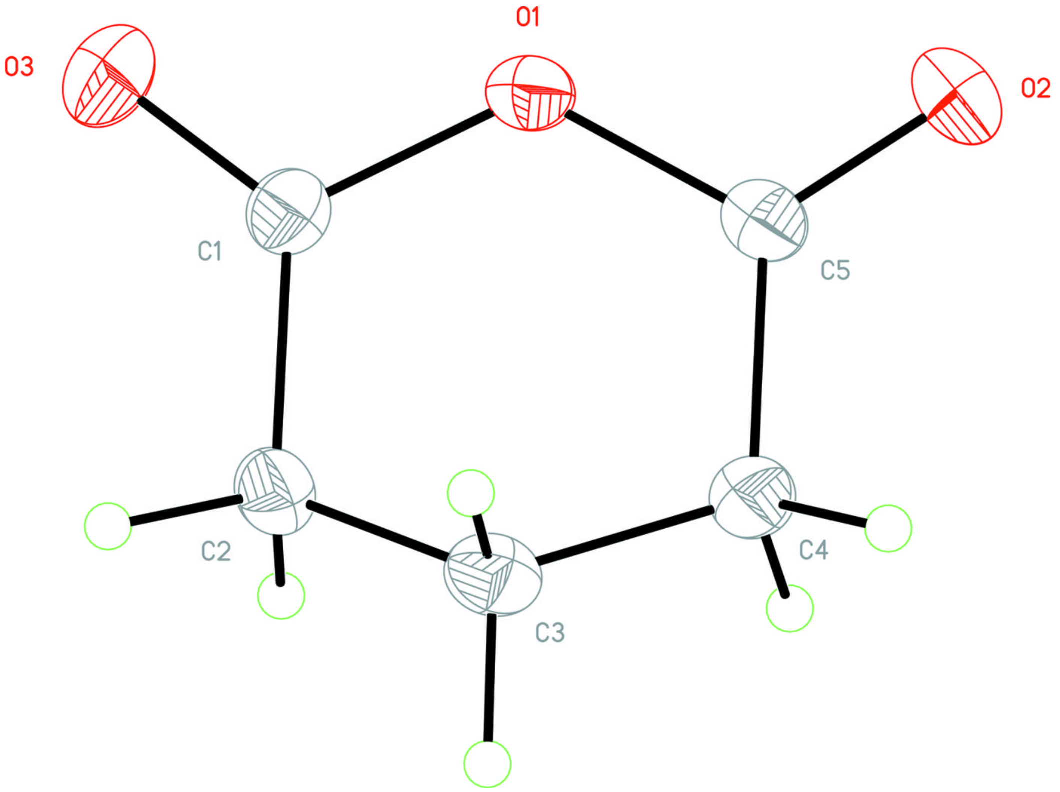

Fig. 2