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(2*R*,3*R*)-Diacetyltartaric acid anhydride

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Key indicators

Single-crystal X-ray study
T = 293 K
Mean σ(C–C) = 0.003 Å
R factor = 0.035
wR factor = 0.100
Data-to-parameter ratio = 10.0

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

The title compound, $C_8H_8O_7$, was synthesized from tartaric acid and acetic anhydride. The structure is stabilized by an intermolecular hydrogen bond from an H atom of a methyl group to a carbonyl O atom on the five-membered ring.

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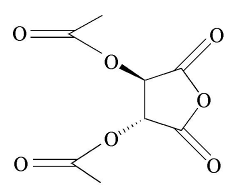
Comment

Diacetyltartaric acid anhydride, (I), is an important intermediate in the synthesis of anionic oil-in-water emulsifiers, such as the diacetyltartaric esters of monoglycerides (DATEMs), which are used throughout the world as improvers in bread-making (Köhler & Grosch, 1999). This compound has also been used in studies of the kinetic acylation of various racemic alcohols and amines, a process that sometimes helps to separate racemates into their pure enantiomeric components (Mravik *et al.*, 1996). Compound (I) was prepared by the reaction of tartaric acid and acetic anhydride (Ireland & Thompson, 1979). The crystal structure determination of the title compound was carried out in order to elucidate its molecular conformation and is reported here.



(I)

The molecular structure of (I) is shown in Fig. 1. Atoms C3 and C2 have distorted tetrahedral geometry, with the C4—C3—C2 [103.49 (16)°] and C1—C2—C3 [102.46 (15)°] angles deviating significantly from ideal tetrahedral values. Because they are in a five-membered ring, the C3—C4—O1 [108.04 (15)°] and C2—C1—O1 [109.28 (16)°] angles are significantly less than the normal (120°) trigonal value, as are the C8—C7—O6 [110.5 (2)°] and C6—C5—O4 [110.4 (2)°] angles in the acetyl groups. Weak C—H···O intermolecular hydrogen bonds (Table 2) link a row of molecules into a zigzag chain along the *b* axis (Fig. 2). There are several short non-bonded approaches between carbonyl groups in this crystal structure, the shortest being O7···C4, at 2.79 (2) Å. Similar short approaches have been reported in other acid anhydride crystal structures; an O···C approach of only 2.75 Å was reported by Jefford *et al.* (1992).

Experimental

To a mixture of pulverized commercial tartaric acid (10 g, 67 mmol) and acetic anhydride (22 ml) was added concentrated sulfuric acid

(1 ml), and the resulting solution was stirred at room temperature for 3 h. After the solution had been heated on a steam bath for a few minutes, the reaction was cooled in an ice bath, and the white crystalline product was collected by vacuum filtration on a medium frit. The filtercake was washed with benzene (5 ml) and dried in a vacuum desiccator over paraffin for three days, yielding pure crystalline diacetyltartaric anhydride (yield 13.2 g, 92%; m.p. 406–408 K). IR (KBr, νcm^{-1}): 2940, 1900, 1826, 1763, 1380; ^{1}H NMR ($CDCl_3$, p.p.m.): δ 2.22 (*s*, 6H), 5.68 (*s*, 2H).

Crystal data

$C_8H_8O_7$
M_r = 216.14
Orthorhombic, $P2_12_12_1$
a = 5.4066 (7) Å
b = 10.3363 (14) Å
c = 17.434 (2) Å
V = 974.3 (2) Å^3
Z = 4
D_x = 1.474 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 1835 reflections
θ = 2.3–21.9°
μ = 0.13 mm^{-1}
T = 293 (2) K
Prism, colourless
0.24 × 0.20 × 0.18 mm

Data collection

Bruker SMART CCD area-detector diffractometer
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
T_{min} = 0.969, T_{max} = 0.976
6584 measured reflections
1381 independent reflections
1093 reflections with $I > 2\sigma(I)$
R_{int} = 0.023
θ_{max} = 27.9°
$h = -5 \rightarrow 7$
$k = -13 \rightarrow 13$
$l = -22 \rightarrow 22$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.035
$wR(F^2)$ = 0.100
S = 1.07
1381 reflections
138 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0644P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ < 0.001
$\Delta\rho_{max}$ = 0.13 e Å^{-3}
$\Delta\rho_{min}$ = −0.17 e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

O2—C1	1.176 (2)	O5—C5	1.191 (3)
O3—C4	1.175 (2)	O7—C7	1.180 (3)
O1—C1—C2	109.28 (16)	O1—C4—C3	108.04 (15)
C3—C2—C1	102.46 (15)	O4—C5—C6	110.4 (2)
C2—C3—C4	103.49 (16)	O6—C7—C8	110.5 (2)
C4—O1—C1—C2	−5.8 (2)	C1—O1—C4—C3	−10.9 (2)
O1—C1—C2—C3	19.7 (2)	C2—C3—C4—O1	22.8 (2)
C1—C2—C3—C4	−24.66 (19)		

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

D—H···A	D—H	H···A	D···A	D—H···A
C8—H8A···O3^{i}	0.96	2.54	3.412 (4)	151
C6—H6C···O5^{ii}	0.96	2.60	3.528 (4)	162
C2—H2···O7^{iii}	0.98	2.60	3.109 (3)	112
C3—H3···O7	0.98	2.24	2.631 (3)	102
C2—H2···O5	0.98	2.27	2.634 (3)	101

Symmetry codes: (i) $-x, y+\frac{1}{2}, -z+\frac{1}{2}$; (ii) $x-1, y, z$; (iii) $x+1, y, z$.

There is no firm chemical evidence for the assignment of the absolute configuration of this compound, which has probably undergone spontaneous resolution during crystallization. In the absence of significant anomalous dispersion effects, Friedel pairs were averaged. All H atoms were positioned geometrically (with C—H = 0.96–0.98 Å) and were refined with riding constraints. For the methyl groups, U_{iso}(H) values were set to $1.5U_{eq}$(carrier atom) and for the CH groups they were set to $1.2U_{eq}$(carrier atom).

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

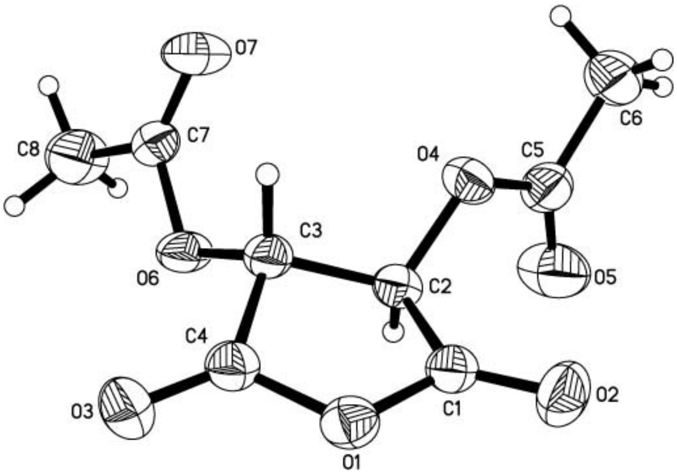


Figure 1
A view of the molecule of (I), showing the atom-labelling scheme. Displacement ellipsoids are drawn at the 35% probability level.

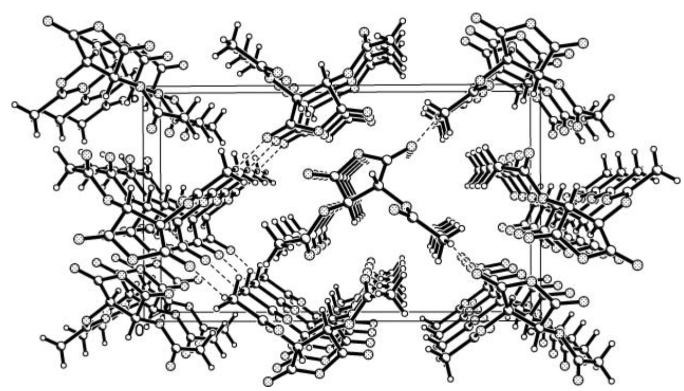


Figure 2
The crystal structure of (I), viewed along the *a* axis. Dashed lines indicate C—H···O interactions.

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