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## 3-Acetoxycyclohex-4-ene-1,2-dicarboxylic anhydride

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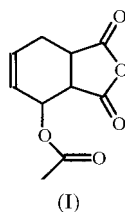
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The title compound, C<sub>10</sub>H<sub>10</sub>O<sub>5</sub>, was found to exist as the *endo-cis* isomer with a pair of enantiomers in the asymmetric unit. The cyclohexene ring is folded about the methylene-to-CH(acetoxy) vector to give a boat conformation.

## Comment

The title compound, (I), was synthesized as part of investigations into the specificity of reactions catalysed by antibody H11, a species which can achieve catalysis of Diels–Alder reactions as well as hydrolysing acetoxy groups (Khalaf *et al.*, 2000; Suckling *et al.*, 1993). The structure of (I) was elucidated to confirm its identity as the *endo-cis* isomer. The structure also confirms that (I) exists as an enantiomeric pair as would be expected as the starting butadiene was a mixture of *E* and *Z* isomers.



Two independent conformations were found in the asymmetric unit but these have essentially identical bond lengths and angles. The largest difference between these two mirror-image conformations lies in a slight difference in the magnitude of the torsion angles at the acetoxy groups [C9–O4–C6–C5 and C19–O9–C16–C15 are  $-164.41(14)$  and  $168.45(14)^\circ$ , respectively]. The cyclohexene rings adopt boat conformations with internal folds of  $134.55(8)$  and  $140.86(12)^\circ$ . This is a flatter conformation than those found in most similar compounds. A search of the Cambridge Structural Database (Allen & Kennard, 1993) found only one compound, [(C<sub>9</sub>H<sub>9</sub>O<sub>5</sub>)FeCp(CO)<sub>2</sub>], with a similar fold of  $142.0^\circ$  (Lee *et al.*, 1988). All other known structures have folds

of between  $110.0$  and  $129.5^\circ$ . The olefin C=C distances are a good fit with those of other related and non-strained compounds (Bolte & Bauch, 1999; Ben Fredj *et al.*, 1998), but shorter than those found in strained compounds (Baggio *et al.*, 1972).

## Experimental

1-Acetoxy-1,3-butadiene (2.187 g, 19.50 mmol) was added at room temperature to a suspension of maleic anhydride (1.750 g, 17.84 mmol) in benzene (40 ml) and stirred overnight. The solvent was removed under reduced pressure and the crude product purified by passing an ethyl acetate/*n*-hexane (1:3) solution through silica gel.  $R_f = 0.2$ ; yield 52%, 2.135 g; m.p. 326–329 K; IR (KBr):  $\nu_{\max}$  1855, 1784, 1736 cm<sup>-1</sup>.

## Crystal data

C <sub>10</sub> H <sub>10</sub> O <sub>5</sub>	$Z = 4$
$M_r = 210.18$	$D_x = 1.449 \text{ Mg m}^{-3}$
Triclinic, $P\bar{1}$	Mo $K\alpha$ radiation
$a = 10.4676(18) \text{ \AA}$	Cell parameters from 25 reflections
$b = 10.4774(15) \text{ \AA}$	$\theta = 16.44\text{--}17.80^\circ$
$c = 10.5727(18) \text{ \AA}$	$\mu = 0.118 \text{ mm}^{-1}$
$\alpha = 71.878(12)^\circ$	$T = 193(2) \text{ K}$
$\beta = 64.619(12)^\circ$	Plate, colourless
$\gamma = 69.533(12)^\circ$	$0.65 \times 0.40 \times 0.15 \text{ mm}$
$V = 963.5(3) \text{ \AA}^3$	

## Data collection

Rigaku AFC-7S diffractometer	$h = -12 \rightarrow 12$
$\omega/2\theta$ scans	$k = -12 \rightarrow 0$
4001 measured reflections	$l = -13 \rightarrow 12$
3784 independent reflections	3 standard reflections
2733 reflections with $I > 2\sigma(I)$	every 150 reflections
$R_{\text{int}} = 0.014$	intensity decay: 1.14%
$\theta_{\max} = 26.02^\circ$	

## Refinement

Refinement on $F^2$	$w = 1/[\sigma^2(F_o^2) + (0.0597P)^2 + 0.2317P]$
$R[F^2 > 2\sigma(F^2)] = 0.039$	where $P = (F_o^2 + 2F_c^2)/3$
$wR(F^2) = 0.118$	$(\Delta/\sigma)_{\max} = 0.002$
$S = 1.006$	$\Delta\rho_{\max} = 0.30 \text{ e \AA}^{-3}$
3784 reflections	$\Delta\rho_{\min} = -0.26 \text{ e \AA}^{-3}$
273 parameters	
H-atom parameters constrained	

All H atoms were observed in difference syntheses but were included in calculated positions and in riding modes. The orientations of the methyl groups were obtained by allowing refinement of rotational parameters about the C–Me bonds.

Data collection: *MSC/AFC Diffractometer Control Software* (Molecular Structure Corporation, 1985); cell refinement: *MSC/AFC Diffractometer Control Software*; data reduction: *TEXSAN* (Molecular Structure Corporation, 1993); program(s) used to solve structure: *SIR* (Burla *et al.*, 1989); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); software used to prepare material for publication: *SHELXL97*.

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