Data reduction, structure solution and refinement were carried out using *SHELXTL-Plus* (Sheldrick, 1991).

Lists of structure factors, anisotropic displacement parameters and H-atom coordinates have been deposited with the IUCr (Reference: AB1148). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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2-Phenylmaleic Anhydride

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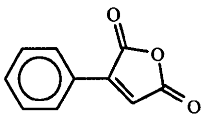
Abstract

The title compound, $C_{10}H_6O_3$, has been studied by both X-ray diffraction and molecular-mechanics calculations to determine the ring–ring dihedral angle. Calculated and observed values of 5.4 and 5.53 (6)° for this dihedral angle in the crystal were found to be in good agreement; these values were quite different from the calculated value of 40° for the gas-phase structure.

Comment

The structure of (1) is particularly important in relation to its use as a dienophile in Diels–Alder chemistry. In this context, possible differences between the solid-state and gas-phase structures are of considerable interest. The present X-ray crystallographic results show the solid-state structure of (1) to be almost planar [ring–ring dihedral angle = 5.53 (6)°]. In general, bond lengths and bond angles within (1) are similar to those found for 1,4-phenylenebis(phenylmaleic anhydride) (Fields *et al.*, 1990).



(1)

Force-field calculations were carried out to determine both the minimized crystal and gas-phase structures of (1). The *POLYGRAF* software (POLYGRAF, 1992) with the Dreiding 11 (Mayo, Olafson & Goddard, 1990) force field was employed for all molecular-mechanics calculations. The partial atomic charges in (1) for both the crystal and the gas-phase calculations were estimated by Del Re's method (Del Re, Pullman & Yonezawa, 1963) as implemented in the *POLYGRAF* software. Within *POLYGRAF*, the crystal field was provided by both van der Waals and electrostatic Ewald sums as previously described (Karasawa & Goddard, 1989).

The observed solid-state bond lengths and angles are compared with the calculated values in Table 2. Since the computed bond distances are based on empirically fit radii, there is some imperfection in the agreement between the Dreiding-based bond lengths and the experimental results, but the overall agreement with the experimental results is quite reasonable. The r.m.s. deviation between the solid-state observed and calculated values was found to be 0.05 Å for the bond lengths and 2.7° for the bond angles. The ring–ring dihedral angle calculated for the *POLYGRAF*-minimized solid-state structure was 5.4°, which is in excellent agreement with the corresponding observed value (see above). The ring–ring dihedral angle for the gas-phase molecule, as minimized by using *POLYGRAF*, was estimated to be 40°. The difference between the almost planar solid-state structure of (1) and the distinctly non-planar gas-phase structure must be attributable to the sum of the intermolecular forces acting on the molecules in the solid

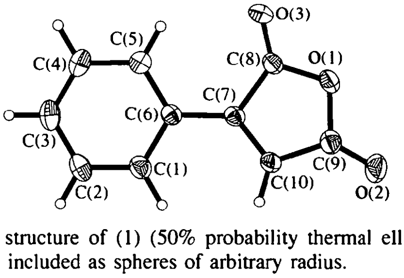


Fig. 1. The structure of (1) (50% probability thermal ellipsoids). H atoms are included as spheres of arbitrary radius.

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state. The almost planar structure is favored in the solid phase despite the fact that the distances between the *ortho* H atoms of the phenyl ring and the O and H atoms nearest to them on the five-membered ring [H(5)···O(3) = 2.36 (1), H(1)···H(10) = 2.25 (1) Å] are less than the distances that would be estimated from van der Waals radii.

Experimental

Crystals of (1) were provided by Dr Carlos Angüis of Colorado State University.

Crystal data

$C_{10}H_6O_3$
$M_r = 174.16$
Triclinic
$P\bar{1}$
$a = 6.547$ (2) Å
$b = 7.057$ (3) Å
$c = 9.687$ (4) Å
$\alpha = 107.48$ (3)°
$\beta = 102.07$ (3)°
$\gamma = 102.23$ (3)°
$V = 399.0$ (2) Å^3
$Z = 2$
$D_x = 1.45$ Mg m^{-3}

Mo $K\alpha$ radiation
$\lambda = 0.71073$ Å
Cell parameters from 25 reflections
$\theta = 10.18$–$19.96°$
$\mu = 0.101$ mm^{-1}
$T = 155$ K
Prism
0.54 × 0.45 × 0.34 mm
Colorless

Data collection

Siemens *R*3*m* diffractometer
$\theta/2\theta$ scans
Absorption correction: none
1547 measured reflections
1408 independent reflections
1320 observed reflections [$F > 2.5\sigma(F)$]
$R_{\text{int}} = 0.0116$

$\theta_{\max} = 25°$
$h = -8 \rightarrow 8$
$k = -9 \rightarrow 9$
$l = -12 \rightarrow 0$
3 standard reflections monitored every 97 reflections intensity decay: none

Refinement

Refinement on *F*
$R = 0.049$
$wR = 0.074$
$S = 1.207$
1320 reflections
148 parameters
H atoms: see text
$w = 1/[\sigma^2(F) + 0.00341F^2]$

$(\Delta/\sigma)_{\max} = 0.006$
$\Delta\rho_{\max} = 0.28$ e Å^{-3}
$\Delta\rho_{\min} = -0.36$ e Å^{-3}
Extinction correction: none
Atomic scattering factors from *International Tables for X-ray Crystallography* (1974, Vol. IV)

Table 1. *Fractional atomic coordinates and equivalent isotropic displacement parameters* (Å^2)

$$U_{\text{eq}} = (1/3)\Sigma_i\Sigma_j U_{ij}a_i^*a_j^*\mathbf{a}_i.\mathbf{a}_j.$$

	x	y	z	U_{eq}
C(1)	−0.2100 (2)	0.1843 (2)	−0.0410 (2)	0.028 (1)
C(2)	−0.3208 (3)	0.0537 (2)	−0.1893 (2)	0.034 (1)
C(3)	−0.2056 (3)	−0.0012 (2)	−0.2922 (2)	0.035 (1)
C(4)	0.0200 (3)	0.0757 (2)	−0.2469 (2)	0.035 (1)
C(5)	0.1319 (2)	0.2062 (2)	−0.0984 (2)	0.028 (1)
C(6)	0.0182 (2)	0.2615 (2)	0.0063 (1)	0.023 (1)
C(7)	0.1300 (2)	0.3998 (2)	0.1638 (2)	0.022 (1)
C(8)	0.3737 (2)	0.4799 (2)	0.2322 (2)	0.025 (1)
O(1)	0.4192 (2)	0.6050 (2)	0.3817 (1)	0.030 (1)
C(9)	0.2219 (2)	0.6106 (2)	0.4126 (2)	0.028 (1)
C(10)	0.0451 (2)	0.4801 (2)	0.2737 (1)	0.025 (1)
O(2)	0.2190 (2)	0.7101 (2)	0.5353 (1)	0.040 (1)
O(3)	0.5174 (2)	0.4506 (2)	0.1795 (1)	0.037 (1)

Table 2. *Selected geometric parameters* (Å, °)

	Observed	Calculated*
C(1)—C(2)	1.387 (2)	1.407
C(1)—C(6)	1.399 (2)	1.422
C(2)—C(3)	1.385 (2)	1.403
C(3)—C(4)	1.384 (2)	1.402
C(4)—C(5)	1.390 (2)	1.406
C(5)—C(6)	1.392 (2)	1.421
C(6)—C(7)	1.466 (2)	1.423
C(7)—C(8)	1.502 (2)	1.405
C(7)—C(10)	1.335 (2)	1.398
C(8)—O(1)	1.380 (2)	1.351
C(8)—O(3)	1.186 (2)	1.249
O(1)—C(9)	1.392 (2)	1.345
C(9)—C(10)	1.467 (2)	1.385
C(9)—O(2)	1.192 (2)	1.249
C(2)—C(1)—C(6)	120.6 (1)	121.9
C(2)—C(3)—C(4)	119.8 (1)	119.7
C(4)—C(5)—C(6)	120.3 (1)	121.9
C(1)—C(6)—C(7)	119.2 (1)	121.2
C(6)—C(7)—C(8)	124.6 (1)	129.9
C(8)—C(7)—C(10)	106.2 (1)	103.1
C(7)—C(8)—O(3)	131.1 (1)	127.9
C(8)—O(1)—C(9)	108.2 (1)	107.0
O(1)—C(9)—O(2)	120.6 (1)	125.0
C(7)—C(10)—C(9)	109.6 (1)	109.0
C(1)—C(2)—C(3)	120.1 (1)	120.0
C(3)—C(4)—C(5)	120.4 (1)	120.1
C(1)—C(6)—C(5)	118.8 (1)	116.5
C(5)—C(6)—C(7)	122.0 (1)	122.3
C(6)—C(7)—C(10)	129.3 (1)	127.1
C(7)—C(8)—O(1)	108.4 (1)	111.8
O(1)—C(8)—O(3)	120.5 (1)	120.4
O(1)—C(9)—C(10)	107.7 (1)	109.2
C(10)—C(9)—O(2)	131.6 (1)	125.9

* Crystal structure minimized using *POLYGRAPH* (POLYGRAPH, 1992).

H atoms were placed in idealized positions with C—H = 0.96 Å and $U(\text{H}) = 1.2U_{\text{iso}}(\text{C})$.

Structure solution: *SHELXTL* (Sheldrick, 1989).

Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the IUCr (Reference: PT1002). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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