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1,5-Dichloro-3,6-diphenyl-3-azabicyclo[3.2.0]hept-6-ene-2,4-dione

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In the title compound, $C_{18}H_{11}Cl_2NO_2$, the succinimide and cyclobutene rings are individually planar, with a dihedral angle of 66.02 (14)° between them. The crystal structure is stabilized by C—H···π interactions involving the phenyl rings.

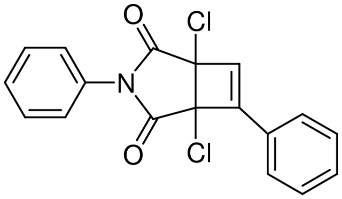
Key indicators

Single-crystal X-ray study
T = 293 K
Mean σ(C–C) = 0.003 Å
R factor = 0.055
wR factor = 0.132
Data-to-parameter ratio = 18.5

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

Comment

The photo-induced reactions of 1-phenyl-3,4-dichloromaleimide with diphenylethylene to give 3-azabicyclo[3.2.0]-heptanedione derivatives have been intensively investigated. Recently, we have reported the structure of one such derivative, 1,5-dichloro-3,6,6-triphenyl-3-azabicyclo[3.2.0]hepta-2,4-dione, (II) (Usman *et al.*, 2001). In a continuation of this study, we have undertaken the X-ray structure analysis of the title compound, (I), a product obtained by the photoreaction of 1-phenyl-3,4-dichloromaleimide with phenylacetylene.



(I)

In the succinimide–cyclobutene ring system of (I) (Fig. 1), the five-membered ring is planar in contrast to a half-chair conformation adopted in the succinimide–cyclobutane ring system of (II) (Usman *et al.*, 2001). The mean plane through the five-membered ring, with N1 deviating by a maximum of −0.032 (2) Å, forms a dihedral angle of 66.02 (14)° with the planar cyclobutene ring. The O1 and O2 atoms deviate from the plane of the attached ring by 0.047 (1) and 0.086 (1) Å, respectively. The phenyl ring substituent at C7 is almost coplanar with the cyclobutene [dihedral angle 12.86 (14)°], whereas that at N1 forms a dihedral angle of 74.83 (12)° with the five-membered ring.

The bond lengths and angles in (I) show normal values (Allen *et al.*, 1987), and agree with those of (II) (Usman *et al.*, 2001), except for the geometry of the cyclobutene ring (Table 1). The C7—C12 bond length of 1.521 (3) Å is slightly longer than that of the typical Csp^3—Csp^2 bond distance and this is considered to be due to the steric effect of the bulky substituent attached at C7. In the title molecule C9 and C12 are chiral centers.

In the crystal, the molecular packing is stabilized by C—H···π interactions (Table 2) involving the phenyl rings. In Table 2, $\pi(A)$ and $\pi(B)$ denote the center of gravity of the phenyl rings C1–C6 and C13–C18, respectively.

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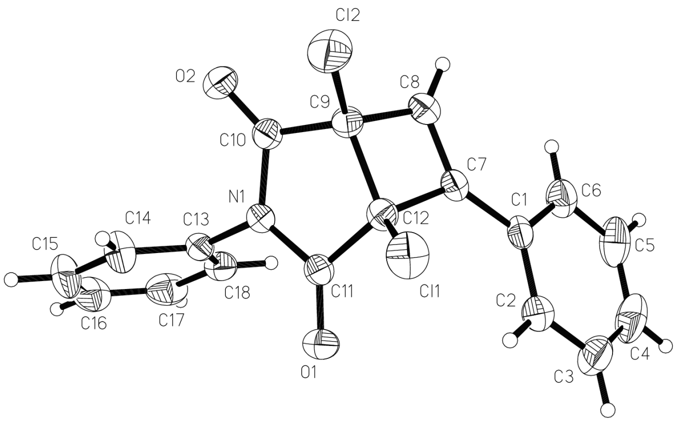

Figure 1
The structure of (I) showing 50% probability displacement ellipsoids and the atom-numbering scheme.

Experimental

The title compound, (I), was prepared by a photoinduced [2+2] cycloaddition reaction. A solution of 1-phenyl-3,4-dichloromaleimide (0.05 *M*) and phenylacetylene (0.2 *M*) in benzene (40 ml) was photolysed by visible light irradiation using a medium-pressure mercury lamp through a Pyrex filter and under constant nitrogen purging. Single crystals suitable for X-ray diffraction were obtained from slow evaporation of a petroleum ether–ethyl acetate solution.

Crystal data

$C_{18}H_{11}Cl_2NO_2$
$M_r = 344.18$
Monoclinic, $P2_1/c$
$a = 7.1468$ (2) Å
$b = 14.8464$ (5) Å
$c = 15.1091$ (5) Å
$\beta = 97.410$ (1)°
$V = 1589.75$ (9) Å^3
$Z = 4$
$D_x = 1.438$ Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 7131 reflections
$\theta = 2.7$–28.3°
$\mu = 0.42$ mm^{-1}
$T = 293$ (2) K
Slab, colorless
0.44 × 0.34 × 0.24 mm

Data collection

Siemens SMART CCD area-detector
ω scans
Absorption correction: empirical (*SADABS*; Sheldrick, 1996)
$T_{\min} = 0.838$, $T_{\max} = 0.907$
10 982 measured reflections
3864 independent reflections
2514 reflections with $I > 2\sigma(I)$
$R_{\rm int} = 0.084$
$\theta_{\max} = 28.3°$
$h = -9 \rightarrow 8$
$k = -19 \rightarrow 19$
$l = -20 \rightarrow 12$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.055$
$wR(F^2) = 0.132$
$S = 0.89$
3864 reflections
209 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0416P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\max} < 0.001$
$\Delta\rho_{\max} = 0.38$ e Å^{-3}
$\Delta\rho_{\min} = -0.43$ e Å^{-3}
Extinction correction: *SHELXTL*
Extinction coefficient: 0.025 (2)

Table 1
Selected geometric parameters (Å, °).

Cl1—C12	1.765 (2)	C1—C7	1.464 (3)
Cl2—C9	1.755 (2)	C7—C8	1.333 (3)
O1—C11	1.202 (2)	C7—C12	1.521 (3)
O2—C10	1.194 (2)	C8—C9	1.507 (3)
N1—C11	1.386 (2)	C9—C10	1.520 (3)
N1—C13	1.441 (3)	C9—C12	1.562 (3)
C12—C9—Cl2	118.95 (15)	C9—C12—Cl1	118.86 (14)

Table 2
Geometry of C—H···π interactions (Å, °).

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C4—H4*A*···$\pi(B^{\rm i})$	0.93	2.74	3.618 (3)	159
C8—H8*A*···$\pi(B^{\rm ii})$	0.93	2.71	3.528 (2)	147
C14—H14*A*···$\pi(A^{\rm iii})$	0.93	2.76	3.612 (3)	153

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

After checking their presence in a difference map, all H atoms were geometrically fixed and allowed to ride on the parent C atoms with isotropic displacement parameters.

Data collection: *SMART* (Siemens, 1996); cell refinement: *SAINT* (Siemens, 1996); data reduction: *SAINT*; program(s) used to solve structure: *SHELXTL* (Sheldrick, 1997); program(s) used to refine structure: *SHELXTL*; molecular graphics: *SHELXTL*; software used to prepare material for publication: *SHELXTL, PARST* (Nardelli, 1995) and *PLATON* (Spek, 1990).

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