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2a,5a-Dichloro-4-methyl-1-methylene-2-phenyl-2a,5a-dihydrocyclobuta[*c*]-pyrrole-3,5-dione

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Key indicators

Single-crystal X-ray study
T = 293 K
Mean σ(C–C) = 0.004 Å
R factor = 0.063
wR factor = 0.138
Data-to-parameter ratio = 16.1

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

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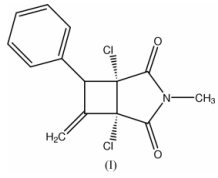
In the title compound, $C_{14}H_{11}Cl_2NO_2$, the pyrrolidine ring is planar within 0.050 (3) Å and the cyclobutane ring is planar within 0.059 (3) Å. The dihedral angle between these two planes is 67.4 (2)°. The packing of the molecules in the crystal is stabilized by C—H···π interactions.

Comment

In our recent investigation on photoinduced reactions of 3,4-dichloromaleimide with styrene derivatives (Zhao & Xu, 2003), we carried out a photoinduced reaction of 3,4-dichloromaleimide with phenylpropadiene. The title compound, (I), was obtained in this reaction as one of the two stereoisomers. It is formed by [2 + 2]-cycloaddition of the triplet-excited dichloromaleimide with phenyl propadiene at its central C=C bond. An X-ray crystallographic analysis was undertaken to elucidate its molecular conformation.



(I)

The bond lengths in (I) are within normal ranges (Allen *et al.*, 1987), except for a long C2—C7 bond, 1.577 (4) Å. This may be due to the steric effect of the bulky substituent attached at atom C7. The pyrrolidinedione moiety is essentially planar, mainly due to C=O and C—N conjugation. The cyclobutane ring (C2/C3/C6/C7) is planar within 0.059 (3) Å; the dihedral angle between the C3/C2/C7 and C3/C6/C7 planes is 12.2 (4)°. The cyclobutane mean plane makes a dihedral angle of 67.4 (2)° with the fused five-membered ring (N1/C1–C4). The phenyl ring (C8–C13) attached at C7 makes dihedral angles of 71.9 (2) and 9.9 (2)° with the cyclobutane

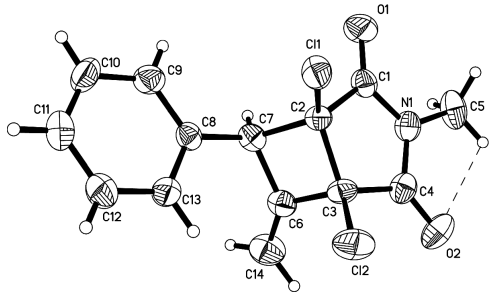


Figure 1
The structure of (I), showing 50% probability displacement ellipsoids and the atom-numbering scheme.

ring and the pyrrolidine ring, respectively. The Cl1—C2—C3—Cl2 torsion angle of 12.7 (3)° indicates that the two Cl atoms lie on the same side of the cyclobutane ring.

In the crystal structure of (I), there is one intramolecular C5—H5*C*···O2 interaction, forming a five-membered ring (Fig. 1). The molecular packing in the crystal structure is stabilized by C—H···π interactions involving the methyl group with the phenyl ring of a symmetry-related molecule (Table 2).

Table 1
Selected geometric parameters (Å, °).

Cl1—C2	1.751 (3)	C2—C3	1.555 (3)
Cl2—C3	1.756 (3)	C2—C7	1.577 (4)
O1—C1	1.202 (3)	C3—C4	1.520 (4)
O2—C4	1.195 (3)	C3—C6	1.525 (4)
N1—C1	1.373 (3)	C6—C14	1.309 (4)
N1—C4	1.387 (4)	C6—C7	1.525 (4)
N1—C5	1.462 (3)	C7—C8	1.513 (4)
C1—C2	1.530 (4)		
C1—N1—C4	115.0 (2)	C4—N1—C5	122.0 (2)
C1—N1—C5	123.0 (2)		

Experimental

The title compound, (I), was prepared by photolysis of a benzene solution of 3,4-dichloromaleimide in the presence of an excess amount of propadiene, followed by chromatographic separation of the reaction mixture on a silica-gel column with petroleum ether (b.p. 333–363 K)–ethyl acetate as eluants. Single crystals suitable for X-ray crystallographic analysis were grown by slow evaporation of solvent from petroleum ether (b.p. 333–363 K)–ethyl acetate (3:1 *v*/*v*).

Crystal data

$C_{14}H_{11}Cl_2NO_2$
M_r = 296.14
Monoclinic, *C*2/*c*
a = 14.8302 (11) Å
b = 7.3973 (5) Å
c = 25.3885 (18) Å
β = 106.167 (1)°
V = 2675.1 (3) Å^3
Z = 8
D_x = 1.471 Mg m^{-3}
Mo *K*α radiation
Cell parameters from 3429 reflections
θ = 2.9–28.1°
μ = 0.48 mm^{-1}
T = 293 (2) K
Plate, colourless
0.48 × 0.20 × 0.16 mm

Data collection

Siemens SMART CCD area-detector diffractometer
ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
T_{min} = 0.802, T_{max} = 0.927
8171 measured reflections
3302 independent reflections
2656 reflections with $I > 2\sigma(I)$
R_{int} = 0.028
θ_{max} = 28.3°
h = −18 → 19
k = −6 → 9
l = −33 → 33

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.063
$wR(F^2)$ = 0.138
S = 1.20
3302 reflections
205 parameters
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0479P)^2 + 3.9853P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 0.31 e Å^{-3}
$\Delta\rho_{min}$ = −0.27 e Å^{-3}

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

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C5—H5*C*···O2	0.96	2.49	2.872 (4)	103
C5—H5*B*···*Cg*P^{i}	0.96	2.75	3.579 (3)	145

Symmetry code: (i) $\frac{1}{2}+x, y-\frac{1}{2}, z$. *Cg*P denotes the centroid of the phenyl ring.

All H atoms were located in difference Fourier maps and were refined isotropically, except for those attached to the methyl C atom, which were positioned geometrically and treated as riding, with C—H distances of 0.96 Å and U_{iso}(H) = 1.5U_{eq}(C). The C—H distances of the refined H atoms lie in the range 0.85 (4)–0.97 (4) Å.

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, 2003).

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