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# 1,5-Dichloro-3,6,6-triphenyl-3-azabicyclo[3.2.0]-heptane-2,4-dione

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**Key indicators**

Single-crystal X-ray study
$T$ = 293 K
Mean $\sigma$(C–C) = 0.003 Å
$R$ factor = 0.045
$wR$ factor = 0.102
Data-to-parameter ratio = 18.0

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

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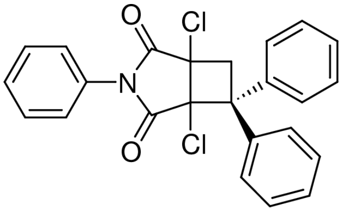
In the title compound, $C_{24}H_{17}Cl_2NO_2$, the succinimide ring adopts a half-chair conformation, and the attached phenyl ring is twisted by an angle of 55.0 (1)° with respect to the succinimide mean plane. The cyclobutane ring is non-planar and is substituted by two phenyl rings oriented at angles of 38.3 (1) and 75.7 (1)° to the cyclobutane mean plane. The dihedral angle between the succinimide and cyclobutane rings is 64.8 (1)°.

## Comment

The title compound, (I), was isolated in order to extend the scope of the photoinduced reactivity of the maleimide derivative and an X-ray crystal structure analysis was undertaken to establish its conformation.



(I)

In the succinimide-cyclobutane ring system (Fig. 1), the succinimide ring adopts a half-chair conformation [$Q_2$ = 0.141 (2) Å and $\varphi_2$ = 89.2 (6)°] twisted about the C3—C6 axis. The cyclobutane ring is non-planar with a dihedral angle between the C3/C4/C6 and C4/C5/C6 planes of 156.1 (2)° and a puckering parameter $Q_2$ = −0.231 (2) Å (Cremer & Pople, 1975). The dihedral angle between the least-square planes through the succinimide and cyclobutane rings is 64.8 (1)°. The bond lengths and angles in (I) have normal values and those within the succinimide are comparable with a related structure (Igonin *et al.*, 1993). In the cyclobutane ring, the C5—C6 bond length of 1.624 (2) Å is longer than typical C$sp^3$—C$sp^3$ bonds and is considered to be of spiro-atom character. The phenyl ring substituent attached to the succinimide ring moiety at N1 is twisted by an angle of 55.0 (1)° to the five-membered ring, corresponding to a bisectional orientation to the succinimide. Atoms O1 and O2 deviate slightly by −0.172 (1) and 0.183 (1) Å, respectively, from the mean plane of this ring. The phenyl rings attached to the cyclobutane ring at C5 are in nearly axial and equatorial positions and they subtend angles of 38.3 (1) and 75.7 (1)°, respectively, at the cyclobutane ring. Both Cl1 and Cl2 atoms

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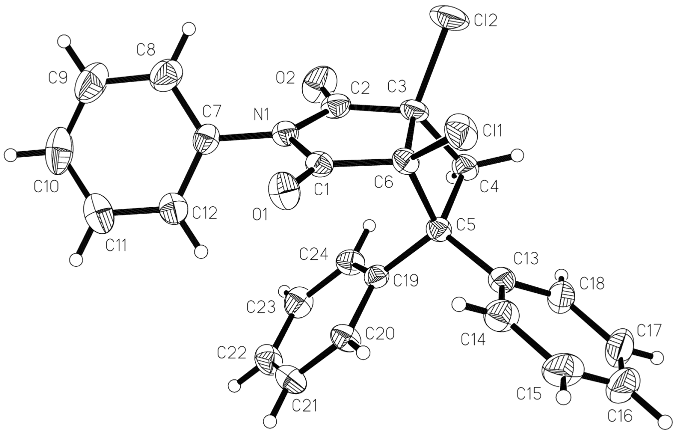

**Figure 1**

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

attached to the succinimide–cyclobutane ring system form an average Cl—C—C angle of 114.8 (1)°, while the Cl1—C6—C3—Cl2 torsion angle is −18.47 (17)°.

## Experimental

The title compound, (I), was prepared by a photoinduced reaction. A solution of 1-phenyl-2,3-dichloromaleimide (0.05 *M*) and 1,1-diphenylethylene (0.2 *M*) in benzene (40 ml) was photolysed by irradiation of light using a medium-pressure mercury lamp (500 W) through a Pyrex filter and under constant nitrogen purging. After irradiation the solvent was removed *in vacuo* and the residue was separated by column chromatography. (I) was obtained as the sole product and was recrystallized from a petroleum ether–ethyl acetate solution.

*Crystal data*

$C_{24}H_{17}Cl_2NO_2$
$M_r = 422.29$
Monoclinic, $P2_1/n$
$a = 11.1302$ (2) Å
$b = 7.3731$ (1) Å
$c = 24.3816$ (4) Å
$\beta = 96.950$ (1)°
$V = 1986.15$ (6) Å$^3$
$Z = 4$
$D_x = 1.412$ Mg m$^{-3}$
Mo $K\alpha$ radiation
Cell parameters from 8192 reflections
$\theta = 1.7$–28.3°
$\mu = 0.35$ mm$^{-1}$
$T = 293$ (2) K
Slab, colourless
0.36 × 0.28 × 0.16 mm

*Data collection*

Siemens SMART CCD diffractometer
$\omega$ scans
Absorption correction: empirical (*SADABS*; Sheldrick, 1996)
$T_{\min} = 0.885$, $T_{\max} = 0.946$
13 108 measured reflections
4726 independent reflections
3204 reflections with $I > 2\sigma(I)$
$R_{\rm int} = 0.078$
$\theta_{\max} = 28.3°$
$h = -14 \rightarrow 14$
$k = -9 \rightarrow 9$
$l = -16 \rightarrow 32$

*Refinement*

Refinement on $F^2$
$R[F^2 > 2\sigma(F^2)] = 0.045$
$wR(F^2) = 0.102$
$S = 0.83$
4726 reflections
263 parameters
H-atom parameters constrained
$w = 1/\sigma^2(F_o^2)$
$(\Delta/\sigma)_{\max} < 0.001$
$\Delta\rho_{\max} = 0.29$ e Å$^{-3}$
$\Delta\rho_{\min} = -0.25$ e Å$^{-3}$
Extinction correction: *SHELXTL*
Extinction coefficient: 0.0133 (11)

**Table 1**

Selected geometric parameters (Å, °).

| | | | |
|---|---|---|---|
| Cl1—C6 | 1.7587 (16) | C2—C3 | 1.529 (2) |
| Cl2—C3 | 1.7655 (16) | C3—C4 | 1.529 (2) |
| O1—C1 | 1.2033 (19) | C3—C6 | 1.545 (2) |
| O2—C2 | 1.202 (2) | C4—C5 | 1.563 (2) |
| N1—C1 | 1.3952 (19) | C5—C6 | 1.624 (2) |
| N1—C2 | 1.400 (2) | C5—C13 | 1.519 (2) |
| N1—C7 | 1.438 (2) | C5—C19 | 1.537 (2) |
| C1—C6 | 1.507 (2) | | |
| C1—N1—C2 | 113.47 (13) | C3—C4—C5 | 90.00 (11) |
| C1—N1—C7 | 122.26 (13) | C4—C5—C6 | 86.85 (11) |
| C2—N1—C7 | 124.00 (13) | C3—C6—C5 | 87.19 (11) |
| C2—C3—C6 | 102.92 (12) | C1—C6—Cl1 | 110.66 (11) |
| C4—C3—C6 | 90.93 (11) | C2—C3—Cl2 | 107.50 (11) |
| C3—C4—C5—C6 | −16.63 (12) | C4—C5—C13—C18 | −37.2 (2) |
| Cl2—C3—C6—Cl1 | −18.47 (17) | C6—C5—C19—C20 | −85.18 (17) |
| C1—N1—C7—C8 | 125.96 (18) | | |

The H atoms were located in difference maps and were subsequently geometrically fixed and allowed to ride on the parent C atoms, with aromatic C—H = 0.93 Å and methylene C—H = 0.97 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, 1990).

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