3a,8c-Dichloro-8b-phenyl-3a,3b,8b,8c-tetrahydro-2-methyl-1H-[1]benzothieno[2′,3′:3,4]cyclobuta-[1,2-c]pyrrole-1,3(2H)-dione

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In the title compound, $C_{19}H_{13}Cl_2NO_2S$, the cyclobutane ring is slightly folded. The crystal structure is stabilized by intra- and intermolecular C—H···O interactions.
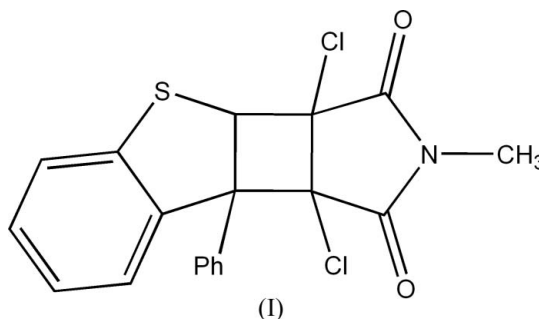
Comment

We have investigated the photo-induced reaction of 3,4-dichloromaleimide and 3-phenylbenzothiophene and obtained the title compound, (I), as one of the products. As part of this study, we have undertaken the X-ray crystallographic analysis of (I), in order to elucidate the conformation and configuration of this cycloadduct product.

The bond lengths and angles in (I) are in good agreement with expected values, except for the C3—C4 [1.586 (5) Å] and the C17—C18 [1.576 (5) Å] bond lengths, which are slightly longer than the normal Csp^3—Csp^3 distance [1.554 (21) Å] reported for cyclobutanes by Allen *et al.* (1987). These bond lengths are comparable with those observed in the related compound 4a,4c,9b,9c-tetrahydro-4b,4c,9b,9c-tetrachloro-cyclobuta[1,2-a:3,4-a′]diindene-5,10-dione [1.5885 (19) Å; Zhang *et al.*, 2003].

The cyclobutane ring slightly folded, the dihedral angle between the C3/C4/C17 plane and the C3/C17/C18 plane being 13.3 (4)°. The benzothiophene moiety is essentially planar, with C17 deviating from the mean plane by 0.241 (5) Å. The two Cl atoms lie on the same side of the maleimide plane. The molecular packing is stabilized by intra- and intermolecular C—H···O interactions, as detailed in Table 2.

Experimental

Compound (I) was prepared by the photo-induced reaction of a benzene solution of 3,4-dichloromaleimide with an excess amount of 3-phenylbenzothiophene, irradiated by light of wavelength longer than 300 nm for 27 h, and was isolated by column chromatography of the reaction mixture after evaporation of the solvent on silica gel. Single crystals of (I) were obtained by slow evaporation of a petroleum ether–ethyl acetate (2:1 *v/v*) solution (yield 84%).

Crystal data

$C_{19}H_{13}Cl_2NO_2S$
$M_r = 390.26$
Triclinic, $P\bar{1}$
$a = 8.3390\ (17)$ Å
$b = 8.6810\ (17)$ Å
$c = 13.268\ (3)$ Å
$\alpha = 71.08\ (3)°$
$\beta = 77.21\ (3)°$
$\gamma = 85.31\ (3)°$
$V = 886.0\ (4)$ Å^3

$Z = 2$
$D_x = 1.463$ Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 25 reflections
$\theta = 1.7$–$25.0°$
$\mu = 0.50$ mm^{-1}
$T = 288\ (2)$ K
Block, colorless
$0.30 \times 0.28 \times 0.28$ mm

Data collection

Enraf–Nonius CAD-4 diffractometer
$\omega/2\theta$ scans
Absorption correction: ψ scan (*XCAD4*; Harms & Wocadlo, 1995)
$T_{min} = 0.846$, $T_{max} = 0.870$
3361 measured reflections
3122 independent reflections

2475 reflections with $I > 2\sigma(I)$
$R_{int} = 0.021$
$\theta_{max} = 25.0°$
$h = 0 \rightarrow 9$
$k = -10 \rightarrow 10$
$l = -15 \rightarrow 15$
3 standard reflections every 200 reflections intensity decay: none

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.050$
$wR(F^2) = 0.160$
$S = 1.00$
3122 reflections
226 parameters
H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.0695P)^2 + 1.998P]$
where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.36$ e Å^{-3}
$\Delta\rho_{min} = -0.43$ e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

Cl1—C18	1.738 (4)	C3—C4	1.586 (5)
Cl2—C3	1.753 (4)	C4—C17	1.562 (5)
C3—C18	1.543 (5)	C17—C18	1.576 (5)
C16—S—C17	91.91 (17)	C18—C3—C4	90.6 (3)
C19—N—C2	114.7 (3)	C17—C4—C3	88.2 (3)
Cl2—C3—C4—C11	27.0 (4)	S—C17—C18—Cl1	1.5 (4)

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

D—H$\cdots A$	D—H	H$\cdots A$	$D\cdots A$	D—H$\cdots A$
C1—H1A$\cdots$O2	0.96	2.50	2.874 (7)	103
C17—H17$\cdots$O2^i	0.98	2.48	3.410 (6)	159

Symmetry code: (i) $-x, -y + 1, -z + 1$.

H atoms were included in the riding-model approximation, with C—H distances of 0.93, 0.96 and 0.98 Å for aromatic, methyl and
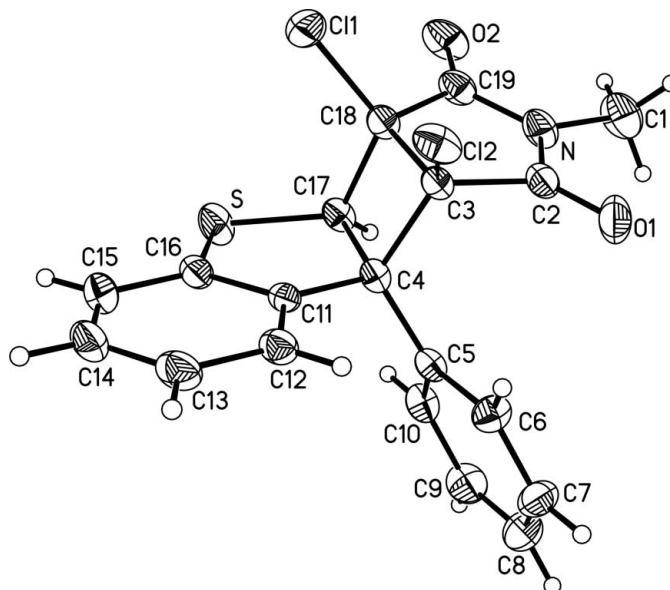

Figure 1
The structure of (I), showing 30% probability displacement ellipsoids and the atom-numbering scheme.

methine H atoms, respectively, and with $U_{iso}(H) = 1.2U_{eq}$(aromatic and methine C) or $1.5U_{eq}$(methyl C).

Data collection: *CAD-4 Software* (Enraf–Nonius, 1989); cell refinement: *CAD-4 Software*; data reduction: *XCAD4* (Harms & Wocadlo, 1995); 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* and *PLATON* (Spek, 2003).

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