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Key indicators

Single-crystal X-ray study
T = 213 K
Mean $\sigma(\text{C}-\text{C}) = 0.005 \text{ \AA}$
R factor = 0.062
wR factor = 0.181
Data-to-parameter ratio = 12.8

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

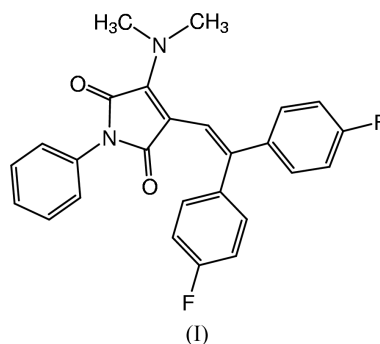
4-[2,2-Bis(4-fluorophenyl)ethenyl]-3-(*N,N*-dimethylamino)-*N*-phenylmaleimide

In the title compound, $\text{C}_{26}\text{H}_{20}\text{F}_2\text{N}_2\text{O}_2$, the dimethylamino group is nearly coplanar with the maleimide, and the *N*-phenyl ring forms a dihedral angle of $51.0(2)^\circ$ with the maleimide. The crystal structure is stabilized by weak intermolecular $\text{C}-\text{H}\cdots\text{O}$ and $\text{C}-\text{H}\cdots\text{F}$ interactions.

Received 1 August 2002
Accepted 13 August 2002
Online 31 August 2002

Comment

The photo-induced reactions of 1-phenyl-3,4-dichloromaleimide with diphenylethylene give the cyclobutane product, 1,5-dichloro-3,6,6-triphenyl-3-azabicyclo[3.2.0]hepta-2,4-dione, (II), whose crystal structure has been reported previously (Usman *et al.*, 2001). This compound undergoes a thermal cyclobutane ring-opening reaction in *N,N*-dimethylacetamide to give the title compound, (I) (Zhao & Xu, 2002). In order to establish the conformation of (I), we undertook an X-ray crystal-structure analysis, the results of which are presented here.



The bond lengths and angles in (I) are within normal ranges (Allen *et al.*, 1987), except for the values within the maleimide moiety (N1/C7–C10). The C8–C9 bond length lengthens, whereas the N1–C8 bond length shortens, compared with typical $\text{Csp}^2-\text{Csp}^2$ and $\text{Csp}^2-\text{Nsp}^2$ distances, respectively, owing to interactions between the bulky substituents attached at the maleimide moiety. This also affects the N2–C9–C10 bond angle, which is about 11.5° larger than N2–C9–C8. The dimethylamino group (N2/C15/C26) attached at C9 is nearly coplanar with the maleimide. This substituent is twisted outwards about the N2–C9 bond by $13.7(2)^\circ$ with respect to the maleimide. Atoms O1 and O2 are displaced by $0.090(2)$ and $0.075(2) \text{ \AA}$, respectively, on opposite sides of the maleimide plane.

The C1–C6 phenyl ring attached at atom N1 forms a dihedral angle of $51.0(2)^\circ$ with the maleimide; this is much smaller than that in (II) [$74.8(1)^\circ$; Usman *et al.*, 2001].

The C11/C12/C13/C19 plane containing the ethylene double bond is planar and makes dihedral angles of $50.1(2)$, $52.3(2)$

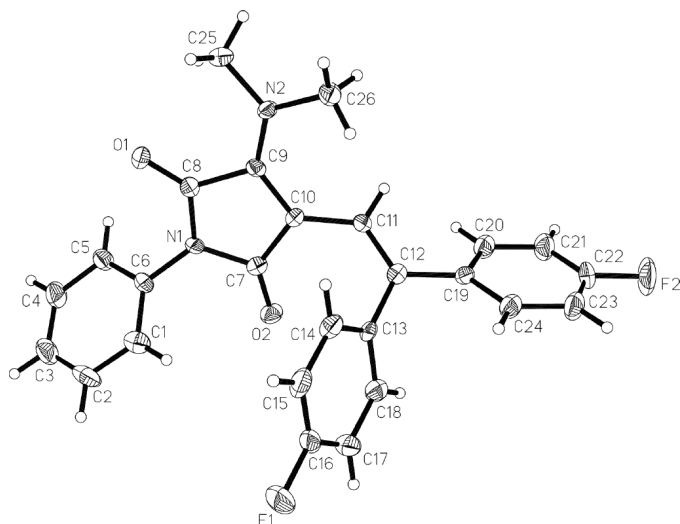


Figure 1
The structure of (I), showing 50% probability displacement ellipsoids and the atom-numbering scheme.

and $35.0(2)^\circ$ with the maleimide, C13–C18 and C19–C24 phenyl rings, respectively (Fig. 1). The dihedral angle between the two phenyl rings is $74.5(2)^\circ$, which is comparable with that of the 2,2-diphenylethenyl moiety in another structure [$75.5(1)^\circ$; Usman *et al.*, 2002]. Atoms F1 and F2 also lie in the planes of their attached phenyl rings, with deviations of 0.015(3) and 0.023(3) Å, respectively.

In the packing, two adjacent molecules form dimers (Fig. 2) through C–H···O intermolecular contacts (Table 1). The molecular dimers are further interconnected by C–H···F interactions (Table 1) to form a three-dimensional network (Fig. 3).

Experimental

The title compound was prepared by thermolysis of 1,5-dichloro-3,6,6-triphenyl-3-azabicyclo[3.2.0]hepta-2,4-dione in *N,N*-dimethylacetamide (Zhao & Xu, 2002). Single crystals, suitable for X-ray diffraction, were obtained by slow evaporation of a petroleum ether–acetone solution.

Crystal data

$C_{26}H_{20}F_2N_2O_2$
 $M_r = 430.44$
Monoclinic, $P2_1/n$
 $a = 14.4125(7)$ Å
 $b = 7.4776(4)$ Å
 $c = 20.3189(7)$ Å
 $\beta = 99.787(2)^\circ$
 $V = 2157.9(2)$ Å³
 $Z = 4$

$D_x = 1.325$ Mg m⁻³
Mo $K\alpha$ radiation
Cell parameters from 2961 reflections
 $\theta = 2.9$ – 28.3°
 $\mu = 0.10$ mm⁻¹
 $T = 213(2)$ K
Needle, yellow
 $0.34 \times 0.20 \times 0.14$ mm

Data collection

Siemens SMART CCD area-detector diffractometer
 ω scans
Absorption correction: multi-scan (SADABS; Sheldrick, 1996)
 $T_{\min} = 0.968$, $T_{\max} = 0.987$
9770 measured reflections

3743 independent reflections
1914 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.107$
 $\theta_{\text{max}} = 25.0^\circ$
 $h = -16 \rightarrow 17$
 $k = -8 \rightarrow 8$
 $l = -24 \rightarrow 17$

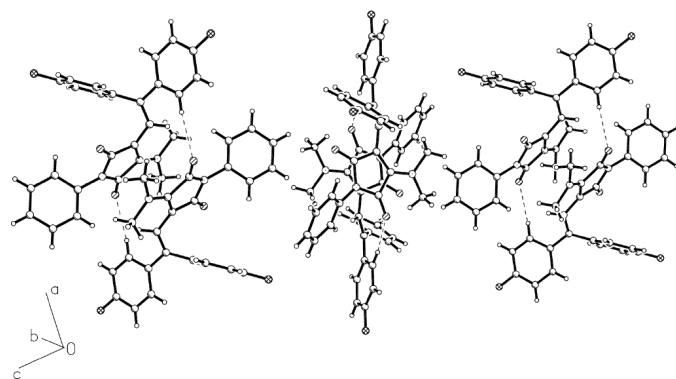


Figure 2
Packing diagram of the title compound, showing the molecular dimers linked by C–H···O interactions (dashed lines).

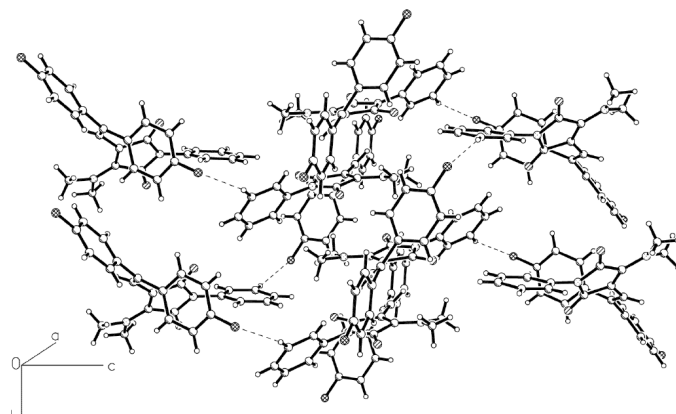


Figure 3
Packing diagram of the title compound, showing the three-dimensional network. The dashed lines denote C–H···F interactions.

Refinement

Refinement on F^2
 $R[F^2 > 2\sigma(F^2)] = 0.062$
 $wR(F^2) = 0.181$
 $S = 0.89$
3743 reflections
292 parameters
H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.0818P)^2]$
where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{\text{max}} < 0.001$
 $\Delta\rho_{\text{max}} = 0.37$ e Å⁻³
 $\Delta\rho_{\text{min}} = -0.41$ e Å⁻³
Extinction correction: SHELXTL
Extinction coefficient: 0.042(3)

Table 1

Intermolecular contacts geometry (Å, °).

$D-H \cdots A$	$D-H$	$H \cdots A$	$D \cdots A$	$D-H \cdots A$
C4–H4B···F1 ⁱ	0.93	2.48	3.266(5)	142
C20–H20A···O1 ⁱⁱ	0.93	2.55	3.470(5)	172

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

The H atoms were placed geometrically and treated as riding on the parent C atoms, with C–H distances in the range 0.93–0.96 Å and $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{C})$. As there was a large fraction of weak data at higher angles, the 2θ maximum was limited to 50° .

Data collection: SMART (Siemens, 1996); cell refinement: SAINT (Siemens, 1996); data reduction: SAINT and SADABS; 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).

The authors thank the Malaysian Government and Universiti Sains Malaysia for research grant R&D No. 305/PFIZIK/610961. AU thanks Universiti Sains Malaysia for a Visiting Postdoctoral Fellowship.

References

- Allen, F. H., Kennard, O., Watson, D. G., Brammer, L., Orpen, A. G. & Taylor, R. (1987). *J. Chem. Soc. Perkin Trans. 2*, pp. S1–19.
- Nardelli, M. (1995). *J. Appl. Cryst.* **28**, 659.
- Sheldrick, G. M. (1996). *SADABS*. University of Göttingen, Germany.
- Sheldrick, G. M. (1997). *SHELXTL*. Version 5.1. Bruker AXS Inc., Madison, Wisconsin, USA.
- Siemens (1996). *SMART* and *SAINT*. Siemens Analytical X-ray Instruments Inc., Madison, Wisconsin, USA.
- Spek, A. L. (1990). *Acta Cryst.* **A46**, C-34.
- Usman, A., Razak, I. A., Fun, H.-K., Chantrapromma, S., Zhao, B.-G. & Xu, J.-H. (2001). *Acta Cryst.* **E57**, o990–o991.
- Usman, A., Razak, I. A., Fun, H.-K., Chantrapromma, S., Zhao, B.-G. & Xu, J.-H. (2002). *Acta Cryst.* **E58**, o756–o757.
- Zhao, B.-G. & Xu, J.-H. (2002). In preparation.