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1,4-Dimethyl-2-phenyl-3*H*,10a*H*-4-aza-cyclobuta[*c*]phenylene-3,5-dione

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Key indicators

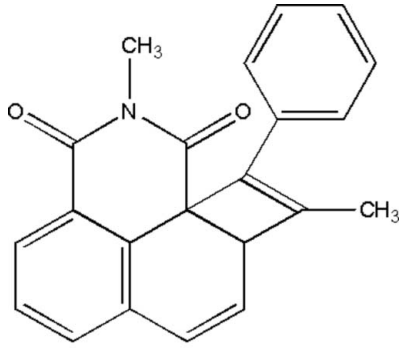
Single-crystal X-ray study
T = 298 K
Mean σ(C–C) = 0.004 Å
R factor = 0.059
wR factor = 0.193
Data-to-parameter ratio = 14.4

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

The title compound, $C_{22}H_{17}NO_2$, was obtained by the photoreaction of *N*-methyl-1,8-naphthalenedicarboximide with 1-phenylpropyne. The planar cyclobutene ring makes a dihedral angle of 71.7 (2)° with the benzene ring of the 1,2-dihydronaphthalene group. There are intramolecular C—H···O hydrogen bonds, and intermolecular C—H···O interactions help to stabilize the crystal structure.

Comment

Photoinduced cycloaddition of aromatic imides to alkenes has long been an active research area in organic photochemistry (Mazzocchi, 1981; Maruyama & Kubo, 1985). As part of our studies on the photoinduced electron-transfer reactions of aromatic imides with various organic electron donors (Xue *et al.*, 2000), we have investigated the photoinduced reactions of naphthalimides with alkynes. We report here the X-ray crystal structure of the title compound, (I), which is one of the products of the photoreaction of *N*-methyl-1,8-naphthalenedicarboximide with 1-phenylpropyne.



(I)

The bond lengths and angles in (I) display normal values, except for the geometry of the cyclobutene ring (Fig. 1 and Table 1). In (I), the naphthalimide group is not planar, as a result of the sp^3 character of atoms C9 and C13. The C9—C13 bond is elongated by the steric effect of the bulky substituents attached at atoms C9 and C13 (Liu *et al.*, 2003). Such an elongation has also been found in another cyclobutene derivative (Usman *et al.*, 2001). The phenyl ring at C16 is twisted by 43.3 (2)° with respect to the attached cyclobutene ring.

In the crystal structure of (I) (Fig.2), the molecules are arranged along the *b* axis *via* intermolecular C—H···O interactions, in which the two neighbouring 1,2-dihydronaphthalene groups overlap face-to-face. Such arrangements take advantage of the π–π stacking interactions between the two neighbouring 1,2-dihydronaphthalene groups, thereby

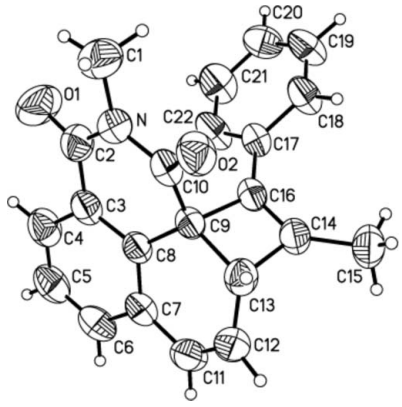


Figure 1
The structure of (I), showing 30% probability displacement ellipsoids and the atom-numbering scheme.

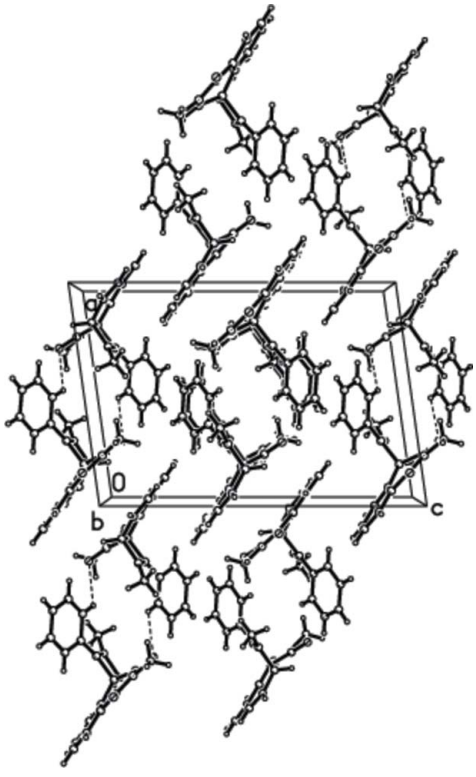


Figure 2
The molecular packing of (I), viewed along the *b* axis. Dashed lines indicate hydrogen bonds.

stabilizing the crystal packing. The perpendicular distance between the ring at (x, y, z) and that at $(-x, y - \frac{1}{2}, \frac{3}{2} - z)$ is 3.424 Å; the distance between their centroids is 5.814 (2) Å.

Experimental

The title compound was prepared by irradiation for 36 h (with light of wavelength longer than 330 nm) of a benzene solution of *N*-methyl-1,8-naphthalenedicarboximide (0.6 mmol, 0.13 g) and 1-phenylpropyne (6 mmol, 0.7 g). The residue was purified by column chromatography on silica gel; the product was crystallized from dichloromethane–petroleum ether solution (1:3 *v*/*v*, yield 52%) to afford (I) as yellow crystals, which were suitable for X-ray analysis (m.p. 437 K).

Crystal data

$C_{22}H_{17}NO_2$
M_r = 327.37
Monoclinic, $P2_1/c$
a = 11.327 (2) Å
b = 9.2270 (18) Å
c = 16.397 (3) Å
β = 97.95 (3)°
V = 1697.2 (6) Å^3
Z = 4
D_x = 1.281 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 25 reflections
θ = 10–13°
μ = 0.08 mm^{-1}
T = 298 (2) K
Block, yellow
0.30 × 0.30 × 0.20 mm

Data collection

Enraf–Nonius CAD-4 diffractometer
$\omega/2\theta$ scans
Absorption correction: ψ scan (*XCAD4*; Harms & Wocadlo, 1995)
T_{min} = 0.963, T_{max} = 0.984
3445 measured reflections
3278 independent reflections
1792 reflections with $I > 2\sigma(I)$
R_{int} = 0.048
θ_{max} = 26.0°
$h = 0 \rightarrow 13$
$k = 0 \rightarrow 10$
$l = -19 \rightarrow 19$
3 standard reflections
every 200 reflections
intensity decay: none

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.059
$wR(F^2)$ = 0.193
S = 1.03
3278 reflections
227 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.1017P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 0.21 e Å^{-3}
$\Delta\rho_{min}$ = −0.24 e Å^{-3}
Extinction correction: *SHELXL97*
Extinction coefficient: 0.022 (3)

Table 1
Selected geometric parameters (Å, °).

N—C2	1.382 (4)	C13—C14	1.525 (4)
N—C10	1.389 (4)	C14—C16	1.335 (4)
N—C1	1.463 (4)	C14—C15	1.484 (4)
C9—C13	1.572 (3)		
C8—C9—C10	113.6 (2)	C10—C9—C13	114.9 (2)
C8—C9—C16	117.3 (2)	C12—C13—C14	112.6 (2)
C10—C9—C16	105.53 (19)	C16—C14—C15	135.3 (3)

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

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C1—H1*A*···O1	0.96	2.25	2.690 (4)	107
C12—H12*A*···O1^{i}	0.93	2.41	3.215 (3)	145
C18—H18*A*···O2^{ii}	0.93	2.58	3.247 (3)	129

Symmetry codes: (i) $x, y - 1, z$; (ii) $-x + 1, -y + 1, -z + 2$.

The H atoms were positioned geometrically and were treated as riding, with C—H = 0.93–0.98 Å, and with U_{iso}(H) = 1.2U_{eq}(parent atom) or U_{iso}(H) = 1.5U_{eq}(C_{methyl}).

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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References

Enraf–Nonius. (1989). *CAD-4 Software*. Version 5. Enraf–Nonius, Delft, The Netherlands.
Harms, K. & Wocadlo, S. (1995). *XCAD4*. University of Marburg, Germany.
Liu, Q.-J., Li, Y.-Z., Shi, D.-Q. & Xu, J.-H. (2003). *Acta Cryst.* C**59**, o36–o37.
Maruyama, K. & Kubo, Y. (1985). *J. Org. Chem.* **50**, 1426–1435.
Mazzocchi, P. H. (1981). *Organic Photochemistry*, Vol. 5, edited by A. Padwa, p. 421. New York: Marcel Dekker.
Sheldrick, G. M. (1997). *SHELXTL*. Version 5.1. Bruker AXS Inc., Madison, Wisconsin, USA.
Spek, A. L. (2003). *J. Appl. Cryst.* **36**, 7–13.
Usman, A., Razak, I. A., Fun, H.-K., Chantrapromma, S., Zhao, B.-G. & Xu, J.-H. (2001). *Acta Cryst.* E**57**, o990–o991.
Xue, J., Zhu, L., Fun, H.-K. & Xu, J. H. (2000). *Tetrahedron Lett.* **41**, 8553–8557.