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9-Bromo-4-methyl-2-phenylbenzo[*de*]cyclobut[*i*]isoquinoline-3,5(2*H*)-dione

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Key indicators

Single-crystal X-ray study
T = 293 K
Mean σ(C–C) = 0.008 Å
R factor = 0.048
wR factor = 0.163
Data-to-parameter ratio = 12.3

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

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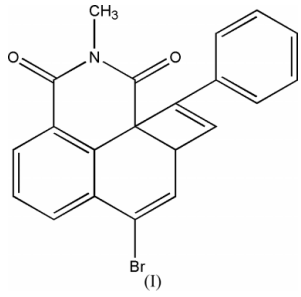
The title compound, $C_{21}H_{14}BrNO_2$, is the main product of the photoreaction of 4-bromo-*N*-methyl-1,8-naphthalenedicarboximide with phenylacetylene. The cyclobutene ring is almost perpendicular to the mean plane of the dihydronaphthalimide moiety, and the phenyl ring substituent is twisted by 6.1 (9)° with respect to the cyclobutene ring.

Comment

The photochemical reactions of imides with alkenes have been studied extensively (Kanaoka, 1978; Mazzocchi, 1981). However, the photochemical reactions of imides with alkynes have not been reported so far. As part of our studies on the photochemistry of aromatic imides, we have investigated the photoreactions of naphthalimides with alkynes. We report here the crystal structure of the title compound, (I), which is the main product of the photoreaction of 4-bromo-*N*-methyl-1,8-naphthalenedicarboximide with phenylacetylene.



(I)

In (I), the naphthalimide moiety has lost planarity as a result of the sp^3 character of atoms C3 and C12. The cyclobutene ring (C1–C3/C12) is almost perpendicular to the mean plane of the dihydronaphthalimide moiety. The phenyl ring at C1 is twisted by 6.1 (9)° with respect to the cyclobutene ring. The bond lengths and angles show normal values, except for the geometry of the cyclobutene ring (Table 1). The C1—C12 bond length [1.552 (7) Å] is much longer than the typical Csp^3—Csp^2 bond distance but is slightly shorter than that in 4-methyl-2-phenylbenzo[*de*]cyclobut[*i*]isoquinoline-3,5(2*H*)-dione [1.562 (3) Å; Liu *et al.*, 2002]. Such elongation is considered to be caused by the steric effect of the bulky substituents attached at atoms C1 and C12.

As illustrated in Fig. 2, the molecules are packed into ladders in which the dihydronaphthalimide moieties are arranged as the rungs, and the phenyl rings together with the cyclobutene rings constitute the uprights of the ladders.

Experimental

Compound (I) was prepared by irradiation (with light of wavelength longer than 300 nm) of a benzene solution, purged with nitrogen, of

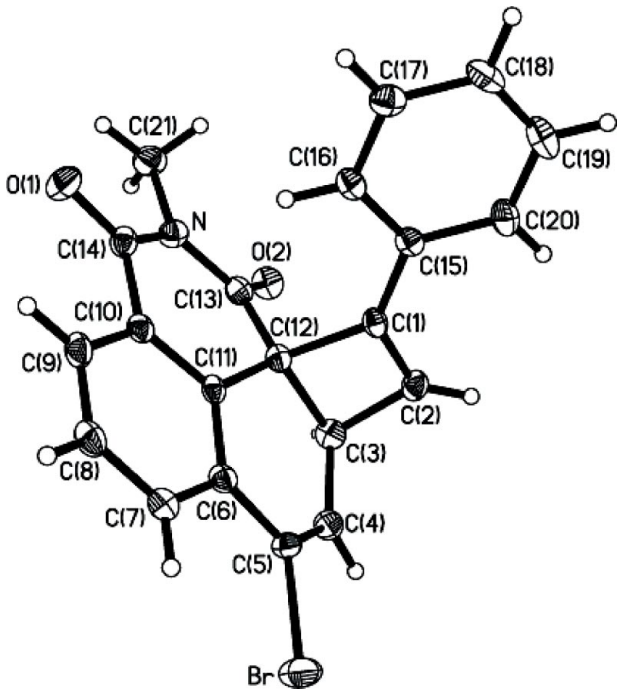


Figure 1
The molecular structure of (I), showing 50% probability displacement ellipsoids.

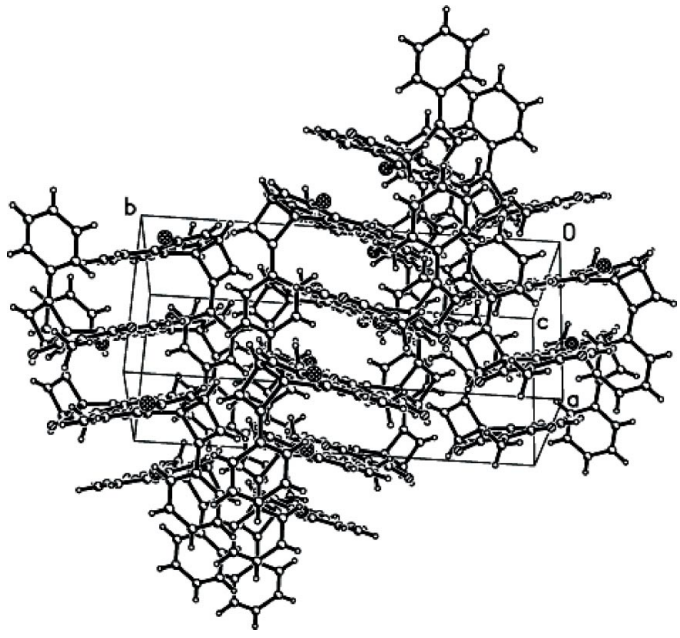


Figure 2
The molecular packing diagram of (I).

4-bromo-*N*-methyl-1,8-naphthalenedicarboximide and phenylacetylene, and isolated as the main product of the photoreaction by flash column chromatography on silica gel; m.p. 473–474 K. Single crystals of (I) suitable for X-ray diffraction were obtained by recrystallization from an ethyl acetate–petroleum ether solution.

Crystal data

$C_{21}H_{14}BrNO_2$
M_r = 392.24
Monoclinic, $P2_1/c$
a = 8.589 (2) Å
b = 19.126 (4) Å
c = 10.176 (2) Å
β = 104.88 (3)°
V = 1615.6 (6) Å^3
Z = 4
D_x = 1.613 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 25 reflections
θ = 11–13°
μ = 2.56 mm^{-1}
T = 293 (2) K
Prism, colourless
0.34 × 0.27 × 0.20 mm

Data collection

Enraf–Nonius CAD-4 diffractometer
ω scans
Absorption correction: ψ scan (North *et al.*, 1968)
T_{min} = 0.441, T_{max} = 0.600
3036 measured reflections
2840 independent reflections
1527 reflections with $I > 2\sigma(I)$
R_{int} = 0.042
θ_{max} = 25.0°
$h = 0 \rightarrow 10$
$k = 0 \rightarrow 22$
$l = -12 \rightarrow 11$
3 standard reflections
every 200 reflections
intensity decay: 4.8%

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.048
$wR(F^2)$ = 0.163
S = 0.903
2840 reflections
230 parameters
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.1P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.36 e Å^{-3}
$\Delta\rho_{min}$ = −0.30 e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

C1—C2	1.333 (7)	C3—C12	1.579 (7)
C1—C15	1.465 (7)	C4—C5	1.325 (8)
C1—C12	1.552 (7)	C11—C12	1.493 (7)
C2—C3	1.510 (8)	C12—C13	1.524 (7)
C3—C4	1.476 (8)		
C2—C1—C15	133.9 (5)	C2—C3—C12	85.6 (4)
C2—C1—C12	93.1 (4)	C11—C12—C1	118.2 (4)
C15—C1—C12	132.9 (5)	C13—C12—C1	107.3 (4)
C1—C2—C3	95.9 (5)	C1—C12—C3	85.0 (4)
C4—C3—C2	110.1 (5)		

Atom H2 bonded to the cyclobutene ring was refined freely, the C—H distance being 0.88 (6) Å. All other H atoms were positioned geometrically (C—H = 0.93–0.98 Å) and treated using a riding model.

Data collection: *XSCANS* (Siemens, 1994); cell refinement: *XSCANS*; data reduction: *SHELXTL* (Sheldrick, 1997); program(s) used to solve structure: *SHELXTL*; program(s) used to refine structure: *SHELXTL*; molecular graphics: *SHELXTL*; software used to prepare material for publication: *SHELXTL*.

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References

Kanaoka, Y. (1978). *Acc. Chem. Res.* **1**, 407–413.
Liu, Q.-J., Shi, D.-Q. & Xu, J.-H. (2002). *J. Chem. Cryst.* **33**, 263–267.
Mazzocchi, P. H. (1981). *Organic Photochemistry*, Vol.5, edited by A. Padwa, p. 421. New York: Marcel Dekker.
North, A. C. T., Phillips, D. C. & Mathews, F. S. (1968). *Acta Cryst.* A**24**, 351–359.
Sheldrick, G. M. (1997). *SHELXTL.* Version 5.1. Bruker AXS Inc., Madison, Wiscosin, USA.
Siemens (1994). *XSCANS.* Version 2.1. Siemens Analytical X-ray Instruments Inc., Madison, Wiscosin, USA.