

2-(4-Methoxyphenyl)-4-methylbenzo[de]-
cyclobut[*i*]isoquinoline-3,5(2*H*)-dioneQingjian Liu,^{a*†} Rongjun Qu^b
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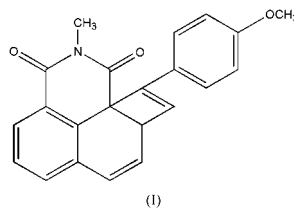
Key indicators

Single-crystal X-ray study
T = 291 K
Mean $\sigma(\text{C}-\text{C}) = 0.003 \text{ \AA}$
R factor = 0.038
wR factor = 0.101
Data-to-parameter ratio = 12.5For details of how these key indicators were automatically derived from the article, see <http://journals.iucr.org/e>.

The title compound, $\text{C}_{22}\text{H}_{17}\text{NO}_3$, is one of the photoreaction products of *N*-methyl-1,8-naphthalenedicarboximide with 4-methoxyphenylacetylene. The planar cyclobutene ring makes a dihedral angle of $70.36(8)^\circ$ with the benzene ring of the tetralin moiety. The substituted phenyl ring is twisted by $21.15(14)^\circ$ with respect to the attached cyclobutene ring.

Comment

The photochemical reaction of imides with alkenes has been extensively studied (Kanaoka, 1978; Mazzocchi, 1981). As part of our studies on the photo-induced electron-transfer reactions of aromatic imides with various organic electron donors (Xue *et al.*, 2000), we have investigated the photo-induced 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 4-methoxyphenylacetylene.



The bond lengths and angles in (I) display normal values, except for the geometry of the cyclobutene ring (Fig. 1 and Table 1). The C7—C10 bond is elongated by the steric effect of the bulky substituents attached at atoms C7 and C10. This C7—C10 bond distance of $1.563(2) \text{ \AA}$ is slightly longer than that in 4-methyl-2-phenylbenzo[de]cyclobut[*i*]isoquinoline-3,5(2*H*)-dione (Liu, Shi & Xu, 2003), but is very similar to that in 9,10-epoxy-4-methyl-2-phenylbenzo[de]cyclobut[*i*]isoquinoline-3,5(2*H*)-dione (Liu, Li *et al.*, 2003). Such an elongation has also been found in another cyclobutene derivative (Usman *et al.*, 2001).

The naphthalimide moiety has lost the coplanarity due to the sp^3 character of atoms C9 and C10. The substituted phenyl ring at C7 is twisted by $21.15(14)^\circ$ with respect to the attached cyclobutene ring.

In the crystal structure of (I) (Fig. 2), the molecules are arranged along the *b* axis into parallel infinite stripes, in which the molecules are orientated head-to-head, the two neighbouring tetrahydronaphthalene moieties lie back-to-back and the other two substituted phenyl rings overlap face-to-face. Such arrangements take advantage of the π - π -stacking interactions involving these aromatic rings, thereby stabilizing the crystal packing.

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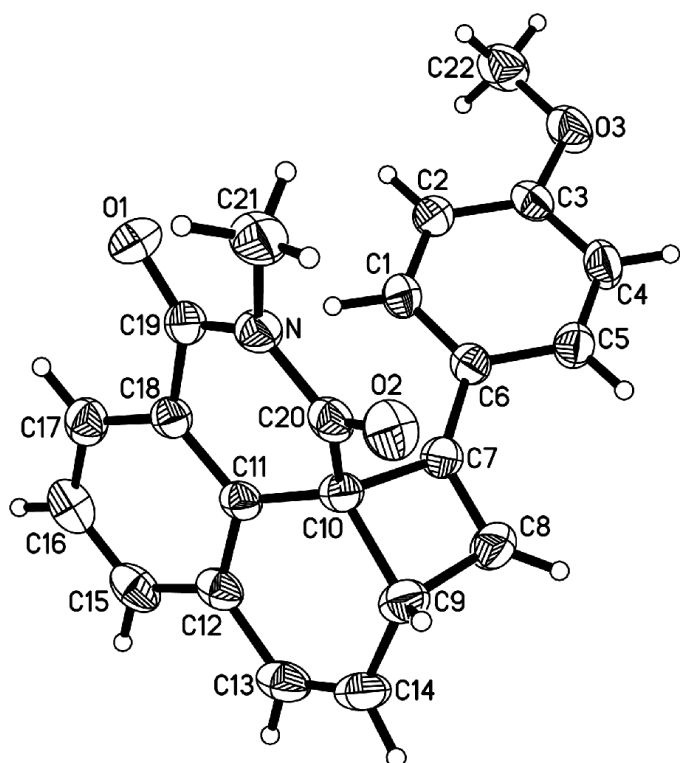


Figure 1
The molecular structure of (I), showing 50% probability displacement ellipsoids and the atom-numbering scheme.

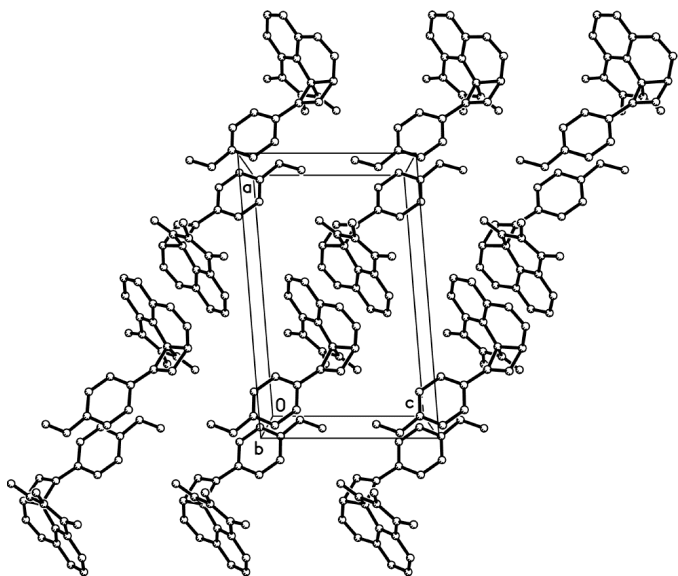


Figure 2
The crystal packing diagram of (I), viewed down the *b* axis.

Experimental

The title compound was prepared *via* irradiation (with light of wavelength longer than 300 nm) of a benzene solution of *N*-methyl-1,8-naphthalenedicarboximide with 4-methoxyphenylacetylene, and isolated as one of the products of the photoreaction by flash column chromatography on silica gel, m.p. 441–442 K. Single crystals of (I) suitable for X-ray diffraction were grown from an ethanol–ethyl acetate solution.

Crystal data

$C_{22}H_{17}NO_3$
 $M_r = 343.37$
Monoclinic, $P2_1/c$
 $a = 13.893$ (2) Å
 $b = 14.248$ (2) Å
 $c = 8.662$ (1) Å
 $\beta = 94.63$ (1)°
 $V = 1709.0$ (4) Å³
 $Z = 4$

$D_x = 1.334$ Mg m⁻³
Mo $K\alpha$ radiation
Cell parameters from 28 reflections
 $\theta = 2.8$ – 14.4 °
 $\mu = 0.09$ mm⁻¹
 $T = 291$ (2) K
Prism, colourless
 $0.30 \times 0.25 \times 0.20$ mm

Data collection

Siemens *P4* diffractometer
 ω scans
Absorption correction: none
3489 measured reflections
3016 independent reflections
1944 reflections with $I > 2\sigma(I)$
 $R_{int} = 0.014$

$\theta_{max} = 25.0$ °
 $h = -16 \rightarrow 16$
 $k = -16 \rightarrow 0$
 $l = 0 \rightarrow 10$
3 standard reflections every 97 reflections
intensity decay: 2.5%

Refinement

Refinement on F^2
 $R[F^2 > 2\sigma(F^2)] = 0.038$
 $wR(F^2) = 0.101$
 $S = 0.94$
3016 reflections
242 parameters
H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.0517P)^2]$
where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{max} < 0.001$
 $\Delta\rho_{max} = 0.17$ e Å⁻³
 $\Delta\rho_{min} = -0.16$ e Å⁻³
Extinction correction: *SHELXTL*
Extinction coefficient: 0.0136 (14)

Table 1

Selected geometric parameters (Å, °).

C7—C8	1.323 (3)	C10—C11	1.499 (3)
C7—C10	1.563 (2)	C11—C12	1.403 (2)
C8—C9	1.505 (3)	C12—C13	1.460 (3)
C9—C14	1.486 (3)	C13—C14	1.318 (3)
C9—C10	1.576 (2)		
C8—C7—C10	92.74 (16)	C11—C10—C7	117.76 (15)
C6—C7—C10	134.11 (16)	C20—C10—C7	107.73 (14)
C7—C8—C9	96.60 (17)	C11—C10—C9	115.83 (15)
C8—C9—C10	85.70 (14)	C7—C10—C9	84.77 (13)
C22—O3—C3—C2	-0.9 (3)	C6—C7—C10—C20	-64.7 (3)
C1—C6—C7—C10	-24.6 (3)	C8—C9—C10—C20	-104.21 (17)
C10—C7—C8—C9	3.48 (16)	C8—C9—C10—C7	2.92 (14)
C8—C7—C10—C11	-119.82 (18)	C8—C9—C14—C13	-94.3 (2)

All H atoms were positioned geometrically with C—H distances in the range 0.93–0.97 Å.

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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