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**Crystal Structure
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# 4,5,9,10-Tetrahydro-4-methyl-2-phenyl-9,10-epoxy-3*H*,10a*H*-cyclobuta[*a*]benzo[2,3,4-*de*]isoquinoline-3,5-dione

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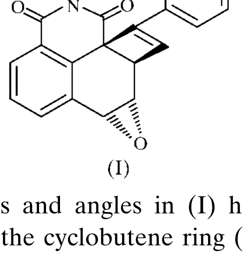
In the title compound, $C_{21}H_{15}NO_3$, which is one of the photoreaction products of *N*-methyl-1,8-naphthalenedicarboximide with phenylacetylene, the cyclobutene and epoxy rings are *trans* to each other across the cyclohexene ring of the tetralin moiety. The dihedral angle between the mean planes of the cyclobutene and cyclohexene rings is 112.80 (2)°, while the latter makes a dihedral angle of 103.70 (9)° with the epoxy ring. The crystal structure is stabilized by C—H···O intermolecular interactions.

## Comment

The photochemistry of imides with alkenes has been extensively studied (Kanaoka, 1978; Mazzocchi, 1981). However, the photochemical reactions of imides with alkynes have not been reported to date. As part of our studies of 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 crystal structure of the title compound, (I), which is one of the products of the photoreaction of *N*-methyl-1,8-naphthalenedicarboximide with phenylacetylene.



(I)

The bond lengths and angles in (I) have normal values, except for those of the cyclobutene ring (Table 1). The C7—C10 bond length of 1.569 (2) Å is much longer than the typical C$sp^3$—C$sp^2$ bond distance and is also slightly longer than that of 4,5,9,10-tetrahydro-4-methyl-2-phenyl-3*H*,10a*H*-cyclobuta[*a*]benz[2,3,4-*de*]isoquinoline-3,5-dione (Liu *et al.*, 2003). Such an elongation has also been observed in another cyclobutene derivative (Usman *et al.*, 2001) and is considered to be caused by the steric effects of the bulky substituents attached at positions C7 and C10.

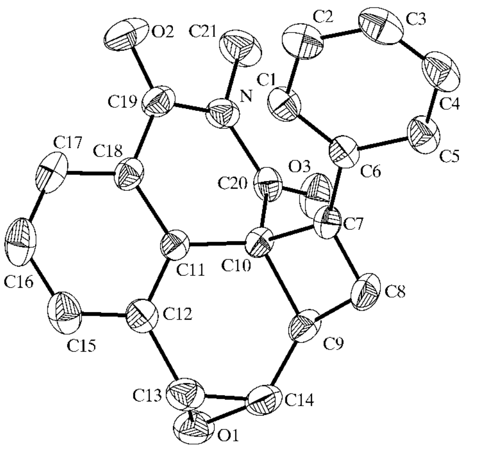


**Figure 1**
The molecular structure of (I), showing 50% probability displacement ellipsoids and the atom-numbering scheme. H atoms have been omitted for clarity.

The naphthalimide moiety has lost its planarity due to the $sp^3$ character of atoms C9, C10, C13 and C14. The C7–C10 cyclobutene ring makes a dihedral angle of 112.80 (2)° with the C9–C14 cyclohexadiene ring, while the latter makes dihedral angles of 6.47 (7) and 18.49 (8)°, respectively, with the C11/C12/C15–C18 benzene ring and the C10/C11/C18/C19/N/C20 ring. The dihedral angle between rings C10/C11/C18/C19/N/C20 and C11/C12/C15–C18 is 15.09 (9)°. The phenyl substituent attached to the C7–C10 cyclobutene ring at C7 is twisted by 150.50 (2)° with respect to this ring. The C13/C14/O1 epoxy ring makes a dihedral angle of 103.70 (9)° with the C9–C14 cyclohexadiene ring.

In the structure of (I), there are intermolecular C4—H4···O1 and C14—H14···O3 close contacts (Table 2).

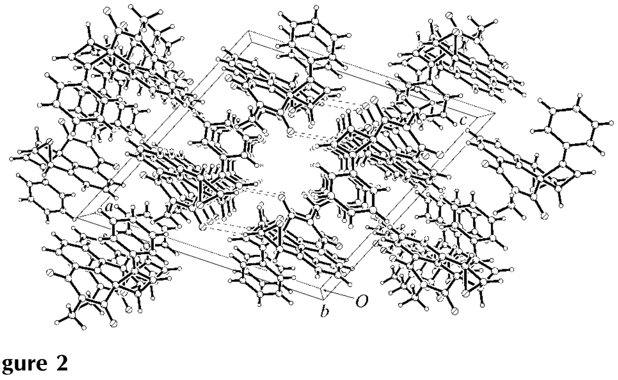


**Figure 2**
The molecular packing diagram for the crystal of (I).

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## Experimental

The title compound was prepared by irradiation (with light of wavelength longer than 300 nm) of a nitrogen-purged benzene solution of *N*-methyl-1,8-naphthalenedicarboximide with phenylacetylene, and isolated as one of the products of the photoreaction using flash column chromatography on silica gel. Single crystals of (I) (m.p. 447.0–448.5 K) suitable for X-ray diffraction were obtained by recrystallization from an ethanol–ethyl acetate (5:1) solution.

### *Crystal data*

$C_{21}H_{15}NO_3$
$M_r = 329.34$
Monoclinic, $P2_1/c$
$a = 14.801$ (3) Å
$b = 8.306$ (2) Å
$c = 14.441$ (2) Å
$\beta = 114.15$ (1)°
$V = 1620.0$ (6) Å$^3$
$Z = 4$
$D_x = 1.350$ Mg m$^{-3}$
Mo $K\alpha$ radiation
Cell parameters from 25 reflections
$\theta = 3.0$–12.6°
$\mu = 0.09$ mm$^{-1}$
$T = 288$ (2) K
Prism, colourless
0.34 × 0.30 × 0.25 mm

**Table 1**
Selected geometric parameters (Å, °).

| | | | |
|---|---|---|---|
| O1—C14 | 1.434 (2) | C8—C9 | 1.505 (3) |
| O1—C13 | 1.464 (2) | C9—C14 | 1.485 (3) |
| C7—C8 | 1.324 (2) | C9—C10 | 1.572 (2) |
| C7—C10 | 1.569 (2) | C13—C14 | 1.460 (3) |
| | | | |
| C14—O1—C13 | 60.52 (13) | C7—C10—C9 | 84.55 (12) |
| C8—C7—C10 | 92.76 (14) | C14—C13—O1 | 58.73 (13) |
| C7—C8—C9 | 96.42 (15) | O1—C14—C13 | 60.74 (13) |
| C8—C9—C10 | 86.09 (13) | | |
| | | | |
| C1—C6—C7—C8 | −150.5 (2) | C6—C7—C10—C20 | 68.1 (2) |
| C10—C7—C8—C9 | −3.40 (16) | C11—C18—C19—N | 15.0 (2) |
| C8—C7—C10—C11 | 121.73 (17) | C11—C10—C20—N | 36.8 (2) |

**Table 2**
Hydrogen-bonding geometry (Å, °).

| $D$—H···$A$ | $D$—H | H···$A$ | $D$···$A$ | $D$—H···$A$ |
|---|---|---|---|---|
| C4—H4···O1$^{i}$ | 0.93 | 2.66 | 3.478 (2) | 147 |
| C14—H14···O3$^{ii}$ | 0.98 | 2.42 | 3.329 (2) | 154 |

Symmetry codes: (i) $x, \frac{1}{2} - y, z - \frac{1}{2}$; (ii) $1 - x, \frac{1}{2} + y, \frac{3}{2} - z$.

### *Data collection*

Siemens *P*4 diffractometer
$\omega$ scans
3306 measured reflections
2852 independent reflections
1870 reflections with $I > 2\sigma(I)$
$R_{\rm int} = 0.011$
$\theta_{\rm max} = 25°$
$h = -17 \rightarrow 16$
$k = -9 \rightarrow 0$
$l = 0 \rightarrow 17$
3 standard reflections
every 97 reflections
intensity decay: 4.8%

### *Refinement*

Refinement on $F^2$
$R[F^2 > 2\sigma(F^2)] = 0.041$
$wR(F^2) = 0.110$
$S = 0.97$
2852 reflections
228 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0603P)^2]$
where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\rm max} = 0.001$
$\Delta\rho_{\rm max} = 0.19$ e Å$^{-3}$
$\Delta\rho_{\rm min} = -0.12$ e Å$^{-3}$
Extinction correction: *SHELXTL*
Extinction coefficient: 0.0108 (15)

The positions of all H atoms were fixed geometrically, with C—H distances in the range 0.93–0.98 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*.

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Supplementary data for this paper are available from the IUCr electronic archives (Reference: OB1086). Services for accessing these data are described at the back of the journal.

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