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2-Methyl-3,3,10-triphenyl-3,4-dihydronaphth[2,1-*c*]-azepine-1,5-dione

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In the title compound, $C_{33}H_{25}NO_2$, the azepine ring adopts a half-chair conformation. The molecules are packed into a three-dimensional network by three intermolecular C—H···O hydrogen bonds.

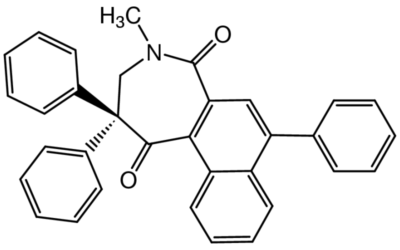
Key indicators

Single-crystal X-ray study
T = 183 K
Mean *σ*(C–C) = 0.002 Å
R factor = 0.060
wR factor = 0.151
Data-to-parameter ratio = 17.6

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

Comment

The photo-induced reactions of *N*-methyl-1,2-naphthalimide with alkenes have been intensively investigated (Kubo *et al.*, 1986). In general, these reactions lead to the expansion of the pyrrolidine ring to give the corresponding two naphthazepine regio-isomers as products, which are readily separated by column chromatography. The regiochemistry of the two isomers was deduced from NMR data and their reactivity with sodium borohydride. In our recent research on the photochemical synthesis of naphthalimides, we have isolated and structurally analysed the title compound, (I). This compound was one of the isomers which resulted from the photo-induced reaction of *N*-methyl-7-phenyl-1,2-naphthalimide with 1,1-diphenylethene. The X-ray crystal structure analysis of (I) was undertaken in order to establish the regiochemistry in the additional ring, and to elucidate its steric conformation and the relative configuration of the rings.



(I)

The bond lengths and angles in (I) are in good agreement with expected values. The C4—C5 bond length is slightly elongated compared with a normal Csp^2—Csp^3 bond (Allen *et al.*, 1987) due to the steric effect of the two bulky phenyl substituents attached at atom C5. The azepine moiety (atoms C1–C6/N1) is not planar, tending towards a boat conformation with a total puckering amplitude Q_T = 0.992 (2) Å (Cremer & Pople, 1975). Ketone atoms O1 and O2 and the C5/C6/N1 plane are displaced on opposite sides of the plane through atoms C1–C4, with both C1 and C4 in an sp^2 hybridization state.

The dihedral angle between phenyl rings C22–C27 and C28–C33 is 80.8 (1)°. The C16–C21 phenyl ring, attached at C9, makes a dihedral angle of 80.1 (1)° with the naphthalene system.

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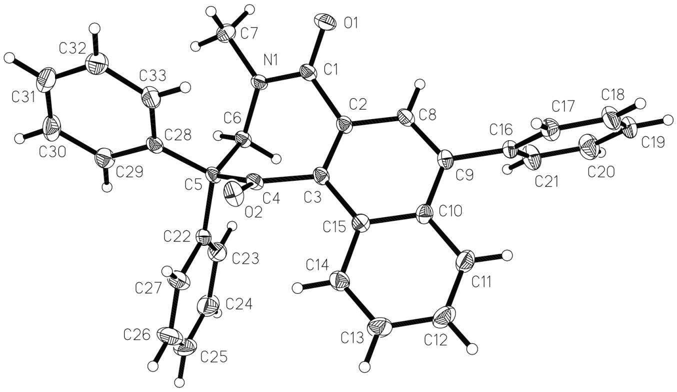

Figure 1
The structure of the title compound, showing 50% probability displacement ellipsoids, and the atom-numbering scheme.

In the packing of the title compound, the molecules are interconnected by an intermolecular C6—H6*B*···O1^{i} hydrogen bond (Table 2) into chains parallel to the *b* axis (Fig. 2). These molecular chains are interconnected by an intermolecular C13—H13···O2^{ii} hydrogen bond into molecular layers (Fig. 3) which are linked by another C21—H21···O2^{iii} hydrogen bond into a three-dimensional molecular network.

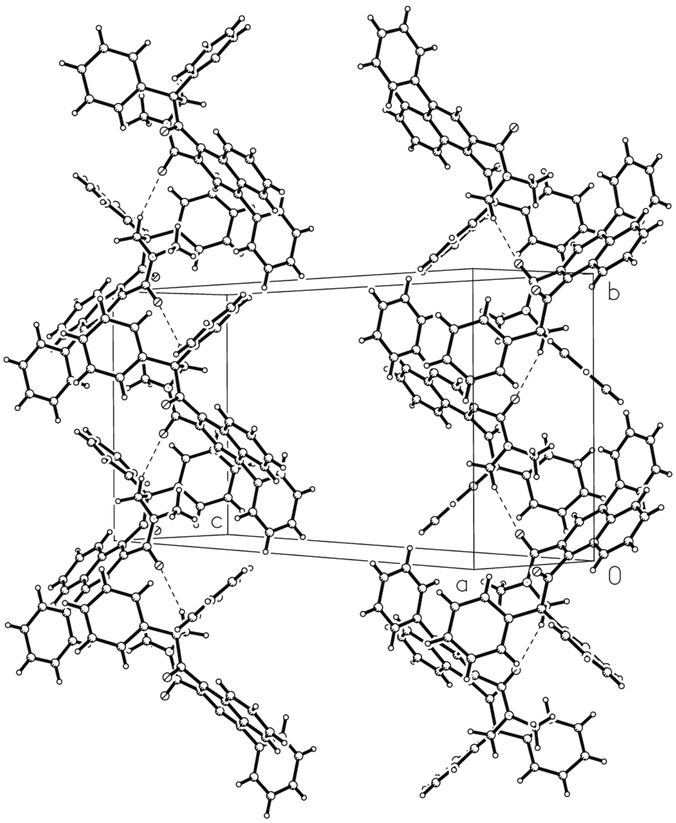

Figure 2
The packing of the title compound, showing the formation of molecular chains.

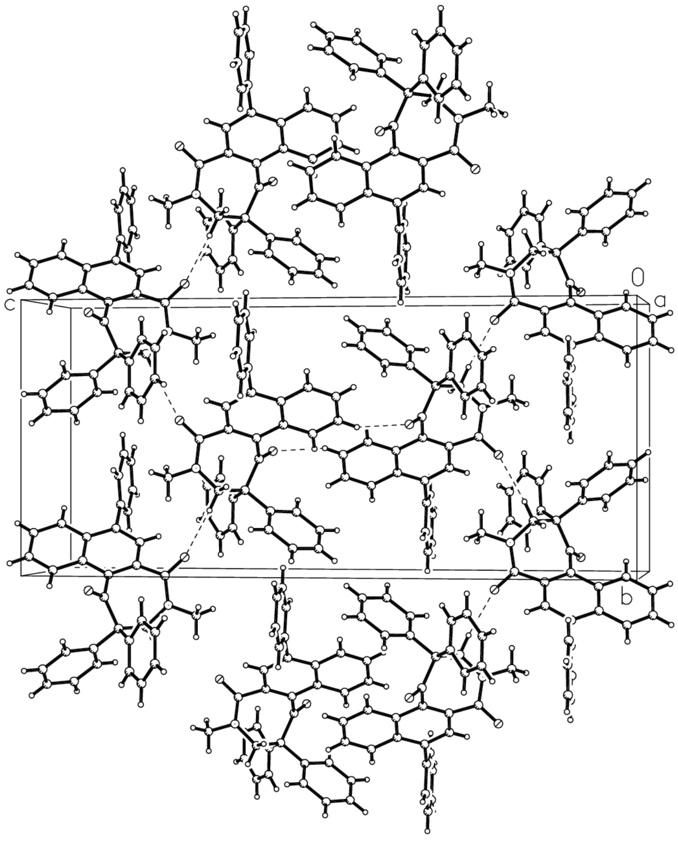

Figure 3
The packing of the title compound, viewed down the *a* axis, showing the formation of molecular layers.

Experimental

The title compound was prepared by a photo-induced reaction of *N*-methyl-5-phenyl-1,2-naphthalimide in the presence of an excess of 1,1-diphenylethene in benzene solution. Single crystals suitable for X-ray diffraction study were obtained by the slow evaporation of an acetone–petroleum ether solution.

Crystal data

$C_{33}H_{25}NO_2$
M_r = 467.54
Monoclinic, $P2_1/n$
a = 7.2788 (1) Å
b = 12.2856 (1) Å
c = 27.1499 (3) Å
β = 94.313 (1)°
V = 2421.0 (1) Å^3
Z = 4

D_x = 1.283 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 8192 reflections
θ = 2.8–28.3°
μ = 0.08 mm^{-1}
T = 183 (2) K
Block, colorless
0.50 × 0.30 × 0.30 mm

Data collection

Siemens SMART CCD area-detector diffractometer
ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
$T_{\min}$ = 0.961, $T_{\max}$ = 0.977
14 058 measured reflections

5767 independent reflections
4083 reflections with $I > 2\sigma(I)$
$R_{\rm int}$ = 0.075
$\theta_{\max}$ = 28.3°
$h = -9 \rightarrow 9$
$k = -16 \rightarrow 15$
$l = -29 \rightarrow 36$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.060$
$wR(F^2) = 0.151$
$S = 0.93$
5767 reflections
327 parameters
H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.0574P)^2]$
where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\max} < 0.001$
$\Delta\rho_{\max} = 0.47$ e Å^{-3}
$\Delta\rho_{\min} = -0.44$ e Å^{-3}
Extinction correction: *SHELXL*97
Extinction coefficient: 0.044 (3)

Table 1
Selected interatomic distances (Å).

N1—C1	1.354 (2)	C3—C4	1.510 (2)
N1—C6	1.466 (2)	C4—C5	1.549 (2)
C1—C2	1.505 (2)	C5—C6	1.544 (2)
C2—C3	1.387 (2)		

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

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C6—H6*B*···O1^{i}	0.97	2.49	3.428 (2)	164
C13—H13···O2^{ii}	0.93	2.50	3.387 (2)	159
C21—H21···O2^{iii}	0.93	2.44	3.364 (2)	172

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

The H atoms were positioned geometrically and treated as riding on the parent C atoms, with C—H distances in the range 0.93–0.98 Å.

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

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