

Lists of structure factors, anisotropic displacement parameters, H-atom coordinates, complete geometry and torsion angles have been deposited with the IUCr (Reference: KA1139). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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## *N*-Methyl-1,1,4-triphenyl-1,2-dihydronaphthalene-2,3-dicarboximide

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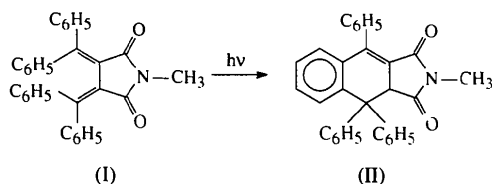
## Abstract

The stereochemistry of the molecular product, C<sub>31</sub>H<sub>23</sub>NO<sub>2</sub>, obtained by photoirradiation of 3,4-bis(diphenylmethylene)-*N*-methylsuccinimide in the solid state, together with the position of the chiral center in the molecule, have been confirmed by X-ray crystal structure analysis.

## Comment

The structure of the title compound, *N*-methyl-1,1,4-triphenyl-1,2-dihydronaphthalene-2,3-dicarboximide, (II), was studied in order to confirm the chemical identity

and stereochemistry of the product obtained by photoirradiation of powdered 3,4-bis(diphenylmethylene)-*N*-methylsuccinimide, (I), in the solid state. In particular, a question was raised with regard to the position of the double bond within the ring system of (II), in view of a possible 1,3-shift isomerization (*i.e.* movement of the chiral center from C5 to C22). It was also of interest to correlate the topological features of the crystalline reactant and product associated with this reaction.



The topochemistry of (I) is characterized by both a helical conformation (due to severe steric hindrance between the phenyl groups) and the close proximity of unsaturated fragments, which facilitates an intramolecular photocyclization reaction in the solid state. Studies of the structure and solid-state photochemistry of chiral and racemic crystalline polymorphs of (I) have been reported elsewhere (Toda, Tanaka, Stein & Goldberg, 1995). It has been shown that irradiation of the chiral crystalline form of (I) yields optically active (II), while irradiation of racemic (I) leads to the formation of racemic crystals of (II). We report here on the detailed structure of racemic (II). Suitable crystals of the optically pure enantiomorph of (II) could not be obtained.

The present analysis indicates clearly that all C—C distances within the five-membered ring correspond to single bonds, with values in the range 1.481 (4)–1.514 (4) Å. The double bond is located adjacent to the

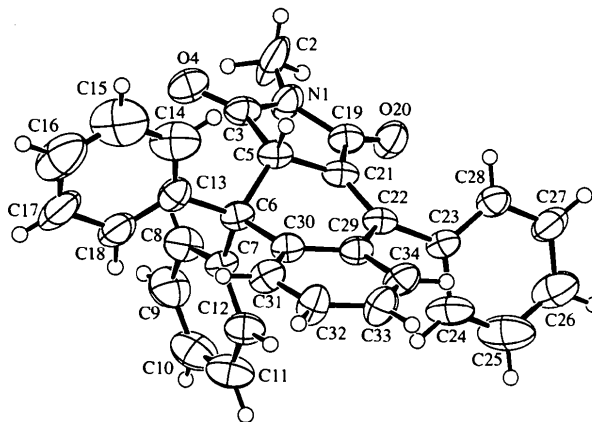


Fig. 1. The molecular structure of compound (II) showing 50% probability displacement ellipsoids and the labelling of the non-H atoms.

phenyl substituent; C21=C22 1.346 (4) Å. Correspondingly, the three bonds around C22 are coplanar, the sum of the bond angles being 360.1 (3)°. The only carbon sites with tetrahedral geometry along the molecular framework are at the C6 and chiral C5 atoms (Fig. 1). All the bond lengths and bond angles conform to standard values. A search of the August 1994 version of the Cambridge Structural Database (Allen & Kennard, 1993) did not reveal any previous crystallographic determinations of a 1,2-dihydronaphthalene-2,3-dicarboximide moiety.

The topological differences between the solid-state structures of racemic (I) and racemic (II) were not sufficiently indicative of a possible phase transformation pathway of the crystalline bulk that could be associated with this photochemical conversion.

## Experimental

Detailed experimental procedures have been reported earlier (Toda, Tanaka, Stein & Goldberg, 1995). Racemic (II) was crystallized from 2-propanol.

### Crystal data

C<sub>31</sub>H<sub>23</sub>NO<sub>2</sub>  
*M<sub>r</sub>* = 441.53  
 Monoclinic  
*P*2<sub>1</sub>/*c*  
*a* = 9.305 (3) Å  
*b* = 25.934 (4) Å  
*c* = 9.9370 (10) Å  
 $\beta$  = 99.020 (10)°  
*V* = 2368.3 (9) Å<sup>3</sup>  
*Z* = 4  
*D<sub>x</sub>* = 1.238 Mg m<sup>-3</sup>

Mo *K*α radiation  
 $\lambda$  = 0.71070 Å  
 Cell parameters from 25 reflections  
 $\theta$  = 7.4–10.3°  
 $\mu$  = 0.072 mm<sup>-1</sup>  
*T* = 293 (2) K  
 Prism  
 0.40 × 0.15 × 0.10 mm  
 Colorless

### Data collection

Enraf–Nonius CAD-4 diffractometer  
 $\omega/2\theta$  scans  
 Absorption correction: none  
 3732 measured reflections  
 3533 independent reflections  
 2420 observed reflections  
 [*I* > 2σ(*I*)]

*R*<sub>int</sub> = 0.0271  
 $\theta_{\max}$  = 24.97°  
*h* = -11 → 10  
*k* = 0 → 30  
*l* = 0 → 11  
 3 standard reflections  
 frequency: 80 min  
 intensity decay: none

### Refinement

Refinement on *F*<sup>2</sup>  
*R*(*F*) = 0.0590  
 $wR(F^2)$  = 0.1784  
*S* = 0.985  
 3533 reflections  
 308 parameters  
 H-atom parameters not refined  
 $w = 1/[\sigma^2(F_o^2) + (0.1000P)^2]$   
 where  $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{\max}$  = -0.057  
 $\Delta\rho_{\max}$  = 0.200 e Å<sup>-3</sup>  
 $\Delta\rho_{\min}$  = -0.261 e Å<sup>-3</sup>  
 Atomic scattering factors from *International Tables for Crystallography* (1992), Vol. C, Tables 4.2.6.8 and 6.1.1.4)

Table 1. Fractional atomic coordinates and equivalent isotropic displacement parameters (Å<sup>2</sup>)

$$U_{eq} = (1/3)\sum_i\sum_j U_{ij}a_i^*a_j^*a_i \cdot a_j$$

	<i>x</i>	<i>y</i>	<i>z</i>	<i>U</i> <sub>eq</sub>
N1	0.2161 (3)	0.71102 (10)	0.7025 (3)	0.0516 (7)
C2	0.0826 (4)	0.7347 (2)	0.6349 (4)	0.0917 (15)
C3	0.2267 (3)	0.68798 (11)	0.8279 (3)	0.0445 (7)
O4	0.1272 (2)	0.68444 (9)	0.8930 (2)	0.0570 (6)
C5	0.3823 (3)	0.66995 (11)	0.8677 (3)	0.0386 (7)
C6	0.4128 (3)	0.61364 (11)	0.9179 (3)	0.0388 (7)
C7	0.3482 (3)	0.57547 (11)	0.8052 (3)	0.0402 (7)
C8	0.1997 (4)	0.57516 (13)	0.7562 (3)	0.0565 (9)
C9	0.1415 (4)	0.5410 (2)	0.6545 (4)	0.0725 (11)
C10	0.2296 (5)	0.50707 (15)	0.5998 (4)	0.0746 (12)
C11	0.3733 (5)	0.50611 (14)	0.6483 (4)	0.0637 (10)
C12	0.4343 (4)	0.53988 (13)	0.7504 (3)	0.0509 (8)
C13	0.3517 (3)	0.60395 (13)	1.0520 (3)	0.0466 (8)
C14	0.3693 (5)	0.6400 (2)	1.1545 (4)	0.0761 (12)
C15	0.3181 (6)	0.6318 (2)	1.2752 (4)	0.100 (2)
C16	0.2487 (6)	0.5868 (3)	1.2963 (5)	0.104 (2)
C17	0.2327 (5)	0.5501 (2)	1.1987 (5)	0.0842 (14)
C18	0.2849 (4)	0.55825 (14)	1.0760 (3)	0.0593 (9)
C19	0.3417 (3)	0.70767 (12)	0.6432 (3)	0.0452 (7)
O20	0.3508 (2)	0.72386 (9)	0.5307 (2)	0.0589 (7)
C21	0.4501 (3)	0.68078 (10)	0.7441 (3)	0.0389 (7)
C22	0.5835 (3)	0.66311 (11)	0.7319 (3)	0.0385 (7)
C23	0.6531 (3)	0.67110 (11)	0.6082 (3)	0.0418 (7)
C24	0.6959 (4)	0.62870 (14)	0.5386 (4)	0.0643 (10)
C25	0.7572 (5)	0.6355 (2)	0.4217 (4)	0.0837 (13)
C26	0.7774 (5)	0.6835 (2)	0.3740 (4)	0.0718 (11)
C27	0.7396 (4)	0.72577 (14)	0.4436 (4)	0.0633 (10)
C28	0.6770 (3)	0.71966 (12)	0.5597 (3)	0.0485 (8)
C29	0.6595 (3)	0.63359 (11)	0.8483 (3)	0.0384 (7)
C30	0.5804 (3)	0.61004 (11)	0.9420 (3)	0.0389 (7)
C31	0.6557 (4)	0.58254 (13)	1.0500 (3)	0.0489 (8)
C32	0.8052 (4)	0.57939 (14)	1.0697 (3)	0.0591 (9)
C33	0.8838 (4)	0.60347 (15)	0.9815 (3)	0.0580 (9)
C34	0.8107 (3)	0.62978 (13)	0.8713 (3)	0.0505 (8)

Data collection: *CAD-4 Software* (Enraf–Nonius, 1989). Cell refinement: *CAD-4 Software*. Data reduction: *CADINT*, local program. Program(s) used to solve structure: *SHELXS86* (Sheldrick, 1990). Program(s) used to refine structure: *SHELXL93* (Sheldrick, 1993). Molecular graphics: *ORTEPII* (Johnson, 1976). Software used to prepare material for publication: *SHELXL93* (Sheldrick, 1993) and *PARST* (Nardelli, 1983).

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