

conformation. This result might support the surprising findings, from the proton two-dimensional nuclear Overhauser effect, of a dipolar connection between the protons of C(29) and those of C(5) [or C(3)] (Sega, Gaggelli & Valensin, 1985).

This stresses the importance of the effect of the length of the alkyloxy side chain on the pharmacological activity.

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Structure of 1,8-Bis(1'-H-1',2',3'-triazolyl)naphthalene*

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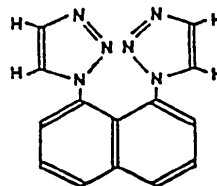
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Abstract. C₁₄H₁₀N₆, *M_r* = 262.27, orthorhombic, *Fdd2*, *a* = 5.489 (1), *b* = 25.241 (1), *c* = 17.675 (1) Å, *U* = 2448.6 (2) Å³, *Z* = 8, *D_x* = 1.423 g cm⁻³, *Cu Kα*, *λ* = 1.5418 Å, *μ* = 7.65 cm⁻¹, *F*(000) = 1088, *T* = 295 K, *R* = 0.069 for 411 observed independent reflections. The title compound has twofold symmetry in the molecule. The naphthalene framework is distorted. The fusion angle of 129.0° is large compared with those of other *peri*-substituted naphthalenes. Severe steric repulsion occurs between the two triazole rings which are in a *trans* conformation to each other.

Introduction. Naphthalenes substituted by bulky groups at the 1 and 8 positions (*peri* substituents) exhibit several unique properties in both reactivity and structure. Recently, we have succeeded in synthesizing novel 1,1'-(1,8-naphthylene)di-1H-1,2,3-triazoles (Honda, Nakanishi, Nagawa & Yabe, 1984). These compounds show very interesting rotational isomerism in solution (Nakanishi, Honda, Yabe, Nagawa & Yamamoto, 1986). In the present paper, we describe the crystal and molecular structure of (I), with the intention

of confirming the conformation of the two triazole rings and the distortion of the naphthalene framework.



(I)

Experimental. Title compound synthesized by method previously reported (Honda *et al.*, 1984); needle-shaped colorless crystals obtained by sublimation. Crystal 0.85 × 0.30 × 0.08 mm. Data collection: Rigaku four-circle diffractometer, graphite-monochromated *Cu Kα* radiation; cell parameters determined by least squares from 2θ values for 25 reflections (84 < 2θ < 110°); systematic absences of *hkl* for *h + k* and *k + l* odd, *Ok_l* for *k + l* ≠ 4*n* and *h0l* for *l + h* ≠ 4*n*; intensity data 4 < 2θ < 120°, ω-2θ scan, 0 ≤ *h* ≤ 7, 0 ≤ *k* ≤ 35, 0 ≤ *l* ≤ 24, scan speed 2° min⁻¹; three standard reflections (040, 020 and 220) showed only statistical

* IUPAC name: 1,1'-(1,8-naphthylene)di-1H-1,2,3-triazole.

variations within $\pm 1\%$; 1089 (including systematically absent) reflections collected, 411 with $|F_o| > 3\sigma(|F_o|)$ used. Data not corrected for absorption. Space group *Fdd2* has 16-fold general positions. However, there are eight molecules in the unit cell, so the molecule is required to have twofold symmetry. Structure solved by direct methods using *MULTAN78* (Main, Hull, Lessinger, Germain, Declercq & Woolfson, 1978). *E* map using $94 |E| > 1.2$ revealed positions of all non-H atoms. H atoms placed at idealized positions. Block-diagonal least-squares refinement (on *F*) using scattering factors from *International Tables for X-ray Crystallography* (1962), anisotropic temperature factors for non-H atoms, isotropic for H, gave $R = 0.069$ and $wR = 0.058$; $w = 1/(a|F_o|^2 + b|F_o| + c)$; $a = 0.0066$, $b = -0.464$, $c = 13.93$. Max. and min. heights in final difference map $\pm 0.2 e \text{ \AA}^{-3}$. Max. $\Delta/\sigma = 0.26$ [z of N(3)]. Calculations carried out on a Facom M-380 with *UNICSIII* (Sakurai & Kobayashi, 1979).

Table 1. Fractional atomic coordinates ($\times 10^4$) and equivalent isotropic thermal parameters with e.s.d.'s in parentheses

	x	y	z	$B_{eq}(\text{\AA}^2)$
C(1)	1555 (12)	378 (3)	3846 (4)	3.5 (2)
C(2)	3181 (12)	680 (3)	3451 (4)	4.1 (3)
C(3)	3317 (16)	627 (4)	2651 (4)	5.2 (2)
C(4)	1708 (13)	317 (3)	2283 (4)	4.4 (2)
C(5)	0	0	3500	3.5 (2)
C(6)	0	0	2680 (5)	3.9 (2)
C(7)	1852 (13)	741 (3)	5788 (4)	4.5 (2)
C(8)	3082 (12)	586 (3)	5155 (4)	4.1 (2)
N(1)	1320 (8)	501 (2)	4644 (3)	3.1 (1)
N(2)	-903 (10)	610 (2)	4927 (4)	3.7 (1)
N(3)	-558 (12)	755 (2)	5629 (4)	4.2 (1)

$$B_{eq} = \frac{1}{3} \sum_i \beta_{ij} a_i \cdot a_j$$

Table 2. Selected bond distances (\AA) and angles ($^\circ$) with e.s.d.'s in parentheses

C(1)–C(2)	1.366 (10)	C(5)–C(6)	1.449 (10)
C(1)–C(5)	1.418 (7)	C(7)–C(8)	1.364 (11)
C(1)–N(1)	1.451 (9)	C(7)–N(3)	1.352 (9)
C(2)–C(3)	1.423 (11)	C(8)–N(1)	1.340 (9)
C(3)–C(4)	1.346 (12)	N(1)–N(2)	1.347 (7)
C(4)–C(6)	1.419 (9)	N(2)–N(3)	1.307 (9)
C(2)–C(1)–C(5)	123.3 (6)	C(4)–C(6)–C(5)	119.7 (5)
C(2)–C(1)–N(1)	115.8 (6)	C(8)–C(7)–N(3)	108.7 (7)
C(5)–C(1)–N(1)	120.6 (5)	C(7)–C(8)–N(1)	104.0 (6)
C(1)–C(2)–C(3)	119.3 (7)	C(1)–N(1)–C(8)	128.7 (5)
C(2)–C(3)–C(4)	120.1 (8)	C(1)–N(1)–N(2)	119.1 (5)
C(3)–C(4)–C(6)	121.4 (7)	C(8)–N(1)–N(2)	111.7 (6)
C(1)–C(5)–C(1 ⁱ)	129.0 (4)	N(1)–N(2)–N(3)	106.2 (5)
C(1)–C(5)–C(6)	115.5 (3)	C(7)–N(3)–N(1)	109.4 (6)
C(4)–C(6)–C(4 ⁱ)	120.7 (8)		

Symmetry code: (i) $-x, -y, z$

Discussion. Final atomic positions and equivalent isotropic thermal parameters are given in Table 1.* Bond distances and angles are given in Table 2. An *ORTEP* (Johnson, 1965) drawing of the molecule with the numbering of atoms is shown in Fig. 1.

The dihedral angle between the mean plane of the naphthalene ring and that of two triazole rings is $50.6(2)^\circ$, which is a little smaller than that of 1,4,5,8-tetraphenylnaphthalene, 58° (Evrard, Piret & Van Meerssche, 1971). The torsion angle C(5)–C(1)–N(1)–N(2), $-48.9(8)^\circ$, shows that N(2) is much closer to the C(5)–C(6) axis than the C(2)–C(3) axis. Since the torsion angle C(6)–C(5)–C(1)–N(1) is $166.0(5)^\circ$, the C(1)–N(1) bond deviates from the naphthalene ring by 14° . The two triazole rings face each other with a dihedral angle of $35.6(3)^\circ$. This indicates the presence of steric repulsion between the two triazole rings. The two triazole rings take a *trans* conformation to each other, which is consistent with the fact that the molecule has twofold rotational symmetry about the axis through the C(5)–C(6) bond.

The fusion angle C(1)–C(5)–C(1ⁱ) is 129.0° , which is subject to distortion by steric strain. The angle is fairly large compared with those of other *peri*-substituted naphthalenes, such as 1,8-bis(dimethylamino)naphthalene, 125.8° (Einspahr, Robert, Marsh & Roberts, 1973), 1,8-dimorpholinonaphthalene, 127.0° (Wong-Ng, Nyburg, Awwal, Jankie & Kresge, 1982) and 1,8-dipiperidinonaphthalene, 126.9° (Wong-Ng *et al.*, 1982). The non-bonding distance of C(1)–

* Lists of structure factors, anisotropic thermal parameters, H-atom coordinates and details of least-squares planes have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 42652 (9 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

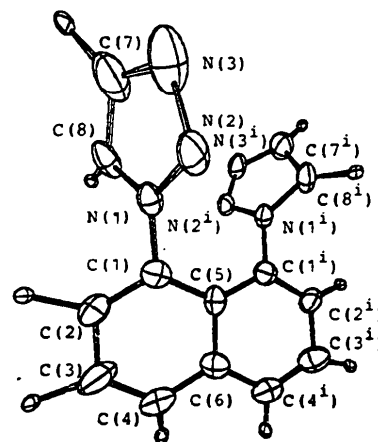


Fig. 1. A perspective view of the molecule with the numbering scheme.

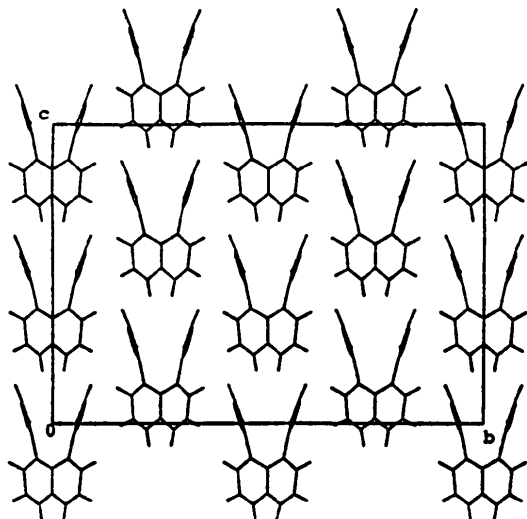


Fig. 2. Packing of the molecule in the unit cell (viewed down *a*).

$C(1^b)$ is longer than that of $C(4)-C(4^b)$ by 0.10 \AA . $C(1)$ and $C(1^b)$ are out of the mean plane formed by $C(4)$, $C(5)$, $C(6)$ and $C(4^b)$ by 8° in opposite directions. This bending is quite large, although it is smaller than that of the overcrowded compound 1,3,6,8-tetra-*tert*-butylnaphthalene, 22° (Handal, White, Franck, Yuh & Allinger, 1977). These distortions in the naphthalene

ring are rationalized by the existence of the bulky triazole rings in *peri* position.

Fig. 2 (a projection along *a*) shows the packing of the molecules in the unit cell. The packing of the molecules in the crystal reveals normal intermolecular contacts equal to the sum of the van der Waals radii.

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The Structure of Carbazole at 168 K

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Abstract. $C_{12}H_9N$, $M_r = 167.21$, orthorhombic, *Pnma*, $a = 7.628(1)$, $b = 18.935(2)$, $c = 5.657(1) \text{ \AA}$, $V = 817.0(2) \text{ \AA}^3$, $Z = 4$, $D_x = 1.36 \text{ g cm}^{-3}$, $\lambda(\text{Mo } K\alpha) = 0.71069 \text{ \AA}$, $\mu = 0.744 \text{ cm}^{-1}$, $F(000) = 352$, $T = 168 \text{ K}$, $R = 0.10$, $wR = 0.045$ for 1047 unique reflections ($R = 0.050$ for reflections having $I > 3\sigma$). The carbazole molecule has mirror symmetry, and is slightly non-planar, the dihedral angle between the two planes in the molecule being 2.5° . The space group is identical to that found for the room-temperature structure. The

H atom on nitrogen does not show the deviation from the mean molecular plane reported for the room-temperature structure.

Introduction. There has been a continuing interest in our laboratory in studying by low-field EPR methods the host-composition dependence of the zero-field splittings of dilute triplet guest molecules in a binary host crystal of mutually soluble components (Lundstedt, 1984). Three molecules that satisfy the conditions formulated by Kitaigorodsky (1973) for the formation of a continuous series of solid solutions and that are otherwise acceptable as potential host systems

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