

Fig. 2. A stereoview of part of the unit cell showing the inter-anion hydrogen bonding which develops infinite chains in the *a* direction.

In addition, the bond lengths in the C(5),C(7),-O(7),N(8),C(9) region [Fig. 1, Table 2, C(5)–C(7) 1.471 (7), C(7)–N(8) 1.350 (7), C(7)–O(7) 1.239 (6), N(8)–C(9) 1.426 (7) Å] are indicative of contributions from forms such as (4*a*) and (4*b*) to the ground-state structure of the anion. The length of the exocyclic C(5)–C(7) bond [1.471 (7) Å] is close to that expected for a C(*sp*<sup>2</sup>)–C(*sp*<sup>2</sup>) single bond, and would imply little conjugation between the resonance forms shown in (3) and (4). The anion is not planar in the solid mainly because of a small rotation [18.4 (6)°] of the phenyl ring about the N(8)–C(9) bond.

The bond lengths in the phenyl ring show a short–long alternation [means 1.350 (8), 1.398 (8) Å] but whether this is a genuine effect or a consequence of the phenyl carbon atoms having maximum  $U_{ij}$  values up to twice as large as those of the tightly hydrogen-bonded pyrimidine ring is not clear. The C atoms of the cation have large vibration parameters, as anticipated (maximum  $U_{ii}$  0.219 Å<sup>2</sup>), and the C–C bond lengths (1.44–1.51 Å) reflect this marked anisotropy and are not at all well determined. The N atom [N(21)] which is hydrogen-bonded to the anion has close to tetrahedral geometry [C–N–C 107.8–113.2 (5)°].

In the crystals, all N–H groups are involved in hydrogen bonding (see Fig. 2). The cation forms a bifurcated hydrogen bond with anion oxygens O(6) and

O(7). Thus the hydrogen H(N21) on cation nitrogen N(21) is essentially equidistant from O(6) (2.06 Å) and O(7) (2.13 Å) with N(21)···O(6) 2.921 (7) Å and N(21)···O(7) 2.811 (7) Å. O(7) is also involved in an intermolecular O···N–O hydrogen bond with the ethanol of solvation, O(7)···O(ethanol) 2.883 (7) Å. Although the hydrogens of the ethanol molecule were not located, because it is only loosely trapped in the crystal lattice, the O···O distance is indicative of O···H–O hydrogen bonding. The hydrogen H(N8) on N(8) is involved in an intra-ion hydrogen bond to O(4) [N(8)···O(4) 2.611 (7), H(N8)···O(4) 1.80 Å]; this is clearly of importance in maintaining the planarity of the exocyclic O(7)–C(7)–N(8) moiety with the pyrimidine ring. The anions are linked into infinite hydrogen-bonded chains extending along the crystallographic *a* axis by inter-ion N–H···O hydrogen bonds [N(1)–H···O(4), N···O 2.758 (7), H···O 1.84 Å, and N(3)–H···O(6), N···O 2.957 (7), H···O 2.02 Å].

Although the structure shows a number of features which might not be easily predictable, the precise reason for antineoplastic activity is not immediately obvious from the structural data. Further work is in progress on related compounds.

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## Structure of 1-(1-Naphthyl)-1*H*-1,2,3-triazole

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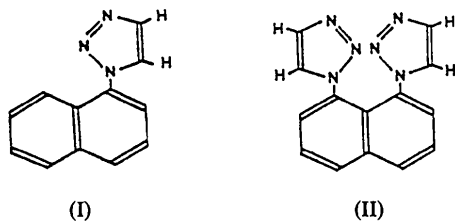
(Received 16 June 1986; accepted 4 August 1986)

**Abstract.** C<sub>12</sub>H<sub>9</sub>N<sub>3</sub>,  $M_r = 195.22$ , orthorhombic, *Pbca*,  $a = 9.089$  (1),  $b = 15.545$  (1),  $c = 13.845$  (1) Å,  $V = 1955.9$  (2) Å<sup>3</sup>,  $Z = 8$ ,  $D_x = 1.326$  g cm<sup>-3</sup>,  $\lambda(\text{Mo } K\alpha)$

$= 0.71069$  Å,  $\mu = 0.90$  cm<sup>-1</sup>,  $F(000) = 816$ ,  $T = 295$  K,  $R = 0.053$  for 785 observed independent reflections. The dihedral angle between the mean plane

of the naphthalene ring and that of the triazole ring in the title compound (I) is  $56.8(2)^\circ$ . The fusion angle of  $123.7(5)^\circ$  is distinctly small compared with that of 1,1'-(1,8-naphthylene)di-1H-1,2,3-triazole (II) due to less steric overcrowding.

**Introduction.** Recently, we have succeeded in synthesizing novel 1,1'-(1,8-naphthylene)di-1H-1,2,3-triazoles (Honda, Nakanishi, Nagawa & Yabe, 1984). Structure determination showed that the naphthalene framework in (II) is distorted (Nagawa, Goto, Honda & Nakanishi, 1986), owing to the steric repulsion between the two triazole rings. On the other hand, only a few crystal structures of 1H-1,2,3-triazoles have been reported (Wamhoff, 1984). The present paper deals with the crystal structure of the title compound (I), in comparison with that of (II).



**Experimental.** Title compound was synthesized by the method previously reported (Honda *et al.*, 1984); needle-shaped colorless crystals (m.p. 375 K) obtained by slow evaporation of a methylene chloride solution. Crystal  $0.80 \times 0.35 \times 0.10$  mm. Data collection: Rigaku four-circle diffractometer, graphite-monochromated Mo  $K\alpha$  radiation; cell parameters determined by least squares from  $2\theta$  values for 25 reflections ( $40 < 2\theta < 50^\circ$ ); systematic absences of  $0kl$  for  $k$  odd,  $h0l$  for  $l$  odd,  $hko$  for  $h$  odd; intensity data  $2 < 2\theta < 55^\circ$ ,  $\omega$ - $2\theta$  scan,  $0 \leq h \leq 11$ ,  $0 \leq k \leq 20$ ,  $0 \leq l \leq 18$ , scan speed  $2^\circ \text{ min}^{-1}$ ; three standard reflections (002, 020 and 200) showed only statistical variations within  $\pm 1\%$ ; 2126 (including systematically absent) reflections collected, 785 with  $|F_o| > 3\sigma(|F_o|)$  used. No correction for absorption. Structure solved by direct methods using *MULTAN78* (Main, Hull, Lessinger, Germain, Declercq & Woolfson, 1978). *E* map using  $141 |E| > 1.2$  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,  $R = 0.053$  and  $wR = 0.050$ ;  $w = 1/(a|F_o|^2 + b|F_o| + c)$ ;  $a = 0.0022$ ,  $b = -0.138$ ,  $c = 2.53$ . Max. and min. heights in final difference map  $\pm 0.2 \text{ e } \text{Å}^{-3}$ . Max.  $\Delta/\sigma = 0.12$  [ $y$  of C(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

$$B_{\text{eq}} = \frac{1}{3} \sum_i \sum_j \beta_{ij} \mathbf{a}_i \cdot \mathbf{a}_j$$

	<i>x</i>	<i>y</i>	<i>z</i>	$B_{\text{eq}}(\text{Å}^2)$
C(1)	2974 (4)	948 (3)	2490 (4)	3.7 (1)
C(2)	2443 (5)	1257 (3)	3347 (3)	4.5 (1)
C(3)	2694 (5)	797 (3)	4200 (3)	5.0 (1)
C(4)	3476 (6)	56 (3)	4189 (4)	5.0 (1)
C(5)	4895 (6)	-1045 (3)	3281 (4)	5.9 (2)
C(6)	5411 (6)	-1352 (3)	2430 (6)	6.6 (2)
C(7)	5117 (6)	-928 (3)	1568 (5)	5.8 (2)
C(8)	4331 (6)	-172 (3)	1550 (4)	4.8 (1)
C(9)	3787 (5)	176 (3)	2431 (4)	3.8 (1)
C(10)	4059 (5)	-273 (3)	3308 (4)	4.2 (1)
C(11)	1695 (5)	2183 (3)	497 (4)	5.0 (2)
C(12)	1381 (5)	1711 (3)	1285 (4)	4.9 (1)
N(1)	2693 (4)	1436 (2)	1629 (3)	3.9 (1)
N(2)	3786 (4)	1747 (3)	1065 (3)	5.3 (1)
N(3)	3174 (5)	2199 (3)	379 (3)	5.4 (1)

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

C(1)–C(2)	1.369 (7)	C(7)–C(8)	1.376 (7)
C(1)–C(9)	1.413 (6)	C(8)–C(9)	1.422 (7)
C(1)–N(1)	1.435 (6)	C(9)–C(10)	1.422 (7)
C(2)–C(3)	1.399 (7)	C(11)–C(12)	1.347 (8)
C(3)–C(4)	1.354 (8)	C(11)–N(3)	1.354 (7)
C(4)–C(10)	1.425 (7)	C(12)–N(1)	1.352 (6)
C(5)–C(6)	1.356 (10)	N(1)–N(2)	1.352 (6)
C(5)–C(10)	1.420 (7)	N(2)–N(3)	1.306 (6)
C(6)–C(7)	1.388 (9)		
C(2)–C(1)–C(9)	122.2 (5)	C(8)–C(9)–C(10)	119.0 (4)
C(2)–C(1)–N(1)	118.2 (4)	C(4)–C(10)–C(5)	121.6 (5)
C(9)–C(1)–N(1)	119.6 (4)	C(4)–C(10)–C(9)	119.4 (4)
C(1)–C(2)–C(3)	119.7 (4)	C(5)–C(10)–C(9)	119.0 (5)
C(2)–C(3)–C(4)	120.7 (5)	C(12)–C(11)–N(3)	108.5 (5)
C(3)–C(4)–C(10)	120.7 (5)	C(11)–C(12)–N(1)	105.7 (4)
C(6)–C(5)–C(10)	120.4 (5)	C(1)–N(1)–C(12)	128.0 (4)
C(5)–C(6)–C(7)	120.8 (5)	C(1)–N(1)–N(2)	122.5 (4)
C(6)–C(7)–C(8)	121.4 (5)	C(12)–N(1)–N(2)	109.4 (4)
C(7)–C(8)–C(9)	119.3 (5)	N(1)–N(2)–N(3)	107.4 (4)
C(1)–C(9)–C(8)	123.7 (5)	C(11)–N(3)–N(2)	109.0 (4)
C(1)–C(9)–C(10)	117.3 (5)		

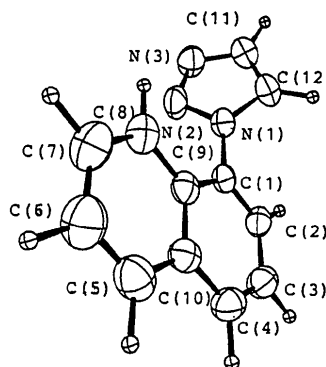


Fig. 1. A perspective view of the molecule, with the numbering scheme.

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

The deviations of each atom in the triazole ring from the mean plane are very small, and the three bonds of N(1) are almost coplanar. From the results above, it is suggested that the triazole ring has aromaticity and the hybridization of the N(1) atom is of the  $sp^2$  type.

The dihedral angle between the mean plane of the naphthalene ring and that of the triazole ring is  $56.8(2)^\circ$ .

As the triazole ring is asymmetric about the C(1)–N(1) axis, it is interesting to know which side of the triazole ring is near to the C(9)–C(10) axis. The torsion angle C(9)–C(1)–N(1)–N(2),  $-58.8(6)^\circ$ , shows that N(2) is much closer to the C(9)–C(10) axis than the C(2)–C(3) axis.

The angle C(1)–C(9)–C(8),  $123.7(5)^\circ$ , is distinctly small compared with that of (II),  $129.0^\circ$  (Nagawa *et al.*, 1986). This is because the steric overcrowding of (I) is less than that of (II).

The packing of the molecules in the crystal is shown in Fig. 2. The crystal structure is stabilized mainly by van der Waals forces; the shortest intermolecular distance is  $3.506(6) \text{ \AA}$  for C(2)( $x, y, z$ )...N(2)( $\frac{1}{2}+x, y, \frac{1}{2}-z$ ).

\* Lists of structure factors, anisotropic thermal parameters, H-atom coordinates, and details of least-squares planes have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 43315 (11 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

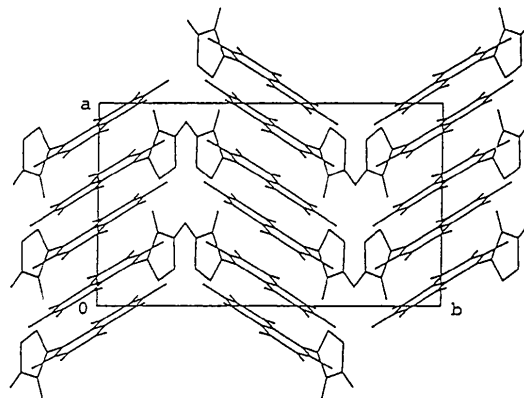


Fig. 2. Packing of molecules in the unit cell. (Positive  $c$  axis is taken downward.)

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### 4,6-Dimethoxy-1,3,5-triazin-2(1H)-one

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**Abstract.**  $C_7H_7N_3O_3$ ,  $M_r = 157.13$ , orthorhombic,  $Pca2_1$ ,  $a = 12.027(3)$ ,  $b = 14.579(3)$ ,  $c = 7.776(2) \text{ \AA}$ ,  $V = 1363.5(5) \text{ \AA}^3$ ,  $Z = 8$ ,  $D_x = 1.530 \text{ Mg m}^{-3}$ ,  $\lambda(\text{Mo } K\alpha) = 0.71069 \text{ \AA}$ ,  $\mu =$

$0.14 \text{ mm}^{-1}$ ,  $F(000) = 656$ ,  $T = 294 \text{ K}$ . Final  $R = 0.034$  for 1184 observed [ $I \geq 2\sigma(I)$ ] reflections. The two independent molecules of the asymmetric unit form dimers by means of two N–H...O(keto) hydrogen

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