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A mesogenic alkenyl compound

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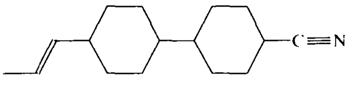
Abstract

In the title compound, 4′-propen-1-ylbicyclohexane-4-carbonitrile, $C_{16}H_{25}N$, both cyclohexane rings adopt chair conformations. The dihedral angle between the rings is 105.90 (5)°. The normals to the least-squares mean planes through the propenyl chain and the adjacent cyclohexane ring make an angle of 97.95 (15)°. Molecules of the compound are packed along the *c* axis. The molecular arrangement is a precursor of a nematic phase.

Comment

The title compound has been reported as a new type of liquid-crystal compound (Schadt *et al.*, 1989). The introduction of a double bond at specific side-chain positions markedly affects the properties of liquid crystals. Different experimental techniques have been applied to these compounds (Schadt *et al.*, 1989; Nath *et al.*, 1995). Upon heating it adopts a nematic liquid-crystal phase from 64.9° until it becomes isotropic at 99.7°. As part of our program to solve the structures of the homologous series of cyanoalkenyl compounds (Gupta *et al.*, 1994), and in order to clarify the precise relationship between nematic structure and the molecular interactions, we have studied the title compound (designated as 1d1CC).



1d1CC

The compound can be considered in three parts, namely the alkenyl chain, the central core and the cyano group (Fig. 1). Both cyclohexane rings adopt chair conformations. The dihedral angle between the rings is 105.90 (5)°. The normals to the least-squares mean planes through the propenyl chain and the adjacent cyclohexane ring make an angle of 97.95 (15)°. Atom C16 of the cyano group is displaced slightly upwards from the plane of the cyclohexane ring *B* (C10–C15) whereas C7 is displaced slightly downwards. The alkenyl-group atom C3 is displaced downwards and C10 is displaced upwards from the plane of the cyclohexane ring *A* (C4–C9). The puckering parameters of the six-

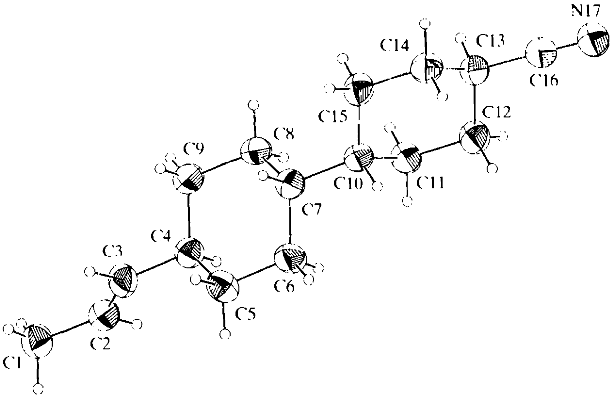


Fig. 1. Perspective drawing (*ZORTEP*; Zsolnai & Huttner, 1994) of the title compound showing the atomic numbering scheme. Displacement ellipsoids are shown at the 30% probability level and H atoms have an arbitrary radius.

membered rings calculated using the method of Cremer & Pople (1975) appear in Table 2. Bond lengths and bond angles (including those involving the double bond) are consistent with our previous observations and have expected values (Gupta *et al.*, 1994).

The packing of the title compound in the *ac* plane is shown in Fig. 2. It can be seen that pairs of molecules, related by centres of symmetry, give rise to a sheet of parallel molecules in the *ac* plane and that these sheets are stacked in an imbricated fashion along the crystallographic *c* axis, as commonly found in a nematogen. With increasing thermal energy, the transformation to the liquid-crystal state is presumably accomplished by the breakdown of the molecular stacking along the *c* axis. This type of packing is normally observed where a transition occurs from the crystalline to the nematic phase in which the molecules possess no positional correlation but have orientational ordering about a director (Bryan & Forcier, 1980). The length of the molecule in the crystalline state is found to be 13.53 Å, corresponding to its most extended possible conformation. It is found that mean distances, *i.e.* the uncorrected distances of C atoms approach the lower bound (*PARST*; Nardelli, 1983), indicating that interatomic contacts are such that C atoms repel each other and undergo correlated parallel motion. Therefore, in the title compound no molecular association, as a result of van der Waals type of interaction, persists in the liquid-crystalline state. A low-angle X-ray diffraction study (Nath *et al.*, 1995) confirms the fact that the average length of the molecules in the nematic phase (13.8 Å) is almost equal to the length of a single molecule.

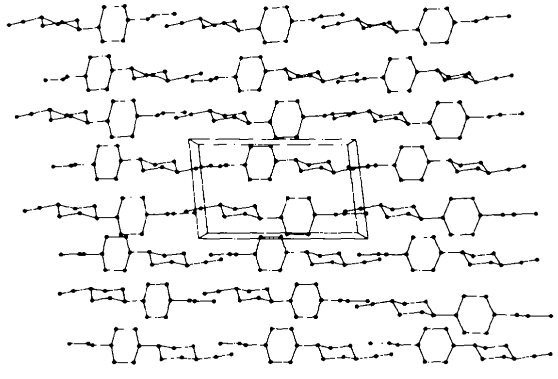


Fig. 2. Packing diagram (*SHELXTL-Plus*; Sheldrick, 1995) of the title compound viewed along [010].

Experimental

The title compound was prepared as described in Schadt *et al.* (1989). Crystals of the title compound were obtained from an acetone solution by slow evaporation at room temperature.

Crystal data

$C_{16}H_{25}N$
$M_r = 231.37$
Triclinic
$P\bar{1}$
$a = 6.834\ (1)$ Å
$b = 9.672\ (1)$ Å
$c = 11.317\ (1)$ Å
$\alpha = 91.72\ (1)^\circ$
$\beta = 96.56\ (1)^\circ$
$\gamma = 97.27\ (1)^\circ$
$V = 736.41\ (15)$ Å^3
$Z = 2$
$D_x = 1.043$ Mg m^{-3}
D_m not measured

Mo $K\alpha$ radiation
$\lambda = 0.71070$ Å
Cell parameters from 25 reflections
$\theta = 12.10–13.91^\circ$
$\mu = 0.060$ mm^{-1}
$T = 293\ (2)$ K
Block
0.5 × 0.4 × 0.3 mm
Colourless

Data collection

Enraf–Nonius CAD-4 diffractometer
ω/θ scans
Absorption correction: ψ scan (North *et al.*, 1968)
$T_{\min} = 0.914$, $T_{\max} = 0.992$
7587 measured reflections
3532 independent reflections

1885 reflections with $I > 2\sigma(I)$
$R_{\rm int} = 0.011$
$\theta_{\max} = 28.02^\circ$
$h = -9 \rightarrow 9$
$k = -12 \rightarrow 12$
$l = -14 \rightarrow 14$
3 standard reflections
frequency: 60 min
intensity decay: 4%

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.045$
$wR(F^2) = 0.151$
$S = 1.030$
3532 reflections
156 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0843P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\max} = -0.034$

$\Delta\rho_{\max} = 0.14$ e Å^{-3}
$\Delta\rho_{\min} = -0.12$ e Å^{-3}
Extinction correction: *SHELXL*97 (Sheldrick, 1997)
Extinction coefficient: 0.045 (8)
Scattering factors from *International Tables for Crystallography* (Vol. C)

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

C2—C3	1.295 (2)	C16—N17	1.141 (2)
C13—C16	1.464 (2)		
C3—C2—C1	126.86 (13)	C16—C13—C14	111.60 (10)
C2—C3—C4	127.51 (13)	N17—C16—C13	178.68 (14)
C16—C13—C12	110.01 (11)		
C2—C3—C4—C9	123.6 (2)	C11—C12—C13—C16	178.57 (10)
C2—C3—C4—C5	−113.0 (2)	C16—C13—C14—C15	−177.40 (10)

Table 2. *Ring-puckering parameters (Å, °) for two rings*

Ring	q_2	q_3	Q_T	θ
A	0.009 (1)	0.562 (1)	0.562 (1)	0.9 (1)
B	0.023 (2)	−0.572 (2)	0.572 (2)	177.7 (2)

The H atoms were allowed to ride on their parent atom with $U_{\rm iso}({\rm H}) = xU_{\rm eq}$(parent), where $x = 1.5$ for methyl and $x = 1.2$ for all others. Rigid-bond restraints (Sheldrick, 1997) were applied to bonds involving C4, C5, C6, C7, C10, C15, C11, C12, C13 and C16.

Data collection: *CAD*-4 *EXPRESS* (Enraf–Nonius, 1995). Cell refinement: *CAD*-4 *EXPRESS*. Data reduction: *XCAD*-4 (Harms & Wocadlo, 1996). Program(s) used to solve structure: *SHELXS*93 (Sheldrick, 1993). Program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997). Molecular graphics: *ZORTEP* (Zsolnai & Huttner, 1994), *SHELXTL-Plus* (Sheldrick, 1995). Software used to prepare material for publication: *SHELXL*97.

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Supplementary data for this paper are available from the IUCr electronic archives (Reference: BM1266). Services for accessing these data are described at the back of the journal.

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5-(4-Pyridyl)-5-phenylhydantoin†

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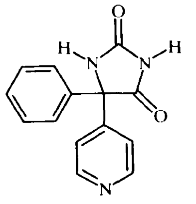
Abstract

The structure of the title compound, $C_{14}H_{11}N_3O_2$, is reported. The hydantoin ring is nearly planar. Bond distances and angles within the hydantoin ring are similar to those of the known 5,5-diphenylhydantoin. The angles between the hydantoin and the phenyl and pyridyl rings are very unequal, unlike those in 5,5-diphenylhydantoin. There is an intermolecular hydrogen-bonding network involving the amido H atoms as donors and the pyridyl-N atom and one carbonyl-O atom as acceptors; there is no intramolecular hydrogen bonding.

Comment

As part of an ongoing search for new stable *N*-halamine bactericides for treatment of aqueous solutions (Kohl *et al.*, 1980; Burkett *et al.*, 1981; Worley & Burkett, 1984; Barnela *et al.*, 1987; Worley *et al.*, 1987; Worley & Williams, 1988; Eknoian *et al.*, 1998), the title compound, (I), was prepared as a precursor. The structure was determined as part of the characterization process.



(I)

The compound crystallizes as a molecular lattice; the molecule is depicted in Fig. 1. Each molecule donates two intermolecular hydrogen bonds (H1 and H2) and accepts two (through N3 and O1); there is no intramolecular hydrogen bonding. The hydantoin ring is nearly planar; no atom deviates from the least-squares plane through the five atoms by more than 0.03 Å. If the ring is viewed as an envelope, with C3 out of the plane of the other atoms, the fold at the C2—N2 line is only 0.9 (2)°. This contrasts strongly with two oxazolidinone structures recently reported

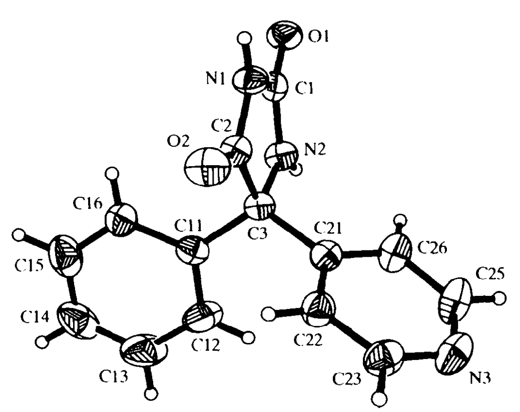


Fig. 1. View of 5-(4-pyridyl)-5-phenylhydantoin (50% probability ellipsoids).

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† IUPAC name: 5-phenyl-5-(4-pyridyl)-2,4-imidazolidinedione.

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