

Acta Crystallographica Section E

Structure Reports

Online

ISSN 1600-5368

3,5-Bis[(*E*)-4-methylbenzylidene]-4-oxo-piperidine-1-carbonitrileM. Subha Nandhini,^a N. Srinivasan,^b R. Ranjithkumar,^c S. Perumal^c and R. V. Krishnakumar^{b*}^aDepartment of Physics, Madurai Kamaraj University, Madurai 625 021, India,^bDepartment of Physics, Thiagarajar College, Madurai 625 009, India, and ^cSchool of Chemistry, Madurai Kamaraj University, Madurai 625 021, India

Correspondence e-mail: mailtorvk@yahoo.co.in

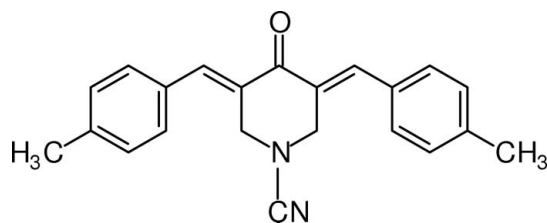
Received 29 July 2007; accepted 1 August 2007

Key indicators: single-crystal X-ray study; $T = 298$ K; mean $\sigma(\text{C}-\text{C}) = 0.002$ Å; R factor = 0.050; wR factor = 0.150; data-to-parameter ratio = 25.2.

In the title compound, $\text{C}_{22}\text{H}_{20}\text{N}_2\text{O}$, the piperidinone ring adopts an envelope conformation. The unequal twists of the 3,5-disubstituted 4-methylphenyl rings [torsion angles 15.7 (1) and 35.7 (1)°] play a role in reducing the molecular symmetry upon crystallization. The structure is a good example of a molecule where competition between intra- and intermolecular interactions is apparent.

Related literature

For related literature, see Cremer & Pople (1975); Dimmock *et al.* (1990, 2001); Suresh *et al.* (2005a, 2005b, 2006); Natarajan *et al.* (2005).



Experimental

Crystal data

 $\text{C}_{22}\text{H}_{20}\text{N}_2\text{O}$ $M_r = 328.40$ Triclinic, $P\bar{1}$ $a = 9.1976$ (2) Å $b = 9.5012$ (2) Å $c = 10.9418$ (3) Å $\alpha = 104.03$ (2)° $\beta = 95.080$ (1)° $\gamma = 108.73$ (1)° $V = 864.01$ (12) Å³ $Z = 2$ Mo $K\alpha$ radiation $\mu = 0.08$ mm⁻¹ $T = 298$ (2) K

0.28 × 0.14 × 0.12 mm

Data collection

Bruker Kappa-APEXII CCD

diffractometer

Absorption correction: multi-scan

(SADABS; Sheldrick, 2004)

 $T_{\min} = 0.88$, $T_{\max} = 0.99$

23388 measured reflections

5753 independent reflections

3427 reflections with $I > 2\sigma(I)$ $R_{\text{int}} = 0.031$

Refinement

 $R[F^2 > 2\sigma(F^2)] = 0.050$ $wR(F^2) = 0.150$ $S = 1.04$

5753 reflections

228 parameters

H-atom parameters constrained

 $\Delta\rho_{\text{max}} = 0.21$ e Å⁻³ $\Delta\rho_{\text{min}} = -0.20$ e Å⁻³

Table 1

Hydrogen-bond geometry (Å, °).

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
$\text{C2}-\text{H2A}\cdots\text{N2}^{\text{i}}$	0.97	2.61	3.4630 (19)	147
$\text{C52}-\text{H52}\cdots\text{O1}^{\text{ii}}$	0.93	2.59	3.4921 (17)	163
$\text{C57}-\text{H57B}\cdots\text{N2}^{\text{iii}}$	0.96	2.57	3.524 (2)	174

Symmetry codes: (i) $-x, -y, -z$; (ii) $-x+1, -y+1, -z+1$; (iii) $-x+1, -y, -z+1$.

Data collection: APEX2 (Bruker, 2004); cell refinement: SAINT-Plus (Bruker, 2004); data reduction: SAINT; program(s) used to solve structure: SHELXS86 (Sheldrick, 1990); program(s) used to refine structure: SHELXL97 (Sheldrick, 1997); molecular graphics: PLATON (Spek, 2003); software used to prepare material for publication: SHELXL97.

The authors thank the Sophisticated Analytical Instrumentation Facility (SAIF), Indian Institute of Technology, Chennai, for the X-ray intensity data collection. RR and SP thank the Department of Science and Technology, Government of India, for financial support.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: BG2085).

References

- Bruker (2004). APEXII and SAINT-Plus (Version 7.06a). Bruker AXS Inc., Madison, Wisconsin, USA.
- Cremer, D. & Pople, J. A. (1975). *J. Am. Chem. Soc.* **97**, 1354–1358.
- Dimmock, J. R., Arora, V. K., Wonko, S. L., Hamon, N. W., Quail, J. W., Jia, Z., Warrington, R. C., Fang, W. D. & Lee, J. S. (1990). *Drug Des. Delivery*, **6**, 183–194.
- Dimmock, J. R., Padmanilayam, M. P., Puthucode, R. N., Nazari, A. J., Motaganahalli, N. L., Zello, G. A., Quail, J. W., Oloo, E. O., Kraatz, H. B., Prisciak, J. S., Allen, T. M., Santos, C. L., Balsarini, J., Clercq, E. D. & Manavathu, E. K. (2001). *J. Med. Chem.* **44**, 586–593.
- Natarajan, S., Krishnakumar, R. V., Subha Nandhini, M., Alex Raja, V. P., Perumal, S. & Ravikumar, K. (2005). *Acta Cryst.* **E61**, o359–o361.
- Sheldrick, G. M. (1990). *Acta Cryst.* **A46**, 467–473.
- Sheldrick, G. M. (1997). SHELXL97. University of Göttingen, Germany.
- Sheldrick, G. M. (2004). SADABS. University of Göttingen, Germany.
- Spek, A. L. (2003). *J. Appl. Cryst.* **36**, 7–13.
- Suresh, J., Alex Raja, V. P., Krishnakumar, R. V., Natarajan, S., Perumal, S. & Mostad, A. (2005a). *Acta Cryst.* **E61**, o3050–o3052.
- Suresh, J., Alex Raja, V. P. A., Krishnakumar, R. V., Natarajan, S., Perumal, S. & Mostad, A. (2005b). *Acta Cryst.* **E61**, o2458–o2460.
- Suresh, J., Raja, V. P. A., Natarajan, S., Perumal, S., Mostad, A. & Krishnakumar, R. V. (2006). *Acta Cryst.* **E62**, o1295–o1297.

supplementary materials

Acta Cryst. (2007). E63, o3749 [doi:10.1107/S1600536807037956]

3,5-Bis[(*E*)-4-methylbenzylidene]-4-oxopiperidine-1-carbonitrile

M. S. Nandhini, N. Srinivasan, R. Ranjithkumar, S. Perumal and R. V. Krishnakumar

Comment

Piperidinones are regarded as precursors of a host of biologically active compounds and natural alkaloids prior to their conversion to piperidines and also possess cytotoxic and anticancer properties (Dimmock *et al.*, 1990, 2001). In addition, precise X-ray crystallographic investigations of the molecular and crystal structures of symmetrically shaped molecules are expected to provide insights into the nature and strength of the competition between inter- and intramolecular forces and their role in effecting symmetry carry-over from the free state to the solid. The crystal structure of the title compound is a good example of a symmetrically shaped molecule loosing its molecular symmetry upon crystallization. In this context, we have already elucidated the crystal structures of cyano substituted (Suresh *et al.*, 2006) and nitroso substituted (Suresh *et al.*, 2005a; 2005b; Natarajan *et al.*, 2005) piperidinone derivatives. The present paper reports the crystal structure of 1-cyano-3,5-bis[(4-methylphenyl)methylidene]-piperidin-4-one.

The piperidinone ring adopts the envelope conformation. Atom N1 deviates by 0.655 (2) Å from the least-squares plane defined by atoms C2, C3, C4, C5 and C6. The envelope conformation is also evident from the puckering amplitudes [$Q = 0.492$ (1) Å, $\theta = 57.9$ (2)°, $\varphi = 349.8$ (2)°] (Cremer & Pople, 1975). As expected, the N—C α —N bond is linear. The 3- and 5- substituted 4-methylphenyl rings are twisted with respect to the plane defined by the piperidinone ring (excluding N1) and the methylidene C atoms by 15.7 (1)° and 35.7 (1)°, respectively. This unequal twists of the rings may be attributed to the fact that atoms C52 and C57 take part in intermolecular interactions (Table 1). Thus the present structure is a good example of a molecule where competition between intra- and intermolecular interactions is apparent.

The molecular aggregation in the crystal is characterized by H-bonded bilayered structures parallel to (−101) plane, with the molecules themselves aligned along the [1−11] direction. Figures 2 and 3 present two views (at 90° from one another) of these two-dimensional structures, where the internal link between layers can be appreciated. This is due to weak non conventional H-bonding (Table 1) as well as $\pi\cdots\pi$ interactions between symmetrically related 4-methylphenyl rings substituted at 3, [with and interplanar distance of 3.973 (1) Å] and at 5 [4.082 (1) Å] (See Fig. 3).

Interactions connecting bilayers are mainly van der Waal's

Experimental

A mixture of 1-methyl-3,5-bis[(*E*)-(4methylphenyl)methylidene]tetrahydro-4(1*H*)-pyridinone (1 g, 3 mmol), cyanogen bromide (0.33 g, 3 mmol) and potassium carbonate (3 mmol) in acetone (20 ml) was refluxed for 30 min. After completion of the reaction as seen from TLC (4:1 v/v petroleum ether:ethyl acetate), the mixture was poured into water (50 ml) and the precipitated 1-cyano-3,5-bis[(*E*)-(4methylphenyl)methylidene]tetrahydro-4(1*H*)- pyridinone was filtered, washed with water and recrystallized from ethanol.

Refinement

H atoms were positioned geometrically and refined using a riding model with C—H = 0.95–0.99 Å and with $U_{\text{iso}}(\text{H}) = 1.2$ (1.5 for methyl groups) times $U_{\text{eq}}(\text{C})$.

Figures

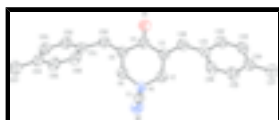


Fig. 1. The molecular structure of (I), with atom labels and 50% probability displacement ellipsoids for non-H atoms. H atoms have been omitted for clarity.

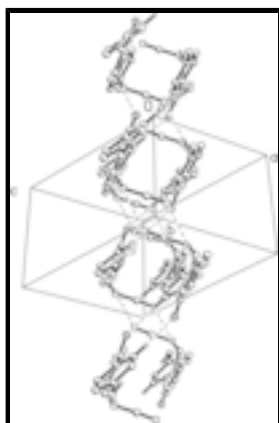


Fig. 2. View of the bilayer along [1-11], showing the two-dimensional structures sideways.

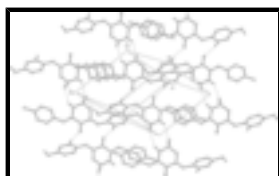


Fig. 3. View of the bilayer normal to (-101). H atoms not involved in hydrogen bonding omitted, for clarity.

3,5-Bis(4-methylbenzylidene)-4-oxopiperidine-1-carbonitrile

Crystal data

$\text{C}_{22}\text{H}_{20}\text{N}_2\text{O}$

$M_r = 328.40$

Triclinic, $P\bar{1}$

Hall symbol: -P 1

$a = 9.1976$ (2) Å

$b = 9.5012$ (2) Å

$c = 10.9418$ (3) Å

$\alpha = 104.03$ (2)°

$\beta = 95.080$ (1)°

$\gamma = 108.73$ (1)°

$V = 864.01$ (12) Å³

$Z = 2$

$F_{000} = 348$

$D_x = 1.262$ Mg m⁻³

Mo $K\alpha$ radiation

$\lambda = 0.71073$ Å

Cell parameters from 5233 reflections

$\theta = 3\text{--}29^\circ$

$\mu = 0.08$ mm⁻¹

$T = 298$ (2) K

Needle, colourless

$0.28 \times 0.14 \times 0.12$ mm

Data collection

Bruker Kappa-APEXII CCD diffractometer	5753 independent reflections
Radiation source: fine-focus sealed tube	3427 reflections with $I > 2\sigma(I)$
Monochromator: graphite	$R_{\text{int}} = 0.031$
$T = 298(2)$ K	$\theta_{\text{max}} = 31.5^\circ$
ω and φ scans	$\theta_{\text{min}} = 2.4^\circ$
Absorption correction: multi-scan (SADABS; Sheldrick, 2004)	$h = -13 \rightarrow 13$
$T_{\text{min}} = 0.88$, $T_{\text{max}} = 0.99$	$k = -13 \rightarrow 13$
23388 measured reflections	$l = -15 \rightarrow 16$

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)] = 0.050$	H-atom parameters constrained
$wR(F^2) = 0.150$	$w = 1/[\sigma^2(F_o^2) + (0.0677P)^2 + 0.0757P]$
$S = 1.04$	where $P = (F_o^2 + 2F_c^2)/3$
5753 reflections	$(\Delta/\sigma)_{\text{max}} = 0.012$
228 parameters	$\Delta\rho_{\text{max}} = 0.21 \text{ e } \text{\AA}^{-3}$
Primary atom site location: structure-invariant direct methods	$\Delta\rho_{\text{min}} = -0.20 \text{ e } \text{\AA}^{-3}$
	Extinction correction: none

Special details

Geometry. All e.s.d.'s (except the e.s.d. in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell e.s.d.'s are taken into account individually in the estimation of e.s.d.'s in distances, angles and torsion angles; correlations between e.s.d.'s in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell e.s.d.'s is used for estimating e.s.d.'s involving l.s. planes.

Refinement. Refinement of F^2 against ALL reflections. The weighted R -factor wR and goodness of fit S are based on F^2 , conventional R -factors R are based on F , with F set to zero for negative F^2 . The threshold expression of $F^2 > 2\sigma(F^2)$ is used only for calculating R -factors(gt) *etc.* and is not relevant to the choice of reflections for refinement. R -factors based on F^2 are statistically about twice as large as those based on F , and R -factors based on ALL data will be even larger.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (\AA^2)

	x	y	z	$U_{\text{iso}}^*/U_{\text{eq}}$
O1	0.48410 (14)	0.53238 (12)	0.31301 (10)	0.0690 (3)
N1	0.30550 (12)	0.11155 (12)	0.05535 (10)	0.0460 (3)
N2	0.07443 (16)	-0.00446 (16)	0.14716 (13)	0.0643 (3)
C1	0.18139 (16)	0.05283 (15)	0.10693 (12)	0.0460 (3)
C2	0.30506 (16)	0.23774 (15)	-0.00010 (13)	0.0461 (3)

supplementary materials

H2A	0.2005	0.2149	-0.0447	0.055*
H2B	0.3740	0.2431	-0.0624	0.055*
C3	0.35695 (14)	0.39263 (15)	0.10000 (12)	0.0426 (3)
C4	0.45051 (15)	0.41260 (15)	0.22607 (13)	0.0461 (3)
C5	0.50289 (14)	0.28455 (14)	0.24356 (12)	0.0430 (3)
C6	0.45772 (15)	0.14170 (16)	0.13125 (13)	0.0482 (3)
H6A	0.5365	0.1551	0.0774	0.058*
H6B	0.4534	0.0531	0.1620	0.058*
C30	0.32067 (15)	0.51409 (15)	0.08403 (13)	0.0465 (3)
H30	0.3595	0.6021	0.1542	0.056*
C31	0.23114 (15)	0.53174 (15)	-0.02403 (13)	0.0448 (3)
C32	0.17379 (16)	0.65369 (16)	-0.00096 (14)	0.0507 (3)
H32	0.1971	0.7214	0.0812	0.061*
C33	0.08403 (16)	0.67599 (16)	-0.09644 (14)	0.0519 (3)
H33	0.0478	0.7582	-0.0774	0.062*
C34	0.04622 (15)	0.57891 (16)	-0.22043 (14)	0.0482 (3)
C35	0.10540 (19)	0.45993 (17)	-0.24448 (14)	0.0563 (4)
H35	0.0831	0.3935	-0.3271	0.068*
C36	0.19605 (18)	0.43723 (17)	-0.14985 (14)	0.0543 (4)
H36	0.2349	0.3570	-0.1702	0.065*
C37	-0.05332 (19)	0.6022 (2)	-0.32468 (16)	0.0635 (4)
H37A	-0.1565	0.5257	-0.3424	0.095*
H37B	-0.0083	0.5917	-0.4007	0.095*
H37C	-0.0589	0.7040	-0.2976	0.095*
C50	0.58049 (14)	0.30007 (15)	0.35805 (13)	0.0476 (3)
H50	0.5946	0.3909	0.4215	0.057*
C51	0.64599 (14)	0.19288 (15)	0.39650 (13)	0.0452 (3)
C52	0.65606 (16)	0.18862 (16)	0.52345 (13)	0.0515 (3)
H52	0.6261	0.2574	0.5826	0.062*
C53	0.70973 (17)	0.08408 (17)	0.56235 (13)	0.0532 (3)
H53	0.7151	0.0835	0.6475	0.064*
C54	0.75601 (15)	-0.02031 (16)	0.47766 (13)	0.0501 (3)
C55	0.75351 (16)	-0.01031 (18)	0.35349 (14)	0.0548 (4)
H55	0.7890	-0.0755	0.2958	0.066*
C56	0.70000 (15)	0.09333 (17)	0.31296 (13)	0.0511 (3)
H56	0.6999	0.0970	0.2288	0.061*
C57	0.8060 (2)	-0.1408 (2)	0.51771 (16)	0.0676 (4)
H57A	0.7203	-0.2381	0.4921	0.101*
H57B	0.8379	-0.1088	0.6091	0.101*
H57C	0.8918	-0.1525	0.4776	0.101*

Atomic displacement parameters (\AA^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.0862 (8)	0.0512 (6)	0.0606 (7)	0.0354 (6)	-0.0140 (6)	-0.0056 (5)
N1	0.0521 (6)	0.0369 (6)	0.0455 (6)	0.0155 (5)	-0.0001 (5)	0.0090 (5)
N2	0.0627 (8)	0.0644 (8)	0.0548 (8)	0.0108 (6)	0.0026 (6)	0.0161 (6)
C1	0.0550 (7)	0.0362 (7)	0.0391 (7)	0.0143 (5)	-0.0048 (6)	0.0043 (5)

C2	0.0543 (7)	0.0398 (7)	0.0437 (7)	0.0176 (6)	0.0051 (5)	0.0113 (6)
C3	0.0436 (6)	0.0391 (6)	0.0451 (7)	0.0151 (5)	0.0075 (5)	0.0112 (5)
C4	0.0463 (6)	0.0396 (7)	0.0486 (7)	0.0168 (5)	0.0026 (5)	0.0057 (6)
C5	0.0404 (6)	0.0395 (7)	0.0468 (7)	0.0149 (5)	0.0046 (5)	0.0089 (5)
C6	0.0507 (7)	0.0445 (7)	0.0498 (8)	0.0229 (6)	0.0033 (6)	0.0080 (6)
C30	0.0497 (7)	0.0400 (7)	0.0497 (8)	0.0172 (5)	0.0085 (6)	0.0112 (6)
C31	0.0495 (7)	0.0384 (7)	0.0514 (8)	0.0181 (5)	0.0131 (6)	0.0170 (6)
C32	0.0580 (8)	0.0397 (7)	0.0537 (8)	0.0203 (6)	0.0075 (6)	0.0088 (6)
C33	0.0574 (8)	0.0410 (7)	0.0616 (9)	0.0249 (6)	0.0095 (6)	0.0127 (6)
C34	0.0488 (7)	0.0451 (7)	0.0546 (8)	0.0174 (6)	0.0112 (6)	0.0197 (6)
C35	0.0792 (10)	0.0518 (8)	0.0455 (8)	0.0326 (7)	0.0138 (7)	0.0137 (6)
C36	0.0766 (9)	0.0500 (8)	0.0514 (8)	0.0375 (7)	0.0198 (7)	0.0183 (7)
C37	0.0645 (9)	0.0658 (10)	0.0648 (10)	0.0282 (8)	0.0047 (7)	0.0224 (8)
C50	0.0462 (7)	0.0419 (7)	0.0495 (8)	0.0152 (5)	0.0021 (6)	0.0067 (6)
C51	0.0407 (6)	0.0435 (7)	0.0468 (7)	0.0131 (5)	0.0007 (5)	0.0098 (6)
C52	0.0565 (8)	0.0498 (8)	0.0416 (7)	0.0209 (6)	0.0005 (6)	0.0015 (6)
C53	0.0625 (8)	0.0554 (8)	0.0400 (7)	0.0227 (7)	0.0022 (6)	0.0103 (6)
C54	0.0480 (7)	0.0511 (8)	0.0481 (8)	0.0192 (6)	-0.0022 (6)	0.0103 (6)
C55	0.0574 (8)	0.0660 (9)	0.0459 (8)	0.0350 (7)	0.0058 (6)	0.0086 (7)
C56	0.0519 (7)	0.0656 (9)	0.0413 (7)	0.0279 (7)	0.0078 (5)	0.0159 (6)
C57	0.0794 (11)	0.0675 (10)	0.0622 (10)	0.0380 (9)	-0.0005 (8)	0.0181 (8)

Geometric parameters (Å, °)

O1—C4	1.2218 (16)	C34—C37	1.502 (2)
N1—C1	1.3381 (18)	C35—C36	1.373 (2)
N1—C6	1.4673 (16)	C35—H35	0.9300
N1—C2	1.4700 (16)	C36—H36	0.9300
N2—C1	1.1431 (18)	C37—H37A	0.9600
C2—C3	1.5060 (18)	C37—H37B	0.9600
C2—H2A	0.9700	C37—H37C	0.9600
C2—H2B	0.9700	C50—C51	1.4609 (18)
C3—C30	1.3448 (17)	C50—H50	0.9300
C3—C4	1.4957 (19)	C51—C56	1.3928 (18)
C4—C5	1.4906 (18)	C51—C52	1.3952 (19)
C5—C50	1.3360 (18)	C52—C53	1.3763 (19)
C5—C6	1.5046 (18)	C52—H52	0.9300
C6—H6A	0.9700	C53—C54	1.385 (2)
C6—H6B	0.9700	C53—H53	0.9300
C30—C31	1.4548 (19)	C54—C55	1.383 (2)
C30—H30	0.9300	C54—C57	1.503 (2)
C31—C36	1.393 (2)	C55—C56	1.3759 (19)
C31—C32	1.3997 (18)	C55—H55	0.9300
C32—C33	1.373 (2)	C56—H56	0.9300
C32—H32	0.9300	C57—H57A	0.9600
C33—C34	1.384 (2)	C57—H57B	0.9600
C33—H33	0.9300	C57—H57C	0.9600
C34—C35	1.3856 (19)		
C1—N1—C6	115.53 (11)	C36—C35—C34	121.90 (14)

supplementary materials

C1—N1—C2	116.42 (10)	C36—C35—H35	119.1
C6—N1—C2	112.59 (10)	C34—C35—H35	119.1
N2—C1—N1	176.60 (14)	C35—C36—C31	121.46 (12)
N1—C2—C3	112.30 (11)	C35—C36—H36	119.3
N1—C2—H2A	109.1	C31—C36—H36	119.3
C3—C2—H2A	109.1	C34—C37—H37A	109.5
N1—C2—H2B	109.1	C34—C37—H37B	109.5
C3—C2—H2B	109.1	H37A—C37—H37B	109.5
H2A—C2—H2B	107.9	C34—C37—H37C	109.5
C30—C3—C4	117.59 (12)	H37A—C37—H37C	109.5
C30—C3—C2	123.71 (12)	H37B—C37—H37C	109.5
C4—C3—C2	118.68 (11)	C5—C50—C51	128.41 (12)
O1—C4—C5	120.60 (12)	C5—C50—H50	115.8
O1—C4—C3	120.45 (12)	C51—C50—H50	115.8
C5—C4—C3	118.95 (11)	C56—C51—C52	117.42 (12)
C50—C5—C4	118.73 (12)	C56—C51—C50	123.55 (12)
C50—C5—C6	124.05 (12)	C52—C51—C50	119.03 (12)
C4—C5—C6	117.15 (11)	C53—C52—C51	120.95 (12)
N1—C6—C5	111.28 (10)	C53—C52—H52	119.5
N1—C6—H6A	109.4	C51—C52—H52	119.5
C5—C6—H6A	109.4	C52—C53—C54	121.54 (13)
N1—C6—H6B	109.4	C52—C53—H53	119.2
C5—C6—H6B	109.4	C54—C53—H53	119.2
H6A—C6—H6B	108.0	C55—C54—C53	117.29 (13)
C3—C30—C31	131.19 (13)	C55—C54—C57	120.90 (13)
C3—C30—H30	114.4	C53—C54—C57	121.80 (13)
C31—C30—H30	114.4	C56—C55—C54	121.84 (13)
C36—C31—C32	116.34 (13)	C56—C55—H55	119.1
C36—C31—C30	125.85 (12)	C54—C55—H55	119.1
C32—C31—C30	117.81 (12)	C55—C56—C51	120.79 (13)
C33—C32—C31	121.72 (13)	C55—C56—H56	119.6
C33—C32—H32	119.1	C51—C56—H56	119.6
C31—C32—H32	119.1	C54—C57—H57A	109.5
C32—C33—C34	121.50 (12)	C54—C57—H57B	109.5
C32—C33—H33	119.2	H57A—C57—H57B	109.5
C34—C33—H33	119.2	C54—C57—H57C	109.5
C33—C34—C35	117.04 (13)	H57A—C57—H57C	109.5
C33—C34—C37	121.56 (13)	H57B—C57—H57C	109.5
C35—C34—C37	121.39 (13)		
C1—N1—C2—C3	81.35 (14)	C31—C32—C33—C34	-0.2 (2)
C6—N1—C2—C3	-55.37 (14)	C32—C33—C34—C35	-1.2 (2)
N1—C2—C3—C30	-157.65 (12)	C32—C33—C34—C37	179.36 (13)
N1—C2—C3—C4	20.61 (16)	C33—C34—C35—C36	0.8 (2)
C30—C3—C4—O1	4.15 (19)	C37—C34—C35—C36	-179.76 (14)
C2—C3—C4—O1	-174.21 (12)	C34—C35—C36—C31	1.0 (2)
C30—C3—C4—C5	-175.46 (11)	C32—C31—C36—C35	-2.2 (2)
C2—C3—C4—C5	6.17 (18)	C30—C31—C36—C35	177.47 (13)
O1—C4—C5—C50	3.3 (2)	C4—C5—C50—C51	-178.33 (12)
C3—C4—C5—C50	-177.08 (11)	C6—C5—C50—C51	4.6 (2)

O1—C4—C5—C6	-179.45 (13)	C5—C50—C51—C56	29.9 (2)
C3—C4—C5—C6	0.16 (17)	C5—C50—C51—C52	-150.15 (14)
C1—N1—C6—C5	-75.18 (14)	C56—C51—C52—C53	-3.3 (2)
C2—N1—C6—C5	61.93 (14)	C50—C51—C52—C53	176.74 (12)
C50—C5—C6—N1	144.17 (13)	C51—C52—C53—C54	0.2 (2)
C4—C5—C6—N1	-32.92 (16)	C52—C53—C54—C55	3.1 (2)
C4—C3—C30—C31	-179.02 (12)	C52—C53—C54—C57	-176.43 (14)
C2—C3—C30—C31	-0.7 (2)	C53—C54—C55—C56	-3.2 (2)
C3—C30—C31—C36	-18.2 (2)	C57—C54—C55—C56	176.34 (14)
C3—C30—C31—C32	161.51 (13)	C54—C55—C56—C51	0.0 (2)
C36—C31—C32—C33	1.8 (2)	C52—C51—C56—C55	3.3 (2)
C30—C31—C32—C33	-177.90 (12)	C50—C51—C56—C55	-176.84 (13)

Hydrogen-bond geometry (Å, °)

<i>D</i> —H \cdots <i>A</i>	<i>D</i> —H	H \cdots <i>A</i>	<i>D</i> \cdots <i>A</i>	<i>D</i> —H \cdots <i>A</i>
C2—H2A \cdots N2 ⁱ	0.97	2.61	3.4630 (19)	147
C52—H52 \cdots O1 ⁱⁱ	0.93	2.59	3.4921 (17)	163
C57—H57B \cdots N2 ⁱⁱⁱ	0.96	2.57	3.524 (2)	174

Symmetry codes: (i) $-x, -y, -z$; (ii) $-x+1, -y+1, -z+1$; (iii) $-x+1, -y, -z+1$.

Fig. 1

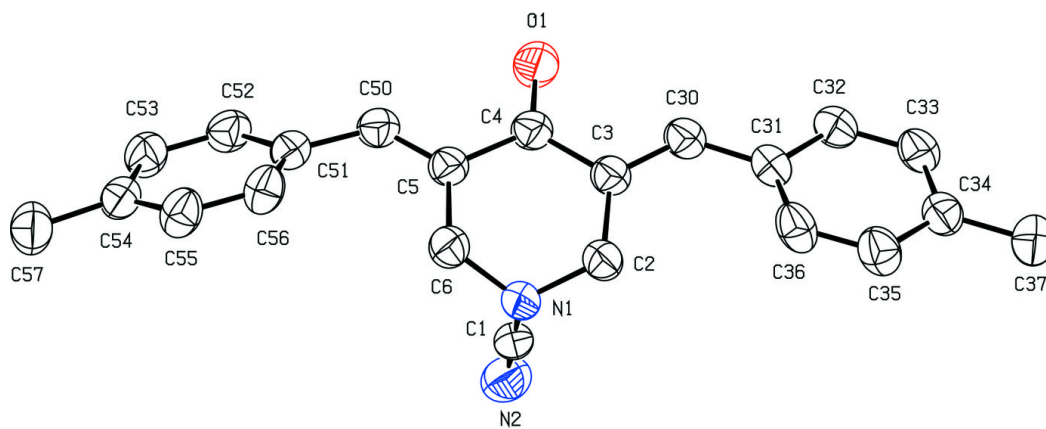


Fig. 2

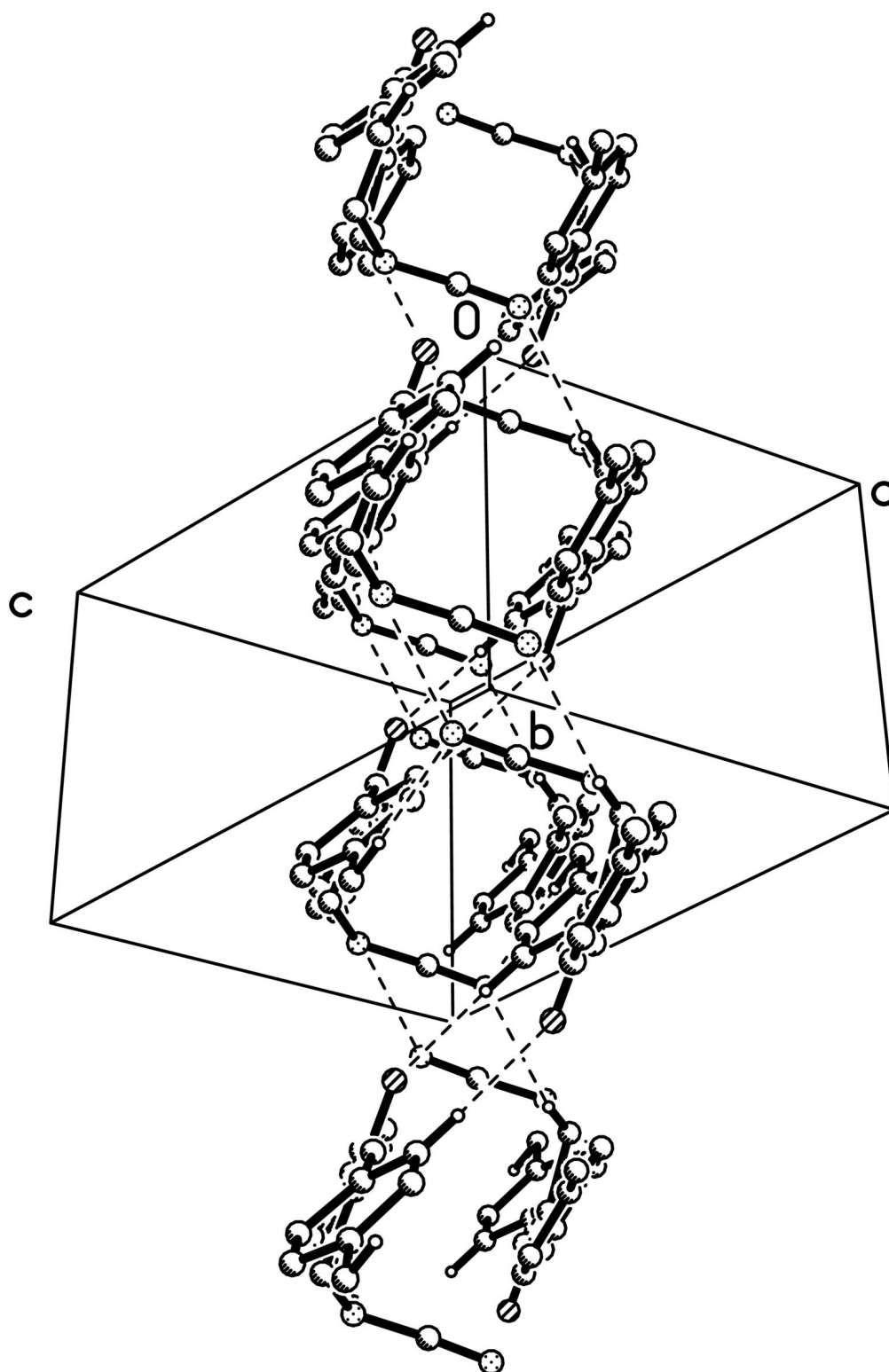


Fig. 3

