Table 2. *Hydrogen-bonding geometry (Å, °)*

D—H···*A*	H···*A*	*D*···*A*	*D*—H···*A*
N1*A*—H1*A*···O1*A*^{i}	2.319 (3)	2.898 (4)	124.88 (9)
N1*A*—H1*A*···O3*A*^{ii}	2.471 (4)	3.110 (4)	131.59 (9)
N1*B*—H1*B*···O1*B*^{iii}	2.340 (4)	2.918 (4)	124.8 (1)
N1*B*—H1*B*···O3*B*^{iv}	2.444 (4)	3.099 (4)	133.45 (9)

Symmetry codes: (i) $x - \frac{1}{2}, \frac{5}{2} - y, z$; (ii) $x, 1 + y, z$; (iii) $\frac{1}{2} + x, -\frac{1}{2} - y, z$; (iv) $x, y - 1, z$.

Extinction conditions indicated that the space group was *Pna*2$_1$ (No. 33) or *Pnam* (No. 62). Successful structure solution by direct methods and successive refinement confirmed the space group to be *Pna*2$_1$. Although two independent enantiomers (molecules *A* and *B*) are related by a local inversion center at $x = 0.86$, $y = 0.50$, $z = 0.33$, this crystal structure can not be described by the centrosymmetric space group (*Pnam*). The absolute structure was not determined [$\chi = 0.3\,(4)$; Flack, 1983]. H atoms were located at geometrically calculated positions and were included in the refinement using the riding model. Polar axis restraints were applied according to the method of Flack & Schwarzenbach (1988). Data collection was performed for the independent region of the reciprocal space. Because some reflections were in a blind region of the equipment, their equivalent reflections, *i.e.* $h\ k\ -l$, were measured instead.

Data collection: *MSC/AFC Diffractometer Control Software* (Molecular Structure Corporation, 1992*a*). Cell refinement: *MSC/AFC Diffractometer Control Software*. Data reduction: *TEXSAN* (Molecular Structure Corporation, 1992*b*). Program(s) used to solve structure: *SHELXS*86 (Sheldrick, 1990). Program(s) used to refine structure: *SHELXL*93 (Sheldrick, 1993). Molecular graphics: *TEXSAN* and *ORTEP*II (Johnson, 1976). Software used to prepare material for publication: *SHELXL*93.

Supplementary data for this paper are available from the IUCr electronic archives (Reference: DE1051). Services for accessing these data are described at the back of the journal.

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4-(1-Phenylpiperidin-4-ylidene)cyclohexanone

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Abstract

The title compound, $C_{17}H_{21}NO$, crystallizes with two independent molecules in the asymmetric unit. The conformation of the molecules is essentially the same. All saturated rings adopt a chair conformation, which is a prerequisite for efficient charge transfer.

Comment

End-functionalized oligo(cyclohexylidenes), which consist of chair-type cyclohexane rings connected *via* double bonds (Hoogesteger *et al.*, 1995), are versatile molecular building blocks for the preparation of supramolecular assemblies, such as non-covalent polymer-like systems (Hoogesteger, Jenneskens, Kooijman, Veldman & Spek, 1996), Langmuir–Blodgett mono- and multilayers (Hoogesteger *et al.*, 1997), and the ripening and growth of microcrystals deposited on silicon by spin-coating (ten Grotenhuis, van der Eerden, Hoogesteger & Jenneskens, 1996). Since oligo(cyclohexylidenes) are semi-rigid aliphatic bridges, possessing a σ–π–σ topology, it was envisaged that end-functionalization with an electron-donor and an electron-acceptor moiety would give opto-electric compounds, *i.e.* materials which give long-range charge separation by electron transfer from the donor to the acceptor upon photoexcitation (Paddon-Row, 1994, and references therein).

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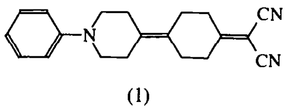
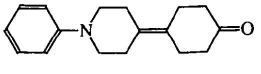
Recently, we have shown that 4-(1-phenylpiperidin-4-ylidene)cyclohexylidenepropanedinitrile, (1), possesses opto-electrical properties (Hoogesteger, 1996). Since the ground-state conformation of the compound is of interest in studies of the occurrence of photo-induced charge separation (Paddon-Row, 1994), we wished to determine the single-crystal X-ray structure of (1). Hitherto, no crystals of (1) have been obtained; however, suitable crystals of its precursor, 4-(1-phenylpiperidin-4-ylidene)cyclohexanone, (2), were obtained. We report here the low-temperature structure of the title compound, (2).



(1)



(2)

The crystal structure of (2) contains two independent molecules. The conformation of both molecules is essentially the same, although there are significant differences in the crystal environments of the molecules at longer range. The cyclohexane-type and piperidine-type rings have adopted slightly distorted chair conformations [all relevant asymmetry parameters (Duax & Norton, 1975) are less then 10°]. It is a prerequisite for the occurrence of efficient charge transfer that both rings adopt a chair-type conformation. The phenyl ring is located in an equatorial position of the piperidine-type ring and is eclipsed with one of the C—N bonds of this ring.

The relation between the independent molecules $A(-x, -y, -z)$ and B can be most easily visualized as a local rotation of 174.3° around an axis parallel to [1.00, −0.55, 0.06]. The r.m.s. fit for this match is 0.94 Å for superpositioned centres of gravity and 0.13 Å for a free fit. The rotation axis in the direct lattice does not coincide with one of the possible twofold rotation axes of the reciprocal lattice; the smallest angle is 18.9° with $(\bar{2}11)$. Rotation symmetry does not hold at longer distances. No other additional symmetry was detected (*MISSYM* option in *PLATON*; Spek, 1990*a*).

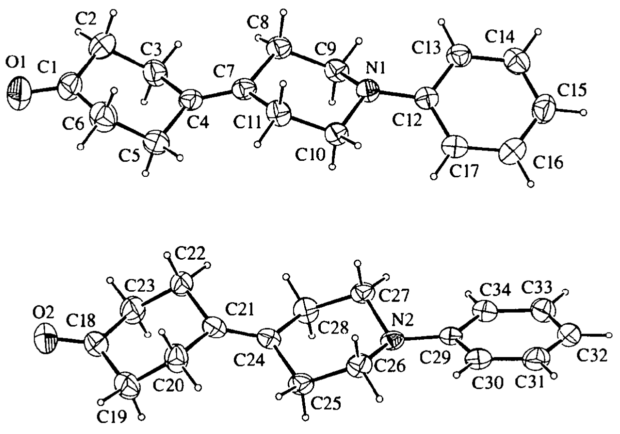


Fig. 1. A displacement ellipsoid plot (50% probability level) of both independent molecules of the title compound. H atoms are shown as spheres with arbitrary radii.

In the crystal, the molecules form layers perpendicular to the b axis, with antiparallel dipole moments. Intermolecular interactions include two short C—H···O contacts (Steiner, 1996), C3—H3A···O2(x, $\frac{1}{2}-y$, $z+\frac{1}{2}$) [C···O 3.440 (3) Å and C—H···O 154.9 (2)°] and C20—H20A···O1(x, $\frac{3}{2}-y$, $z-\frac{1}{2}$) [C···O 3.510 (3) Å and C—H···O 165.1 (3)°].

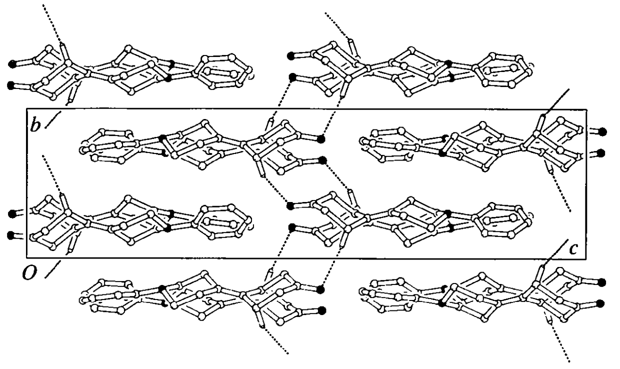


Fig. 2. Packing diagram viewed down the a axis.

Experimental

Details of the synthesis of the title compound are described elsewhere (Hoogesteger, 1996). Crystals were obtained by slow evaporation of a solution in cyclohexane.

Crystal data

$C_{17}H_{21}NO$
$M_r = 255.36$
Monoclinic
$P2_1/c$
$a = 12.3962\ (8)$ Å
$b = 7.8062\ (5)$ Å
$c = 30.305\ (2)$ Å
$\beta = 106.319\ (5)°$
$V = 2814.4\ (3)$ Å^3
$Z = 8$
$D_x = 1.205$ Mg m^{-3}
D_m not measured

Mo $K\alpha$ radiation
$\lambda = 0.71073$ Å
Cell parameters from 25 reflections
$\theta = 10.24$–$13.95°$
$\mu = 0.07$ mm^{-1}
$T = 150$ K
Plate-shaped
0.40 × 0.25 × 0.10 mm
Yellow

Data collection

Enraf–Nonius CAD-4T diffractometer on rotating anode
ω–2θ scans
Absorption correction: none
6561 measured reflections
5797 independent reflections
5797 reflections with $I \geq -3\sigma(I)$

$R_{\rm int} = 0.0378$
$\theta_{\rm max} = 26.45°$
$h = -15 \rightarrow 15$
$k = -9 \rightarrow 0$
$l = -37 \rightarrow 26$
3 standard reflections
frequency: 60 min
intensity decay: 2%

Refinement

Refinement on F^2
$R(F) = 0.063$
$wR(F^2) = 0.148$
$S = 0.960$
5797 reflections
343 parameters
H atoms: see below
$w = 1/[\sigma^2(F_o^2) + (0.0616P)^2]$
where $P = [\min(F_o^2,0) + 2F_c^2]/3$
$(\Delta/\sigma)_{\max} = 0.003$
$\Delta\rho_{\max} = 0.256$ e Å^{-3}
$\Delta\rho_{\min} = -0.287$ e Å^{-3}
Extinction correction: none
Scattering factors from *International Tables for Crystallography* (Vol. C)

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

O1—C1	1.215 (3)	O2—C18	1.213 (3)
N1—C9	1.465 (3)	N2—C26	1.459 (3)
N1—C10	1.460 (3)	N2—C27	1.462 (3)
N1—C12	1.413 (3)	N2—C29	1.413 (3)
C9—N1—C10	110.99 (17)	C26—N2—C27	111.29 (17)
C9—N1—C12	116.06 (18)	C26—N2—C29	117.62 (18)
C10—N1—C12	118.05 (19)	C27—N2—C29	117.95 (19)
C9—N1—C12—C13	50.7 (3)	C26—N2—C29—C30	5.5 (3)
C10—N1—C12—C17	2.3 (3)	C27—N2—C29—C34	−40.4 (3)

The unit-cell parameters were checked for the presence of higher lattice symmetry (Spek, 1988). Data were collected during 13 h of X-ray exposure time. Reflections were measured with a scan angle $\Delta\omega = (0.60 + 0.35\tan\theta)^\circ$; the horizontal and vertical apertures were 3.00 and 4.00 mm, respectively. The H atoms were included in the refinement on calculated positions, riding on their carrier atoms. The H atoms were refined with a fixed isotropic displacement parameter related to the equivalent isotropic displacement parameter of the carrier atom by a factor of 1.2. All non-H atoms were refined with anisotropic displacement parameters. The cited R value is calculated for 3078 reflections with $F > 4\sigma(F)$; wR is calculated for all observed reflections.

Data collection: locally modified *CAD*-4 *Software* (Enraf–Nonius, 1989). Cell refinement: *SET*4 (de Boer & Duisenberg, 1984). Data reduction: *HELENA* (Spek, 1990*b*). Program(s) used to solve structure: *SHELXS*86 (Sheldrick, 1985). Program(s) used to refine structure: *SHELXL*93 (Sheldrick, 1993). Molecular graphics: *PLUTON* (Spek, 1991). Software used to prepare material for publication: *PLATON* (Spek, 1990*a*).

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Supplementary data for this paper are available from the IUCr electronic archives (Reference: JZ1173). Services for accessing these data are described at the back of the journal.

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4-Ethyl-2-(2-hydroxynaphthylmethylidene)-amino-5-methyl-3-thiophenecarbonitrile

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Abstract

The crystal structure of the title compound, $C_{19}H_{16}N_2OS$, has been determined. The core of the molecule (excluding the methyl group) is planar, with the largest deviation of 0.069 (2) Å for the O atom. An intramolecular O—H···N-type hydrogen bond occurs between the hydroxyl and amino groups [2.574 (3) Å].

Comment

Schiff bases have been extensively used as ligands in the field of coordination chemistry (Calligaris, Nardin & Randaccio, 1972). Schiff base compounds can be classified by their photochromic and thermochromic characteristics (Cohen, Schmidt & Flavian, 1964; Moustakali, Mavridis & Hadjoudis, 1978; Hadjoudis, Vitterakis, Moustakali & Mavridis, 1987). The intramolecular hydrogen bond between O and N atoms plays an impor-

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