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1-Methyl-2-phenyldecahydroquinolin-4-one Oxime

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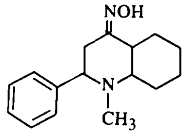
Abstract

The piperidine and cyclohexane rings in the title compound, $C_{16}H_{22}N_2O$, adopt a chair–chair conformation. The methyl group at N1 and the phenyl ring at C2 are oriented equatorially while the rest of the molecule is planar. The molecules are well stacked and the packing in the unit cell is stabilized by O—H···N hydrogen-bonded chains and weaker C—H···O and C—H···N bonds.

Comment

An *ORTEP*II (Johnson, 1976) diagram of the title compound, (I), with the atom-numbering scheme is shown in Fig. 1. The torsion angle H9—C9—C10—H10 [179 (2)°] confirms the *trans* fusion of the piperidine and cyclohexane rings in the molecule. It is evident from the ring torsion angles that the piperidine and cyclohexane rings are in a chair–chair conformation.



(I)

The angles C2—N1—C11 = 109.8 (2), C2—N1—C9 = 109.4 (1) and C9—N1—C11 = 110.7 (1)° indicate tetrahedral geometry at the N atom. The methyl group at N1 [C3—C2—N1—C11 = −173.9 (2)°] and the phenyl ring at C2 [C4—C3—C2—C14 = 178.7 (2)°] are equatorially oriented. The torsion angle C3—C4—N12—O13 [−2.1 (3)°] indicates that the oxime moiety has a planar conformation.

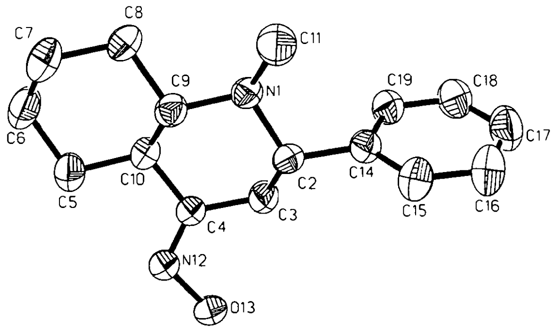


Fig. 1. *ORTEP*II (Johnson, 1976) diagram of the molecule with 50% probability ellipsoids.

From the stereoview of the packing (Fig. 2), it is clear that the phenyl ring of one molecule stacks well with the piperidine ring of the other molecule. It is found that there is an intermolecular O—H···N hydrogen bond (Table 3) which leads to a chain-like arrangement of the molecules (Fig. 2). In addition, an intramolecular C—H···O hydrogen bond and intermolecular C—H···O bonds are also found (Table 3).

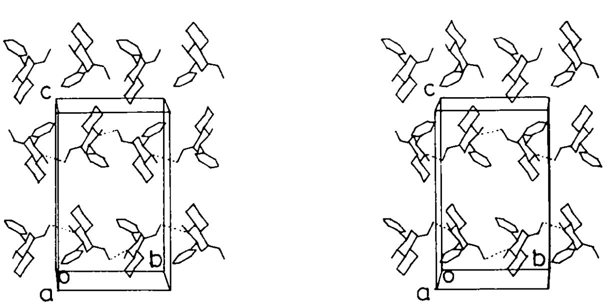


Fig. 2. Stereoview of the molecular packing (down the *a* axis) showing the chain-like arrangement of the molecules formed by intermolecular O—H···N hydrogen bonds.

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Experimental

The title compound was prepared according to the procedure of Baliah, Pandiarajan & Lakshmanan (1978). The solid oxime obtained was filtered and recrystallized from ethanol.

Crystal data

$C_{16}H_{22}N_2O$
$M_r = 258.36$
Monoclinic
$P2_1/n$
$a = 7.6940\ (1)$ Å
$b = 11.1350\ (1)$ Å
$c = 17.6949\ (1)$ Å
$\beta = 101.62\ (1)°$
$V = 1484.9\ (1)$ Å^3
$Z = 4$
$D_x = 1.16$ Mg m^{-3}
D_m not measured

Cu $K\alpha$ radiation
$\lambda = 1.54184$ Å
Cell parameters from 24 reflections
$\theta = 16–25°$
$\mu = 0.54$ mm^{-1}
$T = 298$ K
Needle
0.4 × 0.38 × 0.26 mm
Colourless

Data collection

Enraf–Nonius CAD-4 diffractometer
$\omega/2\theta$ scans
Absorption correction: empirical (*DIFABS*; Walker & Stuart, 1983)
$T_{\min} = 0.835$, $T_{\max} = 0.986$
2859 measured reflections
2431 independent reflections
2007 observed reflections
$[I > 2\sigma(I)]$

$R_{\rm int} = 0.016$
$\theta_{\max} = 65°$
$h = 0 \rightarrow 8$
$k = 0 \rightarrow 13$
$l = -20 \rightarrow 20$
3 standard reflections monitored every 200 reflections
frequency: 120 min
intensity decay: <1.5%

Refinement

Refinement on F
$R = 0.05$
$wR = 0.067$
$S = 0.95$
2007 reflections
260 parameters
All H-atom parameters refined
$w = 1/[\sigma^2(F) + 0.0078F^2]$

$(\Delta/\sigma)_{\max} = 0.017$
$\Delta\rho_{\max} = 0.289$ e Å^{-3}
$\Delta\rho_{\min} = -0.225$ e Å^{-3}
Extinction correction: none
Atomic scattering factors from *International Tables for X-ray Crystallography* (1974, Vol. IV)

Table 1. *Fractional atomic coordinates and equivalent isotropic displacement parameters* (Å^2)

$$B_{\rm eq} = (8\pi^2/3)\Sigma_i\Sigma_j U_{ij}a_i^*a_j^*\mathbf{a}_i.\mathbf{a}_j.$$

	x	y	z	$B_{\rm eq}$
N1	0.4946 (2)	0.6338 (1)	0.1695 (1)	3.03 (5)
C2	0.5584 (3)	0.7195 (2)	0.2337 (1)	3.38 (5)
C3	0.4073 (3)	0.7503 (2)	0.2740 (1)	4.06 (5)
C4	0.2528 (3)	0.8000 (2)	0.2177 (1)	3.31 (5)
C5	0.0572 (4)	0.7945 (2)	0.0828 (1)	4.76 (8)
C6	−0.0031 (4)	0.7138 (3)	0.0128 (2)	5.55 (8)
C7	0.1523 (4)	0.6725 (3)	−0.0204 (1)	5.28 (8)
C8	0.2948 (4)	0.6126 (2)	0.0401 (1)	4.30 (7)
C9	0.3569 (3)	0.6934 (2)	0.1100 (1)	3.36 (5)
C10	0.1966 (3)	0.7308 (2)	0.1436 (1)	3.38 (5)
C11	0.6451 (4)	0.5936 (3)	0.1358 (1)	4.99 (8)
N12	0.1631 (3)	0.8927 (2)	0.2282 (1)	3.69 (5)
O13	0.2219 (2)	0.9435 (1)	0.3019 (1)	4.60 (5)
C14	0.7153 (3)	0.6719 (2)	0.2924 (1)	3.50 (5)
C15	0.8695 (3)	0.7384 (3)	0.3093 (1)	4.48 (7)
C16	1.0118 (4)	0.7001 (3)	0.3645 (2)	5.63 (9)
C17	1.0028 (4)	0.5920 (3)	0.4027 (2)	5.74 (9)
C18	0.8482 (4)	0.5257 (2)	0.3859 (2)	5.24 (8)
C19	0.7047 (3)	0.5648 (2)	0.3320 (1)	4.17 (7)

Table 2. *Selected geometric parameters* (Å, °)

N1—C2	1.489 (2)	C7—C8	1.523 (3)
N1—C9	1.491 (2)	C8—C9	1.526 (3)
N1—C11	1.475 (4)	C9—C10	1.531 (3)
C2—C3	1.520 (3)	N12—O13	1.410 (2)
C2—C14	1.521 (3)	C14—C15	1.379 (3)
C3—C4	1.495 (3)	C14—C19	1.394 (3)
C4—C10	1.507 (3)	C15—C16	1.380 (4)
C4—N12	1.276 (3)	C16—C17	1.389 (5)
C5—C6	1.523 (4)	C17—C18	1.381 (4)
C5—C10	1.532 (3)	C18—C19	1.376 (4)
C6—C7	1.506 (5)		
C9—N1—C11	110.7 (1)	C8—C9—C10	109.7 (2)
C2—N1—C11	109.8 (2)	N1—C9—C10	111.1 (2)
C2—N1—C9	109.4 (1)	C5—C10—C9	111.0 (2)
N1—C2—C14	113.1 (2)	C4—C10—C9	111.4 (2)
N1—C2—C3	109.7 (2)	C4—C10—C5	114.3 (2)
C3—C2—C14	109.6 (2)	C4—N12—O13	112.2 (2)
C2—C3—C4	110.9 (2)	C2—C14—C19	121.5 (2)
C3—C4—N12	125.7 (2)	C2—C14—C15	119.4 (2)
C3—C4—C10	116.2 (2)	C15—C14—C19	119.0 (2)
C10—C4—N12	118.1 (2)	C14—C15—C16	120.9 (3)
C6—C5—C10	110.7 (2)	C15—C16—C17	120.1 (3)
C5—C6—C7	111.2 (3)	C16—C17—C18	118.9 (3)
C6—C7—C8	112.0 (2)	C17—C18—C19	121.1 (2)
C7—C8—C9	112.0 (2)	C14—C19—C18	119.9 (2)
N1—C9—C8	111.8 (2)		

Table 3. *Hydrogen-bonding geometry* (Å, °)

D—H···A	H···A	D···A	D—H···A
O13—H13···N1$^{\rm i}$	1.90 (3)	2.804 (2)	152 (3)
C3—H3B···O13	2.25 (3)	2.681 (3)	105 (2)
C16—H16···O13$^{\rm ii}$	2.74 (3)	3.451 (4)	126 (2)
C8—H8A···O13$^{\rm iii}$	2.64 (2)	3.395 (3)	130 (2)
C10—H10···O13$^{\rm iii}$	2.58 (2)	3.364 (3)	132 (2)
C19—H19···N12$^{\rm iii}$	2.40 (3)	3.406 (3)	169 (3)
C15—H15···N12$^{\rm ii}$	2.68 (3)	3.379 (4)	126 (2)

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

H atoms were located from difference Fourier maps and were refined isotropically.

Data collection: *Enraf–Nonius CAD*-4 *Software* (Enraf–Nonius, 1989). Cell refinement: *SDP* (Frenz, 1978). Data reduction: *SDP*. Program(s) used to solve structure: *SHELXS*86 (Sheldrick, 1990). Program(s) used to refine structure: *SHELX*76 (Sheldrick, 1976). Molecular graphics: *ORTEP*II (Johnson, 1976). Software used to prepare material for publication: *PARST* (Nardelli, 1983).

Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the IUCr (Reference: DE1035). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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An Optically Active Pentacoordinate Silicate: (+)-Bis(triethylammonium) Bis[(*R*,*R*)-tartrato(4−)]diphenyldisilicate

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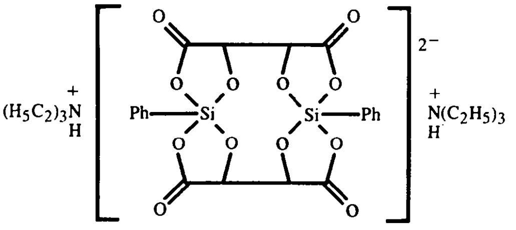
Abstract

The title compound, $2C_6H_{16}N^+.C_{20}H_{14}O_{12}Si_2^{2-}$, was prepared by the reaction of (*R*,*R*)-(+)-tartaric acid with triethoxyphenylsilane and triethylamine in ethanol. The structure of this optically active pentacoordinate phenylsilicate consists of independent bis[(*R*,*R*)-tartrato]diphenyldisilicate anions and triethylammonium cations. In the crystal, the two Si atoms are five-coordinate and the geometry of each of the Si atoms is distorted from a trigonal bipyramid towards a square pyramid along the Berry pseudorotational coordinate by *ca* 9.8 and 8.9%, respectively.

Comment

The study of hypervalent silicon compounds is currently one of the most interesting areas of research in organosilicon chemistry (Holmes, 1990; Chuit, Corriu, Reye & Young, 1993; Tacke, Becht, Lopez-Mras & Sperlich, 1993). In particular, the five-coordinate silicates have drawn much attention over the past few years, their preparation and structures having been investigated extensively (Frye, 1970; Tacke, Lopez-Mras & Jones, 1994; Holmes, Day, Harland, Sau & Holmes, 1984; Small, McCord, Greaces & Shea, 1995; Tamao *et al.*, 1995; Hosomi, Kohra, Ogata, Yauagi & Tominaga, 1990). Tacke, Mühleisen & Jones (1994) reported the preparation and structure of the first optically active pentacoordinate silicate. However, work on the crystal structures of optically active hypervalent organosilicon compounds is quite rare. We report here the crystal structure of a new optically active pentacoordinate silicate: (+)-bis(triethylammonium) bis[(*R*,*R*)-tartrato(4−)]-diphenyldisilicate, (I).



(I)

The disilicate dianion contains two pentacoordinate (formally negatively charged) Si atoms, each of which is coordinated by one phenyl group and four O atoms from two chiral tetradentate (*R*,*R*)-tartrato(4−) ligands which bridge the two Si atoms such that each ligand bonds to each of the Si atoms at the two coordination sites forming a group of four five-membered rings. Charge balance is provided by two triethylammonium cations. The geometry of the two five-coordinate Si atoms, Si(1) and Si(2), is distorted from an ideal trigonal bipyramid towards a square pyramid by *ca* 9.8 and 8.9%, respectively, as calculated by the dihedral-angle method (Holmes & Deiters, 1977). The axial Si—O bond lengths are Si(1)—O(1) 1.838 (4), Si(1)—O(3) 1.799 (4), Si(2)—O(5) 1.848 (4) and Si(2)—O(8) 1.801 (4) Å. The equatorial Si—O bond lengths are Si(1)—O(2) 1.671 (4), Si(1)—O(4) 1.668 (4), Si(2)—O(6) 1.671 (4) and Si(2)—O(7) 1.665 (4) Å.

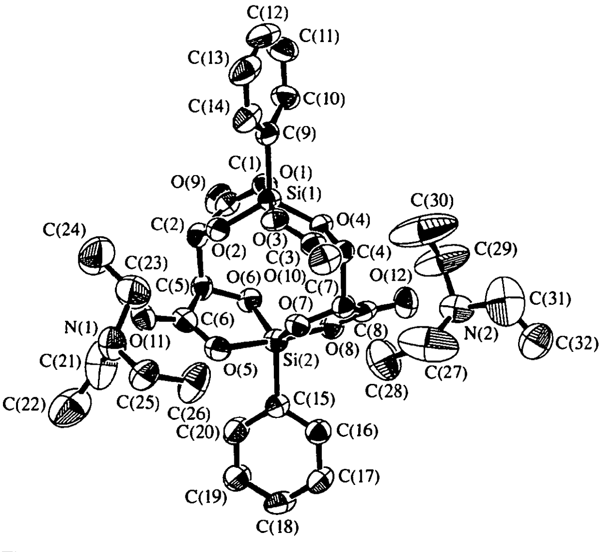


Fig. 1. *ORTEP* (Johnson, 1965) view of (I) showing the atom-labelling scheme. Displacement ellipsoids are drawn at the 50% probability level.

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