Data were collected at room temperature. The structure was solved by direct methods using *SHELXS*86 (Sheldrick, 1990). The refinement was performed using *SHELXL*93 (Sheldrick, 1993) using anisotropic displacement parameters for all non-H atoms and isotropic displacement parameters for the H atoms. The figures were drawn using *ZORTEP* (Zsolnai, 1995) and the tables were prepared using *SHELXL*93. All calculations were carried out on a VAXstation 4000VLC computer system.

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Supplementary data for this paper are available from the IUCr electronic archives (Reference: KH1145). Services for accessing these data are described at the back of the journal.

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3,3-Dimethyl-*N*-nitroso-2-phenyldecahydroquinolin-4-one

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Abstract

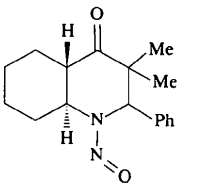
The molecule of the title compound, $C_{17}H_{22}N_2O_2$, consists of a piperidin-4-one ring *trans* fused to a cyclohexane moiety. The piperidine ring has a twist conformation. The ring N atom has a trigonal planar geometry. The cyclohexane ring adopts a chair conformation. The nitroso group is almost coplanar to the best plane of the piperidine ring. The phenyl ring is in the axial position.

Comment

Decahydroquinolines are important fused bicyclic systems containing an N atom. Their stereochemistry is of interest as they occur in many alkaloids such as lupinine, reserpine and yohimbine (Eliel, 1975; Norman, 1978; Nasipuri, 1992). Many nitrosamines are known to exhibit carcinogenic properties (Magee, Montesano & Preussmann, 1976; Ferguson, 1975; Loeppky, Tomasik & Kerrick, 1987). Certain *N*-nitrosoureas are used as antitumor agents or antibiotics (Lomax & Narayanan, 1988; Sapse, Allen & Lown, 1988).

The preferred conformation of the piperidine ring in decahydroquinoline precursors has been shown to be a chair with a slight twist or distortion of the ring dependent upon the position and size of the substituents (Booth & Bostock, 1972; Coz, Martin, Wartski, Penne, Bois & Levisalles, 1990). The substitution of a methyl or a nitroso group at the N-atom position has been shown to exert a large influence on the conformation of the ring and the orientation of the ring substituents (Vierhapper, 1980; Baliah & Natarajan, 1989).

The decahydroquinolines can have either *cis* or *trans* fusion of the two rings. The *trans* form is rigid. The title compound, (I), was found to exist in a *trans* configuration from analysis of the coupling constant of the H9 and H10 protons. From the X-ray crystal structure analysis, the torsion angle H9—C9—C10—H10 is found to be −177°, confirming *trans* fusion.



(I)

In piperidine derivatives, the substituents at nitrogen may be planar or perpendicular with respect to the ring (Lunazzi & Macciantelli, 1981; Lunazzi, Cerioni, Foresti & Macciantelli, 1978; Lunazzi, Cerioni & Ingold, 1976). The former is the case in the present compound. The ring N atom is planar and the N1—N11 bond length falls within the normal range for a partial double bond (Allen *et al.*, 1987).

The phenyl ring is substituted on the C2 atom in an axial position [C9—N1—C2—C21 107.9 (2)°]. The angles between the best planes of the phenyl, piperidine and cyclohexane rings are: phenyl/piperidine 82.6 (1), phenyl/cyclohexane 89.4 (1) and piperidine/cyclohexane 17.3 (2)°.

The dimensions of the cyclohexane ring in the title molecule, (I), are in agreement with those reported for *N*-benzoyl-2-phenyldecahyroquinolin-4-one (Thiruvalluvar, Sankar Raja Raj, Krishna Pillay & Venkatasubramanian, 1995) and *N*-acetyl-3-ethyl-2-phenyldecahydroquinolin-4-one (Thiruvalluvar, Parthasarathi, Natarajan, Bhavani & Bhadbhade, 1996). The conformation of the piperidine ring, however, varies with the substituents.

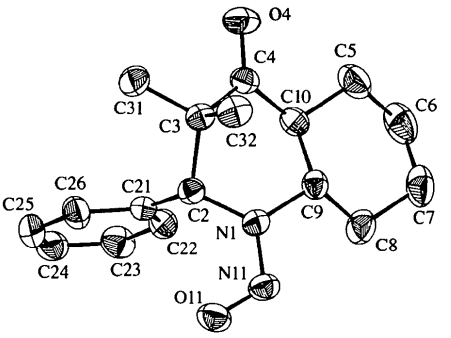


Fig. 1. The molecule structure of (I) showing 50% probability displacement ellipsoids. H atoms have been omitted for clarity.

Experimental

The title compound was prepared according to the procedure of Ravindran, Jeyaraman, Murray & Sing (1991) and recrystallized from ethanol.

Crystal data

$C_{17}H_{22}N_2O_2$
M_r = 286.37
Monoclinic
$P2_1/a$
a = 7.596 (2) Å
b = 22.882 (11) Å
c = 9.311 (2) Å
β = 110.18 (2)°
V = 1519.0 (9) Å^3
Z = 4
D_x = 1.252 Mg m^{-3}
D_m not measured
Cu $K\alpha$ radiation
λ = 1.54180 Å
Cell parameters from 25 reflections
θ = 16–22°
μ = 0.657 mm^{-1}
T = 298 K
Plate-like
0.31 × 0.25 × 0.08 mm
Pale yellow

Data collection

Enraf–Nonius CAD-4 diffractometer
ω–2θ scans
Absorption correction: none
2575 measured reflections
2563 independent reflections
2042 reflections with $I > 2\sigma(I)$
R_{int} = 0.065
θ_{max} = 65°
$h = -6 \rightarrow 8$
$k = -25 \rightarrow 26$
$l = -10 \rightarrow 10$
3 standard reflections
every 200 reflections
intensity decay: negligible

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.059
$wR(F^2)$ = 0.168
S = 1.076
2563 reflections
190 parameters
H atoms not refined
$w = 1/[\sigma^2(F_o^2) + (0.0933P)^2 + 0.4749P]$
where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = -0.006$
$\Delta\rho_{max}$ = 0.222 e Å^{-3}
$\Delta\rho_{min}$ = −0.242 e Å^{-3}
Extinction correction: none
Scattering factors from *International Tables for Crystallography* (Vol. C)

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

O4—C4	1.207 (3)	N1—C2	1.474 (3)
O11—N11	1.239 (2)	N1—C9	1.476 (3)
N1—C2—C21	110.7 (2)	N1—C9—C8	111.9 (2)
N1—C2—C3	110.3 (2)	N1—C9—C10	109.8 (2)
O4—C4—C10	121.6 (2)	O11—N11—N1	115.0 (2)
O4—C4—C3	120.9 (2)		

H atoms were located from difference Fourier maps using geometrical considerations and were included in structure-factor calculations. The parameters were not, however, refined.

Data collection: *SDP* (Frenz, 1978). Cell refinement: *SDP*. Data reduction: *SDP*. Program(s) used to solve structure: *SHELXS*86 (Sheldrick, 1985). Program(s) used to refine structure: *SHELXL*93 (Sheldrick, 1993). Molecular graphics: *ORTEP*II (Johnson, 1976). Software used to prepare material for publication: *SHELXL*93.

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Supplementary data for this paper are available from the IUCr electronic archives (Reference: VJ1058). Services for accessing these data are described at the back of the journal.

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(–)-(*R*)-5,5-Dimethylmorpholinyl-2-acetic Acid Ethyl Ester Hydrochloride,† $C_{10}H_{20}NO_3^+.Cl^-$

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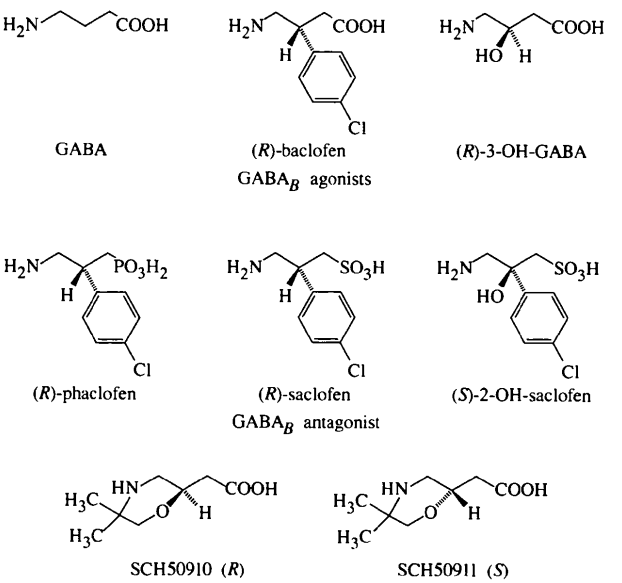
Abstract

One of the enantiomers of 5,5-dimethylmorpholinyl-2-acetic acid is observed to be a $GABA_B$ receptor antagonist. The absolute configuration of the inactive enantiomer is found to be the *R* configuration. The morpholine ring adopts a chair conformation with the acetic acid moiety in an equatorial position. In the crystal packing, hydrogen bonds are observed between the ammonium group and the chloride ions.

Comment

4-Aminobutyric acid (GABA) is the major inhibitory neurotransmitter in the mammalian central nervous system and operates through $GABA_A$ and $GABA_B$, and probably also $GABA_C$ receptors (Krujevic, 1974; Olsen & Venter, 1986; Barnard & Costa, 1989; Enna & Bowery, 1997). The present investigation was performed in order to obtain knowledge of the stereochemical requirements for the ligands to interact with the $GABA_B$ receptor recognition site. (*R*)-4-Amino-3-hydroxybutyric acid [(*R*)-3-OH-GABA] and (*R*)-baclofen are both $GABA_B$ receptor agonists (Falch *et al.*, 1986), showing that $GABA_B$ receptors are capable of accommodating the hydroxyl group of the first and the 4-chlorophenyl group of the second in different stereochemical orientations (see Scheme I). (*R*)-Phaclofen (Frydenvang *et al.*, 1994), (*R*)-saclofen (Carruthers *et al.*, 1995) and (*S*)-2-OH-saclofen (Prager, Schafer, Hamon & Massy-Westropp, 1995) are all $GABA_B$ receptor antagonists. The stereochemical orientations of the hydroxy groups of the $GABA_B$ receptor agonist, (*R*)-3-OH-GABA, and the $GABA_B$ receptor antagonist, (*S*)-2-OH-saclofen, are identical (see Scheme I). Recently, a series of morpholinyl-2-acetic acid analogues was synthesized and tested as $GABA_B$ receptor ligands, and one of these cyclized 3-oxygenated GABA analogues, namely, (+)-5,5-dimethylmorpholinyl-2-acetic acid (SCH50911), was shown to be a competitive $GABA_B$ receptor antagonist (Bolser *et al.*, 1995), while the (–)-enantiomer, SCH50910, was inactive. The absolute configuration of SCH50911 was established to be *S* by X-ray analysis of a derivative of SCH50911 containing bulky substituents (Blythin *et al.*, 1996) (see Scheme I). The stereochemical orientation of the oxygen function of SCH50911 is thus the opposite of the hydroxyl groups of (*R*)-3-OH-GABA and (*S*)-2-OH-saclofen.



Scheme I

† Alternative name: (*R*)-2-(ethoxycarbonylmethyl)-5,5-dimethylmorpholin-4-ium chloride.