

Table 1. Selected geometric parameters (Å, °)

F1—C4	1.319 (4)	N1—C3	1.357 (4)
F2—C4	1.335 (4)	N1—C24	1.428 (4)
F3—C4	1.332 (4)	N2—C6	1.266 (4)
O1—C1	1.364 (5)	N2—C10	1.483 (4)
O1—C21	1.365 (4)	C3—C5	1.340 (4)
O2—C6	1.357 (3)	C5—C6	1.441 (4)
O2—C7	1.443 (4)		
C6—O2—C7	105.2 (3)	N2—C6—O2	118.3 (3)
C3—N1—C24	128.3 (2)	N2—C6—C5	127.1 (3)
C5—C3—N1	123.5 (3)	C22—C21—O1	124.9 (4)
C3—C5—C6	122.3 (3)	C23—C24—C25	119.1 (3)
C24—N1—C3—C5	-176.2 (3)	C3—C5—C6—O2	-179.4 (3)
N1—C3—C5—C6	0.5 (5)	C1—O1—C21—C22	15.0 (6)
C7—O2—C6—C5	176.4 (3)	C3—N1—C24—C25	91.3 (4)

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

D—H...A	D—H	H...A	D...A	D—H...A
N1—H1...N2	0.86	2.05	2.728 (4)	135
C5—H5...F3	0.93	2.30	2.668 (4)	103

Data collection: *CAD-4 Software* (Enraf–Nonius, 1989). Cell refinement: *MolEN* (Fair, 1989). Data reduction: *PROCESS in MolEN*. Program(s) used to solve structure: *MULTAN11/82* (Main *et al.*, 1982). Program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997). Molecular graphics: *ORTEPII* (Johnson, 1976) in *MolEN*. Software used to prepare material for publication: *SHELXL93*. Computer facilities: University of Valencia.

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Supplementary data for this paper are available from the IUCr electronic archives (Reference: NA1290). Services for accessing these data are described at the back of the journal.

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## (±)-6,7-Dihydro-1,6,11-trimethyl-5H-dibenz[*c,e*]azepine

DRAGINJA MRVOŠ-SERMEK,<sup>a</sup> LINDA LONČAR-TOMAŠKOVIĆ,<sup>b</sup> ANTONIJA HERGOLD-BRUNDIĆ,<sup>a</sup> MLADEN MINTAS<sup>b</sup> AND ANTE NAGL<sup>c</sup>

<sup>a</sup>Laboratory of General and Inorganic Chemistry, Faculty of Science, University of Zagreb, Ul. kralja Zvonimira 8, 10 000 Zagreb, Croatia, <sup>b</sup>Department of Organic Chemistry, Faculty of Chemical Engineering and Technology, University of Zagreb, Marulićev trg 20, 10 000 Zagreb, Croatia, and <sup>c</sup>Faculty of Textile Technology, University of Zagreb, Pierottijeva 6, 10 000 Zagreb, Croatia. E-mail: mrvos@zagreb.zoak.pmf.hr

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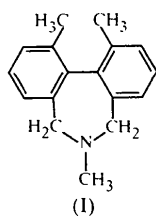
## Abstract

In the title compound, C<sub>17</sub>H<sub>19</sub>N, the central seven-membered azepine ring adopts a twisted-boat conformation. The molecule exhibits a huge deviation from planarity by rotation of the two fused *ortho*-methyl-substituted phenyl rings around the central C12—C14 single bond. The dihedral angle between the planes of these two rings amounts to 56.05 (4)°, with a *cisoid* arrangement around the C12—C14 bond. Because of this non-planarity, the molecule is axially chiral.

## Comment

Dihydrodibenzazepine derivatives have been shown to be potent hypolipidemic agents in rodents; treatment with 6,7-dihydro-5H-dibenz[*c,e*]azepine resulted in a reduction of the cholesterol, neutral lipid and triglyceride content in mouse and rat liver (Hall *et al.*, 1986). In addition, some of these compounds have been shown to be specific anti-epinephrine antagonists (Wenner, 1951). In the search for compounds related to such classes of

pharmacologically active compounds, we have prepared and structurally elucidated the title compound, (I).



The ground state of molecule (I) is non-planar, which gives rise to axial chirality. Both enantiomeric forms are present in the unit cell. Bond lengths and angles are generally as expected (Nyburg *et al.*, 1988; Kamenar *et al.*, 1989). The shape of the molecule (Fig. 1) is best described in terms of three planes: a seven-membered azepine ring, *A*, and two fused *ortho*-methyl-substituted phenyl rings, *B* and *C*. The angles between the least-squares best plane through atoms N, C5, C12 and C13 of the seven-membered ring, and rings *B* and *C* are 56.39 (7) and 46.11 (7)°, respectively. The dihedral angle between phenyl rings *B* and *C* is 56.05 (4)°, showing the expected *cisoid* arrangement around the C12—C14 bond (Bandarage *et al.*, 1995). As usual, the seven-membered ring adopts a twisted-boat conformation. The angles between the least-squares best plane through atoms N, C5, C12 and C13, and the planes through atoms N, C7 and C13, and atoms C12, C14, C15 and C5 are 49.18 (9) and 55.60 (8)°, respectively. The torsion angles C5—N—C7—C13 and C7—N—C5—C15 were found to be 44.9 (2) and 44.3 (2)°, respectively, indicating that the C—N—C—C bonds adopt the expected *gauche* arrangement (Desper *et al.*, 1993). There are no unusual intermolecular contacts; the crystal structure is stabilized entirely by van der Waals interactions.

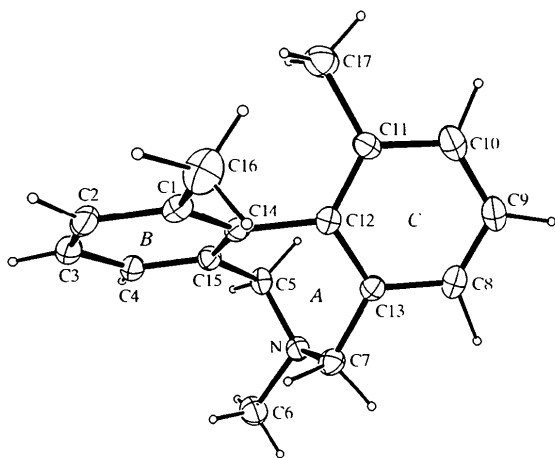


Fig. 1. ORTEP (Johnson, 1976) view of the title molecule with the atom-labelling scheme. Displacement ellipsoids are drawn at the 50% probability level.

## Experimental

The title compound (Mislow *et al.*, 1964; Fraser & Schuber, 1970) was prepared by the reaction of 2,2'-bis(bromomethyl)-6,6-biphenyl (Fritsch *et al.*, 1992) with methylamine using an analogous procedure to that given in the literature (Wenner, 1951). Diffraction quality crystals were grown by slow evaporation at room temperature of a very dilute solution in an ethyl acetate/ethanol (1:5) mixture.

### Crystal data

C <sub>17</sub> H <sub>19</sub> N	Mo K $\alpha$ radiation
$M_r = 237.33$	$\lambda = 0.71073 \text{ \AA}$
Monoclinic	Cell parameters from 26 reflections
$P2_1/c$	$\theta = 8.6\text{--}16.8^\circ$
$a = 9.168 (1) \text{ \AA}$	$\mu = 0.067 \text{ mm}^{-1}$
$b = 9.313 (3) \text{ \AA}$	$T = 295 (1) \text{ K}$
$c = 16.590 (9) \text{ \AA}$	Rhombohedral
$\beta = 105.88 (2)^\circ$	$0.60 \times 0.60 \times 0.20 \text{ mm}$
$V = 1362.4 (9) \text{ \AA}^3$	Colourless
$Z = 4$	
$D_x = 1.157 \text{ Mg m}^{-3}$	
$D_m = 1.150 (3) \text{ Mg m}^{-3}$	
$D_m$ measured by flotation in DMF/DMSO	

### Data collection

Philips PW1100 diffractometer updated by Stoe	$\theta_{\max} = 30.02^\circ$
$\omega/2\theta$ scans	$h = -12 \rightarrow 12$
Absorption correction: none	$k = 0 \rightarrow 13$
3952 measured reflections	$l = 0 \rightarrow 23$
3952 independent reflections	3 standard reflections
2556 reflections with $I > 2\sigma(I)$	frequency: 60 min
	intensity decay: 4.8%

### Refinement

Refinement on $F^2$	$(\Delta/\sigma)_{\max} = 0.013$
$R[F^2 > 2\sigma(F^2)] = 0.046$	$\Delta\rho_{\max} = 0.254 \text{ e \AA}^{-3}$
$wR(F^2) = 0.146$	$\Delta\rho_{\min} = -0.143 \text{ e \AA}^{-3}$
$S = 1.028$	Extinction correction: SHELXL93
3949 reflections	Extinction coefficient: 0.137 (8)
240 parameters	Scattering factors from International Tables for Crystallography (Vol. C)
All H atoms refined	
$w = 1/[\sigma^2(F_o^2) + (0.0716P)^2 + 0.1470P]$	
where $P = (F_o^2 + 2F_c^2)/3$	

Table 1. Selected geometric parameters ( $\text{\AA}$ ,  $^\circ$ )

N—C6	1.458 (2)	N—C7	1.478 (2)
N—C5	1.476 (2)		
C6—N—C5	109.89 (14)	C5—N—C7	111.73 (10)
C6—N—C7	111.62 (12)		

All non-H atoms in the structure were found by direct methods, whereas all H atoms were located in subsequent difference Fourier maps.

Data collection: DIF4 (Stoe & Cie, 1992a). Cell refinement: DIF4. Data reduction: REDU4 (Stoe & Cie, 1992b). Program(s) used to solve structure: SHELXS86 (Sheldrick, 1985). Program(s) used to refine structure: SHELXL93 (Sheldrick, 1993). Molecular graphics: ORTEP (Johnson, 1976). Software used to prepare material for publication: SHELXL93.

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Supplementary data for this paper are available from the IUCr electronic archives (Reference: SK1156). Services for accessing these data are described at the back of the journal.

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## Racemic Ethyl 4-(2-Fluorophenyl)-2,6,6-trimethyl-5-oxo-1,4,5,6,7,8-hexahydroquinoline-3-carboxylate

ANTHONY LINDEN,<sup>a</sup> CIHAT ŞAFAK<sup>b</sup> AND RAHIME ŞİMŞEK<sup>b</sup>

<sup>a</sup>*Institute of Organic Chemistry, University of Zürich, Winterthurerstrasse 190, CH-8057 Zürich, Switzerland,* and <sup>b</sup>*Department of Pharmaceutical Chemistry, Faculty of Pharmacy, Hacettepe University, 06100 Ankara, Turkey.*  
E-mail: alinden@oct.unizh.ch

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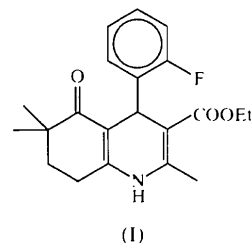
## Abstract

The title compound, C<sub>21</sub>H<sub>24</sub>FNO<sub>3</sub>, has potential calcium modulatory properties. The 1,4-dihydropyridine ring has a shallow boat conformation with the 2-fluorophenyl substituent in an axial synperiplanar orientation. The quinoline ring has a half-chair conformation. Steric

interactions between the adjacent methyl and ethoxy-carbonyl substituents cause local bond-angle distortions. The molecules are linked into chains by intermolecular N—H···O hydrogen bonds.

## Comment

Ca<sup>2+</sup> ions play a vital role in the maintenance of cardiac contractility. Calcium-channel modulators affect the passage of these ions through calcium channels and it is well established that these modulator drugs are important anti-anginal and antihypertensive agents (Janis & Triggle, 1983; Wehinger & Gross, 1986; Kendall & Luscombe, 1987; Nayler, 1988; Triggle, 1988). Those drugs that have a 1,4-dihydropyridine (1,4-DHP) moiety have been studied extensively and thoroughly reviewed (Bossert & Vater, 1989; Goldmann & Stoltefuss, 1991). Active compounds have been obtained by the introduction of the 1,4-DHP moiety to condensed systems such as acridine and quinoline. In these compounds, stereochemical differences affect their activities as an agonist and/or as an antagonist (Franckowiak *et al.*, 1985; Hof *et al.*, 1985; Rose & Dräger, 1992). Therefore, the determination of both the three-dimensional conformation of such systems and the absolute configuration of enantiomerically pure compounds, when available, is important. The structure of a racemic sample of the title compound, (I), as determined by X-ray diffraction, is reported here and corroborates the evidence obtained by IR, <sup>1</sup>H NMR, MS and elemental analyses. The calcium modulatory properties of this compound will be described in a later publication.



The 1,4-DHP ring in the structure of (I) has a shallow boat conformation, with atoms N1 and C4 0.152 (2) and 0.297 (2) Å, respectively, from the plane defined by atoms C2, C3, C4a and C8a. The maximum deviation of these latter four atoms from their mean plane is 0.0090 (6) Å for C8a. The shallowness of the boat is indicated by the puckering parameters (Cremer & Pople, 1975)  $Q = 0.263$  (1) Å,  $\theta = 77.3$  (2) and  $\varphi_2 = 184.0$  (3)°. For an ideal boat,  $\theta$  and  $\varphi_2$  are 90 and  $n \times 60$ °, respectively. The 2-fluorophenyl ring occupies an axial position and thereby lies above the 1,4-DHP boat. The plane of the 2-fluorophenyl ring is almost parallel to the N1···C4 axis [the N1···C4—C13—C18 torsion angle is 7.57 (15)°], which is sterically the most favourable