C8	0.7145 (4)	−0.0678 (4)	−0.1208 (4)	0.056 (4)
C9	0.7439 (4)	−0.0106 (4)	−0.0240 (4)	0.058 (4)
C10	0.6550 (4)	0.0710 (4)	−0.0039 (4)	0.061 (4)
C11	0.6267 (5)	0.1995 (5)	0.1327 (5)	0.091 (5)
C12	0.7073 (7)	0.2541 (6)	0.2428 (6)	0.129 (5)
C13	0.7044 (4)	−0.3176 (4)	−0.0803 (4)	0.064 (4)
C14	0.6330 (4)	−0.4242 (4)	−0.0997 (4)	0.069 (4)
C15	0.5776 (5)	−0.5110 (5)	−0.1133 (5)	0.086 (5)
C16	0.8062 (5)	−0.0955 (5)	−0.2674 (5)	0.089 (5)
O17	0.7045 (3)	0.1180 (3)	0.1014 (3)	0.067 (3)
O18	0.5526 (3)	0.0940 (3)	−0.0721 (3)	0.077 (3)
O19	0.6013 (3)	−0.2392 (3)	−0.2716 (3)	0.065 (3)

Table 2. *Bond lengths* (Å)

C2—C1	1.570 (6)	C7—C6	1.502 (7)
C7—C1	1.538 (6)	C9—C8	1.305 (6)
C13—C1	1.535 (6)	C10—C9	1.478 (6)
O19—C1	1.442 (5)	O17—C10	1.337 (5)
C3—C2	1.545 (6)	O18—C10	1.201 (5)
C8—C2	1.513 (6)	C12—C11	1.483 (8)
C16—C2	1.554 (6)	O17—C11	1.448 (5)
C4—C3	1.531 (7)	C14—C13	1.455 (6)
C5—C4	1.470 (8)	C15—C14	1.171 (6)
C6—C5	1.473 (7)		

Table 3. *Hydrogen-bond parameters* (Å, °)

D—H···O	*D*—H	H···O	*D*···O	H_{norm}···O	*D*—H_{norm}···O
O19—H···O18^{i}	0.79 (4)	2.12 (4)	2.852 (5)	1.96	151
C15—H···O19^{i}	0.95 (4)	2.47 (4)	3.338 (7)	2.32	157

Symmetry code: (i) $1-x, y-\frac{1}{2}, -\frac{1}{2}-z$.

As the crystal was very small, only diffraction data of moderate quality could be obtained. H atoms were refined isotropically, except for those bonded to C4, C5 and C6. These did not refine realistically and were included in the model in their ideal positions (Sheldrick, 1976).

Data collection: *CAD*-4 *Software* (Enraf–Nonius, 1989). Cell refinement: *CAD*-4 *Software*. Data reduction: *CAD*-4 *Software*. Program(s) used to solve structure: *SHELXS*86 (Sheldrick, 1985). 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, bond distances involving H atoms and bond angles involving non-H atoms have been deposited with the IUCr (Reference: DE1006). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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3,3-(Oxydiethyl)-1,2-diphenylguanidine

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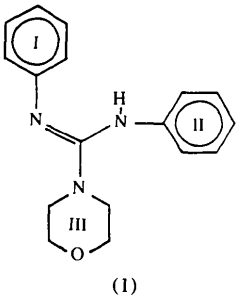
Abstract

The title molecule, 2-morpholino-1,3-diphenyl-1,3-diazapropene, $C_{17}H_{19}N_3O$, contains two phenyl rings joined to a guanidine skeleton that incorporates a morpholine ring. The morpholine ring adopts a chair conformation. All substituent rings are tilted out of the guanidine plane; the aromatic rings are almost perpendicular to each other. Crystal cohesion is due to van der Waals interactions and an intermolecular N—H···O hydrogen bond.

Comment

The title compound, (1), which was synthesized at the SPIC Science Foundation, Madras, belongs to a class of substances known for their pharmacological activity. Several cyclic guanidines are used to treat high blood pressure, and some are potent antidiabetic agents (Maryanoff, 1985). The molecular geometry and the atomic numbering scheme are shown in Fig. 1.



(1)

The morpholine ring is in a chair conformation (Fig. 1). The pyramidalization of the N atom in a morpholine ring varies through a wide range, with the sum of angles at N reported to be between 337 and 359° (Wong-Ng, Nyburg, Awwal, Jankie & Kresge, 1982). In the present case, the sum of the angles at

N10 is 351.8 (9)°. All substituent rings are tilted out of the guanidine plane and the dihedral angles relative to the guanidine moiety are 118.0 (3), 68.1 (3) and 54.8 (3)° for the rings I, II and III, respectively. The two aromatic rings are almost perpendicular to each other [the dihedral angle between rings I and II is 103.3 (3)°].

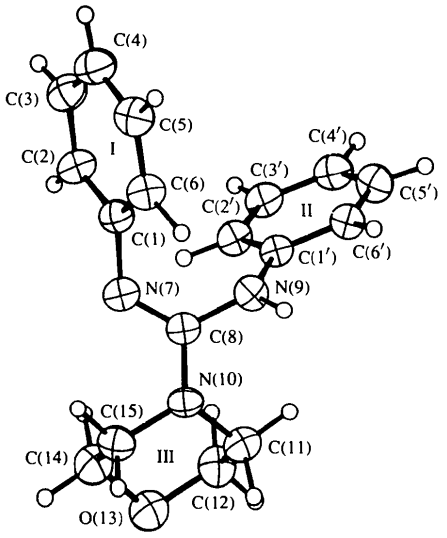


Fig. 1. Molecular structure and atomic numbering scheme; projection is on the guanidine plane. Displacement ellipsoids are plotted at the 30% probability level.

The intermolecular cohesive forces are dominated by van der Waals interactions, with one additional intermolecular N—H···O hydrogen bond from N(9)—H to O(13)(x, 3/2 − y, z + 1/2): N···O = 2.938 (8), N—H = 0.91 (7), H···O = 2.07 Å, N—H···O = 167 (6)° [for the normalized H-atom position based on an ideal N—H bond length of 1.04 Å, H···O = 1.95 Å and N—H···O = 166°].

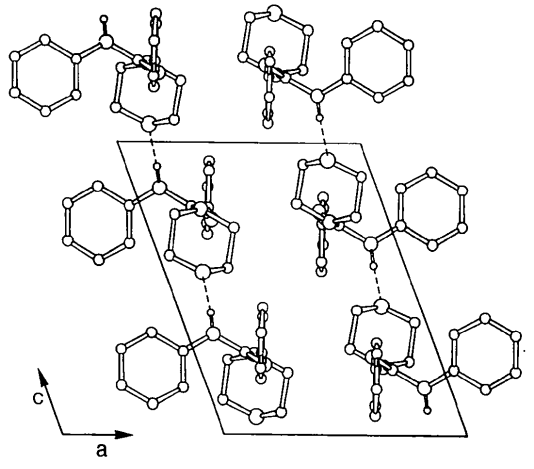


Fig. 2. Crystal-packing and hydrogen-bonding scheme. For clarity, H atoms are omitted except for H(N9) which is involved in hydrogen bonding.

Experimental

The compound was synthesized and supplied by SPIC Science Foundation, Madras. D_m was measured by flotation in KI + water.

Crystal data

$C_{17}H_{19}N_3O$	Cu $K\alpha$ radiation
M_r = 281.4	λ = 1.5418 Å
Monoclinic	Cell parameters from 22 reflections
$P2_1/c$	θ = 20–34°
a = 9.211 (4) Å	μ = 0.62 mm^{-1}
b = 14.671 (4) Å	T = 294 K
c = 11.533 (4) Å	Plate
β = 111.14 (4)°	0.4 × 0.25 × 0.1 mm
V = 1453.6 (10) Å^3	Colourless
Z = 4	
D_x = 1.29 Mg m^{-3}	
D_m = 1.30 Mg m^{-3}	

Data collection

Enraf–Nonius CAD-4 diffractometer	R_{int} = 0.019
ω–θ scans	θ_{max} = 60°
Absorption correction: none	h = −10 → 10
2412 measured reflections	k = 0 → 16
1574 independent reflections	l = 0 → 12
1229 observed reflections [$F > 3\sigma(F)$]	3 standard reflections frequency: 60 min intensity decay: 2.5%

Refinement

Refinement on F	$(\Delta/\sigma)_{max}$ = 0.002
R = 0.065	$\Delta\rho_{max}$ = 0.19 e Å^{-3}
wR = 0.065	$\Delta\rho_{min}$ = −0.18 e Å^{-3}
S = 1.26	Extinction correction: none
1229 reflections	Atomic scattering factors from *SHELX*76 (Sheldrick, 1976)
248 parameters	
Unit weights applied	

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

$$U_{eq} = (1/3)\Sigma_i\Sigma_j U_{ij}a_i^*a_j^*\mathbf{a}_i.\mathbf{a}_j.$$

	x	y	z	U_{eq}
C(1)	0.2767 (7)	0.4403 (5)	0.2756 (6)	0.059 (7)
C(2)	0.2342 (8)	0.3721 (4)	0.1871 (7)	0.069 (8)
C(3)	0.2491 (9)	0.2827 (5)	0.2237 (8)	0.078 (9)
C(4)	0.3058 (9)	0.2582 (6)	0.3466 (9)	0.081 (9)
C(5)	0.3505 (8)	0.3263 (6)	0.4345 (7)	0.080 (9)
C(6)	0.3362 (8)	0.4175 (5)	0.4020 (7)	0.068 (8)
C(1′)	−0.0433 (7)	0.5433 (4)	0.2797 (5)	0.051 (6)
C(2′)	−0.0986 (8)	0.5186 (4)	0.1548 (6)	0.058 (7)
C(3′)	−0.2493 (8)	0.4903 (5)	0.0976 (7)	0.061 (7)
C(4′)	−0.3490 (8)	0.4832 (5)	0.1603 (7)	0.065 (8)
C(5′)	−0.2941 (8)	0.5064 (5)	0.2849 (7)	0.072 (8)
C(6′)	−0.1447 (7)	0.5356 (5)	0.3448 (6)	0.058 (7)
N(7)	0.2729 (6)	0.5321 (4)	0.2355 (5)	0.065 (6)
C(8)	0.2091 (7)	0.5949 (4)	0.2795 (6)	0.055 (7)
N(9)	0.1062 (6)	0.5778 (4)	0.3416 (5)	0.059 (6)
N(10)	0.2438 (6)	0.6840 (3)	0.2699 (4)	0.058 (6)
C(11)	0.1284 (8)	0.7569 (4)	0.2516 (6)	0.066 (7)
C(12)	0.0461 (9)	0.7778 (5)	0.1146 (6)	0.073 (8)
O(13)	0.1585 (5)	0.8011 (3)	0.0587 (4)	0.073 (5)
C(14)	0.2676 (8)	0.7284 (5)	0.0744 (6)	0.073 (8)
C(15)	0.3534 (8)	0.7071 (5)	0.2091 (6)	0.065 (8)

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

C(1)—C(2)	1.381 (9)	C(4′)—C(5′)	1.383 (11)
C(1)—C(6)	1.401 (10)	C(5′)—C(6′)	1.367 (9)
C(1)—N(7)	1.421 (9)	N(7)—C(8)	1.291 (9)
C(2)—C(3)	1.370 (10)	C(8)—N(9)	1.402 (10)
C(3)—C(4)	1.370 (13)	C(8)—N(10)	1.359 (8)
C(4)—C(5)	1.377 (12)	N(10)—C(11)	1.469 (8)
C(5)—C(6)	1.384 (11)	N(10)—C(15)	1.462 (10)
C(1′)—C(2′)	1.391 (8)	C(11)—C(12)	1.517 (9)
C(1′)—C(6′)	1.398 (11)	C(12)—O(13)	1.444 (10)
C(1′)—N(9)	1.398 (7)	O(13)—C(14)	1.432 (8)
C(2′)—C(3′)	1.370 (9)	C(14)—C(15)	1.500 (9)
C(3′)—C(4′)	1.362 (12)		
C(6)—C(1)—N(7)	121.2 (6)	C(1′)—C(6′)—C(5′)	120.0 (6)
C(2)—C(1)—N(7)	118.6 (6)	C(1)—N(7)—C(8)	120.7 (6)
C(2)—C(1)—C(6)	119.9 (6)	N(7)—C(8)—N(10)	120.1 (6)
C(1)—C(2)—C(3)	119.7 (7)	N(7)—C(8)—N(9)	124.1 (6)
C(2)—C(3)—C(4)	122.0 (8)	N(9)—C(8)—N(10)	115.9 (6)
C(3)—C(4)—C(5)	118.2 (8)	C(1′)—N(9)—C(8)	121.8 (5)
C(4)—C(5)—C(6)	122.0 (7)	C(8)—N(10)—C(15)	119.1 (6)
C(1)—C(6)—C(5)	118.4 (7)	C(8)—N(10)—C(11)	122.2 (6)
C(6′)—C(1′)—N(9)	118.7 (5)	C(11)—N(10)—C(15)	110.5 (5)
C(2′)—C(1′)—N(9)	123.3 (6)	N(10)—C(11)—C(12)	111.2 (6)
C(2′)—C(1′)—C(6′)	118.0 (6)	C(11)—C(12)—O(13)	110.1 (6)
C(1′)—C(2′)—C(3′)	120.4 (6)	C(12)—O(13)—C(14)	110.6 (5)
C(2′)—C(3′)—C(4′)	121.8 (7)	O(13)—C(14)—C(15)	111.7 (6)
C(3′)—C(4′)—C(5′)	118.0 (7)	N(10)—C(15)—C(14)	110.4 (6)
C(4′)—C(5′)—C(6′)	121.7 (7)		

C(2)—C(1)—N(7)—C(8)	133.0 (7)
C(6)—C(1)—N(7)—C(8)	−53.1 (9)
C(1)—N(7)—C(8)—N(9)	−17.2 (10)
C(1)—N(7)—C(8)—N(10)	161.0 (6)
N(7)—C(8)—N(9)—C(1′)	−69.2 (9)
N(10)—C(8)—N(9)—C(1′)	112.6 (7)
C(8)—N(9)—C(1′)—C(6′)	−174.3 (6)
C(8)—N(9)—C(1′)—C(2′)	3.8 (9)
N(7)—C(8)—N(10)—C(11)	145.6 (6)
N(7)—C(8)—N(10)—C(15)	0.2 (9)
N(9)—C(8)—N(10)—C(11)	−36.0 (8)
N(9)—C(8)—N(10)—C(15)	178.5 (6)

H atoms were refined isotropically, except for those bonded to C2, C5, C11 and C14. These did not refine realistically and were included in the model in their ideal positions (Sheldrick, 1976). The *R* factor is relatively high due to the quality of data collected.

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: *SHELX*76 (Sheldrick, 1976). Molecular graphics: *ORTEP*II (Johnson, 1976). Software used to prepare material for publication: *PARST* (Nardelli, 1983).

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Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the IUCr (Reference: KA1123). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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Chiral *N*-(6-Amino-3-pyridyl)-*N′*-bicycloalkyl-*N″*-cyanoguanidine Derivative: a Novel Potassium-Channel Opener

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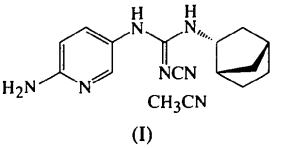
Abstract

The crystal structure of (+)-1-(6-amino-3-pyridyl)-3-[(1*S**,2*R**,4*R**)-bicyclo[2.2.1]hept-2-yl]-2-cyanoguanidine (AL0670) acetonitrile solvate, $C_{14}H_{18}N_6.CH_3CN$, has been determined by X-ray diffraction.

Comment

The title compound, AL0670 acetonitrile solvate, (I), was synthesized and selected as a potent antihypertensive agent. It has a different pharmacological profile from pinacidil, although both are regarded as potassium-channel openers. The synthesis of the compound and the absolute configuration of its hydrochloride have been reported previously (Eda *et al.*, 1994).



(I)

AL0670 has more than four polymorphic forms. In this paper, the crystal structure of AL0670 acetonitrile solvate, which is one of the polymorphic forms, is reported. The molecular structure is shown in Fig. 1. The N2—C6 bond has a *cis* configuration with respect to the aminopyridyl group and the bicycloalkyl group, while it has the *trans* configuration in the case of AL0670 hydrochloride (Eda *et al.*, 1994).