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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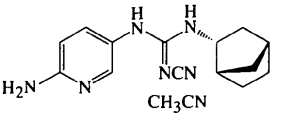
Abstract

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).

The crystal structure is shown in Fig. 2. The hydrogen bond N5···HN6—N6 connects symmetry-related AL0670 molecules into a ribbon along the *a* axis, while N1···HN2—N2 does so along the *b* axis. Acetonitrile is bound to AL0670 *via* an N7···HN3—N3 hydrogen bond, such that the molecular axis of acetonitrile is almost parallel to the *a* axis.

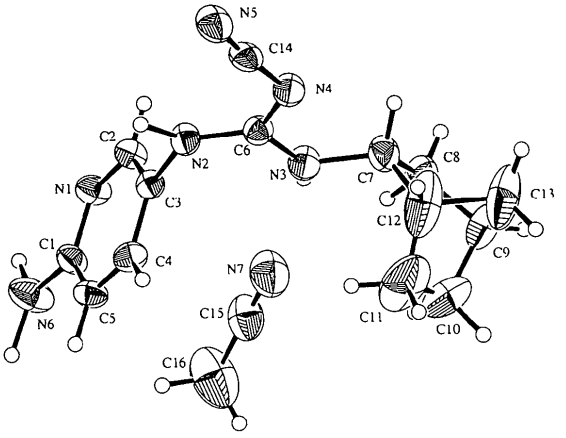


Fig. 1. A perspective view of the molecules (30% probability ellipsoids) with the atomic numbering scheme.

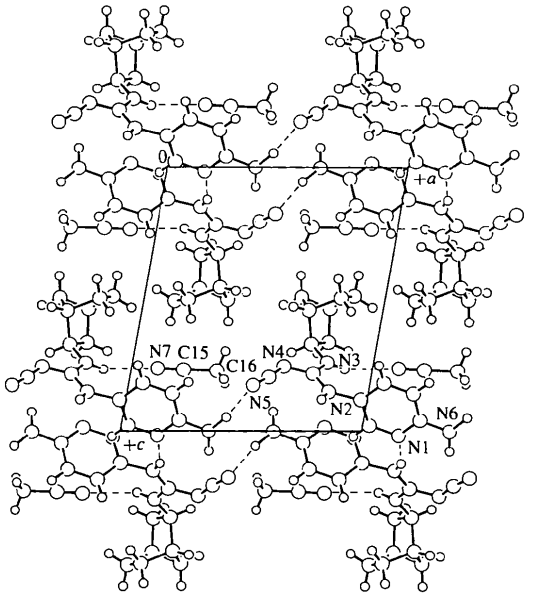


Fig. 2. The crystal structure viewed along the *b* axis. Hydrogen bonds are represented as dashed lines.

Experimental

The title compound was synthesized according to the method of Eda *et al.* (1994), and recrystallized from acetonitrile.

Crystal data

$C_{14}H_{18}N_6.CH_3CN$	Cu $K\alpha$ radiation
M_r = 311.39	λ = 1.5418 Å
Monoclinic	Cell parameters from 25 reflections
$P2_1$	θ = 28.85–29.85°
a = 10.427 (1) Å	μ = 0.610 mm^{-1}
b = 7.197 (1) Å	T = 296 K
c = 11.805 (1) Å	Prismatic
β = 99.755 (8)°	0.35 × 0.30 × 0.10 mm
V = 873.0 (2) Å^3	Colorless
Z = 2	
D_x = 1.184 Mg m^{-3}	

Data collection

Rigaku AFC-7*R* diffractometer	1195 observed reflections [$I > 3\sigma(I)$]
ω–2θ scans [width (1.68 + 0.30tanθ)°; speed 16° min^{-1} in ω]	R_{int} = 0.012
	θ_{max} = 60.05°
Absorption correction: ψ scan	$h = -11 \rightarrow 11$
T_{min} = 0.844, T_{max} = 1.000	$k = -8 \rightarrow 0$
1494 measured reflections	$l = 0 \rightarrow 13$
1419 independent reflections	3 standard reflections monitored every 150 reflections
	intensity decay: none

Refinement

Refinement on F	$\Delta\rho_{max}$ = 0.17 e Å^{-3}
R = 0.045	$\Delta\rho_{min}$ = −0.17 e Å^{-3}
wR = 0.071	Extinction correction: secondary
S = 1.45	Extinction coefficient: 2.61694 × 10^{-5}
1195 reflections	Atomic scattering factors from *International Tables for X-ray Crystallography* (1974, Vol. IV)
208 parameters	
H-atom parameters not refined	
$w = 1/\sigma^2(F)$	
$(\Delta/\sigma)_{max}$ = 0.03	

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

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

	x	y	z	B_{eq}
N(1)	0.6557 (3)	0.0000	0.5143 (2)	5.23 (7)
N(2)	0.3360 (3)	0.1291 (7)	0.3500 (2)	4.72 (7)
N(3)	0.3082 (3)	−0.1389 (8)	0.2415 (3)	5.21 (7)
N(4)	0.1312 (3)	0.0380 (8)	0.2532 (3)	5.67 (8)
N(5)	0.0178 (3)	0.2997 (9)	0.3250 (4)	6.7 (1)
N(6)	0.8653 (3)	0.0861 (9)	0.4976 (3)	7.8 (1)
N(7)	0.3905 (5)	0.225 (1)	0.7375 (5)	9.6 (2)
C(1)	0.7352 (3)	0.0918 (8)	0.4555 (3)	4.87 (8)
C(2)	0.5285 (3)	0.0126 (8)	0.4752 (3)	4.96 (8)
C(3)	0.4743 (3)	0.1085 (7)	0.3795 (3)	4.14 (7)
C(4)	0.5582 (3)	0.1995 (8)	0.3171 (3)	4.73 (8)
C(5)	0.6887 (4)	0.1924 (8)	0.3558 (3)	4.99 (8)
C(6)	0.2578 (3)	0.0106 (8)	0.2817 (3)	4.48 (8)
C(7)	0.2286 (4)	−0.2753 (9)	0.1712 (4)	6.3 (1)
C(8)	0.2995 (6)	−0.463 (1)	0.1645 (4)	7.1 (1)
C(9)	0.2784 (6)	−0.503 (1)	0.0379 (5)	7.8 (1)
C(10)	0.370 (1)	−0.382 (2)	−0.0105 (6)	12.2 (3)
C(11)	0.313 (2)	−0.191 (2)	−0.0050 (6)	15.6 (4)
C(12)	0.1914 (8)	−0.226 (1)	0.0433 (7)	10.9 (2)
C(13)	0.1511 (7)	−0.421 (2)	−0.0058 (7)	12.1 (2)
C(14)	0.0768 (3)	0.1798 (9)	0.2952 (4)	5.23 (9)
C(15)	0.2836 (5)	0.220 (1)	0.7304 (5)	7.5 (1)
C(16)	0.1438 (6)	0.221 (2)	0.730 (1)	12.9 (3)

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

N(1)—C(1)	1.342 (5)	N(1)—C(2)	1.332 (5)
N(2)—C(3)	1.433 (4)	N(2)—C(6)	1.349 (5)
N(3)—C(6)	1.320 (5)	N(3)—C(7)	1.452 (6)
N(4)—C(6)	1.320 (5)	N(4)—C(14)	1.306 (6)
N(5)—C(14)	1.149 (6)	N(6)—C(1)	1.364 (5)
N(7)—C(15)	1.104 (6)	C(1)—C(5)	1.397 (6)
C(2)—C(3)	1.362 (5)	C(3)—C(4)	1.398 (5)
C(4)—C(5)	1.361 (5)	C(7)—C(8)	1.547 (7)
C(7)—C(12)	1.54 (1)	C(8)—C(9)	1.502 (7)
C(9)—C(10)	1.48 (1)	C(9)—C(13)	1.47 (1)
C(10)—C(11)	1.50 (2)	C(11)—C(12)	1.49 (1)
C(12)—C(13)	1.55 (1)	C(15)—C(16)	1.456 (8)
C(1)—N(1)—C(2)	117.0 (3)	C(3)—N(2)—C(6)	124.2 (3)
C(6)—N(3)—C(7)	122.2 (3)	C(6)—N(4)—C(14)	120.0 (3)
N(1)—C(1)—N(6)	117.3 (3)	N(1)—C(1)—C(5)	122.3 (3)
N(6)—C(1)—C(5)	120.3 (3)	N(1)—C(2)—C(3)	124.8 (3)
N(2)—C(3)—C(2)	120.9 (3)	N(2)—C(3)—C(4)	121.1 (3)
C(2)—C(3)—C(4)	117.8 (3)	C(3)—C(4)—C(5)	119.0 (3)
C(1)—C(5)—C(4)	119.1 (3)	N(2)—C(6)—N(3)	119.6 (3)
N(2)—C(6)—N(4)	122.3 (4)	N(3)—C(6)—N(4)	118.1 (3)
N(3)—C(7)—C(8)	112.8 (4)	N(3)—C(7)—C(12)	115.5 (5)
C(8)—C(7)—C(12)	101.2 (4)	C(7)—C(8)—C(9)	103.3 (5)
C(8)—C(9)—C(10)	106.0 (5)	C(8)—C(9)—C(13)	104.1 (5)
C(10)—C(9)—C(13)	103.3 (8)	C(9)—C(10)—C(11)	103.5 (8)
C(10)—C(11)—C(12)	103.5 (8)	C(7)—C(12)—C(11)	108.9 (5)
C(7)—C(12)—C(13)	99.6 (7)	C(11)—C(12)—C(13)	101.6 (8)
C(9)—C(13)—C(12)	93.7 (5)	N(4)—C(14)—N(5)	173.2 (4)
N(7)—C(15)—C(16)	175.7 (8)		

N(1)—C(1)—C(5)—C(4)	0.6 (6)
N(1)—C(2)—C(3)—C(4)	0.5 (6)
N(2)—C(6)—N(3)—C(7)	177.6 (4)
N(3)—C(6)—N(2)—C(3)	2.6 (5)
N(3)—C(7)—C(8)—C(9)	−130.7 (5)
N(3)—C(7)—C(12)—C(13)	162.3 (5)
N(4)—C(6)—N(3)—C(7)	−2.2 (6)
N(6)—C(1)—N(1)—C(2)	177.3 (4)
C(1)—N(1)—C(2)—C(3)	1.2 (6)
C(2)—N(1)—C(1)—C(5)	−1.8 (6)
C(2)—C(3)—C(4)—C(5)	−1.6 (6)
C(6)—N(3)—C(7)—C(8)	−162.9 (4)
C(7)—C(8)—C(9)—C(10)	77.0 (7)
C(7)—C(12)—C(11)—C(10)	72.3 (8)
C(8)—C(7)—C(12)—C(11)	−65.8 (8)
C(8)—C(9)—C(10)—C(11)	−71.7 (8)
C(9)—C(8)—C(7)—C(12)	−6.7 (6)
C(9)—C(13)—C(12)—C(11)	53.2 (8)
C(10)—C(11)—C(12)—C(13)	−32.2 (8)
N(1)—C(2)—C(3)—N(2)	−174.0 (4)
N(2)—C(3)—C(4)—C(5)	172.8 (4)
N(2)—C(6)—N(4)—C(14)	−2.8 (6)
N(3)—C(6)—N(4)—C(14)	177.0 (4)
N(3)—C(7)—C(12)—C(11)	56.4 (8)
N(4)—C(6)—N(2)—C(3)	−177.6 (3)
N(5)—C(14)—N(4)—C(6)	173 (3)
N(6)—C(1)—C(5)—C(4)	−178.4 (4)
C(1)—C(5)—C(4)—C(3)	1.1 (6)
C(2)—C(3)—N(2)—C(6)	−90.9 (4)
C(4)—C(3)—N(2)—C(6)	94.8 (5)
C(6)—N(3)—C(7)—C(12)	81.4 (6)
C(7)···C(8)···C(9)—C(13)	−31.6 (7)
C(7)—C(12)—C(13)—C(9)	−58.5 (7)
C(8)—C(7)—C(12)—C(13)	40.1 (7)
C(8)—C(9)—C(13)—C(12)	55.4 (8)
C(9)—C(10)—C(11)—C(12)	−1.7 (9)
C(10)—C(9)—C(13)—C(12)	−55.1 (8)
C(11)—C(10)—C(9)—C(13)	37.4 (7)

The structure was solved by direct methods using *SHELXS*86 (Sheldrick, 1985) and expanded by Fourier techniques using *DIRDIF*92 (Beurskens *et al.*, 1992). The structure was refined by a full-matrix least-squares procedure. All calculations were performed using *TEXSAN* (Molecular Structure Corporation, 1992).

Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the IUCr (Reference: OH1073). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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Bis(5-chloro-2-nitrophenyl) Disulfide

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Abstract

The title molecule, $C_{12}H_6Cl_2N_2O_4S_2$, lies on a twofold axis in the crystal. The C—S—S—C group has a skewed non-planar conformation with a torsion-angle magnitude of 86.7 (1)°. The S—S—C bond angle is 104.11 (5)°, and the S—S, S—C, C—N and C—Cl bond lengths are 2.0432 (5), 1.789 (1), 1.462 (2) and 1.726 (1) Å, respectively. The nitro group is rotated 15.2 (2)° out of the plane of the phenyl ring.

Comment

The title compound, (I), consists of two identical monomeric units linked by a disulfide bridge lying on a twofold axis. The torsion angle about the S—S bond is 86.7 (1)°.

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