Foundation (grant CHE 8708625), as well as the use of the computing facilities of the University of Birmingham Computer Centre, the University of Manchester Regional Computing Centre and Staffordshire University, Stoke site. Special thanks are due to Dr M. Horder at Birmingham and Mr A. Sherwood at Stoke for help with file conversions and plotting.

Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the IUCr (Reference: HH1069). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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L-Nitroarginine Monohydrochloride Monohydrate

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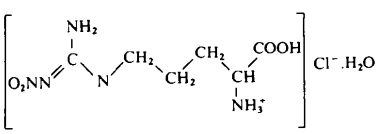
Abstract

Two conformers are present in the asymmetric unit of the title compound, $C_6H_{14}N_5O_4^+.Cl^-.H_2O$, whose conformational differences are characterized by rotations about the C^α—C^β and C^δ—N^ε bonds. The nitroguanidyl moieties of both conformers are nearly planar; each is involved in an intramolecular hydrogen bond between the N^ε atom and an O atom of the nitro group. Molecules are held together by a hydrogen-bond network involving the nitroguanidyl moieties, the protonated α-amino and carboxyl groups, two water molecules and two chloride ions.

Comment

L-Nitroarginine (1) is a specific and potent inhibitor of nitric oxide (NO) generation (Mülsch & Busse, 1990) which is formed by the enzymatic oxidation of L-arginine and plays a crucial role in non-adrenergic and non-cholinergic inhibitory neuromuscular transmission in certain smooth muscles (Moncada, Palmer & Higgs, 1991; Rand, 1992). For this reason, it is important to determine the precise structure of L-nitroarginine in order to clarify the relationship between its structure and function.



(1)

L-Nitroarginine crystallizes with two conformers per asymmetric unit [forms (I) and (II)]. Both conformers have extended conformations as shown in Fig. 1. The prominent conformational differences between forms (I) and (II) are characterized by two rotations; one is a rotation about the C^α—C^β bond [N(1)—C(2)—C(3)—C(4): −53 (2) for (I), 64 (1)° for (II)], and the other is about the C^δ—N^ε bond [C(4)—C(5)—N(2)—C(6): −91 (1) for (I), 94 (1)° for (II)]. The nitroguanidyl group adopts a nearly planar conformation [C(5)—N(2)—C(6)—N(3), N(2)—C(6)—N(4)—N(5), C(6)—N(4)—N(5)—O(4): 4 (2), −3 (1) and −5 (1)° for (I), −8 (1), 0 (1) and 7 (1)° for (II)]. The hydrocarbon part of the side chain has a *trans* conformation [C(2)—C(3)—C(4)—C(5): −171 (1) for (I), −171.1 (7)° for (II)] and is oriented almost perpendicular to the nitroguanidyl plane. Intramoleculear hydrogen bonds, N(2)—H(2)···O(4) [2.57 (1) Å] and N(2′)—H(2′)···O(4′) [2.58 (1) Å], are present in the nitroguanidyl plane. Two conformers are linked through an intermolecular hydrogen-bonding network in which the $—NH_3^+$, —COOH and $—NH_2$ groups and H_2O molecules act as proton donors, and the Cl^- ions, the O atoms of the $—NO_2$ and —COOH groups, H_2O molecules and the —N= atoms all function as proton acceptors. All H atoms of the above donor groups take part in hydrogen bonding to the proton acceptors. The details of the hydrogen-bond geometry are available as part of the supplementary material. The thermal motion of the atoms C(3) and C(4) of form (I) is quite high, resulting in the unusual C(3)—C(4) bond length and C(2)—C(3)—C(4) and C(3)—C(4)—C(5) angles as compared with the normal values of form (II).

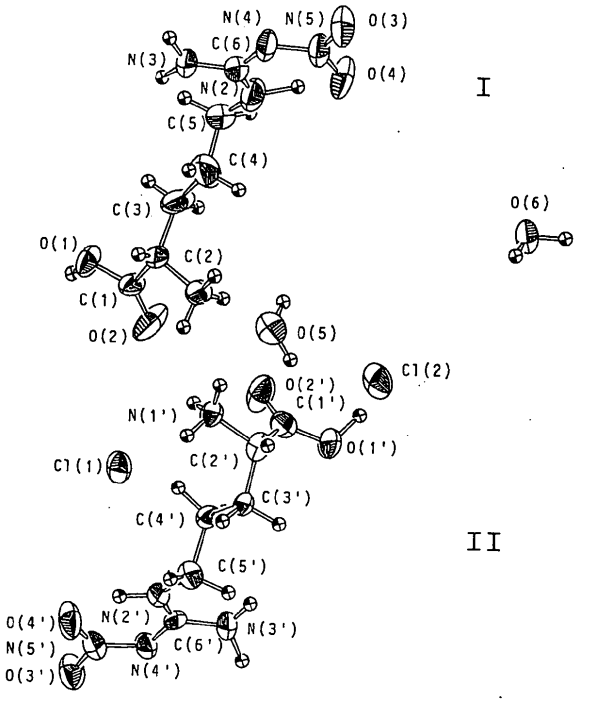


Fig. 1. Perspective view of the two conformers [(I) and (II)] of the title compound with the atomic numbering viewed down the *b* axis.

The crystal structures of L-arginine dihydrate and diarsenate have been reported elsewhere (Lehmann, Verbist, Hamilton & Koetzle, 1973; Zalkin, Eimerl & Velsko, 1989).

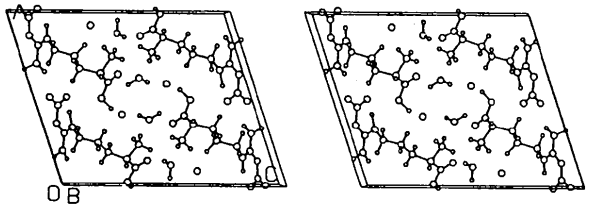


Fig. 2. Stereoview of the title compound showing the packing in the unit cell viewed down the *b* axis.

Experimental

Crystal data

$C_6H_{14}N_5O_4^+.Cl^-.H_2O$
$M_r = 273.68$
Monoclinic
$P2_1$
$a = 13.559$ (3) Å
$b = 5.936$ (3) Å
$c = 16.339$ (3) Å
$\beta = 107.80$ (2)°
$V = 1252$ (1) Å^3
$Z = 4$
$D_x = 1.452$ Mg m^{-3}
$D_m = 1.449$ (4) Mg m^{-3}

Mo $K\alpha$ radiation
$\lambda = 0.71069$ Å
Cell parameters from 25 reflections
$\theta = 4.3$–$7.35°$
$\mu = 0.320$ mm^{-1}
$T = 296$ K
Plate
0.40 × 0.30 × 0.20 mm
Colorless
Crystal source: from 0.05 *N* HCl/50% ethanol

Data collection

Rigaku AFC-5*R* diffractometer
$\omega/2\theta$ scans
Absorption correction: empirical (*DIFABS*; Walker & Stuart, 1983)
$T_{min} = 0.66$, $T_{max} = 1.06$
3292 measured reflections
3164 independent reflections
1510 observed reflections [$I > 2.5\sigma(I)$]

$R_{int} = 0.022$
$\theta_{max} = 27.5°$
$h = 0 \rightarrow 17$
$k = -7 \rightarrow 0$
$l = -18 \rightarrow 17$
3 standard reflections monitored every 150 reflections
intensity variation: −1.6%

Refinement

Refinement on *F*
$R = 0.056$
$wR = 0.057$
$S = 1.59$
1510 reflections
306 parameters
H-atom parameters not refined

$w = 4F_o^2/\sigma^2(F_o^2)$
$(\Delta/\sigma)_{max} = 0.01$
$\Delta\rho_{max} = 1.41$ e Å^{-3}
$\Delta\rho_{min} = -3.60$ e Å^{-3}
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)

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

	x	*y*	*z*	B_{eq}
Cl(1)	0.9202 (2)	−0.3397	0.3596 (2)	4.4 (1)
Cl(2)	0.4100 (2)	−0.2670 (6)	0.3718 (2)	5.6 (1)
O(1)	0.9883 (4)	−0.433 (1)	0.6932 (3)	4.3 (3)
O(1′)	0.5029 (4)	0.106 (1)	0.2968 (4)	4.9 (3)
O(2)	0.8620 (5)	−0.469 (2)	0.5713 (4)	7.4 (4)
O(2′)	0.6404 (5)	0.024 (1)	0.4077 (4)	6.5 (4)
O(3)	0.4920 (5)	0.496 (2)	0.9310 (5)	5.9 (4)
O(3′)	1.0059 (5)	−0.499 (1)	0.0648 (4)	5.2 (3)
O(4)	0.4912 (6)	0.201 (1)	0.8531 (5)	6.3 (4)
O(4′)	1.0115 (5)	−0.205 (1)	0.1444 (5)	6.1 (4)
O(5)	0.6213 (5)	−0.413 (1)	0.5037 (4)	5.1 (3)
O(6)	0.1187 (4)	−0.451 (1)	0.5208 (4)	4.7 (3)
N(1)	0.7665 (5)	−0.094 (1)	0.5930 (4)	3.8 (3)
N(1′)	0.7373 (5)	0.405 (1)	0.3990 (4)	3.5 (3)
N(2)	0.6679 (6)	0.016 (2)	0.8695 (5)	5.1 (4)
N(2′)	0.8361 (5)	−0.031 (1)	0.1419 (5)	3.5 (3)
N(3)	0.8012 (6)	0.234 (2)	0.9502 (5)	4.2 (4)
N(3′)	0.6988 (6)	−0.251 (2)	0.0617 (5)	4.3 (4)
N(4)	0.6426 (6)	0.359 (2)	0.9385 (5)	4.2 (4)
N(4′)	0.8590 (5)	−0.370 (2)	0.0694 (4)	3.5 (3)
N(5)	0.5381 (6)	0.344 (2)	0.9058 (6)	4.5 (4)
N(5′)	0.9614 (6)	−0.347 (2)	0.0943 (5)	4.3 (4)
C(1)	0.9004 (6)	−0.375 (2)	0.6372 (5)	3.8 (4)
C(1′)	0.5947 (7)	0.145 (2)	0.3484 (6)	4.0 (4)
C(2)	0.8517 (7)	−0.177 (2)	0.6664 (5)	4.0 (4)
C(2′)	0.6432 (7)	0.351 (2)	0.3252 (6)	3.6 (4)
C(3)	0.8134 (9)	−0.251 (3)	0.7413 (6)	11 (1)
C(3′)	0.6703 (6)	0.327 (2)	0.2412 (5)	3.1 (4)
C(4)	0.7570 (9)	−0.117 (4)	0.7746 (8)	11 (1)
C(4′)	0.7399 (6)	0.137 (2)	0.2383 (5)	3.5 (4)
C(5)	0.7310 (7)	−0.167 (2)	0.8517 (6)	4.8 (5)
C(5′)	0.7729 (6)	0.159 (2)	0.1568 (6)	4.2 (4)
C(6)	0.7021 (7)	0.198 (2)	0.9172 (6)	3.9 (5)
C(6′)	0.8016 (7)	−0.212 (2)	0.0943 (5)	3.4 (4)

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

O(1)—C(1)	1.307 (9)	N(3)—C(6)	1.30 (1)
O(1′)—C(1′)	1.29 (1)	N(3′)—C(6′)	1.35 (1)
O(2)—C(1)	1.18 (1)	N(4)—N(5)	1.36 (1)
O(2′)—C(1′)	1.21 (1)	N(4)—C(6)	1.36 (1)
O(3)—N(5)	1.24 (1)	N(4′)—N(5′)	1.329 (9)

O(3′)—N(5′)	1.26 (1)	N(4′)—C(6′)	1.36 (1)
O(4)—N(5)	1.24 (1)	C(1)—C(2)	1.49 (1)
O(4′)—N(5′)	1.23 (1)	C(1′)—C(2′)	1.49 (1)
N(1)—C(2)	1.48 (1)	C(2)—C(3)	1.53 (1)
N(1′)—C(2′)	1.50 (1)	C(2′)—C(3′)	1.53 (1)
N(2)—C(5)	1.47 (1)	C(3)—C(4)	1.33 (2)
N(2)—C(6)	1.33 (1)	C(3′)—C(4′)	1.48 (1)
N(2′)—C(5′)	1.48 (1)	C(4)—C(5)	1.44 (1)
N(2′)—C(6′)	1.32 (1)	C(4′)—C(5′)	1.53 (1)
C(5)—N(2)—C(6)	126.6 (9)	N(1)—C(2)—C(3)	111.9 (7)
C(5′)—N(2′)—C(6′)	126.5 (8)	C(1)—C(2)—C(3)	109 (1)
N(5)—N(4)—C(6)	118.8 (9)	N(1′)—C(2′)—C(1′)	107.6 (7)
N(5′)—N(4′)—C(6′)	118.5 (8)	N(1′)—C(2′)—C(3′)	111.3 (7)
O(3)—N(5)—O(4)	121.7 (8)	C(1′)—C(2′)—C(3′)	113.4 (8)
O(3)—N(5)—N(4)	113 (1)	C(2)—C(3)—C(4)	122 (1)
O(4)—N(5)—N(4)	125 (1)	C(2′)—C(3′)—C(4′)	115.4 (7)
O(3′)—N(5′)—O(4′)	121.0 (8)	C(3)—C(4)—C(5)	124 (2)
O(3′)—N(5′)—N(4′)	112.7 (9)	C(3′)—C(4′)—C(5′)	108.7 (8)
O(4′)—N(5′)—N(4′)	126.1 (9)	N(2)—C(5)—C(4)	110 (1)
O(1)—C(1)—O(2)	124.4 (9)	N(2′)—C(5′)—C(4′)	114.4 (8)
O(1)—C(1)—C(2)	112.4 (8)	N(2)—C(6)—N(3)	121 (1)
O(2)—C(1)—C(2)	123.2 (8)	N(2)—C(6)—N(4)	126.3 (9)
O(1′)—C(1′)—O(2′)	125 (1)	N(3)—C(6)—N(4)	113 (1)
O(1′)—C(1′)—C(2′)	113.2 (9)	N(2′)—C(6′)—N(3′)	120.6 (9)
O(2′)—C(1′)—C(2′)	122.1 (9)	N(2′)—C(6′)—N(4′)	127.1 (9)
N(1)—C(2)—C(1)	108.6 (7)	N(3′)—C(6′)—N(4′)	112.2 (9)

The crystal was sealed in a capillary to prevent sublimation. The positions of the H atoms on atoms C(3) and C(4) were calculated geometrically. Other H atoms were subsequently located in successive difference Fourier maps. Program used for data collection and cell refinement: *MSC/AFC Diffractometer Control Software* (Molecular Structure Corporation, 1988). Programs used to solve structure: *SHELXS*86 (Sheldrick, 1985) and *DIRDIF* (Beurskens, 1984). All calculations, including data reduction, were carried out using the *TEXSAN* crystallographic package (Molecular Structure Corporation, 1985).

Lists of structure factors, anisotropic displacement parameters, H-atom coordinates, complete geometry, torsion angles and hydrogen-bond geometry details have been deposited with the IUCr (Reference: AS1098). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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trans-1-(4-Iodophenyl)-2-phenyl-1-{4-[2-(1-pyrrolidinyl)ethoxy]phenyl}-1-butene, $C_{28}H_{30}INO$

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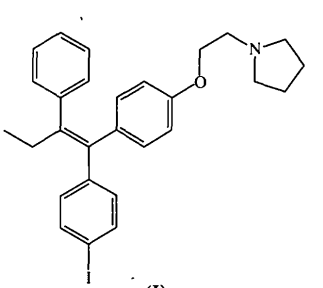
Abstract

The title compound is a tamoxifen derivative. The crystal structure shows a central ethylene bond about which three phenyl rings adopt a propeller conformation. The [2-(1-pyrrolidinyl)ethoxy]phenyl ring lies *trans* with respect to the ethyl group across the ethylene bond.

Comment

This study reports the structure of a *trans*-tamoxifen derivative. *trans*-Tamoxifen and its derivatives show antioestrogenic activity and are important for the treatment of hormone-sensitive breast cancer. A number of tamoxifen structures have been reported previously, and include *trans*-tamoxifen (Precigoux, Courseille, Geoffre & Hospital, 1979), 2-hydroxytamoxifen, 3-hydroxytamoxifen, 2-methyl-4-hydroxytamoxifen (Kuroda, Cutbush, Neidle & Leung, 1985), 4-methylthiotamoxifen (Blackburn, Goodman & Smith, 1988) and tamoxifen citrate (Goldberg & Becker, 1987). The structure of the *E* and *Z* isomers of an iodotamoxifen have been reported (Hunter, Payne, Rahman, Richardson & Ponce, 1983). The *Z* isomer of iodotamoxifen differs from the title compound (I) in having a dimethylammine group in place of the pyrrolidinyl group and a different iodo-substituted phenyl ring.



(I)

The overall conformation of the structure is similar to that seen for the other tamoxifen structures. The central triphenylethylene system adopts a propeller conformation. The dihedral angles formed between the (1) unsubstituted phenyl, (2) 4-iodophenyl and (3) [2-