Howard-Lock, H., Lock, C. J. L. & Smalley, P. (1983). Unpublished work.
International Tables for X-ray Crystallography (1974). Vol. IV. Birmingham: Kynoch Press.
Johnson, C. K. (1976). *ORTEP* II. Report ORNL-5138. Oak Ridge National Laboratory, Tennessee.
Jönsson, P.-G. & Olovsson, I. (1968). *Acta Cryst.* B**24**, 559–564.
Kitano, M. & Kuchitsu, K. (1973). *Bull. Chem. Soc. Jpn*, **46**, 3048–3051.
Kvick, A., Jönsson, P.-G. & Olvosson, J. (1969). *Inorg. Chem.* **8**, 2775–2780.
Muir, K. W. & Speakman, J. C. (1979). *J. Chem. Res.* (*S*), p. 277.
Pippy, M. E. & Ahmed, F. R. (1978). *NRC*22. *Mean Plane and Torsion Angles*. National Research Council of Canada, Ottawa, Canada.
Sheldrick, G. M. (1976). *SHELX*. Program for crystal structure determination. Univ. of Cambridge, England.
Stewart, J. M. (1976). Editor, the *XRAY*76 system. Tech. Rep. TR-446. Computer Science Center, Univ. of Maryland, College Park, Maryland.
Sundaralingam, M. & Chwang, A. K. (1976). *Carbonium Ions*, 5, 2427–2476.
Vinogradov, S. N. & Linnell, R. H. (1971). *Hydrogen Bonding*. New York: Van Nostrand-Reinhold.

Acta Cryst. (1984). C**40**, 1294–1295

1-(*p*-Chlorophenyl)biguanide Hydrochloride, $C_8H_{11}ClN_5^+.Cl^-$

By C. J. Brown and L. Sengier

Department of Metallurgy and Materials Engineering, City of London Polytechnic, Central House, Whitechapel High Street, London E1 7PF, England

(*Received* 30 *November* 1983; *accepted* 9 *April* 1984)

Abstract. $M_r = 248{\cdot}1$, monoclinic, $P2_1/n$, $a = 13{\cdot}604\,(7)$, $b = 5{\cdot}719\,(5)$, $c = 15{\cdot}376\,(8)$ Å, $\beta = 108{\cdot}26\,(4)°$, $V = 1136{\cdot}03$ Å^3, $Z = 4$, $D_m = 1{\cdot}433\,(5)$, $D_x = 1{\cdot}450$ Mg m^{-3}, Mo $K\alpha$, $\lambda = 0{\cdot}71069$ Å, $\mu = 0{\cdot}55$ mm^{-1}, $F(000) = 512$, room temperature, $R = 0{\cdot}046$ for 1843 observed reflexions [$I > \sigma(I)$]. The Cl^- ion is coordinated octahedrally to six N atoms at a mean distance of 3·272 (2) Å. The C—N bonds of the biguanide moiety are within the range 1·307 (3)–1·352 (3) Å; it consists of two planes at an angle of 50·72 (5)° to each other.

Introduction. The title compound is one of a series of substances well known for their pharmacological activity. Crystal data for a variant unit cell of this and for a number of related compounds have already been published (Brown, 1967). This structure determination was carried out to confirm the configuration of the biguanide residue.

Experimental. Acicular crystals (1·0 × 0·5 × 0·2 mm) prepared by Dr J. A. Hendry, Imperial Chemical Industries Limited (Pharmaceuticals Division). Lattice parameters obtained initially from rotation and Weissenberg photographs and refined by least-squares fit to 26 reflexions on the diffractometer. D_m by flotation in NaI solution. Nonius CAD-4 diffractometer. Two standard reflexions, no variation in intensity. $\theta_{max} = 25°$ (Mo $K\alpha$ radiation). Index range h 0 to 16, k 0 to 6, l −18 to +17. Corrections for Lp effects but not for absorption or extinction. Structure solved by direct methods with *MULTAN* (Main, Hull, Lessinger, Germain, Declercq & Woolfson, 1978) and refined by least squares on F with $\sqrt{w} = 1/F_o$ using programs of Ahmed, Hall, Pippy & Huber (1970) implemented on our DEC-10 computer. B_{iso} used initially and B_{ij} finally. Approximate H-atom coordinates from difference Fourier maps; included in structure factor calculations without refining at idealized positions with $B_{iso} = 5{\cdot}0$ Å^2 for phenyl H and 6·0 Å^2 for biguanide H. 153 reflexions [$I < \sigma(I)$] discarded. Δ/σ during last refinement cycle <0·2. Max. $\Delta\rho$ in final difference map ±0·2 e Å^{-3}. Scattering factors from *International Tables for X-ray Crystallography* (1974). $R = 0{\cdot}046$, $R_w = 0{\cdot}127$ for the 1843 observed reflexions.

Discussion. The atomic coordinates and equivalent isotropic temperature factors are given in Table 1,* and bond lengths and angles in Table 2. The atom numbering and molecular geometry are shown in Fig. 1.

The Cl^- ion is coordinated in the form of a severely distorted octahedron to six N atoms at distances shown in Fig. 1 [mean Cl^- to N = 3·272 (2) Å, compared with 3·270 Å in the 5-isopropyl derivative (Brown, 1967)]. There is some uncertainty as to whether these are hydrogen bonds or not; suffice it to say that there is an

---

* Lists of structure factors, anisotropic thermal parameters, calculated H-atom parameters, least-squares-planes calculations and intermolecular distances have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 39387 (16 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

0108-2701/84/071294-02$01.50

H atom available for each of the N—H···Cl⁻ coordinations, but the determination of the exact location of each H atom was not feasible with the data available.

Table 1. *Final atomic parameters and e.s.d.'s*

$$B_{\mathrm{eq}} = \tfrac{4}{3}\left(\frac{\beta_{11}}{a^{*2}} + \frac{\beta_{22}}{b^{*2}} + \frac{\beta_{33}}{c^{*2}}\right).$$

	x	y	z	B_{eq}(Å^2)
Cl⁻(1)	0·68073 (4)	0·03026 (10)	0·08590 (3)	4·40 (1)
Cl(2)	0·31673 (6)	1·34549 (13)	0·42800 (6)	5·88 (1)
C(1)	0·5163 (2)	0·8507 (4)	0·3281 (1)	3·62 (3)
C(2)	0·4102 (2)	0·8061 (4)	0·3092 (2)	5·02 (4)
C(3)	0·3480 (2)	0·9591 (5)	0·3380 (2)	4·63 (4)
C(4)	0·3934 (2)	1·1573 (4)	0·3872 (2)	4·50 (4)
C(5)	0·4979 (2)	1·1971 (4)	0·4081 (2)	4·86 (4)
C(6)	0·5581 (2)	1·0445 (4)	0·3780 (2)	4·35 (4)
C(7)	0·5594 (2)	0·5526 (4)	0·2298 (1)	4·14 (3)
C(8)	0·4321 (2)	0·4038 (4)	0·1033 (1)	4·02 (3)
N(1)	0·5821 (1)	0·6961 (3)	0·3025 (1)	4·39 (3)
N(2)	0·6369 (2)	0·4071 (4)	0·2276 (1)	4·52 (4)
N(3)	0·4699 (2)	0·5728 (4)	0·1654 (1)	4·61 (3)
N(4)	0·3778 (2)	0·4628 (4)	0·0200 (1)	5·59 (4)
N(5)	0·4399 (2)	0·1750 (3)	0·1241 (1)	5·46 (4)

Table 2. *Bond lengths* (Å) *and interbond angles* (°)

Cl(2)—C(4)	1·747 (3)	C(5)—C(6)	1·373 (4)
C(1)—C(2)	1·403 (4)	C(7)—N(1)	1·342 (3)
C(1)—C(6)	1·366 (3)	C(7)—N(2)	1·352 (3)
C(1)—N(1)	1·399 (3)	C(7)—N(3)	1·312 (3)
C(2)—C(3)	1·384 (4)	C(8)—N(3)	1·343 (3)
C(3)—C(4)	1·394 (4)	C(8)—N(4)	1·307 (3)
C(4)—C(5)	1·374 (4)	C(8)—N(5)	1·343 (3)
C(2)—C(1)—C(6)	119·0 (2)	C(1)—C(6)—C(5)	121·2 (2)
C(2)—C(1)—N(1)	122·2 (2)	N(1)—C(7)—N(2)	114·8 (2)
C(6)—C(1)—N(1)	118·7 (2)	N(1)—C(7)—N(3)	119·4 (2)
C(1)—C(2)—C(3)	120·7 (2)	N(2)—C(7)—N(3)	125·6 (2)
Cl(2)—C(4)—C(3)	118·8 (2)	N(3)—C(8)—N(4)	119·0 (2)
Cl(2)—C(4)—C(5)	120·3 (2)	N(3)—C(8)—N(5)	123·2 (2)
C(3)—C(4)—C(5)	120·8 (2)	N(4)—C(8)—N(5)	117·6 (2)
C(2)—C(3)—C(4)	118·4 (2)	C(1)—N(1)—C(7)	128·3 (2)
C(4)—C(5)—C(6)	119·9 (2)	C(7)—N(3)—C(8)	122·1 (2)

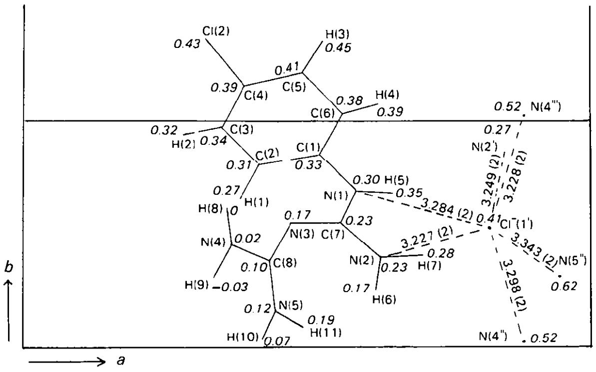


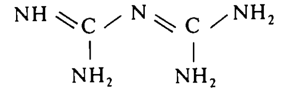
Fig. 1. Diagram of one molecule, showing relation to axial directions, numbering of atoms and Cl⁻ coordination (distances in Å). The italicized numbers alongside each atom are the z fractional coordinates. Symmetry code: (i) $\frac{1}{2}-x, \frac{1}{2}+y, \frac{1}{2}-z$; (ii) $\frac{1}{2}+x, \frac{1}{2}-y, \frac{1}{2}+z$; (iii) $\frac{1}{2}+x, \frac{3}{2}-y, \frac{1}{2}+z$.

There is a close resemblance between the dimensions of the biguanide residues in this structure and in Paludrine (Brown, 1967), particularly in the shorter bonds, C(7)—N(3) 1·312 (3), 1·307 (5) and C(8)—N(4) 1·307 (3), 1·320 (5) Å, respectively, and in view of the fact that N(3) carries no H, these two bonds must be regarded as formal double bonds in both structures.

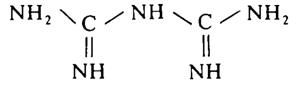
Since C(8)—N(4) is appreciably shorter than the other terminal bonds, it is probable that the proton of the HCl has become attached to N(4), although the small differences in C—N lengths indicate considerable resonance and distribution of the positive charge amongst the other H atoms.

The two halves of the biguanide residue are each slightly pyramidal, with C(7) −0·030 (2) Å out of the N(1), N(2), N(3) plane, and C(8) +0·037 (2) Å out of the N(3),N(4),N(5) plane (equations of planes have been deposited). These two halves make a dihedral angle of 50·72 (5)° compared with 58·9° in Paludrine (Brown, 1967). The benzene ring is planar (r.m.s. deviation 0·008 Å).

As a result of this structure determination, it would seem that the preferred configuration for the biguanide is


$\mathrm{NH{=}C(NH_2){-}N{=}C(NH_2){-}NH_2}$

with the formal double bonds in the positions shown rather than


$\mathrm{NH_2{-}C({=}NH){-}NH{-}C({=}NH){-}NH_2}$

which is usually found in textbooks.

Our thanks are due to Dr M. B. Hursthouse for arranging the data collection on a Nonius CAD-4 diffractometer at Queen Mary College.

References

Ahmed, F. R., Hall, S. R., Pippy, M. E. & Huber, C. P. (1970). *NRC* crystallographic programs for the IBM/360 system. National Research Council, Ottawa, Canada.
Brown, C. J. (1967). *J. Chem. Soc. A*, pp. 60–65.
International Tables for X-ray Crystallography (1974). Vol. IV, pp. 72–98. Birmingham: Kynoch Press.
Main, P., Hull, S. E., Lessinger, L., Germain, G., Declercq, J.-P. & Woolfson, M. M. (1978). *MULTAN. A System of Computer Programs for the Automatic Solution of Crystal Structures from X-ray Diffraction Data.* Univs. of York, England, and Louvain-la-Neuve, Belgium.