Plus (Sheldrick, 1990*b*). Software used to prepare material for publication: *SHELXL*93.

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References

Desiraju, G. R. (1991). *Acc. Chem. Res.* **24**, 290–296.
Herrnstadt, C., Mootz, D., Wunderlich, H. & Möhrle, H. (1979). *J. Chem. Soc. Perkin Trans.* 2, pp. 735–740.
Shapiro, S. L., Parrino, V. A. & Freedman, L. (1959). *J. Am. Chem. Soc.* **81**, 2220–2223.
Sheldrick, G. M. (1990*a*). *Acta Cryst.* A**46**, 467–473.
Sheldrick, G. M. (1990*b*). *SHELXTL-Plus*. Siemens Analytical X-ray Instruments Inc., Madison, Wisconsin, USA.
Sheldrick, G. M. (1993). *SHELXL*93. *Program for the Refinement of Crystal Structures*. University of Göttingen, Germany.
Siemens (1991). *XSCANS Users Manual*. Siemens Analytical X-ray Instruments Inc., Madison, Wisconsin, USA.

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Cyclopropylcarboxamidinium Chloride

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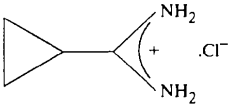
Abstract

Cyclopropylcarboxamidine hydrochloride, $C_4H_9N_2^+.Cl^-$, the first structural example of an alicyclic central C-atom substituted amidine, contains almost equal C—N bond lengths of 1.316 (4) and 1.304 (4) Å, indicating that it is an amidinium salt.

Comment

The widespread interest in amidines as a result of their importance in the pharmaceutical, biological and coordination chemistry fields is reflected in the number of structural papers which have appeared (Dehnicke, 1990; Edelmann, 1994; Barker & Kilner, 1994; Alcock *et al.*, 1994; Barker *et al.*, 1997). The present study of cyclopropylcarboxamidine hydrochloride, (I), was carried out to furnish data with regard to the effect of an alicyclic group on the amidine fragment.



(I)

The C—N bond lengths [1.316 (4) and 1.304 (4) Å] are neither single nor double bond in character. They are similar to those found in the straight-chain isopropylamidine hydrochloride analogue, (II) [with mean bond lengths 1.317 (5) and 1.300 (6) Å; Barker & Powell, 1996], acetamidinium chloride, (III) [1.305 (2) and 1.310 (3) Å; Cannon *et al.*, 1976], and bis(acetamidinium) carbonate monohydrate, (IV) [1.308 (5) and 1.315 (5) Å; Norrestam, 1984], but differ somewhat from those found for benzamidine hydrochloride monohydrate, (V) [1.293 (7) and 1.328 (7) Å; Thailambal *et al.*, 1986]. These C—N bond lengths are indicative of an amidinium salt configuration. Comparison of their C—N bond lengths shows the aryl substituent to be more restrictive toward delocalization than the alicyclic group. The N—C—N angle [120.5 (3)°] is similar to that of compounds (II) [120.4 (4)°] and (III) [120.5 (2)°], and there is a slight (but statistically insignificant) deviation from those found for (IV) [121.6 (3)°] and (V) [121.6 (4)°]. This indicates that the amidinium–

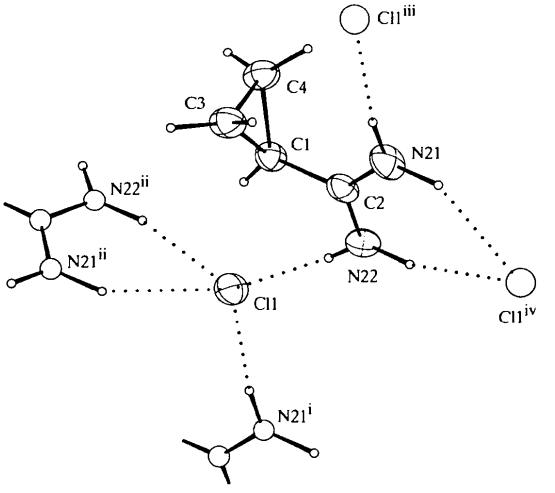


Fig. 1. The molecular structure of cyclopropylamidinium chloride showing the hydrogen bonding of both counterions. The asymmetric unit is drawn with 50% probability displacement ellipsoids and symmetry-related atoms with spheres. The symmetry codes are: (i) $x-\frac{1}{2}, \frac{1}{2}+y, z$; (ii) $x-\frac{1}{2}, \frac{1}{2}-y, z-\frac{1}{2}$; (iii) $x+\frac{1}{2}, y-\frac{1}{2}, z$; (iv) $x+\frac{1}{2}, \frac{1}{2}-y, z+\frac{1}{2}$.

chlorine interaction is similar in acetamidinium chloride, isopropylamidinium chloride, cyclopropylamidine hydrochloride and benzamidine hydrochloride. The delocalization in the title compound around the NCN fragment is also responsible for a short C—CNN bond [1.459 (4) Å], which is shorter than in its straight-chain analogue, (II) [1.488 (3) Å].

The NCN plane is perpendicular [90.35°] to the cyclopropyl-ring plane, presumably due to the steric hindrance between the NH_2 groups and the cyclopropyl ring. The cyclopropyl-ring geometry is in general agreement with that of other published structures (Allen *et al.*, 1987), with bond lengths C3—C4 1.483 (4), C1—C3 1.515 (4) and C1—C4 1.512 (4) Å indicating a slight elongation of the τ-bonded triangle towards the amidino-substituted C atom.

There are four N···Cl contacts in the range 3.223 (3)–3.377 (3) Å, indicating an extended hydrogen-bonded structure.

Experimental

Crystals of (I) were obtained by slow evaporation (over several days) of an acetone solution in the dark.

Crystal data

$C_4H_9N_2^+.Cl^-$
M_r = 120.58
Monoclinic
$C2/c$
a = 11.591 (1) Å
b = 10.891 (1) Å
c = 10.903 (1) Å
β = 118.00 (2)°
V = 1215.2 (3) Å^3
Z = 8
D_x = 1.32 Mg m^{-3}
D_m not measured

Mo $K\alpha$ radiation
λ = 0.71073 Å
Cell parameters from 25 reflections
θ = 74.31–74.96°
μ = 0.51 mm^{-1}
T = 293 K
Irregular block
0.30 × 0.30 × 0.20 mm
Colourless

Data collection

Rigaku R-Axis-IIc image-plate diffractometer
Arndt–Wonacott (1997) method; 30 non-overlapping images, each with an exposure time of 30 min and an oscillation range of 6.0°
Absorption correction: none

2865 measured reflections
969 independent reflections
898 reflections with $I > 3\sigma(I)$
R_{int} = 0.03
θ_{max} = 24.43°
$h = -13 \rightarrow 11$
$k = -12 \rightarrow 12$
$l = -12 \rightarrow 11$

Refinement

Refinement on F
R = 0.052
wR = 0.053
S = 1.098
898 reflections
101 parameters
All H atoms refined

$(\Delta/\sigma)_{max}$ = 0.0017
$\Delta\rho_{max}$ = 0.16 e Å^{-3}
$\Delta\rho_{min}$ = −0.17 e Å^{-3}
Extinction correction: Larson (1970), equation 22
Extinction coefficient: 100 (9) × 10

Weights: Chebychev polynomial (Carruthers & Watkin, 1979)

Scattering factors from *International Tables for X-ray Crystallography* (Vol. IV)

Table 1. *Selected geometric parameters (Å, °)*

C1—C2	1.459 (4)	C3—C4	1.483 (4)
C1—C3	1.515 (4)	Cl1···N21^{i}	3.377 (3)
C1—C4	1.512 (4)	Cl1···N21^{ii}	3.376 (3)
C2—N21	1.316 (4)	Cl1···N22	3.305 (3)
C2—N22	1.304 (4)	Cl1···N22^{ii}	3.223 (3)
C2—C1—C3	121.9 (2)	C1—C2—N22	117.8 (2)
C2—C1—C4	120.4 (2)	N21—C2—N22	120.5 (3)
C3—C1—C4	58.7 (2)	C1—C3—C4	60.6 (2)
C1—C2—N21	121.7 (2)	C1—C4—C3	60.8 (2)

Symmetry codes: (i) $x - \frac{1}{2}, \frac{1}{2} + y, z$; (ii) $x - \frac{1}{2}, \frac{1}{2} - y, z - \frac{1}{2}$.

The image-plate detector allowed data collection from a rather poor quality crystal which gave somewhat diffuse diffraction spots. The unit-cell dimensions were determined on a four-circle diffractometer (Cu $K\alpha$ radiation) with a better crystal which became available later.

Data collection: *R-Axis Processing Software* (Molecular Structure Corporation, 1993*a*). Cell refinement: *AFC-7R Processing Software* (Molecular Structure Corporation, 1993*b*). Data reduction: *R-Axis Processing Software*. Program(s) used to solve structure: *SIR*92 (Altomare *et al.*, 1994). Program(s) used to refine structure: *CRYSTALS* (Watkin, Prout, Carruthers & Betteridge, 1996). Molecular graphics: *CAMERON* (Watkin, Prout & Pearce, 1996). Software used to prepare material for publication: *CRYSTALS*.

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Supplementary data for this paper are available from the IUCr electronic archives (Reference: HA1206). Services for accessing these data are described at the back of the journal.

References

Alcock, N. W., Blacker, N. C., Errington, W., Wallbridge, M. G. H. & Barker, J. (1994). *Acta Cryst.* C**50**, 456–458.
Allen, F. H., Kennard, O., Watson, D. G., Brammer, L., Orpen, A. G. & Taylor, R. (1987). *J. Chem. Soc. Perkin Trans.* 2, pp. S1–S19.
Altomare, A., Cascarano, G., Giacovazzo, C., Guagliardi, A., Burla, M. C., Polidori, G. & Camalli, M. (1994). *J. Appl. Cryst.* **27**, 435.
Arndt, U. W. & Wonacott A. J. (1997). Editors. *The Rotation Method in Crystallography*. Amsterdam: North-Holland.
Barker, J., Barr, D., Barnett, N. D. R., Clegg, W., Cragg-Hine, I., Davidson, M. G., Davies, P., Hodgson, S. M., Howard, J. A. K., Kilner, M., Lehmann, C. W., Lopez-Solera, I., Mulvey, R. E., Raithby, P. R. & Snaith, R. (1997). *J. Chem. Soc. Dalton. Trans.* pp. 951–955.
Barker, J. & Kilner, M. (1994). *Coord. Chem. Rev.* **133**, 219–300.
Barker, J. & Powell, H. R. (1996). *Acta Cryst.* C**52**, 2307–2309.
Cannon, J. R., White, A. H. & Willis, A. C. (1976). *J. Chem. Soc. Perkin Trans.* **2**, pp. 271–272.
Carruthers, J. R. & Watkin, D. J. (1979). *Acta Cryst.* A**35**, 698–699.
Dehnicke, K. (1990). *Chem. Ztg*, **114**, 295–304..
Edelmann, F. T. (1994). *Coord. Chem. Rev.* **137**, 403–481.
Larson, A. C. (1970). *Crystallographic Computing*, edited by F. R. Ahmed, S. R. Hall & C. P. Huber, pp. 291–294. Copenhagen: Munksgaard.

Molecular Structure Corporation (1993*a*). *R-Axis Processing Software*. MSC, 3200 Research Forest Drive, The Woodlands, TX 77381, USA.
Molecular Structure Corporation (1993*b*). *AFC-7R Processing Software*. MSC, 3200 Research Forest Drive, The Woodlands, TX 77381, USA.
Norrestam, R. (1984). *Acta Cryst.* C**40**, 297–299.
Thailambal, V. G., Pattabhi, V. & Guru Row, T. N. (1986). *Acta Cryst.* C**42**, 587–589.
Watkin, D. J., Prout, C. K., Carruthers, R. J. & Betteridge, P. (1996). *CRYSTALS*. Issue 10. Chemical Crystallography Laboratory, University of Oxford, England.
Watkin, D. J., Prout, C. K. & Pearce, L. J. (1996). *CAMERON*. Chemical Crystallography Laboratory, University of Oxford, England.

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myo-Inositol 1,3,5-Bicyclic Phosphate

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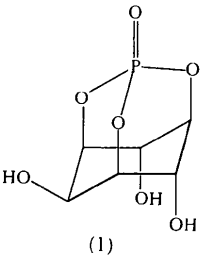
Abstract

In the crystal structure of the title compound (4,6,10-trihydroxy-2,8,9-trioxa-1-phosphatricyclo[3.3.1.$1^{3,7}$]decane *P*-oxide, $C_6H_9O_7P$), the cyclohexane ring is in a chair conformation. Two hydroxy substituents are in axial orientations and the third is equatorial. There is an intramolecular hydrogen bond involving the two axial hydroxy groups.

Comment

A number of phosphate esters of D-*myo*-inositol are of considerable importance inasmuch as they display profound biological activity in the cell as second messengers (Berridge, 1987). Our studies directed towards the synthesis of these phosphate esters (Gaffney & Reese, 1997) led us to the preparation of the title compound, (1), which has an adamantane-like structure of particular interest. The preparation of compound (1) has also been reported by Chinese workers (Yuan & Zhai, 1992). This is the first reported crystal structure of a cyclic phosphate of inositol; the structures of three other non-cyclic phosphates are available in the literature (Spiers *et al.*, 1995).



(1)

The cyclohexane ring adopts the expected chair conformation, with the 2-, 4- and 6-hydroxy substituents being axial, equatorial and axial, respectively. There is an intramolecular hydrogen bond between the O6 atom as donor and O5 as acceptor (Table 2). All three hydroxy groups, as well as the phosphate oxygen atom O1, participate in intermolecular hydrogen bonding in the crystal lattice. Atom O5 participates in an asymmetric bifurcated hydrogen bond with acceptors O6 and O7. The H atom donated from O5 is evidently closer to O7, as judged by the angles involved.

The bicyclic phosphate/cyclohexane moiety has approximate threefold symmetry. The three P—O single bonds, of average length 1.580 Å, are all equivalent within significant error, as are the O—P—O bond angles (average 104.5°). The P—O1 bond is of pure double-bond character (Allen *et al.*, 1987).

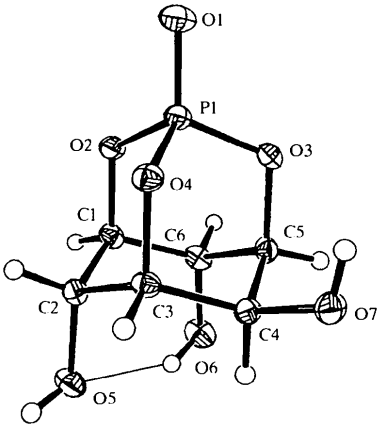


Fig. 1. View of the title structure. Displacement ellipsoids are shown at the 50% probability level. H atoms have been drawn as small circles of arbitrary radii.

Experimental

The title compound was crystallized by slow evaporation from a methanol/2-propanol solution.

Crystal data

$C_6H_9O_7P$ Mo $K\alpha$ radiation
M_r = 224.10 λ = 0.7107 Å