shown that subtle changes in the tetraamine ligand structure causes a change in the *cis* N—Co—N bond angle and a dramatic change in the reactivity of the cobalt complex. Knowledge of such detailed structure–reactivity relationship is valuable since it can give insights into the rational design of highly reactive cobalt complexes.

We thank Professor André L. Beauchamp for valuable comments. Support for this work from the National Sciences and Engineering Reseach Council of Canada is gratefully acknowledged.

References

BERNAL, I. (1985). *Inorg. Chim. Acta*, **96**, 99–110.
CHIN, J. & BANASZCZYK, M. (1989*a*). *J. Am. Chem. Soc.* **111**, 2724–2726.
CHIN, J. & BANASZCZYK, M. (1989*b*). *J. Am. Chem. Soc.* **111**, 4103–4105.
CHIN, J., BANASZCZYK, M., JUBIAN, V. & ZOU, X. (1989). *J. Am. Chem. Soc.* **111**, 186–190.
CHIN, J. & ZOU, X. (1988). *J. Am. Chem. Soc.* **110**, 223–225.
DERVAN, P. B. & MOSER, H. E. (1987). *Science*, **238**, 645–650.
GABE, E. J., LEE, F. L. & LE PAGE, Y. (1985). *The NRCVAX Crystal Structure System*. In *Crysallagraphic Computing* 3: *Data Collection, Structure Determination, Proteins and Databases*, edited by G. M. SHELDRICK, C. KRÜGER & R. GODDARD, pp. 167–174. Oxford: Clarendon Press.
IITAKA, Y., SHINA, M. & KIMURA, E. (1974). *Inorg. Chem.* **13**, 2886–2891.
International Tables for X-ray Crystallography (1974). Vol. IV. Birmingham: Kynoch Press. (Present distributor Kluwer Academic Publishers, Dordrecht.)
LARSON, A. C. (1967). *Acta Cryst.* **23**, 664–665.
LEOHLIN, J. H. & FLEISCHER, E. B. (1976). *Acta Cryst.* B**32**, 3063–3066.
SCHLEMPER, E. O., SEN GUPTA, P. K. & DASGUPTA, T. P. (1983). *Acta Cryst.* C**39**, 1012–1015.
ZACHARIASEN, W. H. (1963). *Acta Cryst.* **16**, 1139–1147.

Acta Cryst. (1990). C**46**, 1025–1027

Structure of 1-Methyl-1*H*-imidazole-5-ethanamine Dihydrochloride

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(*Received* 7 *June* 1989; *accepted* 30 *August* 1989)

Abstract. 4-(2-Ammonioethyl)-1-methyl-3H^+-imidazolium chloride, $C_6H_{13}N_3^{2+}.2Cl^-$, $M_r = 198{\cdot}10$, monoclinic, $P2_1/n$, $a = 4{\cdot}60\ (1)$, $b = 13{\cdot}57\ (1)$, $c = 15{\cdot}43\ (2)$ Å, $\beta = 98{\cdot}37\ (3)°$, $V = 952{\cdot}9$ Å^3, $Z = 4$, $D_x = 1{\cdot}381$, $D_m = 1{\cdot}38$ Mg m^{-3}, $F(000) = 416$, $T = 293$ K, Mo $K\alpha$ ($\lambda = 0{\cdot}71069$ Å), $\mu = 6{\cdot}29$ mm^{-1}, $R = 0{\cdot}030$ for 1484 reflections used in the refinement. Diprotonated methylhistamine molecules are hydrogen bonded through chloride ions. The ethylamine chain is in a *trans* conformation and the plane defined by three non-H atoms of the chain is perpendicular to the plane of the imidazole ring.

Introduction. Ganellin's works on conformation and activity of histamine and methylhistamines have shown that the *trans* conformation of the ethylamine chain dominates in aqueous solution for all protonated forms and that *N*-methylation has little influence on the chain conformation (Ganellin, 1973; Ganellin, Pepper, Port & Richards, 1973; Ganellin, Port & Richards, 1973). However, *pros*-methylation seems to be important for the orientation of the ethylamine chain. Thus we undertook this study as a continuation of the program on structures of histamines and their complexes (Główka, Gałdecki, Kazimierczak & Maśliński, 1980; Główka & Gilli, 1989).

Experimental. The crystals of the compound were recrystallized from 2-propanol. D_m by flotation. The intensity data to $\theta_{max} = 25°$ were obtained from a crystal of size $0{\cdot}44 \times 0{\cdot}38 \times 0{\cdot}15$ mm by $\omega/2\theta$ scan technique on a CAD-4 diffractometer. Cell constants from setting angles of 25 reflections with $9 < \theta < 15°$. 3 standard reflections, measured every 100 reflections; 2% variation. $-5 \le h \le 5$; $0 \le k \le 15$; $0 \le l \le 18$. From the 1860 reflections measured, 1637 had $I > 3\sigma(I)$. Absorption was neglected. The structure was solved by direct methods with *SHELXS*86 (Sheldrick, 1986) and H-atom positions were found from difference Fourier maps. The final agreement factors of 0·030 (R) and 0·041 (wR) were obtained after full-matrix least-squares refinement on F based on 1484 intensities with $F_o > 3\sigma(F_o)$ (non-H atoms with anisotropic and H atoms with isotropic thermal parameters). The final weighting scheme used was $w = k/[\sigma^2(F_o) + dF_o^2]$, where k and d were 0·382 and 0·0079, respectively. $(\Delta/\sigma)_{max} = 0{\cdot}08$. Max. and min.

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heights in final difference synthesis 0·11, −0·16 e Å^{-3}. The calculations were performed on a 1512 Amstrad PC microcomputer with *SHELX*76 (Sheldrick, 1976). Atomic scattering factors were those in *SHELX*76.

The same crystal structure solved earlier (Główka, 1982) has shown disordered position of Cl(2) atom. The population parameters for both positions of the Cl(2) atom refined independently during isotropic refinement procedure had the values of 0·45 for Cl(2*A*) and 0·55 for Cl(2*B*). The partially occupied sites were separated by only 0·223 (1) Å.

Discussion. Final atomic parameters and standard deviations are given in Table 1,* while the calculated distances and angles for non-H atoms are collected in Table 2. Fig. 1 shows the overall conformation of the *pros*-methylhistamine dication and the numbering system used in this study. The crystal structure of the title compound comprises *pros*-methylhistamine dications hydrogen bonded through chloride anions (Table 2). The molecular geometry of the *pros*-methylhistamine ion in this structure is very similar to that predicted by Ganellin, Pepper, Port & Richards (1973) for the histamine dication by quantum mechanics and to that found in the histamine diphosphate monohydrate (Veidis, Palenik, Schaffrin & Trotter, 1969). The angles θ_1 and θ_2, where θ_1 [C(4)—C(5)—C(6)—C(7)] and θ_2 [C(5)—C(6)—C(7)—N(7)] (Ganellin, Pepper, Port & Richards, 1973), are 94·2 (2) and 174·3 (1)°, respectively, close to 90 and 180° for the ideal *trans* conformation of the ethylamine chain. This and other X-ray studies on diprotonated histamine compounds suggest that the packing is the main factor affecting the ethylamine chain conformation in the crystalline state, as the angle between the imidazole ring and the plane defined by the three non-H atoms of the side chain varies from 4·4° in histamine sulfate (Yamane, Ashida & Kakudo, 1973), through 7·0° in the tetrachlorocobaltate (Bonnet & Jeannin, 1972), 30° in the dibromide (Decou, 1964), 78·1° in the *tele*-methylhistamine dihydrochloride complex with copper chloride (Główka & Gilli, 1989), and 82·5° in histamine diphosphate monohydrate (Veidis, Palenik, Schaffrin & Trotter, 1969) to 96·5° in this study. Each chloride ion takes part in two hydrogen bonds with Cl···H(N) distances between 2·2 and 2·4 Å, and with H···Cl···H angles between 89 and 100°.

* Lists of structure factors, anisotropic thermal parameters and H-atom parameters have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 52588 (13 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Table 1. *Atomic positional parameters* (× 10^5 *for* Cl *and* × 10^4 *for other non*-H *atoms*) *and equivalent isotropic thermal parameters*

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

	x	*y*	*z*	B_{eq} (Å^2)
Cl(1)	17301 (9)	41329 (3)	9872 (3)	2·84 (1)
Cl(2)	37694 (10)	33190 (3)	45107 (3)	3·25 (2)
N(1)	6289 (3)	446 (1)	1803 (1)	2·55 (3)
N(3)	3762 (4)	1584 (1)	1083 (1)	3·34 (5)
N(7)	10828 (4)	1248 (1)	4541 (1)	2·61 (4)
C(1)	7276 (6)	−546 (1)	2090 (2)	3·57 (6)
C(2)	4117 (4)	625 (2)	1151 (1)	3·03 (4)
C(4)	5755 (4)	2041 (1)	1693 (1)	3·20 (5)
C(5)	7381 (4)	1336 (1)	2157 (1)	2·41 (4)
C(6)	9725 (4)	1406 (1)	2922 (1)	2·56 (5)
C(7)	8478 (4)	1279 (1)	3775 (1)	2·47 (4)

Table 2. *Bond lengths* (Å) *and angles* (°) *and hydrogen-bond geometry*

N(1)—C(1)	1·468 (2)	C(1)—N(1)—C(2)	124·0 (1)
N(1)—C(2)	1·333 (2)	C(1)—N(1)—C(5)	126·9 (2)
N(1)—C(5)	1·389 (2)	C(2)—N(1)—C(5)	109·1 (1)
N(3)—C(2)	1·314 (2)	C(2)—N(3)—C(4)	109·4 (2)
N(3)—C(4)	1·364 (2)	N(1)—C(2)—N(3)	108·2 (1)
N(7)—C(7)	1·482 (2)	N(3)—C(4)—C(5)	107·9 (1)
C(4)—C(5)	1·353 (2)	N(1)—C(5)—C(4)	105·4 (1)
C(5)—C(6)	1·481 (2)	N(1)—C(5)—C(6)	123·3 (1)
C(6)—C(7)	1·520 (2)	C(4)—C(5)—C(6)	131·2 (1)
		C(5)—C(6)—C(7)	111·1 (2)
		C(6)—C(7)—N(7)	111·7 (2)

N—H···Cl	N···Cl	N—H	H···Cl	N—H···Cl
N(7)—H(1)···Cl(1)^{i}	3·166 (2)	0·90 (3)	2·32 (3)	158 (1)
N(7)—H(2)···Cl(2)^{ii}	3·122 (2)	0·80 (2)	2·34 (2)	169 (1)
N(7)—H(3)···Cl(1)^{iii}	3·229 (2)	0·91 (3)	2·41 (3)	150 (1)
N(3)—H···Cl(2)^{iv}	3·091 (2)	0·82 (3)	2·35 (3)	150 (1)

Symmetry codes: (i) 0·5 + *x*, 0·5 − *y*, 0·5 + *z*; (ii) 1 + *x*, *y*, *z*; (iii) 1·5 − *x*, *y* − 0·5, 0·5 − *z*; (iv) *x* − 0·5, 0·5 − *y*, *z* − 0·5.

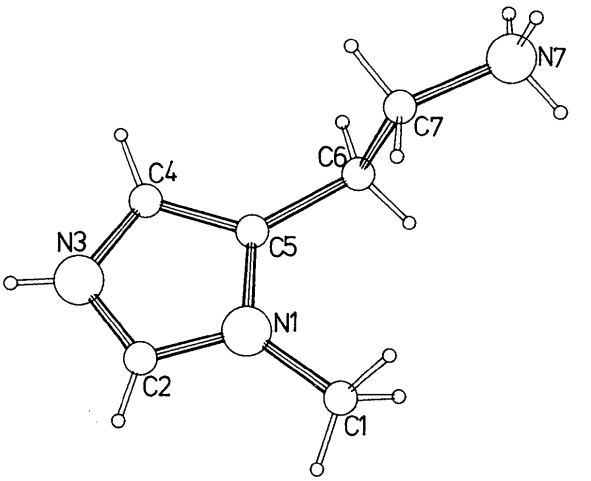

Fig. 1. A view of the molecule and the atomic numbering scheme.

The authors wish to thank Dr C. Robin Ganellin for the gift of the crystals and the Polish Ministry of National Education (project RP.II.10) for financial support.

References

BONNET, J. J. & JEANNIN, Y. (1972). *Acta Cryst.* B**28**, 1079–1085.
DECOU, D. F. JR (1964). Dissertation No. 64-9987. Univ. Microfilms Inc., Ann Arbor, Michigan, USA.
GANELLIN, C. R. (1973). *J. Med. Chem.* **16**, 620–623.
GANELLIN, C. R., PEPPER, E. S., PORT, G. N. J. & RICHARDS, W. G. (1973). *J. Med. Chem.* **16**, 610–616.
GANELLIN, C. R., PORT, G. N. J. & RICHARDS, W. G. (1973). *J. Med. Chem.* **16**, 616–620.
GŁÓWKA, M. L. (1982). Unpublished results.
GŁÓWKA, M. L., GAŁDECKI, Z., KAZIMIERCZAK, W. & MAŚLIŃSKI, C. (1980). *Acta Cryst.* B**36**, 2148–2150.
GŁÓWKA, M. L. & GILLI, G. (1989). *Acta Cryst.* C**45**, 408–410.
SHELDRICK, G. M. (1976). *SHELX*76. A program for crystal structure determination. Univ. of Cambridge, England.
SHELDRICK, G. M. (1986). *SHELXS*86. Program for crystal structure solution. Univ. Göttingen, Federal Republic of Germany.
VEIDIS, M. V., PALENIK, G. J., SCHAFFRIN, R. & TROTTER, J. (1969). *J. Chem. Soc. A*, pp. 2659–2666.
YAMANE, T., ASHIDA, T. & KAKUDO, M. (1973). *Acta Cryst.* B**29**, 2884–2891.

Acta Cryst. (1990). C**46**, 1027–1029

Structure of 1-Morpholinobenzo[*c*]cinnoline

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(*Received* 5 *June* 1989; *accepted* 29 *August* 1989)

Abstract. $C_{16}H_{15}N_3O$, $M_r = 265{\cdot}316$, monoclinic, $P2_1/c$, $a = 10{\cdot}572\ (5)$, $b = 11{\cdot}954\ (6)$, $c = 11{\cdot}222\ (2)$ Å, $\beta = 108{\cdot}13\ (2)°$, $V = 1347{\cdot}8\ (1{\cdot}4)$ Å^3, $Z = 4$, $D_m = 1{\cdot}30$, $D_x = 1{\cdot}307$ g cm^{-3}, λ(Cu $K\alpha$) = 1·54180 Å, $\mu = 6{\cdot}373$ cm^{-1}, $F(000) = 560$, $T = 293$ K, $R = 0{\cdot}046$ for 2103 observed reflections. The two benzenoid rings are each planar but twisted with respect to each other with a torsion angle of 11·7°; in benzo[*c*]cinnoline the corresponding torsion angle is 2·5°.

Introduction. Benzo[*c*]cinnoline is known to be a mutagen (Leary, Lafleur, Liber & Blemann, 1983), and some of its derivatives are known to have antirheumatic (Matter, 1957; Erlenmeyer, 1958), herbicidal (Entwistle, Terence & Barton, 1981) and carcinogenic activity (Ashby, Styles & Paton, 1980). The structures of benzo[*c*]cinnoline (van der Meer, 1972) and octachlorobenzo[*c*]cinnoline (King, MacBride, Muir & Wright, 1983) have been described, as have those of benzo[*c*]cinnoline complexes with bis(tricarbonyliron) (Doedens, 1970) and copper(I) benzoato (Toth, Floriani, Chiesi-Villa & Guastini, 1987).

As far as we know, there are no reports on the structures of benzo[*c*]cinnolines substituted with alkyl, alkoxy, aminoalkyl or dialkylamino groups. The present paper is the first of a series devoted to structural studies on related pyrrolidino-, piperidino- and morpholinobenzo[*c*]cinnolines.

A structure determination of the title compound, which contains a morpholino group, was undertaken to permit a comparison of its structure with those of previously reported benzo[*c*]cinnolines to be made.

Experimental. 1-Bromobenzo[*c*]cinnoline was prepared by bromination of benzo[*c*]cinnoline (Barton & Lapham, 1979) obtained by reductive cyclization of 2,2′-dinitrobiphenyl with hydrazine hydrate using palladium on carbon as a catalyst. 1-Morpholinobenzo[*c*]cinnoline was synthesized from the reaction of 1 mmol 1-bromobenzo[*c*]cinnoline in 15 ml morpholine containing 5 ml dimethyl sulfoxide by refluxing for 96 h. After purification by column chromatography using silica gel and dichloromethane–diethyl ether, the product was recrystallized from petroleum ether. In about 3 d yellow prismatic crystals were obtained from *n*-hexane–diethyl ether. Experimental data, the method used to solve the structure and other related parameters and procedures are given in Table 1. Non-H atoms were included with anisotropic thermal parameters, while H atoms were located on difference syntheses and refined isotropically.

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0108-2701/90/061027-03$03.00