$\Delta = (|F_{hkl}| - |F_{\overline{hkl}}|)/(|F_{hkl}| + |F_{\overline{hkl}}|)$ for the correct absolute configuration. It can be seen from Table 1 that the signs of Δ_c and Δ_o are the same for 27 reflections but there are three contradictions in the $h00$ reflections. These three are denoted by asterisks in Table 1. Final R was 0·050 (wR = 0·055) for all observed reflections. Max. shift/e.s.d. = 0·3 in x of H(C18). Max. and min. $\Delta\rho = 0\cdot3$ and $-0\cdot2$ e Å^{-3}, respectively. IBM-3083 computer. Final atomic coordinates are listed in Table 2, and bond lengths and angles in Table 3.* A view of the molecule is given in Fig. 1.

Discussion. The X-ray analysis confirms the stereochemistry given by Gosset-Garnier, Le Men & Janot (1961, 1965). The B ring [N(1), C(2), C(7), C(8) and C(13)] has a chair form with asymmetry parameter $\Delta C_m^2 = 0\cdot5°$, so the C(2) atom is out of the plane defined by the remaining four atoms. These four atoms are in the same plane as the benzene A ring. The D ring [C(2), C(3), N(4), C(5), C(6) and C(7)] has a chair form with asymmetry parameter $\Delta C_m^3 = 6\cdot1°$. The B and D rings are *trans*-fused. The C ring [C(5), C(6), C(7), C(16) and C(17)] has a skew-envelope form with asymmetry parameter $\Delta C_m^6 = 15\cdot9°$, while the two remaining six-membered rings [E: C(3), N(4), C(5), C(14), C(15) and C(16); F: C(3), N(4), C(14), C(15), C(20) and C(21)] have a skew-sofa form with asymmetry parameters $\Delta C_2^{4-21} = 28\cdot7$, $\Delta C_2^3 = 19\cdot7$, $\Delta C_2^5 = 20\cdot6$ and $\Delta C_2^{3-4} = 25\cdot2°$. The stress between the C(15)—C(16) atoms produces a lengthening of this bond [1·566(8)] and C(16)—C(17) [1·560 (8) Å]; the smal-

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* 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 44079 (11 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

lest angles are C(7)—C(6)—C(5) [98·9 (5)], C(8)—C(7)—C(2) [99·9 (4)] and N(4)—C(3)—C(2) [105·9 (5)°] and the largest C(8)—C(13)—N(1) [110·6 (5)] and C(7)—C(2)—C(3) [114·9 (4)°]. The values of C(8)—C(7)—C(2) and C(8)—C(13)—N(1) are 104·3 (3) and 109·6 (3)° in oxymetavincadifformine (Rodier, Ceolin, Croquelois, Kunesch & Poisson, 1978); 106(1)° in vincamine 2-oxoglutarate (Fayos & Martinez-Ripoli, 1981) and 107·3 (2) and 107·3 (2)° in vincamone (Chiesi-Villa, Gaetani Manfredotti, Guastini, Chiari & Viterbo (1973). The three substituent radicals occupy *exo* sites.

References

CHIESI-VILLA, A., GAETANI MANFREDOTTI, A., GUASTINI, C., CHIARI, G. & VITERBO, D. (1973). *Cryst. Struct. Commun.* **2**, 599–603.
FAYOS, J. & MARTINEZ-RIPOLL, M. (1981). *Acta Cryst.* B**37**, 760–763.
GOSSET-GARNIER, J., LE MEN, J. & JANOT, M. M. (1961). *Bull. Soc. Chim. Fr.* pp. 1033–1035.
GOSSET-GARNIER, J., LE MEN, J. & JANOT, M. M. (1965). *Bull. Soc. Chim. Fr.* pp. 676–678.
International Tables for X-ray Crystallography (1974). Vol. IV, pp. 99–100, 149. Birmingham: Kynoch Press. (Present distributor D. Reidel, Dordrecht.)
MAIN, P., FISKE, S. E., HULL, S. L., LESSINGER, L., GERMAIN, G., DECLERCQ, J.-P. & WOOLFSON, M. M. (1980). *MULTAN*80. *A System of Computer Programs for the Automatic Solution of Crystal Structures from X-ray Diffraction Data.* Univs. of York, England, and Louvain, Belgium.
NOVIKOVA, ZH, N., GOTSIRIDZE, I. A. & ABULADZE, G. V. (1984). *Izv. Akad. Nauk. Gruz.* **10**, 54–61.
RODIER, N., CEOLIN, R., CROQUELOIS, G., KUNESCH, N. & POISSON, J. (1978). *Acta Cryst.* B**34**, 3682–3685.
SHELDRICK, G. M. (1976). *SHELX.* Program for crystal structure determination. Univ. of Cambridge, England.
TAGIEVA, Z. D., BABAEV, N. A. & ALIEV, A. M. (1979). *Azerb. Med. Zh.* **56**, 11–26.

Structure of *N*-Methylaminoacetonitrile Hydrochloride

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Abstract. (Cyanomethyl)methylammonium chloride, $C_3H_7N_2^+.Cl^-$, $M_r = 106\cdot56$, orthorhombic, *Pbca*, $a = 11\cdot518$ (1), $b = 10\cdot201$ (1), $c = 9\cdot166$ (1) Å, $V = 1076\cdot9$ (2) Å^3, $Z = 8$, $D_x = 1\cdot314$ Mg m^{-3}, Mo $K\alpha_1$, $\lambda = 0\cdot70930$ Å, $\mu = 0\cdot56$ mm^{-1}, $F(000) = 448$, $T = 295$ K, $R = 0\cdot031$ for 1175 unique observed reflections with $I \geq 3\sigma(I)$. The structure consists of centrosymmetric hydrogen-bonded dimers in which two NCCH$_2$-NH$_2$CH$_3^+$ cations are bridged by two Cl$^-$ anions *via* N—H···Cl hydrogen bonds, Cl···H = 2·22 (2) and 2·23 (2) Å. The cation adopts a *gauche* conformation with respect to the central C(sp^3)—N(sp^3) bond.

Table 1. *Final positional (fractional* $\times 10^5$, H $\times 10^4$*) and isotropic or equivalent isotropic thermal parameters* ($\text{Å}^2 \times 10^3$) *with estimated standard deviations in parentheses*

$U_{eq} = \frac{1}{3}$(trace of diagonalized U).

	x	y	z	U_{eq}/U_{iso}
Cl	10559 (3)	68443 (4)	−3015 (5)	39
N(1)	15412 (16)	12407 (16)	23641 (21)	54
N(2)	9821 (11)	44926 (12)	18671 (15)	31
C(1)	17007 (15)	22632 (16)	19185 (19)	37
C(2)	19015 (15)	35860 (16)	13613 (20)	37
C(3)	9327 (17)	46387 (20)	34698 (22)	44
H(N2a)	1085 (16)	5320 (20)	1424 (27)	48 (6)
H(N2b)	302 (21)	4266 (21)	1523 (26)	52 (6)
H(2a)	1943 (21)	3565 (24)	338 (28)	65 (7)
H(2b)	2638 (18)	3916 (21)	1692 (21)	46 (6)
H(3a)	645 (26)	5501 (30)	3800 (39)	104 (10)
H(3b)	465 (20)	3852 (25)	3825 (26)	66 (6)
H(3c)	1756 (26)	4684 (33)	3878 (37)	113 (11)

Table 2. *Bond lengths* (Å) *and angles* (°) *with estimated standard deviations in parentheses*

N(1)–C(1)	1·135 (2)	N(2)–C(3)	1·478 (2)
N(2)–C(2)	1·480 (2)	C(1)–C(2)	1·461 (2)
N(2)–H(N2a)	0·94 (2)	C(3)–H(3a)	0·99 (3)
N(2)–H(N2b)	0·88 (2)	C(3)–H(3b)	1·02 (3)
C(2)–H(2a)	0·94 (3)	C(3)–H(3c)	1·02 (3)
C(2)–H(2b)	0·96 (2)		

C(2)–N(2)–C(3)	113·70 (13)	N(2)–C(2)–C(1)	110·74 (13)
N(1)–C(1)–C(2)	179·3 (2)		
C(2)–N(2)–H(N2a)	109·5 (13)	C(1)–C(2)–H(2b)	110·7 (13)
C(2)–N(2)–H(N2b)	111·2 (15)	H(2a)–C(2)–H(2b)	106 (2)
C(3)–N(2)–H(N2a)	110·0 (15)	N(2)–C(3)–H(3a)	114 (2)
C(3)–N(2)–H(N2b)	110·5 (15)	N(2)–C(3)–H(3b)	104·9 (13)
H(N2a)–N(2)–H(N2b)	101 (2)	N(2)–C(3)–H(3c)	109 (2)
N(2)–C(2)–H(2a)	111·3 (15)	H(3a)–C(3)–H(3b)	115 (2)
N(2)–C(2)–H(2b)	108·3 (13)	H(3a)–C(3)–H(3c)	99 (2)
C(1)–C(2)–H(2a)	109·6 (15)	H(3b)–C(3)–H(3c)	114 (2)

Introduction. The crystal structure of the title compound has been determined to assist in the interpretation of solid-state NMR spectra.

Experimental. Colourless crystals, dimensions 0·38 × 0·40 × 0·40 mm, faces {1,0,0}, {0,0,1}, {1,1,1}. Enraf–Nonius CAD-4F diffractometer, lattice parameters from 25 reflections with $\theta = 20$–26°. Intensities for $\theta \le 32\cdot5°$, *hkl*: 0 to 17, 0 to 15, 0 to 13, ω–2θ scan, ω scan width $(0\cdot60 + 0\cdot35$ tan$\theta)°$ at $1\cdot2$–10° min^{-1}, extended 25% on each side for background measurement, three standard reflections (no decay). Lp and absorption corrections (numerical integration), transmission factors 0·836–0·854, 1935 unique reflections measured, 1175 with $I \ge 3\sigma(I)$, where $\sigma^2(I) = S + 4(B_1 + B_2) + (0\cdot04S)^2$, S = scan, B_1 and B_2 = background counts. Structure by Patterson and Fourier methods, refined by full-matrix least squares on F, H atoms refined with isotropic thermal parameters, $w \approx 1/\sigma^2(F)$, scattering factors and anomalous-scattering corrections for Cl from *International Tables for X-ray Crystallography* (1974), locally written or

locally modified versions of standard computer programs, final $R = 0\cdot031$, $wR = 0\cdot040$ for 1175 reflections, $S = 1\cdot71$, 84 parameters, isotropic type I extinction, $g = 4\cdot8$ (8) $\times10^4$, $R = 0\cdot068$ for all 1935 reflections, $\Delta/\sigma = 0\cdot01$ (mean), 0·07 (maximum), maximum final difference density −0·4 to +0·2 e Å^{-3}.

Discussion. Final positional and isotropic or equivalent isotropic thermal parameters are given in Table 1, and geometrical data appear in Table 2.* A view of the asymmetric unit is shown in Fig. 1, and the packing arrangement is shown in Fig. 2.

The crystal structure consists of centrosymmetric hydrogen-bonded dimers in which two NCCH$_2$NH$_2$-CH$_3^+$ cations are bridged by two Cl$^-$ anions *via* N–H···Cl hydrogen bonds [N(2)–H(N2a)···Cl and N(2)–H(N2b)···Cl ($-x$, $1-y$, $-z$), H···Cl = 2·22 (2) and 2·23 (2), N···Cl = 3·117 (1) and 3·071 (1) Å, N–H···Cl = 159 (2) and 160 (2)°, respectively, H··· Cl···H = 90·7 (8)°], to form central eight-membered N$_2$H$_4$Cl$_2$ rings. Interdimer distances correspond to van der Waals contacts; the most significant are Cl···C(1) ($\frac{1}{2}-x$, $\frac{1}{2}+y$, z) = 3·317 (2), Cl···C(2) ($\frac{1}{2}-x$, $\frac{1}{2}+y$,
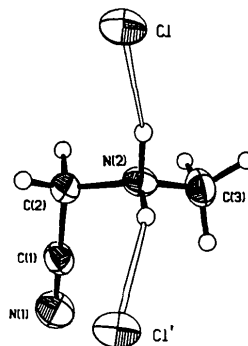
Fig. 1. View of the title compound with atom numbering.

Fig. 2. Packing diagram; broken lines are N–H···Cl hydrogen bonds.

$z) = 3 \cdot 319$ (2), Cl$\cdots$H(3a) $(x, \frac{3}{2}-y, z-\frac{1}{2}) = 2 \cdot 87$ (3) and N(1)$\cdots$H(2b) $(\frac{1}{2}-x, y-\frac{1}{2}, z) = 2 \cdot 63$ (2) Å.

Bond lengths and angles in the cation (Table 2) are generally as expected. The cation adopts a *gauche* conformation with respect to the N(2)–C(2) bond, C(1)–C(2)–N(2)–C(3) = 61·9 (2)°. The C(2)–N(2)–C(3) bond angle [113·7 (1)°] is larger than the tetrahedral value as a result of steric interaction between the cyano and methyl groups [C(1)$\cdots$C(3) = 2·946 (2), C(1)$\cdots$H(3b) = 2·78 (2) Å].

References

International Tables for X-ray Crystallography (1974). Vol. IV, pp. 99–102 and 149. Birmingham: Kynoch Press. (Present distributor D. Reidel, Dordrecht.)

Structures en Séries Mutagène:
les Nitro-2 et Méthyl-3 Nitro-2 Tétrahydro-6,7,8,9 Naphto[1,2-*b*]furannes

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Abstract. (I) 2-Nitro-6,7,8,9-tetrahydronaphtho[1,2-*b*]-furan, $C_{12}H_{11}NO_3$, $M_r = 217 \cdot 2$, monoclinic, $P2_1/c$, $a = 11 \cdot 042$ (1), $b = 9 \cdot 149$ (2), $c = 12 \cdot 056$ (2) Å, $\beta = 123 \cdot 08$ (1)°, $V = 1020 \cdot 7$ Å^3, $Z = 4$, $D_m = 1 \cdot 40$, $D_x = 1 \cdot 413$ Mg m^{-3}, λ(Cu $K\alpha$) = 1·54178 Å, $\mu = 0 \cdot 861$ mm^{-1}, $F(000) = 456$, $T = 298$ K, $R = 0 \cdot 041$ for 1573 observed reflexions. (II) 3-Methyl-2-nitro-6,7,8,9-tetrahydronaphtho[1,2-*b*]furan, $C_{13}H_{13}NO_3$, $M_r = 231 \cdot 2$, orthorhombic, $Pca2_1$, $a = 21 \cdot 386$ (2), $b = 6 \cdot 845$ (5), $c = 7 \cdot 678$ (2) Å, $V = 1123 \cdot 9$ Å^3, $Z = 4$, $D_m = 1 \cdot 36$, $D_x = 1 \cdot 367$ Mg m^{-3}, λ(Cu $K\alpha$) = 1·54178 Å, $\mu = 0 \cdot 815$ mm^{-1}, $F(000) = 488$, $T = 298$ K, $R = 0 \cdot 037$ for 737 observed reflexions. These compounds are mutagenic agents. Partial atomic charges and molecular electrostatic potentials were calculated using semi-quantitative methods. The increase of mutagenic potency on introduction of a methyl group in position 3 of the 2-nitrofuran ring might be related to the increase of electrostatic potential at the NO_2 position.

Introduction. Un nombre important de molécules, correspondant aux nitro-2 naphto[2,1-*b*]-, [2,3-*b*]- et [1,2-*b*]furannes respectivement, a été synthétisé (Royer & Buisson, 1980; Royer, 1986; Royer & Buisson, 1986). Divers substituants ont été introduits, notamment en position β du cycle furanne et en diverses positions sur l'homocycle extérieur. Plusieurs analogues hydrogénés sur l'homocycle extérieur ont été également synthétisés. C'est le cas des deux molécules faisant l'objet de cette étude.

Certaines de ces molécules présentent des propriétés bactéricide et protozoocide remarquables (Cavier,

Buisson, Lemoine & Royer, 1981). Elles sont également douées d'un pouvoir mutagène plus ou moins élevé suivant le type de nitro-2 naphtofuranne, la nature et la position des substituants, l'hydrogénation de l'homocycle extérieur (Arnaise *et al.*, 1986). Le méthoxy-7 méthyl-3 nitro-2 naphto[2,1-*b*]furanne est le composé le plus mutagène de cette série, et l'un des plus puissants mutagènes connus. Son pouvoir mutagène déterminé au moyen du Mutatest, est de $1,9 \times 10^6$ mutants nmol^{-1}.

Dans le but d'établir des relations structure–activité, nous avons entrepris l'étude de quelques molécules de cette série correspondant à un large domaine du pouvoir mutagène (Bravic, Bideau & Courseille, 1982; Bideau, Bravic, Cotrait & Courseille, 1983, 1984; Bideau, Bravic & Cotrait, 1984, 1985; Ajana, Bideau & Cotrait, 1987).

Les analogues hydrogénés ont une moindre activité bactéricide et génotoxique. Mais l'effet défavorable de l'hydrogénation de l'homocycle extérieur peut être compensé par une méthylation en position β de l'hétérocycle (Einhorn, Demerseman, René, Royer & Cavier, 1983).

Les nitro-2 et méthyl-3 nitro-2 tétrahydro-6,7,8,9 naphto[1,2-*b*]furannes, respectivement désignés ici par composé (I) et (II), ont un pouvoir mutagène respectif (Mutatest) de $8,9 \times 10^2$ et $2,4 \times 10^4$ mutants nmol^{-1}.

Partie expérimentale. Les composés (I) et (II) ont été synthétisés par l'équipe du Professeur Royer (Institut Curie, Paris); cristaux jaunes (benzène–acétone); $0,40 \times 0,38 \times 0,16$ mm (I) et $0,50 \times 0,40 \times 0,20$ mm