Crystal structure of 5-chloro-2-methylanilinium chloride hydrate, $(ClC_7H_6NH_3)Cl \cdot H_2O$

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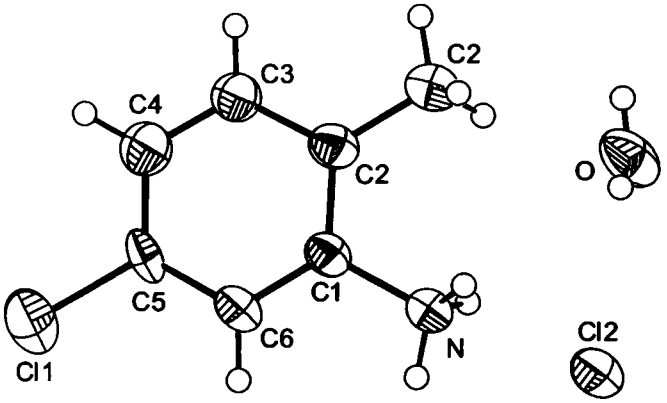
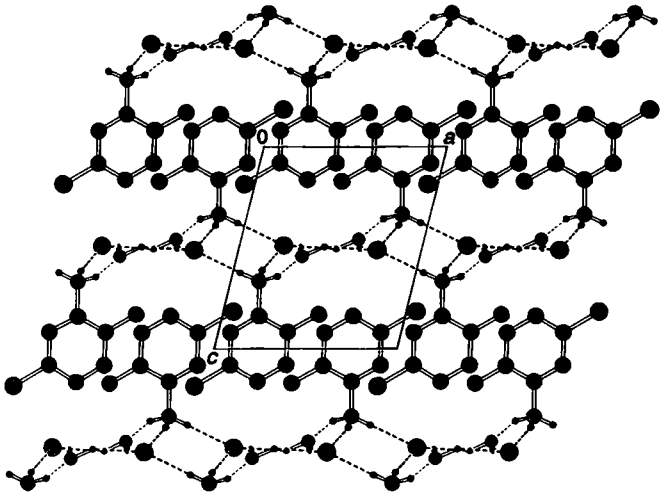
Received May 18, 2004, accepted in revised form and available on-line January 14, 2005; CCDC no. 1267/1340





Abstract

$C_7H_{11}Cl_2NO$, monoclinic, $P12_11$ (no. 4), $a = 7.582(2)$ Å, $b = 7.397(6)$ Å, $c = 8.696(1)$ Å, $\beta = 103.81(1)°$, $V = 473.6$ Å^3, $Z = 2$, $R_{gt}(F) = 0.034$, $wR_{ref}(F) = 0.038$, $T = 296$ K.

Discussion

In the atomic arrangement of 5-chloro-2-methylanilinium chloride monohydrate (figure, top), helical chains built up by chloride anions and water molecules are spreading along the polar *b*-axis. These chains are interconnected to layers parallel to the *a*,*b*-plane by a set of N–H···O and N–H···Cl bonds generated by the organic entities (figure, bottom). These latters reflect favorably the interaction between the organic cations and lead to a non-centrosymmetric compound, which could be a promoter material for quadratic non-linear optical applications.

The atoms C1, C2, C3, C4, C5 and C6 of the phenyl ring have a good coplanarity and they form a conjugated plane with average deviation of 0.0029 Å, the bond lengths of C1—C2, C1—C6, C2—C3, C3—C4, C4—C5 and C5—C6 are 1.392(2) Å, 1.385(2) Å, 1.391(2) Å, 1.380(2) Å, 1.379(2) Å, and 1.389(2) Å, respectively, which are between single bond and double bond and agree with that in benzene [1]. Furthermore, the distances C2—C7, C1—N of 1.500(2) Å and 1.467(1) Å, respectively, clearly indicate two single bonds. The bond length of C5—Cl1 is 1.733(1) Å and agrees also with data of another chloride [2]. The correctness of the absolute structure was confirmed by the Flack parameter [3] value $x = -0.07(7)$.

Table 1. Data collection and handling.

Crystal:	colorless prism, size 0.40 × 0.45 × 0.85 mm
Wavelength:	Ag K_α radiation (0.5608 Å)
μ:	3.26 cm^{-1}
Diffractometer, scan mode:	MACH3, $\omega/2\theta$
$2\theta_{max}$:	49.92°
$N(hkl)_{measured}$, $N(hkl)_{unique}$:	6926, 1889
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 3\,\sigma(I_{obs})$, 1546
$N(param)_{refined}$:	100
Programs:	SIR92 [4], SHELXL-97 [5]

Table 2. Atomic coordinates and displacement parameters (in Å^2).

Atom	Site	x	y	z	U_{iso}
H(1)	2*a*	0.0412	0.4851	0.1320	0.059
H(2)	2*a*	0.1675	0.3395	0.1240	0.059
H(3)	2*a*	0.2294	0.5261	0.1345	0.059
H(7)	2*a*	0.5339	0.3477	0.6358	0.057
H(8)	2*a*	0.3123	0.3899	0.7725	0.059
H(9)	2*a*	–0.0504	0.4937	0.3624	0.048
H(4)	2*a*	0.4851	0.2833	0.2425	0.074
H(5)	2*a*	0.6324	0.3128	0.4009	0.074
H(6)	2*a*	0.5596	0.4780	0.2912	0.074
H(10)	2*a*	–0.4659	0.1646	0.9596	0.098
H(11)	2*a*	–0.3295	0.2956	0.9651	0.098

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Table 3. Atomic coordinates and displacement parameters (in $Å^2$).

Atom	Site	x	y	z	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
Cl(1)	2*a*	–0.05594(6)	0.47571(6)	0.68508(5)	0.0624(2)	0.0695(2)	0.0656(2)	0.0089(2)	0.0392(2)	0.0014(2)
N	2*a*	0.1556(1)	0.4476(2)	0.1652(1)	0.0329(4)	0.0440(5)	0.0407(4)	0.0035(4)	0.0098(3)	0.0006(4)
C(1)	2*a*	0.2028(2)	0.4349(2)	0.3385(1)	0.0321(5)	0.0335(5)	0.0402(5)	0.0016(4)	0.0114(4)	0.0017(4)
C(2)	2*a*	0.3805(2)	0.3923(2)	0.4162(1)	0.0325(5)	0.0375(5)	0.0441(6)	0.0030(4)	0.0113(4)	0.0041(4)
C(3)	2*a*	0.4166(2)	0.3764(2)	0.5801(2)	0.0388(6)	0.0577(8)	0.0466(6)	0.0072(6)	0.0095(5)	0.0108(6)
C(4)	2*a*	0.2844(2)	0.4017(2)	0.6629(2)	0.0514(7)	0.0581(8)	0.0407(6)	0.0032(6)	0.0150(5)	0.0075(5)
C(5)	2*a*	0.1103(2)	0.4447(2)	0.5810(2)	0.0460(6)	0.0407(6)	0.0488(6)	0.0023(5)	0.0233(5)	0.0020(5)
C(6)	2*a*	0.0667(2)	0.4632(2)	0.4174(1)	0.0348(5)	0.0404(6)	0.0480(6)	0.0039(4)	0.0151(4)	0.0015(5)
C(7)	2*a*	0.5276(2)	0.3641(2)	0.3300(2)	0.0344(5)	0.0631(9)	0.0522(7)	0.0092(6)	0.0146(5)	0.0045(6)
O	2*a*	–0.3557(2)	0.1837(2)	0.9586(2)	0.0484(6)	0.0576(6)	0.0967(8)	–0.0070(5)	0.0323(6)	–0.0236(6)
Cl(2)	2*a*	–0.24240(4)	0.59144(5)	0.01144(3)	0.0379(1)	0.0451(1)	0.0547(2)	0.0052(1)	0.0168(1)	0.0062(1)

Acknowledgment. We thank the Secretary of State for Scientific Research and Technology for financial support.

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