Crystal structure of *o*-cyanoaniline hydrochloride, $(NCC_6H_4NH_3)Cl$

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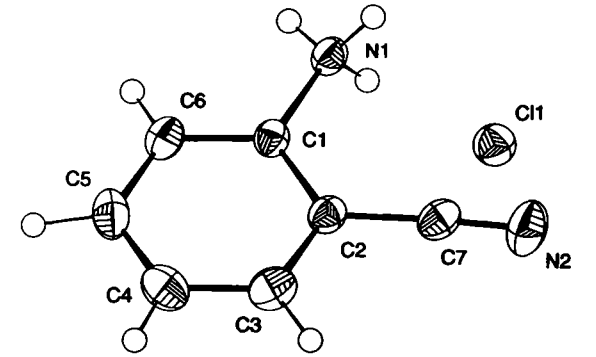

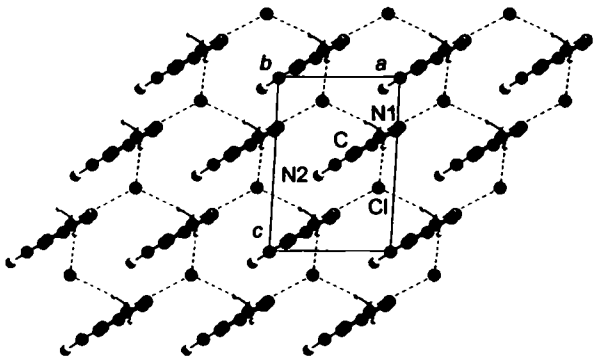

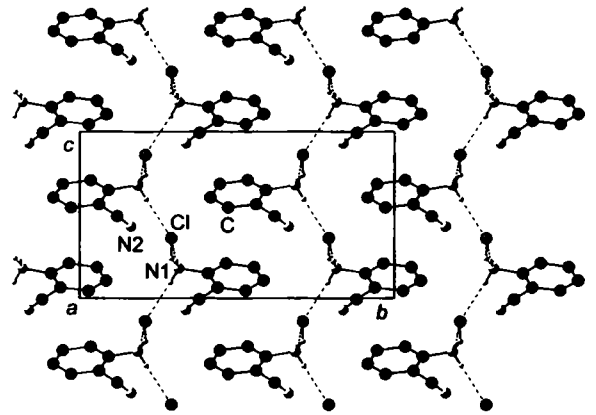

Abstract

$C_7H_7ClN_2$, monoclinic, $C1c1$ (no. 9), $a = 5.658(5)$ Å, $b = 15.406(9)$ Å, $c = 8.234(8)$ Å, $\beta = 93.18(9)°$, $V = 716.6$ Å^3, $Z = 4$, $R_{gt}(F) = 0.020$, $wR_{ref}(F^2) = 0.052$, $T = 293$ K.

Source of material

Crystals of *o*-cyanoaniline hydrochloride (synonym 2-cyanoanilinium chloride) were prepared by dissolving of 5 mmol of purified 2-cyanoaniline in 10 mL of an ethanol-water solution (*v*/*v* 1:1) containing 5 mmol HCl at room temperature. After some days of evaporation, crystals of suitable dimensions for X-ray analysis appeared in the solution.

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Discussion

The crystal structure of the title compound is built up by a network of intermolecular hydrogen bonds between the constituents (figure, top), with a chloride anion acting as a triple acceptor as in other compounds [1,2] (figure, middle). The (N–)H···Cl distances varying between 2.28 Å and 2.45 Å are smaller than the sum of the Van der Waals radii of the chlorine and hydrogen atoms (r(Cl) + r(H) = 2.81 Å). Consequently, these values correspond well to strong hydrogen bonds. Multiple hydrogen bonds connect the different entities of the compound to form corrugated layers parallel to the *a*,*c* plane (figure, bottom). Examination of geometrical features of the organic moiety shows that the atoms C1, C2, C3, C4, C5 and C6 of the phenyl ring have a good co-planarity and they form a conjugated plane with an average deviation of 0.0066 Å. The mean value of C—C bond lengths is 1.384 Å which is between that of a single bond and a double bond and comparable to those in other substituted benzene compounds [3-5]. Furthermore, the bond length of C7—N2 is 1.139(4) Å, which clearly indicates a triple bond. The distances C2—C7 and C1—N1 of 1.434(4) Å and 1.467(3) Å, respectively, indicate two single bonds. Refining the structure in the acentric space group gives a value of –0.01(5) for the Flack parameter [6]. This value shows that the atomic arrangement corresponds to the correct absolute structure.

Table 1. Data collection and handling.

Crystal:	colorless prism, size 0.2 × 0.4 × 0.6 mm
Wavelength:	Mo K_α radiation (0.71070 Å)
μ:	4.48 cm^{-1}
Diffractometer, scan mode:	Enraf-Nonius Mach3, $\omega/2\theta$
$2\theta_{max}$:	50.18°
$N(hkl)_{measured}$, $N(hkl)_{unique}$:	2471, 1263
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\,\sigma(I_{obs})$, 1260
$N(param)_{refined}$:	120
Programs:	SIR92 [7], SHELXL-97 [8]

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

Atom	Site	x	y	z	U_{iso}
H(1)	4*a*	–0.257(4)	–0.204(1)	–0.761(2)	0.029(5)
H(2)	4*a*	–0.384(4)	–0.218(1)	–0.916(3)	0.040(5)
H(3)	4*a*	–0.505(4)	–0.200(1)	–0.774(3)	0.038(5)
H(7)	4*a*	–0.006(4)	0.044(1)	–1.026(2)	0.042(5)
H(6)	4*a*	–0.276(4)	0.141(2)	–0.907(3)	0.049(6)
H(5)	4*a*	–0.599(4)	0.096(1)	–0.773(2)	0.037(5)
H(4)	4*a*	–0.630(4)	–0.058(1)	–0.732(3)	0.039(5)

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)	4*a*	0.12742(5)	−0.20851(2)	−0.63907(4)	0.0248(2)	0.0366(2)	0.0381(2)	0.0024(1)	0.0072(1)	0.0085(2)
N(1)	4*a*	−0.3798(2)	−0.18422(8)	−0.8205(2)	0.0243(6)	0.0285(6)	0.0301(6)	−0.0014(5)	0.0063(5)	−0.0006(5)
N(2)	4*a*	0.1280(3)	−0.1628(1)	−1.0680(2)	0.0336(7)	0.0521(9)	0.0541(8)	0.0016(7)	0.0169(6)	−0.0057(7)
C(1)	4*a*	−0.3558(2)	−0.09146(9)	−0.8501(2)	0.0208(7)	0.0287(7)	0.0242(6)	0.0005(5)	0.0005(5)	−0.0018(5)
C(2)	4*a*	−0.1656(3)	−0.0612(1)	−0.9353(2)	0.0217(7)	0.0339(8)	0.0272(6)	−0.0005(6)	0.0024(5)	0.0000(6)
C(3)	4*a*	−0.1358(3)	0.0278(1)	−0.9552(2)	0.0321(8)	0.0381(9)	0.0359(9)	−0.0053(7)	0.0037(6)	0.0018(6)
C(4)	4*a*	−0.2957(3)	0.0849(1)	−0.8940(2)	0.044(1)	0.0270(7)	0.0455(9)	−0.0013(6)	−0.0002(7)	0.0014(6)
C(5)	4*a*	−0.4863(3)	0.0546(1)	−0.8130(2)	0.0372(9)	0.0364(8)	0.0424(9)	0.0093(7)	0.0040(7)	−0.0072(7)
C(6)	4*a*	−0.5155(3)	−0.0341(1)	−0.7901(2)	0.0242(7)	0.0375(8)	0.0330(7)	0.0003(6)	0.0064(6)	−0.0025(6)
C(7)	4*a*	−0.0021(3)	−0.1193(1)	−1.0062(2)	0.0242(8)	0.0373(8)	0.0331(8)	−0.0042(6)	0.0057(6)	0.0004(6)

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References

1. Oueslati, A.; Rayes, A.; Ben Nasr, C.; Rzaigui, M.: Crystal structure of 5-chloro-2-methylanilinium chloride hydrate, $[ClC_7H_6NH_3]Cl \cdot H_2O$. Z. Kristallogr. **220** (2005) 105-106.
2. Kaabi, K.; Ben Nasr, C.; Rzaigui, M.: Synthesis and characterization of a new monophosphate $[2,5\text{-}(CH_3)_2C_6H_3NH_3]H_2PO_4$. J. Phys. Chem. Solids **65** (2004) 1759-1764.
3. Frey, W.; Henneböhle, M.; Jäger, V.: Crystal structure of (3*S*,4*S*)-4,5-*O*-cyclohexylidene-4,5-dihydroxy-3-methylamino-pentanoic acid hydrochloride, $(C_{12}H_{22}NO_4)Cl$. Z. Kristallogr. **219** (2004) 193-194.
4. Li, Z. J.; Chen, X. M.; Ren, Z. X.; Li, Y.; Chen, X. A.; Huang, Z. T.: Crystal and molecular structure of hexahydro-2-(nitro-benzolmethylene)pyrimidine. Chinese J. Struct. Chem. **16** (1997) 311-314.
5. López-Duplá, E.; Jones, P. G.; Vancea, F.: Secondary Bonding Interactions in Some Di- and Trihaloanilinium Halides. Z. Naturforsch. **58b** (2003) 191-200.
6. Flack, H. D.; Bernardinelli, G.: Absolute structure and absolute configuration. Acta Crystallogr. **A55** (1999) 908-915.
7. Altomare, A.; Cascarano, M.; Giacovazzo, G.; Guagliardi, A.; Burla, M. C.; Polidori, G.; Camalli, M.: SIR92 – a program for automatic solution of crystal structures by direct methods. J. Appl. Crystallogr. **27** (1994) 435.
8. Sheldrick, G. M.: SHELXL-97. Program for the Refinement of Crystal Structures. University of Göttingen, Germany 1997.