Crystal structure of *rac*-3-ammoniopyrrolidinium perchlorate, $C_4H_{12}Cl_2N_2O_8$

D. Kuppert, C. Roth, J. Sander and K. Hegetschweiler*

Universität des Saarlandes, Anorganische Chemie, Postfach 151150, D-66041 Saarbrücken, Germany

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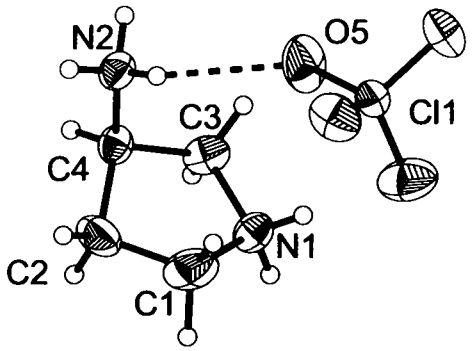

Abstract

$C_4H_{12}Cl_2N_2O_8$, monoclinic, $P12_1/c1$ (No. 14), $a = 7.927(2)$ Å, $b = 12.819(3)$ Å, $c = 10.171(2)$ Å, $\beta = 98.24(3)°$, $V = 1022.9$ Å^3, $Z = 4$, $R_{gt}(F) = 0.035$, $wR_{ref}(F^2) = 0.097$, $T = 293$ K.

Source of material

(±)-3-Aminopyrrolidine dihydrochloride was purchased from Lancaster Syntheses GmbH. An aqueous solution of the free base was obtained by deprotonation on an anion exchange resin (Dowex 2, OH^- form). Crystals of the title compound were grown by slow evaporation of an aqueous solution containing 3-aminopyrrolidine and two equivalents of perchloric acid.

Source of material

All hydrogen atoms were located in a difference Fourier map and were refined with variable isotropic displacement parameters.

Discussion

Cyclic organic polyamines represent a prominent class of metal complexing agents [1]. Their protonation products can serve as donors in hydrogen bonds and are therefore interesting building blocks for supramolecular structures [2]. The metal complexing properties of 3-aminopyrrolidine have recently been described [3], however, a crystal structure determination of this ligand, its protonated form or its metal complexes has not been reported to the best of our knowledge.

The structure of the title compound can be described in terms of layers, formed by cations of the same chirality together with one of the perchlorate anions (Cl1). The layers are aligned parallel to the *xy*-plane, and the primary ammonium group (N2) forms a N—H···O hydrogen bond to O(5) of the anion (N···O distance: 2.84 Å. All other N···O distances are longer than 3.00 Å. The layers are interconnected by the second perchlorate anion (Cl2). The pyrrolidine ring (Figure) has an envelope conformation with C2 in the apex (puckering parameters according to Cremer and Pople [4]: $Q = 0.336$ Å, $\varphi = 74.25°$). The remaining four ring atoms C1, N1, C3 and C4 are practically coplanar with deviations from their mean plane smaller than 0.007 Å.

Table 1. Data collection and handling.

Crystal:	colorless prism, size 0.2 × 0.2 × 0.3 mm
Wavelength:	Mo K_α radiation (0.71073 Å)
μ:	6.68 cm^{-1}
Diffractometer, scan mode:	Stoe Stadi 4, ω/θ
$2\theta_{max}$:	45°
$N(hkl)_{measured}$, $N(hkl)_{unique}$:	1338, 1338
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\,\sigma(I_{obs})$, 1244
$N(param)_{refined}$:	193
Programs:	SHELXS-90 [5], SHELXL-97 [6]

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

Atom	Site	x	y	z	U_{iso}
H(1NB)	4*e*	1.041(5)	1.201(3)	0.246(4)	0.06(1)
H(1CB)	4*e*	−0.108(6)	0.929(4)	0.568(5)	0.11(2)
H(3CB)	4*e*	0.850(5)	0.661(3)	0.415(4)	0.06(1)
H(2NA)	4*e*	0.551(5)	0.812(3)	−0.223(4)	0.05(1)
H(4C)	4*e*	0.665(4)	1.000(2)	−0.145(3)	0.033(8)
H(1NA)	4*e*	0.962(5)	1.113(3)	0.257(3)	0.05(1)
H(2NB)	4*e*	0.526(5)	0.852(3)	−0.104(5)	0.07(1)
H(1CA)	4*e*	−0.253(5)	0.819(3)	0.590(4)	0.07(1)
H(2CA)	4*e*	0.774(6)	1.043(4)	0.679(5)	0.09(2)
H(3CA)	4*e*	0.928(4)	0.546(3)	0.400(3)	0.05(1)
H(2NC)	4*e*	0.453(5)	0.893(3)	−0.221(4)	0.06(1)
H(2CB)	4*e*	0.606(5)	0.972(3)	0.611(4)	0.06(1)

* Correspondence author
(e-mail: hegetschweiler@mx.uni-saarland.de)

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*e*	0.75047(8)	0.90943(5)	0.22370(7)	0.0313(4)	0.0251(4)	0.0260(4)	–0.0004(3)	0.0013(3)	0.0012(3)
Cl(2)	4*e*	0.25786(8)	0.74682(5)	0.53099(6)	0.0274(4)	0.0320(5)	0.0269(4)	–0.0001(3)	0.0023(3)	–0.0036(3)
O(1)	4*e*	0.7652(3)	1.0057(2)	0.2956(2)	0.060(2)	0.032(1)	0.046(1)	–0.003(1)	0.000(1)	–0.008(1)
O(2)	4*e*	0.2480(3)	0.6444(2)	0.4739(2)	0.046(1)	0.034(1)	0.044(1)	0.002(1)	–0.001(1)	–0.012(1)
O(3)	4*e*	0.1172(3)	0.8073(2)	0.4690(3)	0.044(1)	0.049(2)	0.073(2)	0.017(1)	–0.005(1)	0.003(1)
O(4)	4*e*	0.6257(3)	0.9213(2)	0.1090(2)	0.046(1)	0.058(2)	0.037(1)	0.007(1)	–0.011(1)	–0.005(1)
O(5)	4*e*	0.7027(4)	0.8290(2)	0.3060(2)	0.094(2)	0.043(1)	0.048(2)	–0.023(1)	0.011(1)	0.012(1)
O(6)	4*e*	0.4151(3)	0.7941(2)	0.5100(2)	0.038(1)	0.056(2)	0.062(2)	–0.012(1)	0.013(1)	–0.000(1)
O(7)	4*e*	0.9105(3)	0.8830(2)	0.1846(3)	0.041(1)	0.086(2)	0.060(2)	0.016(1)	0.012(1)	–0.006(1)
O(8)	4*e*	0.2518(3)	0.7408(2)	0.6697(2)	0.062(2)	0.074(2)	0.026(1)	–0.012(1)	0.011(1)	–0.009(1)
N(2)	4*e*	0.5446(3)	0.8691(2)	–0.1842(3)	0.027(2)	0.035(2)	0.038(2)	0.001(1)	0.006(1)	–0.001(1)
N(1)	4*e*	1.0643(3)	1.1395(3)	0.2553(3)	0.027(2)	0.041(2)	0.038(2)	–0.000(1)	0.004(1)	–0.002(1)
C(4)	4*e*	0.6881(4)	0.9429(2)	–0.1921(3)	0.032(2)	0.026(2)	0.034(2)	0.002(1)	0.006(1)	–0.005(1)
C(3)	4*e*	0.8584(4)	0.6043(3)	0.3616(3)	0.034(2)	0.050(2)	0.026(2)	0.006(2)	0.001(1)	0.006(2)
C(2)	4*e*	0.7051(5)	0.9709(3)	0.6670(4)	0.041(2)	0.054(2)	0.047(2)	0.002(2)	0.001(2)	0.024(2)
C(1)	4*e*	–0.1839(5)	0.8881(4)	0.6231(3)	0.052(2)	0.085(3)	0.027(2)	0.002(2)	0.009(2)	0.001(2)

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