Crystal structure of 1,1,3,3-tetramethyl-2-(1,3-dimethylimidazolidin-2-yl)guanidinium chloride, $(C_{10}H_{24}N_5)Cl$

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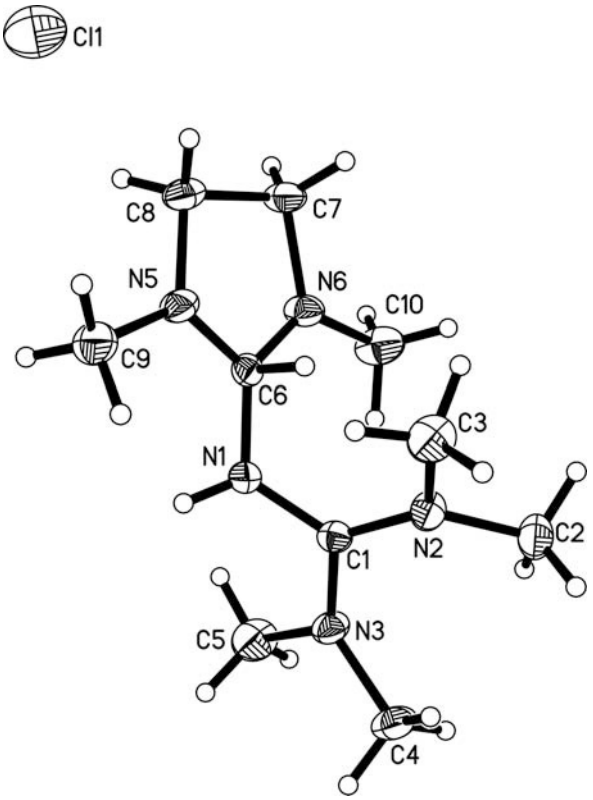

Abstract

$C_{10}H_{24}ClN_5$, monoclinic, $P2_1/n$ (no. 14), a = 12.049(2) Å, b = 6.7268(9) Å, c = 16.947(2) Å, β = 97.634(3)°, V = 1361.4 Å^3, Z = 4, $R_{gt}(F)$ = 0.067, $wR_{ref}(F^2)$ = 0.203, T = 296 K.

Source of material

To a solution of 1,3-dimethyl-2-imidazolidinone (11.4 g, 100 mmol) in toluene (30 ml) was added oxalyl chloride (15.3 g, 120 mmol) at room temperature within 20 min, and the mixture was stirred at 333 - 343 K for 6 hours. The precipitate was filtrated, washed with toluene and dried, affording 2-chloro-1,3-dimethylimidazolinium chloride (DMC). Then, tetramethylguanidine (12 g, 104 mmol) was added to a stirred solution of DMC (17 g, 100 mmol) in dry CH_2Cl_2 (35 ml) at room temperature for 16 hours, affording the crude product as a yellow liquid. By placing the mixture of acetone (10 mol) and crude product (2 g) in a straight glass tube, slow evaporation of acetone leads to the suitable colorless block-shaped crystals after two days.

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Experimental details

H atoms bound to methylene and methyl carbon atoms were assigned to calculated positions with d(C—H) = 0.96 - 0.98 Å and refined using a riding model, with U_{iso}(H) = 1.2 or 1.5 U_{eq}(C). N-bound H atoms were positioned geometrically with d(N—H) = 0.860(1) Å and included in the refinement in the riding model, with U_{iso}(H) = 1.5 U_{eq}(N). The large residual values are caused by the high sensitivity to atmospheric water vapor of the crystal and their crystallinity was partly destroyed during single crystal X-ray determination.

Discussion

Guanidinium compounds are of great interests mainly due to their high chemical and thermal stability, as well as catalytic activity [1-3]. Because of the unique role in molecular recognition, guanidinium and its derivatives become one of the key subjects in life science research [4]. Additionally, guanidinium derivatives have attracted interests during recent years due to the wide use in chemical and biological systems [5].

The asymmetric unit of the title crystal structure contains one cation 1,1,3,3-tetramethyl-2-(1,3-dimethylimidazolidin-2-yl)-guanidinium and one chloride anion. The C—C and C—N bond lengths in the title compound can be compared with the values in other guanidinium derivatives. Analysis of the crystal packing reveals the existence of weak C–H···Cl hydrogen bonds between the chloride anion and methylene and methyl groups from the dimethylimidazolidine moiety. These intermolecular C–H···Cl interactions generate a one-dimensional supramolecular chain along [010].

Table 1. Data collection and handling.

Crystal:	colorless block, size 0.12 × 0.23 × 0.28 mm
Wavelength:	Mo K_α radiation (0.71073 Å)
μ:	2.66 cm^{-1}
Diffractometer, scan mode:	Bruker APEX-II CCD, φ/ω
$2\theta_{max}$:	52°
$N(hkl)_{measured}$, $N(hkl)_{unique}$:	7893, 2675
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\,\sigma(I_{obs})$, 2072
$N(param)_{refined}$:	152
Programs:	SHELXS-97, SHELXL-97, SHELXTL [6]

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

Atom	Site	x	y	z	U_{iso}
H(2A)	4*e*	0.9168	0.0896	0.5928	0.070
H(2B)	4*e*	0.9671	0.3030	0.6089	0.070
H(2C)	4*e*	0.9747	0.1976	0.5272	0.070
H(3A)	4*e*	0.8615	0.4401	0.4396	0.077

Table 2. Continued.

Atom	Site	*x*	*y*	*z*	U_{iso}
H(3B)	4*e*	0.8792	0.5739	0.5160	0.077
H(3C)	4*e*	0.7581	0.5357	0.4723	0.077
H(4A)	4*e*	0.8254	0.0658	0.7039	0.076
H(4B)	4*e*	0.7232	0.1866	0.7269	0.076
H(4C)	4*e*	0.8139	0.2928	0.6835	0.076
H(5A)	4*e*	0.5667	0.0288	0.6451	0.085
H(5B)	4*e*	0.6507	–0.1442	0.6347	0.085
H(5C)	4*e*	0.5833	–0.0440	0.5596	0.085
H(6)	4*e*	0.7031	0.3328	0.4179	0.035
H(7A)	4*e*	0.6642	0.0400	0.2542	0.050

Table 2. Continued.

Atom	Site	*x*	*y*	*z*	U_{iso}
H(7B)	4*e*	0.7499	0.2180	0.2708	0.050
H(8A)	4*e*	0.6013	0.4179	0.2452	0.054
H(8B)	4*e*	0.5168	0.2419	0.2540	0.054
H(9A)	4*e*	0.4321	0.4675	0.3792	0.078
H(9B)	4*e*	0.5058	0.6264	0.3428	0.078
H(9C)	4*e*	0.5328	0.5675	0.4328	0.078
H(10A)	4*e*	0.8466	–0.0039	0.4009	0.082
H(10B)	4*e*	0.7540	–0.1631	0.3746	0.082
H(10C)	4*e*	0.7611	–0.0639	0.4589	0.082
H(1)	4*e*	0.5708	0.2944	0.4994	0.051

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}
C(1)	4*e*	0.7218(2)	0.2203(3)	0.5433(1)	0.034(1)	0.028(1)	0.028(1)	0.0039(8)	0.0053(8)	0.0016(8)
C(2)	4*e*	0.9294(2)	0.2159(4)	0.5692(2)	0.034(1)	0.052(1)	0.054(2)	–0.002(1)	0.002(1)	0.008(1)
C(3)	4*e*	0.8308(2)	0.4782(4)	0.4869(2)	0.057(2)	0.042(1)	0.054(2)	–0.016(1)	0.003(1)	0.015(1)
C(4)	4*e*	0.7735(2)	0.1711(5)	0.6879(1)	0.058(2)	0.066(2)	0.028(1)	–0.004(1)	–0.001(1)	0.002(1)
C(5)	4*e*	0.6200(2)	–0.0214(4)	0.6127(2)	0.060(2)	0.060(2)	0.050(2)	–0.022(1)	0.006(1)	0.018(1)
C(6)	4*e*	0.6397(2)	0.2405(3)	0.4110(1)	0.0266(9)	0.031(1)	0.031(1)	–0.0031(8)	0.0049(8)	0.0037(8)
C(7)	4*e*	0.6826(2)	0.1582(4)	0.2860(1)	0.054(1)	0.047(1)	0.026(1)	0.005(1)	0.0085(9)	0.001(1)
C(8)	4*e*	0.5859(2)	0.3047(4)	0.2775(1)	0.052(1)	0.055(1)	0.028(1)	0.006(1)	0.0037(9)	0.008(1)
C(9)	4*e*	0.5065(2)	0.5193(4)	0.3803(2)	0.056(1)	0.055(2)	0.044(1)	0.023(1)	0.006(1)	0.004(1)
C(10)	4*e*	0.7707(3)	–0.0425(4)	0.4042(2)	0.077(2)	0.043(1)	0.043(1)	0.024(1)	0.005(1)	0.002(1)
Cl(1)	4*e*	0.48752(7)	0.7895(1)	0.18027(5)	0.0837(6)	0.0516(5)	0.0617(5)	–0.0026(4)	0.0093(4)	0.0102(3)
N(1)	4*e*	0.6323(1)	0.2431(3)	0.4884(1)	0.0297(9)	0.046(1)	0.0276(9)	0.0052(7)	0.0049(7)	0.0039(8)
N(2)	4*e*	0.8213(2)	0.3038(3)	0.5363(1)	0.0331(9)	0.0347(9)	0.040(1)	–0.0046(8)	0.0010(7)	0.0091(8)
N(3)	4*e*	0.7094(2)	0.1225(3)	0.6106(1)	0.040(1)	0.045(1)	0.028(1)	–0.0079(8)	0.0009(7)	0.0073(8)
N(5)	4*e*	0.5802(2)	0.3634(3)	0.3594(1)	0.044(1)	0.050(1)	0.028(1)	0.0162(9)	0.0026(8)	0.0066(9)
N(6)	4*e*	0.6959(2)	0.1125(3)	0.3713(1)	0.048(1)	0.037(1)	0.030(1)	0.0092(8)	0.0086(8)	0.0049(8)

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