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Cyanomethanaminium perchlorate

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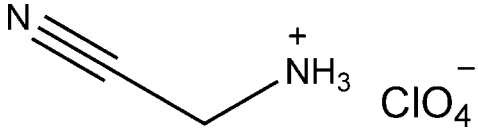
Key indicators: single-crystal X-ray study; T = 293 K; mean σ(C–C) = 0.003 Å; R factor = 0.034; wR factor = 0.096; data-to-parameter ratio = 15.7.

In the crystal of the title salt, $C_2H_5N_2^+\cdot ClO_4^-$, the cations and anions are connected *via* N—H···O and C—H···O hydrogen bonds, forming a three-dimensional network.

Related literature

For general background, see: Haertling (1999); Homes *et al.* (2001). For a related structure, see: Han & Zhang (2010).



Experimental

Crystal data

$C_2H_5N_2^+\cdot ClO_4^-$
M_r = 156.53
Orthorhombic, *Pbca*
a = 9.908 (2) Å
b = 10.398 (2) Å
c = 11.176 (2) Å
V = 1151.4 (4) Å^3
Z = 8
Mo $K\alpha$ radiation
μ = 0.61 mm^{-1}
T = 293 K
0.20 × 0.19 × 0.18 mm

Data collection

Rigaku Mercury2 diffractometer
Absorption correction: multi-scan (*CrystalClear*; Rigaku, 2005)
T_{min} = 0.88, T_{max} = 0.90
10965 measured reflections
1321 independent reflections
1151 reflections with $I > 2\sigma(I)$
R_{int} = 0.044

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.034
$wR(F^2)$ = 0.096
S = 1.12
1321 reflections
84 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.40 e Å^{-3}
$\Delta\rho_{min}$ = −0.37 e Å^{-3}

Table 1
Hydrogen-bond geometry (Å, °).

D—H···A	D—H	H···A	D···A	D—H···A
N1—H1C···O1^{i}	0.89	2.10	2.920 (2)	152
N1—H1D···O4	0.89	2.03	2.919 (2)	175
N1—H1E···O2^{ii}	0.89	2.10	2.914 (3)	152
C1—H1A···O1^{iii}	0.97	2.49	3.456 (3)	172
C1—H1B···O3^{iv}	0.97	2.57	3.532 (3)	169

Symmetry codes: (i) $x, -y+\frac{3}{2}, z+\frac{1}{2}$; (ii) $x+\frac{1}{2}, -y+\frac{3}{2}, -z+1$; (iii) $-x+\frac{1}{2}, -y+1, z+\frac{1}{2}$; (iv) $-x+1, -y+1, -z+1$.

Data collection: *CrystalClear* (Rigaku, 2005); cell refinement: *CrystalClear*; data reduction: *CrystalClear*; program(s) used to solve structure: *SHELXTL* (Sheldrick, 2008); program(s) used to refine structure: *SHELXTL*; molecular graphics: *SHELXTL*; software used to prepare material for publication: *SHELXTL*.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: XU5654).

References

Haertling, G. H. (1999). *J. Am. Ceram. Soc.* **82**, 797–810.
Han, M. T. & Zhang, Y. (2010). *Acta Cryst.* E**66**, o1941.
Homes, C. C., Vogt, T., Shapiro, S. M., Wakimoto, S. & Ramirez, A. P. (2001). *Science*, **293**, 673–676.
Rigaku (2005). *CrystalClear*. Rigaku Corporation, Tokyo, Japan.
Sheldrick, G. M. (2008). *Acta Cryst.* A**64**, 112–122.

supporting information

Acta Cryst. (2012). E**68**, o3480 [doi:10.1107/S1600536812047782]

Cyanomethanaminium perchlorate

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S1. Comment

At present, much attention in ferroelectric material field is focused on developing ferroelectric pure organic or inorganic compounds (Haertling, 1999; Homes *et al.*, 2001). In order to find more dielectric ferroelectric materials, we investigate the physical properties of the title compound (Fig. 1). The dielectric constant of the title compound as a function of temperature indicates that the permittivity is basically temperature-independent (dielectric constant equaling to 3.7 to 5.2), suggesting that this compound should be not a real ferroelectrics or there may be no distinct phase transition occurred within the measured temperature range. Similarly, below the melting point (453 K) of the compound, the dielectric constant as a function of temperature also goes smoothly, and there is no dielectric anomaly observed (dielectric constant equaling to 3.7 to 5.2). Herein, we report the synthesis and crystal structure of the title compound.

Molecules of the title compound have normal geometric parameters. The bond lengths and angles are within their normal ranges (Han & Zhang, 2010). As can be seen from the packing diagram (Fig. 2), molecules are connected *via* intermolecular N—H···O and C—H···O hydrogen bonds to form a three dimensional network.

S2. Experimental

A mixture of aminoacetonitrile hydrochloride (0.095 g, 0.01 mol) and perchloric acid (1.40 g, 0.01 mol) in methanol (20 ml) was stirred until clear. After several days, the title compound was formed and recrystallized from solution to afford colourless prismatic crystals suitable for X-ray analysis.

S3. Refinement

H atoms were positioned geometrically and refined using a riding model with C—H = 0.97 and N—H = 0.89 Å, U_{iso}(H) = 1.2U_{eq}(C) and 1.5U_{eq}(N).

Figure 1

Perspective structure of the title compound with displacement ellipsoids drawn at the 50% probability level. The dashed line indicates N—H···O hydrogen bond.

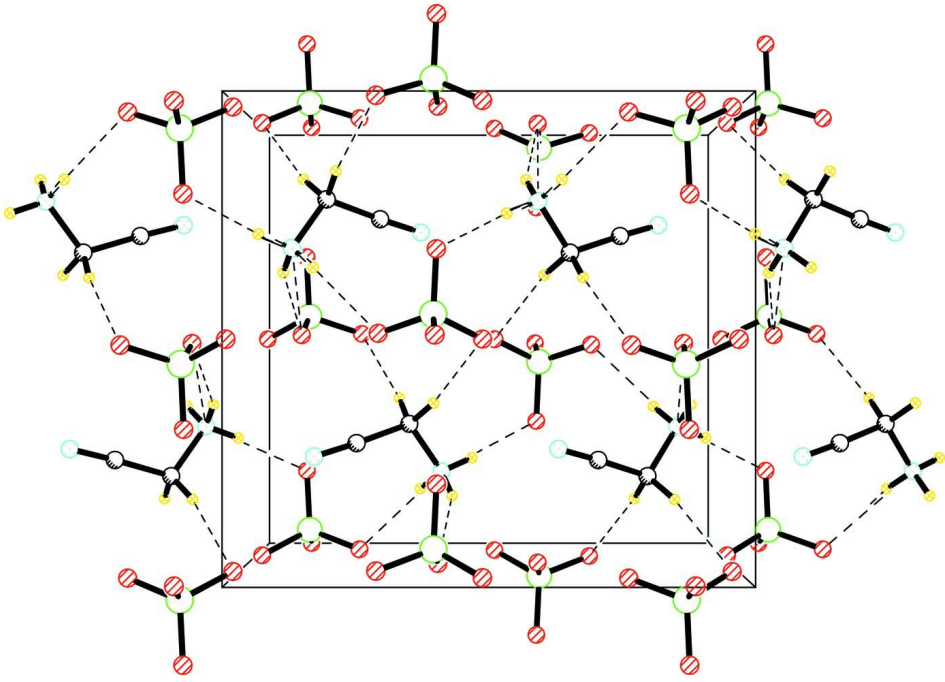

Figure 2

The crystal packing of the title compound viewed along the*a* axis showing the hydrogen bondings network.

Cyanomethanaminium perchlorate

Crystal data

$C_2H_5N_2^+·ClO_4^-$
M_r = 156.53
Orthorhombic, *Pbca*
Hall symbol: -P 2ac 2ab
a = 9.908 (2) Å
b = 10.398 (2) Å
c = 11.176 (2) Å
V = 1151.4 (4) Å^3
Z = 8
F(000) = 640
D_x = 1.806 Mg m^{-3}
Mo *Kα* radiation, *λ* = 0.71073 Å
Cell parameters from 1321 reflections
θ = 2.6–27.5°
μ = 0.61 mm^{-1}
T = 293 K
Prism, colorless
0.20 × 0.19 × 0.18 mm

Data collection

Rigaku Mercury2 diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
Detector resolution: 13.6612 pixels mm^{-1}
CCD_Profile_fitting scans
Absorption correction: multi-scan (*CrystalClear*; Rigaku, 2005)
T_{min} = 0.88, T_{max} = 0.90
10965 measured reflections
1321 independent reflections
1151 reflections with $I > 2\sigma(I)$
R_{int} = 0.044
θ_{max} = 27.5°, θ_{min} = 3.4°
h = −12→12
k = −13→13
l = −14→14

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.034$
$wR(F^2) = 0.096$
$S = 1.12$
1321 reflections
84 parameters
0 restraints
Primary atom site location: structure-invariant direct methods
Secondary atom site location: difference Fourier map
Hydrogen site location: inferred from neighbouring sites
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0373P)^2 + 0.8373P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.002$
$\Delta\rho_{max} = 0.40$ e Å^{-3}
$\Delta\rho_{min} = -0.37$ e Å^{-3}
Extinction correction: *SHELXTL* (Sheldrick, 2008), $Fc^*=kFc[1+0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$
Extinction coefficient: 0.072 (3)

Special details

Geometry. All e.s.d.'s (except the e.s.d. in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell e.s.d.'s are taken into account individually in the estimation of e.s.d.'s in distances, angles and torsion angles; correlations between e.s.d.'s in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell e.s.d.'s is used for estimating e.s.d.'s involving l.s. planes.

Refinement. Refinement of F^2 against ALL reflections. The weighted *R*-factor *wR* and goodness of fit *S* are based on F^2, conventional *R*-factors *R* are based on *F*, with *F* set to zero for negative F^2. The threshold expression of $F^2 > \sigma(F^2)$ is used only for calculating *R*-factors(gt) *etc*. and is not relevant to the choice of reflections for refinement. *R*-factors based on F^2 are statistically about twice as large as those based on *F*, and *R*- factors based on ALL data will be even larger.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²)

	x	*y*	*z*	U_{iso}*/U_{eq}
Cl1	0.24234 (4)	0.55298 (4)	0.39030 (4)	0.0262 (2)
N1	0.38874 (19)	0.80122 (18)	0.60010 (15)	0.0338 (4)
H1C	0.3343	0.8461	0.6478	0.051*
H1D	0.3423	0.7726	0.5374	0.051*
H1E	0.4558	0.8514	0.5750	0.051*
N2	0.5768 (2)	0.7581 (2)	0.85728 (18)	0.0503 (6)
O1	0.30000 (17)	0.51248 (16)	0.27907 (14)	0.0432 (4)
O2	0.10533 (16)	0.50926 (18)	0.39645 (16)	0.0485 (5)
O3	0.31737 (19)	0.50020 (19)	0.48743 (16)	0.0521 (5)
O4	0.24459 (16)	0.69160 (15)	0.39638 (15)	0.0414 (5)
C1	0.4448 (2)	0.6911 (2)	0.6667 (2)	0.0354 (5)
H1A	0.3718	0.6343	0.6900	0.042*
H1B	0.5049	0.6432	0.6147	0.042*
C2	0.5189 (2)	0.7311 (2)	0.77361 (19)	0.0335 (5)

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Cl1	0.0279 (3)	0.0258 (3)	0.0249 (3)	0.00019 (17)	−0.00002 (18)	−0.00046 (17)
N1	0.0368 (10)	0.0363 (10)	0.0283 (9)	−0.0023 (7)	−0.0062 (7)	−0.0005 (7)
N2	0.0541 (12)	0.0613 (15)	0.0354 (11)	−0.0057 (11)	−0.0128 (9)	−0.0011 (10)
O1	0.0516 (10)	0.0449 (9)	0.0331 (9)	0.0040 (8)	0.0122 (7)	−0.0064 (7)
O2	0.0319 (9)	0.0492 (10)	0.0643 (12)	−0.0084 (7)	0.0085 (8)	−0.0033 (8)

O3	0.0638 (12)	0.0515 (10)	0.0409 (10)	0.0079 (9)	−0.0182 (9)	0.0077 (8)
O4	0.0529 (11)	0.0247 (9)	0.0466 (11)	0.0000 (6)	−0.0076 (8)	−0.0046 (6)
C1	0.0415 (11)	0.0306 (11)	0.0341 (11)	−0.0017 (9)	−0.0089 (10)	−0.0009 (9)
C2	0.0308 (10)	0.0386 (11)	0.0310 (11)	−0.0019 (9)	−0.0016 (8)	0.0028 (9)

Geometric parameters (Å, º)

Cl1—O3	1.4256 (17)	N1—H1D	0.8900
Cl1—O1	1.4314 (16)	N1—H1E	0.8900
Cl1—O2	1.4333 (17)	N2—C2	1.133 (3)
Cl1—O4	1.4431 (16)	C1—C2	1.462 (3)
N1—C1	1.475 (3)	C1—H1A	0.9700
N1—H1C	0.8900	C1—H1B	0.9700
O3—Cl1—O1	109.87 (11)	H1C—N1—H1E	109.5
O3—Cl1—O2	109.59 (12)	H1D—N1—H1E	109.5
O1—Cl1—O2	109.04 (11)	C2—C1—N1	112.37 (18)
O3—Cl1—O4	109.92 (11)	C2—C1—H1A	109.1
O1—Cl1—O4	109.18 (10)	N1—C1—H1A	109.1
O2—Cl1—O4	109.22 (10)	C2—C1—H1B	109.1
C1—N1—H1C	109.5	N1—C1—H1B	109.1
C1—N1—H1D	109.5	H1A—C1—H1B	107.9
H1C—N1—H1D	109.5	N2—C2—C1	177.8 (2)
C1—N1—H1E	109.5		

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1—H1*C*···O1^{i}	0.89	2.10	2.920 (2)	152
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