Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

Ammonium imidazolium dichromate

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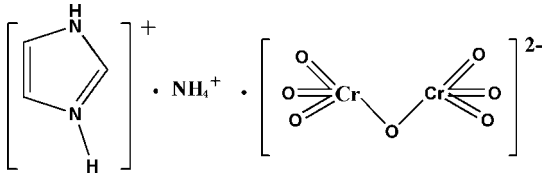
Received 24 February 2012; accepted 4 March 2012

Key indicators: single-crystal X-ray study; T = 293 K; mean σ(C–C) = 0.005 Å; R factor = 0.037; wR factor = 0.085; data-to-parameter ratio = 15.1.

In the crystal structure of the title compound, $(C_3H_5N_2)(NH_4)[Cr_2O_7]$, the anions and cations are linked through N—H···O hydrogen bonds, resulting in a three-dimensional structure which contains three kinds of layers parallel to (001). One layer contains imidazole cations, the other two layers the ammonium cations and dichromate anions. The dichromate anion has an eclipsed conformation with a dihedral angle of 14.65 (18)° between the mean planes of the O—P—O—P—O backbone.

Related literature

The title compound was synthesized as part of a search for ferroelectric materials. For general background to ferroelectric compounds with metal-organic framework structures, see: Fu *et al.* (2009); Ye *et al.* (2006); Zhang *et al.* (2008, 2010). For graph-set motifs, see: Bernstein *et al.* (1995).



Experimental

Crystal data

$(C_3H_5N_2)(NH_4)[Cr_2O_7]$
M_r = 303.13
Monoclinic, $P2_1/c$
a = 5.6260 (11) Å
b = 8.2749 (17) Å
c = 21.593 (4) Å
β = 91.90 (3)°
V = 1004.7 (3) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 2.18 mm^{-1}
T = 293 K
0.32 × 0.27 × 0.22 mm

Data collection

Rigaku SCXmini diffractometer
Absorption correction: multi-scan (*CrystalClear*; Rigaku, 2005)
T_{min} = 0.502, T_{max} = 0.618
10091 measured reflections
2297 independent reflections
1907 reflections with $I > 2\sigma(I)$
R_{int} = 0.047

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.037
$wR(F^2)$ = 0.085
S = 1.09
2297 reflections
152 parameters
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.34 e Å^{-3}
$\Delta\rho_{min}$ = −0.52 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
N2—H2B···O7^{i}	0.86	2.16	3.011 (4)	170
N3—H3B···O1^{ii}	0.86	2.04	2.827 (4)	152
N1—H1B···O7	0.79 (5)	2.16 (5)	2.940 (4)	169 (5)
N1—H1C···O4^{iii}	0.83 (5)	2.19 (5)	2.943 (4)	151 (5)
N1—H1D···O3^{iv}	0.71 (5)	2.29 (5)	3.004 (5)	176 (5)
N1—H1E···O6^{v}	0.74 (5)	2.15 (5)	2.895 (4)	174 (5)

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

Data collection: *CrystalClear* (Rigaku, 2005); cell refinement: *CrystalClear*; data reduction: *CrystalClear*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *DIAMOND* (Brandenburg & Putz, 2005); software used to prepare material for publication: *SHELXL97*.

This work was supported by Southeast University.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: FJ2526).

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supporting information

Acta Cryst. (2012). E**68**, m389 [https://doi.org/10.1107/S1600536812009506]

Ammonium imidazolium dichromate

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S1. Comment

We synthesized the title compound to find ferroelectric material by dielectric measurements of compound as a function of temperature(Fu *et al.*, 2009; Ye *et al.*, 2006; Zhang *et al.*, 2008; Zhang *et al.*, 2010). In the range from 190 K to near its melting point (m.p. >370 K), no dielectric anomaly was observed.

A view of the title compound is shown in Fig.1. The structure is consolidated by multiple intermolecular and intramolecular hydrogen bonds between N and O. This hydrogen bondings (table 1, Fig.2) produces a three-dimensional net work. The N···O distances of the hydrogen bonding are in the range of 2.827 (4) – 3.011 (4) for table 1. Hydrogen bonding is the most reliable desigen element in the non-covalent assembly of molecules with donor and accept functionalities, and as such it is the most important interaction in crystal engineering (Bernstein *et al.*, 1995).

S2. Experimental

A mixture of imidazole (0.68 g, 10 mmol), ammonium dichromate (2.5 g, 10 mmol) in water was stirred for several days at ambient temperature, red sheet crystals were obtained.

S3. Refinement

Hydrogen atom positions were calculated and allowed to ride on their parent atoms with aromtic C–H = 0.93 Å and N–H = 0.86 Å, and with *U*ĩso(H)=1.2*U*eq(C or N).The H atoms on N1 were freely refined.

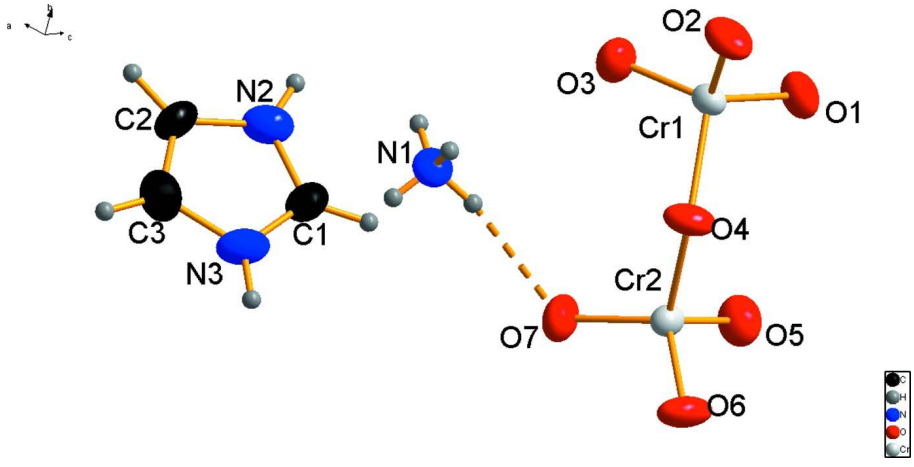

Figure 1

The molecular structure of the title compound, with the displacement ellipsoids drawn at the 30% probability level.

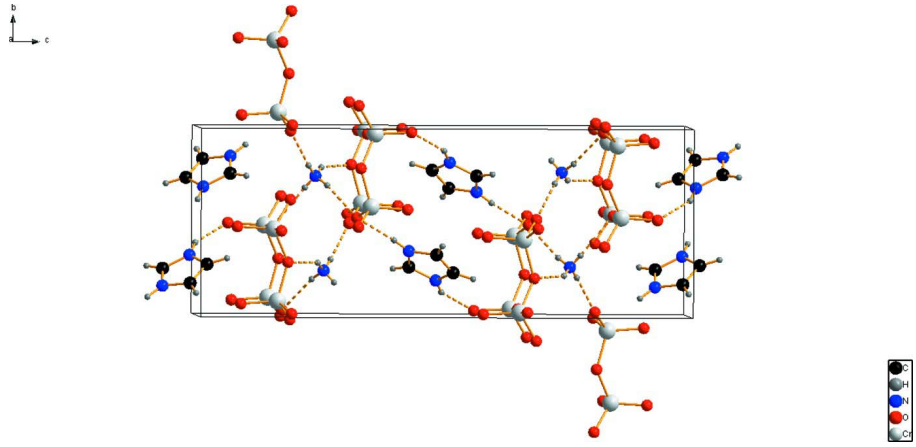

Figure 2

Packing diagram of the title compound, showing the structure along the *a* axis. Hydrogen bonds are shown as dashed lines.

Ammonium imidazolium dichromate

Crystal data

$(C_3H_5N_2)(NH_4)[Cr_2O_7]$
$M_r = 303.13$
Monoclinic, $P2_1/c$
Hall symbol: -P 2ybc
$a = 5.6260$ (11) Å
$b = 8.2749$ (17) Å
$c = 21.593$ (4) Å
$\beta = 91.90$ (3)°
$V = 1004.7$ (3) Å^3
$Z = 4$
$F(000) = 608$
$D_x = 2.004$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 2297 reflections
$\theta = 3.1$–$27.5°$
$\mu = 2.18$ mm^{-1}
$T = 293$ K
Plane, red
0.32 × 0.27 × 0.22 mm

Data collection

Rigaku SCXmini diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
CCD Profile fitting scans
Absorption correction: multi-scan (*CrystalClear*; Rigaku, 2005)
$T_{min} = 0.502$, $T_{max} = 0.618$
10091 measured reflections
2297 independent reflections
1907 reflections with $I > 2\sigma(I)$
$R_{int} = 0.047$
$\theta_{max} = 27.5°$, $\theta_{min} = 3.1°$
$h = -7 \rightarrow 7$
$k = -10 \rightarrow 10$
$l = -28 \rightarrow 27$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.037$
$wR(F^2) = 0.085$
$S = 1.09$
2297 reflections
152 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 atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0321P)^2 + 0.944P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.34$ e Å^{-3}
$\Delta\rho_{min} = -0.52$ e Å^{-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}
C1	0.3469 (6)	0.7554 (4)	0.06526 (17)	0.0396 (8)
H1A	0.2345	0.7447	0.0956	0.048*
C2	0.6599 (6)	0.8268 (4)	0.01522 (17)	0.0390 (8)
H2A	0.8034	0.8757	0.0060	0.047*
C3	0.5350 (7)	0.7289 (5)	−0.02122 (18)	0.0473 (9)
H3A	0.5725	0.6965	−0.0610	0.057*
N1	0.5234 (6)	0.7387 (4)	0.24161 (17)	0.0325 (6)
N2	0.5405 (5)	0.8437 (4)	0.06922 (14)	0.0437 (7)
H2B	0.5851	0.9023	0.1004	0.052*
N3	0.3399 (5)	0.6843 (3)	0.01056 (15)	0.0449 (8)
H3B	0.2299	0.6200	−0.0030	0.054*
O1	0.0282 (4)	0.9810 (3)	0.42701 (10)	0.0378 (5)
O2	−0.0350 (5)	1.1228 (3)	0.31828 (11)	0.0422 (6)
O3	0.3641 (4)	0.9650 (3)	0.34399 (12)	0.0429 (6)
O4	−0.0528 (4)	0.7910 (2)	0.32273 (11)	0.0375 (5)
O5	−0.0001 (5)	0.5720 (3)	0.41884 (11)	0.0458 (6)
O6	−0.2197 (4)	0.4834 (3)	0.31508 (12)	0.0425 (6)
O7	0.2496 (4)	0.5232 (3)	0.32071 (11)	0.0415 (6)
Cr1	0.08035 (8)	0.97044 (5)	0.35383 (2)	0.02452 (14)
Cr2	−0.00312 (8)	0.58625 (5)	0.34477 (2)	0.02334 (14)
H1B	0.439 (8)	0.692 (6)	0.264 (2)	0.066 (16)*
H1C	0.644 (9)	0.783 (6)	0.256 (2)	0.081 (18)*
H1D	0.556 (8)	0.677 (6)	0.221 (2)	0.051 (15)*
H1E	0.439 (8)	0.796 (6)	0.226 (2)	0.059 (15)*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.0366 (18)	0.0382 (19)	0.044 (2)	−0.0071 (15)	0.0056 (15)	0.0040 (16)
C2	0.0287 (16)	0.0377 (18)	0.051 (2)	−0.0066 (14)	0.0055 (15)	0.0081 (16)
C3	0.051 (2)	0.051 (2)	0.040 (2)	0.0087 (18)	0.0061 (17)	−0.0013 (18)
N1	0.0337 (17)	0.0280 (16)	0.0357 (17)	0.0060 (14)	−0.0008 (14)	0.0038 (14)
N2	0.0505 (18)	0.0362 (16)	0.0438 (18)	−0.0065 (14)	−0.0080 (14)	−0.0024 (13)
N3	0.0370 (16)	0.0365 (16)	0.060 (2)	−0.0108 (13)	−0.0108 (14)	−0.0028 (15)
O1	0.0463 (13)	0.0415 (13)	0.0256 (11)	0.0090 (11)	0.0029 (10)	−0.0019 (10)
O2	0.0659 (16)	0.0254 (11)	0.0352 (13)	0.0112 (11)	0.0000 (11)	0.0048 (10)

O3	0.0323 (12)	0.0482 (14)	0.0485 (15)	−0.0032 (11)	0.0072 (11)	−0.0030 (12)
O4	0.0465 (13)	0.0199 (10)	0.0452 (14)	−0.0002 (9)	−0.0130 (11)	0.0003 (9)
O5	0.0538 (15)	0.0505 (15)	0.0334 (13)	0.0077 (12)	0.0042 (11)	0.0057 (11)
O6	0.0324 (12)	0.0254 (11)	0.0686 (17)	−0.0075 (9)	−0.0132 (11)	0.0037 (11)
O7	0.0290 (12)	0.0459 (14)	0.0501 (15)	0.0066 (10)	0.0109 (11)	0.0050 (11)
Cr1	0.0307 (3)	0.0190 (2)	0.0239 (3)	0.00146 (18)	0.00126 (19)	−0.00066 (18)
Cr2	0.0199 (2)	0.0209 (2)	0.0292 (3)	0.00099 (17)	0.00006 (18)	0.00220 (18)

Geometric parameters (Å, º)

C1—N2	1.312 (4)	N1—H1E	0.74 (5)
C1—N3	1.319 (5)	N2—H2B	0.8600
C1—H1A	0.9300	N3—H3B	0.8600
C2—C3	1.316 (5)	O1—Cr1	1.619 (2)
C2—N2	1.372 (5)	O2—Cr1	1.602 (2)
C2—H2A	0.9300	O3—Cr1	1.618 (2)
C3—N3	1.364 (5)	O4—Cr2	1.779 (2)
C3—H3A	0.9300	O4—Cr1	1.784 (2)
N1—H1B	0.79 (5)	O5—Cr2	1.603 (2)
N1—H1C	0.83 (5)	O6—Cr2	1.602 (2)
N1—H1D	0.71 (5)	O7—Cr2	1.616 (2)
N2—C1—N3	107.8 (3)	C2—N2—H2B	125.8
N2—C1—H1A	126.1	C1—N3—C3	109.2 (3)
N3—C1—H1A	126.1	C1—N3—H3B	125.4
C3—C2—N2	107.7 (3)	C3—N3—H3B	125.4
C3—C2—H2A	126.1	Cr2—O4—Cr1	129.15 (13)
N2—C2—H2A	126.1	O2—Cr1—O3	110.17 (13)
C2—C3—N3	106.8 (3)	O2—Cr1—O1	109.98 (12)
C2—C3—H3A	126.6	O3—Cr1—O1	109.94 (13)
N3—C3—H3A	126.6	O2—Cr1—O4	108.44 (11)
H1B—N1—H1C	119 (5)	O3—Cr1—O4	109.34 (12)
H1B—N1—H1D	102 (5)	O1—Cr1—O4	108.94 (12)
H1C—N1—H1D	110 (5)	O6—Cr2—O5	110.06 (14)
H1B—N1—H1E	102 (4)	O6—Cr2—O7	111.42 (13)
H1C—N1—H1E	113 (5)	O5—Cr2—O7	108.47 (13)
H1D—N1—H1E	110 (5)	O6—Cr2—O4	106.74 (11)
C1—N2—C2	108.5 (3)	O5—Cr2—O4	109.46 (12)
C1—N2—H2B	125.8	O7—Cr2—O4	110.69 (12)

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N2—H2*B*···O7^{i}	0.86	2.16	3.011 (4)	170
N3—H3*B*···O1^{ii}	0.86	2.04	2.827 (4)	152
N1—H1*B*···O7	0.79 (5)	2.16 (5)	2.940 (4)	169 (5)
N1—H1*C*···O4^{iii}	0.83 (5)	2.19 (5)	2.943 (4)	151 (5)

N1—H1*D*···O3^{iv}	0.71 (5)	2.29 (5)	3.004 (5)	176 (5)
N1—H1*E*···O6^{v}	0.74 (5)	2.15 (5)	2.895 (4)	174 (5)

Symmetry codes: (i) −*x*+1, *y*+1/2, −*z*+1/2; (ii) *x*, −*y*+3/2, *z*−1/2; (iii) *x*+1, *y*, *z*; (iv) −*x*+1, *y*−1/2, −*z*+1/2; (v) −*x*, *y*+1/2, −*z*+1/2.