Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

1,1′-(Ethane-1,2-diyl)dipyridinium dichromate(VI)

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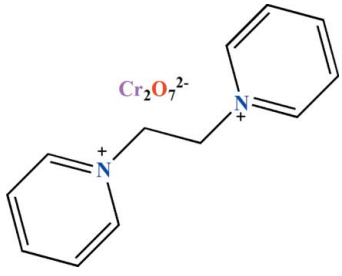
Received 25 January 2012; accepted 7 February 2012

Key indicators: single-crystal X-ray study; T = 100 K; mean σ(C–C) = 0.005 Å; R factor = 0.027; wR factor = 0.067; data-to-parameter ratio = 12.6.

In the cation of the title salt, $(C_{12}H_{14}N_2)[Cr_2O_7]$, the two pyridinium moieties are in an *anti* orientation with respect to one another. The dihedral angle between the pyridine rings is 6.3 (2)°. The N—C—C—N torsion angle is 177.5 (2)°. In the dianion, the Cr^{VI} ions are in a slightly distorted tetrahedral coordination environment and the bond angles at the independent Cr^{VI} ions are in the ranges 105.93 (10)–110.60 (11) and 107.35 (11)–111.07 (12)°. The Cr—O—Cr angle is 127.96 (12)°. The crystal used was an inversion twin with refined components of 0.510 (19) and 0.490 (19).

Related literature

For the crystal structures of the salts with formula $[C_5H_5NCH_2CH_2NC_5H_5]^{2+}\cdot 2X^-$ [X^- = IO_3^-, IO_4^-], the preparation of 1,1′-(ethane-1,2-diyl)dipyridinium dibromide and the orientation of pyridine moieties, see: Gholizadeh, Maleki *et al.* (2011); Gholizadeh, Hojati *et al.* (2011). For dichromate salts, see: Lennartson & Håkansson (2009); Averbuch-Pouchot *et al.* (1984).



Experimental

Crystal data

$(C_{12}H_{14}N_2)[Cr_2O_7]$
M_r = 402.25
Monoclinic, $P2_1$
a = 8.2882 (4) Å
b = 9.0722 (4) Å
c = 10.0179 (5) Å
β = 91.882 (1)°
V = 752.86 (6) Å^3
Z = 2
Mo $K\alpha$ radiation
μ = 1.48 mm^{-1}
T = 100 K
0.22 × 0.15 × 0.15 mm

Data collection

Bruker APEXII CCD diffractometer
Absorption correction: multi-scan (*SADABS*; Bruker, 2005)
T_{min} = 0.737, T_{max} = 0.808
5446 measured reflections
2628 independent reflections
2546 reflections with $I > 2\sigma(I)$
R_{int} = 0.025

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.027
$wR(F^2)$ = 0.067
S = 1.04
2628 reflections
209 parameters
1 restraint
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.47 e Å^{-3}
$\Delta\rho_{min}$ = −0.21 e Å^{-3}
Absolute structure: Flack (1983), 1163 Friedel pairs
Flack parameter: 0.510 (19)

Data collection: *APEX2* (Bruker, 2005); cell refinement: *SAINT* (Bruker, 2005); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *SHELXTL* (Sheldrick, 2008); software used to prepare material for publication: *SHELXTL* and *enCIFer* (Allen *et al.*, 2004).

Support of this investigation by Ferdowsi University of Mashhad is gratefully acknowledged.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: LH5410).

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supporting information

Acta Cryst. (2012). E**68**, m305 [doi:10.1107/S1600536812005430]

1,1′-(Ethane-1,2-diyl)dipyridinium dichromate(VI)

Mostafa Gholizadeh, Mehrdad Pourayoubi, Mehdi Kia, Arnold L. Rheingold and James A. Golen

S1. Comment

In recently published papers, the structure determinations of $[C_5H_5NCH_2CH_2NC_5H_5]^{2+}.2X^-$ [$X^- = IO_3^-$ (Gholizadeh, Maleki *et al.*, 2011) and IO_4^- (Gholizadeh, Hojati *et al.*, 2011)] have been investigated. Structure determination of the title salt, $[C_5H_5NCH_2CH_2NC_5H_5]^{2+}.Cr_2O_7^{2-}$ (Fig. 1), was performed as a part of a project on the synthesis of a new hybrid compound containing an organic cation and an inorganic oxidant anion.

In the dication, two pyridinium moieties are *anti*-oriented with respect to one another similar to those observed in the 1,1′-(ethane-1,2-diyl)dipyridinium salts with iodate and periodate counter ions (Gholizadeh, Maleki *et al.*, 2011; Gholizadeh, Hojati *et al.*, 2011). In the dianion, each Cr^{VI} ion is in a slightly distorted tetrahedral coordination environment. The two pyridinium fragments in the cation and the two CrO_3 units in the anion are not symmetrically equivalent.

The Cr—O bonds (with lengths of 1.777 (2) & 1.790 (2) Å) in the Cr—O—Cr fragment are longer than the other Cr—O bonds (in the range of 1.611 (2) to 1.624 (2) Å). The bond lengths and angles in the dichromate anion are within the expected values in the reported dichromate salts (Lennartson & Håkansson, 2009; Averbuch-Pouchot *et al.*, 1984).

S2. Experimental

1,1′-(ethane-1,2-diyl)dipyridinium dibromide was prepared according to the procedure reported by Gholizadeh, Maleki *et al.*, 2011 and Gholizadeh, Hojati *et al.*, 2011.

Preparation of title salt: To a solution of 1,1′-(ethane-1,2-diyl)dipyridinium dibromide (10 mmol) in H_2O (25 ml), a solution of $K_2Cr_2O_7$ (10 mmol) in H_2O was added and stirred. After 1 h, the precipitate was filtered and washed with H_2O. Orange crystals, suitable for X-ray crystallography, were obtained from a solution of the title salt in H_2O at room temperature.

S3. Refinement

All H atoms were placed in their calculated positions and then refined using the riding model with atom—H lengths of 0.950 Å (CH) or 0.990 Å (CH_2). Isotropic displacement parameters for these atoms were set to 1.20 times U_{eq} of the parent atoms.

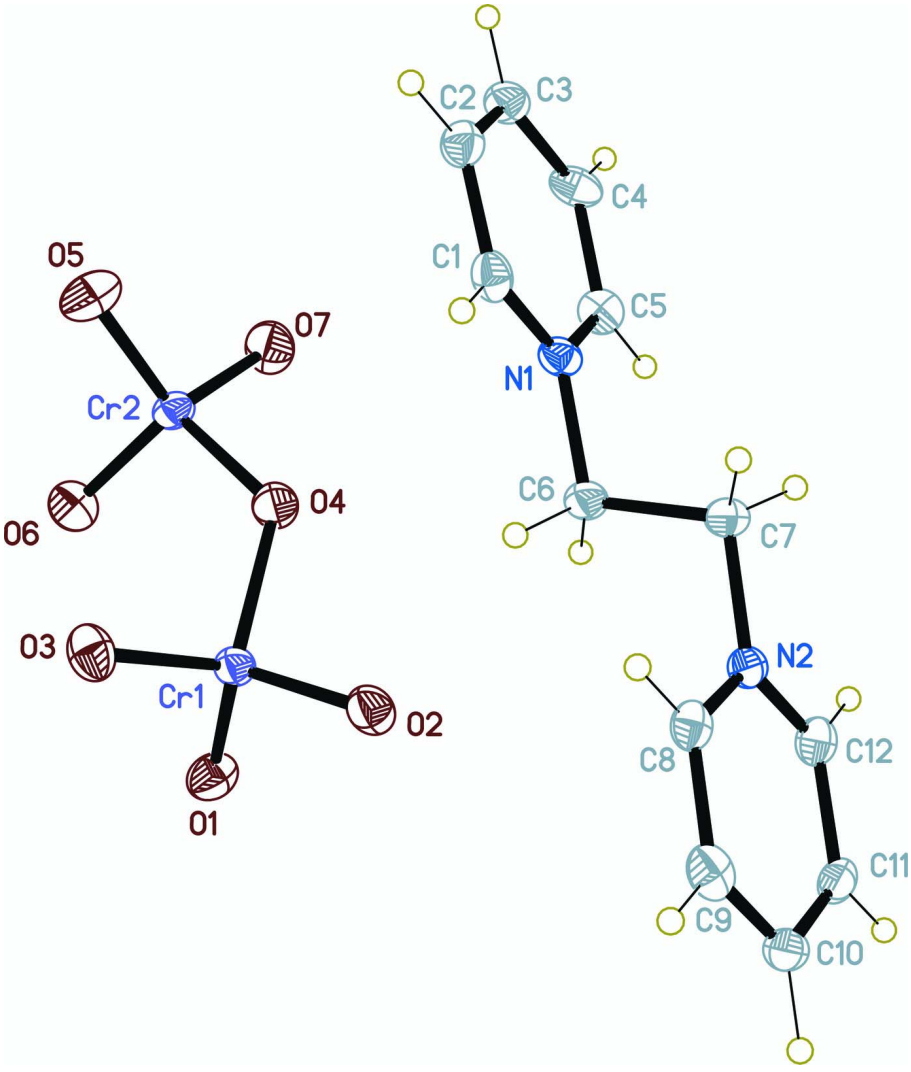

Figure 1

The molecular structure of the title compound. Ellipsoids are given at the 50% probability level.

1,1′-(Ethane-1,2-diyl)dipyridinium dichromate(VI)

Crystal data

$(C_{12}H_{14}N_2)[Cr_2O_7]$
$M_r = 402.25$
Monoclinic, $P2_1$
Hall symbol: P 2yb
$a = 8.2882$ (4) Å
$b = 9.0722$ (4) Å
$c = 10.0179$ (5) Å
$\beta = 91.882$ (1)°
$V = 752.86$ (6) Å^3
$Z = 2$

$F(000) = 408$
$D_x = 1.774$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 3255 reflections
$\theta = 2.5$–26.5°
$\mu = 1.48$ mm^{-1}
$T = 100$ K
Block, orange
0.22 × 0.15 × 0.15 mm

Data collection

Bruker APEXII CCD diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Bruker, 2005)
T_{min} = 0.737, T_{max} = 0.808
5446 measured reflections
2628 independent reflections
2546 reflections with $I > 2\sigma(I)$
R_{int} = 0.025
θ_{max} = 25.3°, θ_{min} = 2.0°
h = −9→9
k = −10→10
l = −12→12

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.027
$wR(F^2)$ = 0.067
S = 1.04
2628 reflections
209 parameters
1 restraint
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
$w = 1/[\sigma^2(F_o^2) + (0.0332P)^2 + 0.2849P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 0.47 e Å^{-3}
$\Delta\rho_{min}$ = −0.21 e Å^{-3}
Absolute structure: Flack (1983), 1163 Friedel pairs
Absolute structure parameter: 0.510 (19)

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 (Å^2)

	x	y	z	U_{iso}*/U_{eq}
Cr1	0.65538 (5)	0.04259 (4)	0.83530 (4)	0.01584 (13)
Cr2	0.35152 (5)	−0.03987 (5)	0.64354 (4)	0.01691 (13)
O1	0.7936 (2)	0.0097 (2)	0.7300 (2)	0.0250 (5)
O2	0.7001 (2)	0.1896 (2)	0.9204 (2)	0.0230 (5)
O3	0.6343 (3)	−0.0968 (2)	0.9351 (2)	0.0241 (5)
O4	0.4684 (3)	0.0804 (2)	0.7502 (2)	0.0255 (5)
O5	0.2177 (3)	−0.1216 (3)	0.7304 (2)	0.0312 (6)
O6	0.4687 (3)	−0.1613 (2)	0.5775 (2)	0.0251 (5)
O7	0.2677 (3)	0.0602 (3)	0.5277 (2)	0.0294 (5)
N1	0.2884 (3)	0.3914 (3)	0.6933 (2)	0.0177 (6)
N2	0.6566 (3)	0.5863 (3)	0.8330 (2)	0.0165 (6)
C1	0.1985 (4)	0.3157 (4)	0.7779 (3)	0.0207 (7)
H1	0.2448	0.2834	0.8608	0.025*
C2	0.0391 (4)	0.2840 (3)	0.7460 (3)	0.0213 (7)
H2	−0.0241	0.2291	0.8058	0.026*

C3	−0.0274 (4)	0.3334 (4)	0.6255 (3)	0.0218 (7)
H3	−0.1367	0.3118	0.6015	0.026*
C4	0.0658 (4)	0.4138 (4)	0.5407 (3)	0.0224 (7)
H4	0.0204	0.4503	0.4589	0.027*
C5	0.2256 (4)	0.4412 (4)	0.5753 (3)	0.0208 (7)
H5	0.2913	0.4949	0.5164	0.025*
C6	0.4621 (4)	0.4188 (3)	0.7282 (3)	0.0193 (7)
H6A	0.5255	0.4160	0.6461	0.023*
H6B	0.5033	0.3406	0.7891	0.023*
C7	0.4825 (3)	0.5674 (4)	0.7949 (3)	0.0192 (7)
H7A	0.4468	0.6467	0.7328	0.023*
H7B	0.4164	0.5724	0.8753	0.023*
C8	0.7158 (3)	0.5188 (3)	0.9456 (3)	0.0179 (6)
H8	0.6471	0.4605	0.9983	0.022*
C9	0.8761 (4)	0.5360 (4)	0.9823 (3)	0.0217 (6)
H9	0.9189	0.4901	1.0611	0.026*
C10	0.9750 (4)	0.6206 (4)	0.9038 (3)	0.0219 (7)
H10	1.0858	0.6328	0.9285	0.026*
C11	0.9116 (4)	0.6876 (4)	0.7886 (3)	0.0197 (7)
H11	0.9783	0.7456	0.7339	0.024*
C12	0.7493 (4)	0.6681 (4)	0.7551 (3)	0.0197 (7)
H12	0.7039	0.7129	0.6768	0.024*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Cr1	0.0149 (2)	0.0163 (3)	0.0162 (2)	0.0001 (2)	−0.00137 (17)	−0.0007 (2)
Cr2	0.0149 (2)	0.0192 (3)	0.0165 (2)	−0.0025 (2)	−0.00182 (17)	0.0006 (2)
O1	0.0229 (11)	0.0267 (13)	0.0256 (11)	−0.0042 (9)	0.0042 (9)	−0.0029 (10)
O2	0.0232 (12)	0.0214 (12)	0.0239 (13)	0.0036 (9)	−0.0064 (9)	−0.0039 (10)
O3	0.0295 (12)	0.0219 (12)	0.0210 (11)	0.0040 (10)	0.0034 (9)	0.0030 (10)
O4	0.0215 (11)	0.0208 (12)	0.0333 (13)	−0.0003 (9)	−0.0105 (9)	−0.0024 (10)
O5	0.0265 (13)	0.0378 (15)	0.0296 (13)	−0.0101 (11)	0.0055 (10)	−0.0017 (11)
O6	0.0278 (12)	0.0271 (13)	0.0206 (11)	0.0027 (10)	0.0020 (9)	−0.0025 (10)
O7	0.0308 (12)	0.0277 (13)	0.0289 (11)	0.0000 (10)	−0.0100 (9)	0.0036 (11)
N1	0.0158 (13)	0.0200 (14)	0.0172 (12)	0.0023 (11)	−0.0016 (10)	−0.0032 (11)
N2	0.0164 (13)	0.0161 (14)	0.0171 (12)	0.0004 (10)	0.0018 (10)	−0.0037 (11)
C1	0.0259 (17)	0.0168 (16)	0.0189 (15)	0.0023 (14)	−0.0047 (12)	−0.0011 (14)
C2	0.0220 (16)	0.0193 (16)	0.0227 (16)	−0.0006 (13)	0.0002 (12)	0.0022 (14)
C3	0.0180 (16)	0.0227 (17)	0.0242 (16)	0.0007 (13)	−0.0032 (12)	−0.0044 (14)
C4	0.0187 (16)	0.0319 (19)	0.0167 (14)	0.0066 (14)	−0.0010 (12)	0.0004 (14)
C5	0.0230 (16)	0.0236 (18)	0.0160 (14)	0.0010 (14)	0.0025 (11)	0.0007 (14)
C6	0.0151 (14)	0.0199 (16)	0.0226 (15)	0.0003 (12)	−0.0034 (12)	−0.0034 (13)
C7	0.0148 (15)	0.0204 (16)	0.0224 (15)	0.0001 (13)	0.0002 (11)	0.0009 (14)
C8	0.0228 (15)	0.0164 (16)	0.0147 (14)	−0.0005 (13)	0.0029 (11)	−0.0033 (12)
C9	0.0294 (16)	0.0213 (16)	0.0142 (13)	0.0052 (15)	−0.0021 (11)	−0.0054 (14)
C10	0.0174 (15)	0.0215 (17)	0.0267 (17)	0.0018 (14)	−0.0025 (12)	−0.0098 (15)
C11	0.0198 (15)	0.0161 (15)	0.0234 (17)	−0.0040 (12)	0.0037 (12)	−0.0034 (13)

C12	0.0241 (17)	0.0182 (16)	0.0169 (15)	−0.0010 (14)	0.0009 (12)	−0.0027 (13)

Geometric parameters (Å, º)

Cr1—O1	1.611 (2)	C3—H3	0.9500
Cr1—O2	1.620 (2)	C4—C5	1.380 (4)
Cr1—O3	1.624 (2)	C4—H4	0.9500
Cr1—O4	1.777 (2)	C5—H5	0.9500
Cr2—O5	1.612 (2)	C6—C7	1.511 (4)
Cr2—O7	1.613 (2)	C6—H6A	0.9900
Cr2—O6	1.624 (2)	C6—H6B	0.9900
Cr2—O4	1.790 (2)	C7—H7A	0.9900
N1—C1	1.337 (4)	C7—H7B	0.9900
N1—C5	1.353 (4)	C8—C9	1.376 (4)
N1—C6	1.491 (4)	C8—H8	0.9500
N2—C12	1.338 (4)	C9—C10	1.386 (4)
N2—C8	1.361 (4)	C9—H9	0.9500
N2—C7	1.491 (3)	C10—C11	1.392 (4)
C1—C2	1.379 (4)	C10—H10	0.9500
C1—H1	0.9500	C11—C12	1.387 (4)
C2—C3	1.385 (4)	C11—H11	0.9500
C2—H2	0.9500	C12—H12	0.9500
C3—C4	1.376 (5)		
O1—Cr1—O2	110.01 (11)	N1—C5—C4	119.8 (3)
O1—Cr1—O3	110.60 (11)	N1—C5—H5	120.1
O2—Cr1—O3	110.15 (11)	C4—C5—H5	120.1
O1—Cr1—O4	110.43 (11)	N1—C6—C7	110.2 (2)
O2—Cr1—O4	105.93 (10)	N1—C6—H6A	109.6
O3—Cr1—O4	109.62 (11)	C7—C6—H6A	109.6
O5—Cr2—O7	111.07 (12)	N1—C6—H6B	109.6
O5—Cr2—O6	109.80 (13)	C7—C6—H6B	109.6
O7—Cr2—O6	109.78 (12)	H6A—C6—H6B	108.1
O5—Cr2—O4	109.08 (11)	N2—C7—C6	108.0 (2)
O7—Cr2—O4	107.35 (11)	N2—C7—H7A	110.1
O6—Cr2—O4	109.72 (10)	C6—C7—H7A	110.1
Cr1—O4—Cr2	127.96 (12)	N2—C7—H7B	110.1
C1—N1—C5	121.2 (3)	C6—C7—H7B	110.1
C1—N1—C6	119.4 (2)	H7A—C7—H7B	108.4
C5—N1—C6	119.3 (3)	N2—C8—C9	119.3 (3)
C12—N2—C8	122.4 (2)	N2—C8—H8	120.4
C12—N2—C7	119.0 (2)	C9—C8—H8	120.4
C8—N2—C7	118.7 (2)	C8—C9—C10	119.6 (3)
N1—C1—C2	120.7 (3)	C8—C9—H9	120.2
N1—C1—H1	119.7	C10—C9—H9	120.2
C2—C1—H1	119.7	C9—C10—C11	119.9 (3)
C1—C2—C3	119.0 (3)	C9—C10—H10	120.1
C1—C2—H2	120.5	C11—C10—H10	120.1

C3—C2—H2	120.5	C12—C11—C10	118.8 (3)
C4—C3—C2	119.6 (3)	C12—C11—H11	120.6
C4—C3—H3	120.2	C10—C11—H11	120.6
C2—C3—H3	120.2	N2—C12—C11	120.0 (3)
C3—C4—C5	119.6 (3)	N2—C12—H12	120.0
C3—C4—H4	120.2	C11—C12—H12	120.0
C5—C4—H4	120.2		
O1—Cr1—O4—Cr2	62.94 (19)	C1—N1—C6—C7	−94.5 (3)
O2—Cr1—O4—Cr2	−177.98 (15)	C5—N1—C6—C7	86.8 (3)
O3—Cr1—O4—Cr2	−59.16 (19)	C12—N2—C7—C6	100.0 (3)
O5—Cr2—O4—Cr1	94.27 (18)	C8—N2—C7—C6	−79.6 (3)
O7—Cr2—O4—Cr1	−145.29 (16)	N1—C6—C7—N2	177.5 (2)
O6—Cr2—O4—Cr1	−26.04 (19)	C12—N2—C8—C9	0.7 (4)
C5—N1—C1—C2	1.1 (5)	C7—N2—C8—C9	−179.7 (3)
C6—N1—C1—C2	−177.6 (3)	N2—C8—C9—C10	−0.5 (5)
N1—C1—C2—C3	−0.8 (5)	C8—C9—C10—C11	0.0 (5)
C1—C2—C3—C4	−0.5 (5)	C9—C10—C11—C12	0.2 (5)
C2—C3—C4—C5	1.6 (5)	C8—N2—C12—C11	−0.5 (5)
C1—N1—C5—C4	0.0 (5)	C7—N2—C12—C11	179.9 (3)
C6—N1—C5—C4	178.7 (3)	C10—C11—C12—N2	0.1 (5)
C3—C4—C5—N1	−1.3 (5)		