

Bis(ethylenediammonium) dichromate oxalate

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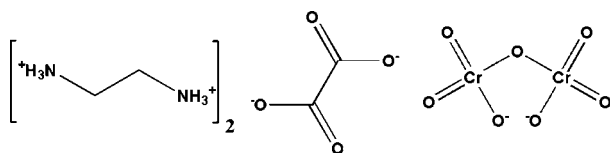
Received 6 May 2007; accepted 6 June 2007

Key indicators: single-crystal X-ray study; $T = 293$ K; mean $\sigma(\text{C}-\text{C}) = 0.004$ Å; disorder in main residue; R factor = 0.041; wR factor = 0.124; data-to-parameter ratio = 12.9.

The structure of the title compound, $[\text{NH}_3(\text{CH}_2)_2\text{NH}_3]_2\text{[Cr}_2\text{O}_7\text{](C}_2\text{O}_4)$, consists of inorganic columns of dichromate anions running along the c axis and organic layers of formula $\{[(\text{C}_2\text{H}_{10}\text{N}_2)_2\cdot\text{C}_2\text{O}_4]_n\}^{2n+}$ parallel to the (100) plane. The dichromate anion lies on a crystallographic twofold rotation axis and the oxalate anion on a centre of symmetry. Three of the four crystallographically independent O atoms of the dichromate anion are disordered, two over two sites and one over three sites; the site occupancy factors are approximately 0.65:0.35, 0.7:0.3 and 0.4:0.3:0.3. Structural cohesion is ensured by $\text{N}-\text{H}\cdots\text{O}$ hydrogen-bonding interactions, which form a three-dimensional framework.

Related literature

For general background, see: Khadhrani, Ben Smail & Driss (2006); Khadhrani, Ben Smail, Driss & Jouini (2006); Chebbi & Driss (2001, 2002*a,b*); Chebbi *et al.* (2000, 2003). For related structures, see: Chebbi & Driss (2004); Fossé & Brohan (1999); Fossé *et al.* (1998, 2001); Lorenzo-Luis *et al.* (1995); Martin-Zarza *et al.* (1995); Srinivasan *et al.* (2003). For related literature, see: Blessing (1986); Brown (1976).



Experimental

Crystal data

$(\text{C}_2\text{H}_{10}\text{N}_2)_2[\text{Cr}_2\text{O}_7](\text{C}_2\text{O}_4)$
 $M_r = 428.26$
Monoclinic, $C2/c$
 $a = 19.591$ (1) Å
 $b = 6.478$ (1) Å
 $c = 12.843$ (2) Å
 $\beta = 100.36$ (2)°

$V = 1603.3$ (4) Å³
 $Z = 4$
Mo $K\alpha$ radiation
 $\mu = 1.42$ mm⁻¹
 $T = 293$ (2) K
 $0.33 \times 0.27 \times 0.15$ mm

Data collection

Enraf-Nonius CAD-4
diffractometer
Absorption correction: ψ scan
(North *et al.*, 1968)
 $T_{\text{min}} = 0.638$, $T_{\text{max}} = 0.809$
2306 measured reflections

1757 independent reflections
1615 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.054$
2 standard reflections
frequency: 120 min
intensity decay: 1.0%

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.041$
 $wR(F^2) = 0.124$
 $S = 1.08$
1757 reflections
136 parameters

15 restraints
H-atom parameters constrained
 $\Delta\rho_{\text{max}} = 0.50$ e Å⁻³
 $\Delta\rho_{\text{min}} = -0.69$ e Å⁻³

Table 1

Hydrogen-bond geometry (Å, °).

| $D-\text{H}\cdots A$ | $D-\text{H}$ | $\text{H}\cdots A$ | $D\cdots A$ | $D-\text{H}\cdots A$ |
|--|--------------|--------------------|-------------|----------------------|
| $\text{N1}-\text{H1}\cdots\text{O5}^i$ | 0.89 | 1.96 | 2.828 (3) | 166 |
| $\text{N1}-\text{H2}\cdots\text{O1}$ | 0.89 | 2.02 | 2.844 (3) | 154 |
| $\text{N1}-\text{H3}\cdots\text{O1}^{ii}$ | 0.89 | 2.07 | 2.909 (4) | 156 |
| $\text{N2}-\text{H6}\cdots\text{O6}^{iii}$ | 0.89 | 1.94 | 2.813 (2) | 167 |
| $\text{N2}-\text{H7}\cdots\text{O5}^{iv}$ | 0.89 | 1.93 | 2.789 (2) | 162 |
| $\text{N2}-\text{H8}\cdots\text{O6}^v$ | 0.89 | 2.22 | 2.910 (2) | 134 |
| $\text{N2}-\text{H8}\cdots\text{O5}^i$ | 0.89 | 2.21 | 3.010 (2) | 149 |

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

Data collection: *CAD-4 EXPRESS* (Duisenberg, 1992; Macíček & Yordanov, 1992); cell refinement: *CAD-4 EXPRESS*; data reduction: *XCAD4* (Harms & Wocadlo, 1995); program(s) used to solve structure: *SHELXS97* (Sheldrick, 1990); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *DIAMOND* (Brandenburg, 1998); software used to prepare material for publication: *SHELXL97*.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: RZ2143).

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supplementary materials

Acta Cryst. (2007). E63, m1859-m1860 [doi:10.1107/S1600536807027808]

Bis(ethylenediammonium) dichromate oxalate

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Comment

Our recent investigations on organic chromates have led to the synthesis of several compounds using slow solvent evaporation at room temperature (Khadhrani, Ben Smail & Driss, 2006; Khadhrani, Ben Smail, Driss & Jouini, 2006; Chebbi & Driss, 2001, 2002*a*, 2002*b*, 2004; Chebbi *et al.*, 2000; Chebbi *et al.*, 2003) These materials result from the interaction between aqueous solutions of chromium(VI) oxide and organic molecules having at least one lone pair of electrons, such as amines and aminoalcohols. These structures have been characterized by single-crystal X-ray diffraction. All the synthesized complexes exhibit three-dimensional framework structures, structural cohesion being established by various kinds of hydrogen bonds. As a continuation of this work, we describe here the synthesis and crystal structure of a new organic oxalate dichromate $[\text{NH}_3(\text{CH}_2)_2\text{NH}_3]_2[\text{C}_2\text{O}_4][\text{Cr}_2\text{O}_7]$, (I).

The asymmetric unit of (I) contains one ethylenediammonium cation, half a dichromate anion and half an oxalate anion (Fig. 1). The crystal packing generates inorganic columns consisting of dichromate anions stacked along the *z* axis and organic layers of formula $[(\text{C}_2\text{H}_{10}\text{N}_2)_2\text{C}_2\text{O}_4]_n^{2n+}$ parallel to (100) plane, formed by ethylenediammonium and oxalate ions linked by N—H \cdots O(oxalate) hydrogen bonds (Fig. 2). There are two organic layers per unit cell at $x=1/4$ and $x=3/4$, while the inorganic groups provide the cohesion among the layers through N—H \cdots O(dichromate) hydrogen bonds. All hydrogen bonds in the structure (Table 1) are weak (Brown, 1976; Blessing, 1986).

The N—C and C—C bond lengths and the C—C—N angles within the cation are comparable with those observed for $[\text{NH}_3\text{-(CH}_2\text{)-NH}_3][\text{Cr}_2\text{O}_7]$ (Lorenzo-Luis *et al.*, 1995; Srinivasan *et al.*, 2003) and $[\text{NH}_3\text{-(CH}_2\text{)-NH}_3][\text{CrO}_4]$ (Chebbi & Driss, 2004; Srinivasan *et al.*, 2003).

The oxalate ion is centrosymmetric. The C—C and C—O distances are in good agreement with those observed in the unique organic oxalate dichromate published (Khadhrani, Ben Smail & Driss, 2006).

The dichromate anion possesses a twofold symmetry axis passing through atom O3. The Cr—O terminal bond lengths are in the range 1.538 (4)–1.712 (8) Å and the bridging Cr—O bonds are longer and in the range 1.760 (7)–1.810 (8) Å. These values are in good agreement with those usually found in organic dichromates (Fossé & Brohan, 1999; Fossé *et al.*, 1998; Fossé *et al.*, 2001). Atoms O2 and O4 of the dichromate anion are disordered over two positions separated by 1.351 (10) and 1.201 (12) Å, respectively. Atom O3 is disordered over three positions, two of which generated by the twofold rotation axis, separated by 0.935 (9) Å. This type of disorder has also been observed in the crystal structure of bis-dihexadecyldimethylammonium dichromate (Fossé & Brohan, 1999) and $(\text{Hdpam})_2\text{Cr}_2\text{O}_7$ (Martin-Zarza *et al.*, 1995).

Experimental

To a solution of CrO_3 (3.2 g) and $(\text{NH}_4)_2\text{C}_2\text{O}_4$ (1.2 g) in water (50 ml) $\text{C}_2\text{N}_2\text{H}_8$ (1 ml) was added under stirring. The reaction mixture was allowed to stand for a week at room temperature. Orange-red single crystals, suitable for X-ray analysis, were

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isolated on slow evaporation of the solvent. The presence of Cr, O, C, and N was confirmed by EDS (energy dispersive spectroscopy) on a scanning electron microscope.

Refinement

Atoms O2 and O4 of the dichromate anion are disordered over two positions with occupancies of (0.65/0.35) and (0.70/0.30), respectively. Atom O3 is disordered over three positions, two of which are generated by a crystallographic twofold rotation axis, with occupancies of 0.42 for the major component and of 0.29 for the minor components, respectively. All H atoms were placed in calculated positions and refined using a riding model with C—H = 0.97 Å, N—H = 0.89 Å, and with $U_{\text{iso}}(\text{H}) = 1.2 U_{\text{eq}}(\text{C})$ or $1.5 U_{\text{eq}}(\text{N})$.

Figures

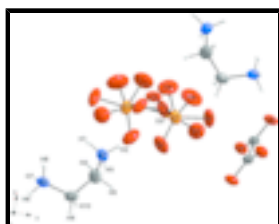


Fig. 1. The molecular structure of (I), with atom labels and 50% probability displacement ellipsoids. Symmetry codes: (a) $1.5 - x, 0.5 - y, -z$; (b) $1 - x, y, 0.5 - z$.

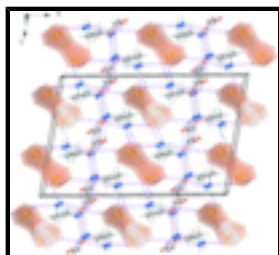
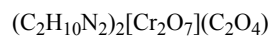


Fig. 2. Packing diagram of (I) viewed approximately along the *b* axis. Dashed lines indicate hydrogen bonds.

Bis(ethylenediammonium) dichromate oxalate

Crystal data



$M_r = 428.26$

Monoclinic, $C2/c$

Hall symbol: $-C 2yc$

$a = 19.591 (1) \text{ \AA}$

$b = 6.478 (1) \text{ \AA}$

$c = 12.843 (2) \text{ \AA}$

$\beta = 100.36 (2)^\circ$

$V = 1603.3 (4) \text{ \AA}^3$

$Z = 4$

$F_{000} = 880$

$D_x = 1.774 \text{ Mg m}^{-3}$

Mo $K\alpha$ radiation

$\lambda = 0.71073 \text{ \AA}$

Cell parameters from 25 reflections

$\theta = 10.0\text{--}10.9^\circ$

$\mu = 1.42 \text{ mm}^{-1}$

$T = 293 (2) \text{ K}$

Prism, orange-red

$0.33 \times 0.27 \times 0.15 \text{ mm}$

Data collection

| | |
|---|------------------------------------|
| Enraf–Nonius CAD-4 diffractometer | $R_{\text{int}} = 0.054$ |
| Radiation source: fine-focus sealed tube | $\theta_{\text{max}} = 27.0^\circ$ |
| Monochromator: graphite | $\theta_{\text{min}} = 2.1^\circ$ |
| $T = 293(2)$ K | $h = -24 \rightarrow 24$ |
| $\omega/2\theta$ scans | $k = -8 \rightarrow 2$ |
| Absorption correction: ψ scan (North <i>et al.</i> , 1968) | $l = 0 \rightarrow 16$ |
| $T_{\text{min}} = 0.638$, $T_{\text{max}} = 0.809$ | 2 standard reflections |
| 2306 measured reflections | every 120 min |
| 1757 independent reflections | intensity decay: 1.0% |
| 1615 reflections with $I > 2\sigma(I)$ | |

Refinement

| | |
|--|--|
| Refinement on F^2 | Secondary atom site location: difference Fourier map |
| Least-squares matrix: full | Hydrogen site location: inferred from neighbouring sites |
| $R[F^2 > 2\sigma(F^2)] = 0.041$ | H-atom parameters constrained |
| $wR(F^2) = 0.124$ | $w = 1/[\sigma^2(F_o^2) + (0.0716P)^2 + 2.7856P]$ |
| $S = 1.08$ | where $P = (F_o^2 + 2F_c^2)/3$ |
| 1757 reflections | $(\Delta/\sigma)_{\text{max}} < 0.001$ |
| 136 parameters | $\Delta\rho_{\text{max}} = 0.50 \text{ e } \text{\AA}^{-3}$ |
| 15 restraints | $\Delta\rho_{\text{min}} = -0.69 \text{ e } \text{\AA}^{-3}$ |
| Primary atom site location: structure-invariant direct methods | Extinction correction: none |

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 $F^2 > 2\sigma(F^2)$ 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 (\AA^2)

| | x | y | z | $U_{\text{iso}}^*/U_{\text{eq}}$ | Occ. (<1) |
|----|--------------|-------------|-------------|----------------------------------|-----------|
| Cr | 0.56119 (2) | 0.41517 (7) | 0.35029 (3) | 0.0375 (2) | |
| O1 | 0.53960 (14) | 0.2065 (4) | 0.4052 (2) | 0.0672 (7) | |

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| | | | | | |
|-----|--------------|-------------|--------------|-------------|------|
| O2 | 0.6161 (2) | 0.3377 (7) | 0.2763 (3) | 0.0661 (10) | 0.65 |
| O2' | 0.6416 (3) | 0.4485 (13) | 0.3638 (7) | 0.069 (2) | 0.35 |
| O3 | 0.5000 | 0.5311 (11) | 0.2500 | 0.054 (2) | 0.42 |
| O3' | 0.5280 (4) | 0.4497 (15) | 0.2147 (5) | 0.0471 (18) | 0.29 |
| O4 | 0.5936 (3) | 0.5807 (6) | 0.4300 (4) | 0.0844 (15) | 0.70 |
| O4' | 0.5329 (7) | 0.6223 (15) | 0.4136 (8) | 0.078 (3) | 0.30 |
| C1 | 0.75386 (11) | 0.3226 (3) | 0.04964 (14) | 0.0224 (4) | |
| O5 | 0.72688 (9) | 0.2589 (3) | 0.12602 (11) | 0.0309 (4) | |
| O6 | 0.78485 (10) | 0.4892 (2) | 0.04552 (12) | 0.0337 (4) | |
| N1 | 0.40427 (12) | 0.0641 (3) | 0.41953 (19) | 0.0397 (5) | |
| H1 | 0.3656 | 0.1386 | 0.4144 | 0.060* | |
| H2 | 0.4394 | 0.1460 | 0.4113 | 0.060* | |
| H3 | 0.4135 | 0.0046 | 0.4830 | 0.060* | |
| C2 | 0.39464 (16) | -0.0993 (4) | 0.3351 (3) | 0.0478 (7) | |
| H4 | 0.3761 | -0.0365 | 0.2674 | 0.057* | |
| H5 | 0.4394 | -0.1591 | 0.3303 | 0.057* | |
| N2 | 0.27317 (10) | -0.2017 (3) | 0.34349 (13) | 0.0276 (4) | |
| H6 | 0.2488 | -0.2975 | 0.3704 | 0.041* | |
| H7 | 0.2563 | -0.1863 | 0.2748 | 0.041* | |
| H8 | 0.2702 | -0.0824 | 0.3767 | 0.041* | |
| C3 | 0.34663 (15) | -0.2666 (4) | 0.3576 (2) | 0.0416 (6) | |
| H9 | 0.3503 | -0.3825 | 0.3111 | 0.050* | |
| H10 | 0.3614 | -0.3136 | 0.4299 | 0.050* | |

Atomic displacement parameters (\AA^2)

| | U^{11} | U^{22} | U^{33} | U^{12} | U^{13} | U^{23} |
|-----|-------------|-------------|-------------|---------------|---------------|--------------|
| Cr | 0.0342 (3) | 0.0480 (3) | 0.0272 (3) | -0.00468 (16) | -0.00284 (18) | 0.00259 (15) |
| O1 | 0.0624 (15) | 0.0668 (16) | 0.0741 (16) | -0.0276 (13) | 0.0170 (12) | 0.0129 (13) |
| O2 | 0.070 (3) | 0.066 (2) | 0.071 (2) | 0.003 (2) | 0.036 (2) | 0.008 (2) |
| O2' | 0.032 (3) | 0.085 (5) | 0.087 (5) | -0.012 (3) | 0.002 (3) | 0.044 (4) |
| O3 | 0.058 (6) | 0.027 (3) | 0.064 (6) | 0.000 | -0.028 (5) | 0.000 |
| O3' | 0.046 (4) | 0.065 (5) | 0.028 (3) | -0.024 (4) | 0.002 (3) | 0.006 (3) |
| O4 | 0.112 (4) | 0.064 (2) | 0.064 (2) | -0.024 (2) | -0.018 (3) | -0.0187 (18) |
| O4' | 0.099 (8) | 0.057 (5) | 0.076 (6) | 0.013 (5) | 0.008 (6) | -0.034 (4) |
| C1 | 0.0278 (10) | 0.0223 (9) | 0.0167 (8) | 0.0025 (7) | 0.0028 (7) | 0.0011 (7) |
| O5 | 0.0443 (9) | 0.0333 (8) | 0.0168 (7) | -0.0065 (7) | 0.0098 (6) | -0.0004 (6) |
| O6 | 0.0505 (11) | 0.0240 (8) | 0.0293 (8) | -0.0079 (7) | 0.0147 (7) | -0.0049 (6) |
| N1 | 0.0338 (11) | 0.0351 (11) | 0.0516 (13) | -0.0053 (8) | 0.0113 (10) | -0.0069 (9) |
| C2 | 0.0413 (15) | 0.0466 (15) | 0.0603 (18) | -0.0022 (12) | 0.0218 (13) | -0.0169 (13) |
| N2 | 0.0403 (10) | 0.0233 (8) | 0.0196 (8) | -0.0022 (7) | 0.0064 (7) | -0.0029 (6) |
| C3 | 0.0444 (14) | 0.0263 (11) | 0.0510 (15) | 0.0049 (10) | 0.0008 (12) | -0.0060 (10) |

Geometric parameters (\AA , $^\circ$)

| | | | |
|--------|-----------|---------------------|-----------|
| Cr—O4 | 1.538 (4) | C1—O6 | 1.244 (3) |
| Cr—O2' | 1.568 (6) | C1—O5 | 1.264 (2) |
| Cr—O1 | 1.615 (2) | C1—C1 ⁱⁱ | 1.569 (4) |

| | | | |
|-------------------------|-------------|---------------------------------------|-------------|
| Cr—O2 | 1.636 (4) | N1—C2 | 1.503 (3) |
| Cr—O4' | 1.712 (8) | N1—H1 | 0.8900 |
| Cr—O3' | 1.760 (7) | N1—H2 | 0.8900 |
| Cr—O3 | 1.762 (3) | N1—H3 | 0.8900 |
| Cr—O3 ⁱ | 1.810 (8) | C2—C3 | 1.497 (4) |
| O2—O2' | 1.351 (10) | C2—H4 | 0.9700 |
| O2'—O4 | 1.621 (11) | C2—H5 | 0.9700 |
| O3—O3 ⁱⁱ | 0.935 (9) | N2—C3 | 1.479 (3) |
| O3—O3' | 0.935 (9) | N2—H6 | 0.8900 |
| O3—Cr ⁱ | 1.762 (3) | N2—H7 | 0.8900 |
| O3'—O3 ⁱⁱ | 1.545 (15) | N2—H8 | 0.8900 |
| O3'—Cr ⁱ | 1.810 (8) | C3—H9 | 0.9700 |
| O4—O4' | 1.201 (12) | C3—H10 | 0.9700 |
| O4—Cr—O2' | 62.9 (4) | Cr ⁱ —O3—Cr | 129.5 (4) |
| O4—Cr—O1 | 113.7 (2) | O3—O3'—Cr | 74.7 (4) |
| O2'—Cr—O1 | 113.8 (3) | O3 ⁱⁱ —O3'—Cr | 66.0 (4) |
| O4—Cr—O2 | 111.4 (3) | O3—O3'—Cr ⁱ | 72.0 (5) |
| O2'—Cr—O2 | 49.8 (4) | O3 ⁱⁱ —O3'—Cr ⁱ | 62.7 (5) |
| O1—Cr—O2 | 104.35 (18) | Cr—O3'—Cr ⁱ | 126.5 (4) |
| O4—Cr—O4' | 42.9 (4) | O4'—O4—Cr | 76.3 (4) |
| O2'—Cr—O4' | 104.1 (6) | O4'—O4—O2' | 132.6 (6) |
| O1—Cr—O4' | 108.4 (4) | Cr—O4—O2' | 59.5 (3) |
| O2—Cr—O4' | 145.0 (5) | O4—O4'—Cr | 60.8 (4) |
| O4—Cr—O3' | 127.2 (3) | O6—C1—O5 | 126.25 (18) |
| O2'—Cr—O3' | 106.1 (4) | O6—C1—C1 ⁱⁱ | 117.5 (2) |
| O1—Cr—O3' | 117.3 (3) | O5—C1—C1 ⁱⁱ | 116.2 (2) |
| O2—Cr—O3' | 68.2 (3) | C2—N1—H1 | 109.5 |
| O4'—Cr—O3' | 106.1 (5) | C2—N1—H2 | 109.5 |
| O4—Cr—O3 | 109.5 (3) | H1—N1—H2 | 109.5 |
| O2'—Cr—O3 | 124.1 (3) | C2—N1—H3 | 109.5 |
| O1—Cr—O3 | 118.6 (2) | H1—N1—H3 | 109.5 |
| O2—Cr—O3 | 97.93 (17) | H2—N1—H3 | 109.5 |
| O4'—Cr—O3 | 77.1 (4) | C3—C2—N1 | 111.9 (2) |
| O3'—Cr—O3 | 30.8 (3) | C3—C2—H4 | 109.2 |
| O4—Cr—O3 ⁱ | 118.0 (3) | N1—C2—H4 | 109.2 |
| O2'—Cr—O3 ⁱⁱ | 154.3 (4) | C3—C2—H5 | 109.2 |
| O1—Cr—O3 ⁱⁱ | 89.9 (3) | N1—C2—H5 | 109.2 |
| O2—Cr—O3 ⁱⁱ | 116.8 (3) | H4—C2—H5 | 107.9 |
| O4'—Cr—O3 ⁱⁱ | 75.7 (5) | C3—N2—H6 | 109.5 |
| O3'—Cr—O3 ⁱⁱ | 51.2 (4) | C3—N2—H7 | 109.5 |
| O3—Cr—O3 ⁱⁱ | 30.3 (3) | H6—N2—H7 | 109.5 |
| O2'—O2—Cr | 62.5 (3) | C3—N2—H8 | 109.5 |
| O2—O2'—Cr | 67.7 (4) | H6—N2—H8 | 109.5 |
| O2—O2'—O4 | 123.6 (5) | H7—N2—H8 | 109.5 |
| Cr—O2'—O4 | 57.6 (3) | N2—C3—C2 | 113.7 (2) |

supplementary materials

| | | | |
|-------------------------------------|------------|-----------|-------|
| O3 ⁱ —O3—O3' | 111.3 (12) | N2—C3—H9 | 108.8 |
| O3 ⁱ —O3—Cr ⁱ | 74.5 (5) | C2—C3—H9 | 108.8 |
| O3'—O3—Cr ⁱ | 77.7 (5) | N2—C3—H10 | 108.8 |
| O3 ⁱ —O3—Cr | 77.7 (5) | C2—C3—H10 | 108.8 |
| O3'—O3—Cr | 74.5 (5) | H9—C3—H10 | 107.7 |

Symmetry codes: (i) $-x+1, y, -z+1/2$; (ii) $-x+3/2, -y+1/2, -z$.

Hydrogen-bond geometry (\AA , $^\circ$)

| $D-H\cdots A$ | $D-H$ | $H\cdots A$ | $D\cdots A$ | $D-H\cdots A$ |
|----------------------------------|-------|-------------|-------------|---------------|
| N1—H1 \cdots O5 ⁱ | 0.89 | 1.96 | 2.828 (3) | 166 |
| N1—H2 \cdots O1 | 0.89 | 2.02 | 2.844 (3) | 154 |
| N1—H3 \cdots O1 ⁱⁱⁱ | 0.89 | 2.07 | 2.909 (4) | 156 |
| N2—H6 \cdots O6 ^{iv} | 0.89 | 1.94 | 2.813 (2) | 167 |
| N2—H7 \cdots O5 ^v | 0.89 | 1.93 | 2.789 (2) | 162 |
| N2—H8 \cdots O6 ^{vi} | 0.89 | 2.22 | 2.910 (2) | 134 |
| N2—H8 \cdots O5 ⁱ | 0.89 | 2.21 | 3.010 (2) | 149 |

Symmetry codes: (i) $-x+1, y, -z+1/2$; (iii) $-x+1, -y, -z+1$; (iv) $-x+1, y-1, -z+1/2$; (v) $x-1/2, y-1/2, z$; (vi) $x-1/2, -y+1/2, z+1/2$.

Fig. 1

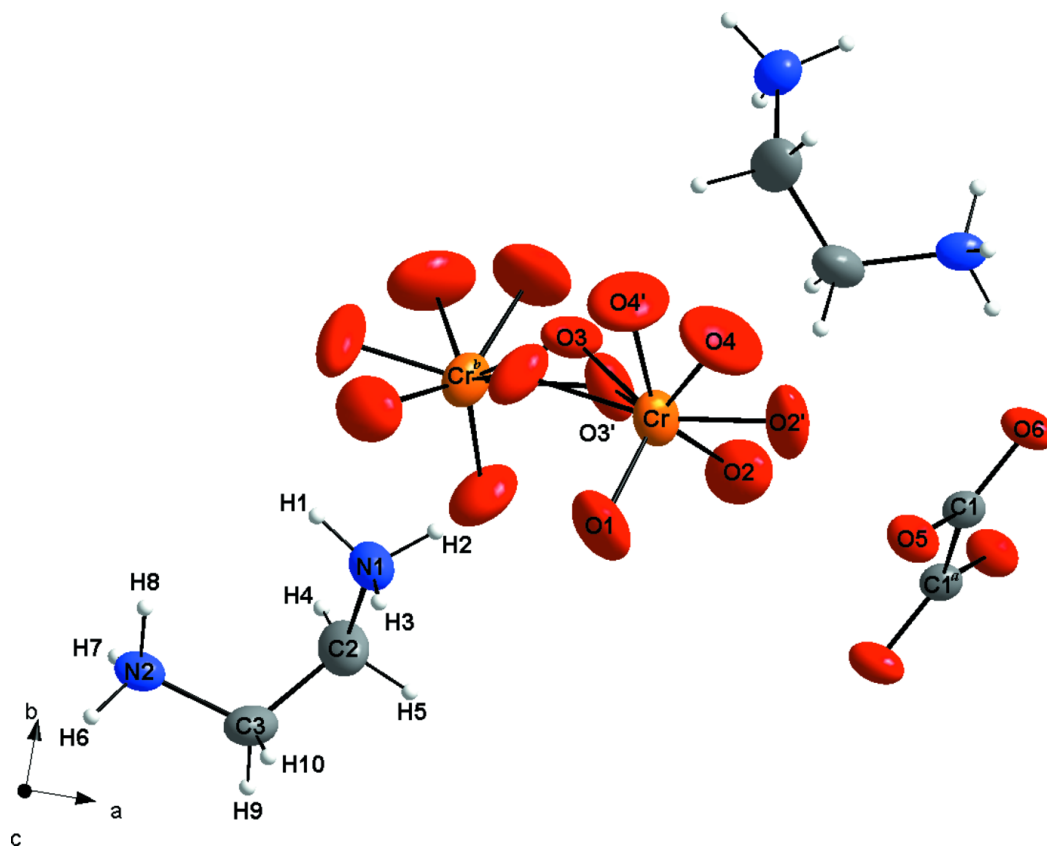


Fig. 2

