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Key indicators

Single-crystal X-ray study  
 T = 293 K  
 Mean  $\sigma(C-C)$  = 0.007 Å  
 R factor = 0.042  
 wR factor = 0.100  
 Data-to-parameter ratio = 16.2

For details of how these key indicators were automatically derived from the article, see <http://journals.iucr.org/e>.

Bis(2,6-dimethylpyridinium) dichromate

In the title compound,  $(C_7H_{10}N)_2[Cr_2O_7]$ , the pyridinium cations and dichromate anion are linked by N—H...O hydrogen bonds.  $\pi$ - $\pi$  stacking and C—H...O interactions play subordinate roles in forming the supramolecular structure.

Comment

The oxidation of primary alcohols to aldehydes and secondary alcohols to ketones with pyridinium chromate is a well established procedure in organic synthesis, owing mainly to the mild conditions employed (Corey & Schmidt, 1979; Kanemoto *et al.*, 1983; Corey *et al.*, 1985). Nowadays the development of newer chromium(VI) reagents (Sarma & Mahanti, 1991; Li & Li 1998; Yli-Kauhalauma *et al.*, 1998) for the oxidation of organic substrates continues to be of interest. In our laboratory, the title salt, (I), has been synthesized.

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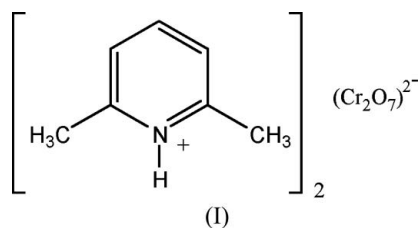
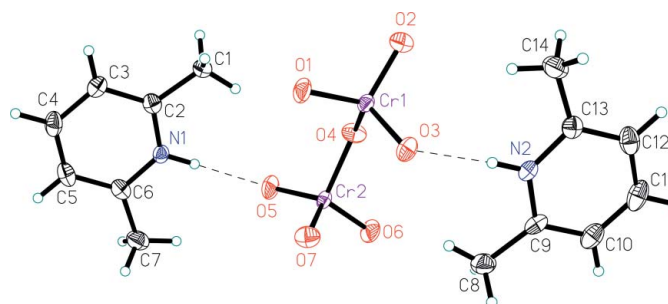
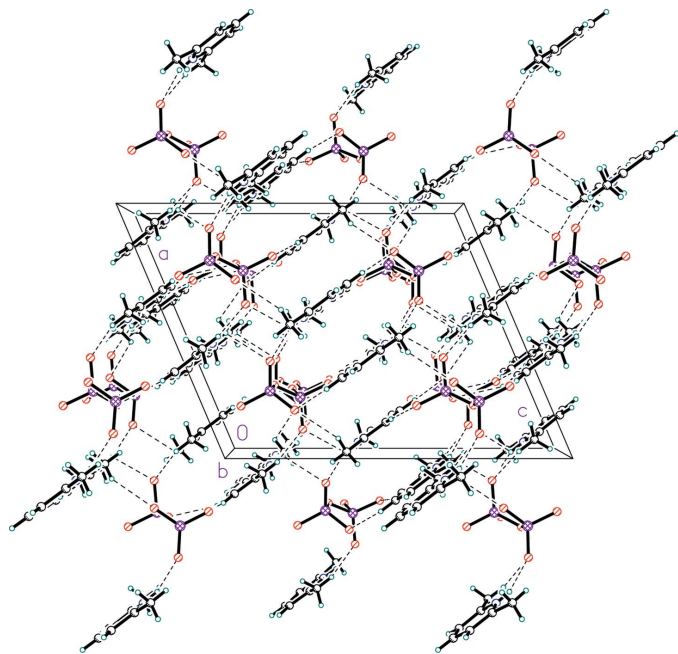


Fig. 1 shows the asymmetric unit of (I), consisting of two 2,6-dimethylpyridinium (DMPM) cations and one dichromate anion. The dichromate has longer bridging Cr—O bonds than terminal Cr—O bonds (Table 1). The shorter distances correspond to double bonds and the longer distances to single bonds. They are in good agreement with those found in bipyridinium dichromate (Martín-Zarza *et al.*, 1995), bis(-



**Figure 1**  
 The structure of the title compound, showing 40% probability displacement ellipsoids and the atom-numbering scheme. Hydrogen bonds are indicated by dashed lines.



**Figure 2**  
A packing diagram, viewed along the *b* axis. Hydrogen bonds are indicated by dashed lines.

octyltrimethylammonium) dichromate (Fossé *et al.*, 1998) and tetramethylammonium dichromate (Fossé *et al.*, 2001). The coordination geometry of each Cr atom is distorted tetrahedral.

The C–N–C angle of the pyridinium cation is always widened in comparison with the parent pyridine. For example, the C–N–C angles are 119.0 (3) and 120° for 2,6-dimethylpyridine (Bond *et al.*, 2001) and the 1:1 2,6-dimethylpyridine–urea complex (Lee & Wallwork, 1965), respectively. However, this angle is widened to 123.83 (2), 123.92 (17) and 124.56 (19)° in 2,6-dimethylpyridinium hydrogen phthalate, fumarate and nitrate (Jin *et al.* 2000, 2003; Pan *et al.*, 2001), respectively. A similar feature is also observed in the title salt (Table 1).

In the crystal structure, there are two pairs of bifurcated hydrogen bonds between the pyridinium and the dichromate ions (Table 2). Furthermore, the pyridinium and dichromate ions are linked by a number of C–H...O hydrogen bonds to form a network structure.

A packing diagram is shown in Fig. 2. The structure is stabilized by  $\pi$ – $\pi$  interactions between pyridinium cations. The relevant centroid–centroid separations are between DMPM (N1/C2–C6) at (*x*, *y*, *z*) and DMPM (N1/C2–C6) at ( $x + \frac{3}{2}$ ,  $y + \frac{1}{2}$ ,  $z + 1$ ) [3.471 (5) Å], and between DMPM (N2/C9–C13) at (*x*, *y*, *z*) and DMPM (N2/C9–C13) at ( $x + \frac{1}{2}$ ,  $y + \frac{1}{2}$ , *z*) [3.483 (5) Å].

## Experimental

2,6-Dimethylpyridine, CrO<sub>3</sub> and H<sub>2</sub>O in a molar ratio of 1:1:8 were mixed together. Crystals of the title salt were formed in the resulting solution by slow evaporation at 293 K for a week.

## Crystal data

(C<sub>7</sub>H<sub>10</sub>N)<sub>2</sub>[Cr<sub>2</sub>O<sub>7</sub>]  
*M<sub>r</sub>* = 432.32  
 Monoclinic, *C*<sub>2</sub>  
*a* = 13.600 (3) Å  
*b* = 8.3478 (17) Å  
*c* = 17.032 (3) Å  
 $\beta$  = 113.10 (3)°  
*V* = 1778.6 (7) Å<sup>3</sup>  
*Z* = 4

*D<sub>x</sub>* = 1.615 Mg m<sup>-3</sup>  
 Mo *K* $\alpha$  radiation  
 Cell parameters from 4141 reflections  
 $\theta$  = 3.5–27.5°  
 $\mu$  = 1.26 mm<sup>-1</sup>  
*T* = 293 (2) K  
 Prism, yellow  
 0.35 × 0.32 × 0.30 mm

## Data collection

Rigaku R-Axis RAPID CCD area-detector diffractometer  
 $\varphi$  and  $\omega$  scans  
 Absorption correction: multi-scan (ABSCOR; Higashi, 1995)  
*T<sub>min</sub>* = 0.650, *T<sub>max</sub>* = 0.685  
 9945 measured reflections

3660 independent reflections  
 2182 reflections with *I* > 2 $\sigma$ (*I*)  
*R<sub>int</sub>* = 0.030  
 $\theta_{max}$  = 27.5°  
*h* = -17 → 14  
*k* = -10 → 10  
*l* = -22 → 22

## Refinement

Refinement on *F*<sup>2</sup>  
*R*[*F*<sup>2</sup> > 2 $\sigma$ (*F*<sup>2</sup>)] = 0.042  
*wR*(*F*<sup>2</sup>) = 0.100  
*S* = 1.10  
 3660 reflections  
 226 parameters  
 H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.0473P)^2 + 2.3684P]$   
 where  $P = (F_o^2 + 2F_c^2)/3$   
 $(\Delta/\sigma)_{max} < 0.001$   
 $\Delta\rho_{max} = 0.40 \text{ e \AA}^{-3}$   
 $\Delta\rho_{min} = -0.43 \text{ e \AA}^{-3}$   
 Absolute structure: Flack (1983),  
 1501 Friedel pairs  
 Flack parameter: 0.00 (2)

**Table 1**

Selected geometric parameters (Å, °).

Cr1–O3	1.630 (3)	N2–C9	1.349 (5)
Cr1–O4	1.779 (3)	N2–C13	1.361 (5)
Cr2–O7	1.606 (3)	C1–C2	1.489 (5)
Cr2–O4	1.793 (3)	C2–C3	1.374 (6)
N1–C2	1.344 (5)	C3–C4	1.390 (6)
N1–C6	1.352 (5)	C4–C5	1.368 (7)
O2–Cr1–O1	110.25 (15)	O6–Cr2–O5	110.30 (16)
O2–Cr1–O3	109.34 (15)	O7–Cr2–O4	109.07 (14)
O1–Cr1–O3	109.57 (16)	O6–Cr2–O4	107.24 (14)
O2–Cr1–O4	107.06 (15)	O5–Cr2–O4	108.31 (15)
O1–Cr1–O4	110.23 (14)	Cr1–O4–Cr2	127.95 (16)
O3–Cr1–O4	110.36 (13)	C2–N1–C6	124.6 (3)
O7–Cr2–O6	110.66 (17)	C9–N2–C13	124.9 (4)
O7–Cr2–O5	111.14 (16)		

**Table 2**

Hydrogen-bond geometry (Å, °).

<i>D</i> –H... <i>A</i>	<i>D</i> –H	H... <i>A</i>	<i>D</i> ... <i>A</i>	<i>D</i> –H... <i>A</i>
N1–H1...O5	0.86	1.96	2.779 (6)	160
N1–H1...O3 <sup>i</sup>	0.86	2.45	2.865 (5)	110
N2–H2...O3	0.86	1.89	2.719 (7)	162
N2–H2...O5 <sup>ii</sup>	0.86	2.58	2.973 (5)	110
C1–H1A...O5	0.96	2.57	3.364 (5)	141
C3–H3...O1 <sup>iii</sup>	0.93	2.53	3.385 (5)	153
C4–H4...O7 <sup>iii</sup>	0.93	2.50	3.164 (5)	128
C5–H5...O1 <sup>iv</sup>	0.93	2.43	3.332 (5)	163
C7–H7B...O7 <sup>i</sup>	0.96	2.58	3.412 (7)	146
C8–H8A...O3	0.96	2.42	3.246 (5)	143
C10–H10...O4 <sup>v</sup>	0.93	2.49	3.325 (5)	149
C11–H11...O2 <sup>v</sup>	0.93	2.54	3.361 (6)	147
C12–H12...O6 <sup>vi</sup>	0.93	2.40	3.257 (5)	153
C14–H14B...O7 <sup>vii</sup>	0.96	2.58	3.152 (5)	119

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

All H atoms were placed in calculated positions and allowed to ride at distances of 0.93 (aromatic), 0.86 (NH) and 0.96 Å (methyl) from their parent atoms, with  $U_{\text{iso}}(\text{H})$  values of 1.2–1.5 times  $U_{\text{eq}}(\text{C,N})$ .

Data collection and cell refinement: *PROCESS-AUTO* (Rigaku, 2001); data reduction: *CrystalStructure* (Rigaku, 2003); program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics and publication material: *SHELXTL* (Bruker, 1998).

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