Crystal structure of bis(2,9-dimethyl-1,10-phenanthrolinium) dichromate dihydrate, $[C_{14}H_{13}N_2]_2[Cr_2O_7] \cdot 2H_2O$

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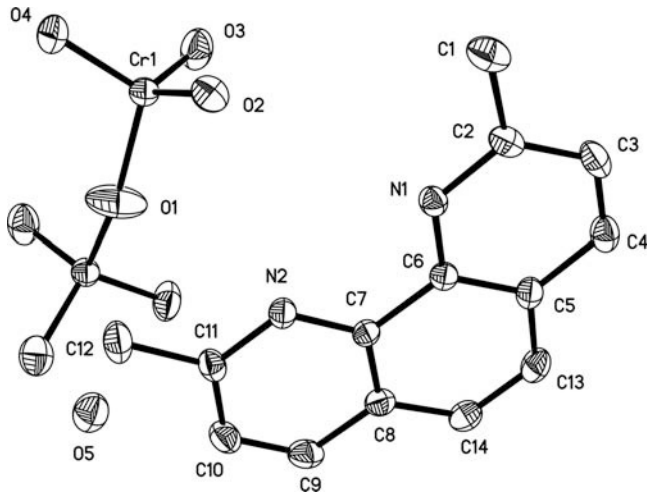

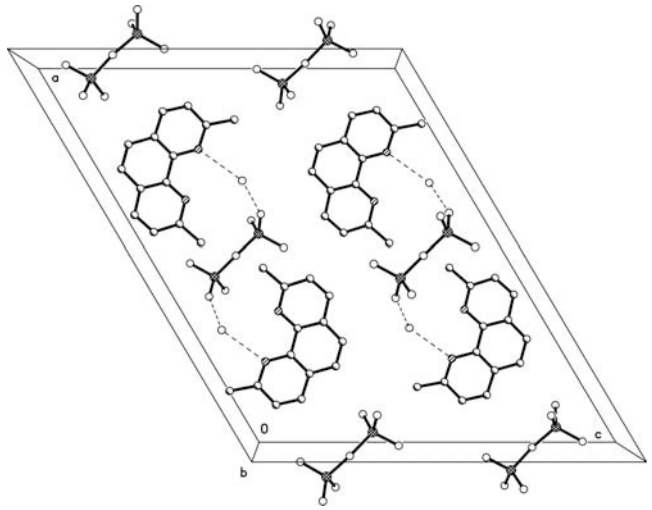

Abstract

$C_{28}H_{30}Cr_2N_4O_9$, monoclinic, *C*12/*c*1 (no. 15), a = 24.268(2) Å, b = 6.8564(8) Å, c = 19.956(2) Å, β = 120.600(2)°, V = 2858.2 Å^3, Z = 4, $R_{gt}(F)$ = 0.039, $wR_{ref}(F^2)$ = 0.112, T = 298 K.

Source of material

All commercially obtained reagent-grade chemicals were used without further purification. A mixture of $NiCl_2$ (0.1 mmol, 0.013 g), 2,9-dimethyl-1,10-phenanthroline (0.1 mmol, 0.02 g), and K_2CrO_4 (0.1 mmol, 0.02 g) were added into 20 mL water with 20 % (*v*/*v*) methanol and heated for 8 h at 140 °C. The solution was obtained by filtration after cooling the reaction to room temperature. Yellow prism-like single crystals suitable for X-ray measurements were obtained after a few weeks. In the preparation, the nickel chloride did not participate in coordination, but it may play a role in the steric hindrance and is conductive to the formation of title compound.

Discussion

2,9-Dimethyl-1,10-phenanthroline, which is the parent for important class chelating agents, has been widely used in the construction of supramolecular architectures. Some 2,9-dimethyl-1,10-phenanthroline complexes have been synthesized and reported [1,2].

The title crystal structure was build up of one dichromate anion, two 2,9-dimethyl-1,10-phenanthrolinium cations and two free water molecules (figure, top). The Cr atom is four-coordinated by O1, O2, O3 and O4 atoms forming an tetrahedron. Two tetrahedra share a vertex atom O1. The mean Cr—O bond lengths are similar to the reported earlier [3]. The torsion angles of O4–Cr1–O1–Cr1′, O3–Cr1–O1–Cr1′ are 84.5(1)° and –36.7(1)°, respectively. Through hydrogen bonding, van der Waals forces, π-π stacking, a two-dimensional layered structure is formed (figure, bottom). The free water molecules are linked by intermolecular O–H···O and O–H···N hydrogen bonds.

Table 1. Data collection and handling.

Crystal:	yellow block, size 0.34 × 0.40 × 0.45 mm
Wavelength:	Mo K_α radiation (0.71073 Å)
μ:	8.22 cm^{-1}
Diffractometer, scan mode:	Bruker SMART CCD, φ/ω
$2\theta_{max}$:	50.02°
$N(hkl)_{measured}$, $N(hkl)_{unique}$:	6805, 2489
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\ \sigma(I_{obs})$, 1765
$N(param)_{refined}$:	198
Programs:	SHELXS-97 [4], SHELXL-97 [5], SHELXTL [6]

Table 2. Atomic coordinates and displacement parameters (in Å^2).

Atom	Site	Occ.	x	y	z	U_{iso}
H(1)	8*f*		0.2637	0.3553	0.6624	0.036
H(1A)	8*f*	0.50	0.1996	0.3513	0.5339	0.079
H(1B)	8*f*	0.50	0.1356	0.4656	0.5065	0.079
H(1C)	8*f*	0.50	0.1358	0.2370	0.5072	0.079
H(1D)	8*f*	0.50	0.1144	0.3513	0.4979	0.079
H(1E)	8*f*	0.50	0.1784	0.2370	0.5253	0.079
H(1F)	8*f*	0.50	0.1782	0.4656	0.5246	0.079
H(3)	8*f*		0.0822	0.3550	0.5939	0.051

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Table 2. Continued.

Atom	Site	Occ.	*x*	*y*	*z*	U_{iso}
H(4)	8*f*		0.1064	0.3571	0.7203	0.047
H(9)	8*f*		0.4129	0.3461	0.9964	0.052
H(10)	8*f*		0.4858	0.3391	0.9560	0.052
H(12A)	8*f*		0.4589	0.2481	0.7757	0.076
H(12B)	8*f*		0.5112	0.3349	0.8558	0.076

Table 2. Continued.

Atom	Site	Occ.	*x*	*y*	*z*	U_{iso}
H(12C)	8*f*		0.4662	0.4747	0.7874	0.076
H(13)	8*f*		0.1906	0.3555	0.8644	0.049
H(14)	8*f*		0.2951	0.3540	0.9589	0.049
H(5A)	8*f*		0.6649	0.4583	0.8873	0.06(1)
H(5B)	8*f*		0.6615	0.2612	0.8770	0.07(1)

Table 3. Atomic coordinates and displacement parameters (in $Å^2$).

Atom	Site	*x*	*y*	*z*	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
Cr(1)	8*f*	0.44385(2)	0.85137(8)	0.65450(3)	0.0326(3)	0.0416(4)	0.0291(3)	–0.0007(3)	0.0145(2)	–0.0015(3)
N(1)	8*f*	0.2338(1)	0.3545(4)	0.6735(1)	0.027(1)	0.031(2)	0.034(1)	0.003(1)	0.015(1)	0.001(1)
N(2)	8*f*	0.3605(1)	0.3544(4)	0.7859(1)	0.030(1)	0.030(2)	0.037(2)	–0.000(1)	0.019(1)	0.000(1)
O(1)	4*e*	½	0.7824(7)	¾	0.105(3)	0.071(3)	0.033(2)	0	0.000(2)	0
O(2)	8*f*	0.3938(1)	0.6744(4)	0.6142(1)	0.046(1)	0.055(2)	0.056(2)	–0.011(1)	0.025(1)	–0.010(1)
O(3)	8*f*	0.4065(1)	1.0420(4)	0.6555(2)	0.055(2)	0.049(2)	0.083(2)	0.005(1)	0.043(2)	–0.004(2)
O(4)	8*f*	0.4795(1)	0.8925(4)	0.6075(1)	0.046(1)	0.081(2)	0.056(2)	–0.008(1)	0.034(1)	–0.008(1)
O(5)	8*f*	0.6871(1)	0.3570(4)	0.8929(2)	0.056(2)	0.052(2)	0.058(2)	0.000(2)	0.036(1)	0.001(1)
C(1)	8*f*	0.1598(2)	0.3517(6)	0.5334(2)	0.048(2)	0.059(3)	0.036(2)	0.003(2)	0.011(2)	0.003(2)
C(2)	8*f*	0.1730(2)	0.3535(5)	0.6153(2)	0.035(2)	0.031(2)	0.033(2)	0.002(2)	0.010(1)	0.000(2)
C(3)	8*f*	0.1249(1)	0.3548(5)	0.6338(2)	0.025(2)	0.039(2)	0.050(2)	0.002(2)	0.009(2)	–0.001(2)
C(4)	8*f*	0.1392(2)	0.3558(5)	0.7092(2)	0.031(2)	0.037(2)	0.052(2)	0.001(2)	0.023(2)	–0.002(2)
C(5)	8*f*	0.2031(1)	0.3549(5)	0.7701(2)	0.031(2)	0.030(2)	0.043(2)	–0.001(1)	0.021(2)	0.000(2)
C(6)	8*f*	0.2506(1)	0.3545(4)	0.7502(2)	0.029(2)	0.023(2)	0.032(2)	0.001(2)	0.017(1)	0.000(2)
C(7)	8*f*	0.3170(1)	0.3535(4)	0.8093(2)	0.029(2)	0.022(2)	0.034(2)	–0.001(1)	0.016(1)	0.002(1)
C(8)	8*f*	0.3332(1)	0.3524(5)	0.8873(2)	0.037(2)	0.031(2)	0.032(2)	–0.001(2)	0.016(1)	0.002(2)
C(9)	8*f*	0.3992(2)	0.3475(5)	0.9435(2)	0.046(2)	0.044(2)	0.031(2)	–0.001(2)	0.013(2)	0.003(2)
C(10)	8*f*	0.4423(2)	0.3450(5)	0.9194(2)	0.030(2)	0.045(2)	0.043(2)	–0.000(2)	0.010(2)	0.002(2)
C(11)	8*f*	0.4219(1)	0.3510(5)	0.8396(2)	0.026(2)	0.028(2)	0.046(2)	–0.001(1)	0.015(2)	–0.002(2)
C(12)	8*f*	0.4687(2)	0.3523(6)	0.8122(2)	0.036(2)	0.050(2)	0.073(3)	0.001(2)	0.032(2)	–0.001(2)
C(13)	8*f*	0.2219(2)	0.3548(5)	0.8507(2)	0.044(2)	0.044(2)	0.049(2)	–0.001(2)	0.034(2)	–0.000(2)
C(14)	8*f*	0.2840(2)	0.3538(5)	0.9069(2)	0.049(2)	0.045(2)	0.035(2)	–0.001(2)	0.026(2)	0.001(2)

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