Hexaaquanickel(II) dichromate bis(hexamethylenetetramine) monohydrate

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Key indicators

Single-crystal X-ray study
$T = 293$ K
Mean σ(Cr–O) = 0.004 Å
Disorder in main residue
R factor = 0.048
wR factor = 0.130
Data-to-parameter ratio = 11.8

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

A novel compound consisting of a hydrated metal cation, an organic molecule, an inorganic complex anion and water of crystallization has been synthesized and is found to crystallize in the monoclinic space group $P2_1/c$. The structure of the title compound, $[Ni(H_2O)_6][Cr_2O_7]\cdot2C_6H_{12}N_4\cdot H_2O$, contains octahedrally coordinated nickel ions and discrete dichromate anions. The hexamethylenetetramine molecules have no direct coordination to the metal ions. There are two cations, two anions, four tetramine molecules and two uncoordinated water molecules in the asymmetric unit. The structure is stabilized by extensive intra- and intermolecular hydrogen bonding.

Comment

It has been shown that hexamethylenetetramine helps to stabilize transition metal dichromate salts (Bronowska *et al.*, 2002) and we intend to use this effect to synthesize organic–inorganic hybrids. The title compound, (I), was formed during this attempt. Fig. 1 shows the components of the asymmetric unit. The packing diagram in Fig. 2 shows how the organic and inorganic moieties are packed together in the crystal structure. The organic molecule and both coordinated and uncoordinated water molecules are held together by several strong O—H···N hydrogen bonds (Table 1). One of the dichromate ions is disordered and hence a detailed analysis of intermolecular interactions cannot be made. It is, however, noted that a set of strong O—H···O hydrogen bonds link both the solvent and coordinated water molecules with the dichromate ions.
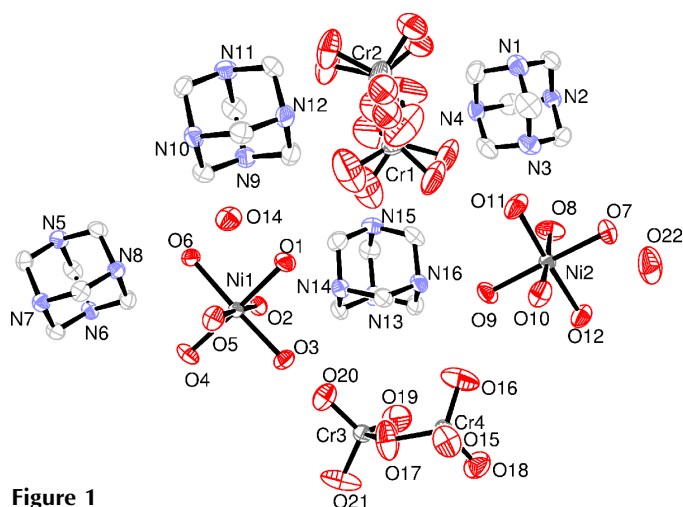

(I)
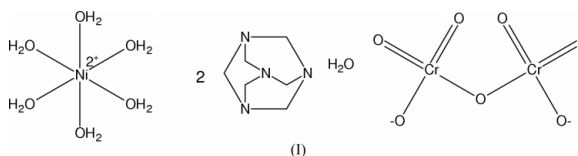
Experimental

The title compound was prepared by mixing aqueous solutions of nickel nitrate hexahydrate, ammonium dichromate and hexamethylenetetramine in the molar ratio 1:1:2. A bright yellow–orange precipitate was immediately produced. Crystals were obtained by complete evaporation of the solvent from an aqueous solution of the product.

Crystal data

$[Ni(H_2O)_6][Cr_2O_7]\cdot2C_6H_{12}N_4\cdot H_2O$	$D_x = 1.741$ Mg m^{-3}
$M_r = 681.19$	Mo $K\alpha$ radiation
Monoclinic, $P2_1/c$	Cell parameters from 765
$a = 27.976$ (5) Å	reflections
$b = 13.333$ (3) Å	$\theta = 1.5–25.5°$
$c = 14.070$ (3) Å	$\mu = 1.62$ mm^{-1}
$\beta = 97.896$ (3)°	$T = 293$ (2) K
$V = 5198.4$ (19) Å^3	Prism, red
$Z = 8$	$0.3 \times 0.2 \times 0.2$ mm

Figure 1
The asymmetric unit of the title compound, with 50% probability ellipsoids. C atoms and disordered O atoms are not labelled, and H atoms have been omitted.

Data collection

Bruker SMART CCD area-detector diffractometer	9189 independent reflections
	7533 reflections with $I > 2\sigma(I)$
φ and ω scans	$R_{int} = 0.036$
Absorption correction: multi-scan ($SADABS$; Sheldrick, 1996)	$\theta_{max} = 25.0°$
	$h = -33 \rightarrow 32$
$T_{min} = 0.623$, $T_{max} = 0.738$	$k = -15 \rightarrow 15$
37222 measured reflections	$l = -16 \rightarrow 16$

Refinement

Refinement on F^2	H atoms treated by a mixture of independent and constrained refinement
$R[F^2 > 2\sigma(F^2)] = 0.048$	
$wR(F^2) = 0.130$	
$S = 1.34$	$w = 1/[\sigma^2(F_o^2) + (0.0611P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
9189 reflections	
779 parameters	$(\Delta/\sigma)_{max} = 0.001$
	$\Delta\rho_{max} = 1.09$ e Å^{-3}
	$\Delta\rho_{min} = -0.77$ e Å^{-3}

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

$D—H\cdots A$	$D—H$	$H\cdots A$	$D\cdots A$	$D—H\cdots A$
O4—H53···N6[i]	0.72 (5)	2.16 (5)	2.874 (4)	177 (3)
O11—H64···N4[ii]	0.79 (5)	2.01 (5)	2.782 (6)	166 (4)
O10—H73···N15[ii]	0.82 (5)	2.34 (5)	2.982 (5)	135 (4)
O1—H58···N13[ii]	0.82 (5)	2.06 (5)	2.862 (5)	168 (5)
O12—H61···N12[iii]	0.81 (5)	1.95 (5)	2.710 (5)	156 (4)
O7—H63···N2[iv]	0.73 (5)	2.06 (5)	2.795 (4)	176 (5)
O22—H70···N1[v]	0.62 (6)	2.22 (6)	2.833 (6)	172 (6)

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

All H atoms bonded to carbon were placed in idealized positions (C—H = 0.93–0.97 Å) and constrained to ride on their parent atoms.
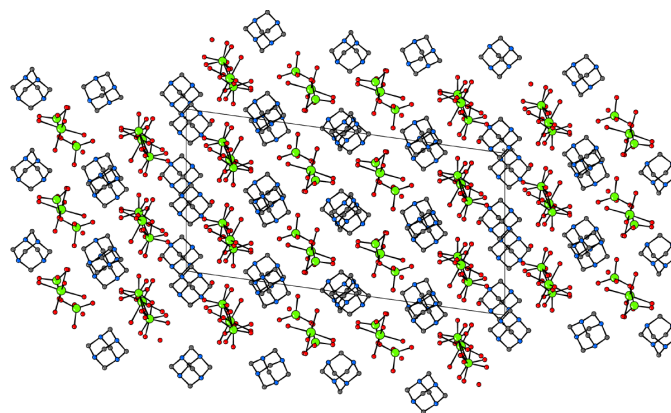
Figure 2
Packing diagram of the title compound, viewed down the c axis. H atoms have been omitted.

The other H atoms were located in a difference Fourier map and were refined with isotropic displacement parameters fixed at 0.05 Å^2. One of the dichromate anions is disordered, with common Cr-atom positions for the two components. Each of the O atoms was split over two sites with equal displacement parameters. The occupancy factors for the two components refined to 0.688 (6):0.312 (6). The largest residual electron density peak is 1.71 Å from O29, one of the atoms of the disordered anion.

Data collection: *SMART* (Bruker, 1998); cell refinement: *SMART*; data reduction: *SAINT* (Bruker, 1998); program(s) used to solve structure: *SIR*92 (Altomare *et al.*, 1993); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); molecular graphics: *ORTEP*-3 *for Windows* (Farrugia, 1997) and *CAMERON* (Watkin *et al.*, 1993); software used to prepare material for publication: *PLATON* (Spek, 1990).

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