Acta Crystallographica Section C
Crystal Structure Communications
ISSN 0108-2701

Disodium chromium(III) hexamolybdoiodate(VII) 24-hydrate, $Na_2Cr[IMo_6O_{24}]\cdot 24H_2O$

Cristina Rosu[a] and Timothy J. R. Weakley[b]*

[a]Department of Inorganic Chemistry, Babes-Bolyai University, RO-3400 Cluj-Napoca, Romania, and [b]Department of Chemistry, University of Oregon, Eugene, OR 97403-1253, USA
Correspondence e-mail: tweakley@oregon.uoregon.edu

Received 27 March 2000
Accepted 5 April 2000

Data validation number: IUC0000096

The title compound can be formulated as $[Cr(H_2O)_6]$-$[Na_2(H_2O)_{10}][IMo_6O_{24}]\cdot 8H_2O$. The anion has the I atom on an inversion centre and has close to $\bar{3}m$ symmetry, with I—O bond lengths in the range 1.881–1.890 (2) Å and Mo—O bond lengths in the ranges 1.697 (3)–1.714 (3), 1.915 (2)–1.948 (2) and 2.317 (2)–2.357 (2) Å.

Comment

This work forms part of an investigation into the interaction of transition metal cations and polyoxometalate anions. The $[IMo_6O_{24}]^{5-}$ anion lies on a crystal inversion center, in an orientation such that the cell is pseudo-body-centered. Its non-crystallographic symmetry is close to $\bar{3}m$ and its dimensions are very similar to those of the anion in $CoNa_3$-$[IMo_6O_{24}]\cdot 14H_2O$ (Rosu & Dickman, 1999) and in $K_5[IMo_6O_{24}]\cdot 5H_2O$ (Kondo *et al.*, 1980). The Mo atoms are coplanar to within 0.007 (1) Å. Whereas in $CoNa_3$-$[IMo_6O_{24}]\cdot 14H_2O$, the Co^{2+} and Na^+ cations are coordinated to the anion, the Na^+ ions in the present structure are present in discrete centrosymmetric $[Na_2(H_2O)_{10}]^{2+}$ units, and despite the preparation at 373 K, O atoms of the anions have not replaced water in the kinetically inert $[Cr(H_2O)_6]^{3+}$ groups.

Experimental

The pH of a slurry containing 15 g $Na_5[IMo_6O_{24}]\cdot 16.5H_2O$ (Rosu & Dickman, 1999) and 4 g $Cr(NO_3)_3\cdot 9H_2O$ in 100 ml water was adjusted to 6. The mixture was heated to reflux for 30 min and filtered while hot. The filtrate was allowed to evaporate at room temperature for 3 d to give 11.7 g of violet crystals. Found (calculated) for $CrNa_2[IMo_6O_{24}]\cdot 24H_2O$: Na 2.8 (2.8), Cr 3.2 (3.2), Mo 35.5 (35.6), H_2O 26.7% (26.7%). The crystal used for data collection was glued to a glass fibre.

Crystal data

$Na_2Cr[IMo_6O_{24}]\cdot 24H_2O$
M_r = 1616.87
Monoclinic, $P2_1/n$
a = 10.3829 (7) Å
b = 14.0871 (12) Å
c = 14.9994 (14) Å
β = 102.931 (9)°
V = 2138.2 (3) Å^3
Z = 2
D_x = 2.511 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 25 reflections
θ = 14.0–14.9°
μ = 2.82 mm^{-1}
T = 295 K
Block, violet
0.29 × 0.23 × 0.18 mm

Data collection

Enraf–Nonius CAD-4 diffractometer
ω–2θ scans
Absorption correction: ψ scan (North *et al.*, 1968)
T_{min} = 0.480, T_{max} = 0.545
6570 measured reflections
6210 independent reflections
4697 reflections with $I > \sigma(I)$
R_{int} = 0.011
θ_{max} = 30.0°
$h = -14 \rightarrow 14$
$k = 0 \rightarrow 19$
$l = -20 \rightarrow 21$
3 standard reflections
every 300 reflections
intensity decay: none

Refinement

Refinement on F^2
$R(F)$ = 0.034
$wR(F^2)$ = 0.050
S = 1.86
6210 reflections
276 parameters
H atoms not refined
$w = 4F_o^2/[\sigma^2(I) + (0.020I)^2]$
$(\Delta/\sigma)_{max}$ = 0.022
$\Delta\rho_{max}$ = 1.21 e Å^{-3} (1.07 Å from I)
$\Delta\rho_{min}$ = −1.11 e Å^{-3} (0.79 Å from Mo1)
Extinction correction: Zachariasen (1967)
Extinction coefficient: 5.5 (2) × 10^{-7}

Table 1
Selected geometric parameters (Å, °).

I—O1	1.890 (2)	Mo3—O7^{i}	1.935 (2)
I—O2	1.885 (2)	Mo3—O10	1.923 (2)
I—O3	1.881 (2)	Mo3—O11	1.714 (3)
Mo1—O1	2.317 (2)	Mo3—O12	1.697 (3)
Mo1—O2	2.353 (2)	Cr—O13	1.968 (3)
Mo1—O4	1.701 (3)	Cr—O13*A*	1.93 (3)
Mo1—O5	1.915 (2)	Cr—O14	1.969 (3)
Mo1—O6	1.711 (3)	Cr—O14*A*	1.90 (3)
Mo1—O7	1.948 (2)	Cr—O15	1.968 (3)
Mo2—O2	2.331 (2)	Cr—O15*A*	1.93 (3)
Mo2—O3	2.337 (2)	Na—O16	2.378 (4)
Mo2—O5	1.930 (2)	Na—O17	2.435 (3)
Mo2—O8	1.694 (3)	Na—O17^{ii}	2.437 (3)
Mo2—O9	1.697 (3)	Na—O18	2.377 (4)
Mo2—O10	1.926 (2)	Na—O19	2.449 (3)
Mo3—O1^{i}	2.331 (2)	Na—O20	2.451 (3)
Mo3—O3	2.347 (2)		
O1—I—O2	86.61 (9)	O2—Mo2—O10	81.22 (9)
O1—I—O3	93.20 (9)	O3—Mo2—O5	81.04 (9)
O2—I—O3	87.31 (10)	O3—Mo2—O8	92.74 (12)
O1—Mo1—O2	67.34 (8)	O3—Mo2—O9	159.62 (11)
O1—Mo1—O4	94.93 (12)	O3—Mo2—O10	73.31 (8)
O1—Mo1—O5	81.69 (9)	O5—Mo2—O8	97.76 (12)
O1—Mo1—O6	156.75 (11)	O5—Mo2—O9	101.36 (12)
O1—Mo1—O7	72.65 (8)	O5—Mo2—O10	149.08 (10)
O2—Mo1—O4	160.98 (12)	O8—Mo2—O9	106.83 (16)
O2—Mo1—O5	72.86 (9)	O8—Mo2—O10	100.38 (12)
O2—Mo1—O6	91.20 (11)	O9—Mo2—O10	97.08 (12)
O2—Mo1—O7	82.04 (9)	O1^{i}—Mo3—O3	67.27 (8)
O4—Mo1—O5	98.66 (12)	O1^{i}—Mo3—O7^{i}	72.54 (9)
O4—Mo1—O6	107.32 (14)	O1^{i}—Mo3—O10	81.39 (9)
O4—Mo1—O7	99.70 (12)	O1^{i}—Mo3—O11	156.65 (11)
O5—Mo1—O6	101.06 (12)	O1^{i}—Mo3—O12	95.37 (11)
O5—Mo1—O7	149.48 (10)	O3—Mo3—O7^{i}	82.26 (10)
O6—Mo1—O7	96.46 (11)	O3—Mo3—O10	73.11 (9)
O2—Mo2—O3	67.68 (8)	O3—Mo3—O11	91.01 (11)
O2—Mo2—O5	73.14 (9)	O3—Mo3—O12	161.15 (11)
O2—Mo2—O8	159.21 (12)	O7^{i}—Mo3—O10	149.51 (10)
O2—Mo2—O9	93.43 (11)	O7^{i}—Mo3—O11	97.01 (11)

O7^{i}—Mo3—O12	100.01 (12)	O17^{ii}—Na—O18	91.74 (14)
O10—Mo3—O11	101.04 (12)	O17^{ii}—Na—O19	172.81 (11)
O10—Mo3—O12	97.88 (12)	O17^{ii}—Na—O20	88.37 (10)
O11—Mo3—O12	107.16 (14)	O18—Na—O19	93.57 (13)
O13—Cr—O13*A*^{iii}	35.5 (9)	O18—Na—O20	90.20 (13)
O13—Cr—O14	90.54 (13)	O19—Na—O20	96.46 (10)
O13—Cr—O15	89.30 (12)	I—O1—Mo1	103.58 (9)
O13*A*—Cr—O14*A*	82.6 (13)	I—O1—Mo3^{i}	103.10 (9)
O13*A*—Cr—O15*A*	85.3 (12)	Mo1—O1—Mo3^{i}	91.61 (8)
O14—Cr—O15	90.36 (13)	I—O2—Mo1	102.46 (9)
O14*A*—Cr—O15*A*	94.8 (15)	I—O2—Mo2	102.55 (9)
O16—Na—O17	89.23 (13)	Mo1—O2—Mo2	89.71 (8)
O16—Na—O17^{ii}	82.65 (13)	I—O3—Mo2	102.45 (9)
O16—Na—O18	172.90 (19)	I—O3—Mo3	102.82 (9)
O16—Na—O19	91.67 (13)	Mo2—O3—Mo3	89.54 (8)
O16—Na—O20	93.98 (13)	Mo1—O5—Mo2	118.43 (12)
O17—Na—O17^{ii}	86.09 (11)	Mo1—O7—Mo3^{i}	118.22 (12)
O17—Na—O18	86.03 (13)	Mo2—O10—Mo3	117.94 (12)
O17—Na—O19	89.44 (10)	Na—O17—Na^{ii}	93.91 (11)
O17—Na—O20	173.20 (11)		

Symmetry codes: (i) $1-x, 1-y, 1-z$; (ii) $-x, 1-y, 1-z$; (iii) $-x, -y, 1-z$.

The intensity data were corrected for absorption (azimuthal scans). Structure solution used an *SIR*92 *E* map (Altomare *et al.*, 1994). Alternative positions were found for the three independent water molecules bonded to Cr, and for two non-coordinated waters. Atoms other than minor-occupancy water molecules were refined anisotropically. H atoms of water molecules could not be located with confidence and no attempt was made to model them. The value of a secondary extinction parameter was refined.

Data collection: *CAD*-4/*PC Diffractometer Software* (Enraf–Nonius, 1993); cell refinement: *CAD*-4/*PC Diffractometer Software*; data reduction: *TEXSAN* (Molecular Structure Corporation, 1997); program(s) used to refine structure: *TEXSAN*; software used to prepare material for publication: *TEXSAN*.

References

Altomare, A., Cascarano, G., Giacovazzo, C., Guagliardi, A., Burla, M. C., Polidori, G. & Camalli, M. (1994). *J. Appl. Cryst.* **27**, 435.

Enraf–Nonius (1993). *CAD*-4/*PC Diffractometer Software*. Version 1.2. Enraf–Nonius, Delft, The Netherlands.

Kondo, H., Kobayashi, A. & Sasaki, Y. (1980). *Acta Cryst.* B**36**, 661–664.

Molecular Structure Corporation (1997). *TEXSAN*. Version 1.7. MSC, 3200 Research Forest Drive, The Woodlands, TX 77381, USA.

North, A. C. T., Phillips, D. C. & Mathews, F. S. (1968). *Acta Cryst.* A**24**, 351–359.

Rosu, C. & Dickman, M. H. (1999). *Acta Cryst*, C**55**, 11–13.

Zachariasen, W. H. (1967). *Acta Cryst.* **23**, 558–564.