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Tris(2,2′-bipyridyl-κ^2N,N')nickel(II) β-octamolybdate tetrahydrate

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Key indicators

Single-crystal X-ray study
T = 273 K
Mean σ(C–C) = 0.005 Å
H-atom completeness 86%
R factor = 0.024
wR factor = 0.072
Data-to-parameter ratio = 18.1

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

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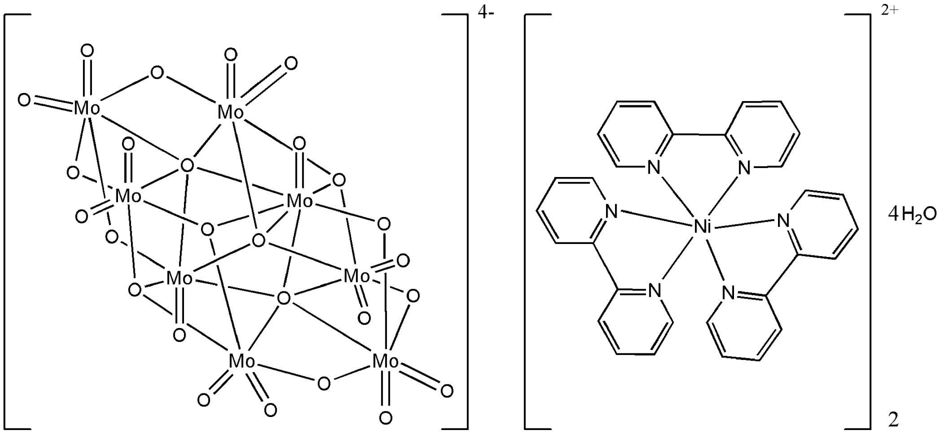
The title compound, $[Ni(C_{10}H_8N_2)_3]_2[Mo_8O_{26}]\cdot 4H_2O$, was synthesized hydrothermally as colorless crystalline blocks. The asymmetric unit consists of one cation, one half-anion and two water molecules; the anion is centrosymmetric.

Comment

Of the various polyoxometalate (POM) structures, the most interesting is the octamolybdate family with a variety of structural isomers including α-, β-, γ-, δ-, ε- and ζ-octamolybdates (Xu *et al.*, 1999; Xiao *et al.*,2005). Recently the structures of several complexes based on the β-octamolybdate anion have been described (Wu *et al.*, 2002; Yang *et al.*, 2002). The geometric parameters of the title compound, (I), are similar to those reported for the β-octamolybdate type polyoxometalate (Lu *et al.*, 2003). The chemistry of POM-based hybrids has been significantly enriched by inclusion of transition-metal coordination complexes in the hybrid system. The organic component is introduced as a ligand to a secondary metal site, which functions as an integral subunit of the structure. Here, the synthesis and crystal structure of (I) is reported.



(I)

The structure of (I) is shown in Fig. 1. The asymmetric unit consists of one cation, one half-anion and two water molecules; the anion is centrosymmetric.. Selected bond lengths are listed in Table 1. In the $[Ni(bpy)_3]^{2+}$ cation, the three bpy molecules are almost perpendicular to each other. The coordination geometry of the Ni atom is octahedral. The well known β-$[Mo_8O_{26}]^{4-}$ cluster consists of eight distorted corner-and/or edge-sharing MoO_6 octahedra.

Experimental

Hexaammonium heptamolybdate tetrahydrate (0.83 g, 0.67 mmol), nickel(II) acetate (0.11 g, 0.45 mmol), 2,2′-bipyridyl (0.10 g, 0.65 mmol) and water (10 ml) were placed in a 20 ml Teflon-lined Parr bomb. The bomb was heated to 413 K for 5 d. Colorless block-shaped crystals were isolated from the cool solution in 65% yield based on Mo. Analysis found: C 31.18, H 2.46, N 7.25%; calculated for $C_{60}H_{56}Mo_8N_{12}Ni_2O_{30}$: C 31.20, H 2.44, N 7.28%.

Crystal data

$[Ni(C_{10}H_8N_2)_3]_2[Mo_8O_{26}]\cdot 4H_2O$
$M_r = 2310.10$
Monoclinic, $P2_1/c$
$a = 12.2823\ (4)$ Å
$b = 13.7818\ (4)$ Å
$c = 21.6970\ (6)$ Å
$\beta = 90.8703\ (7)^\circ$
$V = 3672.28\ (19)$ Å^3
$Z = 2$
Mo $K\alpha$ radiation
$\mu = 1.91$ mm^{-1}
$T = 273\ (2)$ K
0.24 × 0.20 × 0.14 mm

Data collection

Bruker SMART CCD area-detector diffractometer
Absorption correction: multi-scan (*SADABS*; Sheldrick, 2002)
$T_{min} = 0.657$, $T_{max} = 0.776$
36452 measured reflections
9119 independent reflections
7347 reflections with $I > 2\sigma(I)$
$R_{int} = 0.045$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.025$
$wR(F^2) = 0.072$
$S = 1.04$
9119 reflections
505 parameters
24 restraints
H-atom parameters constrained
$\Delta\rho_{max} = 0.69$ e Å^{-3}
$\Delta\rho_{min} = -0.48$ e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

Mo1—O7	1.6925 (18)	Mo3—O13	2.3018 (18)
Mo1—O8	1.9481 (17)	Mo4—O3	1.693 (2)
Mo2—O1	1.6963 (19)	Mo4—O5	1.699 (2)
Mo2—O10	1.8928 (18)	Ni1—N3	2.070 (2)
Mo3—O12	1.699 (2)	Ni1—N5	2.078 (2)
O7—Mo1—O13	103.70 (9)	O10—Mo3—O4^{i}	145.01 (8)
O9—Mo1—O8	150.30 (7)	O3—Mo4—O9	102.05 (9)
O1—Mo2—O2	104.58 (10)	O3—Mo4—O6	160.91 (9)
O1—Mo2—O9	163.75 (8)	N1—Ni1—N5	92.00 (9)
O11—Mo3—O10	98.68 (10)	N3—Ni1—N4	174.02 (9)

Symmetry code: (i) $-x + 2, -y + 1, -z + 2$.

H atoms attached to C atoms were placed in idealized positions, with C—H = 0.93 Å and with $U_{iso}(H) = 1.2U_{eq}(C)$. H atoms of the water molecules were not located in difference Fourier maps and were disregarded in the final model.

Data collection: *SMART* (Bruker, 1998); cell refinement: *SAINT* (Bruker, 1998); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *SHELXTL* (Bruker, 2000); software used to prepare material for publication: *SHELXTL*.

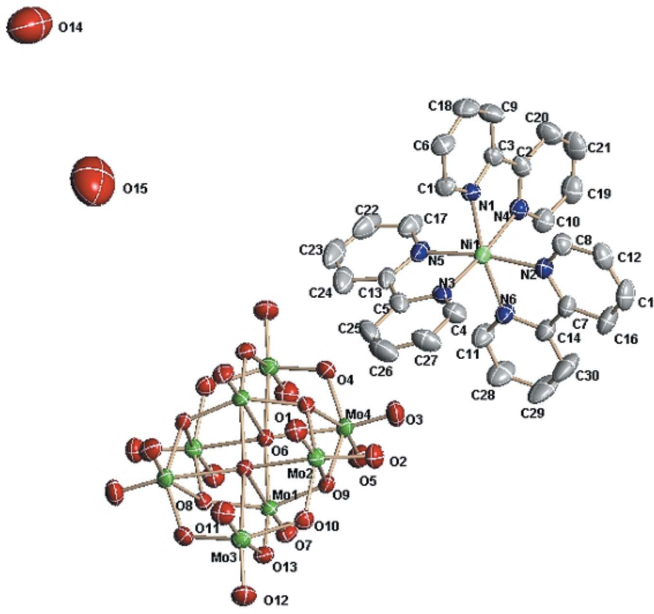


Figure 1
The structure of the components of (I), with the atom-numbering scheme, showing displacement ellipsoids at the 50% probability level. H atoms have been omitted. (Symmetry code to generate equivalent unlabeled atoms: $-x + 2, -y + 1, -z + 2$.)

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