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(μ-Oxalato-$\kappa^4O^1,O^2:O^{1'},O^{2'}$)bis[bis(2,2′-bipyridine-κ^2N,N')cobalt(II)] μ_6-oxido-dodeca-μ_2-oxido-hexaoxido-hexatungstate(VI)

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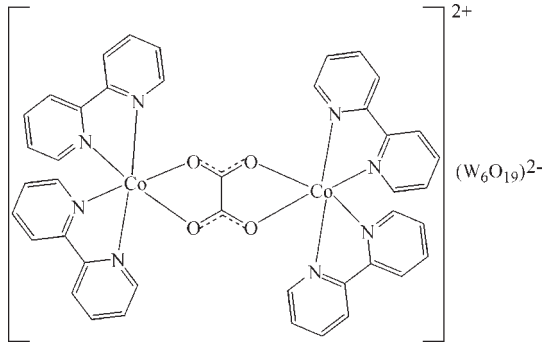
Key indicators: single-crystal X-ray study; T = 293 K; mean σ(C–C) = 0.013 Å; R factor = 0.031; wR factor = 0.076; data-to-parameter ratio = 12.5.

The asymmetric unit of the title compound, $[Co_2(C_2O_4)(C_{10}H_8N_2)_4][W_6O_{19}]$, consists of one half of the complex $[Co_2(C_2O_4)(C_{10}H_8N_2)_4]^{2+}$ cation and one half of the Lindqvist-type $[W_6O_{19}]^{2-}$ isopolyanion. Both constituents are completed by crystallographic inversion symmetry. In the dimeric cation, the Co^{II} atom is surrounded in a distorted octahedral coordination by four N atoms from two chelating 2,2′-bipyridine ligands and by two O atoms from the chelating oxalate anion. The Lindqvist-type anion exhibits the characteristic W—O bond-length distribution, with the shortest bonds being the W—$O_{terminal}$ bonds and the longest being those to the central O atom.

Related literature

For general background to polyoxidometalates, see: Pope & Müller (1991). For polyoxidometalates modified with amines, see: Zhang, Dou *et al.* (2009); Zhang, Wei *et al.* (2009); Zhang *et al.* (2010). For another structure comprising a Lindqvist-type isopolyanion, see: Meng *et al.* (2006). For a related structure, see: Li & Xu (2009).



Experimental

Crystal data

$[Co_2(C_2O_4)(C_{10}H_8N_2)_4][W_6O_{19}]$
M_r = 2237.72
Triclinic, $P\overline{1}$
a = 9.4876 (15) Å
b = 9.8548 (15) Å
c = 14.174 (2) Å
α = 90.769 (2)°
β = 91.576 (2)°
γ = 91.113 (2)°
V = 1324.3 (4) Å^3
Z = 1
Mo $K\alpha$ radiation
μ = 13.67 mm^{-1}
T = 293 K
0.12 × 0.10 × 0.08 mm

Data collection

Bruker APEXII CCD diffractometer
Absorption correction: multi-scan (*SADABS*; Bruker, 2001)
T_{min} = 0.291, T_{max} = 0.408
9331 measured reflections
4613 independent reflections
3755 reflections with $I > 2\sigma(I)$
R_{int} = 0.027

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.031
$wR(F^2)$ = 0.076
S = 1.00
4613 reflections
368 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 2.41 e Å^{-3}
$\Delta\rho_{min}$ = −1.10 e Å^{-3}

Table 1
Selected bond lengths (Å).

Co1—O1	2.104 (6)	O8—W3^{i}	2.3185 (4)
Co1—N1	2.101 (7)	O8—W3	2.3185 (4)
Co1—N4	2.105 (6)	O8—W1	2.3240 (4)
Co1—N2	2.114 (6)	O8—W1^{i}	2.3240 (4)
Co1—N3	2.119 (7)	O8—W2^{i}	2.3252 (5)
Co1—O2	2.134 (6)	O8—W2	2.3252 (5)
O3—W2	1.690 (6)	O9—W2	1.912 (6)
O4—W2	1.919 (6)	O9—W1	1.915 (5)
O4—W3	1.926 (6)	O10—W1	1.914 (6)
O5—W3	1.904 (5)	O10—W3^{i}	1.914 (6)
O5—W2^{i}	1.935 (5)	O11—W1	1.696 (6)
O6—W3	1.698 (6)	O12—W1	1.922 (5)
O7—W3	1.915 (6)	O12—W2^{i}	1.920 (6)
O7—W1	1.931 (6)		

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

Data collection: *APEX2* (Bruker, 2004); cell refinement: *SAINT-Plus* (Bruker, 2001); data reduction: *SAINT-Plus*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *SHELXTL* (Sheldrick, 2008); software used to prepare material for publication: *SHELXTL*.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: WM2358).

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supplementary materials

Acta Cryst. (2010). E**66**, m822-m823 [**doi:10.1107/S1600536810023007**]

(μ-Oxalato-$\kappa^4O^1,O^2:O^{1'},O^{2'}$)bis[bis(2,2'-bipyridine-κ^2N,N')cobalt(II)] μ_6-oxido-dodeca-μ_2-oxido-hexaoxido-hexatungstate(VI)

C. Shi, L. Fan, P. Wei, B. Li and X. Zhang

Comment

There has been extensive interest in polyoxidometalates, owing to their fascinating properties and great potential applications in many fields such in catalysis, material science, medicine and magnetochemistry (Pope & Müller, 1991). Organic amines, such as 3-(2-pyridyl)pyrazole and pyrazine, are used to effectively modify polyoxidomolybdates or heteropolyoxidomolybdates under hydrothermal condictions (Zhang, Dou *et al.*, 2009; Zhang, Wei *et al.*, 2009; Zhang *et al.*, 2010). Here, we describe the synthesis and structural characterization of the title compound.

As shown in Figure 1, the title compound consists of two subunits, *viz.* of a binuclear complex $[Co_2(C_2O_4)(C_{10}H_8N_2)_4]^{2+}$ cation, and one Lindqvist-type $[W_6O_{19}]^{2-}$ isopolyanion. Both constituents exhibit $\bar{1}$ symmetry. The Co^{2+} cation is surrounded in a distorted octahedral coordination by four N atoms from two chelating 2,2'-bipyridine ligands and two O atoms from a chelating oxalate anion. The Co—N and Co—O bond lengths are in the range of 2.101 (7)—2.119 (7) and 2.104 (6)—2.134 (6) Å, respectively, and are in good agreement with the bond lenghts observed for *catena*-poly[[(2,2'-bipyridine-κN,*N*')cobalt(II)]-μ-oxalato- κ^4O^1,*O*^{2}:*O*$^{1'}$,*O*$^{2'}$] (Li & Xu, 2009).

The $[W_6O_{19}]^{2-}$ polyoxidoanion, possessing the well known Lindquist structure, is formed by six WO_6 octahedra connected with each other through edge-sharing oxygen atoms. This anion approaches an approximate O_h symmetry, but actually has $\bar{1}$ symmetry. Three different kinds of oxygen atoms exist in the cluster, *viz.* terminal Oa, double-bridging Ob, and central Oc oxygen atoms. Therefore, W—O bond lengths can be grouped into three sets: W—O*a*: 1.690 (6)—1.698 (6) Å; W—O*b*: 1.904 (5)—1.935 (5) Å; W—O*c*: 2.3185 (4)—2.3252 (5) Å; these bond lengths strictly follow the rule W—O*a* < W—O*b* < W—O*c*, which is in agreement with the Lindqvist-type polyoxidotungstate reported by Meng *et al.* (2006).

Experimental

2,2'-bipyridine (0.5 mmoL 0.07 g) and *p*-carboxyphenylboronic acid were purchased from Jinan Henghua Science & Technology Co. Ltd. A mixture of 2,2'-bipyridine (0.5 mmol 0.07 g), tungstic acid (0.4 mmoL, 0.10 g), oxalic aicd (10 mmol, 0.09), *p*-carboxyphenylboronic acid (0.3 mmol, 0.05 g), and cobalt(II) sulfate heptahydrate (0.2 mmol, 0.05 g) in 14 ml distilled water was sealed in a 25 ml Teflon-lined stainless steel autoclave and was kept at 433 K for three days. Red crystals suitable for the X-ray experiment were obtained. Anal. Calc. for $C_{42}H_{32}Co_2N_8O_{23}W_6$: C, 22.53; H, 1.43; N, 5.01. Found: C, 22.26; H, 1.33; N, 4.85%.

Refinement

All hydrogen atoms bound to carbon were refined using a riding model with distance C—H = 0.93 Å, U_{iso} = 1.2U_{eq}(C). In the final difference Fourier map the highest peak is 2.60 Å from atom H1 and the deepest hole is 0.81 Å from atom W3.

The highest peak is located in the voids of the crystal structure and may be associated with an additional water molecule. However, refinement of this position did not result in a reasonable model.

Figures

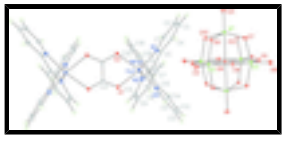


Fig. 1. The cation and anion of the title compound with the atom-labelling scheme. Displacement ellipsoids are drawn at the 30% probability level; H atoms are given as spheres of arbitrary radius.

(μ-Oxalato-κ$^4O^1,O^2$:$O^{1'},O^{2'}$)bis[bis(2,2'- bipyridine-κ$^2N,N'$)cobalt(II)] μ_6-oxido-dodeca-μ_2-oxido-hexaoxidohexatungstate(VI)

Crystal data

$[Co_2(C_2O_4)(C_{10}H_8N_2)_4][W_6O_{19}]$	$Z = 1$
$M_r = 2237.72$	$F(000) = 1022$
Triclinic, $P\overline{1}$	$D_x = 2.806$ Mg m^{-3}
Hall symbol: -P 1	Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
$a = 9.4876$ (15) Å	Cell parameters from 3530 reflections
$b = 9.8548$ (15) Å	$\theta = 2.5$–$27.3°$
$c = 14.174$ (2) Å	$\mu = 13.67$ mm^{-1}
$\alpha = 90.769$ (2)°	$T = 293$ K
$\beta = 91.576$ (2)°	Block, red
$\gamma = 91.113$ (2)°	0.12 × 0.10 × 0.08 mm
$V = 1324.3$ (4) Å^3	

Data collection

Bruker APEXII CCD diffractometer	4613 independent reflections
Radiation source: fine-focus sealed tube	3755 reflections with $I > 2\sigma(I)$
graphite	$R_{int} = 0.027$
φ and ω scans	$\theta_{max} = 25.0°$, $\theta_{min} = 2.6°$
Absorption correction: multi-scan (*SADABS*; Bruker, 2001)	$h = -11 \rightarrow 11$
$T_{min} = 0.291$, $T_{max} = 0.408$	$k = -11 \rightarrow 11$
9331 measured reflections	$l = -16 \rightarrow 16$

Refinement

Refinement on F^2	Primary atom site location: structure-invariant direct methods
Least-squares matrix: full	Secondary atom site location: difference Fourier map
$R[F^2 > 2\sigma(F^2)] = 0.031$	Hydrogen site location: inferred from neighbouring sites
$wR(F^2) = 0.076$	H-atom parameters constrained

$S = 1.00$	$w = 1/[\sigma^2(F_o^2) + (0.0375P)^2 + 1.9139P]$ where $P = (F_o^2 + 2F_c^2)/3$
4613 reflections	$(\Delta/\sigma)_{max} = 0.001$
368 parameters	$\Delta\rho_{max} = 2.41$ e Å^{-3}
0 restraints	$\Delta\rho_{min} = -1.10$ e Å^{-3}

Special details

Geometry. All e.s.d.'s (except the e.s.d. in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell e.s.d.'s are taken into account individually in the estimation of e.s.d.'s in distances, angles and torsion angles; correlations between e.s.d.'s in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell e.s.d.'s is used for estimating e.s.d.'s involving l.s. planes.

Refinement. Refinement of F^2 against ALL reflections. The weighted *R*-factor *wR* and goodness of fit *S* are based on F^2, conventional *R*-factors *R* are based on *F*, with *F* set to zero for negative F^2. The threshold expression of $F^2 > \sigma(F^2)$ is used only for calculating *R*-factors(gt) *etc*. and is not relevant to the choice of reflections for refinement. *R*-factors based on F^2 are statistically about twice as large as those based on *F*, and *R*- factors based on ALL data will be even larger.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å^2)

	x	*y*	*z*	U_{iso}*/U_{eq}
C1	0.5864 (9)	0.5558 (9)	0.7481 (6)	0.039 (2)
H1	0.6557	0.5423	0.7040	0.046*
C2	0.4503 (10)	0.5210 (10)	0.7228 (7)	0.050 (3)
H2	0.4275	0.4831	0.6638	0.060*
C3	0.3498 (10)	0.5440 (10)	0.7869 (7)	0.050 (3)
H3	0.2562	0.5220	0.7714	0.060*
C4	0.3841 (9)	0.5996 (10)	0.8753 (6)	0.045 (2)
H4	0.3150	0.6150	0.9192	0.054*
C5	0.5239 (8)	0.6314 (8)	0.8957 (5)	0.0262 (18)
C6	0.5713 (9)	0.6925 (8)	0.9874 (5)	0.031 (2)
C7	0.4828 (10)	0.7231 (11)	1.0590 (7)	0.050 (3)
H7	0.3869	0.7028	1.0521	0.061*
C8	0.5343 (12)	0.7834 (12)	1.1405 (7)	0.062 (3)
H8	0.4747	0.8067	1.1889	0.074*
C9	0.6775 (12)	0.8085 (12)	1.1486 (7)	0.064 (3)
H9	0.7157	0.8488	1.2035	0.077*
C10	0.7625 (11)	0.7754 (10)	1.0781 (6)	0.046 (2)
H10	0.8589	0.7931	1.0851	0.055*
C11	0.8337 (10)	0.9655 (10)	0.8619 (7)	0.046 (2)
H11	0.7816	0.9641	0.9166	0.055*
C12	0.8660 (11)	1.0894 (10)	0.8234 (7)	0.052 (3)
H12	0.8396	1.1705	0.8515	0.062*
C13	0.9399 (12)	1.0868 (11)	0.7404 (8)	0.062 (3)
H13	0.9621	1.1676	0.7105	0.074*
C14	0.9796 (10)	0.9684 (10)	0.7032 (7)	0.047 (2)
H14	1.0299	0.9669	0.6478	0.057*
C15	0.9459 (8)	0.8496 (9)	0.7469 (6)	0.0306 (19)

C16	0.9845 (8)	0.7135 (9)	0.7095 (6)	0.0294 (19)
C17	1.0644 (9)	0.6960 (10)	0.6295 (6)	0.040 (2)
H17	1.1000	0.7709	0.5981	0.048*
C18	1.0897 (9)	0.5675 (10)	0.5977 (6)	0.043 (2)
H18	1.1428	0.5541	0.5443	0.052*
C19	1.0363 (9)	0.4584 (10)	0.6452 (6)	0.043 (2)
H19	1.0522	0.3703	0.6243	0.051*
C20	0.9589 (9)	0.4815 (9)	0.7240 (6)	0.035 (2)
H20	0.9230	0.4076	0.7564	0.042*
C21	1.0517 (8)	0.5600 (8)	1.0069 (5)	0.0255 (18)
Co1	0.83213 (11)	0.65380 (11)	0.88211 (7)	0.0263 (3)
N1	0.6258 (7)	0.6078 (7)	0.8323 (4)	0.0302 (16)
N2	0.7116 (7)	0.7170 (7)	0.9974 (5)	0.0328 (17)
N3	0.8729 (7)	0.8481 (7)	0.8254 (5)	0.0325 (16)
N4	0.9334 (7)	0.6070 (7)	0.7558 (4)	0.0269 (15)
O1	1.0227 (6)	0.6668 (6)	0.9616 (4)	0.0350 (14)
O2	0.8466 (5)	0.4532 (6)	0.9359 (4)	0.0318 (13)
O3	0.4860 (7)	0.8809 (7)	0.7694 (4)	0.0521 (18)
O4	0.3221 (6)	1.0289 (6)	0.6381 (4)	0.0362 (14)
O5	0.3335 (6)	1.1440 (6)	0.3892 (4)	0.0369 (15)
O6	0.1293 (7)	1.1836 (7)	0.5300 (5)	0.0540 (18)
O7	0.2326 (6)	0.9278 (6)	0.4737 (4)	0.0383 (15)
O8	0.5000	1.0000	0.5000	0.0219 (16)
O9	0.3985 (6)	0.7844 (6)	0.5844 (4)	0.0342 (14)
O10	0.5765 (6)	0.7561 (6)	0.4464 (4)	0.0373 (14)
O11	0.2952 (7)	0.6576 (7)	0.4139 (5)	0.0528 (18)
O12	0.4107 (6)	0.8997 (6)	0.3357 (4)	0.0338 (14)
W1	0.38156 (4)	0.80202 (4)	0.45023 (2)	0.03214 (12)
W2	0.49167 (4)	0.92799 (4)	0.65534 (2)	0.03134 (12)
W3	0.28645 (4)	1.10717 (4)	0.51619 (2)	0.03216 (12)

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.039 (5)	0.051 (6)	0.025 (4)	0.002 (5)	−0.004 (4)	−0.011 (4)
C2	0.047 (6)	0.057 (7)	0.044 (6)	0.004 (5)	−0.018 (5)	−0.008 (5)
C3	0.028 (5)	0.061 (7)	0.058 (6)	−0.010 (5)	−0.014 (5)	0.004 (5)
C4	0.031 (5)	0.063 (7)	0.041 (5)	−0.004 (5)	0.000 (4)	−0.002 (5)
C5	0.024 (4)	0.028 (5)	0.027 (4)	0.000 (4)	−0.003 (3)	0.001 (3)
C6	0.032 (5)	0.031 (5)	0.030 (4)	0.001 (4)	0.012 (4)	0.001 (4)
C7	0.038 (6)	0.064 (7)	0.049 (6)	−0.004 (5)	0.011 (5)	−0.014 (5)
C8	0.057 (7)	0.074 (8)	0.055 (7)	−0.002 (6)	0.027 (5)	−0.027 (6)
C9	0.072 (8)	0.083 (9)	0.036 (6)	−0.003 (7)	0.003 (5)	−0.030 (5)
C10	0.052 (6)	0.051 (6)	0.036 (5)	−0.007 (5)	0.003 (4)	−0.014 (5)
C11	0.044 (6)	0.039 (6)	0.056 (6)	0.007 (5)	0.010 (5)	−0.002 (5)
C12	0.064 (7)	0.023 (5)	0.067 (7)	0.006 (5)	−0.001 (6)	−0.008 (5)
C13	0.066 (8)	0.044 (7)	0.074 (8)	−0.007 (6)	−0.002 (6)	0.013 (6)
C14	0.053 (6)	0.042 (6)	0.047 (6)	−0.007 (5)	0.006 (5)	0.001 (5)

C15	0.021 (4)	0.038 (5)	0.033 (4)	−0.002 (4)	−0.006 (3)	0.001 (4)
C16	0.016 (4)	0.038 (5)	0.034 (4)	−0.009 (4)	−0.009 (3)	0.002 (4)
C17	0.039 (5)	0.049 (6)	0.031 (5)	−0.001 (5)	0.011 (4)	0.009 (4)
C18	0.036 (5)	0.056 (7)	0.038 (5)	0.002 (5)	0.009 (4)	−0.005 (5)
C19	0.047 (6)	0.044 (6)	0.037 (5)	0.004 (5)	0.003 (4)	−0.015 (4)
C20	0.034 (5)	0.028 (5)	0.042 (5)	0.000 (4)	0.002 (4)	−0.002 (4)
C21	0.016 (4)	0.029 (5)	0.032 (4)	−0.004 (3)	0.005 (3)	−0.005 (4)
Co1	0.0243 (6)	0.0289 (6)	0.0258 (6)	0.0001 (5)	0.0035 (4)	−0.0003 (5)
N1	0.026 (4)	0.034 (4)	0.031 (4)	0.001 (3)	0.005 (3)	−0.001 (3)
N2	0.034 (4)	0.031 (4)	0.033 (4)	−0.003 (3)	0.006 (3)	−0.011 (3)
N3	0.034 (4)	0.027 (4)	0.037 (4)	0.001 (3)	0.004 (3)	0.004 (3)
N4	0.026 (4)	0.028 (4)	0.027 (3)	−0.001 (3)	0.003 (3)	−0.002 (3)
O1	0.030 (3)	0.037 (4)	0.038 (3)	−0.002 (3)	−0.003 (3)	0.007 (3)
O2	0.023 (3)	0.036 (4)	0.035 (3)	−0.004 (3)	−0.005 (2)	0.004 (3)
O3	0.065 (5)	0.059 (5)	0.033 (3)	0.007 (4)	0.004 (3)	0.003 (3)
O4	0.032 (3)	0.048 (4)	0.029 (3)	0.001 (3)	0.014 (2)	−0.008 (3)
O5	0.037 (3)	0.041 (4)	0.032 (3)	0.009 (3)	0.000 (3)	0.000 (3)
O6	0.039 (4)	0.068 (5)	0.055 (4)	0.010 (4)	0.002 (3)	−0.014 (4)
O7	0.028 (3)	0.053 (4)	0.034 (3)	0.002 (3)	0.002 (3)	−0.008 (3)
O8	0.021 (4)	0.024 (4)	0.021 (4)	0.004 (3)	0.006 (3)	−0.003 (3)
O9	0.035 (3)	0.033 (4)	0.035 (3)	−0.004 (3)	0.008 (3)	0.000 (3)
O10	0.047 (4)	0.027 (3)	0.039 (3)	0.007 (3)	0.006 (3)	−0.006 (3)
O11	0.054 (4)	0.042 (4)	0.061 (4)	−0.016 (3)	0.009 (3)	−0.013 (3)
O12	0.033 (3)	0.046 (4)	0.022 (3)	0.002 (3)	0.003 (2)	−0.008 (3)
W1	0.0353 (2)	0.0295 (2)	0.0313 (2)	−0.00686 (16)	0.00560 (15)	−0.00922 (15)
W2	0.0381 (2)	0.0338 (2)	0.02229 (18)	0.00114 (16)	0.00478 (14)	−0.00123 (14)
W3	0.0274 (2)	0.0368 (2)	0.0325 (2)	0.00719 (16)	0.00465 (14)	−0.00600 (15)

Geometric parameters (Å, °)

C1—N1	1.333 (10)	C19—C20	1.373 (11)
C1—C2	1.366 (13)	C19—H19	0.9300
C1—H1	0.9300	C20—N4	1.340 (10)
C2—C3	1.355 (13)	C20—H20	0.9300
C2—H2	0.9300	C21—O2^{i}	1.254 (9)
C3—C4	1.388 (13)	C21—O1	1.271 (9)
C3—H3	0.9300	C21—C21^{i}	1.528 (15)
C4—C5	1.379 (11)	Co1—O1	2.104 (6)
C4—H4	0.9300	Co1—N1	2.101 (7)
C5—N1	1.360 (9)	Co1—N4	2.105 (6)
C5—C6	1.479 (11)	Co1—N2	2.114 (6)
C6—N2	1.351 (10)	Co1—N3	2.119 (7)
C6—C7	1.370 (11)	Co1—O2	2.134 (6)
C7—C8	1.366 (13)	O2—C21^{i}	1.254 (9)
C7—H7	0.9300	O3—W2	1.690 (6)
C8—C9	1.378 (15)	O4—W2	1.919 (6)
C8—H8	0.9300	O4—W3	1.926 (6)
C9—C10	1.343 (12)	O5—W3	1.904 (5)

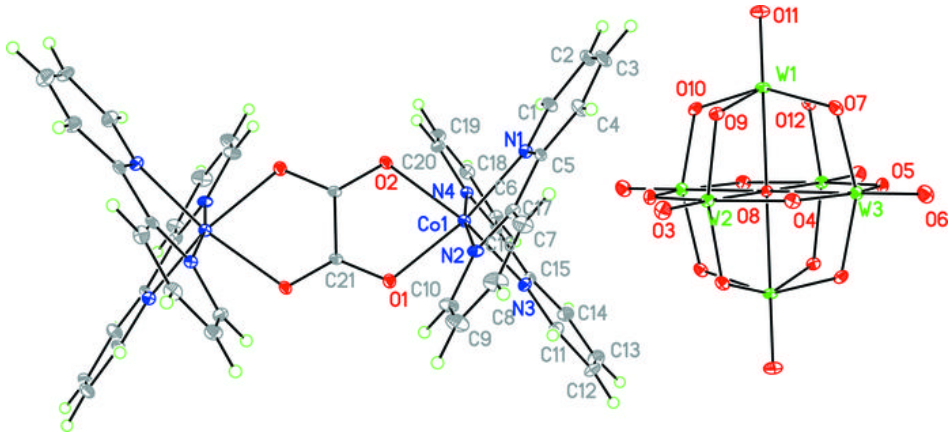
C9—H9	0.9300	O5—W2^{ii}	1.935 (5)
C10—N2	1.346 (10)	O6—W3	1.698 (6)
C10—H10	0.9300	O7—W3	1.915 (6)
C11—N3	1.325 (11)	O7—W1	1.931 (6)
C11—C12	1.377 (13)	O8—W3^{ii}	2.3185 (4)
C11—H11	0.9300	O8—W3	2.3185 (4)
C12—C13	1.386 (14)	O8—W1	2.3240 (4)
C12—H12	0.9300	O8—W1^{ii}	2.3240 (4)
C13—C14	1.339 (14)	O8—W2^{ii}	2.3252 (5)
C13—H13	0.9300	O8—W2	2.3252 (5)
C14—C15	1.368 (12)	O9—W2	1.912 (6)
C14—H14	0.9300	O9—W1	1.915 (5)
C15—N3	1.327 (10)	O10—W1	1.914 (6)
C15—C16	1.491 (11)	O10—W3^{ii}	1.914 (6)
C16—N4	1.336 (10)	O11—W1	1.696 (6)
C16—C17	1.391 (11)	O12—W1	1.922 (5)
C17—C18	1.367 (12)	O12—W2^{ii}	1.920 (6)
C17—H17	0.9300	W2—O12^{ii}	1.920 (6)
C18—C19	1.372 (12)	W2—O5^{ii}	1.935 (5)
C18—H18	0.9300	W3—O10^{ii}	1.914 (6)
N1—C1—C2	124.0 (8)	C1—N1—Co1	127.4 (5)
N1—C1—H1	118.0	C5—N1—Co1	114.6 (5)
C2—C1—H1	118.0	C10—N2—C6	118.9 (7)
C3—C2—C1	117.5 (9)	C10—N2—Co1	126.0 (6)
C3—C2—H2	121.3	C6—N2—Co1	115.1 (5)
C1—C2—H2	121.3	C11—N3—C15	118.4 (8)
C2—C3—C4	121.2 (9)	C11—N3—Co1	125.9 (6)
C2—C3—H3	119.4	C15—N3—Co1	115.7 (6)
C4—C3—H3	119.4	C20—N4—C16	119.2 (7)
C3—C4—C5	117.8 (8)	C20—N4—Co1	125.2 (6)
C3—C4—H4	121.1	C16—N4—Co1	115.4 (5)
C5—C4—H4	121.1	C21—O1—Co1	114.2 (5)
N1—C5—C4	121.5 (7)	C21^{i}—O2—Co1	113.0 (5)
N1—C5—C6	116.4 (7)	W2—O4—W3	117.6 (2)
C4—C5—C6	122.1 (7)	W3—O5—W2^{ii}	117.3 (3)
N2—C6—C7	120.6 (8)	W3—O7—W1	117.5 (3)
N2—C6—C5	115.4 (6)	W3^{ii}—O8—W3	180.0
C7—C6—C5	124.0 (8)	W3^{ii}—O8—W1	89.833 (16)
C8—C7—C6	120.4 (9)	W3—O8—W1	90.167 (16)
C8—C7—H7	119.8	W3^{ii}—O8—W1^{ii}	90.167 (16)
C6—C7—H7	119.8	W3—O8—W1^{ii}	89.833 (16)
C7—C8—C9	117.8 (9)	W1—O8—W1^{ii}	180.0
C7—C8—H8	121.1	W3^{ii}—O8—W2^{ii}	90.173 (13)
C9—C8—H8	121.1	W3—O8—W2^{ii}	89.827 (13)
C10—C9—C8	120.7 (10)	W1—O8—W2^{ii}	90.203 (14)

C10—C9—H9	119.7	W1^{ii}—O8—W2^{ii}	89.797 (14)
C8—C9—H9	119.7	W3^{ii}—O8—W2	89.827 (13)
N2—C10—C9	121.6 (10)	W3—O8—W2	90.173 (13)
N2—C10—H10	119.2	W1—O8—W2	89.797 (14)
C9—C10—H10	119.2	W1^{ii}—O8—W2	90.203 (14)
N3—C11—C12	123.5 (9)	W2^{ii}—O8—W2	180.0
N3—C11—H11	118.2	W2—O9—W1	118.1 (3)
C12—C11—H11	118.2	W1—O10—W3^{ii}	117.8 (3)
C13—C12—C11	116.4 (9)	W1—O12—W2^{ii}	118.0 (3)
C13—C12—H12	121.8	O11—W1—O10	103.9 (3)
C11—C12—H12	121.8	O11—W1—O9	104.0 (3)
C14—C13—C12	120.2 (10)	O10—W1—O9	86.9 (2)
C14—C13—H13	119.9	O11—W1—O12	104.1 (3)
C12—C13—H13	119.9	O10—W1—O12	86.8 (2)
C13—C14—C15	119.8 (9)	O9—W1—O12	151.9 (2)
C13—C14—H14	120.1	O11—W1—O7	104.1 (3)
C15—C14—H14	120.1	O10—W1—O7	152.1 (2)
N3—C15—C14	121.6 (8)	O9—W1—O7	86.5 (2)
N3—C15—C16	115.2 (7)	O12—W1—O7	86.3 (2)
C14—C15—C16	123.2 (8)	O11—W1—O8	180.0 (3)
N4—C16—C17	121.1 (8)	O10—W1—O8	76.11 (17)
N4—C16—C15	115.8 (7)	O9—W1—O8	76.06 (17)
C17—C16—C15	123.0 (8)	O12—W1—O8	75.87 (17)
C18—C17—C16	119.2 (8)	O7—W1—O8	75.96 (18)
C18—C17—H17	120.4	O3—W2—O9	105.6 (3)
C16—C17—H17	120.4	O3—W2—O4	103.1 (3)
C17—C18—C19	119.5 (8)	O9—W2—O4	87.0 (2)
C17—C18—H18	120.2	O3—W2—O12^{ii}	102.5 (3)
C19—C18—H18	120.2	O9—W2—O12^{ii}	152.0 (2)
C18—C19—C20	118.9 (8)	O4—W2—O12^{ii}	86.6 (2)
C18—C19—H19	120.6	O3—W2—O5^{ii}	104.7 (3)
C20—C19—H19	120.6	O9—W2—O5^{ii}	86.7 (2)
N4—C20—C19	122.1 (8)	O4—W2—O5^{ii}	152.2 (2)
N4—C20—H20	118.9	O12^{ii}—W2—O5^{ii}	86.3 (2)
C19—C20—H20	118.9	O3—W2—O8	178.2 (2)
O2^{i}—C21—O1	125.8 (7)	O9—W2—O8	76.08 (16)
O2^{i}—C21—C21^{i}	117.7 (9)	O4—W2—O8	76.11 (15)
O1—C21—C21^{i}	116.4 (9)	O12^{ii}—W2—O8	75.88 (15)
O1—Co1—N1	164.8 (2)	O5^{ii}—W2—O8	76.08 (16)
O1—Co1—N4	93.3 (2)	O6—W3—O5	104.3 (3)
N1—Co1—N4	96.6 (2)	O6—W3—O7	103.2 (3)
O1—Co1—N2	92.9 (2)	O5—W3—O7	87.2 (2)
N1—Co1—N2	78.4 (3)	O6—W3—O10^{ii}	104.2 (3)
N4—Co1—N2	172.1 (3)	O5—W3—O10^{ii}	87.1 (2)
O1—Co1—N3	90.4 (2)	O7—W3—O10^{ii}	152.6 (2)

N1—Co1—N3	103.0 (3)	O6—W3—O4	102.7 (3)
N4—Co1—N3	77.4 (3)	O5—W3—O4	153.0 (2)
N2—Co1—N3	97.7 (3)	O7—W3—O4	86.6 (2)
O1—Co1—O2	78.3 (2)	O10^{ii}—W3—O4	86.3 (2)
N1—Co1—O2	89.4 (2)	O6—W3—O8	178.8 (2)
N4—Co1—O2	94.3 (2)	O5—W3—O8	76.81 (16)
N2—Co1—O2	91.7 (2)	O7—W3—O8	76.38 (16)
N3—Co1—O2	165.6 (2)	O10^{ii}—W3—O8	76.26 (16)
C1—N1—C5	118.0 (7)	O4—W3—O8	76.16 (15)

Symmetry codes: (i) −*x*+2, −*y*+1, −*z*+2; (ii) −*x*+1, −*y*+2, −*z*+1.

Fig. 1


O11
W1
O10
O9
O12
O7
O5
O3
V2
O8
O4
W3
O6
C2
C3
C1
C4
C19
N1
C20
C18
C5
O2
N4
C6
Co1
C17
C7
N2
C15
C21
O1
C10
N3
C14
C8
C9
C11
C13
C12