Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

Tetrapotassium heptacyanidomolybdate(III) dihydrate

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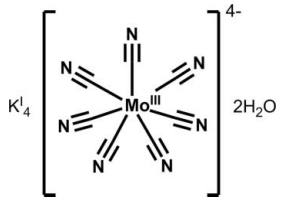
Received 8 July 2009; accepted 28 September 2009

Key indicators: single-crystal X-ray study; T = 90 K; mean σ(N–C) = 0.006 Å; H-atom completeness 46%; disorder in main residue; R factor = 0.054; wR factor = 0.153; data-to-parameter ratio = 16.3.

The asymmetric unit of the title compound, $K^{I}_4[Mo^{III}(CN)_7]\cdot 2H_2O$, consists of one $[Mo(CN)_7]^{4-}$ anion, four K^+ cations, and two water molecules. The $[Mo^{III}(CN)_7]^{4-}$ anion has a seven-coordinated capped-trigonal-prismatic coordination geometry. The site-occupancy factors of the disordered water molecules were set at 0.90, 0.60 and 0.50. The H-atom positions could not be determined for two of the water molecules. The H atoms of the water with a site-occupancy factor of 0.90 were refined using O—H and H···H distance restraints.

Related literature

For the synthesis and spectroscopic information for the title compound, see: Young (1932); Rossman *et al.* (1973). For octacyanidometalate-based materials with photomagnetic and magnetic properties, see: Arimoto *et al.* (2003); Catala *et al.* (2005); Ohkoshi *et al.* (2007, 2008). For the related heptacyanido molybdate(III) crystal structure with D_{5h} and C_{2v} symmetry, see: Hursthouse *et al.* (1980)); Larionova *et al.* (2004); For a heptacyanido molybdate(II) crystal structure with C_s symmetry, see: Drew *et al.* (1977).



Experimental

Crystal data

$K_4[Mo(CN)_7]\cdot 2H_2O$
M_r = 468.29
Triclinic, $P\overline{1}$
a = 8.8813 (5) Å
b = 9.2896 (4) Å
c = 9.7221 (4) Å
α = 86.6480 (13)°
β = 82.2150 (19)°
γ = 71.2570 (17)°
V = 752.48 (6) Å^3
Z = 2
Mo $K\alpha$ radiation
μ = 1.99 mm^{-1}
T = 90 K
0.10 × 0.05 × 0.02 mm

Data collection

Rigaku R-AXIS RAPID diffractometer
Absorption correction: multi-scan (*ABSCOR*; Higashi, 1999)
T_{min} = 0.826, T_{max} = 0.961
7367 measured reflections
3421 independent reflections
2893 reflections with $I > 2\sigma(I)$
R_{int} = 0.025

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.054
$wR(F^2)$ = 0.153
S = 1.25
3421 reflections
210 parameters
3 restraints
H-atom parameters not defined
$\Delta\rho_{max}$ = 2.85 e Å^{-3}
$\Delta\rho_{min}$ = −1.08 e Å^{-3}

Data collection: *PROCESS-AUTO* (Rigaku, 1998); cell refinement: *PROCESS-AUTO*; data reduction: *CrystalStructure* (Rigaku, 2007); program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *CrystalStructure* (Rigaku, 2007); software used to prepare material for publication: *ORTEP-3* (Farrugia, 1997) and *PyMOLWin* (DeLano, 2007).

The present research was supported in part by a Grant-in-Aid for Young Scientists (S) from JSPS, a grant for the Global COE Program, Chemistry Innovation through Cooperation of Science and Engineering, the Photon Frontier Network Program from MEXT, Japan, the Kurata Memorial Hitachi Science and Technology Foundation, and the Inamori Foundation. YT is grateful to JSPS Research Fellowships for Young Scientists. A part of this work was conducted at the Center for Nano Lithography & Analysis at the University of Tokyo, supported by the Ministry of Education, Culture, Sports, Science and Technology (MEXT), Japan.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: SI2189).

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supporting information

Acta Cryst. (2009). E**65**, i79–i80 [https://doi.org/10.1107/S1600536809039336]

Tetrapotassium heptacyanidomolybdate(III) dihydrate

Koji Nakabayashi, Keisuke Tomono, Yoshihide Tsunobuchi, Wataru Kosaka and Shin-ichi Ohkoshi

S1. Comment

In the field of molecule-based magnets, cyano-bridged metal assemblies have been much studied to demonstrate novel functionalities. Octacyanometalate-based materials show interesting functionalities such as photomagnetism (Arimoto *et al.*, 2003; Catala *et al.*, 2005; Ohkoshi *et al.*, 2008) and chemically sensitive magnetism (Ohkoshi *et al.*, 2007). As a versatile class of building blocks, a heptacyanomolybdate ion $[Mo^{III}(CN)_7]^{4-}$ is also attractive because this metal complex can adopt various spatial configurations, depending on the chemical environment, pentagonal bipyramid ($D_{5\,h}$) and capped trigonal prism (C_{2v}) (Rossman *et al.*, 1973; Larionova *et al.*, 2004). Although the coordination geometry of pentagonal bipyramid ($D_{5\,h}$) has been observed with the mixed salt $Na^{I}K^{I}{}_3[Mo(CN)_7]2H_2O$, that of capped trigonal prism ($C_{2\,v}$) has not been reported (Hursthouse *et al.*, 1980, Drew *et al.*, 1977). In this paper, we report the crystal structure of $K^{I}{}_4[Mo^{III}(CN)_7]2H_2O$ with capped trigonal prism (C_{2v}) symmetry, which has the asymmetric unit of one $[Mo^{III}(CN)_7]^{4-}$ anion, four K^+ cations, and two water molecules distributed over three sites with site occupation factors (s.o.f's) of 0.90, 0.60 and 0.50. (Fig. 1). The synthesis and the spectral information of the title compound as been reported by Young (1932) and Rossman *et al.*, (1973).

S2. Experimental

The title compound was prepared by reacting $(NH_4)_2[MoCl_5(H_2O)]$ (1.81 g) with KCN (4 g) in H_2O (23 ml) at room temperature. The prepared compound was a green plate-type crystal. Elemental analyses: calcd for $K^{I}{}_4[Mo^{III}(CN)_7]2.75H_2O$, Calculated: Mo, 19.82%; C, 17.37%; H, 1.15%; N, 20.26%. Found: Mo, 19.44%; C, 17.65%; H, 1.13%; N, 20.01%. In the Infrared (IR) spectra, cyano stretching peaks were observed at 2114 and 2069 cm^{-1}.

S3. Refinement

The H atoms of the solvent water molecules, O2 and O3, could not be located. The maximum and minimum residual electron density peaks were located 0.18 and 0.91 Å from the K3 atom and the Mo1 atom, respectively.

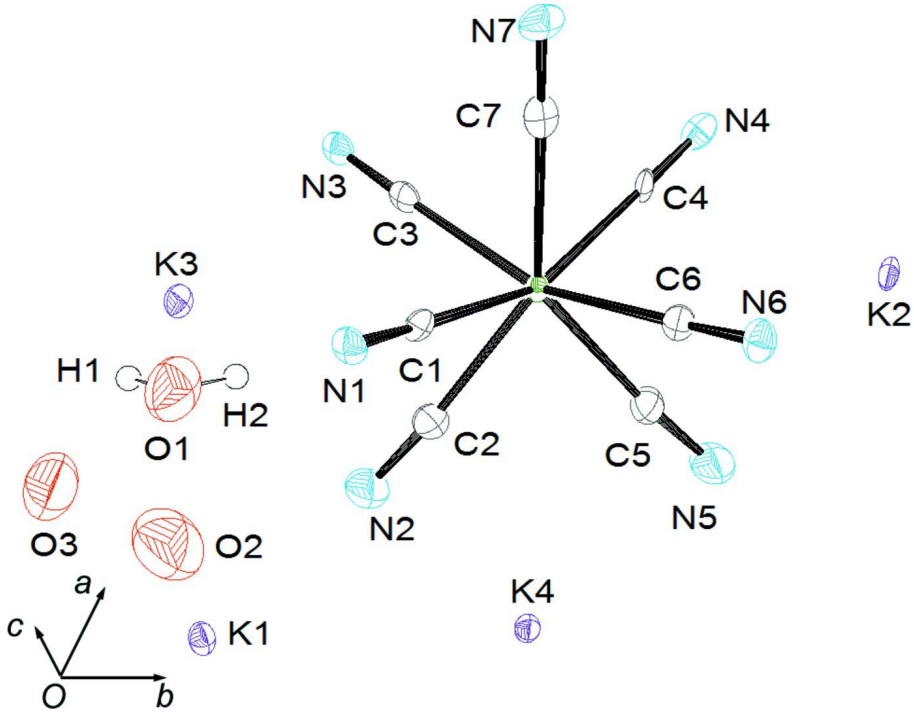

Figure 1

Thermal ellipsoid plots (50% probability level) of independent atoms of $K^{I}_4[Mo^{III}(CN)_7]2H_2O$. Positions of hydrogen atoms of the disordered water molecules could not be determined.

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Crystal data

$K_4[Mo(CN)_7]\cdot 2H_2O$
$M_r = 468.29$
Triclinic, $P\overline{1}$
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$\beta = 82.2150$ (19)°
$\gamma = 71.2570$ (17)°
$V = 752.48$ (6) Å^3
$Z = 2$
$F(000) = 454$
$D_x = 2.067$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71075$ Å
Cell parameters from 6568 reflections
$\theta = 3.0$–27.5°
$\mu = 1.99$ mm^{-1}
$T = 90$ K
Platelet, green
0.10 × 0.05 × 0.02 mm

Data collection

Rigaku R-AXIS RAPID diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
ω scans
Absorption correction: multi-scan (*ABSCOR*; Higashi, 1999)
$T_{min} = 0.826$, $T_{max} = 0.961$
7367 measured reflections
3421 independent reflections
2893 reflections with $I > 2\sigma(I)$
$R_{int} = 0.025$
$\theta_{max} = 27.5°$, $\theta_{min} = 3.0°$
$h = -11\rightarrow 11$
$k = -12\rightarrow 11$
$l = -12\rightarrow 11$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.054$
$wR(F^2) = 0.153$
$S = 1.25$
3421 reflections
210 parameters
3 restraints
Primary atom site location: structure-invariant direct methods
Secondary atom site location: difference Fourier map
H-atom parameters not defined
$w = 1/[\sigma^2(F_o^2) + (0.0908P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 2.85$ e Å^{-3}
$\Delta\rho_{min} = -1.08$ 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. Some weak peaks appeared around all O atoms (O1, O2, and O3). Although we could assign the H atoms of O1 with restraints of *DFIX* (O—H 0.85 0.02 Å and H···H distances about 1.38 0.02 Å), the H atoms of O2 and O3 could not be placed using restraints of *DFIX*.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²)

	x	*y*	*z*	U_{iso}*/U_{eq}	Occ. (<1)
Mo1	−0.28013 (4)	0.18814 (3)	0.22632 (3)	0.00989 (16)	
K4	0.28925 (10)	0.01355 (9)	0.17415 (9)	0.0124 (2)	
K1	0.27995 (11)	−0.37242 (10)	0.36699 (9)	0.0146 (2)	
K2	−0.28226 (10)	0.58985 (9)	0.03172 (9)	0.0129 (2)	
K3	−0.27856 (10)	−0.21632 (9)	0.42126 (9)	0.0133 (2)	
N3	−0.3585 (4)	−0.1085 (4)	0.1198 (4)	0.0150 (7)	
N4	−0.3673 (5)	0.3229 (4)	−0.0868 (4)	0.0182 (8)	
N1	−0.3641 (5)	0.0960 (4)	0.5568 (4)	0.0157 (7)	
N2	0.0322 (5)	−0.0987 (5)	0.2950 (5)	0.0298 (10)	
N5	0.0203 (5)	0.3068 (5)	0.0908 (5)	0.0336 (11)	
C6	−0.3280 (5)	0.4105 (4)	0.3111 (4)	0.0144 (8)	
C4	−0.3348 (5)	0.2756 (4)	0.0224 (4)	0.0127 (7)	
C5	−0.0799 (5)	0.2568 (5)	0.1380 (5)	0.0219 (9)	
C1	−0.3329 (5)	0.1282 (4)	0.4402 (4)	0.0125 (7)	
C3	−0.3286 (5)	−0.0076 (4)	0.1562 (4)	0.0136 (8)	
C2	−0.0739 (5)	0.0069 (5)	0.2682 (5)	0.0206 (9)	
N6	−0.3562 (5)	0.5301 (4)	0.3555 (4)	0.0174 (8)	
C7	−0.5434 (6)	0.2691 (4)	0.2568 (4)	0.0166 (8)	
N7	−0.6817 (5)	0.3103 (4)	0.2748 (4)	0.0209 (8)	
O1	−0.0067 (7)	−0.3954 (6)	0.2344 (7)	0.0609 (15)	0.90
O2	−0.0141 (11)	−0.2434 (11)	0.5544 (12)	0.075 (3)	0.60
O3	−0.0025 (13)	−0.4462 (11)	0.4867 (14)	0.070 (3)	0.50
H1	0.019 (9)	−0.488 (3)	0.199 (6)	0.05 (2)*	0.90

H2	0.007 (9)	−0.343 (6)	0.157 (4)	0.05 (2)*	0.90

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Mo1	0.0125 (2)	0.0083 (2)	0.0092 (2)	−0.00373 (15)	−0.00109 (14)	−0.00049 (14)
K4	0.0150 (5)	0.0119 (5)	0.0117 (5)	−0.0060 (4)	−0.0028 (4)	0.0013 (4)
K1	0.0201 (5)	0.0125 (4)	0.0116 (5)	−0.0058 (4)	−0.0027 (4)	0.0011 (3)
K2	0.0167 (5)	0.0075 (4)	0.0163 (5)	−0.0065 (4)	−0.0022 (4)	0.0014 (4)
K3	0.0172 (5)	0.0080 (4)	0.0156 (5)	−0.0044 (4)	−0.0034 (4)	−0.0020 (4)
N3	0.0166 (18)	0.0182 (17)	0.0096 (16)	−0.0053 (14)	−0.0019 (13)	0.0032 (13)
N4	0.0192 (19)	0.0188 (18)	0.0187 (19)	−0.0099 (15)	0.0020 (15)	−0.0051 (14)
N1	0.0215 (19)	0.0164 (17)	0.0104 (17)	−0.0066 (14)	−0.0051 (14)	0.0010 (13)
N2	0.020 (2)	0.030 (2)	0.037 (3)	−0.0057 (17)	−0.0057 (18)	0.006 (2)
N5	0.018 (2)	0.036 (3)	0.045 (3)	−0.0082 (19)	−0.004 (2)	0.012 (2)
C6	0.018 (2)	0.0144 (18)	0.0123 (19)	−0.0064 (15)	−0.0022 (15)	−0.0001 (14)
C4	0.019 (2)	0.0074 (16)	0.0137 (19)	−0.0076 (14)	−0.0029 (15)	0.0011 (14)
C5	0.016 (2)	0.016 (2)	0.033 (3)	−0.0037 (16)	−0.0025 (18)	0.0030 (17)
C1	0.0159 (19)	0.0104 (17)	0.0136 (19)	−0.0065 (14)	−0.0026 (15)	−0.0040 (14)
C3	0.0150 (19)	0.0171 (19)	0.0077 (17)	−0.0042 (15)	−0.0020 (14)	0.0036 (14)
C2	0.018 (2)	0.023 (2)	0.023 (2)	−0.0085 (17)	−0.0036 (17)	0.0039 (17)
N6	0.024 (2)	0.0174 (18)	0.0123 (18)	−0.0094 (15)	−0.0003 (14)	0.0027 (13)
C7	0.027 (2)	0.0129 (18)	0.0099 (18)	−0.0064 (16)	−0.0014 (16)	−0.0054 (14)
N7	0.019 (2)	0.0197 (19)	0.026 (2)	−0.0070 (15)	−0.0041 (16)	−0.0097 (16)
O1	0.056 (4)	0.035 (3)	0.090 (4)	−0.013 (3)	−0.004 (3)	−0.006 (3)
O2	0.058 (6)	0.070 (6)	0.088 (7)	−0.008 (5)	−0.014 (5)	0.015 (5)
O3	0.052 (6)	0.054 (6)	0.105 (9)	−0.024 (6)	−0.009 (6)	0.022 (6)

Geometric parameters (Å, º)

Mo1—C2	2.120 (4)	K2—N4^{vii}	2.934 (4)
Mo1—C5	2.135 (4)	K2—N7^{vii}	3.096 (4)
Mo1—C1	2.150 (4)	K2—C4	3.109 (4)
Mo1—C4	2.156 (4)	K2—N4	3.134 (4)
Mo1—C6	2.160 (4)	K2—C6	3.150 (4)
Mo1—C3	2.164 (4)	K2—N6	3.181 (4)
Mo1—C7	2.198 (5)	K2—N5	3.188 (4)
K4—N1^{i}	2.836 (4)	K2—C5	3.229 (4)
K4—N2	2.888 (5)	K2—C4^{vii}	3.329 (4)
K4—N3^{ii}	2.948 (4)	K2—C7^{vii}	3.380 (4)
K4—N3^{iii}	2.983 (4)	K3—O2	2.767 (10)
K4—N7^{ii}	3.076 (4)	K3—N6^{viii}	2.788 (4)
K4—N4^{iii}	3.119 (4)	K3—O3	2.806 (11)
K4—N5	3.141 (4)	K3—O1	2.912 (6)
K4—C3^{iii}	3.185 (4)	K3—N1^{ix}	2.989 (4)
K4—C2	3.250 (4)	K3—N7^{ix}	3.039 (4)
K4—C4^{iii}	3.274 (4)	K3—N1	3.081 (4)
K4—C3^{ii}	3.317 (4)	K3—C1	3.093 (4)

K4—C7^{ii}	3.362 (4)	K3—C3	3.125 (4)
K1—N4^{iii}	2.783 (4)	K3—N3	3.140 (4)
K1—O3	2.888 (10)	K3—C2	3.339 (4)
K1—N2	2.900 (4)	K3—N2	3.344 (5)
K1—O2	2.921 (10)	N3—C3	1.140 (6)
K1—N7^{iv}	3.031 (4)	N4—C4	1.161 (6)
K1—N6^{i}	3.046 (4)	N1—C1	1.174 (5)
K1—N1^{i}	3.051 (4)	N2—C2	1.167 (6)
K1—N6^{iv}	3.051 (4)	N5—C5	1.161 (6)
K1—O1	3.075 (6)	C6—N6	1.154 (5)
K1—C6^{i}	3.200 (4)	C7—N7	1.154 (6)
K1—C1^{i}	3.210 (4)	O1—K2^{viii}	3.383 (6)
K1—C7^{iv}	3.362 (4)	O1—H1	0.89 (2)
K2—N3^{v}	2.819 (4)	O1—H2	0.89 (2)
K2—N5^{vi}	2.882 (5)	O3—O3^{x}	1.005 (18)

C2—Mo1—C5	74.20 (16)	C5—K2—C4^{vii}	129.62 (10)
C2—Mo1—C1	75.73 (16)	N3^{v}—K2—C7^{vii}	88.06 (10)
C5—Mo1—C1	126.45 (17)	N5^{vi}—K2—C7^{vii}	86.38 (12)
C2—Mo1—C4	125.11 (16)	N4^{vii}—K2—C7^{vii}	68.44 (11)
C5—Mo1—C4	75.08 (17)	N7^{vii}—K2—C7^{vii}	19.92 (11)
C1—Mo1—C4	155.91 (15)	C4—K2—C7^{vii}	95.04 (10)
C2—Mo1—C6	120.52 (17)	N4—K2—C7^{vii}	74.03 (10)
C5—Mo1—C6	75.93 (16)	C6—K2—C7^{vii}	142.50 (11)
C1—Mo1—C6	82.82 (14)	N6—K2—C7^{vii}	143.29 (11)
C4—Mo1—C6	93.98 (14)	N5—K2—C7^{vii}	133.68 (11)
C2—Mo1—C3	77.55 (16)	C5—K2—C7^{vii}	135.26 (11)
C5—Mo1—C3	122.98 (16)	C4^{vii}—K2—C7^{vii}	48.32 (10)
C1—Mo1—C3	91.60 (14)	O2—K3—N6^{viii}	121.8 (2)
C4—Mo1—C3	82.69 (14)	O2—K3—O3	42.0 (3)
C6—Mo1—C3	158.53 (16)	N6^{viii}—K3—O3	80.6 (2)
C2—Mo1—C7	144.39 (15)	O2—K3—O1	75.4 (3)
C5—Mo1—C7	141.38 (15)	N6^{viii}—K3—O1	74.07 (13)
C1—Mo1—C7	77.75 (15)	O3—K3—O1	51.2 (3)
C4—Mo1—C7	78.21 (15)	O2—K3—N1^{ix}	144.9 (2)
C6—Mo1—C7	78.68 (15)	N6^{viii}—K3—N1^{ix}	77.83 (11)
C3—Mo1—C7	79.88 (14)	O3—K3—N1^{ix}	148.6 (2)
C2—Mo1—K2	125.94 (11)	O1—K3—N1^{ix}	139.68 (15)
C5—Mo1—K2	51.79 (11)	O2—K3—N7^{ix}	66.5 (2)
C1—Mo1—K2	132.54 (9)	N6^{viii}—K3—N7^{ix}	88.30 (11)
C4—Mo1—K2	48.64 (10)	O3—K3—N7^{ix}	69.5 (3)
C6—Mo1—K2	49.76 (10)	O1—K3—N7^{ix}	119.81 (15)
C3—Mo1—K2	131.31 (10)	N1^{ix}—K3—N7^{ix}	87.22 (11)
C7—Mo1—K2	89.59 (10)	O2—K3—N1	76.9 (2)
C2—Mo1—K3	54.13 (11)	N6^{viii}—K3—N1	151.78 (12)
C5—Mo1—K3	128.27 (11)	O3—K3—N1	118.3 (3)
C1—Mo1—K3	47.57 (9)	O1—K3—N1	133.85 (13)
C4—Mo1—K3	131.21 (10)	N1^{ix}—K3—N1	75.93 (11)

C6—Mo1—K3	130.36 (11)	N7^{ix}—K3—N1	80.28 (9)
C3—Mo1—K3	48.53 (10)	O2—K3—C1	83.5 (2)
C7—Mo1—K3	90.34 (10)	N6^{viii}—K3—C1	154.61 (11)
K2—Mo1—K3	179.844 (19)	O3—K3—C1	124.7 (2)
N1^{i}—K4—N2	74.36 (12)	O1—K3—C1	118.24 (13)
N1^{i}—K4—N3^{ii}	79.64 (10)	N1^{ix}—K3—C1	79.58 (10)
N2—K4—N3^{ii}	136.55 (11)	N7^{ix}—K3—C1	102.07 (10)
N1^{i}—K4—N3^{iii}	155.68 (12)	N1—K3—C1	21.93 (10)
N2—K4—N3^{iii}	128.12 (12)	O2—K3—C3	118.1 (2)
N3^{ii}—K4—N3^{iii}	76.83 (11)	N6^{viii}—K3—C3	104.25 (11)
N1^{i}—K4—N7^{ii}	83.60 (11)	O3—K3—C3	129.1 (3)
N2—K4—N7^{ii}	120.85 (12)	O1—K3—C3	80.92 (15)
N3^{ii}—K4—N7^{ii}	89.44 (11)	N1^{ix}—K3—C3	78.63 (10)
N3^{iii}—K4—N7^{ii}	90.10 (11)	N7^{ix}—K3—C3	158.45 (10)
N1^{i}—K4—N4^{iii}	88.28 (10)	N1—K3—C3	80.57 (10)
N2—K4—N4^{iii}	70.89 (11)	C1—K3—C3	59.65 (10)
N3^{ii}—K4—N4^{iii}	74.16 (11)	O2—K3—N3	132.2 (2)
N3^{iii}—K4—N4^{iii}	91.13 (11)	N6^{viii}—K3—N3	83.31 (10)
N7^{ii}—K4—N4^{iii}	162.80 (11)	O3—K3—N3	125.4 (3)
N1^{i}—K4—N5	128.70 (12)	O1—K3—N3	74.17 (15)
N2—K4—N5	86.40 (12)	N1^{ix}—K3—N3	74.35 (10)
N3^{ii}—K4—N5	136.58 (11)	N7^{ix}—K3—N3	160.97 (11)
N3^{iii}—K4—N5	68.27 (11)	N1—K3—N3	99.33 (10)
N7^{ii}—K4—N5	66.26 (12)	C1—K3—N3	79.57 (10)
N4^{iii}—K4—N5	129.74 (12)	C3—K3—N3	20.96 (10)
N1^{i}—K4—C3^{iii}	155.52 (11)	O2—K3—C2	69.3 (2)
N2—K4—C3^{iii}	111.36 (12)	N6^{viii}—K3—C2	137.97 (11)
N3^{ii}—K4—C3^{iii}	80.82 (10)	O3—K3—C2	93.7 (2)
N3^{iii}—K4—C3^{iii}	20.96 (10)	O1—K3—C2	70.34 (13)
N7^{ii}—K4—C3^{iii}	110.84 (11)	N1^{ix}—K3—C2	117.51 (11)
N4^{iii}—K4—C3^{iii}	72.25 (10)	N7^{ix}—K3—C2	128.83 (11)
N5—K4—C3^{iii}	75.78 (11)	N1—K3—C2	65.60 (11)
N1^{i}—K4—C2	88.58 (11)	C1—K3—C2	47.92 (11)
N2—K4—C2	20.85 (11)	C3—K3—C2	48.92 (11)
N3^{ii}—K4—C2	157.13 (11)	N3—K3—C2	66.16 (10)
N3^{iii}—K4—C2	115.65 (11)	O2—K3—N2	54.2 (2)
N7^{ii}—K4—C2	108.80 (11)	N6^{viii}—K3—N2	130.22 (11)
N4^{iii}—K4—C2	86.05 (11)	O3—K3—N2	73.6 (2)
N5—K4—C2	65.55 (11)	O1—K3—N2	56.52 (13)
C3^{iii}—K4—C2	104.29 (11)	N1^{ix}—K3—N2	137.62 (10)
N1^{i}—K4—C4^{iii}	109.02 (10)	N7^{ix}—K3—N2	119.73 (11)
N2—K4—C4^{iii}	77.45 (11)	N1—K3—N2	77.35 (10)
N3^{ii}—K4—C4^{iii}	78.98 (10)	C1—K3—N2	63.97 (10)
N3^{iii}—K4—C4^{iii}	72.38 (10)	C3—K3—N2	64.95 (10)
N7^{ii}—K4—C4^{iii}	160.72 (11)	N3—K3—N2	78.27 (10)
N4^{iii}—K4—C4^{iii}	20.75 (10)	C2—K3—N2	20.11 (10)
N5—K4—C4^{iii}	112.42 (12)	C3—N3—K2^{viii}	154.0 (3)
C3^{iii}—K4—C4^{iii}	52.41 (10)	C3—N3—K4^{xi}	98.7 (3)

C2—K4—C4^{iii}	86.57 (11)	K2^{viii}—N3—K4^{xi}	106.90 (12)
N1^{i}—K4—C3^{ii}	77.63 (10)	C3—N3—K4^{iii}	89.6 (3)
N2—K4—C3^{ii}	148.30 (11)	K2^{viii}—N3—K4^{iii}	89.18 (11)
N3^{ii}—K4—C3^{ii}	19.86 (10)	K4^{xi}—N3—K4^{iii}	103.17 (11)
N3^{iii}—K4—C3^{ii}	78.16 (9)	C3—N3—K3	78.8 (3)
N7^{ii}—K4—C3^{ii}	69.59 (10)	K2^{viii}—N3—K3	92.09 (10)
N4^{iii}—K4—C3^{ii}	93.89 (10)	K4^{xi}—N3—K3	99.76 (11)
N5—K4—C3^{ii}	123.26 (11)	K4^{iii}—N3—K3	155.59 (14)
C3^{iii}—K4—C3^{ii}	88.77 (10)	C4—N4—K1^{iii}	151.2 (3)
C2—K4—C3^{ii}	166.20 (10)	C4—N4—K2^{vii}	99.5 (3)
C4^{iii}—K4—C3^{ii}	98.25 (10)	K1^{iii}—N4—K2^{vii}	109.07 (13)
N1^{i}—K4—C7^{ii}	80.29 (10)	C4—N4—K4^{iii}	87.2 (3)
N2—K4—C7^{ii}	136.66 (12)	K1^{iii}—N4—K4^{iii}	84.27 (10)
N3^{ii}—K4—C7^{ii}	69.42 (10)	K2^{vii}—N4—K4^{iii}	99.88 (11)
N3^{iii}—K4—C7^{ii}	85.77 (10)	C4—N4—K2	78.1 (3)
N7^{ii}—K4—C7^{ii}	20.02 (10)	K1^{iii}—N4—K2	97.92 (11)
N4^{iii}—K4—C7^{ii}	143.19 (10)	K2^{vii}—N4—K2	104.52 (11)
N5—K4—C7^{ii}	82.72 (12)	K4^{iii}—N4—K2	153.20 (14)
C3^{iii}—K4—C7^{ii}	106.31 (10)	C1—N1—K4^{i}	154.3 (3)
C2—K4—C7^{ii}	128.06 (11)	C1—N1—K3^{ix}	99.3 (3)
C4^{iii}—K4—C7^{ii}	144.98 (11)	K4^{i}—N1—K3^{ix}	106.16 (12)
C3^{ii}—K4—C7^{ii}	49.57 (10)	C1—N1—K1^{i}	86.9 (3)
N4^{iii}—K1—O3	126.5 (3)	K4^{i}—N1—K1^{i}	84.65 (10)
N4^{iii}—K1—N2	75.70 (12)	K3^{ix}—N1—K1^{i}	102.10 (11)
O3—K1—N2	79.8 (2)	C1—N1—K3	79.6 (3)
N4^{iii}—K1—O2	132.7 (2)	K4^{i}—N1—K3	97.05 (11)
O3—K1—O2	40.3 (3)	K3^{ix}—N1—K3	104.07 (11)
N2—K1—O2	58.2 (2)	K1^{i}—N1—K3	152.14 (14)
N4^{iii}—K1—N7^{iv}	83.94 (11)	C2—N2—K4	97.4 (3)
O3—K1—N7^{iv}	72.6 (3)	C2—N2—K1	176.0 (4)
N2—K1—N7^{iv}	124.93 (12)	K4—N2—K1	86.55 (12)
O2—K1—N7^{iv}	112.8 (2)	C2—N2—K3	79.7 (3)
N4^{iii}—K1—N6^{i}	151.13 (12)	K4—N2—K3	176.65 (16)
O3—K1—N6^{i}	73.8 (3)	K1—N2—K3	96.37 (12)
N2—K1—N6^{i}	132.09 (12)	C5—N5—K2^{vi}	176.1 (4)
O2—K1—N6^{i}	76.2 (2)	C5—N5—K4	91.7 (3)
N7^{iv}—K1—N6^{i}	83.89 (11)	K2^{vi}—N5—K4	85.04 (11)
N4^{iii}—K1—N1^{i}	90.63 (10)	C5—N5—K2	81.6 (3)
O3—K1—N1^{i}	124.6 (3)	K2^{vi}—N5—K2	101.74 (12)
N2—K1—N1^{i}	71.04 (12)	K4—N5—K2	173.03 (16)
O2—K1—N1^{i}	84.5 (2)	N6—C6—Mo1	178.8 (4)
N7^{iv}—K1—N1^{i}	160.62 (11)	N6—C6—K2	81.0 (3)
N6^{i}—K1—N1^{i}	92.27 (10)	Mo1—C6—K2	98.68 (14)
N4^{iii}—K1—N6^{iv}	79.25 (11)	N6—C6—K1^{i}	71.9 (3)
O3—K1—N6^{iv}	143.8 (2)	Mo1—C6—K1^{i}	108.80 (14)
N2—K1—N6^{iv}	135.46 (11)	K2—C6—K1^{i}	146.00 (14)
O2—K1—N6^{iv}	141.7 (2)	N6—C6—K1^{xii}	62.6 (3)
N7^{iv}—K1—N6^{iv}	87.65 (11)	Mo1—C6—K1^{xii}	116.23 (15)

N6^{i}—K1—N6^{iv}	74.18 (11)	K2—C6—K1^{xii}	90.47 (10)
N1^{i}—K1—N6^{iv}	73.05 (10)	K1^{i}—C6—K1^{xii}	94.81 (10)
N4^{iii}—K1—O1	77.78 (15)	N4—C4—Mo1	178.7 (4)
O3—K1—O1	48.9 (3)	N4—C4—K2	80.5 (3)
N2—K1—O1	59.89 (13)	Mo1—C4—K2	100.01 (13)
O2—K1—O1	70.8 (3)	N4—C4—K4^{iii}	72.1 (3)
N7^{iv}—K1—O1	66.03 (12)	Mo1—C4—K4^{iii}	107.93 (13)
N6^{i}—K1—O1	120.18 (14)	K2—C4—K4^{iii}	144.67 (13)
N1^{i}—K1—O1	130.92 (12)	N4—C4—K2^{vii}	60.4 (3)
N6^{iv}—K1—O1	146.53 (13)	Mo1—C4—K2^{vii}	118.33 (15)
N4^{iii}—K1—C6^{i}	155.00 (11)	K2—C4—K2^{vii}	96.36 (10)
O3—K1—C6^{i}	78.4 (3)	K4^{iii}—C4—K2^{vii}	89.15 (10)
N2—K1—C6^{i}	115.01 (12)	N5—C5—Mo1	174.1 (4)
O2—K1—C6^{i}	66.0 (2)	N5—C5—K2	77.6 (3)
N7^{iv}—K1—C6^{i}	104.87 (11)	Mo1—C5—K2	96.91 (15)
N6^{i}—K1—C6^{i}	21.12 (10)	N5—C5—K4	68.2 (3)
N1^{i}—K1—C6^{i}	73.26 (10)	Mo1—C5—K4	117.19 (16)
N6^{iv}—K1—C6^{i}	77.82 (10)	K2—C5—K4	145.76 (14)
O1—K1—C6^{i}	127.22 (14)	N1—C1—Mo1	179.0 (3)
N4^{iii}—K1—C1^{i}	112.02 (11)	N1—C1—K3	78.5 (3)
O3—K1—C1^{i}	108.7 (3)	Mo1—C1—K3	101.55 (13)
N2—K1—C1^{i}	78.95 (12)	N1—C1—K1^{i}	71.7 (3)
O2—K1—C1^{i}	71.3 (2)	Mo1—C1—K1^{i}	108.74 (13)
N7^{iv}—K1—C1^{i}	154.97 (11)	K3—C1—K1^{i}	141.57 (14)
N6^{i}—K1—C1^{i}	72.96 (10)	N1—C1—K3^{ix}	60.7 (3)
N1^{i}—K1—C1^{i}	21.43 (10)	Mo1—C1—K3^{ix}	118.36 (15)
N6^{iv}—K1—C1^{i}	77.16 (10)	K3—C1—K3^{ix}	95.18 (10)
O1—K1—C1^{i}	134.42 (12)	K1^{i}—C1—K3^{ix}	90.83 (9)
C6^{i}—K1—C1^{i}	52.82 (10)	N3—C3—Mo1	178.1 (4)
N4^{iii}—K1—C7^{iv}	78.81 (10)	N3—C3—K3	80.3 (3)
O3—K1—C7^{iv}	90.8 (2)	Mo1—C3—K3	100.21 (13)
N2—K1—C7^{iv}	139.24 (12)	N3—C3—K4^{iii}	69.5 (3)
O2—K1—C7^{iv}	129.9 (2)	Mo1—C3—K4^{iii}	110.74 (14)
N7^{iv}—K1—C7^{iv}	19.95 (11)	K3—C3—K4^{iii}	143.04 (14)
N6^{i}—K1—C7^{iv}	80.85 (10)	N3—C3—K4^{xi}	61.5 (3)
N1^{i}—K1—C7^{iv}	140.67 (11)	Mo1—C3—K4^{xi}	116.62 (15)
N6^{iv}—K1—C7^{iv}	67.80 (10)	K3—C3—K4^{xi}	92.56 (10)
O1—K1—C7^{iv}	84.04 (12)	K4^{iii}—C3—K4^{xi}	91.23 (10)
C6^{i}—K1—C7^{iv}	101.40 (10)	N2—C2—Mo1	175.1 (4)
C1^{i}—K1—C7^{iv}	140.79 (11)	N2—C2—K4	61.8 (3)
N3^{v}—K2—N5^{vi}	74.20 (12)	Mo1—C2—K4	123.11 (17)
N3^{v}—K2—N4^{vii}	79.00 (11)	N2—C2—K3	80.2 (3)
N5^{vi}—K2—N4^{vii}	143.65 (11)	Mo1—C2—K3	94.90 (14)
N3^{v}—K2—N7^{vii}	92.82 (10)	K4—C2—K3	141.94 (14)
N5^{vi}—K2—N7^{vii}	69.19 (12)	C6—N6—K3^{v}	154.2 (3)
N4^{vii}—K2—N7^{vii}	88.33 (11)	C6—N6—K1^{i}	87.0 (3)
N3^{v}—K2—C4	154.84 (11)	K3^{v}—N6—K1^{i}	93.00 (11)
N5^{vi}—K2—C4	130.86 (12)	C6—N6—K1^{xii}	97.7 (3)

N4^{vii}—K2—C4	78.98 (11)	K3^{v}—N6—K1^{xii}	107.02 (12)
N7^{vii}—K2—C4	98.55 (10)	K1^{i}—N6—K1^{xii}	105.82 (11)
N3^{v}—K2—N4	152.82 (11)	C6—N6—K2	78.0 (3)
N5^{vi}—K2—N4	123.51 (12)	K3^{v}—N6—K2	91.82 (10)
N4^{vii}—K2—N4	75.48 (11)	K1^{i}—N6—K2	154.44 (14)
N7^{vii}—K2—N4	77.34 (10)	K1^{xii}—N6—K2	96.69 (11)
C4—K2—N4	21.43 (10)	N7—C7—Mo1	178.9 (4)
N3^{v}—K2—C6	103.06 (11)	N7—C7—K1^{xii}	63.6 (2)
N5^{vi}—K2—C6	131.04 (12)	Mo1—C7—K1^{xii}	116.63 (15)
N4^{vii}—K2—C6	78.58 (11)	N7—C7—K4^{xi}	65.9 (3)
N7^{vii}—K2—C6	156.93 (10)	Mo1—C7—K4^{xi}	113.90 (14)
C4—K2—C6	60.55 (10)	K1^{xii}—C7—K4^{xi}	129.43 (15)
N4—K2—C6	81.01 (10)	N7—C7—K2^{vii}	66.0 (3)
N3^{v}—K2—N6	82.07 (9)	Mo1—C7—K2^{vii}	115.03 (15)
N5^{vi}—K2—N6	123.91 (12)	K1^{xii}—C7—K2^{vii}	83.05 (10)
N4^{vii}—K2—N6	74.98 (11)	K4^{xi}—C7—K2^{vii}	74.38 (9)
N7^{vii}—K2—N6	163.17 (11)	N7—C7—K3^{ix}	62.5 (3)
C4—K2—N6	80.61 (10)	Mo1—C7—K3^{ix}	116.39 (16)
N4—K2—N6	100.02 (10)	K1^{xii}—C7—K3^{ix}	77.65 (9)
C6—K2—N6	21.00 (10)	K4^{xi}—C7—K3^{ix}	82.09 (10)
N3^{v}—K2—N5	127.59 (12)	K2^{vii}—C7—K3^{ix}	128.45 (14)
N5^{vi}—K2—N5	78.26 (12)	C7—N7—K1^{xii}	96.4 (3)
N4^{vii}—K2—N5	138.08 (11)	C7—N7—K3^{ix}	97.8 (3)
N7^{vii}—K2—N5	117.52 (11)	K1^{xii}—N7—K3^{ix}	88.49 (10)
C4—K2—N5	65.63 (11)	C7—N7—K4^{xi}	94.1 (3)
N4—K2—N5	78.79 (11)	K1^{xii}—N7—K4^{xi}	169.07 (15)
C6—K2—N5	65.05 (11)	K3^{ix}—N7—K4^{xi}	93.01 (11)
N6—K2—N5	77.64 (11)	C7—N7—K2^{vii}	94.0 (3)
N3^{v}—K2—C5	136.31 (12)	K1^{xii}—N7—K2^{vii}	93.66 (11)
N5^{vi}—K2—C5	99.08 (12)	K3^{ix}—N7—K2^{vii}	167.66 (15)
N4^{vii}—K2—C5	117.27 (11)	K4^{xi}—N7—K2^{vii}	82.64 (10)
N7^{vii}—K2—C5	125.79 (11)	K3—O1—K1	102.27 (19)
C4—K2—C5	48.67 (11)	K3—O1—K2^{viii}	85.71 (15)
N4—K2—C5	66.00 (11)	K1—O1—K2^{viii}	169.2 (2)
C6—K2—C5	48.91 (11)	K3—O1—H1	133 (5)
N6—K2—C5	65.90 (11)	K1—O1—H1	109 (5)
N5—K2—C5	20.83 (10)	K2^{viii}—O1—H1	70 (5)
N3^{v}—K2—C4^{vii}	79.86 (10)	K3—O1—H2	110 (4)
N5^{vi}—K2—C4^{vii}	128.22 (11)	K1—O1—H2	97 (5)
N4^{vii}—K2—C4^{vii}	20.13 (10)	K2^{viii}—O1—H2	73 (5)
N7^{vii}—K2—C4^{vii}	68.23 (10)	H1—O1—H2	100 (3)
C4—K2—C4^{vii}	83.64 (10)	K3—O2—K1	110.1 (4)
N4—K2—C4^{vii}	72.96 (10)	O3^{x}—O3—K3	124.8 (14)
C6—K2—C4^{vii}	97.92 (10)	O3^{x}—O3—K1	121.3 (14)

N6—K2—C4^{vii}	95.04 (10)	K3—O3—K1	109.9 (3)
N5—K2—C4^{vii}	149.11 (11)		

Symmetry codes: (i) −*x*, −*y*, −*z*+1; (ii) *x*+1, *y*, *z*; (iii) −*x*, −*y*, −*z*; (iv) *x*+1, *y*−1, *z*; (v) *x*, *y*+1, *z*; (vi) −*x*, −*y*+1, −*z*; (vii) −*x*−1, −*y*+1, −*z*; (viii) *x*, *y*−1, *z*; (ix) −*x*−1, −*y*, −*z*+1; (x) −*x*, −*y*−1, −*z*+1; (xi) *x*−1, *y*, *z*; (xii) *x*−1, *y*+1, *z*.