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Poly[tetra-μ_2-aqua-diaqua-μ_2-chlorido-di-μ_2-oxo-dioxo[μ_7-*N*,*N*,*N*′,*N*′-(propane-1,2-diyldinitrilo)tetraacetato]dimolybdenum(V)trisodium(I)]

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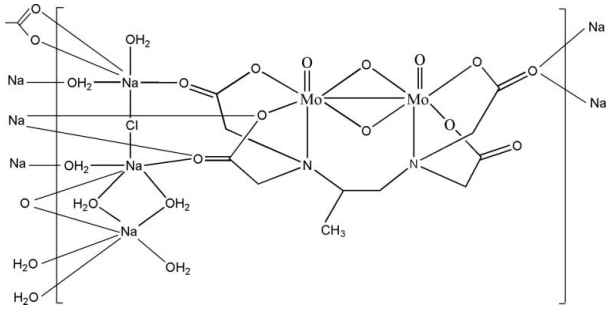
Key indicators: single-crystal X-ray study; T = 293 K; mean σ(C–C) = 0.008 Å; R factor = 0.036; wR factor = 0.061; data-to-parameter ratio = 11.5.

The title compound, $[Na_3Mo_2(C_{11}H_{14}N_2O_8)ClO_4(H_2O)_6]_n$, contains dioxo-bridged Mo_2O_4 units. Each Mo^V atom has a distorted octahedral coordination geometry, and the two coordination octahedra share a common edge to form a dinuclear complex. The dinuclear complexes lie in layers, between which Na^+ cations, Cl^- anions and water molecules form a complex network.

Related literature

For related literature, see: Blake *et al.* (1964); Haynes & Sawyer (1967); Hong & Liu (1985); Kimiko *et al.* (1980); Mitchell & Williams (1962); Pecsok & Sawyer (1956).



Experimental

Crystal data

$[Na_3Mo_2(C_{11}H_{14}N_2O_8)ClO_4(H_2O)_6]$
M_r = 770.64
Monoclinic, *Cc*
a = 9.419 (3) Å
b = 10.443 (3) Å
c = 25.409 (9) Å
β = 97.262 (5)°
V = 2479.2 (15) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 1.26 mm^{-1}
T = 293.2 K
0.18 × 0.15 × 0.12 mm

Data collection

Rigaku Mercury70 CCD diffractometer
Absorption correction: multi-scan (*CrystalClear*; Rigaku, 2000)
T_{min} = 0.793, T_{max} = 0.866
9442 measured reflections
4257 independent reflections
3894 reflections with $I > 2\sigma(I)$
R_{int} = 0.039

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.036
$wR(F^2)$ = 0.061
S = 1.07
4257 reflections
371 parameters
14 restraints
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.64 e Å^{-3}
$\Delta\rho_{min}$ = −0.60 e Å^{-3}
Absolute structure: Flack (1983), 1408 Friedel pairs
Flack parameter: 0.00 (3)

Data collection: *CrystalClear* (Rigaku, 2000); cell refinement: *CrystalClear*; data reduction: *CrystalClear*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997*a*); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997*a*); molecular graphics: *SHELXTL* (Sheldrick, 1997*b*); 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: BI2185).

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supplementary materials

Acta Cryst. (2007). E**63**, m2106 [**doi:10.1107/S1600536807021769**]

Poly[tetra-μ_2-aqua-diaqua-μ_2-chlorido-di-μ_2-oxo-dioxo[μ_7-*N*,*N*,*N*',*N*'-(propane-1,2-diyldinitrilo)tetraacetato]dimolybdenum(V)trisodium(I)]

Z.-H. Dang, J. Zhao, Y.-J. Wang, Y.-Z. Ye and L. Xu

Comment

Although dinuclear oxomolybdenum(V) compounds of the chelating ligands ethylenediaminetetraacetate (edta) and propylenediaminetetraacetate (PDTA) were widely studied during the late 1950 s and early 1960 s (Pecsok & Sawyer, 1956; Mitchell & Williams, 1962; Blake *et al.*, 1964), the first such crystal structure of a potassium salt did not appear until 1967 (Haynes & Sawyer, 1967). Structures of sodium salts were reported in 1980 (Kimiko *et al.*, 1980) and 1985 (Hong & Liu, 1985). This paper describes the synthesis of a new compound, trisodium bis(μ$_2$-oxo)-(μ$_2$-propylene-1,2-diaminetetraacetato)-bis(oxo- molybdenum(V)) chloride hexahydrate, (I).

The asymmetric unit of (I) is shown in Fig. 1. The PDTA ligand coordinates to each Mo atom through two carboxylato-O atoms and an N atom, together with one terminal and two bridging O atoms to complete a distorted octahedral environment. Two terminal oxo ligands lie *cis* to the bridging oxide. The N atom and carboxlato-O atoms occupy the axial (*trans* to the terminal O) and equatorial positions, respectively. The short Mo···Mo distance (2.5483 (10) Å) indicates a strong interaction between the two Mo atoms.

As shown in Fig. 2, the $[Mo_2O_4(PDTA)]^{2-}$ complexes lie in layers parallel to the (001) planes. Between these layers, Na^+ cations, Cl^- anions and water molecules form a complex network stabilized by O—H···O and O—H···Cl hydrogen bonds (Table 2).

Experimental

A mixture of H_4PDTA (1.58 g, 5 mmol) and $Na_2MoO_4{\cdot}2H_2O$ (2.42 g, 10 mmol) was dissolved in 10 ml water at room temperature. $Na_2S_2O_4$ (0.87 g, 5 mmol) was added while stirring, and the color of the solution changed to red. Orange crystals were obtained after standing for several days.

Refinement

H atoms bound to C atoms were placed in calculated positions and treated using a riding-model approximation (C—H = 0.99 Å for methylene H atoms with U_{iso}(H) = 1.2U_{eq}(C), C—H = 0.98 Å for methyl H atoms with U_{iso}(H) = 1.5U_{eq}(C) and C—H = 1.00 Å for tertiary H with U_{iso}(H) = 1.2U_{eq}(C)). H atoms bound to O atoms were visible in difference Fourier maps and were included in the refinement with O—H distances restrained to 0.90 (2) Å, and with U_{iso}(H) = 1.5U_{eq}(O).

Figures

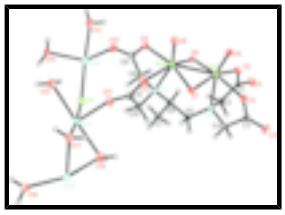

Fig. 1. The asymmetric unit in (I), with displacement ellipsoids drawn at the 30% probability level for non-H atoms.

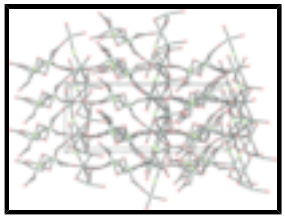

Fig. 2. Packing diagram of (I) viewed along the *a* axis. H atoms have been omitted for clarity.

Poly[tetra-μ$_2$-aqua-diaqua-μ$_2$-chlorido-di-μ$_2$-oxo-dioxo[μ$_7$-*N*,*N*,*N*',*N*'- (propane-1,2-diyldinitrilo)tetraacetato]dimolybdenum(*V*)trisodium(I)]

Crystal data

$[Na_3Mo_2(C_{11}H_{14}N_2O_8)ClO_4(H_2O)_6]$	F_{000} = 1536
M_r = 770.64	D_x = 2.065 Mg m^{-3}
Monoclinic, *Cc*	Mo *K*α radiation λ = 0.71073 Å
Hall symbol: C -2yc	Cell parameters from 3223 reflections
a = 9.419 (3) Å	θ = 2.4–27.5º
b = 10.443 (3) Å	μ = 1.26 mm^{-1}
c = 25.409 (9) Å	*T* = 293.2 K
β = 97.262 (5)º	Block, orange
V = 2479.2 (15) Å^3	0.18 × 0.15 × 0.12 mm
Z = 4	

Data collection

Rigaku Mercury70 CCD diffractometer	4257 independent reflections
Radiation source: fine-focus sealed tube	3894 reflections with $I > 2\sigma(I)$
Monochromator: graphite	R_{int} = 0.039
T = 293(2) K	θ_{max} = 27.5º
ω scans	θ_{min} = 2.9º
Absorption correction: multi-scan (CrystalClear; Rigaku, 2000)	*h* = −12→11
T_{min} = 0.793, T_{max} = 0.866	*k* = −13→13
9442 measured reflections	*l* = −27→32

Refinement

Refinement on F^2	Hydrogen site location: inferred from neighbouring sites
Least-squares matrix: full	H atoms treated by a mixture of independent and constrained refinement
$R[F^2 > 2\sigma(F^2)] = 0.036$	$w = 1/[\sigma^2(F_o^2) + (0.0125P)^2 + 3.2472P]$ where $P = (F_o^2 + 2F_c^2)/3$
$wR(F^2) = 0.061$	$(\Delta/\sigma)_{max} = 0.001$
$S = 1.07$	$\Delta\rho_{max} = 0.64$ e Å^{-3}
4257 reflections	$\Delta\rho_{min} = -0.60$ e Å^{-3}
371 parameters	Extinction correction: none
14 restraints	Absolute structure: Flack (1983), 1408 Friedel pairs
Primary atom site location: structure-invariant direct methods	Flack parameter: 0.00 (3)
Secondary atom site location: difference Fourier map	

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}
Mo1	0.68634 (4)	0.27602 (4)	0.774649 (19)	0.01927 (10)
Mo2	0.72208 (4)	0.17925 (4)	0.86745 (2)	0.01976 (10)
Cl1	1.23667 (17)	0.05188 (14)	1.04123 (7)	0.0373 (4)
C1	0.8469 (6)	0.2226 (5)	0.6825 (2)	0.0211 (12)
C2	0.9743 (6)	0.2587 (5)	0.7218 (2)	0.0268 (13)
H2A	1.0244	0.1800	0.7357	0.032*
H2B	1.0420	0.3100	0.7037	0.032*
C3	0.7845 (6)	0.5120 (5)	0.7215 (2)	0.0227 (12)
C4	0.9243 (6)	0.4734 (5)	0.7522 (2)	0.0264 (13)
H4A	1.0025	0.4918	0.7307	0.032*
H4B	0.9410	0.5255	0.7850	0.032*
C5	1.0376 (5)	0.3277 (5)	0.81541 (19)	0.0185 (11)
H5A	1.0049	0.3834	0.8430	0.022*
H5B	1.1286	0.3637	0.8063	0.022*
C6	1.0684 (5)	0.1933 (5)	0.8395 (2)	0.0224 (12)

H6	1.0390	0.1292	0.8110	0.027*
C7	1.2296 (6)	0.1802 (5)	0.8547 (2)	0.0288 (14)
H7A	1.2610	0.2382	0.8843	0.043*
H7B	1.2788	0.2023	0.8242	0.043*
H7C	1.2526	0.0918	0.8655	0.043*
C8	1.0155 (6)	0.0285 (5)	0.9010 (2)	0.0289 (13)
H8A	1.0308	−0.0218	0.8692	0.035*
H8B	1.1049	0.0254	0.9261	0.035*
C9	0.8976 (6)	−0.0300 (5)	0.9264 (2)	0.0251 (12)
C10	1.0260 (6)	0.2485 (5)	0.9324 (2)	0.0272 (13)
H10A	1.0504	0.3350	0.9204	0.033*
H10B	1.1123	0.2122	0.9534	0.033*
C11	0.9067 (6)	0.2585 (5)	0.9668 (2)	0.0226 (12)
N1	0.9287 (5)	0.3343 (4)	0.76691 (17)	0.0190 (10)
N2	0.9832 (5)	0.1654 (4)	0.88510 (17)	0.0187 (9)
O1	0.8635 (5)	0.1945 (4)	0.63630 (16)	0.0370 (11)
O2	0.7257 (4)	0.2154 (3)	0.69963 (15)	0.0285 (9)
O3	0.7812 (5)	0.5935 (4)	0.68608 (16)	0.0374 (11)
O4	0.6737 (4)	0.4579 (3)	0.73618 (16)	0.0281 (9)
O5	0.5061 (4)	0.2665 (3)	0.76182 (16)	0.0278 (9)
O6	0.7564 (4)	0.1099 (3)	0.79953 (14)	0.0208 (8)
O7	0.7362 (4)	0.3533 (3)	0.84310 (15)	0.0253 (9)
O8	0.5492 (4)	0.1545 (4)	0.87545 (15)	0.0288 (9)
O9	0.7688 (4)	0.0026 (4)	0.90722 (16)	0.0310 (10)
O10	0.9228 (4)	−0.1099 (4)	0.96216 (16)	0.0338 (10)
O11	0.9301 (5)	0.2746 (4)	1.01484 (15)	0.0351 (11)
O12	0.7777 (4)	0.2559 (4)	0.94321 (16)	0.0311 (10)
O13	1.0124 (5)	−0.3908 (5)	0.9867 (2)	0.0425 (11)
H13A	0.997 (8)	−0.390 (7)	0.9509 (8)	0.064*
H13B	0.930 (5)	−0.413 (7)	0.998 (3)	0.064*
O14	1.1359 (5)	−0.2467 (4)	1.10336 (17)	0.0342 (10)
H14B	1.183 (6)	−0.195 (5)	1.127 (2)	0.051*
H14A	1.046 (3)	−0.268 (6)	1.105 (3)	0.051*
O15	1.0060 (5)	0.0945 (4)	1.12619 (18)	0.0406 (11)
H15A	1.047 (7)	0.048 (6)	1.103 (2)	0.061*
H15B	1.051 (7)	0.062 (6)	1.1551 (17)	0.061*
O16	1.2246 (5)	0.4494 (4)	1.03599 (18)	0.0367 (11)
H16B	1.292 (6)	0.423 (6)	1.017 (2)	0.055*
H16A	1.172 (7)	0.484 (6)	1.008 (2)	0.055*
O17	1.0118 (5)	0.4499 (4)	1.11941 (16)	0.0356 (10)
H17B	0.978 (7)	0.417 (6)	1.1470 (18)	0.053*
H17A	0.962 (7)	0.487 (5)	1.0915 (18)	0.053*
O18	1.2844 (5)	0.5725 (4)	1.21464 (19)	0.0413 (12)
H18A	1.301 (8)	0.510 (5)	1.239 (2)	0.062*
H18B	1.361 (5)	0.615 (6)	1.231 (3)	0.062*
Na1	1.1254 (3)	0.2723 (2)	1.08039 (9)	0.0319 (6)
Na2	1.1410 (3)	−0.1912 (2)	1.01298 (9)	0.0318 (5)
Na3	1.2537 (3)	0.5442 (2)	1.12192 (10)	0.0366 (6)

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Mo1	0.0151 (2)	0.0204 (2)	0.0218 (2)	0.00048 (19)	0.00022 (17)	0.0006 (2)
Mo2	0.0139 (2)	0.0257 (2)	0.0195 (2)	0.0006 (2)	0.00159 (17)	−0.0003 (2)
Cl1	0.0354 (9)	0.0337 (8)	0.0426 (9)	0.0054 (6)	0.0043 (7)	−0.0019 (7)
C1	0.025 (3)	0.018 (3)	0.020 (3)	0.000 (2)	0.000 (2)	0.001 (2)
C2	0.024 (3)	0.035 (3)	0.021 (3)	−0.001 (2)	0.004 (2)	−0.004 (2)
C3	0.028 (3)	0.016 (3)	0.024 (3)	0.000 (2)	0.001 (2)	0.000 (2)
C4	0.028 (3)	0.021 (3)	0.029 (3)	−0.007 (2)	−0.001 (2)	0.005 (2)
C5	0.018 (3)	0.025 (3)	0.013 (2)	−0.004 (2)	0.002 (2)	0.001 (2)
C6	0.015 (3)	0.032 (3)	0.021 (3)	−0.001 (2)	0.004 (2)	−0.002 (2)
C7	0.016 (3)	0.034 (3)	0.037 (4)	0.001 (2)	0.005 (3)	0.001 (3)
C8	0.026 (3)	0.031 (3)	0.031 (3)	0.011 (2)	0.010 (3)	0.009 (3)
C9	0.028 (3)	0.023 (3)	0.026 (3)	0.006 (2)	0.009 (2)	0.001 (2)
C10	0.023 (3)	0.043 (3)	0.015 (3)	0.000 (2)	0.001 (2)	0.002 (2)
C11	0.021 (3)	0.025 (3)	0.021 (3)	0.002 (2)	0.002 (2)	−0.002 (2)
N1	0.019 (3)	0.022 (2)	0.016 (2)	−0.0031 (18)	−0.0005 (18)	0.0039 (18)
N2	0.014 (2)	0.021 (2)	0.020 (2)	−0.0009 (17)	0.0020 (18)	0.0043 (18)
O1	0.043 (3)	0.049 (3)	0.019 (2)	0.001 (2)	0.0041 (19)	−0.0085 (19)
O2	0.024 (2)	0.041 (2)	0.020 (2)	−0.0071 (19)	−0.0004 (17)	−0.0055 (17)
O3	0.044 (3)	0.038 (2)	0.031 (2)	0.002 (2)	0.006 (2)	0.0136 (19)
O4	0.024 (2)	0.027 (2)	0.033 (2)	0.0016 (17)	0.0004 (18)	0.0102 (17)
O5	0.016 (2)	0.027 (2)	0.039 (2)	−0.0007 (16)	−0.0031 (18)	0.0042 (18)
O6	0.019 (2)	0.0199 (18)	0.023 (2)	−0.0016 (15)	0.0012 (15)	−0.0052 (15)
O7	0.022 (2)	0.0191 (18)	0.035 (2)	0.0009 (15)	0.0028 (18)	0.0003 (16)
O8	0.015 (2)	0.044 (2)	0.028 (2)	0.0015 (17)	0.0058 (17)	0.0010 (18)
O9	0.019 (2)	0.034 (2)	0.039 (3)	−0.0023 (16)	−0.0009 (18)	0.0154 (18)
O10	0.034 (3)	0.032 (2)	0.035 (2)	0.0065 (18)	0.0038 (19)	0.0159 (19)
O11	0.035 (3)	0.048 (3)	0.021 (2)	0.007 (2)	−0.0019 (19)	−0.010 (2)
O12	0.016 (2)	0.047 (3)	0.030 (2)	0.0011 (18)	−0.0013 (17)	−0.0090 (19)
O13	0.036 (3)	0.049 (3)	0.043 (3)	0.002 (2)	0.008 (2)	0.012 (2)
O14	0.038 (3)	0.039 (3)	0.026 (2)	−0.011 (2)	0.007 (2)	−0.0063 (18)
O15	0.040 (3)	0.048 (3)	0.034 (3)	−0.001 (2)	0.005 (2)	0.005 (2)
O16	0.034 (3)	0.049 (3)	0.028 (2)	0.005 (2)	0.007 (2)	0.003 (2)
O17	0.033 (3)	0.050 (3)	0.024 (3)	0.003 (2)	0.0031 (19)	0.000 (2)
O18	0.040 (3)	0.045 (3)	0.036 (3)	−0.016 (2)	−0.005 (2)	0.003 (2)
Na1	0.0319 (14)	0.0294 (12)	0.0328 (13)	−0.0010 (10)	−0.0016 (11)	−0.0003 (10)
Na2	0.0358 (15)	0.0351 (13)	0.0266 (13)	0.0055 (11)	0.0117 (11)	0.0021 (10)
Na3	0.0347 (15)	0.0404 (14)	0.0336 (14)	−0.0030 (11)	−0.0001 (11)	−0.0021 (11)

Geometric parameters (Å, °)

Mo1—O5	1.690 (4)	C9—O9	1.295 (6)
Mo1—O7	1.921 (4)	C10—N2	1.495 (7)
Mo1—O6	1.933 (3)	C10—C11	1.513 (8)
Mo1—O2	2.086 (4)	C10—H10A	0.990
Mo1—O4	2.133 (4)	C10—H10B	0.990

Mo1—N1	2.395 (4)	C11—O11	1.224 (6)
Mo1—Mo2	2.5483 (10)	C11—O12	1.285 (7)
Mo2—O8	1.686 (4)	O1—Na1^{i}	2.522 (5)
Mo2—O7	1.930 (4)	O1—Na3^{i}	2.706 (5)
Mo2—O6	1.936 (4)	O10—Na2	2.437 (4)
Mo2—O12	2.089 (4)	O11—Na1	2.320 (4)
Mo2—O9	2.122 (4)	O11—Na2^{ii}	2.742 (5)
Mo2—N2	2.448 (4)	O12—Na2^{ii}	2.384 (5)
Cl1—Na2	2.758 (3)	O13—Na2	2.460 (5)
Cl1—Na1	2.766 (3)	O13—H13A	0.90 (2)
C1—O1	1.238 (7)	O13—H13B	0.89 (5)
C1—O2	1.275 (7)	O14—Na2	2.375 (5)
C1—C2	1.508 (7)	O14—Na3^{iii}	2.468 (5)
C2—N1	1.499 (7)	O14—H14B	0.88 (5)
C2—H2A	0.990	O14—H14A	0.88 (2)
C2—H2B	0.990	O15—Na3^{iv}	2.422 (6)
C3—O3	1.236 (6)	O15—Na1	2.529 (5)
C3—O4	1.282 (7)	O15—H15A	0.89 (6)
C3—C4	1.499 (7)	O15—H15B	0.87 (5)
C4—N1	1.499 (6)	O16—Na3	2.381 (5)
C4—H4A	0.990	O16—Na1	2.415 (5)
C4—H4B	0.990	O16—H16B	0.89 (6)
C5—N1	1.502 (6)	O16—H16A	0.89 (6)
C5—C6	1.543 (7)	O17—Na1	2.416 (5)
C5—H5A	0.990	O17—Na3	2.475 (5)
C5—H5B	0.990	O17—H17B	0.88 (5)
C6—N2	1.519 (7)	O17—H17A	0.89 (5)
C6—C7	1.525 (7)	O18—Na3	2.356 (5)
C6—H6	1.000	O18—H18A	0.90 (5)
C7—H7A	0.980	O18—H18B	0.90 (6)
C7—H7B	0.980	Na1—O1^{v}	2.522 (5)
C7—H7C	0.980	Na2—O12^{vi}	2.384 (5)
C8—C9	1.484 (8)	Na2—O11^{vi}	2.742 (5)
C8—N2	1.506 (6)	Na3—O15^{vii}	2.422 (6)
C8—H8A	0.990	Na3—O14^{viii}	2.468 (5)
C8—H8B	0.990	Na3—O1^{v}	2.706 (5)
C9—O10	1.235 (6)		
O5—Mo1—O7	108.89 (18)	C4—N1—C5	104.1 (4)
O5—Mo1—O6	107.66 (16)	C2—N1—C5	111.8 (4)
O7—Mo1—O6	92.66 (15)	C4—N1—Mo1	105.6 (3)
O5—Mo1—O2	95.52 (18)	C2—N1—Mo1	107.3 (3)
O7—Mo1—O2	154.64 (16)	C5—N1—Mo1	118.7 (3)
O6—Mo1—O2	86.20 (15)	C10—N2—C8	108.1 (4)
O5—Mo1—O4	88.14 (16)	C10—N2—C6	112.7 (4)
O7—Mo1—O4	91.95 (15)	C8—N2—C6	106.2 (4)
O6—Mo1—O4	161.05 (16)	C10—N2—Mo2	106.0 (3)

O2—Mo1—O4	81.81 (16)	C8—N2—Mo2	105.6 (3)
O5—Mo1—N1	160.66 (16)	C6—N2—Mo2	117.7 (3)
O7—Mo1—N1	80.86 (16)	C1—O1—Na1^{i}	106.9 (4)
O6—Mo1—N1	88.01 (15)	C1—O1—Na3^{i}	104.7 (4)
O2—Mo1—N1	73.79 (15)	Na1^{i}—O1—Na3^{i}	75.70 (13)
O4—Mo1—N1	74.59 (15)	C1—O2—Mo1	124.4 (3)
O5—Mo1—Mo2	99.62 (14)	C3—O4—Mo1	121.7 (3)
O7—Mo1—Mo2	48.71 (11)	Mo1—O6—Mo2	82.38 (13)
O6—Mo1—Mo2	48.85 (11)	Mo1—O7—Mo2	82.88 (14)
O2—Mo1—Mo2	135.02 (11)	C9—O9—Mo2	122.4 (3)
O4—Mo1—Mo2	140.39 (11)	C9—O10—Na2	134.1 (4)
N1—Mo1—Mo2	99.26 (10)	C11—O11—Na1	137.8 (4)
O8—Mo2—O7	106.84 (18)	C11—O11—Na2^{ii}	87.0 (3)
O8—Mo2—O6	108.83 (17)	Na1—O11—Na2^{ii}	135.18 (19)
O7—Mo2—O6	92.30 (15)	C11—O12—Mo2	123.4 (4)
O8—Mo2—O12	94.66 (18)	C11—O12—Na2^{ii}	102.5 (3)
O7—Mo2—O12	85.14 (16)	Mo2—O12—Na2^{ii}	132.7 (2)
O6—Mo2—O12	156.04 (16)	Na2—O13—H13A	106 (5)
O8—Mo2—O9	87.41 (17)	Na2—O13—H13B	123 (5)
O7—Mo2—O9	162.28 (15)	H13A—O13—H13B	107 (7)
O6—Mo2—O9	92.95 (15)	Na2—O14—Na3^{iii}	109.70 (19)
O12—Mo2—O9	83.19 (16)	Na2—O14—H14B	116 (5)
O8—Mo2—N2	158.75 (16)	Na3^{iii}—O14—H14B	104 (4)
O7—Mo2—N2	90.35 (15)	Na2—O14—H14A	104 (4)
O6—Mo2—N2	82.19 (15)	Na3^{iii}—O14—H14A	100 (4)
O12—Mo2—N2	74.02 (15)	H14B—O14—H14A	122 (6)
O9—Mo2—N2	73.63 (14)	Na3^{iv}—O15—Na1	129.0 (2)
O8—Mo2—Mo1	98.96 (13)	Na3^{iv}—O15—H15A	111 (5)
O7—Mo2—Mo1	48.41 (11)	Na1—O15—H15A	81 (5)
O6—Mo2—Mo1	48.76 (10)	Na3^{iv}—O15—H15B	109 (5)
O12—Mo2—Mo1	133.55 (12)	Na1—O15—H15B	118 (5)
O9—Mo2—Mo1	141.29 (12)	H15A—O15—H15B	99 (6)
N2—Mo2—Mo1	101.84 (10)	Na3—O16—Na1	84.05 (17)
Na2—Cl1—Na1	137.16 (10)	Na3—O16—H16B	128 (5)
O1—C1—O2	122.4 (5)	Na1—O16—H16B	111 (4)
O1—C1—C2	120.1 (5)	Na3—O16—H16A	123 (5)
O2—C1—C2	117.4 (5)	Na1—O16—H16A	118 (5)
O1—C1—Na1^{i}	50.7 (3)	H16B—O16—H16A	93 (6)
O2—C1—Na1^{i}	75.6 (3)	Na1—O17—Na3	82.06 (17)
C2—C1—Na1^{i}	160.0 (4)	Na1—O17—H17B	105 (5)
N1—C2—C1	110.9 (5)	Na3—O17—H17B	124 (5)
N1—C2—H2A	109.5	Na1—O17—H17A	103 (4)
C1—C2—H2A	109.5	Na3—O17—H17A	104 (5)
N1—C2—H2B	109.5	H17B—O17—H17A	127 (7)
C1—C2—H2B	109.5	Na3—O18—H18A	125 (5)
H2A—C2—H2B	108.0	Na3—O18—H18B	120 (5)

O3—C3—O4	124.7 (5)	H18A—O18—H18B	90 (6)
O3—C3—C4	120.2 (5)	O11—Na1—O16	88.59 (17)
O4—C3—C4	115.0 (5)	O11—Na1—O17	86.27 (16)
N1—C4—C3	112.7 (4)	O16—Na1—O17	79.71 (17)
N1—C4—H4A	109.1	O11—Na1—O1^{v}	166.07 (18)
C3—C4—H4A	109.1	O16—Na1—O1^{v}	77.81 (16)
N1—C4—H4B	109.1	O17—Na1—O1^{v}	94.12 (17)
C3—C4—H4B	109.1	O11—Na1—O15	88.83 (17)
H4A—C4—H4B	107.8	O16—Na1—O15	176.26 (19)
N1—C5—C6	116.2 (4)	O17—Na1—O15	97.42 (18)
N1—C5—H5A	108.2	O1^{v}—Na1—O15	104.90 (16)
C6—C5—H5A	108.2	O11—Na1—Cl1	93.00 (13)
N1—C5—H5B	108.2	O16—Na1—Cl1	106.41 (15)
C6—C5—H5B	108.2	O17—Na1—Cl1	173.83 (16)
H5A—C5—H5B	107.4	O1^{v}—Na1—Cl1	88.07 (13)
N2—C6—C7	113.2 (4)	O15—Na1—Cl1	76.43 (13)
N2—C6—C5	112.7 (4)	O14—Na2—O12^{vi}	137.83 (18)
C7—C6—C5	108.3 (4)	O14—Na2—O10	118.61 (18)
N2—C6—H6	107.4	O12^{vi}—Na2—O10	100.68 (16)
C7—C6—H6	107.4	O14—Na2—O13	89.24 (17)
C5—C6—H6	107.4	O12^{vi}—Na2—O13	83.98 (17)
C6—C7—H7A	109.5	O10—Na2—O13	78.34 (16)
C6—C7—H7B	109.5	O14—Na2—O11^{vi}	95.33 (16)
H7A—C7—H7B	109.5	O12^{vi}—Na2—O11^{vi}	49.73 (13)
C6—C7—H7C	109.5	O10—Na2—O11^{vi}	145.47 (16)
H7A—C7—H7C	109.5	O13—Na2—O11^{vi}	110.36 (17)
H7B—C7—H7C	109.5	O14—Na2—Cl1	91.07 (13)
C9—C8—N2	111.7 (4)	O12^{vi}—Na2—Cl1	102.55 (13)
C9—C8—H8A	109.3	O10—Na2—Cl1	92.49 (12)
N2—C8—H8A	109.3	O13—Na2—Cl1	169.69 (16)
C9—C8—H8B	109.3	O11^{vi}—Na2—Cl1	79.88 (12)
N2—C8—H8B	109.3	O18—Na3—O16	162.65 (19)
H8A—C8—H8B	107.9	O18—Na3—O15^{vii}	86.12 (18)
O10—C9—O9	122.6 (5)	O16—Na3—O15^{vii}	97.47 (19)
O10—C9—C8	120.9 (5)	O18—Na3—O14^{viii}	94.32 (17)
O9—C9—C8	116.5 (5)	O16—Na3—O14^{viii}	101.28 (17)
N2—C10—C11	111.1 (4)	O15^{vii}—Na3—O14^{viii}	103.37 (17)
N2—C10—H10A	109.4	O18—Na3—O17	94.12 (17)
C11—C10—H10A	109.4	O16—Na3—O17	79.18 (17)
N2—C10—H10B	109.4	O15^{vii}—Na3—O17	169.07 (18)
C11—C10—H10B	109.4	O14^{viii}—Na3—O17	87.51 (17)
H10A—C10—H10B	108.0	O18—Na3—O1^{v}	89.09 (16)
O11—C11—O12	120.6 (6)	O16—Na3—O1^{v}	74.85 (15)
O11—C11—C10	122.2 (5)	O15^{vii}—Na3—O1^{v}	80.68 (17)

O12—C11—C10	117.1 (5)	O14^{viii}—Na3—O1^{v}	174.85 (16)
C4—N1—C2	108.8 (4)	O17—Na3—O1^{v}	88.40 (16)
O5—Mo1—Mo2—O8	−2.14 (18)	O3—C3—O4—Mo1	156.8 (4)
O7—Mo1—Mo2—O8	104.6 (2)	C4—C3—O4—Mo1	−25.7 (6)
O6—Mo1—Mo2—O8	−107.24 (19)	O5—Mo1—O4—C3	−164.8 (4)
O2—Mo1—Mo2—O8	−110.1 (2)	O7—Mo1—O4—C3	86.4 (4)
O4—Mo1—Mo2—O8	96.7 (2)	O6—Mo1—O4—C3	−17.6 (7)
N1—Mo1—Mo2—O8	173.66 (17)	O2—Mo1—O4—C3	−68.9 (4)
O5—Mo1—Mo2—O7	−106.8 (2)	N1—Mo1—O4—C3	6.4 (4)
O6—Mo1—Mo2—O7	148.2 (2)	Mo2—Mo1—O4—C3	92.4 (4)
O2—Mo1—Mo2—O7	145.3 (2)	O5—Mo1—O6—Mo2	−87.40 (18)
O4—Mo1—Mo2—O7	−7.9 (2)	O7—Mo1—O6—Mo2	23.38 (15)
N1—Mo1—Mo2—O7	69.05 (19)	O2—Mo1—O6—Mo2	178.01 (15)
O5—Mo1—Mo2—O6	105.10 (19)	O4—Mo1—O6—Mo2	127.3 (4)
O7—Mo1—Mo2—O6	−148.2 (2)	N1—Mo1—O6—Mo2	104.13 (14)
O2—Mo1—Mo2—O6	−2.8 (2)	O8—Mo2—O6—Mo1	85.40 (17)
O4—Mo1—Mo2—O6	−156.1 (2)	O7—Mo2—O6—Mo1	−23.27 (15)
N1—Mo1—Mo2—O6	−79.10 (17)	O12—Mo2—O6—Mo1	−106.5 (3)
O5—Mo1—Mo2—O12	−107.4 (2)	O9—Mo2—O6—Mo1	173.66 (13)
O7—Mo1—Mo2—O12	−0.7 (2)	N2—Mo2—O6—Mo1	−113.31 (14)
O6—Mo1—Mo2—O12	147.5 (2)	O5—Mo1—O7—Mo2	86.24 (17)
O2—Mo1—Mo2—O12	144.7 (2)	O6—Mo1—O7—Mo2	−23.44 (15)
O4—Mo1—Mo2—O12	−8.6 (2)	O2—Mo1—O7—Mo2	−110.2 (3)
N1—Mo1—Mo2—O12	68.40 (19)	O4—Mo1—O7—Mo2	174.95 (15)
O5—Mo1—Mo2—O9	94.9 (2)	N1—Mo1—O7—Mo2	−111.00 (15)
O7—Mo1—Mo2—O9	−158.3 (2)	O8—Mo2—O7—Mo1	−87.07 (18)
O6—Mo1—Mo2—O9	−10.2 (2)	O6—Mo2—O7—Mo1	23.40 (15)
O2—Mo1—Mo2—O9	−13.0 (2)	O12—Mo2—O7—Mo1	179.52 (16)
O4—Mo1—Mo2—O9	−166.2 (2)	O9—Mo2—O7—Mo1	130.6 (5)
N1—Mo1—Mo2—O9	−89.2 (2)	N2—Mo2—O7—Mo1	105.60 (15)
O5—Mo1—Mo2—N2	173.48 (16)	O10—C9—O9—Mo2	157.7 (4)
O7—Mo1—Mo2—N2	−79.77 (19)	C8—C9—O9—Mo2	−25.5 (7)
O6—Mo1—Mo2—N2	68.38 (17)	O8—Mo2—O9—C9	−164.3 (5)
O2—Mo1—Mo2—N2	65.57 (18)	O7—Mo2—O9—C9	−20.2 (8)
O4—Mo1—Mo2—N2	−87.7 (2)	O6—Mo2—O9—C9	86.9 (4)
N1—Mo1—Mo2—N2	−10.71 (14)	O12—Mo2—O9—C9	−69.3 (4)
O1—C1—C2—N1	158.7 (5)	N2—Mo2—O9—C9	5.9 (4)
O2—C1—C2—N1	−25.2 (7)	Mo1—Mo2—O9—C9	94.5 (4)
Na1^{i}—C1—C2—N1	101.9 (11)	O9—C9—O10—Na2	−175.9 (4)
O3—C3—C4—N1	−144.9 (5)	C8—C9—O10—Na2	7.4 (8)
O4—C3—C4—N1	37.4 (7)	O12—C11—O11—Na1	173.6 (4)
N1—C5—C6—N2	−96.4 (5)	C10—C11—O11—Na1	−9.9 (9)
N1—C5—C6—C7	137.6 (5)	O12—C11—O11—Na2^{ii}	−4.8 (5)
N2—C8—C9—O10	−146.2 (5)	C10—C11—O11—Na2^{ii}	171.7 (5)
N2—C8—C9—O9	37.0 (7)	O11—C11—O12—Mo2	−162.0 (4)
N2—C10—C11—O11	150.2 (5)	C10—C11—O12—Mo2	21.3 (6)
N2—C10—C11—O12	−33.2 (6)	O11—C11—O12—Na2^{ii}	5.6 (6)

N2—C10—C11—Na2^{ii}	−66.3 (17)	C10—C11—O12—Na2^{ii}	−171.1 (4)
C3—C4—N1—C2	85.8 (6)	O8—Mo2—O12—C11	158.2 (4)
C3—C4—N1—C5	−154.8 (5)	O7—Mo2—O12—C11	−95.3 (4)
C3—C4—N1—Mo1	−29.0 (5)	O6—Mo2—O12—C11	−10.6 (7)
C1—C2—N1—C4	−87.8 (5)	O9—Mo2—O12—C11	71.3 (4)
C1—C2—N1—C5	157.7 (4)	N2—Mo2—O12—C11	−3.5 (4)
C1—C2—N1—Mo1	26.0 (5)	Mo1—Mo2—O12—C11	−94.8 (4)
C6—C5—N1—C4	−178.8 (5)	O8—Mo2—O12—Na2^{ii}	−5.3 (3)
C6—C5—N1—C2	−61.5 (6)	O7—Mo2—O12—Na2^{ii}	101.3 (3)
C6—C5—N1—Mo1	64.2 (5)	O6—Mo2—O12—Na2^{ii}	−174.0 (2)
O5—Mo1—N1—C4	40.7 (7)	O9—Mo2—O12—Na2^{ii}	−92.1 (3)
O7—Mo1—N1—C4	−81.4 (3)	N2—Mo2—O12—Na2^{ii}	−167.0 (3)
O6—Mo1—N1—C4	−174.4 (3)	Mo1—Mo2—O12—Na2^{ii}	101.8 (3)
O2—Mo1—N1—C4	99.0 (3)	C11—O11—Na1—O16	62.4 (6)
O4—Mo1—N1—C4	13.2 (3)	Na2^{ii}—O11—Na1—O16	−120.0 (3)
Mo2—Mo1—N1—C4	−126.7 (3)	C11—O11—Na1—O17	142.2 (6)
O5—Mo1—N1—C2	−75.2 (6)	Na2^{ii}—O11—Na1—O17	−40.2 (3)
O7—Mo1—N1—C2	162.7 (3)	C11—O11—Na1—O1^{v}	50.1 (11)
O6—Mo1—N1—C2	69.7 (3)	Na2^{ii}—O11—Na1—O1^{v}	−132.3 (7)
O2—Mo1—N1—C2	−16.9 (3)	C11—O11—Na1—O15	−120.3 (6)
O4—Mo1—N1—C2	−102.7 (3)	Na2^{ii}—O11—Na1—O15	57.3 (3)
Mo2—Mo1—N1—C2	117.4 (3)	C11—O11—Na1—Cl1	−44.0 (6)
O5—Mo1—N1—C5	157.0 (5)	Na2^{ii}—O11—Na1—Cl1	133.7 (2)
O7—Mo1—N1—C5	34.9 (3)	Na3—O16—Na1—O11	130.10 (17)
O6—Mo1—N1—C5	−58.2 (3)	Na3—O16—Na1—O17	43.64 (15)
O2—Mo1—N1—C5	−144.8 (4)	Na3—O16—Na1—O1^{v}	−52.90 (15)
O4—Mo1—N1—C5	129.5 (4)	Na3—O16—Na1—Cl1	−137.19 (12)
Mo2—Mo1—N1—C5	−10.4 (3)	Na3—O16—Na1—C1^{v}	−41.02 (16)
C11—C10—N2—C8	−86.0 (5)	Na3—O17—Na1—O11	−131.05 (16)
C11—C10—N2—C6	156.9 (4)	Na3—O17—Na1—O16	−41.81 (14)
C11—C10—N2—Mo2	26.9 (5)	Na3—O17—Na1—O1^{v}	35.00 (15)
C9—C8—N2—C10	84.6 (6)	Na3—O17—Na1—O15	140.62 (15)
C9—C8—N2—C6	−154.3 (5)	Na3—O17—Na1—C1^{v}	55.82 (15)
C9—C8—N2—Mo2	−28.6 (5)	Na3^{iv}—O15—Na1—O11	−25.5 (3)
C7—C6—N2—C10	56.9 (6)	Na3^{iv}—O15—Na1—O17	60.6 (3)
C5—C6—N2—C10	−66.4 (5)	Na3^{iv}—O15—Na1—O1^{v}	156.8 (2)
C7—C6—N2—C8	−61.3 (5)	Na3^{iv}—O15—Na1—Cl1	−118.9 (3)
C5—C6—N2—C8	175.4 (4)	Na3^{iv}—O15—Na1—C1^{v}	145.9 (3)
C7—C6—N2—Mo2	−179.3 (3)	Na3^{iv}—O15—Na1—Na3	101.2 (3)
C5—C6—N2—Mo2	57.4 (5)	Na2—Cl1—Na1—O11	−51.88 (19)
O8—Mo2—N2—C10	−73.6 (6)	Na2—Cl1—Na1—O16	−141.29 (16)
O7—Mo2—N2—C10	71.0 (3)	Na2—Cl1—Na1—O1^{v}	142.02 (16)
O6—Mo2—N2—C10	163.3 (3)	Na2—Cl1—Na1—O15	36.19 (17)
O12—Mo2—N2—C10	−13.9 (3)	Na2—Cl1—Na1—C1^{v}	120.46 (16)

O9—Mo2—N2—C10	−101.3 (3)	Na2—Cl1—Na1—Na3	173.35 (12)
Mo1—Mo2—N2—C10	118.4 (3)	Na3^{iii}—O14—Na2—O12^{vi}	−22.2 (3)
O8—Mo2—N2—C8	40.9 (6)	Na3^{iii}—O14—Na2—O10	134.23 (19)
O7—Mo2—N2—C8	−174.5 (3)	Na3^{iii}—O14—Na2—O13	58.0 (2)
O6—Mo2—N2—C8	−82.2 (3)	Na3^{iii}—O14—Na2—O11^{vi}	−52.4 (2)
O12—Mo2—N2—C8	100.7 (4)	Na3^{iii}—O14—Na2—Cl1	−132.34 (17)
O9—Mo2—N2—C8	13.2 (3)	C9—O10—Na2—O14	145.3 (5)
Mo1—Mo2—N2—C8	−127.1 (3)	C9—O10—Na2—O12^{vi}	−50.5 (5)
O8—Mo2—N2—C6	159.2 (4)	C9—O10—Na2—O13	−132.0 (5)
O7—Mo2—N2—C6	−56.2 (3)	C9—O10—Na2—O11^{vi}	−22.9 (7)
O6—Mo2—N2—C6	36.1 (3)	C9—O10—Na2—Cl1	52.7 (5)
O12—Mo2—N2—C6	−141.0 (4)	Na1—Cl1—Na2—O14	−71.71 (18)
O9—Mo2—N2—C6	131.5 (4)	Na1—Cl1—Na2—O12^{vi}	148.50 (15)
Mo1—Mo2—N2—C6	−8.8 (3)	Na1—Cl1—Na2—O10	46.98 (18)
O2—C1—O1—Na1^{i}	25.9 (6)	Na1—Cl1—Na2—O13	20.0 (9)
C2—C1—O1—Na1^{i}	−158.2 (4)	Na1—Cl1—Na2—O11^{vi}	−166.93 (14)
O2—C1—O1—Na3^{i}	−53.3 (6)	Na1—O16—Na3—O18	26.0 (7)
C2—C1—O1—Na3^{i}	122.5 (4)	Na1—O16—Na3—O15^{vii}	127.04 (17)
Na1^{i}—C1—O1—Na3^{i}	−79.2 (2)	Na1—O16—Na3—O14^{viii}	−127.65 (17)
O1—C1—O2—Mo1	−174.4 (4)	Na1—O16—Na3—O17	−42.42 (15)
C2—C1—O2—Mo1	9.6 (6)	Na1—O16—Na3—O1^{v}	48.85 (14)
Na1^{i}—C1—O2—Mo1	−154.0 (3)	Na1—O16—Na3—Na2^{viii}	−142.31 (16)
O5—Mo1—O2—C1	168.3 (4)	Na1—O17—Na3—O18	−121.22 (16)
O7—Mo1—O2—C1	3.8 (6)	Na1—O17—Na3—O16	42.64 (15)
O6—Mo1—O2—C1	−84.4 (4)	Na1—O17—Na3—O15^{vii}	−30.4 (10)
O4—Mo1—O2—C1	80.9 (4)	Na1—O17—Na3—O14^{viii}	144.63 (16)
N1—Mo1—O2—C1	4.7 (4)	Na1—O17—Na3—O1^{v}	−32.25 (14)
Mo2—Mo1—O2—C1	−82.2 (4)	Na1—O17—Na3—Na2^{viii}	111.14 (11)

Symmetry codes: (i) *x*−1/2, −*y*+1/2, *z*−1/2; (ii) *x*−1/2, *y*+1/2, *z*; (iii) *x*, *y*−1, *z*; (iv) *x*−1/2, *y*−1/2, *z*; (v) *x*+1/2, −*y*+1/2, *z*+1/2; (vi) *x*+1/2, *y*−1/2, *z*; (vii) *x*+1/2, *y*+1/2, *z*; (viii) *x*, *y*+1, *z*.

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O13—H13A···O8^{vi}	0.90 (2)	2.09 (4)	2.929 (6)	154 (7)
O13—H13B···Cl1^{iv}	0.89 (5)	2.27 (6)	3.152 (5)	169 (7)
O14—H14B···O3^{v}	0.88 (5)	1.97 (5)	2.850 (6)	173 (6)
O14—H14A···O1^{ix}	0.88 (2)	2.13 (5)	2.850 (7)	139 (6)
O15—H15A···Cl1	0.89 (6)	2.52 (4)	3.281 (5)	144 (6)
O15—H15B···O4^{v}	0.87 (5)	2.24 (3)	3.081 (6)	163 (6)
O16—H16B···O10^{vii}	0.89 (6)	2.00 (3)	2.876 (6)	168 (7)
O16—H16A···O13^{viii}	0.89 (6)	2.02 (5)	2.778 (6)	143 (7)
O17—H17B···O3^{x}	0.88 (5)	2.22 (5)	2.953 (7)	142 (6)
O17—H17A···Cl1^{ii}	0.89 (5)	2.43 (4)	3.239 (5)	152 (7)

O18—H18A···O6^{v}	0.90 (5)	2.08 (4)	2.914 (6)	156 (7)
O18—H18B···O5^{xi}	0.90 (6)	1.93 (6)	2.827 (6)	170 (7)

Symmetry codes: (vi) *x*+1/2, *y*−1/2, *z*; (iv) *x*−1/2, *y*−1/2, *z*; (v) *x*+1/2, −*y*+1/2, *z*+1/2; (ix) *x*, −*y*, *z*+1/2; (vii) *x*+1/2, *y*+1/2, *z*; (viii) *x*, *y*+1, *z*; (x) *x*, −*y*+1, *z*+1/2; (ii) *x*−1/2, *y*+1/2, *z*; (xi) *x*+1, −*y*+1, *z*+1/2.

Fig. 1

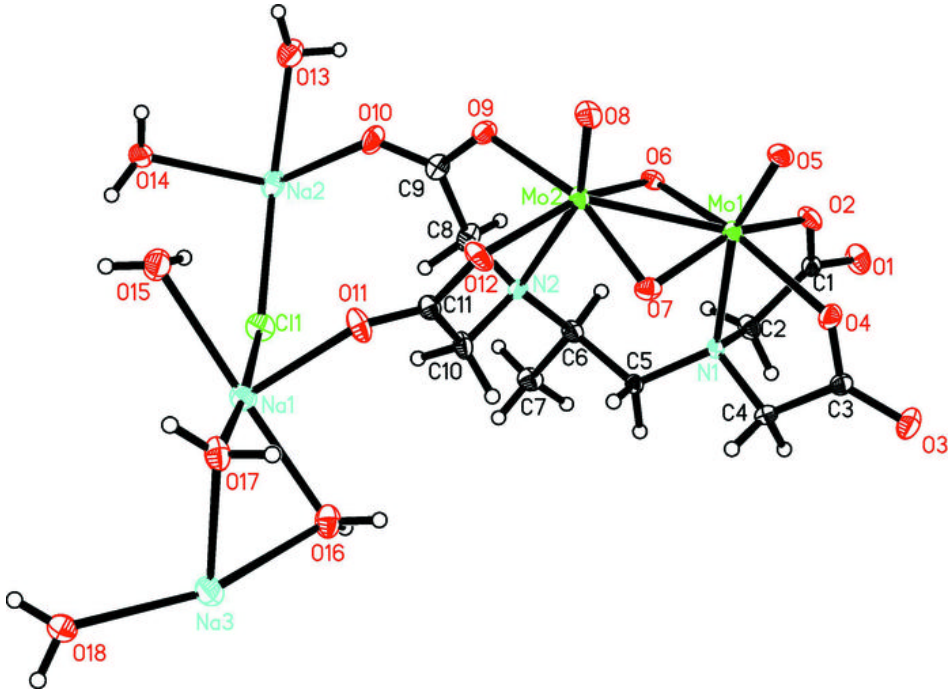
O13
O10
O9
O8
O6
O5
O14
Na2
C9
Mo2
Mo1
O2
C8
O1
O15
O12
N2
C1
O11
C11
O7
C2
O4
Cl1
C6
C5
C10
N1
Na1
C7
C4
C3
O3
O17
O16
Na3
O18

Fig. 2