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Bis(2,4-dioxopentan-3-ido-κ^2O,O')dioxidomolybdenum(VI): a redetermination

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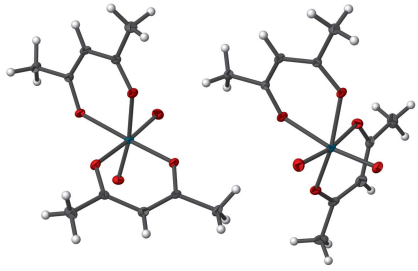
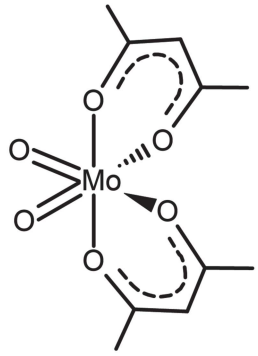
Structural data: full structural data are available from iucrdata.iucr.org

The title compound, $[Mo(C_5H_7O_2)_2O_2]$ or *cis*-$[MoO_2(acac)_2]$ (acac is acetylacetonate), contains a molybdenum(VI) atom coordinated by two acetylacetonate ligands and two doubly bonded oxido ligands in a distorted octahedral shape. The molecule is chiral and the asymmetric unit contains two independent molecules (one Δ, one Λ). Extensive C—H···O contacts are present throughout the structure. Data were collected at 100 K, providing higher precision of unit-cell parameters and atomic positions than previous determinations [Kamenar *et al.* (1973). *Cryst. Struct. Commun.* **2**, 41–44.; Krasochka *et al.* (1975). *Zh. Strukt. Khim.* **16**, 696–698].

3D view



Chemical scheme



Structure description

The title compound is a versatile starting material for the preparation of *cis*-dioxidomolybdenum complexes, including complexes containing organodinitrogen ligands (Bustos *et al.*, 1994) and molybdenyl adducts of platinum μ-S dimers (Henderson *et al.*, 2011). $MoO_2(acac)_2$ has also been used to prepare dioxidomolybdenum(VI) complexes with *O*,*N*,*N*′ chelating ligands (Ceylan *et al.*, 2015) and an amine bis(phenolate) ligand (Bowen & Wile, 2021). Many of these complexes have been prepared and studied for their catalytic activities, including complexes with acylpyrazolonate ligands that catalyze the deoxygenation of epoxides (Hills *et al.*, 2013; Begines *et al.*, 2018) and dioxidomolybdenum(VI) complexes with salicylamide ligands for the epoxidation of olefins (Annese *et al.*, 2019). Molybdenum(VI) dioxido complexes with acetylacetonato ligands have also been investigated for their catalytic properties in the dehydrogenation of alcohols (Korstanje *et al.*, 2013). These complexes are of particular interest due to their close structural similarities to the active sites of several molybdoenyzmes such as sulfite oxidase, xanthine oxidase, and DMSO reductase (Sousa & Fernandes, 2015).

Table 1
Selected geometric parameters (Å, °).

Mo1—O1	1.7029 (9)	Mo2—O7	1.6996 (9)
Mo1—O2	1.7001 (9)	Mo2—O8	1.7021 (9)
Mo1—O3	2.1825 (8)	Mo2—O9	2.1808 (8)
Mo1—O4	2.1921 (8)	Mo2—O10	2.1848 (9)
Mo1—O5	2.0060 (8)	Mo2—O11	1.9898 (8)
Mo1—O6	1.9897 (8)	Mo2—O12	2.0106 (8)
O2—Mo1—O1	105.40 (4)	O7—Mo2—O8	105.59 (5)

Two previous structural determinations of *cis*-dioxidobis(acetylacetonato)molybdenum(VI) were published in the mid-1970s (Kamenar *et al.*, 1973; Krasochka *et al.*, 1975) based on photographic methods and room-temperature data collections. Additionally, Craven *et al.* (1971) cite an unpublished diffraction study that also confirms the *cis* coordination and includes additional structural information consistent with the current study. None of the previously published structure solutions attempted to locate the positions of any of the hydrogen atoms. Several closely related structures have been determined, including *cis*-dioxido-molybdenum complexes with 1,3-diphenylpropanedianoto ligands (Kojić-Prodić *et al.*, 1974; Korstanje *et al.*, 2013) and *tert*-butylacetylacetonato ligands (Nass *et al.*, 2001). The structure of the product from the reaction of *cis*-$[MoO_2(acac)_2]$ with the strong Lewis acid $B(C_6F_5)_3$ (Galsworthy *et al.*, 1997) displays a nearly linear Mo═O—B arrangement [171.2 (1)°] and lengthening of the donating Mo═O bond by about 0.1 Å.

The asymmetric unit of the title compound contains two crystallographically independent *cis*-$[MoO_2(acac)_2]$ molecules, one each of the Δ and Λ forms (Fig. 1). The molecular structure adopts a distorted octahedral arrangement around the Mo^{VI} atoms, with oxido ligands in a *cis* arrangement and oxido-molybdenum-oxido angles of 105.40 (4) and 105.59 (5)°. As observed previously (Krasochka, 1973; Kojić-Prodić *et al.*, 1974), the Mo—O bond distances *trans* to the molybdenum-oxygen double bonds are significantly lengthened [avg = 2.185 (5) Å] relative to the other molybdenum–oxygen distances [avg = 1.999 (11) Å] (see Table 1 for selected bond distances and angles). The four molybdenum oxygen distances for the doubly-bonded oxido ligands average 1.7012 (16) Å, in agreement with the average distance found for over 140 similar *cis*-dioxido molybdenum complexes in the Cambridge Structural Database (Groom *et al.*, 2016). These metrics are also in agreement with relatively narrow distribution of molybdenum–oxygen distances observed by Mayer (1988) for *cis*-dioxido complexes.

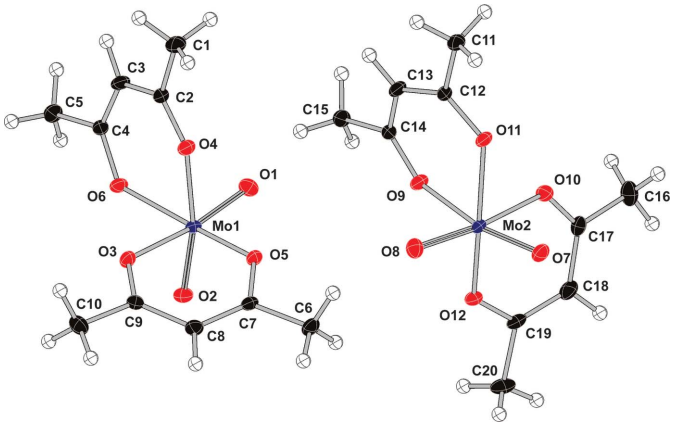


Figure 1
Displacement ellipsoid (50% probability) diagram of the two independent molecules with the numbering scheme for the non-hydrogen atoms.

Table 2
Hydrogen-bond geometry (Å, °).

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C1—H1*A*···O8^{i}	0.93 (1)	2.62 (2)	3.4834 (16)	155 (2)
C5—H5*B*···O10^{ii}	0.98 (1)	2.50 (1)	3.4652 (15)	170 (2)
C6—H6*A*···O8	0.97 (1)	2.51 (2)	3.3894 (15)	151 (2)
C8—H8···O7^{iii}	0.91 (1)	2.79 (2)	3.4071 (14)	126 (1)
C10—H10*A*···O6^{iv}	0.95 (1)	2.68 (2)	3.3334 (15)	127 (1)
C10—H10*C*···O1^{v}	0.97 (1)	2.50 (2)	3.3126 (15)	141 (2)
C11—H11*B*···O3^{i}	0.99 (1)	2.48 (1)	3.4415 (14)	163 (2)
C15—H15*A*···O1^{ii}	0.93 (1)	2.55 (2)	3.4592 (15)	167 (2)
C15—H15*C*···O4	0.96 (1)	2.53 (2)	3.4018 (15)	152 (2)
C16—H16*A*···O11^{vi}	0.94 (1)	2.66 (2)	3.3127 (15)	128 (2)
C16—H16*B*···O8^{vii}	0.96 (2)	2.52 (2)	3.3971 (17)	153 (2)
C18—H18···O2^{viii}	0.92 (1)	2.82 (2)	3.4646 (15)	128 (1)

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

All of the hydrogen-bonding contacts are weak C—H···O interactions with *D*···*A* distances between 3.3 and 3.5 Å (see Table 2 and Fig. 2). There are contacts between C—H atoms and all four of the oxido ligands, including two contacts to O1 and three contacts to O8. Additional C—H contacts are made to most of the acetylacetonate oxygen atoms as well.

Synthesis and crystallization

The title compound was prepared using the *Inorganic Syntheses* procedure (Chakravorti & Bandyopadhyay, 1992) with some modifications adapted from Arnáiz (1995). A sample of 3.0 grams of ammonium *para*-molybdate was dissolved in 6.0 ml of 24%$_{wt}$ aqueous ammonia. A syringe was used to add 7.0 ml of 2,4-pentanedione with stirring. Concentrated nitric acid (5.0 ml) was added and the solution was stirred for

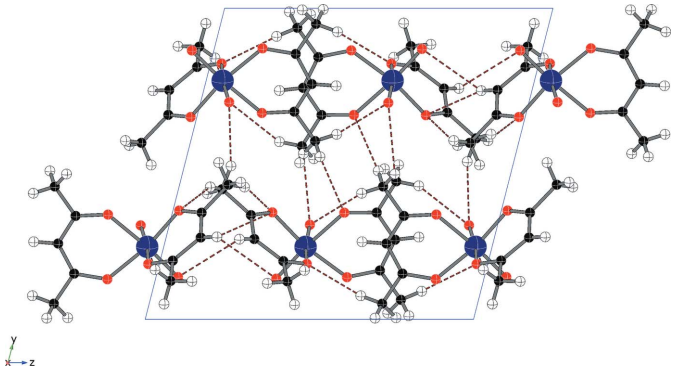


Figure 2
Packing diagram (viewed along *a*), showing extensive weak C—H···O contacts (red dotted lines) throughout the crystal structure.

Table 3
Experimental details.

Crystal data	
Chemical formula	$[Mo(C_5H_7O_2)_2O_2]$
M_r	326.15
Crystal system, space group	Triclinic, $P\overline{1}$
Temperature (K)	100
a, b, c (Å)	8.0111 (3), 12.4143 (4), 12.6847 (4)
α, β, γ (°)	75.649 (1), 89.272 (1), 87.072 (1)
V (Å^3)	1220.56 (7)
Z	4
Radiation type	Mo $K\alpha$
μ (mm^{-1})	1.09
Crystal size (mm)	0.28 × 0.22 × 0.14
Data collection	
Diffractometer	Bruker APEXII CCD
Absorption correction	Multi-scan (*SADABS*; Krause *et al.*, 2015)
T_{min}, T_{max}	0.676, 0.747
No. of measured, independent and observed [$I > 2\sigma(I)$] reflections	82074, 11855, 10556
R_{int}	0.035
(sin $\theta/\lambda)_{max}$ (Å^{-1})	0.835
Refinement	
$R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, S	0.023, 0.061, 1.04
No. of reflections	11855
No. of parameters	391
No. of restraints	28
H-atom treatment	Only H-atom coordinates refined
$\Delta\rho_{max}$, $\Delta\rho_{min}$ (e Å^{-3})	1.19, −1.11

Computer programs: *APEX3* (Bruker, 2020), *SAINT* (Bruker, 2020), *SHELXT* (Sheldrick, 2015*a*), *SHELXL* (Sheldrick, 2015*b*), *CrystalMaker* (Palmer, 2019), *OLEX2* (Dolomanov *et al*., 2009) and *publCIF* (Westrip, 2010).

30 min. The product precipitated as a pale-yellow solid and was isolated by filtration and washed with deionized water (2 × 10 ml), followed by ethanol (1 × 10 ml), and diethyl ether (1 × 10 ml). Over multiple preparations the yield averaged around 90%. Characterization by ^{1}H NMR and FTIR agrees with previously reported values (Chakravorti & Bandyopadhyay, 1992; Arnáiz, 1995).

Three different crystallization methods were utilized: slow evaporation from a concentrated solution in 2,4-pentanedione, vapor diffusion (dichloromethane/diethyl ether), and layering (dichloromethane/diethyl ether) in a standard 5 mm NMR tube. All three methods produced crystals, but the highest quality crystals and those used in this study were produced from solvent layering.

Refinement

Crystal data, data collection and structure refinement details are summarized in Table 3.

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full crystallographic data

IUCrData (2021). **6**, x210778 [https://doi.org/10.1107/S2414314621007781]

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Crystal data

$[Mo(C_5H_7O_2)_2O_2]$
M_r = 326.15
Triclinic, $P\overline{1}$
a = 8.0111 (3) Å
b = 12.4143 (4) Å
c = 12.6847 (4) Å
α = 75.649 (1)°
β = 89.272 (1)°
γ = 87.072 (1)°
V = 1220.56 (7) Å^3
Z = 4
$F(000)$ = 656
D_x = 1.775 Mg m^{-3}
Mo $K\alpha$ radiation, λ = 0.71073 Å
Cell parameters from 9760 reflections
θ = 3.0–36.3°
μ = 1.09 mm^{-1}
T = 100 K
Block, yellow
0.28 × 0.22 × 0.14 mm

Data collection

Bruker APEXII CCD diffractometer
φ and ω scans
Absorption correction: multi-scan (SADABS; Krause *et al.*, 2015)
T_{min} = 0.676, T_{max} = 0.747
82074 measured reflections
11855 independent reflections
10556 reflections with $I > 2\sigma(I)$
R_{int} = 0.035
θ_{max} = 36.4°, θ_{min} = 1.7°
h = −13→13
k = −20→20
l = −21→21

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.023
$wR(F^2)$ = 0.061
S = 1.04
11855 reflections
391 parameters
28 restraints
0 constraints
Only H-atom coordinates refined
$w = 1/[\sigma^2(F_o^2) + (0.0307P)^2 + 0.524P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.003
$\Delta\rho_{max}$ = 1.19 e Å^{-3}
$\Delta\rho_{min}$ = −1.11 e Å^{-3}

Special details

Refinement. All H atoms were located in a difference-Fourier map. Hydrogen atom positions were refined with C—H distances restrained to 0.98 (2) Å (CH_3) or 0.95 (2) Å (ring C) and with U_{iso}(H) = 1.5U_{eq}(C) (methyl) or U_{iso}(H) = 1.2U_{eq}(C) (ring).

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

	x	y	z	U_{iso}*/U_{eq}
Mo1	0.19729 (2)	0.76693 (2)	0.56793 (2)	0.01071 (2)

O1	0.01992 (11)	0.69604 (7)	0.57195 (8)	0.01646 (15)
O2	0.14825 (12)	0.86914 (7)	0.63247 (7)	0.01718 (15)
O3	0.45296 (11)	0.82176 (7)	0.55088 (7)	0.01484 (14)
O4	0.32120 (11)	0.65709 (7)	0.47653 (7)	0.01407 (14)
O5	0.30012 (10)	0.65609 (7)	0.69609 (7)	0.01323 (13)
O6	0.17058 (11)	0.86448 (7)	0.41831 (7)	0.01522 (14)
C1	0.37036 (18)	0.55651 (10)	0.34261 (11)	0.0194 (2)
H1A	0.408 (2)	0.5708 (16)	0.2707 (13)	0.029*
H1B	0.457 (2)	0.5162 (15)	0.3884 (16)	0.029*
H1C	0.286 (2)	0.5032 (14)	0.3486 (16)	0.029*
C2	0.30151 (14)	0.65672 (9)	0.37795 (9)	0.01315 (17)
C3	0.21648 (16)	0.74411 (10)	0.30091 (9)	0.01650 (19)
H3	0.205 (2)	0.7355 (14)	0.2306 (12)	0.020*
C4	0.16005 (14)	0.84288 (9)	0.32317 (9)	0.01329 (17)
C5	0.08566 (17)	0.93714 (10)	0.23647 (10)	0.0183 (2)
H5A	0.170 (2)	0.9885 (14)	0.2000 (15)	0.028*
H5B	0.033 (2)	0.9095 (15)	0.1802 (14)	0.028*
H5C	0.002 (2)	0.9801 (15)	0.2663 (16)	0.028*
C6	0.42700 (16)	0.58188 (10)	0.86696 (9)	0.0170 (2)
H6A	0.416 (2)	0.5069 (12)	0.8582 (16)	0.026*
H6B	0.333 (2)	0.5953 (15)	0.9090 (15)	0.026*
H6C	0.5274 (19)	0.5743 (15)	0.9049 (15)	0.026*
C7	0.42379 (14)	0.66499 (9)	0.75910 (8)	0.01228 (16)
C8	0.54477 (15)	0.74177 (10)	0.73039 (9)	0.01541 (18)
H8	0.6317 (19)	0.7423 (14)	0.7754 (13)	0.018*
C9	0.56069 (13)	0.81282 (9)	0.62473 (9)	0.01234 (17)
C10	0.71393 (15)	0.87800 (10)	0.59652 (11)	0.0180 (2)
H10A	0.689 (2)	0.9523 (12)	0.5569 (15)	0.027*
H10B	0.776 (2)	0.8840 (15)	0.6561 (13)	0.027*
H10C	0.788 (2)	0.8426 (15)	0.5532 (15)	0.027*
Mo2	0.31234 (2)	0.23314 (2)	0.94971 (2)	0.01062 (2)
O7	0.35933 (12)	0.13590 (7)	1.06697 (7)	0.01702 (15)
O8	0.49058 (11)	0.30206 (7)	0.91316 (8)	0.01682 (15)
O9	0.18784 (11)	0.33673 (7)	0.80520 (7)	0.01435 (14)
O10	0.05669 (11)	0.17773 (7)	0.96628 (7)	0.01557 (15)
O11	0.33915 (11)	0.12896 (7)	0.85341 (7)	0.01430 (14)
O12	0.20855 (11)	0.34966 (7)	1.01892 (7)	0.01405 (14)
C11	0.42605 (16)	0.05038 (9)	0.70948 (10)	0.01590 (19)
H11A	0.506 (2)	0.0052 (14)	0.7626 (15)	0.024*
H11B	0.481 (2)	0.0759 (15)	0.6381 (12)	0.024*
H11C	0.338 (2)	0.0040 (14)	0.6990 (15)	0.024*
C12	0.35213 (13)	0.14698 (9)	0.74799 (9)	0.01179 (16)
C13	0.29618 (16)	0.24372 (9)	0.67571 (9)	0.01583 (19)
H13	0.312 (2)	0.2494 (14)	0.6000 (11)	0.019*
C14	0.20743 (13)	0.33250 (9)	0.70739 (9)	0.01210 (16)
C15	0.13314 (16)	0.42696 (10)	0.62128 (10)	0.01670 (19)
H15A	0.095 (2)	0.4053 (15)	0.5610 (13)	0.025*
H15B	0.043 (2)	0.4672 (15)	0.6472 (16)	0.025*

H15C	0.219 (2)	0.4773 (14)	0.5942 (15)	0.025*
C16	−0.20236 (16)	0.12360 (11)	1.04748 (12)	0.0219 (2)
H16A	−0.178 (2)	0.0492 (13)	1.0454 (17)	0.033*
H16B	−0.280 (2)	0.1597 (16)	0.9911 (15)	0.033*
H16C	−0.265 (2)	0.1222 (16)	1.1124 (14)	0.033*
C17	−0.04876 (14)	0.18876 (9)	1.03822 (9)	0.01463 (18)
C18	−0.03130 (16)	0.26312 (11)	1.10536 (10)	0.0182 (2)
H18	−0.116 (2)	0.2651 (15)	1.1543 (14)	0.022*
C19	0.08525 (14)	0.34274 (9)	1.08878 (9)	0.01445 (18)
C20	0.07649 (18)	0.43275 (12)	1.14881 (11)	0.0220 (2)
H20A	0.087 (3)	0.5039 (13)	1.1019 (16)	0.033*
H20B	−0.027 (2)	0.4408 (16)	1.1843 (16)	0.033*
H20C	0.163 (2)	0.4253 (17)	1.1994 (15)	0.033*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Mo1	0.01212 (4)	0.01239 (4)	0.00833 (4)	−0.00019 (3)	−0.00151 (3)	−0.00391 (3)
O1	0.0145 (3)	0.0165 (4)	0.0190 (4)	−0.0011 (3)	−0.0029 (3)	−0.0055 (3)
O2	0.0220 (4)	0.0167 (4)	0.0150 (4)	−0.0011 (3)	0.0008 (3)	−0.0078 (3)
O3	0.0153 (3)	0.0185 (4)	0.0102 (3)	−0.0043 (3)	−0.0011 (3)	−0.0016 (3)
O4	0.0174 (4)	0.0143 (3)	0.0106 (3)	0.0010 (3)	−0.0004 (3)	−0.0037 (3)
O5	0.0147 (3)	0.0152 (3)	0.0094 (3)	−0.0023 (3)	−0.0017 (3)	−0.0018 (3)
O6	0.0229 (4)	0.0129 (3)	0.0099 (3)	0.0008 (3)	−0.0041 (3)	−0.0031 (3)
C1	0.0265 (6)	0.0172 (5)	0.0161 (5)	0.0006 (4)	0.0036 (4)	−0.0079 (4)
C2	0.0152 (4)	0.0140 (4)	0.0111 (4)	−0.0026 (3)	0.0016 (3)	−0.0043 (3)
C3	0.0244 (5)	0.0163 (5)	0.0096 (4)	−0.0001 (4)	−0.0026 (4)	−0.0048 (3)
C4	0.0157 (4)	0.0138 (4)	0.0102 (4)	−0.0028 (3)	−0.0027 (3)	−0.0020 (3)
C5	0.0240 (5)	0.0165 (5)	0.0127 (4)	−0.0004 (4)	−0.0066 (4)	0.0000 (4)
C6	0.0228 (5)	0.0171 (5)	0.0096 (4)	0.0007 (4)	−0.0027 (4)	−0.0004 (3)
C7	0.0156 (4)	0.0126 (4)	0.0090 (4)	0.0017 (3)	−0.0015 (3)	−0.0037 (3)
C8	0.0176 (5)	0.0159 (4)	0.0126 (4)	−0.0016 (4)	−0.0050 (4)	−0.0029 (3)
C9	0.0133 (4)	0.0114 (4)	0.0134 (4)	0.0000 (3)	−0.0004 (3)	−0.0051 (3)
C10	0.0146 (5)	0.0172 (5)	0.0234 (6)	−0.0040 (4)	0.0003 (4)	−0.0070 (4)
Mo2	0.01235 (4)	0.01240 (4)	0.00711 (4)	0.00003 (3)	0.00073 (3)	−0.00262 (3)
O7	0.0226 (4)	0.0168 (4)	0.0105 (3)	−0.0003 (3)	−0.0010 (3)	−0.0013 (3)
O8	0.0153 (4)	0.0165 (4)	0.0181 (4)	−0.0016 (3)	0.0027 (3)	−0.0033 (3)
O9	0.0188 (4)	0.0146 (3)	0.0095 (3)	0.0024 (3)	−0.0003 (3)	−0.0034 (3)
O10	0.0151 (3)	0.0185 (4)	0.0147 (4)	−0.0040 (3)	0.0028 (3)	−0.0066 (3)
O11	0.0214 (4)	0.0131 (3)	0.0083 (3)	0.0013 (3)	0.0016 (3)	−0.0029 (2)
O12	0.0161 (3)	0.0159 (3)	0.0115 (3)	−0.0007 (3)	0.0016 (3)	−0.0062 (3)
C11	0.0213 (5)	0.0137 (4)	0.0135 (4)	0.0010 (4)	0.0031 (4)	−0.0052 (3)
C12	0.0132 (4)	0.0124 (4)	0.0102 (4)	−0.0018 (3)	0.0025 (3)	−0.0037 (3)
C13	0.0244 (5)	0.0145 (4)	0.0085 (4)	0.0012 (4)	0.0011 (4)	−0.0031 (3)
C14	0.0146 (4)	0.0116 (4)	0.0100 (4)	−0.0021 (3)	−0.0006 (3)	−0.0020 (3)
C15	0.0233 (5)	0.0129 (4)	0.0127 (4)	−0.0002 (4)	−0.0046 (4)	−0.0009 (3)
C16	0.0159 (5)	0.0220 (5)	0.0248 (6)	−0.0044 (4)	0.0025 (4)	0.0002 (4)
C17	0.0139 (4)	0.0151 (4)	0.0127 (4)	0.0001 (3)	0.0004 (3)	0.0007 (3)

C18	0.0185 (5)	0.0222 (5)	0.0141 (5)	0.0002 (4)	0.0065 (4)	−0.0054 (4)
C19	0.0173 (4)	0.0173 (4)	0.0091 (4)	0.0039 (4)	−0.0006 (3)	−0.0050 (3)
C20	0.0263 (6)	0.0251 (6)	0.0181 (5)	0.0048 (5)	−0.0002 (4)	−0.0135 (5)

Geometric parameters (Å, º)

Mo1—O1	1.7029 (9)	Mo2—O7	1.6996 (9)
Mo1—O2	1.7001 (9)	Mo2—O8	1.7021 (9)
Mo1—O3	2.1825 (8)	Mo2—O9	2.1808 (8)
Mo1—O4	2.1921 (8)	Mo2—O10	2.1848 (9)
Mo1—O5	2.0060 (8)	Mo2—O11	1.9898 (8)
Mo1—O6	1.9897 (8)	Mo2—O12	2.0106 (8)
O3—C9	1.2624 (13)	O9—C14	1.2623 (13)
O4—C2	1.2635 (13)	O10—C17	1.2632 (14)
O5—C7	1.3067 (13)	O11—C12	1.3036 (13)
O6—C4	1.3041 (13)	O12—C19	1.3104 (14)
C1—H1A	0.933 (14)	C11—H11A	0.984 (14)
C1—H1B	0.947 (15)	C11—H11B	0.988 (14)
C1—H1C	0.957 (14)	C11—H11C	0.962 (14)
C1—C2	1.5010 (16)	C11—C12	1.4961 (15)
C2—C3	1.4183 (16)	C12—C13	1.3761 (16)
C3—H3	0.931 (14)	C13—H13	0.953 (14)
C3—C4	1.3782 (16)	C13—C14	1.4191 (16)
C4—C5	1.4971 (16)	C14—C15	1.4942 (16)
C5—H5A	0.983 (14)	C15—H15A	0.932 (14)
C5—H5B	0.975 (14)	C15—H15B	0.961 (14)
C5—H5C	0.968 (15)	C15—H15C	0.957 (14)
C6—H6A	0.973 (14)	C16—H16A	0.941 (14)
C6—H6B	0.946 (14)	C16—H16B	0.961 (15)
C6—H6C	0.931 (14)	C16—H16C	0.955 (15)
C6—C7	1.4952 (16)	C16—C17	1.4944 (17)
C7—C8	1.3762 (16)	C17—C18	1.4152 (17)
C8—H8	0.907 (14)	C18—H18	0.917 (14)
C8—C9	1.4184 (16)	C18—C19	1.3709 (17)
C9—C10	1.4955 (16)	C19—C20	1.4986 (17)
C10—H10A	0.948 (14)	C20—H20A	0.942 (15)
C10—H10B	0.931 (14)	C20—H20B	0.950 (15)
C10—H10C	0.965 (14)	C20—H20C	0.935 (15)
O1—Mo1—O3	165.66 (4)	O7—Mo2—O8	105.59 (5)
O1—Mo1—O4	89.35 (4)	O7—Mo2—O9	164.58 (4)
O1—Mo1—O5	93.60 (4)	O7—Mo2—O10	87.98 (4)
O1—Mo1—O6	98.03 (4)	O7—Mo2—O11	95.53 (4)
O2—Mo1—O1	105.40 (4)	O7—Mo2—O12	96.98 (4)
O2—Mo1—O3	88.56 (4)	O8—Mo2—O9	89.81 (4)
O2—Mo1—O4	165.18 (4)	O8—Mo2—O10	166.15 (4)
O2—Mo1—O5	97.16 (4)	O8—Mo2—O11	97.67 (4)
O2—Mo1—O6	95.35 (4)	O8—Mo2—O12	94.14 (4)

O3—Mo1—O4	76.80 (3)	O9—Mo2—O10	76.68 (3)
O5—Mo1—O3	81.17 (3)	O11—Mo2—O9	81.38 (3)
O5—Mo1—O4	82.99 (3)	O11—Mo2—O10	83.47 (3)
O6—Mo1—O3	83.63 (3)	O11—Mo2—O12	159.82 (4)
O6—Mo1—O4	80.95 (3)	O12—Mo2—O9	82.36 (3)
O6—Mo1—O5	160.01 (4)	O12—Mo2—O10	81.22 (3)
C9—O3—Mo1	127.94 (7)	C14—O9—Mo2	128.23 (7)
C2—O4—Mo1	128.41 (7)	C17—O10—Mo2	127.20 (8)
C7—O5—Mo1	130.40 (7)	C12—O11—Mo2	131.47 (7)
C4—O6—Mo1	132.43 (7)	C19—O12—Mo2	129.74 (7)
H1A—C1—H1B	108.5 (17)	H11A—C11—H11B	109.9 (16)
H1A—C1—H1C	105.8 (17)	H11A—C11—H11C	108.6 (15)
H1B—C1—H1C	103.1 (16)	H11B—C11—H11C	106.5 (15)
C2—C1—H1A	115.0 (12)	C12—C11—H11A	111.4 (11)
C2—C1—H1B	113.3 (13)	C12—C11—H11B	110.9 (10)
C2—C1—H1C	110.2 (12)	C12—C11—H11C	109.4 (11)
O4—C2—C1	117.22 (10)	O11—C12—C11	114.43 (9)
O4—C2—C3	123.67 (10)	O11—C12—C13	124.15 (10)
C3—C2—C1	119.10 (10)	C13—C12—C11	121.35 (10)
C2—C3—H3	117.7 (11)	C12—C13—H13	118.0 (10)
C4—C3—C2	123.42 (10)	C12—C13—C14	123.58 (10)
C4—C3—H3	118.8 (11)	C14—C13—H13	118.3 (10)
O6—C4—C3	124.17 (10)	O9—C14—C13	123.53 (10)
O6—C4—C5	114.20 (10)	O9—C14—C15	117.53 (10)
C3—C4—C5	121.60 (10)	C13—C14—C15	118.93 (10)
C4—C5—H5A	112.3 (11)	C14—C15—H15A	113.4 (12)
C4—C5—H5B	110.8 (11)	C14—C15—H15B	113.3 (12)
C4—C5—H5C	111.2 (12)	C14—C15—H15C	108.1 (11)
H5A—C5—H5B	106.9 (16)	H15A—C15—H15B	107.6 (16)
H5A—C5—H5C	107.7 (16)	H15A—C15—H15C	105.6 (16)
H5B—C5—H5C	107.8 (16)	H15B—C15—H15C	108.5 (15)
H6A—C6—H6B	105.2 (16)	H16A—C16—H16B	110.9 (18)
H6A—C6—H6C	101.7 (16)	H16A—C16—H16C	107.2 (17)
H6B—C6—H6C	112.9 (17)	H16B—C16—H16C	102.8 (17)
C7—C6—H6A	110.9 (12)	C17—C16—H16A	112.3 (12)
C7—C6—H6B	110.0 (12)	C17—C16—H16B	109.7 (12)
C7—C6—H6C	115.2 (12)	C17—C16—H16C	113.5 (12)
O5—C7—C6	114.28 (10)	O10—C17—C16	117.36 (11)
O5—C7—C8	124.26 (10)	O10—C17—C18	123.12 (11)
C8—C7—C6	121.45 (10)	C18—C17—C16	119.44 (11)
C7—C8—H8	121.1 (11)	C17—C18—H18	115.8 (12)
C7—C8—C9	123.91 (10)	C19—C18—C17	124.01 (10)
C9—C8—H8	114.2 (11)	C19—C18—H18	119.2 (11)
O3—C9—C8	123.05 (10)	O12—C19—C18	124.32 (10)
O3—C9—C10	117.50 (10)	O12—C19—C20	114.39 (11)
C8—C9—C10	119.42 (10)	C18—C19—C20	121.27 (11)
C9—C10—H10A	112.2 (12)	C19—C20—H20A	112.1 (13)
C9—C10—H10B	114.7 (12)	C19—C20—H20B	114.9 (12)

C9—C10—H10C	109.6 (11)	C19—C20—H20C	112.8 (13)
H10A—C10—H10B	105.2 (16)	H20A—C20—H20B	102.8 (17)
H10A—C10—H10C	108.5 (16)	H20A—C20—H20C	104.7 (18)
H10B—C10—H10C	106.2 (16)	H20B—C20—H20C	108.6 (18)
Mo1—O3—C9—C8	14.91 (16)	Mo2—O9—C14—C13	11.74 (16)
Mo1—O3—C9—C10	−167.23 (8)	Mo2—O9—C14—C15	−167.95 (8)
Mo1—O4—C2—C1	−165.78 (8)	Mo2—O10—C17—C16	−167.33 (8)
Mo1—O4—C2—C3	13.14 (16)	Mo2—O10—C17—C18	15.86 (16)
Mo1—O5—C7—C6	159.72 (8)	Mo2—O11—C12—C11	160.69 (8)
Mo1—O5—C7—C8	−21.55 (16)	Mo2—O11—C12—C13	−22.43 (17)
Mo1—O6—C4—C3	−21.18 (18)	Mo2—O12—C19—C18	−20.31 (17)
Mo1—O6—C4—C5	160.91 (8)	Mo2—O12—C19—C20	161.61 (8)
O4—C2—C3—C4	6.18 (19)	O10—C17—C18—C19	10.74 (19)
O5—C7—C8—C9	−7.08 (18)	O11—C12—C13—C14	−4.41 (19)
C1—C2—C3—C4	−174.93 (11)	C11—C12—C13—C14	172.26 (11)
C2—C3—C4—O6	−3.75 (19)	C12—C13—C14—O9	8.31 (19)
C2—C3—C4—C5	174.01 (11)	C12—C13—C14—C15	−172.00 (11)
C6—C7—C8—C9	171.56 (11)	C16—C17—C18—C19	−166.01 (12)
C7—C8—C9—O3	9.00 (18)	C17—C18—C19—O12	−10.0 (2)
C7—C8—C9—C10	−168.82 (11)	C17—C18—C19—C20	167.99 (12)

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C1—H1*A*···O8^{i}	0.93 (1)	2.62 (2)	3.4834 (16)	155 (2)
C5—H5*B*···O10^{ii}	0.98 (1)	2.50 (1)	3.4652 (15)	170 (2)
C6—H6*A*···O8	0.97 (1)	2.51 (2)	3.3894 (15)	151 (2)
C8—H8···O7^{iii}	0.91 (1)	2.79 (2)	3.4071 (14)	126 (1)
C10—H10*A*···O6^{iv}	0.95 (1)	2.68 (2)	3.3334 (15)	127 (1)
C10—H10*C*···O1^{v}	0.97 (1)	2.50 (2)	3.3126 (15)	141 (2)
C11—H11*B*···O3^{i}	0.99 (1)	2.48 (1)	3.4415 (14)	163 (2)
C15—H15*A*···O1^{ii}	0.93 (1)	2.55 (2)	3.4592 (15)	167 (2)
C15—H15*C*···O4	0.96 (1)	2.53 (2)	3.4018 (15)	152 (2)
C16—H16*A*···O11^{vi}	0.94 (1)	2.66 (2)	3.3127 (15)	128 (2)
C16—H16*B*···O8^{vii}	0.96 (2)	2.52 (2)	3.3971 (17)	153 (2)
C18—H18···O2^{viii}	0.92 (1)	2.82 (2)	3.4646 (15)	128 (1)

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