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Octakis[2,2',5,5'-tetrathiafulvalenium(0.5+)] bis[hexamolybdate(2-)] acetonitrile solvate

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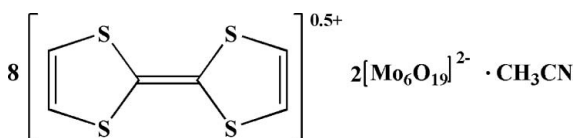
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 Key indicators: single-crystal X-ray study; $T = 100$ K; mean $\sigma(\text{C}-\text{C}) = 0.008$ Å; R factor = 0.036; wR factor = 0.073; data-to-parameter ratio = 17.8.

The asymmetric unit of the title compound, $(\text{C}_6\text{H}_4\text{S}_4)_8 \cdot [\text{Mo}_6\text{O}_{19}]_2 \cdot 2\text{CH}_3\text{CN}$, contains two halves of two centrosymmetric $[\text{Mo}_6\text{O}_{19}]^{2-}$ hexamolybdate anions, which are each built up from six distorted MoO_6 octahedra sharing common edges and one common vertex at the central O atom, six tetrathiafulvalene cations (three of which are located on mirror planes) to balance the charge and a half of an acetonitrile solvent molecule, likewise located on a mirror plane. The two central hexamolybdate O atoms occupy special positions $2a$ and $2d$, respectively. The cations and anions are interlinked through $\text{C}-\text{H} \cdots \text{O}$ contacts.

Related literature

For the chemical and physical properties of polyoxomolybdates, see: Shi *et al.* (2006); Wang *et al.* (2004); Hagrman *et al.* (1999). For the structure of ammonium tris(tetraethylammonium) hexacosaoxidooctamolybdate, see: Zebiri *et al.* (2008). The title compound is isostructural with its tungsten analogue, see: Triki *et al.* (1993). The structure of the anions is the same as that in bis[2-(pyrimidin-2-ylamino)pyrimidinium] hexamolybdate, see: Yeh *et al.* (2008). For Mo—O distances, see: Boyle *et al.* (1998); Deng *et al.* (2006); Maeda *et al.* (2006).



Experimental

Crystal data

 $(\text{C}_6\text{H}_4\text{S}_4)_8[\text{Mo}_6\text{O}_{19}]_2 \cdot 2\text{C}_2\text{H}_3\text{N}$
 $M_r = 3434.99$
 Monoclinic, $P2_1/m$
 $a = 14.3179$ (8) Å
 $b = 20.2299$ (10) Å
 $c = 16.7625$ (10) Å

 $\beta = 101.266$ (3)°
 $V = 4761.7$ (5) Å³
 $Z = 2$
 Mo $K\alpha$ radiation

 $\mu = 2.31$ mm⁻¹
 $T = 100$ K
 $0.25 \times 0.15 \times 0.10$ mm

Data collection

 Nonius KappaCCD diffractometer
 Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
 $T_{\min} = 0.597$, $T_{\max} = 0.802$
 45527 measured reflections
 11170 independent reflections
 8144 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.072$

Refinement

 $R[F^2 > 2\sigma(F^2)] = 0.036$
 $wR(F^2) = 0.073$
 $S = 1$
 11170 reflections
 629 parameters
 H-atom parameters constrained
 $\Delta\rho_{\max} = 0.88$ e Å⁻³
 $\Delta\rho_{\min} = -0.70$ e Å⁻³

 Table 1
 Hydrogen-bond geometry (Å, °).

| $D-\text{H} \cdots A$ | $D-\text{H}$ | $\text{H} \cdots A$ | $D \cdots A$ | $D-\text{H} \cdots A$ |
|--|--------------|---------------------|--------------|-----------------------|
| $\text{C5}-\text{H5} \cdots \text{O11}^{\text{i}}$ | 0.93 | 2.28 | 3.192 (6) | 167 |
| $\text{C7}-\text{H7} \cdots \text{O19}^{\text{ii}}$ | 0.93 | 2.47 | 3.251 (7) | 141 |
| $\text{C7}-\text{H7} \cdots \text{O19}^{\text{iii}}$ | 0.93 | 2.47 | 3.251 (7) | 141 |
| $\text{C9}-\text{H9} \cdots \text{O12}$ | 0.93 | 2.54 | 3.388 (5) | 151 |
| $\text{C13}-\text{H13} \cdots \text{O17}^{\text{iv}}$ | 0.93 | 2.41 | 3.239 (6) | 149 |
| $\text{C14}-\text{H14} \cdots \text{O19}^{\text{v}}$ | 0.93 | 2.59 | 3.175 (5) | 121 |
| $\text{C18}-\text{H18} \cdots \text{O18}^{\text{vi}}$ | 0.93 | 2.41 | 3.145 (5) | 136 |
| $\text{C19}-\text{H19} \cdots \text{O10}^{\text{vii}}$ | 0.93 | 2.41 | 3.298 (5) | 160 |
| $\text{C20}-\text{H20} \cdots \text{O12}$ | 0.93 | 2.54 | 3.437 (5) | 162 |
| $\text{C24}-\text{H24} \cdots \text{O8}^{\text{viii}}$ | 0.93 | 2.25 | 3.075 (6) | 148 |
| $\text{C28}-\text{H28} \cdots \text{O4}^{\text{viii}}$ | 0.93 | 2.53 | 3.384 (6) | 152 |
| $\text{C29}-\text{H29} \cdots \text{O5}^{\text{viii}}$ | 0.93 | 2.52 | 3.256 (6) | 137 |
| $\text{C29}-\text{H29} \cdots \text{O6}^{\text{viii}}$ | 0.93 | 2.55 | 3.284 (6) | 136 |

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

Data collection: *COLLECT* (Nonius, 2002); cell refinement: *DENZO* and *SCALEPACK* (Otwinowski & Minor, 1997); data reduction: *DENZO* and *SCALEPACK*; program(s) used to solve structure: *SIR92* (Altomare *et al.*, 1993); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *ORTEP-3* (Farrugia, 1997) and *Mercury* (Macrae *et al.*, 2006); software used to prepare material for publication: *WinGX* (Farrugia, 1999) and *PARST* (Nardelli, 1995).

This work was supported by Laboratoire de Chimie des Matériaux Faculté des Sciences, Université Mentouri. We would like to thank J.-Y. Saillard and Thierry Roinsel from Rennes 1 University for providing diffraction facilities.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: HG2545).

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supplementary materials

Acta Cryst. (2009). E65, m1063-m1064 [doi:10.1107/S1600536809031092]

Octakis[2,2',5,5'-tetrathiafulvalenium(0.5+)] bis[hexamolybdate(2-)] acetonitrile solvate

I. Zebiri, B. Anak, Y. Djebli, S. Boufas and L. Bencharif

Comment

As part of an ongoing study of materials containing polyoxomolybdates, we have just recently determined the structure of ammonium tris(tetraethylammonium) hexacosaoxidooctamolybdate (Zebiri *et al.*, 2008). These materials show interesting chemical and physical properties (Shi *et al.*, 2006; Wang, *et al.*, 2004; Hagrman, *et al.*, 1999).

The asymmetric unit of the title compound consists of a half neutral acetonitrile; two hexamolybdate anions and six tetrathiafulvalene in which two ones are (+1) charged and the rest ones with +0.5 charge (Fig. 1). The title compound is isostructural to (TTF)₄W₆O₁₉·0.5(CH₃CN) reported by Triki *et al.* (1993). The structure of the anions, as reported recently in Bis[2-(pyrimidin-2-ylamino)pyrimidinium] hexamolybdate (Yeh *et al.*, 2008), is constructed from an array of six edge-shared MoO₆ octahedra with six O(*t*), ten O(μ 2) and one O(μ 6) atoms.

The Mo—O distances, ranging from 1.681 (3) to 2.3363 (4) Å, agree with those reported for other [Mo₆O₁₉]²⁻ anions in the literature (Deng *et al.*, 2006; Maeda *et al.*, 2006; Boyle *et al.*, 1998) and can be grouped into three sets bridging groups [Mo—O(terminal) 1.681 (3)–1.696 (3) Å, Mo—O(μ 2): 1.850 (3)–2.027 (3) (1) Å and Mo—O(μ 6): 2.3151 (4) (1)–2.3363 (4) (1) Å.

Hexamolybdate anions spread along the *b* axis (Fig. 2) between which organic moieties intercalate. The cations and anions are interlinked through C—H···O contacts.

Experimental

Single crystals of the title compound were prepared from a mixture of (NH₄)₆Mo₇O₂₄·1.5 H₂O (137 mg, 1 mmol), C₆S₄H₄ (TTF) (612 mg, 3 mmol) and 3 ml H₂O, heated in a Teflon-lined steel autoclave inside a programmable electric furnace at 160 °C for 3 days. After cooling the autoclave to room temperature for 72 h, colorless crystals were obtained, filtered, washed with H₂O, EtOH, Et₂O and dried in air.

Refinement

H atoms were positioned geometrically and treated as riding, with C—H = 0.93 Å with $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{C})$. The methyl H atoms were constrained to an ideal geometry (C—H = 0.96 Å) with $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{C})$, but were allowed to rotate freely about the C—C bonds.

Figures

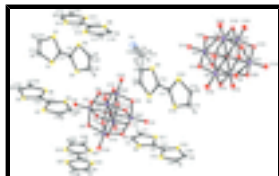


Fig. 1. The independent components of (I), showing the atom-labelling scheme. Displacement ellipsoids are drawn at the 50% probability level.

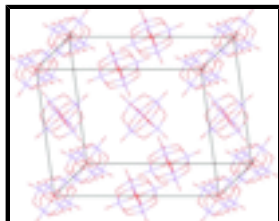


Fig. 2. hexamolybdate anions positions on the unit cell.

Octakis[2,2',5,5'-tetrathiafulvalenium(0.5+)] bis[hexamolybdate(2-)] acetonitrile solvate

Crystal data

$(C_6H_4S_4)_8[Mo_6O_{19}]_2 \cdot C_2H_3N$

$M_r = 3434.99$

Monoclinic, $P2_1/m$

Hall symbol: -P 2yb

$a = 14.3179$ (8) Å

$b = 20.2299$ (10) Å

$c = 16.7625$ (10) Å

$\beta = 101.266$ (3)°

$V = 4761.7$ (5) Å³

$Z = 2$

$F_{000} = 3324$

$D_x = 2.396$ Mg m⁻³

Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å

Cell parameters from 2190 reflections

$\theta = 2.8$ – 27.3 °

$\mu = 2.31$ mm⁻¹

$T = 100$ K

Plates, colourless

$0.25 \times 0.15 \times 0.1$ mm

Data collection

Nonius KappaCCD
diffractometer

Radiation source: fine-focus sealed X-ray tube

Monochromator: graphite

$T = 100$ K

φ scans, and ω scans with κ offsets

Absorption correction: multi-scan
(SADABS; Sheldrick, 1996)

$T_{\min} = 0.597$, $T_{\max} = 0.802$

45527 measured reflections

11170 independent reflections

8144 reflections with $I > 2\sigma(I)$

$R_{\text{int}} = 0.072$

$\theta_{\max} = 27.5$ °

$\theta_{\min} = 1.5$ °

$h = -18 \rightarrow 18$

$k = -26 \rightarrow 26$

$l = -21 \rightarrow 21$

Refinement

Refinement on F^2

Least-squares matrix: full

Hydrogen site location: inferred from neighbouring sites

H-atom parameters constrained

| | |
|--|---|
| $R[F^2 > 2\sigma(F^2)] = 0.036$ | $w = 1/[\sigma^2(F_o^2) + (0.021P)^2]$ |
| $wR(F^2) = 0.073$ | where $P = (F_o^2 + 2F_c^2)/3$ |
| $S = 1$ | $(\Delta/\sigma)_{\max} = 0.003$ |
| 11170 reflections | $\Delta\rho_{\max} = 0.88 \text{ e } \text{\AA}^{-3}$ |
| 629 parameters | $\Delta\rho_{\min} = -0.70 \text{ e } \text{\AA}^{-3}$ |
| Primary atom site location: structure-invariant direct methods | Extinction correction: SHELXL97 (Sheldrick, 2008), $F_c^* = kFc[1+0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$ |
| Secondary atom site location: difference Fourier map | Extinction coefficient: 0.08 (2) |

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.

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

| | <i>x</i> | <i>y</i> | <i>z</i> | $U_{\text{iso}}^*/U_{\text{eq}}$ | Occ. (<1) |
|-----|---------------|---------------|--------------|----------------------------------|-----------|
| Mo1 | 0.56723 (3) | 0.411518 (19) | 0.58067 (2) | 0.01535 (9) | |
| Mo2 | 0.37343 (3) | 0.500847 (18) | 0.56738 (2) | 0.01262 (8) | |
| Mo5 | 0.41862 (3) | 0.425991 (19) | 0.40423 (2) | 0.01562 (9) | |
| Mo3 | 0.09471 (3) | 0.007942 (18) | 0.90255 (2) | 0.01440 (9) | |
| Mo4 | 0.12057 (3) | 0.050345 (18) | 1.09450 (2) | 0.01550 (9) | |
| Mo6 | -0.06607 (3) | 0.102673 (18) | 0.96110 (2) | 0.01461 (9) | |
| S4 | 0.69926 (8) | 0.32365 (5) | 0.32774 (7) | 0.0171 (2) | |
| S1 | 0.83864 (9) | 0.32314 (6) | 0.19478 (7) | 0.0194 (3) | |
| S2 | 0.31973 (9) | 0.32349 (5) | 0.68472 (7) | 0.0193 (3) | |
| S3 | 1.13888 (8) | 0.41650 (5) | 0.47871 (7) | 0.0141 (2) | |
| S5 | 0.85700 (8) | 0.41673 (5) | 0.53863 (7) | 0.0134 (2) | |
| S6 | 0.94719 (8) | 0.41562 (5) | 0.37833 (7) | 0.0148 (2) | |
| S7 | 1.04691 (8) | 0.41757 (5) | 0.64209 (7) | 0.0158 (2) | |
| S8 | 0.19893 (11) | 0.75 | 0.39380 (10) | 0.0162 (3) | |
| S9 | 0.38303 (8) | 0.16665 (6) | 0.03769 (7) | 0.0186 (3) | |
| S10 | -0.09582 (11) | 0.75 | 0.43604 (10) | 0.0167 (3) | |
| S11 | 0.01173 (12) | 0.75 | 0.27956 (10) | 0.0187 (4) | |
| S12 | 0.09410 (11) | 0.75 | 0.54638 (9) | 0.0157 (3) | |
| S13 | 0.57537 (8) | 0.16549 (5) | 0.13620 (7) | 0.0169 (2) | |
| S14 | 0.47171 (8) | 0.16799 (6) | -0.12637 (7) | 0.0174 (2) | |
| S15 | 0.66417 (8) | 0.16465 (6) | -0.02771 (7) | 0.0204 (3) | |
| S18 | 0.21028 (8) | 0.32348 (5) | 0.83822 (7) | 0.0177 (2) | |
| O1 | 0 | 0 | 1 | 0.0106 (9) | |
| O2 | 0.5 | 0.5 | 0.5 | 0.0119 (9) | |
| O5 | 0.4513 (2) | 0.43340 (14) | 0.62260 (17) | 0.0153 (7) | |
| O4 | 0.2801 (2) | 0.50318 (14) | 0.61450 (18) | 0.0159 (7) | |
| O3 | 0.6587 (2) | 0.43007 (14) | 0.51664 (18) | 0.0169 (7) | |

supplementary materials

| | | | | |
|-----|-------------|---------------|--------------|-------------|
| O6 | 0.4587 (2) | 0.56620 (14) | 0.62598 (17) | 0.0150 (7) |
| O12 | 0.4888 (2) | 0.36829 (14) | 0.49387 (18) | 0.0161 (7) |
| O7 | 0.6179 (2) | 0.49293 (14) | 0.64424 (17) | 0.0166 (7) |
| O8 | -0.0228 (2) | -0.03840 (14) | 0.84668 (18) | 0.0195 (7) |
| O9 | 0.3326 (2) | 0.44003 (14) | 0.47865 (18) | 0.0159 (7) |
| O10 | 0.0391 (2) | 0.12578 (13) | 1.03983 (18) | 0.0166 (7) |
| O11 | 0.0124 (2) | 0.08657 (13) | 0.88356 (18) | 0.0178 (7) |
| O13 | 0.1560 (2) | -0.03770 (14) | 1.10491 (19) | 0.0204 (7) |
| O14 | 0.1549 (2) | 0.01313 (14) | 0.82549 (19) | 0.0215 (7) |
| O15 | 0.1685 (2) | 0.05279 (13) | 0.99087 (18) | 0.0158 (7) |
| O16 | 0.3610 (2) | 0.36980 (15) | 0.33835 (19) | 0.0234 (8) |
| O19 | -0.1227 (2) | 0.17476 (14) | 0.93106 (19) | 0.0221 (7) |
| O18 | 0.2048 (2) | 0.09129 (14) | 1.1612 (2) | 0.0245 (8) |
| O17 | 0.6166 (2) | 0.35055 (15) | 0.64365 (19) | 0.0245 (8) |
| O20 | 0.1308 (2) | -0.07465 (14) | 0.95163 (19) | 0.0200 (7) |
| C1 | 0.7991 (4) | 0.25 | 0.2352 (4) | 0.0127 (13) |
| C4 | 0.9789 (3) | 0.4167 (2) | 0.5449 (3) | 0.0145 (9) |
| C3 | 0.1983 (5) | 0.75 | 0.2897 (4) | 0.0204 (15) |
| H3 | 0.2548 | 0.75 | 0.2703 | 0.025* |
| C2 | 0.7423 (4) | 0.25 | 0.2902 (4) | 0.0143 (13) |
| C7 | 0.1143 (5) | 0.75 | 0.2388 (4) | 0.0224 (15) |
| H7 | 0.1107 | 0.75 | 0.1828 | 0.027* |
| C8 | 0.0738 (5) | 0.75 | 0.3820 (4) | 0.0170 (14) |
| C6 | 0.8635 (3) | 0.4170 (2) | 0.6423 (3) | 0.0166 (10) |
| H6 | 0.8088 | 0.4169 | 0.6643 | 0.02* |
| C5 | 0.9505 (3) | 0.4174 (2) | 0.6901 (3) | 0.0178 (10) |
| H5 | 0.9585 | 0.4176 | 0.7465 | 0.021* |
| C11 | 0.2420 (4) | 0.25 | 0.7930 (4) | 0.0149 (13) |
| C10 | 0.2854 (4) | 0.25 | 0.7292 (4) | 0.0160 (14) |
| C15 | 0.5424 (3) | 0.1666 (2) | -0.0301 (3) | 0.0174 (10) |
| C16 | 0.5044 (3) | 0.1662 (2) | 0.0406 (3) | 0.0172 (10) |
| C13 | 0.5655 (3) | 0.1660 (2) | -0.1775 (3) | 0.0216 (11) |
| H13 | 0.5553 | 0.166 | -0.234 | 0.026* |
| C12 | 0.1572 (3) | 0.2831 (2) | 0.9098 (3) | 0.0223 (11) |
| H12 | 0.1297 | 0.3068 | 0.9468 | 0.027* |
| C14 | 0.6539 (3) | 0.1643 (2) | -0.1321 (3) | 0.0210 (11) |
| H14 | 0.7074 | 0.163 | -0.1559 | 0.025* |
| C9 | 0.3514 (3) | 0.2832 (2) | 0.6016 (3) | 0.0231 (11) |
| H9 | 0.3676 | 0.307 | 0.5588 | 0.028* |
| C22 | 1.0177 (3) | 0.41639 (19) | 0.4749 (3) | 0.0132 (9) |
| C17 | 0.4818 (3) | 0.1637 (2) | 0.1873 (3) | 0.0180 (10) |
| H17 | 0.4923 | 0.1623 | 0.2438 | 0.022* |
| C18 | 0.3931 (3) | 0.1643 (2) | 0.1421 (3) | 0.0185 (10) |
| H18 | 0.3396 | 0.1635 | 0.1659 | 0.022* |
| C21 | 0.0295 (4) | 0.75 | 0.4457 (4) | 0.0165 (14) |
| C20 | 0.6422 (3) | 0.2833 (2) | 0.3968 (3) | 0.0211 (11) |
| H20 | 0.6127 | 0.307 | 0.4325 | 0.025* |
| C19 | 0.9255 (3) | 0.2830 (2) | 0.1533 (3) | 0.0235 (11) |
| H19 | 0.9704 | 0.3067 | 0.1319 | 0.028* |

| | | | | | |
|------|-------------|-------------|-------------|-------------|-----|
| C24 | 1.0417 (3) | 0.4110 (2) | 0.3287 (3) | 0.0197 (10) | |
| H24 | 1.0322 | 0.4084 | 0.2723 | 0.024* | |
| C23 | 1.1297 (3) | 0.4114 (2) | 0.3748 (3) | 0.0174 (10) | |
| H23 | 1.1836 | 0.4089 | 0.3516 | 0.021* | |
| C26 | -0.0077 (4) | 0.75 | 0.5908 (4) | 0.0184 (14) | |
| H26 | -0.0027 | 0.75 | 0.6469 | 0.022* | |
| C25 | -0.0915 (5) | 0.75 | 0.5408 (4) | 0.0186 (14) | |
| H25 | -0.1474 | 0.75 | 0.5613 | 0.022* | |
| S16 | 0.43075 (9) | 0.99818 (6) | 0.10594 (7) | 0.0200 (3) | |
| S27 | 0.63331 (9) | 0.99422 (6) | 0.09037 (7) | 0.0203 (3) | |
| C27 | 0.5135 (3) | 0.9988 (2) | 0.0403 (3) | 0.0166 (10) | |
| C29 | 0.5178 (4) | 0.9954 (2) | 0.1949 (3) | 0.0245 (11) | |
| H29 | 0.5008 | 0.9954 | 0.2457 | 0.029* | |
| C28 | 0.6083 (4) | 0.9931 (2) | 0.1884 (3) | 0.0247 (11) | |
| H28 | 0.6568 | 0.991 | 0.2342 | 0.03* | |
| N1 | 0.1799 (6) | 0.25 | 0.1110 (5) | 0.061 (2) | |
| C30 | 0.1822 (6) | 0.25 | 0.1791 (7) | 0.049 (3) | |
| C31 | 0.1876 (6) | 0.25 | 0.2689 (6) | 0.050 (2) | |
| H31A | 0.1353 | 0.2749 | 0.2815 | 0.076* | 0.5 |
| H31B | 0.2466 | 0.2697 | 0.2953 | 0.076* | 0.5 |
| H31C | 0.1845 | 0.2054 | 0.2876 | 0.076* | 0.5 |

Atomic displacement parameters (\AA^2)

| | U^{11} | U^{22} | U^{33} | U^{12} | U^{13} | U^{23} |
|-----|--------------|------------|--------------|---------------|--------------|---------------|
| Mo1 | 0.0150 (2) | 0.0195 (2) | 0.0125 (2) | 0.00474 (16) | 0.00522 (16) | 0.00528 (16) |
| Mo2 | 0.01036 (18) | 0.0189 (2) | 0.00921 (18) | 0.00057 (16) | 0.00335 (15) | 0.00047 (15) |
| Mo5 | 0.0138 (2) | 0.0217 (2) | 0.01209 (19) | -0.00355 (16) | 0.00420 (16) | -0.00492 (16) |
| Mo3 | 0.0168 (2) | 0.0162 (2) | 0.01122 (19) | -0.00087 (16) | 0.00517 (16) | 0.00087 (15) |
| Mo4 | 0.0164 (2) | 0.0170 (2) | 0.01202 (19) | -0.00345 (16) | 0.00029 (16) | -0.00233 (15) |
| Mo6 | 0.0185 (2) | 0.0131 (2) | 0.01240 (19) | 0.00125 (16) | 0.00348 (16) | 0.00127 (15) |
| S4 | 0.0199 (6) | 0.0162 (6) | 0.0154 (6) | 0.0025 (5) | 0.0038 (5) | -0.0010 (5) |
| S1 | 0.0213 (6) | 0.0191 (6) | 0.0189 (6) | -0.0011 (5) | 0.0062 (5) | 0.0029 (5) |
| S2 | 0.0241 (7) | 0.0177 (6) | 0.0166 (6) | -0.0050 (5) | 0.0055 (5) | 0.0010 (5) |
| S3 | 0.0110 (5) | 0.0164 (6) | 0.0148 (6) | -0.0019 (4) | 0.0023 (5) | -0.0003 (4) |
| S5 | 0.0114 (5) | 0.0163 (6) | 0.0122 (5) | 0.0001 (4) | 0.0018 (4) | 0.0009 (4) |
| S6 | 0.0148 (6) | 0.0171 (6) | 0.0117 (5) | -0.0021 (4) | 0.0006 (5) | 0.0014 (4) |
| S7 | 0.0152 (6) | 0.0186 (6) | 0.0121 (5) | -0.0005 (5) | -0.0008 (5) | 0.0000 (4) |
| S8 | 0.0155 (8) | 0.0175 (8) | 0.0151 (8) | 0 | 0.0017 (7) | 0 |
| S9 | 0.0149 (6) | 0.0223 (6) | 0.0184 (6) | -0.0005 (5) | 0.0028 (5) | -0.0022 (5) |
| S10 | 0.0151 (8) | 0.0176 (8) | 0.0161 (8) | 0 | -0.0006 (7) | 0 |
| S11 | 0.0227 (9) | 0.0184 (8) | 0.0124 (8) | 0 | -0.0027 (7) | 0 |
| S12 | 0.0155 (8) | 0.0173 (8) | 0.0126 (8) | 0 | -0.0016 (7) | 0 |
| S13 | 0.0163 (6) | 0.0215 (6) | 0.0127 (6) | 0.0001 (5) | 0.0021 (5) | -0.0004 (5) |
| S14 | 0.0188 (6) | 0.0213 (6) | 0.0118 (6) | 0.0011 (5) | 0.0019 (5) | -0.0001 (5) |
| S15 | 0.0158 (6) | 0.0278 (7) | 0.0182 (6) | 0.0030 (5) | 0.0046 (5) | 0.0017 (5) |
| S18 | 0.0210 (6) | 0.0147 (6) | 0.0177 (6) | -0.0002 (5) | 0.0046 (5) | -0.0021 (5) |
| O1 | 0.013 (2) | 0.012 (2) | 0.007 (2) | -0.0012 (17) | 0.0024 (17) | -0.0010 (16) |

supplementary materials

| | | | | | | |
|-----|-------------|-------------|-------------|--------------|--------------|--------------|
| O2 | 0.010 (2) | 0.016 (2) | 0.011 (2) | 0.0002 (17) | 0.0043 (18) | 0.0001 (17) |
| O5 | 0.0175 (17) | 0.0193 (16) | 0.0103 (15) | 0.0010 (13) | 0.0054 (13) | 0.0021 (12) |
| O4 | 0.0135 (16) | 0.0218 (17) | 0.0133 (16) | -0.0001 (13) | 0.0047 (13) | -0.0004 (13) |
| O3 | 0.0134 (16) | 0.0221 (16) | 0.0164 (17) | 0.0041 (13) | 0.0052 (14) | 0.0028 (13) |
| O6 | 0.0153 (16) | 0.0199 (16) | 0.0109 (15) | -0.0017 (13) | 0.0053 (13) | -0.0024 (13) |
| O12 | 0.0207 (17) | 0.0149 (16) | 0.0149 (16) | -0.0004 (13) | 0.0091 (14) | -0.0004 (12) |
| O7 | 0.0131 (16) | 0.0259 (17) | 0.0102 (16) | 0.0016 (13) | 0.0012 (13) | 0.0014 (13) |
| O8 | 0.0240 (18) | 0.0230 (17) | 0.0114 (16) | -0.0007 (14) | 0.0032 (14) | -0.0036 (13) |
| O9 | 0.0137 (16) | 0.0207 (17) | 0.0149 (16) | -0.0036 (13) | 0.0068 (13) | -0.0031 (13) |
| O10 | 0.0193 (17) | 0.0122 (15) | 0.0188 (17) | -0.0036 (13) | 0.0049 (14) | -0.0019 (13) |
| O11 | 0.0200 (17) | 0.0164 (16) | 0.0168 (17) | -0.0021 (13) | 0.0026 (14) | 0.0020 (13) |
| O13 | 0.0192 (17) | 0.0219 (17) | 0.0173 (17) | -0.0010 (14) | -0.0034 (14) | 0.0015 (14) |
| O14 | 0.0254 (19) | 0.0192 (17) | 0.0229 (19) | -0.0013 (14) | 0.0120 (16) | 0.0011 (14) |
| O15 | 0.0126 (16) | 0.0171 (16) | 0.0183 (17) | -0.0035 (13) | 0.0040 (13) | -0.0032 (13) |
| O16 | 0.0220 (18) | 0.0321 (19) | 0.0167 (18) | -0.0051 (15) | 0.0054 (15) | -0.0125 (14) |
| O19 | 0.0264 (19) | 0.0181 (17) | 0.0227 (18) | 0.0025 (14) | 0.0066 (16) | 0.0043 (14) |
| O18 | 0.0244 (18) | 0.0251 (18) | 0.0215 (18) | -0.0084 (14) | -0.0017 (15) | -0.0043 (14) |
| O17 | 0.028 (2) | 0.0275 (18) | 0.0197 (19) | 0.0115 (15) | 0.0097 (16) | 0.0075 (14) |
| O20 | 0.0219 (18) | 0.0160 (16) | 0.0245 (19) | -0.0015 (13) | 0.0102 (15) | -0.0005 (14) |
| C1 | 0.015 (3) | 0.013 (3) | 0.011 (3) | 0 | 0.006 (3) | 0 |
| C4 | 0.011 (2) | 0.012 (2) | 0.019 (2) | 0.0005 (17) | -0.0005 (19) | 0.0019 (18) |
| C3 | 0.022 (4) | 0.028 (4) | 0.012 (3) | 0 | 0.006 (3) | 0 |
| C2 | 0.018 (3) | 0.014 (3) | 0.009 (3) | 0 | -0.002 (3) | 0 |
| C7 | 0.036 (4) | 0.021 (4) | 0.012 (3) | 0 | 0.010 (3) | 0 |
| C8 | 0.026 (4) | 0.019 (3) | 0.005 (3) | 0 | -0.001 (3) | 0 |
| C6 | 0.016 (2) | 0.017 (2) | 0.018 (2) | 0.0003 (18) | 0.006 (2) | 0.0009 (19) |
| C5 | 0.026 (3) | 0.017 (2) | 0.011 (2) | -0.002 (2) | 0.005 (2) | -0.0006 (18) |
| C11 | 0.014 (3) | 0.015 (3) | 0.014 (3) | 0 | -0.003 (3) | 0 |
| C10 | 0.016 (3) | 0.015 (3) | 0.015 (3) | 0 | 0.000 (3) | 0 |
| C15 | 0.016 (2) | 0.021 (2) | 0.014 (2) | 0.0026 (19) | 0.001 (2) | -0.0020 (19) |
| C16 | 0.019 (2) | 0.018 (2) | 0.015 (2) | -0.0004 (19) | 0.004 (2) | -0.0005 (19) |
| C13 | 0.032 (3) | 0.021 (3) | 0.014 (2) | 0.005 (2) | 0.010 (2) | 0.0023 (19) |
| C12 | 0.019 (3) | 0.028 (3) | 0.021 (3) | 0.005 (2) | 0.009 (2) | -0.001 (2) |
| C14 | 0.020 (3) | 0.028 (3) | 0.017 (3) | 0.006 (2) | 0.010 (2) | 0.001 (2) |
| C9 | 0.024 (3) | 0.031 (3) | 0.016 (3) | -0.003 (2) | 0.008 (2) | 0.002 (2) |
| C22 | 0.014 (2) | 0.007 (2) | 0.016 (2) | -0.0029 (17) | -0.0017 (19) | -0.0001 (17) |
| C17 | 0.023 (3) | 0.019 (2) | 0.013 (2) | 0.001 (2) | 0.007 (2) | -0.0021 (19) |
| C18 | 0.021 (3) | 0.016 (2) | 0.022 (3) | -0.0033 (19) | 0.011 (2) | -0.0040 (19) |
| C21 | 0.014 (3) | 0.018 (3) | 0.013 (3) | 0 | -0.006 (3) | 0 |
| C20 | 0.024 (3) | 0.030 (3) | 0.010 (2) | 0.006 (2) | 0.007 (2) | -0.0007 (19) |
| C19 | 0.023 (3) | 0.034 (3) | 0.017 (3) | -0.003 (2) | 0.011 (2) | 0.001 (2) |
| C24 | 0.025 (3) | 0.022 (3) | 0.011 (2) | -0.005 (2) | 0.002 (2) | 0.0009 (19) |
| C23 | 0.021 (2) | 0.020 (2) | 0.012 (2) | -0.0021 (19) | 0.005 (2) | 0.0018 (18) |
| C26 | 0.021 (4) | 0.015 (3) | 0.020 (4) | 0 | 0.005 (3) | 0 |
| C25 | 0.027 (4) | 0.013 (3) | 0.017 (3) | 0 | 0.006 (3) | 0 |
| S16 | 0.0263 (7) | 0.0219 (6) | 0.0124 (6) | 0.0039 (5) | 0.0053 (5) | -0.0005 (5) |
| S27 | 0.0219 (6) | 0.0245 (6) | 0.0127 (6) | 0.0035 (5) | -0.0012 (5) | -0.0004 (5) |
| C27 | 0.021 (2) | 0.015 (2) | 0.013 (2) | 0.0028 (19) | 0.003 (2) | -0.0002 (18) |
| C29 | 0.039 (3) | 0.022 (3) | 0.010 (2) | 0.002 (2) | -0.001 (2) | -0.0004 (19) |

| | | | | | | |
|-----|-----------|-----------|-----------|-----------|------------|--------------|
| C28 | 0.035 (3) | 0.022 (3) | 0.012 (2) | 0.005 (2) | -0.007 (2) | -0.0015 (19) |
| N1 | 0.071 (6) | 0.060 (5) | 0.050 (5) | 0 | 0.002 (5) | 0 |
| C30 | 0.038 (5) | 0.026 (5) | 0.084 (8) | 0 | 0.014 (6) | 0 |
| C31 | 0.051 (6) | 0.033 (5) | 0.071 (7) | 0 | 0.021 (5) | 0 |

Geometric parameters (Å, °)

| | | | |
|-----------------------|------------|------------------------|------------|
| Mo1—O17 | 1.686 (3) | S15—C14 | 1.727 (5) |
| Mo1—O12 | 1.872 (3) | S15—C15 | 1.736 (5) |
| Mo1—O3 | 1.886 (3) | S18—C12 | 1.743 (5) |
| Mo1—O5 | 1.974 (3) | S18—C11 | 1.768 (4) |
| Mo1—O7 | 2.018 (3) | O1—Mo6 ⁱⁱ | 2.3222 (4) |
| Mo1—O2 | 2.3345 (4) | O1—Mo3 ⁱⁱ | 2.3234 (4) |
| Mo2—O4 | 1.680 (3) | O1—Mo4 ⁱⁱ | 2.3363 (4) |
| Mo2—O5 | 1.887 (3) | O2—Mo2 ⁱ | 2.3151 (4) |
| Mo2—O9 | 1.931 (3) | O2—Mo5 ⁱ | 2.3335 (4) |
| Mo2—O6 | 1.932 (3) | O2—Mo1 ⁱ | 2.3345 (4) |
| Mo2—O3 ⁱ | 1.974 (3) | O3—Mo2 ⁱ | 1.974 (3) |
| Mo2—O2 | 2.3151 (4) | O6—Mo5 ⁱ | 1.928 (3) |
| Mo5—O16 | 1.684 (3) | O7—Mo5 ⁱ | 1.860 (3) |
| Mo5—O7 ⁱ | 1.860 (3) | O8—Mo4 ⁱⁱ | 1.879 (3) |
| Mo5—O6 ⁱ | 1.928 (3) | O13—Mo6 ⁱⁱ | 2.013 (3) |
| Mo5—O9 | 1.937 (3) | O20—Mo6 ⁱⁱ | 1.962 (3) |
| Mo5—O12 | 2.010 (3) | C1—C2 | 1.343 (8) |
| Mo5—O2 | 2.3335 (4) | C1—S1 ⁱⁱⁱ | 1.766 (3) |
| Mo3—O14 | 1.690 (3) | C4—C22 | 1.393 (6) |
| Mo3—O15 | 1.876 (3) | C3—C7 | 1.331 (9) |
| Mo3—O20 | 1.889 (3) | C3—H3 | 0.93 |
| Mo3—O11 | 1.968 (3) | C2—S4 ⁱⁱⁱ | 1.774 (4) |
| Mo3—O8 | 1.993 (3) | C7—H7 | 0.93 |
| Mo3—O1 | 2.3234 (4) | C8—C21 | 1.345 (9) |
| Mo4—O18 | 1.692 (3) | C6—C5 | 1.343 (6) |
| Mo4—O13 | 1.851 (3) | C6—H6 | 0.93 |
| Mo4—O8 ⁱⁱ | 1.879 (3) | C5—H5 | 0.93 |
| Mo4—O15 | 1.989 (3) | C11—C10 | 1.339 (9) |
| Mo4—O10 | 2.028 (3) | C11—S18 ⁱⁱⁱ | 1.768 (4) |
| Mo4—O1 | 2.3363 (4) | C10—S2 ⁱⁱⁱ | 1.774 (4) |
| Mo6—O19 | 1.696 (3) | C15—C16 | 1.398 (6) |
| Mo6—O10 | 1.858 (3) | C13—C14 | 1.343 (6) |
| Mo6—O11 | 1.905 (3) | C13—H13 | 0.93 |
| Mo6—O20 ⁱⁱ | 1.962 (3) | C12—C12 ⁱⁱⁱ | 1.340 (9) |
| Mo6—O13 ⁱⁱ | 2.013 (3) | C12—H12 | 0.93 |
| Mo6—O1 | 2.3222 (4) | C14—H14 | 0.93 |
| S4—C20 | 1.746 (5) | C9—C9 ⁱⁱⁱ | 1.343 (9) |
| S4—C2 | 1.774 (4) | C9—H9 | 0.93 |

supplementary materials

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|-------------|-------------|---------------------------------------|--------------|
| S1—C19 | 1.739 (5) | C17—C18 | 1.347 (6) |
| S1—C1 | 1.766 (3) | C17—H17 | 0.93 |
| S2—C9 | 1.749 (5) | C18—H18 | 0.93 |
| S2—C10 | 1.774 (4) | C20—C20 ⁱⁱⁱ | 1.347 (9) |
| S3—C23 | 1.723 (4) | C20—H20 | 0.93 |
| S3—C22 | 1.724 (4) | C19—C19 ⁱⁱⁱ | 1.335 (9) |
| S5—C6 | 1.721 (5) | C19—H19 | 0.93 |
| S5—C4 | 1.727 (4) | C24—C23 | 1.344 (6) |
| S6—C24 | 1.724 (5) | C24—H24 | 0.93 |
| S6—C22 | 1.732 (4) | C23—H23 | 0.93 |
| S7—C4 | 1.728 (4) | C26—C25 | 1.323 (9) |
| S7—C5 | 1.728 (5) | C26—H26 | 0.93 |
| S8—C3 | 1.743 (6) | C25—H25 | 0.93 |
| S8—C8 | 1.764 (7) | S16—C29 | 1.748 (5) |
| S9—C18 | 1.728 (5) | S16—C27 | 1.767 (5) |
| S9—C16 | 1.728 (5) | S27—C28 | 1.749 (5) |
| S10—C25 | 1.746 (6) | S27—C27 | 1.759 (5) |
| S10—C21 | 1.769 (6) | C27—C27 ^{iv} | 1.330 (8) |
| S11—C7 | 1.737 (7) | C29—C28 | 1.322 (7) |
| S11—C8 | 1.772 (6) | C29—H29 | 0.93 |
| S12—C21 | 1.759 (6) | C28—H28 | 0.93 |
| S12—C26 | 1.761 (7) | N1—C30 | 1.136 (12) |
| S13—C16 | 1.722 (5) | C30—C31 | 1.492 (13) |
| S13—C17 | 1.725 (5) | C31—H31A | 0.96 |
| S14—C15 | 1.729 (4) | C31—H31B | 0.96 |
| S14—C13 | 1.729 (5) | C31—H31C | 0.96 |
| O17—Mo1—O12 | 105.04 (14) | Mo2—O2—Mo5 ⁱ | 89.589 (13) |
| O17—Mo1—O3 | 104.43 (14) | Mo2 ⁱ —O2—Mo5 ⁱ | 90.411 (14) |
| O12—Mo1—O3 | 91.61 (13) | Mo2—O2—Mo5 | 90.411 (14) |
| O17—Mo1—O5 | 102.70 (14) | Mo2 ⁱ —O2—Mo5 | 89.589 (13) |
| O12—Mo1—O5 | 87.90 (12) | Mo5 ⁱ —O2—Mo5 | 180.000 (15) |
| O3—Mo1—O5 | 152.02 (12) | Mo2—O2—Mo1 | 89.800 (13) |
| O17—Mo1—O7 | 101.69 (14) | Mo2 ⁱ —O2—Mo1 | 90.200 (13) |
| O12—Mo1—O7 | 152.99 (12) | Mo5 ⁱ —O2—Mo1 | 89.969 (14) |
| O3—Mo1—O7 | 85.42 (12) | Mo5—O2—Mo1 | 90.031 (14) |
| O5—Mo1—O7 | 82.51 (12) | Mo2—O2—Mo1 ⁱ | 90.200 (13) |
| O17—Mo1—O2 | 176.68 (11) | Mo2 ⁱ —O2—Mo1 ⁱ | 89.800 (13) |
| O12—Mo1—O2 | 77.94 (9) | Mo5 ⁱ —O2—Mo1 ⁱ | 90.031 (14) |
| O3—Mo1—O2 | 76.77 (9) | Mo5—O2—Mo1 ⁱ | 89.969 (14) |
| O5—Mo1—O2 | 75.78 (8) | Mo1—O2—Mo1 ⁱ | 180 |
| O7—Mo1—O2 | 75.24 (8) | Mo2—O5—Mo1 | 116.42 (14) |
| O4—Mo2—O5 | 103.76 (13) | Mo1—O3—Mo2 ⁱ | 117.16 (14) |
| O4—Mo2—O9 | 103.37 (13) | Mo5 ⁱ —O6—Mo2 | 116.11 (14) |
| O5—Mo2—O9 | 88.74 (12) | Mo1—O12—Mo5 | 116.50 (14) |
| O4—Mo2—O6 | 102.89 (13) | Mo5 ⁱ —O7—Mo1 | 116.59 (14) |

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|--------------------------------------|-------------|-----------------------------|-------------|
| O5—Mo2—O6 | 89.50 (12) | Mo4 ⁱⁱ —O8—Mo3 | 117.46 (15) |
| O9—Mo2—O6 | 153.33 (12) | Mo2—O9—Mo5 | 117.05 (14) |
| O4—Mo2—O3 ⁱ | 102.71 (13) | Mo6—O10—Mo4 | 116.53 (14) |
| O5—Mo2—O3 ⁱ | 153.52 (12) | Mo6—O11—Mo3 | 116.24 (15) |
| O9—Mo2—O3 ⁱ | 84.73 (12) | Mo4—O13—Mo6 ⁱⁱ | 116.26 (15) |
| O6—Mo2—O3 ⁱ | 85.07 (12) | Mo3—O15—Mo4 | 116.10 (14) |
| O4—Mo2—O2 | 178.36 (10) | Mo3—O20—Mo6 ⁱⁱ | 116.81 (15) |
| O5—Mo2—O2 | 77.87 (9) | C2—C1—S1 | 123.08 (17) |
| O9—Mo2—O2 | 76.54 (9) | C2—C1—S1 ⁱⁱⁱ | 123.08 (17) |
| O6—Mo2—O2 | 77.07 (9) | S1—C1—S1 ⁱⁱⁱ | 113.8 (3) |
| O3 ⁱ —Mo2—O2 | 75.66 (8) | C22—C4—S5 | 120.9 (3) |
| O16—Mo5—O7 ⁱ | 104.41 (14) | C22—C4—S7 | 123.4 (3) |
| O16—Mo5—O6 ⁱ | 103.77 (14) | S5—C4—S7 | 115.7 (3) |
| O7 ⁱ —Mo5—O6 ⁱ | 90.34 (12) | C7—C3—S8 | 118.0 (5) |
| O16—Mo5—O9 | 103.27 (14) | C7—C3—H3 | 121 |
| O7 ⁱ —Mo5—O9 | 89.55 (12) | S8—C3—H3 | 121 |
| O6 ⁱ —Mo5—O9 | 152.09 (12) | C1—C2—S4 ⁱⁱⁱ | 122.83 (18) |
| O16—Mo5—O12 | 101.98 (14) | C1—C2—S4 | 122.83 (18) |
| O7 ⁱ —Mo5—O12 | 153.60 (12) | S4 ⁱⁱⁱ —C2—S4 | 114.3 (4) |
| O6 ⁱ —Mo5—O12 | 84.01 (12) | C3—C7—S11 | 118.3 (5) |
| O9—Mo5—O12 | 83.78 (12) | C3—C7—H7 | 120.8 |
| O16—Mo5—O2 | 177.36 (11) | S11—C7—H7 | 120.8 |
| O7 ⁱ —Mo5—O2 | 78.16 (9) | C21—C8—S8 | 122.5 (5) |
| O6 ⁱ —Mo5—O2 | 76.70 (8) | C21—C8—S11 | 123.0 (5) |
| O9—Mo5—O2 | 75.98 (8) | S8—C8—S11 | 114.5 (4) |
| O12—Mo5—O2 | 75.45 (8) | C5—C6—S5 | 117.6 (4) |
| O14—Mo3—O15 | 106.28 (14) | C5—C6—H6 | 121.2 |
| O14—Mo3—O20 | 104.51 (14) | S5—C6—H6 | 121.2 |
| O15—Mo3—O20 | 91.14 (13) | C6—C5—S7 | 117.0 (4) |
| O14—Mo3—O11 | 101.82 (14) | C6—C5—H5 | 121.5 |
| O15—Mo3—O11 | 87.94 (12) | S7—C5—H5 | 121.5 |
| O20—Mo3—O11 | 152.81 (13) | C10—C11—S18 ⁱⁱⁱ | 122.77 (18) |
| O14—Mo3—O8 | 100.35 (14) | C10—C11—S18 | 122.77 (18) |
| O15—Mo3—O8 | 153.12 (13) | S18 ⁱⁱⁱ —C11—S18 | 114.5 (4) |
| O20—Mo3—O8 | 85.58 (13) | C11—C10—S2 | 123.05 (18) |
| O11—Mo3—O8 | 83.12 (12) | C11—C10—S2 ⁱⁱⁱ | 123.05 (18) |
| O14—Mo3—O1 | 175.05 (11) | S2—C10—S2 ⁱⁱⁱ | 113.8 (4) |
| O15—Mo3—O1 | 78.15 (9) | C16—C15—S14 | 122.5 (3) |
| O20—Mo3—O1 | 77.32 (9) | C16—C15—S15 | 122.4 (3) |
| O11—Mo3—O1 | 75.90 (9) | S14—C15—S15 | 115.1 (3) |
| O8—Mo3—O1 | 75.10 (9) | C15—C16—S13 | 122.1 (4) |
| O18—Mo4—O13 | 105.18 (14) | C15—C16—S9 | 122.2 (4) |
| O18—Mo4—O8 ⁱⁱ | 102.97 (15) | S13—C16—S9 | 115.7 (3) |
| O13—Mo4—O8 ⁱⁱ | 92.59 (13) | C14—C13—S14 | 117.2 (4) |

supplementary materials

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| O18—Mo4—O15 | 103.98 (14) | C14—C13—H13 | 121.4 |
| O13—Mo4—O15 | 88.36 (13) | S14—C13—H13 | 121.4 |
| O8 ⁱⁱ —Mo4—O15 | 151.77 (12) | C12 ⁱⁱⁱ —C12—S18 | 117.92 (15) |
| O18—Mo4—O10 | 101.72 (13) | C12 ⁱⁱⁱ —C12—H12 | 121 |
| O13—Mo4—O10 | 152.84 (12) | S18—C12—H12 | 121 |
| O8 ⁱⁱ —Mo4—O10 | 84.97 (12) | C13—C14—S15 | 117.2 (4) |
| O15—Mo4—O10 | 81.52 (12) | C13—C14—H14 | 121.4 |
| O18—Mo4—O1 | 176.53 (11) | S15—C14—H14 | 121.4 |
| O13—Mo4—O1 | 78.29 (9) | C9 ⁱⁱⁱ —C9—S2 | 117.77 (15) |
| O8 ⁱⁱ —Mo4—O1 | 76.83 (9) | C9 ⁱⁱⁱ —C9—H9 | 121.1 |
| O15—Mo4—O1 | 75.75 (8) | S2—C9—H9 | 121.1 |
| O10—Mo4—O1 | 74.81 (8) | C4—C22—S3 | 122.2 (3) |
| O19—Mo6—O10 | 105.57 (14) | C4—C22—S6 | 122.1 (3) |
| O19—Mo6—O11 | 104.81 (14) | S3—C22—S6 | 115.7 (3) |
| O10—Mo6—O11 | 91.35 (13) | C18—C17—S13 | 117.3 (4) |
| O19—Mo6—O20 ⁱⁱ | 101.65 (14) | C18—C17—H17 | 121.3 |
| O10—Mo6—O20 ⁱⁱ | 88.78 (13) | S13—C17—H17 | 121.3 |
| O11—Mo6—O20 ⁱⁱ | 152.48 (12) | C17—C18—S9 | 117.0 (4) |
| O19—Mo6—O13 ⁱⁱ | 100.43 (14) | C17—C18—H18 | 121.5 |
| O10—Mo6—O13 ⁱⁱ | 153.81 (12) | S9—C18—H18 | 121.5 |
| O11—Mo6—O13 ⁱⁱ | 84.97 (13) | C8—C21—S12 | 121.4 (5) |
| O20 ⁱⁱ —Mo6—O13 ⁱⁱ | 82.91 (13) | C8—C21—S10 | 123.7 (5) |
| O19—Mo6—O1 | 175.58 (11) | S12—C21—S10 | 115.0 (4) |
| O10—Mo6—O1 | 78.25 (9) | C20 ⁱⁱⁱ —C20—S4 | 117.88 (15) |
| O11—Mo6—O1 | 77.08 (8) | C20 ⁱⁱⁱ —C20—H20 | 121.1 |
| O20 ⁱⁱ —Mo6—O1 | 76.01 (9) | S4—C20—H20 | 121.1 |
| O13 ⁱⁱ —Mo6—O1 | 75.65 (8) | C19 ⁱⁱⁱ —C19—S1 | 117.84 (15) |
| C20—S4—C2 | 94.7 (2) | C19 ⁱⁱⁱ —C19—H19 | 121.1 |
| C19—S1—C1 | 94.0 (2) | S1—C19—H19 | 121.1 |
| C9—S2—C10 | 94.6 (2) | C23—C24—S6 | 117.3 (4) |
| C23—S3—C22 | 94.9 (2) | C23—C24—H24 | 121.4 |
| C6—S5—C4 | 94.8 (2) | S6—C24—H24 | 121.4 |
| C24—S6—C22 | 94.7 (2) | C24—C23—S3 | 117.4 (4) |
| C4—S7—C5 | 94.9 (2) | C24—C23—H23 | 121.3 |
| C3—S8—C8 | 94.6 (3) | S3—C23—H23 | 121.3 |
| C18—S9—C16 | 95.0 (2) | C25—C26—S12 | 117.1 (5) |
| C25—S10—C21 | 94.1 (3) | C25—C26—H26 | 121.4 |
| C7—S11—C8 | 94.5 (3) | S12—C26—H26 | 121.4 |
| C21—S12—C26 | 94.7 (3) | C26—C25—S10 | 119.1 (5) |
| C16—S13—C17 | 95.0 (2) | C26—C25—H25 | 120.4 |
| C15—S14—C13 | 95.3 (2) | S10—C25—H25 | 120.4 |
| C14—S15—C15 | 95.2 (2) | C29—S16—C27 | 94.5 (2) |
| C12—S18—C11 | 94.8 (2) | C28—S27—C27 | 95.1 (2) |
| Mo6—O1—Mo6 ⁱⁱ | 180.000 (19) | C27 ^{iv} —C27—S27 | 123.3 (5) |
| Mo6—O1—Mo3 ⁱⁱ | 89.861 (13) | C27 ^{iv} —C27—S16 | 122.4 (5) |

| | | | |
|---|---------------|---------------|------------|
| Mo6 ⁱⁱ —O1—Mo3 ⁱⁱ | 90.139 (13) | S27—C27—S16 | 114.3 (2) |
| Mo6—O1—Mo3 | 90.139 (13) | C28—C29—S16 | 118.5 (4) |
| Mo6 ⁱⁱ —O1—Mo3 | 89.861 (13) | C28—C29—H29 | 120.8 |
| Mo3 ⁱⁱ —O1—Mo3 | 180.0000 (10) | S16—C29—H29 | 120.8 |
| Mo6—O1—Mo4 | 90.402 (14) | C29—C28—S27 | 117.5 (4) |
| Mo6 ⁱⁱ —O1—Mo4 | 89.598 (13) | C29—C28—H28 | 121.3 |
| Mo3 ⁱⁱ —O1—Mo4 | 90.504 (14) | S27—C28—H28 | 121.3 |
| Mo3—O1—Mo4 | 89.496 (14) | N1—C30—C31 | 178.7 (11) |
| Mo6—O1—Mo4 ⁱⁱ | 89.598 (13) | C30—C31—H31A | 109.5 |
| Mo6 ⁱⁱ —O1—Mo4 ⁱⁱ | 90.402 (14) | C30—C31—H31B | 109.5 |
| Mo3 ⁱⁱ —O1—Mo4 ⁱⁱ | 89.496 (14) | H31A—C31—H31B | 109.5 |
| Mo3—O1—Mo4 ⁱⁱ | 90.504 (14) | C30—C31—H31C | 109.5 |
| Mo4—O1—Mo4 ⁱⁱ | 180.0000 (10) | H31A—C31—H31C | 109.5 |
| Mo2—O2—Mo2 ⁱ | 180 | H31B—C31—H31C | 109.5 |

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

Hydrogen-bond geometry ($\text{\AA}, ^\circ$)

| $D-H\cdots A$ | $D-H$ | $H\cdots A$ | $D\cdots A$ | $D-H\cdots A$ |
|--------------------------------------|-------|-------------|-------------|---------------|
| C5—H5 \cdots O11 ^v | 0.93 | 2.28 | 3.192 (6) | 167 |
| C7—H7 \cdots O19 ^{vi} | 0.93 | 2.47 | 3.251 (7) | 141 |
| C7—H7 \cdots O19 ^{vii} | 0.93 | 2.47 | 3.251 (7) | 141 |
| C9—H9 \cdots O12 | 0.93 | 2.54 | 3.388 (5) | 151 |
| C13—H13 \cdots O17 ^{viii} | 0.93 | 2.41 | 3.239 (6) | 149 |
| C14—H14 \cdots O19 ^{ix} | 0.93 | 2.59 | 3.175 (5) | 121 |
| C18—H18 \cdots O18 ^x | 0.93 | 2.41 | 3.145 (5) | 136 |
| C19—H19 \cdots O10 ^{xi} | 0.93 | 2.41 | 3.298 (5) | 160 |
| C20—H20 \cdots O12 | 0.93 | 2.54 | 3.437 (5) | 162 |
| C24—H24 \cdots O8 ^{xii} | 0.93 | 2.25 | 3.075 (6) | 148 |
| C28—H28 \cdots O4 ^{xii} | 0.93 | 2.53 | 3.384 (6) | 152 |
| C29—H29 \cdots O5 ^{xii} | 0.93 | 2.52 | 3.256 (6) | 137 |
| C29—H29 \cdots O6 ^{xii} | 0.93 | 2.55 | 3.284 (6) | 136 |

Symmetry codes: (v) $x+1, -y+1/2, z$; (vi) $-x, y+1/2, -z+1$; (vii) $-x, -y+1, -z+1$; (viii) $x, -y+1/2, z-1$; (ix) $x+1, y, z-1$; (x) $x, y, z-1$; (xi) $x+1, -y+1/2, z-1$; (xii) $-x+1, y+1/2, -z+1$.

Fig. 1

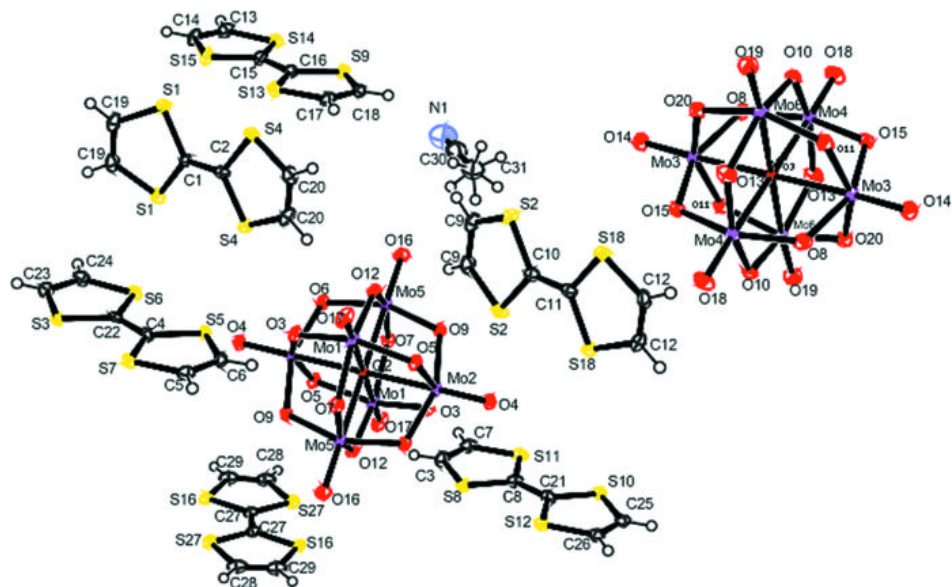


Fig. 2

