

Molecular structure of *fac*-[Mo(CO)₃(DMSO)₃]

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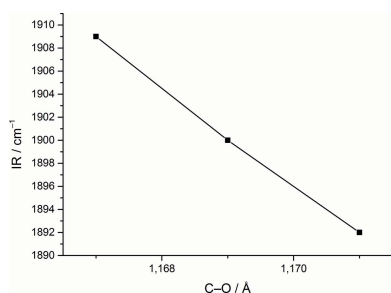
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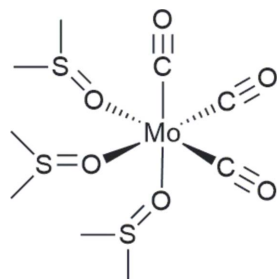
The title compound, tricarbonyltris(dimethyl sulfoxide)molybdenum, [Mo(C₂H₆OS)₃(CO)₃] or *fac*-[Mo(CO)₃(DMSO)₃], crystallizes in the triclinic space group $P\bar{1}$ with two molecules in the unit cell. The geometry around the central molybdenum is slightly distorted octahedral and the *facial* isomer is found exclusively. The packing within the crystal is stabilized by three-dimensional non-classical intermolecular hydrogen-bonding contacts between individual methyl substituents of dimethyl sulfoxide and the oxygen atoms of either another dimethyl sulfoxide or a carbonyl ligand on adjacent complex molecules. The observed bond lengths in the carbonyl ligands and between carbonyl carbon atoms and molybdenum are correlated to the observed FT-IR bands for the carbonyl stretches and compared to respective metrical parameters of related complexes.

1. Chemical context

[Mo(CO)₆] is a commercially available starting material that is easy to handle. It is, however, not particularly reactive. In order to facilitate quicker and/or more complete reactions, it can be activated by replacing some of the CO ligands by solvent ligands. This is often done with acetonitrile, which results in *fac*-[Mo(CH₃CN)₃(CO)₃] complexes. Other examples comprise, for instance, [Mo(CO)₅(THF)] (THF = tetrahydrofuran), [Mo(CO)₄(nbd)] (nbd = norbornadiene) and *fac*-[Mo(CO)₃(DMF)₃] (DMF = dimethyl formamide) (Wieland & van Eldik, 1991; Mukerjee *et al.* 1988; Villanueva *et al.*, 1996). Depending on the co-ligand, the stability and reactivity of the resultant complex can be fine-tuned. It was, for example, previously emphasized that the pyridine complexes surpass acetonitrile complexes in reactivity (Kuhl *et al.*, 2000). In cases where the carbonyl ligands are supposed to be retained, stronger carbonyl–metal interactions and very weak metal–co-ligand interactions are preferred. In cases where the carbonyl ligands shall also be replaced, the opposite is true. The grade of activation is reflected in the C≡O bond lengths and the Mo–C_{carbonyl} bond lengths. For the former, infrared spectroscopy provides an easy way to probe the strength of the bond between carbon and oxygen with stretching vibration bands in a normally not populated region of the infrared wavenumber range (around 2000 cm⁻¹). This bond strength depends directly on the metal–carbon interaction as the stronger the metal carbon bond, the weaker the carbon–oxygen bond becomes (Elschenbroich, 2003) and these again depend on the strengths of the *trans*-located co-ligand-to-metal interactions. A short and strong C≡O bond is, hence, indicative of only weak carbonyl metal–ligand interactions and concomitantly impaired complex stability. FT-IR



therefore constitutes a particularly helpful assessment tool, in particular in cases where no crystal structure is available. On the other hand, it is also quite useful to combine both methods, if possible, for validation purposes and adding reliability to future spectroscopic evaluation of related species. In the course of synthesizing molybdenum–carbonyl complexes as starting materials and in a search for the optimum balance between reactivity and stability, various solvent complexes were tested in our group. During these experiments, DMSO was considered beneficial and the title complex *fac*-[Mo(CO)₃(DMSO)₃] was prepared and crystallized. This complex was first reported in the literature in 1959 (Hieber *et al.*, 1959), but its crystal structure remained, apparently, elusive to date. Notably, it also appears that since then the complex has never been mentioned again. As very nice and suitable crystals of the title compound were obtained, an X-ray diffraction structural analysis was carried out. The respective high-quality results, along with the signatory carbonyl FT–IR stretch bands are presented here.



2. Structural commentary

fac-[Mo(CO)₃(DMSO)₃] crystallizes in the triclinic space group $P\bar{1}$. The asymmetric unit represents the entire molecule (Fig. 1) while $Z = 2$. The central zero-valent molybdenum is coordinated in a *facial* fashion by three neutral dimethyl sulfoxide and three neutral carbonyl ligands, *i.e.* it is embraced by a C₃O₃ donor set. The coordination geometry of the complex is essentially octahedral, showing an almost perfect Bailar twist angle (Wentworth, 1972) of 59.08°. The average *cis*-donor–Mo–donor angle between the three coordinated DMSO molecules is, at approximately 79°, slightly more acute compared to that of the carbonyl ligands, at approximately 84° despite dimethyl sulfoxide being considerably more bulky. The three *trans* angles across molybdenum range from 173.76 (16) to 178.08 (18)°, indicating a slight distortion from ideal octahedral geometry.

The structures of the title compound and those of chemically very closely related *fac*-[Mo(CH₃CN)₃(CO)₃] (refcode: IZUQAV; Antonini *et al.*, 2004) and *fac*-[Mo(CO)₃(DMF)₃] (refcode: WAJWIN; Pasquali *et al.*, 1992) are, as expected, quite similar in the immediate coordination sphere surrounding molybdenum, which is also evident from the overlaid molecular structures (Fig. 2). Still, some specifics in the metrical parameter details in the individual species are quite notable.

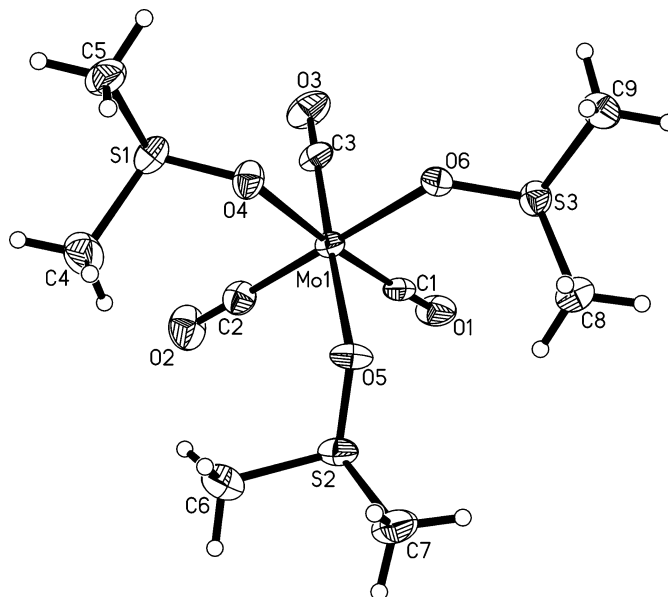


Figure 1
Molecular structure of *fac*-[Mo(CO)₃(DMSO)₃] with ellipsoids at the 50% level.

In particular the C–O and Mo–C distances are interesting when compared to those of *fac*-[Mo(CH₃CN)₃(CO)₃] and *fac*-[Mo(CO)₃(DMF)₃]. Whereas the average C–O bond length in *fac*-[Mo(CO)₃(DMSO)₃] is 1.170 (6) Å and the average Mo–C distance is 1.911 (5) Å, in *fac*-[Mo(CH₃CN)₃(CO)₃] the average C–O and Mo–C distances are 1.167 and 1.923 Å, respectively. In *fac*-[Mo(CO)₃(DMF)₃], these values are 1.172 Å (C–O) and 1.909 Å (Mo–C). The complexes with the O-donor solvent coordination exhibit longer C–O and shorter Mo–C distances, which is indicative of stronger bonds between carbonyl and molybdenum than in the case of the N-donor solvent. At the same time, this suggests that the share of electron density between molybdenum and coordinated solvent is decreased in the case of O-donor solvents and increased in the case of the N-donor solvent. This is also reflected in the reported IR data. In the case of acetonitrile,

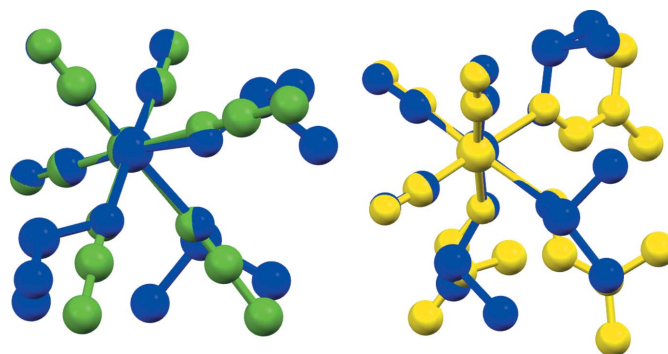
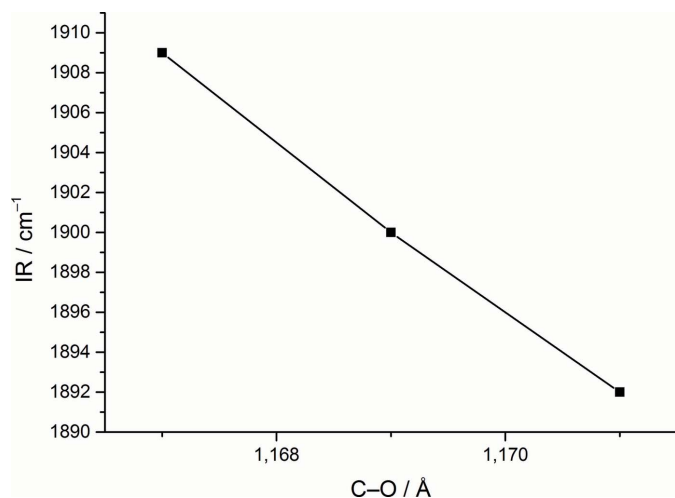


Figure 2
Structural overlay between *fac*-[Mo(CO)₃(DMSO)₃] in blue with *fac*-[Mo(CH₃CN)₃(CO)₃] (refcode: IZUQAV; Antonini *et al.*, 2004) in green (left) and with *fac*-[Mo(CO)₃(DMF)₃] (refcode: WAJWIN; Pasquali *et al.*, 1992) in yellow (right) generated with Mercury 2020.3.0 (Macrae *et al.*, 2020).

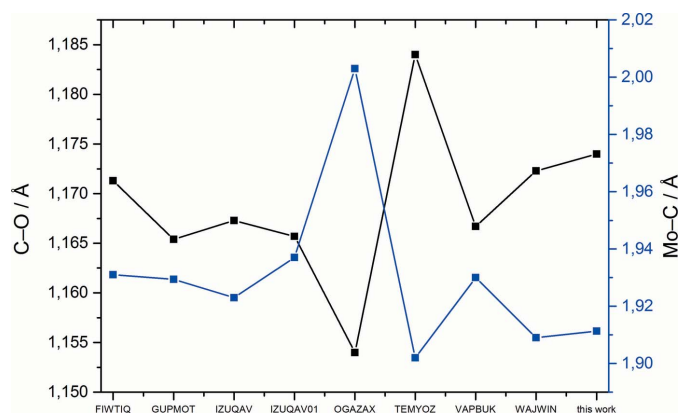

Figure 3

Correlation between the highest energy infrared band for the C–O stretching vibration and the shortest of the observed C–O bonds in the molecular structures of *fac*-[Mo(CO)₃(DMSO)₃], *fac*-[Mo(CH₃CN)₃(CO)₃] (refcode: IZUQAV; Antonini *et al.*, 2004) and *fac*-[Mo(CO)₃(DMF)₃] (refcode: WAJWIN; Pasquali *et al.*, 1992).

two C–O bands are reported, and for the other two complexes, three. In perfectly octahedral symmetry, only two bands would be expected (Elschenbroich, 2003). The presence of three bands therefore indicates a distortion of the complex from perfect symmetry. The comparison of the highest energy infrared bands with the shortest observed C–O bond lengths in these three species reveals a perfect correlation (Fig. 3).

The O-donor solvents, therefore, appear to be slightly better suited for those reactions in which the carbonyl ligands are supposed to be retained on the metal, while the co-ligands are more labile. In the case of *fac*-[Mo(CO)₃(DMSO)₃], it was observed that the complex is very sensitive to moisture, for instance, which supports the anticipated facile exchange of the coordinated solvents.

When larger co-ligands are also included in the C–O and Mo–C bond-length analysis, these observations are generally confirmed (Fig. 4). Only the structures with methyl-pyridine


Figure 4

Average C–O and Mo–C distances in the molecular structures of neutral bisleptic tricarbonyl molybdenum(0) complexes with oxygen or nitrogen donor co-ligands.

Table 1

Hydrogen-bond geometry (Å, °).

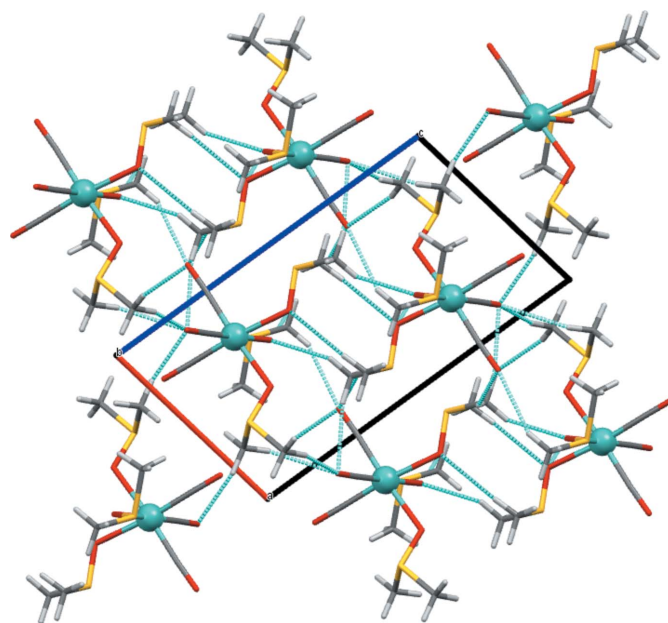
<i>D</i> –H... <i>A</i>	<i>D</i> –H	H... <i>A</i>	<i>D</i> ... <i>A</i>	<i>D</i> –H... <i>A</i>
C5–H5C...O1 ⁱ	0.98	2.52	3.461 (7)	161
C4–H4C...O3 ⁱⁱ	0.98	2.60	3.503 (8)	154
C7–H7C...O1 ⁱⁱ	0.98	2.51	3.419 (7)	154
C9–H9C...O5 ⁱⁱⁱ	0.98	2.45	3.239 (7)	138
C4–H4B...O3 ^{iv}	0.98	2.38	3.334 (8)	165
C9–H9A...O1 ^v	0.98	2.59	3.328 (7)	132
C9–H9B...O3 ^v	0.98	2.52	3.469 (7)	163
C7–H7B...O6 ^{vi}	0.98	2.55	3.398 (6)	144

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

(refcode: TEMYOZ; Schut *et al.*, 1996) and pyrazole (refcode: OGAZAX; Ardizzoia *et al.*, 2002) exhibit somewhat extreme values with a particularly short and strong C–O bond in the latter and an exceptionally long and weak C–O bond in the former, which even surpasses the effect of the O-donor co-ligands. The other considered structures comprise a second one with acetonitrile (refcode: IZUQAV01; Sala *et al.*, 2018), one with propionitrile (refcode: FIWTIQ; Hering *et al.*, 2014), one with thiophene-acetonitrile (refcode: VAPBUK; Baker *et al.*, 2003), and one with pyridine (refcode: GUPMOT, Kuhl *et al.*, 2000).

3. Supramolecular features

The unit cell is relatively small and contains only two molecules. Non-classical hydrogen-bonding contacts stabilize the crystal packing (Table 1). All hydrogen atoms are part of


Figure 5

Crystal packing viewed along the *b* axis with hydrogen-bonding contacts shown as light blue dashed lines, generated with Mercury 2020.3.0 (Macrae *et al.*, 2020).

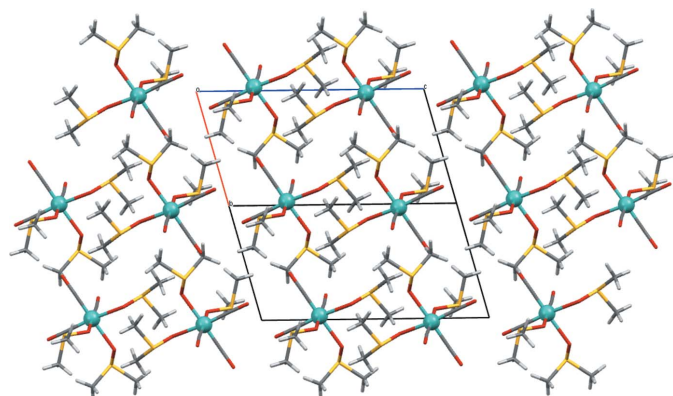


Figure 6
Crystal packing viewed along the *ab* diagonal with the *c* axis in horizontal alignment along which the orientations of the complex molecules alternate, generated with *Mercury* 2020.3.0 (Macrae *et al.*, 2020).

methyl groups and these are thereby the only available donors. Oxygen atoms of DMSO (O5, O6) and of carbonyl ligands (O1, O3) serve as hydrogen-bonding acceptors. Hydrogen bonding within the unit cell involves exclusively DMSO. Hydrogen bonding between unit cells is exclusively between DMSO and carbonyl oxygen atoms (Fig. 5). The orientations of the molecules strictly alternate in the *c*-axis direction, as is evident when viewed along the *ab* diagonal (Fig. 6) while they are identical to those of their neighbours in the *a*- and *b*-axis directions.

4. Database survey

A search of the CSD database with *ConQuest* (Bruno *et al.*, 2002) for bisleptic triscarbonyl molybdenum(0) complexes and three neutral co-ligands with N, O, S or P donor atoms results, in addition to the eight known molecular structures with oxygen or nitrogen donors, which are already discussed in the structural commentary, only in species with phosphorous donor atoms. These are structures with the refcodes DUSHAA (Tarassoli *et al.*, 1986), DUSHAA10 (Chen *et al.*, 1986), JEWPII (Nakazawa *et al.*, 2006), KETQIJ (Campbell *et al.*, 1999), KOBISIE (Fukumoto & Nakazawa, 2008), LALSEW (Willey *et al.*, 1993), NIPTAH and NIPTTEL (Alyea *et al.*, 1997), NITFOM (Tallis *et al.*, 2008), SANMOJ (Bent *et al.*, 1989), SANMOJ10 (Bent *et al.*, 1990), TAWNIO (Edwards *et al.*, 1996), TIRYUP (Thirupathi *et al.*, 2007), YAZSAT and YAZSIB (Kang *et al.*, 1994), YAZSAT10 and YAZSIB10 (Hockless *et al.*, 1996), YEPWAR (Fischer *et al.*, 1994), and ZEXCIO (Alyea *et al.*, 1995). The average C—O bond lengths in the molecular structures with phosphorous donor atoms range from 1.141 Å (KOBISIE) to 1.228 Å (DUSHAA). This means that both shorter as well as longer bonds are observed in the P-donor species than in the O- and N-donor complexes. In total only 27 examples of complexes are found in the database that meet the search criteria. Considering the simplicity of the complexes this is a surprisingly small number.

Table 2
Experimental details.

Crystal data	
Chemical formula	[Mo(C ₂ H ₆ OS) ₃ (CO) ₃]
<i>M_r</i>	414.35
Crystal system, space group	Triclinic, <i>P</i> $\bar{1}$
Temperature (K)	170
<i>a</i> , <i>b</i> , <i>c</i> (Å)	8.2027 (16), 8.4059 (17), 13.465 (3)
α , β , γ (°)	78.58 (3), 75.69 (3), 63.94 (3)
<i>V</i> (Å ³)	803.8 (4)
<i>Z</i>	2
Radiation type	Mo <i>K</i> α
μ (mm ⁻¹)	1.22
Crystal size (mm)	0.27 × 0.09 × 0.04
Data collection	
Diffractometer	Stoe IPDS2T
Absorption correction	Numerical face indexed (<i>X-RED32</i> and <i>X-SHAPE</i> ; Stoe & Cie, 2010)
<i>T_{min}</i> , <i>T_{max}</i>	0.909, 0.989
No. of measured, independent and observed [<i>I</i> > 2 σ (<i>I</i>)] reflections	8903, 4417, 3408
<i>R_{int}</i>	0.054
(<i>sin</i> θ / λ) _{max} (Å ⁻¹)	0.693
Refinement	
<i>R</i> [<i>F</i> ² > 2 σ (<i>F</i> ²)], <i>wR</i> (<i>F</i> ²), <i>S</i>	0.053, 0.135, 1.07
No. of reflections	4417
No. of parameters	178
H-atom treatment	H-atom parameters constrained
$\Delta\rho_{max}$, $\Delta\rho_{min}$ (e Å ⁻³)	1.10, -2.32

Computer programs: *X-AREA* (Stoe & Cie, 2016), *SHELXT2018* (Sheldrick, 2015a), *SHELXL2018/3* (Sheldrick, 2015b) and *XP* (Sheldrick, 2008), *CIFTAB* (Sheldrick, 2008).

5. Synthesis and crystallization

A tempered reaction vessel (293 K) was charged with [Mo(CO)₆] (165 mg, 0.625 mmol, 1 eq.) and the atmosphere was replaced by argon. 10 ml of absolute tetrahydrofuran (THF) and 0.2 ml of absolute dimethyl sulfoxide (DMSO, 2.82 mmol, 4.5 eq.) were added and the reaction vessel was irradiated for 2 h with HPM13 and HPA1200 halogen lamps as in previously described activation procedures (Elvers *et al.*, 2019). The resulting yellow solution was transferred anaerobically into a Schlenk flask and dried *in vacuo*. The golden-yellow solid precipitate was re-dissolved in THF and layered with *n*-hexane. Light-yellow, prismatic crystals of the title compound formed after three days of slow diffusion. Yield: 66.9% (160.7 mg, 0.418 mmol). IR (as KBr pellet given in cm⁻¹): 2260 (*w*, *br*); 1890 (*s*); 1750 (*s*); 1724 (*s*); 1308 (*sh*); 1246 (*s*); 1153 (*s*); 1020 (*sh*); 978 (*s*); 824 (*s*); 760 (*s*) (*w* = weak/*s* = strong/*sh* = shoulder/*br* = broad) .

6. Refinement

Crystal data, data collection and structure refinement details are summarized in Table 2. All hydrogen atoms belong to methyl substituents. They were attached to their parent atom in calculated positions based on the presence of electron density (HFIX 137) and treated as riding with *U*_{iso}(H) = 1.5 *U*_{eq}(C). One reflection was omitted from the refinement as a

clear outlier. *WinGX* was used as GUI for solving and refining the structure (Farrugia, 2012).

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supporting information

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Computing details

Data collection: *X-AREA* (Stoe & Cie, 2016); cell refinement: *X-AREA* (Stoe & Cie, 2016); data reduction: *X-AREA* (Stoe & Cie, 2016); program(s) used to solve structure: *SHELXT2018* (Sheldrick, 2015a); program(s) used to refine structure: *SHELXL2018/3* (Sheldrick, 2015b); molecular graphics: *XP* (Sheldrick, 2008); software used to prepare material for publication: *CIFTAB* (Sheldrick, 2008).

tricarbonyltris(dimethyl sulfoxide)molybdenum

Crystal data

[Mo(C₂H₆OS)₃(CO)₃]

$M_r = 414.35$

Triclinic, $P\bar{1}$

$a = 8.2027$ (16) Å

$b = 8.4059$ (17) Å

$c = 13.465$ (3) Å

$\alpha = 78.58$ (3)°

$\beta = 75.69$ (3)°

$\gamma = 63.94$ (3)°

$V = 803.8$ (4) Å³

$Z = 2$

$F(000) = 420$

$D_x = 1.712$ Mg m⁻³

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

Cell parameters from 8904 reflections

$\theta = 6.5$ – 59.0 °

$\mu = 1.22$ mm⁻¹

$T = 170$ K

Needle, yellow

$0.27 \times 0.09 \times 0.04$ mm

Data collection

Stoe IPDS2T

diffractometer

Radiation source: fine-focus sealed tube

Detector resolution: 6.67 pixels mm⁻¹

ω scans

Absorption correction: numerical

face indexed (X-Red32 and X-Shape; Stoe & Cie, 2010)

$T_{\min} = 0.909$, $T_{\max} = 0.989$

8903 measured reflections

4417 independent reflections

3408 reflections with $I > 2\sigma(I)$

$R_{\text{int}} = 0.054$

$\theta_{\max} = 29.5$ °, $\theta_{\min} = 3.3$ °

$h = -11 \rightarrow 11$

$k = -11 \rightarrow 11$

$l = -16 \rightarrow 18$

Refinement

Refinement on F^2

Least-squares matrix: full

$R[F^2 > 2\sigma(F^2)] = 0.053$

$wR(F^2) = 0.135$

$S = 1.07$

4417 reflections

178 parameters

0 restraints

Primary atom site location: dual

Secondary atom site location: difference Fourier map

Hydrogen site location: inferred from neighbouring sites

H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.0653P)^2 + 1.7985P]$

where $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{\max} < 0.001$

$\Delta\rho_{\max} = 1.10$ e Å⁻³

$\Delta\rho_{\min} = -2.32$ e Å⁻³

Special details

Geometry. All esds (except the esd in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell esds are taken into account individually in the estimation of esds in distances, angles and torsion angles; correlations between esds in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell esds is used for estimating esds 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>	$U_{\text{iso}}^*/U_{\text{eq}}$
Mo1	0.26439 (5)	0.70392 (5)	0.25843 (3)	0.01937 (12)
S1	0.64463 (15)	0.46716 (16)	0.11748 (9)	0.0276 (3)
S2	0.31315 (15)	1.05860 (15)	0.28585 (10)	0.0267 (2)
S3	0.16962 (15)	0.60278 (16)	0.50846 (9)	0.0259 (2)
O1	−0.1521 (5)	0.9399 (5)	0.3125 (3)	0.0366 (9)
O2	0.2294 (6)	0.9145 (6)	0.0427 (3)	0.0437 (10)
O3	0.1020 (5)	0.4935 (6)	0.1823 (3)	0.0425 (10)
O4	0.5662 (4)	0.5143 (5)	0.2275 (3)	0.0261 (7)
O5	0.3793 (5)	0.8603 (4)	0.3182 (3)	0.0276 (7)
O6	0.3177 (4)	0.5503 (4)	0.4125 (2)	0.0241 (7)
C1	0.0078 (6)	0.8542 (6)	0.2948 (4)	0.0244 (9)
C2	0.2447 (6)	0.8356 (6)	0.1249 (4)	0.0277 (10)
C3	0.1697 (6)	0.5673 (6)	0.2123 (4)	0.0269 (9)
C4	0.7296 (8)	0.6302 (8)	0.0556 (5)	0.0441 (14)
H4A	0.625940	0.747086	0.050873	0.066*
H4B	0.797261	0.599555	−0.013743	0.066*
H4C	0.812511	0.633876	0.095684	0.066*
C5	0.8601 (7)	0.2841 (7)	0.1252 (4)	0.0333 (11)
H5A	0.935178	0.317406	0.156388	0.050*
H5B	0.924538	0.250564	0.055791	0.050*
H5C	0.839851	0.183013	0.167560	0.050*
C6	0.4774 (8)	1.0822 (8)	0.1761 (5)	0.0404 (13)
H6A	0.602434	1.005903	0.189192	0.061*
H6B	0.461592	1.206568	0.162583	0.061*
H6C	0.458693	1.046979	0.116050	0.061*
C7	0.3772 (8)	1.1383 (7)	0.3759 (5)	0.0384 (12)
H7A	0.299763	1.132681	0.443629	0.058*
H7B	0.360358	1.261909	0.353195	0.058*
H7C	0.507049	1.064490	0.380701	0.058*
C8	0.2159 (9)	0.7503 (8)	0.5644 (5)	0.0450 (15)
H8A	0.344626	0.694301	0.574038	0.068*
H8B	0.133983	0.776499	0.631347	0.068*
H8C	0.194380	0.861157	0.518762	0.068*
C9	0.2439 (8)	0.4168 (7)	0.6010 (4)	0.0360 (11)
H9A	0.234863	0.315606	0.580728	0.054*
H9B	0.165563	0.446406	0.668472	0.054*
H9C	0.372287	0.385368	0.605036	0.054*

Atomic displacement parameters (\AA^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Mo1	0.01869 (18)	0.01643 (18)	0.02248 (19)	-0.00617 (13)	-0.00398 (13)	-0.00316 (13)
S1	0.0218 (5)	0.0294 (6)	0.0292 (6)	-0.0054 (4)	-0.0042 (4)	-0.0106 (5)
S2	0.0237 (5)	0.0172 (5)	0.0394 (7)	-0.0073 (4)	-0.0082 (5)	-0.0026 (5)
S3	0.0216 (5)	0.0270 (6)	0.0252 (6)	-0.0072 (4)	-0.0028 (4)	-0.0029 (5)
O1	0.0227 (17)	0.0234 (17)	0.056 (2)	-0.0051 (13)	-0.0022 (16)	-0.0054 (17)
O2	0.044 (2)	0.047 (2)	0.030 (2)	-0.0096 (18)	-0.0119 (17)	0.0041 (18)
O3	0.040 (2)	0.043 (2)	0.055 (3)	-0.0181 (18)	-0.0106 (19)	-0.023 (2)
O4	0.0189 (14)	0.0297 (17)	0.0239 (16)	-0.0050 (12)	-0.0023 (12)	-0.0042 (13)
O5	0.0325 (17)	0.0176 (15)	0.0384 (19)	-0.0131 (13)	-0.0134 (14)	0.0002 (14)
O6	0.0269 (16)	0.0184 (15)	0.0235 (16)	-0.0079 (12)	-0.0030 (12)	-0.0001 (12)
C1	0.029 (2)	0.0158 (19)	0.030 (2)	-0.0111 (17)	-0.0045 (18)	-0.0030 (17)
C2	0.023 (2)	0.027 (2)	0.029 (2)	-0.0056 (18)	-0.0068 (18)	-0.0029 (19)
C3	0.024 (2)	0.023 (2)	0.034 (3)	-0.0080 (17)	-0.0023 (18)	-0.0117 (19)
C4	0.041 (3)	0.033 (3)	0.041 (3)	-0.008 (2)	0.000 (2)	0.007 (2)
C5	0.024 (2)	0.026 (2)	0.045 (3)	-0.0066 (19)	0.000 (2)	-0.011 (2)
C6	0.047 (3)	0.031 (3)	0.043 (3)	-0.021 (2)	-0.004 (3)	0.002 (2)
C7	0.041 (3)	0.031 (3)	0.049 (3)	-0.017 (2)	-0.005 (2)	-0.014 (2)
C8	0.069 (4)	0.034 (3)	0.035 (3)	-0.027 (3)	0.004 (3)	-0.012 (2)
C9	0.048 (3)	0.030 (3)	0.031 (3)	-0.019 (2)	-0.007 (2)	0.002 (2)

Geometric parameters (\AA , $^\circ$)

Mo1—C3	1.901 (5)	C4—H4A	0.9800
Mo1—C1	1.915 (5)	C4—H4B	0.9800
Mo1—C2	1.919 (5)	C4—H4C	0.9800
Mo1—O6	2.249 (3)	C5—H5A	0.9800
Mo1—O4	2.264 (3)	C5—H5B	0.9800
Mo1—O5	2.269 (3)	C5—H5C	0.9800
S1—O4	1.518 (3)	C6—H6A	0.9800
S1—C5	1.772 (5)	C6—H6B	0.9800
S1—C4	1.777 (6)	C6—H6C	0.9800
S2—O5	1.516 (3)	C7—H7A	0.9800
S2—C7	1.773 (6)	C7—H7B	0.9800
S2—C6	1.782 (6)	C7—H7C	0.9800
S3—O6	1.522 (3)	C8—H8A	0.9800
S3—C9	1.771 (6)	C8—H8B	0.9800
S3—C8	1.782 (6)	C8—H8C	0.9800
O1—C1	1.174 (6)	C9—H9A	0.9800
O2—C2	1.177 (6)	C9—H9B	0.9800
O3—C3	1.170 (6)	C9—H9C	0.9800
C3—Mo1—C1	82.54 (19)	S1—C4—H4C	109.5
C3—Mo1—C2	85.0 (2)	H4A—C4—H4C	109.5
C1—Mo1—C2	85.0 (2)	H4B—C4—H4C	109.5
C3—Mo1—O6	99.49 (18)	S1—C5—H5A	109.5

C1—Mo1—O6	99.79 (17)	S1—C5—H5B	109.5
C2—Mo1—O6	173.76 (16)	H5A—C5—H5B	109.5
C3—Mo1—O4	97.31 (16)	S1—C5—H5C	109.5
C1—Mo1—O4	175.59 (16)	H5A—C5—H5C	109.5
C2—Mo1—O4	99.35 (16)	H5B—C5—H5C	109.5
O6—Mo1—O4	75.88 (12)	S2—C6—H6A	109.5
C3—Mo1—O5	178.08 (18)	S2—C6—H6B	109.5
C1—Mo1—O5	97.90 (16)	H6A—C6—H6B	109.5
C2—Mo1—O5	96.89 (18)	S2—C6—H6C	109.5
O6—Mo1—O5	78.60 (12)	H6A—C6—H6C	109.5
O4—Mo1—O5	82.10 (13)	H6B—C6—H6C	109.5
O4—S1—C5	104.3 (2)	S2—C7—H7A	109.5
O4—S1—C4	104.6 (3)	S2—C7—H7B	109.5
C5—S1—C4	97.9 (3)	H7A—C7—H7B	109.5
O5—S2—C7	104.0 (2)	S2—C7—H7C	109.5
O5—S2—C6	105.7 (2)	H7A—C7—H7C	109.5
C7—S2—C6	98.1 (3)	H7B—C7—H7C	109.5
O6—S3—C9	104.2 (2)	S3—C8—H8A	109.5
O6—S3—C8	106.0 (2)	S3—C8—H8B	109.5
C9—S3—C8	97.2 (3)	H8A—C8—H8B	109.5
S1—O4—Mo1	115.64 (18)	S3—C8—H8C	109.5
S2—O5—Mo1	118.64 (19)	H8A—C8—H8C	109.5
S3—O6—Mo1	120.18 (18)	H8B—C8—H8C	109.5
O1—C1—Mo1	175.0 (4)	S3—C9—H9A	109.5
O2—C2—Mo1	178.2 (5)	S3—C9—H9B	109.5
O3—C3—Mo1	175.6 (4)	H9A—C9—H9B	109.5
S1—C4—H4A	109.5	S3—C9—H9C	109.5
S1—C4—H4B	109.5	H9A—C9—H9C	109.5
H4A—C4—H4B	109.5	H9B—C9—H9C	109.5
C5—S1—O4—Mo1	-167.4 (2)	C6—S2—O5—Mo1	-94.2 (3)
C4—S1—O4—Mo1	90.3 (3)	C9—S3—O6—Mo1	164.8 (2)
C7—S2—O5—Mo1	163.1 (2)	C8—S3—O6—Mo1	-93.3 (3)

Hydrogen-bond geometry (Å, °)

<i>D</i> —H... <i>A</i>	<i>D</i> —H	H... <i>A</i>	<i>D</i> ... <i>A</i>	<i>D</i> —H... <i>A</i>
C5—H5C...O1 ⁱ	0.98	2.52	3.461 (7)	161
C4—H4C...O3 ⁱⁱ	0.98	2.60	3.503 (8)	154
C7—H7C...O1 ⁱⁱ	0.98	2.51	3.419 (7)	154
C9—H9C...O5 ⁱⁱⁱ	0.98	2.45	3.239 (7)	138
C4—H4B...O3 ^{iv}	0.98	2.38	3.334 (8)	165
C9—H9A...O1 ^v	0.98	2.59	3.328 (7)	132
C9—H9B...O3 ^v	0.98	2.52	3.469 (7)	163
C7—H7B...O6 ^{vi}	0.98	2.55	3.398 (6)	144

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