



Synthesis and structure of two isomers of a molybdenum(II) 2-butyne complex stabilized by bioinspired *S,N*-bidentate ligands

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Received 29 November 2021

Accepted 21 February 2022

Edited by W. Lewis, University of Sydney, Australia

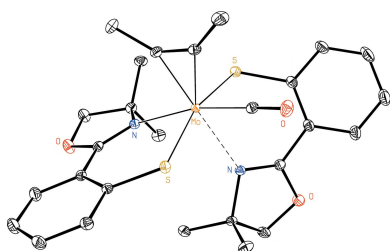
Keywords: isomer; 2-butyne complex; molybdenum(II); benzenethiol; crystal structure.**CCDC references:** 2153636; 2153635**Supporting information:** this article has supporting information at journals.iucr.org/c

The synthesis and structural determination of two isomers of the molybdenum(II) complex (η^2 -but-2-yne)carbonylbis[2-(4,4-dimethyl-4,5-dihydro-1,3-oxazol-2-yl)benzenethiolato- κ^2N,S]molybdenum(II), $[\text{Mo}(\text{C}_{11}\text{H}_{12}\text{NOS})_2(\text{C}_4\text{H}_6)(\text{CO})]$ or $\text{Mo}(\text{CO})(\text{C}_2\text{Me}_2)(\text{S-Phoz})_2$, are presented. The *N,N-cis-S,S-trans* isomer **1** shows quite different bond lengths to the metal atom [$\text{Mo}-\text{N} = 2.4715$ (10) versus 2.3404 (11) Å; $\text{Mo}-\text{S} = 2.4673$ (3) versus 2.3665 (3) Å]. In the *N,N-trans-S,S-cis* isomer **2**, which is isotopic with the corresponding W complex, the $\text{Mo}-\text{N}$ bond lengths [2.236 (2) and 2.203 (2) Å], as well as the $\text{Mo}-\text{S}$ bond lengths [2.5254 (8) and 2.5297 (8) Å], are almost the same.

1. Introduction

In order to explore the interaction of Mo and W centres with acetylene (C_2H_2), which is accepted as a substrate by the tungstoenzyme acetylene hydratase (Schink, 1985; Rosner & Schink, 1995), our group has focused on the synthesis of W^{II} and Mo^{II} complexes containing bioinspired *S,N*-bidentate ligands and their subsequent oxidation to the respective W^{IV} and Mo^{IV} complexes. Although *N*-donor ligands are not the closest structural mimics of the dithiolene ligands in the active site of acetylene hydratase (Seiffert *et al.*, 2007) and other members of the dimethyl sulfoxide (DMSO) reductase enzyme family (Seelmann *et al.*, 2020), the use of these ligands has resulted in the discovery of new reactivities at W centres (Vidovič *et al.*, 2019; Ehweiner *et al.*, 2021c), the isolation of a so-far-elusive Mo^{IV} C_2H_2 complex (Ehweiner *et al.*, 2021a) and a detailed comparison of W and Mo complexes with a variety of coordinated alkynes (Ehweiner *et al.*, 2021b). One of the early publications of our group in this research field focused on the reversible activation of C_2H_2 at a W^{IV} centre coordinated by two 2-(4,4-dimethyloxazolin-2-yl)thiophenolate (S-Phoz) ligands (Peschel *et al.*, 2015a). Thereafter, the reversible binding of C_2Me_2 and C_2Ph_2 (Peschel *et al.*, 2019) was investigated, with a particular focus on the flexibility of the S-Phoz ligand. The latter has also found application in Ni, Pd and Pt compounds (Peschel *et al.*, 2015b; Holzer *et al.*, 2018), as well as in Zn (Mugesh *et al.*, 1999) and Fe (Bottini *et al.*, 2010) complexes.

Herein we report an improved synthetic procedure for $\text{Mo}(\text{CO})_2(\text{S-Phoz})_2$ and the preparation and structural characterization of carbonyl(η^2 -1,2-dimethylethyne)[2-(4,4-dimethyloxazolin-2-yl)benzenethiolato- κ^2N,S]molybdenum(II), $\text{Mo}(\text{CO})(\text{C}_2\text{Me}_2)(\text{S-Phoz})_2$, which forms two isomers (**1** and **2**) in solution, as well as in the solid state (see Scheme 1). This behaviour is different from that observed for the W variant



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Table 1

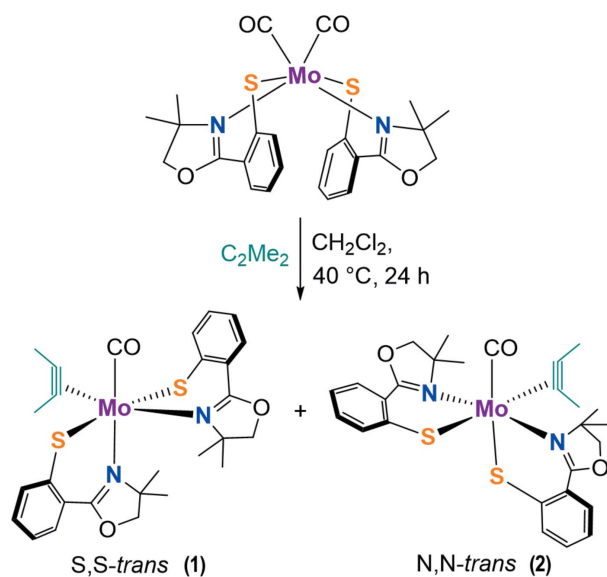
Experimental details.

For both structures: $[\text{Mo}(\text{C}_{11}\text{H}_{12}\text{NOS})_2(\text{C}_4\text{H}_6)(\text{CO})]$, $M_r = 590.59$, $Z = 4$. Experiments were carried out at 100 K with Mo $K\alpha$ radiation using a Bruker APEXII CCD diffractometer. Absorption was corrected for by multi-scan methods (SADABS; Bruker, 2013). Refinement was on 332 parameters. Only H-atom displacement parameters were refined.

	(1)	(2)
Crystal data		
Crystal system, space group	Monoclinic, $P2_1/n$	Monoclinic, $P2_1/c$
a, b, c (Å)	10.6159 (5), 8.9300 (4), 27.3801 (12)	9.1512 (4), 21.3515 (12), 13.1781 (7)
β (°)	96.189 (2)	98.483 (3)
V (Å ³)	2580.5 (2)	2546.7 (2)
μ (mm ⁻¹)	0.70	0.71
Crystal size (mm)	0.18 × 0.18 × 0.10	0.23 × 0.07 × 0.07
Data collection		
$T_{\text{min}}, T_{\text{max}}$	0.884, 1.000	0.776, 1.000
No. of measured, independent and observed [$I > 2\sigma(I)$] reflections	30042, 11363, 9549	22009, 7415, 5339
R_{int}	0.029	0.068
$(\sin \theta/\lambda)_{\text{max}}$ (Å ⁻¹)	0.807	0.703
Refinement		
$R[F^2 > 2\sigma(F^2)], wR(F^2), S$	0.028, 0.071, 1.04	0.043, 0.087, 1.01
No. of reflections	11363	7415
$\Delta\rho_{\text{max}}, \Delta\rho_{\text{min}}$ (e Å ⁻³)	0.72, -0.64	0.52, -0.83

Computer programs: APEX2 (Bruker, 2013), SAINT (Bruker, 2013), SHELXL97 (Sheldrick, 2008), SHELXL2014 (Sheldrick, 2015) and modified ORTEP (Johnson, 1965).

which crystallized solely as the *N,N-trans* isomer and showed the presence of a second isomer in solution only to a minor extent.



2. Experimental

Synthetic manipulations were performed under a nitrogen atmosphere using standard Schlenk and glove-box techniques. Solvents were purified *via* a Pure Solv Solvent Purification System. Chemicals were purchased from commercial sources and used without further purification. The precursor $\text{MoI}_2(\text{CO})_3(\text{NCMe})_2$ was synthesized according to a literature procedure (Baker *et al.*, 1986). For the synthesis of $\text{Mo}(\text{CO})_2(\text{S-Phoz})_2$, a slight modification of a published procedure was

used (Peschel *et al.*, 2013). ¹H NMR spectra were recorded on a Bruker Avance III 300 MHz spectrometer at ambient temperature and are referenced to residual protons in the solvent. The multiplicity of peaks is denoted as singlet (*s*), doublet (*d*), doublet of doublets (*dd*) or multiplet (*m*). NMR solvents were stored over molecular sieves. Solid-state IR spectra were measured on a Bruker ALPHA ATR-FT-IR spectrometer at a resolution of 2 cm⁻¹. The relative intensity of signals is declared as strong (*s*), medium (*m*) and weak (*w*). Electron impact mass spectroscopy (EI-MS) measurements were performed with an Agilent 5973 MSD mass spectrometer with a push rod.

2.1. Synthesis and crystallization

2.1.1. Preparation of $\text{Mo}(\text{CO})_2(\text{S-Phoz})_2$. A solution of Li(S-Phoz) (853 mg, 4.00 mmol) in MeCN (8 ml) was added dropwise to a solution of $\text{MoI}_2(\text{CO})_3(\text{NCMe})_2$ (1.03 g, 2.00 mmol) in MeCN (8 ml). The resulting blood-red solution was stirred for 2 h at 35 °C, whereupon the solvent was removed by evaporation. The residue was suspended in toluene (20 ml) and the resulting suspension was filtered through Celite. The blood-red filtrate was then evaporated to dryness. After repeated recrystallization from CH_2Cl_2 /heptane at -25 °C, $\text{Mo}(\text{CO})_2(\text{S-Phoz})_2$ (yield 790 mg, 70%) was obtained as dark red crystals. NMR and IR data are in agreement with previously published results (Peschel *et al.*, 2013).

2.1.2. Preparation of $\text{Mo}(\text{CO})_2(\text{C}_2\text{Me}_2)(\text{S-Phoz})_2$. $\text{Mo}(\text{CO})_2(\text{S-Phoz})_2$ (339 mg, 0.60 mmol) was dissolved in CH_2Cl_2 (20 ml), whereupon 2-butyne (0.38 ml, 4.80 mmol) was added to the solution at 0 °C under stirring. The cooling bath was removed and the solution was heated under reflux for 24 h. Evaporation of the solvent gave a dark brown powder. Single

crystals suitable for X-ray diffraction were obtained from CH_2Cl_2 /heptane solutions at -35°C . Crystals of both isomers (green plates of **1** and yellow needles of **2**) were obtained from the same batch. The product is very sensitive to air and should be stored in a glove-box.

2.1.3. Analytical data. ^1H NMR for **1** (CD_2Cl_2 , 300 MHz, *S,S-trans* isomer, 34%): δ 8.07 (*dd*, $J = 8.1, 1.1$ Hz, 1H, PhH), 7.78–7.72 (*m*, 3H, PhH), 7.35 (*dd*, $J = 7.8, 1.1$ Hz, 1H, PhH), 7.32–7.27 (*m*, 2H, PhH), 7.21–7.01 (*m*, 1H, PhH), 4.46 (*d*, $J = 8.2$ Hz, 1H, CH_2), 4.18 (*d*, $J = 8.1$ Hz, 1H, CH_2), 4.11 (*d*, $J = 8.3$ Hz, 1H, CH_2), 3.78 (*d*, $J = 8.2$ Hz, 1H, CH_2), 2.70 (*s*, 3H, $\text{C}\equiv\text{CCH}_3$), 2.55 (*s*, 3H, $\text{C}\equiv\text{CCH}_3$), 1.89 (*s*, 3H, CH_3), 1.81 (*s*, 3H, CH_3), 1.57 (*s*, 3H, CH_3), 1.44 (*s*, 3H, CH_3); ^1H NMR for **2** (CD_2Cl_2 , 300 MHz, *N,N-trans* isomer, 66%): δ 7.67–7.62 (*m*, 2H, PhH), 7.43 (*dd*, $J = 8.1, 1.4$ Hz, 1H, PhH), 7.21–7.01 (*m*, 4H, PhH), 6.90–6.84 (*m*, 1H, PhH), 4.11 (*d*, $J = 8.3$ Hz, 1H, CH_2), 3.93–3.90 (*m*, 3H, CH_2), 2.90 (*s*, 3H, $\text{C}\equiv\text{CCH}_3$), 2.46 (*s*, 3H, $\text{C}\equiv\text{CCH}_3$), 1.63 (*s*, 3H, CH_3), 1.34 (*s*, 3H, CH_3), 0.77 (*s*, 3H, CH_3), 0.58 (*s*, 3H, CH_3). IR (cm^{-1}): 2995 (*w*), 2962 (*w*), 2928 (*w*), 2916 (*w*), 2894 (*w*), 1898 (*s*, $\text{C}\equiv\text{O}$), 1856 (*m*, $\text{C}\equiv\text{O}$), 1590 (*s*), 1572 (*s*), 1539 (*m*, $\text{C}=\text{N}$), 1455 (*m*), 1357 (*m*), 1326 (*m*), 1280 (*m*), 1246 (*m*), 1208 (*m*), 1160 (*m*), 1139 (*m*), 1053 (*s*), 966 (*m*), 818 (*m*), 776 (*m*), 741 (*s*), 695 (*m*), 653 (*m*). EI-MS (70 eV) m/z : $[\text{M} - 2\text{CO} + \text{O}]^+$ 526.1.

2.2. Refinement

Crystal data, data collection, and structure refinement details are summarized in Table 1. The H atoms of the CH_2

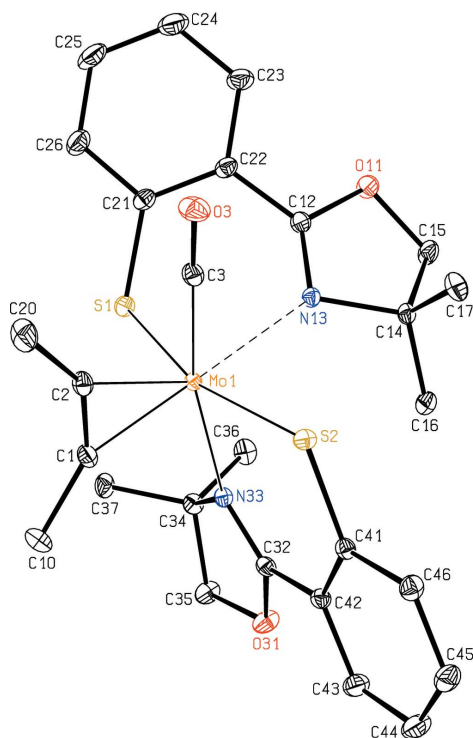


Figure 1
The molecular structure of isomer **1**. Displacement ellipsoids are drawn at the 50% probability level and H atoms have been omitted for clarity. The rather long Mo–N distance [$\text{Mo1}-\text{N13} = 2.4715(10)$ Å] is indicated by a dashed line.

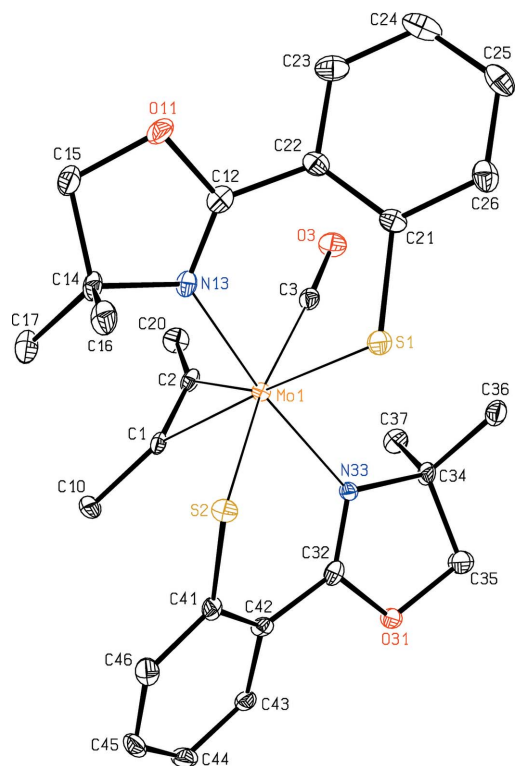


Figure 2
The molecular structure of isomer **2**. Displacement ellipsoids are drawn at the 50% probability level and H atoms have been omitted for clarity.

groups were placed at positions with approximately tetrahedral angles and C–H distances of 0.99 Å, and common isotropic displacement parameters were refined for the H atoms of the same group. The H atoms of the arene rings were placed at the external bisectors of the C–C–C angles at C–H distances of 0.95 Å, and common isotropic displacement parameters were refined for the H atoms of the same ring. The H atoms of the methyl groups were refined with common isotropic displacement parameters for the H atoms of the same group and idealized geometries with tetrahedral angles, enabling rotations around the C–C bonds, and with C–H distances of 0.98 Å.

3. Results and discussion

3.1. Crystal structure analysis

Isomers **1** and **2** crystallize without any solvent molecules in the monoclinic space groups $P2_1/n$ and $P2_1/c$, respectively, and both have one metal complex in the asymmetric unit. In *N,N-cis* isomer **1** (Fig. 1), the Mo–N distance of the oxazole ring *trans* to the butyne ligand [$\text{Mo1}-\text{N13} = 2.4715(10)$ Å] is much longer than that *trans* to the carbonyl ligand [$\text{Mo1}-\text{N33} = 2.3404(11)$ Å]. In *N,N-trans* isomer **2** (Fig. 2), these distances [$\text{Mo1}-\text{N13} = 2.236(2)$ Å and $\text{Mo1}-\text{N33} = 2.203(2)$ Å] are comparable to those observed in the dicarbonyl derivative [$2.2333(9)$ Å; Peschel *et al.*, 2013] or in the isotypic W compound [$\text{W1}-\text{N13} = 2.2153(16)$ Å and $\text{W1}-\text{N33} = 2.1862(16)$ Å; Peschel *et al.*, 2019]. In contrast to this,

Table 2

Selected geometric parameters (\AA , $^\circ$) for $M(\text{CO})(\text{C}_2\text{R}_2)(\text{S-Phoz})_2$ complexes.The labels C1 and C2 of the alkyne ligand were chosen such that the torsion angle $\text{C2}-\text{C1}-\text{M}-\text{C3}$ is approximately 0° . The selected ligand containing atoms S1 and N13 was that in which one of these atoms is *trans* to the alkyne ligand.

<i>M, R</i>	W, H ^a	W, CH ₃ ^b	Mo, CH ₃ ^c	Mo, CH ₃ ^c	W, Ph ^b	W, Ph ^b
	<i>N,N-trans</i>	<i>N,N-trans</i>	<i>N,N-trans</i>	<i>S,S-trans</i>	<i>S,S-trans</i>	<i>S,S-trans</i>
<i>M-C1</i>	2.0268 (17)	2.0210 (17)	2.024 (3)	2.0310 (12)	2.0510 (19)	2.036 (4)
<i>M-C2</i>	2.0548 (18)	2.0565 (17)	2.059 (3)	2.0664 (12)	2.078 (2)	2.057 (4)
<i>M-C3</i>	1.9623 (18)	1.9535 (19)	1.953 (3)	1.9417 (13)	1.949 (2)	1.966 (4)
<i>C3-O3</i>	1.160 (2)	1.164 (2)	1.157 (3)	1.1555 (16)	1.155 (3)	1.154 (5)
<i>M-N13</i>	2.2120 (14)	2.2153 (16)	2.236 (2)	2.4715 (10)	2.3087 (18)	2.350 (3)
<i>M-N33</i>	2.1987 (14)	2.1862 (16)	2.203 (2)	2.3404 (11)	2.2975 (17)	2.304 (4)
<i>M-S1</i>	2.5050 (4)	2.5232 (4)	2.5254 (8)	2.4673 (3)	2.4620 (5)	2.4741 (12)
<i>M-S2</i>	2.5067 (4)	2.5243 (4)	2.5297 (8)	2.3665 (3)	2.3698 (5)	2.3773 (11)
<i>C1-C2</i>	1.327 (3)	1.314 (3)	1.314 (4)	1.2965 (18)	1.309 (3)	1.305 (6)
<i>N13-M-N33</i>	169.58 (5)	167.56 (6)	168.04 (8)	92.41 (3)	83.29 (6)	86.47 (13)
<i>S1-M-S2</i>	78.869 (14)	78.972 (15)	79.54 (3)	162.979 (11)	175.564 (18)	169.56 (4)
<i>C1-M-N13</i>	92.88 (6)	97.14 (7)	96.97 (9)	173.53 (4)	165.94 (7)	169.64 (15)
<i>C2-M-N13</i>	93.66 (7)	94.92 (6)	94.67 (9)	146.80 (4)	150.09 (7)	148.68 (15)
<i>C3-M-N33</i>	94.24 (6)	94.52 (7)	94.51 (9)	168.19 (4)	159.92 (8)	164.04 (15)
<i>C1-M-S1</i>	164.79 (6)	164.06 (5)	163.76 (8)	97.54 (3)	85.61 (5)	91.62 (13)
<i>C2-M-S1</i>	153.79 (6)	156.79 (5)	156.87 (8)	96.29 (3)	87.98 (5)	89.33 (12)
<i>C3-M-S2</i>	163.06 (5)	166.23 (5)	167.27 (9)	85.88 (4)	87.58 (6)	87.74 (14)

References: (a) Peschel *et al.* (2015a); (b) Peschel *et al.* (2019); (c) this work.

the Mo–S distances of the benzenethiolate residues in isomer **1** are significantly different, although they are *trans* to one another, and both are clearly shorter [$\text{Mo1}-\text{S1} = 2.4673$ (3) \AA and $\text{Mo1}-\text{S2} = 2.3665$ (3) \AA] than in isomer **2** [$\text{Mo1}-\text{S1} = 2.5254$ (8) \AA and $\text{Mo1}-\text{S2} = 2.5297$ (8) \AA] or in the W compound [$\text{W}-\text{S} = 2.5232$ (4)– 2.5243 (4) \AA]. On the other hand, in both isomers, the distances are almost the same between the central atom and the butyne ligands [2.0310 (12)– 2.0664 (12) *versus* 2.024 (3)– 2.059 (3) \AA] and to the carbonyl ligands [1.9417 (13) *versus* 1.953 (3) \AA], although both are arranged in *trans* positions with respect to the N atoms of the oxazole rings in **1**, and *trans* to the S atoms of the benzenethiolate groups in **2**. In both isomers, the CO ligands [$\text{C3}-\text{O3} = 1.1555$ (16) and 1.157 (3) \AA] lie roughly in the best planes through the butyne ligands [$\text{C1}-\text{C2} = 1.2965$ (18) and 1.314 (4) \AA] and the Mo atoms.

Comparing all known structures of $M(\text{CO})(\text{C}_2\text{R}_2)(\text{S-Phoz})_2$ complexes (Table 2), the following conclusions can be made: whereas *N,N-trans* conformations for $R = \text{H}$ and CH_3 , and *S,S-trans* conformations for $R = \text{Ph}$ were observed (Peschel *et al.*, 2015a, 2019) for the W complexes, both conformations were found in the first two crystal structures of the analogous Mo complexes with $R = \text{CH}_3$. In general, the Mo–N distances are clearly longer in the *S,S-trans* conformers, and slightly longer for the S-Phoz ligands *trans* to the alkyne ligands than those *trans* to the carbonyl ligand (*e.g.* $M-\text{N13}$ is larger than $M-\text{N33}$). In isomer **1**, the Mo–N distance of the S-Phoz ligand *trans* to the butyne ligand is exceptionally large due to the wide $\text{C1}-\text{Mo1}-\text{N13}$ angle of 173.53 (4) $^\circ$ and large $\text{C}-\text{M}-\text{S1}$ angles. The Mo–S distances are the same in the *N,N-trans* conformers, but in the *S,S-trans* conformers, $M-\text{S1}$ is distinctly longer than $M-\text{S2}$. Therefore, the S-Phoz ligands whose oxazole rings are *trans* to the alkyne ligands are more weakly bound to the metal centre than the others. In all six

complexes (Table 2), the $M-\text{C1}$ distance is significantly shorter than $M-\text{C2}$, presumably due to the carbonyl ligand near atom C2.

3.2. NMR spectroscopy

^1H NMR spectra recorded in CD_2Cl_2 and CD_3CN show a 1:2 ratio of the two isomers of $\text{Mo}(\text{CO})(\text{C}_2\text{Me}_2)(\text{S-Phoz})_2$, while a 1:1 ratio is observed in CDCl_3 . The NMR data of isomer **2**, which presumably adopts the *N,N-trans* configuration, are almost identical with those of the W analogue (Peschel *et al.*, 2019), of which only the *N,N-trans* isomer was crystallized. In CD_2Cl_2 solutions, the two isomers of $\text{W}(\text{CO})(\text{C}_2\text{Me}_2)(\text{S-Phoz})_2$ exhibit a 95:5 ratio, with a clear preference for the *N,N-trans* configuration of isomer **2**.

3.3. IR spectroscopy

The IR spectrum of an average sample of $\text{Mo}(\text{CO})(\text{C}_2\text{Me}_2)(\text{S-Phoz})_2$ shows a very strong band at 1898 cm^{-1} which is attributed to the $\text{C}\equiv\text{O}$ bond. Due to weaker π -backbonding of the Mo centre, this bond is stronger by 18 cm^{-1} compared to that in the respective W compound (Peschel *et al.*, 2019), which is in accordance with previous observations on Mo and W carbonyl complexes (Ehweiner *et al.*, 2021a,b,c). Despite the existence of two isomers, only one $\text{C}\equiv\text{O}$ bond is visible.

Acknowledgements

Financial support by NAWI Graz is gratefully acknowledged.

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supporting information

Acta Cryst. (2022). C78, 218-222 [https://doi.org/10.1107/S2053229622002029]

Synthesis and structure of two isomers of a molybdenum(II) 2-butyne complex stabilized by bioinspired *S,N*-bidentate ligands

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Computing details

For both structures, data collection: *APEX2* (Bruker, 2013); cell refinement: *SAINTE* (Bruker, 2013); data reduction: *SAINTE* (Bruker, 2013); program(s) used to solve structure: *SHELXL97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL2014* (Sheldrick, 2015); molecular graphics: modified *ORTEP* (Johnson, 1965); software used to prepare material for publication: *SHELXL2014* (Sheldrick, 2015).

N,N-*cis*-(η^2 -But-2-yne)carbonylbis[2-(4,4-dimethyl-4,5-dihydro-1,3-oxazol-2-yl)benzenethiolato]molybdenum(II) (1)

Crystal data

[Mo(C₁₁H₁₂NOS)₂(C₄H₆)(CO)]

$M_r = 590.59$

Monoclinic, $P2_1/n$

$a = 10.6159$ (5) Å

$b = 8.9300$ (4) Å

$c = 27.3801$ (12) Å

$\beta = 96.189$ (2)°

$V = 2580.5$ (2) Å³

$Z = 4$

$F(000) = 1216$

$D_x = 1.520$ Mg m⁻³

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

Cell parameters from 9935 reflections

$\theta = 2.4$ – 35.8 °

$\mu = 0.70$ mm⁻¹

$T = 100$ K

Plate, green

$0.18 \times 0.18 \times 0.10$ mm

Data collection

Bruker APEXII CCD
diffractometer

Radiation source: Incoatec microfocus sealed
tube

Multilayer monochromator

φ and ω scans

Absorption correction: multi-scan
(SADABS; Bruker, 2013)

$T_{\min} = 0.884$, $T_{\max} = 1.000$

30042 measured reflections

11363 independent reflections

9549 reflections with $I > 2\sigma(I)$

$R_{\text{int}} = 0.029$

$\theta_{\max} = 35.0$ °, $\theta_{\min} = 1.5$ °

$h = -17$ → 17

$k = -14$ → 11

$l = -44$ → 42

Refinement

Refinement on F^2

Least-squares matrix: full

$R[F^2 > 2\sigma(F^2)] = 0.028$

$wR(F^2) = 0.071$

$S = 1.04$

11363 reflections

332 parameters

0 restraints

Primary atom site location: structure-invariant
direct methods

Secondary atom site location: difference Fourier
map

Hydrogen site location: inferred from
neighbouring sites

Only H-atom displacement parameters refined
 $w = 1/[\sigma^2(F_o^2) + (0.0331P)^2 + 0.5019P]$
 where $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{\max} = 0.008$
 $\Delta\rho_{\max} = 0.72 \text{ e } \text{\AA}^{-3}$
 $\Delta\rho_{\min} = -0.64 \text{ e } \text{\AA}^{-3}$

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.

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 $F^2 > 2\sigma(F^2)$ 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.

The non-hydrogen atoms were refined with anisotropic displacement parameters without any constraints.

The H atoms of the CH_2 groups were put at positions with approx. tetrahedral angles and C-H distances of 0.99 Å, and common isotropic displacement parameters were refined for the H atoms of the same group (AFIX 23 of SHELXL).

The H atoms of the phenyl rings were put at the external bisectors of the C-C-C angles at C-H distances of 0.95 Å and common isotropic displacement parameters were refined for the H atoms of the same ring (AFIX 43 of SHELXL).

The H atoms of the methyl groups were refined with common isotropic displacement parameters for the H atoms of the same group and idealized geometries with tetrahedral angles, enabling rotations around the C-C bonds, and C-H distances of 0.98 Å (AFIX 137 of SHELXL).

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

	x	y	z	$U_{\text{iso}}^*/U_{\text{eq}}$
Mo1	0.89263 (2)	0.62618 (2)	0.60957 (2)	0.00748 (3)
C10	0.79768 (14)	0.34484 (15)	0.53122 (5)	0.0169 (2)
H101	0.8405	0.3231	0.5020	0.032 (3)*
H102	0.7171	0.3954	0.5213	0.032 (3)*
H103	0.7816	0.2510	0.5480	0.032 (3)*
C1	0.87961 (12)	0.44360 (14)	0.56508 (4)	0.0118 (2)
C2	0.99846 (12)	0.46043 (14)	0.58069 (4)	0.0116 (2)
C20	1.12488 (14)	0.39396 (16)	0.57708 (6)	0.0196 (3)
H201	1.1150	0.2896	0.5663	0.065 (5)*
H202	1.1748	0.3975	0.6093	0.065 (5)*
H203	1.1685	0.4507	0.5533	0.065 (5)*
C3	1.07172 (12)	0.65704 (14)	0.62980 (4)	0.0117 (2)
O3	1.17882 (9)	0.67813 (12)	0.63972 (4)	0.01788 (19)
O11	0.93259 (10)	0.95082 (11)	0.74208 (3)	0.01669 (18)
C12	0.94202 (12)	0.82783 (14)	0.71368 (4)	0.0116 (2)
N13	0.88974 (10)	0.83507 (11)	0.66865 (3)	0.00967 (17)
C14	0.84945 (12)	0.99714 (13)	0.66127 (4)	0.01058 (19)
C15	0.84074 (13)	1.04622 (14)	0.71414 (4)	0.0151 (2)
H151	0.8632	1.1533	0.7188	0.015 (3)*
H152	0.7545	1.0296	0.7237	0.015 (3)*
C16	0.72358 (12)	1.01441 (14)	0.63024 (5)	0.0140 (2)
H161	0.7321	0.9824	0.5965	0.024 (3)*
H162	0.6973	1.1196	0.6302	0.024 (3)*
H163	0.6597	0.9525	0.6439	0.024 (3)*

C17	0.95572 (13)	1.08459 (15)	0.64041 (5)	0.0162 (2)
H171	0.9662	1.0476	0.6074	0.023 (3)*
H172	1.0350	1.0709	0.6618	0.023 (3)*
H173	0.9339	1.1912	0.6387	0.023 (3)*
S1	0.86588 (3)	0.49578 (3)	0.68722 (2)	0.01150 (6)
C21	0.99285 (12)	0.55484 (14)	0.72917 (4)	0.0120 (2)
C22	1.01709 (12)	0.70709 (14)	0.73914 (4)	0.0122 (2)
C23	1.11465 (13)	0.74917 (17)	0.77533 (4)	0.0175 (2)
H23	1.1303	0.8523	0.7821	0.028 (3)*
C24	1.18814 (15)	0.64158 (18)	0.80126 (5)	0.0225 (3)
H24	1.2544	0.6707	0.8255	0.028 (3)*
C25	1.16465 (15)	0.49087 (18)	0.79168 (5)	0.0225 (3)
H25	1.2149	0.4169	0.8095	0.028 (3)*
C26	1.06824 (14)	0.44795 (16)	0.75624 (5)	0.0178 (2)
H26	1.0529	0.3445	0.7502	0.028 (3)*
O31	0.48698 (9)	0.68836 (11)	0.54235 (3)	0.01576 (18)
C32	0.61444 (11)	0.70736 (13)	0.54906 (4)	0.01009 (19)
N33	0.67394 (10)	0.64237 (11)	0.58729 (4)	0.00932 (17)
C34	0.57211 (11)	0.57021 (14)	0.61487 (4)	0.0110 (2)
C35	0.45570 (12)	0.57671 (16)	0.57686 (5)	0.0154 (2)
H351	0.4402	0.4785	0.5605	0.022 (3)*
H352	0.3794	0.6060	0.5924	0.022 (3)*
C36	0.55298 (13)	0.66662 (15)	0.65962 (5)	0.0155 (2)
H361	0.4919	0.6179	0.6788	0.019 (3)*
H362	0.5207	0.7652	0.6487	0.019 (3)*
H363	0.6341	0.6785	0.6800	0.019 (3)*
C37	0.59886 (13)	0.40709 (14)	0.62857 (5)	0.0148 (2)
H371	0.6322	0.3555	0.6011	0.029 (3)*
H372	0.5202	0.3585	0.6358	0.029 (3)*
H373	0.6614	0.4023	0.6576	0.029 (3)*
S2	0.91487 (3)	0.81414 (3)	0.55017 (2)	0.01031 (5)
C41	0.78108 (11)	0.85979 (13)	0.51004 (4)	0.00942 (19)
C42	0.65755 (12)	0.80444 (13)	0.51052 (4)	0.01027 (19)
C43	0.56414 (13)	0.84871 (15)	0.47232 (5)	0.0155 (2)
H43	0.4802	0.8117	0.4722	0.020 (2)*
C44	0.59196 (14)	0.94420 (16)	0.43528 (5)	0.0184 (2)
H44	0.5284	0.9694	0.4095	0.020 (2)*
C45	0.71325 (13)	1.00322 (15)	0.43593 (5)	0.0159 (2)
H45	0.7324	1.0715	0.4112	0.020 (2)*
C46	0.80578 (13)	0.96192 (14)	0.47282 (4)	0.0128 (2)
H46	0.8883	1.0033	0.4732	0.020 (2)*

Atomic displacement parameters (\AA^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Mo1	0.00739 (4)	0.00758 (4)	0.00757 (4)	0.00003 (3)	0.00130 (3)	-0.00047 (3)
C10	0.0187 (6)	0.0167 (6)	0.0149 (5)	-0.0037 (5)	0.0000 (4)	-0.0055 (4)
C1	0.0156 (6)	0.0095 (5)	0.0105 (4)	-0.0010 (4)	0.0020 (4)	-0.0013 (4)

C2	0.0134 (5)	0.0106 (5)	0.0113 (4)	0.0013 (4)	0.0036 (4)	-0.0019 (4)
C20	0.0141 (6)	0.0183 (6)	0.0274 (7)	0.0035 (5)	0.0063 (5)	-0.0073 (5)
C3	0.0134 (5)	0.0110 (5)	0.0108 (4)	0.0002 (4)	0.0021 (4)	-0.0020 (4)
O3	0.0106 (4)	0.0223 (5)	0.0205 (4)	-0.0016 (4)	0.0008 (3)	-0.0043 (4)
O11	0.0243 (5)	0.0138 (4)	0.0110 (4)	0.0063 (4)	-0.0023 (3)	-0.0052 (3)
C12	0.0127 (5)	0.0112 (5)	0.0109 (4)	0.0016 (4)	0.0015 (4)	-0.0021 (4)
N13	0.0106 (4)	0.0086 (4)	0.0098 (4)	0.0009 (3)	0.0011 (3)	-0.0003 (3)
C14	0.0123 (5)	0.0082 (4)	0.0112 (4)	0.0011 (4)	0.0011 (4)	-0.0010 (4)
C15	0.0205 (6)	0.0119 (5)	0.0128 (5)	0.0063 (5)	0.0011 (4)	-0.0017 (4)
C16	0.0137 (6)	0.0118 (5)	0.0163 (5)	0.0027 (4)	0.0003 (4)	0.0010 (4)
C17	0.0171 (6)	0.0118 (5)	0.0201 (6)	-0.0038 (5)	0.0042 (5)	-0.0016 (4)
S1	0.01297 (13)	0.01097 (12)	0.01060 (11)	-0.00069 (10)	0.00153 (9)	0.00193 (9)
C21	0.0122 (5)	0.0142 (5)	0.0096 (4)	0.0022 (4)	0.0019 (4)	0.0015 (4)
C22	0.0125 (5)	0.0143 (5)	0.0096 (4)	0.0031 (4)	0.0007 (4)	0.0000 (4)
C23	0.0178 (6)	0.0204 (6)	0.0134 (5)	0.0033 (5)	-0.0031 (4)	-0.0033 (4)
C24	0.0200 (7)	0.0277 (7)	0.0179 (6)	0.0063 (6)	-0.0065 (5)	-0.0030 (5)
C25	0.0229 (7)	0.0256 (7)	0.0177 (6)	0.0096 (6)	-0.0042 (5)	0.0049 (5)
C26	0.0204 (6)	0.0161 (6)	0.0165 (5)	0.0041 (5)	-0.0003 (5)	0.0038 (4)
O31	0.0087 (4)	0.0192 (5)	0.0189 (4)	-0.0033 (3)	-0.0008 (3)	0.0071 (3)
C32	0.0088 (5)	0.0101 (5)	0.0114 (4)	-0.0017 (4)	0.0012 (4)	-0.0012 (4)
N33	0.0087 (4)	0.0090 (4)	0.0104 (4)	-0.0010 (3)	0.0020 (3)	0.0002 (3)
C34	0.0090 (5)	0.0118 (5)	0.0127 (5)	-0.0022 (4)	0.0028 (4)	0.0011 (4)
C35	0.0103 (5)	0.0175 (6)	0.0182 (5)	-0.0040 (5)	0.0006 (4)	0.0063 (4)
C36	0.0167 (6)	0.0152 (5)	0.0156 (5)	-0.0013 (5)	0.0070 (4)	0.0005 (4)
C37	0.0146 (6)	0.0099 (5)	0.0201 (5)	-0.0026 (4)	0.0022 (4)	0.0028 (4)
S2	0.00911 (12)	0.01204 (12)	0.00983 (11)	-0.00215 (10)	0.00123 (9)	0.00168 (9)
C41	0.0105 (5)	0.0092 (5)	0.0089 (4)	-0.0001 (4)	0.0023 (4)	-0.0009 (3)
C42	0.0112 (5)	0.0099 (5)	0.0096 (4)	-0.0012 (4)	0.0006 (4)	0.0003 (4)
C43	0.0142 (6)	0.0170 (6)	0.0145 (5)	-0.0014 (4)	-0.0017 (4)	0.0030 (4)
C44	0.0177 (6)	0.0207 (6)	0.0157 (5)	-0.0007 (5)	-0.0030 (4)	0.0066 (5)
C45	0.0191 (6)	0.0155 (6)	0.0131 (5)	-0.0001 (5)	0.0020 (4)	0.0047 (4)
C46	0.0147 (6)	0.0126 (5)	0.0116 (5)	-0.0009 (4)	0.0030 (4)	0.0019 (4)

Geometric parameters (Å, °)

Mo1—C1	2.0310 (12)	C22—C23	1.4052 (18)
Mo1—C2	2.0664 (12)	C23—C24	1.384 (2)
Mo1—C3	1.9417 (13)	C23—H23	0.95
Mo1—N13	2.4715 (10)	C24—C25	1.389 (2)
Mo1—N33	2.3404 (11)	C24—H24	0.95
Mo1—S1	2.4673 (3)	C25—C26	1.386 (2)
Mo1—S2	2.3665 (3)	C25—H25	0.95
C1—C2	1.2965 (18)	C26—H26	0.95
C1—C10	1.4899 (17)	O31—C32	1.3564 (15)
C10—H101	0.98	O31—C35	1.4370 (15)
C10—H102	0.98	C32—N33	1.2996 (15)
C10—H103	0.98	C32—C42	1.4759 (16)
C2—C20	1.4804 (19)	N33—C34	1.5264 (15)

C20—H201	0.98	C34—C37	1.5233 (18)
C20—H202	0.98	C34—C35	1.5285 (18)
C20—H203	0.98	C34—C36	1.5288 (17)
C3—O3	1.1555 (16)	C35—H351	0.99
O11—C12	1.3558 (15)	C35—H352	0.99
O11—C15	1.4494 (16)	C36—H361	0.98
C12—N13	1.2978 (15)	C36—H362	0.98
C12—C22	1.4710 (17)	C36—H363	0.98
N13—C14	1.5166 (15)	C37—H371	0.98
C14—C16	1.5120 (18)	C37—H372	0.98
C14—C15	1.5247 (16)	C37—H373	0.98
C14—C17	1.5322 (18)	S2—C41	1.7477 (12)
C15—H151	0.99	C41—C42	1.4029 (17)
C15—H152	0.99	C41—C46	1.4131 (16)
C16—H161	0.98	C42—C43	1.4177 (17)
C16—H162	0.98	C43—C44	1.3811 (18)
C16—H163	0.98	C43—H43	0.95
C17—H171	0.98	C44—C45	1.390 (2)
C17—H172	0.98	C44—H44	0.95
C17—H173	0.98	C45—C46	1.3810 (18)
S1—C21	1.7551 (13)	C45—H45	0.95
C21—C26	1.4046 (18)	C46—H46	0.95
C21—C22	1.4051 (18)		
N13—Mo1—N33	92.41 (3)	H172—C17—H173	109.5
S1—Mo1—S2	162.979 (11)	C21—S1—Mo1	105.69 (4)
C1—Mo1—N13	173.53 (4)	C26—C21—C22	118.23 (12)
C2—Mo1—N13	146.80 (4)	C26—C21—S1	119.66 (10)
C3—Mo1—N33	168.19 (4)	C22—C21—S1	121.97 (9)
C3—Mo1—C1	106.63 (5)	C21—C22—C23	120.09 (11)
C3—Mo1—C2	69.81 (5)	C21—C22—C12	122.60 (11)
C1—Mo1—C2	36.88 (5)	C23—C22—C12	117.30 (12)
C1—Mo1—N33	83.79 (4)	C24—C23—C22	120.52 (13)
C2—Mo1—N33	120.63 (4)	C24—C23—H23	119.7
C3—Mo1—S2	85.88 (4)	C22—C23—H23	119.7
C1—Mo1—S2	99.36 (3)	C23—C24—C25	119.73 (13)
C2—Mo1—S2	98.42 (3)	C23—C24—H24	120.1
N33—Mo1—S2	86.95 (3)	C25—C24—H24	120.1
C3—Mo1—S1	91.22 (4)	C26—C25—C24	120.28 (13)
C1—Mo1—S1	97.54 (3)	C26—C25—H25	119.9
C2—Mo1—S1	96.29 (3)	C24—C25—H25	119.9
N33—Mo1—S1	92.90 (2)	C25—C26—C21	121.14 (13)
C3—Mo1—N13	77.70 (4)	C25—C26—H26	119.4
S2—Mo1—N13	85.62 (2)	C21—C26—H26	119.4
S1—Mo1—N13	77.38 (2)	C32—O31—C35	107.19 (9)
C1—C10—H101	109.5	N33—C32—O31	116.38 (10)
C1—C10—H102	109.5	N33—C32—C42	132.61 (11)
H101—C10—H102	109.5	O31—C32—C42	110.99 (10)

C1—C10—H103	109.5	C32—N33—C34	106.17 (10)
H101—C10—H103	109.5	C32—N33—Mo1	128.30 (8)
H102—C10—H103	109.5	C34—N33—Mo1	125.49 (7)
C2—C1—C10	139.10 (12)	C37—C34—N33	113.73 (10)
C2—C1—Mo1	73.04 (7)	C37—C34—C35	108.63 (10)
C10—C1—Mo1	147.76 (10)	N33—C34—C35	102.17 (9)
C1—C2—C20	142.05 (12)	C37—C34—C36	112.24 (10)
C1—C2—Mo1	70.08 (7)	N33—C34—C36	108.73 (10)
C20—C2—Mo1	147.87 (10)	C35—C34—C36	110.91 (11)
C2—C20—H201	109.5	O31—C35—C34	104.29 (10)
C2—C20—H202	109.5	O31—C35—H351	110.9
H201—C20—H202	109.5	C34—C35—H351	110.9
C2—C20—H203	109.5	O31—C35—H352	110.9
H201—C20—H203	109.5	C34—C35—H352	110.9
H202—C20—H203	109.5	H351—C35—H352	108.9
Mo1—C3—O3	176.84 (11)	C34—C36—H361	109.5
C12—O11—C15	105.09 (9)	C34—C36—H362	109.5
N13—C12—O11	117.01 (11)	H361—C36—H362	109.5
N13—C12—C22	129.87 (11)	C34—C36—H363	109.5
O11—C12—C22	113.06 (10)	H361—C36—H363	109.5
C12—N13—C14	105.05 (9)	H362—C36—H363	109.5
C12—N13—Mo1	123.36 (8)	C34—C37—H371	109.5
C14—N13—Mo1	131.02 (7)	C34—C37—H372	109.5
C16—C14—N13	113.06 (10)	H371—C37—H372	109.5
C16—C14—C15	111.33 (10)	C34—C37—H373	109.5
N13—C14—C15	101.12 (9)	H371—C37—H373	109.5
C16—C14—C17	112.34 (10)	H372—C37—H373	109.5
N13—C14—C17	109.12 (10)	C41—S2—Mo1	117.58 (4)
C15—C14—C17	109.25 (10)	C42—C41—C46	118.72 (11)
O11—C15—C14	103.06 (9)	C42—C41—S2	127.52 (9)
O11—C15—H151	111.2	C46—C41—S2	113.74 (9)
C14—C15—H151	111.2	C41—C42—C43	118.36 (11)
O11—C15—H152	111.2	C41—C42—C32	125.16 (11)
C14—C15—H152	111.2	C43—C42—C32	116.43 (11)
H151—C15—H152	109.1	C44—C43—C42	121.73 (12)
C14—C16—H161	109.5	C44—C43—H43	119.1
C14—C16—H162	109.5	C42—C43—H43	119.1
H161—C16—H162	109.5	C43—C44—C45	119.73 (12)
C14—C16—H163	109.5	C43—C44—H44	120.1
H161—C16—H163	109.5	C45—C44—H44	120.1
H162—C16—H163	109.5	C46—C45—C44	119.55 (12)
C14—C17—H171	109.5	C46—C45—H45	120.2
C14—C17—H172	109.5	C44—C45—H45	120.2
H171—C17—H172	109.5	C45—C46—C41	121.81 (12)
C14—C17—H173	109.5	C45—C46—H46	119.1
H171—C17—H173	109.5	C41—C46—H46	119.1
C1—C2—Mo1—C3	-176.82 (9)	C22—C21—C26—C25	-0.4 (2)

C10—C1—C2—C20	-2.9 (3)	S1—C21—C26—C25	-176.27 (11)
Mo1—C1—C2—C20	-179.67 (19)	C35—O31—C32—N33	-9.48 (15)
Mo1—C2—C1—C10	176.81 (17)	C35—O31—C32—C42	171.98 (10)
C15—O11—C12—N13	11.37 (15)	O31—C32—N33—C34	-3.53 (14)
C15—O11—C12—C22	-171.33 (11)	C42—C32—N33—C34	174.63 (12)
O11—C12—N13—C14	8.08 (15)	O31—C32—N33—Mo1	174.03 (8)
C22—C12—N13—C14	-168.70 (13)	C42—C32—N33—Mo1	-7.82 (19)
O11—C12—N13—Mo1	-179.70 (8)	C32—N33—C34—C37	130.83 (11)
C22—C12—N13—Mo1	3.52 (19)	Mo1—N33—C34—C37	-46.81 (13)
C12—N13—C14—C16	-141.83 (11)	C32—N33—C34—C35	13.97 (12)
Mo1—N13—C14—C16	46.79 (14)	Mo1—N33—C34—C35	-163.67 (8)
C12—N13—C14—C15	-22.71 (13)	C32—N33—C34—C36	-103.33 (11)
Mo1—N13—C14—C15	165.91 (8)	Mo1—N33—C34—C36	79.03 (11)
C12—N13—C14—C17	92.37 (12)	C32—O31—C35—C34	17.70 (13)
Mo1—N13—C14—C17	-79.01 (12)	C37—C34—C35—O31	-139.33 (11)
C12—O11—C15—C14	-25.04 (13)	N33—C34—C35—O31	-18.86 (12)
C16—C14—C15—O11	148.99 (11)	C36—C34—C35—O31	96.87 (12)
N13—C14—C15—O11	28.64 (12)	Mo1—S2—C41—C42	4.37 (12)
C17—C14—C15—O11	-86.35 (12)	Mo1—S2—C41—C46	-174.05 (7)
Mo1—S1—C21—C26	-129.28 (10)	C46—C41—C42—C43	2.65 (17)
Mo1—S1—C21—C22	55.02 (10)	S2—C41—C42—C43	-175.70 (9)
C26—C21—C22—C23	0.06 (18)	C46—C41—C42—C32	-174.71 (11)
S1—C21—C22—C23	175.83 (10)	S2—C41—C42—C32	6.94 (18)
C26—C21—C22—C12	-178.65 (12)	N33—C32—C42—C41	-5.9 (2)
S1—C21—C22—C12	-2.89 (16)	O31—C32—C42—C41	172.29 (11)
N13—C12—C22—C21	-34.6 (2)	N33—C32—C42—C43	176.66 (13)
O11—C12—C22—C21	148.52 (12)	O31—C32—C42—C43	-5.11 (15)
N13—C12—C22—C23	146.65 (14)	C41—C42—C43—C44	-0.19 (19)
O11—C12—C22—C23	-30.23 (16)	C32—C42—C43—C44	177.40 (12)
C21—C22—C23—C24	0.4 (2)	C42—C43—C44—C45	-2.2 (2)
C12—C22—C23—C24	179.16 (13)	C43—C44—C45—C46	2.0 (2)
C22—C23—C24—C25	-0.5 (2)	C44—C45—C46—C41	0.5 (2)
C23—C24—C25—C26	0.1 (2)	C42—C41—C46—C45	-2.86 (18)
C24—C25—C26—C21	0.3 (2)	S2—C41—C46—C45	175.71 (10)

N,N-trans-(η^2 -But-2-yne)carbonylbis[2-(4,4-dimethyl-4,5-dihydro-1,3-oxazol-2-yl)benzenethiolato]molybdenum(II) (2)

Crystal data

[Mo(C₁₁H₁₂NOS)₂(C₄H₆)(CO)]

$M_r = 590.59$

Monoclinic, $P2_1/c$

$a = 9.1512$ (4) Å

$b = 21.3515$ (12) Å

$c = 13.1781$ (7) Å

$\beta = 98.483$ (3)°

$V = 2546.7$ (2) Å³

$Z = 4$

$F(000) = 1216$

$D_x = 1.540$ Mg m⁻³

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

Cell parameters from 3132 reflections

$\theta = 2.4$ – 26.8°

$\mu = 0.71$ mm⁻¹

$T = 100$ K

Needle, yellow

$0.23 \times 0.07 \times 0.07$ mm

Data collection

Bruker APEXII CCD diffractometer	22009 measured reflections 7415 independent reflections
Radiation source: Incoatec microfocus sealed tube	5339 reflections with $I > 2\sigma(I)$ $R_{\text{int}} = 0.068$
Multilayer monochromator	$\theta_{\text{max}} = 30.0^\circ$, $\theta_{\text{min}} = 1.8^\circ$
φ and ω scans	$h = -9 \rightarrow 12$
Absorption correction: multi-scan (SADABS; Bruker, 2013)	$k = -23 \rightarrow 30$
$T_{\text{min}} = 0.776$, $T_{\text{max}} = 1.000$	$l = -18 \rightarrow 17$

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)] = 0.043$	Only H-atom displacement parameters refined
$wR(F^2) = 0.087$	$w = 1/[\sigma^2(F_o^2) + (0.0231P)^2 + 1.0578P]$
$S = 1.01$	where $P = (F_o^2 + 2F_c^2)/3$
7415 reflections	$(\Delta/\sigma)_{\text{max}} = 0.001$
332 parameters	$\Delta\rho_{\text{max}} = 0.52 \text{ e } \text{\AA}^{-3}$
0 restraints	$\Delta\rho_{\text{min}} = -0.83 \text{ e } \text{\AA}^{-3}$
Primary atom site location: structure-invariant direct methods	

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.

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 $F^2 > 2\sigma(F^2)$ 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.

The non-hydrogen atoms were refined with anisotropic displacement parameters without any constraints. The H atoms of the CH_2 groups were put at positions with approx. tetrahedral angles and C-H distances of 0.99 Å, and common isotropic displacement parameters were refined for the H atoms of the same group (AFIX 23 of SHELXL). The H atoms of the phenyl rings were put at the external bisectors of the C-C-C angles at C-H distances of 0.95 Å and common isotropic displacement parameters were refined for the H atoms of the same ring (AFIX 43 of SHELXL). The H atoms of the methyl groups were refined with common isotropic displacement parameters for the H atoms of the same group and idealized geometries with tetrahedral angles, enabling rotations around the C-C bonds, and C-H distances of 0.98 Å (AFIX 137 of SHELXL).

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	0.72006 (2)	0.85241 (2)	0.32592 (2)	0.00779 (6)
C10	0.6039 (3)	0.75606 (14)	0.5024 (2)	0.0153 (6)
H101	0.6362	0.7619	0.5760	0.029 (6)*
H102	0.6544	0.7197	0.4784	0.029 (6)*
H103	0.4969	0.7491	0.4899	0.029 (6)*
C1	0.6403 (3)	0.81274 (14)	0.4461 (2)	0.0101 (6)
C2	0.6263 (3)	0.87368 (14)	0.4544 (2)	0.0111 (6)
C20	0.5702 (3)	0.92014 (14)	0.5225 (2)	0.0153 (6)

H201	0.4685	0.9315	0.4945	0.026 (5)*
H202	0.6326	0.9576	0.5270	0.026 (5)*
H203	0.5724	0.9021	0.5910	0.026 (5)*
C3	0.6944 (3)	0.94287 (14)	0.3361 (2)	0.0111 (6)
O3	0.6808 (2)	0.99615 (10)	0.34780 (16)	0.0158 (4)
O11	1.16158 (19)	0.91979 (10)	0.45152 (16)	0.0163 (5)
C12	1.0293 (3)	0.91404 (14)	0.3892 (2)	0.0129 (6)
N13	0.9525 (2)	0.86506 (11)	0.40345 (18)	0.0111 (5)
C14	1.0531 (3)	0.82224 (14)	0.4740 (2)	0.0134 (6)
C15	1.1612 (3)	0.87050 (15)	0.5275 (2)	0.0159 (6)
H151	1.1270	0.8866	0.5904	0.018 (6)*
H152	1.2610	0.8522	0.5459	0.018 (6)*
C16	1.1309 (3)	0.77888 (15)	0.4072 (2)	0.0174 (7)
H161	1.2006	0.7520	0.4510	0.019 (5)*
H162	1.1847	0.8040	0.3625	0.019 (5)*
H163	1.0575	0.7528	0.3650	0.019 (5)*
C17	0.9738 (3)	0.78611 (16)	0.5474 (2)	0.0181 (7)
H171	0.9163	0.8151	0.5836	0.023 (5)*
H172	1.0461	0.7642	0.5973	0.023 (5)*
H173	0.9073	0.7555	0.5092	0.023 (5)*
S1	0.85728 (7)	0.87267 (4)	0.17639 (6)	0.01253 (15)
C21	0.9358 (3)	0.94634 (14)	0.2090 (2)	0.0119 (6)
C22	1.0035 (3)	0.96176 (14)	0.3094 (2)	0.0123 (6)
C23	1.0611 (3)	1.02195 (15)	0.3305 (3)	0.0174 (7)
H23	1.1063	1.0319	0.3981	0.022 (4)*
C24	1.0534 (3)	1.06682 (15)	0.2551 (3)	0.0212 (7)
H24	1.0889	1.1080	0.2709	0.022 (4)*
C25	0.9933 (3)	1.05120 (15)	0.1556 (2)	0.0187 (7)
H25	0.9914	1.0814	0.1025	0.022 (4)*
C26	0.9359 (3)	0.99211 (15)	0.1327 (2)	0.0162 (6)
H26	0.8957	0.9823	0.0639	0.022 (4)*
O31	0.30893 (19)	0.78898 (10)	0.14341 (16)	0.0149 (4)
C32	0.4389 (3)	0.78648 (14)	0.2073 (2)	0.0115 (6)
N33	0.5126 (2)	0.83815 (11)	0.22034 (17)	0.0091 (5)
C34	0.4180 (3)	0.88879 (14)	0.1625 (2)	0.0115 (6)
C35	0.3001 (3)	0.84988 (15)	0.0959 (2)	0.0187 (6)
H351	0.2008	0.8684	0.0954	0.020 (6)*
H352	0.3212	0.8472	0.0245	0.020 (6)*
C36	0.5039 (3)	0.92864 (14)	0.0960 (2)	0.0145 (6)
H361	0.5788	0.9531	0.1400	0.017 (5)*
H362	0.4360	0.9571	0.0541	0.017 (5)*
H363	0.5523	0.9014	0.0512	0.017 (5)*
C37	0.3473 (3)	0.92796 (14)	0.2388 (2)	0.0152 (6)
H371	0.2912	0.9006	0.2786	0.025 (5)*
H372	0.2806	0.9589	0.2015	0.025 (5)*
H373	0.4246	0.9496	0.2852	0.025 (5)*
S2	0.77527 (7)	0.74136 (3)	0.27649 (6)	0.01212 (15)
C41	0.6123 (3)	0.69967 (14)	0.2814 (2)	0.0113 (6)

C42	0.4687 (3)	0.72389 (13)	0.2520 (2)	0.0105 (6)
C43	0.3449 (3)	0.68583 (14)	0.2601 (2)	0.0122 (6)
H43	0.2486	0.7026	0.2409	0.020 (4)*
C44	0.3595 (3)	0.62558 (15)	0.2947 (2)	0.0171 (6)
H44	0.2749	0.6010	0.3010	0.020 (4)*
C45	0.5018 (3)	0.60073 (14)	0.3209 (2)	0.0175 (6)
H45	0.5140	0.5584	0.3430	0.020 (4)*
C46	0.6238 (3)	0.63729 (14)	0.3149 (2)	0.0157 (6)
H46	0.7193	0.6197	0.3341	0.020 (4)*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Mo1	0.00742 (9)	0.00788 (11)	0.00833 (12)	0.00024 (9)	0.00205 (7)	-0.00025 (10)
C10	0.0191 (13)	0.0148 (15)	0.0124 (16)	-0.0046 (11)	0.0039 (11)	0.0026 (12)
C1	0.0082 (11)	0.0157 (15)	0.0061 (14)	-0.0007 (10)	0.0003 (9)	0.0018 (11)
C2	0.0069 (11)	0.0175 (15)	0.0085 (14)	0.0012 (10)	-0.0001 (10)	-0.0001 (11)
C20	0.0174 (13)	0.0158 (16)	0.0143 (16)	0.0014 (11)	0.0076 (11)	-0.0044 (12)
C3	0.0086 (11)	0.0140 (14)	0.0106 (15)	-0.0005 (10)	0.0008 (10)	0.0014 (12)
O3	0.0153 (9)	0.0126 (11)	0.0199 (12)	0.0009 (8)	0.0038 (8)	-0.0017 (9)
O11	0.0099 (8)	0.0235 (12)	0.0144 (12)	-0.0047 (8)	-0.0015 (7)	0.0004 (9)
C12	0.0098 (11)	0.0157 (15)	0.0137 (16)	0.0025 (10)	0.0033 (10)	-0.0024 (12)
N13	0.0087 (9)	0.0133 (13)	0.0111 (13)	0.0033 (8)	0.0010 (8)	0.0011 (10)
C14	0.0090 (11)	0.0184 (16)	0.0119 (15)	0.0034 (10)	-0.0010 (10)	0.0043 (12)
C15	0.0123 (12)	0.0229 (17)	0.0119 (16)	-0.0001 (11)	0.0002 (10)	0.0026 (13)
C16	0.0117 (12)	0.0212 (17)	0.0192 (17)	0.0048 (11)	0.0016 (11)	0.0052 (14)
C17	0.0132 (12)	0.0270 (18)	0.0142 (17)	0.0022 (12)	0.0023 (11)	0.0053 (14)
S1	0.0135 (3)	0.0140 (4)	0.0107 (4)	-0.0017 (3)	0.0041 (3)	-0.0009 (3)
C21	0.0086 (11)	0.0130 (14)	0.0154 (16)	-0.0009 (10)	0.0063 (10)	-0.0001 (12)
C22	0.0083 (11)	0.0137 (15)	0.0159 (16)	-0.0006 (10)	0.0054 (10)	0.0011 (12)
C23	0.0112 (12)	0.0189 (17)	0.0234 (18)	-0.0029 (11)	0.0063 (11)	-0.0030 (13)
C24	0.0203 (14)	0.0123 (16)	0.034 (2)	-0.0031 (12)	0.0143 (13)	-0.0012 (14)
C25	0.0180 (13)	0.0178 (17)	0.0227 (18)	-0.0003 (12)	0.0107 (12)	0.0065 (14)
C26	0.0153 (13)	0.0214 (17)	0.0138 (16)	0.0002 (11)	0.0081 (11)	0.0026 (13)
O31	0.0130 (9)	0.0117 (11)	0.0176 (12)	-0.0015 (7)	-0.0059 (8)	0.0037 (9)
C32	0.0099 (11)	0.0162 (15)	0.0084 (15)	0.0028 (10)	0.0019 (10)	-0.0011 (12)
N33	0.0090 (9)	0.0074 (12)	0.0105 (13)	0.0005 (8)	-0.0002 (8)	0.0011 (9)
C34	0.0123 (11)	0.0102 (14)	0.0107 (15)	0.0035 (10)	-0.0023 (10)	0.0020 (11)
C35	0.0189 (13)	0.0135 (15)	0.0211 (18)	-0.0005 (12)	-0.0055 (12)	0.0023 (14)
C36	0.0173 (13)	0.0140 (15)	0.0113 (16)	0.0021 (11)	-0.0008 (11)	0.0007 (12)
C37	0.0119 (12)	0.0166 (16)	0.0169 (17)	0.0021 (11)	0.0013 (11)	0.0001 (13)
S2	0.0110 (3)	0.0106 (3)	0.0153 (4)	0.0014 (2)	0.0037 (2)	-0.0013 (3)
C41	0.0152 (12)	0.0127 (15)	0.0067 (14)	0.0007 (10)	0.0036 (10)	-0.0012 (11)
C42	0.0128 (12)	0.0085 (14)	0.0105 (15)	0.0012 (10)	0.0022 (10)	-0.0037 (11)
C43	0.0141 (12)	0.0129 (15)	0.0096 (15)	-0.0021 (10)	0.0015 (10)	-0.0022 (11)
C44	0.0220 (14)	0.0150 (15)	0.0151 (17)	-0.0083 (12)	0.0055 (12)	-0.0017 (13)
C45	0.0319 (16)	0.0080 (14)	0.0131 (16)	0.0002 (12)	0.0049 (12)	0.0004 (12)
C46	0.0189 (13)	0.0148 (16)	0.0137 (16)	0.0033 (11)	0.0033 (11)	0.0003 (12)

Geometric parameters (Å, °)

Mo1—C1	2.024 (3)	C22—C23	1.401 (4)
Mo1—C2	2.059 (3)	C23—C24	1.375 (4)
Mo1—C3	1.953 (3)	C23—H23	0.95
Mo1—N13	2.236 (2)	C24—C25	1.386 (4)
Mo1—N33	2.203 (2)	C24—H24	0.95
Mo1—S1	2.5254 (8)	C25—C26	1.383 (4)
Mo1—S2	2.5297 (8)	C25—H25	0.95
C1—C2	1.314 (4)	C26—H26	0.95
C1—C10	1.483 (4)	O31—C32	1.353 (3)
C10—H101	0.98	O31—C35	1.440 (4)
C10—H102	0.98	C32—N33	1.291 (3)
C10—H103	0.98	C32—C42	1.469 (4)
C2—C20	1.479 (4)	N33—C34	1.518 (3)
C20—H201	0.98	C34—C36	1.521 (4)
C20—H202	0.98	C34—C37	1.523 (4)
C20—H203	0.98	C34—C35	1.531 (4)
C3—O3	1.157 (3)	C35—H351	0.99
O11—C12	1.364 (3)	C35—H352	0.99
O11—C15	1.453 (3)	C36—H361	0.98
C12—N13	1.289 (4)	C36—H362	0.98
C12—C22	1.459 (4)	C36—H363	0.98
N13—C14	1.515 (3)	C37—H371	0.98
C14—C17	1.505 (4)	C37—H372	0.98
C14—C16	1.525 (4)	C37—H373	0.98
C14—C15	1.527 (4)	S2—C41	1.746 (3)
C15—H151	0.99	C41—C46	1.402 (4)
C15—H152	0.99	C41—C42	1.412 (4)
C16—H161	0.98	C42—C43	1.411 (4)
C16—H162	0.98	C43—C44	1.365 (4)
C16—H163	0.98	C43—H43	0.95
C17—H171	0.98	C44—C45	1.401 (4)
C17—H172	0.98	C44—H44	0.95
C17—H173	0.98	C45—C46	1.374 (4)
S1—C21	1.756 (3)	C45—H45	0.95
C21—C26	1.403 (4)	C46—H46	0.95
C21—C22	1.414 (4)		
N13—Mo1—N33	168.04 (8)	H172—C17—H173	109.5
S1—Mo1—S2	79.54 (3)	C21—S1—Mo1	101.22 (10)
C1—Mo1—S1	163.76 (8)	C26—C21—C22	117.5 (3)
C2—Mo1—S1	156.87 (8)	C26—C21—S1	119.5 (2)
C3—Mo1—S2	167.27 (9)	C22—C21—S1	123.0 (2)
C3—Mo1—C1	107.43 (12)	C23—C22—C21	120.0 (3)
C3—Mo1—C2	69.91 (12)	C23—C22—C12	118.8 (3)
C1—Mo1—C2	37.52 (11)	C21—C22—C12	120.9 (3)
C3—Mo1—N33	94.51 (9)	C24—C23—C22	121.2 (3)

C1—Mo1—N33	93.43 (9)	C24—C23—H23	119.4
C2—Mo1—N33	97.17 (9)	C22—C23—H23	119.4
C3—Mo1—N13	88.01 (9)	C23—C24—C25	119.2 (3)
C1—Mo1—N13	96.97 (9)	C23—C24—H24	120.4
C2—Mo1—N13	94.67 (9)	C25—C24—H24	120.4
C3—Mo1—S1	87.86 (8)	C26—C25—C24	120.7 (3)
N33—Mo1—S1	90.70 (6)	C26—C25—H25	119.6
N13—Mo1—S1	77.69 (6)	C24—C25—H25	119.6
C1—Mo1—S2	85.29 (9)	C25—C26—C21	121.3 (3)
C2—Mo1—S2	122.81 (9)	C25—C26—H26	119.3
N33—Mo1—S2	83.92 (6)	C21—C26—H26	119.3
N13—Mo1—S2	91.04 (6)	C32—O31—C35	107.1 (2)
C1—C10—H101	109.5	N33—C32—O31	116.3 (2)
C1—C10—H102	109.5	N33—C32—C42	131.4 (2)
H101—C10—H102	109.5	O31—C32—C42	112.3 (2)
C1—C10—H103	109.5	C32—N33—C34	107.1 (2)
H101—C10—H103	109.5	C32—N33—Mo1	125.91 (18)
H102—C10—H103	109.5	C34—N33—Mo1	126.39 (17)
C2—C1—C10	137.3 (3)	N33—C34—C36	112.5 (2)
C2—C1—Mo1	72.66 (18)	N33—C34—C37	109.0 (2)
C10—C1—Mo1	150.0 (2)	C36—C34—C37	112.3 (2)
C1—C2—C20	139.6 (3)	N33—C34—C35	101.7 (2)
C1—C2—Mo1	69.82 (18)	C36—C34—C35	110.7 (2)
C20—C2—Mo1	150.5 (2)	C37—C34—C35	110.1 (2)
C2—C20—H201	109.5	O31—C35—C34	104.5 (2)
C2—C20—H202	109.5	O31—C35—H351	110.8
H201—C20—H202	109.5	C34—C35—H351	110.8
C2—C20—H203	109.5	O31—C35—H352	110.8
H201—C20—H203	109.5	C34—C35—H352	110.8
H202—C20—H203	109.5	H351—C35—H352	108.9
Mo1—C3—O3	176.3 (2)	C34—C36—H361	109.5
C12—O11—C15	104.9 (2)	C34—C36—H362	109.5
N13—C12—O11	116.1 (3)	H361—C36—H362	109.5
N13—C12—C22	129.6 (2)	C34—C36—H363	109.5
O11—C12—C22	114.0 (2)	H361—C36—H363	109.5
C12—N13—C14	106.5 (2)	H362—C36—H363	109.5
C12—N13—Mo1	122.26 (18)	C34—C37—H371	109.5
C14—N13—Mo1	131.18 (17)	C34—C37—H372	109.5
C17—C14—N13	113.4 (2)	H371—C37—H372	109.5
C17—C14—C16	111.8 (3)	C34—C37—H373	109.5
N13—C14—C16	107.7 (2)	H371—C37—H373	109.5
C17—C14—C15	113.0 (3)	H372—C37—H373	109.5
N13—C14—C15	99.8 (2)	C41—S2—Mo1	105.32 (10)
C16—C14—C15	110.5 (2)	C46—C41—C42	117.3 (2)
O11—C15—C14	103.8 (2)	C46—C41—S2	118.1 (2)
O11—C15—H151	111.0	C42—C41—S2	124.6 (2)
C14—C15—H151	111.0	C43—C42—C41	119.6 (3)
O11—C15—H152	111.0	C43—C42—C32	116.8 (2)

C14—C15—H152	111.0	C41—C42—C32	123.6 (2)
H151—C15—H152	109.0	C44—C43—C42	121.9 (3)
C14—C16—H161	109.5	C44—C43—H43	119.1
C14—C16—H162	109.5	C42—C43—H43	119.1
H161—C16—H162	109.5	C43—C44—C45	118.8 (3)
C14—C16—H163	109.5	C43—C44—H44	120.6
H161—C16—H163	109.5	C45—C44—H44	120.6
H162—C16—H163	109.5	C46—C45—C44	120.3 (3)
C14—C17—H171	109.5	C46—C45—H45	119.9
C14—C17—H172	109.5	C44—C45—H45	119.9
H171—C17—H172	109.5	C45—C46—C41	122.3 (3)
C14—C17—H173	109.5	C45—C46—H46	118.9
H171—C17—H173	109.5	C41—C46—H46	118.9
C1—C2—Mo1—C3	178.5 (2)	C22—C21—C26—C25	-3.2 (4)
C10—C1—C2—C20	1.1 (6)	S1—C21—C26—C25	178.4 (2)
Mo1—C1—C2—C20	179.2 (4)	C35—O31—C32—N33	7.8 (3)
Mo1—C2—C1—C10	-178.1 (3)	C35—O31—C32—C42	-173.6 (2)
C15—O11—C12—N13	-8.7 (3)	O31—C32—N33—C34	4.4 (3)
C15—O11—C12—C22	177.1 (2)	C42—C32—N33—C34	-173.8 (3)
O11—C12—N13—C14	-11.2 (3)	O31—C32—N33—Mo1	175.82 (17)
C22—C12—N13—C14	162.0 (3)	C42—C32—N33—Mo1	-2.4 (5)
O11—C12—N13—Mo1	171.36 (17)	C32—N33—C34—C36	-132.2 (3)
C22—C12—N13—Mo1	-15.5 (4)	Mo1—N33—C34—C36	56.4 (3)
C12—N13—C14—C17	145.1 (3)	C32—N33—C34—C37	102.6 (3)
Mo1—N13—C14—C17	-37.8 (4)	Mo1—N33—C34—C37	-68.8 (3)
C12—N13—C14—C16	-90.7 (3)	C32—N33—C34—C35	-13.7 (3)
Mo1—N13—C14—C16	86.5 (3)	Mo1—N33—C34—C35	174.92 (18)
C12—N13—C14—C15	24.7 (3)	C32—O31—C35—C34	-16.0 (3)
Mo1—N13—C14—C15	-158.15 (19)	N33—C34—C35—O31	17.6 (3)
C12—O11—C15—C14	24.1 (3)	C36—C34—C35—O31	137.4 (2)
C17—C14—C15—O11	-149.7 (2)	C37—C34—C35—O31	-97.9 (3)
N13—C14—C15—O11	-29.0 (3)	Mo1—S2—C41—C46	-144.6 (2)
C16—C14—C15—O11	84.3 (3)	Mo1—S2—C41—C42	36.7 (3)
Mo1—S1—C21—C26	-138.2 (2)	C46—C41—C42—C43	1.8 (4)
Mo1—S1—C21—C22	43.6 (2)	S2—C41—C42—C43	-179.5 (2)
C26—C21—C22—C23	3.1 (4)	C46—C41—C42—C32	-174.7 (3)
S1—C21—C22—C23	-178.6 (2)	S2—C41—C42—C32	4.0 (4)
C26—C21—C22—C12	-170.1 (2)	N33—C32—C42—C43	152.4 (3)
S1—C21—C22—C12	8.2 (4)	O31—C32—C42—C43	-25.9 (4)
N13—C12—C22—C23	153.7 (3)	N33—C32—C42—C41	-31.0 (5)
O11—C12—C22—C23	-33.0 (4)	O31—C32—C42—C41	150.7 (3)
N13—C12—C22—C21	-33.0 (4)	C41—C42—C43—C44	-0.7 (4)
O11—C12—C22—C21	140.3 (3)	C32—C42—C43—C44	176.1 (3)
C21—C22—C23—C24	-0.1 (4)	C42—C43—C44—C45	-1.3 (5)
C12—C22—C23—C24	173.2 (2)	C43—C44—C45—C46	2.1 (5)
C22—C23—C24—C25	-2.8 (4)	C44—C45—C46—C41	-1.0 (5)
C23—C24—C25—C26	2.7 (4)	C42—C41—C46—C45	-1.0 (4)

C24—C25—C26—C21

0.4 (4)

S2—C41—C46—C45

−179.8 (2)
