IUCrData

ISSN 2414-3146

Tetracarbonyl[4,4-dimethyl-2-(pyridin-2-yl)-2-oxazoline-$\kappa^2 N,N'$]molybdenum(0)

Christoph Steinlechner, Anke Spannenberg, Henrik Junge and Matthias Beller*

Leibniz-Institut für Katalyse e. V. an der Universität Rostock, Albert-Einstein-Str. 29a, 18059 Rostock, Germany. *Correspondence e-mail: matthias.beller@catalysis.de

Received 29 January 2019
Accepted 25 February 2019

Edited by W. Imhof, University Koblenz-Landau, Germany

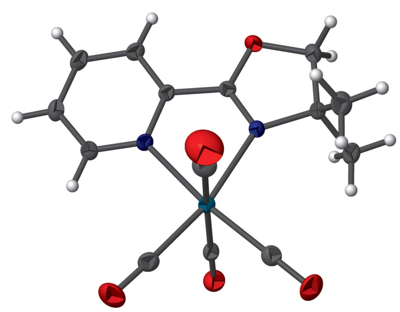
Keywords: crystal structure; bidentate; diimine; molybdenum.

CCDC reference: 1899206

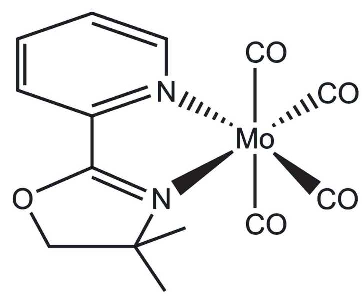
Structural data: full structural data are available from iucrdata.iucr.org

In the title compound, $[Mo(C_{10}H_{12}N_2O)(CO)_4]$, the molybdenum(0) center is surrounded by a bidentate diimine [4,4-dimethyl-2-(pyridin-2-yl)-2-oxazoline] and four carbonyl ligands in a distorted octahedral coordination geometry. The diimine ligand coordinates *via* the two nitrogen atoms.

3D view



Chemical scheme



Structure description

A diimine and four carbonyl ligands are coordinated to a molybdenum(0) atom which shows a distorted octahedral coordination geometry (Fig. 1). The largest deviations from 90° are observed for N2—Mo1—N1 72.27 (3)° and C11—Mo1—N2 99.97 (4)°. The diimine ligand coordinates *via* the two nitrogen atoms to the metal center. The two carbonyl ligands coordinated perpendicular to the diimine ligand are slightly bent [Mo1—C12—O3 173.7 (1), Mo1—C14—O5 173.5 (1)°]. This deviation was expected as it has already been observed for similar tetra-carbonyl molybdenum(0) complexes containing 2-(pyridin-2-yl)benzoxazole (Datta *et al.*, 2011) or 2,6-bis[(4*S*)-isopropyl-oxazolin-2-yl]pyridine acting as bidentate ligands (Heard *et al.*, 1998).

In the crystal, molecules of the title compound are linked by weak intermolecular C—H···O interactions (Table 1).

Synthesis and crystallization

A mixture of $Mo(CO)_6$ (0.10 g, 0.38 mmol) and an equimolar amount of the diimine ligand (0.67 g, 0.38 mmol) in dry toluene was refluxed under argon atmosphere and light exclusion overnight resulting in a deep-red solution. The solvent was removed *in vacuo* and the red residue was washed three times with *n*-pentane and diethyl ether. Recrystallization from CH_2Cl_2/*n*-pentane 2:1 gave deep-red crystals suitable for X-ray crystal structure analysis. Yield 0.112 (76%). ^{1}**H NMR** (300 MHz, CD_2Cl_2, p.p.m.) δ = 9.05 (*d*, *J* =

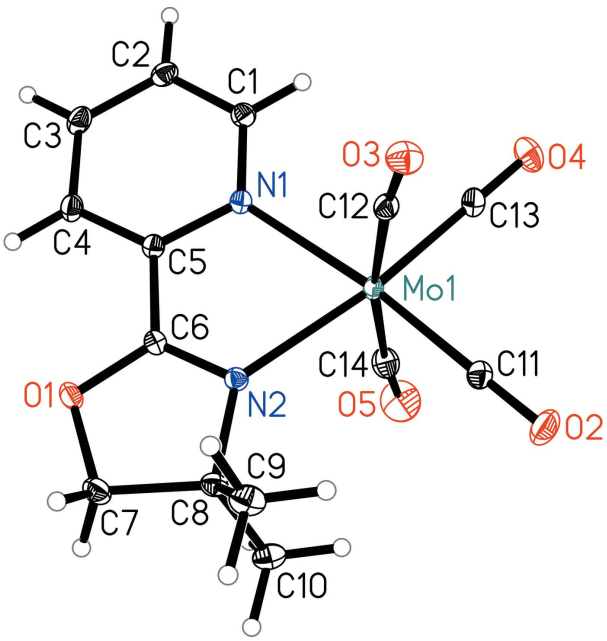


Figure 1
Molecular structure of the title compound showing the atom-labelling scheme. Displacement ellipsoids correspond to 30% probability.

5.3 Hz, 1H), 7.95 (*td*, *J* = 7.7 Hz, 1.6 Hz, 1H), 7.87–7.83 (*m*, 1H), 7.50 (*ddd*, *J* = 7.3 Hz, 5.8 Hz, 1.5 Hz, 1H), 4.52 (*s*, 2H), 1.56 (*s*, 6H); **^{13}C NMR** (75 MHz, $CDCl_3$, p.p.m.) δ = 256.8 (CO), 223.2 (CO), 203.7 (CO), 154.0 (C_{6Py}), 137.6 (C_{4Py}), 127.1 (C_{5Py}), 124.6 (C_{3Py}), 81.8 (C_{4Pyrox}), 68.5 (C_{5Pyrox}), 27.8 (CH_3), signals for the quaternary carbon atoms C_{2Py} and C_{2Pyrox} were not detectable as it was also observed for a highly related manganese complex with the same diimine ligand (Steinlechner *et al.*, 2019); **IR:** $\tilde{\nu}_{(CO)}$/cm^{-1} = 2010, 1891, 1851, 1810; **HR–MS (ESI):** calcd. mass $C_{13}H_{12}BrMoN_2O_4$: 357.98511; found: 357.98100; **elemental analysis:** (calculated) C:43.77, H: 3.15, N:7.29; (found) C: 43.77, H: 3.07, N: 7.43.

Refinement

Crystal data, data collection and structure refinement details are summarized in Table 2.

Funding information

CS acknowledges financial support from EU fund H2020-MSCA-ITN-2015 in Horizon 2020 as part of the NoNoMeCat (grant agreement No. 675020). The publication of this article was funded by the Open Access Fund of the Leibniz Association.

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

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C1—H1···O2^{i}	0.95	2.61	3.2786 (15)	128
C10—H10*A*···O4^{ii}	0.98	2.64	3.4880 (17)	145

Symmetry codes: (i) $x+\frac{1}{2}, -y+\frac{1}{2}, z+\frac{1}{2}$; (ii) $-x+\frac{3}{2}, y+\frac{1}{2}, -z+\frac{3}{2}$.

Table 2
Experimental details.

Crystal data	
Chemical formula	[Mo($C_{10}H_{12}N2O$)(CO)$_4$]
M_r	384.20
Crystal system, space group	Monoclinic, $P2_1/n$
Temperature (K)	150
a, *b*, *c* (Å)	8.8166 (3), 12.2837 (5), 14.0636 (6)
β (°)	99.3347 (11)
V (Å^3)	1502.93 (10)
Z	4
Radiation type	Mo *Kα*
μ (mm^{-1})	0.90
Crystal size (mm)	0.50 × 0.49 × 0.36
Data collection	
Diffractometer	Bruker APEXII CCD
Absorption correction	Multi-scan (*SADABS*; Bruker, 2014)
T_{min}, T_{max}	0.69, 0.74
No. of measured, independent and observed [$I > 2\sigma(I)$] reflections	29586, 3993, 3889
R_{int}	0.017
(sin $\theta/\lambda)_{max}$ (Å^{-1})	0.682
Refinement	
$R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, *S*	0.016, 0.043, 1.10
No. of reflections	3993
No. of parameters	201
H-atom treatment	H-atom parameters constrained
$\Delta\rho_{max}$, $\Delta\rho_{min}$ (e Å^{-3})	0.41, −0.44

Computer programs: *APEX2* (Bruker, 2014), *SAINT* (Bruker, 2013), *XP* in *SHELXTL* and *SHELXS97* (Sheldrick, 2008), *SHELXL2014* (Sheldrick, 2015) and *publCIF* (Westrip, 2010).

References

Bruker (2013). *SAINT*. Bruker AXS Inc., Madison, Wisconsin, USA.

Bruker (2014). *APEX2* and *SADABS*. Bruker AXS Inc., Madison, Wisconsin, USA.

Datta, P., Sardar, D., Mukhopadhyay, A. P., López-Torres, E., Pastor, C. J. & Sinha, C. (2011). *J. Organomet. Chem.* **696**, 488–495.

Heard, P. J. & Tocher, D. A. (1998). *J. Chem. Soc. Dalton Trans.* pp. 2169–2176.

Sheldrick, G. M. (2008). *Acta Cryst.* A**64**, 112–122.

Sheldrick, G. M. (2015). *Acta Cryst.* C**71**, 3–8.

Steinlechner, C., Roesel, A. F., Oberem, E., Päpcke, A., Rockstroh, N., Gloaguen, F., Lochbrunner, S., Ludwig, R., Spannenberg, A., Junge, H., Francke, R. & Beller, M. (2019). *ACS Catal.* pp. 2091–2100.

Westrip, S. P. (2010). *J. Appl. Cryst.* **43**, 920–925.

full crystallographic data

IUCrData (2019). **4**, x190283 [https://doi.org/10.1107/S2414314619002839]

Tetracarbonyl[4,4-dimethyl-2-(pyridin-2-yl)-2-oxazoline-κ^2N,N']molybdenum(0)

Christoph Steinlechner, Anke Spannenberg, Henrik Junge and Matthias Beller

Tetracarbonyl[4,4-dimethyl-2-(pyridin-2-yl)-2-oxazoline-κ^2N,N']molybdenum(0)

Crystal data

[Mo($C_{10}H_{12}$N2O)(CO)$_4$]
M_r = 384.20
Monoclinic, $P2_1/n$
a = 8.8166 (3) Å
b = 12.2837 (5) Å
c = 14.0636 (6) Å
β = 99.3347 (11)°
V = 1502.93 (10) Å^3
Z = 4
F(000) = 768
D_x = 1.698 Mg m^{-3}
Mo $K\alpha$ radiation, λ = 0.71073 Å
Cell parameters from 9837 reflections
θ = 2.2–30.6°
μ = 0.90 mm^{-1}
T = 150 K
Prism, red
0.50 × 0.49 × 0.36 mm

Data collection

Bruker APEXII CCD diffractometer
Radiation source: fine-focus sealed tube
Detector resolution: 8.3333 pixels mm^{-1}
φ and ω scans
Absorption correction: multi-scan (SADABS; Bruker, 2014)
T_{min} = 0.69, T_{max} = 0.74
29586 measured reflections
3993 independent reflections
3889 reflections with $I > 2\sigma(I)$
R_{int} = 0.017
θ_{max} = 29.0°, θ_{min} = 2.2°
h = −11→12
k = −16→16
l = −19→19

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.016
$wR(F^2)$ = 0.043
S = 1.10
3993 reflections
201 parameters
0 restraints
Hydrogen site location: inferred from neighbouring sites
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0202P)^2 + 0.6769P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.004
$\Delta\rho_{max}$ = 0.41 e Å^{-3}
$\Delta\rho_{min}$ = −0.44 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 (Å²)

	x	*y*	*z*	U_{iso}*/U_{eq}
C1	0.80476 (13)	0.45885 (9)	0.99223 (8)	0.0202 (2)
H1	0.8765	0.4015	1.0089	0.024*
C2	0.81678 (13)	0.55152 (10)	1.04953 (8)	0.0228 (2)
H2	0.8953	0.5569	1.1042	0.027*
C3	0.71361 (14)	0.63589 (10)	1.02638 (9)	0.0248 (2)
H3	0.7219	0.7008	1.0636	0.030*
C4	0.59747 (13)	0.62380 (9)	0.94757 (9)	0.0222 (2)
H4	0.5233	0.6795	0.9305	0.027*
C5	0.59241 (12)	0.52863 (9)	0.89456 (8)	0.01714 (19)
C6	0.47421 (12)	0.50561 (9)	0.81159 (8)	0.01724 (19)
C7	0.26379 (14)	0.53003 (10)	0.70282 (9)	0.0248 (2)
H7A	0.2551	0.5782	0.6458	0.030*
H7B	0.1597	0.5170	0.7183	0.030*
C8	0.34159 (12)	0.42100 (9)	0.68335 (8)	0.0191 (2)
C9	0.23763 (15)	0.32331 (11)	0.68853 (10)	0.0273 (2)
H9A	0.2944	0.2563	0.6801	0.041*
H9B	0.1481	0.3286	0.6374	0.041*
H9C	0.2033	0.3220	0.7514	0.041*
C10	0.40366 (16)	0.42340 (11)	0.58851 (9)	0.0282 (2)
H10A	0.4746	0.4848	0.5886	0.042*
H10B	0.3181	0.4317	0.5350	0.042*
H10C	0.4582	0.3553	0.5808	0.042*
C11	0.61991 (14)	0.18720 (10)	0.71624 (9)	0.0228 (2)
C12	0.57667 (13)	0.20789 (9)	0.89897 (8)	0.0206 (2)
C13	0.87057 (14)	0.23449 (10)	0.85597 (9)	0.0234 (2)
C14	0.78619 (15)	0.38044 (11)	0.70982 (10)	0.0288 (3)
Mo1	0.67341 (2)	0.30510 (2)	0.80894 (2)	0.01647 (4)
N1	0.69605 (10)	0.44698 (7)	0.91437 (7)	0.01694 (17)
N2	0.47339 (10)	0.41686 (7)	0.76431 (7)	0.01688 (17)
O1	0.36316 (10)	0.57885 (7)	0.78477 (6)	0.02363 (17)
O2	0.58250 (12)	0.11532 (8)	0.66411 (7)	0.0349 (2)
O3	0.52810 (11)	0.14502 (8)	0.94630 (7)	0.03051 (19)
O4	0.99039 (12)	0.19647 (8)	0.88247 (8)	0.0361 (2)
O5	0.85256 (15)	0.41239 (12)	0.65282 (10)	0.0525 (3)

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.0178 (5)	0.0222 (5)	0.0203 (5)	0.0002 (4)	0.0024 (4)	−0.0003 (4)
C2	0.0204 (5)	0.0283 (6)	0.0193 (5)	−0.0023 (4)	0.0016 (4)	−0.0047 (4)
C3	0.0249 (5)	0.0240 (5)	0.0253 (6)	−0.0021 (4)	0.0038 (4)	−0.0094 (4)
C4	0.0218 (5)	0.0193 (5)	0.0257 (5)	0.0014 (4)	0.0038 (4)	−0.0049 (4)
C5	0.0160 (4)	0.0168 (5)	0.0188 (5)	−0.0004 (4)	0.0032 (4)	−0.0013 (4)
C6	0.0160 (4)	0.0162 (5)	0.0195 (5)	0.0011 (4)	0.0030 (4)	0.0008 (4)
C7	0.0201 (5)	0.0238 (5)	0.0277 (6)	0.0024 (4)	−0.0041 (4)	−0.0006 (4)

C8	0.0188 (5)	0.0186 (5)	0.0186 (5)	−0.0018 (4)	−0.0007 (4)	0.0009 (4)
C9	0.0239 (6)	0.0263 (6)	0.0299 (6)	−0.0083 (5)	−0.0015 (5)	0.0019 (5)
C10	0.0324 (6)	0.0332 (6)	0.0187 (5)	−0.0029 (5)	0.0031 (5)	0.0012 (5)
C11	0.0219 (5)	0.0238 (5)	0.0229 (5)	0.0034 (4)	0.0039 (4)	−0.0025 (4)
C12	0.0191 (5)	0.0209 (5)	0.0206 (5)	0.0018 (4)	−0.0002 (4)	−0.0049 (4)
C13	0.0237 (5)	0.0202 (5)	0.0268 (6)	0.0023 (4)	0.0053 (4)	−0.0028 (4)
C14	0.0244 (6)	0.0292 (6)	0.0338 (6)	0.0018 (5)	0.0074 (5)	0.0004 (5)
Mo1	0.01612 (5)	0.01496 (5)	0.01826 (5)	0.00169 (3)	0.00256 (3)	−0.00258 (3)
N1	0.0160 (4)	0.0164 (4)	0.0186 (4)	−0.0001 (3)	0.0034 (3)	−0.0015 (3)
N2	0.0157 (4)	0.0169 (4)	0.0175 (4)	0.0000 (3)	0.0011 (3)	−0.0004 (3)
O1	0.0221 (4)	0.0188 (4)	0.0278 (4)	0.0061 (3)	−0.0028 (3)	−0.0021 (3)
O2	0.0370 (5)	0.0319 (5)	0.0348 (5)	0.0000 (4)	0.0031 (4)	−0.0164 (4)
O3	0.0329 (5)	0.0296 (5)	0.0291 (5)	−0.0049 (4)	0.0050 (4)	0.0036 (4)
O4	0.0253 (5)	0.0372 (6)	0.0451 (6)	0.0118 (4)	0.0038 (4)	0.0035 (4)
O5	0.0479 (7)	0.0630 (8)	0.0537 (7)	−0.0011 (6)	0.0295 (6)	0.0126 (6)

Geometric parameters (Å, º)

C1—N1	1.3411 (14)	C8—C9	1.5190 (16)
C1—C2	1.3888 (16)	C8—C10	1.5219 (16)
C1—H1	0.9500	C9—H9A	0.9800
C2—C3	1.3825 (17)	C9—H9B	0.9800
C2—H2	0.9500	C9—H9C	0.9800
C3—C4	1.3895 (16)	C10—H10A	0.9800
C3—H3	0.9500	C10—H10B	0.9800
C4—C5	1.3833 (15)	C10—H10C	0.9800
C4—H4	0.9500	C11—O2	1.1608 (15)
C5—N1	1.3550 (14)	C11—Mo1	1.9545 (12)
C5—C6	1.4605 (15)	C12—O3	1.1469 (15)
C6—N2	1.2764 (14)	C12—Mo1	2.0265 (12)
C6—O1	1.3381 (13)	C13—O4	1.1598 (16)
C7—O1	1.4587 (14)	C13—Mo1	1.9592 (12)
C7—C8	1.5492 (16)	C14—O5	1.1363 (18)
C7—H7A	0.9900	C14—Mo1	2.0587 (13)
C7—H7B	0.9900	Mo1—N2	2.2427 (9)
C8—N2	1.4899 (13)	Mo1—N1	2.2758 (9)
N1—C1—C2	122.69 (11)	H9A—C9—H9C	109.5
N1—C1—H1	118.7	H9B—C9—H9C	109.5
C2—C1—H1	118.7	C8—C10—H10A	109.5
C3—C2—C1	119.50 (11)	C8—C10—H10B	109.5
C3—C2—H2	120.2	H10A—C10—H10B	109.5
C1—C2—H2	120.2	C8—C10—H10C	109.5
C2—C3—C4	118.64 (11)	H10A—C10—H10C	109.5
C2—C3—H3	120.7	H10B—C10—H10C	109.5
C4—C3—H3	120.7	O2—C11—Mo1	176.39 (11)
C5—C4—C3	118.38 (11)	O3—C12—Mo1	173.73 (10)
C5—C4—H4	120.8	O4—C13—Mo1	177.11 (11)

C3—C4—H4	120.8	O5—C14—Mo1	173.48 (13)
N1—C5—C4	123.57 (10)	C11—Mo1—C13	90.12 (5)
N1—C5—C6	113.01 (9)	C11—Mo1—C12	84.24 (5)
C4—C5—C6	123.41 (10)	C13—Mo1—C12	88.20 (5)
N2—C6—O1	119.03 (10)	C11—Mo1—C14	88.40 (5)
N2—C6—C5	121.64 (10)	C13—Mo1—C14	85.87 (5)
O1—C6—C5	119.32 (9)	C12—Mo1—C14	170.54 (5)
O1—C7—C8	105.58 (9)	C11—Mo1—N2	99.97 (4)
O1—C7—H7A	110.6	C13—Mo1—N2	168.48 (4)
C8—C7—H7A	110.6	C12—Mo1—N2	98.28 (4)
O1—C7—H7B	110.6	C14—Mo1—N2	88.83 (4)
C8—C7—H7B	110.6	C11—Mo1—N1	171.17 (4)
H7A—C7—H7B	108.8	C13—Mo1—N1	98.04 (4)
N2—C8—C9	109.77 (9)	C12—Mo1—N1	92.61 (4)
N2—C8—C10	108.88 (9)	C14—Mo1—N1	95.51 (5)
C9—C8—C10	111.28 (10)	N2—Mo1—N1	72.27 (3)
N2—C8—C7	101.97 (9)	C1—N1—C5	117.14 (9)
C9—C8—C7	112.74 (10)	C1—N1—Mo1	126.21 (7)
C10—C8—C7	111.75 (10)	C5—N1—Mo1	116.61 (7)
C8—C9—H9A	109.5	C6—N2—C8	107.86 (9)
C8—C9—H9B	109.5	C6—N2—Mo1	116.06 (7)
H9A—C9—H9B	109.5	C8—N2—Mo1	135.66 (7)
C8—C9—H9C	109.5	C6—O1—C7	105.34 (9)

N1—C1—C2—C3	−0.11 (18)	C4—C5—N1—Mo1	−175.19 (9)
C1—C2—C3—C4	1.91 (18)	C6—C5—N1—Mo1	5.07 (11)
C2—C3—C4—C5	−1.37 (18)	O1—C6—N2—C8	2.20 (13)
C3—C4—C5—N1	−1.03 (17)	C5—C6—N2—C8	−178.33 (9)
C3—C4—C5—C6	178.69 (11)	O1—C6—N2—Mo1	175.93 (8)
N1—C5—C6—N2	−0.35 (15)	C5—C6—N2—Mo1	−4.60 (13)
C4—C5—C6—N2	179.90 (11)	C9—C8—N2—C6	−123.82 (11)
N1—C5—C6—O1	179.12 (9)	C10—C8—N2—C6	114.14 (11)
C4—C5—C6—O1	−0.63 (16)	C7—C8—N2—C6	−4.08 (11)
O1—C7—C8—N2	4.53 (11)	C9—C8—N2—Mo1	64.25 (13)
O1—C7—C8—C9	122.16 (10)	C10—C8—N2—Mo1	−57.79 (13)
O1—C7—C8—C10	−111.62 (11)	C7—C8—N2—Mo1	−176.01 (8)
C2—C1—N1—C5	−2.20 (16)	N2—C6—O1—C7	0.96 (14)
C2—C1—N1—Mo1	175.57 (8)	C5—C6—O1—C7	−178.52 (10)
C4—C5—N1—C1	2.79 (16)	C8—C7—O1—C6	−3.51 (12)
C6—C5—N1—C1	−176.95 (9)		

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C1—H1···O2[i]	0.95	2.61	3.2786 (15)	128
C10—H10*A*···O4[ii]	0.98	2.64	3.4880 (17)	145

Symmetry codes: (i) *x*+1/2, −*y*+1/2, *z*+1/2; (ii) −*x*+3/2, *y*+1/2, −*z*+3/2.