Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

(2,2′-Bipyridyl)tricarbonyl(di-2-pyridylamine)molybdenum(0) revisited

Kathleen J. Muir, Geoffrey P. McQuillan and William T. A. Harrison*

Department of Chemistry, University of Aberdeen, Meston Walk, Aberdeen AB24 3UE, Scotland
Correspondence e-mail: w.harrison@abdn.ac.uk

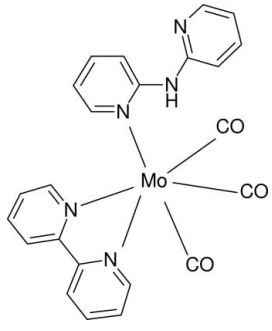
Received 26 October 2007; accepted 30 October 2007

Key indicators: single-crystal X-ray study; T = 298 K; mean σ(C–C) = 0.004 Å; R factor = 0.021; wR factor = 0.061; data-to-parameter ratio = 13.1.

The previous structure report [Howie & McQuillan (1986). *J. Chem. Soc. Dalton Trans*. pp. 759–764] of the title compound, $[Mo(C_{10}H_8N_2)(C_{10}H_9N_3)(CO)_3]$, has been corrected. In the present structure, the precision of the Mo—C and Mo—N bond distances for the *fac*-MoC_3N_3 centre are significantly improved, and an N atom and a C—H group have been exchanged. Possible intramolecular N—H···C and C—H···N and intermolecular C—H···O interactions are described.

Related literature

For the previous structure, see: Howie & McQuillan (1986). For a related structure, see: Braga *et al.* (2007). For background, see: Chisholm *et al.* (1981); Muir *et al.* (2007). For reference structural data, see: Allen *et al.* (1987).



Experimental

Crystal data

$[Mo(C_{10}H_8N_2)(C_{10}H_9N_3)(CO)_3]$
M_r = 507.36
Monoclinic, $P2_1/c$
a = 10.6273 (4) Å
b = 12.9092 (5) Å
c = 16.5581 (7) Å
β = 107.728 (1)°
V = 2163.73 (15) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.64 mm^{-1}
T = 298 (2) K
0.28 × 0.17 × 0.15 mm

Data collection

Bruker SMART1000 CCD diffractometer
Absorption correction: multi-scan (*SADABS*; Bruker, 1999)
T_{min} = 0.841, T_{max} = 0.910
12826 measured reflections
3811 independent reflections
3067 reflections with $I > 2\sigma(I)$
R_{int} = 0.023

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.021
$wR(F^2)$ = 0.061
S = 1.06
3811 reflections
292 parameters
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.26 e Å^{-3}
$\Delta\rho_{min}$ = −0.30 e Å^{-3}

Table 1
Selected bond lengths (Å).

Mo1—C1	1.917 (2)	Mo1—N2	2.2465 (16)
Mo1—C2	1.946 (2)	Mo1—N1	2.2631 (16)
Mo1—C3	1.947 (2)	Mo1—N3	2.3626 (16)

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

D—H···A	D—H	H···A	D···A	D—H···A
N4—H1···C3	0.75 (2)	2.40 (2)	2.978 (3)	135 (2)
C17—H17···N5	0.93	2.36	2.902 (3)	117
C10—H10···O1^{i}	0.93	2.43	3.318 (3)	160
C16—H16···O1^{ii}	0.93	2.38	3.305 (3)	172

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

Data collection: *SMART* (Bruker, 1999); cell refinement: *SAINT* (Bruker, 1999); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *ORTEP-3* (Farrugia, 1997); software used to prepare material for publication: *SHELXL97*.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: LH2540).

References

Allen, F. H., Kennard, O., Watson, D. G., Brammer, L., Orpen, A. G. & Taylor, R. (1987). *J. Chem. Soc. Perkin Trans. 2*, pp. S1–19.
Braga, S. S., Coelho, A. C., Gonçalves, I. S. & Almeida Paz, F. A. (2007). *Acta Cryst.* E**63**, m780–m782.
Bruker (1999). *SMART*, *SAINT* and *SADABS*. Bruker AXS Inc., Madison, Wisconsin, USA.
Chisholm, M. H., Huffman, J. C., Rothwell, I. P., Kress, N. & Woodruff, W. H. (1981). *J. Am. Chem. Soc.* **103**, 4945–4947.
Farrugia, L. J. (1997). *J. Appl. Cryst.* **30**, 565.
Howie, R. A. & McQuillan, G. P. (1986). *J. Chem. Soc. Dalton Trans.* pp. 759–764.
Muir, K. J., McQuillan, G. P. & Harrison, W. T. A. (2007). Unpublished work.
Sheldrick, G. M. (1997). *SHELXS97* and *SHELXL97*. University of Göttingen, Germany.

  doi:10.1107/S160053680705461X

supplementary materials

Acta Cryst. (2007). E**63**, m2918 [**doi:10.1107/S160053680705461X**]

(2,2'-Bipyridyl)tricarbonyl(di-2-pyridylamine)molybdenum(0) revisited

K. J. Muir, G. P. McQuillan and W. T. A. Harrison

Comment

The synthesis and structure of the title compound, (I), $Mo(CO)_3$(bipy)(dipyam) (bipy = 2,2-bipyridine, $C_{10}H_8N_2$; dipyam = di-2-pyridylamine, $C_{10}H_9N_3$) was reported by Howie & McQuillan (1986). We report here a corrected structure, in which an N atom and C—H group are exchanged (Fig. 1). The previous determination did not locate the H atoms, and swapped atoms C20 and N5 (using the present numbering scheme). The revised structure is supported by (i) a lower *R*-factor (compared to swapped N and C atoms); (ii) a significant difference peak near C20 corresponding to an H atom, but little or no evidence of such a feature near N5; (iii) the plausible formation of an intramolecular C17—H17···N5 interaction; (iv) the C19—N5 bond length of 1.320 (3)Å found here would be extremely short for an aromatic C—C bond, but is quite acceptable for an aromatic C—N bond.

Otherwise, the structure of (I) is similar to the previous determination with improved precision [*e.g.* σ values for the Mo—N bonds in (I) = 0.0016Å compared to 0.006Å in the previous study], and features a distorted *mer*-MoC_3N_3 octahedron for the metal atom (Table 1). Mo1—C1 is slightly shorter than the other two Mo—C distances, perhaps because the weak π* acceptor capabilities of the N atoms of the bipy and bonded dipyam rings are slightly different (Chisholm *et al.*, 1981).

The N—Mo—N bite angle for the bipy molecule in (I) is 71.86 (6)°, which is typical (Braga *et al.*, 2007). The dihedral angle between the N1/C4—C8 and N2/C9—C13 ring systems of 3.56 (16)° indicates a small degree of twisting about the linking C8—C9 bond. Even so, Mo1 is significantly displaced from both the N1/C4—C8 and N2/C9—C13 ring planes, by 0.329 (3)Å and 0.168 (3) Å, respectively. The dihedral angle between the N3/C14—C18 and N5/C19—C23 ring planes is 24.91 (9)°. Otherwise, all the organic bond lengths and angles in (I) may be regarded as normal (Allen *et al.*, 1987).

The most unusual feature of (I) is a short intramolecular N—H···C distance of 2.40 (2)Å (Table 2). The van der Waals radius sum for H and C is 2.90 Å. This novel interaction – or incidental steric contact – will be discussed in more detail elsewhere (Muir *et al.*, 2007).

In the crystal of (I), two relatively short C—H···O interactions (Table 2) may help to establish the packing. Any aromatic π-π stacking in (I) must be weak, with a minimum centroid-centroid separation of 3.94 Å.

Experimental

The title compound was prepared by the method of Howie & McQuillan (1986), to result in dark green facted chunks of (I).

Refinement

The N-bound H atom was located in a difference map and its position was freely refined with U_{iso}(H) = 1.2U_{eq}(C). The C-bound hydrogen atoms were geometrically placed (C—H = 0.93 Å) and refined as riding with U_{iso}(H) = 1.2U_{eq}(C).

Figures

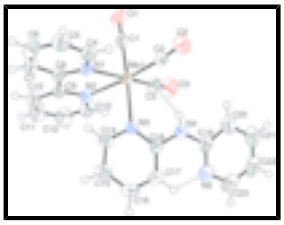


Fig. 1. View of the molecular structure of (I) showing 50% displacement ellipsoids (arbitrary spheres for the H atoms). The C—H···N and N—H···C contacts are indicated by double-dashed lines.

(2,2'-Bipyridyl)tricarbonyl(di-2-pyridylamine)molybdenum(0)

Crystal data

$[Mo(C_{10}H_8N_2)(C_{10}H_9N_3)(CO)_3]$	F_{000} = 1024
M_r = 507.36	D_x = 1.557 Mg m^{-3}
Monoclinic, $P2_1/c$	Mo $K\alpha$ radiation λ = 0.71073 Å
Hall symbol: -P 2ybc	Cell parameters from 6454 reflections
a = 10.6273 (4) Å	θ = 2.6–25.0º
b = 12.9092 (5) Å	μ = 0.64 mm^{-1}
c = 16.5581 (7) Å	T = 298 (2) K
β = 107.728 (1)º	Faceted chunk, dark green
V = 2163.73 (15) Å^3	0.28 × 0.17 × 0.15 mm
Z = 4	

Data collection

Bruker SMART1000 CCD diffractometer	3811 independent reflections
Radiation source: fine-focus sealed tube	3067 reflections with $I > 2\sigma(I)$
Monochromator: graphite	R_{int} = 0.023
T = 298(2) K	θ_{max} = 25.0º
ω scans	θ_{min} = 2.0º
Absorption correction: multi-scan (SADABS; Bruker, 1999)	h = −12→12
T_{min} = 0.841, T_{max} = 0.910	k = −15→8
12826 measured reflections	l = −19→19

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: difmap and geom
$R[F^2 > 2\sigma(F^2)]$ = 0.021	H atoms treated by a mixture of independent and constrained refinement
$wR(F^2)$ = 0.061	$w = 1/[\sigma^2(F_o^2) + (0.0355P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
S = 1.06	$(\Delta/\sigma)_{max} < 0.001$

3811 reflections	$\Delta\rho_{max} = 0.26$ e Å^{-3}
292 parameters	$\Delta\rho_{min} = -0.30$ e Å^{-3}
Primary atom site location: structure-invariant direct methods	Extinction correction: none

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.

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 $F^2 > 2\text{sigma}(F^2)$ 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.

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

	x	*y*	*z*	U_{iso}*/U_{eq}
Mo1	0.293621 (15)	0.246228 (12)	0.062933 (10)	0.03100 (7)
C1	0.4599 (2)	0.20447 (19)	0.14032 (14)	0.0466 (5)
O1	0.56041 (18)	0.17911 (17)	0.18766 (11)	0.0830 (7)
C2	0.3533 (2)	0.38617 (17)	0.09857 (14)	0.0441 (5)
O2	0.39753 (18)	0.46686 (13)	0.12363 (13)	0.0774 (6)
C3	0.2294 (2)	0.23659 (16)	0.16098 (15)	0.0434 (5)
O3	0.1968 (2)	0.22860 (15)	0.22132 (12)	0.0708 (5)
C4	0.4438 (2)	0.29588 (18)	−0.07405 (13)	0.0430 (5)
H4	0.4450	0.3628	−0.0531	0.052*
C5	0.5102 (2)	0.2769 (2)	−0.13158 (15)	0.0514 (6)
H5	0.5549	0.3298	−0.1494	0.062*
C6	0.5094 (2)	0.1782 (2)	−0.16237 (15)	0.0569 (6)
H6	0.5551	0.1630	−0.2007	0.068*
C7	0.4403 (2)	0.10208 (18)	−0.13597 (14)	0.0490 (6)
H7	0.4374	0.0351	−0.1571	0.059*
C8	0.37499 (19)	0.12607 (15)	−0.07757 (12)	0.0359 (4)
C9	0.29805 (19)	0.04888 (15)	−0.04587 (12)	0.0367 (4)
C10	0.2805 (2)	−0.05268 (17)	−0.07389 (16)	0.0565 (6)
H10	0.3158	−0.0751	−0.1158	0.068*
C11	0.2103 (3)	−0.1206 (2)	−0.03941 (19)	0.0695 (8)
H11	0.1989	−0.1893	−0.0572	0.083*
C12	0.1579 (3)	−0.08492 (19)	0.02146 (18)	0.0646 (7)
H12	0.1095	−0.1288	0.0454	0.078*
C13	0.1779 (2)	0.01607 (17)	0.04632 (15)	0.0492 (6)
H13	0.1417	0.0395	0.0875	0.059*
C14	0.0659 (2)	0.25571 (15)	−0.11820 (13)	0.0408 (5)
H14	0.1342	0.2228	−0.1322	0.049*
C15	−0.0520 (2)	0.26647 (17)	−0.18039 (14)	0.0472 (5)

H15	−0.0637	0.2406	−0.2346	0.057*
C16	−0.1531 (2)	0.31653 (19)	−0.16073 (14)	0.0510 (6)
H16	−0.2348	0.3243	−0.2016	0.061*
C17	−0.1328 (2)	0.35484 (17)	−0.08060 (14)	0.0445 (5)
H17	−0.2000	0.3896	−0.0667	0.053*
C18	−0.01035 (19)	0.34098 (14)	−0.02039 (12)	0.0343 (4)
C19	−0.0613 (2)	0.41246 (15)	0.10733 (14)	0.0420 (5)
C20	−0.0031 (3)	0.45981 (18)	0.18505 (15)	0.0553 (6)
H20	0.0880	0.4679	0.2059	0.066*
C21	−0.0838 (3)	0.4944 (2)	0.23039 (18)	0.0704 (8)
H21	−0.0478	0.5280	0.2820	0.084*
C22	−0.2169 (3)	0.4793 (2)	0.1995 (2)	0.0757 (9)
H22	−0.2731	0.5029	0.2289	0.091*
C23	−0.2650 (3)	0.4286 (3)	0.1243 (2)	0.0771 (9)
H23	−0.3556	0.4170	0.1039	0.093*
N1	0.37697 (16)	0.22289 (13)	−0.04629 (10)	0.0338 (4)
N2	0.24692 (16)	0.08371 (12)	0.01458 (10)	0.0362 (4)
N3	0.09003 (15)	0.28956 (13)	−0.03790 (10)	0.0334 (4)
N4	0.02063 (17)	0.38006 (14)	0.06062 (12)	0.0425 (4)
H1	0.092 (2)	0.3747 (18)	0.0863 (15)	0.051*
N5	−0.18931 (19)	0.39405 (18)	0.07744 (13)	0.0613 (6)

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Mo1	0.02958 (11)	0.03322 (11)	0.03088 (11)	0.00259 (7)	0.01021 (7)	−0.00083 (7)
C1	0.0443 (13)	0.0572 (14)	0.0381 (12)	0.0153 (11)	0.0123 (10)	−0.0112 (11)
O1	0.0575 (11)	0.1274 (18)	0.0493 (10)	0.0485 (12)	−0.0058 (9)	−0.0205 (11)
C2	0.0337 (12)	0.0440 (13)	0.0542 (13)	0.0039 (10)	0.0129 (10)	−0.0072 (11)
O2	0.0640 (12)	0.0469 (11)	0.1135 (16)	−0.0102 (9)	0.0157 (11)	−0.0252 (11)
C3	0.0446 (13)	0.0456 (13)	0.0414 (12)	0.0071 (10)	0.0152 (10)	0.0041 (10)
O3	0.0838 (14)	0.0897 (14)	0.0520 (11)	0.0104 (10)	0.0404 (10)	0.0140 (10)
C4	0.0430 (12)	0.0423 (12)	0.0446 (12)	−0.0060 (10)	0.0148 (10)	0.0014 (10)
C5	0.0495 (14)	0.0606 (15)	0.0492 (14)	−0.0088 (11)	0.0224 (11)	0.0077 (12)
C6	0.0608 (16)	0.0700 (17)	0.0523 (15)	0.0061 (13)	0.0359 (13)	0.0064 (13)
C7	0.0567 (14)	0.0491 (13)	0.0484 (13)	0.0056 (11)	0.0267 (11)	−0.0032 (11)
C8	0.0349 (11)	0.0383 (11)	0.0348 (11)	0.0042 (9)	0.0111 (9)	0.0010 (9)
C9	0.0379 (11)	0.0334 (10)	0.0400 (11)	0.0035 (8)	0.0133 (9)	0.0010 (9)
C10	0.0677 (16)	0.0409 (13)	0.0696 (16)	−0.0010 (12)	0.0338 (14)	−0.0110 (12)
C11	0.085 (2)	0.0335 (13)	0.096 (2)	−0.0095 (12)	0.0367 (18)	−0.0079 (14)
C12	0.0745 (18)	0.0425 (14)	0.0856 (19)	−0.0114 (12)	0.0373 (16)	0.0078 (14)
C13	0.0529 (14)	0.0426 (13)	0.0602 (14)	−0.0023 (10)	0.0289 (12)	0.0056 (11)
C14	0.0407 (12)	0.0436 (12)	0.0388 (12)	0.0034 (9)	0.0130 (9)	0.0007 (10)
C15	0.0467 (13)	0.0551 (14)	0.0360 (12)	−0.0026 (10)	0.0068 (10)	−0.0013 (10)
C16	0.0344 (12)	0.0646 (16)	0.0466 (13)	−0.0024 (11)	0.0016 (10)	0.0084 (12)
C17	0.0331 (12)	0.0490 (13)	0.0512 (13)	0.0056 (9)	0.0123 (10)	0.0047 (11)
C18	0.0329 (11)	0.0283 (10)	0.0424 (12)	−0.0008 (8)	0.0127 (9)	0.0009 (9)
C19	0.0472 (13)	0.0297 (11)	0.0547 (13)	0.0061 (9)	0.0237 (11)	0.0007 (10)

C20	0.0629 (16)	0.0502 (14)	0.0593 (15)	−0.0063 (12)	0.0282 (13)	−0.0088 (12)
C21	0.094 (2)	0.0616 (17)	0.0717 (18)	−0.0064 (15)	0.0492 (17)	−0.0165 (14)
C22	0.085 (2)	0.0731 (19)	0.090 (2)	0.0147 (16)	0.0577 (19)	−0.0087 (17)
C23	0.0532 (16)	0.097 (2)	0.092 (2)	0.0115 (15)	0.0374 (16)	−0.0075 (19)
N1	0.0320 (9)	0.0344 (9)	0.0361 (9)	−0.0006 (7)	0.0123 (7)	−0.0003 (7)
N2	0.0376 (9)	0.0333 (9)	0.0394 (9)	0.0016 (7)	0.0145 (8)	0.0031 (7)
N3	0.0317 (9)	0.0330 (8)	0.0363 (9)	−0.0003 (7)	0.0116 (7)	0.0011 (7)
N4	0.0336 (10)	0.0474 (11)	0.0454 (11)	0.0060 (9)	0.0104 (8)	−0.0069 (9)
N5	0.0445 (12)	0.0743 (15)	0.0701 (14)	0.0044 (10)	0.0251 (11)	−0.0134 (12)

Geometric parameters (Å, °)

Mo1—C1	1.917 (2)	C12—H12	0.9300
Mo1—C2	1.946 (2)	C13—N2	1.346 (3)
Mo1—C3	1.947 (2)	C13—H13	0.9300
Mo1—N2	2.2465 (16)	C14—N3	1.348 (3)
Mo1—N1	2.2631 (16)	C14—C15	1.365 (3)
Mo1—N3	2.3626 (16)	C14—H14	0.9300
C1—O1	1.163 (3)	C15—C16	1.375 (3)
C2—O2	1.166 (3)	C15—H15	0.9300
C3—O3	1.158 (3)	C16—C17	1.370 (3)
C4—N1	1.342 (3)	C16—H16	0.9300
C4—C5	1.369 (3)	C17—C18	1.389 (3)
C4—H4	0.9300	C17—H17	0.9300
C5—C6	1.371 (3)	C18—N3	1.360 (2)
C5—H5	0.9300	C18—N4	1.376 (3)
C6—C7	1.376 (3)	C19—N5	1.320 (3)
C6—H6	0.9300	C19—C20	1.388 (3)
C7—C8	1.386 (3)	C19—N4	1.393 (3)
C7—H7	0.9300	C20—C21	1.375 (3)
C8—N1	1.351 (2)	C20—H20	0.9300
C8—C9	1.483 (3)	C21—C22	1.363 (4)
C9—N2	1.353 (2)	C21—H21	0.9300
C9—C10	1.384 (3)	C22—C23	1.360 (4)
C10—C11	1.383 (3)	C22—H22	0.9300
C10—H10	0.9300	C23—N5	1.352 (3)
C11—C12	1.372 (4)	C23—H23	0.9300
C11—H11	0.9300	N4—H1	0.75 (2)
C12—C13	1.364 (3)		
C1—Mo1—C2	84.81 (10)	N2—C13—H13	118.1
C1—Mo1—C3	84.86 (9)	C12—C13—H13	118.1
C2—Mo1—C3	88.34 (9)	N3—C14—C15	124.5 (2)
C1—Mo1—N2	91.96 (8)	N3—C14—H14	117.8
C2—Mo1—N2	172.47 (7)	C15—C14—H14	117.8
C3—Mo1—N2	98.16 (7)	C14—C15—C16	118.2 (2)
C1—Mo1—N1	89.51 (7)	C14—C15—H15	120.9
C2—Mo1—N1	101.27 (7)	C16—C15—H15	120.9
C3—Mo1—N1	168.40 (7)	C17—C16—C15	119.7 (2)
N2—Mo1—N1	71.86 (6)	C17—C16—H16	120.2

C1—Mo1—N3	176.56 (7)	C15—C16—H16	120.2
C2—Mo1—N3	97.94 (7)	C16—C17—C18	119.1 (2)
C3—Mo1—N3	97.25 (8)	C16—C17—H17	120.5
N2—Mo1—N3	85.06 (6)	C18—C17—H17	120.5
N1—Mo1—N3	87.93 (6)	N3—C18—N4	114.60 (17)
O1—C1—Mo1	179.6 (2)	N3—C18—C17	122.26 (18)
O2—C2—Mo1	175.0 (2)	N4—C18—C17	123.12 (18)
O3—C3—Mo1	176.8 (2)	N5—C19—C20	123.0 (2)
N1—C4—C5	123.4 (2)	N5—C19—N4	119.0 (2)
N1—C4—H4	118.3	C20—C19—N4	118.0 (2)
C5—C4—H4	118.3	C21—C20—C19	118.2 (2)
C4—C5—C6	118.6 (2)	C21—C20—H20	120.9
C4—C5—H5	120.7	C19—C20—H20	120.9
C6—C5—H5	120.7	C22—C21—C20	119.8 (3)
C5—C6—C7	119.3 (2)	C22—C21—H21	120.1
C5—C6—H6	120.4	C20—C21—H21	120.1
C7—C6—H6	120.4	C23—C22—C21	118.0 (2)
C6—C7—C8	119.4 (2)	C23—C22—H22	121.0
C6—C7—H7	120.3	C21—C22—H22	121.0
C8—C7—H7	120.3	N5—C23—C22	124.1 (3)
N1—C8—C7	121.44 (19)	N5—C23—H23	118.0
N1—C8—C9	115.60 (16)	C22—C23—H23	118.0
C7—C8—C9	122.96 (19)	C4—N1—C8	117.80 (17)
N2—C9—C10	121.29 (19)	C4—N1—Mo1	123.83 (14)
N2—C9—C8	115.31 (17)	C8—N1—Mo1	117.73 (12)
C10—C9—C8	123.40 (18)	C13—N2—C9	117.56 (18)
C11—C10—C9	119.8 (2)	C13—N2—Mo1	123.65 (14)
C11—C10—H10	120.1	C9—N2—Mo1	118.63 (13)
C9—C10—H10	120.1	C14—N3—C18	116.25 (17)
C12—C11—C10	118.8 (2)	C14—N3—Mo1	118.60 (13)
C12—C11—H11	120.6	C18—N3—Mo1	125.01 (12)
C10—C11—H11	120.6	C18—N4—C19	130.30 (18)
C13—C12—C11	118.8 (2)	C18—N4—H1	114.7 (18)
C13—C12—H12	120.6	C19—N4—H1	114.1 (18)
C11—C12—H12	120.6	C19—N5—C23	116.8 (2)
N2—C13—C12	123.8 (2)		
C6—C7—C8—N1	0.4 (3)	N3—Mo1—N1—C8	−93.96 (14)
C6—C7—C8—C9	179.9 (2)	C12—C13—N2—C9	0.5 (3)
N1—C8—C9—N2	−4.2 (3)	C12—C13—N2—Mo1	−174.87 (19)
C7—C8—C9—N2	176.34 (19)	C10—C9—N2—C13	−0.2 (3)
N1—C8—C9—C10	177.0 (2)	C8—C9—N2—C13	−179.04 (18)
C7—C8—C9—C10	−2.4 (3)	C10—C9—N2—Mo1	175.43 (16)
N2—C9—C10—C11	−0.5 (4)	C8—C9—N2—Mo1	−3.4 (2)
C8—C9—C10—C11	178.2 (2)	C1—Mo1—N2—C13	92.67 (18)
C9—C10—C11—C12	0.9 (4)	C3—Mo1—N2—C13	7.58 (19)
C10—C11—C12—C13	−0.6 (4)	N1—Mo1—N2—C13	−178.49 (18)
C11—C12—C13—N2	−0.1 (4)	N3—Mo1—N2—C13	−89.06 (17)
N3—C14—C15—C16	1.0 (3)	C1—Mo1—N2—C9	−82.70 (15)
C14—C15—C16—C17	0.7 (3)	C3—Mo1—N2—C9	−167.79 (15)

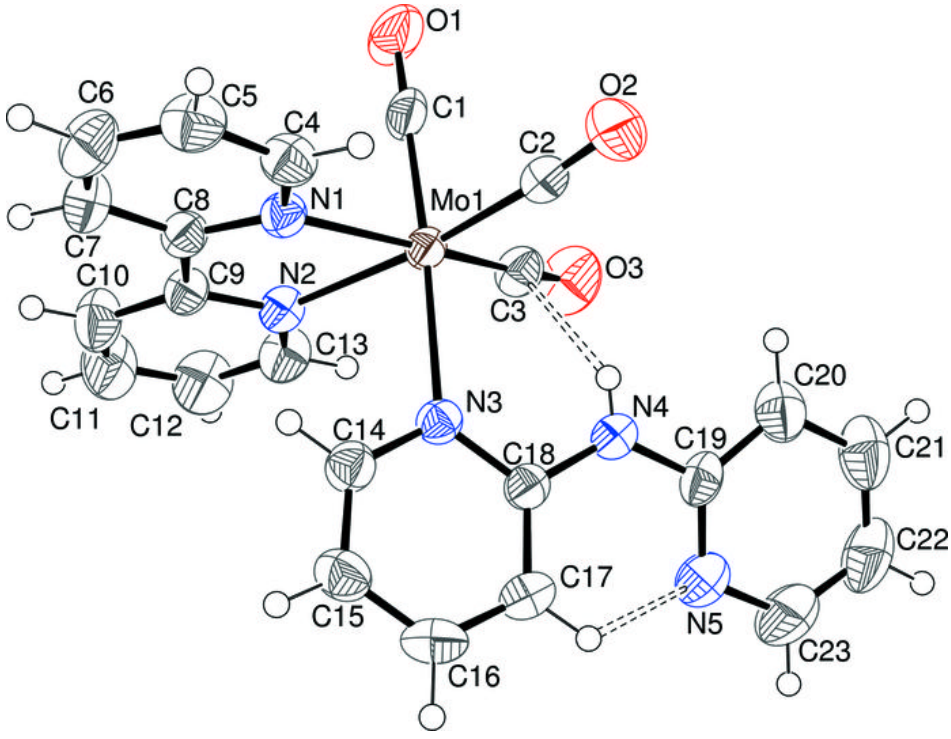
C15—C16—C17—C18	−0.8 (3)	N1—Mo1—N2—C9	6.14 (14)
C16—C17—C18—N3	−0.7 (3)	N3—Mo1—N2—C9	95.57 (14)
C16—C17—C18—N4	177.5 (2)	C15—C14—N3—C18	−2.5 (3)
N5—C19—C20—C21	−3.9 (4)	C15—C14—N3—Mo1	173.32 (16)
N4—C19—C20—C21	178.3 (2)	N4—C18—N3—C14	−176.06 (17)
C19—C20—C21—C22	1.6 (4)	C17—C18—N3—C14	2.3 (3)
C20—C21—C22—C23	0.8 (4)	N4—C18—N3—Mo1	8.4 (2)
C21—C22—C23—N5	−1.4 (5)	C17—C18—N3—Mo1	−173.20 (14)
C5—C4—N1—C8	−0.5 (3)	C2—Mo1—N3—C14	123.55 (15)
C5—C4—N1—Mo1	170.18 (17)	C3—Mo1—N3—C14	−147.12 (15)
C7—C8—N1—C4	0.4 (3)	N2—Mo1—N3—C14	−49.50 (14)
C9—C8—N1—C4	−179.05 (18)	N1—Mo1—N3—C14	22.46 (14)
C7—C8—N1—Mo1	−170.80 (16)	C2—Mo1—N3—C18	−61.04 (16)
C9—C8—N1—Mo1	9.7 (2)	C3—Mo1—N3—C18	28.29 (16)
C1—Mo1—N1—C4	−86.90 (18)	N2—Mo1—N3—C18	125.91 (15)
C2—Mo1—N1—C4	−2.29 (18)	N1—Mo1—N3—C18	−162.13 (15)
C3—Mo1—N1—C4	−147.7 (4)	N3—C18—N4—C19	−161.7 (2)
N2—Mo1—N1—C4	−179.13 (17)	C17—C18—N4—C19	19.9 (3)
N3—Mo1—N1—C4	95.40 (17)	N5—C19—N4—C18	9.2 (3)
C1—Mo1—N1—C8	83.75 (16)	C20—C19—N4—C18	−172.9 (2)
C2—Mo1—N1—C8	168.36 (14)	C20—C19—N5—C23	3.4 (4)
C3—Mo1—N1—C8	22.9 (4)	N4—C19—N5—C23	−178.9 (2)
N2—Mo1—N1—C8	−8.48 (13)	C22—C23—N5—C19	−0.7 (4)

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N4—H1···C3	0.75 (2)	2.40 (2)	2.978 (3)	135 (2)
C17—H17···N5	0.93	2.36	2.902 (3)	117
C10—H10···O1^{i}	0.93	2.43	3.318 (3)	160
C16—H16···O1^{ii}	0.93	2.38	3.305 (3)	172

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

Fig. 1


O1
C1
O2
C2
C6
C5
C4
C8
N1
Mo1
C7
C10
C9
N2
C3
O3
C13
C11
C12
N3
N4
C20
C14
C18
C19
C21
C22
C17
C15
N5
C23
C16