

Table 1. Fractional atomic coordinates and equivalent isotropic displacement parameters (\AA^2)
$$U_{eq} = (1/3)\sum_i \sum_j U_{ij} a_i^* a_j^* a_i \cdot a_j$$

	x	y	z	U_{eq}
Mn	0.0720 (1)	0.2069 (1)	0.0516 (1)	0.041 (1)
O(1)	0.0103 (2)	0.0965 (2)	-0.1208 (2)	0.068 (1)
Cl(1)	0.1356 (1)	0.3568 (1)	0.2197 (1)	0.053 (1)
C(1')	0.3163 (3)	0.1078 (3)	0.0979 (3)	0.046 (1)
C(2')	0.2567 (3)	0.0518 (3)	0.1531 (3)	0.045 (1)
C(3')	0.3016 (3)	-0.0469 (3)	0.2193 (3)	0.051 (1)
C(4')	0.4025 (3)	-0.0869 (3)	0.2330 (3)	0.056 (1)
C(5')	0.4617 (3)	-0.0307 (3)	0.1814 (3)	0.055 (1)
C(6')	0.4197 (3)	0.0649 (3)	0.1140 (3)	0.054 (1)
N(1'a)	0.1888 (2)	0.2618 (2)	0.0079 (2)	0.042 (1)
C(1'a)	0.2797 (3)	0.2108 (3)	0.0300 (3)	0.050 (1)
C(2'a)	0.1621 (3)	0.3713 (3)	-0.0558 (3)	0.052 (1)
O(2')	0.1606 (2)	0.0879 (2)	0.1459 (2)	0.049 (1)
Cl(2)	0.5917 (1)	-0.0805 (1)	0.2039 (1)	0.072 (1)
C(1'')	-0.1812 (3)	0.3008 (3)	-0.0297 (3)	0.042 (1)
C(2'')	-0.1464 (3)	0.2044 (3)	0.0447 (3)	0.041 (1)
C(3'')	-0.2181 (3)	0.1564 (3)	0.0879 (3)	0.046 (1)
C(4'')	-0.3200 (3)	0.2030 (3)	0.0594 (3)	0.052 (1)
C(5'')	-0.3538 (3)	0.2979 (3)	-0.0140 (3)	0.051 (1)
C(6'')	-0.2868 (3)	0.3459 (3)	-0.0591 (3)	0.049 (1)
N(1''a)	-0.0167 (2)	0.3265 (2)	-0.0624 (2)	0.042 (1)
C(1''a)	-0.1158 (3)	0.3543 (3)	-0.0834 (3)	0.046 (1)
C(2''a)	0.0382 (3)	0.3767 (3)	-0.1315 (3)	0.052 (1)
O(2'')	-0.0493 (2)	0.1546 (2)	0.0748 (2)	0.045 (1)
Cl(3)	-0.4846 (1)	0.3561 (1)	-0.0516 (1)	0.072 (1)

Table 2. Selected geometric parameters (\AA , $^\circ$)

Mn—O(1)	2.319 (3)	Mn—O(2')	1.874 (3)
Mn—N(1'a)	1.973 (3)	N(1'a)—C(1'a)	1.280 (5)
Mn—N(1''a)	1.986 (2)	N(1''a)—C(1''a)	1.285 (5)
Mn—Cl(1)	2.572 (1)	N(1'a)—C(2'a)	1.468 (5)
Mn—O(2')	1.883 (2)	N(1'a)—C(2''a)	1.472 (4)
O(1)—Mn—Cl(1)	170.1 (1)	O(1)—Mn—O(2')	91.7 (1)
Cl(1)—Mn—N(1'a)	88.1 (1)	N(1'a)—Mn—O(2')	92.6 (1)
Cl(1)—Mn—O(2')	96.4 (1)	Cl(1)—Mn—N(1''a)	88.5 (1)
O(1)—Mn—N(1''a)	83.1 (1)	O(2')—Mn—N(1''a)	173.4 (1)
N(1'a)—Mn—N(1''a)	83.1 (1)	Cl(1)—Mn—O(2'')	96.5 (1)
O(1)—Mn—O(2'')	87.9 (1)	O(2')—Mn—O(2'')	91.3 (1)
N(1'a)—Mn—O(2'')	173.6 (1)	O(1)—Mn—N(1'a)	87.0 (1)
N(1''a)—Mn—O(2'')	92.5 (1)		

The position of the Mn atom was identified in a Patterson vector map. Direct methods of phase determination lead to an electron-density map from which all the non-H atoms were identified.

Siemens standard procedures and programs were used for all data collection operations. *SHELXTL-Plus* (Sheldrick, 1990) was used for phase determination and structure refinement.

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Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the IUCr (Reference: FG1002). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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cis,trans-(2,2'-Bipyridine-*N,N'*)dibromobis(4-methylpyridine-*N*)tungsten(II)

IVAN LEBAN, JURIJ V. BRENČIČ AND BORIS ČEH

Department of Chemistry and Chemical Technology,
University of Ljubljana, Aškerčeva 5, PO Box 537,
61001 Ljubljana, Slovenia

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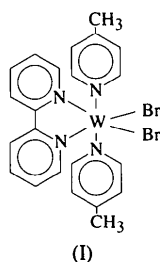
Abstract

The title compound, $[\text{WBr}_2(\text{C}_6\text{H}_7\text{N})_2(\text{C}_{10}\text{H}_8\text{N}_2)]$, is a neutral tungsten(II) mononuclear octahedral coordination complex. The two 4-methylpyridine molecules are in the *trans* staggered conformation with an interplanar angle of $86.5(3)^\circ$. The bidentate bipyridine ligand is slightly twisted, the angle between separate planar pyridine moieties being $5.7(5)^\circ$.

Comment

Although there are numerous organometallic compounds of divalent tungsten, only a few mononuclear coordination compounds of W^{II} , most of which are six- (Colquhoun & Williams, 1984) or seven-coordinate (Barrera, Sabat & Harman, 1991), have been structurally characterized by X-ray diffraction. WCl_6 and WBr_5 are well known starting substances for the syntheses of tungsten complexes with lower oxidation states, *i.e.* from four (Brenčič, Čeh & Šegedin, 1980) down to zero (Anderson & Richards, 1986). Using the same synthetic procedure known for obtaining mononuclear anionic complexes of W^{III} with the formula *trans*-(BaseH)[WX_4L_2] [Base = pyridine, 4-methylpyridine, NH_3 ; X = Br, Cl; L = pyridine, 4-methylpyridine (Brenčič, Čeh & Leban, 1979, 1990; Brenčič, Čeh &

Šegedin, 1980)], our aim was to synthesize the similar *cis* species starting from WBr₄(bipy). However, the higher sensitivity of *cis* in comparison to *trans* W^{IV} neutral complexes with regard to reduction with 4-picoline resulted in the title compound, (I).



[WBr₂(bipy)pic₂] (bipy = 2,2'-bipyridine, pic = 4-methylpyridine) is an example of a neutral octahedral complex of W^{II} with an equatorial arrangement of the two Br atoms and bidentate bipyridine ligand, and with two coordinated molecules of 4-methylpyridine in a *trans* staggered conformation. The W—Br and W—N(pic) distances are close to those found, for example, in the anion WBr₄pic₂⁻

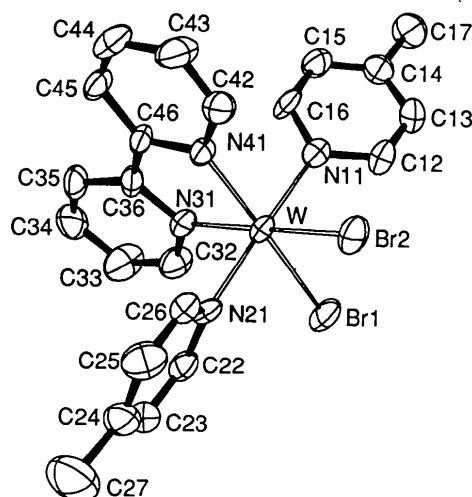


Fig. 1. ORTEP (Johnson, 1971) view of the molecule with atomic numbering. Anisotropic displacement ellipsoids are at the 30% probability level. H atoms are omitted for clarity.

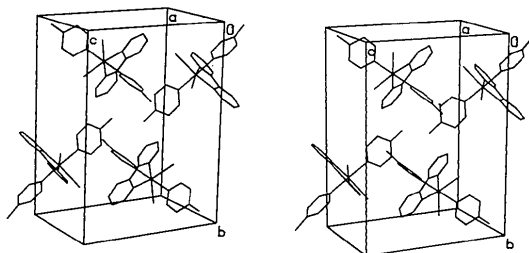


Fig. 2. PLUTON stereodrawing (Spek, 1991) of the crystal packing.

(Brenčič, Čeh & Leban, 1979, 1990), in the compound [WBr(Ph₂PCH₂CH₂PPh₂){N₂CN(Me)CH₂CH₂NMe}]PF₆ (Colquhoun & Williams, 1984) and in the compound [W{C(Ph)CHCHMe}Br₂(CO)₂(pic)] (Mayr, Asaro & Gilnes, 1987). The two W—N(bipy) distances are almost the same, but are shorter than those found, for example, in [W(bipy)(PMe₃)₂Cl(CH₃CN)]PF₆ (Barrera, Sabat & Harman, 1991) or in [AsPh₄][WO(bipy)(CN)₃].0.5bipy.2H₂O (Szklarzewicz, Samotus, Alcock & Moll, 1990). The bipyridine ligand is slightly distorted. The two pyridine moieties are twisted by 5.7 (5)° about the C36—C46 bond. This value is close to the average value of 8° found in the survey by Durham, Wilson, Hodgson & Meyer (1980) and earlier by McKenzie (1971). All other distances and angles are generally as expected. There are no unusual close intermolecular contacts.

Experimental

A mixture of 0.80 g of WBr₄(bipy) and 40 ml of 4-methylpyridine was degassed *in vacuo* and stirred for 20 h at room temperature. This was followed by filtration under an argon atmosphere. The mixture of 10 ml of degassed water and 10 ml of filtrate was left on ice for a week. From the crystalline product, a well shaped dark red crystal of the compound was selected and sealed into a glass capillary under argon atmosphere. Crystals proved to be sensitive to air.

Crystal data

[WBr₂(C₆H₇N)₂(C₁₀H₈N₂)]

M_r = 686.11

Monoclinic

*P*2₁/*n*

a = 9.5690 (1) Å

b = 17.651 (2) Å

c = 13.485 (2) Å

β = 94.26 (2)°

V = 2271.4 (5) Å³

Z = 4

D_x = 2.006 Mg m⁻³

Mo *K*α radiation

λ = 0.7107 Å

Cell parameters from 25 reflections

θ = 9–12°

μ = 8.695 mm⁻¹

T = 293 (1) K

Plate

0.42 × 0.32 × 0.06 mm

Dark red

Data collection

Enraf–Nonius CAD-4

diffractometer

Variable rate θ/2θ scans

Absorption correction:

by integration from crystal shape

T_{min} = 0.100, *T_{max}* =

0.596

9921 measured reflections

4973 independent reflections

3071 observed reflections

[*I* > 2σ(*I*)]

R_{int} = 0.0823

θ_{max} = 27.11°

h = -12 → 12

k = 0 → 22

l = 0 → 17

3 standard reflections

monitored every 600

reflections

intensity decay: 5.44%

Refinement

Refinement on *F*²

R[*F*² > 2σ(*F*²)] = 0.0447

w*R*(*F*²) = 0.0893

S = 1.070

(Δ/σ)_{max} = 0.257

Δρ_{max} = 2.189 e Å⁻³

Δρ_{min} = -1.584 e Å⁻³

Extinction correction: none

4973 reflections
266 parameters
Only H-atom U 's refined
 $w = 1/[\sigma^2(F_o^2) + (0.0528P)^2]$
where $P = (F_o^2 + 2F_c^2)/3$

Atomic scattering factors
from *International Tables*
for *Crystallography* (1992),
Vol. C, Tables 4.2.6.8 and
6.1.1.4)

C16—N11—C12	115.5 (7)	C46—C36—C35	125.8 (7)
C16—N11—W	121.6 (6)	N31—C36—C35	120.2 (8)
C12—N11—W	122.7 (5)	C42—N41—C46	118.0 (7)
C13—C12—N11	122.5 (8)	C42—N41—W	125.0 (5)
C12—C13—C14	121.2 (8)	C46—N41—W	116.8 (5)
C15—C14—C13	115.9 (9)	C43—C42—N41	123.4 (8)
C15—C14—C17	122.9 (8)	C42—C43—C44	119.5 (9)
C13—C14—C17	121.1 (8)	C45—C44—C43	118.5 (8)
C16—C15—C14	121.2 (8)	C44—C45—C46	121.4 (8)
N11—C16—C15	123.7 (8)	N41—C46—C36	115.4 (6)
C26—N21—C22	114.7 (7)	N41—C46—C45	119.1 (8)
C26—N21—W	121.2 (5)	C36—C46—C45	125.4 (7)
C22—N21—W	123.9 (6)		

Table 1. *Fractional atomic coordinates and equivalent isotropic displacement parameters* (\AA^2)

$$U_{eq} = (1/3)\sum_i \sum_j U_{ij} a_i^* a_j^* \mathbf{a}_i \cdot \mathbf{a}_j$$

	x	y	z	U_{eq}
W	0.24514 (3)	0.27437 (2)	0.09138 (2)	0.03370 (11)
Br1	0.43385 (9)	0.18450 (7)	0.17461 (6)	0.0541 (3)
Br2	0.43587 (8)	0.36401 (7)	0.03220 (6)	0.0553 (3)
N11	0.2485 (7)	0.2063 (4)	-0.0426 (5)	0.039 (2)
C12	0.3702 (8)	0.1826 (6)	-0.0799 (6)	0.048 (2)
C13	0.3719 (9)	0.1340 (6)	-0.1584 (6)	0.050 (2)
C14	0.2488 (9)	0.1049 (6)	-0.2040 (5)	0.050 (2)
C15	0.1274 (9)	0.1301 (6)	-0.1676 (6)	0.050 (2)
C16	0.1298 (8)	0.1795 (6)	-0.0898 (6)	0.048 (2)
C17	0.2514 (10)	0.0452 (6)	-0.2854 (6)	0.058 (3)
N21	0.2484 (6)	0.3420 (4)	0.2267 (4)	0.036 (2)
C22	0.2561 (8)	0.3122 (6)	0.3193 (5)	0.041 (2)
C23	0.2487 (9)	0.3534 (6)	0.4035 (6)	0.045 (2)
C24	0.2365 (9)	0.4312 (7)	0.3998 (6)	0.052 (2)
C25	0.2259 (10)	0.4635 (7)	0.3044 (6)	0.060 (3)
C26	0.2311 (8)	0.4161 (6)	0.2230 (6)	0.042 (2)
C27	0.2251 (15)	0.4788 (9)	0.4898 (8)	0.099 (5)
N31	0.0728 (6)	0.2193 (4)	0.1435 (4)	0.0345 (15)
C32	0.0807 (10)	0.1634 (6)	0.2112 (6)	0.052 (2)
C33	-0.0353 (10)	0.1347 (7)	0.2531 (7)	0.067 (3)
C34	-0.1681 (10)	0.1668 (8)	0.2268 (7)	0.065 (3)
C35	-0.1790 (9)	0.2230 (6)	0.1610 (6)	0.045 (2)
C36	-0.0597 (8)	0.2510 (5)	0.1179 (5)	0.036 (2)
N41	0.0748 (6)	0.3333 (4)	0.0250 (4)	0.034 (2)
C42	0.0828 (9)	0.3889 (6)	-0.0443 (5)	0.044 (2)
C43	-0.0307 (9)	0.4245 (6)	-0.0881 (6)	0.055 (3)
C44	-0.1666 (10)	0.4029 (6)	-0.0631 (6)	0.051 (2)
C45	-0.1779 (8)	0.3473 (6)	0.0042 (6)	0.047 (2)
C46	-0.0579 (7)	0.3106 (6)	0.0498 (5)	0.036 (2)

Table 2. *Selected geometric parameters* (\AA , $^\circ$)

W—N41	2.079 (6)	C24—C25	1.403 (12)
W—N31	2.082 (6)	C24—C27	1.486 (14)
W—N11	2.172 (7)	C25—C26	1.384 (13)
W—N21	2.179 (7)	N31—C32	1.342 (11)
W—Br2	2.5860 (10)	N31—C36	1.405 (10)
W—Br1	2.5953 (10)	C32—C33	1.379 (12)
N11—C16	1.346 (10)	C33—C34	1.412 (14)
N11—C12	1.368 (10)	C34—C35	1.331 (15)
C12—C13	1.364 (12)	C35—C36	1.407 (11)
C13—C14	1.386 (12)	C36—C46	1.398 (12)
C14—C15	1.368 (11)	N41—C42	1.361 (11)
C14—C17	1.524 (13)	N41—C46	1.395 (9)
C15—C16	1.362 (12)	C42—C43	1.352 (11)
N21—C26	1.318 (12)	C43—C44	1.419 (13)
N21—C22	1.352 (9)	C44—C45	1.346 (14)
C22—C23	1.354 (11)	C45—C46	1.419 (11)
C23—C24	1.379 (15)		
N41—W—N31	76.3 (3)	N21—C22—C23	124.3 (9)
N41—W—N11	88.8 (2)	C22—C23—C24	120.9 (8)
N31—W—N11	94.8 (2)	C23—C24—C25	115.9 (8)
N41—W—N21	92.8 (2)	C23—C24—C27	123.0 (9)
N31—W—N21	86.3 (2)	C25—C24—C27	121.0 (11)
N11—W—N21	178.3 (2)	C26—C25—C24	118.5 (10)
N41—W—Br2	96.3 (2)	N21—C26—C25	125.6 (8)
N31—W—Br2	170.1 (2)	C32—N31—C36	117.6 (7)
N11—W—Br2	91.6 (2)	C32—N31—W	124.5 (5)
N21—W—Br2	87.5 (2)	C36—N31—W	117.0 (6)
N41—W—Br1	171.9 (2)	N31—C32—C33	122.8 (9)
N31—W—Br1	96.4 (2)	C32—C33—C34	119.1 (10)
N11—W—Br1	88.2 (2)	C35—C34—C33	119.4 (9)
N21—W—Br1	90.4 (2)	C34—C35—C36	120.8 (9)
Br2—W—Br1	91.32 (3)	C46—C36—N31	113.9 (7)

The space group $P2_1/n$ was determined from the intensity statistics. H atoms were included in the refinement at calculated positions. Two common isotropic displacement parameters for H atoms were refined to $0.088 (16) \text{\AA}^2$ (for CH_3 groups) and to $0.055 (7) \text{\AA}^2$ for others.

Calculations were performed on the VAX 8550 cluster at the University Computer Centre, Ljubljana, under VMS 5.5-2 and partially on PC 486.

Data collection: *CAD-4 Software* (Enraf-Nonius, 1989). Cell refinement: *CAD-4 Software*. Data reduction: *NRCVAX* (Gabe, Le Page, Charland, Lee & White, 1989) and *GX* (Mallinson & Muir, 1985). Program(s) used to solve structure: *SHELXS86* (Sheldrick, 1985). Program(s) used to refine structure: *SHELXL93* (Sheldrick, 1993). Molecular graphics: *ORTEPII* (Johnson, 1971) and *PLUTON* (Spek, 1991). Software used to prepare material for publication: *SHELXL93*.

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Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the IUCr (Reference: HU1136). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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Tris(2,2'-bipyridine)manganese(II) Perchlorate Hemihydrate

XIAO-MING CHEN,* RUI-QIN WANG AND ZHI-TAO XU

Department of Chemistry, Zhongshan University,
135 Xingang Road W., Guangzhou,
People's Republic of China

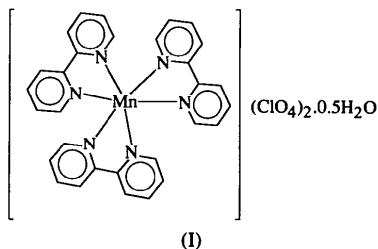
(Received 28 July 1994; accepted 28 October 1994)

Abstract

The crystal structure of tris(2,2'-bipyridine)manganese(II) perchlorate hemihydrate, [Mn(C₁₀H₈N₂)₃](ClO₄)₂·0.5H₂O, has been established by X-ray crystallography. The metal atom is coordinated by three chelating 2,2'-bipyridine ligands in a highly distorted octahedral MnN₆ arrangement with Mn—N bond lengths ranging from 2.219 (4) to 2.294 (4) Å.

Comment

In an attempt to prepare a manganese(II) complex containing 2,2'-bipyridine (bpy) and betaine ligands, we obtained an unusual singly carboxylato-bridged dinuclear manganese(II) complex [Mn₂(bpy)₄(Me₃NCH₂CO₂)(H₂O)₂](ClO₄)₄·2H₂O, together with golden needle-like crystals as a minor product (Chen & Mak, 1993). The latter complex has been characterized by IR spectral data and elemental analysis as the monomeric complex [Mn(bpy)₃](ClO₄)₂·0.5H₂O, (I). We report here the single-crystal structure of this complex.



The crystal structure of the title complex is composed of monomeric [Mn(bpy)₃]²⁺ cations, perchlorate anions and lattice water molecules. As illustrated in Fig. 1, the metal atom is surrounded by six N atoms from three chelating bpy ligands in a highly distorted octahedral MnN₆ arrangement. The Mn—N bond lengths range from 2.219 (4) to 2.294 (4) Å with the most distorted N—Mn—N angle (*ca* 73°) resulting from a chelating bpy ligand. The Mn—N bond lengths are significantly longer than those (1.932–2.172 Å) found for the analogous cations [Zn(bpy)₃]²⁺ (Chen, Wang & Yu, 1995), [Co(bpy)₃]³⁺ (Yanagi, Ohashi, Sasada, Kaizu & Kobayashi, 1981), [Fe(bpy)₃]³⁺ (Figgis, Skelton & White, 1978) and [Ni(bpy)₃]²⁺ (Wada, Katayama & Tanaka, 1976). The Mn—N bond lengths are longer than the *M*—N bond lengths found for analogous [M(bpy)₃]^{*n*+} complexes (*M* = Zn²⁺, Co³⁺, Fe³⁺, Ni²⁺); this may be attributed to the larger ionic radius of the Mn²⁺ cation. It is noteworthy that each pair of pyridine rings in the three bpy ligands are not coplanar, the dihedral angles between each pair ranging from 13 to 25°.

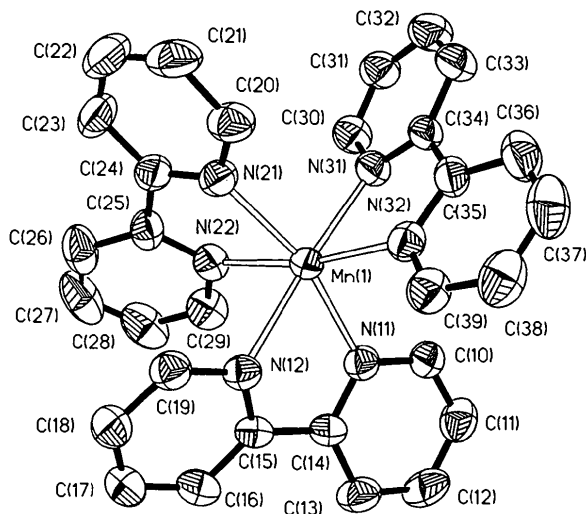


Fig. 1. ORTEP (Johnson, 1965) drawing of the [Mn(bpy)₃]²⁺ cation showing 45% probability ellipsoids.

Experimental

The crystals were a by-product of the work reported in XVI Congress and General Assembly of International Union of Crystallography (Chen & Mak, 1993).

Crystal data

[Mn(C₁₀H₈N₂)₃](ClO₄)₂·
0.5H₂O
M_r = 730.4
Triclinic
*P*1

Mo *K*α radiation
λ = 0.71073 Å
Cell parameters from 25
reflections
θ = 1.5–25°