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CRYSTAL AND MOLECULAR STRUCTURE OF 3,3,4,4-TETRAMETHYL-3,4-DIHYDRODIAZETE BIS(PENTACARBONYLTUNGSTEN), $(N_2C_2Me_4)[W(CO)_5]_2$

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Summary

Crystals of $C_{16}H_{12}N_2O_{10}W_2$ are orthorhombic, a 18.331(5), b 17.877(3), c 13.112(2) Å; $Z = 8$; space group *Pbca*. The structure analysis was carried out by employing 2686 independent observed reflections collected on a X-ray diffractometer and the least-squares refinement yielded final index $R = 0.042$. The crystal structure consists of discrete unsymmetrical molecules, in which each W atom is octahedrally surrounded by five terminal carbonyl groups and one nitrogen atom of the 3,3,4,4-tetramethyl-1,2-diazetine ligand. There is no intramolecular W $\cdots$ W interaction.

Introduction

There exists an extensive series of transition-metal complexes of diazenes [1–31]. In a preceding paper [32] the crystal and molecular structure of the complex $[W(N_2C_2Me_4)(CO)_4]_2$ (I), were discussed. In this paper the structure of the complex $W_2(N_2C_2Me_4)(CO)_{10}$ is reported, and the structural features of this compound are compared with those of related compounds. Some light is thrown on the bond character in these complexes, and on their different stabilities.

Results and discussion

The crystal structure of the $W_2(N_2C_2Me_4)(CO)_{10}$ complex consists of discrete molecules. All the atoms of the molecule occupy positions of general crystallographic symmetry and the molecule exhibits no real symmetry, though it has virtual C_{2v} symmetry and each $W(CO)_5$ fragment has virtual C_{4v} local symmetry. A representation of the molecule with the numbering scheme, drawn with the program ORTEP [33], is shown in Fig. 1. A listing of the interatomic distances and bond angles uncorrected for the effects of thermal motion is given in Table 1.

Each W atom exhibits a deformed octahedral coordination, bonded to five carbon atoms of carbonyl groups and to one nitrogen atom of the diazetine ligand. All the

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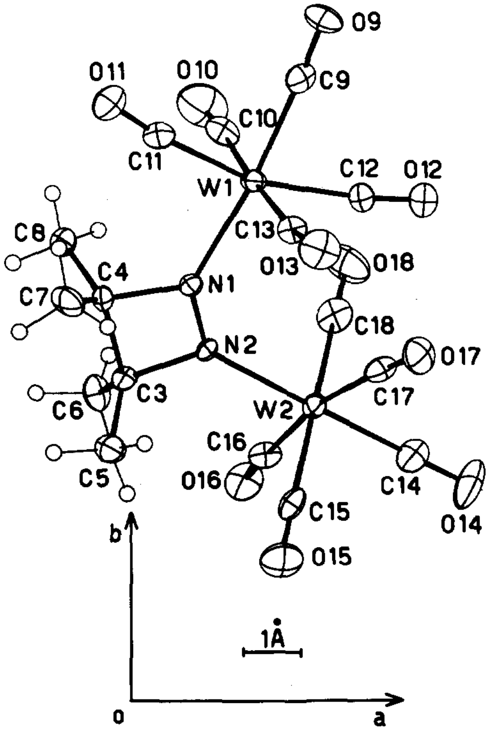

Fig. 1. ORTEP plot and numbering scheme of atoms, down *c* axis of cell. Thermal ellipsoids include 30% probability of electron density.

carbonyl groups are terminal. The IR spectrum of the complex [31] shows five ν(CO) bands instead of the three bands expected for a local C_{4v} symmetry and this suggests that the molecule in solution retains the unsymmetrical solid state structure, with restricted rotation around the W–N bond. There is no W ··· W interaction, since the corresponding distance is 4.473 Å, significantly longer than the value 4.230 Å previously found in $[W(N_2C_2Me_4)(CO)_4]_2$ (I) [32]. The W–N bond lengths (average 2.207 Å) are longer than those (2.160 Å) in (I), and the W–N–N angles (average 134.1°) larger than those in the latter complex (133.0°), in agreement with the steric requirement for sufficient separation of the two $W(CO)_5$ fragments.

The W–C bond lengths (average 2.036 Å) are comparable with those found in (I) (2.020 Å). The W–C bonds in *trans* positions to the nitrogen atoms are shorter than those in *cis* positions (2.009 vs. 2.043 Å), a consequence of a *trans*-effect of the diazetine ligand, which is a π-acceptor poorer than CO groups. A similar remark was made concerning I [32]. The W–C–O angles are in the range 179.6–172.4° and those of the groups in *cis* to the nitrogen atoms are more bended than those of the groups in *trans*. The C–O bond lengths are in the range 1.116–1.166 Å (average 1.139 Å) and can be compared with the value 1.143 Å previously found [32]. The bond lengths and angles in the ligand are also comparable with those previously found [30,32]. The N–N bond (1.292 Å) is longer than a double bond, a consequence of the σ-donation of the two lone-pairs from nitrogen to metal.

TABLE 1

INTERATOMIC DISTANCES (Å) AND BOND ANGLES (°) WITH e.s.d.'s IN PARENTHESES

W(1)···W(2)	4.4734(8)		
In the coordination octahedra			
W(1)–N(1)	2.211(8)	W(2)–N(2)	2.203(8)
W(1)–C(9)	2.010(11)	W(2)–C(14)	2.008(13)
W(1)–C(10)	1.991(16)	W(2)–C(15)	2.063(12)
W(1)–C(11)	2.039(13)	W(2)–C(16)	2.046(11)
W(1)–C(12)	2.043(13)	W(2)–C(17)	2.070(12)
W(1)–C(13)	2.068(13)	W(2)–C(18)	2.019(13)
N(1)–W(1)–C(9)	174.2(4)	N(2)–W(2)–C(14)	179.0(4)
N(1)–W(1)–C(10)	88.9(5)	N(2)–W(2)–C(15)	92.3(4)
N(1)–W(1)–C(11)	92.8(4)	N(2)–W(2)–C(16)	95.2(4)
N(1)–W(1)–C(12)	102.9(4)	N(2)–W(2)–C(17)	94.7(4)
N(1)–W(1)–C(13)	93.1(4)	N(2)–W(2)–C(18)	89.7(4)
C(9)–W(1)–C(10)	88.6(5)	C(14)–W(2)–C(15)	88.7(5)
C(9)–W(1)–C(11)	82.1(5)	C(14)–W(2)–C(16)	84.5(5)
C(9)–W(1)–C(12)	82.2(5)	C(14)–W(2)–C(17)	85.7(5)
C(9)–W(1)–C(13)	89.8(5)	C(14)–W(2)–C(18)	89.3(5)
C(10)–W(1)–C(11)	91.7(6)	C(15)–W(2)–C(16)	90.6(4)
C(10)–W(1)–C(12)	88.5(6)	C(15)–W(2)–C(17)	86.6(5)
C(10)–W(1)–C(13)	176.8(5)	C(15)–W(2)–C(18)	175.2(5)
C(11)–W(1)–C(12)	164.2(5)	C(16)–W(2)–C(17)	169.9(5)
C(11)–W(1)–C(13)	90.8(5)	C(16)–W(2)–C(18)	84.9(5)
C(12)–W(1)–C(13)	88.5(5)	C(17)–W(2)–C(18)	97.6(5)
In the carbonyls			
C(9)–O(9)	1.133(14)	C(14)–O(14)	1.142(16)
C(10)–O(10)	1.166(20)	C(15)–O(15)	1.130(15)
C(11)–O(11)	1.133(16)	C(16)–O(16)	1.116(14)
C(12)–O(12)	1.135(15)	C(17)–O(17)	1.139(15)
C(13)–O(13)	1.142(16)	C(18)–O(18)	1.155(17)
W(1)–C(9)–O(9)	177.1(1.0)	W(2)–C(14)–O(14)	179.3(1.1)
W(1)–C(10)–O(10)	176.8(1.2)	W(2)–C(15)–O(15)	176.3(1.1)
W(1)–C(11)–O(11)	172.6(1.0)	W(2)–C(16)–O(16)	174.0(1.0)
W(1)–C(12)–O(12)	173.7(1.1)	W(2)–C(17)–O(17)	174.2(1.0)
W(1)–C(13)–O(13)	172.4(1.1)	W(2)–C(18)–O(18)	179.6(1.1)
In the 3,3,4,4-tetramethyl-3,4-dihydrodiazete ligand			
N(1)–N(2)	1.292(12)	C(5)–H(5c)	0.98(9)
N(1)–C(4)	1.523(14)	C(6)–H(6a)	1.03(10)
N(2)–C(3)	1.512(13)	C(6)–H(6b)	0.90(10)
C(3)–C(4)	1.580(15)	C(6)–H(6c)	1.03(10)
C(3)–C(5)	1.525(16)	C(7)–H(7a)	0.99(11)
C(3)–C(6)	1.504(16)	C(7)–H(7b)	1.04(10)
C(4)–C(7)	1.515(19)	C(7)–H(7c)	1.00(11)
C(4)–C(8)	1.514(17)	C(8)–H(8a)	1.04(10)
C(5)–H(5a)	1.04(9)	C(8)–H(8b)	1.01(10)
C(5)–H(5b)	1.00(9)	C(8)–H(8c)	0.99(11)
W(1)–N(1)–N(2)	137.5(7)	H(5a)–C(5)–H(5b)	110(8)
W(2)–N(2)–N(1)	134.7(6)	H(5a)–C(5)–H(5c)	108(8)
W(1)–N(1)–C(4)	127.7(6)	H(5b)–C(5)–H(5c)	113(8)
W(2)–N(2)–C(3)	128.9(6)	C(3)–C(6)–H(6a)	104(6)
N(1)–N(2)–C(3)	96.4(7)	C(3)–C(6)–H(6b)	114(6)

TABLE 1 (continued)

N(2)–N(1)–C(4)	94.5(7)	C(3)–C(6)–H(6c)	113(6)
C(3)–C(4)–N(1)	84.9(7)	H(6a)–C(6)–H(6b)	106(8)
C(4)–C(3)–N(2)	84.2(7)	H(6a)–C(6)–H(6c)	106(8)
N(1)–C(4)–C(7)	113.4(1.0)	H(6b)–C(6)–H(6c)	112(8)
N(1)–C(4)–C(8)	110.2(9)	C(4)–C(7)–H(7a)	113(7)
C(7)–C(4)–C(8)	113.9(1.0)	C(4)–C(7)–H(7b)	109(6)
C(3)–C(4)–C(7)	116.5(9)	C(4)–C(7)–H(7c)	110(7)
C(3)–C(4)–C(8)	114.6(1.0)	H(7a)–C(7)–H(7b)	107(9)
N(2)–C(3)–C(5)	112.0(9)	H(7a)–C(7)–H(7c)	111(9)
N(2)–C(3)–C(6)	114.0(9)	H(7b)–C(7)–H(7c)	107(9)
C(4)–C(3)–C(5)	115.5(9)	C(4)–C(8)–H(8a)	110(6)
C(4)–C(3)–C(6)	117.8(9)	C(4)–C(8)–H(8b)	112(6)
C(5)–C(3)–C(6)	110.9(9)	C(4)–C(8)–H(8c)	111(7)
C(3)–C(5)–H(5a)	108(5)	H(8a)–C(8)–H(8b)	106(8)
C(3)–C(5)–H(5b)	111(6)	H(8a)–C(8)–H(8c)	107(9)
C(3)–C(5)–H(5c)	107(6)	H(8b)–C(8)–H(8c)	109(9)

The equation defining the best plane of the ligand ring is:

$$0.0762\,X - 0.4215\,Y + 0.9036\,Z - 0.0172 = 0$$

with displacements of atoms: N(1) 0.005(9), N(2) −0.005(9), C(3) 0.006(11) and C(4) −0.009(13) Å; the probability that the ring is non-planar is $P = 76\%$, since the quantity $\Sigma(d/\sigma_{\perp})^2$ distributed like χ^2 $(n = 1)$ is 1.40. The plane leaves W(1) and W(2) out of this on the same side with displacements: W(1) −0.1326(5), W(2)

TABLE 2

SHORTEST INTERMOLECULAR APPROACHES (Å)

N(1)···O(16) (i)	3.497	O(12)···C(8) (vii)	3.392
N(2)···O(16) (i)	3.442	O(12)···O(15) (v)	3.252
C(5)···O(16) (i)	3.485	C(13)···O(11) (iii)	3.274
C(6)···O(10) (ii)	3.335	C(13)···O(16) (i)	3.139
C(7)···O(11) (iii)	3.429	O(13)···O(12) (iv)	3.092
C(9)···O(18) (iv)	3.392	O(13)···C(16) (i)	3.263
O(9)···O(18) (iv)	3.198	O(13)···O(16) (i)	3.026
O(9)···O(15) (v)	3.454	O(13)···O(18) (iv)	3.330
O(9)···C(17) (v)	3.450	C(15)···O(16) (i)	3.454
O(9)···O(17) (v)	3.248	O(15)···O(18) (i)	3.364
O(10)···O(14) (v)	3.175	C(17)···O(16) (i)	3.364
O(11)···O(13) (iii)	3.127	O(17)···O(14) (i)	3.243
O(11)···O(15) (vi)	3.490	O(17)···O(16) (i)	3.433
O(11)···O(16) (vi)	3.338		

Symmetry code

(i)	x	$1/2 - y$	$1/2 - z$
(ii)	$-x$	$1 - y$	$-z$
(iii)	$-x$	$1 - y$	$1 - z$
(iv)	$1/2 - x$	$1 - y$	$1/2 + z$
(v)	$1/2 - x$	$1/2 + y$	z
(vi)	$-x$	$1/2 + y$	$1/2 - z$
(vii)	$1/2 + x$	y	$1/2 - z$

$-0.0750(5)$ Å. The C–H distances in the methyl groups are normal (average 1.00 Å). The ^{1}H NMR spectrum, showing a single signal at δ 0.6 ppm (shifted by 0.35 ppm to high field, compared to free ligand) [31], confirms the symmetrical coordination of the diazetine to the $W(CO)_5$ fragments. The packing of the molecules in the crystal shows normal Van der Waals distances. Table 2 lists the shortest intermolecular distances. The shortest distance between an hydrogen atom and an oxygen atom in other molecule is 2.48 Å. In the crystal each molecule is in contact with twelve others, thus giving a molecular coordination number 12, which affords adequately close packing. The coefficient of molecular packing of the structure k [38], specifying the packing density, is 0.69, which represents a rather high value for a molecule of somewhat irregular shape.

Experimental

The $W_2(N_2C_2Me_4)(CO)_{10}$ complex was prepared by treating $W(CO)_5(THF)$ with 3,3,4,4-tetramethyl-1,2-diazetine in the ratio 2/1 in THF under argon and stirring the solution at room temperature for 4 h. After evaporation of the solvent, the products $W_2(N_2C_2Me_4)(CO)_{10}$ (30% yield) and $W(N_2C_2Me_4)(CO)_5$ (51% yield) were separated by chromatography on silica gel. Crystals of the title compound suitable for a single crystal X-ray diffractometric analysis were obtained by crystallization from toluene/petroleum ether [31].

Data collection

A needle-shaped red crystal of approximate dimensions 0.20 × 0.24 × 0.40 mm was used for data collection. Accurate cell parameters were obtained by a least-squares treatment of 2ϑ values for 25 reflections measured on a single-crystal Philips PW 1100 computer controlled diffractometer (using the graphite monochromated Mo-K_α radiation) of Centro di Studio per la Cristallografia strutturale del C.N.R., Pavia, Italy. The $\omega/2\vartheta$ scan technique with a constant scan speed of 0.060° sec^{-1} in ω was used to collect the intensities of 4934 independent reflections within the angular range $2 \leqslant \vartheta \leqslant 27.5°$. Three standard reflections were measured every 240 min and showed small random deviations about their mean intensities. The intensities were corrected for Lorentz and polarization factors and for absorption (according to North et al. [34]) and treated by Wilson's method [35] to give an approximate absolute scale and an overall isotropic temperature factor. 2686 reflections having $I \geqslant 3\sigma(I)$ were employed in the structure analysis.

The statistical distribution of the normalized structure factors $|E|$ is consistent with a centrosymmetric space group.

Crystal data

$C_{16}H_{12}N_2O_{10}W_2$, F.W. 759.98; orthorhombic, a 18.331(5), b 17.877(3), c 13.112(2) Å; V 4296.8 Å^3; $Z = 8$, D_c 2.35 g cm^{-3}; $F(000) = 2800$; λ(Mo-K_α) 0.7107 Å, μ(Mo-K_α) 113.9 cm^{-1}. Systematic absences $hk0$, $h \neq 2n$; $h0l$, $l \neq 2n$; $0kl$, $k \neq 2n$; $h00$, $h \neq 2n$; $0k0$, $k \neq 2n$; $00l$, $l \neq 2n$ defined the space group $Pbca$.

Structure determination and refinement

The coordinates of the two unique tungsten atoms were derived from a three-dimensional Patterson map. Their positional and isotropic thermal parameters were

TABLE 3

FINAL COORDINATES WITH e.s.d.'s IN PARENTHESES

Atom	*x*	*y*	*z*
W(1)	0.11560(3)	0.50466(3)	0.29761(4)
W(2)	0.17184(3)	0.28449(3)	0.15581(4)
N(1)	0.0561(5)	0.4026(4)	0.2514(7)
N(2)	0.0710(4)	0.3393(4)	0.2085(7)
C(3)	−0.0069(6)	0.3120(5)	0.2012(8)
C(4)	−0.0261(6)	0.3897(6)	0.2517(10)
C(5)	−0.0202(6)	0.2435(6)	0.2679(10)
C(6)	−0.0324(6)	0.2982(7)	0.0938(9)
C(7)	−0.0587(7)	0.3871(7)	0.3578(11)
C(8)	−0.0632(6)	0.4449(6)	0.1810(10)
C(9)	0.1600(6)	0.6028(6)	0.3395(9)
C(10)	0.0863(8)	0.5519(8)	0.1663(13)
C(11)	0.0270(7)	0.5480(7)	0.3706(9)
C(12)	0.2172(7)	0.4880(7)	0.2376(11)
C(13)	0.1521(7)	0.4570(7)	0.4320(10)
C(14)	0.2639(7)	0.2359(7)	0.1058(10)
C(15)	0.1507(6)	0.1895(7)	0.2401(9)
C(16)	0.1255(6)	0.2367(6)	0.0297(8)
C(17)	0.2340(7)	0.3201(6)	0.2784(9)
C(18)	0.1917(7)	0.3721(8)	0.0627(10)
O(9)	0.1821(5)	0.6592(4)	0.3639(8)
O(10)	0.0676(7)	0.5765(6)	0.0882(9)
O(11)	−0.0192(5)	0.5795(5)	0.4088(8)
O(12)	0.2749(5)	0.4854(5)	0.2065(7)
O(13)	0.1778(5)	0.4370(5)	0.5064(7)
O(14)	0.3166(5)	0.2083(6)	0.0784(8)
O(15)	0.1403(6)	0.1353(5)	0.2819(8)
O(16)	0.1058(5)	0.2110(5)	−0.0429(6)
O(17)	0.2700(5)	0.3337(5)	0.3467(7)
O(18)	0.2030(6)	0.4225(6)	0.0099(8)
H(5a)	0.012(5)	0.248(5)	0.332(7)
H(5b)	−0.073(5)	0.241(6)	0.289(8)
H(5c)	−0.004(6)	0.200(5)	0.229(8)
H(6a)	−0.089(5)	0.299(5)	0.099(8)
H(6b)	−0.021(5)	0.335(5)	0.050(7)
H(6c)	−0.019(5)	0.246(6)	0.066(8)
H(7a)	−0.104(6)	0.358(6)	0.361(9)
H(7b)	−0.022(6)	0.362(6)	0.407(8)
H(7c)	−0.067(7)	0.439(6)	0.384(9)
H(8a)	−0.030(5)	0.457(5)	0.119(8)
H(8b)	−0.110(5)	0.425(6)	0.152(8)
H(8c)	−0.073(6)	0.493(6)	0.216(9)

refined in three cycles of least-squares refinement to conventional *R* index of 0.134. The remaining 28 non-hydrogen atoms were located from a three-dimensional difference Fourier map, phased on the W atoms. Three cycles of least-squares refinement, including scale factor, individual isotropic thermal parameters for C, N, O atoms and anisotropic thermal parameters for W atoms, reduced the *R* index to 0.047. With other three refinement cycles with individual anisotropic thermal parameters, the R index was reduced to 0.044. The 12 hydrogen atoms were located from a three-dimensional difference Fourier synthesis following the anisotropic refinement, and included in the subsequent least-squares refinement with the same isotropic thermal parameters as the atom to which they were attached. Three cycles with unit weights and other three with weights $= \sigma^{-2}(|F_0|)$ gave $R = 0.042$ ($R_w = 0.042$). The atomic scattering factors for the non-hydrogen atoms were taken from

TABLE 4

THERMAL PARAMETERS WITH e.s.d.'s IN PARENTHESES[a]

Atom	β_{11}	β_{22}	β_{33}	β_{12}	β_{13}	β_{23}
W(1)	0.00199(1)	0.00152(1)	0.00401(3)	−0.00005(1)	−0.00020(2)	−0.00004(2)
W(2)	0.00169(1)	0.00195(1)	0.00450(3)	0.00024(1)	−0.00003(2)	−0.00053(2)
N(1)	0.0017(3)	0.0015(3)	0.0051(7)	0.0000(2)	−0.0007(4)	0.0006(4)
N(2)	0.0014(3)	0.0016(3)	0.0057(8)	0.0004(2)	−0.0009(4)	0.0012(4)
C(3)	0.0019(4)	0.0018(4)	0.0027(7)	0.0002(3)	−0.0004(5)	−0.0012(5)
C(4)	0.0013(4)	0.0021(4)	0.0074(10)	0.0000(3)	−0.0004(6)	−0.0008(6)
C(5)	0.0029(5)	0.0025(4)	0.0064(11)	−0.0003(4)	0.0015(6)	−0.0008(6)
C(6)	0.0023(4)	0.0043(6)	0.0038(9)	−0.0005(4)	−0.0004(5)	0.0003(6)
C(7)	0.0033(5)	0.0029(5)	0.0076(12)	−0.0010(4)	0.0023(7)	−0.0001(7)
C(8)	0.0020(4)	0.0026(4)	0.0096(13)	0.0002(4)	−0.0012(6)	0.0012(6)
C(9)	0.0028(5)	0.0026(4)	0.0039(8)	0.0005(4)	−0.0011(6)	−0.0003(6)
C(10)	0.0035(6)	0.0028(5)	0.0103(15)	−0.0008(4)	0.0005(8)	0.0011(8)
C(11)	0.0031(5)	0.0022(4)	0.0051(10)	0.0002(4)	0.0001(6)	0.0003(5)
C(12)	0.0020(4)	0.0027(5)	0.0082(12)	−0.0001(4)	−0.0005(6)	−0.0005(7)
C(13)	0.0026(5)	0.0025(5)	0.0052(10)	0.0001(4)	0.0006(6)	−0.0006(6)
C(14)	0.0032(5)	0.0036(6)	0.0047(9)	0.0007(4)	−0.0013(6)	−0.0019(6)
C(15)	0.0023(5)	0.0033(5)	0.0035(8)	0.0012(4)	0.0006(5)	−0.0004(5)
C(16)	0.0032(5)	0.0022(4)	0.0023(7)	0.0001(4)	0.0000(5)	−0.0003(4)
C(17)	0.0032(5)	0.0021(4)	0.0052(11)	0.0007(4)	−0.0001(6)	−0.0006(5)
C(18)	0.0038(6)	0.0040(6)	0.0041(10)	0.0005(5)	0.0018(6)	−0.0031(6)
O(9)	0.0045(4)	0.0024(3)	0.0127(10)	−0.0011(3)	−0.0014(6)	−0.0023(5)
O(10)	0.0079(7)	0.0058(6)	0.0100(11)	−0.0012(5)	−0.0027(7)	0.0023(7)
O(11)	0.0035(4)	0.0037(4)	0.0102(9)	0.0009(3)	0.0011(5)	−0.0007(5)
O(12)	0.0025(3)	0.0038(4)	0.0094(8)	0.0001(3)	0.0002(5)	−0.0002(5)
O(13)	0.0054(5)	0.0052(4)	0.0049(7)	−0.0002(4)	−0.0024(5)	0.0010(5)
O(14)	0.0029(4)	0.0081(6)	0.0118(10)	0.0026(4)	0.0002(5)	−0.0027(7)
O(15)	0.0057(5)	0.0038(4)	0.0100(10)	0.0004(4)	−0.0002(6)	0.0033(5)
O(16)	0.0045(4)	0.0047(4)	0.0045(6)	0.0007(4)	−0.0006(4)	−0.0030(5)
O(17)	0.0031(4)	0.0045(4)	0.0079(8)	0.0000(3)	−0.0021(5)	−0.0016(5)
O(18)	0.0066(6)	0.0048(5)	0.0084(10)	−0.0021(4)	0.0020(6)	0.0006(6)

Atom	B (Å^2)	Atom	B (Å^2)	Atom	B (Å^2)
H(5a)	3.83	H(6b)	3.73	H(7c)	4.50
H(5b)	3.83	H(6c)	3.73	H(8a)	4.21
H(5c)	3.83	H(7a)	4.50	H(8b)	4.21
H(6a)	3.73	H(7b)	4.50	H(8c)	4.21

[a] The anisotropic parameters are in the form: exp $-(\beta_{11}h^2 + \beta_{22}k^2 + \beta_{33}l^2 + 2\beta_{12}hk + 2\beta_{13}hl + 2\beta_{23}kl)$.

standard tables [36] and those for hydrogen atoms from a paper ref. [37]. Anomalous dispersion effects were included in the scattering factors for W.

The positional and thermal parameters are listed in Tables 3 and 4. A list of structure factors can be obtained from the author on request.

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