

NIOBIUM ORGANOMETALLIC CHEMISTRY

V*. CRYSTAL AND MOLECULAR STRUCTURE OF BIS(η^5 -CYCLOPENTADIENYL)PEROXOCHLORONIObIUM(V) [$(\eta^5\text{-C}_5\text{H}_5)_2\text{Nb}(\text{O}-\text{O})\text{Cl}$]

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Summary

The structure of bis(η^5 -cyclopentadienyl) peroxochloroniobium(V) has been determined from diffractometer data by X-ray methods; $a = 17.81$, $b = 6.343$, $c = 26.156$ Å, $\beta = 138.28^\circ$, space group $P2_1/c$, Mo- K_α radiation, 3706 independent reflections with $I > 2\sigma(I)$, final $R = 0.036$. Each Nb atom is η^5 bonded to two C_5H_5 rings. The two oxygen atoms ($\text{O}-\text{O} = 1.47$, $\text{Nb}-\text{O} = 1.97$ and 2.00 Å) and the chlorine atom ($\text{Nb}-\text{Cl} = 2.483$ Å) are in the plane (within ± 0.02 Å) which bisects the $(\eta^5\text{-C}_5\text{H}_5)_2\text{Nb}$ bent sandwich system.

Cyclooctasulfur reacts with the bis(η -cyclopentadienyl) compounds Cp_2MX_2 ($\text{Cp} = \eta^5\text{-C}_5\text{H}_5$; $\text{M} = \text{Ti}$, $\text{X} = \text{R}$; $\text{M} = \text{Mo}$, W , $\text{X} = \text{H}$) to undergo substitution reactions which afford the sulfurmetallocyclic complexes Cp_2TiS_5 [1] and Cp_2MS_4 ($\text{M} = \text{Mo}$, W) [2]. With niobium, starting from the dialkyl derivatives Cp_2NbR_2 , we observed the formation of $[\text{Cp}_2\text{Nb}(\text{S}-\text{S})\text{R}]$, i.e. the oxidation of a seventeen electron niobium(IV) species into an eighteen electron niobium(V) derivative [3]. The presence in these complexes of a three-membered NbS_2 ring, clearly shown by a crystal structure determination [4], prompted us to study the formation of the oxo-analogs, i.e. the peroxoderivatives. With Group VIII metals such complexes may be prepared from molecular dioxygen [5]. With niobium, starting from the dialkyls Cp_2NbR_2 , this reaction gives only oxo derivatives like

* For Part IV see ref. 9.

$\text{Cp}_2\text{Nb}(\text{O})\text{R}$ [6] and $[(\text{Cp}_2\text{NbR})_2\text{O}]$ [7], while, after reduction with sodium, Cp_2NbCl_2 gives $\text{Cp}_2\text{Nb}(\text{O})\text{Cl}$ [8]. We recently reported the synthesis of peroxo-complexes $[(\text{R}-\text{Cp})_2\text{Nb}(\text{O}-\text{O})\text{Cl}]$ from the reaction of $[(\text{R}-\text{Cp})_2\text{NbX}_2]$ with hydrogen peroxide [9]. Since these compounds seem to be the first well-characterized organometallic peroxoderivatives of Group IV, V and VI metals, it appeared to us important to carry out an X-ray structure analysis.

The structural data we report here confirm the molecular structure we previously proposed on the basis of a molecular orbital study of Cp_2MX_n complexes [10].

Results

The interatomic distances and angles are given in Table 1. The crystal structure consists of the packing of discrete molecules separated by Van der Waals contacts. The asymmetric unit consists of two independent $\text{Cp}_2\text{Nb}(\text{O}-\text{O})\text{Cl}$ molecules. Both of them display the overall geometry expected for a $(\eta^5\text{-Cp})_2\text{MX}_3$ compound: the two oxygen atoms of a side-on bonded O_2 ligand and the chlorine atom are in the plane (within ± 0.02 Å) bisecting the $(\eta^5\text{-C}_5\text{H}_5)_2\text{Nb}$ bent sandwich system (Table 2 and Figure 1). From another point of view the niobium coordination can be regarded as pseudotetrahedral, the pseudotetrahedron around the metal consisting of the two centroids of the cyclopentadienyl groups, the O_2 midpoint, and the chlorine atom.

The $\text{O}-\text{O}$ bond lengths [$1.47(1)$ Å] are in the range of those found in other peroxoniobium compounds (Table 3). The $\text{Nb}-\text{O}$ bond lengths and the $\text{O}-\text{Nb}-\text{O}'$ and $\text{Nb}-\text{O}-\text{O}'$ angles fall within the expected range (Table 3). The side-on bonding of the O_2^{2-} ligand to the central atom is not significantly asymmetric [$\text{Nb}-\text{O}(1) = 2.00(1)$; $\text{Nb}-\text{O}(2) = 1.97(1)$ Å] but similar differences exist in other $[\text{Cp}_2\text{Nb}(\text{X}-\text{Y})\text{Y}]$ compounds such as $[\text{Cp}_2\text{Nb}(\text{S}-\text{S})(\text{CN})][\text{Nb}-\text{S} =$

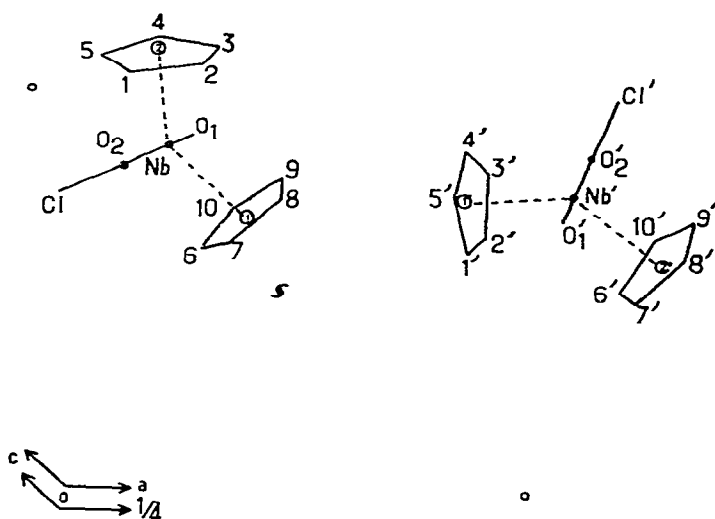


Fig. 1. View of the structure down the b axis.

TABLE 1

SELECTED INTERATOMIC DISTANCES (Å) AND ANGLES (°) (STANDARD DEVIATIONS ARE GIVEN IN PARENTHESES)

<i>Distances</i>			
Nb—Cl	2.483(3)	O(1)—O(2)	1.47(1)
Nb—O(1)	2.00(1)	Cl—O(1)	3.93(1)
Nb—O(2)	1.97(1)	Cl—O(2)	2.84(1)
Nb—C(1)	2.45(1)	C(1)—C(2)	1.40(2)
Nb—C(2)	2.46(1)	C(2)—C(3)	1.40(2)
Nb—C(3)	2.41(1)	C(3)—C(4)	1.41(2)
Nb—C(4)	2.44(1)	C(4)—C(5)	1.37(2)
Nb—C(5)	2.45(1)	C(5)—C(1)	1.41(2)
Nb—C(6)	2.44(2)	C(6)—C(7)	1.33(2)
Nb—C(7)	2.44(2)	C(7)—C(8)	1.39(2)
Nb—C(8)	2.44(2)	C(8)—C(9)	1.38(2)
Nb—C(9)	2.44(2)	C(9)—C(10)	1.42(2)
Nb—C(10)	2.41(2)	C(10)—C(6)	1.39(2)
	2.44 (av)		1.39 (av)
<i>Distances</i>			
Nb—Cp(1)	2.13(1) ^a		
Nb—Cp(2)	2.13(1) ^a		
Nb'—Cl'	2.480(3)	O(1')—O(2')	1.47(1)
Nb'—O(1')	2.00(1)	Cl'—O(1')	3.92(1)
Nb'—O(2')	1.97(1)	Cl'—O(2')	2.83(1)
Nb'—C(1')	2.41(1)	C(1')—C(2')	1.42(2)
Nb'—C(2')	2.46(1)	C(2')—C(3')	1.38(2)
Nb'—C(3')	2.44(1)	C(3')—C(4')	1.40(2)
Nb'—C(4')	2.44(1)	C(4')—C(5')	1.38(2)
Nb'—C(5')	2.44(2)	C(5')—C(1')	1.38(2)
Nb'—C(6')	2.43(2)	C(6')—C(7')	1.42(3)
Nb'—C(7')	2.43(2)	C(7')—C(8')	1.38(2)
Nb'—C(8')	2.46(1)	C(8')—C(9')	1.35(2)
Nb'—C(9')	2.44(2)	C(9')—C(10')	1.37(3)
Nb'—C(10')	2.41(2)	C(10')—C(6')	1.40(3)
	2.44 (av)		1.39 (av)
<i>Distances</i>			
Nb'—Cp(1')	2.13(1) ^a		
Nb'—Cp(2')	2.13(1) ^a		
<i>Angles</i>			
Cl—Nb—O(1)	121.9(3)	Cl'—Nb'—O(1')	121.6(3)
Cl—Nb—O(2)	78.2(3)	Cl'—Nb'—O(2')	78.1(3)
O(1)—Nb—O(2)	43.6(3)	O(1')—Nb'—O(2')	43.6(3)
Nb—O(1)—O(2)	66.9(7)	Nb'—O(1')—O(2')	67.1(7)
Nb—O(2)—O(1)	69.5(7)	Nb'—O(2')—O(1')	69.3(7)
C(1)—C(2)—C(3)	107(2)	C(1')—C(2')—C(3')	108(2)
C(2)—C(3)—C(4)	108(2)	C(2')—C(3')—C(4')	108(2)
C(3)—C(4)—C(5)	109(2)	C(3')—C(4')—C(5')	107(2)
C(4)—C(5)—C(1)	107(2)	C(4')—C(5')—C(1')	110(3)
C(5)—C(1)—C(2)	109(2)	C(5')—C(1')—C(2')	107(2)
C(6)—C(7)—C(8)	110(3)	C(6')—C(7')—C(8')	106(3)
C(7)—C(8)—C(9)	107(3)	C(7')—C(8')—C(9')	110(3)
C(8)—C(9)—C(10)	107(3)	C(8')—C(9')—C(10')	110(3)
C(9)—C(10)—C(6)	106(3)	C(9')—C(10')—C(6')	107(3)
C(10)—C(6)—C(7)	109(3)	C(10')—C(6')—C(7')	108(3)
Cp(1)—Nb—Cp(2)	128.1(4)	Cp(1')—Nb'—Cp(2')	128.6(4)
Cp(1)—Nb—O ₂ (midpoint)	110.5(4)	Cp(1')—Nb'—O ₂ ' (midpoint)	109.9(4)
Cp(2)—Nb—O ₂ (midpoint)	108.7(4)	Cp(2')—Nb'—O ₂ ' (midpoint)	109.2(4)
Cl—Nb—O ₂ (midpoint)	100.3(3)	Cl'—Nb'—O ₂ ' (midpoint)	100.0(3)
Cl—Nb—Cp(1)	101.5(4)	Cl'—Nb'—Cp(1')	102.0(4)
Cl—Nb—Cp(2)	103.6(4)	Cl'—Nb'—Cp(2')	102.6(4)

^a Average perpendicular distances between Nb atom and the cyclopentadienyl rings.

TABLE 2

EQUATIONS OF AND DISTANCES FROM LEAST-SQUARES BEST PLANES AND INTERPLANAR ANGLES

(a) Equations of least-squares planes

The equations are of the form $Ax + By + Cz + D = 0$ where x, y, z (in Å) refer to the orthogonal system a, b, c^* .

Plane	Atoms in plane	A	B	C	D
P	Nb, Cl, O(1) and O(2)	0.4293	-0.0405	-0.9023	5.8625
P'	Nb', Cl', O(1') and O(2')	0.9467	-0.0053	-0.321	-7.2096
P1	five atoms C(1) to C(5)	0.0829	-0.332	-0.940	9.150
P2	five atoms C(6) to C(10)	0.697	0.250	-0.672	1.302
P'1	five atoms C(1') to C(5')	-0.954	0.299	-0.015	6.945
P'2	five atoms C(6') to C(10')	0.739	0.303	-0.602	-5.894

(b) Distances (Å) from the least-squares planes

from P: Nb: 0.0017; Cl: -0.0051; O(1): -0.024; O(2): 0.020.

from P': Nb': 0.0001; Cl': -0.0003; O(1)': -0.001; O(2)': 0.001.

from P1: C(1): 0.003; C(2): -0.012; C(3) 0.018; C(4): -0.016; C(5): 0.008.

from P2: C(6): 0.008; C(7): 0.000; C(8): -0.008; C(9): 0.013; C(10): -0.012

from P'1: C(1)': -0.026; C(2)': 0.021; C(3)': -0.010; C(4)': -0.006; C(5)': 0.020.

from P'2: C(6)': 0.021; C(7)': -0.018; C(8)': 0.009; C(9)': 0.000; C(10)': -0.012.

(c) Interplanar angles (°)

P-P1: 26.3; P-P2: 26.5; P'-P'1: 25.9; P'-P'2: 26.9.

TABLE 3

COMPARISON OF PEROXO GROUP PARAMETERS IN DIFFERENT NIOBIUM DERIVATIVES

Compound	O-O' (Å)	Nb-O (Å)	O-Nb-O' (°)	Nb-O-O' (°)	ref.
(NH ₂) ₃ [Nb(O-O) ₂ (C ₂ O ₄) ₂] · H ₂ O	1.480(6)	{1.961(4) 1.981(4)}	44.1(2)	68.7(2) 67.2(2)	11
	1.485(6)	{1.982(4) 1.969(4)}	44.1(2)	67.5(2) 68.4(2)	
K[Nb(O-O) ₃ (C ₁₂ H ₈ N ₂)] · 3 H ₂ O	1.496(11)	{1.980(7) 1.981(7)}	44.4(3)	67.8(4) 67.8(4)	12
	1.470(10)	{1.988(7) 1.981(7)}	43.5(3)	68.0(4) 68.5(4)	
	1.500(8)	{2.014(7) 1.979(7)}	44.1(3)	66.7(4) 69.2(4)	
KMg[Nb(O-O) ₄] · 7 H ₂ O	1.500(3)	{1.993(4) 2.066(3)}	43.3(1)	70.9(1) 65.7(1)	13
	1.504(6)	{2.011(2) 2.004(3)}	44.0(1)	67.7(1) 68.3(1)	
	1.495(4)	{2.015(2) 2.063(2)}	43.0(1)	70.2(1) 66.8(1)	
	1.502(3)	{2.014(2) 2.038(2)}	43.5(1)	69.1(1) 67.4(1)	
(C ₅ H ₅) ₂ Nb(O-O)Cl	1.47(1)	{2.00(1) 1.97(1)}	43.6(3)	66.9(7) 69.5(7)	this work
	1.47(1)	{2.00(1) 1.97(1)}	43.6(3)	67.1(7) 69.3(7)	

TABLE 4

COMPARISON OF $[(\eta^5\text{-C}_5\text{H}_5)_2\text{Nb}]$ MOIETY PARAMETERS IN DIFFERENT NIOBIUM DERIVATIVES

	ω ($^\circ$) ^a	Nb-ring (Å)	Nb—C(av) (Å)	ref.
$[\text{Cp}_2\text{Nb}(\text{S—S})(\text{CH}_3)]$	128.9	2.112 2.108	2.419(5) 2.418(7)	4
$[\text{Cp}_2\text{Nb}(\text{S—S})(\text{CN})]$	131.8 132.7		2.40(2) 2.41(2)	14
$[\text{Cp}_2\text{Nb}(\text{C}_2\text{H}_4)(\text{C}_2\text{H}_5)]$	132.4	2.096	2.402(5)	15
$[\{\text{Cp}_2\text{Nb}(\text{SMe})_2\}_2\text{Ni}]^{2+}$	129.4 129.9	2.14 2.08 2.09 2.10	2.43	16
$[\text{Cp}_2\text{NbH}(\text{CO})]$	143	2.04(4)	2.36(4)	17
$[\text{Cp}_2\text{NbH}_3]$	141.6	2.057(10) 2.061(11)	2.375(4)	18
$[\text{Cp}_2\text{Nb}(\text{O—O})\text{Cl}]$	127.3 127.2	2.13(1)	2.44(2)	this work

^a The bending angle ω is defined as the angle between the normals to the C_5H_5 rings.

2.521(6) and 2.458(5)] [14], $[\text{Cp}_2\text{Nb}(\text{C}_2\text{H}_4)(\text{C}_2\text{H}_5)]$ [for C_2H_5 : Nb—C = 2.320(9) and 2.277(9) Å] [15] and $[\text{Cp}_2\text{Nb}(\text{S—S})(\text{CH}_3)]$ [Nb—S = 2.515(2) and 2.432(3) Å] [4], the shortest Nb—X bond corresponding in all these cases to the X ligand occupying the central position, i.e. lying near the Y atom.

The Nb—Cl bond lengths [2.483(3) and 2.480(3)] are slightly longer than that reported in $[\text{Cp}_2\text{Nb}(\text{S—S})\text{Cl}]$ (2.43 Å) [14].

The geometry of the $[(\eta^5\text{-C}_5\text{H}_5)_2\text{Nb}]$ moiety agrees well with that found in other bent bis(η^5 -cyclopentadienyl)systems (Table 4). The η^5 -cyclopentadienyl rings are staggered, the Nb atom being at 2.13 Å along the normals to the rings. The bending angle, defined as the angle between the normals to the C_5H_5 rings, is 127.2°. The C—C bonds are in the range 1.33–1.42 Å (mean 1.39 Å) and show no systematic variation. The Nb—C distances are in the range 2.41–2.46 Å (mean 2.44 Å) in good agreement with the mean distance (2.43 Å) in $[\{\text{Cp}_2\text{Nb}(\text{SMe})_2\}_2\text{Ni}](\text{BF}_4)_2 \cdot 2 \text{H}_2\text{O}$ [16].

Experimental

The compound was prepared by reaction of hydrogen peroxide with bis(η^5 -cyclopentadienyl)niobium dichloride $[(\eta^5\text{-C}_5\text{H}_5)_2\text{NbCl}_2]$ in dichloromethane or chloroform following the procedure previously described [9]. Yellow crystals, slightly air unstable, were obtained by recrystallisation from a $\text{CH}_2\text{Cl}_2\text{—CH}_3\text{CN}$ mixture.

Crystal data

$\text{C}_{10}\text{ClH}_{10}\text{NbO}_2$. $M = 290.55$, monoclinic; $a = 17.81(1)$, $b = 6.343(5)$, $c = 26.156(6)$ Å, $\beta = 138.28(5)^\circ$, $V = 1965$ Å³, $D_m = 1.93$, $Z = 8$, $D_c = 1.96$, Mo- K_α radiation, $\lambda = 0.7107$ Å, $\mu(\text{Mo-}K_\alpha) = 14.14$ cm⁻¹. Systematic absences:

(continued on p. 314)

TABLE 5
FINAL ATOMIC FRACTIONAL COORDINATES AND THERMAL PARAMETERS^a

Atom	x	y	z	β_{11}	β_{22}	β_{33}	β_{12}	β_{13}	β_{23}
Nb	5022.5(4)	1390.9(7)	4203.2(3)	35.4(3)	90(1)	18.4(2)	-3.5(5)	19.2(2)	-1.5(4)
Cl	3786(1)	2652(3)	3610(1)	33(1)	312(5)	29(1)	8(2)	21(1)	-2(2)
O(1)	5010(4)	-1676(6)	4969(3)	117(5)	116(11)	54(2)	31(6)	64(3)	14(4)
O(2)	4864(4)	-1277(7)	3983(3)	91(4)	160(12)	43(2)	-50(6)	50(3)	-23(4)
C(1)	6185(5)	4034(9)	5125(3)	52(5)	146(15)	26(2)	-2(7)	25(4)	-15(5)
C(2)	7067(5)	3924(9)	5218(3)	45(4)	179(17)	22(2)	-36(7)	20(3)	-18(5)
C(3)	7522(5)	1889(11)	5477(3)	32(4)	303(22)	21(2)	13(8)	15(3)	-7(6)
C(4)	6946(5)	806(10)	5575(3)	61(5)	182(17)	21(2)	10(8)	22(3)	1(6)
C(5)	6112(5)	2081(10)	5345(3)	54(6)	198(17)	19(2)	-17(7)	25(3)	-9(5)
C(6)	4588(6)	2572(13)	2925(4)	77(6)	387(27)	27(3)	35(11)	34(4)	40(7)
C(7)	5013(6)	694(12)	3018(4)	94(7)	291(23)	35(3)	-47(11)	47(4)	-26(7)
C(8)	6183(6)	796(13)	3602(4)	91(7)	332(25)	45(3)	22(11)	56(4)	17(7)
C(9)	6483(6)	2849(13)	3870(4)	84(7)	439(30)	33(3)	-100(12)	45(4)	-35(8)
C(10)	5469(8)	3983(10)	3453(5)	173(10)	151(18)	48(3)	-25(11)	81(6)	-3(6)
Nb'	9383.5(4)	1536.9(7)	3572.8(3)	35.4(3)	94.3(1.1)	18.2(2)	9.0(5)	18.8(2)	5.0(4)
Cl'	11223(1)	2811(3)	4859(1)	36(1)	349(6)	23(1)	-20(2)	15(1)	0(2)
O(1')	9002(4)	-1417(6)	3310(3)	104(5)	96(10)	44(2)	-7(6)	50(3)	-12(4)
O(2')	10133(4)	-1063(6)	4101(3)	85(4)	168(12)	39(2)	60(6)	45(3)	38(4)
C(1')	7488(5)	1997(12)	2926(4)	31(4)	347(24)	24(2)	-13(8)	18(3)	-19(6)
C(2')	7931(5)	4084(10)	3146(4)	59(5)	215(19)	35(3)	51(8)	37(3)	28(6)
C(3')	8813(5)	4156(10)	3938(4)	63(6)	182(17)	33(2)	10(8)	35(3)	-14(5)
C(4')	8895(5)	2184(10)	4219(3)	55(5)	262(20)	28(2)	40(8)	32(3)	19(6)
C(5')	8063(5)	919(10)	3995(4)	59(5)	166(18)	53(3)	3(8)	47(4)	14(6)
C(6')	8661(8)	3627(16)	2481(5)	139(9)	628(41)	54(4)	236(17)	80(6)	161(11)

	x	y	z	$B(\text{\AA}^3)$						
C(7')	8702(6)	1470(15)	2352(4)	63(6)	534(33)	21(2)	-20(12)	26(3)	-7(8)	
C(8')	9834(6)	962(10)	2900(4)	91(6)	201(18)	34(3)	26(9)	49(4)	10(6)	
C(9')	10462(6)	2624(12)	3364(4)	89(7)	280(22)	43(3)	-31(10)	54(4)	-10(7)	
C(10')	9770(8)	4289(11)	3131(5)	165(10)	178(19)	66(4)	25(12)	94(6)	23(8)	

	x	y	z	$B(\text{\AA}^3)$
H(1)	586(4)	532(10)	503(3)	3.4
H(2)	727(5)	492(10)	508(3)	3.1
H(3)	816	134	559	3.5
H(4)	703(5)	-39(10)	571(3)	3.8
H(5)	567(5)	183(9)	537(3)	3.8
H(6)	365(5)	296(10)	257(4)	2.4
H(7)	462(5)	-34(10)	277(3)	2.9
H(8)	650(5)	1(10)	380(4)	2.9
H(9)	716(5)	321(10)	425(4)	3.0
H(10)	553(5)	508(11)	355(4)	2.3
H(1')	692(5)	166(10)	247(8)	3.0
H(2')	760(5)	514(10)	272(3)	2.9
H(3')	911(5)	522(10)	419(3)	2.9
H(4')	943(5)	195(10)	473(3)	2.6
H(5')	797(5)	-36(10)	360(3)	4.7
H(6')	797	448	218	4.7
H(7')	804	55	194	4.1
H(8')	1005(5)	-44(10)	294(3)	2.7
H(9')	1135(5)	274(11)	387(4)	3.5
H(10')	993(5)	532(11)	331(4)	3.9

^a For non hydrogen atoms, fractional coordinates and thermal parameters $\times 10^4$. Form of the thermal ellipsoid: $\exp[-(\beta_{11}h^2 + \beta_{22}k^2 + \beta_{33}l^2 + 2\beta_{12}hk + 2\beta_{13}hl + 2\beta_{23}kl)]$ for hydrogen atoms: fractional coordinates $\times 10^3$ and thermal parameters.

$h0l : l = 2n + 1$ and $0k0 : k = 2n + 1$; space group : $P2_1/c$. (density measured by flotation in a $C_2H_5I-CCl_4$ solution). Preliminary cell dimensions were derived from Weissenberg and precession films. Accurate values were obtained from diffractometer settings.

The crystal selected for X-ray analysis was approximately a parallelepiped ($0.60 \times 0.48 \times 0.28$ mm) and was sealed in a Lindemann tube. The intensity data were collected on a Nonius CAD-4 automatic four circle diffractometer using $Mo-K_\alpha$ radiation with a graphite crystal monochromator. Intensity data for reflections with $\theta < 30^\circ$ were collected using the $\omega-2\theta$ stepscan technique. Three standard reflections were measured periodically to check crystal and electronic stability. Of the 4383 independent intensities measured at room temperature, only 3706 for which $I > 2\sigma(I)$ were accepted as being significantly above background and were used in subsequent calculations. No absorption or extinction corrections were applied.

Structure solution and refinement

Starting with atomic positional parameters generated from the best electron density map produced by MULTAN [19], all non-hydrogen atoms were located after a Fourier synthesis. All but three H atoms were located on a difference map, positions for the others were calculated. The structure was refined by the full matrix least-squares method, the weighting scheme was $1/\sigma^2(F)$ where $\sigma(F)$ were determined from counting statistics. Scattering factors (including real anomalous dispersion correction for Nb) were taken from International Tables of Crystallography [20]. The thermal factors of the hydrogen atoms were set equal to those of carbon binding atoms and were not allowed to vary. Anisotropic refinement of the atoms excluding hydrogen resulted in a final R value of 0.036 ($R_w = 0.043$). The positional and thermal parameters obtained from the last least squares refinement cycle are presented in Table 4. Interatomic distances and angles, with their estimated standard deviations, are provided in Table 1 in which the hydrogen atoms are given with the number of the carbon atom to which they are bonded. The figure shows a view of the structure down the b axis and the numbering of the atoms.

Tables of structure factors can be obtained from the authors.

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