Crystal structure of bromo-(*N,N,N',N'*-tetramethyl-1,3-propanediamine)-tricarbonyl-rhenium(I), $Re(CO)_3(C_7H_{18}N_2)Br$

E. Horn*[I,II] and S. Onai[I]

[I] Rikkyo University, Department of Chemistry, 3-34-1 Nishi-Ikebukuro, Tosima-ku, Tokyo 171-8501, Japan
[II] University of Tsukuba, Tsukuba Advanced Research Alliance Center and Department of Chemistry, Tsukuba, Ibaraki 305-8577, Japan

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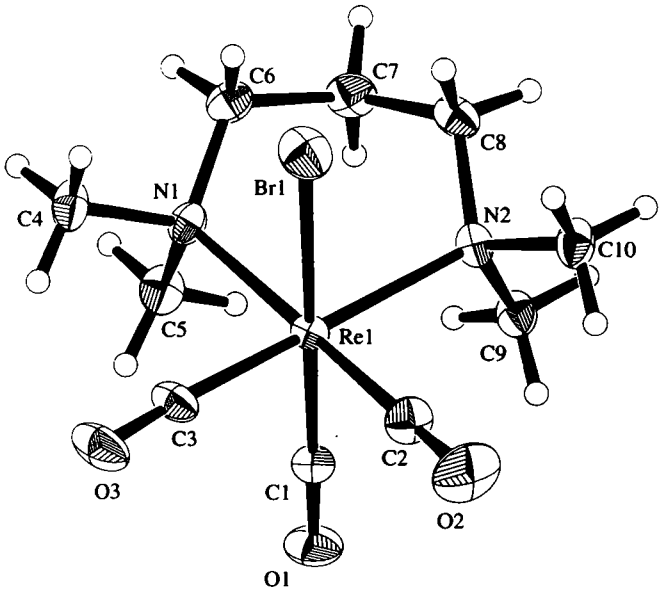

Abstract

$C_{10}H_{18}BrN_2O_3Re$, orthorhombic, *Pbca* (No. 61), $a = 14.496(2)$ Å, $b = 13.338(2)$ Å, $c = 14.722(2)$ Å, $V = 2846.5$ Å^3, $Z = 8$, $R_{gt}(F) = 0.042$, $wR_{all}(F) = 0.074$, $T = 296$ K.

Source of material

The rhenium carbonyl complex was prepared by refluxing a petroleum ether (373-393 K) solution containing $Re(CO)_5Br$ and a 10% molar excess of *N,N,N',N'*-tetramethyl-1,3-propanediamine (LN) for two hours, in a similar way as reported earlier [1]. Transparent yellow-brown crystals of the product were obtained by slow recrystallization from dichloromethane at room temperature.

Discussion

Transition metal (Mn, Re) carbonyl halide complexes with nitrogen or phosphorus containing are stable precursors for the synthesis of a variety of compounds [2,3]. The second interest is the correlation between the CO stretching frequencies and the respective molecular structures [4]. This X-ray analysis report characterizes the stereochemistry of the three carbonyl ligands of the $[Re(CO)_3(LN)Br]$ complex.

The ORTEP plot (30% probability ellipsoids) shows the rhenium atom in a slightly distorted octahedral environment with the respective coordination bond angles in the ranges 86.8(2)°–94.1(4)° and 178.2(4)°–178.9(3)°. The three carbonyl groups are in a mutually *cis* stereochemistry at a distance of between 1.89(1) Å and 1.92(1) Å from the metal atom. The bidentate ligand coordinates in the equatorial positions with an average Re—N bond distance equal to 2.308(9) Å. The bromine atom is located in an axial posit ion with Re(1)—Br(1) = 2.583(2) Å. All other bond lengths and angles are normal.

Table 1. Data collection and handling.

Crystal:	yellow-brown, prismatic, size 0.100 × 0.100 × 0.300 mm
Wavelength:	Mo K_α radiation (0.7107 Å)
μ:	113.57 cm^{-1}
Diffractometer, scan mode:	Rigaku AFC7R, ω/2θ
$2\theta_{max}$:	60°
$N(hkl)_{measured}$, $N(hkl)_{unique}$:	4633, 4633
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 3\ \sigma(I_{obs})$, 2849
$N(param)_{refined}$:	154
Programs:	SIR-92 [5], TEXSAN [6], DIFABS [7], ORTEP-II [8]

Table 2. Atomic coordinates and displacement parameters (in Å^2).

Atom	Site	*x*	*y*	*z*	U_{iso}
H(1)	8*c*	0.8204	0.1195	0.9792	0.059
H(2)	8*c*	0.8171	0.2306	0.9414	0.059
H(3)	8*c*	0.7987	0.2098	1.0457	0.059
H(4)	8*c*	0.6637	0.1386	1.0973	0.054
H(5)	8*c*	0.5870	0.0970	1.0304	0.054
H(6)	8*c*	0.6841	0.0428	1.0369	0.054
H(7)	8*c*	0.6610	0.2977	1.0369	0.052
H(8)	8*c*	0.6823	0.3191	0.9332	0.052
H(9)	8*c*	0.5163	0.2310	0.9878	0.058
H(10)	8*c*	0.5270	0.3489	0.9800	0.058
H(11)	8*c*	0.5631	0.3233	0.8220	0.051
H(12)	8*c*	0.4589	0.3036	0.8489	0.051
H(13)	8*c*	0.3940	0.1467	0.8431	0.051
H(14)	8*c*	0.4512	0.0490	0.8190	0.051
H(15)	8*c*	0.4642	0.1015	0.9147	0.051
H(16)	8*c*	0.5505	0.2313	0.6866	0.054
H(17)	8*c*	0.5019	0.1250	0.6851	0.054
H(18)	8*c*	0.4442	0.2221	0.7099	0.054

* Correspondence author (e-mail: horn@rikkyo.ne.jp)

Table 3. Atomic coordinates and displacement parameters (in $Å^2$).

Atom	Site	x	y	z	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
Re(1)	8*c*	0.66827(2)	0.09782(2)	0.82529(2)	0.0285(1)	0.0280(1)	0.0284(1)	0.0010(1)	0.0050(1)	0.0020(1)
Br(1)	8*c*	0.73900(7)	0.25830(8)	0.75610(8)	0.0559(5)	0.0610(6)	0.0630(6)	-0.0005(5)	0.0068(5)	0.0136(6)
O(1)	8*c*	0.5881(5)	-0.0941(5)	0.9045(5)	0.058(4)	0.035(3)	0.073(4)	-0.011(3)	0.014(3)	0.011(3)
O(2)	8*c*	0.6482(7)	0.0009(7)	0.6395(5)	0.114(7)	0.089(6)	0.045(3)	0.001(5)	0.008(4)	-0.035(4)
O(3)	8*c*	0.8557(4)	-0.0043(5)	0.8347(6)	0.037(3)	0.050(4)	0.118(6)	0.014(3)	0.015(4)	0.017(4)
N(1)	8*c*	0.6888(4)	0.1725(5)	0.9654(4)	0.032(3)	0.041(3)	0.030(2)	-0.007(2)	0.001(2)	-0.001(2)
N(2)	8*c*	0.5269(4)	0.1757(5)	0.8126(4)	0.028(2)	0.043(3)	0.029(3)	0.002(2)	0.004(2)	0.004(2)
C(1)	8*c*	0.6178(5)	-0.0210(6)	0.8742(5)	0.034(4)	0.033(3)	0.032(4)	-0.001(3)	0.008(3)	0.000(3)
C(2)	8*c*	0.6549(7)	0.0372(7)	0.7087(6)	0.052(5)	0.053(5)	0.032(3)	0.002(4)	0.005(4)	-0.006(3)
C(3)	8*c*	0.7875(5)	0.0356(6)	0.8328(7)	0.036(3)	0.034(4)	0.057(5)	0.008(3)	0.009(4)	0.008(4)
C(4)	8*c*	0.7903(6)	0.1841(7)	0.9846(6)	0.035(3)	0.062(6)	0.049(5)	-0.007(4)	-0.010(4)	0.005(5)
C(5)	8*c*	0.6527(6)	0.1071(7)	1.0389(5)	0.048(5)	0.054(5)	0.033(4)	-0.009(4)	0.003(3)	0.011(3)
C(6)	8*c*	0.6499(6)	0.2754(6)	0.9751(6)	0.048(4)	0.042(4)	0.040(4)	-0.003(3)	0.003(4)	-0.009(4)
C(7)	8*c*	0.5477(6)	0.2850(7)	0.9562(6)	0.048(4)	0.047(5)	0.048(4)	0.008(4)	0.011(4)	-0.012(4)
C(8)	8*c*	0.5216(6)	0.2795(6)	0.8552(6)	0.038(4)	0.039(3)	0.049(4)	0.007(3)	0.007(3)	0.006(3)
C(9)	8*c*	0.4527(5)	0.1129(7)	0.8506(6)	0.028(3)	0.047(5)	0.052(5)	-0.006(3)	0.000(3)	0.011(4)
C(10)	8*c*	0.5038(6)	0.1897(7)	0.7151(5)	0.046(5)	0.057(5)	0.032(3)	-0.006(4)	-0.010(4)	0.009(4)

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