Crystal structure of dichlorotetrakis(cyclohexylisocyanide)dioxo-μ_2-oxo-dirhenium(V), $C_{28}H_{44}Cl_4N_4O_3Re_2$

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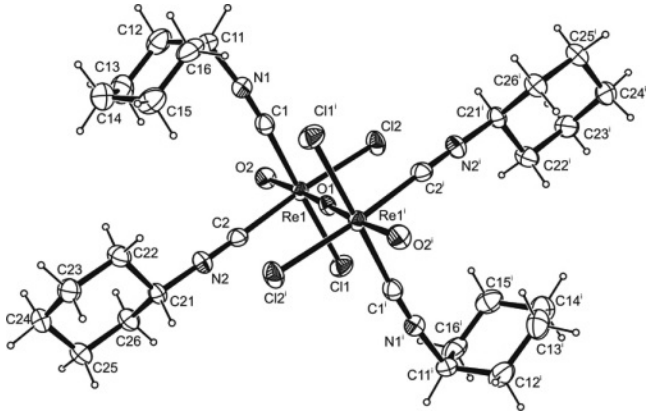

Abstract

$C_{28}H_{44}Cl_4N_4O_3Re_2$, monoclinic, $P2_1/n$ (no. 14), a = 12.1765(6) Å, b = 9.8302(5) Å, c = 14.3784(7) Å, β = 99.695(2)°, V = 1696.5 Å^3, Z = 2, $R_{gt}(F)$ = 0.0221, $wR_{ref}(F^2)$ = 0.0642, T = 200 K.

Table 1. Data collection and handling.

Crystal:	blue platelets, size 0.05×0.46×0.52 mm
Wavelength:	Mo K_α radiation (0.71073 Å)
μ:	74.79 cm^{-1}
Diffractometer, scan mode:	Bruker APEX-II CCD, φ and ω
$2\theta_{max}$:	56.66°
$N(hkl)_{measured}$, $N(hkl)_{unique}$:	29529, 4217
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\ \sigma(I_{obs})$, 3797
$N(param)_{refined}$:	187
Programs:	SHELX [8], WinGX [9], MERCURY [10], PLATON [11]

Source of material

The compound was obtained upon reacting $Re(Me_2S)OCl_3(OPPh_3)$ with cyclohexyl isocyanide in dichloromethane, ethanol and acetonitrile. Crystals suitable for the diffraction study were obtained upon free evaporation of the reaction batch at room temperature.

Experimental details

Carbon-bound H atoms were placed in calculated positions (C–H 0.99 Å for methylene groups and C–H 1.00 Å for methine groups) and were included in the refinement in the riding model approximation, with U_{iso}(H) set to 1.2U_{eq}(C).

Discussion

Next to cardiovascular diseases, cancer has become one of the main fatal diseases in industrialized countries. Apart from classical surgery, chemo- and radiotherapeutic treatments have entered the arsenal of possible cures for certain types of cancer. All methods, however, suffer from their own set of problematic side-effects and, as a consequence, the development of radiopharmaceuticals – combining the advantages of chemotherapy as well as radiation methods while at the same time avoiding their unique respective undesired side-effects – has been a topic of research [1, 2]. Tailoring and fine-tuning of the envisioned radiopharmaceuticals' properties such as lipophilicity and, in particular, inertness is of paramount importance with respect to possible future *in vivo* applications in contemporary medicine and requires sound knowledge about structural parameters of the ligands applied if a more heuristic approach in the synthesis is to triumph over pure trial-and-error as it is encountered in this specific field of coordination chemistry up to the present day. In continuation of our study of rhenium-based coordination compounds that might serve as radiopharmaceuticals, the title compound was synthesized. The results of a structural analysis of a similar compound featuring aromatic isonitrile ligands have recently been described in the literature [3] as has been the case for a compound featuring bridging methanolate ligands [4]. The compound is a dinuclear centrosymmetric rhenium(V) coordination compound. The rhenium metal centers are hexacoordinated. The respective octahedral coordination sphere is set up by two cyclohexylisocyanido ligands in *trans* position to a chlorido ligand, an oxido ligand and a bridging μ_2-oxido ligand. The Re–Cl bond lengths were measured at 2.3920(9) Å and 2.3953(8) Å while the Re–C bond lengths were found at 2.079(3) Å and 2.090(4) Å. The two rhenium-oxygen bond lengths clearly allow for a separation into the shorter Re=O double bond with 1.701(2) Å and the Re–O single bond with 1.91189(13) Å. These two values are in good agreement with data available for other compounds featuring a O=Re–O–Re=O motif whose metrical parameters have been deposited with the Cambridge Structural Database [5]. The angles at the rhenium atom between *trans* orientated ligands adopt values of 168.46(9)°, 171.77(10) and 175.53(10)° with the smallest value realized for the O=Re–O fragment. A conformational analysis of the cyclohexyl rings according to Cremer & Pople [6] shows these to adopt a $^1C_4$ and a $^4C_1$ conformation [7].

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

Atom	Site	*x*	*y*	*z*	U_{iso}
H(11)	4*e*	0.3611	0.3087	0.7405	0.041
H(12A)	4*e*	0.5338	0.3990	0.7264	0.050
H(12B)	4*e*	0.4797	0.3431	0.6244	0.050
H(13A)	4*e*	0.5626	0.5626	0.6164	0.053
H(13B)	4*e*	0.4334	0.5619	0.5701	0.053
H(14A)	4*e*	0.5229	0.6648	0.7535	0.054
H(14B)	4*e*	0.4676	0.7550	0.6651	0.054
H(15A)	4*e*	0.2926	0.6629	0.6709	0.050
H(15B)	4*e*	0.3426	0.7228	0.7728	0.050
H(16A)	4*e*	0.2621	0.5042	0.7838	0.048
H(16B)	4*e*	0.3910	0.5061	0.8307	0.048
H(21)	4*e*	0.1405	0.9313	0.4206	0.030
H(22A)	4*e*	0.3552	0.8452	0.5181	0.038
H(22B)	4*e*	0.2501	0.8769	0.5685	0.038
H(23A)	4*e*	0.2515	1.1073	0.5240	0.042
H(23B)	4*e*	0.3733	1.0689	0.5784	0.042
H(24A)	4*e*	0.4412	1.0521	0.4373	0.045
H(24B)	4*e*	0.3812	1.1967	0.4393	0.045
H(25A)	4*e*	0.3317	1.0874	0.2903	0.041
H(25B)	4*e*	0.2244	1.1187	0.3376	0.041
H(26A)	4*e*	0.2094	0.8940	0.2784	0.036
H(26B)	4*e*	0.3298	0.8562	0.3367	0.036

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)	4*e*	0.10149(1)	0.42018(1)	0.427726(8)	0.02050(9)	0.01815(8)	0.02346(8)	–0.00136(4)	0.00345(5)	–0.00295(4)
Cl(1)	4*e*	–0.03192(8)	0.4475(1)	0.28653(7)	0.0345(5)	0.0394(4)	0.0288(4)	–0.0002(4)	–0.0047(4)	–0.0037(4)
Cl(2)	4*e*	0.02359(8)	0.20049(8)	0.44769(7)	0.0349(5)	0.0199(3)	0.0458(5)	–0.0059(3)	0.0095(4)	–0.0055(3)
O(1)	2*a*	0	½	½	0.022(2)	0.020(1)	0.023(2)	–0.002(1)	0.009(1)	–0.000(1)
O(2)	4*e*	0.2104(2)	0.3721(3)	0.3750(2)	0.030(1)	0.028(1)	0.036(1)	0.003(1)	0.011(1)	–0.001(1)
N(1)	4*e*	0.2736(3)	0.3944(3)	0.6235(2)	0.027(2)	0.029(1)	0.034(2)	–0.002(1)	0.000(1)	0.004(1)
N(2)	4*e*	0.1773(3)	0.7329(3)	0.4174(2)	0.032(2)	0.022(1)	0.032(2)	–0.004(1)	0.007(1)	–0.000(1)
C(1)	4*e*	0.2087(3)	0.4023(3)	0.5569(3)	0.027(2)	0.020(1)	0.034(2)	–0.001(1)	0.008(2)	–0.002(1)
C(2)	4*e*	0.1503(3)	0.6218(3)	0.4190(2)	0.029(2)	0.025(2)	0.022(2)	0.000(1)	–0.001(1)	0.001(1)
C(11)	4*e*	0.3614(3)	0.3967(4)	0.7058(3)	0.028(2)	0.034(2)	0.036(2)	–0.003(2)	–0.007(2)	0.012(2)
C(12)	4*e*	0.4729(4)	0.4135(4)	0.6724(3)	0.030(2)	0.041(2)	0.051(2)	0.007(2)	–0.002(2)	–0.005(2)
C(13)	4*e*	0.4860(4)	0.5532(5)	0.6303(3)	0.035(2)	0.057(2)	0.043(2)	–0.008(2)	0.012(2)	–0.001(2)
C(14)	4*e*	0.4641(4)	0.6661(4)	0.6968(3)	0.044(2)	0.039(2)	0.050(2)	–0.008(2)	–0.001(2)	0.000(2)
C(15)	4*e*	0.3518(3)	0.6508(4)	0.7265(3)	0.041(2)	0.042(2)	0.040(2)	0.007(2)	–0.003(2)	–0.008(2)
C(16)	4*e*	0.3388(3)	0.5129(5)	0.7701(3)	0.030(2)	0.062(3)	0.026(2)	0.005(2)	–0.002(2)	0.005(2)
C(21)	4*e*	0.2091(3)	0.8748(3)	0.4220(2)	0.028(2)	0.017(1)	0.029(2)	–0.003(1)	0.005(1)	–0.001(1)
C(22)	4*e*	0.2873(3)	0.9015(3)	0.5148(3)	0.041(2)	0.027(2)	0.026(2)	–0.002(2)	0.003(2)	0.002(1)
C(23)	4*e*	0.3188(4)	1.0523(4)	0.5202(3)	0.042(2)	0.029(2)	0.032(2)	–0.005(2)	–0.004(2)	–0.003(1)
C(24)	4*e*	0.3683(4)	1.0972(4)	0.4357(3)	0.034(2)	0.027(2)	0.049(2)	–0.008(2)	0.001(2)	0.005(2)
C(25)	4*e*	0.2932(4)	1.0635(4)	0.3434(3)	0.037(2)	0.029(2)	0.036(2)	–0.004(2)	0.008(2)	0.009(1)
C(26)	4*e*	0.2622(3)	0.9120(3)	0.3373(3)	0.035(2)	0.029(2)	0.029(2)	–0.003(1)	0.009(2)	0.001(1)

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