The crystal structure of factricarbonyl(4,4-dimethyl-2,2-dipyridyl-κ2 N,N#)- (pyrazole-κN)rhenium(I) nitrate, C18H16O3N4Re

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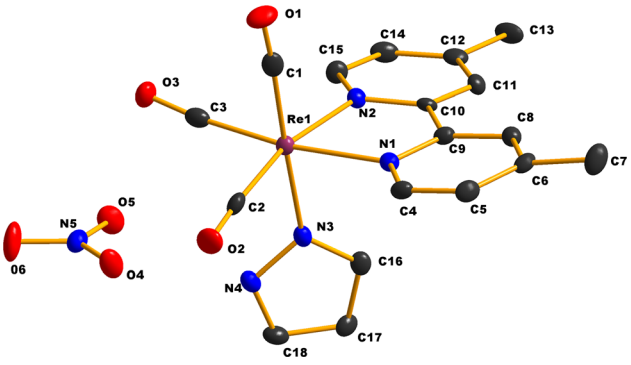
The crystal structure of *fac*-tricarbonyl(4,4-dimethyl-2,2-dipyridyl-κ^2*N*,*N*′)-(pyrazole-κ*N*)rhenium(I) nitrate, $C_{18}H_{16}O_3N_4Re$



https://doi.org/10.1515/ncrs-2020-0458
Received August 27, 2020; accepted October 12, 2020; published online November 6, 2020

Abstract

$C_{18}H_{16}O_3N_4Re$, monoclinic, $P2_1/c$ (no. 14), a = 9.8409(6) Å, b = 14.0933(9) Å, c = 13.9153(9) Å, β = 90.558(2)°, V = 1929.8(2) Å^3, Z = 4, $R_{gt}(F)$ = 0.0266, $wR_{ref}(F^2)$ = 0.0584, T = 100(2) K.

CCDC no.: 2036274

The molecular structure is shown in the Figure. Table 1 contains crystallographic data, and Table 2 contains the list of the atoms including atomic coordinates and displacement parameters.

Table 1: Data collection and handling.

Crystal:	Yellow block
Size:	0.20 × 0.17 × 0.10 mm
Wavelength:	Mo *K*α radiation (0.71073 Å)
μ:	6.35 mm^{-1}
Diffractometer, scan mode:	Bruker APEX-II, φ and ω
θ_{max}, completeness:	26.1°, >99%
$N(hkl)_{measured}$, $N(hkl)_{unique}$, R_{int}:	25,596, 3854, 0.064
Criterion for I_{obs}, $N(hkl)_{gt}$:	I_{obs} > 2 σ(I_{obs}), 3315
$N(param)_{refined}$:	273
Programs:	Bruker [1], SIR97 [2], Olex2 [3], SHELX [4], Diamond [5]

Source of material

The starting complex was synthesized according to a published procedure [14]: *fac*-[Re(Ddpy)(CO)$_3$(H$_2$O)]$^+$ (30.00 mg; 0.063 mmol) was dissolved in methanol (5 ml) and pyrazole (4.316 mg, 0.063 mmol) was added; followed by refluxing for 24 h. The bright yellow solid was obtained by evaporation of the solvent, and the product was recrystallized using cold methanol.

IR (FTIR cm^{-1}): ν_{co} = 2021.6, 1888.1, 1618.

Experimental details

All the hydrogen atoms were positioned geometrically and refined discernibly using a riding model, with fixed C-H$_{methyl}$ = 0.96 Å; C-H$_{Aromatic}$ = 0.97 Å. The H atoms isotropic displacement parameters were fixed; U_{iso}(H) = 1.2U_{eq}(C) for aromatic and U_{iso}(H) = 1.5U_{eq}(C) for methyl, allowing them to ride on the parent atom. The graphics were obtained using the DIAMOND program with 50% probability ellipsoids. All the H-atoms on the title structure were omitted for clarity.

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Table 2: Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å^2).

Atom	x	y	z	U_{iso}*/U_{eq}
Re1	0.36523 (2)	0.36667 (2)	0.15167 (2)	0.01396 (7)
O1	0.4160 (3)	0.3151 (2)	−0.0595 (2)	0.0264 (7)
O2	0.5057 (3)	0.1840 (2)	0.2198 (2)	0.0262 (7)
O3	0.6380 (3)	0.4703 (2)	0.1279 (2)	0.0243 (7)
O4	0.6958 (3)	0.3629 (2)	0.3347 (2)	0.0241 (7)
O5	0.6997 (3)	0.5165 (2)	0.3356 (2)	0.0257 (7)
O6	0.8885 (3)	0.4380 (2)	0.3395 (3)	0.0359 (9)
N1	0.1627 (3)	0.3057 (2)	0.1449 (2)	0.0137 (7)
N2	0.2300 (3)	0.4857 (2)	0.1288 (2)	0.0145 (7)
N3	0.3273 (3)	0.4026 (3)	0.3010 (2)	0.0160 (7)
N4	0.4256 (3)	0.4002 (3)	0.3699 (2)	0.0177 (8)
H4	0.511688	0.387514	0.359227	0.021*
N5	0.7630 (3)	0.4398 (3)	0.3361 (2)	0.0200 (8)
C1	0.3933 (4)	0.3347 (3)	0.0188 (3)	0.0193 (10)
C2	0.4571 (4)	0.2529 (3)	0.1925 (3)	0.0189 (9)
C3	0.5366 (4)	0.4316 (3)	0.1429 (3)	0.0186 (10)
C4	0.1367 (4)	0.2118 (3)	0.1458 (3)	0.0160 (9)
H4A	0.209268	0.169252	0.159136	0.019*
C5	0.0098 (4)	0.1753 (3)	0.1285 (3)	0.0179 (9)
H5	−0.003982	0.108613	0.129190	0.021*
C6	−0.0989 (4)	0.2360 (3)	0.1097 (3)	0.0163 (9)
C7	−0.2374 (4)	0.1983 (3)	0.0841 (3)	0.0257 (11)
H7A	−0.303757	0.250042	0.086171	0.039*
H7B	−0.262891	0.148950	0.130138	0.039*
H7C	−0.235731	0.171266	0.019210	0.039*
C8	−0.0722 (4)	0.3320 (3)	0.1119 (3)	0.0152 (9)
H8	−0.144425	0.375800	0.101995	0.018*
C9	0.0581 (4)	0.3657 (3)	0.1285 (3)	0.0148 (9)
C10	0.0936 (4)	0.4671 (3)	0.1268 (3)	0.0118 (8)
C11	−0.0010 (4)	0.5396 (3)	0.1253 (3)	0.0152 (9)
H11	−0.095228	0.525011	0.123859	0.018*
C12	0.0410 (4)	0.6336 (3)	0.1258 (3)	0.0164 (9)
C13	−0.0610 (4)	0.7129 (3)	0.1215 (3)	0.0208 (10)
H13A	−0.073396	0.733384	0.054624	0.031*
H13B	−0.028121	0.766444	0.160198	0.031*
H13C	−0.148006	0.690739	0.146723	0.031*
C14	0.1789 (5)	0.6515 (3)	0.1301 (3)	0.0197 (10)
H14	0.210879	0.715056	0.132844	0.024*
C15	0.2698 (4)	0.5775 (3)	0.1306 (3)	0.0169 (9)
H15	0.364265	0.591403	0.132226	0.020*
C16	0.2138 (4)	0.4253 (3)	0.3478 (3)	0.0165 (9)
H16	0.127329	0.432742	0.317796	0.020*
C17	0.2380 (4)	0.4367 (3)	0.4455 (3)	0.0178 (9)
H17	0.174469	0.452672	0.493854	0.021*
C18	0.3742 (4)	0.4196 (3)	0.4565 (3)	0.0191 (10)
H18	0.423711	0.421314	0.515383	0.023*

Comment

The applications of the *fac*-$[M(H_2O)_3(CO)_3]^+$ synthon, (M = Tc-99*m*, Re) and the introduction of nanotechnology merged with coordination chemistry, generally referred to as nano-medicine is a highly investigated field due to their prospective and existing applications in radiochemistry [6–9]. Nanoparticles are the major system used in nano-medicine as theranostic agents because of their high molecular specificity which permit them to absorb high quantities of drugs [10–12].

The molecular structure of the title complex comprises three facial tricarbonyl ligands, a *4,4*-dimethyl-*2,2*-dipyridyl bidentate ligand in the equatorial plane which is *trans* to two of the carbonyl ligands and a *N*-coordinated pyrazole monodentate ligand in the axial position. The complex was obtained from the [2 + 1] mixed ligand system on the *fac*-$[M(H_2O)_3(CO)_3]^+$ moiety.

The model involves the substitution of the two labile water molecules in the equatorial plane, followed by the coordination of the mono-dentate aqua ligand in the axial position; and the complex is neutralized by a nitrate counter-ion. The polyhedron geometry of the title complex reveals an octahedral distortion around rhenium(I). The distortion is evident in the angles of 167.56(14)° and 171.99(14)° for C2–Re1–N2 and C3–Re1–N1 respectively. The bite angle of the chelate ligand of 74.76(12)° from the title structure correlates well with those observed in literature reported similar structures [13–14]. The bond distances between rhenium and the coordinated N atoms in the first coordination sphere ranges between 2.162(3) Å and 2.174(3) Å whereas the bond distances between the rhenium and the carbonyl carbon atoms ranges between 1.923(5) Å and 1.926(5) Å.

Author contribution: All the authors have accepted responsibility for the entire content of this submitted manuscript and approved submission.

Research funding: The authors would like to express the gratitude towards NRF (Thuthuka grant specific number 113629) for financial assistance. We would also like to acknowledge Tshwane University of Technology for Institutional support.

Conflict of interest: The authors declare no conflicts of interest regarding this article.

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