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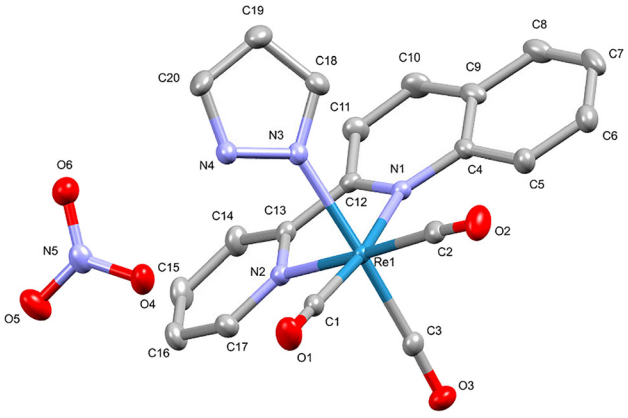
The crystal structure of *fac*-tricarbonyl(2-pyridin-2-yl-quinoline-κ^2*N*,*N*′)-(pyrazole-κ*N*)rhenium(I) nitrate, $C_{20}H_{14}N_4O_3ReNO_3$



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Abstract

$C_{20}H_{14}N_4O_3ReNO_3$, monoclinic, $P2_1/c$ (no. 14), a = 12.9572 (2), b = 9.1568 (2), c = 17.3658 (3) Å, β = 97.267 (2)°, V = 2043.84 (7) Å^3, Z = 4, $R_{gt}(F)$ = 0.0231, $wR_{ref}(F^2)$ = 0.0514, T = 154 K

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Table 1 contains crystallographic data and Table 2 contains the list of the atoms including atomic coordinates and displacement parameters.

1 Source of materials

fac-[Re(*N*,*N*′)(CO)$_3$(pyrazole)] (*N*,*N*′ = 2-pyridin-2-yl-quinoline) was prepared according to previously reported procedures, using the [2 + 1] mixed ligand approach [15, 16].

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Table 1: Data collection and handling.

Crystal:	Yellow blade
Size:	0.30 × 0.19 × 0.08 mm
Wavelength:	Mo *Kα* radiation (0.71073 Å)
μ:	6.00 mm^{-1}
Diffractometer, scan mode:	XtaLAB Synergy R, ω
θ_{max}, completeness:	31.0°, >99 %
$N(hkl)_{measured}$, $N(hkl)_{unique}$, R_{int}:	33,676, 5471, 0.057
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\sigma(I_{obs})$, 4763
$N(param)_{refined}$:	289
Programs:	CrysAlis Pro [1], Olex2 [2], WinGX [3], Shelx [4, 5]

fac-[Re(*N*,*N*′)(CO)$_3$(Cl)] (40 mg, 0.078 mmol) was dissolved in methanol (3 mL), and pyrazole (5.50 mg, 0.081 mmol) dissolved in (2 mL) methanol was added. The solution was then refluxed for 24 h at 60 °C and a yellow solution was formed. The solvent was evaporated and a yellow solid formed, which was recrystallized in dichloromethane. (Yield = 32.20 mg, 70.77 %), **IR (FTIR cm^{-1})**: ν_{co} = 2016.3, 1881.

2 Experimental details

All hydrogen atoms were positioned geometrically and refined using riding models, with fixed C–H$_{Aromatic}$ = 0.95 Å. The H atoms isotropic displacement parameters were fixed; U_{iso}(H) = 1.2U_{eq} (C), allowing them to ride on the parent atom. The graphics were obtained using the MERCURY program with 50 % probability ellipsoids. All the H-atoms on the title structure were omitted for clarity.

3 Comment

This work forms part of ongoing research that attempts to design Re(I) tricarbonyl complexes for medicinal purposes [6–8]. Recently researchers have embarked on designing various Re(I) complexes aimed at treating diseases like Chagas disease [9], Malaria [10], and lung and breast cancer [11, 12], just to name a few. These Re(I) complexes were found to be active against all these diseases, therefore research must continue using this metal core kinetic behavior as well as medical applications.

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

Atom	*x*	*y*	*z*	U_{iso}*/U_{eq}
C1	0.7644 (2)	−0.0159 (3)	0.39006 (16)	0.0209 (5)
C2	0.5794 (2)	0.0668 (3)	0.33831 (16)	0.0204 (5)
C3	0.6361 (2)	0.0637 (3)	0.49450 (16)	0.0194 (5)
C4	0.52059 (19)	0.4247 (3)	0.39775 (15)	0.0178 (5)
C5	0.4399 (2)	0.3257 (3)	0.37236 (18)	0.0239 (6)
H5	0.452647	0.223606	0.376026	0.029*
C6	0.3432 (2)	0.3754 (3)	0.34245 (18)	0.0279 (6)
H6	0.289954	0.307100	0.325583	0.034*
C7	0.3217 (2)	0.5257 (3)	0.33633 (17)	0.0294 (6)
H7	0.255203	0.558552	0.313691	0.035*
C8	0.3966 (2)	0.6238 (3)	0.36300 (17)	0.0269 (6)
H8	0.381192	0.725178	0.360106	0.032*
C9	0.4972 (2)	0.5774 (3)	0.39498 (15)	0.0216 (6)
C10	0.5752 (2)	0.6753 (3)	0.42536 (18)	0.0261 (6)
H10	0.562215	0.777406	0.422933	0.031*
C11	0.6695 (2)	0.6248 (3)	0.45833 (17)	0.0239 (6)
H11	0.721321	0.690662	0.480938	0.029*
C12	0.68892 (19)	0.4729 (3)	0.45837 (15)	0.0171 (5)
C13	0.78958 (19)	0.4155 (3)	0.49553 (15)	0.0174 (5)
C14	0.8607 (2)	0.4984 (3)	0.54343 (17)	0.0258 (6)
H14	0.848679	0.599565	0.550604	0.031*
C15	0.9493 (2)	0.4324 (3)	0.58066 (18)	0.0290 (6)
H15	0.998261	0.487701	0.614011	0.035*
C16	0.9660 (2)	0.2858 (3)	0.56911 (17)	0.0268 (6)
H16	1.026870	0.239267	0.593828	0.032*
C17	0.8924 (2)	0.2074 (3)	0.52073 (16)	0.0218 (5)
H17	0.903429	0.106039	0.513297	0.026*
C18	0.7106 (2)	0.3830 (3)	0.26962 (16)	0.0238 (6)
H18	0.641023	0.416735	0.267870	0.029*
C19	0.7818 (2)	0.4342 (3)	0.22258 (17)	0.0298 (7)
H19	0.770989	0.507325	0.183606	0.036*
C20	0.8712 (2)	0.3563 (3)	0.24450 (18)	0.0271 (6)
H20	0.934878	0.365331	0.223178	0.033*
N1	0.61884 (16)	0.3757 (2)	0.42703 (12)	0.0158 (4)
N2	0.80612 (16)	0.2701 (2)	0.48409 (12)	0.0170 (4)
N3	0.75349 (16)	0.2801 (2)	0.31747 (12)	0.0169 (4)
N4	0.85224 (17)	0.2651 (2)	0.30150 (13)	0.0208 (5)
H4	0.897967	0.203845	0.325261	0.025*
N5	1.07929 (19)	0.1526 (2)	0.38341 (15)	0.0231 (5)
O1	0.80534 (16)	−0.1221 (2)	0.37628 (13)	0.0307 (5)
O2	0.51666 (16)	0.0040 (2)	0.29833 (12)	0.0333 (5)
O3	0.60773 (15)	0.0082 (2)	0.54741 (12)	0.0281 (4)
O4	0.99778 (15)	0.0773 (2)	0.38182 (13)	0.0321 (5)
O5	1.15321 (18)	0.1328 (2)	0.43534 (15)	0.0363 (5)
O6	1.08544 (16)	0.2463 (2)	0.33183 (13)	0.0327 (5)
Re1	0.68730 (2)	0.15786 (2)	0.40810 (2)	0.01422 (4)

In this study, the crystal structure of the presented complex consists of three facial tricarbonyl ligands, with 2-pyridin-2-yl-quinoline bidentate ligand in the equatorial plane which is *trans* to two of the carbonyl ligands, and an axial position *N*-coordinated pyrazole monodentate ligand. The complex was synthesized by utilizing the [2 + 1] mixed ligand approach from the *fac*-$[Re(H_2O)_3(CO)_3]^+$ moiety. The complex is neutralized by a nitrate $[NO_3^-]$ counter-ion after the substitution of two labile water molecules in the equatorial plane. The model of the crystal structure was found to possess an octahedral distortion, as seen in the angles 171.88 (9)° for C1–Re1–N1 and 177.24 (9)° for C2–Re1–N2, which diverge significantly from 180°. The bite angle of 75.48 (8)° from the title structure correlates well with those observed in the literature that reported similar structures [13, 14]. The bond distances between rhenium and the nitrogen atoms directly attached to the rhenium sphere range between 2.157(2) Å and 2.224(2) Å whereas the bond distances between the rhenium and the carbonyl carbon atoms range between 1.920(3) Å and 1.925(3) Å. The title complexes are arranged in a head-to-head manner and are stabilized by couple of intra- and intermolecular hydrogen bonds.

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