The crystal structure of \textit{fac-tricarbonyl(bis(3,5-dimethyl-4H-pyrazole)-κ^1N)-((nitrato)-κ^1O)-rhenium(I)— 3,5-dimethyl-4H-pyrazole(1/1), C}_{18}H_{23}N_{7}O_{6}Re}

Abstract

\[ \text{C}_{18}H_{23}N_{7}O_{6}Re, \text{ triclinic, } P \bar{1} \text{ (no. 2), } a = 10.527(2) \text{ Å, } b = 10.849(2) \text{ Å, } c = 11.215(2) \text{ Å, } \alpha = 72.798(2) \text{°, } \beta = 69.767(2) \text{°, } \gamma = 87.496(10) \text{°, } V = 1145.52(4) \text{ Å}^3, Z = 2, R_{gt} = 0.0273, wR_{ref} = 0.0727, T = 150(2) K.} \]

Table 1: Data collection and handling.

| Crystal: | Colorless needles |
| Size: | 0.29 × 0.25 × 0.19 mm |
| Wavelength: | MoK\(\alpha\) radiation (0.71073 Å) |
| \(\mu\): | 5.35 mm\(^{-1}\) |
| Diffractometer, scan mode: | Rigaku XtaLAB Synergy R, \(\omega\) |
| \(\theta_{\text{max}}\) completeness: | 26.4°, >99% |
| \(N(hkl)\text{measured} - N(hkl)\text{unique}\) \(\%\): | 37,457, 4680, 0.088 |
| Criterion for \(I_{\text{obs}}\): \(I_{\text{obs}} > 2\sigma(I_{\text{obs}})\): | 4447, 295 |
| Programs: | Olex2 \(^1\), Sheldx \(^2\) |

CCDC no.: 2354778

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.

1 Source of material

The title compound was crystallized from the intermediate \textit{fac-Re(\(\text{CO}_3\text{(OCH}_3\text{)}_3\))N\text{O}_3 in the presence of excess pyrazole ligands during the reaction indicated in the literature.}\(^5\) The title compound was crystallized from the methanol filtrate in high purity. IR (ATR, cm\(^{-1}\)): \(\nu_{\text{CO}}\text{) 2020, 1892, 1879.}\)

2 Experimental details

All hydrogen atoms were positioned geometrically using a riding model, with fixed C–H aromatic = 0.97 Å. The H atoms isotropic displacement parameters were fixed; \(U_{\text{iso}}(H) = 1.2U_{\text{eq}}(C)\) for aromatic, 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

The radionuclide \(^{99m}\text{Tc}\) is the most significant radioisotope in radiopharmaceuticals mainly for imaging purposes.\(^5\) This isotope has first to be stabilized by chelator ligands...
The crystal structure exhibits an octahedral distortion for ruthenium(II)-based complexes as indicated by the N1–Re1–N3 bond angle of 86.45(12)° and O4–Re1–N1 bond angle of 78.15(11)°. The bond distance between the rhenium (Re1) and the oxygen (O4) of the nitrate moiety is 2.170(3) Å which is in accordance with literature related structures.14,15 This title compound is further stabilized by N–H⋯O, –N⋯N, C–H⋯O, and C–H⋯N intra-intermolecular hydrogen contacts. All bond distances and angles of the presented complex correlate well with other structures already reported in the literature.14–17

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

Research funding: National Research Foundation of South Africa (Grant No. 129468 and TTK2204193773), Tshwane University of Technology, University of the Western Cape and the University of Pretoria.

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


