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Tricarbonyl[tris(pyrazol-1-yl)methanesulfonato-κ^3N,N',N'']rhenium(I) acetone solvate

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Key indicators

Single-crystal X-ray study
T = 183 K
Mean σ(C–C) = 0.010 Å
R factor = 0.035
wR factor = 0.086
Data-to-parameter ratio = 21.0

For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

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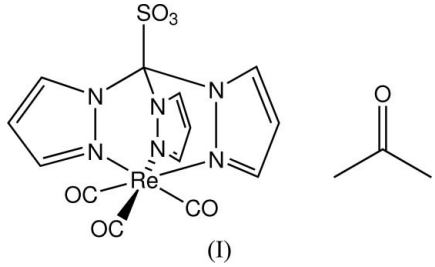
The title compound, $[Re(C_{10}H_9N_6O_3S)(CO)_3]\cdot C_3H_6O$, was prepared in almost quantitative yield by reaction of $Re(CO)_5Br$ with the thallium salt of the tris(pyrazol-yl)methanesulfonate (tpms) ligand. It shows a slightly distorted octahedral geometry for the *fac*-$Re(CO)_3N_3$ unit, with the ligand forming three six-membered N-bonded chelate rings. A C—H···O interaction between the main molecule and the solvent molecule helps to establish the crystal packing.

Comment

Tris(pyrazolyl)methane (tpm) type ligands are easily modified at the central methane C atom by deprotonation and reaction of the resulting carbanion with electrophiles (Reger & Grattan, 2003). Furthermore, it is known that tris(pyrazol-yl)methane type ligands readily coordinate to group VII metal(I) carbonyls (Reger *et al.*, 2002). Therefore derivatives of tpm type ligands should be suitable as anchor groups for ^{99m}Tc or ^{188}Re binding to biomolecules or biologically active macromolecules. The tris(pyrazolyl)methanesulfonato (tpms) ligand is a well studied tpm derivative and was designed to be a water-soluble N,N',N''-ligand (Kläui *et al.*, 2003).



(I)

We report here the synthesis and structural characterization of the title compound, (I) (Fig. 1). During our work, the unsolvated complex $[(tpms)Re(CO)_3]$ has been described elsewhere (Herrick *et al.*, 2006). Compound (I) was prepared in nearly quantitative yield by reaction of $Re(CO)_5Br$ with Tl(tpms) under refluxing THF conditions.

The distorted octahedral ReC_3N_3 coordination environment of the rhenium centre (Table 1) is defined by three facially bound CO ligands and the three pyrazolyl nitrogen donor atoms, *i.e.* the tpms acts as a κ^3-N,N',N''-tridentate ligand. This coordination mode is also found in the copper(I) carbonyl complex of tpms^{tBu} but in solution exists in equilibrium with κ^3-N,N',O-coordination, with the sulfonato group coordinating to the metal centre (Kläui *et al.*, 2003). Such κ^3-N,N',O modes are observed in some tpms–3*d*-transition metal complexes, resembling the $M(CO)_3$ (M = Mn, Re) complexes of bis(pyrazol-1-yl)acetate ligands (Burzlaff *et al.*, 2001).

A weak non-classical intermolecular C—H···O hydrogen bond is formed between C19/H19 of one pyrazolyl ring and O20 of the cocrystallized acetone molecule [C19···O20 = 3.35 (1) Å].

Experimental

The thallium salt of the tpms ligand was prepared according to the literature procedure (Kläui *et al.*, 2000). For the preparation of (I), Tl(tpms) (0.25 mmol, 0.13 g) and $Re(CO)_5Br$ (0.25 mmol, 0.10 g) were dissolved in THF (10 ml) and the resulting solution refluxed for 4 h. The precipitated TlBr was removed by filtration and the colourless reaction mixture was evaporated to dryness. The solid thus obtained was crystallized by slow evaporation of an acetone solution to yield colourless plates of (I). IR (KBr): $\nu_{(CO)}$/ cm^{-1} = 2037, 1916. HPLC was performed on a Merck L7000 system using a Macherey–Nagel EC 2503 Nucleosil 100-5 C18HD column. HPLC: t min^{-1} = 19.9. HPLC solvents were 0.1% trifluoroacetic acid as solvent *A* and methanol as solvent *B*. The flow-rate was 0.5 ml min^{-1}. Detection was performed at 250 nm. The gradient used for analysis was: 0–3 min 100% *A*, 3.1–9 min. 75% *A*, 9.1–20 min. 66% to 0% *A* 20–25 min 0% *A*, 25.1–30 min. 100 % *A*.

Crystal data

$[Re(C_{10}H_9N_6O_3S)(CO)_3]\cdot C_3H_6O$
M_r = 621.60
Triclinic, $P\overline{1}$
a = 8.9610 (6) Å
b = 8.9726 (6) Å
c = 14.3248 (11) Å
α = 80.455 (9)°
β = 84.444 (9)°
γ = 70.235 (8)°
V = 1067.87 (13) Å^3
Z = 2
D_x = 1.933 Mg m^{-3}
Mo $K\alpha$ radiation
μ = 5.84 mm^{-1}
T = 183 (2) K
Thick plate, colourless
0.24 × 0.22 × 0.13 mm

Data collection

Stoe IPDS diffractometer
φ oscillation scans
Absorption correction: numerical (*X-RED*; Stoe & Cie, 1999)
T_{min} = 0.250, T_{max} = 0.493
12823 measured reflections
5872 independent reflections
4848 reflections with $I > 2\sigma(I)$
R_{int} = 0.046
θ_{max} = 30.4°

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.035
$wR(F^2)$ = 0.086
S = 0.92
5872 reflections
280 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0551P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.004
$\Delta\rho_{max}$ = 1.56 e Å^{-3}
$\Delta\rho_{min}$ = −1.41 e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

Re1—C1	1.933 (5)	Re1—N10	2.165 (4)
Re1—C2	1.935 (5)	Re1—N12	2.165 (4)
Re1—C3	1.937 (5)	Re1—N14	2.179 (4)
N10—Re1—N12	81.47 (15)	N12—Re1—N14	80.39 (14)
N10—Re1—N14	79.64 (14)		

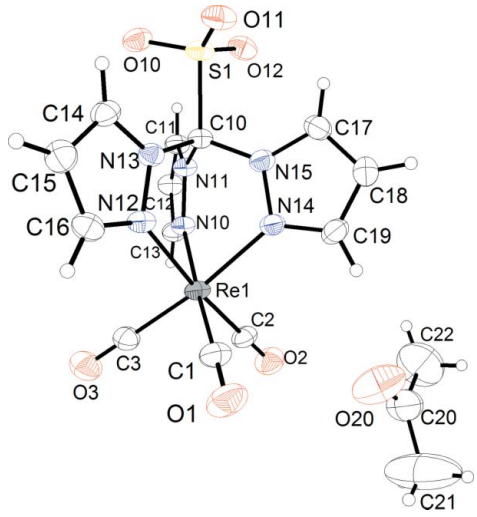


Figure 1
View of the molecular structure of (I), showing 50% probability displacement ellipsoids (arbitrary spheres for H atoms).

The low data completeness (90.8%) is caused by the symmetry of the triclinic space group and the use of a one-circle IPDS diffractometer. The H atoms were placed geometrically (C—H = 0.95–0.98 Å) and refined as riding with U_{iso}(H) = 1.2U_{eq}(C). The highest residual density peak is located 1.12 Å away from N12, the deepest hole 0.77 Å from Re1.

Data collection: *IPDS Software* (Stoe & Cie, 1999); cell refinement: *IPDS Software*; data reduction: *X-RED* (Stoe & Cie, 1999); program(s) used to solve structure: *SIR97* (Altomare *et al.*, 1999); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *ORTEP-3* (Farrugia, 1997); software used to prepare material for publication: *SHELXL97*.

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