Acta Crystallographica Section E
Structure Reports
Online
ISSN 1600-5368

[*N*,*N*′-Bis(2,4-dimethylbenzene)-2,3-dimethyl-1,4-diazabuta-1,3-diene]-*fac*-tricarbonyl-(trifluoroacetato)rhenium(I)

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Received 7 February 2005
Accepted 14 February 2005
Online 19 February 2005

In the title compound, $[Re(CF_3COO)(C_{20}H_{24}N_2)(CO)_3]$, the Re atom has a distorted octahedral configuration. The 1,4-diazabutadiene part of the donor ligand and two carbonyl groups occupy the equatorial plane of the complex, with the third carbonyl ligand and the trifluoroacetate group in the axial positions. The equatorial diimine ligand is bound to the Re center in an *N*,*N*′-bidentate chelating fashion, leading to a five-membered ReN_2C_2 chelate ring with an N—Re—N bite angle of 73.4 (2)°.

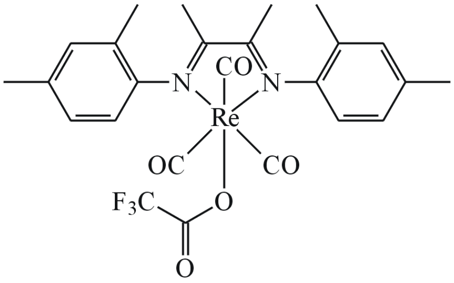
Key indicators

Single-crystal X-ray study
T = 293 K
Mean σ(C–C) = 0.013 Å
Disorder in main residue
R factor = 0.042
wR factor = 0.087
Data-to-parameter ratio = 16.0

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

Comment

We report here the results of an X-ray structure investigation of the title complex, (I). The Re atom has a distorted octahedral configuration (Fig. 1) and is coordinated by two carbonyl groups [Re—C = 1.900 (9) and 1.901 (9) Å] and the diimine group of the organic ligand [Re—N = 2.156 (5) and 1.157 (5) Å] in the equatorial plane. Octahedral coordination is completed at the axial positions by another carbonyl ligand [Re—C = 1.912 (8) Å] and a carboxyl O atom of the trifluoroacetate anion [Re—O = 2.157 (4) Å]. The orientation of the coordinated trifluoroacetate anion can be defined by the O2—C8—C9—F1 torsion angle of −16 (2)°. The equatorial arrangement, defined by the Re and donor atoms (N1/N4/C5/C6) of the corresponding diimine and carbonyl ligands is essentially planar; the deviation of the Re atom from the N1/N4/C5/C6 plane is 0.068 (3) Å.



(I)

The equatorial diimine ligand is bound to the Re center in a *N*,*N*′-bidentate chelating fashion, leading to a five-membered ReN_2C_2 chelate ring with an N1—Re1—N4 bite angle of 73.4 (2)°. The other *cis*-equatorial angles at Re1 are C5—Re1—C6 of 88.3 (3)°, N1—Re1—C5 of 98.9 (3)° and N4—Re1—C6 of 97.0 (2)°. The axial C7—Re1—O1 angle is 177.4 (3)°. The aromatic rings are oriented essentially at right angles to the chelate ring plane [dihedral angle between C11–C16 and Re1/N1/C2 is 84.9 (2)° and between C21–C26 and Re1/N4/C3 is 87.4 (3)°]; this orientation leads to C121···O7

doi:10.1107/S1600536805005003

and C221···O7 intramolecular separations of 3.737 (11) and 3.738 (18) Å, respectively. The CF_3 group has high thermal motion consistent with some unresolved rotational disorder, as is often found in other trifluoroacetate compounds (Rodrigues *et al.*, 2000, 2001).

Experimental

Compound (I) was synthesized as described in the literature (Klein *et al.*, 1996). Dark red block-shaped crystals of (I) suitable for X-ray structure analysis were obtained by slow evaporation of a solution in dichloromethane/*n*-hexane at room temperature.

Crystal data

$[Re(C_2F_3O_2)(C_{20}H_{24}N_2)(CO)_3]$
$M_r = 675.66$
Monoclinic, $P2_1/c$
$a = 12.818$ (3) Å
$b = 13.026$ (3) Å
$c = 16.554$ (4) Å
$\beta = 106.07$ (2)°
$V = 2655.9$ (11) Å^3
$Z = 4$
$D_x = 1.690$ Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 25 reflections
$\theta = 14.0$–$15.9°$
$\mu = 4.63$ mm^{-1}
$T = 293$ (2) K
Block, dark red
0.55 × 0.30 × 0.30 mm

Data collection

Enraf–Nonius CAD-4 diffractometer
ω–2θ scans
Absorption correction: ψ scan (*DATCOR*; Reibenspies, 1989)
$T_{min} = 0.137$, $T_{max} = 0.250$
5775 measured reflections
5369 independent reflections
2977 reflections with $I > 2\sigma(I)$
$R_{int} = 0.015$
$\theta_{max} = 26.3°$
$h = 0 \rightarrow 15$
$k = 0 \rightarrow 16$
$l = -20 \rightarrow 19$
3 standard reflections frequency: 60 min
intensity decay: 3%

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.042$
$wR(F^2) = 0.087$
$S = 0.92$
5369 reflections
335 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0422P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.004$
$\Delta\rho_{max} = 0.76$ e Å^{-3}
$\Delta\rho_{min} = -0.63$ e Å^{-3}

One of the *m*-xylol rings of the diimine ligand has orientational disorder and the refined site-occupancy factors of the disordered parts (methyl groups C221/C226 and associated H atoms) are 0.651 (15) and 0.349 (15). None of the the H atoms of the methyl groups was clearly resolved in difference maps and they were subsequently included as six half-occupancy H atoms on each group. Other H atoms were located in difference maps and were subsequently repositioned geometrically and refined as riding, with C—H distances of 0.93 (aromatic) and 0.96 Å (methyl), and with $U_{iso}(H) = 1.2$ (aromatic) or 1.5 (methyl) times $U_{eq}(C)$.

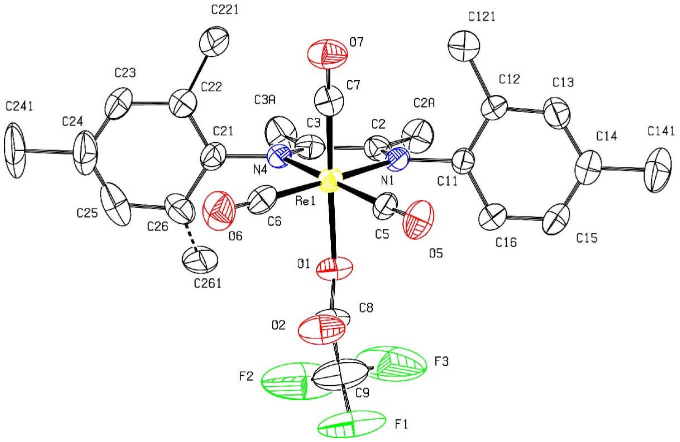


Figure 1
The molecular structure of (I). Displacement ellipsoids are drawn at the 30% probability level. Atom C261 is the minor-occupancy methyl C atom. For clarity, H atoms have been omitted.

Data collection: *CAD-4 EXPRESS* (Enraf–Nonius, 1992); cell refinement: *CAD-4 EXPRESS*; data reduction: *XCAD4* (Harms, 1996); program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *PLATON* (Spek, 2003); software used to prepare material for publication: *SHELXL97*.

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