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fac-Bis(acetonitrile- κN)tricarbonyl-(trifluoroacetato- κO)rhenium(I)

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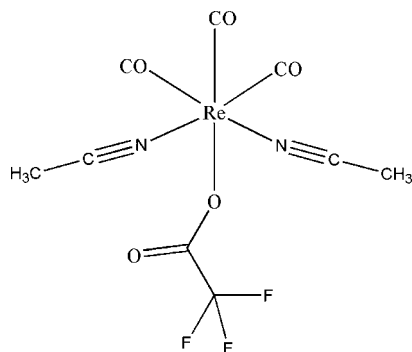
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Key indicators: single-crystal X-ray study; $T = 100$ K; mean $\sigma(C-C) = 0.007$ Å; disorder in main residue; R factor = 0.032; wR factor = 0.079; data-to-parameter ratio = 27.6.

In the title compound, $[Re(CF_3COO)(CH_3CN)_2(CO)_3]$, the Re atom has a distorted octahedral configuration. The two acetonitrile molecules and two of the three carbonyl groups occupy the equatorial plane of the complex, with the third carbonyl ligand and the trifluoroacetato ligand in the axial positions. The three carbonyl ligands are arranged in a *fac* configuration around the Re atom. The CF_3 segment of the trifluoroacetato ligand shows rotational disorder and the refined site-occupancy factors of the disordered parts are *ca* 0.5/0.5. The crystal structure is stabilized by $C-H \cdots O$ and $C-H \cdots F$ hydrogen bonds.

Related literature

For values of standard bond lengths, see: Allen *et al.* (1987). For related structures, see, for example: Chan *et al.* (1977); Lazarova *et al.* (2004). For background on the applications, see, for example: Davies & Hartely (1981); Collin & Sauvage (1989); Balzani *et al.* (1996); Meyer (1989).



Experimental

Crystal data

$[Re(C_2F_3O_2)(C_2H_3N)_2(CO)_3]$
 $M_r = 465.36$
 Monoclinic, $P2_1/c$
 $a = 10.8243$ (2) Å
 $b = 10.4745$ (2) Å
 $c = 14.4772$ (3) Å
 $\beta = 125.584$ (1)°

$V = 1334.90$ (4) Å³
 $Z = 4$
 Mo $K\alpha$ radiation
 $\mu = 9.16$ mm⁻¹
 $T = 100.1$ (1) K
 $0.32 \times 0.23 \times 0.19$ mm

Data collection

Bruker SMART APEXII CCD
 area-detector diffractometer
 Absorption correction: multi-scan
 (SADABS; Bruker, 2005)
 $T_{min} = 0.098$, $T_{max} = 0.175$

24096 measured reflections
 5819 independent reflections
 5087 reflections with $I > 2\sigma(I)$
 $R_{int} = 0.037$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.031$
 $wR(F^2) = 0.078$
 $S = 1.06$
 5819 reflections

211 parameters
 H-atom parameters constrained
 $\Delta\rho_{max} = 2.80$ e Å⁻³
 $\Delta\rho_{min} = -1.27$ e Å⁻³

Table 1

Hydrogen-bond geometry (Å, °).

$D-H \cdots A$	$D-H$	$H \cdots A$	$D \cdots A$	$D-H \cdots A$
$C4-H4B \cdots O5^i$	0.98	2.42	3.113 (5)	127
$C6-H6A \cdots F1A^{ii}$	0.98	2.35	3.237 (8)	150
$C6-H6B \cdots O5^{iii}$	0.98	2.46	3.075 (5)	121

Symmetry codes: (i) $-x + 1, y - \frac{1}{2}, -z + \frac{3}{2}$; (ii) $-x + 2, y - \frac{1}{2}, -z + \frac{5}{2}$; (iii) $-x + 2, -y, -z + 2$.

Data collection: APEX2 (Bruker, 2005); cell refinement: APEX2; data reduction: SAINT (Bruker, 2005); program(s) used to solve structure: SHELXTL (Sheldrick, 2008); program(s) used to refine structure: SHELXTL; molecular graphics: SHELXTL; software used to prepare material for publication: SHELXTL and PLATON (Spek, 2003).

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: AT2632).

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supplementary materials

Acta Cryst. (2008). E64, m1314 [doi:10.1107/S1600536808029966]

***fac*-Bis(acetonitrile- κ N)tricarbonyl(trifluoroacetato- κ O)rhenium(I)**

R. Kia and H.-K. Fun

Comment

The synthesis of solvent-coordinated complexes is a matter of considerable interest since they are useful sources for synthesis of new species resulting from the substitution of the coordinated solvent by a more basic ligand (Davies & Hartely, 1981). The lability of the solvent ligand easily gives rise to a highly reactive $16e^-$ electrophilic fragment able to activate small molecules thus providing an important step in many chemical processes. On the other hand, Rhenium tricarbonyl complexes have been the subject of much attention, mainly because of their photophysical (Meyer 1989) and photochemical properties (Collin & Sauvage 1989) and in supramolecular chemistry (Balzani *et al.*, 1996).

In the title compound (I) (Fig. 1), the Re atom adopts a distorted octahedral geometry. The bond lengths (Allen *et al.*, 1987) and angles are within the normal ranges and are comparable to the related structures (Chan *et al.*, 1977; Lazarova *et al.*, 2004). The two acetonitriles and two carbonyl groups occupy the equatorial plane of the complex, with the third carbonyl ligands and the trifluoroacetate in the axial positions. The three carbonyl ligands at Re atom are arranged in a *fac* configuration. The *cis*-equatorial angle of N1–Re1–N2 is $81.88(13)^\circ$. The deviation of the Re atom from the C1/C2/N1/N2 plane is $-0.048(1) \text{ \AA}$.

The crystal structure is stabilized by C—H \cdots O and C—H \cdots F hydrogen bonds (Table 1, Fig. 2).

Experimental

The synthetic method has been described earlier (Chan *et al.*, 1977). Single crystals suitable for X-ray diffraction were obtained by evaporation of an acetonitrile solution at room temperature.

Refinement

The hydrogen atoms of the methyl groups were first located from the difference Fourier map and then constrained to refine using a rotating-group model. The CF₃ segment of the trifluoroacetate ligand has rotational disorder and the refined site-occupancy factors of the disorder parts are 0.501(2)/0.499(2). The highest peak (2.80 e\AA^{-3}) is located 0.61 \AA from Re1 and the deepest hole (-1.27 e\AA^{-3}) is located 1.01 \AA from Re1.

Figures

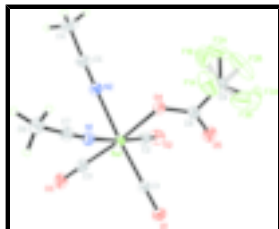


Fig. 1. The molecular structure of the title compound, showing 40% probability displacement ellipsoids and the atomic numbering scheme. The open bonds indicate the disordered parts.

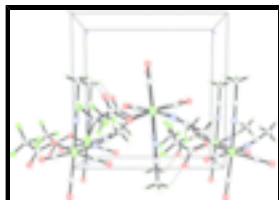


Fig. 2. The crystal packing of the major component of (I), viewed down the *c*-axis. Inter-molecular interactions are shown as dashed lines.

fac-Bis(acetonitrile- κ N)tricarbonyl(trifluoroacetato- κ O)rhenium(I)

Crystal data

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Monoclinic, $P2_1/c$

Hall symbol: -P 2ybc

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$b = 10.4745$ (2) Å

$c = 14.4772$ (3) Å

$\beta = 125.5840$ (10)°

$V = 1334.90$ (4) Å³

$Z = 4$

$F_{000} = 864$

$D_x = 2.316$ Mg m⁻³

Mo $K\alpha$ radiation

$\lambda = 0.71073$ Å

Cell parameters from 9896 reflections

$\theta = 2.6$ – 40.1 °

$\mu = 9.16$ mm⁻¹

$T = 100.1$ (1) K

Block, colourless

$0.32 \times 0.23 \times 0.19$ mm

Data collection

Bruker SMART APEXII CCD area-detector diffractometer

Radiation source: fine-focus sealed tube

Monochromator: graphite

$T = 100.0$ (1) K

φ and ω scans

Absorption correction: multi-scan (SADABS; Bruker, 2005)

$T_{\min} = 0.098$, $T_{\max} = 0.175$

24096 measured reflections

5819 independent reflections

5087 reflections with $I > 2\sigma(I)$

$R_{\text{int}} = 0.037$

$\theta_{\text{max}} = 35.0$ °

$\theta_{\text{min}} = 2.6$ °

$h = -17 \rightarrow 14$

$k = -10 \rightarrow 16$

$l = -23 \rightarrow 23$

Refinement

Refinement on F^2

Secondary atom site location: difference Fourier map

Least-squares matrix: full

$$R[F^2 > 2\sigma(F^2)] = 0.031$$

$$wR(F^2) = 0.078$$

$$S = 1.06$$

5819 reflections

211 parameters

Primary atom site location: structure-invariant direct methods

Hydrogen site location: inferred from neighbouring sites

H-atom parameters constrained

$$w = 1/[\sigma^2(F_o^2) + (0.0375P)^2 + 1.8212P]$$

$$\text{where } P = (F_o^2 + 2F_c^2)/3$$

$$(\Delta/\sigma)_{\max} = 0.002$$

$$\Delta\rho_{\max} = 2.80 \text{ e } \text{\AA}^{-3}$$

$$\Delta\rho_{\min} = -1.27 \text{ e } \text{\AA}^{-3}$$

Extinction correction: none

Special details

Experimental. The low-temperature data was collected with the Oxford Cyrosystem Cobra low-temperature attachment.

Geometry. All esds (except the esd in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell esds are taken into account individually in the estimation of esds in distances, angles and torsion angles; correlations between esds in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell esds is used for estimating esds involving l.s. planes.

Refinement. Refinement of F^2 against ALL reflections. The weighted R-factor wR and goodness of fit S are based on F^2 , conventional R-factors R are based on F , with F set to zero for negative F^2 . The threshold expression of $F^2 > 2\sigma(F^2)$ is used only for calculating R-factors(gt) etc. and is not relevant to the choice of reflections for refinement. R-factors based on F^2 are statistically about twice as large as those based on F , and R- factors based on ALL data will be even larger.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (\AA^2)

	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{iso}}^*/U_{\text{eq}}$	Occ. (<1)
Re1	0.681282 (13)	-0.021815 (12)	0.800806 (9)	0.02552 (4)	
F1A	0.9350 (14)	0.2534 (7)	1.1194 (5)	0.073 (4)	0.501 (19)
F2A	0.7397 (8)	0.3650 (13)	1.0179 (9)	0.075 (4)	0.501 (19)
F3A	0.938 (2)	0.4097 (12)	1.0310 (12)	0.101 (6)	0.501 (19)
F1B	0.818 (2)	0.2830 (10)	1.0906 (11)	0.096 (7)	0.499 (19)
F2B	0.7687 (18)	0.4304 (11)	0.9808 (9)	0.104 (6)	0.499 (19)
F3B	0.9837 (11)	0.358 (2)	1.0782 (13)	0.121 (8)	0.499 (19)
O1	0.9541 (3)	0.0126 (3)	0.7902 (3)	0.0368 (5)	
O2	0.5020 (4)	0.1538 (3)	0.5934 (2)	0.0477 (7)	
O3	0.5809 (4)	-0.2505 (3)	0.6407 (3)	0.0461 (6)	
O4	0.7533 (3)	0.1237 (2)	0.9276 (2)	0.0308 (4)	
O5	0.8149 (4)	0.2725 (3)	0.8489 (2)	0.0425 (6)	
N1	0.7910 (3)	-0.1433 (3)	0.9467 (2)	0.0307 (5)	
N2	0.4997 (4)	-0.0360 (3)	0.8190 (3)	0.0338 (6)	
C1	0.8537 (4)	-0.0010 (3)	0.7956 (3)	0.0284 (5)	
C2	0.5700 (4)	0.0891 (4)	0.6720 (3)	0.0353 (7)	
C3	0.6165 (4)	-0.1638 (4)	0.7004 (3)	0.0339 (6)	
C4	0.2941 (6)	-0.0491 (6)	0.8572 (5)	0.0620 (15)	
H4A	0.3436	-0.0624	0.9386	0.093*	
H4B	0.2247	-0.1201	0.8144	0.093*	
H4C	0.2368	0.0311	0.8336	0.093*	

supplementary materials

C5	0.4088 (5)	-0.0429 (4)	0.8350 (3)	0.0401 (8)
C6	0.8744 (5)	-0.3017 (4)	1.1107 (3)	0.0398 (7)
H6A	0.9143	-0.2542	1.1811	0.060*
H6B	0.9531	-0.3591	1.1211	0.060*
H6C	0.7859	-0.3517	1.0915	0.060*
C7	0.8301 (4)	-0.2132 (3)	1.0199 (3)	0.0327 (6)
C8	0.8016 (4)	0.2303 (3)	0.9217 (3)	0.0322 (6)
C9	0.8499 (5)	0.3206 (4)	1.0215 (3)	0.0405 (7)

Atomic displacement parameters (\AA^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Re1	0.02296 (6)	0.02704 (7)	0.02680 (6)	-0.00061 (4)	0.01461 (5)	0.00002 (4)
F1A	0.093 (8)	0.062 (4)	0.035 (3)	0.002 (4)	0.020 (3)	-0.009 (2)
F2A	0.045 (3)	0.095 (8)	0.076 (6)	0.009 (4)	0.030 (4)	-0.042 (6)
F3A	0.165 (15)	0.083 (7)	0.109 (9)	-0.093 (9)	0.110 (11)	-0.062 (6)
F1B	0.173 (17)	0.072 (6)	0.102 (8)	-0.060 (9)	0.113 (11)	-0.049 (6)
F2B	0.127 (11)	0.060 (6)	0.068 (5)	0.036 (6)	0.025 (5)	-0.015 (4)
F3B	0.037 (4)	0.202 (19)	0.112 (10)	-0.039 (7)	0.037 (5)	-0.108 (12)
O1	0.0305 (13)	0.0433 (14)	0.0409 (13)	-0.0065 (10)	0.0232 (11)	-0.0090 (10)
O2	0.0454 (16)	0.0530 (17)	0.0389 (13)	0.0121 (14)	0.0212 (12)	0.0132 (12)
O3	0.0558 (18)	0.0402 (14)	0.0468 (14)	-0.0161 (13)	0.0324 (14)	-0.0126 (12)
O4	0.0333 (12)	0.0281 (10)	0.0346 (10)	-0.0033 (9)	0.0218 (10)	-0.0029 (8)
O5	0.0503 (16)	0.0378 (13)	0.0446 (13)	-0.0049 (12)	0.0306 (13)	0.0024 (11)
N1	0.0304 (13)	0.0288 (12)	0.0331 (12)	-0.0023 (10)	0.0187 (11)	-0.0003 (10)
N2	0.0314 (14)	0.0352 (14)	0.0377 (13)	-0.0035 (11)	0.0218 (12)	-0.0036 (11)
C1	0.0280 (14)	0.0268 (12)	0.0309 (13)	-0.0012 (11)	0.0175 (12)	-0.0028 (10)
C2	0.0330 (16)	0.0398 (18)	0.0342 (14)	0.0019 (13)	0.0202 (13)	0.0022 (13)
C3	0.0306 (15)	0.0362 (16)	0.0339 (14)	-0.0062 (13)	0.0182 (13)	-0.0017 (12)
C4	0.050 (3)	0.086 (4)	0.073 (3)	-0.026 (3)	0.049 (3)	-0.037 (3)
C5	0.0329 (17)	0.051 (2)	0.0396 (17)	-0.0085 (15)	0.0229 (15)	-0.0123 (15)
C6	0.0372 (18)	0.0422 (19)	0.0367 (15)	0.0006 (15)	0.0196 (14)	0.0103 (14)
C7	0.0289 (15)	0.0321 (15)	0.0351 (14)	-0.0015 (12)	0.0175 (12)	0.0000 (12)
C8	0.0299 (15)	0.0305 (14)	0.0344 (13)	0.0002 (12)	0.0178 (12)	-0.0008 (11)
C9	0.044 (2)	0.0376 (17)	0.0414 (17)	-0.0063 (15)	0.0254 (16)	-0.0054 (14)

Geometric parameters (\AA , $^\circ$)

Re1—C3	1.905 (4)	O3—C3	1.155 (4)
Re1—C2	1.914 (4)	O4—C8	1.257 (4)
Re1—C1	1.924 (3)	O5—C8	1.227 (4)
Re1—N2	2.135 (3)	N1—C7	1.147 (4)
Re1—N1	2.138 (3)	N2—C5	1.136 (5)
Re1—O4	2.153 (2)	C4—C5	1.454 (6)
F1A—C9	1.354 (8)	C4—H4A	0.9800
F2A—C9	1.253 (7)	C4—H4B	0.9800
F3A—C9	1.285 (9)	C4—H4C	0.9800
F1B—C9	1.296 (8)	C6—C7	1.444 (5)
F2B—C9	1.356 (9)	C6—H6A	0.9800

F3B—C9	1.241 (10)	C6—H6B	0.9800
O1—C1	1.143 (4)	C6—H6C	0.9800
O2—C2	1.149 (4)	C8—C9	1.541 (5)
C3—Re1—C2	89.26 (15)	H6A—C6—H6B	109.5
C3—Re1—C1	89.64 (14)	C7—C6—H6C	109.5
C2—Re1—C1	88.16 (15)	H6A—C6—H6C	109.5
C3—Re1—N2	94.75 (13)	H6B—C6—H6C	109.5
C2—Re1—N2	93.44 (14)	N1—C7—C6	178.2 (4)
C1—Re1—N2	175.34 (12)	O5—C8—O4	129.8 (3)
C3—Re1—N1	92.03 (13)	O5—C8—C9	116.0 (3)
C2—Re1—N1	175.23 (12)	O4—C8—C9	114.2 (3)
C1—Re1—N1	96.43 (13)	F3B—C9—F2A	128.8 (8)
N2—Re1—N1	81.88 (12)	F3B—C9—F3A	36.0 (9)
C3—Re1—O4	173.21 (12)	F2A—C9—F3A	111.5 (9)
C2—Re1—O4	96.39 (13)	F3B—C9—F1B	108.5 (9)
C1—Re1—O4	94.28 (11)	F2A—C9—F1B	57.9 (7)
N2—Re1—O4	81.20 (10)	F3A—C9—F1B	130.9 (7)
N1—Re1—O4	82.04 (10)	F3B—C9—F1A	69.5 (10)
C8—O4—Re1	122.0 (2)	F2A—C9—F1A	106.7 (7)
C7—N1—Re1	170.6 (3)	F3A—C9—F1A	104.8 (8)
C5—N2—Re1	176.2 (3)	F1B—C9—F1A	50.1 (7)
O1—C1—Re1	178.5 (3)	F3B—C9—F2B	103.4 (10)
O2—C2—Re1	178.7 (4)	F2A—C9—F2B	46.1 (7)
O3—C3—Re1	178.3 (3)	F3A—C9—F2B	71.6 (10)
C5—C4—H4A	109.5	F1B—C9—F2B	101.3 (9)
C5—C4—H4B	109.5	F1A—C9—F2B	140.3 (6)
H4A—C4—H4B	109.5	F3B—C9—C8	116.3 (6)
C5—C4—H4C	109.5	F2A—C9—C8	113.1 (5)
H4A—C4—H4C	109.5	F3A—C9—C8	111.8 (6)
H4B—C4—H4C	109.5	F1B—C9—C8	116.1 (4)
N2—C5—C4	178.6 (5)	F1A—C9—C8	108.4 (4)
C7—C6—H6A	109.5	F2B—C9—C8	109.4 (5)
C7—C6—H6B	109.5		
C2—Re1—O4—C8	-43.2 (3)	O4—C8—C9—F2A	-72.6 (9)
C1—Re1—O4—C8	45.4 (3)	O5—C8—C9—F3A	-19.7 (11)
N2—Re1—O4—C8	-135.7 (3)	O4—C8—C9—F3A	160.5 (10)
N1—Re1—O4—C8	141.3 (3)	O5—C8—C9—F1B	171.4 (10)
Re1—O4—C8—O5	1.2 (5)	O4—C8—C9—F1B	-8.3 (11)
Re1—O4—C8—C9	-179.1 (2)	O5—C8—C9—F1A	-134.7 (7)
O5—C8—C9—F3B	-59.0 (13)	O4—C8—C9—F1A	45.5 (8)
O4—C8—C9—F3B	121.3 (13)	O5—C8—C9—F2B	57.6 (11)
O5—C8—C9—F2A	107.1 (9)	O4—C8—C9—F2B	-122.1 (10)

Hydrogen-bond geometry (\AA , $^\circ$)

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
C4—H4B \cdots O5 ⁱ	0.98	2.42	3.113 (5)	127
C6—H6A \cdots F1A ⁱⁱ	0.98	2.35	3.237 (8)	150

supplementary materials

C6—H6B \cdots O5ⁱⁱⁱ

0.98

2.46

3.075 (5)

121

Symmetry codes: (i) $-x+1, y-1/2, -z+3/2$; (ii) $-x+2, y-1/2, -z+5/2$; (iii) $-x+2, -y, -z+2$.

Fig. 1

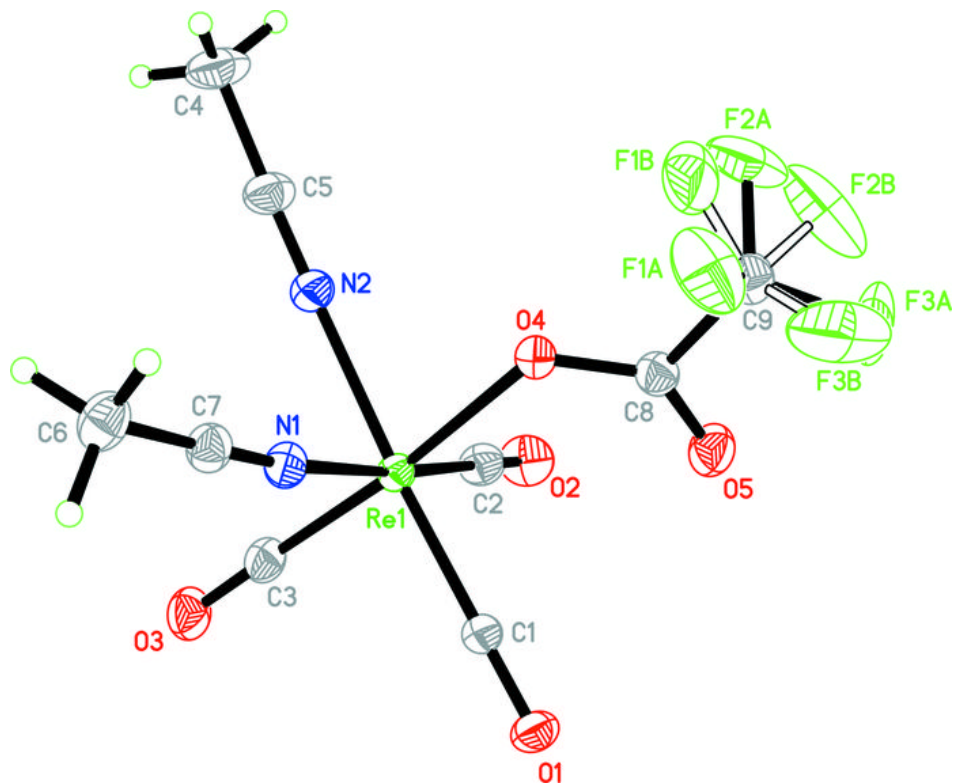


Fig. 2

