Crystal structure of 2,2′-bipyrimidine-tricarbonyl-pyridine-rhenium(I) trifluoromethanesulfonate hemihydrate, $(C_{16}H_{11}N_5O_3Re)(CF_3O_3S) \cdot 0.5H_2O$

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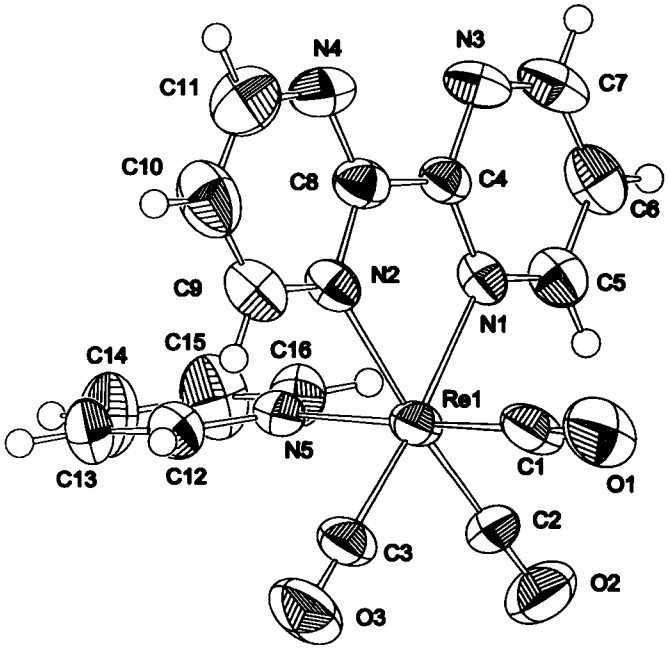

Abstract

$C_{17}H_{12}F_3N_5O_{6.50}ReS$, triclinic, $P\bar{1}$ (No. 2), $a = 7.949(2)$ Å, $b = 10.966(3)$ Å, $c = 14.947(2)$ Å, $\alpha = 104.62(2)°$, $\beta = 101.89(2)°$, $\gamma = 93.47(2)°$, $V = 1225.0$ Å^3, $Z = 2$, $R_{gt}(F) = 0.037$, $wR_{all}(F^2) = 0.093$, $T = 293$ K.

Source of material

To a solution of 200 mg (0.51 mmol) of $(\eta^3\text{-}C_3H_5)Re(CO)_3(py)$ [1] in 10 ml of dichloromethane 43.9 µl (0.50 mmol) of trifluoromethane sulfonic acid are added at 273 K. After 20 min to this mixture a solution of 190 mg (1.20 mmol) of 2,2′-bipyrimidine in 10 ml of dichloromethane is added dropwise at 273 K. After another 10 min the orange mixture is allowed to warm up to room temperature and after 1.5 h a yellow precipitate is formed which is separated, washed with n-pentane and dried in vacuo (yield 170 mg, 52 %; mp 403 K – 405 K. Crystals were obtained from a dichloromethane/acetone solution which was overlayed with n-pentane.

Discussion

Protonation of η^3-allylrhenium complexes $(\eta^3\text{-allyl})Re(CO)_3L$ by strong acids to generate double unsaturated, 14 electron cations "$Re(CO)_3L^+$" and subsequent addition of other ligands L′ is a general method for the synthesis of complexes $[Re(CO)_3LL'_2]^+$ [1-3]. Other methods include abstraction of chloride from chloro rhenium carbonyls using silver salts [4,5]. In the cation, the three CO ligands have the fac geometry as it was observed for other complexes of this type, e.g. in $[Re(CO)_3(bpym)(CH_3CN)]^+$ [4] and $[Re(CO)_3(bpym)CMeQ]^{2+}$ [4,6]. The comparable Re-C and Re-N bond lengths in the title complex are very similar to that in the latter cations.

Table 1. Data collection and handling.

Crystal:	yellow plate, size 0.07 × 0.20 × 0.40 mm
Wavelength:	Mo K_α radiation (0.71073 Å)
µ:	51.10 cm^{-1}
Diffractometer, scan mode:	Enraf-Nonius CAD4, ω/2θ
$2\theta_{max}$:	47.94°
$N(hkl)_{measured}$, $N(hkl)_{unique}$:	4130, 3819
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\,\sigma(I_{obs})$, 3130
$N(param)_{refined}$:	307
Programs:	SHELXS-86 [7], SHELXL-93 [8], ZORTEP [9], MolEN [10]

Table 2. Atomic coordinates and displacement parameters (in Å^2).

Atom	Site	Occ.	x	y	z	U_{iso}
H(5)	2*i*		0.07(1)	0.18(1)	0.815(7)	0.062
H(6)	2*i*		–0.20(1)	0.24(1)	0.766(7)	0.071
H(7)	2*i*		–0.43(1)	0.09(1)	0.706(8)	0.072
H(9)	2*i*		0.15(1)	–0.39(1)	0.790(8)	0.070
H(10)	2*i*		–0.08(2)	–0.55(1)	0.73(1)	0.092
H(11)	2*i*		–0.35(2)	–0.49(1)	0.68(1)	0.091
H(12)	2*i*		0.31(1)	–0.30(1)	0.672(7)	0.061
H(13)	2*i*		0.35(1)	–0.35(1)	0.521(8)	0.075
H(14)	2*i*		0.32(2)	–0.19(1)	0.437(8)	0.092
H(15)	2*i*		0.23(2)	–0.00(1)	0.508(8)	0.095
H(16)	2*i*		0.19(1)	0.04(1)	0.660(7)	0.069
H(7A)	2*i*	0.50	0.08(7)	0.42(5)	0.91(4)	0.360
H(7B)	2*i*	0.50	0.18(7)	0.37(5)	1.03(4)	0.360

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Table 3. Atomic coordinates and displacement parameters (in $Å^2$).

Atom	Site	Occ.	x	y	z	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
Re(1)	2*i*		0.2235(5)	–0.0854(4)	0.8307(3)	0.031(2)	0.056(3)	0.036(2)	0.008(2)	0.009(1)	0.012(2)
O(1)	2*i*		0.18(1)	–0.04(1)	1.035(5)	0.07(5)	0.15(9)	0.04(4)	0.03(5)	0.03(4)	0.02(5)
O(2)	2*i*		0.47(1)	0.160(9)	0.892(6)	0.07(5)	0.08(6)	0.07(5)	–0.02(5)	0.01(4)	–0.00(5)
O(3)	2*i*		0.53(1)	–0.23(1)	0.885(6)	0.06(5)	0.13(8)	0.07(5)	0.05(6)	0.01(4)	0.03(5)
N(1)	2*i*		–0.013(9)	–0.005(7)	0.785(5)	0.04(4)	0.04(4)	0.05(4)	0.01(3)	0.02(3)	0.01(3)
N(2)	2*i*		0.021(9)	–0.246(7)	0.775(5)	0.04(4)	0.04(5)	0.05(4)	0.01(4)	0.02(3)	0.02(4)
N(3)	2*i*		–0.31(1)	–0.054(9)	0.719(6)	0.03(4)	0.07(6)	0.07(5)	0.01(4)	0.01(4)	0.03(5)
N(4)	2*i*		–0.28(1)	–0.305(9)	0.710(6)	0.04(5)	0.07(6)	0.07(6)	–0.01(4)	0.01(4)	0.01(5)
N(5)	2*i*		0.247(9)	–0.127(7)	0.681(5)	0.03(4)	0.04(4)	0.04(4)	0.01(3)	0.01(3)	0.02(4)
C(1)	2*i*		0.20(1)	–0.05(1)	0.960(7)	0.04(5)	0.09(8)	0.05(6)	0.02(5)	0.02(4)	0.02(6)
C(2)	2*i*		0.38(1)	0.07(1)	0.868(7)	0.04(5)	0.07(7)	0.04(5)	–0.00(5)	0.00(4)	–0.01(5)
C(3)	2*i*		0.42(1)	–0.17(1)	0.865(7)	0.04(5)	0.09(8)	0.04(5)	0.02(6)	0.01(4)	0.01(5)
C(4)	2*i*		–0.16(1)	–0.086(9)	0.749(6)	0.03(5)	0.05(6)	0.04(5)	0.01(4)	0.01(4)	0.01(4)
C(5)	2*i*		–0.03(1)	0.12(1)	0.791(7)	0.05(6)	0.05(6)	0.06(6)	0.01(5)	0.02(5)	0.02(5)
C(6)	2*i*		–0.19(1)	0.16(1)	0.762(7)	0.07(8)	0.06(7)	0.07(7)	0.03(6)	0.03(6)	0.03(6)
C(7)	2*i*		–0.32(1)	0.07(1)	0.726(8)	0.04(6)	0.09(9)	0.07(7)	0.03(6)	0.02(5)	0.04(6)
C(8)	2*i*		–0.14(1)	–0.221(9)	0.743(6)	0.04(5)	0.06(6)	0.04(5)	0.01(5)	0.01(4)	0.02(4)
C(9)	2*i*		0.04(1)	–0.37(1)	0.769(8)	0.06(6)	0.06(7)	0.07(7)	0.02(6)	0.02(5)	0.03(6)
C(10)	2*i*		–0.09(2)	–0.46(1)	0.73(1)	0.09(9)	0.05(7)	0.1(1)	0.02(7)	0.04(8)	0.03(7)
C(11)	2*i*		–0.25(2)	–0.43(1)	0.71(1)	0.08(9)	0.05(7)	0.10(9)	–0.01(6)	0.02(7)	0.02(6)
C(12)	2*i*		0.29(1)	–0.24(1)	0.639(7)	0.05(6)	0.05(6)	0.05(6)	0.01(5)	0.01(5)	0.02(5)
C(13)	2*i*		0.32(1)	–0.27(1)	0.548(8)	0.07(7)	0.06(7)	0.06(7)	0.01(6)	0.03(5)	–0.00(5)
C(14)	2*i*		0.30(2)	–0.18(1)	0.498(8)	0.1(1)	0.08(9)	0.05(6)	0.02(8)	0.03(7)	0.02(6)
C(15)	2*i*		0.25(2)	–0.06(1)	0.540(8)	0.1(1)	0.09(9)	0.06(7)	0.03(8)	0.04(7)	0.04(7)
C(16)	2*i*		0.22(1)	–0.04(1)	0.632(7)	0.06(7)	0.05(7)	0.06(6)	0.00(5)	0.02(5)	0.01(5)
S(1)	2*i*		0.309(4)	0.345(3)	0.672(2)	0.07(2)	0.05(2)	0.10(2)	0.02(1)	0.03(2)	0.03(2)
F(1)	2*i*		0.34(1)	0.409(9)	0.521(7)	0.13(8)	0.12(7)	0.12(7)	0.01(6)	0.05(6)	0.07(6)
F(2)	2*i*		0.19(2)	0.23(1)	0.492(7)	0.2(1)	0.11(8)	0.11(7)	–0.01(7)	–0.01(7)	0.01(6)
F(3)	2*i*		0.09(1)	0.41(1)	0.540(8)	0.10(7)	0.16(9)	0.2(1)	0.04(7)	–0.01(7)	0.08(8)
O(4)	2*i*		0.34(1)	0.478(8)	0.722(7)	0.08(6)	0.06(5)	0.11(7)	0.02(5)	0.04(5)	0.02(5)
O(5)	2*i*		0.17(2)	0.28(1)	0.692(9)	0.1(1)	0.09(8)	0.2(1)	–0.00(7)	0.08(8)	0.06(7)
O(6)	2*i*		0.46(1)	0.28(1)	0.668(7)	0.11(8)	0.10(7)	0.11(8)	0.07(6)	0.03(6)	0.04(6)
C(17)	2*i*		0.23(2)	0.35(1)	0.55(1)	0.07(9)	0.07(9)	0.1(1)	0.00(7)	0.01(8)	0.03(9)
O(7)	2*i*	0.50	0.16(7)	0.45(5)	0.96(4)	0.3(3)	0.3(3)	0.3(3)	0.0(1)	0.1(1)	0.1(1)

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