Crystal structure of *tert*-butylisocyanide-tricarbonyl-1,10-phenanthroline-rhenium(I) trifluoromethanesulfonate, $[Re(C_5H_9N)(CO)_3(C_{12}H_8N_2)](F_3CSO_3)$

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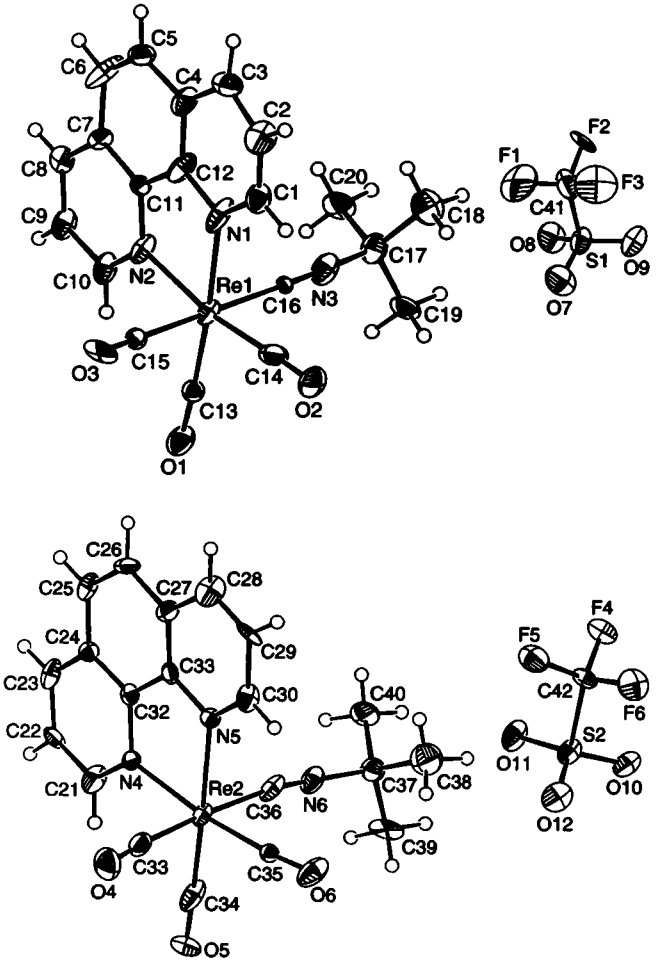

Abstract

$C_{21}H_{17}F_3N_3O_6ReS$, triclinic, $P\bar{1}$ (no. 2), $a = 11.931(1)$ Å, $b = 14.313(1)$ Å, $c = 14.804(1)$ Å, $\alpha = 91.63(1)°$, $\beta = 102.74(1)°$, $\gamma = 102.52(1)°$, $V = 2399.6$ Å^3, $Z = 4$, $R_{gt}(F) = 0.060$, $wR_{obs}(F^2) = 0.202$, $T = 193$ K.

Source of material

To a solution of $(\eta^3\text{-}C_3H_5)Re(CO)_3(t\text{-BuNC})$ [1] (100 mg, 0.25 mmol) in dichloromethane (10 ml) trifluoromethane sulfonic acid (21.5 µl, 0.245 mmol) was added at 0 °C. After 15 min 1.10-phenanthroline (47 mg, 0.26 mmol) was added and the mixture was allowed to warm up to room temperature. After 2 h *n*-hexane (10 ml) was added and the solvents were removed in vacuo. The oily product was stirred with *n*-pentane to give a yellow powder which was centrifuged off (yield 130 mg, 74 %). Crystals were obtained by diffusion of *n*-pentane to a solution of the complex in THF/acetone (4:1) (m.p. 147-151°C, decomposition). Elemental analysis: found – C, 37.56 %; H, 2.37 %; N, 6.30 %; calc. for $C_{21}H_{17}F_3N_3O_6ReS$: C, 36.95 %; H, 2.51 %; N, 6.16 %. IR, ^{1}H NMR and ^{13}C NMR data are available in the CIF file.

Experimental details

Additional crystallographic symmetry was not observed for the two independent molecular units. Some carbon atoms show unusual anisotropy.

Discussion

The reaction of $(\eta^3\text{-}C_3H_5)Re(CO)_3(t\text{-BuNC})$ [1] with trifluoromethane sulfonic acid yields under elimination of propene the very reactive organo-metallic Lewis acid $Re(CO)_3(t\text{-BuNC})$(triflate) [2]. By addition of 1,10-phenanthroline the title complex was obtained. This complex was recently reported by Dumas et al. [3] and Meyer et al. [4] starting from $Re(CO)_5Cl$. Analogous complexes could be synthesized from $Re(CO)_3(t\text{-BuNC})$(triflate) and 1,2-diphenylphosphinoethane [5] or 2,2′-bipyrimidine [6] and were also characterized by X-ray analysis. Carbonyl-rhenium complexes with *N*-heterocycles have attracted tremendous interest because of their remarkable photophysical properties. As in other tricarbonylrhenium complexes the carbonyl ligands occupy the fac positions [3-7]. The donor function of the nitrogen atoms of the phenanthroline ligand leads to a larger back donation in the *trans* Re–C–O groups and to a shortening of the Re—C distances, d(Re2—C34) = 1.92(2) Å, d(Re2—C35) = 1.94(2) Å compared with d(Re2—C33) = 2.00(2) Å). The Re—C(NBu) and Re—N bonds of the title complex are of similar lengths as in other comparable complexes [5-11]. The analogous manganese complex with perchlorate, $Mn(CO)_3$(CN-*tert*-butyl)(1,10-phenanthroline)ClO_4, has the same structure [12] and shows the same altering in the *M*—C distances as the title compound.

Table 1. Data collection and handling.

Crystal:	orange prism, size 0.25 × 0.25 × 0.3 mm
Wavelength:	Mo K_α radiation (0.71073 Å)
μ:	52.16 cm^{-1}
Diffractometer, scan mode:	Siemens SMART CCD, φ/ω
$2\theta_{max}$:	58.48°
$N(hkl)_{measured}$, $N(hkl)_{unique}$:	13857, 7436
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\,\sigma(I_{obs})$, 6858
$N(param)_{refined}$:	637
Programs:	SHELXTL [13], SHELXL-93 [14]

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

Atom	Site	x	y	z	U_{iso}
H(1A)	2*i*	0.340(1)	−0.299(1)	−0.097(1)	0.049
H(2A)	2*i*	0.285(2)	−0.243(1)	0.025(2)	0.060
H(3A)	2*i*	0.422(2)	−0.129(1)	0.139(1)	0.050
H(5A)	2*i*	0.612(2)	0.008(1)	0.166(1)	0.048
H(6A)	2*i*	0.779(2)	0.073(1)	0.126(2)	0.068
H(8B)	2*i*	0.912(1)	0.082(1)	0.010(1)	0.041
H(9B)	2*i*	0.942(1)	0.018(1)	−0.125(1)	0.046
H(10B)	2*i*	0.804(1)	−0.110(1)	−0.212(1)	0.043
H(18A)	2*i*	0.67(1)	−0.440(2)	0.088(7)	0.084
H(18B)	2*i*	0.721(6)	−0.530(9)	0.085(8)	0.084
H(18C)	2*i*	0.606(6)	−0.525(9)	0.012(2)	0.084
H(19A)	2*i*	0.714(2)	−0.553(6)	−0.116(7)	0.064
H(19B)	2*i*	0.83(1)	−0.555(6)	−0.037(1)	0.064
H(19C)	2*i*	0.84(1)	−0.484(1)	−0.115(7)	0.064
H(20A)	2*i*	0.848(2)	−0.324(6)	0.075(8)	0.073
H(20B)	2*i*	0.916(6)	−0.343(8)	−0.001(2)	0.073
H(20C)	2*i*	0.912(6)	−0.409(2)	0.081(7)	0.073

Table 2. Continued.

Atom	Site	x	y	z	U_{iso}
H(21A)	2*i*	0.465(1)	0.216(1)	0.621(1)	0.045
H(22A)	2*i*	0.515(1)	0.114(1)	0.522(1)	0.036
H(23A)	2*i*	0.370(1)	0.016(1)	0.407(1)	0.043
H(25A)	2*i*	0.153(1)	−0.0376(9)	0.330(1)	0.039
H(26A)	2*i*	−0.035(1)	−0.016(1)	0.312(1)	0.037
H(28A)	2*i*	−0.178(1)	0.065(1)	0.371(2)	0.052
H(29A)	2*i*	−0.209(1)	0.177(1)	0.470(1)	0.031
H(30A)	2*i*	−0.049(1)	0.266(1)	0.581(1)	0.039
H(38A)	2*i*	0.104(2)	0.519(5)	0.41(1)	0.086
H(38B)	2*i*	0.177(7)	0.626(6)	0.41(1)	0.086
H(38C)	2*i*	0.165(8)	0.58(1)	0.501(2)	0.086
H(39A)	2*i*	0.384(7)	0.628(8)	0.546(4)	0.083
H(39B)	2*i*	0.394(8)	0.663(5)	0.448(6)	0.083
H(39C)	2*i*	0.459(2)	0.584(4)	0.49(1)	0.083
H(40A)	2*i*	0.222(5)	0.459(8)	0.310(4)	0.073
H(40B)	2*i*	0.358(8)	0.471(9)	0.349(1)	0.073
H(40C)	2*i*	0.31(1)	0.559(2)	0.310(4)	0.073

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

Atom	Site	x	y	z	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
Re(1)	2*i*	0.55051(4)	−0.24335(4)	−0.20591(5)	0.0254(3)	0.0207(3)	0.0417(5)	0.0026(2)	0.0116(3)	−0.0052(3)
Re(2)	2*i*	0.23485(4)	0.29056(3)	0.64585(5)	0.0194(3)	0.0173(3)	0.0391(5)	0.0036(2)	0.0082(3)	−0.0033(2)
N(1)	2*i*	0.491(1)	−0.2052(8)	−0.083(1)	0.030(6)	0.021(6)	0.09(1)	0.001(5)	0.027(7)	−0.014(7)
N(2)	2*i*	0.6909(9)	−0.1241(8)	−0.131(1)	0.021(5)	0.022(5)	0.05(1)	0.003(4)	0.006(6)	−0.009(6)
N(3)	2*i*	0.688(1)	−0.3825(9)	−0.077(1)	0.033(6)	0.033(7)	0.07(1)	0.000(5)	0.020(7)	−0.005(7)
N(4)	2*i*	0.2960(9)	0.1887(7)	0.5686(8)	0.021(2)	0.020(2)	0.020(2)	0.005(1)	0.005(1)	0.002(1)
N(5)	2*i*	0.0752(9)	0.2123(7)	0.5471(9)	0.021(2)	0.022(2)	0.021(2)	0.005(1)	0.005(1)	0.002(1)
N(6)	2*i*	0.276(1)	0.4473(9)	0.498(1)	0.027(6)	0.022(6)	0.06(1)	0.001(5)	0.011(6)	−0.002(6)
O(1)	2*i*	0.664(1)	−0.2774(8)	−0.368(1)	0.050(6)	0.034(6)	0.06(1)	0.011(5)	0.025(6)	−0.003(6)
O(2)	2*i*	0.343(1)	−0.4165(8)	−0.283(1)	0.048(6)	0.036(6)	0.06(1)	−0.014(5)	0.014(6)	−0.006(6)
O(3)	2*i*	0.415(1)	−0.1132(9)	−0.3256(8)	0.052(7)	0.067(8)	0.015(6)	0.034(6)	−0.006(6)	0.014(6)
O(4)	2*i*	0.197(1)	0.1606(9)	0.808(1)	0.060(8)	0.041(7)	0.06(1)	0.012(6)	0.009(8)	0.017(7)
O(5)	2*i*	0.4713(9)	0.3946(8)	0.7740(8)	0.034(5)	0.053(7)	0.020(7)	0.004(5)	−0.002(5)	0.002(5)
O(6)	2*i*	0.115(1)	0.4293(8)	0.731(1)	0.035(5)	0.041(6)	0.07(1)	0.008(5)	0.016(6)	−0.022(6)
O(7)	2*i*	−0.082(1)	0.260(1)	0.090(1)	0.062(9)	0.10(1)	0.07(1)	−0.018(8)	0.008(9)	0.01(1)
O(8)	2*i*	0.119(1)	0.300(1)	0.188(1)	0.043(7)	0.08(1)	0.08(1)	−0.006(6)	0.024(8)	0.010(9)
O(9)	2*i*	−0.015(1)	0.154(1)	0.209(1)	0.060(8)	0.042(7)	0.09(1)	0.007(6)	0.014(8)	−0.008(8)
O(10)	2*i*	0.3146(9)	0.9043(7)	0.2547(9)	0.033(5)	0.031(5)	0.047(8)	0.002(4)	0.012(5)	−0.009(5)
O(11)	2*i*	0.3583(9)	0.7461(8)	0.257(1)	0.026(5)	0.037(6)	0.08(1)	0.014(4)	0.009(6)	−0.011(6)
O(12)	2*i*	0.271(1)	0.8019(9)	0.375(1)	0.038(6)	0.048(7)	0.049(9)	0.007(5)	0.022(6)	−0.001(6)
S(1)	2*i*	0.0005(4)	0.2491(3)	0.1813(4)	0.046(2)	0.034(2)	0.074(4)	0.006(2)	0.027(3)	0.004(2)
S(2)	2*i*	0.2879(3)	0.8080(2)	0.2817(3)	0.021(1)	0.027(2)	0.048(3)	0.004(1)	0.011(2)	−0.005(2)
F(1)	2*i*	−0.057(2)	0.401(1)	0.234(2)	0.25(3)	0.09(1)	0.14(2)	0.12(2)	0.03(2)	0.00(1)
F(2)	2*i*	0.014(2)	0.313(1)	0.3514(8)	0.16(1)	0.081(9)	0.016(6)	−0.007(9)	0.013(8)	0.022(6)
F(3)	2*i*	−0.168(1)	0.266(2)	0.259(1)	0.059(8)	0.16(2)	0.14(2)	0.05(1)	0.046(9)	0.00(1)
F(4)	2*i*	0.1387(7)	0.7603(7)	0.1216(7)	0.031(4)	0.055(6)	0.030(6)	0.008(4)	0.003(4)	0.008(4)
F(5)	2*i*	0.1077(8)	0.6619(7)	0.2262(8)	0.041(5)	0.043(5)	0.048(7)	−0.007(4)	−0.002(5)	0.012(5)
F(6)	2*i*	0.0599(7)	0.7990(8)	0.2302(8)	0.026(4)	0.064(6)	0.062(8)	0.017(4)	0.013(5)	0.000(6)
C(1)	2*i*	0.391(1)	−0.246(1)	−0.059(1)	0.038(8)	0.032(7)	0.05(1)	0.003(6)	0.021(8)	0.004(7)
C(2)	2*i*	0.359(2)	−0.216(1)	0.016(2)	0.038(8)	0.049(9)	0.07(2)	0.011(7)	0.027(9)	0.000(9)
C(3)	2*i*	0.439(2)	−0.144(1)	0.082(1)	0.049(9)	0.051(9)	0.03(1)	0.020(8)	0.013(8)	0.004(8)
C(4)	2*i*	0.544(1)	−0.098(1)	0.058(1)	0.044(8)	0.028(7)	0.05(1)	0.016(6)	0.019(8)	−0.004(7)
C(5)	2*i*	0.628(2)	−0.017(1)	0.112(1)	0.06(1)	0.038(8)	0.03(1)	0.024(8)	0.009(8)	−0.001(7)
C(6)	2*i*	0.726(2)	0.023(1)	0.088(2)	0.042(9)	0.040(9)	0.08(2)	0.012(7)	−0.00(1)	−0.04(1)
C(7)	2*i*	0.753(1)	−0.009(1)	0.003(1)	0.036(7)	0.027(7)	0.017(8)	0.016(6)	−0.015(7)	−0.001(6)
C(8)	2*i*	0.856(1)	0.031(1)	−0.025(1)	0.033(7)	0.034(7)	0.029(9)	0.013(6)	−0.015(7)	0.008(7)
C(9)	2*i*	0.874(1)	−0.008(1)	−0.105(1)	0.027(6)	0.029(7)	0.06(1)	0.002(5)	0.013(7)	0.000(7)
C(10)	2*i*	0.790(1)	−0.085(1)	−0.158(1)	0.027(6)	0.025(6)	0.06(1)	0.005(5)	0.016(7)	0.004(7)
C(11)	2*i*	0.673(1)	−0.0864(9)	−0.052(1)	0.031(6)	0.023(6)	0.017(8)	0.011(5)	−0.001(6)	0.004(5)
C(12)	2*i*	0.566(1)	−0.130(1)	−0.027(1)	0.025(6)	0.028(7)	0.06(1)	0.010(5)	0.001(7)	−0.012(7)
C(13)	2*i*	0.623(1)	−0.2666(9)	−0.304(1)	0.026(3)	0.026(3)	0.026(3)	0.006(1)	0.006(1)	0.002(1)
C(14)	2*i*	0.422(2)	−0.351(1)	−0.252(1)	0.051(9)	0.044(9)	0.02(1)	0.019(7)	0.006(8)	0.000(7)
C(15)	2*i*	0.465(1)	−0.1562(9)	−0.285(1)	0.024(3)	0.024(3)	0.023(3)	0.005(1)	0.006(1)	0.003(1)
C(16)	2*i*	0.639(1)	−0.3346(8)	−0.1229(9)	0.016(2)	0.015(2)	0.014(2)	0.003(1)	0.003(1)	0.003(1)
C(17)	2*i*	0.756(1)	−0.440(1)	−0.018(1)	0.033(7)	0.033(7)	0.05(1)	0.007(6)	0.007(8)	0.003(7)

Table 3. Continued.

Atom	Site	x	y	z	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
C(18)	2*i*	0.680(2)	−0.488(2)	0.048(2)	0.043(9)	0.06(1)	0.06(2)	0.006(8)	0.02(1)	0.01(1)
C(19)	2*i*	0.785(2)	−0.515(1)	−0.077(1)	0.06(1)	0.05(1)	0.020(9)	0.021(8)	0.002(8)	0.010(8)
C(20)	2*i*	0.869(2)	−0.373(1)	0.040(1)	0.043(9)	0.06(1)	0.03(1)	−0.002(8)	−0.011(8)	0.003(8)
C(21)	2*i*	0.405(1)	0.179(1)	0.575(1)	0.018(6)	0.028(7)	0.07(1)	0.004(5)	0.010(7)	−0.006(7)
C(22)	2*i*	0.436(1)	0.116(1)	0.515(1)	0.029(6)	0.041(8)	0.020(8)	0.017(6)	−0.002(6)	0.007(6)
C(23)	2*i*	0.350(1)	0.059(1)	0.446(1)	0.045(8)	0.024(6)	0.05(1)	0.016(6)	0.025(8)	0.004(7)
C(24)	2*i*	0.233(1)	0.0658(9)	0.436(1)	0.022(3)	0.022(3)	0.022(3)	0.005(1)	0.005(1)	0.002(1)
C(25)	2*i*	0.138(1)	0.0082(9)	0.368(1)	0.041(8)	0.017(6)	0.04(1)	0.006(5)	0.018(8)	0.004(6)
C(26)	2*i*	0.026(1)	0.019(1)	0.360(1)	0.034(7)	0.032(7)	0.018(9)	0.000(6)	−0.002(6)	−0.007(6)
C(27)	2*i*	0.001(1)	0.086(1)	0.424(1)	0.028(6)	0.025(6)	0.03(1)	0.006(5)	0.001(7)	−0.005(6)
C(28)	2*i*	−0.115(1)	0.101(1)	0.416(2)	0.023(7)	0.037(8)	0.07(2)	−0.005(6)	0.019(8)	0.000(8)
C(29)	2*i*	−0.134(1)	0.167(1)	0.474(1)	0.021(6)	0.050(8)	0.011(7)	0.013(5)	0.003(6)	0.017(6)
C(30)	2*i*	−0.036(1)	0.221(1)	0.540(1)	0.021(6)	0.031(7)	0.05(1)	0.009(5)	0.014(7)	0.004(7)
C(31)	2*i*	0.091(1)	0.1429(9)	0.488(1)	0.023(6)	0.020(6)	0.028(9)	0.009(5)	0.002(6)	0.006(6)
C(32)	2*i*	0.209(1)	0.1323(9)	0.496(1)	0.024(6)	0.019(5)	0.023(8)	0.003(5)	0.008(6)	0.007(5)
C(33)	2*i*	0.210(1)	0.205(1)	0.747(2)	0.032(7)	0.030(7)	0.05(1)	0.012(6)	0.001(8)	−0.011(8)
C(34)	2*i*	0.384(1)	0.357(1)	0.723(2)	0.029(7)	0.021(6)	0.07(2)	0.001(5)	0.022(8)	−0.007(7)
C(35)	2*i*	0.161(1)	0.3760(8)	0.704(1)	0.019(2)	0.019(2)	0.018(3)	0.004(1)	0.004(1)	0.002(1)
C(36)	2*i*	0.261(1)	0.388(1)	0.545(1)	0.021(6)	0.022(7)	0.06(1)	0.001(5)	0.014(7)	−0.013(7)
C(37)	2*i*	0.282(1)	0.531(1)	0.440(1)	0.031(3)	0.031(3)	0.031(3)	0.007(1)	0.007(1)	0.003(1)
C(38)	2*i*	0.172(2)	0.567(2)	0.438(2)	0.057(5)	0.058(5)	0.057(5)	0.014(2)	0.014(2)	0.006(2)
C(39)	2*i*	0.389(2)	0.609(1)	0.485(1)	0.09(2)	0.041(9)	0.009(8)	−0.014(9)	−0.008(9)	0.019(7)
C(40)	2*i*	0.294(2)	0.502(1)	0.343(1)	0.08(1)	0.041(9)	0.019(9)	0.009(9)	0.003(9)	0.011(7)
C(41)	2*i*	−0.055(3)	0.314(2)	0.260(2)	0.12(2)	0.06(1)	0.09(2)	0.05(1)	0.07(2)	0.04(1)
C(42)	2*i*	0.142(1)	0.754(1)	0.212(1)	0.032(7)	0.038(7)	0.009(7)	0.007(6)	0.006(6)	0.009(6)

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