Crystal structure of *t*-butylisocyanide-tricarbonyl-bis(1,2-diphenylphosphinoethane)-rhenium(I) trifluoromethanesulfonate, (C₃₄H₃₃NO₃P₂Re)(CF₃O₃S)

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Abstract

C₃₅H₃₃F₃NO₃P₂ReS, monoclinic, *P*12₁/*n*1 (No. 14), *a* = 13.573(3) Å, *b* = 14.305(3) Å, *c* = 19.344(5) Å, β = 95.07(2)°, *V* = 3741.2 Å³, *Z* = 4, *R*_g(*F*) = 0.034, *wR*_{all}(*F*²) = 0.119, *T* = 293 K.

Source of material

To a solution of 79 mg (0.20 mmol) of (η³-C₃H₅)Re(CO)₃-(*t*-BuNC) [1] in 10 ml of dichloromethane 17.1 μl (0.195 mmol) of trifluoromethane sulfonic acid are added at 273 K. After 15 min 77.7 mg (0.195 mmol) of bis(1,3-diphenylphosphinoethane) are added and the mixture is allowed to warm up to room temperature, whereby it becomes yellow. After 2 h, 10 ml of *n*-hexane are added and the solvent is removed in vacuo. The residue is dissolved in trichloromethane and colorless crystals were obtained by diffusion of *n*-pentane into the solution (yield 61 mg, 35 %).

Discussion

The reaction of (η³-C₃H₅)Re(CO)₃-(*t*-BuNC) [1] with trifluoromethane sulfonic acid yields under elimination of propene [2] the very reactive Lewis acid Re(CO)₃-(*t*-BuNC)-(triflate) and by addition of 1,2-diphenylphosphinoethane (DPPE) the title complex was obtained. In the cation [Re(CO)₃-(*t*-BuNC)(DPPE)]⁺ the three CO groups show the fac geometry as has been observed for

many tricarbonyl rhenium complexes of the type [Re(CO)₃LL'₂]⁺ [3,4]. The bond lengths *d*(Re—CN) = 2.46 Å and *d*(Re—P) = 2.46 Å lie in the expected range [1,5].

Table 1. Data collection and handling.

Crystal:	transparent orange plate, size 0.17 × 0.33 × 0.53 mm
Wavelength:	Mo K _α radiation (0.71069 Å)
μ:	34.48 cm ⁻¹
Diffractometer, scan mode:	Enraf-Nonius CAD4, ω/2θ
2θ _{max} :	49.94°
<i>N</i> (<i>hkl</i>) _{measured} , <i>N</i> (<i>hkl</i>) _{unique} :	6772, 6565
Criterion for <i>I</i> _{obs} , <i>N</i> (<i>hkl</i>) _{gt} :	<i>I</i> _{obs} > 2 σ(<i>I</i> _{obs}), 5376
<i>N</i> (<i>param</i>) _{refined} :	523
Programs:	SHELXS-86 [6], SHELXL-93 [7], ZORTEP [8], MolEN [9]

Table 2. Atomic coordinates and displacement parameters (in Å²).

Atom	Site	<i>x</i>	<i>y</i>	<i>z</i>	<i>U</i> _{iso}
H(6A)	4e	0.285(2)	0.475(6)	0.3490(8)	0.178
H(6B)	4e	0.224(6)	0.558(2)	0.375(2)	0.178
H(6C)	4e	0.189(4)	0.455(5)	0.386(2)	0.178
H(7A)	4e	0.389(5)	0.4043(7)	0.517(3)	0.160
H(7B)	4e	0.368(6)	0.365(2)	0.442(2)	0.160
H(7C)	4e	0.285(2)	0.363(2)	0.493(4)	0.160
H(8A)	4e	0.365(1)	0.621(2)	0.435(4)	0.139
H(8B)	4e	0.437(3)	0.539(3)	0.421(3)	0.139
H(8C)	4e	0.423(3)	0.569(4)	0.4975(9)	0.139
H(9A)	4e	0.2808(5)	0.8581(4)	0.6436(4)	0.079
H(9B)	4e	0.2176(5)	0.8207(4)	0.7021(4)	0.079
H(10A)	4e	0.3707(5)	0.7434(5)	0.7123(4)	0.084
H(10B)	4e	0.3557(5)	0.7117(5)	0.6344(4)	0.084
H(12)	4e	0.3341(7)	0.8334(6)	0.5419(5)	0.110
H(13)	4e	0.3881(9)	0.8530(9)	0.4311(7)	0.153
H(14)	4e	0.287(1)	0.8085(8)	0.3354(6)	0.141
H(15)	4e	0.1337(9)	0.7486(6)	0.3454(4)	0.122
H(16)	4e	0.0805(6)	0.7292(5)	0.4557(3)	0.089
H(18)	4e	0.1490(6)	0.9650(5)	0.5884(4)	0.084
H(19)	4e	0.0313(6)	1.0807(5)	0.5898(4)	0.093
H(20)	4e	-0.1345(7)	1.0412(6)	0.5838(4)	0.099
H(21)	4e	-0.1810(6)	0.8865(5)	0.5887(4)	0.092
H(22)	4e	-0.0623(5)	0.7710(5)	0.5942(4)	0.080
H(24)	4e	0.1353(6)	0.5323(5)	0.7940(3)	0.084
H(25)	4e	0.1093(8)	0.5460(7)	0.9109(4)	0.115
H(26)	4e	0.1978(9)	0.6548(9)	0.9785(4)	0.131
H(27)	4e	0.3138(8)	0.7440(7)	0.9339(5)	0.124

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Table 2. Continued.

Atom	Site	x	y	z	U _{iso}
H(28)	4e	0.3421(6)	0.7308(6)	0.8190(4)	0.102
H(30)	4e	0.2751(6)	0.4220(5)	0.6618(4)	0.091
H(31)	4e	0.3963(6)	0.3110(6)	0.6734(5)	0.105

Table 2. Continued.

Atom	Site	x	y	z	U _{iso}
H(32)	4e	0.5543(6)	0.3489(6)	0.7177(4)	0.097
H(33)	4e	0.5891(6)	0.4987(7)	0.7503(4)	0.100
H(34)	4e	0.4683(5)	0.6118(6)	0.7419(4)	0.092

Table 3. Atomic coordinates and displacement parameters (in Å²).

Atom	Site	Occ.	x	y	z	U ₁₁	U ₂₂	U ₃₃	U ₁₂	U ₁₃	U ₂₃
Re(1)	4e		0.10782(2)	0.60350(2)	0.62205(1)	0.0510(2)	0.0481(2)	0.0438(2)	-0.00842(9)	0.0019(1)	-0.00085(9)
P(1)	4e		0.1509(1)	0.7654(1)	0.59380(8)	0.0570(9)	0.0502(8)	0.0492(8)	-0.0094(6)	-0.0012(6)	0.0040(6)
P(2)	4e		0.2596(1)	0.6179(1)	0.70156(8)	0.0555(9)	0.0565(9)	0.0487(8)	-0.0082(7)	-0.0031(7)	0.0041(6)
O(1)	4e		-0.0655(4)	0.6090(3)	0.5081(3)	0.074(3)	0.081(3)	0.081(4)	-0.009(2)	-0.019(3)	-0.012(2)
O(2)	4e		0.0642(5)	0.3975(3)	0.6582(3)	0.110(5)	0.056(3)	0.105(4)	-0.018(3)	0.027(4)	0.008(3)
O(3)	4e		-0.0284(4)	0.6703(4)	0.7331(3)	0.098(4)	0.091(4)	0.073(3)	0.011(3)	0.028(3)	-0.006(3)
N(1)	4e		0.2439(4)	0.5274(4)	0.5060(3)	0.063(3)	0.074(4)	0.065(3)	0.003(3)	0.015(3)	0.006(3)
C(1)	4e		-0.0025(5)	0.6063(4)	0.5509(3)	0.057(4)	0.054(3)	0.056(3)	-0.005(3)	-0.008(3)	-0.006(2)
C(2)	4e		0.0813(5)	0.4728(5)	0.6461(3)	0.062(4)	0.058(4)	0.065(4)	-0.011(3)	0.013(3)	-0.001(3)
C(3)	4e		0.0219(5)	0.6476(5)	0.6929(3)	0.070(4)	0.056(3)	0.056(3)	-0.004(3)	0.005(3)	-0.003(3)
C(4)	4e		0.1971(4)	0.5568(4)	0.5480(3)	0.057(3)	0.054(3)	0.052(3)	-0.003(3)	0.004(3)	0.004(3)
C(5)	4e		0.3063(6)	0.4925(6)	0.4540(4)	0.089(5)	0.095(6)	0.083(5)	0.012(4)	0.038(4)	0.006(4)
C(6)	4e		0.2456(9)	0.495(1)	0.3847(5)	0.14(1)	0.24(2)	0.067(6)	0.02(1)	0.032(6)	-0.016(7)
C(7)	4e		0.340(1)	0.3980(7)	0.4785(8)	0.17(1)	0.095(8)	0.15(1)	0.031(7)	0.07(1)	0.003(6)
C(8)	4e		0.3905(7)	0.5617(8)	0.4518(6)	0.100(7)	0.126(8)	0.130(8)	0.001(6)	0.058(6)	0.012(7)
C(9)	4e		0.2473(5)	0.8036(4)	0.6600(4)	0.074(4)	0.053(3)	0.067(4)	-0.014(3)	-0.011(3)	0.003(3)
C(10)	4e		0.3217(5)	0.7244(5)	0.6755(4)	0.066(4)	0.067(4)	0.073(4)	-0.021(3)	-0.010(3)	0.021(3)
C(11)	4e		0.2013(5)	0.7805(4)	0.5097(3)	0.078(4)	0.057(4)	0.060(4)	0.003(3)	0.011(3)	0.014(3)
C(12)	4e		0.2933(7)	0.8165(6)	0.5027(5)	0.087(5)	0.096(6)	0.095(6)	-0.014(5)	0.026(4)	0.030(5)
C(13)	4e		0.3260(9)	0.8279(9)	0.4363(7)	0.111(8)	0.14(1)	0.14(1)	-0.001(7)	0.051(8)	0.046(8)
C(14)	4e		0.265(1)	0.8015(8)	0.3792(6)	0.16(1)	0.103(7)	0.103(8)	0.024(7)	0.061(8)	0.044(6)
C(15)	4e		0.1742(9)	0.7656(6)	0.3847(4)	0.16(1)	0.078(5)	0.067(5)	0.015(6)	0.019(5)	0.012(4)
C(16)	4e		0.1427(6)	0.7546(5)	0.4510(3)	0.103(6)	0.063(4)	0.058(4)	0.003(4)	0.009(4)	0.009(3)
C(17)	4e		0.0565(5)	0.8560(4)	0.5935(3)	0.069(4)	0.049(3)	0.046(3)	-0.001(3)	-0.001(3)	-0.002(2)
C(18)	4e		0.0829(6)	0.9490(5)	0.5901(4)	0.077(4)	0.062(4)	0.071(4)	-0.005(3)	0.001(3)	0.003(3)
C(19)	4e		0.0124(6)	1.0182(5)	0.5890(4)	0.101(6)	0.047(4)	0.082(5)	-0.003(4)	0.002(4)	0.003(3)
C(20)	4e		-0.0866(7)	0.9945(6)	0.5869(4)	0.094(6)	0.075(5)	0.076(5)	0.014(4)	0.002(4)	0.001(4)
C(21)	4e		-0.1145(6)	0.9023(5)	0.5893(4)	0.075(5)	0.078(5)	0.076(5)	0.001(4)	0.000(4)	-0.006(4)
C(22)	4e		-0.0434(5)	0.8334(5)	0.5926(4)	0.064(4)	0.065(4)	0.072(4)	-0.001(3)	0.005(3)	-0.008(3)
C(23)	4e		0.2403(5)	0.6322(5)	0.7932(3)	0.068(4)	0.059(3)	0.046(3)	0.006(3)	-0.012(3)	0.001(3)
C(24)	4e		0.1712(6)	0.5757(5)	0.8217(3)	0.089(5)	0.072(4)	0.049(3)	0.002(4)	0.002(3)	0.003(3)
C(25)	4e		0.1555(8)	0.5838(7)	0.8918(4)	0.119(7)	0.111(6)	0.059(4)	0.032(6)	0.008(5)	0.011(4)
C(26)	4e		0.2092(9)	0.6482(9)	0.9321(4)	0.125(8)	0.145(9)	0.055(5)	0.048(7)	-0.005(5)	-0.019(6)
C(27)	4e		0.2774(8)	0.7015(7)	0.9056(5)	0.110(7)	0.115(8)	0.080(6)	0.018(6)	-0.025(5)	-0.036(5)
C(28)	4e		0.2939(6)	0.6938(6)	0.8366(4)	0.094(5)	0.081(5)	0.074(5)	0.010(4)	-0.023(4)	-0.018(4)
C(29)	4e		0.3568(5)	0.5291(5)	0.7030(3)	0.059(3)	0.073(4)	0.044(3)	0.002(3)	0.005(3)	0.008(3)
C(30)	4e		0.3380(6)	0.4382(5)	0.6810(4)	0.071(4)	0.063(4)	0.093(5)	0.001(3)	0.000(4)	0.013(4)
C(31)	4e		0.4108(6)	0.3720(6)	0.6872(5)	0.080(5)	0.078(5)	0.105(6)	0.012(4)	0.005(5)	0.020(5)
C(32)	4e		0.5051(6)	0.3943(6)	0.7135(4)	0.079(5)	0.085(6)	0.081(5)	0.023(4)	0.019(4)	0.023(4)
C(33)	4e		0.5251(6)	0.4829(7)	0.7331(4)	0.062(4)	0.111(7)	0.078(5)	0.017(4)	0.013(4)	0.005(5)
C(34)	4e		0.4526(5)	0.5509(6)	0.7282(4)	0.062(4)	0.096(6)	0.072(4)	0.000(4)	0.005(3)	-0.016(4)
S(1)	4e	0.62(1)	0.5713(3)	0.8353(4)	0.6518(3)	0.071(3)	0.104(3)	0.139(4)	-0.032(2)	0.010(3)	-0.034(3)
F(1)	4e	0.62	0.7271(7)	0.878(1)	0.7260(6)	0.083(7)	0.28(2)	0.14(1)	-0.003(8)	-0.014(6)	0.01(1)
F(2)	4e	0.62	0.657(1)	0.9894(7)	0.6594(9)	0.23(2)	0.111(9)	0.26(2)	-0.06(1)	0.04(1)	0.06(1)
F(3)	4e	0.62	0.591(1)	0.946(1)	0.7536(7)	0.16(1)	0.26(2)	0.17(1)	-0.03(1)	0.07(1)	-0.10(1)
O(4)	4e	0.62	0.565(1)	0.7572(8)	0.7015(9)	0.11(1)	0.13(1)	0.26(2)	-0.062(9)	-0.01(1)	0.09(1)
O(5)	4e	0.62	0.4779(6)	0.8811(8)	0.6345(8)	0.070(7)	0.14(1)	0.19(1)	-0.021(6)	-0.039(8)	0.047(9)
O(6)	4e	0.62	0.632(1)	0.815(1)	0.5960(6)	0.18(2)	0.22(2)	0.11(1)	-0.06(1)	0.04(1)	-0.07(1)
C(35)	4e	0.62	0.6397(8)	0.9155(8)	0.7004(6)	0.09(1)	0.11(1)	0.11(1)	-0.026(9)	0.024(9)	-0.04(1)
S(1A)	4e	0.38	0.604(1)	0.800(1)	0.6538(8)	0.21(1)	0.21(2)	0.22(1)	-0.02(1)	0.13(1)	0.02(1)
F(1A)	4e	0.38	0.677(3)	0.946(2)	0.708(2)	0.33(4)	0.18(3)	0.20(3)	-0.12(3)	0.03(3)	-0.11(2)
F(2A)	4e	0.38	0.610(3)	0.958(2)	0.600(2)	0.38(5)	0.44(5)	0.40(5)	0.09(4)	0.03(4)	0.01(4)
F(3A)	4e	0.38	0.515(2)	0.945(2)	0.686(3)	0.35(4)	0.44(4)	0.35(4)	0.09(4)	0.08(4)	0.03(3)
O(4A)	4e	0.38	0.581(3)	0.766(2)	0.722(1)	0.25(4)	0.23(3)	0.13(2)	-0.04(3)	-0.06(2)	0.06(2)
O(5A)	4e	0.38	0.526(2)	0.782(2)	0.598(1)	0.11(2)	0.31(4)	0.18(2)	0.01(2)	-0.03(2)	0.03(2)
O(6A)	4e	0.38	0.703(1)	0.779(2)	0.637(2)	0.16(2)	0.15(2)	0.30(3)	-0.05(2)	0.06(2)	-0.02(2)
C(35A)	4e	0.38	0.602(2)	0.918(1)	0.662(1)	0.6(1)	0.6(1)	0.6(1)	0.00(2)	0.06(2)	-0.00(2)

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