

displacement parameters were set to 1.2 times (1.5 for methyl groups) the equivalent isotropic displacement parameters of the atoms to which the H atoms are bonded. For all methyl groups one torsion angle was refined. Compound (3) was refined as a racemic twin using the method of Flack (1983).

For all compounds, data collection: *DIF4* (Stoe & Cie, 1988a); cell refinement: *DIF4*; data reduction: *REDU4* (Stoe & Cie, 1988b); program(s) used to solve structures: *SHELX90* (Sheldrick, 1990); program(s) used to refine structures: *SHELXL93* (Sheldrick, 1993); molecular graphics: *SHELXTL-Plus* (Sheldrick, 1994); software used to prepare material for publication: *SHELXL93*.

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Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the IUCr (Reference: JZ1049). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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## A Cationic Rhenium–Platinum Complex: *fac*-Tricarbonylrhenium-bis[ $\mu$ -methylaminobis(difluorophosphine)]-triphenylphosphineplatinum Trifluoromethanesulfonate

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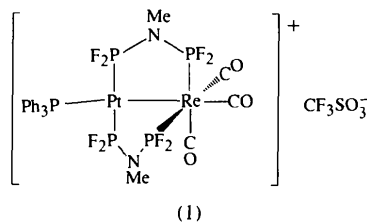
(Received 10 February 1995; accepted 16 May 1995)

### Abstract

The title compound, *fac*-tricarbonyl- $1\kappa^3C$ -bis[ $\mu$ -methylaminobis(difluorophosphine)- $1\kappa P:2\kappa P'$ ]triphenylphosphine- $2\kappa P$ -platinumrhenium (*Pt*—*Re*) trifluoromethanesulfonate, [PtRe(CO)<sub>3</sub>(CH<sub>3</sub>F<sub>4</sub>NP<sub>2</sub>)<sub>2</sub>(C<sub>18</sub>H<sub>15</sub>P)]·CF<sub>3</sub>SO<sub>3</sub>, exhibits distorted octahedral and square-planar coordination about the Re and Pt atoms, respectively. A Pt—Re single bond of length 2.818 (1) Å is present.

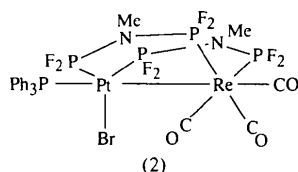
### Comment

Previous studies of the 'metalloligands' CpMCl{ $\eta^1$ -MeN(PF<sub>2</sub>)<sub>2</sub>}<sub>2</sub> (*M* = Fe, Ru) and *fac*-ReBr(CO)<sub>3</sub>( $\eta^1$ -L<sub>2</sub>)<sub>2</sub> [L<sub>2</sub> = MeN(PF<sub>2</sub>)<sub>2</sub>, CH<sub>2</sub>(PMe<sub>2</sub>)<sub>2</sub> (dmpm)] (Mague & Lin, 1992, 1994; Mague, 1994) have shown them to be useful for the directed synthesis of heterobimetallic complexes. To date, the reactions of these complexes have proven quite limited; in an attempt to increase reactivity, the weakly bound Br ligand in *fac*-Re(CO)<sub>3</sub>{ $\mu$ -MeN(PF<sub>2</sub>)<sub>2</sub>}<sub>2</sub>PtBr(PPh<sub>3</sub>), (2) (Mague & Lin, 1994), was removed by reacting with silver trifluoromethanesulfonate to give the title compound, (1). Little increase in the reactivity of (1) over its precursor was seen, suggesting that the trifluoromethanesulfonate ion might be coordinated in place of the Br atom. As spectroscopic data were inconclusive on this point, the structure of (1) was determined by X-ray analysis.

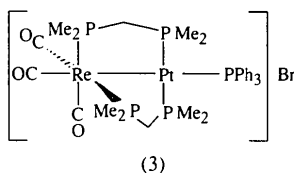


In the crystal form, (1) consists of well separated [Re(CO)<sub>3</sub>{ $\mu$ -MeN(PF<sub>2</sub>)<sub>2</sub>}<sub>2</sub>Pt{P(C<sub>6</sub>H<sub>5</sub>)<sub>3</sub>}]<sup>+</sup> cations and CF<sub>3</sub>SO<sub>3</sub><sup>-</sup> anions. Both ions have crystallographically imposed mirror symmetry and while the displacement

parameters for the axial carbonyl group (C2—O2) and the C atoms of the phenyl ring C10—C15 suggest slight disorder of these groups across the mirror, it is to a much lesser extent than was found in (2) (Mague & Lin, 1994).



Of particular note is the Pt—Re distance of 2.818 (1) Å which is considerably shorter than that in (2) [2.899 (1) Å; Mague & Lin, 1994] and the length of 2.868 (1) Å found in the close analogue [Re(CO)<sub>3</sub>(μ-dmpm)<sub>2</sub>Pt(PPh<sub>3</sub>)]Br, (3) (Mague, 1994). It is also shorter than those distances found for the unsupported Pt—Re bonds in CpRe(H)(CO)<sub>2</sub>Pt(H)(PPh<sub>3</sub>)<sub>2</sub> [2.838 (1) Å; Casey, Rutter & Haller, 1987] and *trans*-Pt(CO)<sub>2</sub>{Re(CO)<sub>5</sub>}<sub>2</sub> [2.831 (1) Å; Urbancic, Wilson & Shapley, 1984], and is thus considered to represent a Pt—Re single bond.



The coordination about the Re atom is roughly octahedral and little different from that found in (2) and (3). The distortions are primarily the result of the bridging ligands occupying *cis* positions on the Re atom but approximately *trans* positions on the Pt

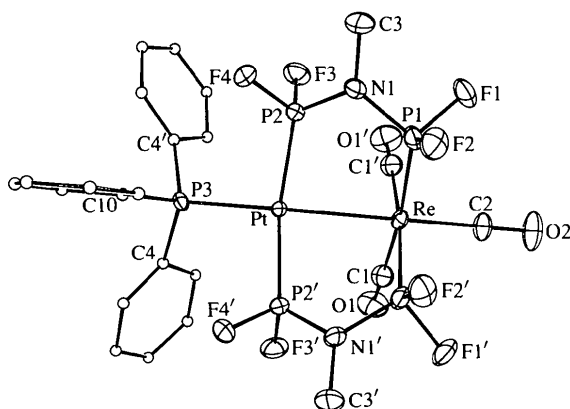


Fig. 1. Perspective view of the cation of (1). Non-H atoms are represented by displacement ellipsoids drawn at the 25% probability level except for the phenyl C atoms which are represented as circles for clarity. H atoms are omitted. Primed atoms are related to non-primed atoms by the crystallographic mirror. P1' is bonded to F1', F2' and N1'.

atom. This in turn results in the coordination about the Pt atom being severely distorted from square planar; this can be described as a 'tetrahedral' distortion. The values of the Re—Pt—P3 and P2—Pt—P2' angles are virtually identical to the corresponding values found in (3), but differ considerably from those in (2) [178.64 (6) and 145.08 (3)°, respectively]. Thus, removal of the Br ligand from (2) allows the triphenylphosphine ligand to swing toward the vacated site and the angle between the ends of the bridging ligands to open up.

## Experimental

Under nitrogen, 0.038 g (0.15 mmol) of Ag(CF<sub>3</sub>SO<sub>3</sub>) was added to a solution of 0.196 g (0.15 mmol) of Re(CO)<sub>3</sub>{MeN(PF<sub>2</sub>)<sub>2</sub>}<sub>2</sub>PtBr(PPh<sub>3</sub>) in 6 ml of dichloromethane. The mixture was stirred for 4 h at room temperature, filtered to remove precipitated AgBr and taken to dryness *in vacuo*. Recrystallization was from dichloromethane/diethyl ether at 263 K. A crystal of (1), obtained as above, was cut to size and mounted on a glass fibre with a coat of epoxy cement.

### Crystal data

[PtRe(CO)<sub>3</sub>(CH<sub>3</sub>F<sub>4</sub>NP<sub>2</sub>)<sub>2</sub>-  
(C<sub>18</sub>H<sub>15</sub>P)]CF<sub>3</sub>SO<sub>3</sub>  
M<sub>r</sub> = 1210.65  
Monoclinic  
P2<sub>1</sub>/m  
a = 11.274 (2) Å  
b = 14.2626 (9) Å  
c = 12.375 (1) Å  
β = 114.56 (1)°  
V = 1809.8 (7) Å<sup>3</sup>  
Z = 2  
D<sub>x</sub> = 2.22 Mg m<sup>-3</sup>

Mo Kα radiation  
λ = 0.71073 Å  
Cell parameters from 23  
reflections  
θ = 13–17°  
μ = 7.657 mm<sup>-1</sup>  
T = 293 K  
Block  
0.35 × 0.30 × 0.27 mm  
Yellow

### Data collection

Enraf–Nonius CAD-4  
diffractometer  
θ/2θ scans  
Absorption correction:  
ψ scan (North, Phillips  
& Mathews, 1968)  
T<sub>min</sub> = 0.548, T<sub>max</sub> =  
0.998  
3896 measured reflections  
3698 independent reflections

2976 observed reflections  
[I > 3σ(I)]  
R<sub>int</sub> = 0.035  
θ<sub>max</sub> = 25.98°  
h = 0 → 13  
k = 0 → 17  
l = -15 → 13  
2 standard reflections  
frequency: 120 min  
intensity decay: 5.9%

### Refinement

Refinement on F  
R = 0.028  
wR = 0.039  
S = 1.327  
2976 reflections  
253 parameters  
H-atom parameters not  
refined  
w = 4F<sub>o</sub><sup>2</sup>/[σ<sup>2</sup>(F<sub>o</sub><sup>2</sup>)  
+ 0.0016F<sub>o</sub><sup>4</sup>]

(Δ/σ)<sub>max</sub> = 0.014  
Δρ<sub>max</sub> = 1.34 e Å<sup>-3</sup>  
Δρ<sub>min</sub> = -0.22 e Å<sup>-3</sup>  
Extinction correction: none  
Atomic scattering factors  
from *International Tables*  
for X-ray Crystallography  
(1974)

Table 1. Fractional atomic coordinates and equivalent isotropic displacement parameters (Å<sup>2</sup>)
$$U_{eq} = (1/3)\sum_i \sum_j U_{ij} a_i^* a_j^* \mathbf{a}_i \cdot \mathbf{a}_j$$

	x	y	z	<i>U</i> <sub>eq</sub>
Pt	0.20512 (2)	1/4	0.27906 (2)	0.02835 (7)
Re	-0.06524 (3)	1/4	0.13588 (3)	0.03849 (9)
P1	-0.0768 (1)	0.1274 (1)	0.2547 (1)	0.0465 (4)
P2	0.1842 (1)	0.0978 (1)	0.2991 (1)	0.0396 (4)
P3	0.4303 (2)	1/4	0.3357 (2)	0.0324 (5)
F1	-0.1812 (4)	0.0509 (3)	0.1895 (4)	0.081 (1)
F2	-0.1171 (3)	0.1427 (3)	0.3588 (3)	0.074 (1)
F3	0.1948 (3)	0.0273 (3)	0.2095 (3)	0.068 (1)
F4	0.2927 (3)	0.0548 (3)	0.4110 (3)	0.062 (1)
O1	-0.0208 (6)	0.3929 (4)	-0.0343 (4)	0.090 (2)
O2	-0.3645 (7)	1/4	-0.0020 (7)	0.101 (3)
N1	0.0550 (4)	0.0612 (3)	0.3166 (4)	0.048 (1)
C1	-0.0375 (6)	0.3433 (5)	0.0307 (5)	0.055 (2)
C2	-0.2516 (8)	1/4	0.0465 (8)	0.060 (3)
C3	0.0542 (7)	-0.0305 (5)	0.3742 (7)	0.085 (3)
C4	0.4725 (5)	0.3536 (4)	0.2712 (4)	0.042 (1)
C5	0.5493 (7)	0.4230 (5)	0.3395 (6)	0.061 (2)
C6	0.5700 (7)	0.5045 (5)	0.2896 (7)	0.076 (3)
C7	0.5103 (7)	0.5177 (5)	0.1713 (6)	0.082 (2)
C8	0.4295 (9)	0.4515 (6)	0.1013 (6)	0.105 (3)
C9	0.4096 (8)	0.3696 (6)	0.1506 (6)	0.084 (3)
C10	0.5377 (7)	1/4	0.4919 (6)	0.036 (2)
C11	0.4819 (7)	1/4	0.5731 (6)	0.050 (2)
C12	0.5623 (8)	1/4	0.6933 (7)	0.071 (4)
C13	0.694 (1)	1/4	0.7308 (8)	0.070 (3)
C14	0.7504 (8)	1/4	0.6507 (8)	0.056 (3)
C15	0.6742 (7)	1/4	0.5327 (7)	0.048 (2)
S	0.8586 (2)	3/4	0.3777 (2)	0.0489 (6)
F5	0.6504 (6)	3/4	0.1749 (7)	0.113 (3)
F6	0.8094 (6)	0.8243 (4)	0.1717 (4)	0.127 (2)
O3	0.8138 (4)	0.8342 (4)	0.4103 (4)	0.081 (2)
O4	0.9936 (5)	3/4	0.4007 (6)	0.064 (2)
C16	0.779 (1)	3/4	0.218 (1)	0.076 (4)

Table 2. Selected geometric parameters (Å, °)

Pt—Re	2.818 (1)	Re—C1	1.975 (7)
Pt—P2	2.208 (1)	Re—C2	1.925 (9)
Pt—P3	2.336 (2)	O1—C1	1.144 (8)
Re—P1	2.323 (2)	O2—C2	1.16 (1)
Re—Pt—P2	86.13 (4)	P1—Re—P1'	97.62 (8)
Re—Pt—P3	160.96 (6)	P1—Re—C1	172.0 (2)
P2—Pt—P2'	158.77 (8)	P1—Re—C2	91.6 (2)
P2—Pt—P3	96.88 (4)	C1—Re—C2	93.2 (3)
Pt—Re—P1	86.15 (4)	Re—C1—O1	175.8 (6)
Pt—Re—C1	89.3 (2)	Re—C2—O2	177 (1)
Pt—Re—C2	176.6 (3)		

Symmetry code: (i) *x*,  $\frac{1}{2}$  - *y*, *z*.

General procedures for crystal orientation and cell determination and refinement have been published (Mague & Lloyd, 1989). The choice between the possible space groups *P2*<sub>1</sub> and *P2*<sub>1</sub>/*m* was made on the basis of intensity statistics and the successful refinement in the latter. Most H atoms were seen in a difference map near the end of the refinement and all were included as fixed contributions in calculated positions (C—H = 0.95 Å) with isotropic displacement parameters 20% larger than those of the attached C atoms, and updated periodically. The structure was solved by Patterson and Fourier methods.

Data collection: *CAD-4 Software* (Enraf-Nonius, 1989). Cell refinement: *CAD-4 Software*. Data reduction: *MolEN PROCESS* (Fair, 1990). Program(s) used to refine structure: *MolEN LSFM*. Molecular graphics: *ORTEPII* (Johnson, 1976). Software used to prepare material for publication: *MolEN CIF VAX*.

We thank the Chemistry Department of Tulane University for generous financial support of the X-ray diffraction facility.

Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the IUCr (Reference: CRI192). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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## Tris(1,2-dimethoxyethane)lithium 1,1,4,4-Tetraphenyl-1,3-butadiene Radical Anion

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## Abstract

The crystal structure of [Li(C<sub>4</sub>H<sub>10</sub>O<sub>2</sub>)<sub>3</sub>](C<sub>28</sub>H<sub>22</sub>) has been determined at 100 K. The structure of the radical anion displays a slightly distorted butadiene skeleton with reversed bond order compared to the neutral 1,1,4,4-tetraphenyl-1,3-butadiene molecule. The Li counterion is hexacoordinated by three dimethoxyethane molecules forming a solvent-separated ion pair.