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Di-μ-chlorido-bis[chlorido(η^5-pentamethylcyclopentadienyl)rhodium(III)] chloroform trisolvate

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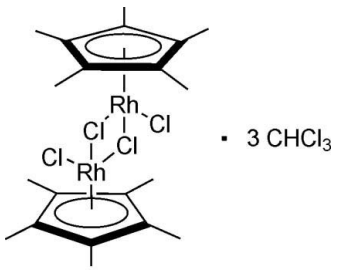
In the title dinuclear rhodium complex, $[Rh_2(C_{10}H_{15})_2Cl_4]\cdot 3CHCl_3$, there are two independent centrosymmetric dinuclear complexes per unit cell.

Comment

The title compound, (I), first synthesized by Kang *et al.*, (1969) and characterized by X-ray structure analysis at 295 K a few years later (Churchill *et al.*, 1977), has been crystallized with solvent molecules. Crystallization of (I) from chloroform gives rise not only to a different packing, but also to different geometric parameters for the dinuclear complex $[Rh(\eta^5\text{-}C_5Me_5)(\mu\text{-}Cl)Cl]_2$, (I).



(I)

In the chloroform trisolvate structure determined at 173 K, two symmetrically independent halves of the dinuclear complex are found per asymmetric unit. Each complex possesses a crystallographic inversion center at the mid-point of the Rh···Rh vector (Fig. 1). Interestingly, compared to the solvent-free structure reported by Churchill, the new determination including solvent molecules shows different structural parameters for the dinuclear rhodium complex. The Rh···Rh distances are shorter in (I) [3.6578 (5) and 3.6601 (5) Å] than in the solvent-free structure [3.7191 (6)Å]. The bridging Rh—Cl—Rh angles are smaller by about 2°, while the Rh—Cl distances remain the same. The Rh—C distances are slightly longer, the mean difference being, however, only 0.015 Å.

Weak interactions between the dinuclear rhodium complexes and the chloroform molecules are observed. The strongest involves the terminal atom Cl3 and atom C21 of a chloroform molecule, the C···Cl distance being 3.483 (4) Å, with a C—H···Cl angle of 155°.

Experimental

$[Rh(\eta^5\text{-}C_5Me_5)(\mu\text{-}Cl)Cl]_2$ was disolved in chloroform and crystals suitable for X-ray diffraction analysis were obtained after 3 d by slow evaporation of the chloroform solution.

Crystal data

$[Rh_2(C_{10}H_{15})_2Cl_4]\cdot 3CHCl_3$
M_r = 976.16
Monoclinic, $P2_1/c$
a = 16.2812 (8) Å
b = 16.7815 (6) Å
c = 13.8303 (7) Å
β = 106.733 (4)°
V = 3618.8 (3) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 1.89 mm^{-1}
T = 173 (2) K
0.30 × 0.15 × 0.15 mm

Data collection

Stoe IPDS2 diffractometer
Absorption correction: multi-scan (Blessing, 1995)
T_{min} = 0.699, T_{max} = 0.848
(expected range = 0.621–0.753)
46686 measured reflections
6449 independent reflections
4874 reflections with $I > 2\sigma(I)$
R_{int} = 0.038

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.027
$wR(F^2)$ = 0.061
S = 1.01
6449 reflections
400 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.56 e Å^{-3}
$\Delta\rho_{min}$ = −0.53 e Å^{-3}

H atoms were included in calculated positions (C—H = 0.96 Å) and refined as riding atoms with U_{iso}(H) = 1.5U_{eq}(carrier). The disordered C_5Me_5 groups (C11–C20), constrained to an ideal geometry, were located over two sites in a 65:35 ratio. The occupancy factor was first determined, before being fixed, by linking it to a free variable, while the isotropic displacement parameters were fixed at 0.05 Å^2.

Data collection: *X-AREA* (Stoe & Cie, 2000); cell refinement: *X-AREA*; data reduction: *X-RED32* (Stoe & Cie, 2000); program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *ORTEP-3* (Farrugia, 1997); software used to prepare material for publication: *SHELXL97*.

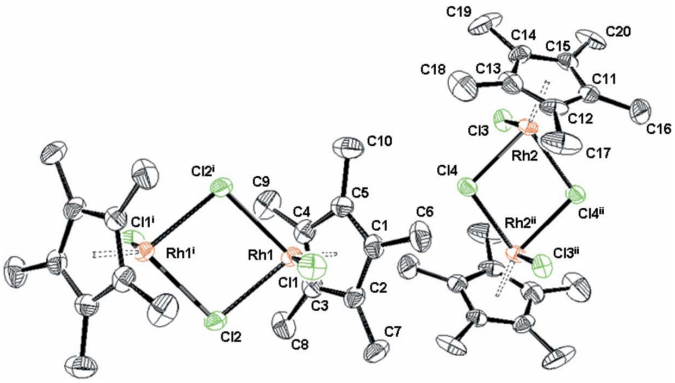


Figure 1
The molecular structure of (I), showing 50% displacement ellipsoids. H atoms and chloroform molecules have been omitted for clarity. Only the major component of the disordered C_5Me_5 ligand is shown. [Symmetry codes: (i) $-x, 1-y, 2-z$; (ii) $1-x, 1-y, 1-z$.]

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