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Carbonyl(3,7-dichlorotropolonato)(triphenylphosphine)rhodium(I)

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Key indicators

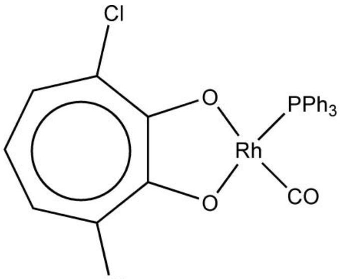
Single-crystal X-ray study
T = 100 K
Mean σ(C–C) = 0.004 Å
R factor = 0.027
wR factor = 0.061
Data-to-parameter ratio = 17.0

For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

The title compound, [Rh($C_7H_3Cl_2O_2$)($C_{18}H_{15}P$)(CO)], is a rhodium(I) derivative of chlorinated tropolonate. It crystallizes with a nearly square-planar geometry about the rhodium(I) metal centre. The most important bond distances and angles include: Rh—O(*trans* P) = 2.086 (2) Å, Rh—O(*trans* carbonyl) = 2.056 (2) Å, Rh—P = 2.2377 (8) Å, Rh—*C*(carbonyl) = 1.809 (2) Å, O—Rh—O = 76.60 (6)° and O—C—C—O = −3.0 (3)°.

Comment

Tropolonate complexes of rhodium(I) and palladium(II) have been investigated by our research group (Steyl & Roodt, 2005; Steyl, 2006). Although functionalization of the tropolonate group has been reported [NO_2: Steyl & Roodt (2004); Br_3: Roodt *et al.* (2003) and Steyl & Roodt (2006)], the synthetically challenging dihalogenated tropolones have been elusive. The title compound, (I), is presented as an example of a dihalogenated tropolonate unit coordinated to a rhodium(I) metal centre having carbonyl and triphenylphosphine ligands.



(I)

The title compound (Fig. 1) crystallizes with a slightly distorted square-planar geometry about the rhodium(I) metal centre. The Rh^I atom is elevated by 0.0097 (4) Å above the plane defined by the four coordinated atoms (O1, O2, P and C01). The Rh—O bond distances (Table 1) are comparable with those observed for [Rh(Trop)CO(PFcPh_2)] (Trop = 2-hydroxy-2,4,6-cycloheptatrienone and Fc = ferrocenyl) (Steyl *et al.*, 2001). The O—Rh—O bidentate bite angle [76.60 (6)°] in (I) is the smallest to date for four-coordinated rhodium(I) complexes containing a tropolonate ligand. A small torsional twist is observed for the tropolonate group (O1—C1—C2—O2, Table 1).

The crystal structure (Fig. 2) is stabilized by weak intermolecular C—H···O hydrogen bonds (Table 2) and van der Waals forces. The relatively short distance C1···C7($1 - x$, $1 - y$, $1 - z$) of 3.687 (3) Å between neighbouring tropolonate fragments may indicate a weak π–π interaction.

Experimental

The title complex was synthesized by the addition of PPh_3 (83 mg, 0.316 mmol) to an acetone solution (10 ml) of the [Rh(3,7-Cl_2Trop)$(CO)_2$] complex (100 mg, 0.287 mmol). On slow evaporation of the solvent, crystals suitable for X-ray crystallography were obtained (yield: 45 mg, 30%).

Crystal data

$[Rh(C_7H_3Cl_2O_2)(C_{18}H_{15}P)(CO)]$
$M_r = 583.18$
Monoclinic, $P2_1/n$
$a = 8.823$ (5) Å
$b = 17.662$ (5) Å
$c = 15.563$ (5) Å
$\beta = 106.450$ (5)°
$V = 2325.9$ (17) Å^3
$Z = 4$
$D_x = 1.665$ Mg m^{-3}
Mo $K\alpha$ radiation
$\mu = 1.06$ mm^{-1}
$T = 100$ (2) K
Plate, yellow
0.18 × 0.10 × 0.03 mm

Data collection

Bruker SMART 4K CCD area-detector diffractometer
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Bruker, 1998)
$T_{min} = 0.882$, $T_{max} = 0.97$
16578 measured reflections
5065 independent reflections
4322 reflections with $I > 2\sigma(I)$
$R_{int} = 0.031$
$\theta_{max} = 27.0°$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.027$
$wR(F^2) = 0.061$
$S = 1.04$
5065 reflections
298 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0234P)^2 + 1.6672P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.001$
$\Delta\rho_{max} = 0.71$ e Å^{-3}
$\Delta\rho_{min} = -0.45$ e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

Rh—C01	1.809 (2)	Cl3—C3	1.749 (2)
Rh—O1	2.0563 (16)	Cl7—C7	1.737 (2)
Rh—O2	2.0860 (16)	O01—C01	1.153 (3)
Rh—P	2.2377 (8)		
C01—Rh—O1	175.37 (8)	O1—Rh—P	97.25 (5)
C01—Rh—O2	99.15 (8)	O2—Rh—P	173.85 (4)
O1—Rh—O2	76.60 (6)	O01—C01—Rh	179.2 (2)
C01—Rh—P	87.00 (7)		
O2—C2—C1—O1	−3.0 (3)		

Table 2
Hydrogen-bond geometry (Å, °).

D—H⋯A	D—H	H⋯A	D⋯A	D—H⋯A
C13—H13⋯O2^{i}	0.93	2.38	3.301 (3)	171
C34—H34⋯O2^{ii}	0.93	2.54	3.387 (3)	152

Symmetry codes: (i) $x - \frac{1}{2}, -y + \frac{1}{2}, z - \frac{1}{2}$; (ii) $-x + \frac{1}{2}, y + \frac{1}{2}, -z + \frac{1}{2}$.

All H atoms were positioned geometrically and allowed to ride on their parent atoms, with $U_{iso}(H) = 1.2U_{eq}$(parent) and C—H = 0.93 Å.

Data collection: *APEX2* (Bruker, 2005); cell refinement: *SAINT-Plus* (Bruker, 2004); data reduction: *SAINT-Plus* and *XPREP* (Bruker, 2004); program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *DIAMOND* (Brandenburg, 2004); software used to prepare material for publication: *SHELXL97*.

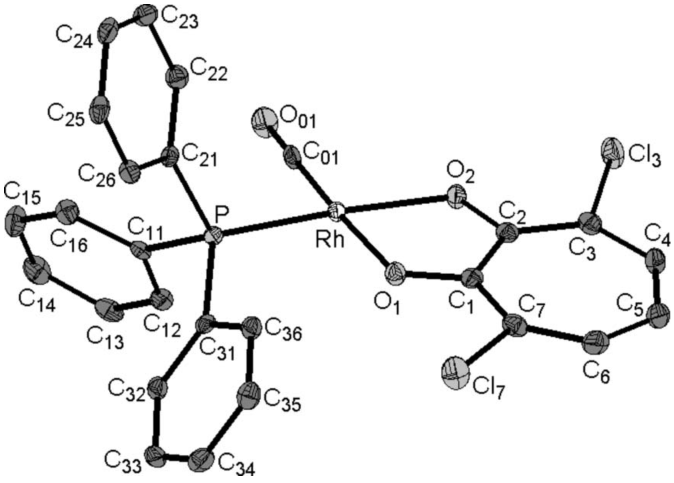


Figure 1
The molecular structure of the title compound, (I), showing the numbering scheme and displacement ellipsoids drawn at the 50% probability level. H atoms have been omitted for clarity.

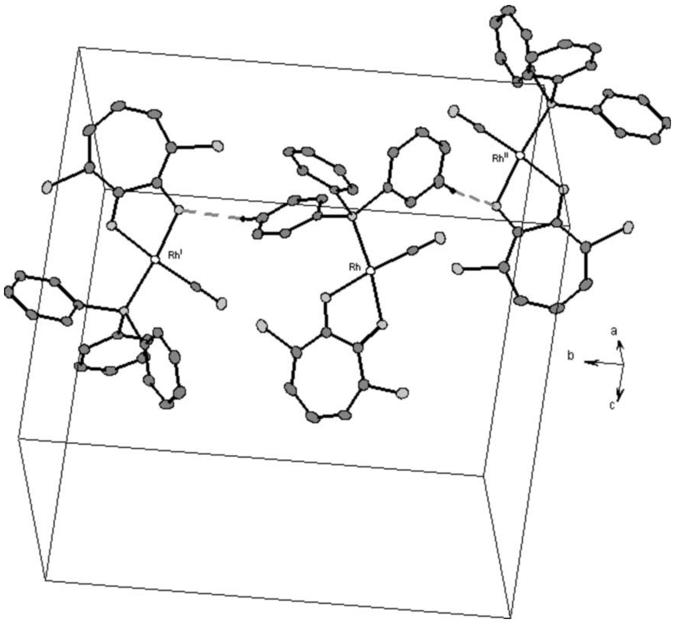


Figure 2
Part of the crystal packing showing the C—H⋯O interactions as dashed lines [symmetry codes: (i) $\frac{1}{2} - x, \frac{1}{2} + y, \frac{1}{2} - z$; (ii) $-\frac{1}{2} + x, \frac{1}{2} - y, -\frac{1}{2} + z$]. H atoms not involved in hydrogen bonding have been omitted for clarity.

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