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The crystal structure tetrakis(μ_2 -*o*-chlorobenzoato- $\kappa^2 O:O'$)-bis(methanol- $\kappa^1 O$) dirhodium(II), $C_{30}H_{24}Cl_4O_{10}Rh_2$

Table 1: Data collection and handling.

Crystal:	Blue needle
Size:	0.10 × 0.05 × 0.02 mm
Wavelength:	Cu K α radiation (1.54184 Å)
μ :	11.7 mm ⁻¹
Diffractometer, scan mode:	XtaLAB AFC12 (RINC), ω
$\theta_{\max}$, completeness:	74.0°, 96%
$N(hkl)_{\text{measured}}$, $N(hkl)_{\text{unique}}$, R_{int} :	7741, 3185, 0.047
Criterion for I_{obs} , $N(hkl)_{\text{gt}}$:	$I_{\text{obs}} > 2 \sigma(I_{\text{obs}})$, 2824
$N(\text{param})_{\text{refined}}$:	212
Programs:	CrysAlis ^{PRO} [1], SHELX [2–4], Diamond [5]

Table 2 contains the list of the atoms including atomic coordinates and displacement parameters.

Source of material

Rhodium(II) acetate was prepared by a method described in the literature [6]. The title compound was prepared by a reaction of rhodium(II) acetate with 2-chlorobenzoic acid. A solution of rhodium(II) acetate (0.100 g, 0.226 mmol), 2-chlorobenzoic acid (2.575 g, 0.0164 mol) in 5 mL of diethyleneglycoldimethylether was stirred for 6 h at 190 °C. After evaporation of the solvent, recrystallization from methanol gave blue needle crystals of the title compound (71.8% based on $[Rh_2(O_2CCH_3)_4]$).

Experimental details

Coordinates of hydrogen atoms were refined without any constraints or restraints. Their U_{iso} values were set to $1.2U_{\text{eq}}$ of the parent atoms.

Comment

Rhodium acetate is a rhodium homogeneous catalyst with a dinuclear structure. It has significant catalytic activity for cyclopropanation and hydroformylation of olefin, and has been widely used in basic chemical and pharmaceutical chemical fields. Based on the application of rhodium acetate, many new rhodium bicarbonate compounds have important industrial applications. In this paper, two

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Abstract

$C_{30}H_{24}Cl_4O_{10}Rh_2$, monoclinic, $P2_1/n$ (no. 14), $a = 7.5270(4)$ Å, $b = 12.3533(6)$ Å, $c = 17.4904(8)$ Å, $\beta = 91.780(4)^\circ$, $V = 1625.52(14)$ Å³, $Z = 2$, $R_{\text{gt}}(F) = 0.0458$, $wR_{\text{ref}}(F^2) = 0.1285$, $T = 150(1)$ K.

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The molecular structure is shown in the figure (Radii are of arbitrary size.). Table 1 contains crystallographic data and

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Table 2: Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²).

Atom	x	y	z	<i>U</i> _{iso} */ <i>U</i> _{eq}
Rh1	0.35549(4)	0.03850(2)	0.505925(16)	0.02537(15)
Cl1	0.6057(2)	0.20646(14)	0.28108(8)	0.0614(4)
Cl2	0.5212(2)	0.30023(10)	0.67455(8)	0.0516(4)
O1	0.4573(4)	0.1808(3)	0.46988(18)	0.0325(7)
O2	0.7310(4)	0.1097(3)	0.45753(17)	0.0301(7)
O3	0.4278(4)	0.0783(3)	0.61492(16)	0.0304(7)
O4	0.7028(4)	0.0071(3)	0.60403(17)	0.0306(7)
O5	0.0865(4)	0.1225(3)	0.5180(2)	0.0415(8)
H5	−0.011(2)	0.111(4)	0.493(2)	0.0622(12)*
C0	0.7259(9)	0.3750(5)	0.4690(3)	0.0522(14)
H0	0.7242(9)	0.3647(5)	0.5216(3)	0.0627(17)*
C1	0.6172(6)	0.1854(4)	0.4518(2)	0.0307(9)
C2	0.6776(6)	0.2917(4)	0.4209(3)	0.0348(10)
C3	0.6770(7)	0.3100(4)	0.3426(3)	0.0412(11)
C4	0.7268(7)	0.4096(5)	0.3130(3)	0.0496(14)
H4	0.7260(7)	0.4210(5)	0.2604(3)	0.0595(16)*
C5	0.7771(8)	0.4907(5)	0.3627(4)	0.0531(15)
H5a	0.8116(8)	0.5574(5)	0.3435(4)	0.0637(18)*
C6	0.7772(11)	0.4745(5)	0.4402(4)	0.0599(17)
H6	0.8115(11)	0.5300(5)	0.4734(4)	0.072(2)*
C7	0.5852(6)	0.0584(4)	0.6390(2)	0.0294(9)
C8	0.6330(6)	0.0987(4)	0.7177(3)	0.0324(9)
C9	0.7097(7)	0.0274(4)	0.7707(3)	0.0394(11)
H9	0.7374(7)	−0.0429(4)	0.7561(3)	0.0472(14)*
C10	0.7448(8)	0.0608(5)	0.8451(3)	0.0460(13)
H10	0.7953(8)	0.0126(5)	0.8803(3)	0.0551(15)*
C11	0.7057(7)	0.1653(5)	0.8678(3)	0.0446(12)
H11	0.7274(7)	0.1864(5)	0.9182(3)	0.0535(15)*
C12	0.6352(8)	0.2375(5)	0.8158(3)	0.0446(12)
H12	0.6109(8)	0.3082(5)	0.8306(3)	0.0535(15)*
C13	0.6000(7)	0.2046(4)	0.7405(3)	0.0370(11)
C14	0.0525(8)	0.1838(6)	0.5831(4)	0.0602(18)
H14a	−0.016(6)	0.247(2)	0.5686(5)	0.090(3)*
H14b	−0.013(6)	0.1408(14)	0.6182(13)	0.090(3)*
H14c	0.1630(8)	0.206(4)	0.6070(17)	0.090(3)*

rhodium(II) ions are bridged by four 2-carboxylato ligands in a syn-syn fashion to give a lantern-like dimer. The Rh1–Rh1a distance is 2.3892(6) Å, which is slightly shorter than those of other complexes of rhodium(II)

benzoate (2.405(1) Å for [Rh₂(O₂CC₆H₅)₄(DMSO)₂] · C₆H₅CH₃ [7], 2.402(1) Å for [Rh₂(O₂CC₆H₅)₄(py)₂] [8], and 2.391(1) Å for [Rh₂(O₂CC₆H₅)₄(pyz)]_n [9, 10]), but in the range typical for such dimers (2.35–2.45 Å). The coordination geometries around the rhodium(II) ions are both (crystallographically related) square-pyramidal. The axial positions are occupied by oxygen atoms of the methanol molecules with Rh–Oax distances of 2.291 Å (for Rh1–O5 and Rh1a–O5a).

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