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Key indicators

Single-crystal X-ray study T = 294 K Mean σ (C–C) = 0.008 Å R factor = 0.042 wR factor = 0.109 Data-to-parameter ratio = 17.5

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

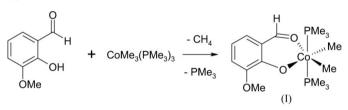
(2-Formyl-6-methoxyphenolato- $\kappa^2 O, O'$)*cis*-dimethyl-*trans*-bis(trimethylphosphine- κP)cobalt(III)

The title complex, $[Co(CH_3)_2(C_8H_7O_3)(C_3H_9P)_2]$, crystallizes with two molecules in the asymmetric unit. The Co^{III} centre has a distorted octahedral geometry in both molecules.

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Comment

The reaction of substituted salicylaldehydes with $[CoMe_3(PMe_3)_3]$ has been reported recently (Li *et al.*, 2005). 3-Methoxysalicylaldehyde reacts with $[CoMe_3(PMe_3)_3]$ by elimination of methane and trimethylphosphine (see scheme and *Experimental*), using both the phenolate and the keto functions to afford the hexacoordinated title Co^{III} complex, (I).



The molecular structure of (I) is shown in Fig. 1. The asymmetric unit contains two molecules with similar geometry. Co^{III} ions present an octahedral coordination geometry, with two equatorial *cis*-methyl groups (C15/C16 and C31/C32), two axial trimethylphosphines, and a bidentate 2-formyl-6-methoxyphenolate ligand. The *trans*-P-Co-P angles of 175.32 (6) and 175.52 (6)° indicate a slight distortion from an ideal octahedron. The substituted salicylaldehyde ligands have the expected Co-O coordination bond lengths, ranging from 2.000 (4) to 2.016 (3) Å, and the chelate rings are almost planar.

Experimental

Standard vacuum techniques were used for the manipulation of volatile and air-sensitive materials. 3-Methoxysalicylaldehyde (700 mg, 4.61 mmol) in diethyl ether (20 ml) was combined with $[CoMe_3(PMe_3)_3]$ (1550 mg, 4.67 mmol) in diethyl ether (20 ml) at 298 K. The mixture was stirred for 18 h. During this period, the solution turned red. The volatiles were removed *in vacuo* and the residue was extracted with *n*-pentane. Crystallization at 246 K afforded red crystals.

Crystal data

$$\begin{split} & \left[\text{Co}(\text{CH}_3)_2(\text{C}_8\text{H}_7\text{O}_3)(\text{C}_3\text{H}_9\text{P})_2 \right] \\ & M_r = 392.28 \\ & \text{Orthorhombic, } P2_12_12_1 \\ & a = 9.0948 \ (9) \text{ Å} \\ & b = 12.9794 \ (13) \text{ Å} \\ & c = 35.070 \ (3) \text{ Å} \\ & V = 4139.8 \ (7) \text{ Å}^3 \end{split}$$

Z = 8 D_x = 1.259 Mg m⁻³ Mo K α radiation μ = 0.99 mm⁻¹ T = 294 (2) K Block, red 0.30 × 0.24 × 0.15 mm

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metal-organic papers

Data collection

Bruker APEX-II CCD areadetector diffractometer φ and ω scans Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996) $T_{\min} = 0.755, T_{\max} = 0.866$

Refinement

Refinement on F^2 $R[F^2 > 2\sigma(F^2)] = 0.042$ $wR(F^2) = 0.109$ S = 0.977286 reflections 416 parameters H-atom parameters constrained 19456 measured reflections 7286 independent reflections 5070 reflections with $I > 2\sigma(I)$ $R_{\text{int}} = 0.048$ $\theta_{\text{max}} = 25.0^{\circ}$

$$\begin{split} &w = 1/[\sigma^2(F_o^2) + (0.0518P)^2 \\ &+ 1.3024P] \\ &where \ P = (F_o^2 + 2F_c^2)/3 \\ (\Delta/\sigma)_{max} = 0.028 \\ \Delta\rho_{max} = 0.40 \ e^{-3} \\ \Delta\rho_{min} = -0.37 \ e^{-3} \\ Absolute \ structure: \ Flack \ (1983), \\ &3165 \ Friedel \ pairs \\ Flack \ parameter: \ 0.50 \ (2) \end{split}$$

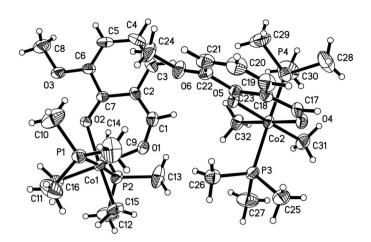


Figure 1

The asymmetric unit of (I), with displacement ellipsoids drawn at the 30% probability level.

The crystal was an inversion twin with equal contributions of the two components according to the Flack (1983) parameter refinement 0.50 (2). H atoms were placed in idealized positions and refined as riding on their parent atoms, with methyl C–H = 0.96 Å and aromatic C–H = 0.93 Å. Isotropic displacement parameters for H atoms were $U_{\rm iso}({\rm H}) = 1.2U_{\rm eq}({\rm aromatic C})$ and $U_{\rm iso}({\rm H}) = 1.5U_{\rm eq}({\rm meeq}({\rm methyl C})$. Rigid methyl groups were allowed to rotate about their C–C bonds to fit the electron density.

Data collection: *SMART* (Bruker, 1997); cell refinement: *SAINT* (Bruker, 1997); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *SHELXTL* (Bruker, 1997); software used to prepare material for publication: *SHELXTL*.

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