Acta Crystallographica Section E

## Structure Reports

Online
ISSN 1600-5368

## Jin Zhou, Shimin Fang, Hongjian Sun and Xiaoyan Li*

School of Chemistry and Chemical Engineering, Shandong University, Shanda Nanlu 27, Jinan 250100, People's Republic of China

Correspondence e-mail: xli63@sdu.edu.cn

## Key indicators

Single-crystal X-ray study
$T=294 \mathrm{~K}$
Mean $\sigma(\mathrm{C}-\mathrm{C})=0.008 \AA$
$R$ factor $=0.042$
$w R$ 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.
(C) 2006 International Union of Crystallography All rights reserved

## (2-Formyl-6-methoxyphenolato- $\kappa^{2} O, O^{\prime}$ )-cis-dimethyl-trans-bis(trimethylphosphine- $\kappa P$ )cobalt(III)

The title complex, $\left[\mathrm{Co}\left(\mathrm{CH}_{3}\right)_{2}\left(\mathrm{C}_{8} \mathrm{H}_{7} \mathrm{O}_{3}\right)\left(\mathrm{C}_{3} \mathrm{H}_{9} \mathrm{P}\right)_{2}\right]$, crystallizes with two molecules in the asymmetric unit. The $\mathrm{Co}^{\mathrm{III}}$ centre has a distorted octahedral geometry in both molecules.

## Comment

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

(I)

The molecular structure of (I) is shown in Fig. 1. The asymmetric unit contains two molecules with similar geometry. $\mathrm{Co}^{\mathrm{III}}$ 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-6methoxyphenolate ligand. The trans $-\mathrm{P}-\mathrm{Co}-\mathrm{P}$ angles of 175.32 (6) and 175.52 (6) ${ }^{\circ}$ indicate a slight distortion from an ideal octahedron. The substituted salicylaldehyde ligands have the expected $\mathrm{Co}-\mathrm{O}$ coordination bond lengths, ranging from 2.000 (4) to 2.016 (3) $\AA$, and the chelate rings are almost planar.

## Experimental

Standard vacuum techniques were used for the manipulation of volatile and air-sensitive materials. 3-Methoxysalicylaldehyde ( $700 \mathrm{mg}, 4.61 \mathrm{mmol}$ ) in diethyl ether ( 20 ml ) was combined with $\left[\mathrm{CoMe}_{3}\left(\mathrm{PMe}_{3}\right)_{3}\right](1550 \mathrm{mg}, 4.67 \mathrm{mmol})$ 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

| $\left[\mathrm{Co}\left(\mathrm{CH}_{3}\right)_{2}\left(\mathrm{C}_{8} \mathrm{H}_{7} \mathrm{O}_{3}\right)\left(\mathrm{C}_{3} \mathrm{H}_{9} \mathrm{P}\right)_{2}\right]$ | $Z=8$ |
| :--- | :--- |
| $M_{r}=392.28$ | $D_{x}=1.259 \mathrm{Mg} \mathrm{m}^{-3}$ |
| Orthorhombic, $P_{2} 2_{1} 2_{1} 2_{1}$ | Mo $K \alpha$ radiation |
| $a=9.0948(9) \AA \AA \AA$a <br> $b=12.9794(13) \AA$ | $\mu=0.99 \mathrm{~mm}^{-1}$ |
| $c=35.070(3) \AA$ | $T=294(2) \mathrm{K}$ |
| $V=4139.8(7) \AA^{3}$ | Block, red |
|  | $0.30 \times 0.24 \times 0.15 \mathrm{~mm}$ |

Received 9 October 2006
Accepted 15 November 2006

## Data collection

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

## Refinement

Refinement on $F^{2}$
$R\left[F^{2}>2 \sigma\left(F^{2}\right)\right]=0.042$
$w R\left(F^{2}\right)=0.109$
$S=0.97$
7286 reflections
416 parameters
H -atom parameters constrained

19456 measured reflections 7286 independent reflections 5070 reflections with $I>2 \sigma(I)$ $R_{\text {int }}=0.048$
$\theta_{\text {max }}=25.0^{\circ}$

$$
\begin{aligned}
& w=1 /\left[\sigma^{2}\left(F_{\mathrm{o}}^{2}\right)+(0.0518 P)^{2}\right. \\
& \quad+1.3024 P] \\
& \quad \text { where } P=\left(F_{\mathrm{o}}^{2}+2 F_{\mathrm{c}}^{2}\right) / 3 \\
& (\Delta / \sigma)_{\max }=0.028 \\
& \Delta \rho_{\max }=0.40 \mathrm{e} \AA^{-3} \\
& \Delta \rho_{\min }=-0.37 \mathrm{e}^{-3} \AA^{-3} \\
& \text { Absolute structure: Flack }(1983), \\
& \quad 3165 \text { Friedel pairs } \\
& \text { Flack parameter: } 0.50(2)
\end{aligned}
$$

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 $\mathrm{C}-\mathrm{H}=0.96 \AA$ and aromatic $\mathrm{C}-\mathrm{H}=0.93 \AA$. Isotropic displacement parameters for H atoms were $U_{\text {iso }}(\mathrm{H})=1.2 U_{\text {eq }}($ aromatic C $)$ and $U_{\text {iso }}(\mathrm{H})=1.5 U_{\mathrm{eq}}(\mathrm{me}-$ eq(methyl C). Rigid methyl groups were allowed to rotate about their $\mathrm{C}-\mathrm{C}$ 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.

Financial support of this work by the Excellent Young Teachers Program of MOE, People's Republic of China, the


Figure 1
The asymmetric unit of (I), with displacement ellipsoids drawn at the $30 \%$ probability level.

Scientific Research Foundation for Returned Overseas Chinese Scholars/State Education Ministry, and the Natural Science Foundation of Shandong University for Young Scientists is gratefully acknowledged.

## References

Bruker (1997). SMART (Version 5.6), SAINT (Version 5.06A) and SHELXTL (Version 6.12). Bruker AXS Inc., Madison, Wisconsin, USA.
Flack, H. D. (1983). Acta Cryst. A39, 876-881.
Li, X., Sun, H., Brand, A. \& Klein, H.-F. (2005). Inorg. Chim. Acta, 358, 33293333.

Sheldrick, G. M. (1996). SADABS. University of Göttingen, Germany.
Sheldrick, G. M. (1997). SHELXS97 and SHELXL97. University of Göttingen, Germany.

