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mer-(2-Formylatocyclohexen-1-olato-κ^2C^2,O^1)-iodotris(trimethylphosphine-κP)cobalt(III)

Fengli Yu, Shuli Dong, Jin Zhou, Aichen Wang, Hongjian Sun and Xiaoyan Li*

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

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

Key indicators

Single-crystal X-ray study
T = 293 K
Mean σ(C–C) = 0.011 Å
Disorder in main residue
R factor = 0.053
wR factor = 0.152
Data-to-parameter ratio = 21.7

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

In the title compound, $[CoI(C_7H_8O_2)(C_3H_9P)_3]$, the cobalt center is in a distorted octahedral geometry.

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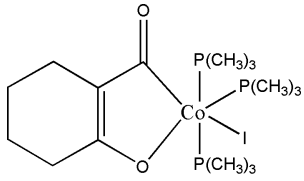
Comment

In 1998, Klein and co-workers reported some novel acyl–hydrido–cobalt(III) complexes, which are stabilized through 2-acylphenolato-chelating ligands and with the support of trimethylphosphine (Klein *et al.*, 1998*a*). They can react with acetic acid, hydrogen halides, substituted salicylaldehyde, substituted malondialdhyde, alkyl halides, perchloric acid and 2-nitrophenol. (Klein *et al.*, 1998*b*, 2003). In addition, insertion of phenylethyne into a Co—H bond of these acyl–hydrido–phenolato–cobalt(III) complexes was found and afforded vinyl–cobalt(III) complexes (Klein *et al.*, 2000).



(I)

The title compound, (I), was synthesized by the reported method of Klein *et al.* (1998*b*). Single crystals suitable for X-ray diffraction analysis were obtained. A view of the molecular structure is given in Fig. 1. The cobalt center is in a distorted octahedral geometry. The Co—I distance of 2.715 (1) Å is relatively long due to the strong *trans* influence of the acyl group. The cyclohexene ring in the structure showed a twofold positional disorder.

Experimental

Standard techniques were used in manipulations of volatile and air-sensitive material. The title compound was synthesized by the reaction of *mer*-(1-carbonyl-2-oxocyclohexenediyl)hydridotris(trimethylphosphine)cobalt(III) (1 g, 2.42 mmol) with iodomethane (450 mg, 3.17 mmol) in diethyl ether (80 ml) for 18 h at room temperature. Brown crystals suitable for X-ray diffraction analysis were obtained by crystallization at 269 K from diethyl ether.

Crystal data

$[CoI(C_7H_8O_2)(C_3H_9P)_3]$
M_r = 538.18
Orthorhombic, $P2_12_12_1$
a = 10.184 (2) Å
b = 12.182 (2) Å
c = 18.987 (3) Å
V = 2355.5 (7) Å^3
Z = 4
D_x = 1.518 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 3854 reflections
θ = 2.2–24.3°
μ = 2.25 mm^{-1}
T = 293 (2) K
Block, brown
0.26 × 0.18 × 0.16 mm

Data collection

Bruker SMART CCD area-detector diffractometer	4800 independent reflections
φ and ω scans	3421 reflections with $I > 2\sigma(I)$
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)	$R_{int} = 0.054$
$T_{min} = 0.546$, $T_{max} = 0.698$	$\theta_{max} = 26.4^\circ$
13537 measured reflections	$h = -5 \rightarrow 12$
	$k = -15 \rightarrow 15$
	$l = -23 \rightarrow 23$

Refinement

Refinement on F^2	$w = 1/[\sigma^2(F_o^2) + (0.0646P)^2 + 5.7779P]$ where $P = (F_o^2 + 2F_c^2)/3$
$R[F^2 > 2\sigma(F^2)] = 0.053$	$(\Delta/\sigma)_{max} = 0.001$
$wR(F^2) = 0.152$	$\Delta\rho_{max} = 1.01$ e Å^{-3}
$S = 1.08$	$\Delta\rho_{min} = -1.20$ e Å^{-3}
4800 reflections	Absolute structure: Flack (1983), 2071 Friedel pairs
221 parameters	Flack parameter = −0.08 (4)
H-atom parameters constrained	

H atoms were placed in idealized positions (C—H = 0.96 Å) with fixed isotropic displacement parameters and allowed to ride on their parent C atoms. The cyclohexene ring is disordered over two positions, with occupancy factors of 0.567 (16) and 0.433 (16). The C—C distance of the disordered ring was restrained to 1.52 Å. The highest peak in the difference map was 0.52 Å from atom Co1 and the depest hole was 0.70 Å from I1.

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* (Sheldrick, 2001); software used to prepare material for publication: *SHELXTL*.

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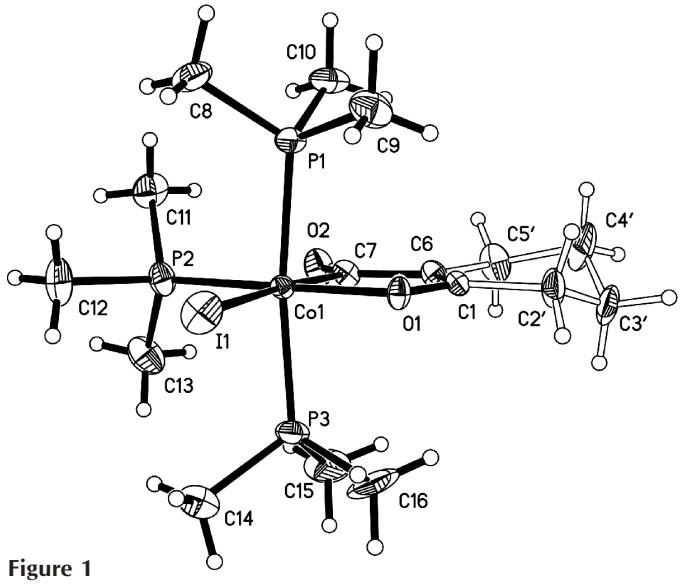

Figure 1
The molecular structure of the title complex. Displacement ellipsoids are drawn at the 50% probability level. Only one component of the disorder is shown.

References

Bruker (1997). *SMART* (Version 5.6) and *SAINT* (Version 5.06A). Bruker AXS Inc., Madison, Wisconsin, USA.
Flack, H. D. (1983). *Acta Cryst.* A**39**, 876–881.
Klein, H.-F., Haller, S., Sun, H., Li, X. Y., Jung, T., Röhr, C., Flörke, U. & Haupt, H.-J. (1998*a*). *Z. Naturforsch. Teil B*, **53**, 587–598.
Klein, H.-F., Haller, S., Sun, H., Li, X. Y., Jung, T., Röhr, C., Flörke, U. & Haupt, H.-J. (1998*b*). *Z. Naturforsch. Teil B*, **53**, 856–864.
Klein, H.-F., Li, X. Y., Flörke, U. & Haupt, H.-J. (2000) *Z. Naturforsch. Teil B*, **55**, 707–717.
Klein, H.-F., Li, X. Y., Flörke, U. &Haupt, H.-J. (2003). *Inorg. Chim. Acta*, **342**, 179–184.
Sheldrick, G. M. (1996). *SADABS*. University of Göttingen, Germany.
Sheldrick, G. M. (1997). *SHELXS97* and *SHELXL97*. University of Göttingen, Germany.
Sheldrick, G. M. (2001). *SHELXTL*. Version 6.12. Bruker AXS Inc., Madison, Wisconsin, USA.