

trans-(Methanol)(methyldiphenylphosphine)bis(pentane-2,4-dionato)cobalt(III) hexafluorophosphate hydrate

Takayoshi Suzuki

Department of Chemistry, Graduate School of Science, Osaka University, Toyonaka 560-0043, Japan

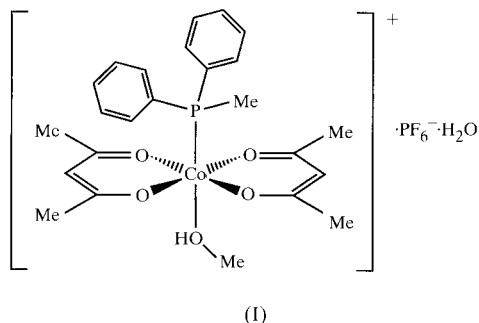
Correspondence e-mail: suzuki@chem.sci.osaka-u.ac.jp

Received 2 February 2000

Accepted 23 February 2000

Data validation number: IUC0000049

The title compound $[\text{Co}(\text{C}_5\text{H}_7\text{O}_2)_2(\text{C}_{13}\text{H}_{13}\text{P})(\text{CH}_4\text{O})]\text{PF}_6\cdot\text{H}_2\text{O}$, (I), which was converted from *trans*- $[\text{Co}(\text{acac})_2(\text{PMePh}_2)(\text{H}_2\text{O})]\text{PF}_6$ (acac is pentane-2,4-dionato) by recrystallization from aqueous methanol, has been confirmed as have a coordinated methanol ligand. The molecular structure of the complex cation, *trans*- $[\text{Co}(\text{acac})_2(\text{PMePh}_2)(\text{MeOH})]^+$, is similar to that of the above aqua complex found in the ClO_4 salt [Kashiwabara *et al.* (1995). *Bull. Chem. Soc. Jpn.*, **68**, 883–888]. The Co–O bond length for the coordinated methanol is 2.059 (3) Å. There is an intermolecular hydrogen bond between the OH group of the coordinated methanol and one of the O atoms of the acac ligands in an adjacent complex cation $[\text{O5}\cdots\text{O3}' = 2.914 (4) \text{ \AA}]$, giving a centrosymmetric dimeric dicationic complex.



Comment

This is, to my knowledge, the first X-ray crystallographic study of a non-organometallic cobalt(III) complex bearing a methanol ligand, although two organo-cobaloxime derivatives with a coordinated methanol ligand have been reported (Flohre *et al.*, 1978; Dreos *et al.*, 1995).

Experimental

The corresponding aqua complex, *trans*- $[\text{Co}(\text{acac})_2(\text{PMePh}_2)(\text{H}_2\text{O})]\text{PF}_6$, was prepared according to the literature method of Kashiwabara *et al.* (1982). The aqua complex was recrystallized from aqueous methanol, depositing purple prismatic crystals of the title methanol complex.

Crystal data

$[\text{Co}(\text{C}_5\text{H}_7\text{O}_2)_2(\text{C}_{13}\text{H}_{13}\text{P})(\text{CH}_4\text{O})]\text{PF}_6\cdot\text{H}_2\text{O}$
 $M_r = 652.37$
 Triclinic, $P\bar{1}$
 $a = 10.904 (3) \text{ \AA}$
 $b = 13.184 (4) \text{ \AA}$
 $c = 10.352 (2) \text{ \AA}$
 $\alpha = 99.74 (2)^\circ$
 $\beta = 97.24 (2)^\circ$
 $\gamma = 80.06 (2)^\circ$
 $V = 1437.4 (6) \text{ \AA}^3$

$Z = 2$
 $D_x = 1.507 \text{ Mg m}^{-3}$
 Mo $K\alpha$ radiation
 Cell parameters from 24 reflections
 $\theta = 14.8\text{--}15.0^\circ$
 $\mu = 0.784 \text{ mm}^{-1}$
 $T = 293 (2) \text{ K}$
 Prismatic, purple
 $0.50 \times 0.25 \times 0.20 \text{ mm}$

Data collection

Rigaku AFC-7R diffractometer
 ω - 2θ scans
 Absorption correction: ψ scan (North *et al.*, 1968)
 $T_{\text{min}} = 0.769$, $T_{\text{max}} = 0.855$
 8835 measured reflections
 8405 independent reflections
 3541 reflections with $I > 2\sigma(I)$

$R_{\text{int}} = 0.032$
 $\theta_{\text{max}} = 30.03^\circ$
 $h = -15 \rightarrow 15$
 $k = -18 \rightarrow 18$
 $l = -14 \rightarrow 0$
 3 standard reflections every 150 reflections intensity decay: 1.02%

Refinement

Refinement on F^2
 $R(F) = 0.055$
 $wR(F^2) = 0.167$
 $S = 0.965$
 8405 reflections
 396 parameters
 H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.0481P)^2 + 2.2359P]$
 where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{\text{max}} = 0.003$
 $\Delta\rho_{\text{max}} = 0.627 \text{ e \AA}^{-3}$
 $\Delta\rho_{\text{min}} = -0.817 \text{ e \AA}^{-3}$

Table 1

Hydrogen-bonding geometry (Å, °).

$D\text{---}H\cdots A$	$D\text{---}H$	$H\cdots A$	$D\cdots A$	$D\text{---}H\cdots A$
$\text{O5---H5}\cdots\text{O3}^i$	0.82	2.10	2.914 (4)	173

Symmetry codes: (i) $1 - x, -y, 1 - z$.

There were positional disorders for four equatorial F atoms (F2, F3, F4 and F5) of the PF_6^- anion over two sites, both of which corresponded to a regular octahedral arrangement with two axial F atoms (F1 and F6). The population of these disordered atoms were assumed to be 0.5. The O atom of water of crystallization was treated as two partial-occupancy isotropic O atoms (O61 and O62). H atoms bound to the O atom were not included in the calculation. All other H atoms were included in the structural calculation by means of a riding model.

Data collection: *Rigaku/AFC Diffractometer Control Software* (Rigaku, 1985); cell refinement: *Rigaku/AFC Diffractometer Control Software*; data reduction: *teXsan* (Molecular Structure Corporation & Rigaku, 1999); program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); software used to prepare material for publication: *SHELXL97*.

The author thanks Professors Kazuo Kashiwabara (Nagoya University) and Sumio Kaizaki (Osaka University) for valuable discussions.

References

- Dreos, R., Tazher, G., Vuano, S., Asaro, F., Pellizer, G., Nardin, G., Randaccio, L. & Geremia, S. (1995). *J. Organomet. Chem.* **505**, 135–138.
- Flohr, H., Pannhorat, W. & Retey, J. (1978). *Helv. Chim. Acta*, **61**, 1565–1587.
- Kashiwabara, K., Katoh, K., Ohishi, T., Fujita, J. & Shibata, M. (1982). *Bull. Chem. Soc. Jpn.* **55**, 149–155.
- Kashiwabara, K., Kita, M., Masuda, H., Kurachi, S. & Ohba, S. (1995). *Bull. Chem. Soc. Jpn.* **68**, 883–888.
- Molecular Structure Corporation & Rigaku (1999). *teXsan*. Version 1.10. MSC, 3200 Research Forest Drive, The Woodlands, TX 77381, USA, and Rigaku Co. Ltd, Akishima, Tokyo, Japan.
- North, A. C. T., Phillips, D. C. & Mathews, F. S. (1968). *Acta Cryst.* **A24**, 351–359.
- Rigaku (1985). *Rigaku/AFC Diffractometer Control Software*. Rigaku Co. Ltd, Akishima, Tokyo, Japan.