

Bis(2-hydroxybenzotrile- κN)dimethyl-bis(trimethylphosphine- κP)cobalt(III) iodide

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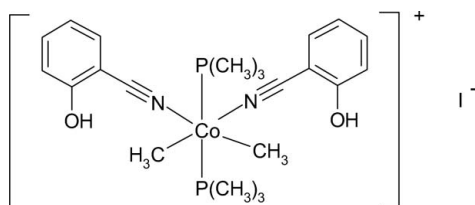
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Key indicators: single-crystal X-ray study; $T = 294$ K; mean $\sigma(C-C) = 0.008$ Å; R factor = 0.039; wR factor = 0.101; data-to-parameter ratio = 20.0.

In the title compound, $[Co(CH_3)_2(C_7H_5NO)_2(C_3H_9P)_2]I$, the Co^{III} atom lies in the centre of a distorted octahedron. The central Co^{III} atom is coordinated by the N atoms of two 2-hydroxybenzotrile ligands, which are positioned in the equatorial plane. The cation lies on a mirror plane (passing through Co and the phosphine ligands) and has approximate C_{2v} symmetry except for the OH groups. The anion also lies on a mirror plane.

Related literature

For related literature, see: Daran *et al.* (1984); Duff *et al.* (1990); Pozdeeva *et al.* (1983); Wang *et al.* (2006).



Experimental

Crystal data

$[Co(CH_3)_2(C_7H_5NO)_2(C_3H_9P)_2]I$
 $M_r = 606.28$
 Orthorhombic, $Pnma$
 $a = 13.749$ (6) Å
 $b = 13.817$ (6) Å
 $c = 14.771$ (7) Å
 $V = 2806$ (2) Å³
 $Z = 4$

Mo $K\alpha$ radiation
 $\mu = 1.84$ mm⁻¹

$T = 294$ (2) K
 $0.22 \times 0.18 \times 0.16$ mm

Data collection

Bruker SMART CCD area-detector diffractometer
 Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
 $T_{min} = 0.687$, $T_{max} = 0.757$
 15120 measured reflections
 2980 independent reflections
 1677 reflections with $I > 2\sigma(I)$
 $R_{int} = 0.067$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.039$
 $wR(F^2) = 0.102$
 $S = 0.99$
 2980 reflections
 149 parameters
 H atoms treated by a mixture of independent and constrained refinement
 $\Delta\rho_{max} = 0.73$ e Å⁻³
 $\Delta\rho_{min} = -0.46$ e Å⁻³

Table 1
 Hydrogen-bond geometry (Å, °).

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
$O-H1\cdots I$	0.88 (5)	2.62 (5)	3.490 (4)	175 (4)

Data collection: *SMART* (Bruker, 1997); cell refinement: *SAINTE* (Bruker, 1997); data reduction: *SAINTE*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1990); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *ORTEP-3* (Farrugia, 1997); software used to prepare material for publication: *SHELXTL* (Sheldrick, 2001).

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: LX2017).

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supplementary materials

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Bis(2-hydroxybenzointrile- κN)dimethylbis(trimethylphosphine- κP)cobalt(III) iodide

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Comment

Many organometallic nitrile complexes were synthesized. Pozdeeva and co-workers prepared the palladium(II) complexes with benzonitrile ligands in an aprotic medium and studied electrochemical reduction of these compounds (Pozdeeva *et al.*, 1983). Wang and co-workers reported the synthesis and the crystal structure of nickel(II) complex with 2-hydroxybenzointrile. (Wang *et al.*, 2006).

The title compound, $[\text{Co}(\text{CH}_3)_2(\text{C}_3\text{H}_9\text{P})_2(\text{C}_7\text{H}_5\text{NO})_2]\text{I}$, was synthesized by the reaction of (dimethyl)tris(trimethylphosphine)cobalt(III) iodide with 2-hydroxybenzointrile. The Co^{III} atom is hexa-coordinated by two N atoms of *cis*-(2-hydroxybenzointrile) ligands and two *cis*-methyl groups and two axial *trans*-trimethylphosphine ligands, and is in the center of distorted octahedral configuration (Fig. 1). The title compound has $C_{2v}(mm2)$ symmetry. The angular deviation of $\text{P1}-\text{Co}-\text{P2} = 170.53(8)^\circ$ from 180° may result from the steric hindrance of equatorial 2-hydroxybenzointrile ligands. The C—N distance of 1.135(5) Å is relatively long due to the coordination of the N atom to the Co atom. The Co—N distance of 1.956(4) Å is longer than the Ni—N distance of 1.858(5) Å in nickel(II) complex with 2-hydroxybenzointrile. (Wang *et al.*, 2006).

Experimental

All air-sensitive and volatile materials were handled *in vacuo* or under argon atmosphere using standard Schlenk techniques. The title compound was synthesized through the reaction of (dimethyl)tris(trimethylphosphine)cobalt(III) iodide (0.53 g, 1.17 mmol) with 2-hydroxybenzointrile (0.28 g, 2.36 mmol) in diethyl ether (80 ml) for 18 h at room temperature. Yellow crystals suitable for X-ray diffraction analysis were obtained from the solution of the title compound in diethyl ether at 253 K.

Refinement

The O bond H atom is positioned in the different Fourier maps and freely refined. Other all H atoms were positioned geometrically and refined using a riding model, with C—H = 0.93 Å for aromatic H atoms and 0.96 Å for methyl H atoms, respectively, and with $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{C})$ for aromatic H atoms and 1.5 $U_{\text{eq}}(\text{C})$ for methyl H atoms. The highest peak in the difference map is 0.85 Å from Co and the largest hole is 1.70 Å from H8C.

Figures

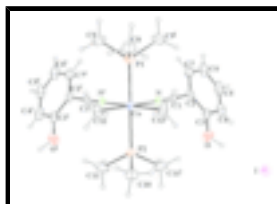


Fig. 1. The molecular structure of the title complex, shown with 50% probability displacement ellipsoids. [Symmetry code: (i) $x, 3/2 - y, z$.]

Bis(2-hydroxybenzotrile- κN)dimethylbis(trimethylphosphine- κP)cobalt(III) iodide

Crystal data

$[\text{Co}(\text{CH}_3)_2(\text{C}_7\text{H}_5\text{NO})_2(\text{C}_3\text{H}_9\text{P})_2]\text{I}$	$F_{000} = 1224$
$M_r = 606.28$	$D_x = 1.435 \text{ Mg m}^{-3}$
Orthorhombic, $Pnma$	Mo $K\alpha$ radiation
Hall symbol: -p 2ac 2n	$\lambda = 0.71073 \text{ \AA}$
$a = 13.749 (6) \text{ \AA}$	Cell parameters from 2687 reflections
$b = 13.817 (6) \text{ \AA}$	$\theta = 2.5\text{--}22.1^\circ$
$c = 14.771 (7) \text{ \AA}$	$\mu = 1.84 \text{ mm}^{-1}$
$V = 2806 (2) \text{ \AA}^3$	$T = 294 (2) \text{ K}$
$Z = 4$	Block, yellow
	$0.22 \times 0.18 \times 0.16 \text{ mm}$

Data collection

Bruker SMART CCD area-detector diffractometer	2980 independent reflections
Radiation source: fine-focus sealed tube	1677 reflections with $I > 2\sigma(I)$
Monochromator: graphite	$R_{\text{int}} = 0.067$
Detector resolution: 10 pixels mm^{-1}	$\theta_{\text{max}} = 26.4^\circ$
$T = 294(2) \text{ K}$	$\theta_{\text{min}} = 2.0^\circ$
φ and ω scans	$h = -17 \rightarrow 14$
Absorption correction: multi-scan (SADABS; Sheldrick, 1996)	$k = -16 \rightarrow 17$
$T_{\text{min}} = 0.687$, $T_{\text{max}} = 0.757$	$l = -15 \rightarrow 18$
15120 measured reflections	

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)] = 0.039$	H atoms treated by a mixture of independent and constrained refinement
$wR(F^2) = 0.102$	$w = 1/[\sigma^2(F_o^2) + (0.0371P)^2 + 2.8161P]$
$S = 0.99$	where $P = (F_o^2 + 2F_c^2)/3$
2980 reflections	$(\Delta/\sigma)_{\text{max}} < 0.001$
149 parameters	$\Delta\rho_{\text{max}} = 0.73 \text{ e \AA}^{-3}$
Primary atom site location: structure-invariant direct methods	$\Delta\rho_{\text{min}} = -0.46 \text{ e \AA}^{-3}$
	Extinction correction: none

Special details

Geometry. All e.s.d.'s (except the e.s.d. in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell e.s.d.'s are taken into account individually in the estimation of e.s.d.'s in distances, angles and torsion angles; correlations between e.s.d.'s in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell e.s.d.'s is used for estimating e.s.d.'s involving l.s. planes.

Refinement. Refinement of F^2 against ALL reflections. The weighted R -factor wR and goodness of fit S are based on F^2 , conventional R -factors R are based on F , with F set to zero for negative F^2 . The threshold expression of $F^2 > \sigma(F^2)$ is used only for calculating R -factors(gt) *etc.* and is not relevant to the choice of reflections for refinement. R -factors based on F^2 are statistically about twice as large as those based on F , and R -factors based on ALL data will be even larger.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (\AA^2)

	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{iso}}^*/U_{\text{eq}}$	Occ. (<1)
Co	0.51637 (6)	0.7500	0.12124 (5)	0.0395 (2)	
I	0.35233 (4)	0.2500	0.38891 (3)	0.0675 (2)	
P1	0.52454 (12)	0.7500	-0.02964 (11)	0.0497 (4)	
P2	0.53498 (15)	0.7500	0.27142 (12)	0.0614 (5)	
N	0.4156 (3)	0.6501 (3)	0.1238 (2)	0.0441 (9)	
O	0.3357 (3)	0.4629 (3)	0.2627 (3)	0.0831 (13)	
H1	0.338 (3)	0.412 (4)	0.298 (3)	0.071 (17)*	
C1	0.3565 (3)	0.5930 (3)	0.1243 (3)	0.0451 (10)	
C2	0.2783 (3)	0.5229 (3)	0.1237 (3)	0.0484 (11)	
C3	0.2679 (4)	0.4589 (3)	0.1967 (4)	0.0570 (13)	
C4	0.1891 (4)	0.3960 (4)	0.1970 (4)	0.0737 (16)	
H4	0.1798	0.3543	0.2456	0.088*	
C5	0.1248 (4)	0.3953 (4)	0.1259 (5)	0.087 (2)	
H5	0.0723	0.3528	0.1269	0.104*	
C6	0.1366 (4)	0.4564 (4)	0.0530 (5)	0.0834 (18)	
H6	0.0932	0.4541	0.0049	0.100*	
C7	0.2130 (4)	0.5209 (4)	0.0517 (4)	0.0692 (15)	
H7	0.2210	0.5628	0.0030	0.083*	
C8	0.6466 (5)	0.7500	-0.0769 (5)	0.076 (2)	
H8A	0.6775	0.8109	-0.0643	0.115*	0.50
H8B	0.6431	0.7405	-0.1412	0.115*	0.50
H8C	0.6838	0.6986	-0.0501	0.115*	0.50
C9	0.4688 (4)	0.8524 (4)	-0.0866 (3)	0.0831 (18)	
H9A	0.4841	0.8501	-0.1500	0.125*	
H9B	0.4933	0.9115	-0.0611	0.125*	
H9C	0.3995	0.8497	-0.0788	0.125*	
C10	0.6603 (6)	0.7500	0.3117 (5)	0.093 (3)	
H10A	0.6612	0.7633	0.3754	0.139*	0.50
H10B	0.6966	0.7989	0.2802	0.139*	0.50
H10C	0.6890	0.6878	0.3006	0.139*	0.50
C11	0.4839 (5)	0.8528 (5)	0.3307 (4)	0.111 (2)	
H11A	0.4142	0.8492	0.3289	0.166*	
H11B	0.5049	0.9115	0.3021	0.166*	

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H11C	0.5054	0.8522	0.3925	0.166*
C12	0.6168 (3)	0.8572 (3)	0.1187 (3)	0.0610 (13)
H12A	0.6079	0.8957	0.0652	0.091*
H12B	0.6808	0.8295	0.1184	0.091*
H12C	0.6092	0.8974	0.1713	0.091*

Atomic displacement parameters (\AA^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Co	0.0443 (5)	0.0331 (4)	0.0411 (5)	0.000	-0.0009 (4)	0.000
I	0.0849 (4)	0.0531 (3)	0.0645 (3)	0.000	-0.0021 (3)	0.000
P1	0.0541 (11)	0.0531 (11)	0.0420 (10)	0.000	0.0048 (8)	0.000
P2	0.0831 (15)	0.0558 (12)	0.0452 (11)	0.000	-0.0119 (10)	0.000
N	0.054 (2)	0.0358 (19)	0.043 (2)	-0.0006 (18)	-0.0003 (18)	0.0010 (16)
O	0.102 (3)	0.073 (3)	0.075 (3)	-0.031 (2)	-0.010 (2)	0.025 (2)
C1	0.053 (3)	0.034 (2)	0.048 (3)	0.002 (2)	0.001 (2)	0.003 (2)
C2	0.045 (3)	0.036 (2)	0.065 (3)	-0.003 (2)	0.004 (2)	-0.005 (2)
C3	0.056 (3)	0.042 (3)	0.073 (4)	-0.005 (3)	0.015 (3)	-0.001 (2)
C4	0.066 (4)	0.050 (3)	0.105 (5)	-0.015 (3)	0.028 (4)	0.002 (3)
C5	0.048 (3)	0.051 (3)	0.162 (7)	-0.013 (3)	0.007 (4)	-0.010 (4)
C6	0.064 (4)	0.059 (4)	0.127 (5)	-0.005 (3)	-0.026 (4)	-0.014 (4)
C7	0.070 (4)	0.055 (3)	0.082 (4)	-0.001 (3)	-0.020 (3)	0.002 (3)
C8	0.070 (5)	0.086 (6)	0.074 (5)	0.000	0.025 (4)	0.000
C9	0.102 (4)	0.095 (4)	0.052 (3)	0.030 (4)	0.006 (3)	0.017 (3)
C10	0.113 (7)	0.081 (6)	0.084 (6)	0.000	-0.048 (5)	0.000
C11	0.160 (6)	0.118 (6)	0.054 (4)	0.040 (5)	-0.006 (4)	-0.030 (4)
C12	0.059 (3)	0.051 (3)	0.073 (3)	-0.016 (2)	-0.004 (3)	-0.007 (3)

Geometric parameters (\AA , $^\circ$)

Co—N	1.956 (4)	C4—H4	0.9300
Co—N ⁱ	1.956 (4)	C5—C6	1.378 (8)
Co—C12	2.026 (4)	C5—H5	0.9300
Co—C12 ⁱ	2.026 (4)	C6—C7	1.377 (7)
Co—P1	2.232 (2)	C6—H6	0.9300
Co—P2	2.233 (2)	C7—H7	0.9300
Co—C1	3.089 (5)	C8—H8A	0.9600
P1—C9 ⁱ	1.816 (5)	C8—H8B	0.9600
P1—C9	1.816 (5)	C8—H8C	0.9600
P1—C8	1.818 (7)	C9—H9A	0.9600
P2—C11 ⁱ	1.810 (6)	C9—H9B	0.9600
P2—C11	1.810 (6)	C9—H9C	0.9600
P2—C10	1.822 (7)	C10—H10A	0.9600
N—C1	1.133 (5)	C10—H10B	0.9600
O—C3	1.350 (6)	C10—H10C	0.9600
O—H1	0.88 (5)	C11—H11A	0.9600
C1—C2	1.447 (6)	C11—H11B	0.9600
C2—C7	1.391 (6)	C11—H11C	0.9600

C2—C3	1.402 (6)	C12—H12A	0.9600
C3—C4	1.389 (6)	C12—H12B	0.9600
C4—C5	1.372 (7)	C12—H12C	0.9600
N—Co—N ⁱ	89.8 (2)	C6—C5—H5	119.4
N—Co—C12	177.90 (17)	C7—C6—C5	119.9 (6)
N ⁱ —Co—C12	88.08 (18)	C7—C6—H6	120.1
N ⁱ —Co—C12 ⁱ	177.90 (17)	C5—C6—H6	120.1
C12—Co—C12 ⁱ	94.0 (3)	C6—C7—C2	119.6 (5)
N—Co—P1	93.14 (10)	C6—C7—H7	120.2
C12—Co—P1	86.96 (14)	C2—C7—H7	120.2
N—Co—P2	93.57 (10)	P1—C8—H8A	109.5
C12—Co—P2	86.59 (14)	P1—C8—H8B	109.5
P1—Co—P2	170.53 (8)	H8A—C8—H8B	109.5
N ⁱ —Co—C1	89.53 (13)	P1—C8—H8C	109.5
C12—Co—C1	177.60 (16)	H8A—C8—H8C	109.5
C12 ⁱ —Co—C1	88.36 (16)	H8B—C8—H8C	109.5
P1—Co—C1	92.88 (8)	P1—C9—H9A	109.5
P2—Co—C1	93.85 (9)	P1—C9—H9B	109.5
C9 ⁱ —P1—C9	102.4 (4)	H9A—C9—H9B	109.5
C9—P1—C8	102.2 (2)	P1—C9—H9C	109.5
C9—P1—Co	116.20 (17)	H9A—C9—H9C	109.5
C8—P1—Co	115.5 (3)	H9B—C9—H9C	109.5
C11 ⁱ —P2—C11	103.3 (5)	P2—C10—H10A	109.5
C11—P2—C10	102.1 (3)	P2—C10—H10B	109.5
C11—P2—Co	115.8 (2)	H10A—C10—H10B	109.5
C10—P2—Co	115.6 (3)	P2—C10—H10C	109.5
C1—N—Co	179.0 (4)	H10A—C10—H10C	109.5
C3—O—H1	114 (3)	H10B—C10—H10C	109.5
N—C1—C2	177.7 (5)	P2—C11—H11A	109.5
C2—C1—Co	177.1 (3)	P2—C11—H11B	109.5
C7—C2—C3	120.6 (4)	H11A—C11—H11B	109.5
C7—C2—C1	119.8 (4)	P2—C11—H11C	109.5
C3—C2—C1	119.5 (4)	H11A—C11—H11C	109.5
O—C3—C4	124.2 (5)	H11B—C11—H11C	109.5
O—C3—C2	117.3 (4)	Co—C12—H12A	109.5
C4—C3—C2	118.4 (5)	Co—C12—H12B	109.5
C5—C4—C3	120.3 (5)	H12A—C12—H12B	109.5
C5—C4—H4	119.8	Co—C12—H12C	109.5
C3—C4—H4	119.8	H12A—C12—H12C	109.5
C4—C5—C6	121.1 (5)	H12B—C12—H12C	109.5
C4—C5—H5	119.4		
N—Co—P1—C9 ⁱ	-15.3 (2)	N ⁱ —Co—P2—C11	-15.6 (3)
N ⁱ —Co—P1—C9 ⁱ	-105.3 (3)	C12—Co—P2—C11	72.2 (3)
C12—Co—P1—C9 ⁱ	166.8 (3)	C12 ⁱ —Co—P2—C11	166.5 (3)
C12 ⁱ —Co—P1—C9 ⁱ	72.6 (3)	C1—Co—P2—C11	-105.4 (3)
C1—Co—P1—C9 ⁱ	-15.6 (2)	N—Co—P2—C10	134.98 (10)

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N—Co—P1—C9	105.3 (3)	N ⁱ —Co—P2—C10	-134.98 (10)
N ⁱ —Co—P1—C9	15.3 (2)	C12—Co—P2—C10	-47.12 (14)
C12—Co—P1—C9	-72.6 (3)	C12 ⁱ —Co—P2—C10	47.12 (14)
C12 ⁱ —Co—P1—C9	-166.8 (3)	C1—Co—P2—C10	135.24 (8)
C1—Co—P1—C9	105.0 (2)	C7—C2—C3—O	177.9 (4)
N—Co—P1—C8	-135.01 (10)	C1—C2—C3—O	-3.4 (6)
N ⁱ —Co—P1—C8	135.01 (10)	C7—C2—C3—C4	-2.3 (7)
C12—Co—P1—C8	47.10 (14)	C1—C2—C3—C4	176.4 (4)
C12 ⁱ —Co—P1—C8	-47.10 (14)	O—C3—C4—C5	-178.4 (5)
C1—Co—P1—C8	-135.30 (8)	C2—C3—C4—C5	1.8 (7)
N—Co—P2—C11 ⁱ	15.6 (3)	C3—C4—C5—C6	-0.1 (9)
N ⁱ —Co—P2—C11 ⁱ	105.7 (3)	C4—C5—C6—C7	-1.2 (9)
C12—Co—P2—C11 ⁱ	-166.5 (3)	C5—C6—C7—C2	0.7 (8)
C12 ⁱ —Co—P2—C11 ⁱ	-72.2 (3)	C3—C2—C7—C6	1.1 (7)
C1—Co—P2—C11 ⁱ	15.9 (3)	C1—C2—C7—C6	-177.6 (5)
N—Co—P2—C11	-105.7 (3)		

Symmetry codes: (i) $x, -y+3/2, z$.

Hydrogen-bond geometry ($\text{\AA}, ^\circ$)

D—H \cdots A	D—H	H \cdots A	D \cdots A	D—H \cdots A
O—H1 \cdots I	0.88 (5)	2.62 (5)	3.490 (4)	175 (4)

Fig. 1

