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{Bis[2-(diisopropylphosphanyl)ethyl]amine}carbonyl(tetrahydroborato)cobalt(I)

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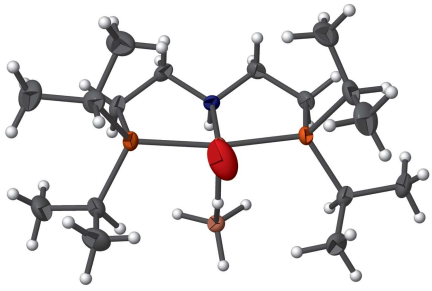
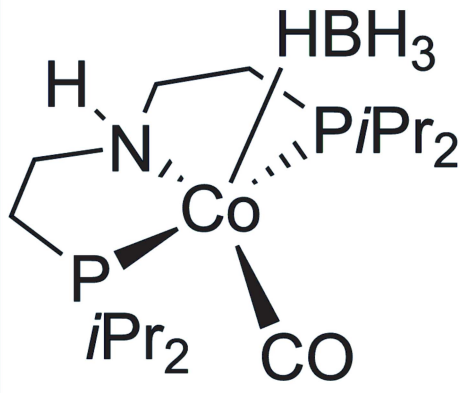
Structural data: full structural data are available from iucrdata.iucr.org

In the structure of title borohydride pincer complex, $[Co(BH_4)(C_{16}H_{37}NP_2)(CO)]$, the cobalt(I) metal exhibits a distorted square-pyramidal coordination geometry with the basal positions occupied by the P and N atoms of the tridentate ligand and by the C atom of the carbon monoxide ligand. In the crystal, molecules interact only by van der Waals forces.

3D view



Chemical scheme



Structure description

The title cobalt(I) borohydride species was formed by the reduction of the complex $[CoBr_2(CO)\{HN((CH_2CH_2)PiPr_2)_2\}]$ with five equivalents of $NaBH_4$. The title Co^I 18-electron complex consists of a PNP, one CO and a HBH_3 ligand coordinating to the Co^I atom (Fig. 1). The coordination geometry at the Co^I atom is best described as distorted square-pyramidal with the HBH_3 ligand in the apical position (τ = 0.25; Addison *et al.*, 1984). The metal atom is displaced by 0.2757 (7) Å from the mean plane through the P, N and C atoms occupying the basal positions (r.m.s. deviation = 0.135 Å). Both five-membered chelate rings involving the PNP ligand display an envelope conformation, with the flap atoms C2 and C3 lying 0.564 (2) and 0.599 (2) Å, respectively, from the Co1/P1/C1/N1 and Co1/P2/C4/N1 plane. The crystal packing is governed only by van der Waals interactions.

Synthesis and crystallization

The synthesis of the starting complex $[CoBr_2(CO)\{HN((CH_2CH_2)PiPr_2)_2\}]$ was described before (Junge *et al.*, 2018). $[CoBr_2(CO)\{HN((CH_2CH_2)PiPr_2)_2\}]$ (475.1 g, 0.86 mmol) and five equivalents of $NaBH_4$ (160.4 mg in 45 ml absolute EtOH) were added in a 100 ml Schlenk tube in an inert atmosphere. The mixture was stirred for 3 h at room temperature

Table 1
Experimental details.

Crystal data	
Chemical formula	$[Co(BH_4)(C_{16}H_{37}NP_2)(CO)]$
M_r	407.19
Crystal system, space group	Monoclinic, $P2_1/n$
Temperature (K)	150
a, b, c (Å)	8.0249 (2), 27.3200 (8), 10.3546 (3)
β (°)	97.101 (1)
V (Å^3)	2252.73 (11)
Z	4
Radiation type	Mo $K\alpha$
μ (mm^{-1})	0.91
Crystal size (mm)	0.38 × 0.32 × 0.14
Data collection	
Diffractometer	Bruker APEXII CCD
Absorption correction	Multi-scan (*SADABS*; Bruker, 2014)
T_{min}, T_{max}	0.72, 0.89
No. of measured, independent and observed [$I > 2\sigma(I)$] reflections	38885, 5449, 4886
R_{int}	0.026
(sin $\theta/\lambda)_{max}$ (Å^{-1})	0.660
Refinement	
$R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, S	0.030, 0.075, 1.08
No. of reflections	5449
No. of parameters	236
H-atom treatment	H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$, $\Delta\rho_{min}$ (e Å^{-3})	0.66, −0.42

Computer programs: *APEX2* and *SAINT* (Bruker, 2014), *SHELXS97* (Sheldrick, 2008), *SHELXL2014* (Sheldrick, 2015) and *publCIF* (Westrip, 2010).

giving a dark-brown solution. The solvent was removed under vacuum and the residue was suspended in Et_2O. After filtration, Et_2O was removed in vacuum and the solid was extracted several times with *n*-heptane (40 ml). Finally, *n*-heptane was removed leaving a reddish powder. Pure red crystals suitable for single crystal X-ray diffraction analysis were grown from a solution of Et_2O layered with *n*-heptane. Yield: 158.1 mg (45%). IR ATR ν (CO): 2971, 1914, 1871, ν (NH): 3400, ν (CH): 2959, 2871, 2783, ν (BH): 2368 cm^{-1}.

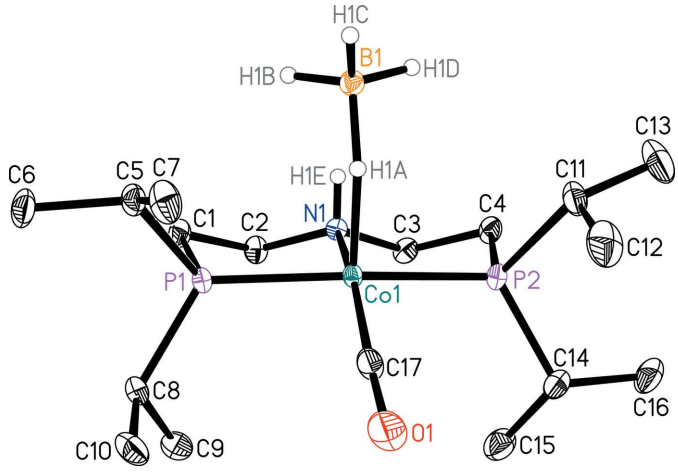


Figure 1
The molecular structure of the title compound with displacement ellipsoids drawn at 30% probability level. Hydrogen atoms except H1*A*–H1*E* are omitted for clarity.

Refinement

Crystal data, data collection and structure refinement details are summarized in Table 1.

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full crystallographic data

IUCrData (2018). **3**, x180471 [https://doi.org/10.1107/S2414314618004716]

{Bis[2-(diisopropylphosphanyl)ethyl]amine}carbonyl(tetrahydroborato)cobalt(I)

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Crystal data

$[Co(BH_4)(C_{16}H_{37}NP_2)(CO)]$
M_r = 407.19
Monoclinic, $P2_1/n$
a = 8.0249 (2) Å
b = 27.3200 (8) Å
c = 10.3546 (3) Å
β = 97.101 (1)°
V = 2252.73 (11) Å^3
Z = 4
$F(000)$ = 880
D_x = 1.201 Mg m^{-3}
Mo $K\alpha$ radiation, λ = 0.71073 Å
Cell parameters from 9366 reflections
θ = 2.5–28.8°
μ = 0.91 mm^{-1}
T = 150 K
Prism, red
0.38 × 0.32 × 0.14 mm

Data collection

Bruker APEXII CCD diffractometer
Radiation source: fine-focus sealed tube
Curved graphite monochromator
φ and ω scans
Absorption correction: multi-scan (SADABS; Bruker, 2014)
T_{min} = 0.72, T_{max} = 0.89
38885 measured reflections
5449 independent reflections
4886 reflections with $I > 2\sigma(I)$
R_{int} = 0.026
θ_{max} = 28.0°, θ_{min} = 1.5°
h = −10→10
k = −36→36
l = −13→13

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.030
$wR(F^2)$ = 0.075
S = 1.08
5449 reflections
236 parameters
0 restraints
Hydrogen site location: mixed
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0317P)^2 + 1.3762P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.66 e Å^{-3}
$\Delta\rho_{min}$ = −0.42 e Å^{-3}

Special details

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

Refinement. Atoms H1A–H1E could be located in a difference Fourier map and were refined freely. All other H atoms were placed in idealized positions with d(C—H) = 1.00 Å (CH), 0.99 Å (CH_2), 0.98 Å (CH_3) and refined using as riding with U_{iso}(H) fixed at 1.2 U_{eq}(C) for CH, CH_2 and 1.5 U_{eq}(C) for CH_3. A rotating model was used for the methyl H atoms.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²)

	x	*y*	*z*	U_{iso}*/U_{eq}
B1	−0.1303 (2)	0.04214 (7)	0.13590 (18)	0.0227 (3)
C1	0.3578 (2)	0.03751 (6)	0.24641 (16)	0.0265 (3)
H1F	0.4702	0.0332	0.2963	0.032*
H1G	0.2932	0.0069	0.2529	0.032*
C2	0.3747 (2)	0.04845 (6)	0.10518 (16)	0.0256 (3)
H2A	0.4645	0.0730	0.1001	0.031*
H2B	0.4059	0.0182	0.0612	0.031*
C3	0.2323 (2)	0.08158 (6)	−0.09605 (15)	0.0248 (3)
H3A	0.2656	0.0527	−0.1446	0.030*
H3B	0.3208	0.1068	−0.0963	0.030*
C4	0.0664 (2)	0.10156 (6)	−0.16071 (15)	0.0269 (3)
H4A	−0.0158	0.0747	−0.1786	0.032*
H4B	0.0822	0.1175	−0.2443	0.032*
C5	0.1334 (2)	0.05913 (7)	0.43592 (15)	0.0291 (4)
H5	0.0789	0.0297	0.3912	0.035*
C6	0.2471 (3)	0.03910 (8)	0.55335 (18)	0.0403 (5)
H6A	0.3005	0.0664	0.6042	0.060*
H6B	0.3338	0.0182	0.5233	0.060*
H6C	0.1800	0.0199	0.6079	0.060*
C7	−0.0108 (2)	0.09020 (8)	0.47412 (17)	0.0394 (4)
H7A	−0.0722	0.0716	0.5340	0.059*
H7B	−0.0870	0.0987	0.3960	0.059*
H7C	0.0343	0.1202	0.5169	0.059*
C8	0.4209 (2)	0.12393 (7)	0.40695 (18)	0.0325 (4)
H8	0.4845	0.1006	0.4691	0.039*
C9	0.5423 (2)	0.14279 (8)	0.3157 (2)	0.0435 (5)
H9A	0.4805	0.1624	0.2465	0.065*
H9B	0.5955	0.1150	0.2772	0.065*
H9C	0.6288	0.1630	0.3650	0.065*
C10	0.3582 (3)	0.16526 (8)	0.4871 (2)	0.0453 (5)
H10A	0.4544	0.1826	0.5338	0.068*
H10B	0.2889	0.1517	0.5499	0.068*
H10C	0.2911	0.1881	0.4293	0.068*
C11	−0.2425 (2)	0.14304 (8)	−0.07824 (18)	0.0341 (4)
H11	−0.2731	0.1127	−0.0325	0.041*
C12	−0.3163 (3)	0.18517 (9)	−0.0076 (2)	0.0506 (6)
H12A	−0.3013	0.2158	−0.0539	0.076*
H12B	−0.2588	0.1874	0.0813	0.076*
H12C	−0.4363	0.1793	−0.0049	0.076*
C13	−0.3250 (3)	0.13749 (11)	−0.2184 (2)	0.0572 (6)
H13A	−0.4430	0.1282	−0.2187	0.086*
H13B	−0.2664	0.1121	−0.2622	0.086*
H13C	−0.3186	0.1686	−0.2645	0.086*
C14	0.0493 (2)	0.20699 (6)	−0.10655 (18)	0.0313 (4)
H14	0.0024	0.2317	−0.0497	0.038*

C15	0.2386 (3)	0.21286 (8)	−0.0862 (2)	0.0471 (5)
H15A	0.2890	0.1915	−0.1469	0.071*
H15B	0.2814	0.2038	0.0034	0.071*
H15C	0.2678	0.2470	−0.1019	0.071*
C16	−0.0198 (3)	0.21952 (8)	−0.2475 (2)	0.0476 (5)
H16A	0.0344	0.2494	−0.2742	0.071*
H16B	−0.1413	0.2248	−0.2536	0.071*
H16C	0.0034	0.1924	−0.3047	0.071*
C17	0.0784 (2)	0.17611 (6)	0.20485 (17)	0.0305 (4)
Co1	0.09870 (2)	0.11960 (2)	0.14107 (2)	0.01695 (6)
H1A	−0.065 (3)	0.0841 (8)	0.157 (2)	0.037 (5)*
H1B	−0.034 (3)	0.0148 (8)	0.172 (2)	0.037 (6)*
H1C	−0.241 (3)	0.0384 (8)	0.188 (2)	0.037 (6)*
H1D	−0.161 (3)	0.0376 (8)	0.027 (2)	0.049 (6)*
N1	0.21356 (16)	0.06750 (5)	0.03935 (12)	0.0187 (2)
O1	0.0642 (2)	0.21577 (5)	0.24275 (16)	0.0561 (4)
P1	0.24794 (5)	0.08916 (2)	0.31343 (4)	0.02044 (9)
P2	−0.01034 (5)	0.14623 (2)	−0.04995 (4)	0.02156 (9)
H1E	0.147 (2)	0.0444 (7)	0.0339 (18)	0.022 (5)*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
B1	0.0229 (8)	0.0229 (8)	0.0225 (8)	−0.0002 (7)	0.0032 (7)	0.0005 (7)
C1	0.0223 (7)	0.0280 (8)	0.0284 (8)	0.0071 (6)	0.0001 (6)	0.0051 (6)
C2	0.0198 (7)	0.0296 (8)	0.0280 (8)	0.0084 (6)	0.0050 (6)	0.0004 (6)
C3	0.0305 (8)	0.0261 (8)	0.0197 (7)	0.0018 (6)	0.0106 (6)	−0.0003 (6)
C4	0.0381 (9)	0.0268 (8)	0.0155 (7)	0.0014 (7)	0.0024 (6)	0.0008 (6)
C5	0.0304 (8)	0.0383 (9)	0.0180 (7)	−0.0017 (7)	0.0014 (6)	0.0042 (7)
C6	0.0432 (11)	0.0521 (12)	0.0246 (9)	0.0009 (9)	0.0006 (8)	0.0161 (8)
C7	0.0363 (10)	0.0616 (13)	0.0212 (8)	0.0040 (9)	0.0069 (7)	−0.0010 (8)
C8	0.0304 (9)	0.0334 (9)	0.0298 (9)	−0.0032 (7)	−0.0115 (7)	0.0038 (7)
C9	0.0310 (10)	0.0447 (11)	0.0520 (12)	−0.0119 (8)	−0.0061 (9)	0.0058 (9)
C10	0.0531 (12)	0.0415 (11)	0.0365 (11)	−0.0053 (9)	−0.0136 (9)	−0.0112 (9)
C11	0.0230 (8)	0.0481 (11)	0.0297 (9)	0.0017 (7)	−0.0023 (7)	0.0102 (8)
C12	0.0283 (10)	0.0668 (15)	0.0578 (14)	0.0146 (10)	0.0095 (9)	0.0066 (11)
C13	0.0343 (11)	0.096 (2)	0.0382 (12)	−0.0045 (12)	−0.0089 (9)	0.0083 (12)
C14	0.0337 (9)	0.0252 (8)	0.0351 (9)	0.0038 (7)	0.0054 (7)	0.0069 (7)
C15	0.0371 (11)	0.0400 (11)	0.0643 (14)	−0.0026 (9)	0.0064 (10)	0.0167 (10)
C16	0.0615 (14)	0.0382 (11)	0.0422 (11)	0.0023 (10)	0.0028 (10)	0.0180 (9)
C17	0.0318 (9)	0.0290 (9)	0.0284 (9)	0.0075 (7)	−0.0059 (7)	−0.0058 (7)
Co1	0.01843 (10)	0.01766 (10)	0.01438 (10)	0.00293 (7)	0.00046 (7)	−0.00097 (7)
N1	0.0181 (6)	0.0198 (6)	0.0186 (6)	0.0015 (5)	0.0043 (5)	−0.0003 (5)
O1	0.0681 (11)	0.0346 (8)	0.0595 (10)	0.0175 (7)	−0.0161 (8)	−0.0228 (7)
P1	0.02065 (18)	0.0236 (2)	0.01616 (18)	0.00142 (15)	−0.00147 (14)	0.00128 (14)
P2	0.0239 (2)	0.0226 (2)	0.01789 (19)	0.00427 (15)	0.00123 (14)	0.00319 (14)

Geometric parameters (Å, º)

B1—H1A	1.27 (2)	C9—H9B	0.9800
B1—H1B	1.10 (2)	C9—H9C	0.9800
B1—H1C	1.10 (2)	C10—H10A	0.9800
B1—H1D	1.13 (2)	C10—H10B	0.9800
C1—C2	1.515 (2)	C10—H10C	0.9800
C1—P1	1.8440 (17)	C11—C12	1.522 (3)
C1—H1F	0.9900	C11—C13	1.527 (3)
C1—H1G	0.9900	C11—P2	1.8520 (18)
C2—N1	1.4796 (19)	C11—H11	1.0000
C2—H2A	0.9900	C12—H12A	0.9800
C2—H2B	0.9900	C12—H12B	0.9800
C3—N1	1.4792 (19)	C12—H12C	0.9800
C3—C4	1.516 (2)	C13—H13A	0.9800
C3—H3A	0.9900	C13—H13B	0.9800
C3—H3B	0.9900	C13—H13C	0.9800
C4—P2	1.8327 (17)	C14—C15	1.516 (3)
C4—H4A	0.9900	C14—C16	1.535 (3)
C4—H4B	0.9900	C14—P2	1.8431 (18)
C5—C7	1.526 (3)	C14—H14	1.0000
C5—C6	1.528 (2)	C15—H15A	0.9800
C5—P1	1.8480 (17)	C15—H15B	0.9800
C5—H5	1.0000	C15—H15C	0.9800
C6—H6A	0.9800	C16—H16A	0.9800
C6—H6B	0.9800	C16—H16B	0.9800
C6—H6C	0.9800	C16—H16C	0.9800
C7—H7A	0.9800	C17—O1	1.163 (2)
C7—H7B	0.9800	C17—Co1	1.6949 (17)
C7—H7C	0.9800	Co1—N1	2.0555 (12)
C8—C10	1.523 (3)	Co1—P2	2.1863 (4)
C8—C9	1.528 (3)	Co1—P1	2.1869 (4)
C8—P1	1.8531 (18)	Co1—H1A	1.66 (2)
C8—H8	1.0000	N1—H1E	0.823 (19)
C9—H9A	0.9800		

H1A—B1—H1B	107.3 (14)	C12—C11—C13	112.53 (18)
H1A—B1—H1C	109.8 (14)	C12—C11—P2	109.38 (14)
H1B—B1—H1C	110.3 (15)	C13—C11—P2	117.68 (14)
H1A—B1—H1D	108.0 (15)	C12—C11—H11	105.4
H1B—B1—H1D	108.5 (16)	C13—C11—H11	105.4
H1C—B1—H1D	112.8 (16)	P2—C11—H11	105.4
C2—C1—P1	108.44 (11)	C11—C12—H12A	109.5
C2—C1—H1F	110.0	C11—C12—H12B	109.5
P1—C1—H1F	110.0	H12A—C12—H12B	109.5
C2—C1—H1G	110.0	C11—C12—H12C	109.5
P1—C1—H1G	110.0	H12A—C12—H12C	109.5
H1F—C1—H1G	108.4	H12B—C12—H12C	109.5

N1—C2—C1	109.67 (12)	C11—C13—H13A	109.5
N1—C2—H2A	109.7	C11—C13—H13B	109.5
C1—C2—H2A	109.7	H13A—C13—H13B	109.5
N1—C2—H2B	109.7	C11—C13—H13C	109.5
C1—C2—H2B	109.7	H13A—C13—H13C	109.5
H2A—C2—H2B	108.2	H13B—C13—H13C	109.5
N1—C3—C4	109.02 (12)	C15—C14—C16	110.30 (17)
N1—C3—H3A	109.9	C15—C14—P2	110.25 (13)
C4—C3—H3A	109.9	C16—C14—P2	115.20 (14)
N1—C3—H3B	109.9	C15—C14—H14	106.9
C4—C3—H3B	109.9	C16—C14—H14	106.9
H3A—C3—H3B	108.3	P2—C14—H14	106.9
C3—C4—P2	107.94 (11)	C14—C15—H15A	109.5
C3—C4—H4A	110.1	C14—C15—H15B	109.5
P2—C4—H4A	110.1	H15A—C15—H15B	109.5
C3—C4—H4B	110.1	C14—C15—H15C	109.5
P2—C4—H4B	110.1	H15A—C15—H15C	109.5
H4A—C4—H4B	108.4	H15B—C15—H15C	109.5
C7—C5—C6	112.97 (15)	C14—C16—H16A	109.5
C7—C5—P1	112.85 (13)	C14—C16—H16B	109.5
C6—C5—P1	113.96 (13)	H16A—C16—H16B	109.5
C7—C5—H5	105.3	C14—C16—H16C	109.5
C6—C5—H5	105.3	H16A—C16—H16C	109.5
P1—C5—H5	105.3	H16B—C16—H16C	109.5
C5—C6—H6A	109.5	O1—C17—Co1	176.79 (18)
C5—C6—H6B	109.5	C17—Co1—N1	154.31 (8)
H6A—C6—H6B	109.5	C17—Co1—P2	90.14 (6)
C5—C6—H6C	109.5	N1—Co1—P2	85.50 (4)
H6A—C6—H6C	109.5	C17—Co1—P1	95.61 (6)
H6B—C6—H6C	109.5	N1—Co1—P1	85.40 (4)
C5—C7—H7A	109.5	P2—Co1—P1	169.253 (18)
C5—C7—H7B	109.5	C17—Co1—H1A	112.2 (7)
H7A—C7—H7B	109.5	N1—Co1—H1A	93.4 (7)
C5—C7—H7C	109.5	P2—Co1—H1A	93.1 (7)
H7A—C7—H7C	109.5	P1—Co1—H1A	93.2 (7)
H7B—C7—H7C	109.5	C3—N1—C2	110.05 (12)
C10—C8—C9	111.44 (17)	C3—N1—Co1	114.21 (9)
C10—C8—P1	112.85 (13)	C2—N1—Co1	115.41 (10)
C9—C8—P1	110.08 (13)	C3—N1—H1E	106.0 (13)
C10—C8—H8	107.4	C2—N1—H1E	106.4 (13)
C9—C8—H8	107.4	Co1—N1—H1E	103.8 (13)
P1—C8—H8	107.4	C1—P1—C5	102.71 (8)
C8—C9—H9A	109.5	C1—P1—C8	103.12 (8)
C8—C9—H9B	109.5	C5—P1—C8	105.84 (8)
H9A—C9—H9B	109.5	C1—P1—Co1	102.88 (5)
C8—C9—H9C	109.5	C5—P1—Co1	117.40 (6)
H9A—C9—H9C	109.5	C8—P1—Co1	122.06 (6)
H9B—C9—H9C	109.5	C4—P2—C14	106.27 (8)

C8—C10—H10A	109.5	C4—P2—C11	106.20 (9)
C8—C10—H10B	109.5	C14—P2—C11	106.73 (8)
H10A—C10—H10B	109.5	C4—P2—Co1	102.51 (5)
C8—C10—H10C	109.5	C14—P2—Co1	119.79 (6)
H10A—C10—H10C	109.5	C11—P2—Co1	114.19 (6)
H10B—C10—H10C	109.5		

P1—C1—C2—N1	−44.85 (16)	C9—C8—P1—C5	169.94 (13)
N1—C3—C4—P2	48.02 (15)	C10—C8—P1—Co1	73.05 (15)
C4—C3—N1—C2	−179.27 (13)	C9—C8—P1—Co1	−52.15 (16)
C4—C3—N1—Co1	−47.59 (15)	C3—C4—P2—C14	98.19 (12)
C1—C2—N1—C3	176.00 (13)	C3—C4—P2—C11	−148.41 (12)
C1—C2—N1—Co1	44.96 (16)	C3—C4—P2—Co1	−28.31 (12)
C2—C1—P1—C5	148.85 (12)	C15—C14—P2—C4	−67.28 (16)
C2—C1—P1—C8	−101.28 (12)	C16—C14—P2—C4	58.33 (16)
C2—C1—P1—Co1	26.47 (12)	C15—C14—P2—C11	179.69 (15)
C7—C5—P1—C1	−161.14 (13)	C16—C14—P2—C11	−54.70 (17)
C6—C5—P1—C1	68.27 (15)	C15—C14—P2—Co1	48.01 (16)
C7—C5—P1—C8	91.05 (14)	C16—C14—P2—Co1	173.62 (12)
C6—C5—P1—C8	−39.53 (16)	C12—C11—P2—C4	−170.08 (14)
C7—C5—P1—Co1	−49.16 (14)	C13—C11—P2—C4	−40.04 (19)
C6—C5—P1—Co1	−179.75 (12)	C12—C11—P2—C14	−57.00 (16)
C10—C8—P1—C1	−172.37 (14)	C13—C11—P2—C14	73.03 (19)
C9—C8—P1—C1	62.42 (15)	C12—C11—P2—Co1	77.72 (14)
C10—C8—P1—C5	−64.86 (15)	C13—C11—P2—Co1	−152.24 (16)