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Cyanidotetrakis(trimethylphosphine)-cobalt(I)

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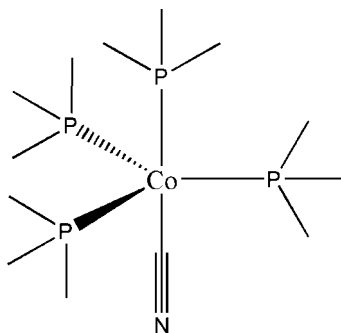
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 Key indicators: single-crystal X-ray study; $T = 293$ K; mean $\sigma(\text{P}-\text{C}) = 0.010$ Å; R factor = 0.068; wR factor = 0.216; data-to-parameter ratio = 24.0.

The title compound, $[\text{Co}(\text{CN})(\text{C}_3\text{H}_9\text{P})_4]$, was obtained as a product of the reaction of $[\text{Co}(\text{PMe}_3)_4]$ with a molar equivalent of 2,6-difluorobenzonitrile in diethyl ether. This compound is stable in the air for several hours, but rapidly decomposes at room temperature in solution. The cobalt(I) atom has a trigonal-bipyramidal coordination environment in which the cyano group and one of the PMe_3 groups are in the axial positions.

Related literature

For related cobalt(II) compounds, see: Yu *et al.* (2008); Li *et al.* (2006).



Experimental

Crystal data

 $[\text{Co}(\text{CN})(\text{C}_3\text{H}_9\text{P})_4]$
 $M_r = 389.24$

 Monoclinic, $P2_1/c$
 $a = 13.160$ (3) Å
 $b = 9.6136$ (19) Å
 $c = 17.971$ (4) Å
 $\beta = 93.09$ (3)°
 $V = 2270.3$ (8) Å³
 $Z = 4$
 Mo $K\alpha$ radiation
 $\mu = 1.03$ mm⁻¹
 $T = 293$ K
 $0.25 \times 0.23 \times 0.22$ mm

Data collection

 Bruker APEXII diffractometer
 Absorption correction: multi-scan
 (*SADABS*; Bruker, 2001)
 $T_{\min} = 0.395$, $T_{\max} = 0.434$

 11732 measured reflections
 4438 independent reflections
 3663 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.020$

Refinement

 $R[F^2 > 2\sigma(F^2)] = 0.068$
 $wR(F^2) = 0.216$
 $S = 1.06$
 4438 reflections
 185 parameters

 150 restraints
 H-atom parameters constrained
 $\Delta\rho_{\max} = 1.06$ e Å⁻³
 $\Delta\rho_{\min} = -2.00$ e Å⁻³
Table 1

Selected geometric parameters (Å, °).

C13—N1	1.166 (7)	Co1—P1	2.2082 (17)
Co1—C13	1.896 (5)	Co1—P4	2.2115 (17)
Co1—P2	2.2018 (15)	Co1—P3	2.2272 (17)
N1—C13—Co1	178.8 (6)	P1—Co1—P4	119.14 (8)
C13—Co1—P2	179.02 (19)	C13—Co1—P3	84.66 (19)
C13—Co1—P1	83.92 (19)	P2—Co1—P3	94.75 (7)
P2—Co1—P1	97.05 (7)	P1—Co1—P3	118.05 (8)
C13—Co1—P4	83.39 (18)	P4—Co1—P3	119.55 (8)
P2—Co1—P4	96.24 (6)		

Data collection: *APEX2* (Bruker, 2004); cell refinement: *SAINTE-Plus* (Bruker, 2001); data reduction: *SAINTE-Plus*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *SHELXTL* (Sheldrick, 2008); software used to prepare material for publication: *SHELXTL*.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: OM2401).

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 Bruker (2004). *APEX2*. Bruker AXS Inc., Madison, Wisconsin, USA.
 Li, X., Sun, H. & Yu, F. (2006). *Organometallics*, **25**, 4695–4697.
 Sheldrick, G. M. (2008). *Acta Cryst.* **A64**, 112–122.
 Yu, F., Wang, Q. & Li, X. (2008). *Acta Cryst.* **E64**, m112.

supporting information

Acta Cryst. (2011). E67, m503 [doi:10.1107/S160053681101083X]

Cyanidotetrakis(trimethylphosphine)cobalt(I)

Xiaofeng Xu, Lei Feng and Xiaoyan Li

S1. Comment

In the title molecule (Fig. 1, Table 1) the cobalt(I) atom has trigonal planar coordination geometry. Three PMe_3 groups using P1, P3 and P4 form the trigonal plane and the fourth PMe_3 group, using P2, and the cyano group are in the axial position. The axial groups are linear with P2-Co1-C13 of $179.02(1)^\circ$. The sum of the bond angles in the trigonal plane ($356.75(8)^\circ$) indicates only slightly distorted planarity. We have previously reported two related cobalt(II) structures. In one, (Yu *et al.*, 2008), the axial PMe_3 group is replaced by a phenyl and in the other, it is replaced by a 2,6-difluorophenyl group (Li *et al.*, 2006). These structures show a distorted square pyramidal coordination, which is different from the title compound.

S2. Experimental

A sample of $\text{Co}(\text{PMe}_3)_4$ (1.0 g, 2.75 mmol) in 30 ml of diethyl ether was combined with a solution of 2,6-difluorobenzonitrile (0.19 g, 1.36 mmol) in diethyl ether (20 ml) at -80°A . The reaction mixture was warmed to ambient temperature and stirred for 24 h to form a red solution. The volatiles were removed *in vacuo*, and the resulting solid was extracted with pentane (50 ml). Crystallization from its pentane solution at -15°C afforded red crystals suitable for X-ray diffraction analysis (yield 0.22 g, 42%), dec $> 120^\circ\text{C}$. The datum crystal was coated with perfluoropolyether to retard decomposition due to air sensitivity during data collection.

S3. Refinement

All H atoms on C were placed in calculated positions with a C—H bond distance of 0.96 \AA and $U_{\text{iso}}(\text{H}) = 1.5U_{\text{eq}}$ of the carrier atom. Large thermal motion of the methyl groups required that restraints be applied to the thermal parameters of several atoms, namely, SIMU 0.001 0.002 3.8 P3 C7 C8 C9; SIMU 0.01 0.02 3.8 P4 C10 C11 C12; ISOR 0.01 \$C. The maximum and minimum peaks in the final electron density map had values of 1.06 e/\AA^3 and -2.00 e/\AA^3 which were located 0.03 \AA from P3 and 0.05 \AA from C7, respectively.

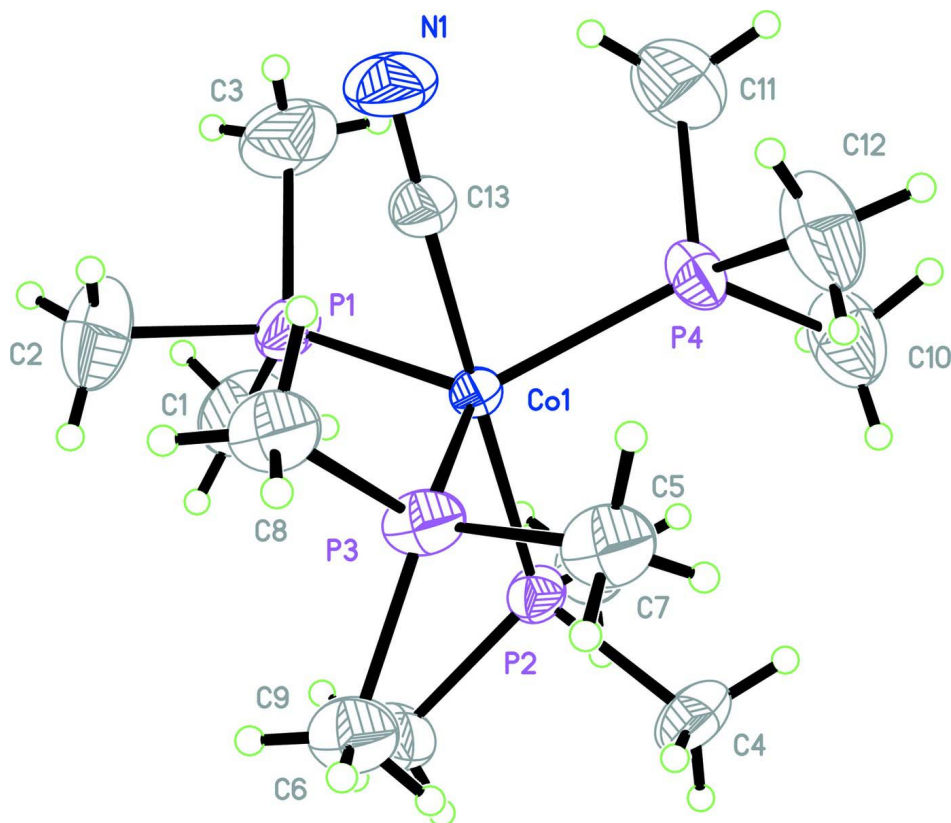


Figure 1

A view of the structure of the title compound showing 30% probability displacement ellipsoids.

Cyanidotetrakis(trimethylphosphine)cobalt(I)

Crystal data

[Co(CN)(C₃H₉P)₄]

$M_r = 389.24$

Monoclinic, $P2_1/c$

Hall symbol: -P 2ybc

$a = 13.160 (3) \text{ \AA}$

$b = 9.6136 (19) \text{ \AA}$

$c = 17.971 (4) \text{ \AA}$

$\beta = 93.09 (3)^\circ$

$V = 2270.3 (8) \text{ \AA}^3$

$Z = 4$

$F(000) = 832.0$

$D_x = 1.139 \text{ Mg m}^{-3}$

Mo $K\alpha$ radiation, $\lambda = 0.71073 \text{ \AA}$

Cell parameters from 4976 reflections

$\theta = 2.3\text{--}26.0^\circ$

$\mu = 1.03 \text{ mm}^{-1}$

$T = 293 \text{ K}$

Block, red

$0.25 \times 0.23 \times 0.22 \text{ mm}$

Data collection

Bruker APEXII

diffractometer

Radiation source: fine-focus sealed tube

Graphite monochromator

φ and ω scans

Absorption correction: multi-scan

(SADABS; Bruker, 2001)

$T_{\min} = 0.395$, $T_{\max} = 0.434$

11732 measured reflections

4438 independent reflections

3663 reflections with $I > 2\sigma(I)$

$R_{\text{int}} = 0.020$

$\theta_{\max} = 26.0^\circ$, $\theta_{\min} = 2.3^\circ$

$h = -15 \rightarrow 16$

$k = -11 \rightarrow 11$

$l = -15 \rightarrow 22$

Refinement

Refinement on F^2
 Least-squares matrix: full
 $R[F^2 > 2\sigma(F^2)] = 0.068$
 $wR(F^2) = 0.216$
 $S = 1.06$
 4438 reflections
 185 parameters
 150 restraints
 Primary atom site location: structure-invariant
 direct methods

Secondary atom site location: difference Fourier
 map
 Hydrogen site location: inferred from
 neighbouring sites
 H-atom parameters constrained
 $w = 1/[\sigma^2(F_o^2) + (0.1278P)^2 + 4.380P]$
 where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{\max} < 0.001$
 $\Delta\rho_{\max} = 1.06 \text{ e } \text{\AA}^{-3}$
 $\Delta\rho_{\min} = -2.00 \text{ e } \text{\AA}^{-3}$

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)

	x	y	z	$U_{\text{iso}}^*/U_{\text{eq}}$
C1	0.0427 (7)	0.5719 (11)	0.6108 (6)	0.112 (3)
H1A	-0.0142	0.5545	0.6407	0.167*
H1B	0.0756	0.4855	0.6002	0.167*
H1C	0.0194	0.6154	0.5649	0.167*
C2	0.1727 (9)	0.5681 (11)	0.7382 (6)	0.125 (4)
H2A	0.2114	0.6174	0.7766	0.187*
H2B	0.2134	0.4948	0.7192	0.187*
H2C	0.1130	0.5292	0.7585	0.187*
C3	0.0419 (8)	0.7949 (12)	0.7113 (7)	0.123 (4)
H3A	-0.0118	0.7361	0.7271	0.184*
H3B	0.0140	0.8651	0.6782	0.184*
H3C	0.0754	0.8383	0.7540	0.184*
C4	0.3468 (6)	0.7624 (9)	0.4375 (4)	0.080 (2)
H4A	0.4136	0.7418	0.4588	0.120*
H4B	0.3416	0.8603	0.4273	0.120*
H4C	0.3353	0.7112	0.3920	0.120*
C5	0.1325 (6)	0.7398 (9)	0.4449 (4)	0.0768 (19)
H5A	0.1354	0.6858	0.4002	0.115*
H5B	0.1263	0.8366	0.4323	0.115*
H5C	0.0748	0.7115	0.4716	0.115*
C6	0.2570 (7)	0.5211 (8)	0.4942 (4)	0.080 (2)
H6A	0.1998	0.4794	0.5167	0.120*
H6B	0.3189	0.4877	0.5186	0.120*
H6C	0.2557	0.4971	0.4424	0.120*
C7	0.5231 (5)	0.8559 (9)	0.6188 (4)	0.0783 (9)

H7A	0.5086	0.8925	0.5697	0.117*
H7B	0.5871	0.8078	0.6203	0.117*
H7C	0.5264	0.9309	0.6541	0.117*
C8	0.4623 (6)	0.7086 (8)	0.7401 (4)	0.0771 (9)
H8A	0.4308	0.6273	0.7597	0.116*
H8B	0.4438	0.7887	0.7682	0.116*
H8C	0.5349	0.6975	0.7436	0.116*
C9	0.4766 (5)	0.5759 (8)	0.6060 (4)	0.0770 (9)
H9A	0.5466	0.5704	0.6237	0.115*
H9B	0.4727	0.5797	0.5525	0.115*
H9C	0.4407	0.4954	0.6222	0.115*
C10	0.1996 (9)	1.0851 (10)	0.4832 (5)	0.114 (3)
H10A	0.1993	1.1849	0.4826	0.172*
H10B	0.1330	1.0512	0.4683	0.172*
H10C	0.2479	1.0513	0.4493	0.172*
C11	0.1338 (10)	1.1173 (12)	0.6267 (7)	0.134 (3)
H11A	0.1330	1.0866	0.6775	0.201*
H11B	0.0691	1.0984	0.6016	0.201*
H11C	0.1470	1.2154	0.6255	0.201*
C12	0.3430 (9)	1.1417 (11)	0.5971 (7)	0.123 (3)
H12A	0.4025	1.1046	0.5757	0.185*
H12B	0.3557	1.1521	0.6499	0.185*
H12C	0.3270	1.2308	0.5754	0.185*
C13	0.2731 (5)	0.8878 (6)	0.7120 (3)	0.0545 (13)
Co1	0.26139 (5)	0.80567 (6)	0.61596 (3)	0.0371 (3)
N1	0.2797 (6)	0.9405 (7)	0.7705 (3)	0.0869 (19)
P1	0.13485 (13)	0.68895 (18)	0.66233 (9)	0.0621 (5)
P2	0.25026 (12)	0.71209 (15)	0.50400 (7)	0.0479 (4)
P3	0.41947 (12)	0.7316 (2)	0.64284 (9)	0.0605 (4)
P4	0.23568 (14)	1.02279 (15)	0.57888 (9)	0.0598 (4)

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.099 (5)	0.129 (7)	0.109 (6)	-0.062 (5)	0.023 (5)	-0.026 (5)
C2	0.133 (7)	0.130 (7)	0.111 (6)	-0.039 (6)	0.015 (5)	0.045 (6)
C3	0.097 (6)	0.142 (8)	0.136 (7)	-0.010 (5)	0.061 (6)	-0.021 (6)
C4	0.091 (5)	0.105 (5)	0.046 (3)	-0.014 (4)	0.021 (3)	-0.007 (3)
C5	0.079 (4)	0.086 (4)	0.063 (4)	-0.010 (4)	-0.019 (3)	-0.003 (3)
C6	0.104 (5)	0.061 (4)	0.076 (4)	0.000 (4)	0.002 (4)	-0.019 (3)
C7	0.0674 (16)	0.0953 (17)	0.0713 (16)	0.0130 (15)	-0.0047 (14)	-0.0044 (15)
C8	0.0674 (15)	0.0951 (17)	0.0678 (16)	0.0156 (15)	-0.0053 (14)	-0.0044 (15)
C9	0.0670 (15)	0.0934 (17)	0.0696 (16)	0.0167 (15)	-0.0053 (14)	-0.0069 (15)
C10	0.149 (6)	0.085 (5)	0.106 (5)	0.007 (5)	-0.026 (5)	0.023 (4)
C11	0.153 (7)	0.102 (6)	0.147 (7)	0.044 (5)	0.013 (6)	0.008 (5)
C12	0.141 (6)	0.082 (5)	0.142 (6)	-0.031 (5)	-0.036 (5)	0.022 (5)
C13	0.063 (3)	0.056 (3)	0.044 (3)	0.002 (3)	0.003 (2)	-0.005 (2)
Co1	0.0414 (4)	0.0375 (4)	0.0323 (4)	0.0004 (2)	0.0023 (3)	-0.0018 (2)

N1	0.117 (5)	0.090 (4)	0.053 (3)	0.013 (4)	0.001 (3)	-0.026 (3)
P1	0.0630 (10)	0.0687 (10)	0.0564 (9)	-0.0182 (7)	0.0193 (7)	-0.0050 (7)
P2	0.0575 (8)	0.0509 (8)	0.0353 (7)	-0.0059 (6)	0.0012 (6)	-0.0048 (5)
P3	0.0509 (8)	0.0800 (10)	0.0498 (8)	0.0160 (7)	-0.0038 (6)	-0.0075 (7)
P4	0.0747 (10)	0.0405 (8)	0.0628 (9)	0.0027 (7)	-0.0097 (8)	0.0039 (6)

Geometric parameters (Å, °)

C1—P1	1.863 (8)	C7—H7C	0.9600
C1—H1A	0.9600	C8—P3	1.820 (7)
C1—H1B	0.9600	C8—H8A	0.9600
C1—H1C	0.9600	C8—H8B	0.9600
C2—P1	1.840 (10)	C8—H8C	0.9600
C2—H2A	0.9600	C9—P3	1.816 (7)
C2—H2B	0.9600	C9—H9A	0.9600
C2—H2C	0.9600	C9—H9B	0.9600
C3—P1	1.850 (9)	C9—H9C	0.9600
C3—H3A	0.9600	C10—P4	1.858 (9)
C3—H3B	0.9600	C10—H10A	0.9600
C3—H3C	0.9600	C10—H10B	0.9600
C4—P2	1.855 (7)	C10—H10C	0.9600
C4—H4A	0.9600	C11—P4	1.866 (11)
C4—H4B	0.9600	C11—H11A	0.9600
C4—H4C	0.9600	C11—H11B	0.9600
C5—P2	1.850 (7)	C11—H11C	0.9600
C5—H5A	0.9600	C12—P4	1.833 (10)
C5—H5B	0.9600	C12—H12A	0.9600
C5—H5C	0.9600	C12—H12B	0.9600
C6—P2	1.847 (7)	C12—H12C	0.9600
C6—H6A	0.9600	C13—N1	1.166 (7)
C6—H6B	0.9600	Co1—C13	1.896 (5)
C6—H6C	0.9600	Co1—P2	2.2018 (15)
C7—P3	1.881 (8)	Co1—P1	2.2082 (17)
C7—H7A	0.9600	Co1—P4	2.2115 (17)
C7—H7B	0.9600	Co1—P3	2.2272 (17)
P1—C1—H1A	109.5	P4—C10—H10B	109.5
P1—C1—H1B	109.5	P4—C10—H10C	109.5
H1A—C1—H1B	109.5	P4—C11—H11A	109.5
P1—C1—H1C	109.5	P4—C11—H11B	109.5
H1A—C1—H1C	109.5	P4—C11—H11C	109.5
H1B—C1—H1C	109.5	P4—C12—H12A	109.5
P1—C2—H2A	109.5	P4—C12—H12B	109.5
P1—C2—H2B	109.5	P4—C12—H12C	109.5
H2A—C2—H2B	109.5	N1—C13—Co1	178.8 (6)
P1—C2—H2C	109.5	C13—Co1—P2	179.02 (19)
H2A—C2—H2C	109.5	C13—Co1—P1	83.92 (19)
H2B—C2—H2C	109.5	P2—Co1—P1	97.05 (7)

P1—C3—H3A	109.5	C13—Co1—P4	83.39 (18)
P1—C3—H3B	109.5	P2—Co1—P4	96.24 (6)
P1—C3—H3C	109.5	P1—Co1—P4	119.14 (8)
P2—C4—H4A	109.5	C13—Co1—P3	84.66 (19)
P2—C4—H4B	109.5	P2—Co1—P3	94.75 (7)
H4A—C4—H4B	109.5	P1—Co1—P3	118.05 (8)
P2—C4—H4C	109.5	P4—Co1—P3	119.55 (8)
H4A—C4—H4C	109.5	C2—P1—C3	98.9 (6)
H4B—C4—H4C	109.5	C2—P1—C1	97.5 (5)
P2—C5—H5A	109.5	C3—P1—C1	98.0 (5)
P2—C5—H5B	109.5	C2—P1—Co1	114.9 (3)
H5A—C5—H5B	109.5	C3—P1—Co1	115.5 (4)
P2—C5—H5C	109.5	C1—P1—Co1	127.1 (3)
H5A—C5—H5C	109.5	C6—P2—C5	97.6 (4)
H5B—C5—H5C	109.5	C6—P2—C4	99.2 (4)
P2—C6—H6A	109.5	C5—P2—C4	100.1 (4)
P2—C6—H6B	109.5	C6—P2—Co1	119.5 (3)
H6A—C6—H6B	109.5	C5—P2—Co1	118.6 (3)
P2—C6—H6C	109.5	C4—P2—Co1	117.9 (3)
H6A—C6—H6C	109.5	C9—P3—C8	97.9 (3)
H6B—C6—H6C	109.5	C9—P3—C7	96.9 (4)
P3—C7—H7A	109.5	C8—P3—C7	96.1 (4)
P3—C7—H7B	109.5	C9—P3—Co1	125.8 (2)
P3—C7—H7C	109.5	C8—P3—Co1	119.0 (2)
P3—C8—H8A	109.5	C7—P3—Co1	115.3 (2)
P3—C8—H8B	109.5	C12—P4—C10	96.9 (5)
P3—C8—H8C	109.5	C12—P4—C11	100.4 (6)
P3—C9—H9A	109.5	C10—P4—C11	96.6 (5)
P3—C9—H9B	109.5	C12—P4—Co1	115.5 (4)
P3—C9—H9C	109.5	C10—P4—Co1	127.6 (3)
P4—C10—H10A	109.5	C11—P4—Co1	115.0 (4)
