Inorganic Chemistry
including bioinorganic chemistry
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STRUCTURE DETERMINATION SUMMARY

Crystal Data

Empirical Formula	$C_{18} H_{29} Co F_{12} N_2 P_2$
Color; Habit	pale yellow plates
Crystal Size (mm)	0.64 x 0.24 x 0.12
Crystal System	Triclinic
Space Group	$P\bar{1}$
Unit Cell Dimensions	$a = 6.314(2)$ Å
	$b = 7.137(2)$ Å
	$c = 13.452(2)$ Å
	$\alpha = 103.66(2)^{\circ}$
	$\beta = 90.25(2)^{\circ}$
	$\gamma = 92.89(2)^{\circ}$
Volume	588.2(3) $Å^3$
Z	1
Formula Weight	622.3
Density(calc.)	1.757 Mg/m^3
Absorption Coefficient	8.011 mm^{-1}
F(000)	316

Data Collection

Diffractometer Used	Siemens P4
Radiation	CuKα (λ = 1.54178 Å)
Temperature (K)	153
Monochromator	Highly oriented graphite crystal
2θ Range	4.0 to 100.0°
Scan Type	ω
Scan Speed	Variable; 6.00 to 60.00°/min. in ω
Scan Range (ω)	3.00°
Background Measurement	Stationary crystal and stationary counter at beginning and end of scan, each for 25.0% of total scan time
Standard Reflections	4 measured every 100 reflections
Index Ranges	$-4 \leq h \leq 6$, $-6 \leq k \leq 6$ $-12 \leq \ell \leq 12$
Reflections Collected	1455
Independent Reflections	1065 (R_{int} = 6.31%)
Observed Reflections	932 ($F > 4.0\sigma(F)$)
Absorption Correction	N/A

Solution and Refinement

System Used	Siemens SHELXTL IRIS
Solution	Direct Methods
Refinement Method	Full-Matrix Least-Squares
Quantity Minimized	$\sum w(F_o - F_c)^2$
Absolute Structure	N/A
Extinction Correction	N/A
Hydrogen Atoms	Riding model, fixed isotropic U
Weighting Scheme	Unit weights
Number of Parameters Refined	161
Final R Indices (obs. data)	R = 4.01 %, wR = 4.23 %
R Indices (all data)	R = 4.69 %, wR = 4.75 %
Goodness-of-Fit	1.02
Largest and Mean Δ/σ	0.596, 0.090
Data-to-Parameter Ratio	5.8:1
Largest Difference Peak	0.26 $eÅ^{-3}$
Largest Difference Hole	-0.24 $eÅ^{-3}$

Table 1. Atomic coordinates ($x10^4$) and equivalent isotropic displacement coefficients ($Å^2 x10^3$)

	x	y	z	U(eq)
Co(1)	5000	5000	0	20(1)
P(1)	9818(3)	325(2)	2517(1)	28(1)
F(1)	9490(6)	53(5)	1326(3)	63(1)
F(2)	12307(5)	204(5)	2352(3)	51(1)
F(3)	9622(5)	-1952(4)	2374(3)	54(1)
F(4)	7333(5)	436(4)	2675(3)	42(1)
F(5)	10026(5)	2606(5)	2647(3)	62(1)
F(6)	10163(6)	596(5)	3705(3)	65(1)
N(1)	5330(6)	4030(6)	-4298(3)	25(1)
C(1)	6272(7)	7038(6)	-680(4)	30(1)
C(2)	5046(7)	7899(7)	184(4)	35(1)
C(3)	2959(7)	7117(7)	-2(4)	33(1)
C(4)	2887(7)	5758(6)	-964(4)	27(1)
C(5)	4925(7)	5679(6)	-1387(4)	22(1)
C(6)	5558(7)	4408(7)	-2376(4)	31(1)
C(7)	4998(7)	5292(6)	-3269(4)	26(1)
C(8)	3784(8)	2347(7)	-4525(4)	46(1)
C(9)	7507(7)	3449(7)	-4437(4)	41(1)

* Equivalent isotropic U defined as one third of the trace of the orthogonalized U_{ij} tensor

Table 2. Bond lengths (Å)

Co(1)-C(1)	2.028	(5)	
Co(1)-C(3)	2.035	(5)	
Co(1)-C(5)	2.036	(5)	
Co(1)-C(2A)	2.024	(5)	
Co(1)-C(4A)	2.036	(5)	
P(1)-F(1)	1.580	(4)	
P(1)-F(3)	1.589	(3)	
P(1)-F(5)	1.594	(4)	
N(1)-C(7)	1.485	(6)	
N(1)-C(9)	1.456	(6)	
C(1)-C(2)	1.431	(7)	
C(1)-H(1)	0.978	(5)	
C(2)-H(2)	0.963	(5)	
C(3)-H(3)	0.969	(5)	
C(4)-H(4)	0.977	(4)	
C(6)-C(7)	1.531	(8)	
C(6)-H(6)	0.959	(5)	
C(7)-H(8)	0.956	(5)	
C(8)-H(10)	0.957	(5)	
C(9)-H(12)	0.969	(6)	
C(9)-H(14)	0.957	(5)	
H(15)-N(1B)	1.318	(4)	
Co(1)-C(2)	2.024	(5)	
Co(1)-C(4)	2.036	(5)	
Co(1)-C(1A)	2.029	(5)	
Co(1)-C(3A)	2.035	(5)	
Co(1)-C(5A)	2.036	(5)	
P(1)-F(2)	1.592	(3)	
P(1)-F(4)	1.588	(3)	
P(1)-F(6)	1.576	(4)	
N(1)-C(8)	1.482	(6)	
N(1)-H(15A)	1.318	(4)	
C(1)-C(5)	1.429	(6)	
C(2)-C(3)	1.402	(6)	
C(3)-C(4)	1.421	(6)	
C(4)-C(5)	1.408	(6)	
C(5)-C(6)	1.491	(6)	
C(6)-H(5)	0.960	(5)	
C(7)-H(7)	0.958	(4)	
C(8)-H(9)	0.956	(5)	
C(8)-H(11)	0.964	(6)	
C(9)-H(13)	0.953	(5)	
H(15)-N(1A)	1.318	(4)	

Table 3. Bond angles (°)

C(1)-Co(1)-C(2)	41.4(2)
C(2)-Co(1)-C(3)	40.4(2)
C(2)-Co(1)-C(4)	68.6(2)
C(1)-Co(1)-C(5)	41.2(2)
C(3)-Co(1)-C(5)	68.9(2)
C(1)-Co(1)-C(1A)	180.0(1)
C(3)-Co(1)-C(1A)	111.6(2)
C(5)-Co(1)-C(1A)	138.8(2)
C(2)-Co(1)-C(2A)	180.0(1)
C(4)-Co(1)-C(2A)	111.4(2)
C(1A)-Co(1)-C(2A)	41.4(2)
C(2)-Co(1)-C(3A)	139.6(2)
C(4)-Co(1)-C(3A)	139.1(2)
C(1A)-Co(1)-C(3A)	68.4(2)
C(1)-Co(1)-C(4A)	111.9(2)
C(3)-Co(1)-C(4A)	139.1(2)
C(5)-Co(1)-C(4A)	139.5(2)
C(2A)-Co(1)-C(4A)	68.6(2)
C(1)-Co(1)-C(5A)	138.8(2)
C(3)-Co(1)-C(5A)	111.1(2)
C(5)-Co(1)-C(5A)	180.0(1)
C(2A)-Co(1)-C(5A)	69.8(2)
C(4A)-Co(1)-C(5A)	40.5(2)
F(1)-P(1)-F(3)	89.9(2)
F(1)-P(1)-F(4)	90.4(2)
F(3)-P(1)-F(4)	90.4(2)
F(2)-P(1)-F(5)	90.3(2)
F(4)-P(1)-F(5)	89.9(2)
F(2)-P(1)-F(6)	90.2(2)
F(4)-P(1)-F(6)	90.0(2)
C(7)-N(1)-C(8)	111.5(4)
C(8)-N(1)-C(9)	111.9(4)
C(8)-N(1)-H(15A)	106.1(3)
Co(1)-C(1)-C(2)	69.1(3)
C(2)-C(1)-C(5)	108.5(4)
C(2)-C(1)-H(1)	125.7(4)
Co(1)-C(2)-C(1)	69.5(3)
C(1)-C(2)-C(3)	107.4(4)
C(1)-C(2)-H(2)	126.7(4)
Co(1)-C(3)-C(2)	69.4(3)
C(2)-C(3)-C(4)	108.3(4)
C(2)-C(3)-H(3)	126.0(4)
Co(1)-C(4)-C(3)	69.5(3)
C(3)-C(4)-C(5)	109.1(4)
C(3)-C(4)-H(4)	125.7(4)
Co(1)-C(5)-C(1)	69.1(3)
C(1)-C(5)-C(4)	106.7(4)
C(1)-C(5)-C(6)	126.7(4)
C(5)-C(6)-C(7)	110.4(4)
C(7)-C(6)-H(5)	109.2(4)
C(7)-C(6)-H(6)	109.1(4)
N(1)-C(7)-C(6)	114.6(4)
C(6)-C(7)-H(7)	107.9(4)
C(6)-C(7)-H(8)	107.9(4)
N(1)-C(8)-H(9)	111.1(4)
H(9)-C(8)-H(10)	108.4(5)
C(1)-Co(1)-C(3)	68.4(2)
C(1)-Co(1)-C(4)	68.1(2)
C(3)-Co(1)-C(4)	40.9(2)
C(2)-Co(1)-C(5)	69.8(2)
C(4)-Co(1)-C(5)	40.5(2)
C(2)-Co(1)-C(1A)	138.6(2)
C(4)-Co(1)-C(1A)	111.9(2)
C(1)-Co(1)-C(2A)	138.6(2)
C(3)-Co(1)-C(2A)	139.6(2)
C(5)-Co(1)-C(2A)	110.2(2)
C(1)-Co(1)-C(3A)	111.6(2)
C(3)-Co(1)-C(3A)	180.0(1)
C(5)-Co(1)-C(3A)	111.1(2)
C(2A)-Co(1)-C(3A)	40.4(2)
C(2)-Co(1)-C(4A)	111.4(2)
C(4)-Co(1)-C(4A)	180.0(1)
C(1A)-Co(1)-C(4A)	68.1(2)
C(3A)-Co(1)-C(4A)	40.9(2)
C(2)-Co(1)-C(5A)	110.2(2)
C(4)-Co(1)-C(5A)	139.5(2)
C(1A)-Co(1)-C(5A)	41.2(2)
C(3A)-Co(1)-C(5A)	68.9(2)
F(1)-P(1)-F(2)	89.3(2)
F(2)-P(1)-F(3)	89.3(2)
F(2)-P(1)-F(4)	179.7(2)
F(1)-P(1)-F(5)	89.6(2)
F(3)-P(1)-F(5)	179.3(2)
F(1)-P(1)-F(6)	179.6(2)
F(3)-P(1)-F(6)	90.1(2)
F(5)-P(1)-F(6)	90.5(2)
C(7)-N(1)-C(9)	112.3(4)
C(7)-N(1)-H(15A)	109.3(3)
C(9)-N(1)-H(15A)	105.3(4)
Co(1)-C(1)-C(5)	69.7(3)
Co(1)-C(1)-H(1)	127.4(4)
C(5)-C(1)-H(1)	125.8(4)
Co(1)-C(2)-C(3)	70.2(3)
Co(1)-C(2)-H(2)	125.0(4)
C(3)-C(2)-H(2)	125.9(5)
Co(1)-C(3)-C(4)	69.6(3)
Co(1)-C(3)-H(3)	127.8(4)
C(4)-C(3)-H(3)	125.7(4)
Co(1)-C(4)-C(5)	69.8(3)
Co(1)-C(4)-H(4)	127.0(4)
C(5)-C(4)-H(4)	125.2(4)
Co(1)-C(5)-C(4)	69.8(3)
Co(1)-C(5)-C(6)	125.6(3)
C(4)-C(5)-C(6)	126.6(4)
C(5)-C(6)-H(5)	110.2(4)
C(5)-C(6)-H(6)	110.0(4)
H(5)-C(6)-H(6)	107.9(5)
N(1)-C(7)-H(7)	108.9(4)
N(1)-C(7)-H(8)	109.1(4)
H(7)-C(7)-H(8)	108.2(4)
N(1)-C(8)-H(10)	111.1(5)
N(1)-C(8)-H(11)	110.5(5)

H(9)-C(8)-H(11)	107.8(5)	H(10)-C(8)-H(11)	107.7(5)
N(1)-C(9)-H(12)	110.4(5)	N(1)-C(9)-H(13)	111.6(4)
H(12)-C(9)-H(13)	107.6(5)	N(1)-C(9)-H(14)	111.2(4)
H(12)-C(9)-H(14)	107.3(5)	H(13)-C(9)-H(14)	108.6(5)
N(1A)-H(15)-N(1B)	180.0(1)		

Table 4. Anisotropic displacement coefficients ($Å^2 x 10^3$)

	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
Co(1)	32(1)	18(1)	10(1)	4(1)	5(1)	4(1)
P(1)	27(1)	26(1)	27(1)	5(1)	6(1)	0(1)
F(1)	70(1)	91(1)	29(1)	19(1)	10(1)	14(1)
F(2)	26(1)	54(1)	68(1)	8(1)	5(1)	2(1)
F(3)	51(1)	27(1)	80(1)	3(1)	-1(1)	3(1)
F(4)	23(1)	47(1)	55(1)	8(1)	9(1)	10(1)
F(5)	46(1)	29(1)	108(1)	3(1)	7(1)	10(1)
F(6)	75(1)	85(1)	28(1)	21(1)	2(1)	-3(1)
N(1)	34(1)	29(1)	12(1)	11(1)	6(1)	1(1)
C(1)	43(1)	26(1)	29(1)	-1(1)	8(1)	21(1)
C(2)	54(1)	29(1)	27(1)	8(1)	0(1)	15(1)
C(3)	44(1)	34(1)	25(1)	21(1)	5(1)	10(1)
C(4)	41(1)	26(1)	19(1)	16(1)	4(1)	9(1)
C(5)	36(1)	17(1)	17(1)	6(1)	0(1)	11(1)
C(6)	46(1)	35(1)	15(1)	18(1)	11(1)	10(1)
C(7)	37(1)	29(1)	13(1)	11(1)	9(1)	5(1)
C(8)	62(1)	38(1)	36(1)	-4(1)	4(1)	7(1)
C(9)	43(1)	54(1)	31(1)	25(1)	11(1)	15(1)

The anisotropic displacement factor exponent takes the form:

$-2\pi^2(h^2a^{*2}U_{11} + \ldots + 2hka^*b^*U_{12})$

Table 5. H-Atom coordinates ($x10^4$) and isotropic displacement coefficients ($Å^2 x10^3$)

	x	y	z	U
H(1)	7776	7338	-774	41
H(2)	5552	8832	789	47
H(3)	1763	7462	441	42
H(4)	1627	5002	-1285	37
H(5)	7056	4236	-2370	42
H(6)	4840	3160	-2474	42
H(7)	5848	6469	-3199	35
H(8)	3542	5605	-3209	35
H(9)	2362	2747	-4431	57
H(10)	3909	1612	-5214	57
H(11)	4026	1502	-4077	57
H(12)	7886	2744	-3934	51
H(13)	7699	2629	-5097	51
H(14)	8483	4548	-4350	51
H(15)	5000	5000	5000	50(1)