Inorganic Chemistry
including bioinorganic chemistry
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Table S1. STRUCTURE DETERMINATION SUMMARY

Crystal Data

Empirical Formula	$C\ H_6\ Cs\ O_7\ P_2\ V_{0.5000}$
Color; Habit	Blue plate
Crystal size (mm)	0.18 x 0.17 x 0.09
Crystal System	Monoclinic
Space Group	C2
Unit Cell Dimensions	a = 10.991(2) Å
	b = 10.161(2) Å
	c = 7.4450(10) Å
	β = 92.97(3)°
Volume	830.3(2) $Å^3$
Z	4
Formula weight	348.4
Density(calc.)	2.787 Mg/m^3
Absorption Coefficient	5.367 mm^{-1}
F(000)	650

Data Collection

Diffractometer Used	Rigaku AFC5S
Radiation	MoKα (λ = 0.71073 Å)
Temperature (K)	253
Monochromator	Highly oriented graphite crystal
2θ Range	2.0 to 50.0^{o}
Scan Type	2θ-θ
Scan Speed	Variable; 4.00 to 12.00^{o}/min. in ω
Scan Range (ω)	1.31^{o} plus Kα-separation
Background Measurement	Stationary crystal and stationary counter at beginning and end of scan, each for 50.0% of total scan time
Standard Reflections	5 measured every 150 reflections
Index Ranges	$0 \leq h \leq 15$, $0 \leq k \leq 14$ $-10 \leq l \leq 10$
Reflections Collected	1276
Independent Reflections	1276 (R_{int} = 3.03%)
Observed Reflections	627 ($F > 6.0\sigma(F)$)
Absorption Correction	Based on ψ scans for 5 reflections

Solution and Refinement

System Used	Siemens SHELXTL PLUS (PC Version)
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	$w^{-1} = \sigma^2(F) + 0.0002F^2$
Number of Parameters Refined	64
Final R Indices (obs. data)	R = 4.76 %, wR = 4.87 %
Goodness-of-Fit	1.28
Largest and Mean Δ/σ	0.009, 0.001
Data-to-Parameter Ratio	9.8:1
Largest Difference Peak	1.09 $e\text{Å}^{-3}$
Largest Difference Hole	-0.92 $e\text{Å}^{-3}$

Table S2. Atomic coordinates ($x10^4$) and equivalent isotropic displacement coefficients ($Å^2 x10^3$)

	x	y	z	U(eq)
Cs(1)	0	0	0	40(2)
Cs(2)	0	5569	0	43(2)
V(1)	0	-2213(17)	5000	33(1)
P(1)	2429(3)	-2257(14)	2583(5)	17(1)
P(2)	2761(4)	-1965(10)	6667(6)	25(3)
O(1)	0	-3846(14)	5000	70(6)
O(2)	0	-220(24)	4861(22)	23(6)
O(3)	1080(8)	-2150(33)	2941(12)	24(2)
O(4)	2676(12)	-1204(14)	1161(18)	28(3)
O(5)	2909(13)	-3584(16)	2055(21)	39(4)
O(6)	1421(8)	-2052(19)	6829(12)	19(2)
O(7)	3122(12)	-538(15)	6610(19)	36(4)
O(8)	3529(11)	-2647(11)	8240(16)	28(3)
C(1)	3189(18)	-2795(20)	4632(26)	35(5)

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

Table S3. Bond lengths (Å)

Cs(1)-O(2)	3.626 (23)	Cs(1)-O(3)	3.272 (23)
Cs(1)-O(4)	3.260 (13)	Cs(1)-O(2A)	3.626 (23)
Cs(1)-O(3A)	3.272 (23)	Cs(1)-O(4A)	3.260 (13)
Cs(1)-O(5)	3.171 (16)	Cs(1)-O(5B)	3.171 (16)
Cs(1)-O(6)	3.570 (13)	Cs(1)-O(6A)	3.570 (13)
Cs(1)-O(8)	3.135 (11)	Cs(1)-O(8A)	3.135 (11)
Cs(2)-O(1)	3.770 (1)	Cs(2)-O(1A)	3.770 (1)
Cs(2)-O(3)	3.363 (24)	Cs(2)-O(3A)	3.363 (24)
Cs(2)-O(4)	3.278 (14)	Cs(2)-O(4A)	3.278 (14)
Cs(2)-O(5)	3.577 (15)	Cs(2)-O(5A)	3.577 (15)
Cs(2)-O(6)	3.773 (14)	Cs(2)-O(6A)	3.773 (14)
Cs(2)-O(7)	3.370 (14)	Cs(2)-O(7A)	3.370 (14)
V(1)-O(1)	1.660 (17)	V(1)-O(2)	2.027 (20)
V(1)-O(3)	1.988 (9)	V(1)-O(6)	2.025 (9)
V(1)-O(3A)	1.988 (9)	V(1)-O(6A)	2.025 (9)
P(1)-O(3)	1.524 (10)	P(1)-O(4)	1.539 (17)
P(1)-O(5)	1.508 (20)	P(1)-C(1)	1.787 (20)
P(2)-O(6)	1.487 (10)	P(2)-O(7)	1.504 (18)
P(2)-O(8)	1.569 (13)	P(2)-C(1)	1.817 (21)

Table S4. Bond angles (°)

O(1)-V(1)-O(2)	177.1(15)	O(1)-V(1)-O(3)	91.8(11)
O(2)-V(1)-O(3)	85.8(16)	O(1)-V(1)-O(6)	94.6(7)
O(2)-V(1)-O(6)	87.2(12)	O(3)-V(1)-O(6)	92.6(4)
O(1)-V(1)-O(3A)	91.8(11)	O(2)-V(1)-O(3A)	90.5(16)
O(3)-V(1)-O(3A)	176.3(21)	O(6)-V(1)-O(3A)	87.1(4)
O(1)-V(1)-O(6A)	94.6(7)	O(2)-V(1)-O(6A)	83.5(12)
O(3)-V(1)-O(6A)	87.1(4)	O(6)-V(1)-O(6A)	170.8(14)
O(3A)-V(1)-O(6A)	92.6(4)	O(3)-P(1)-O(4)	106.2(12)
O(3)-P(1)-O(5)	117.8(15)	O(4)-P(1)-O(5)	111.4(8)
O(3)-P(1)-C(1)	106.8(9)	O(4)-P(1)-C(1)	135.1(9)
O(5)-P(1)-C(1)	78.1(10)	O(6)-P(2)-O(7)	108.9(10)
O(6)-P(2)-O(8)	114.2(8)	O(7)-P(2)-O(8)	108.3(7)
O(6)-P(2)-C(1)	109.9(9)	O(7)-P(2)-C(1)	110.1(9)
O(8)-P(2)-C(1)	105.4(9)	V(1)-O(3)-P(1)	139.3(6)
V(1)-O(6)-P(2)	133.1(6)	P(1)-C(1)-P(2)	116.1(11)

Table S5. Anisotropic displacement coefficients ($Å^2 x10^3$)

	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
Cs(1)	18(1)	63(3)	36(3)	0	-13(1)	0
Cs(2)	56(2)	43(3)	31(3)	0	0(2)	0
V(1)	15(2)	71(3)	14(2)	0	2(1)	0
P(1)	21(2)	16(3)	15(2)	-9(4)	3(1)	-2(5)
P(2)	19(2)	46(7)	12(2)	1(3)	2(2)	3(3)

The anisotropic displacement exponent takes the form:

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

Table S6. H-Atom coordinates ($x10^4$) and isotropic displacement coefficients ($Å^2 x10^3$)

	x	y	z	U
H(2A)	632	41	4355	80
H(2B)	-632	41	4258	80
H(1A)	4048	-2669	4527	80
H(1B)	3050	-3722	4762	80

Table S7. STRUCTURE DETERMINATION SUMMARY

Crystal Data

Empirical Formula	$C_2 H_6 Cs_2 O_{14} P_4 V_2$
Color; Habit	Blue plate
Crystal size (mm)	0.24 x 0.25 x 0.11
Crystal System	Monoclinic
Space Group	C2
Unit Cell Dimensions	a = 10.212(2) Å
	b = 10.556(2) Å
	c = 14.699(3) Å
	β = 94.57(2)°
Volume	1579.5(8) $Å^3$
Z	4
Formula weight	746.8
Density(calc.)	3.141 Mg/m^3
Absorption Coefficient	6.201 mm^{-1}
F(000)	1382.40

Data Collection

Diffractometer Used	Rigaku AFC5S
Radiation	MoKα (λ = 0.71073 Å)
Temperature (K)	253
Monochromator	Highly oriented graphite crystal
2θ Range	2.0 to 55.0°
Scan Type	ω-2θ
Scan Speed	Variable; 2.00 to 8.00°/min. in ω
Scan Range (2θ)	(1.31 + tanθ)$^{\circ}$
Background Measurement	Stationary crystal and stationary counter at beginning and end of scan, each for 50% of total scan time
Standard Reflections	3 measured every 100 reflections
Index Ranges	$0 \le h \le 14$, $0 \le k \le 14$ $-20 \le l \le 20$
Reflections Collected	2553
Independent Reflections	2428 (R_{int} = 6.36%)
Observed Reflections	2148 ($F > 6.0\sigma(F)$)
Absorption Correction	Based on ψ scans for 5 reflections

Solution and Refinement

System Used	Siemens SHELXTL PLUS (PC Version)
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	$w^{-1} = \sigma^2(F) + 0.0001F^2$
Number of Parameters Refined	217
Final R Indices (obs. data)	R = 4.56 %, wR = 5.18 %
R Indices (all data)	R = 5.19 %, wR = 5.24 %
Goodness-of-Fit	2.37
Largest and Mean Δ/σ	0.097, 0.002
Data-to-Parameter Ratio	9.9:1
Largest Difference Peak	1.07 $e\text{Å}^{-3}$
Largest Difference Hole	-1.46 $e\text{Å}^{-3}$

Table S8. Atomic Positional Parameters ($x10^4$) and Isotropic Temperature Factors ($Å^2 \times 10^3$) for $Cs[VO(HO_3PCH_2PO_3)]$ (2).

	x	y	z	U(eq)
Cs(2)	0	8782	0	23(1)
Cs(1)	0	8843(2)	5000	39(1)
Cs(3)	0	4673(1)	5000	22(1)
Cs(4)	0	4630(2)	0	41(1)
V(1A)	-35(5)	6440(7)	2531(3)	11(1)
V(1B)	75(22)	6443(31)	7558(14)	13(7)
V(2A)	2179(5)	9170(5)	2456(3)	9(1)
V(2B)	2768(11)	4186(12)	7438(7)	6(3)
P(1)	2290(3)	12021(3)	13338(2)	10(1)
P(2)	2290(3)	11794(3)	11348(2)	10(1)
P(3)	-2790(3)	6535(3)	8332(2)	11(1)
P(4)	2223(3)	11756(3)	6346(2)	11(1)
O(1)	737(7)	8570(8)	12438(6)	13(2)
O(2)	632(7)	5046(8)	7424(6)	15(2)
O(3)	1222(7)	7260(9)	6635(6)	14(2)
O(4)	1972(7)	10609(9)	3354(6)	12(2)
O(5)	1705(8)	2704(8)	4133(6)	17(2)
O(6)	1217(7)	7023(9)	8558(5)	12(2)
O(7)	1974(7)	10394(8)	11447(6)	13(2)
O(8)	1682(7)	12332(9)	10444(6)	29(3)
O(9)	-1330(7)	6184(8)	8369(6)	14(2)
O(10)	-2997(7)	7978(9)	8341(6)	13(2)
O(11)	1554(7)	10921(8)	9114(6)	13(2)
O(12)	1314(7)	6410(9)	3580(6)	14(2)
O(13)	2018(7)	3183(9)	6446(6)	14(2)
O(14)	1542(7)	11302(9)	5438(6)	16(2)
C(1)	1596(10)	12660(11)	12262(8)	11(3)
C(2)	1489(10)	10954(12)	7243(9)	13(3)

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

Table S9. Bond lengths (Å)

Cs(2)-O(1)	3.609	(8)	Cs(2)-O(1A)	3.609	(8)
Cs(2)-O(6)	3.147	(8)	Cs(2)-O(6A)	3.147	(8)
Cs(2)-O(7)	3.286	(8)	Cs(2)-O(7A)	3.286	(8)
Cs(2)-O(11)	3.105	(8)	Cs(2)-O(11A)	3.105	(8)
Cs(1)-O(3)	3.106	(8)	Cs(1)-O(4)	3.763	(9)
Cs(1)-O(12)	3.633	(9)	Cs(1)-O(14)	3.078	(9)
Cs(1)-P(1D)	4.031	(3)	Cs(1)-O(3A)	3.106	(8)
Cs(1)-O(4A)	3.763	(9)	Cs(1)-O(5)	3.703	(8)
Cs(1)-O(5A)	3.703	(8)	Cs(1)-O(12)	3.633	(9)
Cs(1)-O(14)	3.078	(9)	Cs(3)-O(2)	3.592	(9)
Cs(3)-O(3)	3.783	(9)	Cs(3)-O(5)	3.055	(9)
Cs(3)-O(12)	3.156	(9)	Cs(3)-O(13)	3.247	(8)
Cs(3)-O(2A)	3.592	(9)	Cs(3)-O(3A)	3.783	(9)
Cs(3)-O(5A)	3.055	(9)	Cs(3)-O(12A)	3.156	(9)
Cs(3)-O(13A)	3.247	(8)	Cs(4)-O(6)	3.584	(8)
Cs(4)-O(6A)	3.584	(8)	Cs(4)-O(8)	3.013	(9)
Cs(4)-O(8A)	3.013	(9)	Cs(4)-O(9)	3.125	(8)
Cs(4)-O(9A)	3.125	(8)	Cs(4)-O(10)	3.737	(9)
Cs(4)-O(10A)	3.737	(9)	V(1)-O(12)	1.985	(9)
V(1)-O(1)	2.390	(11)	V(1)-O(2)	1.596	(11)
V(1)-O(3)	1.989	(10)	V(1)-O(6)	2.022	(9)
V(1)-O(9)	2.016	(10)	V(2)-O(4)	2.034	(11)
V(2)-O(1)	1.601	(9)	V(2)-O(2)	2.413	(9)
V(2)-O(7)	1.966	(10)	V(2)-O(10)	1.952	(10)
V(2)-O(13)	2.038	(10)	P(1)-C(1)	1.810	(11)
P(1)-O(3)	1.538	(7)	P(1)-O(4)	1.526	(10)
P(1)-O(5)	1.534	(10)	P(2)-O(7)	1.523	(9)
P(2)-O(8)	1.531	(9)	P(2)-C(1)	1.815	(12)
P(2)-O(6)	1.539	(7)	P(3)-O(9)	1.533	(8)
P(3)-O(10)	1.538	(10)	P(3)-O(11)	1.521	(9)
P(3)-C(2)	1.814	(13)	P(4)-O(14)	1.531	(9)
P(4)-C(2)	1.782	(13)	P(4)-O(12)	1.534	(8)
P(4)-O(13)	1.529	(10)			

Table S10. Bond angles (°)

O(1)-Cs(2)-O(1A)	172.9(3)	O(1)-Cs(2)-O(6)	124.7(2)
O(6)-Cs(2)-O(6A)	107.7(3)	O(1)-Cs(2)-O(7)	138.0(2)
O(6)-Cs(2)-O(7)	119.1(2)	O(6)-Cs(2)-O(7A)	97.1(2)
O(7)-Cs(2)-O(7A)	117.6(3)	O(1)-Cs(2)-O(11)	112.8(2)
O(1)-Cs(2)-O(11A)	72.7(2)	O(6)-Cs(2)-O(11)	84.5(2)
O(6)-Cs(2)-O(11A)	162.5(2)	O(7)-Cs(2)-O(11)	65.6(2)
O(7)-Cs(2)-O(11)	70.1(2)	O(11)-Cs(2)-O(11A)	86.7(3)
O(3)-Cs(1)-O(4)	124.1(2)	O(3)-Cs(1)-O(12)	85.4(2)
O(4)-Cs(1)-O(12)	74.9(2)	O(3)-Cs(1)-O(14)	97.1(2)
O(4)-Cs(1)-O(14)	54.9(2)	O(12)-Cs(1)-O(14)	120.7(2)
O(3)-Cs(1)-O(3A)	114.9(3)	O(4)-Cs(1)-O(3A)	88.4(2)
O(14)-Cs(1)-O(3A)	141.5(2)	O(3)-Cs(1)-O(4A)	88.4(2)
O(4)-Cs(1)-O(4A)	120.6(3)	O(12)-Cs(1)-O(4)	163.9(2)
O(14)-Cs(1)-O(4)	74.9(2)	O(4)-Cs(1)-O(5)	117.2(2)
O(12)-Cs(1)-O(5)	86.5(2)	O(14)-Cs(1)-O(5)	141.4(2)
O(2)-Cs(3)-O(3)	42.8(2)	O(2)-Cs(3)-O(5)	115.2(2)
O(3)-Cs(3)-O(5)	125.9(2)	O(2)-Cs(3)-O(12)	122.7(2)
O(3)-Cs(3)-O(12)	82.2(2)	O(5)-Cs(3)-O(12)	80.4(2)
O(2)-Cs(3)-O(13)	49.4(2)	O(3)-Cs(3)-O(13)	76.9(2)
O(5)-Cs(3)-O(13)	65.8(2)	O(12)-Cs(3)-O(13)	115.7(2)
O(2)-Cs(3)-O(2A)	167.4(3)	O(3)-Cs(3)-O(2A)	125.1(2)
O(5)-Cs(3)-O(2A)	74.0(2)	O(12)-Cs(3)-O(2A)	48.1(2)
O(13)-Cs(3)-O(2A)	139.2(2)	O(2)-Cs(3)-O(3A)	125.1(2)
O(3)-Cs(3)-O(3A)	87.6(3)	O(5)-Cs(3)-O(3A)	113.3(2)
O(12)-Cs(3)-O(3A)	45.3(2)	O(13)-Cs(3)-O(3A)	157.8(2)
O(6)-Cs(4)-O(6A)	90.3(3)	O(6)-Cs(4)-O(8A)	118.5(2)
O(6)-Cs(4)-O(8A)	131.1(2)	O(8)-Cs(4)-O(8A)	72.8(3)
O(6)-Cs(4)-O(9)	86.2(2)	O(8)-Cs(4)-O(9)	142.6(2)
O(8)-Cs(4)-O(9A)	92.9(2)	O(6)-Cs(4)-O(10)	161.5(2)
O(8)-Cs(4)-O(10)	55.5(2)	O(8)-Cs(4)-O(10A)	79.4(2)
O(9)-Cs(4)-O(10A)	88.4(2)	O(9A)-Cs(4)-O(10A)	121.2(2)
O(12)-V(1)-O(1)	81.3(4)	O(12)-V(1)-O(2)	101.3(5)
O(1)-V(1)-O(2)	176.8(4)	O(12)-V(1)-O(3)	87.0(4)
O(1)-V(1)-O(3)	81.2(4)	O(2)-V(1)-O(3)	97.1(5)
O(12)-V(1)-O(6)	162.5(5)	O(1)-V(1)-O(6)	81.2(4)
O(2)-V(1)-O(6)	96.2(5)	O(3)-V(1)-O(6)	90.1(4)
O(12)-V(1)-O(9)	92.0(4)	O(1)-V(1)-O(9)	80.8(4)
O(2)-V(1)-O(9)	100.9(5)	O(3)-V(1)-O(9)	161.9(5)
O(6)-V(1)-O(9)	85.5(4)	O(4)-V(2)-O(1)	99.4(4)
O(4)-V(2)-O(2)	79.1(3)	O(1)-V(2)-O(2)	176.7(5)
O(4)-V(2)-O(7)	89.4(4)	O(1)-V(2)-O(7)	101.8(4)
O(2)-V(2)-O(7)	81.1(4)	O(4)-V(2)-O(10)	160.5(4)
O(1)-V(2)-O(10)	99.9(5)	O(2)-V(2)-O(10)	81.5(4)
O(7)-V(2)-O(10)	89.7(4)	O(4)-V(2)-O(13)	85.7(4)
O(1)-V(2)-O(13)	97.0(4)	O(2)-V(2)-O(13)	80.0(3)
O(7)-V(2)-O(13)	161.1(4)	O(10)-V(2)-O(13)	88.9(4)
C(1)-P(1)-O(3)	106.2(5)	C(1)-P(1)-O(4)	108.0(5)
O(3)-P(1)-O(4)	111.8(5)	C(1)-P(1)-O(5)	109.9(5)
O(3)-P(1)-O(5)	110.3(5)	O(4)-P(1)-O(5)	110.5(5)
O(7)-P(2)-O(8)	111.6(5)	O(7)-P(2)-C(1)	108.7(5)
O(8)-P(2)-C(1)	107.5(5)	O(7)-P(2)-O(6)	111.1(5)
O(8)-P(2)-O(6)	110.5(5)	C(1)-P(2)-O(6)	107.3(5)
O(9)-P(3)-O(10)	111.9(5)	O(9)-P(3)-O(11)	110.8(5)
O(10)-P(3)-O(11)	110.3(5)	O(9)-P(3)-C(2)	106.0(5)
O(10)-P(3)-C(2)	107.2(5)	O(11)-P(3)-C(2)	110.5(5)
O(14)-P(4)-C(2)	107.9(5)	O(14)-P(4)-O(12)	111.1(5)

C(2)-P(4)-O(12)	107.4(5)	O(14)-P(4)-O(13)	109.6(5)
C(2)-P(4)-O(13)	109.1(5)	O(12)-P(4)-O(13)	111.6(5)
V(1)-O(1)-V(2)	132.7(5)	V(2)-O(2)-V(1)	135.2(6)
V(1)-O(3)-P(1)	121.9(8)	V(2)-O(4)-P(1)	133.3(6)
V(1)-O(6)-P(2)	124.1(9)	P(2)-O(7)-V(2)	131.1(6)
V(1)-O(9)-P(3)	134.7(9)	P(3)-O(10)-V(2)	124.6(5)
V(1)-O(12)-P(4)	131.6(5)	V(2)-O(13)-P(4)	124.7(6)
P(1)-C(1)-P(2)	108.0(6)	P(4)-C(2)-P(3)	109.1(6)

Table S11. Anisotropic displacement coefficients ($Å^2 x10^3$)

	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
Cs(2)	28(1)	14(1)	27(1)	0	10(1)	0
Cs(1)	50(1)	15(1)	49(1)	0	-27(1)	0
Cs(3)	27(1)	14(1)	25(1)	0	10(1)	0
Cs(4)	51(1)	16(1)	51(1)	0	-29(1)	0
P(1)	12(1)	5(1)	12(1)	0(1)	3(1)	-1(1)
P(2)	12(1)	7(1)	11(1)	1(1)	1(1)	0(1)
P(3)	11(1)	8(1)	13(1)	-1(1)	2(1)	1(1)
P(4)	12(1)	10(1)	12(2)	0(1)	0(1)	-1(1)
O(1)	15(4)	10(4)	14(4)	-4(3)	3(3)	-7(3)
O(2)	12(3)	10(4)	24(5)	2(3)	4(3)	-3(3)
O(3)	12(3)	12(4)	18(5)	4(3)	4(3)	8(4)
O(4)	12(3)	8(4)	18(4)	-1(3)	7(3)	0(3)
O(5)	23(4)	10(4)	18(5)	0(3)	7(3)	-2(3)
O(6)	14(3)	15(4)	7(4)	4(3)	0(3)	-1(3)
O(7)	18(4)	10(4)	11(4)	-2(3)	3(3)	2(3)
O(8)	38(4)	20(4)	28(5)	4(4)	-3(3)	3(4)
O(9)	11(3)	18(5)	12(4)	0(3)	2(3)	3(3)
O(10)	18(4)	9(4)	12(4)	1(3)	4(3)	-3(3)
O(11)	19(4)	6(4)	16(4)	-5(3)	10(3)	1(3)
O(12)	14(3)	11(4)	16(4)	1(3)	1(3)	0(4)
O(13)	13(3)	7(4)	22(5)	2(3)	-1(3)	0(4)
O(14)	18(4)	18(4)	13(4)	-3(3)	-2(3)	0(3)
C(1)	17(5)	6(5)	9(5)	4(4)	-2(4)	-4(4)
C(2)	13(5)	10(5)	17(6)	0(4)	4(4)	-1(4)

The anisotropic displacement exponent takes the form:

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

Table S12. H-Atom coordinates ($x10^4$) and isotropic displacement coefficients ($Å^2 x10^3$)

	x	y	z	U
H(1A)	658	12567	12217	80
H(1B)	1817	13541	12224	80
H(2A)	1886	10245	7567	80
H(2B)	845	11332	7600	80

Table S13. STRUCTURE DETERMINATION SUMMARY

Crystal Data

Empirical Formula	$C_2 H_8 Cs O_{16} P_4 V_3$
Color; Habit	Blue plate
Crystal size (mm)	0.22 x 0.20 x 0.12
Crystal System	Monoclinic
Space Group	C2/m
Unit Cell Dimensions	a = 9.724(2) Å
	b = 8.136(2) Å
	c = 10.268(2) Å
	β = 103.75(3)°
Volume	789.1(4) $Å^3$
Z	2
Formula weight	697.7
Density(calc.)	2.936 Mg/m^3
Absorption Coefficient	4.504 mm^{-1}
F(000)	664

Data Collection

Diffractometer Used	Rigaku AFC5S
Radiation	MoKα (λ = 0.71073 Å)
Temperature (K)	253
Monochromator	Highly oriented graphite crystal
2θ Range	2.0 to 45.0°
Scan Type	ω-2θ
Scan Speed	Variable; 4.00 to 8.00°/min. in ω
Scan Range (2θ)	(1.31 + tanθ)°
Background Measurement	Stationary crystal and stationary counter at beginning and end of scan, each for 50% of total scan time
Standard Reflections	3 measured every 100 reflections
Index Ranges	$0 \le h \le 13$, $0 \le k \le 11$ $-14 \le l \le 14$
Reflections Collected	1295
Independent Reflections	1228 (R_{int} = 2.60%)
Observed Reflections	671 ($F > 6.0\sigma(F)$)
Absorption Correction	Based on ψ scans for 5 reflections

Solution and Refinement

System Used	Siemens SHELXTL PLUS (PC Version)
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	$w^{-1} = \sigma^2(F) + 0.0001F^2$
Number of Parameters Refined	78
Final R Indices (obs. data)	R = 6.02 %, wR = 6.77 %
Goodness-of-Fit	2.02
Largest and Mean Δ/σ	0.006, 0.001
Data-to-Parameter Ratio	8.6:1
Largest Difference Peak	1.26 $e\text{Å}^{-3}$
Largest Difference Hole	-1.01 $e\text{Å}^{-3}$

Table S14. Atomic coordinates ($x10^4$) and equivalent isotropic displacement coefficients ($Å^2 x10^3$)

	x	y	z	U(eq)
Cs	631(3)	0	277(2)	37(1)
V(1)	7637(3)	0	3024(3)	12(1)
V(2)	5000	0	0	23(2)
P(1)	4719(3)	1802(4)	2656(3)	27(1)
O(1)	4442(7)	1716(8)	1131(6)	18(2)
O(2)	3691(7)	-1726(9)	-3316(6)	22(2)
O(3)	882(10)	-1649(13)	-3111(8)	37(4)
O(4)	3059(10)	0	-1253(9)	14(3)
O(5)	1663(12)	0	-5166(11)	30(5)
C(1)	6095(21)	0	-3145(21)	37(5)

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

Table S15. Bond lengths (Å)

Cs-O(3)	3.790 (9)	Cs-O(4)	3.131 (11)
Cs-O(1)	3.119 (7)	Cs-O(1A)	3.031 (7)
Cs-O(3A)	3.804 (10)	V(1)-O(2)	1.978 (7)
V(1)-O(2A)	1.978 (7)	V(1)-O(3)	1.954 (10)
V(1)-O(3A)	1.954 (10)	V(1)-O(4)	1.783 (9)
V(1)-O(5)	2.142 (11)	V(2)-O(1)	1.973 (7)
V(2)-O(4)	2.016 (8)	V(2)-O(1A)	1.973 (7)
V(2)-O(1B)	1.973 (7)	V(2)-O(1C)	1.973 (7)
V(2)-O(4A)	2.016 (8)	P(1)-O(1)	1.526 (7)
P(1)-O(2)	1.535 (7)	P(1)-O(3)	1.510 (9)
P(1)-C(1)	1.793 (10)		

Table S16. Bond angles (°)

O(2)-V(1)-O(2A)	90.5(4)	O(2)-V(1)-O(3)	90.4(4)
O(2)-V(1)-O(3A)	168.7(3)	O(3)-V(1)-O(3A)	86.7(6)
O(2)-V(1)-O(4)	93.0(3)	O(3)-V(1)-O(4)	98.2(3)
O(2)-V(1)-O(5)	84.5(3)	O(3)-V(1)-O(5)	84.4(3)
O(4)-V(1)-O(5)	176.4(5)	O(1)-V(2)-O(4)	91.7(3)
O(1)-V(2)-O(1A)	180.0(1)	O(4)-V(2)-O(1)	88.3(3)
O(1)-V(2)-O(1A)	89.9(4)	O(1)-V(2)-O(1A)	90.1(4)
O(1)-P(1)-O(2)	111.4(4)	O(1)-P(1)-O(3)	111.7(4)
O(2)-P(1)-O(3)	108.7(5)	O(1)-P(1)-C(1)	105.5(7)
O(2)-P(1)-C(1)	108.0(6)	O(3)-P(1)-C(1)	111.5(7)
V(2)-O(1)-P(1)	129.1(4)	V(1)-O(2)-P(1)	125.9(4)
V(1)-O(3)-P(1)	153.4(5)	V(2)-O(4)-V(1)	136.2(6)
P(1)-C(1)-P(1A)	109.6(12)		

Table S17. Anisotropic displacement coefficients ($Å^2 x10^3$)

	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
Cs	41(2)	17(1)	31(1)	0	28(1)	0
V(1)	15(1)	18(1)	26(1)	0	8(1)	0
V(2)	24(4)	16(2)	21(2)	0	17(2)	0
P(1)	22(2)	21(2)	12(1)	17(2)	-9(1)	-14(1)
O(1)	24(4)	15(3)	10(3)	14(3)	-6(3)	-6(3)
O(2)	20(4)	16(4)	20(4)	19(3)	-14(3)	-10(3)
O(3)	33(7)	37(8)	28(4)	-21(6)	-17(4)	12(5)
O(4)	17(5)	30(6)	15(4)	0	-2(4)	0
O(5)	19(6)	30(12)	11(6)	0	1(5)	0

The anisotropic displacement exponent takes the form:

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

Table S18. H-Atom coordinates ($x10^4$) and isotropic displacement coefficients ($Å^2 x10^3$)

	x	y	z	U
H(4A)	1122(253)	2040(1606)	-5113(229)	80
H(4B)	1667(290)	-1731(390)	-6082(270)	80
H(1)	6152(217)	0	-4084(211)	80
H(2)	6951(224)	0	-2844(226)	80

Table S19. STRUCTURE DETERMINATION SUMMARY

Crystal Data

Empirical Formula	$C_2 H_{10} O_{14} P_4 V_2$
Color; Habit	Green block
Crystal size (mm)	0.19 x 0.18 x 0.20
Crystal System	Monoclinic
Space Group	$P2_1/n$
Unit Cell Dimensions	a = 5.3410(10) Å
	b = 11.516(2) Å
	c = 10.558(2) Å
	β = 99.89(1)°
Volume	639.7(2) Å^3
Z	2
Formula weight	483.9
Density(calc.)	2.512 Mg/m^3
Absorption Coefficient	2.040 mm^{-1}
F(000)	480

Data Collection

Diffractometer Used	Rigaku AFC5S
Radiation	MoKα (λ = 0.71073 Å)
Temperature (K)	296
Monochromator	Highly oriented graphite crystal
2θ Range	2.0 to 55.0°
Scan Type	2θ–θ
Scan Speed	Variable; 4.00 to 16.00°/min. in ω
Scan Range (ω)	1.31° plus Kα-separation
Background Measurement	Stationary crystal and stationary counter at beginning and end of scan, each for 50.0% of total scan time
Standard Reflections	5 measured every 200 reflections
Index Ranges	$0 \le h \le 6$, $0 \le k \le 16$ $-14 \le l \le 14$
Reflections Collected	1567
Independent Reflections	1567 (R_{int} = 3.03%)
Observed Reflections	827 ($F > 6.0\sigma(F)$)
Absorption Correction	Based on ψ scans for 5 reflections

Solution and Refinement

System Used	Siemens SHELXTL PLUS (PC Version)
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	$w^{-1} = \sigma^2(F) + 0.0001F^2$
Number of Parameters Refined	103
Final R Indices (obs. data)	R = 4.72 %, wR = 5.00 %
Goodness-of-Fit	1.47
Largest and Mean Δ/σ	0.000, 0.000
Data-to-Parameter Ratio	8.0:1
Largest Difference Peak	0.68 $e\text{Å}^{-3}$
Largest Difference Hole	-0.82 $e\text{Å}^{-3}$

Table S20. Atomic coordinates ($x10^4$) and equivalent isotropic displacement coefficients ($Å^2 x10^3$)

	x	y	z	U(eq)
V(1)	0	0	5000	8(1)
V(2)	0	5000	5000	8(1)
P(1)	655(4)	305(2)	1871(2)	7(1)
P(2)	2224(3)	2521(2)	6194(2)	9(1)
O(1)	963(10)	264(5)	3326(5)	15(2)
O(2)	2987(10)	-170(5)	1426(5)	14(2)
O(3)	-1768(10)	-366(5)	1280(5)	11(2)
O(4)	351(10)	3304(5)	5373(5)	13(2)
O(5)	2397(10)	1301(5)	5660(5)	11(2)
O(6)	1478(10)	2394(5)	7576(5)	15(2)
O(7)	3114(10)	-1071(5)	5408(6)	17(2)
C(1)	346(15)	1817(7)	1378(8)	13(2)

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

Table S21. Bond lengths (Å)

V(1)-O(1)	1.947 (6)	V(1)-O(5)	2.016 (5)
V(1)-O(7)	2.056 (6)	V(1)-O(1A)	1.947 (6)
V(1)-O(5A)	2.016 (5)	V(1)-O(7A)	2.056 (6)
V(2)-O(4)	1.995 (6)	V(2)-O(2)	2.004 (6)
V(2)-O(2A)	2.004 (6)	V(2)-O(3)	2.045 (5)
V(2)-O(3A)	2.045 (5)	V(2)-O(4A)	1.995 (6)
P(1)-O(1)	1.518 (6)	P(1)-O(2)	1.507 (6)
P(1)-O(3)	1.544 (6)	P(1)-C(1)	1.816 (8)
P(2)-O(4)	1.506 (6)	P(2)-O(5)	1.523 (6)
P(2)-O(6)	1.584 (6)	P(2)-C(1)	1.813 (8)

Table S22. Bond angles (°)

O(1)-V(1)-O(5)	86.9(2)	O(1)-V(1)-O(7)	87.3(2)
O(5)-V(1)-O(7)	86.0(2)	O(1)-V(1)-O(1A)	180.0(1)
O(5)-V(1)-O(1A)	93.1(2)	O(7)-V(1)-O(1A)	92.7(2)
O(1)-V(1)-O(5A)	93.1(2)	O(5)-V(1)-O(5A)	180.0(1)
O(7)-V(1)-O(5A)	94.0(2)	O(1A)-V(1)-O(5A)	86.9(2)
O(1)-V(1)-O(7A)	92.7(2)	O(5)-V(1)-O(7A)	94.0(2)
O(7)-V(1)-O(7A)	180.0(1)	O(1A)-V(1)-O(7A)	87.3(2)
O(5A)-V(1)-O(7A)	86.0(2)	O(4)-V(2)-O(2)	90.5(2)
O(4)-V(2)-O(2A)	89.5(2)	O(2)-V(2)-O(2A)	180.0(1)
O(4)-V(2)-O(3)	88.2(2)	O(2)-V(2)-O(3)	89.1(2)
O(2A)-V(2)-O(3)	90.9(2)	O(4)-V(2)-O(3A)	91.8(2)
O(2)-V(2)-O(3A)	90.9(2)	O(2A)-V(2)-O(3A)	89.1(2)
O(3)-V(2)-O(3A)	180.0(1)	O(4)-V(2)-O(4A)	180.0(1)
O(2)-V(2)-O(4A)	89.5(2)	O(2A)-V(2)-O(4A)	90.5(2)
O(3)-V(2)-O(4A)	91.8(2)	O(3A)-V(2)-O(4A)	88.2(2)
O(1)-P(1)-O(2)	110.6(3)	O(1)-P(1)-O(3)	109.3(3)
O(2)-P(1)-O(3)	111.8(3)	O(1)-P(1)-C(1)	108.0(4)
O(2)-P(1)-C(1)	107.4(4)	O(3)-P(1)-C(1)	109.6(3)
O(4)-P(2)-O(5)	114.5(3)	O(4)-P(2)-O(6)	109.8(3)
O(5)-P(2)-O(6)	107.3(3)	O(4)-P(2)-C(1)	108.5(3)
O(5)-P(2)-C(1)	108.3(3)	O(6)-P(2)-C(1)	108.2(3)
V(1)-O(1)-P(1)	157.5(4)	P(1)-O(2)-V(2)	141.0(3)
P(1)-O(3)-V(2)	135.8(3)	V(2)-O(4)-P(2)	136.9(3)
V(1)-O(5)-P(2)	137.6(3)	P(1)-C(1)-P(2)	118.1(5)

Table S23. Anisotropic displacement coefficients ($Å^2 x10^3$)

	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
V(1)	8(1)	9(1)	8(1)	0(1)	2(1)	0(1)
V(2)	6(1)	9(1)	9(1)	0(1)	2(1)	-1(1)
P(1)	7(1)	8(1)	7(1)	1(1)	3(1)	0(1)
P(2)	9(1)	8(1)	10(1)	-1(1)	2(1)	-1(1)
O(1)	14(3)	21(3)	9(3)	-1(3)	0(2)	2(3)
O(2)	13(3)	12(3)	17(3)	5(2)	4(2)	4(2)
O(3)	5(3)	12(3)	15(3)	-5(2)	-2(2)	-1(2)
O(4)	10(3)	11(3)	20(3)	1(2)	4(2)	2(3)
O(5)	7(3)	11(3)	13(3)	0(2)	1(2)	-4(2)
O(6)	20(3)	13(3)	12(3)	-6(3)	5(2)	-5(3)
O(7)	13(3)	17(3)	21(3)	6(2)	3(3)	3(3)
C(1)	14(4)	5(3)	20(4)	0(3)	1(3)	6(3)

The anisotropic displacement exponent takes the form:

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

Table S24. H-Atom coordinates ($x10^4$) and isotropic displacement coefficients ($Å^2 x10^3$)

	x	y	z	U
H(6)	1336	2394	8470	80
H(7A)	4637	-842	5974	80
H(7B)	2965	-1901	5412	80
H(1A)	-827	2085	644	80
H(1B)	841	2456	1950	80