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Table S1. Crystallographic Details for $K_4Pd(PS_4)_2$.

I. Crystal Data

Empirical Formula	$K_4Pd(PS_4)_2$
Formula Weight	581.18
Crystal Color, Habit	orange-red, rod
Crystal Dimensions (mm)	0.19 x 0.11 x 0.11
Crystal System	Triclinic
Lattice Parameters	a = 6.380(1) Å
	b = 6.897(1) Å
	c = 8.999(2) Å
	α = 87.777(8)°
	β = 81.581(8)°
	γ = 106.61(1)°
	V = 389.8(2) Å^3
Space Group	$P\bar{1}$ (#2)
Z value	1
D_{calc}	2.469 g/cm^3
μ(MoKα)	17.8 cm^{-1}

II. Intensity Measurements

Diffractometer	Siemens P4
Radiation	AgK-$L_{2,3}$ (λ = 0.56086 Å)
Temperature	23°C
Scan Type	ω
$2\theta_{max}$	55°
No. of Reflections measured	Total : 6814
	Unique : 3408 (R_{int} = 0.029)
Corrections	Lorentz-polarization

III. Structure Solution and Refinement

Structure Solution	Direct Methods
Refinement	Full-matrix least squares
Function minimized	Σ w (|F_o| - |F_c|)^2
Least-squares Weights	$4F_o^2$ / $\sigma^2(F_o^2)$
Anomalous Dispersion	All non-hydrogen atoms
No. of Observations (I > 3.00σ (I))	1541

No. of Variables	71
Residuals : R; R_w	0.039; 0.050
Goodness of Fit Indicator	1.37
Max Shift / Error in Final Cycle	0.00
Maximum Peak in Final Diff. Map	0.78 e^- / Å^3
Minimum Peak in Final Diff. Map	-0.68 e^- / Å^3

Table S2. Crystallographic Details for $Cs_4Pd(PSe_4)_2$.

I. Crystal Data

Empirical Formula	$Cs_4Pd(PSe_4)_2$
Formula Weight	1331.65
Crystal Color, Habit	dark-red, irregular rod
Crystal Dimensions (mm)	0.53 x 0.39 x 0.36
Crystal System	Monoclinic
Lattice Parameters	a = 7.491(2) Å
	b = 13.340(2) Å
	c = 10.030(3) Å
	β = 92.21(2)°
	V = 1001.6(4) Å^3
Space Group	$P2_1/c$ (#14)
Z value	2
D_{calc}	4.415 g/cm^3
μ(MoKα)	225.64 cm^{-1}

II. Intensity Measurements

Diffractometer	Rigaku AFC6S
Radiation	MoKα (λ = 0.71069 Å)
Temperature	-100°C
Scan Type	ω–2θ
$2\theta_{max}$	50°
No. of Reflections measured	Total : 1990
	Unique : 1846 (R_{int} = 0.107)
Corrections	Lorentz-polarization
	Empirical Absorption

	(ψ-scan, DIFABS)
min./max. transmission	0.748

III. Structure Solution and Refinement

Structure Solution	Direct Methods
Refinement	Full-matrix least squares
Function minimized	Σ w ($\|F_o\|$ - $\|F_c\|$)^2
Least-squares Weights	$4F_o^2$ / $\sigma^2(F_o^2)$
Anomalous Dispersion	All non-hydrogen atoms
No. of Observations (I > 3.00σ (I))	1391
No. of Variables	70
Residuals : R; R_w	0.029; 0.038
Goodness of Fit Indicator	1.53
Max Shift / Error in Final Cycle	0.00
Maximum Peak in Final Diff. Map	0.95 e^- / Å^3
Minimum Peak in Final Diff. Map	-1.14 e^- / Å^3

Table S3. Crystallographic Details for $Cs_{10}Pd(PSe_4)_4$.

I. Crystal Data

Empirical Formula	$Cs_{10}Pd(PSe_4)_4$
Formula Weight	2822.71
Crystal Color, Habit	red, rod
Crystal Dimensions (mm)	0.84 x 0.28 x 0.28
Crystal System	Tetragonal
Lattice Parameters	a = 13.949(2) Å
	b = 13.949(2) Å
	c = 11.527(2) Å
	V = 2242.7(6) Å^3
Space Group	$P\bar{4}2c$ (#112)
Z value	2
D_{calc}	4.180 g/cm^3
μ(MoKα)	213.61 cm^{-1}

II. Intensity Measurements

Diffractometer	Rigaku AFC6S
Radiation	MoKα (λ = 0.71069 Å)
Temperature	23°C
Scan Type	ω–2θ
$2\theta_{max}$	55°
No. of Reflections measured	Total : 1543
	Unique : 1543
Corrections	Lorentz-polarization
	Empirical Absorption (ψ-scan, DIFABS)
min./max. transmission	0.629

III. Structure Solution and Refinement

Structure Solution	Direct Methods
Refinement	Full-matrix least squares
Function minimized	Σ w ($\|F_o\|$ - $\|F_c\|$)^2
Least-squares Weights	$4F_o^2$ / $\sigma^2(F_o^2)$
Anomalous Dispersion	All non-hydrogen atoms
No. of Observations (I > 3.00σ (I))	830
No. of Variables	73
Residuals : R; R_w	0.038; 0.043
Goodness of Fit Indicator	1.32
Max Shift / Error in Final Cycle	0.00
Maximum Peak in Final Diff. Map	1.77 e$^-$ / Å^3
Minimum Peak in Final Diff. Map	-1.13 e$^-$ / Å^3

Table S4. Crystallographic Details for $KPdPS_4$.

I. Crystal Data

Empirical Formula	$KPdPS_4$
Formula Weight	304.5
Crystal Color, Habit	orange-red, rod
Crystal Dimensions (mm)	0.23 x 0.13 x 0.11

Crystal System	Tetragonal
Lattice Parameters	a = 8.5337(2) Å
	b = 8.5337(2) Å
	c = 10.5595(5) Å
	V = 769.0(1) Å^3
Space Group	$P4_2/mmm$ (#136)
Z value	4
D_{calc}	2.631 g/cm^3
μ(MoKα)	21.4 cm^{-1}

II. Intensity Measurements

Diffractometer	Siemens P4
Radiation	AgK-$L_{2,3}$ (λ = 0.56086 Å)
Temperature	23°C
Scan Type	ω
$2\theta_{max}$	55°
No. of Reflections measured	Total : 3366
	Unique : 1252 (R_{int} = 0.024)
Corrections	Lorentz-polarization

III. Structure Solution and Refinement

Structure Solution	Direct Methods
Refinement	Full-matrix least squares
Function minimized	Σ w ($\|F_o\| - \|F_c\|$)^2
Least-squares Weights	$4F_o^2 / \sigma^2(F_o^2)$
Anomalous Dispersion	All non-hydrogen atoms
No. of Observations (I > 3.00σ (I))	430
No. of Variables	26
Residuals : R; R_w	0.025; 0.030
Goodness of Fit Indicator	0.82
Max Shift / Error in Final Cycle	0.00
Maximum Peak in Final Diff. Map	0.68 e$^-$ / Å^3
Minimum Peak in Final Diff. Map	-0.48 e$^-$ / Å^3

Table S5. Crystallographic Details for $K_2PdP_2S_6$.

I. Crystal Data

Empirical Formula	$K_2PdP_2S_6$
Formula Weight	438.9
Crystal Color, Habit	red, rod
Crystal Dimensions (mm)	0.13 x 0.04 x 0.03
Crystal System	Orthorhombic
Lattice Parameters	a = 15.612(2) Å
	b = 7.0724(7) Å
	c = 10.080(1) Å
	V = 1113.0(2) Å^3
Space Group	Pnma (#62)
Z value	4
D_{calc}	2.619 g/cm^3
μ(MoKα)	30.7 cm^{-1}

II. Intensity Measurements

Diffractometer	Enraf-Nonius CAD4-F
Radiation	MoKα (λ = 0.71069 Å)
Temperature	23°C
Scan Type	ω–2θ
$2\theta_{max}$	35°
No. of Reflections measured	Total : 7674
	Unique : 2594 (R_{int} = 0.058)
Corrections	Lorentz-polarization

III. Structure Solution and Refinement

Structure Solution	Direct Methods
Refinement	Full-matrix least squares
Function minimized	Σ w (|F_o| - |F_c|)^2
Least-squares Weights	$4F_o^2$ / $\sigma^2(F_o^2)$
Anomalous Dispersion	All non-hydrogen atoms
No. of Observations (I > 3.00σ (I))	741
No. of Variables	61
Residuals : R; R_w	0.048; 0.054
Goodness of Fit Indicator	1.29

Max Shift / Error in Final Cycle	0.00
Maximum Peak in Final Diff. Map	1.85 e^- / Å^3
Minimum Peak in Final Diff. Map	-1.88 e^- / Å^3

Table S6. Crystallographic Details for $Cs_2PdP_2Se_6$.

I. Crystal Data

Empirical Formula	$Cs_2PdP_2S_6$
Formula Weight	907.92
Crystal Color, Habit	black, plate
Crystal Dimensions (mm)	0.50 x 0.21 x 0.08
Crystal System	Monoclinic
Lattice Parameters	a = 12.9750 (4) Å
	b = 8.3282 (2) Å
	c = 13.0568 (1) Å
	β = 102.940(2)°
	V = 1375.07 (5) Å^3
Space Group	C2/c (#15)
Z value	4
D_{calc}	4.385 g/cm^3
μ(MoKα)	226.37 cm^{-1}

II. Intensity Measurements

Diffractometer	Siemens SMART Platform CCD
Radiation	MoKα (λ = 0.71073 Å)
Temperature	-141°C
$2\theta_{max}$	47°
No. of Reflections measured	Total : 3029
	Unique : 1072 (R_{int} = 0.097)
Corrections	Empirical Absorption SADABS
min./max. transmission	0.125

III. Structure Solution and Refinement

Structure Solution	Direct Methods
Refinement	Full-matrix least squares

Function minimized	Σ w ($\|F_o\|$ - $\|F_c\|$)^2
Least-squares Weights	$4F_o^2$ / $\sigma^2(F_o^2)$
No. of Observations (I > 3.00σ (I))	650
No. of Variables	80
Residuals : R; R_w	0.067; 0.075
Goodness of Fit Indicator	1.67
Max Shift / Error in Final Cycle	0.00
Maximum Peak in Final Diff. Map	1.62 e^- / Å^3
Minimum Peak in Final Diff. Map	-1.83 e^- / Å^3

Table S7. Table of Anisotropic Thermal Parameters for $K_4Pd(PS_4)_2$

Atom	U(1,1)	U(2,2)	U(3,3)	U(1,2)	U(1,3)	U(2,3)
Pd	0.0199(3)	0.0163(3)	0.0183(3)	-0.0041(2)	-0.0018(2)	0.0039(2)
S (1)	0.0337(8)	0.0216(7)	0.0316(8)	-0.0087(6)	-0.0098(6)	0.0025(6)
S (2)	0.0209(7)	0.0232(6)	0.0284(8)	0.0015(5)	-0.0018(6)	0.0074(5)
S (3)	0.0197(6)	0.0261(7)	0.0224(7)	-0.0053(5)	-0.0037(5)	0.0067(5)
S (4)	0.0294(8)	0.0425(9)	0.0182(7)	-0.0056(6)	0.0002(6)	0.0030(6)
P	0.01929(7)	0.0201(6)	0.0170(7)	-0.0044(5)	-0.0031(5)	0.0035(5)
K (1)	0.0374(8)	0.0351(7)	0.0299(7)	0.0018(6)	-0.0089(6)	-0.0011(6)
K (2)	0.0348(8)	0.0441(8)	0.0388(8)	-0.0115(6)	-0.0148(6)	0.0114(6)

Table S8. Table of Anisotropic Thermal Parameters for $Cs_4Pd(PSe_4)_2$

Atom	U(1,1)	U(2,2)	U(3,3)	U(1,2)	U(1,3)	U(2,3)
Cs(1)	0.0159(4)	0.0149(4)	0.0155(4)	-0.0038(3)	-0.0016(3)	0.0010(3)
Cs(2)	0.0140(4)	0.0145(4)	0.0146(3)	-0.0002(3)	0.0033(3)	-0.0007(3)
Pd	0.0068(6)	0.0104(6)	0.0081(5)	0.0014(4)	-0.0003(4)	-0.0006(4)
Se(1)	0.0179(6)	0.0160(6)	0.0085(5)	-0.0011(5)	0.0021(4)	-0.0017(4)
Se(2)	0.0119(6)	0.0078(5)	0.0124(5)	0.0003(4)	-0.0021(4)	0.0010(4)
Se(3)	0.0122(6)	0.0115(6)	0.0147(5)	-0.0039(4)	-0.0029(4)	0.0004(4)
Se(4)	0.0097(6)	0.0165(6)	0.0142(5)	-0.0037(5)	0.0033(4)	0.0014(4)
P	0.008(1)	0.006(1)	0.008(1)	0.001(1)	0.001(1)	-0.001(1)

Table S9. Table of Anisotropic Thermal Parameters for $Cs_{10}Pd(PSe_4)_4$

Atom	U(1,1)	U(2,2)	U(3,3)	U(1,2)	U(1,3)	U(2,3)
Cs(1)	0.0222(8)	0.031(1)	0.053(2)	0	0	-0.013(4)
Cs(2)	0.064(1)	0.044(1)	0.020(1)	0.005(6)	0	0
Cs(3)	0.041(2)	0.023(2)	0.0261(7)	-0.0003(5)	0.002(2)	0.005(2)
Cs(4)	0.0264(9)	0.0267(9)	0.053(1)	0	0	0.006(4)
Pd(1)	0.0186(8)	0.0186	0.016(1)	0	0	0
Se(1)	0.0186(8)	0.0210(9)	0.036(2)	-0.0015(7)	0.004(3)	0.004(3)
Se(2)	0.036(1)	0.032(1)	0.026(1)	0.014(3)	-0.005(4)	-0.007(1)
Se(3)	0.025(1)	0.031(1)	0.029(1)	-0.0094(8)	0.009(3)	-0.008(3)
Se(4)	0.030(1)	0.028(1)	0.021(1)	-0.001(4)	-0.002(4)	0.005(1)
P(1)	0.019(3)	0.016(3)	0.020(4)	0	0.00(1)	0
P(2)	0.019(3)	0.021(3)	0.015(4)	0	-0.00(1)	0

Table S10. Table of Anisotropic Thermal Parameters for $KPdPS_4$

Atom	U(1,1)	U(2,2)	U(3,3)	U(1,2)	U(1,3)	U(2,3)
Pd (1)	0.0246(2)	0.0246(2)	0.0289(3)	-0.0093(2)	0	0
Pd (2)	0.0279(2)	0.0279(2)	0.0296(3)	0.0127(2)	0	0
S (1)	0.0423(4)	0.0423(4)	0.0295(5)	0.0204(5)	-0.0005(3)	0.0005(3)
S (2)	0.0270(5)	0.0324(5)	0.0428(6)	-0.0079(4)	0	0
P	0.0309(4)	0.0309(4)	0.0283(7)	0.0128(6)	0	0
K	0.0533(6)	0.0533(6)	0.0357(7)	0	0	0

Table S11. Table of Anisotropic Thermal Parameters for $K_2PdP_2S_6$

Atom	U(1,1)	U(2,2)	U(3,3)	U(1,2)	U(1,3)	U(2,3)
Pd	0.0175(6)	0.0171(5)	0.0145(6)	-0.0001(9)	-0.0015(9)	-0.000(1)
S (1)	0.030(2)	0.024(2)	0.015(1)	-0.013(2)	0.003(2)	-0.001(2)
S (2)	0.031(2)	0.025(2)	0.038(2)	0.012(2)	-0.008(2)	-0.006(3)
S (3)	0.016(2)	0.021(2)	0.014(3)	0	-0.002(2)	0
P (4)	0.019(3)	0.015(2)	0.014(3)	0	-0.005(3)	0
P (1)	0.012(3)	0.016(2)	0.016(3)	0	-0.002(2)	0
S (2)	0.033(3)	0.039(3)	0.010(3)	0	-0.007(3)	0
K (1)	0.020(3)	0.040(3)	0.027(3)	0	0.006(2)	0
K (2)	0.038(3)	0.040(3)	0.060(4)	0	-0.010(3)	0

Table S12. Table of Anisotropic Thermal Parameters for $Cs_2PdP_2Se_6$

Atom	U(1,1)	U(2,2)	U(3,3)	U(1,2)	U(1,3)	U(2,3)
Cs	0.058(1)	0.061(1)	0.045(1)	-0.0165(9)	0.0129(9)	-0.0102(8)
Pd	0.018(2)	0.054(2)	0.112(3)	0.003(1)	0.001(2)	-0.043(2)
Se(1)	0.033(2)	0.050(2)	0.073(2)	0.013(1)	-0.007(1)	0.004(1)
Se(2)	0.058(2)	0.048(2)	0.038(2)	-0.019(1)	-0.001(1)	-0.008(1)
Se(3)	0.039(2)	0.075(2)	0.065(2)	0.008(2)	0.022(1)	-0.013(1)
P(1)	0.02(2)	0.06(2)	0.06(2)	0.01(1)	0.00(1)	-0.01(2)
P(2)	0.04(2)	0.05(2)	0.05(2)	-0.02(2)	0.02(2)	-0.01(2)
P(3)	0.03(1)	0.04(1)	0.03(1)	0.01(1)	0.01(1)	0.02(1)
P(4)	0.01(2)	0.04(1)	0.05(2)	0.00(1)	0.00(1)	-0.02(1)

Table S13. Calculated and Observed X-ray Powder Pattern of $K_4Pd(PS_4)_2$.

h	k	l	d_{obs} (Å)	d_{calc} (Å)	I_{obs} (%)
0	0	1	8.8892	8.9002	36
0	1	0	6.8531	6.8624	19
1	0	0	6.2767	6.2849	44
1	0	1	5.5158	5.5200	100
1	1	1	4.5218	4.5200	19
1	-1	0	4.4387	4.4351	23
-1	1	1	3.8414	3.8419	36
0	1	2	3.7778	3.7771	10
0	-1	2	3.6931	3.6919	19
1	1	2	3.5150	3.5163	20
-1	0	2	3.4085	3.4088	2
0	2	1	3.2289	3.2287	71
0	-2	1	3.1765	3.1750	7
2	0	0	3.1419	3.1425	38
2	0	1	3.1065	3.1062	10
-1	-1	2	3.0900	3.0914	4
1	2	1	3.0501	3.0493	9
-1	1	2	3.0190	3.0159	3
0	0	3	2.9654	2.9667	40
2	1	0	2.9654	2.9624	40
2	1	1	2.9424	2.9426	4
-2	0	1	2.8396	2.8383	8
1	-2	1	2.7948	2.7954	10
0	2	2	2.7500	2.7507	9
0	-1	3	2.6967	2.6987	44
-2	-1	1	2.6967	2.6957	44
1	2	2	2.6860	2.6855	32
0	-2	2	2.6860	2.6850	32
1	-1	3	2.5696	2.5688	13
-2	1	1	2.5551	2.5555	7
-1	0	3	2.5458	2.5458	3
1	-2	2	2.4816	2.4818	23
2	2	0	2.4314	2.4321	16
2	0	3	2.3301	2.3303	22
0	3	0	2.2888	2.2874	2
0	2	3	2.2725	2.2725	7
-2	1	2	2.2334	2.2337	4
0	0	4	2.2254	2.2250	6
2	-2	0	2.2171	2.2175	9
0	-2	3	2.2171	2.2169	9
0	-3	1	2.2017	2.2022	8
1	1	4	2.1292	2.1296	3
1	-2	3	2.1220	2.1224	6
-1	-3	1	2.1133	2.1140	3
2	-2	2	2.0559	2.0557	24
-2	-2	2	2.0274	2.0273	12
-2	0	3	2.0166	2.0178	13
-1	0	4	2.0088	2.0089	12
-3	0	1	1.9771	1.9770	4

-3	-1	1	1.9413	1.9424	7
3	-1	2	1.8792	1.8786	4
2	-1	4	1.8402	1.8400	11
0	3	3	1.8329	1.8338	4
1	0	5	1.7804	1.7809	7
2	2	4	1.7576	1.7581	6
0	4	0	1.7151	1.7156	13
-1	-3	3	1.7151	1.7152	13
2	0	5	1.6544	1.6537	3
1	3	4	1.6326	1.6325	12
3	3	1	1.6326	1.6326	12
1	-4	0	1.6173	1.6175	8
1	-4	1	1.5946	1.5952	6
2	4	1	1.5702	1.5698	3
0	-2	5	1.5638	1.5642	8

Table S14. Calculated and Observed X-ray Powder Pattern of $Cs_4Pd(PSe_4)_2$.

h	k	l	d_{obs} (Å)	d_{calc} (Å)	I_{obs} (%)
0	1	1	8.014	8.035	23
1	1	-1	5.556	5.566	16
1	2	1	4.415	4.417	2
1	0	2		4.092	
0	3	1	4.085	4.065	14
1	1	-2		4.042	
2	0	0	3.743	3.744	13
1	3	-1	3.595	3.594	36
2	1	-1	3.432	3.435	27
0	4	0	3.335	3.332	100
0	1	3	3.241	3.245	24
2	2	-1	3.135	3.136	7
2	2	1	3.073	3.073	4
0	2	3	2.987	2.985	37
2	0	2	2.945	2.945	10
1	4	1	2.902	2.909	16
2	1	2	2.876	2.876	13
2	2	-2	2.778	2.776	14
2	3	-1	2.775	2.774	32
2	3	1	2.732	2.735	5
0	3	3	2.671	2.675	40
2	3	2	2.455	2.454	16
2	2	-3	2.375	2.375	20
1	5	-2	2.258	2.257	16
2	4	2	2.207	2.207	30
2	0	-4	2.120	2.119	12
0	5	3	2.085	2.084	16
2	5	-2	2.009	2.009	12
4	0	0	1.871	1.871	10
4	1	0	1.853	1.853	11

Table S15. Calculated and Observed X-ray Powder Pattern of $KPdPS_4$.

h	k	l	d_{obs} (Å)	d_{calc} (Å)	I_{obs} (%)
1	1	0	6.0226	6.0355	80
0	0	2	5.2764	5.2806	76
1	1	1	5.2450	5.2402	68
2	0	0	4.2699	4.2677	53
1	1	2	3.9751	3.9742	83
2	1	0	3.8206	3.8172	19
2	1	1	3.5903	3.5899	12
2	0	2	3.3200	3.3192	7
1	0	3	3.2557	3.2545	15
2	2	0	3.0175	3.0177	30
2	2	1	2.9013	2.9016	4
3	0	1	2.7471	2.7472	18
3	1	0	2.6986	2.6991	6
0	0	4	2.6411	2.6403	23
2	2	2	2.6192	2.6201	3
2	1	3	2.5893	2.5879	7
3	1	2	2.4035	2.4034	100
2	2	3	2.2905	2.2912	3
2	0	4	2.2446	2.2453	16
2	1	4	2.1720	2.1715	5
4	0	0	2.1352	2.1339	28
4	1	1	2.0309	2.0315	4
3	3	0	2.0113	2.0118	16
2	2	4	1.9869	1.9871	10
4	0	2	1.9780	1.9784	24
4	2	0	1.9084	1.9086	15
3	1	4	1.8872	1.8874	16
3	3	2	1.8797	1.8800	20
4	2	2	1.7946	1.7949	2
0	0	6	1.7602	1.7602	10
1	1	6	1.6895	1.6898	10
4	0	4	1.6596	1.6596	29
5	1	2	1.5958	1.5957	15
5	2	1	1.5677	1.5674	2
4	2	4	1.5468	1.5468	7
4	3	3	1.5360	1.5360	6
4	4	0	1.5088	1.5089	22