Supplementary Material (ESI) for Dalton Transactions

STRUCTURE DETERMINATION SUMMARY

Crystal Data for 207336

Empirical Formula	C32 H36 I4 O3 P2 Pd2
Crystal System	Orthorhombic
Space Group	Pnma
Unit Cell Dimensions	a = 16.594(3) Δ
	b = 24.734(3) Δ
	c = 9.3914(10) Δ
Volume	3855(2) Δ^3
Z	4
Formula weight	1250.9
Density(calc.)	2.156 Mg/m^3
Absorption Coefficient	4.249 mm^{-1}
F(000)	2344

Data Collection

Diffractometer Used	Enraf Nonius CAD4
Radiation	MoK_α (λ = 0.71073 Δ)
Temperature (K)	298
Monochromator	Highly oriented graphite crystal
θ Range	5.0 to 45.0°
Index Ranges	-17 < h < 17, -26 < k < 26, 0 < l < 10
Reflections Collected	9809
Independent Reflections	2580 (R_{int} = 1.88%)
Observed Reflections	2580 (F > 0.3σ(F))
Absorption Correction	Semi-empirical
Min./Max. Transmission	0.1801 / 0.3071

Solution and Refinement

System Used	Siemens SHELXTL PLUS (PC Version)
Solution	Direct Methods
Refinement Method	Full-Matrix Least-Squares
Refinement on	F^2
Absolute Structure	N/A
Extinction Coefficient	0.000106(15)
Hydrogen Atoms	Riding model, fixed isotropic U
Weighting Scheme	$w^{-1} = \sigma 2(F) + 0.0008F^2$
Number of Parameters Refined	200
Final R Indices (obs. data)	R = 4.52 %, wR = 5.24 %
R Indices (all data)	R = 4.52 %, wR = 5.24 %
Goodness-of-Fit	1.29
Largest and Mean shift/error	0.000, 0.000
Data-to-Parameter Ratio	12.9:1
Largest Difference Peak	1.17 $e\Delta^{-3}$
Largest Difference Hole	-0.82 $e\Delta^{-3}$

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

	X	y	z	U(eq)
I(1)	173(1)	2500	-1972(1)	69(1)
I(2)	1301(1)	2500	1227(1)	51(1)
I(3)	1126(1)	972(1)	1402(1)	60(1)
Pd(1)	636(1)	1718(1)	-345(1)	38(1)
P(1)	50(1)	1081(1)	-1726(2)	39(1)
O(3)	-1642(3)	1364(3)	-234(6)	79(2)
O(6)	-2267(5)	2500	439(13)	119(6)
C(1)	-684(4)	666(3)	-764(9)	58(3)
C(2)	-1192(4)	943(4)	366(9)	67(3)
C(4)	-2254(9)	1535(6)	615(13)	146(7)
C(5)	-2694(6)	2009(6)	25(14)	124(6)
C(11)	752(4)	596(3)	-2471(8)	45(2)
C(12)	458(5)	139(3)	-3151(9)	58(3)
C(13)	989(5)	-219(3)	-3807(10)	68(3)
C(14)	1774(6)	-157(4)	-3762(10)	77(4)
C(15)	2076(5)	277(5)	-3046(12)	96(5)
C(16)	1558(5)	665(4)	-2434(10)	76(4)
C(21)	-436(4)	1339(3)	-3333(8)	44(2)
C(22)	-1277(5)	1429(4)	-3393(9)	62(3)
C(23)	-1589(6)	1646(4)	-4614(12)	81(4)
C(24)	-1103(9)	1788(4)	-5770(12)	93(5)
C(25)	-304(7)	1706(3)	-5670(10)	73(4)
C(26)	36(5)	1473(3)	-4476(8)	57(3)

* Equivalent isotropic U defined as one third of the trace of the orthogonalized Uij tensor

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Table 2. Bond lengths (Δ)

I(1)-Pd(1)	2.582 (1)
I(1)-Pd(1A)	2.582 (1)
I(2)-Pd(1)	2.672 (1)
I(2)-Pd(1A)	2.672 (1)
I(3)-Pd(1)	2.600 (1)
Pd(1)-P(1)	2.260 (2)
P(1)-C(1)	1.832 (8)
P(1)-C(11)	1.813 (7)
P(1)-C(21)	1.826 (7)
O(3)-C(2)	1.399 (11)
O(3)-C(4)	1.359 (15)
O(6)-C(5)	1.459 (14)
O(6)-C(5A)	1.459 (14)
C(1)-C(2)	1.520 (12)
C(4)-C(5)	1.488 (20)
C(11)-C(12)	1.387 (10)
C(11)-C(16)	1.349 (10)
C(12)-C(13)	1.393 (12)
C(13)-C(14)	1.312 (13)
C(14)-C(15)	1.363 (15)
C(15)-C(16)	1.410 (14)
C(21)-C(22)	1.415 (10)
C(21)-C(26)	1.369 (11)
C(22)-C(23)	1.368 (14)
C(23)-C(24)	1.397 (16)
C(24)-C(25)	1.345 (18)
C(25)-C(26)	1.382 (12)

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Table 3. Bond angles (°)

Pd(1)-I(1)-Pd(1A)	97.0(1)
Pd(1)-I(2)-Pd(1A)	92.8(1)
I(1)-Pd(1)-I(2)	84.7(1)
I(1)-Pd(1)-I(3)	176.7(1)
I(2)-Pd(1)-I(3)	92.1(1)
I(1)-Pd(1)-P(1)	93.1(1)
I(2)-Pd(1)-P(1)	177.8(1)
I(3)-Pd(1)-P(1)	90.1(1)
Pd(1)-P(1)-C(1)	113.2(3)
Pd(1)-P(1)-C(11)	114.0(2)
C(1)-P(1)-C(11)	104.2(3)
Pd(1)-P(1)-C(21)	114.9(2)
C(1)-P(1)-C(21)	108.1(3)
C(11)-P(1)-C(21)	101.3(3)
C(2)-O(3)-C(4)	113.2(8)
C(5)-O(6)-C(5A)	112.8(11)
P(1)-C(1)-C(2)	117.4(6)
O(3)-C(2)-C(1)	110.5(7)
O(3)-C(4)-C(5)	113.1(10)
O(6)-C(5)-C(4)	108.6(10)
P(1)-C(11)-C(12)	119.4(5)
P(1)-C(11)-C(16)	123.0(6)
C(12)-C(11)-C(16)	117.6(7)
C(11)-C(12)-C(13)	119.9(7)
C(12)-C(13)-C(14)	122.7(8)
C(13)-C(14)-C(15)	118.2(9)
C(14)-C(15)-C(16)	120.8(8)
C(11)-C(16)-C(15)	120.6(8)
P(1)-C(21)-C(22)	121.6(6)
P(1)-C(21)-C(26)	118.7(6)
C(22)-C(21)-C(26)	119.7(7)
C(21)-C(22)-C(23)	118.0(8)
C(22)-C(23)-C(24)	122.1(10)
C(23)-C(24)-C(25)	118.5(10)
C(24)-C(25)-C(26)	121.5(10)
C(21)-C(26)-C(25)	120.2(8)

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

	U11	U22	U33	U12	U13	U23
I(1)	120(1)	27(1)	61(1)	0	-42(1)	0
I(2)	67(1)	35(1)	51(1)	0	-19(1)	0
I(3)	67(1)	42(1)	73(1)	4(1)	-18(1)	15(1)
Pd(1)	44(1)	26(1)	44(1)	2(1)	-5(1)	1(1)
P(1)	40(1)	27(1)	51(1)	-1(1)	-2(1)	-2(1)
O(3)	64(3)	107(5)	67(4)	29(4)	19(3)	11(4)
O(6)	48(5)	185(13)	125(10)	0	-11(6)	0
C(1)	57(5)	48(5)	70(5)	-8(4)	0(4)	9(4)
C(2)	57(5)	81(6)	64(6)	5(4)	5(4)	16(5)
C(4)	140(11)	223(17)	74(8)	92(11)	7(8)	-25(10)
C(5)	85(8)	175(14)	112(9)	26(9)	-9(7)	-62(10)
C(11)	39(4)	38(4)	56(5)	3(3)	1(3)	-4(4)
C(12)	59(4)	42(5)	73(5)	7(4)	-13(4)	-13(4)
C(13)	93(7)	36(5)	76(6)	12(4)	-12(5)	-20(4)
C(14)	68(6)	71(7)	92(7)	15(5)	18(5)	-35(6)
C(15)	50(5)	112(9)	125(9)	4(5)	25(6)	-25(8)
C(16)	52(5)	65(6)	111(8)	-22(4)	21(5)	-34(6)
C(21)	58(4)	29(4)	45(4)	-1(3)	-8(3)	-9(3)
C(22)	60(5)	66(6)	60(5)	7(4)	-14(4)	7(5)
C(23)	81(6)	82(7)	82(7)	15(5)	-28(6)	9(6)
C(24)	172(12)	44(6)	65(7)	2(7)	-45(8)	5(5)
C(25)	108(7)	58(6)	54(6)	-13(5)	7(6)	0(5)
C(26)	75(5)	36(4)	60(5)	-11(4)	-2(4)	2(4)

The anisotropic displacement exponent takes the form:$-2\pi^2(h^2a^{*2}U11 + ... + 2hka^*b^*U12)$

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

	X	y	z	U
H(1A)	-404	366	-344	59
H(1B)	-1053	520	-1452	59
H(2A)	-1545	686	809	67
H(2B)	-839	1087	1082	67
H(4A)	-2064	1620	1553	145
H(4B)	-2630	1241	692	145
H(5A)	-2704	1980	-994	126
H(5B)	-3239	2029	362	126
H(12)	-112	75	-3183	58
H(13)	769	-529	-4285	69
H(14)	2125	-405	-4246	77
H(15)	2649	316	-2941	95
H(16)	1781	977	-1971	76
H(22)	-1615	1343	-2596	62
H(23)	-2160	1703	-4684	81
H(24)	-1340	1940	-6613	93
H(25)	32	1816	-6450	74
H(26)	604	1401	-4444	57