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J1695-1

STRUCTURE DETERMINATION SUMMARY

Empirical Formula	$C_{16}H_{11}O_5PW$
Crystal size	0.20mm x 0.20mm x 0.10mm
Crystal System	Orthorhombic
Space Group	$P2_12_12_1$
Unit Cell Dimensions	$\underline{a}$ = 6.6763(9) Å $\underline{b}$ = 13.5269(21) Å $\underline{c}$ = 18.8822(27) Å
Volume	1705.2(9) $Å^3$
Z	4
Formula weight	498.1
Density(calc.)	1.940 Mg/m^3
Absorption Coefficient	6.888 mm^{-1}
Diffractometer Used	Enraf Nonius CAD4
Radiation	MoKα (λ = 0.71073 Å)
Temperature (K)	298
Monochromator	Highly oriented graphite crystal
2θ Range	5.0 to 45.0°
Scan Type	ω
Scan Speed	Variable; 2.00 to 29.30°/min. in ω
Scan Range (ω)	0.55°
Standard Reflections	3 measured every 6000 seconds
Index Ranges	$0 \le h \le 7$, $0 \le k \le 14$ $-20 \le l \le 20$
Reflections Collected	3059
Independent Reflections	2216 (R_{int} = 1.81%)
Observed Reflections	1964 ($F > 6.0\sigma(F)$)
Absorption Correction	Semi-empirical
Min./Max. Transmission	0.2245 / 0.3180
System Used	Siemens SHELXTL PLUS (PC Version)
Refinement Method	Full-Matrix Least-Squares
Quantity Minimized	$\sum w(F_o-F_c)^2$
Absolute Structure	η = 1.07(3)
Hydrogen Atoms	Riding model, fixed isotropic U
Weighting Scheme	$w^{-1} = \sigma^2(F) + 0.0010F^2$
Number of Parameters Refined	210
Final R Indices (6σ data)	R = 2.47 %, Rw = 2.83 %
R Indices (all data)	R = 2.85 %, Rw = 3.77 %
Goodness-of-Fit	0.85
Largest and Mean Δ/σ	0.001, 0.000
Data-to-Parameter Ratio	10.6:1
Largest Difference Peak	0.67 $eÅ^{-3}$
Largest Difference Hole	-0.87 $eÅ^{-3}$

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

	x	y	z	U(eq)
W(1)	2585(1)	9574(1)	5834(1)	42(1)
P(1)	3782(3)	11179(2)	6311(1)	41(1)
O(1)	1000(13)	7523(5)	5283(4)	87(3)
O(2)	708(13)	9068(8)	7326(4)	103(4)
O(3)	6763(11)	8692(6)	6340(5)	97(3)
O(4)	4591(12)	10056(6)	4352(4)	83(3)
O(5)	-1406(11)	10631(6)	5315(4)	90(3)
C(1)	1576(15)	8253(8)	5469(5)	60(3)
C(2)	1365(15)	9250(8)	6796(5)	66(4)
C(3)	5230(15)	8990(6)	6158(6)	60(3)
C(4)	3876(14)	9889(7)	4881(5)	55(3)
C(5)	28(13)	10237(7)	5501(5)	54(3)
C(11)	2881(14)	12404(6)	6037(4)	58(3)
C(12)	5015(14)	12132(6)	5784(5)	62(3)
C(13)	6342(17)	12880(7)	6117(5)	74(4)
C(14)	5169(20)	13487(7)	6532(6)	78(5)
C(15)	3152(22)	13287(7)	6509(5)	80(5)
C(21)	4691(13)	11260(7)	7216(4)	48(3)
C(22)	3327(16)	11444(7)	7743(4)	64(3)
C(23)	3952(23)	11425(8)	8448(5)	80(5)
C(24)	5923(26)	11186(9)	8612(5)	95(6)
C(25)	7252(21)	11005(8)	8080(5)	90(5)
C(26)	6629(14)	11023(7)	7369(5)	63(3)

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

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

	x	y	z	U
H(11)	1826	12417	5691	57
H(12)	5184	11990	5289	63
H(13A)	7760	12930	6037	74
H(13B)	7016	13259	5759	74
H(13C)	7334	12557	6403	74
H(14)	5756	13989	6826	79
H(15A)	2115	13642	6753	80
H(15B)	2478	13843	6301	80
H(15C)	2612	13179	6974	80
H(22)	1965	11605	7624	65
H(23)	2995	11533	8820	80
H(24)	6352	11212	9097	96
H(25)	8604	10821	8195	90
H(26)	7547	10889	6990	63

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Table 3. Bond lengths (Å)

W(1)-P(1)	2.482	(2)	W(1)-C(1)	2.030	(10)
W(1)-C(2)	2.040	(10)	W(1)-C(3)	2.029	(10)
W(1)-C(4)	2.040	(9)	W(1)-C(5)	2.028	(9)
P(1)-C(11)	1.837	(9)	P(1)-C(12)	1.826	(9)
P(1)-C(21)	1.816	(8)	O(1)-C(1)	1.116	(12)
O(2)-C(2)	1.119	(13)	O(3)-C(3)	1.153	(13)
O(4)-C(4)	1.129	(12)	O(5)-C(5)	1.150	(12)
C(11)-C(12)	1.547	(13)	C(11)-C(15)	1.501	(13)
C(12)-C(13)	1.484	(14)	C(13)-C(14)	1.379	(16)
C(14)-C(15)	1.374	(20)	C(21)-C(22)	1.372	(13)
C(21)-C(26)	1.364	(13)	C(22)-C(23)	1.396	(13)
C(23)-C(24)	1.390	(23)	C(24)-C(25)	1.362	(18)
C(25)-C(26)	1.406	(14)			

Table 4. Bond angles (°)

P(1)-W(1)-C(1)	178.4(3)	P(1)-W(1)-C(2)	89.6(3)
C(1)-W(1)-C(2)	88.8(4)	P(1)-W(1)-C(3)	87.2(3)
C(1)-W(1)-C(3)	92.7(4)	C(2)-W(1)-C(3)	89.7(4)
P(1)-W(1)-C(4)	90.1(3)	C(1)-W(1)-C(4)	91.5(4)
C(2)-W(1)-C(4)	178.5(4)	C(3)-W(1)-C(4)	88.8(4)
P(1)-W(1)-C(5)	89.8(3)	C(1)-W(1)-C(5)	90.3(4)
C(2)-W(1)-C(5)	92.0(4)	C(3)-W(1)-C(5)	176.6(4)
C(4)-W(1)-C(5)	89.5(4)	W(1)-P(1)-C(11)	125.5(3)
W(1)-P(1)-C(12)	124.4(3)	C(11)-P(1)-C(12)	50.0(4)
W(1)-P(1)-C(21)	120.2(3)	C(11)-P(1)-C(21)	108.6(4)
C(12)-P(1)-C(21)	108.6(4)	W(1)-C(1)-O(1)	178.5(8)
W(1)-C(2)-O(2)	179.4(9)	W(1)-C(3)-O(3)	177.5(8)
W(1)-C(4)-O(4)	179.5(7)	W(1)-C(5)-O(5)	178.6(9)
P(1)-C(11)-C(12)	64.6(5)	P(1)-C(11)-C(15)	120.7(7)
C(12)-C(11)-C(15)	105.1(8)	P(1)-C(12)-C(11)	65.4(5)
P(1)-C(12)-C(13)	121.3(7)	C(11)-C(12)-C(13)	104.9(7)
C(12)-C(13)-C(14)	107.9(10)	C(13)-C(14)-C(15)	114.9(10)
C(11)-C(15)-C(14)	107.0(10)	P(1)-C(21)-C(22)	118.1(7)
P(1)-C(21)-C(26)	120.2(7)	C(22)-C(21)-C(26)	121.2(8)
C(21)-C(22)-C(23)	119.4(10)	C(22)-C(23)-C(24)	120.0(11)
C(23)-C(24)-C(25)	119.6(10)	C(24)-C(25)-C(26)	120.5(12)
C(21)-C(26)-C(25)	119.2(9)		

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Table 5. Anisotropic displacement coefficients ($Å^2 x 10^3$)

	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
W(1)	37(1)	48(1)	41(1)	-1(1)	-2(1)	-2(1)
P(1)	43(1)	44(1)	36(1)	1(1)	-2(1)	0(1)
O(1)	102(6)	55(4)	106(6)	-24(4)	-28(5)	-12(4)
O(2)	91(6)	159(9)	58(5)	-23(6)	9(4)	22(5)
O(3)	52(4)	83(5)	157(8)	6(4)	-29(5)	13(5)
O(4)	83(5)	107(6)	58(4)	-10(4)	23(4)	-2(4)
O(5)	59(4)	119(6)	93(5)	28(5)	-7(4)	20(5)
C(1)	57(6)	59(6)	64(6)	0(5)	-16(5)	3(5)
C(2)	56(6)	89(7)	52(6)	-1(5)	2(5)	8(5)
C(3)	59(6)	42(5)	78(7)	-6(5)	0(5)	3(5)
C(4)	45(5)	68(6)	52(6)	-4(5)	7(4)	-16(5)
C(5)	40(5)	64(6)	59(6)	5(5)	0(4)	-1(5)
C(11)	62(6)	55(5)	57(5)	11(5)	-14(5)	10(4)
C(12)	82(6)	59(5)	46(5)	3(5)	5(5)	14(5)
C(13)	87(7)	56(6)	77(6)	-19(6)	7(6)	15(5)
C(14)	95(9)	46(6)	94(9)	-6(6)	-11(7)	4(5)
C(15)	120(11)	47(5)	71(6)	31(6)	-17(7)	-11(5)
C(21)	56(5)	46(5)	43(5)	-1(4)	-7(4)	-1(4)
C(22)	74(7)	62(6)	57(6)	-12(5)	9(5)	-8(4)
C(23)	131(11)	68(7)	42(5)	-13(8)	19(6)	-6(5)
C(24)	167(14)	72(7)	46(6)	-36(9)	-24(8)	-6(6)
C(25)	97(9)	103(8)	71(6)	-18(8)	-46(7)	29(6)
C(26)	60(6)	64(6)	65(6)	0(5)	-7(5)	-5(5)

The anisotropic displacement exponent takes the form:

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