Diastereocontrol in Asymmetric Allyl-Allyl Cross Coupling: Stereocontrolled Reaction of Prochiral Allylboronates with Prochiral Allyl Chlorides

Laura A. Brozek, Michael J. Ardolino, James P. Morken*

Department of Chemistry, Merkert Chemistry Center, Boston College, Chestnut Hill, MA 02467

Table 1. Crystal data and structure refinement for C30H24Cl2O6P2Pd.

Identification code	C30H24Cl2O6P2Pd	
Empirical formula	C30 H24 Cl2 O6 P2 Pd	
Formula weight	719.73	
Temperature	100(2) K	
Wavelength	0.71073 ≈	
Crystal system	Tetragonal	
Space group	P4(1)2(1)2	
Unit cell dimensions	a = 11.0719(3) ≈	α= 90∞.
	b = 11.0719(3) ≈	β= 90∞.
	c = 23.9032(7) ≈	γ = 90∞.
Volume	2930.22(14) ≈^{3}	
Z	4	
Density (calculated)	1.631 Mg/m^3	
Absorption coefficient	0.968 mm^{-1}	
F(000)	1448	
Crystal size	0.16 x 0.15 x 0.12 mm^3	
Theta range for data collection	2.03 to 28.35∞.	
Index ranges	-14<=h<=14, -13<=k<=14, -29<=l<=31	
Reflections collected	25662	
Independent reflections	3655 [R(int) = 0.0196]	
Completeness to theta = 28.35∞	100.0 %	
Absorption correction	Semi-empirical from equivalents	
Max. and min. transmission	0.8927 and 0.8605	
Refinement method	Full-matrix least-squares on F^2	
Data / restraints / parameters	3655 / 0 / 187	
Goodness-of-fit on F^2	1.119	
Final R indices [I>2sigma(I)]	R1 = 0.0145, wR2 = 0.0376	
R indices (all data)	R1 = 0.0149, wR2 = 0.0378	
Absolute structure parameter	-0.006(14)	
Extinction coefficient	na	
Largest diff. peak and hole	0.273 and -0.348 e.≈$^{-3}$	

Table 2. Atomic coordinates (x 10^4) and equivalent isotropic displacement parameters (≈^{2}x 10^3) for C30H24Cl2O6P2Pd. U(eq) is defined as one third of the trace of the orthogonalized U^{ij} tensor.

	x	y	z	U(eq)
Pd(1)	10183(1)	10183(1)	0	11(1)
Cl(1)	12198(1)	10220(1)	294(1)	23(1)
P(1)	9929(1)	8442(1)	456(1)	11(1)
O(1)	10132(1)	6020(1)	337(1)	20(1)
O(2)	5465(1)	7352(1)	95(1)	17(1)
O(3)	10814(1)	9358(1)	1432(1)	20(1)
C(1)	10401(1)	7121(1)	100(1)	14(1)
C(2)	11033(1)	6971(2)	-380(1)	22(1)
C(3)	11169(2)	5702(2)	-450(1)	31(1)
C(4)	10617(1)	5175(2)	-12(1)	28(1)
C(5)	8368(1)	8233(1)	672(1)	12(1)
C(6)	7487(1)	7820(1)	294(1)	12(1)
C(7)	6282(1)	7770(1)	474(1)	14(1)
C(8)	5959(1)	8172(2)	1006(1)	19(1)
C(9)	6838(1)	8618(2)	1363(1)	22(1)
C(10)	8039(1)	8646(1)	1205(1)	17(1)
C(11)	4248(1)	7225(2)	285(1)	22(1)
C(12)	10834(1)	8382(1)	1078(1)	14(1)
C(13)	11701(2)	9144(2)	1820(1)	24(1)
C(14)	12257(1)	8087(1)	1724(1)	22(1)
C(15)	11696(1)	7582(1)	1239(1)	17(1)

Table 3. Bond lengths [≈] and angles [∞] for C30H24Cl2O6P2Pd.

Pd(1)-P(1)	2.2332(3)
Pd(1)-P(1)#1	2.2332(3)
Pd(1)-Cl(1)#1	2.3389(3)
Pd(1)-Cl(1)	2.3389(3)
P(1)-C(1)	1.7711(14)
P(1)-C(12)	1.7931(14)
P(1)-C(5)	1.8180(13)
O(1)-C(4)	1.3640(19)
O(1)-C(1)	1.3767(16)
O(2)-C(7)	1.3605(17)
O(2)-C(11)	1.4290(17)
O(3)-C(13)	1.3724(18)
O(3)-C(12)	1.3726(17)
C(1)-C(2)	1.3551(19)
C(2)-C(3)	1.423(2)
C(2)-H(2)	0.9500
C(3)-C(4)	1.344(3)
C(3)-H(3)	0.9500
C(4)-H(4)	0.9500
C(5)-C(10)	1.4020(18)
C(5)-C(6)	1.4069(17)
C(6)-C(7)	1.4021(19)
C(6)-C(6)#1	1.500(2)
C(7)-C(8)	1.3955(19)
C(8)-C(9)	1.386(2)
C(8)-H(8)	0.9500
C(9)-C(10)	1.383(2)
C(9)-H(9)	0.9500
C(10)-H(10)	0.9500
C(11)-H(11A)	0.9800
C(11)-H(11B)	0.9800
C(11)-H(11C)	0.9800
C(12)-C(15)	1.358(2)
C(13)-C(14)	1.342(2)

C(13)-H(13)	0.9500
C(14)-C(15)	1.429(2)
C(14)-H(14)	0.9500
C(15)-H(15)	0.9500

P(1)-Pd(1)-P(1)#1	91.193(18)
P(1)-Pd(1)-Cl(1)#1	166.502(13)
P(1)#1-Pd(1)-Cl(1)#1	89.329(12)
P(1)-Pd(1)-Cl(1)	89.329(12)
P(1)#1-Pd(1)-Cl(1)	166.501(13)
Cl(1)#1-Pd(1)-Cl(1)	93.297(19)
C(1)-P(1)-C(12)	101.71(7)
C(1)-P(1)-C(5)	108.19(6)
C(12)-P(1)-C(5)	106.92(6)
C(1)-P(1)-Pd(1)	116.15(5)
C(12)-P(1)-Pd(1)	111.48(5)
C(5)-P(1)-Pd(1)	111.61(4)
C(4)-O(1)-C(1)	105.68(12)
C(7)-O(2)-C(11)	116.67(11)
C(13)-O(3)-C(12)	105.66(11)
C(2)-C(1)-O(1)	110.62(12)
C(2)-C(1)-P(1)	131.39(12)
O(1)-C(1)-P(1)	117.97(9)
C(1)-C(2)-C(3)	105.88(15)
C(1)-C(2)-H(2)	127.1
C(3)-C(2)-H(2)	127.1
C(4)-C(3)-C(2)	106.88(15)
C(4)-C(3)-H(3)	126.6
C(2)-C(3)-H(3)	126.6
C(3)-C(4)-O(1)	110.94(14)
C(3)-C(4)-H(4)	124.5
O(1)-C(4)-H(4)	124.5
C(10)-C(5)-C(6)	120.62(12)
C(10)-C(5)-P(1)	117.64(10)
C(6)-C(5)-P(1)	121.22(10)
C(7)-C(6)-C(5)	118.44(12)

C(7)-C(6)-C(6)#1	120.74(11)
C(5)-C(6)-C(6)#1	120.78(10)
O(2)-C(7)-C(8)	123.04(13)
O(2)-C(7)-C(6)	116.22(12)
C(8)-C(7)-C(6)	120.71(13)
C(9)-C(8)-C(7)	119.71(14)
C(9)-C(8)-H(8)	120.1
C(7)-C(8)-H(8)	120.1
C(10)-C(9)-C(8)	120.99(14)
C(10)-C(9)-H(9)	119.5
C(8)-C(9)-H(9)	119.5
C(9)-C(10)-C(5)	119.44(13)
C(9)-C(10)-H(10)	120.3
C(5)-C(10)-H(10)	120.3
O(2)-C(11)-H(11A)	109.5
O(2)-C(11)-H(11B)	109.5
H(11A)-C(11)-H(11B)	109.5
O(2)-C(11)-H(11C)	109.5
H(11A)-C(11)-H(11C)	109.5
H(11B)-C(11)-H(11C)	109.5
C(15)-C(12)-O(3)	110.46(12)
C(15)-C(12)-P(1)	130.70(11)
O(3)-C(12)-P(1)	118.21(10)
C(14)-C(13)-O(3)	111.27(13)
C(14)-C(13)-H(13)	124.4
O(3)-C(13)-H(13)	124.4
C(13)-C(14)-C(15)	106.32(13)
C(13)-C(14)-H(14)	126.8
C(15)-C(14)-H(14)	126.8
C(12)-C(15)-C(14)	106.29(13)
C(12)-C(15)-H(15)	126.9
C(14)-C(15)-H(15)	126.9

Symmetry transformations used to generate equivalent atoms:

#1 y,x,-z

Table 4. Anisotropic displacement parameters ($\approx^2$x 10^3) for C30H24Cl2O6P2Pd. The anisotropic displacement factor exponent takes the form: $-2\pi^2[h^2 a^{*2}U^{11} + ... + 2 h k a^* b^* U^{12}]$

	U^{11}	U^{22}	U^{33}	U^{23}	U^{13}	U^{12}
Pd(1)	10(1)	10(1)	14(1)	1(1)	-1(1)	-2(1)
Cl(1)	12(1)	23(1)	33(1)	9(1)	-5(1)	-6(1)
P(1)	10(1)	11(1)	12(1)	0(1)	1(1)	-1(1)
O(1)	22(1)	12(1)	27(1)	1(1)	-3(1)	-1(1)
O(2)	11(1)	23(1)	18(1)	-1(1)	0(1)	-5(1)
O(3)	22(1)	19(1)	18(1)	-4(1)	-7(1)	2(1)
C(1)	13(1)	12(1)	19(1)	-1(1)	0(1)	0(1)
C(2)	17(1)	27(1)	22(1)	-5(1)	4(1)	2(1)
C(3)	23(1)	32(1)	38(1)	-18(1)	-1(1)	11(1)
C(4)	28(1)	15(1)	43(1)	-9(1)	-11(1)	6(1)
C(5)	11(1)	12(1)	13(1)	1(1)	1(1)	0(1)
C(6)	14(1)	10(1)	13(1)	0(1)	1(1)	-1(1)
C(7)	14(1)	14(1)	16(1)	1(1)	0(1)	-2(1)
C(8)	13(1)	26(1)	17(1)	0(1)	4(1)	0(1)
C(9)	19(1)	31(1)	14(1)	-4(1)	3(1)	1(1)
C(10)	16(1)	22(1)	13(1)	-1(1)	0(1)	-1(1)
C(11)	13(1)	30(1)	23(1)	4(1)	0(1)	-6(1)
C(12)	14(1)	15(1)	13(1)	0(1)	-1(1)	-2(1)
C(13)	26(1)	28(1)	18(1)	-2(1)	-10(1)	-2(1)
C(14)	19(1)	26(1)	20(1)	5(1)	-7(1)	-2(1)
C(15)	15(1)	18(1)	19(1)	3(1)	0(1)	-2(1)

Table 5. Hydrogen coordinates (x 10^4) and isotropic displacement parameters (≈^{2}x 10 ^{3}) for C30H24Cl2O6P2Pd.

	x	y	z	U(eq)
H(2)	11326	7587	-622	26
H(3)	11573	5303	-748	37
H(4)	10569	4327	45	34
H(8)	5140	8140	1124	22
H(9)	6612	8909	1722	26
H(10)	8636	8942	1456	20
H(11A)	4235	6736	626	33
H(11B)	3766	6827	-5	33
H(11C)	3909	8025	363	33
H(13)	11895	9675	2119	29
H(14)	12896	7743	1936	26
H(15)	11889	6835	1064	21

Table 6. Torsion angles [∞] for C30H24Cl2O6P2Pd.

P(1)#1-Pd(1)-P(1)-C(1)	84.04(5)
Cl(1)#1-Pd(1)-P(1)-C(1)	176.14(7)
Cl(1)-Pd(1)-P(1)-C(1)	-82.46(5)
P(1)#1-Pd(1)-P(1)-C(12)	-160.10(5)
Cl(1)#1-Pd(1)-P(1)-C(12)	-68.00(8)
Cl(1)-Pd(1)-P(1)-C(12)	33.39(5)
P(1)#1-Pd(1)-P(1)-C(5)	-40.61(4)
Cl(1)#1-Pd(1)-P(1)-C(5)	51.48(8)
Cl(1)-Pd(1)-P(1)-C(5)	152.88(5)
C(4)-O(1)-C(1)-C(2)	-0.11(16)
C(4)-O(1)-C(1)-P(1)	-178.70(10)
C(12)-P(1)-C(1)-C(2)	-111.98(15)
C(5)-P(1)-C(1)-C(2)	135.63(15)
Pd(1)-P(1)-C(1)-C(2)	9.24(17)
C(12)-P(1)-C(1)-O(1)	66.26(11)
C(5)-P(1)-C(1)-O(1)	-46.13(12)
Pd(1)-P(1)-C(1)-O(1)	-172.52(8)
O(1)-C(1)-C(2)-C(3)	0.03(18)
P(1)-C(1)-C(2)-C(3)	178.37(12)
C(1)-C(2)-C(3)-C(4)	0.06(19)
C(2)-C(3)-C(4)-O(1)	-0.13(19)
C(1)-O(1)-C(4)-C(3)	0.15(17)
C(1)-P(1)-C(5)-C(10)	139.33(11)
C(12)-P(1)-C(5)-C(10)	30.46(13)
Pd(1)-P(1)-C(5)-C(10)	-91.68(11)
C(1)-P(1)-C(5)-C(6)	-48.88(12)
C(12)-P(1)-C(5)-C(6)	-157.74(11)
Pd(1)-P(1)-C(5)-C(6)	80.11(11)
C(10)-C(5)-C(6)-C(7)	-3.4(2)
P(1)-C(5)-C(6)-C(7)	-174.91(10)
C(10)-C(5)-C(6)-C(6)#1	174.24(14)
P(1)-C(5)-C(6)-C(6)#1	2.69(19)
C(11)-O(2)-C(7)-C(8)	-6.1(2)
C(11)-O(2)-C(7)-C(6)	176.09(13)

C(5)-C(6)-C(7)-O(2)	-179.31(12)
C(6)#1-C(6)-C(7)-O(2)	3.1(2)
C(5)-C(6)-C(7)-C(8)	2.8(2)
C(6)#1-C(6)-C(7)-C(8)	-174.80(14)
O(2)-C(7)-C(8)-C(9)	-178.15(14)
C(6)-C(7)-C(8)-C(9)	-0.4(2)
C(7)-C(8)-C(9)-C(10)	-1.5(2)
C(8)-C(9)-C(10)-C(5)	1.0(2)
C(6)-C(5)-C(10)-C(9)	1.5(2)
P(1)-C(5)-C(10)-C(9)	173.38(12)
C(13)-O(3)-C(12)-C(15)	0.19(16)
C(13)-O(3)-C(12)-P(1)	-171.65(10)
C(1)-P(1)-C(12)-C(15)	2.38(16)
C(5)-P(1)-C(12)-C(15)	115.73(14)
Pd(1)-P(1)-C(12)-C(15)	-122.03(14)
C(1)-P(1)-C(12)-O(3)	172.27(11)
C(5)-P(1)-C(12)-O(3)	-74.38(12)
Pd(1)-P(1)-C(12)-O(3)	47.85(11)
C(12)-O(3)-C(13)-C(14)	-0.19(18)
O(3)-C(13)-C(14)-C(15)	0.13(19)
O(3)-C(12)-C(15)-C(14)	-0.11(16)
P(1)-C(12)-C(15)-C(14)	170.38(12)
C(13)-C(14)-C(15)-C(12)	-0.01(17)

Symmetry transformations used to generate equivalent atoms:

#1 y,x,-z