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Template effects on the asymmetric cycloaddition reaction between 3,4-dimethyl-1-phenylarsole and diphenylvinylphosphine and their arsenic elimination reaction

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ABSTRACT

The organopalladium complex containing *ortho*-metalated (*S*)-[1-(dimethylamino)ethyl]naphthalene as the chiral template was employed to promote the asymmetric cycloaddition reaction between 3,4-dimethyl-1-phenylarsole and diphenylvinylphosphine. It has been proven that the chiral template incorporating napthylamine is more efficient than the benzylamine based analogue as evidenced by the drastic improvement in stereoselectivity and reaction rate. However, when no chiral template was employed, *trans*-[PdI_2(3,4-dimethyl-1-phenylarsole)$_2$] reacted with *trans*-[PdI_2(diphenylvinylphosphine)$_2$] producing a structurally novel diiodo complex, as a result of an interesting selective cleavage of one As–C bond in the norbornene skeleton and subsequent rearrangements within the skeletal framework. The molecular structure of the diiodo product has been confirmed by X-ray crystallography. Structural analysis showed that in addition to the normal As–P five-membered ring, there is one new five-membered ring containing As–O bond being formed during the course of the reaction along with another seven-membered ring incorporating a hydroxy group.

1. Introduction

Quite recently, we have reported that the asymmetric cycloaddition reaction between diphenylvinylphosphine and 3,4-dimethyl-1-phenylarsole (DMPA) can be promoted by the organopalladium complex containing *ortho*-metalated (*S*)-[1-(dimethylamino)ethyl]phenylene as the chiral auxiliary, the arsenic donor in the dichloro and dibromo palladium complexes containing the diphenylphosphino-substituted asymmetrical heterobidentate arsanorbornene ligand could be eliminated stereospecifically under mild reaction conditions to generate the corresponding 1-(diphenylphosphino)-3,4-dimethyl-2,4-cyclohexadiene, which remained as a bidentate ligand at the $PdCl_2$ unit *via* phosphorus and the η^2-C–C double bond [1]. The arsenic elimination process in such cases was found to be influenced by the halo ligand in the [PdX_2(As–P)] complex.

Compared to the stable phosphole complexes in which a few phosphorus elimination reactions have been reported [2–4], arsenic elimination appeared to be a common phenomenon in the cycloaddition reactions involving arsoles [5–7]. For example, 2,3,4,5-tetramethyl-1-phenylarsole and 1,2,3,4,5-pentamethylarsole metal complexes readily undergo Diels–Alder reaction with acetylenedicarboxylic acid dimethyl ester. The 7-arsanorbornadienes formed as intermediates are unstable and decompose into arene and arsinidene complex which in one case has been trapped through consecutive insertion reactions [8].

In order to further understand the intricacies of the stereoselectivity and stability factors influencing the As–C bonds in such systems, we undertook a synthesis of the cycloadduct where the reaction was allowed to proceed under the assistance and influence of the corresponding naphthylamine auxiliary as well as in the absence of any chiral auxiliary. It was observed that the stereoselectivity of the Diels–Alder reaction between DMPA and diphenylvinylphosphine has been vastly improved using the organopalladium complex containing *ortho*-metalated (*S*)-[1-(dimethylamino)ethyl]naphthalene as the chiral template in comparison with the earlier reported cycloaddition [1]. More interestingly, the *trans*-[PdI_2(DMPA)$_2$] complex reacted with *trans*-[PdI_2(DPVP)$_2$] (DPVP = diphenylvinylphosphine) as a result of which a different mode of arsenic elimination reaction coupled with a novel skeletal rearrangement was found to occur.

2. Results and discussion

2.1. Asymmetric cycloaddition reaction between DMPA and diphenylvinylphosphine promoted by the Pd naphthylamine template

Treatment of complex (+)-**1** with silver perchlorate yielded the intermediate perchlorate complex in essentially quantitative yield. This highly reactive species was not isolated and upon subsequent

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removal of AgCl and excess silver perchlorate, the dichloromethane solution of the complex was treated directly with a stoichiometric amount of diphenylvinylphosphine (Scheme 1). This cycloaddition reaction was monitored by $^{31}P\{^{1}H\}$ NMR spectroscopy and was found to be completed within 40 min at room temperature. Prior to purification, the $^{31}P\{^{1}H\}$ NMR spectrum of the crude Diels–Alder reaction in $CDCl_3$ exhibited only two singlets at δ 50.2 and 48.6 in the ratio 19:1, thus indicating that only two stereochemically distinct cycloadducts were formed with high stereoselectivity being exercised. Compared to the same reaction conducted using benzylamine auxiliary as chiral template, the selectivity improved roughly six fold (from 3.2:1 to 19:1) and the reaction time reduced significantly from 3 h to 40 min [1]. The reason is that the naphthylamine auxiliary is superior to the benzylamine analogue in certain asymmetric synthesis scenarios due to the unique stereochemistry associated with the rigid *ortho*-metalated naphthylamine ring [9]. For example, there is an internal steric repulsion between the methyl substituent on the stereogenic carbon and its neighboring naphthylene proton in complex (+)-**1**. The crystallographic determinations and rotating overhauser effect (ROESY) NMR investigations confirmed that the organometallic ring is locked into the static λ conformation, both in the solid state and in solution [10]. Thus the prochiral NMe groups are fixed into the non-equivalent axial and equatorial positions. These NMe groups control the stereochemistry of the neighboring coordination sites. On the other hand, the stereochemistry of the five-membered organometallic ring in the corresponding benzylamine complex cannot be well defined, as the puckered benzylamine ring undergoes rapid transformation between the two non-equivalent δ and λ conformations in solution.

The major diastereomer (−)-**2** was obtained as pale yellow needles from dichloromethane–diethyl ether in 73% isolated yield, $[\alpha]_D = -33.0°$ (*c* 0.6, CH_2Cl_2). The molecular structure and absolute confirmation of complex (−)-**2** have been resolved by X-ray crystallography (Fig. 1). Selected bond lengths and angles are listed in Table 1. It is structurally the same as the one obtained by utilizing the analogous benzyl complex, the arsenic is *trans* to the aromatic carbon and the phosphorus is *trans* to nitrogen as expected from the hard soft preference exerted by the template in such systems. Four new chiral centers have been generated with *R* absolute stereochemistry at As(1) and *R*, *R* and *S* stereochemistry at C(21), C(26) and C(27). The naphthylamine auxiliary could be removed chemoselectively from (−)-**2** by treatment with concentrated hydrochloric acid to generate the dichloro complex (−)-**3** at room temperature. The same phenomenon as that in the case of our earlier report was observed with the two As–C bridgehead bonds in

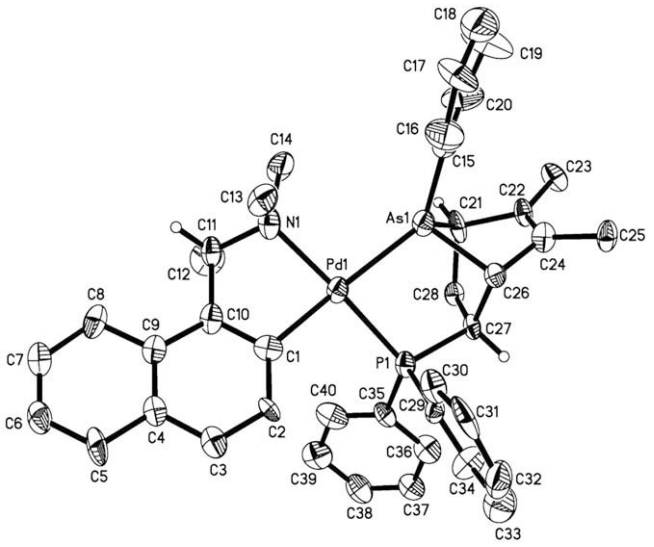
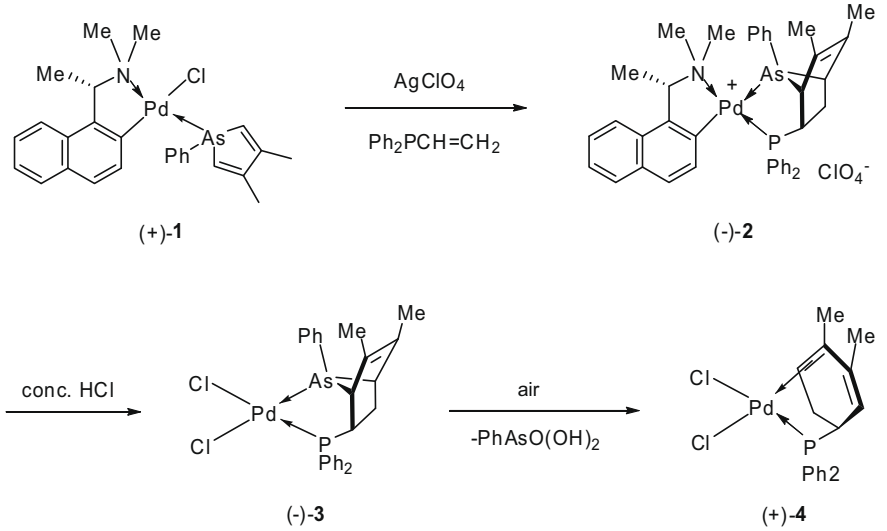


Fig. 1. Molecular structure of complex (−)-**2**.

Table 1
Selected bond lengths (Å) and angles (°) for complex (−)-**2**.

Pd1–C1	2.024(14)	Pd1–N1	2.095(13)
Pd1–As1	2.443(2)	Pd1–P1	2.264(4)
As1–C21	1.984(13)	As1–C26	1.968(14)
C21–C28	1.57(2)	C21–C22	1.579(19)
C22–C24	1.277(18)	C24–C26	1.548(19)
C26–C27	1.590(18)	C27–C28	1.540(17)
C1–Pd1–N1	82.3(5)	C1–Pd1–P1	95.7(4)
N1–Pd1–P1	174.0(3)	C1–Pd1–As1	173.7(4)
N1–Pd1–As1	99.4(3)	P1–Pd1–As1	83.2(1)
C26–As1–C21	77.5(6)	C28–C21–C22	105.3(12)
C24–C22–C21	112.0(12)	C22–C24–C26	113.0(13)
C24–C26–C27	110.0(10)	C28–C27–C26	105.0(10)
C27–C28–C21	109.1(10)		



Scheme 1.

(−)-**3** easily collapsing to give the new η^2-P palladium complex (+)-**4** [1].

2.2. Cycloaddition reaction between DMPA and diphenylvinylphosphine without template

DMPA was coordinated to the $[PdCl_2(NCMe)_2]$, resulting in the *cis* and *trans* isomeric mixture of the dichloro complex **5** with the two DMPA ligands coordinated to the Pd center (Scheme 2). In fact, the mixture of *trans* and *cis* dichloro complexes of **5** need not be separated because when the complex **5** was subsequently converted to the analogous diiodo complex **6** in high yield (97%) by treatment with sodium iodide at room temperature, only the *trans* isomer was obtained. This can be confirmed by the single crystal X-ray analysis of complex **6** in which the arsenic is *trans* to each other (Fig. 2). Selected bond lengths and angles are listed in Table 2. The geometry at palladium is a distorted square planar with bond angles at metal center ranging between 87.7(1)–92.3(1) and 180.0(1)°.

The diiodo complex **6** was then allowed to react with *trans*-$[PdI_2(DPVP)_2]$ at room temperature. The Diels–Alder reaction was monitored by the $^{31}P\{^1H\}$ NMR spectrum. Analysis of the crude reaction mixture indicated the formation of several products amongst which complex **7** was subsequently isolated by recrystallization and other products cannot be isolated. The molecular structure of complex **7** was confirmed by X-ray crystallography and indicated the formation of a ligand system was the result of a very unusual rearrangement in norbornene skeletal system formed as an intermediate (Fig. 3). Selected bond lengths and angles are listed in Table 3. Upon further detailed analysis of the $^{31}P\{^1H\}$ NMR spectral data of the reaction it was seen that when the two *trans* diiodo complexes, **6** and $[PdI_2(DPVP)_2]$, were mixed together, a new peak at δ 13.0 appeared immediately which can be attributed to the *trans* or *cis* intermediate $[PdI_2(DMPA)(DPVP)]$ [11]. After 1 day, the Diels–Alder reaction product was found to be formed as observed from the $^{31}P\{^1H\}$ signal at δ 37.7 which is identical to the value obtained for the enantiomerically pure

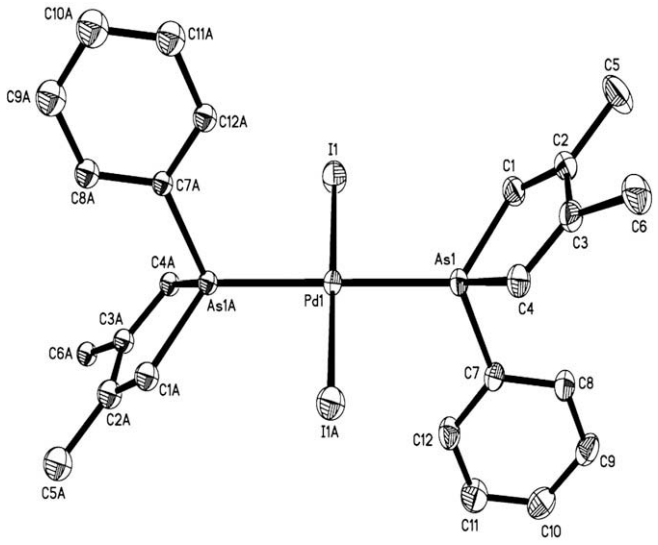
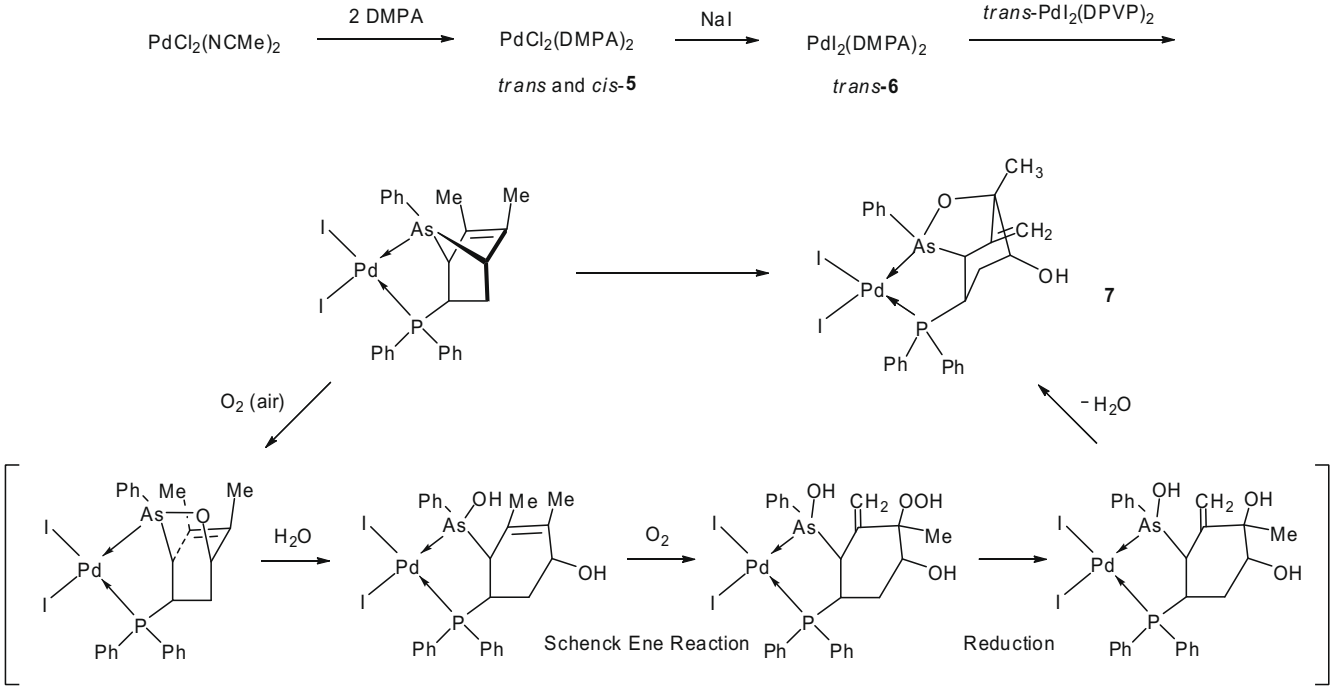


Fig. 2. Molecular structure of complex **6**.

Table 2
Selected bond lengths (Å) and angles (°) for complex **6**.

Pd1–I1	2.600(1)	Pd1–I1A	2.600(1)
Pd1–As1	2.394(1)	Pd1–As1A	2.394(1)
As1–C1	1.911(2)	As1–C4	1.916(2)
C1–C2	1.341(3)	C2–C3	1.497(3)
C2–C5	1.496(3)	C3–C4	1.340(3)
C3–C6	1.500(3)		
I1–Pd1–I1A	180.0(1)	I1A–Pd1–As1	87.7(1)
I1–Pd1–As1	92.3(1)	I1A–Pd1–As1A	92.3(1)
I1–Pd1–As1A	87.7(1)	As1–Pd1–As1A	180.0(1)
C1–As1–C4	88.0(1)	C2–C1–As1	110.5(1)
C1–C2–C3	115.4(2)	C4–C3–C2	115.9(2)
C3–C4–As1	110.1(2)		



Scheme 2.

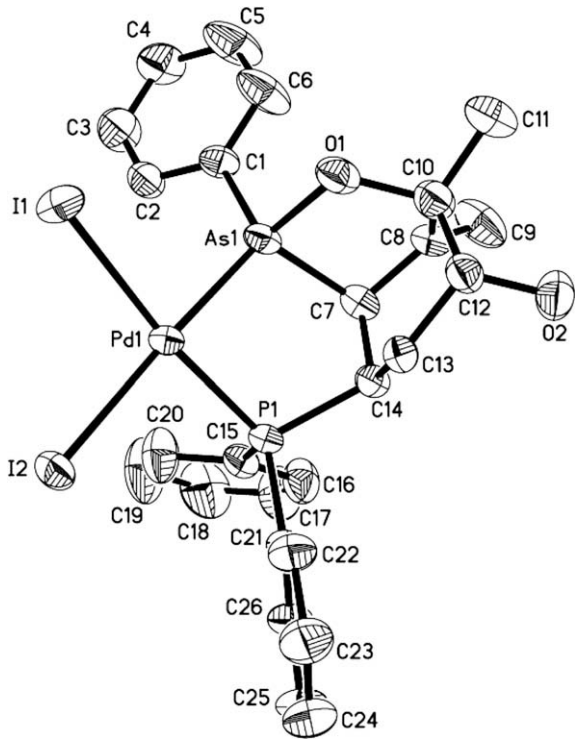


Fig. 3. Molecular structure of complex **7**.

Table 3
Selected bond lengths (Å) and angles (°) for complex **7**.

Pd1–P1	2.276(1)	Pd1–As1	2.345(1)
Pd1–I2	2.620(1)	Pd1–I1	2.649(1)
As1–O1	1.786(4)	As1–C7	1.976(6)
C7–C8	1.490(7)	C7–C14	1.537(7)
C8–C9	1.304(9)	C8–C10	1.509(8)
C10–C11	1.516(8)	C10–O1	1.531(7)
C10–C12	1.549(8)	C12–O2	1.416(7)
C12–C13	1.525(7)	C13–C14	1.553(7)
P1–Pd1–As1	83.7(1)	P1–Pd1–I2	91.8(1)
As1–Pd1–I2	173.4(1)	P1–Pd1–I1	172.7(1)
As1–Pd1–I1	89.2(1)	I2–Pd1–I1	95.4(1)
O1–As1–C7	89.6(2)	C8–C7–C14	112.9(5)
C9–C8–C7	125.9(6)	C9–C8–C10	126.8(6)
C7–C8–C10	107.3(5)	C8–C10–O1	106.9(5)
C8–C10–C12	108.5(5)	O1–C10–C12	106.5(4)
O2–C12–C13	113.0(5)	O2–C12–C10	107.1(5)
C13–C12–C10	111.2(4)	C12–C13–C14	116.3(4)
C7–C14–C13	111.7(4)		

version of the cycloadduct obtained as part of our previous studies involving DMPA and diphenylvinyphosphine albeit in that case the reaction was promoted by the chiral auxiliary [1]. Cyclic phosphine oxides with contracted internal C–P–C angles have been found to undergo oxygen insertion into a C–P bond [12,13]. The same phenomenon may occur in the cycloadduct resulting in oxygen being inserted into the As–C bond of the complex and thereafter, due to the inherent instability of such compounds, it results in a hydrolysis [12]. Subsequently a Schenck ene reaction [14] caused double bond migration to a methyl group [15]. Finally, the reduction of the peroxide [16] gave the alcohol intermediate which eliminated a molecular of water to yield the complex **7**. Taking into consideration the low yield and numerous unidentified side products obtained along with the fact that the presence of phenylarsonic acid in similar reactions have been reported earlier [1], the possibility of the acid acting as a reductant in this reaction could not be ruled out.

The diiodo complex **7** is very different from the η^2-P palladium complexes formed as a result of As–C bridghead bond collapse, only one As–C bond broke in this case, and meanwhile one new five-member ring containing As–O and the seven-member ring containing hydroxyl group were generated during the course of the reaction. To our knowledge this is the first instance of such unique bridgehead collapse coupled with rearrangement observed in cyclic arsenic systems. We are currently in the process of fine-tuning the conditions in order to improve the yield of the reaction which will allow us to do a more thorough investigation of the mechanism involved as well as pave the way for potential application of the ligand system in various catalytic scenarios.

In conclusion, we have found that the template has a vital effect on the reaction between DMPA and diphenylvinylphosphine. Compared to the benzyl analogue, the naphthylamine is a better chiral auxiliary in exerting stereospecific control over the formation of the hetero bidentate As–P ligand. However, in the absence of any template, a new arsenic elimination coupled with ring rearrangement was observed. Finally, it is confirmed that it is a common feature that As–C bonds cleaved in such arsanorbornene (As–P) units but interestingly there was a selective cleavage of only one As–C bridgehead bond in the latter scenario where no chiral template was employed. We are currently investigating the reactions employing other substrates in order to further understand the processes involved in both the template promoted and non-template promoted reactions involving cyclic arsines.

3. Experimental

Reactions involving air-sensitive compounds were performed under an inert atmosphere of argon using standard Schlenk techniques. Solvents were dried and freshly distilled according to standard procedures and degassed prior to use when necessary. The ^{1}H and ^{31}P{^{1}H} NMR spectra were recorded at 25 °C on Bruker Avance 300 and 400 spectrometers. Optical rotations were measured on the specified solution in a 0.1 dm cell at 20 °C with a Perkin–Elmer 341 polarimeter. Elemental analysis was performed by the Elemental Analysis Laboratory of the Division of Chemistry and Biological Chemistry at Nanyang Technological University. Melting points are uncorrected.

(+)-**1** [17], diphenylvinylphosphine [18] and DMPA [19] were prepared following the literature procedures.

Caution! Perchlorate salts of metal complexes are potentially explosive compounds and should be handled with care.

3.1. Cycloaddition reaction: preparation of complex (−)-*2*

A solution of (+)-**1** (0.59 g, 1.03 mmol) in dichloromethane (50 mL) was stirred for 2 h in the presence of a solution of silver perchlorate (0.36 g) in water (1 mL). The organic layer, after the removal of AgCl, was then washed with water (3 × 50 mL), dried ($MgSO_4$), and subsequently treated with diphenylvinylphosphine (0.22 g, 1.03 mmol) for 40 min at room temperature. Removal of the solvent gave (−)-**2** as a yellow solid, which was then recrystallized from dichloromethane–diethyl ether to give the complex as pale yellow needle crystals (0.66 g, 73%). $[\alpha]_D = -33.0°$ (*c* 0.6, CH_2Cl_2). M.p.: 161–162 °C. Anal. Calc. for $C_{40}H_{42}AsClNO_4PPd \cdot 1/3CH_2Cl_2$: C, 55.3; H, 4.9; N, 1.6. Found: C, 55.7; H, 5.0; N, 1.7%. ^{31}P{^{1}H} NMR ($CDCl_3$, δ): 50.2. ^{1}H NMR ($CDCl_3$, δ): 1.41 (s, 3H, =CCH_3), 1.61 (s, 3H, =CCH_3), 1.92 (m, 1H, CHCH_2), 2.03 (d, $^3J_{HH}$ = 6.1 Hz, 3H, CHCH_3), 2.52 (dd, $^3J_{PH}$ = 24.5, $^2J_{HH}$ = 13.6 Hz, 1H, CHCH_2), 2.79 (s, 3H, NCH_3), 2.84 (s, 1H, As*CH*), 2.93 (d, $^4J_{PH}$ = 3.1 Hz, 3H, NCH_3), 3.15 (t, $^3J_{HH}$ = 9.0 Hz, 1H, P*CH*), 3.81 (s, 1H, As*CH*), 4.46 (qn, $^3J_{HH} = {}^4J_{PH}$ = 5.9 Hz, 1H, *CH*CH_3), 6.68–8.26 (m, 21H, aromatics).

Table 4
Crystallographic data for complexes (°)-**2**, **6** and **7**.

	(−)-**2**	**6**	**7**
Formula	$C_{40}H_{42}AsClNO_4PPd \cdot 1/3CH_2Cl_2$	$C_{24}H_{26}As_2I_2Pd$	$C_{26}H_{26}AsI_2O_2PPd \cdot 1/2CH_2Cl_2$
Formula weight	876.79	824.49	878.52
Space group	*C*2	*C*2/c	$P\bar{1}$
Crystal system	Monoclinic	Monoclinic	Triclinic
a (Å)	30.3585(16)	17.0458(9)	9.9521(3)
b (Å)	17.5355(7)	7.5236(3)	11.7499(4)
c (Å)	25.7191(8)	20.0657(8)	14.3409(5)
α (°)	90	90	108.790(2)
β (°)	90.066(5)	95.381(3)	91.610(2)
γ (°)	90	90	111.672(2)
V (Å^3)	13691.6(10)	2562.0(2)	1454.62(8)
Z	12	4	2
T (K)	173(2)	173(2)	173(2)
D_{calc} (g cm^{-3})	1.276	2.138	2.006
λ (Å)	0.71073	0.71073	0.71073
μ (mm^{-1})	1.294	5.707	4.058
F(000)	5352	1552	837
GOF on F^2	0.909	1.135	1.038
R_1 (observed data)	0.0864	0.0179	0.0459
wR_2 (observed data)	0.2032	0.0428	0.1041

3.2. Synthesis of the dichloro complex **5**

The complex $[PdCl_2(NCMe)_2]$ (0.11 g, 0.43 mmol) and the ligand DMPA (0.20 g, 0.86 mmol) in dichloromethane (60 mL) were stirred at room temperature overnight. The solvent was removed and the residue was crystallized with chloroform–diethyl ether to give the product (*trans* and *cis* mixture) as yellow crystals (0.45 g, 82%), which was further recrystallized with dichloromethane–diethyl ether to produce pure *trans* isomer **5**. M.p.: 177–178 °C. Anal. Calc. for $C_{24}H_{26}As_2Cl_2Pd$: C, 44.9; H, 4.1. Found: C, 44.4; H, 4.4%. ^{1}H NMR ($CDCl_3$, δ): 2.04 (s, 6H, *CH*$_3$), 6.60 (s, 2H, =*CH*), 7.33–7.62 (m, 5H, aromatics).

3.3. Synthesis of the trans-diiodo complex **6**

The solution of $[PdCl_2(DMPA)_2]$ (*trans* and *cis* mixture) (0.45 g, 0.70 mmol) in dichloromethane (50 mL) was added to sodium iodide (0.5 g) in acetone (50 mL) and was stirred vigorously for 10 min. The solvent was removed and the residue was extracted with dichloromethane, Removal of solvent gave **6** as a solid, which was then recrystallized from dichloromethane–diethyl ether to give the product as red crystals (0.56 g, 97%). M.p.: 192–193 °C. Anal. Calc. for $C_{24}H_{26}As_2I_2Pd$: C, 35.0; H, 3.2. Found: C, 34.8; H, 3.1%. ^{1}H NMR ($CDCl_3$, δ): 2.11 (s, 6H, *CH*$_3$), 7.03 (s, 2H, =*CH*), 7.33–7.72 (m, 5H, aromatics).

3.4. Synthesis of the diiodo complex **7**

The complexes $[PdI_2(DMPA)_2]$ (0.45 g, 0.55 mmol) and $[PdI_2(DPVP)_2]$ (0.43 g, 0.55 mmol) in dichloromethane (90 mL) were stirred at 30 °C for 5 days. The solvent was removed and the residue was recrystallized with dichloromethane–diethyl ether from −78 °C to room temperature to produce brown crystals (0.05 g, 5%). M.p.: 180–181 °C. ^{31}P{^{1}H} NMR (CD_2Cl_2, δ): 79.0. ^{1}H NMR (CD_2Cl_2, δ): 1.64 (s, 3H, C*CH*$_3$), 2.04 (s, 1H, O*CH*), 2.74 (m, 1H, CH*CH*$_2$), 3.02 (d, $^4J_{HH}$ = 3.4 Hz, 1H, As*CH*), 3.26 (m, 1H, CH*CH*$_2$), 3.92 (d, $^2J_{PH}$ = 2.6 Hz, 1H, P*CH*), 5.00 (s, 1H, =*CH*$_2$), 5.13 (d, $^4J_{HH}$ = 4.2 Hz, 1H, =*CH*$_2$), 7.00–8.19 (m, 15H, aromatics). ^{13}C NMR (CD_2Cl_2, δ) [20]: 20.1, 30.5, 38.6, 38.9, 73.1, 73.2, 86.6, 113.9, 125.2, 128.1, 128.2, 128.7, 129.8, 129.9, 130.3, 132.1, 132.4, 132.9, 134.1, 134.2, 135.0, 135.1, 136.6. EI-MS: *m*/*z* 837.3, $[M]^+$.

3.5. X-ray crystal structure determination of complexes (−)-**2**, **6** and **7**

Crystal data for all complexes and a summary of the crystallographic analysis are given in Table 4. Diffraction data were collected on a Bruker X8 CCD diffractometer with Mo Kα radiation (graphite monochromator). SADABS absorption corrections were applied. All non-hydrogen atoms were refined anisotropically, while the hydrogen atoms were introduced at calculated positions and refined riding on their carrier atoms. The absolute configuration of the chiral complex was determined unambiguously by using the Flack parameter.

4. Supplementary material

CCDC 694659, 694661 and 649745 contain the supplementary crystallographic data for (−)-**2**, **6** and **7**. These data can be obtained free of charge from the Cambridge Crystallographic Data Centre *via* www.ccdc.cam.ac.uk/data_request/cif.

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