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Synthesis and structure of the chiral palladium–fullerene C60 and C70 complexes with enantiomeric ligand 2,2′,5,5′-tetramethyl-4,4′-bis(diphenylphosphino)-3,3′-bithiophene [(−)tetraMe-BITIOP]

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Abstract

η^2-Fullerene (C_{60} and C_{70}) palladium optically active complexes with the axially chiral enantiomeric ligand of bithienyl series, [(−)tetraMe-BITIOP], have been synthesized and investigated by ^{31}P–$\{^1H\}$ NMR, electronic spectroscopy, CD spectroscopy, cyclic voltammetry and by single crystal X-ray diffraction.
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1. Introduction

Coordinatively unsaturated phosphine complexes of 10 group metals (Pt, Pd) are known to add on the double bonds of fullerenes to afford *exo*-metal η^2-fullerene complexes [1], however, a mixture of adducts is often formed with different number of the metal fragments up to six [2]; organometallic complexes of fullerenes have been reviewed [3]. We have been interested for several years in developing new routes leading selectively to *mono*-metallated fullerenes. It was found that fullerenes are able to abstract the ML_2 moiety from such compounds as $RHgPtL_2R'$ [4] or R_2PtL_2 [5]. The most practical preparation of palladium η^2-complexes appeared

to be the direct synthesis using Pd_2dba_3, fullerene and a free phosphine ligand [6–8].

Keeping in mind the catalytic activity of palladium η^2-fullerene complexes found in the hydrogenation reaction [9,10], one can expect that the optically active analogues might be interesting for enantioselective catalysis. In addition, the fullerene core is a chromophore with many absorption bands in the electronic spectra; at least some of them may be optical active in a chiral environment and therefore interesting for chiroptical study.

However, just few optically active organometallic fullerene derivatives have been reported till present time. More than ten years ago we have synthesized the first optically active organometallic η^2-C_{60} complex with palladium [6], then with platinum [11] using a well-known bidentate enantiomeric tertiary diphosphine ligands (+)DIOP (**1**) (CD spectra of (η^2-C_{60})M[(+)DIOP],

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M = Pd, Pt in different solvents were reported elsewhere [12]). Later on the chiral palladium complex with racemic monodentate ligand phenylbenzylferrocenylphosphine (2) (η^2-C$_{60}$)Pd(PPhBnFc)$_2$ has been prepared [13]; for review of all chiral and optically active derivatives of fullerenes see [14]. Recently our work [11] was repeated by another research group with (−)DIOP enantiomer to afford the same results in X-ray structure and CD spectra [15]. In addition, optically active η^2-C$_{60}$ molybdenum and tungsten complexes have been reported with enantiomeric bidentate ligands (η^2-C$_{60}$)M(CO)$_3$[L-L], where [L-L] is either the same DIOP [15] or the optically active *N,N*-ligand (what is unusual for η^2-fullerenyl metal complexes) bpy*-(+) or (−) 4,5-pinenobipyridine (M = Mo only) [16].
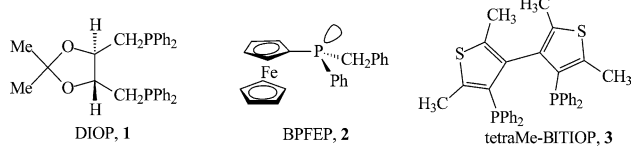
Ligands **1** and **2** have chiral centres at either carbon or phosphorus atoms. Now we wish to report on the synthesis of the palladium–fullerene complexes with the enantiomeric ligand **3** with axial chirality.

2. Results and discussion

Enantiomeric ligand 2,2′,5,5′-tetramethyl-4,4′-bis(diphenylphosphino)-3,3′-bithienyl, (−)-(tetraMe-BITIOP), **3**, had been synthesized previously and its chiroptical properties investigated. Chirality of this molecule is of axial nature (atropoisomerism) due to the restricted rotation around the thiophene–thiophene bond assisted by four substituents in α,β-positions. tetraMe-BITIOP (in the form of Ru or Rh complexes) is

one of the most efficient chiral chelate ligands for enantioselective homogeneous hydrogenation [17].

When a fullerene (C$_{60}$ or C$_{70}$) reacts with Pd$_2$(dba)$_3$·C$_6$H$_6$ (**4**), a known synthetic equivalent of palladium atom, in toluene or benzene in the presence of **3**, colour of the solution immediately turns to green or brown, correspondingly, and crystals of the products **5** or **6** separate slowly in nearly quantitative yield (Scheme 1).

The crystalline samples of (η^2-C$_{60}$) Pd(tetraMe–BITIOP)·Et$_2$O (**5**) and (η^2-C$_{70}$)Pd(tetraMe-BITIOP) (**6**) were characterized by elemental analysis. Complexes **5** and **6** are sparingly soluble in most common solvents but are sufficiently soluble in 1,2-dichlorobenzene (ODCB) to permit recording of their NMR spectra. ^{31}P–^{1}{H} NMR spectrum of **5** exhibited a single resonance at 22.9 ppm that is deshielded as compared with that of the free ligand, −21.1 ppm. Both phosphorus nuclei are equivalent. The coordination shift for **5** is 44.0 ppm. On the contrary, ^{31}P–^{1}{H} NMR spectrum of **6** showed two doublets of magnetically nonequivalent phosphorus nuclei (Fig. 1). This is a typical AB system with δ(P$_A$) 23.6 ppm, δ(P$_B$) 23.2 ppm, J(P$_A$,P$_B$) = 23.2 Hz. The coordination shifts for **6** are 44.7 and 44.3 ppm. This pattern is characteristic of the metal η^2 complexes formed on the most reactive C$_a$–C$_b$ bond in C$_{70}$.

The electronic absorption spectra of **5** and **6** in toluene (Fig. 2) are similar to those of C$_{60}$ and C$_{70}$. Broad peaks occur at positions: [λ_{max} (nm), log ε, molar extinction coefficients]; 665 (3.50), 624 (3.88), 452 (4.10), 355 (4.59) (for **5**), and 695 (3.74), 603 (3.88), 471 (4.29), 380 (4.31) (for **6**). The most characteristic bands observed in the spectra of **5** and **6** are the charge-transfer bands 452 and 471 nm correspondingly.

CD spectra of **5** and **6** were registered in toluene as well (Fig. 3); they exhibited ill-resolved Cotton effects shifted to longer wavelengths compared with the
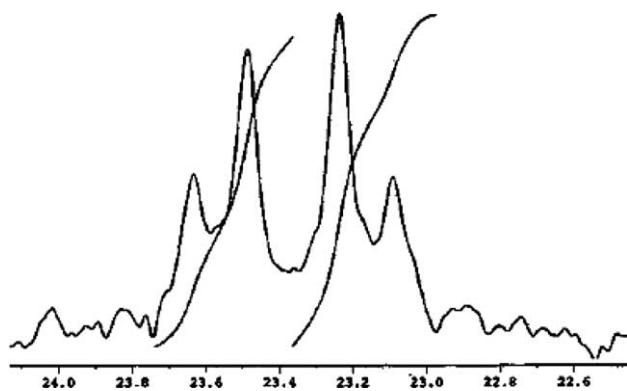

Ligands: DIOP, **1**; BPFEP, **2**; tetraMe-BITIOP, **3**
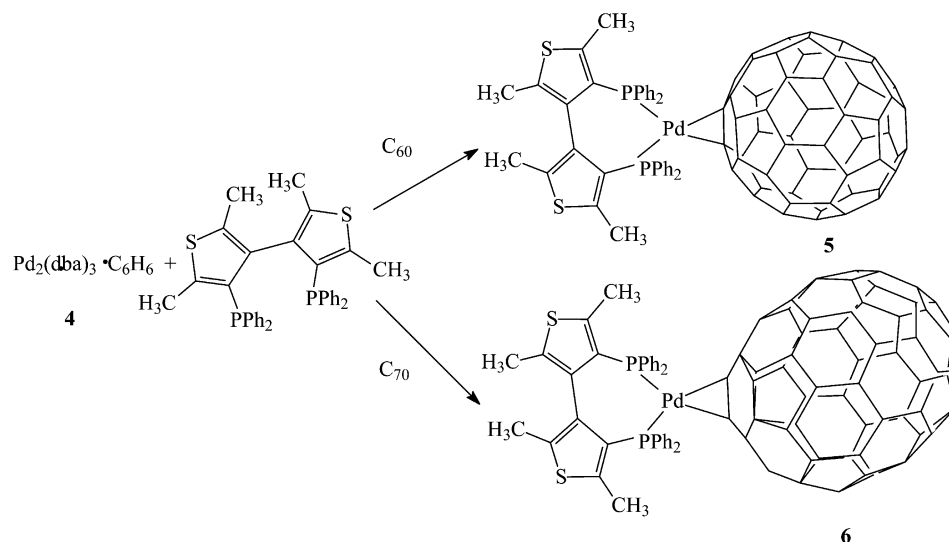
Scheme 1.

Fig. 1. $^{31}P–\{^1H\}$ NMR spectrum of **6** in 1,2-dichlorobenzene (ODCB).

starting ligand **3** that probably points to the electron density transfer from the ligand to fullerene as in other palladium–fullerene complexes [14]. $R(-)$ absolute configurations for the ligand **3** previously deduced from CD spectra only has been now confirmed by direct X-ray study of the complex **5** (see below).

Electrochemical experiments were performed at 25 °C under nitrogen in three-electrode cells. The compound **5** was dissolved in 1,2-dichlorobenzene (ODCB) with tetrabutylammonium perchlorate (TBAP) previously dried under vacuum at 70 °C as supporting electrolyte. The counter-electrode was platinum and the reference electrode was a silver/0.1 M silver perchlorate in acetonitrile (0.39 V versus Ag/AgCl). The working electrode for cyclic voltammetry was a platinum minidisk electrode (0.003 cm^2).

The cyclic voltammogram of the compound **5** (1×10^{-3} M) in ODCB + 0.1 M TBAP displays in the reductive scan three one-electron reversible processes

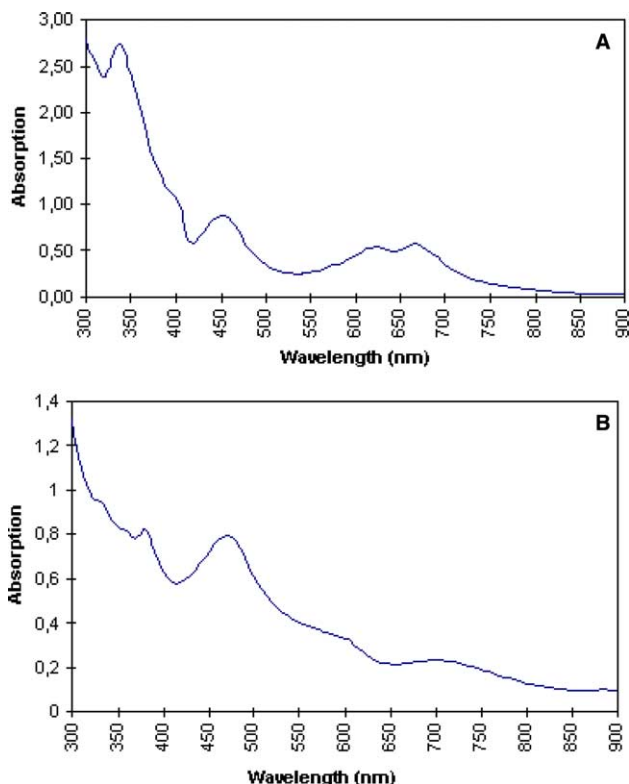Fig. 2. Visible–ultraviolet absorption spectra of **5** (A) and **6** (B) in toluene.

typical of C$_{60}$ (Fig. 4). They are displayed at $E_{1/2}=$ -1.11; -1.49; and -1.95 V. The three waves are shifted to more negative potential by about 0.05 V relative to those of free C$_{60}$ that is consistent with the presence of a donating ligand. Analogous by sign shifts of the reduc-
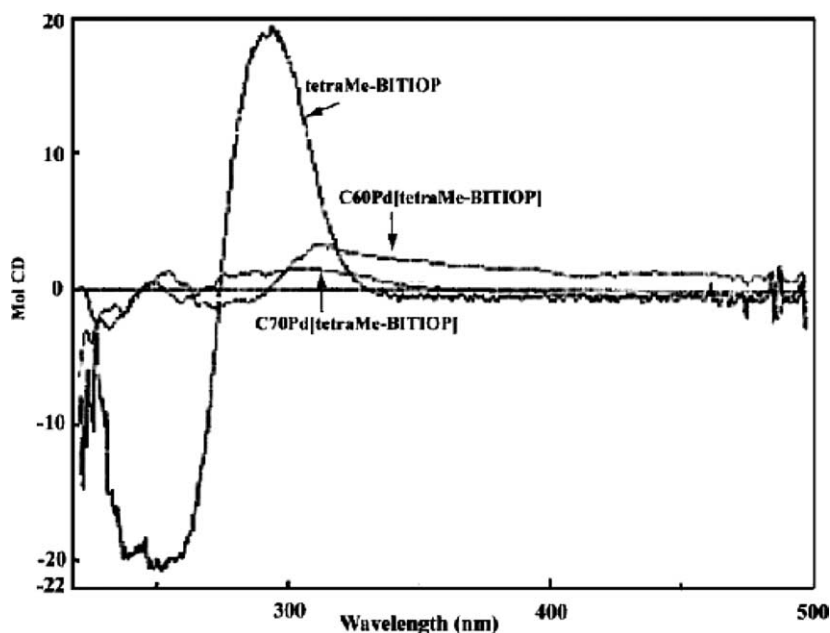

Fig. 3. Circular dichroism spectra of the complexes **5** and **6** together with the ligand **3**.
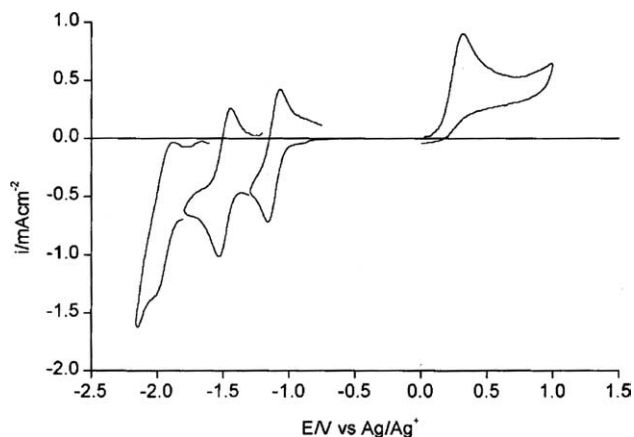
Fig. 4. Cyclic voltammogram for $(\eta^2-C_{60})Pd[(-)tetraMe-BITIOP]$ in ODCB + 0.1 M TBAP. Scan rate: 0.1 V/s.

tion waves were recently observed [18] in the electrochemical study of palladium–fullerene complexes with metallocene-phosphine ligands. In the positive scan the compound **5** is irreversibly oxidized in a two-electron process at $E_p = 0.32$ V.

The molecular structure of **5**·Et$_2$O was established by a single-crystal X-ray study. The structure of complex **5** with the atomic numbering scheme is shown in Fig. 5; important geometrical parameters are listed in Table 1.
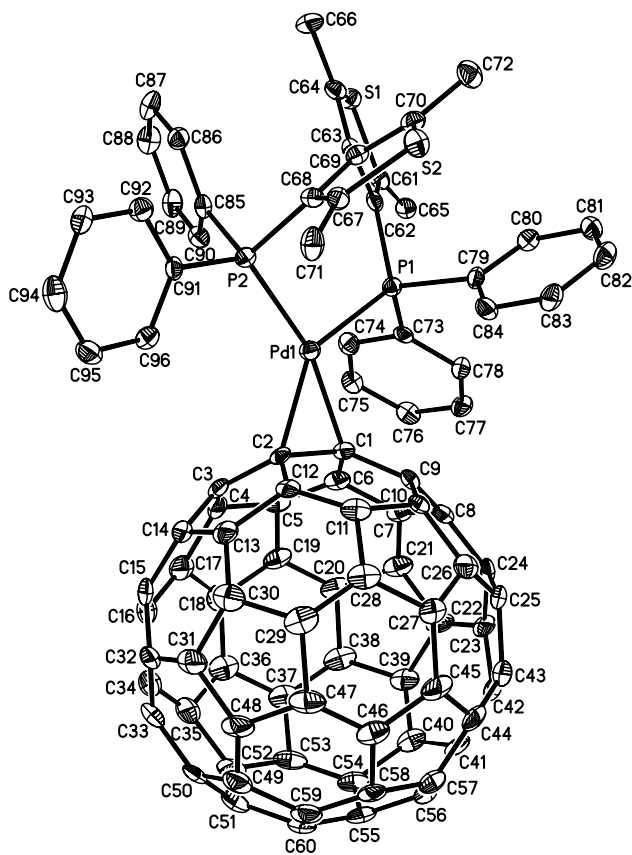

Fig. 5. ORTEP drawing of $(\eta^2-C_{60})Pd[(-)tetraMe-BITIOP]$. Hydrogen atoms are omitted for clarity.

Table 1
Selected bond lengths and angles for **5**

Bond lengths (Å)	
Pd(1)–P(1)	2.327(2)
Pd(1)–P(2)	2.299(2)
Pd(1)–C(1)	2.083(5)
Pd(1)–C(2)	2.135(6)
S(1)–C(61)	1.735(6)
S(1)–C(64)	1.726(6)
S(2)–C(67)	1.722(6)
S(2)–C(70)	1.710(6)
P(1)–C(62)	1.824(6)
P(1)–C(73)	1.802(6)
P(1)–C(79)	1.818(6)
P(2)–C(68)	1.812(6)
P(2)–C(85)	1.835(6)
P(2)–C(91)	1.818(6)
C(1)–C(2)	1.482(8)
C(1)–C(9)	1.461(8)
C(1)–C(6)	1.489(8)
C(2)–C(3)	1.464(8)
C(2)–C(12)	1.483(8)
C(61)–C(62)	1.358(8)
C(61)–C(65)	1.483(8)
C(62)–C(63)	1.451(7)
C(63)–C(64)	1.352(8)
C(63)–C(69)	1.487(8)
C(64)–C(66)	1.476(8)
C(67)–C(68)	1.347(8)
C(67)–C(71)	1.520(8)
C(68)–C(69)	1.452(8)
C(69)–C(70)	1.364(9)
C(70)–C(72)	1.520(9)

Bond angles (°)	
C(1)–Pd(1)–C(2)	41.1(2)
C(1)–Pd(1)–P(2)	160.6(2)
C(2)–Pd(1)–P(2)	119.6(2)
C(1)–Pd(1)–P(1)	101.5(2)
C(2)–Pd(1)–P(1)	142.6(2)
P(2)–Pd(1)–P(1)	97.73(6)
C(64)–S(1)–C(61)	93.0(3)
C(70)–S(2)–C(67)	92.2(3)
C(73)–P(1)–C(79)	108.8(3)
C(73)–P(1)–C(62)	105.3(2)
C(79)–P(1)–C(62)	102.9(3)
C(73)–P(1)–Pd(1)	103.7(2)
C(79)–P(1)–Pd(1)	114.0(2)
C(62)–P(1)–Pd(1)	121.6(2)
C(68)–P(2)–C(91)	106.2(3)
C(68)–P(2)–C(85)	105.4(3)
C(91)–P(2)–C(85)	104.0(3)
C(68)–P(2)–Pd(1)	104.2(2)
C(91)–P(2)–Pd(1)	121.8(2)
C(85)–P(2)–Pd(1)	114.0(2)
C(62)–C(61)–C(65)	133.0(6)
C(62)–C(61)–S(1)	109.8(5)
C(65)–C(61)–S(1)	117.1(4)
C(61)–C(62)–C(63)	113.6(6)
C(61)–C(62)–P(1)	128.0(5)
C(63)–C(62)–P(1)	118.4(4)
C(64)–C(63)–C(62)	112.3(5)
C(64)–C(63)–C(69)	123.5(5)
C(62)–C(63)–C(69)	123.2(5)
C(63)–C(64)–C(66)	128.7(6)
C(63)–C(64)–S(1)	111.1(4)

Table 1 (*continued*)

C(66)–C(64)–S(1)	120.2(4)
C(68)–C(67)–C(71)	130.6(6)
C(68)–C(67)–S(2)	112.1(5)
C(71)–C(67)–S(2)	117.2(4)
C(67)–C(68)–C(69)	111.9(5)
C(67)–C(68)–P(2)	125.3(4)
C(69)–C(68)–P(2)	121.4(4)
C(70)–C(69)–C(68)	112.3(5)
C(70)–C(69)–C(63)	119.6(5)
C(68)–C(69)–C(63)	127.9(5)
C(69)–C(70)–C(72)	127.0(6)
C(69)–C(70)–S(2)	111.5(5)
C(72)–C(70)–S(2)	121.4(5)

Palladium atom is η^2-coordinated to C_{60} on (6:6) bond C(1)–C(2) with non-equal bond lengths Pd–C(1) 2.083(5) Å and Pd–C(2) 2.135(6) Å. Due to coordination C(1)–C(2) bond is much longer [1.482(8) Å] than the non-coordinated (6:6) bonds having the average length 1.391 Å.

Palladium atom has a slightly distorted planar-square coordination, dihedral angle between planes P(1)–Pd–P(2) and C(1)–Pd–C(2) being 2.6°. Two similar Pd–P bonds have also different lengths (see Table 1), in addition, they are inclined to the fullerene core in a different way (angles are following: P(1)–Pd–C(2) 142.6(2)° and P(2)–Pd–C(1) 160.6(2)°). This may be due to the non-identical orientations of phenyl groups C(73)–C(78) and C(91)–C(96) at P(1) and P(2) correspondingly, torsion angles being C(74)–C(73)–P(1)–Pd 60.5° and C(96)–C(91)–P(2)–Pd 15.6°. Thus the phenyl group C(91)–C(96) is directed by its "edge" to fullerene and situated nearly *cis* to the Pd–P(2) bond; torsion angle C(91)–P(2)–Pd–X, where X is a midpoint of C(1)–C(2), is 7.7° whereas phenyl groups at P(1) are situated in *gauche* position to fullerene concerning Pd–P(1) bond with torsion angle C(73)–P(1)–Pd–X equal to 40.8°.

As a result of coordination with metal atom the fullerene core undergoes the substantial distortion which reflects in the increase of not only C(1)–C(2) length but also of all C–C bonds involving either C(1) or C(2) atom. Their lengths are within the interval 1.461–1.489 Å (in average 1.474 Å) whereas the average length for the rest of (5:6) bonds of the fullerene amounts 1.448 Å. Besides, 5- and 6-membered cycles involving C(1) and C(2) atoms are declined of planarity to the conformations of envelope and sofa, respectively. Atoms C(1) and C(2) go out of the planes of these cycles by 0.14 Å in average while the rest of 5- and 6-membered cycles of the fullerene core are planar better than 0.008Å.

Distortion of the fullerene ligand in complex **5** can be described by its "radius" in different directions. So, distances from the fullerene centre (determined without taking into account C(1), C(2) and symmetrically opposite C(59) and C(60) positions) to C(1) and C(2) are 3.694 and 3.683 Å, respectively, whereas the average dis-

tance for the rest of the fullerene carbons is 3.521 Å, interval being 3.488–3.549 Å.

Planes of two thiophene cycles form the dihedral angle equal to 75.5° (torsion angle C(62)–C(63)–C(69)–C(68) is 76.9°).

Intermolecular distances in the crystal correspond the usual van-der-Waals contacts.

Absolute configurations of two enantiomers of free tetraMe-BITIOP have been previously assigned by comparison of their CD curves with other biheteroaryl diphosphines [17]. This work establishes by anomalous dispersion effects in diffraction measurements on the crystal (see Section 3) that (–)tetraMe-BITIOP in complex **5** has *R* configuration that is in agreement with previous assignment.

3. Experimental

All the reactions were carried out using standard Schlenk technique under argon atmosphere. The solvents were dried, degassed, and distilled under argon prior to use. ^{31}P–{^{1}H} (at 162 MHz) and ^{1}H NMR spectra (at 400 MHz) were recorded on a Bruker AMX-400 FT instrument; the chemical shifts were measured relative to 1% H_3PO_4 and TMS, respectively. Electronic absorption spectra were registered on ⟨⟨UV-2201 SHIMADZU⟩⟩, circular dichroism spectra on JASCO-720. Electrochemical measurements were made using the voltammetric apparatus (AMEL, Italy) including a 551 potentiostat modulated by a 568 programmable function generator and coupled to a 731 digital integrator.

3.1. Preparation of (η^2-C_{60})Pd[(–)tetraMe-BITIOP] (5)

A mixture of C_{60} (72 mg, 1×10^{-4} mol), $Pd_2(dba)_3$ ·C_6H_6 (50 mg, 5×10^{-5} mol), and tetraMe-BITIOP (60 mg, 1.01×10^{-4} mol) was stirred in benzene (10 ml of C_6H_6 and 1 ml of C_6D_6) at room temperature. From time to time aliquots of the solution were examined by ^{31}P NMP spectroscopy. Colour of the solution gradually turned to green and dark-green precipitate formed. It was separated after 5 days, thoroughly washed with diethyl ether and dried in vacuo. The yield of complex **5** was 137 mg (97%). NMR ^{31}P–{^{1}H}(ODCB, δ, ppm): 22.87, singlet. To obtain single crystals suitable for X-ray study **5** was dissolved in 1,2-dichlorobenzene (ODCB), solution was filtered into Schlenk vessel, pentane and diethyl ether were added and solution was left for 40 days at room temperature isolated from daylight. Then crystals were separated from mother liquor, washed with dry ether and dried in argon stream to give 80.9 mg of **5** monosolvate (η^2-C_{60})Pd[(–)tetraMe-BITIOP]. Et_2O as dark green plate crystals. Analysis.

Found (%): C 79.95; H 2.72; S 4.08. $C_{96}H_{32}P_2PdS_2 \cdot$ $C_4H_{10}O$ calc. (%): C 80.51; H 2.84; S 4.30%. $^{31}P-\{^1H\}$ NMR (ODCB-C_6D_6, δ, ppm): singlet, 22.86. ^1H NMR (ODCB-C_6D_6, δ, ppm): singlet, 1.995 (6H, 2CH$_3$); singlet, 2.038 (6H, 2CH$_3$).

3.2. Preparation of (η^2-C_{70})Pd[(−)tetraMe-BITIOP] (6)

A mixture of C_{70} (92.4 mg, 1.1×10^{-4} mol), Pd$_2$ (dba)$_3 \cdot C_6H_6$ (50 mg, 5×10^{-5} mol), and tetraMe-BI-TIOP (61 mg, 1.03×10^{-4} mol) was stirred in benzene (7 ml of C_6H_6 and 0.56 ml of C_6D_6) at room temperature with monitoring by ^{31}P NMP spectroscopy. After 12 h only non-complexed starting diphosphine 3, δ(P) 21.11 ppm, could be revealed in solution. The product precipitated was filtered off, washed with diethyl ether, pentane and dried in vacuo to afford quantitative yield (154 mg) of complex 6 as dark-brown small crystals. Found (%): C 82.62; H 2.10; P 3.56. $C_{106}H_{32}P_2PdS_2$ calc. (%): C 82.78; H 2.10; P 4.03. $^{31}P-^1H$ NMR (ODCB-C_6D_6, δ, ppm): AB-system, $\delta(P_A) = 23.57$, $\delta(P_B) = 23.18$, $J(P_A,P_B) = 23.2$ Hz.

3.3. X-ray study of 5

$C_{96}H_{32}P_2S_2Pd \cdot C_4H_{10}O$ ($M = 1491.80$), orthorhombic, space group $P2_12_12_1$, $a = 14.5760(9)$ Å, $b = 17.087(1)$ Å, $c = 24.919(2)$ Å, $V = 6206.0(7)$ Å^3, $Z = 4$, $\mu = 0.480$ mm^{-1}, crystal size $0.40 \times 0.20 \times 0.05$ mm. Single-crystal X-ray diffraction experiment was carried out with a Bruker SMART 1000 CCD area detector, using graphite monochromated Mo Kα radiation ($\lambda = 0.71073$ Å, ω-scan with 0.3° step in ω and 20 s per frame exposure, $2\theta < 54°$) at 120 K. Reflection intensities were integrated using SAINT software [19] and semiempirical method SADABS [20] was applied for absorption correction ($T_{min}/T_{max} = 0.831/0.976$). A total of 42 773 reflections were measured, 13 595 ($R_{int} = 0.0847$) independent reflections were used in further calculations and refinement. The structure was solved by direct methods and refined by the full-matrix least-squares against F^2 in anisotropic (for non-hydrogen atoms) approximation. All hydrogen atoms were placed geometrically and included in the structure factor calculation in the riding motion approximation. Absolute configuration was determined from the value of Flack parameter ($x = 0.00(3)$) [21]. The final refinement was converged to $R_1 = 0.0633$ (for 9395 observed reflections with $I > 2\sigma(I)$) and $wR_2 = 0.1242$ (for all unique reflections); the number of the refined parameters is 955. All calculations were performed on an IBM PC/AT using the SHELXTL software [22]. Crystallographic data for the structural analysis of 5 has been deposited with the Cambridge Crystallographic Data Centre, CCDC No. 272221.

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