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4,5-Bis(diphenylthiophosphinoyl)-1,2,3-triazole, $L^{T\text{-}S2}$: a new varidentate ligand containing diphenylthiophosphinoyl moieties

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In memory of Luigi M. Venanzi

Abstract

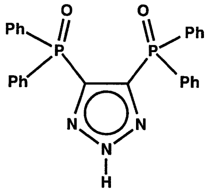
The novel ligand 4,5-bis(diphenylthiophosphinoyl)-1,2,3-triazole, $L^{T\text{-}S2}H$, has been synthesized, converted to the triethylamine salt, and to the palladium complexes $Pd[L^{T\text{-}S2}]_2$ and $Pd[L^{T\text{-}S2}][\eta^3\text{-methallyl}]$. Structures of $L^{T\text{-}S2}H$, of its 2-acetyl derivative, of $Pd[L^{T\text{-}S2}]_2$ and $Pd[L^{T\text{-}S2}][\eta^3\text{-methallyl}]$ were determined by X-ray crystallography. In the last two complexes the $L^{T\text{-}S2}$ ligand was N,S-bonded. © 2002 Elsevier Science B.V. All rights reserved.

Keywords: Crystal structures; Palladium complexes; Polydentate ligand complexes; Thiophosphinoyl complexes

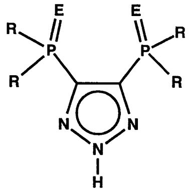
1. Introduction

The design of ligands to enable a desired function of a coordinated metal spans the whole range of inorganic and bioinorganic fields, either in the extraction of metal ions, in providing the proper steric and electronic environment for catalysis in chemical industry, or in the modeling of biologically important enzymatic processes. Yet, truly novel ligands with impressive properties do not appear very often.

We have reported recently a novel and versatile ligand, 4,5-bis(diphenylphosphinoyl)-1,2,3-triazole, $L^{T\text{-}O2}H$, which is thermally, oxidatively and hydrolytically very stable, and thus can be used under harsh conditions [1]. It combines the coordinating ability of the two –P(O)Ph_2 groups with the nitrogen donor atoms of the triazole ring, and is the prototype of a much broader family of ligands, having the general structure $L^{T\text{-}E2}H$,[1] where E can be O, S, Se, NR′ or NAr, while R may be a variety of alkyl or aryl groups.



$L^{T\text{-}O2}H$



$L^{T\text{-}E2}H$

There exist other ligands containing the –P(E)Ph_2 grouping, exemplified mainly by structures **A** or **B**, where E can be O, S, Se, or NR′ (R is most often Ph or Me). They usually coordinate as the deprotonated anions, although examples of complexes without deprotonation, as in the five-coordinate $Sb\{H_2C[P(O)Ph_2]_2\}Cl_3$, are also known [2].

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[1] In Ref. [1] the abbreviation L^T was used for the 4,5-bis(diphenylphosphinoyl)-1,2,3-triazole ligand. Since more ligands of this type are now being prepared, the abbreviation has been modified, to specify the phosphorus substituents. Thus, $L^{T\text{-}E2}H$ will denote the 4,5-bis(diphenylphosphino)-1,2,3-triazole, where each phosphorus contains a P=E bond. When the phosphorus E substituents are different, the abbreviation will be $L^{T\text{-}E,E'}$, as for instance $L^{T\text{-}O,S}$, $L^{T\text{-}O,Se}$, and so forth. The designation L^TH will be reserved for the parent compound, 4,5-bis(diphenylphosphino)-1,2,3-triazole.

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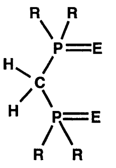
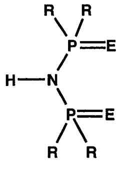


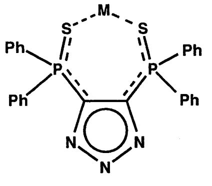
A



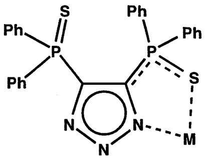
B

Most often the lithium salt of **A** was prepared (the dilithio salt had a complicated structure) [3,4] and then treated with various metal salts. These reactions were not always clean, and products often contained four-membered rings, involving bonding to the central carbon [5–8], and sometimes six-membered rings [9–13]. Similarly, the imidophosphonates **B** containing NH in place of the central CH_2 unit have also been prepared [14–19], and converted to chelates, such as $M[N(P(S)R_2)]_2$ for Mn(II), Fe(II), Ni(II) and Cu(II), where R was Ph or Me [20–25]. In general, ligands of type **A** and **B** do not possess good stability, nor do the complexes derived therefrom.

To overcome some of the limitations of previous thiophosphinoyl ligands, we have prepared the second member of the $[L^{T\text{-}E2}]H$ ligand class with E = S, $[L^{T\text{-}S2}]H$, and are reporting here some of the preliminary results. As can be seen from structures **C** and **D** there are two possible modes of chelation for the $[L^{T\text{-}S2}]^-$ ligand: a symmetric one (C_{2v}), through the two S termini, forming a seven-membered chelate ring, and an asymmetric one, through one S and the nearest N, resulting in a five-membered ring.



C



D

2. Experimental

All chemicals were of commercial reagent grade and were used as received. Elemental analyses were done by Microanalysis, Inc., Wilmington, DE. Infrared spectra were obtained as Nujol mulls or as KBr pellets with a Perkin–Elmer 1625 FTIR spectrophotometer, using 16 scans. Proton NMR spectra were obtained with a Nicolet NT360WB spectrometer. The compounds were studied under typical conditions of 16 K data points, a sweep width of 3000–4000 Hz, 90° pulse angles, and a recycle time of 4–5 s.

2.1. 4,5-Bis(diphenylthiophosphinoyl)acetylene

The synthesis described below is an improvement over the literature procedure [26]. A mixture of 102 g $Ph_2PC{\equiv}CPPh_2$ (0.26 mol) plus 32 g (1.0 mol) sulfur was heated with stirring in 500 ml THF. A vigorous reaction ensued and the solids dissolved forming a red solution, except for the excess sulfur. After 2 h the liquid layer was decanted, the sulfur residue was washed with a small amount of acetone, and the combined washings were concentrated down to a thick slurry. Filtration and washing with acetone yielded a pinkish solid (102 g, 86.5%). Further evaporation of the filtrates yielded a second crop of 16 g. Both crops were slightly contaminated with sulfur, but this did not interfere with the next step.

2.2. 4,5-Bis(diphenylthiophosphinoyl)-1,2,3-triazole, $[L^{T\text{-}S2}]H$

The whole crude batch from the above reaction was stirred with 19.2 g NaN_3 (small excess) in 500 ml DMF, and after the moderately exothermic reaction subsided, the red solution was decanted from a small amount of undissolved sulfur into 3 l of water containing 200 ml of concentrated HCl. The precipitated creamy solid was filtered off, washed with several portions of water, plus a few small portions of acetone, and was dried. The yield was 97.4 g (76.0% over-all for the two steps). After recrystallization from DMF the compound melted at 300 °C. Calc. $C_{26}H_{21}N_3P_2S_2$: C, 62.3; H, 4.19; N, 8.38. Found: C, 63.1; H, 4.30; N, 8.16%.

2.3. 2-Acetyl-4,5-bis(diphenylthiophosphinoyl)-1,2,3-triazole

A small sample of $[L^{T\text{-}S2}]H$ was boiled for 5 min in excess acetic anhydride, and the solution was evaporated. The residue was recrystallized from xylene; m.p. 173–175 °C. IR (cm^{-1}): ν(CO) 1719. ^1H NMR (ppm): 2.54 (s, 3H, Me), 7.38 (m, 8H, Ph), 7.48 (m, 4H, Ph), 7.72 (m, 8H, Ph). ^{31}P NMR (ppm): singlet at 31.2. Calc. $C_{28}H_{23}N_3OP_2S_2$: C, 61.9; H, 4.24; N, 7.73. Found: C, 62.2; H, 4.47; N, 7.58%.

2.4. Triethylamonium salt of 4,5-bis(diphenylthiophosphinoyl)-1,2,3-triazole, $[L^{T\text{-}S2}][HNEt_3]$

Adding an excess of triethylamine to a stirred slurry of $[L^{T\text{-}S2}]H$ in methanol resulted in a clear solution, which was evaporated, and the residue was slurried with acetone, in which the salt is poorly soluble, and filtered producing, after drying, the pinkish salt in 94% yield. It was recrystallized from DMF. M.p. sinters from 188 °C, dec. 196–198 °C. ^1H NMR (ppm): 1.02 (t, 12H, Me), 3.00 (q, 8H, CH_2), 7.16 (m, 8H, Ph), 7.27 (m, 4H, Ph), 7.63 (m, 8H, Ph). ^{31}P NMR (ppm): 32.6. Calc. for $C_{32}H_{36}N_4P_2S_2$: C, 63.8; H, 5.98; N, 9.30. Found: C, 64.0; H, 6.15, N, 9.13%.

2.5. Pd[$L^{T\text{-}S2}$][η^3-methallyl]

An equimolar mixture of [$L^{T\text{-}S2}$][$HNEt_3$] and [Pd(η^3-methallyl)Cl]$_2$ was stirred in methylene chloride at room temperature for 1 h. Water was added, the layers were separated, the pale yellowish organic layer was filtered through alumina, and evaporated. The residue was recrystallized from toluene. The compound starts yellowing from 188 °C, and decompose to a red froth at 210–212 °C. ^1H NMR (ppm): 2.01 (S, 3H, Me), 2.67 (s, 1H, *anti*), 3.44 (s, 1H, *anti*), 3.65 (d, 1H, *syn*), 4.60 (d, 1H, *syn*), 7.24 (m, 8H, phenyl), 7.40 (m, 4H, phenyl), 7.53 (m, 4H, phenyl), 7.73 (m, 4H, phenyl). ^{31}P NMR (ppm): 46.8, 28.8. Calc. for $C_{30}H_{27}N_3P_2PdS_2$: C, 54.5; H, 4.08; N, 6.35. Found: C, 54.4; H, 4.19; N, 6.25%.

2.6. Pd[$L^{T\text{-}S2}$]$_2$·2py

To a stirred methanolic solution of 2 equiv. of [$L^{T\text{-}S2}$][$HNEt_3$] was added dropwise 1 equiv. of a methanolic solution of Na_2PdCl_4, and after 1 h the solution was diluted with more water. The precipitated solid was filtered off, washed with three portions of water, and dried. It was recrystallized from pyridine, forming a yellow bissolvate. After drying at 150 °C/0.1 Torr to remove pyridine, it did not melt up to 310 °C. Calc. for $C_{52}H_{40}N_6P_4PdS_4$: C, 56.4; H, 3.62; N, 7.59. Found: C, 56.6; H, 3.81; N, 7.56%.

2.7. Crystallography

Crystallographic data are collected in Table 1. All specimens were mounted in Paratone N mineral oil and placed in the cold stream. Corrections for absorption were applied using SADABS. Systematic absences uniquely determined the space group for **3**, and for the others the centrosymmetric option was chosen by the results of refinement. All structures were solved by direct methods. All non-hydrogen atoms were refined with anisotropic thermal parameters, and hydrogen atoms were treated as idealized contributions. For **1** the asymmetric unit is a half molecule residing on a two-fold axis. The Pd complex in **4** is accompanied by two molecules of pyridine. All software is contained in the SHELXTL (5.1) library (G.M. Sheldrick, Bruker AXS, Madison, WI, USA).

3. Results and discussion

The sodium salts of [$L^{T\text{-}S2}$]H and Na[$L^{T\text{-}S2}$] were obtained directly in one step by the reaction of sodium azide with bis(diphenylthiophosphinoyl)acetylene in DMF [Eq. (1)], and this solution could be used for the preparation of metal complexes, or for obtaining the free acid, [$L^{T\text{-}S2}$]H, through acidification with hydrochloric acid. The addition of azide anion to bis(diphenylthiophosphinoyl)acetylene required DMF as solvent, rather than methanol, as was the case in the

Table 1
Crystal data and structure refinement parameters for **1**–**4**

	1	**2**	**3**	**4**
Empirical formula	$C_{26}H_{21}N_3P_2S_2$	$C_{28}H_{23}N_3OP_2S_2$	$C_{30}H_{27}N_3P_2PdS_2$	$C_{62}H_{50}N_8P_4PdS_4$
Formula weight	501.52	543.55	662.01	1265.62
Crystal system	monoclinic	triclinic	monoclinic	triclinic
Space group	$C2/c$	$P\bar{1}$	$P2_1/n$	$P\bar{1}$
Unit cell dimensions				
a (Å)	15.9311(3)	9.0061(2)	9.892(2)	9.2097(2)
b (Å)	7.2861(2)	9.6793(2)	25.041(9)	10.9655(2)
c (Å)	23.1967(2)	15.6127(3)	12.336(4)	28.3762(6)
α (°)		88.4379(9)		95.0766(5)
β (°)	105.5155(8)	79.5000(9)	98.57(2)	94.1875(2)
γ (°)		81.2939(6)		90.5218(3)
V (Å^3)	2553.20(8)	1322.78(3)	3021.7(13)	2846.46(18)
Z, Z'	4, 0.5	2, 1	4, 1	2, 1
Crystal color	colorless	colorless	pale yellow	yellow
D_{calc} (g cm^{-3})	1.305	1.365	1.455	1.477
μ (Mo Kα) (cm$^{-1)}$	3.53	3.50	8.82	6.34
Temperature (K)	173	173	235	173
Radiation	Mo Kα ($\lambda = 0.71073$ Å)			
Reflections collected	5528	7459	5703	18 100
Independent reflections	2789	5139	4611	10 873
$R(F)$ (%)	4.23	7.67	4.26	6.55
$R(wF)$ (%)	15.06	20.70	9.98	16.04
$\Delta(\rho)$ (e Å^{-3})	0.519	1.073	0.901	1.424

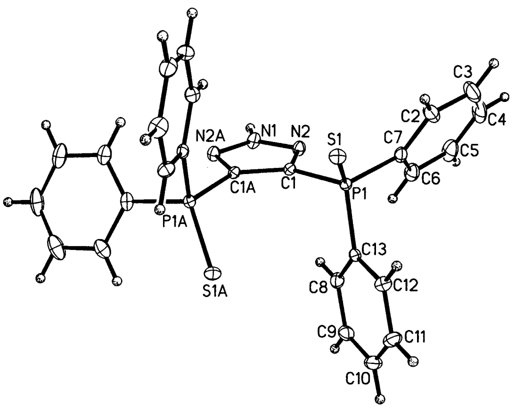


Fig. 1. ORTEP plot of the structure of $[L^{T\text{-}S2}]H$, **1**. Selected bond distances (Å) and angles (°): P(1)–S(1) 1.9633(7); P(1)–C(1) 1.8263(17); C(1)–N(2) 1.254(2); N(1)–N(2) 1.337(2); C(13)–P(1)–C(7) 105.75(8); C(13)–P(1)–S(1) 114.30(6); C(7)–P(1)–S(1) 114.00(7); C(13)–P(1)–C(1) 106.43(8); C(1)–P(1)–C(7).

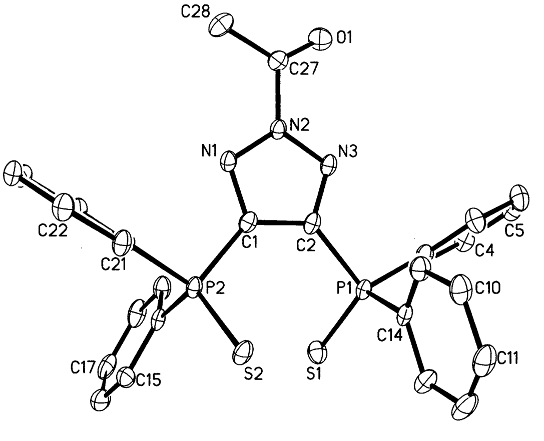
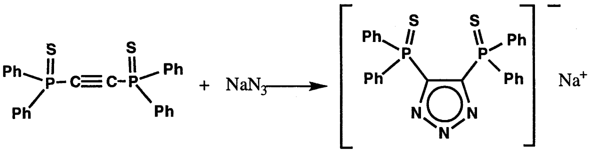


Fig. 2. ORTEP plot of the structure of $[L^{T\text{-}S2}][2\text{-}Ac]$, **2**. Selected bond distances (Å) and angles (°): P(1)–C(8) 1.821(4); P(1)–S(1) 1.9380(14); P(1)–C(14) 1.812(3); P(1)–C(2) 1.830(3); C(14)–P(1)–C(8) 105.13(16); C(14)–P(1)–C(2) 104.24(14); C(8)–P(1)–C(2) 104.51(17); C(14)–P(1)–S(1) 116.27(14); C(2)–P(1)–S(1) 111.41(12).

synthesis of $Na[L^{T\text{-}O2}]$, and longer reaction time. This reaction was only mildly exothermic, commensurate with the stronger electron-withdrawing power of a $-P(O)Ph_2$ group as compared with $-P(S)Ph_2$.



(1)

$[L^{T\text{-}S2}]H$ (**1**) is thermally quite stable, melting sharply at 300 °C. Its structure was confirmed by X-ray crystallography (Fig. 1). The P=S distance was 1.9633(7) Å, which is significantly longer compared to the P=S distance of 1.881(4) Å in $HN[P(S)Ph_2][P(O)Ph_2]$ [28].

The 4,5-bis(diphenylthiophosphinoyl)-1,2,3-triazole is not as acidic as the oxo analogue, $L^{T\text{-}O2}H$, but is still acidic enough to form the stable, halocarbon-soluble triethylamine salt, $[L^{T\text{-}S2}][Et_3NH]$, which can be used for reactions of the $[L^{T\text{-}S2}]^-$ ligand in organic solvents. The NMR spectrum of $[L^{T\text{-}S2}][HNEt_3]$ was consistent with a symmetrical structure of the anion, and the ^{31}P was at 32.6 ppm, and thus in the typical P=S range.

Acetylation of $[L^{T\text{-}S2}]H$ with acetic anhydride occurred exclusively at 2-N, and the C_{2v} symmetry of acylated product, $[L^{T\text{-}S2}][2\text{-}Ac]$ (**2**) was confirmed by NMR, which exhibited a single ^{31}P signal at 31.2 ppm, as well as by X-ray crystallography (Fig. 2). The P=S distances were 1.9380(14) and 1.9415(12), respectively, the slightly longer value found in the P=S '*trans*' to the acyl oxygen.

We also wanted to check whether the coordination of the $[L^{T\text{-}S2}]^-$ ligand with Pd(II) will lead to the five-membered S,N chelate ring, or to the seven-membered S,S-chelate ring. In the case of $[L^{T\text{-}O2}]H$, several examples of both types of complexes have been synthesized, and structurally characterized [1]. The reaction of the salt $[L^{T\text{-}S2}][Et_3NH]$ in chloroform with $[Pd(\eta^3\text{-methallyl})]_2$ produced the heteroleptic complex $Pd[L^{T\text{-}S2}][\eta^3\text{-methallyl}]$ (**3**). The fact that its structure was of type **D** was suggested by the presence of two very different ^{31}P signals at 46.8 and 28.8 ppm, assigned to the coordinated and uncoordinated P=S, respectively, since the latter value is much closer to the one found in $[L^{T\text{-}S2}][Et_3NH]$. Asymmetry of the molecule also caused each of the methallyl *syn* and *anti* protons to be unique. The structure was confirmed by X-ray crystallography (Fig. 3).

The molecule contains two different P=S bonds: the uncoordinated P=S bond is 1.9519(17) Å, while the coordinated one is distinctly longer, at 2.0084(18) Å. Similarly, the distances from P to the triazole carbon were different: for the coordinated $C\text{–}P(S)Ph_2$ group,

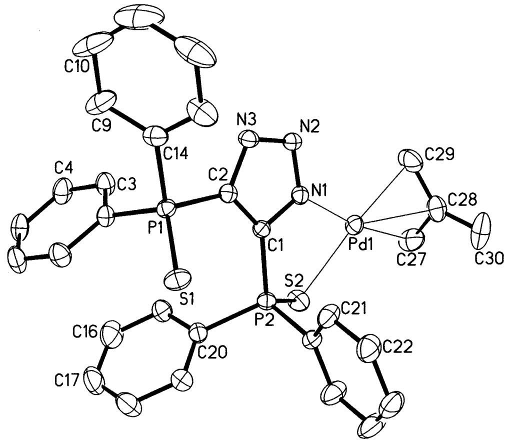


Fig. 3. ORTEP plot of the structure of $Pd[L^{T\text{-}S2}][\eta^3\text{-methallyl}]$, **3**. Selected bond distances (Å) and angles (°): Pd(1)–N(1) 2.070(4); Pd(1)–C(27) 2.104(5); Pd(1)–C(29) 2.124(5); Pd(1)–C(28) 2.132(6); Pd(1)–S(2) 2.3796(14); S(1)–P(1) 1.9519(17); S(2)–P(2) 2.0084(18); P(1)–C(2) 1.810(4); P(2)–C(1) 1.789(4).

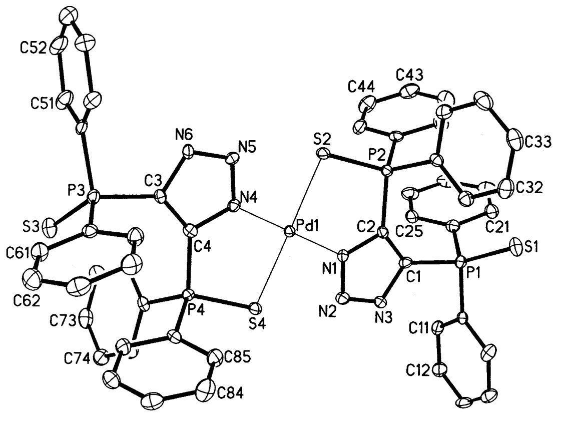
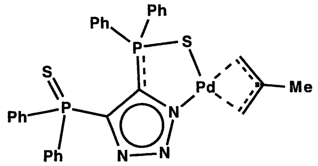


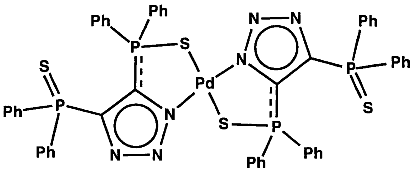
Fig. 4. ORTEP plot of the structure of Pd[$L^{T\text{-}S2}$]$_2$·2py, **4**. The two uncoordinated molecules of pyridine have been omitted. Selected bond distances (Å) and angles (°): Pd(1)–N(4) 1.997(4); Pd(1)–N(1) 2.003(4); Pd(1)–S(4) 2.3159(13); Pd(1)–S(2) 2.3228(13); S(1)–P(1) 1.9476(19); S(2)–P(2) 2.0187(18); S(3)–P(3) 1.949(2); S(4)–P(4) 2.0306(17); N(4)–Pd(1)–N(1) 179.63(18); N(4)–Pd(1)–S(4) 88.83(12); N(l)–Pd(l)–S(4) 90.94(12); N(4)–Pd(1)–S(2) 91.45(12); N(1)–Pd(1)–S(2) 88.79(12); S(4)–Pd(1)–S(2) 179.53(5); P(2)–S(2)–Ps(1) 100,26(6); P(4)–S(4)–Pd(1) 101.06(6).

the P–C bond was 1.789(4) Å, while for the uncoordinated C–P(S)Ph$_2$ group the P–C bond was 1.8109(4) Å. Thus, coordination of sulfur from the C–P(S)Ph$_2$ group leads to a lengthening of the P=S bond, and to shortening of the P–C bond. The Pd–S distance was 2.3796(14) Å, which is somewhat longer than the values of 2.3478(8) and 2.3333(8) Å found in the complex Pd[N(P(S)Ph$_2$)$_2$]$_2$ [27].



Pd[$L^{T\text{-}S2}$][η^3-methallyl]

The homoleptic complex Pd[$L^{T\text{-}S2}$]$_2$ (**4**) was also synthesized by the reaction of the triethylamine salt of $L^{T\text{-}S2}$H with Na$_2$PdCl$_4$ in methanol. Considering that the chelation of $L^{T\text{-}S2}$ in **3** was of type **D**, involving N,S-bonding, one could have expected the presence of *cis*- and *trans*-isomers. However, X-ray crystallography showed that the complex was the *trans*-isomer (Fig. 4). The reason why no S,S-bonded structure was obtained is not clear, since other S$_4$ tetracoordinated Pd complexes, for instance Pd[N(P(S)Ph$_2$)$_2$]$_2$, have been reported. In complex **4** the coordinated P=S bond lengths were 2.0187(18) and 2.0306(17) Å, the uncoordinated ones were 1.9476(19) and 1.9492(2) Å, the P–C bonds to the triazole carbon were 1.814(5) and 1.819(5) Å for the free –P(S)Ph$_2$ groups, and for the coordinated ones they were 1.794(5) and 1.790(5) Å. Again, coordination to Pd resulted in lengthening of the P=S bonds, and shortening of the P–C bonds. By comparison, in complex Pd[N(P(S)Ph$_2$)$_2$]$_2$ the P=S bond lengths were very similar, 2.0255(13) and 2.258(11) Å, to the coordinated P=S of compound **4**. The Pd–S bond lengths were 2.3478(8) and 2.3333(8) Å in the complex Pd[N(P(S)Ph$_2$)$_2$]$_2$, and 2.3159(13) and 2.3228(13) Å in complex **4**.



Pd[$L^{T\text{-}S2}$]$_2$

We have also found that the anion [$L^{T\text{-}S2}$]$^-$ extracts readily transition metals from aqueous solutions into chloroform or into dichloromethane. These and other reactions of the [$L^{T\text{-}S2}$]$^-$ ligand are being investigated, and will be reported in due course.

In summary, the second member of the $L^{T\text{-}E2}$H family, $L^{T\text{-}S2}$H, has been synthesized, and found to form homo- and heteroleptic S,N-bonded complexes with Pd(II), all of which were structurally characterized.

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