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Note

Displacement of triphenylphosphine from $Cu(PPh_3)_2NO_3$ and $Co(PPh_3)_2Cl_2$ by a diselenoimidodiphosphinato ligand. X-ray crystal structure of $(PPh_3)Cu[Ph_2P(Se)NP(Se)Ph_2]$ and $Co[Ph_2P(Se)NP(Se)Ph_2]_2$ containing the novel $CuSe_2P_2N$ and $CoSe_2P_2N$ inorganic metallocycles

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Abstract

The reaction of $Cu(PPh_3)_2NO_3$ with $K[Ph_2P(Se)–N–P(Se)Ph_2]$ produced a tricoordinate copper(I) complex, $Cu(PPh_3)[Ph_2P(Se)–N–P(Se)Ph_2]$, containing the novel inorganic (carbon-free) $CuSe_2P_2N$ metallocycle. The same diselenium reagent gave with $Co(PPh_3)_2Cl_2$ a spirobicyclic compound $Co[Ph_2P(Se)–N–P(Se)Ph_2]_2$, containing the novel inorganic $CoSe_2P_2N$ metallocycle. The crystal structure of the two compounds was investigated by X-ray diffraction. In both compounds the inorganic metallocycles are nonplanar and display strongly distorted twisted boat conformations. © 1999 Elsevier Science S.A. All rights reserved.

Keywords: Crystal structures; Copper complexes; Diselenoimidodiphosphinato complexes

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1. Introduction

Dichalcogenoimidodiphosphinato anions $[R_2P(E)–N–P(E)R_2]^-$ (E = O, S, Se) are versatile ligands, with a strong tendency to form inorganic (carbon-free) chelate rings [1–4]. For many years after their introduction by Schmidpeter et al. [5–7], dichalcogenoimidodisphosphinates received only scant attention, but recently the interest in their complexes has been renewed and dioxo [8–15], dithio [16–27], and even mixed oxo-thio [28–30] derivatives have been investigated. Although known for several years [31], diselenoimidodiphosphinate has been only recently used as a ligand [32–37].

All dichalcogenoimidodisphosphinates **1** tend to form six-membered inorganic (carbon-free) chelate rings **2** and seldom act as bridging ligands; only the dithioimidodiphosphinates are known to form larger bimetallic rings **3** (R = Ph, M = Au [38], TePh [39], $TeC_6H_4OCH_3$ [40]) and only one example is known when a supramolecular helical polymer **4** (R = Ph, M = $SnMe_3$) is formed via secondary M···S interactions [41].

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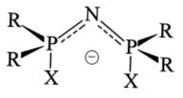
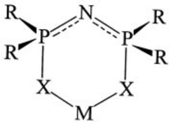


1



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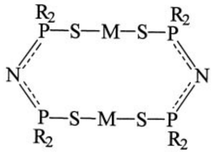

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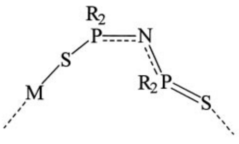


4

The discovery of heterogeometrism [42] in some dichalcogenoimidodiphosphinates, i.e. isomeric square pyramidal and square planar tin(II) diselenoimidodiphosphinates [35], illustrates another feature of interest in the structural chemistry of these complexes: the possibility of (unpredictable) major structural changes (even in the coordination geometry) produced by minor variations in the composition of the ligand (e.g. differing peripheral groups) within a series of compounds containing the same metal as coordination centre. With differing donor properties of the atoms (oxygen, sulfur or selenium) at the two ends of the diphosphazene fragment, the comparison of the structures and properties of various dichalcogenoimidodiphosphinato ligands becomes interesting.

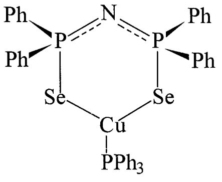
Since few diselenoimidodiphosphinato metal chelates have been investigated so far, we found it worthwhile to extend this class of compounds. We now report copper(I) and cobalt(II) diselenoimidodiphosphinates, prepared and investigated for comparison with the corresponding thio analogues.

2. Results and discussion

It appears that the diselenoimidodiphosphinate is a strong complexing reagent, able to displace coordinated triphenylphosphine, when the geometric and chemical requirements of chelate ring formation allow it. Thus, bis(triphenylphosphine)copper(I) nitrate reacts with potassium tetraphenyldiselenoimidodiphosphinate, to give (triphenylphosphine) (tetraphenyldiselenoimidodiphosphinato)copper(I) as a result of combined metathetical anion exchange and displacement of a triphenylphosphine molecule (Eq. (1)):

$$\mathrm{Cu(PPh_3)_2NO_3 + K[Ph_2P(Se){-}N{-}P(Se)Ph_2]}$$
$$\mathrm{\rightarrow Cu(PPh_3)[Ph_2P(Se){-}N{-}P(Se)Ph_2] + PPh_3 + KNO_3} \quad (1)$$

The chelate ring compound formed in reaction (1) contains a new inorganic (carbon-free) $CuSe_2P_2N$ chelate ring **5** in which copper(I) is three-coordinate.



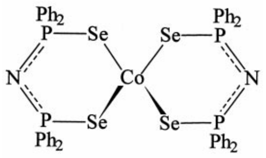
5

Complex **5** is a colourless crystalline solid (m.p. 223°C), moderately soluble in common organic solvents.

In the reaction of bis(triphenylphosphine)cobalt(II) dichloride with potassium tetraphenyldiselenoimidodiphosphinate, the halogens were metathetically exchanged for two diselenoimidodiphosphinato ligands. The chelate ring closure with two ligands, resulting in a four-coordinate complex, requires two additional coordination sites, and thus both triphenylphosphine molecules are liberated (Eq. (2)). No five- or six-coordinate complex containing coordinated triphenylphosphine was obtained in this reaction.

$$\mathrm{Co(PPh_3)_2Cl_2 + 2K[Ph_2P(Se){-}N{-}P(Se)Ph_2]}$$
$$\mathrm{\rightarrow Co[Ph_2P(Se){-}N{-}P(Se)Ph_2]_2 + 2PPh_3 + 2KCl} \quad (2)$$

The resulting compound is a bis(chelate) complex **6**, which can also be described as a spirobicyclic compound consisting of two inorganic (carbon-free) $CoSe_2P_2N$ rings, sharing a tetrahedral cobalt(II) atom as a coordination centre.



6

Complex **6** is a colourless crystalline solid, moderately soluble in common organic solvents and can be recrystallized from methylene dichloride.

Partial or complete displacement of triphenylphosphine by tetraphenyldiselenoimidodiphosphinate has also been observed in reactions with $Re(PPh_3)_2OCl_3$ [32], $Re(PPh_3)_2NCl$ and $Re(PPh_3)_2(NMe)Cl_3$ [33], but not with $[M(PMe_2Ph)Cl_2$ (M = Pd, Pt) [33]. Similar reactions also occur with tetraphenyldithioimidodiphosphinate [33]. Obviously, in all these cases the preferred coordination geometry of the central metal atom dictates the reaction path and the composition of the final product.

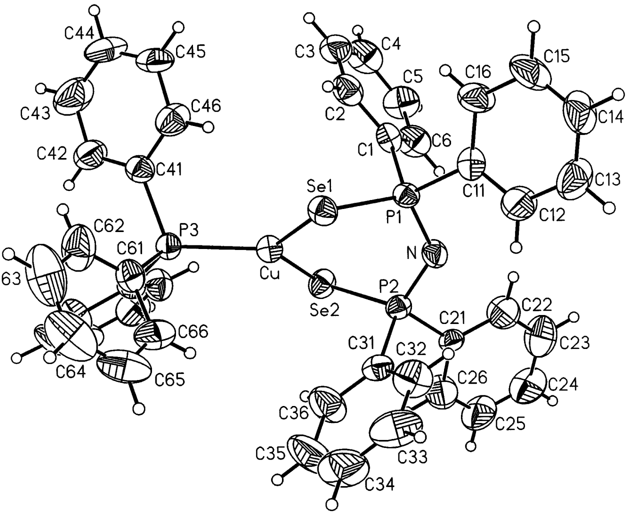


Fig. 1. An ORTEP diagram of the molecular structure of **5**.

The molecular structures of both **5** and **6** were determined by single-crystal X-ray diffraction. An ORTEP diagram of the molecular structure of the copper compound **5** is shown in Fig. 1 with the atom numbering scheme and important interatomic distances and bond angles are listed in Table 1.

Table 1
Selected bond lengths (Å) and angles (°) in compounds **5** and **6**

Compound **5**		Compound **6**	
Bond lengths (Å)			
Cu–Se(1)	2.3918(14)	Co–Se(1)	2.4746(10)
Cu–Se(2)	2.376(2)	Co–Se(2)	2.3622(10)
Cu–P(3)	2.222(2)	Co–Se(3)	2.4394(8)
		Co–Se(4)	2.3835(11)
P(1)–Se(1)	2.166(2)	P(1)–Se(1)	2.0992(14)
P(2)–Se(2)	2.163(2)	P(2)–Se(2)	2.1913(13)
		P(3)–Se(3)	2.2056(14)
		P(4)–Se(4)	2.1640(13)
P(1)–N	1.600(7)	P(1)–N(1)	1.577(7)
P(2)–N	1.592(7)	P(2)–N(1)	1.609(4)
		P(3)–N(2)	1.532(4)
		P(4)–N(2)	1.575(4)
Bond angles (°)			
Cu–Se(1)–P(1)	92.16(7)	Co–Se(1)–P(1)	97.91(4)
Cu–Se(2)–P(2)	90.22(7)	Co–Se(2)–P(2)	98.21(4)
		Co–Se(3)–P(3)	97.00(4)
		Co–Se(4)–P(4)	103.33(4)
Se(1)–Cu–Se(2)	121.48(6)	Se(1)–Co–Se(2)	113.38(3)
P(3)–Cu–Se(1)	115.62(7)	Se(1)–Co–Se(3)	111.55(3)
P(3)–Cu–Se(3)	122.90(7)	Se(1)–Co–Se(4)	106.18(3)
		Se(2)–Co–Se(3)	106.91(3)
		Se(2)–Co–Se(4)	104.72(4)
		Se(3)–Co–Se(4)	114.06(3)
N–P(1)–Se(1)	116.9(3)	N(1)–P(1)–Se(1)	115.0(2)
N–P(2)–Se(2)	118.1(3)	N(1)–P(2)–Se(2)	119.7(2)
		N(2)–P(3)–Se(3)	117.9(2)
		N(2)–P(4)–Se(4)	120.7(2)
P(1)–N–P(2)	127.0(4)	P(1)–N–P(2)	130.3(3)
		P(3)–N–P(4)	133.1(3)

Table 2
Deviations (Å) of the ring atoms from the best plane in the $CuSe_2P_2N$ ring

N	0.046(8)
Cu	0.009(2)
P(1)	−0.694(3)
P(2)	0.747(3)
Se(1)	0.209(2)
Se(2)	−0.242(2)

The six-membered $CuSe_2P_2N$ ring is nonplanar (twisted boat conformation). The deviations of the ring atoms from the weighted least-squares best plane are listed in Table 2. The three-coordinate copper(I) atom displays a distorted trigonal planar geometry with P(3)–Cu–Se(1) = 115.62(7)°, P(3)–Cu–Se(2) = 122.90(7)° and Se(1)–Cu–Se(2) = 121.48(6)°. This distortion is somewhat similar to that observed in the copper(I) thio analogue $Cu(PPh_3)_2[Ph_2P(S)–N–P(S)Ph_2]$ with P–Cu–S = 126.1(1) and 112.2(1)° and S–Cu–S = 121.5(1)°.

The ring is basically symmetric. Thus, the selenium–copper bonds are practically equal in the six-membered ring [Cu–Se 2.3918(14) and 2.376(2) Å] and so are the phosphorus–selenium bonds [P(1)–Se(1) = 2.166(2) Å and P(2)–Se(2) = 2.163(2) Å].

The P–N bonds are also equal within the e.s.d. [P(1)–N = 1.600(7) and P(2)–N = 1.592(7) Å] and these interatomic distances suggest some double-bond character as in other inorganic (carbon-free) metallocycles derived from dichalcogenoimidodiphosphinates.

An ORTEP diagram of the molecular structure of the cobalt(II) compound **6** is shown in Fig. 2, with the atom numbering scheme. Significant interatomic distances and bond angles are listed in Table 1.

The six-membered $CoSe_2P_2N$ rings are nonplanar and severely distorted, displaying twisted boat confor-

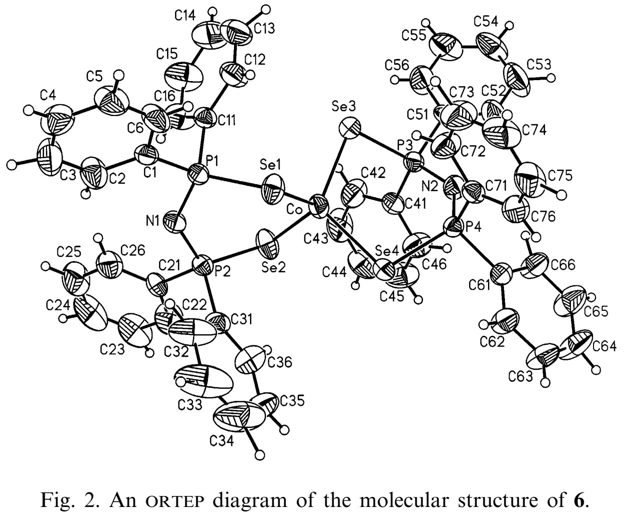
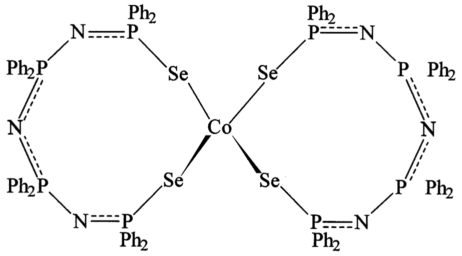


Fig. 2. An ORTEP diagram of the molecular structure of **6**.

mation imposed by the non-parallel orientation of the two P–Se bonds in each ligand moiety. The cobalt–selenium bond lengths differ slightly and each ring contains two unequal (one longer and one shorter) cobalt–selenium bonds [Co–Se(1) 2.475(1) and Co–Se(3) 2.439(1) Å ('long') with Co–Se(2) 2.362(1) and Co–Se(4) 2.383(1) Å ('short')]. The cobalt(II) centre is tetrahedrally coordinated with Se–Co–Se intra-ring (endocyclic) bond angles slightly larger [e.g. (exocyclic) bond angles [Se(1)–Co–Se(2) 113.38(3)° and Se(3)–Co–Se(4) 114.06(3)°]. The opposite is observed in the methyl thio analogue $Co[Me_2P(S)–N–P(S)Me_2]_2$ with exocyclic S–Co–S bond angles (111.7(1) and 114.3(1)°) larger than endocyclic S–Co–S bond angles (109.6(1) and 110.3(1)°) [16].

The structure of the cobalt(II) compound **6** can be compared with that of a related bis(diselenodiimidotriphosphinate) $Co[Ph_2P(Se)–N–P(Ph_2)–N–P(Se)Ph_2]_2$ (**7**) reported recently, which contains two eight-membered $CoSe_2P_3N_2$ inorganic rings [43].



7

In **7** the coordination geometry of cobalt(II) is also distorted tetrahedral. In the ten-membered rings the Se–Co–Se bond angles are larger (113.6(1) and 113.7(1)°) than the exocyclic bond angles (106.0(1) and 110.2(1)°). The Co–Se and P–N bond lengths in **6** and **7** are comparable.

3. Experimental

3.1. Preparation of $Cu(PPh_3)[Ph_2P(Se)–N–P(Se)Ph_2]$ (5)

A solution of 0.300 g (0.516 mmol) $K[Ph_2P(Se)–N–P(Se)Ph_2]$ dissolved in 50 cm^3 methanol was mixed with stirring with a solution of 0.355 g (0.516 mmol) $Cu(PPh_3)_2NO_3$ dissolved in 25 cm^3 chloroform. After stirring for 30 min the precipitate was filtered off and the filtrate was concentrated to give a solid. The product was recrystallized from CH_2Cl_2.

Yield: 86% $Cu(PPh_3)[Ph_2P(Se)–N–P(Se)Ph_2]$; m.p. 223°C.

Anal. Found: C, 57.9; H, 4.0; N, 1.6. Calc. for $C_{42}H_{35}CuNP_3Se_2$: C, 58.1; H, 4.1; N, 1.6%.

^{31}P NMR ($CDCl_3$, rel. to 85% H_3PO_4): δ 26.14 with two P–Se satelites $^1J_{P-Se} = 576$ Hz.

IR (KBr pellets, cm^{-1}): 3050 (νCH), 1170 ($\nu_{as}P_2N$), 1101, 757 (ν_sP_2N), 693, 550 (νPSe), 519, 505. Mass spec. (FAB$^+$): m/z 869 [M^+], 670, 460, 341.

3.2. Preparation of $Co[Ph_2P(Se)–N–P(Se)Ph_2]_2$ (6)

A solution of 0.300 g (0.516 mmol) $K[Ph_2P(Se)–N–P(Se)Ph_2]$ dissolved in 50 cm^3 methanol was mixed with stirring with a solution of 0.169 g (0.258 mmol) $Co(PPh_3)_2Cl_2$ dissolved in 25 cm^3 chloroform. After stirring for 30 min the precipitate was filtered off and

Table 3
Crystal data and structure determination of compounds **5** and **6**

	5	**6**
Crystal data		
Empirical formula	$C_{42}H_{35}CuNP_3Se_2$	$C_{48}H_{40}CoN_2P_4Se_4$
Formula weight	868.08	1143.47
Crystal dimensions (mm)	0.50 × 0.20 × 0.10	0.65 × 0.40 × 0.20
Crystal system	triclinic	triclinic
Space group	$P\bar{1}$	$P\bar{1}$
Lattice parameters		
a (Å)	9.180(2)	13.743(3)
b (Å)	12.056(2)	13.830(3)
c (Å)	18.642(4)	14.329(2)
α (°)	79.24(3)	82.623(14)
β (°)	79.28(3)	66.25(2)
γ (°)	73.34(3)	66.98(2)
Cell volume (Å^3)	1922.5(7)	2292.9(8)
Z	2	1
D_{calc} (mg m^{-3})	1.500	1.656
F(000)	872	1130
Absorption coefficient (mm^{-1})	2.617	3.725
Intensity measurements		
Scan type	ω–2θ	ω–2θ
Index range	$-11<h<10$, $-14<k<14$, $0<l<22$	$-15<h<13$, $-16<k<16$, $0<l<17$
No. reflections collected	6433	8153
No. independent reflections	6237 (R_{int} 0.1070)	7848 (R_{int} 0.0203)
Corrections	none	none
Structure solution and refinement		
Structure solution	heavy atom (SHELX-86)	
Refinement	full-matrix least-squares on F^2	
No. of observations used [$I>2\sigma(I)$]	6237	7848
No. of variables	477	693
Final R indices	$R_1 = 0.0558$ $wR_2 = 0.1634$	$R_1 = 0.0345$ $wR_2 = 0.0887$
R all data	$R_1 = 0.1042$ $wR_2 = 0.1897$	$R_1 = 0.0644$ $wR_2 = 0.0992$
Max. peak in final difference map (e Å^{-3})	0.674	0.573
Min. peak in final difference map (e Å^{-3})	−1.318	−0.802

the filtrate was concentrated to give a colourless solid, which was recrystallized from CH_2Cl_2.

Yield: 81% $Co[Ph_2P(Se)-N-P(Se)Ph_2]_2$.

Anal. Found: C, 54.0; H, 3.7; N, 2.6. Calc. for $C_{48}H_{40}CoN_2P_4Se_4$: C, 53.7; H, 3.7; N, 2.5%.

^{31}P NMR ($CDCl_3$, rel. to 85% H_3PO_4): δ 27.7 (singlet). IR (KBr pellets, cm^{-1}): 3067 (νCH), 1479, 1436,1199 ($\nu_{as}P_2N$), 1106, 746 (ν_sP_2N), 692, 545 (νPSe). Mass spec. (FAB^+): 1143 [M^+], 542, 384, 307.

4. Crystallographic data

The single-crystal diffraction studies were performed on a KUMA KM4 four-circle κ-axis diffractometer. Details about cell dimensions, data collection, structure solving and refinement are shown in Table 3. The crystal stability during the data collection was checked by measuring three standard reflections (020, 002 and 210) after every 300 measurements; no significant change of their intensities was detected. The structures were solved by the heavy-atom method, using the SHELX-86 program. All atoms, except the hydrogens, were refined anisotropically by the full-matrix least-squares procedure (program SHELX-93). The hydrogen atoms were positioned from difference Fourier maps and refined isotropically. Extinction and absorption corrections were not applied because their application did not reduce the *R* values or e.s.d.'s.

Acknowledgements

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