

*Dedicated to Professor Eugen Segal  
on the occasion of his 80<sup>th</sup> anniversary*

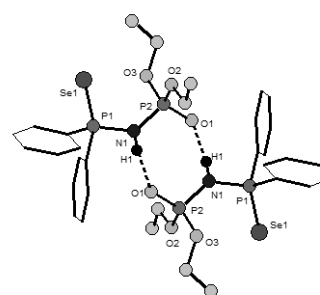
## NEW GROUP 12 METAL COMPLEXES WITH ANIONIC $[\{\text{OP}(\text{OEt})_2\}(\text{SePPh}_2)\text{N}]^-$ LIGANDS

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The organophosphorus acid  $(\text{SePPh}_2)[\text{OP}(\text{OEt})_2]\text{NH}$  (**1**) as well as its potassium salt  $\text{K}[(\text{SePPh}_2)\{\text{OP}(\text{OEt})_2\}\text{N}]$  (**2**) were prepared and structurally characterized. One-pot reactions between  $\text{MCl}_2$ ,  $^t\text{BuOK}$  and **1**, in a 1:2:2 molar ratio, resulted in group 12 metal complexes of type  $\text{M}[(\text{SePPh}_2)\{\text{OP}(\text{OEt})_2\}\text{N}]_2$  [ $\text{M} = \text{Zn}$  (**3**),  $\text{Cd}$  (**4**)]. All compounds were investigated by multinuclear NMR ( $^1\text{H}$ ,  $^{13}\text{C}$ ,  $^{31}\text{P}$ ,  $^{77}\text{Se}$ ). Single-crystal X-ray diffraction studies revealed dimeric associations in case of acid **1**, established through  $\text{NH}\cdots\text{O}=\text{P}$  hydrogen bonding.



### INTRODUCTION

Tetraorganodichalcogenoimidodiphosphinato ligands of type  $[(\text{XPR}_2)(\text{YPR}'_2)\text{N}]^-$  ( $\text{X}, \text{Y} = \text{O}, \text{S}, \text{Se}, \text{Te}$ ;  $\text{R}, \text{R}' = \text{alkyl}, \text{aryl}, \text{OR}$ ) attracted a considerable increased interest in the last three decades due to their versatility towards both main group and transition metals. Both the free acids and their metal-containing compounds were on a large extent discussed in several review papers in last years.<sup>1-5</sup> The high flexibility of the  $\text{XPNPY}$  skeleton and its monometallic biconnective behavior results in the formation of chelate rings in most of the metal complexes. Other coordination patterns, *i.e.* monodentate, monometallic monoconnective, or bridging either in a bimetallic triconnective or a bimetallic biconnective pattern are also known.<sup>3</sup>

Different metal complexes of such ligands are good candidates for applications in selective metal extraction,<sup>6-8</sup> catalysis,<sup>9-12</sup> materials science<sup>4,13-16</sup> or as NMR shift reagents.<sup>17</sup> Group 12 metal-containing species with ligands from this class found applications as precursors for materials for electronic devices.<sup>18-23</sup>

For many years our interests were focused on various tetraorganodichalcogenoimidodiphosphinic acids, as well as on their metal derivatives.<sup>1,24-28</sup> We described several zinc(II) and cadmium(II) complexes with ligands belonging to this class, with either monomeric, *e.g.*  $\text{Cd}[(\text{SPMe}_2)_2\text{N}]_2$ ,<sup>24</sup>  $\text{Zn}[(\text{OPPh}_2)_2\text{N}]_2$ ,<sup>28</sup> or dimeric structures, *e.g.*  $[\text{Cd}\{(\text{OPMe}_2)(\text{SPPH}_2)\text{N}\}_2]_2$  and  $[\text{Cd}\{(\text{OPPh}_2)_2\text{N}\}_4(\text{H}_2\text{O})]$ .<sup>24</sup> As a continuation of our studies we report here on the synthesis and spectroscopic characterization of the new acid  $(\text{SePPh}_2)[\text{OP}(\text{OEt})_2]\text{NH}$  (**1**), its

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potassium salt  $K[(SePPh_2)\{OP(OEt)_2\}N]$  (**2**) and the group 12 metal complexes  $M[(SePPh_2)\{OP(OEt)_2\}N]_2$  [ $M = Zn$  (**3**),  $Cd$  (**4**)].

## RESULTS

### Synthesis

The organophosphorus acid **1** was obtained as depicted in Scheme 1, by reacting the lithiated organophosphorus amide with  $Ph_2P(Se)Cl$ , similarly with the procedure reported for other related compounds.<sup>1</sup>

The free acid was deprotonated with  $tBuOK$ , while the metal complexes were obtained by a one pot synthesis procedure, involving a reaction mixture of **1**,  $tBuOK$  and  $MCl_2$  in a 2:2:1 molar ratio (Scheme 1).

Compounds **1** (yellow), **2** (colorless) and **4** (orange) were isolated as microcrystalline solids, while compound **3** as a yellow oil. The compounds were investigated by multinuclear NMR ( $^1H$ ,  $^{13}C$ ,  $^{31}P$  and  $^{77}Se$ ). For the acid **1** the molecular structure was determined by single-crystal X-ray diffraction.

## DISCUSSION

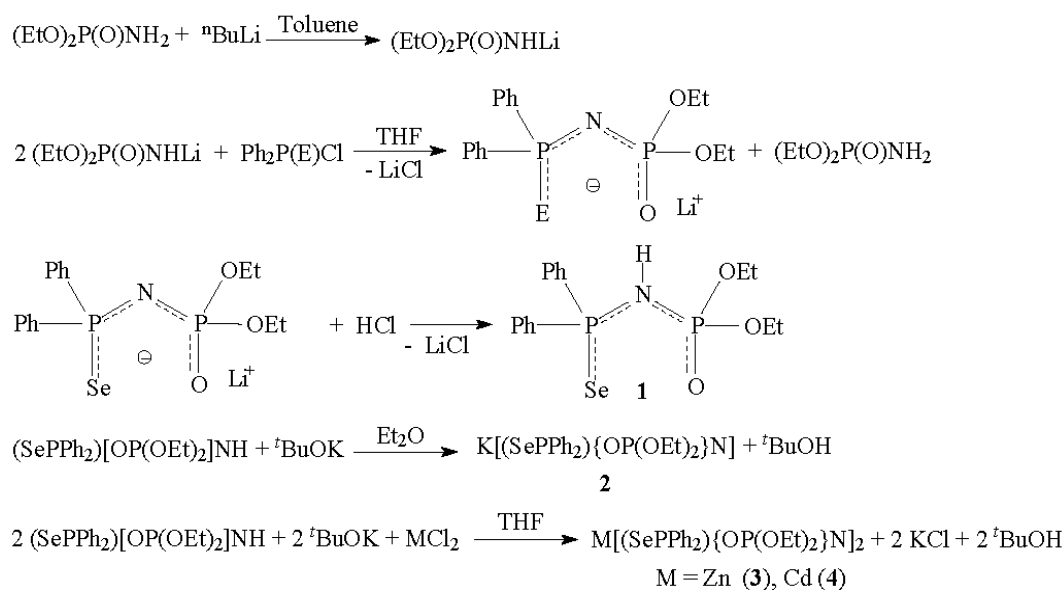
### Spectroscopy

The  $^1H$  and  $^{13}C$  NMR spectra show the expected resonances for the organic groups attached to

phosphorus, with a multiplicity determined by the proton-proton and phosphorus-proton or phosphorus-carbon couplings, respectively. The broad resonance observed in the  $^1H$  NMR spectrum of **1** at 6.80 ppm was assigned to the  $NH$  proton. All four compounds show two resonances in the  $^{31}P$  NMR spectra, either with a doublet pattern or with a broad aspect, due to the phosphorus-phosphorus coupling. They are accompanied by phosphorus-carbon and phosphorus-selenium satellites. The  $\delta[^{31}P(O)]$  values for the metal-containing species are high field and the  $\delta[^{31}P(Se)]$  values are low field shifted in comparison with the free acid, as it was also observed for the related potassium salt  $K[(SPPH_2)\{OP(OEt)_2\}N]$ .<sup>29</sup> For the cadmium and zinc complexes the NMR spectra suggest the equivalence of the two ligands of a molecular unit in solution. Only in case of the free acid **1** the  $^{77}Se$  NMR resonance could be observed, as a doublet due to the coupling with phosphorus.

### Crystal and molecular structure of $(SePPh_2)[OP(OEt)_2]NH$ (**1**)

The compound crystallizes in the  $P2(1)/n$  space group. The ORTEP-like diagram with the numbering scheme is depicted in Fig. 1, while interatomic distances and bond angles are given in Table 1.



Scheme 1

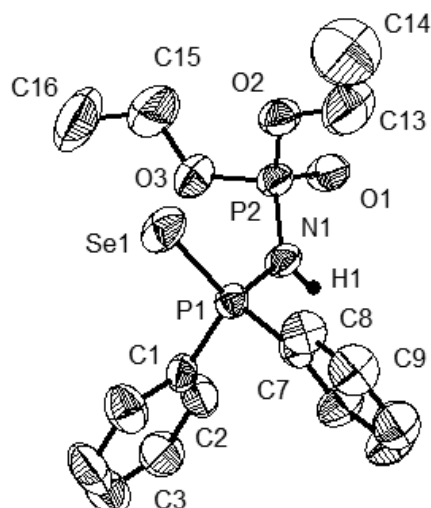
Fig. 1 – ORTEP representation at 50% probability and atom numbering scheme for **1**.

Table 1

Selected interatomic distances (Å) and angles (°) in (SePPh<sub>2</sub>)[OP(OEt)<sub>2</sub>]NH (**1**)

N(1)–P(1)	1.684(3)	P(2)–O(2)	1.551(3)
N(1)–P(2)	1.626(3)	P(2)–O(3)	1.560(3)
N(1)–H(1)	0.813(18)	P(1)–C(1)	1.810(4)
N(1)···H(1) <sup>a</sup>		P(1)–C(7)	1.810(4)
P(1)–Se(1)	2.0850(11)	C(13)–O(2)	1.452(6)
P(1)–O(1)	1.459(3)	C(15)–O(3)	1.411(6)
P(1)–N(1)–P(2)	131.0(2)	N(1)–P(2)–O(1)	110.79(17)
P(1)–N(1)–H(1)	111(3)	N(1)–P(2)–O(2)	109.57(17)
P(2)–N(1)–H(1)	117(3)	N(1)–P(2)–O(3)	104.41(18)
N(1)–P(1)–C(1)	105.87(18)	O(1)–P(2)–O(2)	114.26(18)
N(1)–P(1)–C(7)	104.06(17)	O(1)–P(2)–O(3)	114.45(19)
N(1)–P(1)–Se(1)	114.55(12)	O(2)–P(2)–O(3)	102.64(17)
C(1)–P(1)–Se(1)	113.38(13)	C(13)–O(2)–P(2)	118.8(3)
C(7)–P(1)–Se(1)	113.93(13)	C(15)–O(3)–P(2)	125.8(4)
C(1)–P(1)–C(7)	103.97(18)		

<sup>a</sup> Symmetry equivalent positions  $1-x, -y, -z$  are given by “prime”.

The compound has a similar molecular structure with other related species previously described.<sup>1,29</sup> The acidic proton, as was observed also in solution, is attached to nitrogen [N–H 0.813(18) Å]. The phosphorus–chalcogen interatomic distances have typical values for P=O and P=Se double bonds [P(2)–O(1) 1.459(3) Å vs. P=O 1.486(6), P–O 1.526(6) Å in Ph<sub>2</sub>P(=O)OH<sup>30</sup> and P(1)–Se(1) 2.0850(11) Å vs. P=Se 2.085(1) and 2.101(1) in (Se=PPh<sub>2</sub>)<sub>2</sub>NH,<sup>31</sup> respectively]. The phosphorus–nitrogen interatomic distances have typical values for single P–N bonds [1.626(3) and 1.684(3) Å vs. P=N 1.562(2) and P–N 1.610(2) Å in Ph<sub>2</sub>P(=S)–N=P(SMe)Ph<sub>2</sub>.<sup>32</sup> The P–N–P system

is angular [P(1)–N(1)–P(2) 131.0(2)°] with the sum of the angles at the nitrogen atom 359°, thus suggesting a *sp*<sup>2</sup> hybridization, as was also noted for other imidodiphosphinic acids.<sup>1</sup> The Se(1)P(1)N(1)P(2)O(1) skeleton is almost planar. The conformation of the XPNPY skeleton can be described by the torsion angle X–P···P–Y, assigning the term *anti* for values in the range 150–180° and *syn* for values smaller than 90°, respectively, taking in account the relative orientation of the phosphorus–chalcogen bonds.<sup>1</sup> Accordingly, for the molecule of **1** an *anti* conformation can be considered, with a O–P···P–Se torsion angle of 173.6°.

The  $(\text{SePPh}_2)[\text{OP}(\text{OEt})_2]\text{NH}$  molecules are associated into centrosymmetric dimers (Fig. 2) through  $\text{N}-\text{H}\cdots\text{O}=\text{P}$  hydrogen bonding [ $\text{H}(1)\cdots\text{O}(1')$  2.002 Å,  $\text{N}(1)-\text{H}(1)\cdots\text{O}(1')$  166.78°, vs.  $\Sigma r_{\text{vdW}}(\text{H},\text{O})$  2.60 Å<sup>33</sup>], similarly with the situation found in the monothio derivative  $(\text{SPPH}_2)[\text{OP}(\text{OEt})_2]\text{NH}$  [ $(\text{N})\text{H}\cdots\text{O}(\text{P})$  1.79 Å],<sup>29</sup> but different from the monothioimidodiphosphinates  $(\text{OPPh}_2)(\text{SPPH}_2)\text{NH}$ ,<sup>34</sup>  $(\text{SPMe}_2)(\text{OPPh}_2)\text{NH}$  and  $(\text{OPMe}_2)(\text{SPPH}_2)\text{NH}$ <sup>35</sup> for which polymeric chains are formed through such  $(\text{N})\text{H}\cdots\text{O}(\text{P})$  hydrogen bonds.

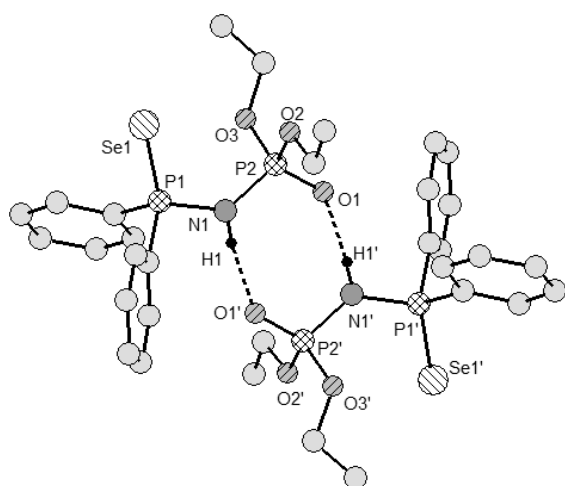


Fig. 2 – Dimeric association in the crystal of **1**. Hydrogen atoms, other than those involved in intermolecular interactions were omitted for clarity.

## EXPERIMENTAL

Starting materials, *i.e.*  $[\text{Ph}_2\text{P}(\text{Cl})]$ ,  $(\text{EtO})_2\text{P}(\text{O})\text{NH}_2$ ,  $\text{ZnCl}_2$ ,  $\text{CdCl}_2$  and  ${}^t\text{BuOK}$  were used as purchased (Aldrich).  $\text{Ph}_2\text{P}(\text{Se})\text{Cl}$  was prepared as previously reported.<sup>36</sup> The solvents were dried and freshly distilled under argon prior to use. Elemental analyses were performed on a Flash EA 1112 machine, while melting points were measured on an Electrothermal 9200 apparatus and are not corrected.  ${}^1\text{H}$ ,  ${}^{13}\text{C}$  and  ${}^{31}\text{P}$  NMR spectra were recorded in  $\text{CDCl}_3$  solution on a BRUKER Avance DRX 300 instrument. The chemical shifts are reported in  $\delta$  units (ppm) relative to the residual peak of the deuterated solvent (ref.  $\text{CHCl}_3$ :  ${}^1\text{H}$  7.26 ppm,  ${}^{13}\text{C}$  77.0 ppm) and  $\text{H}_3\text{PO}_4$  85%, respectively. The NMR spectra were processed using the MestReC and MestReNova software.<sup>37</sup>

### Preparation of $(\text{SePPh}_2)[\text{OP}(\text{OEt})_2]\text{NH}$ (**1**)

A solution of  ${}^n\text{BuLi}$  in *n*-hexane (26.1 mL, 1.6 M, 41.76 mmol) was added dropwise at room temperature to a stirred solution of  $(\text{EtO})_2\text{P}(\text{O})\text{NH}_2$  (6.38 g, 41.67 mmol) in 50 mL of anhydrous toluene, under argon atmosphere. The reaction mixture was stirred for 2 h at room temperature. Then a solution of  $\text{Ph}_2\text{P}(\text{Se})\text{Cl}$  (6.24 g, 20.83 mmol), in 30 mL of anhydrous THF was added dropwise and was maintained under stirring for additional 24 hours. The yellow reaction mixture was treated with water (60 mL). The aqueous phase

was treated with HCl 37% (3–4 mL), when a solid yellow precipitate formed. This solid product was separated by filtration and dried in vacuum. Recrystallization from a chloroform/*n*-hexane mixture (1/4, v/v) gave the title compound as a microcrystalline yellow solid. Yield: 5.97 g (68.9%). M.p. = 97°C. Anal. Calcd. for  $\text{C}_{16}\text{H}_{21}\text{NO}_3\text{P}_2\text{Se}$  (MW 416.26): C, 46.17; H, 5.09; N, 3.36%. Found: C, 46.32; H, 5.07; N, 3.41%.  ${}^1\text{H}$  NMR:  $\delta$  1.13 (dt, 6H,  $\text{OCH}_2\text{CH}_3$ ,  ${}^3J_{\text{HH}} = 7.1$ ,  ${}^3J_{\text{PH}} = 1.0$  Hz), 3.93 (dq, 4H,  $\text{OCH}_2\text{CH}_3$ ,  ${}^3J_{\text{HH}} = 7.1$ ,  ${}^3J_{\text{PH}} = 7.1$  Hz), 6.80 (s, br., 1H, NH), 7.45 (m, 6H,  $\text{C}_6\text{H}_5$ -*meta+para*), 7.98 (ddd, 4H,  $\text{C}_6\text{H}_5$ -*ortho*,  ${}^3J_{\text{HH}} = 7.3$ ,  ${}^4J_{\text{HH}} = 1.4$ ,  ${}^3J_{\text{PH}} = 14.6$  Hz).  ${}^{13}\text{C}$  NMR:  $\delta$  15.8 (d,  $\text{OCH}_2\text{CH}_3$ ,  ${}^3J_{\text{PC}} = 7.6$  Hz), 63.4 (d,  $\text{OCH}_2\text{CH}_3$ ,  ${}^2J_{\text{PC}} = 5.6$  Hz), 128.16 (d,  $\text{C}_6\text{H}_5$ -*meta*,  ${}^3J_{\text{PC}} = 13.7$  Hz), 131.84 (d,  $\text{C}_6\text{H}_5$ -*para*,  ${}^4J_{\text{PC}} = 3.3$  Hz), 131.96 (d,  $\text{C}_6\text{H}_5$ -*ortho*,  ${}^2J_{\text{PC}} = 12.3$  Hz), 133.7 (dd,  $\text{C}_6\text{H}_5$ -*ipso*,  ${}^1J_{\text{PC}} = 91.9$ ,  ${}^3J_{\text{PC}} = 2.3$  Hz).  ${}^{31}\text{P}$  NMR:  $\delta$  -0.03 [d,  $\text{P}(\text{O})$ ,  ${}^2J_{\text{PP}} = 13$  Hz], 47.4 [d,  $\text{P}(\text{Se})$ ,  ${}^2J_{\text{PP}} = 13.3$ ,  ${}^1J_{\text{PSe}} = 784.5$ ,  ${}^1J_{\text{PC}} = 91.9$  Hz].  ${}^{77}\text{Se}$  NMR:  $\delta$  -207.4 (d,  ${}^1J_{\text{PSe}} = 784.2$  Hz).

### Preparation of $\text{K}[(\text{SePPh}_2)\{\text{OP}(\text{OEt})_2\}\text{N}]$ (**2**)

A reaction mixture of **1** (6.38 g, 15.32 mmol) and  ${}^t\text{BuOK}$  (1.72 g, 15.32 mmol) in 50 mL of anhydrous diethyl ether was stirred at room temperature for 24 h. The resulting solid was separated by filtration and then dried under vacuum. The title compound was obtained quantitatively as a colorless powder. M.p. 110–111°C. Anal. Calcd. for  $\text{C}_{16}\text{H}_{20}\text{KNO}_3\text{P}_2\text{Se}$  (MW 454.35): C, 42.30; H, 4.44; N, 3.08%. Found: C, 42.61; H, 4.57; N, 3.11%.  ${}^1\text{H}$  NMR:  $\delta$  1.16 (t, 6H,  $\text{OCH}_2\text{CH}_3$ ,  ${}^3J_{\text{HH}} = 7.0$  Hz), 3.99 (dq, 4H,  $\text{OCH}_2\text{CH}_3$ ,  ${}^3J_{\text{HH}} = 6.9$ ,  ${}^3J_{\text{PH}} = 7.0$  Hz), 7.33 (m, 6H  $\text{C}_6\text{H}_5$ -*meta+para*), 7.99 (dd, 4H,  $\text{C}_6\text{H}_5$ -*ortho*,  ${}^3J_{\text{HH}} = 7.2$ ,  ${}^3J_{\text{PH}} = 13.6$  Hz).  ${}^{31}\text{P}$  NMR:  $\delta$  4.8 [d,  $\text{P}(\text{O})$ ,  ${}^2J_{\text{PP}} = 18.5$  Hz], 25.6 [s, br.,  $\text{P}(\text{Se})$ ].

### Preparation of $\text{Zn}[(\text{SePPh}_2)\{\text{OP}(\text{OEt})_2\}\text{N}]_2$ (**3**)

A reaction mixture of **1** (2 g, 4.80 mmol),  ${}^t\text{BuOK}$  (0.58 g, 5.20 mmol) and  $\text{ZnCl}_2$  (0.327 g, 2.4 mmol) in 50 mL of anhydrous THF was stirred at room temperature for 2 hours. The solvent was removed in vacuum and dichloromethane (30 mL) was added. From the yellow solution the solvent was removed in vacuum and the title compound resulted as a yellow oily product. Yield: 1.64 g (76.3%). Anal. Calcd. for  $\text{C}_{32}\text{H}_{40}\text{N}_2\text{O}_6\text{P}_4\text{Se}_2\text{Zn}$  (MW 895.87): C, 42.90; H, 4.50; N, 3.13%. Found: C, 42.71; H, 4.58; N, 3.27%.  ${}^1\text{H}$  NMR:  $\delta$  1.20 (t, 6H,  $\text{OCH}_2\text{CH}_3$ ,  ${}^3J_{\text{HH}} = 7.1$  Hz), 3.96 (dq, 4H,  $\text{OCH}_2\text{CH}_3$ ,  ${}^3J_{\text{HH}} = 7.2$ ,  ${}^3J_{\text{PH}} = 7.1$  Hz), 7.41 (m, 6H,  $\text{C}_6\text{H}_5$ -*meta+para*), 7.92 (dd, 4H,  $\text{C}_6\text{H}_5$ -*ortho*,  ${}^3J_{\text{HH}} = 5.3$ ,  ${}^3J_{\text{PH}} = 14.2$  Hz).  ${}^{13}\text{C}$  NMR:  $\delta$  16.04 (d,  $\text{OCH}_2\text{CH}_3$ ,  ${}^3J_{\text{PC}} = 7.7$  Hz), 62.27 (d,  $\text{OCH}_2\text{CH}_3$ ,  ${}^2J_{\text{PC}} = 5.6$  Hz), 128.04 (d,  $\text{C}_6\text{H}_5$ -*meta*,  ${}^3J_{\text{PC}} = 13.5$  Hz), 130.78 (d,  $\text{C}_6\text{H}_5$ -*ortho*,  ${}^2J_{\text{PC}} = 11.9$  Hz), 130.9 (s, br.,  $\text{C}_6\text{H}_5$ -*para*),  $\text{C}_6\text{H}_5$ -*ipso* was not observed.  ${}^{31}\text{P}$  NMR ( $\text{CDCl}_3$ ): 4.53 [d,  $\text{P}(\text{O})$ ,  ${}^2J_{\text{PP}} = 25.75$  Hz], 24.07 [d,  $\text{P}(\text{Se})$ ,  ${}^2J_{\text{PP}} = 22.3$ ,  ${}^1J_{\text{PSe}} = 507$  Hz].

### Preparation of $\text{Cd}[(\text{SePPh}_2)\{\text{OP}(\text{OEt})_2\}\text{N}]_2$ (**4**)

Compound **4** was prepared using a similar one-pot procedure as described for the Zn analogue **3** from **1** (0.98 g, 2.36 mmol),  ${}^t\text{BuOK}$  (0.26 g, 2.36 mmol) and  $\text{CdCl}_2$  (0.22 g, 1.18 mmol). Yield: 0.87 g (78.4%). M.p. 101–102°C. Anal. Calcd. for  $\text{C}_{32}\text{H}_{40}\text{N}_2\text{O}_6\text{P}_4\text{Se}_2\text{Cd}$  (MW 942.90): C, 40.76; H, 4.28; N, 2.97%. Found: C, 40.43; H, 4.23; N, 3.02%.  ${}^1\text{H}$  NMR:  $\delta$  1.19 (t, 6H,  $\text{OCH}_2\text{CH}_3$ ,  ${}^3J_{\text{HH}} = 7.0$  Hz), 3.94 (dq, 4H,  $\text{OCH}_2\text{CH}_3$ ,  ${}^3J_{\text{HH}} = 7.1$ ,  ${}^3J_{\text{PH}} = 7.2$  Hz), 7.39 (m, 6H,  $\text{C}_6\text{H}_5$ -*meta+para*), 7.93 (ddd, 4H,  $\text{C}_6\text{H}_5$ -*ortho*,  ${}^3J_{\text{HH}} = 7.3$ ,

$^4J_{\text{HH}} = 2.2$ ,  $^3J_{\text{PH}} = 14.2$  Hz).  $^{31}\text{P}$  NMR:  $\delta$  4.27 [d,  $P(\text{O})$ ],  $^2J_{\text{PP}} = 24.7$  Hz], 22.74 [s, br.,  $P(\text{Se})$ ].

#### X-ray structure determination

The details of the crystal structure determination and refinement for compound **1** are given in Table 2.

Data were collected on a Bruker SMART APEX diffractometer by using graphite-monochromated Mo- $K_{\alpha}$  radiation ( $\lambda = 0.71073$  Å). The crystal was attached with paraton/N oil on cryoloops and the data were collected at room temperature (297 K). The structure was refined with anisotropic thermal parameters. The hydrogen atoms were refined with a riding model and a mutual isotropic thermal parameter. For structure solving and refinement the software package SHELX-97 was used.<sup>38</sup> The drawings were created with the Diamond program.<sup>39</sup>

## CONCLUSIONS

The new imidodiphosphinic acid  $(\text{SePPh}_2)[\text{OP}(\text{OEt})_2]\text{NH}$  (**1**) was obtained and

structurally characterized by multinuclear NMR in solution and by single-crystal X-ray diffraction in solid state. Deprotonation with  $^t\text{BuOK}$  resulted in the potassium salt  $\text{K}[(\text{SePPh}_2)\{\text{OP}(\text{OEt})_2\}\text{N}]$  (**2**), while reactions with  $\text{MCl}_2$  ( $\text{M} = \text{Zn}, \text{Cd}$ ) in presence of  $^t\text{BuOK}$  lead to the complexes  $\text{M}[(\text{SePPh}_2)\{\text{OP}(\text{OEt})_2\}\text{N}]_2$  [ $\text{M} = \text{Zn}$  (**3**),  $\text{Cd}$  (**4**)]. The NMR spectra for the group 12 metal complexes revealed the equivalence of the two ligand units in solution.

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#### Supplementary material

CCDC 941305 contains the supplementary crystallographic data for compound **1**. These data can be obtained free of charge from The Cambridge Crystallographic Data Centre via [www.ccdc.cam.ac.uk/data\\_request/cif](http://www.ccdc.cam.ac.uk/data_request/cif).

Table 2

X-ray crystal data and structure refinement for  $(\text{SePPh}_2)[\text{OP}(\text{OEt})_2]\text{NH}$  (**1**)

	<b>1</b>
Empirical formula	$\text{C}_{16}\text{H}_{21}\text{NO}_3\text{P}_2\text{Se}$
Formula weight	416.24
Temperature (K)	297(2)
Wavelength (Å)	0.71073
Crystal system	Monoclinic
Space group	$P2(1)/n$
Unit cell dimensions	
<i>a</i> (Å)	9.294(2)
<i>b</i> (Å)	8.6078(18)
<i>c</i> (Å)	23.687(5)
$\alpha$ (°)	90.00
$\beta$ (°)	97.856(4)
$\gamma$ (°)	90.00
Volume (Å <sup>3</sup> )	1877.2(7)
<i>Z</i>	4
$D_c$ (g/cm <sup>3</sup> )	1.473
Absorption coefficient (mm <sup>-1</sup> )	2.183
$F(000)$	848
Crystal size, mm	0.34 x 0.30 x 0.26
$\theta$ range for data collections (°)	1.74 to 25.00
Reflections collected	13087
Independent reflections	3297
	[ $R(\text{int}) = 0.0506$ ]
Refinement method	Full-matrix least-squares on $F^2$
Data/restraints/parameters	3297 / 1 / 214
Goodness-of-fit on $F^2$	1.069
Final <i>R</i> indices [ $F^2 > 2\sigma(F^2)$ ]	$R1 = 0.0510$ , $wR2 = 0.1089$
<i>R</i> indices (all data)	$R1 = 0.0693$ , $wR2 = 0.1164$
Largest diff. peak and hole, eÅ <sup>-3</sup>	0.560 and -0.303

## REFERENCES

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