

The hydrogen-bonded dimers of N,N',N'' -tricyclohexylphosphoric triamide in new tin(IV) and copper(II) complexes

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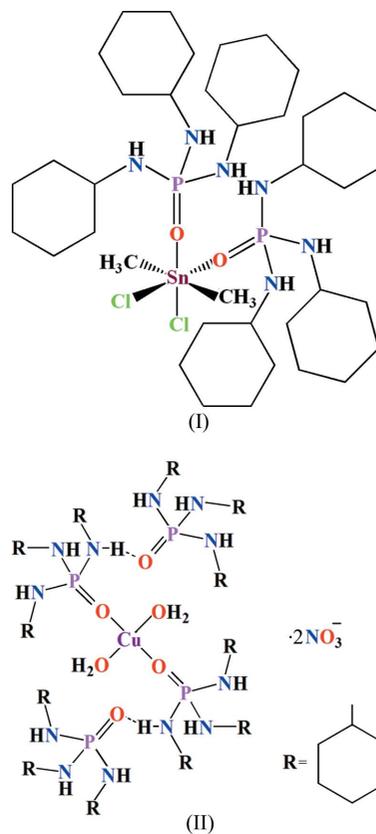
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In the new tin(IV) and copper(II) complexes, *cis*-dichlorido-*trans*-dimethyl-*cis*-bis(N,N',N'' -tricyclohexylphosphoric triamide- κO)tin(IV), [Sn(CH₃)₂Cl₂(C₁₈H₃₆N₃OP)₂], (I), and *trans*-diaquabis(N,N',N'' -tricyclohexylphosphoric triamide- κO)copper(II) dinitrate- N,N',N'' -tricyclohexylphosphoric triamide (PTA) ligands exist as hydrogen-bonded dimers *via* P=O...H-N interactions around the metal center. The asymmetric unit in (I) consists of one complete complex molecule located on a general position. The Sn^{IV} coordination geometry is octahedral with two *cis* hydrogen-bonded PTA ligands, two *cis* chloride ligands and two *trans* methyl groups. The asymmetric unit in (II) contains one half of a [Cu(PTA)₂(H₂O)₂]²⁺ dication on a special position (site symmetry $\bar{1}$ for the Cu atom), one nitrate anion and one free PTA molecule, both on general positions. The complex adopts a square-planar *trans*-[CuO₂O₂] coordination geometry, with the Cu^{II} ion coordinated by two PTA ligands and two water molecules. Each of the noncoordinated PTA molecules is hydrogen bonded to a neighboring coordinated PTA molecule and an adjacent water molecule; the phosphoryl O atom acts as a double-H-atom acceptor. The P atoms in the PTA ligands of both complexes and in the noncoordinated hydrogen-bonded molecules in (II) adopt a slightly distorted tetrahedral environment.

Comment

The P=O...H-N hydrogen bond exists in the structures of phosphoramidate compounds containing the P(O)NH moiety. In the process of coordination to a metal cation (M), one would anticipate that this interaction would be replaced with a

P=O... M bond (Gholivand *et al.*, 2006). Here we discuss two cases where this type of hydrogen bond remains in a coordinated phosphoric triamide ligand in two new complexes and consider its possible role in the coordination mode of the ligand.



Organotin(IV) complexes, particularly those with organophosphorus ligands, have attracted attention because of their versatile biological properties (Gholivand *et al.*, 2010). Similar to organophosphorus ligands, phosphoramidates are suitable for reacting with organotin(IV) compounds. In this series, a few organotin(IV)-phosphoramidate compounds have been reported with some phosphoramidate ligands. A search of the Cambridge Structural Database (CSD, Version 5.32, November 2010 update; Allen, 2002) shows that in all reported dialkyltin(IV) dihalide tris(alkylamido)phosphate compounds, the phosphoramidate ligands adopt a *trans* arrangement. We present here the first structure of this class of organotin(IV) complexes with two phosphoric triamide ligands in *cis* positions (Fig. 1). The Sn coordination geometry in *cis*-dichlorido-*trans*-dimethyl-*cis*-bis(N,N',N'' -tricyclohexylphosphoric triamide- κO)tin(IV), (I), is octahedral with the N,N',N'' -tricyclohexylphosphoric triamide (PTA) and chloride ligands *cis*, but with the methyl groups *trans* (Fig. 1 and Table 1). The P atoms in the PTA ligands adopt a slightly distorted tetrahedral environment. The Sn—O—P angles are both slightly greater than 140°. These geometric parameters are consistent with those observed in related structures: [(Me₂N)₃PO]₂X₂Me₂Sn, with X = Br (CSD refcode BDMSAP; Aslanov *et al.*, 1977), Cl (CDMSAP01; Rheingold *et al.*, 1984); [(Me₂N)₃PO]₂X₂Et₂Sn, with X = I (DUCJAM; Tursina *et al.*,

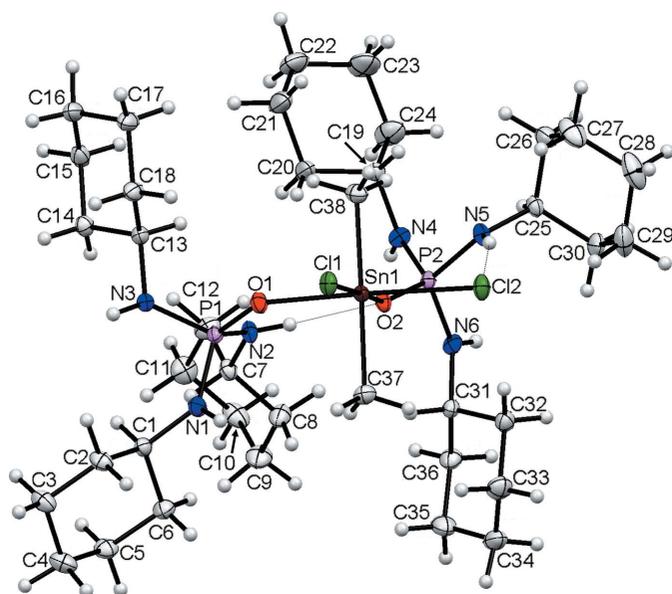


Figure 1

The molecular structure and atom-labeling scheme for (I), with displacement ellipsoids drawn at the 50% probability level. Intramolecular hydrogen bonds are shown as thin dotted lines.

1985), Br (FILCIM and FILCIM01; Tursina *et al.*, 1987); $[R_2\{\text{PhC(O)NH}\}\text{PO}]_2\text{Cl}_2\text{Me}_2\text{Sn}$ (Gholivand & Shariatinia, 2006), with $R = t\text{-BuNH}$ (YERGEI), $\text{C}_4\text{H}_8\text{N}$ (YERGIM), $\text{C}_5\text{H}_{10}\text{N}$ (YERGOS) and $(\text{Me})(\text{Cy})\text{N}$ (Cy is cyclohexyl; YERGUY); $\{(\text{C}_5\text{H}_{10}\text{N})_2[\textit{p}\text{-F-PhC(O)NH}\}\text{PO}\}_2\text{Cl}_2\text{Me}_2\text{Sn}$ (LEHWIF; Gholivand *et al.*, 2006).

The two symmetry-independent PTA ligands in (I) are involved in an intramolecular $\text{N2-H2N}\cdots\text{O2-P2}$ hydrogen bond (Table 2) in which the amide group of one ligand (P1) interacts with the phosphoryl O atom of the other ligand. This hydrogen bond may be considered as a factor helping to stabilize the previously unobserved *cis* geometry. The P2-O2 bond length is a little longer than the P1-O1 bond length, the latter O atom not serving as a hydrogen-bond acceptor. An intramolecular $\text{N5-H5N}\cdots\text{Cl2}$ hydrogen bond is also found in the structure. The environment of each N atom in the PTA ligands is almost planar and it does not form any hydrogen bonds as an acceptor, showing its low Lewis-base character. In previously reported phosphoramidate compounds and their dialkyl dichloride tin(IV)-phosphoramidate complexes (Gholivand *et al.*, 2004, 2006; Gholivand & Shariatinia, 2006), the $\text{N-H}\cdots\text{O}$ hydrogen bonds which appear in the structures of the free phosphoramidate molecules are replaced by $\text{N-H}\cdots\text{Cl}$ hydrogen bonds in the complexes, as the phosphoryl O atoms are involved in Sn-O covalent bonds in the latter. This fact may be supported by IR spectroscopy in which the N-H stretching frequencies in the tin complexes are higher than in the free molecules as they are involved in weaker $\text{N-H}\cdots\text{Cl}$ hydrogen bonds.

The two-dimensional hydrogen-bonded network in (I) is produced *via* some different weak $\text{N-H}\cdots\text{Cl}$ hydrogen bonds between the complex and three neighboring complexes in the crystal (Fig. 2). In this case, the Cl1 and Cl2 atoms are, respectively, involved in $\text{N6-H6N}\cdots\text{Cl1}^{\text{ii}}$ and $\text{N4-H4N}\cdots\text{Cl2}^{\text{ii}}$ interactions [symmetry code: (ii) $x - \frac{1}{2}, -y + \frac{1}{2}, z - \frac{1}{2}$] to build an $R_2^2(8)$ ring (Etter *et al.*, 1990; Bernstein *et al.*, 1995).

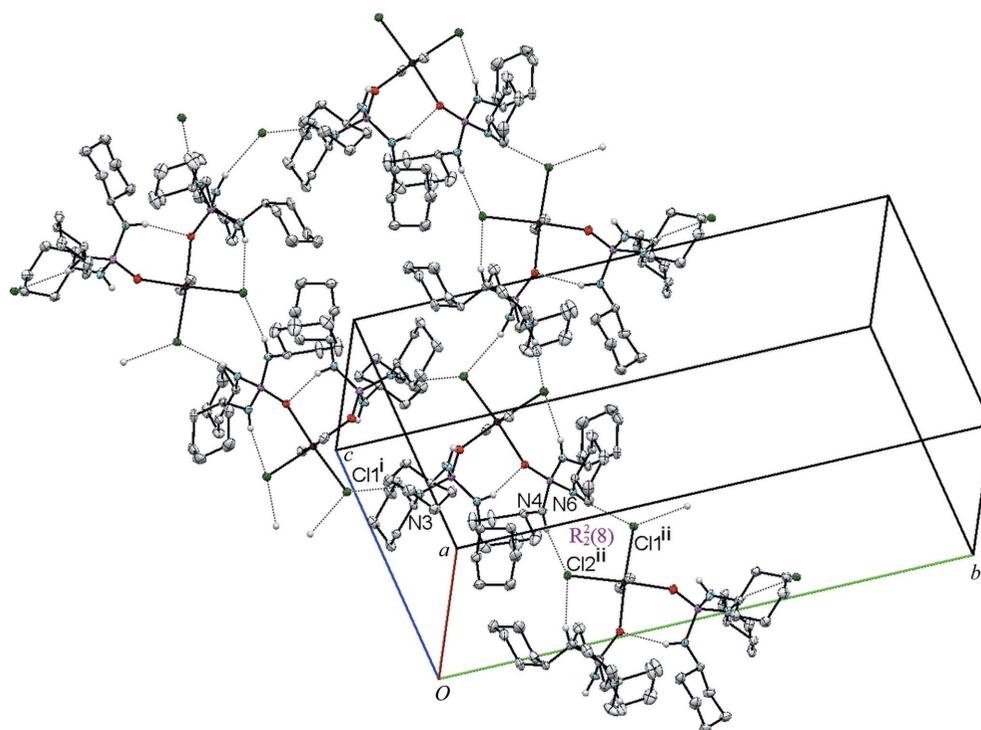


Figure 2

A view of the crystal packing of (I). Hydrogen bonds are shown as thin dotted lines. H atoms on C atoms are not shown for clarity. [Symmetry codes: (i) $-x + 2, -y, -z + 1$; (ii) $x - \frac{1}{2}, -y + \frac{1}{2}, z - \frac{1}{2}$]

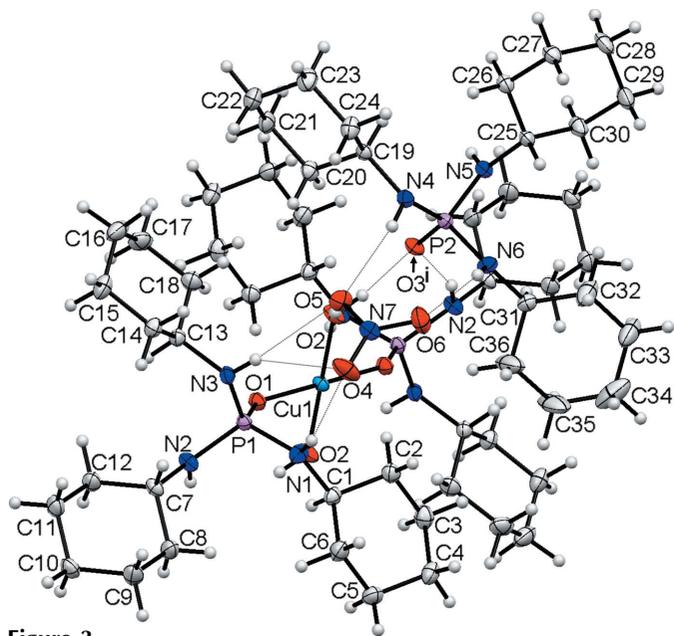


Figure 3
The molecular structure of (II), with displacement ellipsoids drawn at the 50% probability level. Hydrogen bonds are shown as thin dotted lines. [Symmetry code: (i) $-x + 1, -y + 1, -z + 1$.]

The Cl2 atom is also involved in an intramolecular N—H...Cl hydrogen bond, which has been noted earlier, while the Cl1 atom co-operates in another intermolecular N3—H3N...Cl1ⁱ hydrogen bond [symmetry code: (i) $-x + 2, -y, -z + 1$] (Table 2).

Copper complexes have been of interest as the interface between biochemistry and coordination chemistry (Collinson & Fenton, 1996; Hossain *et al.*, 1996; Tarafder *et al.*, 2002). The PTA ligand was also used to prepare a new copper(II) phosphoramidate complex, namely *trans*-diaquabis(*N,N,N'*-tricyclohexylphosphoric triamide- κO)copper(II) dinitrate-*N,N,N'*-tricyclohexylphosphoric triamide (1/2), (II) (Fig. 3). The asymmetric unit contains one half of a $[\text{Cu}(\text{PTA})_2(\text{H}_2\text{O})_2]^{2+}$ dication, one nitrate anion and one free PTA molecule. The Cu^{II} ion is located on a crystallographic inversion center. The complex thus adopts a square-planar *trans*- $[\text{CuO}_2\text{O}_2]$ coordination geometry, with the Cu^{II} ion coordinated by two PTA ligands and two water molecules. The phosphoryl O atom makes a shorter Cu—O bond than the water O atom (Table 3). The P atoms in the PTA ligands adopt a slightly distorted tetrahedral environment and the Cu—O—P angle is a little under 140° . These geometric parameters are consistent with those in related structures: $[\text{Cu}_4\text{OCl}_6\{(\text{O})\text{P}(\text{NH}^t\text{Bu})_3\}_4]$ (LIDNIW; Fu & Chivers, 2007), $[\text{Cu}(\text{ONO}_2)_2\{(\text{O})\text{P}(\text{NH}^t\text{Bu})_3\}_2]$ (LIDPAQ; Fu & Chivers, 2007) and $[\text{CuCl}_2(\text{NHC}_4\text{H}_8\text{O})\{(\text{O})\text{P}(\text{NMe}_2)_3\}]$ (YIBZEP; Xie *et al.*, 2007).

Each of the noncoordinated PTA molecules is hydrogen bonded to a neighboring coordinated PTA molecule *via* an N2—H2N...O3ⁱ hydrogen bond [Table 4; symmetry code: (i) $-x + 1, -y + 1, -z + 1$] and to an adjacent water molecule *via* an O2—H2WB...O3 hydrogen bond. The noncoordinated PTA phosphoryl O atom thus acts as a double-H-atom acceptor (Steiner, 2002). Atom O5 of the nitrate anion co-

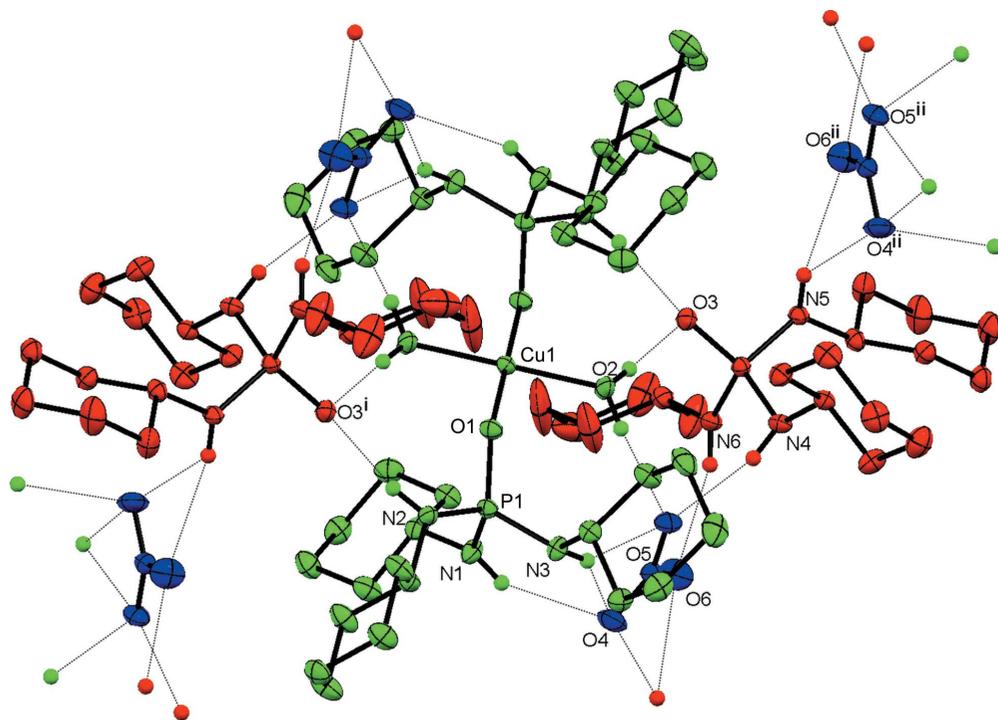


Figure 4
A view of the centrosymmetric hydrogen-bonded pentamer containing one $[\text{Cu}(\text{PTA})_2(\text{H}_2\text{O})_2]^{2+}$ dication (green in the electronic version of the paper), two PTA molecules (red) and two nitrate anions (blue); the two other nitrate anions in the top right and bottom left of the figure, which co-operate in two weak N5—H5N...O4ⁱⁱ and N5—H5N...O6ⁱⁱ hydrogen bonds, assemble neighboring pentamers in a two-dimensional array. Hydrogen bonds are shown as thin dotted lines. [Symmetry codes: (i) $-x + 1, -y + 1, -z + 1$; (ii) $x - \frac{1}{2}, -y + \frac{1}{2}, z - \frac{1}{2}$.]

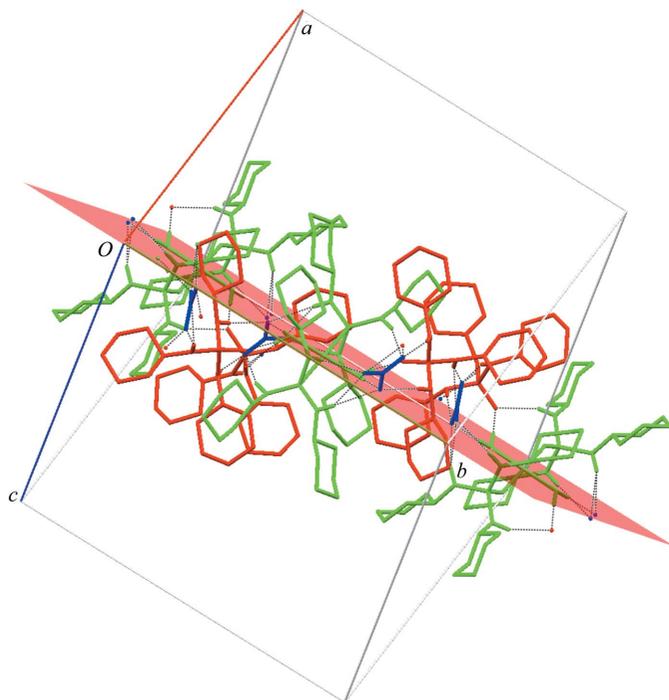


Figure 5
The two-dimensional arrangement of molecules in the structure of (II) viewed along the (101) plane.

operates in O2—H2WA···O5, N3—H3N···O5 and N4—H4N···O5 hydrogen bonds, respectively, with neighboring coordinated water, and coordinated and noncoordinated PTA molecules (Fig. 4 and Table 4). Moreover, atom O4 of the nitrate anion co-operates in hydrogen-bonding interactions with N1—H1N and N3—H3N groups of the adjacent coordinated PTA. These interactions make a centrosymmetric hydrogen-bonded pentamer containing one dication, two PTA molecules and two nitrate anions. The N5—H5N group of free PTA molecules is involved in two weak hydrogen bonds, *viz.* N5—H5N···O4ⁱⁱ and N5—H5N···O6ⁱⁱ [symmetry code: (ii) $x - \frac{1}{2}, -y + \frac{1}{2}, z - \frac{1}{2}$] (Fig. 4). These hydrogen bonds assemble the centrosymmetric pentamers in a two-dimensional arrangement along the (101) plane (Fig. 5).

Experimental

N,N',N''-Tricyclohexylphosphoric triamide (PTA) was prepared according to the procedure reported by Audrieth & Toy (1942). IR (KBr, cm^{-1}): 3272 (NH), 2930, 2852, 2667, 1659, 1440, 1237, 1145, 1111, 1004, 926, 882. For the synthesis of $[\text{Sn}(\text{CH}_3)_2\text{Cl}_2(\text{PTA})_2]$ (I), a solution of dimethyltin(IV) dichloride (0.253 g, 1.15 mmol) in chloroform (5 ml) was added dropwise to a solution of PTA (0.787 g, 2.30 mmol) in chloroform (15 ml). The clear solution was stirred for 5 h. Colorless crystals were obtained after a few days in a refrigerator (at 278 K). An effort to prepare the *trans* complex under reflux conditions (24 h) in CH_3OH was not successful and led to the *cis* complex, which was confirmed by X-ray crystallography (yield: approximately 80%). IR (KBr, cm^{-1}): 3285 (NH), 2921, 2852, 1444, 1301, 1242, 1112, 1023, 936, 792. $^{31}\text{P}\{^1\text{H}\}$ NMR (202.45 MHz, $\text{DMSO}-d_6$, 300.0 K, H_3PO_4 external): δ 12.83 (s). ^1H NMR (500.13 MHz,

$\text{DMSO}-d_6$, 300.0 K, TMS): δ 1.02 (s, 6H, CH_3), 1.13 (m, 30H), 1.49 (m, 6H), 1.63 (m, 12H), 1.79 (m, 12H), 2.80 (m, 6H, CH), 3.49 (b, 6H, NH). ^{13}C NMR (125.75 MHz, $\text{DMSO}-d_6$, 300.0 K, TMS): δ 22.95 (s, CH_3), 25.01 (s), 25.23 (s), 35.48 [$d, {}^3J(\text{P},\text{C}) = 4.7$ Hz], 49.48 (s).

For the synthesis of $[\text{Cu}(\text{PTA})_2(\text{H}_2\text{O})_2](\text{NO}_3)_2 \cdot 2\text{PTA}$ (II), a solution of copper(II) nitrate trihydrate (0.547 g, 2.26 mmol) in CH_3CN (5 ml) was added dropwise to a solution of PTA (1.423 g, 4.17 mmol) in ethanol (10 ml). The resulting clear solution was stirred for 2 h at room temperature. Single crystals were obtained from a mixture of dimethylformamide/ethanol after slow evaporation at room temperature (yield: approximately 30%). IR (KBr, cm^{-1}): 3345, 3273, 2925, 2847, 1650, 1432, 1394, 1232, 1101, 1028, 950, 882, 828. ^{13}C NMR (125.75 MHz, $\text{DMSO}-d_6$, 300.0 K, TMS): δ 26.13 (s), 37.02 (s), 41.96 (b), 49.29 (s).

Compound (I)

Crystal data

$[\text{Sn}(\text{CH}_3)_2\text{Cl}_2(\text{C}_{18}\text{H}_{36}\text{N}_3\text{OP})_2]$
 $M_r = 902.59$
Monoclinic, $P2_1/n$
 $a = 12.0882$ (6) Å
 $b = 25.1607$ (13) Å
 $c = 15.8103$ (8) Å
 $\beta = 108.879$ (1)°

$V = 4550.0$ (4) Å³
 $Z = 4$
Mo $K\alpha$ radiation
 $\mu = 0.79$ mm⁻¹
 $T = 100$ K
 $0.35 \times 0.25 \times 0.25$ mm

Data collection

Bruker SMART APEXII CCD
area-detector diffractometer
Absorption correction: multi-scan
(*APEX2*; Bruker, 2005)
 $T_{\min} = 0.772$, $T_{\max} = 0.825$

71686 measured reflections
12101 independent reflections
10758 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.030$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.024$
 $wR(F^2) = 0.080$
 $S = 1.00$
12101 reflections

462 parameters
H-atom parameters constrained
 $\Delta\rho_{\text{max}} = 0.84$ e Å⁻³
 $\Delta\rho_{\text{min}} = -0.48$ e Å⁻³

Compound (II)

Crystal data

$[\text{Cu}(\text{C}_{18}\text{H}_{36}\text{N}_3\text{OP})_2(\text{H}_2\text{O})_2](\text{NO}_3)_2 \cdot 2\text{C}_{18}\text{H}_{36}\text{N}_3\text{OP}$
 $M_r = 1589.46$
Monoclinic, $P2_1/n$
 $a = 15.4495$ (12) Å
 $b = 18.6924$ (14) Å
 $c = 16.5229$ (12) Å

$\beta = 116.656$ (1)°
 $V = 4264.5$ (6) Å³
 $Z = 2$
Mo $K\alpha$ radiation
 $\mu = 0.39$ mm⁻¹
 $T = 100$ K
 $0.24 \times 0.20 \times 0.18$ mm

Data collection

Bruker APEXII CCD
diffractometer
Absorption correction: multi-scan
(*SADABS*; Bruker, 2009)
 $T_{\min} = 0.911$, $T_{\max} = 0.932$

32668 measured reflections
9496 independent reflections
7117 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.054$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.043$
 $wR(F^2) = 0.106$
 $S = 1.04$
9496 reflections
490 parameters
9 restraints

H atoms treated by a mixture of independent and constrained refinement
 $\Delta\rho_{\text{max}} = 0.47$ e Å⁻³
 $\Delta\rho_{\text{min}} = -0.51$ e Å⁻³

Table 1
Selected geometric parameters (Å, °) for (I).

Sn1—C38	2.1114 (16)	P1—O1	1.5032 (11)
Sn1—O1	2.2281 (11)	P1—N1	1.6397 (14)
Sn1—Cl1	2.5479 (4)	P2—O2	1.5116 (11)
C38—Sn1—C37	176.31 (6)	O2—P2—N6	107.63 (6)
C38—Sn1—O1	90.69 (6)	P1—O1—Sn1	142.63 (7)
O1—Sn1—O2	87.31 (4)	P2—O2—Sn1	144.10 (6)
C38—Sn1—Cl1	90.83 (5)	C1—N1—P1	128.23 (11)
O2—Sn1—Cl1	176.07 (3)	C7—N2—P1	123.14 (10)
O1—P1—N3	117.82 (7)	C13—N3—P1	120.32 (10)
O1—P1—N2	106.76 (7)	C19—N4—P2	124.77 (10)
O2—P2—N4	117.89 (7)	C25—N5—P2	122.46 (11)

Table 2
Hydrogen-bond geometry (Å, °) for (I).

<i>D</i> —H... <i>A</i>	<i>D</i> —H	H... <i>A</i>	<i>D</i> ... <i>A</i>	<i>D</i> —H... <i>A</i>
N2—H2N...O2	0.86	2.16	2.9900 (16)	161
N3—H3N...Cl1 ⁱ	0.92	2.61	3.4403 (13)	151
N4—H4N...Cl2 ⁱⁱ	0.88	2.46	3.3296 (12)	169
N5—H5N...Cl2	0.83	2.51	3.3135 (15)	162
N6—H6N...Cl1 ⁱⁱ	0.87	2.53	3.3736 (14)	165

Symmetry codes: (i) $-x + 2, -y, -z + 1$; (ii) $x - \frac{1}{2}, -y + \frac{1}{2}, z - \frac{1}{2}$.

Table 3
Selected geometric parameters (Å, °) for (II).

P1—O1	1.5090 (14)	Cu1—O2	1.9229 (15)
P1—N2	1.6240 (17)	P2—O3	1.5024 (15)
P1—N3	1.6274 (18)	P2—N5	1.6288 (16)
P1—N1	1.6372 (17)	P2—N6	1.6348 (17)
Cu1—O1	1.8888 (12)	P2—N4	1.6495 (17)
O1—P1—N2	103.41 (8)	P1—O1—Cu1	139.13 (9)
O1—P1—N3	118.43 (9)	O3—P2—N5	106.71 (8)
N2—P1—N3	106.46 (9)	O3—P2—N6	114.77 (9)
O1—P1—N1	111.53 (8)	N5—P2—N6	107.65 (8)
N2—P1—N1	115.45 (9)	O3—P2—N4	116.43 (8)
N3—P1—N1	102.03 (9)	N5—P2—N4	110.75 (9)
O1—Cu1—O2	91.42 (6)	N6—P2—N4	100.25 (8)

Table 4
Hydrogen-bond geometry (Å, °) for (II).

<i>D</i> —H... <i>A</i>	<i>D</i> —H	H... <i>A</i>	<i>D</i> ... <i>A</i>	<i>D</i> —H... <i>A</i>
N1—H1N...O4	0.85 (1)	2.28 (1)	3.064 (2)	154 (2)
N3—H3N...O5	0.85 (1)	2.45 (1)	3.199 (2)	148 (2)
N3—H3N...O4	0.85 (1)	2.47 (1)	3.232 (2)	149 (2)
O2—H2WA...O5	0.82 (1)	1.99 (1)	2.811 (2)	175 (2)
O2—H2WB...O3	0.83 (1)	1.77 (1)	2.5926 (19)	170 (2)
N2—H2N...O3 ⁱ	0.85 (1)	2.18 (1)	2.983 (2)	159 (2)
N4—H4N...O5	0.85 (1)	2.23 (1)	3.050 (2)	163 (2)
N5—H5N...O6 ⁱⁱ	0.84 (1)	2.43 (1)	3.244 (2)	164 (2)
N5—H5N...O4 ⁱⁱ	0.84 (1)	2.73 (2)	3.359 (2)	133 (2)
N6—H6N...O6	0.84 (1)	2.46 (1)	3.258 (2)	159 (2)

Symmetry codes: (i) $-x + 1, -y + 1, -z + 1$; (ii) $x - \frac{1}{2}, -y + \frac{1}{2}, z - \frac{1}{2}$.

For (I), N-bonded H atoms were found in difference Fourier maps. C-bonded H-atom positions were calculated, and the C—H distances

were normalized at standard values of 0.98–1.00 Å, depending on the hybridization. All the H atoms were treated as riding atoms with their $U_{\text{iso}}(\text{H})$ parameters equal to $1.5U_{\text{eq}}(\text{C})$ for methyl H atoms and $1.2U_{\text{eq}}(\text{C})$ otherwise. For (II), H atoms on N and O atoms were found in difference Fourier maps and were allowed to refine with N—H = 0.86 (1) Å, O—H = 0.84 (1) Å and H2WA...H2WB = 1.20 (2) Å, and with isotropic displacement parameters fixed at $1.2U_{\text{eq}}$ of the parent atom. All other H atoms were placed in calculated positions and included as riding atoms, with C—H = 1.00 (CH) or 0.99 Å (CH₂) and $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{C})$.

For both compounds, data collection: *APEX2* (Bruker, 2005). Cell refinement: *APEX2* for (I); *SAINT* (Bruker, 2008) for (II). Data reduction: *APEX2* for (I); *SAINT* for (II). Program(s) used to solve structure: *SHELXTL* (Sheldrick, 2008) for (I); *SHELXS97* (Sheldrick, 2008) for (II). Program(s) used to refine structure: *SHELXTL* for (I); *SHELXL97* (Sheldrick, 2008) for (II). For both compounds, molecular graphics: *Mercury* (Macrae *et al.*, 2008); software used to prepare material for publication: *SHELXTL*.

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Supplementary data for this paper are available from the IUCr electronic archives (Reference: SQ3282). Services for accessing these data are described at the back of the journal.

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