

Dinuclear 3D metal complexes based on a carbacylamidophosphate ligand: redetermination of the ligand crystal structure

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Abstract Dinuclear Ni(II), Co(II) and Zn(II) complexes of general formula $[M_2^{II}Cl_4(HL)_4(i\text{-PrOH})_2 \cdot 2(i\text{-PrOH})]$ with a carbacylamidophosphate ligand, namely 2,2,2-trichloro-*N*-(dipiperidine-1-yl-phosphoryl)acetamide ($CCl_3C(O)N(H)P(O)[N(CH_2)_5]_2$), were synthesized and characterized by physicochemical and spectroscopic methods. Electronic absorption spectra of the nickel and cobalt complexes were measured at room temperature in toluene and in the solid state. The crystal structures of HL and $[Ni_2Cl_4(HL)_4(i\text{-PrOH})_2 \cdot 2(i\text{-PrOH})]$ have been determined by single-crystal XRD studies. Earlier, the structure of a monoclinic $C2/c$ modification of HL was reported. In this paper, redetermination of the structure of HL with triclinic crystal system, $P\bar{1}$, was made. The nickel complex is a chloro-bridged dimer, in which the Ni(II) centers are in a distorted octahedral geometry and the neutral HL is coordinated via its phosphoryl oxygen atom. This coordination mode was determined for the first time for 3D metal carbacylamidophosphate complexes.

Introduction

Carbacylamidophosphates (CAPHs), $RC(O)N(H)P(O)R_2^1$, containing both nitrogen–phosphoryl and nitrogen–

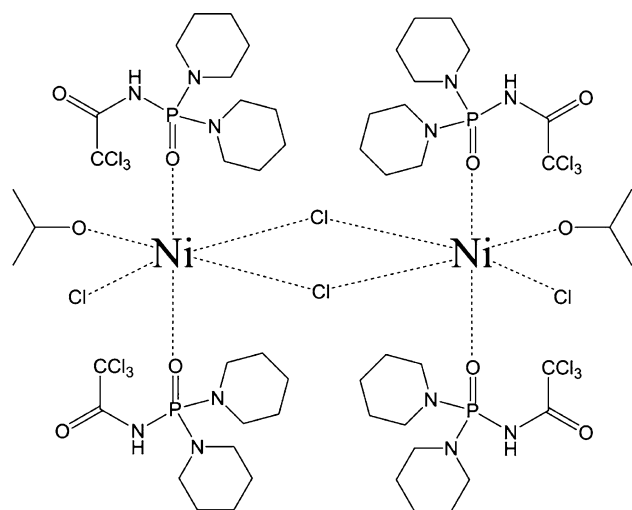
carbonyl bonds in the same molecule, have attracted scientific attention as ligands with a wide range of coordination possibilities for d- and f-block metals [1–4]. Moreover, there is a practical interest in these complexes due to their inhibition of PlsY acyltransferase and aspartate semi-aldehyde dehydrogenase ASA-DH [5–7], antiviral [8] and anticancer activities [9, 10] and their powerful extraction ability (liquid–liquid extraction of the transuranium elements) [11, 12].

The presence of the phosphoryl group in CAPH, possessing a high affinity for Lewis acids, allows us to use this type of ligand in lanthanide and actinide coordination chemistry [1, 2]. Chelating O,O-coordination of the deprotonated form (L^-) and monodentate coordination via the phosphoryl oxygen of the neutral form (HL) are the most characteristic CAPH complexation modes in lanthanide coordination compounds [13–15]. Bidentate chelate CAPH coordination to 3D metals has been reported [3, 4, 16]. Much effort has been devoted to detect the neutral CAPH coordination to 3D metal centers via the phosphoryl oxygen, similar to phosphorylic ligands in lanthanide complexes, but so far without success.

As one part of our ongoing research into the coordination chemistry of CAPHs, we report herein the synthesis and X-ray crystal structure of a CAPH ligand and some of its dinuclear 3D metal complexes; specifically, $[M_2^{II}Cl_4(HL)_4(i\text{-PrOH})_2 \cdot 2(i\text{-PrOH})]$ [where $M^{II} = Ni, Co$ or Zn ; $HL = CCl_3C(O)N(H)P(O)[N(CH_2)_5]_2$ —2,2,2-trichloro-*N*-(dipiperidine-1-yl-phosphoryl)acetamide]. Monodentate HL coordination via the phosphoryl oxygen to nickel was determined for the nickel complex (Scheme 1) by single-crystal X-ray analysis. The nickel and zinc coordination compounds are isostructural according to X-ray powder diffraction data.

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Scheme 1 Structural formula of $[\text{Ni}_2\text{Cl}_4(\text{HL})_4(\text{i-PrOH})_2 \cdot 2(\text{i-PrOH})]$

Experimental

Materials and instruments

Solvents were used as supplied or were distilled using standard methods. Elemental analyses (C, H, N) were obtained on an Elementar Vario Micro Cube elemental analyzer. Metal ions were titrated by a complexometric method, using EDTA and murexide as indicator. IR spectra were recorded using KBr pellets on a Perkin–Elmer Spectrum BX FTIR spectrophotometer in the range of 4,000 to 400 cm^{-1} . ^1H (TMS) and ^{31}P (H_3PO_4 in D_2O) NMR spectra were recorded using a pulse microwave spectrometer, Varian AMX 400, at room temperature in DMSO-d_6 solutions. The XRD data were recorded on a DRON-3 XRD system using monochromatic CuK_α radiation ($\lambda = 1.5406 \text{ \AA}$) with step size of 0.5 min^{-1} . UV–VIS absorption spectra ($12,000\text{--}30,000 \text{ cm}^{-1}$) of the complex solutions in absolute toluene and acetone at room temperature were obtained using a KSVU-23 “LOMO” UV spectrophotometer interfaced with an IBM PC. The electronic diffuse reflectance spectra were recorded in the same range using a SPECORD M-40 spectrometer.

X-ray analysis

Single-crystal XRD data for HL (**1**) and $[\text{Ni}_2\text{Cl}_4(\text{HL})_4(\text{i-PrOH})_2 \cdot 2(\text{i-PrOH})]$ (**2**) were collected at 20 °C on an “Xcalibur 3” diffractometer (MoK_α radiation, CCD detector, graphite monochromator, ω -scan). Data processing was carried out using the Patterson method, and the structures were solved using the programs SHELXS and SHELXL-97 [17, 18]. Full-matrix least-squares refinement against F^2 in anisotropic approximation for non-hydrogen atoms was used. Positions of the H atoms were located from electron density difference maps and refined by “riding” model with

Table 1 Crystal data and structure refinement parameters for **1** and **2**

	1	2
Empirical formula	$\text{C}_{12}\text{H}_{21}\text{N}_3\text{O}_2\text{P}_4\text{Cl}_3$	$\text{C}_{28.5}\text{H}_{54}\text{Cl}_8\text{N}_6\text{NiO}_{5.5}\text{P}_2$
Crystal color	Colorless	Yellow
Formula weight ($\text{g}^{-1} \text{ mol}^{-1}$)	376.64	973.03
Wavelength (\AA^{-1})	0.71073	0.71073
Crystal system	Triclinic	Monoclinic
Space group	P-1	$\text{P}2_1/\text{n}$
a (\AA)	9.3410(12)	14.553(9),
b (\AA)	10.0710(15)	15.432(15)
c (\AA)	10.72610(13)	19.277(3)
α ($^\circ$)	78.391(11)	90
β ($^\circ$)	75.336(11)	90.848(9)
γ ($^\circ$)	63.297(14)	90
V (\AA^3)	867.(6)	4,329.1(9)
Z	2	2
ρ_{calcd} ($\text{mg}^{-1} \text{ m}^{-3}$)	1.442	1.493
F(000)	392	2,020
θ range for data coll.	$3.16^\circ\text{--}27.50^\circ$	$4.32^\circ\text{--}30.00^\circ$
hkl range	$-12 \leq h \leq 12,$ $-13 \leq k \leq 13,$ $-13 \leq l \leq 13$	$-20 \leq h \leq 20,$ $-21 \leq k \leq 19,$ $-27 \leq l \leq 26$
Measured reflections	7,841	12,483
Independent reflections [R_{int}]	3,887 [0.0255]	7,881 [0.0434]
Data/restraints/parameters	3,887/0/190	12,483/0/503
Goodness of fit on F^2	1.051	0.877
Final R indices [$I > 2\sigma(I)$]	$R_1 = 0.0424,$ $wR_2 = 0.1149$	$R_1 = 0.0520,$ $wR_2 = 0.1367$
R indices (all data)	$R_1 = 0.0574,$ $wR_2 = 0.1249$	$R_1 = 0.0922,$ $wR_2 = 0.1250$

fixed thermal parameters. Further details concerning the crystal data collection and refinement are given in Table 2. Crystallographic data (excluding structure factors) for the structures reported in this paper have been deposited with the Cambridge Crystallographic Data Centre as supplementary publication numbers CCDC-871746 and CCDC-871747 [19]. Copies of the data can be obtained free of charge on application to CCDC, 12 Union Road, Cambridge CB2 1EZ, UK [fax: (internat.) +44 1223/336-033; e-mail: deposit@ccdc.cam.ac.uk]. Details of the data collection and refinements are given in Table 1.

Preparation of HL

A solution of piperidine (17 g, 0.2 mol) and triethylamine (20.2 g, 0.2 mol) in dioxane (100 ml) was cooled to 268 K, and then, a solution of the dichloranhydride of trichloroacetamidophosphoric acid (27.9 g, 0.1 mol) in dioxane (400 ml) was added dropwise under vigorous

stirring. The temperature was not allowed to rise above 278 K. The stirring was then continued for 1 h. The resulting precipitate of $N(C_2H_5)_3HCl$ was filtered off, and the filtrate was evaporated. The oily precipitate of 2,2,2-trichloro-*N*-(dipiperidine-1-yl-phosphoryl)acetamide) was isolated and recrystallized from 2-propanol as a white crystalline powder. Colorless crystals of HL were obtained by slow evaporation of the mother liquor, washed with cold 2-propanol (10 ml) and finally dried in air (yield 85 %). 1H NMR (400 MHz, DMSO- d_6 , 25 °C): δ = 1.51 (d, 4H $_{\beta}$, CH $_2$), 1.58 (d, 2H $_{\gamma}$, CH $_2$), 3.16 (s, 4H $_{\alpha}$, CH $_2$), 9.37 (s, 1H, NH). ^{31}P NMR (162 MHz, DMSO- d_6 , 25 °C): δ = 10.18 (m).

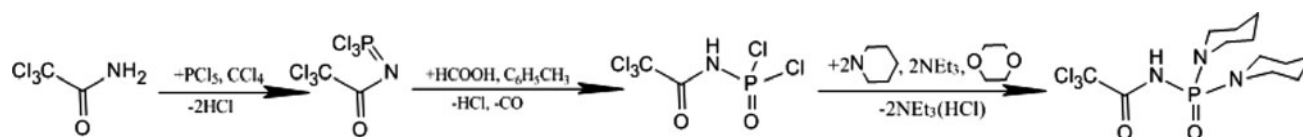
Preparation of the complexes

Solutions of hydrated cobalt(II), nickel(II) or zinc(II) chloride (0.1 mmol) in dry methanol (10 ml) and HL (0.075 g, 0.2 mmol) in 2-propanol (15 ml) were combined. The mixture was left at ambient temperature for crystallization in a vacuum desiccator over $CaCl_2$. The resulting crystals were filtered off, washed with cold isopropanol and dried in air. Yield 81–85 %. Anal. Calc. for $C_{57}C_{116}N_{12}Ni_2O_{11}P_4$: Ni, 5.8; C, 2.9; H, 5.8; N, 0.6. Found: Ni, 5.9; C, 3.1; H, 5.5; N, 0.5. Anal. Calc. for $C_{57}C_{116}N_{12}Co_2O_{11}P_4$: Ni, 5.9; C, 2.9; H, 5.8; N, 0.6. Found: Co, 6.0; C, 3.2; H, 5.5; N, 0.4. Anal. Calc. for $C_{57}C_{116}N_{12}Zn_2O_{11}P_4$: Zn, 6.3; C, 2.9; H, 5.7; N, 0.6. Found: Zn, 6.5; C, 3.2; H, 5.8; N, 0.5.

All of the compounds, excluding 2, were isolated as air-stable crystalline products. Compound 2 was decomposed after losing solvent molecules. According to the X-ray diffraction data, the prepared coordination complexes with nickel(II) and zinc(II) are isostructural, but the cobalt(II) complex has a different structure. The complexes are readily soluble in acetone, alcohols and toluene, less soluble in benzene and essentially insoluble in water. 1H NMR (400 MHz, DMSO- d_6 , 25 °C) of $[Zn_2Cl_4(HL)_4(i-PrOH)_2 \cdot 2(i-PrOH)]$: δ = 1.03 (d, 6H, CH $_3$), 1.50 (d, 4H $_{\beta}$, CH $_2$), 1.57 (d, 2H $_{\gamma}$, CH $_2$), 3.13 (s, 4H $_{\alpha}$, CH $_2$), 3.19 (s, 1H, OH), 3.69 (sep, 1H, CH), 9.57 (s, 1H, NH). ^{31}P NMR (162 MHz, DMSO- d_6 , 25 °C): δ = 9.57 (m).

Results and discussion

The dichloranhydride of trichloroacetylamidophosphoric acid was prepared according to the method reported by Kirsanov [19], Scheme 2.



Scheme 2 Synthesis of HL

IR spectra (4,000–400 cm^{-1}) of HL and the complexes are mainly characterized by the vibrational absorptions of the phosphoryl and carbonyl groups, which are sensitive to the coordination mode of the CAPH ligand [1, 3, 4, 20, 21]. Two strong bands at 1,729 and 1,194 cm^{-1} are attributable to the stretching vibrations of the C=O and P=O groups, respectively, and are indicative of monodentate HL behavior, as was observed previously for lanthanide complexes [22, 23].

The FTIR spectral data of HL and the complexes are listed in Table 2. The coordination is confirmed by the ligand's characteristic IR spectroscopic bands: $\nu_{as}(C=O)$, $\nu_{as}(P=O)$, $\nu(\text{Amide II})$ and $\nu(\text{NH})$ [24]. The low-frequency shift of the $\nu(P=O)$ band ($\Delta\nu(PO) = 23\text{--}31\text{ cm}^{-1}$) is diagnostic of the ligand's coordination to the metal. Also, the $\nu(C=O)$ absorption band in the spectra of the complexes slightly shifted to higher frequencies in comparison with free HL, due to an increase in the CO bond order upon complexation.

The UV–VIS absorption data provide evidence of an octahedral environment for the Ni(II) and Co(II) atoms in their complexes. Thus, in the spectrum of 0.01 M $[Ni_2Cl_4(HL)_4(i-PrOH)_2 \cdot 2(i-PrOH)]$ toluene solution, an intense and broad band at 21,000–24,000 cm^{-1} is assigned to $^3A_{2g} \rightarrow ^3T_{1g}$, $^3T_{1g}(P)$ transitions. An intense and broad band at 13,500–19,000 cm^{-1} in the spectrum of 0.01 M $[Co_2Cl_4(HL)_4(i-PrOH)_2 \cdot 2(i-PrOH)]$ toluene solution corresponds to $^4A_{2g} \rightarrow ^4T_{1g}$ transition [25]. The same d–d transitions are observed in the diffusion reflectance spectra of the Ni and Co complexes in the solid state. Thus, the infrared and electronic spectra of the complexes are in good agreement with their structures.

An X-ray diffraction study was carried out for HL (1) and $[Ni_2Cl_4(HL)_4(i-PrOH)_2 \cdot 2(i-PrOH)]$ (2). Selected bond distances and angles with the estimated standard deviations are shown in Table 3. Parameters of the hydrogen bonds are listed in Table 4. The HL structure is a redetermination; a monoclinic C2/c modification of 2,2,2-trichloro-*N*-(dipiperidine-1-yl-phosphoryl)acetamide, obtained by slow evaporation of an *n*-heptane–chloroform (1:5 v/v) solution, was described earlier [26]. In the present work, the HL structure is in monoclinic crystal system, P2 $_1$ /n. The unit cell consists of centrosymmetric (HL) $_2$ dimers (Fig. 1). Molecules are linked via hydrogen bonds involving the phosphorylic oxygen atoms and amide hydrogen atoms of neighboring molecules. The parameters of these hydrogen bonds (Table 4) are typical for dimers of CAPHs [27, 28].

Table 2 FTIR data (cm⁻¹) for HL and its complexes

Vibration frequency (cm ⁻¹)	HL	[M ₂ Cl ₄ (HL) ₄ (i-PrOH) ₂ ·2(i-PrOH)]		
		Ni	Co	Zn
v(NH)	3,027 m, b	3,030 m, b	3,038 m, b	3,041 m, b
v(C=O)	1,729 vs, sp	1,731 vs, sp	1,735 vs, sp	1,736 vs, sp
v(P=O)	1,194 s, sp	1,165 s, sp	1,170 s, sp	1,169 s, sp
Amide II	1,430 m, sp	1,465 m, sp	1,440 m, sp	1,440 m, sp

s strong, *vs* very strong, *m* medium, *b* broad, *sp* sharp

Table 3 Selected bond lengths (Å) and angles (°) of **1** and **2** with the estimated standard deviations

		1		2	
P1–O1	1.4741(15)	P1–O1	1.481(2)	Ni1–O1	2.049(2)
		P2–O3	1.477(2)	Ni1–O3	2.016(2)
P1–N3	1.6268(16)	P1–N3	1.621(3)	Ni1–O5	2.106(2)
		P2–N5	1.625(3)	Ni1–Cl7	2.421(8)
P1–N2	1.639(2)	P1–N2	1.635(3)	Ni1–Cl8	2.386(8)
		P2–N6	1.624(3)	Ni1–Cl8.1	2.414(8)
P1–N1	1.7019(16)	P1–N1	1.689(3)		
		P2–N4	1.691(2)		
C1–O2	1.200(2)	C1–O2	1.203(4)		
		O4–C13	1.201(4)		
O1–P1–N3	111.22(9)	O1–P1–N3	112.66(14)	O3–Ni–Cl8	175.22(6)
		O3–P2–N5	107.63(13)	O3–Ni–Cl8.1	89.34(6)
O1–P1–N2	119.44(10)	O1–P1–N2	116.46(14)	Cl8–Ni–Cl8.1	89.16(3)
		O3–P2–N6	119.94(13)	O1–Ni–Cl8	93.41(6)
O1–P1–N1	105.60(9)	O1–P1–N1	105.05(12)	Ni–Cl8–Ni1.1	175.70(6)
		O3–P2–N4	106.21(12)	Ni–Cl8.1–Ni	90.84(3)
N3–P1–N1	111.06(9)	N3–P1–N1	111.42(15)		
		N5–P2–N4	112.42(13)		
C1–N1–P1	125.09(14)	C1–N1–P1	126.50(2)		
		C13–N4–P2	126.50(2)		

Table 4 Geometry of the hydrogen bonds D–H...A for **1** and **2**

	Donor		Acceptor (A)	d (Å)			Angle
	(D)	(H)		D–H	A...H	D...A	
1	N1	H1B	O(1)*	0.86	2.00	2.776	150.3
2	N1	H1A	Cl(7)	0.86	2.42	3.204	151.1
	N4	H4A	Cl(7)	0.86	2.37	3.180	156.7
	O(5)	H(5C)	O(2)	0.82	2.09	2.909	174.9

Symmetry transformations used to generate equivalent atoms:

* $-x + 1, -y + 1, -z + 2$

The molecules of HL display the O–C–N–P–O fragment in anti conformation, with the oxygen atoms placed on the opposite sides of the CNP plane (Fig. 1). The O=P...C=O

torsion angle is 161.09°. The phosphorus atom of HL has a slightly distorted tetrahedral configuration. The OPN angles [105.60°–119.44° (see Table 4)] differ from the ideal value of 109.4°, probably because of the formation of hydrogen bonds of the P=O...H–N type. The carbon–oxygen bond distance [1.200 Å (Table 3)] and the phosphorus–nitrogen bond distances (range 1.626–1.701 Å) are typical of C=O double and P–N single bonds, respectively. The phosphorus–oxygen bond length (1.473 Å) is longer than a normal P=O bond (1.45 Å) [29]. It is, however, characteristic of a hydrogen-bonded phosphorus atom with amide-type substituents.

The CNHP system is nearly planar, thus suggesting considerable sp² character of the nitrogen atom, while the C–N–P system is nonlinear (angle is 125.09°). The structural studies of HL crystals show that the P–N_{amine}

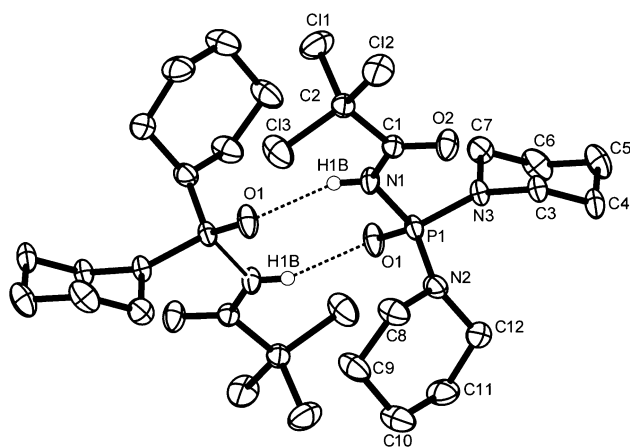


Fig. 1 Association of HL molecules in dimers by intermolecular hydrogen bonds (*dashed lines*). Displacement ellipsoids are shown at 30 % probability level. View normal to (001)

bonds are longer than the P–N_{amide} bond. Moreover, they are shorter than a typical P–N single bond (1.77 Å for NaHPO₃NH₂) [29], but longer than a P=N double bond (1.57 Å) [29]. The piperidine rings are in a chair conformation.

The molecule of di- μ -chlorido-bis[chlorido(2,2,2-trichloro-*N*-(dipiperidine-1-yl)-phosphoryl)acetamide]-nickel(II) (**2**) is a centrosymmetric dimer with each Ni^{II} center in a distorted octahedral coordination environment (Fig. 2). The neutral phosphoryl ligands are coordinated in a monodentate manner via oxygen atoms O1 and O3 of the phosphoryl groups. The present structure differs from those previously reported for 3D metal complexes, which only showed bidentate coordinated CAPH ligands in neutral form [4, 5]. The chlorides Cl8 and Cl8.1 ($-x, -y, 1-z$) act as bridging ligands between the nickel atoms, while the axial Cl atoms (Cl7) take part in the formation of two intramolecular N–H \cdots Cl hydrogen bonds with the amide groups of HL ligands. The octahedral coordination of the nickel atom is completed by an oxygen atom from an isopropanol ligand. The complex molecule is located around an inversion center and exhibits a planar Ni₂(μ -Cl)₂ framework (Fig. 2b). The Ni \cdots Ni interatomic distance of 3.42 Å virtually excludes any specific interactions between these atoms.

The nickel(II) coordination polyhedra occur as the fac isomer (Fig. 2b). The values of the bond lengths for Ni1–Cl8 and Ni1–Cl8.1 [2.3860(8) and 2.4140(8) Å, respectively] are similar to those of Ni1–Cl7 and Ni1–Cl7.1 [2.4216(8) Å] (Table 3). The Cl8–Ni1–Cl8.1 ($-x, -y, 1-z$) and Ni1–Cl8–Ni1.1 ($-x, -y, 1-z$) angles in the [Ni₂(μ -Cl)₂] cycle lie in the range of 89.16(3)°

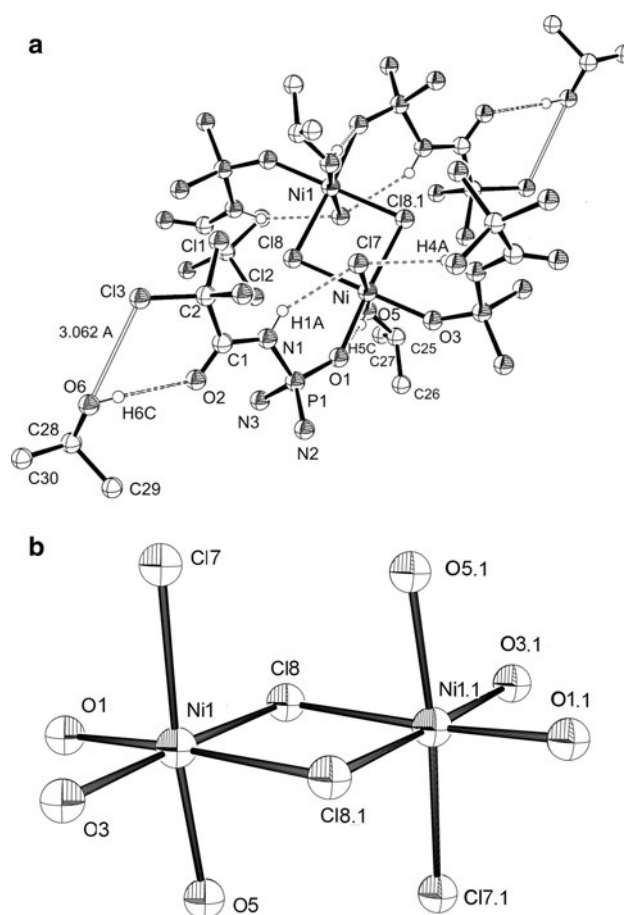
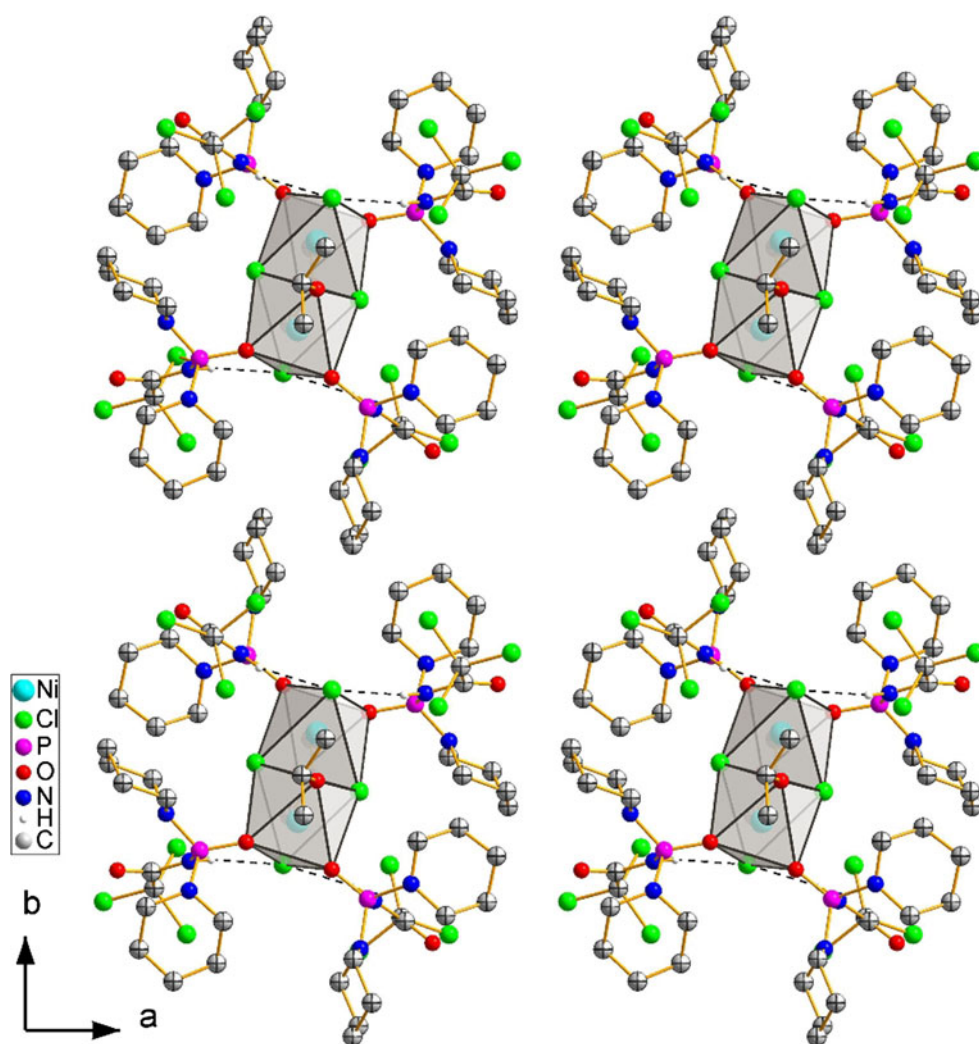


Fig. 2 **a** Structural representation of **2** with the atom numbering scheme and 30 % probability thermal ellipsoids; piperidine rings are omitted for clarity. **b** The coordination environment of Ni^{II} in **2**

and 90.84(3)°, respectively. The other angles are also close to the ideal octahedral angles (Table 3).

The single-crystal X-ray data reveal both phosphorus–oxygen bond elongation and phosphorus–nitrogen bond shortening upon coordination of HL (values are given in the Table 3). This can be caused by the displacement of electron density from the ligand phosphoryl oxygen to the nickel center. The coordination also results in some changes in valence angles. Specifically, angles N2,3–P1–N1, C1–N1–P1 and O2–C1–N1 are increased (see Table 3). The carbonyl carbon atom and the amide nitrogen atom have sp² character, and the HL ligands preserve their anti conformation under coordination. The hydrogen atoms H1A and H4A of the amide groups of the HL ligands take part in hydrogen bond formation with the axial chlorine atoms Cl7 (Figs. 2a, 3). Also, there are hydrogen bonds between H5C of the isopro-

Fig. 3 Crystal packing of **2** along the *c* axis, displaying the hydrogen-bonding interactions. Hydrogen atoms of piperidine rings are omitted for clarity



panol hydroxyl group and the carbonyl oxygen atom O2 (Table 4).

Conclusion

New dimeric 3D metal coordination compounds with a carbacylamidophosphate-type ligand were obtained in the crystalline state. The coordination of HL with M^{II} atoms by its phosphoryl oxygen was confirmed by the shift of $\nu(\text{P}=\text{O})$ and $\nu(\text{C}=\text{O})$ stretching vibrations in the IR spectra of the complexes compared to the free ligand HL. The nickel complex was structurally characterized; the metal is six-coordinated by three chloride ligands, two oxygen atoms of HL phosphoryl groups and one oxygen atom from an isopropanol ligand. The monodentate neutral coordination mode of a CAPH ligand to 3D metals contrasts with previously reported structures and has not been observed previously.

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