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# Novel tetranuclear cubane-like Co(II) complexes involving chelate phosphoramide ligands

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#### Abstract

Interaction of cobalt(II) ions and sodium carbacylamidophosphates Na(L) (HL = PhC(O)NHP(O)(NR<sub>2</sub>)<sub>2</sub>; where NR<sub>2</sub> are morpholyl, HL<sup>1</sup>; NMe<sub>2</sub>, HL<sup>2</sup>; NEt<sub>2</sub>, HL<sup>3</sup>) in methanol solution afforded polynuclear alkoxo complexes  $[Co_4\{L^1\}_3(OCH_3)_4(OH)(H_2O)_5 \cdot 3H_2O]$  **1** and  $[Co_4\{L\}_4(OCH_3)_4(CH_3OH)_4]$  (L = L<sup>2</sup> **2**, L<sup>3</sup> **3**). Data of spectral and TGA studies are presented. Coordination compounds **1** and **3** have been characterized by means of X-ray diffraction. Both the structures consist of tetranuclear cubane alkoxo clusters with methoxide ions bridging three metal centers (Co-O 2.068(3)-2.093(4) Å) and phosphorylic ligands coordinated in a bidentatechelate fashion via the carbonyl oxygen atoms (Co-O 1: 2.050(2); **3**: 2.031(4) Å) and the phosphoryl groups (2.093(2) and 2.106(4) Å). Isolation of these cubane alkoxo complexes is an important proof for close resemblance in behavior of carbacylamidophosphate systems and  $\beta$ -diketonates towards transition metal ions. © 2002 Elsevier Science Ltd. All rights reserved.

Keywords: Cobalt(II); Cubane complexes; Carbacylamidophosphates; X-ray data

## 1. Introduction

The cubane-like structure of tetranuclear metal complexes is a characteristic motif in coordination chemistry of different metal ions. It may dominate assembly of relatively simple compounds like thallium alkoxides [1] as well as of complicated molecular and supramolecular architectures [2]. This structural motif with close spatial alignment of metal centers is important also in the chemistry of transition metals where it may have an essential impact considering possible magnetic and catalytical properties of the compounds [3,4]. The structural and magnetochemical features of numerous Co(II) and Co(III) based cubane molecules have been reported [5–7]. Forming the complete or incomplete cubane complexes in solution is competitive for Co(II) complexes just as for Co(III). The cubane structure is also common in the realm of metal diketonates, recent examples include series of cobalt(II) and nickel (II) compounds [8-11]. We were interested in the generation of the cubane structure for new transition metal derivatives involving structural analogs of  $\beta$ -diketones. Such species may allow further functionalization of the metal-organic core, and phosphazo analogs of β-diketones are of special interest in this context: these Nacylated phosphoramides bear a close structural resemblance to the  $\beta$ -diketone frame, they usually act as effective chelating groups, and stoichiometry, structure and properties of coordination compounds thereof may be clearly related with known types of metal diketonates.

In this contribution we report on the engineering of a tetrameric cubane structure involving Co(II) ions and three chelating phosphoramide ligands.

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## 2. Experimental

2.1. Preparation of  $Co_4L_{4-n}(\mu_3-OCH3)_4(OH)_n \cdot mSolv$ (Solv =  $H_2O$  or  $CH_3OH$ )

Dimorpholido-*N*-benzoylamidophosphate  $HL^1$ , *N*,*N'*-tetramethyl-*N"*-benzoyltriamido-phosphate  $HL^2$ *N*,*N'*-tetraethyl-*N"*-benzoyltriamidophosphate  $HL^3$ and their sodium salts were obtained and identified according to [12,13]. 

## 2.2. Methods

IR spectra of the compounds were recorded on an UR-10 spectrometer in the range 400-4000 cm<sup>-1</sup> (samples as KBr pellets). Differential thermal analyses were carried out in the temperature range 20-500 °C



Coordination compounds of cobalt(II) were synthesized reacting the metal chloride and sodium salts of the ligands in methanol solution:

$$4\text{CoCl}_2 \cdot 6\text{H}_2\text{O} + 8\text{NaL}$$

$$\overset{\text{CH}_3\text{OH}}{\rightarrow}_{-\text{NaCl}} \text{Co}_4\text{L}_{4-n}(\mu_3\text{OCH}_3)_4(\text{OH})_n \cdot m\text{Solv} + 4\text{HL}$$

where  $L^- = (L^1)^-$ ,  $(L^2)^-$ ,  $(L^3)^-$ .

In a typical synthesis, CoCl<sub>2</sub>·6H<sub>2</sub>O (0.238 g, 1 mmol) was dissolved in methanol (15 ml) and combined with the solution of NaL<sup>1</sup> (0.722 g, 2 mmol) in 10 ml methanol. The mixture was allowed to stand in a vacuum desiccator over CaCl<sub>2</sub>. After a while the pinkviolet crystals of the complex  $[Co_4 \{L^1\}_3(OCH_3)_4(OH)]$ - $(H_2O)_5 \cdot 3H_2O$ ] 1 precipitated from the solution in the mixture along with neutral ligand HL<sup>1</sup>. The latter was separated out by washing the product with methanol and identified according to IR spectra. Crystals of the complex were additionally washed with cool acetone, and dried over CaCl<sub>2</sub> (yield: 0.248 g, 65.6%). Complexes  $[Co_4 \{L\}_4 (OCH_3)_4 (CH_3OH)_4]$  (L = L<sup>2</sup> 2, L<sup>3</sup> 3) were prepared in the same manner. Single crystals of 1 and 3 were prepared by slow crystallization from acetonemethanol mixture.

The complexes are unstable in air and decompose under long-term storage. They were soluble in nonpolar aprotic solvents, less soluble in acetone, isopropanol and ethyl alcohol.

The compositions of the compounds were established on the basis of the elemental analysis. *Anal.* for **1**. Found: C 37.53, H 5.56, N 7.75. Calc. for  $C_{49}H_{92}N_9Co_4O_{28}P_3$ : C 38.32, H 5.8, N 8.2%. *Anal.* for **2**. Found: C 41.67, H 6.78, N 10.01. Calc. for using a Q-1500 'Quasi' thermoanalyzer at a heating rate of 5 °C min<sup>-1</sup>. Electronic diffuse reflectance spectra were recorded on a SPECORD M-40 (Carl Zeiss, Jena) UV–Vis spectrometer.

X-ray diffraction data for 1 were collected using an Enraf-Nonius CAD-4 diffractometer. For 3 crystallographic measurements were made using a SMART CCD area-detector diffractometer (Siemens). Table 2 lists the cell parameters and details of data acquisition and structure refinement. Selected geometrical parameters of 1 and 3 are listed in Table 3. The structures were solved by direct methods and refined by full-matrix least-square methods using programs SHELXS-86 and SHELXL-93 [14,15]. All the non-hydrogen atoms were refined anisotropically. The hydrogen atoms were localized by difference Fourier techniques without further refinement.

Table 1

Main IR absorption bands and their assignments for carbacylamido-phosphate compounds,  $\rm cm^{-1}$ 

Compound	v(NH)	ν(C= Ο)	v(C- N)	ν(P= Ο)
HL <sup>1</sup> NaL <sup>1</sup> $[Co_4{L^1}_3(OCH_3)_4(OH)(H_2O)_5 \cdot$ 3H_O]	3100	1685 1545 1530	1440 1385 1380	1200 1115 1110
$HL^{2}$ NaL <sup>2</sup> $[Co_{4}\{L^{2}\}_{4}(OCH_{3})_{4}(CH_{3}OH)_{4}]$	3070	1680 1595 1535	1460 1380 1380	1280 1130 1085
HL <sup>3</sup> NaL <sup>3</sup> [Co <sub>4</sub> {L <sup>3</sup> } <sub>4</sub> (OCH <sub>3</sub> ) <sub>4</sub> (CH <sub>3</sub> OH) <sub>4</sub> ]	3070	1675 1535 1530	1450 1380 1380	1280 1120 1090

Table 2 Crystal data and structure refinement for **1** and **3** 

	1	3
Empirical formula	C <sub>49</sub> H <sub>92</sub> Co <sub>4</sub> N <sub>9</sub> O <sub>25</sub> P <sub>3</sub>	C <sub>68</sub> H <sub>128</sub> Co <sub>4</sub> N <sub>12</sub> O <sub>16</sub> P <sub>4</sub>
Temperature (K)	293	223
Wavelength (Å)	0.71073	0.71073
Crystal system	rhombohedral	tetragonal
Space group	$R\overline{3}$	$P\overline{4}2_1c$
Unit cell dimensions		
a (Å)	21.604(3)	16.921(2)
c (Å)	26.014(5)	15.404(3)
$V(Å^3)$	10515(3)	4410.5(11)
Z	6	2
Crystal size (mm)	$0.25 \times 0.25 \times 0.30$	0.2  imes 0.2  imes 0.25
$2\theta_{\rm max}$ (°)	50.1	51.4
Reflections collected	5178	16341
Independent reflections	3449	4059
Reflections observed ( > $2\sigma$ )	2488	2490
Data used/parameters refined	3449/271	4059/266
Goodness-of-fit on $F^2$	0.998	1.050
$R_1, wR_2$ (observed)	0.037, 0.090	0.057, 0.106
$R_1$ , $wR_2$ (all data)	0.068, 0.101	0.121, 0.142
Largest difference peak and hole (e $Å^{-3}$ )	0.52, -0.39	0.48, -0.43

## 3. Results and discussion

## 3.1. Spectroscopy

The IR spectra of the coordination compounds have a number of common features (Table 1). The IR bands in the region 3070-3100 cm<sup>-1</sup>, observed in spectra of the neutral carbacylamidophosphates, may be ascribed to v(NH) vibrations of the amide protons. These bands are absent in the spectra of the sodium and cobalt(II) carbacylamidophosphates 1-3, which is evidence for ligand coordination in the deprotonated form. The spectra reveal significantly lower frequencies of v(PO)as may be compared with those for the sodium salts  $(\Delta v(PO) = 45 \text{ and } 30 \text{ cm}^{-1} \text{ for compounds } 2 \text{ and } 3$ , respectively), reflecting coordination via the phosphoryl oxygen atom [8,9]. In the case of compound 1, the position of the v(PO) band could not be related identically. The v(CO) values in spectra of the complexes are shifted to lower frequencies  $(\Delta v(CO) = 5-60)$  $cm^{-1}$ ). These facts suggest a bidentate chelate coordination of the anionic ligands  $L^{-}$  via the oxygen atoms of phosphoryl and carbonyl groups.

UV-Vis absorption spectra reflect the octahedral environment the cobalt(II) ions in 1-3 [16]. The bands related to the electron transition  ${}^{4}T_{1g}(P) \rightarrow {}^{4}T_{1g}$  are observed at 18000, 19000 and 21000 cm<sup>-1</sup> for 1, 2 and 3, respectively. The transition  ${}^{4}A_{2g} \rightarrow {}^{4}T_{1g}$  is very weak and appears as a shoulder.

Table 3			
Selected bond	lengths (Å) and	angles (°) fo	r 1 and 3

1		3	
Bond lengths			
Co(1)-O(2)	2.050(2)	Co(1)-O(2)	2.031(4)
$Co(1) - O(7)^{a}$	2.083(2)	$Co(1) - O(3)^{a}$	2.068(3)
Co(1)-O(6)	2.086(2)	$Co(1)-O(3)^{b}$	2.084(3)
Co(1)-O(7)	2.092(2)	Co(1)-O(3)	2.091(4)
Co(1)-O(l)	2.093(2)	Co(1)-O(1)	2.106(4)
Co(1)-O(5)	2.161(2)	Co(1)-O(4)	2.204(4)
Bond angles			
P(1) - O(1)	1.509(2)	P(1)–O(1)	1.523(4)
P(1) - N(1)	1.617(3)	P(1) - N(1)	1.625(5)
O(2) - C(1)	1.266(4)	O(2) - C(1)	1.264(7)
N(1)-C(1)	1.314(5)	N(1)-C(1)	1.294(7)
$O(2)-Co(1)-O(7)^{a}$	90.47(8)	$O(2)-Co(1)-O(3)^{a}$	176.23(14)
$O(7)^{a} - Co(1) - O(6)$	83.16(9)	$O(2)-Co(1)-O(3)^{b}$	93.55(14)
O(2)-Co(1)-O(7)	93.00(8)	O(2)-Co(1)-O(3)	98.1(2)
O(2)-Co(1)-O(1)	91.55(9)	$O(3)^{a}-Co(1)-O(3)$	82.73(14)
$O(7)^{a} - Co(1) - O(1)$	173.61(8)	O(2)-Co(1)-O(1)	89.9(2)
$O(7)^{a}-Co(1)-O(5)$	89.13(8)	$O(3)^{a}-Co(1)-O(1)$	93.76(15)
O(1)-Co(1)-O(5)	96.80(8)	$O(3)^{b}-Co(1)-O(1)$	175.09(13)
O(1) - P(1) - N(1)	118.37(14)	O(3)-Co(1)-O(1)	94.06(14)
C(1)-N(1)-P(1)	127.7(2)	O(2)-Co(1)-O(4)	93.1(2)
$Co(1)^{b}-O(7)-Co(1)$	96.46(9)	$O(3)^{a} - Co(1) - O(4)$	85.58(14)
		O(3)-Co(1)-O(4)	166.36(13)
		O(1)-Co(1)-O(4)	93.70(15)
		O(1) - P(1) - N(1)	118.7(2)
		C(1)-N(1)-P(1)	124.7(4)
		$Co(1)^{c}-O(3)-Co(1)^{b}$	96.74(14)
		$Co(1)^{c}-O(3)-Co(1)$	96.52(14)
		$Co(1)^{b}-O(3)-Co(1)$	97.7(2)

Symmetry transformations used to generate equivalent atoms for 1: (a) 1-x+y, 1-x, z; (b) 1-y, x-y, z; for 3: (a) 1-y, x, -z; (b) 1-x, 1-y, z; (c) -y, 1-x, -z.

### 3.2. Thermogravimetry

The thermal behavior of the cobalt complexes 2 and 3 is very similar. According to the TGA data, these complexes are stable up to 100 °C. In the temperature range 100 °C-290 °C, the thermoanalytical curves indicate two endothermic effects. The first of them (115 °C) is connected with the elimination of four molecules of methanol. This relatively elevated temperature of the methanol molecule removal indirectly indicates their disposition in the internal coordination sphere of the metal ions. The other endothermic stage corresponds to the beginning of the decomposition of the complex. It takes place at the same temperature for the both compounds, namely 265 °C. Full decomposition of the complex is accompanied by its vigorous exothermic oxidation at 285 °C. It is necessary to note that a difference in the temperatures of the maximal onset of the processes is not essential and it is equal to within 20 °C. This fact shows the important role of the methanol molecules in the formation of the crystal lattice of the cobalt complexes 2 and 3. The similar character of the decomposition of these compounds and the same temperature interval of thermal effects may suggest a similarity of their structures.

In the case of complex 1, the TGA results are rather different. The thermoanalytical curves also reflect two endothermic effects at 130 and 200 °C, and an exothermic one at 335 °C. The first effect is connected with the elimination of three water molecules from the external sphere of the complex. The compound decomposes at 200 °C. The fact that the further dehydration of 1 is accompanied by its decomposition is evidence of the involvement of the other five water molecules in the internal coordination sphere and their strong bonding with the central atoms. The full exothermic decomposition of the complex takes place at 335 °C.

#### 3.3. Description of the structures

We succeeded in confirming the conclusions about the structure of the obtained compounds based on IR and UV–Vis spectroscopy data by the results of full X-ray analysis of compounds 1 and 3.

The solid-state structure of **3** contains discrete tetrameric molecules.  $[Co_4 \{L^3\}_4(\mu_3\text{-}OCH_3)_4(CH_3OH)_4]$ , each octahedral Co(II) ion being bound to three  $\mu_3\text{-}OMe^$ ligands, one chelating carbacylphosphortriamide ligand (L<sup>3</sup>), and one terminal MeOH ligand. Concerning the composition of the compound and the results of spectral and TGA studies, the same structure may also be ascribed for compound **2**. The molecule occupies a special position on an inversion fourfold axis and has virtual  $D_{2d}$  symmetry.

For both structures, the tetrameric skeletons are formed by four cobalt atoms and four oxygen atoms of methoxy groups. The octahedral environment for each cobalt atom in **3** and for three of four cobalt atoms in **1** comprises three oxygen atoms of methoxy groups, carbonylic and phosphoryl oxygen atoms of the chelate anionic ligands and an oxygen atom of an alcohol molecule in structure **3** (Fig. 1). The fourth cobalt atom in structure **1** is situated on a threefold axis and is connected with three oxygen atoms of methoxy groups, oxygen atoms of two water molecules and one  $OH^$ group. These hydro and hydroxo ligands are structurally equivalent and all are related by a threefold axis.

The cubane-like frameworks in the present cases are slightly distorted, the angles O–Co–O are about 5° smaller than 90°, while the angles Co–O–Co are approximately 97°. The Co–O(2) bond with the carbonyl oxygen atom is the shortest in the cobalt environment in both structures (1: 2.050(2) and 3: 2.031(4) Å). The Co–O(CH<sub>3</sub>) bond lengths are typical for  $\mu_2$ - and  $\mu_3$ -OMe groups [17] and are in the range of 2.068(3)–2.093(4) Å for both structures. The distances to the phosphoryl oxygen atoms, Co–O(1) in 1 and 3, are 2.093 (2), and 2.106(4) Å, respectively. The Co–O(4) bond with a coordinated water molecule (2.161(2) Å) for



Fig. 1. Molecular structure of the cobalt cubane alkoxo complexes: A- $Co_4\{L^1\}_3(OCH_3)_4(OH)(H_2O)_5 \cdot 3H_2O$  **1**, hydrogen atoms oxamethylene linkage of morpholine cycles and two of the three phenyl groups are omitted; B- $Co_4\{L^3\}_4(OCH_3)_4(CH_3OH)_4$  **3**, hydrogen atoms, ethyl and methyl fragments of the phosphorylic, metoxy and methoxo ligands are omitted for clarity, and only one of the four chelate frames is shown. Thermal ellipsoids are at 40% probability level and possible hydrogen bonding is shown as dotted lines.

1 and the Co–O bond with a coordinated methanol oxygen atom (2.204(4) Å) in the case of compound 3 are the longest.

The ligand deprotonation leads to the appearance of  $\pi$ -conjugation in the chelate ring of the compounds, which may be illustrated by the changes of the bond lengths. Thus the distances P=O (1: 1.509; 3: 1.523 Å) and C=O (1: 1.266; 3: 1.264 Å) both appeared slightly longer with respect to those for the non-coordinated molecules HL<sup>1</sup> and HL<sup>3</sup> [12,18]. The increase of the bond order for P–N and C–N caused by the increase of the  $\pi$ -bonding leads to the 0.05–0.09 Å shortening of these bond lengths in the complexes with respect to that observed for HL<sup>1</sup> and HL<sup>3</sup>. The OPNCO chelate

fragment in both structures are planar to within 0.1 Å and the deviation of the metal atoms from these planes does not exceed 0.5 Å.



The phosphorus atoms have a distorted tetrahedral environment. The atoms forming the angles O(1)-P(1)-N(1) (118.37°, 118.7°) for both cases have the largest deviations because they are involved in the  $\pi$ -conjugation. All the other angles in the **3** are nearly tetrahedral.

For each type of the cubane molecules the OH and O counterparts are involved in extensive intramolecular hydrogen bonding. Thus, in **3** each MeOH hydroxy group forms an hydrogen bond with the phosphoryl oxygen atom of carbacylphosporamide ligand coordinated to a neighboring Co ion (O···O 2.78 Å). A similar pattern of intramolecular hydrogen bonding has also been observed in other alkoxides cubane complexes containing Co(II), Co(III) and Ni(II) and coordinated alcohol molecules [7,19].

## 4. Conclusions

Isolation of cobalt(II) tetranuclear cubane alkoxo complexes with carbacylamidophosphates is an important proof for close resemblance of behaviour of these ligand systems and  $\beta$ -diketonates towards transition metal ions. In the present case, the remarkable stability of (Co{L})<sub>4</sub>(OCH<sub>3</sub>)<sub>4</sub> or (Co{L})<sub>3</sub>(CoOH)(OCH<sub>3</sub>)<sub>4</sub> cores dominates the pathways of interaction in the system  $M^{2+} -L^{-}$  and corresponding cubane molecules appear to be a most stable and common product. This effective stabilization of a cubane metal–alkoxide cluster with a carbacylamidophosphate anion may allow the preparation of novel functionalized condensed metal–alkoxide complexes.

## 5. Supplementary material

Crystallographic data for the structural analysis have been deposited at the Cambridge Crystallographic Data Center, CCDC numbers CSD-142056 (1) and CSD-166804 (3). Copies of this information may be obtained free of charge from the Director CCDC, 12 Union Road, Cambridge, CB2 1EZ, UK (fax: +44-1223-336033; e-mail: deposit@ccdc.cam.ac.uk or www: http://www.ccdc.cam.ac.uk).

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