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Polynuclear Co(II) Myrtenates with 2,4-Lutidine

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Abstract—The reaction of cobalt(II) chloride with potassium myrtenate (KMyr) followed by addition of 2,3-lutidine in ethanol gives the trinuclear compound $[Co_3(Myr)_6(2,4\text{-}Lut)_2]$ (**I**). The central cobalt(II) atom is linked to each of the two other metal atoms by three myrtenic acid anions. The reaction of a solution of compound **I** with lithium myrtenate (LiMyr) results in the formation of tetranuclear heterometallic complex $[Li_2Co_2(Myr)_6(2,4\text{-}Lut)_2]$ (**II**). Compound **II** is composed of two binuclear $\{LiCo(Myr)_3(2,4\text{-}Lut)\}$ moieties, in which the lithium(I) atoms are linked to cobalt(II) atoms by bridging myrtenate anions. The lithium atoms are connected by two oxygen atoms of acid anions. Compounds **I** and **II** were characterized by X-ray diffraction (CIF files CCDC nos. 1898096 (**I**), 1898097 (**II**)).

Keywords: cobalt(II) carboxylate complexes, myrtenic acid, X-ray diffraction

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INTRODUCTION

Among numerous polynuclear carboxylates containing divalent 3*d* metal atoms, the simplest and the most abundant ones are binuclear compounds with the $\{M(\mu\text{-}O_2CR)_4M\}$ metal moiety [1–6] and linear trinuclear compounds with the $\{M(\mu\text{-}O_2CR)_3M(\mu\text{-}O_2CR)_3M\}$ metal moiety [7–16]. These metal moieties are quite typical for layered and framework polymers with dicarboxylic and polycarboxylic acids [17–23]. These systems are actively studied by various researchers for their catalytic activity, magnetic or optical properties, or biological activity, depending on the nature of metal centers [1, 4, 13–15, 18]. Molecular systems are fabricated using adducts of these simple bi- and trinuclear moieties with various organic neutral donor molecules. The structure of the metal core of these systems can be controlled by various methods depending on the geometric and electronic properties of apical organic ligands [1–3]. One more possible way to affect the structure of the metal carboxylate core is to change the nature of the substituent at the carboxyl group; this effect is approximately the same for Co(II), Fe(II), Mn(II), and Zn(II) derivatives [7, 12]. The difficulty of accurate prediction of the structures of complexes resulting from varying the substituent in the carboxylate anion and the possibility of obtaining two types of complexes (with bi- and trinuclear metal carboxylate core) for two types of carboxylate anions indicate that the thermodynamic stabilities of these bi- and trinuclear complexes are often similar and that some of the compounds can be quite long-lived metastable products, in particular, metastable isomers. This can be illustrated by the unique example of coexistence of binuclear $[Zn_2(O_2CPh)_4(Py)_2]$ and trinuclear $[Zn_3(O_2CPh)_6(Py)_2]$ molecules in the crystal [24]. The effect of substituent at the carboxylate group can be demonstrated by the example of naphthoate complexes: zinc(II) complex with α-naphthoic acid (α-HNaph) and 2,3-lutidine (2,3-Lut), $[Zn_2(\alpha\text{-}Naph)_4(2,3\text{-}Lut)_2]$, is binuclear [7], but replacement of the acid by β-naphthoic (β-HNaph) promotes the formation of a linear trinuclear complex, $[Zn_3(\beta\text{-}Naph)_6(2,3\text{-}Lut)_2]$ [16].

An obvious analogy with benzoate complexes is observed for complexes of carboxylic acids with an unsaturated substituent, such as crotonic acid anion. For zinc(II), as in the case of benzoate complexes, a binuclear [25, 26] and trinuclear complexes were obtained [27, 28] with quinoline.

In this paper, we pursued the studies on the effect of substituent in the carboxylic acid. The paper addresses the formation of the metal carboxylate core based on the myrtenate anion (Myr^-), which is of interest due to the presence of the C=C double bond able to participate in π–π-conjugation with the carboxylate group, like that in the crotonate anion. However, in the case of myrtenate anion, the double bonded carbon atoms belong to a bulky bicyclic moiety.

[†] Deceased.

Table 1. Crystallographic parameters and structure refinement details for **I** and **II**

Parameter	Value	
	I	**II**
Molecular formula	$C_{74}H_{96}N_2O_{12}Co_3$	$C_{78}H_{102}N_4O_{12}Co_2Li_2$
M	1382.32	1419.38
T, K	184(2)	184(2)
System	Triclinic	Monoclinic
Space group	$P\bar{1}$	$P2_1$
a, Å	11.619(3)	10.8075(14)
b, Å	12.325(3)	18.449(2)
c, Å	13.635(3)	18.927(3)
α, deg	111.380(3)	90
β, deg	100.092(4)	96.460(2)
γ, deg	92.950(4)	90
V, Å^3	1775.9(7)	3749.8(8)
Z	1	2
ρ(calcd.), g/cm^3	1.293	1.257
μ, mm^{-1}	0.753	0.504
θ_{max}, deg	29.13	28.28
T_{min}/T_{max}	0.929/0.956	0.942/0.951
Number of measured reflections	19954	28286
Number of unique reflections	9493	15479
Number of reflections with $I > 2\sigma(I)$	5756	11664
R_{int}	0.0568	0.058
Number of refined parameters	648	883
GOOF	1.39	1.07
R_1 ($I > 2\sigma(I)$)	0.124	0.057
wR_2 ($I > 2\sigma(I)$)	0.378	0.129
Residual electron density (min/max), e/Å^3	–3.833/1.003	–0.436/0.682

EXPERIMENTAL

Gas liquid chromatography (GLC) was carried out on a SHIMADZU GC-2010AF chromatograph with an HP1 column, flame ionization detector, and helium as the carrier gas. The IR spectrum of myrtenic acid was recorded in thin film on an IR Prestige 21 Shimadzu instrument. NMR spectra were measured on a Bruker AVANCE-II-300 instrument operating at 300 MHz (^{1}H) and 75 MHz (^{13}C) in $CDCl_3$ using standard Bruker pulse software for 1D and 2D experiments. Optical rotation was measured on a P3002RS automatic polarimeter (Kruss, Germany). The IR spectra of the complexes were recorded on a Perkin Elmer Spectrum 65 spectrometer in the ATR mode in the 4000–400 cm^{-1} range. Elemental analysis was done on an EA-3000 automatic C, H, N, S-analyzer (Euro Vector).

Single crystal X-ray diffraction study of complexes **I** and **II** was performed on a Bruker Apex II diffractometer (CCD-detector, MoK_α radiation at λ = 0.71073 Å, graphite monochromator) [29]. The structures were solved by direct methods and refined in the full-matrix anisotropic approximation for all non-hydrogen atoms. The hydrogen atoms attached to the carbon atoms of organic ligands were generated geometrically and refined in the riding model. The calculations were carried out using the SHELX-97 software [30]. The crystallographic parameters of **I** and **II** are summarized in Table 1.

The full set of X-ray diffraction data is deposited with the Cambridge Crystallographic Data Centre (nos. 1898096 (**I**), 1898097 (**II**); deposit@ccdc.cam.ac.uk or http://www.ccdc.cam.ac.uk/data_request/cif).

Column chromatography was performed with silica gel 60 (70–230 mesh; Alfa-Aesar). Thin layer chromatography (TLC) was done on Sorbfil plates with reagent grade hexane and analytical grade diethyl ether as eluents and visualization by a 10% aqueous solution of $KMnO_4$. Myrtenic acid was prepared using 1(*R*)(–)-myrtenol (Aldrich), $[\alpha]_D$ –47.1 (*c* 1.0 EtOH), reagent grade pyridine, reagent grade Na_2CO_3, and reagent grade $MgSO_4$. Chlorine dioxide (ClO_2) as an aqueous solution (6–7 g/L) was provided by the Mondi Syktyvkar Pulp and Paper Mill.

Synthesis of HMmyr. Chlorine dioxide was passed for 8–10 h through a stirred solution of (–)-myrtenol (5 g, 0.033 mol) in pyridine (50 mL) at 35–40°C. The reaction was monitored by TLC and GLC. The reaction mixture was cooled, the solvent was removed under reduced pressure, the residue was treated with a 5% aqueous solution of Na_2CO_3 (100 mL), and the mixture was extracted with Et_2O to separate neutral compounds. The aqueous phase was acidified with 10% aqueous H_2SO_4 (reagent grade) and thoroughly extracted with Et_2O. The Et_2O extract was washed with saturated solution of NaCl and dried over anhydrous $MgSO_4$. After removal of the solvent, the yield of myrtenic acid as a thick dark oil was 2.0–2.2 g (40–44% based on myrtenol). Column chromatography on SiO_2 (hexane–diethyl ether (10 : 1)) gave a colorless oil, which gradually crystallized. R_f 0.56 (hexane–diethyl ether (3 : 2)), $[\alpha]_D$ –42.4 (*c* 0.4 EtOH). IR (ν, cm^{-1}): 3100–2500 (br., ν, OH), 1690 (C=O), 1630 (C=C), 1430, 1280 (ν, C–O), 1080, 960 (OH), 790, 760, 720. ^{13}C NMR (δ, ppm): 171.8 (C10), 139.8 (C2), 139.3 (C3), 40.8 (C1), 40.2 (C5), 37.6 (C6), 32.3 (C4), 31.2 (C7), 25.8 (C8), 20.9 (C9). ^1H NMR (δ, ppm): 0.80 (s, 3H, C8), 1.12 (d, 1H, *J* = 9.1, C7β), 1.34 (s, 3H, C9), 2.13 (ddd, 1H, C5, J^1 = 5.7, J^2 = 2.9, J^3 = 1.5), 2.45 (dd, 1H, $C4_\beta$, J^1 = 13.5, J^2 = 2.9), 2.46 (d, 1H, $C4_\alpha$, *J* = 13.5) 2.48 (d, 1H, *J* = 9.1, C7α), 2.79 (td, 1H, J^1 = 1.5, J^2 = 5.7, C1), 6.99 (m, 1H, *J* = 1.5, C3), 12.10 (br, 1H, COOH).

Complexes **I** and **II** were synthesized in air using ethyl alcohol (96%), and acetonitrile (99%) and commercially available chemicals: $CoCl_2 \cdot 6H_2O$, 2,4-lutidine (>99%), KOH, and LiOH (all analytical grade). The coordination compounds were prepared using myrtenic acid with different optical purities (–37° to –42.4°). The above-described synthesis of the acid refers to the optimal procedure using optically pure initial myrtenol.

Synthesis of $[Co_3(Myr)_6(2,4\text{-}Lut)_2]$ (I). Weighed portions of KOH (0.047 g, 8.4 mmol) and HMyr (0.1395 g, 8.4 mmol) were dissolved in ethanol (30 mL), then $CoCl_2 \cdot 6H_2O$ (0.1 g, 4.2 mmol) and 2,4-lutidine (0.064 mL, 6.3 mmol) were added. The resulting violet-colored reaction mixture was stirred for 30 min, filtered to remove the precipitated KCl, and kept at room temperature. The violet crystals suitable for X-ray diffraction, which formed after 24 h, were separated from the mother liquor by decantation, washed with cold EtOH, and dried in air. The yield of **I** was 0.110 g (57% based on the initial amount of cobalt(II)).

For $C_{74}H_{96}N_2O_{12}Co_3$

Anal. calcd., %	C, 64.30	H, 7.00	N, 2.03
Found, %	C, 64.45	H, 7.01	N, 2.75

IR (ATR; ν, cm^{-1}): 2982 m, 2915 m, 2881 m, 2822 w, 1680 w br, 1631 m, 1622 m, 1578 s, 1516 m, 1494 m, 1400 s, 1387 s, 1365 m, 1330 s, 1321 s, 1300 m, 1266 m, 1238 m, 1216 m, 1202 m, 1177 m, 1150 m, 1123 w, 1096 m, 1079 m, 1055 m, 1037 m, 1027 m, 966 m, 949 m, 926 m, 889 m, 847 m, 833 m, 807 m, 761 s, 734 m, 726 m, 633 m, 594 m, 539 m.

Synthesis of $[Li_2Co_2(Myr)_6(2,4\text{-}Lut)_2]$ (II). A solution containing LiOH (0.010 g, 4.2 mmol) and HMyr (0.070 g, 4.2 mmol) in ethanol (15 mL) was added to the reaction mixture obtained in the synthesis of **I** after KCl separation. The resulting violet solution was concentrated at 80°C to 20 mL, cooled to room temperature, and kept for 24 h. The violet crystals thus formed, suitable for X-ray diffraction, were washed with cold acetonitrile (–5°C) and dried in air. The yield of compound **II** was 0.128 g (43% based on the initial amount of cobalt(II)).

For $C_{78}H_{102}N_4O_{12}Li_2Co_2$

Anal. calcd., %	C, 66.00	H, 7.24	N, 3.95
Found, %	C, 65.48	H, 7.16	N, 4.34

IR (ATR; ν, cm^{-1}): 2982 m, 2935 m, 2910 m, 2883 m, 2823 w, 1634 m, 1622 m, 1590 s, 1554 s, 1491 m, 1443 m, 1395 s, 1366 m, 1333 m, 1315 s, 1297 m, 1265 m, 1235 w, 1220 m, 1200 m, 1175 m, 1149 w, 1102 w, 1091 m, 1079 m, 1057 m, 1026 m, 967 m, 958 m, 949 w, 923 w, 890 m, 858 w, 830 m, 823 m, 809 m, 766 s, 728 m, 632 m, 604 m, 592 m, 540 m.

RESULTS AND DISCUSSION

Myrtenic acid is a derivative of naturally abundant optically active bicyclic monoterpenes, α- and β-pinenes. Here it was prepared by oxidation of commercially available myrtenol with chlorine dioxide (industrial product used for pulp bleaching and wastewater treatment) in pyridine. The yield of the acid isolated via the water-soluble sodium salt and purified by column chromatography on silica gel was 35–37%.

We found that the reaction of cobalt(II) chloride with potassium myrtenate (prepared in situ by the reaction of myrtenic acid with potassium hydroxide) followed by addition of 2,3-lutidine (the excess does not influence the product yield) in ethanol afforded a

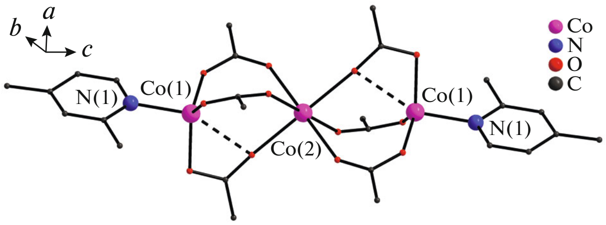

Fig. 1. Molecular structure of complex **I** (hydrogen atoms and cyclic moieties of the myrtenate anion are omitted).

bright violet solution, from which violet crystals of compound **I** were isolated afterwards. The reaction of a solution of compound **I** with lithium myrtenate in EtOH afforded a bright yellow solution, from which violet crystals of heterometallic complex **II** precipitated upon concentration.

According to X-ray diffraction data, complex **I** crystallizes in the triclinic space group $P\bar{1}$, with the inversion center coinciding with the central Co(2) atom (Fig. 1). The pairs of metal atoms (Co···Co, 3.8192(11) Å) of the trinuclear molecule are connected by three carboxyl groups. The central Co(2) atom has an octahedral environment composed of six O atoms of the bridging myrtenate anions (Co–O, 2.295(5)–2.322(5) Å). The Co(1) environment is a tetrahedron consisting of three myrtenate O atoms (Co–O, 1.927(5)–1.997(5) Å) and one 2,4-lutidine N atom (Co–N 2.085(5) Å). The Co(1) atom forms a contact with the second O atom of one bridging carboxyl group (Co(1)···O(6), 2.560(5) Å).

Complex **II** crystallizes in the monoclinic space group $P2_1$. The molecule of **II** (Fig. 2) is composed of two binuclear $\{LiCo(Myr)_3(2,4\text{-}Lut)\}$ moieties (Co···Li, 3.103(8) and 3.134(8) Å), in which Li atoms (Li···Li, 2.722(8) Å) are linked by O atoms of the bridging carboxylate groups, thus forming a zigzag-like Co–Li–Li–Co metal skeleton (CoLiLi angles, 122.0(3)° and 123.3(3)°). The geometry of the central Li_2O_2 group is close to a square (Li–O, 1.933(8)–1.976(8) Å; LiOLi, 91.5(3)°, 92.9(3)°; OLiO, 87.2(3)°, 88.3(3)°). The cobalt and lithium atoms are connected by the bridging carboxyl groups (Co–O, 1.931(3)–1.963(3) Å; Li–O, 1.868(8)–1.976(8) Å). The environment of each cobalt(II) atom is completed to a tetrahedron by coordination of the 2,4-lutidine N atom (Co–N, 2.067(4) and 2.078(4) Å).

The structure of complex **I** is similar to the structures of previously obtained complexes with aromatic acids and 2,3-lutidine [7, 8]. It could be suggested that π–π conjugation between the O–C=O and C=C groups of the substituent at the carboxyl group plays a substantial role in the formation of the trinuclear structure. Analysis of the structural data of trinuclear transition metal complexes demonstrated that in the case of 2-naphthoic anions, the relative position of these groups can vary from virtually coplanar to rotated (the OCC=C torsion angle varies from 5° to 73°) [7, 16]. In the case of complexes with 9-anthracenecarboxylate anions, no π–π conjugation is present (the OCC=C torsion angle varies from 44° to 90°), because of steric strain. For comparison, we analyzed the torsion angles in bi- and trinuclear complexes with sterically unhindered crotonate and benzoate anions; they were in the ranges of 0°–15° [31–39] and 1°–24° [8, 40–43], respectively. The OCC=C torsion angles for the newly synthesized compounds were 11.1(13)°–22.4(8)° for **I** and 2.61(4)°–17.57(3)° for **II**; thus, they differ little from the angles in the binuclear complexes with crotonate anions. This indicates that the free rotation of the bulky substituent in the myrtenate anion is unhindered.

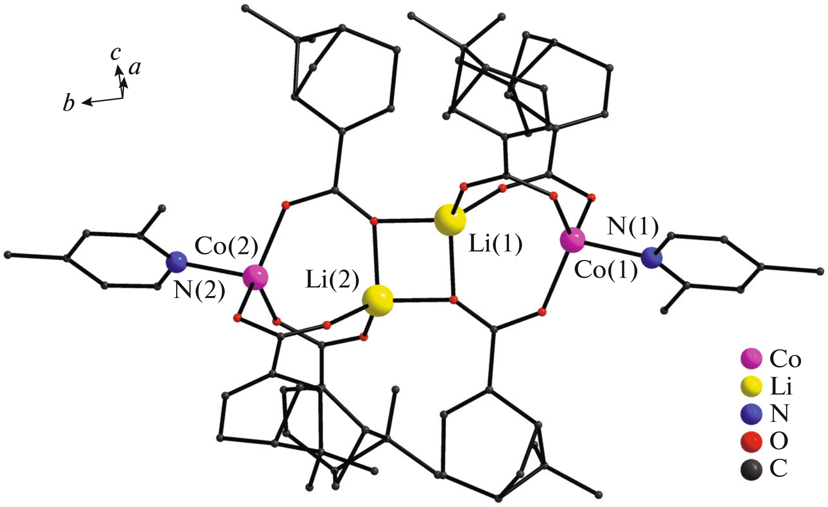

Fig. 2. Molecular structure of complex **II** (hydrogen atoms are omitted).

The formation of trinuclear complex **I** even with excess α-substituted pyridine implies that the complexation equilibrium is shifted towards the formation of a linear trinuclear metal carboxylate core. A similar situation was observed on going from benzoate to 2-naphthoate complexes: in the case of Co(II) benzoates with pyridine and its monodentate derivatives, both types of complexes can be obtained [8, 44, 45], while in the case of 2-naphthoates with excess 2,3-lutidine, only the linear trinuclear complex was isolated [7].

Thus, we have demonstrated that the myrtenate anion forms trinuclear complex **I** with cobalt(II) in the presence of 2,3-lutidine, while in the presence of lithium(I) ions, tetranuclear heterometallic Li–Co complex **II** is produced. Analysis of the torsion angles between the O–C=O and C=C groups of the substituent at the carboxyl showed that these angles for newly prepared complexes are close to those of the bi- and trinuclear complexes with sterically unhindered crotonate and benzoate anions, which suggests the absence of steric restrictions to the free rotation of the bulky substituent in the myrtenate anion. This should not hamper the formation of both the described trinuclear complex **I** and the binuclear complex, which could not be isolated as single crystals. The formation of Li–Co complex **II**, an analogue of the previously prepared Li–M complexes (M = Cu, Co, Ni, Zn) with pivalate, *tert*-butylacetate, and β-naphthoate anions [46], confirms that the structure and nature of the substituent at the carboxylate group has little effect on the metal core of the heterometallic 3*d* metal compounds.

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CONFLICT OF INTEREST

The authors declare that they have no conflicts of interest.

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