

COORDINATION COMPOUNDS

Synthesis and Molecular Structures of the $\text{Co}_3(\mu\text{-OOCPh})_4(\mu,\eta^2\text{-OOCPh})_2[\text{OC}(\text{Ph})\text{OHNET}_3]_2$ and $\text{Co}(\text{Hdmpz})_2(\text{OOCPh})_2$ Complexes (Hdmpz = 3,5-Dimethylpyrazole)

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Abstract—The product of the thermal reaction between cobalt acetate hydrate and benzoic acid reacts with a triethylamine excess to form the trinuclear complex $\text{Co}_3(\mu\text{-OOCPh})_4(\mu,\eta^2\text{-OOCPh})_2[\text{OC}(\text{Ph})\text{OHNET}_3]_2$, and its reaction with 3,5-dimethylpyrazole yields the mononuclear complex $\text{Co}(\text{Hdmpz})_2(\text{OOCPh})_2$. The compound structures are discussed on the basis of X-ray crystallographic data.

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The deprotonation behavior of 3,5-dimethylpyrazole (Hdmpz) and the peculiarities of the structures of the pyrazolate-bridged transition metal compounds formed are determined by the nature of the transition metal, the R substituent in the carboxylate anion, and the reaction conditions.

For example, we have previously shown that the reactions of binuclear pivalates $\text{M}_2(\mu\text{-OOCMe})_4(\text{NEt}_3)_2$, containing donor *tert*-butyl substituents in the carboxylate anion, with Hdmpz depends on the transition metal nature and leads to deprotonation of pyrazole at room temperature with formation of the pyrazolate-bridged dimers $\text{M}_2(\mu\text{-dmpz})_2(\text{Hdmpz})_2(\text{OOCBu}')_2$ ($\text{M} = \text{Zn}, \text{Co}$), binuclear complexes $\text{M}_2(\mu\text{-OOCBu}')_4(\text{Hdmpz})_2$ ($\text{M} = \text{Cu}, \text{Ni}$), or the mononuclear complex $\text{Cu}(\text{Hdmpz})_2(\text{OOCBu}')_2$ [1–6].

Note that the complexes obtained can be used to produce more complex polynuclear clusters and even metal-containing polymers with unsaturated bridging organic moieties and transition metal atoms [7–11].

In this work, reactions of cobalt benzoate, which contains an acceptor phenyl substituent in the carboxylate anion, with triethylamine and Hdmpz are considered in comparison with analogous reactions of cobalt pivalate and acetate containing donor *tert*-butyl and methyl substituents. It is well known that the strength of carboxylic acids changes as follows: $\text{PhCOOH} > \text{MeCOOH} > \text{Bu}'\text{COOH}$ [12].

EXPERIMENTAL

All procedures relating to the synthesis and isolation of complexes were carried out in a pure argon atmosphere with the use of dry solvents.

IR spectra were recorded as KBr pellets on a Nexus-Nicolet spectrophotometer in the range of 400–4000 cm^{-1} .

X-ray diffraction studies were performed by the standard procedure on an automated Bruker SMART Apex II diffractometer with a CCD detector (λMo , graphite monochromator, ω scan). The structures were solved with the use of the SHELXTL PLUS (PC version) and refined with the SHELXTL-97 program packages [13, 14]. Crystal data and refinement parameters are shown in Table 1. Atomic coordinates and selected geometric parameters are compiled in Tables 2–5.

Synthesis of $\text{Co}_3(\mu\text{-OOCPh})_4(\mu,\eta^2\text{-OOCPh})_2[\text{OC}(\text{Ph})\text{OHNET}_3]_2$ (1). To 1 g (4.01 mmol) of cobalt acetate tetrahydrate, 1.47 g (12.03 mmol) of benzoic acid in 20 mL of *o*-xylene was added. The mixture obtained was refluxed with stirring for 3 h. The suspension formed was cooled to room temperature, and a triethylamine excess (4 mL, 28.78 mmol) was added. The resulting solution was concentrated to 10 mL and kept at -15°C for a day. The violet crystals formed were isolated by decantation, washed with cold hexane, and dried in an argon flow. Yield, 2.11 g (78.3%).

For $\text{Co}_3\text{C}_{68}\text{H}_{72}\text{N}_2\text{O}_{16}$ anal. calcd (%): C, 60.22; H, 5.80. Found (%): C, 60.19; H, 5.77.

Table 1. Crystal data and refinement details for structures **1** and **2**

	1	2
Chemical formula	C ₆₈ H ₇₂ Co ₃ N ₂ O ₁₆	C ₂₄ H ₂₆ CoN ₄ O ₄
FW	1350.07	493.42
T, K	120(2)	150(2)
Color	Violet	Violet
Crystal system	Monoclinic	Monoclinic
Space group	<i>P</i> 2(1)/ <i>n</i>	<i>P</i> 2(1)/ <i>n</i>
Unit cell parameters (Å, deg)	<i>a</i> = 13.4012(15) <i>b</i> = 15.6336(17) <i>c</i> = 16.6327(18) α = 90 β = 108.837(2) γ = 90	<i>a</i> = 12.2366(5) <i>b</i> = 14.0892(6) <i>c</i> = 14.2885(6) α = 90 β = 95.461(1) γ = 90
<i>V</i> , Å ³	3298.1(6)	2452.21(18)
<i>Z</i>	2	4
ρ _{calcd} , g/cm ³	1.359	1.336
μ, mm ⁻¹	0.814	0.736
<i>F</i> (000)	1406	1028
Crystal dimensions, mm	0.14 × 0.12 × 0.10	0.16 × 0.14 × 0.12
θ scan range, deg	1.71–27.00	2.03–29.00
Reflection index range	−17 ≤ <i>h</i> ≤ 17, −19 ≤ <i>k</i> ≤ 19, −21 ≤ <i>l</i> ≤ 19	−16 ≤ <i>h</i> ≤ 16, −19 ≤ <i>k</i> ≤ 19, −17 ≤ <i>l</i> ≤ 19
Reflections measured	22 296	21 346
Independent reflections	7092 [<i>R</i> (int) = 0.0337]	6453 [<i>R</i> (int) = 0.0533]
GOOF	1.017	1.001
<i>R</i> [<i>I</i> > 2σ(<i>I</i>)]	<i>R</i> 1 = 0.0441, <i>wR</i> 2 = 0.1089	<i>R</i> 1 = 0.0330, <i>wR</i> 2 = 0.0818
<i>R</i> (for all reflections)	<i>R</i> 1 = 0.0654, <i>wR</i> 2 = 0.1227	<i>R</i> 1 = 0.0462, <i>wR</i> 2 = 0.0854
<i>R</i> (Residual electron density (max/min), e Å ^{−3})	0.755 and −0.687	0.393 and −0.293

IR (cm^{-1}): 3438 m, 3028 m, 2853 w, 2222 w, 1610 s, 1567 s, 1491 m, 1472 m, 1448 m, 1366 s, 1339 m, 1175 m, 1158 m, 1130 m, 1068 m, 1026 m, 937 w, 839 m, 819 w, 720 s, 676 s, 691 m, 554 w, 468 m, 442 w.

Synthesis of $\text{Co}(\text{Hdmpz})_2(\text{OOCBu}')_2$ (2). To a solution of 0.25 g (0.18 mmol) of complex **1** in 7 mL of benzene, a solution of 0.106 g (1.11 mmol) of 3,5-dimethylpyrazole in 5 mL of benzene was added. The resulting solution was concentrated to 10 mL and kept at +5°C for 2 days. The violet crystals formed were isolated by decantation, washed with cold hexane, and dried in an argon flow. Yield, 0.14 g (82.8%).

For $\text{CoC}_{24}\text{H}_{26}\text{N}_4\text{O}_4$ anal. calcd (%): C, 58.42; H, 5.31. Found (%): C, 58.37; H, 5.37.

IR (cm^{-1}): 3189 m, 3138 m, 3105 m, 3064 m, 3030 m, 2972 m, 2927 m, 2862 m, 2738 m, 2677 m, 2491 w, 1975 w, 1922 w, 1605 s, 1566 s, 1545 s, 1491 m, 1475 m, 1449 m, 1397 s, 1292 s, 1171 m, 1152 m, 1069 m, 1045 m, 1024 m, 981 w, 943 w, 850 m, 840 m, 806 m, 784 w, 742 w, 723 s, 707 m, 571 w, 447 m, 422 m.

Reaction of $\text{Co}_2(\mu\text{-dmpz})_2(\text{Hdmpz})_2(\text{OOCBu}')_2$ with PhCOOH. Benzoic acid (0.068 g, 0.56 mmol) was added to a solution of 0.1 g (0.14 mmol) of $\text{Co}_2(\mu\text{-dmpz})_2(\text{Hdmpz})_2(\text{OOCBu}')_2$ in 15 mL of *o*-xylene, and the mixture was stirred at 140°C for 3 h. The solution obtained was concentrated to 5 mL and kept at +5°C for several days. The violet crystals formed were isolated by decantation, washed with cold hexane, and dried in an argon flow. Yield of **2**, 0.098 g (71%).

For $\text{CoC}_{24}\text{H}_{26}\text{N}_4\text{O}_4$ anal. calcd (%): C, 58.42; H, 5.31; N, 11.35. Found (%): C, 58.99; H, 4.97; N, 11.03.

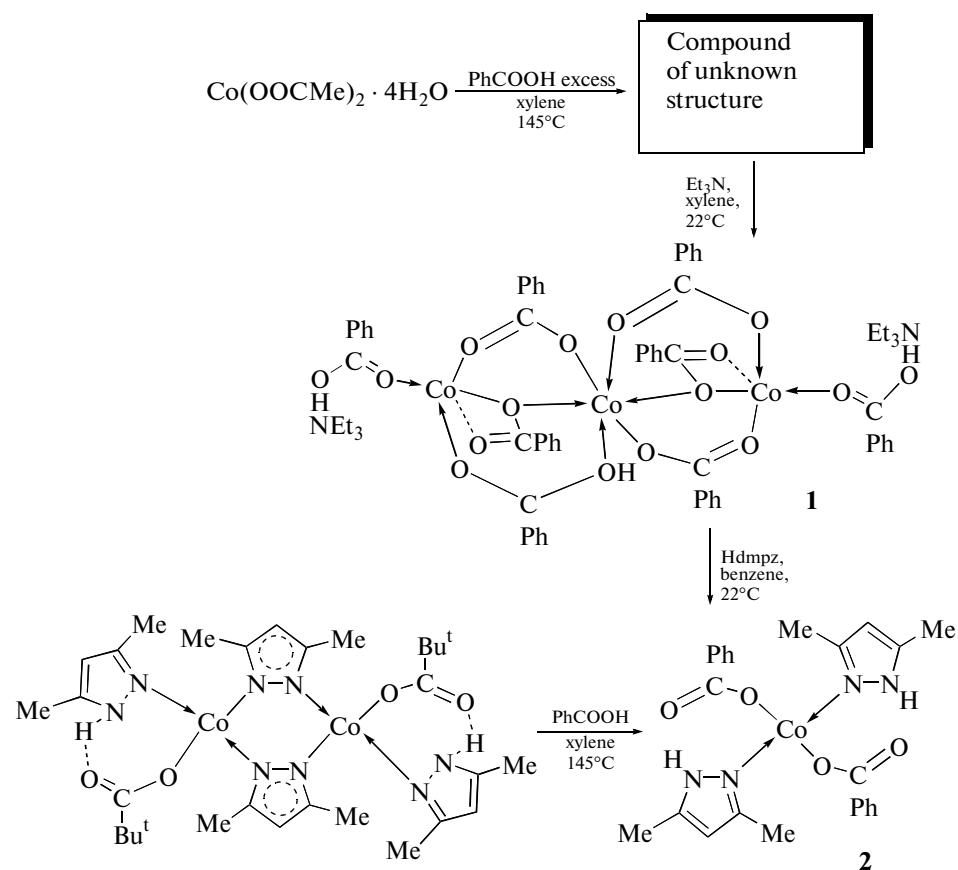
IR (cm^{-1}): 3189 m, 3138 m, 3105 m, 3064 m, 3030 m, 2972 m, 2927 m, 2862 m, 2738 m, 2677 m, 2491 w, 1975 w, 1922 w, 1605 s, 1566 s, 1545 s, 1491 m, 1475 m, 1449 m, 1397 s, 1292 s, 1171 m, 1152 m, 1069 m, 1045 m, 1024 m, 981 w, 943 w, 850 m, 840 m, 806 m, 784 w, 742 w, 723 s, 707 m, 571 w, 447 m, 422 m.

RESULTS AND DISCUSSION

It was found that the compound obtained by the thermal reaction between cobalt acetate hydrate and benzoic acid reacts with excess triethylamine to produce violet trinuclear complex $\text{Co}_3(\mu\text{-OOCPh})_4(\mu,\eta^2\text{-OOCPh})_2[\text{OC}(\text{Ph})\text{OHNET}_3]_2$ (**1**) containing axially coordinated adduct of carboxylic acid and triethylamine:

Table 2. Atomic coordinates ($\times 10^4$) and equivalent isotropic factors ($\text{\AA}^2 \times 10^3$) for complex **1**

Atom	<i>x</i>	<i>y</i>	<i>z</i>	<i>U</i> _{eq}
Co(1)	5000	5000	0	35(1)
Co(2)	3624(1)	6095(1)	958(1)	41(1)
O(1)	4380(1)	6301(1)	103(1)	46(1)
O(2)	4978(2)	7282(2)	1093(1)	75(1)
O(3)	5552(2)	4837(1)	1297(1)	54(1)
O(4)	4527(2)	5468(1)	1944(1)	55(1)
O(5)	6448(1)	5535(1)	127(1)	51(1)
O(6)	7491(1)	4716(1)	-354(1)	53(1)
O(7)	2958(2)	6937(1)	1505(2)	65(1)
O(8)	2456(2)	7602(2)	266(1)	79(1)
C(1)	4833(2)	7033(2)	362(2)	49(1)
C(2)	5081(2)	7588(2)	-270(2)	54(1)
C(3)	5363(3)	7259(2)	-930(2)	70(1)
C(4)	5569(3)	7798(3)	-1515(3)	95(1)
C(5)	5448(4)	8661(3)	-1463(3)	103(2)
C(6)	5147(4)	9001(3)	-824(4)	106(2)
C(7)	4980(3)	8467(2)	-210(3)	79(1)
C(8)	5292(2)	5007(2)	1932(2)	45(1)
C(9)	5951(2)	4651(2)	2773(2)	50(1)
C(10)	5560(3)	4638(2)	3453(2)	64(1)
C(11)	6199(4)	4342(3)	4232(2)	90(1)
C(12)	7199(4)	4052(3)	4334(3)	101(2)
C(13)	7576(3)	4063(3)	3668(3)	103(2)
C(14)	6951(3)	4354(3)	2886(2)	77(1)
C(15)	7311(2)	5353(2)	50(2)	41(1)
C(16)	8233(2)	5923(2)	462(2)	46(1)
C(17)	9197(2)	5757(2)	360(2)	65(1)
C(18)	10066(3)	6267(3)	753(3)	79(1)
C(19)	9955(3)	6939(3)	1228(3)	87(1)
C(20)	8997(3)	7112(3)	1336(3)	96(1)
C(21)	8138(3)	6607(2)	950(2)	70(1)
C(22)	2631(2)	7592(2)	1045(2)	53(1)
C(23)	2512(2)	8393(2)	1499(2)	46(1)
C(24)	2440(3)	9179(2)	1110(2)	66(1)
C(25)	2418(3)	9921(2)	1550(3)	81(1)
C(26)	2437(3)	9888(3)	2370(3)	84(1)
C(27)	2471(3)	9124(3)	2761(2)	81(1)
C(28)	2521(2)	8363(2)	2329(2)	59(1)
C(29)	1854(9)	7543(7)	-1905(4)	251(7)
C(30)	2428(5)	8202(5)	-1867(4)	153(3)
C(31)	263(7)	7788(6)	-1640(5)	232(6)
C(32)	-264(4)	8030(5)	-1048(4)	132(2)
C(33)	1141(5)	6394(3)	-1374(3)	123(2)
C(34)	506(4)	5965(3)	-2196(3)	112(2)
N(1)	1197(3)	7310(2)	-1377(2)	79(1)



According to the X-ray diffraction data, three cobalt atoms of **1** that lie on the same line are at a non-

bonding distance $\text{Co}\cdots\text{Co}$ of $3.2811(5)$ Å. The atoms are connected in pairs by two $\mu\text{-O},\text{O}'\text{-benzoate}$ bridges

Table 3. Selected bond lengths (d) and bond angles (ω) for complex **1**

Bond	d , Å	Bond	d , Å
Co(1)O(3)	2.0580(18)	Co(1)O(3)#1	2.0580(18)
Co(1)O(5)	2.0607(17)	Co(1)O(5)#1	2.0607(17)
Co(1)O(1)	2.2249(17)	Co(1)O(1)#1	2.2250(17)
Co(2)O(4)	1.9586(19)	Co(2)O(6)#1	1.9688(19)
Co(2)O(7)	1.970(2)	Co(2)O(1)	2.0209(17)
Angle	ω , deg	Angle	ω , deg
O(3)Co(1)O(3)#1	179.999(1)	O(3)Co(1)O(5)	86.27(8)
O(3)#1Co(1)O(5)	93.73(8)	O(3)Co(1)O(5)#1	93.73(8)
O(3)#1Co(1)O(5)#1	86.27(8)	O(5)Co(1)O(5)#1	180.0
O(3)Co(1)O(1)	92.84(7)	O(3)#1Co(1)O(1)	87.16(7)
O(5)Co(1)O(1)	89.07(7)	O(5)#1Co(1)O(1)	90.93(7)
O(3)Co(1)O(1)#1	87.16(7)	O(3)#1Co(1)O(1)#1	92.84(7)
O(5)Co(1)O(1)#1	90.93(7)	O(5)#1Co(1)O(1)#1	89.07(7)
O(1)Co(1)O(1)#1	180.0	O(4)Co(2)O(6)#1	105.50(9)
O(4)Co(2)O(7)	101.34(9)	O(6)#1Co(2)O(7)	106.48(9)
O(4)Co(2)O(1)	110.77(8)	O(6)#1Co(2)O(1)	102.37(8)
O(7)Co(2)O(1)	128.54(8)	C(1)O(1)Co(2)	101.20(16)
C(1)O(1)Co(1)	133.07(16)	Co(2)O(1)Co(1)	101.11(7)
C(8)O(3)Co(1)	139.38(18)	C(8)O(4)Co(2)	123.99(18)
C(15)O(5)Co(1)	140.94(18)	C(15)O(6)Co(2)#1	122.19(16)
C(22)O(7)Co(2)	112.36(19)		

Symmetry codes: #1 $-x + 1, -y + 1, -z$.

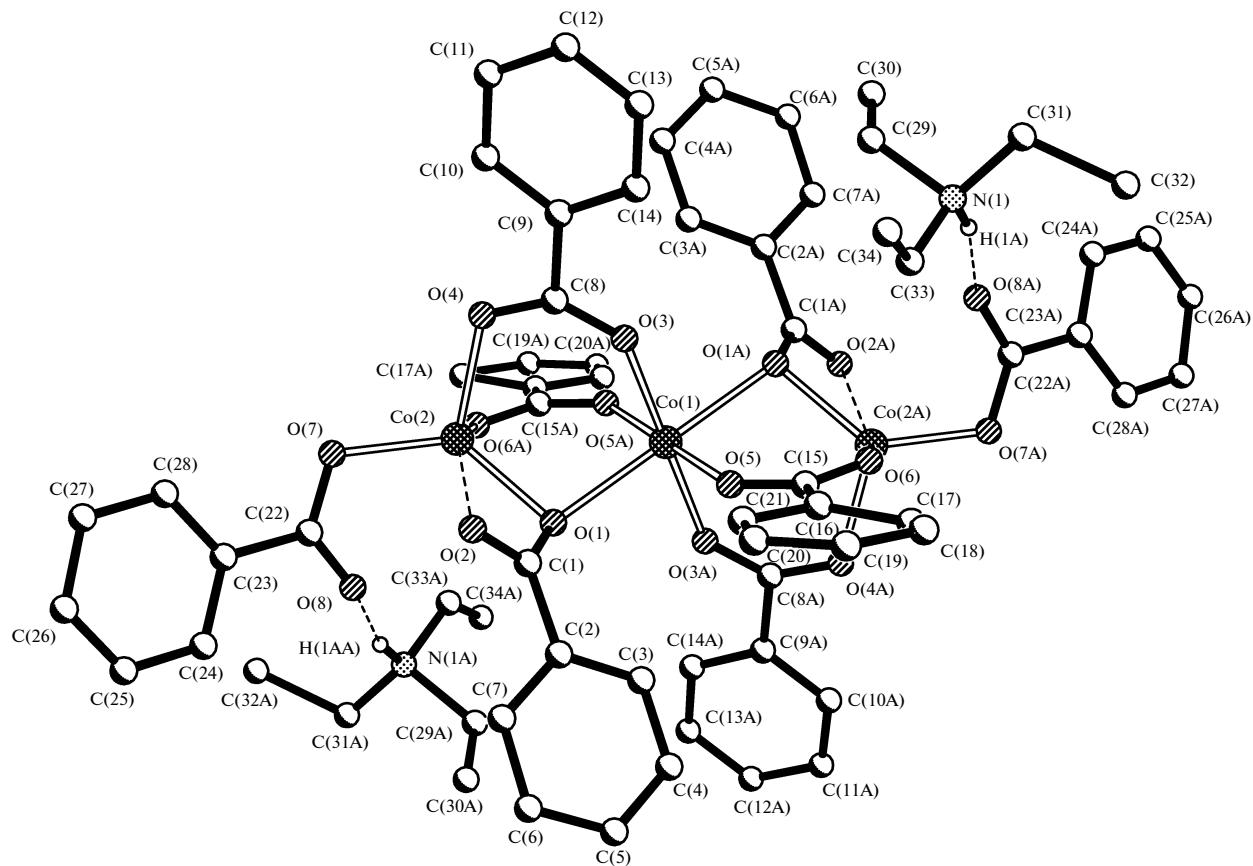
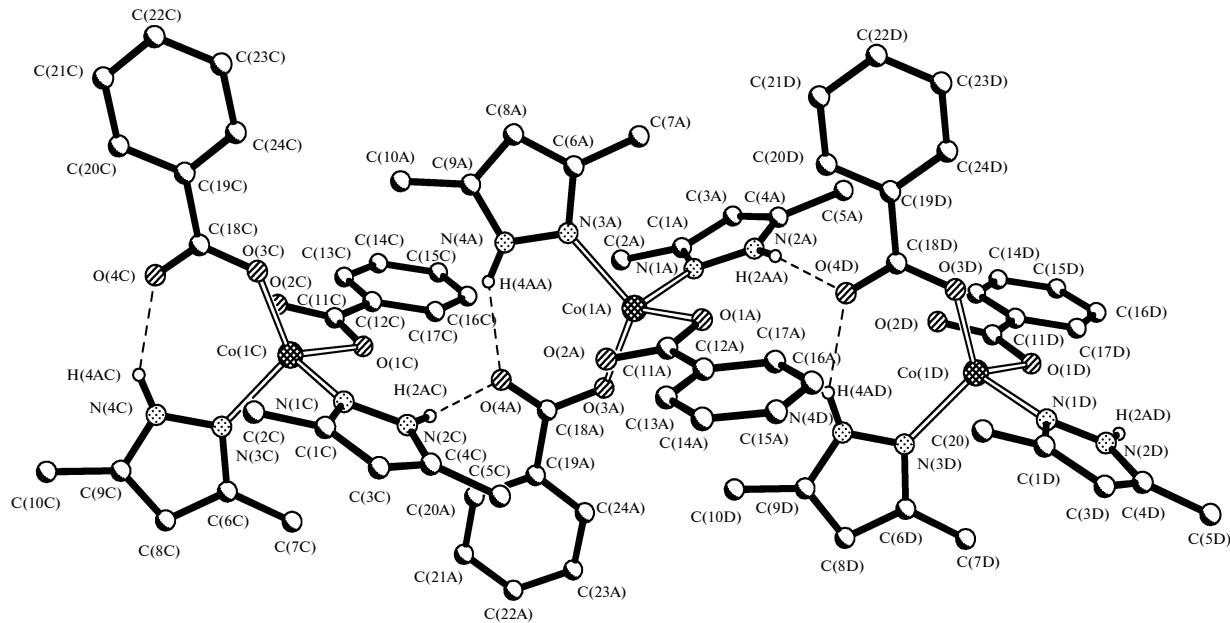
**Fig. 1.** Structure of complex **1**.**Fig. 2.** A fragment of crystal packing of complex **2**.

Table 4. Atomic coordinates ($\times 10^4$) and equivalent isotropic factors ($\text{\AA}^2 \times 10^3$) for complex **2**

Atom	<i>x</i>	<i>y</i>	<i>z</i>	<i>U</i> _{eq}
Co(1)	2690(1)	472(1)	2133(1)	24(1)
O(1)	1519(1)	-491(1)	2183(1)	33(1)
O(2)	924(1)	865(1)	2697(1)	35(1)
O(3)	3502(1)	892(1)	3314(1)	29(1)
O(4)	3028(1)	2418(1)	3195(1)	29(1)
N(1)	3806(1)	-417(1)	1625(1)	26(1)
N(2)	3550(1)	-1310(1)	1309(1)	27(1)
N(3)	2477(1)	1472(1)	1109(1)	26(1)
N(4)	2710(1)	2401(1)	1273(1)	27(1)
C(1)	4818(1)	-253(1)	1382(1)	32(1)
C(2)	5368(2)	679(1)	1614(1)	44(1)
C(3)	5195(1)	-1041(1)	922(1)	40(1)
C(4)	4371(1)	-1705(1)	889(1)	32(1)
C(5)	4290(2)	-2683(1)	480(1)	47(1)
C(6)	2188(1)	1403(1)	186(1)	30(1)
C(7)	1849(2)	472(1)	-253(1)	43(1)
C(8)	2243(1)	2294(1)	-227(1)	33(1)
C(9)	2578(1)	2918(1)	482(1)	30(1)
C(10)	2790(2)	3961(1)	467(1)	41(1)
C(11)	775(1)	10(1)	2519(1)	28(1)
C(12)	-275(1)	-476(1)	2692(1)	29(1)
C(13)	-1141(1)	44(1)	2988(1)	42(1)
C(14)	-2111(2)	-418(2)	3150(2)	53(1)
C(15)	-2213(2)	-1382(2)	3030(1)	50(1)
C(16)	-1354(1)	-1901(1)	2737(1)	41(1)
C(17)	-388(1)	-1449(1)	2563(1)	32(1)
C(18)	3457(1)	1735(1)	3643(1)	23(1)
C(19)	3990(1)	1897(1)	4620(1)	23(1)
C(20)	4130(1)	2819(1)	4962(1)	31(1)
C(21)	4634(1)	2977(1)	5855(1)	35(1)
C(22)	5018(1)	2221(1)	6411(1)	34(1)
C(23)	4889(1)	1305(1)	6076(1)	34(1)
C(24)	4370(1)	1142(1)	5183(1)	28(1)

(Co(1)–O(3), 2.055(2) Å; Co(1)–O(5), 2.061(2) Å; Co(2)–O(4), 1.955(2) Å; Co(2)–O(6), 1.967(2) Å) and one μ -O bridging benzoate anion (Co(1)–O(1), 2.227(2) Å; Co(2)–O(1), 2.019(2) Å). The central cobalt atom has a distorted octahedral environment, and each of the peripheral atoms is in a tetragonal-pyramidal environment, which, in addition to oxygen atoms of the bridging anions, is completed by contacts with the O(2) atom of the μ -O bridging moiety (Co(2)–O(2), 2.554(3) Å) and the oxygen atom of the adduct PhCOO \cdots HNEt₃ (Co(2)–O(7) 1.970(2) Å) (Fig. 1, Tables 2, 3).

When complex **1** interacts with excess Hdmpz in benzene, deprotonation of pyrazole does not occur, as distinct from the cobalt complexes containing pivalate or acetate anions, and the violet mononuclear complex Co(OOCPh)₂(Hdmpz)₂ (**2**) is formed. In this complex, according to X-ray diffraction data, the cobalt(II) atom has a distorted tetrahedral environment of two oxygen atoms of the two terminal benzoate anions (Co(1)–O(1), 1.979(1) Å; Co(1)–O(3), 1.966(1) Å) and two nitrogen atoms of the coordinated Hdmpz molecules (Co(1)–N(1), 2.038(1) Å; Co(1)–N(3), 2.031(1) Å). In the crystal unit of the complex, linear polymers are formed due to intermolecular hydrogen bonds of the pyrazole NH fragments and oxygen atoms of the benzoate anions (N \cdots O, 2.681(2) Å) (Fig. 2, Tables 4, 5).

Complex **2** was unexpectedly obtained in high yield when we tried to replace the terminal pivalate anions in Co₂(μ -dmpz)₂(Hdmpz)₂(OOCBu $'$)₂ with more basic benzoate anions. However, this reaction likely proceeded through protonation of the bridging pyrazolate anions due to the presence of strong benzoic acid, as it occurred with the pyrazolate-bridged dimer in the reaction with trifluoromethanesulfonic acid [1, 15], and pivalic acid was formed.

Thus, the studies performed have shown that the structures of the products of the reactions of zinc, copper, and cobalt carboxylates with triethylamine and pyrazole are determined by the Lewis acidity of the metal center, which depends on the donor ability of the R substituent in the carboxylate anion. For less basic benzoate and acetate anions, the adduct [OC(R)O] $^-($ HNEt₃) $^+$ (R = Ph, Me) is coordinated in the zinc, copper, and cobalt complexes at an excess of triethylamine in the presence of the corresponding acids; whereas for the pivalate anion, complexes M₂(μ -OOCBu $'$)₄(NEt₃)₂ were obtained under the same conditions. The reactions of zinc benzoate and acetate with Hdmpz in the presence of triethylamine lead to deprotonation of pyrazole and formation of binuclear complexes containing intramolecular hydrogen bonds of the pyrazole NH moieties with oxygen atoms of the carboxylate anions [16–18]. Cobalt forms a trinuclear complex with intermolecular hydrogen bonds in case of acetate and a mononuclear complex in case of benzoate [16, 17]. The interaction of {[Cu₂(μ -OOCMe)₄[μ -OOCMe(HNEt₃)]}_n with 3,5-dimethyl-

Table 5. Selected bond lengths (d) and bond angles (ω) for complex 2

Bond	d , Å	Bond	d , Å
Co(1)O(3)	1.9658(10)	Co(1)O(1)	1.9789(11)
Co(1)N(3)	2.0309(12)	Co(1)N(1)	2.0378(13)
O(1)C(11)	1.2813(19)	O(2)C(11)	1.2404(18)
O(3)C(18)	1.2812(16)	O(4)C(18)	1.2431(16)
N(1)C(1)	1.337(2)	N(1)N(2)	1.3630(16)
N(2)C(4)	1.339(2)	N(3)C(6)	1.3350(19)
N(3)N(4)	1.3556(16)	N(4)C(9)	1.3417(19)
Angle	ω , deg	Angle	ω , deg
O(3)Co(1)O(1)	118.94(5)	O(3)Co(1)N(3)	115.46(5)
O(1)Co(1)N(3)	117.25(5)	O(3)Co(1)N(1)	100.92(5)
O(1)Co(1)N(1)	95.90(5)	N(3)Co(1)N(1)	102.36(5)
C(11)O(1)Co(1)	100.25(9)	C(18)O(3)Co(1)	123.94(9)
C(1)N(1)N(2)	105.24(12)	C(1)N(1)Co(1)	130.89(10)
N(2)N(1)Co(1)	123.04(9)	C(6)N(3)Co(1)	131.67(10)
N(4)N(3)Co(1)	122.29(9)		

pyrazole does not lead to deprotonation of pyrazole, and mono- and binuclear complexes were isolated as was observed for pivalates. However, the binuclear complex with bridging acetate anions contains intermolecular hydrogen bonds, whereas the binuclear complex with bridging pivalate anions contains intramolecular hydrogen bonds between the NH fragments of the coordinated pyrazole and oxygen atoms of the corresponding anion [18].

Note also that, in the absence of triethylamine, deprotonation of pyrazole with zinc and cobalt acetate hydrates occurs on heating to 150°C and leads to formation of the complexes $M_3(\mu\text{-dmpz})_4(\text{Hdmpz})_2(\text{OOCMe})_2$, in which the character of hydrogen bonds depends on the natures of the transition metal and solvation molecules [16].

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