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COORDINATION COMPOUNDS

Interaction of 3,5-Dimethylpyrazole with Cobalt(II) Carboxylates Containing Coordinated 1,10-Phenanthroline or 2,2'-Dipyridyl

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Abstract—The reaction of $Co_3(\mu\text{-}OOCBu^t)_6(NEt_3)_2$ with 1,10-phenanthroline or 2,2'-dipyridyl in benzene at room temperature yields $L_2Co_2(\mu\text{-}OH_2)(\mu\text{-}OOCBu^t)_4$ complexes (L_2 = Phen (**1a**) and Dipy (**1b**)). The reaction of **1a** (**1b**) with 3,5-dimethylpyrazole gives a mixture of $L_2Co(Hdmpz)_2(OOCBu^t)_2$ complexes (**2a**, **2b**) and $L_2Co(Hdmpz)(OOCBu^t)_2$ (**3a**, **3b**), and their yield is determined by the ratio of the initial reagents. As distinct from pivalates, for cobalt(II) phenanthroline-benzoate, only the $(Phen)Co(Hdmpz)_2(OOCPh)_2$ complex (**4**) has been isolated. The structures of the synthesized compounds have been determined by X-ray crystallography.

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Deprotonation of coordinated pyrazole leads to the formation of pyrazolate-bridged bi- and polynuclear compounds, which have attracted much recent interest in the context of the discussed analogies between three-electron-donating pyrazolate and carboxylate anions [1–12].

Previously, it was shown that, owing to deprotonation of Hdmpz, the reaction of $Co_3(\mu\text{-}OOCBu^t)_6(NEt_3)_2$ with 3,5-dimethylpyrazole (Hdmpz) under mild conditions (benzene, room temperature) yields almost quantitatively pyrazolate-bridged binuclear complex $Co_2(\mu\text{-}dmpz)_2(OOCBu^t)_2(Hdmpz)_2$ [13–15]. When an electron-withdrawing phenyl substituent is substituted for the electron-donating *tert*-butyl substituent in the carboxylate anion, the analogous reaction yields only the mononuclear complex $Co(OOCPh)_2(Hdmpz)_2$, in which the coordinated pyrazole can be deprotonated with cobalt(II) acetate to give the binuclear pyrazolate-bridged complex $Co_2(\mu\text{-}dmpz)_2(OOCPh)_2(Hdmpz)_2$ [16, 17].

In the present work, we consider structural features of the products of the reactions of Hdmpz with cobalt(II) pivalate or benzoate containing, as distinct from monodentate triethylamine (which can also act as a proton acceptor), strong bidentate donors, 1,10-phenanthroline and 2,2'-dipyridyl.

EXPERIMENTAL

All operations dealing with 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 Specord M-80 and Nexus-Nicolet spectrophotometers in the range 400–4000 cm^{-1}.

X-ray diffraction experiments were carried out using a routine technique on a Bruker SMART Apex II automated diffractometer with a CCD detector (λMo, graphite monochromator, ω scan). The structures were calculated with the SHELXTL PLUS program package (PC version). The structures were refined with the SHELXTL-97 program package [18, 19].

The crystal data and refinement details are listed in Table 1, and the atomic coordinates and selected bond lengths and bond angles in the complexes studied are presented in Tables 2–11. Complete tables of atomic coordinates, bond lengths, and bond angles were deposited with the Cambridge Structural Database (CCDC 891697–891701).

$L_2Co_2(\mu\text{-}OH_2)(\mu\text{-}OOCBu^t)_4$ (L_2 = Phen (1a), Dipy (1b)). Aqueous phenanthroline (0.12 g, 0.6 mmol) was added to a solution of 0.2 g (0.2 mmol) of $Co_3(\mu\text{-}OOCBu^t)_6(NEt_3)_2$ in 20 mL of benzene. The resulting red solution was concentrated to 10 mL and kept at +5°C for 24 h. The resulting orange crystals were separated from the mother solution by decantation, washed with hexane, and dried in an argon flow. The yield was 0.21 g (84%).

For $Co_2C_{40}H_{54}N_4$ (powder) anal. calcd. (%): C, 56.34; H, 6.38; N, 6.57. Found (%): C, 56.98; H, 6.29; N, 6.51.

IR (KBr) (ν, cm^{-1}): 3437 m, 2954 m, 2922 w, 1600 s, 1516 s, 1481 s, 1455 w, 1411 s, 1367 m, 1356 m,

Table 1. Crystallographic parameters and refinement details for structures **1–4**

	1a × 0.5CH_2Cl_2	**2b**	**3a**	**4 × C_6H_6**	**4 × C_6H_6 (4-1)**
Empirical formula	$C_{44.25}H_{54.50}Cl_{0.50}Co_2N_4O_9$	$C_{30}H_{42}CoN_6O_4$	$C_{27}H_{34}CoN_4O_4$	$C_{42}H_{40}CoN_6O_4$	$C_{42}H_{40}CoN_6O_4$
FW	922.00	609.63	537.51	751.73	751.73
T, K	173(2)	173(2)	173(2)	173(2)	173(2)
Color	Orange	Red-orange	Yellow-orange	Orange	Orange
Symmetry system	Triclinic	Orthorhombic	Monoclinic	Triclinic	Monoclinic
Space group	P-1	$Pna2(1)$	$P2(1)/n$	P-1	$P2/c$
Unit cell parameters, Å, deg	$a = 9.596(4)$	$a = 19.467(3)$	$a = 11.1338(11)$	$a = 12.3416(17)$	$a = 16.273(2)$
	$b = 12.893(5)$	$b = 11.5346(15)$	$b = 16.6989(17)$	$b = 12.7078(17)$	$b = 10.1345(14)$
	$c = 20.322(7)$	$c = 14.0539(16)$	$c = 14.6518(15)$	$c = 15.220(2)$	$c = 23.741(3)$
	$\alpha = 100.840(6)$	$\alpha = 90$	$\alpha = 90$	$\alpha = 69.033(2)$	$\alpha = 90$
	$\beta = 90.304(6)$	$\beta = 90$	$\beta = 90.924(2)$	$\beta = 79.195(2)$	$\beta = 101.794(2)$
	$\gamma = 106.785(6)$	$\gamma = 90$	$\gamma = 90$	$\gamma = 63.366(2)$	$\gamma = 90$
V, Å^3	2359.6(15)	3155.7(7)	2723.7(5)	1991.4(5)	3832.7(9)
Z	2	4	4	2	4
ρ_{calc}, g/cm^3	1.298	1.283	1.311	1.254	1.303
μ, mm^{-1}	0.786	0.587	0.668	0.479	0.497
F(000)	965	1292	1132	786	1572
Crystal size, mm	0.14 × 0.12 × 0.10	0.14 × 0.12 × 0.10	0.16 × 0.14 × 0.12	0.14 × 0.12 × 0.10	0.14 × 0.12 × 0.10
θ range, deg	2.14–25.00	2.05–27.00	2.28–28.00	2.07–28.00	2.37–28.00
Index ranges	$-11 \le h \le 11$,	$-24 \le h \le 10$,	$-14 \le h \le 14$,	$-16 \le h \le 16$,	$-15 \le h \le 21$,
	$-15 \le k \le 15$,	$-4 \le k \le 14$,	$-22 \le k \le 22$,	$-16 \le k \le 16$,	$-12 \le k \le 13$,
	$-24 \le l \le 24$	$-16 \le l \le 6$	$-18 \le l \le 19$	$-19 \le l \le 20$	$-31 \le l \le 31$
Number of reflections	19210	5727	19577	20275	25004
Number of unique reflections	8278 [R(int) = 0.0542]	4270 [R(int) = 0.0361]	6503 [R(int) = 0.0519]	9500 [R(int) = 0.0592]	9211 [R(int) = 0.0406]
GOOF	0.993	0.911	1.005	0.848	0.968
R ($I > 2\sigma(I)$)	$R1 = 0.0869$, $wR2 = 0.2431$	$R1 = 0.0466$, $wR2 = 0.1080$	$R1 = 0.0447$, $wR2 = 0.1061$	$R1 = 0.0402$, $wR2 = 0.0608$	$R1 = 0.0442$, $wR2 = 0.1056$
R (over all reflections)	$R1 = 0.1336$, $wR2 = 0.2855$	$R1 = 0.0619$, $wR2 = 0.1136$	$R1 = 0.0667$, $wR2 = 0.1189$	$R1 = 0.0791$, $wR2 = 0.0666$	$R1 = 0.0824$, $wR2 = 0.1272$
max/min electron density peaks, e Å^{-3}	1.824 and −1.686	0.453 and −0.334	0.663 and −0.291	0.424 and −0.419	0.364 and −0.327

Table 2. Atomic coordinates ($\times 10^4$) and equivalent isotropic factors ($Å^2 \times 10^3$) for complex **1a**

Atom	x	y	z	U_{eq}	Atom	x	y	z	U_{eq}
Co(1)	6824(1)	8030(1)	3282(1)	40(1)	C(17)	2519(12)	3317(9)	2824(7)	101(2)
Co(2)	6254(1)	5563(1)	2076(1)	41(1)	C(18)	2322(12)	2464(9)	2080(7)	101(2)
O(1)	5285(5)	6772(4)	2565(2)	44(1)	C(19)	3244(12)	2620(9)	3082(7)	101(2)
O(2)	7561(6)	6949(4)	3730(2)	56(1)	C(20)	1087(14)	3314(12)	2971(13)	208(11)
O(3)	7713(6)	5663(4)	2850(3)	57(1)	C(21)	4263(8)	7278(6)	4241(4)	54(2)
O(4)	8450(5)	8159(4)	2609(2)	52(1)	C(22)	3580(10)	7339(8)	4836(5)	72(2)
O(5)	7816(5)	6769(4)	1718(2)	52(1)	C(23)	4196(10)	8168(8)	5373(4)	68(2)
O(7)	6046(6)	9183(4)	2919(2)	51(1)	C(24)	5498(10)	8959(7)	5307(4)	60(2)
O(8)	5058(6)	8277(4)	1897(2)	56(1)	C(25)	6152(8)	8846(6)	4677(3)	47(2)
O(9)	4741(6)	4366(4)	2479(3)	55(1)	C(26)	7502(8)	9617(6)	4587(3)	45(2)
O(10)	3111(6)	5229(5)	2839(3)	71(2)	C(27)	6248(12)	9883(8)	5827(4)	72(3)
N(1)	5507(6)	8017(4)	4146(3)	45(1)	C(28)	7490(11)	10609(8)	5737(4)	67(2)
N(2)	8087(6)	9443(5)	3988(3)	45(1)	C(29)	8169(9)	10503(6)	5114(4)	55(2)
N(3)	4699(6)	4993(5)	1203(3)	51(2)	C(30)	9503(10)	11230(7)	4989(5)	72(3)
N(4)	7042(7)	4348(5)	1456(3)	51(2)	C(31)	10057(10)	11076(7)	4375(5)	67(2)
C(1)	7748(10)	6034(7)	3474(4)	66(1)	C(32)	9317(9)	10170(6)	3881(4)	58(2)
C(2)	8177(10)	5395(7)	3945(4)	66(1)	C(33)	3502(9)	5266(8)	1089(4)	65(2)
C(3)	7736(10)	4144(6)	3630(4)	66(1)	C(34)	2545(10)	4805(8)	521(5)	75(3)
C(4)	9925(16)	5770(10)	3989(9)	146(7)	C(35)	2853(11)	4002(8)	49(5)	77(3)
C(5)	7700(30)	5613(13)	4620(6)	197(11)	C(36)	4088(10)	3660(7)	151(4)	65(2)
C(6)	8733(9)	7565(8)	2091(5)	71(1)	C(37)	4972(8)	4165(6)	739(4)	54(2)
C(7)	10340(9)	7856(8)	1924(5)	71(1)	C(38)	6234(9)	3854(6)	869(4)	54(2)
C(8)	11151(15)	9012(12)	2183(10)	173(8)	C(39)	4484(14)	2876(9)	−329(5)	88(3)
C(9)	11076(9)	7298(8)	2407(5)	71(1)	C(40)	5646(15)	2558(9)	−211(5)	94(4)
C(10)	10560(14)	7280(20)	1259(8)	236(14)	C(41)	6607(11)	3026(7)	396(4)	70(3)
C(11)	5724(10)	9158(7)	2313(5)	66(1)	C(42)	7861(13)	2753(8)	548(6)	84(3)
C(12)	6248(9)	10228(7)	2055(5)	66(1)	C(43)	8676(12)	3262(8)	1125(6)	84(3)
C(13)	7687(9)	10260(7)	1735(5)	66(1)	C(44)	8220(10)	4059(7)	1580(5)	65(2)
C(14)	6490(20)	11236(9)	2618(6)	153(7)	Cl(1S)	690(20)	1100(20)	338(12)	191(8)
C(15)	5228(15)	10277(11)	1489(7)	122(5)	Cl(2S)	2800(30)	280(30)	292(18)	267(14)
C(16)	3535(13)	4382(9)	2703(7)	101(2)	C(1S)	990(50)	−150(40)	−40(50)	220(40)

1224 m, 1141 w, 1103 w, 1043 w, 867 m, 846 m, 785 m, 729 s, 680 w, 598 m, 422 w.

Complex **1b** was obtained by an analogous procedure (0.18 g, yield 75%).

For $Co_2C_{36}H_{54}O_9N_4$ (powder) anal. calcd. (%): C, 53.73; H, 6.76; N, 6.96. Found (%): C, 56.68; H, 6.69; N, 6.82.

IR (KBr) (ν, cm^{-1}): 3444 m, 2933 m, 1604 s, 1514 s, 1486 s, 1458 w, 1411 m, 1367 m, 1358 m, 1229 w, 1138 w, 1100 w, 868 m, 841 m, 788 m, 722 s, 687 m, 560 m.

Reactions of 1a and 1b with **3,5-dimethylpyrazole. Synthesis of $L_2Co(OOCBu^t)_2(Hdmpz)_2$ (2a, 2b) and $L_2Co(\eta^2\text{-}OOCBu^t)(OOCBu^t)(Hdmpz)$ (3a, 3b).** General procedure: Hdmpz was added to a red solution of

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

Bond	*d*, Å	Bond	*d*, Å
Co(1)–O(4)	2.072(5)	Co(1)–O(2)	2.076(5)
Co(1)–O(7)	2.087(5)	Co(1)–N(2)	2.137(6)
Co(1)–O(1)	2.154(4)	Co(1)–N(1)	2.170(6)
Co(2)–O(3)	2.060(5)	Co(2)–O(5)	2.070(5)
Co(2)–O(9)	2.087(5)	Co(2)–O(1)	2.132(4)
Co(2)–N(4)	2.137(6)	Co(2)–N(3)	2.184(6)
Angle	ω, deg	Angle	ω, deg
O(4)Co(1)O(7)	91.8(2)	O(2)Co(1)O(7)	174.74(18)
O(4)Co(1)N(2)	92.9(2)	O(2)Co(1)N(2)	92.7(2)
O(7)Co(1)N(2)	83.8(2)	O(4)Co(1)O(1)	91.93(19)
O(2)Co(1)O(1)	96.0(2)	O(7)Co(1)O(1)	87.20(19)
N(2)Co(1)O(1)	169.9(2)	O(4)Co(1)N(1)	167.8(2)
O(2)Co(1)N(1)	81.2(2)	O(7)Co(1)N(1)	94.1(2)
N(2)Co(1)N(1)	77.2(2)	O(1)Co(1)N(1)	99.0(2)
O(3)Co(2)O(5)	89.7(2)	O(3)Co(2)O(9)	87.7(2)
O(5)Co(2)O(9)	177.3(2)	O(3)Co(2)O(1)	98.15(19)
O(5)Co(2)O(1)	90.53(19)	O(9)Co(2)O(1)	89.29(19)
O(3)Co(2)N(4)	90.3(2)	O(5)Co(2)N(4)	88.4(2)
O(9)Co(2)N(4)	92.2(2)	O(1)Co(2)N(4)	171.5(2)
O(3)Co(2)N(3)	164.6(2)	O(5)Co(2)N(3)	98.5(2)
O(9)Co(2)N(3)	84.1(2)	O(1)Co(2)N(3)	94.8(2)
N(4)Co(2)N(3)	77.1(2)	Co(2)O(1)Co(1)	110.9(2)

$L_2Co_2(\mu\text{-}OH_2)(\mu\text{-}OOCBu^t)_2(OOCBu^t)_2$, and the mixture was stirred for 1 h. The resulting crimson solution was evaporated until dry. The solid product was extracted with hexane, concentrated until the first signs of turbidity appeared, and kept at +5°C for 24 h. The resulting mixture of red-orange (**2a**, **2b**) and yel-

Table 4. Atomic coordinates ($\times 10^4$) and equivalent isotropic factors ($Å^2 \times 10^3$) for complex **2b**

Atom	x	y	z	U_{eq}
Co(1)	1657(1)	5389(1)	3281(1)	24(1)
O(1)	1871(1)	3833(2)	3965(2)	30(1)
O(2)	2702(1)	2770(2)	3302(3)	35(1)
O(3)	1366(1)	6768(2)	2444(2)	33(1)
O(4)	542(2)	7910(3)	3014(2)	49(1)
N(1)	2726(2)	5805(2)	3097(3)	27(1)
N(2)	3192(1)	4938(2)	3191(3)	27(1)
N(3)	1597(2)	6362(3)	4577(2)	27(1)
N(4)	1181(2)	7306(3)	4604(2)	28(1)
N(5)	1516(2)	4379(3)	1997(2)	28(1)
N(6)	580(1)	4860(2)	3336(3)	27(1)
C(1)	3092(2)	6777(3)	3039(3)	28(1)
C(2)	2768(2)	7948(3)	2924(3)	39(1)
C(3)	3795(2)	6526(3)	3104(3)	31(1)
C(4)	3841(2)	5350(3)	3203(4)	27(1)
C(5)	4444(2)	4568(4)	3332(4)	43(1)
C(6)	1872(2)	6291(3)	5458(3)	30(1)
C(7)	2392(2)	5386(4)	5689(3)	46(1)
C(8)	1624(2)	7183(3)	6025(3)	33(1)
C(9)	1185(2)	7807(4)	5469(3)	29(1)
C(10)	779(2)	8884(4)	5647(4)	46(1)
C(11)	2163(2)	2867(3)	3787(3)	26(1)
C(12)	1820(2)	1795(3)	4198(3)	30(1)
C(13)	2241(2)	724(3)	4034(4)	51(1)
C(14)	1719(2)	1969(4)	5286(3)	44(1)
C(15)	1126(2)	1671(4)	3732(4)	55(2)
C(16)	899(2)	7509(4)	2346(3)	35(1)
C(17)	731(3)	7901(5)	1342(3)	58(2)
C(18)	102(3)	7262(9)	1040(4)	160(5)
C(19)	1311(3)	7626(4)	643(3)	51(1)
C(20)	603(5)	9195(6)	1339(5)	191(6)
C(21)	1999(2)	4152(3)	1364(3)	32(1)
C(22)	1898(2)	3483(4)	562(3)	40(1)
C(23)	1255(3)	3010(4)	427(3)	47(1)
C(24)	748(2)	3253(4)	1069(3)	43(1)
C(25)	886(2)	3937(3)	1857(3)	28(1)
C(26)	362(2)	4213(3)	2591(3)	29(1)
C(27)	−316(2)	3828(4)	2535(4)	46(1)
C(28)	−763(2)	4072(4)	3247(5)	54(1)
C(29)	−539(2)	4719(4)	4015(4)	51(1)
C(30)	133(2)	5083(4)	4023(3)	41(1)

low-orange single crystals (**3a**, **3b**) were mechanically separated under a microscope.

L_2	Complex **1** (g, mmol)	Hdmpz (g, mmol)	Yield **2** (%)	Yield **3** (%)
Phen	0.2 (0.22)	0.05 (0.44)	54	12
Phen	0.2 (0.22)	0.1 (0.88)	68	7
Phen	0.2 (0.22)	0.012 (0.11)	14	71
Dipy	0.16 (0.2)	0.038 (0.4)	62	16
Dipy	0.16 (0.2)	0.076 (0.8)	74	5
Dipy	0.16 (0.2)	0.01 (0.1)	10	56

(Phen)Co(OOCBu^t)$_2$(Hdmpz)$_2$ (2a)

For $CoC_{32}H_{42}O_4N_6$ anal. calcd. (%): C, 60.65; H, 6.68; N, 13.27. Found (%): C, 59.96; H, 6.67; N, 13.22.

IR (KBr) (ν, cm^{-1}): 3450 m, 2955 m, 2867 m, 2603 w, 1578 m, 1551 s, 1483 s, 1441 s, 1368 s, 1356 s, 1317 m, 1226 s, 1140 m, 1051 w, 1041 s, 897 m, 832 m, 789 m, 732 s, 640 w, 596 m, 428 m.

(Dipy)Co(OOCBu^t)$_2$(Hdmpz)$_2$ (2b)

For $CoC_{30}H_{42}O_4N_6$ anal. calcd. (%): C, 59.11; H, 6.94; N, 13.79. Found (%): C, 59.13; H, 6.88; N, 13.91.

IR (KBr) (ν, cm^{-1}): 3112 w, 2956 s, 2864 m, 2340 w, 1664 w, 1596 s, 1580 s, 1480 s, 1444 s, 1368 s, 1356 w, 1316 m, 1224 s, 1176 m, 1156 m, 1060 w, 1036 s, 1020 m, 884 m, 832 m, 788 m, 736 s, 628 w, 596 m, 428 m, 396 m, 308 w.

(Phen)Co(η^2-OOCBu^t)(OOCBu^t)(Hdmpz) (3a)

For $CoC_{24}H_{34}O_4N_4$ anal. calcd. (%): C, 57.48; H, 6.83; N, 11.17. Found (%): C, 56.13; H, 6.88; N, 11.01.

IR (KBr) (ν, cm^{-1}): 3435 m, 3052 w, 2957 m, 2864 w, 1578 w, 1551 s, 1511 m, 1482 s, 1427 s, 1408 s, 1366 m 1355 s, 1319 m, 1225 s, 1139 w, 1042 m, 898 m, 851 s, 791 m, 730 s, 640 m, 606 w, 597 w, 556 w.

(Dipy)Co(η^2-OOCBu^t)(OOCBu^t)(Hdmpz) (3b)

For $CoC_{20}H_{34}O_4N_4$ anal. calcd. (%): C, 52.98; H, 7.56; N, 12.35. Found (%): C, 53.03; H, 7.51; N, 12.51.

IR (KBr) (ν, cm^{-1}): 3434 m, 2998 w, 2944 m, 1581 w, 1554 s, 1507 m, 1477 s, 1431 s, 1408 s, 1361 m, 1355 s, 1318 m, 1222 s, 1044 m, 888 m, 850 s, 788 m, 728 s, 640 m, 601 w, 597 w, 551 w, 427 w.

(Phen)Co(OOCPh)$_2$(Hdmpz)$_2$ (4). (1) To a solution of 0.23 g (0.48 mmol) of $Co_3(\mu$-$OOCPh)_6[OC(Ph)OHEt_3]$ in 15 mL of benzene, 0.12 g (0.6 mmol) of aqueous 1,10-phenanthroline was added, and then 0.09 g (0.96 mmol) of Hdmpz was added to the resulting red solution. The solution was concentrated to 7 mL and kept at +5°C for 24 h. The yellow-orange crystals were separated from the mother

Table 5. Selected bond lengths (*d*) and bond angles w) for complex **2b**

Bond	*d*, Å	Bond	*d*, Å
Co(1)–O(3)	2.058(3)	Co(1)–O(1)	2.078(3)
Co(1)–N(3)	2.143(3)	Co(1)–N(1)	2.152(3)
Co(1)–N(5)	2.164(3)	Co(1)–N(6)	2.183(3)
Angle	ω, deg	Angle	ω, deg
O(3)Co(1)O(1)	170.85(11)	O(3)Co(1)N(3)	93.77(12)
O(1)Co(1)N(3)	94.01(12)	O(3)Co(1)N(1)	91.40(11)
O(1)Co(1)N(1)	93.12(10)	N(3)Co(1)N(1)	92.14(13)
O(3)Co(1)N(5)	84.55(11)	O(1)Co(1)N(5)	86.93(12)
N(3)Co(1)N(5)	169.55(12)	N(1)Co(1)N(5)	98.20(13)
O(3)Co(1)N(6)	88.43(11)	O(1)Co(1)N(6)	86.26(11)
N(3)Co(1)N(6)	93.69(13)	N(1)Co(1)N(6)	174.16(15)
N(5)Co(1)N(6)	75.98(13)		

solution by decantation and dried in an argon flow. The yield was 0.22 g (65%).

For $CoC_{36}H_{34}O_4N_6$ (powder) anal. calcd. (%): C, 64,129; H, 5.09; N, 12.48. Found (%): C, 64.26; H, 5.04; N, 12.39.

(2) To a violet solution of 0.1 g (0.2 mmol) of $Co(Hdmpz)_2(OOCPh)_2$ in 15 mL of benzene, 0.036 (0.2 mmol) of 1,10-phenanthroline was added. The resulting red solution was concentrated to 7 mL and kept at +5°C for 24 h. The resulting yellow-orange crystals were separated from the mother solution and dried in an argon flow. The yield was 0.1 g (72%).

For $CoC_{36}H_{34}O_4N_6$ (powder) anal. calcd. (%): C, 64,129; H, 5.09; N, 12.48. Found (%): C, 64.23; H, 4.84; N, 12.47.

IR (KBr) (ν, cm^{-1}): 3434 m, 2956 s, 2860 m, 1604, 1580 m, 1554 s, 1484 s, 1441 s, 1370 s, 1358 s, 1317 m, 1228 s, 1051 w, 1044 s, 897 m, 832 m, 777 m, 732 s, 641 w, 720 s, 594 m, 422 m.

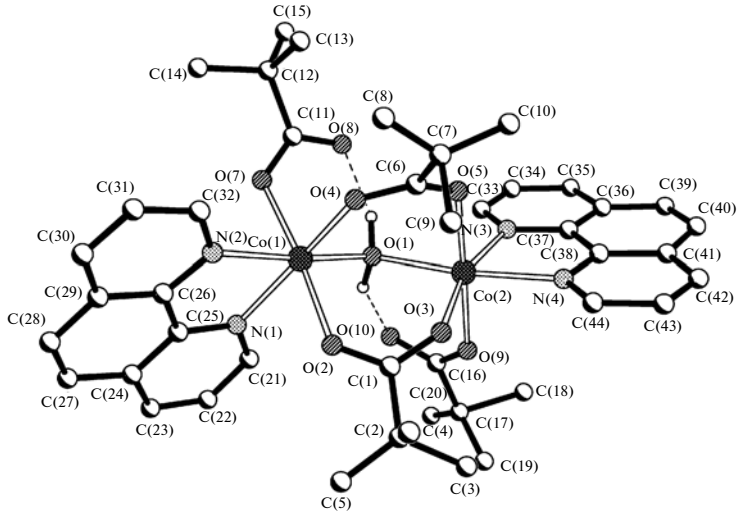

Fig. 1. Structure of complex **1a**.

Table 6. Atomic coordinates ($\times 10^4$) and equivalent isotropic factors ($Å^2 \times 10^3$) for complex **3a**

Atom	x	y	z	U_{eq}
Co(1)	7232(1)	1836(1)	9113(1)	20(1)
O(1)	6854(1)	2678(1)	8012(1)	26(1)
O(2)	5407(1)	2002(1)	8643(1)	27(1)
O(3)	6922(1)	2601(1)	10164(1)	26(1)
O(4)	7771(2)	3783(1)	9899(1)	33(1)
N(1)	9090(2)	1994(1)	9165(1)	24(1)
N(2)	9442(2)	2766(1)	9292(1)	27(1)
N(3)	6923(2)	855(1)	9992(1)	22(1)
N(4)	7228(2)	834(1)	8185(1)	22(1)
C(1)	5759(2)	2500(1)	8058(1)	23(1)
C(2)	4829(2)	2851(1)	7385(2)	29(1)
C(3)	3722(3)	3111(2)	7900(2)	53(1)
C(4)	4491(3)	2188(2)	6725(2)	66(1)
C(5)	5330(3)	3557(2)	6863(3)	70(1)
C(6)	7012(2)	3349(1)	10279(1)	23(1)
C(7)	6093(2)	3733(1)	10908(2)	29(1)
C(8)	6560(3)	4527(2)	11283(2)	48(1)
C(9)	4973(2)	3864(2)	10308(2)	51(1)
C(10)	5772(3)	3165(2)	11681(2)	43(1)
C(11)	10100(2)	1568(1)	9105(2)	28(1)
C(12)	10091(2)	687(2)	8950(2)	40(1)
C(13)	11093(2)	2075(2)	9196(2)	36(1)
C(14)	10640(2)	2832(2)	9324(2)	34(1)
C(15)	11233(3)	3627(2)	9478(2)	56(1)
C(16)	6763(2)	882(1)	10881(1)	26(1)
C(17)	6422(2)	214(1)	11395(2)	29(1)
C(18)	6226(2)	504(1)	10959(2)	28(1)
C(19)	6392(2)	556(1)	10015(2)	24(1)
C(20)	6746(2)	143(1)	9556(1)	21(1)
C(21)	6917(2)	130(1)	8591(1)	21(1)
C(22)	6209(2)	1275(1)	9497(2)	29(1)
C(23)	6401(2)	1293(1)	8589(2)	32(1)
C(24)	6752(2)	586(1)	8104(2)	25(1)
C(25)	6944(2)	570(1)	7160(2)	30(1)
C(26)	7255(2)	144(1)	6756(2)	32(1)
C(27)	7388(2)	828(1)	7287(2)	28(1)

RESULTS AND DISCUSSION

The reaction of aqueous 1,10-phenanthroline or 2,2'-dipyridyl with $Co_3(\mu\text{-}OOCBu^t)_6(NEt_3)_2$ in benzene at room temperature leads to the formation of orange binuclear complexes $L_2Co_2(\mu\text{-}OH_2)(\mu\text{-}OOCBu^t)_2(OOCBu^t)_2$ (L_2 = Phen (**1a**, yield 84%), Dipy (**1b**), yield 87%). According to X-ray crystallo-graphy, two cobalt atoms in **1a** are 3.5312(9) Å apart and are linked by the bridging water molecule (Co–O, 2.154(4), 2.132(4) Å) and two bridging pivalate ions (C–O, 2.072(5)–2.070(5) Å). The octahedral environment of each of the cobalt(II) atoms is completed with two nitrogen atoms of the coordinated phenanthroline molecule (2.137(6)–2.184(6) Å) and the oxygen atom of the terminal pivalate anion (Co–O, 2.087(5), 2.087(5) Å) forming short bonds to the hydrogen atoms of the bridging water molecule (O(1)···O 2.693(7), 2.966(7) Å).

The geometry of complex **1a** is close to that of complex **1b** (Co···Co 3.6041(9), Co–O_w 2.14(1) Å; Co–O, 2.07(1)–2.11(1) Å; Co–N, 2.07(2)–2.16(2) Å) [20].

The reaction of **1a** or **1b** with Hdmpz in benzene gives a mixture of orange red and yellow-red single crystals of the complexes $L_2(OOCBu^t)_2(Hdmpz)_2$ (**2**, L_2 = Phen (**a**), Dipy (**b**)) and $L_2(\eta\text{-}OOCBu^t)(OOCBu^t)(Hdmpz)$ (**3**, L_2 = Phen (**a**), Dipy (**b**)), the yield of which is determined by the ratio of the initial reagents. It should be noted that complexes **2a** and **2b**, as well as complexes **3a** and **3b**, have almost the same IR spectra.

According to X-ray crystallography, the cobalt(II) atom in complex **2b** (Fig. 2, Table 3) has a distorted octahedral environment composed of the four nitrogen atoms of two coordinated pyrazole molecules (Co–N, 2.143(3), 2.152(3) Å) and the bidentate dipyridyl molecule (Co–N, 2.164(3), 2.183(3) Å), as well as of two oxygen atoms of the terminal pivalate ions

Table 7. Selected bond lengths (d) and bond angles ω) for complex **3a**

Bond	d, Å	Bond	d, Å
Co(1)–O(3)	2.0345(14)	Co(1)–N(1)	2.0855(19)
Co(1)–N(3)	2.1153(17)	Co(1)–O(2)	2.1538(16)
Co(1)–N(4)	2.1550(17)	Co(1)–O(1)	2.1757(15)
Angle	**ω, deg**	**Angle**	**ω, deg**
O(3)Co(1)N(1)	94.19(7)	O(3)Co(1)N(3)	89.64(6)
N(1)Co(1)N(3)	104.29(7)	O(3)Co(1)O(2)	89.44(6)
N(1)Co(1)O(2)	157.82(6)	N(3)Co(1)O(2)	97.60(6)
O(3)Co(1)N(4)	164.99(6)	N(1)Co(1)N(4)	96.54(7)
N(3)Co(1)N(4)	77.56(7)	O(2)Co(1)N(4)	84.59(6)
O(3)Co(1)O(1)	97.02(6)	N(1)Co(1)O(1)	97.23(6)
N(3)Co(1)O(1)	156.95(6)	O(2)Co(1)O(1)	60.60(5)
N(4)Co(1)O(1)	92.03(6)		

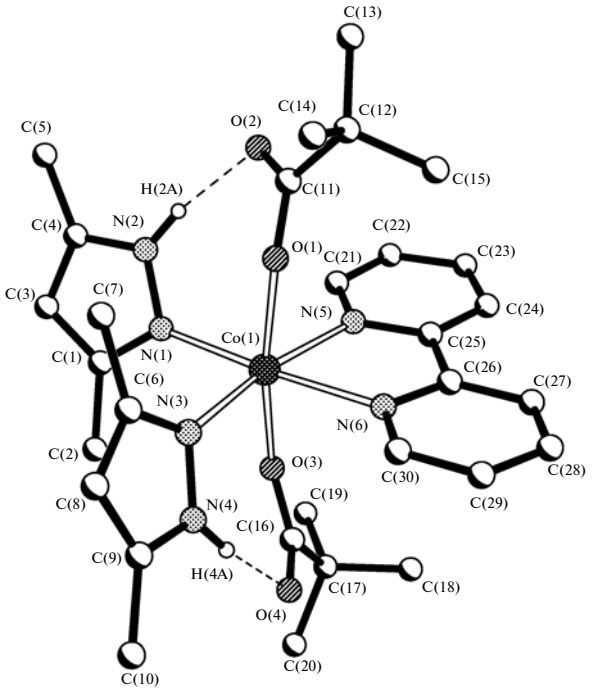

Fig. 2. Structure of complex **2b**.

(Co–O, 2.058(3), 2.078(3) Å). The terminal oxygen atoms of the pivalate ions form short hydrogen bonds with the NH moieties if the coordinated Hdmpz molecules (N···O 2.650(5), 2.680(5) Å).

As distinct from **2b**, according to X-ray crystallographic data, one of the pivalate ions in **3a** is η^2-coordinated, all metal–oxygen bonds (2.1538(16) and 2.1757(15) Å) being noticeably elongated as compared with the Co–O bond of the terminal pivalate ion (2.0345(14 Å). This may be responsible for the shorter Co–N distance (2.0855(19) Å) of the coordinated pyrazole molecule as compared with the distances observed in complex **2b**. Like the Co(II) atom in **2b**, the cobalt(II) atom in **3a** has a distorted octahedral environment completed with two nitrogen atoms of the phenanthroline molecule (Co–N, 2.1153(17), 2.1550(17) Å).

In complex **3a**, the hydrogen atom of the pyrrole moiety of the heterocycle forms a short hydrogen bond with the oxygen atom of the terminal carboxylate anion (N···O 2.680(3) Å).

The reaction of anhydrous cobalt(II) benzoate $Co_3(\mu\text{-}OOCPh)_6[OC(Ph)OHEt_3]$ with 1,10-phenanthroline in benzene and the subsequent addition of an Hdmpz excess to the resulting red solution lead to red-orange single crystals of the $PhenCo(Hdmpz)_2(OOCPh)_2$ complex (**4**, yield 75%). It is worth noting that this reaction does not yield com-

Table 8. Atomic coordinates ($\times 10^4$) and equivalent isotropic factors ($Å^2 \times 10^3$) for complex **4**

Atom	x	y	z	U_{eq}
Co(1)	3062(1)	2144(1)	2346(1)	29(1)
O(1)	4600(1)	1937(1)	2874(1)	38(1)
O(2)	4817(1)	3676(1)	2615(1)	43(1)
O(3)	1634(1)	2367(1)	1655(1)	35(1)
O(4)	1800(1)	523(1)	1697(1)	39(1)
N(1)	2822(1)	767(1)	3560(1)	32(1)
N(2)	2296(1)	77(1)	3479(1)	33(1)
N(3)	1957(1)	3521(1)	2992(1)	31(1)
N(4)	2419(1)	4222(1)	3150(1)	35(1)
N(5)	3419(1)	3407(1)	1056(1)	33(1)
N(6)	4354(1)	919(1)	1589(1)	33(1)
C(1)	2865(2)	531(2)	4487(1)	36(1)
C(2)	3341(2)	1155(2)	4879(1)	53(1)
C(3)	2354(2)	−303(2)	4979(1)	44(1)
C(4)	2005(2)	−579(2)	4322(1)	40(1)
C(5)	1386(2)	−1389(2)	4415(2)	63(1)
C(6)	862(2)	3806(2)	3436(1)	34(1)
C(7)	81(2)	3185(2)	3452(2)	45(1)
C(8)	643(2)	4694(2)	3858(1)	40(1)
C(9)	1655(2)	4932(2)	3668(1)	40(1)
C(10)	1960(2)	5789(2)	3934(2)	64(1)
C(11)	5234(2)	2551(2)	2692(1)	35(1)
C(12)	6576(2)	1842(2)	2576(1)	34(1)
C(13)	7035(2)	656(2)	2522(1)	42(1)
C(14)	8268(2)	−7(2)	2411(2)	54(1)
C(15)	9053(2)	519(2)	2346(2)	56(1)
C(16)	8615(2)	1691(2)	2400(2)	53(1)
C(17)	7382(2)	2359(2)	2510(1)	44(1)
C(18)	1487(2)	1665(2)	1317(1)	32(1)
C(19)	899(2)	2251(2)	380(1)	36(1)
C(20)	475(2)	1632(2)	52(2)	48(1)
C(21)	8(2)	2143(2)	−840(2)	60(1)
C(22)	−22(2)	3272(2)	−1403(2)	68(1)
C(23)	384(2)	3902(2)	−1082(2)	66(1)
C(24)	843(2)	3403(2)	−189(2)	49(1)
C(25)	2887(2)	4628(2)	763(1)	41(1)
C(26)	3217(2)	5348(2)	−90(2)	50(1)
C(27)	4144(2)	4778(2)	−625(2)	49(1)
C(28)	4734(2)	3487(2)	−343(1)	41(1)
C(29)	4316(2)	2831(2)	500(1)	32(1)
C(30)	4847(2)	1509(2)	801(1)	33(1)
C(31)	5732(2)	2803(2)	−858(2)	57(1)
C(32)	6261(2)	1568(2)	−559(2)	59(1)
C(33)	5826(2)	872(2)	275(2)	44(1)
C(34)	6301(2)	−416(2)	602(2)	55(1)
C(35)	5808(2)	−1010(2)	1384(2)	53(1)
C(36)	4834(2)	−302(2)	1859(1)	41(1)
C(37)	−5013(3)	6376(4)	4019(3)	127(1)
C(38)	−4426(3)	5642(3)	3457(3)	108(1)
C(39)	−3329(3)	5568(2)	3068(2)	86(1)
C(40)	−2798(3)	6193(2)	3249(2)	86(1)
C(41)	−3380(3)	6921(3)	3808(2)	95(1)
C(42)	−4480(4)	7020(3)	4201(3)	110(1)

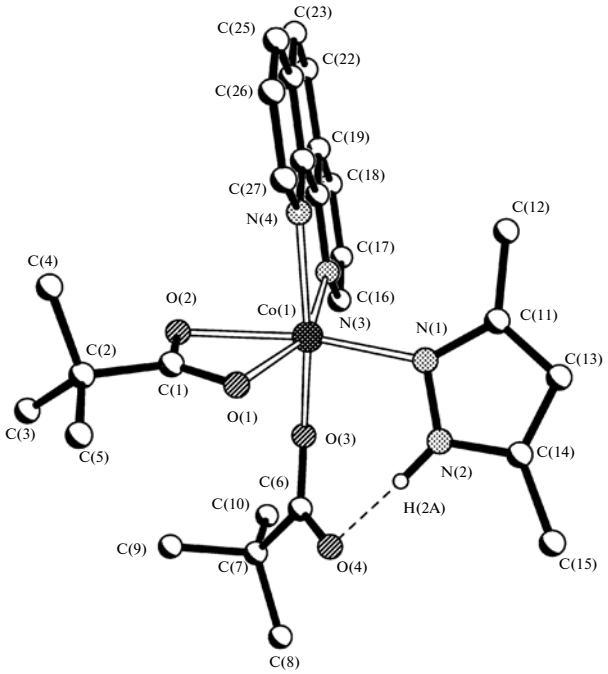

Fig. 3. Structure of complex **3a**.

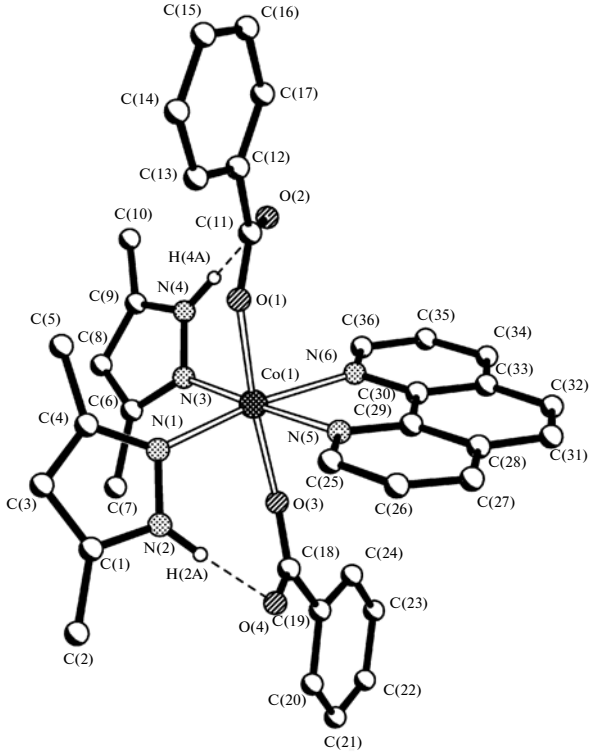

Fig. 4. Structure of complex **4**.

plexes analogous to compound **3**, which is likely due to the lower basicity of the anion of the strong benzoic acid as compared with the anion of weak pivalic acid.

Complex **4** can be obtained in nearly quantitative yield by the reaction of mononuclear tetrahedral Co(II) benzoate $Co(OOCPh)_2(Hdmpz)_2$ in benzene with phenanthroline at room temperature.

According to X-ray crystallographic data, the cobalt(II) atom in compound **4** (Fig. 4, Tables 8, 9), containing a solvate benzene molecule, gas a distorted

Table 9. Selected bond lengths (*d*) and bond angles ω) for complex **4**

Bond	*d*, Å	Bond	*d*, Å
Co(1)–O(1)	2.0713(13)	Co(1)–O(3)	2.0798(13)
Co(1)–N(3)	2.1164(15)	Co(1)–N(1)	2.1235(14)
Co(1)–N(6)	2.1595(14)	Co(1)–N(5)	2.1632(14)
Angle	ω, deg	Angle	ω, deg
O(1)Co(1)O(3)	172.89(5)	O(1)Co(1)N(3)	90.72(5)
O(3)Co(1)N(3)	93.95(5)	O(1)Co(1)N(1)	91.78(5)
O(3)Co(1)N(1)	93.58(5)	N(3)Co(1)N(1)	89.88(5)
O(1)Co(1)N(6)	83.73(5)	O(3)Co(1)N(6)	90.93(5)
N(3)Co(1)N(6)	170.75(6)	N(1)Co(1)N(6)	97.66(6)
O(1)Co(1)N(5)	87.90(5)	O(3)Co(1)N(5)	86.32(5)
N(3)Co(1)N(5)	95.52(6)	N(1)Co(1)N(5)	174.59(6)
N(6)Co(1)N(5)	76.94(6)		

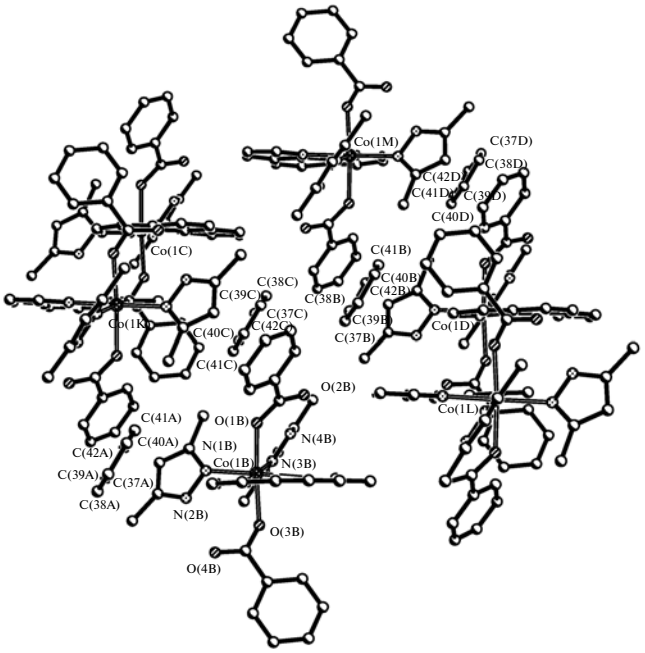

Fig. 5. Fragment of crystal packing of complex **4**.

octahedral environment formed by two oxygen atoms of benzoate anions (Co–O, 2.0713(13) Å, 2.0798(13) Å), two nitrogen atoms of the coordinates pyrazole molecules (Co–N, 2.1164(15), 2.1235(14) Å), and two phenanthroline nitrogen atoms (C–N, 2.1595(14), 2.1632(14) Å). In complex **4**, like in **2** and **3**, there are NH···O hydrogen bonds (2.695(3), 2.749(3) Å).

In the unit cell of complex **4**, the carbon atoms of the coordinated phenanthroline molecules form short stacking contacts (3.429(5)–3.472(5) Å) (Fig. 5).

In addition, it was found that complex **4** can also crystallize in another polymorphous modification (**4-1**), in which such stacking contacts are absent (Fig. 6, Tables 10, 11).

Such a crystals packing of molecules leads to only insignificant changes in bond lengths in complex **4** (Co–O, 2.0783(14), 2.0931(14) Å; Co–N_{pz}, 2.1320(19), 2.1510(19), 2.1510(19) Å; Co–N_{phen}, 2.1699(19), 2.1816(18) Å).

Thus, our study, demonstrates that, independently of the donor nature of the substituent in the carboxylate ion, in the presence of strong bidentate *n* donors, 3,5-dimethylpyrazole is not deprotonated, as distinct from carboxylates incorporating monodentate triethylamine, which cam also act as the proton acceptor.

Table 10. Atomic coordinates ($\times 10^4$) and equivalent isotropic factors ($Å^2 \times 10^3$) for complex **4** (**4-1**)

Atom	*x*	*y*	*z*	U_{eq}
Co(1)	2768(1)	1959(1)	4345(1)	28(1)
O(1)	2812(1)	3091(2)	3622(1)	34(1)
O(2)	4015(1)	2608(2)	3348(1)	33(1)
O(3)	2818(1)	923(2)	5114(1)	32(1)
O(4)	1798(1)	1523(2)	5568(1)	42(1)
N(1)	1432(1)	1735(2)	4072(1)	36(1)
N(2)	946(1)	1732(2)	4475(1)	33(1)
N(3)	2948(1)	156(2)	3924(1)	33(1)
N(4)	3471(1)	121(2)	3546(1)	35(1)
N(5)	2719(1)	3774(2)	4822(1)	32(1)
N(6)	4081(1)	2325(2)	4741(1)	28(1)
C(1)	136(1)	1494(2)	4245(1)	36(1)
C(2)	−520(2)	1436(3)	4602(1)	46(1)
C(3)	88(2)	1349(3)	3662(1)	44(1)
C(4)	902(2)	1496(3)	3570(1)	41(1)
C(5)	1199(2)	1405(3)	3016(1)	55(1)
C(6)	2583(2)	−1036(2)	3885(1)	41(1)
C(7)	1987(2)	−1424(3)	4258(1)	59(1)
C(8)	2872(2)	−1797(2)	3477(1)	48(1)
C(9)	3448(2)	−1036(2)	3273(1)	44(1)
C(10)	4003(2)	−1331(3)	2856(1)	67(1)
C(11)	3352(1)	3253(2)	3312(1)	28(1)
C(12)	3146(1)	4291(2)	2844(1)	31(1)
C(13)	2338(2)	4771(2)	2677(1)	41(1)
C(14)	2140(2)	5642(3)	2218(1)	56(1)
C(15)	2742(2)	6053(3)	1931(1)	57(1)
C(16)	3561(2)	5607(3)	2101(1)	50(1)
C(17)	3757(2)	4721(2)	2557(1)	39(1)
C(18)	2463(1)	935(2)	5544(1)	30(1)
C(19)	2895(1)	136(2)	6057(1)	30(1)
C(20)	2448(2)	−306(3)	6460(1)	46(1)
C(21)	2834(2)	−1131(3)	6907(1)	57(1)
C(22)	3661(2)	−1487(3)	6960(1)	49(1)
C(23)	4113(2)	−1026(3)	6571(1)	42(1)
C(24)	3729(2)	−220(2)	6118(1)	35(1)
C(25)	2051(2)	4506(3)	4845(1)	43(1)
C(26)	2088(2)	5608(3)	5210(1)	56(1)
C(27)	2828(2)	5941(3)	5561(1)	54(1)
C(28)	3550(2)	5200(2)	5546(1)	41(1)
C(29)	3464(2)	4124(2)	5165(1)	32(1)
C(30)	4185(1)	3350(2)	5124(1)	28(1)
C(31)	4363(2)	5498(3)	5894(1)	49(1)
C(32)	5037(2)	4772(3)	5852(1)	46(1)
C(33)	4971(2)	3674(2)	5469(1)	36(1)
C(34)	5659(2)	2887(3)	5415(1)	41(1)
C(35)	5550(2)	1850(2)	5037(1)	39(1)
C(36)	4748(1)	1610(2)	4706(1)	32(1)
C(37)	127(2)	4808(4)	6056(2)	78(1)
C(38)	−371(3)	4841(4)	6468(2)	87(1)
C(39)	−474(2)	3722(4)	6757(1)	74(1)
C(40)	−105(2)	2563(4)	6656(1)	62(1)
C(41)	393(2)	2544(3)	6244(1)	55(1)
C(42)	499(2)	3662(3)	5949(1)	59(1)

Table 11. Selected bond lengths (*d*) and bond angles ω) for complex **4** (**4-1**)

Bond	*d*, Å	Bond	*d*, Å
Co(1)–O(1)	2.0783(14)	Co(1)–O(3)	2.0931(14)
Co(1)–N(3)	2.1320(19)	Co(1)–N(1)	2.1510(19)
Co(1)–N(5)	2.1699(19)	Co(1)–N(6)	2.1816(18)
Angle	ω, deg	Angle	ω, deg
O(1)Co(1)O(3)	174.72(6)	O(1)Co(1)N(3)	93.13(6)
O(3)Co(1)N(3)	89.68(6)	O(1)Co(1)N(1)	90.68(6)
O(3)Co(1)N(1)	93.81(6)	N(3)Co(1)N(1)	89.71(7)
O(1)Co(1)N(5)	88.56(6)	O(3)Co(1)N(5)	88.20(6)
N(3)Co(1)N(5)	174.07(7)	N(1)Co(1)N(5)	95.96(7)
O(1)Co(1)N(6)	93.84(6)	O(3)Co(1)N(6)	81.35(6)
N(3)Co(1)N(6)	97.57(7)	N(1)Co(1)N(6)	171.21(7)
N(5)Co(1)N(6)	76.64(7)		

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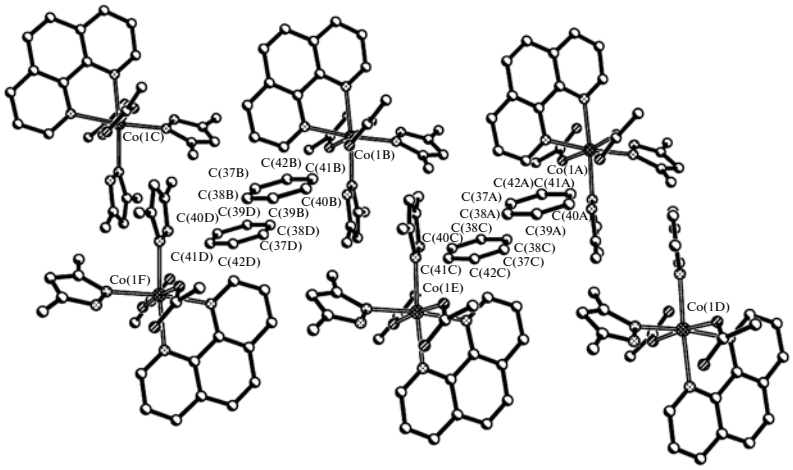

Fig. 6. Fragment of crystal packing of complex **4** in polymorph **4-1**.

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