

Complexes of Zinc, Nickel(II), and Cobalt(II) Cymantrenate Complexes with Pyrazole and 3,5-Dimethylpyrazole: Synthesis and Structural Features

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Abstract—The reaction of cymantrenates $M[(OOC_5H_4)Mn(CO)_3]_2(MeOH)_4$ ($M = Zn, Co(II), Ni(II)$) with pyrazole (HPz) results in replacement of labile methanol molecules by the heterocyclic ligand and gives mononuclear complexes $Zn[(OOC_5H_4)Mn(CO)_3]_2(HPz)_2$ (**I**), $Ni[(OOC_5H_4)Mn(CO)_3]_2(HPz)_4$ (**II**), and $Co[(OOC_5H_4)Mn(CO)_3]_2(HPz)_4$ (**III**). A similar reaction of cobalt cymantrenate with more basic and sterically bulky 3,5-dimethylpyrazole (HDmpz) gives the complex $Co[(OOC_5H_4)Mn(CO)_3]_2(HDmpz)_2$ (**IV**). Compounds **I–IV** were characterized by X-ray diffraction (CCDC nos. 2157671 (**I**), 2157672 (**II**), 2157669 (**III**), and 2157670 (**IV**)), IR spectroscopy, and elemental analysis.

Key words: cymantrenates, X-ray diffraction, cobalt, nickel, zinc, pyrazole, heterometallic complexes

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INTRODUCTION

Development of synthetic approaches to the preparation of heterometallic compounds is a relevant trend of modern coordination chemistry [1–3]. By combination of moieties with various natures of metal centers, it is possible to manufacture materials with unique physicochemical properties [4–10]. There are several approaches to the assembly of heterometallic complexes; one of them is to obtain transition metal carboxylates containing a heterometal atom in the carboxylate anion (cymantrene, chromocene, ferrocene) [11–20]. An additional ligand such as pyrazole also plays a noticeable role in the assembly of heterometallic compounds.

When reacting with transition metal carboxylates, pyrazole can be either coordinated in the monodentate fashion or deprotonated and form pyrazolate-bridged complexes, in some cases, coordination polymers [21–28]. Specific features of the reaction and the composition and structure of the products depend on the nature of the transition metal, substituent R in the carboxylate anion, and substituents in the pyrazole molecule [29–33]. In particular, reactions of manganese(II) cymantrenates with pyrazole and 3,5-dimethylpyrazole do not result in its deprotonation [19–30], while cobalt(II) and zinc carboxylates (pivalates, acetates, benzoates) are converted to pyrazolate-bridged complexes [34–36].

Previously, complexes of cobalt, zinc, nickel, and manganese cymantrenates with four coordinated methanol molecules $M[(OOC_5H_4)Mn(CO)_3]_2(MeOH)_4$ ($M = Zn, Co(II), Ni(II), Mn(II)$) were prepared [37–40]. This communication reports the synthesis and structure of the reaction products formed from zinc, nickel, and cobalt cymantrenates with pyrazole and 3,5-dimethylpyrazole.

EXPERIMENTAL

Commercial methanol, acetonitrile, chloroform, and heptane (Khimmed) and pyrazole and 3,5-dimethylpyrazole (ACROS) were used. The initial complexes $M[(OOC_5H_4)Mn(CO)_3]_2(MeOH)_4$ ($M = Zn, Co(II), Ni(II)$) were synthesized by reported procedures [37–40]; and cymantrenecarboxylic acid was synthesized by the procedure reported in [41].

Synthesis of $Zn[(OOC_5H_4)Mn(CO)_3]_2(HPz)_2$ (I**).** Pyrazole (0.04 g, 0.6 mmol) was added to a solution of pyrazole $Zn[(OOC_5H_4)Mn(CO)_3]_2(MeOH)_4$ (0.1 g, 0.15 mmol) in chloroform (10 mL), and the reaction mixture was refluxed for 1 h. Hexane (2 mL) was added to the resulting solution and the mixture was kept in a refrigerator (5°C) for 24 h. The white crystals thus formed were separated from the mother liquor by

[†] Deceased.

decantation and dried in an argon flow. The yield was 0.1 g (80%).

For $C_{24}H_{16}Mn_2N_4O_{10}Zn$

Anal. calcd., %	C, 41.43	H, 2.32	N, 8.05
Found, %	C, 41.25	H, 2.48	N, 8.12

IR (ν , cm^{-1}): 2961 w, 2854 w, 2021 s, 1928 s, 1580 m, 1482 m, 1390 s, 1362 s, 1259 s, 1189 m, 1067 s, 1013 s, 954 w, 864 w, 793 s, 746 s, 703 w, 665 m, 629 s, 538 m, 464 m, 430 w, 421 w, 411 m.

Synthesis of $Ni[(OCC_5H_4)Mn(CO)_3]_2(HPz)_4$ (II). Pyrazole (0.04 g, 0.6 mmol) was added to a solution of $Ni[(OCC_5H_4)Mn(CO)_3]_2(MeOH)_4$ (0.1 g, 0.15 mmol) in chloroform (10 mL) and the reaction mixture was refluxed for 1 h. Heptane (5 mL) was added to the resulting solution and the mixture was kept in a refrigerator (5°C) for 24 h. The bright green crystals thus formed were separated from the mother liquor by decantation and dried in an argon flow. The yield was 76% (0.095 g).

For $C_{30}H_{24}N_8O_{10}Mn_2Ni$

Anal. calcd., %	C, 43.66	H, 2.93	N, 13.58
Found, %	C, 43.35	H, 2.98	N, 13.18

IR (ν , cm^{-1}): 3134 w, 2893 w, 2013 s, 1924 s, 1570 m, 1536 m, 1474 s, 1390 s, 1355 s, 1264 m, 1211 w, 1167 w, 1136 m, 1047 s, 1059 s, 1028 m, 934 m, 854 w, 840 m, 815 m, 755 s, 663 m, 630 s, 604 s, 540 s, 488 m, 459 m, 441 w.

Synthesis of $Co[(OCC_5H_4)Mn(CO)_3]_2(HPz)_4$ (III). Pyrazole (0.04 g, 0.6 mmol) was added to a solution of $Co[(OCC_5H_4)Mn(CO)_3]_2(MeOH)_4$ (0.1 g, 0.15 mmol) in chloroform (10 mL) and the reaction mixture was refluxed for 1 h. Heptane (5 mL) was added to the resulting solution and the mixture was allowed to stand at room temperature for 24 h. The violet crystals thus formed were separated from the mother liquor by decantation and dried in an argon flow. The yield was 89% (0.11 g).

For $C_{30}H_{24}N_8O_{10}Mn_2Co$

Anal. calcd., %	C, 43.66	H, 2.93	N, 13.58
Found, %	C, 43.98	H, 2.95	N, 13.77

IR (ν , cm^{-1}): 3210 w, 2965 w, 2022 s, 1930 s, 1588 w, 1563 m, 1483 m, 1416 w, 1393 m, 1355 s, 1298 w, 1258 s, 1159 m, 1124 s, 1054 m, 1024 s, 988 w, 910 w, 836 w, 826 m, 797 s, 717 w, 665 m, 625 s, 565 m, 531 s, 475 m, 428 w.

Synthesis of $Co[(OCC_5H_4)Mn(CO)_3]_2(HDmpz)_2$ (IV). 3,5-Dimethylpyrazole (0.028 g, 0.3 mmol) was added to a solution of $Co[(OCC_5H_4)Mn(CO)_3]_2(MeOH)_4$ (0.1 g, 0.15 mmol) in chloroform (10 mL) and the reaction mixture was refluxed for 1 h.

Heptane (5 mL) was added to the resulting solution and the mixture was allowed to stand at room temperature for 24 h. The blue crystals thus formed were separated from the mother liquor by decantation and dried in an argon flow. The yield was 85% (0.095 g).

For $C_{28}H_{24}N_4O_{10}Mn_2Co$

Anal. calcd., %	C, 45.12	H, 3.25	N, 7.51
Found, %	C, 44.58	H, 3.42	N, 7.62

IR (ν , cm^{-1}): 3181 w, 2863 w, 2017 s, 1932 s, 1919 c, 1553 s, 1536 m, 1472 m, 1393 s, 1356 s, 1393 s, 1292 m, 1193 w, 1027 s, 1027 s, 930 m, 815 m, 792 s, 662 m, 630 s, 537 s, 509 w, 449 m, 418 w.

Elemental analysis was carried out on a Carlo Erba EA 1108 automated C,H,N-analyzer. IR spectra were recorded on a Perkin-Elmer Spectrum 65 FTIR spectrometer in the attenuated total reflection (ATR) mode in the frequency range of 400–4000 cm^{-1} .

The single crystals of the complexes for X-ray diffraction study were taken directly from the mother liquor and rapidly transferred to a stream of evaporating liquid nitrogen.

X-ray diffraction analysis of I–IV was carried out by a standard procedure on a Bruker SMART Apex II automated diffractometer equipped with a CCD array detector (MoK_{α} radiation, $\lambda = 0.71073$ Å, graphite monochromator, ω -scan mode). The structures were refined using the SHELXTL PLUS program package (PC version) [42–46]. The crystallographic data and structure refinement details for I–IV are summarized in Table 1, and bond lengths and bond angles are in Tables 2 and 3.

Additional crystallographic data for structures I–IV are deposited with the Cambridge Crystallographic Data Centre (CCDC nos. 2157671 (I), 2157672 (II), 2157669 (III), 2157670 (IV); <http://www.ccdc.cam.ac.uk/>).

RESULTS AND DISCUSSION

The reaction of the mononuclear complex $Zn[(OCC_5H_4)Mn(CO)_3]_2(MeOH)_4$ with an excess of pyrazole (HPz) on refluxing in chloroform affords the mononuclear complex $Zn[(OCC_5H_4)Mn(CO)_3]_2(HPz)_2$ (I) (in 80% yield).

According to X-ray diffraction data, as a result of removal of four labile methanol molecules, the zinc in I occurs in a tetrahedral environment of two oxygen atoms belonging to cymantrenate anions ($Zn-O$, 1.938(6)–1.971(6) Å) and two nitrogen atoms belonging to coordinated pyrazole molecules ($Zn-N$, 1.984(7)–2.009(8) Å) (Fig. 1). In the crystal cell, the molecules of complex I are bound into a linear polymer via intermolecular hydrogen bonds between the oxygen atoms of the cymantrenate anions and the NH

Table 1. Crystallographic data and X-ray experiment and structure refinement details for **I–IV**

Parameter	Value			
	I	II	III	IV
Molecular formula	C ₂₄ H ₁₆ N ₄ O ₁₀ ZnMn ₂	C ₃₀ H ₂₄ N ₈ O ₁₀ Mn ₂ Ni	C ₃₀ H ₂₄ N ₈ O ₁₀ Mn ₂ Co	C ₂₈ H ₂₄ N ₄ O ₁₀ Mn ₂ Co
<i>M</i>	695.68	825.16	825.38	745.32
Temperature, K	150(2)			
Color	Colorless	Bright green	Pink	Pink
System	Triclinic			
Space group	<i>P</i> $\bar{1}$			
<i>a</i> , Å	10.212(3)	8.0100(8)	14.394(4)	11.2348(5)
<i>b</i> , Å	12.222(3)	14.2896	15.846(5)	11.4078(5)
<i>c</i> , Å	22.747(6)	15.8650(16)	17.549(8)	13.0979(5)
α , deg	98.947(4)	108.285(2)	99.890(6)	97.4780(10)
β , deg	99.111(4)	93.491(2)	107.245(6)	103.9410(10)
γ , deg	90.153(4)	95.334(2)	111.063(4)	102.8800(10)
<i>V</i> , Å ³	2768.06(120)	1708.9(3)	3389(2)	1557.97(11)
<i>Z</i>	4	2	4	2
ρ (calcd.), mg/m ³	1.66918	1.604	1.618	1.589
μ , mm ^{−1}	1.819	1.340	1.286	1.385
<i>F</i> (000)	1392	836	1668	754
Crystal size, mm	0.22 × 0.2 × 0.18	0.2 × 0.18 × 0.16	0.24 × 0.22 × 0.2	0.24 × 0.22 × 0.2
Scanning range of θ , deg	3.38–53.994	2.716–59.994	2.558–59.998	5.418–59.998
Ranges of reflection indices	−14 ≤ <i>h</i> ≤ 14, −17 ≤ <i>k</i> ≤ 17, −32 ≤ <i>l</i> ≤ 32	−11 ≤ <i>h</i> ≤ 11, −19 ≤ <i>k</i> ≤ 18, 0 ≤ <i>l</i> ≤ 22	−20 ≤ <i>h</i> ≤ 20, −22 ≤ <i>k</i> ≤ 22, −24 ≤ <i>l</i> ≤ 24	−15 ≤ <i>h</i> ≤ 15, −15 ≤ <i>k</i> ≤ 16, −18 ≤ <i>l</i> ≤ 18
Number of measured reflections	26012	20372	30905	18422
Number of unique reflections (<i>R</i> _{int})	12008 (0.0610)	9903 (0.0765)	18536 (0.0700)	8999 (0.0171)
GOOF	1.043	0.789	0.759	1.027
<i>R</i> (<i>I</i> > 2σ(<i>I</i>))	<i>R</i> ₁ = 0.0834, <i>wR</i> ₂ = 0.2173	<i>R</i> ₁ = 0.0434, <i>wR</i> ₂ = 0.0710	<i>R</i> ₁ = 0.0501, <i>wR</i> ₂ = 0.0692	<i>R</i> ₁ = 0.0293, <i>wR</i> ₂ = 0.0783
<i>R</i> (for all reflections)	<i>R</i> ₁ = 0.1164, <i>wR</i> ₂ = 0.2475	<i>R</i> ₁ = 0.0777, <i>wR</i> ₂ = 0.0783	<i>R</i> ₁ = 0.1410, <i>wR</i> ₂ = 0.0822	<i>R</i> ₁ = 0.0356, <i>wR</i> ₂ = 0.0820
Electron density (max/min), e Å ^{−3}	2.77/−1.41	0.69/−0.64	0.61/−0.82	0.50/−0.34

hydrogen atoms of the coordinated heterocycle (N...O, 2.735–2.957 Å).

The reactions of methanol-containing nickel and cobalt cymantrenates M[(OCC₅H₄)Mn(CO)₃]₂–(MeOH)₄ (M = Co(II), Ni(II)) with PzH on refluxing in chloroform give isostructural mononuclear complexes Ni[(OCC₅H₄)Mn(CO)₃]₂(HPz)₄ (**II**) (76% yield) and Co[(OCC₅H₄)Mn(CO)₃]₂(HPz)₄ (**III**) (89% yield), in which all four labile methanol mole-

cules have been replaced by four pyrazole molecules. According to X-ray diffraction, the central metal atom in **II** retains the octahedral environment typical of Ni(II), which consists of four pyrazole nitrogen atoms (Ni–N, 2.091(3)–2.119(2) Å) and two carboxylate oxygen atoms (Ni–O, 2.091(2) Å) (Fig. 2).

According to X-ray diffraction data, in the centrosymmetric complex Co[(OCC₅H₄)Mn(CO)₃]₂–(HPz)₄ (**III**) (Fig. 3), the cobalt atom also has an octa-

Table 2. Selected bond lengths and bond angles in complexes **I** and **II**

Bond	<i>d</i> , Å	Bond	<i>d</i> , Å
I			
Zn(1)–O(3)	1.940	Zn(1)–N(3)	1.972
Zn(1)–O(1)	1.970	Zn(1)–N(1)	2.017
II			
Ni(1)–O(1)	2.0910	Ni(1)–O(1A)	2.0910
Ni(1)–N(3)	2.0915	Ni(1)–N(3A)	2.0915
Ni(1)–N(1)	2.1191	Ni(1)–N(1A)	2.1191
Angle	ω, deg	Angle	ω, deg
I			
Zn(1)O(3)O(1)	98.263	Zn(1)O(1)N(3)	113.067
Zn(1)O(3)N(3)	111.798	Zn(1)O(1)N(1)	101.639
Zn(1)O(3)N(1)	113.862	Zn(1)N(3)N(1)	116.308
II			
Ni(1)O(1)O(1A)	180.000	Ni(1)N(3A)N(3)	180.000
Ni(1)O(1)N(3)	94.244	Ni(1)N(3)N(1)	91.142
Ni(1)O(1)N(3A)	85.756	Ni(1)N(3A)N(1)	88.858
Ni(1)O(1)N(1)	95.851	Ni(1)N(1A)N(1)	180.000
Ni(1)O(1A)N(1)	84.149	Ni(1)N(3)N(1A)	91.142

hedral environment consisting of four nitrogen atoms belonging to coordinated pyrazole molecules (Co–N, 2.102(4)–2.134(5) Å) and two oxygen atoms of the cymantrenate anions (Co–O, 2.161(3) Å). In the crystals of **II** and **III**, intramolecular hydrogen bonds are formed between the carboxylate oxygen atoms and hydrogen atoms of the coordinated pyrazole molecules (NH...O, 2.751–2.833 Å).

When the methanol cobalt(II) cymantrenate reacts with 3,5-dimethylpyrazole under the same conditions, the mononuclear complex $\text{Co}[(\text{OCC}_5\text{H}_4)\text{Mn}(\text{CO})_3]_2(\text{HDmpz})_2$ (**IV**) is formed (Fig. 4). The introduction of electron-donating methyl groups into positions 3 and 5 of the heterocycle increases the basicity

of the pyridine nitrogen atom compared to that in pyrazole and, hence, decreases the acidity of pyrrole hydrogen atoms. This has a considerable effect on the packing of molecules in the crystal, in which intramolecular NH...O hydrogen bonds are present, and the crystal is formed via unusual π – π contacts.

In complex **IV**, the cobalt(II) atom, unlike that in **III**, has a tetrahedral environment of two cymantrenate oxygen atoms and two nitrogen atoms of

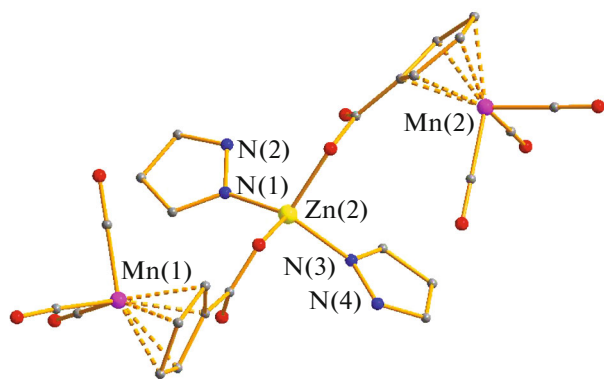
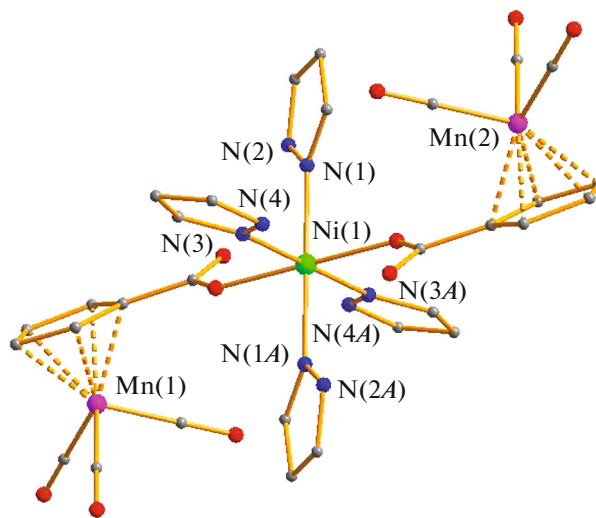
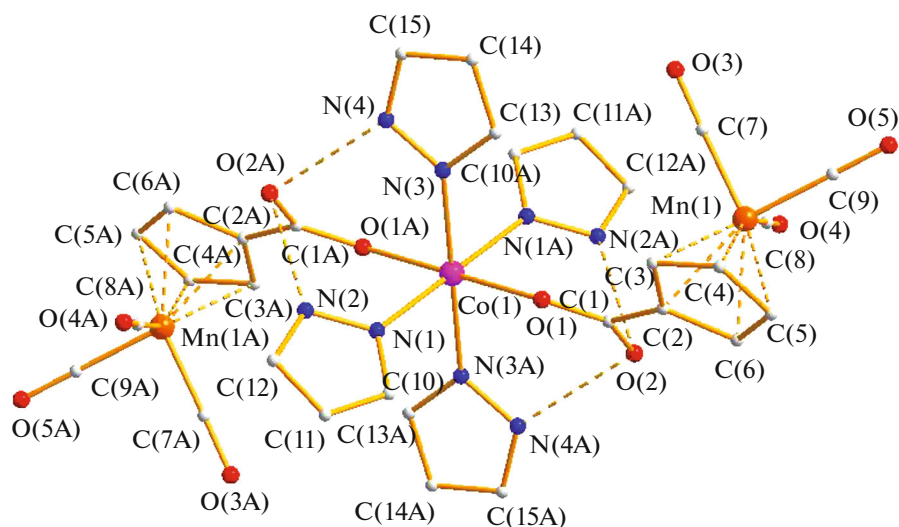
**Fig. 1.** Independent part of complex **I**.**Fig. 2.** Structure of complex **II**.

Table 3. Selected bond lengths and bond angles in complexes **III** and **IV**

Bond	<i>d</i> , Å	Bond	<i>d</i> , Å
III			
Co(1)–N(1)	2.1029	Co(1)–N(3A)	2.1342
Co(1)–N(1A)	2.1029	Co(1)–O(1)	2.1601
Co(1)–N(3)	2.1342	Co(1)–O(1A)	2.1601
IV			
Co(1)–O(1)	1.9324	Co(1)–N(3)	2.0053
Co(1)–O(3)	1.9420	Co(1)–N(1)	2.0176
Angle	ω, deg	Angle	ω, deg
III			
Co(1)N(1)N(1A)	180.000	Co(1)N(1A)O(1)	88.870
Co(1)N(1)N(3)	93.357	Co(1) N(1A)O(1A)	91.130
Co(1)N(1A)N(3)	86.643	Co(1)N(3)N(3A)	180.000
Co(1)N(1)O(1)	91.130	Co(1)N(3)O(1)	94.844
Co(1)N(1)O(1A)	88.870	Co(1)N(3)O(1A)	85.156
Co(1)N(1)N(3A)	86.643	Co(1)N(3A)O(1)	85.156
Co(1)N(1)N(3A)	93.357	Co(1)O(1)O(1A)	180.000
IV			
Co(1)O(1)O(3)	110.147	Co(1)O(3)N(1)	102.681
Co(1)O(1)N(3)	107.927	Co(1)N(3)N(1)	108.727
Co(1)N(1)O(1)	112.142	Co(1)O(1)O(3)	110.147
Co(1)O(3)N(3)	115.233		

the coordinated heterocycles. The tetrahedral environment of the central atom is unsymmetrically distorted: the Co–O bond lengths are 1.932 and 1.942 Å, while the M–N bond lengths differ to a greater extent (2.018 and 2.005 Å). Among strong non-covalent

interactions, mention should be made of the standard intramolecular N–H...O hydrogen bonds and strong intermolecular stacking interactions between the pyrazole molecule and the cyclopentadienyl ring of the cymantrenecarboxylate anion (C(6)...C(25), ~3.17 Å).

**Fig. 3.** Structure of complex **III**.

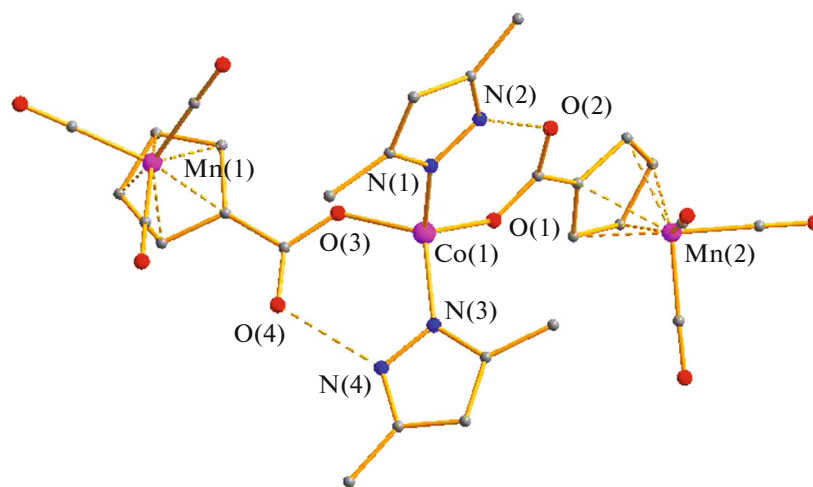


Fig. 4. Independent part of complex IV.

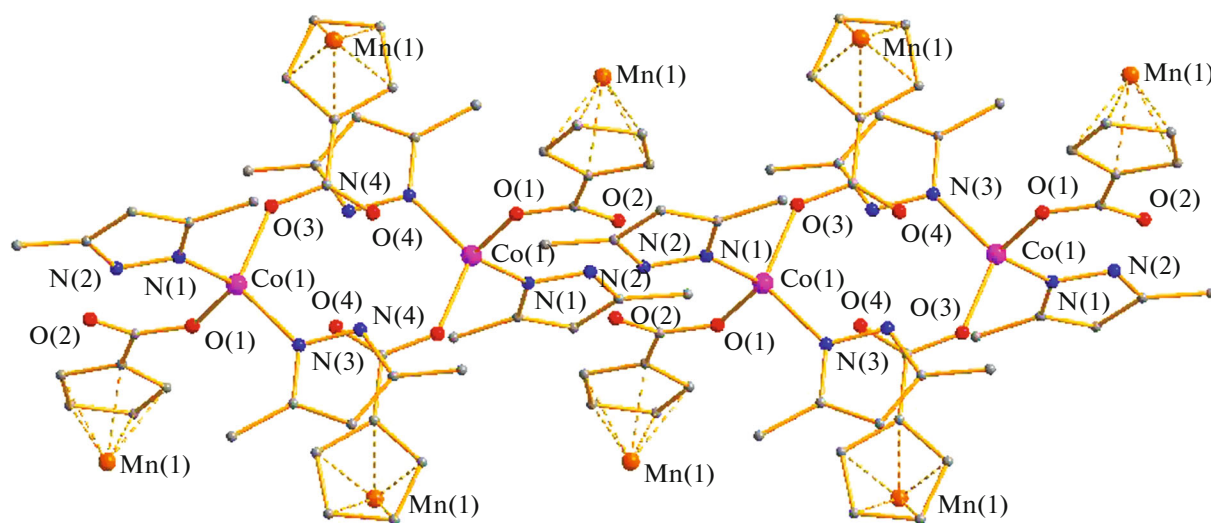


Fig. 5. Fragment of packing of complex IV in the crystal.

These stacking interactions (two per two molecules) give rise to centrosymmetric dimers of the complex molecules; the geometry of these dimers is such that the O(4) atom of one complex molecule is located above the M–N(1) bond of the other molecule (the corresponding O(4)CoN(1) angle is $\sim 176^\circ$; the M...O(4) distance is 3.595(1) Å (Fig. 5).

Thus, it was found that the pyrazole and 3,5-dimethylpyrazole molecules are not deprotonated on treatment with nickel, zinc, and cobalt cymantrenates, but are coordinated in the monodentate fashion, thus forming mononuclear complexes, which are additionally stabilized by hydrogen bonds.

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