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Synthesis and Molecular Structures of Cymantrenecarboxylate Derivatives of Titanium(IV) and Vanadium(III) Cyclopentadienyl Complexes and of Copper(II) and Manganese(II) Lutidine Complexes

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Abstract—The reactions of dimethyltitanocene borohydride and vanadocene with cymantrenecarboxylic acid CymCOOH gave the monomer $(C_5H_4Me)_2Ti(OOCCym)_2$ (**I**) and dimer $(C_5H_5)V(OOCCym)_4V(C_5H_5)$ (**II**), respectively. Treatment of Cu(II) cymantrenylcarboxylate with excess lutidine gives the monomer $(C_7H_9N)_2Cu(COOCym)_2$ (**III**). The reaction of lutidine with a mixture of Cu(II) and Mn(II) bis-cymantrenecarboxylates affords the heterometallic trinuclear complex $(C_7H_9N)Cu(OOCCym)_3Mn(OOCCym)_3Cu(C_7H_9N)$ (**IV**). The structures of **I**–**IV** were established by X-ray diffraction. In **I** and **III**, the cymantrenecarboxylate groups are terminal and in **II** and **IV**, they are bridging, which is also manifested as characteristic OCO stretching bands in the IR spectra.

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INTRODUCTION

Transition metal carboxylates, which are traditional subjects of coordination chemistry, are distinguished by a diversity of coordination modes of carboxyl groups RCOO depending on the electronic and steric characteristics of R and on the nature of metal atom and the ligands it bears. It was shown previously that vanadocene reacts with benzoyl peroxide without heating to give mononuclear vanadocene-bis-benzoate $Cp_2V(OOCPh)_2$ with terminal carboxylate groups [1]; this product, however, readily loses one cyclopentadienyl ligand to give the binuclear complex $CpV(OOCPh)_4VCp$ with four carboxylate bridges. The same compound is formed in the reaction of vanadocene or vanadium cyclopentadienyl tetracarbonyl with benzoic acid on heating [2, 3]. Similarly, heating of vanadocene or vanadium cyclopentadienyl tetracarbonyl with carboxylic acids gives the dimers $CpV(OOCR)_4VCp$, R = CF_3 [4], CMe_3 [5], furyl [6], which were characterized by X-ray diffraction, mass spectrometry [7], magnetochemistry, and ESR [8].

The complex $Cp_2Ti(OOCPh)_2$ with terminal carboxyl groups was fairly stable, so that binuclear monocyclopentadienyl complexes, "lanterns" $CpTi(OOCPh)_4TiCp$, were formed only in the reactions of monocyclopentadienyl titanium complexes, for example, from $CpTi(BH_4)_2$ and benzoic acid [9].

For Cu(II), lantern-type dimers $LCu(OOCR)_4CuL$ are most typical, irrespective of the nature of R, at different L (from water molecules to triphenylphosphine) [10]. However, the steric effects of *ortho*-disubstituted pyridine ligands (quinaldine, acridine, and 2,6-lutidine (Lut = C_6H_9N)) strongly affect the geometry and properties of binuclear complexes [11, 12], in particular, resulting in dissociation of binuclear chromium trifluoroacetate to give unusual square monomer $(Lut)_2Cr(OOCCF_3)_2$ [13].

Finally, polymeric manganese carboxylates have typically octahedral environment of six carboxylate bridges [14].

Since heterometallic carboxylates are promising as precursors for the preparation of mixed-metal nanosized oxides and intermetallic compounds, it appeared of interest to continue studies of complexes of cymantrenecarboxylic acid containing a $C_5H_4Mn(CO)_3$ group as the radical. Previously we studied binuclear copper complexes of this type $LCu(CymCOO)_4CuL$, where L = THF, Et_2O, CymCOOH, and PPh3 [15], and also nickel complexes $LNi(CymCOO)_4NiL$ (L = PPh_3), and the trinuclear cobalt complex $(Lut)Co(CymCOO)_3Co(CymCOO)_3Co(Lut)$ [16]. Furthermore, recently [17–19] cymantrenyl carboxylates of rare earth metals (Ce, Pr, Nd, Sm, Eu, and Gd) were reported.

In this study, lutidine ligands were used in copper and copper–manganese complexes, whereas the above-mentioned bulky cyclopentadienyl ligands served as partners for titanium and vanadium atoms.

EXPERIMENTAL

All reactions reported in this work were performed by the standard Schlenk procedure in an argon atmosphere using anhydrous solvents. The reactions were monitored by TLC and IR spectroscopy. IR spectra were recorded on a Bruker Alpha FT IR spectrometer. Elemental analysis was carried out on a Carlo-Erba CHNS analyzer. X-ray diffraction analysis was performed on an a Bruker APEX II CCD diffractometer. The compounds $(THF)_2Cu_2[(CO)_3MnC_5H_4COO)_4]_2$, $(CO)_3MnC_5H_4COOH$, $(MeCp)_2TiCl_2$, and $(C_5H_5)_2V$ were synthesized by procedures reported in [15, 20–22], respectively.

Synthesis of $(C_5H_4Me)_2Ti(OOCCym)_2$ (I). A. $(MeCp)_2TiCl_2$ (0.23 g, 0.8 mmol) and $NaBH_4$ (0.063 g, 1.6 mmol) were dissolved in THF (10 mL). The reaction mixture was stirred for 15 min, the color being changed from red to violet. The solvent was removed in a water-jet pump vacuum, the residue was extracted with benzene (20 mL), and then $(CO)_3MnC_5H_4COOH$ (0.41 g, 1.6 mmol) was added. The solution color changed to red-brown and a gas evolved. Heptane (1.5 mL) was added to the filtered solution and the mixture was concentrated until crystallization started. After keeping the solution at +5°C, red crystals were separated and dried. Yield 0.06 g (10%).

B. $(MeCp)_2TiCl_2$ (0.4 g, 1.4 mmol) and $(CO)_3MnC_5H_4COOH$ (0.72 g, 2.28 mmol) were dissolved in a 1 : 1 THF–CH_2Cl_2 mixture (15 mL). The reaction mixture was stirred for 10 min, NEt_3 (0.8 mL) was added, and the flask was left overnight. The solvent was removed in a water-jet pump vacuum, the residue was extracted with benzene (25 mL), and the solution was filtered and concentrated until crystallization started. The red-orange-colored mother liquor was kept at +5°C to give red crystals. Yield 0.25 g (25%).

IR (KBr; ν, cm^{-1}): 2330 s, 2010 vs, 1930 vs, 1540 s, 1470 s, 1440 s, 1380 s, 1350 s, 1200 vw, 1160 vw, 1030 vw, 820 m, 650 s, 620 vs, 560 w, 530 w, 490 vw.

For $C_{30}H_{22}O_{10}TiMn_2$ (M = 700.24)		
anal. calcd., %:	C, 55.55;	H, 3.63.
Found, %:	C, 56.70;	H, 3.81.

Synthesis of $(C_5H_5)V(OOCCym)_4V(C_5H_5)$ (II). Benzene (20 mL) was added to $(C_5H_5)_2V$ (0.18 g, 1.0 mmol) and $(CO)_3MnC_5H_4COOH$ (0.49 g, 2.0 mmol) and, within 10 min, the solution color turned bright violet. The reaction mixture was refluxed for 2 h and slowly cooled together with an oil bath. Black-violet crystals precipitated overnight. Yield 0.56 g (46%).

IR (KBr; ν, cm^{-1}): 2020 vs, 1944 vs, 1636 s, 1488 s, 1396 s, 1364 m, 1204 vw, 928 w, 844 w, 808 m, 780 m, 668 m, 632 s, 540 m, 364 vw.

Elemental analysis was not performed as the crystals were unstable in air.

Synthesis of $Lu_2Cu(COOR)_2$ ($R = (CO)_3MnC_5H_4$, $Lu = C_7H_9N$) (III). $Cu(CH_3COO)_2 \cdot H_2O$ (0.08 g, 0.4 mmol) and $(CO)_3MnC_5H_4COOH$ (0.20 g, 0.8 mmol) were dissolved in THF (16 mL) and the reaction mixture was stirred at reflux for 2 h; the color changed from light green to dark green. The solvent was removed in a water-jet pump vacuum and the residue was washed with heptane (15 mL) and extracted with boiling toluene (20 mL). 2,6-Lutidine (0.15 mL) was added to the extract, and the solution became blue. On keeping the solution at +5°C, light violet crystals suitable for X-ray diffraction were formed. Yield 0.20 g (67%).

IR (KBr; ν, cm^{-1}): 2925 w, 2016 vs, 1938 vs, 1612 s, 1476 s, 1385 c, 1349 m, 1192 w, 1171 w, 1118 w, 1045 w, 1027 w, 922 w, 864 w, 839 w, 805 m, 795 m, 667 m, 635 s, 572 w, 543 w, 492 w, 475 w.

For $C_{32}H_{26}N_2O_{10}Mn_2Cu$ (M = 771.98)		
anal. calcd., %:	C, 50.38;	H, 3.47.
Found, %:	C, 49.78;	H, 3.39.

Synthesis of $LutCu(OOCR)_3Mn(OOCR)_3CuLut$ ($R = (CO)_3MnC_5H_4$, $Lut = C_7H_9N$) (IV). A solution of $[(CO)_3MnC_5H_4COO]_2Mn$ (0.1 mmol) was synthesized from $(CO)_3MnC_5H_4COOH$ (0.05 g, 0.2 mmol), KOH (0.01 g, 0.2 mmol), and $MnCl_2 \cdot 4H_2O$ (0.02 g, 0.1 mmol) in C_2H_5OH (5 mL). This solution was added to a solution of $(THF)_2Cu_2[(CO)_3MnC_5H_4COO)_4]_2$ (0.1 mmol) in ethanol (7 mL) and the mixture was stirred for 1 h. Then the solvent was evaporated in a water-jet pump vacuum, the residue was extracted with benzene (10 mL), and 2,6-lutidine (0.02 mL) was added to the filtered extract. On keeping the mixture at +5°C, bright blue crystals suitable for X-ray diffraction were formed. Yield 0.032 g (17%).

IR (KBr; ν, cm^{-1}): 2021 vs, 1924 vs, 1604 s, 1481 m, 1387 s, 1362 m, 1115 vw, 782 vw, 666 m, 633 s, 540 w.

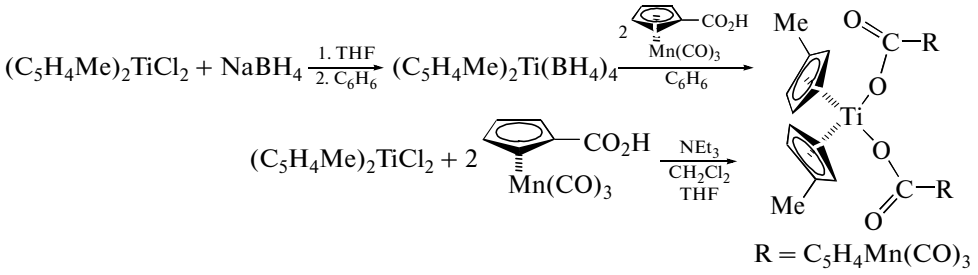
For $C_{68}H_{42}N_2O_{30}Mn_7Cu_2$ (M = 1878.72)		
anal. calcd., %:	C, 44.26;	H, 2.34.
Found, %:	C, 43.47;	H, 2.25.

X-ray diffraction. The crystallographic data and structure refinement details for **I–IV** are summarized in the table. The crystal of complex **II** used for X-ray diffraction turned out to be a twin. Unit cell determination by the CELL_NOW program [23] showed the presence of four twin components rotated through 180°, 84°, and –86° around the x axis relative to one another. Data treatment and refinement of unit cell parameters were done by SAINT program [24] taking account for the presence of four twin components for complex **II**. Absorption corrections were applied by multiple measurement of equivalent reflections using the SADABS program [25] (for complexes **I**, **III**, and

IV) and TWINABS program [26] (for **II**). The structures of **I–IV** were solved by the direct method and refined by the least-squares method relative to F^2 in the anisotropic approximation (except for the C and O atoms of the disordered $NC_5H_3Me_2$ and $C_5H_4Mn(CO)_3$ fragments in **IV** and the disordered solvent molecules in **II**) using the SHELXTL program package [27]. The positions of H atoms were calculated geometrically. Selected bond lengths and bond angles in **I–IV** are given in the captions to Figs. 1–4. Atom coordinates and other structure parameters of **I–IV** are deposited with the Cambridge Crystallographic Data Centre (nos. 941253, 941254, 941256, and 941255 for **I**, **II**, **III**, and **IV**, respectively; http://www.ccdc.cam.ac.uk/data_request/cif).

RESULTS AND DISCUSSION

Bis(methylcyclopentadienyl)titanium dichloride reacts with two moles of $NaBH_4$ to give the Ti(III) complex $(C_5H_4Me)_2Ti(BH_4)$. Treatment of this complex with two equivalents of HOOCCym induces oxidation to Ti(IV) carboxylate, $(C_5H_4Me)_2Ti(OOCCym)_2$ (**I**) in a 10% yield. Meanwhile, direct reaction of titanium dichloride complex with the acid in the presence of triethylamine increases the yield to 25%.



According to X-ray diffraction (Fig. 1, table), the Ti–O bonds (1.973(1) and 1.937(1) Å) are much shorter than the sum of the covalent radii (2.26 Å) [28] due to the additional oxygen–titanium π-bonding in the formally 16-electron environment of titanium. Note that a similar reaction with pivalic acid gave the dicyclopentadienyl complex of trivalent titanium with one chelating carboxylate group [29].

Study of the thermal decomposition of **I** (M = 700.24) demonstrated that below 200°C, only a minor amount of the solvent benzene is lost (1.4% of the weight). The subsequent process has mainly two stages:

(1) 200–300°C: the loss of 15.65% (109.5) due to elimination of $2CO_2$ (88) and H_2O (18) (totally 106);

(2) 300–410°C: the loss of 23.83% (167) due to elimination of 6CO (168);

(3) 400–550°C: the overall loss is 42% (294) and the remainder is 58% (406).

In the first stage (200–300°C), the onset of weight loss is accompanied by endothermic effect, which transforms into exothermic effect; the mass spectrum of the gas phase exhibits not only CO^+ and CO_2^+ ions but also H_2O^+, $C_6H_6^+$, $C_6H_5^+$. The presence of water is indicative of the oxidative destruction of the organic part of the complex; water is evolved almost completely in the first stage of destruction. The appearance of $C_6H_6^+$ and $C_6H_5^+$ ions (after the solvent benzene has been already eliminated) is probably due to the formation of fulvene $C_5H_4CH_2$ and the M–H bond upon

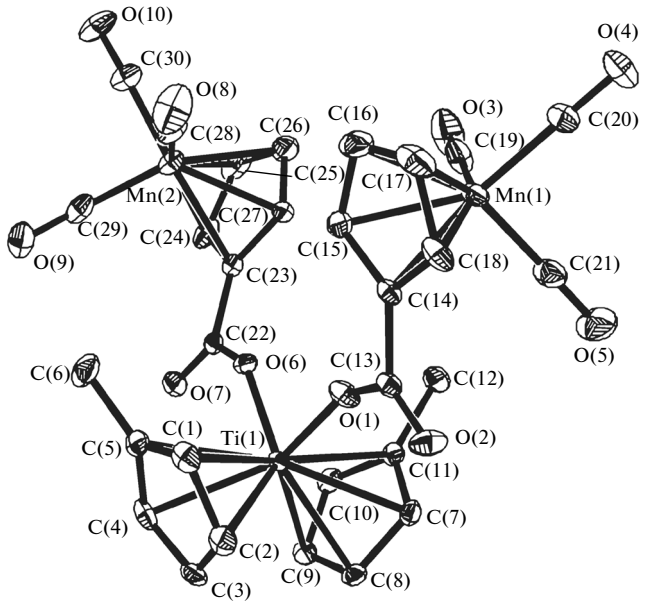


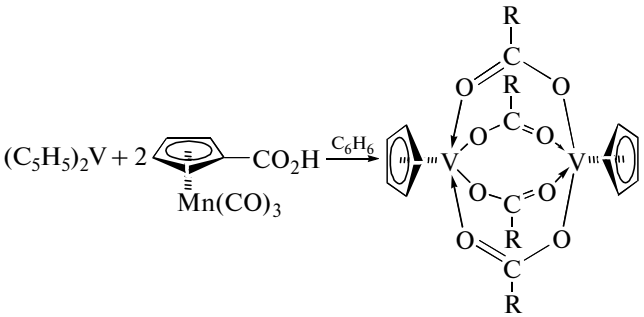
Fig. 1. Molecular structure of complex **I**. Selected bond lengths (Å): Ti–O, 1.973(1) and 1.937(1); Ti···O, 3.58(1) and 3.707(2); C–O(Ti), 1.292(3) and 1.290(1); C–O 1.202(3) and 1.222(1).

Crystal data and structure refinement details for **I–IV**

Parameter	Value			
	III	**IV**	**I**	**II**
M	771.97	1956.80	700.26	1532.74
Diffractometer	Bruker APEX II CCD			
Radiation (λ, Å)	MoK_α (0.71073)			
Temperature, K	296(2)	173(2)	150(2)	150(2)
Space group	$P\bar{1}$	$P\bar{1}$	$C2/c$	$P\bar{1}$
a, Å	8.0945(6)	11.3040(8)	42.287(2)	10.978(2)
b, Å	10.3282(7)	13.703(1)	11.8898(6)	12.756(2)
c, Å	10.6457(7)	15.082(2)	13.4979(6)	13.511(3)
α, deg	93.417(1)	103.716(1)	90	81.712(4)
β, deg	95.637(1)	104.996(1)	98.836(1)	69.701(4)
γ, deg	112.680(1)	113.167(1)	90	69.110(4)
V, Å^3	812.6(1)	1917.5(3)	6706.0(6)	1657.4(5)
Z	1	1	8	1
ρ_{calcd}, g cm^{-3}	1.578	1.695	1.387	1.536
μ, mm^{-1}	1.473	1.742	1.024	1.084
F(000)	391	979	2832	776
Scan range of θ, deg	2.73–29.13	2.13–29.20	0.97–27.92	2.10–25.03
Scan mode	ω			
The number of independent reflections (N_1)	4346 (R_{int} = 0.0163)	10289 (R_{int} = 0.0235)	8024 (R_{int} = 0.0390)	5839 (R_{int} = 0.0883)
The number of reflections with $I > 2\sigma(I)$ (N_2)	3954	7856	6410	5159
The number of refined parameters	216	509	467	399
GOOF (F^2)	1.042	1.010	1.043	1.193
R_1 for N_2	0.0239	0.0375	0.0385	0.0768
wR_2 for N_1	0.0682	0.0962	0.1079	0.2094
$\Delta\rho_{max}/\Delta\rho_{min}$, e Å^{-3}	0.363/–0.381	0.782/–0.535	0.437/–0.379	0.939/–0.612

cleavage of the methylcyclopentadienyl ligand. The intensity of the CO^+-related ion current is much higher in the second stage (300–410°C) than in the first stage. A fundamental distinctive feature of the mass spectrum of the gas phase in the second stage is the presence of the cyclopentadienyl ion $C_5H_5^+$ (m/z = 65). The weight loss continues to a minor extent (2%) in the third stage (400–550°C). Thus, decarboxylation followed by complete decarbonylation takes place with possible formation of H_2O and partial elimination of fulvene from MeCp. The probable composition of the remainder is $(CH_3C_5H_4)(C_5H_4)_2TiMn_2O_2$ (M = 397).

The reaction of vanadocene $(C_5H_5)_2V$ with two equivalents of HOOCCym in benzene gave the dimeric complex **II** with the "Chinese lantern" structure.



(II)

According to X-ray diffraction data (Fig. 2, table), the V–O bonds (2.014(5)–2.032(6) Å) are much shorter than the sum of the covalent radii (2.19 Å) [29], while the V···V distance (3.650(2) Å) is substan-

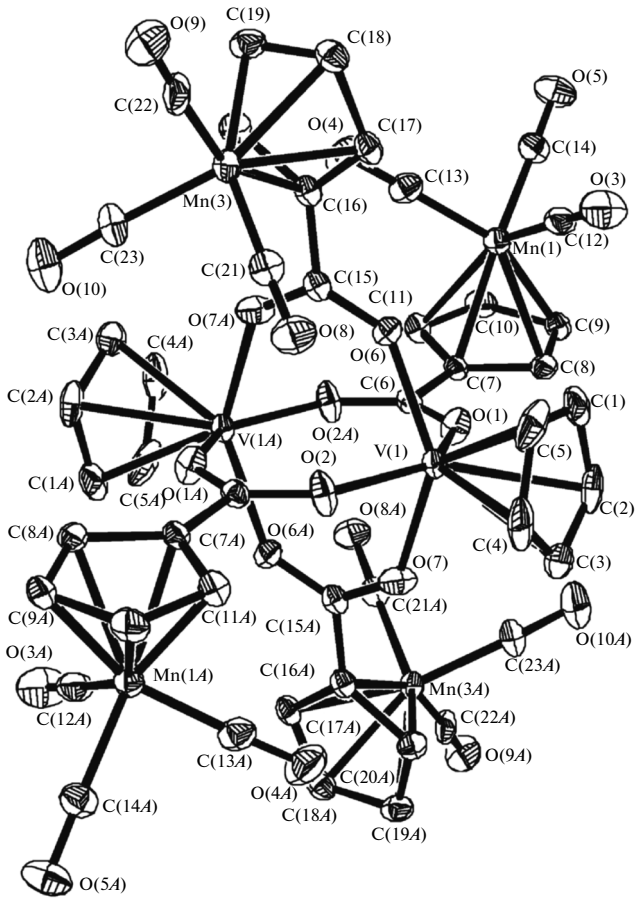
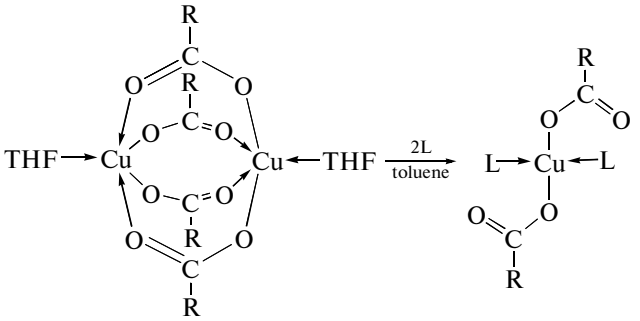


Fig. 2. Molecular structure of complex **II**. Selected bond lengths (Å): V–O, 2.014(5)–2.032(6); V···V, 3.650(2).

tially longer than the sum of the covalent radii of 3.06 Å [29]. The situation is similar for known vanadium complexes $CpV(OOCR)_4VCp$, R = Ph [3], CF_3 [4], furyl [6]. However, the enhanced sensitivity of complex **II** to air oxygen compared with other vanadium carboxylate complexes was unexpected.

Treatment of $(THF)_2Cu_2[(CO)_3MnC_5H_4COO]_4$ with an excess of 2,6-lutidine in toluene results in dimerization of the diene to give a planar mononuclear complex **III** with NCuN and OCuO angles of 180° (Cu–O, 1.9639(9); Cu–N, 2.0292(11) Å) (Fig. 3, table), which resembles the above-noted chromium complex $(Lut)_2Cr(OOCCF_3)_2$ [13].



(III)

Finally, the heterometallic Cu(II)–Mn(II) complex **IV** having bridging cymantrenecarboxylate anions and axial 2,6-lutidine molecules was prepared by the reaction of copper(II) and manganese(II) cymantrenecarboxylates and lutidine in an ethanol–THF mixture.

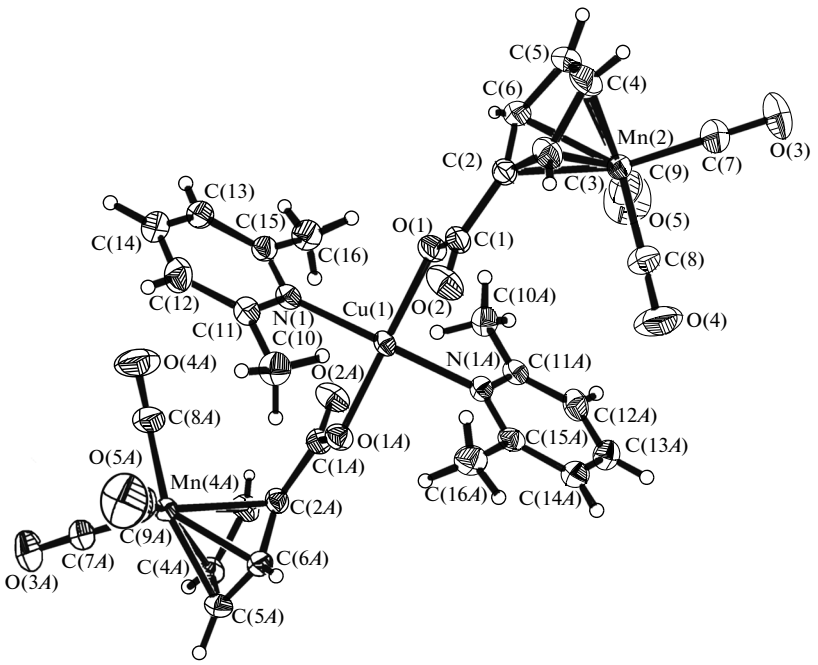
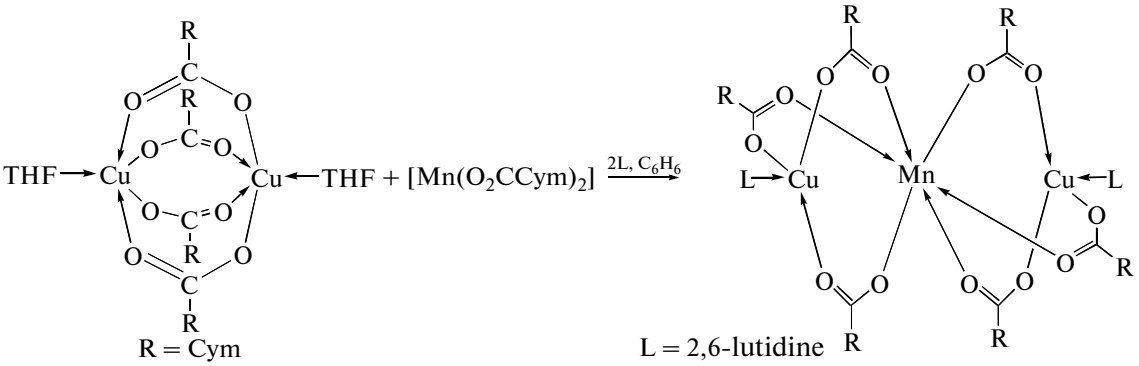


Fig. 3. Molecular structure of complex **III**. Selected bond lengths (Å): Cu–O, 1.9639(9), Cu–N, 2.0292(11).



According to X-ray diffraction data (Fig. 4, table), the copper atom in **IV** has a tetrahedral environment, while manganese(II) has an octahedral environment. The Cu–O and Cu–N bond lengths are roughly equal to the sum of the covalent radii of the atoms, and the Mn–O bond is somewhat longer than this sum (probably due to the Mn(II) d^5 configuration in the octahedron).

Note in conclusion that the mode of binding of cymantrenecarboxylate groups to metal atoms depends appreciably on the steric factors of other ligands bound to the metals, in particular, bis-methylcyclopentadienyl at titanium, cyclopentadienyl at vanadium, and lutidine ligands at copper atoms.

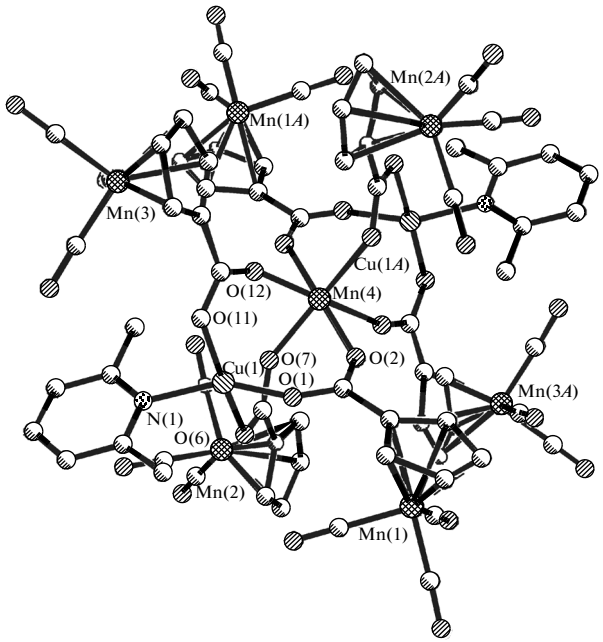


Fig. 4. Molecular structure of complex **IV**. Selected bond lengths (Å): Cu(1)–O(1), 1.9339(17); Cu(1)–O(11), 1.9439(17); Cu(1)–O(6), 1.9526(17); Cu(1)–N(1), 1.962(5); Cu(1)–N(1)', 2.037(5)' Mn–O(2), 2.1153(17)–2.2057(18); Cu···Mn, 3.5033(4).

ACKNOWLEDGMENTS

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