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Dicarbonylcyclopentadienyltellurophenyliron Complexes as Ligands

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Abstract—The reaction of $CpFe(CO)_2TePh$ (**I**) with ferricinium hexafluorophosphate as an oxidant affords ionic complex $\{[CpFe(CO)_2]_2(\mu\text{-}TePh)\}^+PF_6^-$ (**II**) with the simultaneous formation of diphenylditellurium. The decarbonylation of compound **II** by Me_3NO followed by the addition of complex **I** affords trinuclear complex $\{[CpFe(CO)_2(\mu\text{-}TePh)]_2Fe(CO)Cp\}PF_6$ (**III**). The corresponding tetrafluoroborate (**IV**) is synthesized similarly. The heating of compound **I** with PPh_3 gives $CpFe(CO)(PPh_3)TePh$ (**V**) that reacts with ionic complex $[CpMn(CO)_2(NO)]PF_6$ (**VI**) to form binuclear heterometallic ionic complex $[CpFe(CO)(PPh_3)(\mu\text{-}TePh)Mn(CO)(NO)Cp]PF_6$ (**VII**). A similar reaction of $Cp'Fe(CO)_2TePh$ (Cp' is methylcyclopentadienyl) with compound **VI** affords heterometallic $[Cp'Fe(CO)_2(\mu\text{-}TePh)Mn(CO)(NO)Cp]PF_6$ (**VIII**). The structures of compounds **II**, **IV**, **VII**, and **VIII** are determined by X-ray diffraction analysis (CIF files CCDC 963285, 963286, 963288, and 963289, respectively).

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INTRODUCTION

Complexes containing heavy chalcogen atoms, in particular, tellurium, are fairly poorly studied compared to wide investigations of sulfur-containing complexes of transition metals. At the same time, these complexes can serve as ligands in the construction of heterometallic telluride clusters. Due to the detachment of organic groups in the course of pyrolysis, they are convenient precursors for the preparation of valuable inorganic homo- and heterometallic tellurides of specified complicated composition. In this approach, it is easy to vary the ratio of atoms of different metals and tellurium using metal-containing fragments with different numbers of telluorophenyl ligands. It was reported, on the one hand, that the reaction of *fac*-$[Fe(CO)_3(TePh)_3]^-$ with $[Mn(CO)_3(CH_3CN)_3]^+$ afforded complex $(CO)_3Mn(\mu\text{-}TePh)_3Fe(CO)_3$ [1]. On the other hand, the reaction of *cis*-$[PPN][Mn(CO)_4(TePh)_2]$ with $Co(ClO_4)_2$ and Ph_2Te_2 in tetrahydrofuran gave $(CO)_3Mn(\mu\text{-}TePh)_3Co(CO)(\mu\text{-}TePh)_2Mn(CO)_4$ [2]. Complex $[Cp^*Ir(CO)(TeTol)_2]$, which easily displaced cyclooctadienes from complexes $((COD)MCl_2$ (M = Pd or Pt) to form $[Cp^*Ir(CO)(\mu\text{-}TeTol)_2MCl_2]$, was also used as a chelating bis(telluoroaryl) ligand. According to the X-ray diffraction data, in both compounds the Ir and Te atoms are remote at a nonbinding distance (3.9038(6) and 3.8527(4) Å, respectively) [3]. Finally, an example for the monotellurophenyl bridging cluster is the adduct $Me_4C_4(CO)_2Co(\mu\text{-}TePh)W(CO)_5$ in which the unusual shortening of the Co–Te distance is observed along with an additional coordination of the W atom to the Te atom. The pyrolysis of this adduct gives inorganic mixed-metal telluride CoTeW [4].

It seemed interesting to use the recently synthesized [5] complex $CpFe(CO)_2TePh$ as a ligand for the iron or manganese complexes with the purpose of the directed synthesis of homo- or heteropolynuclear tellurium-containing metal clusters.

EXPERIMENTAL

All procedures related to the synthesis and isolation of compounds were carried out in an argon atmosphere in dehydrated solvents. Commercial Ph_2Te_2, $Mn_2(CO)_{10}$, and $[CpFe(CO)_2]_2$ were used without additional purification. Reagents $Fc^+PF_6^-$ [6] and $[CpMn(CO)_2(NO)]PF_6$ [7] were synthesized according to published procedures.

Synthesis of $CpFe(CO)_2TePh$ (I). Toluene (20 mL) was added to $[CpFe(CO)_2]_2$ (1.01 g, 2.84 mmol) and Ph_2Te_2 (1.15 g, 2.81 mmol) in an argon flow with magnetic stirring. The red-brown reaction mixture was heated to 100°C for 1.5 h in an oil bath with a reflux condenser until a green color appeared. The reaction course was also monitored using IR spectroscopy and thin layer chromatography (TLC) in toluene (the IR spectrum and TLC were detected at an interval of 15 min). The green solution was concentrated in a vacuum of a water jet pump to 1/3 of the initial volume, heptane (5 mL) was added, and the product was recrystallized from a toluene–heptane mixture and kept for 24 h at –10°C. Precipitated green-brown crystals were decanted, washed with heptane (2 × 5 mL), and dried in vacuo. The yield was 1.43 g (66%).

For $C_{13}H_{10}O_2FeTe$ (M = 381.67)
anal. calcd., %: C, 40.91; H, 2.64.
Found, %: C, 41.57; H, 2.52.

IR (CH_2Cl_2, ν, cm^{-1}): 2005, 1968.

TLC (Silufol, toluene): green spot, R_f = 0.6.

Synthesis of $\{[CpFe(CO)_2]_2(\mu\text{-}TePh)\}^+PF_6^- \cdot 0.5C_6H_6$ (II). A mixture of compound **I** (0.34 g, 0.88 mmol) and $(C_5H_5)_2FePF_6$ (0.15 g, 0.44 mmol) in CH_2Cl_2 (15 mL) was stirred for 7 h. The red-brown solution was evaporated to dryness and washed with heptane (20 mL). The residue was dissolved in CH_2Cl_2 (15 mL), benzene (10 mL) was added to the filtered solution, and the resulting solution was evaporated to half a volume and kept at –5°C for 36 h. The precipitated red-brown crystals were separated and washed with heptane. The yield was 0.26 g (82%).

For $C_{23}H_{18}O_4Fe_2PF_6Te$ (M = 742.65)
anal. calcd., %: C, 37.19; H, 2.44.
Found, %: C, 36.80; H, 2.45.

IR (KBr, ν, cm^{-1}): 3129 w, 2040 v.s, 2023 v.s, 1991 v.s, 1716 v.w, 1575 v.w, 1475 w, 1439 w, 1430 m, 1422 m, 1115 w, 1073 w, 1016 w, 999 w, 887 m, 843 v.s, 825 v.s, 742 m, 694 w, 609 s, 556 m, 491 w, 458 m.

Synthesis of $[[Cp(CO)_2Fe(\mu\text{-}TePh)]_2Fe(CO)Cp]PF_6 \cdot 0.5C_6H_6$ (III). Compound **II** (0.1 g, 0.14 mmol) was dissolved in CH_2Cl_2 (15 mL) (the solution was yellow-brown), anhydrous $(CH_3)_3NO$ (0.018 g, 0.16 mmol) (obtained from $(CH_3)_3NO \cdot 2H_2O$ by azeotropic drying with benzene) in CH_2Cl_2 (3 mL) was added, and the mixture was stirred for 10 min. Then compound **I** (0.05 g, 0.14 mmol) was added, and the mixture was additionally stirred for 15 min. The solvent was removed in vacuo, the product was extracted with CH_2Cl_2 (15 mL), the solution was filtered, C_6H_6 (20 mL) was added, and the resulting solution was concentrated to the beginning of crystal precipitation and left to stay at room temperature. Black-brown crystals suitable for X-ray diffraction analysis were formed. The yield was 0.037 g (24.5%).

For $C_{32}H_{27}O_5F_6PFe_3Te_2$ (M = 1059.26)
anal. calcd., %: C, 38.27; H, 2.75.
Found, %: C, 38.31; H, 2.58.

IR (KBr, ν, cm^{-1}): 2026v.s, 2012 v.s, 1980 v.s, 1969 v.s, 1931 v.s, 1569 w, 1472 w, 1428 w, 1359 v.w, 1300 v.w, 1064 v.w, 1013 v.w, 997 v.w, 842 v.s, 830 v.s, 735 m, 694 w, 615 m, 573 s, 556 s, 456 w.

Synthesis of $[\{(C_5H_5)Fe(CO)_2TePh\}_2Fe(CO)(C_5H_5)]BF_4$ (IV). Compound $[(C_5H_5Fe(CO)_2)_2TePh]BF_4$ (0.1 g, 0,16 mmol) was dissolved in CH_2Cl_2 (16 mL), a solution of Me_3NO (0.012 g, 0.16 mmol) (obtained from $Me_3NO \cdot 2H_2O$ by azeotropic drying in benzene) in CH_2Cl_2 (5 mL) was added with stirring, and the resulting mixture was stirred for 15 min. Then $C_5H_5Fe(CO)_2TePh$ (0.0592 g, 0.16 mmol) was added, and the mixture was additionally stirred for 20 min and evaporated. The brown residue was obtained and extracted with CH_2Cl_2 (15 mL). The solution was filtered to another flask, benzene (20 mL) was added, and the resulting solution was concentrated in a vacuum of a water jet pump until crystals began to precipitate. After keeping at room temperature, brown crystals were obtained. The yield was 0.055 g (35.5%).

For $C_{32}H_{25}BO_5F_4Fe_3Te_2$ (M = 999.09)
anal. calcd., %: C, 38.47; H 2.52.
Found, %: C, 38.31; H 2.58.

IR (KBr, ν, cm^{-1}): 3445 br.w, 3116 w, 2025 v.s, 2012 v.s, 1978 v.s, 1968 v.s, 1931 v.s, 1569 w, 1423 w, 1426 w, 1105 w, 1077 m, 1046 s, 1013 w, 884 w, 860 m, 825 w, 736 m, 694 w, 616 w, 574 m, 557 m, 543 m, 498 w, 455 w.

Synthesis of $CpFe(CO)(PPh_3)TePh$ (V). A solution of compound **I** (0.16 g, 0.4 mmol) and PPh_3 (0.21 g, 0.8 mmol) was refluxed in benzene (15 mL) for 2 h, evaporated to dryness, and washed with hexane (15 mL). The residue was dissolved in CH_2Cl_2 (15 mL). The obtained solution was filtered, hexane (4 mL) was added to the mother liquor, and the solution was evaporated to half a volume and left to crystallize at –18°C. Brown crystals precipitated. The yield was 0.24 g (49%) .

For $C_{30}H_{25}OPFeTe$ (M = 615.94)
anal. calcd., %: C, 58.50; H, 4.09.
Found, %: C, 58.35; H, 4.04.

IR (KBr, ν, cm^{-1}): 3050 w, 2954 w, 2922 w, 2852 w, 2009 m, 1922 v.s, 1585 w, 1569 m, 1478 m, 1468 m, 1432 m, 1307 v.w, 1182 w, 1157 w, 1115 w, 1090 m, 1070 w, 1027 w, 1016 w, 997 w, 839 w, 823 w, 744 m, 731 m, 693 s, 649 w, 619 w, 599 w, 575 w, 548 s, 526 v.s, 508 m, 496 w, 453 w.

Synthesis of $[CpFe(CO)(PPh_3)(\mu\text{-}TePh)Mn(CO)(NO)Cp]PF_6$ (VII). Compound **V** (0.26 g, 0.43 mmol) was added to a solution of compound **VI** (0.15 g, 0.43 mmol) in CH_3CN (10 mL). The solution turned brown as compound **V** was dissolved, and the reaction mixture was stirred for 1 h. The obtained solution was evaporated to the formation of the brown oil, which was dissolved in CH_2Cl_2 (10 mL). Then the solution was filtered, benzene

(10 mL) was added, and the solution was concentrated in vacuo to ~10 mL. The mother liquor was separated from the precipitated brown oil, and the latter was dried and triturated with Et_2O (10 mL). Brown crystals were obtained from a brown powder (0.37 g) that was recrystallized from a CH_2Cl_2—Et_2O mixture. The yield was 0.10 g (25%).

For $C_{36}H_{30}NO_3F_6P_2FeMnTe$ ($M = 938.96$)

anal. calcd., %:	C, 46.05;	H, 3.22;	N, 1.49.
Found, %:	C, 46.65;	H, 3.07;	N, 1.44.

IR (KBr, ν, cm^{-}): 3441 br.m, 3122 w, 3058 w, 2970 w, 2924 w, 2853 w, 2016 s, 1948 s, 1768 s, 1628 w, 1571 w, 1508 w, 1480 w, 1434 m, 1364 w, 1309 w, 1268 w, 1183 w, 1160 w, 1114 w, 1092 w, 1069 w, 1015 w, 998 w, 840 v.s, 741 w, 696 m, 657 w, 620 w, 590 w, 572 w, 556 s, 549 w, 525 s, 509 w, 492 w, 460 w, 432 w.

Synthesis of [Cp'Fe(CO)$_2$(μ-TePh)Mn(CO)(NO)Cp]PF$_6$ (VIII). A solution of $[Cp'Fe(CO)_2]_2$ (0.20 g, 0.53 mmol) and diphenylditellurium (0.21 g, 0.53 mmol) in benzene (10 mL) was refluxed for 6 h, the solution was evaporated to dryness, and the residue was dissolved in acetonitrile (10 mL). Compound **VII** (0.37 g) was added to the obtained green solution of $Cp'Fe(CO)_2TePh$, and the mixture was stirred for 40 min. The brown solution was evaporated to dryness, and the residue was washed with diethyl ether (20 mL) and dissolved in CH_2Cl_2 (10 mL). The solution was filtered off, benzene (5 mL) was added, and the mixture was evaporated to 1/4 of the volume and kept at 5°C. The yield of brown crystals was 0.38 g (32%)

For $C_{20}H_{17}NO_4F_6PMnFeTe$ ($M = 718.71$)

anal. calcd., %:	C, 33.42;	H, 2.38;	N, 1.95.
Found, %:	C, 35.14;	H, 2.59;	N, 2.55.

IR (KBr, ν, cm^{-1}): 3114 w, 2036 v.s, 2019 v.s, 1980 v.s, 1774 s, 1751 s, 1713 w, 1633 w, 1573 w, 1475 m, 1882 m, 1395 w, 1376 w, 1303 w, 1266 w, 1237 w, 1181 w, 1070 w, 1036 w, 1016 w, 998 w, 843 s, 831 s, 738 m, 695 w, 680 w, 620 w, 608 m, 573 m, 557 s, 487 w, 475 w, 458 w.

A CHNS analyzer (Carlo Erba) was used for chemical analyses. A BrukerAlpha FT-IR spectrometer was used for IR spectroscopy in KBr pellets and in solutions of dehydrated organic solvents.

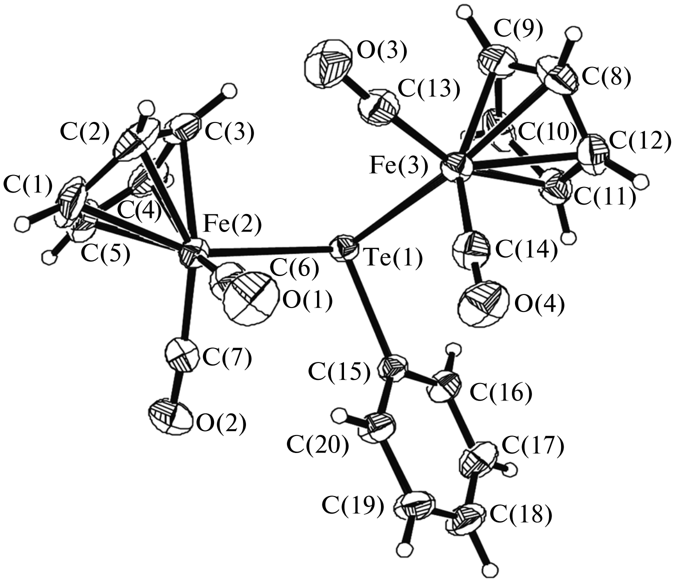


Fig. 1. Molecule structure of complex **II**. Selected bond lengths: Fe(2)–Te(1) 2.5493 (4) and Fe(3)–Te(1) 2.5696(4) Å.

The X-ray diffraction analyses of single crystals of compounds **II**, **IV**, **VII**, and **VIII** were carried out on a Bruker Smart Apex CCD AXS diffractometer at room temperature or at 173 K for complex **IV**. The structures were determined by direct methods and refined by least squares for F^2 in the anisotropic approximation for non-hydrogen atoms (SHELXTL) [8]. The structural contribution of the disordered PF_6^- anion in a crystal of complex **VII** and the disordered solvate CH_2Cl_2 molecule in crystal **VIII** was removed using the SQUEEZE procedure accomplished in the PLATON program [9]. The cation of complex **VIII** turned out to be disordered, so that the fragments $Cp'Fe(CO)_2$ and CpMnCONO occupy the general position of each other. The metal atoms were specified as a superposition of the iron and manganese atoms with common coordinates and thermal parameters. The ratio refined by the least-squares method was 0.707(3)Fe : 0.293(3)Mn in the Fe(1) position. An attempt to refine the disordering of the NO group over all four positions CO/NO with the common coordinates and thermal parameters of the C and N atoms and the superposition of the conditions of a linear dependence of the SUMP type based on one NO group at the manganese atom gave the population of the C(14)O(4) position close to zero. After this position was excluded from the refinement and the population of N(13) was equalized to that of Mn(1), the populations of N(5) and N(21) were refined as 0.24(3) and 0.47(3) of the NO groups, respectively, with the superposition of a linear restraint; population N(5) + N(21) = Mn(2). The most part of structure **IV** turned out to be disordered over two positions with the population about 0.5; however, attempts of the solution in the nonsymmetrical space group or in the cell of a doubled volume with two independent molecules did not give an ordered structure. The positions of the carbon atoms of the disordered cyclopentadienyl and phenyl groups were refined in the isotropic approximation using restrains of the equal distances of the SAME type.

The main structural data are presented in the table. The coordinates of atoms and other parameters of the structures were deposited with the Cambridge Crystal-

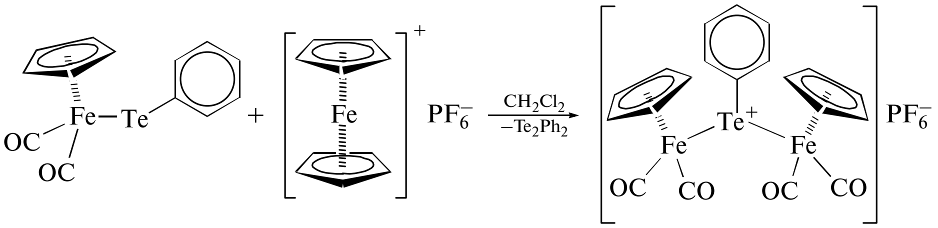
Crystallographic data and refinement parameters for structures **II**, **IV**, **VII**, and **VIII**

Parameter	Value			
	II	**IV**	**VII**	**VIII**
FW	999.08	615.92	793.97	718.71
Diffractometer	Bruker APEX II CCD			
Radiation (λ, Å)	MoK_α (0.71073)			
Temperature, K	296(2)	173(2)	296(2)	296(2)
Space group	$P4_3$	$P\bar{1}$	$P2_1/n$	$P\bar{1}$
a, Å	11.339(1)	9.190(2)	10.8763(14)	7.6365(5)
b, Å	11.339(1)	10.345(2)	25.245(3)	13.2237(8)
c, Å	26.731(2)	14.970(3)	16.534(2)	13.4347(8)
α, deg	90	94.852(3)	90	76.193(1)
β, deg	90	101.278(2)	104.574(3)	76.601(1)
γ, deg	90	111.542(2)	90	87.883(1)
V, Å^3	3436.6(5)	1279.0(4)	4394.3(10)	1281.37(14)
Z	4	2	4	2
ρ_{calcd}, g/cm^3	1.931	1.599	1.200	1.863
μ, mm^{-1}	2.974	1.791	1.332	2.307
F(000)	1920	612	1580	696
Scan range; θ, deg	2.36–30.87	1.41–29.00	1.51–26.38	2.51–24.99
Scan type	ω			
Independent reflections (N_1)	10676 (R_{int} = 0.0313)	6725 (R_{int} = 0.0210)	8743 (R_{int} = 0.0303)	4352 (R_{int}0.0692)
Reflections with $I > 2\sigma(I)$ (N_2)	9192	5622	5491	3981
Number of refined parameters	464	288	373	323
Goodness-of-fit (F^2)	1.042	1.038	0.892	1.031
R_1 for N_2	0.0328	0.0354	0.0469	0.0265
wR_2 for N_1	0.0661	0.0888	0.1190	0.0738
$\Delta\rho_{max}/\Delta\rho_{min}$, e Å^{-3}	0.741/–0.372	1.035/–0.409	0.616/–0.670	0.638–0.881

lographic Data Centre (CCDC 963285 (**II**), 963286 (**IV**), 963288 (**VII**), and 963289 (**VIII**); http://www.ccdc.cam.ac.uk/data_request/cif).

RESULTS AND DISCUSSION

The oxidation of green mononuclear complex **I** by ferricinium hexafluorophosphate (0.5 molar equivalent) in CH_2Cl_2 affords brown complex **II** (Fig. 1) accompanied by the formation of diphenylditellurium. The IR spectrum of the synthesized compound contains four bands of CO stretching vibrations (ν, cm^{-1}: 2044 v.s, 2022 v.s, 1983 v.s, 1972 s). According to the X-ray diffraction data, two Fe–Te bonds are nearly equalized. This is explainable if assuming that the positive charge of the cation is localized on the tellurium atom.

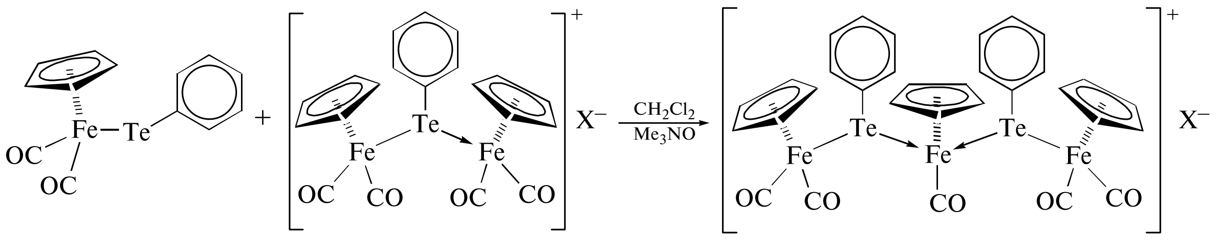


(II)

Evidently, the oxidation of compound **I** affords radical $TePh^{\bullet}$, which dimerizes to the observed diphenylditellurium, and $CpFe(CO)_2^+$, coordinating to the second molecule of compound **I** through the tellurium atom.

Similar processes were earlier observed for the oxidation of $CpFe(CO)_2SR$ [10] and radical $Cp(CO)_2MnTePh^{\bullet}$, which transformed into the binuclear cation with the tellurophenyl bridge $[Cp(CO)_2Mn]_2TePh^+$. The number of electrons in the latter, unlike complex **II**, was by two electrons lower and, hence, this cation contained the Mn–Mn bond (3.010(7) Å) and partially multiple Mn–Te bonds shortened to 2.511 Å [11] compared to the sum of covalent radii of Mn and Te (2.77 Å) [12]. Note that the Fe–Te bonds in compound **II** are also shortened to 2.5493 (4) and 2.5696(4) Å, probably, due to an additional dative interaction of a lone electron pair at the iron atoms with the vacant *d* orbitals of tellurium.

Then complex **II** was introduced into the reaction of trimethylamine oxide in dichloromethane, and CO_2 formation was observed. The decarbonylation product was immediately treated with one equivalent of the starting complex **I** to form brown trinuclear ionic complex **III**. Crystallization from a dichloromethane–benzene system gave single crystals of the solvate with half a benzene molecule, whose composition was characterized by the elemental analysis and IR spectrum: five bands of CO stretching vibrations (ν, cm^{-1}: 2026 v.s, 2012 v.s, 1980 v.s, 1969 v.s, 1931 v.s). Single crystals of tetrafluoroborate **IV** were obtained similarly and characterized by X-ray diffraction analysis. They also contain half a molecule of solvate benzene.

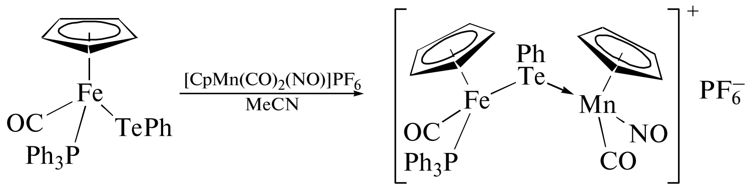


X = PF_6 (**IIa**), BF_4 (**IIb**)

X = PF_6 (**III**), BF_4 (**IV**)

Trinuclear cationic complex **IV** contains two bridging TePh groups between three iron atoms. The Fe–Te bond lengths in complex **IV** (Fig. 2) are close to those found in complex **II** and are also shortened compared to the sum of covalent radii. The IR spectrum of compound **IV** contains five bands of CO stretching vibrations (ν, cm^{-1}: 2025 v.s, 2012 v.s, 1978 v.s, 1968 v.s, 1931 v.s), which nearly coincide with those found for compound **III**.

Complex **V** synthesized by the heating of compound **I** with PPh_3 readily reacts at room temperature in acetonitrile with cationic complex **VI** to substitute one of the CO groups at the manganese atom and to give binuclear heterometallic cationic complex **VII**, whose crystals suitable for X-ray diffraction analysis were obtained by crystallization from a dichloromethane–diethyl ether mixture.



(**VII**)

According to the X-ray diffraction data (Fig. 3), complex **VII** contains one bridging TePh group between the iron and manganese atoms. The Fe–Te and Mn–Te bonds in the complex are 2.5667(7) and 2.6154(9) Å, respectively. Both M–Te bonds are shortened compared to the sum of covalent radii ($r_{Fe} + r_{Te} =$ 2.70 Å, $r_{Mn} + r_{Te} =$ 2.77 Å) [12], indicating their increased multiplicity, probably, due to the aforementioned additional dative interaction. The IR spectrum of the obtained compound contains two bands of stretching vibrations (ν, cm^{-1}: 2016 s, 1948 s) of CO at the Mn and Fe atoms, respectively, and one stretching vibration band (1770 cm^{-1}, v.s) of the NO group bound to Mn.

Complex **VIII** was synthesized similarly by the reaction of $Cp'Fe(CO)_2TePh]$ (Cp' is methylcyclopentadienyl) with compound **VI** in acetonitrile at room

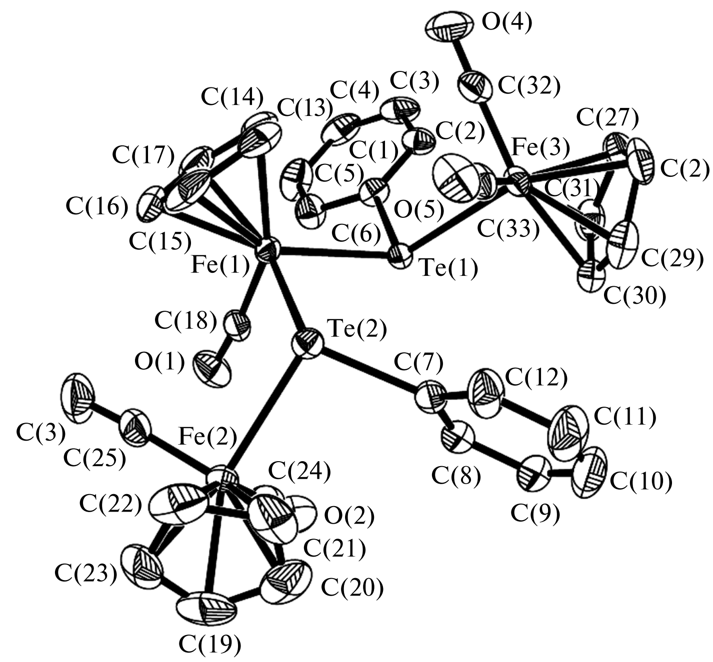
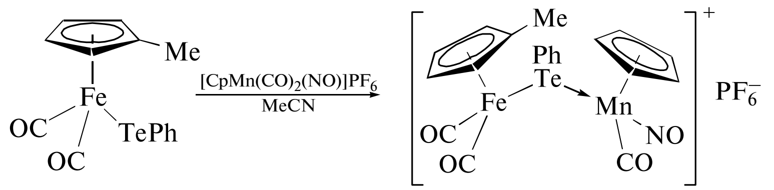


Fig. 2. Molecule structure of complex **IV**. Selected bond lengths: Fe–Te 2.5415(6), 2.5647(6), 2.5471(6), and 2.5559(7) Å.

temperature. Its spectrum contains three intense bands of stretching vibrations of the CO groups bound to Fe (2036, 1980 cm^{-1}) and Mn (2019 cm^{-1}) and a strong stretching vibration band of NO at the Mn atom (1774 cm^{-1}). The structure of complex **VIII** was confirmed by the X-ray diffraction data. However, since the inversion center of the molecule is localized on the tellurium atom, the Cp'Fe(CO)$_2$ fragment is combined with CpMn(CO)(NO), due to which the Fe–Te and Mn–Te bonds are averaged.



(VIII)

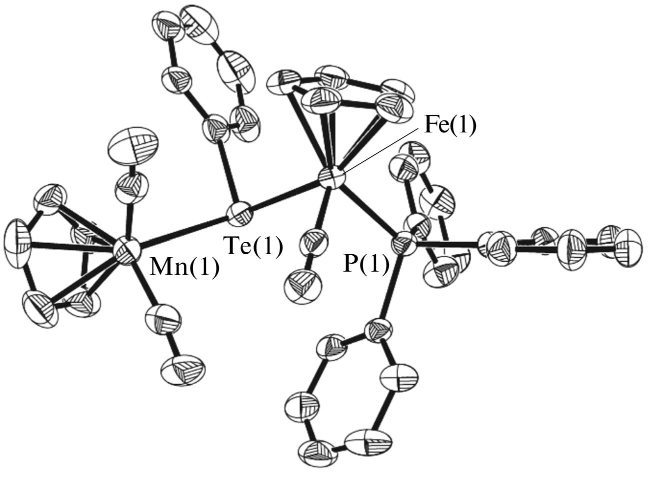


Fig. 3. Structure of the cation in complex **VII**. Selected bond lengths: Te–Fe 2.5667(7) and Te–Mn 2.6154(9) Å.

Thus, complexes Cp'Fe(CO)$_2$TePh,, CpFe(CO)$_2$TePh and CpFe(CO)(PPh$_3$)TePh can act as monodentate ligands for iron and manganese carbonyl-containing complexes, which makes it possible to control the composition of the formed homo- and heteronuclear clusters promising as precursors of inorganic materials of specified composition.

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