Synthesis and molecular structures of heterometallic complexes $[RC_5H_4Fe(CO)_2]_2Sn(TePh)_2$ and their adducts with chromium and tungsten carbonyls or with trimethylplatinum iodide

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Complexes $[RC_5H_4Fe(CO)_2]_2Sn(TePh)_2$ (R = H, Me) containing stable heterometallic Fe—Sn—Fe fragments with two phenyltellurium groups at the tin atom were synthesized from $[RC_5H_4Fe(CO)_2]_2SnCl_2$ (R = H, Me) and sodium phenyltelluride and their structures were established by X-ray analysis. Their chelates with tungsten tetracarbonyl, $[RC_5H_4Fe(CO)_2]_2Sn(TePh)_2[W(CO)_4]$ (R = Me, H), and complexes with two $Cr(CO)_5$ fragments or dimeric trimethylplatinum iodide were synthesized and studied by X-ray analysis. Thermal decomposition of $[RC_5H_4Fe(CO)_2]_2Sn(TePh)_2$ complexes and their adducts with ML fragments (ML = $W(CO)_4$, 2 $Cr(CO)_5$, $(Me_3PtI)_2$) into inorganic tellurides of a preset mixed-metal—chalcogenide composition was studied by differential scanning calorimetry. The temperature of complete elimination of organic fragments from methylcyclopentadienyl complexes is about 100 °C lower than in the case of cyclopentadienyl analogs.

Key words: heterometallic complexes, chelates, metal—metal bonds, organotellurium ligands, thermal decomposition, X-ray analysis.

Starting in the early 1960's,[1] heterometallic compounds with stable Fe—Sn bonds have been much investigated by many authors including A. N. Nesmeyanov and co-workers.[2] These compounds are characterized by shortened Fe—Sn bonds and the ability for exchange of substituents at the tin atom by other nucleophilic reagents to occur,[3] which in the case of telluride ions results in the formation of hexanuclear $[C_5H_5Fe(CO)_2]_4(Sn_2Te_2)$ complexes[4] with bridging tellurium atoms.

In this work, we report the synthesis of the first organotellurium derivatives of the complexes containing the Fe_2Sn fragment, their use as ligands toward tungsten, chromiun, and platinum atoms, as well as the results of X-ray and thermal decomposition studies of heterometallic complexes obtained.

Results and Discussion

Initial tin dihalides containing two cyclopentadienyl- or methylcyclopentadienylirondicarbonyl ligands, $[RC_5H_4Fe(CO)_2]_2SnCl_2$ (R = H (**1**), Me), were obtained by insertion of $SnCl_2$ into the Fe—Fe bond in corresponding $[RC_5H_4Fe(CO)_2]_2$ complexes (Scheme 1). The X-ray study of complex **1** was performed previously[5]; in this work we refined its structure to R = 0.0437 (Table 1). The direct Fe—Sn bonds in molecule **1** are shortened to 2.504(1) Å due to partial double bonding of the atoms, whereas the Sn—Cl bonds (2.416(2) Å) are ordinary. Treatment of complex **1** with sodium phenyltelluride obtained from diphenylditellurium and sodium borohydride in ethanol results in the formation of brown crystals of $[CpFe(CO)_2]_2Sn(TePh)_2$ (**2**). According to the data of X-ray study (Fig. 1, Table 2), the direct Fe—Sn bonds in molecule **2** are lengthened to 2.580(2) Å due to the presence of phenyltellurium substituents at the tin atoms (these substituents are weaker electron acceptors than chlorine atoms). The decrease in the Fe—Sn—Fe angle from 129° to 123° is also in agreement with weakened Fe—Sn double bonding, whereas the lengths of the Te—Sn bonds are characteristic of ordinary bonds (2.794(1) Å).

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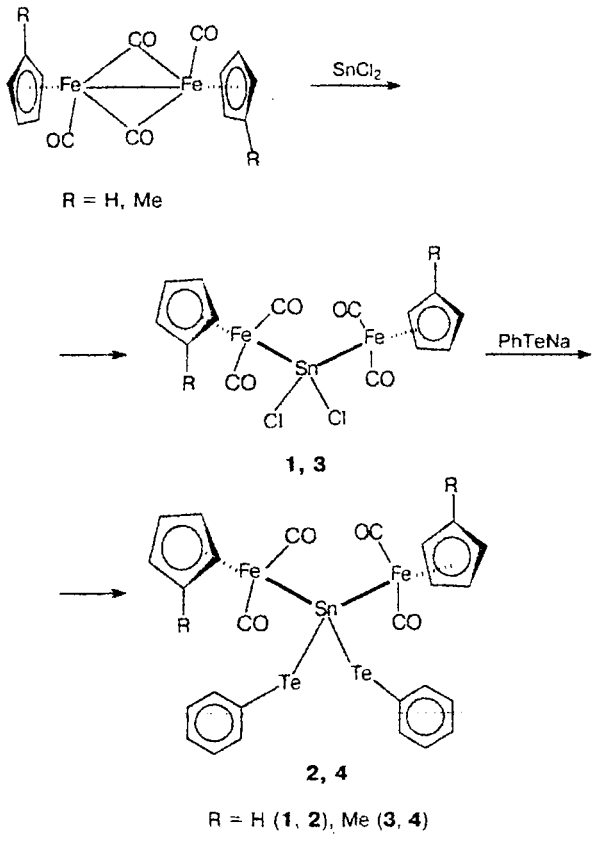
Table 1. Selected crystallographic parameters and conditions of X-ray experiment for complexes **1**—**5**

Parameter	**1**	**2**	**3**	**4**	**5**
Space group	*C2/c*	*P*-1	*Pna*2(1)	*P*-1	*Pbcn*
Temperature/K	293(2)	293(2)	293(2)	293(2)	293(2)
Radiation (λ/Å)			Mo-Kα (λ = 0.71073)		
a/Å	15.056(5)	9.222(2)	21.129(7)	10.163(4)	11.969(4)
b/Å	7.641(2)	11.581(2)	7.238(2)	11.224(6)	18.695(4)
c/Å	15.331(5)	13.618(3)	13.217(4)	14.400(6)	16.196(5)
α/deg	90	100.72(2)	90	89.28(4)	90°
β/deg	94.77(3)	90.17(2)	90	89.72(4)	90°
γ/deg	90	105.38(2)	90	64.24(3)	90°
V/Å^3	1757.6(9)	1375.7(5)	2021.4(11)	1479.2(11)	3624(2)
Z	4	2	4	2	4
d_{calc}/g cm^{-3}	2.054	2.129	1.878	2.043	2.210
θ/2θ scanning range/deg	2.67—60.14	1.52—60.08	1.93—50.10	1.41—49.96	2.18—51.1
Number of measured reflections	2536	6464	1873	5513	2838
Number of reflections with $I > 2\sigma$	1870	4369	1226	4682	1369
R	0.0437	0.0647	0.0614	0.0376	0.0403
R_w	0.0977	0.1863	0.1026	0.0832	0.0817

Scheme 1



The study of thermal decomposition of complex **2** (m.p. 84 °C, with decomp.) revealed a chain of transformations with stepwise elimination of organic fragments and the formation of inorganic mixed-metal telluride Fe_2SnTe_2 (Scheme 2).

Scheme 2

$$[CpFe(CO)_2]_2Sn(TePh)_2 \xrightarrow[-4\ CO]{84\text{—}165\ ^\circ C} \mathbf{2}$$

$$\longrightarrow [CpFe]_2Sn(TePh)_2 \xrightarrow[-2\ Ph]{182\ ^\circ C}$$

$$\longrightarrow [CpFe]_2SnTe_2 \xrightarrow[-2\ Cp]{350\text{—}450\ ^\circ C} Fe_2SnTe_2$$

According to the data of X-ray study of complex $[Cp'Fe(CO)_2]_2SnCl_2$ (**3**, Cp′ = MeC_5H_4) (Fig. 2, see

Table 2. Main geometric characteristics (bond lengths (*d*) and bond angles (ω)) of complexes **2**—**5**

Complex	Bond	*d*/Å	Angle	ω/deg
2	Te(1)—Sn(1)	2.793(1)	C(15)—Te(1)—Sn(1)	96.6(3)
	Te(2)—Sn(1)	2.796(1)	C(21)—Te(2)—Sn(1)	99.9(3)
	Sn(1)—Fe(2)	2.581(2)	Fe(2)—Sn(1)—Fe(1)	122.83(6)
	Sn(1)—Fe(1)	2.578(2)	Te(1)—Sn(1)—Te(2)	98.28(4)
3	Sn(1)—Cl(1)	2.393(6)	Cl(1)—Sn(1)—Cl(2)	94.5(2)
	Sn(1)—Cl(2)	2.461(6)	Fe(1)—Sn(1)—Fe(2)	131.76(7)
	Sn(1)—Fe(1)	2.500(3)		
	Sn(1)—Fe(2)	2.508(3)		
4	Sn(1)—Fe(2)	2.568(2)	Fe(2)—Sn(1)—Fe(1)	120.51(6)
	Sn(1)—Fe(1)	2.570(2)	Te(1)—Sn(1)—Te(2)	105.09(5)
	Sn(1)—Te(1)	2.790(2)	C(17)—Te(1)—Sn(1)	99.4(2)
	Sn(1)—Te(2)	2.819(14)	C(23)—Te(2)—Sn(1)	103.1(2)
5	W(1)—Te(1*)	2.835(1)	Te(1*)—W(1)—Te(1)	87.21(4)
	W(1)—Te(1)	2.835(1)	Fe(1*)—Sn(1)—Fe(1)	125.53(9)
	Sn(1)—Fe(1*)	2.535(2)	Te(1)—Sn(1)—Te(1*)	85.56(5)
	Sn(1)—Fe(1)	2.535(2)	W(1)—Te(1)—Sn(1)	93.61(3)
	Sn(1)—Te(1)	2.879(1)		
	Sn(1)—Te(1*)	2.879(1)		

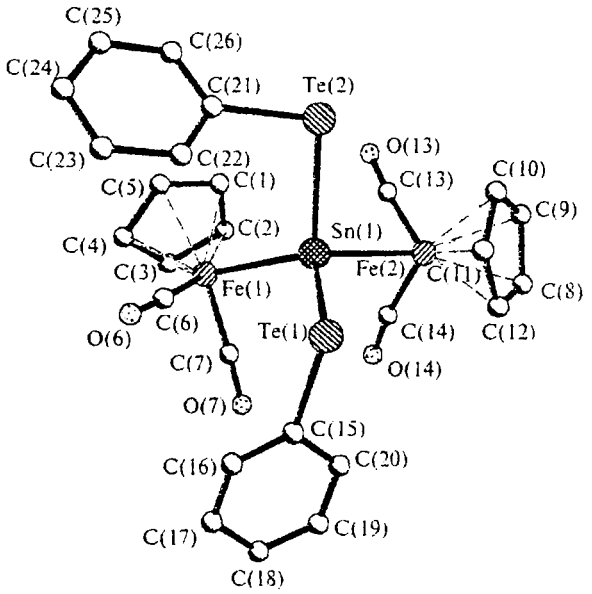


Fig. 1. Molecular structure of complex **2**.

Table 2), replacement of cyclopentadienyl ligands by methylcyclopentadienyl ones has little effect on the dichloride geometry (the average Fe—Sn and Sn—Cl bond lengths are 2.500(1) and 2.430(6) Å, respectively). In the case of complex $[Cp'Fe(CO)_2]_2Sn(TePh)_2$ (**4**), obtained analogously to complex **2** by treatment of dichloride **3** with sodium phenyltelluride and characterized by X-ray analysis (Fig. 3, see Table 2), the phenyl groups are in *syn*-conformation, which does not lead to appreciable changes in the Fe—Sn and Te—Sn bond lengths (2.569(2) and 2.800(2) Å, respectively). Thermal decomposition of **4** at the stage of elimination of CO and phenyl groups occurs analogously to that of the cyclopentadienyl complex **2** (Scheme 3).

Scheme 3

$$[Cp'Fe(CO)_2]_2Sn(TePh)_2 \xrightarrow[-4\ CO]{80-151\ ^\circ C} \mathbf{4}$$

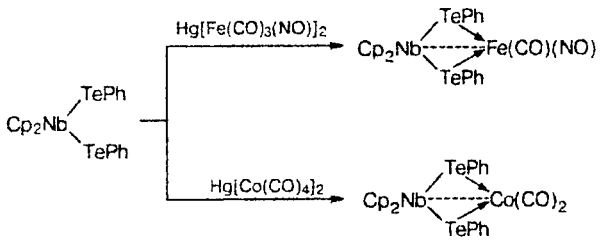
$$\mathbf{4} \longrightarrow [Cp'Fe]_2Sn(TePh)_2 \xrightarrow[-2\ Ph]{185\ ^\circ C}$$

$$[Cp'Fe]_2Sn(TePh)_2 \longrightarrow [Cp'Fe]_2SnTe_2 \xrightarrow[-2\ Cp']{217\ ^\circ C} Fe_2SnTe_2$$

However, elimination of methylcyclopentadienyl ligands occurs at a lower temperature (217 °C) and in a narrower temperature range than elimination of cyclopentadienyl ligands in complex **2** (at 350—450 °C). It might be associated with the possibility of easy decomposition of methylcyclopentadienyl ligands with the formation of a stable fulvene molecule $C_5H_4{=}CH_2$ and an M—H fragment. As in the case of complex **2**, Fe_2SnTe_2 is the final product of thermal decomposition.

It was of interest to test the possibility of using two terminal phenyltellurium groups in chelation reactions, the more so that similar examples are known for $Cp_2Nb(TePh)_2$ (Scheme 4)[6] (unfortunately, no X-ray studies of the adducts were performed in this case[6]).

Scheme 4



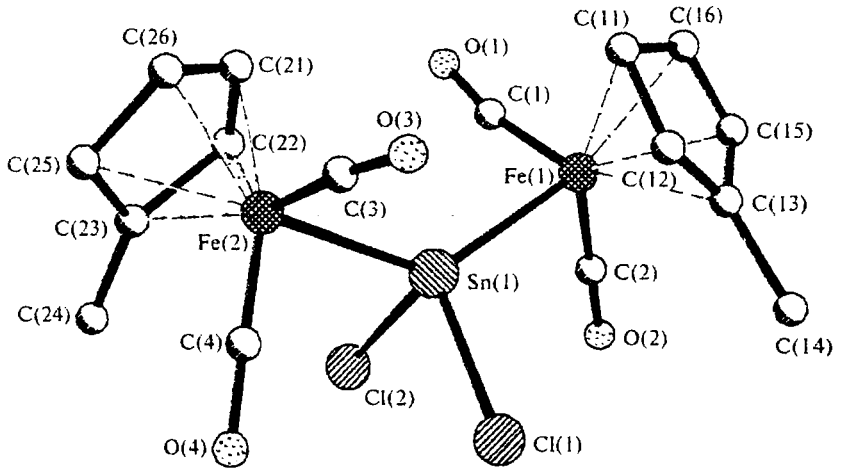


Fig. 2. Molecular structure of complex **3**.

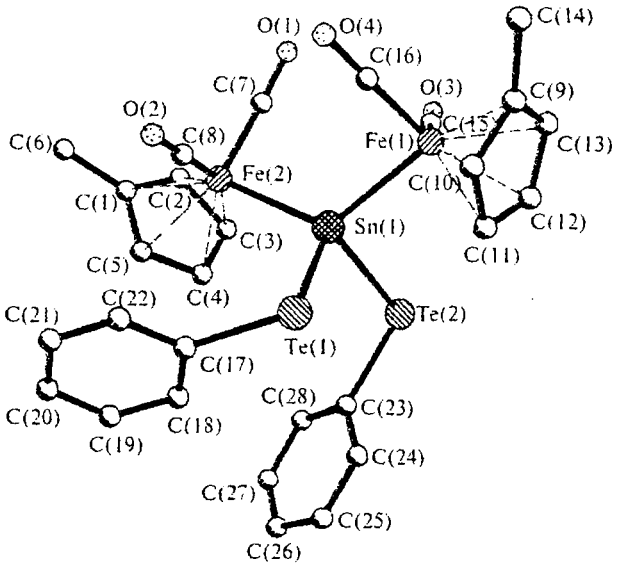


Fig. 3. Molecular structure of complex **4**.

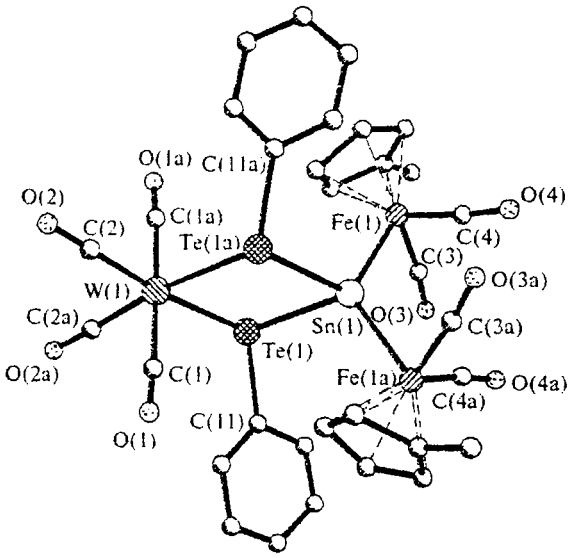
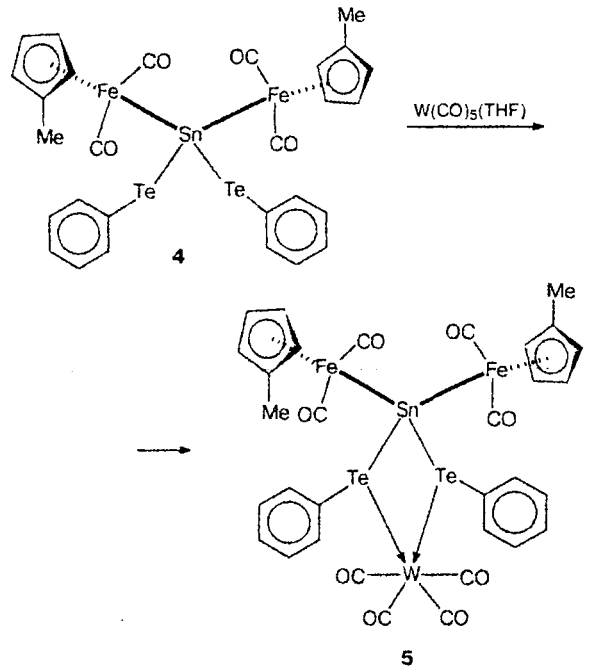


Fig. 4. Molecular structure of complex **5**.

We studied the reactions of complex **4** with $W(CO)_5(THF)$ as well as the reactions of compound **2** with carbonyl complexes $M(CO)_5(THF)$ (M = Cr, W) or with $[Me_3PtI]_4$.

Black prismatic crystals of adduct $[Cp'Fe(CO)_2]_2Sn(TePh)_2[W(CO)_4]$ (**5**) were isolated in the reaction of $[Cp'Fe(CO)_2]_2Sn(TePh)_2$ (**4**) with $W(CO)_5(THF)$ (Scheme 5).

Scheme 5



The X-ray study of complex **5** (Fig. 4, see Table 2) showed the formation of a chelate cycle, the Fe—Sn and Sn—Te bond lengths (2.535(2) and 2.879(1) Å, respectively) being relatively little changed compared to corresponding values in molecule **4**. Thermal decomposition of complex **5** was not studied because of the small amount of the substance.

Only the heavy-atom Fe_2SnTe_2W core geometry of black crystals of the adduct $[CpFe(CO)_2]_2Sn(TePh)_2[W(CO)_4]$ (**6**), which is the product of the reaction of complex **2** with $W(CO)_5(THF)$, is determined to date by X-ray analysis. It is analogous to that found for the methylcyclopentadienyl chelate **5** (we failed to refine the structure of **6** because of the poor quality of the single crystals). Thermal decomposition of adduct **6** occurs with successive elimination of carbonyl groups and organic ligands to give a mixed-metal telluride Fe_2SnTe_2W (Scheme 6).

Scheme 6

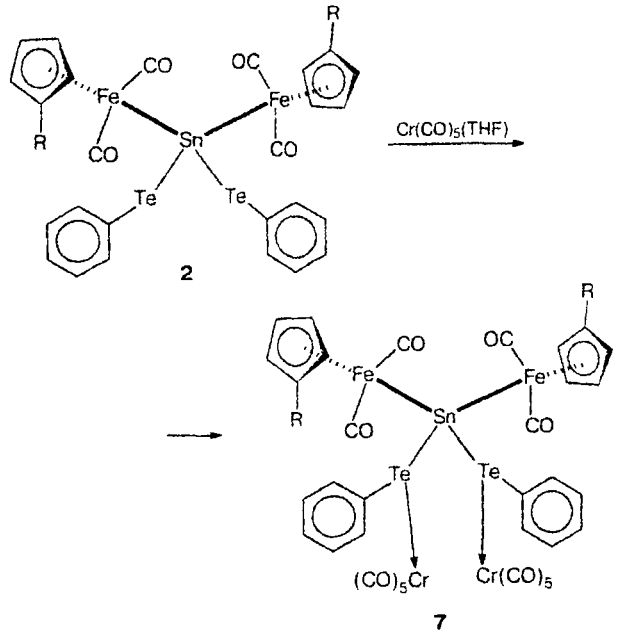
$$[CpFe(CO)_2]_2Sn(TePh)_2[W(CO)_4] \xrightarrow[-8\ CO]{180-210\ ^\circ C} \mathbf{6}$$

$$\mathbf{6} \longrightarrow [CpFe]_2Sn(TePh)_2W \xrightarrow[-2\ Ph,\ -2\ Cp]{245-280\ ^\circ C} Fe_2SnTe_2W$$

The reaction of $[CpFe(CO)_2]_2Sn(TePh)_2$ (**2**) with $Cr(CO)_5(THF)$ results in the formation of cluster $[CpFe(CO)_2]_2Sn(PhTe)_2[Cr(CO)_5]_2$ (**7**, Scheme 7) as red-brown crystals. We have failed to characterize the structure of **7** by X-ray analysis as yet, though its

composition can be unambiguously established on the basis of the differential scanning calorimetry (DSC) data on quantitative elimination of carbonyl and phenyl groups in the course of thermal decomposition, as well as from the IR spectra indicating the presence of $CpFe(CO)_2$ and $Cr(CO)_5$ groups.

Scheme 7



Thermal decomposition of complex **7** occurs with successive elimination of organic fragments to give a mixed-metal telluride $Fe_2SnTe_2Cr_2$ (Scheme 8).

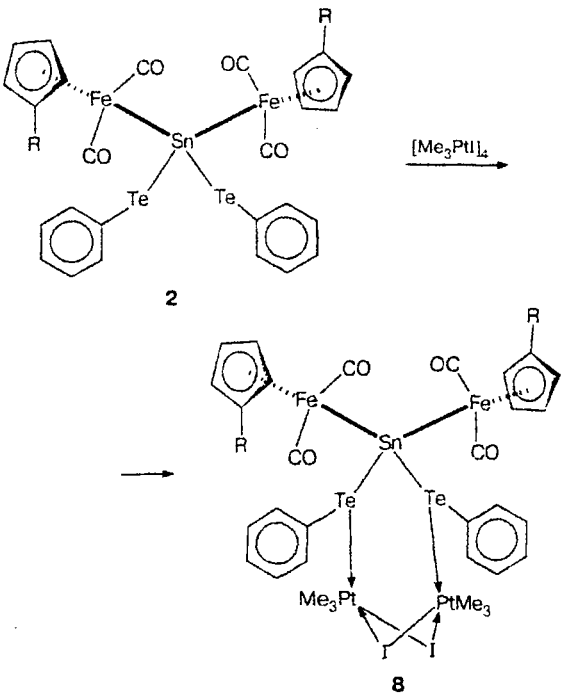
Scheme 8

$$[CpFe(CO)_2]_2Sn(TePh)_2[Cr(CO)_5]_2 \xrightarrow[-14\ CO]{108-150\ ^\circ C} [CpFe]_2Sn(TePh)_2Cr_2 \xrightarrow[-2\ Ph]{160-187\ ^\circ C} [CpFe]_2SnTe_2Cr_2 \xrightarrow[-2\ Cp]{400-470\ ^\circ C} Fe_2SnTe_2Cr_2$$

Finally, the reaction of complex **2** with $[Me_3PtI]_4$ resulted in $[CpFe(CO)_2]_2Sn(TePh)_2(Me_3PtI)_2$ (**8**, Scheme 9) as black-brown microcrystals, whose composition was established by elemental analysis, IR spectra, and quantitative elimination of organic fragments in the course of thermal decomposition (Scheme 10, DSC data).

Two platinum atoms are likely to be bonded to each other through bridging iodine atoms, as in the known complex with a bridging diphenylditellurium molecule[7]

Scheme 9



Scheme 10

$$[CpFe(CO)_2]_2Sn(TePh)_2[Me_3Pt(\mu\text{-}I)]_2 \xrightarrow[-4\ CO,\ -6\ Me]{215\ ^\circ C} \mathbf{8} \longrightarrow [CpFe]_2Sn(TePh)_2[Pt(\mu\text{-}I)]_2 \xrightarrow[-2\ Ph]{227\ ^\circ C} \longrightarrow Cp_2Fe_2SnTe_2(PtI)_2 \xrightarrow[-2\ Cp,\ -2\ I]{350\ ^\circ C} Fe_2SnTe_2Pt$$

or in the heterometallic complex with the $(CpCrSCMe_3)_2S$ bridge we described previously.[8]

Experimental

All operations associated with the synthesis and isolation of complexes were performed in a pure Ar atmosphere using dehydrated solvents. Complexes **1** and **3** were synthesized according to the known procedure.[3] IR spectra were recorded on a Specord 75 IR spectrophotometer in KBr pellets. Thermal decomposition of the complexes was studied by DSC on a Mettler TA-4000 thermoanalyzer. The specimens were heated in a dry nitrogen atmosphere at a constant rate varied from 2 to 7 deg min^{-1}. The weight loss was monitored at each stage and after completion of the experiment.

Bis(η^5-cyclopentadienyldicarbonyliron)tin dichloride, $[CpFe(CO)_2]_2SnCl_2$ (1). To a mixture of anhydr. $SnCl_2$ (5.94 g, 31.26 mmol) and $Cp_2Fe_2(CO)_4$ (8.85 g, 25 mmol), THF (50 mL) was added in an argon atmosphere with stirring and

the mixture was boiled for 30 min. The solution was concentrated *in vacuo* and cooled to −20 °C. This resulted in precipitation of orange crystals, which were filtered off using a Schott filter and washed with ether. The yield was 10.16 g (75%). IR (KBr), ν/cm^{-1}: 495 w, 560 m, 620 w, 840 w, 995 w, 1920 m, 1960 vs, 2000 vs. Single crystals were used for X-ray study.

Bis(η^5-cyclopentadienyldicarbonyliron)tin bis(phenyltelluride), $[CpFe(CO)_2]_2Sn(TePh)_2$ (2). To a mixture of Ph_2Te_2 (2.00 g, 4.9 mmol) and $NaBH_4$ (0.76 g, 20 mmol), ethanol (25 mL) was added in an argon atmosphere with stirring. After several minutes, the solution decolorized. To the PhTeNa solution obtained, $[CpFe(CO)_2]_2SnCl_2$ (2.66 g, 4.9 mmol) was added and the solution immediately turned red-brown. The mixture was stirred for 30 min and the solvent was removed *in vacuo*. The residue was extracted with boiling hexane (5×20 mL). Cooling the extract to −20 °C gave brown crystals of complex **2**. The yield was 3.38 g (78%). IR (KBr), ν/cm^{-1}: 505 m, 562 s, 580 m, 630 m, 725 s, 840 w, 829 w, 989 w, 1010 w, 1926 vs, 1953 s, 1965 s, 2002 s. Found (%): C, 35.0; H, 2.2. $C_{26}H_{20}Fe_2O_4SnTe_2$. Calculated (%): C, 35.4; H, 2.3. Single crystals were used for X-ray study.

DSC study showed quantitative elimination of the following fragments:

	CO	Ph	C_5H_5
Found (%)	13.0	16.6	14.9
Calculated (%)	12.7	17.5	14.7

Bis(η^5-methylcyclopentadienyldicarbonyliron)tin dichloride, $[Cp'Fe(CO)_2]_2SnCl_2$ (3). To a mixture of anhydr. $SnCl_2$ (1.0 g, 5.26 mmol) and $[Cp'Fe(CO)_2]_2$ (2.0 g, 5.24 mmol), benzene (30 mL) was added in an argon atmosphere. The reaction mixture was boiled for 4 h with stirring. The solution was filtered and concentrated to 1/3 of its volume, and heptane (10 mL) was added. Cooling the solution to −20 °C resulted in precipitation of crystals of complex **3**, which were filtered off using a Schott filter and washed with ether. The yield was 1.9 g (63%). Found (%): C, 33.6; H, 3.2. $C_{16}H_{14}Cl_2Fe_2O_4Sn$. Calculated (%): C, 33.6; H, 2.5. IR (KBr), ν/cm^{-1}: 500 m, 580 s, 620 m, 865 m, 920 w, 1020 w, 1940 vs, 1980 vs, 2920 w, 3080 w. Single crystals were used for X-ray study.

Bis(η^5-methylcyclopentadienyldicarbonyliron)tin bis(phenyltelluride), $[Cp'Fe(CO)_2]_2Sn(TePh)_2$ (4). To an orange solution of Ph_2Te_2 (1.42 g, 3.47 mmol) in 30 mL of ethanol, $NaBH_4$ (0.4 g, 14.28 mmol) was added portionwise in an Ar atmosphere until decolorization indicated the formation of PhTeNa. Then $[Cp'Fe(CO)_2]_2SnCl_2$ (2.0 g, 3.47 mmol) was added and the solution immediately turned red-brown. The mixture was stirred for 1 h and the solvent was removed *in vacuo*. The residue was extracted with boiling hexane (3×20 mL). Cooling the extract to −20 °C gave red-brown crystals of complex **4**. The yield was 2.58 g (82%). Found (%): C, 37.0; H, 2.6. $C_{28}H_{24}Fe_2O_4SnTe_2$. Calculated (%): C, 36.9; H, 2.7. IR (KBr), ν/cm^{-1}: 580 s, 620 m, 690 w, 730 s, 830 m, 1010 s, 1920 vs, 1935 vs, 1970 vs, 1994 vs, 2960 s, 3040 s. Single crystals were used for X-ray study.

DSC study showed quantitative elimination of the following fragments:

	Ph	MeC_5H_5
Found (%)	16.5	16.8
Calculated (%)	16.9	17.4

Bis(η^5-methylcyclopentadienyldicarbonyliron)tin-bis(μ-phenyltelluride)tungsten tetracarbonyl, $[Cp'Fe(CO)_2]_2Sn(TePh)_2[W(CO)_4]$ (5). A solution of $W(CO)_6$ (0.35 g, 1 mmol) in 25 mL of THF, placed in a quartz Schlenk vessel cooled using a water-jacket, was irradiated for 3 h in an Ar atmosphere with stirring. After switching off irradiation, $[Cp'Fe(CO)_2]_2Sn(TePh)_2$ (**4**) (0.65 g, 0.71 mmol) was added to the solution and the combined solution was evaporated *in vacuo*. The residue was dissolved in benzene at 70 °C. Cooling the solution gave a yellow resin, which was separated from the mother liquor by adding heptane (30 mL). At room temperature, black prismatic crystals of complex **5** precipitated, which were freed from the admixture of resin by washing with acetone and heptane. The yield was 0.07 g (8%). Found (%): C, 30.2; H, 2.4. $C_{32}H_{20}Fe_2O_8SnTe_2W$. Calculated (%): C, 31.9; H, 2.0. IR (KBr), ν/cm^{-1}: 565 s, 623 m, 684 w, 721 s, 832 w, 848 w, 995 w, 1007 s, 1026 s, 1057 w, 1872 vs, 1980 vs, 1995 vs, 2114 vs, 2920 br, w. Single crystals were used for X-ray study.

Bis(η^5-cyclopentadienyldicarbonyliron)tin-bis(μ-phenyltelluride)tungsten tetracarbonyl, $[CpFe(CO)_2]_2Sn(TePh)_2[W(CO)_4]$ (6). A solution of $W(CO)_6$ (0.46 g, 1.3 mmol) in 22 mL of THF was irradiated for 2.5 h in an Ar atmosphere with stirring. The reaction was conducted in a quartz Schlenk vessel cooled using a water jacket. After switching off irradiation, the yellow solution was poured into a flask containing 0.62 g (0.7 mmol) of $[CpFe(CO)_2]_2Sn(TePh)_2$ (**3**) and evaporated until beginning of crystallization, and heptane (20 mL) was added dropwise. At room temperature, black crystals of complex **6** precipitated, which were filtered off and washed with heptane. The yield was 0.04 g (5%). Found (%): C, 30.7; H, 1.7. $C_{30}H_{20}Fe_2O_8SnTe_2W$. Calculated (%): C, 30.6; H, 1.7. IR (KBr), ν/cm^{-1}: 500 m, 560 s, 620 m, 690 w, 720 m, 830 w, 990 m, 1010 m, 1050 m, 1830 vs, 1880 vs, 1940 vs, 1960 vs, 2000 vs, 2020 vs. Single crystals were used for X-ray study.

Bis(η^5-cyclopentadienyldicarbonyliron)tin-bis(μ-phenyltelluridechromiumpentacarbonyl), $[CpFe(CO)_2]_2Sn(PhTe)_2[Cr(CO)_5]_2$ (7). A solution of $Cr(CO)_6$ (0.22 g, 1 mmol) in 20 mL of THF was irradiated for 1 h in an Ar atmosphere with stirring by a magnetic stirrer. The reaction was conducted in a quartz Schlenk vessel equipped with a reflux condenser until elimination of 1 mmol CO and the formation of orange solution. To the solution obtained, a solution of $[CpFe(CO)_2]_2Sn(TePh)_2$ (0.885 g, 1 mmol) in 10 mL of THF was added and the combined solution was evaporated to dryness *in vacuo*. The residue was dissolved in 5 mL of CH_2Cl_2, silica gel was added, and the mixture was evaporated to dryness *in vacuo* and chromatographed on SiO_2. First, the yellow-orange band was eluted with a hexane–benzene mixture, followed by elution of the dark-red band with ether. The ethereal eluent was concentrated and hexane (5 mL) was added. Cooling the solution to −20 °C gave brown-red crystals of complex **5**. The yield was 0.1 g (8%). IR (KBr), ν/cm^{-1}: 450 w, 550 m, 650 s, 720 w, 830 m, 850 m, 1000 w, 1430 m, 1900 vs, 1980 s, 2010 m, 2030 vs, 2050 m, 2070 vs.

DSC study showed quantitative elimination of the following fragments:

	CO	Ph
Found (%)	31.5	11.7
Calculated (%)	31.0	12.2

Bis(η^5-cyclopentadienyldicarbonyliron)tin-bis(μ-phenyltelluridetrimethylplatinum-μ-iodide), $[CpFe(CO)_2]_2Sn(TePh)_2(Me_3PtI)_2$ (8). A mixture of 0.24 g of $[CpFe(CO)_2]_2Sn(TePh)_2$ (**2**) and trimethylplatinum iodide (0.20 g), taken in a 1 : 2 molar ratio, in 15 mL of benzene was heated for 2 h at 60 °C. The cherry-red solution gradually turned darker until red-brown and TLC (Silufol, a benzene–hexane (1 : 1) mixture as eluent) revealed disappearance of the

dark-red spot (**2**) with R_f 0.45 and the appearance of a new brown spot with R_f 0.7. Then the reaction mixture was concentrated to 5 mL, hexane (5 mL) was added, and the combined mixture was kept for ~24 h at −20 °C. The tiny black-brown crystals of complex **8** that precipitated were separated, washed with hexane, and dried *in vacuo*. The yield was 0.16 g (36%). Found (%): C, 23.4; H, 2.1. $C_{32}H_{38}Fe_2I_2O_4Te_2Pt_2Sn$. Calculated (%): C, 23.8; H, 2.3. IR (KBr), ν/cm^{-1}: 440 m, 520 w, 570 s, 625 m, 720 w, 845 w, 1940 s, 1950 s, 2000 s, 2880 w, 2930 w, 2960 w.

DSC study showed quantitative elimination of the following fragments:

	CO + Me	Ph
Found (%)	13.3	9.5
Calculated (%)	12.5 (6.9 for CO, 5.6 for Me)	9.5

X-ray study of complexes 1—5. Experimental sets of reflections ($\theta/2\theta$ scan, λ(Mo-Kα)) were collected on four-circle automatic Siemens P3/Pc (for **1**, **2**, **4**, **5**) and Enraf Nonius CAD-4 diffractometers (for **3**) at 293 K. The structures were solved by direct methods. All non-hydrogen atoms were refined anisotropically in the full-matrix approximation. All calculations were performed using the SHELXTL Plus 3.0 program complex (PC version).[9] Selected crystallographic parameters of complexes **1—5** are listed in Table 1. Atomic coordinates and thermal parameters of the atoms are deposited at the Cambridge Structural Database.

The X-ray study was carried out in the X-ray Structural Center (at the A. N. Nesmeyanov Institute of Organoelement Compounds of the Russian Academy of Sciences).

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References

1. F. Bonati and G. Wilkinson, *J. Chem. Soc.*, 1964, 179.
2. A. N. Nesmeyanov, K. N. Anisimov, N. E. Kolobova, and V. V. Skripkin, *Izv. Akad. Nauk SSSR, Ser. Khim.*, 1966, 1292 [*Bull. Acad. Sci. USSR, Div. Chem. Sci.*, 1966, **15** (Engl. Transl.)].
3. P. Hackett and A. R. Manning, *J. Chem. Soc., Dalton Trans.*, 1972, 1487.
4. K. Merzweiler and H. Kraus, *Z. Naturforsch.*, 1994, **B49**, 621.
5. J. E. O'Connor and E. R. Corey, *Inorg. Chem.*, 1967, **6**, 968.
6. M. Sato and T. Youshida, *J. Organomet. Chem.*, 1997, **94**, 403.
7. H. B. Singh, V. Regini, J. P. Jasinski, F. S. Paight, and R. J. Butcher, *J. Organomet. Chem.*, 1994, **466**, 283.
8. A. A. Pasynskii, Y. V. Torubaev, S. E. Nefedov, I. L. Eremenko, O. G. Ellert, V. K. Belsky, and A. I. Stastch, *J. Organomet. Chem.*, 1997, 536.
9. G. M. Sheldrick, in *Crystallographic Computing 3: Data Collection, Structure Determination, Proteins, and Databases*, Clarendon Press, New York, 1985, 175.

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