# Chelating and Tellurolate Ligand-Transfer Studies of the Complex fac-[Fe(CO)<sub>3</sub>(TePh)<sub>3</sub>]<sup>-</sup>: Crystal Structures of Heterodinuclear (CO)<sub>3</sub>Mn(μ-TePh)<sub>3</sub>Fe(CO)<sub>3</sub> and CpNi(TePh)(PPh<sub>3</sub>)

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Complex fac-[Fe(CO)<sub>3</sub>(TePh)<sub>3</sub>]<sup>-</sup> was employed as a "metallo chelating" ligand to synthesize the neutral (CO)<sub>3</sub>Mn( $\mu$ -TePh)<sub>3</sub>Fe(CO)<sub>3</sub> obtained in a one-step synthesis by treating fac-[Fe(CO)<sub>3</sub>(TePh)<sub>3</sub>]<sup>-</sup> with fac-[Mn-(CO)<sub>3</sub>(CH<sub>3</sub>CN)<sub>3</sub>]<sup>+</sup>. It seems reasonable to conclude that the d<sup>6</sup> Fe(II) [(CO)<sub>3</sub>Fe(TePh)<sub>3</sub>]<sup>-</sup> fragment is isolobal with the d<sup>6</sup> Mn(I) [(CO)<sub>3</sub>Mn(TePh)<sub>3</sub>]<sup>2</sup>- fragment in complex (CO)<sub>3</sub>Mn( $\mu$ -TePh)<sub>3</sub>Fe(CO)<sub>3</sub>. Addition of fac-[Fe(CO)<sub>3</sub>(TePh)<sub>3</sub>]<sup>-</sup> to the CpNi(I)(PPh<sub>3</sub>) in THF resulted in formation of the neutral CpNi(TePh)(PPh<sub>3</sub>) also obtained from reaction of CpNi(I)(PPh<sub>3</sub>) and [Na][TePh] in MeOH. This investigation shows that fac-[Fe(CO)<sub>3</sub>(TePh)<sub>3</sub>]<sup>-</sup> serves as a tridentate metallo ligand and tellurolate ligand-transfer reagent. The study also indicated that the fac-[Fe(CO)<sub>3</sub>(SePh)<sub>3</sub>]<sup>-</sup> may serve as a better tridentate metallo ligand and chalcogenolate ligand-transfer reagent than fac-[Fe(CO)<sub>3</sub>(TePh)<sub>3</sub>]<sup>-</sup> in the syntheses of heterometallic chalcogenolate complexes.

#### INTRODUCTION

The investigation of metal-tellurolate chemistry has been actively pursued recently, motivated primarily by the bonding diversity, reactivity, and the potential use as precursors for metal-telluride materials.<sup>2</sup>

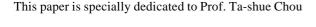
Recent work in this laboratory has shown that fac-[Fe(CO)<sub>3</sub>(SePh)<sub>3</sub>] can serve as a chelating and selenolate ligand-transfer reagent to synthesize (1) distorted square planar [Ni(CO)(SePh)<sub>3</sub>]<sup>-</sup>, the potential biomimetic nickel-site structure of CO dehydrogenases, from reaction of fac-[Fe(CO)<sub>3</sub>(SePh)<sub>3</sub>]<sup>-</sup>, [CpNi(CO)]<sub>2</sub>, and (SePh)<sub>2</sub> via chelating and selenolate ligand-transfer,<sup>3</sup> (2) heterotrimetallic Fe(II)-M-Fe(II)-selenolate complexes  $(CO)_3$ Fe $(\mu$ -SePh $)_3$ M $(\mu$ - $SePh)_3Fe(CO)3(M = Fe, Ni, Zn, Cd)$  with a unique homoleptic hexaselenolatometal (MSe<sub>6</sub>) core, respectively,<sup>4</sup> and  $[(CO)_3Mn(\mu-S-2-S-C_4H_3S)_3Ni(\mu-SePh)_3Fe(CO)_3]^$ heteroleptic hexachalcogenolatonickel (NiS<sub>3</sub>Se<sub>3</sub>) core;<sup>5</sup> and (3) neutral  $[Fe(CO)_2(phen)(SePh)_2]$  (phen = 1, 10-phenanthroline) requiring intermetal selenolate and CO ligands-transfer from fac-[Fe(CO)<sub>3</sub>(SePh)<sub>3</sub>] to [Fe(phen)<sub>3</sub>]<sup>2+</sup> (redistribution reaction).4

Here we examined the reactivity of the analogue, iron(II)-tellurolate species *fac*-[Fe(CO)<sub>3</sub>(TePh)<sub>3</sub>]<sup>-,6</sup> by react-

ing it with fac-[Mn(CO)<sub>3</sub>(CH<sub>3</sub>CN)<sub>3</sub>]<sup>+</sup> and CpNi(I)(PPh<sub>3</sub>) individually. Specifically, synthesis and characterization of the heterodinuclear Fe<sup>II</sup>-Mn<sup>I</sup>-tellurolate and the neutral Ni<sup>II</sup>-tellurolate complex by employing fac-[Fe(CO)<sub>3</sub>(TePh)<sub>3</sub>]<sup>-</sup> as a chelating and intermetal tellurolate ligand-transfer reagent are described.

## RESULTS AND DISCUSSION

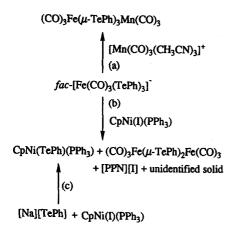
As illustrated in Scheme Ia, when a THF solution of fac- $[Fe(CO)_3(TePh)_3]^-$  and  $[Mn(CO)_3(CH_3CN)_3][PF_6]$  are stirred under  $N_2$ , a reaction ensues over the course of 12 h at ambient temperature to yield the neutral heterodinuclear  $(CO)_3Mn(\mu\text{-TePh})_3Fe(CO)_3$  (1) in 80% isolated yield after removal of  $[PPN][PF_6]$  by diethyl-ether/hexane and recrystallization with hexane-diethyl ether. The brown purple, heterodimetallic complex 1 which is soluble in common organic solvents like THF and  $CH_2Cl_2$ , displays no tendency to decompose under nitrogen at room temperature. The carbonyl stretching bands 2063 s, 2003 vs of complex 1 (compared to  $\nu(CO)$  2021 s, 1963 s of fac- $[Fe(CO)_3(TePh)_3]^-)^6$  were assigned to the  $Fe^{II}(CO)_3$  fragment, and the stretching bands 1923 s, 2003 vs (compared to  $\nu(CO)$  1896 s, 1970 s of





[(CO)<sub>3</sub>Mn(μ-TePh)<sub>3</sub>Mn(CO)<sub>3</sub>]<sup>-</sup>)<sup>8</sup> were attributed to the CO stretching bands of the Mn<sup>I</sup>(CO)<sub>3</sub> fragment of complex 1. The IR  $\nu$ (CO) data suggested its formulation of (CO)<sub>3</sub>Mn(μ-TePh)<sub>3</sub>Fe(CO)<sub>3</sub> with the two metal ions, Mn<sup>I</sup> and Fe<sup>II</sup>, being bridged by three phenyltellurolates, which was also confirmed by X-ray crystallography. <sup>1</sup>H NMR spectra are consistent with the presence of the low-spin octahedrally coordinated d<sup>6</sup> Fe(II) and d<sup>6</sup> Mn(I) ions with facial carbonyls. In light of this result, the neutral (CO)<sub>3</sub>Mn(μ-TePh)<sub>3</sub>Fe(CO)<sub>3</sub> feature octahedral coordination about each of the Mn(I) and Fe(II) ions with three terminal carbonyls in the facial positions and bears a closer structural resemblance to the reported [(CO)<sub>3</sub>Mn(μ-TePh)<sub>3</sub>Mn(CO)<sub>3</sub>]<sup>-</sup>,<sup>8</sup> i.e. the d<sup>6</sup> Fe(II) [Fe(CO)<sub>3</sub>]<sup>2+</sup> fragment is isolobal with the d<sup>6</sup> Mn(I) [Mn(CO)<sub>3</sub>] fragment.<sup>9</sup>

#### Scheme I



The neutral complex  ${\bf 1}$  appears to have no precedent in heterodinuclear-tellurolate chemistry.

In contrast, reactions of fac-[Fe(CO)<sub>3</sub>(TePh)<sub>3</sub>] $^-$  with  $Cd(NO_3)_2\cdot 4H_2O$ ,  $Zn(BF_4)_2\cdot 6H_2O$ ,  $Ni(NO_3)_2\cdot 6H_2O$ ,  $Fe(ClO_4)_2\cdot H_2O$  were not successful in synthesizing heterotrimetallic tellurolates  $(CO)_3Fe(\mu$ -TePh)<sub>3</sub>M( $\mu$ -TePh)<sub>3</sub>Fe(CO)<sub>3</sub> (M = Cd, Zn, Ni, Fe) with a homoleptic hexatellurolatometal core M(Te)<sub>6</sub>, respectively. This is in contrast to the observation that heterotrimetallic selenolate complexes  $(CO)_3Fe(\mu$ -SePh)<sub>3</sub>M( $\mu$ -SePh)<sub>3</sub>Fe(CO)<sub>3</sub> were readily obtained under similar reaction conditions.<sup>4</sup>

In comparison, the addition of one equiv of fac-[Fe(CO)<sub>3</sub>(TePh)<sub>3</sub>]<sup>-</sup> to a THF solution of CpNi(I)(PPh<sub>3</sub>) under N<sub>2</sub> led to the isolation of dark red-brown CpNi(TePh)(PPh<sub>3</sub>), the known (CO)<sub>3</sub>Fe( $\mu$ -TePh)<sub>2</sub>Fe(CO)<sub>3</sub>, <sup>10</sup> [PPN][I], and the unidentified solid (Scheme Ib). The complex CpNi(TePh)(PPh<sub>3</sub>) (2) is soluble and stable in THF, and crystallized from THF-hexane at -15 °C. The most likely explanation for the formation of complex 2 is that fac-[Fe(CO)<sub>3</sub>(TePh)<sub>3</sub>] $^-$  was employed as an

intermetal tellurolate ligand-transfer reagent. The  $^{1}H$  and  $^{13}C$  NMR spectrum of complex **2** shows the expected signals ( $^{1}H$  NMR ( $C_{4}D_{8}O$ ):  $\delta$  4.99 (s) ppm (Cp);  $^{13}C$  NMR ( $C_{4}D_{8}O$ ):  $\delta$  93.96 (s) ppm (Cp)) for the Cp ring involved and displayed characteristics of diamagnetic  $d^{8}$  Ni<sup>II</sup> species. Additionally, when CpNi(I)(PPh<sub>3</sub>) was reacted in stoichiometric proportions with [Na][TePh] (obtained from reaction of [Na][BH<sub>4</sub>] and (TePh)<sub>2</sub> in 2:1 ratio) in MeOH under N<sub>2</sub>, the neutral red brown complex **2** was also obtained after being separated from NaI (Scheme Ic).

The X-ray structural analysis of complex 1 is depicted in Fig. 1; selected bond distances and angles are collected in Table 2. In a first approximation, the MnTe<sub>3</sub>(CO)<sub>3</sub>/FeTe<sub>3</sub>(CO)<sub>3</sub> fragments may be described as face-sharing octahedra. The phenyl groups of three bridging benzenetellurolates in 1 form a regular propeller-like arrangement around the Te<sub>3</sub> plane defined by the three telluriums. In a sense, neutral complex 1 is isostructural with the anion  $[(CO)_3Mn(\mu\text{-TePh})_3Mn(CO)_3]^-$ . Complex 1 contains discrete dinuclear units in which the d<sup>6</sup> Mn(I) and d<sup>6</sup> Fe(II) ions are unsymmetrically bridged by three benzenetellurolates. The average Fe-TePh bond length of 2.631(1) Å in 1 is comparable with the reported terminal Fe-TePh bond length of 2.630(4) Å in fac-[Fe(CO)<sub>3</sub>(TePh)<sub>3</sub>]<sup>-.6</sup> The bridging Mn-TePh distances (average 2.654(1) Å) in complex 1 are comparable to those in  $[(CO)_3Mn(\mu\text{-TePh})_3Mn(CO)_3]^-$  (average 2.666(2) Å),<sup>8</sup> in cis-[Mn(CO)<sub>4</sub>(TePh)<sub>2</sub>]<sup>-</sup> (average 2.673(1) Å).<sup>8</sup> The distinctly differential Fe-TePh (2.631(1) Å) and Mn-TePh (2.654(1) Å) bond distances (Fe-TePh distance is ca. 0.02 Å shorter than the Mn-TePh distance) also support the formation of complex 1 as a heterodinuclear Fe<sup>II</sup>-Mn<sup>I</sup>-tellurolate complex consistent with the conclusion in IR  $\nu(CO)$  data. The larger Te-Fe-Te angle (average 82.08(3)°) compared with the Te-Mn-Te angles (average

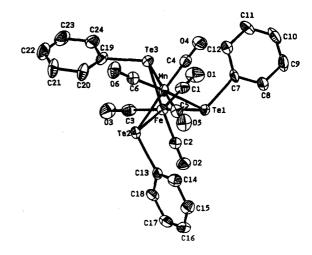


Fig. 1. ORTEP drawing and labeling scheme of the neutral (CO) $_3$ Mn( $\mu$ -TePh) $_3$ Fe(CO) $_3$  with thermal ellipsoids drawn at the 30% probability level.

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Table 1. Crystallographic Data of Complexes 1, and 2

|  | 1  | 2                                     |
|--|--|---------------------------------------|
| chem formula                               | C <sub>24</sub> H <sub>15</sub> O <sub>6</sub> MnFeTe <sub>3</sub> | C <sub>29</sub> H <sub>25</sub> PTeNi |
| fw   | 892.95   | 590.79                                |
| cryst syst                                 | monoclinic   | triclinic                             |
| space group                                | $P 2_1/n$  | P 1                                   |
| $\lambda$ , $\mathring{A}(Mo\ K_{\alpha})$ | 0.7107   | 0.7107                                |
| a, Å                                       | 8.961(1)   | 9.744(2)                              |
| b, Å                                       | 17.496(3)  | 11.466(3)                             |
| c, Å                                       | 17.309(2)  | 11.805(1)                             |
| α, deg                                     |  | 80.07(2)                              |
| β, deg                                     | 97.86(1)   | 73.66(1)                              |
| γ, deg                                     |  | 82.64(2)                              |
| V, Å <sup>3</sup>                          | 2688.2(7)  | 1242.3(4)                             |
| Z  | 4  | 2                                     |
| d <sub>calcd</sub> , g cm <sup>-3</sup>    | 2.206  | 1.579                                 |
| F(000)                                     | 1650   | 578                                   |
| μ, cm <sup>-1</sup>                        | 42.283   | 20.156                                |
| T, °C                                      | 23   | 23                                    |
| $R^{a}$                                    | 0.032  | 0.027                                 |
| $R_{\mathrm{w}}^{}\mathrm{b}}$             | 0.028  | 0.027                                 |
| GOF <sup>c</sup>                           | 1.23   | 2.42                                  |

 $<sup>^{</sup>a}\,R = \Sigma |(F_{o}\text{-}F_{c})|/\Sigma F_{o}.\ ^{b}R_{w} = \left[\Sigma \omega (F_{o}\text{-}F_{c})^{2}/\Sigma \omega {F_{o}}^{2}\right]^{1/2}.$ 

81.20(3)°), observed in complex **1**, is ascribed to the proximity of the bridging benzenetellurolate groups to the Fe(II) center.

The crystal structure of the neutral CpNi(TePh)(PPh<sub>3</sub>) was determined with X-ray diffraction (Fig. 2). The terminal Ni<sup>II</sup>-TePh bond length of 2.4804(6) Å observed in complex  ${\bf 2}$  is comparable to Ni-Te distance in CpNi(Te-C<sub>6</sub>H<sub>3</sub>Me<sub>3</sub>)-

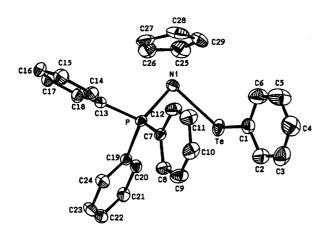


Fig. 2. ORTEP drawing and labeling scheme of the CpNi(TePh)(PPh<sub>3</sub>) with thermal ellipsoids drawn at the 30% probability level.

 $(P(C_2H_5)_3)$ .<sup>11</sup> Of particular significance, the Ni-C(27) bond of length 2.056(4) Å in complex **2** is significantly shorter than the average Ni-C bonds (average 2.115(4) Å); it is assumed that the steric effect of the Te atom on the metal-bound cyclopentadiene atoms causes significant bonding between Ni and C(27) atoms (the bond angle of Te-Ni-C(27) 163.92(2)°).

In these reactions the complex fac-[Fe(CO)<sub>3</sub>(TePh)<sub>3</sub>]<sup>-</sup> acts as a potential chelating metallo ligand", and the chelating property of the iron(II) tellurolate complex offers novel routes toward heterometallic tellurolate species. This investigation also indicated that the fac-[Fe(CO)<sub>3</sub>(SePh)<sub>3</sub>]<sup>-</sup> may serve as a better tridentate metallo ligand and chalcogenolate ligand-transfer reagent than fac-[Fe(CO)<sub>3</sub>(TePh)<sub>3</sub>]<sup>-</sup> in the syntheses of heterometallic chalcogenolate complexes.

#### EXPERIMENTAL SECTION

Manipulations, reactions, and transfers of samples were conducted under nitrogen according to standard Schlenk techniques or in a glovebox (argon gas). Solvents were distilled under nitrogen from appropriate drying agents (diethyl ether from CaH2; acetonitrile from CaH2-P2O5; hexane and tetrahydrofuran (THF) from Na-benzophenone) and stored in dried, N<sub>2</sub>-filled flasks over 4 Å molecular sieves. A nitrogen purge was used on these solvents before use and transfers to reaction vessels were via stainless-steel cannula under N2 at a positive pressure. The reagents manganese decacarbonyl, iron pentacarbonyl, diphenyl ditelluride, bis(triphenylphosphoranylidene)ammonium chloride (Aldrich) were used as received. Infrared spectra were recorded on (Bio-Rad FTS-185 and FTS-7 FTIR spectrometer) with sealed solution cells (0.1 mm) and KBr windows, NMR spectra on a Bruker AC 200 spectrometer, <sup>1</sup>H and <sup>13</sup>C chemical shifts being relative to tetramethylsilane and UV/VIS spectra on a GBC 918 spectrophotometer. Analyses of carbon, hydrogen were obtained with a CHN analyzer (Heraeus).

## Preparation of (CO)<sub>3</sub>Mn(μ-TePh)<sub>3</sub>Fe(CO)<sub>3</sub> (1)

A solution containing *fac*-[PPN][Fe(CO)<sub>3</sub>(TePh)<sub>3</sub>] (1 mmol, 1.29 g)<sup>6</sup> and [Mn(CO)<sub>3</sub>(CH<sub>3</sub>CN)<sub>3</sub>][PF<sub>6</sub>] (1 mmol, 0.407 g)<sup>7</sup> in THF (3 mL) was stirred under nitrogen at ambient temperature. After stirring of the reaction solution overnight, the orange purple solution accompanied by a gray precipitate, presumably [PPN][PF<sub>6</sub>], was formed. Diethyl ether (5 mL) was added into the orange purple solution and then the solution was filtered to remove [PPN][PF<sub>6</sub>]. The filtrate was dried under vacuum and then diethyl ether (5 mL)-hexane (3 mL) were added to extract the brown purple solid. The mixture solution was then stored still at room temperature for two days to

 $<sup>^</sup>c$  GOF =  $\left[\Sigma[\omega(F_o\text{-}F_c)^2/(M\text{-}N)]^{1/2}$  where M = number of reflections and N = number of parameters.

Table 2. Selected Bond Distances (Å) and Angles (deg) for (a) 1, (b) 2

| (a) <b>1</b>      |            |                   |            |
|-------------------|------------|-------------------|------------|
| Fe-Te(1)          | 2.628(1)   | Mn-Te(1)          | 2.638(1)   |
| Fe-Te(2)          | 2.640(1)   | Mn-Te(2)          | 2.678(1)   |
| Fe-Te(3)          | 2.623(1)   | Mn-Te(3)          | 2.646(1)   |
| Fe-Te(1)-Mn       | 82.26(3)   | Te(1)-Fe- $Te(2)$ | 79.83(3)   |
| Fe-Te(2)-Mn       | 81.29(3)   | Te(1)-Fe- $Te(3)$ | 84.04(3)   |
| Fe-Te(3)-Mn       | 82.21(3)   | Te(1)-Fe-C(1)     | 93.6(3)    |
| Te(1)-Mn-Te(2)    | 78.97(3)   | Te(1)-Fe-C(2)     | 89.69(22)  |
| Te(1)-Mn- $Te(3)$ | 83.40(3)   | Te(1)-Fe-C(3)     | 168.0(3)   |
| Te(1)-Mn-C(4)     | 93.43(24)  | Te(1)-Mn-C(5)     | 93.22(23)  |
| Te(1)-Mn-C(6)     | 172.99(22) |                   |            |
| (b) <b>2</b>      |            |                   |            |
| Ni-Te             | 2.4804(6)  | Ni-C(25)          | 2.117(4)   |
| Ni-C(26)          | 2.113(4)   | Ni-C(27)          | 2.056(4)   |
| Ni-C(28)          | 2.124(4)   | Ni-C(29)          | 2.104(4)   |
| Te-Ni-P           | 92.69(3)   | Te-Ni-C(25)       | 104.45(13) |
| Te-Ni-C(26)       | 135.98(15) | Te-Ni-C(27)       | 163.92(12) |
| Te-Ni-C(28)       | 127.41(15) | Te-Ni-C(29)       | 99.65(13)  |
|                   |            |                   |            |

precipitate the pure brown purple solid  $(CO)_3Mn(\mu-TePh)_3Fe(CO)_3$ . The yield was 0.717~g~(80%). Diffusion of hexane into  $(CO)_3Mn(\mu-TePh)_3Fe(CO)_3$  diethyl ether solution at -15 °C for 4 weeks led to formation of orange purple crystals of complex 1 suitable for X-ray crystallography. IR (THF):  $\nu(CO)$  2063 m, 2003 vs, 1923 m, br cm<sup>-1</sup>. <sup>1</sup>H NMR (CD<sub>2</sub>Cl<sub>2</sub>):  $\delta$  7.24-7.88 (m) ppm (Ph). <sup>13</sup>C NMR (CD<sub>2</sub>Cl<sub>2</sub>):  $\delta$  204.93 ppm (Fe-CO), 138.30 (s), 129.81 (s), 129.54 (s), 105.25 (s) ppm (Ph). Absorption spectrum (THF) [ $\lambda_{max}$ , nm ( $\epsilon$ , M<sup>-1</sup> cm<sup>-1</sup>)]: 385 (3245), 534 (890). Anal. Calcd for  $C_{24}H_{15}O_6Te_3FeMn$ : C, 32.28; H, 1.69. Found: C, 32.18; H, 1.75.

#### Preparation of CpNi(TePh)(PPh<sub>3</sub>) (2)

A solution of [Na][TePh] (obtained from reaction of [Na][BH<sub>4</sub>] (1 mmol, 0.038 g) and (TePh)<sub>2</sub> (1 mmol, 0.410 g) in MeOH) in 15 mL of MeOH was added to CpNi(I)(PPh<sub>3</sub>) (1 mmol, 0.513 g) under nitrogen at ambient temperature. After stirring of the reaction solution for 1 h, the brown solution accompanied by a red brown precipitate was formed. The solution was removed under positive N<sub>2</sub> and then the red brown solid was washed with MeOH twice. The yield of complex **2** was 0.438 g (74%). Diffusion of hexane into CpNi(TePh)(PPh<sub>3</sub>) THF solution at -15 °C for 5 weeks led to formation of crystals of complex **2** suitable for X-ray crystallography. <sup>1</sup>H NMR (C<sub>4</sub>D<sub>8</sub>O):  $\delta$  4.99 (s) ppm (Cp). <sup>13</sup>C NMR (C<sub>4</sub>D<sub>8</sub>O):  $\delta$  93.96 (s) ppm (Cp). Anal. Calcd for C<sub>29</sub>H<sub>25</sub>PTeNi: C, 58.95; H, 4.27. Found: C, 59.27; H, 4.49.

## Reaction of fac-[Fe(CO)<sub>3</sub>(TePh)<sub>3</sub>] and CpNi(I)(PPh<sub>3</sub>)

A solution of fac-[Fe(CO)<sub>3</sub>(TePh)<sub>3</sub>]<sup>-</sup> (0.4 mmol, 0.516 g) in THF (5 mL) was stirred with CpNi(I)(PPh<sub>3</sub>) (0.4 mmol, 0.206 g) overnight at ambient temperature, resulting in the formation of a red brown solution and the solvent-insoluble gray solid [PPN][I], and an unidentified solid. The red brown solution was transferred into another flask and dried under vacuum. Hexane was added to extract the neutral hexane-soluble product identified as the known Fe<sub>2</sub>(CO)<sub>6</sub>( $\mu$ -TePh)<sub>2</sub> by IR (IR  $\nu$ <sub>CO</sub> (hexane) 2056 m, 2021 vs, 1980 m, 1962 sh). The hexane-insoluble red brown solid was washed twice with hexane and identified as the neutral CpNi(TePh)(PPh<sub>3</sub>) (yield 11%).

# Crystallography

Crystallographic data for complexes 1, and 2 are collected in Table 1 and in the Supporting Information. All crystals were chunky: 1, brown, ca.  $0.30 \times 0.30 \times 0.50$  mm; 2, dark brown,  $0.55 \times 0.50 \times 0.40$  mm. Each was mounted on a glass fiber and quickly coated in epoxy resin. The unit-cell parameters were obtained by the least-square refinement from 25 reflections with  $2\theta$ between 18.82 and 28.14° for 1 and between 18.90° and 29.22° for 2. Diffraction measurements for complexes 1 and 2 were carried out at 23 °C on a Nonius CAD 4 diffractometer with graphite-monochromated Mo  $K_{\alpha}$  radiation ( $\lambda$  0.7107 Å) employing the  $\theta$ -2 $\theta$  scan mode. <sup>12</sup> A  $\psi$ -scan absorption correction was made. In the structure determination, the positions of Fe and Mn atoms are determined on the basis of the synthetic route, not from the X-ray diffraction data for complex 1. The NRCC-SDP-VAX package of programs was employed and atomic scattering factors were from ref. 13.

#### ACKNOWLEDGMENT

The support of the National Science Council (Taiwan) is gratefully acknowledged.

Received September 14, 1999.

## **Key Words**

Tellurolate; Heterodimetallic tellurolate.

# **Supporting Information Available**

Tables of crystal data and experimental conditions for the X-ray studies, atomic coordinates and  $B_{eq}$  values, bond lengths and angles for  $(CO)_3Mn(\mu\text{-TePh})_3Fe(CO)_3$ , and



CpNi(TePh)(PPh<sub>3</sub>) (11 pages). Ordering information is on any current masthead page.

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