Synthesis and spectroscopic characterization of the complex 
Bu₂Sn[Fe(η²-C₅H₅)(CO)₂] and (PPh₃)₂Pd[Fe(η²-C₅H₅)(CO)₂] 

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Na[Fe(η²-C₅H₅)(CO)₂] reacts with both Bu₂SnCl₂ and (PPh₃)_2PdCl₂ in 2:1 molar ratio in THF at room temperature to give the metal-metal bonded complexes Bu₂Sn[Fe(η²-C₅H₅)(CO)₂] and (PPh₃)_2Pd[Fe(η²-C₅H₅)(CO)₂] respectively. The complexes have been characterized by IR, PMR, mass spectra and elemental analysis.

The chemistry of compounds containing a direct bond between two metals, although relatively new, has now become an exciting field of research because of the novel structures, bonding and physical and chemical properties of such compounds. Of the various methods for the formation of metal-metal bond, the reaction of carbonyl anion on another metal halide seems to be the most widely used one. The reagent Na[Fe(η²-C₅H₅)(CO)₂] is one such and has been used to make Fe-M bonds with most of the metals, transition or non-transition. We too have reported several metal-iron bonded complexes prepared through the use of this reagent. In the present note, we report two more new complexes.

**Experimental**

The reactions were carried out under dry nitrogen atmosphere. THF was dried by refluxing over Na wire and freshly distilled prior to use. Bu₂SnCl₂ and [Fe(η²-C₅H₅)(CO)₂] were Aldrich reagent grade chemicals. (PPh₃)_2PdCl₂ was prepared by shaking an ethanolic solution of Na₂PdCl₄ with PPh₃. Solution of Na[Fe(η²-C₅H₅)(CO)₂] were prepared by stirring a THF solution of [Fe(η²-C₅H₅)(CO)₂] with an excess of sodium amalgam. The melting points are uncorrected. The IR spectra were recorded on a Shimadzu FT IR instrument in cyclohexane solution, PMR spectra in CDCl₃ on a Bruker AM-300 spectrometer and mass spectra on a VG ZAB F-1 instrument.

**Notes**

Preparation of Bu₂Sn[Fe(η²-C₅H₅)(CO)₂]

A solution of Bu₂SnCl₂ (0.858 g, 2.8 mmol) in THF (20 cm³) was slowly added to a solution of Na[Fe(η²-C₅H₅)(CO)₂] (5.6 mmol) in THF (40 cm³) and the mixture stirred at room temperature for 24 h. The colour of the solution turned from orange to reddish brown by this time. The solvent was removed in vacuo to get a reddish brown solid which was extracted with pet. ether (b.p. 40-60°C). The removal of solvent and subsequent recrystallization of the solid from n-hexane gave Bu₂Sn[Fe(η²-C₅H₅)(CO)₂] (I) as reddish brown needles (1.2 g, 73%), m.p., 85°C [Found: C, 44.72; H, 4.83; C₂₂H₂₂O₄Fe₂Sn requires: C, 44.99; H, 4.77%]. The complex (PPh₃)_2Pd[Fe(η²-C₅H₅)(CO)₂] (II), another reddish brown crystalline solid, m.p. 128°C [Found: C, 59.36; H, 4.12; C₉₀H₄₀O₄P₂Fe₂Pd requires: C, 60.95; H, 4.06%], was prepared in 62% yield by a similar method using (PPh₃)_2PdCl₂ instead of Bu₂SnCl₂.

**Results and discussion**

The reaction between Bu₂SnCl₂ and Na[Fe(η²-C₅H₅)(CO)₂] in 1:2 molar ratio gave the complex Bu₂Sn[Fe(η²-C₅H₅)(CO)₂] (I) whose structure has been suggested on the basis of its IR, PMR, mass spectra. The IR spectrum showed two "CO band at 1975(s) and 1931(s) cm⁻¹ which are characteristic of terminal CO groups. The PMR spectrum [CDCl₃, δ 4.80 (s, C₅H₅), 1.53 (m, CH₂), 1.36 (m, CH₂), 1.34 (m, CH₂), 0.91 (t, CH₃)] showed a sharp resonance for the cyclopentadienyl protons and four multiplets for the butyl protons in the correct intensity ratio. The mass spectrum clearly showed the molecular ion peak at m/z 587. Other characteristic peaks for the different decomposition fragments of I were observed at m/z 531 [M⁺ - 2CO], 475 [M⁺ - 4CO], 418 [M⁺ - 4CO-Bu], 361 [M⁺ - 4CO-2Bu], 410 [Bu₂SnFe(η²-C₅H₅)(CO)₂]⁺, 354 [Bu₂SnFe(η²-C₅H₅)(CO)₂]⁺, 297 [Bu₂SnFe(η²-C₅H₅)⁺], 240 [SnFe(η²-C₅H₅)⁺], 121 [Fe(η²-C₅H₅)⁺] and 56 [Fe⁺]. Thus the spectral data of complex (I) are consistent with the proposed structure.

Treatment of (PPh₃)_2PdCl₂ with Na[Fe(η²-C₅H₅)(CO)₂] afforded the complex (PPh₃)_2Pd[Fe(η²-C₅H₅)(CO)₂] (II) whose structure is proposed from
spectroscopic results. The IR spectrum \( \nu(\text{CO}) : 1996\text{s} \) and 1956\text{s} \( \text{cm}^{-1} \) indicated the presence of terminal CO groups and the PMR spectrum \( \text{CDCl}_3, \delta 4.78 \text{ (s, C}_2\text{H}_5\), 7.42-7.80 \text{ (m, PPh}_3\) showed the presence of both cyclopentadienyl and triphenylphosphine protons in the proper intensity ratio. The mass spectrum did not show the molecular ion but showed a peak at \( m/z \) 956 \( [\text{M}^+ - \text{CO}] \) due to the loss of one carbonyl group. Other peaks for successive loss of CO groups were also observed: \( m/z \) 900 \( [\text{M}^+ - 3\text{CO}] \), 872 \( [\text{M}^+ - 4\text{CO}] \). Mass peaks were also observed for fragments after successive losses of phenyl groups from the molecular ion: 907 \( [\text{M}^+ - \text{Ph}] \), 830 \( [\text{M}^+ - 2\text{Ph}] \), 753 \( [\text{M}^+ - 3\text{Ph}] \), 676 \( [\text{M}^+ - 4\text{Ph}] \). The other important mass peaks were at \( m/z \) 808 \( [\text{PPh}_3\text{PdFe}(\eta^5\text{-C}_5\text{H}_5)(\text{CO})_2]^+ \), 780 \( [\text{PPh}_3\text{PdFe}(\eta^5\text{-C}_5\text{H}_5)(\text{CO})]^+ \), 752 \( [\text{PPh}_3\text{PdFe}(\eta^5\text{-C}_5\text{H}_5)(\text{CO})]^+ \), 631 \( [\text{PPh}_3\text{Pd}]^+ \), 611 \( [\text{M}^+ - 4\text{Ph}\text{-C}_5\text{H}_5] \), 555 \( [\text{M}^+ - 4\text{Ph}\text{-C}_5\text{H}_5\text{-2CO}] \), 177 \( [\text{Fe}(\eta^5\text{-C}_5\text{H}_5)(\text{CO})_2]^+ \), 121 \( [\text{Fe}(\eta^5\text{-C}_5\text{H}_5)]^+ \) and 56 \( [\text{Fe}]^+ \). The expected isotopic pattern was clearly observed in many of the peak containing palladium. Thus all the spectral evidences established the proposed structure of complex (II).

Both complexes I and II are soluble in most of the common organic solvents including pet. ether. They are stable for a long time when stored under nitrogen.

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References