



## NOTE

### Synthesis and Characterization of Dicobalt-Iron Complex $(\mu_3\text{-S})\text{FeCo}_2(\text{CO})_8(\text{Ph}_2\text{PMe})$

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Treatment of the dicobalt-iron complex  $(\mu_3\text{-S})\text{FeCo}_2(\text{CO})_9$  (**1**) with a monophosphine ligand  $\text{Ph}_2\text{PMe}$  in  $\text{CH}_2\text{Cl}_2$  produced the dicobalt-iron complex  $(\mu_3\text{-S})\text{FeCo}_2(\text{CO})_8(\text{Ph}_2\text{PMe})$  (**2**) in 41 % yield. The new complex **2** was structurally characterized by IR,  $^1\text{H}$  NMR,  $^{31}\text{P}\{^1\text{H}\}$  NMR and  $^{13}\text{C}\{^1\text{H}\}$  NMR spectroscopy.

**Keywords:** Dicobalt-iron complex, Monophosphine, Synthesis, Carbonyl substitution.

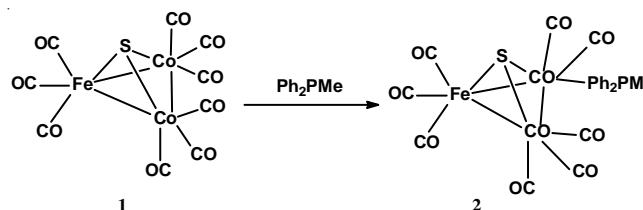
Transition metal carbonyl complexes have attracted much attention in fields of organometallic and inorganic chemistry because of their widely applications in most catalytic reactions<sup>1-7</sup>. The  $\mu_3\text{-S}$  dicobalt-iron complex  $(\mu_3\text{-S})\text{FeCo}_2(\text{CO})_9$  was prepared many years ago<sup>8</sup>. Carbonyl substitution reactions of  $(\mu_3\text{-S})\text{FeCo}_2(\text{CO})_9$  with monophosphine or diphosphine ligands gave the corresponding dicobalt-iron derivatives<sup>9</sup>. We recently investigate the reaction of the parent complex **1** with  $\text{Ph}_2\text{PMe}$  and have successfully synthesized a new dicobalt-iron complex with the monophosphine ligand  $\text{Ph}_2\text{PMe}$ .

Reaction and operation was carried out under a dry, oxygen free nitrogen atmosphere with standard Schlenk and vacuum line techniques. Complex **1**<sup>8</sup> was prepared according to the literature procedures.  $\text{Ph}_2\text{PMe}$  was available commercially and used as received. IR spectra were recorded on a Nicolet 670 FTIR spectrometer.  $^1\text{H}$  ( $^{31}\text{P}\{^1\text{H}\}$ ,  $^{13}\text{C}\{^1\text{H}\}$ ) NMR spectra were obtained on a Bruker Avance 500 MHz spectrometer.

**Synthesis:** A solution of  $(\mu_3\text{-S})\text{FeCo}_2(\text{CO})_9$  (**1**) (0.092 g, 0.2 mmol) and  $\text{Ph}_2\text{PMe}$  (0.040 g, 0.2 mmol) in  $\text{CH}_2\text{Cl}_2$  (10 mL) was stirred at room temperature overnight. The solvent was reduced *in vacuo* and the residue was subjected to TLC separation using  $\text{CH}_2\text{Cl}_2$ /petroleum ether (*v/v* = 1:5) as eluent. From the main brown band afforded 0.052 g (41 %) of **2** as a black solid. IR (KBr, disk,  $\text{cm}^{-1}$ ):  $\nu_{\text{C=O}}$  2073 (vs), 2035 (vs), 2015 (vs), 2004 (vs), 1993 (vs), 1978 (vs), 1943 (vs).  $^1\text{H}$  NMR (500 MHz,  $\text{CDCl}_3$ ): 7.58-7.45 (m, 10H, PhH), 2.02 (d, *J* = 9.5 Hz, 3H,  $\text{CH}_3$ ) ppm.  $^{31}\text{P}\{^1\text{H}\}$  NMR (200 MHz,  $\text{CDCl}_3$ , 85 %  $\text{H}_3\text{PO}_4$ ): 31.19 (s) ppm.  $^{13}\text{C}\{^1\text{H}\}$  NMR (125 MHz,  $\text{CDCl}_3$ ): 206.05 (C=O), 136.19 (d, *J*<sub>P-C</sub> = 43.4 Hz, *i*-PhC), 135.76 (d, *J*<sub>P-C</sub> = 43.2 Hz, *i*-PhC), 131.58 (t, *J*<sub>P-C</sub> = 10.4 Hz, *m*-PhC), 131.17 (t, *J*<sub>P-C</sub> = 10.1 Hz, *m*-PhC), 130.72, 130.59 (2s, *p*-PhC),

128.78 (t, *J*<sub>P-C</sub> = 11.0 Hz, *o*-PhC), 20.04 (d, *J*<sub>P-C</sub> = 27.9 Hz,  $\text{CH}_3$ ) ppm.

As shown in **Scheme-I**, treatment of the dicobalt-iron complex **1** with 1 equivalent of  $\text{Ph}_2\text{PMe}$  in  $\text{CH}_2\text{Cl}_2$  at room temperature afforded the dicobalt-iron complex **2** in 41 % yield. The new complex **2** was air-stable black solids, which has been characterized by IR,  $^1\text{H}$  NMR,  $^{31}\text{P}\{^1\text{H}\}$  NMR and  $^{13}\text{C}\{^1\text{H}\}$  NMR spectroscopy.



**Scheme-I:** Synthesis of the title complex **2**

**Infrared spectrum:** As shown in Fig. 1, the IR spectrum of complex **2** showed seven strong absorption bands in the range of 2073-1943  $\text{cm}^{-1}$  for the terminal carbonyls and the values are shifted towards lower frequencies relative to those of complex **1** (2106, 2067, 2054, 2041, 2029 and 1973  $\text{cm}^{-1}$ )<sup>10</sup> because  $\text{Ph}_2\text{PMe}$  is stronger electron-donating than CO.

**$^1\text{H}$  NMR spectrum:** As shown in Fig. 2, the  $^1\text{H}$  NMR spectrum of the title complex **2** displayed a multiplet at  $\delta$  7.58-7.45 ppm for the phenyl protons and a doublet at  $\delta$  2.02 ppm with coupling constant of 9.5 Hz for the methyl protons.

**$^{31}\text{P}\{^1\text{H}\}$  NMR spectrum:** As shown in Fig. 3, the  $^{31}\text{P}\{^1\text{H}\}$  NMR spectrum of the title complex **2** exhibited a singlet at  $\delta$  31.19 ppm for the phosphorus atom of  $\text{Ph}_2\text{PMe}$  coordinated to the cobalt atom.

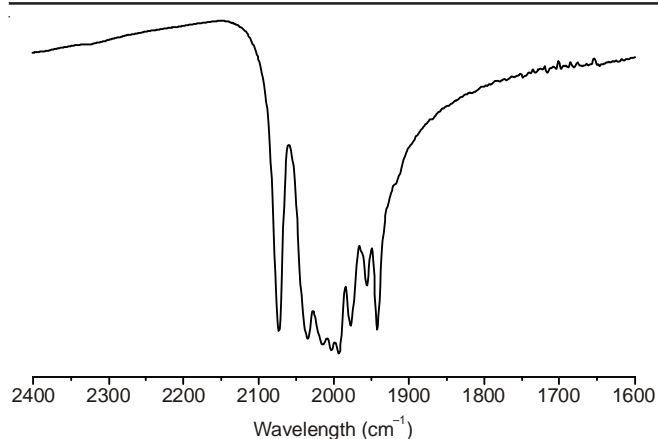
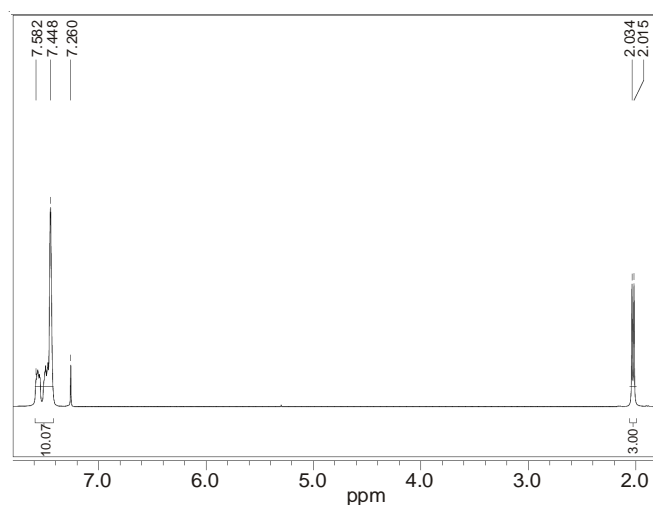
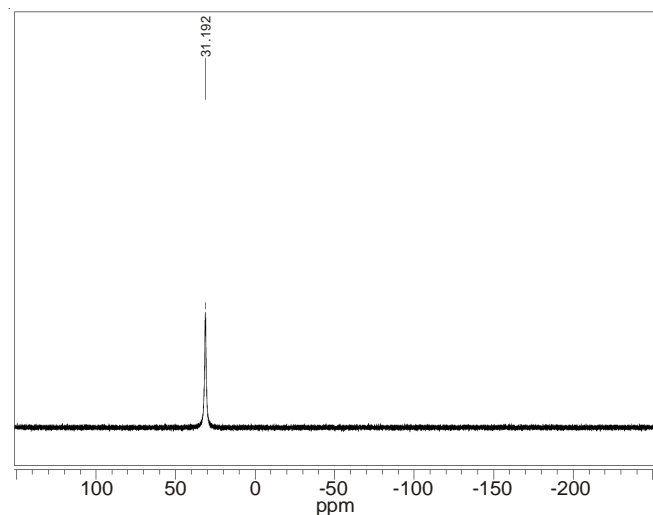
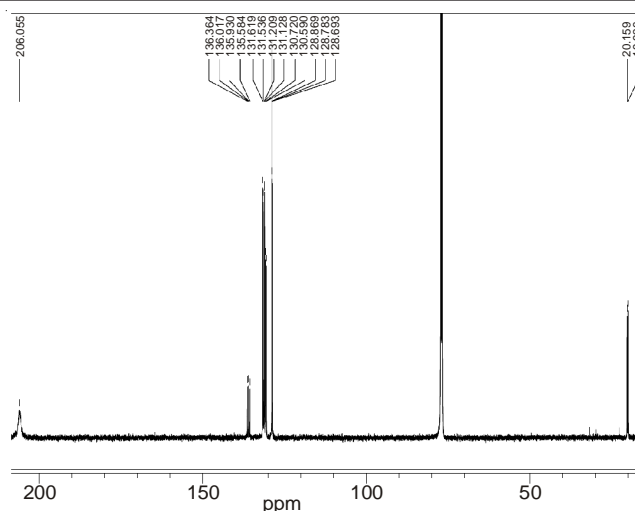


Fig. 1. IR spectrum of the title complex 2

Fig. 2. <sup>1</sup>H NMR spectrum of the title complex 2Fig. 3. <sup>31</sup>P{<sup>1</sup>H} NMR spectrum of the title complex 2

**<sup>13</sup>C{<sup>1</sup>H} NMR spectrum:** As shown in Fig. 4, the <sup>13</sup>C{<sup>1</sup>H} NMR spectrum of the title complex 2 demonstrated a singlet at  $\delta$  206.05 ppm for the terminal carbonyls, two doublets at  $\delta$  136.19 and 135.76 ppm for the ipso-phenyl carbons, two

Fig. 4. <sup>13</sup>C{<sup>1</sup>H} NMR spectrum of the title complex 2

triplets at  $\delta$  131.58 and 131.17 ppm for the *meta*-phenyl carbons, two singlets at  $\delta$  130.72 and 130.59 ppm for the *para*-phenyl carbons, a triplet at  $\delta$  128.78 ppm for the *ortho*-phenyl carbons and a doublet at  $\delta$  20.04 ppm for the methyl carbon.

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