

## $\eta^3:\eta^1 \rightleftharpoons \eta^2$ -TAUTOMERISM IN CARBONYLIRON COMPLEXES. INFLUENCE OF SUBSTITUENTS

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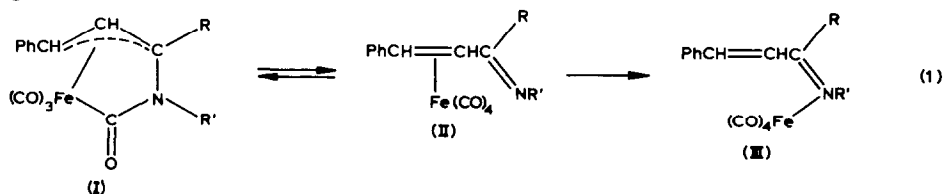
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### Summary

A number of chelate  $\eta^3$ -allylcarbonyl iron complexes (I) with different substituents (R in the allyl ligand and R' at the nitrogen atom) were synthesized. The influence of structural features on the equilibrium between the complexes I and their  $\eta^2$ -azadiene tautomers (II) was studied by IR spectroscopy. It was established that the equilibrium position is determined in the first place by steric factors. When R is a bulky substituent the equilibrium is shifted towards the cyclic form I, whereas the branching of the alkyl substituent R' at the nitrogen atom favours the open olefinic form II. Furthermore  $\pi$ - $\sigma$ -(N) rearrangement of complexes II to  $\sigma$ -(N) derivatives (III), and the conversion of III into  $\eta^4$ -azadiene complexes (IV) also depend on the steric requirements of the substituents R and R'.

### Introduction

Tricarbonyl- $\sigma$ -[N-(1-R-3-phenyl-1-3- $\eta$ -allyl)-NR'-carbonyl] iron complexes (I) synthesized by the reaction of  $\text{BF}_3$  adducts of  $((\eta^2\text{-RCOCH=CHPh})\text{Fe}(\text{CO})_4)$  complexes with primary amines ( $\text{R}'\text{NH}_2$ ) [1,2] exhibit a chelate  $\pi$ -allylic structure in the solid state, as defined by X-ray analysis [1,3]. In solution, they are reversibly isomerized to open  $\pi$ -olefinic tetracarbonyl(1-R'-2-R-4-phenyl-3,4- $\eta$ -1-azadiene)iron complexes (II) [2]. In a previous paper, the thermodynamic characteristics of such an equilibrium isomerization were determined, and it was concluded that the interconversion of  $\text{I} \rightleftharpoons \text{II}$  is related to a new case of ring-chain valent tautomerism in organometallic compounds [4]. Besides, complex II is capable of being rearranged in solution to an isomeric tetracarbonyl(1-R'-2-R-4-phenyl-1- $\eta^1$ -1-azadiene)iron compound (III) having a  $\sigma$ -bond N-Fe [5].



As was briefly noted [6], the capability of I to isomerize into II depends on the nature of the substituent R in the allyl ligand and R' at the nitrogen atom. The present paper reports the results of an investigation of the influence of these substituents on the position of tautomeric equilibrium, as well as the specific features of the structure of complex II required for  $\pi$ - $\sigma$ -(N) rearrangement into complex III.

## Results and discussion

Complexes I, with different R and R' substituents, were synthesized by the method described earlier [1,2] (see Experimental); their IR spectra were studied in the  $\nu(\text{CO})$  range (Table 1). The concentrations of the chelate and the open forms I and II were determined from the integrated intensities of the corresponding absorption bands, and the tautomeric equilibrium constants,  $K_T$ , were calculated (Table 2). The measurements and calculation techniques have been given in a previous paper [4].

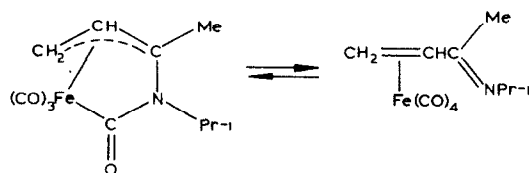
TABLE 1  
IR SPECTRAL DATA FOR THE EQUILIBRIUM

(I)  $\rightleftharpoons$  (II)

Nr.	R	R'	$\nu(\text{C}\equiv\text{O}), \text{cm}^{-1} (\text{hexane})^a$	
			I	II
1.	H	t-Bu	2070 <sup>b</sup>	2087,2018,2008,1987
2.	Me	Me	2068,2009,1991	2085,2017,1980 <sup>b</sup>
3.	Me	Et	2065,2010,1992	2085,2021,1982 <sup>b</sup>
4.	Me	t-Bu	2065,2011,1995	2087,2020,1985 <sup>b</sup>
5.	Me	c-C <sub>6</sub> H <sub>11</sub>	2068,1992 <sup>b</sup>	2085,2017,2008,1980
6.	Me	i-Pr	2068,1993 <sup>b</sup>	2085,2017,2008,1980
7.	Et	Me	2065,2010,1994	2085 <sup>b</sup>
8.	Et	Et	2065,2010,1994	2085 <sup>b</sup>
9.	Et	c-C <sub>6</sub> H <sub>12</sub>	2068,2011,1994	2087,2021,1985 <sup>b</sup>
10.	n-Bu	Me	2065,2006,1993	2087 <sup>b</sup>
11.	n-Bu	c-C <sub>6</sub> H <sub>11</sub>	2065,2006,1992	2086,2016,1985 <sup>b</sup>
12.	Ph	H	2065,2009,1994	2088,2023,2015,1986
13.		c	2071 <sup>b</sup>	2088,2018,2011,1985

<sup>a</sup> Spectra Nos. 1–4, 7, 8 and 10–12 also reveal the bands of complexes III and IV. The assignments were made according to data of ref. 2. <sup>b</sup> The remaining bands overlapped with bands of another tautomer.

<sup>c</sup> For the equilibrium



From Table 2, it can be seen that the position of tautomeric equilibrium is determined, first of all, by the steric requirements of the substituents R and R', with the influence of R at C(1) in the allyl group being particularly pronounced.

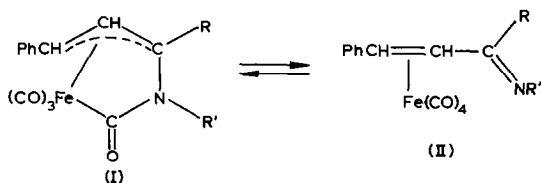
Steric models of both tautomeric forms were constructed in order to explain this effect. Examination of the models shows that two kinds of steric interactions involving R and R' may occur in complexes I and II.

The first is associated with the mutual repulsion of the substituents, which is greater in the open olefinic form II and decreases in passing over to the cyclic allylic form I. The second is connected with the interaction of the bulky R' substituents with the oxygen atom of the carbamoyl group which leads to destabilization of cyclic form I; in other words, the bulky substituents at the nitrogen atom hinder the attack of the imine group on the carbonyl ligand, i.e. cyclization.

In view of this, increasing the bulk of substituent R must lead to a shift in the tautomeric equilibrium towards the chelate allyl form. In fact, the tautomeric equilibrium constant,  $K_T$ , decreases 7 to 9 times when complexes with R = Me are replaced by complexes with R = Et. This is clearly seen when the following pairs are compared: (a)-(f), (b)-(g), (d)-(h) and (e)-(i) (Table 2). With further branching of the substituent R (e.g. R = i-Pr or t-Bu), the equilibrium shifts towards complex I to such an extent that only one isomer is observed in the IR spectrum.

The influence of R' at the carbamoyl nitrogen atom probably manifests itself as the sum of the two steric interactions mentioned above and acting in opposite directions, with the destabilizing effect of the bulky substituent R' in the allyl form apparently prevailing. This is consistent with the general lesser influence of R', compared with R, and with the observed shift in the equilibrium towards the open form (II) when the steric requirements of substituent R' are increased. Elongation of

TABLE 2  
EQUILIBRIUM CONSTANTS FOR THE EQUILIBRIUM



Com- pounds	R	R'	$K_T^a$	Com- pounds	R	R'	$K_T^a$
a	Me	Me	$0.81 \pm 0.03$	j	n-Bu	Me	$0.12 \pm 0.02$
b	Me	Et	$0.80 \pm 0.03$	k	n-Bu	c-C <sub>6</sub> H <sub>11</sub>	$0.63 \pm 0.02$
c	Me	n-Bu	$0.79 \pm 0.02$	l	i-Pr	Me	< 0.05
d	Me	i-Bu	$2.33 \pm 0.11$	m	t-Bu	Me	< 0.05
e	Me	c-C <sub>6</sub> H <sub>11</sub>	$2.38 \pm 0.10$	n	Ph	Me	< 0.05
f	Et	Me	$0.09 \pm 0.01$	o	Ph	i-Pr	< 0.05
g	Et	Et	$0.10 \pm 0.01$	p	Ph	c-C <sub>6</sub> H <sub>11</sub>	< 0.05
h	Et	i-Pr	$0.35 \pm 0.03$				
i	Et	c-C <sub>6</sub> H <sub>11</sub>	$0.36 \pm 0.03$				

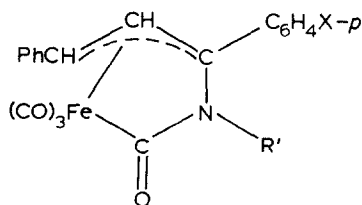
<sup>a</sup> The arithmetic mean value and the mean deviation for 6-9 measurements are given. When one cyclic form in solution is revealed by the IR spectra, it is assumed that  $K_T < 0.05$ .

the normal alkyl chain of R' hardly affects the position of equilibrium (complexes Ia, Ic or If, Ig); however, on changing to the branched R' substituents bonded to nitrogen via the second carbon atom, the tautomeric equilibrium constant increases 3 to 4 times. Such a shift towards the open olefinic form is established for three groups of complexes: a-e, f-i and j-k.

Destabilization of the ring structure with a branched alkyl substituent at the nitrogen atom, for example in the case of ring-chain isomerization of amides of acylcarboxylic acids, has been reported in the literature [7].

Although the electron-releasing properties in the sequence Me, Et, i-Pr and t-Bu increase slightly, we may assume that such a change does not affect the tautomeric equilibrium position, and the main influence is caused by increasing the bulk of the substituent in this sequence.

The advantage of the influence of steric factors is also witnessed by the results of an investigation of the chelate complexes I, where the substituents (R) are phenyl groups having electron-releasing and electron-withdrawing substituents in the *para*-position.



(I, X = Me, OMe, NMe<sub>2</sub>, Br,  
R' = Me, c-C<sub>6</sub>H<sub>11</sub>)

Irrespective of the electron effect of the aryl substituent, the IR spectra of all these complexes revealed, as in the case of the unsubstituted phenyl radical, bands of only one chelate allyl form.

The important role of the reciprocal repulsion of the substituents R and R' for relative destabilization of the open  $\pi$ -olefin form (II) is confirmed by a study of the tautomeric conversions of complexes in which R is H and in which, consequently, there is no such steric effect. Earlier it was shown that complexes prepared from ( $\eta^2$ -PhCH=CHCHO)Fe(CO)<sub>4</sub> (i.e. with R = H) and primary amines are  $\sigma$ -(N) derivatives (III) [5]. IR spectral investigation revealed that the reaction mixture also contains complexes I and II which further undergo  $\pi$ - $\sigma$ -(N) rearrangement into III (see reaction (1)). In this case, tautomeric equilibrium I  $\rightleftharpoons$  II is strongly shifted towards open form II. Unfortunately, it is impossible to make a quantitative estimate of the equilibrium position because of the successive fast  $\pi$ - $\sigma$ -(N) rearrangement in reactions with most of the primary amines, R'NH<sub>2</sub> (R' = Me, c-C<sub>6</sub>H<sub>11</sub>, Ph). Only in the case of an amine with a bulky t-butyl substituent were we able to isolate the chelate  $\pi$ -allyl complex (I, R = H, R' = t-Bu) as a light-yellow solid of low stability at room temperature. According to the IR spectrum, in hexane solution this complex isomerizes into olefinic form II by more than 90%. A considerable increase in the relative stability of the open form is, in this case, probable, because of the absence of repulsion between R and R' in the open form, and also the presence of the bulky substituent at the nitrogen atom which, as mentioned above, destabilizes the cyclic form.

Also of interest was an investigation of the behaviour of the complex in which the substituent at the nitrogen atom (R') was H. In this case, the steric effects of R and

TABLE 3

YIELDS, DECOMPOSITION POINTS AND ELEMENTAL ANALYSIS OF THE  $(\eta^2\text{-PhCH=CHCOR})\text{Fe}(\text{CO})_4$  COMPLEXES

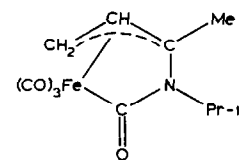
R	Yield (%)	Decomp. point (°C) <sup>a</sup>	(Found (calcd.)(%))			Molecular formula	IR (cm <sup>-1</sup> )	
			C	H	Fe		$\nu(\text{C=O})$ (heptane)	$\nu(\text{C=O})$ (KBr)
Et	62	50–51	54.61 (54.90)	3.57 (3.69)	16.71 (17.02)	C <sub>15</sub> H <sub>12</sub> FeO <sub>5</sub>	2095,2031 2020,1992	1670
n-Bu	65	64–65	57.33 (57.33)	4.43 (4.53)	15.70 (15.68)	C <sub>17</sub> H <sub>16</sub> FeO <sub>5</sub>	2093,2028 2015,1989	1660
i-Pr	53	82–83	55.96 (56.17)	4.15 (4.12)	16.83 (16.32)	C <sub>16</sub> H <sub>14</sub> FeO <sub>5</sub>	2097,2035 2024,1995	1660
t-Bu	42	51–52	56.82 (57.33)	4.71 (4.53)	15.74 (15.68)	C <sub>17</sub> H <sub>16</sub> FeO <sub>5</sub>	2098,2032 2021,1992	1670
C <sub>6</sub> H <sub>4</sub> Br	83	103–104	50.04 (50.15)	2.17 (2.43)	–	C <sub>19</sub> H <sub>11</sub> BrFeO <sub>5</sub>	2096,2032 2020,1995	1630
C <sub>6</sub> H <sub>4</sub> Me	67	104–105	62.32 (61.56)	3.71 (3.62)	13.77 (14.31)	C <sub>20</sub> H <sub>14</sub> FeO <sub>5</sub>	2095,2033 2018,1995	1640
C <sub>6</sub> H <sub>4</sub> OMe	68	90–91	59.20 (59.14)	3.40 (3.47)	–	C <sub>20</sub> H <sub>14</sub> FeO <sub>6</sub>	2097,2035 2024,1995	1640
C <sub>6</sub> H <sub>4</sub> N(Me) <sub>2</sub> <sup>b</sup>	53	105–106	60.54 (60.16)	4.19 (4.09)	–	C <sub>21</sub> H <sub>17</sub> FeNO <sub>5</sub>	2093,2028 2015,1989	1600

<sup>a</sup> Recrystallized from hexane. <sup>b</sup> Found (calculated): N, 3.26 (3.34)%.

TABLE 4

YIELDS, DECOMPOSITION POINTS AND ELEMENTAL ANALYSIS OF COMPLEXES I

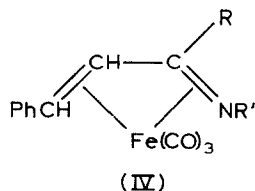
R	R'	Yield (%)	Decomp. pt. (°C)	(Found (calcd.) (%))				Molecular formula
				C	H	Fe	N	
H	t-Bu	50	—	57.36 (57.49)	4.82 (4.82)	15.41 (15.73)	—	C <sub>17</sub> H <sub>17</sub> FeNO <sub>4</sub>
Me	Et	43	76–77	56.35 (56.33)	4.63 (4.43)	16.42 (16.37)	4.06 (4.10)	C <sub>13</sub> H <sub>15</sub> FeNO <sub>4</sub>
Me	n-Bu	43	74–75	58.81 (58.56)	5.45 (5.18)	15.15 (15.13)	—	C <sub>18</sub> H <sub>19</sub> FeNO <sub>4</sub>
Et	Me	47	82–83	56.30 (56.33)	4.42 (4.43)	16.34 (16.37)	4.70 (4.10)	C <sub>13</sub> H <sub>15</sub> FeNO <sub>4</sub>
Et	Et	45	85–86	57.53 (57.49)	4.84 (4.82)	15.74 (15.73)	3.87 (3.94)	C <sub>17</sub> H <sub>17</sub> FeNO <sub>4</sub>
Et	i-Pr	38	81–82	58.51 (58.56)	5.03 (5.19)	15.17 (15.13)	3.65 (3.79)	C <sub>18</sub> H <sub>19</sub> FeNO <sub>4</sub>
Et	c-C <sub>6</sub> H <sub>11</sub>	44	92–93	61.55 (61.66)	5.71 (5.62)	13.60 (13.66)	3.65 (3.42)	C <sub>21</sub> H <sub>23</sub> FeNO <sub>4</sub>
n-Bu	Me	49	79–80	58.76 (58.76)	5.29 (5.19)	15.62 (15.13)	3.79 (3.79)	C <sub>18</sub> H <sub>19</sub> FeNO <sub>4</sub>
n-Bu	c-C <sub>6</sub> H <sub>11</sub>	45	82–83	63.16 (63.17)	6.24 (6.22)	13.29 (12.77)	3.16 (3.20)	C <sub>23</sub> H <sub>27</sub> FeNO <sub>4</sub>
Ph	H	47	93–94	60.43 (60.83)	3.45 (3.49)	15.10 (14.89)	3.70 (3.73)	C <sub>19</sub> H <sub>13</sub> FeNO <sub>4</sub>

Ph	i-Pr	43	–	63.25 (63.33)	4.40 (4.59)	–	–	C <sub>22</sub> H <sub>19</sub> FeNO <sub>4</sub>
Ph	C <sub>6</sub> H <sub>13</sub>	38	95–96	65.12 (65.52)	5.70 (5.46)	11.62 (12.10)	–	C <sub>25</sub> H <sub>25</sub> FeNO <sub>4</sub>
i-Pr	Me	53	103–104	57.50 (57.45)	4.86 (4.82)	15.62 (15.68)	4.15 (3.95)	C <sub>17</sub> H <sub>17</sub> FeNO <sub>4</sub>
t-Bu	Me	50	127–129	58.67 (58.55)	5.01 (5.19)	–	4.10 (3.79)	C <sub>18</sub> H <sub>19</sub> FeNO <sub>4</sub>
<i>p</i> -C <sub>6</sub> H <sub>4</sub> Me	Me	85	121–122	62.29 (62.55)	4.29 (4.25)	14.58 (13.85)		C <sub>21</sub> H <sub>17</sub> FeNO <sub>4</sub>
<i>p</i> -C <sub>6</sub> H <sub>4</sub> OMe	<i>c</i> -C <sub>6</sub> H <sub>11</sub>	50	106–107	63.82 (64.08)	5.25 (5.17)	11.53 (11.46)		C <sub>26</sub> H <sub>25</sub> FeNO <sub>4</sub>
<i>p</i> -C <sub>6</sub> H <sub>4</sub> N(Me) <sub>2</sub>	Me	52	100	61.26 (61.13)	4.80 (4.66)	12.66 (12.92)	6.36 (6.48)	C <sub>22</sub> H <sub>20</sub> FeN <sub>2</sub> O <sub>4</sub>
<i>p</i> -C <sub>6</sub> H <sub>4</sub> Br	Me	61	119–120	50.84 (51.32)	2.93 (3.01)	11.63 (11.93)	2.85 (2.99)	C <sub>20</sub> H <sub>14</sub> BrFeNO <sub>4</sub>
		49	–	46.94 (47.34)	4.66 (4.70)	20.15 (20.01)		C <sub>11</sub> H <sub>13</sub> FeNO <sub>4</sub>

$R'$  were minimal. The complexes (I,  $R = \text{Ph}$ ,  $t\text{-Bu}$  and  $R' = \text{H}$ ) were prepared by the reaction of  $(\eta^2\text{-PhCH=CHCOR})\text{Fe}(\text{CO})_4$  with  $\text{BF}_3$  and ammonia. They are less stable, especially in solutions, than complexes I having an alkyl-substituted nitrogen atom. Complex I, ( $R = \text{Ph}$ ,  $R' = \text{H}$ ) was isolated in an analytically pure form. In hexane solution \* it reversibly isomerized into the open  $\pi$ -olefinic form (II), the tautomeric equilibrium being notably shifted towards the open isomer, according to IR spectral data. Approximately equal amounts of both tautomeric forms were detected in the solution of the complex with  $R = t\text{-Bu}$  and  $R' = \text{H}$ .

These results should be compared with the data obtained for the corresponding complexes I with alkyl substituents at the nitrogen atom. As already discussed above, these complexes do not isomerize in solution into the open  $\pi$ -olefinic form (II) sufficiently to warrant IR investigation of such an equilibrium (Table 2, complexes m-p).

Steric factors also play an important role in the formation of  $\sigma$ -( $N$ ) derivatives (III) and influence their stability. These complexes were isolated when  $R = \text{H}$ , or when  $R$  and  $R'$  were small, for example when  $R = \text{Me}$  and  $R' = \text{Me}$ ,  $\text{Et}$  [5]. In other cases when  $R = \text{Et}$ ,  $n\text{-Bu}$  and  $R' = \text{Me}$ ,  $\text{Et}$ ,  $n\text{-Bu}$ , or when  $R = \text{Ph}$ ,  $t\text{-Bu}$  and  $R' = \text{H}$ , the formation of complexes III was only observed spectrally. We may suppose that the difficulty in formation and the instability of complexes III having bulky  $R$  and  $R'$  substituents are due to the steric interactions in a planar molecule of an azadiene ligand, similar to those observed in the  $\pi$ -olefinic form (II). Besides, the substituent  $R'$  at the nitrogen atom hinders the coordination of the  $\text{Fe}(\text{CO})_4$  groups which is more significant the bulkier the substituent. The common property of the  $\sigma$ -( $N$ ) complexes (III) is their facile decarbonylation into  $(\eta^4\text{-azadiene})\text{tricarboxyliron}$  complexes (IV). This conversion strongly hinders the isolation of III;  $\eta^4\text{-azadiene}$  complexes (IV) may also be obtained from  $\eta^2\text{-olefinic}$  tetracarbonyl compounds (II) in the same way as takes place with oxadiene analogues [8].



For the compounds given in Table 2, the  $\pi$ - $\sigma$ -( $N$ ) rearrangement and the decarbonylation of complexes II proceed at a significantly slower rate than the tautomeric conversion  $\text{I} \rightleftharpoons \text{II}$ . Therefore we assume that they do not effectively influence the tautomeric equilibrium constant  $K_T$ .

## Experimental

The complexes  $(\eta^2\text{-PhCH=CHCOR})\text{Fe}(\text{CO})_4$  ( $R = \text{Et}$ ,  $n\text{-Bu}$ ,  $i\text{-Pr}$ ,  $t\text{-Bu}$ ,  $p\text{-C}_6\text{H}_4\text{Br}$ ,  $p\text{-C}_6\text{H}_4\text{Me}$ ,  $p\text{-C}_6\text{H}_4\text{OMe}$ ,  $p\text{-C}_6\text{H}_4\text{N}(\text{Me})_2$ ) were obtained by interaction of the corresponding ketones [9,10] with  $\text{Fe}_2(\text{CO})_9$  according to the procedures usually

\* The addition of a small amount of dichloromethane is needed because of the poor solubility of the complex.



employed in the synthesis of ( $\eta^2$ -olefin)tetracarbonyl iron complexes [11]. ( $\eta^2$ -CH<sub>2</sub>=CHCOMe)Fe(CO)<sub>4</sub> was synthesized in the same way; its IR and <sup>1</sup>H NMR spectra were identical to those described in ref. 12. The yields and properties of the tetracarbonyliron complexes are given in Table 3.

$\pi$ -Allyl- $\sigma$ -carbonyl tricarbonyliron complexes (I) were synthesized as described in ref. 2. Their yields and properties are given in Table 4.

Complex III (R = Me, R' = Et) was synthesized according to ref. 5; dark-red crystals, unstable in air, yield 25%. Found: C, 56.58; H, 4.56; Fe, 16.59; N, 3.92. C<sub>13</sub>H<sub>15</sub>FeNO<sub>4</sub> calcd.: C, 56.33; H, 4.43; Fe, 16.37; N, 4.10%. IR (hexane):  $\nu$ (C=O) 2048, 1969, 1962, 1937 cm<sup>-1</sup>; (KBr):  $\nu$ (C=N) 1620 cm<sup>-1</sup>.

#### *Synthesis of complex IV (R = Me, R' = C<sub>6</sub>H<sub>11</sub>)*

0.9 g (23 mmol) of complex I (R = Me, R' = C<sub>6</sub>H<sub>11</sub>) was dissolved in 50 ml methanol and heated for 2.5 h at 50°C. The reaction mixture was filtered and was evaporated to a minimal volume. The residue was recrystallized from ethanol. Orange-red crystals (0.2 g; 50% \*) were obtained, m.p. 104–107°C (decomp.). Found: C, 62.20; H, 5.76; Fe, 15.14; N, 3.98. C<sub>19</sub>H<sub>21</sub>FeO<sub>3</sub> calcd.: C, 62.14; H, 5.76; Fe, 15.21; N, 3.81%. IR (hexane)  $\nu$ (C=O) 2049, 1988, 1968 cm<sup>-1</sup>. <sup>1</sup>H NMR:  $\delta$  (ppm) 2.45 (3H, s), 5.74 (1H, d), 2.94 (1H, d, *J* 9.75 Hz), 7.06–7.64 (5H, m), 0.97–1.95 (11H, m).

The synthesis and isolation of the complexes were carried out under argon atmosphere. The IR absorption spectra of the equilibrium mixtures I  $\rightleftharpoons$  II (Table 1) were recorded on a Specord IR-75 spectrophotometer. The KBr cell thickness was 0.1–0.3 cm.

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\* According to the equation 2LFe(CO)<sub>4</sub> → LFe(CO)<sub>3</sub> + L + Fe(CO)<sub>5</sub> [8].