Pergamon

PII: S0277-5387(97)00052-1

*Polyhedron* Vol. 16, No. 17, pp. 2979–2985, 1997

0277-5387/97 $17.00+0.00

# Reaction of lithium dimethyl cuprate with carbonyl iron $\alpha,\beta$-unsaturated ketone complexes: the structure of a novel $\eta^3,\eta^3$-[(1,6-diphenyl)-3,4-(distyr-1-yl)-1,3,5-hexatriene]-Fe(CO)$_2$ complex

Olivia García-Mellado,[a]* René Gutiérrez-Pérez,[b] Cecilio Alvarez-Toledano,[c]* R. Alfredo Toscano[c] and Armando Cabrera[c]

[a]Facultad de Estudios Superiores Cuautitlán-UNAM. Depto. de Ciencias Químicas, Sección de Química Orgánica, Av. 1o. de Mayo s/n, Campo 1, Colonia Santa María de las Torres, Cuautitlán Izcalli, 54740, Edo. de México

[b]Centro de Investigación de la Facultad de Ciencias Químicas, Universidad Autónoma de, Puebla, Av. San Claudio y 14 Sur, Cd. Universitaria, Puebla, Pue

[c]Instituto de Química, UNAM, Cto. Exterior, Cd. Universitaria, Coyoacán, 04510, México, D.F.

(Received 15 July 1996; accepted 28 January 1997)

**Abstract**—The reaction between $\eta^4$-$\alpha,\beta$-unsaturated ketone-Fe(CO)$_3$ complexes and Me$_2$CuLi in the absence of CO atmosphere yields the corresponding $\eta^4$-vinylketene complexes **2**, **4** and **6**. The reaction between $\eta^2$-dibenzylideneacetone-Fe(CO)$_4$ and Me$_2$CuLi gives a new dimeric complex **8**, which was characterized by mass spectrometry, IR, $^1$H and $^{13}$C NMR spectroscopy and its structure confirmed by single-crystal X-ray analysis. © 1997 Elsevier Science Ltd

*Keywords*: Me$_2$CuLi; ketene; dimeric complex; iron(0).

Transition metal complexes of vinylketones are important intermediates in the synthesis of organic products such as $\beta$-lactams [1], amino acids [2], phenols [3] and heterocyclic compounds [4]. Some stable vinylketones complexes including those of chromium [5], cobalt [6,7] and iron [8] have been obtained, characterized and their chemical reactions extensively studied. There are some methods to produce those compounds, for example, the $\eta^4$-vinylketenes of iron(0) [8] complexes have been prepared by the reaction of the corresponding $\eta^4$-vinylketone of iron(0) compounds with organolithium reagents under a CO atmosphere. In this context, Thomas found that tricarbonyl complexes of $\alpha,\beta$-insaturated ketones react *via* a nucleophilic addition pathway with MeMgBr, MeLi and Me$_2$CuLi leading to 1,4-diketones [9].

In connection with our investigations on this subject, recently we have reported the synthesis of three new complexes of iron(0) derived from dibenzylideneacetone [10], using the Thomas' method. This gave a $\eta^4$-divinylketene of iron(0) and a new $\mu$-alkylidene complexes, the latter representing the stable carbene, like the intermediate suggested by Thomas [8].

In this work we report the results of the behavior of some $\eta^4$ and $\eta^2$ complexes of $\alpha,\beta$-unsaturated ketone iron(0) towards Me$_2$CuLi.

## EXPERIMENTAL

### *General methods*

$^1$H NMR and $^{13}$C NMR spectra were recorded on a Varian 300S spectrometer, using CDCl$_3$ as solvent and TMS as internal reference. IR spectra were rec-

---

* Authors to whom correspondence should be addressed.

orded on a Perkin–Elmer 283 B or 1420 spectrometer. The electronic impact (EI) ionization mass spectra were acquired on a JEOL JMS-AX505 HA mass spectrometer operated in the positive ion mode. The acquisition conditions were ion source temperature 230°C, ionization energy 70 eV, emission current 0.14 $\mu$A and ionization current 100 $\mu$A. Melting points were measured using a Mel-Temp II apparatus and are uncorrected. Column chromatography was performed with Merck silica-gel (70–230 mesh) using ethyl acetate : hexane in different ratios as eluent. All reagents were obtained from commercial suppliers and used as received. Reactions were performed under nitrogen atmosphere in carefully dried glassware. Diethyl ether and benzene were distilled from sodium–benzophenone ketyl under argon atmosphere and toluene was distilled over sodium wires. Complexes **1**, **3**, **5** and **7** were prepared by known procedures [10,11].

### *Reaction of lithium dimethylcuprate with tricarbonyl ($\eta^4$-vinylketone)iron(0) : formation of complexes* **2**, **4** *and* **6**

The preparation of complex **2** is representative of the procedure used for **4** and **6**. Methyl lithium (6.8 cm$^3$, 1.4 M in $Et_2O$) was added to a suspension of cuprous iodide (0.91 g, 4.8 mmol) in anhydrous diethyl ether (250 cm$^3$) at $-78$°C under nitrogen and the mixture was stirred by mechanical shaking for 15 min at $-78$°C and allowed to heat at $-23$°C for 45 min. The resulting clear and pale yellow solution was cooled at $-78$°C and treated with a solution of complex **1** (0.90 g 2.4 mmol) in anhydrous diethyl ether (100 cm$^3$) and the mixture was stirred at $-78$°C for 3.5 h. The resulting dark green solution was quenched with $NH_4Cl/NH_4OH$ aqueous solution (50 cm$^3$) at $-20$°C and allowed to warm at room temperature. The reaction mixture was extracted with diethyl ether, then dried with $Na_2SO_4$ anhydrous. The solvent was removed under reduced pressure and the residue was chromatographed on silica-gel. Elution with hexane : ethyl acetate first gave the complex **9** in less 1% yield, then **2** (0.10 g, 11%) as a yellow solid (m.pt: 143°C, decomposed) and finally dibenzylideneacetone (0.240 g, 42%). The yields for complexes **4** and **6** were 9 and 10% and their melting points are 137 and 111°C, respectively. The complexes **2, 4** and **6** were identified by comparison of its $^1$H, $^{13}$C NMR, EI-MS and IR spectra with literature values [8,10].

### *Reaction of lithium dimethylcuprate with $\eta^2$-tetracarbonyl(dibenzilideneacetone)iron(0) : formation of complex* **8**

A solution of **7** (0.96 g, 2.4 mmol) in diethyl ether (100 cm$^3$) was treated as above with lithium dimethyl cuprate (prepared with MeLi 6.8 cm$^3$, 1.4 M in $Et_2O$ and CuI 0.91 g, 4.8 mmol). Silica-gel chromatography gave complex **8** (0.31 g, 24%) as an orange solid; m.pt: 150°C (decomposed); IR (KBr): 1992, 1978, 1937, 1915 cm$^{-1}$, (M—CO); $^1$H NMR (200 MHz, $CDCl_3$) : $\delta$ 7.5–7.25 (m, 20 H, Ar), 6.65 (d, 2H, $J = 16$ Hz, $\mathrm{CCH{=}C\underline{H}Ph}$); 6.15 (d, 2H, $J = 16$ Hz, $\mathrm{CC\underline{H}{=}CHPh}$); 5.75 [d, 2H, $J = 10$ Hz, $\mathrm{Fe(CCH{=}C\underline{H}Ph]}$; 3.25 ppm [d, 2H, $J = 10$ Hz, $\mathrm{Fe(CC\underline{H}{=}CH)Ph]}$. $^{13}$C NMR (50 MHz, $CDCl_3$ : $\delta$ 214 (M—CO); 142–124 (Ar, C=C); 94 [$\mathrm{Fe(CH{=}\underline{C}H)Ph}$]; 88 [$\mathrm{Fe(\underline{C}H{=}CH)Ph}$]; 61 ppm [$\mathrm{{=}HC{-}\underline{C}{-}(CH{=})Fe}$]. EI-MS: 548 *m/z* ($M^{+\cdot}$), 492 ($M^+ - 2CO$), 436 for $C_{34}H_{28}$.

### *Formation of complex* **8** *from complex* **2** *or complex* **9**

A solution of complex **2** (0.25 g, 0.60 mmol) in benzene (50 cm$^3$) was refluxed for 24 h. The solution was evaporated under vacuum and the reaction mixture chromatographed on silica-gel with hexane : ethyl acetate to give **8** (0.03 g, 17%). Complex **9** (0.10 g, 0.20 mmol) was dissolved in toluene (50 cm$^3$) and was heated for 72 h. The solution was treated as above to give complex **8** (0.008 g, 8%).

### *X-ray data collection, structure solution and refinements for* **8**

The single crystals of complex **8** were grown by slow evaporation from a hexane : dichloromethane solution to 0°C as orange plates. A crystal summary is given in Table 2. The crystal dimensions were 0.60 × 0.44 × 0.06 mm. Intensity data were collected on a Siemens P4/PC diffractometer equipped with Mo-$K_\alpha$ radiation ($\lambda = 0.71073$ Å) using variable scan speed in the range 4.00–60.0° min$^{-1}$ in $\omega$, scan range ($\omega$) 1.20°, and selected the background/scan ratio to 50%. The data were corrected Lp and a semiempirical face-indexed absorption correction was applied.

The structure was solved by direct methods (SIR92) [12] and refined by full-matrix least-squares using the Siemens SHELXTL PLUS (PC version) [12] program, which gave final discrepancy indices $R = 4.71$, $wR = 5.48\%$ ($w = 1/[\sigma^2(F) + 0.0008\ F^2]$) with 1874 [$F > 4.0\sigma(F)$] observed reflections. Atomic coordinates have been deposited with the Cambridge Crystallographic Data Centre.

## RESULTS AND DISCUSSION

The complex **1** was treated with 2 equiv. lithium dimethylcuprate to give complex **2** as an air-stable yellow solid with 11% yield, $\mu$-alkylidene complex **9** [10] in less than 1% yield and dibenzylidenecacetone in 42% yield. Complex **2** was identified on the basis of their spectroscopic data (IR, $^1$H NMR, $^{13}$C NMR and mass spectrometry) as the tricarbonyl($\eta^4$-divinylketene)iron(0) complex previously reported. Complexes **3** and **5** under similar conditions also led to the isolation of compounds **4** and **6** in 9 and 10% yields,

Table 1. Bond lengths (Å) and angles (°) for $C_{34}H_{28}Fe(CO)_2$

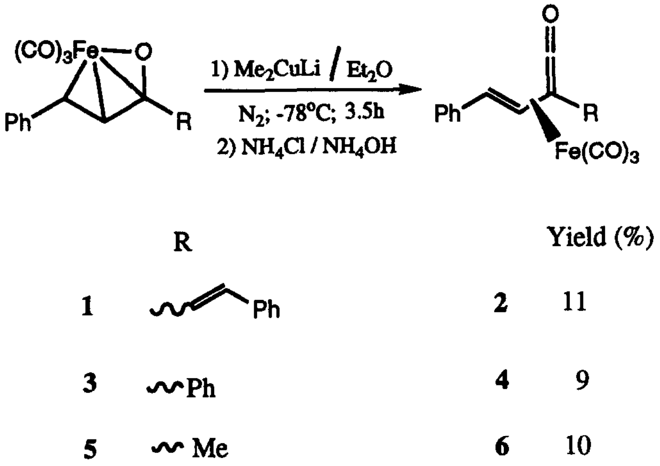
| | | | |
|---|---|---|---|
| Fe—C(1) | 2.157(3) | Fe—C(2) | 2.085(3) |
| Fe(1)—C(3) | 2.081(4) | Fe(1)—C(18) | 1.771(4) |
| C(1)—H(1) | 0.951(44) | C(1)—C(2) | 1.415(5) |
| C(1)—C(6) | 1.473(5) | C(2)—H(2) | 0.967(42) |
| C(2)—C(3) | 1.409(5) | C(3)—C(4) | 1.452(5) |
| C(3)—C(3′) | 1.500(6) | C(4)—H(4) | 0.917(44) |
| C(4)—C(5) | 1.337(5) | C(5)—H(5) | 0.885(43) |
| C(5)—C(12) | 1.466(6) | C(6)—C(7) | 1.385(6) |
| C(6)—C(11) | 1.380(6) | C(7)—C(8) | 1.383(7) |
| C(8)—C(9) | 1.387(9) | C(9)—C(10) | 1.347(9) |
| C(10)—C(11) | 1.377(8) | C(12)—C(13) | 1.400(6) |
| C(12)—C(17) | 1.386(6) | C(13)—C(14) | 1.372(7) |
| C(14)—C(15) | 1.365(8) | C(15)—C(16) | 1.367(8) |
| C(16)—C(17) | 1.393(7) | C(18)—O(18) | 1.143(6) |
| | | | |
| C(1)—Fe—C(2) | 38.9(1) | C(1)—Fe—C(3) | 71.2(1) |
| C(2)—Fe—C(3) | 39.5(1) | C(1)—Fe—C(18) | 108.2(2) |
| C(1)—Fe—C(1′) | 152.8(2) | C(2)—Fe—C(1′) | 120.5(1) |
| C(3)—Fe—C(1′) | 83.3(1) | C(18)—Fe—C(1′) | 90.4(2) |
| C(1)—Fe—C(2′) | 120.5(1) | C(2)—Fe—C(2′) | 110.0(2) |
| C(3)—Fe—C(2′) | 72.6(1) | C(18)—Fe—C(2′) | 129.2(2) |
| C(1)—Fe—C(3′) | 83.3(1) | C(2)—Fe—C(3′) | 72.6(1) |
| C(3)—Fe—C(3′) | 42.2(2) | C(18)—Fe—C(3′) | 149.7(2) |
| C(1)—Fe—C(18′) | 90.4(2) | C(2)—Fe—C(18′) | 129.7(2) |
| C(3)—Fe—C(18′) | 149.7(2) | C(18)—Fe—C(18′) | 94.5(3) |
| Fe—C(1)—C(2) | 67.8(2) | Fe—C(1)—C(6) | 125.9(3) |
| C(2)—C(1)—C(6) | 121.6(3) | Fe—C(2)—C(1) | 73.3(2) |
| Fe—C(2)—C(3) | 70.1(2) | C(1)—C(2)—C(3) | 121.9(3) |
| Fe—C(3)—C(2) | 70.4(2) | Fe—C(3)—C(4) | 127.6(3) |
| C(2)—C(3)—C(4) | 121.8(3) | Fe—C(3)—C(3′) | 68.9(1) |
| C(2)—C(3)—C(3′) | 116.0(3) | C(4)—C(3)—C(3′) | 122.1(3) |
| C(3)—C(4)—C(5) | 123.5(3) | C(4)—C(5)—C(12) | 126.8(3) |
| C(1)—C(6)—C(7) | 123.3(4) | C(1)—C(6)—C(11) | 119.4(4) |
| C(7)—C(6)—C(11) | 117.2(4) | C(6)—C(7)—C(8) | 121.3(4) |
| C(7)—C(8)—C(9) | 119.7(5) | C(8)—C(9)—C(10) | 119.5(6) |
| C(9)—C(10)—C(11) | 120.8(5) | C(6)—C(11)—C(10) | 121.5(5) |
| C(5)—C(12)—C(13) | 120.0(4) | C(5)—C(12)—C(17) | 122.6(4) |
| C(13)—C(12)—C(17) | 117.4(4) | C(12)—C(13)—C(14) | 121.5(4) |
| C(13)—C(14)—C(15) | 119.8(5) | C(14)—C(15)—H(15) | 119.3(3) |
| C(14)—C(15)—C(16) | 120.6(5) | C(15)—C(16)—C(17) | 119.8(5) |
| C(12)—C(17)—C(16) | 120.8(4) | Fe—C(18)—O(18) | 176.9(4) |

respectively (Scheme 1). The spectroscopic data correspond to those reported in the literature [8].

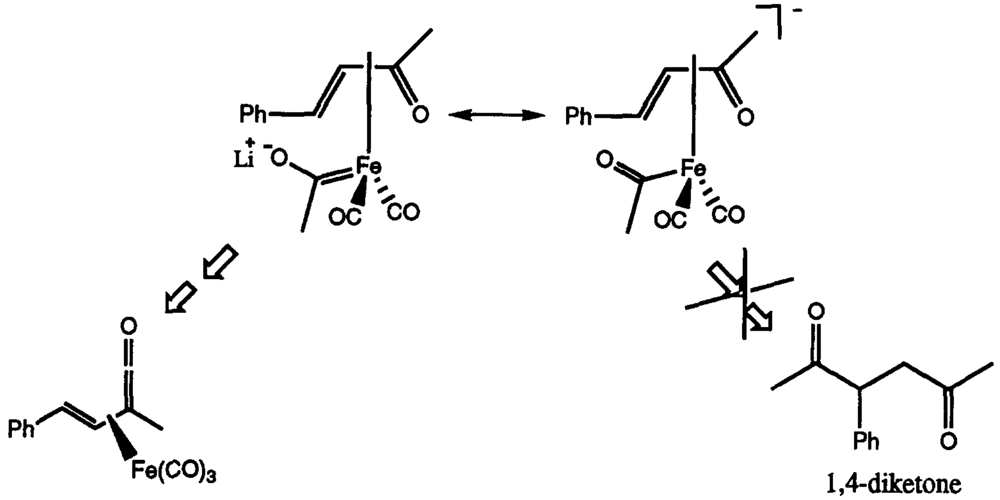
The mechanism for the formation of $\eta^4$-vinylketene can be thought to be similar to that proposed by Thomas [8], with the key metal acyl intermediate, suffering an acyl elimination instead of the acyl group migration to a $\beta$-carbon of ligand. Vinylcarbene then undergoes carbonylation, taking the CO from the decomposition of tricarbonyl($\eta^4$-dibenzylideneacetone)iron(0) to generate in this way the $\eta^4$-vinylketene of iron (Scheme 2).

When the tetracarbonyl($\eta^2$-vinylketone)iron(0) complex **7** was treated with 2 equiv. of lithium dimethylcuprate in the aforementioned conditions, the reaction product was an air-stable orange crystalline compound in 24% yield. The structure was established by spectroscopic and confirmed by X-ray crystal analysis as a new dimeric complex of iron(0) **8** similar to one complex previously reported by Alvarez *et al.* (Scheme 3) [13].

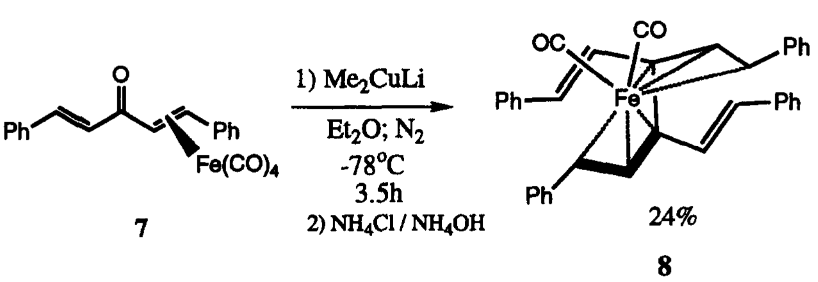
Complex **8** exhibits in the IR spectrum $\nu$(CO) frequencies of high intensity at 1992, 1978, 1937 and 1915 $cm^{-1}$, which correspond to the M—CO bond, and no ketone carbonyl frequency was found. The mass spectrum shows $M^+$ at 548 *m*/*z* and a signal with significant relative intensity at 492 *m*/*z* corresponding to the M—2CO fragment, in 436 *m*/*z* appears a fragment which belongs to $C_{34}H_{28}$, all ions suggest that two $C_{17}H_{14}$ units are coupled through carbon–carbon bond formation. The $^1H$ NMR spectrum showed the aromatic protons between $\delta$ 7.5 and 7.1 ppm, at $\delta$ 6.65 and 6.15 ppm ($J = 16$ Hz), as two doublets coupled, which belong to the free double bond, at high field displayed at $\delta$ 5.75 and 3.25 ppm as two doublets



Scheme 1.



Scheme 2.



Scheme 3.

($J = 10$ Hz) owing to the coordinated double bond. The proton counting $^1$H NMR spectrum suggests that the formed complex must be in a high symmetrical arrangement. This fact was confirmed by the simplicity of the $^{13}$C NMR spectrum which exhibits a resonance for a terminal CO at $\delta$ 214 ppm, resonance for phenyl groups and free double bonds between $\delta$ 142 and 124 ppm (nine peaks) and three signals at $\delta$ 94, 89 and 61 ppm, which correspond to coordinated double bonds and a quaternary carbon atom.

The structure of complex **8** was finally assessed by an X-ray diffraction study. Its ORTEP projection is shown in Fig. 1 whereas the bond distances (Å) and the bond angles (deg) are gathered in Table 1. In the crystal, the whole molecule lies on a special position with local $C_2$ symmetry, as a result the organic ligand

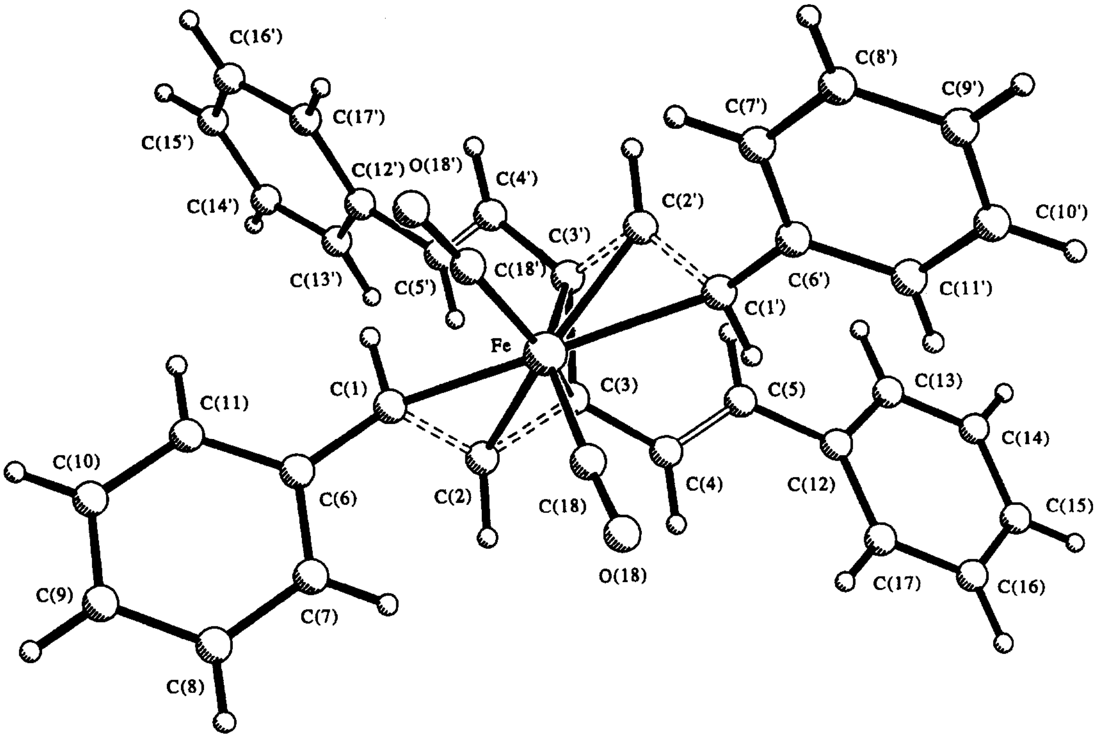


Fig. 1.

Table 2. Crystal data and refinement for complex **8**

| | |
|---|---|
| Formula | $C_{36}H_{28}FeO_2$ |
| $M_w$ | 548.4 |
| Crystal system | Monoclinic |
| Space group | $C_2/c$ |
| $a$ (Å) | 24.422(3) |
| $b$ (Å) | 9.202(2) |
| $c$ (Å) | 12.833(2) |
| $\beta$ (°) | 95.15(2) |
| $V$ (Å$^3$) | 2872.3 |
| $Z$ | 4 |
| $D_c$ (Mg m$^{-3}$) | 1.268 |
| $F$(000) | 1144 |
| Diffractometer | Siemens P4/PC |
| Radiation | Mo-$K_\alpha$ |
| Scan type | $\omega$–$2\theta$ |
| Scan range (°) | $3 < 2\theta < 50$ |
| Data collected | 2591 |
| Data used [criterion] | 1874 [$F > 4\sigma(F)$] |
| $R(R_w)$(%) | 4.71 (5.48) |

Table 3. Atomic coordinates ( × 10$^4$) and equivalent isotropic displacement coefficient (Å$^2$ × 10$^3$) for complex **8**

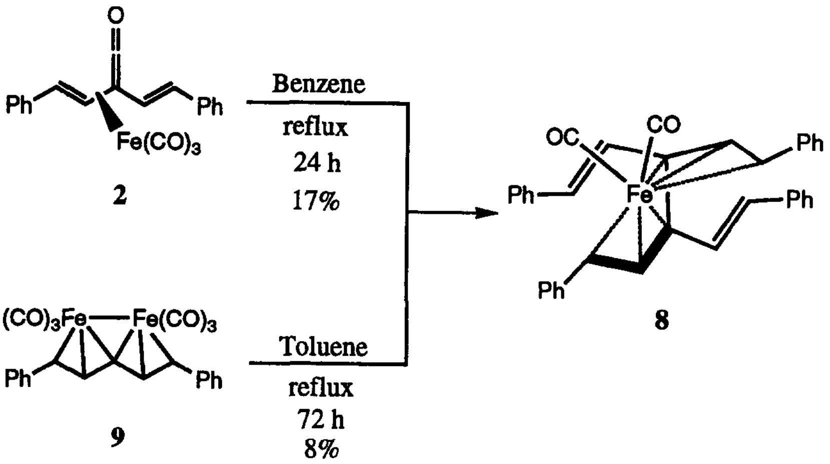
| | $x$ | $y$ | $z$ | $U$(eq)$^a$ |
|---|---|---|---|---|
| Fe | 10000 | 889(1) | 7500 | 35(1) |
| C(1) | 9138(1) | 1441(4) | 7305(3) | 41(1) |
| C(2) | 9445(1) | 2190(4) | 6590(3) | 39(1) |
| C(3) | 9918(1) | 2999(4) | 6923(2) | 39(1) |
| C(4) | 10248(2) | 3706(4) | 6183(3) | 42(1) |
| C(5) | 10644(2) | 4670(4) | 6464(3) | 46(1) |
| C(6) | 8686(2) | 451(4) | 6946(3) | 46(1) |
| C(7) | 8685(2) | −382(5) | 6047(3) | 60(1) |
| C(8) | 8237(2) | −1231(6) | 5707(4) | 83(2) |
| C(9) | 7777(3) | −1245(7) | 6268(5) | 97(3) |
| C(10) | 7776(2) | −443(8) | 7145(5) | 96(3) |
| C(11) | 8225(2) | 388(6) | 7492(3) | 69(2) |
| C(12) | 11033(2) | 5308(5) | 5788(3) | 47(1) |
| C(13) | 11337(2) | 6541(5) | 6117(3) | 56(1) |
| C(14) | 11712(2) | 7150(6) | 5513(4) | 70(2) |
| C(15) | 11797(2) | 6545(6) | 4570(4) | 73(2) |
| C(16) | 11513(2) | 5334(6) | 4221(4) | 69(2) |
| C(17) | 11127(2) | 4720(5) | 4825(3) | 57(1) |
| C(18) | 10130(2) | −417(5) | 6539(3) | 53(1) |
| O(18) | 10238(2) | −1255(5) | 5933(3) | 97(2) |

$^a$ Equivalent isotropic $U$ defined as one third of the trace of the orthogonalized $U_{ij}$ tensor.

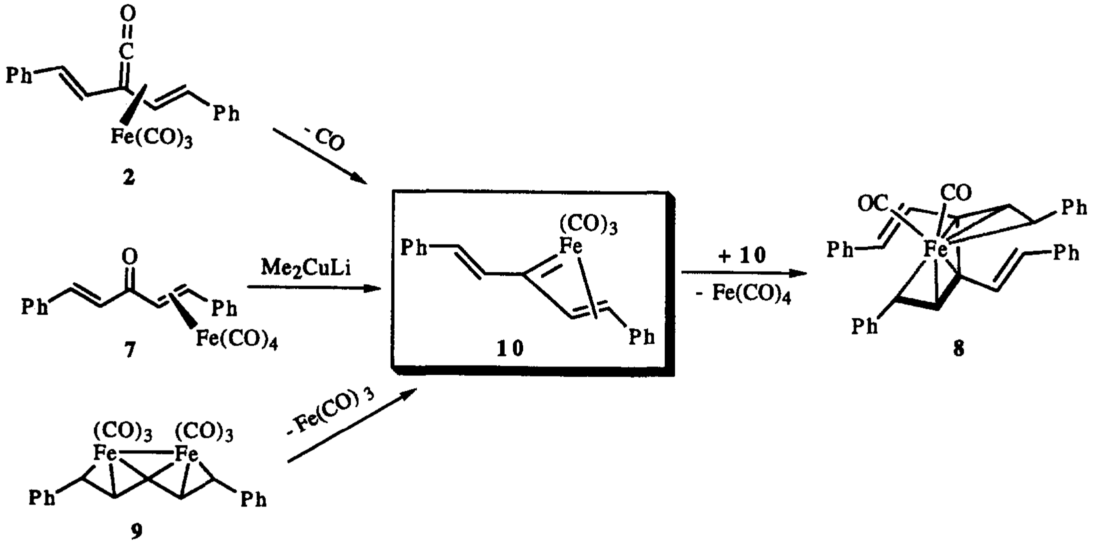
comprises two nearly coplanar moieties connected through the C3—C3′ bond and making a dihedral angle of 104° between the two halves.

Inspection of the bond lengths and angles reveals the presence of an allylic system (Cl—C2—C3) [14] and a formal C═C double bond (C4—C5) on each half. Phenyl rings (C6 to C11 and C12 to C17) are slightly twisted (34 and 28°, respectively), towards the same side, from the mean plane calculated through Cl to C5.

The coordination polyhedron can be best described



Scheme 4.



Scheme 5.

as a tetrahedron with two CO groups and two allyl moieties coordinated to the iron atom. The most important feature of complex **8** is the coordination of the $C_{34}H_{28}$ unit as a bis chelating ($\pi$-allyl) ligand to $Fe(CO)_2$ [15,16].

On the other hand, the same dimeric complex **8** was obtained in 17% yield when the $\eta^4$-vinylketene of iron no **2** was refluxed in benzene during 24 h and complex **8** was also produced in 8% yield when a solution of complex **9** was refluxed in toluene during 72 h (Scheme 4).

Formation of complex **8** in all these cases can be explained *via* the Thomas' intermediate [17,18,19]: this carbene probably undergoes a coupling reaction leading to dimeric complex **8** either *via* free carbene or complex carbene **10** with loss of a $Fe(CO)_4$ unit [20] (Scheme 5).

## CONCLUSION

In conclusion, in this work we found that reaction of $\eta^4$-$\alpha,\beta$-unsaturated ketone-$Fe(CO)_3$ complexes with $Me_2CuLi$ proceeded in the absence of CO atmosphere to afford the corresponding $\eta^4$-vinylketene complexes in low yields instead of generating the 1,4-diketone. Also noteworthy was the formation of a new dimeric complex when $Me_2CuLi$ reacts with $\eta^2$-dibenzylideneacetone-$Fe(CO)_4$.

*Acknowledgements*—The authors gratefully acknowledge DGAPA-UNAM-IN204395 for financial support and Rocío Patiño Maya, Javier Flores and Isabel Chávez for technical assistance.

## REFERENCES

1. Hegedus, L. S., Imwinkelreid, R., Alarid-Sargent,

M., Drorak, D. and Saton, Y., *J. Am. Chem. Soc.*, 1990, **112,** 1109.

2. Hegedus, L. S., Schwindt, M. A., Delombert, S. and Imwinkelreid, R., *J. Am. Chem. Soc.*, 1990, **112,** 2264.
3. Morris, G., Saberi, S. P. and Thomas, S. E., *J. Chem. Soc., Chem. Commun.*, 1993, 209.
4. Reduto dos Reis, A. C. and Hegedus, L. S., *Organometallics*, 1995, **14,** 1585.
5. (a) Anderson, B. A., Wulff, W. D. and Rheingold, A. L., *J. Am. Chem. Soc.*, 1990, **112,** 8615; (b) Anderson, B. A., Bao, J., Branvold, T. A., Challener, C. A., Wulff, W. D., Xu, Y.-C. and Rheingold, A. L., *J. Am. Chem. Soc.*, 1993, **115,** 10671.
6. (a) Wulff, W. D., Gilberton, S. R. and Springer, J. P., *J. Am. Chem. Soc.*, 1986, **108,** 520; (b) Huffman, M. A. and Liebeskind, L. S., *J. Am. Chem. Soc.*, 1990, **112,** 8617.
7. Huffman, M. A., Liebeskind, L. S. and Pennington, W. T., *Organometallics*, 1992, **11,** 255.
8. Alcock, N. W., Richards, C. J. and Thomas, S. E., *Organometallics*, 1991, **10,** 231.
9. (a) Thomas, S. E., *J. Chem. Soc., Chem. Commun.*, 1987, 226; (b) Dank, T. N., Rakshit, D. and Thomas, S. E., *J. Chem. Soc., Perkin Trans.* (*I*), 1988, 2091.
10. (a) Cano, A. C., Zuñiga-Villareal, N., Alvarez, C., Toscano, R. A., Cervantes, M. and Díaz, A., *J. Organomet. Chem.*, 1994, **464,** C23; (b) Bernès, S., Toscano, R. A., Cano, A. C., García Mellado, O., Alvarez-Toledano, C., Rudler, H. and Daran, J.-C., *J. Organomet. Chem.*, 1995, **489,** 15.
11. Brodie, A. M., Johnson, B. F. G., Josty, P. L. and Lewis, J., *J. Chem. Soc., Dalton Trans.*, 1972, 2031.
12. Sheldrich, G. M., *SHELXTLS/PC User's Manual.* Siemens Analytical X-ray Instruments, Madison, WI, 1990.
13. Alvarez-Toledano, C., Cano, A. C., Toscano, R. A., Parlier, A. and Rudler, H., *Bull. Soc. Chim. Fr.*, 1993, **130,** 601.
14. Chen, J.-B., Lei, G.-X., Shao, M.-C., Xu, X.-J. and Zhang, Z.-Y., *J. Chem. Soc., Chem. Commun.*, 1988, 1296 and refs therein.
15. King, R. B. and Harmon, C. A., *J. Am. Chem. Soc.*, 1976, **98,** 2408.
16. Nesmeyanov, A. N., Ustynyuk, Y. A., Kristaya, I. I. and Shechembelov, G. A., *J. Organomet. Chem.*, 1968, **14,** 395.
17. Bkonche-Waksman, I., Ricci, J. S., Koetzle, T. F., Wiechmann, J. and Herrmann, W. A., *Inorg. Chem.*, 1985, **24,** 1492.
18. Heirman, W. A., Gimeno, J., Wichman, J., Ziegler, M. L. and Balbach, B., *J. Organomet. Chem.*, 1982, **231,** C69.
19. Geoffroy, G. L. and Bassner, S. L., *Adv. Organomet. Chem.*, 1988, **28,** 1.
20. Alcock, N. W., Richards, C. J. and Thomas, S. E., *Organometallics*, 1992, **11,** 154.