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Photolysis of 3-methyl- and 3-isopropyl-3-ferrocenylcyclopropenes

Elena I. Klimova [a,*], Marcos Martinez Garcia [b], Tatiana Klimova [a],
Cecilio Alvarez Toledano [b], Ruben Alfredo Toscano [b], Lena Ruiz Ramirez [a]

[a] *Facultad de Química, Universidad Nacional Autónoma de México, Cd. Universitaria, Coyoacán, C.P. 04510 Mexico D.F., Mexico*
[b] *Instituto de Química, Universidad Nacional Autónoma de México, Cd. Universitaria, Coyoacán, C.P. 04510 Mexico D.F., Mexico*

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Abstract

The photochemistry of 3-methyl- and 3-isopropyl-3-ferrocenylcyclopropenes (**10** and **11**) has been studied. Upon triplet excitation (sensitized irradiation) both cyclopropenes are dimerized to a tricyclohexane dimers whereas in the singlet state (direct irradiation) cyclopropene **10** is converted to 3-ferrocenyl-3-methylidenepropyne and *trans*-2-ferrocenyl-2-butene indicating that there is an intermediate vinyl carbene. Cyclopropene **11** is converted to 1-ferrocenyl-4,4-dimethylcyclobutene. Direct irradiation of the mixture of **11** with 2-ferrocenyl-3-methyl-1-butene leads to the formation of 1,3-diferrocenyl-6,6-dimethyl-3-isopropenylcyclohexene (**24**). A possible reaction pathway via intermediate radicals is discussed. X-ray structural data are presented for 3,6-diferrocenyl-3,6-diisopropyltricyclo$[3.1.0.0.^{2,4}]$hexane and compound **24**. © 2000 Elsevier Science S.A. All rights reserved.

Keywords: Ferrocene; Cyclopropene; Vinylcarbenes; Photolysis; X-ray structural analysis; Radical; Cyclobutene; Tricyclohexanes

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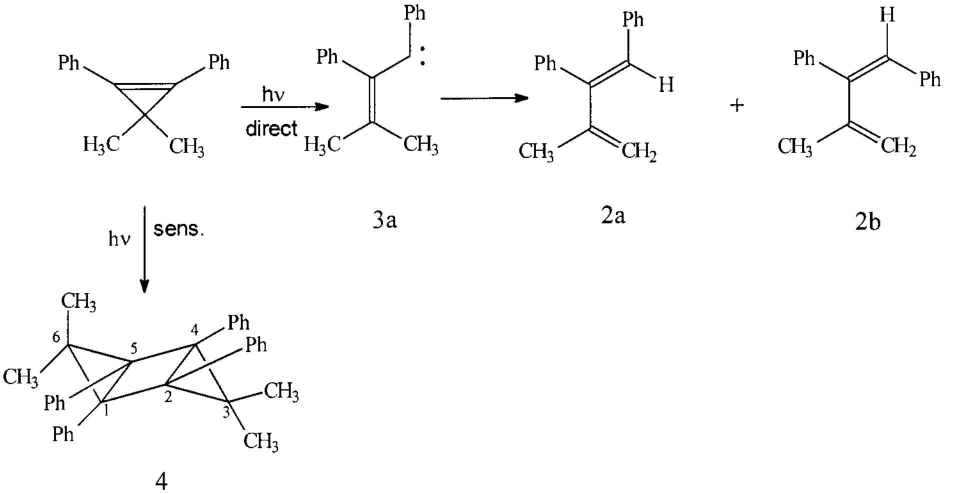
1. Introduction

Photochemical transformations of the majority of cyclopropenes with alkyl and aryl substituents depend largely on the multiplicities of the excited states [1]. The singlet excitation (direct irradiation) results, as in the case of thermolysis, in the cleavage of the σ-bond and generation of vinylcarbene intermediates, which are then transformed into the reaction products. Thus direct irradiation of 3,3-dimethyl-1,2-diphenylcyclopropene (**1**) results in two isomeric 3-methyl-1,2-diphenylbuta-1,3-dienes (**2a** and **2b**), which are formed via an intermediate vinylcarbene (**3a**) [2]:



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* Corresponding author. Tel./fax: + 525-622-5366.
E-mail address: klimova@servidor.unam.mx (E.I. Klimova).

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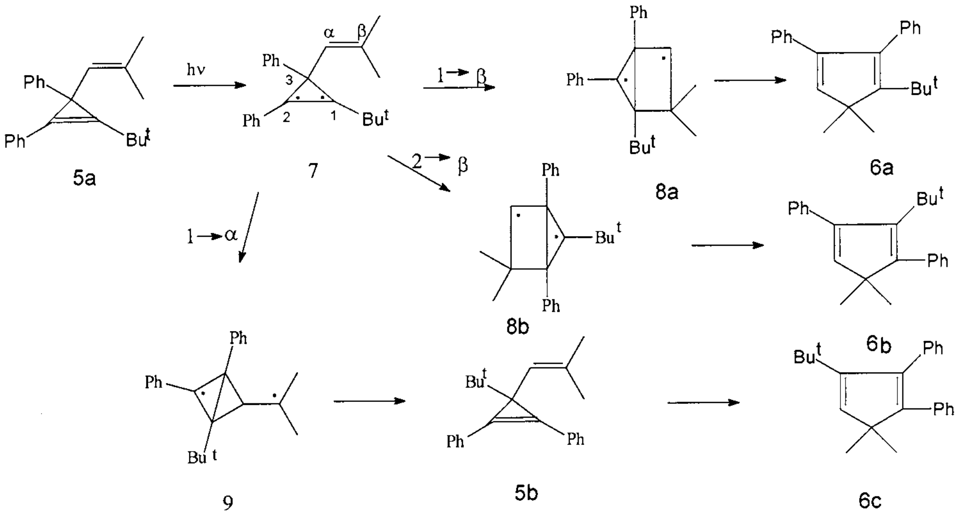
The triplet excitation (irradiation in the presence of sensitizers) of the same compound affords a [2 + 2]-cyclodimerization product, viz., 3,3,6,6-tetramethyl-1,2,4,5-tetraphenyltricyclo[3.1.0.0^{2,4}]-hexane (**4**) [3].

2-Vinyl-substituted cyclopropenes present exceptions. Their photolysis results preferentially in mixtures of isomeric cyclopentadienes irrespective of the multiplicities of the excited states. Thus 1-*tert*-butyl-3-isobutenyl-2,3-diphenylcyclopropene (**5a**) [4,5] is converted into a mixture of three cyclopentadienes **6a**, **6b**, and **6c** and an isomeric cyclopropene (**5b**) upon any type of irradiation.

Presumably, phototransformation of 3-vinylcyclopropenes occurs through a mechanism [4,5] implying the intermediacy of di-π-methane derivative **7** [6,7] with its subsequent transformation into biradicals **8a**, **8b**, and **9**:



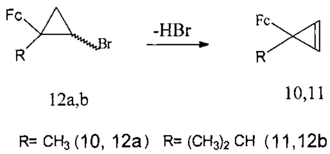
Isolation of small amounts of the isomeric compound **5b** corroborates the suggested scheme for the transformation of cyclopropene **5a**.

Recently, cyclopropenes with the ferrocenyl substituent at position three of the three-membered ring [8–11] have become the subject of studies. It was shown that on thermolysis ferrocenylvinylcarbene intermediates are formed, which then undergo transformation or disproportionation.

Photochemical transformations of ferrocenylcyclopropenes have not been studied so far, although many compounds of the ferrocene series are known to undergo photolysis. The main characteristic feature of photolytic transformations of these compounds is the cleavage of the iron–cyclopentadiene bond. This is especially facile in ferrocene derivatives which contain positively charged atoms (total or partial positive charge) in α-positions relative to the five-membered rings [12–14]. In 3-ferrocenylcyclopropenes, the α-positions relative to the cyclopentadienyl rings of the metallocene moieties are occupied by the carbon atoms with elevated electron density [15–17]. Hence, one could expect that the organometallic system would survive under conditions of the photochemical transformations of the three-membered ring, which, in our opinion, is of considerable interest.

2. Results and discussion

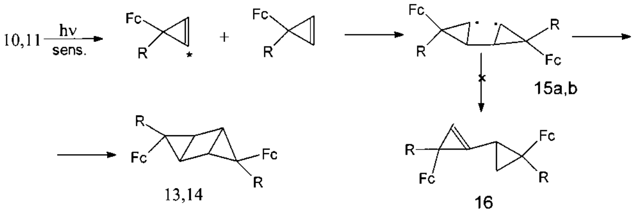
In the present work, photolysis of 3-ferrocenyl-3-methyl- and 3-isopropylcyclopropenes (**10** and **11**) is studied. The starting cyclopropenes were obtained by dehydrobromination of the corresponding monobromoferrocenylcyclopropanes (**12a**, **b**) with ^{t}BuOK in DMSO [8,9].



Compounds **10** and **11** are unstable on storage under ordinary conditions (room temperature) and even at low temperature (0°C); therefore, freshly prepared samples were used for photolysis.

2.1. *Photolysis of 3-alkyl-3-ferrocenylcyclopropenes (**10** and **11**) in the presence of a sensitizer*

Solutions of cyclopropenes **10** and **11** in acetonitrile were subjected to photolysis in the presence of benzophenone using a 200 W Hanovia medium pressure lamp. Following irradiation, the solvent was distilled off and the ^{1}H-NMR of the product mixture was recorded. As followed from the spectral data, dimeric products (**13** and **14**, respectively) were formed in both cases in ca. 70% yields. The absence in the ^{1}H-NMR spectra of singlets for the olefinic protons inherent in the starting cyclopropenes ($\delta = 7.15$ and 6.95 ppm) and the appearance of high-field singlets for the aliphatic protons ($\delta = 1.64$ and 1.58 ppm) give evidence in favor of the formation of dimeric products. These were purified by column chromatography on alumina (Brockmann activity III), and analytically pure samples were obtained by crystallization.



R=CH$_3$ (10,13,15a); R= (CH$_3$)$_2$CH (11,14,15b)

The structure of tricyclohexane (**14**) was confirmed by X-ray diffraction analysis. A single crystal of this compound was grown from a hexane solution. The general view of the molecule of **14** is shown in Fig. 1, and its molecular structure is shown in Fig. 2. An interesting feature of the crystal structure is the presence of only one molecule of compound **14** in the unit cell (Fig. 2).

As follows from the X-ray diffraction data, compound **14** is anti-3,6-diferrocenyl-3,6-diisopropyltricyclo[3.1.0.0^{2,4}]hexane. The tricyclohexane system is the basic element in the structure of the dimer **14**, the ferrocenyl and isopropyl substituents occupying *trans*-positions at the C(11) and C(11a) atoms of the tricyclic system. The central four-membered ring is planar; the dihedral angles between the four-membered and three-membered rings are 114°. The lengths of the C(12)–C(13) and C(12a)–C(13a) bonds in the three-membered ring are somewhat larger (1.557 Å), while those of the C(12)–C(13a) and C(12a)–C(13) bonds in the four-membered ring are somewhat smaller (1.500 Å) than the standard values. Typical C–C bond lengths in cyclopropanes are ca. 1.51 Å [18,19] and those in cyclobutanes are ca. 1.55 Å [20–22]. The mean Fe–C distance is equal to 2.033 Å, and the mean C–C bond length in the cyclopentadiene rings is equal to 1.392 Å.

As expected, the ferrocenyl fragments of the starting compounds **10** and **11** survived under the irradiation conditions. Only cyclopropene fragments underwent photochemical transformations, as has been described earlier for alkyl- and aryl-substituted cyclopropenes [5,23]. The dimerization occurred, presumably, via an analogous stepwise mechanism involving the biradical intermediates (**15a**, **b**) [23]. However, we failed to detect any cyclopropylcyclopropane dimers of the type **16a** and **b**, which would corroborate the stepwise character of the process.

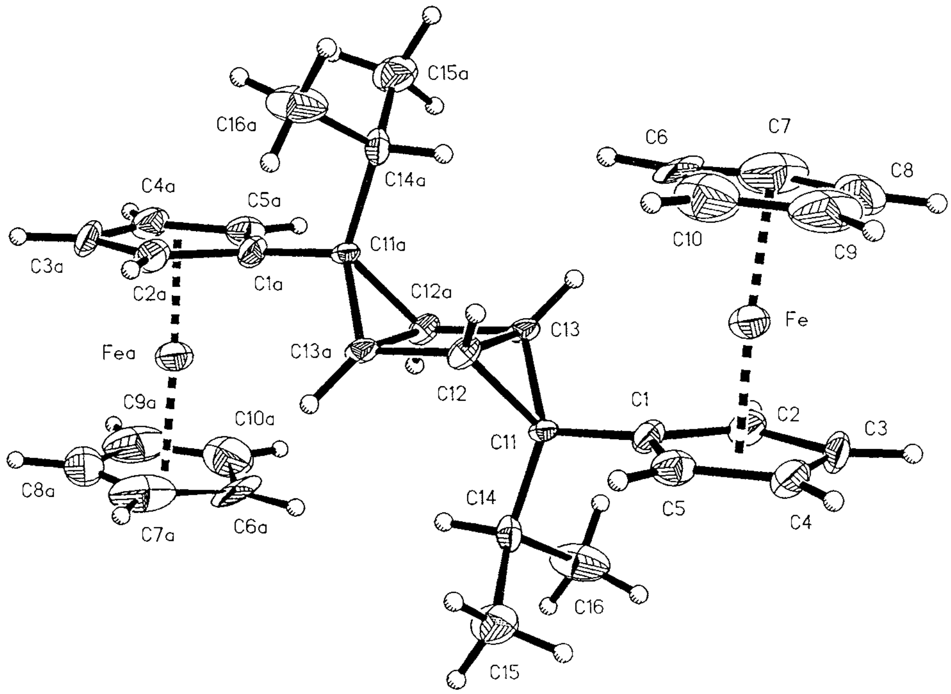


Fig. 1. Crystal structure of **14**. Selected bond lengths (Å): C_{11}–$C_{12} = 1.521(13)$; C_{11}–$C_{13} = 1.515(12)$; C_{12}–$C_{13} = 1.557(15)$; C_{12a}–$C_{13a} = 1.557(15)$; C_{12}–$C_{13a} = 1.500(11)$; C_{12a}–$C_{13} = 1.500(11)$. Selected bond angles (°): C_{13a}–C_{12}–$C_{11} = 113.9(9)$; C_{12a}–C_{13}–$C_{11} = 114.1(9)$; C_{11}–C_{12}–$C_{13} = 59.0(6)$; C_{12a}–C_{13}–$C_{12} = 90.0(9)$.

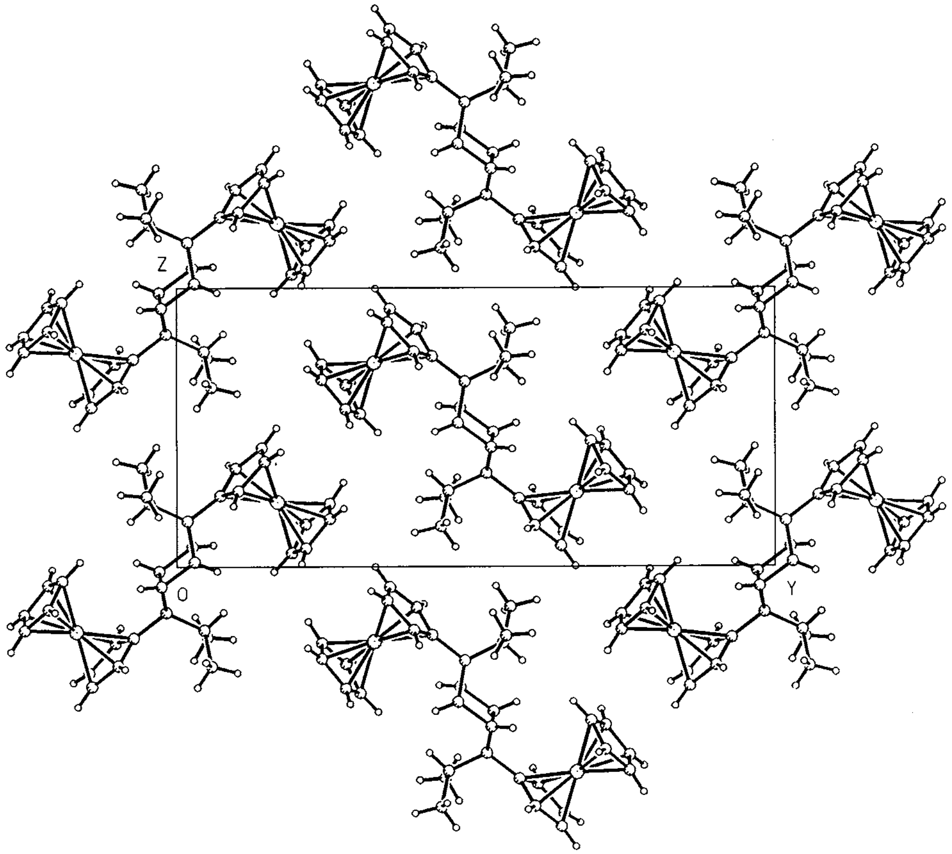
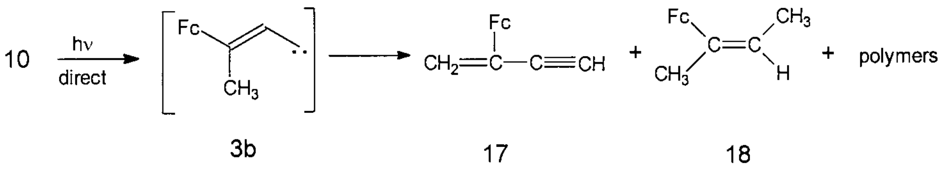


Fig. 2. Crystal packing of **14**.

*2.2. Direct photolysis of 3-alkyl-3-ferrocenylcyclopropenes (**10** and **11**)*

Irradiation of solutions of compounds **10** and **11** in benzene was carried out with the use of a 200 W Hanovia medium pressure lamp and a Vycor filter. Following the photolysis, the solvent was distilled off in vacuo and the residue was chromatographed on alumina.

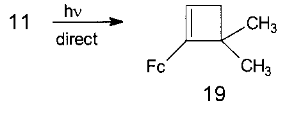
Direct photolysis of cyclopropene **10** resulted in 2-ferrocenylbut-1-en-3-yne (**17**), *trans*-2-ferrocenylbut-2-ene (**18**) [24] and unidentified polymeric products:



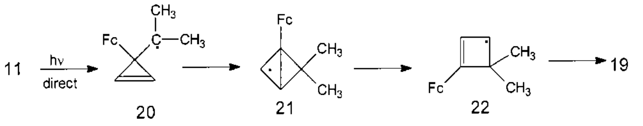
Obviously, the reaction occurs via ferrocenylvinylcarbene intermediate **3b**, which undergoes subsequent disproportionation.

An analogous process has recently been described by us for thermolysis of 3-alkyl-3-ferrocenylcyclopropenes (alkyl = methyl, isopropyl, cyclobutyl) [24]. The formation of ferrocenylvinylacetylenes and ferrocenylalkenes was the predominant process and occurred as a result of heterolysis of a σ-bond of the small ring leading to ferrocenylvinylcarbenes and their subsequent disproportionation.

It was found that the photochemical transformations of 3-isopropyl-3-ferrocenylcyclopropene (**11**) differed from those of compound **10**. Direct irradiation of cyclopropene **11** was accompanied by substantial polymerisation of the reaction mixture so that only one reaction product could be isolated by chromatography in 27% yield. Its ^{1}H-NMR spectrum contained signals for one ferrocenyl group, two singlets for two methyl groups, a triplet for one olefinic proton, and a doublet for two aliphatic protons. These data allowed us to ascribe the structure of 1-ferrocenyl-4,4-dimethylcyclobutene (**19**) to the product obtained.

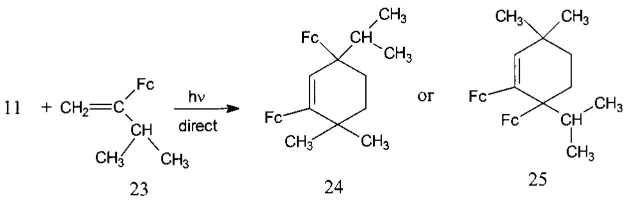


In our opinion, the formation of cyclobutene **19** involves initially generation of cyclopropenyl(dimethyl)-methyl radical (**20**), its transformation into bicyclobutyl and cyclobutenyl radicals **21** and **22**, respectively:



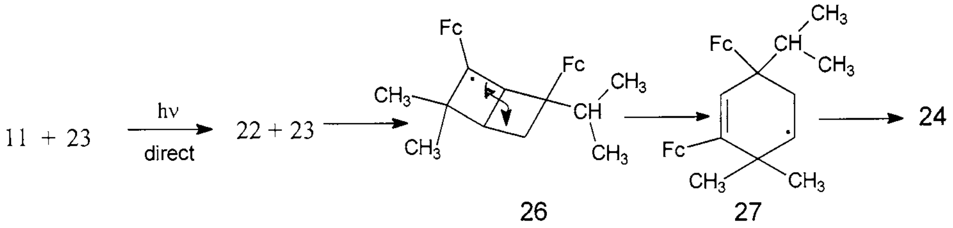
Compound **19** is an orange oil which is rapidly decomposed on storage even at low temperature, this was characterized by ^{13}C-NMR spectral and elemental analysis data.

In order to support the suggested reaction scheme, we performed photochemical reaction of cyclopropene **11** with 2-ferrocenyl-3-methylbut-1-ene (**23**) as a potential free-radical acceptor. In this case, the regioselective reaction afforded a single product in high yield. Its ^{1}H-NMR spectrum contained signals for two ferrocenyl substituents, one isopropyl and two methyl groups, a singlet for one olefinic proton, and two multiplets for two methylene groups of a CH_2CH_2 fragment. These data are in accord with either of two structures, viz., **24** or **25**:



Conclusive evidence of the structure of the reaction product was obtained from X-ray diffraction analysis of its single crystal obtained by crystallization from *n*-hexane. It was established that this compound is 1,3-diferrocenyl-3-isopropyl-6,6-dimethylcyclohexene (**24**) (Fig. 3). The six-membered ring exists in a half-chair conformation (see legend to Fig. 3). The mean Fc_1–C and Fc_2–C distances are equal to 2.035 and 2.051 Å, respectively, the mean C–C bond lengths in the cyclopentadienyl rings are equal to 1.406 (Fc_1) and 1.401 Å (Fc_2).

The photochemical reaction of cyclopropene **11** with butene **23** seems to occur according to a scheme involving intermediate bicyclohexane and cyclohexene radicals (**26** and **27**):



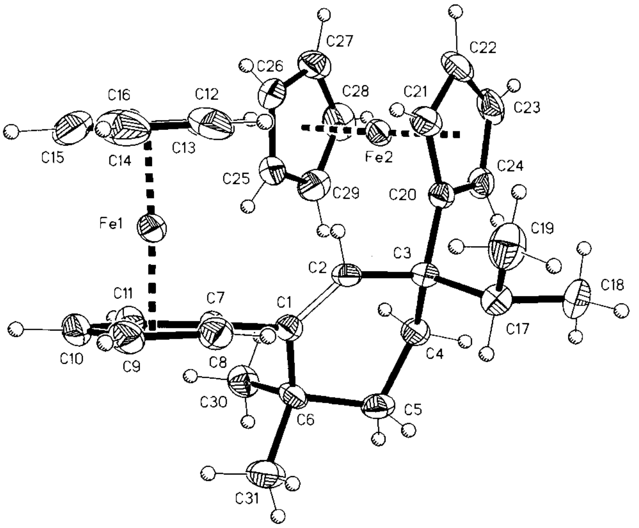


Fig. 3. Crystal structure of **24**. Selected bond lengths (Å): C_1–C_2 = 1.320(9); C_2–C_3 = 1.514(8); C_3–C_4 = 1.549(9); C_4–C_5 = 1.505(10); C_5–C_6 = 1.546(9); C_1–C_6 = 1.528(9). Selected bond angles (°): C_1–C_6–C_5 = 108.6(6); C_5–C_4–C_3 = 112.5(6); C_2–C_1–C_6 = 123.0(5); C_1–C_2–C_3 = 127.5(6); C_2–C_3–C_4 = 107.6(5); C_4–C_5–C_6 = 112.3(5); C_2–C_1–C_7 = 120.4(6); C_2–C_3–C_{17} = 108.7(5); C_{20}–C_3–C_4 = 110.1(5).

It should be mentioned that there are no literature data on photochemical reactions of cyclopropenes in the presence of potential free-radical scavengers.

Thus, the results of this study show that the character of photochemical transformations of 3-alkyl-3-ferrocenylcyclopropenes (**10** and **11**) depends on both the multiplicities of the excited states and the nature of the alkyl substituent. The metallocene fragment remained intact in all the photochemical reactions, as expected.

3. Experimental

The solvents were dried by standard methods and distilled prior to use. Column chromatography was carried out on Al_2O_3 (activity III according to Brockmann). The ^{1}H-NMR spectra were registered in $CDCl_3$ on a Unity Inova Varian spectrometer at 300 MHz using Me_4Si as the internal standard. The parameters of the unit cell and the X-ray diffraction intensities were recorded on a Siemens P4/PC. The crystallographic data, the experimental conditions and corrections are given in Table 1. The photochemical reactions were

Table 1
Crystal data, data collection and refinement parameters for **14** and **24**

Data	**14**	**24**
Molecular formula	$C_{32}H_{36}Fe_2$	$C_{31}H_{36}Fe_2$
Formula weight (g mol^{-1})	532.30	520.30
Temperature (K)	293	293
Crystal system	Monoclinic	Monoclinic
Space group	$P2_1/n$	$C2/c$
a (Å)	6.064(1)	20.483(3)
b (Å)	21.328(2)	8.665(1)
c (Å)	10.138(1)	30.293(4)
α (°)	90.0	90.0
β (°)	100.54(1)	109.68(1)
γ (°)	90.0	90.0
V (Å^3)	1289.1(3)	5062.5(12)
Z	2	8
D_{calc} (mg m^{-3})	1.371	1.365
Absorption coefficient (mm^{-1})	1.144	1.163
F(000)	560	2192
Radiation, Mo–K$_\alpha$ λ (Å)	0.71073	0.71073
Monochromator	Graphite	Graphite
θ range (°)	1.50–25.00	1.50–25.0
Reflections collected	2484	4599
Independent reflections	2263	4464
R_{int}	0.0859	0.1322
Final R indices [$I>2\sigma(I)$]	$R_1=0.0955$, $wR_2=0.1323$ [a]	$R_1=0.0587$, $wR_2=0.1106$ [b]
R indices (all data)	$R_1=0.2614$, $wR_2=0.1876$ [a]	$R_1=0.1457$, $wR_2=0.1445$ [b]
Data/restraints/parameters	2263/0/154	4464/0/298
Refinement method	Full-matrix least-squares on F^2	Full-matrix least-squares on F^2
Goodness-of-fit	0.993	1.065
Min. and max. residual electron density (e·Å^{-3})	−0.451 and 0.383	−0.380 and 0.386
Hydrogen atoms	Riding	Riding

[a] Weighting scheme: $w^{-1}=\sigma^2(F_o^2)+(0.0735P)^2$ where $P=(F_o^2+2F_c^2)/3$

[b] Weighting scheme: $w^{-1}=\sigma^2(F)+0.0008F^2$

carried out in dry nitrogen atmosphere and absolute grade solvents.

3.1. *3-Alkyl-3-ferrocenylcyclopropenes (**10** and **11**)*

Cyclopropenes **10** and **11** were prepared as reported earlier [8,9] by dehydrobromination of Z-/E-2-bromo-1-alkyl-1-ferrocenylcyclopropanes (**12a** and **b**) by ^{t}BuOK in DMSO.

3-Ferrocenyl-3-methylcyclopropene (**10**), orange oil, yield 44%. ^{1}H-NMR, δ: 1.46 (3H, s), 4.01 (4H, m, C_5H_4), 4.15 (5H, s, C_5H_5), 7.15 (2H, s, CH=).

3-Ferrocenyl-3-isopropylcyclopropene (**11**), orange crystals, yield 61%, m. p. 62°C. ^{1}H-NMR, δ: 0.79 (6H, d, $J=6.76$ Hz), 2.41 (1H, m, $J=6.76$ Hz), 4.02 (4H, m, C_5H_4), 4.12 (5H, s, C_5H_5), 6.95 (2H, s, CH=).

3.2. *Sensitized photolysis of cyclopropenes **10** and **11***

A solution of 0.24 g or 0.27 g (1 mmol) of cyclopropenes **10** or **11** in 100 ml of acetonitrile (distilled from P_2O_5) with 5 mg benzophenone was irradiated for 5 h in a Pyrex container using a 200 W Hanovia medium pressure lamp. After evaporation of the acetonitrile the residue was chromatographed on Al_2O_3 (hexane as the eluent). Compounds **13** and **14** being obtained:

3.2.1. *Compound **13***

0.18 g (73%) of the 3,6-diferrocenyl-3,6-dimethyltricyclo[3.1.0.0^{2,4}]hexane (**13**), orange crystals, m.p. 131–132°C. ^{1}H-NMR, δ: 1.64 (4H, s, CH), 1.66 (6H, s, CH_3), 4.01 (4H, m, C_5H_4), 4.03 (4H m, C_5H_4), 4.15 (10H, s, 2C_5H_5). Anal. Found: C, 70.53; H, 5.89; Fe, 23.56. $C_{28}H_{28}Fe_2$; Calc.: C, 70.62; H, 5.93; Fe, 23.44%.

3.2.2. *Compound **14***

0.19 g (70%) 3,6-diferrocenyl-3,6-diisopropyltricyclo[3.1.0.0^{2,4}]hexane (**14**), orange crystals, m.p. 157–158 °C. ^{1}H-NMR, δ: 0.85 (12H, d, $J=6.8$ Hz), 1.58 (4H, s, CH), 2.64 (2H, m, $J=6.8$ Hz), 3.96 (4H, m, C_5H_4), 4.03 (4H m, C_5H_4), 4.13 (10H, s, 2C_5H_5). Anal. Found: C, 72.16; H, 6.73; Fe, 21.07. $C_{32}H_{36}Fe_2$; Calc.: C, 72.21; H, 6.81; Fe, 20.98%.

3.3. *Direct photolysis of 3-ferrocenyl-3-methylcyclopropene (**10**)*

In a typical experiment 0.24 g (1 mmol) of the cyclopropene **10** was dissolved in 100 ml of benzene and irradiated for 4 h in a nitrogen atmosphere with a 200 W Hanovia medium pressure lamp and Vycor filter. Following the removal of the solvent, the residue chromatographed on a plate with neutral alumina (Brockmann activity III) in hexane to give compounds **17** and **18**.

3.3.1. *Compound **17***

3-Ferrocenyl-3-methylidenepropyne (**17**), R_f 0.72, orange oil, yield 0.065 g (23%).^{1}H-NMR, δ: 3.02 (1H, s, CH=), 4.24 (2H, m, C_5H_4), 4.51 (2H, m, C_5H_4), 4.16 (5H, s, C_5H_5), 5.47 (1H, d, $J=0.9$ Hz, CH_2=), 5.60 (1H, d, $J=0.9$ Hz, CH_2=). ^{13}C-NMR, δ: 66.67, 69.09 (C_5H_4), 69.65 (C_5H_5), 76.51 (CH=), 82.93 (C_{ipso}Fc), 89.97 (C=), 118.15 (CH_2=), 128.42 (C). Anal. Found: C, 71.38; H, 5.03; Fe, 23.75. $C_{14}H_{12}Fe$; Calc.: C, 71.22; H, 5.12; Fe, 23.66%.

3.3.2. *Compound **18***

Trans-2-Ferrocenyl-2-butene **18** [24], R_f 0.65, orange oil, yield 0.076 g (30%). ^{1}H-NMR, δ: 1.44 (3H, d, $J=6.67$ Hz), 1.78 (3H, s), 4.01 (2H, m, C_5H_4), 4.23 (2H, m, C_5H_4), 4.06 (5H, s, C_5H_5), 5.52 (1H, q, $J=6.67$

Hz, CH=). Anal. Found: C, 69.87; H, 6.93; Fe, 23.41. $C_{14}H_{16}Fe$; Calc.: C, 70.02; H, 6.72; Fe, 23.26%.

*3.4. Direct photolysis of 3-ferrocenyl-3-isopropylcyclopropene (**11**)*

Analogously, compound **11** (1 mmol, 0.27 g) in 150 ml of benzene was irradiated for 3.5 h.The solvent was distilled in vacuo and the residue was chromatographed on Al_2O_3 (hexane as the eluent) to give 1-ferrocenyl-4,4-dimethylcyclobutene (**19**), orange oil, yield 0.07 g (27%). ^{1}H-NMR, δ: 1.83 (3H, s, CH_3), 2.07 (3H, s, CH_3), 2.93 (2H, d, $J = 7.8$ Hz, CH_2), 4.15 (2H, m, C_5H_4), 4.19 (2H, m, C_5H_4), 4.10 (5H, s, C_5H_5), 5.81 (1H, t, $J = 7.8$ Hz, CH=). Anal. Found: C, 72.09; H, 7.03; Fe, 20.85. $C_{16}H_{18}Fe$; Calc.: C, 72.21; H, 6.81; Fe, 20.98%.

*3.5. Direct photolysis of cyclopropene **11** with 2-ferrocenyl-3-methyl-1-butene (**23**)*

Analogously, a solution of cyclopropene **11** (1 mmol, 0.27 g) and alkene **23** (1 mmol, 0.25 g) in 200 ml of benzene was irradiated for 5 h. The solvent was distilled in vacuo and the residue was chromatographed on Al_2O_3 (hexane) to give 1,3-diferrocenyl-6,6-dimethyl-3-isopropylcyclohexene (**24**), orange crystals, yield 0.43 g (83%), m.p. 164-165°C. ^{1}H-NMR, δ: 0.67 (3H, d, $J =$ 6.8 Hz), 0.80 (3H, d, $J = 6.8$ Hz), 0.97 (3H, s, CH_3), 1.07 (3H, s, CH_3), 1.65 (2H, dd, $J = 5.2$, 7.1 Hz, CH_2), 1.88 (2H,dd, $J = 7.1$, 12.2 Hz, CH_2), 1.98 (1H, m, CH), 4.04 (1H, m, C_5H_4), 4.11 (1H, m, C_5H_4), 4.16 (2H, m, C_5H_4), 4.18 (2H, m, C_5H_4), 4.21 (1H, m, C_5H_4), 4.24 (1H, m, C_5H_4), 4.19 (5H, s, C_5H_5), 4.27 (5H, s, C_5H_5), 6.48 (1H, s, CH=). Anal. Found: C, 71.43; H, 7.06; Fe, 21.51. $C_{31}H_{36}Fe_2$; Calc.: C, 71.56; H, 6.97; Fe, 21.47%.

4. Supplementary material

Crystallographic data for the structural analysis have been deposited with the Cambridge Crystallographic Data Centre, CCDC nos. 140679 and 140680 for compounds **14** and **24**, respectively. Copies of this information may be obtained free of charge from The Director, CCDC, 12 Union Road, Cambridge CB2 1EZ (fax: +44-1223-336033; e-mail: deposit@ccdc.cam.ac.uk or www: http://www.ccdc.cam.ac.uk).

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