0.0498 ($I > 2\sigma(I)$), $wR_2 = 0.1073$ ($I > 2\sigma(I)$) (GOF = $[\Sigma[w(F_o^2 - F_c^2)^2]/(n-p)]^{1/2}$, where n and p denote the number of data and parameters; $R_1 = (\Sigma||F_o| - |F_c||/\Sigma|F_o|$; $wR_2 = [\Sigma[w(F_o^2 - F_c^2)^2]/\Sigma[w(F_o^2)^2]]^{1/2}$ with $w = 1/[\sigma^2(F_o^2) + (a \cdot P)^2 + b \cdot P]$ and $P = [\max(F_o^2, 0) + 2(F_c^2)/3]$).

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[2+1] Cycloadditions of Diazoalkanes to Enol Ethers Catalyzed by Chromium Complexes—The First Direct Spectroscopic Observation of a Carbene Complex Intermediate**

Jürgen Pfeiffer and Karl Heinz Dötz*

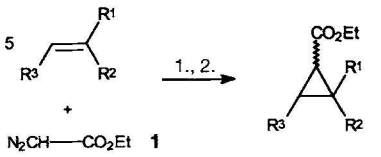
Dedicated to Professor Dieter Seebach on the occasion of his 60th birthday

Reactions of aliphatic diazo compounds with alkenes catalyzed by transition metal complexes are among the most important methods for obtaining cyclopropanes.[1] The generally accepted mechanism involves the formation of a reactive carbene complex intermediate, resulting from electrophilic attack at the carbon atom of the diazo group and subsequent elimination of N_2. Transfer of the carbene fragment to the alkene regenerates the catalytically active species and the catalytic cycle starts again. Although a carbene complex intermediate has not yet been observed directly, convincing experimental results support this model:

1. The high asymmetric induction observed when chiral metal complexes are used requires strong steric interactions between alkene, metal complex, and carbene during the face-selective reaction step.[2]
2. Reactivity/selectivity correlations between reactions catalyzed by $[Rh_2(OAc)_4]$ and stoichiometric reactions with $[(CO)_5W{=}C(H)Ph]$ indicate a similar mechanism for the two types of reactions.[3]
3. The synthesis of stable carbene complexes from diazoalkanes[4,5] as well as that of cyclopropanes from carbene complexes support the mechanism outlined above.[1a,6] Furthermore, a chiral ruthenium complex that catalyzes the [2+1] cycloaddition of ethyl diazoacetate and styrene in high enantiomeric excess was obtained recently; the corresponding carbene complex, which also catalyzes the reaction, is isolable in the absence of styrene.[7]

The ability of chromium(0) complexes to catalyze [2+1] cycloadditions was hitherto studied in less detail.[8] During our research aimed at the synthesis of stable chromium carbene complexes from diazoalkanes and chromium complexes of the type $[(CO)_5CrL]$ (L = THF, *cis*-cyclooctene), we became interested in the utiliziation of these compounds for catalytic cyclopropanations of alkenes with diazoalkanes.

Reactions of ethyl diazoacetate **1** with electron-rich alkenes (5 equivalents) in the presence of pentacarbonyl(η^2-*cis*-cyclooctene)chromium(0) (**2**) (5 mol%; Scheme 1) afford the donor–acceptor substituted cyclopropanes **3**–**5** in good



Scheme 1. Synthesis of the cyclopropanes **3**–**6**. 1: **2** (5 mol%), 5 °C, 4 h, CH_2Cl_2, $-N_2$, $-C_8H_{14}$; 2: 20 °C, 8 h, CH_2Cl_2; 28–79%.

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yields (Table 1).[10] Compound **6**, which is formed by the reaction with styrene, could only be obtained in poor yield, whereas alkyl- and acceptor-substituted alkenes gave no [2+1] cycloaddition products under these conditions.[11] Attempts to trap or provide evidence for a carbene complex

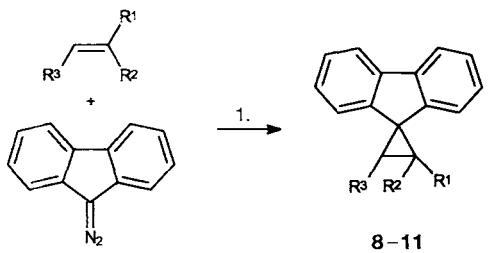
Table 1. Chromium-catalyzed cyclopropanations of electron-rich alkenes with ethyl diazoacetate **1** [a,b].

Product	R^1	R^2	R^3	Yield [%]	*cis/trans* [c]
3	OC_2H_5	H	H	79	1:1.8
4	OCH_3	CH_3	H	77	1:2.1
5	H	$-O-CH_2-CH_2-$		72	1:4.0 [d]
6	C_6H_5	H	H	28	1:2.0

[a] Reaction at 5 °C for 4 h in dichloromethane. [b] Elemental analyses and/or high-resolution mass spectra were obtained for **3–6**; the identification and assignment of the isomers were performed by comparison with data from the literature [12]. [c] Assigned by ^{1}H NMR spectroscopy and GC-MS. [d] *endo/exo*.

intermediate during these reactions have not been successful.

Reactions of equimolar amounts of 9-(9*H*)-diazofluorene (**7**) with enol ethers in the presence of **2** (2 mol%) also afford the corresponding spirocyclopropanes **8**[13]–**11** in good to very good yields (Scheme 2, Table 2).[14] Again, no [2+1] cycloaddition was observed with alkyl-substituted alkenes.[11] A control experiment with ethyl acrylate in the absence of **2** indicates that **12** is formed by an uncatalyzed [2+1] cycloaddition.[15] All catalyzed reactions occur with darkening of the reaction mixture; thin-layer chromatography (TLC) shows the typical violet spot for pentacarbonyl[9-(9*H*)-fluorenylidene]chromium(0) (**13**), which, in the absence of alkenes, is isolable from stoichiometric reactions of **2** with **7**.[9]



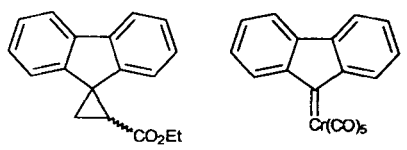
7 8–11

Scheme 2. Synthesis of the cyclopropanes **8–11**. 1: **2** (2 mol%), 20 °C, 16 h, CH_2Cl_2, $-N_2$, $-C_8H_{14}$; 25–93%.

Table 2. Chromium-catalyzed reactions of 9-(9*H*)-diazofluorene **7** with electron-rich alkenes [a].

Product	R^1	R^2	R^3	Yield [%]	Workup	Eluent [b]
8	OC_2H_5	H	H	93	A	PE/CH_2Cl_2 2:3
9	OCH_3	CH_3	H	87	A	PE/CH_2Cl_2 1:1
10	H	$-O-CH_2-CH_2-$		73	A	PE/CH_2Cl_2 1:2
11	C_6H_5	H	H	25	B	PE/Et_2O 5:1

[a] Reaction at 20 °C for 8 h in dichloromethane. [b] PE = petroleum ether (40/60).



12 13

Direct evidence for **13** during the reaction of **7** with ethyl vinyl ether in the presence of **2** (5 mol%) by ^{13}C NMR spectroscopy is provided by "freezing" the reaction by cooling to −30 °C. In the range between $\delta = 200$ and 380, the ^{13}C NMR spectrum (Figure 1) exhibits the signals of **13** ($\delta =$ 217.58, 238.92, 361.32), **2** ($\delta = 216.04$, 224.31), and of hexacarbonylchromium(0). In addition, further signals are observed which could not be assigned.

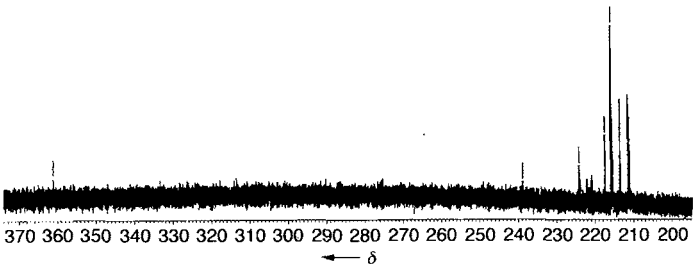
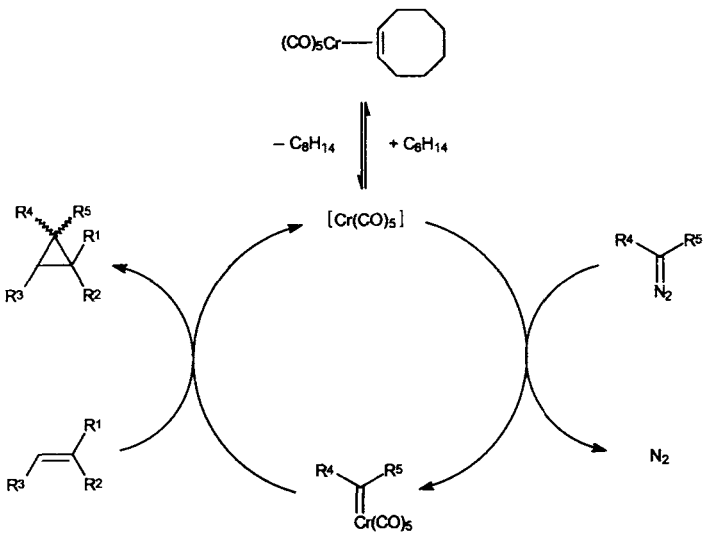


Figure 1. Section of the ^{13}C NMR spectrum (125.6 MHz, $CDCl_3$, 243 K) recorded during the reaction of **7** with ethyl vinyl ether catalyzed by **2**.

Scheme 3 presents a catalytic cycle that is consistent with the experimental results; however, the detailed mechanism of the carbene transfer from the carbene complex to the alkene remains unclear.



Scheme 3. Catalytic cycle of the chromium complex catalyzed [2+1] cycloadditions.

The chromium-catalyzed reactions of **1** and **7** with alkenes proceed with pronounced chemoselectivity with regard to the electronic properties of the olefin substituents. The preference of electron-rich C–C double bonds can be rationalized in terms of the electrophilic character of the postulated or detected intermediates $[(CO)_5Cr{=}C(H)CO_2Et]$ and **13**, respectively.

We have demonstrated that chromium(0) complexes can efficiently catalyze cyclopropanations. The presence of a weakly coordinated ligand such as *cis*-cyclooctene, which dissociates under mild conditions, generating the catalytically active species "$Cr(CO)_5$", is crucial. Furthermore, the observation of **13** by ^{13}C NMR spectroscopy represents the first

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direct evidence for a carbene complex intermediate during a [2+1] cycloaddition catalyzed by a transition metal complex. Further studies on the mechanism and the stereochemical course of the reactions are in progress.

Experimental Section

General procedures: All reactions were performed under argon. Solvents were dried and degassed according to standard procedures. Yields refer to products isolated after column chromatography. Compounds **1** [16], **2** [17], and **7** [18] were synthesized according to literature methods.

3–6: A solution of **1** (1.14 g, 10 mmol) in CH_2Cl_2 (20 mL) was added dropwise over 4 h to a stirred solution of **2** (0.15 g, 0.5 mmol, 5 mol%) in the corresponding alkene (50 mmol), precooled to 5 °C. The reaction mixture changed color from yellow to green-brown under evolution of N_2. After the mixture had been stirred for a further 8 h at 20 °C and the solvent and excess alkene had been removed under reduced pressure, the residue was purified by column chromatography (petroleum ether (40/60)/Et_2O 2:1).

8–11: A solution of **7** (0.57 g, 3 mmol) in CH_2Cl_2 (30 mL) was added dropwise over 8 h to a stirred solution of the corresponding alkene (3 mmol) and **2** (0.02 g, 0.06 mmol, 2 mol%) in CH_2Cl_2 (10 mL). The reaction mixture changed color from yellow to brown-violet under the evolution of N_2. After the mixture had been stirred for a further 8 h at 20 °C and the solvent had been removed under reduced pressure, workup was performed according to one of the following procedures (see Table 2).

A: Workup by column chromatography.

B: The residue was washed several times with portions of petroleum ether(40/60) (10 mL) and filtered until only bis[9-(9*H*)-fluorenylidene]azine could be detected in the solution (TLC-control, petroleum ether (40/60)/CH_2Cl_2 1:1, $R_f = 0.35$). The combined filtrates were concentrated, and purified by column chromatographic workup.

9: ^{1}H NMR (500 MHz, $CDCl_3$): $\delta = 7.89$ (d, 1H), 7.88 (d, 1H), 7.52 (d, 1H), 7.41 (t, 1H), 7.39 (t, 1H), 7.34 (t, 1H), 7.32 (t, 1H), 7.18 (d, 1H), 3.04 (s, 3H), 2.27 (dd, $^2J = 6.15$ Hz, $^4J = 0.55$ Hz, 1H; H-3), 1.91 (d, $^2J = 6.15$ Hz, 1H; H-3), 1.82 (d, $^4J = 0.49$ Hz, 3H; CH_3); ^{13}C NMR (125.6 MHz, $CDCl_3$): $\delta = 144.9$, 144.7, 141.1, 139.9 (4quart. C), 126.5 (CH), 126.0 (CH), 125.82 (CH), 125.78 (CH), 122.7 (CH), 121.5 (CH), 119.9 (CH), 119.5 (CH), 70.3 (s, 1C; C-2), 54.8 (q, 1C; OCH_3), 41.5 (s, 1C; C-1), 30.4 (t, $^1J = 160.8$ Hz, 1C; C-3), 16.8 (q, 1C, CH_3); IR (KBr): $\tilde{\nu}$(cm^{-1}) = 3059 (m), 2962 (m), 1473 (s), 1442 (s), 1240 (vs), 1065 (vs), 813 (s), 736 (vs); MS (EI, 70 eV): *m/z* (%): 236 (95) [M^+], 221 (100) [$M^+ - CH_3$], 205 (35) [$M^+ - OCH_3$], 178 (50) [$C_{14}H_{10}^+$], 165 (44) [$C_{13}H_9^+$], 152 (25) [$C_{12}H_8^+$]; elemental analysis calcd for $C_{17}H_{16}O$ (236.31): C 86.41, H 6.82; found: C 86.11, H 6.82.

10: ^{1}H NMR (500 MHz, $CDCl_3$): $\delta = 7.87$ (d, 1H), 7.75 (d, 1H), 7.40 (t, 1H), 7.32 (t, 2H), 7.25 (d, 1H), 7.24 (t, 1H), 6.80 (d, 1H), 4.65 (d, $^3J = 5.76$ Hz, 1H; H-1), 4.48–4.39 (m, 2H; H-3), 2.62 (ddd, $^3J = 1.76$, 5.68, 7.37 Hz, 1H; H-5), 2.48–2.31 (m, 2H; H-4); ^{13}C NMR (125.6 MHz, $CDCl_3$): $\delta = 145.9$, 141.6, 141.4, 138.3 (4quart. C), 126.8 (CH), 126.2 (CH), 125.9 (CH), 125.8 (CH), 123.0 (CH), 120.2 (CH), 119.5 (CH), 118.4 (CH), 77.0 (t, 1C; C-3), 71.6 (d, $^1J = 199.43$ Hz, 1C; C-1), 43.8 (s, 1C; C-6), 33.6 (d, $^1J = 173.3$ Hz, 1C; C-5), 25.6 (t, 1C; C-4); IR (KBr): $\tilde{\nu}$(cm^{-1}) = 3032 (m), 2968 (m), 1438 (vs), 1340 (vs), 1039 (s), 927 (s), 744 (vs); MS (EI, 70 eV): *m/z* (%): 234 (78) [M^+], 205 (85) [$M^+ - C_2H_5$], 178 (100) [$C_{14}H_{10}^+$], 165 (25) [$C_{13}H_9^+$]; elemental analysis calcd for $C_{17}H_{14}O$ (234.30): C 87.15, H 6.02; found: C 86.73, H 6.05.

11: ^{1}H NMR (400 MHz, $CDCl_3$): $\delta = 7.85$ (d, 1H), 7.80 (d, 1H), 7.41 (t, 1H), 7.36 (t, 1H), 7.30–7.19 (m, 7H), 6.92 (t, 1H), 6.15 (d, 1H), 3.38 (t, $^3J = 8.41$ Hz, 1H; H-2), 2.22 (d, $^3J = 8.41$ Hz, 2H; H-3); ^{13}C NMR (100.6 MHz, $CDCl_3$): $\delta = 148.2$, 144.2, 140.4, 139.6, 137.1 (5quart. C), 130.1 (2CH), 128.1 (2CH), 126.8 (CH), 126.7 (CH), 126.0 (CH), 125.8 (CH), 125.7 (CH), 121.5 (CH), 119.7 (CH), 119.6 (CH), 118.5 (CH), 35.5 (s, 1C; C-1), 34.9 (d, $^1J = 160.6$ Hz, 1C; C-2), 22.3 (t, $^1J = 162.3$ Hz, 1C; C-3); IR (KBr): $\tilde{\nu}$(cm^{-1}) = 3055 (m), 3034 (m), 1496 (m), 1444 (s), 777 (vs), 748 (vs), 696 (vs); MS (EI, 70 eV): *m/z* (%): 268 (100) [M^+], 252 (40) [$M^+ - CH_2$], 165 (25) [$C_{13}H_9^+$], 91 (17) [$C_7H_7^+$]; MS-HR calcd for $C_{21}H_{16}$: 268.1252; found: 268.1249.

Two control experiments in the absence of **2** and in the presence of $[Cr(CO)_6]$ (10 mol%), respectively, have been performed for each catalytic reaction under the reaction conditions described above. A [2+1] cycloaddition was observed in the absence of the chromium complexes [15] only in the reaction of ethyl acrylate with **7**.

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Creation of Enantioselective Biocatalysts for Organic Chemistry by In Vitro Evolution

Manfred T. Reetz,* Albin Zonta, Klaus Schimossek, Klaus Liebeton, and Karl-Erich Jaeger*

The development of chiral catalysts for the enantioselective synthesis of optically active compounds is of great academic[1] and industrial interest.[2] Inspite of worldwide intensive research in the area of homogeneous transition metal catalysis, the number of really efficient enantioselective catalysts is limited. Owing to a lack of general principles, the development of a single highly effective chiral catalyst requires laborious preparation and testing of many ligands. Alternatively, biocatalysts can be used, but by nature the problem of limited substrate specificity persists.[3] In some cases site-directed mutagenesis can be applied to improve enzyme activity and selectivity but not in a general way,

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