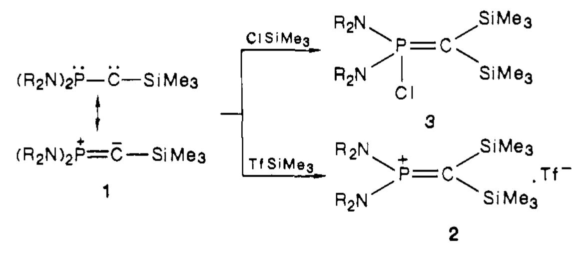


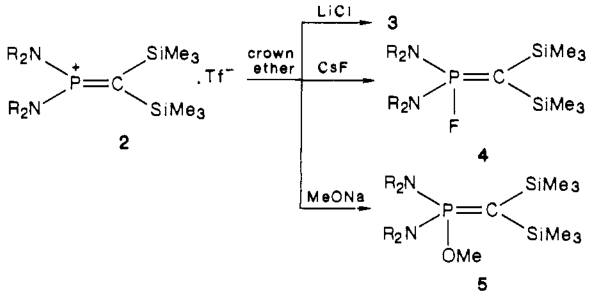
R = iPr; Tf = CF_3SO_3

In contrast to P-halogenated ylide **3**, derivative **2** is poorly soluble in nonpolar solvent. The ^{31}P chemical shift (+130.8 ppm) and the ^{13}C chemical shift of the tricoordinated carbon (+76.51 ppm, J_{PC} = 87.6 Hz), as expected for a cationic species, are significantly deshielded compared with those of related phosphorus ylides.[13] The ionic structure of **2** was unambiguously determined by an X-ray crystal structure analysis.[14] The thermal ellipsoid diagram of the molecule is shown in Figure 1 as well as the pertinent metric parameters.

Several aspects of the structure merit discussion. No interaction with the triflate is observed, demonstrating the ionic character of **2**. The positive charge is delocalized onto the nitrogen atoms, indicative of a PN partial double bond character, as shown by their planar geometry[15] and the short P–N bond lengths (1.61 vs 1.68 Å in H_2PNH_2).[16] The structural parameters for the framework atoms (P1 and C1) are as expected for a valence isoelectronic olefin: (i) the P–C distance of 1.62 Å is short, even compared to that in phosphaalkenes (1.64–1.69 Å),[1] and in good agreement with that from a theoretical study on the parent compound (1.624 Å);[10] (ii) the geometry at both P1 and C1 is strictly planar (±0.01 Å). However, the dihedral angle between these two planes corresponds to a twist of the double bond by 60°! This value is significantly larger than that reported by Sakurai (50.2°) for the "most crowded olefin".[17]

In order to rationalize the simultaneous existance of the short P–C bond and the large twist angle, a model developed by Trinquier[18] to describe the bonding in phosphorus ylides can be employed. This involves the interaction of a phosphenium ion with a p_π^2 closed-shell singlet excited carbene. The energy required for reaching the p_π^2 configuration from a n_σ^2 configuration is largely compensated by the building of the ylidic bond which is basically a dative bond (phosphenium lone pair → carbene empty n_σ) plus a partial back-donation (carbene occupied p_π → phosphenium empty d_π).

All attempts to involve the P–C double bond of **2** in reactions failed. However, in the presence of crown ether, substitution reactions occur with lithium chloride, cesium fluoride, and sodium methoxide, yielding the corresponding phosphorus ylides **3**–**5**.



R = iPr; Tf = CF_3SO_3

Acknowledgment. Thanks are due to Drs. G. Trinquier and G. Gillette for helpful discussions and to the CNRS (GRECO basses coordinences) for financial support of this work.

Supplementary Material Available: Tables of bond distances and angles with estimated standard deviations and atomic coordinates and thermal parameters (5 pages); listing of structure factors (10 pages). Ordering information is given on any current masthead page.

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[12] To an ether solution (10 mL) of carbene **1** (0.2 g, 0.6 mmol), a slight excess of trimethylsilyl triflate (0.12 mL, 0.7 mmol) was added dropwise at room temperature. Methylenephosphonium salt **2** precipitated from the solution as a yellow powder. After filtration, **2** was recrystallized at −10 °C from a dichloromethane/toluene solution. ^{31}P NMR (CD_2Cl_2) +130.8; ^{13}C NMR (CD_2Cl_2) 2.93 (d, $J(PC)$ = 7.1 Hz, $SiCH_3$), 24.55, 24.72 (s, CH_3CH), 48.88 (d, $J(PC)$ = 6.1 Hz, $CHCH_3$), 76.51 (d, $J(PC)$ = 87.6 Hz, P=C), 124.23 (q, $J(CF)$ = 321.1 Hz, CF_3SO_3); ^1H NMR (C_6D_6) 0.32 (d, $J(PH)$ = 0.6 Hz, 18 H, $SiCH_3$), 1.47 (d, $J(HH)$ = 6.9 Hz, 24 H, CH_3CH), 4.16 (sept d, $J(HH)$ = 6.9 Hz, $J(PH)$ = 12.8 Hz, 4 H, $CHCH_3$); ^{29}Si NMR (C_6D_6) −0.24 (s); ^{19}F NMR (C_6D_6) −3.37 (s).

[13] ^{31}P NMR: **3** +72.1; 4 +70.3; **5** +71.0 ppm. ^{13}C NMR (C=): **3** 12.35 (J_{PC} = 151.5 Hz); 4 7.25 (J_{PC} = 149.2 Hz); **5** 5.45 (J_{PC} = 135.6 Hz) ppm.

[14] A single crystal of **2** was grown from a dichloromethane/toluene solution, at −10 °C. **2** crystallizes in the monoclinic space group $P2_1/c$, a = 11.323 (6) Å, b = 9.432 (5) Å, c = 28.070 (20) Å; β = 90.67 (5)°; V = 2998 Å^3; Z = 4. The 3331 observed data ($I > 2\sigma I$) were collected by using Mo Kα radiation in the ω-scan mode and were used in the least-squares refinement to yield R = 0.050; R_w = 0.031 for 465 variables. The hydrogen atoms were refined isotropically in observed positions except for 4 methyl groups which were refined as rigid groups. Tables of bond lengths, bond angles, atomic coordinates, thermal parameters, and structure factors are given in the supplementary material.

[15] The geometry of the $(R_2N)_2P$ fragment is quite similar to that reported for the corresponding phosphenium ion: Cowley, A. H.; Cushner, M. C.; Szobota, J. S. *J. Am. Chem. Soc.* **1978**, *100*, 7784.

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Pericyclic Reactions of Cyclopropylcarbene-Chromium Complexes

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Recently, we reported that cyclopropylcarbene–chromium complexes react with alkynes to give highly substituted cyclopentenones.[1] Two mechanisms were proposed for this reaction, one of which involves ring expansion of the cyclopropylcarbene complex **1** to a chromacyclopentene (**2**) (Scheme I) as the initial step of the reaction. The penultimate product of the reaction is the cyclopentadienone **3**, which is reduced to the cyclopentenone **4** under the conditions of the reaction.[2] Rearrangement of a cyclopropylcarbene–transition metal complex to a metallacyclopentene is not precedented,[3] but this step can be viewed as an analogue of the vinylcyclopropane rearrangement.[4] We have studied the thermal chemistry of cyclopropylcarbene–chromium complexes[5] as a probe for the viability of this step. Herein we report our preliminary results of this study.

Carbene complex **1** was unchanged after 1 h when heated to reflux in benzene or p-dioxane. Since the reaction between carbene complex **1** and alkynes typically proceeded rapidly at 65 °C, the unreactivity of carbene complex **1** under the above conditions would seem to rule out the mechanism in Scheme I, provided that the conversion of **1** to **2** is not strongly influenced by the presence of alkyne ligands at chromium. In the all-carbon system, vinylcyclopropane rearrangements typically require a great deal of activation energy and usually proceed only at high temperatures.[4]

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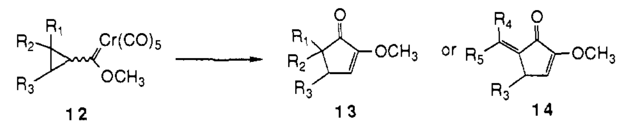
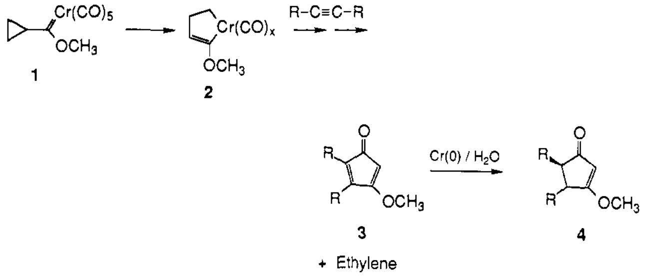
Table I. Thermal Reactions of Cyclopropylcarbene–Chromium Complexes



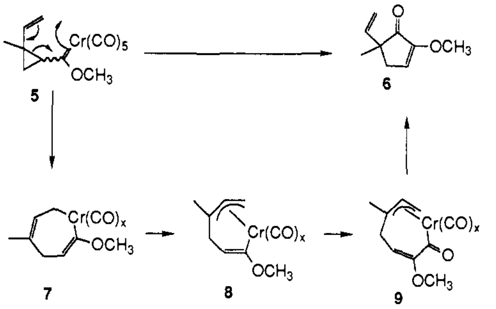
entry[a]	R_1	R_2	R_3	R_4	R_5	trans:cis complex[b]	reacn time/ temp[c,d]	yield of **13**,[e] %
A	vinyl	CH_3	H			42:58	3 h/101 °C	43
B	Ph	H	H			46:54	14 h/101 °C[f]	22
C	Ph	CH_3	H			68:32	14 h/101 °C[f]	27
D[k]	1-cyclopentenyl	H	H	$-(CH_2)_4-$			1 h/101 °C	69[i]
E	H	$-CH{=}CH(CH_2)_2-$				100:0	4.5 h/101 °C[f]	0
F	1-propenyl	H	CH_3	CH_2CH_3	H	53:47[g]	1 h/100 °C	53[g-i,l]
G	vinyl	H	H	CH_3	H	49:51	2.0 h/100 °C	49[i,j,m]
H	vinyl	$-(CH_2)_4-$				41:59	1.5 h/101 °C	67[n]
I	vinyl	$-(CH_2)_5-$				78:22	1.5 h/100 °C	57[h,n]

[a] Table entry letters define R_1, R_2, and R_3 for compounds **12–14**. [b] The trans:cis designation refers to the relative stereochemistry of the chromium carbene and the alkenyl (aryl) substituents in the starting carbene complex. [c] The reaction was performed in anhydrous *p*-dioxane unless otherwise noted. [d] All reactions were allowed to proceed until the carbene complex had completely disappeared unless otherwise noted. [e] The yields refer to products obtained after chromatographic purification. [f] The starting material had not disappeared when the reaction was stopped. [g] The relative stereochemistry of the methyl and propenyl groups is trans. [h] The reaction was conducted in DMF. [i] The yield refers to the product isolated after isomerization of the double bond (compound **14**). [j] The reaction was conducted in THF in a pressure bottle. [k] The starting carbene complex was stereoisomerically pure: a cis or trans assignment was not possible. [l] The *E*/*Z* ratio is 26:74. [m] The *E*/*Z* ratio is 36:64. [n] The stereochemistry of the ring fusion has been assigned as cis based on NOE results.

Scheme I



Scheme II



A related thermal reaction that also causes cyclopropane ring opening is the divinylcyclopropane rearrangement, which often proceeds at or below room temperature.[6] We therefore synthesized the vinylcyclopropylcarbene complex **5** as a 1:1 mixture of stereoisomers. Thermolysis of complex **5** in refluxing tetrahydrofuran for a period of 2 h led to formation of the 2-methoxy-2-cyclopentenone derivative **6** in 30% yield along with recovered starting material. The recovered starting material was now enriched in the isomer where the carbene complex is trans to the vinyl group. This indicates that the cis isomer converts to product faster than the trans isomer; the same order of reactivity of stereoisomers has been observed in the divinylcyclopropane rearrangement.[5] The first-order rate constant for disappearance of the cis isomer at 52 °C in THF was $(3.0 \pm 0.1) \times 10^{-5}\ s^{-1}$ ($\Delta G^{\ddagger} = 25.8$ kcal/mol), while that for the trans isomer was $(8.3 \pm 0.6) \times 10^{-6}\ s^{-1}$ ($\Delta G^{\ddagger} = 26.6$ kcal/mol). We propose the mechanism in Scheme II to explain the formation of cyclopentenone **6**. This mechanism involves a divinylcyclopropane rearrangement, possibly after complexation of chromium with the double bond,[7] as the ring-opening step.[8] The initially formed metallacycloheptadiene complex **7** then rearranges to the π-allyl complex **8**.[9] CO insertion and reductive elimination[10] then occur to give cyclopentenone **6**. In DMF or *p*-dioxane at 100 °C, both the cis and trans isomers reacted to completion. Cyclopentenone **6** was obtained in 68% yield when DMF was used as the solvent. Under the more vigorous conditions, cis–trans isomerization of the carbene complex can occur. The isomerization was facilitated by the slightly basic nature of DMF since protons adjacent to carbene complexes are typically highly acidic.[11] If the reaction was conducted in 99:1 DMF–D_2O, then compound **6** was obtained completely deuterated in the 3-position.

An alternate explanation for the enhanced reactivity of complex **5** is that the vinyl substituent weakens the carbon–carbon bond of the cyclopropane ring,[12] making the mechanism in Scheme I more likely. It is not clear how the relative stereochemistry of the vinyl and carbene complex substituents would affect the reaction if it proceeded by this mechanism. As a further test for the Cope rearrangement mechanism, we studied thermolysis reactions of the phenyl-substituted carbene complex **10** (Scheme III). The phenyl substituent should be similar to vinyl in its ability to activate the carbon–carbon bond for a cleavage reaction[12] but should be much poorer in its ability to participate in the Cope rearrangement.[13] Carbene complex **10** is unreactive when heated to 95 °C for a period of 6 h, but it does rearrange very slowly at 100 °C. Other γ,δ-unsaturated carbene complexes have been prepared, but usually do not undergo a Cope rearrangement.[14]

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(6) Schneider, M. P.; Rau, A. *J. Am. Chem. Soc.* **1979**, *101*, 4426–4427.

(7) The reaction is inhibited by external CO pressure, suggesting that CO dissociation and perhaps alkene complexation are important steps.

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(10) Reductive elimination from these types of complexes typically gives five-rather than seven-membered rings. For an example, see: Trost, B. M.; Walchli, R. A. *J. Am. Chem. Soc.* **1987**, *109*, 3487–3489.

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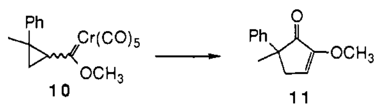
Scheme III



In one case, a reaction reminiscent of the Claisen rearrangement has been observed.[14c] Presumably, ring opening of the cyclopropane ring provides the necessary driving force to induce the Cope-like process is these systems.

We have explored the scope and limitations of this rearrangement with a variety of 2-alkenylcyclopropylcarbene–chromium complexes; the results are summarized in Table I. The reaction appears to be general for a wide variety of these types of complexes. Only in one case (entry E) does the reaction fail. Bicyclic carbene complex **12E** fails to rearrange, presumably because rearrangement requires the alkene and carbene complex functionalities to attain the cis relative configuration. This configuration is hard to attain because of steric interactions between the cyclohexyl ring and the carbene complex. In cases where R_2 = H, the reaction mixtures also contain some of the alkene isomerization product as an impurity.

In summary, we have explored rearrangements of cyclopropylcarbene–chromium complexes. These rearrangements reveal interesting similarities with classical electrocyclic reactions, and they may be one of the first examples where a dπ–pπ bond participates in a Cope rearrangement. We are continuing to explore the mechanism, the scope, and the limitations of these novel rearrangement reactions.

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Supplementary Material Available: Spectra and procedures for the synthesis of the carbene complexes and for the reactions in Table I (28 pages). Ordering information is given on any current masthead page.

Heptadecanuclear Mixed Metal Iron Oxo–Hydroxo Complexes, $[Fe_{16}MO_{10}(OH)_{10}(O_2CPh)_{20}]$, M = Mn or Co, Structurally Comprised of Two Fragments Derived from $[Fe_{11}O_6(OH)_6(O_2CPh)_{15}]$

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Recent interest in polynuclear oxo–hydroxo complexes of iron and manganese has been stimulated by their occurrence in biology, prominent examples including ferritin[1] and photosystem II.[2] The hydrolytic polymerization of iron[3] is of particular importance in biomineralization[4] and corrosion.[5] Because of the difficulty in controlling hydrolytic chemistry above pH 1–2, examples of discrete soluble polyiron or -manganese oxo–hydroxo complexes are rare.[6] Structurally characterized species include M_3,[7] M_4,[8] M_6 (M = Fe, Mn),[9] Fe_8,[10] Mn_9,[11] Mn_{10},[12] Fe_{11},[13] and Mn_{12}[14] complexes, most of which were prepared since 1984. Despite this progress, there remains a need to understand the synthetic pathways by which such molecular aggregates form and to elu-

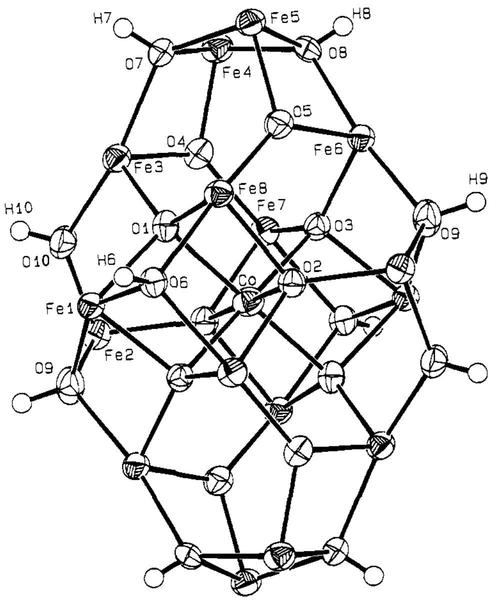

Figure 1. ORTEP drawing of the metal oxo–hydroxo core in **2**, showing the 50% probability thermal ellipsoids and atom labels for the metal and oxygen atoms. Hydrogens are presented as small spheres for clarity.

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