were employed between 80 and 120 °C, and solutions containing DTBP were employed between 130 and 160 °C. The relative rate of β-scission to reduction was determined by using the integrated rate equation (eq 19). The Arrhenius parameters for trans-β-scission were calculated from linear least-squares regression analysis of ln $(k_{\text{trans}}/k_{\text{abs}})$ vs $1/RT$ as depicted in Figure 2. The Arrhenius parameters for cis-β-scission were calculated from ratios of *trans*- to *cis*-β-methylstyrene and the estimated ratio $A_{\text{trans}}/A_{\text{cis}} = 1.17$ (see text), yielding the relative rate expression log $(k_{\beta,\text{trans}}/k_{\beta,\text{cis}}) = 0.04 - 2.4/\theta$. The expression was combined with the absolute rate expression for trans-β-scission to yield log $(k_{\beta,\text{cis}}/\text{s}^{-1}) = 13.41 - 19.3/\theta$.

Regioselectivity and Solvent Effects in Cyclopropanation of Alkadienes

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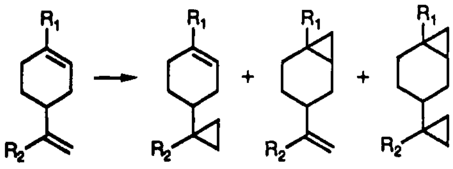
A comparison of the regioselectivities for monocyclopropanation of limonene and 4-vinylcyclohexene using diiodomethane with zinc dust and copper(I) chloride in ether, with diethylzinc in toluene, or with triethylaluminum in toluene–dichloromethane has been carried out. Some studies were also done using dibromomethane with zinc dust and copper(I) chloride in ether. The procedure using diethylzinc in toluene exhibited the least steric hindrance toward cyclopropanation at more highly substituted, electron-rich double bonds. In a study of solvent effects on the diiodomethane, zinc dust, and copper(I) chloride procedure, use of diisopropyl ether as a reaction solvent proved to be a satisfactory method for almost doubling the steric requirements of the cyclopropanating reagent.

In connection with other studies in our laboratory, it became of interest to carry out an investigation of regioselectivities in monocyclopropanation of dienes using organometallic carbenoids generated from diiodomethane or dibromomethane by various methodologies. The methodologies, which were of special interest to us because of their operational simplicity, low cost, and adaptability to larger scale reactions, were those using zinc dust and copper(I) chloride for in situ generation of a zinc–copper couple,[1] diethylzinc[2] and triethylaluminum.[3]

Some studies[4,5] of intermolecular alkene competitions reported for the diiodomethane and zinc–copper couple cyclopropanating system have revealed that the organozinc intermediate is electrophilic but rather sterically hindered. In the few cases of intramolecular competitions using this methodology that have appeared,[6-8] the results are conflicting. With the diiodomethane–diethylzinc system, no intramolecular alkene cyclopropanation competitions have been reported. However, some intermolecular competition studies[9] have suggested that the diethylzinc system, especially in noncoordinating solvents, gives a much less sterically hindered cyclopropanating intermediate than that formed with the zinc–copper couple in ether. Finally, with the only recently reported triethylaluminum method,[3] neither intra- nor intermolecular competition was reported with hydrocarbon dienes. However, with several dienols, cyclopropanation using diiodomethane and triethylaluminum in toluene–dichloromethane took place exclusively at the olefinic site distant from the hydroxyl group. The opposite behavior is observed in cyclopropanation using diiodomethane and diethylzinc in ether.

Results and Discussion

For our initial studies, which are reported in Table I, we investigated the regioselectivities of all of the cyclopropanating systems of interest toward limonene (**1a**).



1a, $R_1 = R_2 = CH_3$
b, $R_1 = R_2 = H$
2
3
4

The solvent used in each case was that usually associated with the method. Two different mole ratios of reactants were examined in each case. For the zinc dust–copper(I) chloride procedure, we used the acetyl chloride promotion method recently developed in our laboratory[10] to ensure rapid, high-yield reactions. The use of dibromomethane was examined only when the zinc dust–copper(I) chloride procedure was employed because, with diethylzinc or triethylaluminum, dibromomethane has not been reported to react.

It is immediately seen that the diiodomethane–diethylzinc system in toluene exhibits the lowest regioselectivity in monocyclopropanation of limonene, presumably due to use of the noncoordinating toluene solvent. With limonene, the trisubstituted ring double bond is the most electron rich but also the most sterically hindered. Thus, lower regioselectivity in the direction shown in the limonene system should be associated with a cyclopropanating reagent having lower steric requirements. The observation that with use of zinc dust and copper(I) chloride the cyclopropanations involving diiodomethane and dibromomethane in ether exhibited such closely similar regioselectivities toward the double bonds of limonene was unexpected. Also, it is seen that the regioselectivity for cyclopropanation for the triethylaluminum with diiodomethane in toluene–dichloromethane procedure lies between those for the zinc dust–copper(I) chloride and diethylzinc methods.

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Table I. Regioselectivity of Cyclopropanation of Limonene (1a) under Various Conditions[a]

			composition of volatiles, mol %				
conditions	mole ratio diene:CH_2X_2:Zn	recovery of volatiles,[b] %	diene **1a**	exocyclic CP, **2a**	ring CP, **3a**[c]	$(CP)_2$, **4a**[c]	mole ratio **2a**:**3a**
CH_2I_2–Zn, CuCl, AcCl, in diethyl ether	1:2:3	82	10	36	10	44	3.6
	1:1:1.5	84	46	31	13	10	2.4
CH_2Br_2–Zn, CuCl, AcCl, in diethyl ether	1:2:3	74	22	37	12	29	3.0
	1:1:1.5	79	60	25	10	5	2.5
CH_2I_2–Et_2Zn, in toluene	1:1:1	100	39	19	25	17	0.76
	1:1:0.5	78	39	22	22	17	1.0
CH_2I_2–Et_3Al, in toluene–CH_2Cl_2	1:1:1	*d*	46	26	13	12	2.0
	1:1:0.5	*d*	55	23	13	7	1.8

[a] The specific conditions are given in the Experimental Section. [b] Based on distilled weight and GLC analysis. [c] The ratios of the cis and trans stereoisomers were approximately 1:1.1. [d] Could not be determined due to the presence of diiodomethane in the distillate.

Table II. Regioselectivity of Cyclopropanation of 4-Vinylcyclohexene (1b) under Various Conditions[a]

			composition of volatiles, mol %				
conditions	mole ratio diene:CH_2X_2:Zn	recovery of volatiles,[b] %	diene **1b**	exocyclic CP, **2b**	ring CP, **3b**[c]	$(CP)_2$, **4b**[c]	mole ratio **2b**:**3b**
CH_2I_2–Zn, CuCl, AcCl, in diethyl ether	1:1:1.5	72	61	13	25	1	0.52
CH_2Br_2–Zn, CuCl, AcCl, in diethyl ether	1:1:1.5	60	89	4	7	0	0.57
CH_2I_2–Et_2Zn, in toluene	1:1:0.5	76	47	7	45	1	0.16
CH_2I_2–Et_3Al, in toluene–CH_2Cl_2	1:1:0.5	*d*	40	13	39	8	0.33

[a] The specific reaction conditions are indicated in the Experimental Section. [b] Based on distilled weight and GLC analysis. [c] The cis and trans stereoisomers were not separated. [d] Could not be determined due to the presence of CH_2I_2.

The effects of changing reactant mole ratios upon the cyclopropanation regioselectivities for the various systems are of some interest. In the case of the reactions of diiodomethane or dibromomethane with zinc dust and copper(I) chloride, the reason for the apparent higher selectivities when 1:2:3 mole ratios of limonene to dihalomethane to zinc are used is related to the formation of considerable amounts of dicyclopropanated material **4a**, especially with the diiodomethane. Thus, on further cyclopropanation of the initially formed mixture of monocyclopropanes, the isomer **3a** having the 1-methylethenyl group reacts faster than **2a** having the more sterically hindered ring trisubstituted double bond, thereby amplifying the ratio of the remaining monocyclopropanes. In the cases of the diethylzinc and triethylaluminum reactions, there is also a change in regioselectivity associated with changing the reactant mole ratios. However, an explanation for the behavior in these cases is not readily forthcoming.

To provide confirmation for the conclusions derived from the studies with limonene, we also carried out the corresponding studies on 4-vinylcyclohexene (**1b**) reported in Table II. Because both double bonds of 4-vinylcyclohexene are less sterically hindered than those of limonene, it was anticipated that **1b** would be preferentially cyclopropanated at the more electron-rich, disubstituted ring double bond in all cases. This was borne out by the results. Also, in accord with the limonene results, the diethylzinc–diiodomethane reaction in toluene exhibited the least steric hindrance toward cyclopropanation of the disubstituted ring double bond, while the zinc–copper(I) chloride procedure showed the most.

From the data given in Tables I and II, it is apparent that if one were to desire high-yield preparations of two regioisomeric monocyclopropanated dienes, the diiodomethane with diethylzinc in toluene and the diiodomethane with zinc dust and copper(I) chloride procedures would be the methods of choice among those studied thus far to give the widest range of regioselectivities. However, for efficient syntheses, a larger range would be desirable. Thus, since solvent changes can often have profound effects on the steric requirements of organometallic reagents, we considered this for the present investigation as a possible way to extend the range of regioselectivities available for diene cyclopropanations.

Some studies of solvent effects on relative rates of intermolecular alkene cyclopropanations had already been reported for the diethylzinc–diiodomethane system.[9] However, from these studies it appeared that the only way to achieve a sterically less demanding diethylzinc–diiodomethane reagent than that already studied in toluene would be to switch to an alkane solvent, although the effect should only be slight. Thus, we turned our attention to the possible use of solvent effects to increase the steric requirements of the diiodomethane–zinc copper couple derived cyclopropanating reagent.

One problem with proposals to change to solvents other than ether for alkene cyclopropanations using diiodomethane and a zinc–copper couple prepared either in situ from zinc dust and copper(I) chloride or in a prior step was that prior attempts to do this had usually proven unsuccessful.[11] However, we hoped that this problem might be overcome by use of the acetyl chloride promotion procedure developed in our laboratory,[10] and our expectations were realized. The data in Table III for solvent effects in cyclopropanation of limonene (**1a**) point to several solvents, diisopropyl ether and 1,2-dimethoxyethane, in which the regioselectivity for cyclopropanation is significantly greater toward the less sterically hindered 1-methylethenyl group than in ether. The only problem with use of these solvents is the somewhat lower conversions of diene.

To confirm the results seen with limonene (**1a**), diisopropyl ether was chosen as the solvent for further studies. This is because it is relatively low boiling, inexpensive, and available as an anhydrous reagent. Thus, an acetyl chloride promoted cyclopropanation was carried out on 4-vinylcyclohexene using diiodomethane and zinc dust–copper(I) chloride on a 1:1:1.5 mole ratio in diisopropyl ether. Also, because of the problem of low alkene conversions based on starting diiodomethane, cyclopropanations were carried out in diisopropyl ether for both limonene (**1a**) and 4-vinylcyclohexene (**1b**) with diidomethane and zinc dust on 1:2:3 mole ratios. The results are given in Table IV and

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Table III. Regioselectivity of Acetyl Chloride Promoted Cyclopropanation of Limonene (1a) with Diiodomethane, Zinc Dust, and Copper(I) Chloride in Various Solvents[a]

				composition of volatiles, mol %				
solvent	temp, °C	time, h	recovery of volatiles,[b] %	diene **1a**	exocyclic CP, **2a**	ring CP, **3a**[c]	$(CP)_2$, **4a**[c]	mole ratio **2a**:**3a**
diethyl ether	48–50	6	84	46	31	13	10	2.4
1,2-dimethoxyethane	50	8	86	55	30	10	5	3.0
tetrahydrofuran	55–60	23	100	76	16	7	1	2.2
diisopropyl ether	55–60	10	86	60	29	9	2	3.2
ethyl acetate	48–50	48	97	77	14	8	1	1.8
dioxane	60–70	24	81	80	13	6	1	2.2
1,2-dichloroethane	55–70	24	23	22	40	8	18	5.0

[a] Run on 0.1-mol diene scale with a 1:1:1.5 mol ratio of limonene–diiodomethane–zinc dust in about 75–80 mL of solvent with 10 mol % copper(I) chloride based on zinc and 2.5 mol % acetyl chloride based on diiodomethane. [b] Based on distilled weight and GLC analysis. [c] The ratios of cis and trans stereoisomers were approximately 1:1.2.

Table IV. Regiochemistry of Acetyl Chloride Promoted Cyclopropanation of Limonene (1a) and 4-Vinylcyclohexene (1b) with Diiodomethane, Zinc Dust, and Copper(I) Chloride in Diethyl Ether or Diisopropyl Ether[a]

				composition of volatiles, mol %				
diene	solvent	mole ratio diene:CH_2I_2:Zn	recovery of volatiles,[b] %	diene **1**	exocyclic CP, **2**	ring CP, **3**	$(CP)_2$, **4**	mole ratio **2**:**3**
limonene (**1a**)	diethyl ether	1:1:1.5	84	46	31	13	10	2.4
	diisopropyl ether	1:1:1.5	86	60	29	9	2	3.2
		1:2:3	69	40	40	9	11	4.4
4-vinylcyclohexene (**1b**)	diethyl ether	1:1:1.5	72	61	13	25	1	0.52
	diisopropyl ether	1:1:1.5	68	70	15	14	1	1.1
		1:2:3	54	41	30	18	11	1.7

[a] Run on 0.1-mol diene scale in 75–80 mL of solvent with 10 mol % copper(I) chloride based on zinc and 2.5 mol % acetyl chloride based on diiodomethane. [b] Determined by distillation and GLC analysis.

compared with those obtained in diethyl ether solvent. It is seen that the steric effect enhancing results seen in diisopropyl ether with limonene are confirmed with 4-vinylcyclohexene. Also, it was gratifying to observe with both dienes in diisopropyl ether that use of the higher mole ratios of diiodomethane to diene not only resulted in better diene conversions but also achieved higher regioselectivities. This is a result of the ring monocyclopropanated product being more reactive toward further cyclopropanation. One might thus anticipate that, with an even higher ratio of diiodomethane to diene, a further improvement in the ratio of monocyclopropanated products would be obtained, favoring that in which the least substituted double bond is cyclopropanated.

Experimental Section

Methods. ^1H NMR spectra were measured at 300 MHz with a GE QE-300 instrument. GC analyses were accomplished using a modified Varian Aerograph (Series 1400) instrument with hydrogen flame detector and equipped with a 30 m × 0.246 mm (i.d.) SE-54 J&W fused silica column. The limonene, 4-vinylcyclohexene, and other reagents were obtained commercially and in general were used without further purification.

General Procedure for Acetyl Chloride Promoted Cyclopropanations Using Diiodomethane or Dibromomethane, Zinc Dust, and Copper(I) Chloride. A 300-mL, three-necked, round-bottomed flask was equipped with a double condenser, a mechanical stirrer, and a 125-mL pressure-equalizing dropping funnel containing 25 mL of solvent to use in replenishing solvent losses. A typical reaction was run on 0.1-mol diene scale in 75–80 mL of solvent with 10 mol % copper(I) chloride based on zinc and 2.5 mol % acetyl chloride based on dihalomethane. All of the reactants and solvent except the acetyl chloride were added to the reaction flask, and after stirring was started, the acetyl chloride was added by syringe. The reaction mixture was then heated in an oil bath at 48–50 °C until all of the dihalomethane had reacted as determined by ^1H NMR examination of an aliquot. Upon completion, the reaction mixture was diluted with pentane and worked up by adding saturated aqueous ammonium chloride solution. The organic layer was separated, washed with 10% aqueous NaOH and saturated aqueous NaCl, and dried over sodium sulfate. After removal of volatile solvents, the unreacted diene, monocyclopropanated products, and dicyclopropanated product were distilled from polymeric side products under vacuum as one fraction over a wide boiling range. This distillate was subsequently analyzed by capillary GC.

General Procedure for Cyclopropanations Using Diiodomethane and Diethylzinc. A 250-mL, three-necked, round-bottomed flask equipped with a thermometer, pressure-equalizing dropping funnel, and magnetic stirrer was sealed with serum caps, evacuated, and filled with nitrogen. The diene (0.025 mol) and diethylzinc, the latter as a 1.1 M solution in toluene, were added by syringe, and the diiodomethane was added dropwise over 30–45 min at room temperature while the mixture was being stirred. After reacting at room temperature for about 5 h, the mixture was worked up with saturated aqueous ammonium chloride. The organic layer was separated, washed with 10% aqueous NaOH, and dried over sodium sulfate before distillation and GC analysis.

General Procedure for Cyclopropanations Using Diiodomethane and Triethylaluminum. An oven-dried, 250-mL, three-necked, round-bottomed flask equipped with a thermometer, nitrogen inlet, and serum cap was filled with nitrogen. The diene (0.025 mol) and diiodomethane dissolved in dichloromethane (40 mL) were added to the flask followed by the triethylaluminum as a 1.9 M solution in toluene. The mixture was allowed to react at room temperature for almost 5 h and then was worked up by dilution with dichloromethane and treated at 0 °C with a solution of 4 g of sodium fluoride and 1.5 mL of water. The organic layer was separated, washed with saturated aqueous NaCl, and dried over sodium sulfate before distillation and GC analysis.

Analysis of Product Mixtures from Cyclopropanations of Limonene. The wide-range-distilled product mixtures obtained from partial cyclopropanations of limonene under the various conditions were analyzed by capillary GC on a 30-m SE-54 column. The retention times at 80 °C for unreacted limonene and its various cyclopropanated products, in minutes from time of injection, were as follows: limonene, 6.7; *cis*- and *trans*-4-(1-methylethenyl)-1-methylbicyclo[4.1.0]heptanes, 8.5 and 8.7 (almost completely resolved); 4-(1-methylcyclopropyl)-1-methylcyclohexene, 11.2; *cis*- and *trans*-4-(1-methylcyclopropyl)-1-methylbicyclo[4.1.0]heptanes, 14.5 and 14.7 (poorly resolved). The identities of each of the products were established by ^1H NMR examination of pure samples collected by GC from a SE-30 packed column. The isomeric 4-(1-methylethenyl)-1-methylbicyclo[4.1.0]heptanes were characterized by the absence of a ring olefinic proton absorption at δ 5.42 and the presence of absorptions for the vinylic protons for the two stereoisomers at δ 4.60 and 4.71. The 4-(1-methylcyclopropyl)-1-methylcyclohexene was characterized by the absence of a vinylic proton absorption at δ 4.72 and the presence of a δ 5.38 ring olefinic proton absorption. The isomeric 4-(1-methylcyclopropyl)-1-methylbicyclo[4.1.0]heptanes exhibited no absorptions in the alkenyl proton region. All of these materials had been characterized earlier by other workers.[11,12]

Analysis of Product Mixtures from Cyclopropanation of 4-Vinylcyclohexene. The wide-range-distilled product mixtures obtained from partial cyclopropanation of 4-vinylcyclohexene under the various conditions were analyzed by capillary GC with a SE-54 column. The retention times at 80 °C for unreacted 4-vinylcyclohexene and its different cyclopropanated products, in minutes from time of injection, were as follows: 4-vinylcyclohexene, 2.7; *cis*- and *trans*-3-cyclopropylnorcaranes, 5.0 (two isomers did not separate); 4-cyclopropylcyclohexene, 5.3; *cis*- and *trans*-3-cyclopropylnorcaranes, 11.3 and 11.5 (partially resolved). The identities of each of the products were established by ^{1}H NMR examination of pure samples collected by GC from a SE-30 packed column. All of the materials had been characterized earlier by other workers.[13]

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Registry No. **1a**, 138-86-3; **1b**, 100-40-3; **2a**, 60059-22-5; **2b**, 80105-51-7; *cis*-**3a**, 62740-28-7; *trans*-**3a**, 62740-29-8; *cis*-**3b**, 131791-69-0; *trans*-**3b**, 71268-78-5; *cis*-**4a**, 62740-27-6; *trans*-**4a**, 62869-85-6; *cis*-**4b**, 131791-70-3; *trans*-**4b**, 131791-71-4.

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Structure and Inversion Barrier of Phosphirene and 1,2-Dihydrophosphete. An ab Initio Study

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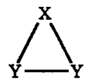
The geometry of 1*H*-phosphirene (**1**), 2*H*-phosphirene (**2**), and 1,2-dihydrophosphete (**3**) were optimized at HF/6-31G*, and single-point calculations at MP2 were performed. The transition structure for inversion at P of **1** and **3** were also optimized (**1pl** and **3pl**). The inversion barrier of **1** and **3** is 92.65 kcal mol^{-1} and 46.62 kcal mol^{-1}, respectively. **1** is not antiaromatic based on the highly pyramidal P atom and no energetic destabilization. Analysis of the electron density distribution and comparison with the inversion barriers of N analogues indicate that the antiaromatic contribution in **1pl** is small.

Small-membered rings have long fascinated organic chemists for their structural simplicity and strained bonds. Small rings incorporating multiple bonds severely test the limits of chemical bonds and ring strain and offer excellent opportunities to evaluate bonding theories. Recently, Regitz[1-3] and Mathey[4-6] have independently developed synthetic strategies for preparing both 1*H*-phosphirene (**1**) and 2*H*-phosphirene (**2**). Of interest is the relative energy of these compounds, particularly in comparison to the nitrogen analogues, since Regitz found that chlorine substituted 2*H*-phospirenes will rearrange to give 1*H*-phosphirenes, contrary to azirenes.[3] The inversion barrier of **1** is of interest since this process goes through a cyclic, planar, formally 4π-electron geometry which may be antiaromatic. Comparison with the inversion barrier of 1*H*-azirine will provide information concerning the effects of pyramidalization and bond lengths on cyclic 4π-electron interactions. Determining the inversion barrier of 1,2-dihydrophosphete (**3**) allows for comparison of the effects of ring size. Following up on our previous theoretical studies of phosphirane **4** and phosphetane **5**,[7] we report here the continuation of our examination of strained-ring organophosphorus systems. Using ab initio calculations, we have determined the geometries of **1–3** and the inversion barrier of **1** and **3**. Comparisons of the geometries, energies, and electron density distributions are made with both their saturated and nitrogen analogues.

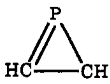
Table I. Energies of 1–3 Using the 6-31G* Basis Set

compd	E(HF)[a]	rel E[b]	E(MP2)[a]	rel E[b]
1	−418.107 704	0.0	−418.462 482	0.0
1pl	−417.951 072	98.29	−418.314 835	92.65
2	−418.112 186	2.81	−418.482 100	−12.31
3	−457.174 108	0.0	−457.656 699	0.0
3pl	−457.090 647	52.37	−457.582 399	46.62

[a] In atomic units. [b] Relative energy in kilocalories per mole.



	X	Y
1	PH	CH
4	PH	CH$_2$
6	NH	CH
7	NH	CH$_2$



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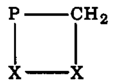

	X
3	CH
5	CH$_2$

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