MS calcd for $C_{13}H_{11}NOSe$, 277; found, 277 (M^+), 261, 199, 157, 122, 93, 77. Anal. Calcd for $C_{13}H_{11}NOSe$: C, 56.53; H, 4.01; N, 5.07. Found: C, 56.56; H, 3.99; N, 5.06.

***N*-[2,4-Dimethyl-6-oxo-2,4-cyclohexadien-1-ylidene]benzeneselenenamide (19).** An authentic sample of the selenoimine **19** was prepared in 62% yield from the reaction of 3,5-dimethylphenol (**18**) with **2a** and **3** in CH_2Cl_2 at room temperature. The product upon isolation gave ^1H NMR ($CDCl_3$) δ: 7.86–7.79 (dd, J = 8.38, 1.44 Hz, 2 H), 7.49–7.40 (m, 2 H), 7.36–7.26 (m, 1 H), 6.43 (m, 2 H), 2.35 (s, 3 H), 2.17 (s, 3 H). ^{13}C NMR ($CDCl_3$) δ: 177.7, 154.4, 152.9, 141.9, 137.8, 129.0, 127.8, 127.6, 125.7, 121.8, 22.8, 18.0. ^{77}Se NMR ($CDCl_3$) δ: 1067. IR (CCl_4) ν: 3057, 2967, 2921, 1633, 1594, 1549, 1477, 1444, 1225, 1020, 840, 785. UV/vis (abs) λ: 485, 232. MS calcd for $C_{14}H_{13}NOSe$, 291; found, 291 (M^+), 214, 157, 134, 106, 91, 77. Anal. Calcd for $C_{14}H_{13}NOSe$: C, 57.94; H, 4.51; N, 4.83. Found: C, 57.80; H, 4.52; N, 4.75.

***N*-[2,4-Bis(1,1-dimethylethyl)-6-oxo-2,4-cyclohexadien-1-ylidene]methaneselenenamide (21).** Reaction of methaneseleninic anhydride (**1b**) with hexamethyldisilazane and 3,5-di-*tert*-butylphenol (added after 1 h of reaction time) under argon gave 67% of the selenoimine **21** upon isolation. ^1H NMR ($CDCl_3$) δ: 6.64 (d, J = 1.94 Hz, 1 H), 6.52 (d, J = 1.94 Hz, 1 H), 2.73 [s, 3 H (the satellite doublet of 3.69% intensity, $^2J_{Se,H}$ = 14 Hz), 1.38 (s, 9 H), 1.21 (s, 9 H). ^{13}C NMR ($CDCl_3$) δ: 178.1, 163.0, 153.9, 147.8, 118.9, 118.4, 36.5, 35.7, 30.6, 28.9, 20.7. ^{77}Se NMR ($CDCl_3$) δ: 1052. IR (film) ν: 2962, 2870, 1623, 1589, 1534, 1441, 1327, 1244, 1180, 1023, 909, 867, 733. UV/vis (abs in $CHCl_3$) λ: 400, 241. MS calcd for $C_{15}H_{23}NOSe$, 313; found, 313 (M^+), 298, 218, 191, 174, 162, 124, 91, 57. Anal. Calcd for $C_{15}H_{23}NOSe$: C, 57.69; H, 7.42; N, 4.48. Found: C, 57.49; H, 7.40; N, 4.47.

Acknowledgment. We thank the Robert A. Welch Foundation (Grant No. 455060) and Quest International for the financial support of this work. We also thank Dr. Howard Williams of this department for assisting us with the ^{15}N NMR studies. We thank Professor D. H. Russel of this department for conducting TOF-MS experiments for us. Thanks are also extended to Professor Jaan Laane of this department for the use of a Laser Raman spectrophotometer. We thank Dr. Josesh Riebenspies of this department for crystal structure determination of selenoimine **8**. Thanks are also due to Dr. David Jacobs of this department for carrying out FAB-MS analyses.

Structure and Bonding in Cyclic Tetrameric Selenium–Nitrogen and Phosphorus–Nitrogen Systems. A Comparative View

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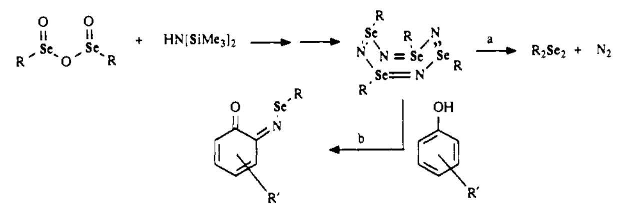
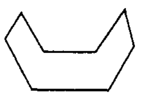
Abstract: Ab initio calculations have been used to study the relative stabilities of various conformations in the tetrameric cyclic $(RSeN)_4$ and $(R_2PN)_4$ systems. The high stability of the "tub" conformer has been rationalized on the basis of electronic and electrostatic interactions. The orientation of the p_π orbitals of the nitrogen atoms is crucial to the stability of these ring systems. Compared to other conformers, the tub conformer allows the maximum number of d orbitals of the Se or P atoms to participate in the π delocalization. Two important differences between the P–N and Se–N ring systems are the stronger ionic character of the Se–N bonds and the steric effect of the Se lone pair.

In the preceding paper, a reactive intermediate was identified in the mechanistic study of the oxidation of phenols to (phenylseleno)iminoquinones by benzeneseleninic anhydride with hexamethyldisilazane, as shown in Scheme I.[1] From extensive NMR evidence the nature of this molecule was determined to be the symmetrical cyclic tetrameric entity $(RSeN)_4$. These cyclic intermediates were found to be very reactive as they disproportionated to the corresponding diselenides and nitrogen gas. It was clear that the factors associated with their assembly, stability, and reactivity needed to be investigated further. Although to the best of our knowledge no structural information can be found on this class of molecules, one can easily relate this intermediate to its phosphorus and sulfur analogues, tetrameric phosphazenes $[(X_2PN)_4$,[2] X can be halide, alkyl, or alkoxyl group] and tetrathiatetraazaenes $(FSN)_4$.[3] Most tetrameric phosphazenes are found in "tub" (S_4), "chair" (C_{2h}), "crown" (C_{4v}), and "saddle" (S_4) (or hybrid) conformations (**1–4**). Only one example, $(F_2PN)_4$, has been found to be planar.[4]

Scheme I





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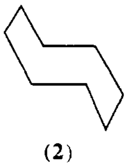

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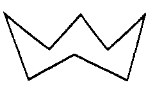

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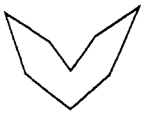

(4)

In this paper, ab initio calculations were used to study the relative stability of the four different conformations mentioned above and to rationalize the structure and bonding in these Se–N

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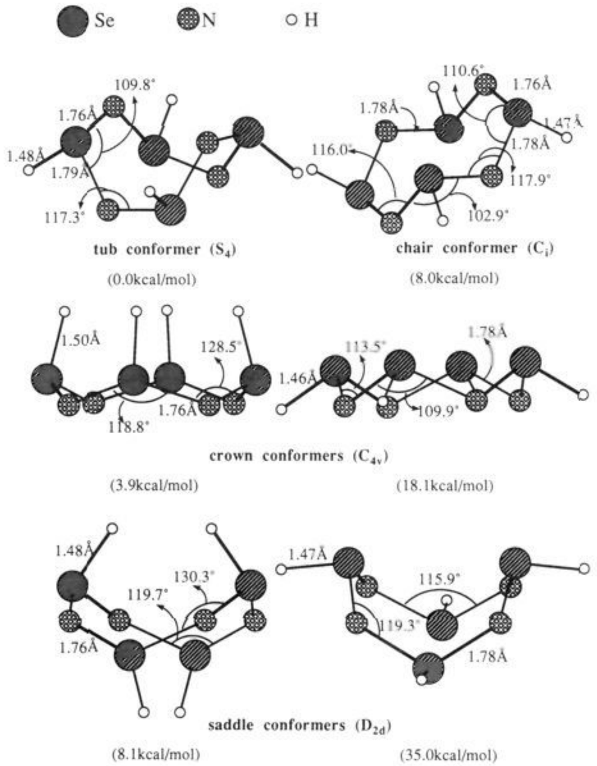


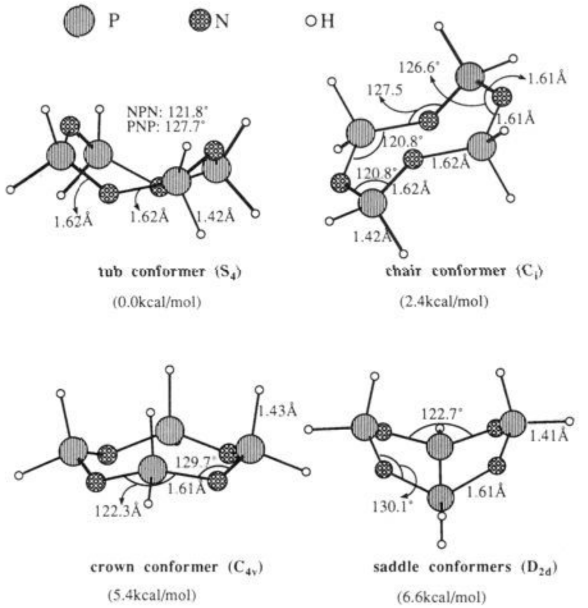
tub conformer (S_4) (0.0kcal/mol)

chair conformer (C_i) (8.0kcal/mol)

crown conformers (C_{4v}) (3.9kcal/mol) (18.1kcal/mol)

saddle conformers (D_{2d}) (8.1kcal/mol) (35.0kcal/mol)

Figure 1. Calculated geometries and relative energies for different conformations of model $(HSeN)_4$ cyclic oligomer.



tub conformer (S_4) (0.0kcal/mol)

chair conformer (C_i) (2.4kcal/mol)

crown conformer (C_{4v}) (5.4kcal/mol)

saddle conformers (D_{2d}) (6.6kcal/mol)

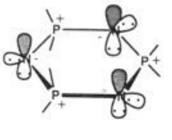
Figure 2. Calculated geometries and relative energies for different conformations of model $(H_2PN)_4$ cyclic oligomer.

systems as well as in their P–N analogues. The planar structure is not included in this study since it requires an impossibly large N–Se–N bond angle for the Se atom. In the ab initio calculations, the effective core potential (ECP) and double-ζ basis sets of Hay and Wadt,[5a] augmented with polarized functions ($\zeta_d = 0.338$), were employed for the Se atoms. For P and N atoms, the ECPs and basis sets with a double-ζ representation in Stevens, Basch, Krauss were used.[5b] Polarization functions were also added to both P ($\zeta_d = 0.340$) and N ($\zeta_d = 0.864$) atoms. The Dunning–Huzinaga double-ζ basis set (31) were used for the H atom.[5c,d]

Results and Discussion

Ab initio full-gradient geometry optimizations were performed on the model compound $(HSeN)_4$ to obtain "tub", "chair", "crown", and "saddle" conformers. These calculated structures are shown in Figure 1 together with relative energies and structural parameters. For comparison, we also optimized the model phosphazene compound $(H_2PN)_4$. These results are shown in Figure 2.

All phosphazenes contain the formally unsaturated alternating double and single bonds, $-N{=}PR_2-N{=}PR_2-$. However, the structural information in this class of compounds gives equal (or almost equal) skeletal P–N bond distances. Thus far, all theoretical investigations[6] generally have supported the so called "island" model of Dewar et al.[6a] to describe the bonding in trimeric phosphazene (see **5**). In the island model, the phosphorus is assigned a 1+ formal charge while the nitrogen is assigned at 1− formal charge. The model is consistent with sp^2 hybridization of the nitrogen and approximately sp^3 hybridization of phosphorus. The π electrons, which are essentially localized on nitrogen, can be significantly stabilized through interaction with empty phosphorus d orbitals. The stabilizing effect of the phosphorus d orbitals is, at least, partially responsible for the existence of the variety of conformations of the tetrameric phosphazenes. The importance of the phosphorus d orbitals in these systems has recently been confirmed.[6i]



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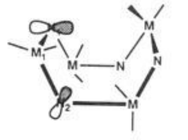

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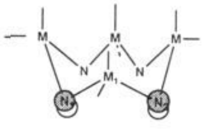

(7)

As we can see from Figure 2, the energy differences among the four conformations of tetrameric phosphazenes are relatively small. However, the corresponding energy differences for tetrameric Se–N conformations become significant (see Figure 1). Strikingly, the two conformers of the crown structure (and the saddle structure, see Figure 1) have a significant energy difference. In this discussion, we attempt to explain the following points: (1) the extensive delocalization of π electrons in a nonplanar ring geometry in the phosphazenes and the Se–N analogues; (2) the high stability of the tub structure; (3) the difference between phosphazenes and Se–N analogues; and (4) the large energy differences between the two conformers of crown structure (and saddle structure).

The bonding in phosphazenes and related Se–N compounds is not adequately described by the concepts of aromatic hydrocarbon systems in which the extensive electron delocalization is via p_π–p_π bonding. In phosphazenes and the corresponding Se–N analogues, the d orbitals of phosphorus and selenium provide flexibility of d_π–p_π bonding without the restriction of planar geometry. Even for planar geometry, the participation of more than one d orbital from each phosphorus allows the P–N systems to disobey the Hückel rule of $(4n + 2)$ π electrons. Examples include planar $(F_2PN)_4$ (8 π electrons) and planar cyclodiphosphazene $[(R_2N)_2PN]_2$ (4 π electrons). Figure 3 provides an illustration of π interactions for a planar 8-membered ring P–N system. Each phosphorus has two d orbitals with π symmetry. The interaction between these d_π orbitals of phosphorus and p_π orbitals of nitrogens

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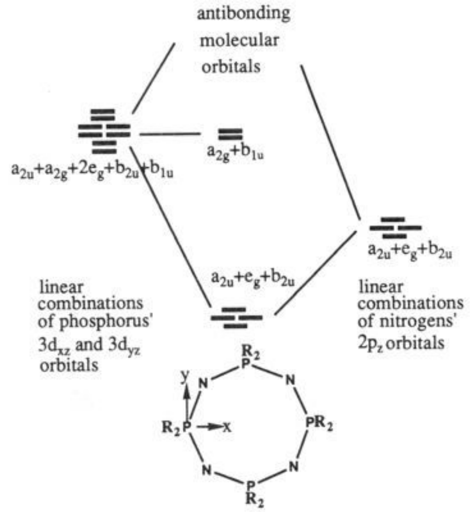


Figure 3. Molecular orbital interaction scheme for a planar $(R_2PN)_4$ cyclic oligomer.

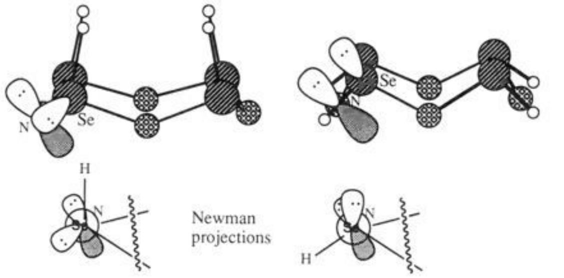
gives four strong bonding molecular orbitals, two nonbonding orbitals, and six antibonding orbitals. This result is significantly different from the typical p_π molecular orbitals derived for planar aromatic ring systems that obey the Hückel rule.

Since the tub structure is the most stable conformation for both phosphazene and the Se–N analogues (see Figures 1 and 2), the extensive delocalization in such a structure will be discussed in detail. An illustration of p_π orbitals of nitrogens for the tub structure is shown in **6**. The p_π (N_1) and p_π (N_2) orbitals are almost perpendicular to each other. This perpendicular arrangement allows maximum π interaction between M_1 and N_1, and M_1 and N_2, because N_1 and N_2 do not need to share the d_π orbitals from M_1. In contrast, the two adjacent p_π (N) orbitals in a crown structure [see **7** for p_π (N_1) and p_π (N_2)] are almost coplanar. Here they must share the d_π orbitals of the metal for efficient M–N π interactions. We can see that the chair structure has π interactions almost identical to those of the tub conformer. In the eight-membered planar structure, each phosphorus (or selenium) also provides two d_π orbitals for π bonding. However, the reduction in the skeletal strain upon the planar-to-nonplanar transformation (because the phosphorus (or selenium) does not allow large angles around it) is responsible for the lack of planar examples.

Since each phosphorus (or selenium) in the tub and chair structures provides more d_π orbitals for the π bonding than that in crown and saddle structures, one could expect the tub and chair conformers to be more stable than the crown and saddle ones. In phosphazene, the tub and chair isomers are indeed more stable than the crown and saddle ones. However, in the Se–N analogues, one of the two crown conformers is more stable than the chair conformer. Thus, factors other than d_π bonding must influence the stabilities in these systems.

In hydrocarbon chemistry the chair conformation is more stable than the tub one. We found the situation to be quite the contrary in phosphazene and Se–N analogues. Here, the influence of the electrostatic force plays a considerable role in the overall stability of various conformations. The calculations performed for electrostatic attraction potentials on the basis of the point charge model[7] showed that in the selenium–nitrogen cyclic polymers the potential in the tub conformer is greater than that in the chair one, whereas these potentials were of comparable values and much smaller in magnitudes in the P–N analogues. This clearly showed that these electrostatic interactions are less significant in phosphazanes than in Se–N systems. This is presumably due to more ionic character of the Se–N bonds compared to P–N bonds. Moreover, we also found that in the $(RSeN)_4$ the crown conformer (the more stable one) had much higher electrostatic attraction potential than the chair conformer. This difference in electrostatic attraction can explain why one of the two crown Se–N isomers is even more stable than the chair conformer. In the P–N systems the electrostatic effect is less important, and hence, the crown conformer is less stable than the chair one.

Now let us examine why the energy of two Se–N crown conformations (and the two saddle conformations, see Figure 1) differ so significantly. As mentioned above, the π electrons in the P–N and Se–N systems are essentially localized on the nitrogens. The orientation of these p_π orbitals is shown in **8** for the two crown isomers (different view of the structure). The orientation of lone pairs on selenium depends on the positions of the hydrogens and is significantly different in the two rotational isomers **8**. In the lower energy conformers the selenium lone pairs are located nearly on the nodal plane of the p_π orbitals of nitrogens, while in the higher energy conformers the Se lone pairs are almost parallel to the p_π orbitals of the neighboring nitrogens. Therefore, significant electron–electron and Pauli repulsion is expected for the higher energy isomer. The repulsion also lengthens the Se–N bonds significantly (see Figure 1). Other lower symmetry crown and saddle conformers with different combinations in the orientations of the R groups are possible. Based on the arguments above, we conclude that the two crown (or saddle) conformers in Figure 1 represent the lowest and the highest energy structures. Therefore, all the other possible isomers fall between these two extremes in energy. In phosphazenes, two P–R bonds, instead of a Se–R bond and a lone pair, lead to no difference between the two corresponding isomers.



(**8**)

For theoretical simplicity, we replaced the phenyl groups in $(PhSeN)_4$ with hydrogens. This simplification ignores possible steric interactions among the R groups in various conformations of $(RSeN)_4$ molecule. Variation in the steric bulk of the substituents in the phosphazenes have not shown any dramatic dependence on the overall stability of the ring conformations.[2] Since the substituents are much further away from the ring system in most conformers of $(RSeN)_4$ molecule, the p_π–d_π interactions and extensive delocalization in these nonplanar structures should dominate the relative energies of various conformers. Nonetheless, significant steric interactions among R groups are expected for the two lower energy crown and saddle conformers, as shown in Figure 1. Therefore, for real $(RSeN)_4$ compounds the two lower energy crown and saddle conformers may have higher energies than the chair conformers. We do not expect large steric interactions for tub, chair, higher energy crown, or saddle conformers because of the orientations of the R groups.

Conclusion

In summary, a tub structure is determined to be the most stable structure for the tetrameric $(RSeN)_4$ intermediate observed in the oxidation reaction of phenols to (phenylseleno)iminoquinones. The evidence presented in the preceding paper[1] showed that the cyclic tetrameric Se–N molecules must have nonequilibrating formal single–double bonds. The present study concludes that the tub conformer does render nonequivalent bond lengths. The stability of this tub structure in the tetrameric Se–N intermediate is discussed together with other possible structures, such as chair, crown, and saddle. For comparison, we have also discussed the relative stabilities among different conformations of the tetrameric phosphazenes. The participation of more than one d orbitals in

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(7) Atomic charges are derived from Mulliken population analyses.

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the π bonding in the P–N and Se–N systems is important in stabilizing the low-energy conformer. This π bonding together with the electrostatic interaction is responsible for the high stability of the tub structure.

Acknowledgment. We thank the Robert A. Welch Foundation (Grants Nos. A-648 and 455060) for financial support of this work. We would also like to thank M. F. Guest for providing the GAMESS package of programs.

Electronic and Steric Control in Carbon–Hydrogen Insertion Reactions of Diazoacetoacetates Catalyzed by Dirhodium(II) Carboxylates and Carboxamides

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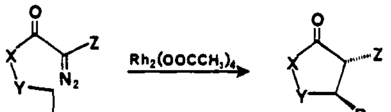
Abstract: Carboxylate and carboxamide ligands on dirhodium(II) catalysts can provide enormous regiocontrol in carbon–hydrogen insertion reactions of diazoacetate esters. Whereas 2,3,4-trimethyl-3-pentyl diazoacetoacetate (**1**) forms γ-lactone products from insertion into primary and tertiary C–H bonds in a statistical distribution (61:39) with dirhodium(II) tetrakis(perfluorobutyrate), only tertiary C–H insertion is observed with dirhodium(II) tetraacetamide. Similar results are obtained with 2-methyl-2-octyl diazoacetoacetate (**3**), where competition for insertion exists between secondary and primary C–H bonds and electronic factors govern regioselection. However, with 2-methyl-3-isopropyl-3-heptyl diazoacetoacetate (**2**) and 2-methyl-1-phenyl-2-propyl diazoacetoacetate (**4**), product distributions from C–H insertion are invariant with the dirhodium(II) ligands; insertion into a secondary C–H bond is favored over tertiary C–H insertion with **2** (95:5), and insertion into a primary C–H bond is preferred to benzylic secondary C–H insertion with **4** (70:30). In such cases, which are amenable to analyses by MM2 calculations, regioselectivity is determined by conformational preferences for which C–H insertion selectivity can be as random as that found with **2** and **4**. When only one C–H bond site is available for insertion to form a five-membered ring product, only one γ-lactone is observed from reactions catalyzed by dirhodium(II) tetraacetate, and that product is not necessarily the one predicted by presumed electronic preferences.

The development of dirhodium(II) catalysts for intramolecular carbon–hydrogen insertion reactions of diazo carbonyl compounds has been a significant synthetic achievement.[1,2] β-Keto-α-diazo esters, phosphonates, and sulfones have been employed for the construction of cyclopentanone derivatives (eq 1: X = Y = CH_2; Z = COOEt, $PO(OR)_2$, SO_2Ar),[3-6] diazoacetates and diazoacetoacetates form γ-lactones (eq 1: X = O; Y = CH_2; Z = H, $COCH_3$),[7] 3-alkoxy-1-diazoacetates provide access to 2(3*H*)-dihydrofuranones (eq 1, X = CH_2, Y = O, Z = H),[8] and diazomalonate esters are reported to produce either or both γ-lactones and β-lactones.[9] These reactions occur in moderate to high yield and, ordinarily, with an overwhelming preference for the formation of a five-membered ring. There are, however, notable exceptions to five-membered ring formation, especially in reactions of diazo amides[10] and of sterically constrained systems,[11,12] that limit predictability for these constructions.



(1)

Regiocontrol in carbon–hydrogen insertion reactions is one of the major advantages attributed to the use of rhodium(II) acetate as the catalyst.[2] For reactions involving five-membered ring formation, there is general agreement that insertion into a tertiary C–H bond is favored over insertion into a secondary C–H bond, and primary C–H insertion, when observed, is the least favorable.[7,13,14] Surprisingly, for what is commonly regarded to be an electrophilic transformation, allylic or benzylic C–H bonds are reported to be less reactive than secondary C–H bonds for insertion,[13] although, more recently, allylic C–H insertion has been shown to be greatly preferred to nonallylic secondary C–H in-

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