mp 234-235 °C]. Similarly, tetramethylurea was prepared from dimethylamine using twice the amount of catalyst. **Tetramethylurea**: colorless oil; bp 180 °C [lit.[24] bp 176.5 °C]. **^{13}C NMR Study on the Reaction Path.** Butylamine (1.3 mmol), Ph_3SbO (0.1 mmol), P_4S_{10} (0.2 mmol), and benzene-d_6 (0.5 mL) were charged into a NMR tube, and then the tube was sealed under CO_2 (0.2 MPa at room temperature). Monitoring the carbonylation by ^{13}C NMR spectra was done in a step-by-step manner as follows: the tube was shaken in the incubator at 15 °C overnight (step 1), at 30 °C overnight (step 2), at 60 °C overnight (step 3), and at 80 °C for 24 h (step 4). Compound 1 was prepared separately by placing butylamine under CO_2 atmosphere. **Butylammonium butylcarbamate (1)**: white crystalline solid; mp dec above 80 °C; ^{13}C NMR ($CDCl_3$) δ 162.8 (s), 40.9 (t, $^1J(^{13}C^1H)$ = 133.4 Hz, CH_2NHCO_2), 38.7 (t, $^1J(^{13}C^1H)$ = 143.0 Hz, CH_2N^+), 32.2 (t, $^1J(^{13}C^1H)$ = 123.0 Hz), 30.0 (t, $^1J(^{13}C^1H)$ = 128.2 Hz), 19.5 (t, $^1J(^{13}C^1H)$ = 126.8 Hz), 13.1 (q, $^1J(^{13}C^1H)$ = 127.8 Hz). In step 1, signals of 1 were observed predominantly. New signals appeared in the mixture after step 2. These were assignable to those of triphenylantimony bis(butylcarbamate) (2) by means of the correlation of $\delta(^{13}C)$ of phenyl carbon with the nature of the ligands in triphenylantimony(V) derivatives based on the data collected by Havránek and Lyčka.[25] 2: 162.3 (s), 139.0 (s, ipso), 133.8 (d, o), 131.6 (d, p), 129.7 (d, m), 40.5 (t), 33.4 (t), 20.8 (t), 14.3 (q). Further, the generation of butylammonium *N*-butylcarbamothioate (3) was suggested by an appearance of a signal at 185.6 ppm[26] during steps 3 and 4. Finally, signals of dibutylurea were only observed after step 4.

Ph_3SbO-Catalyzed Carbonylation of Diamines by CO_2. Imidazolidinone. Ethylene diamine (20 mmol, 1.2 g) with 1.0 mmol of Ph_3SbO and 2.0 mmol of P_4S_{10} was autoclaved under pressure of CO_2 (4.9 MPa). Imidazolidinone was isolated by column chromatography (Silica gel, eluted by ethyl acetate/hexane, 1/1 in volume) (yield 1.5 g; 85%): colorless crystals; mp 130 °C [lit.[27] mp 129-131 °C]. Similarly, the following imidazolidinones were prepared from the corresponding diamines. **1-Methyl-2-imidazolidinone**: colorless crystals; mp 111-113 °C [lit.[28] mp 112 °C]. **1-Phenyl-2-imidazolidinone**: colorless crystals; mp 167 °C [lit.[29] mp 166 °C]. **1-(2-Hydroxypropyl)-2-imidazolidinone**: colorless; mp 72-73 °C; IR (KBr) 1675 cm^{-1}; ^1H NMR ($CDCl_3$) δ 5.24 (bd, 1 H, NH), 3.88-4.07 (A_2X_3 type m, 1 H, CH), 3.30-3.80 (m, 5 H, ring CH_2 and OH), 3.17 (d, 2 H, J = 5.5 Hz), 1.17 (d, 3 H, J = 6.2 Hz, CH_3); ^{13}C NMR ($CDCl_3$) δ 163.9 (s), 66.5 (d), 52.0 (t), 47.0 (t), 38.5 (t), 20.8 (q); MS (EI) m/z 144 (M^+). Anal. Calcd for $C_6H_{12}N_2O_2$: C, 49.99; H, 8.39; N, 19.43. Found: C, 50.00; H, 8.38; N, 19.41. **1-(2-Hydroxyethyl)-2-imidazolidinone**: colorless crystals; mp 52 °C [lit.[30] mp 53.5-57.5 °C]. **1,3-Dimethyl-2-imidazolidinone (8)**: colorless oil; bp 88 °C/1.3 kPa [lit.[31] bp 106-108 °C/2.3 kPa]; ^{13}C NMR ($CDCl_3$) δ 160.2 (s), 43.6 (t), 30.0 (q).

Synthesis of Trisubstituted Ureas. This series of reactions was carried out using 1.0 mmol of Ph_3SbO, 2.0 mmol of P_4S_{10}, 10 mmol of butylamine, and 10 mmol of diethylamine or 10 mmol of *N,N'*-dimethylethylenediamine. After the general workup the yields of the products were estimated by ^{13}C NMR spectra using benzophenone as an internal standard with respect to the predetermined calibration factors. Isolation of the ureas was accomplished by column chromatography (silica gel) eluted by hexane, ethyl acetate/hexane (1/1 in volume), ethyl acetate, methanol/ethyl acetate (1/1 in volume), and methanol, successively. **1-Butyl-3,3-diethylurea**: colorless oil; bp 150 °C/1.1 kPa; IR (KRS-5) 1630 cm^{-1}; ^1H NMR ($CDCl_3$) δ 4.44 (bd, 1 H), 3.06-3.19 (m, 6 H), 1.32-1.43 (m, 2 H), 1.16-1.28 (m, 2 H), 1.00 (t, 6 H, J = 7.1 Hz), 0.81 (t, 3 H, J = 7.1 Hz); ^{13}C NMR ($CDCl_3$) δ 156.7 (s), 40.1 (t), 39.8 (t), 31.9 (t), 19.4 (t), 13.1 (q); HRMS (EI) m/z 172.1562; calcd for $C_9H_{20}N_2O$ 172.1589. **1-[2-(Methylamino)ethyl]-3-butyl-1-methylurea (6)**: slightly yellow oil; bp 150-155 °C/1.3 Pa; IR (KRS-5) 1620 cm^{-1}; ^1H NMR ($CDCl_3$) δ 5.68 (bd, 1 H), 3.07-3.31 (m, 4 H), 2.81 (s, 3 H), 2.65 (t, 2 H, J = 5.7 Hz), 2.35 (s, 3 H), 1.61 (s, 1 H), 1.15-1.45 (m, 4 H), 0.84 (t, 3 H, J = 6.4 Hz); ^{13}C NMR ($CDCl_3$) δ 158.2 (s), 50.3 (t), 49.4 (t), 40.4 (t), 36.1 (q), 34.9 (q), 32.1 (t), 19.9 (t), 13.6 (q); MS (CI) m/z 188 (M^+ + 1); Anal. Calcd for $C_9H_{21}N_3O$: C, 57.72; H, 11.30; N, 22.44. Found C, 58.00; H, 11.12; N, 22.49. ***N,N'*-Bis(butylcarbamoyl)-*N,N'*-dimethylethylenediamine (7)**: slightly yellow crystals; mp 112-114 °C: IR (KBr) 1650 cm^{-1}; ^1H NMR ($CDCl_3$) δ 4.86 (bd, 2 H), 3.36 (s, 4 H), 3.18 (t, 4 H, J = 6.2 Hz), 2.90 (s, 6 H), 1.23-1.56 (m, 8 H), 0.92 (t, 6 H, J = 6.6 Hz); ^{13}C NMR ($CDCl_3$) δ 158.3 (s), 47.3 (t), 40.6 (t), 35.1 (q), 32.3 (t), 20.0 (t), 13.7 (q); MS (CI) m/z 287 (M^+ + 1). Anal. Calcd for $C_{14}H_{30}N_4O_2$: C, 58.74; H, 10.49; N, 19.58. Found C, 58.86; H, 10.55; N, 19.62.

Acknowledgment. Part of this work was supported by a Grant-in-Aid for Scientific Research (No. 03855177) from the Ministry of Education, Science, and Culture, Japan.

(24) Lüttringhaus, A.; Dirksen, H. W. *Angew. Chem., Int. Ed. Engl.* **1960**, *3*, 260.
(25) Havránek, J.; Lyčka, A. *Sb. Věd. Prací. Vys. Škola Chem. Technol., Pardubice* **1980**, *43*, 123.
(26) Breitmaier, E.; Voelter, W. *Carbon-13 NMR Spectroscopy*, 3rd ed.; VCH: Verlag, Weinheim, 1987; pp 134-138.
(27) Wilson, A. L. *US Patent* 2,517,750, 1950.
(28) Etemad-Moghadam, G.; Gasc, M. B.; Klaebe, A.; Perie, J. *Nouv. J. Chem.* **1984**, *8*, 285.
(29) Araki, T.; Nogami, F.; Tenkube, H.; Nagata, K.; Iyoshi, S. *J. Polym. Sci., Polym. Chem. Ed.* **1978**, *16*, 1037.
(30) Ebetino, F. F. *US Patent* 3,254,075, 1966; *Chem. Abstr.* **1966**, *65*, 7187.
(31) Kohn, H.; Cravey, M. J.; Arceneaux, J. H.; Cravey, R. L.; Willcott, M. R., III. *J. Org. Chem.* **1977**, *42*, 941.

Notes

Oxidation of Aliphatic Amines by $HOF \cdot CH_3CN$ Complex Made Directly from F_2 and Water

Shlomo Rozen* and Moshe Kol

School of Chemistry, Raymond and Beverly Sackler Faculty of Exact Sciences, Tel-Aviv University, Tel-Aviv 69978, Israel

Received May 11, 1992

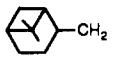
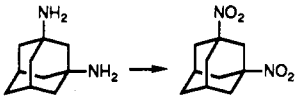
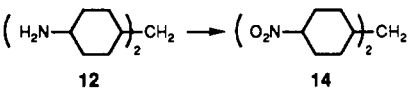
Recently we have developed a powerful, yet relatively stable, oxygen transfer reagent, $HOF \cdot CH_3CN$, simply by passing fluorine through aqueous acetonitrile.[1] It proved to be an effective epoxidizing agent,[2] was used for hydroxylation of tertiary unactivated C-H bonds,[3] and could convert aromatic amines into the corresponding nitroarenes.[4]

The synthesis of aliphatic nitro compounds is usually more difficult than the aromatic ones. There are a few specific methods for this purpose, some described in an

(1) Rozen, S.; Brand, M. *Angew. Chem., Int. Ed. Engl.* **1986**, *25*, 554.
(2) Rozen, S.; Kol, M. *J. Org. Chem.* **1990**, *55*, 5155. Hung, M. H.; Smart, B. E.; Feiring, A. E.; Rozen, S. *J. Org. Chem.* **1991**, *56*, 3187.
(3) Rozen, S.; Brand, M.; Kol, M. *J. Am. Chem. Soc.* **1989**, *111*, 8325.
(4) Kol, M.; Rozen, S. *J. Chem. Soc., Chem. Commun.* **1991**, 567.

0022-3263/92/1957-7342$03.00/0 © 1992 American Chemical Society

Table I

RNH_2	$\xrightarrow{HOF\cdot CH_3CN}$	RNO_2	yield (%)
1	R = cyclohexyl	2	90
3	R = $CH_3(CH_2)_{10}CH_2$	6	85
4	R = $PhCH_2$	7	95
5		8	80
			80
9		10	80
	 11 13		80
	 12 14		70

early review[5] and some in more recent works based on various oxidants including dimethyldioxirane used by Murray.[6] Another approach consists of condensing at least two smaller molecules, one of which already contains the nitro group.[7] However, most methods require either lengthy multistep reactions or metal-containing oxidizers which can create disposal problems. We describe here a new fast and efficient method for oxidizing aliphatic amines to the corresponding nitro derivatives with the unique feature of using the water oxygen as the ultimate oxidizer.

Unlike the case of aromatic amines, the $HOF\cdot CH_3CN$ complex does not react with the aliphatic counterpart and only gradual decomposition of the oxidizing power was observed. The most conspicuous difference between aromatic and aliphatic amines is, of course, that the latter are stronger bases by about 6–7 pK_a units. Since during the synthesis of the $HOF\cdot CH_3CN$ at least one molecule of HF is also formed[8] we reckoned that only the free amine is susceptible to oxidation and salt formation in damp acetonitrile suppresses its concentration. In order to minimize the effect of the hydrofluoric acid we added NaF to the $HOF\cdot CH_3CN$ solution and allowed the mixture to stir for about 10 min at 0 °C. While this procedure hardly affected the oxidizing power, it bound most of the HF in the form of the sparingly soluble $NaHF_2$ salt.

When cyclohexylamine (1) was added to the above mixture, the $HOF\cdot CH_3CN$ disappeared in a few minutes and nitrocyclohexane (2) was formed in excellent yield. This method proved efficient for a wide variety of aliphatic amines, including straight chain, benzylic, and polycyclic ones. Thus, dodecyl- (3), benzyl- (4) and adamantylamine (5) were oxidized to the corresponding nitro derivatives 6, 7[9] and 8.[10] The absorption of the HF by NaF had another beneficial aspect and an acid sensitive system such as the bicyclo mirtanylamine (9) was converted to the corresponding nitro compound (10) without any undesired rearrangements. This method is not confined to monofunctional amines only, and 1,3-diaminoadamantane (11) and 4,4'-methylenebis(cyclohexylamine) (12) were also easily converted to the corresponding dinitro derivatives (13) and (14) in good yields.

As with other reactions of the $HOF\cdot CH_3CN$ system no partial oxidized products were found. However, the difference in the oxidation states between the amino and the nitro group (six units) and the fact that an excess of 3 mol/equiv of $HOF\cdot CH_3CN$ is needed for this oxidation point to a series of two-electron oxidation processes which may indicate that the following reaction sequence is taking place:[11]

$$RNH_2 \xrightarrow[-HF]{HOF\cdot CH_3CN} [RNHOH] \xrightarrow[-HF;\ -H_2O]{HOF\cdot CH_3CN} [RNO] \xrightarrow[-HF]{HOF\cdot CH_3CN} RNO_2$$

The fact that the oxygen atoms originate directly from the water presents a very convenient way for easy and economical labeling of the nitro group with other than the ^{16}O isotope. Thus, reacting 1-aminoadamantane with the oxidative solution obtained from the action of F_2 on $H_2{}^{18}O/CH_3CN$ resulted in the formation of 1-nitroadamantane with both oxygens of the nitro group being the ^{18}O isotope as the MS m/e = 185 (M^+) indicates.

Experimental Section

^1H NMR spectra were recorded with a Bruker WH-360 with $CDCl_3$ as solvent and Me_4Si as an internal standard. The proton broad-band decoupled ^{13}C NMR spectra were recorded at 90.5 MHz. Here too, $CDCl_3$ served as a solvent and TMS as internal standard. Mass spectra were measured with a Du Pont 21-491B spectrometer. IR spectra were recorded as neat films, in $CHCl_3$ solution or as KBr pellets on a Perkin-Elmer 177 spectrophotometer.

General Procedure for Working with Fluorine. Fluorine is a strong oxidizer and a very corrosive material. An appropriate vacuum line made from copper or monel in a well-ventilated area should be constructed for working with this element. For more experimental details see, for example, ref 12. For the occasional user, however, various premixed mixtures of F_2 in inert gases are commercially available, simplifying the whole process. The reactions themselves can be carried out in glass vessels. If elementary precautions are taken, work with fluorine is relatively simple and we have had no bad experiences working with this element.

General Procedure for Producing the Oxidizing $HOF\cdot CH_3CN$. Mixtures of 10% F_2 with nitrogen were used in this work. The gas mixture was prepared in a secondary container before the reaction was started. This mixture was then passed at a rate of about 400 mL per minute through a cold (−10 °C) and vigorously stirred mixture of 400 mL of CH_3CN and 40 mL of H_2O. The formation of the oxidizing power was monitored by reacting aliquots with an acidic aqueous solution of KI. The liberated iodine was then titrated with thiosulfate. It is thus possible to achieve concentrations of more than 1 mol/L of the oxidizing reagent.

General Oxidation Procedure for Aliphatic Amines. About 10 mmol of an amine were dissolved in 10–20 mL of $CHCl_3$ or CH_2Cl_2. The mixture was then cooled to −15 °C and added to the glass reactor containing 5 g of NaF and 30–35 mmol of the oxidizing $HOF\cdot CH_3CN$ in cold (−15 °C) aqueous CH_3CN (100 mL). The reaction was allowed to proceed for 10–15 min when most of the oxidizing compound had been consumed. It was neutralized with saturated sodium bicarbonate solution, poured into 1500 mL of water, extracted with CH_2Cl_2, and washed with $NaHCO_3$ and water until neutral. The organic layer was dried over $MgSO_4$ and the solvent evaporated, preferably at room temperature. The crude product was usually purified by vacuum flash chromatography using Silicagel 60-H (Merck). The spectral and physical properties of the known products thus obtained were compared either with those of authentic samples or with the properties reported in the literature. In every case excellent agreement was

(5) Seebach, D.; Colvin, E. W.; Lehr, F.; Weller, T. *Chimia* **1979**, *33*, 1.
(6) Murray, R. W.; Rajadhyaksha, S. N.; Mohan, L. *J. Org. Chem.* **1989**, *54*, 5783.
(7) Korenblum, N. *Aldrichim. Acta* **1990**, *23*, 71.
(8) See, however: Appelman, E. H.; Jache, A. W. *J. Am. Chem. Soc.* **1987**, *109*, 1754.
(9) Shapiro, M. J. *J. Org. Chem.* **1976**, *41*, 3197.
(10) Smith, G. W.; Williams, H. D. *J. Org. Chem.* **1961**, *26*, 2207.
(11) Keinan, E.; Mazur, Y. *J. Org. Chem.* **1977**, *42*, 844.
(12) Hebel, D.; Rozen, S. *J. Org. Chem.* **1991**, *56*, 6298.

obtained. Data for new compounds or for ones which have not been well-defined in the literature are given below.

Nitromyrtane (10): IR 1545, 1385 cm^{-1}; ^1H NMR 4.40 (2 H, d, J = 8 Hz), 2.91 (1 H, m), 2.41 (1 H, m), 1.95 (5 H, m), 1.53 (1 H, m), 1.21 (3 H, s), 1.02 (3 H, s), 1.02 (1 H, d, J = 8 Hz); ^{13}C NMR 81.1 ppm. Anal. Calcd for $C_{10}H_{17}NO_2$: C, 65.54, H, 9.28. Found: C, 65.62, H, 9.44.

1,3-Dinitroadamantane (13):[10] mp (subl) 211 °C; IR 1530, 1370 cm^{-1}; ^1H NMR 2.71 (2 H, s), 2.59 (2 H, m), 2.22 (8 H, m), 1.73 (2 H, m); ^{13}C NMR 84.8, 42.7, 39.3, 3.36, 30.2 ppm; MS m/z 180 [$(M - NO_2)^+$].

4,4'-Methylenebis(nitrocyclohexane) (14): mp 124 °C; IR 1550, 1540, 1385 cm^{-1}; ^1H NMR 4.50 (1 H, q, J = 4.5 Hz), 4.34 (1 H, tt, J_1 = 12 Hz, J_2 = 4 Hz); MS m/z 224 [$(M - NO_2)^+$], 178 [$(M - 2NO_2)^+$], 177 [$(M - 2NO_2 - H)^+$]. Anal. Calcd for $C_{13}H_{22}N_2O_4$: C, 57.76, H, 8.14. Found: C, 57.68, H, 8.10.

Acknowledgment. This research was supported by Grant No. 89-00079 from the USA-Israel Binational Science Foundation (BSF), Jerusalem, Israel.

Registry No. 1, 108-91-8; 2, 1122-60-7; 3, 124-22-1; 4, 100-46-9; 5, 768-94-5; 6, 16891-99-9; 7, 622-42-4; 8, 7575-82-8; 8-$N^{18}O_2$, 143957-73-7; 9, 74837-99-3; 10, 143957-74-8; 11, 10303-95-4; 12, 1761-71-3; 13, 55100-59-9; 14, 143957-75-9; HOF, 14034-79-8; CH_3CN, 75-05-8.

Halogen-Assisted Alkylation of Ester Enolates. Facile Synthesis of C_{10}-Functionalized Tricyclo[5.2.1.$0^{2,6}$]decenes

Subrata Ghosh,* Goutam Saha, Golam Mostafa,† and Siddhartha Ray†

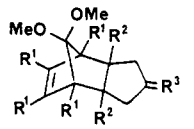
Departments of Organic Chemistry and Solid State Physics, Indian Association for the Cultivation of Science, Jadavpur, Calcutta-700 032, India

Received May 28, 1992

Tricyclo[5.2.1.$0^{2,6}$]decenes are valuable synthetic intermediates. However, utilization of these derivatives in the synthesis[1] of natural products is limited to the cyclopentanoid family due to lack of methods for preparation of various functionalized derivatives. Recently, we have demonstrated[2] that appropriately functionalized tricyclo[5.2.1.$0^{2,6}$]decenes may provide bridged eight-membered rings related to the A/B ring of taxanes. An extension of this route for the synthesis of taxane diterpenes required the synthesis of C_{10}-functionalized tricyclo[5.2.1.$0^{2,6}$]decenes **1** which could not be prepared by the commonly used[3] Diels-Alder reaction of 5,5-disubstituted cyclopentadiene derivatives. An alternative route[4] involving ring annelation via alkylation of the vicinal dianion generated from **2** with 1,3-dihalides also failed. Intrigued by the neighboring heteroatom influence[5] on the reactivity and facial selectivity during addition of electrophiles to nucleophilic double bonds, we investigated the behavior of the dienolate available in principle from the parent tetrachloro diester **3** toward electrophiles. We report here the results of this investigation and demonstrate a dramatic influence of the chlorine atoms on the reactivity of the dienolate of the tetrachloro diester **3** resulting in facile alkylation and annelation.

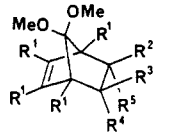
Results and Discussion

Treatment of the diester **2**[6] with LDA in THF followed by treatment of the resulting solution with 1 equiv of 3-chloro-2-(chloromethyl)propene or 2 equiv of MeI in the presence of HMPA failed to afford any alkylated product. Instead, simply a syn-anti isomerization of the ester moieties was observed,[7] giving rise to **4**. On the other hand, in a parallel experiment, the tetrachloro diester **3**,[8] under identical conditions, on reaction with 2 equiv of methyl iodide gave a solid in 93% isolated yield as a mixture of two chromatographically inseparable components in ca. a 1:1 ratio (t_R 2.20 and 2.47). Although theoretically three dimethylated products **5X**, **5N**, and **5T** may arise, the symmetrical nature of the ^1H and ^{13}C NMR spectra of the crude product suggested the presence of only *exo*-**5X** and *endo*-**5N** isomers. The assignment of the structure as exo to the component with t_R 2.20 in GLC, obtained by multiple fractional crystallization of the crude product, could be made by a single-crystal X-ray structure determination. Since the crude mixture and the crystallized product **5X** displayed indistinguishable ^1H and ^{13}C NMR spectra, the second component with t_R 2.47 in the mixture was assigned the endo structure **5N**, and the possibility of the presence of the trans dimethylated compound **5T** in the product mixture could thus be excluded. Successful achievement in alkylation of the diester **3** led us to investigate alkylation with other electrophiles.

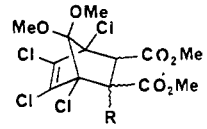


1

a, R^1=Cl, R^2=CO_2Me, R^3=H_2
b, R^1=Cl, R^2=CO_2Me, R^3=CH_2
c, R^1=H, R^2=CH_2OH, R^3=H_2
d, R^1=H, R^2=CH_2OH, R^3=CH_2



2, R^1=R^2=R^3=H, R^4=R^5=CO_2Me
3, R^1=Cl, R^2=R^3=H, R^4=R^5=CO_2Me
4, R^1=R^2=R^4=H, R^3=R^5=CO_2Me
5X, R^1=Cl, R^2=R^3=Me, R^4=R^5=CO_2Me
5N, R^1=Cl, R^2=R^3=CO_2Me, R^4=R^5=Me
5T, R^1=Cl, R^2=R^4=Me, R^3=R^5=CO_2Me



6, R=$CH_2CH=CH_2$
7, R=$CH_2C(=CH_2)CH_2Cl$

Thus, when the alkylation of **3** was carried out with 1 equiv of a bulkier electrophile (i.e., allyl bromide) again a 1:1 mixture of the monoallylated products **6** was obtained in 82% yield. Attempted addition of a second allyl group to **6** was not successful. Ring annelation on **3** was next examined using bifunctional electrophilic reagents. Treatment of the enolate of **3** with 1 equiv of 1,3-dibromopropane under the above conditions gave the ring

† Department of Solid State Physics.

(1) Applications of tricyclo[5.2.1.$0^{2,6}$]decenes in the synthesis of cyclopentanoid natural products: (a) Ito, T.; Tomiyoshi, N.; Nakamura, K.; Azuma, S.; Izawa, M.; Maruyama, F.; Yanagiya, M.; Shirahama, H.; Matsumoto, T. *Tetrahedron Lett.* **1982**, *23*, 1721. (b) Tsunoda, T.; Kodama, M.; Ito, S. *Tetrahedron Lett.* **1983**, *24*, 83. (c) Marchand, A. P.; Vidyasagar, V. *J. Org. Chem.* **1988**, *53*, 4412. (d) Takano, S.; Inomata, K.; Ogasawara, K. *J. Chem. Soc., Chem. Commun.* **1989**, 271. (e) Klunder, A. J. H.; Zwanenburg, B.; Liu, Z. Y. *Tetrahedron Lett.* **1991**, *32*, 3131.

(2) (a) Saha, G.; Bhattacharya, A.; Saha Roy, S.; Ghosh, S. *Tetrahedron Lett.* **1990**, *31*, 1483. (b) Ghosh, S.; Karpha, A.; Saha, G.; Patra, D. *Tetrahedron Lett.* **1992**, *33*, 2363.

(3) Camps, P.; Castane, J.; Feliz, M.; Figueredo, M. *Tetrahedron* **1984**, *40*, 5235.

(4) (a) Garratt, P. J.; Hollowood, F. *J. Org. Chem.* **1982**, *47*, 68. (b) Girard, C.; Bloch, R. *Tetrahedron Lett.* **1982**, *23*, 3683.

(5) Kahn, S. D.; Pau, C. F.; Hehre, W. J. *J. Am. Chem. Soc.* **1986**, *108*, 7396.

(6) Mehta, G.; Khan, F. A. *J. Am. Chem. Soc.* **1990**, *112*, 6140.

(7) Saha, G.; Karpha, A.; Saha Roy, S.; Ghosh, S. *J. Chem. Soc., Perkin Trans. 1* **1992**, 1587.

(8) Newcomer, J. S.; McBee, E. T. *J. Am. Chem. Soc.* **1949**, *71*, 946.

0022-3263/92/1957-7344$03.00/0 © 1992 American Chemical Society