mL of 3 M in Et_2O, 1.35 mmol, 10% excess) was added dropwise via syringe over the course of 10 min. The reaction mixture was allowed to stir for an additional 4 h, and then Et_2O (20 mL) was added slowly to precipitate salts. The cold mixture was then quickly filtered through a plug of alumina, and the solvent was removed in vacuo to give a red solid (0.42 g, 87% crude). NMR spectroscopy showed the presence of Fp_2 and Fp-phenyl, but attempts to remove these via column chromatography led to significant decomposition and rearrangement to compound **18**. Only traces of pure **11c** were recovered and used for elemental analysis. Spectra were taken on samples containing the impurities: IR (CH_2Cl_2) 2000, 1940 cm^{-1} (C≡O); ^1H NMR ($CDCl_3$/TMS) δ 7.6–7.2 (m, 5 H, Ph), 6.95 (br s, 1 H, Fp-C*H*), 4.75 (s, 5 H, Cp), 4.55 (s, 1 H, N≡CC*H*), 3.70, 3.50 (2 m, 2 H, OC*H*C*H*O), 1.75 (br m, 4 H, OCHCH_2's), 1.30 (br m, 4 H, OCHCH$_2$CH_2's); ^{13}C NMR ($CDCl_3$) δ 217.6, 217.1 (C≡O), 142.3 (iPh), 86.4 (Cp), 77.1, 75.0 (O*C*HCHO), 74.7 (Fp-*C*H), 73.3 (*C*HC≡N), 30.3, 29.7 (OCHCH$_2$'s), 24.5, 24.2 (OCHCH$_2$CH$_2$). Anal. Calcd for $C_{21}H_{22}FeO_4$: C, 63.98; H, 5.62. Found: C, 63.67; H, 5.42.

Dicarbonyl(η⁵-cyclopentadienyl)(*trans*,*trans*-2,3,4a,5,6,7,8,8a-octahydrobenzodioxin-2-yl)iron(II) (12a). Same procedure as **11a** using compound **10**. Crude yield was quantitative. Column chromatography gave a yellow solid (0.42 g, 66%). The lower yield after chromatography may be due to ring opening and decomposition as in the attempted purification of **11c**: IR (CH_2Cl_2) 2010, 1954 cm^{-1} (C≡O); ^1H NMR (C_6D_6/CS_2/TMS, 0 °C) δ 5.50 (d of d, 1 H, J = 3 Hz/11 Hz, Fp-C*H*), 4.23 (s, 5 H, Cp), 3.77 (app t, 1 H, J = 11 Hz/12 Hz, Fp-CHCH_{ax}), 3.67 (d of d, 1 H, J = 3 Hz/12 Hz, Fp-CHCH_{eq}), 3.41, 2.34 (2 m, 2 H, OC*H*C*H*O), 1.84–0.97 (br m, 8 H, CH_2's); ^{13}C NMR (C_6D_6/CS_2/TMS, 0 °C) δ 217.3, 216.9 (C≡O), 85.2 (Cp), 79.4 (Fp-CHCH$_2$), 76.9, 74.5 (O*C*HCHO), 68.5 (Fp-*C*H), 31.4, 24.9, 24.7, 20.6 (*C*H$_2$'s). Anal. Calcd for $C_{15}H_{18}FeO_4$: C, 56.63; H, 5.70. Found: C, 57.18; H, 5.34.

Dicarbonyl(*trans*,*trans*,*trans*-3-cyano-2,3,4a,5,6,7,8,8a-octahydrobenzodioxin-2-yl)(η⁵-cyclopentadienyl)iron(II) (12b). Same procedure as **11b**, using compound **10** as the starting material. Column chromatography on alumina (50% ether/petroleum ether) gave a yellow oil (0.22 g, 46%): IR (CH_2Cl_2) 2010, 1950 cm^{-1} (C≡O); ^1H NMR ($CDCl_3$/TMS) δ 5.36 (d, 1 H, J = 10.5 Hz, Fp-C*H*), 4.90 (s, 5 H, Cp), 4.56 (d, 1 H, J = 10.5 Hz, N≡CC*H*), 3.90, 3.50 (2 m, 2 H, OC*H*C*H*O), 2.1–1.30 (br m, 8 H, CH_2's); ^{13}C NMR ($CDCl_3$) δ 215.7, 214.3 (C≡O), 118.1 (C≡N), 85.4 (Cp), 76.5 (*C*HC≡N), 75.7, 75.0 (O*C*HCHO), 66.1 (Fp-*C*H), 30.4, 24.0, 23.4, 19.6 (*C*H$_2$'s). Anal. Calcd for $C_{16}H_{17}FeNO_4$: C, 56.00; H, 4.99; N, 4.08. Found: C, 55.70; H, 5.21; N, 3.87.

Dicarbonyl(η⁵-cyclopentadienyl)(formylmethyl)iron(II) *cis*-Cyclohexane-1,2-diyl Acetal (13a and 13b). To a 5-mm NMR tube containing a solution of **12a** (100 mg, 0.31 mmol in 1 mL of $CDCl_3$) was added via syringe 0.29 mL of a solution of BF_3·Et_2O in $CDCl_3$ (0.109 M, 0.031 mmol, 0.1 equiv) at room temperature. The rearrangement was followed by ^1H NMR spectroscopy and was complete within 3 h. The reaction mixture was then filtered through a short plug of alumina, the plug was washed with ether, and the solvent was removed in vacuo from the combined organics to give a brown oil. Column chromatography of the oil on alumina with 50% ether/hexane gave two fractions: mixed products **13a** and **13b** (55 mg, 55%) and hydrolyzed product (Fp-acetaldehyde): IR ($CDCl_3$) 2010, 1957 cm^{-1} (C≡O); ^1H NMR ($CDCl_3$/TMS) δ 5.31, 5.04 (2 t, 2 H, J = 6 Hz, Fp-CH$_2$C*H*), 4.82, 4.81 (2 s, 10 H, Cp's), 4.06, 4.00 (br s, 4 H, OC*H*C*H*O's), 1.74 (br s, 8 H, OCHCH_2's), 1.54, 1.28 (2 m, 8 H, OCHCH$_2$CH_2's), 1.45, 1.43 (2 d, 4 H, J = 6 Hz, Fp-CH_2); ^{13}C NMR ($CDCl_3$/TMS) δ 216.8 (C≡O), 110.6, 109.0 (Fp-CH$_2$*C*H), 85.04, 85.00 (Cp's), 74.5, 73.8 (O*C*HCHO), 28.9, 27.3 (OCH*C*H$_2$), 21.2, 21.0 (OCHCH$_2$*C*H$_2$), 5.3, 4.4 (Fp-*C*H$_2$).

Dicarbonyl(η⁵-cyclopentadienyl)(cyanoformylmethyl)iron(II) *cis*-Cyclohexane-1,2-diyl Acetal (15). This compound was prepared in the same manner as **13** using **12b** as the starting material. From 63 mg of **12b**, 28 mg (44%) of **15** was recovered: IR ($CDCl_3$) 2005, 1950 cm^{-1} (C≡O); ^1H NMR ($CDCl_3$/TMS) δ 5.10 (d, 1 H, J = 7 Hz, Fp-CHC*H*), 4.75 (s, 5 H, Cp's), 4.10 (m, 2 H, OC*H*C*H*O), 2.40 (d, 1 H, J = 7 Hz, Fp-C*H*), 1.80 (br s, 4 H, OCHCH_2's), 1.30 (br m, 4 H, OCHCH$_2$CH_2's); ^{13}C NMR ($CDCl_3$/TMS) δ 216.8 (C≡O), 124.5 (C≡N), 98.3 (Fp-CH*C*H), 84.7 (Cp's), 75.2, 72.2 (O*C*HCHO), 29.7, 28.2 (OCH*C*H$_2$), 21.1, 20.5 (OCHCH$_2$*C*H$_2$), 1.5 (Fp-*C*H$_2$).

Dicarbonyl(η⁵-cyclopentadienyl)(formylphenylmethyl)iron(II) *trans*-Cyclohexane-1,2-diyl Acetal (18a and 18b). This compound was prepared in the same manner as **13** using **11c** as the starting material. From 121 mg of **11c**, 74 mg (61%) of **18** was recovered. The compounds are formed in a 3:1 ratio (by NMR). Where both are detected spectroscopically, values will be given as major/minor: IR ($CDCl_3$) 2000, 1945 cm^{-1} (C≡O); ^1H NMR ($CDCl_3$/TMS) δ 7.3 (br m, 5 H, Ph), 5.40/5.35 (d, 1 H, J = 7 Hz, Fp-CHC*H*), 4.60 (s, 5 H, Cp), 3.90–3.60 (br m, 3 H, OC*H*C*H*O, Fp-C*H*), 2.10–1.40 (br m, 8 H, CH_2's); ^{13}C NMR ($CDCl_3$/TMS) δ 211.8 (C≡O), 145.9/147.1 (Fp-*C*H), 135.9/134.2 (ipso-Ph), 128.1, 127.9 (o-Ph, m-Ph), 125.6 (p-Ph), 105.9/108.1 (Fp-CH*C*H), 84.5/84.3 (Cp), 73.4, 73.5 (O*C*HCHO), 32.0, 30.6 (OCH*C*H$_2$), 24.0, 23.7 (OCHCH$_2$*C*H$_2$).

Acknowledgment. We gratefully acknowledge financial support from the National Institutes of Health #37067 (K.-H.C. and M.R.) and the Faculty Development Fund of Clark University (H.A.E., undergraduate research support).

Nickel-Mediated Elimination of Hydrogen Halide from Primary and Secondary Alkyl Bromides and Iodides. Synthetic Aspects

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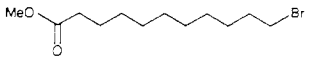
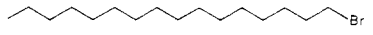
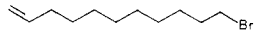
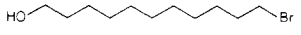
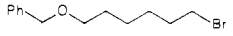
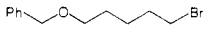
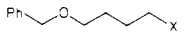
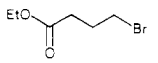
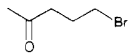
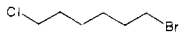
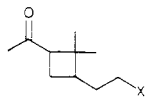
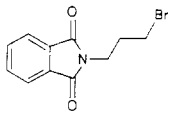
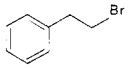
Treatment of alkyl bromides or iodides with a low-valent nickel complex generated in situ and 1,8-diazabicyclo[5.4.0]undec-7-ene in THF under argon leads on oxidative workup to alkenes. Primary halides give predominantly or exclusively the terminal alkene whereas acyclic secondary halides give a mixture. The thermodynamically most stable alkene is isolated from the cyclic congeners, 3α- and 3β-bromocholestane, and from cholesteryl bromide.

The elimination of hydrogen halide from alkyl bromides or iodides can be achieved under a number of experimental conditions.[1] Most often bases of varying strengths are used with or without considerable heating. Subsitution

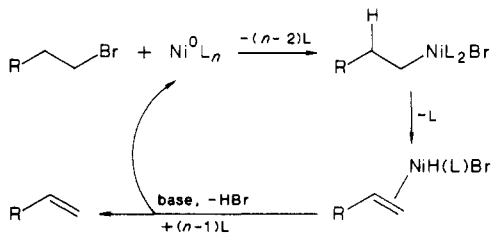
0022-3263/89/1954-3015$01.50/0

Table I. Elimination of Hydrogen Halide from Primary Substrates

entry	halide	yield, %	ratio term:int	isolation method
1		100	78:22	A
2		78	90:10	A, C
3		61	76:24	A
4		96	85:15	A, B
5		66	78:22	D
6		49	80:20	D
7		68 (X = Br) 56 (X = I)	100:0 100:0	A
8		77	0:100[a]	B
9		49	0:100[a]	B
10		20[b]	100:0	B
11		82 (X = Br) 50 (X = I)	100:0 100:0	A
12		43	100:0	C
13		≈50		

[a] Conjugation of the double bond occurs. [b] Some losses due to volatility.

Scheme I[a]



[a] L = ligand.

is a common accompaniment when using amine or alkoxide bases that are not sterically hindered, especially in the case of primary substrates.[2] A new approach to this type of elimination, which is devoid of substitution pathways, occurs under mild conditions, and is based on a familiar precept of organometallic chemistry, involves oxidative addition of the halide to a low-valent metal complex followed by β-hydride elimination (Scheme I). Herein we record the present scope of this reaction using nickel as the mediating metal.[3]

Results and Discussion

Generation of the Low-Valent Nickel Complex. We first needed a convenient method to produce nickel(0), preferably in situ so that transfer of air-sensitive materials would be avoided. Reduction of nickel(II) species to nickel(0) complexes is quite often accomplished by using aluminum alkyls in the presence of the appropriate ligand.[4] Since we were not interested in the isolation of the intermediate low-valent nickel species we were concerned that the aluminum chloride formed by such a procedure might not be compatible with a number of groups sensitive to Lewis acids. Hence, we routinely used *n*-BuLi for the reduction,[5] in the expectation that the resultant lithium chloride would be less harmful. We assumed that tetrakis(triphenylphosphine)nickel(0) would be formed in this step. Indeed, an authentic sample of this complex obtained commercially performed the elimination in almost identical fashion with that of the in situ experiment (cetyl bromide: yield 65% compared to 65–70%; ratio of internal to terminal olefin 1:6.9 compared to 0.8:7). Qualitatively there was little difference in the times of reaction, although the rates of oxidative addition in the two experiments have not be quantified. No oxidative addition of the cetyl bromide was observed when the nickel(1) reagent, ClNi-$(PPh_3)_3$, was used.

Primary Halides. In Table I are given examples of substrates that contain functional groups capable of surviving the reaction conditions. Thus, benzyl ether, ketone, ester, hydroxy, olefinic, phthalimido, and phenyl groups do not interfere with the elimination. Simple alkyl chlorides do not eliminate, neither do they interfere with reaction in a bromo–chloro compound (entry 10), the terminal alkene product of such a reaction showing no trace of bromine by mass spectrometry. 2-Phenethyl bromide

(1) Baciocchi, E. In *Chemistry of Halides, Pseudo-halides and Azides*; Patai, S., Rappoport, Z., Eds.; Wiley: New York, 1983; Part 2, pp 1173–1227.

(2) If the primary halide has a 2-alkyl side chain, nucleophilic attack by DBU is prohibited and elimination proceeds well; see: Wolff, S.; Huecas, M. E.; Agosta, W. L. *J. Org. Chem.* **1982**, *47*, 4358.

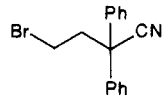
(3) For a preliminary communication of parts of this work, see: Jeropoulos, S.; Smith, E. H. *J. Chem. Soc., Chem. Commun.* **1986**, 1621.

(4) Schunn, R. A. *Inorg. Synth.* **1972**, *13*, 124. Jolly, P. W.; Jonas, K. *Inorg. Synth.* **1974**, *15*, 29.

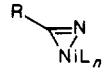
(5) Although our control experiment suggested that $Ni(PPh_3)_4$ is the active agent in the elimination reaction, we are not completely assured of that assumption; for example, for the unpredictable effects of alkyllithium species on *palladium*-catalyzed reactions, see: Negishi, E.; Akiyoshi, K.; Takahashi, T. *J. Chem. Soc., Chem. Commun.* **1987**, 477.

eliminates hydrogen bromide slowly with 1,5-diazabicyclo[5.4.0]undec-7-ene (DBU) alone but the reaction is faster when the nickel method is used (entry 13); the styrene produced appears to be stable toward polymerization under the reaction conditions.

Groups that are not compatible with the reaction conditions are aldehyde and cyanide. The cyanide 1 reacts



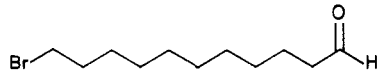
1



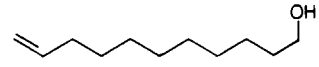
2

to give a crude product whose ^1H NMR spectrum indicates the absence of olefinic peaks. Starting material is recovered in low yield (33%) and the remainder of the product material (an orange oil) is comprised of a number of compounds by TLC, which together show C=N and NH bands, but no cyano stretch, in the IR spectrum. The known ability of nickel(0) to oxidatively add cyano groups to produce a nickelacyclopropene intermediate[6] as in 2 might explain this result providing the latter process was faster than oxidative addition to the C–Br bond.

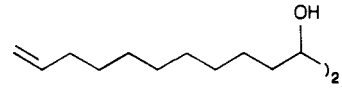
Bromo aldehyde 3 requires the use of 2 equiv of the nickel mediator and provides the desired alkene in approximately 40% yield together with a mixture of alcohols whose IR and ^1H NMR spectra are consistent with structures 4 and 5 (combined yield ≈ 10%).



3



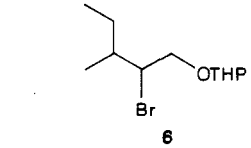
4



5

Synthetically the method suffers from a major disadvantage when the halide is an unbranched one (entries 1–6). Thus, in alkyl halides with a straight chain longer than four carbon atoms we observe a mixture of internal olefins contaminating the major terminal olefin. The extent of this contamination is maximal (11–32%) in chains longer than six. A GC/mass spectrometric investigation (Carlo-Erba GC, oven temperature = 140 °C, BPO5 capillary column, FID detector) of the products from cetyl bromide showed 1-hexadecene (m/e 224 = M^+, 7.8 min retention time identical with that of an authentic sample) plus internal alkenes (multiple peaks at retention time ≈ 7.6 min, m/e 224 = M^+), suggestive of random scrambling of the double bond along the chain (see below).

Secondary Bromides. It was quickly established that the reaction was not useful for the regiospecific synthesis of acyclic, internal olefins. Thus, the reaction of the bromotetrahydropyranyl (THP) ether 6 (in separate experiments using 50% 2*S*,3*S* and 50% 2*R*,3*S*, or 100% 2*S*,3*S*) leads (by a 500-MHz ^1H NMR spectrum of the crude products) to all the possible olefins, barring the enol ether, notwithstanding the branching in the chain. The ratio of the olefins depends slightly on the stereochemistry of the starting material, but otherwise there is little difference between the experiments. Considerable loss of material occurred in this reaction, presumably because of concomitant elimination of Br and OTHP to give the volatile 3-methyl-1-pentene.



6

(6) Bassi, I. W.; Benedicenti, C.; Calcaterra, M.; Intrito, R.; Rucci, G.; Santini, C. *J. Organomet. Chem.* **1978**, *144*, 225.

Scheme II

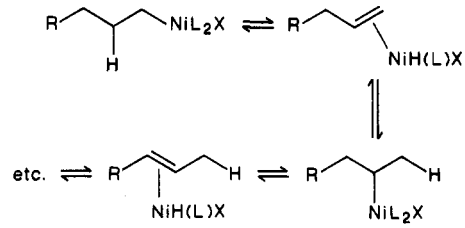
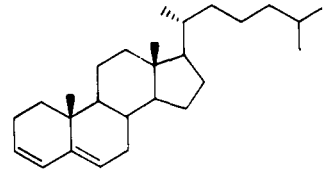


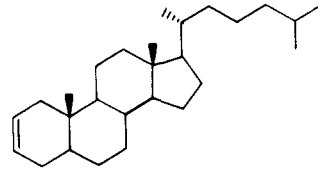
Table II. Effect of Base on the Elimination from Cetyl Bromide

amine	$pK_a(R_3NH^+)$	ratio term:int
N-methylimidazole	6.95	0:100
imidazole	7.05	0:100
DABCO	8.7	0:100
TMEDA	9.1	28:72
triethylamine	10.65	0:100
DBU	11.5	90:10
DBN		82:18
TMG	13.6	42:58
$Me_2NCH_2NMe_2$		0:100

In the cholesterol series the reaction proved to be more promising. Cholesteryl bromide gave the conjugated 3,5-diene 7 in 73% yield. The 3β- and 3α-bromocholestanes gave the same 2-cholestene (8) in 100% and 79% yields,



7



8

respectively. No evidence for the production of isomeric products could be found in these reactions and the thermodynamically most stable olefin is formed in each case.[7]

Influence of Base on the Reaction. In the absence of base the internal olefin isomers are obtained. No terminal olefin can be detected in the crude product. If we assume that the mechanism shown in Scheme I is correct, then presumably the former are produced by a reversal of the β-hydride elimination but with the opposite regiochemistry to give a secondary alkyl nickel complex that undergoes further elimination toward the center of the chain (Scheme II). It is emphasized here that this represents a working hypothesis at the moment although there is ample precedent for olefin isomerization by this mech-

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anism.[8] Thus the base hinders this process, albeit less effectively for long-chain substrates.

Also, the *structure* of the base is very important for the success of the reaction. Of a number of bases tried, only DBU, 1,5-diazabicyclo[4.3.0]non-5-ene (DBN), tetramethylguanidine (TMG), and tetramethylethylenediamine (TMEDA) gave the terminal olefin (Table II) from cetyl bromide. The pK_a of the conjugate base does not appear to be important since TMG is a stronger base than DBU but is only just over half as effective. A predominant feature of three of the successful bases is the presence of the amidine unit, $R_2NC{=}NR$. Whether this is the critical structural requirement for success and, if so, what is the means by which it achieves this success are the topics we are currently studying.

It should be noted that the method requires a stoichiometric amount of the nickel complex. The reaction could not be made catalytic in nickel to any great degree (when 10 equiv of cetyl bromide and 20 equiv of DBU were used per equiv of nickel complex the yield of all of the alkenes was 31% instead of 10%). We believe that this is due to the base, inhibiting recycling of the nickel possibly by complexation.

In summary the elimination method described herein is very useful for primary bromides or iodides in which the halogen does not terminate a long chain and for some cyclic secondary substrates. It is tolerant of quite a wide range of functionality and occurs under exceptionally mild conditions (cholesteryl bromide also eliminates at 0 °C with no loss in yield compared to normal reaction at room temperature). Despite the precedent for the mechanisms as written, we have little data to assume they are correct beyond the control reactions with authentic $Ni(PPh_3)_4$ and $ClNi(PPh_3)_3$.

Experimental Section

General Methods. Melting points are uncorrected and were determined on a Gallenkamp apparatus. Dry THF was obtained from potassium diphenylketyl under argon. Light petroleum ether (bp 60–80 °C) used for column chromatography was distilled. DBU was distilled periodically (once every 2 months suffices) in order to destroy contained carbonates that reduce yields of terminal alkenes. Merck 9385 and Sorbsil C60-40/60 (Crosfield Chemicals) silica gels were used for gravity column chromatography. Ether refers to diethyl ether.

IR spectra were recorded for samples either neat (liquids) or as Nujol mulls (solids) on Perkin-Elmer 298 and 881 spectrophotometers. ^1H NMR spectra were recorded on a Varian EM360A (60 MHz), JEOL FX90Q (90 MHz), Bruker WM250 (250 MHz), or JEOL GSX270 (270 MHz) spectrometer for solutions in $CDCl_3$ using TMS as internal standard. Mass spectra were recorded on an AEI MS9 or a VG Micromass 7070 instrument.

Product alkenes were identified by comparison of selected spectral data with those in the literature.

Dichlorobis(triphenylphosphine)nickel(II). Although the elimination can be done using the reagent prepared in glacial acetic acid according to the literature,[9] more reproducible results were obtained with the complex prepared by the following procedure: nickel(II) chloride hexahydrate (11.9 g, 50 mmol) was dissolved in 150 mL of absolute ethanol and the solution was concentrated to dryness on a rotary evaporator. This was repeated once more and then the residue was dissolved in 150 mL of absolute alcohol and treated with triphenylphosphine (26.2 g, 100 mmol). The mixture was boiled and stirred for 3 h when it became dark green. After cooling to room temperature, the dark green complex was filtered off, washed with absolute ethanol, and dried under vacuum.

Elimination Reaction. General Procedure. Dichlorobis(triphenylphosphine)nickel(II) (1 equiv) was suspended in dry THF (40 mL per mmol) containing triphenylphosphine (2 equiv). The suspension was subjected to four cycles of evacuation at room temperature by means of an oil pump followed by argon ingress to ensure removal of dissolved oxygen. A solution of *n*-butyllithium (1.6 M in hexanes, Aldrich, 2 equiv) was added dropwise to the stirred suspension under argon. After the addition the mixture was a rich red-brown color (if this color does not develop the reaction should be abandoned). A solution of the alkyl halide (1 equiv) and DBU (2 equiv, see General Method) in dry THF (10 mL per mmol) was degassed as above and added to the red-brown solution over 5 min. No color change occurred. The mixture was stirred overnight at room temperature and then exposed to air for 0.5 h. The solvent was removed on a rotary evaporator and the residue was treated in one of four ways to remove triphenylphosphine.

Method A. The residue was triturated with light petroleum ether (bp 40–60 °C) and the triturate was filtered through a pad of silica gel. The filtrate was concentrated to dryness and the resultant crude product was chromatographed on silica gel using light petroleum ether.

Method B. The residue was subjected to Kugelrohr distillation and the product was collected as the distillate.

Method C. The residue was triturated with petroleum ether (bp 40–60 °C) and the triturate was treated with excess methyl iodide. The mixture was left to stand overnight at room temperature and then concentrated on the rotary evaporator. The concentrate was triturated with 1:1 ether:petroleum ether (bp 40–60 °C) and filtered. The filtrate was passed through a short silica gel pad and concentrated to the product.

Method D. The residue was dissolved in 1:1 ether:petroleum ether (bp 40–60 °C), passed through a pad of silica gel, and concentrated to dryness. The concentrate was dissolved in THF and stirred overnight at room temperature with 30% H_2O_2. The mixture was evaporated carefully to remove THF and poured into excess water and the product was extracted with ether.

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Supplementary Material Available: Experimental data for compounds synthesized in this study (5 pages). Ordering information is given on any current masthead page.

Registry No. **1**, 39186-58-8; **3**, 70326-40-8; **4**, 112-43-6; **5**, 120446-58-4; (2*S*,3*S*)-**6**, 120446-59-5; (2*R*,3*S*)-**6**, 120522-42-1; **7**, 747-90-0; **8**, 15910-23-3; DABCO, 280-57-9; TMEDA, 110-18-9; DBU, 6674-22-2; DBN, 3001-72-7; TMG, 80-70-6; $MeOCO(CH_2)_{10}Br$, 6287-90-7; $C_{16}H_{33}Br$, 112-82-3; $CH_2{=}CH(CH_2)_9Br$, 7766-50-9; $HO(CH_2)_{11}Br$, 1611-56-9; $PhCH_2O(CH_2)_6Br$, 54247-27-7; $PhCH_2O(CH_2)_5Br$, 1014-93-3; $PhCH_2O(CH_2)_4Br$, 60789-54-0; $PhCH_2O(CH_2)_4I$, 50873-94-4; $EtOCO(CH_2)_3Br$, 2969-81-5; $CH_3CO(CH_2)_3Br$, 3884-71-7; $Cl(CH_2)_6Br$, 6294-17-3; $Ph(CH_2)_2Br$, 103-63-9; $MeOCO(CH_2)_8CH{=}CH_2$, 111-81-9; $MeOCOC_{10}H_{19}$, 66573-84-0; $C_{14}H_{29}CH{=}CH_2$, 629-73-2; $C_{16}H_{32}$, 26952-14-7; $CH_2{=}CH(CH_2)_7CH{=}CH_2$, 13688-67-0; $CH_2{=}CHC_9H_{17}$, 29825-95-4; $HOCH_2C_{10}H_{19}$, 25377-71-3; $PhCH_2O(CH_2)_4CH{=}CH_2$, 59137-50-7; $PhCH_2OCH_2C_5H_9$, 120446-60-8; $PhCH_2O(CH_2)_3CH{=}CH_2$, 81518-74-3; $PhCH_2OCH_2C_4H_7$, 120446-61-9; $PhCH_2O(CH_2)_2CH{=}CH_2$, 70388-33-9; (*E*)-$EtOCOCH{=}CHCH_3$, 623-70-1; (*E*)-$CH_3COCH{=}CHCH_3$, 3102-33-8; $Cl(CH_2)_4CH{=}CH_2$, 928-89-2; $PhCH{=}CH_2$, 100-42-5; $Cl_2Ni(PPh_3)_2$, 14264-16-5; $Ni(PPh_3)_4$, 15133-82-1; (*S*)-$CH_2{=}CHCH(CH_3)C_2H_5$, 5026-95-9; NEt_3, 121-44-8; $Me_2NCH_2NMe_2$, 51-80-9; 1-acetyl-2,2-dimethyl-3-(2-bromoethyl)cyclobutane, 120446-57-3; 1-acetyl-2,2-dimethyl-3-(2-iodoethyl)cyclobutane, 109682-69-1; *N*-(3-bromopropyl)-phthalimide, 5460-29-7; 1-acetyl-2,2-dimethyl-3-vinylcyclobutane, 109682-70-4; *N*-allylphthalimide, 5428-09-1; cholesteryl bromide, 516-91-6; 3β-bromocholestane, 51154-61-1; 3α-bromocholestane, 2309-03-7; *N*-methylimidazole, 616-47-7; imidazole, 288-32-4.

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