3 H, $OCH_3$), 3.2 (m, 2 H, $CH_2$); CIMS $m/z$ (relative intensity) 290 ($M^+ + 1 - HCl$) (100). Anal. Calcd for $C_{16}H_{17}Cl_2NO_2$: C, 58.91; H, 5.25; N, 4.29; Cl, 21.73. Found: C, 58.79; H, 5.25; N, 4.04; Cl, 22.05.

**(*S*)-4-(1-Naphthalenyl)phenylalanine methyl ester hydrochloride salt (4d)**: mp 176-177 °C; $[\alpha]^{20}_D$ +9.41° ($c$ = 1, $CH_3OH$); $^1H$ NMR ($CDCl_3$) $\delta$ 8.77 (s, 3 H, $NH_3$), 8.0 (dd, $J$ = 8.1, 8.2 Hz, 2 H, NapH), 7.8 (d, $J$ = 8.0 Hz, 1 H, NapH), 7.35-7.6 (m, 8 H, ArH + NapH), 4.35 (t, $J$ = 6.2 Hz, 1 H, $\alpha$-CH), 3.72 (s, 3 H, $OCH_3$), 3.26 (m, 2 H, $CH_2$); CIMS $m/z$ (relative intensity) 306 ($M^+ + 1 - HCl$) (100). Anal. Calcd for $C_{20}H_{20}ClNO_2$: C, 70.27; H, 5.90; N, 4.10; Cl, 10.37. Found: C, 70.27; H, 5.90; N, 4.10; Cl, 10.56.

**(*S*)-4-(2-Furanyl)phenylalanine methyl ester hydrochloride salt (4e)**: mp 207-208 °C; $[\alpha]^{20}_D$ +16.36° ($c$ = 1, $CH_3OH$); $^1H$ NMR ($CDCl_3$) $\delta$ 8.67 (s, 3 H, $NH_3$), 7.75 (d, $J$ = 1.6 Hz, 1 H, $Fur_5H$), 7.67 (d, $J$ = 8.1 Hz, 2 H, $Ar_3H$), 7.27 (d, $J$ = 8.1 Hz, 2 H, $Ar_2H$), 6.95 (d, $J$ = 3.2 Hz, 1 H, $Fur_3H$), 6.57 (dd, $J$ = 3.1, 1.8 Hz, 1 H, $Fur_4H$), 4.3 (t, $J$ = 6.4 Hz, 1 H, $\alpha$-CH), 3.68 (s, 3 H, $OCH_3$), 3.15 (m, 2 H, $CH_2$); CIMS $m/z$ (relative intensity) 246 ($M^+ + 1 - HCl$) (100). Anal. Calcd for $C_{14}H_{16}ClNO_3$: C, 59.68; H, 5.72; N, 4.97; Cl, 12.58. Found: C, 59.47; H, 5.60; N, 4.86; Cl, 12.37.

**Typical Procedure for the Determination of the Enantiomeric Purity of Amino Ester.** The amino ester **4a** was derivatized with 2,3,4,6-tetra-*O*-acetyl-$\beta$-D-glucopyranosyl isothiocyanate (GITC) by the procedures reported by Kinoshita[9] to form thio urea diastereomers. A 5-$\mu$L aliquot was injected directly into the Waters HPLC system equipped with a C-18 column (Waters $\mu$Bondapak, 300 × 3.9 mm). The sample was eluted with an isocratic solution of aqueous ammonium formate (0.025M, pH = 4) and methanol, 40/60 (v/v), at a constant flow rate of 1.2 mL/min at room temperature and was monitored at 254 nm. A typical retention time for an (*S*)-**4a** derivative is 33 min and for an (*R*)-**4a** derivative is 40 min. The limit of detection of each diastereomer monitored at 254 nm is less than 1% by weight.

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**Registry No.** **1**, 112766-18-4; **2a**, 98-80-6; **2b**, 5720-05-8; **2c**, 1679-18-1; **2d**, 13922-41-3; **2e**, 13331-23-2; **3a**, 137255-86-8; **3b**, 137255-87-9; **3c**, 137255-88-0; **3d**, 137255-89-1; **3e**, 137255-90-4; **4a**, 63024-30-6; **4b**, 137255-91-5; **4c**, 137255-92-6; **4d**, 137255-93-7; **4e**, 137255-94-8; Boc-Tyr-OMe, 4326-36-7; $O(SO_2CF_3)_2$, 358-23-6; Pd, 7440-05-3.

## Chromium(VI) Oxidation of Tertiary Unsaturated Alcohols. Oxidative Fragmentation of 2-Substituted Bicyclo[2.2.1]hept-5-en-2-ols

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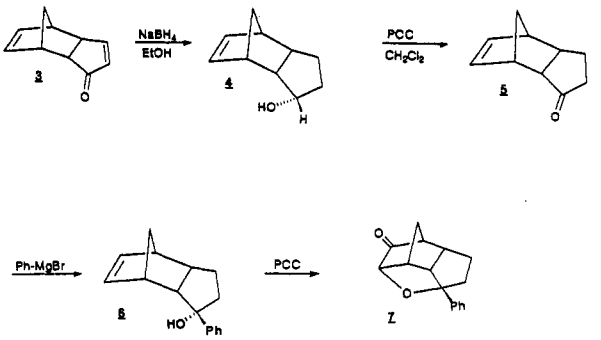
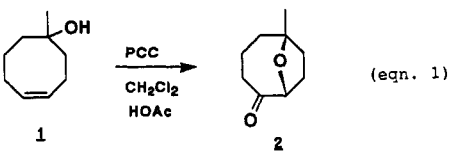
Tertiary alcohols are generally inert toward oxidation by Cr(VI) reagents.[1] Some exceptions, however, have been reported such as strained cyclopropanols[2] and bicyclic [2.2.1] tertiary alcohols[3] which react with chromic acid to yield ketones resulting from C-C bond cleavage. When an alkene double bond is suitably disposed near the tertiary OH site, Schlecht has observed synthetically useful intramolecular C=C oxidations initiated by pyridinium chlorochromate (PCC), for example **1** → **2**.[4] Furthermore, tertiary allylic alcohols react with PCC to give conjugated ketones through allylic rearrangements.[5] Since the mechanisms of some of these reactions remain in doubt and since the synthetic potential of tertiary alcohol oxidations has not been fully exploited, we became interested in investigating further examples of PCC oxidation of tertiary unsaturated alcohols.

**Scheme I**




(eqn. 1)

### Results and Discussion

One such compound is 3-phenyltricyclo[5.2.1.$0^{2,6}$]dec-8-en-*endo*-3-ol (**6**) prepared from the known ketone **3**[6] according to Scheme I. Thus, $NaBH_4$ reduction of **3** yielded the alcohol **4** in which the conjugated double bond of **3** was also saturated.[7] PCC oxidation of **4** followed by PhMgBr treatment gave **6**, where the endo orientation of the OH group is assured since the nucleophilic Grignard reagent must attack from the least hindered exo face of the tricyclic ketone **5**.[8] Within the carbon framework of **6**, the tertiary OH is positioned in a conformationally fixed orientation with respect to the alkene group (in contrast to **1**). As a consequence, we might expect PCC oxidation of **6** to proceed smoothly to a $\beta$ keto ether in the manner of the **1** to **2** conversion.[4] Indeed, oxidation of tertiary unsaturated alcohol **6** gave crystalline **7**, $C_{16}H_{16}O_2$, in 70% yield. This compound was fully characterized, and spectral data are listed in the Experimental Section.

The mechanism of this reaction very likely parallels the concept elegantly demonstrated by Schlecht for PCC oxidation of substrates such as **1**. Here, the chromate ester of the OH group rapidly forms and participates in a transannular, intramolecular alkene oxidation.[4] Since the strained double bond of **6** might be more reactive toward PCC than the same group in **1**, we have considered the possibility of an intermolecular attack of PCC on the 8-ene

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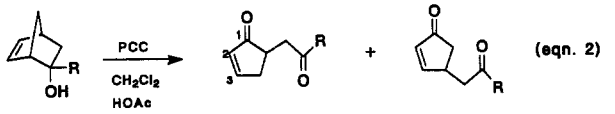
group of 6. However, in a control experiment, we observed that the 8-ene of dicyclopentadiene reacts only very slowly with PCC under identical conditions to give a 25% conversion to α-chloro ketones (C-8/9 regioisomers).[9] Thus, although an intermolecular mechanism cannot be ruled out, it would not seem to account for the clean production of 7.

In the bicyclic tertiary alcohols 8 and 9, the endo OH is again (as in 6) conformationally fixed with respect to the 5-ene double bond. However, in this case, if the mechanism follows the same track as 1 → 2 and 6 → 7, a highly strained 5-keto-2,6-*oxetane* product would be expected. Parenthetically, we have observed 2,6-oxetanes to form readily in related compounds.[10]




8: R = $C_6H_5$
9: R = n-butyl

10 : R = $C_6H_5$
11 : R = n-butyl

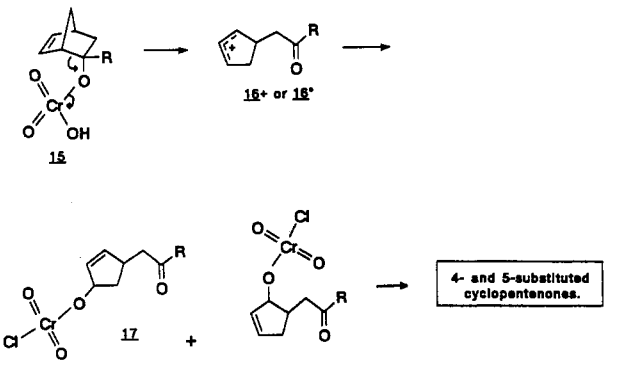
12 : R = $C_6H_5$
13 : R = n-butyl

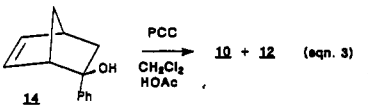
When 2-phenylbicyclo[2.2.1]hept-5-en-2-ol (8) was treated with PCC under Schlecht's conditions,[4] a crystalline product $C_{13}H_{12}O_2$ was obtained in 62% yield. At first we considered this product to be a single cyclopentenone isomer (e.g., 10) on the basis of IR, MS, and NMR (60 MHz) data and on its sharp melting point and homogeneity on TLC (see Experimental Section). However, upon close examination of the 200-MHz $^1H$ NMR and $^{13}C$ NMR spectra, it became clear that isomers 12 and 10 were both present in a ratio of 1.7:1. Particularly, the H-2 region under high resolution revealed two signals at δ 6.22 (dd, $J$ = 2, 5.6 Hz) and 6.29 (multiplet) in a peak area ratio of 1.7:1. This is exactly what would be expected for a mixture of 12 (major) and 10 (minor).[11] The $^{13}C$ NMR spectrum of 12/10 is completely consistent. For example, major/minor signals were clearly seen for C-3 at δ 168.1 and 164.5. In addition, for each of the two $CH_2$ groups and the one CH that the structure demands, a minor signal accompanied the major. The Experimental Section lists all chemical shifts and gives a description of an APT experiment which confirmed $^{13}C$ NMR peak assignments.

PCC oxidation of the *n*-butyl analogue 9 gave a liquid product in 73% yield. This material is also an inseparable mixture of the corresponding isomers 13 (major) and 11 (minor). Now, both alkene protons H-2 and H-3 are clearly visible at δ 6.20 and 7.70. Each signal is represented by a doubled doublet (major) and a slightly downfield multiplet (minor), and this is exactly what is expected of a 13 (major)/11 (minor) mixture.[11]

The following observations will allow us to formulate a likely mechanism for these oxidative fragmentations. (1) Chromate esters rapidly form with tertiary alcohols.[12] (2) PCC alone only very slowly attacks an alkene C=C under the reaction conditions[4] (and vide infra). (3) The rearrangement step requires PCC since a control run without PCC gave recovered starting material. (4) The 2,6-oxetane is not an intermediate since if it were, the O–$C_2$ bond would break giving the tertiary (benzylic) carbocation leading to products not observed in the present case.[13] (5) Significantly, PCC oxidation of the exo alcohol 14[14] gives the same fragmentation products 12 and 10 in good yield. Thus, an intramolecular oxidation of the 5-ene (analogous to 1 → 2) is not involved.

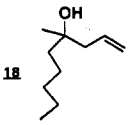
**Scheme II**




14

Taking into account these observations along with the nature of our product mixtures, we propose the mechanism illustrated in Scheme II. Here the required chromate ester 15 suffers fragmentation to give the allylic carbocation 16. Capture of 16 by a chromate species and subsequent elimination leads to the 4- and 5-substituted cyclopentenones. It is noteworthy that this mechanism also explains the observed product ratio since 16 is not symmetrical and capture of the chromate unit at the least hindered positive center gives 17 and the 4-substituted isomer as the major product. An analogous one-electron process passing through an allylic *radical* remains possible, although the formation of an alkoxy radical is unlikely in view of the stability of 18[10] toward the reaction conditions.



18

In summary, although the cleavage of strained ring alcohols during Cr(VI) oxidation is known[1,2] such a process on tertiary norbornenols appears to be new, as do the product compounds 10–13. Furthermore, we note that the carbon framework and functionality of 10–13 seem very appropriate for subsequent conversion to novel prostaglandin derivatives.[15]

## Experimental Section

NMR spectra were obtained on a Nicolet NT 200-MHz, a Bruckner AC 250, or a JEOL JNM-PMX 60 instrument. Analytical TLC made use of Merck precoated silica gel plates (0.25 mm). The pyridinium chlorochromate (PCC) reagent is available from Aldrich Chemical Co. Organic solutions were dried over anhydrous $MgSO_4$.

The starting tertiary alcohols 8 and 9 are known compounds[16] synthesized by addition of the appropriate Grignard reagent to dehydronorcamphor.[17]

**Tricyclo[5.2.1.$0^{2,6}$]dec-8-en-*endo*-3-ol (4).** To 0.958 g (6.56 mmol) of the ketone 3[6] in 30 mL of EtOH was added 0.50 g (13.2 mmol) of $NaBH_4$. The reaction mixture was stirred at rt for 2.3 h, diluted with 3% NaOH, and extracted with ether (3 × 35 mL). The combined ether layer was washed with water, dried, filtered,

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and concentrated by rotary evaporation to give 0.831 g (86%) of **4**, mp 127–9 °C; TLC (1:1 ether/hexane) $R_f$ = 0.4; IR ($CHCl_3$) 3360, 1550 $cm^{-1}$; $^1H$ NMR ($CDCl_3$) δ 6.20 (m, 2 H), 4.26 (broad m, 1 H), 2.3–3.0 (complex, 5 H), 1.0–2.0 (complex, 6 H).

**3-Phenyltricyclo[5.2.1.0$^{2,6}$]dec-8-en-*endo*-3-ol (6).** To a solution of 0.831 g (5.54 mmol) of **4** in 50 mL of $CH_2Cl_2$ with stirring was added 1.795 g of PCC (8.33 mmol), 15 drops of HOAc, and 3.6 g of Celite. The reaction slurry was stirred at rt under $N_2$ for 22 h, then diluted with ether, and passed through a column of neutral alumina (10 × 2.2 cm) with exhaustive ether washing. The combined ether solution (200 mL) was concentrated to a small volume and used directly in the next step.

To a stirred solution of the ketone **5** in 40 mL of dry ether at 0 °C was slowly added via syringe 2.7 mL of 3.0 M PhMgBr (8.1 mmol) in ether. The reaction mixture was stirred at 0 °C for 1 h ($CaCl_2$ drying tube) then for an additional 1 h at rt. Again at 0 °C, 10 mL of saturated $Na_2SO_4$ was slowly added with continued stirring. The mixture was gravity filtered with ether washing, dried, filtered, and evaporated to yield 0.892 g of a pale yellow oil. This crude product was purified by chromatography on a column of silica gel (2.8 × 24 cm) prepared in and eluted with 10% acetone in hexane, collecting 10-mL fractions. Fraction numbers 7–13 were evaporated to give crystalline **6**, mp 66–71 °C, 0.517 g (45%): IR (film) 3430, 1635, 1595, 1575, 1490, 1440 $cm^{-1}$; EIMS (*m/e*, base peak) 226 ($M^+$) (4.6), 208 (4.6), 165 (5.5), 142 (100), 115 (20.2), 105 (12.8), 91 (12.8), 77 (24.8), 66 (40); $^1H$ NMR ($CDCl_3$) (in part) δ 7.4 (complex, 5 H), 6.33 (m, 2 H).

**PCC Oxidation of 6.** A solution of a 0.208 g sample of **6** (0.923 mmol) in 20 mL $CH_2Cl_2$ was stirred at rt as 0.571 g of PCC (2.65 mmol), five drops of HOAc, and 1.8 g of Celite were added in succession. This suspension was stirred and refluxed for 4 h under $N_2$ then stirred at rt overnight. The reaction was worked up as described above to give the β keto ether **7**, 0.155 g (70%). The solid material was recrystallized from ether/petroleum ether, mp 95.5–97.5 °C: IR (film) 1760 $cm^{-1}$ (no OH); EIMS (*m/e* base peak): 240 ($M^+$) (56), 212 (53), 183 (21), 155 (24), 143 (100), 128 (20), 115 (25), 105 (7.7), 91 (20), 77 (25); $^1H$ NMR ($CDCl_3$) δ 7.5–7.20 (m, 5 H, ArH), 4.10 (d, *J* = 5.4 Hz, 1 H, H-9), 3.2–2.90 (m, 3 H), 2.55 (d, *J* = 3.8 Hz, 1 H), 2.30 (m, 3 H), 2.10 (m, 1 H), 2.0–1.80 (q, *J* = 12.8 Hz 2 H). Anal. Calcd for $C_{16}H_{16}O_2$: C, 80.00; H. 6.67. Found: C, 79.88; H.6.76.

**4- and 5-(2-Oxo-2-phenylethyl)-2-cyclopentenones (10 and 12).**[4] To a solution of 0.304 g (1.63 mmol) of **8** in 20 mL of $CH_2Cl_2$ were added 1.04 g (4.82 mmol) of PCC and 2.0 g of Celite. This slurry was charged further with six drops of acetic acid, and the resulting mixture was stirred and refluxed under $N_2$ for 4 h then stirred overnight at rt. The reaction slurry was diluted with ether and passed through a column of neutral alumina (10.3 × 2.2 cm) with exhaustive ether washing. The total ether filtrate was evaporated to give the crystalline product (0.201 g, 63%), one spot on TLC ($R_f$ 0.7, 5% acetone in ether), mp 84–6 °C, positive 2,4-DNP test: IR ($CHCl_3$) 1710, 1685, 1600, 1585 $cm^{-1}$; $^1H$ NMR ($CDCl_3$) δ 7.96 (m, 2 H, ortho H), 7.74 (dd, *J* = 2.5, 5.6 Hz, 1 H, H-3), 7.66–7.40 (complex, 3 H, ArH), 6.29 (m, 0.37 H, H-2, isomer **10**), 6.22 (dd, *J* = 2.0, 5.6 Hz, 0.62 H, H-2, isomer **12**), 3.60 (1 H, methine); the spectrum between δ 3.2–2.0 was very complex due to the presence of **12**/**10**, but consistent with the structural assignments; UV $\lambda_{max}$ 239 (COPh), 220 (sh) nm; EIMS (*m/e*, base peak): 200 (42) ($M^+$), 105 (100), 95 (24), 77 (50). Anal. Calcd for $C_{13}H_{12}O_2 \cdot 0.2\ H_2O$; C, 76.62; H, 6.09. Found: C, 76.64; H, 5.92.

The $^{13}C$ NMR spectrum shows more than 13 signals due to the presence of the **12** (major)/**10** (minor) isomeric mixture: δ 209.9, 198.4 (C=O), 168.1, 164.5 (H-3), 137.2, 135.2, 134.4, 134.2, 129.6, 129.5, 128.8 (H-2, aromatic) 44.0, 42.2, 41.9, 41.6, 37.8, 37.3 ($CH_2$, CH). An attached proton test (APT) was performed which is consistent with the structural assignments. Particularly, in the δ 44.0–37.0 region, the six signals were revealed as 2 × $CH_2$ and 1 × CH, each with a corresponding minor signal associated with it, representing the accompanying isomer. All other carbon signals also behaved as expected.

**4- and 5-(2-Oxo-2-hexyl)-2-cyclopentenone (11 and 13).** The identical procedure [4] was followed for **9** to give the product as a clear oil (0.218 g, 73%), one spot on TLC ($R_f$ 0.75, 5% acetone in ether)(trace starting material): IR (neat) 1710, 1590 $cm^{-1}$; UV $\lambda_{max}$ 219 nm(cyclopentenone); EIMS (*m/e*, base peak): 180 (96)($M^+$), 138 (59), 123 (71), 95 (100), 85 (73), 81 (23), 67 (33), 57 (51); $^1H$ NMR ($CDCl_3$) δ 7.74 (m, 0.37 H) 7.67 (dd, *J* = 2.4, 5.6 Hz, 0.62 H, H-3), 6.23 (m, 0.37 H), 6.17 (dd, *J* = 2.2, 5.8 Hz, 0.62 H, H-2), 3.40 (m, 1 H), 3.00 (m, 1 H), 2.50 (complex, 3 H), 1.92 (dd, *J* = 1.9 Hz, 1 H), 1.58 (complex, 2 H), 1.32 (complex, 2 H), 0.90 (2 t, 3 H, $CH_2CH_3$).

The above procedure run on tertiary alcohol **9** but without PCC gave 84% recovered starting material. In another control experiment, this procedure run on **18**[10] gave 82% recovered starting material.

***endo*-2-Phenylbicyclo[2.2.1]hept-5-en-*exo*-2-ol(14) was** synthesized according to the method of Brown and Peters[14] and reacted with PCC as described above. The product (75% yield) was a mixture of **12** (major) and **10** (minor).

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# Novel Preparation of Highly Electrophilic Species for Benzenetellurenylation or Benzenesulfenylation by Nitrobenzenesulfonyl Peroxide in Combination with Ditelluride or Disulfide. Application to Intramolecular Ring Closures

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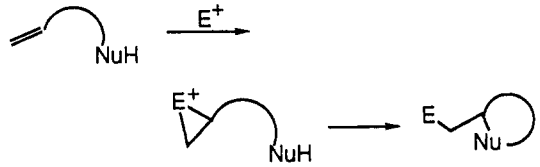
Taking advantage of the strong electrophilicity of nitrobenzenesulfonyl peroxide (NBSP), various synthetic reactions have been developed.[1-4] Though NBSP is known to attack the π-bond of simple olefins electrophilicially,[1,2] intramolecular cyclization did not occur with 4-penten-1-ol using *p*-nitrobenzenesulfonyl peroxide (*p*-NBSP) or *m*-nitrobenzenesulfonyl peroxide (*m*-NBSP) as an electrophilic promoter for the cyclization. However, we found that the electrophilic intramolecular ring closures of unsaturated alcohols and acids were effected by NBSP in combination with diphenyl diselenide in excellent yields.[5] Ring closures proceeding by intramolecular capture of the cationic intermediate initiated by suitable electrophilic reagents (represented by eq 1) are useful methods for the




(1)

synthesis of natural products.[6] In the course of our studies

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