2 L of water was added 200 g of chromium trioxide. Formation of the reagent occurred very smoothly and no heat was evolved as the mixture was stirred for several hours. After filtration, the resin was washed repeatedly with water until the filtrate was colorless. In general, the reagent was used without drying. However, the reagent could also be dried in vacuo at 50 °C for storage (typically, 1 g of PVP affords 1.9 g of the dry PVPDC). The dry reagent was found to perform very poorly in oxidation reactions, but its activity could be restored completely by soaking in water prior to use.

Titration[2] of the reagent showed that it contained 14 mequiv of oxidizing agent per gram (or 2.3 mmol of $(PVPH)_2{}^+Cr_2O_7{}^{2-}$ per gram). This indicated that >98% of the pyridine units have been converted into dichromate units. The infrared spectrum of the dry reagent showed bands at 930 and 765 cm^{-1}, characteristic of dichromate ion.[5] Anal. Calcd: Cr, 24.28; N, 6.54. Found: Cr, 23.93; N, 6.67.

Oxidation of Alcohols with PVPDC. The standard procedure involved the reaction of 1.9 g of PVPDC with 5–8 mmol of alcohol in 10 mL of cyclohexane or hexane at 70 °C. The progress of the reaction was monitored by gas chromatography. After completion of the reaction the products could be obtained in the soluble phase by washing of the resin. Scaling up of the reaction to oxidize mole quantities of alcohols could be done easily. Thus, 266 g of PVPDC made from 140 g of poly(vinylpyridine) resin was suspended in 1 L of cyclohexane and 122 g (1 mol) of 1-phenylethanol was added. The reaction mixture was stirred at 65 °C and reaction proceeded rapidly and then slowed down as it neared completion. After 48 h, chromatographic analysis showed no starting material remaining. The reaction mixture was filtered and the spent resin was washed with cyclohexane, ether, and dichloromethane. After distillation of the filtrate, 113 g (94% yield) of pure ketone was obtained.

Although cyclohexane was the solvent of choice, hexane was used in reactions in which low-boiling aldehydes were produced to avoid interference of the solvent peak in the GC analysis. In addition, sealed tubes were used as reaction vessels in these cases to prevent loss of the volatile products.

Titration of spent reagent used to oxidize 8 mmol of benzyl alcohol showed that 18 mequiv of oxidizing agent had been consumed, a value which compares favorably with the theoretical value[8] of 16 mequiv.

Recyling of PVPDC. The dark spent resin could be easily recycled by repeated sequential washings with 2 N HCl and 2 N NaOH until the PVP polymer had returned to its original white color, drying, and reacting with CrO_3 to regenerate the reagent. A 200-g batch of PVP used in a reaction such as the one above was recycled and reused in the oxidation of 1 mol of cyclohexanol to produce a 91% conversion to cyclohexanone. Reactions on a smaller scale were also quite effective with easy recycling of the polymer after each use. For example, a 1-g batch of PVP resin was carried through five successive cycles of formation of reagent, oxidation of 8 mmol of benzyl alcohol, washing, and regeneration with no loss of oxidizing capability and only a small decrease in reaction rate. In addition, the weight of the polymer remained essentially constant throughout, the small variations being due to the varying amounts of chromium salts left in the resin after washing.

Acknowledgment. Financial support of this investigation by the Natural Science and Engineering Research Council of Canada is gratefully acknowledged. In addition thanks are due to Dr. G. Gelbard for a fruitful discussion.

Registry No. Benzyl alcohol, 100-51-6; 1-phenylethanol, 98-85-1; cinnamyl alcohol, 104-54-1; cyclopentanol, 96-41-3; cyclohexanol, 108-93-0; 3-pentanol, 584-02-1; 1-butanol, 71-36-3; 1-hexanol, 111-27-3; benzaldehyde, 100-52-7; 1-phenylethanone, 98-86-2; 3-phenyl-2-propenal, 104-55-2; cyclopentanone, 120-92-3; cyclohexanone, 108-94-1; 3-pentanone, 96-22-0; butanal, 123-72-8; hexanal, 66-25-1; 4-vinylpyridine–divinylbenzene copolymer, 9017-40-7; chromium trioxide, 1333-82-0.

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(8) Gelbard (CNRS, Solaise, France) has informed us that reduction of the polymer-bound chromate or chloroformate may in fact produce Cr^{IV} rather than Cr^{III}. We have made no attempt to characterize fully the reduced species bound to the polymer.

A Novel Rearrangement of Tricyclo[4.2.2.0^{2,5}]deca-3,7-dien-9-ones to Functionalized Bicyclo[3.2.1]octa-2,6-dienes

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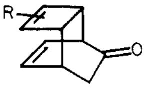
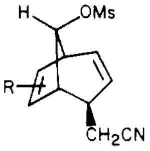
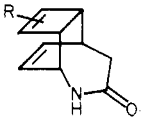
The bicyclo[3.2.1]octa-2,6-diene framework is endowed with many structural features that make it an attractive substrate for synthetic and mechanistic investigations.[1] Conventionally, synthetic entry to this ring system and its derivatives is gained either from bicyclo[2.2.1]hepta-2,5-diene (norbornadiene) via a dihalocarbene addition–ring-expansion sequence[2] or through the thermal [$_\pi2_s$ + $_\sigma2_s$ + $_\pi2_s$] (Cope) rearrangement of 6-vinylbicyclo[3.1.0]hex-2-enes.[3] In this communication, we describe a novel entry to the bicyclo[3.2.1]octa-2,6-diene ring system from tricyclo[4.2.2.0^{2,5}]deca-3,7-dien-9-ones (I) and (II) via Schmidt fragmentation and carbonium ion rearrangement. The interesting and useful aspect of this rearrangement is the ready availability[4] of precursors I and II and formation of bicyclo[3.2.1]octanes carrying functionality in all three bridges and in particular at C_8.



I, R = H
II, R = Br



IV, R = H
VIII, R = Br



V, X = H
X, X = Br

Reaction of the tricyclic dienone I, prepared according to the sequence depicted in Scheme I from the COT–acrylonitrile adduct[5] (III), with sodium azide (1 mol equiv) in methanesulfonic acid (MsOH)–methylene chloride and chromatography of the resulting reaction mixture led to the isolation of IV (55%) and V (22%). While the structure of the minor product (V, mp 178 °C), a lactam, could be readily deduced in a straightforward manner, the formulation of IV (mp 65–66 °C) necessitated an incisive analysis of the spectral data. The elemental composition, $C_{11}H_{13}O_3SN$, and IR spectrum of the major product (IV) indicated the presence of a cyano (2250 cm^{-1}) and mesylate ester (1360, 1180 cm^{-1}) moiety. The ^1H NMR spectrum indicated the presence of four olefinic protons at δ 6.25 (1 H, dd, J_1 = 6 Hz, J_2 = 3 Hz), 5.75–6.15 (2 H, m), and 5.64 (1 H, $^1/_2$AB, J = 10 Hz) and a proton attached to a carbon bearing the mesylate group at δ 5.05 (1 H, t, J = 5 Hz) besides other expected resonances (see the Experimental

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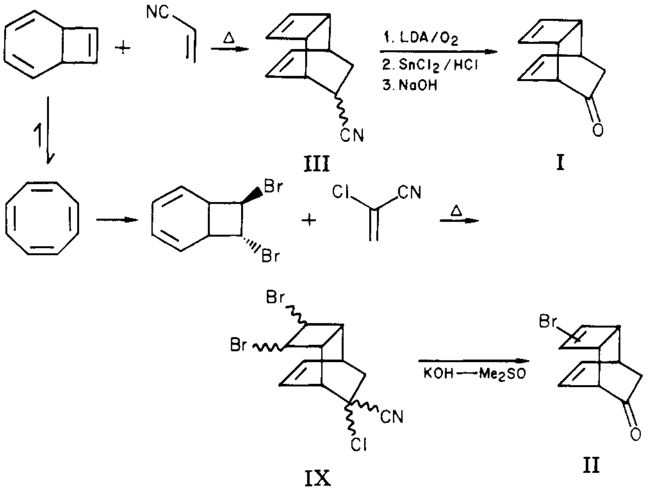
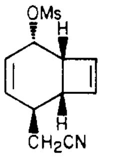
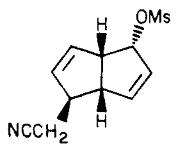
Scheme I



Section). In conformity with these structural features, the ^{13}C NMR spectrum displayed resonances attributable to four olefinic carbons at δ 139.0 (d), 129.5 (d), 128.1 (d), and 127.5 (d) and a carbon bearing the mesylate group at δ 78.4 (d). These spectral data are compatible with any of the structures IV, VI, and VII, derivable from I through reasonable mechanistic deductions. A distinction between these alternatives was achieved with the aid of ^{1}H NMR double-resonance experiments. For example, irradiation of the triplet due to the proton attached to the carbon bearing the mesylate group at δ 5.05 produced no significant change in the olefinic proton resonances. Similarly, irradiation of the olefinic proton signals in the region δ 5.7–6.3 made little impression on the proton attached to the carbon bearing the mesylate group at δ 5.05 and corroborated the earlier observation that the proton attached to the mesylate-bearing carbon was not coupled with the olefinic protons. Clearly, this observation can not be reconciled with formulations VI and VII. Furthermore, it was shown by the double-resonance experiments that the multiplicity of the proton attached to the mesylate-bearing carbon arose from coupling with the protons in the region δ 2.6–3.1, which is again indicative of the disposition present in IV rather than in systems VI and VII. Finally, a close scrutiny and comparison of ^{13}C NMR chemical shifts with those reported for various bicyclo[3.2.1]octa-2,6-dienes[6] and bicyclo[3.3.0]octa-2,6-dienes[7] led to unambiguous formulation IV for the major product obtained from I. The stereochemistry of the mesylate group in IV follows from mechanistic considerations (vide infra) and more securely from the ^{1}H NMR coupling constant of 5 Hz between the C_8 exo proton and the bridgehead protons at C_1 and C_5. It is well established[1b,2,8] that the *exo*-C_8-H and C_1-H or C_5-H coupling constants are of the order of 4–5 Hz compared to the *endo*-C_8-H and C_1-H or C_5-H coupling which is much smaller (0–1 Hz) in the bicyclo[3.2.1]octane system.

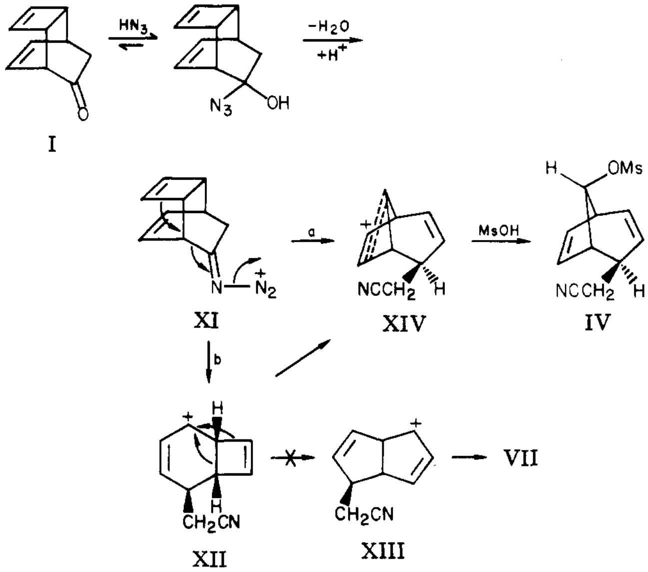


VI



VII

Scheme II



The generality of the I to IV type of transformation was further demonstrated through the rearrangement of the bromo-substituted tricyclo[4.2.2.0^{2,5}]deca-3,7-dien-9-one (II, prepared from dibromo COT and chloroacrylonitrile adduct IX as shown in Scheme I) to the bicyclo[3.2.1]octa-2,6-diene derivative VIII, mp 110 °C, under Schmidt reaction conditions. The structure of VIII follows from complementary spectral characteristics (^{1}H and ^{13}C NMR) summarized in the Experimental Section. In particular, the ^{1}H NMR double-resonance experiments gave results analogous to that obtained in the case of IV. A small amount of lactam (X) was also formed in the reaction of II, but it could not be fully characterized due to the paucity of the material.

A plausible mechanism for the formation of IV from I is displayed in Scheme II. It is not quite clear whether IV is formed directly from the Schmidt intermediate XI in a concerted manner (path a) or through the intermediacy of the allylic cation (XII, path b) bearing the bicyclo[4.2.0]octane skeleton. The driving force for the further rearrangement of the bicyclo[4.2.0]octane-based allylic cation XII is obviously the release of strain through the expansion of the cyclobutene ring. It is interesting to note that XII rearranges exclusively to the dihomoaromatic species XIV and rearrangement products derived from the bicyclo[3.3.0]octane-based allylic cation XIII, having comparable stability, are not encountered. This behavior is in harmony with the earlier recorded[9] observations on bicyclo[4.2.0]oct-7-en-2-yl cations.

Experimental Section

All melting points were recorded on a Buchi SMP-20 apparatus and are uncorrected. IR, ^{1}H NMR (60 and 100 MHz), and ^{13}C NMR (25.0 and 22.64 MHz) spectra were recorded on a Perkin-Elmer 297 spectrophotometer, Varian A-60 spectrometer, JEOL MH-100 spectrometer, JEOL FX-100 spectrometer, and Brucker WH-90 spectrometer, respectively. ^{1}H NMR and ^{13}C NMR chemical shifts are reported on the δ scale relative to internal Me_4Si. Off-resonance multiplicities in the ^{13}C NMR spectra, when recorded, are shown in parentheses and abbreviations s, d, t, q, and m correspond to singlet, doublet, triplet, quartet, and multiplet, respectively. All solvent extracts were washed with brine, dried over anhydrous Na_2SO_4, and concentrated on a rotary

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evaporator. All operations that required exclusion of moisture were carried under dry N_2, using standard syringe-septum techniques.

Tricyclo[4.2.2.0^{2,5}]deca-3,7-dien-9-one (I).[4,10] To a solution of lithium diisopropylamide [6 mmol, prepared by adding 5 mL of 1.6 M *n*-butyllithium in hexane to a stirred solution of diisopropylamine (605 mg, 6 mmol) in 5 mL of dry THF at -78 °C] was added 9-cyanotricyclo[4.2.2.0^{2,5}]deca-3,7-diene (630 mg, 4 mmol)[5] in 5 mL of dry THF at -78 °C. Dry oxygen gas was bubbled at a moderate flow rate into the lithionitrile solution for 30 min at the same temperature. The reaction was quenched with 12 mL of 1 M stannous chloride in 2 M HCl and stirred for 1 h at ice temperature. The reaction mixture was diluted with water (50 mL) and extracted with ether (3 × 25 mL). Washing of the ether extract with 1 M NaOH (3 × 20 mL) and concentration yielded (600 mg) of an oily residue. Filtration through a silica gel (20 g) column and direct bulb-to-bulb distillation, bp 90 °C (bath) (8 mm), afforded 450 mg (77%) of I as a low-melting solid: IR (neat) 1720 cm^{-1} (carbonyl); ^{1}H NMR (100 MHz, $CDCl_3$) δ 5.7–6.3 (m, 4 H), 2.6–3.15 (m, 4 H), 2.1 (dd, 1 H, J_1 = 16 Hz, J_2 = 4 Hz), 1.75 (dd, 1 H, J_1 = 16 Hz, J_2 = 2 Hz).

3,4-Dibromo-9-chloro-9-cyanotricyclo[4.2.2.0^{2,5}]dec-7-ene (IX). Dibromocyclooctatetraene (9.25 g, 0.035 mol),[11] α-chloroacrylonitrile (9.25 g, 0.105 mol), and 2,6-di-*tert*-butylphenol (0.1 g) were heated in a sealed glass tube (3.5 × 12 cm, N_2 atmosphere) to 105 °C for 8 h. The sealed tube was cooled and carefully opened and its contents were filtered through a silica gel (100 g) column, using 20% benzene in petroleum ether as eluant. Crystallization of appropriate fractions from carbon tetrachloride furnished 7.5 g (61%) of IX: mp 181–182 °C; IR (KBr) 2250 cm^{-1} (cyano); ^{1}H NMR (60 MHz, $CDCl_3$) δ 6.3–6.9 (m, 2 H), 4.6–5.0 (m, 1 H), 4.1–4.4 (m, 1 H), 2.8–3.9 (m, 4 H), 2.0–2.6 (m, 2 H). Anal. Calcd for $C_{11}H_{10}Br_2ClN$: C, 37.55; H, 2.84; N, 3.98. Found: C, 37.58; H, 3.04; N, 4.04.

3-(or 4-)-Bromotricyclo[4.2.2.0^{2,5}]deca-3,7-dien-9-one (II).[4,5] To a solution of the adduct IX (3.52 g, 10 mmol) in 12 mL of Me_2SO was added a hot solution of 1.5 g of KOH (assay 85%) in 1 mL of water. After being stirred at room temperature for 13 h, the reaction mixture was poured in ice–water (40 mL) and extracted with hexane (3 × 25 mL). The organic extract was washed well with water (3 × 25 mL) and concentrated. Direct sublimation of the residue afforded 1.95 g (86%) of II. The product appeared to be a regioisomeric mixture of two bromides from which the major bromide (II) crystallized from hexane: mp 83–84 °C; IR (KBr) 1730 cm^{-1} (carbonyl); ^{1}H NMR (60 MHz, $CDCl_3$) δ 5.9–6.5 (m, 3 H, olefinic), 2.7–3.3 (m, 4 H, ring CH), 1.5–2.25 (dq, 2 H, $CH_2C{=}O$); ^{13}C NMR (22.64 MHz, $CFCl_3$) δ 139.4, 134.1, 127.0, 119.0 (all olefinic), 53.5, 50.0, 46.7, 37.4, 35.7 (carbonyl carbon not seen). Anal. Calcd for $C_{10}H_9BrO$: C, 53.33; H, 4.0. Found: C, 53.58; H, 3.79.

Schmidt Fragmentation of Tricyclo[4.2.2.0^{2,5}]deca-3,7-dien-9-one (I). To a stirred ice-cold solution of I (90 mg, 0.615 mmol) and methanesulfonic acid (1 mL) in dry dichloromethane (5 mL) was added sodium azide (41 mg, 0.615 mmol) in small portions. After being stirred for 5 min more, the solution was quenched in aqueous $NaHCO_3$ and extracted with CH_2Cl_2 (2 × 15 mL). TLC examination (silica gel plates, solvent system 40% ethyl acetate in benzene) indicated the presence of two components. The reaction mixture was charged on a silica gel (10 g) column and eluted with 20% ethyl acetate in benzene to furnish the cyano mesylate (IV, 80 mg, 55%). Bulb-to-bulb distillation [165 °C (0.6 mm)] and crystallization from carbon tetrachloride yielded the crystalline compound: mp 65–66 °C; IR (KBr) 2250 (cyano), 1360 and 1180 cm^{-1} (mesylate ester); ^{1}H NMR (100 MHz, $CDCl_3$) δ 6.25 (dd, 1 H, J_1 = 6 Hz, J_2 = 3 Hz), 5.75–6.15 (m, 2 H), 5.64 ($^1/_2$AB, 1 H, J = 10 Hz), 5.05 (t, 1 H, J = 5 Hz), 3.02 (s, 3 H, OSO_2CH_3), 2.85–3.05 (m, 1 H), 2.2–2.8 (m, 4 H); ^{13}C NMR (25.0 MHz, $CDCl_3$) δ 139.0 (d), 129.5 (d), 128.1 (d), and 127.5 (d) (olefinic), 118.6 (s, CN), 78.4 (d, COMs), 42.3 (d) and 40.3 (d) (bridge head C's), 38.6 (q, SO_3CH_3), 33.5 (d, allylic), 21.2 (t, CH_2CN). Anal. Calcd for $C_{11}H_{13}O_3SN$: C, 55.23; H, 5.48; N, 5.85. Found: C, 55.25; H, 5.21; N, 5.76.

Further elution of the column with 50% ethyl acetate in benzene yielded 22 mg (22%) of 7-azatricyclo[4.3.2.0^{2,5}]undeca-3,11-dien-8-one (V): mp 178 °C (crystallized from ether); IR (CH_2Cl_2) 3410, 1665 cm^{-1} (lactam); ^{1}H NMR (100 MHz, $CDCl_3$) δ 5.6–6.45 (m, 5 H, olefinic and NH), 3.3–3.6 (m, 3 H), 2.95–3.3 (m, 2 H), 2.55 (m, 1 H). Anal. Calcd for $C_{10}H_{11}NO$: C, 74.51; H, 6.88; N, 8.69. Found: C, 74.42; H, 6.99; N, 8.75.

Schmidt Fragmentation of 3-(or 4-)-Bromotricyclo[4.2.2.0^{2,5}]deca-3,7-dien-9-one (II). Reaction of bromo ketone II (100 mg, 0.44 mmol) with sodium azide (29 mg, 0.45 mmol) in methanesulfonic acid–methylene chloride as described above resulted in 120 mg of crude mixture. Filtration through a silica gel (10 g) column, using 20% ethyl acetate in benzene as eluant, yielded 100 mg of the cyano mesylate VIII (70%): mp 110 °C (white needles from dichloromethane–petroleum ether); IR (KBr) 2255 (cyano), 1595 (olefinic), 1360, 1180 cm^{-1} (mesylate ester); ^{1}H NMR (100 MHz, $CDCl_3$) δ 6.0–6.25 (m, 2 H), 5.8 ($^1/_2$AB, 1 H, J = 8 Hz), 5.18 (t, 1 H, J = 5 Hz), 3.05 (s, 3 H), 3.0–3.2 (m, 1 H), 2.3–2.9 (m, 4 H); ^{13}C NMR (25.0 MHz, $CDCl_3$) δ 129.0, 128.7, 128.0, and 126.3 (olefinic), 118.3 (CN), 76.2 (COMs), 47.1 and 43.8 (bridgehead C's), 38.5 (CH_3SO_3), 32.8 (allylic), 20.2 (CH_2CN). Anal. Calcd for $C_{11}H_{12}O_3NSBr$: C, 41.5; H, 3.71; N, 4.48. Found: C, 41.32; H, 3.71; N, 4.34.

Acknowledgment. We thank SERC, Department of Science and Technology, for partial support of this research.

Registry No. I, 16282-05-6; II, 73382-94-2; III, 73395-57-0; IV, 76446-95-2; V, 76447-09-1; VIII, 76447-08-0; IX, 73374-41-1; α-chloroacrylonitrile, 920-37-6; dibromocyclooctatetraene, 76447-10-4.

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Ethyl *N*-[(Trifluoromethanesulfonyl)oxy]carbamate: A New Reagent for the Synthesis of *N*-(Ethoxycarbonyl)sulfilimines

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Despite current interest in the chemistry of sulfilimines,[1] there are no general methods available for the synthesis of *N*-(alkoxycarbonyl)sulfilimines. The most useful procedures involve direct amination of sulfides of *N*-chlorocarbamates[2] and (alkoxycarbonyl)nitrenes (generated from azidoformates[3,4] or *N*-[(arenesulfonyl)oxy]carbamates),[5] but all of them suffer from limited applications. We now introduce ethyl *N*-[(trifluoromethanesulfonyl)oxy]carbamate (1)[6] as a new reactive aminating reagent of sulfides.

The reagent 1 was prepared in 72% overall yield by the reaction of the thallium salt[7] of ethyl *N*-hydroxycarbamate with trifluoromethanesulfonic anhydride[8] in methylene chloride at -30 to -40 °C (eq 1). The use of the sodium

$$\mathrm{HONHCO_2Et} \xrightarrow[\text{2. Tf}_2\text{O}]{\text{1. TlOEt or NaOEt}} \underset{\mathbf{1}}{\mathrm{TfONHCO_2Et}} \qquad (1)$$

$$\mathrm{Tf} = \mathrm{CF_3SO_2}$$

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