Synthesis of Tetraacetal Pentaoxa-Cages and Convex Oxa-Cages by Ozonolysis of 7-Oxabicyclo[2.2.1]heptenes

Chu-Chung Lin, Hsien-Jen Wu*

Department of Applied Chemistry, National Chiao Tung University, Hsinchu, Taiwan, Republic of China

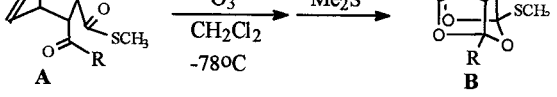
Fax +886(35)723764

Received 11 August 1995; revised 22 November 1995

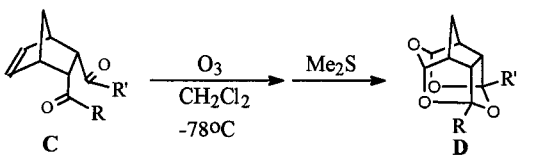
Tetraacetal pentaoxa-cage compounds **4a** and **4b** and convex oxa-cage compounds **6**, **7** and **8** are synthesized from alkylfurans in three steps. Ozonolysis of the *endo* adducts **2a** and **2b** in dichloromethane at −78 °C followed by reduction with dimethyl sulfide gave the tetraacetal pentaoxa-cages **4a** and **4b** in 85–90% yields, respectively. Ozonolysis of **2a** and **2b** in dichloromethane at −78 °C followed by treatment with triethylamine gave the convex oxa-cages **6**, **7** and **8** in 85–90% yields, respectively. The synthesis of the tetraacetal pentaoxa-cage **12**, possessing aromatic substituents directly on the skeleton of the oxa-cages, has also been accomplished.

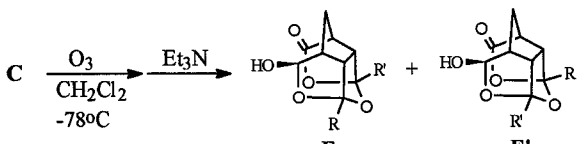
The synthesis and chemistry of polycyclic cage compounds have attracted considerable attention in recent years.[1] The vast majority of the work reported in this area has dealt with polycarbocyclic cage compounds. Although the synthesis and chemistry of heterocyclic cage compounds have received less attention, there are some reports regarding the chemistry[2] and synthesis[3–8] of oxa-cage compounds in the literature. This class of heterocyclic cage compounds is synthesized by intramolecular alkene–oxirane (2σ–2π) photocycloaddition,[3] by transannular cyclization of suitable compounds,[4] by tandem cyclization,[5] by dehydration of diols having the proper stereochemistry,[6] by base-promoted rearrangement,[7] and by intramolecular etherification of an alkene bond with an organoselenium reagent.[8]

Recently, we reported[9] the formation of novel heterocyclic cage compounds **B** by ozonolysis of thioesters **A**, Scheme 1. We also accomplished[10] the synthesis of new tetraoxa-cages **D** and novel convex tetraquinane oxa-cages **E** and **E'** by ozonolysis of the bis-*endo*-1,4-diones **C**, Scheme 2. Both the thioesters **A** and the 1,4-diones **C** are derivatives of norbornene. In this paper, we report the synthesis of the tetraacetal pentaoxa-cages **4a**, **4b** and **12** and the convex oxa-cages **6**, **7** and **8** in a short sequence.



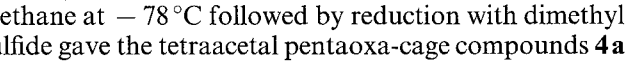
Scheme 1

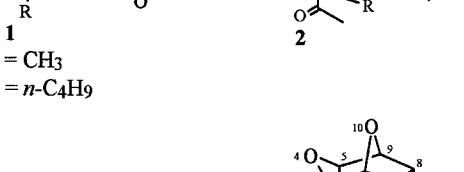




Scheme 2

Diels–Alder reaction of compounds **1a** and **1b**, which were obtained by oxidation of 2,5-dialkylfurans with *m*-chloroperoxybenzoic acid,[10] with furan at 25 °C gave the *endo* adducts **2a** and **2b** as the major product and the *exo* adducts **3a** and **3b** as the minor product in 30–35% yields, respectively. The low yields of the Diels–Alder reaction are attributed to the aromaticity of furan. If the Diels–Alder reaction was conducted in a sealed tube or under pressure, the yield was increased (50–60%). If the Diels–Alder reaction was conducted in the presence of Lewis acids, a Michael addition reaction took place.[11] Ozonolysis of the *endo* adducts **2a** and **2b** in dichloromethane at −78 °C followed by reduction with dimethyl sulfide gave the tetraacetal pentaoxa-cage compounds **4a** and **4b** in 80–85% yields, respectively, Scheme 3.



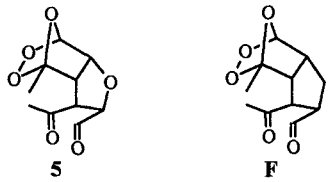


Scheme 3

The IR spectra of **4a** and **4b** lacked carbonyl adsorptions and showed strong absorptions near 1050 cm^{-1} for the ether C–O bonds. The ^1H NMR spectrum of **4a** revealed one doublet at $\delta = 5.48$ for the two acetal protons on C-3 and C-5. The absorption at $\delta = 2.12$ singlet for the methyl ketone protons of **2a** shifted to $\delta = 1.58$ for the angular methyl protons of **4a**. The ^{13}C NMR spectrum of **4a** lacked any carbonyl absorption and displayed one peak at $\delta = 97.4$ for the acetal carbons, one singlet at $\delta = 115.5$ for the quarternary carbons and one peak at $\delta = 25.3$ for the angular methyl carbons. Both the ^1H and ^{13}C NMR spectra of **4a** showed that compound **4a** possesses a symmetry plane. The IR spectrum and ^1H and ^{13}C NMR spectra of **4b** revealed that compound **4b** possesses the same skeleton as **4a**. The conformation of the oxygen atom O-4 with respect to the bridged oxygen atom O-10 was assigned as having a boat conformation based on molecular modeling and the similarity to the previously reported tetraacetal oxa-cage compounds.[9]

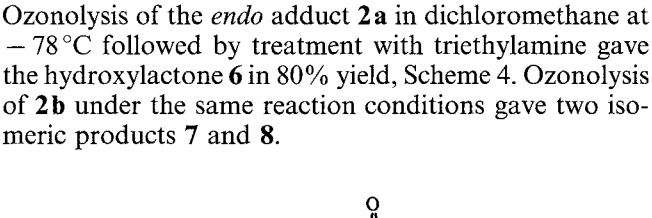
In the ozonolysis of the norbornene derivatives **C** (R = R' = Me) (Scheme 2), we observed by low temperature NMR spectroscopy that the final monomeric ozonide **F** was formed exclusively.[10] Consequently, the

tetraacetal oxa-cages **D** were formed in high yields (85–90%) by reduction of the final ozonide **F** with dimethyl sulfide. In order to understand the effect of an oxygen atom at the apex position of the norbornene derivatives **C** on formation of the final monomeric ozonide, we have performed ozonolysis of a small amount of **2a** in $CDCl_3$ at − 78 °C. We observed by low temperature NMR spectroscopy that the final monomeric ozonide **5** was formed exclusively, Figure 1. Consequently, the oxa-cage **4a** was formed in high yield by reduction of the final ozonide **5** with dimethyl sulfide. The ^{1}H NMR spectrum of **5** was taken at − 30 °C right after the ozonation process without purification.



5 **F**

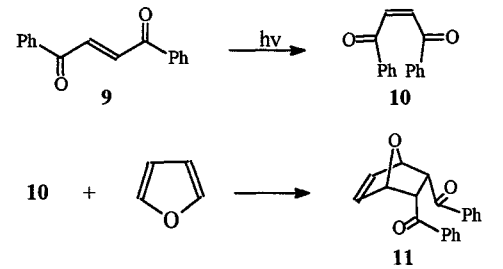
Ozonolysis of the *endo* adduct **2a** in dichloromethane at − 78 °C followed by treatment with triethylamine gave the hydroxylactone **6** in 80% yield, Scheme 4. Ozonolysis of **2b** under the same reaction conditions gave two isomeric products **7** and **8**.



Scheme 4

In the reaction of final ozonides with triethylamine, Razumovskii et al.[12] concluded that an oxidation–reduction electron-transfer process occurred between the amine and the ozonide. In our own results, reaction of the final ozonide **5** with dimethyl sulfide gave the pentaoxa-cages **4**, whereas reduction of the final ozonide **5** with triethylamine gave the convex oxa-cages **6**, **7** and **8**. These results support that an acid–base proton-transfer process occurs between the final ozonides and triethylamine.[13] In other words, triethylamine acts as a base rather than as a reducing agent in the reaction with the final ozonides if there is at least one proton present on the trioxolane ring.

In order to synthesize oxa-cages which possess aromatic substituents directly on the skeleton of the oxa-cages, compound **11** was prepared. Photoisomerization[14] of the commercially available compound **9** with sunlight gave the *cis* isomer **10**. Diels–Alder reaction of **10** with furan at 25 °C gave the *endo* adduct **11** as the major product in 30% yield. Ozonolysis of **11** in dichloromethane at − 78 °C followed by reduction with dimethyl sulfide gave the pentaoxa-cage **12** in 85% yield, Scheme 5.



Scheme 5

Thus, we have accomplished the synthesis of tetraacetal pentaoxa-cage compounds **4a**, **4b** and **12** and convex oxa-cages **6**, **7** and **8** in three steps from alkylfurans.

Melting points were determined in capillary tubes with a Laboratory Devices melting point apparatus and are uncorrected. Infrared spectra were recorded in $CHCl_3$ solutions or on neat thin films

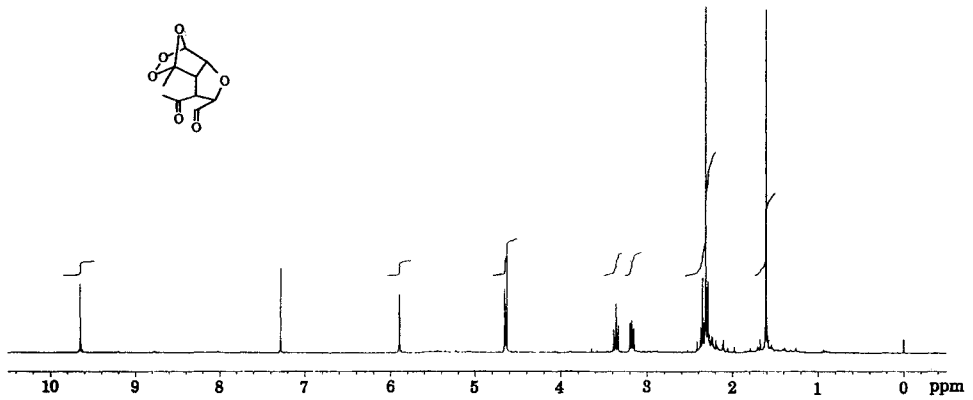


Figure 1. The ^{1}H NMR spectrum of the final ozonide **5**.

between NaCl disks on a Nicolet 520 spectrometer. ^{1}H NMR spectra were taken in $CDCl_3$ solutions on a Varian UNITY-300FT spectrometer and were referenced to chloroform (δ = 7.24), tetramethylsilane (δ = 0.00), acetone (δ = 2.04) or MeOH (δ = 3.30). ^{13}C NMR spectra were recorded in $CDCl_3$ solutions unless otherwise stated on a Varian UNITY-300FT spectrometer with a center line of internal $CDCl_3$ (δ = 77.0), acetone (δ = 29.8) or MeOH (δ = 49.0) as reference. The multiplicities of ^{13}C signals were determined by DEPT techniques. Mass spectra were taken on a JEOL JMS-D100 or TRIO-2000 mass spectrometer. High resolution mass values were obtained with a JEOL JMS-D300 mass spectrometer. Elemental analyses were performed at the microanalysis laboratory of National Taiwan University using a Perkin-Elmer 240C analyzer. For TLC analysis, Merck precoated TLC plates (Kieselgel 60 F_{254}, 0.2 mm) were used, and column chromatography was performed using Merck Kieselgel 60 (70–230 mesh) as the stationary phase.

***endo*-2,3-Diacetyl-7-oxabicyclo[2.2.1]hept-5-ene (2a) and *exo*-2,3-Diacetyl-7-oxabicyclo[2.2.1]hept-5-ene (3a); Typical Procedure:**
To a solution of **1a** (2.0 g, 17.9 mmol) in CH_2Cl_2 (3 mL) was added furan (3.7 g, 53.7 mmol) at 25 °C. The mixture was stirred at 25 °C for 2 days. The solvent was evaporated and the crude product was purified by column chromatography to give the *endo* adduct **2a** as a pale yellow oil; yield: 0.92 g (29%) and the *exo* adduct **3a** as a pale yellow oil; yield: 0.19 g (6.0%).

Spectral data for **2a**:

IR ($CHCl_3$): ν = 2970, 1710, 1595, 1150 cm^{-1}.

^{1}H NMR: δ = 6.50 (br s, 2H), 5.14 (d, J = 1.5 Hz, 2H), 3.56 (d, J = 1.5 Hz, 2H), 2.12 (s, 6H).

^{13}C NMR: δ = 204.74 (2C), 134.37 (2CH), 80.32 (2CH), 56.63 (2CH), 30.32 (2CH_3).

LRMS: m/z (%) = 180 (M^+, 3), 111 (37), 68 (100).

HRMS (EI) calc. for $C_{10}H_{12}O_3$ 180.0786, found 180.0764.

Spectral data for **3a**:

IR ($CHCl_3$): ν = 2970, 1710, 1595, 1150 cm^{-1}.

^{1}H NMR: δ = 6.47 (d, J = 1 Hz, 2H), 5.22 (d, J = 1 Hz, 2H), 2.88 (br s, 2H), 2.21 (s, 6H).

^{13}C NMR: δ = 207.10 (2C), 136.47 (2CH), 79.77 (2CH), 55.06 (2CH), 29.68 (2CH_3).

LRMS: m/z (%) = 180 (M^+, 2), 111 (41), 68 (100).

HRMS (EI): calc. for $C_{10}H_{12}O_3$ 180.0786, found 180.0783.

Spectral data for **2b**:

IR ($CHCl_3$): ν = 2970, 1710, 1595, 1150 cm^{-1}.

^{1}H NMR: δ = 6.54 (dd, J = 5.9 Hz, J = 1.2 Hz, 1H), 6.44 (dd, J = 5.9 Hz, J = 1 Hz, 1H), 5.13 (dd, J = 4.5 Hz, J = 1 Hz, 2H), 3.55 (ABq, J = 9.3 Hz, 1H), 3.54 (ABq, J = 9.3 Hz, 1H), 2.42–2.30 (m, 2H), 2.09 (s, 3H), 1.62–1.51 (m, 2H), 1.39–1.22 (m, 2H), 0.90 (t, J = 7.2 Hz, 3H).

^{13}C NMR: δ = 207.10 (C), 204.85 (C), 134.72 (CH), 134.22 (CH), 80.56 (CH), 80.41 (CH), 56.93 (CH), 55.88 (CH), 43.06 (CH_2), 30.41 (CH_3), 25.69 (CH_2), 22.25 (CH_2), 13.80 (CH_3).

LRMS: m/z (%) = 222 (M^+, 8), 111 (43), 97 (88), 71 (100).

HRMS (EI) calc. for $C_{13}H_{18}O_3$ 222.1256, found 222.1253.

Spectral data for **3b**:

IR ($CHCl_3$: ν = 2970, 1710, 1595, 1150 cm^{-1}.

^{1}H NMR: δ = 6.46 (br s, 2H), 5.22 (br s, 1H), 5.18 (br s, 1H), 2.93 (d, J = 9 Hz, 1H), 2.80 (d, J = 9 Hz, 1H), 2.58–2.43 (m, 2H), 2.195 (s, 3H), 1.63–1.51 (m, 2H), 1.42–1.22 (m, 2H), 0.94–0.88 (m, 3H).

^{13}C NMR: δ = 208.90 (C), 207.45 (C), 136.56 (CH), 136.50 (CH), 79.83 (2CH), 54.74 (CH), 54.45 (CH), 42.36 (CH_2), 29.65 (CH_3), 25.75 (CH_2), 22.25 (CH_2), 13.86 (CH_3).

LRMS: m/z (%) = 222 (M^+, 6), 111 (32), 97 (100).

HRMS (EI) calc. for $C_{13}H_{18}O_3$ 222.1256, found 222.1249.

Diels–Alder Reaction of 1a and 1b with Furan in Sealed Tube.
A solution of **1a** (0.5 g, 4.5 mmol) in furan (3.7 g, 53.7 mmol) was sealed in a Pyrex tube and was heated to 60 °C for 10 h. After cooling, excess furan was evaporated and the crude product was purified by column chromatography to give the *endo* adduct **2a** (0.42 g 50%) and the *exo* adduct **3a** (0.08 g, 12%).

1,7:3,5:4,8-Triepoxy-1,3-dimethyloctahydrofuro[3,4-*d*]oxepin (4a) and 1-Butyl-1,7:3,5:4,8-triepoxymethyloctahydro[3,4-*d*]oxepin (4b):
A solution of **2a** (0.5 g, 2.8 mmol) in CH_2Cl_2 (20 mL) was cooled to − 78 °C and ozone was bubbled through it at − 78 °C until the solution turned light blue. To this solution was added $(CH_3)_2S$ (0.52 g, 8.4 mmol) at − 78 °C. Then, the mixture was stirred at r.t. for 5 h. The solvent was evaporated and the crude product was purified by column chromatography to give **4a** as a white waxy solid; yield: 0.5 g (85%).

Mp 162–163 °C.

IR ($CHCl_3$): ν = 2980, 2880, 1050 cm^{-1}.

^{1}H NMR: δ = 5.48 (d, J = 6.0 Hz, 2H), 4.89–4.84 (m, 2H), 3.39 (dd, J = 4.4 Hz, J = 3.0 Hz, 2H), 1.54 (s, 6H).

^{13}C NMR: δ = 115.46 (2C), 97.40 (2CH), 80.58 (2CH), 57.28 (2CH), 25.25 (CH_3).

LRMS: m/z (%) = 212 (M^+, 7), 127 (51), 99 (83), 81 (100).

HRMS (EI) calc. for $C_{10}H_{12}O_5$ 212.0685, found 212.0675.

4b: white waxy solid; yield: (80%); mp 157–159 °C.

IR ($CHCl_3$): ν = 2980, 2880, 1050 cm^{-1}.

^{1}H NMR: δ = 5.47 (d, J = 6.0 Hz, 2H), 4.92–4.79 (m, 2H), 3.34–3.31 (m, 2H), 1.81–1.73 (m, 2H), 1.53 (s, 3H), 1.40–1.25 (m, 4H), 0.94–0.89 (m, 3H).

^{13}C NMR: δ = 117.68 (C), 115.40 (C), 97.37 (CH), 97.19 (CH), 80.56 (CH), 80.50 (CH), 56.84 (CH), 55.35 (CH), 37.32 (CH_2), 26.22 (CH_2), 25.08 (CH_3), 22.52 (CH_2), 13.89 (CH_3).

LRMS: m/z (%) = 254 (M^+, 6), 127 (25), 85 (100).

HRMS (EI) calc. for $C_{13}H_{18}O_5$ 254.1154, found 254.1170.

7-Acetyl-1,4-epoxy-6-formyl-1-methyltetrahydro-1*H*,4*H*-furo[2,3-*d*][1,2]dioxin (5):
A solution of **2a** (0.05 g, 0.28 mmol) in $CDCl_3$ (1.0 mL) was cooled to − 78 °C, and ozone was bubbled through it at − 78 °C until the solution turned light blue. The solution was then transferred to an NMR tube and the ^{1}H and ^{13}C NMR spectra were taken at − 30 °C.

^{1}H NMR: δ = 9.65 (s, 1H), 5.89 (s, 1H), 4.65 (d, J = 7.2 Hz, 1H), 4.64 (d, J = 6 Hz, 1H), 3.36 (dd, J = 8.1 Hz, J = 7.2 Hz, 1H), 3.17 (dd, J = 8.1 Hz, J = 6 Hz, 1H), 2.31 (s, 3H), 1.61 (s, 3H).

^{13}C NMR: δ = 201.33 (C=O), 199.78 (CH), 119.63 (C), 101.59 (CH), 83.32 (CH), 82.42 (CH), 57.60 (CH), 49.93 (CH), 30.64 (CH_3), 13.98 (CH_3).

3,4-Epoxy-6a,7a-dimethyl-2-oxooctahydrodifuro[2,3-*b*,3′,2′-*d*]furan (6); Typical Procedure:
A solution of **2a** (0.50 g, 2.8 mmol) in CH_2Cl_2 (20 mL) was cooled to − 78 °C, and ozone was bubbled through it at − 78 °C until the solution turned light blue. To this solution was added Et_3N (0.28 g, 2.8 mmol) at − 78 °C. Then, the mixture was stirred at r.t. for 3 h. The solvent was evaporated and the crude product was purified by column chromatography to give **6** as a white waxy solid; yield: 0.57 g (90%).

Spectral data for **6**:

Mp 159–160 °C.

IR ($CHCl_3$): ν = 3450, 2960, 1767, 1110 cm^{-1}.

^{1}H NMR (CD_3OD): δ = 5.31 (s, 1H), 4.81 (d, J = 8.7 Hz, 1H), 4.52 (d, J = 4.8 Hz, 1H), 3.76–3.59 (m, 2H), 3.20 (br s, 1H), 1.54 (s, 3H), 1.48 (s, 3H).

^{13}C NMR (CD_3COCD_3): δ = 175.51 (C=O), 121.70 (C), 117.27 (C), 104.13 (CH), 92.73 (CH), 83.32 (CH), 60.68 (CH), 57.80 (CH), 27.06 (CH_3), 25.02 (CH_3).

LRMS: m/z (%) = 228 (M^+, 14), 210 (100).

HRMS (EI) calc. for $C_{10}H_{12}O_6$ 228.0634, found 228.0629.

6a-Butyl-3,4-epoxy-7a-methyl-2-oxooctahydrodifuro[2,3-*b*,3′,2′-*d*]furan (7) and 7a-Butyl-3,4-epoxy-6a-methyl-2-oxooctahydrodifuro[2,3-*b*,3′,2′-*d*]furan (8):

White waxy solid; combined yield (85%); mp 148–150°C.

IR ($CHCl_3$): ν = 3450, 2960, 1767, 1110 cm^{-1}.

^1H NMR (CD_3COCD_3): δ = 5.55 (s, 2H), 4.91–4.73 (m, 4H), 3.69–3.63 (m, 4H), 3.07 (br s, 2H), 2.05–1.72 (m, 4H), 1.70 (s, 3H), 1.69 (s, 3H), 1.50–1.24 (m, 8H), 0.94–0.88 (m, 6H).

^{13}C NMR (CD_3COCD_3): δ = 177.73 (C=O), 177.64 (C=O), 127.23 (C), 125.25 (C), 122.25 (C), 120.36 (C), 107.91 (CH), 107.83 (CH), 96.46 (CH), 87.17 (CH), 86.76 (CH), 83.41 (CH), 64.33 (CH), 63.04 (CH), 61.30 (CH), 59.96 (CH), 44.40 (CH_2), 42.15 (CH_2), 31.34 (CH_3), 30.88 (CH_2), 30.32 (CH_2), 29.39 (CH_3), 27.64 (CH_2), 27.41 (CH_2), 18.52 (CH_3), 18.41 (CH_3).

LRMS: *m/z* (%) = 270 (M^+, 23), 253 (100).

HRMS (EI) calc. for $C_{13}H_{18}O_6$ 270.1103, found 270.1096.

***endo*-2,3-Dibenzoyl-7-oxabicyclo[2.2.1]hept-5-ene (11):**

The same reaction conditions and procedure for the synthesis of **2a** and **2b** from Diels–Alder reaction of **10** with furan were applied to the synthesis of **11**; yield: (28%).

IR ($CHCl_3$): ν = 2970, 1680, 1595, 1150 cm^{-1}.

^1H NMR: δ = 7.95–7.82 (m, 4H), 7.60–7.37 (m, 6H), 6.53 (br s, 2H), 5.34 (br s, 2H), 4.48 (br s, 2H).

^{13}C NMR: δ = 196.52 (2C), 137.28 (2C), 137.14 (2CH), 132.71 (2CH), 128.54 (4CH), 127.76 (4CH), 80.82 (2CH), 50.49 (2CH).

LRMS: *m/z* (%) = 304 (M^+, 4), 275 (63), 123 (100).

HRMS (EI) calc. for $C_{20}H_{16}O_3$ 304.1099, found 304.1095.

1,7:5,3:4,8-Triepoxy-1,3-diphenyloctahydrofuro[3,4-*d*]oxepin (12):

The same reaction conditions and procedure for the synthesis of **4a** and **4b** from ozonolysis of **2a** and **2b** were applied to the ozonolysis of **11**; yield: (85%).

Mp 202–203°C.

IR ($CHCl_3$): ν = 3070, 2980, 1580, 1060 cm^{-1}.

^1H NMR: δ = 7.60–7.29 (m, 10H), 5.82 (d, J = 6 Hz, 2H), 5.08–5.04 (m, 2H), 3.79 (dd, J = 5.1 Hz, J = 3 Hz, 2H).

^{13}C NMR: δ = 139.88 (2C), 128.81 (2CH), 128.43 (4CH), 125.57 (4CH), 116.51 (2C), 98.04 (2CH), 80.73 (2CH), 60.01 (2CH).

LRMS: *m/z* (%) = 336 (M^+, 100), 307 (67), 231 (57).

HRMS (EI) calc. for $C_{20}H_{16}O_5$ 336.0998, found 336.0989.

(1) For reviews, see:
Eaton, P.E. *Angew. Chem. Int. Ed. Engl.* **1992**, *31*, 1421.
Griffin, G.W.; Marchand, A.P. *Chem. Rev.* **1989**, *89*, 997.
Marchand, A.P. *Chem. Rev.* **1989**, *89*, 1011.
Paquette, L.A. *Chem. Rev.* **1989**, *89*, 1051.
Klunder, A.J.H.; Zwanenburg, B. *Chem. Rev.* **1989**, *89*, 1035.
Mehta, G.; Marchand, A.P.; Dilling, W.L. In *Carbocyclic Cage Compounds*; Osawa, E.; Yonemitsu, O. Ed.; VCH: New York, 1992.

(2) Mehta, G.; Nair, M.S. *J. Chem. Soc., Chem. Commun.* **1983**, 439.
Shen, K.W. *J. Am. Chem. Soc.* **1971**, *93*, 3064.
Allred, E.L.; Beck, B.R. *Tetrahedron Lett.* **1974**, 437.
Barborak, J.C.; Khoury, D.; Maier, W.F.; Schleyer, P.V.R.; Smith, E.C.; Smith, Jr., W.F.; Wyrick, C. *J. Org. Chem.* **1979**, *44*, 4761.

(3) Prinzbach, H.; Klaus, M. *Angew. Chem., Int. Ed. Engl.* **1969**, **8**, 276.
Marchand, A.P.; Reddy, G.M.; Watson, W.H.; Kashyap, R. *Tetrahedron* **1990**, *46*, 3409.

(4) Sasaki, T.; Eguchi, S.; Kiriyama, T.; Hiroaki, O. *Tetrahedron* **1974**, *30*, 2707.
Singh, P. *J. Org. Chem.* **1979**, *44*, 843.
Coxon, J.M.; Fong, S.T.; McDonald, D.Q.; O'Connell, M.J.; Steel, P.J. *Tetrahedron Lett.* **1991**, *32*, 7115.

(5) Suri, S.C. *J. Org. Chem.* **1993**, *58*, 4153.

(6) Mehta, G.; Srikrishna, A.; Reddy, A.V.; Nair, M.S. *Tetrahedron* **1981**, *37*, 4543.
Mehta, G.; Nair, M.S. *J. Am. Chem. Soc.* **1985**, *107*, 7519.
Marchand, A.P.; LaRoe, W.D.; Sharma, G.V.M.; Suri, S.C.; Reddy, D.S. *J. Org. Chem.* **1986**, *51*, 1622.
Fessner, W.D.; Prinzbach, H. *Tetrahedron* **1986**, *42*, 1797.
Smith, E.C.; Barborak, J.C. *J. Org. Chem.* **1976**, *41*, 1433.

(7) Marchand, A.P.; Chou, T.C. *Tetrahedron* **1975**, *31*, 2655.
Mehta, G.; Reddy, K.R. *J. Org. Chem.* **1987**, *52*, 460.

(8) Mehta, G.; Rao, H.S.P. *J. Chem. Soc., Chem. Commun.* **1986**, 472.
Mehta, G.; Rao, H.S.P.; Reddy, K.R. *J. Chem. Soc., Chem. Commun.* **1987**, 78.

(9) Wu, H.J.; Huang, F.J.; Lin, C.C. *J. Chem. Soc., Chem. Commun.* **1991**, 770.

(10) Wu, H.J.; Lin, C.C. submitted for publication (1995).

(11) Chen, K.L. Masters Thesis, National Chiao Tung University (1990).

(12) Razumovskii, S.D.; Zaikov, G.E. *Ozone and its Reactions with Organic Compounds*, Chapter 9, p 370, Elsevier: Netherlands, 1984.
Pobedimskii, D.G.; Razumovskii, S.D. *Izv. Akad. Nauk SSSR, Ser. Khim.* **1970**, *3*, 602; *Chem. Abstr.* **1970**, *73*, 13853n.

(13) Hon, Y.S.; Yan, J.L. *Tetrahedron Lett.* **1993**, *34*, 6591.

(14) Pasto, D.J.; Duncan, J.A.; Silversmith, E.F. J. Chem. Educ. **1974**, *51*, 277.
Silversmith, E.F.; Dunson, F.C. J. Chem. Educ. **1973**, *50*, 568.