Ozonides from the Ozonolyses of Indene

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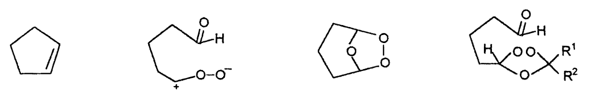
Ozonolysis reactions of indene **5** in the presence of carbonyl compounds **6** provided the corresponding indenemonoozonide **9** and cross-ozonides **10a-c** and **11a-c**. Further reactions of ozonides **10** and **11** with the independently prepared carbonyl oxide **13** gave diozonides of structure **14a-c** and **15a-c**.

Introduction

Reactions of substituted indenes with ozone in non-participating solvents generally gave rise to the corresponding bicyclic ozonides in high yield as a consequence of intramolecular recombination of either or both carbonyl oxide-carbonyl pairs.[1-5] However, studies by Warnel and Shriner[6] on the ozonolysis of unsubstituted indene in ethanol have demonstrated the presence of the cyclic peroxide as an anomalous product.

Ozonolysis of cyclopentene **1** in non-participating solvents has been reported to the corresponding intermediate of type **2**.[7] Recently, we have been able to make use of this mode of generation of intermediate for the preparation of monoozonides type **3** and cross ozonides type **4** by ozonolysis of cyclopetene in the presence of carbonyl compounds.[8] We have now tried whether this mode of reaction can be extended to the ozonolysis of indene **5**. Ozonides of type **4** would represent functionalized ozonides which could undergo subsequent reactions at the aldehyde groups to give diozonides.

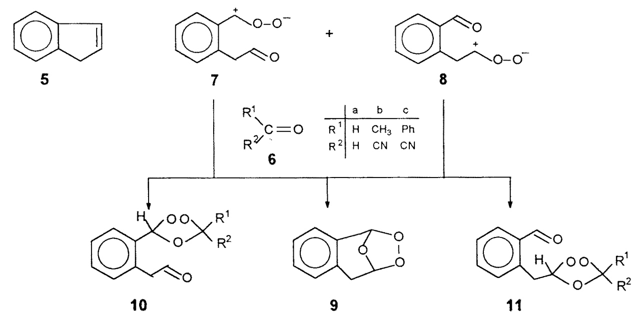


1 **2** **3** **4**

Results and Discussion

In pursuit of our goals, we have ozonized the indene **5** in inert solvents in the presence of two molar equivalents of a carbonyl compounds **6a**, **6b** and **6c**. Ozonolyses of indene **5** in the presence of carbonyl compounds **6** as a good dipolarophile[9,10] afforded in each case the corresponding normal ozonide **9** and cross ozonides **10** and **11**, whereas in this reactions no evidence was found for the formation of other peroxidic products as outlined in Scheme 1.

Cycloreversion process of primary ozonides can provide two possible different intermediates **7** and **8** in the case of indene. Ozonolysis of indene **5** in the presence of carbonyl compounds **6a-c** did provide the corresponding monoozonide **9**, **10a-c** and **11a-c**, which were obtained in yields of 35%, 27% and 15%, respectively. Ozonolyses of **5** in the presence of **6** gave higher yields of the cross-ozonide **10a-c**, suggesting the formation of carbonyl oxide moiety **7** rather



Scheme 1

than **8** is more favored.[5] The formation of ozonide **9** is in line with the known fact that 5-membered cycloolefins[11,12] give high yields of ozonides, *i.e.* intramolecular reaction of the carbonyl oxide in **7** and **8** can compete with intermolecular reaction with carbonyl compounds **6**.

All of the peroxidic products have been isolated by column chromatography on silica gel. The unsymmetrically substituted ozonides **10b** and **10c** were 1 : 2-mixtures of the cis-and trans-isomers, from which only trans-isomers could be isolated. The stereoisomers have been tentatively assigned based on the assumption that, as in other reported cases, the ^1H NMR signal of the hydrogen attached to the ozonide ring appears in a higher position for the trans-isomer than for the cis-isomer.[13,14] The structures of all isolated ozonides were established by ^1H and ^{13}C NMR spectroscopy, and their reduction with triphenylphosphine to give the expected fragments, viz. dialdehydes of structure O–CH-Ar-CH_2-CH–O **12** and the corresponding carbonyl compounds **6** in a ratio of 1 : 1. Characteristic signals in the ^1H NMR spectra of all ozonides of type **10a-c** and **11a-c** were those for the R-C-H groups in the ozonide rings and the CH–O groups in the side chains. The R-C-H signals and CH–O signals for ozonides **10a-c** and **11a-c** appeared in the range of δ – 5.04-6.51 and 9.64-10.18, respectively.

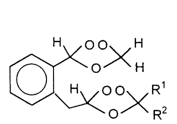
12 **6**

Characteristic signals in the ^{13}C NMR spectra of all ozonides of structure **10a-c** and **11a-c** were those of the magnetically nonequivalent (R,H)C-atoms and (H,H)C- or (R^1,R^2) C-atoms in the ozonide rings and of the C–O atoms in the side chains. The signals for the C-atoms in the ozonide rings appeared in the range of δ = 94.45-101.14 and δ = 102.23-107.11, respectively. The C=O signals of ozonides appeared in the range of δ = 193.16-199.42.

In an attempt to make use of the functionalized ozonides of type **10a-c** and **11a-c**, we have generated formaldehyde-O-oxide **13** in the presence of ozonides **10a-c** and **11a-c** on purpose to induce a cycloaddition reaction between **13** and the aldehyde group in **10** and **11**. To this end, isopropenyl acetate[15] has been ozonized in the presence of one half molar equivalent of one of the ozonides in dichloromethane at -78 °C. Ozonolyses of **10a-c** and **11a-c** in the presence of formaldehyde-O-oxide **13** afforded corresponding diozonides **14a-c** and **15a-c**, respectively. All of the diozonides were isolated by column chromatographic methods.

$\overset{+}{C}H_2-O-O^-$

13



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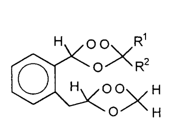

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The structrural assignments of diozonides are based on their reduction with TPP to give the corresponding dialdehyde O=CH-R-CH=O **12** and carbonyl compounds **6**. Characteristic signals in the ^{1}H NMR spectra were those of the CH_2 and CH groups in the ozonide ring appearing in the range of δ = 4.89-6.71 for diozonides **14a-c** and **15a-c**. Characteristic signals in the ^{13}C NMR spectra were those in the range of δ = 94.53-107.56 for all diozonides.

The results in this study provide ample evidences that carbonyl oxides which are formed in the ozonolysis of indene in aprotic solvent can be readily trapped by "foreign" carbonyl compounds to give cross-ozonides. As one of several conceiveable aldehyde reactions, the cycloaddition with formaldehyde-O-oxide was realized to give a variety of diozonides. This represents another new short-path synthesis for ozonides which were not known previously.

Experimental Section

All NMR spectra were recorded with Brucker FT-NMR (300 MHz), using TMS as an internal reference. The ozonides were isolated by flash chromatography on 80 g silica gel using diethyl ether/*n*-pentane in a ratio of 1 : 4. HPLC separation was carried out on a Shimadzu chromatograph SPD-6AV.

Ozonolyses and Reductions of Ozonides. Unless otherwise mentioned, the following procedure was used: The ozonolysis reaction was carried out in dichloromethane at -78 °C until the solution turned blue. Residual ozone was flushed out with nitrogen, the solvent was distilled off at room temperature under reduced pressure, and the residue was separated by flash chromatography. Reductions of isolated ozonides were carried out on *ca.* 20-40 mg samples in *ca.* 0.6 mL of $CDCl_3$ with an excess of triphenylphosphine and the products were analyzed by ^{1}H NMR spectroscopy.

Caution: All ozonolysis reactions, chromatographic separations, and reductions of ozonides were carried out behind protective safety glass shields in hood. Safety glasses and gloves must be worn.

Ozonolysis of 5 and 6a: Ozonolysis of 0.35 g (3 mmol) of **5** and 1 mL of **6a** (freshly prepared by pyrolysis of paraformaldehyde) in 50 mL of dichloromethane followed by distillation of the solvent under reduced pressure gave a liquid residue. From which 0.16 g (0.84 mmol, 28%) of **10a**, 0.10 g (0.51 mmol, 17%) of **11a** and 0.18 g (1.1 mmol, 36%) of **9** were isolated [solvent: diethyl ether/*n*-pentane, 1 : 4].

Indenemonoozonide (9): Colorless solid. mp 62-63 °C (Lit.,[16] 62-63 °C); ^{1}H NMR δ 3.03 (d, J = 18.3 Hz, 1H), 3.33 (d, J = 18.3 Hz, 1H), 6.13 (s, 1H), 6.34 (s, 1H), 6.94-7.37 (m, 4H); ^{13}C NMR δ 35.34, 100.17 100.99, 125.70, 126.95, 129.25, 130.33, 130.41, 133.83. Anal. calcd for $C_9H_8O_3$: C, 65.83; H, 4.91. found: C, 65.77; H, 4.86.

Reduction of 9 with TPP gave 2-(*o*-formylphenyl)ethanal **12** [^{1}H NMR δ 4.14 (s, 2H), 7.24-8.41 (m, 4H), 9.82 (s, 1H), 10.03 (s, 1H); ^{13}C NMR δ 48.74, 128.63-137.75 (m), 193.79, 198.80].

***o*-[(1,2,4-Trioxolan-3-yl)phenyl]acetaldehyde (10a)**: Colorless liquid; ^{1}H NMR δ 3.78 (s, 2H), 5.22 (s, 1H), 5.34 (s, 1H), 6.08 (s, 1H), 7.17-7.60 (m, 4H), 9.64 (s, 1H); ^{13}C NMR δ 47.84, 95.32, 102.23, 128.27, 128.38, 131.09, 131.68, 132.20, 132.47, 199.42. Anal. calcd for $C_{10}H_{10}O_4$: C, 61.85; H, 5.19. found: C, 61.07; H, 5.23.

Reduction of 10a with TPP gave **12**.

***o*-[(1,2,4-Trioxolan-3-yl)methyl]benzaldehyde (11a)**: Colorless liquid; ^{1}H NMR δ 3.50 (m, 2H), 5.04 (s, 1H), 5.07 (s, 1H), 5.44 (t, J = 6.3 Hz, 1H), 7.34-7.84 (m, 4H), 10.18 (s, 1H); ^{13}C NMR δ 35.72, 94.45, 103.10, 128.19, 133.20, 133.57, 134.05, 134.98, 136.78, 193.16. Anal. calcd for $C_{10}H_{10}O_4$: C, 61.85; H, 5.19. found: C, 60.73: H, 5.04.

Reduction of 11a with TPP gave **12.**

Ozonolysis of 5 and 6b: Ozonolysis of 0.35 g (3 mmol) of indene **5** and 0.63 g (9 mmol) of **6b** in 50 mL of dichloromethane, followed by distillation of the solvent under reduced pressure gave a liquid residue. From which 0.20 g (0.87 mmol, 29%) of **10b**, 0.10 g (0.45 mmol, 15%) of **11b** and 0.17 g (1.0 mmol, 34%) of **9** were isolated [solvent: diethyl ether/*n*-pentane, 1 : 1]. By HPLC (3.2 × 25 cm LiChrosorb Si 60, solvent: dichloromethane/*n*-pentane 15 : 1) separation of 0.69 g (3 mmol) of *cis*-and *trans* of **11b**, one obtained 0.45 g (1.95 mmol, 65%) of *trans*-**11b**.

***o*-[5-Cyano-5-methyl-(1,2,4-trioxolan-3-yl)-phenyl]acetaldehyde (10b)**: Colorless liquid (only one isomer of unknown stereochemistry could be isolated.); ^{1}H NMR δ 1.89 (s, 3H), 3.81 (s, 2H), 6.21 (s, 1H), 7.17-7.80 (m, 4H), 9.65 (s, 1H); ^{13}C NMR δ 21.47, 47.67, 99.87, 103.94, 117.09, 128.06, 128.33, 128.63, 128.83, 132.08, 132.75, 198.84. Anal. calcd for $C_{12}H_{11}O_4N$: C, 61.81; H, 4.75. found: C, 62.03; H, 4.63.

Reduction of 10b with TPP gave **12** and **6b** in a ratio of *ca.* 1 : 1.

***cis*-and *trans*-*o*-[5-Cyano-5-methyl-(1,2,4-trioxolan-3-yl)methyl]benzaldehyde (11b)**: Colorless liquid; ^{1}H NMR δ [1.80 (s), 1.85 (s)] (3H), [3.30 (m), 3.66 (m)] (2H), [5.48 (t, J = 3.4 Hz), 6.12 (t, J = 3.4 Hz)] (1H), 7.18-8.30 (m, 4H), 10.11 (s, 1H); ^{13}C NMR δ 20.96, 21.21, 34.10, 34.20, 98.46, 98.79, 105.75, 116.40, 116.78, 128.82, 133.65, 134.31, 134.75, 135.29, 135.70, 193.87. Anal. calcd for $C_{12}H_{11}O_4N$: C, 61.81; H, 4.75. found: C, 61.74; H, 4.82.

Reduction of 11b with TPP gave **12** and **6b** in a ratio of *ca.* 1 : 1.

***trans*-11b**: ^{1}H NMR δ 1.80 (s, 3H), 3.66 (d, J = 3.4 Hz, 2H), 5.48 (t, J = 3.4 Hz, 1H), 7.18-8.30 (m, 4H), 10.11 (s, 1H); ^{13}C NMR δ 21.21, 34.20, 98.79, 105.75, 116.78, 128.82, 133.65, 134.31, 134.75, 135.29, 135.70, 193.87.

Ozonolysis of 5 and 6c: Ozonolysis of 0.35 g (3 mmol) of **5** and 1.18 g (9 mmol) of **6c** in 50 mL of dichloromethane, followed by distillation of the solvent under reduced pressure gave a liquid residue. From which 0.23 g (0.78 mmol, 26%) of **10c**, 0.12 g (0.39 mmol, 13%) of **11c** and 0.16 g (0.99 mmol, 33%) of **9** were isolated [solvent: diethyl ether/*n*-pentane, 1 : 1]. By HPLC (3.2 × 25 cm LiChrosorb Si 60, solvent: dichloromethane/*n*-pentane 15 : 1) separation of 0.89 g (3 mmol) of *cis*-and *trans*-**11c**, one obtained 0.62 g (2.1 mmol, 69%) of *trans*-**11c**.

***o*-[5-Cyano-5-phenyl-(1,2,4-trioxolan-3-yl)-phenyl]acetaldehyde (10c)**: Colorless liquid (only one isomer of unknown stereochemistry could be isolated.); ^{1}H NMR δ 3.92 (s, 2H), 6.52 (s, 1H), 7.29-7.80 (m, 9H), 9.77 (s, 1H); ^{13}C NMR δ 47.98, 102.32, 105.29, 116.34, 127.54, 128.42, 128.96, 129.06, 129.64, 129.65, 132.04, 132.11, 132.21, 132.45, 132.66, 132.74, 198.4. Anal. calcd for $C_{17}H_{13}O_4N$: C, 69.51; H, 4.38. found: C, 69.32; H, 4.41.

Reduction of 10c with TPP gave **12** and **6c** in a ratio of *ca.* 1 : 1.

***cis*-and *trans*-*o*-[5-Cyano-5-phenyl-(1,2,4-trioxolan-3-yl)methyl]benzaldehyde (11c)**: Colorless liquid; ^{1}H NMR δ 3.82 (m, 2H), [5.76 (t, J = 3.2 Hz), 6.09 (t, J = 3.2 Hz)] (1H), 7.41-8.09 (m, 9H), [10.09 (s), 10.13 (s)] (1H); ^{13}C NMR δ 34.37, 101.14, 101.50, 106.76, 107.00, 115.92, 116.12, 127.56, 128.67, 128.85, 129.04, 129.46, 132.55, 133.72, 134.32, 134.36, 134.66, 134.86, 135.29, 193.77, 193.88. Anal. calcd for $C_{17}H_{13}O_4N$: C, 69.51; H, 4.38. found: C, 69.24; H, 4.33.

Reduction of 11c with TPP gave **12** and **6c** in a ratio of *ca.* 1 : 1.

trans-**11c**: ^{1}H NMR δ 3.82 (m, 2H), 5.76 (t, J = 3.2 Hz, 1H), 7.41-8.09 (m, 9H), 10.13 (s, 1H); ^{13}C NMR δ 34.37, 101.50, 107.11, 116.12, 127.56, 128.67, 128.85, 129.04, 129.46, 132.55, 133.72, 134.32, 134.36, 134.66, 134.86, 135.29, 193.88.

Ozonolysis of 10a and isopropenyl acetate: Ozonolysis of 0.58 g (3.0 mmol) of **10a** and 0.60 g (6 mmol) of isopropenyl acetate in 40 mL of dichloromethane gave a solid residue, from which 0.27 g (1.1 mmol, 37%) of **14a** was isolated [solvent: diethyl ether/*n*-pentane, 1 : 1].

3-[*o*-(1,2,4-Trioxolan-3-yl)-benzyl]-1,2,4-trioxolane (14a): Colorless liquid; ^{1}H NMR δ 3.16 (d, J = 3.4 Hz, 2H), 5.05 (s, 1H), 5.07 (s, 1H), 5.22 (s, 1H), 5.34 (s, 1H), 5.42 (t, J = 6.3 Hz, 1H), 6.28 (s, 1H), 7.27-7.63 (m, 4H); ^{13}C NMR δ 35.32, 94.53, 95.45, 101.66, 103.52, 127.92, 130.63, 131.81, 132.09, 132.12, 134.63. Anal. calcd for $C_{11}H_{12}O_6$: C, 55.00; H, 5.04. found: C, 55.13; H, 5.13.

Reduction of 14a with TPP gave **12**.

Ozonolysis of 11a and isopropenyl acetate: Ozonolysis of 0.58 g (3.0 mmol) of **11a** and 0.60 g (6 mmol) of isopropenyl acetate in 40 mL of dichloromethane gave a solid residue, from which 0.25 g (1.0 mmol, 35%) of **14a** was isolated [solvent: diethyl ether/*n*-pentane, 1 : 1].

Ozonolysis of 10b and isopropenyl acetate: Ozonolysis of 0.70 g (3.0 mmol) of **10b** and 0.60 g (6 mmol) of isopropenyl acetate in 40 mL of dichloromethane gave a solid residue, from which 0.29 g (1.05 mmol, 35%) of **14b** was isolated [solvent: diethyl ether/*n*-pentane, 1 : 1].

3-[*o*-(5-Cyano-5-methyl-1,2,4-trioxolane-3-yl)-benzyl]-1,2,4-trioxolane (14b): Colorless liquid (only one isomer of unknown stereochemistry could be isolated.); ^{1}H NMR δ 1.87 (s, 3H), 3.00-3.07 (m, 2H), 4.93 (d, J = 9.2 Hz, 1H), 4.96 (d, J = 9.2 Hz, 1H), 5.27 (t, J = 3.4 Hz, 1H), 6.34 (s, 1H), 7.19-7.74 (m, 4H); ^{13}C NMR δ 21.59, 35.12, 35.41, 94.58, 99.80, 103.03, 103.19, 103.78, 103.84, 117.13, 128.25, 128.54, 131.59, 132.09, 132.21, 134.99. Anal. calcd for $C_{13}H_{13}O_6N$: C, 55.91; H, 4.69. found: C, 56.14; H, 4.63.

Reduction of 14b with TPP gave **12** and **6b** in a ratio of *ca.* 1 : 1.

Ozonolysis of 11b and isopropenyl acetate: Ozonolysis of 0.70 g (3.0 mmol) of *cis*- and *trans*-**11b** and 0.60 g (6 mmol) of isopropenyl acetate in 40 mL of dichloromethane gave a solid residue, from which 0.28 g (1.0 mmol, 34%) of **15b** was isolated [solvent: diethyl ether/*n*-pentane, 1 : 1].

3-[*o*-(1,2,4-Trioxolan-3-yl)-benzyl]-5-cyano-5-methyl-1,2,4-trioxolane (15b): Colorless liquid (a mixture of two stereoisomers); ^{1}H NMR δ 1.80 (s, 3H), [3.09 (m), 3.34 (m)] (2H), 5.29 (s, 1H), 5.43 (s, 1H), [5.44 (t, J = 3.4 Hz, 5.85 (t, J = 3.4 Hz)] (1H), 6.21 (s, 1H), 7.317.90 (m, 4H); ^{13}C NMR δ 20.96, 21.20, 33.56, 33.68, 95.49, 98.53, 98.88, 101.80, 106.24, 106.31, 116.37, 116.81, 128.36, 130.74, 131.57, 132.36, 132.96, 133.76. Anal. calcd for $C_{13}H_{13}O_6N$: C, 55.91; H, 4.69. found: C, 55.74; H, 4.58.

Reduction of 15b with TPP gave **12** and **6b** in a ratio of *ca.* 1 : 1.

Ozonolysis of 10c and isopropenyl acetate: Ozonolysis of 0.89 g (3.0 mmol) of **10c** and 0.60 g (6 mmol) of isopropenyl acetate in 40 mL of dichloromethane gave a solid residue, from which 0.32 g (0.93 mmol, 31%) of **14c** was isolated [solvent: diethyl ether/*n*-pentane, 1 : 1].

3-[*o*-(5-Cyano-5-phenyl-1,2,4-trioxolan-3-yl)-benzyl]-1,2,4-trioxolane (14c): Colorless liquid (only one isomer of unknown stereochemistry could be isolated.); ^{1}H NMR δ 3.08-3.28 (m, 2H), 4.99-5.10 (m, 2H), 5.39 (t, J = 3.4 Hz, 1H), 6.69 (s, 1H), 7.32-8.15 (m, 9H); ^{13}C NMR δ 35.23, 35.66, 94.63, 97.01, 103.04, 103.25, 105.14, 105.22, 116.48, 127.53, 128.52, 128.67, 129.36, 129.6, 130.90, 131.69, 132.11, 132.26, 132.64, 134.97, 137.29. Anal. calcd for $C_{18}H_{15}O_6N$: C, 63.35; H, 4.43. found: C, 63.13; H, 4.56.

Reduction of 14c with TPP gave **12** and **6c** in a ratio of *ca.* 1 : 1.

Ozonolysis of 11c and isopropenyl acetate: Ozonolysis of 0.89 g (3.0 mmol) of *cis*- and *trans*-**11c** and 0.60 g (6 mmol) of isopropenyl acetate in 40 mL of dichloromethane gave a solid residue, from which 0.33 g (0.96 mmol, 32%) of

15c was isolated [solvent: diethyl ether/*n*-pentane, 1 : 1].

3-[*o*-(1,2,4-Trioxolan-3-yl)-benzyl]-5-cyano-5-phenyl-1,2,4-trioxolane (15c): Colorless liquid (a mixture of two stereoisomers); ^{1}H NMR δ 3.41-3.55 (m, 2H), 5.34 (s, 1H), 5.47 (s, 1H), 5.74 (t, J = 3.4 Hz, 1H), 6.27 (s, 1H), 7.38-7.71 (m, 9H); ^{13}C NMR: δ 33.98, 95.50, 100.50, 102.00, 107.56, 116.08, 127.60, 128.38, 128.90, 129.48, 130.58, 130.99, 131.58, 132.38, 132.47, 132.59, 132.81, 132.89. Anal. calcd for $C_{18}H_{15}O_6N$: C, 63.35; H, 4.43. found: C, 63.52; H, 4.37.

Reduction of 15c with TPP gave **12** and **6c** in a ratio of *ca.* 1 : 1.

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