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# Oxidation of Alkenes with Hydrogen Peroxide, Catalyzed by Boron Trifluoride. Synthesis of Vicinal Methoxyalkanols

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**Abstract**—In oxidation of alkenes with the $BF_3$–$H_2O_2$ system, boron trifluoride induces transfer of available oxygen from hydrogen peroxide, accompanied by the formation of epoxides. The oxidation in methanol occurs as a one-pot two-step process involving epoxidation of the C=C bond followed by opening of the oxirane ring, with the formation of methoxyalkanols.

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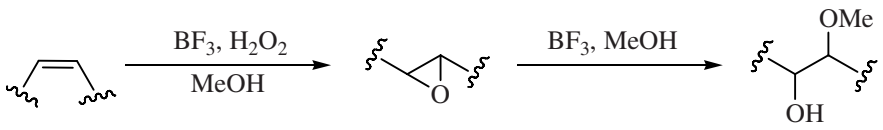
The development of new methods for oxidation of unsaturated componds attracts enduring interest of organic chemists. An appreciable share of studies in this field concern the search for catalysts that transfer available oxygen from hydrogen peroxide. These studies are performed within the framework of the development of methods of fine organic synthesis. At the same time, they also serve as a basis for the development of commercial-scale oxidation processes, because hydrogen peroxide is cheap and environmentally friendly. Compounds of many metals (W, V, Mo, Rh, Re, Ir, Ru, Pd, Ti, Co, Mg, Cu, Mn, Fe, Zr, Ag, Cr, Au are used as catalysts of oxidative transformations involving $H_2O_2$ [1–7].

Boron-containing catalysts in combination with hydrogen peroxide were not used for oxidation of alkenes. Only oxidation reactions with perborates, performed in carboxylic acids, in which the actual oxidizing species is per acid generated in situ from hydrogen peroxide and carboxylic acid, have been reported [8].

We used previously the $BF_3$–$H_2O_2$ system for preparing geminal bishydroperoxides from enol ethers [9]. In this study, when attempting to perform hydroperoxidation of related unsaturated compounds, alkenes, we discovered that in this system boron trifluoride, commonly known as a Lewis acid, shows a different behavior, inducing oxygen transfer from hydrogen peroxide to alkene. In oxidation of alkenes **Ia–If** in methanol, the major products are epoxides **IIa–IIf** and methoxyalkanols **IIIa–IIIf** (Scheme 1). Alkyl hydroperoxides were not detected, although it was reported that they are formed in oxidation of alkenes with $H_2O_2$, catalyzed by acids (e.g., $H_2SO_4$) [10, 11].

**Scheme 1**. Oxidation of alkenes **Ia–If** with the $BF_3$–$H_2O_2$ system in MeOH



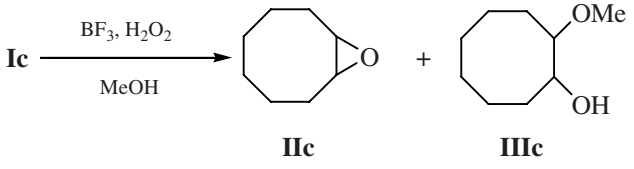

**Ia–If** **IIa–IIf** **IIIa–IIIf**

The capability of $H_2O_2$ in combination with $BF_3$ to induce Baeyer–Villiger oxidative rearrangement of ketones is well known [12, 13]. It is assumed in these papers that initially $H_2O_2$ and then the product of addition of $H_2O_2$ to the ketone form with $BF_3$ a complex in which the O–O bond is polarized, which is the necessary condition for the rearrangement to occur. Apparently, polarization of the O–O bond in the $BF_3$–$H_2O_2$ system imparts to the peroxy fragment the properties of a per acid which is the active species oxidizing the alkenes. Boron trifluoride is coordinated with the forming epoxide **II** and in the absence of an external nucleophile induces its polymerization and isomerization into a ketone. To make the synthesis of methoxyalkanols **III** more selective and prevent side reactions, the oxidation was performed in excess methanol. Previously $BF_3$ was already used as acid catalyst for opening of the oxirane ring with alcohols [14–18]. The interest in this reaction is due to the fact that opening of epoxides (e.g., styrene or indene epoxide [15], or glycidyl ethers [16–18]) with an alcohol yields, as a rule, a single regioisomer.

The objects of our study were cycloalkanes (cyclopentene **Ia**, cyclohexene **Ib**, cyclooctene **Ic**, cyclododecene **Id**; **Id**, as a mixture of *cis* and *trans* isomers) and alkenes with the double bond conjugated with an aromatic ring (styrene **Ie**, indene **If**).

The influence of the experimental conditions on the reaction of olefins with the $BF_3$–$H_2O_2$ system was studied with oxidation of cyclooctene **Ic** as example. The reaction yielded two major products: epoxycyclooctane **IIc** and 1-hydroxy-2-methoxycyclooctane **IIIc** (Scheme 2).

**Scheme 2**. Oxidation of cyclooctene **Ic** with the $BF_3$–$H_2O_2$ system in MeOH




To perform the oxidation, we prepared a mixture of methanol with a 7% solution of $H_2O_2$ in ether and $BF_3{\cdot}Et_2O$, after which compound **Ic** was added, and the homogeneous mixture was kept for 4–120 h at various temperatures: from –16 to –12, from 0 to 5, from 20 to 22, and from 35 to 38°C. As follows from Table 1, the temperature strongly affects the conversion of cyclooctene and the composition and yield of the products.

In the temperature range from –16 to 22°C, oxidation of cyclooctene **Ic** is slow. For example, at 20–22°C 70% conversion of cyclooctene was attained only in 80 h. At 35–38°C, the oxidation rate noticeably increases, and 41% conversion is attained in 4 h. An increase in the amount of $BF_3{\cdot}Et_2O$ from 0.5 to 1 equiv also accelerates the reaction: The conversion of **Ic** in 4 and 5 h becomes 75 and 91%, respectively.

Oxidation of **Ia–If** (Table 2) was performed under the conditions optimal for selective formation of methoxyalkanols. Namely, the majority of experiments were performed at 40–42 and 50–55°C in excess MeOH.

Thus, alkenes **Ia–If** in the temperature ranges 40–42 and 50–55°C in 10–22 h under the action of a threefold molar excess of $H_2O_2$ and one- or twofold molar excess of $BF_3{\cdot}Et_2O$ undergo oxidation with a high degree of conversion (80–100%). The oxidation results only slightly depend on the structure of the starting cycloalkene **Ia–If**. Methoxyalkanols **IIIa–IIIf** were prepared under optimized conditions in 61–72% yields, which is a good result taking into account the two-step reaction course (epoxidation followed by ring opening).

**Table 1**. Influence of reaction temperature and time on the conversion of cyclooctene **Ic** and yields of oxidation products **IIc** and **IIIc**[a]

| *T*, °C | Mol $BF_3{\cdot}Et_2O$/mol **Ic** | Conversion, % | Time, h | Yield, %[b] | |
|---|---|---|---|---|---|
| | | | | **IIc** | **IIIc** |
| From –16 to –12[c] | 0.5 | 12 | 120 | 73 | 10 |
| 0–5[d] | 0.5 | 28 | 120 | 70 | 12 |
| 20–22[d] | 0.5 | 70 | 80 | 57 | 27 |
| 35–38[d] | 0.5 | 41 | 4 | 62 | 28 |
| 35–38[d] | 1 | 75 | 4 | 45 | 38 |
| 35–38[d] | 1 | 91 | 5 | 38 | 42 |

[a] To 7% solution of $H_2O_2$ in $Et_2O$ (2.7 or 8.1 mmol of $H_2O_2$) we added 0.3 g of cyclooctene, 0.192 or 0.384 g of $BF_3{\cdot}Et_2O$, and 2 ml of MeOH. [b] GLC data; yields based on converted cyclooctene **Ic**. [c] 7% solution of $H_2O_2$ in $Et_2O$, 2.7 mmol. [d] 7% solution of $H_2O_2$ in $Et_2O$, 8.1 mmol.

**Table 2**. Results of oxidation of alkenes **Ia–If**[a]

| Alkene | Mol $H_2O_2$/mol **I** | Mol $BF_3$·$Et_2O$/mol **I** | *T*, °C | Time, h | Conversion, % | Products, yield, %[b] | |
|---|---|---|---|---|---|---|---|
| **Ia** | 3 | 1 | 40–42 | 22 | 100 | **IIa**, 0 | **IIIa**, 61 |
| **Ib** | 1 | 0.5 | 20–25 | 100 | 70 | **IIb**, 2 | **IIIb**, 42 |
| **Ib** | 3 | 1 | 20–25 | 40 | 41 | **IIb**, 2 | **IIIb**, 29 |
| **Ib** | 3 | 1 | 50–55 | 14 | 100 | **IIb**, 0 | **IIIb**, 69 |
| **Ic** | 3 | 1 | 50–55 | 12 | 100 | **IIc**, 0 | **IIIc**, 72 |
| **Id**[c] | 1 | 2 | 40–42 | 10 | 80 | **IId**, 2 | **IIId**, 51 |
| **Id**[c] | 3 | 1 | 50–55 | 15 | 100 | **IId**, 0 | **IIId**, 67 |
| **Ie** | 3 | 2 | 50–55 | 14 | 95 | **IIe**, 11 | **IIIe**, 62 |
| **Ie** | 3 | 2 | 50–55 | 16 | 100 | **IIe**, 0 | **IIIe**, 67 |
| **If** | 3 | 1 | 50–55 | 12 | 100 | **IIf**, 0 | **IIIf**, 69 |

[a] Reaction conditions: 2.7 mmol of alkene; 7% solution of $H_2O_2$ in $Et_2O$ (2.7 or 8.1 mmol of $H_2O_2$); 0.192, 0.384 or 0.767 g of $BF_3$·$Et_2O$; 6 ml of MeOH. [b] Based on alkene taken into reaction. [c] Cyclododecene **Id** was used as a mixture of *cis* (41%) and *trans* (59%) isomers.

The structure of the epoxides and methoxyalkanols obtained was determined by NMR spectroscopy and mass spectrometry. Oxidation of cyclododecene **Id** taken as a mixture of isomers yielded epoxide **IId** as a mixture of *cis* and *trans* isomers and 2-methoxy-1-cyclododecanol **IIId** as a mixture of *erythro* and *threo* isomers, which was confirmed by GLC–MS, doubling of signals in the $^{13}C$ NMR spectra, and comparison of the TLC data for **IId** and **IIId** with those for the references prepared by epoxidation of cyclododecene and methanolysis of its epoxide [19, 20]. 1-Methoxy-2,3-dihydro-1*H*-inden-1-ol **IIIf** was prepared as a mixture of *cis* and *trans* isomers, which was found by comparison with the known NMR spectra [21, 22]; the isomer ratio was ~1/2 (GC–MS).

Thus, we have discovered a new property of boron trifluoride: capability to activate hydrogen peroxide in oxidation of alkenes in methanol. The first step involves formation of an epoxide, and in the second step $BF_3$ catalyzes the epoxy ring opening with methanol, yielding 2-methoxy-alkan-1-ols. Alkyl hydroperoxides, expected products of acid-catalyzed addition of hydrogen peroxide, are not formed under these conditions.

## EXPERIMENTAL

The NMR spectra were recorded on Bruker AC-200 (200.13 MHz for $^1H$, 50.32 MHz for $^{13}C$), Bruker WM-250 (250.13 MHz for $^1H$, 62.9 MHz for $^{13}C$), and Bruker AM-300 (300.13 MHz for $^1H$, 75.4 MHz for $^{13}C$) spectrometers in $CDCl_3$ solutions. The TLC analysis was performed on Silufol UV-254 plates, sorbent Silpearl, eluent hexane–ethyl acetate (3:1 by volume). Column chromatography was performed with silica gel, 63–200 mesh (Merck). Cyclopentene, cyclohexene, cyclooctene, and indene were purchased from Acros. Styrene (pure grade), cyclododecene (pure grade), $Et_2O$, hexane, MeOH, ethyl acetate, $Na_2S_2O_3$·$5H_2O$, and 37% aqueous $H_2O_2$ (Russia) were used without additional purification. A solution of $H_2O_2$ in $Et_2O$ was prepared by extraction from 37% aqueous $H_2O_2$ solution, followed by drying over $MgSO_4$ [23]. The concentration of peroxides was determined by iodometric titration [24]. GC–MS analysis was performed with a Varian 3400 chromatograph equipped with an HP-101 capillary column (25 m×0.2 mm×0.2 μm). The injector temperature was 270°C. The carrier gas was helium, flow rate 1 ml $min^{-1}$. An MS Finnigan MAT ITP-700 ion trap was used. Mass range monitored: *m/z* 40–300. Ionizing electron energy 70 eV. Software: ITDS (Finnigan MAT), version 4.10.

**Oxidation of alkenes** *(general procedure)*. To a 7% solution of $H_2O_2$ in $Et_2O$ (2.7–8.1 mmol), we added an alkene (2.7 mmol), 0.192–0.767 g of $BF_3$·$Et_2O$, and 2 or 6 ml of MeOH. The homogeneous reaction mixture was kept at a temperature from –16 to 55°C for 4–240 h. In the course of synthesis at temperatures exceeding 35°C, the required amount of $Et_2O$ was evaporated in the initial period of the reaction to increase the boiling point of the mixture. After the reaction completion, 40 ml of $Et_2O$ and 10 ml of a 5% solution of $Na_2S_2O_3$·$5H_2O$ were added, the mixture

was stirred, and the ether layer was separated, washed with 2 × 5 ml of $H_2O$, and dried over $MgSO_4$. The mixtures were analyzed by GC–MS. Compounds **IIb–IIe** and **IIIa–IIIf** were isolated pure by column chromatography.

**2-Methoxycyclopentanol (IIIa)** [25]. Oil, $R_f$ 0.44. $^1$H NMR spectrum (250.13 MHz, CDCl3), δ, ppm: 1.45–2.03 m (6H, $CH_2$), 3.0–3.25 br.s (1H, OH), 3.31 s (3H, $CH_3$), 3.50–3.59 m (1H, C*H*COCH$_3$), 4.03–4.10 m (1H, C*H*OH). Mass spectrum, *m/z* ($I_{rel}$, %): 116 $[M]^+$ (9), 84 (100).

**7-Oxabicyclo[4.1.0]heptane (IIb)** [26, 27]. Mobile liquid. $^1$H NMR spectrum (300 MHz, $CDCl_3$), δ, ppm: 1.11–1.45 m (4H, $CH_2$), 1.69–1.94 m (4H, $CH_2$), 3.08 br.s (3H, $OCH_3$).

**2-Methoxycyclohexanol (IIIb)** [25]. Oil. $R_f$ 0.33. $^1$H NMR spectrum (250.13 MHz, $CDCl_3$), δ, ppm: 0.85–2.05 m (8H, $CH_2$), 2.75–2.90 m (1H, C*H*COCH$_3$), 3.20–3.41 m (5H, C*H*OH, $CH_3$, OH). $^{13}$C NMR spectrum (62.9 MHz, CDCl3), $\delta_C$, ppm: 23.7, 23.8, 28.1, 32.0, 56.0, 73.2, 84.6. Mass spectrum, *m/z* ($I_{rel}$, %): 130 $[M]^+$ (45), 112 $[M - H_2O]^+$ (8), 71 (100).

**9-Oxabicyclo[6.1.0]nonane (IIc)** [28]: mp 54–56°C (mp 55–56°C [29]). $R_f$ 0.81. $^1$H NMR spectrum (250.13 MHz, $CDCl_3$), δ, ppm: 1.10–1.65 m (12H, $CH_2$), 2.01–2.19 m (1H, CH), 2.78–2.91 m (1H, CH). $^{13}$C NMR spectrum (62.9 MHz, $CDCl_3$), δ, ppm: 25.4, 26.1, 26.4, 55.4. Mass spectrum, *m/z* ($I_{rel}$, %): 125 $[M - H]^+$ (1), 55 (100).

**2-Methoxycyclooctanol (IIIc)** [19]. Oil. $R_f$ 0.49. $^1$H NMR spectrum (250.13 MHz, $CDCl_3$), δ, ppm: 1.32–1.88 m (12H, $CH_2$), 2.90–3.12 m (2H, C*H*COCH$_3$, OH), 3.33 s (3H, $OCH_3$), 3.57 m (1H, C*H*OH). $^{13}$C NMR spectrum (62.9 MHz, $CDCl_3$), $\delta_C$, ppm: 23.5, 24.6, 25.7, 26.2, 26.8, 30.2, 56.3 ($CH_3$), 74.6 (CHOH), 86.4 (CHOMe). Mass spectrum, *m/z* ($I_{rel}$, %): 158 $[M]^+$ (0.5), 143 $[M - CH_3]^+$ (11), 71 (100).

**13-Oxabicyclo[10.1.0]tridecane (IId)** [20] (mixture of *cis* and *trans* isomers). $R_f$ 0.82, 0.88. $^1$H NMR spectrum (300 MHz, $CDCl_3$), δ, ppm: 1.14–1.61 m (36H, $CH_2$), 1.72–2.19 m (4H, C$H_2$CHO), 2.62–2.90 m (4H, CHO). $^{13}$C NMR spectrum (75 MHz, $CDCl_3$), δ, ppm: 22.4, 23.4, 23.6, 23.8, 23.9, 24.0, 25.0, 25.5, 25.9, 26.6, 31.3 ($CH_2$), 58.0, 58.1, 59.8, 59.9 (CHO). Mass spectrum, *m/z* ($I_{rel}$, %): 164 $[M - H_2O]^+$ (8), 55 (100).

**2-Methoxycyclododecanol (IIId)** [19] (mixture of *erythro* and *threo* isomers). $R_f$ 0.52, 0.67. $^1$H NMR spectrum (250.13 MHz, $CDCl_3$), δ, ppm: 1.18–1.70 m (40H, $CH_2$), 3.65–3.9 br.s (2H, OH), 3.11–3.39 m (8H, $CH_3$, CHOMe), 3.65–3.87 m (2H, C*H*OH). $^{13}$C NMR spectrum (62.9 MHz, $CDCl_3$), δ, ppm: 21.5–25.1 (18C, $CH_2$), 28.2, 29.3 ($CH_2$), 57.1, 57.3 ($CH_3$), 69.7, 69.9 (CHOH), 82.4, 82.7 (CHOMe). Erythro and threo isomers. Mass spectrum, *m/z* ($I_{rel}$, %): 214 $[M]^+$ (3), 199 $[M - CH_3]^+$ (15), 71 (100) and 214 $[M]^+$ (2), 199 $[M - CH_3]^+$ (14), 71 (100).

**2-Phenyloxirane (IIe)** [28]. Oil, $R_f$ 0.81. $^1$H NMR spectrum (300 MHz, $CDCl_3$), δ, ppm: 2.82 d.d (1H, $CH_2$, *J* 2.6, 5.4 Hz), 3.16 d.d (1H, $CH_2$, *J* 4.1, 5.4 Hz), 3.88 d.d (1H, CH, *J* 2.6, 4.1 Hz), 7.28–7.41 m (5H, Ph). Mass spectrum, *m/z* ($I_{rel}$, %): 120 $[M]^+$ (12), 91 $[PhCH_2]^+$ (100).

**2-Methoxy-2-phenylethanol (IIIe)** [25]. Oil, $R_f$ 0.38. $^1$H NMR spectrum (300 MHz, $CDCl_3$), δ, ppm: 2.51–2.73 br.s (1H, OH), 3.30 s (3H, $CH_3$), 3.58–3.71 m (2H, $CH_2$), 4.30 d.d (1H, CH, *J* 3.7, 8.5 Hz), 7.27–7.40 m (5H, Ph). Mass spectrum, *m/z* ($I_{rel}$, %): 135 $[M - H_2O]^+$ (2), 121 $[M - MeO]^+$ (100).

**1-Methoxy-2,3-dihydro-1*H*-inden-2-ol (IIIf)** [21, 22] [mixture of cis and trans isomers, 2:1 (GLC–MS)]. $R_f$ 0.21, 0.28. $^1$H NMR spectrum (300 MHz, $CDCl_3$), δ, ppm: 2.45–3.38 m (3H, $CH_2$, OH), 3.54, 3.57 s ($CH_3$), 4.46–4.68 m (2H, CH), 7.19–7.45 m (4H, CH, Ar). $^{13}$C NMR spectrum (75 MHz, $CDCl_3$), δ, ppm: 39.0, 39.1 ($CH_2$), 57.1, 57.2 ($CH_3$), 72.3, 78.0 (CHOH), 83.9, 90.3 (CHOMe), 125.1, 125.2, 125.5, 126.5, 126.8, 129.0, 139.6, 140.1, 140.13, 141.0 (C, Ar). Mass spectrum, *m/z* ($I_{rel}$, %): 164 $[M]^+$ (12), 104 $[PhCH{=}CH_2]^+$ (100).

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