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Transformations of Peroxide Products of Olefin Ozonolysis in Tetrahydrofuran in Reactions with Hydroxylamine and Semicarbazide Hydrochlorides

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Abstract—Treatment with hydroxylamine and semicarbazide hydrochlorides of peroxide products obtained by ozonolysis of olefins in tetrahydrofuran gives mainly carboxylic acids and their derivatives.

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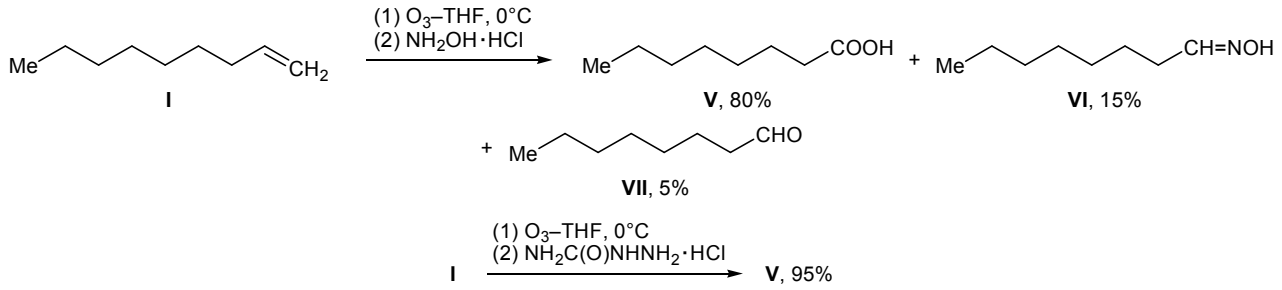
Solvent nature is the crucial factor determining the structure of peroxide ozonolysis products and their subsequent transformations [1, 2]. We previously demonstrated the efficiency of semicarbazide and hydroxylamine hydrochlorides in the transformations of intermediate peroxides in alcohols into carbonyl compounds and their derivatives [3, 4]. In continuation of these studies, in the present work we examined transformations of peroxide products of olefin ozonolysis in tetrahydrofuran by the action of the same reagents. As substrates we selected olefins with mono-, di-, and trisubstituted double bond, namely non-1-ene (**I**), cyclooctene (**II**), (–)-α-pinene (**III**, *ee* 86%), and Δ^3-carene (**IV**, *ee* 100%).

Ozonolysis of non-1-ene (**I**) in THF at 0°C, followed by reduction of the peroxide ozonolysis products with hydroxylamine hydrochloride gave octanoic acid (**V**) and small amounts of octanal oxime (**VI**), and octanal (**VII**). Ozonolysis of **I** under the same conditions and subsequent treatment with semicarbazide hydrochloride afforded acid **V** in a high yield (95%; Scheme 1).

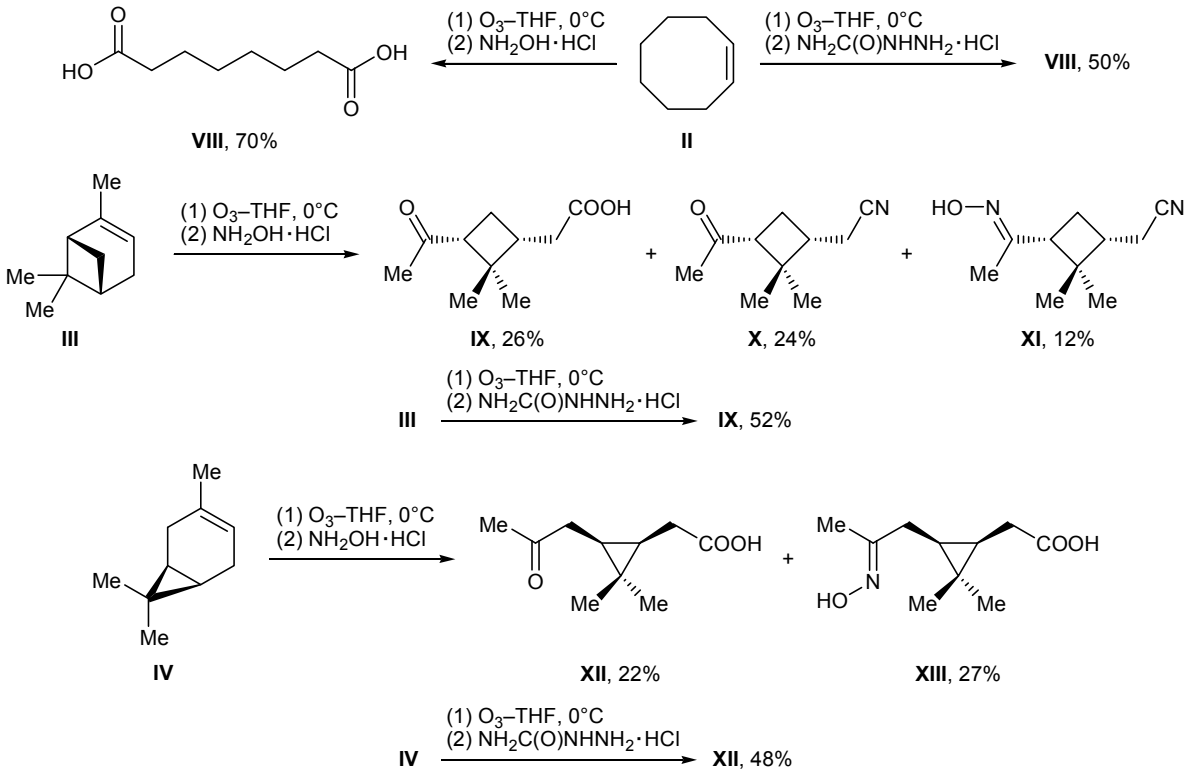
By ozonolysis of cyclic olefins **II**–**IV** in tetrahydrofuran and subsequent treatment with hydroxylamine hydrochloride we obtained the corresponding acids **VIII**, **IX**, and **XII**. In addition, in the reaction with α-pinene (**III**) we isolated precursors of acid **IX**, nitrile **X** and hydroxyimino nitrile **XI**. Hydroxyimino carboxylic acid **XIII** was formed in the ozonolysis of (+)-3-carene (**IV**). When the peroxide ozonolysis products derived from cyclooctene (**II**) and monoterpenes **III** and **IV** were reduced with semicarbazide hydro-

Scheme 1.



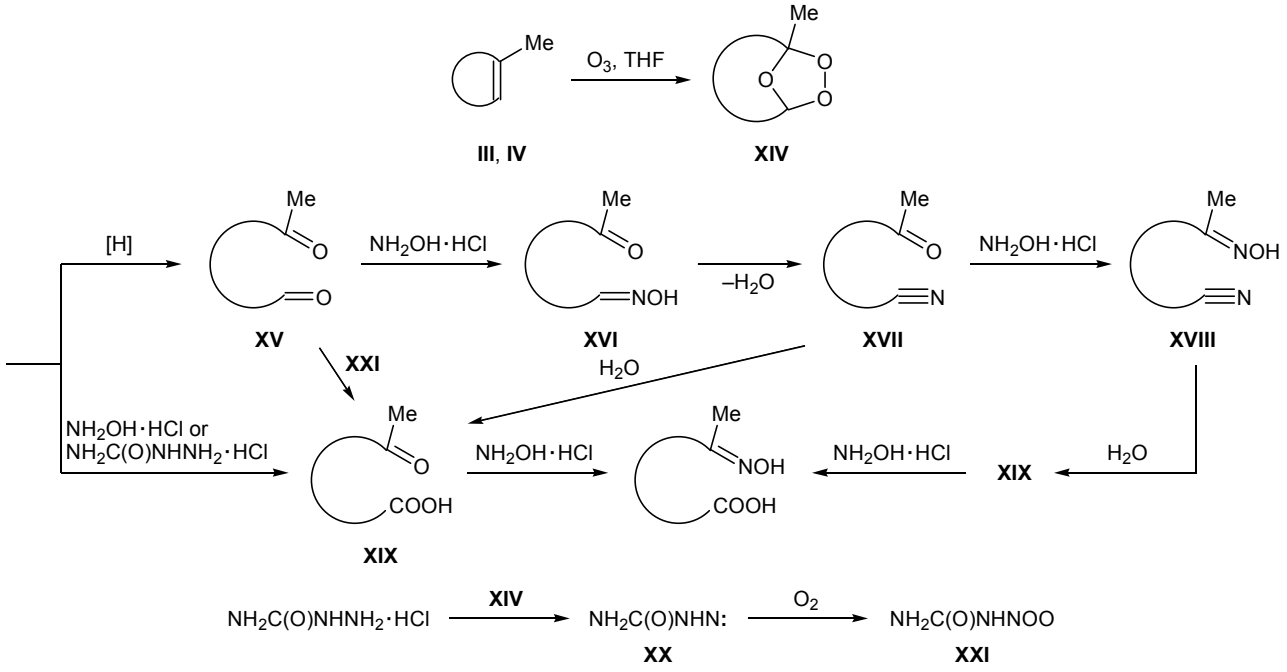
† Deceased.

Scheme 2.



chloride, only the corresponding carboxylic acids **VIII**, **IX**, and **XII** were isolated in moderate yields (Scheme 2). Presumably, ozonolysis of olefins **III** and **IV** in an ether-like solvent (tetrahydrofuran) gives ozonides **XIV** as primary products, and hydroxylamine and semicarbazide hydrochlorides act as reducing agents which convert ozonides into carbonyl compounds **XV** and also promote their isomerization to carboxylic acids **XIX** (Scheme 3). On the other hand, intermediate keto aldehydes **XV** can undergo the trans-

Scheme 3.



formation sequence aldehyde **XV** → oxime **XVI** → nitrile **XVII** or be oxidized with nitroso oxide **XXI** generated by oxidation of semicarbazide hydrochloride first with ozonide **XIV** and then with oxygen through intermediate nitrene **XX** [5]. Furthermore, oximation of the ketone carbonyl group is possible (Scheme 3).

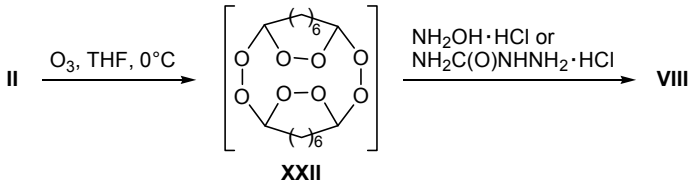
Peroxide **XXII** formed in the ozonolysis of cyclooctene **II** is enriched in active oxygen [1, 2], and it is capable of undergoing direct rearrangement into dicarboxylic acid **VIII** by the action of semicarbazide or hydroxylamine hydrochloride (Scheme 4).

Our attempts to raise the yield of carboxylic acids by promoting formation of intermediate hydroxy hydroperoxides [6] via addition of water as co-solvent were unsuccessful. In all cases, strong tarring and reduction of both overall yield and carboxylic acid fraction were observed. When hydroxylamine hydrochloride was used as reducing agent, the reaction mixtures contained precursors of carboxylic acids, namely octanenitrile (**XXIII**) in the reaction with non-1-ene (**I**), dinitrile **XXIV** and 7-cyanoheptanoic acid (**XXV**) in the ozonolysis of cyclooctene (**II**), and oxime **XXVI** in the reaction with α-pinene (**III**).

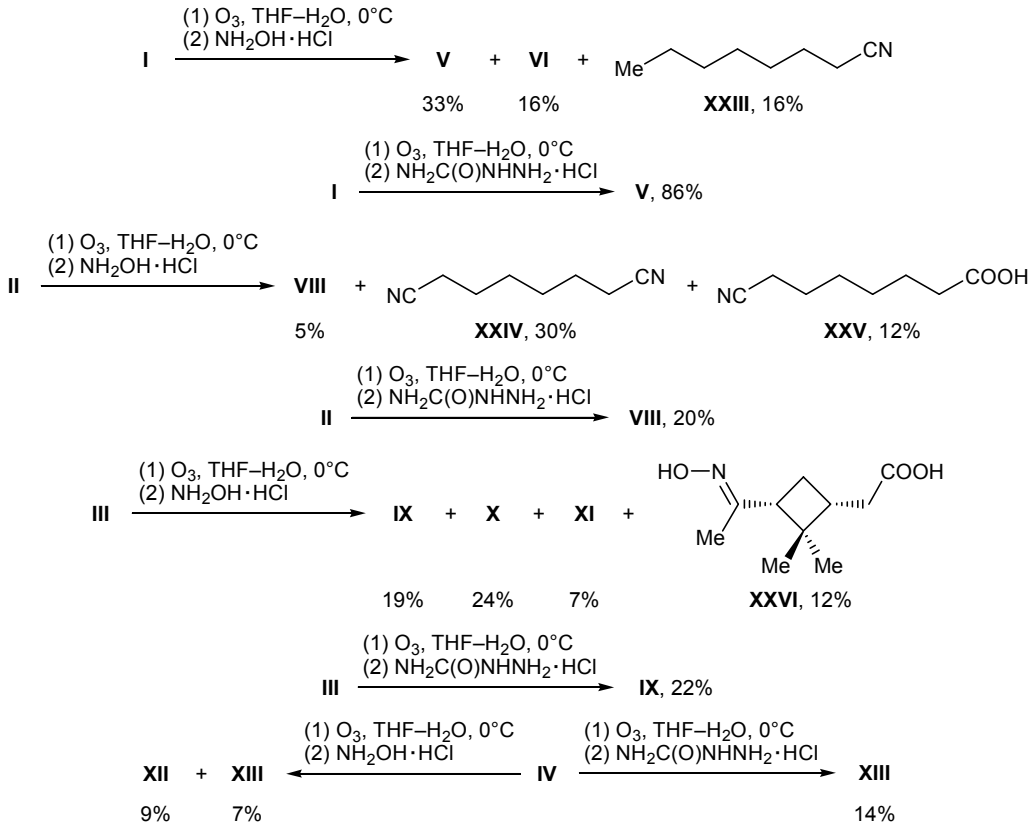
EXPERIMENTAL

The IR spectra were recorded from thin films on a Shimadzu IR Prestige-21 spectrometer with Fourier transform. The NMR spectra were measured on Bruker AM-300 and Bruker Avance III 500 high-resolution spectrometers (300 and 500 MHz for ^{1}H and 75.47 MHz for ^{13}C) using $CDCl_3$ as solvent and tetramethylsilane as internal reference. The ^{1}H signals were

Scheme 4.



Scheme 5.



assigned, and the coupling constants were determined, with the aid of double resonance and COSY ^1H–^1H two-dimensional homonuclear correlation techniques. The ^{13}C NMR spectra were recorded with broad-band decoupling from protons and in the JMOD mode. GLC analysis was performed on Chrom-5 (1.2-m column packed with 5% of SE-30 on Chromaton N-AW-DMCS, 0.16–0.20 mm; oven temperature 50–300°C) and Chrom-41 chromatographs (2.4-m column packed with PEG-6000; oven temperature 50–200ºC); carrier gas helium. *Sorbfil* silica gel (Russia) was used for analytical thin-layer chromatography. The products were isolated by column chromatography on silica gel (70–230 mesh, Lancaster, England). The optical rotations were measured on a Perkin Elmer 241-MC polarimeter. The elemental compositions of all isolated compounds were consistent with the theoretical ones. The ozonizer efficiency was 40 mmol/h.

Ozonolysis of olefins I–IV (*general procedure*). A solution of 10.0 mmol of olefin **I–IV** in 30 mL of THF was cooled to 0°C, and an ozone–oxygen mixture was bubbled through the solution until 10.5 mmol of ozone was absorbed.

Treatment of peroxide ozonolysis products with hydroxylamine hydrochloride (*general procedure*). The mixture obtained after ozonolysis was purged with argon, 2.43 g (35.0 mmol) of hydroxylamine hydrochloride was added under stirring at 0°C, and the mixture was stirred at room temperature until peroxide products disappeared (starch–iodine test). The solvent was distilled off, the residue was dissolved in 150 mL of chloroform, and the solution was washed with brine (4×15 mL), dried over Na_2SO_4, and evaporated.

The residue obtained in the reaction with non-1-ene (**I**), 1.37 g, was separated by chromatography on silica gel using hexane–*tert*-butyl methyl ether (20:1 to 1:1) as eluent to isolate 1.15 g (80%) of octanoic acid (**V**), 0.20 g (15%) of octanal oxime (**VI**), and 0.07 g (5%) of octanal (**VII**).

Octanoic acid (V). R_f 0.25 (hexane–*tert*-butyl methyl ether, 2:1). ^1H NMR spectrum, δ, ppm: 0.87 t (3H, 8-H, J = 6.6 Hz), 1.20–1.37 m (6H, 4-H, 5-H, 6-H, 1.43–1.58 m (2H, 7-H), 1.56–1.69 m (2H, 3-H), 2.24 t (2H, 2-H, J = 6.7 Hz), 11.45 br.s (1H, OH). The IR and ^{13}C NMR spectra were identical to those reported previously [7].

Octanal oxime (VI). R_f 0.53 (hexane–*tert*-butyl methyl ether, 2:1). The IR and NMR spectra were identical to those reported previously [4].

Octanal (VII). IR spectrum (KBr): ν 1718 cm^{-1} (C=O). The NMR spectra were identical to those reported in [8].

The residue obtained in the reaction with cyclooctene (**II**), 1.64 g, was separated by silica gel chromatography using hexane–*tert*-butyl methyl ether (20:1 to 1:1) as eluent to isolate 1.21 g (70%) of octanedioic acid (**VIII**), R_f 0.21 (hexane–*tert*-butyl methyl ether, 2:1). IR spectrum (KBr), ν, cm^{-1}: 1712 (C=O), 3242 (OH). ^1H NMR spectrum, δ, ppm: 1.31–1.38 m (4H, 4-H, 5-H), 1.50–1.70 m (4H, 3-H, 6-H), 2.49 t (4H, 2-H, 7-H, J = 6.3 Hz). ^{13}C NMR spectrum, δ_C, ppm: 24.56 t (C^4, C^5), 29.43 t (C^3, C^6), 33.82 t (C^2, C^7), 177.06 s (COOH) [9].

The residue obtained in the ozonolysis of (–)-α-pinene (**III**), 2.0 g, was separated by silica gel chromatography using hexane–*tert*-butyl methyl ether (20:1 to 1:1) as eluent to isolate 0.47 g (26%) of keto acid **IX**), 0.40 g (24%) of keto nitrile **X**, and 0.21 g (12%) of hydroxyimino nitrile **XI**.

(3-Acetyl-2,2-dimethylcyclobutyl)acetic acid (IX) [10]**.** R_f 0.21 (hexane–*tert*-butyl methyl ether, 4:1), $[\alpha]_D^{20}$ = –39.8° (c = 0.8164, CH_2Cl_2). IR spectrum (KBr), ν, cm^{-1}: 1715 (C=O), 3330 (OH). ^1H NMR spectrum, δ, ppm: 0.83 s (3H, *cis*-CH_3), 1.29 s (3H, *trans*-CH_3), 1.83–1.92 m (1H, *cis*-4-H), 1.94 d.d (1H, *trans*-4-H, J = 10.1, 10.7 Hz), 2.03 s (3H, CH_3CO), 2.12–2.36 m (3H, 1-H, 1-CH_2), 2.83 d.d (1H, 3-H, J = 10.1, 7.5 Hz), 9.70 br.s (1H, COOH). ^{13}C NMR spectrum, δ_C, ppm: 16.78 q (*cis*-CH_3), 22.12 t (C^4), 29.68 q ($\mathbf{C}H_3CO$), 30.26 q (*trans*-CH_3), 35.95 t (1-CH_2), 37.10 d (C^1), 42.94 s (C^2), 52.88 d (C^3), 178.28 s (COOH), 208.27 s (C=O).

(3-Acetyl-2,2-dimethylcyclobutyl)acetonitrile (X) [10]**.** R_f 0.33 (hexane–*tert*-butyl methyl ether, 3:2), $[\alpha]_D^{20}$ = –131.16° (c = 1.02, $CHCl_3$) [10]. IR spectrum (KBr): ν 2220 cm^{-1} (CN). ^1H NMR spectrum, δ, ppm: 0.95 s (*cis*-CH_3), 1.40 s (*trans*-CH_3), 1.94 d.t (1H, *cis*-4-H, J = –11.0, 9.5 Hz), 2.03 d.t (1H, *trans*-4-H, J = –11.0, 8 Hz), 2.08 s (3H, CH_3CO), 2.32 d.d (1H, 1-CH_2, J = 15.2, 8.00 Hz), 2.32 d.d (1H, 1-CH_2, J = 15.2, 7.8 Hz), 2.37–2.43 m (1H, 1-H), 2.90 d.d (1H, 3-H, J = 9.5, 8.0 Hz). ^{13}C NMR spectrum, δ_C, ppm: 16.54 q (CH_3), 17.25 t (CH_2), 22.89 t (C^4), 29.86 q (CH_3), 30.27 q ($\mathbf{C}H_3CO$) 38.16 d (C^1), 42.97 s (C^2), 53.81 d (C^3), 118.56 s (CN), 206.63 s (C=O).

{3-[(*E*)-(1-Hydroxyimino)ethyl]-2,2-dimethylcyclobutyl}acetonitrile (XI). R_f 0.30 (hexane–*tert*-butyl methyl ether, 3:2). The IR and NMR spectra of **XI** were identical to those given in [4].

The residue obtained in the ozonolysis of (+)-3-carene (**IV**), 1.70 g, was separated by silica gel chromatography using hexane–*tert*-butyl methyl ether (20:1 to 1:1) as eluent to isolate 0.54 g (27%) of hydroxyimino carboxylic acid **XIII** and 0.40 g (22%) of keto acid **XII**.

[2,2-Dimethyl-3-(2-oxopropyl)cyclopropyl]acetic acid (XII). R_f 0.19 (hexane–*tert*-butyl methyl ether, 4:1), $[\alpha]_D^{20}$ = –14° (*c* = 2.23, CH_2Cl_2). IR spectrum (KBr), ν, cm^{-1}: 1712 (C=O), 3331 (OH). ^1H NMR spectrum, δ, ppm: 0.94 s (3H, *cis*-CH_3), 0.98 d.d (1H, 1-H, *J* = 10.2, 6.3 Hz), 1.09 d.d (1H, 3-H, *J* = 10.2, 5.1 Hz), 1.10 s (3H, *trans*-CH_3), 2.04 s (3H, CH_3CO), 2.11–2.20 m (2H, 1-CH_2), 2.30–2.38 m (3-CH_2). ^{13}C NMR spectrum, δ_C, ppm: 14.12 q (*cis*-CH_3), 17.09 s (C^2), 20.90 d (C^1), 22.27 d (C^3), 28.41 q (*trans*-CH_3), 30.78 t (3-CH_2), 30.78 q ($COCH_3$), 32.96 t (1-CH_2), 177.02 s (COOH), 212.38 s (C=O).

{3-[(2*E*)-2-(Hydroxyimino)propyl]-2,2-dimethylcyclopropyl}acetic acid (XIII) [6]**.** R_f 0.17 (hexane–*tert*-butyl methyl ether, 4:1). IR spectrum (KBr), ν, cm^{-1}: 1633 (C=N), 1714 (C=O), 3334 (OH). ^1H NMR spectrum, δ, ppm: 0.76 d.d.d (1H, 1-H, *J* = 9.2, 7.2, 1.8 Hz), 0.94 d.d.d (1H, 3-H, *J* = 9.2, 7.7, 2.1 Hz), 0.95 s (3H, *cis*-CH_3), 1.09 s (3H, *trans*-CH_3), 1.93 s (3H, CH_3), 2.27–2.33 m (1H, 1-CH_2), 2.28 d.d (1H, 3-CH_2, *J* = –13.1, 5.2 Hz), 2.31 d.d (1H, 3-CH_2, *J* = –13.1, 7.1 Hz), 2.48 d.d (1H, 1-CH_2, *J* = 15.7, 6.9 Hz), 8.00 br.s (2H, OH). ^{13}C NMR spectrum, δ_C, ppm: 13.49 q (**C**H_3C=N), 14.90 q (CH_3), 17.42 s (C^3), 21.79 d (C^1), 22.38 d (C^2), 28.53 q (CH_3), 29.66 t (1-CH_2), 30.88 t (3-CH_2), 158.74 s (C=N), 175.05 s (COOH).

Treatment of peroxide ozonolysis products of olefins I–IV with semicarbazide hydrochloride (*general procedure*). The reaction mixture obtained after ozonolysis was purged with argon, 3.90 g (35.0 mmol) of semicarbazide hydrochloride was added under stirring at 0°C, and the mixture was stirred at room temperature until peroxide compounds disappeared (starch–iodine test). The solvent was distilled off, the residue was dissolved in chloroform (150 mL), and the solution was washed with brine (4×15 mL), dried over Na_2SO_4, and evaporated.

From non-1-ene (**I**) we obtained 1.36 g (95%) of octanoic acid (**V**).

From cyclooctene (**II**) we obtained 0.88 g (50%) of octanedioic acid (**VIII**).

The residue obtained in the ozonolysis of (–)-α-pinene (**III**), 1.72 g, was subjected to silica gel chromatography using hexane–*tert*-butyl methyl ether (20:1 to 1:1) as eluent to isolate 0.95 g (52%) of acid **IX**.

The residue, 1.80 g, obtained in the ozonolysis of (+)-3-carene (**IV**) was subjected to silica gel chromatography using hexane–*tert*-butyl methyl ether (20:1 to 1:1) as eluent to isolate 0.88 g (48%) of acid **XII**.

Ozonolysis of olefins I–IV in the system THF–water (*general procedure*). A solution of 10.0 mmol of olefin **I**–**IV** in a mixture of 34 mL of THF and 1.8 mL of water was cooled to 0°C, and an ozone–oxygen mixture was bubbled through the solution until 10.5 mmol of ozone was absorbed.

Treatment of peroxide ozonolysis products with hydroxylamine hydrochloride (*general procedure*). The mixture obtained after ozonolysis was purged with argon, 2.43 g (35.0 mmol) of hydroxylamine hydrochloride was added under stirring at 0°C, and the mixture was stirred at room temperature until peroxide products disappeared (starch–iodine test). The solvent was distilled off, the residue was dissolved in 150 mL of chloroform, and the solution was washed with brine (4×15 mL), dried over Na_2SO_4, and evaporated.

The residue obtained in the ozonolysis of non-1-ene (**I**), 1.10 g, was separated by silica gel chromatography (hexane–*tert*-butyl methyl ether, 20:1 to 1:1) to isolate 0.47 g (33%) of octanoic acid (**V**), 0.24 g (16%) of octanal oxime (**VI**), and 0.20 g (16%) of nitrile **XXIII** (R_f 0.26, hexane–*tert*-butyl methyl ether, 2:1). The IR and NMR spectra of **XXIII** were identical to those reported in [11].

The residue obtained in the ozonolysis of cyclooctene (**II**), 0.72 g, was separated by silica gel chromatography (hexane–*tert*-butyl methyl ether, 20:1 to 1:1) to isolate 0.41 g (30%) of octanedinitrile (**XXIV**), 0.19 g (12%) of 7-cyanoheptanoic acid (**XXV**), and 0.09 g (5%) of dicarboxylic acid **VIII**.

Octanedinitrile (XXIV). R_f 0.25 (hexane–*tert*-butyl methyl ether, 2:1). IR spectrum (KBr): ν 2245 cm^{-1} (CN). The ^1H NMR spectrum was identical to that given in [12]. ^{13}C NMR spectrum, δ_C, ppm: 16.98 t (C^2, C^7), 25.02 t (C^3, C^6), 27.78 t (C^4, C^5), 119.65 s (CN).

7-Cyanoheptanoic acid (XXV). R_f 0.23 (hexane–*tert*-butyl methyl ether, 2:1). IR spectrum (KBr), ν, cm^{-1}: 3242 (OH), 2245 (CN), 1712 (C=O). ^1H NMR spectrum, δ, ppm: 1.41–1.60 m (4H, 4-H, 5-H), 1.62–1.73 m (4H, 3-H, 6-H), 2.25 t (2H, 2-H), 2.35 t (7-H). ^{13}C NMR spectrum, δ_C, ppm: 16.82 t (C^7), 29.05 t (C^3), 28.08 t (C^4), 28.26 t (C^5), 25.45 t (C^6), 33.51 t (C^2), 119.38 s (CN), 177.52 s (COOH).

The residue obtained in the ozonolysis of (–)-α-pinene (**III**), 1.29 g, was separated by silica gel chromatography (hexane–*tert*-butyl methyl ether, 20:1 to 1:1) to isolate 0.39 g (24%) of keto nitrile **X**, 0.34 g (19%) of keto acid **IX**, 0.14 g (12%) of hydroxyimino acid **XXVI**, and 0.22 g (7%) of hydroxyimino nitrile **XI**.

{3-[(*E*)-1-(Hydroxyimino)ethyl]-2,2-dimethylcyclobutyl}acetic acid (XXVI). R_f 0.23 (hexane–*tert*-butyl methyl ether, 4:1). IR spectrum (KBr), ν, cm^{-1}: 3330 (OH), 1715 (C=O). ^1H NMR spectrum, δ, ppm: 0.83 s (3H, *cis*-CH_3), 1.17 s (3H, *trans*-CH_3), 1.77 s (3H, CH_3CO), 6.5 br.s (NOH), 9.7 br.s (COOH). ^{13}C NMR spectrum, δ_C, ppm: 14.52 q (CH_3C=N), 17.11 q (CH_3), 24.48 t (C^4), 29.93 q (CH_3), 34.63 t (1-CH_2), 39.38 d (C^1), 42.12 s (C^2), 47.74 d (C^3), 157.98 s (C=N), 177.78 s (COOH).

The residue obtained in the ozonolysis of (+)-3-carene (**IV**), 1.02 g, was separated by silica gel chromatography (hexane–*tert*-butyl methyl ether, 20:1 to 1:1) to isolate 0.18 g (9%) of hydroxyimino acid **XIII** and 0.12 g (7%) of keto acid **XII**.

Treatment of peroxide ozonolysis products of olefins I–IV with semicarbazide hydrochloride (*general procedure*). The reaction mixture obtained after ozonolysis was purged with argon, 3.90 g (35.0 mmol) of semicarbazide hydrochloride was added under stirring at 0°C, and the mixture was stirred at room temperature until peroxide compounds disappeared (starch–iodine test). The solvent was distilled off, the residue was dissolved in chloroform (150 mL), and the solution was washed with brine (4×15 mL), dried over Na_2SO_4, and evaporated.

From non-1-ene (**I**) we obtained 1.24 g (86%) of octanoic acid (**V**).

From cyclooctene (**II**) we obtained 0.34 g (20%) of octanedioic acid (**VIII**).

The residue obtained in the ozonolysis of (–)-α-pinene (**III**), 1.93 g, was subjected to silica gel chromatography (hexane–*tert*-butyl methyl ether, 20:1 to 1:1) to isolate 0.41 g (22%) of keto acid **IX**.

The residue obtained in the ozonolysis of (+)-3-carene (**IV**), 1.67 g, was subjected to silica gel chromatography (hexane–*tert*-butyl methyl ether, 20:1 to 1:1) to isolate 0.25 g (14%) of keto acid **XII**.

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