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Specificity of the Reaction of Tetranitromethane with Alkenes in Nitromethane

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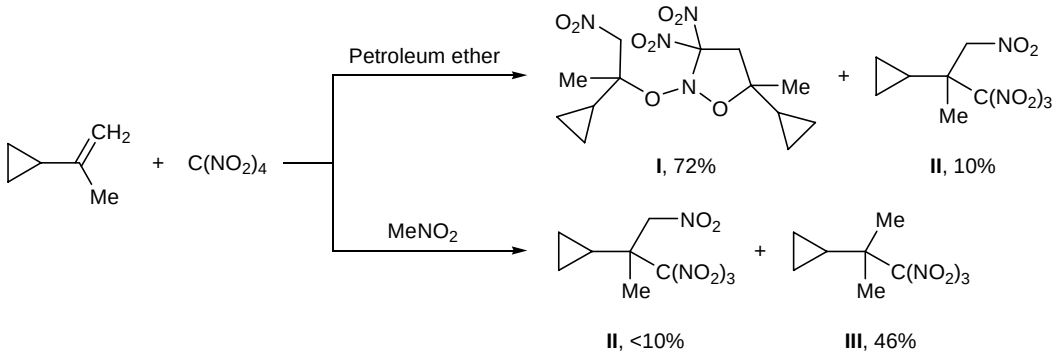
Abstract—Reactions of tetranitromethane with a number of di- and trisubstituted olefins in nitromethane were studied. Vinylcyclopropanes and phenylcycloalkenes reacted with tetranitromethane in an unexpected fashion, leading to formation of alkyltrinitromethanes and vicinal nitro alcohols.

Reactions of tetranitromethane with alkenes have been the subject of numerous studies [1–3]. According to published data, tetranitromethane reacts with olefins to afford, depending on the substrate structure, 3,3-dinitroisoxazolidines, tetranitropropanes, nitroalkenes, or α-nitro ketones [1]. The data on solvent effect in reactions of tetranitromethane with alkenes are few in number and are often contradictory. Ratsino *et al*. [4] were the only to report that the reaction of tetranitromethane with substituted styrenes follow different pathways, depending on the polarity of aprotic solvent [4].

In continuation of our recent studies on reactions of tetranitromethane with olefins containing small rings [5–8], in the present work we made an attempt to examine solvent effect on these reactions. We previously showed that methylidenecyclobutane and bicyclobutylidene react with tetranitromethane in such solvents as petroleum ether and methylene chloride to give isoxazolidine derivatives [5–8]. We also found that the use of nitromethane as solvent changes neither the reaction direction nor the yield of the corresponding isoxazolidines.

However, solvent effect on the reactions of tetranitromethane with vinylcyclopropanes and phenylcycloalkenes may be considerable since the substrates could give rise to stable carbocations [9, 10]; therefore, unusual products may be formed, depending on the conditions. According to our previous data [6], tetranitromethane reacts with 2-cyclopropylpropene in petroleum ether to give a mixture of isoxazolidine **I** and 2-cyclopropyl-2-methyl-1,1,1,3-tetranitropropane (**II**). When the same reaction was carried out in nitromethane, the major product was trinitro derivative **III** (Scheme 1). Formalistically, compound **III** may be regarded as product of addition of $CH(NO_2)_3$ to the initial alkene. The yield of the expected adduct **II** did not exceed 10%. The structure of **III** was unambiguously confirmed by the ^1H and ^{13}C NMR and mass spectra and elemental analysis (see Experimental). It should be noted that formation of trinitro derivatives in

Scheme 1.



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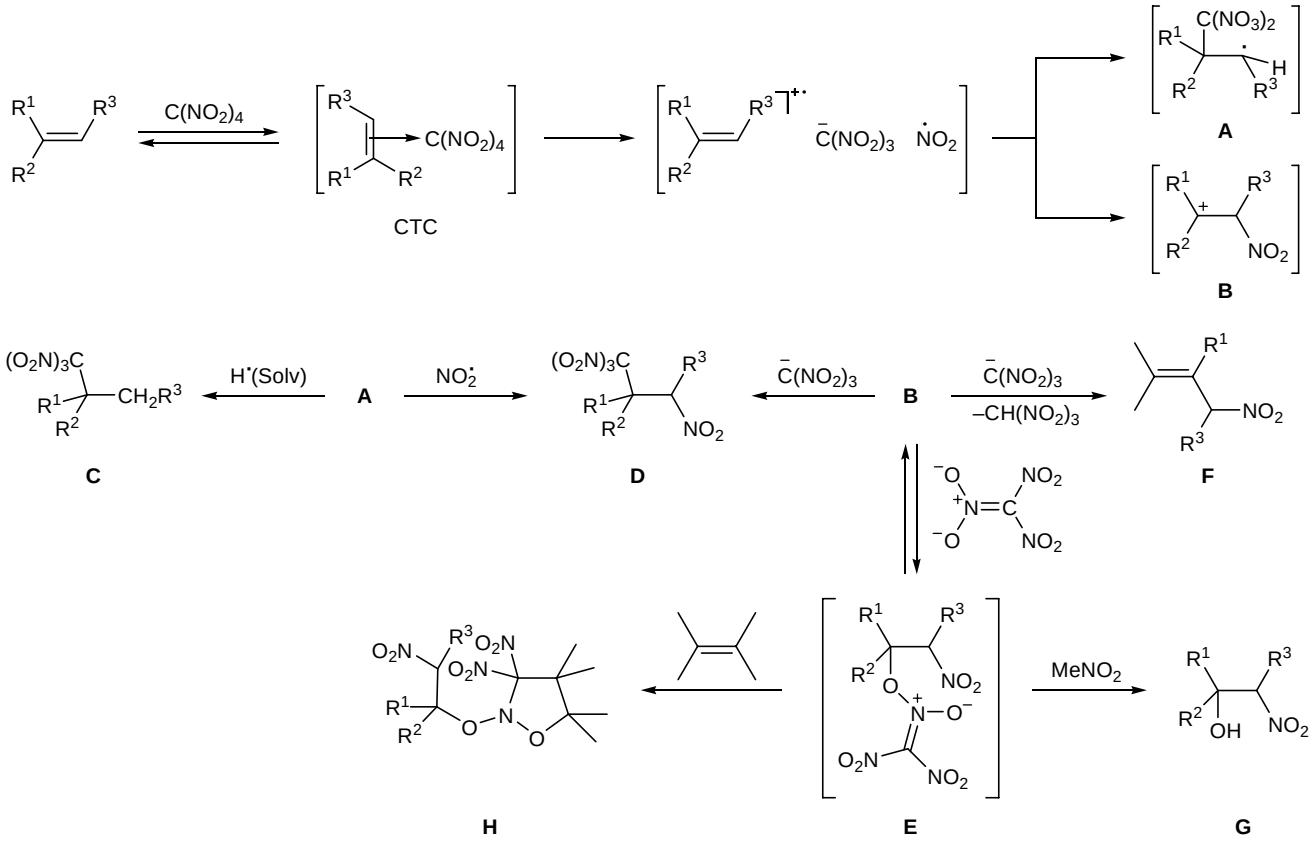
Scheme 2.



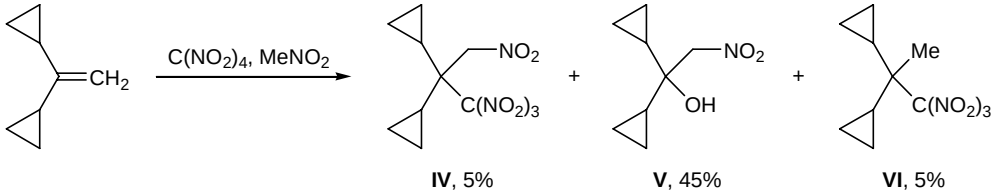
reactions of olefins with tetranitromethane was not reported previously.

Scheme 2 illustrates the reaction mechanism which was proposed on the basis of modern views on the mechanism of tetranitromethane reactions with olefins, including the results of our studies [8, 11]. Presumably, tetranitromethane and olefin initially form charge-transfer complex (CTC). Electron transfer in CTC from the donor (olefin) to tetranitromethane gives the corresponding radical cation, nitryl radical, and trinitromethyl anion. Recombination of the radical–ion pair (vinylcyclopropane radical cation and trinitromethyl anion) leads to radical **A**. The regioselectivity of this process is controlled by strong electron density deficit on the internal carbon atom in substituted vinylcyclopropane. Primary radical **A** abstracts hydrogen from the solvent to produce trinitroalkane **C** or takes up $^{\cdot}NO_2$ thus leading to tetranitro derivative **D**.

The reaction of 1,1-dicyclopropylethene with tetranitromethane in petroleum ether gives 2,2-dicyclopropyl-1,1,1,3-tetranitropropane (**IV**) as the only product (yield 80%) [6]. Surprisingly, the same substrate reacted with tetranitromethane in nitromethane to give vicinal nitro alcohol **V** as the major product, though trace amounts of adduct **IV** and trinitro derivative **VI** were also formed (Scheme 3).

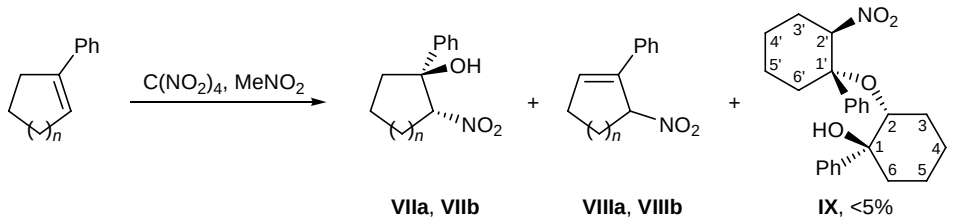
Reactions of 1-phenylcycloalkenes with tetranitromethane also showed an appreciable dependence on the solvent. According to the data of [12], the products of these reactions in petroleum ether were the corresponding tetranitro derivatives. 1-Phenylcyclopentene and 1-phenylcyclohexene reacted with tetranitro-

Scheme 3.



IV, 5% **V**, 45% **VI**, 5%

Scheme 4.



VII, *n* = 1, yield 40% (**a**); *n* = 2, 41% (**b**); **VIII**, *n* = 1, 26% (**a**); *n* = 2, 33% (**b**).

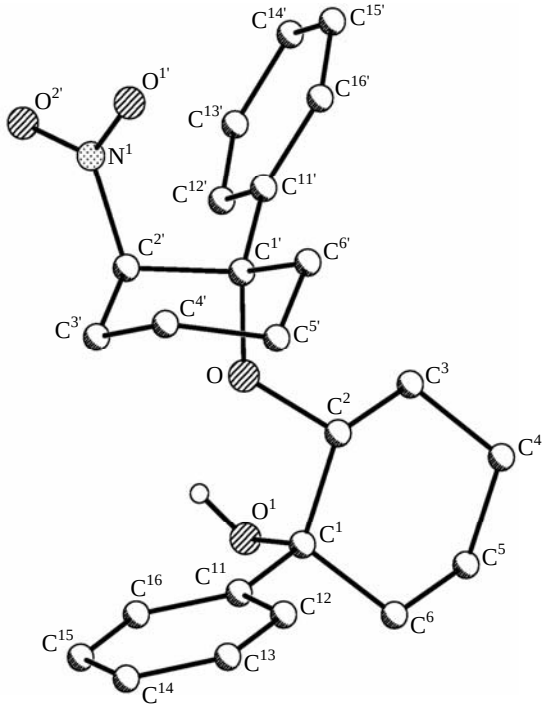
methane in nitromethane to afford vicinal nitro alcohols **VIIa** and **VIIb**, as well as nitroalkenes **VIIIa** and **VIIIb**, respectively. In addition, a small amount of ether **IX** was isolated in the reaction of 1-phenylcyclohexene with tetranitromethane (Scheme 4).

According to the NMR data, compounds **VIIa** and **VIIb** were formed as a single diastereoisomer, assumingly with *trans* orientation of the hydroxy and nitro groups. The ^{1}H and ^{13}C NMR spectra of *trans*-nitro alcohol synthesized from 2-nitrocyclohexanone and PhMgBr [13] fully coincided with those of compound **VIIb**. Nitro alcohols **V**, **VIIa**, and **VIIb** were identified via conversion into stable tetrahydropyranyl derivatives **Xa**–**Xc**. The structure of cyclohexanol **IX** was unambiguously proved by the X-ray diffraction data (see figure). The NO_2 and RO groups, as well as RO and OH, are arranged *trans* with respect to each other. The mechanism of formation of compound **IX** still remains unclear, and no published analogies of such transformation were found.

Analysis of the structure of products **V** and **VII**–**IX** suggests that the first reaction stage involves recombination of nitryl radical with the radical cation derived from olefin to give nitro-substituted carbocation **B**. Obviously, vicinal nitro alcohols **V**, **VIIa**, and **VIIb** are formed via predominant O-alkylation of cation **B** with trinitromethyl anion, followed by solvolysis of unstable nitronate **E** (Scheme 2). The formation of appreciable amounts of nitro alkenes **F** from phenylcycloalkenes is explained by preferential stabilization of nitro carbocation **B** via elimination of proton. An analogous behavior was described previously for some olefins having bulky substituents [14]. In the reaction of tetranitromethane with sterically unhindered alkenes, [2+3]-cycloaddition of nitronate **E** to the substrate leads to isoxazolidine derivatives **H** [1–8].

According to the data of [4], the major product of the reaction of α-methylstyrene with tetranitromethane in nitromethane is (1-methyl-2,2,2-trinitro-1-nitromethylethyl)benzene. We made an attempt to reproduce this procedure and failed to isolate the corresponding product as individual substance. The reaction resulted in formation of a complex mixture of products which were difficult to separate, and isoxazolidine derivative was detected among the products.

Thus the different behavior of the examined alkenes in reaction with tetranitromethane in nitromethane originates from the existence of two possible paths of recombination of intermediate species (olefin radical cation, trinitromethyl anion, and nitryl radical), i.e., collapse of ion or radical pair. The olefins react with tetranitromethane differently in petroleum ether and nitromethane due to competing nucleophilic attack by ambident trinitromethyl anion on intermediate nitro-substituted carbocation (C- and O-alkylation), leading



Structure of the molecule of 2-(2-nitro-1-phenylcyclohexyloxy)-1-phenylcyclohexanol (**IX**) according to the X-ray diffraction data.

to tetranitropropanes and vicinal nitro alcohols, respectively. Presumably, nitromethane (as more polar solvent) stabilizes more polar form of trinitromethyl anion, favoring addition of the latter at the oxygen atom.

To conclude, it should be noted that the examined reactions of tetranitromethane with olefins in nitromethane open new prospects in the synthesis of such difficultly accessible but promising compounds as alkyltrinitromethanes and vicinal nitro alcohols in which the hydroxy group is attached to a tertiary carbon atom.

EXPERIMENTAL

The ^{1}H and ^{13}C NMR spectra were recorded on Varian VXR-400 (400 and 100 MHz, respectively) and Bruker DPX-300 spectrometers (300 and 75 MHz, respectively); chloroform-*d* was used as solvent and internal reference (δ 7.24, δ_C 77.10 ppm). The mass spectra (electron impact, 70 eV) were obtained on a Varian MAT-311A instrument.

Tetranitromethane was prepared from acetic anhydride and concentrated nitric acid (d_4^{20} = 1.5 g/cm^3) according to the procedure described in [15]. 2-Cyclopropylpropene [16] and 1,1-dicyclopropylethene [17] were synthesized by known methods. 1-Phenylcyclohexene, 1-phenylcyclopentene, and α-methylstyrene were commercial products.

X-Ray analysis of a single crystal of compound **IX** was performed on a Siemens P3/PC diffractometer [λ(MoK_α) = 0.71073 Å, θ/2θ-scanning, 2θ < 56°]. Monoclinic crystals with the following unit cell parameters: a = 10.907(2), b = 10.736(2), c = 17.937(4) Å; β = 91.36(3)°; Z = 4; V = 2099.9(7) Å^3; space group $P2_1/n$; d_{calc} = 1.251 g/cm^3; μ = 0.85 cm^{-1}; F(000) = 848; M 395.48. Intensities of 5358 reflections were measured at room temperature; 5101 independent reflections (R_{int} = 0.0204) were used in subsequent refinement. The structure was solved by the direct method and was refined with respect to F^2 by the least-squares procedure in full-matrix isotropic–anisotropic approximation. Hydrogen atoms were localized from the Fourier difference series of electron density, and their positions were refined in isotropic approximation. The final divergence factors were R_1 = 0.0441 [from 3916 reflections with $I > 2\sigma(I)$] and wR_2 = 0.1312 (all reflections); GOF = 1.059. All calculations were performed using SHELXTL V5.10 software package [18]. Additional information is available from the authors.

Reaction of olefins with tetranitromethane (*general procedure*). A solution of 2.5 mol of tetranitromethane in 2 ml of nitromethane was cooled to 0°C, and a solution of 2.5 mmol of the corresponding olefin in 2 ml of nitromethane was added dropwise under stirring. The mixture was kept for 3 days at room temperature, the solvent was distilled off, and the residue was subjected to column chromatography.

2-Cyclopropyl-2-methyl-1,1,1-trinitropropane (III). Yield 46%, R_f 0.6 ($CHCl_3$). ^{1}H NMR spectrum, δ, ppm: 0.47 m (2H, cyclorpopane), 0.60 m (2H, cyclopropane), 1.35 s (6H, CH_3), 1.64 m (1H, CH, cyclopropane). ^{13}C NMR spectrum, δ_C, ppm ($^1J_{CH}$, Hz): 2.20 (CH_2, cyclopropane, J = 163), 17.51 (CH, cyclopropane, J = 163), 22.22 (CH_3, J = 130), 44.57 (C), 136.60 [$C(NO_2)_3$]. Mass spectrum, m/z, (I_{rel}, %): 233 (1) [M]$^+$, 203 (35) [M – NO], 121 (51), 109 (35), 95 (34), 93 (37), 83 (100) [M – $C(NO_2)_3$], 69 (45), 55 (71). Found, %: C 36.37; H 4.93. $C_7H_{11}N_3O_6$. Calculated, %: C 36.05; H 4.72.

1,1-Dicyclopropyl-3-nitro-2-propanol (V). Yield 45%, R_f 0.4 ($CHCl_3$). ^{1}H NMR spectrum, δ, ppm: 0.33–0.57 m (8H, CH_2, cyclopropane), 0.88 m (2H, CH, cyclopropane), 4.54 s (2H, CH_2). ^{13}C NMR spectrum, δ_C, ppm ($^1J_{CH}$, Hz): –0.75 (CH_2, cyclopropane, J = 163), 0.37 (CH_2, cyclopropane, J = 162), 17.14 (CH, cyclopropane, J = 156), 70.20 (C), 85.09 (CH_2, J = 128). Mass spectrum, m/z (I_{rel}, %): 154 (38) [M – OH], 111 (100) [M – CH_2NO_2], 107 (31), 93 (10), 79 (12). Found, %: C 56.51; H 8.02; N 7.15. $C_8H_{13}NO_3$. Calculated, %: C 56.13; H 7.65; N 8.18.

2-Nitro-1-phenylcyclopentanol (VIIa). Yield 40%, R_f 0.3 ($CHCl_3$). ^{1}H NMR spectrum, δ, ppm (J, Hz): 2.01–2.86 m (6H, CH_2), 5.06 d.d.d (1H, $CHNO_2$, ^3J = 3.5, 7.7, ^4J = 1.1), 7.29–7.50 m (5H, Ph). ^{13}C NMR spectrum, δ_C, ppm ($^1J_{CH}$, Hz): 21.95 (CH_2, J = 132), 30.09 (CH_2, J = 133), 36.67 (CH_2, J = 132), 88.63 (C), 96.07 ($CHNO_2$, J = 156), 126.20 (2CH, Ph), 128.52 (2CH, Ph), 128.72 (CH, Ph), 139.86 (C, Ph). Mass spectrum, m/z (I_{rel}, %): 261 (21) [M – NO_2], 143 (2) [M – NO_2 – H_2O], 105 (100), 77 (38), 51 (15). Found, %: C 64.59; H 6.67. $C_{11}H_{13}NO_3$. Calculated, %: C 63.76; H 6.32.

2-Nitro-1-phenylcyclohexanol (VIIb) [13]**.** Yield 41%, mp 93–94°C; published data [16]: mp 93°C; R_f 0.3 ($CHCl_3$). ^{1}H NMR spectrum, δ, ppm (J, Hz): 1.55–3.10 m (8H, CH_2), 4.85 t (1H, $CHNO_2$, J = 5.0), 7.26–7.52 m (5H, Ph). ^{13}C NMR spectrum, δ_C, ppm: 20.56 (CH_2), 20.60 (CH_2), 27.45 (CH_2), 34.09 (CH_2), 73.08 (C), 90.43 ($CHNO_2$), 125.85 (2CH, Ph), 128.13

(CH, Ph), 128.44 (2CH, Ph), 143.21 (C, Ph). Mass spectrum, *m*/*z* (I_{rel}, %): 221 (1) $[M]^+$, 204 (2) [*M* – OH], 186 (9), 175 (8) [*M* – NO_2], 174 (20) [*M* – HNO_2], 173 (25), 145 (15), 133 (21), 105 (100), 98 (25), 91 (46), 83 (24), 77 (51), 70 (30), 55 (24). Found, %: C 65.29; H 7.67; N 6.16. $C_{12}H_{15}NO_3$. Calculated, %: C 65.14; H 6.83; N 6.33.

(5-Nitro-1-cyclopentenyl)benzene (VIIIa) [19]**.** Yield 26%, R_f 0.7 ($CHCl_3$). ^{1}H NMR spectrum, δ, ppm: 2.01–3.05 m (4H, CH_2), 5.96 m (1H, CH), 6.70 m (1H, CH), 7.29–7.50 m (5H, Ph). ^{13}C NMR spectrum, δ_C, ppm: 31.45 (CH_2), 32.15 (CH_2), 92.48 ($CHNO_2$), 125.85 (2CH, Ph), 128.39 (CH, Ph), 129.31 (2CH, Ph), 133.13 (C), 136.72 (CH=), 138.72 (C).

(6-Nitro-1-cyclohexenyl)benzene (VIIIb) [20]**.** Yield 33%, mp 36°C; published data [18]: mp 36–37°C; R_f 0.8 ($CHCl_3$). ^{1}H NMR spectrum, δ, ppm (*J*, Hz): 1.68–2.50 m (6H, CH_2), 5.56 m (1H, $CHNO_2$), 6.42 d.d (1H, CH=, ^3J = 3.6, 4.4), 7.31 m (5H, Ph). ^{13}C NMR spectrum, δ_C, ppm: 17.51 (CH_2), 25.63 (CH_2), 29.19 (CH_2), 83.24 ($CHNO_2$), 125.70 (2CH, Ph), 128.20 (CH, Ph), 128.88 (2CH, Ph), 131.49 (C), 134.08 (CH=), 139.02 (C).

(1*RS*,2*RS*,1'*SR*,2'*RS*)-2-(2-Nitro-1-phenylcyclohexyloxy)-1-phenylcyclohexanol (IX). Yield 4%, mp 194–195°C. ^{1}H NMR spectrum, δ, ppm (*J*, Hz): 1.00–2.00 m (15H, cyclohexane), 2.76 d.d.d (1H, cyclohexane, *J* = 4.7, 14.5, 14.1), 3.89 m (1H, CHO), 4.70 m (1H, $CHNO_2$), 7.27–7.52 m (10H, Ph), 10.13 (1H, OH).

Tetrahydropyranyl derivatives of alcohols V, VIIa, and VIIb (*general procedure***).** Alcohol **V**, **VIIa**, or **VIIb**, 2 mmol, was added to a solution of 3 mmol of dihydropyran and 0.01 mmol of pyridinium *p*-toluenesulfonate in 7 ml of methylene chloride. The mixture was stirred for 4 h and left overnight. It was then washed with a saturated aqueous solution of sodium chloride (2×3 ml) and evaporated, and the product was isolated from the residue by column chromatography.

2-(1,1-Dicyclopropyl-2-nitroethoxy)tetrahydro-2*H*-pyran (Xa). Yield 76%, R_f 0.7 (petroleum ether–ethyl acetate, 4:1). ^{1}H NMR spectrum, δ, ppm (*J*, Hz): 0.03–1.93 m (16H), 3.51 m (1H, CH_2O), 4.87 m (1H, CH_2O), 4.61 d (1H, CH_2NO_2, ^2J = 10.2,), 4.76 d (1H, CH_2NO_2, ^2J = 10.2), 4.99 m (1H, CH). ^{13}C NMR spectrum, δ_C, ppm: –0.99 (CH_2, cyclopropyl), 1.12 (CH_2, cyclopropyl), 1.45 (CH_2, cyclopropyl), 2.56 (CH_2, cyclopropyl), 13.66 (CH, cyclopropyl), 14.55 (CH, cyclopropyl), 19.75 (CH_2), 25.58 (CH_2), 30.80 (CH_2), 63.01 (CH_2O), 84.08 (CH_2NO_2), 90.01 (C), 94.71 (CH). Mass spectrum, *m*/*z* (I_{rel}, %): 195 (1) [*M* – CH_2NO_2], 154 (18) [*M* – $C_5H_9O_2$], 111 (16), 85 (100) (C_5H_9O), 41 (15) (cyclopropyl).

2-(2-Nitro-1-phenylcyclopentyloxy)tetrahydro-2*H*-pyran (Xb). Yield 73%, R_f 0.5 (petroleum ether–ethyl acetate, 4:1). ^{1}H NMR spectrum, δ, ppm (a mixture of two diastereoisomers, A and B, at a ratio of 8:3): 1.32–2.53 m (12H + 12H, A, B), 3.19 m (1H, CH_2O, B), 3.48 m (1H, CH_2O, A), 3.69 m (1H, CH_2O, B), 3.90 m (1H, CH_2O, A), 4.35 m (1H, $CHNO_2$, A), 4.48 m (1H, $CHNO_2$, B), 5.15 m (1H, CH, A), 5.56 m (1H, CH, B), 7.26–7.51 m (5H + 5H, Ph, A, B). ^{13}C NMR spectrum, δ_C, ppm (A+B): 20.09 (CH_2, A), 20.20 (CH_2, B), 22.03 (CH_2, B), 22.23 (CH_2, A), 25.17 (CH_2, B), 25.27 (CH_2, A), 25.55 (CH_2, B), 30.49 (CH_2, A), 30.79 (CH_2, B), 30.85 (CH_2, A), 31.85 (CH_2, A), 32.10 (CH_2, B), 63.48 (CH_2O, B), 63.84 (CH_2O, A), 91.54 (C, B), 92.69 (C, A), 94.15 ($CHNO_2$, A), 94.90 ($CHNO_2$, B), 95.90 (CH, A), 96.36 (CH, B), 127.98 (2CH, Ph), 128.06 (2CH, Ph), 128.12 (2CH, Ph), 128.39 (2CH, Ph), 128.75 (CH, Ph), 128.79 (CH, Ph), 135.06 (C, Ph), 137.12 (C, Ph). Found, %: C 66.01; H 7.79; N 4.81. $C_{16}H_{21}NO_4$. Calculated, %: C 65.98; H 7.27; N 4.81.

2-(2-Nitro-1-phenylcyclohexyloxy)tetrahydro-2*H*-pyran (Xc). Yield 86%, R_f 0.6 (petroleum ether–ethyl acetate, 4:1). ^{1}H NMR spectrum, δ, ppm (a mixture of two diastereoisomers, A and B, at a ratio of 3:1): 1.30–2.90 m (14H + 14H, A, B), 3.41 m (1H, CH_2O, A), 3.75 m (1H, CH_2O, B), 3.90 m (1H, CH_2O, B), 3.98 m (1H, CH_2O, A), 4.22 m (1H, $CHNO_2$, A), 4.33 m (1H, $CHNO_2$, B), 5.04 m (1H, CH, A), 5.32 m (1H, CH, B), 7.23–7.55 m (5H + 5H, Ph, A, B). ^{13}C NMR spectrum, δ_C, ppm (A+B): 19.32 (CH_2, A), 19.43 (CH_2, B), 20.40 (CH_2, A), 20.46 (CH_2, A), 20.56 (CH_2, B), 20.77 (CH_2, B), 25.24 (CH_2, B), 25.35 (CH_2, A), 27.40 (CH_2, A), 27.58 (CH_2, A), 27.78 (CH_2, A), 28.61 (CH_2, B), 32.05 (CH_2, A), 32.37 (CH_2, B), 63.81 (CH_2O, B), 64.25 (CH_2O, A), 78.44 (C, A, B), 88.86 ($CHNO_2$, B), 90.21 ($CHNO_2$, A), 94.82 (CH, B), 95.55 (CH, A), 127.22 (2CH, Ph), 127.33 (2CH, Ph), 128.15 (2CH, Ph, A, B), 128.54 (2CH, Ph), 128.63 (2CH, Ph), 140.60 (2C, Ph). Found, %: C 67.31; H 7.87; N 4.68. $C_{17}H_{23}NO_4$. Calculated, %: C 66.86; H 7.59; N 4.59.

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