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Spatial Arrangement of Norbornanedicarboxylic Acids N-Arylimides

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Abstract—The effect of substituents on the spatial arrangement of norbornane compounds was investigated. Norbornanedicarboxylic acids N-arylimides with OCH_3, Cl, NO_2 groups in the *ortho*-position of the aromatic ring form as conformational isomers with hindered rotation. The specific spatial isomerism is confirmed by the quantum-chemical calculations.

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The oxidation of bicyclo[2.2.2]oct-2-ene with performic acid is known to afford bicyclo[2.2.2]-octane-*trans*-2,3-diol, whereas the oxidation of bicyclo[2.2.1]-hept-2-ene under the same conditions is accompanied by Wagner–Meerwein rearrangement and 1,3-hydride shift [1–3].

The introduction into the norbornene ring of electron-acceptor substituents act as a stabilizing factor fixing the boat-like conformation of the cyclohexene fragment and thus prevents the hydride shifts and rearrangements [4, 5]. In the oxidation of norbornenes containing reactive groups in the *endo*-position forms as a rule a new lactone ring, whereas the presence of these groups in the *exo*-position of the norbornene results in normal oxidation products [6, 7]. This rule is conserved in the oxidation of norbornene dicarboxylic acids, their anhydrides **I, II,** and the nitrogen-containing derivatives with the *endo*- and *exo*-configuration [8–10]. The epoxidation of *exo*-compounds proceeds stereoselectively with the formation of an oxirane ring of *exo*-configuration, a lactone is formed from the corresponding *endo*-derivatives [11–13] (Scheme 1).

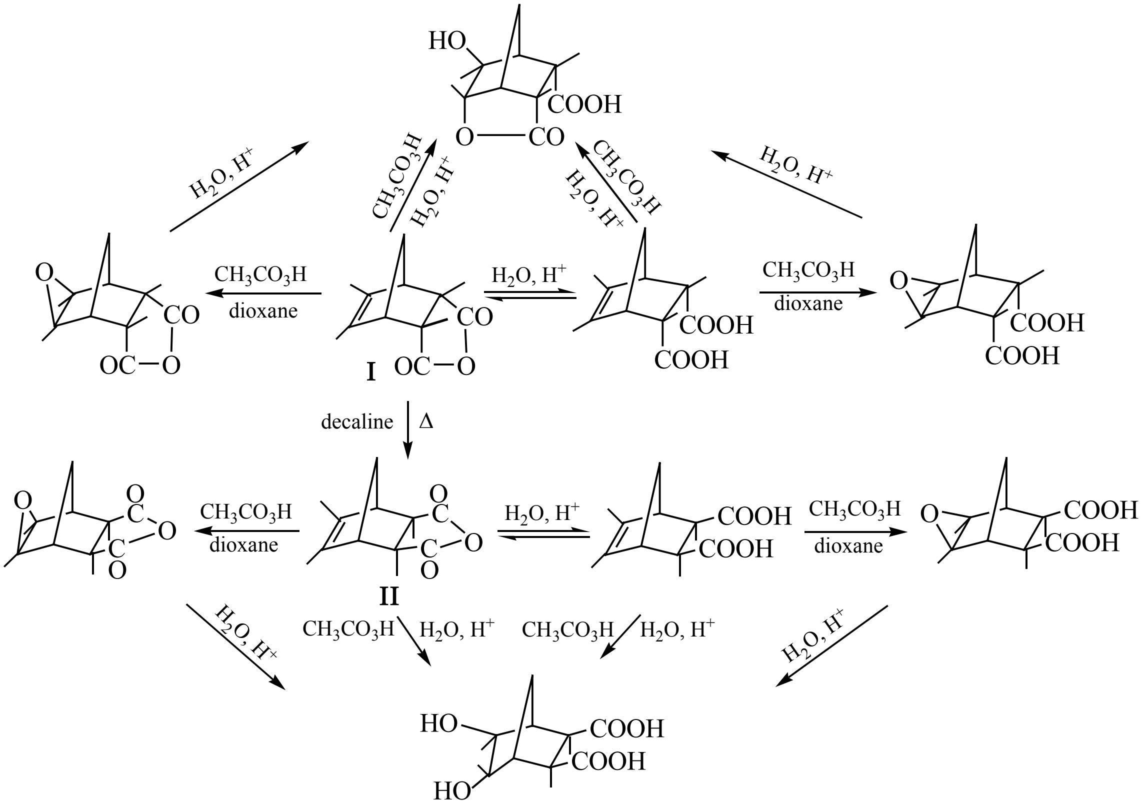
Our investigation of the oxidation of norbornenedicarboxylic acids N-arylimides showed that independent of the *endo*- or *exo*-configuration of the imide ring and also of the electronic nature of the substituents in the *ortho*-, *meta*, or *para*-positions in the N-arylimide fragment only stereoisomeric epoxy or dihydroxy derivatives formed, and no lactonization and Wagner–Meerwein rearrangement was observed [14, 15]. The strong electron-acceptor imide moiety decreases the electron density of the π-bond of norbornene and diminishes the ionization of the C–O bond which is the first stage in the Wagner–Meerwein rearrangement, and the low strain of the five-membered imide ring and its chemical inertness prevents the lactonization (Scheme 2).

The role of electronic and sterical effects and the positions (*ortho-, meta-, para-*) of substituents in the formation of the conformational structure of norbornane N-arylimides remains unclear.

Our quantum-chemical calculations, spectral analysis, and the consideration of model structures of N-arylimides **III–LXVI** of bicyclo[2.2.1]hept-5-ene-*endo*- and -*exo*-2,3-dicarboxylic acid showed that when in the *ortho*-position of the aromatic ring were located OCH_3, Cl, NO_2 groups the large bulk of the substituents hampered the free rotation, and as a result a stable conformational isomer of N-arylimide formed where the benzene ring at the nitrogen atom was perpendicular to the plane of the five-membered heterocycle, and the N–C bond suffered elongation compared to its average length [16].

This fact is confirmed by the presence of two

Scheme 1.



unequal signals from the OCH_3 group in the ^1H NMR spectra of compounds **VIIa, VIIb, XXIIIa, XXIIIb, XXXIXa, XXXIXb** etc. For instance, the protons of methoxy groups in the spectra of N-arylimides of 5,6-dihydroxybicyclo[2.2.1]hept-5-ene-2,3-dicarboxylic acid **XXXIXa, XXXIXb** give rise to a double singlet at 3.75 and 3.80 ppm indicating the different poitions of the methoxy group in the ring. Double signals from OCH_3 group are lacking in the spectra of the corresponding *meta-* and *para-*isomers **VIII, IX, XXIV, XXV, XL, XLI, LVI, LVII**; in the ^1H NMR spectra of N-arylimides **IV–VI, XX–XXII, XXXVI–XXXVIII, LII–LIV** no two signals from the methyl groups are observed even in the *ortho*-isomers.

The consideration of the models of possible isomeric forms of the molecule shows that in the *syn*-isomer due to the steric interaction exists prevailingly only **B** conformation, whereas the *anti*-isomer can exist in both **C** and **D** conformations.

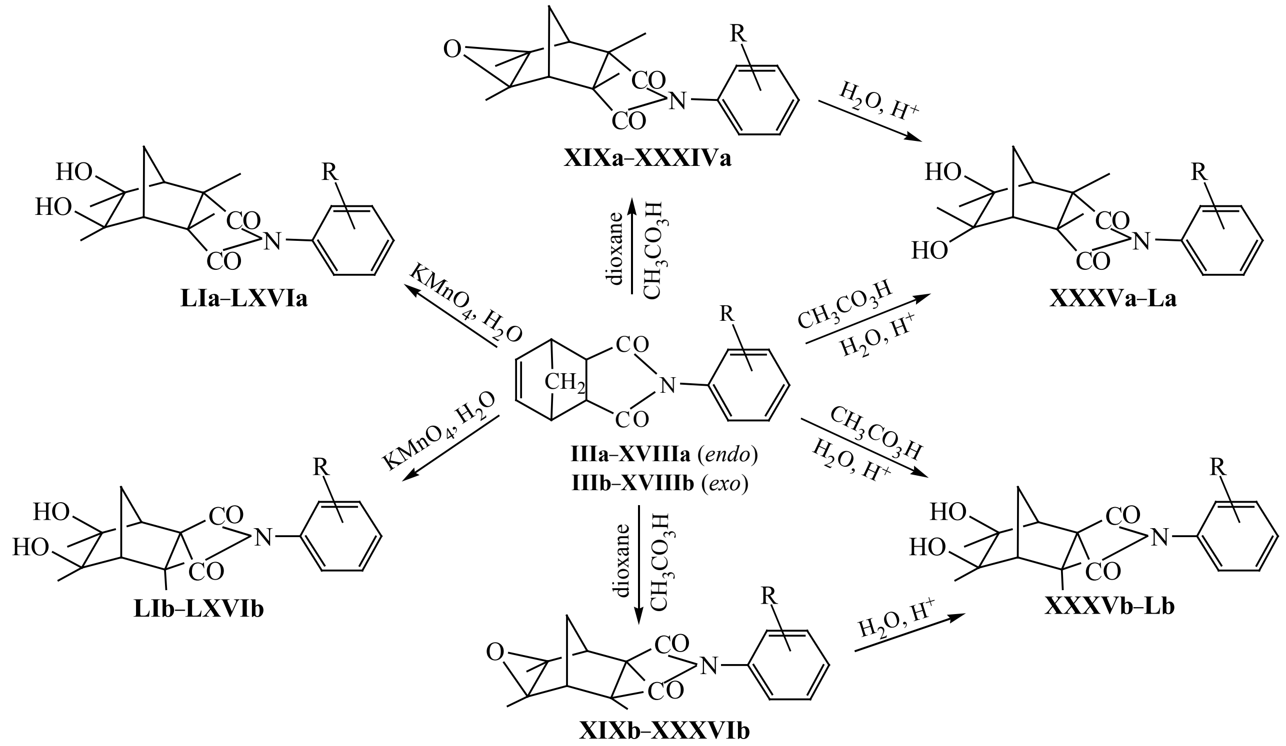
In the ^1H NMR spectra of compounds **XXXIXa, XXXIXb** the less strong singlet of the methoxy group (δ 3.75 ppm) corresponds to form **B** where the OCH_3 group is relatively distant from the magnetically anisotropic C=O group, whereas in the interconvertible conformations **C** and **D** the methoxy group protons are partially screened by the carbonyl groups of the imide ring, and therefore the more intense signal corresponding to forms **C** and **D** is slightly shifted upfield (δ 3.80 ppm).

In keeping with the integral intensity of the signals the ratio of *syn*-form **B** to *anti*-forms **C** and **D** equals 25 to 75%, but the separation of stereoisomers **XXXIXa, XXXIXb** is materially impossible. (Scheme 3)

The experimental and calculation data show that the radius of the OCH_3 group (0.145 nm) is insufficient for the complete overlapping with the *ortho*-substituent therefore compounds **VIIa, VIIb, XXIIIa, XXIIIb, XXXIXa, XXXIXb**, **LVa, LVb** are conformationally labile.

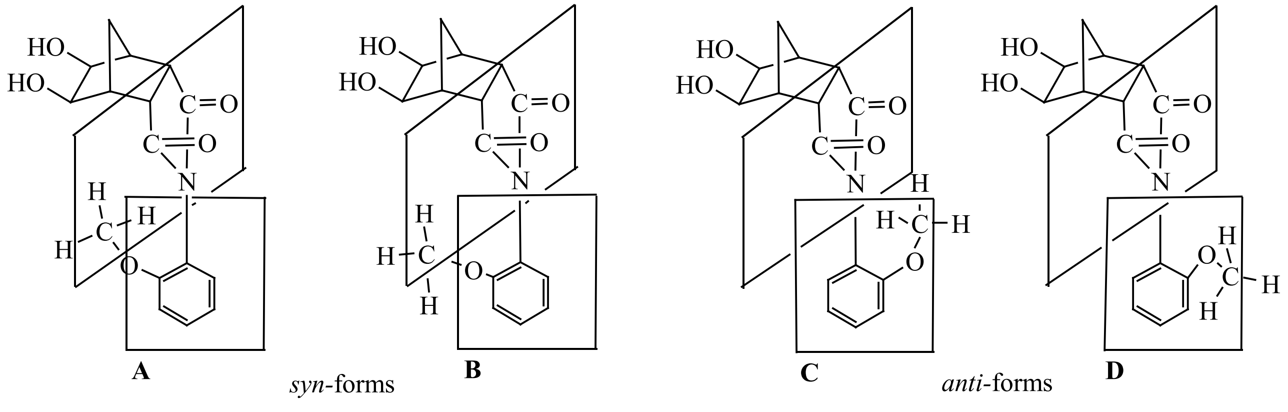
In order to confirm the experimentally found fact of the conformational isomerism we attempted to find a relation between the structure and quantum-chemical description of N-arylimides of bicycloheptene-*endo-* and *exo-*dicarboxylic acids containing in the aryl ring substituents of various electronic and steric character **III–XVIII**. The analysis of the results of the quantum-chemical calculations shows that although the other parameters also vary consistently, in this case the most appropriate parameter

Scheme 2.



R = H (**III, XIX, XXXV, LI**), *o*-CH_3 (**IV, XX, XXVI, LII**), *m*-CH_3 (**V, XXI, XXXVII, LIII**), *p*-CH_3 (**VI, XXII, XXXVIII, LIV**), *o*-OCH_3 (**VII, XXIII, XXXIX, LV**), *m*-OCH_3 (**VIII, XXIV, XL, LVI**), *p*-OCH_3 (**IX, XXV, XLI, LVII**), *o*-Cl (**X, XXVI, XLII, LVIII**), *m*-Cl (**XI, XXVII, XLIII, LIX**), *p*-Cl (**XII, XXVIII, XLIV, LX**), *o*-PO_2 (**XIII, XXIX, XLV, LXI**), *m*-PO_2 (**XIV, XXX, XLVI, LXII**), *p*-PO_2 (**XV, XXXI, XLVII, LXIII**), *o*-COOH (**XVI, XXXII, XLVIII, LXIV**), *m*-COOH (**XVII, XXXIII, XLIX, LXV**), *p*-COOH (**XVIII, XXXIV, L, LXVI**).

Scheme 3.



for establishing the relation between the structure and property is the heat of formation (Q_{form}). The conformational isomers are stable at room temperature when the value of the free activation enthalpy ΔG^* is no less than 65–85 kJ mol^{-1} [17].

The heat of formation is known [18] to include the sum of energy of all chemical bonds and separate structural fragments of the molecule. Therefore it is expectable that the changes in the composition and structure would affect the Q_{form} values. This conclusion is confirmed by the calculations. Q_{form} of anhydrides **I,II** (60.61 and 61.91 kcal mol^{-1}) is lower than Q_{form} of *endo*- and *exo*-N-phenylimides **IIIa, IIIb** (72.72 and 73.39 kcal mol^{-1}, respectively). In going to N-arylimides containing electron-donor and electron-acceptor substituents Q_{form} regularly changes: in the N-arylimides with the electron-donor substituents in the benzene ring (**IVa, IVb, VIIa, VIIb, Xa, Xb**) it decreases, and in the N-arylimides with the electron-acceptor substituents (**XIIIa, XIIIb**) it increases. The value Q_{form} of the *ortho*-isomers as a rule is twice as large as that of *meta*- and *para*-isomers of the same molecules notwithstanding the electronic character of the substituent. The reason of this large difference between the isomeric N-arylimides is the large barrier to the rotation around the N–C bond in the *ortho*-isomers arising from the steric effect and increased or decreased by additional factors.

The results of the quantum-chemical calculations permit a conclusion that in all *ortho*-substituted *endo*- and *exo*-isomers of N-arylimide **IVa, IVb, VIIa, VIIb, Xa, Xb**, **XIIIa, XIIIb** the hindered rotation exists; however if in N-*o*-methoxyphenylimides **VIIa, VIIb** a fast racemization occurs (Q_{form} 89.54 and 88.78 kcal mol^{-1} respectively), the data for *o*-chlorophenylimides **Xa, Xb** (Q_{form} 137.95 and 137.48 kcal mol^{-1}) and for *o*-nitrophenylimides **XIIIa, XIIIb** (Q_{form} 148.86 and 150.49 kcal mol^{-1}) allow a statement that in these events arise stable spatial conformers. The consideration of the models of the structure of norbornenedicarboxylic acids N-arylimides shows that besides the dimensions of these groups a significant role in the formation of the spatial arrangement plays the unshared electron pair which increases the stability of the spatial isomer through the conjugation. (Complete data of quantum-chemical calculations of anhydrides and N-arylimides of bicyclo[2.2.1]hept-5-ene- *endo*- and *exo*-2,3-dicarboxylic acid are available from the authors by e-mail: salahov_mustafa@mail.ru).

It is possible to conclude from the theoretical considerations that in formation of one of individual *syn*- or *anti*-isomers the governing importance belongs to the intermediate complex of one or another configuration. In the first stage in the forming amidoacid the nitrogen atom has a pyramidal configuration, and the hydrogen atom of NH group is involved in an intramolecular hydrogen bond with the oxygen atom of the carbonyl group. This arrangement determines the configuration of the obtained intermediate product. The second stage of the reaction consists in water elimination and in the formation of flat imide ring with the *syn*- or *anti*-orientation of the substituent, and the latter prevails.

EXPERIMENTAL

NMR spectra were registered on a spectrometer Tesla BS-487 at operating frequency 80 MHz in $CDCl_3$, internal reference HMDS [19]. IR spectra were recorded on a spectrophotometer UR-20 in the region 400–3800 cm^{-1} from mulls in mineral oil. UV spectra were measured on a spectrophotometer Specord M-40 [20].

The purity of compounds was checked by TLC, sorbent silica L 5/40μ, development under UV irradiation [21].

Quantum-chemical calculations were performed along Schroedinger equation, for multielectron systems with the use of the method of Hartree–Fock–Roothaan [22, 23]. Multicenter integrals were calculated using semiempirical methods of Pariser–Parr–Pople [24]. In the calculations the information was used on the system under consideration (serial number of the atom, its position in the Periodic Table, bond lengths, bond and dihedral angles) and on the basis.

N-Arylimides of bicyclo[2.2.1]hept-5-ene-*endo*- and -*exo*-2,3-dicarboxylic acids **IIIa, IIIb–XVIIIa**, **XVIIIb** were obtained as described in [15].

***N*-Arylimides of 5,6-*exo*-epoxybicyclo[2.2.1]-heptane-*endo*- and -*exo*-2,3-dicarboxylic acid XIXa, XIXb–XXXIVa, XXXIVb.** In 15 ml of dioxane was dissolved 0.01 mol of *N*-arylimide **IIIa, IIIb–XVIIIa**, **XVIIIb**, and at 8–10°C 10 ml of peracetic acid solution in anhydrous dioxane was added at constant cooling (10°C). After complete consumtion of peracetic acid (3.5–4 h) the acetic acid was distilled off in a vacuum of a water-jet pump, and the reaction product was crystallized from anhydrous benzene.

N-Phenylimide **XIXa**. Yield 80%, mp 185–187°C (from ethanol), $\mathbf{R_f}$ 0.59. Found, %: C 75.01; H 5.00; N 4.79; $C_{15}H_{13}NO_3$. Calculated, %: C 74.90; H 5.09; N 5.49.

N-(*o*-Methoxyphenyl)imide **XXIIIa**. Yield 87%, mp 160°C (from ethanol), R_f 0.60. Found, %: C 67.17; H 5.16; N 4.80; $C_{16}H_{15}NO_4$. Calculated, %: C 67.37; H 5.26; N 4.91.

N-(*m*-Methoxyphenyl)imide **XXIVa**. Yield 90%, mp 154°C (from ethanol), R_f 0.60. Found, %: C 67.07; H 5.30; N 5.19. $C_{16}H_{15}NO_4$. Calculated, %: C 67.37; H 5.26; N 4.91.

N-(*p*-Methoxyphenyl)imide **XXVa**. Yield 87%, mp 164–165°C (from ethanol), R_f 0.54. Found, %: C 66.81; H 5.19; N 5.10. $C_{16}H_{15}NO_4$. Calculated, %: C 67.37; H 5.26; N 4.91.

N-(*o*-Chlorophenyl)imide **XXVIa**. Yield 90%, mp 190–192°C (from ethanol), R_f 0.56. Found, %: C 62.43; H 4.10; Cl 12.53; N 4.67. $C_{15}H_{12}ClNO_3$. Calculated, %: C 62.18; H 4.14; Cl 12.26; N 4.48.

N-(*m*-Chlorophenyl)imide **XXVIIa**. Yield 91%, mp 174–175°C (from ethanol), R_f 0.66. Found, %: C 62.12; H 3.76; Cl 12.12; N 4.19. $C_{15}H_{12}ClNO_3$. Calculated, %: C 62.18; H 4.14; Cl 12.26; N 4.48.

N-(*p*-Chlorophenyl)imide **XXVIIIa**. Yield 89%, mp 190–191°C (from ethanol), R_f 0.69. Found, %: C 61.12; H 4.11; Cl 12.30; N 4.89. $C_{15}H_{12}ClNO_3$. Calculated, %: C 62.18; H 4.14; Cl 12.26; N 4.48.

N-(*o*-Carboxyphenyl)imide **XXXIIa**. Yield 87%, mp 207–208°C (from ethanol), R_f 0.82. Found, %: C 63.62; H 4.17; N 5.02. $C_{16}H_{13}NO_5$. Calculated, %: C 64.21; H 4.34; N 4.68.

N-(*m*-Carboxyphenyl)imide **XXXIIIa**. Yield 90%, mp 241–242°C (from ethanol), R_f 0.78. Found, %: C 64.60; H 3.75; N 4.79. $C_{16}H_{13}NO_5$. Calculated, %: C 64.21; H 4.34; N 4.68.

N-(*p*-Carboxyphenyl)imide **XXXIVa**. Yield 86%, mp 219–220°C (from ethanol), R_f 0.80. Found, %: C 64.13; H 4.33; N 4.19. $C_{16}H_{13}NO_5$. Calculated, %: C 64.21; H 4.34; N 4.68.

***N*-Arylimides of *trans*-5,6-dihydroxybicyclo-[2.2.1] heptane-*endo*- and -*exo*-2,3-dicarboxylic acid (XXXVa, XXXVb–La, Lb.** *a*. In 30 ml of acetic acid was dissolved 0.01 mol of N-arylimide **IIIa, IIIb–XVIIIa, XVIIIb**, 1–2 drops of concn. H_2SO_4 was added, and at constant stirring a mixture of 10 ml of 30% H_2O_2 in 10 ml of acetic acid was added to the above solution. The mixture was heated for 2 h at 70°C. Then the reaction mixture was diluted with 15 ml of water, and the heating was continued for 1 h. On completion of the reaction the separated crystals were filtered off, washed with water, and recrystallized from ethanol.

N-Phenylimide (**XXXVa**). Yield 91%, mp 199–200°C (from ethanol), R_f 0.69. ^{1}H NMR spectrum, δ, ppm: 1.15 m (H^{7an}), 1.75 m (H^{7s}), 1.95 s ($H^{1,4}$), 3.15 br.m ($H^{2,3}$), 3.30 br.m ($H^{5,6}$), 3.35 s (OH), 7.30–7.60 m (H_{arom}). Found, %: C 66.31; H 5.05; N 6.05; $C_{15}H_{15}NO_4$. Calculated, %: C 66.42; H 4.79; N 5.17.

N-(*o*-Methoxyphenyl)imide **XXXIXa**. Yield 83%, mp 169–170°C (from ethanol), R_f 0.79. ^{1}H NMR spectrum, δ, ppm: 1.10 m (H^{7an}), 1.65 m (H^{7s}), 2.10 s ($H^{1,4}$), 3.15 br.m ($H^{2,3}$), 3.32 br.m ($H^{5,6}$), 3.36 s (OH), 7.10–7.70 m ($MeOC_6H_4$), 3.75–3.80 s (3H, MeO). Found, %: C 64.22; H 5.91; N 4.83. $C_{16}H_{17}NO_5$. Calculated, %: C 63.66; H 5.61; N 4.60.

N-(*m*-Methoxyphenyl)imide **XLa**. Yield 85%, mp 163–164°C (from ethanol), R_f 0.72. ^{1}H NMR spectrum, δ, ppm: 1.12 m (H^{7an}), 1.73 m (H^{7s}), 2.00 s ($H^{1,4}$), 3.13 br.m ($H^{2,3}$), 3.27 br.m ($H^{5,6}$), 3.30 s (OH), 7.00–7.50 m ($MeOC_6H_4$), 3.70–3.77 s (3H, MeO). Found, %: C 64.22; H 5.91; N 4.83. $C_{16}H_{17}NO_5$. Calculated, %: C 63.66; H 5.61; N 4.60.

N-(*p*-Methoxyphenyl)imide **XLIa**. Yield 91%, mp 184–185°C (from ethanol), R_f 0.60. ^{1}H NMR spectrum, δ, ppm: 1.10 m (H^{7an}), 1.70 m (H^{7s}), 2.05 s ($H^{1,4}$), 3.15 br.m ($H^{2,3}$), 3.25 br.m ($H^{5,6}$), 3.34 s (OH), 7.10–7.55 m ($MeOC_6H_4$), 3.85 s (3H, MeO). Found, %: C 64.22; H 5.91; N 4.83. $C_{16}H_{17}NO_5$. Calculated, %: C 63.66; H 5.61; N 4.60.

N-(*o*-Chlorophenyl)imide **XLIIa**. Yield 93%, mp 201–202°C (from ethanol), R_f 0.75. Found, %: C 59.01; H 4.61; Cl 12.01; N 4.72. $C_{15}H_{14}Cl\ NO_4$. Calculated, %: C 58.53; H 4.55; Cl 11.54; N 4.55.

N-(*m*-Chlorophenyl)imide **XLIIIa**. Yield 95%, mp 227–228°C (from ethanol), R_f 0.76. Found, %: C 59.42; H 4.47; Cl 11.17; N 4.92. $C_{15}H_{14}ClNO_4$. Calculated, %: C 58.53; H 4.55; Cl 11.54; N 4.55.

N-(*p*-Chlorophenyl)imide **XLIVa**. Yield 90%, mp 202–203°C (from ethanol), R_f 0.70. Found, %: C 59.13; H 5.11; Cl 12.03; N 4.52. $C_{15}H_{14}ClNO_4$. Calculated, %: C 58.53; H 4.55; Cl 11.54; N 4.55.

N-(*o*-Carboxyphenyl)amandд **XLVIIIa**. Yield 83%, mp 225–226°C (from ethanol), R_f 0.86. Found, %: C 60.47; H 4.57; N 4.77. $C_{16}H_{15}NO_6$. Calculated, %: C 60.56; H 4.73; N 4.41.

N-(*m*-Carboxyphenyl)imide **XLIXa**. Yield 91%, mp 285–287°C (from ethanol), R_f 0.83. Found, %: C 60.60; H 5.19; N 4.76. $C_{16}H_{15}NO_6$. Calculated, %: C 60.56; H 4.73; N 4.41.

N-(*p*-Carboxyphenyl)imide **La**. Yield 94%, mp 260–261°C (from ethanol), R_f 0.81. Found, %: C 60.79; H 4.82; N 4.53. $C_{16}H_{15}NO_6$. Calculated, %: C 60.56; H 4.73; N 4.41.

b. In 30 ml of 5 H_2SO_4 % 0.01 mol of *N*-arylimides of 5,6-*exo*-epoxybicyclo-[2.2.1]heptane-*endo*- (**XIXa–XXXIVa**) or -*exo*-(**XIXb–XXXIVb**)-2,3-dicarboxylic acids was heated for 4 h at 60°C. The reaction product was filtered off, washed with dilstilled water, and dried. Yield of *trans*-imidodiols **XXXVa, XXXVb–La, Lb** 78–90%.

***N*-Arylimides of *exo-cis*-5,6-dihydroxybicyclo-[2.2.1]heptane-*endo*-2,3-dicarboxylic acid (LIa–LXVIa).** To a solution of 0.01 mol of *N*-arylimide **IIIa–XVIIIa** in 100 ml of acetone at room temperature while vigorous stirring was added dropwise a solution of 1.6 g of potassium permanganate in 250 ml of distilled water. After the complete consumption of the potassium permanganate (disappearance of pink color) a dark brown precipitate separated that after treatment with dilute hydrochloric acid turned colorless. The reaction mixture was left standing for 12 h. The separated crystals were filtered off and recrystallized from methanol.

N-Phenylimide **LIa**. Yield 70%, mp 207°C (from ethanol), R_f 0.71. Found, %: C 66.0; H 5.42; N 5.07. $C_{15}H_{15}NO_4$. Calculated, %: C 65.93; H 5.49; N 5.13.

N-(*o*-Methoxyphenyl)imide **LVa**. Yield 60%, mp 239°C (from ethanol), R_f 0.73. Found, %: C 63.14; H 5.25; N 4.21. $C_{16}H_{17}NO_5$. Calculated, %: C 63.37; H 5.61; N 4.62.

N-(*m*-Methoxyphenyl)imide **LVIa**. Yield 68%, mp 252°C (from ethanol), R_f 0.70. Found, %: C 63.50; H 5.37; N 4.12. $C_{16}H_{17}NO_5$. Calculated, %: C 63.37; H 5.61; N 4.62.

N-(*p*-Methoxyphenyl)imide **LVIIa**. Yield 63%, mp 255°C (from ethanol), R_f 0.68. Found, %: C 63.16; H 5.39; N 4.20. $C_{16}H_{17}NO_5$. Calculated, %: C 63.37; H 5.61; N 4.62.

N-(*o*-Chlorophenyl)imide **LVIIIa**. Yield 75%, mp 196°C (from ethanol), R_f 0.75. Found, %: C 58.40; H 4.25; Cl 11.14; N 4.31. $C_{15}H_{14}ClNO_4$. Calculated, %: C 58.53; H 4.55; Cl 11.54; N 4.55.

N-(*m*-Chlorophenyl)imide **LIXa**. Yield 75%, mp 237°C (from ethanol), R_f 0.71. Found, %: C 58.13; H 4.09; Cl 11.06; N 4.15. $C_{15}H_{14}ClNO_4$. Calculated, %: C 58.53; H 4.55; Cl 11.54; N 4.55.

N-(*p*-Chlorophenyl)imide **LXa**. Yield 73%, mp 218°C (from ethanol), R_f 0.74. Found, %: C 58.11; H 4.15; Cl 11.12; N 4.18. $C_{15}H_{14}ClNO_4$. Calculated, %: C 58.53; H 4.55; Cl 11.54; N 4.55.

N-(*o*-Carboxyphenyl)imide **LXIVa**. Yield 61%, mp 236°C (from ethanol), R_f 0.68. Found, %: C 60.12; H 4.51; N 4.37. $C_{16}H_{15}NO_6$. Calculated, %: C 60.56; H 4.73; N 4.41.

N-(*m*-Carboxyphenyl)imide **LXVa**. Yield 61%, mp 255°C (from ethanol), R_f 0.70. Found, %: C 68.18; H 4.29; N 4.27. $C_{16}H_{15}NO_6$. Calculated, %: C 60.56; H 4.73; N 4.41.

N-(*p*-Carboxyphenyl)imide **LXVIa**. Yield 61%, mp 265°C (from ethanol), R_f 0.80. Found, %: C 60.10; H 4.24; N 4.18. $C_{16}H_{15}NO_6$. Calculated, %: C 60.56; H 4.73; N 4.41.

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