Chemistry of Heterocyclic Compounds, Vol. 43, No. 9, 2007

SYNTHESIS AND MOLECULAR STRUCTURE OF 4-NITRO-9-PHENYL-1H- AND 9-HYDROXY-3-OXO-9-PHENYL-2,3-DIHYDRO-9H-INDENO-[2,1-*c*]PYRIDINES AND 3,7-DIPHENYL-3a,4,5,6-TETRA-HYDROINDENO[2,1-*c*]ISOXAZOLO[5,4-*d*]PYRIDINE

S. V. Volkov[1], A. N. Levov[1], A. T. Soldatenkov[1], N. M. Kolyadina[1], O. E. Volkova[1], and V. N. Khrustalev[2]

The 4-nitro derivatives and an oxidation by-product, 9-hydroxy-2-methyl-3-oxo-9-phenyl-2,3-dihydro-9H-indeno[2,1-c]pyridine were obtained by the nitration of N-alkyl-9-phenyl-2,3-dihydro-1H-indeno-[2,1-c]pyridines with sodium nitrite in acetic acid. Their molecular structures were studied by X-ray structural analysis. The product of [2+3] cycloaddition, 5-methyl-3,7-diphenyl-3a,4,5,6-tetrahydro-indeno[2,1-c]isoxazolo[5,4-d]pyridine, was obtained by the interaction of 2-chloro-1-hydroxy-2-phenylazomethine with 2-methyl-9-phenyl-2,3-dihydro-1H-indeno[2,1-c]pyridine.

Keywords: N-alkyl-4-nitro-9-phenyl-2,3-dihydro-1H-indeno[2,1-*c*]pyridines, 9-hydroxy-2-methyl-3-oxo-9-phenyl-2,3-dihydro-9H-indeno[2,1-*c*]pyridine, 3,7-dimethyl-3a,4,5,6-tetrahydroindeno[2,1-*c*]isoxazolo-[5,4-*d*]pyridine, X-ray structural analysis.

The principle of chemical modification of known natural and synthetic biologically active substances and their precursors is, up to the present time, one of the bases in the strategy of designing new drugs [1], chemical agents for protecting and treating plants and animals [2], and also food additives [3]. As a continuation of the systematic investigation of the chemistry [4, 5] and biological activity [6, 7] of indenopyridine derivatives we have in the present work introduced the problem of studying the direction of the conversions of N-alkyl-9-phenyl-2,3-dihydro-1H-indeno[2,1-*c*]pyridines **1a,b** on interacting them with sodium nitrite in acid medium, and also with benzohydroxymoyl chloride in the presence of base. The importance of the functionalization of these close precursors of the antiallergic agent *thephorin* [1] is evident, although it is impossible to consider the target chemical modification of the latter a very simple problem, if the extremely significant chemical lability of the precursors is taken into consideration [5]. First of all, on studying the reaction between sodium nitrite and indenopyridines **1a,b** it was established that the main products are the 4-nitro derivatives **2a,b**, isolated by column chromatography as garnet-red crystals in yields of 50 and 49% respectively.

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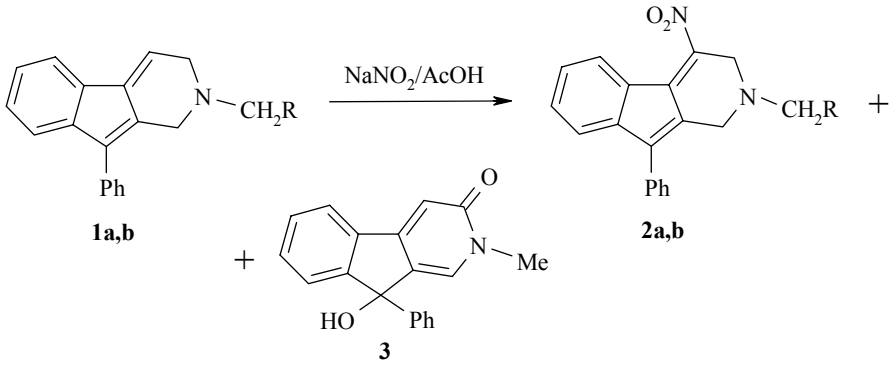
[1]People's Friendship University of Russia, Moscow 117198; e-mail: swelfen@mail.ru. [2]A. N. Nesmeyanov Institute of Organoelement Compounds, Russian Academy of Sciences, Moscow 119991; Translated from Khimiya Geterotsiklicheskikh Soedinenii, No. 9, pp. 1390-1399, September, 2007. Original article submitted July 21, 2006.

0009-3122/07/4309-1181



1,2 a R = H, **b** R = Ph

High intensity absorption bands, characteristic of a nitro group conjugated with an olefinic bond, were observed in the IR spectra of the synthesized compounds at 1320 and 1514 cm^{-1}. In the mass spectra of both compounds peaks of various intensities were present for the molecular ions M^+, $[M–H]^+$, $[M–OH]^+$, $[M–HNO]^+$, and $[M–NO_2]^+$ ions, confirming the empirical formulas of products **2a,b**. The fact that the nitro group is found at position C(4) of the tetrahydropyridine ring is indicated first by the absence of a triplet signal of the H-4 proton (in the initial compounds it is observed at 6.87 and 6.80 ppm), and secondly by the significant displacement towards low field ($\Delta\delta$ = 0.6 ppm) of the doublet-doublet signal of the H-5 benzene proton. This may be linked primarily with the occurrence of the electron-withdrawing NO_2 group in a pseudo-*peri* position to this proton and its deshielding effect. For unequivocal confirmation of the fact that under electrophilic nitrosation conditions oxidation of the nitroso group to nitro occurs, an X-ray structural investigation of nitro compound **2a** was carried out (Fig. 1, Tables 1 and 2).

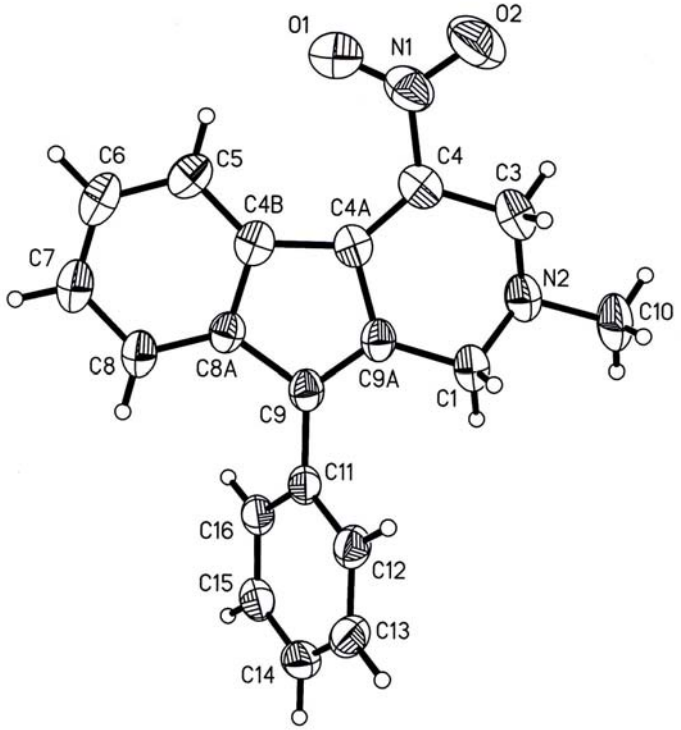


Fig. 1. Molecular structure of compound **2a** (atoms are shown as 40%-probability ellipsoids of anisotropic displacements).

TABLE 1. Bond Lengths (*l*) in Structure **2a**

Bond	*l*, Å	Bond	*l*, Å
O(1)–N(1)	1.193(3)	C(5)–C(6)	1.388(4)
O(2)–N(1)	1.205(3)	C(6)–C(7)	1.362(4)
N(1)–C(4)	1.460(3)	C(7)–C(8)	1.397(3)
C(1)–N(2)	1.444(3)	C(8)–C(8A)	1.373(3)
C(1)–C(9A)	1.483(3)	C(8A)–C(9)	1.481(3)
N(2)–C(3)	1.442(3)	C(9)–C(9A)	1.353(3)
N(2)–C(10)	1.456(3)	C(9)–C(11)	1.470(3)
C(3)–C(4)	1.492(3)	C(11)–C(12)	1.387(3)
C(4)–C(4A)	1.356(3)	C(11)–C(16)	1.398(3)
C(4A)–C(9A)	1.466(3)	C(12)–C(13)	1.371(3)
C(4A)–C(4B)	1.484(3)	C(13)–C(14)	1.373(3)
C(4B)–C(5)	1.381(3)	C(14)–C(15)	1.367(3)
C(4B)–C(8A)	1.416(3)	C(15)–C(16)	1.369(3)

TABLE 2. Valence Angles (ω) in Structure **2a**

Angle	ω, deg	Angle	ω, deg
O(1)–N(1)–O(2)	121.9(3)	C(6)–C(7)–C(8)	120.2(2)
O(1)–N(1)–C(4)	120.8(2)	C(8A)–C(8)–C(7)	118.6(2)
O(2)–N(1)–C(4)	117.3(3)	C(8)–C(8A)–C(4B)	121.36(19)
N(2)–C(1)–C(9A)	109.70(19)	C(8)–C(8A)–C(9)	129.6(2)
C(3)–N(2)–C(1)	111.79(18)	C(4B)–C(8A)–C(9)	108.98(17)
C(3)–N(2)–C(10)	110.9(2)	C(9A)–C(9)–C(11)	126.70(19)
C(1)–N(2)–C(10)	111.9(2)	C(9A)–C(9)–C(8A)	108.16(19)
N(2)–C(3)–C(4)	112.31(18)	C(11)–C(9)–C(8A)	125.14(17)
C(4A)–C(4)–N(1)	124.0(2)	C(9)–C(9A)–C(4A)	110.12(18)
C(4A)–C(4)–C(3)	122.3(2)	C(9)–C(9A)–C(1)	128.5(2)
N(1)–C(4)–C(3)	113.7(2)	C(4A)–C(9A)–C(1)	121.40(19)
C(4)–C(4A)–C(9A)	116.04(19)	C(12)–C(11)–C(16)	117.9(2)
C(4)–C(4A)–C(4B)	137.4(2)	C(12)–C(11)–C(9)	120.85(19)
C(9A)–C(4A)–C(4B)	106.52(17)	C(16)–C(11)–C(9)	121.27(18)
C(5)–C(4B)–C(8A)	118.8(2)	C(13)–C(12)–C(11)	121.1(2)
C(5)–C(4B)–C(4A)	135.0(2)	C(12)–C(13)–C(14)	119.8(2)
C(8A)–C(4B)–C(4A)	106.18(18)	C(15)–C(14)–C(13)	120.2(2)
C(4B)–C(5)–C(6)	119.1(2)	C(14)–C(15)–C(16)	120.4(2)
C(7)–C(6)–C(5)	121.8(2)	C(15)–C(16)–C(11)	120.5(2)

The indenopyridine portion of molecule **2a** (with the exception of atom N(2)) was practically planar (mean square deviation of atoms from the mean plane was 0.018 Å). Atom N(2) emerges from this plane by 0.618 Å, and consequently the tetrahydropyridine ring has a *sofa* conformation.The nitro group is disposed in a practically coplanar manner with the indenopyridine (angle between the corresponding planes is 60°), while the phenyl substituent is opened at an angle of 50.3° in relation to the same plane.

Atom N(2) has a pyramidal configuration (sum of valence angles at atom N(2) is equal to 334.6°).

The bond lengths and valence angles in the **2a** molecule have average values [8].

In the synthesis of nitro compound **2a** the product of partial oxidation of the initial **1a**, *viz.* 9-hydroxy-3-oxo-2,3-dihydroindenopyridine **3**, was also isolated from the reaction mixture in 17% yield. This compound is formed as a result of isomerization of the *s-trans* diene fragment into the *s-cis* diene system fixed by oxidation of the 3-CH_2 group into a keto group with the formation of the thermodynamically stable α-pyridone system.

The similar processes may be accompanied by a C(1)→C(9) prototropic shift, but the high C(9)-H acidity of the triarylmethine proton [9] leads to facile oxidation of this fragment to the corresponding carbinol. It is necessary to mention that compound **3** has melting point, IR, ^{1}H NMR, and mass spectra identical to those of the substance obtained previously by us by the oxidation of the initial **1a** with manganese dioxide at room temperature [5]. With the aim of finally establishing its structure and stereochemical features, an X-ray structural analysis was carried out on compound **3** (Fig. 2, Tables 3 and 4).

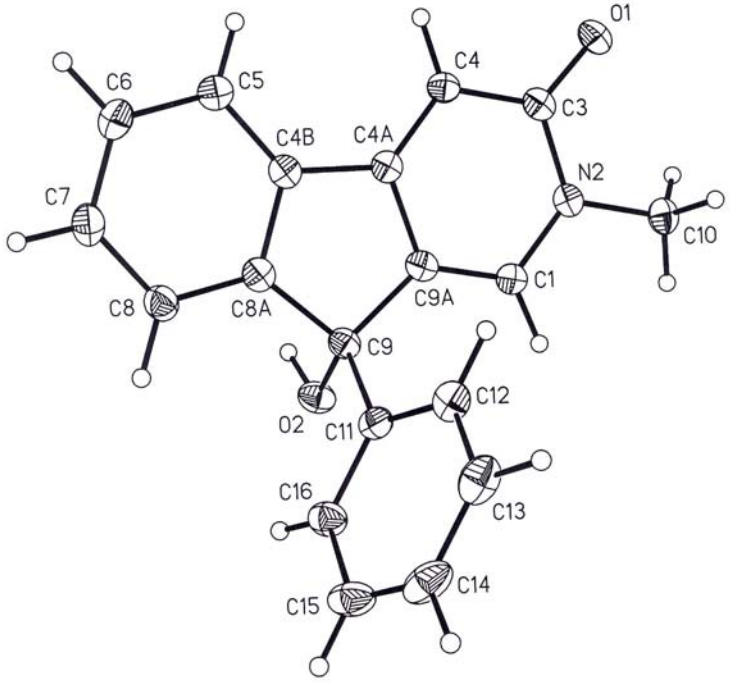


Fig. 2. Molecular structure of compound **3** (atoms are shown as 40% probability ellipsoids of anisotropic displacements).

TABLE 3. Bond Lengths (*l*) in Structure **3**

Bond	*l*, Å	Bond	*l*, Å
O(1)–C(3)	1.2575(14)	C(6)–C(7)	1.3939(19)
O(2)–C(9)	1.4319(14)	C(7)–C(8)	1.3951(19)
C(1)–C(9A)	1.3547(17)	C(8)–C(8A)	1.3854(17)
C(1)–N(2)	1.3756(15)	C(8A)–C(9)	1.5387(17)
N(2)–C(3)	1.3894(16)	C(9)–C(11)	1.5247(16)
N(2)–C(10)	1.4678(16)	C(9)–C(9A)	1.5281(16)
C(3)–C(4)	1.4360(17)	C(11)–C(16)	1.3902(18)
C(4)–C(4A)	1.3643(16)	C(11)–C(12)	1.3945(18)
C(4A)–C(9A)	1.4240(16)	C(12)–C(13)	1.3923(19)
C(4A)–C(4B)	1.4655(16)	C(13)–C(14)	1.386(2)
C(4B)–C(5)	1.3932(17)	C(14)–C(15)	1.381(2)
C(4B)–C(8A)	1.4024(16)	C(15)–C(16)	1.3886(19)
C(5)–C(6)	1.3907(18)		

TABLE 4. Main Valence Angles (ω) in Structure **3**

Angle	ω, deg	Angle	ω, deg
C(9A)–C(1)–N(2)	20.41(11)	O(2)–C(9)–C(11)	107.05(9)
C(1)–N(2)–C(3)	122.83(10)	O(2)–C(9)–C(9A)	112.92(9)
C(1)–N(2)–C(10)	119.88(10)	C(11)–C(9)–C(9A)	113.90(10)
C(3)–N(2)–C(10)	117.28(10)	O(2)–C(9)–C(8A)	111.77(10)
O(1)–C(3)–N(2)	119.28(11)	C(11)–C(9)–C(8A)	110.86(9)
O(1)–C(3)–C(4)	124.06(11)	C(6)–C(5)–C(4B)	118.15(12)
N(2)–C(3)–C(4)	116.66(10)	C(5)–C(6)–C(7)	120.95(12)
C(4A)–C(4)–C(3)	120.13(11)	C(1)–C(9A)–C(9)	129.65(11)
C(6)–C(7)–C(8)	120.76(12)	C(16)–C(11)–C(9)	119.65(11)
C(8A)–C(8)–C(7)	118.64(12)	C(12)–C(11)–C(9)	121.47(11)
C(8)–C(8A)–C(9)	127.92(11)		

The indenopyridine fragment of the molecule **3** is practically planar (mean square deviation of atoms from the average plane was 0.024 Å). The plane of the phenyl substituent is disposed at angle of 74.7° to the plane of the indenopyridine fragment but the plane of the three-atom grouping (C(9)–O(2)–H(2O)) is at an angle of 84.5°.

The N(2) atom has a planar trigonal configuration (sum of valence angles at the N(2) atom is 360.0°).

The bond lengths and valence angles in the molecule **3** have average values [8].

In the crystal the molecules **3** form centrosymmetric dimers as a result of intermolecular hydrogen bonds O(2)-H(2O)···O(1) [-*x*, -*y*+1, -*z*+1] [O···O 2.759(2), H···O 1.81(2) Å, angle O-H···O 177(1)°] (Fig. 3).

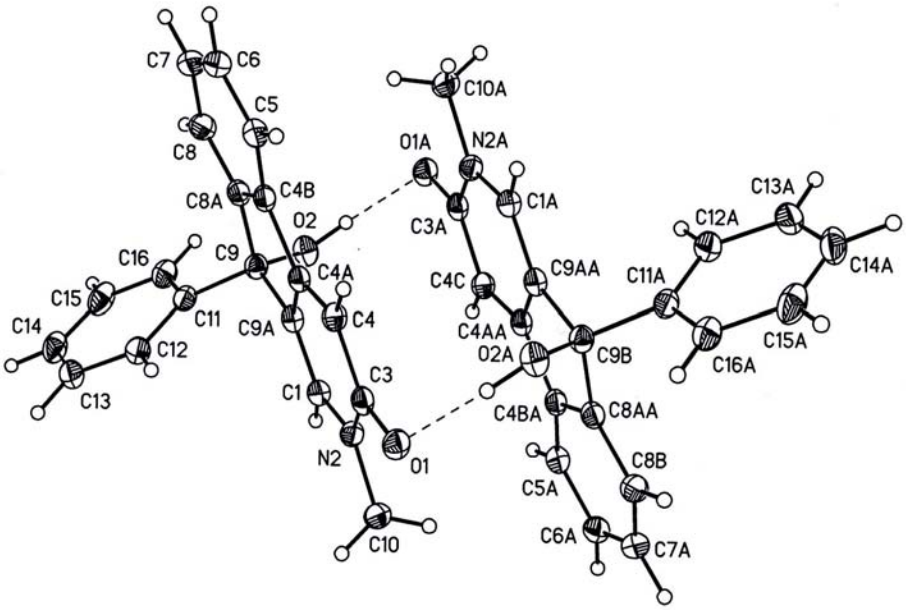
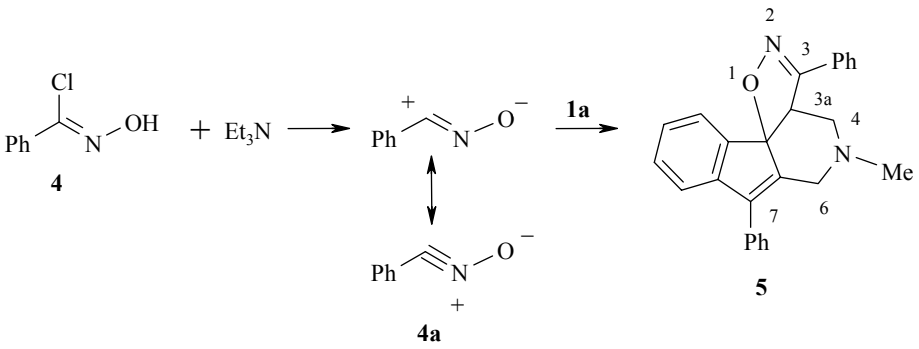


Fig. 3. Centrosymmetric dimers of compound **3** in the crystal, dotted lines show hydrogen bonds.

Reaction of indenopyridine **1a** was also carried out with chlorobenzaldehyde oxime **4**. In the presence of triethylamine the latter is converted into zwitter-ion **4a,** which reacts with compound **1a** by a [2+3] cycloaddition reaction with the possible formation of two isomers. As a result of chromatographic separation only one substance was isolated (in 29% yield) having, according to spectral data, the structure of tetrahydro-indeno[2,1-*c*]isoxazolo[5,4-*d*]pyridine **5**.



In its ^{1}H NMR spectrum the two H-4 protons experience the significant shielding effect of the benzaldimine fragment and resonate as two doublet-doublet signals at 2.51 and 2.82 ppm (in the spectrum of the initial compound **1a** the analogous two H-3 protons give one singlet signal at 3.41 ppm). The geminal coupling constants of these protons are ^2J = 11.0, but ^3J = 7.0 and 4.0 Hz respectively. This indicates the axial disposition of the H-3a methine proton, which resonates as a triplet signal at 3.97 ppm. Consideration of a three-dimensional Dreiding model of molecule **5** also shows that, due to the sp^2 configuration of the C(6a) atom and also participation of atoms C(3a), C(6a), and C(11b) in a rigid spiro-linked system of two five-membered rings, the piperidine ring must have a *sofa* configuration with axial disposition of the H-3a proton. A NOESY analysis was carried out for additional confirmation of the structure of compound **5**. The spatial structure of the isomeric adduct of cycloaddition **5** was established with the aid of the two-dimensional Overhauser effect on ^{1}H nuclei. Thus, in the 2D NOESY spectrum a cross-peak was observed between the H-3a protons (3.97 ppm) and the *ortho* protons of the phenyl substituent at C(3) (7.78 ppm), which was absent from the COSY spectrum. This fact indicates that the nuclei indicated are spatially close, which is only possible in the case of structure **5**.

TABLE 5. Main Crystallographic Data and Refinement Parameters for Compounds **2a** and **3**

Compound	**2a**	**3**
Empirical formula	$C_{19}H_{16}N_2O_2$	$C_{19}H_{15}NO_2$
M	304.34	289.32
T, K	293	120
System	Monoclinic	Monoclinic
Space group	$P2_1/c$	$P2_1/c$
a, Å	10.757(2)	13.3169(9)
b, Å	15.027(3)	9.5943(6)
c, Å	10.025(2)	11.0655(7)
β, deg	107.65(2)	93.932(5)
V, Å^3	1544.2(5)	1410.47(16)
Z	4	4
d_c, g/cm^{-3}	1.309	1.362
F(000)	640	608
μ, mm^{-1}	0.086	0.089
$2\theta_{max}$, deg.	54	54
Number of		
reflections measured	3527	12 949
independent reflections	3335	3054
observed reflections with $I > 2\sigma(I)$	1959	2612
refined parameters	208	259
R_1 ($I > 2\sigma(I)$)	0.056	0.042
wR_2 (all data)	0.140	0.116
GOOF	1.004	1.024

In conclusion we note that nitro compound **2a**, according to the prediction of the internet program PASS [10], is promising for biotesting as an agent for the treatment of psychosexual disorders, Alzheimer's disease, and hypertonia.

EXPERIMENTAL

The ^{1}H NMR spectra were recorded on a Bruker WM-400 (400 MHz) spectrometer in $CDCl_3$, internal standard was TMS. The mass spectra (EI) were obtained on a MAT-112 instrument with direct insertion of sample into the ion source at an ionizing voltage of 70 eV. The IR spectra were recorded on an IR-75 spectrometer in KBr disks. Silufol UV-254 plates were used for TLC (visualization with iodine vapor). Column chromatography was carried out on silica gel (Silicagel L 32/63). Compounds **1a** and **1b** were obtained as described in [11].

X-ray Structural Investigation of Compounds 2a and 3. Crystals of compounds **2a** and **3** were grown in benzene. Parameters of the unit cells and the intensity of reflections were measured on a Syntex $P2_1$ automatic diffractometer (T = 293 K, λMoKα radiation, graphite monochromator, θ/2θ scanning) (compound **2a**) and a Bruker SMART 1000 CCD automatic diffractometer (T = 120 K, λMoKα radiation, graphite monochromator, φ and ω scanning) (compound **3**). The main crystallographic data and the refinement parameters are given in Table 5. The structures of both compounds were determined by the direct method and were refined by the full matrix least squares method in an anisotropic approach for the non-hydrogen atoms. The positions of the hydrogen atoms in the case of compound **2a** were calculated geometrically and were refined in an isotropic approach with fixed positions ("rider" model) and thermal parameters (U_{iso}(H) = 1.5U_{eq}(C) for CH_3 groups and $U(H)_{iso}$ = 1.2U_{eq}(C) for all other groups). The hydrogen atoms in the case of compound **3** were localized objectively in Fourier difference syntheses and were refined isotropically. All calculations were carried out using the SHELXTL PLUS set of programs [12]. Tables of atomic coordinates, bond lengths, valence angles, and anisotropic thermal parameters for compounds **2a** and **3** have been deposited in the Cambridge Structural Data Bank (CCDC 658025 and 658026 respectively).

2-Methyl-4-nitro-9-phenyl-2,3-dihydro-1H-indeno[2,1-*c*]pyridine (2a). Sodium nitrite (0.53 g, 7.7 mmol) was added in portions during 1 h to a solution of indenopyridine **1a** (2 g, 7.7 mmol) in AcOH (15 ml). The mixture was stirred for a further 0.5 h at 20°C, then neutralized with saturated sodium carbonate solution (pH 7), and extracted with ether. The extract was dried over $MgSO_4$, the solvent evaporated, and the residue resolved on a chromatographic column (eluent hexane, then ethyl acetate–hexane, 1:5). Compound **2a** (1.17 g, 50%) was obtained first as garnet-red crystals, mp 102-103°C. IR spectrum, ν, cm^{-1}: 1320, 1514 (NO_2). ^{1}H NMR spectrum, δ, ppm (*J*, Hz): 2.48 (3H, s, CH_3); 3.53 (2H, s, H-1); 3.78 (2H, s, H-3); 7.20-7.50 (8H, m, H_{arom}); 8.18 (1H, dd, ^3J = 7.6, ^4J = 1.0, H-5). Mass spectrum, *m/z* (I_{rel}, %): 304 $[M]^+$ (4), 303 (7), 302 (10), 287 $[M–OH]^+$ (7), 286 (11), 278 (12), 274 (27), 273 $[M–H–NO]^+$ (100), 257 (30), 242 (11), 230 (19), 215 (15), 202 (17), 201 (22), 189 (7), 77 (4). Found, %: C 75.21; H 5.44; N 9.15. $C_{19}H_{16}N_2O_2$. Calculated, %: C 75.00; H 5.26; N 9.21. M 304.

9-Hydroxy-2-methyl-3-oxo-9-phenyl-2,3-dihydro-9H-indeno[2,1-*c*]pyridine (3) was then eluted from the chromatographic column as beige crystals. Yield was 0.38 g (17%). In mp (143-145°C), ^{1}H NMR, mass, and IR spectra substance **3** was identical with the substance isolated and described previously in [5].

2-Benzyl-4-nitro-9-phenyl-2,3-dihydro-1H-indeno[2,1-*c*]pyridine (2b) was obtained analogously to compound **2a**. Yield was 49%. Dark claret-crystals, mp 80-82°C. IR spectrum, ν, cm$^{-1:}$ 1328, 1521 (NO_2). ^{1}H NMR spectrum, δ, ppm (*J*, Hz): 3.39 (2H, s, H-1); 3.73 (2H, s, H-3); 3.83 (2H, s, $NCH_2C_6H_5$); 7.23-7.49 (8H, m, H_{arom}); 8.16 (1H, dd, ^3J = 7.5 and ^4J = 1.0, H-5). Mass spectrum, *m/z* (I_{rel}, %); 380 $[M]^+$ (8), 379 (8), 363 $[M–OH]^+$ (20), 349 $[M–H–NO]^+$ (5), 334 $[M–NO_2]^+$ (7), 333 (21), 242 (11), 215 (15), 91 (100), 65 (23). Found, %: C 78.73; H 5.47; N 7.34. $C_{25}H_{20}N_2O_2$. Calculated, %: C 78.95; H 5.26; N 7.37. M 380.

5-Methyl-3,7-diphenyl-3a,4,5,6-tetrahydroindeno[2,1-*c*]isoxazolo[5,4-*d*]pyridine (5). Triethylamine (0.19 g, 0.27 ml, 1.93 mmol) was added gradually (during 10 min) to a solution of benzohydroxymoyl chloride **4** (0.3 g, 1.93 mmol) in absolute ether (20 ml) cooled to 0-5°C. The mixture was stirred for 0.5 h, the solid filtered off, and a solution of indenopyridine **1a** (0.5 g, 1.93 mmol) in absolute ether (30 ml) was added carefully to the filtrate (benzonitrile N-oxide) cooled to 0°C. The mixture was then boiled for 6 h. The solvent was evaporated in vacuum, the residue was resolved on a chromatographic column, eluent was ethyl acetate–hexane, 1:1. Compound **5** (0.21 g, 29%) was obtained as light-yellow crystals, mp 145-147°C. ^{1}H NMR spectrum, δ, ppm (*J*, Hz): 2.27 (3H, s, CH_3); 2.50 (1H, dd, ^2J = 12.1 and ^3J = 7.1, H_e-4); 2.82 (1H, dd, ^2J = 12.1 and ^3J = 5.8, H_a-4); 3.42 (1H, d, ^2J = 13.8, H-6); 3.61 (1H, d, ^2J = 13.8, H-6); 3.96 (1H, t, ^3J = 7.0 and ^3J = 5.9, H-3a); 7.09-7.79 (14H, m, H_{arom}). Mass spectrum, *m/z* (I_{rel}, %): 378 $[M]^+$ (28), 335 $[M–CH_2NCH_3]^+ = \Phi_1$ (23), 334 (16), 258 $[\Phi_1\text{-Ph}]^+$ (37), 231 (67), 204 (100), 203 (87), 202 (46), 145 (42), 105 (29), 77 (51), 42 (87). Found, %: C 82.38; H 5.75; N 7.62. $C_{26}H_{22}N_2O$. Calculated, %: C 82.54; H 5.82; N 7.41. M 378.

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