Synthesis of 3,4-dihydroisoquinolines using nitroalkanes in polyphosphoric acid

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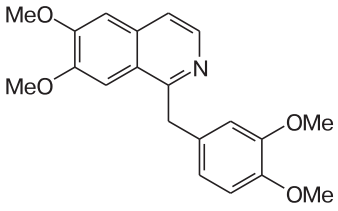
A new method for the synthesis of 6,7-dimethoxy-3,4-dihydroisoquinoline based on the reaction of 2-(3,4-dimethoxyphenyl)ethan-1-amine (homoveratrylamine) with aliphatic nitro compounds in polyphosphoric acid (PPA) was developed.

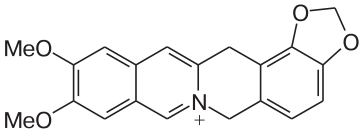
Key words: phenylethylamines, 6,7-dimethoxy-3,4-dihydroisoquinoline, nitro compounds, polyphosphoric acid, alkaloids.

Modern organic chemistry is mainly governed by the interest of the pharmaceutical industry to generation of new synthetic drugs, as well as modification of already existing ones in order to increase their activity and reduce their toxicity. The solution of these problems is impossible without using new effective synthetic methods. Such techniques should not involve usage of heavy metals and other toxic compounds that complicate the following purification of the target products. Therefore, the search for methods of assembling new structures that enables one to effectively synthesize large libraries of compounds for screening biological activity is still an urgent task. The isoquinoline fragment as well as partially hydrogenated isoquinoline derivatives are often found in natural compounds, for example, in the structure of alkaloids of the isoquinoline series, such as papaverine **1**, barberine **2**, scoulerine **3**, cochlaurine **4**, No-spa **5**, *etc*. Many of these alkaloids demonstrate different physiological activities and therefore various derivatives of this heterocyclic system, including partially hydrogenated, are widely used for the search of new drugs.[1—7]

The synthesis of 3,4-dihydroisoquinolines usually involves the assembly of the pyridine core by the Bischler—Napieralski,[8—10] Ritter reactions,[11—13] and by other methods (see, for example, Ref. 14). Carboxylic acid derivatives, which are not always readily available, are often utilized in these methods. In addition, the possibility of functionalization of isoquinolines and their derivatives with partially hydrogenated heterocycles should be mentioned.[15—17]



1 (papaverine)



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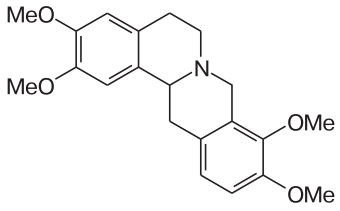
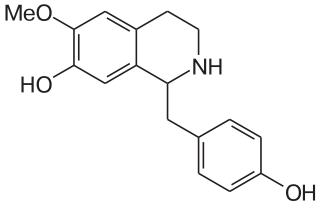
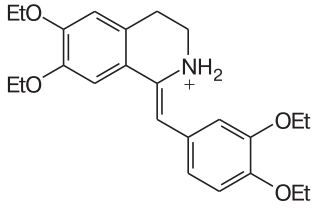


3 (scoulerine)



4 (cochlaurine)



5 (No-spa)

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In the present work, we report on a highly effective modification of the Bischler—Napiralski reaction, which serves to produce 3,4-dihydroisoquinolines from activated nitroalkanes but not from carboxylic acid derivatives.

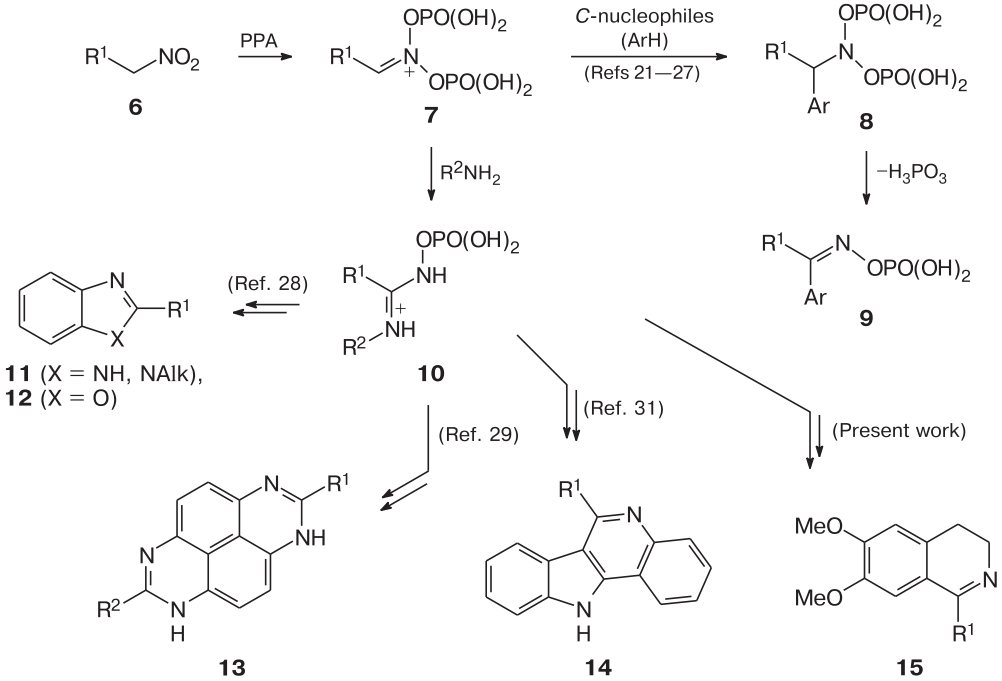
The binary phosphoric anhydride—water system has great potential from the viewpoint of the design of "smart" reaction media for conducting organic reactions catalyzed by Brønsted acids. Thus, when the content of phosphoric anhydride is over 75%, polyphosphoric acid (PPA) is present in the system, which allows one to tune such physical and chemical parameters of the medium as polarity, effective acidity, and anhydride activity. The behavior of nitroalkanes in PPA is of particular interest. Nitro compounds are one of the most convenient and widely used in organic synthesis of synthons for building the carbon—carbon and carbon—heteroatom bonds.[18,19] The availability of aliphatic nitro compounds, as well as their high reactivity, make them attractive building blocks in the synthesis of a wide variety of natural and biologically active nitrous compounds.[20] Nevertheless, utilization of nitro compounds in organic synthesis is limited mainly by the introduction of electrophilic reagents in the α-position. This fact, evidently, limits the scope of their application to basic and weakly acidic media. In PPA, nitroalkanes **6** form a stable, double phosphorylated *aci*-form **7**, which can act as an electrophilic component in many selective processes that proceed similarly to the Nef reaction with *C*-nucleophiles[21—27] and *N*-nucleophiles[28—30] participating. Some examples of such reactions in which *C*- and *N*-nucleophiles participate simultaneously (Scheme 1) are reported.[31]

The reaction of the *aci*-form **7** with electron rich aromatic hydrocarbons usually results in phosphorylated *N*-hydroxyhydroxylamines **8**, which further eliminate orthophosphoric acid to form phosphorylated oximes **9**. Under the same conditions, oximes **9** can participate in either the Beckmann rearrangement or the Wagner—Meerwein rearrangement providing an additional opportunity for the introduction of functional groups into the aromatic ring (see Scheme 1).[21—30] The reaction of the *aci*-form **7** with *N*-nucleophiles yields a stabilized amidinium ion **10**. In the presence of a second nucleophilic functional group or an aromatic ring, intramolecular cyclocondensation is possible, which allowed us to develop effective synthetic approaches to imidazoles **11**, oxazoles **12**,[28] tetraazopyrenes **13**,[29] and 11*H*-indolo[3,2-*c*]quinolines **14**[31] (see Scheme 1).

Application of nitro compounds instead of carboxylic acids has several advantages. First, nitro compounds, in particular 2-aryl-1-nitroethanes, synthesized by the reduction of the corresponding nitroalkenes, are readily available compounds. Secondly, utilization of nitro compounds made it possible to decrease the reaction temperature by 100 °C compared to a similar method in which carboxylic acids were used.[32]

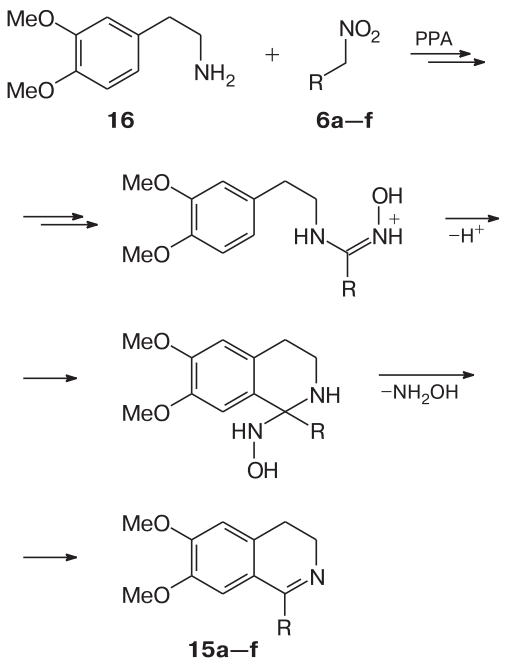
We suggested that the same strategy can be applied in the synthesis of 3,4-dihydroisoquinolines **15** by the reaction of nitro alkanes, as activated electrophiles, with 2-(3,4-dimethoxyphenyl)ethane-1-amine (**16**). The usage of aliphatic amines in such transformations has not been reported earlier. To verify this suggestion, the mixture of

Scheme 1



amine **16** with nitroethane **6b** (R = Me) was stirred in 86% polyphosphoric acid at 100 °C for 2 h. Under these conditions, the corresponding 6,7-dimethoxy-3,4-dihydroisoquinoline (**15b**) was obtained with a yield of 63% (Scheme 2). Similar reactions were carried out with nitroalkanes **6a**, **c**—**f**, however, in these cases, the reaction temperature was elevated in accordance with a decrease in the volatility of the nitroalkane used, which allowed us to reduce the reaction time. Reactions involving all the used nitroalkanes **6a—f** proceeded smoothly to give 6,7-dimethoxy-3,4-dihydroisoquinolines **15a**—**f** in the yields of 36–63% (see Scheme 2).

Scheme 2



Compounds **6** and **15**	R	Yield of **15** (%)
a	H	50
b	Me	63
c	Et	36
d	C_7H_{15}	52
e	Ph	48
f	Bn	39

In summary, in the present work, a convenient method for the synthesis of 6,7-dimethoxy-3,4-dihydroisoquinolines was developed. The reaction of nitroalkanes activated by PPA with aliphatic amines was carried out for the first time.

Experimental

^{1}H and ^{13}C NMR spectra were recorded on a Bruker AVANCE III HD (400.40 (^{1}H) and 100.61 (^{13}C) MHz) instrument in $CDCl_3$, using residual signals of $CHCl_3$ as an internal standard. Electrospray ionization mass spectra were obtained using a Bruker maXis impact time-of-flight mass spectrometer equipped with a direct injection system. A mixture of HCO_2Na—HCO_2H was used to calibrate the instrument. IR spectra were measured on a Shimadzu IR Affinity-1S FT-IR spectrometer equipped with an attenuated total reflectance attachment. Purity of isolated compounds were controlled by TLC on Silufol UV-254 plates, eluting with ethanol—ethyl acetate (2 : 3); UV radiation was used for the visualization of the spots of compounds. Melting points were measured on a Stuart SMP30 apparatus.

11*H*-Indolo[3,2-*c*]isoquinolines 15a—f (general protocol). A mixture containing 90 mg (0.5 mmol) of homoveratrylamine **16**, 1.5 mmol of nitroalkane **6a—f**, and polyphosphoric acid (1.0 g, P_2O_5 content was 86%) was placed in an Erlenmeyer flask and stirred at 100—120 °C (100 °C in the case of nitromethane (**6a**) and nitroethane (**6b**)) until complete consumption of the reactants (*ca.* 1.5 h, TLC monitoring). Then the reaction mixture was colled down to room temperature, diluted with water (40 mL), and neutralized with 20% aqueous ammonia (*ca.* 7 mL) to obtain alkaline medium. The resulted mixture was extracted with ethyl acetate (4×15 mL), solvents were evaporated *in vacuo*, and the product was isolated by column chromatography (ethanol—ethyl acetate, 1 : 10→1 : 1.5).

6,7-Dimethoxy-3,4-dihydroisoquinoline (15a). Yield 50%, light yellow liquid. R_f 0.52 (ethyl acetate—EtOH, 3 : 2). IR, ν/cm^{-1}: 2949, 2835, 1630, 1611, 1577, 1519, 1458, 1275, 1119. ^{1}H NMR, δ: 2.73—2.63 (m, 2 H, C(4)); 3.72 (t, 2 H, C(3), J = 7.2 Hz); 3.89 (s, 3 H, C(6)OMe); 3.91 (s, 3 H, C(7)OMe); 6.66 (s, 1 H, C(5)); 6.80 (s, 1 H, C(8)); 8.22 (s, 1 H, C(1)). ^{13}C NMR, δ: 24.9(C(4)), 47.4 (C(3)), 56.1 (C(6)OMe), 56.2 (C(7)OMe), 110.4 (C(7)), 110.5 (C(8)), 121.5 (C(4a)), 130.0 (C(8a)), 147.9 (C(6)), 151.3 (C(7)), 159.8 (C(1)). MS, found: m/z 192.1024 $[M + H]^+$. Calculated for $C_{11}H_{14}NO_2$: 192.1019.

6,7-Dimethoxy-1-methyl-3,4-dihydroisoquinoline (15b). Yield 63%, light brown solid. M.p. 87.2—89.5 °C. R_f 0.21 (ethyl acetate—EtOH, 3 : 2). IR, ν/cm^{-1}: 2945, 2839, 2362, 1668, 1626, 1607, 1577, 1515, 1378, 1325, 1275, 1218, 1161, 1060. ^{1}H NMR, δ: 2.26 (s, 3 H, C(1)Me); 2.52 (t, 2 H, C(4), J = 7.6 Hz); 3.51 (t, 2 H, C(3), J = 7.6 Hz); 3.79 (s, 3 H, C(6)OMe); 3.80 (s, 3 H, C(7)OMe); 6.58 (s, 1 H, C(5)); 6.88 (s, 1 H, C(8)). ^{13}C NMR, δ: 23.1 (C(1)Me), 25.5 (C(4)), 46.6 (C(3)), 55.8 (C(6)OMe), 56.0 (C(7)OMe), 108.8 (C(8)), 110.1 (C(5)), 122.2 (C(4a)), 130.9 (C(8a)), 147.3 (C(6)), 150.7 (C(7)), 163.7 (C(1)). MS, found: m/z 206.1171 $[M + H]^+$. Calculated for $C_{12}H_{16}NO_2$: 206.1176.

1-Ethyl-6,7-dimethoxy-3,4-dihydroisoquinoline (15c). Yield 36%, brown liquid. R_f 0.29 (ethyl acetate—EtOH, 3 : 2). IR, ν/cm^{-1}: 2938, 2835, 2366, 1664, 1607, 1573, 1515, 1458, 1325, 1271, 1210, 1149, 1073, 1023. ^{1}H NMR, δ: 1.23 (t, 3 H, C(1)Et, J = 7.5 Hz); 2.65 (t, 2 H, C(4), J = 7.5 Hz); 2.77 (q, 2 H, C(1)Et, J = 7.4 Hz); 3.65 (t, 2 H, C(3), J = 7.5 Hz); 3.90 (s, 3 H, C(6)OMe); 3.91 (s, 3 H, C(7)OMe); 6.70 (s, 1 H, C(5)), 7.01 (s, 1 H, C(8)). ^{13}C NMR, δ: 11.7 (C(1)Et), 25.9 (C(4)), 28.7 (C(1)Et), 46.2 (C(3)), 56.1 (C(6)OMe), 56.4 (C(7)OMe), 109.0 (C(8)), 110.5 (C(5)), 121.4 (C(4a)), 131.9 (C(8a)), 147.7 (C(6)), 151.4 (C(7)), 168.9 (C(1)). MS, found: m/z 220.1333 $[M + H]^+$. Calculated for $C_{13}H_{18}NO_2$: 220.1332.

1-Heptyl-6,7-dimethoxy-3,4-dihydroisoquinoline (15d). Yield 52%, light yellow liquid. R_f 0.58 (ethyl acetate—EtOH, 3 : 2). IR, ν/cm^{-1}: 2938, 2366, 1680, 1577, 1520, 1275, 1153, 1031. ^{1}H NMR, δ: 0.86 (t, 3 H, C(1)C_7H_{15}, J = 6.7 Hz); 1.46—1.21

(m, 8 H, C(1)C_7H_{15}); 1.69—1.59 (m, 2 H, C(1)C_7H_{15}); 2.63 (t, 2 H, C(4), J = 7.5 Hz); 2.70 (t, 2 H, C(1)C_7H_{15}, J = 7.8 Hz); 3.63 (t, 2 H, C(3), J = 7.5 Hz); 3.90 (s, 3 H, C(6)OMe); 3.92 (s, 3 HC(7)OMe); 6.70 (s, 1 H, C(5)); 7.00 (s, 1 H, C(8)). ^{13}C NMR, δ: 14.2 (C(1)C_7H_{15}), 26.0 (C(1)C_7H_{15}), 22.8 (C(4)), 27.5 (C(1)C_7H_{15}), 29.2 (C(1)C_7H_{15}), 29.6 (C(1)C_7H_{15}), 31.9 (C(1)C_7H_{15}), 35.9 (C(1)C_7H_{15}), 46.4 (C(3)), 56.1 (C(6)OMe), 56.4 (C(7)OMe), 109.1 (C(8)), 110.5 (C(5)), 121.7 (C(4a)), 131.9 (C(8a)), 147.6 (C(6)), 151.2 (C(7)), 167.9 (C(1)). MS, found: m/z 290.2118 $[M + H]^+$. Calculated for $C_{18}H_{28}NO_2$: 290.2115.

6,7-Dimethoxy-1-phenyl-3,4-dihydroisoquinoline (15e). Yield 48%, light yellow liquid. R_f 0.19 (ethyl acetate—EtOH, 3 : 2). IR, ν/cm^{-1}: 2938, 2838, 2351, 1611, 1561, 1515, 1462, 1363, 1279, 1218, 1119, 1031. ^{1}H NMR, δ: 2.74 (t, 2 H, C(3), J = 7.4 Hz); 3.73 (s, 3 H, C(4)); 3.82 (t, 2 H, C(6)OMe, J = 7.4 Hz); 3.95 (s, 3 H, C(7)OMe); 6.79 (s, 1 H, C(5)); 6.79 (s, 1 H, C(8)); 7.48—7.40 (m, 3 H, C(3)$_{C(1)Ph}$, C(4)$_{C(1)Ph}$, C(5)$_{C(1)Ph}$); 7.61 (dd, 2 H, C(2)$_{C(1)Ph}$, C(6)$_{C(1)Ph}$, J = 6.8 Hz, J = 2.4 Hz). ^{13}C NMR, δ: 26.1 (C(4)), 47.7 (C(3)), 56.2 (C(6)OMe), 56.3 (C(7)OMe), 110.3 (C(8)), 111.7 (C(5)), 121.6 (C(4a)), 128.3 (2 C, C(3)$_{C(1)Ph}$, C(5)$_{C(1)Ph}$), 128.9 (2 C, C(2)$_{C(1)Ph}$, C(6)$_{C(1)Ph}$), 129.5 (C(4)$_{C(1)Ph}$), 132.8 (C(8a)), 139.1 (C(1)$_{C(1)Ph}$), 147.2 (C(6)), 151.1 (C(7)), 167.0 (C(1)). MS, found: m/z 268.1337 $[M + H]^+$. Calculated for $C_{17}H_{18}NO_2$: 268.1332.

1-Benzyl-6,7-dimethoxy-3,4-dihydroisoquinoline (15g). Yield 39%, light yellow liquid. R_f 0.55 (ethyl acetate —EtOH, 3 : 2). IR, ν/cm^{-1}: 2949, 2839, 1672, 1611, 1573, 1515, 1458, 1367, 1321, 1275, 1153, 1046. ^{1}H NMR, δ: 2.65 (t, 2 H, C(4), J = 7.7 Hz); 3.71 (s, 3 H, C(7)OMe); 3.71 (t, 2 H, C(3), J = 7.7 Hz); 3.86 (s, 3 H, C(6)OMe); 4.04 (s, 2 H, C$\underline{H_2}$Ph); 6.65 (s, 1 H, C(5)); 6.94 (s, 1 H, C(8)); 7.31—7.23 (m, 5 H, $CH_2\underline{Ph}$). ^{13}C NMR, δ: 25.8 (C(4)), 43.4 ($\underline{C}H_2$Ph), 47.1 (C(3)), 55.9 (C(6)OMe), 56.0 (C(7)OMe), 109.6 (C(8)), 110.3 (C(5)), 121.5 (C(4a)), 126.5 (C(8a)), 128.6 (2 C, C(3)$_{Bn}$, C(5)$_{Bn}$), 128.7 (2 C, C(2)$_{Bn}$, C(6)$_{Bn}$), 131.8 (C(8a)), 138.1 (C(1)$_{Bn}$), 147.2 (C(6)), 150.7 (C(7)), 165.6 (C(1)). MS, found: m/z 282.1482 $[M + H]^+$. Calculated for $C_{18}H_{20}NO_2$: 282.1489.

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