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REDUCTION OF 2,3,4-SUBSTITUTED QUINOLINES WITH SODIUM BOROHYDRIDE

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UDC 547.831.07

1,2-Dihydroquinolines were obtained by the reduction of 3-substituted 2-methyl-4-phenylquinolines with sodium borohydride in aliphatic carboxylic acids; N-alkyl derivatives are also formed. The corresponding 1,4-dihydroquinoline was obtained in the reaction of 2-methyl-3-nitroquinolinium perchlorate with sodium borohydride.

Little study has been devoted to 1,4-dihydroquinolines (1,4-DHQ) [1-3], although they are close analogs of 1,4-dihydropyridines, which are used in practice as coronary dilating agents and antioxidants. 2-Methyl-4-aryl-N-unsubstituted 1,4-DHQ are most interesting from this point of view. However, this group of compounds has not been adequately studied. Only patent data [2] on the possibility of the synthesis of 2-methyl-4-phenyl-1,4-DHQ by the condensation of α-(2-aminophenyl)benzyl alcohol with ethyl acetoacetate are available. A greater amount of study has been devoted to 2- and 4-unsubstituted 1,4-DHQ obtained chiefly by the reduction of 3-substituted quinolines [1, 4, 5] or by the hydrogenation of the corresponding quinolinium salts [4, 6, 7].

It has been shown [1] that the controlling condition for obtaining 1,4-DHQ by the reduction of quinolines with sodium borohydride in ethanol is the presence of electron-acceptor groups in the 3 position and the absence of substituents in the 2 and 4 positions. The reduction of 3-halo-substituted quinolines or quaternary N-methylquinolinium salts leads primarily to 1,2-DHQ [1, 6]. 2-Substituted quinolines are reduced by lithium aluminum hydride or sodium borohydride to 1,2-DHQ [8]. No data on the reduction of 2,4-disubstituted quinolines are available.

We have established that 2-methyl-4-phenyl-3-substituted quinolines Ia-h are not reduced by sodium borohydride even in the case of prolonged refluxing in ethanol; this is in agreement with the proposed [1] mechanism for the reduction of the quinoline ring.

Quinolines Ia-h (Table 1), which are substituted in the 2, 3, and 4 positions, are reduced at 20°C by sodium borohydride in acetic acid to give 1,2-DHQ IIa-h. In addition to reduction of the ring, the hydrogen atom attached to the nitrogen atom is replaced by an ethyl group (see the scheme).

Similarly, N-methyl-1,2-DHQ IIIa-c were obtained in good yields by hydrogenation of quinolines Ia-c with sodium borohydride in formic acid (see the experimental section, method A). It should be noted that formic acid is used in a fivefold molar ratio with respect to Ia-h in ethanol, since reduction does not occur in it without a solvent. Quinolines Ia-c are also satisfactorily reduced by sodium borohydride in propionic acid, but N-propyl-1,2-DHQ were not isolated because of their instability. The structure of the latter is confirmed by the characteristic (for 1,2-DHQ) absorption maxima in the electronic spectra of the reaction mixtures.

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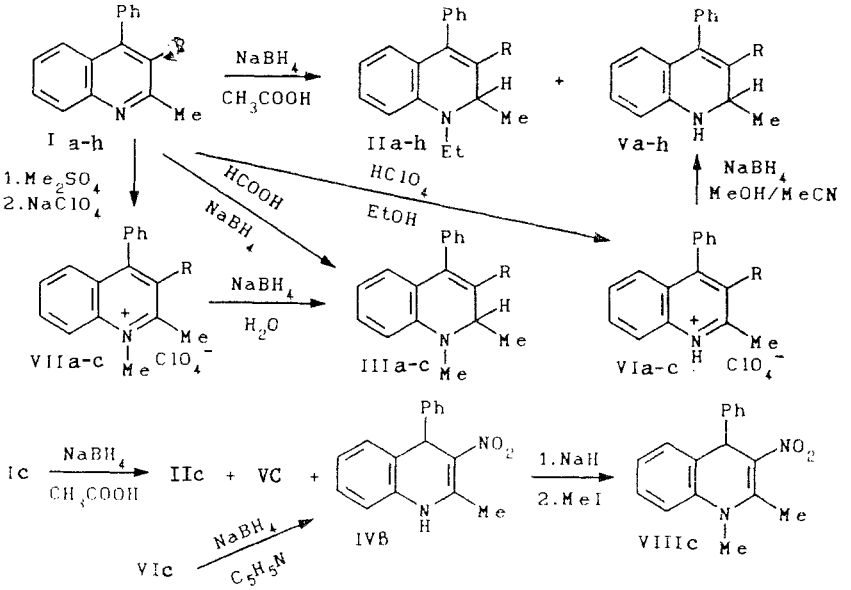
TABLE 1. Characteristics of Quinolines Ic, f-h and Quinolinium Salts VIa-c and VIIa-c

Compound	Empirical formula	mp, °C	Yield %	Compound	Empirical formula	mp, °C	Yield, %
Ic	$C_{16}H_{12}N_2O_2$	149 ... 151	89	VIb	$C_{17}H_{13}ClN_2O_4$	203	66
If	$C_{17}H_{14}N_2O$	241 ... 243	82	VIc	$C_{16}H_{13}ClN_2O_6$	286	64
Ig	$C_{19}H_{17}NOS$	100	85	VIIa	$C_{20}H_{20}ClNO_6$	143	65
Ih	$C_{22}H_{17}NO_2S$	130 ... 134	60	VIIb	$C_{13}H_{15}ClN_2O_4$	210	99
VIa	$C_{19}H_{18}ClNO_6$	140	92	VIIc	$C_{17}H_{15}ClN_2O_6$	230	93

Mixtures of products, which are separated by preparative TLC, are formed in the reduction of quinolines Ia-h.

Scheme 4



I, II a R=$COOC_2H_5$, b R=CN, c R=NO_2, d R=$COCH_3$, e R=COC_6H_5, f R=$CONH_2$, g R=$COSC_2H_5$, h R=$SO_2C_6H_5$; III—VII a R=$COOC_2H_5$, b R=CN, c R=NO_2

The course of the reaction evidently depends on the electron-acceptor properties of the β substituents. Relatively weak electron-acceptor groups in the 3 position of the quinoline ring (Ia, f) determine the formation of chiefly N-ethyl-1,2-DHQ IIa, f, which were isolated from the reaction mixtures in good yields.

In the reduction of 3-nitroquinoline Ic by sodium borohydride in acetic acid 3-nitro-1,4-DHQ was isolated by preparative TLC as the principal product, along with N-ethyl-1,2-DHQ IIc and 1,2-DHQ Vc, which are stable under ordinary conditions. Nevertheless, in other cases, when relatively strong electron-acceptor groups are present in the β position, 1,4-DHQ cannot be detected in the reaction mixtures (by TLC). Thus the reduction of quinolines Ib, d, e, g, h with sodium borohydride in acetic acid leads to mixtures of N-ethyl-1,2-DHQ IIb, d, e, g, h and Vb, d, e, g, h. The mixtures are separated by preparative TLC. During chromatography, N-unsubstituted Vb, d, e, g, h are rapidly oxidized to the corresponding quinolines Ib, d, e, g, h. The introduction of an N-alkyl group into 1,2-DHQ prevents air oxidation; this is probably associated with facilitated detachment of hydrogen during oxidation for N-unsubstituted 1,2-DHQ as compared with the N-alkyl derivatives. In the opinion of Dauphinee and Forrest [8], the presence of a methyl group in the 2 position in 1,4-unsubstituted 1,2-DHQ leads to an increase in their instability: the latter are readily oxidized in air or undergo disproportionation to quinolines and tetrahydroquinolines.

The formation of N-ethyl-1,2-DHQ IIa during the reduction of the quinolines in acetic acid was proved by alternative synthesis. The reduction of quinolinium perchlorate VIa leads to unstable 1,2-DHQ Va, which, without isolation, is alkylated with ethyl bromide in DMSO in the presence of sodium hydride. N-Ethyl-1,2-DHQ, which was identical to IIa, was isolated by preparative TLC.

TABLE 2. Characteristics of 3-Substituted Dihydroquinolines II-V and VIII

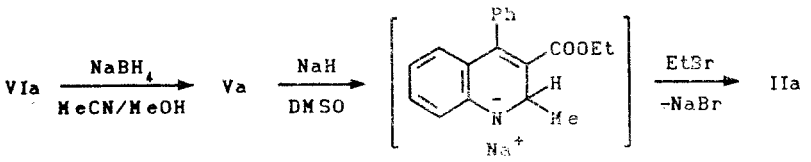
Compound	Empirical formula	mp, °C	UV spectrum, λ_{max}, nm (log ε)	PMR spectrum (d_6-DMSO), δ, ppm: 2-CH_3, 3H	N—H, 1H ($N—CH_3$, 3H)	N—CH_2CH_3, 3H, t	N—CH_2, 2H, q	2-CH, 1H, q	R	Arom. protons, 9H, m	Reaction time, h	Yield, %
IIa	$C_{21}H_{23}NO_2$	81	208 (4,38), 257 (4,39), 302 (3,64), 420 (3,51)	1,16 d	—	1,27	3,78	4,49	0,71 t	6,44 ... 7,38	3	51
IIb	$C_{19}H_{18}N_2$	88	205 (4,41), 255 (4,48), 300 sh (3,70), 423 (3,83)	1,30 d	—	1,42	3,38	4,29	—	6,44 ... 7,46	3	38
IIc	$C_{18}H_{18}N_2O_2$	91	207 (4,48), 267 (4,15), 329 (3,78), 498 (3,51)	1,20 d	—	1,24	3,47	4,89	—	6,49 ... 7,40	1	22
IId	$C_{20}H_{21}NO$	101 ... 103	204 (4,35), 263 (4,36), 313 (3,81), 446 (3,51)	1,00 d	—	1,20	3,37	4,49	1,47 s	6,44 ... 7,44	3	43
IIe	$C_{25}H_{23}NO$	93	207 (4,30), 240 (4,25), 320 sh (3,47), 435 (3,45)	1,18 d	—	1,22	3,38	4,33	—	6,49 ... 7,62	2	42
IIf	$C_{19}H_{20}N_2O$	153 ... 158	205 (4,36), 249 (4,36), 298 sh (3,30), 395 (3,30)	1,13 d	—	1,22	3,31	4,31	6,38 d	6,45 ... 7,36	2	51
IIg	$C_{21}H_{23}NOS$	62 ... 68	207 (4,38), 260 (4,30), 312 (3,78), 444 (3,64)	1,02	—	1,29	3,38	4,47	0,93 t 2,60 q	6,44 ... 7,40	3	34
IIh	$C_{24}H_{23}NO_2S$	148 ... 152	208 (4,40), 256 (4,30), 303 sh (3,72), 416 (3,51)	1,04	—	1,16	3,27	4,56	—	6,29 ... 7,33	1	58
Va	$C_{19}H_{19}NO_2$	93 ... 96	205 (4,21), 337 (4,32), 408 (3,60)	—	—	—	—	—	—	—	0,5	20
Vb*	$C_{17}H_{14}N_2$	70 ... 75	220 (4,10), 245 (4,42), 416 (3,24)	—	—	—	—	—	—	—	2	27
Vc	$C_{16}H_{14}N_2O_2$	140 ... 145	210 (4,35), 267 (4,13), 330 (3,70), 488 (3,32)	1,31 d	7,02 d	—	—	4,82 m	—	6,38 ... 7,44	2	35
IVc	$C_{16}H_{14}N_2O_2$	220	205 (4,51), 222 sh (4,32), 256 (4,05), 408 (4,26)	2,62 s	10,20 s	—	—	—	—	7,18, s*2	0,5	92
IIIa	$C_{20}H_{21}NO_2$	90 ... 94	208 (4,38), 254 (4,44), 304 (3,56), 416 (3,30)	2,00 d*3	(3,78 s)	—	—	5,29	1,60 t, 4,67 q	7,29 ... 8,09	1	60 (81)*4
IIIb	$C_{18}H_{16}N_2$	65 ... 68	208 (4,46), 252 (4,40), 304 (3,73), 422 (3,42)	1,19 d	(2,91 s)	—	—	4,38	—	6,52 ... 7,49	1	45 (70)*4
IIIc	$C_{17}H_{16}N_2O_2$	105 ... 107	208 (4,60), 269 (4,28), 333 (3,97), 496 (3,74)	1,18 d	(2,96 s)	—	—	4,80	—	6,38 ... 7,40	1	72 (85)*4
VIII	$C_{17}H_{16}N_2O_2$	136 ... 138	205 (4,13), 220 sh (4,00), 263 sh (3,50), 416 (4,15)	2,44 s	(3,58 s)	—	—	—	—	7,04 ... 7,42*5	—	53

*The substance contained admixed Ib.

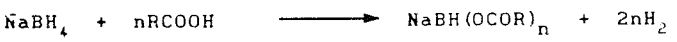
**The signal of the proton attached to the $C_{(4)}$ atom shows up in the form of a singlet at 5.44 ppm.

***The spectrum was recorded in $CDCl_3$.

****The yield obtained by method A; the yield obtained by method B is given in parentheses.

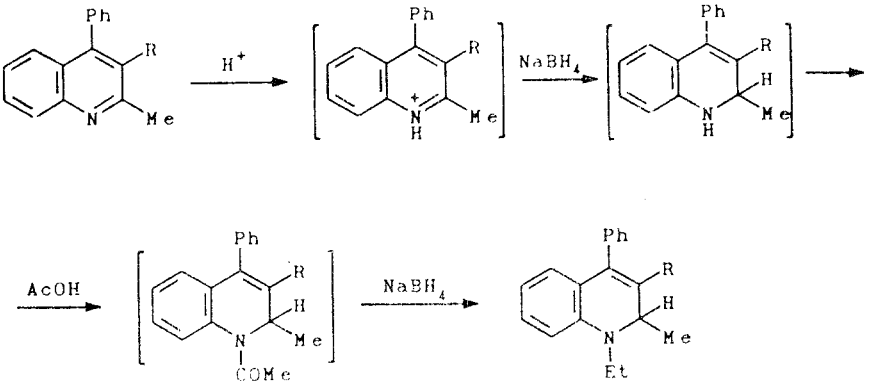


In a study of the reduction of compounds of various classes with sodium borohydride in carboxylic acids it was established [9] that the reducing agent is a sodium acyloxoborohydride:



The use of this reaction for the reduction of quinoline led to N-ethyl-1,2,3,4-tetrahydroquinoline [10]. A reaction mechanism that includes reduction of the protonated form of quinoline with subsequent N-alkylation of the resulting 1,2-DHQ was proposed [11, 12].

It seems likely to us that the formation of N-alkyl-1,2-DHQ IIa-h and IIIa-c may also proceed via a similar pathway. The reduction of the protonated form of the quinoline leads to a 1,2-DHQ, which is acylated by the acid, and the N-acyl group is reduced by sodium borohydride to an alkyl group.



The reduction of quinolinium salts VIa-c with sodium borohydride in a mixture of acetonitrile and methanol at room temperature in an argon atmosphere gives 1,2-DHQ Va-c. As noted above, Va, b are extremely unstable and are partially oxidized during isolation and crystallization. On the other hand, 3-nitro-1,2-DHQ Vc – a stable substance – was obtained in quantitative yield in the reduction of perchlorate VIc. Only 1,4-DHQ IVc was obtained in almost quantitative yield by the action of sodium borohydride in pyridine on quinolinium salt VIc [13]. Perchlorates VIa, b, which contain weaker electron-acceptor groupings than VIc, are not reduced by sodium borohydride in pyridine. 1,4-DHQ IVc displays the characteristic properties peculiar to hydrogenated nitrogen-containing heterocycles, i.e., it is oxidized by nitric acid to the corresponding quinoline Ic and, by the action of sodium hydride in dimethoxyethane, forms an anion that is methylated by methyl iodide to give N-methyl-1,4-DHQ VIII.

Quinoline perchlorates VIa-c are obtained in good yields by the action of perchloric acid on quinolines Ia-c in alcohol in a molar ratio of 1:2.

As in the synthesis of pyridinium salts [14], a preparative method for obtaining N-methylquinolinium slats VIIa-c is methylation of the corresponding quinolines Ia-c with dimethyl sulfate with the subsequent action of sodium perchlorate on the methylsulfates obtained. In turn, the reduction of N-methylquinolinium perchlorates VIIa-c with sodium borohydride in water or pyridine leads to N-methyl-1,2-DHQ IIIa-c (method B) in almost quantitative yields.

The structures of all of the synthesized compounds were confirmed by spectral methods. In the PMR spectra of N-ethyl-1,2-dihydro isomers IIa-h the signal of the 2-CH_3 group shows up in the form of a doublet that is overlapped by the triplet of the signal of the protons of the N—CH_2—CH_3 group. The signals of the protons of the N—CH_2 group show up in the form of two overlapped quartets: this constitutes evidence for hindered rotation of the N-ethyl group, which leads to nonequivalence of the hydrogen atoms of the N—CH_2 group. The presence of strong electron-acceptor groups in the β position causes a shift of the 2-CH signal (IIb, c) to weaker field as compared with IIa, f (quartets at ~4.5 ppm). For 1,2-DHQ Vc the signal of

the 2-CH proton shows up in the form of a multiplet, which reflects spin-spin coupling with the protons of the 2-CH_3 and N—H groups. This is in agreement with the PMR spectra of the corresponding 1,2-dihydropyridines [15] and confirms the isomeric 1,2-dihydro structure. For 1,4-DHQ IVc one observes a shift of the signal of the protons of the N—H group (10.18 ppm) to weak field as compared with the corresponding 1,4-dihydropyridine [16].

Three absorption bands that are characteristic for 1,2-DHQ and 1,2-dihydropyridines are observed in the UV region of the spectrum for II, IV, and V (Table 2); there is a bathochromic shift (~35 nm) of the long-wave maximum of IIa, b as compared with the corresponding 1,2-dihydropyridines [15], which constitutes evidence for lengthening of the conjugation chain in the 1,2-DHQ molecule, while the long-wave absorption maximum for 1,4-DHQ IVc and 2,6-dimethyl-3-ethoxycarbonyl-4-phenyl-1,4-dihydroquinoline (335 nm) [3] has a small hypsochromic shift as compared with 2,6-dimethyl-4-phenyl-3,5-diethoxycarbonyl-1,4-dihydropyridine (356 nm). The absorption maximum of the long-wave band of 1,2-DHQ Vc is shifted bathochromically by 78 nm as compared with 1,4-DHQ IVc, which is in good agreement with the difference ($\Delta\lambda$ = 73 nm) of the long-wave maxima between 1,2-DHQ Va and 2-methyl-4-phenyl-3-ethoxycarbonyl-1,4-DHQ [3]. The absorption of 1,2-DHQ in both the visible and UV regions is sensitive to a change in the substituent attached to the nitrogen atom. Thus in the case of N-ethyl derivatives IIa-c, h the absorption maximum is shifted bathochromically by 5-20 nm as compared with Va-c, h, depending on the β substituent.

1,4-DHQ IVc is a weaker N—H acid than 2,6-dimethyl-3-nitro-4-phenyl-5-ethoxycarbonyl-1,4-dihydropyridine, as well as 1,2-DHQ Vc. 1,4-DHQ IVc forms an anion only in DMSO in the presence of sodium hydride, while the corresponding 1,4-dihydropyridines and 1,2-DHQ Vc are dissociated completely in a 0.1 M solution of potassium hydroxide in 90% alcohol.

EXPERIMENTAL

The UV spectra of solutions of the compounds in ethanol were obtained with a Specord UV-vis spectrophotometer. The PMR spectra were obtained with a Bruker WH/90 spectrometer (90 MHz) with tetramethylsilane (TMS) as the internal standard. The individuality of the synthesized substances was verified by TLC on Silufol UV-254 plates in a chloroform—hexane—acetone (4:10:0.5) system.

The starting quinolines Ia-h were obtained by condensation of 2-aminobenzophenone with derivatives of β-keto carboxylic acids under the conditions of the Friedlaender synthesis [17, 18].

The results of elementary analysis of Ic-h, IIa-h, IIIa-c, IVc, VIa-c, VIIa-c, and VIII were in agreement with the calculated values.

3-Substituted 1-Ethyl-2-methyl-4-phenyl-1,2-dihydroquinolines IIa-h. A 5-mmole sample of Ia-h was dissolved in 30 ml of glacial acetic acid, and argon was passed through the solution for 30 min. A 1.9-g (50 mmole) sample of sodium borohydride was added in portions in the course of 10 min, and the mixture was maintained under argon at 20°C for 1-3 h. The solution was poured into 150 ml of water, and the precipitated oil was washed with water. Compounds IIa, f were crystallized from methanol. In the remaining cases the yellow oil was dissolved in 10 ml of acetone and chromatographed in three stages on a preparative plate (220 by 260 mm; the thickness of the loose layer of silica gel 100/160 μ was 2-3 mm). The most mobile bright-yellow or red bands of 1-ethyl-1,2-DHQ IIb-e, g, h (R_f ~ 0.78), the centrally located yellow or red bands of 1,2-DHQ Vb, c, d-h (R_f ~ 0.34), and, in the case of IVc, the lowest band of the 1,4-DHQ (R_f ~ 0.10) were sorbed. The dihydroquinolines were eluted from the silica gel with acetone, the solvent was evaporated in vacuo, and the residue was recrystallized from ethanol.

3-Substituted 2-Methyl-4-phenylquinolinium Perchlorates VIa-c. A 0.6-ml (5 mmole) sample of 57% perchloric acid was added to a solution of 5 mmole of quinolines Ia-c in 30 ml of ethanol, and the mixture was refluxed for 3 h. The solvent was evaporated in vacuo, and the residue was cooled and treated with 20 ml of ether. The white crystals were removed by filtration and recrystallized from isopropyl alcohol.

3-Substituted 2-Methyl-4-phenyl-1,2-dihydroquinolines Va-c. A 3-mmole sample of perchlorate VIa-c was dissolved in 50 ml of acetonitrile containing 5 ml of methanol, and argon was passed through the solution for 20 min. A 0.57-g (15 mmole) sample of sodium borohydride was added with stirring in the course of 10 min, and the mixture was maintained at 20°C for 3 h. It was then diluted with a tenfold amount of water under argon, and the precipitated oil was washed with water (350 ml), dissolved in 5 ml of chloroform, and chromatographed in one stage on a preparative plate as described above. The yellow bands of Va (R_f 0.40) and Vb (R_f 0.30) and dark-red Vc (R_f 0.37) were collected. The yellow substances obtained contained admixed quinoline and were oxidized rapidly during isolation and crystallization. Compound Vc was crystallized from dilute ethanol.

2-Methyl-3-nitro-4-phenyl-1,4-dihydroquinoline (IVc). A 0.94-g (25 mmole) sample of sodium borohydride was added in portions under argon to a solution of 1.88 g (5 mmole) of quinolinium perchlorate VIc in 20 ml of dry pyridine, after which the mixture was maintained at 20°C for 12 h. It was then diluted with 80 ml of water, and the bright-yellow crystals were filtered and crystallized from ethanol. According to the PMR spectral data and the results of elementary analysis, IVc crystallized with one molecule of ethanol. Drying at 80°C in vacuo (10 mm) gave 1.22 g of IVc with mp 222°C.

3-Substituted 1,2-Dimethyl-4-phenylquinolinium Perchlorates VIIa-c. A mixture of the corresponding quinoline Ia-c and 1.42 ml (16 mmole) of freshly distilled dimethyl sulfate was heated at 60°C for 6 h, after which the mixture was cooled and washed with ether (3 × 10 ml). The residue was dissolved in 20 ml of water, and a saturated solution of sodium perchlorate was added until the liberation of a colorless substance ceased. The colorless substance was filtered, washed with water, and crystallized from isopropyl alcohol.

3-Substituted 1,2-Dimethyl-4-phenyl-1,2-dihydroquinolines IIIa-c. A. A 5-mmole sample of quinoline Ia-c was dissolved in 20 ml of ethanol, 1.15 g (25 mmole) of formic acid was added, and 0.95 g (25 mmole) of sodium borohydride was added under argon at 20°C. The mixture was allowed to stand for 24 h, after which it was diluted to 100 ml with water, and the yellow crystals were filtered and crystallized from 70% ethanol.

B. A 0.95-g (25 mmole) sample of sodium borohydride was added with stirring in portions in the course of 20 min under argon to a suspension of 5 mmole of the corresponding 1-methylquinolinium perchlorate VIIa-c in 50 ml of water, after which the mixture was stirred for 2 h at 20°C under argon. The bright-yellow or red crystals were filtered and crystallized from 70% ethanol.

1,2-Dimethyl-3-nitro-4-phenyl-1,4-dihydroquinoline (VIII). A 0.24-g (10 mmole) sample of sodium hydride was added to a solution of 1.07 g (4 mmole) of IVc in 30 ml of dry dimethoxyethane, the mixture was heated for 5 min on a water bath, 0.62 ml (10 mmole) of methyl iodide was added, and the mixture was refluxed on a water bath for 3 h. It was then cooled and poured into 50 ml of water, and the aqueous mixture was extracted with ethyl acetate (3 × 40 ml), washed with water (3 × 30 ml), and dried with anhydrous sodium sulfate. The solvent was removed in vacuo, and the residue was dissolved in 10 ml of acetone and chromatographed in two stages on a preparative plate as described above with a chloroform—hexane—acetone (9:9:1) solvent system. The bright-yellow band (R_f 0.47) was collected and eluted with ethanol, the solvent was evaporated in vacuo, and the residue was crystallized from ethanol.

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