

The Synthesis of Benzofuroquinolines. I. Some
Benzofuro[2,3-*b*]quinoline and Benzofuro[3,2-*c*]quinoline Derivatives

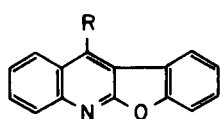
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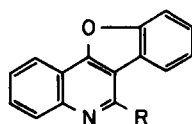
Benzofuro[2,3-*b*]quinoline (Ia) and its 11-methyl derivative (Ib) were synthesized by demethyl-cyclization of 3-(*o*-methoxyphenyl)-1,2-dihydroquinolin-2-ones (VIa,b). Benzofuro[2,3-*b*]quinoline-11-carboxylic acid (Id) was synthesized by chlorination followed by the action of potassium hydroxide of a lactone (IX) prepared by demethyl-cyclization of 3-(*o*-methoxyphenyl)-2-oxo-1,2-dihydroquinoline-4-carboxylic acid (VIII). Isomeric benzofuro[3,2-*c*]quinoline (IIa) and its 6-methyl derivative (IIb) were synthesized by demethyl-cyclization of 3-(*o*-methoxyphenyl)-1,4-dihydroquinolin-4-ones (XIa,b). Both the methyl derivatives (Ib and IIb) were converted to the carboxylic acids (Id and IIId) through condensation with benzaldehyde followed by oxidation. The benzofuroquinolines (Ia,b,d and IIa,b) thus obtained were oxidized to the corresponding *N*-oxides (IIIa,b,d and IVa,b).

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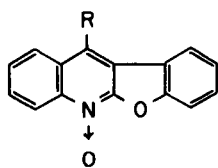
A number of studies concerning furoquinoline derivatives have been carried out because of the interest in furoquinoline alkaloids (1). In the course of our studies on polycyclic heteroaromatics (2), we studied the synthesis of analogous benzofuroquinolines to investigate their chemical reactivities and also to test their activities as mutagens, carcinogens, and further as anti-tumor substances. In this paper, we will report the synthesis of benzofuro[2,3-*b*]quinoline and isomeric benzofuro[3,2-*c*]quinoline derivatives.



I a, b, c, d



II a, b, c, d



III a, b, d



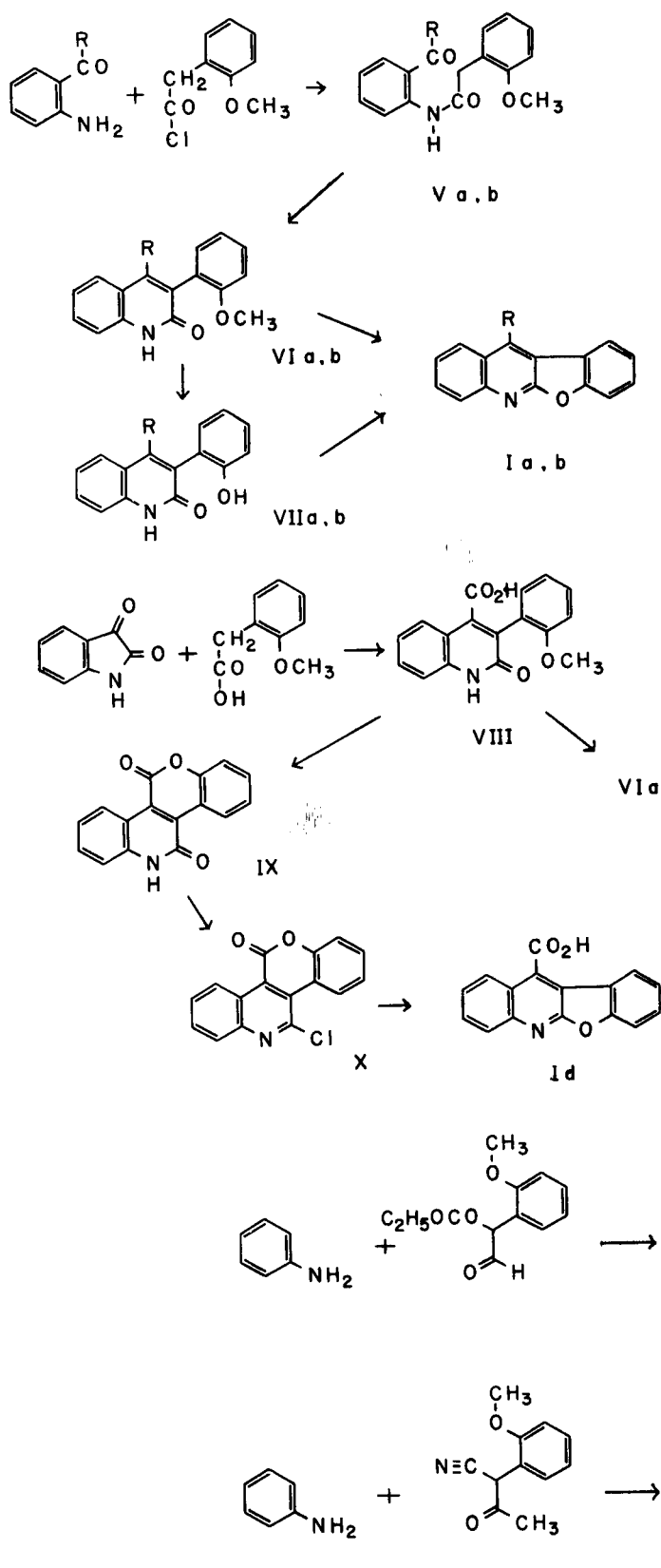
IV a, b

- a) R = H b) R = CH₃ c) R = CH=CHC₆H₅
d) R = CO₂H

It was reported by Mohanty, *et al.* (3) that benzofuro[2,3-*b*]quinoline (Ia) was synthesized by condensation of 2(3*H*)benzofuranone and *o*-nitrobenzaldehyde

followed by reductive cyclization. Analogously, the 11-hydroxy derivative of Ia was synthesized by condensation of 2(3*H*)benzofuranone and anthranilic acid. This reported method seems to be inconvenient, since the preparation of 2(3*H*)benzofuranone is difficult. Therefore, we investigated the synthesis of benzofuro[2,3-*b*]quinoline derivatives through another route.

3-(*o*-Methoxyphenyl)-1,2-dihydroquinolin-2-one (VIa) and its 4-methyl (VIb) and 4-carboxy (VIII) derivatives, the intermediates in our method, were prepared by analogous procedures (4-6) described for the preparation of quinolones without methoxy groups on the 3-phenyl group. The amide (Va), prepared from *o*-methoxyphenylacetyl chloride and *o*-aminobenzaldehyde, was cyclized by the action of sodium hydroxide to VIa. The quinolone (VIb) was also prepared analogously from the amide (Vb); the acid (VIII) was prepared by the condensation of isatin and *o*-methoxyphenylacetic acid. The decarboxylation of VIII with copper in quinoline gave VIa. Demethyl-cyclization of the quinolones (VIa,b) by heating with pyridine hydrochloride furnished benzofuro[2,3-*b*]quinoline (Ia) and its 11-methyl derivative (Ib), respectively. Mild demethylation of VIa,b afforded 3-(*o*-hydroxyphenyl)-1,2-dihydroquinolin-2-ones (VIIa,b), which were easily converted to Ia,b by heating vigorously with pyridine hydrochloride. Demethylation of the acid (VIII) afforded 11,12-dihydro-5*H*[1]benzopyrano[4,3-*c*]quinoline-5,11-dione (IX), the lactone structure of which was indicated from its ir spectrum, having two ν co bands at 1735 (lactone) and 1655 cm⁻¹ (amide). The lactone (IX) was converted by chlorination with phosphorus pentachloride to 11-chloro-5*H*[1]benzopyrano[4,3-*c*]quinolin-5-one (X), the structure of which was suggested by the ir spectrum,



having a ν co lactone band at 1725 cm^{-1} , lacking that of a lactam. The chloro lactone (**X**) was treated with potassium hydroxide in dimethyl sulfoxide to give benzofuro[2,3-*b*]quinoline-11-carboxylic acid (**Id**).

In order to synthesize the isomeric benzofuro[3,2-*c*]quinoline derivatives, the intermediate compounds (**XIa, b**), were prepared analogously to the quinolones without methoxy groups on the 3-phenyl group. The quinolone (**XIa**) was prepared by the condensation of aniline and ethyl α -formyl-*o*-methoxyphenylacetate in an analogous manner to the method of Elderfield, *et al.* (7), while the quinolone (**XIb**) was prepared by the condensation of aniline and α -acetyl-*o*-methoxyphenylacetoneitrile (8) in an analogous manner to the method of Hauser, *et al.* (9). However, the preparations of **XIa** from aniline and α -formyl-*o*-methoxyphenylacetoneitrile and of **XIb** from aniline and ethyl α -acetyl-*o*-methoxyphenylacetate were unsuccessful. Demethyl-cyclization of the quinolones (**XIa, b**) with pyridine hydrochloride afforded benzofuro[3,2-*c*]quinoline (**IIa**) and its 6-methyl derivative (**IIb**).

The condensation of angular methylbenzofuroquinoline (**IIb**) with benzaldehyde was effected by heating in acetic anhydride. The condensation of linear **Ib** with benzaldehyde was unsuccessful under the same conditions, but the condensation to 11-styrylbenzofuro[2,3-*b*]quinoline (**Ic**) was effected by heating it with excess benzaldehyde in the presence of anhydrous zinc chloride. Both the styryl derivatives (**Ic** and **IId**) were oxidized to the corresponding carboxylic acids (**Id** and **IId**).

Benzofuroquinolines (**Ia, b** and **IIa, b**) thus obtained were converted to the corresponding *N*-oxides (**IIIa, b** and **IVa, b**) by oxidation with peracetic acid. The oxidation of the linear carboxylic acid (**Id**) to the *N*-oxide (**IIId**) required trifluoroperacetic acid, the oxidation of the angular carboxylic acid (**IIId**) was unsuccessful under these conditions.

It seemed that quinolone with a hydroxy group in the 2- or 4-position could possess two structures, namely, quinolone and quinolinol. However, Ewing, *et al.* (10), and Mason (11) suggested quinolone structures for these compounds because of their ir spectra. The compounds VIa,b, VIIa,b VIII, IX, and XIa,b seem to possess principally the quinolone structures since they have a ν co band around 1650 cm^{-1} in their ir spectra. Moreover, in the nmr spectra of XIa,b measured in DMSO- d_6 , doublet signals at about 8.5 ppm may be assigned to the 5-H protons, which are deshielded by neighbouring carbonyl groups, suggest that XIa,b have 4-quinolone structures in DMSO.

It was observed that the melting points of the linear benzofuro[2,3-*b*]quinolines (Ia,b,c,d) were generally higher than those of the angular benzofuro[3,2-*c*]quinolines (IIa,b,c,d). Particularly, the melting point of the linear carboxylic acid (Id) was much higher than that of the angular carboxylic acid (IId). In the uv spectra, the linear benzofuro[2,3-*b*]quinolines (Ia,b,d) have strong bands around 220, 260, and 320 nm; those of their *N*-oxides (IIIa,b,d) are red-shifted by about 5 nm (Table 2). However, the isomeric angular benzofuro[3,2-*c*]quinolines (IIa,b,d) have absorption curves which are somewhat different from those of the linear isomers having strong bands around 290 nm (Table 3), and the whole shape of absorption curves of IIa,b,d is comparable to that of chrysene. The *N*-oxides (IVa,b) have strong bands red-shifted by about 15 nm (Table 3).

EXPERIMENTAL

All melting points were determined on a micro melting point apparatus (Yanagimoto) or in a salt bath, and are uncorrected. Ir spectra were taken on a Hitachi EPI-S2 spectrophotometer as potassium bromide disk; uv spectra were taken on a Hitachi 124 spectrophotometer in ethanol (95%) solution. Mass spectra were recorded on a JEOL LMS-OISG-2 mass spectrometer; nmr spectra were recorded on a JEOL JNM-MH-60 nmr spectrometer. The elemental analyses and uv data are summarized in Tables 1-4.

3-(*o*-Methoxyphenyl)-1,2-dihydroquinolin-2-one (VIa).

According to the procedure of Bishler, *et al.* (4), the preparation of VIa was effected by the reaction of the amide (Va) with sodium hydroxide. The crude amide (Va) was obtained as a yellow oil; ir ν max: 3250 (NH), 2850 and 2775 (CHO), 1690 (aldehyde), 1670 cm^{-1} (amide). It was used for the next step without further purification. The quinolone (VIa) was recrystallized from ethanol to give yellow needles, m.p. $240\text{--}242^\circ$; ir ν max: 1690 cm^{-1} (amide). The quinolone (VIa) was obtained in 15% yield from *o*-methoxyphenylacetic acid.

3-(*o*-Methoxyphenyl)-4-methyl-1,2-dihydroquinolin-2-one (VIb).

According to the procedure of Camps (5), the preparation of VIb was effected by the reaction of amide (Vb) with sodium hydroxide. The amide (Vb) was recrystallized from ethanol to give a dark yellow powder (31% yield), m.p. $96\text{--}97^\circ$; ir ν max: 1690 cm^{-1} (ketone), 1660 cm^{-1} (amide).

3-(*o*-Methoxyphenyl)-2-oxo-1,2-dihydroquinoline-4-carboxylic Acid (VIII).

According to the procedure of Gysae (6), the preparation of VIII was effected by the Pfitzinger reaction of isatin and *o*-methoxyphenylacetic acid with anhydrous sodium acetate. The carboxylic acid (VIII) was recrystallized from acetic acid to give a colorless powder (52% yield), m.p. $313\text{--}315^\circ$; ir ν max: $1700\text{--}1600\text{ cm}^{-1}$ (broad) (carboxylic acid and amide). The carboxylic acid (VIII) afforded the quinolone (VIa) in 55% yield by decarboxylation with copper in quinoline by the procedure of Hubner (12).

Table I

Elemental Analyses of the New Compounds

Compound No.	C (%)	Found H (%)	N (%)	Empirical Formula	Calcd. C (%)	Calcd. H (%)	N (%)
Ib	82.20	4.86	5.78	$C_{16}H_{11}NO$	82.38	4.75	6.01
Ic	86.07	4.47	4.16	$C_{23}H_{15}NO$	85.96	4.71	4.36
Id	73.29	3.23	5.18	$C_{16}H_9NO_3$	73.00	3.45	5.32
IIa	82.29	4.16	6.54	$C_{15}H_9NO$	82.17	4.14	6.39
IIb	82.34	4.96	5.98	$C_{16}H_{11}NO$	82.38	4.75	6.01
IIc	85.81	4.76	4.27	$C_{23}H_{15}NO$	85.96	4.71	4.36
IId	73.06	3.16	5.35	$C_{16}H_9NO_3$	73.00	3.45	5.32
IIIa	76.35	3.59	5.92	$C_{15}H_9NO_2$	76.58	3.86	5.96
IIIb	77.38	4.16	5.49	$C_{16}H_{11}NO_2$	77.09	4.45	5.62
IIIc	68.81	3.24	4.80	$C_{16}H_9NO_4$	68.82	3.25	5.01
IVa	76.41	3.90	6.02	$C_{15}H_9NO_2$	76.58	3.86	5.96
IVb	76.81	4.61	5.88	$C_{16}H_{11}NO_2$	77.09	4.45	5.62
Vb	71.91	5.92	4.82	$C_{17}H_{17}NO_2$	72.06	6.05	4.94
VIa	76.52	5.30	5.63	$C_{16}H_{13}NO_2$	76.47	5.22	5.57
VIb	76.93	5.45	5.13	$C_{17}H_{15}NO_2$	76.96	5.70	5.28
VIIa	76.06	4.47	6.01	$C_{15}H_{11}NO_2$	75.93	4.67	5.90
VIIb	76.63	5.26	5.41	$C_{16}H_{13}NO_2$	76.47	5.22	5.57
VIII	69.17	4.55	4.74	$C_{17}H_{13}NO_4$	69.14	4.44	4.74
IX	72.76	3.61	5.61	$C_{16}H_9NO_3$	73.00	3.45	5.32
X	68.06	2.90	4.70	$C_{16}H_9ClNO_2$	68.22	2.86	4.97
XIa	76.44	5.46	5.71	$C_{16}H_{13}NO_2$	76.47	5.22	5.57
XIb	76.73	5.55	5.02	$C_{17}H_{15}NO_2$	76.96	5.70	5.28

3-(*o*-Methoxyphenyl)-1,4-dihydroquinolin-4-one (XIa).

According to the procedure of Elderfield, *et al.* (7), the preparation of XIa was effected by the condensation of aniline and ethyl α -formyl-*o*-methoxyphenylacetate in diphenyl ether. The quinolone (XIa) was recrystallized from ethanol to give a colorless powder (30% yield), m.p. 242-243°; ν max: 1620 cm^{-1} (cross conjugated ketone); nmr (DMSO- d_6): δ 8.4 (5-H, d, J = 9 Hz), 8.1 (2-H, s), 3.8 ppm (CH_3O , s).

3-(*o*-Methoxyphenyl)-2-methyl-1,4-dihydroquinolin-4-one (XIb).

According to the procedure of Hauser, *et al.* (9), the preparation of XIb was effected by the condensation of aniline and α -acetyl-*o*-methoxyphenylacetoneitrile (8) with polyphosphoric acid ($n = 2.5$). The quinolone (XIb) was recrystallized from acetic acid to give a colorless powder (45% yield), m.p. 301-302.5°; ν max: 1640 cm^{-1} (cross conjugated ketone); nmr (DMSO- d_6): δ 8.5 (5-H, d, J = 9 Hz), 3.8 (CH_3O , s), 2.2 ppm (2- CH_3 , s).

Demethyl-Cyclization of the Quinolones (VIa,b and XIa,b) to the Benzofuroquinolines (Ia,b and IIa,b).

A mixture of the quinolone (VIa,b or XIa,b) and a tenfold excess of pyridine hydrochloride was refluxed vigorously for 1.5 hours. After cooling, the reaction mixture was treated with water, and made basic with aqueous sodium bicarbonate solution. The precipitates formed were collected and recrystallized. Benzofuro[2,3-*b*]quinoline (Ia), yield 64%, had m.p. 188-189° (brown needles from ethanol) (lit. m.p. 108°). The reported melting point seems to be too low perhaps because of impurities in the sample, considering its reported elemental analysis (3); ms: m/e 219

Table 2

The Uv Spectral Data of Linear Benzofuroquinolines and their *N*-Oxides (a)

Compound No.	λ max (log ϵ) (nm)
Ia	219 (4.52), 249 sh (4.51), 257 (4.81), 276 (3.55), 318 (4.24), 330 sh (4.16)
Ib	222 (4.56), 250 sh (4.61), 257 (4.74), 279 (3.70), 292 sh (3.76), 310 sh (4.13), 324 (4.42), 337 (4.20)
Ic	205 (4.64), 223.5 (4.68), 260.5 (4.69), 340 (4.35)
Id	221 (4.60), 250 sh (4.60), 258 (4.76), 322 (4.31), 334 sh (4.26)
IIIa	224 (4.34), 250 sh (4.34), 262 (4.68), 281 sh (4.22), 320 (3.96), 335 (4.15), 351 sh (3.93), 364 (3.85)
IIIb	224 (4.34), 251 sh (4.36), 263 (4.75), 281 sh (4.32), 323 sh (3.93), 338 (4.11), 350 sh (3.92), 366.5 (3.90)
IIIc	223 (4.34), 254 sh (4.47), 264 (4.73), 281 sh (4.32), 323 sh (3.93)

(a) Sh = shoulder.

Table 3

The Uv Spectral Data of Angular Benzofuroquinolines and their *N*-Oxides (a)

Compound No.	λ max (log ϵ) (nm)
IIa	212 (4.42), 228 (4.42), 248 (4.65), 256 (4.83), 282 (4.24), 290 (4.26), 297 sh (4.17), 317 (3.32), 333 (3.07)
IIb	213 (4.36), 229 (4.44), 247 (4.75), 256 (4.87), 281 (4.24), 290 (4.26), 315 (3.35), 329 (3.06)
IIc	207 (4.88), 226 (4.89), 260 (4.98), 289 (4.92), 330 sh (4.29)
IId	212 (4.26), 228 (4.23), 250 sh (4.54), 258.5 (4.68), 282 (4.16), 292 (4.12)
IVa	214 (4.37), 236 (4.50), 248 sh (4.37), 258 sh (4.34), 272 (4.50), 287 sh (4.36), 295 (4.34), 328 (3.87), 342 (3.89)
IVb	215 (4.36), 237 sh (4.44), 248 (4.39), 256 sh (4.43), 271 (4.62), 285 sh (4.33), 294 (4.36), 328 (3.79), 339 (3.78), 355 sh (3.65)

(a) Sh = shoulder.

(M⁺).

Anal. Calcd. for $\text{C}_{15}\text{H}_9\text{NO}$: C, 82.17; H, 4.14; N, 6.39. Found: C, 82.27; H, 3.99; N, 6.45.

11-Methylbenzofuro[2,3-*b*]quinoline (Ib), yield 71% had m.p. 189-190.5° (a brown powder from ethanol); ms: m/e 233 (M⁺). Benzofuro[3,2-*c*]quinoline (IIa), yield 17%, had m.p. 135-138° (a colorless powder from ethanol); ms: m/e 219 (M⁺). 6-Methylbenzofuro[3,2-*c*]quinoline (IIb), yield 30%, had m.p. 132-133° (a colorless powder from cyclohexane); ms: m/e 233 (M⁺).

Demethylation of the Quinolones (VIa,b).

The quinolone (VIa,b) was heated at 230-250° with a tenfold excess of pyridine hydrochloride for 1 hour. After cooling, the reaction mixture was treated with water. The precipitates were collected and dissolved in aqueous sodium hydroxide solution. Acidification of the filtered alkaline solution afforded the demethylated quinolone. 3-(*o*-Hydroxyphenyl)-1,2-dihydroquinolin-2-one (VIIa), yield 49%, had m.p. 284-286° (a colorless powder from ethanol); ν max: 3400 (OH), 1645 cm^{-1} (amide). 3-(*o*-Hydroxyphenyl)-4-methyl-1,2-dihydroquinolin-2-one (VIIb), yield 14%, had m.p. 280-281° (a colorless powder from ethanol); ν max: 3250 (OH), 1635 cm^{-1} (amide). The quinolone (VIIa) was vigorously refluxed with a tenfold excess of pyridine hydrochloride for 3 hours and afforded Ia in 35% yield.

Demethyl-Cyclization of VIII to the Lactone (IX).

A mixture of the acid (VIII) (0.70 g.) and pyridine hydrochloride (7.0 g.) was refluxed for 1 hour, and the cooled reaction mixture was treated with water. The precipitates formed were collected and recrystallized from acetic acid to give 0.50 g. (80%) of IX as a colorless powder, m.p. 364-365°, ν max: 1735 (lactone), 1655 cm^{-1} (amide).

Chlorination of IX to the Chloro Lactone (X).

A mixture of IX (2.3 g.), phosphoryl chloride (10 ml.), and phosphorus pentachloride (2.6 g.) was vigorously refluxed for 2 hours. After cooling the reaction mixture was treated with water. The white precipitates formed were collected and recrystallized from ethanol to give 1.6 g. (64%) of X as a colorless powder, m.p. 237-237°; ν max: 1725 cm^{-1} (lactone).

Conversion of X into the Carboxylic Acid (Id).

A mixture of X (1.6 g.), aqueous potassium hydroxide solution (containing 1.2 g. potassium hydroxide in 1.2 ml. of water), and dimethyl sulfoxide (58 ml.) was refluxed for 6 hours. The cooled reaction mixture was treated with water and filtered. The filtrate was acidified to pH 4 with dilute hydrochloric acid. The white precipitates were collected and recrystallized from ethanol to give 0.68 g. (47%) of Id as a colorless powder, m.p. 326° dec.; ν max: 1705 cm^{-1} (carboxylic acid); ms: m/e 263 (M⁺).

Table 4

The Uv Spectral Data of Quinolones

Compound No.	λ max (log ϵ) (nm)
VIa	222 (4.61), 267 sh (3.94), 279.5 (3.98), 332 (4.04), 345 sh (3.96)
VIb	223 (4.60), 275 (3.95), 317 sh (3.89), 328 (3.98), 342 sh (3.83)
VIIa	220 (4.50), 267 (3.61), 290.5 (3.65), 340 (3.93)
VIIb	207 sh (4.15), 223 (4.27), 248 sh (4.06), 277 (3.93), 317 sh (3.91), 329 (4.00), 341 sh (3.86)
VIII	222 (4.54), 278 (3.90), 316 (3.82), 326 (3.89), 338 sh (3.77)
XIa	212 (4.57), 249 (4.24), 266 sh (4.05), 283 (3.89), 310 sh (3.86), 322.5 (3.94), 335 (3.94)
XIb	213 (4.58), 240 (4.37), 247 (4.38), 281 (3.84), 320 (4.04), 332 (4.04)

(a) Sh = shoulder.

Condensation of Ib with Benzaldehyde.

A mixture of Ib (2.4 g.), benzaldehyde (30 ml.), and anhydrous zinc chloride (0.2 g.) was refluxed for 18 hours. Excess benzaldehyde was distilled off by steam distillation, and 20% aqueous sodium hydroxide solution (100 ml.) was added to the cooled reaction mixture. The precipitates formed were collected and recrystallized from ligroin to give 2.4 g. (71%) of Ic as a colorless powder, m.p. 176-180°; ms: m/e 321 (M⁺).

Condensation of IIb with Benzaldehyde.

A mixture of IIb (1.8 g.), benzaldehyde (3.0 g.), and acetic anhydride (2.0 g.) was refluxed for 3 hours. The cooled reaction mixture was treated with water. The precipitates formed were collected and recrystallized from benzene to give 1.5 g. (61%) of IIc as a colorless powder, m.p. 177-179°; ms: m/e 321 (M⁺).

Oxidation of the Styrylbenzofuroquinolines (Ic and IIc) to the Carboxylic Acids (Id and IID).

To a solution of the styrylbenzofuroquinolines (Ic or IIc) (5.3 mmoles) in pyridine (10 ml.), a solution of potassium permanganate (2.7 g.) in water (20 ml.) was added, and the mixture was heated at about 50° for 2 hours with stirring. The cooled reaction mixture was filtered, and the filtrate was treated with sodium bisulfite to decompose the manganese dioxide and acidified to pH 4 with dilute hydrochloric acid. The precipitates formed were collected and recrystallized. Benzofuro[2,3-*b*]quinoline-11-carboxylic acid (Id), yield 22%, had m.p. 326° dec. (a colorless powder from ethanol). Benzofuro[3,2-*c*]quinoline-6-carboxylic acid (IID), yield 58%, had m.p. 171-172° (a colorless powder from benzene); ν max: 1705 and 1675 cm⁻¹ (carboxylic acid); ms: m/e 263 (M⁺).

N-Oxidation of the Benzofuroquinolines (Ia,b and IIa,b).

To a solution of the benzofuroquinoline (Ia,b or IIa,b) (5.5 mmoles) in acetic acid (10 ml.), 30% hydrogen peroxide aqueous solution (1.0 ml.) was added, and the mixture was heated at 60-70° for 3 hours. The heating was continued at the same temperature for 6 additional hours, with the addition of more 30% hydrogen peroxide aqueous solution (0.5 ml.). The reaction mixture was concentrated under reduced pressure and neutralized with aqueous sodium carbonate solution. The precipitates formed were collected and recrystallized. Compound IIIa, yield 23%,

had m.p. 238-239° (a yellow powder from ethanol); ms: m/e 235 (M⁺). Compound IIIb, yield 50%, had m.p. 220-222° (a pale yellow powder from benzene); ms: m/e 249 (M⁺). Compound IVa, yield 22%, had m.p. 206-208° (a pale yellow powder from benzene); ms: m/e 235 (M⁺). Compound IVb, yield 15%, had m.p. 205-207° (a yellow powder from benzene); ms: m/e 249 (M⁺).

N-Oxidation of the Carboxylic Acid (Id).

Analogously, a solution of Id (0.57 g.) in trifluoroacetic acid (3 ml.) was treated with 30% hydrogen peroxide aqueous solution (0.5 ml. and an additional 0.5 ml.) to give the crude *N*-oxide, which was recrystallized from methanol to give 0.22 g. (37%) of IIId as a pale yellow powder, m.p. 285-287°; ms: m/e 279 (M⁺).

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