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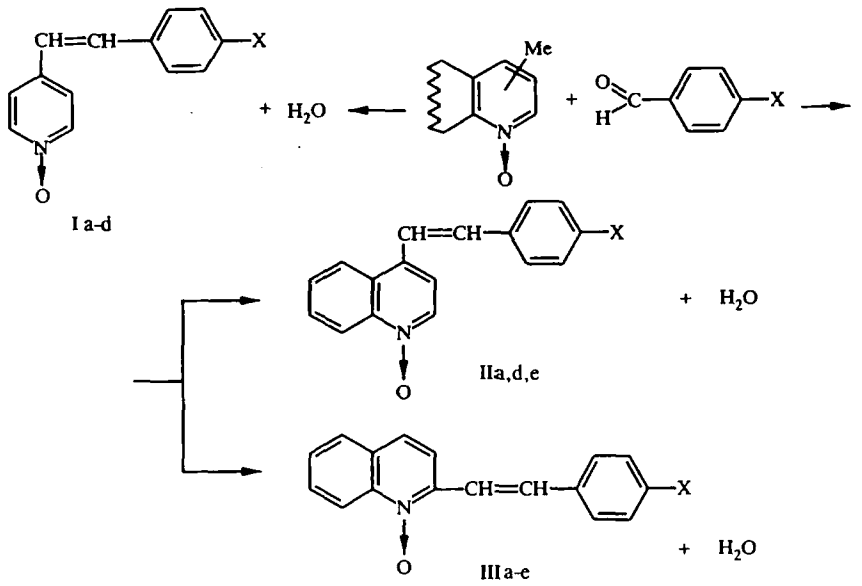
INTRAMOLECULAR CHARGE TRANSFER PROCESSES IN A SERIES OF STYRYL DERIVATIVES OF PYRIDINE AND QUINOLINE N-OXIDES

V. P. Andreev, E. G. Batotsyrenova, A. V. Ryzhakov, and L. L. Rodina

Intramolecular charge transfer processes in a series of the trans-*isomers of styryl derivatives of pyridines and quinolines, their hydrochlorides and molecular complexes with BF_3 have been studied by electronic and IR spectroscopy. Electron donor groups involved in direct resonance conjugation with the N-oxide group intensify these processes, while electron acceptor nitro groups somewhat weaken them. Protons and BF_3 coordinate to the oxygen of the N→O group to form strong 1:1 complexes, except in the cases of 4-(4-dimethylaminostyryl)quinoline and pyridine N-oxides, in which a second acceptor molecule adds to the amino group.*

Heteroaromatic N-oxides are unique in that the N-oxide group can act as both a donor and acceptor of electron density, depending on the structure of the molecule. We have previously studied intramolecular charge transfer processes by electronic and IR spectroscopy in functionally substituted pyridine and quinoline N-oxides in which the substituents were in direct resonance conjugation with the N-oxide group [1]. It has been shown [2, 3] that when these compounds form complexes with the typical ν-acceptor BF_3 and also on salt formation with HCl, redistribution of electron density in the N-oxide molecules is considerably affected.



I, III a X = H, b OMe, c 2,4-OMe, d NMe_2, e NO_2

Petrozavodsk State University, Petrozavodsk 185640, Russia. Translated from Khimiya Geterotsiklicheskikh Soedinenii, No. 8, 1093-1102, August, 1998. Original paper submitted September 10, 1997.

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TABLE 1. Characteristics of Styryl Derivatives of N-Oxides, Their Molecular Complexes with BF_3, and Their Hydrochlorides

N-Oxide	Substituent	Acceptor	Color	mp, °C	Yield, %
Ia	H	BF_3	Beige	75...77	83
Ib	OCH_3	BF_3	Yellow	216...217	75
Ic	2,4-OCH_3	—	Yellow	129...131	70
Ic	2,4-OCH_3	BF_3	Mustard	145...147	83
Ic	2,4-OCH_3	HCl	Brick red	182...184	94
Id	$N(CH_3)_2$	$2BF_3$	Dark yellow Oily substance	—	90
IIa	H	BF_3	Pale yellow	201...203	65
IId	$N(CH_3)_2$	—	Red orange	182...183	55
IId	$N(CH_3)_2$	$2BF_3$	Yellow brown Oily substance	—	92
IIe	NO_2	—	Mustard	200...202	58
IIe	NO_2	BF_3	Yellow orange	188...190	60
IIe	NO_2	HCl	Bright yellow	220...221	90
IIIa	H	BF_3	Bright yellow	192...194	78
IIIa	H	HCl	Yellow	184...186	93
IIIb	OCH_3	BF_3	Light orange	210...211	85
IIIb	OCH_3	HCl	Orange	206...208	92
IIIc	2,4-OCH_3	BF_3	Orange	203...205	58
IIId	$N(CH_3)_2$	BF_3	Dark violet	199...200	98
IIId	$N(CH_3)_2$	HCl	Blackish violet	198...200	96
IIIe	NO_2	BF_3	Orange	195...196	52

In a continuation of these investigations we have studied the processes of intramolecular electron transfer in styryl derivatives of pyridine N-oxides Ia-d and quinoline N-oxides IIa, d, e and IIIa-e which contain a more extended conjugated system which is considerably more sensitive to internal and external influences. Special attention was paid to the effect of complex formation of these N-oxides with BF_3 and HCl on the ability of various types of substituent in the benzene ring of the styryl unit to conjugate intramolecularly with the N→O group.

The styryl derivatives Ia-d, IIa, d, e and IIIa-e were prepared by a new method developed by us which includes the condensation the N-oxides of 4-methylpyridine, 2- and 4-methylquinoline with aromatic aldehydes in the presence of KOH in ethanol [4].

As expected from the mechanism of nucleophilic addition [5], the reaction occurs readily with aldehydes containing electron acceptor groups. For example, with 4-nitrobenzaldehyde condensation occurs at room temperature whereas in other cases the mixture must be heated at 60°C. The characteristics of the synthesized styryl derivatives of N-oxides are given in Tables 1 and 2.

A bathochromic shift of the maxima of the lone wavelength bands and a more complicated structure, in comparison with the initial N-oxides containing a methyl group in the heterocyclic ring, was observed in the electronic spectra of all the styryl derivatives. In compounds containing donor groups entering into direct conjugation with the N-oxide group, a bathochromic shift, accompanied by a simultaneous increase in the extinction coefficients, occurred in comparison with the unsubstituted analogs. The long wavelength shift in the electronic spectra is caused by intramolecular charge transfer [1], the extent of which depends on the electron donor power of the substituent which reaches a maximum with the dimethylamino group.

The IR spectra of the styryl derivatives of pyridine and quinoline N-oxides contain absorption bands in the 1310-1255 cm^{-1} range, characteristic for the N→O group. The strong absorption bands at 1520 and 1340 cm^{-1} for the N-oxides IIe and IIIe can be assigned to the antisymmetric and symmetric stretches of the N−O of the nitro group according to the literature [6].

The ^1H NMR spectra also confirm the structures of the new compounds. The presence of signals for protons attached to the double bond in the regions 7.72-7.86 and 7.02-7.39 ppm (two doublets, J = 16 Hz) in the styryl derivatives of the quinoline and pyridine N-oxides respectively indicates that they are *trans* isomers [7, 8].

Complexes of the N-oxides with BF_3 were obtained by reaction with boron trifluoride etherate in chloroform and the hydrochlorides by reaction with concentrated hydrochloric acid or with HCl gas. Their properties are cited in Tables 1 and 2.

TABLE 2. Electronic and IR Spectra of N-Oxides and Products of Their Reactions with BF_3 and HCl

Compound	X	Acceptor	Solvent	Electronic spectra, λ_{max} (nm), log ε		IR spectra	
				field 1	field 2	N → O↔N⁺ — O⁻	O—B; B—F
1	2	3	4	5	6	7	8
Ia	H	—	Ethanol–chloroform	200(4,05); 205 sh; 230(4,08); 281(3,73)	337(4,52); 349(4,72); 364 sh	1265 s	
		BF_3	Chloroform	243(2,69); 348(3,12)		—	1110 br.s; 940
Ib	OCH_3	—	Ethanol–chloroform	211(3,92); 227(3,95); 237(3,92)	356(4,52); 363(4,81); 380 sh	1310 w; 1260 s	
		BF_3	Chloroform	256(3,15)	382(3,40)	1295 w; 1260 s	1155; 955 av.; 1095 s; 935 av.
Ic	2,4-OCH_3	—	Chloroform	265 plateau 3,70)	370(4,22)	1320 av; 1285	
		BF_3	Chloroform	260(3,84)	402(4,21)	1310 av.	1140 br.s; 970 av; 1040 s; 940 av.
		HCl	Chloroform	261(3,80)	403(4,13)	1300 av.	
Id	$N(CH_3)_2$	—	Ethanol–chloroform	207(4,28); 223 sh; 243 sh; 272 sh; 247 (3,89)	303 sh; 346(4,16); 420(4,37); 340(4,22); 408(4,31)	1240 av.	
		$2BF_3$	Chloroform	244(3,93)	338(4,13)	1240...1030 br.s	
		BF_3	Chloroform	244	340; 480		
I		—	Chloroform	279(4,31)		1255 av.	
		BF_3	Chloroform	262(3,31); 268(3,24)		1280	1160; 970 br.s
IIa	H	—	Chloroform	258(3,99); 292(3,80)	362(3,96); 385(3,99)		
		BF_3	Chloroform	243(3,13)	335 sh; 380(2,97)	1290	1130; 925; 1080
IId	$N(CH_3)_2$	—	Chloroform	252(4,28); 270 sh	342(4,05); 442(4,25)		
		$2BF_3$	Chloroform	247(4,45	330(4,06)	1210...1020 br.s	
		BF_3	Chloroform	247	335; 518		

TABLE 2 (continued)

1	2	3	4	5	6	7	8
IIe	NO_2	—	Chloroform	252	325 sh; 345; 355	1520 s; 1340 w.	
		BF_3	Chloroform	252; 266	308; 319; 335 sh; 361 sh	1520 s; 1345 s	1145; 900 s; 1095 av.
		HCl	Chloroform	252; 270 sh	325 sh; 342; 355 sh		
II		—	Chloroform		328 sh; 342(3,88); 355 sh	1300 w.	1155; 920 s
		BF_3	Chloroform		309 sh; 320(3,76); 333 sh; 360 sh		1100 s
IIIa	H	—	HCl (conc)	212; 239	306; 322; 382	1360 s; 1280 av.	
			HCl (conc)—ethanol (1:20)	218; 238 sh	306; 322; 390		
			Ethanol	202(4,26); 208 sh; 237(4,23); 247 sh; 300(4,54) 308(4,55) 350 sh (4,35); 360 (4,16)			
			Chloroform	243(4,26); 302 sh; 311(4,62); 352 sh (4,28); 365 (4,32)		1360 av.; 1300 av.	1150 s; 940 s; 1090 s; 920 v. s
		BF_3	Chloroform	244(4,30); 293 sh; 302(4,28) 330 sh (4,62); 375 br.s. (4,46)			
		HCl	Chloroform	255 sh	309(4,38); 348 sh; 377(4,31); 394 sh		
IIIb	OCH_3	—	HCl (conc)	213; 244; 259 sh	316; 415	1360 s; 1290 av.	
			HCl (conc)—ethanol (1:20)	218; 242 sh; 260	317; 420		
			Ethanol	202(4,09); 222(4,04); 247(4,14); 311 sh (4,31); 320(4,34); 377(4,35)			
			Chloroform	242(4,27); 248 sh	316 sh; 326(4,56); 379(4,41)		
		BF_3	Chloroform	257(4,31); 292 sh	314(4,06); 326 sh; 408(4,53)	1355 w	1160; 920; 1140; 1100
		HCl	Chloroform	256(4,18)	325(4,34); 417(4,42)		
IIIc	2,4-OCH_3	—	Ethanol	203(4,29); 238(4,18); 262 sh (4,01); 306(4,14); 322(4,14); 391(4,26)		1305 s; 1280 s; 1250 av.	
			Chloroform				
		BF_3	Chloroform	258(4,14)	312(3,99); 428(4,42)	1300 s	1160s; 920; 1130 s
		HCl	Chloroform	253(4,15)	312(3,80); 432(4,23)		

TABLE 2 (continued)

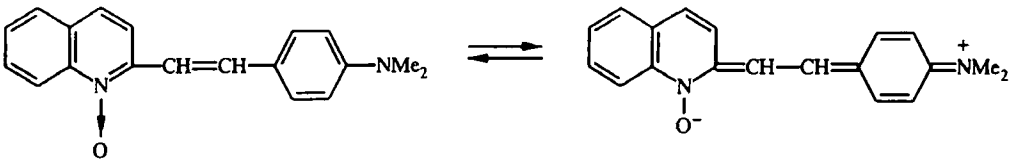
1	2	3	4	5	6	7	8
IIId	$N(CH_3)_2$	—	HCl (conc.)	247 298 sh	370; 384 sh	1345 w	
			HCl (conc.)—ethanol (1 : 20)	240(4,42); 295(4,42)	366(4,42); 518(4,29)	1295 s	
			Ethanol	198(4,47); 228(4,22); 268(4,18)	308 sh; 338(4,06); 420(4,41)		
			Chloroform	240 sh; 272(4,30)	309 sh; 346(4,36); 430(4,59)		
		BF_3	Chloroform	243(4,24); 273(4,11)	332(4,01); 508(4,37)	1360 av	1070 s; 920 av
		$2BF_3$	Chloroform	243(4,14); 290 sh; 298(4,16)	359(4,30); 377 sh		
		HCl	Chloroform	240 sh; 242 sh; 273(4,17); 435(4,10); 520(4,30)	309(4,15); 340(4,10); 363 sh;	1350 av; 1300 av	
IIIe	NO_2	—	HCl (conc.)	211; 237; 280	312; 321	1520 s; 1340 s	
			HCl (conc.)—ethanol (1 : 20)	216; 236; 273	310; 321	1235	
			Ethanol	202; 235; 270	315 sh; 330		
			Chloroform	248; 272	329; 343		
		BF_3	Chloroform	252 272	312; 324; 343 sh	1520 s	1120 vs
		HCl	Chloroform	250 268	312 sh; 325; 343 sh	1340 av; 1315 w.	
III		—	Ethanol	213 sh; 230 sh; 237(4,60)	319(3,79); 328 sh	1345; 1320 av.	
			Chloroform		330(3,83); 341(3,82)		
		BF_3	Chloroform		310(3,81); 321(3,85); 343 sh (3,13)	1330 av.	1145 v. s; 940 av; 1110 s. 915 vs 1010 w.

*As a consequence of the poor solubility of compounds IIe, IIIe, their hydrochlorides and their complexes with BF_3 in chloroform and aqueous solution, the electronic spectra of saturated solutions were recorded and the extinction coefficients were not determined in these cases.

*2Compounds Id·BF_3, IId·BF_3 and IIId·$2BF_3$ were not isolated in the pure state. The electronic spectrum of IIId·$2BF_3$ was recorded after adding a manyfold excess of BF_3·etherate to a solution of IIId in chloroform. The electronic spectra of Id·BF_3 and IId·BF_3 were recorded after stepwise addition of a very dilute solution of BF_3·etherate in chloroform to solutions of Id and IId in chloroform.

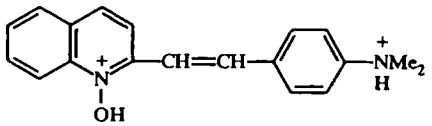
We observed that formation of molecular complexes with BF_3 and HCl caused the electronic spectra to become more complex than the spectra of the initial N-oxides and that there was a bathochromic shift which depended on the nature of the functional group in the styryl unit. As with the N-oxides, the long wavelength shift was at a maximum for the dimethylamino group which is a strong electron donor in direct resonance conjugation with the N→O group.

For example the following resonance structures are possible for the N-oxide IIId via intramolecular charge transfer:



The "quinonoid" structures should be further stabilized by formation of covalent bonds the N→O group which explains the intensified bathochromic shift of the long wavelength bands in the complexes of compounds Ia-c, II, IIIa-d with BF_3 and HCl. The electronic spectra of their hydrochlorides in which protonation occurs at the oxygen atom of the N→O group are similar to those of the complexes with BF_3 which indicates that the BF_3 is also coordinates to the same center.

It is interesting that there is a very intense shortwave shift (λ 384 nm) in the electronic spectra of N-oxide IIId and its hydrochloride in concentrated HCl, whereas in dilute ethanolic hydrochloric acid solution an absorption band appears at 518 nm which is present in the electronic spectrum of the hydrochloride IIId·HCl. This can be explained by the existence of compound IIId in concentrated HCl as the dication IV in which there is no direct resonance conjugation:

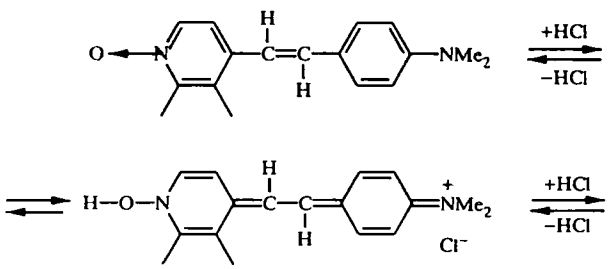


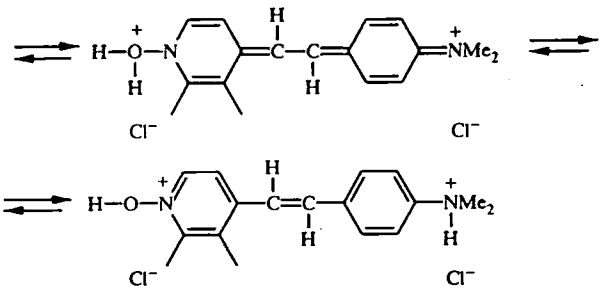
IV

Dilution of the solution with ethanol causes removal of the proton from the nitrogen atom, shifts the electron density into the π-system by conjugation with the dimethylamino group and causes the appearance of a long wavelength absorption band, explicable by the intramolecular transfer of electron density. Thus even in 62% H_2SO_4 4-aminopyridine N-oxide is protonated at the N→O oxygen atom but not at the amino group nitrogen atom, whereas in 95% H_2SO_4 it exists as the dication [9].

Reaction of complex IIId·BF_3 in chloroform solution with excess BF_3 also led to a hypsochromic shift of the long wavelength absorption band from 508 to 359 nm and decolorization of the solution, but subsequent gradual addition of 20% trimethylamine solution gave rise to reappearance of absorption bands characteristic of the complex IIId·BF_3 and then of the N-oxide IIId.

Unlike the other styryl derivatives, the N-oxides Id and IId reacted with excess BF_3 or HCl extremely easily to form stable 1:2 complexes. Evidently such behavior of these *trans*-isomers arises from the especially electron distribution in the adducts on complex formation with simultaneous participation of the two diametrically opposite N→O and $N(CH_3)_2$ groups which are similar in basicity. The interaction with HCl in this case can be described as follows:





The other styryl derivatives of the N-oxides without a strongly basic second donor center (X = H, OCH_3, NO_2) forms adducts with only one molecule of BF_3 or HCl and the electronic spectra of the complexes change only slightly or not at all in the presence of a large excess of the acceptor (Table 2).

IR spectroscopy also confirmed the formation of *n,π*-complexes between the N-oxides Ia-d, IIa, d, e, and IIIa-e and BF_3. In these spectra the absorption bands at about 1300 cm^{-1}, characteristic of the N→O group, are either absent or decreased in intensity while new bands appear in the 1160-900 cm^{-1} region. According to literature data absorption bands for N→O (in aliphatic N-oxides), B−F, and B−O bonds occur in this region [10, 11].

The N-oxides Ie and IIe and their complexes which contain the electron accepting nitro group deserve special attention. In their electronic spectra the long wavelength absorption band appears at higher frequency than in the spectra of 4-nitroquinoline N-oxide (λ 383 nm) and their unsubstituted styryl analogs IIA and IIIa (Table 2) which evidently indicates that electron density in the N-oxides IIe and IIIe is more evenly distributed.

In distinction from the other N-oxides no major changes were observed when the nitro derivatives formed complexes with HCl and BF_3. We believe that this observation can be explained as follows.

Both the N→O and NO_2 normally possess electron acceptor properties, but the former may appear as an electron donor in some cases. When there is a short π-system between them the competition for electrons is sufficiently strong that the comparatively easily polarizable N→O group shows donor properties [9]. The dipole moment of the molecule is directed toward the nitro group [12] and on account of the presence of the quinonoid form an absorption band appears at longer wavelength in the electronic spectrum of 4-nitropyridine n-oxide (λ 330 nm, μ 0.62 D) than in the spectrum of pyridine N-oxide (λ 263, μ 3.35 D). In concentrated HCl this band underwent a hypsochromic shift (λ 247, 290 nm, shoulder) as a result of protonation of the oxygen of the N→O group and removal of the unshared pair of electrons from direct resonance conjugation with the nitro group [1].

In compounds with a sufficiently long conjugated chain including N→O and NO_2 groups, they may both show electron acceptor properties to some extent because of the easy polarizability of the π-bonds between carbon atoms. For example, introduction of a nitro group at position 4 of quinoline N-oxide leads to a considerable decrease in the bathochromic shift in the electronic spectrum in 96% ethanol (from 340 to 375 nm), and in the styryl derivatives IIe and IIIe there is the opposite effect — a short wavelength shift by 22 and 30 nm respectively. In similar compounds even complex formation with acceptors may not produce much change in the electronic spectrum or in the dipole moment of the molecule as a whole.

In fact, reaction of 4-nitroquinoline N-oxide [2], 4-(4-nitrostyryl)pyridine N-oxide [13], and styryl derivatives of the N-oxides IIe and IIIe, containing nitro groups, with BF_3 and HCl did not lead to a noticeable shift in the position of the absorption bands in the long wavelength region of their electronic spectra. Moreover, their spectra differ very little in this region from the spectrum of 4-nitroquinoline N-oxide in concentrated HCl (λ 333 nm), in which conjugation between N→O and the NO_2 group is impossible.

From a comparison of the intensities of the C=C and C≡C absorption bands in the IR spectra of styryl- and phenylethynylpyridine N-oxides it was concluded [14] that the 2- and 4-(pyridyl)N-oxide groups behaved as electron acceptors even in the presence of other electron acceptor groups.

A comparison of the pK_a of nitro derivatives of 4-*trans*-styryl- and 4-phenylethynylpyridines and their N-oxides confirmed a weak interaction of the nitro groups with the basic centers of the heterocycle — nitrogen or oxygen, respectively [15].

The N-oxides IIe and IIIe, unlike the 4-nitro N-oxides of quinoline and pyridine, react with gaseous hydrogen chloride in chloroform solution to give stable salts which indicates the considerably weaker interaction between the N→O and NO_2 groups.

Thus, we have shown that the direction of complex formation of heteroaromatic N-oxides depends on the structure of the N-oxide (the presence of donor or acceptor groups, the length of the conjugated chain, the symmetry of the molecule) and the ν acceptor (see [2] for example). The adducts formed are generally stable and have a 1:1 or 1:2 N-oxide-acceptor composition, but some rapidly undergo further reaction. For example, we have shown previously that the interaction of 4-nitroquinoline N-oxide with dry HCl in aprotic solvents leads to replacement of the nitro group by chlorine [16, 17].

EXPERIMENTAL

IR spectra of Nujol mulls were recorded on a UR-20 instrument and electronic spectra of chloroform, 96% ethanol, and concentrated HCl solutions were recorded with a Specord UV-Vis spectrophotometer. ^{1}H NMR spectra of DMSO-D_6 solutions with TMS as internal standard were recorded with a Bruker AM-500 instrument.

Styryl Derivatives of Quinoline and Pyridine N-Oxides. 2-(4-Nitrostyryl)quinoline N-Oxide (IIIe). A mixture of 2-methylquinoline N-oxide (1.59 g, 10 mmole) and 4-nitrobenzaldehyde (1.81 g, 10 mmole) in a 10% solution of KOH in absolute ethanol (10 ml) was kept at about 20°C for 3 h. The precipitate was washed with water (3 × 15 ml), ethanol (2 × 5 ml) and chloroform (2 × 5 ml), and dried in vacuum to give compound IIIe (2.34 g, 80%), mp 213-215°C.

4-(4-Nitrostyryl)quinoline N-oxide (IIe) was made analogously. Found, %: C 69.90, H 4.24, N 9.70. Calc. for $C_{17}H_{12}N_2O_3$, %: C 69.85, H 4.14, N 9.50.

4-(4-Dimethylaminostyryl)quinoline N-Oxide (IId). A mixture of 4-methylquinoline N-oxide (0.80 g, 5 mmole) and 4-dimethylaminobenzaldehyde (0.90 g, 6 mmole) in 10% KOH in absolute ethanol (5 ml) was heated at 60°C for 3 h. The reaction mixture was evaporated to dryness in vacuum, treated with water (3 × 15 ml) and the red-brown mass was triturated with a glass rod. The residue was centrifuged, dried in the air and washed with ether (2 × 5 ml) to give compound IId (0.80 g, 55%), mp 182-183°C. Found, %: C 78.60, H 6.30, N 9.80. Calc. for $C_{19}H_{18}N_2O$, %: C 78.59, H 6.25, N 9.65.

The remaining styryl derivatives were made analogously to compound IId. The melting points of compounds Ia-d, IIa, and IIIa-e agree with literature values [4, 15, 18].

Molecular Complexes of Quinoline and Pyridine N-Oxides with BF_3. Boron trifluoride etherate (0.08 ml) was added to a solution of an N-oxide (0.05 mmol) in chloroform (1.5 ml). The precipitate which formed over 2 h (in some cases a small amount of ether was added to facilitate crystallization) was centrifuged, washed with chloroform (2 × 0.5 ml) and ether (2 × 1 ml) and dried in air. The least stable complexes, from N-oxides IIe and IIIe, were not washed with ether.

Hydrochlorides of Quinoline and Pyridine N-Oxides. Concentrated HCl (0.1 ml) was added to a saturated solution of the N-oxide (0.25 mmole) in ethanol. After several minutes the hydrochloride was precipitated with ether. The precipitate was centrifuged, washed with ether (2 × 1 ml) and dried in air.

The hydrochloride IIe·HCl was obtained by passing dry HCl through a suspension of N-oxide IIe in chloroform. Found, %: C 62.33, H 4.12, N 8.55. Calc. for $C_{17}H_{13}ClN_2O_3$, %: C 61.10, H 3.99, N 8.52.

The ratio of N-oxide to HCl in the hydrochlorides was determined by titration of their aqueous solutions with aqueous 0.1 M NaOH with phenolphthalein as indicator. Since the hydrochloride of IIe N-oxide was insoluble in water, the ratio was determined by elemental analysis.

The ratio of N-oxide to BF_3 in molecular complexes was determined by a literature method [19].

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