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INDO- AND BENZINDOCYANINE DYES WITH FLUORINE-CONTAINING SUBSTITUENTS

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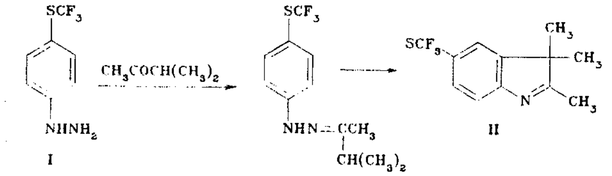
Syntheses are reported for 2,3,3-trimethyl-3H-indoles and 1,1,2-trimethyl-1H-benz[e]indoles with fluorine-containing substituents (CF_3, CF_3S, and SO_2CF_3) at C-5 and for carbocyanine, merocyanine, and styryl dye derivatives of these indoles. The fluorine-containing substituents studied produce a bathochromic effect in the styryl dyes, hypsochromic effect in the merocyanine dyes, and, as a rule, a hypsochromic effect in the carbocyanine dyes.

Indocyanine dyes with substituents containing fluorine have been the subject of only a few studies. The effect of a trifluoromethyl group has been studied only at C-4, C-5, and C-6 in the case of indocarbocyanines [1]. Indolenines containing SCF_3 and SO_2CF_3 have been noted in the patent literature [2] but the syntheses for these compounds and the spectral characteristics of their dye derivatives were not given. Benzindolenines with fluorine-containing substituents have not been reported. We have obtained indolinines containing either a trifluoromethyl or trifluoromethylsulfonyl group at C-5 and benz[e]indolenines with a CF_3, SCF_3, or SO_2CF_3 substieutnt at C-5. The spectral properties of carbocyanines, merocyanines and styryl dyes derived from these bases were studied.

Diazotization of p-trifluoromethylthioaniline [3] and reduction of the diazo salt by $SnCl_2$ gave p-trifluoromethylthiophenylhydrazine (I), which was then converted to the corresponding hydrazone. Fischer cyclization of this hydrazone gave indolenine II.



The yield of p-trifluoromethylsulfonylphenylhydrazine (III) by this method was low, the results had poor reproducibility, and the product was obtained in a mixture with the starting amine. The synthesis of hydrazine III was accomplished in 90% yield by the action of anhydrous hydrazine on p-trifluoromethylsulfonylchlorobenzene [4]. Hydrazine II was obtained previously by this same method, but in lower yield [5]. 2,3,3-Trifluoromethyl-5-trifluoromethylsulfonyl-3H-indole (IV) was prepared analogously to indolenine II.

The synthesis of 5-trifluoromethyl-, 5-trifluoromethylthio-, and 5-trifluoromethylsulfonyl derivatives of benzindolenines requires the development of methods of preparation for 3-nitronaphthalenes with these substituents at C-1. The fluorination of 3-nitro-1-naphthalenecarboxylic acid (V) [6] by highly pure sulfur tetrafluoride not containing HF leads to 1-trifluoromethyl-3-nitronaphthalene (VI) [7] in 40% yield. For this purpose, we used technical sulfur tetrafluoride without purification using a newly reported method for SF_4 fluorination in HF solution [8]. In the case of naphthalene derivatives, this method has limitations since HF is a catalyst for Friedel–Crafts reactions [9] and its presence may lead to the complete conversion of the reaction mixture into tars [7]. Acid V was fluorinated using SF_4 to tri-

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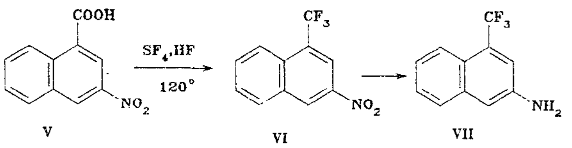
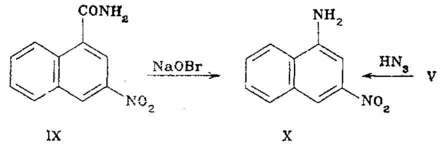
fluoromethylnaphthalene (VI) at 120°C in 90% yield. Carrying out this reaction at 80-100°C gives a lower yield while tar formation from naphthalene VI occurs at 160°C. Nitro compound VI was reduced to amine (VII) using $SnCl_2$.

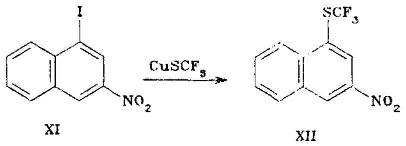


Acid V gave acid chloride (VIII) and amide (IX). 1-Amino-3-nitronaphthalene (X) was obtained by the Hofmann reaction from amide (IX). This amide has poor solubility and, under ordinary conditions [10], does not undergo the Hofmann reaction. We discovered that the use of dimethylformamide as the solvent in this reaction permits obtaining amine X in 40% yield. The yield of amine X is increased to 70% in N-methylpyrrolidone. Amine X was separated from the starting amide by crystallization from benzene (the amide is almost insoluble in this solvent). Amine X may also be obtained directly from acid V by the Schmidt reaction in 90% yield. The reaction proceeds in concentrated sulfuric acid.

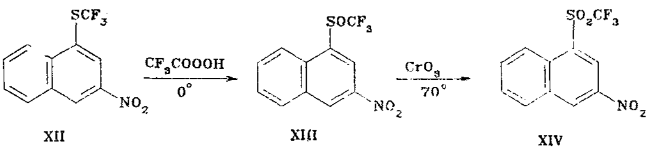


1-Iodo-3-nitronaphthalene (XI) was obtained from amine X by the Sandmeyer reaction. Thus, substituted nitronaphthalenes X and XI are now readily available compounds. Previously, these compounds were obtained in low yield by a multistep procedure involving separation of an isomer mixture [11, 12].

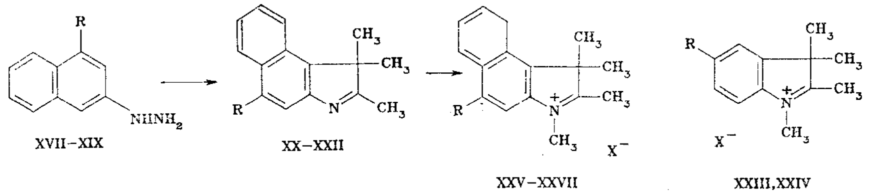
We have recently developed a method for the direct introduction of trifluoromethylthio groups into aromatic and heterocyclic compounds by the action of cuprous trifluoromethylmercaptide on aryl and heteryl iodides [13]. $CuSCF_3$ also gives this reaction with good yields for naphthalene derivatives. This method was used to obtain the first derivative of naphthalene containing a trifluoromethylthio group, namely, 1-trifluoromethylthio-3-nitronaphthalene (XII), from iodide XI.



The oxidation of sulfide XII by CrO_3 in acetic acid or by trifluoroperacetic acid at 70°C leads to destruction of the naphthalene system. The action of trifluoroperacetic acid on the naphthalene system. The action of trifluoroperacetic acid on sulfide XII at 0°C gives sulfoxide (XIII), while at 20°C, this reaction gives a mixture of XIII and sulfone (XIV). This sulfone may be obtained as a pure product by the oxidation of sulfoxide XIII by CrO_3 at 70°C.

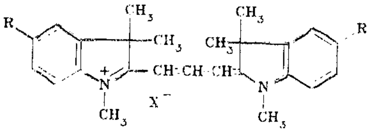


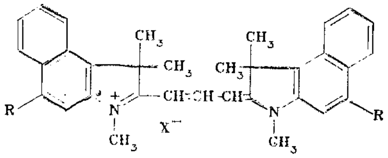
1-Trifluoromethylthio- (XV) and 1-trifluoromethylsulfonyl-3-aminonaphthalenes (XVI) were obtained analogously to amine VII. Amines VII, XV, and XVI were converted to the corresponding 1-trifluoromethyl- (XVII), 1-trifluoromethylthio- (XVIII), and 1-trifluoromethylsulfonyl-3-hydrazinonaphthalenes (XIX). The Fischer reaction was used to convert hydrazines XVII-XIX into the corresponding 5-trifluoromethyl- (XX), 5-trifluoromethylthio- (XXI), and 5-trifluoromethylsulfonyl-1,1,2-trimethyl-1H-benz[e]indoles (XXII). Indolenines II and IV and benzindolenines XX-XXII give the corresponding quaternary salts (XXIII-XXVII) upon heating with methyl iodide.

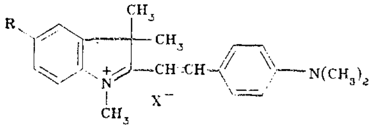


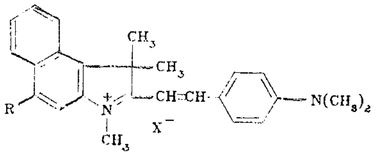
XVII, XX, XXV R=CF_3; XVIII, XXI, XXIII, XXVI R=SCF_3; XIX, XXII, XXIV, XXVII R=SO_2CF_3; X=I, BF_4

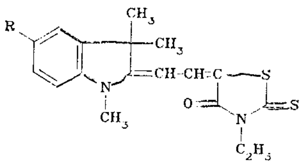
Quaternary salts XXIII-XXVII yield the corresponding indocyanines (XXVIIIa,b and XXIXa-c), styryl dyes (XXXa,b and XXXIa-c), and merocyanines (XXXIIa,b and XXXIIIa,c).

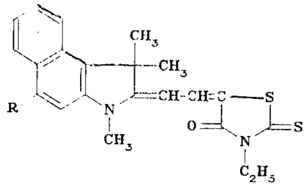

XXVIII a, b


XXIX a-c


XXX a, b


XXXI a-c


XXXII a, b


XXXIII a, c

a R=SCF_3; b R=SO_2CF_3; c R=CF_3; X=I, BF_4

Fluorine-containing CF_3, SCF_3, and SO_2CF_3 substituents in the heterocyclic systems of cyanine dyes which are derivatives of benzothiazole, benzimidazole, and quinoline, as a rule, produce a bathochromic shift of the long-wavelength absorption bands [14]. However, a tendency toward a reduction in the band maximum shift ($\Delta\lambda$) is noted with decreasing electron-withdrawing properties of the heterocyclic residues (in going from quinoline and benzimidazole to benzothiazole). We have shown that in the case of dyes with the indolenine residue having the least electron-withdrawing properties, these effects are even less (0 for SCF_3, 12 nm for SO_2CF_3) and may actually be hypsochromic (−6 nm for CF_3). All these groups in benzindolenine carbocyanines have a significant hypsochromic effect (Table 1).

Another general feature was found in a study of the spectra of asymmetric indolenine dyes: The substituent effect for styryl dues XXX and XXXI are bathochromic and are greater than the effects in the carbocyanines, while only hypsochromic effects are found in the merocyanines XXXII and XXXIII. This finding is related to the electron-donor properties of the indolenine system due to the presence of electron-withdrawing CF_3, SCF_3, and SO_2CF_3 groups,

TABLE 1. Spectral Characteristics of Dyes Studied

R	$\lambda_{max}(\Delta\lambda)$,nm	$\lambda_{max}(\Delta\lambda)$, nm	D, nm	$\lambda_{max}(\Delta\lambda)$, nm	D, nm
	XXVIII	XXX		XXXII	
H	548	545	34	504	40
CF_3 [1]	542 (−6)				
SCF_3	548 (0)	570 (25)	11	496 (−8)	48
SO_2CF_3	560 (12)	581 (36)	2	482 (−20)	66
	XXIX	XXXI		XXXIII	
H	594	558	44	524	43
CF_3	580 (−14)	572 (14)	23	516 (−8)	44
SCF_3	579 (−15)	573 (15)	21,5	515 (−9)	44,5
SO_2CF_3	572 (−22)	576 (18)	15		

which results in a decrease in the electronic asymmetry of the styryl dyes (the deviations are reduced) but in its enhancement in the merocyanines (the deviations increase).

EXPERIMENTAL

The absorption spectra of the ethanolic solutions were taken on an SF-10 spectrophotometer. The physical indices and yields of the compounds synthesized are given in Table 2.

p-Trifluoromethylthiophenylhydrazine (I). A suspension of 6 g (30 mmoles) p-trifluoromethylthioaniline in 20 ml conc. HCl was diazotized by a solution of 2.3 g (33 mmoles) $NaNO_2$ in 10 ml water at 0°C. The diazo solution was filtered and as sample of 15 g (66 mmoles) $SnCl_2 \cdot 2H_2O$ in 17 ml conc. HCl was added at 0°C. The reaction mixture was stirred for 1 h. The precipitate was filtered off and it was mixed with 70 ml 25% aqueous ammonia. After 10 min, hydrazine I was extracted by ether and dried over $MgSO_4$.

Hydrazine XVIII was obtained by analogy.

2,3,3-Trimethyl-5-trifluoromethylthio-3H-indole (II). A sample of 2 g (10 mmoles) hydrazine I and 0.09 g (10 mmoles) methyl isopropyl ketone was heated for 4 h at 70°C. The reaction mixture was cooled, dissolved in 25 ml ether, and dried over $MgSO_4$. The ether was distilled off in vacuum. The residue, 2.7 g (9.7 mmoles) p-trifluoromethylthiophenylhydrazone of methyl isopropyl ketone, was heated at reflux for 3 h with 2.6 g (10 mmoles) boron trifluoride etherate in 15 ml acetic acid. The precipitate was filtered off and washed with 5 ml acetic acid. The filtrate was evaporated in vacuum. A sample of 25 ml 10% aqueous ammonia was added to the residue which was then extracted by ether and dried over $MgSO_4$.

Products IV and XX-XXII were obtained by analogy.

p-Trifluoromethylsulfonylphenylhydrazine (III). A sample of 1.2 g (4.9 mmoles) p-chlorophenyltrifluoromethylsulfone, 5 ml anhydrous ethanol, and 1 g (30 mmoles) anhydrous hydrazine was heated for 20 min at 70°C. The reaction mixture was diluted with water. The precipitate was filtered off, washed with water, and dried to yield 1.1 g (93%) III, mp 93-94°C (from ethanol) (95°C, [5]).

1-Trifluoromethyl-3-nitronaphthalene (VI). A sample of 10 g (46 mmoles) acid V and 10 ml anhydrous HF was placed in a 100-ml stainless steel autoclave and 18 g (160 mmoles) sulfur tetrafluoride was condensed in. The reaction mixture was heated for 10 h at 120°C and cooled. Then, the reaction mixture was poured into 100 ml 20% aq. NaOH and heated for 1 h at 80°C, extracted with ether, and dried over $MgSO_4$. The ether was filtered off to yield 10 g (91%) VI with mp 127-128°C (from hexane) [7].

1-Trifluoromethyl-3-aminonaphthalene (VII). A solution of 2 g (8 mmoles) naphthalene VI in 6 ml ethanol and 7.8 g (34 mmoles) $SnCl_2 \cdot 2H_2O$ in 7 ml conc. HCl was heated for 2 h at 100°C and then poured onto ice. The pH was brought to 14 by adding 40% aq. NaOH. The reaction mixture was extracted with ether and dried over $MgSO_4$. The ether was distilled off and the residue was crystallized to yield 1.2 g (87%) VII with mp 56-57°C (from pentane) [7].

Amines XV and XVI were obtained by analogy.

Acid chloride of 3-nitro-1-naphthalenecarboxylic acid (VIII). A sample of 10 g (46 mmoles) acid V and 30 ml thionyl chloride was heated for 2 h at reflux. Excess $SOCl_2$ was distilled off and the residue was crystallized.

Amide of 3-nitro-1-naphthalenecarboxylic acid (IX). Ammonia was passed through a solution of 10 g (42 mmoles) acid chloride VIII in 200 ml anhydrous benzene at 10°C until the cessation of gas absorption. The precipitate was filtered off, washed with water, dried, and recrystallized.

1-Amino-3-nitronaphthalene (X). A. A solution of 1 g (4.5 mmoles) amide IX in 7 ml N-methylpyrrolidone was added to a hypobromite solution prepared by mixing 1 g (6 mmoles) Br_2 and 1 g (25 mmoles) NaOH in 18 ml water at 0°C. The reaction mixture was stirred for 20 min at 0-5°C and for 1 h at 70°C, diluted with water, extracted with benzene, and dried over $MgSO_4$. The solvent was distilled off in vacuum. The residue was recrystallized from benzene to yield 0.65 g (70%) X with mp 136-137°C [7].

B. A suspension of 4.2 g (20 mmole) of acid V in 20 ml H_2SO_4 (d 1.83) was stirred for 10 min, and 2 g (30 mmole) of sodium azide was added portionwise in the course of 20 min. After the turbulent evolution of nitrogen, the mixture was heated for 1 h at 60°. It was

TABLE 2. Characteristics of Compounds Synthesized

Compound	Mp, deg C[a]	Found, %			Chemical formula	Calculated, %			Yield, %
		C	H	F (N)		C	H	F (N)	
I	63—64	40,4	3,2	(13,1)	$C_7H_7F_3N_2S$	40,4	3,4	(13,5)	48
II	60—61	55,3	4,4		$C_{12}H_{12}F_3NS$	55,6	4,6		55
IV	90—92	49,5	4,0		$C_{12}H_{12}F_3NO_2S$	49,5	4,1		68
VIII	170—172	56,2	2,6		$C_{11}H_6ClNO_3$	56,2	2,6		90
IX	268—270	61,0	4,0	(13,1)	$C_{11}H_8N_2O_3$	61,1	3,7	(12,9)	85
XII	83—84	45,3	2,0	20,6	$C_{11}H_6F_3NO_2S$	45,7	2,2	20,9	72
XIII	165—167	45,6	2,1	19,7	$C_{11}H_6F_3NO_3S$	45,7	2,1	19,7	78
XIV	145—147	43,4	2,0	18,4	$C_{11}H_6F_3NO_4S$	43,2	1,9	18,7	68
XV	98—100	54,5	3,4	23,7	$C_{11}H_8F_3NS$	54,3	3,3	23,4	82
XVI	132—134	48,2	3,0	20,6	$C_{11}H_8F_3NO_2S$	48,0	2,9	20,7	81
XVII	75—76	58,6	4,1	(12,3)	$C_{11}H_9F_3N_2$	58,4	4,0	(12,4)	68
XVIII	90—92	51,0	3,6	(10,9)	$C_{11}H_9F_3N_2S$	51,2	3,5	(10,8)	72
XIX	138—140	45,7	3,2	(9,7)	$C_{11}H_9F_3N_2O_2S$	45,5	3,1	(9,7)	78
XX	215—216	51,5	3,4	11,4 (12,5)	$C_{22}H_{17}F_3N_4O_7$	51,4	3,3	11,1 (12,5)	66
XXI	—[b]	62,1	4,6		$C_{16}H_{14}F_3NS$	62,1	4,5		70
XXII	122—124	56,4	4,1	16,8	$C_{16}H_{14}F_3NO_2S$	56,3	4,1	16,7	56
XXIII	215—217 (dec.)	39,0	4,0		$C_{13}H_{15}F_3INS$	38,9	3,7		97
XXIV	178—180 (dec.)	39,5	4,0	33,7	$C_{13}H_{15}BF_7NO_2S$	39,7	3,8	33,8	92
XXV	210—212 (dec.)	53,9	4,5	35,1	$C_{17}H_{17}BF_7N$	53,8	4,5	35,1	95
XXVI	260—262 (dec.)	44,9	3,8		$C_{17}H_{17}F_3INS$	45,2	3,8		93
XXVII	270—273 (dec.)	42,1	3,7		$C_{17}H_{17}F_3INO_2S$	42,2	3,5		90
XXVIIIa	283—285	47,4	4,1		$C_{27}H_{27}F_6IN_2S_2$	47,4	3,9		38
XXVIIIb	285—287	40,9	3,8		$C_{27}H_{27}F_6IN_2O_4S_2$	41,4	3,7		42
XXIXa	218—220	53,6	3,8		$C_{35}H_{31}F_6IN_2S_2$	53,5	3,9		40
XXIXb	248—250	49,5	3,5		$C_{35}H_{31}F_6IN_2S_2O_4$	49,5	3,6		39
XXIXc	248—250	61,8	4,6		$C_{35}H_{31}BF_{10}N_2$	61,7	4,5		44
XXXa	206—207	49,8	4,6		$C_{22}H_{24}F_3IN_2S$	49,6	4,5		80
XXXb	230—232	49,3	4,2		$C_{22}H_{24}F_3INO_2$	49,2	4,0		78
XXXIa	259—260	53,4	4,2		$C_{26}H_{26}F_3IN_2S$	53,6	4,4		77
XXXIb	265—267	50,9	4,0		$C_{26}H_{26}F_3IN_2O_2S$	50,8	4,2		80
XXXIc	255—257	61,2	5,2		$C_{26}H_{26}BF_7N_2$	61,1	5,1		75
XXXIIa	95—97	51,3	4,2		$C_{19}H_{19}F_3N_2OS_3$	51,4	4,3		40
XXXIIb	128—129	48,0	4,1		$C_{19}H_{19}F_3N_2O_3S_3$	47,9	4,0		34
XXXIIIa	133—134	56,0	4,1		$C_{23}H_{21}F_3N_2OS_3$	55,9	4,2		60
XXXIIIc	132—134	59,9	5,7		$C_{23}H_{21}F_3N_2OS_2$	59,7	4,6		50

[a] The recrystallization solvents were pentane (I, II, IV, and XII), hexane (XIII, XVI-XIX, and XXII), benzene (VIII, XX, XXXLI, and XXXIII), methanol (XV), ethanol (XXIII-XXXI), and DMF (IX). [b] Bp 170-173°C (0.3 mm).

then poured onto ice and made basic with 20% NaOH. The precipitate was filtered, dried, and crystallized. Yield 3.4 g (90%), mp 136-137°.

1-Iodo-3-nitrolnaphthalene (XI). A suspension of 1g (5.3 mmoles) amine X and 10 ml conc. HCl was stirred for 15 min at 70°C and then cooled to 10°C. A solution of 0.6 g (8.7 mmoles) $NaNO_2$ in 5 ml water was added and the reaction mixture was stirred for an additional 20 min. The diazo solution was added to a solution of 1 g (6 mmoles) KI and 1 g (3.9 mmoles) iodine in 8 ml water and heated for 1 h at 80°C, then cooled and extracted with ether. The ethereal solution was washed with water and dried over $MgSO_4$. The ether was distilled off to yield 0.9 g (60%) XI with mp 146-147°C (from ethanol) [11].

1-Trifluoromethylthio-3-nitronaphthalene (XII). A solution of 9 g (33 mmoles) naphthalene XI and 8.2 g (49 mmoles) cuprous trifluoromethylmercaptide in 50 ml anhydrous N-methylpyrrolidone was heated for 4 h at 140°C. The mixture was diluted with 300 ml ether. The ethereal solution was decanted, washed with three 50-ml portions of water, and dried over $MgSO_4$.

1-Trifluoromethylthionyl-3-nitronaphthalene (XIII). A sample of 0.5 ml (12 mmoles) 80% hydrogen peroxide was added to 4 ml trifluoroacetic anhydride and stirred for 20 min at −20°C and for 10 min at 20°C. The reaction mixture was cooled to −5°C and 0.27 g (1.1 mmole) naphthalene XII was added. Then, the reaction mixture was stirred for 1 h at 0°C and for 30 min at 20°C and diluted with water. The product was filtered off and dried.

1-Trifluoromethylsulfonyl-3-nitronaphthalene (XIV). A solution of 0.3 g (1 mmole) sulfoxide XIII and 0.4 g (4 mmoles) CrO_3 in 4 ml acetic acid was heated for 40 min at 70°C. The mixture was poured onto ice. The precipitate was filtered off, washed with water, and dried.

1-Trifluoromethylthio-3-hydrazinonaphthalene (XVIII). A solution of 0.5 g (2 mmoles) amine XV in 6 ml acetic acid was added to nitrosylsulfuric acid prepared from 0.5 g (2.2 mmoles) $NaNO_2$ and 2.55 ml H_2SO_4 (d 1.83), stirred for 3 h at 20°C, and added to a solution of 4 g (14 mmoles) $SnCl_2 \cdot 2H_2O$ in 10 ml conc. HCl at 10°C. The reaction mixture was stirred for 1 h. The suspension was made basic with 25% aq. NaOH with ice cooling, extracted with ether, and dried over $MgSO_4$.

Product XIX was prepared by analogy.

1,2,3,3-Tetramethyl-5-trifluoromethylthio-3H-indolium iodide (XXIII). A sample of 2.5 g (10 mmoles) base II and 2.8 g (20 mmoles) methyl iodide was heated in an ampule for 6 h at 75°C. The ampule was cooled, opened, and the product was washed with ether.

Products XXIV, XXVI, and XXVII were prepared by analogy.

1,1,2,3-Tetramethyl-5-trifluoromethyl-1H,benz[e]indolium tetrafluoroborate (XXV) was prepared by the action of 1 mmole $AgBF_4$ and 2 mmoles methyl iodide in 3 ml anhydrous benzene on 1 mmole base XX.

5-R-2-[3-(5R-1,2-dihydro-1,3,3-trimethyl-3H-indol-2-ylidene)-1-propyl]-1,3,3-trimethyl-3H-indolium iodides (XXVIIIa and XXVIIIb) and 5-R-2-[3-(5-R-2,3-dihydro-1,3,3-trimethyl-1H-benz[e]indol-2-ylidene)-1-propenyl]-1,1,3-trimethyl-1H-benz[e]indolium iodides (XXIXa and XXIXb) and tetrafluoroborate (XXIXc). A sample of 1 mmole of the corresponding quaternary salt was heated with 1 ml ethyl orthoformate in 2 ml acetic anhydride for 1 h at 120°C. The dyes were precipitated by ether, filtered off, and crystallized.

5-R-2-[2-(4-Dimethylaminophenyl)ethenyl]-1,3,3-trimethyl-3H-indolium iodides (XXXa and XXXb) and 5-R-2-[2-(4-dimethylaminophenyl)ethenyl]-1,1,3-trimethyl-1H-benz[e]indolium iodides (XXXIa and XXXIb) and tetrafluoroborate (XXXIc). A sample of 1 mmole of the corresponding quaternary salt and 1 mmole p-dimethylaminobenzaldehyde in 2 ml acetic anhydride was heated at reflux for 1 h. The dyes were precipitated by ether, filtered off, and crystallized.

5-[2-(5-R-1,2-Dihydro-1,3,3-trimethyl-3H-indol-2-yliden)ethylidene]-2-thioxo-3-ethyl-4-thiazolidinones (XXXIIa and XXXIIb) and 5-[2-(5-R-2,3-dihydro-1,1,3-trimethyl-1H-benz[e]-indol-2-yliden)ethylidene]-2-thioxo-3-ethyl-4-thiazolidonones (XXXIIIa and (XXXIIIc). A sample of 1 mmole of the corresponding quaternary salt, 1 mmole acetanilidovinylrhodanine, and 1 mmole triethylamine in 2 ml acetic anhydride was heated for 1 h at 130°C, cooled, and poured into 100 ml water. The dye was filtered off, dried, and crystallized.

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