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SYNTHESIS OF THIAPENTA- AND THIAHEXACARBOCYANINE DYES FROM ALKOXY-SUBSTITUTED POLYMETHINE SALTS WITH DIMETHYLAMINE RESIDUES

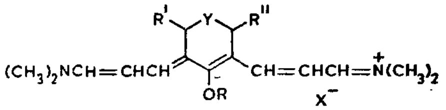
N. V. Monich, Zh. A. Krasnaya,
I. I. Levkoev,* and V. F. Kucherov*

UDC 668.819.45:547.789.61'812.5

The possibility of the use of alkoxypolymethine salts with dimethylamine residues to obtain thiapenta- and thiahexacarbocyanine dyes is demonstrated. The synthesis of a number of meso-alkoxythiapentacarbocyanines that contain various condensed rings in the 12 and 14 positions of the polymethine chain was realized, and their photographic properties were investigated. It was established that the dyes obtained are effective infrachromatic sensitizers. The electronic absorption spectra of the synthesized compounds were measured.

Polymethine dyes are starting compounds for the preparation of various cyanine dyes. Pentacarbocyanines that are unsubstituted in the chain can be synthesized by the condensation of quaternary heterocyclic salts that contain an active methyl group with salts of dianils, bis(tetrahydroquinolides) [1], or di(N-alkyl)anils [2] of nonatrienedial. Pentacarbocyanines with condensed rings in the chromophore are formed when salts of di(N-alkyl)anils or bis(tetrahydroquinolides) of substituted nonatrienedials are used [3].

In the present research we studied the possibility of obtaining dyes from readily accessible and stable alkoxypolymethine salts with dimethylamine residues (I), which were recently synthesized in [4, 5].



I

$R = CH_3$, C_2H_5, C_4H_9; R′ and R″ = H, CH_3; $X = CH_3SO_4$, $C_2H_5SO_4$, I, BF_4; $Y = CH_2$, $CHCH_3$, $CHC_4H_9\text{-}t$, O

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*Deceased.

All-Union State Scientific-Research and Design Institute of the Photographic Chemical Industry, Moscow 125167. N. D. Zelinskii Institute of Organic Chemistry, Academy of Sciences of the USSR, Moscow 117913. Translated from Khimiya Geterotsiklicheskikh Soedinenii, No. 12, pp. 1631-1636, December, 1981. Original article submitted May 20, 1980; revision submitted October 14, 1980.

0009-3122/81/1712-1192$07.50

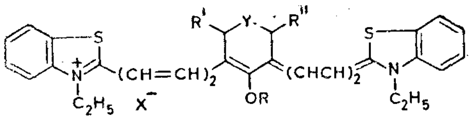
TABLE 1. Electronic Absorption Spectra of Thiapentacarbocyanines IIc–j

Compound*	R′	R″	X	Y	In ethanol		mp, °C (dec.)
					λ_{max}, nm		
IIc	H	H	BF_4	CH_2	994	7,0	1012
IId	H	CH_3	I	CH_2	988	7,0	1005
IIe	H	H	TsO	$CHCH_3$	991	7,0	1008
IIf	H	CH_3	I	O	985	6,0	1000
IIg	H	CH_3	I	$CHCH_3$	988	7,4	1005
IIh	CH_3	CH_3	I	CH_2	988	7,7	1004
IIi	H	H	I	CH_2CH_2	964	3,5	980
IIj	H	H	TsO	CHC_4H_9-*t*	991	6,8	1008

*$R = C_2H_5$.

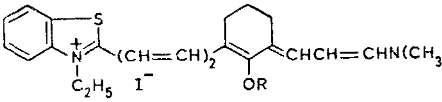
We found that salts I are extremely stable in methanol solutions at 20°C as compared with nonatrienedial di(N-methyl)anil chloride. In the case of a solution of the latter in absolute methanol ($c = 0.5 \cdot 10^{-4}$ mole/liter) a decrease by a factor of almost three was observed in the optical density (D) of the absorption maximum 45 min after dissolving. In the case of the analogous solutions of the methylsulfate of Ia ($R = CH_3$, $R' = R'' = H$, $Y = CH_2$) and the tetrafluoroborate of Ib ($R = C_2H_5$, $R' = R'' = H$, $Y = CH_2$) the D values underwent only 5% changes after 24 h.

The reaction of the iodide of Ib ($R = C_2H_5$, $R' = R'' = H$, $Y = CH_2$) under the conditions of the synthesis of 3,3′-diethylthiapentacarbocyanine [by condensation with nonatrienedial di(N-methyl)anil chloride in methanol or acetic anhydride in the presence of triethylamine] [2] led to the rapid formation of a copious crystalline precipitate consisting of a mixture of thiapentacarbocyanine IIa, the corresponding hemicyanine IIIa, and the starting nonamethylidyne salt Ib (X = I).



IIa-j

II a $R=C_2H_5$, b $R=C_4H_9$; IIa, b $R'=R''=H$, $X=I$, Y —CH_2; c-j (see Table 1)



IIIa, b

IIIa $R=C_2H_5$; IIIb $R=C_4H_9$

Despite variation of the reaction conditions, the dye always contained appreciable amounts of the hemicyanine. It should be noted that the separation of this sort of mixture is extremely difficult. A similar pattern was also observed in the case of the iodide of Ic ($R = C_4H_9$, $R' = R'' = H$, $Y = CH_2$). However, pure thiapentacarbocyanine tetrafluoroborate IIc was obtained (in 60% yield) in the reaction of 2-methyl-3-ethylbenzothiazolium toluenesulfonate and the tetrafluoroborate of Ib (in acetic anhydride in the presence of triethylamine at room temperature).

The use of alkoxynonamethylidyne salts I in the form of ethylsulfates, which have very high solubilities, proved to be extremely successful. A number of meso-alkoxythiapentacarbocyanines that contain various condensed rings in the 12 and 14 positions of the polymethine chain (IId-j, Tables 1 and 2) were synthesized in 33-60% yields by condensation of ethylsulfates I and 2-methyl-3-ethylbenzothiazolium toluenesulfonate under the conditions indicated above.

An attempt to obtain indolenine analogs of thiapentacarbocyanines II by the reaction of alkoxynonamethylidyne salts I ($R = CH_3$, C_2H_5; $R' = R'' = H$; $Y = CH_2$, $CHCH_3$) and 1,2,3,3-tetramethyl-3H-indolium iodide was unsuccessful (see [2]). Despite extensive variation of the reaction conditions, including the use of potassium tert-butoxide in dimethyl sulfoxide

TABLE 2. Conditions for the Preparation of Thiapentacarbocyanines IIe-j and Their Characteristics

Compound	Reaction time, min*		mp, °C (dec.)	Wash solvent†	I (S) found, %	Empirical formuls	I (S) calc., %	Yield, %
	25°	0°						
IIe	15	15	150—152‡	Ethanol 50%, B–C (5 : 1)	(12,6)	$C_{42}H_{46}N_2O_4S_3 \cdot 0{,}5C_2H_5OH$	(12,6)	50
IIf	15	15	190—193	Ethanol B–C (3 : 2)	16,0	$C_{34}H_{37}IN_2O_2S_2 \cdot 2C_2H_5OH$	16,1	43
IIg	10	10	171—173	B–C (5 : 1)	16,7	$C_{36}H_{41}IN_2OS_2 \cdot C_2H_5OH$	16,8	40
IIh	5	10	170—173‡	B–C (5 : 1)	16,6	$C_{36}H_{41}IN_2OS_2 \cdot C_2H_5OH$	16,8	40
IIi	30	—**	193—195‡	B–C (3 : 2)	16,5	$C_{35}H_{39}IN_2OS_2 \cdot 1{,}5C_2H_5OH$	16,6	57
IIj	15	15	155—157‡	Ethanol	(10,8)	$C_{45}H_{52}N_2O_4S_3 \cdot 2C_2H_5OH$	(11,0)	33

*The reaction was carried out initially at room temperature and was completed at 0°C.

†The abbreviation B–C indicates a mixture of benzene with methylene chloride.

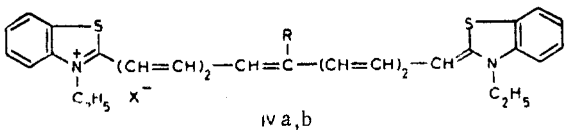
‡The dye precipitated from the reaction solution in the form of crystals.

**The dye was obtained from 2-methyl-3-ethylbenzothiazolium iodide.

(DMSO) as the condensing agent [6], we did not detect even traces of an indopentacarbocyanine dye in any of the experiments.

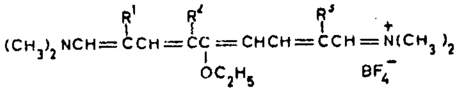
It should be noted that alkoxynonamethylidyne salts I proved to be identical to nonatrienedial bis(dimethylimine) perchlorate with respect to their tendency to undergo condensation with quaternary heterocyclic salts that contain an active methyl group and with respect to their stability in methanol solution.

We subsequently accomplished the synthesis of 13-ethoxythiapentacarbocyanine from 2-methyl-3-ethylbenzothiazolium toluenesulfonate and tetrafluoroborate Va [4, 5]. The results of elementary analysis of the dye obtained are in good agreement with the formula of toluenesulfonate IVa.



IV a,b

IVa $R{=}OC_2H_5$, $X{=}TsO$; IVb $R{=}H$, $X{=}ClO_4$



$(CH_3)_2NCH{=}\overset{R^1}{C}CH{=}\overset{R^2}{C}\underset{OC_2H_5}{C}{=}CHCH{=}\overset{R^3}{C}CH{=}\overset{+}{N}(CH_3)_2\ BF_4^-$

Va-c

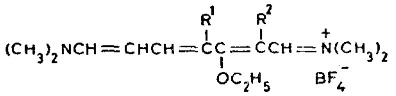
Va $R^1{=}R^2{=}R^3{=}H$; Vb $R^1{=}R^3{=}H$, $R^2{=}CH_3$; Vc $R^1{=}R^3{=}CH_3$, $R^2{=}H$

It has been previously shown that the methyl groups in ethoxynonamethylidyne salt Va decrease its stability markedly. Salt Vb, which contains methyl and ethoxy groups attached to adjacent carbon atoms, is particularly unstable. It decomposes completely under the conditions of the synthesis of the pentacarbocyanine. The formation of the corresponding pentacarbocyanine was observed in the case of the reaction of 2-methyl-3-ethylbenzothiazolium toluenesulfonate and salt Vc; however, it is present in a mixture with a number of compounds that absorb at 460, 520, 550, and 670 nm.

A study of the color of the thiapentacarbocyanines II (Table 1) showed that a methyl or tert-butyl group in the 4 position of the cyclohexene ring gives rise to a hypsochromic shift (4 nm in CH_2Cl_2) of the absorption maximum of dye IIc. Transfer of the methyl group to the 3 position, the absence of two methyl groups in the 3 and 4 or 3 and 5 positions of the ring, and replacement of the methylidyne grouping in the 4 position of the ring by an oxygen atom lead to a further shift of the absorption maximum of the pentacarbocyanine to the short-wave part of the spectrum (5-8 nm in CH_2Cl_2). The introduction of a seven-membered ring in place of the six-membered ring (IIi) in the polymethine chain of the dye is accompanied by a hypsochromic shift of the absorption maximum (32 nm in CH_2Cl_2).

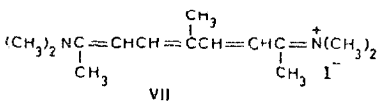
The absorption maximum of pentacarbocyanine IVa is located at 920 nm (in CH_2Cl_2). Thus the incorporation of an ethoxy group in the meso position of the chromophore of 3,3'-diethylthiapentacarbocyanine (IVb) [2] leads to a pronounced hypsochromic shift of its absorption maximum (89 nm in CH_2Cl_2). A similar phenomenon was previously observed in the thiatricarbocyanine series [7].

In order to synthesize thiatetracarbocyanine dyes we investigated the conditions for the reaction of 2-methyl-3-ethylbenzothiazolium toluenesulfonate with heptamethylidyne salts VIa, b [4] and VII [8].



$(CH_3)_2NCH{=}CHCH{=}\overset{R^1}{C}\underset{OC_2H_5}{C}{=}\overset{R^2}{C}CH{=}\overset{+}{N}(CH_3)_2 \; BF_4^-$

VIa, b

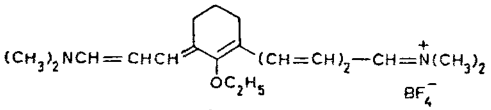


$(CH_3)_2N\underset{CH_3}{C}{=}CHCH{=}\overset{CH_3}{C}CH{=}CH\underset{CH_3}{C}{=}\overset{+}{N}(CH_3)_2 \; I^-$

VII

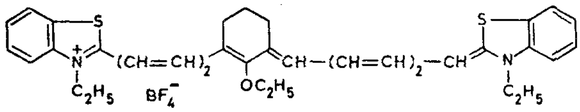
VIa $R^1{=}R^2{=}H$; VIb $R^1{+}R^2{=}(CH_2)_3$

Starting from tetrafluoroborate VIb we obtained the corresponding tetracarbocyanine (in methanol in the presence of triethylamine), which, however, was contaminated with an appreciable amount of the hemicyanine. Salt VIa, as well as the heptamethylidyne salt with three methyl groups in the chain (VII), could not be subjected to condensation even when we used potassium tert-butoxide in DMSO as the condensing agent.



VIII

Undecamethylidyne salt VIII proved to be extremely reactive. Thiahexacarbocyanine IX was synthesized by the reaction of the latter with 2-methyl-3-ethylbenzothiazolium toluenesulfonate. Its absorption maximum was located at 1125 nm (in CH_2Cl_2), i.e., it was shifted 5 nm to the short-wave region of the spectrum as compared with the absorption maximum of the chain-unsubstituted analog [9].



IX

A study of the effectiveness of the spectral sensitization of highly sensitive silver bromide and iodide emulsions that contain the synthesized thiapentacarbocyanines showed that the 13-ethoxy derivative (IV) and the dye with a condensed seven-membered ring in the chain (IIi) are ineffective. On the other hand, thiapentacarbocyanines IIc-h and IIj are effective infrachromatic sensitizers. With respect to additional light sensitivity (behind a KS-14 light filter), the photolayers subjected to their influence are not inferior to photolayers sensitized with dye IVb (X = I). With respect to its sensitizing effect, thiahexacarbocyanine IX does not differ from the chain-unsubstituted analog.

The authors thank A. G. Vakar and M. A. Chizhova for performing the photographic tests.

EXPERIMENTAL

The electronic absorption spectra were recorded with a Cary-14 spectrophotometer.

The presence of crystallization alcohol in dyes II was determined by gas chromatography with a Chrom-3 chromatograph (Czechoslovak SSR) with a thermal conductivity detector (a katharometer) and Porapak Q as the filler.

[9-Dimethylamino-4,6-(2-tert-butyltrimethylene)-5-ethoxynona-2,4,6,8-tetraenylidene]-dimethylammonium Ethylsulfate. This compound was obtained in 90% yield by the method in [4] by heating the corresponding bis(dimethylamino) ketone with diethyl sulfate for 5 h. UV spectrum, λ_{max} in ethanol: 640 nm (ε_{max} $1.7 \cdot 10^5$).

(11-Dimethylamino-6,8-trimethylene-7-ethoxyundeca-2,4,6,8,10-pentaenylidene)dimethylammonium Tetrafluoroborate (VIII). This compound was obtained in 85% yield in the form of a crystalline precipitate by the method in [4] from the corresponding bis(dimethylamino) ketone [10] at —10 to 0°C. UV spectrum, λ_{max} in ethanol: 770 nm (ε $8.04 \cdot 10^4$).

2-[11-(3-Ethylbenzothiazolin-2-ylidene)-6-ethoxy-5,7-trimethyleneundeca-1,3,5,7,9-pentaen-1-yl]-3-ethylenebenzothiazolium Tetrafluoroborate (IIc) [3]. A mixture of 0.28 g (0.8 mole) of 2-methyl-3-ethylbenzothiazolium toluenesulfonate, 0.08 g (0.2 mmole) of tetrafluoroborate Ib, 4 ml of acetic anhydride, and 1.2 ml of a 1 N solution of triethylamine in acetic anhydride was maintained at room temperature for 1.5 h. The resulting precipitate was removed by filtration, washed successively with ether, water, and ether, and stirred with ethanol to give 0.09 g (60%) of fine brown crystals with a copper lustre and mp 257-259°C (dec.). Found: C 63.5; H 5.8; N 4.8; S 10.5%. $C_{34}H_{37}BF_4N_2OS_2$. Calculated: C 63.7; H 5.8; N 4.4; S 10.0%.

2-[11-(3-Ethylbenzothiazolin-2-ylidene)-6-ethoxy-5,7-(1-methyltrimethylene)undeca-1,3,5,7,9-pentaen-1-yl]-3-ethylbenzothiazolium Iodide (IId). A mixture of 0.28 g (0.8 mmole) of 2-methyl-3-ethylbenzothiazolium toluenesulfonate, 0.085 g (0.2 mmole) of ethylsulfate I ($R = C_2H_5$, $R' = H$, $R'' = CH_3$, $Y = CH_2$), 3 ml of acetic anhydride, and 1.2 ml of a 1 N solution of triethylamine in acetic anhydride was maintained at room temperature for 15 min and in ice for 15 min. The solution was then mixed with ether, the liquid was decanted, and the viscous dark oil was stirred with water. The crystals were removed by filtration, washed with water and ether, and dissolved in ethanol. The alcohol solution was mixed with a 15% aqueous solution of potassium iodide, and the resulting precipitate was removed by filtration, washed on the filter with 50% aqueous alcohol and ether, and stirred successively with ethanol and benzene–methylene chloride (3:2) to give 0.05 g (36%) of fine brown crystals with mp 202-203°C (dec.). Found: C 59.4; H 6.2; I 16.4; N 3.7%. $C_{35}H_{39}IN_2OS_2 \cdot 1.5\ C_2H_5OH$. Calculated: C 59.8; H 6.3; I 16.6; N 3.7%.

Dyes IIe-j (Table 2) were similarly obtained.

2-[11-(3-Ethylbenzothiazolin-2-ylidene)-6-ethoxyundeca-1,3,5,7,9-penta en-1-yl]-3-ethylbenzothiazolium Toluenesulfonate (IVa). A mixture of 0.28 g (0.8 mmole) of 2-methyl-3-ethylbenzothiazolium toluenesulfonate, 0.07 g (0.2 mmole) of tetrafluoroborate Va, 1 ml of acetic anhydride, and 1.2 ml of a 1 N solution of triethylamine in acetic anhydride was maintained at room temperature for 1 h. The resulting crystals were removed by filtration, washed successively with ether, water, and ether, and stirred successively with 50% aqueous ethanol and 96% ethanol to give 0.05 g (40%) of fine brown crystals with a copper lustre and mp 152-154°C (dec.). UV spectrum, λ_{max} in ethanol: 860 nm (ε_{max} $5.20 \cdot 10^4$); λ_{max} in CH_2Cl_2 920 nm. Found: C 65.6; H 6.0; N 3.6%. $C_{38}H_{40}N_2O_4S_3 \times C_2H_5OH$. Calculated: C 65.7; H 6.3; N 3.7%.

(9-Dimethylaminonona-2,4,6,8-tetraenylidene)dimethylammonium Perchlorate (X) [11]. A solution of 0.14 g (0.4 mmole) of N-[9-(N-methylanilino)nona-2,4,6,8-tetraenylidene]-N-methylanilinium chloride [12] in 6 ml of absolute ethanol was mixed with 0.4 ml of a 33% aqueous solution of dimethylamine (3 mmole), and the resulting mixture was mixed with a solution of 0.56 g (4 mmole) of dimethylamine perchlorate in 6 ml of absolute ethanol. The mixture was then maintained at room temperature for 1.5 h, and the bright blue liquid was stirred with ether. The resulting precipitate was removed by filtration, washed successively with ether, a small amount of water, ether, and benzene, and dried to give 0.10 g (83%) of fine dark-gray crystals with a steel lustre and mp 121-122°C (dec.). UV spectrum, λ_{max} in CH_2Cl_2: 625 nm (ε_{max} $2.63 \cdot 10^5$). Found: C 51.1; H 7.5; Cl 10.2; N 7.8%. $C_{13}H_{21}ClN_2O_4 \cdot C_2H_5OH$. Calculated: C 51.4; H 7.8; Cl 10.2; N 8.0%.

2-[11-(3-Ethylbenzothiazolin-2-ylidene)undeca-1,3,5,7,9-pentaen-1-yl]-3-ethylbenzothiazolium Perchlorate (IVb). A mixture of 0.06 g (0.2 mmole) of perchlorate X, 0.24 g (0.8 mmole) of 2-methyl-3-ethylbenzothiazolium iodide, 4 ml of acetic anhydride, and 1.2 ml of a 1 N solution of triethylamine in acetic anhydride was maintained at room temperature for 30 min, after which the resulting precipitate was removed by filtration, washed successively with ether, water, a small amount of ethanol, and benzene, and dried. The crystals were dissolved in methylene chloride, a solution of 0.08 g of anhydrous sodium perchlorate in 1 ml of ethanol, was added, 30 ml of ether was added, and the mixture was maintained at room temperature for 20-30 min. The precipitate was removed by filtration, washed successively with ether, water, and ether, and stirred with methanol to give 0.05 g (45%) of fine dark-brown crystals with a metallic luster and mp 202-204°C (dec.). UV spectrum, λ_{max} in ethanol: 985 nm (ε_{max} $5.5 \cdot 10^{-4}$); λ_{max} in CH_2Cl_2 1009 nm. Found: C 61.1; H 5.2; Cl 6.0%. $C_{29}H_{29}ClN_2O_4S_2$. Calculated: C 61.2; H 5.1; Cl 6.2%.

2-[13-(3-Ethylbenzothiazolin-2-ylidene)-6-ethoxy-5,7-trimethylenetrideca-1,3,5,7,9,11-hexaen-1-yl]-3-ethylbenzothiazolium Tetrafluoroborate (IX). A 0.5-ml sample of a 1 N solution of triethylamine in acetic anhydride (cooled with ice) was added to a mixture of 0.08 g (0.2 mmole) of undecamethylidyne salt VIII, 0.18 g (0.5 mmole) of 2-methyl-3-ethylbenzothiazolium toluenesulfonate, and 2.5 ml of acetic anhydride, and the reaction mixture was allowed to stand in ice for 15 min. The resulting precipitate was removed by filtration, washed successively with ether, water, and ether, and dried. The dye was then washed by stirring with benzene—methylene chloride (3:1). This operation was repeated twice to give fine dark-brown crystals with mp 214-215°C (dec.). The yield was 0.05 g (38%). UV spectrum, λ_{max} in ethanol: 1100 nm (ε_{max} $2.45 \cdot 10^4$) λ_{max} in CH_2Cl_2 1125 nm. Found: C 64.5; H 6.0; N 3.9%. $C_{36}H_{39}BF_4N_2OS_2$. Calculated: C 64.9; H 5.9; N 4.2%.

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