The Reaction Products of 2*H*-Isoindole-4,7-dione Derivatives with 2-Aminobenzenethiol

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From 2,3,4-Trisubstituted oxazolium-5-oxides and 2-methyl-, 2-phenyl- or 2-bromo-1,4-benzoquinone 1,2,3,5-tetrasubstituted 2*H*-isoindole-4,7-dione derivatives were prepared. These compounds were condensed with 2-aminobenzenethiol to produce 1,2,3-trisubstituted or 1,2,3,5-tetrasubstituted 4*H*-pyrrolo[3,4-*a*]phenothiazin-4-one derivatives. In the case of 1,2,3-trisubstituted 5-methyl-2*H*-isoindole-4,7-dione 1,2,3,5-tetrasubstituted 7-(2-mercaptophenyl)imino-2*H*-isoindole-4-one was obtained instead of the expected phenothiazinones.

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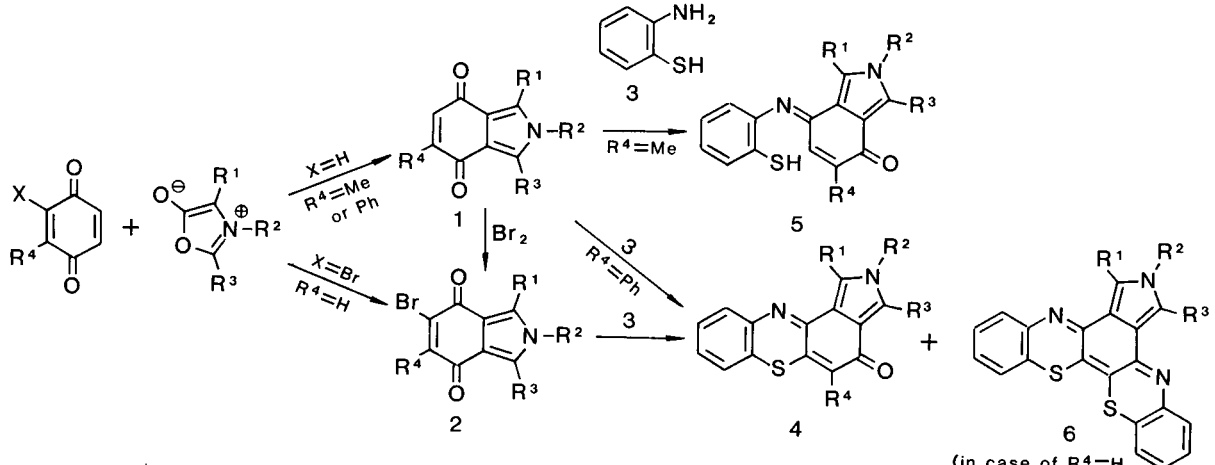
As part of our program to prepare the cyclic iminoquinone derivatives, we have synthesized several benzophenoxazone, pyridophenoxazone and benzophenothiazone derivatives [1-3].

In this work, 4*H*-pyrrolo[3,4-*a*]phenothiazin-4-ones and their related compounds were prepared by the reaction of 2*H*-isoindole-4,7-dione derivatives with 2-aminobenzenethiol (**3**). 2,5-Dimethyl-1,3-diphenyl-2*H*-isoindole-4,7-dione (**1a**) was brominated in acetic acid with bromine in the presence of sodium acetate to the 6-bromo derivative **2a** and then was reacted with **3** in the presence of potassium acetate to produce 2,5-dimethyl-1,3-diphenyl-4*H*-pyrrolo[3,4-*a*]phenothiazin-4-one (**4a**). In the case of the reaction of **1a**, without bromination, with **3** 2,5-dimethyl-1,3-diphenyl-7-(2-mercaptophenyl)imino-2*H*-isoindol-4-one (**5a**) was obtained in good yield. In contrast to **1a**, 2-methyl-1,3,4-triphenyl-2*H*-isoindole-4,7-dione (**1b**) produced by the condensation with **3** in the presence of 15% hydrochloric acid 2-methyl-1,3,5-triphenyl-4*H*-pyrrolo[3,4-*a*]phenothiazin-4-one (**4b**) instead of **5b**. Compound **4b** was also prepared from 6-bromoisoindoledione (**2b**) with **3**.

5-Bromo-2-methyl-1,3-diphenyl-2*H*-isoindole-4,7-dione (**2c**) afforded by the condensation with **3**, 2-methyl-1,3-diphenyl-4*H*-pyrrolo[3,4-*a*]phenothiazin-4-one (**4c**) and a small amount of 7-methyl-6,8-diphenylpyrrolo[3,4-*a*][1,4]-benzothiazino[3,2-*c*]phenothiazine (**6c**).

Reaction of 2-phenyl-3,4-trimethyleneoxazolium 5-oxide, generated in solution, with 2-substituted 1,4-benzoquinones was non-regiospecific, giving mixtures of

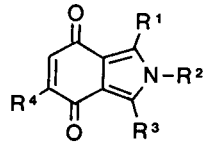


Scheme I

	R¹	R²	R³	R⁴
a	Ph	Me	Ph	Me
b	Ph	Me	Ph	Ph
c	Ph	Me	Ph	H
d (A or B)	$-(CH_2)_3-$		Ph	Me
d (B or A)	Ph	$-(CH_2)_3-$		Me
e (A or B)	$-(CH_2)_3-$		Ph	Ph
e (B or A)	Ph	$-(CH_2)_3-$		Ph
f (A or B)	Ph	$-(CH_2)_3-$		H
f (B or A)	$-(CH_2)_3-$		Ph	H

Table 1

Physical and Spectroscopic Data for Compounds **1** and **2**



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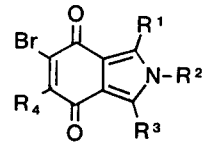
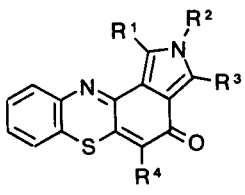


2

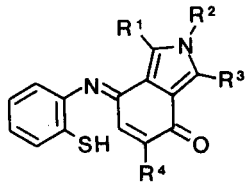
Compound	R^1	R^2	R^3	R^4	Yield (%)	Mp (°C) (recrystallized)	Molecular Formula	Mass (M^+)	IR (cm^{-1})	^{1}H-NMR (Deuteriochloroform) (δ)
1a	Ph	Me	Ph	Me	35 37 [4]	183.0-185.0 (*i*-PrOH) 172-174 [4]	$C_{22}H_{17}NO_2$ (327.4)	327	1638 (C=O)	7.54 (s, 10H), 6.54 (q, 1H, quinone H), 3.34 (s, 3H, NCH_3), 2.04 (d, 3H, quinone CH_3)
1b	Ph	Me	Ph	Ph	35	216.0-217.5 (*i*-PrOH)	$C_{27}H_{19}NO_2$ (389.5)	389	1640 (C=O)	7.62-7.48 (m, 13H), 7.39 (m, 2H), 6.79 (s, 1H, quinone H), 3.36 (s, 3H, NCH_3)
1dA or **B**	-$(CH_2)_3$-		Ph	Me		193.5-195.0 (Ethanol)	$C_{18}H_{15}NO_2$ (277.3)	277	1635 (C=O)	7.64 (m, 2H), 7.46 (m, 3H), 6.53 (q, 1H, quinone H), 4.02 (t, 2H, NCH_2), 3.17 (t, 2H, pyrrole CH_2), 2.58 (p, 2H, $CH_2CH_2CH_2$), 2.07 (d, 3H, quinone CH_3)
					30 28 [4]	178-179 [4]				
1dB or **A**	Ph	-$(CH_2)_3$-		Me		177.5-179.5 (Ethanol)	$C_{18}H_{15}NO_2$ (277.3)	277	1635 (C=O)	7.64 (m, 2H), 7.46 (m, 3H), 6.56 (q, 1H, quinone H), 4.01 (t, 2H, NCH_2), 3.18 (t, 2H, pyrrole CH_2), 2.58 (p, 2H, $CH_2CH_2CH_2$), 2.06 (d, 3H, quinone CH_3)
1eA or **B**	-$(CH_2)_3$-		Ph	Ph		147.0-149.0 (Ethanol)	$C_{27}H_{17}NO_2$ (339.4)	339	1635 (C=O) 1630 (C=O)	7.75-7.38 (m, 9H), 7.01 (m, 1H), 6.80 (s, 1H, quinone H), 4.07 (t, 2H, NCH_2), 3.23 (t, 2H, pyrrole CH_2), 2.61 (p, 2H, $CH_2CH_2CH_2$)
					32					
1eB or **A**	Ph	-$(CH_2)_3$-		Ph		203.0-205.0 (Ethanol)	$C_{27}H_{17}NO_2$ (339.4)	339	1638 (C=O) 1630 (C=O)	7.74-7.37 (m, 10H), 6.82 (s, 1H, quinone H), 4.03 (t, 2H, NCH_2), 3.23 (t, 2H, pyrrole CH_2), 2.61 (p, 2H, $CH_2CH_2CH_2$)
2a	Ph	Me	Ph	Me	31	234.0-235.5	$C_{22}H_{16}BrNO_2$ (406.3)	405/407	1655 (C=O) 1647 (C=O)	7.54 (s, 10H), 3.35 (s, 3H, NCH_3), 2.26 (s, 3H, quinone CH_3)
2b	Ph	Me	Ph	Ph	60	215.5-217.0 (Acetone)	$C_{27}H_{18}BrNO_2$ (4.68.4)	467/469	1680 (C=O) 1658 (C=O)	7.64-7.46 (m, 14H), 7.41 (m, 1H), 3.38 (s, 3H, NCH_3)
2c	Ph	Me	Ph	H	43	218.0-220.5 (Ethanol-Acetone)	$C_{21}H_{14}BrNO_2$ (392.3)	391/393	1653 (C=O) 1635 (C=O)	7.54 (s, 10H), 7.22 (s, 1H, quinone H), 3.36 (s, 3H, NCH_3)
2fA or **B**	Ph	-$(CH_2)_3$-		H		194.0-196.5 (Ethanol)	$C_{17}H_{12}BrNO_2$ (342.2)	341/343	1662 (C=O) 1632 (C=O)	7.64 (m, 2H), 7.50 (m, 3H), 7.22 (s, 1H, quinone H), 4.07 (t, 2H, NCH_2), 3.23 (t, 2H, pyrrole CH_2), 2.63 (p, 2H, $CH_2CH_2CH_2$)
					27					
2fB or **A**	-$(CH_2)_3$-		Ph	H		192.0-194.0 (Ethanol)	$C_{17}H_{12}BrNO_2$ (342.2)	341/343	1655 (C=O) 1635 (C=O)	7.62 (m, 2H), 7.48 (m, 3H), 7.25 (s, 1H, quinone H), 4.04 (t, 2H, NCH_2), 3.19 (t, 2H, pyrrole CH_2), 2.61 (p, 2H, $CH_2CH_2CH_2$)

Table 2

Physical and Analytical Data for Compounds **4**, **5** and **6**



4



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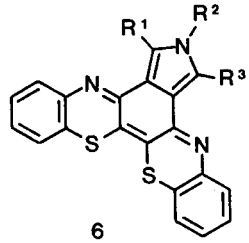

6

Compound	R¹	R²	R³	R⁴	Yield (%)	Mp °C (recrystallized)	Molecular Formula	Elemental Analyses (%) Found/(Calcd.) C	H	N	S
4a	Ph	Me	Ph	Me	89	246.0-247.5 (Benzene-Chloroform)	$C_{28}H_{20}N_2OS$ (432.5)	77.87 (77.75)	4.53 (4.66)	6.34 (6.48)	7.32 (7.41)
4b	Ph	Me	Ph	Ph	45 (from **2b**) 60 (from **1b**)	294.0-295.0 (Acetone)	$C_{33}H_{22}N_2OS$ (494.6)	80.21 (80.14)	4.29 (4.48)	5.67 (5.66)	6.50 (6.48)
4c	Ph	Me	Ph	H	39	253.0-256.1 (Acetone-Ethanol)	$C_{27}H_{18}N_2OS + H_2O$ (418.5 + 18.0)	74.61 (74.29) 418.1156 [a] (418.1141)	4.44 (4.62)	6.77 (6.42)	7.12 (7.35)
4eA or **B**	-(CH₂)₃-		Ph	Ph	12 (from **1eA** or **B**)	233.5-235.0 (Acetone-Ethanol)	$C_{29}H_{20}N_2OS$ (444.6)	78.64 (78.35)	4.28 (4.53)	5.93 (6.30)	
4eB or **A**	Ph	-(CH₂)₃-		Ph	10 (from **1eB** or **A**)	286.5-288.0 (Acetone-Ethanol)	$C_{29}H_{20}N_2OS$ (444.6)	78.27 (78.35)	4.49 (4.53)	5.98 (6.30)	
4fA or **B**	Ph	-(CH₂)₃-		H	11 (from **2fA** or **B**)	244.2-247.0 (Acetone-Ethanol)	$C_{23}H_{16}N_2OS$ (368.5)	74.84 (74.97)	4.43 (4.38)	7.33 (7.60)	
4fB or **A**	-(CH₂)₃-		Ph	H	10 (from **2fB** or **A**)	235.1-238.0 (Acetone-Ethanol)	$C_{23}H_{16}N_2OS$ (368.5)	74.63 (74.97)	4.43 (4.38)	7.48 (7.60)	
5a	Ph	Me	Ph	Me	80	244.0-245.0 (Ethanol)	$C_{28}H_{22}N_2OS$ (434.6)	77.18 (77.39)	4.95 (5.10)	6.72 (6.45)	7.39 (7.38)
6c	Ph	Me	Ph	—	3	280.0-282.5 (Ethanol)	$C_{33}H_{21}N_3S_2$ (523.7)	76.22 (75.69) 523.1186 [a] (523.1179)	4.35 (4.04)	7.58 (8.02)	

[a] Exact mass spectra were measured at Kyoto Pharmaceutical University.

isomers, 5-methyl-1,2-trimethylene-3-phenyl- (**1dA** or **1dB**) and 5-methyl-2,3-trimethylene-1-phenyl-2*H*-isoindole-4,7-dione (**1dB** or **1dA**), 1,2-trimethylene-3,5-diphenyl- (**1eA** or **1eB**) and 2,3-trimethylene-1,5-diphenyl-2*H*-isoindole-4,7-dione (**1eB** or **1eA**) as well as 2,3-trimethylene-1-phenyl- (**2fA** or **2fB**) and 1,2-trimethylene-3-phenyl-2*H*-isoindole-4,7-dione (**2fB** or **2fA**).

From the mixture of **1dA** and **1dB**, **1dA** was isolated by the column chromatography as the first fraction, showing a quartet at δ 6.53 for vinylic hydrogen in the nmr spectrum. From the second fraction **1dB** was obtained, exhibiting a quartet at δ 6.56. Similarly **1eA** and **2fA** were isolated as the first fraction from the mixtures of their isomers, showing singlets at δ 6.80 and δ 7.22 for vinylic hydrogen respectively. The nmr spectra of **1eB** and **2fB** for vinylic hydrogen revealed singlets at δ 6.82 and δ 7.25. The assignment of the structures for **A** or **B** of **1d**, **1e** and **2f** is subject to further investigation.

Because of the reaction of **1dA** and **1dB** with bromine in acetic acid produced a variety of brominated compounds, subsequent condensation with **3** are still under examination. The condensations of **1eA** and **1eB** with **3** were carried out in the presence of 15% hydrochloric acid to afford **4eA** (1,2-trimethylene-3,5-diphenyl- or 2,3-trimethylene-1,5-diphenyl-4*H*-pyrrolo[3,4-*a*]phenothiazin-4-one) and **4eB** respectively. Compounds **2fA** and **2fB** reacted with **3** in the presence of potassium acetate giving **4fA** (2,3-trimethylene-1-phenyl- or 1,2-trimethylene-3-phenyl-4*H*-pyrrolo[3,4-*a*]phenothiazin-4-one) and **4fB** respectively. The formation of 6,7-trimethylene-8-phenylpyrrolo[3,4-*a*][1,4]benzothiazino[3,2-*c*]phenothiazine (**6f**) was inferred from its mass spectrum.

The structures of the compounds **4**, **5** and **6** were determined by elemental analyses and from their spectroscopic

Table 3

Spectroscopic Data for Compounds **4**, **5** and **6**

Compound	Mass (M^+)	IR (cm^{-1})	UV (Methanol) λ max (nm) (log ε)	^{1}H-NMR (Deuteriochloroform) (δ)
4a	432	1618 (C=O)	477 (4.22), 374sh (3.90), 327 (4.11), 259 (4.62)	7.56 (m, 9H), 7.40-7.26 (m, 2H), 7.20 (m, 2H), 7.01 (m, 1H), 3.41 (s, 3H, NCH_3), 2.07 (s, 3H, iminoquinone CH_3)
4b	494	1622 (C=O)	487 (4.08), 377sh (3.80), 329 (4.01), 259 (4.54)	7.68-7.52 (m, 8H), 7.47 (m, 4H), 7.34-7.26 (m, 4H), 7.22-7.10 (m, 2H), 6.99 (m, 1H), 3.45 (s, 3H, NCH_3)
4c	418	1615 (C=O)	486 (4.20), 377sh (3.94), 324 (4.09), 257 (4.67)	7.70-7.42 (m, 10H), 7.39 (s, 1H), 7.20 (m, 2H), 7.01 (m, 1H), 6.51 (s, 1H, iminoquinone H), 5.14 (br, 2H, H_2O), 3.42 (s, 3H, NCH_3)
4eA or **B**	444	1615 (C=O)	495 (4.20), 374 (3.91) 331 (3.91), 260 (4.60)	7.72 (m, 2H), 7.58-7.32 (m, 10H), 7.22 (m, 2H), 4.20 (t, 2H, NCH_2), 3.50 (t, 2H, pyrrole CH_2), 2.70 (p, 2H, $CH_2CH_2CH_2$)
4eB or **A**	444	1615 (C=O)	497 (4.13), 372 (3.97), 325 (4.06), 273sh (4.46), 244 (4.59)	7.75 (m, 2H), 7.61-7.33 (m, 8H), 7.19 (m, 4H), 4.09 (t, 2H, NCH_2), 3.30 (t, 2H, pyrrole CH_2), 2.60 (p, 2H, $CH_2CH_2CH_2$)
4fA or **B**	368	1605 (C=O)	492 (4.20), 375 (3.90), 321 (3.88), 257 (4.60)	7.83-7.60 (m, 4H), 7.55-7.36 (m, 5H), 6.72 (s, 1H, iminoquinone H), 4.16 (t, 2H, NCH_2), 3.46 (m, 2H, pyrrole CH_2), 2.66 (m, 2H, $CH_2CH_2CH_2$)
4fB or **A**	368	1610 (C=O)	494 (4.14), 373 (3.97), 323 (3.98), 283sh, 243 (4.56)	7.73 (m, 2H), 7.51 (m, 3H), 7.39 (s, 1H), 7.32-7.21 (m, 3H), 6.86 (s, 1H, iminoquinone H), 4.13 (t, 2H, NCH_2), 3.36 (t, 2H, pyrrole CH_2), 2.64 (m, 2H, $CH_2CH_2CH_2$)
5a	434	1673 (C=O)	489 (3.14), 367 (4.23), 345sh, 258 (4.60)	7.53 (m, 10H), 7.27-7.20 (m, 1H), 7.09 (m, 2H), 6.86 (m, 1H), 3.38 (s, 3H, NCH_3), 3.21 (d, J = 16 Hz, 1H, iminoquinone H), 2.86 (d, J = 16 Hz, 1H, SH), 1.35 (s, 3H, iminoquinone CH_3)
6c	523	1642 (C=N)	501, 375, 333, 255	7.75-7.35 (m, 14H), 7.20-7.00 (m, 4H), 3.17 (s, 3H, NCH_3)

data. The reactions investigated are summarized in Scheme I. The analytical and spectral data for the compounds obtained in these reactions are listed in Tables 1, 2 and 3.

EXPERIMENTAL

Melting points were determined on a Yanaco micro-melting point apparatus and are uncorrected. The infrared spectra were taken on a JASCO A-102 spectrometer using potassium bromide disks and the ultraviolet spectra were recorded with a JASCO UVIDEC-505. The nuclear magnetic resonance spectra were measured on a Varian XL-200 spectrometer, using tetramethylsilane as the internal standard. Mass spectra were obtained with a Hitachi M-52 or ESCO EMD-05B spectrometer. For column chromatography, aluminium oxide 90 (Merck, 70-230 mesh ASTM) and silica gel (Kieselgel 60, Merck, 70-230 mesh ASTM and Mallinckrodt, 100 mesh) were used. 2-Aminobenzenethiol and 2-methyl-1,4-benzoquinone purchased from Tokyo Kasei Kogyo Co., Ltd.

1,2,3,5-Tetrasubstituted 2*H*-Isoindole-4,7-diones (**1**).

Compounds **1** were synthesized by the method for the preparation of 2,5-dimethyl-1,3-diphenyl-2*H*-isoindole-4,7-dione (**1a**) [4]. 2,3,4-Trisubstituted oxazolium 5-oxides were generated in solution and were reacted with 2-methyl-1,4-benzoquinone or 2-phenyl-1,4-benzoquinone [5] respectively.

Compound **1b**.

Anal. Calcd. for $C_{27}H_{19}NO_2$: C, 83.27; H, 4.92; N, 3.60. Found: C, 82.90; H, 4.92; N, 3.77.

Compound **1eA**.

Anal. Calcd. for $C_{23}H_{17}NO_2$: C, 81.40; H, 5.05; N, 4.13. Found: C, 81.62; H, 4.81; N, 3.85.

Compound **1eB**.

Anal. Calcd. for $C_{23}H_{17}NO_2$: C, 81.40; H, 5.05; N, 4.13. Found: C, 81.76; H, 4.77; N, 3.90.

1,2,3,5-Tetrasubstituted 6-Bromo-2*H*-isoindole-4,7-diones (**2**).

To a solution of **1** (10 mmoles) in 50 ml of acetic acid were added 0.8 ml (15 mmoles) of bromine and 3.3 g (40 mmoles) of sodium acetate. The mixture was allowed to stand in the dark at room temperature for several days. After the addition of water to the dark brown mixture, separated product was filtered and recrystallized or column chromatographed.

Compound **2a**.

Anal. Calcd. for $C_{22}H_{16}BrNO_2$: C, 65.04; H, 3.97; N, 3.45. Found: C, 65.32; H, 4.11; N, 3.29.

Compound **2b**.

Anal. Calcd. for $C_{27}H_{18}BrNO_2$: C, 69.24; H, 3.87; N, 2.99. Found: C, 69.34; H, 3.82; N, 2.80.

1,2,3-Trisubstituted 6-bromo-2*H*-isoindole-4,7-diones **2c** and **2f**.

2,3,4-Trisubstituted oxazolium 5-oxides were reacted with 2-bromo-1,4-benzoquinone [6] in the manner of the preparation of **1**.

Compound **2c**.

Anal. Calcd. for $C_{21}H_{14}BrNO_2$: C, 64.30; H, 3.60; N, 3.57. Found: C, 64.48; H, 3.64; N, 3.37.

Compound **2fA**.

Anal. Calcd. for $C_{17}H_{12}BrNO_2$: C, 59.67; H, 3.53; N, 4.09. Found: C, 59.31; H, 3.40; N, 3.72.

Compound **2fB**.

Anal. Calcd. for $C_{17}H_{12}BrNO_2$: C, 59.67; H, 3.53; N, 4.09. Found: C, 59.28; H, 3.49; N, 3.88.

2,5-Dimethyl-1,3-diphenyl-7-(2-mercaptophenyl)imino-2*H*-isoindol-4-one (**5a**).

To a solution of 1 mmole of **1a** in 35 ml of ethanol, 1 mmole of **3** in 8 ml of 15% hydrochloric acid was added and the resulting mixture was stirred at room temperature for 15 hours. The resulting solid was filtered and recrystallized from ethanol.

2,5-Dimethyl-1,3-diphenyl-4*H*-pyrrolo[3,4-*a*]phenothiazin-4-one (**4a**).

To a solution of 1 mmole of **2a** in 20 ml of ethanol-benzene (4:1) 0.1 g of potassium acetate and 1 mmole of **3** were added and the resulting mixture was stirred at room temperature for 2-3 hours. After the mixture was evaporated *in vacuo* the residue was recrystallized from benzene-chloroform.

2-Methyl-1,3,5-triphenyl-4*H*-pyrrolo[3,4-*a*]phenothiazin-4-one (**4b**).

Method A.

From **2b** and **3** in a similar manner as for the preparation of **4a**, **4b** was obtained. By the column chromatography on aluminium oxide using benzene as the eluent **4b** was purified.

Method B.

From **1b** and **3** in ethanol-benzene in the presence of 15% hydrochloric acid by stirring for 5-15 hours as the preparation of **5a**, **4b** was produced instead of **5b**.

2-Methyl-1,3-diphenyl-4*H*-pyrrolo[3,4-*a*]phenothiazin-4-one (**4c**).

From the reaction of **2c** (1 mmole) with **3** (1 mmole) in a manner similar to the preparation of **4a**, **4c** was produced and was purified by the column chromatography with silica gel using benzene-ethyl acetate (97:3) as the eluent. On the above chromatography, 7-methyl-6,8-diphenylpyrrolo[3,4-*a*][1,4]benzothiazino[3,2-*c*]phenothiazine (**6c**) was obtained from the first reddish purple fraction.

1,2-Trimethylene-3,5-diphenyl- and 2,3-trimethylene-1,5-diphenyl-4*H*-pyrrolo[3,4-*a*]phenothiazin-4-one **4eA** or **4eB**.

From **1eA** or **1eB** (1 mmole) with **3** (1 mmole) by method B for the preparation of **4b** at about 60°, stirring for 24 hours, **4eA** or **4eB** was obtained respectively.

2,3-Trimethylene-1-phenyl- and 1,2-trimethylene-3-phenyl-4*H*-pyrrolo-[3,4-*a*]phenothiazin-4-one **4fA** or **4fB**.

From **2fA** or **2fB** (1 mmole) with **3** (1 mmole) in the same way as for the preparation of **4c** by stirring for 1 hour, **4fA** or **4fB** was produced.

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