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# Synthesis of new basic dimethine dyes containing a phenothiazine moiety

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Condensation of 1-carbamoylmethyl-2,3,3-trimethyl-3*H*-indolium chloride with *N*-alkylphenothiazine-3-carboxaldehydes in acetic acid and following work-up of the reaction mixture with base afforded 9a-[2-(phenothiazin-3-yl)ethenyl]imidazo[1,2-*a*]indol-2-ones as new basic dimethine dyes. 10a-[2-(Phenothiazin-3-yl)ethenyl]pyrimido[1,2-*a*]indol-2-ones were obtained by reaction of the mentioned heterocyclic aldehyde with 10,10,10a-trimethylpyrimido[1,2-*a*]indol-2-one derivatives.

**Key words**: 3*H*-indole, imidazo[1,2-*a*]indole, pyrimido[1,2-*a*]indole, phenothiazine, basic dimethine dyes

## INTRODUCTION

Reaction of 2,3,3-trimethyl-3H-indole with such bifunctional alkylating agents as 2-bromoethanol or ethylene oxide produces indolo[1,2-*b*]oxazole derivatives whose products of condensation with aromatic aldehydes are basic dimethine dyes [1, 2]. These styryl-like dyes showed a colour-developing and bleaching behaviour and found application as colour formers in information registration processes or basic dyes for textile materials [3–7].

We previously reported the synthesis of similar basic dimethine dyes by condensation of imidazo- and pyrimido[1,2-*a*]indole derivatives with various benzaldehydes, indole-3- and carbazole-3-carboxaldehydes [8–10]. This report deals with preparation of new styryl-like colour formers by condensation of 1-carbamoylalkyl-2,3,3-trimethyl-3*H*-indolium salts or their cyclic bases with *N*-alkylphenothiazine-3-carboxaldehydes.

## RESULTS AND DISCUSSION

The starting 1-carbamoylmethyl-3*H*-indolium chlorides **1a,b** were prepared by alkylation of 2,3,3-trimethyl- and 2,3,3,5-tetramethyl-3*H*-indole with 2-chloroacetamide by the method described in [11]. Condensation of **1a, b** with 10-methyl- and 10-ethylphenothiazine-3-carboxaldehydes was carried out in glacial acetic acid at 100 °C. Treatment of the formed styrylic derivatives **2a–d** with potassium hydroxide afforded 9a-[2-(phenothiazin-3-yl)ethenyl]imidazo[1,2-*a*]indol-2-ones **3a–d** (Table 1). The structure of compounds **3a**–**d** was confirmed by spectral investigations.

The IR spectra of compounds **3a–d** contain absorption bands characteristic for five-membered lactams [12] at 3162–3219 (N-H) and 1688–1705 (C = O) $cm^{-1}$ (Table 2). The $^1$H NMR spectra of compounds **3a–d** are characterized by the presence of two singlets of diastereotopic 9,9-methyl groups in the area of 1.04–1.38 and AB-quadruplet ($^2J_{AB}$ = 16.2–16.5 Hz) of the $NCH_2CO$ moiety protons in the area of 3.37–3.78 ppm. Protons of the vinyl bridge give two doublets in the area of 6.35–6.71 ppm. The spin–spin coupling constant of vicinal ethene protons is 15.9 Hz and attests to their *trans*-orientation. The singlet of the *N*-methyl group of the phenothiazine moiety (for **3a, c**) is present at 3.30, while the corresponding *N*-ethyl group (for **3b, d**) gives a triplet and a quadruplet at 1.31 and 3.92 ppm, respectively. In the $^{13}$C NMR spectrum of the compound **3a**, signals of carbon atoms of the imidazolidinone ring are present at 57.17 (C-3), 95.66 (C-9a) and 176.35 ppm (C = O), while signals of methyl groups are situated at 24.59 (9-$CH_3$), 33.36 (9-$CH_3$) and 37.88 (N-$CH_3$) ppm. Eighteen $sp^2$-hybridized carbons of aromatic rings and two ethene carbons give overlapping signals in the area of 115.16–153.52 ppm. The $^{13}$C NMR spectra of compounds **3b–d** have a similar character.

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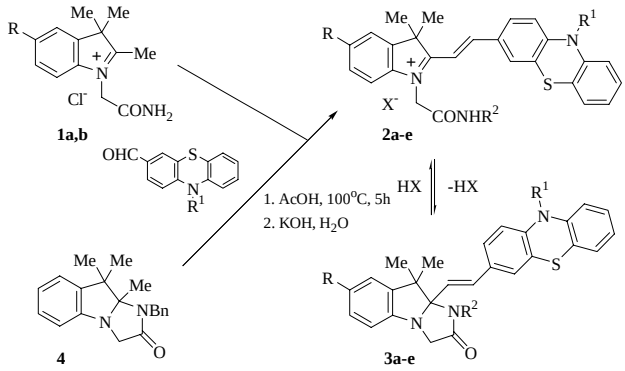
Table 1. **9a-[2-(10-Alkylphenothiazin-3-yl)ethenyl]-1,2,3,9a-tetrahydroimidazo[1,2-*a*]indol-2(9*H*)-ones (3 a–e) and 10a-[2-(10-alkylphenothiazin-3-yl)ethenyl]-1,2,3,4,10,10a-hexahydropyrimido[1,2-*a*]indol-2-ones (7a–d)**

| Compound | Empirical formula | Found,% / Calculated, % | | | m.p., °C (solvent for crystallization) | Yield, % |
|---|---|---|---|---|---|---|
| | | C | H | N | | |
| **3a** | $C_{27}H_{25}N_3OS$ | 73.81 / 73.77 | 5.67 / 5.73 | 8.71 / 9.56 | 196–197 (acetone) | 24 |
| **3b** | $C_{28}H_{27}N_3OS$ | 74.02 / 74.14 | 5.82 / 6.00 | 8.91 / 9.26 | 205–206 (DMF) | 33 |
| **3c** | $C_{28}H_{27}N_3OS$ | 74.07 / 74.14 | 6.45 / 6.00 | 8.89 / 9.26 | 212–213 (acetone) | 24 |
| **3d** | $C_{29}H_{29}N_3OS$ | 74.21 / 74.48 | 6.02 / 6.25 | 8.51 / 8.98 | 130–131 (acetone) | 27 |
| **3e** | $C_{35}H_{33}N_3OS$ | 77.01 / 77.31 | 6.28 / 6.12 | 7.43 / 7.73 | 134–135 (acetone) | 43 |
| **7a** | $C_{28}H_{27}N_3OS$ | 73.80 / 74.14 | 6.40 / 6.00 | 9.14 / 9.26 | 256–257 (DMF) | 66 |
| **7b** | $C_{29}H_{29}N_3OS$ | 74.28 / 74.48 | 6.05 / 6.25 | 8.48 / 8.98 | 257–258 (DMF) | 68 |
| **7c** | $C_{29}H_{29}N_3OS$ | 74.20 / 74.48 | 6.50 / 6.25 | 8.52 / 8.98 | 251–252 (DMF) | 76 |
| **7d** | $C_{30}H_{31}N_3OS$ | 74.61 / 74.81 | 6.54 / 6.49 | 8.38 / 8.72 | 273–274 (DMF) | 35 |

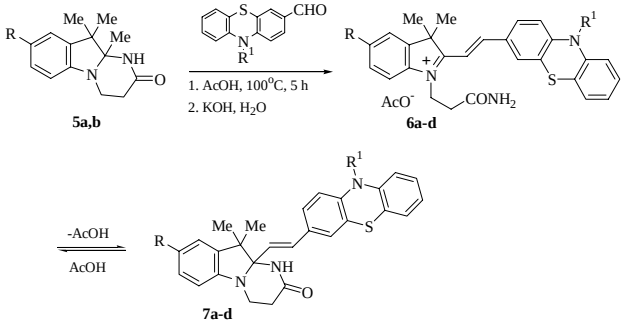
Scheme 1




**1a** R = H, **b** R = Me; **2, 3a** R = $R^2$ = H, $R^1$ = Me; **b** R = $R^2$ = H, $R^1$ = Et; **c** R = $R^1$ = Me, $R^2$ = H; **d** R = Me, $R^1$ = H, $R^2$ = Et; **e** R = H, $R^1$ = Et, $R^2$ = Bn; **2 a–d** X = Cl; **2e** X = AcO

When 10-ethylphenothiazine-3-carboxaldehyde was condensed with 1-benzylimidazo[1,2-*a*]indol-2-one **4** in acetic acid, formation of coloured acetate **2e** took place. Work-up of the reaction mixture with a strong base gave the target cyclic product **3e**. The IR spectrum of the compound **3e** contains a carbonyl group absorption band in the area of 1693 $cm^{-1}$, and no absorption band characteristic of the N–H bond is observed. The presence of the benzyl group in the molecule of **3e** was confirmed by observation of the AB-quadruplet of benzylic methylene protons at 4.50–4.77 ppm ($^2J_{AB}$ = 16.5 Hz) in the $^1$H NMR spectrum and the signal of the corresponding methylene carbon at 51.39 ppm in the $^{13}$C NMR spectrum.

Scheme 2




**5a** R = H, **b** R = Me; **6, 7a** R = H, $R^1$ = Me; **b** R = H, $R^1$ = Et; **c** R = $R^1$ = Me; **d** R = Me, $R^1$ = Et

10a-[2-(Phenothiazin-3-yl)ethenyl]pyrimido[1,2-*a*]indol-2-ones **7a–d** were synthesized by reaction of 10-methyl- and 10-ethylphenothiazine-3-carboxaldehydes with pyrimido[1,2-*a*]indol-ones **5a, b**. The starting **5a, b** were obtained by quaternization of 2,3,3-trimethyl-3*H*-indole with acrylamide [8]. Basification of the reaction mixtures containing intermediate coloured acetates **6a–d** with a solution of potassium hydroxide afforded the target products **7a–d** in moderate yields.

In the IR spectrum of compounds **7a–d** containing the annelated six-membered lactam ring the absorption band of the C=O group is shifted by approximately 20 $cm^{-1}$ towards the lower values in comparison with the corresponding band of the five-membered lactam derivatives **3a–e**. In the $^1$H NMR spectra the methylene protons of the relatively rigid he-

Table 2. **IR, $^1$H and $^{13}$C NMR spectra of 9a-[2-(10-alkylphenothiazin-3-yl)ethenyl]-1,2,3,9a-tetrahydroimidazo[1,2-*a*]indol-2(9*H*)-ones (3a–e) and 10a-[2-(10-alkylphenothiazin-3-yl)ethenyl]-1,2,3,4,10,10a-hexahydropyrimido[1,2-*a*]indol-2-ones (7a–d)**

| Compound | ν, $cm^{-1}$ | Chemical shifts, ppm |
|---|---|---|
| **3a** | 1705 (C=O), 3213 (NH) | $^1$H NMR: 1.05 (3H, s, 9-$CH_3$); 1.35 (3H, s, 9-$CH_3$); 3.30 (3H, s, N-$CH_3$); 3.37–3.76 (2H, AB-q, *J* = 16.2 Hz, $NCH_2$); 6.37 (1H, d, *J* = 15.9 Hz, CH-ethenyl); 6.69 (1H, d, *J* = 15.9 Hz, CH-ethenyl); 6.89–7.34 (11H, m, Ar-H); 8.85 (1H, br.s, NH). $^{13}$C NMR: 24.59 (9-$CH_3$), 33.36 (9-$CH_3$), 37.88 (N-$CH_3$), 49.92 (C-9), 57.17 (C-3), 95.66 (C-9a), 115.16-153.52 (18xC-Ar and 2xC-ethenyl), 176.35 (C=O). |
| **3b** | 1689 (C=O), 3214 (NH) | $^1$H NMR: 1.07 (3H, s, 9-$CH_3$); 1.31 (3H, t, *J* = 6.6 Hz, $CH_2CH_3$); 1.38 (3H, s, 9-$CH_3$); 3.58–3.78 (2H, AB-q, *J* = 16.2 Hz, $NCH_2CO$); 3.92 (2H, q, *J* = 6.6 Hz, $CH_2CH_3$); 6.38 (1H, d, *J* = 15.9 Hz, CH-ethenyl); 6.71 (1H, d, *J* = 15.9 Hz, CH-ethenyl); 6.91-7.34 (11H, m, Ar-H); 8.87 (1H, br.s, NH). $^{13}$C NMR: 13.29 ($CH_2CH_3$), 22.61 (9-$CH_3$), 28.67 (9-$CH_3$), 41.89 ($CH_2CH_3$), 47.94 (C-9), 55.19 (C-3), 93.71 (C-9a), 113.21–151.56 (18xC-Ar and 2xC-ethenyl), 174.40 (C=O). |
| **3c** | 1688 (C=O), 3162 (NH) | $^1$H NMR: 1.04 (3H, s, 9-$CH_3$); 1.33 (3H, s, 9-$CH_3$); 2.23 (3H, s, 7-$CH_3$); 3.30 (3H, s, N-$CH_3$); 3.52–3.69 (2H, AB-q, *J* = 16.5 Hz, $NCH_2$); 6.35 (1H, d, *J* = 15.9 Hz, CH-ethenyl); 6.68 (1H, d, *J* = 15.9 Hz, CH-ethenyl); 6.76–7.34 (10H, m, Ar-H); 8.80 (1H, br.s, NH). $^{13}$C NMR: 23.29 (7-$CH_3$), 24.51 (9-$CH_3$), 30.62 (9-$CH_3$), 37.88 (N-$CH_3$), 49.90 (C-9), 57.31 (C-3), 95.87 (C-9a), 114.94–151.24 (18xC-Ar and 2xC-ethenyl), 176.37 (C=O). |
| **3d** | 1705 (C=O), 3219 (NH) | $^1$H NMR: 1.05 (3H, s, 9-$CH_3$); 1.31 (3H, t, *J* = 6.6 Hz, $CH_2CH_3$); 1.34 (3H, s, 9-$CH_3$); 2.25 (3H, s, 7-$CH_3$); 3.52-3.69 (2H, AB-q, *J* = 16.2 Hz, $NCH_2CO$); 3.92 (2H, q, *J* = 6.6 Hz, $CH_2CH_3$); 6.35 (1H, d, *J* = 15.9 Hz, CH-ethenyl); 6.67 (1H, d, *J* = 15.9 Hz, CH-ethenyl); 6.78–7.32 (10H, m, Ar-H); 8.80 (1H, br.s, NH). $^{13}$C NMR: 13.29 ($CH_2CH_3$), 21.34 (7-$CH_3$), 22.54 (9-$CH_3$), 28.68 (9-$CH_3$), 41.09 ($CH_2CH_3$), 47.94 (C-9), 55.34 (C-3), 93.92 (C-9a), 113.00-149.29 (18xC-Ar and 2xC-ethenyl), 174.44 (C=O). |
| **3e** | 1693 (C=O) | $^1$H NMR: 1.10 (3H, s, 9-$CH_3$); 1.27 (3H, s, 9-$CH_3$); 1.28 (3H, t, *J* = 6.6 Hz, $CH_2CH_3$); 3.87–4.08 (2H, AB-q, *J* = 16.5 Hz, $NCH_2CO$); 3.88 (2H, q, *J* = 6.6 Hz, $CH_2CH_3$); 4.50-4.77 (2H, AB-q, *J* = 16.5 Hz, $CH_2C_6H_5$); 6.59–6.76 (2H, AB-q, *J* = 16.0 Hz, CH=CH); 6.90–7.26 (16H, m, Ar-H). $^{13}$C NMR: 15.24 ($CH_2CH_3$), 25.99 (9-$CH_3$), 30.45 (9-$CH_3$), 43.81 ($CH_2CH_3$), 48.06 (C-9), 51.39 ($CH_2$-benzyl), 56.29 (C-3), 99.24 (C-9a), 115.06-152.54 (24xC-Ar and 2xC-ethenyl), 175.71 (C = O). |
| **7a** | 1658 (C=O), 3193 (NH) | $^1$H NMR: 1.07 (3H, s, 10-$CH_3$); 1.31 (3H, s, 10-$CH_3$); 1.92–2.08 (1H, m, 1/2$CH_2$); 2.15–2.35 (1H, m, 1/2$CH_2$); 3.35 (3H, s, N-$CH_3$); 3.38-3.52 (1H, m, 1/2$CH_2$); 3.76–3.86 (1H, m, 1/2$CH_2$); 6.39 (1H, d, *J* = 16.5 Hz, CH-ethenyl); 6.71 (1H, d, *J* = 16.5 Hz, CH-ethenyl); 6.78–7.35 (11H, m, Ar-H); 7.92 (1H, br.s, NH). |
| **7b** | 1659 (C=O), 3181 (NH) | $^1$H NMR: 1.08 (3H, s, 10-$CH_3$); 1.34 (3H, s, 10-$CH_3$); 1.34 (3H, t, *J* = 6.6 Hz, $CH_2CH_3$); 1.88–2.07 (1H, m, 1/2$CH_2$); 2.27–2.45 (1H, m, 1/2$CH_2$); 3.26–3.40 (1H, m, 1/2$CH_2$); 3.74-3.84 (1H, m, 1/2$CH_2$); 3.93 (2H, q, *J* = 6.6 Hz, $CH_2CH_3$); 6.31 (1H, d, *J* = 16.0 Hz, CH-ethenyl); 6.69 (1H, d, *J* = 16.0 Hz, CH-ethenyl); 6.75-7.29 (11H, m, Ar-H); 7.42 (1H, br.s, NH). |
| **7c** | 1660 (C=O), 3195 (NH) | $^1$H NMR: 1.08 (3H, s, 10-$CH_3$); 1.29 (3H, s, 10-$CH_3$); 1.93–2.00 (1H, m, 1/2$CH_2$); 2.18–2.37 (1H, m, 1/2$CH_2$); 2.24 (3H, s, 8-$CH_3$); 3.18–3.32 (1H, m, 1/2$CH_2$); 3.34 (3H, s, N-$CH_3$); 3.70–3.81 (1H, m, 1/2$CH_2$); 6.36 (1H, d, *J* = 15.9 Hz, CH-ethenyl); 6.70 (1H, d, *J* = 15.9 Hz, CH-ethenyl); 6.71–7.32 (10H, m, Ar-H); 7.84 (1H, br.s, NH). $^{13}$C NMR: 25.57, 26.10 (8-$CH_3$, 10-$CH_3$), 32.66 (10-$CH_3$), 40.70, 42.42 (C-3, $NCH_3$), 54.87 (C-4, C-10), 92.35 (C-10a), 114.46–150.39 (18xC-Ar and 2xC-ethenyl), 174.20 (C=O). |
| **7d** | 1659 (C=O), 3193 (NH) | $^1$H NMR: 1.03 (3H, s, 10-$CH_3$); 1.27 (3H, s, 10-$CH_3$); 1.32 (3H, t, *J* = 6.6 Hz, $CH_2CH_3$); 2.23 (3H, s, 7-$CH_3$); 1.94–2.02 (1H, m, 1/2$CH_2$); 2.16–2.35 (1H, m, 1/2$CH_2$); 3.16–3.32 (1H, m, 1/2$CH_2$); 3.68–3.79 (1H, m, 1/2$CH_2$); 3.91 (2H, q, *J* = 6.6 Hz, $CH_2CH_3$); 6.33 (1H, d, *J* = 16.2 Hz, CH-ethenyl); 6.68 (1H, d, *J* = 16.2 Hz, CH-ethenyl); 6.71–7.28 (10H, m, Ar-H); 7.82 (1H, br.s, NH). |

Table 3. **Electronic spectra of 9a-[2-(10-alkylphenothiazin-3-yl)ethenyl]-1,2,3,9a-tetrahydroimidazo[1,2-*a*]indol-2(9*H*)-ones (3a–e) and 10a-[2-(10-alkylphenothiazin-3-yl)ethenyl]-1,2,3,4,10,10a-hexahydropyrimido[1,2-*a*]indol-2-ones (7a–d)**

| Compound | $\lambda_{max}$ (lg ε) | |
|---|---|---|
| | acetonitrile | acetic acid |
| **3a** | 204 (4.63); 238 (4.35); 268 (4.51) | 250 (4.22); 398 (4.06); 562 (4.33) |
| **3b** | 204 (4.64); 238 (4.38); 270 (4.50) | 252 (4.17); 400 (3.99); 574 (4.23) |
| **3c** | 204 (4.65); 238 (4.38); 268 (4.53) | 248 (4.31); 400 (4.13); 558 (4.42) |
| **3d** | 206 (4.66); 238 (4.43); 270 (4.54) | 252 (4.37); 402 (4.21); 570 (4.48) |
| **3e** | 204 (4.66); 240 (4.42); 270 (4.53) | 254 (4.35); 400 (4.21); 574 (4.47) |
| **7a** | 204 (3.98); 242 (3.67); 268 (3.78) | 250 (4.10); 394 (3.97); 558 (4.21) |
| **7b** | 204 (3.83); 242 (3.50); 270 (3.62) | 254 (4.32); 398 (4.20); 570 (4.46) |
| **7c** | 206 (4.56); 242 (4.26); 268 (4.40) | 246 (4.18); 400 (3.99); 554 (4.27) |
| **7d** | 206 (4.11); 242 (3.80); 270 (3.91) | 252 (4.23); 400 (4.08); 566 (4.35) |

xahydropyrimidinone cycle give four complex multiplets in the area of 1.88–3.86 ppm. The *trans*-configuration of the molecules is confirmed by the value of the spin–spin coupling constant of the ethene moiety protons ($^3J$ = 15.9–16.5 Hz). The $^{13}$C NMR spectrum of the compound **7c** exibits a signal of the indole α-carbon (C-10a) at 92.35 ppm.

Solutions of compounds **3a–e** and **7a–d** in acetonitrile or ethanol do not have absorption peaks in a visible part of the electronic spectra. However, the action of organic or inorganic protic acids on the mentioned compounds leads to opening of the lactam ring and formation of coloured 3*H*-indolium salts which possess a long chain of conjugated double bonds. For example, electronic spectra of the solutions of compounds **3a, e** and **7a** in acetic acid exhibit $\lambda_{max}$ at 562, 574 and 558 nm, respectively (Table 3).

## EXPERIMENTAL

Melting points were determined on a *Kleinfeld* melting point apparatus. IR spectra were recorded on a Perkin Elmer Spectrum BXII spectrophotometer using KBr pellets. The UV spectra were determined with a Spectronic Genesys 8 spectrophotometer. $^1$H NMR spectra were recorded at 300 MHz and $^{13}$C NMR spectra were recorded at 75 MHz on a Varian Gemini 2000 instrument. Tetramethylsilane was used as the internal standard. For thin layer chromatographic (TLC) analyses, Merck precoated TLC plates (silica gel 60 F254) were used.

**General method for the synthesis of 9a-[2-(10-alkylphenothiazin-3-yl)ethenyl]-1,2,3,9a-tetrahydro-9,9-dimethylimidazo[1,2-*a*]indol-2(9*H*)-ones (3 a–e) and 10a-[2-(10-alkylphenothiazin-3-yl)ethenyl]-1,2,3,4,10,10a-hexahydro-10,10-dimethylpyrimido[1,2-*a*]indol-2-ones (7a–d)**. A solution of 1-carbamoylmethyl-3H-indolium chloride (**1a, b**) (3 mmol), or 1-benzylimidazo[1,2-*a*]indol-2-one (**4**), or pyrimido[1,2-*a*]indol-2-one (**5a, b**) and 10-methyl- or 10-ethylphenothiazine-3-carboxaldehyde (3 mmol) in glacial acetic acid (6 ml) was heated at 100 °C for 5 h. Then the reaction mixture was poured into water (50 ml), the solution made alkaline with 10% potassium hydroxide, extracted with ether (20 ml) and the mixture allowed to stay at 5 °C for 24 h. Then the precipitated substance was filtered off, washed with water (20 ml), and crystallized from acetone or DMF to afford a yellowish crystalline material. Yields, solvents for crystallization and data of elemental analysis of the synthesized new compounds are presented in Table 1. Data of IR, $^1$H and $^{13}$C NMR spectra are presented in Table 2. Data of electronic spectra are presented in Table 3.

## CONCLUSION

Condensation of 1-carbamoylmethyl-2,3,3-trimethyl-3*H*-indolium chloride or 1,2,3,4,10,10a-hexahydro-10,10,10a-trimethylpyrimido[1,2-*a*]indol-one with 10-alkylphenothiazine-3-carboxaldehydes in glacial acetic acid and the subsequent work-up of the reaction mixture with potassium hydroxide yields basic dimethine dyes possessing phenothiazine moiety.

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**NAUJŲ BAZINIŲ DIMETINO DAŽŲ SU FENTIAZINO ŽIEDU SINTEZĖ**

S a n t r a u k a

Kondensuojant acto rūgštyje 1-karbamoilmetil-2,3,3-trimetil-3H-indolio chloridą su *N*-alkilfenotiazin-3-karbaldehidu ir toliau apdorojus reakcijos mišinį stipriomis bazėmis, susidaro 9a-[2-(fentiazin-3-il)etenil]imidazo[1,2-*a*]indol-2-onai. 10a-[2-(Fentiazin-3-il)etenil]pirimido[1,2-*a*]indol-2-onai buvo gauti panašiu būdu iš minėtų heterociklinių aldehidų ir 10,10,10a-trimetilpirimido[1,2-*a*]indol-2-ono darinių. Tokiu būdu susintetinti nauji baziniai dimetino dažai, kurių struktūroje yra fentiazino žiedas.