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Synthesis and photochromic properties of dithienylmaleimides with sulfur-containing fragments

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A number of sulfur-containing dithienylmaleimide derivatives were synthesized. Spectral and kinetic characteristics of the compounds obtained were studied, which showed that all the dithienylethenes synthesized possessed photochromic properties.

Key words: synthesis, dithienylmaleic anhydrides, sulfur-containing dithienylmaleimides, photochromism, photocoloration, photodecoloration.

Diarylethenes are subject to the valence isomerization between two thermally stable forms: open and cyclic.[1] These compound are the most promising candidates for the development on their basis of molecular photoswitches[1,2] and carriers of information of large capacity used for recording, processing, and storage of data.[2—4] Photochromic compounds, which can be used for such a purpose, should possess a certain set of operating characteristics: the thermal stability of both isomeric forms, the high quantum yield, as well as the high fatigue resistance of phototransformations.

1,2-Dithienylethenes, in particular, the products containing maleic anhydride or maleimide fragments as the ethene bridge,[2] are one class of photochromic compounds meeting these conditions. Photocontrolled systems developed based on photochromic dithienylmaleimides and nanoparticles of noble metals are of special interest.[5] To bring into being efficient molecular interactions of photochromic compounds with nanoparticles of noble metals, the presence of a fragment providing binding of the photochromic molecule with the metal nanoparticle is necessary in the structure of photochrom. Thiol groups can serve as such fragments,[6,7] however, the synthesis of the corresponding derivatives meets certain difficulties caused by their low stability.

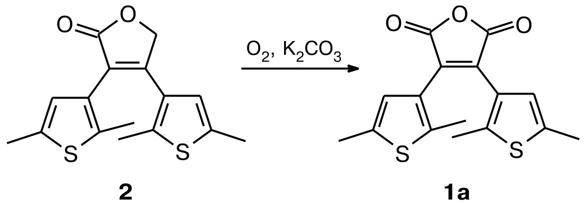
The purpose of the present work was to develop an approach to the synthesis of photochromic dithienylmaleic anhydrides with thiazole substituents, to functionalize the bridging fragment of the products obtained with sulfur-containing nitrogen nucleophiles, and to study spectral and kinetic characteristics of the compounds synthesized.

Results and Discussion

Synthesis of dithienylmaleimides with sulfide substituents in the bridging fragment. In the present work, we synthesized a number of sulfur-containing dithienylmaleimides in order to subsequently compare complexation properties of these products.

In the literature, we found descriptions of several approaches to the preparation of photochromic dithienylsubstituted maleimide derivatives based on the corresponding maleic anhydrides and primary aliphatic and aromatic amines.[8,9] The starting dithienylmaleic anhydride (**1a**) was obtained by the oxidation of furanone **2** with atmospheric oxygen in the presence of anhydrous potassium carbonate (Scheme 1).

Scheme 1



The synthesis of the thiazole-containing maleic anhydride **1b** is given in Scheme 2. The condensation of thienylchloroketone **3** with thioacetamide resulted in thienylthiazole **4**, whose chloroacetylation furnished haloketone **5**. The reaction of compound **5** with (2,5-dimethylthiophen-3-yl)acetic acid resulted in the formation of the target maleic anhydride **1b**.

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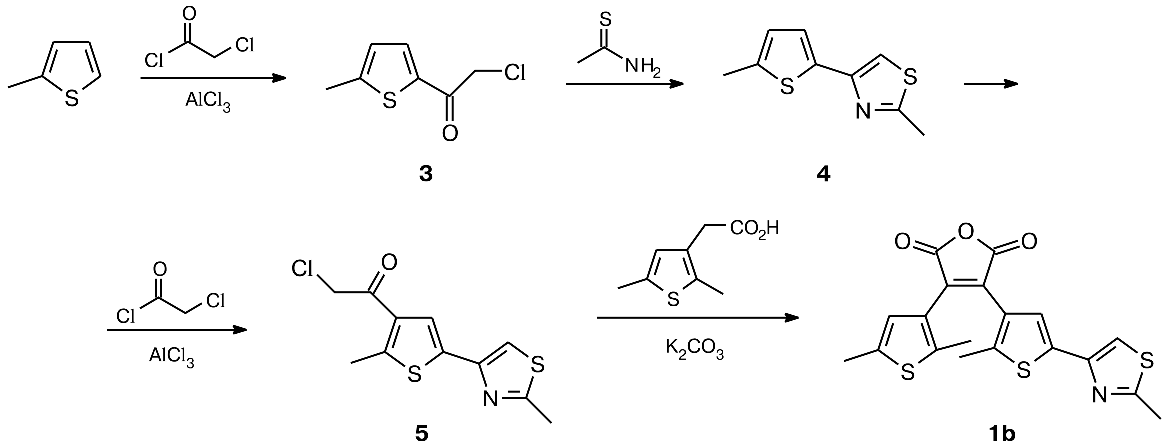
Scheme 2



We studied the reaction of dithienylmaleic anhydride **1b** with 2-(methylthio)aniline (**6**) and 2-(benzylthio)ethylamine (**7**) (Scheme 3). The reaction was carried out by reflux in ethanol in the presence of catalytic amounts of *p*-toluenesulfonic acid. In the case of aliphatic amine **7**, the reaction proceeded faster than with aromatic amine **6**, that was attributable to the different nucleophilicity of these amines. This resulted in the synthesis of sulfur-containing maleimides **8** and **9** (see Scheme 3).

Synthesis of *N*-thiocarbamide-substituted dithienylmaleimide. Earlier, we have shown[10] that photochromic dithienylperfluorocyclopentenes, in which the thiosemicarbazide fragment was bonded to the thiophene rings, formed stable complexes with silver nanoparticles. In the present work, we suggest an approach enabling one to incorporate a thiosemicarbazide fragment into the bridging maleimide ring. It should be noted that there are no literature data describing reactions of disubstituted maleic anhydrides with thiosemicarbazides.

The reaction of dithienylmaleic anhydrides **1a**,**b** with unsubstituted thiosemicarbazide was carried out by prolonged reflux in acetic acid (Scheme 4). As a result, maleimides **10a**,**b** containing a thiocarbamide fragment at the ring nitrogen atom were obtained in moderate yields (35—40%).

When the reaction was carried out with phenylthiosemicarbazide under similar conditions, the target product **11** was not isolated. The desired condensation was successfully carried out by reflux in Pr^iOH in the presence of catalytic amount of *p*-toluenesulfonic acid (Scheme 5).

To sum up, we suggested a general approach enabling one to synthesize dithienyl-substituted maleimides containing sulfide and thiocarbamide fragments at the nitrogen atom of the bridging ring.

Spectral and kinetic studies of photochromic properties of dithienylmaleimides with sulfur-containing fragments. The results of the studies are given in Table 1 and in Fig. 1.

Scheme 3

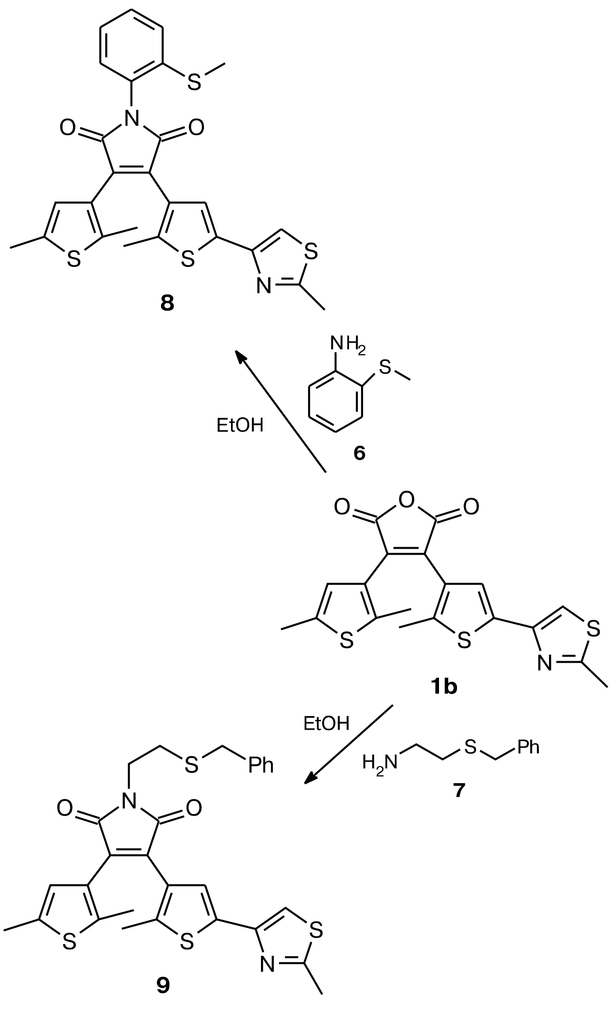
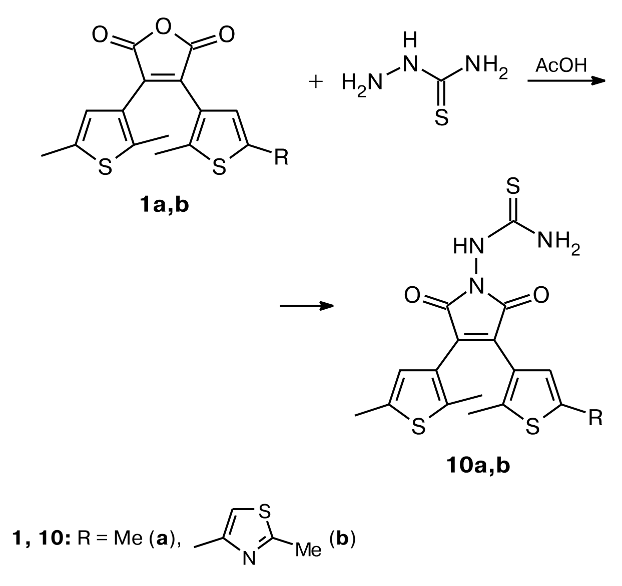
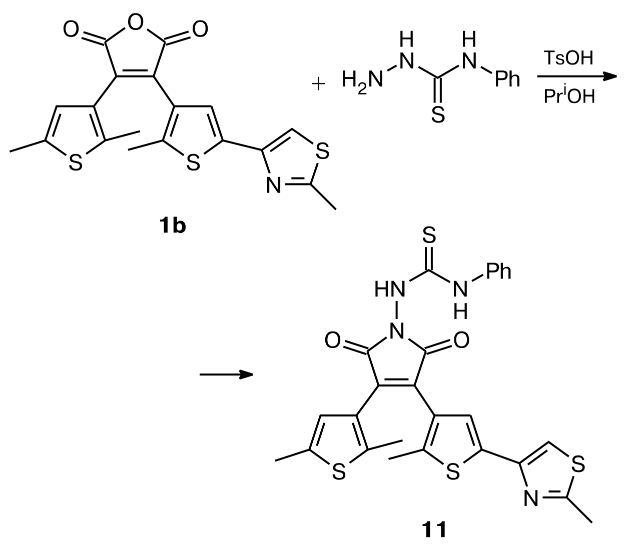


Figure 1 demonstrates typical absorption spectra of the initial open and the photoinduced cyclic form of the synthesized dithienylmaleimides with sulfur-containing fragments.

Scheme 4



Scheme 5



The reverse photoinduced transformations of the absorption spectra under the influence of UV (see Fig. 1, curve *2*) and visible (see Fig. 1, curve *3*) irradiation indicate that the compounds **8**—**11** possess photochromic properties.

Analysis of the results of spectral and kinetic studies of photochromism given in Table 1 show that all the synthesized compounds exhibit photochromic properties characteristic of diarylethenes.

From Table 1, it is seen that the introduction of the thiazole group into the thiophene ring (*cf.* the data for compounds **10a** and **10b**) leads to a bathochromic shift of the absorption maxima of both the open and the cyclic form. For the latter, it reached 41 nm. As it follows from the comparison of spectroscopic data for compounds with the thiazole substituent in the thiophene fragment (compounds **8**, **9**, **10b**, and **11**), the position of the absorption bands of the cyclic form slightly depends on the nature of substituents in the structure of the bridge. In this case, only the introduction of the benzylthioethyl group (compound **9**) led to a hypsochromic shift of the absorption band of the cyclic form.

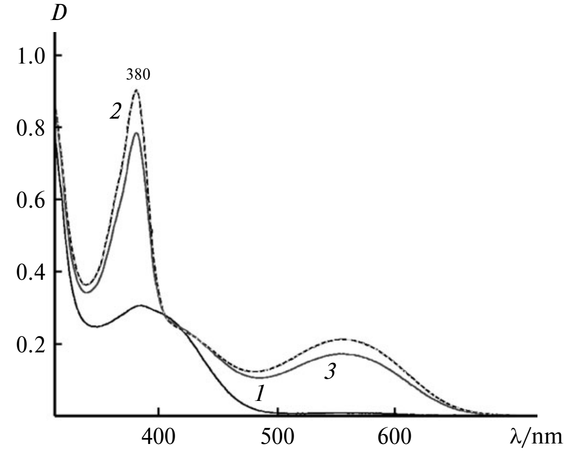


Fig. 1. Absorption spectra of compound **10b** in acetonitrile ($C = 4 \cdot 10^{-5}$ mol L^{-1}) before irradiation (*1*), after UV irradiation through the UFS-1 light filter (*2*) and subsequent irradiation with visible light through the ZhS-16 light filter (*3*).

Table 1. Spectral and kinetic characteristics of dithienylmaleimides with sulfur-containing fragments

Compound	λ^A /nm	ε^A /M^{-1} cm^{-1}	λ^B /nm	ΔD^B	$\Delta D^B/D^A$	k_{AB}	k_{BA}
						s^{-1}	
8	382	6750	552	0.24	0.89	0.32	0.01
	405	6000					
9	380	6000	544	0.33	1.38	0.39	0.01
	403	5500					
10a	367	5750	515	0.08	0.32	0.53	0.02
	400	6250					
10b	382	7750	556	0.20	0.65	0.31	0.01
	407	7000					
11	384	4250	552	0.07	0.41	0.25	0.01

Note: λ^A and λ^B are the wavelengths in the absorption maxima of the open and the cyclic forms, respectively; ε is the coefficient of molar extinction in the absorption maxima of the open form A; ΔD^B is the maximum photoinduced change in the optical density of solution in the absorption maximum of cyclic form in the photostationary state; $\Delta D^B/D^A$ is the maximum photoinduced change in the optical density of solution in the absorption maximum of cyclic form in the photostationary state normalized on the value of optical density in the absorption maximum of open form; k_{AB}, k_{BA} are the rate constants of the process of photocoloration of solution upon irradiation through the UFS-1 light filter and photodecoloration through the ZhS-11 light filter, respectively.

For all the diarylethenes under study, considerable photoinduced changes in optical density in the absorption maximum of the cyclic form were found. In this case, the introduction of the thiazole substituent into the thiophene fragment (*cf.* data for compounds **10a** and **10b**) increases susceptibility to UV irradiation evaluated by the value of the normalized photoinduced optical density, whereas the phenyl substituent in the bridging fragment (compound **11**) decreases it. Diarylethene with the benzylthioethyl substituent (compound **9**) is characterized by the highest susceptibility to UV irradiation (see Table 1). Compound **8** is also highly light susceptible.

From Table 1, it follows that the rate constant of photocoloration decreases upon the introduction of additional substituents in both the thiophene and the bridging fragment (*cf.* data for compounds **10a**, **10b**, and **11**). Among compounds with the thiazole fragment, the nature of substituents in the maleimide bridge (compounds **8**, **9**, and **10b**) virtually does not affect the rate of photocoloration.

The process of photodecoloration under the influence of visible irradiation is characterized by virtually identical rate constants for all the compounds under study (see Table 1).

In conclusion, the results of spectral and kinetic studies indicate that the introduction of the thiazole fragment into the thiophene ring of diarylethene leads to a bathochromic shift of the absorption bands of both forms, to the increase in light susceptibility to UV irradiation, to the formation of cyclic form with higher value of optical density. The change in the nature of substituents in the maleimide bridge has considerably smaller influence on the spectroscopic characteristics of both forms, except of the sulfide substituents, whose introduction causes a considerable increase in the susceptibility to UV irradiation.

Experimental

Spectrophotometric measurements (photostationary spectra), as well as kinetics studies of the photocoloration and photodecoloration processes in the photoinduced cyclic form of compound under study in solution were performed on a Varian Cary 50 Bio spectrophotometer (Australia). Acetonitrile from Aldrich was used as the solvent, with the concentration of the solution being $4 \cdot 10^{-5}$ mol L^{-1}. The measurements were made in 2-mm thick (kinetic measurements) and 10-mm thick quartz cuvettes (spectrophotometric measurements).

Hamamatsu lightning cure$^{\text{TM}}$ LC lamps (50—60 Hz, Japan) and UFS-1, ZhS-11, and ZhS-16 colored optical glasses were used for the irradiation. The irradiance through the UFS-1 and ZhS-16 light filters was 140 and 30 W m^{-2}, respectively.

The rate constants of photocoloration and photodecoloration of solutions of diarylethenes were determined from the monoexponential kinetic curves using the Origin program.

The efficiency of photocoloration and photodecoloration was evaluated based on the rate constant k, which was determined on the suggestion that the kinetic curves of photocoloration ($[D^{\mathrm{B}}] = [D^{\mathrm{B}}{}_{\infty}](1 - \exp(-kt))$) and photodecoloration ($[D^{\mathrm{B}}] = [D^{\mathrm{B}}{}_0]\exp(-kt)$) of solutions of diarylethenes are monoexponential. In this case, the molar extinction coefficients of the colored forms in the maxima of their bands of the longwave absorption were assumed to be approximately equal, which can be justified for a number of the structurally similar compounds. The concentration of irradiated solutions was selected in such a way as to provide the similar amount of the absorbed light.

^{1}H and ^{13}C NMR spectra were recorded on a Bruker AM-300 (300 MHz) spectrometer. Melting points were measured a Boetius heating stage and were not corrected. Reaction progress and purity of compounds obtained were monitored by TLC (Merck Silica gel 60 F_{254} plates, eluent ethyl acetate—hexane).

Furanone **2**,[11] 2-(methylthio)aniline (**6**),[12] 2-(benzylthio)ethylamine (**7**),[13] 2-chloroacetyl-5-methylthiophene (**3**),[14] and 2,5-dimethyl-3-thienylacetic acid[15] were obtained according to the procedures described in the literature.

3,4-Bis(2,5-dimethyl-3-thienyl)furan-2,5-dione (1a). A mixture of furanone **2** (0.1 g, 0.33 mmol) and (0.1 g, 0.66 mmol) potassium carbonate in DMF (5 mL) was stirred for 16 h at 80 °C. Then, the reaction mixture was poured into water (20 mL) and extracted with ethyl acetate (3×20 mL). The organic layer was discarded, the aqueous layer was diluted with concentrated hydrochloric acid (2 mL), a precipitate formed was filtered off, washed with water and diethyl ether on the filter. The yield was 0.065 g (65%), m.p. 202—204 °C (see Ref. 16: m.p. 203—204 °C). ^{1}H NMR ($CDCl_3$), δ: 1.92 (s, 6 H, 2 CH_3); 2.42 (s, 6 H, 2 CH_3); 6.78 (s, 2 H, 2 H_{Het}).

2-Methyl-4-(5-methyl-2-thienyl)-1,3-thiazole (4). A mixture of chloroketone **3** (1 g, 5.73 mmol) and thioacetamide (0.51 g, 6.87 mmol) in ethanol (20 mL) was refluxed for 2 h. Then, the mixture was poured into water (50 mL), followed by the addition of potassium hydroxide (0.77 g, 0.013 mol). A precipitate formed was filtered off and washed with water. The yield was 78%, m.p. 66—68 °C. ^{1}H NMR ($CDCl_3$), δ: 2.48 (s, 3 H, CH_3); 2.72 (s, 3 H, CH_3); 6.69 (s, 1 H, CH_{Het}); 7.09 (s, 1 H, CH_{Het}); 7.21 (s, 1 H, CH_{Het}). Found (%): C, 55.43; H, 4.69; N, 7.25. $C_9H_9NS_2$. Calculated (%): C, 55.35; H, 4.64; N, 7.17.

2-Chloro-1-[2-methyl-5-(2-methyl-1,3-thiazol-4-yl)-3-thienyl]ethanone (5). Chloroacetyl chloride (0.35 g, 3.07 mmol) was added dropwise to a mixture of thienylthiazole **4** (0.5 g, 2.56 mmol) and aluminum chloride (1.2 g, 8.97 mmol) in dichloromethane (25 mL). The reaction mixture was stirred for 2 h at room temperature. Then, the mixture was poured into water (100 mL) and extracted with chloroform (3×50 mL). The organic layer was washed with saturated aqueous sodium hydrogen carbonate (3×100 mL) and water (3×100 mL), dried with Na_2SO_4, the solvent was evaporated. The residue was recrystallized from hexane. The yield was 86%. m.p. 97—99 °C. ^{1}H NMR ($CDCl_3$), δ: 2.72 (s, 3 H, CH_3); 2.80 (s, 3 H, CH_3); 4.60 (s, 2 H, CH_2); 7.19 (s, 1 H, CH_{Het}); 7.52 (s, 1 H, CH_{Het}). Found (%): C, 48.82; H, 3.86; N, 5.27. $C_{11}H_{10}ClNOS_2$. Calculated (%): C, 48.61; H, 3.71; N, 5.15.

3-(2,5-Dimethylthiophen-3-yl)-4-[2-methyl-5-(2-methylthiazol-4-yl)thiophen-3-yl]furan-2,5-dione (1b). A mixture of (0.11 g, 0.622 mmol) (2,5-dimethylthiophen-3-yl)acetic acid, 2-chloro-1-[2-methyl-5-(2-methylthiazol-4-yl)-thiophen-3-yl]ethanone **5** (0.17 g, 0.622 mmol), and anhydrous potassium carbonate (0.28 g, 2 mmol) in DMF (4 mL) was stirred for 16 h at 80 °C. Then, the reaction mixture was poured into water and extracted with ethyl acetate (3×30 mL), the organic layer was discarded, the aqueous solution was acidified with 10% aq. hydrochloric acid to pH = 2, followed by the addition of ethyl acetate (30 mL) and stirring for 3 h at room temperature. Then, the organic layer was separated, the aqueous layer was extracted with ethyl acetate (2×30 mL), the extracts were dried with Na_2SO_4. The solvent was evaporated, the residue was subjected to chromatography on silica gel (eluent hexane—ethyl acetate (3 : 1)) and recrystallized from diethyl ether. The yield was 56%, m.p. 181—183 °C. ^{1}H NMR ($CDCl_3$), δ: 1.98 (s, 6 H, 2 CH_3); 2.41 (s, 3 H, CH_3); 2.74 (s, 3 H, CH_3); 6.78 (s, 1 H, CH_{Het}); 7.21 (s, 1 H, CH_{Het}); 7.42 (s, 1 H, CH_{Het}). Found (%): C, 56.97; H, 3.87; N, 3.57. $C_{19}H_{15}NO_3S_3$. Calculated (%): C, 56.84; H, 3.77; N, 3.49.

3-(2,5-Dimethyl-3-thienyl)-4-[2-methyl-5-(2-methyl-1,3-thiazol-4-yl)-3-thienyl]-1-[2-(methylthio)phenyl]-1*H*-pyrrole-2,5-dione (8). A mixture of dithienylmaleic anhydride **1b** (0.4 g, 1 mmol) and 2-(methylthio)aniline **6** (0.15 g, 1.1 mmol) in acetic acid (15 mL) was refluxed for 20 h. Then, the mixture was poured into water, a precipitate formed was filtered off, dried, and subjected to chromatography on silica gel (eluent hexane—ethyl acetate (3 : 1)). The yield was 57%, m.p. 118—121 °C. ^{1}H NMR (DMSO-d_6), δ: 1.90 (s, 3 H, CH_3); 1.98 (s, 3 H, CH_3); 2.41 (s, 3 H, CH_3); 2.48 (s, 3 H, CH_3); 2.70 (s, 3 H, CH_3); 6.72 (s, 1 H, CH_{Het}); 7.31—7.62 (m, 5 H, CH_{arom}); 7.79 (s, 1 H, CH_{Het}). Found (%): C, 59.96; H, 4.37; N, 5.48. $C_{26}H_{22}N_2O_2S_4$. Calculated (%): C, 59.74; H, 4.24; N, 5.36.

1-[2-Benzylthioethyl]-3-(2,5-dimethyl-3-thienyl)-4-[2-methyl-5-(2-methyl-1,3-thiazol-4-yl)-3-thienyl]-1*H*-pyrrole-2,5-dione (9). A mixture of dithienylmaleic anhydride **1b** (0.1 g, $3.7 \cdot 10^{-4}$ mol), 2-benzylthioethylamine **7** (0.07 g, $4 \cdot 10^{-4}$ mol), and *p*-toluenesulfonic acid (5 mg, $3 \cdot 10^{-5}$ mol) in EtOH (10 mL) was refluxed for 15 h. Then, the reaction mixture was poured into water and filtered. A precipitate formed was dried and subjected to chromatography on silica gel (eluent hexane—ethyl acetate (3 : 1)). The yield was 78%, m.p. 68—72 °C. ^{1}H NMR (DMSO-d_6), δ: 1.90 (s, 3 H, CH_3); 1.98 (s, 3 H, CH_3); 2.39 (s, 3 H, CH_3); 2.68 (s, 3 H, CH_3, CH_2); 3.70—3.82 (m, 4 H, 2 CH_2); 6.71 (s, 1 H, CH_{Het}); 7.2—7.45 (m, 5 H, CH_{arom}); 7.42 (s, 1 H, CH_{arom}), 7.78 (s, 1 H, CH_{Het}). Found (%): C, 61.27; H, 4.89; N, 5.23. $C_{28}H_{26}N_2O_2S_4$. Calculated (%): C, 61.06; H, 4.76; N, 5.09.

***N*-[3,4-Bis(2,5-dimethyl-3-thienyl)-2,5-dioxo-2,5-dihydro-1*H*-pyrrol-1-yl]thiourea (10a).** A mixture of dithienylmaleic anhydride **1a** (0.2 g, $6.27 \cdot 10^{-4}$ mol) and thiosemicarbazide (0.07 g, $7.5 \cdot 10^{-4}$ mol) in acetic acid (3 mL) was refluxed for 16 h. Then, the mixture was poured into water, a precipitate formed was filtered off, dried, and subjected to chromatography on silica gel (eluent hexane—ethyl acetate (3 : 1)). The yield was 43%, m.p. 265—267 °C. ^{1}H NMR (DMSO-d_6), δ: 1.84 (s, 6 H, 2 CH_3); 2.40 (s, 6 H, 2 CH_3); 6.68 (s, 2 H, CH_{Het}); 8.15 (s, 1 H, NH); 8.38 (s, 1 H, NH). ^{13}C NMR (DMSO-d_6), δ: 182.6 (C = S), 167.3 (C = O), 138.7 (C_{thioph}), 136.2 (C_{thioph}), 131.6 (C_{thioph}), 129.3 (C_{thioph}), 126.4 (C = C), 14.7 (CH_3), 14.2 (CH_3). Found (%): C, 52.25; H, 4.46; N, 10.86. $C_{17}H_{17}N_3O_2S_3$. Calculated (%): C, 52.15; H, 4.38; N, 10.73.

***N*-{3-(2,5-Dimethyl-3-thienyl)-4-[2-methyl-5-(2-methyl-1,3-thiazol-4-yl)-3-thienyl]-2,5-dioxo-2,5-dihydro-1*H*-pyrrol-1-yl}thiourea (10b).** A mixture of dithienylmaleic anhydride **1b** (0.2 g, $4.96 \cdot 10^{-3}$ mol) and thiosemicarbazide (0.04 g, $4.96 \cdot 10^{-3}$ mol) in acetic acid (10 mL) was refluxed for 16 h. Then, the mixture was poured into water, a precipitate formed was filtered off, dried, and subjected to chromatography on silica gel (eluent hexane—ethyl acetate (3 : 1)). The yield was 39%, m.p. 273—274 °C. ^{1}H NMR (DMSO-d_6), δ: 1.89 (s, 3 H, CH_3); 1.98 (s, 3 H, CH_3); 2.40 (s, 3 H, CH_3); 2.68 (s, 3 H, CH_3); 6.70 (s, 1 H, CH_{Het}); 7.39 (s, 1 H, CH_{Het}), 7.78 (s, 1 H, CH_{Het}); 8.17 (s, 1 H, NH); 8.39 (s, 1 H, NH). Found (%): C, 50.83; H, 3.98; N, 12.02. $C_{20}H_{18}N_4O_2S_4$. Calculated (%): C, 50.61; H, 3.82; N, 11.80.

***N*-{3-(2,5-Dimethyl-3-thienyl)-4-[2-methyl-5-(2-methyl-1,3-thiazol-4-yl)-3-thienyl]-2,5-dioxo-2,5-dihydro-1*H*-pyrrol-1-yl}-N´-phenylthiourea (11).** A mixture of dithienylmaleic anhydride **1b** (0.1 g, $3.73 \cdot 10^{-4}$ mol), phenylthiosemicarbazide (0.045 g, $3.8 \cdot 10^{-4}$ mol), and *p*-toluenesulfonic acid (5 mg, $3 \cdot 10^{-5}$ mol) in propan-2-ol (15 mL) was refluxed for 16 h. Then, the mixture was poured into water, a precipitate formed was filtered off and dried. The residue was subjected to chromatography on silica gel (eluent hexane—ethyl acetate (3 : 1)). The yield was 36%, m.p. 276—279 °C. ^{1}H NMR (DMSO-d_6), δ: 1.89 (s, 3 H, CH_3); 1.98 (s, 3 H, CH_3); 2.39 (s, 3 H, CH_3); 2.60 (s, 3 H, CH_3); 6.67—6.80 (m, 3 H, CH_{arom}); 7.11—7.15 (m, 2 H, CH_{arom}); 7.48 (s, 1 H, CH_{Het}); 7.70—7.80 (m, 2 H, CH_{arom}); 9.94 (s, 1 H, NH). Found (%): C, 56.85; H, 4.21; N, 10.32. $C_{26}H_{22}N_4O_2S_4$. Calculated (%): C, 56.70; H, 4.03; N, 10.17.

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