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Synthesis of New (Arylsulfanyl)maleimide Derivatives

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Abstract—Previously unknown 2-(arylsulfanyl)-3-hydroxymaleimide and 2-(arylsulfanyl)-3-chloromaleimide derivatives have been synthesized, and several synthetic approaches to 2-(arylamino)-3-(arylsulfanyl)-maleimides have been tested. The reactivities of 2-chloro-3-(4-methylphenylsulfanyl)maleimide and 2,3-bis(4-methylphenylsulfanyl)maleimide toward nitrogen and sulfur nucleophiles have been studied, and products of substitution of one or two arylsulfanyl groups have been obtained.

Keywords: maleimides, (arylsulfanyl)maleimides, antibacterial agents, (arylamino)maleimides.

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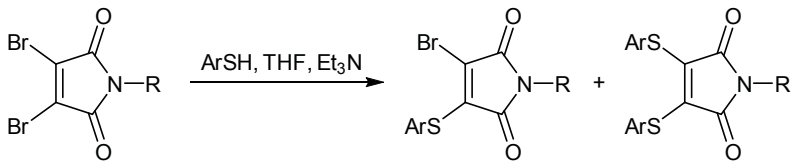
Maleimide derivatives are used not only as polymers [1, 2] or fluorescent dyes [3] but also as biologically active compounds. In particular, some antitumor agents [4] and protein kinase inhibitors [5] are derivatives of maleimide. A 2,3-bis(arylsulfanyl)maleimide fragment was used to modify the antibiotic teicoplanin with the goal of extending its spectrum of action [6]. Antibacterial and antifungal activities of *N*-alkyl-2-(arylsulfanyl)-maleimides have also been reported [7]. We previously synthesized a series of bis(phenylsulfanyl)maleimides which showed antimicrobial activity [8, 9]. In this work we synthesized a series of new related compounds.

New symmetrical bis(arylsulfanyl)maleimides **3a** and **3b** were synthesized by reaction of dibromomaleimide **1a** or *N*-methyldibromomaleimide **1b** with 4-methylbenzenethiol in the presence of triethylamine (Scheme 1) [9]. Only traces of 2-bromo-3-(arylsulfanyl)-maleimides **2** were detected, presumably due to their high reactivity. 2-(Arylamino)-3-(arylsulfanyl)maleimides **5a**–**5c** may be regarded as the closest analogs of **3a** and **3b**. The reaction of **1a** with substituted anilines in the presence of ethyl(diisopropyl)amine (DIPEA) initially involved substitution of one bromine atom with the formation of 2-(arylamino)-3-bromomaleimides **4a** and **4b**. It should be noted that some 2-(arylamino)-3-bromomaleimides reacted with amines, such as butylamine, piperidine, and morpholine to give the corresponding diamino derivatives [10]. However, the reactivity of **4a** and **4b** was relatively low, and the bromine atom therein was replaced only by the action of benzenethiols to produce compounds **5a**–**5c** (Scheme 2).

We succeeded in obtaining mono(arylsulfanyl) derivatives **7** and **8** using a modified Faul's procedure [11, 12]. The condensation of 2-(4-methylphenylsulfanyl)acetamide (**6**) with diethyl oxalate afforded hydroxymaleimide **7**, and treatment of the latter with oxalyl chloride in THF produced chloro derivative **8** (Scheme 3) [13]. No desired products were obtained with the use of other reagents such as phosphoryl chloride or thionyl chloride.

Like 2-(arylamino)-3-hydroxymaleimides [12], compound **7** reacted with aniline and dihydroindole to give products **9** and **10** as a result of substitution of the hydroxy group (Scheme 4). However, no similar

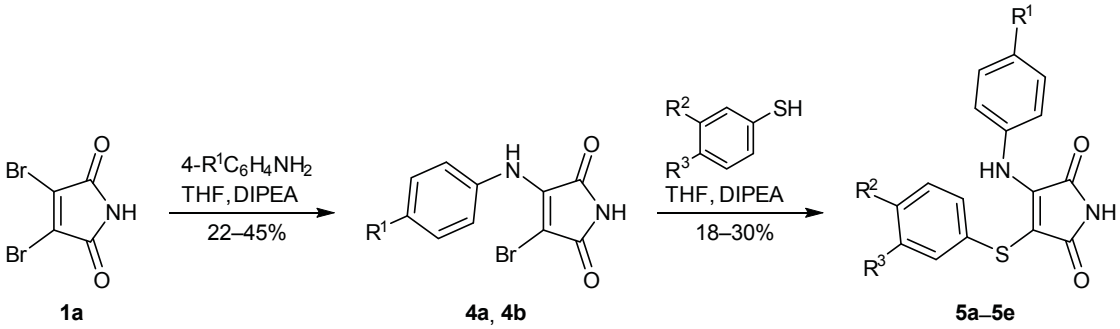
Scheme 1.



1a, **1b** **2** **3a** (85%), **3b** (77%)

1, R = H (**a**), Me (**b**); **3**, R = H (**a**, 85%), Me (**b**, 77%); Ar = 4-MeC_6H_4.

Scheme 2.



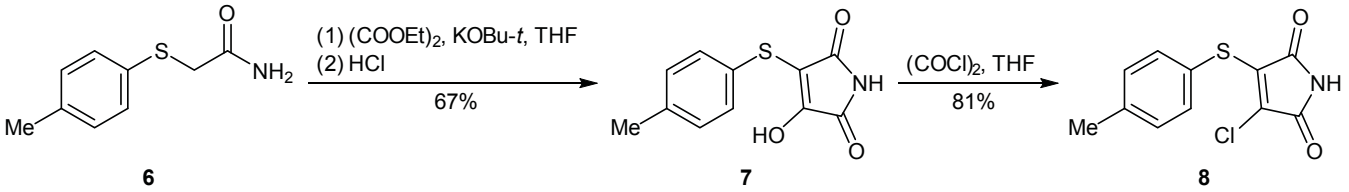
1a **4a**, **4b** **5a–5e**

4, R^1 = F (**a**), Cl (**b**); **5**, R^1 = F, $R^2 = R^3$ = H (**a**), R^1 = Cl, $R^2 = R^3$ = H (**b**), R^1 = Cl, $R^2 = R^3$ = MeO (**c**).

reaction occurred with benzenethiol. On the other hand, the reaction of **8** with 4-methoxybenzenethiol in THF in the presence of triethylamine led to the formation of symmetrically substituted maleimide **11** rather than the expected unsymmetrical derivative (Scheme 5). Presumably, the reaction involved replacement of both chlorine atom and phenylsulfanyl group. The same product was obtained by heating compound **3a** in dioxane with a large excess of 4-methoxybenzenethiol in the presence of triethylamine.

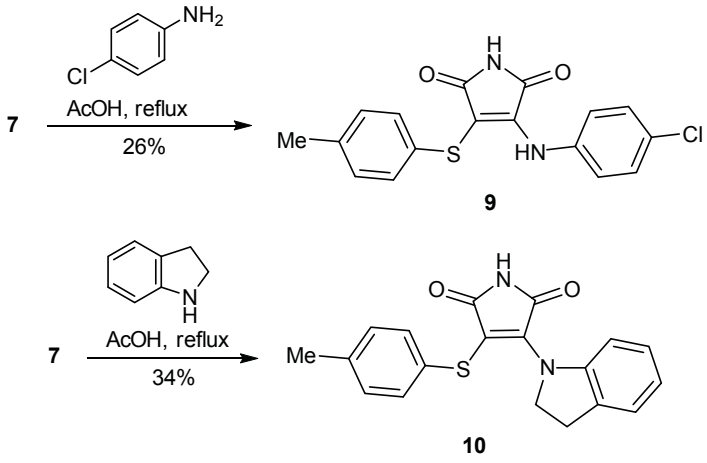
In fact, mass spectral analysis of the reaction mixture obtained under similar conditions from chloromaleimide **8** and an equimolar amount of benzenethiol revealed the presence of three bis(arylsulfanyl)maleimides

Scheme 3.



6 **7** **8**

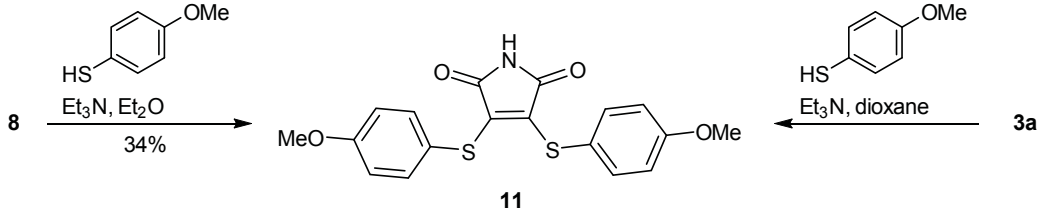
Scheme 4.



9

10

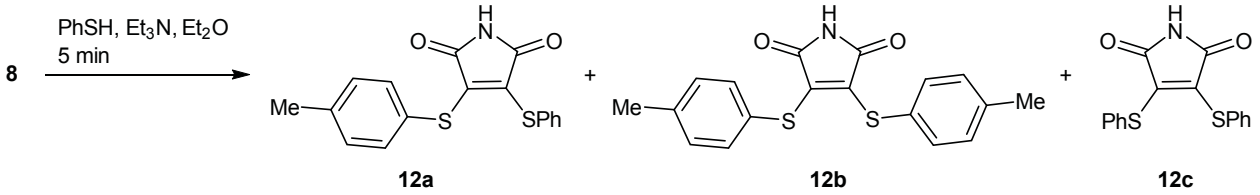
Scheme 5.



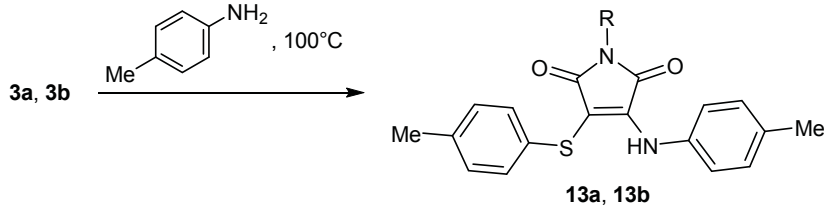
8 **3a**

11

Scheme 6.



Scheme 7.



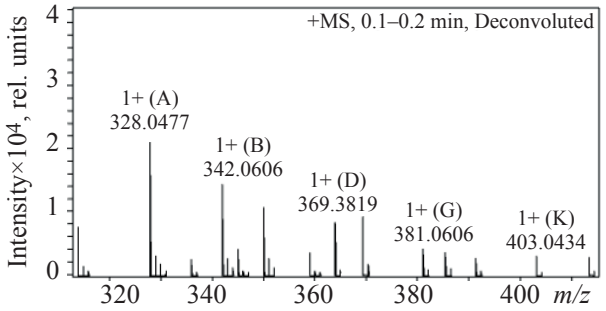
13, R = H (**a**), Me (**b**).

12a–**12c** (*m/z* 327, 342, and 314, respectively; Fig. 1, Scheme 6). Presumably, bis(arylsulfanyl)maleimides, especially unsymmetrical ones, are quite reactive toward nucleophiles. This was confirmed by the transformation of **3a** and **3b** into 2-(4-methylanilino)-3-(4-methylphenylsulfanyl)maleimides **13a** and **13b**, respectively, on heating with excess *p*-toluidine at 100°C (Scheme 7).

EXPERIMENTAL

The melting points were measured with a Buchi SMP-20 melting point apparatus and are uncorrected. The ^{1}H and ^{13}C NMR spectra were recorded on a Varian VXR-400 spectrometer at 400 and 100 MHz, respectively, using DMSO-d_6 as solvent and tetramethylsilane as standard. The high-resolution mass spectra (electrospray ionization) were obtained on a Bruker Daltonics MicrOTOF-Q II instrument (Germany); samples were introduced as solutions in methanol or acetonitrile (c = 0.1 mg/mL) directly into the ion source using a syringe pump (flow rate 3 μL/min); capillary voltage –4.5 and +4 kV for negative and positive ions, respectively; nebulizer gas (nitrogen) pressure 0.4 bar (5.8 psi), drying gas flow rate 4.0 L/min; ion source temperature 180°C; the mass analyzer was calibrated using a 1% ESI calibration solution (Sigma–Aldrich, Switzerland) in 95% aqueous acetonitrile; the accuracy was 0.43 ppm in the a.m.u. range from 118.086255 to 2721.894829; LC/MS grade solvents with a purity of higher than 98% were used. The electron impact mass spectra were recorded on a Finnigan SAQ 710 mass spectrometer (UK) with direct sample admission into the ion source (70 eV, ion source temperature 150°C).

Analytical TLC was performed on Silica gel F_{254} plates (Merck), and Silica gel 60 (Merck) was used for column chromatography. Extracts were dried over anhydrous sodium sulfate and were evaporated under reduced pressure. The purity of the isolated compounds was checked by HPLC on a Shimadzu LC10 instrument equipped with a Gemini 110A-C18 column (4.6×250 mm, grain size 5 μm; Phenomenex, USA) and a Shimadzu SPD-10A UV-Vis detector



m/z	*I*	*I*, %
314.0321	7656	36.0
328.0477	21283	100.0
342.0606	15259	71.7
345.0712	4279	20.1
350.0257	11047	51.9
359.0881	3968	18.6
364.0425	8401	39.5
369.3819	9696	45.6
381.0604	4348	20.4
385.3795	3717	17.5

Fig. 1. Mass spectrum of product mixture **12a**–**12c**.

(Japan); detection at the wavelengths corresponding to absorption maxima; eluent 0.2% ammonium formate in acetonitrile (pH 4.5). Reagents and solvents were commercial products.

Compound **6** was synthesized as described in [14]. Compounds **3a** and **3b** were synthesized according to the procedure reported in [9].

3,4-Bis[(4-methylphenyl)sulfanyl]-1*H*-pyrrole-2,5-dione (3a). Yield 290 mg (85%), yellow crystals, mp 104–106°C. ^{1}H NMR spectrum, δ, ppm: 2.27 s (6H, CH_3), 7.09 d (4H, J = 8.1 Hz), 7.16 d (4H, J = 8.1 Hz), 11.28 s (1H, NH). ^{13}C NMR spectrum, δ_C, ppm: 20.65, 125.97, 129.64, 131.08, 136.37, 137.71, 167.42. Mass spectrum: m/z 342.0622 $[M + H]^+$. $C_{18}H_{15}NO_2S_2$. Calculated: M + H 342.0617.

1-Methyl-3,4-bis[(4-methylphenyl)sulfanyl]-1*H*-pyrrole-2,5-dione (3b). Yield 273 mg (77%), yellow crystals, mp 114–115°C. ^{1}H NMR spectrum, δ, ppm: 2.26 s (6H, CH_3), 2.84 s (3H, NCH_3), 7.08 d (4H, J = 8.1 Hz), 7.15 d (4H, J = 8.1 Hz). ^{13}C NMR spectrum, δ_C, ppm: 21.12, 24.90, 126.33, 130.11, 131.58, 136.32, 138.27, 166.81. Mass spectrum: m/z 356.0778 $[M + H]^+$. $C_{19}H_{17}NO_2S_2$. Calculated: M + H 356.0773.

3-Bromo-4-(4-fluoroanilino)-1*H*-pyrrole-2,5-dione (4a). Dibromomaleimide **1a**, 2.0 g (7.84 mmol), was dissolved in 15 mL of anhydrous DMF, and 0.9 mL (9.51 mmol) of 4-fluoroaniline and 2 mL (11.5 mmol) of DIPEA were added. The mixture was stirred for 24 h at 50°C and poured into a mixture of water and ethyl acetate. The organic layer was separated, washed with dilute aqueous HCl, and evaporated under reduced pressure, and the residue was purified by column chromatography using petroleum ether–ethyl acetate (5 : 1) as eluent. Yield 503 mg (22%), pale yellow crystals, mp 208–210°C; HPLC: τ = 11.09 min, 96%. ^{1}H NMR spectrum, δ, ppm: 7.15–7.23 m (4H), 9.60 s (1H, 4-NH), 10.96 s (1H, N^{1}H). ^{13}C NMR spectrum, δ_C, ppm: 80.39, 114.83 d (J = 22.8 Hz), 126.64 d (J = 8.5 Hz), 132.89, 141.73, 159.63 d (J = 242.0 Hz), 167.04, 168.4. Mass spectrum: m/z 284.9703 $[M + H]^+$. $C_{10}H_6BrFN_2O_2$. Calculated: M + H 284.9669.

3-Bromo-4-(4-chloroanilino)-1*H*-pyrrole-2,5-dione (4b) was synthesized in a similar way from 2.0 g (7.84 mmol) of **1b** and 1.1 g (8.66 mmol) of 4-chloroaniline in 20 mL of DMF. Yield 1.16 g (45% based on 4-chloroaniline), pale yellow crystals, mp 230–235°C (decomp., from EtOAc). ^{1}H NMR spectrum, δ, ppm: 7.17 d (2H, J = 8.6 Hz), 7.38 d (2H, J = 8.6 Hz), 9.68 s (1H, 4-NH), 11.01 s (1H, N^{1}H). ^{13}C NMR spectrum, δ_C, ppm: 82.67, 125.81, 128.07, 128.96, 135.68, 141.30, 167.16, 168.38. Mass spectrum: m/z 300.9296 $[M + H]^+$. $C_{10}H_6BrClN_2O_2$. Calculated: M + H 300.9374.

3-(4-Fluoroanilino)-4-(phenylsulfanyl)-1*H*-pyrrole-2,5-dione (5a). Compound **4a**, 150 mg (0.53 mmol), was dissolved in 5 mL of DMF, 0.2 mL (1.81 mmol) of benzenethiol and 0.3 mL (1.72 mmol) of DIPEA were added, and the mixture was stirred for 12 h at 50°C. The mixture was poured into a mixture of water and ethyl acetate, the organic layer was separated, and the aqueous layer was extracted with 20 mL of ethyl acetate. The extracts were evaporated with addition of toluene to remove traces of water. The residue was purified by column chromatography using petroleum ether–ethyl acetate (5 : 1) as eluent. Yield 50 mg (30%), yellow crystals, mp 203–204°C (from EtOAc). HPLC: τ = 8.4 min, 96.6%. ^{1}H NMR spectrum, δ, ppm: 7.54–6.66 m (9H), 9.88 s (1H), 10.92 s (1H). ^{13}C NMR spectrum, δ_C, ppm: 114.5, 114.73, 125.53, 126.32, 126.78, 129.13, 132.85, 136.34, 146.66, 158.9, 161.31, 167.73, 171.96. Mass spectrum: m/z 313.0616 $[M - H]^-$. $C_{16}H_{11}FN_2O_2S$. Calculated: M – H 313.0453.

3-(4-Chloroanilino)-4-(phenylsulfanyl)-1*H*-pyrrole-2,5-dione (5b) was synthesized as described above for **5a**. Yield 31 mg (18%), yellow crystals, mp 205–207°C. HPLC: τ = 10.9 min, 96.5%. ^{1}H NMR spectrum, δ, ppm: 6.84 d (2H, J = 8.6 Hz), 6.95 d (2H, J = 8.0 Hz), 7.07–7.16 m (5H), 9.94 s (1H, NH), 10.97 s (1H, NH). ^{13}C NMR spectrum, δ_C, ppm: 125.66, 125.97, 126.53, 127.75, 129.11, 129.53, 135.48, 135.74, 145.74, 167.79, 171.81. Mass spectrum: m/z 329.0364 $[M - H]^-$. $C_{16}H_{11}ClN_2O_2S$. Calculated: M – H 329.0157.

3-(4-Chloroanilino)-4-(3,4-dimethoxyphenylsulfanyl)-1*H*-pyrrole-2,5-dione (5c) was synthesized as described above for **5a**. Yield 62 mg (30%), yellow crystals, mp 176–181°C (from EtOAc). ^{1}H NMR spectrum, δ, ppm: 3.60 s (1H), 3.65 s (1H), 6.28 s (1H), 6.36 d (1H, J = 8.2 Hz), 6.69 d (1H, J = 8.4 Hz), 6.91 d (1H, J = 8.7 Hz), 7.19 d (1H, J = 8.7 Hz), 10.55 s (1H, NH). ^{13}C NMR spectrum, δ, ppm: 55.73, 56.13, 111.68, 112.79, 120.43, 125.39, 125.70, 127.76, 128.95, 135.58, 135.61, 143.75, 147.86, 149.13, 168.08, 171.95. Mass spectrum: m/z 391.0577. $C_{18}H_{15}ClN_2O_4S$. Calculated: M + H 391.0514.

3-Hydroxy-4-(4-methylphenylsulfanyl)-1*H*-pyrrole-2,5-dione (7). 2-(4-Methylphenylsulfanyl)-acetamide (**6**), 1.03 g (5.7 mmol), was dissolved in 25 mL of anhydrous DMF, 0.83 g (5.6 mmol) of diethyl oxalate was added, and 2.5 g (22.3 mmol, 3.9 equiv) of potassium *tert*-butoxide was then added in one portion.

The mixture turned yellow and warmed up. After 24 h, the mixture was treated with 50 mL of 25% aqueous HCl, kept for 10 min, and extracted with ethyl acetate (2 × 20 mL). The combined extracts were evaporated, and the residue was purified by column chromatography using ethyl acetate–ethanol (10:1) as eluent. Yield 912 mg (67%), yellow crystals, mp 175–180°C. ^{1}H NMR spectrum, δ, ppm: 2.18 s (3H, CH_3), 6.89 d (2H, J = 8.1 Hz), 6.97 d (2H, J = 8.1 Hz), 9.66 s (1H, NH). ^{13}C NMR spectrum, δ_C, ppm: 20.87, 80.48, 125.17, 129.43, 133.09, 138.26, 169.84, 174.65, 175.62. Mass spectrum: m/z 234.0168 $[M - H]^-$. $C_{11}H_9NO_3S$. Calculated: M – H 234.0230.

3-Chloro-4-(4-methylphenylsulfanyl)-1*H*-pyrrole-2,5-dione (8). Maleimide **7**, 82 mg (0.39 mmol), was dissolved in 5 mL of DMF, and 0.1 mL (1.16 mmol) of oxalyl chloride was added dropwise with stirring. After 2 h (complete conversion according to the TLC data), the mixture was poured into 15 mL of water and extracted with ethyl acetate (20 mL). The organic layer was separated and evaporated, and the residue was purified by column chromatography using petroleum ether–ethyl acetate (5:1) as eluent. Yield 80 mg (81%), mp 169–171°C. ^{1}H NMR spectrum, δ, ppm: 2.30 s (3H, CH_3), 7.21 d (2H, J = 7.8 Hz), 7.44 d (2H, J = 7.8 Hz), 11.50 s (1H, NH). ^{13}C NMR spectrum, δ_C, ppm: 21.21, 123.48, 130.35, 130.53, 133.83, 137.42, 139.70, 165.73, 167.10. Mass spectrum: m/z 251.9931 $[M - H]^-$. $C_{11}H_8ClNO_2S$. Calculated: M – H 251.9892.

3-(4-Chloroanilino)-4-(4-methylphenylsulfanyl)-1*H*-pyrrole-2,5-dione (9). Maleimide **7**, 117 mg (0.5 mmol), was dissolved in 10 mL of acetic acid, 70 mg (0.55 mmol) of 4-chloroaniline was added, and the mixture was refluxed for 3 h with stirring. The mixture was evaporated under reduced pressure, and the residue was purified by column chromatography using petroleum ether–ethyl acetate (3:1) as eluent. Yield 46 mg (26%), mp 224–225°C (from EtOAc). ^{1}H NMR spectrum, δ, ppm: 2.18 s (3H, CH_3), 6.73 d (2H, J = 8.0 Hz), 6.90–6.97 m (4H), 7.16 d (2H, J = 8.6 Hz), 9.86 s (1H, NH), 10.93 s (1H, NH). ^{13}C NMR spectrum, δ_C, ppm: 20.89, 91.30, 125.93, 126.95, 127.79, 129.47, 129.77, 132.12, 135.19, 135.61, 145.45, 167.84, 171.81. Mass spectrum: m/z 345.0440 $[M + H]^+$. $C_{17}H_{13}ClN_2O_2S$. Calculated: M + H 345.0459.

3-(2,3-Dihydro-1*H*-indol-1-yl)-4-(4-methylphenylsulfanyl)-1*H*-pyrrole-2,5-dione (10). Maleimide **7**, 150 mg (0.64 mmol), was dissolved in 10 mL of acetic acid, 100 mg (0.84 mmol) of 2,3-dihydro-1*H*-indole was added, and the mixture was refluxed for 1 h with stirring. The mixture was evaporated under reduced pressure, and the residue was purified by column chromatography using petroleum ether–ethyl acetate (5:1) as eluent. Yield 73 mg (34%), light beige crystals, mp 163–164°C. ^{1}H NMR spectrum, δ, ppm: 2.15 s (3H, CH_3), 2.82 t (2H, CH_2), 4.20 t (2H, CH_2), 6.84–6.93 m (6H, H_{arom}), 7.04 t (2H, H_{arom}), 11.01 br.s (1H, NH). ^{13}C NMR spectrum, δ_C, ppm: 20.87, 29.20, 53.91, 100.91, 116.12, 123.30, 124.52, 126.24, 127.39 (2C), 129.70 (2C), 131.33, 133.13, 135.43, 141.91, 144.12, 167.70, 170.61. Mass spectrum: m/z 337.1008 $[M + H]^+$. $C_{19}H_{16}N_2O_2S$. Calculated: M + H 337.1005.

3,4-Bis[(4-methoxyphenyl)sulfanyl]-1*H*-pyrrole-2,5-dione (11). Maleimide **8**, 80 mg (0.316 mmol), was dissolved in 10 mL of diethyl ether, and a solution of 150 mg (1.22 mmol) of 4-methoxybenzenethiol and 0.2 mL (1.43 mmol) of triethylamine in 5 mL of diethyl ether was added in portions over a period of 30 min. The mixture was heated to the boiling point and was refluxed for 1 h until the reaction was complete (TLC). The mixture was evaporated, and the residue was purified by column chromatography using petroleum ether–ethyl acetate (3:1) as eluent. Yield 40 mg (34%). ^{1}H NMR spectrum, δ, ppm: 3.74 s (6H, OCH_3), 6.87 d (4H, J = 8.8 Hz), 7.26 d (4H, J = 8.8 Hz), 11.17 br.s (1H, NH). ^{13}C NMR spectrum, δ_C, ppm: 55.30, 114.70, 119.64, 133.58, 136.13, 159.58, 167.51. Mass spectrum: m/z 374.0552 $[M + H]^+$. $C_{18}H_{15}NO_4S_2$. Calculated: M + H 374.0515.

3-(4-Methylanilino)-4-[(4-methylphenyl)sulfanyl]-1*H*-pyrrole-2,5-dione (13a). Maleimide **3a**, 341 mg (1 mmol), was dissolved on heating in 3 mL of *p*-toluidine, and the mixture was stirred for 2 h at 100°C. The mixture was poured into water (20 mL), acidified with aqueous HCl, and extracted with ethyl acetate. The extract was evaporated under reduced pressure, and the residue was purified by column chromatography using petroleum ether–ethyl acetate (3:1) as eluent. Yield 150 mg (46%). ^{1}H NMR spectrum, δ, ppm: 2.19 s (3H), 2.22 s (3H), 6.80 d (2H, J = 8.1 Hz), 6.95 m (2H), 9.83 br.s (1H), 10.75 br.s (1H). ^{13}C NMR spectrum, δ_C, ppm: 20.39, 20.50, 88.48, 124.21, 126.31, 128.04, 129.30, 132.80, 133.78, 134.41, 134.51, 146.42, 167.32, 171.43. Mass spectrum: m/z 325.1037 $[M + H]^+$. $C_{18}H_{16}N_2O_2S$. Calculated: M + H 325.1005.

***N*-Methyl-3-(4-methylanilino)-4-[(4-methylphenyl)sulfanyl]-1*H*-pyrrole-2,5-dione (13b)** was synthesized as described above for **13a**. Yield 128 mg (38%). ^{1}H NMR spectrum, δ, ppm: 2.19 s (3H), 2.22 s (3H), 2.93 s (3H), 6.82 d (2H, J = 7.0 Hz), 6.95 m (6H), 9.98 s (1H, NH). ^{13}C NMR spectrum, δ_C, ppm: 20.45, 20.55, 24.09, 87.19, 124.31, 126.27, 128.09, 129.35,

132.85, 133.77, 134.45, 134.72, 146.56, 166.26, 170.66. Mass spectrum: m/z 339.1185 $[M + H]^+$. $C_{19}H_{18}N_2O_2S$. Calculated: M + H 339.1162.

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CONFLICT OF INTERESTS

The authors declare the absence of conflict of interests.

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