A New Access to Diarylmaleic Anhydrides

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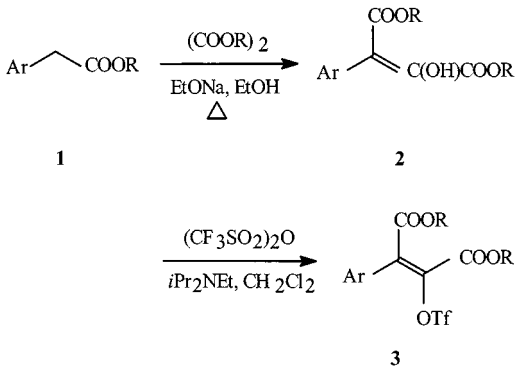
A new three-step synthesis of diarylmaleic anhydrides **6**, starting from 3-aryl-2-hydroxybut-2-enedioates **2**, is reported.

Diindolylmaleic anhydrides are useful intermediates in the synthesis of some indole alkaloids.[1–5] Such compounds, including aryl(indolyl)maleic anhydrides, have recently been obtained from (1*H*-indol-3-yl)oxoacetyl chloride and arylacetic acids.[6] These anhydrides are also good precursors of the corresponding maleimides,[7] a class of compounds that have been found to be selective PKC inhibitors.[5]

We report here a new and general method for the synthesis of maleic anhydrides, starting from 3-aryl-2-hydroxybut-2-enedioates **2**, which are readily accessible from the corresponding arylacetates. Diesters **2** were first transformed into the triflates **3**, as shown in Scheme 1.[8]



Scheme 1. Formation of triflates **3** from arylacetates **1**

1, **2**	R	Ar
a	Et	phenyl
b	Me	1-methyl-2-pyrrolyl
c	Et	2-thienyl
d	Et	3-indolyl

3	R	Ar
a	Et	phenyl
b	Me	1-methyl-2-pyrrolyl
c	Et	thien-2-yl
d	Et	1-ethoxycarbonyl-3-indolyl

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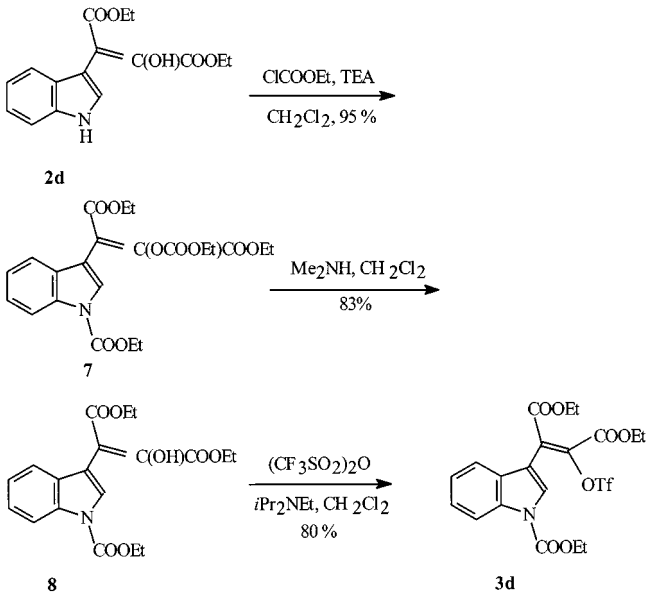
However, diethyl 2-(1-ethoxycarbonyl-1*H*-indol-3-yl)-3-(trifluoromethanesulfonyloxy)but-2-enedioate (**3d**) was prepared by a different route, analogous to the preparation of ethyl 3-[1-cyano-2-ethoxycarbonyl-2-(trifluoromethanesulfonyloxy)vinyl]indole-1-carboxylate.[9] Thus, from the known diethyl 2-hydroxy-3-(1*H*-indol-3-yl)but-2-enedioate (**2d**),[10] compound **7** was prepared by reaction with ethyl chlorocarbonate and triethylamine. Compound **8** was then obtained from **7** by reaction with dimethylamine. Finally, the triflate **3d** was prepared from **8** and trifluoromethanesulfonic anhydride in good yield (Scheme 2).



Scheme 2. Sequence of events leading to triflate **3d**

The prepared triflates **3** were obtained as pure (*E*) or (*Z*) isomers with the exception of **3b**, which was obtained as a 5:1 mixture of the two isomers. These isomers exhibit identical ^{1}H-NMR spectra, but have different melting points and R_f values and thus could easily be separated by column chromatography on silica gel. The subsequent coupling reactions of the triflates **3** with the aryltributylstannanes **4** afforded the isomerically pure products **5** in good yields (Scheme 3). The reactions were carried out in anhydrous THF containing tetrakis(triphenylphosphane)palladium, essentially under Stille conditions,[11] but LiCl and CuI[12] were also added to the mixtures in all cases other than the reaction giving **5ae**. In the latter case, better results were obtained when the reaction was carried out at 75°C in

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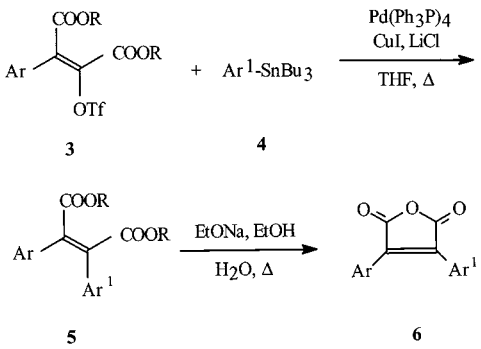
DMF containing triphenylphosphane with $Pd_2(dba)_3$ as the catalyst.



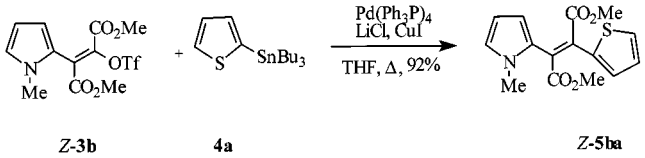
Scheme 3. Synthesis of anhydrides **6** from the triflates **3**

4	Ar[1]
a	2-thienyl
b	2-furyl
c	1-benzenesulfonyl-2-indolyl
d	1-benzenesulfonyl-3-indolyl
e	phenyl

5	R	Ar	Ar[1]
aa	Et	phenyl	2-thienyl
ac	Et	phenyl	1-benzenesulfonyl-2-indolyl
ad	Et	phenyl	1-phenylsulfonyl-3-indolyl
ae	Et	phenyl	phenyl
ba	Me	1-methyl-2-pyrrolyl	2-thienyl
bb	Me	1-methyl-2-pyrrolyl	2-furyl
bc	Me	1-methyl-2-pyrrolyl	1-phenylsulfonyl-2-indolyl
bd	Me	1-methyl-2-pyrrolyl	1-phenylsulfonyl-3-indolyl
cb	Et	2-thienyl	2-furyl
cc	Et	2-thienyl	1-phenylsulfonyl-2-indolyl
da	Et	1-ethoxycarbonyl-3-indolyl	2-thienyl
db	Et	1-ethoxycarbonyl-3-indolyl	2-furyl
dc	Et	1-ethoxycarbonyl-3-indolyl	1-phenylsulfonyl-2-indolyl
dd	Et	1-ethoxycarbonyl-3-indolyl	1-phenylsulfonyl-3-indolyl

6	Ar	Ar[1]
aa	phenyl	2-thienyl
ac	phenyl	2-indolyl
ad	phenyl	3-indolyl
ae	phenyl	phenyl
ba	1-methyl-2-pyrrolyl	2-thienyl
bb	1-methyl-2-pyrrolyl	2-furyl
bc	1-methyl-2-pyrrolyl	2-indolyl
bd	1-methyl-2-pyrrolyl	3-indolyl
cb	2-thienyl	2-furyl
cc	2-thienyl	2-indolyl
da	3-indolyl	2-thienyl
db	3-indolyl	2-furyl
dc	3-indolyl	2-indolyl
dd	3-indolyl	3-indolyl

In order to assign the stereochemistry of the esters **5**, and hence of the triflates **3**, the two isomers of the triflate **3b** were treated separately with tributyl(thien-2-yl)stannane (**4a**). The major isomer of triflate **3b** afforded the corresponding ester (*E*)-**5ba**, while the minor isomer afforded the ester (*Z*)-**5ba** (Scheme 4).



Scheme 4. Pd^0-catalyzed formation of compound *Z*-**5ba**

The stereochemistry of the esters **5ba**, and hence that of the corresponding triflates **3b**, was assigned on the basis of NOE experiments (Figure 1).

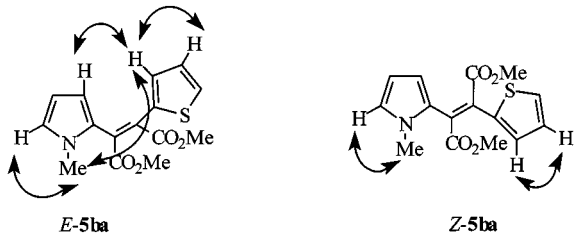


Figure 1. Selected NOE correlation of compounds *E*-**5ba** and *Z*-**5ba**

To confirm the assigned stereochemistry for all esters **5** and triflates **3**, the triflate **3a** was treated with tributyl(phenyl)stannane (**4e**) to afford diethyl diphenylmaleate (**5ae**).

Alkaline hydrolysis of esters **5** afforded, after acid treatment, the corresponding anhydrides **6**. From the ester (*Z*)-**5ba**, no anhydride could be obtained. In the case of compounds **5** containing an *N*-protected indole ring, alkaline hydrolysis afforded the corresponding *N*-deprotected anhydrides **6**.

Experimental Section

General: Melting points were determined with a Büchi 510 apparatus and are uncorrected. − IR spectra were recorded with a JASCO IR Report 100 instrument, in Nujol mulls for solids and as liquid films for oils. − ^{1}H-NMR spectra were recorded with a Varian Gemini 200 or a Bruker AVANCE DRX 300 spectrometer, in $CDCl_3$ solution unless otherwise stated; chemical shifts (δ) are expressed in ppm relative to TMS, coupling constants (*J*) in Hz. The NMR assignments were made by comparison with literature spectroscopic data and by analysis of NOESY and COSY spectra where necessary. − Column chromatography was performed on Merck Kieselgel 60, 0.063−0.2 mm. − Organic extracts were dried with Na_2SO_4; solvents were evaporated in vacuo in a rotary evaporator. − Compounds **2a**,[14] **2d**,[10] **4c**,[15] and **4d**[13] were prepared according to literature procedures. 2,3-Diphenylmaleic anhydride (**6ae**) is commercially available from Aldrich, m.p. 159−162°C.

Dimethyl 2-Hydroxy-3-(1-methyl-1*H*-pyrrol-2-yl)but-2-enedioate (2b): To a solution of EtONa, prepared from Na (1.6 g, 70 mmol) and ethanol (120 mL), were added methyl (1-methyl-1*H*-pyrrol-2-yl)acetate (**1b**) (7.5 mL, 50 mmol) and dimethyl oxalate (8.26 g, 70 mmol). The resulting mixture was refluxed for 1.5 h. After evaporation of the solvent, water (80 mL) was added and the mixture was extracted with Et_2O (2 × 50 mL). The aqueous layer was then acidified (9% HCl, 30 mL) and extracted with CH_2Cl_2 (2 × 50 mL). The organic phase was dried, filtered, and the solvent was

evaporated. The residue was purified by chromatography on silica gel (eluent hexane/Et_2O, 1:1) to give pure **2b**, 7.5 g (63%), m.p. 59−60°C. − IR: $\tilde{\nu}$ = 3400, 1730, 1650, 1605 cm^{-1}. − ^1H NMR: δ = 3.47 (s, 3 H, NMe), 3.64 (s, 3 H, OMe), 3.82 (s, 3 H, OMe), 5.96 (dd, *J* = 1.8/3.6 Hz, 1 H, pyrrole 4-H), 6.08 (m, 1 H, pyrrole 3-H), 6.88 (m, 1 H, pyrrole 5-H), 12.76 (s, 1 H, H/D exchangeable). − $C_{11}H_{13}NO_5$ (239.22): calcd. C 55.23, H 5.48, N 5.85; found C 55.10, H 5.43, N 5.78.

Diethyl 2-Hydroxy-3-(thien-2-yl)but-2-enedioate (2c): To a solution of EtONa, prepared from Na (1.38 g, 60 mmol) and ethanol (100 mL), were added ethyl 2-thiopheneacetate (**1c**) (8.5 g, 50 mmol) and diethyl oxalate (8.15 mL, 60 mmol). The resulting mixture was refluxed for 1 h, then the solvent was evaporated, and the crude residue was taken up in H_2O (40 mL) and extracted with Et_2O (2 × 40 mL). The aqueous layer was acidified (9% HCl, 30 mL) and then extracted with CH_2Cl_2 (2 × 40 mL). The organic phase was dried, filtered, and the solvent was evaporated. Purification of the residue by chromatography on silica gel (eluent pentane/CH_2Cl_2, 2:1) gave pure **2c**, 11.3 g (84%), oil. − IR (film): $\tilde{\nu}$ = 3390, 1720−1730, 1639 cm^{-1}. − ^1H NMR ([D_6]DMSO): δ = 1.27 (m, 6 H, CH_3), 4.27 (m, 4 H, OCH_2), 7.10 (m, 2 H, thiophene 3-H and 4-H), 7.63 (dd, *J* = 1.3/5.1 Hz, 1 H, thiophene 5-H). − $C_{12}H_{14}O_5S$ (270.30): calcd. C 53.32, H 5.22; found C 53.13, H 5.28.

Alkyl (2-Aryl-3-trifluoromethanesulfonyloxy)but-2-enedioates 3. − General Method: To a solution of compound **2** (10 mmol) in CH_2Cl_2 (50 mL) was added *N*,*N*-diisopropylethylamine (2.57 mL, 15 mmol). The stirred reaction mixture was cooled to 0°C, whereupon a solution of trifluoromethanesulfonic anhydride (2.2 mL, 13 mmol) in CH_2Cl_2 (5 mL) was added. After 10 min at 0°C, the mixture was washed with water (2 × 30 mL). The organic phase was dried, filtered, and the solvent was evaporated, and the residue was purified by chromatography on silica (eluents reported for each individual compound).

3a: Eluent pentane/CH_2Cl_2 (1:1); oil; yield 85%. − IR (film): $\tilde{\nu}$ = 1710−1738, 1620 cm^{-1}. − ^1H NMR: δ = 1.36 (m, 6 H, CH_3), 4.37 (m, 4 H, OCH_2), 7.40 (m, 5 H, arom.). − $C_{15}H_{15}F_3O_7S$ (396.34): calcd. C 45.46, H 3.81; found C 45.33, H 3.78.

(*E*)-3b: Eluent pentane/CH_2Cl_2 (5:1); m.p. 45−46°C (pentane); yield 75%. − IR: $\tilde{\nu}$ = 1718, 1575 cm^{-1}. − ^1H NMR: δ = 3.54 (s, 3 H, NMe), 3.74 (s, 3 H, OMe), 3.87 (s, 3 H, OMe), 6.17 (dd, *J* = 2.8/3.9 Hz, 1 H, pyrrole 4-H), 6.28 (dd, *J* = 1.7/3.9 Hz, 1 H, pyrrole 3-H), 6.80 (br. t, *J* = 2.2 Hz, 1 H, pyrrole 5-H). − $C_{12}H_{12}F_3NO_7S$ (371.28): calcd. C 38.82, H 3.26, N 3.77; found C 38.64, H 3.21, N 3.73.

(*Z*)-3b: Eluent pentane/Et_2O (3:1); oil; yield 18%. − IR: $\tilde{\nu}$ = 1718, 1580 cm^{-1}. − ^1H NMR: δ = 3.54 (s, 3 H, NMe), 3.74 (s, 3 H, OMe), 3.87 (s, 3 H, OMe), 6.17 (dd, *J* = 2.8/3.9 Hz, 1 H, pyrrole 4-H), 6.28 (dd, *J* = 1.7/3.9 Hz, 1 H, pyrrole 3-H), 6.80 (br. t, *J* = 2.2 Hz, 1 H, pyrrole 5-H). − $C_{12}H_{12}F_3NO_7S$ (371.28): calcd. C 38.82, H 3.26, N 3.77; found C 38.61, H 3.18, N 3.72.

3c: Eluent pentane/Et_2O (1:1); oil; yield 90%. − IR (film): $\tilde{\nu}$ = 1722, 1600 cm^{-1}. − ^1H NMR: δ = 1.30−1.40 (m, 6 H, CH_3), 4.30−4.50 (m, 4 H, OCH_2), 7.13 (dd, *J* = 3.9/5.1 Hz, 1 H, thiophene 4-H), 7.46 (dd, *J* = 1.1/3.9 Hz, 1 H, thiophene 3-H), 7.66 (dd, *J* = 1.1/5.1 Hz, 1 H, thiophene 5-H). − $C_{13}H_{13}F_3O_7S_2$ (402.36): calcd. C 38.81, H 3.26; found C 38.69, H 3.20.

3d: Prepared starting from compound **8**; eluent pentane/CH_2Cl_2 (1:1); m.p. 59−60°C (pentane/Et_2O); yield 80%. − IR: $\tilde{\nu}$ = 1738, 1708, 1602 cm^{-1}. − ^1H NMR: δ = 1.36 (t, *J* = 7.2 Hz, 3 H, NCH_3), 1.41 (t, *J* = 7.2 Hz, 3 H, CH_3), 1.49 (t, *J* = 7.2 Hz, 3 H, CH_3), 4.40 (m, 4 H, OCH_2), 4.54 (q, *J* = 7.2 Hz, 2 H, OCH_2), 7.29−7.40 (m, 2 H, indole 5-H and 6-H), 7.62 (dd, *J* = 1.2/7.6 Hz, 1 H, indole 4-H), 8.03 (s, 1 H, indole 2-H), 8.25 (d, *J* = 7.6 Hz, 1 H, indole 7-H). − $C_{20}H_{20}F_3NO_9S$ (507.44): calcd. C 47.34, H 3.97, N 2.76; found C 47.20, H 3.85, N 2.72.

Alkyl 1,2-Diarylbut-2-enedioates 5. − General Method: Compound **3** (2 mmol) was dissolved in anhydrous THF (40 mL). To this solution were added LiCl (254 mg, 6 mmol), CuI (190 mg, 1 mmol), $Pd(Ph_3P)_4$ (60 mg, 0.04 mmol), and the aryltributylstannane **4** (3 mmol). The reaction mixture was heated under reflux for the specified time, the solvent was evaporated, and the residue was purified by chromatography on silica gel using the eluent indicated, to give pure compound **5**.

5aa: Prepared from **3a** and **4a**; reflux for 2 h; eluent pentane/CH_2Cl_2 (3:1); m.p. 82°C (pentane/Et_2O); yield 93%. − IR: $\tilde{\nu}$ = 1703, 1698 cm^{-1}. − ^1H NMR: δ = 1.21 (t, *J* = 7.2 Hz, 3 H, CH_3), 1.42 (t, *J* = 7.2 Hz, 3 H, CH_3), 4.20 (q, *J* = 7.2 Hz, 2 H, OCH_2), 4.45 (q, *J* = 7.2 Hz, 2 H, OCH_2), 6.88 (dd, *J* = 3.8/5.0 Hz, 1 H, thiophene 4-H), 7.03 (dd, *J* = 1.1/3.8 Hz, 1 H, thiophene 3-H), 7.28 (m, 3 H, arom.), 7.40 (m, 3 H, arom.). − $C_{18}H_{18}O_4S$ (330.40): calcd. C 65.44, H 5.49; found C 65.33, H 5.42.

5ac: Prepared from **3a** and **4c**; reflux for 2 h; eluent pentane/CH_2Cl_2 (2:1) to CH_2Cl_2; m.p. 109°C (pentane/Et_2O); yield 70%. − IR: $\tilde{\nu}$ = 1712, 1600 cm^{-1}. − ^1H NMR: δ = 1.23 (t, *J* = 7.2 Hz, 3 H, CH_3), 1.37 (t, *J* = 7.2 Hz, 3 H, CH_3), 4.22 (q, *J* = 7.2 Hz, 2 H, OCH_2), 4.38 (q, *J* = 7.2 Hz, 2 H, OCH_2), 6.28 (s, 1 H, indole 3-H), 6.80 (m, 2 H, arom.), 7.00 (m, 2 H, arom.), 7.10−7.60 (m, 7 H, arom.), 7.90 (m, 2 H, arom.), 8.04 (d, *J* = 8.3 Hz, 1 H, indole 7-H). − $C_{28}H_{25}NO_6S$ (503.57): calcd. C 66.78, H 5.00, N 2.78; found C 66.69, H 4.91, N 2.71.

5ad: Prepared from **3a** and **4d**; reflux for 2 h; eluent pentane to CH_2Cl_2; m.p. 118°C (pentane/CH_2Cl_2); yield 72%. − IR: $\tilde{\nu}$ = 1711, 1690 cm^{-1}. − ^1H NMR: δ = 1.23 (t, *J* = 7.2 Hz, 3 H, CH_3), 1.32 (t, *J* = 7.2 Hz, 3 H, CH_3), 4.27 (q, *J* = 7.2 Hz, 2 H, OCH_2), 4.32 (q, *J* = 7.2 Hz, 2 H, OCH_2), 7.05−7.40 (m, 11 H, arom.), 7.52 (m, 1 H, arom.), 7.61 (m, 2 H, arom.), 7.88 (d, *J* = 8.2 Hz, 1 H, indole 7-H). − $C_{28}H_{25}NO_6S$ (503.57): calcd. C 66.78, H 5.00, N 2.78; found C 66.70, H 4.90, N 2.70.

(*E*)-5ba: Prepared from (*E*)-**3b** and **4a**; reflux for 1.5 h; eluent hexane/CH_2Cl_2 (2:1); m.p. 100−101°C (hexane/Et_2O); yield 92%. − IR: $\tilde{\nu}$ = 1719, 1684 cm^{-1}. − ^1H NMR: δ = 3.32 (s, 3 H, NMe), 3.76 (s, 3 H, OMe), 4.00 (s, 3 H, OMe), 6.13 (dd, *J* = 1.7/3.7 Hz, 1 H, pyrrole 3-H), 6.27 (dd *J* = 2.6/3.7 Hz, 1 H, pyrrole 4-H), 6.81 (m, 1 H, pyrrole 5-H), 6.95 (dd, *J* = 3.8/5.1 Hz, 1 H, thiophene 4-H), 7.10 (dd, *J* = 1.1/3.8 Hz, 1 H, thiophene 3-H), 7.38 (dd, *J* = 1.1/5.1 Hz, 1 H, thiophene 5-H). − $C_{15}H_{15}NO_4S$ (305.35): calcd. C 59.00, H 4.95, N 4.59; found C 58.79, H 4.90, N 4.51.

(*Z*)-5ba: Prepared from (*Z*)-**3b** and **4a**; reflux for 1.5 h; eluent hexane/CH_2Cl_2 (2:1); m.p. 106−107°C (hexane/Et_2O); yield 92%. − IR: $\tilde{\nu}$ = 1709, 1695 cm^{-1}. − ^1H NMR: δ = 3.57 (s, 3 H, NMe), 3.62 (s, 3 H, OMe), 3.72 (s, 3 H, OMe), 6.10 (dd, *J* = 2.6/3.7 Hz, 1 H, pyrrole 4-H), 6.24 (dd, *J* = 1.8/3.7 Hz, 1 H, pyrrole 3-H), 6.67 (m, 1 H, pyrrole 5-H), 7.03 (dd, *J* = 3.7/5.1 Hz, 1 H, thiophene 4-H), 7.16 (dd, *J* = 1.3/3.7 Hz, 1 H, thiophene 3-H), 7.41 (dd, *J* = 1.3/5.1 Hz, 1 H, thiophene 5-H). − $C_{15}H_{15}NO_4S$ (305.35): calcd. C 59.00, H 4.95, N 4.59; found C 58.82, H 4.92, N 4.53.

5bb: Prepared from **3b** and **4b**; reflux for 1.5 h; eluent pentane/CH_2Cl_2 (2:1); m.p. 96°C (pentane/Et_2O), yield 71%. − IR: $\tilde{\nu}$ = 1710, 1692, 1580 cm^{-1}. − ^1H NMR: δ = 3.32 (s, 3 H, NMe), 3.77 (s, 3 H, OMe), 3.99 (s, 3 H, OMe), 5.29 (d, *J* = 4.0 Hz, 1 H, furan 3-H), 6.09 (dd, *J* = 1.8/3.7 Hz, 1 H, pyrrole 3-H), 6.23 (dd, *J* = 2.6/3.7 Hz, 1 H, pyrrole 4-H), 6.33 (dd, *J* = 1.7/3.6 Hz, 1 H, furan

4-H), 6.75 (dd, J = 1.8/2.6 Hz, 1 H, pyrrole 5-H), 7.44 (d, J = 1.7 Hz, 1 H, furan 5-H). − $C_{15}H_{15}NO_5$ (289.28): calcd. C 62.28, H 5.23, N 4.84; found C 62.12, H 5.10, N 4.75.

5bc: Prepared from **3b** and **4c**; reflux for 2 h; eluent pentane/CH_2Cl_2 (1:1); m.p. 131−132°C (pentane/Et_2O); yield 95%. − IR: $\tilde{\nu}$ = 1720, 1702, 1560 cm^{-1}. − ^1H NMR: δ = 3.45 (s, 3 H, NMe), 3.62 (s, 3 H, OMe), 3.91 (s, 3 H, OMe), 5.67 (dd, J = 1.7/3.9 Hz, 1 H, pyrrole 3-H), 5.87 (dd, J = 2.6/3.9 Hz, 1 H, pyrrole 4-H), 6.45 (s, 1 H, indole 3-H), 6.55 (dd, J = 1.7/2.6 Hz, 1 H, pyrrole 5-H), 7.25 (m, 2 H, arom.), 7.40−7.55 (m, 4 H, arom.), 7.85 (m, 3 H, arom.). − $C_{25}H_{22}N_2O_6S$ (478.52): calcd. C 62.75, H 4.63, N 5.85; found C 62.78, H 4.58, N 5.72.

5bd: Prepared from **3b** and **4d**; reflux for 2 h; eluent pentane/CH_2Cl_2 (1:1) to CH_2Cl_2/Et_2O (30:1); m.p. 152−153°C (pentane/CH_2Cl_2); yield 85%. − IR: $\tilde{\nu}$ = 1719, 1695 cm^{-1}. − ^1H NMR: δ = 3.08 (s, 3 H, NMe), 3.84 (s, 3 H, OMe), 3.85 (s, 3 H, OMe), 6.10 (m, 2 H, pyrrole), 6.52 (d, J = 2.1 Hz, 1 H, pyrrole 5-H), 7.07 (s, 1 H, indole 2-H), 7.20 (m, 3 H, arom.), 7.50 (m, 3 H, arom.), 7.80 (m, 2 H, arom.), 7.95 (d, J = 8.1 Hz, 1 H, indole 7-H). − $C_{25}H_{22}N_2O_6S$ (478.52): calcd. C 62.75, H 4.63, N 5.85; found C 62.63, H 4.55, N 5.80.

5cb: Prepared from **3c** and **4b**; reflux for 1.5 h; eluent pentane/Et_2O (3:1); m.p. 48°C (pentane); yield 98%. − IR: $\tilde{\nu}$ = 1718, 1698, 1590 cm^{-1}. − ^1H NMR: δ = 1.30 (m, 6 H, CH_3), 4.35 (m, 4 H, OCH_2), 6.08 (d, J = 3.7 Hz, 1 H, furan 3-H), 6.38 (dd, J = 1.8/3.6 Hz, 1 H, furan 4-H), 7.04 (m, 2 H, thiophene), 7.43 (m, 2 H, arom.). − $C_{16}H_{16}O_5S$ (320.36): calcd. C 59.99, H 5.03; found C 59.88, H 4.99.

5cc: Prepared from **3c** and **4c**; reflux for 1 h; eluent pentane/CH_2Cl_2 (1:1); m.p. 142°C (Et_2O); yield 98%. − IR: $\tilde{\nu}$ = 1718, 1698, 1595 cm^{-1}. − ^1H NMR: δ = 1.24 (t, J = 7.2 Hz, 3 H, CH_3), 1.52 (t, J = 7.2 Hz, 3 H, CH_3), 4.25 (m, 2 H, OCH_2), 4.57 (q, J = 7.2 Hz, 2 H, OCH_2), 6.79 (s, 1 H, indole 3-H), 6.80 (dd, J = 3.8/5.1 Hz, 1 H, thiophene 4-H), 6.92 (dd, J = 1.1/5.1 Hz, 1 H, thiophene 5-H), 7.06 (dd, J = 1.1/3.8 Hz, 1 H, thiophene 3-H), 7.14 (m, 2 H, arom.), 7.30 (m, 2 H, arom.), 7.48 (m, 1 H, indole), 7.62 (d, J = 7.7 Hz, 1 H, indole 4-H), 7.90 (m, 2 H, arom.), 8.39 (d, J = 7.7 Hz, 1 H, indole 7-H). − $C_{26}H_{23}NO_6S_2$ (509.59): calcd. C 61.28, H 4.55, N 2.75; found C 61.16, H 4.46, N 2.68.

5da: Prepared from **3d** and **4a**; reflux for 1.5 h; eluent pentane to CH_2Cl_2; m.p. 88−89°C (pentane/Et_2O); yield 98%. − IR: $\tilde{\nu}$ = 1718, 1698, 1690 cm^{-1}. − ^1H NMR: δ = 1.20 (t, J = 7.2 Hz, 3 H, CH_3), 1.44 (t, J = 7.2 Hz, 3 H, CH_3), 1.48 (t, J = 7.2 Hz, 3 H, CH_3), 4.21 (q, J = 7.2 Hz, 2 H, OCH_2), 4.50 (m, 4 H, OCH_2), 6.86 (dd, J = 3.7/5.1 Hz, 1 H, thiophene 4-H), 7.12 (dd, J = 1.3/3.7 Hz, 1 H, thiophene 3-H), 7.15 (m, 2 H, arom.), 7.35 (m, 2 H, arom.), 7.68 (s, 1 H, indole 2-H), 8.23 (d, J = 8.4 Hz, 1 H, indole 7-H). − $C_{23}H_{23}NO_6S$ (441.50): calcd. C 62.57, H 5.25, N 3.17; found C 62.46, H 5.19, N 3.11.

5db: Prepared from **3d** and **4b**; reflux for 1 h; eluent pentane/Et_2O (3:1); m.p. 85°C (pentane/Et_2O); yield 99%. − IR: $\tilde{\nu}$ = 1738, 1719, 1700 cm^{-1}. − ^1H NMR: δ = 1.22 (t, J = 7.2 Hz, 3 H, CH_3), 1.42 (t, J = 7.2 Hz, 3 H, CH_3), 1.48 (t, J = 7.2 Hz, 3 H, CH_3), 4.23 (q, J = 7.2 Hz, 2 H, OCH_2), 4.48 (m, 4 H, OCH_2), 6.10 (d, J = 3.5 Hz, 1 H, furan 3-H), 6.23 (dd, J = 1.8/3.5 Hz, 1 H, furan 4-H), 7.15 (m, 1 H, arom.), 7.30 (m, 3 H, arom.), 7.66 (s, 1 H, indole 2-H), 8.21 (d, J = 8.4 Hz, 1 H, indole 7-H). − $C_{23}H_{23}NO_7$ (425.44): calcd. C 64.93, H 5.45, N 3.29; found C 64.81, H 5.40, N 3.21.

5dc: Prepared from **3d** and **4c**; reflux for 1.5 h; eluent pentane/Et_2O (2:1); m.p. 143−145°C (Et_2O); yield 87%. − IR: $\tilde{\nu}$ = 1733, 1721, 1710, 1595 cm^{-1}. − ^1H NMR: δ = 1.16 (t, J = 7.2 Hz, 3 H, CH_3), 1.25 (t, J = 7.2 Hz, 3 H, CH_3), 1.38 (t, J = 7.2 Hz, 3 H, CH_3), 4.22 (m, 4 H, OCH_2), 4.44 (q, J = 7.2 Hz, 2 H, OCH_2), 6.49 (s, 1 H, indole 3′-H), 6.75 (s, 1 H, indole 2-H), 7.15−7.45 (m, 7 H, arom.), 7.62 (m, 2 H, arom.), 7.90 (m, 2 H, arom.), 8.06 (m, 2 H, arom.). − ^1H NMR (C_6D_6): δ = 0.72 (t, J = 7.1 Hz, 3 H, CH_3), 1.10 (t, J = 7.1 Hz, 3 H, CH_3), 1.23 (t, J = 7.1 Hz, 3 H, CH_3), 3.70 (q, J = 7.1 Hz, 2 H, $NCOOCH_2$), 4.20 (q, J = 7.1 Hz, 2 H, OCH_2), 4.49 (q, J = 7.1 Hz, 2 H, OCH_2), 6.35 (s, 1 H, 3′-H), 6.74 (m, 3 H, arom.), 6.96 (t, J = 7.4 Hz, 1 H, indole 5-H), 7.07 (m, 2 H, indole), 7.11 (t, J = 8.4 Hz, 1 H, indole 6-H), 7.24 (m, 2 H, indole), 8.03 (m, 2 H, arom.), 8.20 (m, 1 H, 4′-H), 8.34 (d, J = 8.4 Hz, 1 H, indole 7-H), 8.40 (br. s, 1 H, indole 7′-H). − $C_{33}H_{30}N_2O_8S$ (614.67): calcd. C 64.48, H 4.92, N 4.56; found C 64.35, H 4.82, N 4.45.

5dd: Prepared from **3d** and **4d**; reflux for 3 h; eluent pentane/CH_2Cl_2 (1:1) to CH_2Cl_2; m.p. 175−176°C (Et_2O); yield 90%. − IR: $\tilde{\nu}$ = 1730, 1719, 1697 cm^{-1}. − ^1H NMR ([D_6]DMSO): δ = 1.26 (t, J = 7.2 Hz, 3 H, CH_3), 1.31 (t, J = 7.2 Hz, 3 H, CH_3), 1.37 (t, J = 7.2 Hz, 3 H, CH_3), 4.25 (m, 4 H, OCH_2), 4.38 (q, J = 7.2 Hz, 2 H, OCH_2), 6.98 (t, J = 7.1 Hz, 1 H, indole), 7.15 (m, 2 H, arom.), 7.28 (m, 3 H, arom.), 7.50 (m, 2 H, arom.), 7.62 (s, 1 H, indole 2-H), 7.70 (m, 3 H, arom.), 7.76 (s, 1 H, indole 2′-H), 7.80 (d, J = 8.1 Hz, 1 H, indole 7′-H), 8.03 (d, J = 8.2 Hz, 1 H, indole 7-H). − $C_{33}H_{30}N_2O_8S$ (614.67): calcd. C 64.48, H 4.92, N 4.56; found C 64.37, H 4.81, N 4.58.

Diethyl 2,3-Diphenylmaleate (5ae):[16] Compound **3a** (900 mg, 2.28 mmol) was dissolved in DMF (6 mL) and n-Bu_3SnPh (**4e**) (1.1 mL, 3.4 mmol), Ph_3P (60 mg, 0.23 mmol) and tris(dibenzylideneacetone)palladium (105 mg, 0.11 mmol) were added. The resulting mixture was heated at 75−80°C for 3 h, then poured into a saturated NaCl solution (150 mL), and extracted with Et_2O (2 × 30 mL). The organic phase was dried, filtered, and the solvent was evaporated. Purification of the residue by chromatography on silica gel with pentane/CH_2Cl_2 (3:1) as eluent afforded **5ae**; m.p. 56°C, yield 88%.

Diarylmaleic Anhydrides 6. − General Method: To a solution of the appropriate compound **5** (1.5 mmol) in EtOH (30 mL) was added a solution of NaOH (540 mg, 13.5 mmol) in EtOH (10 mL) and H_2O (4 mL). The mixture was heated to reflux for 30 min. The solvent was then evaporated, the residue was acidified with 5% HCl, and extracted with CH_2Cl_2 (2 × 20 mL). The organic phase was dried, filtered, and the solvent was evaporated. The residue was purified either by chromatography on silica gel or by crystallization.

6aa: Purified by chromatography on silica gel, eluent pentane/CH_2Cl_2 (1:1); m.p. 149°C (sublimes) (pentane/CH_2Cl_2); yield 60%. − IR: $\tilde{\nu}$ = 1800, 1732, 1600 cm^{-1}. − ^1H NMR: δ = 7.11 (dd, J = 3.9/5.1 Hz, 1 H, thiophene 4-H), 7.52−7.55 (m, 5 H), 7.59 (dd, J = 1.1/5.1 Hz, 1 H, thiophene 5-H), 7.95 (dd, J = 1.1/3.9 Hz, 1 H, thiophene 3-H). − $C_{14}H_8O_3S$ (256.28): calcd. C 65.61, H 3.15; found C 65.81, H 3.09.

6ac: Purified by chromatography on silica gel, eluent pentane/CH_2Cl_2 (1:1); m.p. 259°C (pentane/CH_2Cl_2); yield 98%. − IR: $\tilde{\nu}$ = 3360, 1800, 1738, 1604 cm^{-1}. − ^1H NMR: δ = 7.12 (m, 1 H), 7.35 (m, 3 H, arom.), 7.60 (m, 4 H, arom.), 7.70 (m, 2 H, arom.), 9.55 (s, 1 H, H/D exchangeable). − $C_{18}H_{11}NO_3$ (289.29): calcd. C 74.73, H 3.83, N 4.84; found C 74.52, H 3.69, N 4.75.

6ad: Purified by chromatography on silica gel, eluent pentane/CH_2Cl_2 (1:2); m.p. 218−219°C (hexane/CH_2Cl_2); yield 68%. − IR: $\tilde{\nu}$ = 3395, 1803, 1730 cm^{-1}. − ^1H NMR: δ = 6.39 (d, J = 8.1 Hz, 1 H, indole 7-H), 6.84 (dt, J = 1.0/8.1 Hz, 1 H, indole 5-H), 7.20 (dt, J = 1.0/8.1 Hz, 1 H, indole 6-H), 7.40 (m, 4 H, arom.), 7.66

(m, 2 H, arom.), 8.16 (d, J = 3.1 Hz, 1 H, indole 2-H), 8.83 (s, 1 H, H/D exchangeable). − $C_{18}H_{11}NO_3$ (289.29): calcd. C 74.73, H 3.83, N 4.84; found C 74.62, H 3.71, N 4.71.

6ae: m.p. 159−160°C (hexane/AcOEt); yield 86%.

6ba: Purified by chromatography on silica gel, eluent pentane/CH_2Cl_2 (1:2); m.p. 146°C (hexane/CH_2Cl_2); yield 52%. − IR: $\tilde{\nu}$ = 1810, 1740 cm^{-1}. − ^1H NMR: δ = 3.46 (s, 3 H, NMe), 6.35 (dd, J = 2.6/3.9 Hz, 1 H, pyrrole 4-H), 6.66 (dd, J = 1.6/3.9 Hz, 1 H, pyrrole 3-H), 6.93 (m, 1 H, pyrrole 5-H), 7.15 (dd, J = 3.9/5.1 Hz, 1 H, thiophene 4-H), 7.60 (dd, J = 1.1/5.1 Hz, 1 H, thiophene 5-H), 7.91 (dd, J = 1.1/3.9 Hz, 1 H, thiophene 3-H). − $C_{13}H_9NO_3S$ (259.28): calcd. C 60.22, H 3.50, N 5.40; found C 60.11, H 3.41, N 5.36.

6bb: Purified by crystallization; m.p. 128°C (hexane/CH_2Cl_2); yield 98%. − IR: $\tilde{\nu}$ = 1820, 1740, 1610 cm^{-1}. − ^1H NMR: δ = 3.54 (s, 3 H, NMe), 6.33 (dd, J = 2.6/3.9 Hz, 1 H, pyrrole 4-H), 6.63 (dd, J = 1.8/3.6 Hz, 1 H, furan 4-H), 6.76 (dd, J = 1.7/3.9 Hz, 1 H, pyrrole 3-H), 6.94 (m, 1 H, furan 5-H), 7.48 (dd, J = 0.7/3.6 Hz, 1 H, furan 3-H), 7.58 (dd, J = 1.8/2.6 Hz, 1 H, pyrrole 5-H). − $C_{13}H_9NO_4$ (243.22): calcd. C 64.20, H 3.73, N 5.76; found C 64.05, H 3.68, N 5.69.

6bc: Purified by chromatography on silica gel, eluent pentane/Et_2O (2:1); m.p. 218−219°C (CH_2Cl_2/Et_2O); yield 82%. − IR: $\tilde{\nu}$ = 3360, 1802, 1740 cm^{-1}. − ^1H NMR ([D_6]DMSO): δ = 3.54 (s, 3 H, NMe), 6.32 (dd, J = 2.6/3.8 Hz, 1 H, pyrrole 4-H), 6.58 (dd, J = 1.7/3.8 Hz, 1 H, pyrrole 3-H), 6.92 (d, J = 1.7 Hz, 1 H, pyrrole 5-H), 7.05 (dt, J = 1.2/8.0 Hz, 1 H, indole), 7.19 (s, 1 H, indole 3-H), 7.21 (m, 1 H, indole), 7.59 (m, 2 H, indole), 11.27 (s, 1 H, H/D exchangeable). − $C_{17}H_{12}N_2O_3$ (292.28): calcd. C 69.86, H 4.14, N 9.58; found C 69.72, H 4.07, N 9.42.

6bd: Purified by chromatography on silica gel, eluent pentane/CH_2Cl_2 (1:1) to CH_2Cl_2; m.p. 221−223°C (hexane/CH_2Cl_2); yield 69%. − IR: $\tilde{\nu}$ = 3350, 3300, 1810, 1730 cm^{-1}. − ^1H NMR: δ = 3.22 (s, 3 H, NMe), 6.33 (dd, J = 2.7/3.9 Hz, 1 H, pyrrole 4-H), 6.50 (d, J = 8.3 Hz, 1 H, indole 7-H), 6.67 (dd, J = 1.7/3.9 Hz, 1 H, pyrrole 3-H), 6.74 (m, 1 H, pyrrole 5-H), 6.92 (dt, J = 1.1/7.2 Hz, 1 H, indole), 7.25 (dt, J = 1.1/8.3 Hz, 1 H, indole), 7.40 (d, J = 8.3 Hz, 1 H, indole 4-H), 8.16 (d, J = 3.0 Hz, 1 H, indole 2-H), 8.75 (s, 1 H, H/D exchangeable). − $C_{17}H_{12}N_2O_3$ (292.28): calcd. C 69.86, H 4.14, N 9.58; found C 69.72, H 4.07, N 9.46.

6cb: Purified by crystallization; m.p. 184°C (sublimes) (CH_2Cl_2/Et_2O); yield 95%. − IR: $\tilde{\nu}$ = 1802, 1745, 1580 cm^{-1}. − ^1H NMR: δ = 6.73 (dd, J = 1.9/3.7 Hz, 1 H, furan 4-H), 7.24 (m, 2 H, arom.), 7.63 (d, J = 3.7 Hz, 1 H, furan 3-H), 7.75 (d, J = 1.8 Hz, 1 H, thiophene 5-H), 8.33 (d, J = 3.9 Hz, 1 H, thiophene 3-H). − $C_{12}H_6O_4S$ (246.24): calcd. C 58.53, H 2.46; found C 58.43, H 2.50.

6cc: Purified by crystallization; m.p. 221−222°C (CH_2Cl_2/Et_2O); yield 80%. − IR: $\tilde{\nu}$ = 3360, 1800, 1735 cm^{-1}. − ^1H NMR: δ = 7.15 (m, 1 H, indole), 7.29 (d, J = 3.8 Hz, 1 H, thiophene 4-H), 7.35 (m, 2 H, indole), 7.65 (d, J = 7.2 Hz, 1 H, indole), 7.73 (s, 1 H, indole 3-H), 7.74 (dd, J = 1.1/5.1 Hz, 1 H, thiophene 5-H), 8.08 (dd, J = 1.1/3.8 Hz, 1 H, thiophene 3-H), 9.70 (s, 1 H, H/D exchangeable). − $C_{16}H_9NO_3S$ (295.31): calcd. C 65.08, H 3.07, N 4.74; found C 64.92, H 3.01, N 4.61.

6da: Purified by chromatography on silica gel, eluent pentane/CH_2Cl_2 (1:1) to CH_2Cl_2; m.p. 216°C (hexane/CH_2Cl_2); yield 82%. − IR: $\tilde{\nu}$ = 3350, 1800, 1738 cm^{-1}. − ^1H NMR: δ = 6.84 (d, J = 8.1 Hz, 1 H, indole), 7.06 (m, 2 H, arom.), 7.28 (m, 1 H, indole), 7.43 (dd, J = 1.1/3.8 Hz, 1 H, thiophene 3-H), 7.50 (d, J = 8.0 Hz, 1 H, indole), 7.58 (dd, J = 1.0/5.1 Hz, 1 H, thiophene 5-H), 8.00 (d, J = 3.0 Hz, 1 H, indole 2-H), 8.85 (s, 1 H, H/D exchangeable). − $C_{16}H_9NO_3S$ (295.31): calcd. C 65.08, H 3.07, N 4.74; found C 64.95, H 3.02, N 4.63.

6db: Purified by crystallization; m.p. 165°C (hexane/CH_2Cl_2); yield 98%. − IR: $\tilde{\nu}$ = 3310, 1799, 1739 cm^{-1}. − ^1H NMR: δ = 6.40 (dd, J = 1.8/3.6 Hz, 1 H, furan 4-H), 7.09−7.26 (m, 3 H, arom.), 7.35 (d, J = 3.6 Hz, 1 H, furan 3-H), 7.45 (m, 2 H, arom.), 7.99 (d, J = 3.0 Hz, 1 H, indole 2-H), 8.87 (s, 1 H, H/D exchangeable). − $C_{16}H_9NO_4$ (279.25): calcd. C 68.82, H 3.25, N 5.02; found C 68.61, H 3.18, N 4.92.

6dc: The reaction mixture was heated to reflux for 2 h; product purified by chromatography on silica gel, eluent pentane/CH_2Cl_2 (1:1) to CH_2Cl_2; m.p. > 300°C (Et_2O/acetone); yield 98%. − IR: $\tilde{\nu}$ = 3350, 3300, 1800, 1730 cm^{-1}. − ^1H NMR ([D_6]DMSO): δ = 6.80−6.90 (m, 3 H, arom.), 7.01 (t, J = 7.0 Hz, 1 H, indole), 7.18 (m, 2 H, arom.), 7.43 (d, J = 7.8 Hz, 1 H, indole), 7.55 (m, 2 H, indole), 8.13 (s, 1 H, indole 2-H), 11.26 (s, 1 H, H/D exchangeable), 12.23 (s, 1 H, H/D exchangeable). − $C_{20}H_{12}N_2O_3$ (328.32): calcd. C 73.16, H 3.68, N 8.53; found C 73.01, H 3.58, N 8.49.

6dd:[1] The reaction mixture was heated to reflux for 2.5 h; product purified by chromatography on silica gel, eluent pentane/CH_2Cl_2 (1:1) to CH_2Cl_2/Et_2O (10:1); yield 77%; m.p. 243°C (Et_2O/acetone).

Diethyl 2-(1-Ethoxycarbonyl-1*H*-indol-3-yl)-3-(ethoxycarbonyloxy)-but-2-enedioate (7): To a stirred solution of compound **2d** (2.67 g, 8.8 mmol) in CH_2Cl_2 (70 mL) was added triethylamine (3.68 mL, 26.4 mmol). The mixture was cooled to 0°C, whereupon ethyl chlorocarbonate (2.1 mL, 22 mmol) was slowly added dropwise. After 1 h at room temperature, the solution was washed with H_2O (50 mL). The organic phase was dried, filtered, and the solvent was evaporated. Purification of the residue by chromatography on silica gel (eluent pentane/Et_2O, 2:1) gave pure compound **7**, 3.75 g (96%), oil. − IR (film): $\tilde{\nu}$ = 1758, 1730, 1720 cm^{-1}. − ^1H NMR: δ = 1.26 (t, J = 7.2 Hz, 3 H, CH_3), 1.34 (m, 6 H, CH_3), 1.47 (t, J = 7.2 Hz, 3 H, CH_3), 4.21 (q, J = 7.2 Hz, 2 H, OCH_2), 4.35 (m, 4 H, OCH_2), 4.50 (q, J = 7.2 Hz, 2 H, OCH_2), 7.28 (m, 1 H, indole), 7.37 (m, 1 H, indole), 7.62 (dd, J = 1.1/7.1 Hz, indole 4-H), 7.90 (s, 1 H, indole 2-H), 8.21 (d, J = 8.0 Hz, 1 H, indole 7-H). − $C_{22}H_{25}NO_9$ (447.44): calcd. C 59.06, H 5.63, N 3.13; found C 59.15, H 5.58, N 3.04.

Diethyl 2-(1-Ethoxycarbonyl-1*H*-indol-3-yl)-3-(hydroxy)but-2-enedioate (8): Compound **7** (2.9 g, 6.48 mmol) was dissolved in CH_2Cl_2 (40 mL) and then a 33% ethanolic dimethylamine solution (2 mL, 11 mmol) was added. The reaction mixture was stirred at room temperature for 10 min and then washed with 4.5% aq. HCl (50 mL). The organic phase was dried, filtered, and the solvent was evaporated. Purification of the residue by chromatography on silica gel (eluent pentane/Et_2O, 2:1) gave pure compound **8**, 1.95 g (83%), oil. − IR (film): $\tilde{\nu}$ = 3400, 1725, 1695, 1640 cm^{-1}. − ^1H NMR: δ = 1.18−1.28 (m, 6 H, CH_3), 1.50 (t, J = 7.5 Hz, 3 H, CH_3), 4.20−4.40 (m, 4 H, OCH_2), 4.49 (q, J = 7.5 Hz, 2 H, OCH_2), 7.20−7.40 (m, 3 H, indole), 7.78 (s, 1 H, indole 2-H), 8.19 (m, 1 H, indole 7-H), 13.05 (s, 1 H, H/D exchangeable). − $C_{19}H_{21}NO_7$ (375.38): calcd. C 60.79, H 5.64, N 3.73; found C 60.69, H 5.59, N 3.82.

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