Synthesis and Diels–Alder Reactivity of *ortho*-Carbazolequinones

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Oxidation of 2- and 3-hydroxycarbazoles with Frémy's salt gave the corresponding *ortho*-carbazolequinones. These molecules react as carbodienophiles in Diels–Alder reaction with 1-acetoxy-1,3-butadiene and 1,3-cyclopentadiene to provide the novel benzocarbazolequinone structures 15, 16, 18 and 19.

Key words *ortho*-carbazolequinone; Diels–Alder reaction; benzocarbazolequinone

The carbazole-3,4-quinones represent an important family of carbazole alkaloids. Carquinostatins A and B, lavanduquinocin and carbazoquinocins A—F represent the first carbazole alkaloids containing an *ortho*-quinone system[1] and possess various biologic activities (Chart 1).

The carquinostatins A and B were isolated from *Streptomyces exfoliatus 2419-SVT2* and were shown to be potent neuronal protecting substances.[2,3] Lavanduquinocin was isolated from *Streptomyces viridochromogenes 2942-SVS3* and exhibit a strong neuronal cell protecting activity.[4] The carbazoquinocins A—F were isolated from *Streptomyces violaceus 2448-SVT2* and they showed strong inhibitory activity against lipid peroxydation.[5]

As part of our continuing search for biologically active compounds with a quinone moiety, we were particularly interested in the synthesis and in the Diels–Alder chemistry of carbazolequinones.[6] In a previous work we reported the synthesis,[7,8] the induction of caspase-dependent cell death[9] and the *Toxoplasma gondii* purine nucleoside phosphorylase inhibitory activity[10] of some carbazole-1,4-quinones.

The purpose of this work is to obtain carbazole-1,2 and -3,4-dione derivatives and to report their behaviour as dienophiles in [4+2] cycloaddition reactions.

Results and Discussion

Carbazole-1,2-quinone **3**[11] was obtained by oxidation of the commercially available hydroxycarbazole **1** with Frémy's salt. Treatment of **1** with sodium hydride in the presence of methyl iodide in the solvent system THF–DMF, gave chemoselectively the *N*-methylated compound **2**.[12] Then, oxidation of **2** with Frémy's salt afforded carbazole-1,2-quinone **4**.

On the other hand, carbazole-3,4-quinone **7** was obtained in two steps from the commercially available 3-aminocarbazole **5**. The formation of the corresponding diazonium salt at 0 °C followed by heating the latter in highly acidic aqueous solution gave the 3-hydroxycarbazole **6**, which was oxidized as above to yield quinone **7** (Chart 2).

Our attempts to obtain **7** through a direct oxidation of **5** with Frémy's salt provided the dimeric carbazole compounds **12** and **13** only. The reaction may proceed first by formation of a mixture of amino phenol **8** and *ortho*-quinone imine **9**. Then, a nucleophilic addition of primary amine **5** or **8** to quinone imine **9** led to intermediates **10** and **11** which were subsequently oxidized to the dimeric carbazoles **12** and **13** (Chart 3). Aromatic amines which have a position *para* to the amino group substituted by an alkyl or alkoxy group have been reported to undergo a similar reaction.[13,14]

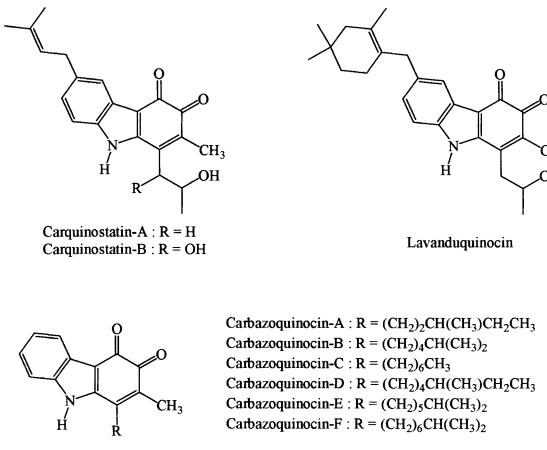

Chart 1

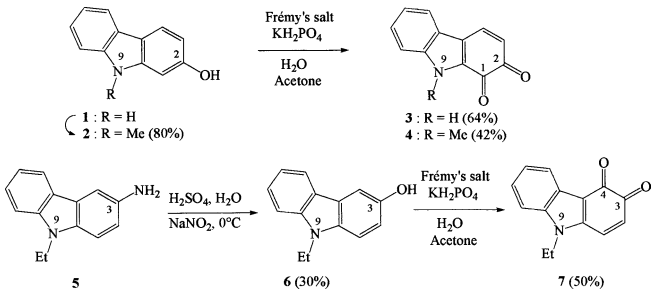

Chart 2

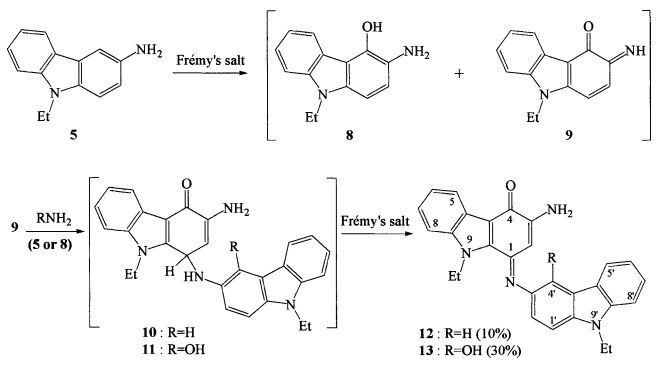

Chart 3

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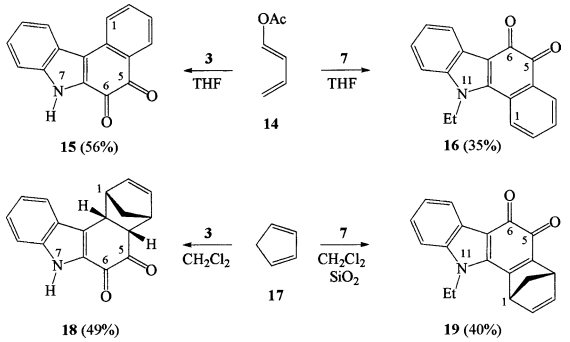

Chart 4

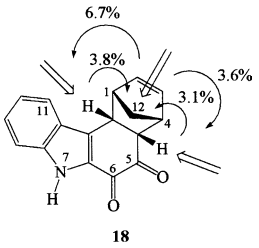

Chart 5

The Diels–Alder reactions of *ortho*-quinones previously reported have been essentially performed with carbodienes. Recently, our group reported the first examples of [4+2] cycloaddition of *ortho*-benzofurandione[15] and *ortho*-indoloquinone[16] towards 1-azadienes. Thus the *ortho*-benzofurandione reacts as a carbodienophile and *ortho*-indoloquinone can react either as a carbodienophile or as heterodienophile.

In continuation of this work focused on heterocyclic *ortho*-quinones we planned to investigate the chemical behaviour of *ortho*-carbazolequinones **3** and **7** towards carbodienes and 1-azadienes.

The [4+2] cycloaddition between carbazolequinone **3** or **7** and 1-acetoxy-1,3-butadiene **14** was performed at reflux in dry THF and provided directly the benzocarbazolequinones **15** and **16**, respectively. The reaction between 1,3-cyclopentadiene **17** and carbazolequinone **3**, carried out in dichloromethane at −10 °C for 5 h then at room temperature for 12 h led to the stable cycloadduct endo **18**. In contrast, reaction of **7** with 1,3-cyclopentadiene **17** as above gave an unstable tetrahydro cycloadduct which was converted into the dihydro derivative **19** after its treatment with silicagel at 40 °C (Chart 4).

The stereochemistry endo of **18** was confirmed by ^{1}H-NMR NOE DIFF experiment (Chart 5). Irradiation at 3.42 ppm (H-4a) gives responses on H-11c at 4.12 ppm and H-12 at 1.71 ppm. Irradiation at 4.12 ppm (H-11c) gives responses on H-4a and H-12. Finally, an irradiation at 1.71 ppm (H-12) gives two responses on H-4a and H-11c.

Attempted cycloadditions of *ortho*-carbazolequinones **3** and **7**, as dienophiles, with different 1-azadienes gave a complex mixture of products and failed to yield the expected pyridocarbazolequinones, probably due to the versatile reactivity profile of *ortho*-quinones in Diels–Alder reactions. Potentially they can react as carbodienes, heterodienes or dienophiles.

Experimental

Melting points were taken in a capillary tube using a Büchi 510 apparatus. The IR spectra were obtained on a Perkin–Elmer 1310 spectrophotometer. The NMR spectra were recorded with a Bruker AM 300 spectrometer (^{1}H-NMR: 300 MHz, ^{13}C-NMR: 75 MHz). Chemical shifts are reported in ppm using tetramethylsilane (TMS) as an internal reference. Coupling constant values are given in Hz. Elemental analyses were performed at the Centre de Microanalyse du CNRS at Solaize, France.

2-Hydroxy-9-methyl-9*H*-carbazole (2) A solution of 2-hydroxycarbazole **1** (0.457 g, 2.5 mmol) and DMF (0.37 ml, 4.76 mmol) in dry THF (5 ml) was added dropwise to 60% NaH (0.25 g, 6.25 mmol) under a nitrogen atmosphere and stirring at room temperature. After 10 min, CH_3I (0.17 ml, 2.75 mmol) was added and the stirring was continued for 2 h. The resulting mixture was cooled to 0 °C and quenched with water (2 ml). After removing the solvent under vacuum, the crude product was washed with acidic aqueous solution and purified by column chromatography on silica gel with CH_2Cl_2/MeOH (98 : 2) as the eluent. Compound **2** was obtained as a yellow solid in 80% yield (ref. 12, 89%), mp 162 °C (ref. 12, 166—167 °C). IR (KBr) cm^{-1}: 3360. ^{1}H-NMR (DMSO-d_6) δ: 9.53 (1H, s, OH), 7.96 (1H, d, J=7.7 Hz, H-5), 7.89 (1H, d, J=8.3 Hz, H-4), 7.48 (1H, d, J=8.2 Hz, H-8), 7.34 (1H, td, J=8.2, 1.1 Hz, H-7), 7.13 (1H, td, J=7.7, 1.0 Hz, H-6), 6.86 (d, 1H, J=2.0 Hz, H-1), 6.68 (1H, dd, J=8.3, 2.0 Hz, H-3), 3.76 (3H, s, CH_3-9).

General Method for the Oxidation of Hydroxycarbazoles with Frémy's Salt An aqueous solution (20 ml) of Frémy's salt (0.37 g, 1.37 mmol) and potassium dihydrogen orthophosphate (0.02 g, 0.15 mmol) was added to a solution of hydroxycarbazole **1**, **2** or **6** (0.55 mmol) in acetone (20 ml). The reaction mixture was stirred at room temperature for 30 min, extracted with CH_2Cl_2, dried over Na_2SO_4 and concentrated under vacuum.

1,2-Dihydrocarbazole-1,2(9*H*)-dione (3) The quinone **3** was obtained from hydroxycarbazole **1**. The crude mixture was purified by column chromatography on silica gel using CH_2Cl_2/MeOH (95 : 5) as the eluent. Compound **3** was obtained as a dark green solid in 64% yield (ref. 11, 83%), mp 181 °C (ref. 11, 178 °C). IR (KBr) cm^{-1}: 3260, 1665, 1640. ^{1}H-NMR (DMSO-d_6) δ: 12.61 (1H, s, NH), 7.87 (2H, m, H-4, H-5), 7.43—7.18 (3H, m, H-6, H-7, H-8), 5.97 (1H, d, J=9.9 Hz, H-3).

9-Methyl-1,2-dihydrocarbazole-1,2(9*H*)-dione (4) The quinone **4** was obtained from hydroxycarbazole **2**. The crude mixture was purified by column chromatography on silica gel using CH_2Cl_2 as the eluent. Compound **4** was obtained as a dark green solid in 42% yield, mp 177 °C. IR (KBr) cm^{-1}: 1670, 1640. ^{1}H-NMR (DMSO-d_6) δ: 7.90 (2H, m, H-4, H-5), 7.59 (1H, d, J=8.7 Hz, H-8), 7.45 (1H, m, H-6 or H-7), 7.26 (1H, m, H-6 or H-7), 6.01 (1H, d, J=9.8 Hz, H-3), 4.01 (3H, s, CH_3-9). ^{13}C-NMR (DMSO-d_6) δ: 182.7, 172.4, 141.2, 138.2, 131.2, 128.6, 123.9, 123.8, 123.3, 121.9, 112.7, 32.6. HR-MS *m/z*: 211.0630 (Calcd for $C_{13}H_9NO_2$: 211.0633).

3-Hydroxy-9-ethyl-9*H*-carbazole (6) A solution of 3-amino-9-ethylcarbazole **5** (1 g, 4.76 mmol), ice (3 g) and concentrated H_2SO_4 (1 ml) in water (1.5 ml) was stirred at 0 °C. Then, a cooled solution of $NaNO_2$ (0.391 g, 5.11 mmol) in water (1 ml) was added dropwise and the stirring was continued for 10 min. The resulting mixture was added to acidic aqueous solution (3 ml of concentrated H_2SO_4 in 2.5 ml of water) heated at reflux. After 5 min the reaction mixture was poured into ice water (30 ml), extracted with CH_2Cl_2 and dried with Na_2SO_4. After removing the solvent under vacuum, the crude product was purified by column chromatography on silica gel with CH_2Cl_2 as the eluent. Compound **6** was obtained as a beige solid in 15% yield, mp 106 °C. IR (KBr) cm^{-1}: 3300. ^{1}H-NMR ($CDCl_3$) δ: 8.03 (1H, d, J=7.7, 1.1 Hz, H-5), 7.54 (1H, d, J=2.3 Hz, H-4), 7.47 (1H, td, J=8.3, 1.1 Hz, H-7), 7.38 (1H, d, J=8.3 Hz, H-8), 7.27 (1H, d, J=8.7 Hz, H-1), 7.19 (1H, td, J=7.7, 1.1 Hz, H-6), 7.04 (1H, dd, J=8.7, 2.3 Hz, H-2), 4.85 (1H, s, OH), 4.33 (2H, q, J=7.3 Hz, $\underline{CH_2}CH_3$), 1.42 (3H, t, J=7.3 Hz, $CH_2\underline{CH_3}$). *Anal.* Calcd for $C_{14}H_{13}NO \cdot 0.4\,H_2O$: C, 76.97; H, 6.37; N, 6.41. Found: C, 77.16; H, 6.40; N, 6.23.

9-Ethyl-3,4-dihydrocarbazole-3,4(9*H*)-dione (7) The quinone **7** was obtained from hydroxycarbazole **6** by oxidation with Frémy's salt according to the general method described above. The crude mixture was purified by column chromatography on silica gel using CH_2Cl_2/MeOH (99.5 : 0.5) as the eluent. Compound **7** was obtained as a red solid in 50% yield, mp 195 °C. IR (KBr) cm^{-1}: 1670, 1640. ^{1}H-NMR ($CDCl_3$) δ: 8.09 (1H, m, H-5), 7.31—7.15 (4H, m, H-1, H-6, H-7, H-8), 6.16 (1H, d, J=10.2 Hz, H-2), 4.14 (2H, q, J=7.4 Hz, $\underline{CH_2}CH_3$), 1.43 (3H, t, J=7.4 Hz, $CH_2\underline{CH_3}$). ^{13}C-NMR (DMSO-d_6) δ: 184.7, 173.2, 143.9, 138.4, 131.9, 129.5, 126.3, 125.6, 125.5, 121.6, 113.5, 113.1, 39.3, 16.7. *Anal.* Calcd for $C_{14}H_{11}NO_2 \cdot 0.3\,H_2O$: C, 72.90; H, 5.07; N, 6.07. Found: C, 72.81; H, 4.90; N, 6.07.

Oxidation of 3-Amino-9-ethylcarbazole 5 with Frémy's Salt An aqueous solution (200 ml) of Frémy's salt (3.2 g, 11.9 mmol) and potassium dihydrogen orthophosphate (0.16 g, 1.19 mmol) was added to a solution of aminocarbazole **5** (1 g, 4.76 mmol) in acetone (200 ml). The reaction mixture was stirred at room temperature for 1 h, extracted with CH_2Cl_2, dried over $MgSO_4$ and concentrated under vacuum. The crude residue was purified by column chromatography on silica gel using CH_3COOEt/Petroleum ether (20 : 80) as the eluent to give compounds **12** and **13**.

3-Amino-9-ethyl-1-[(9-ethyl-9*H*-carbazol-3-yl)imino]-1,9-dihydro-4*H*-carbazol-4-one (12) Compound **12** was obtained as a dark red solid in 10% yield, mp 236 °C. IR (KBr) cm^{-1}: 3460, 3360, 1635, 1595. ^{1}H-NMR (DMSO-d_6) δ: 8.19 (2H, d, J=7.4 Hz, H), 7.77 (1H, d, J=7.4 Hz), 7.76 (1H, s, H-4′), 7.68 (1H, d, J=8.5 Hz), 7.62 (1H, d, J=8.5 Hz), 7.49—7.33 (3H, m), 7.21—7.13 (2H, m), 6.34 (2H, br s, NH_2), 5.93 (1H, s, H-2), 4.96 (2H, q, J=7.0 Hz, $\underline{CH_2}CH_3$), 4.48 (2H, q, J=7.0 Hz, $\underline{CH_2}CH_3$), 1.45 (3H, t, J=7.0 Hz, $CH_2\underline{CH_3}$), 1.36 (3H, t, J=7.0 Hz, $CH_2\underline{CH_3}$). *Anal.* Calcd for $C_{28}H_{24}N_4O \cdot 0.4\,H_2O$: C, 76.48; H, 5.68; N, 12.74. Found: C, 76.51; H, 5.73; N, 12.59.

3-Amino-9-ethyl-1-[(9-ethyl-4-hydroxy-9*H*-carbazol-3-yl)imino]-1,9-dihydro-4*H*-carbazol-4-one (13) Compound **13** was obtained as a dark blue solid in 30% yield, mp 248 °C. IR (KBr) cm^{-1}: 3500, 3380, 1625, 1585, 1550. ^{1}H-NMR (DMSO-d_6) δ: 9 (1H, s, OH), 8.26 (1H, d, J=7.4 Hz), 8.19 (1H, d, J=7.4 Hz), 7.75 (1H, d, J=8.1 Hz), 7.57 (1H, d, J=8.1 Hz), 7.44—7.32 (3H, m), 7.19 (1H, m), 7.14 (1H, d, J=8.5 Hz), 7.01 (1H, d, J=8.5 Hz), 6.32 (2H, br s, NH_2), 6.04 (1H, s, H-2), 5.03 (2H, q, J=7.0 Hz, $\underline{CH_2}CH_3$), 4.42 (2H, q, J=7.4 Hz, $\underline{CH_2}CH_3$), 1.41 (3H, t, J=7.4 Hz, $CH_2\underline{CH_3}$), 1.35 (3H, t, J=7.0 Hz, $CH_2\underline{CH_3}$). FAB-MS m/z 449 $(M+H)^+$. *Anal.* Calcd for $C_{28}H_{24}N_4O_2 \cdot 0.4\,H_2O$: C, 73.79; H, 5.49; N, 12.29. Found: C, 73.87; H, 5.41; N, 12.36.

Benzo[c]carbazole-5,6(7*H*)-dione (15) 1-Acetoxy-1,3-butadiene **14** (0.503 g, 4.5 mmol) in dry THF (1 ml) was added dropwise to a solution of carbazoledione **3** (0.3 g, 1.52 mmol) in 14 ml of the same solvent. The reaction mixture was stirred at reflux for 15 h. The solvent was evaporated off to afford a residue which was purified by column chromatography on silica gel with CH_2Cl_2/MeOH (95 : 5) as the eluent to give compound **15** as a black solid in 56% yield, mp >300 °C. IR (KBr) cm^{-1}: 3320, 1710, 1680. ^{1}H-NMR ($CDCl_3$) δ: 12.4 (1H, s, NH), 8.29 (1H, d, J=8.3 Hz), 8.18 (1H, d, J=7.6 Hz), 7.89 (1H, dd, J=7.5, 1.3 Hz), 7.68 (1H, td, J=7.6, 1.3 Hz), 7.44 (2H, m), 7.34 (1H, td, J=7.5, 1.0 Hz), 7.25 (1H, td, J=6.6, 1.5 Hz). ^{13}C-NMR (DMSO-d_6) δ: 181.5, 172.3, 141.0, 136.4, 135.7, 132.9, 130.6, 130.5, 128.5, 127.9, 125.6, 123.9, 123.8, 123.3, 122.2, 114.7. *Anal.* Calcd for $C_{16}H_9NO_2 \cdot 0.3\,H_2O$: C, 76.06; H, 3.83; N, 5.54. Found: C, 76.38; H, 4.05; N, 5.55.

11-Ethylbenzo[a]carbazole-5,6(11*H*)-dione (16) Synthesis of compound **16** was performed starting from **7** following the procedure used for **15**. Compound **16** was purified by column chromatography on silica gel with CH_2Cl_2/MeOH (98 : 2) as the eluent and obtained as an orange solid in 35% yield, mp 255 °C. IR (KBr) cm^{-1}: 1690, 1630. ^{1}H-NMR ($CDCl_3$) δ: 8.30 (1H, dd, J=6.6, 1.8 Hz), 8.16 (1H, dd, J=7.6, 1.0 Hz), 7.80 (1H, d, J=7.9 Hz), 7.68 (1H, td, J=7.6, 1.3 Hz), 7.46 (1H, t, J=7.4 Hz), 7.40—7.28 (3H, m), 4.59 (2H, q, J=7.3 Hz, $\underline{CH_2}CH_3$), 1.69 (3H, t, J=7.3 Hz, $CH_2\underline{CH_3}$). ^{13}C-NMR (DMSO-d_6) δ: 182.4, 174.5, 151.3, 143.6, 139.7, 136.3, 132.1, 131.2, 130.8, 129.9, 125.9, 125.82, 125.2, 121.9, 115.1, 112.4, 41.5, 15.6. *Anal.* Calcd for $C_{18}H_{13}NO_2 \cdot 0.25\,H_2O$: C, 77.26; H, 4.86; N, 5.00. Found: C, 77.31; H, 4.80; N, 4.78.

1,4,4a,11c-Tetrahydro-1,4-methanobenzo[c]carbazole-5,6(7*H*)-dione (18) Freshly distilled cyclopentadiene **17** (1.05 g, 15.9 mmol) was added dropwise to a solution of the carbazoledione **3** (1 g, 5.3 mmol) in CH_2Cl_2 (40 ml) stirred and cooled at −10 °C. The stirring was continued for 5 h at −10 °C then for 12 h at room temperature. The solution was concentrated under vacuum and the resulting residue was chromatographed on silica gel using CH_2Cl_2/MeOH (97 : 3) as the eluent. Compound **18** was obtained as a yellow solid in 49% yield, mp 182 °C. IR (KBr) cm^{-1}: 3260 1700, 1640. ^{1}H-NMR ($CDCl_3$) δ: 8.9 (1H, s, NH), 7.87 (1H, d, J=7.9 Hz, H-11), 7.55—7.20 (3H, m, H-8, H-9, H-10), 6.1 (1H, t, J=5.3, 3.0 Hz, H-2 or H-3), 5.58 (1H, t, J=5.3, 3.0 Hz, H-2 or H-3), 4.1 (1H, dd, J=7.2, 3.8 Hz, H-11c), 3.59 (2H, s, H-1, H-4), 3.42 (1H, dd, J=7.2, 3.8 Hz, H-4a), 1.71 (2H, s, H-12). ^{13}C-NMR (DMSO-d_6) δ: 199.1, 172.2, 140.8, 136.3, 135.2, 134.9, 129.4, 126.1, 124.6, 123.4, 121.4, 113.9, 51.5, 49.8, 49.1, 48.2, 37.4. *Anal.* Calcd for $C_{17}H_{13}NO_2 \cdot 0.7\,H_2O$: C, 74.00; H, 5.26; N, 5.07. Found: C, 73.98; H, 4.89; N, 5.23.

11-Ethyl-1,4-dihydro-1,4-methanobenzo[a]carbazole-5,6(11*H*)-dione (19) Freshly distilled cyclopentadiene **17** (0.4 g, 6.06 mmol) was added dropwise to a solution of the carbazoledione **7** (0.15 g, 0.67 mmol) in CH_2Cl_2 (6 ml) stirred and cooled at 0 °C. The stirring was continued for 6 h at 0 °C. Then, 1.5 g of SiO_2 was added and the reaction mixture was stirred for 48 h at 40 °C. The mixture was filtered and SiO_2 washed with CH_2Cl_2. The combined filtrates were concentrated under vacuum. Column chromatography of the residue, eluting with a mixture of CH_2Cl_2/MeOH (98 : 2) gave compound **19** as a dark green solid in 26% yield, mp 191 °C (decomp.). IR (KBr) cm^{-1}: 1625. ^{1}H-NMR ($CDCl_3$) δ: 8.15 (1H, m, J=7.9, 1.1 Hz, H-7), 7.34—7.25 (3H, m, H-8, H-9, H-10), 6.96 (1H, dd, J=4.9, 3.2 Hz, H-2 or H-3), 6.82 (1H, dd, J=4.9, 3.2 Hz, H-2 or H-3), 4.45 (2H, q, J=7.4 Hz, $\underline{CH_2}CH_3$), 4.33 (1H, s, H-1 or H-4), 4.16 (1H, s, H-1 or H-4), 2.36 (2H, m, H-12), 1.58 (3H, t, J=7.4 Hz, $CH_2\underline{CH_3}$). ^{13}C-NMR (DMSO-d_6) δ: 178.6, 176.4, 160.9, 151.4, 151.2, 144.4, 141.2, 138.8, 126.8, 125.6, 125.3, 121.2, 112.7, 111.3, 71.0, 52.3, 47.8, 17.1. *Anal.* Calcd for $C_{19}H_{15}NO_2 \cdot 0.5\,H_2O$: C, 76.49; H, 5.41; N, 4.69. Found: C, 76.56; H, 5.29; N, 4.67.

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