Two directions in spiroheterocyclization of 1*H*-pyrrole-2,3-diones upon the action of 3-arylamino-1*H*-inden-1-ones*

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4-Ethoxycarbonyl-5-phenyl-substituted 1*H*-pyrrole-2,3-diones react with 3-arylamino-1*H*-inden-1-ones in the ratios 1 : 1 and 1 : 2 to form substituted spiro{indeno[1,2-*b*]quinoline-10,3′-pyrroles} and substituted spiro{diindeno[1,2-*b*:2′,1′-*e*]pyridine-11,3′-pyrroles}, respectively.

Key words: 1*H*-pyrrole-2,3-diones, spiroheterocyclization, 3-arylamino-1*H*-inden-1-ones, spiro{indeno[1,2-*b*]quinoline-10,3′-pyrroles}, spiro{diindeno[1,2-*b*:2′,1′-*e*]pyridine-11,3′-pyrroles}.

Earlier, we have shown that the reaction of monocyclic 1*H*-pyrrole-2,3-diones with enamines is directed by the structure of substituents in the pyrroledione ring.[1] 1-Aryl-4-isopropoxalyl-5-phenyl-1*H*-pyrrole-2,3-diones react with enamines to add to the NH and β-CH groups of the enamino fragment to the atoms C(2) and C(3) of pyrrolediones, with the cleavage of the pyrroledione ring and subsequent cyclization. Three ways of the reaction of 1-aryl-4-aroyl-5-methoxycarbonyl-1*H*-pyrrole-2,3-diones with enamines are described. The most frequent is a sequential addition of the β-CH and NH groups of the enamino fragment to the atoms C(5) and COOMe group at position 5 of pyrrolediones.[2—5] A sequential addition of the NH and β-CH groups of the enamino fragment to the atoms C(2) and C(3) of pyrroledione with the cleavage of the pyrroledione ring and subsequent cyclization is reported as a minor direction.[4,5] Enamines unsubstituted at the N atom tend to sequentially add the β-CH and NH groups of the enamino fragment to the atoms C(5) and C(3) of pyrrolediones.[5] The reaction of 1-alkyl-4-ethoxycarbonyl-5-phenyl-1*H*-pyrrole-2,3-diones with *N*-aryl-substituted 3-amino-5,5-dimethylcyclohex-2-enones proceeds as a sequential addition of the *o*-CH group of the aryl substituent and the β-CH group of the enamino fragment of enamines to the pyrroledione atom C(3).[6,7] In order to study the reactivity of 4-ethoxycarbonyl-5-phenyl-1*H*-pyrrole-2,3-diones (ethyl 4,5-dioxo-1-phenyl-4,5-dihydro-1*H*-pyrrole-3-carboxylates), we studied their reactions with the other class of *N*-aryl-substituted cyclic enamines, *viz.*, 3-arylamino-1*H*-inden-1-ones.

The reaction of pyrrolediones **1a—c** with 3-arylamino-1*H*-inden-1-ones **2a—c** in the ratio 1 : 1, carried out by reflux of a solution of reagents in anhydrous *m*-xylene at 139—140 °C over 5—6 h, leads to the substituted ethyl carboxylates **3a—g** (Scheme 1).** The reaction course was monitored by chromatography.

Compounds **3a—g** are orange crystalline compounds with high melting points, readily soluble in DMSO and DMF, poorly soluble in common organic solvents, insoluble in alkanes and water.

In the ^{1}H NMR spectra of compounds **3a—g**, besides the signals for the protons of substituents at position 1′, aromatic rings, and bonded to them groups, a singlet for the NH group in the region δ 10.89—11.04, a triplet and a quartet for the ethyl group of the ethoxycarbonyl substituent in the regions δ 0.59—0.72 and 3.55—3.87, respectively, are also present. In the ^{13}C NMR spectrum of compound **3c**, the signals characteristic of the carbon atoms of the keto, ester, and lactam carbonyl groups at δ 187.83, 179.75, and 161.48, respectively, are present, as well as the signal for the spiro carbon atom at δ 43.88. The structure of compound **3a** was established by X-ray diffraction studies (Fig. 1).

The formation of compounds **3a—g** is apparently effected by initial addition of the β-CH group of the enamino fragment of enamines **2a—c** to the carbon atom at position 3 of pyrrolediones **1a—c** and formation of the

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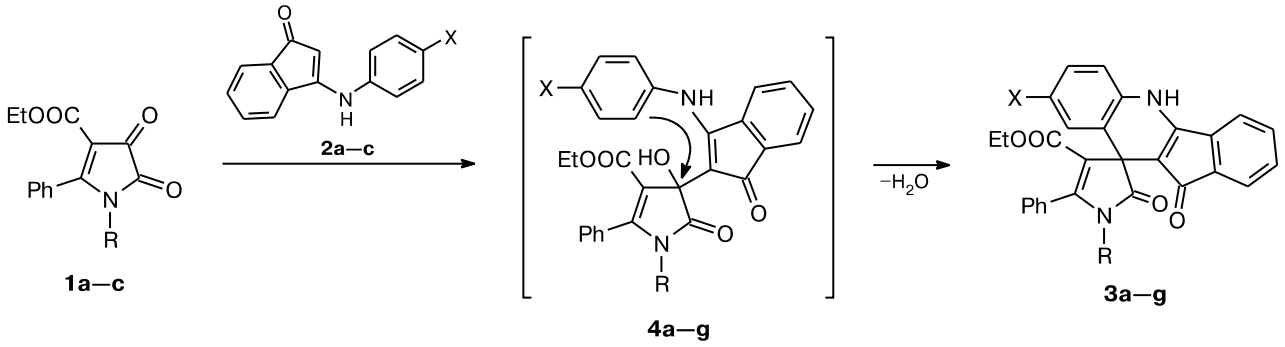
Scheme 1



1: R = CH_2Ph (**a**), Ph (**b**), *cyclo*-C_6H_{11} (**c**);
2: X = H (**a**), Me (**b**), OMe (**c**);
3, 4: R = CH_2Ph, X = H (**a**), Me (**b**), OMe (**c**); R = Ph, X = H (**d**), Me (**e**), OMe (**f**); R = *cyclo*-C_6H_{11}, X = OMe (**g**)

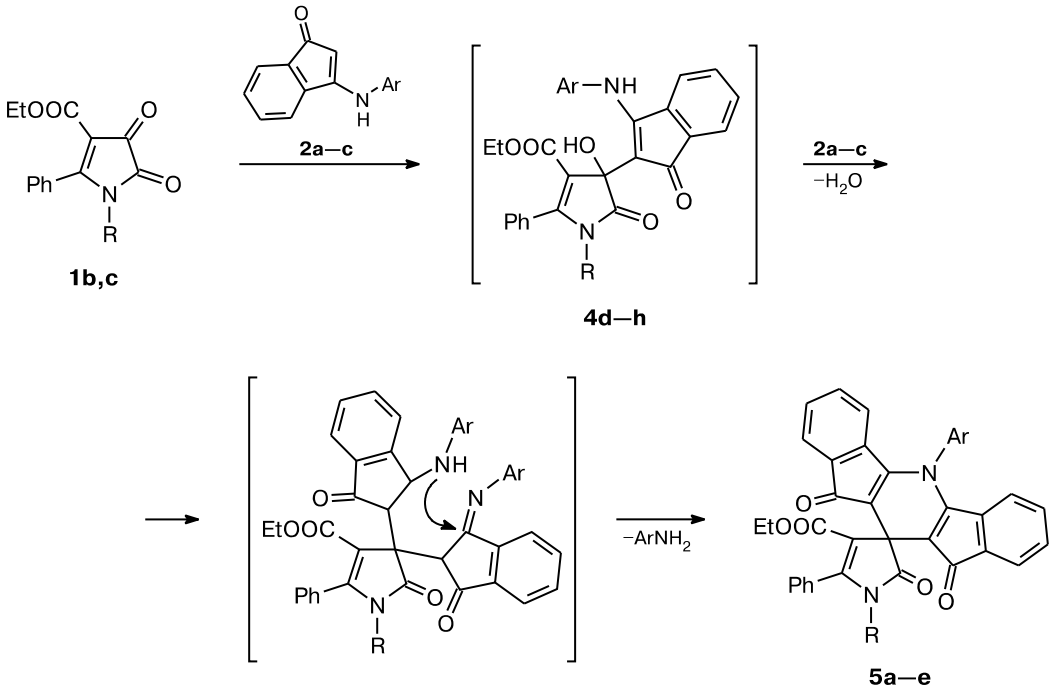
intermediate compounds **4a**—**g** with subsequent intramolecular cyclization involving an activated *o*-CH group of the aryl substituent of enamines **2a**—**c**. We observed a similar scheme of spiroheterocyclization in the reaction of pyrrolediones **1a**,**c** with *N*-aryl-substituted 3-amino-5,5-dimethylcyclohex-2-enones.[6,7]

In the reactions of pyrrolediones **1b**,**c** with indenones **2a**—**c**, besides compounds **3d**—**g**, the substituted ethyl esters **5a**—**d** were isolated as the minor products (Scheme 2). If similar conditions are applied to the reaction of pyrroledione **1c** with enamines **2b**, compound **5e** is isolated as the only product.

Compounds **5a**—**e** are bright crimson crystalline substances, readily soluble in common organic solvents and insoluble in alkanes and water.

In the ^1H NMR spectra of compounds **5a**—**e**, in addition to the signals for the protons of substituents at position 1′, aromatic rings, and bonded to them groups,

Scheme 2



2: Ar = Ph (**a**), C_6H_4Me-4 (**b**), C_6H_4OMe-4 (**c**);
4: R = Ph, Ar = Ph (**d**), C_6H_4Me (**e**), C_6H_4OMe (**f**); R = *cyclo*-C_6H_{11}, Ar = C_6H_4OMe (**g**), C_6H_4Me-4 (**h**);
5: R = Ph, Ar = Ph (**a**), C_6H_4Me-4 (**b**), C_6H_4OMe-4 (**c**); *R* = *cyclo*-C_6H_{11}, Ar = C_6H_4OMe-4 (**d**), C_6H_4Me-4 (**e**)

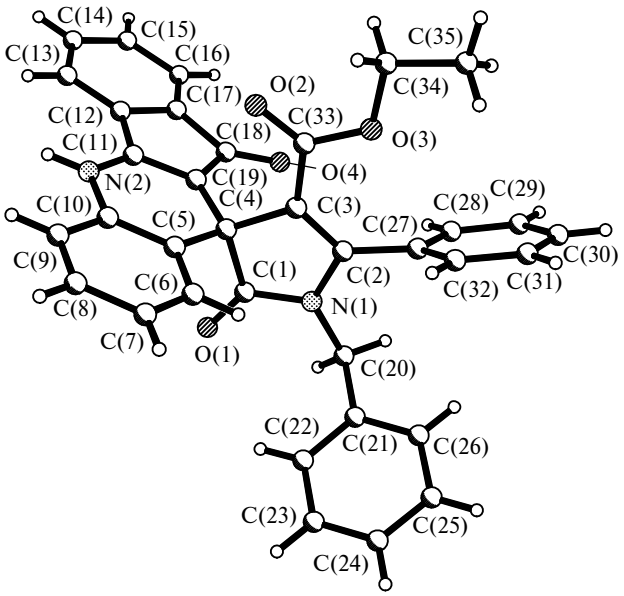


Fig. 1. Molecular structure of **3a**.

a triplet and a quartet for the ethyl group of the ethoxycarbonyl substituent in the regions δ 0.63—0.72 and 3.74—3.87, respectively, are present. In the ^{13}C NMR spectrum of compound **5b**, the signals characteristic of the carbon atoms of the keto, ester, and lactam carbonyl groups at δ 189.88, 177.58, and 162.10, respectively, are found, as well as the signal for the spiro carbon atom at δ 46.90. The structure of compound **5a** was established by X-ray diffraction studies (Fig. 2).

The formation of compounds **5a**—**e** is apparently effected by initial addition of the β-CH group of the enamino fragment of enamines **2a**—**c** to the carbon atom at position 3 of pyrrolediones **1b**,**c** and the formation of the intermediate pyrrolones **4d**—**h** with subsequent assembly of the diindeno[1,2-*b*:2′,1′-*e*]pyridine structural fragment as a result of a sequential nucleophilic addition of the β-CH group and the NH group of the enamino fragment from the second molecule of enamines **2a**—**c** to the carbon atom at position 3 and the imino group of a tautomeric form of pyrrolones **4d**—**h**, respectively, with elimination of water and arylamine.

The synthesis of compounds **5a**—**e** is a three-component reaction, therefore, its conduction with the ratio of reagents 1 : 2, in our opinion, should have led to the increase in the yields of these compounds. However, the use of the ratio 1 : 2 under all the conditions studied gave no noticeable increase in the yields.

The reactions described illustrate an example of a direct spiroheterocyclization of substituted pyrroledione upon the action of *N*-aryl-substituted enamine, involving into the reaction as the CH,CH-1,5-binucleophiles, and a direct three-component spiroheterocyclization of substituted pyrroledione upon the action of enamine, involving into the reaction as the CH,NH-1,3-binucleophiles, as well as represent a convenient preparative approach to the synthesis of the earlier unavailable heterocyclic systems of spiro{indeno[1,2-*b*]quinoline-10,3′-pyrrole} and spiro{diindeno[1,2-*b*:2′,1′-*e*]pyridine-11,3′-pyrrole}.

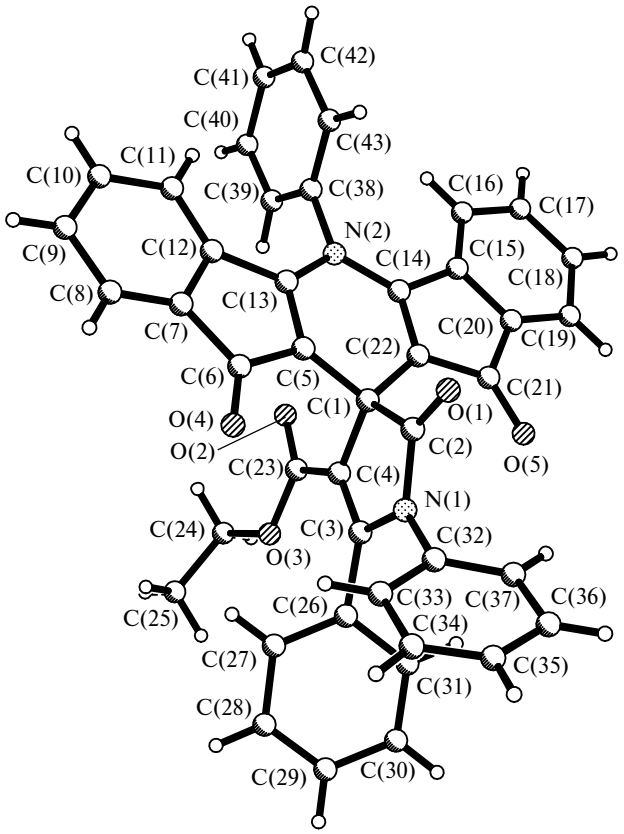


Fig. 2. Molecular structure of **5a**.

It should be noted that the indenopyridine fragment is a structural part of the 4-azafluorenone group alkaloids.[9] Substituted indenopyridines manifest cytitoxic,[10] anti-inflammatory, and antiallergic activity,[11] as well as are phosphodiesterase inhibitors[12] and adenosine A2a receptor antagonists.[13]

Experimental

IR spectra were recorded on a FSM-1201 spectrophotometer. ^1H and ^{13}C NMR spectra were recorded on a Bruker AM-400 spectrometer (^1H 400 MHz; ^{13}C 100 MHz) in DMSO-d_6, using Me_4Si as an internal standard. Individuality of the synthesized compounds was confirmed by TLC on Silufol plates, using ethyl acetate and benzene—ethyl acetate (5 : 1) as eluents.

Ethyl 1′-benzyl-2′,11-dioxo-5′-phenyl-1′,2′,5,11-tetrahydrospiro{indeno[1,2-*b*]quinoline-10,3′-pyrrole}-4′-carboxylate (3a). A solution of pyrroledione **1a** (1.0 mmol) and enamine **2a** (1.0 mmol) in anhydrous *m*-xylene (15 mL) was refluxed for 5 h, cooled, an orange precipitate formed was filtered off. The yield was 54%, m.p. 239—241 °C (ethyl acetate). IR, ν/cm^{-1}: 3252

(NH); 1694, 1688, 1626 (C=O). ^{1}H NMR, δ: 0.60 (t, 3 H, $CH_2C\underline{H}_3$, J = 7.1 Hz); 3.58 (q, 2 H, $C\underline{H}_2CH_3$, J = 7.1 Hz); 4.51, 4.67 (both d, 1 H each, $C\underline{H}_2Ph$, J = 16.3 Hz); 7.08—7.67 (m, 18 H, Ar); 11.02 (s, 1 H, NH). Found (%): C, 77.98; H, 4.84; N, 5.15. $C_{35}H_{26}N_2O_4$. Calculated (%): C, 78.05; H, 4.87; N, 5.20.

Compounds **3b,c** were synthesized similarly.

Ethyl 1′-benzyl-8-methyl-2′,11-dioxo-5′-phenyl-1′,2′,5,11-tetrahydrospiro{indeno[1,2-*b*]quinoline-10,3′-pyrrole}-4′-carboxylate (3b). The yield was 61%, m.p. 290—292 °C (ethyl acetate—1,2-dichloroethane (1 : 1)). IR, ν/cm^{-1}: 3191 (NH); 1678, 1626 (C=O). ^{1}H NMR, δ: 0.61 (t, 3 H, $CH_2C\underline{H}_3$, J = 7.2 Hz); 2.24 (s, 3 H, Me); 3.58 (m, 2 H, $C\underline{H}_2CH_3$); 4.57, 4.61 (both d, 1 H each, $C\underline{H}_2Ph$, J = 16.7 Hz); 6.79—7.65 (m, 17 H, Ar); 10.96 (s, 1 H, NH). Found (%): C, 78.15; H, 5.08; N, 5.03. $C_{36}H_{28}N_2O_4$. Calculated (%): C, 78.24; H, 5.11; N, 5.07.

Ethyl 1′-benzyl-8-methoxy-2′,11-dioxo-5′-phenyl-1′,2′,5,11-tetrahydrospiro{indeno[1,2-*b*]quinoline-10,3′-pyrrole}-4′-carboxylate (3c). The yield was 59%, m.p. 293—294 °C (ethyl acetate). IR, ν/cm^{-1}: 3195 (NH); 1686, 1622 (C=O). ^{1}H NMR, δ: 0.61 (t, 3 H, $CH_2C\underline{H}_3$, J = 7.0 Hz); 3.58 (m, 2 H, $C\underline{H}_2CH_3$); 3.69 (s, 3 H, OMe); 4.57, 4.61 (both d, 1 H each, $C\underline{H}_2Ph$, J = 16.1 Hz); 6.50—7.64 (m, 17 H, Ar); 11.02 (s, 1 H, NH). ^{13}C NMR, δ: 13.12 ($OCH_2\underline{C}H_3$); 43.88 (C(10)); 51.56 ($\underline{C}H_2Ph$); 55.13 (OMe); 58.48 ($O\underline{C}H_2CH_3$); 99.19 (C(4′)); 111.51 (C(10a)); 114.12—136.70, 153.56 (C(5′)); 155.85 (C(8)); 156.19 (C(4b)); 161.48 (C(2′)); 179.75 (<u>C</u>OOEt); 187.83 (C(11)). Found (%): C, 75.97; H, 4.91; N, 4.90. $C_{36}H_{28}N_2O_5$. Calculated (%): C, 76.04; H, 4.96; N, 4.93.

Ethyl 2′,11-dioxo-1′,5′-diphenyl-1′,2′,5,11-tetrahydrospiro{indeno[1,2-*b*]quinoline-10,3′-pyrrole}-4′-carboxylate (3d) and ethyl 2,10,12-trioxo-1′,5′,5-triphenyl-1′,2′,10,12-tetrahydro-5*H*-spiro{diindeno[1,2-*b*:2′,1′-*e*]pyridine-11,3′-pyrrole}-4′-carboxylate (5a). A solution of pyrroledione **1b** (1.0 mmol) and enamine **2a** (1.0 mmol) in anhydrous *m*-xylene (15 mL) was refluxed for 5 h, cooled, an orange precipitate of compound **3d** was filtered off. The yield was 53%, m.p. 279—281 °C (ethyl acetate). IR, ν/cm^{-1}: 3262 (NH); 1697, 1628 (C=O). ^{1}H NMR, δ: 0.66 (t, 3 H, $CH_2C\underline{H}_3$, J = 7.1 Hz); 3.63 (m, 2 H, $C\underline{H}_2CH_3$); 7.11—7.87 (m, 18 H, Ar); 11.07 (s, 1 H, NH). Found (%): C, 77.80; H, 4.57; N, 5.29. $C_{34}H_{24}N_2O_4$. Calculated (%): C, 77.85; H, 4.61; N, 5.34. The mother liquor was concentrated to dryness, the residue was dissolved in boiling ethyl acetate (10 mL), cooled, a crimson precipitate of compound **5a** was filtered off. The yield was 14%, m.p. 336—337 °C (ethyl acetate). IR, ν/cm^{-1}: 1734, 1694 (C=O). ^{1}H NMR, δ: 0.72 (t, 3 H, Me, J = 7.2 Hz); 3.87 (q, 2 H, OCH_2, J = 7.2 Hz); 5.55 (d, 2 H, C(4)H, C(6)H, J = 7.5 Hz); 7.16—8.07 (m, 21 H, Ar). Found (%): C, 79.07; H, 4.29; N, 4.25. $C_{43}H_{28}N_2O_5$. Calculated (%): C, 79.13; H, 4.32; N, 4.29.

Compounds **3e—g** and **5b—d** were synthesized similarly.

The results of X-ray diffraction studies will be published separately.

Ethyl 8-methyl-2′,11-dioxo-1′,5′-diphenyl-1′,2′,5,11-tetrahydrospiro{indeno[1,2-*b*]quinoline-10,3′-pyrrole}-4′-carboxylate (3e) and ethyl 2,10,12-trioxo-1′,5′-diphenyl-5-(4-tolyl)-1′,2′,10,12-tetrahydro-5*H*-spiro{diindeno[1,2-*b*:2′,1′-*e*]pyridine-11,3′-pyrrole}-4′-carboxylate (5b). The yield of compound **3e** was 50%, m.p. 307—308 °C (ethyl acetate). IR, ν/cm^{-1}: 3244 (NH); 1694, 1630 (C=O). ^{1}H NMR, δ: 0.66 (t, 3 H, $CH_2C\underline{H}_3$, J = 7.0 Hz); 2.32 (s, 3 H, Me); 3.63 (m, 2 H, $C\underline{H}_2CH_3$); 7.05—7.66 (m, 17 H, Ar); 11.02 (s, 1 H, NH). Found (%): C, 77.96; H, 4.80; N, 5.14. $C_{35}H_{26}N_2O_4$. Calculated (%): C, 78.05; H, 4.87; N, 5.20. The yield of compound **5b** was 17%, m.p. 338—339 °C (ethyl acetate). IR, ν/cm^{-1}: 1740, 1688 (C=O). ^{1}H NMR, δ: 0.71 (t, 3 H, $CH_2C\underline{H}_3$, J = 7.0 Hz); 2.58 (s, 3 H, Me); 3.86 (q, 2 H, OCH_2, J = 7.0 Hz); 5.61 (d, 2 H, C(4)H, C(6)H, J = 7.5 Hz); 7.16—7.91 (m, 20 H, Ar). ^{13}C NMR, δ: 13.47 ($OCH_2\underline{C}H_3$); 21.01 (Me); 46.90 (C(10)); 58.92 ($O\underline{C}H_2CH_3$); 110.11, 110.41, 121.42, 121.66, 127.57—136.08, 141.55 (C(5a), C(4b)); 156.98 (C(5′)); 162.10 (C(2′)); 177.58 (<u>C</u>OOEt); 189.88 (C(10), C(12)). Found (%): C, 79.20; H, 4.55; N, 4.14. $C_{44}H_{30}N_2O_5$. Calculated (%): C, 79.26; H, 4.54; N, 4.20.

Ethyl 8-methoxy-2′,11-dioxo-1′,5′-diphenyl-1′,2′,5,11-tetrahydrospiro{indeno[1,2-*b*]quinoline-10,3′-pyrrole}-4′-carboxylate (3f) and ethyl 5-(4-methoxyphenyl)-2,10,12-trioxo-1′,5′-diphenyl-1′,2′,10,12-tetrahydro-5*H*-spiro{diindeno[1,2-*b*:2′,1′-*e*]pyridine-11,3′-pyrrole}-4′-carboxylate (5c). The yield of compound **3f** was 52%, m.p. 287—289 °C (ethyl acetate). IR, ν/cm^{-1}: 3231 (NH); 1692, 1618 (C=O). ^{1}H NMR, δ: 0.67 (t, 3 H, $CH_2C\underline{H}_3$, J = 7.2 Hz); 3.64 (m, 2 H, $C\underline{H}_2CH_3$); 3.78 (s, 3 H, OMe); 6.74—7.65 (m, 17 H, Ar); 11.04 (s, 1 H, NH). Found (%): C, 75.73; H, 4.69; N, 5.00. $C_{35}H_{26}N_2O_5$. Calculated (%): C, 75.80; H, 4.73; N, 5.05. The yield of compound **5c** was 16%, m.p. 305—307 °C (benzene—ethyl acetate (1 : 1)). IR, ν/cm^{-1}: 1742, 1692 (C=O). ^{1}H NMR, δ: 0.72 (t, 3 H, $CH_2C\underline{H}_3$, J = 7.0 Hz); 3.86 (q, 2 H, OCH_2, J = 7.0 Hz); 3.97 (s, 3 H, OMe); 5.69 (d, 2 H, C(4)H, C(6)H, J = 7.6 Hz); 7.19—7.95 (m, 20 H, Ar). Found (%): C, 77.36; H, 4.40; N, 4.04. $C_{44}H_{30}N_2O_6$. Calculated (%): C, 77.41; H, 4.43; N, 4.10.

Ethyl 1′-cyclohexyl-8-methoxy-2′,11-dioxo-5′-phenyl-1′,2′,5,11-tetrahydrospiro{indeno[1,2-*b*]quinoline-10,3′-pyrrole}-4′-carboxylate (3g) and ethyl 1′-cyclohexyl-5-(4-methoxyphenyl)-2,10,12-trioxo-5′-phenyl-1′,2′,10,12-tetrahydro-5*H*-spiro{diindeno[1,2-*b*:2′,1′-*e*]pyridine-11,3′-pyrrole}-4′-carboxylate (5d). The yield of compound **3g** was 48%, m.p. 275—276 °C (ethyl acetate). IR, ν/cm^{-1}: 3195 (NH); 1688, 1620 (C=O). ^{1}H NMR, δ: 0.59 (t, 3 H, $CH_2C\underline{H}_3$, J = 7.0 Hz); 0.87—2.07 (m, 10 H, *cyclo*-C_6H_{11}); 3.15 (m, 1 H, *cyclo*-C_6H_{11}); 3.55 (q, 2 H, $C\underline{H}_2CH_3$, J = 7.0 Hz); 3.73 (s, 3 H, OMe); 6.46—7.62 (m, 12 H, Ar); 10.89 (s, 1 H, NH). Found (%): C, 75.11; H, 5.69; N, 5.06. $C_{35}H_{32}N_2O_5$. Calculated (%): C, 74.98; H, 5.75; N, 5.00. The yield of compound **5d** was 18%, m.p. 270—273 °C (benzene—ethyl acetate (1 : 1)). IR, ν/cm^{-1}: 1723, 1690 (C=O). ^{1}H NMR, δ: 0.65 (t, 3 H, $CH_2C\underline{H}_3$, J = 7.1 Hz); 0.91—2.12 (m, 10 H, *cyclo*-C_6H_{11}); 3.19 (m, 1 H, *cyclo*-C_6H_{11}); 3.75 (q, 2 H, $C\underline{H}_2CH_3$, J = 7.1 Hz); 3.95 (s, 3 H, OMe); 5.65 (d, 2 H, C(4)H, C(6)H, J = 7.6 Hz); 6.94—7.91 (m, 15 H, Ar). Found (%): C, 76.79; H, 5.35; N, 4.18. $C_{44}H_{36}N_2O_6$. Calculated (%): C, 76.73; H, 5.27; N, 4.07.

Ethyl 1′-cyclohexyl-2,10,12-trioxo-5′-phenyl-5-(4-tolyl)-1′,2′,10,12-tetrahydro-5*H*-spiro{diindeno[1,2-*b*:2′,1′-*e*]pyridine-11,3′-pyrrole}-4′-carboxylate (5e). A solution of pyrroledione **1c** (1.0 mmol) and enamine **2b** (1.0 mmol) in anhydrous *m*-xylene (15 mL) was refluxed for 5 h, cooled, the solvent was evaporated to dryness. The residue was dissolved in boiling ethyl acetate (20 mL), cooled, a crimson precipitate formed was filtered off. The yield was 28%, m.p. 318—319 °C (ethyl acetate). IR, ν/cm^{-1}: 1734, 1694, 1676 (C=O). ^{1}H NMR, δ: 0.63 (t, 3 H, $CH_2C\underline{H}_3$, J = 7.0 Hz); 0.80—2.10 (m, 10 H, *cyclo*-C_6H_{11}); 2.57 (s, 3 H, Me); 3.18 (m, 1 H, *cyclo*-C_6H_{11}); 3.74 (q, 2 H, OCH_2, J = 7.0 Hz); 5.58 (d, 2 H, C(4)H, C(6)H, J = 7.6 Hz); 7.15—7.85

(m, 15 H, Ar). Found (%): C, 78.45; H, 5.32; N, 4.11. $C_{44}H_{36}N_2O_5$. Calculated (%): C, 78.55; H, 5.39; N, 4.16.

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