Synthesis of 11*H*-indolo[3,2-*c*]quinolines by $SnCl_4$-catalyzed cyclization of indole-3-carbaldehyde oximes*

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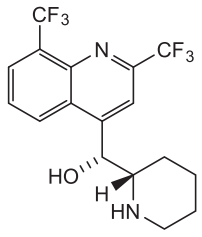
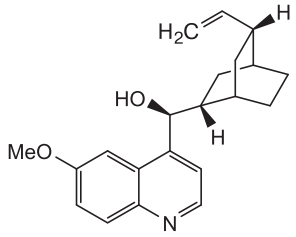
A new method for synthesizing 11*H*-indolo[3,2-*c*]quinolines by $SnCl_4$-catalyzed intramolecular electrophilic amination of 2-arylindole-3-carbaldehyde *O*-acetyl oximes was developed.

Key words: indoloquinolines, alkaloids, amination, quinoline.

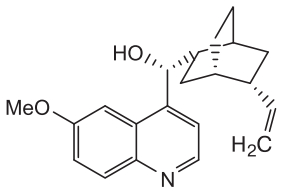
Quinoline is an important structural constituent of a number of natural and synthetic biologically active substances, *e.g.*, Mefloquine,[1] quinine,[2] and quinidine.[3] Despite the fact that several name reactions are suitable to construct this heterocyclic system (see, for instance, Refs 4—7), considerable efforts are been made to explore new synthetic methods tolerating a wide range of substrates and expanding the structural variety of the products.



Mefloquine



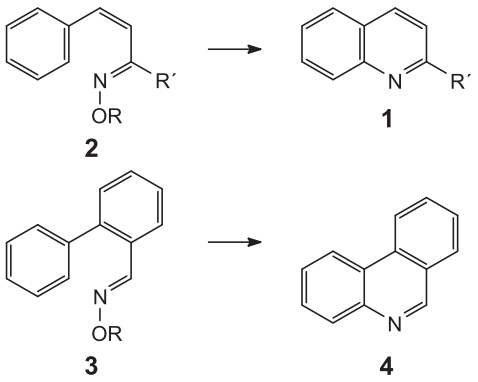
Quinine



Quinidine

Exploring such advantageous methods, we paid attention to the synthesis of quinolines **1** *via* intramolecular cyclization of oximes of unsaturated carbonyl compounds. The starting compounds in these reactions are often the derivatives of cinnamon aldehydes and 3-arylpropanones **2**.[8—13] The synthesis can also be started from 2-arylbenzaldehyde oximes **3** to be converted to phenantridines **4**.[14—19] This reaction could proceed under photochemical,[14—16] electrochemical,[17] microwave,[18] and thermal activations[19] (Scheme 1).

Scheme 1



R = H, Ar, Ac, Alk, EtCOO; R′ = H, Alk

The results described above were used to develop an interesting approach[20,21] to indolo[2,3-*c*]quinolines **6** involving base-mediated intramolecular cyclization of *O*-(2,4-dinitrophenyl)-3-arylindole-2-ketoximes **5** (Scheme 2). Deprotonation of indole increases the electron density at the *ortho* position of 3-aryl substituent thus accelerating the reaction. This is why the similar conditions are unsuitable for the synthesis of indolo[3,2-*c*]quinolines.

The indoloquinoline alkaloids exhibit a wide range of biological activities[22—24] that attracts our attention to

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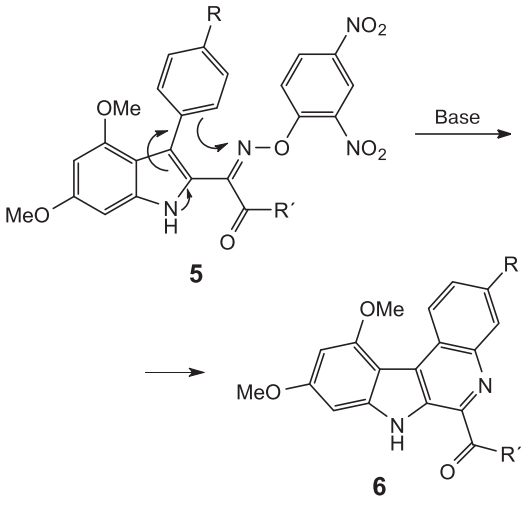
Scheme 2



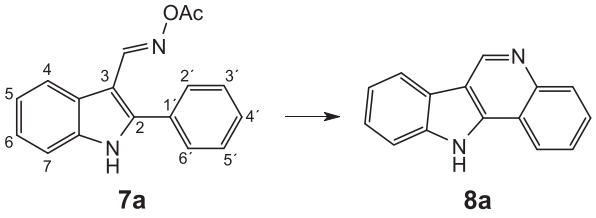
R = H, Me, Cl, Br, OMe; R′ = OEt, NMe_2

heterocyclic systems of this type.[25,26] We have recently developed an efficient *one-pot* synthetic approach to indolo[3,2-*c*]quinolines featuring the synthesis of indoles from 2-aminoacetophenones and aryl hydrazines under the Fischer conditions followed by treatment of the intermediate indoles with acylation reagents,[27] nitroalkanes,[28] and nitrostyrenes[29] in polyphosphoric acid (PPA). We also have recently reported the synthesis of indoloquinolines by the reaction of 2-nitroacetophenones with aryl hydrazines followed by *one-pot* reduction and acylation of the intermediate 2-(2-nitrophenyl)indoles in PPA.[30]

We hypothesized that in the presence of Lewis acids 2-arylindole-3-carbaldehyde oximes will readily undergo intramolecular electrophilic amination to give indolo[3,2-*c*]quinolines. Earlier,[14—21] low reactivity of unsubstituted oximes in intramolecular amination has been shown; therefore, the starting compounds of choice for this research were 2-aryl-1*H*-indole-3-carbaldehyde *O*-acetyl oximes **7a—f**. It should be underlined that Rodríguez and co-workers[31] have found that irradiation of oxime **7a** produces only 2-phenyl-1*H*-indole-3-carbonitrile but not the desired cyclization products. Therefore, we decided to study cyclization of oximes **7** under thermal activation conditions in the presence of different Lewis acids. The reaction conditions for the intramolecular cyclization of oximes **7** were optimized using *O*-acetyl oxime **7a** as a model compound (Scheme 3, Table 1).

The highest yield of the target product **8a** was achieved by refluxing oxime **7a** in toluene in the presence of tin tetrachloride for 7 h. As expected, *O*-unsubstituted oximes were unreactive under these conditions. To evaluate the

Scheme 3



Reagents and conditions: Lewis acid (3 equiv.), toluene, reflux, 7—12 h.

Table 1. Optimization of the reaction conditions for the synthesis of compound **8a**

Entry	Lewis acid	Solvent	*t*/h	Yield of **8a** (%)
1	$SnCl_4$	Toluene	8	46
2	$Fe(AcAc)_3$	EtOH	10	5
3	$ZnCl_2$	EtOH	10	15
4	$SnCl_2 \cdot 2H_2O$	Toluene	12	—[a]
5	$TiCl_4$	Toluene	7	—[b]

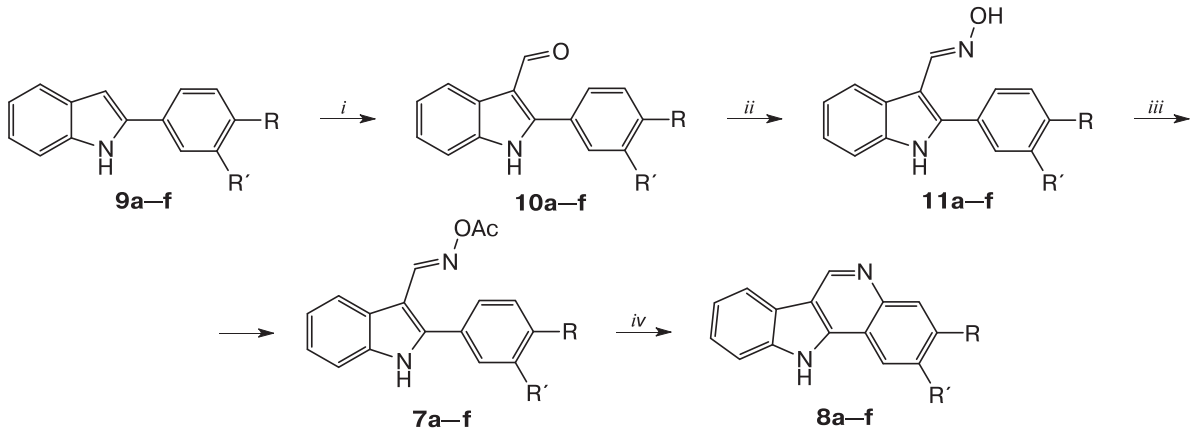
[a] No reaction occurs.
[b] Resinification.

synthetic scope of this reaction, 2-arylindoles **9a—f** were formylated[32] to give the corresponding 2-arylindole-3-carbaldehydes **10a—f**. Aldehydes **10a—f** were isolated pure by crystallization and quantitatively converted into the corresponding aldoximes **11a—f** and then to *O*-acetyl oximes **7a—f**. Without additional purification, *O*-acetyl oximes **7a—f** were subjected to heterocyclization to afford indolo[3,2-*c*]quinolines **8a—f** in 25—49% yields. It is of note that 2-β-naphthyl-1*H*-indole-3-carbaldehyde *O*-acetyl oxime (**7b**) gives exclusively 12*H*-benzo[*g*]-indolo[3,2-*c*]quinoline (**8b**) and no 13*H*-benzo[*h*]-indolo[3,2-*c*]quinoline was detected (Scheme 4).

Under similar conditions 3-acetyl-3-arylindoles **14** undergo the Beckmann rearrangement to compounds **15** (Scheme 5). Intermediate oximes **13a,b** and *O*-acetyl oximes **14** derived from them were used in the next steps without purification; the yields of 3-acetamino-2-arylindoles **15** were calculated based on the amounts of 3-acetyl-2-arylindoles **12** used.

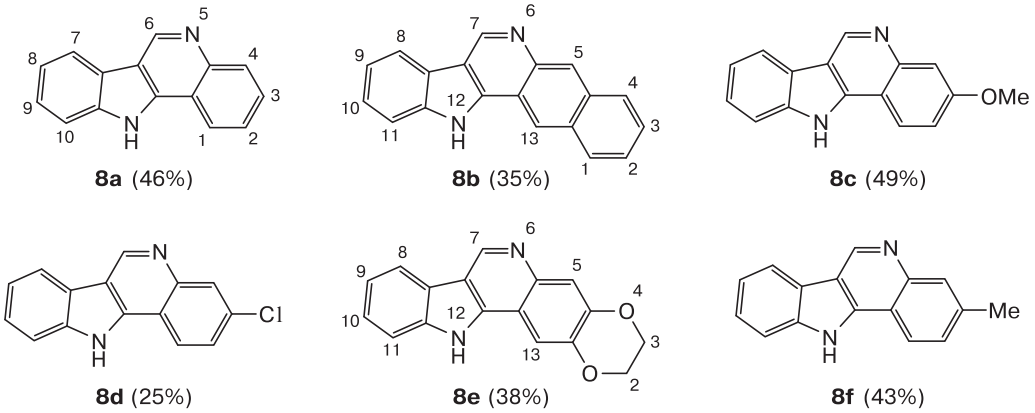
In summary, we elaborated the synthetic approach to 11*H*-indolo[3,2-*c*]quinolines involving electrophilic amination of 2-arylindole-3-carbaldehyde *O*-acetyl oximes. The developed procedure opens up the prospects for the construction of the hardly accessible indolo[3,2-*c*]quinolines including 12*H*-benzo[*g*]indolo[3,2-*c*]quinoline. The synthesized compounds can be transformed into the isocryptoleptine derivatives by treatment with iodomethane following the standard procedure.[33]

Scheme 4

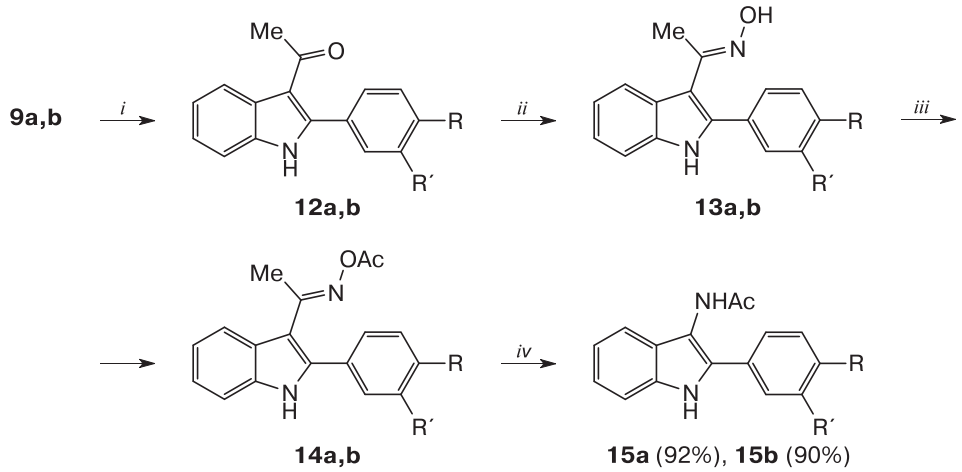


7—11: R = R′ = H (**a**), R + R′ = —CH=CH—CH=CH— (**b**), R = OMe, R′ = H (**c**), R = Cl, R′ = H (**d**), R + R′ = —OCH_2CH_2O— (**e**), R = Me, R′ = H (**f**)

Reagents and conditions: *i*. $POCl_3$, DMF, 30 min; *ii*. NH_2OH (3 equiv.), Et_3N (3 equiv.), EtOH, reflux, 2 h, then 20 °C, 2 h; *iii*. AcCl (1.3 equiv.), pyridine, 20 °C, 2 h; *iv*. $SnCl_4$ (3 equiv.), toluene, reflux, 7—12 h.



Scheme 5



7, 12—15: R = R′ = H (**a**), R + R′ = —CH=CH—CH=CH— (**b**)

Reagents and conditions: *i*. $SnCl_4$, CH_2Cl_2, 0 °C, 30 min, then Ac_2O, nitromethane, 20 °C, 3 h; *ii*. NH_2OH (3 equiv.), Et_3N (3 equiv.), EtOH, reflux, 2 h, then 20 °C, 2 h; *iii*. AcCl (1.3 equiv.), pyridine, 20 °C, 2 h; *iv*. $SnCl_4$, toluene.

Experimental

Lewis acids ($SnCl_4$ (98%), $Fe(AcAc)_3$ (99%), $TiCl_4$ (99%), $SnCl_2 \cdot 2H_2O$ (98%), and $ZnCl_2$ (97%)), $POCl_3$, 2- phenylindole, and nitromethane were purchased from Alfa Aesar. Commercially available 2-(2-naphthyl)indole, 2-(4-methoxyphenyl)-1*H*-indole, 2-(4-chlorophenyl)indole, and 2-(4-methylphenyl)-1*H*-indole were purchased from abcr. Gute Chimie (Germany). 2-(2,3-Dihydro[*b*][1,4]dioxin-6-yl)-1*H*-indole was synthesized as described earlier.[34] Zinc chloride was calcined prior to use in order to remove the water traces, other reagents were used as received.

^1H and ^{13}C NMR spectra were run on a Bruker AVANCE III HD instrument (working frequencies of 400.13 (^1H) and 100.61 MHz (^{13}C)) in DMSO-d_6. The chemical shifts are given in the δ scale relative to the residual solvent signals. High resolution electrospray ionization mass spectrometry was performed with a Bruker Maxis impact instrument using a direct inlet, HCO_2Na—HCO_2H was used for calibration. IR spectra were recorded with a Shimadzu IRAffinity-1S FTIR spectrophotometer equipped with an attenuated total reflection attachment. Purity of the compounds was monitored by TLC on the precoated Silufol UV-254 plates with visualization under UV-light. Melting points were measured with a Stuart SMP30 apparatus.

2-Arylindole-3-carbaldehydes 10a—f (general procedure). Compounds **10a—f** were synthesized by known procedure.[35] A solution of 2-aryl-1*H*-indole **9a—f** (0.01 mol) in a minimum amount of DMF was added to the Vilsmeier—Haack reagent (was prepared from phosphorus oxychloride (1 mL) and DMF (3.15 mL)) maintaining the reaction temperature between 10 and 20 °C. The reaction mixture was heated at 45 °C for 30 min and poured into a mixture of ice—water (100 mL) and 10% aqueous NaOH (20 mL). The resulting mixture was refluxed for 1 h and cooled to room temperature. The precipitate was collected by filtration, washed with water, dried, and recrystallized.

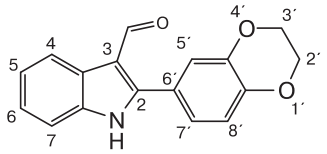
2-Phenyl-1*H*-indole-3-carbaldehyde (10a). White substance, yield 194 mg (88%), m.p. 236—240 °C (dioxane) (*cf.* Ref. 33: m.p. 249—250 °C). ^1H NMR (DMSO-d_6), δ: 7.41—7.21 (m, 2 H, H(5), H(6)); 7.52 (d, 1 H, H(7), J = 7.9 Hz); 7.67—7.56 (m, 3 H, H(3′), H(4′), H(5′)); 7.85—7.76 (m, 2 H, H(2′), H(6′)); 8.23 (d, 1 H, H(4), J = 7.5 Hz); 9.98 (s, 1 H, CHO); 12.43 (s, 1 H, NH). ^{13}C NMR (DMSO-d_6), δ: 112.5 (C(3)), 113.9 (C(7)), 121.5 (C(5)), 122.9 (C(6)), 124.2 (C(4)), 126.2 (C(2)), 129.5 (2 C, C(2′), C(6′)); 130.2 (C(3a)), 130.3 (C(4′)), 130.4 (2 C, C(3′), C(5′)); 136.4 (C(1′)), 149.6 (C(7a)), 186.0 (CHO). MS (ESI), *m/z*: 244.0731 $[M + Na]^+$. $C_{15}H_{11}NNaO$. Calculated: M = 244.0733.

2-(Naphthalen-2-yl)-1*H*-indole-3-carbaldehyde (10b). White substance, yield 228 mg (84%), m.p. 242—244 °C (ethanol). ^1H NMR (DMSO-d_6), δ: 7.16—7.39 (m, 2 H, H(5), H(6)); 7.55 (d, 1 H, H(7), J = 7.8 Hz); 7.71—7.59 (m, 2 H, H(6′), H(7′)); 7.91 (dd, 1 H, H(3′), J = 8.5 Hz, J = 1.7 Hz); 8.04 (dd, 1 H, H(4′), J = 5.8 Hz, J = 3.6 Hz); 8.19—8.09 (m, 2 H, H(5′), H(8′)); 8.26 (d, 1 H, H(6′), J = 7.3 Hz); 8.40 (s, 1 H, H(4)); 10.09 (s, 1 H, CHO); 12.60 (s, 1 H, NH). ^{13}C NMR (DMSO-d_6), δ: 112.1 (C(7)), 113.8 (C(3)), 121.2 (C(4)), 122.6 (C(5)), 123.9 (C(6)), 125.9 (C(2)), 126.9 (C(6′)), 127.1 (C(7′)), 127.3 (C(8a′)), 127.5 (C(1′)), 127.8 (C(2′)), 128.6 (2 C, C(4′), C(5′)); 129.8 (C(8′)), 132.7 (C(3a′)), 133.2 (C(2′)), 136.2 (C(4a′)), 149.0 (C(7a)), 185.9 (CHO). MS (ESI), *m/z*: 294.0884 $[M + Na]^+$. $C_{19}H_{13}NNaO$. Calculated: M = 294.0889. R_f 0.66 (EtOAc—hexane, 1 : 1).

2-(4-Methoxyphenyl)-1*H*-indole-3-carbaldehyde (10c). White substance, yield 221 mg (88%), m.p. 208—210 °C (ethanol) (*cf.* Ref. 33: m.p. 207—209 °C). ^1H NMR (DMSO-d_6), δ: 3.86 (s, 3 H, OMe); 7.16 (d, 2 H, H(3′), H(5′), J = 8.7 Hz); 7.20—7.30 (m, 2 H, H(5), H(6)); 7.48 (d, 1 H, H(7), J = 7.8 Hz); 7.73 (d, 2 H, H(2′), H(6′), J = 8.7 Hz); 8.19 (d, 1 H, H(4), J = 7.5 Hz); 9.95 (s, 1 H, CHO); 12.30 (s, 1 H, NH). ^{13}C NMR (DMSO-d_6), δ: 55.5 (OMe), 111.9 (C(7)), 113.1 (C(3)), 114.5 (2 C, C(3′), C(5′)); 121.0 (C(5)), 122.1 (C(2)), 122.4 (C(6)), 123.5 (C(4)), 125.9 (C(4a)), 131.3 (2 C, C(2′), C(6′)); 135.9 (C(1′)), 149.3 (C(7a)), 160.6 (C(4′)), 185.5 (CHO). MS (ESI), *m/z*: 274.0840 $[M + Na]^+$. $C_{16}H_{13}NNaO_2$. Calculated: M = 274.0838. R_f 0.54 (EtOAc—hexane, 1 : 4).

2-(4-Chlorophenyl)-1*H*-indole-3-carbaldehyde (10d). White substance, yield 219 mg (86%), m.p. 262—264 °C (acetone) (*cf.* Ref. 36: m.p. 263—265 °C). ^1H NMR (DMSO-d_6), δ: 7.15—7.38 (m, 2 H, H(5), H(6)); 7.52 (d, 1 H, H(7), J = 7.8 Hz); 7.66 (d, 2 H, H(3′), H(5′), J = 8.2 Hz); 7.81 (d, 2 H, H(2′), H(6′), J = 8.2 Hz); 8.22 (d, 1 H, H(4), J = 7.6 Hz); 9.97 (s, 1 H, CHO); 12.50 (s, 1 H, NH). ^{13}C NMR (DMSO-d_6), δ: 112.1 (C(7)), 113.7 (C(3)), 121.1 (C(5)), 122.6 (C(4)), 123.9 (C(6)), 125.8 (C(2)), 128.7 (C(3a)), 129.1 (2 C, C(2′), C(6′)); 131.6 (2 C, C(3′), C(5′)); 134.8 (C(1′)), 136.0 (C(4′)), 147.5 (C(7a)), 185.5 (C=O). MS (ESI), *m/z*: 278.0349 $[M + Na]^+$. $C_{15}H_{10}C1NNaO$. Calculated: M = 278.0343. R_f 0.71 (EtOAc—hexane, 1 : 4).

2-(2,3-Dihydro[*b*][1,4]dioxin-6-yl)-1*H*-indole-3-carbaldehyde (10e). White substance, yield 232 mg (83%), m.p. 251.3—253.3 °C (DMF). ^1H NMR (DMSO-d_6), δ: 4.32 (d, 4 H, OCH_2CH_2O, J = 13.6 Hz); 7.07 (d, 1 H, H(5′), J = 8.3 Hz); 7.32—7.17 (m, 4 H, H(5), H(6), H(7′), H(8′)); 7.47 (d, 1 H, H(7), J = 7.8 Hz); 8.17 (d, 1 H, H(4), J = 7.4 Hz); 9.95 (s, 1 H, CHO); 12.27 (s, 1 H, NH). ^{13}C NMR (DMSO-d_6), δ: 64.2 (OCH_2CH_2O), 64.4 (OCH_2CH_2O), 111.9 (C(7)), 113.2 (C(3)), 117.7 (C(5′)), 118.3 (C(8′)), 121.0 (C(7′)), 122.4 (C(5)), 122.8 (C(2)), 123.2 (C(4)), 123.6 (C(6)), 125.9 (C(3a)), 135.8 (C(7′)), 143.7 (C(7a)), 145.1 (C(4a′)), 148.8 (C(8a′)), 185.4 (CHO). MS (ESI), *m/z*: 302.0790 $[M + Na]^+$. $C_{17}H_{13}NNaO_3$. Calculated: M = 302.0788. R_f 0.51 (EtOAc—hexane, 1 : 1).



10e

2-(*p*-Tolyl)-1*H*-indole-3-carbaldehyde (10f). White substance, yield 209 mg (89%), m.p. 232—235 °C (ethanol) (*cf.* Ref. 33: m.p. 239—241 °C). ^1H NMR (DMSO-d_6), δ: 2.41 (s, 3 H, Me); 7.19—7.31 (m, 2 H, H(5), H(6)); 7.41 (d, 2 H, H(3′), H(5′), J = 7.9 Hz); 7.50 (d, 1 H, H(7), J = 7.8 Hz); 7.67 (d, 2 H, H(2′), H(6′), J = 7.9 Hz); 8.20 (d, 1 H, H(4), J = 7.5 Hz); 9.96 (s, 1 H, CHO); 12.39 (s, 1 H, NH). ^{13}C NMR (DMSO-d_6), δ: 21.0 (Me), 112.0 (C(7)), 113.3 (C(3)), 121.0 (C(5)), 122.4 (C(4)), 123.7 (C(6)), 125.9 (C(3)), 127.0 (C(3a)), 129.6 (2 C, C(2′), C(6′)); 129.8 (2 C, C(3′), C(5′)), 135.9 (C(4′)), 139.7 (C(1′)), 149.3 (C(7a)), 185.5 (CHO). MS (ESI), *m/z*: 258.0885 $[M + Na]^+$. $C_{16}H_{13}NNaO$. Calculated: M = 258.0889. R_f 0.65 (EtOAc—hexane, 1 : 4).

3-Acetyl-2-arylindoles 12a,b (general procedure). Compounds **12a,b** were synthesized by the earlier described procedure.[37] To

a stirred solution of indole **9** (10.0 mmol) in CH_2Cl_2 (20 mL), $SnCl_4$ (1.44 mL, 12.0 mmol) was added in one portion at 0 °C. Cooling was removed and the mixture was stirred at room temperature for 30 min. To the resulting suspension, acetic anhydride (2.1 mL, 10.0 mmol) and nitromethane (15 mL) were added by small portions. After 3 h stirring at room temperature, the mixture was treated with ice—water (30 mL), filtered to remove the inorganic impurities, and extracted with ethyl acetate. The organic layer was dried with $MgSO_4$ and concentrated *in vacuo*. The products were purified by either recrystallization or column chromatography (elution with EtOAc—petroleum ether, 1 : 4).

2-Acetyl-3-phenyl-1*H*-indole (12a). Colorless crystals, yield 1 g (43%), m.p. 232—235 °C (ethanol) (*cf.* Ref. 38: m.p. 171 °C). ^{1}H NMR (DMSO-d_6), δ: 2.07 (s, 3 H, Me); 7.27—7.17 (m, 2 H, H(5), H(6)); 7.43 (d, 1 H, H(7), J = 7.3 Hz); 7.52—7.61 (m, 3 H, H(3′), H(4′), H(5′)); 7.61—7.68 (m, 2 H, H(2′), H(6′)); 8.21 (d, 1 H, H(4), J = 7.4 Hz); 12.10 (s, 1 H, NH). ^{13}C NMR (DMSO-d_6), δ: 30.1 (Me), 111.6 (C(7)), 114.3 (C(3)), 121.6 (C(5)), 121.8 (C(6)), 122.9 (C(4)), 127.0 (C(2)), 128.4 (2 C, C(2′), C(6′)); 129.4 (C(4′)), 130.0 (2 C, C(3′), C(5′)); 132.7 (C(3a)), 135.4 (C(1′)), 144.9 (C(7a)), 193.6 (C=O). MS (ESI), m/z: 258.0884 $[M + Na]^+$. $C_{16}H_{13}N_1NaO$. Calculated: M = = 258.0889. R_f 0.31 (EtOAc—hexane, 1 : 4).

3-Acetyl-2-(naphthalen-2-yl)-1*H*-indole (12b). Colorless crystals, yield 1.2 g (42%), m.p. 185.3—187.1 °C (EtOAc). ^{1}H NMR (DMSO-d_6), δ: 2.12 (s, 3 H, Me); 7.18—7.30 (m, 2 H, H(5), H(6)); 7.46 (d, 1 H, H(7), J = 7.7 Hz); 7.59—7.69 (m, 2 H, H(6′), H(7′)); 7.76 (dd, 1 H, H(3′), J = 8.5 Hz, J = 1.4 Hz); 8.13—8.00 (m, 3 H, H(4′), H(5′), H(8′)); 8.22 (d, 1 H, H(4), J = 7.4 Hz); 8.24 (s, 1 H, H(1′)); 12.22 (s, 1 H, NH). ^{13}C NMR (DMSO-d_6), δ: 30.3 (Me), 111.7 (C(7)), 114.5 (C(3)), 121.6 (C(5)), 121.9 (C(4)), 123.0 (C(6)), 126.9 (C(1′)), 127.1 (C(8a)), 127.2 (C(6′)), 127.6 (C(7′)), 127.8 (C(4′)), 127.9 (C(3′)), 128.3 (C(5′)), 129.4 (C(8′)), 130.1 (C(2)), 132.5 (C(3a)), 133.0 (C(2′)), 135.6 (C(4a′)), 144.8 (C(7a)), 193.7 (C=O). MS (ESI), m/z: 308.1052 $[M + Na]^+$. $C_{20}H_{15}NNaO$. Calculated: M = = 308.1046. R_f 0.31 (EtOAc—hexane, 1 : 4).

2-Arylindole-3-carbaldehyde oximes 11a—f and 3-acetyl-2-arylindole oximes 13a,b (general procedure). Compounds **11a—f** and **13a,b** were synthesized by the earlier described modified procedure.[39] A 5-mL round bottom flask was charged with 2-aryl-1*H*-indole-3-carbaldehyde **10a—f** or **12a,b** (1 mmol), hydroxylamine hydrochloride (0.207 g, 3 mmol), triethylamine (0.3 mL, 3 mmol), and EOH (2 mL). The mixture was refluxed for 1—2 h (TLC monitoring). After reaction completion, the solvent was removed *in vacuo*, the residue was dissolved in ethyl acetate (20 mL), and washed with water (3×15 mL). The combined organic layers were dried and concentrated *in vacuo*. The products were obtained in quantitative yields. Compounds **11a—f** and **13a,b** were used in the next step without purification; if necessary, they can be purified by silica gel column chromatography (gradient elution with ethyl acetate—hexane, 1 : 8→1 : 4).

2-Phenyl-1*H*-indole-3-carbaldehyde oxime (11a). White substance, m.p. 179.2—183.1 °C. ^{1}H NMR (DMSO-d_6), δ: 7.13 (t, 1 H, H(5), J = 7.5 Hz); 7.21 (t, 1 H, H(6), J = 7.5 Hz); 7.43 (d, 1 H, H(7), J = 8.0 Hz); 7.48 (t, 1 H, H(4′), J = 6.9 Hz); 7.54—7.64 (m, 4 H, H(2′), H(3′), H(5′), H(6′)); 8.08 (d, 1 H, H(4), J = 7.9 Hz); 8.26 (s, 1 H, CH=N); 10.72 (s, 1 H, NH); 11.76 (s, 1 H, OH). ^{13}C NMR (DMSO-d_6), δ: 105.8 (C(3)), 111.5 (C(7)), 120.5 (C(5)), 121.9 (C(6)), 122.8 (C(4)), 125.5 (C(2)), 128.6 (C(4′)), 129.0 (4 C, C(2′), C(3′), C(5′), C(6′)); 131.3 (C(3a)), 136.0 (C(1′)), 139.7 (C(7a)), 144.3 (CH=N). MS (ESI), m/z: 237.1026 $[M + H]^+$. $C_{15}H_{13}N_2O$. Calculated: M = 237.1022. R_f 0.22 (EtOAc—hexane, 1 : 4).

2-(Naphthalen-2-yl)-1*H*-indole-3-carbaldehyde oxime (11b). White substance, m.p. 182.8—187.2 °C. ^{1}H NMR (DMSO-d_6), δ: 7.15 (ddd, 1 H, H(5), J = 7.9 Hz, J = 7.0 Hz, J = 0.8 Hz); 7.23 (ddd, 1 H, H(6), J = 8.1 Hz, J = 7.0 Hz, J = 1.1 Hz); 7.46 (d, 1 H, H(7), J = 8.0 Hz); 7.64—7.57 (m, 2 H, H(6′), H(7′)); 7.75 (dd, 1 H, H(4′), J = 8.5 Hz, J = 1.7 Hz); 8.03—7.98 (m, 1 H, H(3′)); 8.14—8.05 (m, 3 H, H(4), H(5′), H(8′)); 8.16 (s, 1 H, H(1′)); 8.37 (s, 1 H, CH=N); 10.75 (s, 1 H, NH); 11.89 (s, 1 H, OH). ^{13}C NMR (DMSO-d_6), δ: 106.3 (C(7)), 111.6 (C(3)), 120.6 (C(5)), 122.0 (C(4)), 122.9 (C(6)), 125.6 (C(2)), 126.6 (C(7′)), 126.9 (C(6′)), 126.9 (C(1′)), 127.8 (C(4′)), 128.1 (C(3′)), 128.3 (C(5′)), 128.5 (C(8′)), 128.9 (C(8a′)), 132.6 (C(3a)), 132.9 (C(2′)), 136.6 (C(4a′)), 139.6 (C(7a)), 144.4 (C=N). MS (ESI), m/z: 387.1181 $[M + H]^+$. $C_{19}H_{15}N_2O$. Calculated: M = 387.1179. R_f 0.23 (EtOAc—hexane, 1 : 4).

2-(4-Methoxyphenyl)-1*H*-indole-3-carbaldehyde oxime (11c). White substance, m.p. 203.0—206 °C. ^{1}H NMR (DMSO-d_6), δ: 3.84 (s, 3 H, OMe); 7.15—7.08 (m, 3 H, H(5), H(3′), H(5′)); 7.18 (ddd, 1 H, H(6), J = 8.2 Hz, J = 7.1 Hz, J = 1.2 Hz); 7.40 (d, 1 H, H(7), J = 8.0 Hz); 7.57—7.50 (m, 2 H, H(2′), H(6′)); 8.05 (d, 1 H, H(4), J = 7.8 Hz); 8.24 (s, 1 H, CH=N); 10.65 (s, 1 H, NH); 11.65 (s, 1 H, OH). ^{13}C NMR (DMSO-d_6), δ: 55.3 (OMe), 105.2 (C(3)), 111.4 (C(7)), 114.5 (2 C, C(3′), C(5′)); 120.4 (C(5)), 121.7 (C(4)), 122.5 (C(6)), 123.7 (C(2)), 125.6 (C(1′)), 130.3 (2 C, C(2′), C(6′)), 136.2 (C(3a)), 139.8 (C(7a)), 144.5 (CH=N), 159.6 (C(4′)). MS (ESI), m/z: 267.1123 $[M + H]^+$. $C_{16}H_{15}N_2O_2$. Calculated: M = 267.1128. R_f 0.28 (EtOAc—hexane, 1 : 4).

2-(4-Chlorophenyl)-1*H*-indole-3-carbaldehyde oxime (11d). White substance, m.p. 239.5—256 °C. ^{1}H NMR (DMSO-d_6), δ: 7.13 (ddd, 1 H, H(5), J = 7.9 Hz, J = 7.0 Hz, J = 0.8 Hz); 7.22 (ddd, 1 H, H(6), J = 8.2 Hz, J = 7.0 Hz, J = 1.1 Hz); 7.43 (d, 1 H, H(7), J = 8.0 Hz); 7.58—7.67 (m, 4 H, H(2′), H(3′), H(5′), H(6′)); 8.08 (d, 1 H, H(4), J = 7.9 Hz); 8.25 (s, 1 H, CH=N); 10.78 (s, 1 H, NH); 11.81 (s, 1 H, OH). ^{1}H NMR (DMSO-d_6), δ: 106.3 (C(3)), 111.6 (C(7)), 120.6 (C(5)), 122.0 (C(6)), 123.0 (C(4)), 125.5 (C(3)), 129.0 (2 C, C(2′), C(6′)); 130.2 (C(3a)), 130.6 (2 C, C(3′), C(5′)); 133.3 (C(1′)), 136.4 (C(4′)), 138.2 (C(7a)), 144.0 (CH=N). MS (ESI), m/z: 271.0635 $[M + H]^+$. $C_{15}H_{12}ClN_2O$. Calculated: M = 271.0633. R_f 0.29 (EtOAc—hexane, 1 : 4).

2-(2,3-Dihydrobenzo[*b*][1,4]dioxin-6-yl)-1*H*-indole-3-carbaldehyde oxime (11e). White substance, m.p. 157.0—165.1 °C (DMF). ^{1}H NMR (DMSO-d_6), δ: 4.32 (s, 4 H, OCH_2CH_2O); 7.05—7.07 (m, 2 H, H(5), H(8′)); 7.08 (s, 1 H, H(5′)); 7.10—7.13 (m, 1 H, H(7′)); 7.18 (ddd, 1 H, H(6), J = 8.2 Hz, J = 7.1 Hz, J = 1.2 Hz); 7.39 (d, 1 H, H(7), J = 8.0), 8.04 (d, 1 H, H(4), J = 7.9); 8.24 (s, 1 H, CH=N); 10.67 (s, 1 H, NH); 11.63 (s, 1 H, OH). ^{13}C NMR (DMSO-d_6), δ: 64.2 (OCH_2CH_2O), 64.3 (OCH_2CH_2O), 105.3 (C(3)), 111.4 (C(7)), 117.3 (C(5′)), 117.6 (C(8′)), 120.4 (C(7′)), 121.7 (C(5)), 122.1 (C(4)), 122.6 (C(6)), 124.4 (C(6′)), 125.6 (C(2)), 136.2 (C(3a)), 139.4 (C(7a)), 143.6 (C(8a′)), 144.0 (C(4a′)), 144.4 (CH=N). MS (ESI), m/z: 295.1078 $[M + Na]^+$. $C_{17}H_{15}N_2O_3$. Calculated: M = 295.1077. R_f 0.2 (EtOAc—hexane, 1 : 4).

2-(*p*-Tolyl)-1*H*-indole-3-carbaldehyde oxime (11f). White substance, m.p. 203.3—205 °C. ^{1}H NMR (DMSO-d_6), δ: 2.39

(s, 3 H, Me); 7.12 (ddd, 1 H, H(5), J = 7.7 Hz, J = 7.2 Hz, J = 0.7 Hz); 7.20 (ddd, 1 H, H(6), J = 8.0 Hz, J = 7.3 Hz, J = 0.9 Hz); 7.37 (d, 2 H, H(3′), H(5′), J = 8.0 Hz); 7.42 (d, 1 H, H(7), J = 8.0 Hz); 7.49 (d, 2 H, H(2′), H(6′), J = 8.0 Hz); 8.07 (d, 1 H, H(4), J = 7.9 Hz); 8.26 (s, 1 H, CH=N); 10.69 (s, 1 H, NH); 11.70 (s, 1 H, OH). ^{13}C NMR (DMSO-d_6), δ: 20.9 (Me), 105.5 (C(3)), 111.5 (C(7)), 120.4 (C(5)), 121.8 (C(4)), 122.6 (C(6)), 125.6 (C(2)), 128.5 (C(3a)), 128.8 (2 C, C(2′), C(6′)); 129.5 (2 C, C(3′), C(5′)); 136.3 (C(1′)), 138.2 (C(4′)), 139.8 (C(7a)), 144.4 (CH=N). MS (ESI), *m/z*: 251.1178 $[M + H]^+$. $C_{16}H_{15}N_2O$. Calculated: M = 251.1179. R_f 0.34 (EtOAc—hexane, 1 : 4).

1-(2-Phenyl-1*H*-indol-3-yl)ethan-1-one oxime (13a). White substance, m.p. 159.6—162.9 °C (*cf.* Ref. 40: m.p. 177—180 °C). ^{1}H NMR (DMSO-d_6), δ: 1.95 (s, 3 H, Me); 7.06 (t, 1 H, H(5), J = 7.6 Hz); 7.15 (ddd, 1 H, H(6), J = 8.1 Hz, J = 6.9 Hz, J = 0.9 Hz); 7.44—7.37 (m, 2 H, H(7), H(4′)); 7.53—7.47 (m, 2 H, H(3′), H(5′)); 7.61—7.57 (m, 2 H, H(2′), H(6′)); 7.73 (d, 1 H, H(4), J = 7.9 Hz); 10.89 (s, 1 H, NH); 11.56 (s, 1 H, OH). ^{13}C NMR (DMSO-d_6), δ: 16.0 (Me), 110.7 (C(3)), 111.4 (C(7)), 119.8 (C(5)), 120.2 (C(4)), 122.0 (C(6)), 127.3 (C(2)), 128.1 (C(4′)), 128.5 (2 C, C(2′), C(6′)); 128.6 (2 C, C(3′), C(5′)); 132.7 (C(3a)), 135.9 (C(1′)), 136.3 (C(7a)), 150.6 (C=N). MS (ESI), *m/z*: 251.1175 $[M + H]^+$. $C_{16}H_{15}N_2O$. Calculated: M = 251.1179. R_f 0.22 (EtOAc—hexane, 1 : 4).

1-(2-(Naphthalen-2-yl)-1*H*-indol-3-yl)ethan-1-one oxime (13b). White substance, m.p. 117.7—126.2 °C. ^{1}H NMR (DMSO-d_6), δ: 1.99 (s, 3 H, Me); 7.11—7.02 (m, 1 H, H(5)); 7.21—7.13 (m, 1 H, H(6)); 7.44 (d, 1 H, H(7), J = 7.5 Hz); 7.60—7.51 (m, 2 H, H(6′), H(7′)); 7.70 (d, 1 H, H(3′), J = 8.0 Hz); 7.77 (d, 1 H, H(4′), J = 7.7 Hz); 8.08—7.91 (m, 3 H, H(4), H(5′), H(8′)); 8.14 (s, 1 H, H(1′)); 10.92 (s, 1 H, NH); 11.70 (s, 1 H, OH). ^{13}C NMR (DMSO-d_6), δ: 16.1 (Me), 111.2 (C(3)), 111.4 (C(7)), 119.9 (C(5)), 120.3 (C(4)), 122.2 (C(6)), 126.58 (C(5′)), 126.61 (C(6′)), 126.8 (C(3′)), 127.2 (C(1′)), 127.4 (C(2′)), 127.7 (C(4′)), 128.0 (C(8′)), 128.1 (C(5′)), 130.2 (C(3a)), 132.4 (C(8a′)), 132.9 (C(4a′)), 136.1 (C(2′)), 136.3 (C(7a)), 150.7 (C=N). MS (ESI), *m/z*: 301.1339 $[M + H]^+$. $C_{20}H_{17}N_2O$. Calculated: M = 301.1335. R_f 0.23 (EtOAc—hexane, 1 : 4).

2-Aryl-1*H*-indole-3-carbaldehyde *O*-acetyl oximes 7a—f and 3-acetyl-2-arylindole *O*-acetyl oximes 14a,b (general procedure). Compounds **7a—f** and **14a,b** were synthesized as earlier described.[31] To a solution of oxime **11a—f** or **13a,b** (1 mmol) in pyridine (1.2 mL), acetyl chloride (101 mg, 1.3 mmol) was added dropwise and the mixture was stirred at room temperature for 2 h. The mixture was diluted with ethyl acetate (50 mL) and washed successively by 10% HCl (3×10 mL) and 5% aqueous $NaHCO_3$ (3×10 mL). The organic layer was dried with Na_2SO_4, the drying agent was filtered off. Removal of the solvent *in vacuo* afforded the title products in quantitative yields. Compounds **7a—f** and **14a,b** were used in the next step without purification; if necessary, they can be purified by silica gel column chromatography (gradient elution with ethyl acetate—hexane, 1 : 8→1 : 4).

2-Phenyl-1*H*-indole-3-carbaldehyde *O*-acetyl oxime (7a). White substance, m.p. 154.6—159.4 °C. ^{1}H NMR (DMSO-d_6), δ: 2.19 (s, 3 H, Me); 7.23 (ddd, 1 H, H(6), J = 7.9 Hz, J = 7.2 Hz, J = 0.9 Hz); 7.28 (ddd, 1 H, H(5), J = 8.1 Hz, J = 7.2 Hz, J = 1.2 Hz); 7.49 (d, 1 H, H(7), J = 8.1 Hz); 7.68—7.52 (m, 5 H, H(2′), H(3′), H(4′), H(5′), H(6′)); 8.14 (d, 1 H, H(4), J = 7.9 Hz); 8.55 (s, 1 H, CH=N); 12.20 (s, 1 H, NH). ^{13}C NMR (DMSO-d_6), δ: 19.7 (Me), 103.2 (C(3)), 111.9 (C(7)), 121.5 (C(5)), 122.1 (C(6)), 123.5 (C(4)), 125.2 (C(2)), 129.2 (2 C, C(2′), C(6′)); 129.3 (2 C, C(3′), C(5′)); 129.4 (C(4′)), 130.4 (C(3a)), 136.4 (C(1′)), 144.1 (C(7a)), 151.7 (CH=N), 168.7 (C=O). MS (ESI), *m/z*: 301.0953 $[M + Na]^+$. $C_{17}H_{14}N_2NaO_2$. Calculated: M = 301.0947. R_f 0.29 (EtOAc—hexane, 1 : 4).

2-(Naphthalen-2-yl)-1*H*-indole-3-carbaldehyde *O*-acetyl oxime (7b). White substance, m.p. 130.0—136.1 °C. ^{1}H NMR (DMSO-d_6), δ: 2.20 (s, 3 H, Me); 7.25 (t, 1 H, H(5), J = 7.4 Hz); 7.31 (t, 1 H, H(6), J = 7.7 Hz); 7.53 (d, 1 H, H(7), J = 7.9 Hz); 7.67—7.60 (m, 3 H, H(4′), H(6′), H(7′)); 7.79 (dd, 1 H, H(3′), J = 8.5 Hz, J = 1.6 Hz); 8.05—8.01 (m, 1 H, H(8′)); 8.15—8.08 (m, 2 H, H(1′), H(5′)); 8.18 (d, 1 H, H(4), J = 8.0 Hz); 8.66 (s, 1 H, CH=N); 12.33 (s, 1 H, NH). ^{13}C NMR (DMSO-d_6), δ: 19.7 (Me), 103.6 (C(3)), 111.9 (C(7)), 121.5 (C(5)), 122.2 (C(4)), 123.6 (C(6)), 125.3 (C(2)), 126.5 (C(6′)), 127.0 (C(7′)), 127.2 (C(1′)), 127.8 (C(4′)), 127.8 (C(3a)), 128.5 (C(3′)), 128.7 (C(5′)), 128.8 (C(8′)), 132.8 (C(8a′)), 132.9 (C(2′)), 136.6 (C(4a′)), 144.0 (C(7a)), 151.9 (CH=N), 168.8 (C=O). MS (ESI), *m/z*: 351.1110 $[M + Na]^+$. $C_{21}H_{16}N_2NaO_2$. Calculated: M = 351.1104. R_f 0.22 (EtOAc—hexane, 1 : 4).

2-(4-Methoxyphenyl)-1*H*-indole-3-carbaldehyde *O*-acetyl oxime (7c). White substance, m.p. 150.1—154 °C. ^{1}H NMR (DMSO-d_6), δ: 2.19 (s, 3 H, Me); 3.85 (s, 3 H, OMe); 7.18—7.13 (m, 2 H, H(3′), H(5′)); 7.23—7.18 (m, 1 H, H(5)); 7.23—7.29 (m, 1 H, H(6)); 7.47 (d, 1 H, H(7), J = 7.8 Hz); 7.55—7.63 (m, 2 H, H(2′), H(6′)); 8.11 (d, 1 H, H(4), J = 7.6 Hz); 8.52 (s, 1 H, CH=N); 12.09 (s, 1 H, NH). ^{13}C NMR (DMSO-d_6), δ: 19.7 (Me), 55.4 (OMe), 102.5 (C(3)), 111.7 (C(7)), 114.6 (2 C, C(3′), C(5′)); 121.4 (C(5)), 121.9 (C(4)), 122.6 (C(2)), 123.2 (C(6)), 125.2 (C(3a)), 130.6 (2 C, C(2′), C(6′)); 136.3 (C(1′)), 144.3 (C(7a)), 151.9 (CH=N), 160.2 (C(4′)), 168.7 (C=O). MS (ESI), *m/z*: 331.1053 $[M + Na]^+$. $C_{18}H_{16}N_2NaO_3$. Calculated: M = 331.1053. R_f 0.12 (EtOAc—hexane, 1 : 4).

2-(4-Chlorophenyl)-1*H*-indole-3-carbaldehyde *O*-acetyl oxime (7d). White substance, m.p. 195.7—205 °C. ^{1}H NMR (DMSO-d_6), δ: 2.20 (s, 3 H, Me); 7.24 (ddd, 1 H, H(5), J = 8.0 Hz, J = 7.1 Hz, J = 1.0 Hz); 7.30 (ddd, 1 H, H(6), J = 7.9 Hz, J = 7.2 Hz, J = 1.2 Hz); 7.50 (d, 1 H, H(7), J = 7.9 Hz); 7.67—7.70 (m, 4 H, H(2′), H(3′), H(5′), H(6′)); 8.15 (d, 1 H, H(4), J = 7.7); 8.56 (s, 1 H, CH=N); 12.25 (s, 1 H, NH). ^{13}C NMR (DMSO-d_6), δ: 19.7 (Me), 103.6 (C(3)), 112.0 (C(7)), 121.6 (C(5)), 122.2 (C(4)), 123.6 (C(6)), 125.1 (C(2)), 129.18 (2 C, C(2′), C(6′)); 129.2 (C(3a)), 131.0 (2 C, C(3′), C(5′)); 134.2 (C(1′)), 136.5 (C(4′)), 142.6 (C(7a)), 151.6 (CH=N), 168.7 (C=O). MS (ESI), *m/z*: 335.0553 $[M + Na]^+$. $C_{17}H_{13}Cl_1N_2NaO_2$. Calculated: M = 335.0558. R_f 0.33 (EtOAc—hexane, 1 : 4).

2-(2,3-Dihydrobenzo[*b*][1,4]dioxin-6-yl)-1*H*-indole-3-carbaldehyde *O*-acetyl oxime (7e). White amorphous substance. ^{1}H NMR (DMSO-d_6), δ: 2.19 (s, 3 H, Me); 4.33 (s, 4 H, OCH_2-CH_2O); 7.07 (d, 1 H, H(8′), J = 8.3 Hz); 7.11 (d, 1 H, H(7′), J = 8.4 Hz); 7.15 (d, 1 H, H(5′), J = 1.7 Hz); 7.20 (t, 1 H, H(5), J = 7.4 Hz); 7.26 (t, 1 H, H(6), J = 7.2 Hz); 7.45 (d, 1 H, H(7), J = 7.9 Hz); 8.10 (d, 1 H, H(4), J = 7.7 Hz); 8.53 (s, 1 H, CH=N); 12.07 (s, 1 H, NH). ^{13}C NMR (DMSO-d_6), δ: 19.7 (Me), 64.2 (OCH_2CH_2O), 64.3 (OCH_2CH_2O), 102.7 (C(3)), 111.8 (C(7)), 117.7 (C(5′)), 117.8 (C(8′)), 121.4 (C(5)), 122.0 (C(7′)), 122.4 (C(4)), 123.3 (C(6)), 123.4 (C(2)), 125.2 (C(6′)), 136.3 (C(3a)), 143.7 (C(7a)), 143.9 (C(8a′)), 144.6 (C(4a′)), 151.8 (CH=N), 168.7 (C=O). MS (ESI), *m/z*: 359.0996 $[M + Na]^+$. $C_{19}H_{16}N_2NaO_4$. Calculated: M = 359.1002. R_f 0.15 (EtOAc—hexane, 1 : 4).

2-(*p*-Tolyl)-1*H*-indole-3-carbaldehyde *O*-acetyl oxime (7f). White substance, m.p. 165.1—168.8 °C. ^{1}H NMR (DMSO-d_6), δ: 2.19 (s, 3 H, COMe); 2.40 (s, 3 H, Me); 7.24—7.19 (m, 1 H, H(5)); 7.31—7.24 (m, 1 H, H(6)); 7.40 (d, 2 H, H(2′), H(6′), *J* = 8.0 Hz); 7.48 (d, 1 H, H(7), *J* = 7.9 Hz); 7.54 (d, 2 H, H(3′), H(5′), *J* = 8.1 Hz); 8.12 (d, 1 H, H(4), *J* = 7.7 Hz); 8.53 (s, 1 H, CH=N); 12.14 (s, 1 H, NH). ^{13}C NMR (DMSO-d_6), δ: 19.7 (COMe), 20.9 (Me), 102.9 (C(3)), 111.8 (C(7)), 121.4 (C(5)), 122.0 (C(4)), 123.3 (C(6)), 125.2 (C(2)), 127.5 (C(3a)), 129.1 (2 C, C(2′), C(6′)); 129.7 (2 C, C(3′), C(5′)); 136.4 (C(1′)), 139.1 (C(4′)), 144.3 (C(7a)), 151.8 (CH=N), 168.7 (C=O). MS (ESI), *m/z*: 315.1099 $[M + Na]^+$. $C_{18}H_{16}N_2NaO_2$. Calculated: M = 315.1104. R_f 0.37 (EtOAc—hexane, 1 : 4).

1-(2-Phenyl-1*H*-indol-3-yl)ethan-1-one *O*-acetyl oxime (14a). White substance, m.p. 116.1—120.6 °C. ^{1}H NMR (DMSO-d_6), δ: 2.05 (s, 3 H, Me); 2.21 (s, 3 H, COMe); 7.13 (ddd, 1 H, H(5), *J* = 8.0 Hz, *J* = 6.9 Hz, *J* = 0.8 Hz); 7.21 (ddd, 1 H, H(6), *J* = 7.9 Hz, *J* = 6.9 Hz, *J* = 0.8 Hz); 7.44 (d, 1 H, H(4), *J* = 8.0 Hz); 7.47—7.56 (m, 3 H, H(3′), H(4′), H(5′)); 7.58—7.63 (m, 2 H, H(2′), H(6′)); 7.99 (d, 1 H, H(7), *J* = 7.9 Hz); 11.89 (s, 1 H, NH). ^{13}C NMR (DMSO-d_6), δ: 17.3 ($\underline{C}H_3CO$), 19.8 ($\underline{C}H_3C$=N), 107.9 (C(3)), 111.5 (C(7)), 120.6 (C(5)), 120.9 (C(6)), 122.6 (C(4)), 126.5 (C(2)), 128.7 (2 C, C(2′), C(6′)); 128.8 (C(4′)), 129.1 (2 C, C(3′), C(5′)); 132.1 (C(3a)), 135.8 (C(1′)), 139.7 (C(7a)), 160.4 (C=N), 168.6 (C=O). MS (ESI), *m/z*: 315.1109 $[M + Na]^+$. $C_{18}H_{16}N_2NaO_2$. Calculated: M = 315.1104. R_f 0.32 (EtOAc—hexane, 1 : 4).

1-(2-(Naphthalen-2-yl)-1*H*-indol-3-yl)ethan-1-one *O*-acetyl oxime (14b). Yellow liquid. ^{1}H NMR (DMSO-d_6), δ: 1.80 (s, 3 H, Me); 2.28 (s, 3 H, COMe); 7.21 (t, 1 H, H(5), *J* = 7.2 Hz); 7.30 (t, 1 H, H(6), *J* = 7.1 Hz); 7.45—7.59 (m, 4 H, H(7), H(4′), H(6′), H(7′)); 7.64 (dd, 1 H, H(3′), *J* = 8.5 Hz, *J* = 1.5 Hz); 7.85—7.95 (m, 3 H, H(4), H(5′), H(8′)); 8.63 (s, 1 H, H(1′)); 8.00 (s, 1 H, NH). ^{13}C NMR (DMSO-d_6), δ: 19.6 ($\underline{C}H_3CO$), 21.7 ($\underline{C}H_3C$=N), 108.3 (C(3)), 111.4 (C(7)), 120.7 (C(5)), 121.3 (C(4)), 123.3 (C(6)), 125.2 (C(6′)), 126.7 (C(7′)), 127.0 (C(2)), 127.1 (C(1′)), 127.2 (C(4′)), 128.0 (C(3′)), 128.4 (C(5′)), 129.2 (C(8′)), 129.4 (C(8a′)), 133.3 (C(3a)), 133.4 (C(4a′)), 135.9 (C(2′)), 136.8 (C(7a)), 160.7 (C(C=N)), 168.9 (C=O). MS (ESI), *m/z*: 365.1269 $[M + Na]^+$. $C_{22}H_{18}N_2NaO_2$. Calculated: M = 365.1260. R_f 0.26 (EtOAc—hexane, 1 : 4).

11*H*-Indolo[3,2-*c*]quinolines 8a—f (general procedure). A 5-mL round bottom flask was charged with 2-aryl-1*H*-indole-3-carbaldehyde *O*-acetyl oxime **7a—f** (1 mmol), toluene (1 mL), and $SnCl_4$ (0.354 mL, 3 mmol). The mixture was refluxed for 7—12 h (TLC monitoring). After the reaction completion, the mixture was cooled, poured into water (20 mL), and acidified with 20% aqueous ammonia (appr. 20 mL) to pH 9. The resulting solution was extracted with ethyl acetate (4×20 mL) and the solvent was removed *in vacuo*. The products were purified by silica gel column chromatography (gradient elution with acetone—hexane, 1 : 4→1 : 1).

11*H*-Indolo[3,2-*c*]quinoline (8a). Colorless crystals, yield 100 mg (46%), m.p. 340—341 °C (*cf.* Ref. 30: m.p. 340—341 °C). IR (ATR), ν/cm^{-1}: 3047, 2775, 1571, 1519, 1462, 1373, 1341, 1242, 1158, 933, 771, 740. ^{1}H NMR (DMSO-d_6), δ: 7.34 (t, 1 H, H(8), *J* = 7.3 Hz); 7.55—7.45 (m, 1 H, H(9)); 7.78—7.65 (m, 3 H, H(2), H(3), H(10)); 8.14 (d, 1 H, H(7), *J* = 7.9 Hz); 8.32 (d, 1 H, H(1), *J* = 7.8 Hz); 8.53 (dd, 1 H, H(4), *J* = 8.0 Hz, *J* = 1.1 Hz); 9.60 (s, 1 H, H(6)); 12.73 (s, 1 H, NH). ^{13}C NMR (DMSO-d_6), δ: 111.8 (C(10)), 114.3 (C(6a)), 117.1 (C(6b)), 120.1 (C(9)), 120.6 (C(7)), 121.9 (C(11b)), 122.1 (C(8)), 125.5 (C(2)), 125.7 (C(3)), 128.0 (C(1)), 129.5 (C(4)), 138.8 (C(10a)), 139.8 (C(11a)), 144.8 (C(6)), 145.4 (C(4a)). MS (ESI), *m/z*: 219.0911 $[M + H]^+$. $C_{15}H_{11}N_2$. Calculated: M = 219.0917. R_f 0.19 (ethyl acetate). R_f 0.5 (hexane—acetone, 1 : 1).

12*H*-Benzo[*g*]indolo[3,2-*c*]quinoline (8b). White substance, yield 94 mg (35%), m.p. 400 °C. IR (ATR), ν/cm^{-1}: 3494, 2934, 1741, 1462, 1378, 1260, 1077, 1027. ^{1}H NMR (DMSO-d_6), δ: 7.37 (ddd, 1 H, H(9), *J* = 7.9 Hz, *J* = 7.1 Hz, *J* = 0.9 Hz); 7.50 (ddd, 1 H, H(10), *J* = 8.2 Hz, *J* = 7.2 Hz, *J* = 1.1 Hz); 7.67—7.58 (m, 2 H, H(2), H(3)); 7.76 (d, 1 H, H(11), *J* = 8.1 Hz); 8.27—8.15 (m, 2 H, H(1), H(4)); 8.33 (d, 1 H, H(8), *J* = 7.8 Hz); 8.79 (s, 1 H, H(13)); 9.12 (s, 1 H, H(5)); 9.67 (s, 1 H, H(7)); 13.02 (s, 1 H, NH). ^{13}C NMR (DMSO-d_6), δ: 112.1 (C(11)), 112.6 (C(7a), 117.5 (C(7b)), 119.9 (C(9)), 120.6 (C(8)), 120.9 (C(10)), 122.4 (C(12b)), 125.2 (C(2)), 126.0 (C(3)), 126.2 (C(1)), 127.4 (C(4)), 127.9 (C(13)), 128.5 (C(5)), 130.6 (C(12a)), 132.5 (C(13a)), 138.4 (C(4a)), 139.2 (C(5a)), 142.9 (C(11a)), 146.9 (C(7)). MS (ESI), *m/z*: 269.1077 $[M + H]^+$. $C_{19}H_{13}N_2$. Calculated: M = 269.1073. R_f 0.26 (acetone—hexane, 1 : 2).

3-Methoxy-11*H*-indolo[3,2-*c*]quinoline (8c). White substance, yield 122 mg (49%), m.p. 303.1—307.8 °C. IR (ATR), ν/cm^{-1}: 3437, 2930, 1626, 1561, 1500, 1491, 1447, 1378, 1298, 1237, 1187, 1149, 1035. ^{1}H NMR (DMSO-d_6), δ: 3.94 (s, 3 H, Me); 7.31 (ddd, 1 H, H(8), *J* = 7.9 Hz, *J* = 7.3 Hz, *J* = 0.7 Hz); 7.35 (dd, 1 H, H(2), *J* = 9.0 Hz, *J* = 2.6 Hz); 7.45 (ddd, 1 H, H(9), *J* = 8.2 Hz, *J* = 7.1 Hz, *J* = 1.1 Hz), 7.55 (d, 1 H, H(4), *J* = 2.5 Hz); 7.67 (d, 1 H, H(10), *J* = 8.1 Hz); 8.26 (d, 1 H, H(7), *J* = 7.8 Hz); 8.42 (d, 1 H, H(1), *J* = 9.0 Hz); 9.52 (s, 1 H, H(6)); 12.57 (s, 1 H, NH). ^{13}C NMR (DMSO-d_6), δ: 55.4 (Me), 109.0 (C(10)), 111.7 (C(6a)), 111.4 (C(4)), 113.4 (C(11b)), 117.4 (C(8)), 119.9 (C(7)), 120.5 (C(2)), 122.1 (C(6b)), 123.3 (C(9)), 125.2 (C(1)), 138.7 (C(11a)), 140.3 (C(10a)), 145.0 (C(6)), 147.3 (C(4a)), 159.3 (C(3)). MS (ESI), *m/z*: 249.1022 $[M + H]^+$. $C_{16}H_{13}N_2O$. Calculated: M = 249.1022. R_f 0.17 (acetone—hexane, 1 : 1).

3-Chloro-11*H*-indolo[3,2-*c*]quinoline (8d). Colorless crystals, yield 63 mg (25%), m.p. 310.1—313.7 °C (*cf.* Ref. 41: m.p. 310 °C). IR (ATR), ν/cm^{-1}: 3441, 2930, 2850, 1744, 1626, 1565, 1508, 1454, 1344, 1226, 1138, 1073. ^{1}H NMR (DMSO-d_6), δ: 7.36 (t, 1 H, H(8), *J* = 7.5 Hz); 7.52 (t, 1 H, H(9), *J* = 7.5 Hz); 7.78—7.71 (m, 2 H, H(2), H(10)); 8.17 (d, 1 H, H(4), *J* = 1.7 Hz); 8.34 (d, 1 H, H(1), *J* = 7.8 Hz); 8.56 (d, 1 H, H(7), *J* = 8.8 Hz); 9.64 (s, 1 H, H(6)); 12.86 (s, 1 H, NH). ^{13}C NMR (DMSO-d_6), δ: 112.0 (C(10)), 114.7 (C(6a)), 115.8 (C(11b)), 120.3 (C(8)), 120.9 (C(7)), 121.7 (C(6b)), 124.2 (C(9)), 126.0 (C(1)), 126.2 (C(2)), 128.3 (C(4)), 132.5 (C(11a)), 138.9 (C(3)), 139.6 (C(10a)), 145.9 (C(4a)), 146.1 (C(6)). MS (ESI), *m/z*: 253.0525 $[M + H]^+$. $C_{15}H_{10}ClN_2$. Calculated: M = 253.0527. R_f 0.28 (EtOAc—hexane, 1 : 1). R_f 0.33 (acetone—hexane, 1 : 2).

2,3-Dihydro-12*H*-[1,4]dioxino[2,3-*g*]indolo[3,2-*c*]quinoline (8e). White substance, yield 105 mg (38%), m.p. 390.0—393.3 °C. IR (ATR), ν/cm^{-1}: 2983, 1569, 1512, 1474, 1367, 1344, 1290, 1245, 1187, 1069. ^{1}H NMR (DMSO-d_6), δ: 4.41 (s, 4 H, OCH_2CH_2O); 7.29 (t, 1 H, H(9), *J* = 7.5 Hz); 7.44 (ddd, 1 H, H(10), *J* = 8.2 Hz, *J* = 7.1 Hz, *J* = 1.0 Hz); 7.51 (s, 1 H, H(13)); 7.65 (d, 1 H, H(11), *J* = 8.1 Hz); 7.92 (s, 1 H, H(5)); 8.23 (d, 1 H, H(8), *J* = 7.8 Hz); 9.37 (s, 1 H, H(7)); 12.41 (s, 1 H, NH). ^{13}C NMR (DMSO-d_6), δ: 64.2 (C(2)), 64.3 (C(3)), 107.0 (C(5)), 111.7 (C(13)), 112.2 (C(6a)), 113.1 (C(12b)), 114.6 (C(9)), 119.8 (C(8)), 120.4 (C(10)), 122.1 (C(6b)), 125.1 (C(7)), 138.5 (C(12a)), 139.4 (C(11a)), 141.8 (C(5a)), 143.2 (C(7)), 143.4

(C(13a)), 145.3 (C(4a)). MS (ESI), *m/z*: 277.0974 $[M + H]^+$. $C_{17}H_{13}N_2O_2$. Calculated: M = 277.0972. R_f 0.29 (EtOAc).

3-Methyl-11*H*-indolo[3,2-*c*]quinoline (8f). White substance, yield 100 mg (43%), m.p. 365.7—367.7 °C. IR (ATR), ν/cm^{-1}: 3429, 3044, 2934, 168, 1599, 1565, 1496, 1454, 1363, 1340, 1241, 1027. ^{1}H NMR (DMSO-d_6), δ: 2.56 (s, 3 H, Me); 7.32 (ddd, 1 H, H(8), *J* = 7.9 Hz, *J* = 7.1 Hz, *J* = 0.8 Hz); 7.47 (ddd, 1 H, H(9), *J* = 8.2 Hz, *J* = 7.1 Hz, *J* = 1.1 Hz); 7.53 (dd, 1 H, H(2), *J* = 8.3 Hz, *J* = 1.5 Hz); 7.69 (d, 1 H, H(11), *J* = 8.1 Hz); 7.93 (s, 1 H, H(4)); 8.28 (d, 1 H, H(7), *J* = 7.8 Hz); 8.41 (d, 1 H, H(1), *J* = 8.3 Hz); 9.54 (s, 1 H, H(6)); 12.64 (s, 1 H, NH). ^{13}C NMR (DMSO-d_6), δ: 21.5 (Me), 111.8 (C(10)), 114.0 (C(11b)), 114.9 (C(6a)), 120.0 (C(8)), 120.6 (C(7)), 121.9 (C(9)), 122.0 (C(6b)), 125.4 (C(1)), 127.6 (C(2)), 128.8 (C(4)), 137.7 (C(11a)), 138.7 (C(10a)), 139.9 (C(4a)), 144.8 (C(6)), 145.7 (C(3)). MS (ESI), *m/z*: 233.1076 $[M + H]^+$. $C_{16}H_{13}N_2$. Calculated: M = 233.1073. R_f 0.18 (acetone—hexane, 1 : 2).

3-Acetamino-2-arylindoles 15a,b were synthesized from 3-acetyl-2-arylindole *O*-acetyl oximes **14a,b** by the procedure described above for the synthesis of 11*H*-indolo[3,2-*c*]quinolines **8**. Compounds **15a,b** were isolated by silica gel column chromatography (gradient elution with ethyl acetate—hexane, 1 : 4→1 : 1).

***N*-(2-Phenyl-1*H*-indol-3-yl)acetamide (15a).** White substance, yield 230 mg (92%), m.p. 206.1—207.7 °C (benzene) (*cf.* Ref. 40: m.p. 199–203 °C). IR (ATR), ν/cm^{-1}: 3433, 3265, 2918, 2861, 1664, 1588, 1542, 1477, 1443, 1393, 1371, 1275. ^{1}H NMR (DMSO-d_6), δ: 2.10 (s, 3 H, Me); 7.00 (t, 1 H, H(5), *J* = 7.4 Hz); 7.13 (t, 1 H, H(6), *J* = 7.5 Hz); 7.32 (d, 1 H, H(4), *J* = 8.1 Hz); 7.37 (t, 2 H, H(3′), H(5′), *J* = 7.7 Hz); 7.46—7.52 (m, 2 H, H(4), H(4′)); 7.78 (d, 2 H, H(2′), H(6′), *J* = 7.8 Hz); 9.44 (s, 1 H, NHCO); 11.38 (s, 1 H, NH). ^{13}C NMR (DMSO-d_6), δ: 22.8 (Me), 110.8 (C(3a)), 111.4 (C(7)), 118.5 (C(5)), 119.0 (C(4)), 121.9 (C(6)), 126.2 (C(2)), 126.7 (2 C, C(2), C(6)); 127.4 (C(4′)), 128.7 (2 C, C(3), C(5)); 131.3 (C(7a)), 131.7 (C(3)), 134.5 (C(1′)), 169.6 (C=O). MS (ESI), *m/z*: 273.1003 $[M + Na]^+$. $C_{16}H_{14}N_2NaO$. Calculated: M = 273.0998. R_f 0.17 (EtOAc—hexane, 1 : 1).

***N*-(2-(Naphthalen-2-yl)-1*H*-indol-3-yl)acetamide (15b).** White substance, yield 270 mg (90%), m.p. 189.8—191.1 °C (benzene). IR (ATR), ν/cm^{-1}: 3364, 3258, 3048, 2926, 1649, 1504, 1367, 1344, 1313, 1271, 1245, 1012. ^{1}H NMR (DMSO-d_6), δ: 2.16 (s, 3 H, Me); 7.04 (ddd, 1 H, H(5), *J* = 7.8 Hz, *J* = 6.9 Hz, *J* = 0.7 Hz); 7.17 (ddd, 1 H, H(6), *J* = 8.1 Hz, *J* = 7.0 Hz, *J* = 1.0 Hz); 7.39 (d, 1 H, H(4), *J* = 7.9 Hz); 7.44 (d, 1 H, H(7), *J* = 8.1 Hz); 7.62—7.52 (m, 2 H, H(6′), H(7′)); 7.93—7.99 (m, 3 H, H(3′), H(4′), H(5′)); 8.03 (d, 1 H, H(8′), *J* = 8.8 Hz); 8.31 (s, 1 H, H(1′)), 9.57 (s, 1 H, NHCO); 11.56 (s, 1 H, NH). ^{13}C NMR (DMSO-d_6), δ: 22.9 (Me), 111.4 (C(3a)), 111.4 (C(7)), 118.6 (C(5)), 119.2 (C(4)), 122.1 (C(6)), 124.8 (C(6′)), 125.2 (C(7′)), 126.2 (C(8a′)), 126.3 (C(1′)), 126.7 (C(3′)), 127.7 (C(4′)), 128.0 (C(5′)), 128.2 (C(8′)), 129.3 (C(2)), 131.1 (C(7a)), 132.1 (C(2′)), 133.1 (C(3)), 134.8 (C(4a′)), 169.7 (C=O). MS (ESI), *m/z*: 323.1160 $[M + Na]^+$. $C_{20}H_{16}N_2NaO$. Calculated: M = 323.1155. R_f 0.39 (acetone—hexane, 1 : 1). R_f 0.67 (ethyl acetate—hexane, 1 : 1).

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