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Functionalization of Oxadiazolylindole Systems

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Abstract—A new synthetic approach was developed to the preparation of functional derivatives of 3-(1,3,4-oxadiazol-2-yl)-1*H*-indoles starting with a synthetically available 3-[5-(chloromethyl)-1,3,4-oxadiazol-2-yl]-1*H*-indole. The versatility of the developed strategy was demonstrated in the synthesis of this kind compounds with a wide range of substituents.

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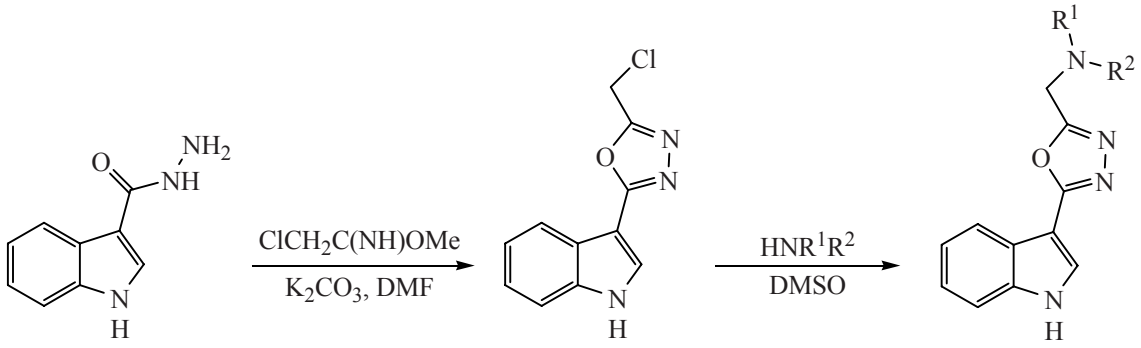
Indoles and their derivatives underlie many natural and synthetic compounds endowed with a wide range of biological action. In particular, among them substances were found exhibiting fungicidal properties [1], compounds simulating the recognition sites on the membranes of brain cells [2]. Some 5-mercapto derivatives of 3-(1,3,4-oxadiazol-2-yl)-1*H*-indoles possess bactericidal qualities [3]. These examples show that the heterocyclic structures containing the 3-(1,3,4-oxadiazol-2-yl)-1*H*-indole fragment belong to an important class of compounds with a wide range of biological action that can be extended by the synthesis of new compounds of this type. The development of efficient approaches to the synthesis of these structure will favorably affect the creation of new physiologically active substances.

In the method reported here of the synthesis of 3-(1,3,4-oxadiazol-2-yl)-1*H*-indoles as well as in a number of methods previously described [4, 5] the key reaction consists in the condensation of methyl 1*H*-indole-3-carboxyimidoate hydrochloride with chloroacetic acid iminoester affording the skeleton of the target structures.

We chose as the initial reagent the synthetically available 3-[5-(chloromethyl)-1,3,4-oxadiazol-2-yl]-1*H*-indole (**I**) prepared from 1*H*-indole-3-carboxylic acid hydrazide (**II**) [4]**.** We developed a sequence of synthetic stages occurring with medium and high yields at a wide range of substituents.

In the first stage of the study we synthesized along the known method [6, 7] in 72–78% yields 2-methylene-

Scheme 1.



II **I** **IIIa–IIId**

R^1,R^2 = morpholin-4-yl (**a**), pyrrolidin-1-yl (**b**), piperidin-1-yl (**c**), 4-methylpiperidin-1-yl (**d**).

amino derivatives of 5-(1*H*-indole-3-yl)-1,3,4-oxadiazol (**III**) (Scheme 1).

The published methods of the preparation of 3-(1,3,4-oxadiazol-2-yl)-1*H*-indoles N-alkyl derivatives involve the indole system alkylation at the starting stages of the synthesis before the formation of the oxadiazole ring. In particular Farghaly Abdel-Rahman [8] used this approach in the synthesis of 1-benxyl-3-[1,3,4]oxadiazol-2-yl-1*H*-indole. The known preparation method of 1-methyl-3-(5-pyridin-4-yl[1,3,4]oxadiazol-2-yl)-1*H*-indole [2] also consisted in a similar approach. The first stage in this synthetic route involved the methylation of the indole system with dimethyl sulfate [9]. We showed for the first time that 3-(1,3,4-oxadiazol-2-yl)-1*H*-indoles could be alkylated with methyl chloroacetate obtaining the target esters in high yields (Scheme 2).

Esters **IVa–IVd** obtained were converted in high yields under mild conditions into the corresponding acids **Va–Vd**. The hydrolysis was performed in the THF–water mixture with lithium hydroxide followed by the treatment with the hydrochloric acid. The acids obtained were used in further stage without additional purification. The amination of acids **Va–Vd** under mild conditions gave a wide range of the corresponding amides **VI** (Scheme 3). The amination was successfully carried out involving various linear and branched aliphatic **VIIa–VIIf**, alicyclic **VIIIa–VIIIe**, aromatic amines like phenylalkylamines **IXa–IXe** and substituted anilines **Xa–Xe**, heterocyclic **XIa–XIf** and heteroaromatic **XIIa–XIIe** amines.

The preparation of amides **VI** from the corresponding acids **V** was carried out by the classic procedure applying carbonyldiimidazole (CDI) as the carboxy group activator. The employed amidation method is of a general character unlke, e.g., the preparation of amides via the corresponding esters **IV**. We failed to obtain target reaction products **VI** attempting to prepare them by the latter method using certain aromatic and heteroaromatic amines, or the reaction did not result in the complete conversion of ester **IV** into the amide, and it decreased the yield of compound **VI** and required its additional purification.

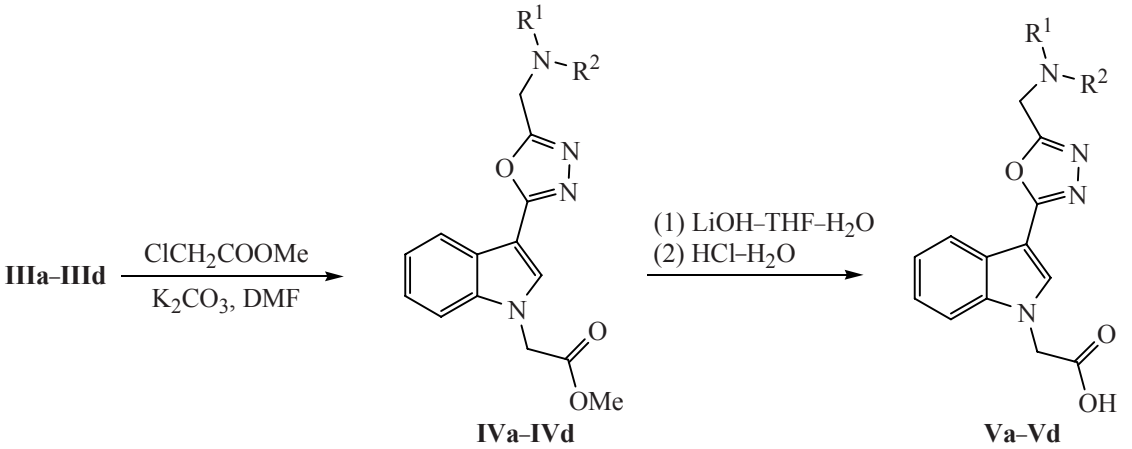
The structure and the composition of all compounds synthesized were confirmed by ^{1}H NMR spectra and elemental analyses. The purity of compounds and their molecular mass were determined by HPLC-MS method. The general procedure and the most typical examples of amides **VI** syntheses are presented in EXPERIMENTAL. The detailed characteristics of the other amides of this series are available from the authors.

The developed synthetic strategy can be efficiently used for creating large combinatorial libraries. The developed procedures can be easily reproduced on large amounts of reagents. The relatively mild reaction conditions, the easy separation and relatively high yields of the target products in the described approach make it a powerful tool in the combinatorial chemistry. The developed synthetic approach provides a possibility of the preparation of a great versatility of new potentially biologically active polysubstituted 3-(1,3,4-oxadiazol-2-yl)-1*H*-indoles.

EXPERIMENTAL

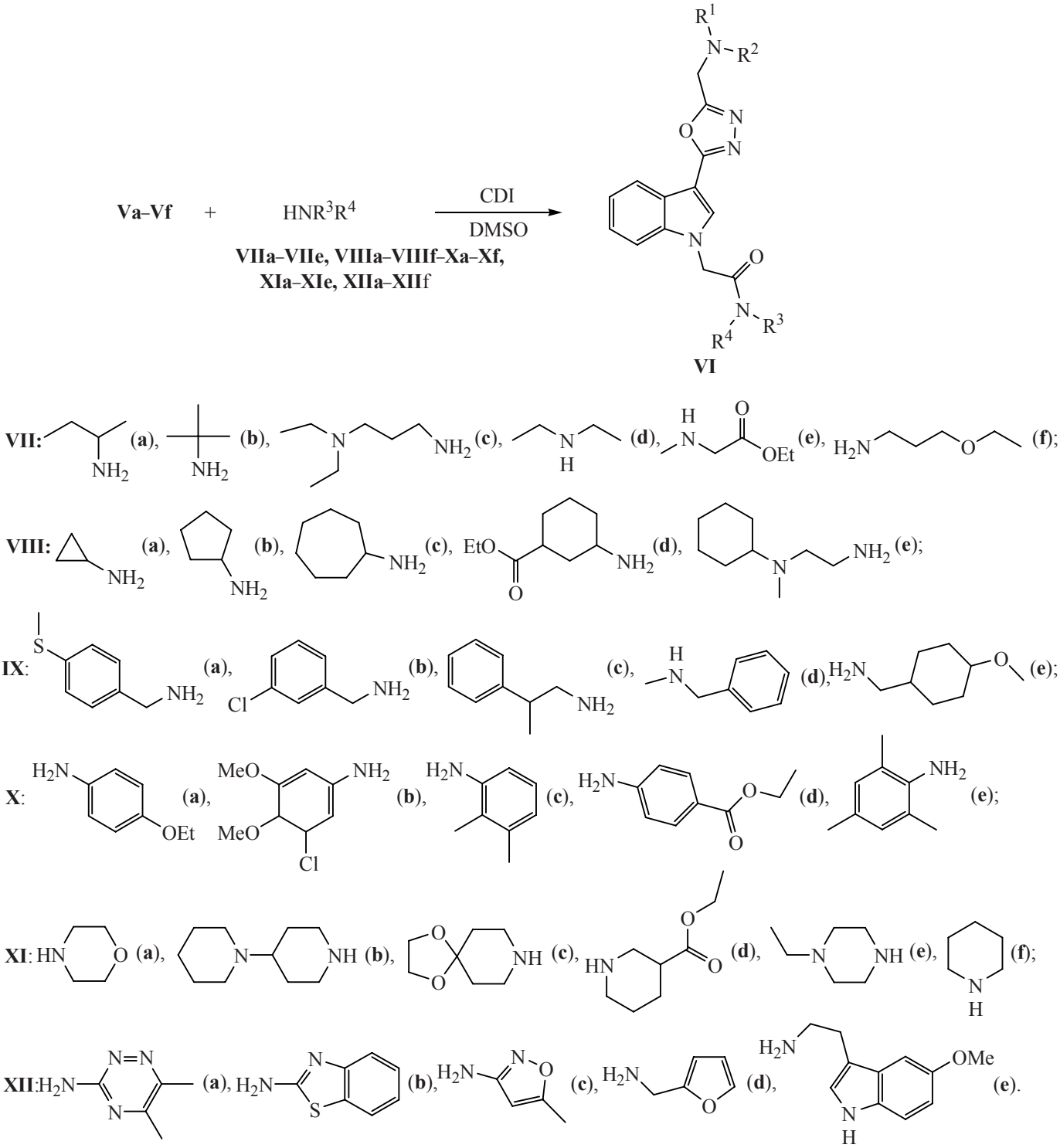
Thin layer chromatography was performed on Kieselgel 60 F-254 plates. ^{1}H NMR spectra from solutions of studied compounds in DMSO-d_6 were registered on

Scheme 2.



IVa–IVd **Va–Vd**

Scheme 3.



a spectrometer Bruker DPX-400 (400.1300 MHz, 25°C), internal reference TMS. Spectra HPLC-MS were obtained with the use of liquid chromatograph PE SCIEX API 150EX equipped with a column C_{18} (100 × 4 mm) and a spectrophotometric detector operating on wavelengths $\lambda_{max.}$ 220 and 254 nm. According to the HPLC-MS data the purity of all compounds obtained exceeded 95%. All the solvents and reagents used in the study were

purchased from Acros Organics, Aldrich, and ChemDiv and were applied without additional purification.

3-[5-(Chloromethyl)-1,3,4-oxadiazol-2-yl]-1*H*-indole (I) [4]. Yield 93%, mp 212–214°C. ^{1}H NMR spectrum, δ, ppm: 7.22–7.32 m (2H, Ar), 7.52–7.58 m (1H, Ar), 8.07–8.13 m (1H, Ar), 8.21 d (1H, C^{2}H, *J* 2.9 Hz), 12.07 br.s (1H, NH). Mass spectrum: *m/z* 235 [*M* + 1]$^+$. Found, %: C 56.66; H 3.51; Cl 15.09; N 18.10. $C_{11}H_8ClN_3O$. Calculated, %: C 56.54; H 3.45; Cl 15.17; N 17.98.

3-[5-(Morpholin-4-ylmethyl)-1,3,4-oxadiazol-2-yl]-1*H*-indole (IIIa) [6]. Yield 77%, mp 179–181°C. ^{1}H NMR spectrum, δ, ppm: 2.52–2.59 m [4H, N$(CH_2)_2$], 3.58–3.65 m [4H, O$(CH_2)_2$], 3.84 s (2H, CH_2), 7.19–7.28 m (2H, Ar), 7.51–7.58 m (1H, Ar), 8.07–8.13 m (2H, C^{2}H and Ar), 11.87 br.s (1H, NH). Mass spectrum: *m/z* 285 [*M* + 1]$^+$. Found, %: C 63.48; H 5.71; N 19.89. $C_{15}H_{16}N_4O_2$. Calculated, %: C 63.37; H 5.67; N 19.71.

3-[5-(Pyrrolidin-1-ylmethyl)-1,3,4-oxadiazol-2-yl]-1*H*-indole (IIIb) [6]. Yield 91%, mp 176–178°C. ^{1}H NMR spectrum, δ, ppm: 1.74–1.81 m [4H, $(CH_2)_2$], 2.60–2.68 m [4H, N$(CH_2)_2$], 3.91 s (2H, CH_2), 7.16–7.27 m (2H, Ar), 7.48–7.54 m (1H, Ar), 8.03–8.12 m (2H, C^{2}H and Ar), 11.88 br.s (1H, NH). Mass spectrum: *m/z* 269 [*M* + 1]$^+$. Found, %: C 67.18; H 6.12; N 20.89. $C_{15}H_{16}N_4O$. Calculated, %: C 67.15; H 6.01; N 20.88.

3-[5-(Piperidin-1-ylmethyl)-1,3,4-oxadiazol-2-yl]-1*H*-indole (IIIc) [6]. Yield 97%, mp 186–188°C. ^{1}H NMR spectrum, δ, ppm: 1.36–1.46 m (2H, CH_2), 1.51–1.61 m [4H, $(CH_2)_2$], 2.47–2.53 m [4H, N$(CH_2)_2$], 3.77 s (2H, CH_2), 7.16–7.27 m (2H, Ar), 7.48–7.54 m (1H, Ar), 8.03–8.12 m (2H, C^{2}H and Ar), 11.94 br.s (1H, NH). Mass spectrum, *m/z* 283 [*M* + 1]$^+$. Found, %: C 68.13; H 6.49; N 19.99. $C_{16}H_{18}N_4O$. Calculated, %: C 68.06; H 6.43; N 19.84.

3-[5-(4-Methylpiperidin-1-ylmethyl)-1,3,4-oxadiazol-2-yl]-1*H*-indole (IIId) [6]. Yield 94%, mp 171–173°C. ^{1}H NMR spectrum, δ, ppm: 0.92 d (3H, CH_3, *J* 6.4 Hz), 1.14–1.27 m (2H, CH_2), 1.30–1.40 m (1H, CH), 1.58–1.65 m (2H, CH_2), 2.10–2.19 m (2H, CH_2), 2.86–2.92 m (2H, CH_2), 3.79 s (2H, CH_2), 7.18–7.27 m (2H, Ar), 7.49–7.53 m (1H, Ar), 8.06 s (1H, C^{2}H), 8.08–8.12 m (2H, Ar), 11.92 br.s (1H, NH). Mass spectrum, *m/z* 297 [*M* + 1]$^+$. Found, %: C 68.93; H 6.94; N 18.99. $C_{17}H_{20}N_4O$. Calculated, %: C 68.89; H 6.80; N 18.90.

Methyl esters IVa–IVd. General procedure. To a solution of 0.1 mol of compound **IIIa–IIId** in 110 ml of anhydrous DMF was added 0.12 mol of methyl chloroacetate and 0.14 mol of calcined K_2CO_3. The reaction mixture was heated at 50°C for 3 h, then it was cooled to room temperature and poured into cold water at vigorous stirring. The formed precipitate was filtered off, washed with cold water and hexane.

Methyl 3-[5-(morpholin-4-ylmethyl)-1,3,4-oxadiazol-2-yl]-1*H*-indole-1-acetate (IVa). Yield 74%, mp 130–132°C. ^{1}H NMR spectrum, δ, ppm: 2.56–2.62 m [4H, N$(CH_2)_2$], 3.60–3.65 m [4H, O$(CH_2)_2$], 3.76 s (3H, OCH_3), 3.82 s (2H, CH_2), 5.27 s (2H, CH_2COOMe), 7.25–7.33 m (2H, Ar), 7.51–7.56 m (1H, Ar), 8.09–8.13 m (1H, Ar), 8.14 s (1H, C^{2}H). Mass spectrum: *m/z* 357 [*M* + 1]$^+$. Found, %: C 60.84; H 5.61; N 15.79. $C_{18}H_{20}N_4O_4$. Calculated, %: C 60.66; H 5.66; N 15.72.

Methyl 3-[5-(pyrrolidin-1-yl-methyl)-1,3,4-oxadiazol-2-yl]-1*H*-indole-1-acetate (IVb). Yield 80%, mp 142–144°C. ^{1}H NMR spectrum, δ, ppm: 1.75–1.83 m [4H, $(CH_2)_2$], 2.62–2.70 m [4H, N$(CH_2)_2$], 3.78 s (3H, OCH_3), 3.88 s (2H, CH_2), 5.24 s (2H, CH_2COOMe), 7.22–7.32 m (2H, Ar), 7.50–7.56 m (1H, Ar), 8.08–8.12 m (1H, Ar), 8.16 s (1H, C^{2}H). Mass spectrum: *m/z* 341 [*M* + 1]$^+$. Found, %: C 63.58; H 5.99; N 16.44. $C_{18}H_{20}N_4O_3$. Calculated, %: C 63.52; H 5.92; N 16.46.

Methyl 3-[5-(piperidin-1-yl-methyl)-1,3,4-oxadiazol-2-yl]-1*H*-indole-1-acetate (IVc). Yield 67%, mp 134–136°C. ^{1}H NMR spectrum, δ, ppm: 1.38–1.46 m (2H, CH_2), 1.53–1.61 m [4H, $(CH_2)_2$], 2.48–2.54 m [4H, N$(CH_2)_2$], 3.74 s (3H, OCH_3), 3.80 s (2H, CH_2), 5.27 s (2H, CH_2COOMe), 7.24–7.34 m (2H, Ar), 7.50–7.55 m (1H, Ar), 8.12–8.15 m (1H, Ar), 8.17 C (1H, C^{2}H). Mass spectrum: *m/z* 355 [*M* + 1]$^+$. Found, %: C 64.48; H 6.29; N 15.88. $C_{19}H_{22}N_4O_3$. Calculated, %: C 64.39; H 6.26; N 15.81.

Methyl 3-{5-[(4-methyl-1-piperidinyl)methyl]-1,3,4-oxadiazol-2-yl}-1*H*-indole-1-acetate (IVd). Yield 87%, mp 127–129°C. ^{1}H NMR spectrum, δ, ppm: 0.91 d (3H, CH_3, *J* 6.4 Hz), 1.14–1.27 m (2H, CH_2), 1.28–1.40 m (1H, CH), 1.58–1.65 m (2H, CH_2), 2.10–2.19 m (2H, CH_2), 2.86–2.92 m (2H, CH_2), 3.73 s (3H, OCH_3), 3.80 s (2H, CH_2), 5.25 s (2H, CH_2COOMe), 7.24–7.32 m (2H, Ar), 7.48–7.54 m (1H, Ar), 8.11–8.13 m (1H, Ar), 8.15 s (1H, C^{2}H). Mass spectrum: *m/z* 369 [*M* + 1]$^+$. Found, %: C 65.24; H 6.69; N 15.29. $C_{20}H_{24}N_4O_3$. Calculated, %: C 65.20; H 6.57; N 15.21.

Acids Va–Vd. *General procedure.* To a solution of 0.1 mol of ester **IVa–IVd** in 100 ml of THF was added a solution of 0.12 mol of lithium hydroxide in 25 ml of water. The reaction mixture was kept for 3–4 h at room

temperature while vigorous stirring [TLC monitoring, (EtOH–CH_2Cl_2, 5 : 95)]. The reaction mixture was diluted with water, and concn. HCl was added till pH 2. The separated precipitate was filtered off, washed with a little of ice water, with ethanol, and hexane.

3-[5-(Morpholin-4-ylmethyl)-1,3,4-oxadiazol-2-yl]-1*H*-indole-1-acetic acid (Va). Yield 69%, mp 239–241°C. ^{1}H NMR spectrum, δ, ppm: 2.70–2.80 m [4H, $N(CH_2)_2$], 3.90–4.02 m [4H, $O(CH_2)_2$], 4.77 s (2H, CH_2), 5.22 s (2H, CH_2COOH), 7.28–7.36 m (2H, Ar), 7.55–7.60 m (1H, Ar), 8.15–8.19 m (1H, Ar), 8.24 s (1H, C^{2}H), 11.54 br.s (1H, COOH). Mass spectrum: *m/z* 343 [*M* + 1]$^+$. Found, %: C 59.74; H 5.33; N 16.59. $C_{17}H_{18}N_4O_4$. Calculated, %: C 59.64; H 5.30; N 16.37.

3-[5-(Pyrrolidin-1-ylmethyl)-1,3,4-oxadiazol-2-yl]-1*H*-indole-1-acetic acid (Vb). Yield 88%, mp 249–251°C. ^{1}H NMR spectrum, δ, ppm: 1.99–2.11 m [4H, $(CH_2)_2$], 3.59–3.66 m [4H, $N(CH_2)_2$], 4.71 b (2H, CH_2), 5.20 b (2H, CH_2COOH), 7.26–7.35 m (2H, Ar), 7.56–7.61 m (1H, Ar), 8.14–8.20 m (1H, Ar), 8.26 b (1H, C^{2}H), 11.70 br.s (1H, COOH). Mass spectrum: *m/z* 327 [*M* + 1]$^+$. Found, %: C 62.68; H 5.59; N 17.21. $C_{17}H_{18}N_4O_3$. Calculated, %: C 62.57; H 5.56; N 17.17.

3-[5-(Piperidin-1-ylmethyl)-1,3,4-oxadiazol-2-yl]-1*H*-indole-1-acetic acid (Vc). Yield 67%, mp 224–227°C. ^{1}H NMR spectrum, δ, ppm: 1.80–1.90 m [4H, $(CH_2)_2$], 3.05–3.20 m (2H, NCH_2), 3.50–3.67 m (2H, NCH_2), 4.73 s (2H, CH_2), 5.23 s (2H, CH_2COOH), 7.27–7.36 m (2H, Ar), 7.57–7.61 m (1H, Ar), 8.16–8.20 m (1H, Ar), 8.25 s (1H, C^{2}H), 11.60 br.s (1H, COOH). Mass spectrum: *m/z* 341 [*M* + 1]$^+$. Found, %: C 63.55; H 5.96; N 16.49. $C_{18}H_{20}N_4O_3$. Calculated, %: C 63.52; H 5.92; N 16.46.

3-{5-[(4-Methylpiperidin-1-yl)methyl]-1,3,4-oxadiazol-2-yl}-1*H*-indole-1-acetic acid (Vd). Yield 93%, mp 231–233°C. ^{1}H NMR spectrum, δ, ppm: 0.93 d (3H, CH_3, *J* 6.4 Hz), 1.50–1.64 m (3H, CH + CH_2), 1.77–1.86 m (2H, CH_2), 3.05–3.17 m (2H, CH_2), 3.65–3.55 m (2H, CH_2), 4.73 s (2H, CH_2), 5.22 s (2H, CH_2COOH), 7.27–7.36 m (2H, Ar), 7.56–7.61 m (1H, Ar), 8.15–8.19 m (1H, Ar), 8.24 s (1H, C^{2}H), 11.45 br.s (1H, COOH). Mass spectrum: *m/z* 355 [*M* + 1]$^+$. Found, %: C 64.34; H 6.39; N 15.79. $C_{19}H_{22}N_4O_3$. Calculated, %: C 64.39; H 6.26; N 15.81.

Amides VI. *General procedure*. To a solution of 2 mmol of acyd **Va–Vd** in 8 ml of DMSO was added 2.4 mmol of carbonyldiimidazole. The mixture was heated to 50°C for 30 min, then to the solution was added 2.2 mmol of amine **VII–XII**. The reaction mixture was stirred for 3–4 h at 60°C, cooled to room temperature, and poured into cold water. The separated precipitate was filtered off, washed with water and hexane. The oily substance was extracted into ethyl acetate, the extract was washed with a NaCl solution and with water, and dried with Na_2SO_4. On removing the solvent under a reduced pressure the reaction products was purified by crystallization from a mixture benzene–hexane or from isooctane, or by column chromatography on silica gel, eluent ethyl acetate–hexane, 25:75.

***N*-{[4-(Methylthio)phenyl]methyl-3-[5-(morpholin-4-ylmethyl)-1,3,4-oxadiazol-2-yl]-1*H*-indol-1-yl}acetamide (VIa).** Yield 84%, mp 152–154°C. ^{1}H NMR spectrum, δ, ppm: 2.46 s (3H, SCH_3), 2.55–2.63 m [4H, $N(CH_2)_2$], 3.62–3.69 m [4H, $O(CH_2)_2$], 3.84 s (2H, CH_2), 4.28 d (2H, $NHCH_2$, *J* 5.9 Hz), 4.97 s (2H, CH_2CONH), 7.17–7.23 m (4H, Ar), 7.24–7.33 m (2H, Ar), 7.46–7.52 m (1H, Ar), 8.07–8.11 m (1H, Ar), 8.13 s (1H, C^{2}H), 8.68 t (1H, NH, *J* 5.9 Hz). Mass spectrum: *m/z* 479 [*M* + 1]$^+$. Found, %: C 62.74; H 5.93; N 14.60. $C_{25}H_{27}N_5O_3S$. Calculated, %: C 62.87; H 5.70; N 14.66.

***N*-Methyl-N-benzyl-{3-[5-(pyrrolidin-1-ylmethyl)-1,3,4-oxadiazol-2-yl]-1*H*-indol-1-yl}acetamide (VIb).** Yield 58%, mp 117–119°C. ^{1}H NMR spectrum, δ, ppm: 1.76–1.84 m [4H, $(CH_2)_2$], 2.67–2.74 m [4H, $N(CH_2)_2$], 3.10 s (3H, NCH_3), 3.88 s (2H, CH_2), 4.55 s (2H, NCH_2), 4.94 s (2H, CH_2CON), 7.20–7.40 m (7H, Ar), 7.50–7.56 m (1H, Ar), 8.06–8.11 m (1H, Ar), 8.14 s (1H, C^{2}H). Mass spectrum: *m/z* 431 [*M* + 1]$^+$. Found, %: C 69.97; H 6.33; N 16.39. $C_{25}H_{27}N_5O_2$. Calculated, %: C 69.91; H 6.34; N 16.31.

***N*-Cycloheptyl-{3-[5-(piperidin-1-ylmethyl)-1,3,4-oxadiazol-2-yl]-1*H*-indol-1-yl}acetamide (VIc).** Yield 59%, mp 89–91°C. ^{1}H NMR spectrum, δ, ppm: 1.35–1.65 m (17H), 1.74–1.84 m (2H, CH_2), 2.46–2.49 m (3H), 3.70–3.78 m (1H, CH), 3.82 s (2H, CH_2), 4.92 s (2H, CH_2CON), 7.24–7.33 m (2H, Ar), 7.44–7.49 m (1H, Ar), 8.07–8.12 m (1H, Ar), 8.17 s (1H, C^{2}H), 8.28 d (1H, NH, *J* 7.7 Hz). Mass spectrum: *m/z* 437 [*M* + 1]$^+$. Found, %: C 69.03; H 7.73; N 16.09. $C_{25}H_{33}N_5O_2$. Calculated, %: C 68.94; H 7.64; N 16.08.

***N*-(2-Furylmethyl)-(3-{5-[(4-methylpiperidin-1-yl)methyl]-1,3,4-oxadiazol-2-yl}-1*H*-indol-1-yl)-acetamide (VId).** Yield 71%, mp 114–116°C. ^{1}H NMR spectrum, δ, ppm: 0.88 d (3H, CH_3, *J* 6.4 Hz), 1.09–1.24 m (2H, CH_2), 1.25–1.37 m (1H, CH), 1.55–1.64 m (2H, CH_2), 2.07–2.19 m (2H, CH_2), 2.82–2.92 m (2H, CH_2), 3.83 s (2H, CH_2), 4.31 d (2H, $NHCH_2$, *J* 6.0 Hz),

5.02 c (2H, CH_2CONH), 6.27 d (1H, C^3H_{furan}, *J* 3.0 Hz), 6.40 d.d (1H, C^4H_{furan}, *J* 1.8, *J* 3.0 Hz), 7.25–7.34 m (2H, Ar), 7.46–7.51 m (1H, Ar), 7.59 d (1H, C^5H_{furan}, *J* 1.8 Hz), 8.08–8.13 m (1H, Ar), 8.20 s (1H, C^2H), 8.78 t (1H, NH, *J* 6.0 Hz). Mass spectrum: *m/z* 435 $[M + 1]^+$. Found, %: C 66.57; H 6.33; N 16.19. $C_{24}H_{27}N_5O_3$. Calculated, %: C 66.49; H 6.28; N 16.16.

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