B. This was obtained in a manner similar to method B for the bromotriazole (Vb) from the ester (Va) and 25% aqueous methylamine. Reaction time 2 h. According to IR and PMR spectroscopy and its melting point the product was identical with that obtained by method A.

4-Benzoyl-5-iodo-1,2,3-triazole trihydrate (VId) was obtained in a similar manner to the iodotriazole (VIa) from diazo compound (Id) and hydrogen iodide. Reaction time 1 day. Colorless prisms.

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SYNTHESIS AND PROPERTIES OF AZOLES AND THEIR DERIVATIVES.

36.* SYNTHESIS OF 2-AMINO-1,3,4-OXADIAZOLES, 2-AMINO-1,3,4-THIADIAZOLES, AND 1,2,4-TRIAZOLINE-3-THIONES CONTAINING INDOLYL RADICALS

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UDC 547.752'793.3:542.953.2:543.422.4

On the heterocyclization of 1-(indol-3-ylcarbonyl)thiosemicarbazides, depending on the conditions of performing the reaction, either 1,2,4-triazoline-3-thiones, 2-amino-1,3,4-thiadiazoles, or 2-amino-1,3,4-oxadiazoles containing an indolyl residue in position 5 are obtained. 1,2,4-Triazoline-3-thiones and 2-amino-1,3,4-thiadazoles have also been synthesized by the reaction of the hydrochloride of ethyl indol-3-carboximidate with thiosemicarbazides.

Continuing an investigation of the synthesis of indolylazoles [1-4], in the present paper we report the preparation of 2-amino-1,3,4-oxadiazoles, 2-amino-1,3,4-thiadiazoles, and 1,2,4-triazoline-3-thiones each containing an indolyl residue. There has been only limited information concerning bisheterocyclic compounds of this type in the literature hitherto [5, 6].

It is known [7, 8] that the starting materials for the synthesis of amino and mercapto derivatives of 1,3,4-oxadiazoles, 1,3,4-thiadiazoles, and 1,2,4-triazoles may be compounds containing a thiosemicarbazide fragment. In the present work, as the initial compound we used the 1-(indol-3-ylcarbonyl)thiosemicarbazides (IIa, b) which are readily formed from indole-3-carbohydrazide (I).

*For communication 35, see [1].

I. M. Gubkin Moscow Institute of the Petrochemical and Gas Industries. Translated from Khimiya Geterotsilicheskikh Soedinenii, No. 9, pp. 1271-1276, September, 1984. Original article submitted January 21, 1984.

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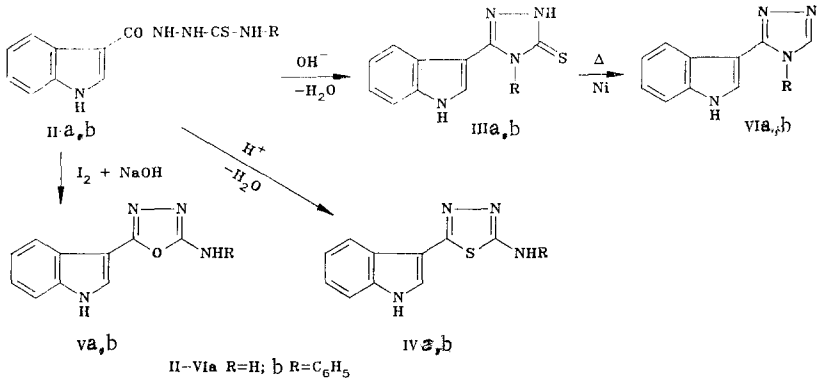


II–VIa R=H; b R=C_6H_5

The study of the heterocyclization of thiosemicarbazides (IIa, b) showed that the nature of the substances formed depends on the conditions of performing the process. It was established that in an alkaline medium the thiosemicarbazides (IIa, b) cyclize into 5-(indol-3-yl)-1,2,4-triazoline-3-thiones (IIIa, b), to which, on the basis of spectral characteristics, the structure of 1,2,4-triazolinethiones, and not of the 3-mercapto-1,2,4-triazoles isomeric with them, was assigned. This was shown by the absence from the IR spectra of absorption in the regions of 2600-2500 cm^{-1} (SH stretching vibrations) and 900-850 cm^{-1} (C—S—H planar deformation vibrations) and by the presence of an intense absorption band at 1525-1518 cm^{-1} belonging to the vibrations of a thioamide group [9] and also of two absorption maxima at 1342 and 1320 cm^{-1} that are characteristic for the vibrations of a C=S group in 1,2,4-triazoline-3-thiones [10]. To the vibrations of the 1,2,4-triazoline ring correspond absorption maxima of medium intensity at 1440, 1245, and 1140 cm^{-1} [11]. Unfortunately, in this case it was impossible clearly to identify the NH group of the 1,2,4-triazoline ring, since the vibrations of the NH group of indole appeared in the same region.

In the mass spectra of compounds (IIIa, b), the peaks of maximum intensity were those of the molecular ions, the m/z values of which corresponded to the calculated molecular masses of the proposed compounds. The general direction of decomposition under the action of electron impact for the 1,2,4-triazoline-3-thiones (IIIa, b) was the detachment of a SH group from the molecular ion; further fragmentation took place by the scheme characteristic for 1,2,4-triazoles [12].

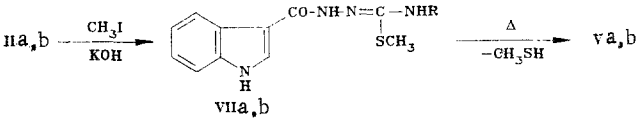
On brief heating with concentrated H_2SO_4, the thiosemicarbazide (IIa) was converted with good yield into 2-amino-5-(indol-3-yl)-1,3,4-thiadiazole (IVa). At the same time, on attempted cyclization of the phenylthiosemicarbazide (IIb) in an acid medium, pronounced resinification of the reaction mixture took place. In order to cyclize compound (IIb) into the corresponding 5-(indol-3-yl)-2-phenylamino-1,3,4-thiadiazole (IVb), we attempted to use $POCl_3$, polyphosphoric acid with heating, and concentrated H_2SO_4 with heating or in the cold. In the first case, judging from the TLC characteristics, the reaction formed not only the 1,3,4-thiadiazole (IVb) but also the 1,2,4-triazoline-3-thione (IIIb) and also several unidentified compounds.

As a result of this series of experiments, it was found that the 1,3,4-thiadiazole (IVb) can be obtained with good yield by performing the reaction in acetyl chloride at room temperature. In this case, no resinification of the reaction mixture or the formation of 1,2,4-triazoline-3-thione (IIIb) as a byproduct was observed.

The IR spectra of compounds (IVa, b) showed absorption bands in the 1515, 1480, 1390, 1235, 1040, and 860 cm^{-1} regions that are characteristic for the vibrations of a 1,3,4-thiadiazole ring [13], and also absorption bands at 1320-1300 cm^{-1}, assigned to the C—N stretching vibrations in aromatic amines.

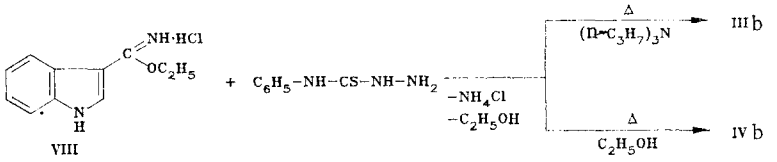
On treatment with an ethanolic solution of iodine in an alkaline medium, the thiosemicarbazides (IIa,b) were converted into the 2-amino-5-(indol-3-yl)-1,3,4-oxadiazoles (Va, b), but the yields of these three compounds did not exceed 50-55%, and a certain amount of the initial thiosemicarbazide was recovered from the reaction mixture unchanged. An increase in the time of the reaction and a rise in the temperature or the replacement of iodine by bromine did not lead to an appreciable increase in the yields of compounds (Va, b).

It is known [14] that 5-substituted 2-amino-1,3,4-oxadiazoles can be obtained from 1-acyl-S-methylisothiosemicarbazides. In the present work we decided to use this method for synthesizing compounds (Va, b). It was established that the 1-(indol-3-ylcarbonyl)-S-methylisocyanosemicarbazides (VIIa, b) formed on the methylation of the corresponding carbazides (IIa, b) readily split out methyl mercaptan on heating and were converted quantitatively into the 2-amino-1,3,4-oxadiazoles (Va, b).



In the IR spectra of the 1,3,4-oxadiazoles (Va, b), absorption bands of variable intensity were observed in the 1625-1620 and 1605-1595 cm^{-1} regions which related to the stretching C=N and C=C vibrations of heteroaromatic nuclei, and in the 1460-1450 cm^{-1} region there were the extremely intense absorption maxima that are characteristic for the 1,3,4-oxadiazole ring [3, 15]. The presence of this ring was also confirmed by absorption bands at 1225-1220 and 1030-1020 cm^{-1}, which relate to the stretching vibrations of the C–O–C fragment in the 1,3,4-oxadiazoles [16].

For the purposes of an independent synthesis of the 1,2,4-triazoline-3-thione (IIIb) and the 2-amino-1,3,4-thiadiazole (IVb), we studied the condensation of the hydrochloride of ethyl indole-3-carboximidate (VIII) with 4-phenylthiosemicarbazide. Similar cases of the cyclocondensation of hydrochlorides of esters of aromatic imidic acids with 4-substituted thiosemicarbazides have been described in the literature, the direction of the reaction usually being determined by the nature of the reaction medium — in the presence of bases 1,2,4-triazoline-3-thiones are formed, and in an acid medium 2-amino-1,3,4-thiadiazoles [17].



In the case of the hydrochloride of the imidic ester (VIII) the direction of cyclization was less clear; for example, when the reaction was performed in pyridine, the two possible isomers (IIIb) and (IVb) were formed in approximately equal amounts. When (VIII) was boiled with 4-phenylthiosemicarbazide in ethanol, the main reaction product was the 2-amino-1,3,4-thiadiazole (IVb), but the 1,2,4-triazole-3-thione (IIIb) was also formed in small amount (15%). Similar results were observed when the reaction was performed in butanol or DMFA. The highest yield (60%) of compound (IIIb) was obtained when the reactants were heated in tripropylamine, but in this case as well, traces of compound (IVb) were detected in the reaction products.

The 1,3,4-thiadiazole (IVa) was also obtained by indirect synthesis — by the reaction of the hydrochloride (VIII) with thiosemicarbazide in ethanol — but this reaction was accompanied by pronounced resinification and the yield of the thiadiazole (IVa) was only 40%.

When ethanolic solutions of the 1,2,4-triazoline-3-thiones (IIIa, b) were boiled with Raney nickel, the corresponding 4-R-3-(indol-3-yl)-1,2,4-triazoles (VIa, b) were formed. In contrast to compounds (IIIa, b), the 2-amino-1,3,4-thiadiazoles (IVa, b) were recovered unchanged even on prolonged boiling in ethanol with Raney nickel.

The IR spectra of the 1,2,4-triazoles (VIa, b) contained, in addition to the absorption frequency characteristic for indoles, a series of bands connected with the vibrations of the 1,2,4-triazole ring [18]: 1475-1470, 1390, 1280-1275, and 1020 cm^{-1}. In addition, there were strong absorption maxima at 1630 and 1145 cm^{-1} which were assigned, respectively, to the vibrations of the C=N group and those of the C–H bond of a 1,2,4-triazole unsubstituted in position 5 [19].

The 1,2,4-triazolinethiones (IIIa, b) gave colored precipitates with heavy-metal salts: with Cu^{2+} — dark green; with Fe^{3+} — bright red; with Ni^{2+} — pale blue; with Co^{2+} — light

TABLE 1. Characteristics of the Compounds (II-VIII) Synthesized

Compound	mp, °C*	R_f (solvent system)	Found				Empirical formula	Calculated				Yield (%) (method of preparation)
			C, %	H, %	N(S), %	M		C, %	H, %	N(S), %	M	
IIa	217—218	0,12 (a)	51,1	4,2	23,3	234	$C_{10}H_{10}N_4OS$	51,3	4,3	23,5	234	60
IIb	203—204	0,16 (a)	62,1	4,4	18,3	310	$C_{16}H_{14}N_4OS$	61,9	4,5	18,1	310	97
IIIa	170—171	0,56 (a)	55,4	3,7	25,7	216	$C_{10}H_8N_4S$	55,5	3,7	25,9	216	58
IIIb	282—284	0,50 (a)	65,7	4,1	19,3	292	$C_{16}H_{12}N_4S$	65,7	4,1	19,2	292	71 (A), 60 (B)
IVa	191—192	0,46 (b)	55,3	3,7	25,8	216	$C_{10}H_8N_4S$	55,5	3,7	25,9	216	76 (A), 50 (B)
IVb	152—154	0,10 (a)	65,6	4,0	19,0	292	$C_{16}H_{12}N_4S$	65,7	4,1	19,2	292	90 (A), 54 (B)
Va	140—141,5	0,20 (a)	60,1	3,9	27,8	200	$C_{10}H_8N_4O$	60,0	4,0	28,0	200	51 (A), 94 (B)
Vb	207—209	0,45 (b)	69,3	4,3	20,5	276	$C_{16}H_{12}N_4O$	69,6	4,3	20,3	276	48 (A), 96 (B)
VIa	144—145	0,22 (b)	65,5	4,4	30,3	184	$C_{10}H_8N_4$	65,4	4,5	30,5	184	74
VIb	217—219	0,34 (b)	74,0	4,6	21,8	260	$C_{16}H_{12}N_4$	73,8	4,6	21,5	260	86
VIIa	184—186 (decomp.)	0,18 (b)			(13,7)		$C_{11}H_{12}N_4OS$			(13,8)		63
VIIb	263—263,5 (decomp.)	0,35 (b)			(10,2)		$C_{17}H_{16}N_4OS$			(9,9)		71

*The compounds were recrystallized from the following solvents: (IIa), from n-butanol; (IIb), from aqueous DMFA; (IIIa, b), (IVa), (Va), and (VIb), from aqueous ethanol; (IVb) and (Vb), from DMFA—water (1:4); (VIa), from aqueous acetone.

green; and with Pb^{2+}, Hg^{2+}, and Zn^{2+} salts — black. To all appearances, in this case compounds (IIIa, b) reacted as mercapto derivatives. In contrast to these compounds, the 2-amino-1,3,4-thiadiazoles (IVa, b) and 2-amino-1,3,4-oxadiazoles (Va, b) did not give colored precipitates or solutions with heavy-metal salts.

EXPERIMENTAL

IR spectra were taken on a UR-20 instrument in tablets with KBr. Mass spectra were obtained on a LKB-2091 instrument using a system for the direct introduction of the sample into the ion source (the energy of the ionizing electrons was 70 eV, the emission current 25 μA, and the temperature of the ion source 200°C), the temperature of evaporation of the samples being 130-140°C. The course of the reactions was followed and the individuality of the substances obtained was checked by the TLC method on Al_2O_3 of Brockmann activity grade III in the solvent systems benzene—methanol (5:1) (a) and benzene—methanol (10:1) (b); the spots were revealed with iodine vapor.

The investigation made use of the indole-3-carbohydrazide (I) and the hydrochloride of ethyl indole-3-carboximidate (VIII) obtained previously ([2] and [3], respectively).

1-(Indol-3-ylcarbonyl)thiosemicarbazide (IIa). A mixture of 1.5 g (8.6 mmole) of (I), 1.1 g (11 mmole) of potassium thiocyanate, and 1.5 ml of concentrated HCl in 20 ml of water was stirred with heating on the water bath for 4 h and was then left overnight. The resulting precipitate was filtered off, dried, and crystallized from n-butanol. IR spectrum, cm^{-1}: 3320-3300, 3200 (NH_2, NH), 1645 (C=O), 1605-1595 (indole ring), 1540, 1455, 1320 (C=S), 1135 (N—N), 750 (CH).

1-(Indol-3-ylcarbonyl)-4-phenylthiosemicarbazide (IIb). To a stirred solution of 1.93 g (11 mmole) of (I) in 25 ml of ethylene glycol at 65-70°C was added 1.5 g (11 mmole) of phenyl isothiocyanate in 20 ml of ethylene glycol—dioxane (1:1). The mixture was stirred in 95-100°C for 2 h and was then cooled and poured into 150 ml of water. The resulting precipitate was filtered off, dried, and crystallized. IR spectrum (cm^{-1}): 3350-3300, 3230 (NH_2, NH), 1650 (C=O), 1605-1590 (indole ring), 1540, 1460, 1440, 1320 (C=S), 1130 (N—N), 750 (CH).

5-(Indol-3-yl)-1,2,4-triazoline-3-thione (IIIa). A mixture of 2.34 g (10 mmole) of (IIa) and 0.75 g (33 mmole) of sodium in 30 ml of absolute ethanol was boiled with stirring for 12 h. Then the reaction mixture was evaporated to dryness under reduced pressure, and

the residue was extracted with hot water (3 × 30 ml). The aqueous extract was cooled to 15 °C and acidified with 10% CH_3COOH to pH 5.5. The resulting precipitate was filtered off, washed with water, and dried. IR spectrum, cm^{-1}: 3300, 3250-3200, 3155 (NH), 1645 (C=N), 1600 (indole ring), 1520 (N–C=S), 1330, 1320 (C=S), 1445, 1230, 1145 (1,2,4-triazoline ring), 1125 (N–N), 750 (CH).

5-(Indol-3-yl)-4-phenyl-1,2,4-triazoline-3-thione (IIIb). A. A suspension of 3.99 g (13 mmole) of (IIIb) in 100 ml of 5% NaOH solution was boiled with stirring for 2 h and was left overnight. The resulting precipitate was filtered off and was suspended with vigorous stirring in 2 ml of water, to which a solution of HCl (1:1) was then added to give pH 4.5-5.0. The resulting precipitate was filtered off, washed with water, dried, and crystallized. IR spectrum, cm^{-1}: 3220-3090 (NH), 1640 (C=N), 1610-1605 (indole ring), 1518 (N–C=S), 1342, 1320 (C=S), 1450, 1220, 1145 (1,2,4-triazole ring), 1120 (N–N), 750 (CH).

B. A mixture of 1.05 g (5 mmole) of the hydrochloride of the imidic ester (VIII) and 0.83 g (5 mmole) of 4-phenylthiosemicarbazide in 20 ml of tripropylamine was boiled with stirring for 10 h. The solvent was driven off in vacuum, the residue was extracted with acetone (2 × 10 ml), and the extract was evaporated to dryness, to give (IIIb). A mixed melting point of the samples of (IIIb) obtained by the two methods gave no depression of the melting point, and their IR spectra were identical.

Compounds (IIIa, b) dissolve at room temperature in acetone, THF, dioxane, and DMFA and, on heating, in alcohol. They do not dissolve in water, hydrocarbons, and chloroform. The reactions of compounds (IIIa, b) with metal salts were carried out in the following way. A few drops of a 1% solution of the substance in acetone was added to 3 ml of a solution of a metal salt.

2-Amino-5-(indol-3-yl)-1,3,4-thiadiazole (IVa). A. A mixture of 1.4 g (6 mmole) of (IIa) and 12 ml of concentrated H_2SO_4 was heated in the water bath for 10 min, cooled to 10 °C, and mixed with 40 ml of water. The reaction mixture was alkalinized with an aqueous solution of ammonia to pH 8.0, heated to the boil and cooled to 0°C, and the resulting precipitate was filtered off. IR spectrum, cm^{-1}: 3350-3300 (NH_2), 3180 (NH of indole), 1610 (C=N), 1600-1595 (indole ring), 1510, 1480, 1240, 1010, 850 (1,3,4-thiadiazole ring), 1340 (C–N), 750 (CH).

Hydrochloride of (IVa): yellow crystals with mp 283-285°C (from ethanol). Found, %: Cl 13.8. $C_{10}H_8N_4 \cdot HCl$. Calculated, %: Cl 14.0.

B. A mixture of 1.05 g (5 mmole) of (VIII) and 0.46 g (5 mmole) of thiosemicarbazide in 15 ml of absolute ethanol was boiled with stirring for 4 h. Then the reaction mixture was evaporated to dryness under reduced pressure and the residual oil was extracted with methanol (2 × 5 ml). The extract was chromatographed on a column of Al_2O_3 (30 × 3.5 cm) with elution by benzene–methanol (10:1). After the solvent had been driven off, 0.54 g of (IVa) was obtained.

5-(Indol-3-yl)-2-phenylamino-1,3,4-thiadiazole (IVb). A. With stirring, 1.18 g (4 mmole) of (IIIb) was added in portions to 10 ml of acetyl chloride. After the end of the vigorous reaction, the reaction mixture was stirred at 20°C for 2 h and was poured onto 50 g of ice. With stirring, the mixture obtained was neutralized with aqueous ammonia to pH 7.0-8.0. The resulting precipitate was filtered off, dried, and crystallized from aqueous ethanol. IR spectrum, cm^{-1}: 3400, 3200-2050 (NH), 1620 (C=N), 1610-1585 (indole ring), 1515, 1475, 1235, 1000, 840 (1,3,4-thiadiazole ring), 1320 (C–N), 750 (CH).

B. A mixture of 1.50 g (7 mmole) of (VIII) and 1.18 g (7.1 mmole) of 4-phenylthiosemicarbazide in 35 ml of absolute ethanol was boiled with stirring for 5 h. The reaction mixture was cooled to 20°C and, with stirring, it was poured dropwise into 200 ml of ice water. The resulting precipitate was filtered off and dried, and crystallized to give (IVb).

Compounds (IVa, b) dissolve at room temperature in alcohols, acetone, and DMFA and are insoluble in hydrocarbons, THF, dioxane, and chloroform.

1-(Indol-3-ylcarbonyl)-S-methylisothiosemicarbazide (VIIa). With stirring, a solution of 0.28 g (5 mmole) of KOH in 10 ml of absolute ethanol was added dropwise to a mixture of 1.17 g (5 mmole) of (IIa) and 2.3 g (14 mmole) of methyl iodide in 25 ml of absolute ethanol. The reaction mixture was stirred at 20°C for 10 h, evaporated to a volume of 8-10 ml, and poured into 50 ml of ice water. After being kept at 0°C for 24 h, the precipitate that had

deposited was filtered off and dried. IR spectrum, cm^{-1}: 3350 (NH_2), 3300-3270, 3180 (NH), 1665 (C=O), 1625 (C=N), 1605-1595 (indole ring), 1335 (C–N), 1150 (C–S), 750 (CH).

1-(Indol-3-ylcarbonyl)-4-phenyl-S-methylisothiosemicarbazide (VIIb) was obtained similarly.

2-Amino-5-(indol-3-yl)-1,3,4-oxadiazole (Va). A. With cooling and stirring, 5 ml of 4 N NaOH was added to a suspension of 2.34 g (10 mmole) of (IIa) in 150 ml of ethanol. Then a solution of iodine in 5% KI solution was added dropwise until the color of the iodine persisted at room temperature. The reaction mixture was heated to the boil and more iodine solution was carefully added until the appearance of permanent coloration of an excess of iodine. Then the mixture was cooled to 20°C and poured onto ice, and the resulting precipitate was filtered off, washed with hot water, and dried. IR spectrum, cm^{-1}: 3410 (NH_2), 3200-3150 (NH), 1625 (C=N), 605-1095 (indole ring), 1455 (1,3,4-oxadiazole ring), 1340 (C–N), 1220, 1030 (C–O–C), 750 (CH).

B. Compound (VIIa) (0.8 g, 3 mmole) was heated at 184-188°C for 5-6 min. Then the reaction mixture was cooled and the residue was crystallized from aqueous ethanol.

5-(Indol-3-yl)-2-phenylamino-1,3,4-oxadiazole (Vb) was obtained in a similar manner to compound (Va) from (IIb) (method A) or from (VIIb) at a temperature of 265-267°C (method B). IR spectrum, cm^{-1}: 3350, 3280-3240 (NH), 1620 (C=N), 1600 (indole ring), 1450 (1,3,4-oxadiazole ring), 1315 (C–N), 1220, 1020 (C–O–C), 760 (CH).

3-(Indol-3-yl)-1,2,4-triazole (VIa). A mixture of 2.16 g (10 mmole) of (IIIa) and 2.5 g of Raney nickel in 50 ml of ethanol was boiled with stirring for 5 h. The reaction mixture was filtered, and the precipitate on the filter was washed with hot ethanol. The filtrate was evaporated to dryness under reduced pressure, giving 1.50 g of (VIa). IR spectrum, cm^{-1}: 3300, 3260-3200, 3120 (NH), 1620 (C=N), 1605-1600 (indole ring), 1470, 1380, 1260, 980 (1,2,4-triazole ring), 1145 (triazole C–H), 750 (CH).

3-(Indol-3-yl)-4-phenyl-1,2,4-diazole (VIb) was obtained in a similar manner to compound (VIa) from 1,2,4-triazoline-3-thione (IIIb). IR spectrum, cm^{-1}: 3300, 3120 (NH), 1605-1590 (indole ring), 1475, 1380, 1270, 990 (1,2,4-triazole ring), 1145 (triazole C–H), 755 (CH).

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