

Synthesis and Selected Transformations of 1-(5-methyl-1-aryl-1*H*-1,2,3-triazol-4-yl)ethanones and 1-[4-(4-*R*-5-methyl-1*H*-1,2,3-triazol-1-yl)phenyl]ethanones

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Abstract—By cycloaddition of arylazides to acetylacetone are obtained derivatives of 1,2,3-triazole. In the reaction of 1-[5-methyl-1-(*R*-phenyl)-1*H*-1,2,3-triazol-4-yl] ethanones (**IIa–IIe**) and 1-[4-(4-*R*-5-methyl-1*H*-1,2,3-triazol-1-yl)phenyl] ethanones (**VIIa–VIIe**) with isatin are obtained 2-[1-(*R*-phenyl)-5-methyl-1*H*-1,2,3-triazol-4-yl]-4-quinolinecarboxylic acids (**IIIa–IIIe**) and 2-[4-(4-*R*-5-methyl-1*H*-1,2,3-triazol-1-yl)phenyl] -4-quinolinecarboxylic acids (**IXa, IXb**), respectively. We found that 1-[5-methyl-1-(*R*-phenyl)-1*H*-1,2,3-triazol-4-yl] ethanones (**IIa–IIe**) readily transform into [5-methyl-1-(*R*-phenyl)-1*H*-1,2,3-triazol-4-yl] acetic acids (**IVa–IVc**) by the method of Wilgerodt–Kindler. The (5-methyl-1-phenyl-1*H*-1,2,3-triazol-4-yl)acetic acid reacts with 5-phenyl-4-amino-4*H*-1,2,4-triazol-3-thiol affording 6-[(5-methyl-1-phenyl-1*H*-1,2,3-triazol-4-yl) methyl]-3-phenyl[1,2,4] triazolo[3,4-*b*] [1,3,4] thiadiazole (**VI**).

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Derivatives of 1,2,3-triazole is an important class of heterocyclic compounds [1–3]. They are applied as pharmaceutical preparations with cytostatic [4], antiviral [5] and antiproliferative [6] action and as γ -aminobutyric acid antagonists [7, 8]. They are used as intermediates at the synthesis of antibiotics [9, 10, 11], antigistamine preparations [12] and neuroleptics [13], muscarin antagonists [14], nucleozides [15, 16], rotaxanes [17] and chemiluminescent compounds [18, 19]. The derivatives of 1,2,3-triazole also are applied as insecticides [20], fungicides [21], plant growth regulators [22, 23], corrosion inhibitors [24] and photostabilizers [25]. Therefore in the last two decades the chemistry of 1,2,3-triazole derivatives was developed intensively. Many publications are denoted to the development of new synthetic methods and improving existing ones [1–3]. Much less were studied transformations of functionalized 1,2,3-triazoles. The triazole ring is known as enough labile and can exert both thermal and photochemical destruction [26]. Therefore chemical behavior of compounds that include triazole fragment remains unknown for many reactions.

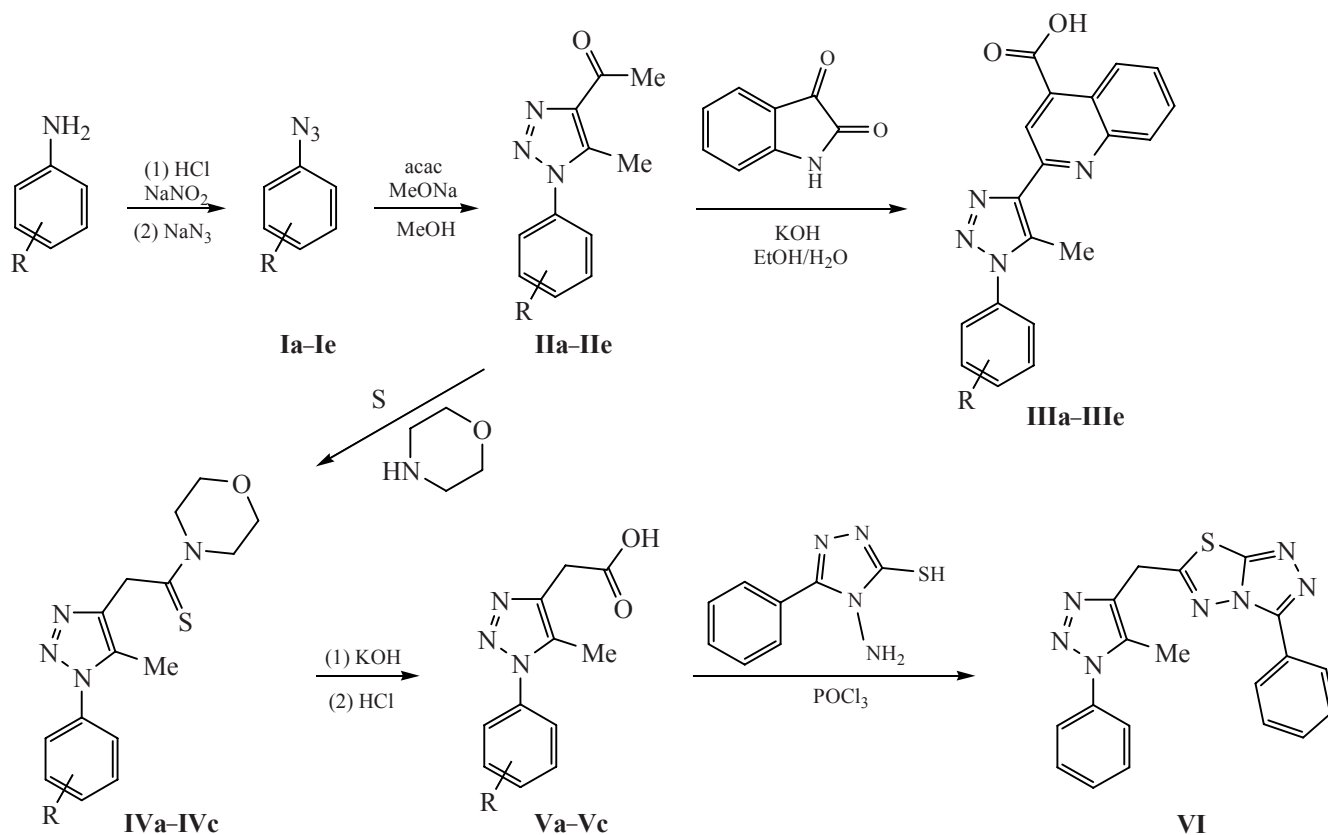
In this study we investigated possibility of application of 1-(5-methyl-1-aryl-1*H*-1,2,3-triazol-4-yl)ethanones (**IIa–IIe**) and 1-[4-(4-*R*-5-methyl-1*H*-1,2,3-triazol-1-yl)phenyl] ethanones (**VIIa–VIIe**) in some reactions that proceed under rigid conditions.

In the reaction of arylazides **Ia–Ie** with acetylacetone we prepared a series of 1-(5-methyl-1-aryl-1*H*-1,2,3-triazol-4-yl)ethanones **IIa–IIe**. We found that 1,2,3-triazole derivatives **IIa–IIe** are formed in a high yield when this reaction is conducted at room temperature in methanol with sodium methylate as a base. Reaction is completed in a week, yield of compounds **IIa–IIe** was 37–77%. Attempted acceleration of this reaction by heating led to tarring of the reaction mixture. With softer bases and at reflux in methanol the degree of conversion of the parent arylazides **Ia–Ie** also diminished.

Note that only restricted data on the reaction of arylazides with acetylacetone has been published [27–30]. The procedure developed by us makes available substituted triazoles **IIa–IIe** that can be used as reagents for further application.

We used 4-acetyltriazaoles **IIa–IIe** in the syntheses of quinoline derivatives along Pfitzinger reaction and of 1,2,3-triazolylacetic acids by the method of Wilgerodt–Kindler. We found that compounds **IIa–IIe** readily react with isatin in strong alkaline medium with construction of quinoline ring. Yields of substituted 4-quinolinecarboxylic acids **IIIa–IIIe** achieve 78–90%, except that of compound **IIIb**, and reaction is fast due to significant acceptor properties of triazole ring that activates acetyl fragment.

Ketones **IIa–IIe** also readily react with sulfur and morpholine under the conditions of Wilgerodt–Kindler reaction affording triazolylacetic acid trimorpholides **IVa–IVc** in high yield (78–86%). By hydrolysis of the latter we obtained triazolylacetic acids **Va–Vc** which can be used as reagents for the creation of combinatory libraries. On one example (compound **VI**) we showed that acids **V** react with 4-amino-1,2,4-triazol-3-thiols to form [1, 2, 4] triazolo[3,4-*b*] [1,3,4] thiadiazoles [31–33].



Using procedures described in [34] we synthesized 1-aryl-1*H*-1,2,3-triazolylethanones **VII** and **VIII** containing acetyl group in the aryl ring. We found that these compounds also react with isatin, the result of this reaction is formation of 4-quinolinecarboxylic acids **IXa**, **IXb** in high yield.

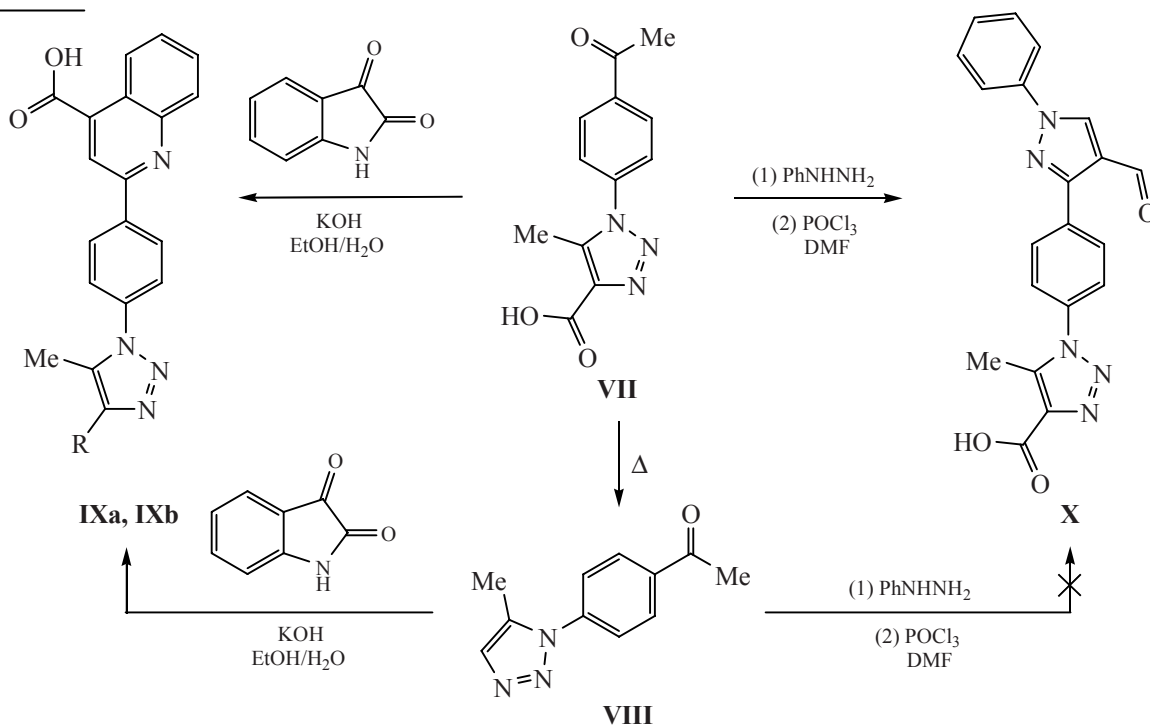
At the study of ketone phenylhydrazones **VII** and **VIII** in the syntheses of pyrazoles was found that at the action of Wilsmeier–Haak complex the final pyrazole **X** is formed only when the parent ketone **VII** contains carboxy group in 4 position of triazole ring.

When a decarboxylated ketone phenylhydrazone **VIII** is used occurs tarring that is explainable by the lability of hydrogen atom in 4 position.

Thus, we elaborated general methodology for the synthesis in high yield of 1,2,3-triazole derivatives by cycloaddition of arylazides to acetylacetone. We showed that 1-(5-methyl-1-aryl-1*H*-1,2,3-triazol-4-yl)-ethanones **IIa–IIe** readily react with isatin in strong alkaline medium, therewith are formed 2-(1-aryl-5-methyl-1*H*-1,2,3-triazol-4-yl)-4-quinolinecarboxylic acids **IIIa–IIIe**. We also elaborated a procedure for the

synthesis of [5-methyl-1-(R-phenyl)-1*H*-1,2,3-triazol-4-yl] acetic acids **Va–Vc**. We studied transformation of 1-[4-(4-R-5-methyl-1*H*-1,2,3-triazol-1-yl)phenyl]-

ethanones **VII** and **VIII** into the derivatives of 4-quinolinecarboxylic acids **IXa** and **IXb** and pyrazolecarbaldehyde **X**.



EXPERIMENTAL

The ^1H NMR spectra of the synthesized compounds were registered on a Varian Mercury 400 instrument with operating frequency 400 MHz, solvent $\text{DMSO}-d_6$, internal reference TMS.

Compounds **Ia–Ie** and 1-(4-azidophenyl)ethanone for the synthesis of compound **VII** are prepared along the procedures in [35], yields 46–84% on the parent anilines: **Ia**, yield 63%, bp 52–53°C (10 mm Hg); **Ib**, yield 46%, bp 62°C (10 mm Hg); **Ic**, yield 67%, bp 78°C (10 mm Hg); **Id**, yield 70%, bp 70°C (10 mm Hg); **Ie**, yield 84%, mp 21–22°C; 1-(4-azidophenyl)ethanone, yield 76%, mp 46–48°C [34].

1-(5-Methyl-1-aryl-1*H*-1,2,3-triazol-4-yl)ethanones (IIa–IIe). A solution of sodium methoxide is prepared from 3 g of sodium and 70 ml of methanol. To the solution at vigorous stirring was added 10 g of acetylacetone and 0.1 mol of arylazide. The mixture was stirred at room temperature until precipitate was formed. The precipitate was filtered off, to the filtrate was added a few milliliters of water and additional

amount of product was extracted. The product was purified by recrystallization from aqueous ethanol. Compound **IIa**, yield 58%, mp 99–100°C; **IIb**, yield 37%, oily substance; **IIc**, yield 67%, mp 74–75°C; **IId**, yield 74%, mp 108–109°C; **IIe**, yield 77%, mp 93–94°C.

Compounds **VII** and **VIII** were prepared along the procedures in [34] and recrystallized from ethanol: **VII**, yield 85%, mp 198–199°C; **VIII**, yield 63%, mp 113–114°C.

Synthesis of cynchonic acids IIIa–IIIe and IXa, IXb. At heating 1.47 g of isatin was dissolved in 25 ml of 8 M solution of KOH, then 0.1 mol of ketone **II**, **VII** or **VIII** was added and ethanol was added until the mixture became homogenous. The mixture was boiled under reflux for 2 h, then cooled and 10 ml of water was added to it, then it was acidified with AcOH to pH $\approx$ 6–7 and filtered. The products were recrystallized from ethanol.

2-(1-Phenyl-5-methyl-1*H*-1,2,3-triazol-4-yl)-4-quinolinecarboxylic acid (IIIa). Yield 88%, mp 294–

295°C. The ^1H NMR spectrum, δ , ppm: 2.88 s (3H, Me), 7.59–7.69 m (6H, Ph + 6-H quinoline), 7.75 t (1H, 7-H quinoline, $^3J = 7.6$ Hz), 8.07 d (1H, 5-H quinoline, $^3J = 8.4$ Hz), 8.79 s (1H, 3-H quinoline), 8.80 d (1H, 8-H quinoline, $^3J = 8.4$ Hz), 13.55 br.s (1H, COOH). Found, %: C 68.91; H 4.14; N 16.84. $\text{C}_{19}\text{H}_{14}\text{N}_4\text{O}_2$. Calculated, %: C 69.08; H 4.27; N 16.96.

2-[1-(2-Methylphenyl)-5-methyl-1*H*-1,2,3-triazol-4-yl]-4-quinolinecarboxylic acid (IIIb). Yield 52%, mp >300°C. The ^1H NMR spectrum, δ , ppm: 2.09 s (3H, Me), 2.68 s (3H, Me), 7.39–7.58 m (4H, C_6H_4), 7.63 pseudo-t (1H, 6-H quinoline), 7.77 pseudo-t (1H, 7-H quinoline), 8.07 d (1H, 5-H quinoline, $^3J = 8.4$ Hz), 8.78 d (1H, 8-H quinoline, $^3J = 8.4$ Hz), 8.79 s (1H, 3-H quinoline), 13.69 br.s (1H, COOH). Found, %: C 69.87; H 4.61; N 16.18. $\text{C}_{20}\text{H}_{16}\text{N}_4\text{O}_2$. Calculated, %: C 69.76; H 4.68; N 16.27.

2-[1-(3-Methylphenyl)-5-methyl-1*H*-1,2,3-triazol-4-yl]-4-quinolinecarboxylic acid (IIIc). Yield 78%, mp 285–286°C. The ^1H NMR spectrum, δ , ppm: 2.50 s (3H, Me), 2.86 s (3H, Me), 7.39 d (2H, C_6H_4 , $^3J = 7.2$ Hz), 7.44 s (1H, 2-H C_6H_4), 7.51 pseudo-t (1H, 5-H C_6H_4), 7.61 pseudo-t (1H, 6-H quinoline), 7.75 pseudo-t (1H, 7-H quinoline), 8.07 d (1H, 5-H quinoline, $^3J = 8.0$ Hz), 8.78 s (1H, 3-H quinoline), 8.79 d (1H, 8-H quinoline, $^3J = 8.4$ Hz). Found, %: C 69.52; H 4.59; N 16.36. $\text{C}_{20}\text{H}_{16}\text{N}_4\text{O}_2$. Calculated, %: C 69.76; H 4.68; N 16.27.

2-[1-(4-Methylphenyl)-5-methyl-1*H*-1,2,3-triazol-4-yl]-4-quinolinecarboxylic acid (IIId). Yield 83%, mp 262–263°C. The ^1H NMR spectrum, δ , ppm: 2.46 s (3H, Me), 2.87 s (3H, Me), 7.43 d (2H, 3,5-H C_6H_4 , $^3J = 7.2$ Hz), 7.50 d (2H, 2,6-H C_6H_4 , $^3J = 7.2$ Hz), 7.62 pseudo-t (1H, 6-H quinoline), 7.76 pseudo-t (1H, 7-H quinoline), 8.07 d (1H, 5-H quinoline, $^3J = 8.4$ Hz), 8.78 d (1H, 8-H quinoline, $^3J = 8.4$ Hz), 8.79 s (1H, 3-H quinoline). Found, %: C 69.64; H 4.56; N 16.14. $\text{C}_{20}\text{H}_{16}\text{N}_4\text{O}_2$. Calculated, %: C 69.76; H 4.68; N 16.27.

2-[1-(4-Chlorophenyl)-5-methyl-1*H*-1,2,3-triazol-4-yl]-4-quinolinecarboxylic acid (IIIe). Yield 90%, mp 261–262°C. The ^1H NMR spectrum, δ , ppm: 2.88 s (3H, Me), 7.60–7.73 m (5H, C_6H_4 + 6-H quinoline), 7.77 pseudo-t (1H, 7-H quinoline), 8.08 d (1H, 5-H quinoline, $^3J = 8.4$ Hz), 8.77 s (1H, 3-H quinoline), 8.79 d (1H, 8-H quinoline, $^3J = 8.4$ Hz), 13.69 br.s (1H, COOH). Found, %: C 62.70; H 3.45; N 15.31. $\text{C}_{19}\text{H}_{13}\text{ClN}_4\text{O}_2$. Calculated, %: C 62.56; H 3.59; N 15.36.

2-[4-(5-Methyl-1*H*-1,2,3-triazol-1-yl)phenyl]-4-quinolinecarboxylic acid (IXa). Yield 79%, mp 233–234°C. The ^1H NMR spectrum, δ , ppm: 2.45 s (3H, Me), 7.61 s (1H, triazole), 7.67 t (1H, 6-H quinoline, $^3J = 7.6$ Hz), 7.76 d (2H, 3,5-H C_6H_4 , $^3J = 8.8$ Hz), 7.82 t (1H, 7-H quinoline, $^3J = 7.6$ Hz), 8.16 d (1H, 5-H quinoline, $^3J = 8.4$ Hz), 8.51 d (2H, 2,6-H C_6H_4 , $^3J = 8.8$ Hz), 8.53 s (1H, 3-H quinoline), 8.78 d (1H, 8-H quinoline, $^3J = 8.4$ Hz). Found, %: C 68.92; H 4.40; N 16.81. $\text{C}_{19}\text{H}_{14}\text{N}_4\text{O}_2$. Calculated, %: C 69.08; H 4.27; N 16.96.

2-[4-(4-Carboxy-5-methyl-1*H*-1,2,3-triazol-1-yl)-phenyl]-4-quinolinecarboxylic acid (IXb). Yield 83%, mp >300°C. The ^1H NMR spectrum, δ , ppm: 2.46 s (3H, Me), 7.68 pseudo-t (1H, 6-H quinoline), 7.78 d (2H, 3,5-H C_6H_4 , $^3J = 8.8$ Hz), 7.81 pseudo-t (1H, 7-H quinoline), 8.16 d (1H, 5-H quinoline, $^3J = 8.4$ Hz), 8.54 d (2H, 2,6-H C_6H_4 , $^3J = 8.8$ Hz), 8.54 s (1H, 3-H quinoline), 8.79 d (1H, 8-H quinoline, $^3J = 8.4$ Hz). Found, %: C 64.08; H 3.54; N 14.85. $\text{C}_{20}\text{H}_{14}\text{N}_4\text{O}_4$. Calculated, %: C 64.17; H 3.77; N 14.97.

Synthesis of [5-methyl-1-aryl-1*H*-1,2,3-triazol-4-yl]-1-(morpholin-4-yl)ethanethiones (Wilgerodt–Kindler reaction) (IVa–IVc). A mixture of 0.1 mol of ketone **II**, 6.4 g of sulfur and 17.4 g of morpholine was heated for 5 h in oil bath (the bath temperature 135°C) with reflux condenser. Warm mixture was carefully poured into 50 ml of hot ethanol and triturated to the beginning of crystallization. The mixture was left overnight in a freezer, then the product was filtered off, washed with little amount of cold ethanol and dried in air. **IVa**, yield 78%, mp 148–149°C; **IVb**, yield 83%, mp 140–141°C. **IVc**, yield 86%, mp 149–150°C.

Synthesis of (5-methyl-1-aryl-1*H*-1,2,3-triazol-4-yl)acetic acids (Va–Vc). To a mixture of 80 g of 50% aqueous solution of KOH and 140 ml of ethanol was added 0.1 mol of crude thiomorpholide **IVa–IVc**. The mixture was refluxed for 6 h and then poured to water and acidified. The solution was cooled, the precipitate formed was filtered off. The acid was recrystallized from aqueous ethanol.

(5-Methyl-1-phenyl-1*H*-1,2,3-triazol-4-yl)acetic acid (Va). Yield 61%, mp 149–150°C. The ^1H NMR spectrum δ , ppm: 2.28 s (3H, Me), 3.73 s (2H, CH_2), 7.57–7.68 m (5H, Ph), 12.63 br.s (1H, COOH). Found, %: C 60.69; H 5.23; N 19.42. $\text{C}_{11}\text{H}_{11}\text{N}_3\text{O}_2$. Calculated, %: C 60.82; H 5.10; N 19.34.

[5-Methyl-1-(4-methylphenyl)-1H-1,2,3-triazol-4-yl]acetic acid (Vb). Yield 65%, mp 196–197°C. The ^1H NMR spectrum, δ , ppm: 2.26 s (3H, Me), 2.44 s (3H, Me), 3.63 s (2H, CH_2), 7.36 d (2H, 3,5-H C_6H_4 , $^3J = 8.4$ Hz), 7.39 d (2H, 2,6-H C_6H_4 , $^3J = 8.4$ Hz), 12.40 br.s (1H, COOH). Found, %: C 62.19; H 5.52; N 18.11. $\text{C}_{12}\text{H}_{13}\text{N}_3\text{O}_2$. Calculated, %: C 62.33; H 5.67; N 18.17.

[5-Methyl-1-(4-chlorophenyl)-1H-1,2,3-triazol-4-yl]-acetic acid (Vc). Yield 74%, mp 170–171°C. The ^1H NMR spectrum, δ , ppm: 2.29 s (3H, Me), 3.65 s (2H, CH_2), 7.60 s (4H, C_6H_4), 12.63 s (1H, COOH). Found, %: C 52.32; H 4.18; N 16.58. $\text{C}_{11}\text{H}_{10}\text{ClN}_3\text{O}_2$. Calculated, %: C 52.50; H 4.01; N 16.70.

6-[(5-Methyl-1-phenyl-1H-1,2,3-triazol-4-yl)methyl]-3-phenyl[1,2,4]triazolo[3,4-b][1,3,4]thiadiazole (VI). To a mixture of 0.96 g of 5-phenyl-4-amino-4H-1,2,4-triazol-3-thiol and 1.1 g of acid (Va) was added 10 ml of POCl_3 . The reaction mixture was refluxed until HCl evolution ceases and then was cooled to room temperature. The obtained viscous mass was added in small portion to a mixture of 20 g of NaOH, 50 ml of water and 50 g of ice at external cooling. The reaction mixture was kept at room temperature for 0.5 h, then alkalinized (if necessary) with 2 M solution of NaOH to pH 8. Precipitate was filtered off, washed on the filter with 2 M solution of NaOH (up to 15 ml) and with warm water (up to 500 ml), dried in air and recrystallized. When necessary, at the recrystallization was added active carbon. Yield 80%, mp 182–183°C. The ^1H NMR spectrum, δ , ppm: 2.43 s (3H, Me), 4.64 s (2H, CH_2), 7.48–7.64 m (8H, Ph), 8.25 d (2,6-H Ph, $^3J = 8.0$ Hz). Found, %: C 61.17; H 3.90; N 26.33. $\text{C}_{19}\text{H}_{15}\text{N}_7\text{S}$. Calculated, %: C 61.11; H 4.05; N 26.26.

Synthesis of 1-[4-(4-formyl-1-phenyl-1H-pyrazol-3-yl)phenyl]-5-methyl-1H-1,2,3-triazol-4-carboxylic acid (X). A mixture of 4.9 g of ketone VIIa and 2.2 g of phenylhydrazine in ethanol was heated until precipitate formed. The precipitate was filtered off and dried. To 2.58 g of dimethylformamide cooled to 0°C was added dropwise 5.4 g of POCl_3 at the temperature maintained below 5°C. At the same temperature was added a solution of 3.9 g of hydrazone in 7 ml of DMFA. The mixture was heated to room temperature and kept for 1 h, then it was heated to 70–80°C and kept at this temperature for 3 h. After cooling to room temperature the mixture was poured to saturated solution of K_2CO_3 cooled with ice. The residue formed was filtered off, washed with water and recrystallized from ethanol. Yield 58%, mp >300°C. The ^1H NMR

spectrum, δ , ppm: 2.62 s (3H, Me), 7.40 pseudo-t (1H, 4-H Ph), 7.55 pseudo-t (2H, 3,5-H Ph), 7.71 d (2H, 3,5-H C_6H_4 , $^3J = 8.4$ Hz), 8.01 d (2H, 2,6-H Ph, $^3J = 8.0$ Hz), 8.25 d (2H, 2,6-H C_6H_4 , $^3J = 8.4$ Hz), 9.40 s (1H, CHO), 10.06 s (1H, pyrazole). Found, %: C 64.20; H 4.21; N 18.64. $\text{C}_{20}\text{H}_{15}\text{N}_5\text{O}_3$. Calculated, %: C 64.34; H 4.05; N 18.76.

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