Synthesis of New Azocompounds and Fused Pyrazolo[5,1-*c*][1,2,4] triazines Using Heterocyclic Components

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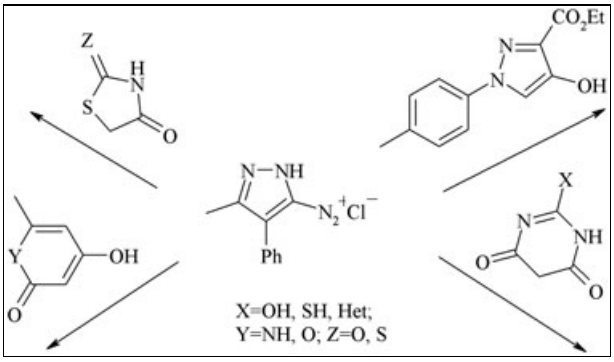

Diazotization of 3-methyl-4-phenyl-1*H*-pyrazol-5-amine **1** in hydrochloric acid has been reported to afford the corresponding diazonium salt **2**. The latter underwent azocoupling with a variety of active methylene compounds (barbituric **3a** and thiobarbituric **3b** acid, 2-hetarylpyrimidine-4,6-dione **6a**,**b**, 4-hydroxy-6-methylpyridin-2(1*H*)-one **10a**, 4-hydroxy-6-methyl-2*H*-pyran-2-one **10b**, 4-hydroxy-1-*p*-tolyl-1*H*-pyrazole-3-carboxylic acid ethyl ester **14**, 1,3-thiazolidine-2,4-dione **16a**, 2-thioxo-1,3-thiazolidin-4-one **16b**) to yield new pyrazolylazo derivatives. Fused pyrazolo[5,1-*c*][1,2,4]triazines **5**, **9a**,**b**, **12**, **13** were obtained by heterocyclization reactions.

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INTRODUCTION

Pyrazolediazonium salts are known as potential building blocks for the synthesis of various heterocycles [1–6]. The pyrazole-annelated nitrogen-containing structures can also be referred to them. The pyrazolo[5,1-*c*][1,2,4]triazines are the most important among them. The considerable biological activity of these heterocycles as purine analogs stimulated interest in the synthesis of derivatives of these ring systems [7–12].

A direct procedure for obtaining pyrazolo[5,1-*c*][1,2,4] triazines consists in azocoupling of pyrazole-3(5)-diazonium salts with aliphatic compounds containing an active methylene/methine followed by the cyclization of formed hydrazones or azocompounds [13–18].

Nevertheless, there are only a few examples in which the heterocyclic rings [19–21] are used as coupling components in reactions with pyrazole-3(5)-diazonium salts.

In the previous article [22], we reported that the reaction of pyrazolediazonium salts with alicyclic methylene active reagents led to pyrazol-5-ylhydrazones, which spontaneously cyclized to substituted pyrazolo[5,1-*c*] [1,2,4]triazines.

In the present work, we investigated the synthetic routes to new azocompounds and tricyclic pyrazolo[5,1-*c*][1,2,4] triazines using different heterocyclic components in the azocoupling reaction with pyrazolediazonium salts.

RESULTS AND DISCUSSION

3-Methyl-4-phenyl-1*H*-pyrazol-5-amine **1** reacted with sodium nitrite and hydrochloric acid to give the corresponding pyrazole-3(5)-diazonium chloride **2**. The latter was isolated in pure form, and further, it was treated *in situ* (Scheme 1).

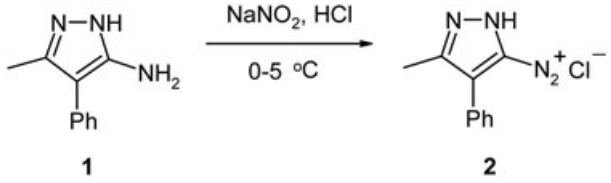
In the present study, the synthetic potential of compounds **2** was investigated through its reactions with heterocyclic coupling components.

Thus, diazonium salt **2** reacted with barbituric acid **3a** and thiobarbituric acid **3b** under the coupling conditions (Scheme 2). For all interactions of this type, several tautomeric forms of the products can be written. Spectral data showed that linear derivatives exist in an azo tautomeric form.

According to the ^{1}H NMR spectrum, there were no signals in the range of δ 11–12 ppm relating to NH-protons of hydrazone form, as reported in the literature [23]. Besides, the analysis showed the hydroxyl group near δ 14.5 ppm and the signals of NH-protons of pyrazole and pyrimidine rings.

Subsequently, the attempts at heterocyclization of derivatives **4a**,**b** by heating with polyphosphoric acid (PPA) for a few hours have been made. As a result, only a new pyrazolo[5,1-*c*]pyrimido[4,5-*e*][1,2,4]triazine-4(3*H*)-one **5** was obtained. Unfortunately, the cyclization of hetarylazocompound **4b** both by heating with PPA and anhydrous sodium acetate in acetic acid failed.

Scheme 1



1 **2**

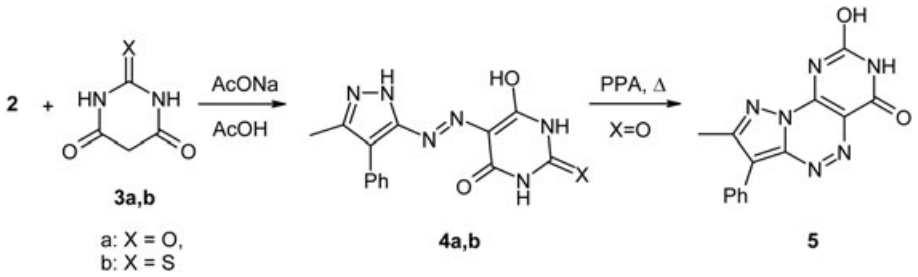
Similarly, diazonium salt **2** coupled smoothly with 2-hetarylpyrimidine-4,6-diones **6a**,**b** in the presence of sodium acetate to yield the colored products (Scheme 3). ^{1}H NMR data of the compounds obtained showed the tautomeric forms **7a**,**b** only.

When the substances obtained were heated with PPA, 8-methyl-7-phenyl-2-hetaryl-pyrazolo[5,1-*c*]pyrimido[4,5-*e*][1,2,4]triazin-4(3*H*)-ones **9a**,**b** were formed in satisfactory yields (65–70%). Alternative structures **8a**,**b** were excepted on the basis of spectral data. The ^{1}H NMR spectra of **9a**,**b** showed the presence of amide NH-proton signal (δ 11.80 ppm) of pyrimidine cycle and the absence of the analogous signal of pyrazole ring in a low field.

Azocoupling of 4-hydroxy-6-methyl-1*H*-pyridine-2-one **10a** and 4-hydroxy-6-methyl-2*H*-pyran-2-one (triacetic acid) **10b** with diazotized 3(5)-aminopyrazole resulted in the formation of the pyrazolo[5,1-*c*][1,2,4]triazine derivatives that were probably formed via intermediacy of the azo form **11a**,**b** (Scheme 4).

In the case of compound **11a**, the heterocyclization was performed by heating the colored linear substrate with acetic acid in ring-closure product **12**. Such procedure is usable as a novel route to obtain the pyrazolo[5,1-*c*]pyrido[4,3-*e*][1,2,4]triazine derivative [24]. Differently, cyclocondensation of intermediate **11b** did not lead to the expected tricyclic structure but gave a new 7-methyl-4-(2-oxopropyl)-8-phenylpyrazolo[5,1-c][1,2,4]triazine-3-carboxylic acid **13**. Probably, this was due to the lactone ring opening under the action of water in acid medium (Scheme 5).

Scheme 2

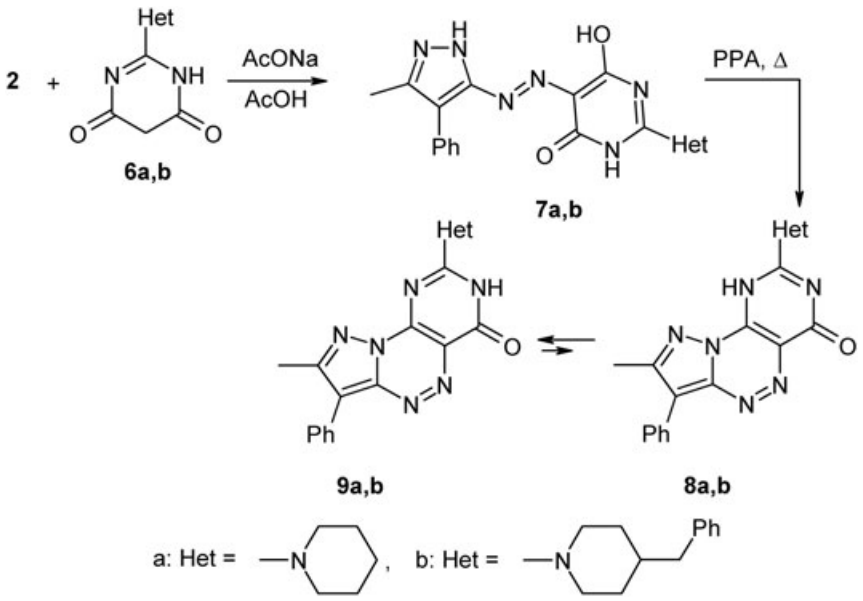


3a,b

a: X = O,
b: X = S

4a,b **5**

Scheme 3



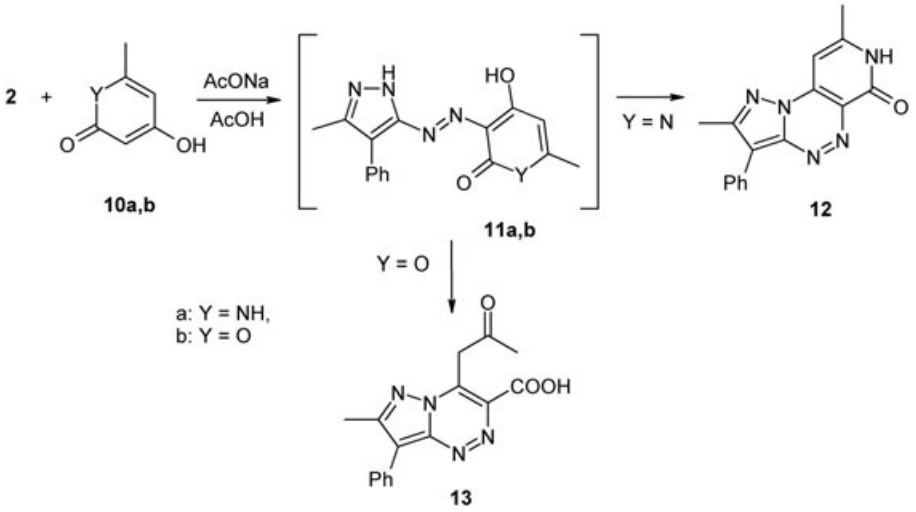
6a,b

7a,b

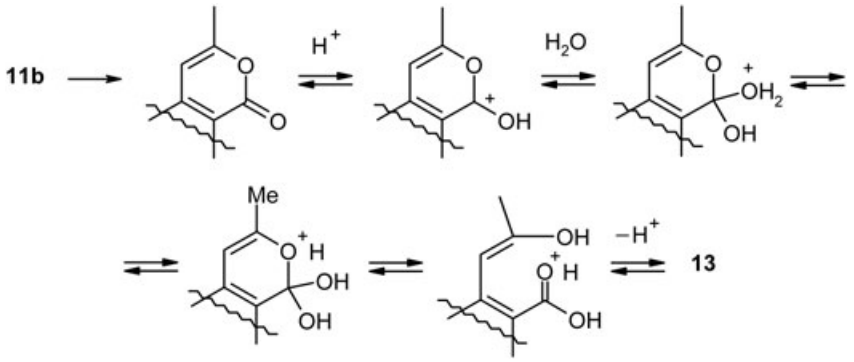
9a,b **8a,b**

a: Het = , b: Het =

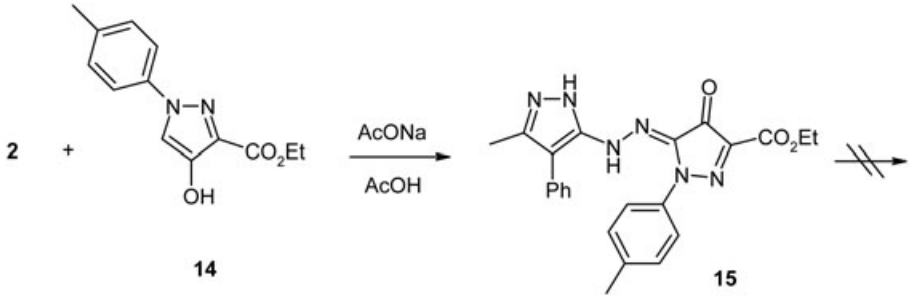
Scheme 4



Scheme 5



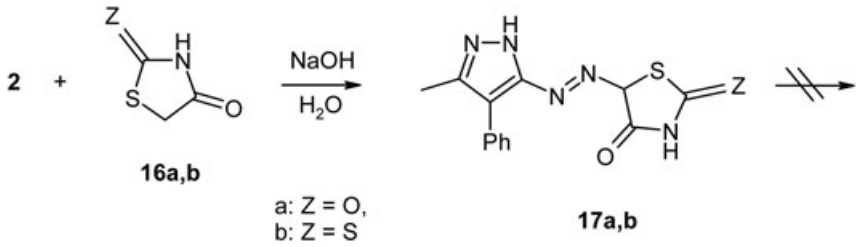
Scheme 6



Moreover, the interaction of pyrazolediazonium salt **2** with 4-hydroxy-1-*p*-tolyl-1*H*-pyrazole-3-carboxylic acid ethyl ester **14** yielded a linear bright red hetarylhydrazone **15** (Scheme 6). The spectral data were in good agreement with the suggested structure. From the NMR data, the broad signals of proton in the region of δ 9.50 ppm proved the presence of the linear C-NH-N═C fragment. The IR spectrum revealed a strong absorption band at 1650 cm^{-1} for conjugated ketone. Neither heating of **15** with acetic acid nor heating with PPA led to a new azoheterocycle. This is, obviously, connected with the hindered nucleophilic attack at C(4)-position in pyrazole ring.

In addition, the synthetic possibilities of 1,3-thiazolidine-2,4-dione and 2-thioxo-1,3-thiazolidin-4-one (rhodanin) as

Scheme 7



methylene active components in reactions with diazonium salt were considered. The aforementioned heterocycles were found to be incorporated into natural subtracts and medicines [25–28]. Therefore, the inclusion of these compounds in synthetic polyheterocyclic systems made it possible to model many potential drugs.

Azocoupling of pyrazole-(3)5-diazonium chloride **2** with active methylene compounds **16a**,**b** yielded colored linear products **17a**,**b**. On the basis of literature data, the tautomeric forms of these compounds have been defined as azo-keto forms [29–31]. The attempts at heterocyclization of azocompounds **17a**,**b** to pyrazolotriazine-containing structures either by refluxing in acetic acid or by heating with PPA for a few hours were unsuccessful because of the ring destruction (Scheme 7).

CONCLUSION

Thus, the convincing examples of an effective synthesis of earlier unknown pyrazol-5-yl-azocompounds and tricyclic pyrazolo[5,1-*c*][1,2,4]triazines have demonstrated the prospects for using different heterocycles as coupling components in the reaction with pyrazolediazonium salts. In addition, pyrazole-containing azocompounds can also find application in industrial azo dyes and analytical indicators.

Finally, many pyrazolo[5,1-*c*][1,2,4]triazine derivatives have been used as potential bioactive compounds because they are structurally related to purines. The aforementioned discussion makes it possible to consider all systems obtained significant for the future research in the area of heterocyclic and medicinal chemistry.

EXPERIMENTAL

General procedures. Melting points were determined with a Kofler apparatus (Heizbank, Reichert, Austria). Reaction progress was monitored by an analytical thin-layer chromatography on plates Silufol UV-254 (Merck, Darmstadt, Germany) using the solvent systems of chloroform, ethyl acetate, and isopropyl alcohol. The products were visualized with UV light. Infrared spectrum was determined using a Specord-82 M spectrometer, and the wave numbers are expressed in cm^{-1}. The ^1H NMR spectra were recorded on Bruker AC-300 spectrometer (Bruker, Flawil, Switzerland) (300 MHz) relative to $SiMe_4$ as an internal standard (DMSO-d_6 solutions). Mass spectra were measured on an LKB-9000 spectrometer (Shimadzu, Tokyo, Japan). Elemental analyses (C, H, N) were conducted using the elemental analyzer Carlo Erba NA 1500 (Heraeus, Hanau, Germany). Starting materials **3a**,**b**, **10b**, and **16a**,**b** were commercially available.

2-Hetarylpyrimidine-4,6-diones 6a,**b**. A typical procedure for the preparation of pyrimidine-4,6-dione derivatives was described in paper [32].

4-Hydroxy-6-methyl-1*H*-pyridine-2-one 10a. The synthetic procedure for obtaining **10a** has been reported previously [33].

4-Hydroxy-1-*p*-tolyl-1*H*-pyrazole-3-carboxylic acid ethyl ester 14. Compound **14** was prepared according to the literature procedure [34].

Preparation of compounds 4a,**b**, **7a**,**b**, **12**, **13**, **15**. A solution of 3(5)-aminopyrazole [35] **1** (5.19 g, 0.03 mol) in water (30 mL) and hydrochloric acid (9 mL, $d = 1.19\ g \cdot mL^{-1}$) was cooled to 0°C. The mixture was treated with the water solution of sodium nitrite (2.07 g, 0.03 mol) by stirring. Then, the cooled (0–5°C) pyrazolediazonium chloride **2** solution was added with stirring to the active methylene compounds **3a**,**b**, **6a**,**b**, **10a**,**b**, **14** (0.03 mol) with sodium acetate (30 g, 0.37 mol) in acetic medium (30 mL). The reaction mixture was stirred at room temperature for nearly 1 h and kept standing overnight. The solid deposit was collected by filtration and washed well with water. All colored products were recrystallized from acetic acid.

Synthesis of tricyclic pyrazolo[5,1-*c*][1,2,4]triazines 5, 9a,**b**. Dried up and crushed crystalline pyrazolylazo compounds **4a**, **7a**,**b** were heated for 3 h with two-multiple excess of solid PPA. The temperature of the reaction mixture was kept between 140 and 160°C. Then, water was added to the cooled reaction mixture, and the latter was neutralized with sodium bicarbonate. The solid deposit was collected by filtration, washed with water, and recrystallized from acetic acid, DMF, or the mixtures of them.

6-Hydroxy-5-[(3-methyl-4-phenyl-1H-pyrazol-5-yl)diazenyl] pyrimidine-2,4(1H,3H)-dione 4a. Yield 93%; yellow crystals (from AcOH); mp >300°C; ^1H NMR δ 2.28 (3H, s, Me), 7.33 (3H, m, H_{Ar}), 7.45 (2H, m, H_{Ar}), 12.38, 12.55 (2H, both br, NH), 12.85 (1H, br, $NH_{pyrazole}$), 14.49 (s, 1H, OH). ms, *m*/*z* 312 $[M]^+$. *Anal*. Calcd for $C_{14}H_{12}N_6O_3$ (*M* 312.28): C, 53.85; H, 3.87; N, 26.91. Found: C, 53.92; H, 3.83; N, 26.88.

6-Hydroxy-5-[(3-methyl-4-phenyl-1H-pyrazol-5-yl)diazenyl]-2-thioxo-2,3-dihydropyrimidin-4(1H)-one 4b. Yield 88%; yellow-red crystal solid (from AcOH); mp 190–192°C; ^1H NMR δ 2.30 (3H, s, Me), 7.10–7.30 (5H, m, H_{Ar}), 12.38, 12.49 (2H, both br, NH), 12.80 (1H, br, $NH_{pyrazole}$), 14.62 (1H, s, OH). MS, *m*/*z* 328 $[M]^+$. *Anal.* Calcd for $C_{14}H_{12}N_6O_2S$ (*M* 328.35):C, 51.21; H, 3.68; N, 25.59. Found: C, 51.30; H, 3.55; N, 25.61.

2-Hydroxy-8-methyl-7-phenylpyrazolo[5,1-c]pyrimido[4,5-e][1,2,4]triazin-4(3H)-one 5. Yield 67%; yellow crystal solid (from AcOH–DMF); mp >300°C; ^{1}H NMR δ 2.69 (3H, s, Me), 7.50 (3H, s, H_{Ar}), 7.88 (2H, s, H_{Ar}), 11.79 (1H, br, NH). ms, *m/z* 294 $[M]^+$. *Anal*. Calcd for $C_{14}H_{10}N_6O_2$ (*M* 294.27): C, 57.14; H, 3.43; N, 28.56. Found: C, 57.39; H, 3.29; N, 28.42.

6-Hydroxy-5-[(3-methyl-4-phenyl-1H-pyrazol-5-yl)diazenyl]-2-piperidin-1-ylpyrimidin-4(3H)-one 7a. Yield 87%; bright yellow crystal solid (from AcOH); mp 182–184°C; ^{1}H NMR δ 1.70 (6H, s, $(CH_2)_3$), 2.33 (3H, s, Me), 3.91 (4H, s, $N(CH_2)_2$), 7.30 (1H, s, H_{Ar}), 7.60 (4H, m, H_{Ar}), 11.56 (1H, br, NH), 12.80 (1H, br, $NH_{pyrazole}$), 16.00 (1H, br, OH). ms, *m/z* 379 $[M]^+$. *Anal*. Calcd for $C_{19}H_{21}N_7O_2$ (*M* 379.42): C, 60.15; H, 5.58; N, 25.84. Found: C, 60.25; H, 5.51; N, 25.80.

2-(4-Benzylpiperidin-1-yl)-6-hydroxy-5-[(3-methyl-4-phenyl-1H-pyrazol-5-yl)diazenyl]pyrimidin-4(3H)-one 7b. Yield 79%; bright yellow crystal solid (from AcOH); mp 198–200°C; ^{1}H NMR δ 1.26 (2H, m, CH_2), 1.75 (2H, m, CH_2), 1.90 (1H, m, CH), 2.31 (3H, s, Me), 2.56 (2H, d, J = 13.0 Hz, CH_2), 4.60 (4H, m, $N(CH_2)_2$), 7.10–7.50 (10H, m, H_{Ar}), 11.60 (1H, br, NH), 12.89 (1H, br, $NH_{pyrazole}$), 16.10 (1H, br, OH). ms, *m/z* 469 $[M]^+$. *Anal*. Calcd for $C_{26}H_{27}N_7O_2$ (*M* 469.54): C, 66.51; H, 5.80; N, 20.88. Found: C, 66.50; H, 5.74; N, 20.90.

8-Methyl-7-phenyl-2-piperidin-1-ylpyrazolo[5,1-c]pyrimido[4,5-e][1,2,4]triazin-4(3H)-one 9a. Yield 65%; brown crystals (from AcOH–DMF); mp >300°C; ^{1}H NMR δ 1.73 (6H, s, $(CH_2)_3$), 2.69 (3H, s, Me), 3.96 (4H, s, $N(CH_2)_2$), 7.39 (1H, s, H_{Ar}), 7.50 (2H, m, H_{Ar}), 7.86 (2H, m, H_{Ar}), 11.81 (1H, br, NH). ms, *m/z* 361 $[M]^+$. *Anal*. Calcd for $C_{19}H_{19}N_7O$ (*M* 361.40): C, 63.14; H, 5.30; N, 27.13. Found: C, 63.20; H, 5.40; N, 27.19.

2-(4-Benzylpiperidin-1-yl)-8-methyl-7-phenylpyrazolo[5,1-c]pyrimido[4,5-e][1,2,4]triazin-4(3H)-one 9b. Yield 50%; brown crystals (from AcOH–DMF); mp >300°C; ^{1}H NMR δ 1.30 (2H, m, CH_2), 1.76 (2H, m, CH_2), 1.90 (1H, m, CH), 2.70 (3H, s, Me), 2.85 (2H, m, CH_2), 4.23 (4H, s, $N(CH_2)_2$), 7.25–7.80 (10H, m, H_{Ar}), 11.80 (1H, br, NH). ms, *m/z* 451 $[M]^+$. *Anal*. Calcd for $C_{26}H_{25}N_7O$ (*M* 451.52): C, 69.16; H, 5.58; N, 21.71. Found: C, 69.22; H, 5.55; N, 21.70.

2,8-Dimethyl-3-phenylpyrazolo[5,1-c]pyrido[4,3-e][1,2,4]triazin-6(7H)-one 12. Yield 45%; brown crystals (from AcOH–DMF); mp >300°C; ^{1}H NMR δ 2.43 (3H, s, Me), 2.67 (3H, s, Me), 6.94 (1H, c, CH), 7.44 (1H, t, J = 7.4 Hz, H_{Ar}), 7.57 (2H, t, J = 7.6 Hz, H_{Ar}), 7.85 (2H, d, J = 7.6 Hz, H_{Ar}), 12.39 (1H, br, NH). ms, *m/z* 291 $[M]^+$. *Anal*. Calcd for $C_{16}H_{13}N_5O$ (*M* 291.31): C, 65.97; H, 4.50; N, 24.04. Found: 66.06; H, 4.41; N, 24.00.

7-Methyl-4-(2-oxopropyl)-8-phenylpyrazolo[5,1-c][1,2,4]triazine-3-carboxylic acid 13. Yield 49%; orange crystals (from AcOH); mp 187°C, decomp.; ^{1}H NMR δ 1.92, 2.59 (6H, both s, Me), 3.05 (1H, d, J = 8.9 Hz, CH_2), 3.24 (1H, d, J = 8.9 Hz, CH_2), 7.43 (1H, t, J = 7.0 Hz, H_{Ar}), 7.55 (2H, t, J = 7.8 Hz, H_{Ar}), 7.78 (2H, d, J = 7.7 Hz, H_{Ar}), 8.28 (1H, s, COOH). ms, *m/z* 310 $[M]^+$. *Anal*. Calcd for $C_{16}H_{14}N_4O_3$ (*M* 310.31): C, 61.93; H, 4.55; N, 18.06. Found: 62.10; H, 4.46; N, 17.98.

Ethyl 1-(p-tolyl)-5-[(3-methyl-4-phenyl-1H-pyrazol-5-yl)hydrazono]-4-oxo-4,5-dihydro-1H-pyrazole-3-carboxylate 15. Yield 66%; orange-red crystals (from AcOH); mp 167–169°C, decomp.; ir 1608, 1650 (C=O), 2961 (C-H_{alip}). ^{1}H NMR δ 1.38 (3H, t, J = 2.8 Hz, $MeCH_2O$), 2.34 (3H, s, Me), 2.40 (3H, s, Me), 4.35 (2H, q, J = 5.0 Hz, $MeCH_2O$), 7.10–7.60 (9H, m, H_{Ar}), 9.50 (1H, br, NH), 13.20 (1H, br, $NH_{pyrazole}$). ms, *m/z* 430 $[M]^+$. *Anal.* Calcd for $C_{23}H_{22}N_6O_3$ (*M* 430.46): C, 64.17; H, 5.15; N, 19.52. Found: C, 64.28; H, 5.10; N, 19.48.

Preparation of thiazolidine derivatives 17a,b. Azothiazolidines **17a,b** were obtained according to the procedure described for the analogous aryl derivatives [36].

A solution of **16a,b** (0.03 mol in 30 mL water) was mixed with 2N aqueous NaOH (15 mL). The cooled solution was added dropwise with stirring to a solution of the appropriate pyrazole-diazonium chloride (prepared from 5.19 g, 0.03 mol of the amine **1** and the appropriate quantities of hydrochloric acid and sodium nitrite). After addition, the mixture was stirred for 40 min at 0°C and then adjusted to pH ~3 with aqueous HCl. The precipitate was filtered, washed with water, and dried.

5-[(3-Methyl-4-phenyl-1H-pyrazol-5-yl)diazenyl]-1,3-thiazolidine-2,4-dione 17a. Yield 62%; yellow crystal solid; mp 188°C, decomp.; ^{1}H NMR δ 2.32 (3H, s, Me), 7.18–7.50 (5H, m, H_{Ar}), 7.63 (1H, s, SCH), 12.46 (1H, br, NH), 13.22 (1H, br, $NH_{pyrazole}$). *Anal*. Calcd for $C_{13}H_{11}N_5O_2S$: C, 51.82; H, 3.68; N, 23.24. Found: C, 51.73; H, 3.60; N, 23.21.

5-[(3-Methyl-4-phenyl-1H-pyrazol-5-yl)diazenyl]-2-thioxo-1,3-thiazolidin-4-one 17b. Yield 44%; orange crystal solid; mp 150°C, decomp.; ^{1}H NMR δ 2.31 (3H, s, Me), 7.20–7.45 (5H, m, H_{Ar}), 7.51 (1H, s, SCH), 10.63 (1H, br, NH), 13.40 (1H, br, $NH_{pyrazole}$). *Anal*. Calcd for $C_{13}H_{11}N_5OS_2$: C, 49.19; H, 3.49; N, 22.07. Found: C, 49.29; H, 3.40; N, 22.00.

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