Synthesis of *peri*-annelated heterocyclic systems based on 3-substituted 1-aryl-4,6-dinitro-1*H*-indazoles.

*A. M. Starosotnikov, A. V. Lobach, V. M. Vinogradov, and S. A. Shevelev**

N. D. Zelinsky Institute of Organic Chemistry, Russian Academy of Sciences, 47 Leninsky prosp., 119991 Moscow, Russian Federation. Fax: +7 (095) 135 5328. E-mail: shevelev@mail.ioc.ac.ru

A method for the synthesis of *peri*-annelated trinuclear heterocycles, including 14π-electron heteroaromatic systems, namely, 1*H*-thiopyrano[4,3,2-*cd*]indazoles and 1,5-dihydropyrazolo[3,4,5-*de*]cinnolines, from 3-R-1-aryl-4,6-dinitro-1*H*-indazoles was developed. The method is based on the high mobility of the NO_2 group in position 4 and consists of either selective nucleophilic substitution of the 4-NO_2 group on treatment with the $HSCH_2CO_2Me$—K_2CO_3 system followed by intramolecular cyclization of the resulting sulfide (R = CHO) or the corresponding sulfone (R = CN) formed upon its oxidation or direct intramolecular substitution of the 4-NO_2 group (R = CH=NNHPh).

Key words: 4,6-dinitro-1*H*-indazoles, nucleophilic substitution, cyclization, *peri*-annelated trinuclear heterocycles, 14π-electron heteroaromatic systems, thiopyranoindazoles, dihydropyrazolocinnolines.

Previously, we developed methods for the synthesis of 3-R-1-aryl-4,6-dinitro-1*H*-indazoles (R = CHO (**1**)[1,2] and CN (**2**)[2]) starting from 2,4,6-trinitrotoluene (TNT). It was found that the nitro group in position 4 of 4,6-dinitroindazoles **1** and **2**, *i.e.*, the group closest to the heterocyclic nucleus, is selectively substituted under mild conditions on treatment with anionic S-, O-, and N-nucleophiles.[1,2] In this study, we employed the high mobility of the 4-NO_2-group in 4,6-dinitroindazoles **1** and **2** to synthesize *peri*-annelated heterocyclic systems.

We have found that 1-aryl-3-formyl-4,6-dinitro-1*H*-indazoles (**1**) can be used to prepare 14π-electron *peri*-annelated tricyclic heteroaromatic systems (for the preliminary communication, see Ref. 3). Thus the reaction of 3-formyl-4,6-dinitroindazoles **1a,b** with methyl thioglycolate in *N*-methylpyrrolidone (NMP) or DMF in the presence of solid K_2CO_3 gives the corresponding *peri*-annelated tricyclic heteroaromatic compounds, namely, methyl 1-aryl-7-nitro-1*H*-thiopyrano[4,3,2-*cd*]indazole-4-carboxylates **3a,b** (Scheme 1). Presumably, first, the 4-NO_2 group is substituted on treatment with the thioglycolate—K_2CO_3 system to give intermediate **A**. The formyl group in **A** undergoes base-catalyzed intramolecular condensation with the active methylene fragment of the SCH_2CO_2Me substituent.

Under standard conditions, 1-aryl-3-formyl-4,6-dinitroindazoles **1a,b** are converted into phenylhydrazones **4a,b**, which undergo intramolecular cyclization with replacement of the 4-NO_2 group on treatment with K_2CO_3 in NMP or DMF yielding the corresponding *peri*-annelated aromatic heterocycles, namely, 1,5-diaryl-7-nitro-1,5-dihydropyrazolo[3,4,5-*de*]cinnolines **5a,b**. It can be assumed that the reaction proceeds *via* preliminary deprotonation of phenylhydrazones **4a,b** to give N-anions **B** (see Scheme 1).

Thus, starting from 3-formyl-4,6-dinitroindazoles **1**, we synthesized representatives of two types of 14π-electron *peri*-annelated tricyclic heteroaromatic systems, namely, 1*H*-thiopyrano[4,3,2-*cd*]indazoles and 1,5-dihydropyrazolo[3,4,5-*de*]cinnolines.

Note that 14π-electron *peri*-annelated heteroaromatic systems consisting of two six-membered and one five-membered rings represent a relatively uncommon type of heterocycles (see Refs. 4, 5). As regards the tricyclic systems obtained in this work, we found no publications describing their synthesis; only a collection of reports[6] contains data on pharmacological properties of some 1*H*-thiopyrano[4,3,2-*cd*]indazoles with substituents other that those considered in our study, but no synthetic procedures are reported.

We studied some transformations of thiopyranoindazoles **3** using tricyclic derivative **3a** as an example. Compound **3a** is easily and selectively oxidized on treatment with aqueous H_2O_2 in CF_3COOH to afford the corresponding sulfone **6**; the same reaction in AcOH results in sulfoxide **7** (Scheme 2). Alkaline hydrolysis of **3a** leads to the corresponding tricyclic carboxylic acid **8**.

Previously we reported[2] that the 4-NO_2 group in 3-cyano-4,6-dinitro-1-phenyl-1*H*-indazole (**2a**) is replaced in the reaction with methyl thioglycolate in the

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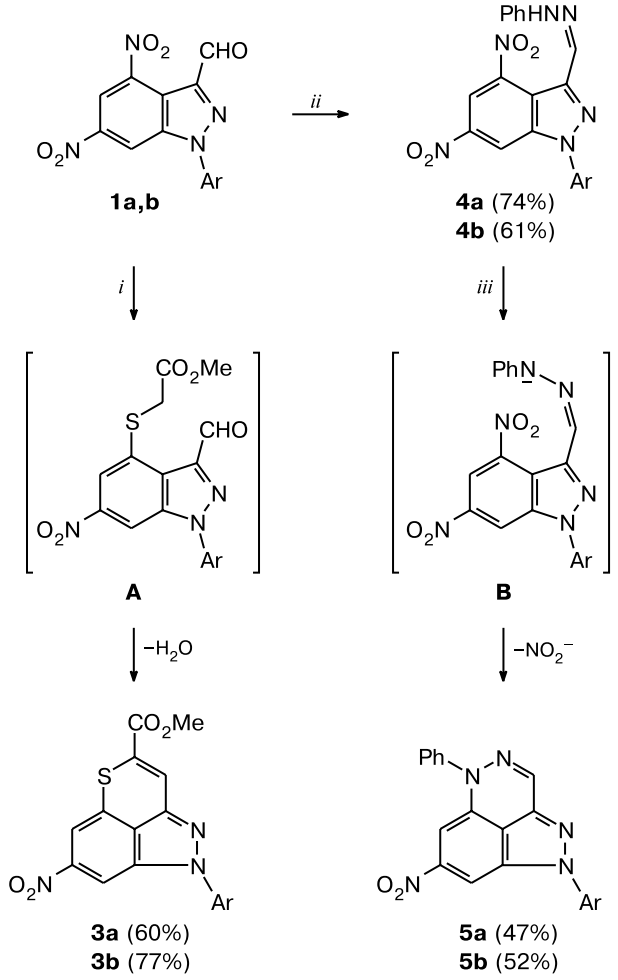
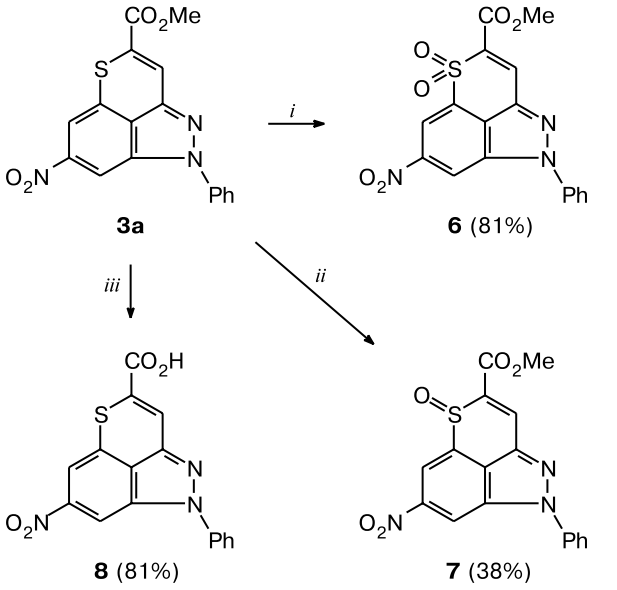
Scheme 1



Reagents and conditions: *i*. 1 equiv. $HSCH_2CO_2CH_3$, 2 equiv. K_2CO_3, NMP, 60 °C, 10 h; *ii*. 1 equiv. $PhNHNH_2 \cdot HCl$, EtOH, 78 °C, 3 h; *iii*. 1 equiv. K_2CO_3, NMP, 80 °C, 10 h.

Scheme 2



Reagents and conditions: *i*. 35% H_2O_2 (8 equiv.), CF_3COOH, 20 °C, 0.5 h; *ii*. 35% H_2O_2 (120 equiv.), AcOH, 60 °C, 12 h; *iii*. 10 equiv. NaOH, H_2O, 100 °C, 20 h.

presence of K_2CO_3, giving rise to the corresponding sulfide **9**. An attempt to perform cyclization of sulfide **9** through the addition of the active methylene bridge to the CN bond (in the presence of bases) to give tricyclic compound **10** (Scheme 3) failed, most likely, because of the insufficient mobility of the hydrogen atoms in the methylene fragment of sulfide **9**. In order to obtain a compound with more reactive methylene bridge, sulfide **9** was oxidized with an aqueous H_2O_2—CF_3COOH mixture to the corresponding sulfone **11**. Easy cyclization of this sulfone on treatment with K_2CO_3 in NMP gave *peri*-annelated tricyclic amino derivative **12**.

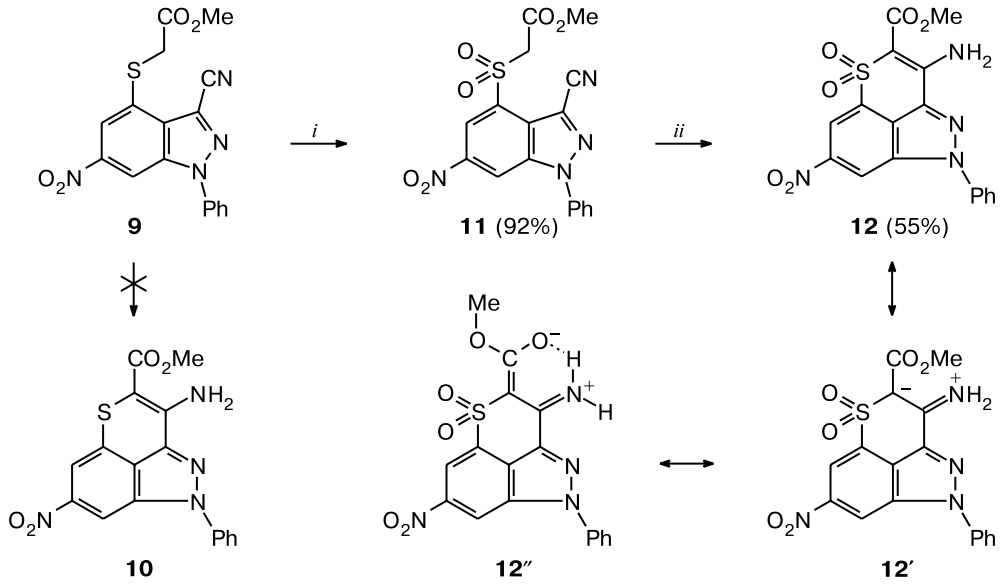
It should be noted that the position of the carbonyl absorption band in the IR spectrum of derivative **12** (1668 cm^{-1}) is unusual for an ester group. The ^1H NMR spectrum of this compound exhibits a very strong downfield shift of the NH_2 signal (9.0 ppm). These data indicate a sizable contribution of structures **12´** and **12″** (see Scheme 3). Indeed, such spectral data are quite inherent in this type of push-pull compounds. For example, in the IR spectrum of derivatives of 3-amino-2-nitroacrylic esters, the absorption bands due to the ester group are located in the 1650—1670 cm^{-1} region and the chemical shifts of the NH_2 protons reach 9.7 ppm (see Ref. 7).

It might be expected that 3-formyl-4,6-dinitro-1-phenyl-1*H*-indazole oxime (**13**)[2] can also be used to obtain a tricyclic system. In particular, under the action of a base, the arising oximate anion will enter into intramolecular replacement of the 4-NO_2 group to form tricyclic compound **14**. However, on treatment with K_2CO_3 in NMP, oxime **13** is converted into 3-cyano-4-hydroxy-6-nitro-1-phenyl-1*H*-indazole (**15**) (Scheme 4).

In our opinion, tricyclic product **14** is formed initially during the reaction but undergoes oxazine ring opening under the action of a base present, being thus converted into nitrile **15**. This type of β-cleavage, —ON=CH— → —OH + N≡C—, induced by bases is well known for 3-unsubstituted benzo[*d*]isoxazoles, which are converted into 2-hydroxybenzonitriles (see Refs. 8, 9), and for *O*-arylaldoximes containing electron-withdrawing substitutes in the *O*-aryl fragment, which give rise to phenols and alkyl(aryl)nitriles (see Ref. 10).

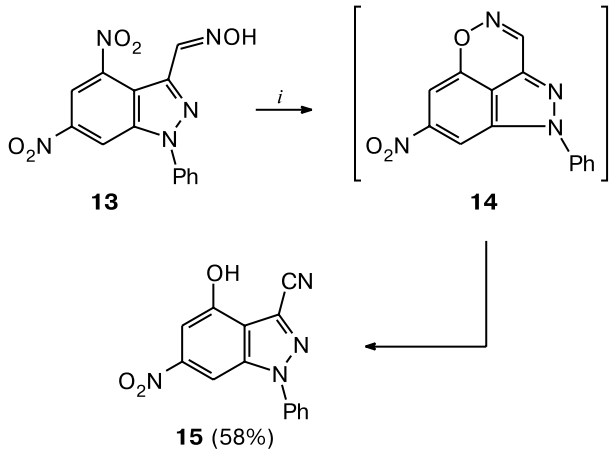
Thus, based on 1-aryl-3-formyl(3-cyano)-4,6-dinitro-1*H*-indazoles and making use of the high mobility of 4-NO_2 group, one can obtain *peri*-annelated trinuclear

Scheme 3



Reagents and conditions: *i*. H_2O_2, CF_3COOH, 20 °C; *ii*. K_2CO_3, NMP, 80 °C.

Scheme 4



i. K_2CO_3, NMP, 80 °C.

heterocycles, including representatives of new 14π-electron heteroaromatic systems.

The structures and compositions of the compounds obtained were proved by ^{1}H NMR data, mass spectra (the molecular ion is observed in all cases), IR spectra, and elemental analysis.

Experimental

^{1}H and ^{13}C NMR spectra were recorded on Bruker AC-200 and Bruker AM-300 instruments, respectively. The chemical shifts (δ) are referred to Me_4Si. The spin-spin coupling constants are given in Hz. IR spectra were measured using a Specord M-80 instrument in KBr pellets. Mass spectra (EI, 70 eV) were recorded using a Kratos MS-30 mass spectrometer. The reactions were monitored and the compound purity was checked using TLC on Silufol UV-254 plates. The solvents were not specially dried. Compounds **1**, **9**, and **13** were obtained according to known procedures.[2]

Preparation of thiopyranoindazoles 3a,b (general procedure). Potassium carbonate (0.55 g, 4 mmol) was added to a solution containing 3-formylindazole **1a** or **1b** (2 mmol) and methyl thioglycolate (0.18 mL, 2 mmol) in 7 mL of NMP, and the mixture was stirred for 8 h at 60 °C. The reaction mixture was cooled and poured in water, and the resulting mixture was acidified to pH = 2. The precipitate was filtered off, washed with acetone, and recrystallized from $CHCl_3$.

Methyl 7-nitro-1-phenyl-1*H*-thiopyrano[4,3,2-*cd*]indazole-4-carboxylate (3a). M.p. 230—231 °C ($CHCl_3$). Found (%): C, 57.11; H, 3.39; S, 8.51. $C_{17}H_{11}N_3O_4S$. Calculated (%): C, 57.78; H, 3.14; S, 9.07. ^{1}H NMR ($CDCl_3$), δ: 3.98 (s, 3 H, CH_3); 7.45 (t, 1 H, Ph, $^3J_{H,H}$ = 7.2 Hz); 7.62 (t, 2 H, Ph, $^3J_{H,H}$ = 7.2 Hz); 7.70 (s, 1 H, H arom.); 7.75 (d, 2 H, Ph, $^3J_{H,H}$ = 7.2 Hz); 7.95 (s, 1 H, H arom.); 8.15 (s, 1 H, H arom.). ^{13}C NMR ($CDCl_3$), δ: 53.5, 103.4, 109.0, 121.6, 124.1, 127.5, 127.9, 130.0, 132.3, 132.9, 137.5, 139.2, 143.6, 150.1, 163.0. MS, *m/z*: 353 $[M]^+$, 307 $[M - NO_2]^+$. IR, ν/cm^{-1}: 1720 (CO_2Me), 1540, 1340 (NO_2).

Methyl 1-(4-chlorophenyl)-7-nitro-1*H*-thiopyrano[4,3,2-*cd*]indazole-4-carboxylate (3b). M.p. >300 °C ($CHCl_3$). Found (%): C, 52.69; Cl, 9.05; H, 2.85; S, 8.18. $C_{17}H_{10}ClN_3O_4S$. Calculated (%): C, 52.65; Cl, 9.14; H, 2.60; S, 8.27. ^{1}H NMR ($CDCl_3$), δ: 3.95 (s, 3 H, CH_3); 7.55 (d, 2 H, H arom., $^3J_{H,H}$ = 8.8 Hz); 7.60 (d, 3 H, H arom., $^3J_{H,H}$ = 8.8 Hz); 7.95 (s, 1 H, H arom.); 8.09 (s, 1 H, H arom.). MS, *m/z*: 387 $[M]^+$, 341 $[M - NO_2]^+$. IR, ν/cm^{-1}: 1712 (CO_2Me), 1520, 1348 (NO_2).

Preparation of 3-formylindazole *N*-phenylhydrazones 4a,b (general procedure). $PhNHNH_2 \cdot HCl$ (0.93 g, 6.4 mmol) was added to a suspension of compound **1a** or **1b** (6.4 mmol) in 30 mL of EtOH and the mixture was refluxed for 3 h. The reaction mixture was cooled and the precipitate was filtered off, washed with EtOH, and dried at 80 °C.

4,6-Dinitro-1-phenyl-1*H*-indazole-3-carboxaldehyde *N*-phenylhydrazone (4a). M.p. 260—261 °C (EtOH). ^{1}H NMR (DMSO-d_6), δ: 6.80 (t, 1 H, Ph, $^3J_{H,H}$ = 7.3 Hz); 7.00 (d, 2 H, Ph, $^3J_{H,H}$ = 7.3 Hz); 7.20 (t, 2 H, Ph, $^3J_{H,H}$ = 7.3 Hz); 7.60 (t, 1 H, Ph, $^3J_{H,H}$ = 7.9 Hz); 7.70 (t, 2 H, Ph, $^3J_{H,H}$ = 7.8 Hz); 7.85 (d, 2 H, Ph, $^3J_{H,H}$ = 7.9 Hz); 8.20 (s, 1 H, CH=N); 8.55 (s, 1 H, H arom.); 8.75 (s, 1 H, H arom.); 10.60 (s, 1 H, NH).

1-(4-Chlorophenyl)-4,6-dinitro-1*H*-indazole-3-carboxaldehyde *N*-phenylhydrazone (4b). M.p. 267—269 °C (EtOH). ^{1}H NMR (DMSO-d_6), δ: 6.88 (m, 1 H, Ph); 7.10—7.35 (m, 4 H, Ph); 7.64 (s, 1 H, CH=N); 7.78 (d, 2 H, H arom., $^3J_{H,H}$ = 8.0 Hz); 8.05 (d, 2 H, H arom., $^3J_{H,H}$ = 8.0 Hz); 8.75 (s, 1 H, H arom.); 8.90 (s, 1 H, H arom.); 11.75 (s, 1 H, NH).

Synthesis of pyrazolocinnolines 5a,b (general procedure). Potassium carbonate (0.21 g, 1.5 mmol) was added to a solution of compound **4a** or **4b** (1.5 mmol) in 10 mL of NMP and the mixture was stirred for 8 h at 90 °C. Then the reaction mixture was cooled and poured in water and the precipitate was filtered off, washed with acetone, and dried in air.

7-Nitro-1,5-diphenyl-1,5-dihydropyrazolo[3,4,5-*de*]cinnoline (5a). M.p. 257—259 °C. Found (%): C, 66.91; H, 3.95. $C_{20}H_{13}N_5O_2$. Calculated (%): C, 67.60; H, 3.69. ^{1}H NMR ($CDCl_3$), δ: 7.12 (s, 1 H, H arom.); 7.40—7.55 (m, 2 H, Ph); 7.55—7.65 (m, 6 H, Ph); 7.80 (d, 2 H, Ph, $^3J_{H,H}$ = 7.2); 7.92 (s, 1 H, H arom.); 8.09 (s, 1 H, CH=N). MS, *m/z*: 355 $[M]^+$, 309 $[M - NO_2]^+$. IR, ν/cm^{-1}: 1596 (C=N), 1544, 1340 (NO_2).

1-(4-Chlorophenyl)-7-nitro-5-phenyl-1,5-dihydropyrazolo[3,4,5-*de*]cinnoline (5b). M.p. 264—265 °C. Found (%): C, 61.19; Cl, 9.50; H, 3.32. $C_{20}H_{12}ClN_5O_2$. Calculated (%): C, 61.63; Cl, 9.10; H, 3.10. ^{1}H NMR (DMSO-d_6) δ: 6.95 (s, 1 H, H arom.); 7.50 (m, 1 H, Ph); 7.60 (m, 6 H, Ph); 8.90 (m, 3 H, Ph, H arom.); 8.22 (s, 1 H, CH=N). MS, *m/z*: 389 $[M]^+$, 343 $[M - NO_2]^+$. IR, ν/cm^{-1}: 1596 (C=N), 1532, 1340 (NO_2).

Methyl 7-nitro-5,5-dioxo-1-phenyl-1,5-dihydro-5λ^6-thiopyrano[4,3,2-*cd*]indazole-4-carboxylate (6). A 35% solution of H_2O_2 (3 mL) was added at 20 °C to a solution of **2a** (0.45 g, 1.28 mmol) in 20 mL of CF_3COOH and the mixture was stirred for 20 min. The reaction mixture was poured in water and the precipitate was filtered off, dried at 100 °C, and recrystallized from $CHCl_3$ to give 0.4 g of compound **6**. M.p. 218—220 °C ($CHCl_3$). Found (%): C, 52.99; H, 3.06; S, 8.04. $C_{17}H_{11}N_3O_6S$. Calculated (%): C, 52.99; H, 2.88; S, 8.32. ^{1}H NMR (DMSO-d_6) δ: 3.97 (s, 3 H, OCH_3); 7.54—7.76 (m, 3 H, Ph); 7.91 (d, 2 H, Ph, $^3J_{H,H}$ = 7.9 Hz); 8.55 (s, 1 H, CH); 8.75 (s, 1 H, H arom.); 8.91 (s, 1 H, H arom.). MS, *m/z*: 385 $[M]^+$, 308 $[M - Ph]^+$. IR, ν/cm^{-1}: 1720 (CO_2Me); 1544, 1344 (NO_2); 1320 (SO_2).

Methyl 7-nitro-5-oxo-1-phenyl-1,5-dihydro-5λ^4-thiopyrano[4,3,2-*cd*]indazole-4-carboxylate (7). A 35% solution of H_2O_2 (5×3 mL, every 2 h) was added at 60 °C to **2a** (0.45 g, 1.28 mmol) in 20 mL of AcOH. After cooling the reaction mixture, the precipitate was filtered off, washed with water, dried at 100 °C, and recrystallized from $CHCl_3$ to give 0.13 g of compound **7**. M.p. 224—225 °C ($CHCl_3$). ^{1}H NMR (DMSO-d_6), δ: 3.98 (s, 3 H, OCH_3); 7.60—7.80 (m, 3 H, Ph); 7.87 (d, 2 H, Ph, $^3J_{H,H}$ = 7.3 Hz); 8.05 (s, 1 H, CH); 8.60 (s, 1 H, H arom.); 8.90 (s, 1 H, H arom.). IR, ν/cm^{-1}: 1728 (CO_2Me), 1536, 1336 (NO_2), 1084 (S=O).

7-Nitro-1-phenyl-1*H*-thiopyrano[4,3,2-*cd*]indazole-4-carboxylic acid (8). A mixture of **2a** (0.18 g, 0.5 mmol), NaOH (0.2 g, 5 mmol), and 15 mL of water was refluxed for 24 h. After cooling, the reaction mixture was acidified to pH = 2 and the precipitate was filtered off, washed with water, dried, and recrystallized from $CHCl_3$ to give 0.14 g of compound **8**. M.p. 303—304 °C ($CHCl_3$). ^{1}H NMR (DMSO-d_6), δ: 7.45 (t, 1 H, Ph, $^3J_{H,H}$ = 6.7 Hz); 7.63 (t, 2 H, Ph, $^3J_{H,H}$ = 7.3 Hz); 7.80 (m, 3 H, Ph, CH); 8.08 (s, 1 H, H arom.); 8.25 (s, 1 H, H arom.). ^{13}C NMR (DMSO-d_6), δ: 103.2, 109.1, 121.4, 127.4, 130.1, 133.8, 137.2, 139.3, 144.2, 150.1, 163.9. MS, *m/z*: 339 $[M]^+$, 293 $[M - NO_2]^+$. IR, ν/cm^{-1}: 1700 (CO_2H), 1530, 1350 (NO_2).

Methyl 2-[(3-cyano-6-nitro-1-phenyl-1*H*-indazol-4-yl)sulfonyl]acetate (11). A 35% solution of H_2O_2 (1 mL) was added at 20 °C to a solution of compound **9** (0.4 g, 1.1 mmol) in 8 mL of CF_3COOH and the mixture was stirred for 1 h. The reaction mixture was poured in water and the precipitate was filtered off, washed with water and EtOH, and dried *in vacuo* to give 0.4 g (92%) of compound **11**. M.p. 185—187 °C. ^{1}H NMR (DMSO-d_6), δ: 3.67 (s, 3 H, OCH_3); 4.98 (s, 2 H, CH_2); 7.60—7.90 (m, 5 H, Ph); 8.70 (s, 1 H, H(5)); 8.90 (s, 1 H, H(7)). ^{13}C NMR (DMSO-d_6), δ: 113.1, 115.5, 120.3, 123.0, 125.3, 130.9, 131.0, 132.8, 137.8, 140.1, 146.9, 163.5. IR, ν/cm^{-1}: 1724 (CO_2Me), 1540, 1340 (NO_2), 1168 (SO_2). MS, *m/z*: 400 $[M]^+$.

Methyl 3-amino-7-nitro-5,5-dioxo-1-phenyl-1,5-dihydro-5λ^6-thiopyrano[4,3,2-*cd*]indazole-4-carboxylate (12). Potassium carbonate (0.21 g, 1.5 mmol) was added to a solution of compound **11** (0.6 g, 1.5 mmol) in 8 mL of NMP and the mixture was stirred at 80 °C for 8 h. After cooling, the reaction mixture was poured in water, the resulting mixture was acidified to pH = 4, and the precipitate was filtered off, thoroughly washed with $CHCl_3$, and dried in air to give 0.33 g (55%) of compound **12**. M.p. >300 °C. Found (%): C, 51.29; H, 2.74; S, 8.04. $C_{17}H_{12}N_4O_6S$. Calculated (%): C, 51.00; H, 3.02; S, 8.01. ^{1}H NMR (DMSO-d_6), δ: 3.90 (s, 3 H, OCH_3); 7.60—7.80 (m, 3 H, Ph); 8.00 (d, 2 H, Ph, $^3J_{H,H}$ = 7.3 Hz); 8.51 (s, 1 H, H(5)); 8.90 (s, 1 H, H(7)); 9.00 (br.s, 2 H, NH_2). IR, ν/cm^{-1}: 1668 (CO_2Me), 1548, 1336 (NO_2), 1260 (SO_2). MS, *m/z*: 400 $[M]^+$, 354 $[M - NO_2]^+$.

3-Cyano-4-hydroxy-6-nitro-1-phenyl-1*H*-indazole (15). Potassium carbonate (0.09 g, 0.6 mmol) was added to a solution of compound **13** (0.2 g, 0.6 mmol) in 6 mL of NMP and the mixture was stirred at 80 to 90 °C for 6 h. The reaction mixture was cooled and poured in water. The resulting mixture was acidified to pH = 2 and extracted with AcOEt (3×15 mL). The solvent was evaporated and the residue was purified by column chromatography (SiO_2/$CHCl_3$). Evaporation of the eluent gave 0.1 g of compound **15**. M.p. 238—240 °C ($CHCl_3$). ^{1}H NMR (DMSO-d_6), δ: 7.48 (s, 1 H, H(5)); 7.50—7.80 (m, 5 H, Ph); 8.01 (s, 1 H, H(7)); 11.90 (br.s, 1 H, OH). MS, *m/z*: 280 $[M]^+$, 234 $[M - NO_2]^+$. IR, ν/cm^{-1}: 2250 (CN), 1660 (C=N), 1540, 1345 (NO_2).

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