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Synthesis of some pyrazolo[3,4-*d*]pyrimidines and their fused triazole and tetrazole derivatives

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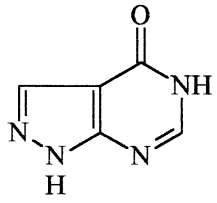
Reaction of **2** with different reagents, namely formic acid, acetic anhydride and trichloroacetonitrile, yielded pyrazolo[3,4-*d*]pyrimidine derivatives **3, 5** and **6,** respectively. Pyrazolo[3,4-*d*]pyrimidine *m*-thiazine(**7**) and 2,4-(1*H*,3*H*)dithione (**8**) derivatives were formed by the action of carbon disulfide on **2**, depending on the reaction medium. Interaction of **7** with hydrazine hydrate yielded the aminoimino derivative **9** which reacted with acetic anhydride, triethyl orthoacetate and/or appropriate aldehydes to give **11, 12** and **13a,b**, respectively. Methylation of compound **8** gave **14**, which reacted with hydrazine hydrate to afford the monohydrazino derivative **15**. Reaction of **15**, with formic acid and nitrous acid afforded pyrazolo[4,3-*e*]triazolo[1,5-*c*]pyrimidine (**16**) and pyrazolo[4,3-*e*]tetrazolo-[1,5-*c*]pyrimidine (**17**) derivatives, respectively. The structures of products **3-17** were identified in light of their elemental analyses and spectra data.

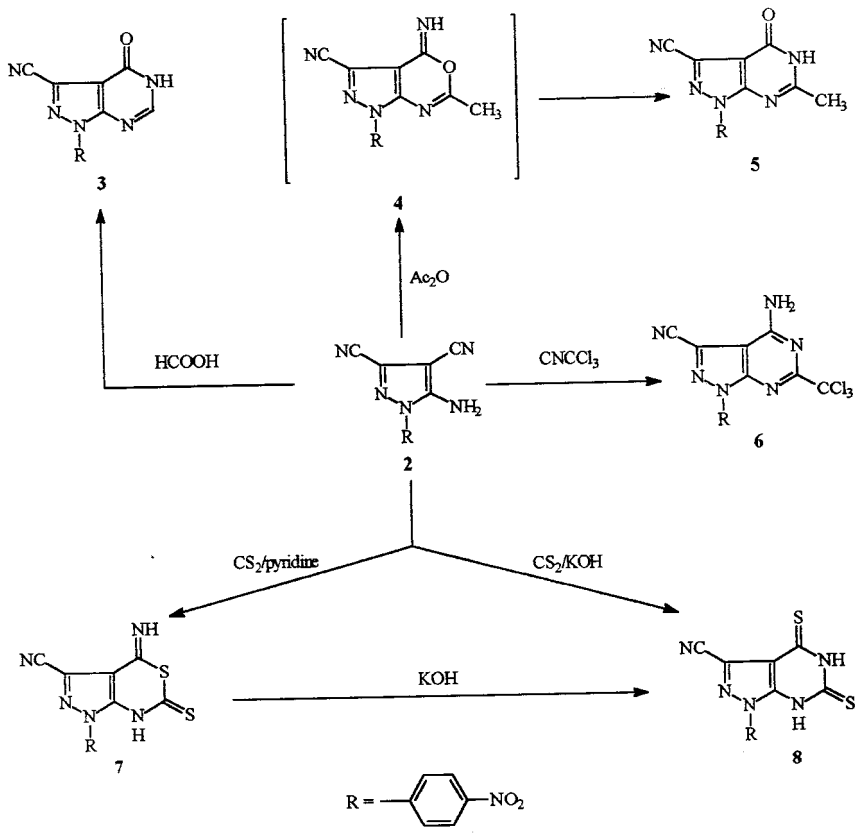
Key words: pyrazolo[3,4-*d*]pyrimidine, *m*-thiazine, rearragnement, triazolo[1,5-*c*]-pyrimidine, tetrazolo[1,5-*c*]pyrimidine.

The best known xanthine oxidase inhibitor is allopurinol (**1**), first synthesized by Robins,[1] and still the drug of choice for treatment of gouty arthritis. Certain pyrazolo[3,4-*d*]pyrimidines exhibit phospho diesterase inhibitory action[2] and others have shown herbicidal activity.[3] These findings encouraged us to undertake the synthesis of some new pyrazolo[3,4-*d*]pyrimidine ring systems, isomeric with various biologically active purine, in the hope that they could be of some promising biological interest.



(1)

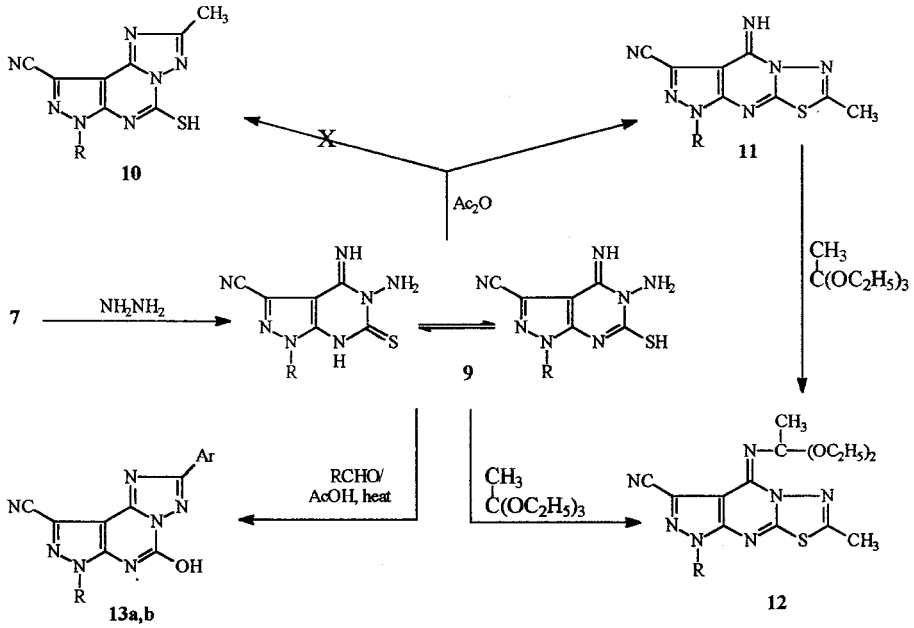
The starting material, 5-amino-3,4-dicyano-1-(4'-nitrophenyl)pyrazole (**2**), used in the present work was prepared by the condensation of *p*-nitrophenylhydrazine with tetracyanoethylene in the presence of piperidine in dioxane as an improvement on the previous method.[4]



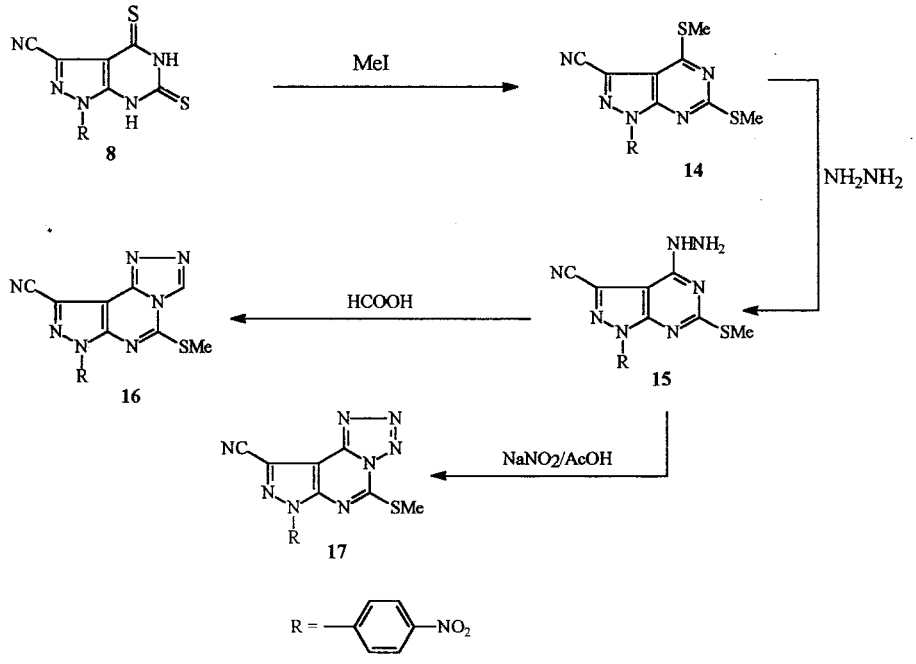
Scheme 1.

Prolonged heating of **2** with formic acid afforded the pyrazolo[3,4-*d*]-pyrimidine-4(3*H*)-one (**3**). The 2-methyl derivative **4** was obtained by refluxing **2** in a mixture of acetic anhydride and acetic acid. A probable route to the formation of **5** is assumed to be the formation of the *N*-acetyl derivative which is converted under the reaction condition to **5** through the *m*-oxazine intermediate **4**. Reaction of **2** with trichloroacetonitrile in refluxing dioxane afforded the 2-trichloromethyl-4-aminopyrazolo[3,4-*d*]pyrimidine derivative **6**.

The obtained products from the reaction of **2** with carbon disulfide varied according to the reaction medium. In pyridine, 5-cyano-4-imino-7-(4'-nitrophenyl)-pyrazolo[3,4-*d*][1,3]thiazine-2-(1*H*)thione (**7**) was formed, however in the presence of aqueous potassium hydroxide, pyrazolo[3,4-*d*]pyrimidine-2,4(1*H*,3*H*)dithione derivative **8** was obtained. Moreover, compound **7** rearranged to product **8** under



a, Ar = C_6H_4-CH_3*p*; b, Ar = furyl-CH_3-2



Scheme 2.

the action of aqueous potassium hydroxide, *via m*-thiazine pyrimidine rearrangement in accordance with previous findings.[5,6]

It was intended in this work to include the synthesis of some new fused pyrazolo[3,4-*d*]pyrimidines. Treatment of the thiazine derivative **7** with hydrazine hydrate at ambient temperature gave the 3-amino-4-iminopyrazolo[3,4-*d*]pyrimidine derivative (**9**). Treatment of the product **9** with acetic anhydride did not afford the expected triazolopyrimidine derivative **10** but a product that was identified as 2-methyl-4-imino-5-cyano-7-(4'-nitrophenyl)pyrazolo[3,4-*d*][1,3,4]-thiadiazolo[3,2-*a*]-pyrimidine (**11**). The ^{1}H-NMR spectrum of compound **11** showed a singlet of an exchangeable proton at δ 7.6 ppm and the IR spectrum band at 3232 cm^{-1} corresponding to the NH group. Moreover, reaction of compound **11** with triethyl orthoacetate yielded product **12** which was also obtained by the reaction of **9** with triethyl orthoacetate in the presence of acetic anhydride. The triazolo[3,2-*c*]pyrimidine derivatives (**13a** and **13b**) were formed as a result of the reaction of the aminoimino derivative **9** with *p*-tolualdehyde or 2-methylfurfural in a refluxing mixture of acetic acid and dioxane, respectively.

The 2,4-dithioxopyrimidine derivative **8** reacted with methyl iodide to produce the dimethylthio derivative **14**. When product **14** was boiled with hydrazine hydrate in dioxane, the 4-hydrazino derivative **15** was obtained. This could be explained by hindrance to nucleophilic attack in the 2-position of the pyrimidine ring.[7] The hydrazino group in the 4-position of the pyrimidine ring in compound **15** was used to produce condensed triazolo- and tetrazolopyrimidines. The indicated compound **15** was condensed with formic acid to afford the pyrazolo[4,3-*e*][1,2,4]triazolo[1,5-*c*]pyrimidine derivative (**16**). The tetrazolo[1,5-*c*]pyrimidine derivative **17** was synthesized by the action of sodium nitrite in acetic acid on compound **15**.

EXPERIMENTAL

Melting points were taken using a Boetius melting point apparatus and were uncorrected. Infrared (IR) spectra were recorded in KBr pellets on a Karl Zeiss IMP 16 spetrophotometer. Nuclear magnetic resonance (^{1}H-NMR) spectra were obtained on a Jeol Ex-270 spectrometer with tetramethylsilane as internal standard. Mass spectra (MS) were measured on a MS 30 (AEL) spectrometer. Elemental analyses (C,H,N and S) were carried out in the microanalytical center, Cairo University, Egypt.

*2-Amino-3,4-dicyano-1-(4'-nitrophenyl)pyrazole (***2***)*

To a solution of tetracyanoethylene (38.5 g; 0.3 mol) and piperidine (3 ml) in dioxane (750 ml), *p*-nitrophenylhydrazine (45.9 g; 0.3 mol) was added in portions, under vigorous stirring, over 1 h. The stirred mixture was left at room temperature overnight, and the formed solid was separated by filtration, recrystallized from dioxane (decolourized with charcoal) to afford **2**, yield: 73 g (96%); m.p.: 249–250 °C [Lit[4], yield: 82%; m.p. 252–253 °C].

5-Cyano-7-(4'-nitrophenyl)pyrazolo[*3,4-*d]*pyrimidin-4-one(***3***)*

A suspension of **2** (1 g; 4 mmol) in formic acid (85%; 20 ml) was heated under reflux for 10 h. A solid product formed on cooling to room temperature, which was filtered off and crystallized from DMF to give **3**; yield: 0.56 g (50%); m.p. 310–311 °C. IR: 2100 (CN), 1630 (CO) cm^{-1}; ^{1}H-NMR(DMSO-d$_6$): δ 13.10 (*s*, 1H, CH–pyridine), 11.2 (*s*, 1H, NH, exchangeable with D$_2$O), 8.4 (*d*,2H, Ph-H), 7.9 (*d*, 2H, Ph–H); MS *m/z* 282 (M$^+$, 100%).

Analysis: calcd. for $C_{12}H_6N_6O_3$: C, 50.07; H, 2.14; N, 29.78%. Found: C, 49.87; H, 2.17; N, 29.93%.

*5-Cyano-7-(4'-nitrophenyl)-2-methylpyrazolo[3,4-*d*]pyrimidin-4-one (***5***)*

A solution of compound **2** (2 g; 8 mmol) in a mixture of acetic anhydride (10 ml) and acetic acid (10 ml) was refluxed for 5 h. The reaction mixture was cooled and the formed solid filtered off, washed with water, dried and crystallized from acetic acid to give **5**, yield: 1.14 g (48%); m.p. 290–291 °C. IR: 2100 (CN), 1650 (CO) cm^{-1}; ^{1}H-NMR (DMSO-d_6): δ 9.5 (*br. s,* 1H, NH, exchangeable with D_2O), 8.4 (*d*, 2H, Ph–H), 7.85 (*d*, 2H, Ph–H), 2.7 (*s*, 3H, CH_3); MS *m/z*: 296 (M^+, 49%).

Analysis: calcd. for $C_{13}H_8N_6O_3$: C, 52.70; H, 2.72; N, 37.14%. Found: C, 52.56; H, 2.82; N, 37.19%.

*4-Amino-5-cyano-7-(4'-nitrophenyl)-2-trichloromethylpyrazolo[3,4-*d*]pyrimidine (***6***)*

A mixture of **2** (0.5 g; 2 mmol), trichloroacetonitrile (0.29 g; 2 mmol) and TEA (4 drops) in dioxane (40 ml) was refluxed for 5 h, then left overnight at room temperature. The excess solvent was distilled off and the residue diluted with water (25 ml). The formed product was filtered off and crystallized from dioxane to afford **6**; yield: 0.48 g (60%); m.p. 240–242 °C. IR: 3480, 3327 (NH_2), 2180 (CN) cm^{-1}; ^{1}H-NMR (DMSO-d_6): δ 8.4 (*d*, 2H, Ph-–H), 7.9 (*d*, 2H, Ph–H), 7.4 (*s*, 2H, NH_2, exchangeable with D_2O); MS *m/z:* 397 (M^+, 7%), 268 (100%).

Analysis: calcd. for $C_{13}H_6N_7O_2Cl_3$: C, 39.16; H, 1.52; N, 24.59%. Found: C, 38.92; H, 1.55; N, 24.74%.

*5-Cyano-4-imino-7-(4'-nitrophenyl)pyrazolo[3,4-*d*]1,3-thiazine-2(1*H*)-thione (***7***)*

To a soluiton of **2** (2 g, 8 mmol) in dry pyridine (20 ml), carbon disulfide (10 ml) was added and the reaction mixture was refluxed for 6 h. After cooling, the mixture was poured onto water, the formed solid was collected by filtration, washed several times with water, dried and recrystallized from dioxane to afford yellow crystals identified as **7**; yield: 2.1 g (80%); m.p. 238–239 °C. IR: 3539 (NH), 3420 (NH), 2120 (CN) cm^{-1}; ^{1}H-NMR (DMSO-d_6): δ 8.4 (*d*, 2H, Ph–H), 7.8 (*d*, 2H, Ph–H), 7.5 (*s*, 2H, 2NH, exchangeable with D_2O); MS *m/z*: 331 (M^++1,3%), 254 (100%).

Analysis: calcd. for $C_{12}H_6N_6O_2S_2$: C, 43.63; H, 1.83; N, 25.44; S, 19.41%. Found: C, 43.40; H, 1.89; N, 25.26; S, 19.58%.

*5-Cyano-7-(4'-nitrophenyl)pyrazolo[3,4-*d*]pyrimidine-2,4-dithione (***8***)*

Method A: To a solution of **2** (2 g, 8 mmol) in 10% alcoholic potassium hydroxide (10 ml), carbon disulfide (10 ml) was added. The reaction mixture was refluxed for 1 h, cooled, poured onto water and neutralized with 1M HCl. The formed solid was filtered off, washed with water, dried and recrystallized from DMF to afford **8** as a yellow powder; yield: 1.23 g (50%); m.p. 297–298 °C. IR: 3435 (NH), 3345 (NH), 2120 (CN) cm^{-1}; ^{1}H-NMR (DMSO-d_6): δ 8.33 (*d*, 2H, Ph–H), 7.96 (*d*, 2H, Ph–H), 7.8 (*s*, 1H, NH, exchangeable with D_2O), 7.59 (*s*, 1H, NH, exchangeable with D_2O); MS *m/z*: 330 (M^+, 5%), 159 (100%).

Analysis: calcd. for $C_{12}H_6N_6O_2S_2$: C, 43.63; H, 1.83; N, 25.44; S, 19.1%. Found: C, 43.42; H, 1.91; N, 25.47; S, 19.38%.

Method B: A solution of **7** (0.33 g, 1 mmol) in 10% alcoholic potassium hydroxide (5 ml) was left under reflux for 1 h. The reaction mixture was cooled, poured onto water, neutralized with 1M HCl to give product **8** as identified by its m.p. and mixed m.p., in addition to its chromatographic behaviour in comparison with an authentic sample from method A.

*3-Amino-4-imino-5,7-(4'-nitrophenyl)pyrazolo[3,4-*d*]pyrimidine-2 (1*H*) thione (***9***)*

A mixture of **6** (6.6 g: 20 mmol), hydrazine hydrate (1.6 g; 30 mmol) in acetic acid (10 ml) was stirred at 0 °C for 2 h. The reaction mixture was poured onto water and the deposited solid was filtered off, washed several times with water to afford **9**; yield: 4.6 g (70%); m.p. 261–262 °C. IR: 3524, 3432,

3365 (NH), 2120 (CN), cm^{-1}; ^{1}H-NMR (DMSO-d_6): δ 8.4 (*d*, 2H, Ph–H), 8.1 (*d*, 2H, Ph–H), 7.6 (*s*, 1H, NH, exchangeable with D_2O), 4.8 (*s*, 2H, NH_2, exchangeable with D_2O), 3.2 (*s*, 1H, SH, exchangeable with D_2O); MS *m/z*: 329 (M+1, 0.62%), 273 (100%).

Analysis: calcd. for $C_{12}H_8N_8O_2S$: C, 43.9; H, 2.46; N, 34.13; S, 9.76%. Found: C, 43.53; H, 2.52; N, 34.24; S, 9.85%.

2-Methyl-4-imino-5-cyano-7-(4'-nitrophenyl)pyrazolo[*3,4*-d][*1,3,4*]-*thiadiazolo*-[*3,2*-a]-*pyrimidine* (**11**)

A mixture of **9** (1.65 g; 5 mmol) in acetic anhydride (10 ml) was refluxed for 6 h. On cooling, a precipitate formed which was filtered off, washed with water, dried and recrystallized from acetic acid to afford **11**; yield: 1.23 g (70%); m.p. 312–313 °C. IR: 3232 (NH), 2120 (CN) cm^{-1}; ^{1}H-NMR (DMSO-d_6): δ 8.3 (*d*, 2H, Ph–H), 7.9 (*d*, 2H, Ph–H), 7.6 (*s*, 1H, NH, exchangeable with D_2O), 2.3 (*s*, 3H, CH_3); MS *m/z*: 352 (M^+, 3%), 279 (100%).

Analysis: calcd. for $C_{14}H_8N_8O_2S$: C, 47.72; H, 2.29; N, 31.81; S, 9.1%. Found: C, 47.53; H, 2.35; N, 31.92; S, 8.94%.

2-Methyl-4-imino-5-cyano-7-(4'-nitrophenyl)pyrazolo[*3,4*-d][*1,3,4*]*thiaziazolo*-[*3,2*-c]-*pyrimidine* (**12**)

Method A: A mixture of **11** (1 g, 3 mmol), triethyl orthoacetate (10 ml) and acetic anhydride (1 ml) was refluxed for 8 h. The formed precipitate was filtered off and recrystallized from acetic acid to afford **12** yield: 0.9 g (65%); m.p. 226–227 °C. IR: 2180 (CN) cm^{-1}; ^{1}H-NMR (DMSO-d_6): δ 8.3 (*d*, 2H, Ph–H), 7.9 (*d*, 2H, Ph–H), 4.35–4.25 (*dq*, 4H, 2×CH_2), 2.4 (*s*, 3H, CH_3), 2.2 (*s*, 3H, CH_3), 1.4–1.3 (*dt*, 6H, 2×CH_3); MS *m/z*: 368 (M^+, 10%), 342 (100%).

Analysis: calcd. for $C_{20}H_{20}N_8O_4S$: C, 51.27; H, 4.3; N, 23.92; S, 6.84%. Found: C, 50.94; H, 4.26; N, 23.85; S, 6.91%.

Method B: A mixture of **9** (0.5 g, 1.5 mmol), triethyl orthoacetate (5 ml) and acetic anhydride (5 ml) was refluxed for 8 h. A precipitate was formed, filtered off and crystallized from acetic acid to give **12;** yield 0.49 g (70%), as identified by TLC, m.p. and mixed m.p., with an authentic sample.

Pyrazolo[*4,3*-e][*1,2,4*]*triazolo*[*3,2*-c]*pyrimidine derivatives* (**13**)

A mixture of **9** (1 g, 3 mmol) and the appropriate aldehyde (3 mmol), namely *p*-tolualdehyde and 5-methylfurfural, in acetic acid (20 ml) was refluxed for 8 h. On cooling, the precipitated solid was filtered off and crystallized from dioxane to afford **13a** and **13b**, respectively.

13a: Yield: 0.9 g (70%); m.p. 289–291 °C. IR: 2140 (CN) cm^{-1}; ^{1}H-NMR (DMSO-d_6): δ 11.7 (*s*, 1H, NH, exchangeable with D_2O), 8.1–7.6 (*m*, 8H, Ph–H), 2.1 (*s*, 3H, CH_3); MS *m/z*: 428 (M^+, 1%), 312 (100%).

Analysis: calcd. for $C_{20}H_{12}N_8O_2S$: C, 56.07; H, 2.82; N, 26.16; S, 7.48%. Found: C, 55.92; H, 2.76; N, 26.25; S. 7.32%.

13b: Yield : 0.95 g (75%); m.p. 279–80 °C. IR: 2135 (CN) cm^{-1}; ^{1}H-NMR (DMSO-d_6): δ 10.5 (*s*, 1H, NH, exchangeable with D_2O), 8.1 (*d*, 2H, Ph–H), 7.8 (*d*, 2H, Ph–H), 7.1 (*s*, 1H, furyl), 6.5 (*s*, 1H, furyl), 2.3 (*s*, 3H, CH_3).

Analysis: calcd. for $C_{18}H_{10}N_8O_3S$: C, 51.67; H, 2.41; N, 26.78; S, 7.66%. Found: C, 51.44; H, 2.36; N, 26.53; S, 7.72%.

5-Cyano-2,4-dimethylthio-7-(4'-nitrophenyl)-pyrazolo[*4,3*-d]-*pyrimidine* (**14**)

To a solution of sodium ethoxide, obtained from sodium (0.46 g, 20 mmol) and absolute ethanol (40 ml), product **8** (3.3 g, 10 mmol) was added. The reaction mixture was stirred for 10 min, then methyl iodide (2.9 g, 20 mmol) was added and the mixture was refluxed for 1 h. After cooling, water (60 ml) was added and the buff yellow solid that formed filtered off, washed with water and dried to give **14**, yield: 3.25 g (80%); m.p. 210–211 °C. IR: 2140 (CN) cm^{-1}; ^{1}H-NMR (DMSO-d_6): δ 8.4 (*d*, 2H, Ph–H), 7.6 (*d*, 2H, Ph–H), 2.8 (*s*, 6H, 2×CH_3); MS *m/z*: 406 (M^+, 18%), 267 (100%).

Analysis: calcd. for $C_{18}H_{10}N_6O_2S_2$: C, 53.19; H, 2.48; N, 20.68; S, 15.78%. Found: C, 53.00; H, 2.46; N, 20.36; S, 15.92%.

5-Cyano-2-methylthio-4-hydrazino-7-(4'-nitrophenyl)pyrazolo[*3,4*-d]*pyrimidine (***15***)*

A mixture of **14** (3 g, 7.5 mmol) and hydrazine hydrate (5 ml, excess) in dioxane (30 ml) was refluxed for 5 h. The formed solid was separated by filtration, washed with water, then methanol and dried to afford **15** as dark green crystals; yield: 1.6 g (62%); m.p. 215–218 °C. IR: 3430, 3370, 3335 (NH), 2160 (CN) cm-1; ^{1}H-NMR (DMSO-d_6): δ 11.2 (*bs*, 1H, NH, exchangeable with D_2O), 8.3 (*d*, 2H, Ph–H), 7.9 (*d*, 2H, Ph–H), 5.4 (*s*, 2H, NH_2, exchangeable with D_2O), 3.25 (*s*, 3H, SCH_3); MS *m/z*: 342 (M^+, 3%), 169 (100%).

Analysis: calcd. for $C_{13}H_{10}N_8O_2S$: C, 45.61; H, 2.94; N, 32.74; S, 9.36%. Found: C, 44.89; H, 2.75; N, 32.98; S, 9.24%.

6-Cyano-2-methylthio-8-(4'-nitrophenyl)pyrazolo[*4,3*-e][*1,2,4*]*triazolo*[*1,5*-c]*-pyrimidine (***16***)*

A mixture of **15** (0.68 g, 2 mmol) and formic acid (10 ml) was refluxed for 5 h. The exceess solvent was distilled off, the residue treated with water (25 ml) and the mixture neutralized with sodium carbonate solution. The separated solid was filtered off, washed with water, dried and crystallized from DMF to give **16** as a pale yelllow powder; yield: 0.5 g (70%); m.p. 250–253 °C. IR: 2180 (CN) cm^{-1}; ^{1}H-NMR (DMSO-d_6): δ 9.1 (*s*, 1H, triazolo-H), 8.2 (*d*, 2H, Ph–H), 7.9 (*d*, 2H, Ph–H), 3.3 (*s*, 3H, SCH_3); MS *m/z*: 352 (M^++H, 0.73%), 159 (100%).

Analysis: calcd. for $C_{14}H_7N_8O_2S$: C, 47.86; H, 2.01; N, 3.19; S, 9.13%. Found: C, 47.38; H, 2.12; N, 32.10; S, 9.21%.

7-Cyano-2-methylthio-9-(4'-nitrophenyl)pyrazolo[*4,3*-e][*1,2,4*]*tetrazolo*[*1,5*-c]*-pyrimidine (***17***)*

To a stirred cold suspension of compound **15** (0.68 g, 2 mmol) in 2M aqueous acetic acid (10 ml), a solution of sodium nitrite (0.2 g; 4 mmol) in water (4 ml) was added dropwise. After stirring for one hour at room temperature, the formed solid was filtered off, washed thoroughly with water and crystallized from DMF to give a pale orange powder identified as **17**; yield: 0.46 g (65%); m.p. 228–230 °C. IR: 2180 (CN) cm^{-1}; ^{1}H-NMR (DMSO-d_6): δ 8.3 (*d*, 2H, Ph–H), 7.9 (*d*, 2H, Ph–H), 3.2 (*s*, 3H, SCH_3); MS *m/z*: 354 (M^+ +H, 0.1%), 159 (100%).

Analysis: calcd. for $C_{13}H_7N_9O_2S$: C, 44.19; H, 1.99; N, 35.68; S, 9.07%. Found: C, 44.12, H, 2.1; N, 35.74; S, 8.96%.

И З В О Д

СИНТЕЗА НЕКИХ ПИРАЗОЛО[3,4-*d*] ПИРИМИДИНА И ЊИХОВИХ ТРИАЗОЛ И ТЕТРАЗОЛ ДЕРИВАТА

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Реаговањем 5-амино-3,4-дицијано-1-(4′-нитрофенил)пиразола (**2**) са мрављом киселином, сирћетним анхидридом или трихлороацетонитрилом добијају се пиразоло [3,4-*d*]пиримидин деривати **3, 5** и **6**. Деривати пиразоло[3,4-*d*]пиримидин *m*-тиазина (**7**) и 2,4-(1*H*,3*H*)дитиона (**8**) настају деловањем угљен-дисулфида на **2**, зависно од реакционе средине. Интеракција **7** са хидразин хидратом даје аминоимино дериват **9** који реагујући са сирћетним анхидридом, триетил ортоацетатом или/и одговарајућим алдехидом даје **11, 12** или **13a,b**. Метилација једињења **8** даје једињење **14**, које реакцијом са хидразин хидратом ствара монохидразино дериват **15**. Реаговањем једињења **15** са мрављом киселином и азотастом киселином добијају се деривати пиразоло[4,3-*e*]триазоло[1,5-

с]пиримидина (**16**) и пиразоло [4,3-*e*]тетразоло[1,5-*c*]пиримидина (**17**). Структура производа **3** – **17** одређена је на основу елементалне анализе и спектралних података.

(Примљено 26. фебруара 1999)

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