

A Simple Synthesis of Some New Thienopyridine and Thienopyrimidine Derivatives

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Abstract: A number of pyridothienopyridine (2, 3), pyridothienopyrimidine (6, 10), pyridothienotriazine (13), pyrimidothienopyrimidine (15, 16, 17a,b) and thienoimidazo-triazines (18) were obtained via interaction of 3-amino-5-phenylamino-2,4-dicarbonitrile (1) with different reagents.

Keywords: pyridothienopyridines, pyridothienopyrimidines, thienotriazopyrimidines, pyridothienotriazines, pyrimidothienopyrimidines.

Introduction

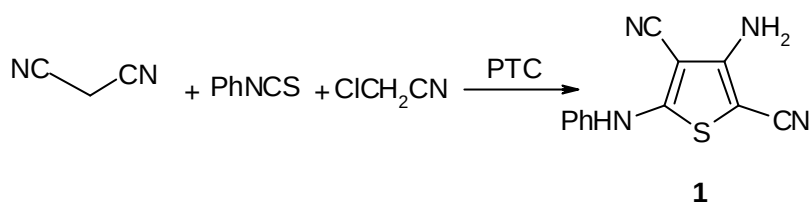
Many thienopyridines have been evaluated pharmacologically and have been found to show activity against, for example, diabetes mellitus [1-3], as analgesics and antiinflammatories [4-6], sedatives [4], anticoagulants [6], antiatherosclerotics [7], and as gonadotropin releasing hormone antagonists [8]. Moreover the pyridothienopyrimidines showed analgesic and antiinflammatory activity [9].

In earlier communications, we have reported the syntheses and reactions of various thiophenes and fused thiophenes [10-12], which were prepared in one-pot reactions using phase transfer techniques (PTC). Also, several papers have reported the synthesis of polyfused heterocycles containing different nuclei [13-16], starting from fused thiophenes prepared in our laboratory.

Results and Discussion

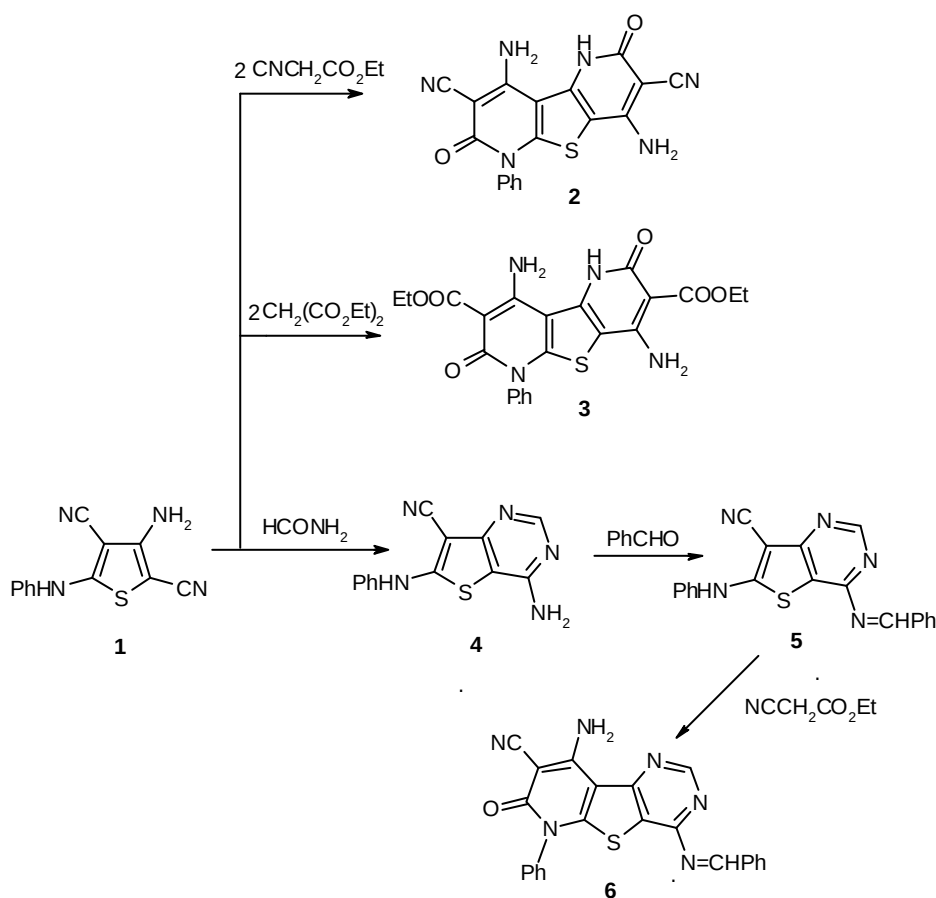
We report herein the synthesis of some new polyfunctionally substituted thienopyridines, thienopyrimidines and pyridothienopyrimidines starting with 3-amino-5-phenylamino-2,4-dicarbonitrile (**1**). Compound **1** was prepared in one-pot reaction using PTC conditions [K_2CO_3 /benzene/ tetrabutylammonium bromide (TBAB)] from malononitrile, PhNCS and $ClCH_2CN$ compounds in a 1:1:1 molar ratio, (Scheme 1) [12].

Scheme 1



Compound **1** was allowed to react with two moles of ethyl cyanoacetate or diethyl malonate in the presence of ammonium acetate-acetic acid at 200°C [17] to give the corresponding 4,9-diamino-2,7-dioxo-6-phenyl-1,2,6,7-tetrahydropyrido[2',3':4,5]thieno[2,3-b]pyridin-3,8-dicarbonitrile (**2**) or 4,9-diamino-3,8-diethoxycarbonyl-6-phenyl-1,2,6,7-tetrahydro[2',3':4,5]thieno[2,3-b]pyridin-2,7-dione (**3**).

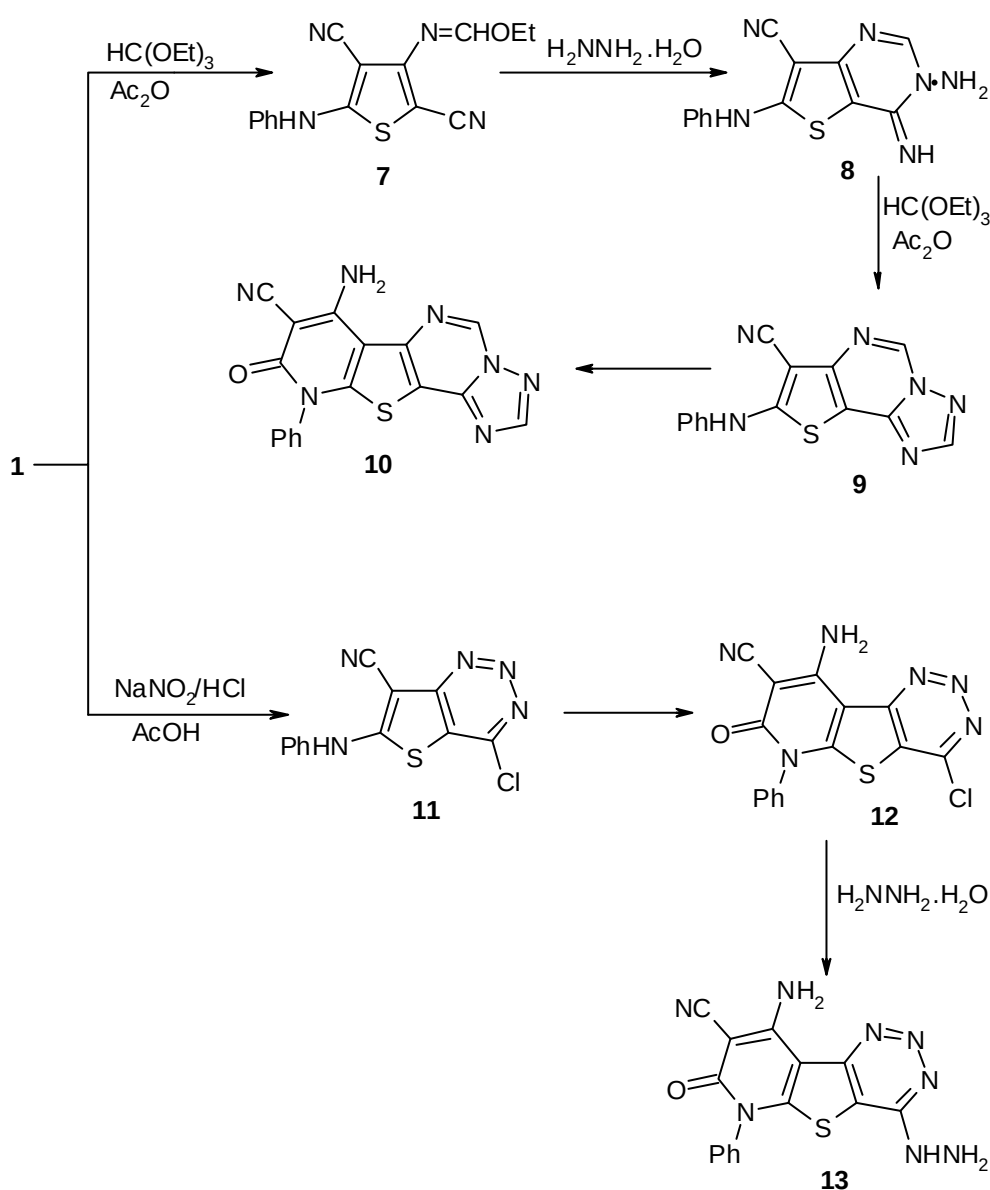
Scheme 2



Reaction of compound **1** with formamide gave the corresponding 4-amino-6-phenylamino-thieno[3,2-d]pyrimidin-7-carbonitrile (**4**) which was allowed then allowed to react with benzaldehyde to yield 6-phenylamino-4-benzalaminothieno[3,2-d]pyrimidin-7-carbonitrile (**5**). 9-Amino-7-oxo-6-phenyl-4-phenylmethanimido-6,7-dihydropyrido-[3',2':4,5]thieno[3,2-d]pyrimidin-8-carbonitrile (**6**) was obtained by treating compound **5** with ethyl cyanoacetate (Scheme 2).

The condensation of compound **1** with triethyl orthoformate in refluxing acetic anhydride gave ethyl N-(2,4-dicyano-5-phenylamino-3-yl)metanimidate (**7**) which underwent further cyclization upon treatment with hydrazine hydrate at room temperature affording 3-amino-3,4-dihydro-4-imino-6-phenylaminothieno[3,2-d]pyrimidin-7-carbonitrile (**8**) (Scheme 3).

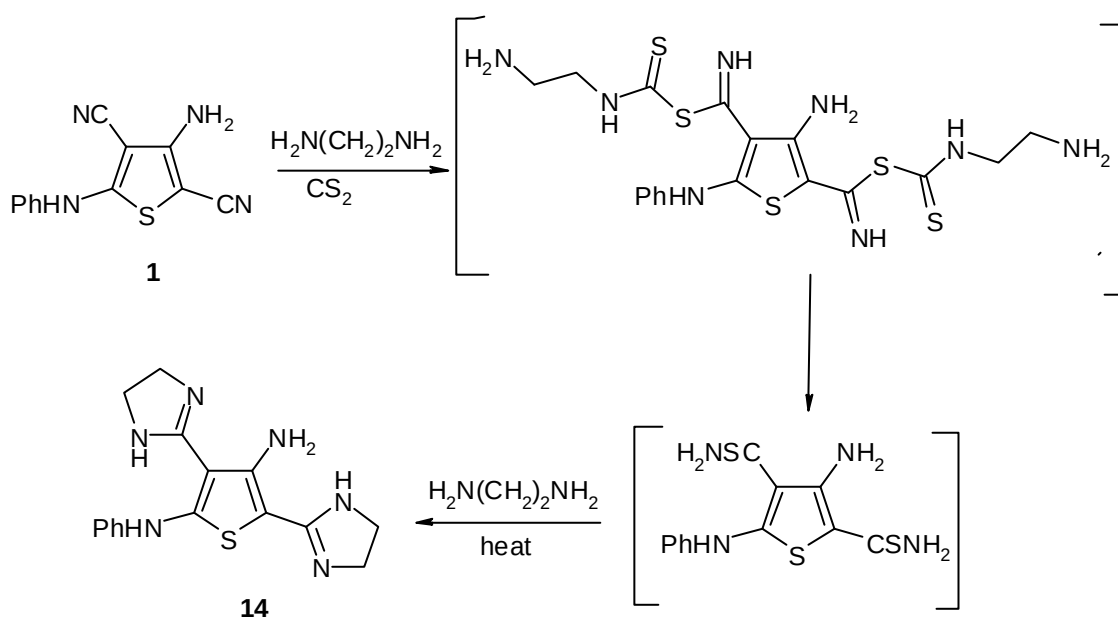
Scheme 3



Reaction of compound **8** with triethyl orthoformate gave 8-phenylaminothieno[3,2-d][1,2,4]-triazolo[3,2-f]pyrimidin-7-carbonitrile (**9**). Fusion of compound **9** with ethyl cyanoacetate in the presence of ammonium acetate yielded 7-amino-9-oxo-10-phenyl-9,10-dihydropyrido[3',2':4,5]-thieno[3,2-d][1,2,4]triazolo[3,2-f]pyrimidin-8-carbonitrile (**10**). Diazotisation of compound **1** with NaNO_2 /concentrated HCl led to the formation of 4-chloro-6-phenylaminothieno[3,2-d]-1,2,3-triazin-7-carbonitrile (**11**) which, in turn, was allowed to react with ethyl cyanoacetate to give 9-amino-4-chloro-7-oxo-6-phenyl-6,7-dihydropyrido[3',2':4,5]thieno[3,2-d][1,2,3]triazin-8-carbonitrile (**12**). Compound **12** was further treated with hydrazine hydrate to afford 9-amino-4-hydrazino-7-oxo-6-phenyl-6,7-dihydropyrido-[3',2':4,5]-thieno[3,2-d][1,2,3]triazin-8-carbonitrile (**13**).

Incorporation of the imidazolyl moiety in pyrimidothienopyrimidine, imidazothienopyrimidine and imidazothienotriazine systems were achieved by converting the nitrile group of compound **1** into a dihydroimidazolyl residue, followed by some additional reactions [18]. Thus, the interaction of compound **1** with ethylenediamine in the presence of carbon disulfide afforded 3-amino-2,4-di(4,5-dihydro-1H-2-imidazolyl)-5-phenylaminothiophene (**14**) (Scheme 4).

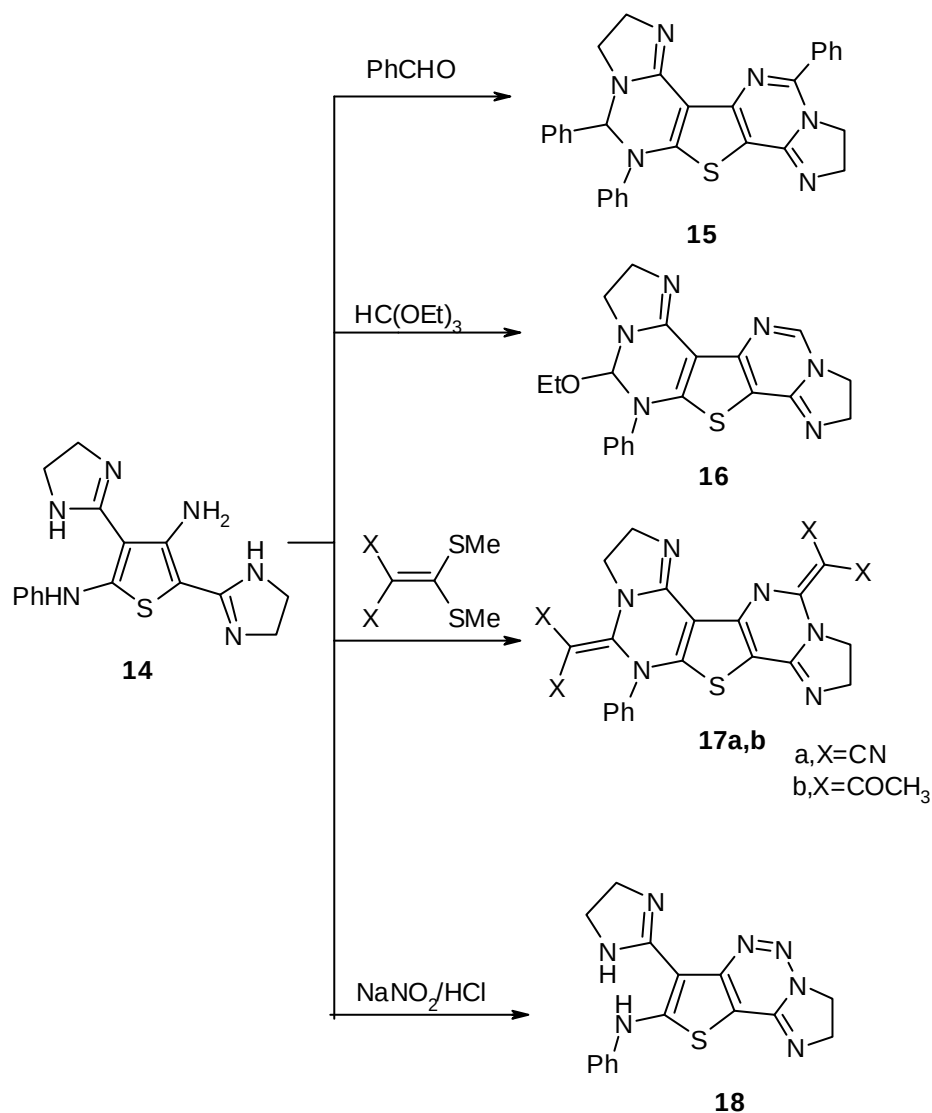
Scheme 4



Treatment of compound **14** with triethyl orthoformate, benzaldehyde, dimethylthiomethylenemalononitrile or dimethylthio-2-acetylbuten-3-one furnished 5,6,12-triphenyl-2,3,5,6,9,10-hexahydroimidazo[1,2-c]imidazo[2'',1'':6',1']pyrimido[4',5':4,5]thieno[3,2-e]pyrimidine (**15**), 5-ethoxy-6-phenyl-2,3,5,6,9,10-hexahydroimidazo[1,2-c]imidazo[2'',1'':6',1']pyrimido[4',5':4,5]-thieno[3,2-e]-pyrimidine (**16**), 2-(12-(1,1-dicyanomethylidene)-6-phenyl-2,3,5,6,9,10,12,13-octahydro-imidazo[1,2-c]imidazo[2'',1'':6',1']pyrimido[4',5':4,5]thieno[3,2-e]pyrimidin-5-ylidenemalononitrile (**17a**) or 2-(12-(1-acetyl-2-oxopropylidene)-6-phenyl-2,3,5,6,9,10,12,13-octahydroimidazo[1,2-c]imidazo[2'',1'':6',1']pyrimido[4',5':4,5]thieno[3,2-

e]pyrimidin-5-yliden)-2,4-pentandione (**17b**), respectively. In addition 7-(4,5-dihydro-1H-2-imidazolyl)-2,3-dihydro-8-phenylaminoimidazo[1,2-c]-thieno[2,3-e][1,2,3]-triazine (**18**) was obtained through the treatment of compound **14** with nitrous acid (Scheme 5). Analytical and spectral data of the newly synthesized compounds were in agreement with the proposed structures (Tables 1 and 2).

Scheme 5



Experimental

General

All melting points were determined on a Koffler melting points apparatus and are uncorrected. IR spectra were obtained on a Nicolet 710 FT-IR spectrometer. ^1H -NMR spectra were recorded on a Varian EM 360 A at 60 MHz using TMS as an internal reference. Elemental analyses were carried out with an elemental

analyzer model 240 C. Satisfactory microanalysis ($C \pm 0.4$, $H \pm 0.4$, $N \pm 0.3\%$) were obtained for all newly prepared compounds.

4,9-diamino-2,7-dioxo-6-phenyl-1,2,6,7-tetrahydropyrido[2',3':4,5]thieno[2,3-b]pyridin-3,8-dicarbonitrile (2) or *4,9-diamino-3,8-diethoxycarbonyl-6-phenyl-1,2,6,7-tetrahydro[2',3':4,5]-thieno[2,3-b]pyridin-2,7-dione (3)*. A mixture of **1** (0.01 mol), ethyl cyanoacetate or diethyl malonate (0.02 mol), ammonium acetate (6 g) and acetic acid (1.2 mL) was heated with stirring at 200 °C for 2h, left to cool and then triturated with ethanol. The solid product thus formed was collected by filtration. Compound **2** (from ethyl cyanoacetate) was recrystallized from ethanol as yellow needles; m. p. 200 °C. Compound **3** (from diethyl malonate) was recrystallized from ethanol as orange crystals; m. p. > 320 °C.

4-Amino-6-phenylaminothieno[3,2-d]pyrimidin-7-carbonitrile (4). A solution of **1** (0.001 mol) in formamide (10 mL) was refluxed for 2h. The precipitate that formed on cooling was filtered off and recrystallized from ethanol to give white powder; m.p. >271 °C.

6-Phenylamino-4-benzalaminothieno[3,2-d]pyrimidin-7-carbonitrile (5). An equimolar mixture of **4** (0.01 mol) and benzaldehyde (0.01 mol) was dissolved in EtOH (30 mL) in the presence of a few drops of piperidine. The reaction mixture was refluxed for 2h and left to cool. The solid product was filtered off and recrystallized from pyridine as white crystals; m.p. 243 °C.

General Procedure for the Preparation of Thienopyridone Derivatives 6, 10 or 12. A mixture of **1** (0.01 mol), ethyl cyanoacetate (0.01 mol), ammonium acetate (3 g) and acetic acid (0.6 mL) was heated with stirring at 200 °C for 2h, then left to cool and triturated with ethanol. The solid product, so formed, was collected by filtration and recrystallized from appropriate solvents.

9-Amino-7-oxo-6-phenyl-4-phenylmethanimido-6,7-dihydropyrido[3',2':4,5]thieno[3,2-d]-pyrimidin-8-carbonitrile (6). Recrystallized from pyridine as white crystals; m.p. 220 °C.

7-Amino-9-oxo-10-phenyl-9,10-dihydropyrido[3',2':4,5]thieno[3,2-d][1,2,4]triazolo-[3,2-f]-pyrimidin-8-carbonitrile (10). Recrystallized from acetonitrile as yellow crystals; m.p. 236 °C.

9-Amino-4-chloro-7-oxo-6-phenyl-6,7-dihydropyrido-[3',2':4,5]thieno[3,2-d][1,2,3]triazin-8-carbonitrile (12). Recrystallized from acetonitrile as yellow crystals; m.p. 158-160 °C.

Ethyl N-(2,4-dicyano-5-phenylamino-3-yl)metanimidate (7). A mixture of **1** (0.005 mol), triethyl orthoformate (3 mL) and acetic anhydride (20 mL) was heated under reflux for 5h. After cooling the precipitated solid was filtered off and recrystallized from ethanol as white crystals; m.p. 215 °C.

3-Amino-3,4-dihydro-4-imino-6-phenylaminoyhieno[3,2-d]pyrimidin-7-carbonitrile (8). Hydrazine hydrate (80%) (4 mL) was added to a suspension of **7** (0.005 mol) in dioxane (40 mL). The reaction mixture was stirred at room temperature for 1h. The precipitate which formed was filtered off, washed with water, dried in air and recrystallized from dioxane as white crystals; m.p. 185 °C.

8-Phenylaminothieno[3,2-d][1,2,4]triazolo[3,2-f]pyrimidin-7-carbonitrile (9). Compound **8** (0.001 mol) in an excess of triethyl orthoformate (7 mL) was refluxed for 1h. After cooling, the precipitated product was collected by filtration and recrystallized from ethanol-chloroform mixture as white needles; m.p. 299 °C.

4-Chloro-6-phenylamino-thieno[3,2-d]-1,2,3-triazin-7-carbonitrile (11). A solution of (0.01 mol) sodium nitrite in 10 mL of water was added to a cold solution of **1** (0.005 mol) in acetic acid (30 mL) and concentrated hydrochloric acid (15 mL). After completion of the addition, the ice bath was removed and stirring continued for an additional 2h. The crude product obtained was recrystallized from ethanol as white needles; m.p. 201 °C.

9-Amino-4-hydrazino-7-oxo-6-phenyl-6,7-dihydropyrido-[3',2':4,5]thieno[3,2-d][1,2,3]triazin-8-carbonitrile (13). A mixture of **12** (0.002 mol) and hydrazine hydrate (3 mL) in ethanol (20 mL) was refluxed for 1h. The precipitate that separated after cooling was recrystallized from dioxane as white crystals; m.p. 262 °C.

3-Amino-2,4-di(4,5-dihydro-1H-2-imidazolyl)-5-phenylaminothiophene (14). To a suspension of **1** (0.002 mol), ethylenediamine (3 mL) and carbon disulfide (1 mL) were added dropwise. The reaction mixture was heated on a water bath for 2h. The precipitated solid was triturated with ethanol (10 mL), filtered off and recrystallized from ethanol to give golden yellow crystals; m.p. 197 °C.

5,6,12-Triphenyl-2,3,5,6,9,10-hexahydroimidazo[1,2-c]imidazo-[2'',1'':6',1']pyrimido[4',5':4,5]-thieno-[3,2-e]pyrimidine (15). A mixture of **14** (0.005 mol), benzaldehyde (0.01 mol) and acetic acid (15 mL) was heated under reflux for 5 h. The precipitated solid was collected and recrystallized from dioxane in the form of white needles; m.p. 215 °C.

5-Ethoxy-6-phenyl-2,3,5,6,9,10-hexahydroimidazo[1,2-c]imidazo[2'',1'':6',1']pyrimido[4',5':4,5]-thieno[3,2-e]pyrimidine (16). Compound **14** (0.001 mol) in triethyl orthoformate (14 mL) was heated under reflux for 3 h. The precipitated solid was collected and recrystallized from pyridine as pale yellow crystals; m.p. 163 °C.

Preparation of 2-(12-(1,1-dicyanomethylidene)-6-phenyl-2,3,5,6,9,10,12,13-octahydroimidazo[1,2-c]imidazo[2'',1'':6',1']pyrimido[4',5':4,5]thieno[3,2-e]pyrimidin-5-yliden)malononitrile (17a) and 2-(12-(1-Acetyl-2-oxopropylidene)-6-phenyl-2,3,5,6,9,10,12,13-octahydroimidazo[1,2-c]imidazo-[2'',1'':6',1']pyrimido[4',5':4,5]thieno[3,2-e]pyrimidin-5-yliden)-2,4-pentandione (17b). A mixture of **1** (0.001

mol) and dimethylthiomethylenemalononitrile or 2-acetyl-1,1-dimethylthiobuten-3-one (0.002 mol) in ethanol (20 mL) was heated under reflux for 24h. The separated solids were collected and recrystallized from the appropriate solvent. Compound **17a**: gray crystals; m.p. 172 °C (from dioxane). Compound **17b**: orange crystals; m.p. 179 °C (from methanol).

7-(4,5-Dihydro-1H-2-imidazolyl)-2,3-dihydro-8-phenylaminoimidazo[1,2-c]thieno[2,3-e][1,2,3]-triazine (18). To a solution of **1** (0.001 mol) in concentrated sulphuric acid (2 mL) and glacial acetic acid (10 mL), sodium nitrite (0.003 mol) dissolved in 5 mL water was added dropwise with constant stirring during 10 minutes. The mixture was stirred without heating for additional 1 h and then diluted with water. The forming precipitate was filtered off and recrystallized from ethanol as white needles; m.p. 269 °C.

Table 1: Analytical data for the newly prepared compounds

Product	Yield %	Mol.Form. Mol. Wt.	Analysis(%) Calcd./Found			
			C	H	N	S
2	76	C ₁₈ H ₁₀ N ₆ O ₂ S 374.37	57.75	2.67	22.44	8.56
			57.64	2.73	22.55	8.62
3	81	C ₂₂ H ₂₀ N ₄ O ₆ S 468.48	56.40	4.27	17.93	6.83
			56.48	4.05	17.85	6.77
4	92	C ₁₃ H ₉ N ₅ S 267.30	58.41	3.63	26.28	11.97
			58.56	3.55	25.99	11.88
5	85	C ₂₀ H ₁₃ N ₅ S 355.41	67.58	3.66	19.69	9.00
			67.67	3.77	19.58	8.98
6	77	C ₂₃ H ₁₄ N ₆ OS 422.46	65.38	3.31	19.88	7.57
			65.44	3.23	19.92	7.61
7	89	C ₁₅ H ₁₂ N ₄ OS 269.21	60.82	4.05	18.90	10.82
			61.02	3.98	19.03	10.76
8	90	C ₁₃ H ₁₀ N ₆ S 282.32	55.30	3.54	29.75	11.33
			55.22	3.46	29.66	11.43
9	78	C ₁₄ H ₈ N ₆ S 292.31	57.52	2.74	28.74	11.94
			57.33	2.87	28.65	11.99
10	66	C ₁₇ H ₉ N ₇ OS 359.36	56.81	2.50	27.27	8.90
			56.77	2.56	27.30	9.12
11	75	C ₁₂ H ₆ N ₅ SCl 287.72	50.10	2.09	24.33	11.12
			49.99	1.98	24.15	10.98

12	81	C ₁₅ H ₇ N ₆ OSC 354.77	50.77	1.97	23.68	9.02
			50.85	2.01	23.44	8.94
13	64	C ₁₅ H ₁₀ N ₈ OS 350.35	51.42	2.85	31.97	9.13
			51.61	2.82	31.99	8.99
14	91	C ₁₆ H ₁₈ N ₆ S 326.41	58.87	5.61	25.73	9.80
			58.84	5.54	25.62	9.91
15	89	C ₃₀ H ₂₄ N ₆ S 500.62	71.97	4.79	211.40	6.39
			71.89	4.81	21.32	6.41
16	78	C ₂₀ H ₂₀ N ₆ OS 392.47	61.20	5.10	21.40	8.15
			60.32	4.94	21.22	8.00
17a	85	C ₂₄ H ₁₄ N ₁₀ S 474.50	60.75	2.95	22.50	6.74
			60.56	3.02	22.65	6.61
17b	82	C ₂₈ H ₂₆ N ₆ O ₄ S 542.61	61.97	4.79	15.48	5.89
			62.13	4.81	15.50	5.75
18	89	C ₁₆ H ₁₅ N ₇ S 337.40	56.95	4.44	29.04	9.48
			57.02	4.32	30.12	9.51

Table 2: Spectroscopic data of the newly prepared compounds.

Product	IR (KBr)^b	¹H-NMR^c (DMSO-d₆)
2	3430,3330,3300 (2NH ₂), 3180 (NH), 2222, 2210 (2CN), 1640, 1630 (C=O)	8.20 (s,1H,NH), 7.70-7.20 (m,5H,arom.), 6.20-5.90 (br,2H,NH ₂)
3	3420,3300,3250(2NH ₂), 3190 (NH), 1720,1710 (C=O ester), 1630 (C=O).	8.30-7.80 (m,5H,arom.), 6.30-6.10 (br,2H,NH ₂), 4.80-4.60 (br,2H,NH ₂), 2.80 (s,3H,COCH ₃), 2.10 (s,3H,CH ₃).
4	3340,3300, (NH ₂), 3200 (NH), 2218 (CN).	9.10 (s,1H,NH), 8.40 (s,1H,CH-Pyridine), 7.70-7.20 (m,5H,arom.), 5.50 (s,2H,NH ₂).
5	3200(NH), 2210 (CN).	9.10 (s,1H,NH), 8.40 (s,1H,CH-Pyridine), 7.90-7.30 (m,10H,arom.), 5.50-5.10 (br,2H,NH ₂).
6	3350,3240 (NH ₂), 2220 (CN), 1640 (C=O).	8.40 (s,1H,CH-Pyridine), 8.10 (s,1H,N=CH), 7.90-7.00 (m,10H,arom.), 6.20 (s,2H,NH ₂).
7	3180 (NH), 2220,2210 (CN), 1630 (C=N).	8.50 (s,1H,NH), 7.70 (s,1H,CH), 7.70-7.00 (m,5H,arom.), 3.80-3.40 (q,2H,OCH ₂), 1.25-1.00 (t,3H,CH ₃).
8	3430,3330 (NH ₂), 3200,3180 (2NH), 2190 (CN).	8.20-8.00 (br,2H,2NH), 7.80 (s,1H,CH), 7.70-7.20 (m,5H,arom.), 5.20 (s,2H,NH ₂).
9	3180 (NH), 2220 (CN), 1600 (C=N).	8.80 (s,1H,NH), 8.40 (s,1H,CH-Pyridine), 8.10 (s,1H,CH-triazole), 7.50-7.00 (m,5H,arom.), 6.20 (s,2H,NH ₂).

10	3400, 3330, 3300 (2NH ₂), 2217 (CN), 1640 (C=O), 1600 (C=N).	8.50 (s,1H,CH-Pyridine), 8.20 (s,1H,CH-triazole), 7.50-7.00 (m,5H,arom.), 6.20 (s,2H, NH ₂).
11	3320 (NH), 2210 (CN), 1640 (C=N).	8.40 (s,1H,NH), 7.70-7.20 (m,5H,arom.).
12	3340,3300, 3270(2NH ₂), 2220 (CN), 1640(C=O), 1620(C=N).	7.70-7.00 (m,5H,arom.), 6.20-6.00 (br,2H, (NH ₂).
13	3350,3330,3210 (2NH ₂), 3180, (NH), 2220 (CN), 1640 (C=O)	8.00 (s,1H,NH), 7.80-7.20 (m,5H,arom.), 6.10 (s,2H,NH ₂), 5.00 (s,2H,NH ₂).
14	3390,3260 (NH ₂), 3200,3180,3100 (3NH).	8.40 (s,1H,NH), 8.10 (s,1H,NH), 7.70-7.10 (m,5H,arom.), 5.60 (s,2H,NH ₂), 4.00-3.50 (m, 8H,4CH ₂ -imidazole).
15	3080 (CH-arom.), 1640 (C=N)	7.90-7.10 (m,15H,arom.), 4.00-3.40 (m,9H, 4CH ₂ -imidazole + 1H, CH-Ph)
16	3050 (CH-arom.), 1640 (C=N).	9.10 (s,1H,CH), 7.70-7.20 (m,5H,arom.), 4.00-3.40 (m,10H,4CH ₂ -imidazole+ OCH ₂), 1.25-1.00 (t,3H,CH ₃).
17a	3200 (NH), 2220,2210,2197 (4CN), 1620 (C=N).	9.00 (s,1H,NH), 7.70-7.20 (m,5H,arom.), 4.00-3.40 (m,8H,4CH ₂ -imidazole).
17b	3200 (NH), 1690,1670 (C=O), 1640 (C=N).	8.90 (s,1H,NH), 7.70-7.00 (m,5H,arom.), 4.00-3.40 (m,,8H,4CH ₂ -imidazole), 2.70-2.60 (ss, 12H,2COCH ₃).
18	3050 (CH-arom.), 1640 (C=N).	8.70 (s,1H,NH), 8.40 (s,1H,NH), 7.70-7.20 (m, 5H,arom.), 4.00-3.50 (m,8H,4CH ₂ -imidazole).

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