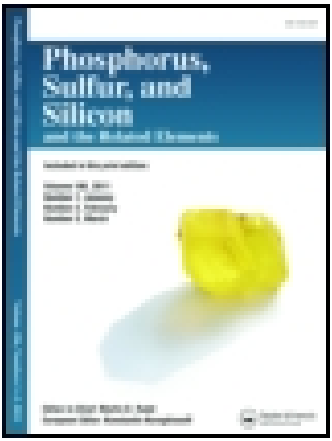

Phosphorus, Sulfur, and Silicon and the Related Elements

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SYNTHESIS OF SOME NEW THIENO[2,3-b]PYRIDINES, PYRIDO[3′,2′:4,5]-THIENO[3,2-d]PYRIMIDINES AND PYRIDO[3′,2′:4,5]THIENO[3,2-d][1,2,3]-TRIAZINES

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SYNTHESIS OF SOME NEW THIENO[2,3-*b*]PYRIDINES, PYRIDO[3′,2′:4,5]-THIENO[3,2-*d*]PYRIMIDINES AND PYRIDO[3′,2′:4,5]THIENO[3,2-*d*][1,2,3]-TRIAZINES

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4-Aryl-3-cyano-2-substituted-methylthiocyclopenta[*b*]pyridines (**3a-c** and **4a-i**) were prepared by reaction of 4-aryl-3-cyanocyclopenta[*b*]pyridine-2(1*H*)-thiones(**2a-c**) with chloroacetonitrile or chloro-*N*-arylacetamides, respectively. On treatment of these products with sodium ethoxide in boiling ethanol, they underwent intramolecular *Thorpe-Ziegler* cyclization to afford the corresponding 3-amino-4-aryl-2-functionallized-cyclopenta[*e*]thieno [2,3-*b*]pyridines (**5a-c** and **6a-i**). Most of the latter thienopyridines were used as synthons for the target cyclopenta[5′,6′]pyrido[3′,2′:4,5]thieno[3,2-*d*]pyrimidines and cyclopenta[5′,6′] pyrido[3′,2′:4,5]thieno[3,2-*d*][1,2,3]triazines.

Keywords: Cyclopenta[*b*]pyridines; Cyclopenta[*e*]thieno[2; 3-*b*]pyridines; Cyclopenta-[5′; 6′]pyrido[3′; 2′:4; 5]thieno[3; 2-*d*]pyrimidines; Cyclopenta[5′; 6′]pyrido[3′; 2′:4; 5]-thieno[3; 2-*d*][1; 2; 3]triazines

INTRODUCTION

In view of the broad spectrum of biological activities associated with many thieno[2,3-*b*]pyridines,[1–6] pyrido[3′,2′:4,5]thieno[2,3-*b*]pyrimidines,[7–10] and pyrido[3′,2′:4,5]thieno[2,3-*b*][1,2,3]triazines[11,12] and as a continuation of our program on annelated thieno[2,3-*b*]pyridines,[13–18] we reported herein the synthesis of the title compounds, as new agents in this field of anticipated biological and medicinal properties.

* Corresponding Author

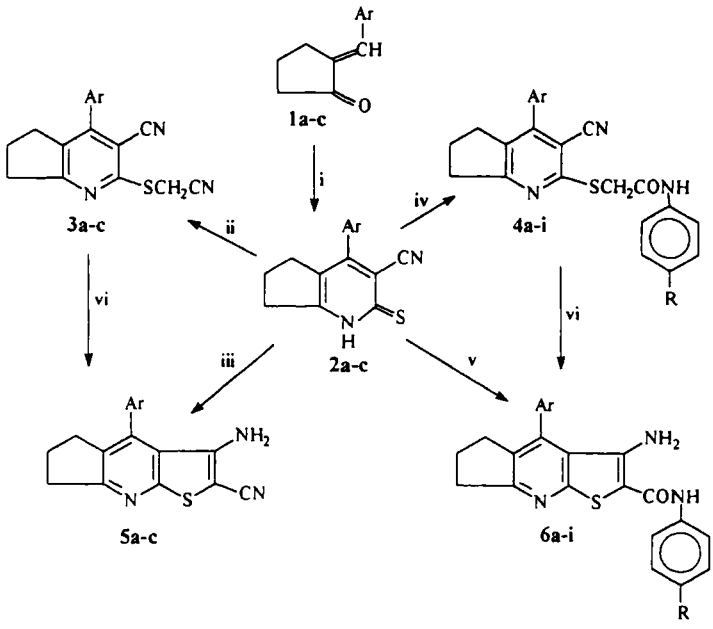
RESULTS AND DISCUSSION

The synthesis of the target compounds started from 4-aryl-3-cyanocyclopenta[*b*]pyridine-2(1*H*)-thiones (**2a-c**) which were prepared according to our previous procedure.[18] The reaction of **2a-c** with chloroacetonitrile or chloro-*N*-arylacetamides in refluxing ethanol containing sodium acetate gave the corresponding *S*-substituted-methylthiopyridines **3a-c** and **4a-i**. On heating these products in ethanol in the presence of catalytic amounts of sodium ethoxide, they underwent intramolecular *Thorpe-Ziegler* cyclization to give the promising 3-amino-4-aryl-2-functionallized-cyclopenta[*e*]thieno[2,3-*b*]pyridines (**5a-c** and **6a-i**). The latter compounds were also obtained *via* direct reaction of **2a-c** with the respective halo compounds in the presence of sodium ethoxide as a basic catalyst (Scheme 1).

3-Amino-4-aryl-2-functionallized-cyclopenta[*e*]thieno[2,3-*b*]pyridines (**5a-c** and **6a-i**) were used as good synthons for the target cyclopentapyridothienopyrimidines and cyclopentapyridothienotriazines. Thus, refluxing of **5a,b** with formamide resulted in the formation of aminopyrimidine derivatives **7a,b**. The interaction of **5a** with carbon disulfide afforded pyrimidinedithione **8**. Also, compound **5a** was reacted with phenyl isothiocyanate to give the thiourea derivative **9** which was cyclized into the corresponding cyclopentapyridothienopyrimidine **10** by heating in methanol containing sodium methoxide (Scheme 2).

Incorporating the imidazolyl moiety in pyridothienopyrimidine and pyridothienotriazine systems was achieved by converting the nitrile group of **5a,b** into a dihydroimidazolyl residue followed by some subsequent reactions. Thus, the interaction of *o*-aminocarbonitriles **5a,b** with ethylenediamine in the presence of carbon disulfide afforded 3-amino-4-aryl-2-(4′,5′-dihydroimidazol-2′-yl)-cyclopenta[*e*]thieno[2,3-*b*]pyridines (**11a,b**) in excellent yields. The compounds **11a,b** were reacted with triethyl orthoformate, *p*-chlorobenzaldehyde, and/or carbon dislufide to furnish the imidazolo[1″,2″-*c*]cyclopenta[5′,6′]pyrido[3′,2′:4,5]thieno[2,3-*e*]pyrimidine derivatives **12a,b, 13a,b** and **14a,b**, respectively. The 1,2,3-triazine analogue **15** was obtained upon treatment of **11a** with nitrous acid (Scheme 3).

The condensation of *o*-aminocarbonitrile **5c** with triethyl orthoformate in refluxing acetic anhydride afforded the methanimidate derivative **16** which underwent further reaction with hydrazine hydrate at room tempera-



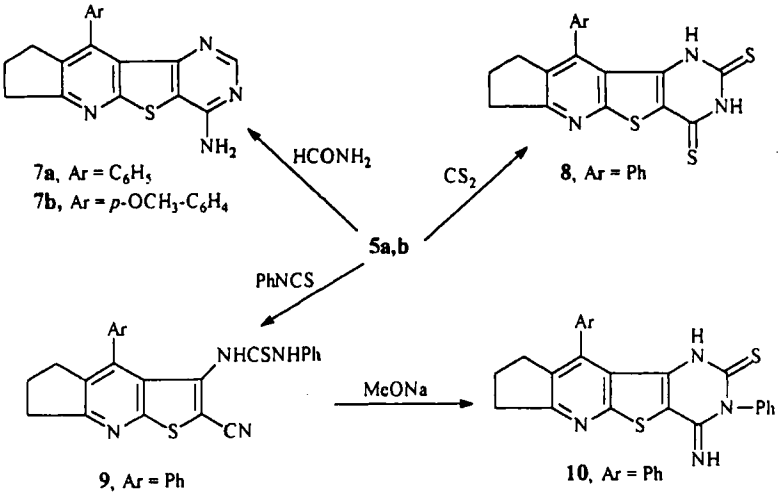
1-3, 5	Ar
a	C_6H_5
b	*p*-OCH_3-C_6H_4
c	*p*-Cl-C_6H_4

4, 6	Ar	R
a	C_6H_5	H
b	*p*-OCH_3-C_6H_4	Cl
c	*p*-Cl-C_6H_4	CH_3
d	C_6H_5	H
e	*p*-OCH_3-C_6H_4	Cl
f	*p*-Cl-C_6H_4	CH_3
g	C_6H_5	H
h	*p*-OCH_3-C_6H_4	Cl
i	*p*-Cl-C_6H_4	CH_3

i: $CNCH_2CSNH_2$ / MeONa; ii: $ClCH_2CN$ / AcONa; iii: $ClCH_2CN$/ EtONa; iv $ClCH_2CONHC_6H_4R(p)$/ AcONa; v: $ClCH_2CONHC_6H_4R(p)$ / EtONa; vi: EtONa

SCHEME 1

ture to give 3-amino-10-(*p*-chlorophenyl)-4-imino-1,2,3,4-tetrahydrocyclopenta[5′,6′]pyrido[3′,2′:4,5]thieno[2,3-*b*]pyrimidine (**17**). The latter compound was reacted with triethyl orthoformate to give *s*-triazole derivative **18**. Fusion of **17** with acetylacetone resulted in the formation of



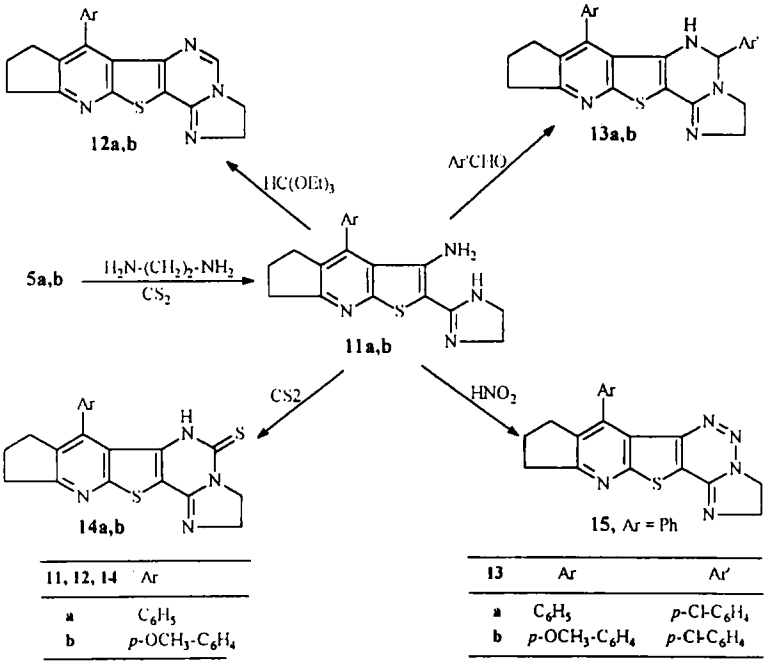
SCHEME 2

methyl-*s*-triazolo compound **20** instead of the expected triazepine derivative **19**. The structure of compound **20** was further confirmed by another route of preparation *via* treatment of **17** with acetic anhydride (Scheme 4).

Diazotisation of compound **5c** led to the formation of 4-chlorotriazine **21** which, in turn, was reacted with hydrazine hydrate to give 10-(*p*-chlorophenyl)-4-hydrazinocylopenta[5′,6′]pyrido[3′,2′:4,5]thieno[3,2-*d*][1,2,3]traizine (**22**). This compound was cyclocondensed with acetylacetone to furnish dimethylpyrazolyltriazine **23** (Scheme 5).

When compounds **6a-i** were allowed to react with triethyl orthoformate in refluxing acetic anhydride, the 3,10-diarylcyclopenta[5′,6′]pyrido [3′,2′:4,5]thieno[2,3-*b*]pyrimidine-4(3*H*)-ones (**24a-i**) were produced in excellent yields. The [1,2,3]triazinone analogs **25a-i** were prepared by treating **6a-i** in a AcOH-H_2SO_4 mixture with sodium nitrite (Scheme 6).

The interaction of **6d** with carbon disulfide in pyridine afforded 10-(*p*-methoxyphenyl)-4-oxo-3-phenyl-1,2,3,4-tetrahydro-2-thioxocyclopenta[5′,6′]pyrido[3′,2′:4,5]thieno[3,2-*d*]pyrimidine (**26**). This compound was also obtained by reaction of 3-amino-4-(*p*-methoxyphenyl)-cyclopenta[*e*]thieno[2,3-*b*]pyridine-2-carboxamide with phenyl isothiocyanate.[18] Compound **26** was reacted with ethyl iodide and/or with



11, 12, 14	Ar
a	C_6H_5
b	p-OCH_3-C_6H_4

13	Ar	Ar'
a	C_6H_5	p-Cl-C_6H_4
b	p-OCH_3-C_6H_4	p-Cl-C_6H_4

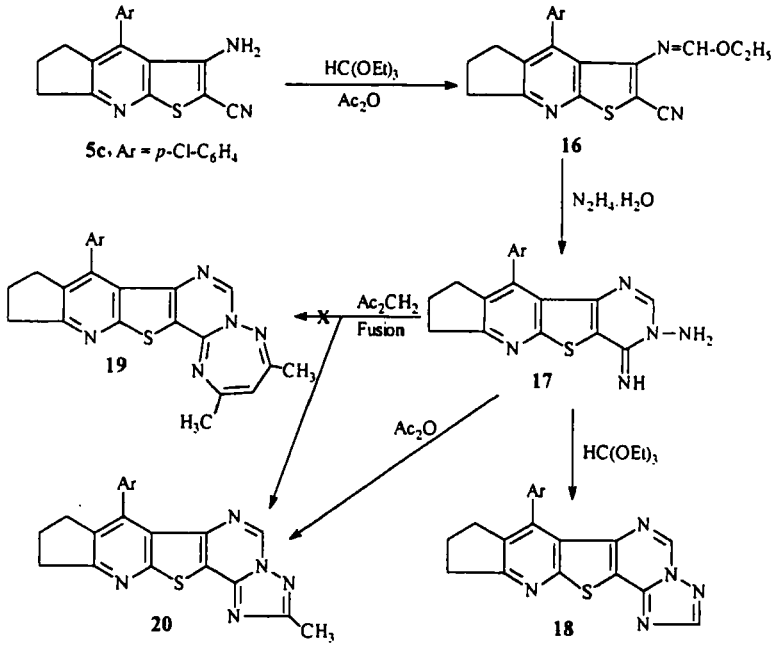
SCHEME 3

hydrazine hydrate to give 2-ethylthiopyrimidinone **27** or 2-hydrazinopyrimidinone **28**, respectively. Refluxing **28** with triethyl orthoformate led to the formation of 11-(*p*-methoxyphenyl)-4-phenylcyclopenta[5′,6′]pyrido [3′,2′:4,5]thieno[2,3-*e*]-*s*-triazolo[4″,3″-*a*]pyrimidine-5(4*H*)-one (**29**).

The structural formulas of all newly synthesized compounds were elucidated and confirmed by elemental and spectral analyses (Tables I–3). Its important to note that the ^{1}HNMR spectra of compounds **11a** and **17** showed no signal for NH proton and this can be explained by the rapid proton exchange as reported before.[19–21]

EXPERIMENTAL

All melting points are uncorrected and measured on a Gallan-Kamp apparatus. IR spectra were recorded on a Shimadzu 470 IR-Spectrophotometer (KBr; υ_{max} in cm^{-1}); ^{1}H-NMR spectra on a Varian EM-390, 90 MHz spec-

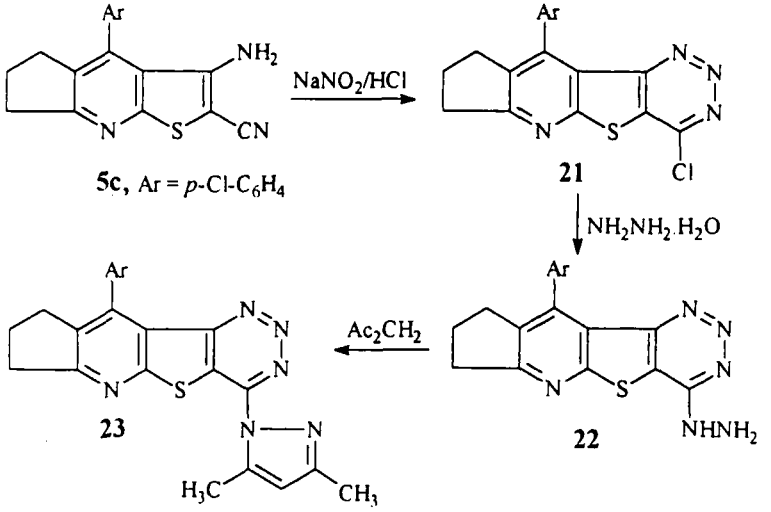


SCHEME 4

trometer, with TMS as an internal standard or on a Jeol LA 400 MHz FT-NMR spectrometer (δ in ppm). MS were taken on a Jeol JMS-600 mass spectrometer, and elemental analyses wre obtained on a Perkin-Elmer 240C elemental analyser.

4-(p-Chlorophenyl)-3-cyanocyclopenta[b]pyridine-2(1H)-thiones (2c)

A mixture of 2-(*p*-chlorobenzylidene)-cyclopentanone (2.07 g, 0.01 mol) and cyanothioacetamide (1.0 g, 0.01 mol) in sodium methoxide solution (0.05 g sodium in 30 mL methanol) was heated at 50 °C for 48 hr. The crystalline solid that formed on cooling was collected and recrystallized (ethanol) to give **2c**. The other cyclopenta[b]-pyridinethiones **2a,b** were prepared in a similar way.[18]



SCHEME 5

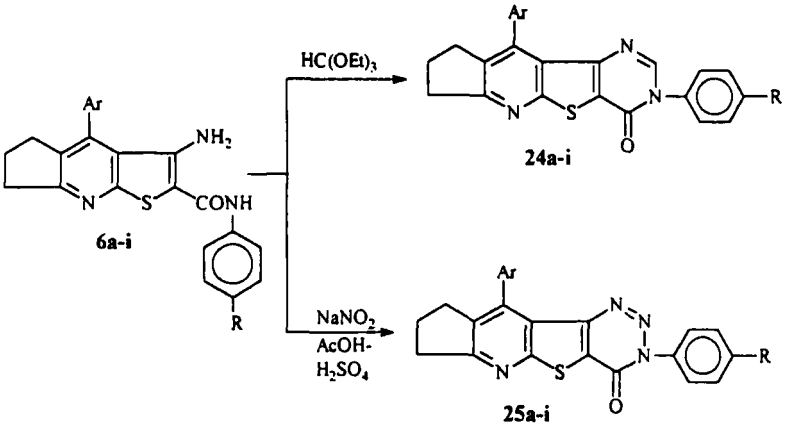
Reaction of 2a-c with chloroacetonitrile or chloro-N-arylacetamides; formation of S-substitutedmethylthiopyridines 3a-c or 4a-i, respectively; general procedure

A mixture of **2a-c** (0.1 mol), the respective halocompound (0.1 mol), and sodium acetate trihydrate (13.6 g, 0.1 mol) in ethanol (200 mL) was heated under reflux for 2 hr. The precipitate thus formed on cooling was filtered off, washed with water, dried in air, and recrystallized (ethanol) to give white crystals of **3a-c** or **4a-i**.

3-Amino-4-aryl-2-functionallized-cyclopenta[e]thieno[2,3-b]pyridines (5a-c and 6a-i); general procedures

A) A suspension of **3a-c** or **4a-i** (0.01 mol) in sodium ethoxide solution (0.05 g of sodium in 30 mL of abs. ethanol) was heated under reflux for 5 min. The solid that formed after cooling was collected and recrystallized (ethanol) to give canary yellow crystals of **5a-c** or **6a-i** (yields are given in Table I).

B) To a suspension of **2a-c** (0.01 mol) in sodium ethoxide solution (0.28 g of sodium in 40 mL of abs. ethanol) was added the respective halocom-



24,25	Ar	R
a	C_6H_5	H
b	*p*-OCH_3-C_6H_4	Cl
c	*p*-Cl-C_6H_4	CH_3
d	C_6H_5	H
e	*p*-OCH_3-C_6H_4	Cl
f	*p*-Cl-C_6H_4	CH_3
g	C_6H_5	H
h	*p*-OCH_3-C_6H_4	Cl
i	*p*-Cl-C_6H_4	CH_3

SCHEME 6

pound (0.01 mol). The resulting mixture was heated under reflux for 20 min. The yellow precipitate that formed after cooling was collected and recrysallized (ethanol) to give compounds **5a-c** and **6a-i** (yields: 60–70 %). These products were identical with those described in method A above.

4-Amino-10-aryl-cyclopenta[5′,6′]pyrido[3′,2′:4,5]thieno[3,2-d] pyrimidines(7a,b)

A solution of **5a,b** (0.001 mol) in formamide (10 mL) was heated under reflux for 2 hr. On cooling, the precipitate that formed was filtered off and recrystallized (ethanol) to give **7a,b**.

TABLE I Melting points, yields and analytical data of all newly synthesized compounds

Compd. No.	M.p., °C Yield, %	Formula (M. Wt.)	Calculated / Found				
			% C	% H	%N	%S	%Cl
2c	265	$C_{15}H_{11}ClN_2S$	62.82	3.87	9.77	11.18	12.36
	65	(286.8)	62.70	3.70	9.65	11.48	12.17
3a	130	$C_{17}H_{13}N_3S$	70.08	4.49	14.42	11.00	–
	90	(291.4)	69.81	4.50	14.23	10.92	–
3b	195	$C_{18}H_{15}N_3OS$	67.27	4.70	13.07	9.98	–
	85	(321.4)	66.94	4.65	13.20	9.58	–
3c	160	$C_{17}H_{12}ClN_3S$	62.67	3.71	12.89	9.84	10.88
	86	(325.8)	62.53	3.45	12.96	10.00	10.60
4a	190	$C_{23}H_{19}N_3OS$	71.66	4.97	10.90	8.32	–
	87	(385.5)	71.56	4.71	10.59	8.46	–
4b	135	$C_{23}H_{18}ClN_3OS$	65.78	4.32	10.00	7.64	8.44
	89	(419.9)	65.96	4.17	9.85	7.50	8.68
4c	160	$C_{24}H_{21}N_3OS$	72.15	5.30	10.52	8.03	–
	86	(399.5)	72.37	5.61	10.35	7.90	–
4d	175	$C_{24}H_{21}N_3O_2S$	69.38	5.09	10.11	7.72	–
	80	(415.5)	69.59	4.90	10.13	7.43	–
4e	165	$C_{24}H_{20}ClN_3O_2S$	64.07	4.48	9.34	7.13	7.88
	83	(450.0)	63.95	4.51	9.21	6.97	7.59
4f	200	$C_{25}H_{23}N_3O_2S$	69.91	5.39	9.78	7.46	–
	90	(429.5)	70.22	5.36	9.70	7.80	–
4g	155	$C_{23}H_{18}ClN_3OS$	65.78	4.32	10.00	7.63	8.44
	81	(419.9)	65.75	4.13	10.11	7.90	8.37
4h	155	$C_{23}H_{17}Cl_2N_3OS$	60.80	3.77	9.24	7.05	15.60
	88	(454.4)	61.13	3.60	9.50	7.20	15.90
4i	168	$C_{24}H_{20}ClN_3OS$	66.43	4.65	9.68	7.38	8.17
	81	(434.0)	66.22	4.60	9.87	7.00	8.28
5a	209	$C_{17}H_{13}N_3S$	70.08	4.49	14.42	11.00	–

Compd. No.	*M.p., °C Yield, %*	*Formula (M. Wt.)*	*Calculated / Found*				
			% C	*% H*	*%N*	*%S*	*%Cl*
	90	(291.4)	69.81	4.20	14.11	10.70	–
5b	195	$C_{18}H_{15}N_3OS$	67.27	4.70	13.07	9.98	–
	92	(321.4)	67.65	4.40	12.84	10.14	–
5c	250	$C_{17}H_{12}ClN_3S$	62.67	3.71	12.89	9.84	10.88
	90	(325.8)	62.59	3.54	12.90	9.51	11.11
6a	230	$C_{23}H_{19}N_3OS$	71.66	4.97	10.90	8.32	–
	95	(385.5)	71.84	4.62	10.97	8.58	–
6b	240	$C_{23}H_{18}ClN_3OS$	65.78	4.32	10.00	7.64	8.44
	93	(419.9)	65.59	4.60	9.71	7.60	8.31
6c	230	$C_{24}H_{21}N_3OS$	72.15	5.30	10.52	8.03	–
	94	(399.5)	72.11	5.51	10.79	8.42	–
6d	275	$C_{24}H_{21}N_3O_2S$	69.38	5.09	10.11	7.72	–
	93	(415.5)	69.10	5.16	9.80	7.60	–
6e	258	$C_{24}H_{20}ClN_3O_2S$	64.07	4.48	9.34	7.13	7.88
	93	(450.0)	63.91	4.60	9.27	6.95	7.89
6f	220	$C_{25}H_{23}N_3O_2S$	69.91	5.39	9.78	7.46	–
	90	(429.5)	69.71	5.09	9.50	7.23	–
6g	260	$C_{23}H_{18}ClN_3OS$	65.78	4.32	10.00	7.63	8.44
	95	(419.9)	65.50	4.20	10.13	7.81	8.20
6h	210	$C_{23}H_{17}Cl_2N_3OS$	60.80	3.77	9.24	7.05	15.60
	94	(454.4)	60.97	3.34	9.60	7.35	15.82
6i	215	$C_{24}H_{20}ClN_3OS$	66.43	4.65	9.68	7.38	8.17
	90	(434.0)	66.31	4.38	10.00	7.18	8.45
7a	310	$C_{18}H_{14}N_4S$	67.90	4.43	17.59	10.07	–
	70	(318.4)	68.00	4.59	17.22	10.10	–
7b	316	$C_{19}H_{16}N_4OS$	65.49	4.62	16.67	9.20	–
	73	(348.4)	65.15	5.01	16.75	9.16	–
8	310	$C_{18}H_{13}N_3S_3$	58.80	3.56	11.46	26.16	–

Compd. No.	M.p., °C Yield, %	Formula (M. Wt.)	Calculated / Found				
			% C	% H	%N	%S	%Cl
	69	(367.6)	59.01	3.60	11.12	26.38	–
9	169	$C_{24}H_{18}N_4S_2$	67.58	4.25	13.13	15.03	–
	85	(426.6)	67.31	4.43	13.04	15.32	–
10	310	$C_{24}H_{18}N_4S_2$	67.58	4.25	13.13	15.03	–
	91	(426.6)	67.18	3.99	12.85	15.18	–
11a	225	$C_{19}H_{18}N_4S$	67.05	5.62	17.37	9.94	–
	81	(334.4)	66.80	5.86	17.78	9.79	–
11b	220	$C_{20}H_{20}N_4OS$	65.90	5.53	15.37	8.79	–
	80	(364.5)	66.00	5.19	15.50	8.50	–
12a	303	$C_{20}H_{16}N_4S$	69.75	4.68	16.26	9.31	–
	80	(344.4)	69.99	4.82	16.50	9.56	–
12b	290	$C_{21}H_{18}N_4OS$	67.34	4.85	14.96	8.56	–
	90	(374.5)	67.13	4.61	14.81	8.37	–
13a	315	$C_{26}H_{21}ClN_4S$	68.33	4.59	12.26	7.02	7.76
	65	(457.0)	68.30	4.29	12.50	7.15	7.50
13b	291	$C_{27}H_{23}ClN_4OS$	66.59	4.76	11.50	6.58	7.27
	60	(487.0)	66.20	4.60	11.22	6.53	7.20
14a	351	$C_{20}H_{16}N_4S_2$	63.80	4.28	14.88	17.03	–
	80	(376.5)	63.50	4.05	14.90	17.20	–
14b	355	$C_{21}H_{18}N_4OS_2$	62.05	4.46	13.79	15.77	–
	85	(406.5)	61.70	4.33	13.97	15.52	–
15	200	$C_{19}H_{15}N_5S$	66.07	4.38	20.27	9.28	–
	73	(345.4)	65.87	4.13	20.15	9.40	–
16	218	$C_{20}H_{16}ClN_3OS$	62.88	4.22	11.03	8.39	9.28
	84	(382.0)	62.58	4.33	11.0	8.50	8.97
17	165	$C_{18}H_{14}ClN_5S$	58.78	3.84	19.04	8.72	9.64
	82	(367.9)	59.02	3.55	19.42	8.59	9.89
18	235	$C_{19}H_{12}ClN_5S$	60.39	3.20	18.53	8.49	9.38

Compd. No.	*M.p., °C Yield, %*	*Formula (M. Wt.)*	*Calculated / Found*				
			% C	*% H*	*%N*	*%S*	*%Cl*
	95	(377.9)	60.11	3.50	18.50	8.30	9.19
20	243	$C_{20}H_{14}ClN_5S$	61.29	3.60	17.87	8.18	9.05
	65	(391.9)	61.21	3.34	18.22	8.50	8.90
21	190	$C_{17}H_{10}Cl_2N_4S$	54.70	2.70	15.01	8.59	18.99
	80	(373.3)	55.02	2.50	14.68	8.58	19.13
22	241	$C_{17}H_{13}ClN_6S$	55.36	3.55	22.78	8.69	9.61
	96	(368.9)	55.00	3.41	22.60	8.30	9.32
23	270	$C_{22}H_{17}ClN_6S$	61.04	3.96	19.41	7.41	8.19
	84	(432.9)	61.11	3.90	19.50	7.60	8.00
24a	266	$C_{24}H_{17}N_3OS$	72.89	4.33	10.62	8.11	–
	90	(395.5)	73.06	4.02	10.90	8.32	–
24b	225	$C_{24}H_{16}ClN_3OS$	67.04	3.75	9.77	7.46	8.25
	92	(429.9)	67.19	3.50	9.60	7.12	8.45
24c	195	$C_{25}H_{19}N_3OS$	73.33	4.68	10.26	7.83	–
	90	(409.5)	73.50	4.49	10.30	8.00	–
24d	210	$C_{25}H_{19}N_3O_2S$	70.57	4.50	9.88	7.54	–
	93	(425.5)	70.37	4.33	9.50	7.46	–
24e	278	$C_{25}H_{18}ClN_3O_2S$	65.28	3.94	9.14	6.97	7.71
	90	(460.0)	65.40	3.89	8.85	7.00	7.58
24f	281	$C_{26}H_{21}N_3O_2S$	71.05	4.82	9.56	7.29	–
	87	(439.5)	71.19	4.69	9.20	6.93	–
24g	300	$C_{24}H_{16}ClN_3OS$	67.05	3.75	9.77	7.46	8.25
	87	(429.9)	67.40	3.90	9.50	7.60	8.00
24h	290	$C_{24}H_{15}Cl_2N_3OS$	62.08	3.26	9.05	6.90	15.27
	80	(464.4)	62.39	3.11	9.40	6.67	15.18
24i	260	$C_{25}H_{18}ClN_3OS$	67.64	4.09	9.46	7.22	7.99
	80	(444.0)	67.50	4.06	9.60	7.00	7.75
25a	225	$C_{23}H_{16}N_4OS$	69.68	4.07	14.13	8.08	–

Compd. No.	M.p., °C Yield, %	Formula (M. Wt.)	Calculated / Found				
			% C	% H	%N	%S	%Cl
	73	(396.5)	69.80	4.02	14.42	8.05	–
25b	186	$C_{23}H_{15}ClN_4OS$	64.11	3.51	13.00	7.44	8.23
	79	(430.9)	64.11	3.22	13.36	7.63	8.56
25c	201	$C_{24}H_{18}N_4OS$	70.22	4.42	13.65	7.81	–
	83	(410.5)	70.41	4.27	13.67	7.69	–
25d	230	$C_{24}H_{18}N_4O_2S$	67.59	4.25	13.14	7.52	–
	82	(426.5)	67.50	4.21	13.41	7.30	–
25e	190	$C_{24}H_{17}ClN_4O_2S$	62.54	3.72	12.15	6.96	7.69
	81	(460.9)	62.49	3.62	12.20	6.88	7.60
25f	200	$C_{25}H_{20}N_4O_2S$	68.16	4.58	12.71	7.28	–
	77	(440.5)	68.00	4.51	12.50	7.12	–
25g	240	$C_{23}H_{15}ClN_4OS$	64.11	3.51	13.00	7.44	8.22
	76	(430.9)	64.50	3.41	12.80	7.50	8.13
25h	210	$C_{23}H_{14}Cl_2N_4OS$	59.33	3.03	12.03	6.89	15.23
	79	(465.6)	59.11	3.00	11.90	7.11	14.90
25i	250	$C_{24}H_{17}ClN_4OS$	64.79	3.85	12.59	7.21	7.97
	70	(445.0)	64.70	3.76	12.20	7.20	7.55
26	285	$C_{25}H_{19}N_3O_2S_2$	65.62	4.18	9.18	14.01	–
	67	(457.6)	65.92	4.17	9.45	13.90	–
27	189	$C_{27}H_{23}N_3O_2S_2$	66.78	4.77	8.65	13.21	–
	75	(485.6)	66.81	4.55	8.40	13.37	–
28	310	$C_{25}H_{21}N_5O_2S$	65.93	4.65	15.37	7.04	–
	>300	(455.4)	65.90	4.37	15.25	7.00	–
29	271	$C_{26}H_{19}N_5O_2S$	67.08	4.11	15.04	6.89	–
	75	(465.5)	67.25	4.00	14.99	6.69	–

TABLE II IR spectral data of the prepared compounds (selected bands)

Assignment compound	υNH_2	υNH	$\upsilon C{\equiv}N$	$\upsilon C{=}O$	$\upsilon C{=}N$
2c	–	3180	2210	–	–
3a-c	–	–	2220, 2200	–	–
4a-i	–	3300–3240	2210–2220	1670–1650	–
5a-c	3450, 3320	–	2200	–	–
6a-i	3490, 3300	3200–3180	–	1640–1630	–
7a,b	3300, 3100	–	–	–	1640
8	–	3330, 3100	–	–	–
9	–	3200, 3100, 2200	–	–	–
10	–	3220, 3120	–	–	1600
11a,b	3480, 3310	3180	–	–	–
12a,b	–	–	–	–	1600
13a,b	–	3400	–	–	–
14a,b	–	3400	–	–	–
15	–	–	–	–	1600
16	–	–	2210	–	1630
17	3490, 3380	3150	–	–	1640
18	–	–	–	–	1600
20	–	–	–	–	1600
21	–	–	–	–	1600
22	3300–3200[a]	–	–	–	1640
23	–	–	–	–	1600
24a-i	–	–	–	1680–1670	–
25a-i	–	–	–	1680–1670	–
26	–	3300	–	1680	–
27	–	–	–	1660	–
28	3300, 3200	3100–2000[b]	–	1660	–
29	–	–	–	1670	–

a. For $NHNH_2$ group
b. Broad band

2,4-Dithioxo-10-phenyl-1,2,3,4-tetrahydrocyclopenta[5′,6′]pyrido-[3′,2′:4,5]-thieno[3,2-d]pyrimidine (8)

A mixture of **5a** (0.58 g, 0.002 mol) and carbon disulfide (1 mL) in dry pyridine (15 mL) was heated on a water bath for 6 hr. On cooling, the precipitate thus formed was filtered off and recrystallized (acetic acid) to give **8** in the form of orange needles.

N-(2-cyano-4-phenylcyclopenta[e]thieno[2,3-b]pyridin-3-yl)-N′-phenylthiourea (9)

A mixture of **5a** (0.58 g, 0.002 mol) and phenyl isothiocyanate (0.24 mL, 0.002 mol) in pyridine (15 mL) was heated on a water bath for 2 hr. The precipitate that formed after cooling and dilution with water was collected and recrystallized (ethanol) to give yellow needles of **9**.

3,10-Diphenyl-4-imino-1,2,3,4-tetrahydro-2-thioxocyclopenta[5′,6′] pyrido[3′,2′:-4,5]thieno[3,2-d]pyrimidine (10)

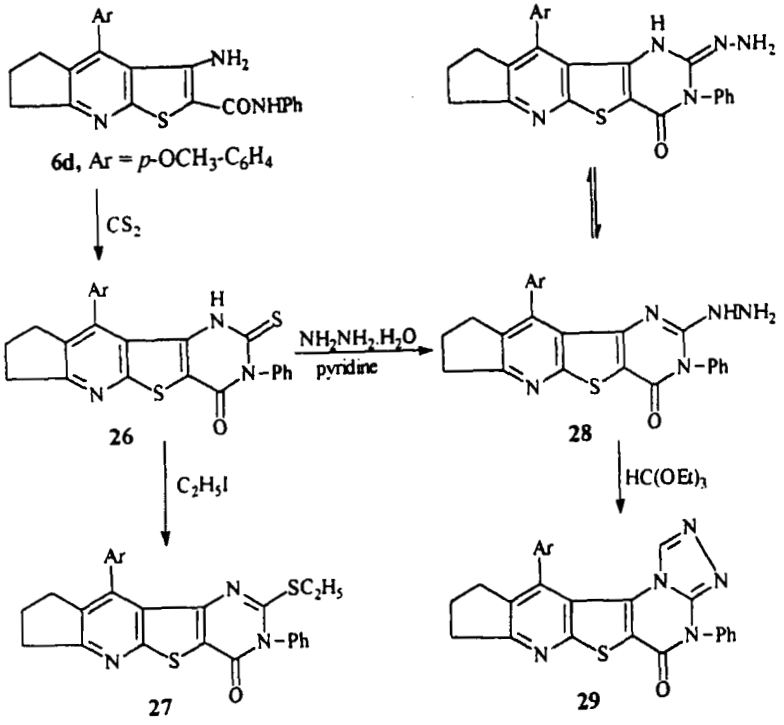
A suspension of **9** (0.85 g, 0.002 mol) in sodium methoxide solution (0.1 g of sodium in 30 mL of methanol) was heated under reflux for 2 hr. The precipitate that formed after cooling and acidification with acetic acid was collected and recrystallized (dioxane) to give yellow plates of **10**.

3-Amino-4-aryl-2-(4,5-dihydroimidazol-2-yl)-cyclopenta[e]thieno [2,3-b]pyridines (11a,b)

To a suspension of **5a,b** (0.002 mol) in ethylenediamine (3 mL) was added dropwise carbon disulfide (1 mL). The reaction mixture was heated on a water bath for 2 hr. The precipitated solid was triturated with ethanol (10 mL), filtered off and recrystallized (ethanol) to give golden yellow crystals of **11a,b**.

7-Aryl-2,3-dihydroimidazolo[1″,2″-c]cyclopenta[5′,6′]pyrido[3′,2′:4,5] thieno[2,3-e]pyrimidines (12a,b)

Compound **11a,b** (0.001 mol) in triethyl orthoformate (7 mL) was heated under reflux for 3 hr. The precipitated solid which formed was collected and recrystallized (pyridine) to give **12a,b** in the form of pale yellow crystals.



SCHEME 7

7-Aryl-5-(p-chlorophenyl)-2,3,5,6-tetrahydroimidazolo[1″,2″-c] cyclopenta[5′,6′]-pyrido[3′,2′:4,5]thieno[2,3-e]pyrimidines (13a,b)

A mixture of **11a,b** (0.005 mol) and *p*-chlorobenzaldehyde (0.7 g, 0.005 mol) in 15 mL of acetic acid was heated under reflux for 4 hr. The precipitate was collected and recrystallized (dioxane) as yellow needles of **13a,b**.

7-Aryl-2,3-dihydroimidazolo[1″,2″-c]cyclopenta[5′,6′]pyrido[3′,2′:4,5] thieno[2,3--e]pyrimidine-5(6H)-thiones (14a,b)

A mixture of **11a,b** (0.001 mol) and carbon disulfide (1 mL) in dry pyridine (10 mL) was heated on a water bath for 8 hr. On cooling, the precipi-

tated product was filtered off and recrystallized (pyridine) as orange crystals of **14a,b**.

2,3-Dihydro-7-phenylimidazolo [1″,2″-c]cyclopenta[5′,6′]pyrido [3′,2′:4,5]thieno-[2,3-e][1,2,3]triazine (15)

To a solution of **11a** (0.33 g, 0.001 mol) in concentrated sulphuric acid (1 mL) and glacial acetic acid (5 mL) was added dropwise sodium nitrite (0.1 g, 0.0015 mol) dissolved in 5 mL water with constant stirring during 10 min. The mixture was stirred cold for one additional hour and diluted with water. The precipitate was filtered off and crystallized (ethanol) as pale yellow crystals of **15**.

Ethyl-N-[(4-p-chlorophenyl)-2-cyanocyclopenta[e]thieno[2,3-b] pyridin-3-yl]-methanimidate (16)

A mixture of **5c** (1.7 g, 0.005 mol), triethyl orthoformate (3 mL) and acetic anhydride (20 mL) was heated under reflux for 5 hr. After cooling, the solid that formed was filtered off and recrystallized (ethanol) as colourless plates of **16**.

3-Amino-3,4-dihydro-4-imino-10-(p-chlorophenyl)-cyclopenta[5′,6′] pyrido-[3′,2′:4,5]thieno[3,2-d]pyrimidine (17)

To a suspension of **16** (1.9 g, 0.005 mol) in dioxane (40 mL) was added hydrazine hydrate (80%) (4 mL). The reaction mixture was stirred at room temperature for one hour. The precipitate which formed was filtered off, washed with water, dried in air and recrystallized (dioxane) as white needles of **17**.

7-(p-Chlorophenyl)-s-triazolo[2″,3″-c]cyclopenta[5′,6′]pyrido [3′,2′:4,5]thieno-[2,3-e]pyrimidine (18)

Compound **17** (0.36 g, 0.001 mol) in an excess amount of triethyl orthoformate (7 mL) was heated under reflux for one hour. The precipitated product was collected and recrystallized (ethanol-chloroform) to give **18** as white needles.

TABLE III ^{1}H-NMR and mass spectral data of some representative compounds

Compd No.	*Spectral data*
2c	^{1}H-NMR (DMSO-d_6): 14.39 (s, 1H, NH); 7.45–7.56 (m, 4H, ArH); 2.89–2.91 (t, 2H, CH_2 at C-7); 2.44–2.49 (t, 2H, CH_2 at C-5); 1.94–2.01 (p, 2H, CH_2 at C-6). MS: 287.8 (M^++1, 17.6%); 286.8 (M^+, 43.2%)
3a	^{1}H-NMR ($CDCl_3$): 7.15–7.60 (m, 5H, ArH); 4.00 (s, 2H, SCH_2); 3.00- 3.25 (t, 2H, CH_2 at C-7); 2.65–2.90 (t, 2H, CH_2 at C-5); 1.90–2.40 (p, 2H, CH_2 at C-6)
4b	^{1}H-NMR ($CDCl_3$): 9.55 (s, 1H, NH); 7.15–7.50 (m, 9H, ArH); 4.00 (s, 2H, SCH_2); 3.00–3.25 (t, 2H, CH_2 at C-7); 2.75–3.00 (t, 2H, CH_2 at C- 5); 1.95–2.30 (p, 2H, CH_2 at C-6).
4d	^{1}H-NMR ($CDCl_3$): 9.40 (s, 1H, NH); 7.00–7.60 (m, 9H, ArH); 4.00 (s, 2H, SCH_2); 3.90 (s, 3H, OCH_3); 3.10–3.35 (t, 2H, CH_2 at C-7); 2.80- 3.10 (t, 2H, CH_2 at C-5); 2.05–2.45 (p, 2H, CH_2 at C-6).
4f	^{1}H-NMR ($CDCl_3$): 9.30 (s, 1H, NH); 6.95–7.50 (m, 8H, ArH), 4.00 (s, 2H, SCH_2); 3.85 (s, 3H, OCH_3); 3.10–3.35 (t, 2H, CH_2 at C-7); 2.80- 3.05 (2H, CH_2 at C-5); 2.00–2.40 (m, 5H, CH_3 and CH_2 at C-6).
5a	^{1}H-NMR ($CDCl_3$): 7.30–7.70 (m, 5H, ArH); 4.35 (s, 2H, NH_2); 3.00- 3.30 (t, 2H, CH_2 at C-7); 2.65–2.90 (t, 2H, CH_2 at C-5); 1.95–2 30 (2H, CH_2 at C-6).
6f	^{1}H-NMR (DMSO-d_6): 9.30 (s, 1H, NH); 7.00–7.70 (m, 8H, ArH); 5.90 (s, 2H, NH_2); 3.90 (s, 3H, OCH_3); 3.05–3.30 (t, 2H, CH_2at C-7); 2.65- 2.90 (t, 2H, CH_2 at C-5); 2.00–2.50 (m, 5H, CH_3 and CH_2 at C-6).
6i	^{1}H-NMR (DMSO-d_6): 8.90 (s, 1H, NH); 7.05–7.70 (m, 8H, ArH); 5.70 (s, 2H, NH_2); 3.00–3.20 (t, 2H, CH_2 at C-7); 2.60–2.80 (t, 2H, CH_2 at C-5); 2.00–2.35 (m, 5H, CH_3 and CH_2 at C-6).
6h	MS: 456 (M^++2, 32%); 457 (M^++3, 13%); 454 (M^+, 100%).
7a	MS: 319 (M^++1, 9%); 318 (M^+, 26%); 317 (M^+-1, 100%).
7b	^{1}H-NMR (DMSO-d_6): 8.20 (s, 1H, CH pyrimidine); 6.90–7.40 (m, 6H, NH_2 and ArH); 3.80 (s, 3H, OCH_3); 3.00–3.25 (t, 2H, CH_2at C-7); 2.70–3.00 (t, 2H, CH_2 at C-9); 1.90–2.25 (t, 2H, CH_2 at C-8).

Compd No.	*Spectral data*
8	^{1}H-NMR (TFA) 7.3–7.7 (m, 5H, ArH); 3.5–3.8 (t, 2H, CH_2 at C-10), 3.0–3.4 (t, 2H, CH_2at C-8); 2.2–2.7 (p, 2H, CH_2 at C-9).
9	^{1}H-NMR ($CDCl_3$): 12.1 (s, 1H, NH); 11.0 (s, 1H, NH); 7.0–7.6 (m, 9H, ArH); 3.8 (s, 3H, OCH_3); 3.0–3.2 (t, 2H, CH_2 at C-7); 2.7–3.0 (t, 2H, CH_2 at C-5); 2.0–2.4 (p, 2H, CH_2 at C-6).
10	MS: 427 (M^++1, 25%); 426 (M^+, 67%); 426 (M^+-1, 100%).
11a	^{1}H-NMR ($CDCl_3$): 7.10–7.70 (m, 5H, ArH); 5.60 (br, 2H, NH_2); 3.50- 4.00 (br, 4H, $2CH_2$ dihydroimidazolyl); 3.00–3.20 (t, 2H, CH_2 at C-7); 2.60–2.80 (t, 2H, CH_2 at C-5); 2.00–2.40 (p, 2H, CH_2 at C-6).
12a	^{1}H-NMR (TFA): 8.50 (s, 1H, CH pyrimidine); 7.30–7.70 (m, 5H, ArH); 4.80–5.20 (t, 2H, CH_2 dihydroimidazolyl); 4.30–4.70 (t, 2H, CH_2 dihydroimidazolyl); 3.50–3.80 (t, 2H, CH_2 at C-10); 3.00–3.40 (t, 2H, CH_2at C-8); 2.20–2.70 (p, 2H, CH_2 at C-9).
12b	MS: 375.4 (M^++1, 36.5%); 374.4 (M^+, 66.8%); 373.4 (M^+-1, 100%)
13a	^{1}H-NMR (DMSO-d_6): 7.0–7.10 (m, 8H, ArH); 6.2 (s, 1H, CH, tetrahydropyrimidine); 6.0 (s, 1H, NH); 3.9 (s, 4H, $2CH_2$ dihydroimidazolyl); 3.5–3.8 (t, 2H, CH_2 at C-10); 3.0–3.3 (t, 2H, CH_2 at C-8); 2.3–2.7 (p, 2H, CH_2 at C-9).
14a	^{1}H-NMR ($CDCl_3$): 7.3–7.7 (m, 5H, ArH); 4.1 (s, 4H, $2CH_2$ dihydroimidazolyl); 3.1–3.3 (t, 2H, CH_2 at C-10); 2.7–2.9 (t, 2H, CH_2at C-8); 2.0- 2.4 (p, 2H, CH_2 at C-9).
15	^{1}H-NMR ($CDCl_3$): 7.4–8.0 (m, 5H, ArH); 4.1 (s, 4H, $2CH_2$ dihydroimidazolyl); 3.0–3.3 (t, 2H, CH_2 at C-10); 2.7–3.0 (t, 2H, CH_2at C-8); 2.0–2.4 (p, 2H, CH_2 at C-9).
16	^{1}H-NMR ($CDCl_3$): 7.6 (s. 1H, N=CH); 7.0–7.5 (dd, 4H, ArH); 3.4–3.8 (q, 2H, OCH_2); 3.0–3.3 (t, 2H, CH_2 at C-7); 2.7–3.0 (t, 2H, CH_2 at C-5); 2.0–2.4 (p, 2H, CH_2 at C-6); 1.0–1.3 (t, 3H, CH_3).
17	^{1}H-NMR (DMSO-d_6): 7.8 (s, 1H, CH pyrimidine); 7.2–7.7 (m, 4H, ArH); 5.2 (s, 2H, NH_2); 3.0–3.2 (t, 2H, CH_2 at C-7); 2.7–2.9 (t, 2H, CH_2 at C-9); 1.9–2.3 (p, 2H, CH_2at C-8).
18	^{1}H-NMR ($CDCl_3$): 9.00 (s, 1H, CH pyrimidine); 8.4 (s, 1H, CH triazole); 7.2–7.5 (m, 4H, ArH); 3.0–3.3 (t, 2H, CH_2 at C-10); 2.7–2.9 (t, 2H, CH_2at C-8); 2.0–2.3 (p, 2H, CH_2 at C-9).

Compd No.	*Spectral data*
20	^{1}H-NMR ($CDCl_3$): 8.93 (s, 1H, CH pyrimidine); 7.24–7.46 (dd, 4H, ArH); 3.18–3.22 (t, 2H, CH_2 at C-10); 2.84–2.88 (t, 2H, CH_2 at C-8); 2.62 (s, 3H, CH_3); 2.16–2.22 (p, 2H, CH_2 at C-9). MS: 391.8 (M^+, 43.9%); 392.9 (M^++1, 71.2%).
21	^{1}H-NMR ($CDCl_3$): 7.3–7.6 (m, 4H, ArH); 3.2–3.4 (t, 2H, CH_2 at C-7); 2.8–3.1 (t, 2H, CH_2 at C-9); 2.0–2.4 (p, 2H, CH_2at C-8).
22	^{1}H-NMR (DMSO-d_6): 9.6 (s, 1H, NH); 7.2–7.6 (m, 4H, ArH); 5.0 (br, 2H, NH_2); 3.0–3.3 (t, 2H, CH_2 at C-7); 2.7–2.9 (t, 2H, CH_2at C-9); 1.9- 2.3 (p, 2H, CH_2 at C-8).
23	^{1}H-NMR (DMSO-d_6): 7.4–7.7 (m, 4H, ArH); 6.1 (s, 1H, CH pyrazole); 3.2–3.4 (t, 2H, CH_2at C-7); 2.8–3.0 (m, 5H, CH_3 attached to pyrazole ring and CH_2 at C-9); 2.1–2.5 (m, 5H, CH_3 attached to pyrazole ring and CH_2 at C-8).
24d	^{1}H-NMR ($CDCl_3$): 7.9 (s, 1H, CH pyrimidine); 6.9–7.6 (m, 9H, ArH); 3.8 (s, 3H, OCH_3); 3.0–3.3 (t, 2H, CH_2 at C-7); 2.7–3.0 (t, 2H, CH_2 at C-9); 1.9–2.3 (p, 2H, CH_2 at C-8).
24f	^{1}H-NMR (TFA): 8.9 (s, 1H, CH pyrimidine); 7.2–7.7 (m, 8H, ArH); 4.0 (s, 3H, OCH_3); 3.6–3.8 (t, 2H, CH_2 at C-7); 3.1–3.4 (t, 2H, CH_2 at C-9); 2.3–2.7 (m, 5H, CH_3 and CH_2 at C-8).
25e	^{1}H-NMR ($CDCl_3$): 6.8–7.6 (m, 8H, ArH); 3.7 (s, 3H, OCH_3); 3.0–3.3 (t, 2H, CH_2 at C-7); 2.7–2.9 (t, 2H, CH_2 at C-9); 1.9–2.3 (p, 2H, CH_2 at C-8).
27	^{1}H-NMR ($CDCl_3$): 6.8–7.7 (m, 9H, ArH); 3.9 (s, 3H, OCH_3); 3.5–3.8 (q, 2H, SCH_2); 3.2–3.4 (t, 2H, CH_2 at C-7); 2.8–3.0 (t, 2H, CH_2 at C-9); 1.9–2.3 (p, 2H, CH_2at C-8); 1.1–1.4 (t, 3H, CH_3).
28	MS: 455.6 (M^+, 30%); 454.6 (M^+-1, 1.5%).
29	^{1}H-NMR (TFA): 9.0 (s, 1H, CH triazole); 7.2–7.7 (m, 9H, ArH); 4.0 (s, 3H, OCH_3); 3.5–3.8 (t, 2H, CH_2 at C-8); 3.1–3.4 (t, 2H, CH_2 at C-10); 2.4–2.7 (p, 2H, CH_2 at C-9).

7-(p-Chlorophenyl)-2-methyl-s-triazolo[2″,3″-c]cyclopenta[5′,6′] pyrido-[3′,2′:4,5]thieno[2,3-e]pyrimidine (20)

A) A mixture of **17** (0.72 g, 0.002 mol) and acetylacetone (4 mL) was heated under reflux for 3 hr. The crystalline precipitate which formed on cooling was collected and recrystallized (ethanol) as pale yellow plates. This product was identified as 7-(*p*-chlorophenyl)-2-methylcyclopenta[5′,6′]pyrido[3′,2′:4,5]thieno[2,3-*e*]-*s*-triazolo[2″,3″-*c*]pyrimidine (**20**).

B) A solution of **17** (0.36 g, 0.001 mol) in acetic anhydride (10 mL) was refluxed for 8 hr. The cooled reaction mixture was diluted with water and the precipitated solid was collected and crystallized (ethanol) to give **20**; yield, 0.27 g (71%); mp 243 °C, mmp 243 °C.

4-Chloro-10-(p-chlorophenyl)-cyclopenta[5′,6′]pyrido[3′,2′:4,5]thieno [3,2-d]-[1,2,3]triazine (21)

To an ice-cold solution of **5c** (1.62 g, 0.005 mol) in 30 mL of acetic acid and 15 mL of concentrated hydrochloric acid was added portionwise a solution of 0.7 g (0.01 mol) sodium nitrite in 10 mL of water. After completion of the addition, the ice bath was removed and stirring continued for two more hours. The crude product that obtained was recrystallized (ethanol) to give **21** as buff needles.

10-(p-Chlorophenyl)-4-hydrazinocyclopenta[5′,6′]pyrido[3′,2′:4,5] thieno[3,2-d]-[1,2,3]triazine (22)

A mixture of **21** (0.67 g, 0.002 mol) and hydrazine hydrate (3 mL) in ethanol (20 mL) was refluxed for one hour. The precipitate that separated after cooling was collected and recrystallized (dioxane) to give **22** as white crystals.

4-(3,5-Dimethylpyrazol-1-yl)-10-(p-chlorophenyl)-cyclopenta[5′,6′] pyrido[3′,2′:4,5]thieno[3,2-d][1,2,3]triazine (23)

A mixture of **22** (0.37 g, 0.001 mol) and acetylacetone (2 mL, 0.02 mol) in ethanol (15 mL) was heated under reflux for 3 hr. The product was collected and recrystallized (ethanol) as colourless needles of **23**.

3,10-Diaryl-cyclopenta[5′,6′]pyrido[3′,2′:4,5]thieno[3,2-d] pyrimidine-4(3H)-ones (24a-i)

A mixture of **6a-i** (0.005 mol) and triethyl orthoformate (2 mL, 0.012 mol) in acetic anhydride (20 mL) was heated under reflux for 4 hr. The crystalline solid thus formed was collected and recrystallized (ethanol) as white crystals of **6a-i**.

3,10-Diaryl-cyclopenta[5′,6′]pyrido[3′,2′:4,5]thieno[3,2-*d*][1,2,3] triazine-4(3*H*)-ones (25a-i)

Sodium nitrite solution (12 mL, 10%; 0.015 mol) was added to a solution of compound **6a-i**(0.01 mol) in concentrated HCl (5 mL) and glacial acetic acid (5 mL) at 0 °C during 5 min. with stirring. The solid thus precipitated on dilution with water was collected and recrystallized (ethanol) to give **25a-i** as white needles.

10-(p-Methoxyphenyl)-4-oxo-3-phenyl-1,2,3,4-tetrahydro-2-thioxocyclopenta-[5′,6′]pyrido[3′,2′:4,5]thieno[3,2-d]pyrimidine (26)

A mixture of **6d** (0.83 g, 0.002 mol) and carbon disulfide (3 mL) in pyridine (15 mL) was heated under reflux on a water bath for 8 hr. The product that separated was filtered, dried and recrystallized (acetic acid) as yellow needles of **26**.

2-Ethylthio-10-(p-methoxyphenyl)-3-phenylcyclopenta[5′,6′]pyrido [3′,2′:4,5]-thieno[3,2-d]pyrimidine-4(3H)-one (27)

A mixture of **26** (0.45 g, 0.001 mol), ethyl iodide (0.002 mol) and anhydrous sodium acetate (0.5 g) in ethanol (10 mL) was heated under reflux for one hour. The product which obtained was recrystallized (ethanol) to give **27** as pale yellow crystals.

2-Hydrazino-10-(p-methoxyphenyl)-3-phenylcyclopenta[5′,6′]pyrido [3′,2′:4,5]-thieno[3,2-d]pyrimidine-4(3H)-one (28)

A mixture of **26** (0.91 g, 0.002 mol) and hydrazine hydrate (6 mL) in pyridine (15 mL) was heated under reflux for 3 hr. The product which precipi-

tated on dilution with water was collected and crystallized (dioxane) as yellow crystals of **28**.

11-(p-Methoxyphenyl)-4-phenyl-s-triazolo[4″,3″-a]cyclopenta[5′,6′] pyrido-3′,2′:4,5]thieno[2,3-e]pyrimidine-5(4H)-one (29)

Compound **28** (0.45 g, 0.001 mol) in triethyl orthoformate (10 mL) was refluxed for 4 hr. On cooling, the precipitate that formed was collected and recrystallized (acetic acid) to give **29**.

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