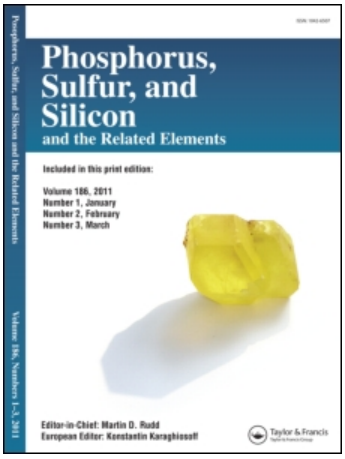

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Synthesis of Some Phenylazopyridothienopyrimidines

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SYNTHESIS OF SOME PHENYLAZOPYRIDOTHIENOPYRIMIDINES

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2-Mercapto-5-phenylazo-4,6-dimethylpyridine-3-carbonitrile (**3**) reacts with halocompounds to give S-alkylated derivatives (**4a–h**) which upon treatment with sod. ethoxide in ethanol, the thienopyridines (**5a–h**) was obtained. Compound (**5**) converted into pyridothienopyrimidines (**6a–e**) or (**7a,b**) boiling with triethyl orthoformate or acetic anhydride. Also it can be converted into pyridothienotriazine (**8a–d**) by treating with nitrous acid. Chloropyridothienopyrimidine (**10a,b**) was obtained by refluxing compound **6a** or **7a** with $POCl_3$.

Key words: Synthesis, reactions, thienopyridine, pyridothienopyrimidine, pyridothienotriazine.

INTRODUCTION

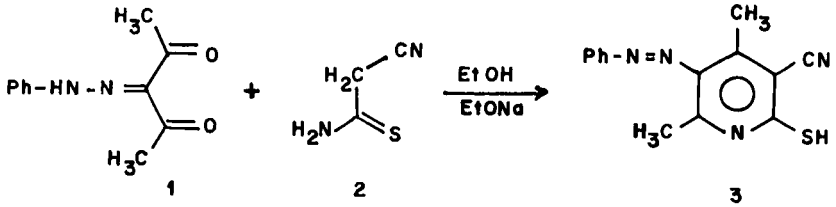
Pyrimidines have coupled a unique place and made a remarkable contribution to biological and medicinal chemistry. Various analogues of thiopurines have profound biological, antifungal, antiviral, insecticidal and miticidal.[1–3]

Also pyrimidines fused heterocycles are of importance in the field of medicinal chemistry thiazolopyrimidine for example have some analgesic activity and are devoid of cerebral system activity.[4]

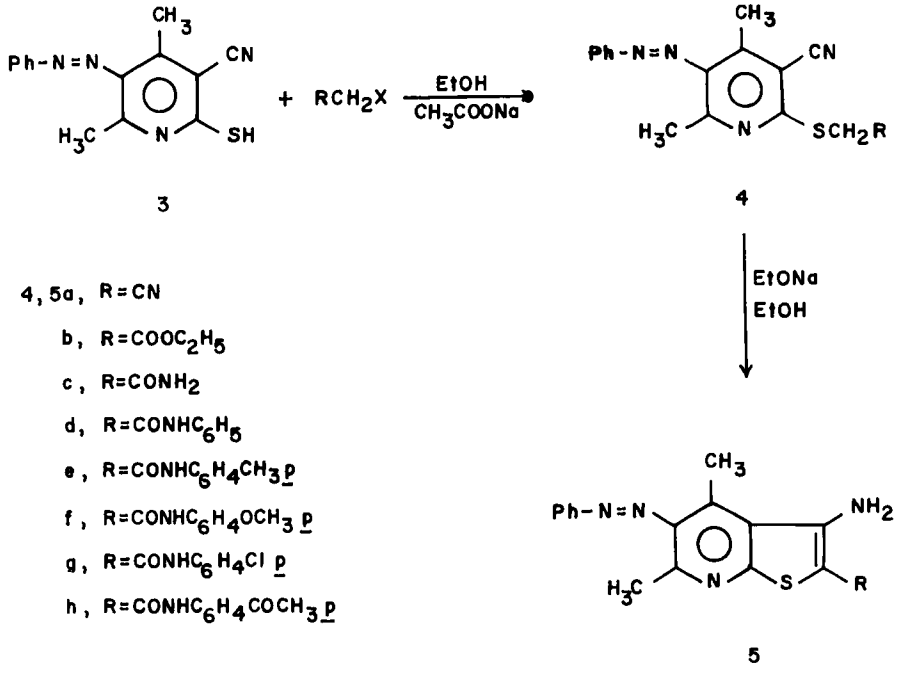
Many of the available thienopyridines have been evaluated pharmacologically and have been found to show activity against diabetes mellitus, as analgesics and antiinflammants.[5–8] Herein and in continuation of our work in the synthesis of thienopyridines,[9–12] we prepared some phenylazopyridothienopyrimidines hoping that may show biological activity.

RESULTS AND DISCUSSION

When phenylhydrazonoacetylacetone (**1**) were reacted with cyanothioacetamide (**2**) in ethanol containing an equivalent amount of sodium ethoxide, 2-mercapto-4,6-dimethyl-5-phenylazopyridine-3-carbonitrile (**3**) was obtained.



The mercaptocompound (**3**), when refluxed with halocompounds like, ethyl chloroacetate, chloroacetonitrile, chloroacetamide, or N-substituted chloroacetamides in ethanol and in the presence of sodium acetate or potassium carbonate, S-alkylation occurred to produce the corresponding S-substituted derivatives (**4a–h**). Compound **4** undergoes cyclization into thienopyridine derivatives (**5a–h**) upon treatment with sodium ethoxide in ethanol.



3

4

4, 5a, R=CN
b, R=COOC$_2$H$_5$
c, R=CONH$_2$
d, R=CONHC$_6$H$_5$
e, R=CONHC$_6$H$_4$CH$_3$ p
f, R=CONHC$_6$H$_4$OCH$_3$ p
g, R=CONHC$_6$H$_4$Cl p
h, R=CONHC$_6$H$_4$COCH$_3$ p

5

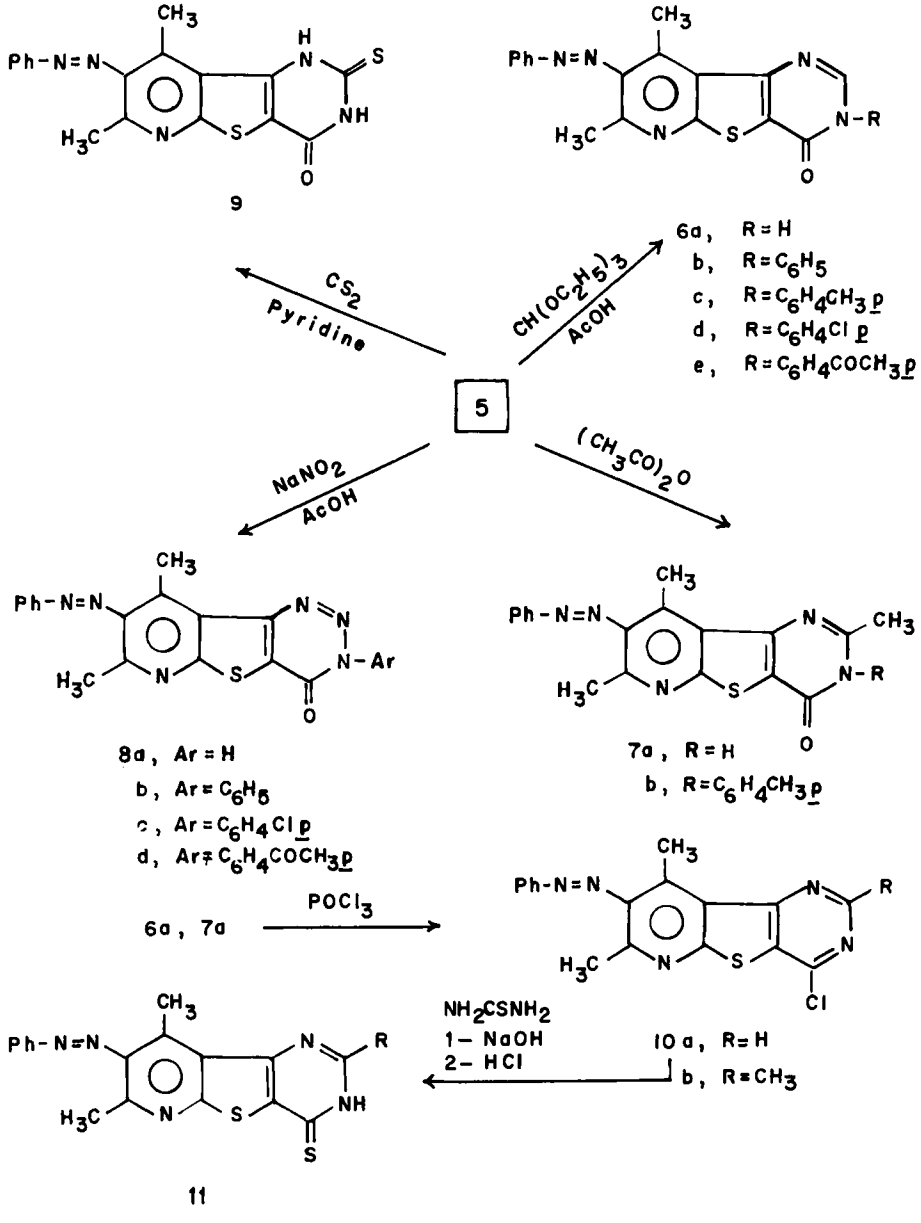
3-Amino-5-phenylazo-5,6-dimethyl thieno[2,3-b]pyridine-3-carboxamide derivatives (**5e–g**) when refluxed with triethyl orthoformate in ethanol in the presence of a few drops of acetic acid, 7,9-dimethyl-5-phenylazo-3-substituted pyrido-[3′,2′:4,5]-thieno[3,2-d]pyrimidin-4(3H)-one (**6a–e**) were obtained.

Also, 2,7,9-trimethyl-8-phenylazo-3-substituted pyrido-[3′,2′:4,5]thieno[2,3-d]pyrimidin-4-one (**7a,b**) resulted when compounds (**5c,e**) were refluxed in acetic anhydride.

When compounds (**5c–h**) were reacted with nitrous acid in acetic acid, 7,9-dimethyl-8-phenylazo pyrido[3′,2′:4,5]thieno-[2,3-d]-3-substituted triazin-4-one (**8a–d**) formed.

Also, 7,9-dimethyl-8-phenylazo-2-thiopyrido[3′,2′:4,5]-thieno[3,2-d]pyrimidin 2,4(1H, 3H)dione (**9**) was obtained by refluxing compound (**5c**) with carbon disulfide in pyridine.

When compound **6a** or **7a** was refluxed with $POCl_3$, the corresponding chloroarylazopyridothienopyrimidine (**10a,b**) was obtained which upon treatment with thiourea and subsequent treatment with sodium hydroxide and hydrochloric acid gave arylazopyridothienopyrimidinethione (**11**).



EXPERIMENTAL

Melting points are uncorrected and were determined on a Mel-Temp II melting point apparatus. IR spectra were recorded on Pye-Unicam SP 3-100 spectrophotometer using KBr Wafer technique. ^{1}H-NMR spectra were recorded on a Varian EM-390 90 MHz NMR spectrometer in the suitable deuterated solvent, using TMS as internal standard. Elemental analysis were determined on a Perkin-Elmer 240 C microanalyser.

2-Mercapto-4,6-dimethyl-5-phenylazopyridine-3-carbonitrile (**3**): It was prepared according to a procedure reported earlier in 70% yield, m.p. 235°C. lit.[13] m.p. 220°C.

TABLE I

Physical constants and analytical data of compounds (4–8)

Compound No.	R	M.P. C°	Yield %	Molecular formula	Analytical data calcd./found C	H	N	S	Cl
4a	CN	180	80	$C_{16}H_{13}N_5S$	62.54	4.23	22.80	10.42	-
					62.80	4.02	23.05	10.32	-
4b	$COOC_2H_5$	102	85	$C_{18}H_{18}N_4O_2S$	61.01	5.08	15.81	9.03	-
					60.89	4.86	16.00	8.80	-
4c	$CONH_2$	220-2	82	$C_{16}H_{15}N_5OS$	59.07	4.61	21.53	9.84	-
					58.82	4.82	21.28	9.62	-
4d	$CONHC_6H_5$	183-5	82	$C_{22}H_{19}N_5OS$	65.83	4.73	17.45	7.98	-
					66.05	5.00	17.60	7.81	-
4e	$CONHC_6H_4CH_3p$	190	80	$C_{23}H_{21}N_5OS$	66.50	5.06	16.86	7.71	-
					66.80	5.00	17.00	7.68	-
4f	$CONHC_6H_4OCH_3p$	200	84	$C_{23}H_{21}N_5O_2S$	64.03	4.87	16.24	7.42	-
					63.80	5.00	16.50	7.88	-
4g	$CONHC_6H_4Clp$	205	86	$C_{22}H_{18}ClN_5OS$	60.62	4.13	16.07	7.34	18.15
					60.82	4.00	15.82	7.50	17.90
4h	$CONHC_6H_4COCH_3P$	190	83	$C_{24}H_{21}N_5O_2S$	65.01	4.74	15.80	7.22	-
					64.83	4.52	16.00	7.40	-
5a	CN	210	85	$C_{16}H_{13}N_5S$	62.54	4.23	22.80	10.42	-
					62.68	4.50	23.00	10.50	-
5b	$COOC_2H_5$	170	90	$C_{18}H_{18}N_4O_2S$	61.01	5.08	15.81	9.03	-
					60.80	4.82	16.04	8.85	-
5c	$CONH_2$	234	88	$C_{16}H_{15}N_5OS$	59.07	4.61	21.53	9.84	-
					58.80	4.78	21.70	9.90	-
5d	$CONHC_6H_5$	240	90	$C_{22}H_{19}N_5OS$	65.83	4.73	17.45	7.98	-
					65.60	4.62	17.60	8.15	-
5e	$CONHC_6H_4CH_3p$	270	92	$C_{23}H_{21}N_5OS$	66.50	5.06	16.86	7.71	-
					66.35	4.80	17.15	7.92	-
5f	$CONHC_6H_4OCH_3p$	275	85	$C_{23}H_{21}N_5O_2S$	64.03	4.87	16.24	7.42	-
					63.90	5.12	16.05	7.60	-
5g	$CONHC_6H_4Clp$	307	90	$C_{22}H_{18}ClN_5OS$	60.62	4.13	16.07	7.34	8.15
					60.45	3.85	15.89	7.52	7.95
5h	$CONHC_6H_4COCH_3p$	265	88	$C_{24}H_{21}N_5O_2S$	65.01	4.74	15.80	7.22	-
					65.25	4.68	16.00	7.00	-

TABLE I (*Continued*)

Compound No.	R	M.P. C°	Yield %	Molecular formula	Analytical data calcd./found C	H	N	S	Cl
6a	H	305	75	$C_{17}H_{13}N_5OS$	54.35	4.52	24.39	11.14	-
					54.52	4.38	24.30	11.00	-
6b	C_6H_5	275	70	$C_{23}H_{17}N_5OS$	67.15	4.13	17.03	7.78	-
					66.95	4.30	16.85	7.88	-
6c	$C_6H_4CH_3$p	>300	72	$C_{24}H_{19}N_5OS$	67.76	4.47	16.47	7.52	-
					66.00	4.30	16.60	7.60	-
6d	C_6H_4Clp	270	75	$C_{23}H_{16}ClN_5OS$	61.95	3.59	15.71	7.18	7.96
					62.10	3.68	15.60	7.00	8.10
6e	$C_6H_4COCH_3$p	284	78	$C_{25}H_{19}N_5O_2S$	66.22	4.19	15.45	7.06	-
					66.00	4.05	15.67	6.90	-
7a	H	300	65	$C_{18}H_{15}N_5OS$	61.89	4.29	20.05	9.16	-
					62.00	4.05	19.80	7.50	-
7b	$C_6H_4CH_3$p	>300	67	$C_{25}H_{21}N_5OS$	68.33	4.78	15.94	7.28	-
					68.15	5.00	16.10	7.05	-
8a	H	215	85	$C_{16}H_{12}N_6OS$	57.14	3.57	25.00	9.52	-
					56.92	3.64	24.85	9.71	-
8b	C_6H_5	230	84	$C_{22}H_{16}N_6OS$	64.07	3.88	20.38	7.76	-
					63.88	4.07	20.50	7.80	-
8c	C_6H_4Cl	270	80	$C_{22}H_{15}ClN_6OS$	59.12	3.35	18.81	7.16	7.95
					59.00	3.30	19.00	7.00	8.10
8d	$C_6H_4COCH_3$	284	78	$C_{24}H_{18}N_6O_2S$	63.43	3.96	18.50	7.04	-
					63.32	4.10	18.68	6.88	-

2-Substituted mercapto-4,6-dimethyl-5-phenylazopyridine-3-carbonitrile (**4a–h**). A mixture of mercaptopyridine (**3**) (0.01 mole), the appropriate halocompound (0.01 mole) and sodium acetate (0.012 mole) in ethanol (30 ml) was refluxed for one hour. After cooling the solid was collected, washed with water and recrystallized from ethanol. The physical constants and spectral data of compound **4a–h** are listed in Table I and Table II.

3-Amino, 4,6-dimethyl-5-phenylazothieno[2,3-b]pyridine-2-substituted carboxamide (**5**): To a sample of compound (**4**) (1 gm) in ethanol absolute (20 ml), a few drops of sodium ethoxide in ethanol was added and refluxed for ½ hour, after cooling the solid was collected and recrystallized from ethanol. The physical constants and spectral data are listed in Table I and Table II.

7,9-Dimethyl-5-phenylazo-3-substituted pyrido[3',2':4,5]thieno-[3,2-d]pyrimidin-4(3H)-one (**6**): To a mixture of compound (**5**) (0.005 mole) and triethyl orthoformate (1 ml) in ethanol (20 ml) a few drops of acetic acid was added and refluxed for 4 hours. After cooling the solid was collected and recrystalized from acetic acid. The physical properties and spectral data of compound (**6**) were listed in Table I and Table II.

TABLE II

Spectral data of compounds (4–8)

Compound No.	IR	^{1}H NMR
4a	ν22200, 221 5 (2C≡N).	in DMSO-d_6: δ2.5, δ2.7(2s, 6H, $2CH_3$), δ4.2 (s, 2H, CH_2), δ7.3-7.8 (m, 5H, Ar-H).
4b	ν22200 cm^{-1} (C≡N), and 1700 cm^{-1} (C=0).	in $CDCl_3$: δ1.3(t, 3H, CH_3), δ2.5, δ2.7(2s, 6H, $2CH_3$), δ4.1(q, 2H, CH_2), δ3.9(s, 2H, CH_2) and δ7.3-7.8(m, 5H, Ar-H).
4c	ν3350 cm^{-1}, 3220 cm^{-1} (NH_2), 2230 cm^{-1} (C N) and 1670 cm^{-1} (C=0).	in DMSO-d_6: δ2.5, δ2.7(2s, 6H, $2CH_3$), δ3.9 (s, 2H, CH_2), δ7.3-7.8(m, 5H, Ar-H) and (s, 2H, NH_2).
4d	ν3460 cm^{-1} (NH), ν2230 cm^{-1} (C≡N), and ν1640 cm^{-1} (C=0).	in DMSO-d_6: δ2.55, δ2.75(2s, 6H, $2CH_3$), δ3.95 (s, 2H, CH_2), δ7.3-8.5(m, 10H, Ar-H) and 11.3(s, 1H, NH).
4e	ν3360 cm^{-1} (NH), ν2230 cm^{-1} (C≡N), and ν1650 cm^{-1} (C=0).	in CF_3COOH: δ2.5, δ2.7, δ3.3(3s, 9H, $3CH_3$), δ3.8(s, 2H, CH_2), δ7.4-8.5(m, 9H, Ar-H) and δ9.8(s, 1H, NH).
4f	ν3260 cm^{-1} (NH), ν2220 cm^{-1} and 1670 cm^{-1} (C=0)	in DMSO-d_6: δ2.5, 2.7, 3.3(3s, 9H, $3CH_3$), δ3.9(s, 2H, CH_2), δ7.4-8.5(m, 9H, Ar-H), and 11.3(s, 1H, NH).
4g	ν3360 cm^{-1} (NH), ν2230 cm^{-1} (C≡N), ν1670 cm^{-1} (C=0).	in DMSO-d_6: δ2.5, δ2.75(2s, 6H, $2CH_3$), δ3.9 (s, 2H, CH_2), δ7.5-8.5(m, 9H, Ar-H), δ10.5 (s, 1H, NH).
4h	ν3300 cm^{-1} (NH), ν2220 cm^{-1} (C≡N), ν1700-1640 cm^{-1} (2C=0).	in DMSO-d_6: δ2.5,2.7,δ3.5(3s, 9H, $3CH_3$), δ3.9 (s, 2H, CH_2), δ7.4-8.6(m, 9H, Ar-H), δ11.3 (s, 1H, NH).
5a	ν3320-3200 cm^{-1} (NH_2), 2220 cm^{-1}(C≡N).	in DMSO-d_6: δ2.5, 2.7(2s, 6H, $2CH_3$), δ7.2 (s, 2H, NH_2), δ7.5-8.3(m, 5H, Ar-H).
5b	ν3450, 3360 cm^{-1} (NH_2), 1660 cm^{-1} (C=0).	1.3(t, 3H, CH_3), δ2.5, δ2.7(2s, 6H, $2CH_3$), δ4.00(q, 2H, CH_2), δ7.1(s, 2H, NH_2), δ7.3-8.2(m, 5H, Ar-H).
5c	ν3500-3200 cm^{-1} ($2NH_2$), 1650 cm^{-1} (C=0).	in CF_3COOH: δ2.5, δ2.7(2s, 6H, $2CH_3$), δ7.3-8.0 (m, 5H, Ar-H).
5d	ν3450-3340 cm^{-1} (NH, NH_2), 1650 (C=0).	in CF_3COOH: δ2.5,δ2.7(2s, 6H, $2CH_3$), δ7.4-8.5 (m, 10H, Ar-H), 9.6(s, 1H, NH).
5e	ν3470-3250 cm^{-1} (NH, NH_2), 1650 cm^{-1} (C=0).	in CF_3COOH: δ2.5, 2.75, 3.2(2s, 9H, $3CH_3$), δ7.3-8.6(m, 9H, Ar-H).
5f	ν3450-3250 cm^{-1} (NH, NH_2), 1650 cm^{-1} (C=0).	in DMSO-d_6: δ2.5, δ2.7, δ3.3(3s, 9H, $3CH_3$), δ7.1(s, 2H, NH_2), δ7.4-8.5(m, 9H, Ar-H),δ11.3 (s, 1H, NH).

TABLE II (*Continued*)

Compound No.	IR	^{1}H NMR
5g	ν3480-3300 cm^{-1} (NH, NH_2), 1650 cm^{-1} (C=O).	in DMSO-d_6: δ2.5, δ2.7(2s, 6H, $2CH_3$), δ7.1 (s, 2H, NH_2), δ7.4-8.6(m, 9H, Ar-H), and δ11.3(s, 1H, NH).
5h	ν3500-3300 cm^{-1} (NH, NH_2), 1690-1640 cm^{-1} (2C=O).	in CF_3COOH: δ2.5, δ2.7, δ3.3(3s, 9H, $3CH_3$), δ7.3-8.6(m, 9H, Ar-H).
6a	ν3350 cm^{-1} (NH), ν1670 cm^{-1} (C=O).	in CF_3COOH: δ2.5, δ2.75(2s, 6H, $2CH_3$), δ7.4-7.9(m, 5H, Ar-H), δ8.9(s, 1H, CH).
6b	ν1680 cm^{-1} (C=O).	in CF_3COOH: δ2.5, δ2.8(2s, 6H, $2CH_3$), δ7.4-8.5(m, 10H, Ar-H) and δ8.9(s, 1H, CH pyrimidine).
6c	ν1680 cm^{-1} (C=O).	in CF_3COOH: δ2.5, δ2.7, δ3.3 (3s, 9H, $3CH_3$), δ7.35-8.5(m, 9H, Ar-H) and δ8.85(s, 1H, CH pyrimidine).
6d	ν1680 cm^{-1} (C=O).	in CF_3COOH: δ2.5, δ2.8(2s, 6H, $2CH_3$), δ7.3-8.5(m, 9H, Ar-H) and δ8.9(s, 1H, CH pyrimidine).
6e	ν1700-1660 cm^{-1} (2CO).	in CF_3COOH: δ2.5, δ2.7, δ3.4(3s, 9H, $3CH_3$), δ7.3-8.5(m, 9H, Ar-H), δ8.9(s, 1H, CH pyrimidine).
7a	ν3350 cm^{-1} (NH), 1680 cm^{-1} (C=O).	in CF_3COOH: δ2.5, δ2.75, δ3.2(3s, 9H, $3CH_3$), δ7.3-7.9(m, 5H, Ar-H).
7b	ν1680 cm^{-1} (C=O).	in CF_3COOH: δ2.5, δ2.7, δ3.3, δ3.4(4s, 12H, $4CH_3$), δ7.3-8.5(m, 9H, Ar-H).
8a	ν3250 cm^{-1} (NH), ν1670 cm^{-1} (C=O).	in DMSO-d_6: δ2.5, δ2.7(2s, 6H, $2CH_3$), δ7.3-7.9 (m, 5H, Ar-H), δ11.3(s, 1H, NH).
8b	ν1680 cm^{-1} (C=O).	in DMSO-d_6: δ2.5, δ2.7(2s, 6H, $2CH_3$), δ7.3-8.5 (m, 10H, Ar-H).
8c	ν1680 cm^{-1} (C=O).	in DMSO-d_6: δ2.5, δ2.7(2s, 6H, $2CH_3$), δ7.3-8.5 (m, 9H, Ar-H).
8d	ν1680-1650 cm^{-1} (2CO).	in DMSO-d_6: δ2.5, δ2.7, δ3.3(3s, 9H, $3CH_3$) and δ7.3-8.5(Ar-H).

2,7,9-Trimethyl-8-phenylazo-3-substituted pyrido[3',2':4,5]thieno-[3,2-d]pyrimidin-4-one (**7**):

General procedure: A sample of compound (**5**) (0.5 gm) in acetic anhydride (5 ml) was refluxed for 5 hours. After cooling the solid was collected and recrystallized from acetic acid. The physical constants and spectral data of compound **7** are listed in Table I and Table II.

7,9-Dimethyl-8-phenylazo-3-substituted pyrido[3',2':4,5]thieno-[2,3-d]-triazin-4-one (**8**):

General procedure: To a cold suspension of compound (**5**) (0.005 mole) in acetic acid (20 ml) a sodium nitrite solution (½ gm. in 2 ml H_2O) was added dropwise with stirring. The stirring was continued for ½ hour and let stand for one hour. The precipitate was collected and recrystallized from ethanol. The physical constants and spectral data for compound (**8**) were listed in Table I and Table II.

7,9-Dimethyl-8-phenylazo-2-thiopyrido[3',2':4,5]thieno[2,3-d]-pyrimidin-2,4(1H,3H)dione (**9**): A mixture of compound **5c** (1.00 gm) and carbon disulphide (2 ml) in pyridine (10 ml) was refluxed for 10 hours. After cooling the solid was collected and recrystallized from dioxane to give yellow crystals in 60% yield, m.p. > 300.

Anal. Calcd. for: $C_{17}H_{13}N_5OS$: C, 60.89; H, 3.88; N, 20.89; S, 9.55%
Found: C, 61.10; H, 4.04; N, 20.78; S, 9.42%.
IR: ν3450, ν3250 cm^{-1} (2NH), ν1650 cm^{-1} (C=O).
^1H NMR in CF_3 COOH: δ2.5, 2.7 (2s, 6H, $2CH_3$), δ7.3–7.9 (m, 5H, Ar—H).

(7,9-Dimethyl or 2,7,9-trimethyl)-4-chloro-8-phenylazopyrido-[3',2':4,5]thienopyrimidine (**10a,b**):

General procedure: A sample of compound **6a** or **7a** (0.5 gm) in phosphorus oxychloride (5 ml) was refluxed for 2 hours. After cooling the reaction mixture was poured into an ice/water mixture. The solid was filtered off and recrystallized from ethanol as buff crystals.

10a: Produced 85% yield, m.p. 170°C.
Anal. Calcd. for: $C_{17}H_{12}ClN_5S$: C, 57.70; H, 3.39; N, 19.80; S, 9.05; Cl, 10.04%
Found: C, 57.80; H, 3.50; N, 19.66; S, 8.85; Cl, 9.85%.
IR: showed the disappearance of band characteristic for (C=O).
^1H NMR in DMSO-d_6: δ2.5, δ2.7 (2s, 6H, $2CH_3$), δ7.3–7.9 (m, 5H, Ar—H), and δ8.8 (s, 1H, CH-pyrimidine).

10b: Produced in 86% yield, m.p. 178°C.
Anal. Calcd. for: $C_{18}H_{14}ClN_5S$: C, 58.77; H, 3.80; Cl, 9.65; N, 19.04; S, 8.70%
Found: C, 59.00; H, 4.04; Cl, 9.48; N, 18.86; S, 8.54%.
IR showed the disappearance of band characteristic for (C=O).
^1H NMR in DMSO-d_6: δ2.5, δ2.7, δ3.2 (3s, 9H, 3CH), δ7.3–7.9 (m, 5H, Ar—H).

(7,9-Dimethyl or 2,7,9-trimethyl)-8-phenylazopyrido[3',2':4,5]-thienopyrimidin-4-thione (**11a,b**):

General procedure: A mixture of compound **10a** or **10b** (0.001 mole) and thiourea (0.005 mole) in ethanol (20 ml) was refluxed for 2 hours or until the thiourenium salt has precipitated. Then the reaction mixture was cooled, sodium hydroxide solution 20 ml (10%) was added and the mixture was warmed for 5 minutes. After acidification with hydrochloric acid the precipitate was collected and recrystallized from dioxane to give yellow crystals.

11a: Produced in 82% yield, m.p. > 300°C.
Anal. Calcd. for: $C_{17}H_{13}N_5S_2$: C, 58.11; H, 3.70; N, 19.94; S, 9.11%
Found: C, 57.90; H, 3.95; N, 20.15; S, 8.88%.
IR: ν3350 cm^{-1} (NH), and 1450 cm^{-1} (C=S). ^1H NMR in CF_3COOH: δ2.5, δ2.7 (2s, 6H, $2CH_3$), δ7.3–8.0 (m, 5H, Ar—H), and 8.9 (s, 1H, CH pyrimidine).

11b Produced in 86% yield, m.p. > 300°C.
Anal. Calcd. for: $C_{18}H_{15}N_5S_2$: C, 59.17; H, 4.10; N, 19.17; S, 8.76%
Found: C, 59.00; H, 3.90; N, 18.95; S, 9.00%.
IR: ν3300 cm^{-1} (NH), and ν1450 cm^{-1} (C=S). ^1H NMR in CF_3COOH: δ2.5, δ2.7, δ3.2 (3s, 9H, $3CH_3$), and 7.3–8.0 (m, 5H, Ar—H).

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