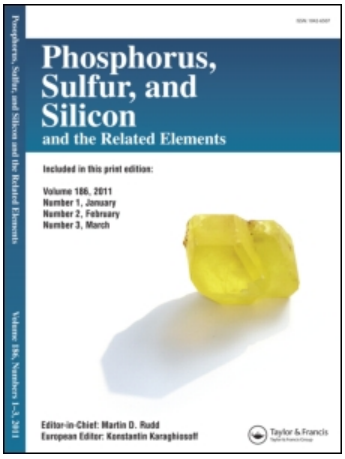

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Carbon Disulfide in Heterocyclic Organic Synthesis; Synthesis of Polyfunctionally Substituted Sulfur and Nitrogen Heteroaromatics

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Carbon Disulfide in Heterocyclic Organic Synthesis; Synthesis of Polyfunctionally Substituted Sulfur and Nitrogen Heteroaromatics

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*Reaction of cayno acetamdie with carbon disulphide in alkaline medium, e.g., potassium hydroxide, or potassium ter.butoxide gave the gem-dithiol (**1**). While in presence of ammonium hydroxide the ammonium salt (**2**) was obtained. Reactions of (**1**) or (**2**) with different reagents in different mediums gave pyridine, pyrimidines, thiophene, and thiopyranones derivatives.*

Keywords Carbon disulphide; keten thioacetals; sulfur chemistry; thioacetals

RESULTS AND DISCUSSION

Ketene gem-dithols have proved useful starting materials for a variety of heterocyclic and homocyclic aromatic compounds.[1–5] Some of its derivatives were found to possess wide applications. It have an antihepatotoxic in preventive curative and curative tests as shown by measuring serum glutamate-oxalacetate transaminase due to radical trapping formation of the thiol group.[6] Also, some derivatives were found to possess activities as herbicidal agents against field and aquatic weeds as well as mullscicides.[7] Moreover, the reactivity are extended to include nematocide,[7] baceriocide,[7] gungicide,[7] and antidates against ionizing radiations.[7–9]

An extensive work has been reported on the synthesis of some heteroaromatics. Although ketene gem- dithioacetals has been extensively utilized in organic synthesis, little have been reported on the utility of this versatile reagents (**1**) or (**2**) for building up a series of new compounds.

In continuation to our work on the behavior of ketene gem-dithiols,[10–12] the precursor (**1**) could be obtained on reacting cayno

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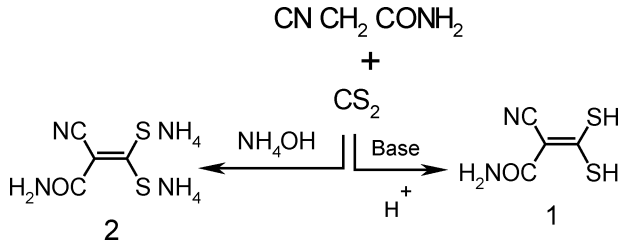
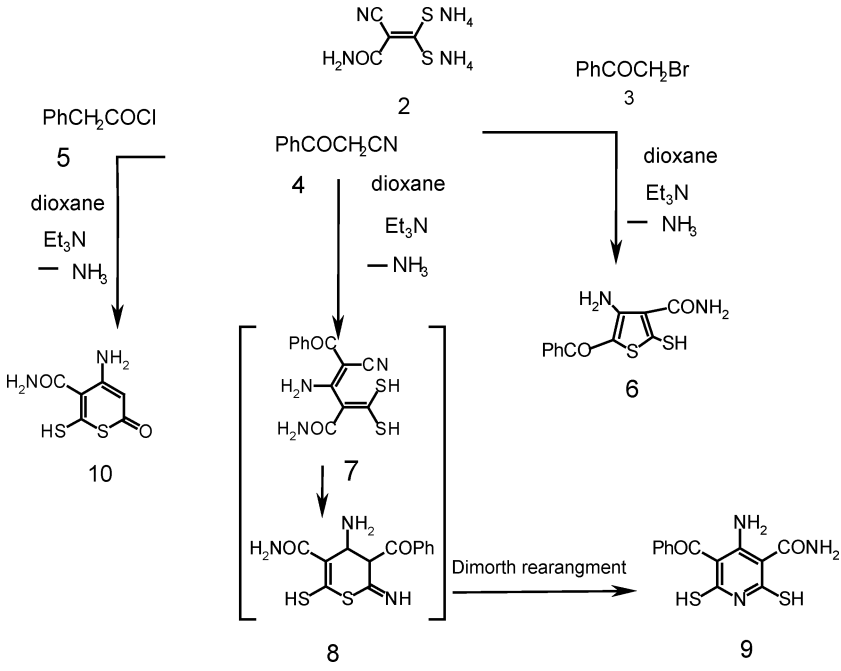


SCHEME 1

acetamide with carbon disulphide in a base (e.g., NaOH, KOH, KO.*ter*Bu) in aqueous medium. Using ammonium hydroxide in place of the above bases, precursor (**2**) was prepared (Scheme 1). On boiling (**2**) in alkaline aqueous medium, the precursor (**1**) was isolated. Reactions of (**2**) with some other reagents e.g., ω-bromo—acetophenone (**3**), benzoyl acetonitrile (**4**), and/or with phenylacetyl chloride (**5**) in boiling dioxane in the presence of triethylamine as a catalyst ,the poly functionally substituted thiophene (**6**), nicotinamide (**9**), and thiopyranone (**10**) derivatives (Scheme 2)



SCHEME 2

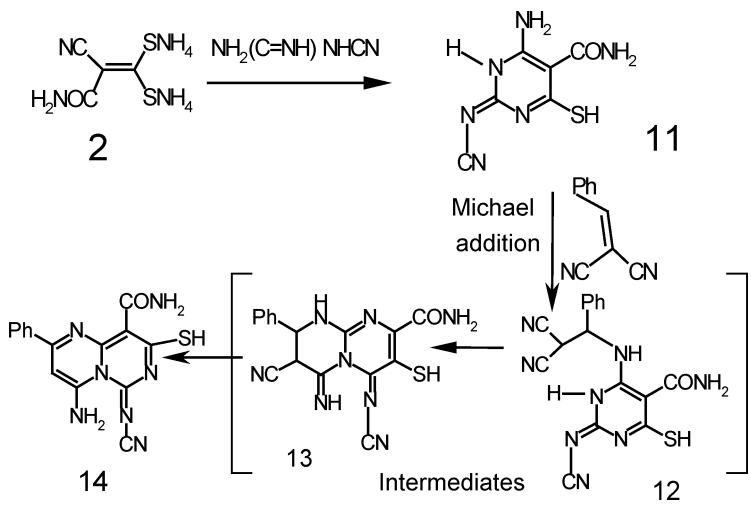
Formation of (**9**) is presumably proceeded via intermediates (**7** and **8**), followed by Dimorth rearrangement. Structure assignment of the products (**6**, **9**, and **10**) was supported by analytical and spectral measurements.

Disappearance of a peak for cayno group and appearance of a peak for amino group confirmed the structures (**6** and **9**). H^1nmr revealed signals that proved presence of the phenyl, sulfohydyl and amidic amino protons, in addition to a broad signal for amino groups.

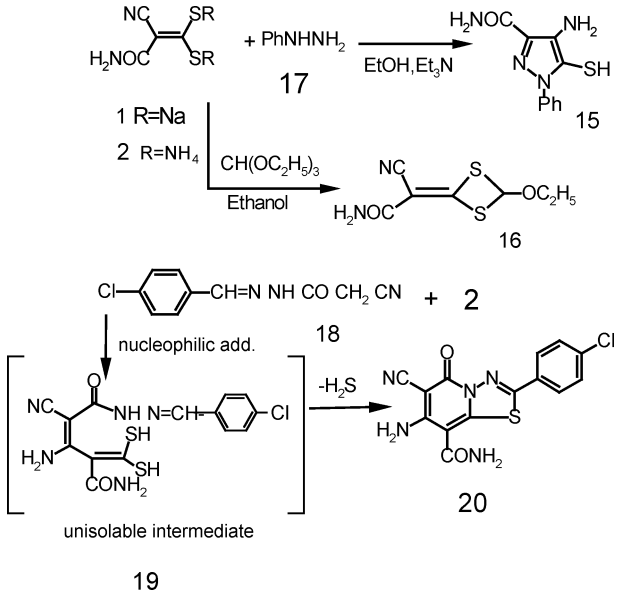
Furthermore, on boiling (**5**) with (**2**) gave the thiopyranone derivative (**10**). Moreover, behavior of (**2**) towards dicyano diamide, in boiling ethanol in the presence of a catalytic amount of triethylamine was studied and gave the polyfunctionlly substituted pyrimidine derivative (**11**). Treating (**11**) with benzylidine malono nitrle in boiling ethanol in the presence of a catalytic amount of triethylamine could form the pyridopyrimidine(**14**) (Scheme 3).

The reaction pathway was assumed to proceed through Michael addition as a first step followed by nucleophilic addition to form the intermediates (**12** and **13**), respectively.

Spectral and analytical measurements gave a satesfy data to confirm structures of (**11**) and (**14**). Reaction of (**2**) with phenyl hydazine in boiling ethanol in the presence of a catalytic amount of triethylamine was also reported to give the pyrazolone derivative (**15**). Substrate (**2**) when reacted with triethyl orthformate in boiling ethanol, the cyclic dithiol derivative (**16**) was produced. Meanwhile, the reaction of (**2**) with



SCHEME 3



SCHEME 4

azmethane derivative (**18**) in boiling dioxane and a catalytic ammount of triethylamine, favored formation of the condensed system (**20**) via formation of the intermediate (**19**) (Scheme 4).

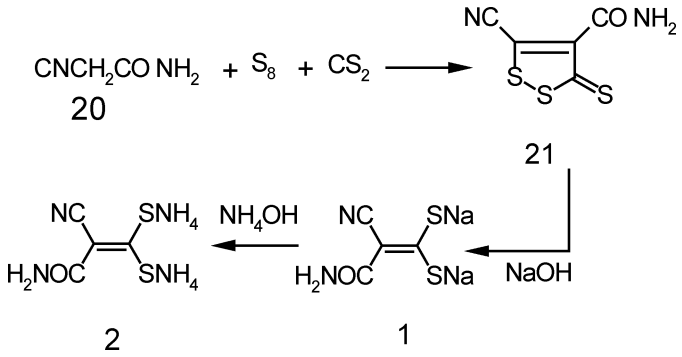
The obtained spectral data ruled out any other probabilities for the reactions of (**1**) or (**2**) with phenyl hydrazine or with triethylorthoformate, since the data offer signals that supported structures of (**15–20**).

The ketene gem-dithiol (**1**) could also be obtained from the trithione derivative (**21**) via reaction of cayno actamide (**20**) with carbon disulfide and elemental sulfur, followed by ring opening under the influence of sodium hydroxide (Scheme 5).

An interesting novel method for the synthesis of substituted pyrazolopyrimidine derivative (**24**) was proceeded via the reaction of the precursor (**22**)with carbon disulfide in alkaline medium. On treating (**23**) with thiourea in a basic medium, the pyrazolopyrimidine derivative (**24**) was isolated (Scheme 6).

Dimerization of (**2**) resulted in formation of the trithione derivative (disaurine) (**25**), which on boiling in ethanol looses a sulfur atom and finally the cyclic ditheitane (**26**) derivative was formed (Scheme 7).

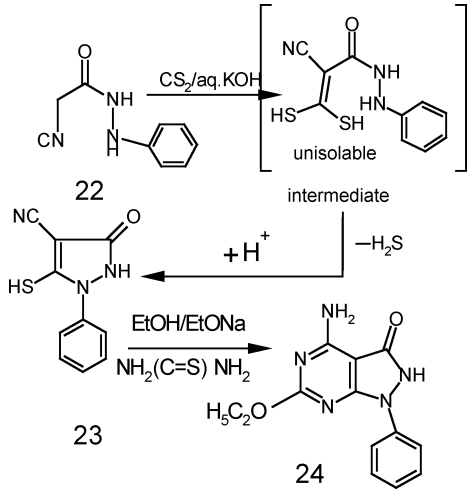
Furthermore, reaction of substrate (**27**) with barbituric acid (**28**) in boiling ethanol/sodium ethoxide, resulted in formation of pyrimidine derivative (**30**) via formation of the unisolable intermediate (**29**). The



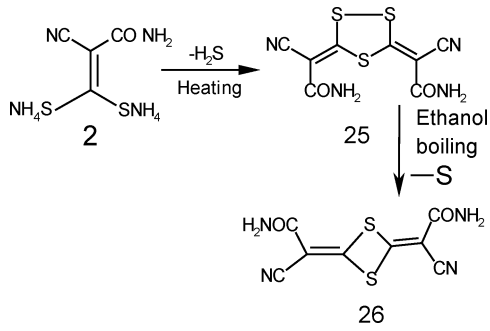
SCHEME 5

reaction is assumed to proceed via condensation of keto group in (**27**) with methylene moiety in (**28**) follweed by water elimination. Meanwhile the reaction of 1,3-cyclohexane dione (**31**) with (**27**) in boling methylne chloride in presence of triethylamine yielded product (**33**) (Scheme 8).

Formation of (**33**) was assumed to proceed via Michael addition to ethylenic moiety in (**27**) followed by water elimnation. Then the second molecule (**27**) reacted with carbonyl moiety followed by water elimnation. Obtained spectral and elemental data supported the structure (**32**) and ruled out any other assumed structures. Benzylidene thiosemicarbazide (**34**) was also subjected to reaction with (**27**)



SCHEME 6



SCHEME 7

in boiling ethanol/sodium ethoxide and gave the pyrimidine derivative (**36**) (Scheme 9).

EXPERIMENTAL

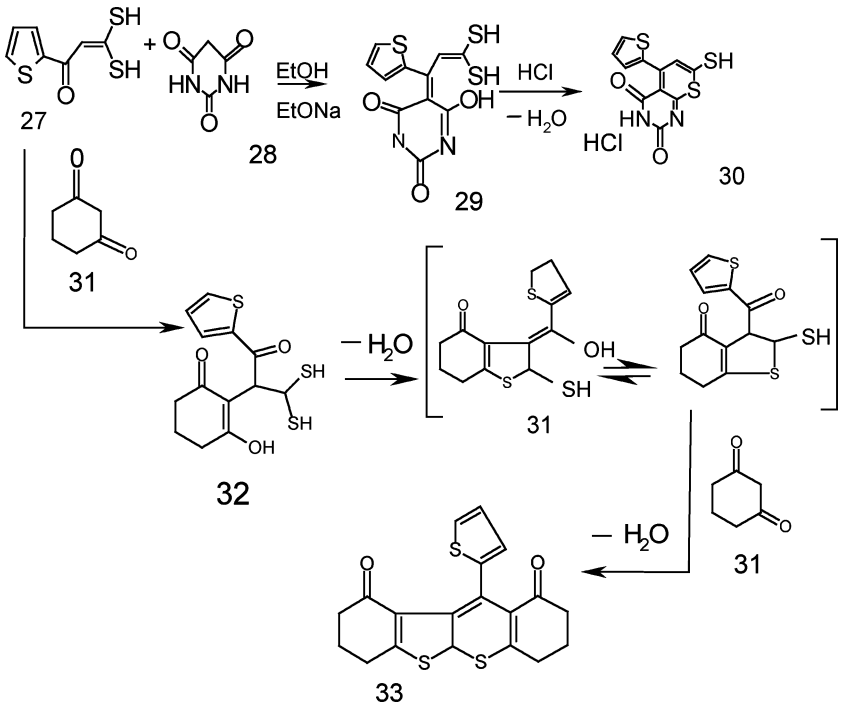
M.Ps are uncorrected and measured by open capillary tubes on a hot plate surface IR spectrum (K Br) were recorded on Shimadzu part No: 200-91506 spectrophotometer. H^1NMR spectra were recorded on Varian–Gemini 200-200MHz (DMSO; Mass spectra were measured on Shimadzu SQ 100 (At Cairo University, analysis unit). H^1NMR spectra (DMSO or CDCl3) were recorded on BRUKER, MCKCYC 53.001AU PROG: 300-MHz (At the Chemistry Department, University of Manitoba, Canada. Elemental analysis values were determined at Cairo University (Egypt) laboratories.

3,3-Dimercapto 2-cyanmide 2 and 3

1 The gem-dithiol (**2**) was prepared according to the reported procedure via action of CS_2/K*ter*. Butoxide in benzene on cayno acetamide in place of NaOH in water, m.p. 112°C (reported m.p. 112°C) yield 89.67%.
2 Gem-dithiol (**1**) could also be prepared from reaction of CS_2 with cayno-acetamide in presence of NH_4OH in cold condition (at room temperature), m.p. 143°C (reported 142°C) yield 88.57%.
3 Heating (**2**) in aqueous NaOH gave (**1**) 87.94%.

General Procedure for the Synthesis of 6, 9, and 10

To the reagents, bromoacetophenone (**3**), benzoyl actonirile (**4**), and phenyl-acetylchloride (**5**) (0.01 mol) was added an equimolar ratio from



SCHEME 8

(**2**) (0.01mol) in dioxane (100 ml) contained a catlytic ammount of Et_3N(2 drops), then the reaction mixtures were heated under reflux for 3 h. On concentration the solvent to the third of its volume and cooling , the precipitated solid products were gathered and recrystallized from DMF.

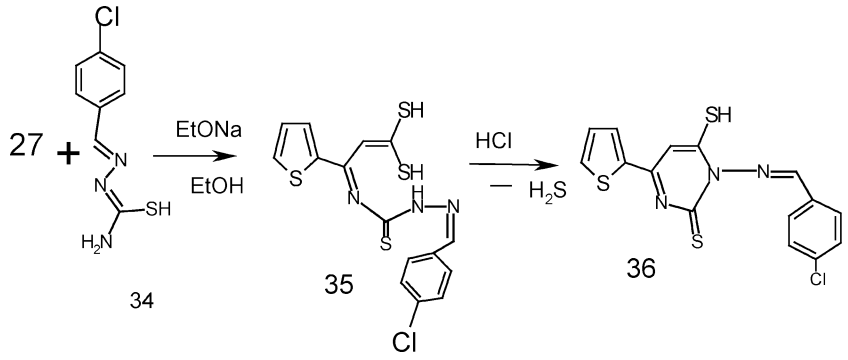
3-Amino 4-Benzoyl 5-Mercapto-Thiophene 2-Carboxylic Acid Amide (6)

Obtained as a brown crystals, m.p. 132 c° m/e 278.02. Analysis for $C_{12}H_{10}N_2O_2S_2$ (278.02) yield; 68.51%. Required % C, 51,78, H, 3.62 N, 10.06; S,23.04, Found, C, 51.77 ;H,3.7; N,11.0.

H^1NMR δ m, 7.45–7.81 5H; C_6H_5; s, 6. 8.1; 2H, $CONH_2$; s, 4.66; 2H NH_2; s, 3.22, 1H, SH.

4-Amino 5-Benzoyl 2,6-Dimercapto Nicotinamide 9.

Obtained as a brown crystals.yield; 77.92% ,M.p. 183°C Analysis for $C_{13}H_{11}N_3O_2S_2$ (305.38) Requires % C,51.13, H,3.63; N,13.76; S,21.00. found %, C 51.2; H, 3.7; N, 13.79, S, 20.76. H^1 nmr, δ m,



SCHEME 9

7.44–8.11, 5H C_6H_5, s, 5,57 2H NH_2;s,5.86 2H, amidic NH_2, s 2.86, 2H 2SH.

4-Amino 2-Mercapto 6-Oxo 6H Thiopyran 3-Carboxylic Acid Amid 10

Collected as a dark brown powder,yield 78.64%; m.p. 212°C. Analysis for $C_6H_6N_2O_2S_2$ (202.25) Required % C, 35.63; H, 2.99; N, 13.85; S, 31.71; Found% C, 35.71; H, 3.11; N,14.0;s, 32.22. Hnmr δ s 6.12 1H CH, s, 5.43 2H, $CONH_2$.

6-Amino 2-Caynoimino 4-Mercapto 1,2-Dihydroppyrimidine 5-Carboxylic Acid Amide 11.

A mixture of (**2**) (0.01 mol, 1.94 g) and caynamide dimer (01.0, 0.084 mol) in absolute ethanol (75 ml) contained (1ml,Et_3N) was heated under reflux for 2 h. After cooling the precipitated product was filtered and recrystallized from DMF as a dark red crystals. Yield 82.43%, m.p. 266°C. Analysis for $C_6H_8N_6OS$ (212.23). Required % C, 33. 96; H, 3.80; N, 39.60; S, 15.11, Found % C, 34.01; H, 4.00; N,40.00; S, 15.53.; I.R2228 /cm^{-1} (CN), 1695/cm^{-1}(C=O amide). H^1nmr δ; s, 8.57 2H NH_2CO s,4.23 2H NH_2, s, 3.87 1H SH.

4-Amino 6-Caynoimino 8-Mercapto 2-Phenyl 6H Pyrimido[1,6-a] Pyrimidine 9-Carboxylic Acid Amide 14

To 11 (0.01 mol, 3.38 gm) in absolute ethanol (100 ml) contained Et_3N (1 ml), benzylidin-malono nitrle (0.01 mol, 1.54 g) was added while heating under reflux for 4 hours (until evolution of H_2S ceased). On cooling, the separated crop was filtered off and recrystallized from DMF yield

76.53%. Analysis for $C_{15}H_{11}N_7OS$ (337. 36) yield 68.32%. Calculated % C, 53.40; H,3.29; N, 29.06; S,9.50 found % C, 53.47; H, 3.32; N,30.00; S, 10.11. IR, 2235 cm^{-1} (CN), H^1NMR, δ ; s, 8.12 2H, CO NH_2m,7.1–7.87 5H C_6H_5; s, 5.98 1H,CH=C s, .4.87 2H NH_2; s,1.66 1H SH.

4-Amino 1-Phenyl 5-Mercapto 1-H Pyrazole 3-Carboxylic Acid Amide 15

To a solution of 2 (0.01 mol; 1.94 g) in absolute ethanol (75 ml) was added to phenylhydrazine (0.013 mol, 1.4 ml) in absolute ethanol (25 ml) in presence of Et_3N (1ml). The reaction mixture was then heated under reflux for 2.5 h, followed by cooling to room temperature. The separated crop was recrystallized from ethanol to give a pale yellow crystals of m.p. 129°C. Analysis for $C_{10}H_{10}N_4OS$ (234.28); yield 87.62%. Required %, C, 51.27; H, 4.30; N, 23.91; S, 13.69, Found % C, 51. 29; H, 4.33; N, 24.00; S, 14.00. I.R/ cm^{-1}, 1692($CONH_2$); 33450 (NH_2).H^1 nmr δ s, .8.12 2H CO NH_2; m,7.00–7.73 5H $C_6H_{5;}$s, 5.32 2H NH_2; S, 1.76 1H SH.

2-Cayno 2-(4-Ethoxy-[1,3] Dithietan 2- Ylidene) Acetamide 16

To 2 (0.01 mol, 1.6 g) in ethanol (50 ml) was added triethylorthoformate (0.01, 1.5 ml) in ethanol (25 ml) drop wise (5 min), while stirring at room temperature for 30 min .The reaction mixture then, heated under reflux for 2 h. On cooling, the separated pale brown solid products was filtered and recrystallized from DMF. Yield 84.33%, m.p. 196°C. (shrinking). Analysis for $C_7H_8N_2O_2S_2$ (216.28), Required %, C, 38.87; H, 3.73; N, 12.95; S, 29.65. Found %, C, 39.00; H, 3.77; N, 13.00; S, 31.00. I.R/ cm^{-1} 2225, CN; 1700, amidic CONH2. H^1nmr δ t,1. 64 3H CH_3 (ester), q, 4.23 2H CH_2(ester), s, 5.67 1H cyclic CH-, s, 8.12 1H $CONH_2$.

7-Amino 2-(4-Chlorophenyl) 6- Cayno 5- Oxo 5H [1,3,4]thiadiazole [3,2-a] Pyridine 8-Carboxylic Acid Amide 20

On heating a mixture of 18 (1.93 g; 0.01 mol) and 2 (1.94 g; 0.01 mol) in dioxane (100 ml) and Et_3N (ml) under reflux for 3 ½ h followed by cooling to room temperature; a dark brown precipitation was gathered m.p. 238°C, yield 67.78%. Analysis for $C_{14}H_8ClN_5O_2S$ (345.76). Required % C, 48.63; H, 2.33; N, 20.25; S, 9.27.Found % C, 48.65; H, 2.35; N, 21.00; S, 9.44. IR/cm^{-1} 1755, C = O; 1675, $CONH_2$; 3325, NH_2; 2225, CN. H^1 nmr δ s, 8.22, 2H NH_2; m, 7.1–7.69,4H C_6H_4, s, 6.82 2H $CONH_2$.

5-Cyano 3-Thioxo 3H (1,2) Dithiole 4-Carboxylic Acid Amide (21)

A mixture of caynoacetamide (20) (0.01 mol, 0. 84 g), carbon disulfide (0.013 mol, 1 ml) and elemental sulfur (0.2 mol, 6.4 g) in DMF (50 ml) and Et_3N(1 ml) was stirred at room temperature for 30 min. Heating the mixture under reflux for 3 h followed by distillation of the excess DMF, a yellow residue was purified from ethanol, m.p. 205°C. Analysis for $C_5H_2N_2OS_3$ (202.28), yield 73.52%. Required % C,29.69; H, 1.00; N, 13.85; S, 47.56; Found % C, 29.72; H, 0.96; N, 13.92; S, 7.66., IR/cm^{-1} 2225; CN, 1725; CONH2.

5-Mercapto 3-Oxo 1-Phenyl 2,3-Dihydro 1H-Pyrazole 4-Carbonitrile 23

To an alcoholic aqueous solution (NaOH, 10 g/50 ml C_2H_5OH/50 ml H_2O) was added caynoacetic acid N-phenyl hydrazide (22) (0.01 mol; 1.75 g) in portions, followed by adding carbon disulfide (0.015 mol; 1.2 ml) while stirring at room temperature. After complete addition, the reaction mixture was stirred for further 3 h. On neutralization with concentration sulfuric acid a yellow residue formed is purified from ethanol to give 23 m.p. 184°C. Analysis for $C_{10}H_7N_3OS$ (217.24), yield 92.42%. Required % C, 55.29; H, 3.25; N, 19.34; S, 14.76. Found % C, 55.31; H, 3.25; N, 19.33; S, 14.82. IR/cm^{-1} 2225 CN; 3350 NH. H^1nmr δ s, 8.00, 1H NH; m, 6.89–7.44, C_6H_5;s,1.55 1H SH.

4-Amino 6-Ethoxy 1-Phenyl 1,2-Dihydro-Pyrazolo[3,4-d]Pyrimidine 3-One 24

A mixture of 23 (0.01 mol; 2.172 g) and thiourea (0.01 mol; 0.76 g) in absolute ethanol(50 ml) contained sodium ethoxide (0.012 mol, 0.81 g. $NaOC_2H_5$) was heated under reflux for 6 h. On neutralization with cold conc. hydrochloric acid, a pale yellow product was obtained and purified from ethanol. m.p. 259°C. Analysis for C13H13N5O2 (271.10) yield 59.78%. Requires % C, 57.56; H, 4.83; N, 25.82. Found % C, 57.58; H, 4.84; N, 25.83. IR/cm^{-1} 3450 NH2; 3330 NH; 1690 CONH.H^1nmr δ; s, 8.12, 1H, NH; m, 6.98–7.44, 5H, C_6H_5; s, 4.42, 2H, NH_2; q, 4.12, 2H, CH_2t, 1.33, 3H, CH_3.

2-[5-(Carbamoyl-cayno-methylene) [1,2,4]trithiolan 3-ylidene]2-cayno Acetamide 25

To an slightly alkaline solution (20 ml) (20% NaOH) was added the ammonium salt 2 (2.0 g) with gentle heating for 15 min (until the

evolution of H_2S gases ceased). Purification of the formed dark brown solid product from benzene gave 25. M.p. 289°C. (shrinking) yield 77.45%. Analysis for $C_8H_4N_4O_2S_3$ (284.338). Requires % C, 33.79; H, '1.42; N, 19.70; S, 33.83. Found % C, 33.77; H, 1.44, N, 19.68; S, 44.00. IR/cm^{-1} 2222&2225, 2CN.

2-[4-(carbamoyl-Cayno Methylene) 1,3 dithietan 2-ylidene2-caynoacetamide 26

On boiling (**25**) in ethanol for 4 h gave a red crystals. Purification of the product from benzene obtained a brown crystals m.p. 212°C yield 65.32%. Analysis for $C_8H_4N_4O_2S_2$ (252.273) Required % C, 30.09; H, 1.60; N, 22.21; S, 25.42. Found % C, 30.00; H, 1.55; N, 22.23; S, 25.55.IR/cm^{-1} 2222&2225, 2CN.

7-Mercapto 5-Thiophene 2-yl Thiopyrano[2,3d] Pyrimidine 2,4-Dione 30

A mixture of equimolar ratio of (**27**), (**30**) and sdium ethoxide (0.01 mol) in ethanol (75 ml) were heated under reflux for 2.5 h. On cooling a white yellowish solid product was separated. Purification from ethanol gave a product of m.p. 179°C. Analysis for $C_{11}H_7ClN_2O_2S_3$ (330.83) yield 69.91%. Required % C, 39.93; H, 2.13; N, 8.47; S, 29.08. Found% C, 40.00; H, 2.15; S, 29.11. H^1nmr δ; t, 7.37–7.39 3H (thiophene); d, 7.3–7.99, 1H, NH s, 5.74.1H C_5H_1.

5-Thiophene 2-yl 2,3,7,8,9,10a Hexahydro 1H 10,11 Dithia Benzo[b]fluorene 33

To (27) (0.01 mol; 2.02 gm)in CH_2Cl_2 (50 ml) in presence of Et_3N (1 ml) was added cyclohexane dione (0.01 mol; 1.12 g) while stirring at room temperature. Strring the reaction mnixture with gentle heating under reflux for 3 h. Leaving over night left a yellow solid product, m.p. 235°C yield 81.44%. Analysis for $C_{19}H_{16}O_2S_3$ (372.52). Required %C, 61.26; H, 4.33;S,25.82. Found % C, 61.29;H, 4.37; S, 26.00. H^1nmr δ,t,7.35–7.38 3H,(thiophene), t, 5.56–5.65, 1H C4; m, 3.35–3.82, 12H, $6CH_2$ (cyclohexane).

1-(4-Chlorobenzylidene) Amino 6-Mercapto 4-Thiophene 2-yl 1-H Pyrimidine 2-Thione 36

A mixture of equimolar ratio from (27) and (34) (0.01 mol) in ethanol (75 ml) and sodium ethoxide (0.022 mol) were heated under reflux for

3 h. Filtration of the separated solid producer and purification from ethanol gave (36) m.p. 218°C yield 79.24%. Analysis for $C_{15}H_{10}ClN_3S_3$ (363.91). Required % C,49.51; H,2.77; N,11.55; S,26.43. Found% C,49.55;H,2.75;S,26.67. H^1nmr δ, m,8.31–8.36., s; 8.01 1H CH = N, m,3H 7.38–7.49, (thiophene); s; 6.44 1H (C_5 pyrimidine) s, 1.32 1H, SH.

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