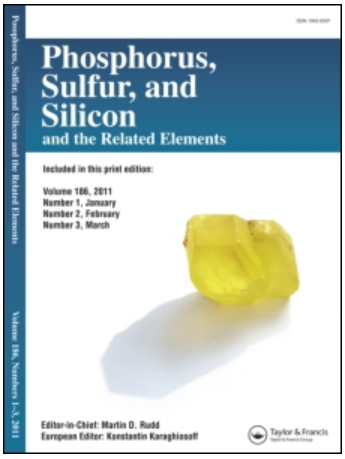

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REACTIONS OF HYDRAZONOYL HALIDES 33[1]: SYNTHESIS OF SOME NEW 2,3-DIHYDRO-1,3,4-THIADIAZOLES CONTAINING PYRAZOL-3-YL, INDOLIN-2-ONE-2-YL AND INDAN-1,3-DIONE-2-YL MOIETIES

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Some new 2,3-dihydro-1,3,4-thiadiazoles containing pyrazol-3-yl, indolin-2-one-2-yl and indan-1,3-dione-2-yl moieties in a good yields obtained from the reaction of hydrazonoyl halides with thiocarbamate and carbodithioate in ethanolic triethylamine respectively. In contrast, pyrazolylthiourea reacts with hydrazonoyl halides under the same condition afford corresponding hydrazonoyl sulfide derivatives.

Keywords: 2,3-Dihydro-1,3,4-thiadiazole; carbodithioate; hydrazonoyl halides; nitrile imine

INTRODUCTION

Many pyrazoles have been reported be use as antipyretic and analgesic drugs,[2–4] bactericides, and fungicides.[5] Isatin derivatives have been used in the development of colour photographic recording materials[6–8] of blood coagulation,[9–12] of liquid crystal components for display devices,[13–15] and in the inhibition of corrosion of aluminum,[16] Fe-Ni alloys[17] and of iron.[18] On the other hand; thiadiazoles are known to be highly biologically active reagents.[19–21] Based on these findings, it was of interest to synthesis some 2,3-dihydro-1,3,4-thiadiazoles directly with pyrazol-3-yl or indolin-2-one-2-yl or indan-1,3-dione-2-yl moieties are expected to posses potential biological activities.

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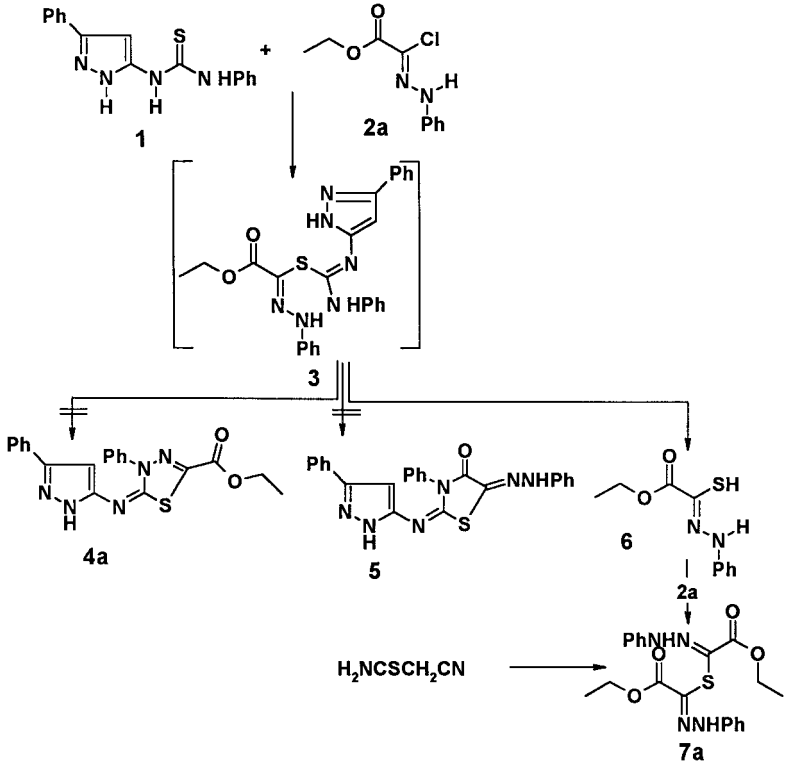
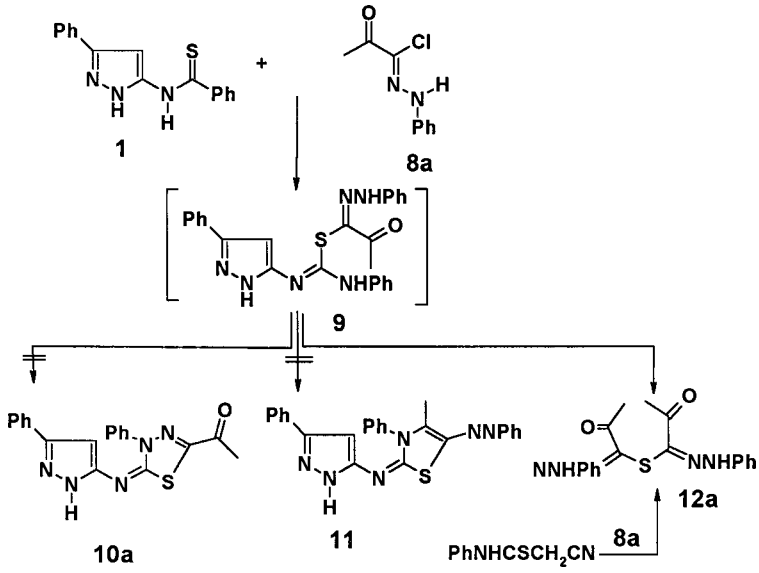
RESULTS AND DISCUSSION

N-phenyl-N-(5-phenylpyrazol-3-yl)thiourea (**1**) reacts with C-ethoxycarbonyl-N-phenylhydrazonoyl chloride (**2a**) to give isolable product. Its structure was confirmed on the basis of analytical and spectroscopic data. Thus, IR (cm^{-1}) spectrum showed bands at 3330 (NH) and 1701 (CO) and its ^1H NMR (δ ppm) spectrum showed signals at δ = 1.22 (t, 6H), 4.20 (q, 4H), 7.01–7.47 (m, 10H) and 10.83 (s, br., 2H). Based on these data the product formulated as ethyl 2-[2-aza-1-(ethoxycarbonyl)-2-(phenylamino)vinylthio]-3-aza-3-(phenyl-amino)prop-2-enoate (**7a**). Structure **7** was further confirmed by independent synthesis. Thus, cyanothioacetamide treated with **2a** to produce a product[22] proved to be identical in all respects (m.p., mixed m.p., and spectra) with **7a**. The formation of **7** can be explained via elimination hydrogen chloride to give the intermediate **3**, which readily to give **6**. Compound **6** reacts with **2a** to give the final product **7a**. On the above date the structures **4** and **5** were ruled out (cf. Scheme 1).



SCHEME 1

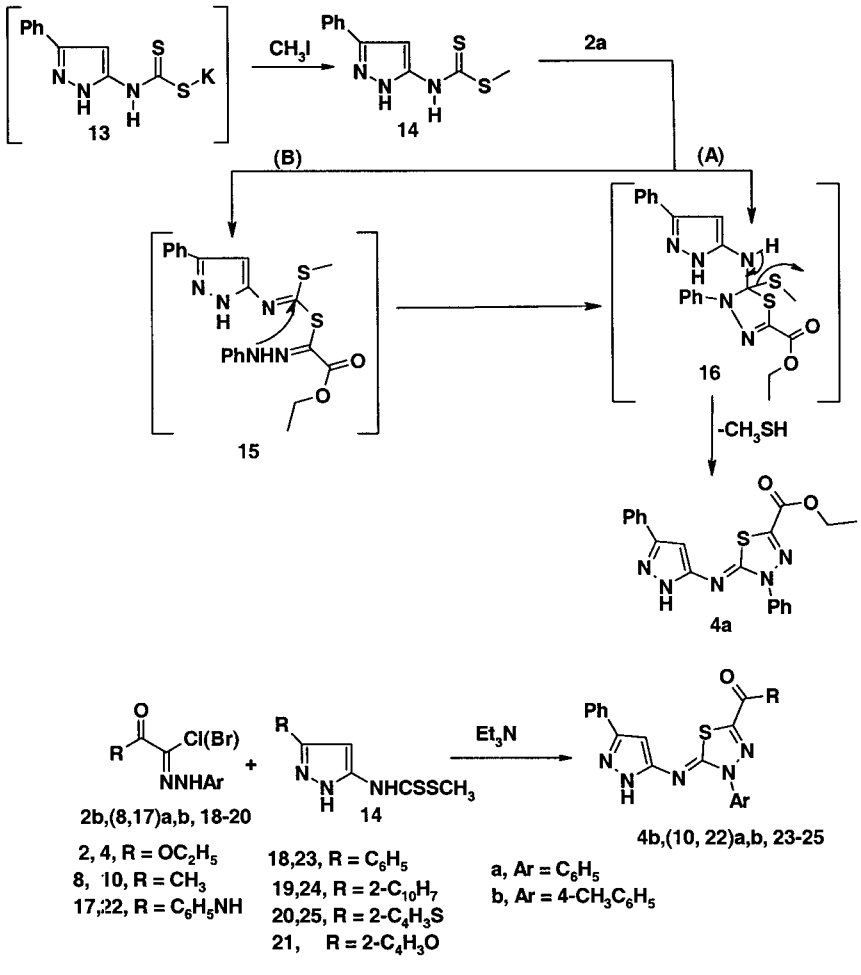
Also, compound **1** react with C-acetyl-N-phenylhydrazonoyl chloride (**8a**) in N,N-dimethylformamide containing potassium hydroxide to give product, identified as (3E)-4-aza-3-{1-[aza(phenylamino)methylene]-2-oxopropylthio}-4-(phenylamino)but-3-en-2-one (**12a**). Structure **12a** was confirmed on the basis of analytical and spectroscopic data. Thus, IR (cm^{-1}) spectrum of **12a** revealed bands at 3425 (NH) and 1658 (CO). Its ^1H NMR (δ ppm) spectrum showed signals at $\delta = 2.53$ (s, 6H), 7.06–7.43 (m, 10H) and 11.55 (s, br., 2H). Unequivocal support for the structure **12a**, it showed no depression in mixed m.p. with authentic sample[23] (which prepared from reaction of N-phenylcyanothioacetamide with **8a**) (cf. Scheme 2).



SCHEME 2

On the other hand, treatment of potassium [(3-phenylpyrazol-5-yl)amino]methanedithioate (**13**)[24] with methyl iodide afforded 3-[(methylthio)thiocarbonoylamino]-5-phenylpyrazole (**14**). Its structure was confirmed on the basis of elemental analyses, spectral data, and chemical transformation (cf. Experimental part). Compound **14** react with C-ethoxycarbonyl-N-Phenylhydrazonoyl chloride (**2a**) in ethanol containing triethylamine gave 2,3-dihydro-1,3,4-thiadiazole **4a**. The structure was deduced from their spectral and elemental analysis. IR (cm^{-1}) spectrum revealed bands at 3330 (NH) and 1710 (CO). Its ^1H NMR (δ ppm) spectrum showed signals at $\delta = 1.44$ (t, 3H, CH_2CH_3), 4.33 (q, 2H, CH_2CH_3), 6.4 (s, IH, pyrazole C-4), 7.21–7.47 (m, 10H,

ArH's), and 8.42 (s, IH, NH). The formation of thiadiazole **4a** can be explained via elimination of methanethiol from cycloadduct **16**, which is assumed to be formed from 1,3-dipolar cycloaddition of nitrile imide (generate in situ by treatment of **2a** with triethylamine) to C=S double bond of **14** (pathway A) or by its stepwise path involving substitution via 1,3-addition of thiol **14** to nitrile imide to give a cyclic hydrazone **15** (pathway B), which transformed to cyclic intermediate **16**. Cyclization of the latter is achieved by elimination of methanethiol to afford the final product **4a**. All attempts to isolate either intermediate hydrazone **15** or cycloadduct **16** were unsuccessful (cf. Scheme 3)



SCHEME 3

Similarly, the 3-[(methylthio)thiocarbonaylamino]-5-phenylpyrazole (**14**) reacted with the appropriate hydrazonoyl halides **2b**, (**8**, **17**)**a**, **b**, **18–20**, in ethanolic triethylamine to give the corresponding 2,3-dihydro-1,3,4-thiadiazole **4b**, (**10**, **22**)**a**, **b**, and **23–25**, respectively (cf. Scheme 3).

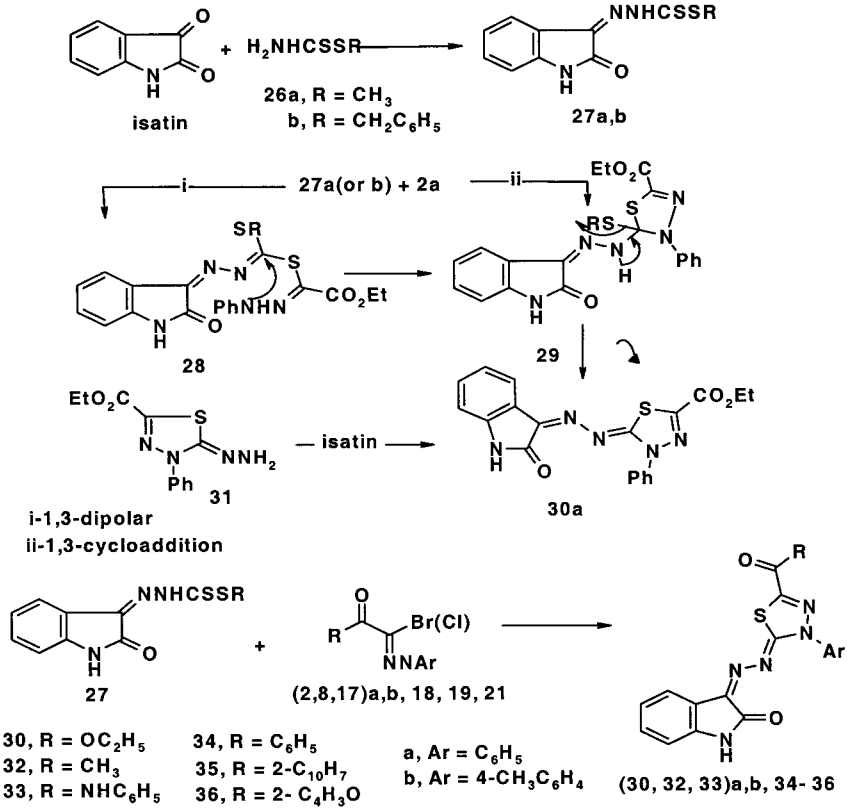
The potential reactivity of compounds **27**(**a**[25], **b**) [which prepared from reaction of isatin with alkyl hydrazinecarbodithioate (**26a**, **b**)] toward hydrazonoyl halides to afford 2,3-dihydro-1,3,4-thiadiazoles derivatives. Thus, **27a** reacted with C-ethoxyl-N-phenylhydrazonoyl chloride (**2a**) in ethanol containing triethylamine to afford 2,3-dihydro-1,3,4-thiadiazole derivatives **30a**.

Structure of **30a** was confirmed on the basis of analytical, spectroscopic data and alternative synthesis methods. Thus IR (cm^{-1}) spectrum revealed bands at 3147 (NH) and 1720, 1705 (CO's). ^1H NMR (δ ppm) spectrum showed signals at $\delta = 1.44$ (t, 3H), 4.34(q, 2H), 7.21–7.74 (m, 9H), and 10.71 (s, br., 1H). On the other hand, the reaction of **27b** with **2a** in ethanol containing triethylamine afforded product identical in all respects (m.p., mixed m.p., and spectra) with **30a**.

Unequivocal support on the structure of **30a** obtained by reaction of 2-hydrazino-2,3-dihydro-1,3,4-thiadiazole[26] (**31**) with isatin (**25**) gave identical product in all respect (m.p., mixed m.p., and spectra) with **30a** (cf. Scheme 4). The formation of unsymmetrical azine **30** can be explained via elimination alkyl thiol (RSH) from cycloadduct **29**, which assumed to be formed from 1,3-dipolar cycloaddition or 1,3-addition of nitrile imine to CS or thiol of **27**.

Similarly, the appropriate hydrazonoyl halides **2b**, (**8**, **17**)**a**, **b**, **18**, **19**, **21** react with the appropriate **27a**, **b** in ethanol containing triethylamine to give the corresponding unsymmetrical azine **30b**, (**32**, **33**)**a**, **b**, and **34–36** respectively. All compounds synthesized are elucidated its structure on the basis elemental analysis and spectroscopic data (cf. Experimental part).

Finally, indon-1,2,3-trione (**37**) reacts with the appropriate alkyl hydrazinecarbodthioate (**26a**, **b**) to give the corresponding 2-{aza-[alkylthiothioxomethyl)-amino]methyelne}indi-one-1,3-dione (**38a**, **b**), respectively. The structure of **38** was confirmed on the basis of analytical, spectroscopic data, and chemical transformation. Thus, IR (cm^{-1}) spectra of **38** revealed bands at 3330 (NH), 1728, and 1698 (COs). ^1H NMR (δ ppm) spectrum of **38a** showed signals at 2.95 (s, 3H) and 7.22–7.46 (m, 4H). Thus, the appropriate of **40a**, **b** reactions with C-ethoxycarbonoyl-N-phenylhydrazonoyl chloride (**2a**) in ethanol containing triethylamine to afford products has molecular formula $C_{20}H_{14}N_4O_4S$. IR (cm^{-1}) spectrum revealed bands near 1740–1715 (CO's). ^1H NMR spectrum showed signals (δ ppm) at 1.36 (t, 3H, CH_2CH_3), 4.42 (q, 2H, CH_2CH_3) and 7.35–8.05 (m, 9H, ArH's). Based on



SCHEME 4

the above data the product was formulated as: ethyl 2-[1,2-diaza-z-(1,3-dioxindan-2-ylidene-3-phenyl-1,3,4-thiadiazdine-5-carboxylate (**41a**).

Unequivocal support on the structure obtained from the study of the products reaction of 2-hydrazino-2,3-dihydro-1,3,4-thiadiazole **31** with indain-1,2,3-trione in ethanol, gave identical properties (m.p., mixed m.p., and spectra) with **41a** (cf. Scheme 5). The formation of **41** can be explained via elimination of alkyl mercaptan from cycloaddition **40**, which assumed to be formed from 1,3-dipalor cycloaddition or 1,3-addition of nitrile imine (which prepared in situ from hydrazonoyl chloride **2a** with triethylamine), to carbodithioate **38** (cf. Scheme 5).

Simiar, the appropriate hydrazonoyl halides **2b**, (**8**, **17**)**a**, **b**, **18–20** react with the appropriate **38a**, **b** in ethanol containing triethylamine to give the corresponding 2,3-dihydro-1,3,4-thiadiazole derivatives **41b**,(**42**, **43**)**a**, **b**, and **44–46**, respectively (cf. Scheme 5).

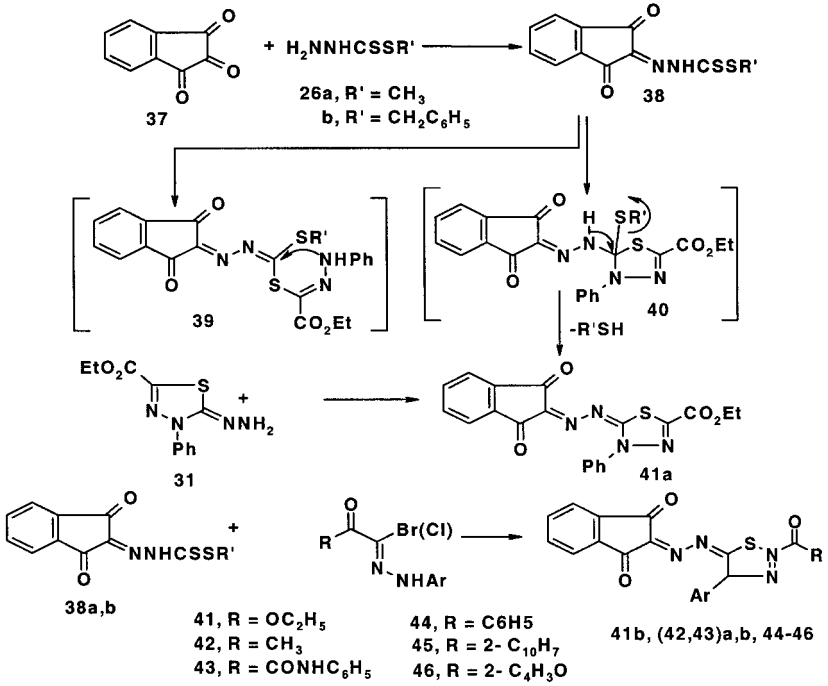
TABLE I Characterization Data of the Newly Synthesized Compounds

Compd. no.	R	Yield (%)	m.p. (°C) solvent	Mol. formula mol. wt.	% Analyses calcd./found			
					C	H	N	S
4a	OC_2H_5	85	98–100	$C_{20}H_{17}N_5O_2S$	61.36	4.37	17.89	8.19
			Pet. ether	391.45	61.30	4.30	17.80	8.10
4b	OC_2H_5	80	95	$C_{21}H_{19}N_5O_2S$	62.20	4.72	17.27	7.90
			Pet. ether	405.47	62.20	4.70	17.20	7.90
10a	CH_3	80	115	$C_{19}H_{15}N_5OS$	63.14	4.18	19.37	8.87
			EtOH	361.42	63.10	4.10	19.30	8.80
10b	CH_3	75	139	$C_{20}H_{17}N_5OS$	63.98	4.56	18.65	8.54
			EtOH	375.45	53.90	4.50	18.60	8.50
14	—	—	168–70	$C_{11}H_{11}N_2S_2$	52.98	4.49	16.85	25.72
			Benzene	249.34	52.50	4.40	16.80	25.70
22a	NHC_6H_5	65	113–115	$C_{24}H_{18}N_6OS$	65.73	4.13	19.16	7.31
			EtOH	438.51	65.70	4.10	19.10	7.30
22b	NHC_6H_5	60	75–77	$C_{25}H_{20}N_6OS$	66.35	4.45	18.57	7.08
			Pet. ether	452.54	66.30	4.40	18.50	7.00
23	C_6H_5	80	58–60	$C_{24}H_{17}N_5OS$	68.07	4.05	16.53	7.57
			Pet. ether	423.49	68.00	4.00	16.50	7.50
24	2-$C_{10}H_7$	70	80–82	$C_{28}H_{19}N_5OS$	71.01	4.04	14.78	6.77
			Pet. ether	473.55	71.00	4.00	14.70	6.74
25	2-C_4H_3S	65	50	$C_{22}H_{15}N_5OS_2$	61.52	3.52	16.30	14.92
			Pet. ether	429.52	61.50	3.50	16.30	14.50
27b	$CH_2C_6H_5$	89	221–23	$C_{16}H_{13}N_3OS_2$	58.69	4.00	12.83	19.58
			EtOH	327.43	58.60	4.00	12.80	19.50
30a	OC_2H_5	85	256	$C_{19}H_{15}N_5O_3S$	58.00	3.84	17.80	8.15
			AcOH	393.42	58.00	3.80	17.80	8.10
30b	OC_2H_5	80	295	$C_{20}H_{17}N_5O_3S$	58.95	4.20	17.19	7.87
			AcOH	407.45	58.90	4.20	17.20	7.80
32a	CH_3	85	298–300	$C_{18}H_{13}N_5O_2S$	59.49	3.60	19.27	8.82
			AcOH	363.39	59.40	3.60	19.29	8.80
32b	CH_3	75	270–72	$C_{19}H_{15}N_5O_2S$	60.46	4.00	18.55	8.49
			AcOH	377.42	60.40	4.00	18.50	8.40
33a	NHC_6H_5	70	330–33	$C_{23}H_{16}N_6O_2S$	62.71	3.66	19.08	7.28
			AcOH	440.48	62.70	3.60	19.00	7.20
33b	NHC_6H_5	65	350–52	$C_{24}H_{18}N_6O_2S$	63.42	3.99	18.49	7.05
			AcOH	454.51	63.40	3.90	18.40	7.00
34	C_6H_5	70	295	$C_{23}H_{15}N_5O_2S$	64.92	3.55	16.46	7.53
			AcOH	425.47	65.00	3.30	16.50	7.20
35	2-$C_{10}H_7$	80	317	$C_{27}H_{17}N_5O_2S$	68.05	3.60	14.72	6.74
			AcOH	475.53	68.00	3.80	14.60	6.70
36	2-C_4H_3O	75	270	$C_{21}H_{13}N_5O_3S$	60.71	3.15	16.85	7.71
			AcOH	415.44	60.50	3.30	16.80	7.60
38a	CH_3	88	108–110	$C_{11}H_8N_2O_2S_2$	49.98	3.05	10.59	24.25
			EtOH	264.32	49.90	3.00	10.50	24.20
38b	$CH_2C_6H_5$	92	130–132	$C_{17}H_{12}N_2O_2S_2$	59.98	3.55	8.22	18.83
			EtOH	340.41	59.90	3.50	8.20	18.80
41a	OC_2H_5	80	190–92	$C_{20}H_{14}N_4O_4S$	59.11	3.47	13.78	7.89
			AcOH	406.42	59.10	3.40	13.70	7.80

(Continued on next page)

TABLE I Characterization Data of the Newly Synthesized Compounds *(Continued)*

Compd. no.	R	Yield (%)	m.p. (°C) solvent	Mol. formula mol. wt.	% Analyses calcd./found C	H	N	S
41b	OC_2H_5	80	180–82	$C_{21}H_{16}N_4O_4S$	59.99	3.83	13.32	7.62
			AcOH	420.45	59.90	3.80	13.30	7.60
42a	CH_3	85	140	$C_{19}H_{12}N_4O_3S$	60.63	3.21	14.88	5.81
			AcOH	376.39	60.60	3.20	14.80	8.50
42b	CH_3	75	170	$C_{20}H_{14}N_4O_3S$	61.53	3.61	14.35	8.21
			AcOH	390.42	61.50	3.60	24.30	8.20
43a	C_6H_5NH	72	185	$C_{24}H_{15}N_5O_3S$	63.57	3.33	15.44	7.07
			AcOH	453.48	63.50	3.30	15.40	7.00
43b	C_6H_5NH	75	190	$C_{25}H_{17}N_5O_3S$	64.23	3.66	14.98	6.86
			AcOH	467.50	64.00	3.60	14.90	6.80
44	C_6H_5	70	145	$C_{24}H_{14}N_4O_3S$	65.74	3.22	12.78	7.31
			AcOH	438.46	65.70	3.20	12.70	7.30
45	2-$C_{10}H_7$	80	125–27	$C_{28}H_{16}N_4O_3S$	68.84	3.30	11.47	6.56
			AcOH	488.50	68.80	3.30	11.40	6.50
46	2-C_4H_3O	75	195	$C_{22}H_{12}N_4O_4S$	61.67	2.82	13.08	7.48
			AcOH	425.43	61.60	2.80	13.00	7.40



SCHEME 5

TABLE II Spectroscopic Data of the Newly Synthesized Compounds

Compd. no.	IR (cm^{-1})	^1H NMR (δ ppm)
4a	3330 (NH) and 1707 (CO)	1.44 (t, 3H, CH_2CH_3), 4.44 (q, 2H, CH_2CH_3), 6.4 (s, IH, pyrazole (C-4′), 7.21–7.92 (m, 11 H, ArHs and NH)
4b	3261 (NH) and 1708 (CO).	1.44 (t, 3H, CH_2CH_3), 2.3 (s, 3H, 4-$CH_3C_6H_4$), 4.44 (q, 2H, CH_2CH_3), 6.4 (s, IH, pyrazole (C-4′), 7.21–7.92 (m, 10 H, ArHs and NH)
10	3330 (NH) and 1701.6 (CO)	1.22 (t, 6H), 4.20 (q, 4H), 7.01–7.47 (m, 10H) and 10.83 (s, br., 2H).
14	3247 (NH) and 1681 (CO)	2.38 (s, 3H), 6.4 (s, 1H), 7.07–7.48 (m, 10H) and 10.68 (s, 1H).
22a	3244 (NH) and 1676	6.42 (s, 1H), 7.22–7.52 (m, 15H), 9.22 (s, 1H) and 10.67 (s, 1H).
22b	3240 (NH) and 1672	2.33 (s, 3H), 6.42 (s, 1H), 7.22–7.52 (m, 9H), 9.22 (s, 1H) and 10.67 (s, 1H).
24	3240 (NH) and 1673 (CO)	6.42 (s, 1H), 7.17–7.66 (m, 18H).
27a	3340 (NH) and 1703 (CO)	2.92 (s, 3H), 6.82–7.53 (m, 4H), 10.71 (s, 1H) and 11.0 (s, 1H).
27b	3340 (NH) and 1703 (CO)	2.66 (s, 2H), 6.82–7.53 (m, 9H), 10.71 (s, 1H) and 11.0 (s, 1H).
30a	3147 (NH) and 1720, 1705 (COs)	1.38 (t, 3H, CH_2CH_3), 4.34 (q, 2H, CH_2CH_3), 7.21–7.74 (m, 9H) and 10.70 (s, br., 1H)
30b	3140 (NH) and 1747 (COs)	1.34 (t, 3H, CH_2CH_3), 2.48 (s, 3H, 4-$CH_3C_6H_4$), 4.42 (q, 2H, CH_2CH_3), and 6.83–7.87 (m, 9H, ArHs and NH proton).
32a	3134 (NH), 1712, 1691 (CO)	2.33 (s, 3H), 7.21–7.74 (m, 9H) and 10.70 (s, br., 1H)
32b	3161 (NH), 1714, 1693 (CO)	2.33 (s, 3H), 244 (s, 3H), 7.21–7.74 (m, 8H) and 10.70 (s, br., 1H).
38a	3330 (NH), 1728 and 1698 (COs)	2.95 (s, 3H), 7.22–7.46 (m, 4H) and 10.55 (s, 1H)
38b	3330 (NH), 1728 and 1698 (COs)	2.65 (s, 2H), 7.22–7.46 (m, 9H) and 10.55 (s, 1H)
41a	1728,1698 (COs)	1.36 (t, 3H, CH_2CH_3), 4.42 (q, 2H, CH_2CH_3) and 7.35–8.05 (m, 9H, ArHs)
41b	1740, 1720 (COs)	1.36 (t, 3H, CH_2CH_3), 2.45 (s, 3H), 4.42 (q, 2H, CH_2CH_3) and 7.35–8.05 (m, 8H, ArHs)
42a	1728, 1689 (COs)	2.32 (s, 3H), and 7.35–8.05 (m, 9H, ArHs)
42b	1728, 1689 (COs)	2.32 (s, 3H), 2.42 (s, 3H), and 7.35–8.05 (m, 8H, ArHs)
45	1728, 1690 (COs)	7.26–8.33 (m, ArHs)

EXPERIMENTAL

All melting points were determined on an electrothermal apparatus and are uncorrected. IR spectra were recorded (KBr discs) on a Shimadzu FT-IR 8201 PC spectrophotometer. ^{1}H NMR spectra were recorded in $CDCl_3$ and $(CD_3)_2SO$ solutions on a Varian Gemini 300 MHz spectrometer and chemical shifts were expressed in δ units using TMS as internal reference. Elemental analyses were carried out at the Microanalytical Center of the Cairo University.

Syntheses of 2,3-Dihydro-1,3,4-thadiazoles (4, 10, 22)a, b, and 23–25

Equimolar amounts of the appropriate 3-amino-5-phenylpyrazole, potassium hydroxide, carbon hydroxide (5 mmol, each) in N,N-dimethylformamide (15 ml) was stirred at room temperature for 3 h until the potassium hydroxide dissolved, and then methyl iodide (0.5 ml) was added. Each hydrazonoyl hydrazonoyl halides (**2**, **8**, **17**)**a**, **b**, **18**, **19**, **21** (5 mmol) was added to the solution with stirring and then triethyamine (0.75 ml) was added. The resulting solid was collected, washed, and then crystallized from acetic acid to afford thiadiazoles (**4**, **10**, **22**)**a**, **b**, and **23–25**, respectively (cf. Tables I and II).

Syntheses of 2,3-Dihydro-1,3,4-thadiazoles (30, 32, 33, 41–43)a, b, 34–36, and 44–46

Triethylamine (0.75 ml, 5 mmol) was added to a mixture of the appropriate (5 mmol) and the appropriate hydrazonoyl bromide (**2**, **8**, **17**)**a**, **b**, **18–20** (5 mmol) and the appropriate alkyl carbodithioates (**29**, **40**)**a**, **b** (5 mmol) in ethanol (20 ml) while stirring. The reaction was stirred for 30 min at room temperature. The formed precipitate was collected, washed with ethanol, and crystallized from acetic acid to give 1,3,4-thiadiazolines (**30**, **32**, **33**, **41–43**)**a**, **b**, **34–36**, and **44–46** respectively.

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