

Reaction with Hydrazonoyl Halides. Part 32 [1]: Reaction of *C*-acyl-*N*-(3-Phenyl-5-pyrazolyl)hydrazonoyl Chlorides with Potassium Thiocyanate and Synthesis of Some New 2,3-dihydro-1,3,4-Thiadiazoles and Slenadiazoles*

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ABSTRACT: *C*-acyl-*N*-(3-phenyl-5-pyrazolyl)hydrazonoyl chlorides **1a,b** react with potassium thiocyanate and potassium selenocyanate to give 5-acyl-2,3-dihydro-2-imino-3-(3'-phenyl)pyrazol-5'-yl)-1,3,4-thiadiazoles **2a,b** and 5-acetyl-2,3-dihydro-2-imino-3-(3'-phenyl)pyrazol-5'-yl)-1,3,4-selenadiazole **10a,b**. Also, 2-[mercapto-(methylthio)methylene]indan-1,3-dione **16** reacts with hydrazonoyl halides **15** and **22–25** to afford 2,3-dihydro-1,3,4-thiadiazoles **19** and **26–29**, respectively. Structures of the newly synthesized compounds are elucidated on the basis of spectral data, chemical transformations, and alternative synthesis methods. © 2001 John Wiley & Sons, Inc. Heteroatom Chem 12:468–474, 2001

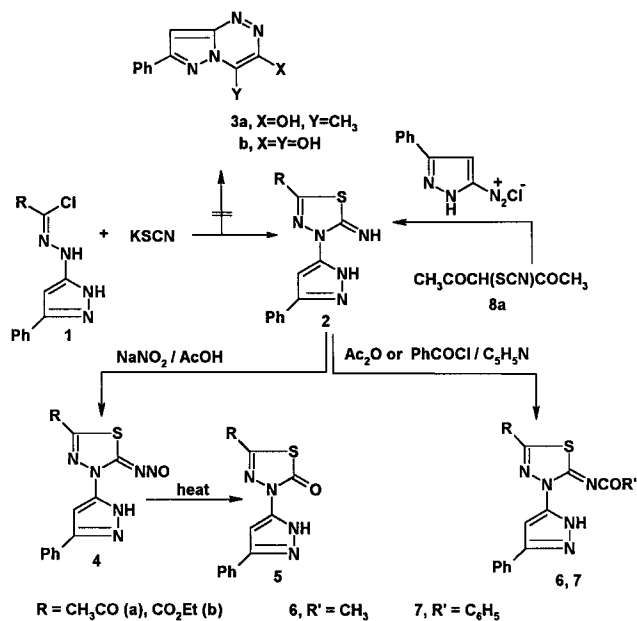
The reported biological activity of 1,3,4-thiadiazoles [1–5] makes them attractive targets for synthesis. The reactions between *C*-acyl-*N*-pyrazolylhydrazonoyl bromides and potassium thiocyanate have

been examined [6] and give exclusively pyrazolo[5,1-*c*] triazines **3**. The formation of 5-acyl-2,3-dihydro-2-imino-3-pyrazol-3'-yl-1,3,4-thiadiazole **2**, although expected in principle, was not observed. In the present study, we investigated this reaction, and we isolated 5-acyl-2,3-dihydro-2-imino-3-(3'-phenyl)pyrazol-5'-yl)-1,3,4-thiadiazoles as the sole product in each case and in about 80% yield. Structures were supported by both spectral data and chemical transformations. Thus, the IR (cm⁻¹) spectrum **2a** revealed bands at 3306 due to the imino (NH) group, 1653 (CO), 1618 (C = N), and no band at 2000–2200 showing the absence of a free SCN group [7]. Its ¹H NMR spectrum showed signals at δ = 2.51 (s, 3H, CH₃CO), 6.49 (s, 1H, pyrazole C-4), 7.20–7.59 (m, 6H, ArHs and NH), and 10.82 (s, br., 1H, NH). Upon shaking compound **2a** with D₂O, a new singlet at 4.38 assignable to DOH was observed. Unequivocal support of the structure of **2a** was further confirmed by treatment of 5-phenylpyrazole-3-diazonium chloride with 3-thiocyanato-2,4-pentandione (**8a**) [8] in ethanolic sodium acetate to produce a product that proved to be identical in all respects (m.p., mixed m.p., and spectra) with **2a** (cf. Scheme 1).

Nitrosation of the imino derivative **2a** gave a 2-*N*-nitroso derivative **4a**. The structure of **4a** was confirmed on the basis of elemental analyses, spectral

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SCHEME 1

data, and its thermal decomposition. Thus, the IR (cm^{-1}) spectrum of **4a** revealed bands at 3310 (NH), 1660 (CO), and 1530 (NO). The 1H NMR spectrum of **4a** showed signals at $\delta = 2.51$ (s, 3H, CH_3CO), 6.56 (s, 1H, pyrazole C-4), 7.20–7.59 (m, 5H, ArHs), and 10.82 (s, br, 1H, NH). The nitroso derivative **4a** decomposed to the corresponding 2,3-dihydrothiadiazolone **5a**, when heated in a refluxing xylene solution. The structure of **5a** was confirmed on the basis of analytical and spectral data studies. The IR (cm^{-1}) spectrum of **5a** revealed absorption bands at 3340 (NH), 1690, 1653 (COs) groups. The 1H NMR spectrum of **5a** showed signals at $\delta = 2.52$ (s, 3H, CH_3CO), 6.56 (s, 1H, pyrazole C-4), 7.20–7.58 (m, 5H, ArHs), and 10.82 (s, 1H, NH). Acylation of **2a** with acetic anhydride (and benzoyl chloride in pyridine) afforded the corresponding acetyl and benzoyl derivatives **6a** and **7a**, respectively (cf. Scheme 1). Both elemental analyses and spectral data were consistent with the assigned structure of the products **6a** and **7a**. The 1H NMR spectrum of **6a** showed signals at $\delta = 2.52$ (s, 3H, CH_3CO), 2.72 (s, 3H, $CH_3CON=$), 6.61 (s, 1H, pyrazole C-4), 7.22–7.51 (m, 5H, ArHs) and 10.56 (s, br, 1H, NH). Its IR (cm^{-1}) spectrum revealed bands at 1664 and 1638 (COs). The 1H NMR spectrum of **7a** showed signals at $\delta = 2.51$ (s, 3H, CH_3), 6.60 (s, 1H, pyrazole C-4), 7.22–7.81 (m, 10H, ArHs), and 10.84 (s, br, 1H, NH). Its IR (cm^{-1}) spectrum revealed bands at 1661 and 1640 (CO groups).

Such results indicate that both the azo coupling of **8a** and the reaction of **1a** with potassium thiocyanate proceed through the hydrazone **9**.

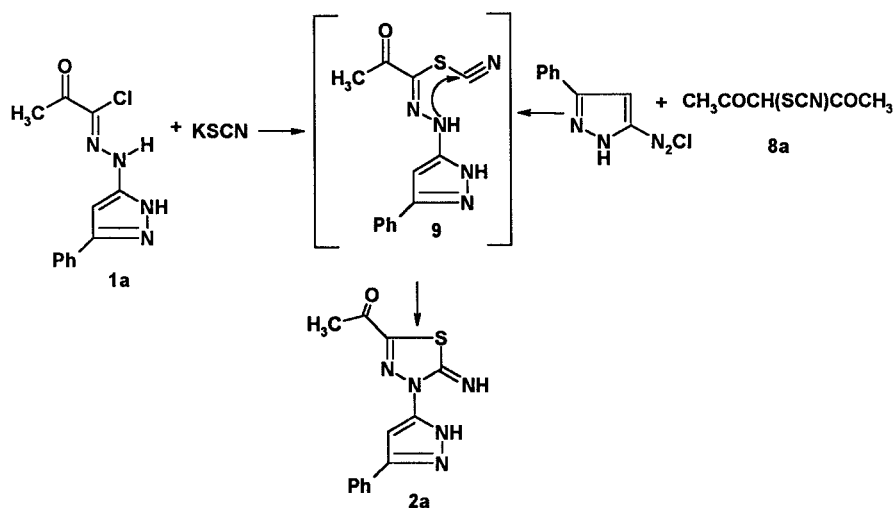
The latter is cyclized readily under the reaction conditions to give 5-acetyl-2-imino-2,3-dihydro-5'-phenylpyrazol-3'-yl-1,3,4-thiadiazole (**2a**).

Also, C-ethoxycarbonyl-*N*-(5-phenyl)pyrazol-3-ylhydrazonoyl chloride (**1b**) reacted with potassium thiocyanate in ethanol at room temperature to afford 5-ethoxycarbonyl-2,3-dihydro-2-imino-3-(5'-phenyl)pyrazol-3'-yl-2,3-dihydro-1,3,4-thiadiazole (**2b**). The structure was elucidated on the basis of elemental analysis, spectral data, and chemical reactions (acylation, nitrosation, thermal decomposition of the nitroso product) (cf. Scheme 1 and Experimental section).

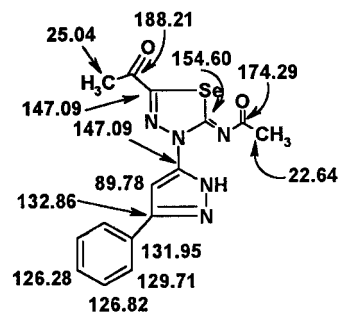
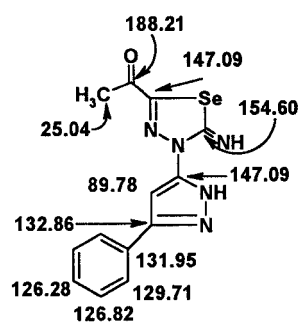
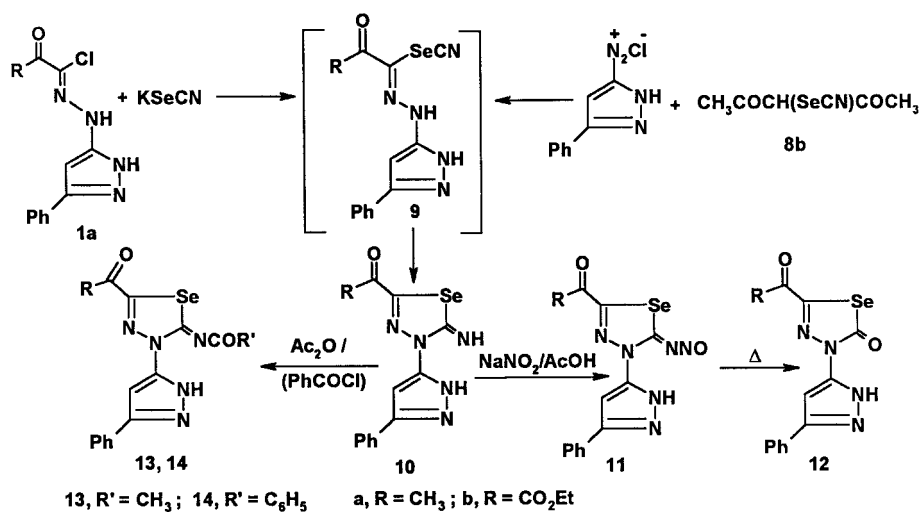
Moreover, hydrazonoyl chlorides **1a,b** reacted with potassium selenocyanate to afford 2,3-dihydro-1,3,4-selenadiazoles **10a,b**. Structure **10a** was elucidated on the basis of spectral data, chemical transformation, and an alternative synthesis (by coupling of diazotized aminopyrazole with 3-selenocyanato-2,4-pentane dione (**8b**) [8] (cf. Scheme 3 and Experimental section).

Next, the reaction of C-ethoxycarbonyl-*N*-phenylhydrazonoyl chloride **15a** with 2-[mercapto(methylthio)methylene]indan-1,3-dione **16** [9] in the presence of triethylamine gave 2,3-dihydro-1,3,4-thiadiazole **19a**. The structure was deduced from its spectra and elemental analysis. The IR (cm^{-1}) spectrum revealed bands at 1741, 1697, 1645 (COs), and 1593 (C=C). Its 1H NMR spectrum showed signals at $\delta = 1.44$ (t, 3H, CH_3CH_2), 4.53 (q, 2H, CH_2CH_3), and 7.25–7.57 (m, 9H, ArHs). The structure **19** was further confirmed by the reaction of hydrazonoyl chloride **15a** with thioanilide **20** (which was prepared from 1,3-indandione and phenyl isothiocyanate) in *N,N*-dimethylformamide solution containing potassium hydroxide to give a product identical in all respects (m.p., mixed m.p. and spectra) with **19a** (cf. Scheme 4). The formation of thiadiazole **19** can be explained on the basis of the elimination of methanethiol from the cycloadduct **18**, which is assumed to be formed by a 1,3-dipolar cycloaddition of the nitrile imide (generated in situ by treatment of **15a** with triethylamine) to the C=S double bond of **16** or by a stepwise path involving substitution via a 1,3-addition of thiol **16** to the nitrile imide to give a cyclic hydrazone **17**, which was transformed into the cyclic intermediate **18**. Cyclization of the latter could be achieved by elimination of methanethiol to afford the final product **19**.

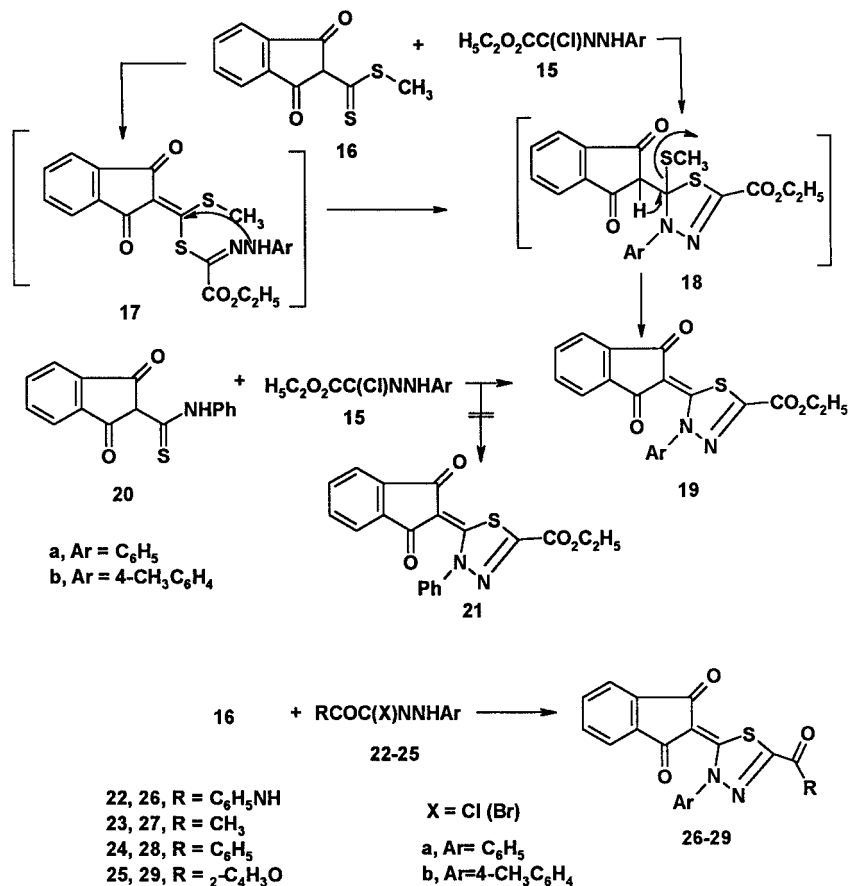
It is worthwhile to mention that the elimination of aniline by the reaction of thioanilide **20** with hydrazonoyl chloride **15a** came about from the thioanilide, based on comparison spectral data with the product obtained from the reaction of thioanilide **20**



SCHEME 2



SCHEME 3



SCHEME 4

with *C*-ethoxycarbonyl-*N-p*-tolylhydrazonoyl chloride (**15b**) (cf. Scheme 4). Thus, The ¹H NMR spectrum of **19b** showed signals at $\delta = 1.44$ (t, 3H, CH₃CH₂), 2.45 (s, 3H, 4-CH₃C₆H₄), 4.52 (q, 2H, CH₃CH₂), and 7.22–7.58 (m, 8H, ArHs). Similarly, the 2-[mercapto(methylthio)methylene]indan-1,3-dione (**16**) reacted with hydrazonoyl halides **22–25** in ethanolic triethylamine to give the corresponding 2,3-dihydro-1,3,4-thiadiazoles **26–29**, respectively (cf. Scheme 4).

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. ¹H NMR spectra were recorded in CDCl₃ and (CD₃)₂SO solutions on a Varian Gemini 300 MHz spectrometer, and chemical shifts are expressed in δ units using tetramethylsilane (TMS) as an internal reference. Elemental analyses were carried out at the Microanalytical Center of the Cairo University. Hydrazonoyl bromides **1a,b**

[6], **15** [10], and **22–25** [11–14] were prepared as previously reported.

Synthesis of 5-Acyl-2,3-dihydro-2-imino-3-(3'-phenyl)pyrazol-5'-yl)-1,3,4-thiadiazole and 5-Acetyl-2,3-dihydro-2-imino-3-(3'-phenyl)pyrazol-5'-yl)-1,3,4-selenadiazole (**2a,b**) and **10a,b**

Method A A mixture of the appropriate *C*-acyl-*N*-5-phenylpyrazol-3-ylhydrazonoyl chloride (**1a,b**) and potassium thiocyanate (or potassium selenocyanate) (5 mmol) in ethanol (25 mL) was stirred at room temperature for 2 hours. The resulting solid was collected and crystallized from ethanol to give the thiadiazole **2a,b** and the selenadiazole **10a,b**, respectively.

Method B Diazotized 3-amino-5-phenylpyrazole [15] (5 mmol) was added to ethanol (50 mL) containing sodium acetate (1.3 g) and 3-thiocyanato- (or 3-selenocyanato)-2,4-pentanedione (5 mmol) at 0–5°C with stirring. Stirring was continued for 3 hours, and then the resulting solid was collected by

TABLE 1 Characterization Data of the Newly Synthesized Compounds

Compound No.	<i>m.p.</i> (°C) Color	Yield (%)	Mol. Formula	Mol. Wt	% Analyses, Calcd./Found			
					%C	%H	%N	%S
2a	197–98	85	C ₁₃ H ₁₁ N ₅ OS	285.33	54.72	3.89	24.54	11.24
	Yellow				54.60	4.10	24.30	11.40
2b	180–82	80	C ₁₄ H ₁₃ N ₅ O ₂ S	315.36	53.32	4.16	22.21	10.17
	Yellow				53.20	4.20	22.40	10.00
4a	142–43 dec	91	C ₁₃ H ₁₀ N ₆ O ₂ S	314.33	49.68	3.21	26.74	10.20
	Red				49.80	3.40	26.60	10.30
4b	134–35 dec	89	C ₁₄ H ₁₂ N ₆ O ₃ S	344.35	48.83	3.51	24.41	9.31
	Red				48.90	3.40	24.60	9.50
5a	215–16	87	C ₁₃ H ₁₀ N ₄ O ₂ S	286.31	54.54	3.52	19.57	11.20
	Pale yellow				54.40	3.70	19.70	11.00
5b	165–67	89	C ₁₄ H ₁₂ N ₄ O ₃ S	316.34	53.16	3.82	17.71	10.14
	Pale yellow				53.00	3.90	17.60	10.30
6a	195–96	88	C ₁₅ H ₁₃ N ₅ O ₂ S	327.37	55.04	4.00	21.39	9.79
	Pale yellow				55.00	4.20	21.10	9.90
6b	213–14	90	C ₁₆ H ₁₅ N ₅ O ₃ S	357.39	53.77	4.23	19.60	8.97
	Pale yellow				53.50	3.30	19.70	8.80
7a	284–85	89	C ₂₀ H ₁₅ N ₅ O ₂ S	389.44	61.68	3.88	17.98	8.23
	Buff				61.80	3.90	18.10	8.40
7b	220–22	91	C ₂₁ H ₁₇ N ₅ O ₃ S	419.47	60.13	4.09	16.70	7.64
	Pale yellow				60.10	3.90	16.50	7.40
10a	179	85	C ₁₃ H ₁₁ N ₅ OSe	332.23	47.00	3.34	21.08	—
	Pale yellow				46.80	3.50	21.20	—
10b	145–147	78	C ₁₄ H ₁₃ N ₅ O ₂ Se	362.25	46.42	3.62	19.33	—
	Yellow				46.40	3.50	19.10	—
11a	132 dec.	92	C ₁₃ H ₁₀ N ₆ O ₂ Se	361.22	43.23	2.79	23.27	—
	Red				43.30	2.60	23.40	—
11b	134 dec.	63	C ₁₄ H ₁₂ N ₆ O ₃ Se	391.25	42.98	3.09	21.48	—
	Red				43.10	3.20	21.60	—
12	187–88	73	C ₁₃ H ₁₀ N ₄ O ₂ Se	333.21	46.86	3.03	16.81	—
	Pale yellow				46.60	3.10	17.00	—
13	197–98	82	C ₁₅ H ₁₃ N ₅ O ₂ Se	374.26	48.14	3.50	18.71	—
	Pale yellow				48.20	3.60	18.60	—
13b	174–6	79	C ₁₆ H ₁₅ N ₅ O ₃ Se	404.29	47.53	3.74	17.32	—
	Pale yellow				47.30	3.60	17.30	—
14	254–5	85	C ₂₀ H ₁₅ N ₅ O ₂ Se	436.33	55.05	3.74	16.05	—
	Pale yellow				54.90	3.60	16.20	—
19a	257–58	81	C ₂₀ H ₁₄ N ₂ O ₄ S	378.41	63.48	3.73	7.40	8.47
	Yellow				63.60	3.60	7.50	8.30
19b	245–48	82	C ₂₁ H ₁₆ N ₂ O ₄ S	392.44	64.27	4.11	7.14	8.17
	Yellow				64.20	4.00	6.90	8.30
26a	312–14	78	C ₂₄ H ₁₅ N ₃ O ₃ S	425.47	67.75	3.55	9.88	7.54
	Yellow				67.60	3.70	10.10	7.40
26b	322–25	87	C ₂₅ H ₁₇ N ₃ O ₃ S	439.50	68.32	3.90	9.56	7.29
	Yellow				68.20	3.80	9.60	7.10
27a	297–98	81	C ₁₉ H ₁₂ N ₂ O ₃ S	348.38	65.51	3.47	8.04	9.20
	Yellow				65.70	3.60	7.80	9.40
27b	326–27	84	C ₂₀ H ₁₄ N ₂ O ₃ S	362.41	66.28	3.89	7.73	8.85
	Yellow				66.10	4.00	7.60	8.70
28a	266–68	79	C ₂₄ H ₁₄ N ₂ O ₃ S	410.45	70.23	3.44	6.82	7.81
	Yellow				70.30	3.30	6.90	7.90
28b	315–18	86	C ₂₅ H ₁₆ N ₂ O ₃ S	424.48	70.74	3.80	6.60	7.55
	Yellow				70.90	3.90	6.50	7.40
29a	153–54	76	C ₂₂ H ₁₂ N ₂ O ₄ S	400.42	65.99	3.02	7.00	8.01
	Yellow				65.80	3.10	6.90	8.20
29b	>330	74	C ₂₃ H ₁₄ N ₂ O ₄ S	414.44	66.66	3.40	6.76	7.74
	Yellow				66.50	3.30	6.60	7.80

TABLE 2 IR and ^1H NMR Spectra of the Newly Synthesized Compounds

Compound No.	IR (cm^{-1})	^1H NMR (δ ppm)
2b	3306 (NH), 1715 (CO), and 1615 (C=N).	1.31 (t, 3H, CH_2CH_3), 4.43 (q, 2H, CH_2CH_3) 6.51 (s, 1H, pyrazole C-4), 7.21–7.56 (m, 6H, ArHs and NH) and 10.82 (s, br., 1H, NH).
4b	3320 (NH), 1715 (CO), and 1535 (NO).	1.34 (t, 3H, CH_2CH_3), 4.22 (q, 2H, CH_2CH_3), 6.52 (s, 1H, pyrazole C-4), 7.21–7.55 (m, 5H, ArHs) and 10.62 (s, br, 1H, NH).
5b	3327 (NH), 1715, and 1695 (COs).	1.41 (t, 3H, CH_2CH_3), 4.21 (q, 2H, CH_2CH_3), 6.43 (s, 1H, pyrazole C-4), 7.21–7.54 (m, 5H, ArHs) and 10.33 (s, 1H, NH).
6b	3330 (NH), 1713, and 1655 (COs)	1.30 (t, 3H, CH_2CH_3), 2.53 (s, 3H, $\text{CH}_3\text{CON}=\text{O}$), 4.42 (q, 2H, CH_2CH_3), 6.60 (s, 1H, pyrazole C-4), 7.21–7.56 (m, 5H, ArHs) and 10.82 (s, br, 1H, NH).
7b	3335 (NH), 1715, and 1660 (COs)	1.32 (t, 3H, CH_2CH_3), 4.21 (q, 2H, CH_2CH_3), 6.45 (s, 1H, pyrazole C-4), 7.21–7.85 (m, 10H, ArHs) and 10.62 (s, br, 1H, NH).
10a	3306 (NH), 1653 (CO), 1618 (C=N)	2.51 (s, 3H, CH_3CO), 6.44 (s, H, pyrazole C-4), 7.49–7.87 (m, 6H, ArHs and NH) and 10.82 (s, br, 1H, NH).
10b	3306 (NH), 1715 (CO), and 1615 (C=N).	1.31 (t, 3H, CH_2CH_3), 4.43 (q, 2H, CH_2CH_3) 6.51 (s, 1H, pyrazole C-4), 7.21–7.56 (m, 6H, ArHs and NH) and 10.82 (s, br, 1H, NH).
12a	3340 (NH), 1690, 1653 (COs)	2.52 (s, 3H, CH_3CO), 6.56 (s, 1H, pyrazole C-4), 7.20–7.58 (m, 5H, ArHs) and 10.32 (s, 1H, NH).
13a	3340 (NH), 1664, and 1638 (COs).	2.52 (s, 3H, CH_3CO), 2.72 (s, 3H, $\text{CH}_3\text{CON}=\text{O}$), 6.64 (s, 1H, pyrazole C-4), 7.42–7.87 (m, 5H, ArHs) and 10.89 (s, br, 1H, NH).
14a	3340 (NH), 1661, and 1640 (COs).	2.52 (s, 3H, CH_3), 6.65 (s, 1H pyrazole C-4), 7.42–7.81 (m, 10H, ArHs) and 10.91 (s, br, 1H, NH).
26a	3350 (NH), 1695, 1680, 1652 (COs) and 1600 (C=C).	7.21–7.82 (m, 14H, ArHs) and 8.34 (s, 1H, NH).
26b	3342 (NH), 1690, 1685, 1655 (COs) and 1595 (C=C)	2.39 (s, 3H, 4- $\text{CH}_3\text{C}_6\text{H}_4$), 7.23–7.81 (m, 13H, ArHs) and 8.41 (s, br, 1H, NH).
27a	1695, 1655, 1652 (COs) and 1600 (C=C).	2.22 (s, 3H, CH_3CO), and 7.21–7.83 (m, 14H, ArHs).
27b	1694, 1660, 1653 (COs) and 1600 (C=C).	2.21 (s, 3H, CH_3CO), 2.41 (s, 3H, 4- $\text{CH}_3\text{C}_6\text{H}_4$) and 7.15–7.82 (m, 13H, ArHs).
28b	1990, 1665, 1652 (COs) and 1600 (C=C)	2.38 (s, 3H, 4- $\text{CH}_3\text{C}_6\text{H}_4$) and 7.23–8.12 (m, 13H, ArHs).

filtration. The crude product was crystallized from ethanol to give a products identical in all respects (m.p., mixed m.p., and spectra) with the compound obtained by method A.

Nitrosation of **2a,b** and **10a,b**

A saturated solution of NaNO_2 (10 mL) was added dropwise to a solution of the appropriate 2,3-dihydro-1,3,4-thiadiazole **2a,b** or 2,3-dihydro-1,3,4-selenadiazole **10a,b** (1 g) in acetic acid (20 mL) with stirring at 0–5°C. The red precipitate in each case was collected, washed with water, and crystallized from ethanol, to give a 5-acyl-2,3-dihydro-2-*N*-nitroso-3-(3'-phenyl)pyrazol-5'-yl)-1,3,4-thiadiazole (**4a,b**) and 5-acetyl-2,3-dihydro-2-imino-3-(3'-phenyl)pyrazol-5'-yl)-1,3,4-selenadiazole (**11a,b**), respectively.

Decomposition of **4a,b** and **11a**

The appropriate *N*-nitroso derivative **4a,b** or **11a** (1 g) in xylene (10 mL) was heated for 10 minutes under

refluxing, conditions and the solution was evaporated under reduced pressure. The residual oil in each case was triturated with petroleum ether (40–60°C), and the resulting solid was collected and crystallized from acetic acid to give 2,3-dihydro-1,3,4-thiadiazolonones **5a,b** and **12a**, respectively.

Acylation of **2a,b** and **10a**

The appropriate **2a,b** and **10a** (1 g) was boiled with acetic anhydride (10 mL) for 5 minutes and poured onto crushed ice (10 g). The resulting solid was collected, washed, and then crystallized from acetic acid to give the *N*-acetyl derivatives **6a**, **7a**, and **13a**, respectively. An equimolar amount of each **2a,b** and **10a** and benzoyl chloride in pyridine was refluxed for 5 minutes, poured onto ice-cold water, and acidified with hydrochloric acid. The solid was collected, washed with hot water, and then crystallized from *N,N*-dimethylformamide to afford *N*-acetyl- and *N*-benzoyl derivatives **6b**, **7b**, and **14a**, respectively.

Syntheses of 2,3-Dihydro-1,3,4-thiadiazoles 19, 26–29 (a,b)

Equimolar amounts of indane-1,3-dione, potassium hydroxide and carbon disulfide (5 mmol, each) in *N,N*-dimethylformamide (15 mL) was stirred at room temperature for 3 hours until the potassium hydroxide dissolved, and then methyl iodide (0.5 mL) was added. Each hydrazonoyl halide **15**, **22–25 (a,b)** (5 mmol) was added to the solution with stirring and then triethylamine (0.75 mL) was added. The resulting solid was collected, washed, and then crystallized from acetic acid to afford thiadiazoles **19** and **26–29 (a,b)**, respectively.

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