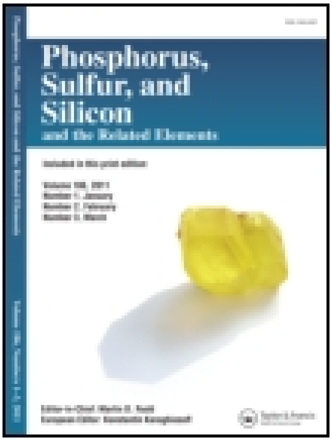

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2-(4-Naphtho[1,2-d]furan-2-yl-1,3-thiazol-2-yl)-ethanenitrile in Heterocyclic Synthesis

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2-(4-Naphtho[1,2-*d*]furan-2-yl-1,3-thiazol-2-yl)-ethanenitrile in Heterocyclic Synthesis

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*Coumarin, thiazole, and 2,3-dihydro-1,3,4-thiadiazole derivatives were synthesized from 2-(4-naphtho[1,2-*d*]furan-2-yl-1,3-thiazol-2-yl)ethanenitrile and different reagents. Structures of the new compounds were elucidated on the basis of elemental analysis, spectral data, and alternative methods of synthesis whenever possible.*

Keywords 2,3-Dihydro-1,3,4-thiadiazole; coumarin; hydrazonoyl halides; thiazole

INTRODUCTION

Recently, a large number of thiazole derivatives have been found to exhibit pharmacological activity.[1,2] They also were used as an anthelmintic,[3] fungicidal,[4] and antifungal activity, inhibiting in vivo the growth of Xanthomonas oryzae,[5] an ingredient of herbicides.[6] As an extension of our study[7–11], we report herein the synthesis of different thiadiazoles, coumarines, and pyrazolo[1,5-*a*]pyrimidines containing thiazole moiety.

RESULTS AND DISCUSSION

2-Bromo-1-naphtho[1,2-*d*]furan-2-ylethan-1-one (**1**) reacted with cyanothioacetamide in boiling ethanol under reflux and afforded 2-(4-naphtho[1,2-*d*]furan-2-yl-1,3-thiazol-2-yl)ethanenitrile (**2**). Structure **2** was confirmed on the basis of elemental analysis, spectral data, and chemical transformation. ^{1}H NMR (δ ppm) spectrum showed signals at $\delta = 4.12$ (s, 2H, CH_2CN), 7.51–8.19 (m, 8H, thiazole C-5, and ArH's). Its IR (cm^{-1}) revealed bands at 3101 (C—H), 2252 (CN) and 1620 (C=C).

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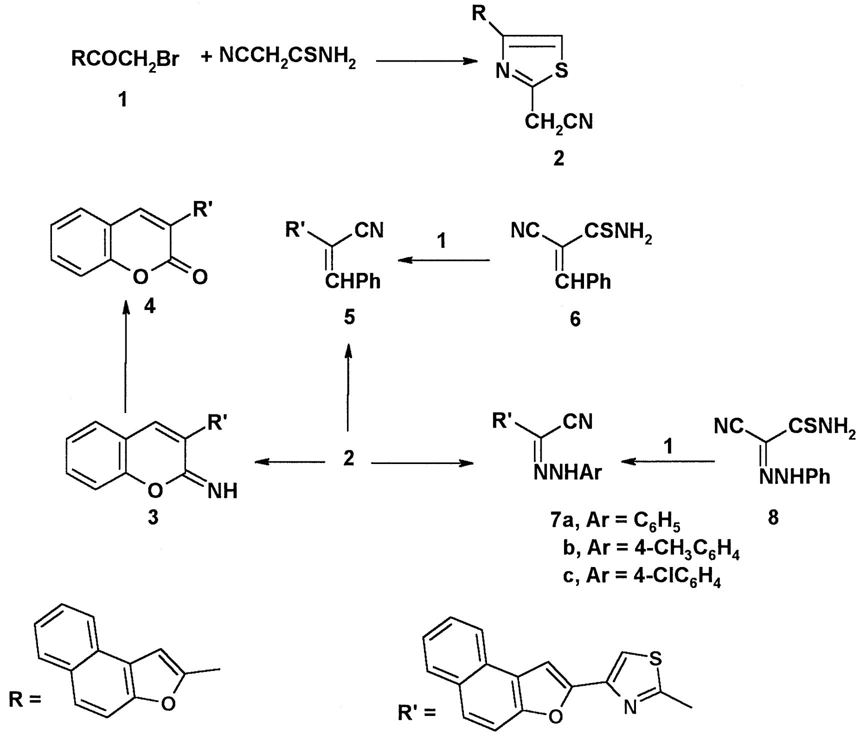
Compound **2** reacted with each of salicylaldehyde and benzaldehyde to give 3-(4-naphtho[1,2-*d*]furan-2-yl-1,3-hiazol-2yl)-2H-chromen-2-imine (**3**) and (2E)-2-(4-naphtho[1,2-*d*]furan-2-yl(1,3-thiazol-2-yl))-3-phenylprop-2-enenitrile (**5**), respectively (Scheme 1).



SCHEME 1

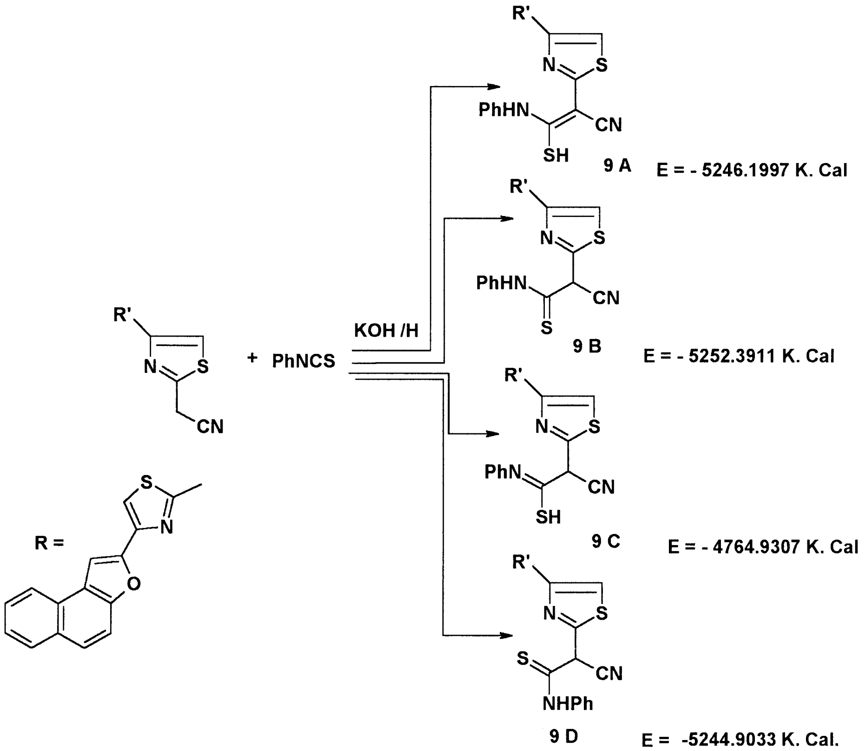
Structures of **3** and **5** were established on the basis of elemental analysis, spectral data, and chemical transformation (or alternative route synthesis). ^{1}H NMR (δ ppm) spectrum of **3** showed signals at $\delta = 6.68$–7.41 (m, 13H, ArH's) and 8.42 (s, 1H). Its IR (cm^{-1}) spectrum revealed bands at 3124 (NH), 3057 (CH), and 1599 (C=C). More evidence on structure **3** came from its conversion to 3-(4-naphtho[1,2-*d*]furan-2-yl-1,3-thiazole-2-yl)-2H-chromen-2-one (**4**) by hydrochloric acid. IR spectrum of **4** revealed bands at 3055 (CH), 1712 (CO), and no bands between 3150–3500 due the absence of NH group. ^{1}H NMR (δ ppm) spectrum of **5** showed one signal at δ = 7.00–7.41 (m, ArH's). Its IR (cm^{-1}) spectrum revealed bands at 3057 (CH), 2214 (CN), and 1604 (C=C). Thus, compound 2-(aminothioxomethyl)-3-phenylprop-2-enenitrile (**6**) reacted with naphtho[1,2-*d*]furan-2-ylethan-1-one (**1**) in boiling ethanol to give a product identical in all respects (mp., mixed mp., and spectra) with **5**.

Treatment of **2** with arenediazonium chloride in ethanolic sodium acetate solution at 0°C afforded (2E)-3-aza-3-(arylamino)-2-naphtho[1,2-*d*]furan-2-yl(1,3-thiazol-2-yl))-2-enenitrile **7a–c**, respectively. Structure **7** was confirmed on the basis of elemental analysis, spectral data, and chemical transformation. Thus, ^{1}H NMR spectrum of **7b** showed signals at δ = 2.37 (s, 3H), 7.26–8.20 (m, 12H), and 14.05 (s, br., 1H). Its IR revealed bands at 3349 (NH) and 2221 (CN). Its ^{13}C NMR showed signals at 24, 102, 111, 115, 116, 119, 121, 124, 127, 128, 129, 130, 140, 143, 153, 155 ppm. Also, treatment of the appropriate (2E)-2-aminothioxomethyl)-3aza)-3-(arylamino)prop-2-enenitrile **8a–c** with 2-Bromo-1-naphtho[1,2-*d*]furan-2-ylethan-1-one (**1**) in boiling acetic acid under reflux afford products identical in all respects (mp., mixed mp., and spectra) with **7a–c**.

Compound **2** reacted with phenyl isothiocyanate in *N*,*N*-dimethylformamide containing potassium hydroxide to give non-isolable product which converted to a product by hydrochloric acid. The resulting product seems to be one of the four isomers **9a–d** (cf. Scheme 2). According to M.O. calculation, using Hyper Chem. AM1 semi-empirical Method, the total energy proved that the most stable isomer formulated was 2-(4-naphtho[1,2-*d*]furan-2-yl(1,3-thiazol-2yl))-3-(phenylamino)-3-thioxopropanenitrile (**9B**). Also, structure **9** was elucidated by microanalytical, spectral data, and chemical transformation. Thus, treatment of **9** with iodomethane afforded (2E)-3-methylthio-2-(4-naphtho[1,2-*d*]furan-2-yl(1,3-thiazol-2-yl))-3-(phenylamino)prop-2-enenitrile (**10**) (cf. Scheme 3).

IR spectrum of **10** revealed characteristic bands at 3386 (NH) and 2198 (CN). Its ^{1}H NMR showed signals at $\delta = 2.27$ (s, 3H), 7.31–8.10 (m, 13H), and 12,53 (s, br., 1H). ^{13}C NMR showed signals at δ = 16, 71, 102, 111, 115, 116, 118, 122, 127, 129, 130, 143, 144, 150, 153, 155, and 167 ppm. Also, compound **10** reacted with hydrazine hydrate in boiling ethanol under reflux gave [5-Amino-4-(4-naphtho[1,2-*d*]furan-2-yl)(1,3-thiazol-2-y))pyrazol-3-yl]phenyl amine (**11**). Structure **11** was elucidated by elemental analysis and spectral data (cf. Experimental).

On the other hand, the appropriate hydrazonoyl halides **12a–h** reacted with **9** in ethanolic triethylamine at room temperature to give 2,3-dihydro-1,3,4-thiadiazole derivatives **15a–h**, respectively (Scheme 3). IR spectra of **15a–h** revealed bands near 2190 (CN) and 1720–1680 (CO). ^{1}H NMR spectrum of **15a** showed signals at $\delta = 4.15$ (s, 3H) and 7.52–8.31 (m, 13H) ppm. Its ^{13}C NMR spectrum showed signals at δ = 14, 61, 89, 115, 119, 118, 121, 122, 127, 128, 129, 130, 146, 149, 150, 153, 154, 155, 162, and 163 ppm.



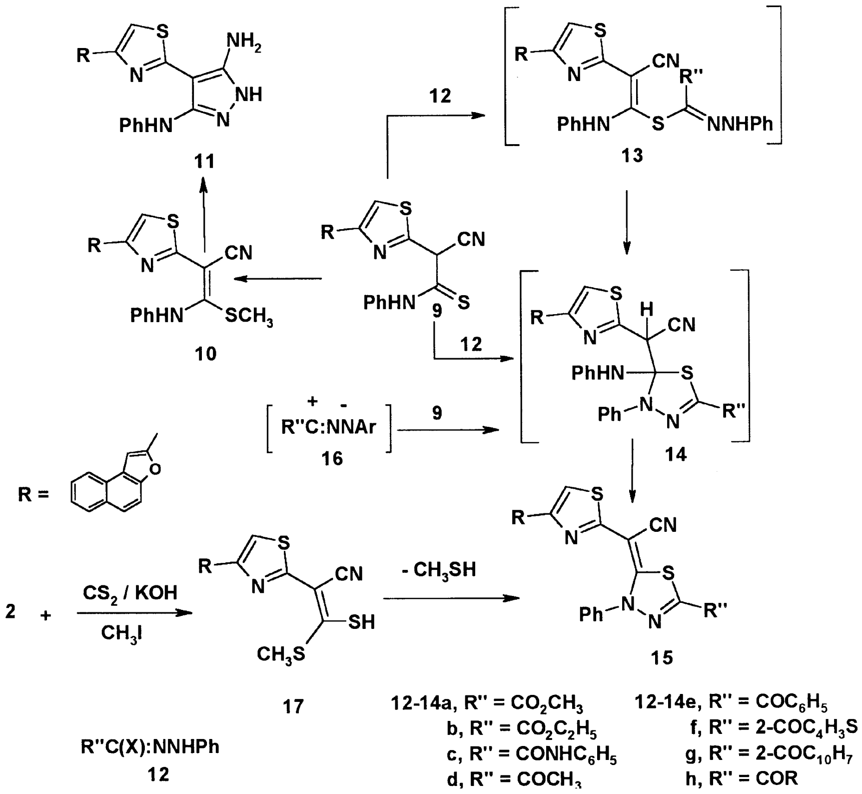
SCHEME 2

Also, C-methoxy-N-phenylhydrazonoyl chloride **12a** reacted with 3-methylthio-2-(4-naphtho[1,2-*d*]furan-2-yl(1,3-thiazol-2-yl)-3-thioxopropanenitrile (**17**) in ethanolic triethylamine to give product identical in all respects (mp., mixed mp., and spectra) with **15a** .

In the light of the forgoing results, the mechanism outlined in Scheme 3 seems to be the most plausible pathway for the formation of **15** from the reaction of the **9** with **12** (or nirile imine **16**, which prepared in situ by treatment of **12** with triethylamine). The reaction involves initial formation of thiohydrazonate **13**, which undergoes intermolecular cyclization as soon as it is formed to yield the intermediate **14** or via 1,3-dipolar cycloaddition of nitrilimine **16** to C=S double bond of **9** to give the final product **15** via elimination of aniline.

EXPERIMENTAL

All melting points were determined on an electrothermal apparatus and are uncorrected. IR spectra were recorded (KBr discs) on a Shimadzu



SCHEME 3

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 are expressed in δ units using TMS as an internal reference. Mass spectra were recorded on a GC-MS QP1000 EX Shimadzu. Elemental analysis were carried out at the Microanalytical Center of the Cairo University. Hydrazonoyl halides were obtained as previously reported.[12–18]

SYNTHESIS OF 2-(4-NAPHTHO[1,2-*d*]FURAN-2-yl-1,3-THIAZOL-2-yl)ETHANENITRILE (2)

A mixture of 2-Bromo-1–naphtho[1,2-*d*]furan-2-ylethan-1-one (**1**),[19] (2.9 g, 0.01 mmol) and cyanothioacetamide (1.0 g, 0.01 mmol) in ethanol (25 mL) was refluxed for 2 h. The reaction mixture was poured onto ice-cold water (50 mL) and drops of conc. ammonium hydroxide (25%) were added. The resulting solid was collected and washed with water, and recrystallized from ethanol to give thiazolylacetonitrile **2** (Table I).

TABLE I Characterization Data of the Newly Synthesized Compounds

Corp. no.	Mp., °C solvent	Color yield%	Mol. formula mol. wt.	Calcd./found%			
				C	H	N	S
2	175–78	Pale Brown	$C_{17}H_{10}N_2OS$	70.33	3.47	9.69	11.04
	EtOH	69	290.35	70.45	3.30	9.80	11.15
3	228–30	Yellow	$C_{24}H_{14}N_2O_2S$	73.08	3.58	7.10	8.13
	DMF	64	394.46	72.85	3.80	7.00	8.25
4	268–70	Yellowish gray	$C_{24}H_{13}NO_3S$	72.90	3.54	12.14	8.11
	AcOH	61	395.44	73.10	3.40	12.00	7.95
5	209–210	Yellow	$C_{24}H_{14}N_2OS$	76.17	3.73	7.40	8.47
	DMF	52	378.46	76.25	3.65	7.20	8.65
7a	232–34	Brown	$C_{23}H_{14}N_4OS$	70.03	3.58	14.20	8.13
	Dioxane	40	394.46	69.80	3.68	13.95	8.25
7b	230–32	Brown	$C_{24}H_{16}N_4OS$	70.57	3.95	13.72	7.85
	DMF	41	408.49	70.70	4.15	13.94	7.71
7c	265–67	Brown	$C_{23}H_{13}ClN_4OS$	64.41	3.06	13.06	7.48
	Dioxane	39	428.90	64.50	2.90	12.90	7.70
9	220–22	Brown	$C_{24}H_{15}N_3OS_2$	67.74	3.55	9.87	15.07
	Dioxane	67	425.53	67.50	3.70	9.70	14.80
10	207–209	Yellowish gray	$C_{25}H_{17}N_3OS_2$	68.31	3.90	9.56	14.59
	DMF	58	439.56	68.20	3.80	9.72	14.82
11	244–46	Brown	$C_{24}H_{17}N_5OS$	68.07	4.05	16.54	7.57
	AcOH	72	423.50	67.90	4.10	16.45	7.40
15a	275–77	Yellow	$C_{27}H_{16}N_4O_3S_2$	63.77	3.17	11.02	12.61
	DMF	60	508.58	63.92	3.10	10.97	12.42
15b	302–304	Yellowish brown	$C_{28}H_{18}N_4O_3S_2$	64.35	3.47	10.72	12.27
	DMF	66	522.61	64.10	3.32	10.90	12.40
15c	274–76	Yellowish brown	$C_{32}H_{19}N_5O_2S_2$	67.47	3.36	12.29	11.26
	DMF	63	569.67	67.29	3.25	12.10	11.40
15d	198–200	Yellowish brown	$C_{27}H_{16}N_4O_2S_2$	65.84	3.27	11.37	13.02
	DMF	59	492.58	65.900	3.35	11.50	12.90
14e	243–45	Brown	$C_{32}H_{18}N_4O_2S_2$	69.30	3.27	10.10	11.56
	DMF	69	554.65	69.40	3.00	9.95	11.65
15f	265–67	Brown	$C_{30}H_{16}N_4O_2S_3$	64.27	2.88	9.99	17.16
	DMF	64	560.68	64.00	2.60	10.10	17.00
15g	265–268	Brown	$C_{36}H_{20}N_4O_2S_2$	71.51	3.33	9.27	10.60
	DMF	67	604.71	71.30	3.50	9.40	10.70
15h	>320	Brown	$C_{38}H_{20}N_4O_3S_2$	70.79	3.13	8.69	9.95
	DMF	61	644.74	70.90	3.10	8.50	10.20
17	272–75	Brown	$C_{19}H_{12}N_2OS_3$	59.97	3.18	7.36	25.28
	DMF	62	380.51	59.70	3.00	7.50	25.40

SYNTHESIS OF 3-(4-NAPHTHO[1,2-*d*]FURAN-2-yl-1,3-HIAZOL-2yl)-2H-CHROMEN-2-IMINE (3) AND (2E)-2-(4-NAPHTHO-[1,2-*d*]FURAN-2-yl(1,3-THIAZOL-2-yl))-3-PHENYLPROP-2-ENENITRILE (5)

Method A: Thiazolylacetonitrile **2** (1.45 g, 5 mmol) and salicylaldehyde or benzaldehyde (5 mmol) in ethanol (20) containing a catalytic amount of piperidine were stirred at room temperature for 2 h. The resulting solid was collected and recrystallized to give **3** and **5**, respectively (Table I).

Method B: Equimolar amounts of 2-(aminothioxomethyl)-3-phenyl-prop-2-enenitrile[20] (**6**) and naphtho[1,2-*d*]furan-2-ylethan-1-one (**1**) (5 mmol) in acetic acid (15 mL) was boiled under reflux for 2 h. The resulting solid was collected and crystallized to give a product identical (mp., mixed mp, and spectra) with **5**

SYNTHESIS OF 3-(4-NAPHTHO[1,2-*d*]FURAN-2-yl-1,3-THIAZOLE-2-yl)-2H-CHROMEN-2-ONE (4)

A mixture of **3** (0.5 g) and hydrochloric acid (5 mL, 6 M) was stirred at room temperature for 1 h. The resulting solid was collected and recrystallized from acetic acid to give substituted coumarin **4** (Table I).

SYNTHESIS OF (2E)-3-AZA-3-(ARYLAMINO)-2-NAPHTHO[1,2-*d*]FURAN-2-yl(1,3-THIAZOL-2-yl))-2-ENENITRILE 7a–c

Method A: The appropriate arenediazonium chlorides (5 mmol) was added dropwise to a cold ethanol solution, containing thiazolyacetonitrile **2** (1.45 g, 5 mmol) and sodium acetate (1 g), (50 mL) at 0–5°C while stirring. The mixture was stirred for 3 h at 0–5°C. The resulting solid was collected and crystallized from dioxane to give **7a–c** (Tables I and II).

Method B: A mixture of the appropriate (2E)-2-aminothioxomethyl)-3aza)-3-(arylamino)prop-2-enenitrile[21] **8a–c** and 2-Bromo-1-naphtho-[1,2-*d*]furan-2-ylethan-1-one (**1**) (5 mmol each) in ethanol (30 mL) was boiled under reflux for 2 h. The resulting solid, which formed after cooling, was collected and crystallized from acetic acid to afford a product identical in all respects (mp., mixed mp., and spectra) with products obtained in method A.

SYNTHESIS OF THIOANILID 9

Phenyl isothiocyanate (0.65 g (0.6 mL), 5 mmol) was added to a mixture of thiazolylacetonitrile (1.45 g, 5 mmol) **2** and potassium hydroxide

TABLE II ^{1}H NMR Spectra of Some Newly Synthesized Compounds

Comp. no.	Spectral data
2	^{1}H NMR: 4.22 (s, 3H), 7.51–8.19 (m, 8H) IR: 2252 (CN)
4	IR: 1660 (C=N), 1604 (C=C)
5	IR: 1712 (CO), 1604 (C=C)
7a	^{1}H NMR: 7.5–8.12 (m, 13H), 8.21 (s, 1H) IR: 3342 (NH), 2214 (CN) Mass: 378 (100%), 223 (4.2%), 195 (42%), 152 (48.8%).
7b	IR: 3349 (NH), 221 (CN)
7c	^{1}H NMR: 2.37 (s, 3H), 7.26–8.20 (m, 12H) and 14.05 (s, br., 1H). IR: 3349 (NH) and 2221 (CN).
9	IR: 3350 (NH), 2214 (CN), 1651 (C=N), 1569 (C=C)
10	IR: 3323 (NH), 2177 (CN), 1623 (C=N)
11	^{1}H NMR: 4.10 (s, br., 2H), 6.70–7.67 (m, 14H), 9.32 (s, br., 1H). IR: 3350, 3280, 3150 (NH_2, NH), 3050 (CH), 1620 (C=N), 1596 (C=C).
15a	^{1}H NMR: 2.27 (s, 3H), 7.31–8.10 (m, 13H) and 12,53 (s, br., 1H). IR: 2191 (CN), 1720 (CO), 1666 (C=N)
15b	^{1}H NMR: 1.56 (t, 3H), 4.55 (s, 2H), 7.60–8.29 (m, 13H) and 12,53 (s, br., 1H). IR: 2191 (CN), 1743 (CO), 1624 (C=N)
15d	^{1}H NMR: 2.20 (s, 3H), 6.46 (d, 4H), 6.72 (s, 1H), 7.01–7.67 (m, 10H), 8.21 (s, 1H). ^{13}C NMR: 23, 89, 102, 111, 115, 118, 121, 124, 127, 128, 129, 143, 146, 149, 150, 153, 154, 155, 194. IR: 2198 (CN), 1685 (CO), 1596 (C=C)
15f	^{1}H NMR: 2.40 (s, 3H), 7.50–8.32 (m, 13) IR: 2191 (CN), 1674 (CO)
15g	IR: 2191 (CN), 1680 (CO), 1635 (C=N)
17	1H NMJR: 1.51 (s, 1H), 2.25 (s, 3H), 6.71–7.76 (m, 8H).

(0.28 g, 5 mmol) in *N*,*N*-dimethylformamide (15 mL) while stirring at room temperature until potassium hydroxide dissolve completely, and the reaction mixture was continued stirring for 1 h. The reaction mixture was poured onto water (30 mL) and acidifies with acetic acid. The resulting solid was collected and recrystallized from dioxin to give thioanilide **9** (Table I).

SYNTHESIS OF (2E)-3-METHYLTHIO-2-(4-NAPHTHO[1,2-*d*]FURAN-2-yl(1,3-THIAZOL-2-yl))-3-(PHENYLAMINO)PROP-2-ENENITRILE (10)

Phenyl isothiocyanate (0.65 g, (0.6 mL), 5 mmol) was added to a mixture of thiazolylacetonitrile (1.45 g, 5 mmol) **2** and potassium hydroxide

(0.28 g, 5 mmol) in *N*,*N*-dimethylformamide (15 mL) while stirring at room temperature until potassium hydroxide dissolve completely, and the reaction mixture was stirred for 1 h, then Iodomethane (0.71 g, 5 mmol) was added dropwise while stirring. The reaction mixture was stirred for 2 h and the resulting solid was collected and recrystallized from *N*,*N*-dimethylformamide to give **10** (Table I).

SYNTHESIS OF [5-AMINO-4-(4-NAPHTHO[1,2-*d*]FURAN-2-yl)(1,3-THIAZOL-2-y)PYRAZOL-3-yl]-PHENYLAMINE (11)

A mixture of **10** (2.18 g, 5 mmol) and hydrazine hydrate (1 m, 99%) in ethanol (20 mL) was boiled under reflux for 4 h. The resulting solid was collected and crystallized from *N*,*N*-dimethylformamide to give **11** (Table I).

SYNTHESIS OF 2,3-DIHYDRO-1,3,4-THIADIAZOLES 15a–h

Method A: Phenyl isothiocyanate (0.65 g (0.6 mL), 5 mmol) was added to a mixture of thiazolylacetonitrile (1.45 g, 5 mmol) **2** and potassium hydroxide (0.28 g, 5 mmol) in N,N-dimethylformamide (15 mL) while stirring at room temperature until potassium hydroxide dissolved completely and the reaction mixture was stirred for 30 min. The appropriate hydrazonoyl halides **12a–h (**5 mmol) were added and stirred for 3 h. The resulting solid was collected and recrystallized from the appropriate solvent to afford **15a–h** (Table I).

Method B: Triethylamine (0.5 g, (0.75 mL), 5 mmol) was added dropwise to a mixture of the appropriate hydrazonoyl halides **12a–h** and **17** (1.9 g, 5 mmol) in ethanol (20 mL) while stirring. Stirred was continued for 1 h and the resulting solid was collected and crystallized to give products identical in all respects (mp., mixed mp., and spectra) with those obtained in method A.

SYNTHESIS OF 3-METHYLTHIO-2-(4)-NAPHTHO[1,2-*d*]FURAN-2-yl(1,3-THIAZOL-2-yl)-3-THIOXOPROPANENITRILE (17)

A mixture of thiazolylacetonitrile **2** (1.45 g, 5 mmol), potassium hydroxide (0.28 g (5 mmol) and carbon disulfide (0.47 g, 5 mmol) in *N*,*N*-dimethylformamide (15 mL) was stirred for 6 h at room temperature. Iodometane (0.71 g, 5 mmol) was added dropwise to the above mixture and stirred for 1 h. The resulting solid was collected and crystallized from acetic acid to give **17** (Tables I and II)

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