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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.

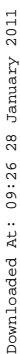
RESULTS AND DISCUSSION

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. ¹H NMR (δ ppm) spectrum showed signals at δ = 4.12 (s, 2H, CH₂CN), 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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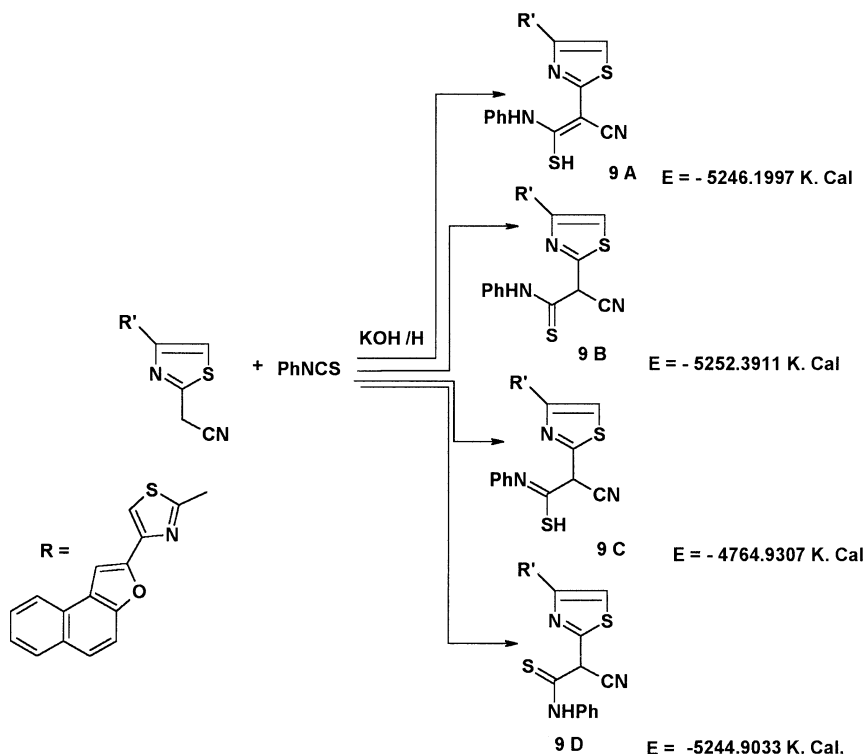
to give a product identical in all respects (mp., mixed mp., and spectra) with **5**.

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, ¹H 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 ¹³C 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-2-yl))-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 ¹H NMR showed signals at δ = 2.27 (s, 3H), 7.31–8.10 (m, 13H), and 12.53 (s, br., 1H). ¹³C 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-yl)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). ¹H NMR spectrum of **15a** showed signals at δ = 4.15 (s, 3H) and 7.52–8.31 (m, 13H) ppm. Its ¹³C 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.



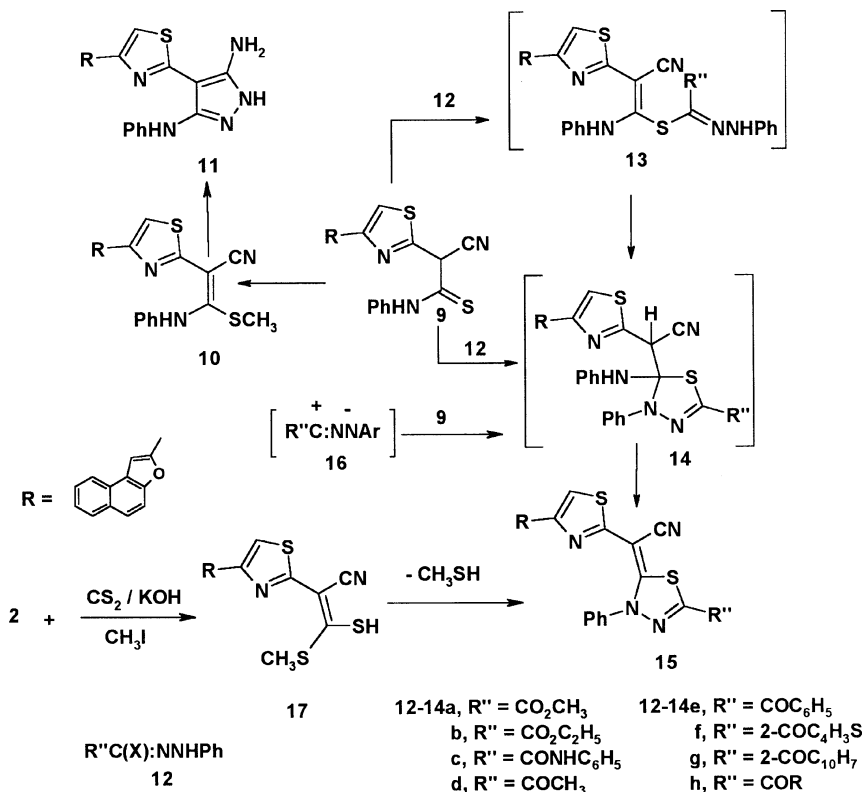
SCHEME 2

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-thioxo-propanenitrile (**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 nitrile imine **16**, which prepared in situ by treatment of **12** with triethylamine). The reaction involves initial formation of thiohydrazone **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. ^1H NMR spectra were recorded in CDCl_3 and $(\text{CD}_3)_2\text{SO}$ 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 EtOH	Pale Brown 69	C ₁₇ H ₁₀ N ₂ OS 290.35	70.33 70.45	3.47 3.30	9.69 9.80	11.04 11.15
3	228–30 DMF	Yellow 64	C ₂₄ H ₁₄ N ₂ O ₂ S 394.46	73.08 72.85	3.58 3.80	7.10 7.00	8.13 8.25
4	268–70 AcOH	Yellowish gray 61	C ₂₄ H ₁₃ NO ₃ S 395.44	72.90 73.10	3.54 3.40	12.14 12.00	8.11 7.95
5	209–210 DMF	Yellow 52	C ₂₄ H ₁₄ N ₂ OS 378.46	76.17 76.25	3.73 3.65	7.40 7.20	8.47 8.65
7a	232–34 Dioxane	Brown 40	C ₂₃ H ₁₄ N ₄ OS 394.46	70.03 69.80	3.58 3.68	14.20 13.95	8.13 8.25
7b	230–32 DMF	Brown 41	C ₂₄ H ₁₆ N ₄ OS 408.49	70.57 70.70	3.95 4.15	13.72 13.94	7.85 7.71
7c	265–67 Dioxane	Brown 39	C ₂₃ H ₁₃ ClN ₄ OS 428.90	64.41 64.50	3.06 2.90	13.06 12.90	7.48 7.70
9	220–22 Dioxane	Brown 67	C ₂₄ H ₁₅ N ₃ OS ₂ 425.53	67.74 67.50	3.55 3.70	9.87 9.70	15.07 14.80
10	207–209 DMF	Yellowish gray 58	C ₂₅ H ₁₇ N ₃ OS ₂ 439.56	68.31 68.20	3.90 3.80	9.56 9.72	14.59 14.82
11	244–46 AcOH	Brown 72	C ₂₄ H ₁₇ N ₅ OS 423.50	68.07 67.90	4.05 4.10	16.54 16.45	7.57 7.40
15a	275–77 DMF	Yellow 60	C ₂₇ H ₁₆ N ₄ O ₃ S ₂ 508.58	63.77 63.92	3.17 3.10	11.02 10.97	12.61 12.42
15b	302–304 DMF	Yellowish brown 66	C ₂₈ H ₁₈ N ₄ O ₃ S ₂ 522.61	64.35 64.10	3.47 3.32	10.72 10.90	12.27 12.40
15c	274–76 DMF	Yellowish brown 63	C ₃₂ H ₁₉ N ₅ O ₂ S ₂ 569.67	67.47 67.29	3.36 3.25	12.29 12.10	11.26 11.40
15d	198–200 DMF	Yellowish brown 59	C ₂₇ H ₁₆ N ₄ O ₂ S ₂ 492.58	65.84 65.900	3.27 3.35	11.37 11.50	13.02 12.90
14e	243–45 DMF	Brown 69	C ₃₂ H ₁₈ N ₄ O ₂ S ₂ 554.65	69.30 69.40	3.27 3.00	10.10 9.95	11.56 11.65
15f	265–67 DMF	Brown 64	C ₃₀ H ₁₆ N ₄ O ₂ S ₃ 560.68	64.27 64.00	2.88 2.60	9.99 10.10	17.16 17.00
15g	265–268 DMF	Brown 67	C ₃₆ H ₂₀ N ₄ O ₂ S ₂ 604.71	71.51 71.30	3.33 3.50	9.27 9.40	10.60 10.70
15h	>320 DMF	Brown 61	C ₃₈ H ₂₀ N ₄ O ₃ S ₂ 644.74	70.79 70.90	3.13 3.10	8.69 8.50	9.95 10.20
17	272–75 DMF	Brown 62	C ₁₉ H ₁₂ N ₂ OS ₃ 380.51	59.97 59.70	3.18 3.00	7.36 7.50	25.28 25.40

SYNTHESIS OF 3-(4-NAPHTHO[1,2-*d*]FURAN-2-yl)-1,3-HIAZOL-2-yl)-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-phenylprop-2-enenitrile²⁰ (**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 thiazolylacetonitrile **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-enenitrile²¹ **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 ^1H NMR Spectra of Some Newly Synthesized Compounds

Comp. no.	Spectral data
2	^1H 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	^1H 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	^1H 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	^1H 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	^1H 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	^1H 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	^1H 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	^1H 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-yl)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 hydrazoneoyl 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 hydrazoneoyl 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. Iodomethane (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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