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Simple Syntheses of Benzothiazol-2-ylazoles

El-Sayed M. A. Yakout, Yahia A. Allam, and Galal A. M. Nawwar

Department of Pesticide Chemistry, National Research Centre, Dokki, Cairo, Egypt

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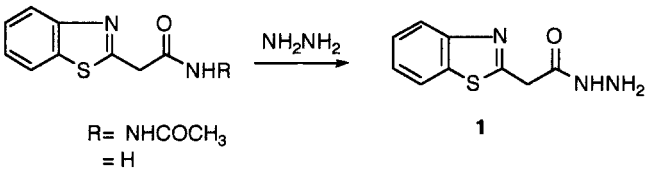
ABSTRACT: *Syntheses of the title ring systems are described starting with benzothiazol-2-ylacetohydrazide* (**1**). *Thus,* **1** *was reacted with carbon disulfide to afford the 2-methyl heteroaryl derivative* **2**, *which on reaction with hydrazine hydrate yielded the corresponding triazole compound* **3**. *Also,* **1** *can undergo a reaction with an isothiocyanate to give the N-thiocarbonyl adduct* **4** *that can then be cyclized to produce a 2-methyl heteroaryl analog* **5** *or* **6**. *Compounds* **8** *or* **9** *could be obtained by the reaction of* **1** *with an aryl aldehyde followed by malononitrile or via its self-cyclization, respectively.* © 1999 John Wiley & Sons, Inc. Heteroatom Chem 10: 177–182, 1999

INTRODUCTION

The chemistry of 2-heteroarylbenzoazoles is quite interesting due to its application in the agrochemical field [1,2]. Furthermore, cyanoaceto-hydrazides are versatile reagents extensively used in organic synthesis [3,4]. On these bases, benzothiazol-2-yl-acetohydrazides were considered to be the precursors of choice in the present work, as they seemed to combine both advantages, that is, potential biological effectiveness and chemical reactivity.

RESULTS AND DISCUSSION

Benzothiazol-2-ylacetohydrazide can be prepared either by the reaction of 2-aminothiophenol with cyanoacetohydrazide [3] or by the reaction of N-acetylbenzothiazol-2-ylacetohydrazide or benzothiazol-2-ylacetamide with hydrazine hydrate in methanol. However, we used the second procedure because it gives a far better yield (80%) than the first one, the yield in the former not exceeding 30% due to the self-cyclization of a part of cyanoacetohydrazide under the reaction conditions to form the corresponding aminopyrazolone.



Benzothiazol-2-ylacetohydrazide (**1**) was reacted with carbon disulfide in ethanol in the presence of a catalytic amount of potassium hydroxide with subsequent treatment with hydrochloric acid. The product isolated showed by ^{1}H-NMR spectroscopy, the signals of methylene group protons similar to those previously detected in the parent compound at δ = 4.0 but with one (D_2O) exchangeable singlet at δ = 10.2 instead of the two (D_2O) exchangeable singlets present in the parent compound. Moreover, the mass spectrum showed the ion base peak at *m/z* (M^+ 176, 100%), which is attributed to the carboxymethyl benzothiazole fragment C_9H_6NOS, while the molecular ion peak appeared at *m/z* (M^+ 249, 14%). These data along with the microanalytical data could be interpreted to be consistent with the benzothiazol-2-methyloxadiazole structure **2**.

Upon repetition of the reaction of **1** with carbon disulfide in ethanol in the presence of potassium hydroxide as a catalyst, followed by treatment with hydrazine hydrate, the benzothiazol-2-methyltriazole derivative **3** was obtained in a moderate yield. The latter showed an IR NH_2 absorption at 3300 cm^{-1} which also appeared as a (D_2O) exchangeable singlet

Correspondence to: Galal A. M. Nawwar
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at $\delta = 5.4$ in its ^{1}H-NMR spectrum together with the resonances of the active methylene group protons. Moreover, the mass spectrum of **3** gave a molecular ion peak, as a base ion peak, at *m/z* (M^+ 263, 100%), compatible with the given structure.

Compound **3** could be obtained when compound **2** was allowed to react with hydrazine hydrate in methanol, a result which is in accord with previous reports [5].

Moreover, it was found that other benzothiazol-2-ylmethyltriazoles could be synthesized starting with the aryl carbamate derivatives **4**, which were obtained in good yield upon reaction of the hydrazide **1** with benzyl isothiocyanate or phenyl isothiocyanate, respectively, in dry dioxane containing a catalytic amount of triethylamine. Structure **4** was established on the basis of elemental analyses, spectral data (cf. experimental), and analogy [6].

When **4a** was treated with sodium carbonate (20%), cyclization occurred, and it was accompanied by loss of a water molecule to produce the expected benzyltriazole derivative **5a,** similar to previous reports [6]. Its IR spectrum lacked the carbonyl absorption present in the parent compound at 1700 cm^{-1}, but the two active methylene groups (similar to those found in **4a**) were detected in its ^{1}H-NMR spectrum at $\delta = 4.6$ and 5.3, as well as the resonances of the aromatic protons at their expected locations. Also, it gave a molecular ion peak at *m/z* (M^+ 338, 100%) compatible with the proposed structure.

A parallel result was obtained upon repeating the reaction sequence with **4b** instead of **4a,** thus affording the corresponding phenyltriazole **5b,** the structure of which was deduced from elemental analyses and spectral data (cf. experimental).

The carbamate **4a** was also heated at 100°C in concentrated H_2SO_4, affording a new product lacking the IR carbonyl group absorption band and the benzyl group protons in its ^{1}H-NMR spectrum but that nevertheless revealed the signals of the methylene group protons similar to those detected in the starting compound at $\delta = 4.7$ together with the benzothiazole protons and a (D_2O) exchangeable signal (2H) at $\delta = 7.15$. Also, its mass spectrum showed the molecular ion peak at *m/z* (M^+ 248, 35%). These data are consistent with the benzothiazol-2-methylthiadiazole structure **6a.** The reaction was also applied to the phenyl derivative **4b,** the product obtained showing spectral features similar to **6a** but also revealing the presence of the phenyl protons in its ^{1}H-NMR spectrum. Accordingly, the phenylaminothiadiazole structure **6b** has been given to the obtained product, and its mass spectrum, with a molecular formula $C_{16}H_{12}N_4S_2$ (*m/z* 324), was in accord with its structure.

It is assumed that the benzylamide side chain in **6a** was removed by acid hydrolysis under the reaction conditions to afford the free amino group, while the aromatic amino group in **6b** was stable under such reaction conditions.

The hydrazide **1** condensed readily with furan-2-aldehyde or 4-chlorobenzaldehyde, respectively, in methanol to afford the corresponding hydrazones **7**. Structure **7** was established by elemental analyses and ^{1}H-NMR and IR spectral data where the anil proton was detected at $\delta = 8.3$ in the ^{1}H-NMR spectra. The mass spectrum of **7b** gave a molecular ion peak compatible with the given structure at *m/z* (M^+ 329, 5%).

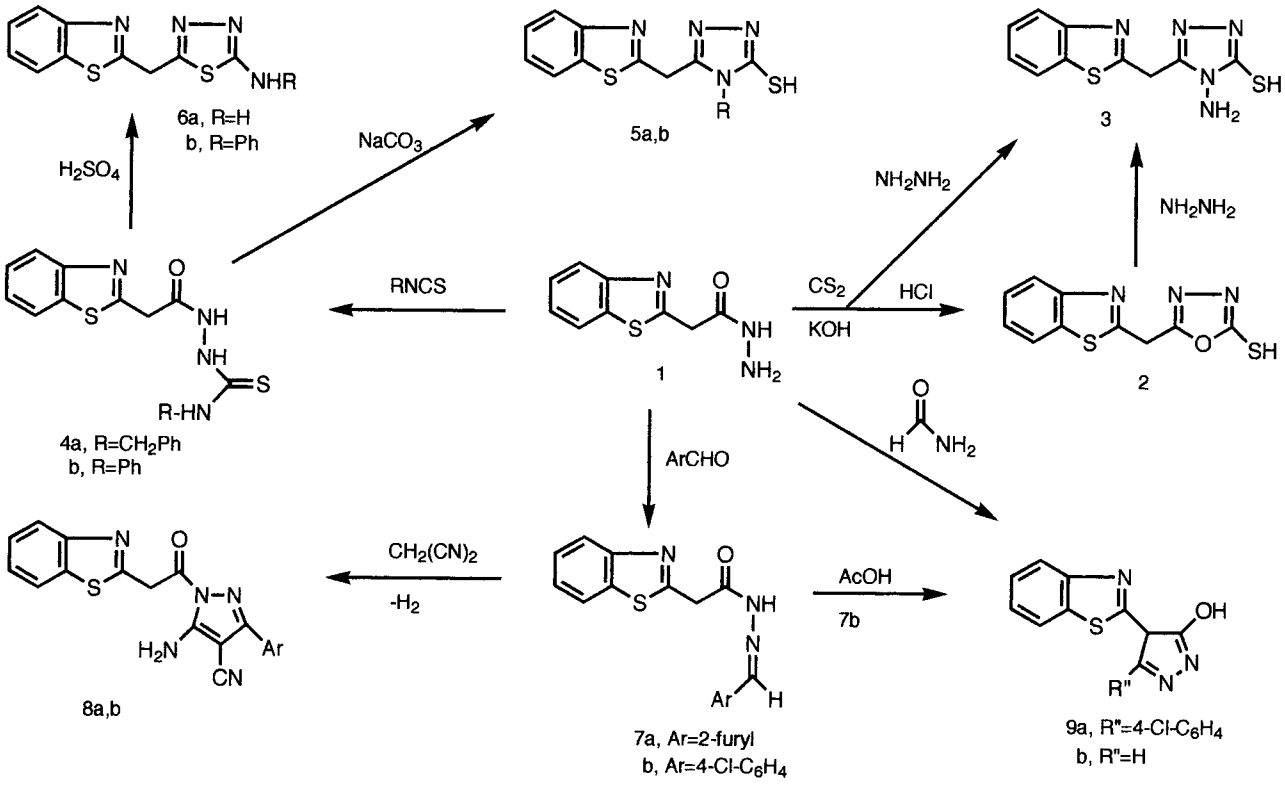
When compound **7a** was treated with malononitrile in methanol in the presence of triethylamine, a new product was obtained showing IR carbonyl, cyano, and amino group absorptions. Its ^{1}H-NMR spectrum revealed, in addition to the aromatic protons, a lower field methylene group signal at $\delta = 5.7$ in comparison with that detected in the parent compound **7a** at $\delta = 4.5$. It also showed a (D_2O) exchangeable singlet (2H) at $\delta = 8.1$. Its mass spectrum showed a molecular formula compatible with $C_{17}H_{11}N_5O_2S$ with *m/z* (M^+ 349, 20%). Among different proposed structures and based on these data, the benzothiazolyl-acetopyrazole structure **8a** is suggested for this product.

A parallel result was obtained when the reaction was carried out with **7b** instead of **7a,** affording **8b** in high yield. Structure **8b** was assigned based on elemental analyses and spectral data that showed similar features to those discussed for **8a** (cf. Experimental).

The formation of **8** probably proceeds via an initial nucleophilic addition of the malononitrile to the C=N group affording the corresponding 1:1 adduct that underwent cyclization to the pyrazoline intermediate, the latter then being dehydrogenated to the final product **8.** Similar dehydrogenation was previously reported by Soto et al. [7], who explained that the benzylidene malononitrile was the reaction oxidant. In our case, we assume that a catalytic amount of benzylidene malononitrile was formed due to the expected arylidene exchange between malononitrile and the hydrazone **7** [8].

The hydrazone **7b** was also refluxed in glacial acetic acid affording another pyrazole derivative for which the structure **9a** was proposed to result from cyclization and dehydrogenation. Thus, it showed a ^{1}H-NMR signal at $\delta = 7.65$ attributable to the pyrazole H-4 together with the aromatic protons at their expected locations in accord with a previous report [9].

It was also found that **9b** could be obtained in



SCHEME 1

satisfactory yield when the hydrazide **1** was heated in formamide at 100°C. Thus, the product obtained showed spectral similarities to compound **9a** except for the disappearance of the phenyl signal and the presence of two doublets at δ = 6.15 and δ = 6.9 attributable to the pyrazole H-3 and H-4 resonances, respectively. Accordingly, structure **9b** was assigned to this product, which was also supported by its mass spectral data.

From these results, it could be concluded that the hydrazide **1** could be used as precursor for the synthesis of benzothiazoles bearing a five-membered ring heterocycle (with two or three hetero atoms) at position **2** through simple procedures.

Experimental

Melting points are uncorrected and were taken on an Electrothermal 9100 apparatus. IR spectra were recorded on a Carl Zeiss spectrophotometer model "UR 10" using KBr. ^{1}H-NMR spectra were obtained on a Jeol 270 MHz instrument using tetramethylsilane as an internal standard. Mass spectra (MS) were recorded on a Finigan SSQ 7000 mass spectrometer. Microanalyses were performed by the Central Service Laboratory at Cairo University.

Benzothiazol-2-ylacetohydrazide (1)

N-Acetylbenzothiazol-2-ylacetohydrazide or benzothiazol-2-yl-acetamide (0.01 mol) was refluxed with hydrazine hydrate 80% (0.02 mol) in methanol (30 mL) for 2 hours. The precipitate formed after cooling of the reaction mixture was filtered off, crystallized from methanol, and identified as **1** (mp and mix. mp 155–157) [3].

2-(Benzothiazol-2-methyl)-1,3,4-oxadiazol-5-thiol (2)

To a solution of compound **1** (0.01 mol, 2.07 g) in ethanolic potassium hydroxide (20 mL), carbon disulfide (20 mL) was dropped in over a period of half hour at room temperature (25°C). After an additional half hour of stirring, a precipitate was formed and collected by filtration. The salt formed was dissolved in water, then acidified with HCl, and the precipitate thus formed was filtered off and crystallized from methanol, mp 157–159°C; yield 60%. Anal. calcd for $C_{10}H_7N_3OS_2$ (249.31): C, 48.18; H, 2.83; N, 16.86; S, 25.72%. Found: C, 48.07; H, 2.70; N, 16.52; S, 25.54%. IR (cm^{-1}): γ 2900–2700 (SH). ^{1}H-NMR (DMSO-d_6): δ 4.0 (s, 2H, CH_2); 7.4–7.5 (m, 2H, benzothiazole H-5 and H-6); 7.9–8.1 (m, 2H, benzothia-

zole H-4 and H-7); 10.2 (brs, 1H, SH). MS: *m/z* (M^+ 249, 14%).

4-Amino-3-(benzothiazol-2-methyl)-1,2,4-triazol-5-thiol **(3)**

Method A. Compound **1** (0.001 mol) was treated with carbon disulfide as previously mentioned. The salt formed was refluxed with an equimolar amount of hydrazine hydrate 80% in methanol (30 mL) for 3 hours. After the mixture had been cooled, drops of water were added until a precipitate formed. The solid thus formed was collected and crystallized from methanol.

Method B. Compound **2** (0.01 mol, 2.49 g) was refluxed with hydrazine hydrate 80% (0.01 mL) in methanol (25 mL) for 2 hours. The solution was partially concentrated and cooled; the precipitate formed was filtered off and crystallized from methanol: mp 233–235°C; yield 45%. Anal. calcd for $C_{10}H_9N_5S_2$ (263.34): C, 45.61; H, 3.45; N, 26.60; S, 24.35%. Found: C, 45.43; H, 3.19; N, 26.37; S, 24.2%. IR (cm^{-1}): γ 3300 (NH_2), 2950–2750 (SH); 1640 (C=N). ^{1}H-NMR (DMSO-d_6): δ 4.7 (s, 2H, CH_2); 5.4 (br s, 2H, NH_2); 7.5 (m, 2H, benzothiazole H-5 and H-6); 8.0 (m, 2H, benzothiazole H-4 and H-7). MS: *m/z* (M^+ 263, 100%).

1-(Benzothiazol-2-acetyl)-4-aryl Thiosemicarbazides **(4a,b)**

General Procedure. Benzothiazol-2-ylacetohydrazide **(1)** (0.01 mol, 2.07 g) was refluxed with an equimolar amount of benzyl or phenyl isothiocyanate in dry dioxane (30 mL) in the presence of triethylamine (3 drops) for 15 minutes. After the solution had been cooled, the solid formed was filtered off and crystallized from dioxane.

1-(Benzothiazol-2-acetyl)-4-benzylthiosemicarbazide **(4a)**

Crystallized from dioxane; mp 203–205°C; yield 85%. Anal. calcd for $C_{17}H_{16}N_4OS_2$ (356.46): C, 57.28; H, 4.53; N, 15.72; S, 17.99. Found: C, 57.06; H, 4.31; N, 15.58; S, 17.66%. IR (cm^{-1}): γ 3240–3150 (NH), 1700 (CO). ^{1}H-NMR (DMSO-d_6): δ 4.1 (s, 2H, CH_2); 4.7 (s, 2H, CH_2Ph); 7.2–7.3 (m, 5H, C_6H_5); 7.4–7.5 (m, 2H, benzothiazole H-5 and H-6); 7.8 (m, 1H, benzothiazole H-4); 8.1 (m, 1H, benzothiazole H-7); 8.6 (brs, 1H, NH); 9.5 (brs, 1H, NH); 10.3 (brs, 1H, NH). MS: *m/z* (M^+ 356, 8%).

1-(Benzothiazol-2-acetyl)-4-phenylthiosemicarbazide **(4b)**

Crystallized from dioxane; mp 216–218°C; yield 80%. Anal. calcd for $C_{16}H_{14}N_4OS_2$ (342.43): C, 56.12; H, 4.12; N, 16.36; S, 18.73. Found: C, 55.20; H, 4.03; N, 16.17; S, 18.47%. IR (cm^{-1}): γ 3250–3150 (NH), 1695 (CO). ^{1}H-NMR (DMSO-d_6): δ 4.2 (s, 2H, CH_2); 7.1–7.5 (m, 7H, C_6H_5, benzothiazole H-5 and H-6); 7.8–8.1 (m, 2H, benzothiazole H-4 and H-7); 9.7–9.8 (brs, 2H, NH, NH); 11.0 (s, 1H, NH).

3-(Benzothiazol-2-methyl)-4-aryl-1,2,4-triazol-5-thiols **(5a,b)**

General Procedure. Each compound **4** (0.01 mol) was refluxed in Na_2CO_3 solution (20%) (30 mL) for 4 hours. The reaction mixture was filtered hot and kept overnight. The solid formed was collected, washed with water, and finally crystallized from dioxane.

3-(Benzothiazol-2-methyl)-4-benzyl-1,2,4-triazol-5-thiol **(5a)**

Crystallized from dioxane; mp 241–243°C; yield 40%. Anal. calcd for $C_{17}H_{14}N_4S_2$ (338.45): C, 60.33; H, 4.17; N, 16.55; S, 18.95. Found: C, 60.14; H, 3.98; N, 16.37; S, 18.80%. IR (cm^{-1}): γ 2950–2700 (SH). ^{1}H-NMR (DMSO-d_6): δ 4.6 (s, 2H, CH_2); 5.3 (s, 2H, CH_2Ph); 7.1–7.2 (m, 5H, C_6H_5); 7.35–7.5 (m, 2H, benzothiazole H-5 and H-6); 7.85 (m, 1H, benzothiazole H-4); 8.1 (m, 1H, benzothiazole H-7). MS: *m/z* (M^+ 338, 100%).

3-(Benzothiazol-2-methyl)-4-phenyl-1,2,4-triazol-5-thiol **(5b)**

Crystallized from dioxane; mp 235–237°C; yield 45%. Anal. calcd for $C_{16}H_{12}N_4S_2$ (324.42): C, 59.24; H, 3.73; N, 17.27; S, 19.77. Found: C, 59.05; H, 3.56; N, 16.89; S, 19.57%. IR (cm^{-1}); γ 2950–2700 (SH). ^{1}H-NMR (DMSO-d_6): δ 4.55 (s, 2H, CH_2); 7.1–7.5 (m, 7H, C_6H_5, benzothiazole H-5 and H-6); 7.9 (m, 1H, benzothiazole H-4); 8.15 (m, 1H, benzothiazole H-7).

5-(Benzothiazol-2-methyl)-2-substituted-1,3,4-thiadiazoles **(6a,b)**

General Procedure. Each compound **4** (0.01 mol) was heated in concentrated H_2SO_4 (3 mL) at 100°C for 10 minutes; the solution was then cooled, and water was added dropwise till precipitation commenced. The solid thus formed was filtered off,

washed with water, and crystallized from the indicated solvent.

2-Amino-5-(benzothiazol-2-methyl)-1,3,4-thiadiazole (6a)

Crystallized from methanol; mp 206–208°C; yield 30%. Anal. calcd for $C_{10}H_8N_4S_2$ (248.32): C, 48.37; H, 3.25; N, 22.56; S, 25.82. Found: C, 48.19; H, 3.02; N, 22.31; S, 25.58%. IR (cm^{-1}): γ 3240–3120 (NH_2). ^{1}H-NMR (DMSO-d_6): δ 4.7 (s, 2H, CH_2); 7.15 (brs, 2H, NH_2); 7.4–7.6 (m, 2H, benzothiazole H-5 and H-6); 7.9 (m, 1H, benzothiazole H-4); 8.1 (m, 1H, benzothiazole H-7). MS: *m/z* (M^+ 248, 35%).

5-(Benzothiazol-2-methyl)-2-phenylamino-1,3,4-thiadiazole (6b)

Crystallized from dioxane; mp 255–257°C; yield 35%. Anal. calcd for $C_{16}H_{12}N_4S_2$ (324.42): C, 59.24; H, 3.73; N, 17.27; S, 19.77. Found: C, 58.90; H, 3.57; N, 17.06; S, 19.62%. IR (cm^{-1}): γ 3250–3150 (NH). ^{1}H-NMR (DMSO-d_6): δ 4.5 (s, 2H, CH_2); 7.2–7.5 (m, 7H, C_6H_5, benzothiazole H-5 and H-6); 7.9 (m, 1H, benzothiazole H-4); 8.1 (m, 1H, benzothiazole H-7); 10.3 (brs, 1H, NH). Mass: *m/z* (M^+ 324, 30%).

N-Arylidenebenzothiazol-2-ylacetohydrazide (7a,b)

General Procedure. Compound **1** (0.01 mol, 2.07 g) was refluxed with the appropriate aldehyde (0.01 mol) in ethanol (30 mL). After half hour reflux, a precipitate was formed. It was filtered off from the hot mixture and crystallized from the indicated solvent.

1-(2-Furylidene)benzothiazol-2-ylacetohydrazide (7a)

Crystallized from methanol; mp 139–141°C; yield 65%. Anal. calcd for $C_{14}H_{11}N_3O_2S$ (285.32): C, 58.94; H, 3.89; N, 14.73; S, 11.24. Found: C, 58.53; H, 3.60; N, 14.38; S, 11.01%. IR (cm^{-1}): γ 3200–3080 (NH), 1670 (CO). ^{1}H-NMR (DMSO-d_6): δ 4.5 (s, 2H, CH_2); 6.6 (dd, 1H, furan H-4); 7.0 (dd, 1H, furan H-3); 7.5 (m, 2H, benzothiazole H-5 and H-6); 7.8–8.2 (m, 4H, furan H-5, ylidene H, benzothiazole H-4 and H-7); 11.4 (brs, 1H, NH). MS: *m/z* (M^+ 285, 25%).

1-(4-Chlorobenzylidene)benzothiazol-2-ylacetohydrazide (7b)

Crystallized from acetic acid; mp 196–198°C; yield 90%. Anal. calcd for $C_{16}H_{12}ClN_3OS$ (329.81): C, 58.27; H, 3.67; Cl, 10.75; N, 12.74; S, 9.72. Found: C, 58.50; H, 3.68; Cl, 10.52; N, 12.37; S, 9.93%. IR (cm^{-1}): γ 3200–3080 (NH), 1670 (CO). ^{1}H-NMR (DMSO-d_6): δ 4.6 (s, 2H, CH_2); 7.4–7.75 (m, 6H, C_6H_4, benzothiazole H-5 and H-6); 7.9–8.1 (m, 2H, benzothiazole H-4 and H-7); 8.3 (s, 1H, ylidene H). MS: *m/z* (M^+ 329, 5%).

5-Amino-3-aryl-1-(benzothiazol-2-acetyl)-4-cyanopyrazoles (8a,b)

General Procedure. Malononitrile (0.01 mL, 0.66 g) was refluxed with each of **(7a,b)** (0.01 mol) in methanol (20 mL) in the presence of triethylamine (1 drop) for 2 hours. A solid product was formed, filtered off from the hot mixture, and crystallized from the indicated solvent.

5-Amino-1-(benzothiazol-2-acetyl)-4-cyano-3-fur-2-yl)pyrazole (8a)

Crystallized from methanol; mp 293–295°C; yield 35%. Anal. calcd for $C_{17}H_{11}N_5O_2S$ (349.37): C, 58.45; H, 3.17; N, 20.05; S, 9.18. Found: C, 58.24; H, 2.94; N, 19.75; S, 9.02%. IR (cm^{-1}): γ 3469–3347 (NH_2), 2180 (CN), 1720 (CO). ^{1}H-NMR (DMSO-d_6): δ 5.7 (s, 2H, CH_2); 6.5–6.7 (m, 2H, furan H-4 and H-3); 7.3–7.45 (m, 2H, benzothiazole H-5 and H-6); 7.6–7.75 (m, 2H, benzothiazole H-4 and H-5); 8.0 (m, 1H, benzothiazole H-7); 8.10 (brs, 2H, NH_2). MS: *m/z* (M^+ 349, 20%).

5-Amino-1-(benzothiazol-2-acetyl)-3-(4-chlorophenyl)-4-cyanopyrazole (8b)

Crystallized from dioxane; mp 308–310°C; yield 30%. Anal. calcd for $C_{19}H_{12}ClN_5OS$ (393.85): C, 57.94; H, 3.07; Cl, 9.00, N, 17.78; S, 8.14. Found: C, 57.08; H, 2.95; Cl, 8.70; N, 17.52; S, 7.83%. IR (cm^{-1}): γ 3345–3200 (NH_2), 2186 (CN), 1700 (CO). ^{1}H-NMR (DMSO-d_6): δ 5.75 (s, 2H, CH_2); 7.2–7.5 (m, 7H, C_6H_4, benzothiazole H-5, H-6 and H-4); 7.9 (m, 1H, benzothiazole H-7); 8.15 (brs, 2H, NH_2).

4-(Benzothiazol-2-yl)-3-(4-chlorophenyl)-4H-pyrazol-5-ol (9a)

Compound **7b** (0.01 mol, 3.29 g) was refluxed in glacial acetic acid (25 mL) for 6 hours; the reaction mixture was then partially concentrated and cooled. The precipitate formed was filtered off and crystallized from AcOH; mp 167–170°C, yield 45%. Anal. calcd for $C_{16}H_{10}ClN_3OS$ (327.79): C, 58.63; H, 3.08; Cl, 10.82; N, 12.82; S, 9.78. Found: C, 58.90; H, 3.34;

Cl, 10.51; N, 12.49; S, 9.57%. IR (cm^{-1}): γ 3450–3380 (OH). ^{1}H-NMR (DMSO-d_6): δ 7.4–7.55 (m, 4H, C_6H_4); 7.65 (s, 1H, pyrazole H-4); 7.7–8.1 (m, 4H, benzothiazole protons).

4-(Benzothiazol-2-yl)-3,4-H-pyrazol-5-ol (9b)

Compound **1** (0.01 mL, 2.07 g) was heated in formamide (20 mL) at 100°C for 6 hours, and the solution was then cooled. The precipitate formed was collected and crystallized from methanol, mp 278–279°C; yield 30%. Anal. calcd for $C_{10}H_7N_3OS$ (217.25): C, 55.29; H, 3.25; N, 19.34; S, 14.76. Found: C, 55.48; H, 3.52; N, 19.08; S, 14.44%. IR (cm^{-1}): γ 3450–3400 (OH). ^{1}H-NMR (DMSO-d_6): δ 6.15 (d, 1H, pyrazole H-3); 6.5 (m, 1H, benzothiazole H-5); 6.9 (d, 1H, pyrazole H-4); 7.1 (m, 1H, benzothiazole H-6); 7.5 (m, 1H, benzothiazole H-4); 8.0 (m, 1H, benzothiazole H-7); 11.0 (s, 1H, OH). MS: *m/z* (M^+ 217, 100%).

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