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Synthesis of New 2-[(Substituted-pyrazolin-1-yl)carbonyl]-thieno[2,3-*b*]pyridine Derivatives

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The reaction of 3-amino-4,6-dimethyl-2-thieno[2,3-*b*]pyridine carbohydrazide (**1**) with appropriate chalcones **2a-2d** in the presence of acid catalyst produced the corresponding 3-amino-2-[(3,5-disubstituted-pyrazolin-1-yl)carbonyl]-4,6-dimethylthieno[2,3-*b*]pyridines **3a-3d**. 3-Amino-2-[(3-substituted-pyrazolin-1-yl)carbonyl]-4,6-dimethylthieno[2,3-*b*]pyridines **7a, 7b** were also obtained by the cyclization reaction of carbohydrazide **1** with Mannich base derivatives **6a, 6b** under basic condition.

INTRODUCTION

Pyrazoline derivatives constitute an interesting class of organic compounds with diverse chemical and pharmacological applications.[1-13] In view of these observations, it was considered of interest to synthesize some new pyrazoline derivatives. Recently, synthesized pyrazolines, as reported by the reaction of hydrazine derivatives with different chalcones, included 1-acetyl-5-aryl-3-(2-furyl)-2-pyrazolines,[14] *N*-phenyl-3-methyl-4-(pyrimido-2-carboxamido)-5-arylpyrazoline,[15] 1-acetyl-3,5-diaryl-2-pyrazolines[16] and 1-acetyl-3-(4′-fluorophenyl)-5-phenyl-4*H*-4,5-pyrazoline.[17] In the preceding papers[27,28] we have described the synthesis of 3-amino-4,6-dimethyl-2-thieno[2,3-*b*]pyridine carbohydrazide (**1**) by refluxing ethyl 3-amino-4,6-dimethyl-2-thieno[2,3-*b*]pyridine carboxylate with 85% excess of hydrazine hydrate in good yield (97%). In continuation of our studies, we synthesized some new 2-[(substituted-pyrazolin-1-yl)carbonyl]-thieno[2,3-*b*]pyridine derivatives from carbohydrazide **1**.

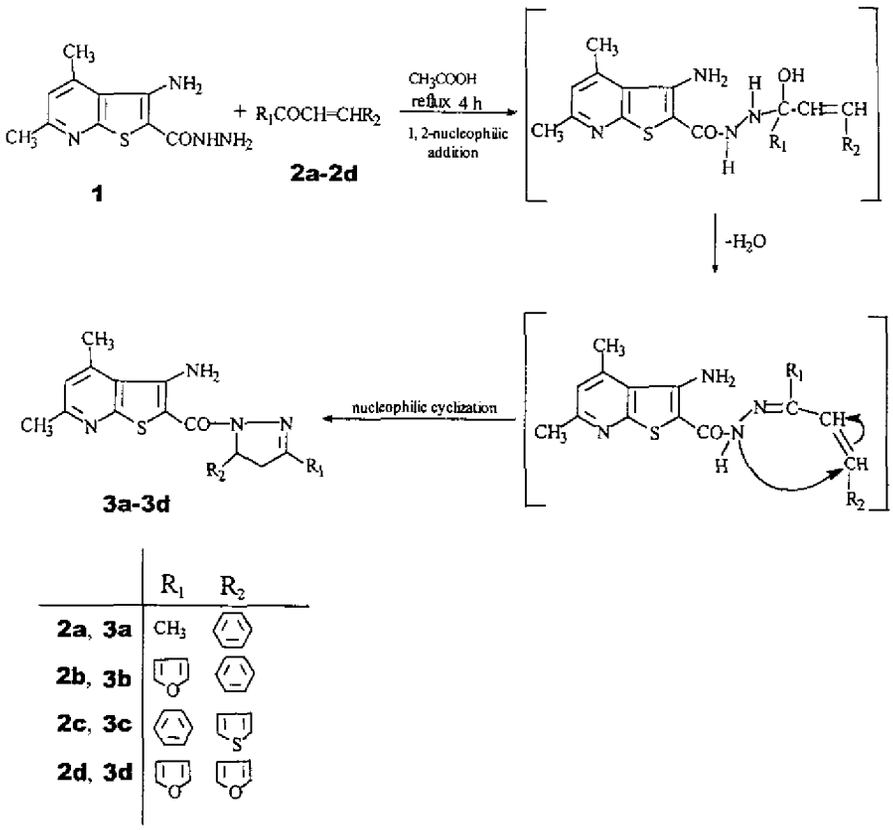
RESULTS AND DISCUSSION

All relevant reactions are depicted in Schemes I-III. The reaction of 3-amino-4,6-dimethyl-2-thieno[2,3-*b*]pyridine carbohydrazide (**1**) with one equivalent of 1-methyl-3-phenyl-2-propen-1-one (**2a**) in refluxing glacial acetic acid for 4 h produced desired product, 3-amino-2-[(3-methyl-5-phenylpyrazolin-1-yl)carbonyl]-4,6-dimethylthieno[2,3-*b*]pyridine (**3a**), in 52% yield (Scheme I). Under similar reaction condition, treatment of carbohydrazide **1** with chalcones **2b-2d** such as 1-(2-furyl)-3-phenyl-2-propen-1-one (**2b**), 1-phenyl-3-(2-thienyl)-2-propen-1-one (**2c**) and 1,3-bis(2-furyl)-2-propen-1-one (**2d**) produced the corresponding 3-amino-2-[(3,5-disubstituted-pyrazolin-1-yl)carbonyl]-4,6-dimethylthieno[2,3-*b*]pyridines **3b-3d**, respectively (Scheme I). The chalcones **2a-2d** were obtained *via* the Claisen-Schmidt condensation.[18-20] Accordingly, condensation of aromatic aldehyde with 2-acetylfuran or 2-acetylthiophene in alcoholic sodium hydroxide solution gave the chalcones **2a-2d**, respectively.[14,26]

The structures of **3a-3d** were established by examining spectral data and elemental analysis. The IR spectra of compounds **3a-3d** are characterized by the strong absorption band of the NH_2 group at 3449-3316 cm^{-1} and the characteristic absorption band of the carbonyl group (C=O) at 1689-1612 cm^{-1}. The ^1H NMR spectra (DMSO-d_6 or CF_3COOD) of compounds **3a-3d** revealed a doublet at δ 7.42-6.57 (2H, d) assigned for the 4-H of the pyrazoline ring. Compounds **3a-3c** showed a downfield multiplet at δ 7.78-7.20 (6H, m) assigned for the 3-phenyl and 5-position protons of the pyrazoline ring. Compound **3d** showed a downfield multiplet at δ 8.03-6.73 (6H, m) assigned for two furyl protons of the pyrazoline ring.

Scheme II depicts the treatment of carbohydrazide **1** with cinnamaldehyde (**2e**) under similar reaction conditions. The major product (75% yield) was 3-amino-2-carbocinnamylidene-hydrazido-4,6-dimethylthieno[2,3-*b*]pyridine (**3e**), instead of the expected 3-amino-2-[(5-phenylpyrazolin-1-yl)carbonyl]-4,6-dimethylthieno[2,3-*b*]pyridine (**4**). The IR spectra of compound **3e** showed strong absorption bands at 3492, 3252 cm^{-1} for the NH_2 and NH group, at 1708 cm^{-1} for the carbonyl group (C=O), and at 972 cm^{-1} for the out of plane vibration from *trans*-olefinic protons (CH=CH). In addition, the structure was supported by ^1H

Scheme I



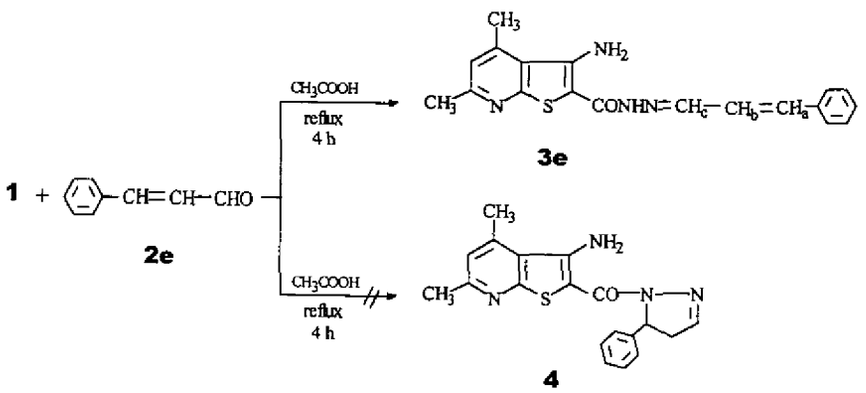
	R_1	R_2
2a, 3a	CH_3	
2b, 3b		
2c, 3c		
2d, 3d		

NMR (DMSO-d_6) spectrum, which showed a broad singlet at δ 11.29 (1H, br) assigned to the CONH proton. The spectrum also revealed a multiplet at δ 7.34-7.29 (2H, m) and a doublet at δ 7.91 (1H, d), which were assigned to the protons of $=CH_c-CH_b=$ and CH_a of cinnamylidene moiety, respectively.

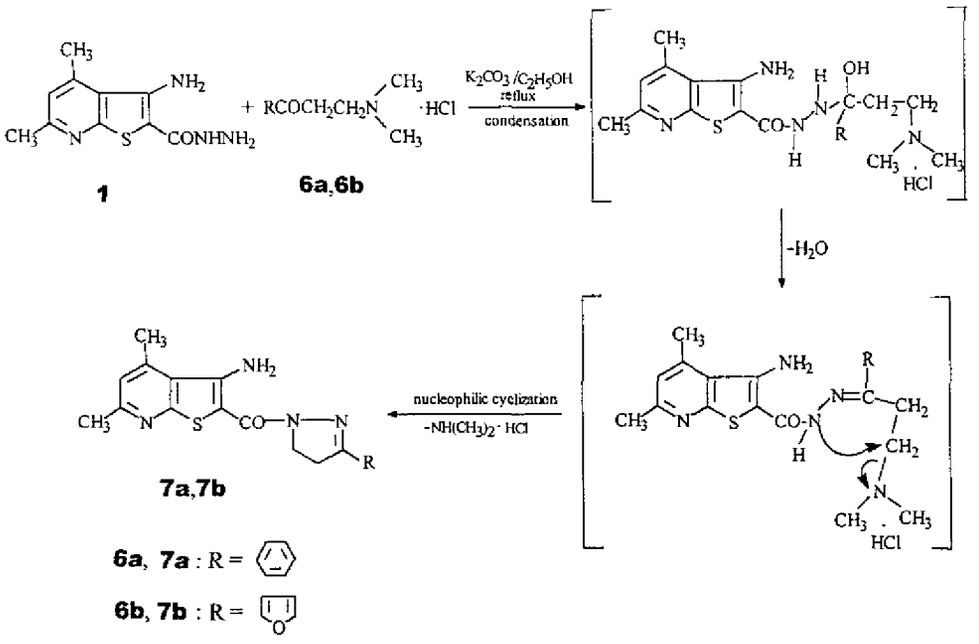
The formation of 3-amino-2-[(3,5-disubstituted-pyrazolin-1-yl)carbonyl]-4,6-dimethylthieno[2,3-*b*]pyridines **3a-3d** can be explained by the reaction pathway depicted in Scheme I. The first step of the mechanism involves 1,2-nucleophilic addition of hydrazide to the carbonyl group followed by dehydration and subsequent nucleophilic cyclization.[14,21]

The synthesis of 3-aminothieno[2,3-*b*]pyridines having 3-substituted-pyrazoline rings is outlined in Scheme III. The Mannich bases such as *N,N*-dimethyl-2-benzoylethy-

Scheme II



Scheme III



lamine hydrochloride (**6a**) and *N,N*-dimethyl-2-furoylethylamine hydrochloride (**6b**), were obtained *via* Mannich reaction.[24,25]

Condensation of the carbohydrazide **1** with Mannich bases **6a** and **6b** in refluxing ethanol for 8 h in the presence of excess anhydrous potassium carbonate afforded the corresponding 3-amino-2-[(3-substituted-pyrazolin-1-yl)carbonyl]-4,6-dimethylthieno[2,3-*b*]pyridines **7a**, **7b** (Scheme III). The first step of the mechanism involves the condensation of hydrazide with the carbonyl group, followed by dehydration, subsequent nucleophilic cyclization, with the loss of *N,N*-dimethylamine hydrochloride. The structures of compounds **7a**, and **7b** were established and confirmed on the basis of their elemental analysis and spectral data. ^{1}H NMR spectra revealed triplets at δ 3.69 (2H, t) and δ 4.46 (2H, t) in compound **7a**, and at δ 3.20 (2H, t) and δ 4.16 (2H, t) in compound **7b**. These triplets were assigned to be from methylene protons at the 4-, 5-position of the pyrazoline ring. Compound **7a** showed a multiplet at δ 7.67-7.60 (5H, m) from phenyl protons of the pyrazoline ring. Compound **7b** showed signals at δ 6.51 (1H, dd), 6.90 (1H, d) and 7.53 (1H, d), which were assigned to be from protons of furyl moiety of the pyrazoline ring.

In conclusion, carbohydrazide **1** shown in this paper is found to be useful reagent for the synthesis of some new 2-[(substituted-pyrazolin-1-yl)carbonyl]-thieno[2,3-*b*]pyridine derivatives.

EXPERIMENTAL SECTION

All melting points were determined in a capillary tube and are uncorrected. The IR spectra were recorded on potassium bromide pellets on a JASCO FTIR-3 spectrophotometer. The ^{1}H NMR spectra were obtained on a Bruker AM-300WB FT-NMR spectrometer, and chemical shifts are expressed in δ ppm using TMS as an internal standard. Electron impact Mass spectra were obtained at 70 eV using a Finingan Mat TSQ-46C spectrometer. All compounds were dried *in vacuo* at 60 °C for 10 h before elemental analyses. The elemental analyses for C, H, and N were performed on a Perkin-Elmer 240 elemental analyzer.

3-Amino-2-[(3-methyl-5-phenylpyrazolin-1-yl)carbonyl]-4,6-dimethylthieno[2,3-*b*]pyridine (3a)

A solution of carbohydrazide **1** (0.50 g, 2.1 mmol) and 1-methyl-3-phenyl-2-propen-1-one (**2a**) (0.31 g, 2.1 mmol) was refluxed in glacial acetic acid (10 mL) for 4 h, and then allowed to stand overnight. The resulting solid product was collected by filtration and recrystallized from glacial acetic acid/dioxane to give 0.4 g of orange needles (52% yield), mp 267-268 °C; IR: ν 3437, 3298 (NH_2), 1612 (C=O) cm^{-1}; ^{1}H NMR (DMSO-d_6): δ 2.14 (3H, s, 3-CH_3 of pyrazoline), 2.49 (3H, s, 4-CH_3), 2.74 (3H, s, 6-CH_3), 6.95 (2H, d, J = 5.0 Hz, 4-H of pyrazoline), 7.08 (1H, s, 5-H), 7.61-7.32 (6H, m, phenyl-H and 5-H of pyrazoline); MS: 364 (M^+).

Anal. Calcd. for $C_{20}H_{20}N_4OS$: C, 65.91; H, 5.53; N, 15.37. Found: C, 65.77; H, 5.37; N, 15.35.

3-Amino-2-[(3-furyl-5-phenylpyrazolin-1-yl)carbonyl]-4,6-dimethylthieno[2,3-*b*]pyridine (3b)

To a mixture of carbohydrazide **1** (0.50 g, 2.1 mmol) and 1-(2-furyl)-3-phenyl-2-propen-1-one (**2b**) (0.42 g, 2.1 mmol) in dioxane (5 mL), a few drops of trifluoroacetic acid were added. The mixture was heated under reflux for 6 h, and then allowed to stand overnight. The resulting solid product was collected by filtration and recrystallized from glacial acetic acid/dioxane to give 0.51 g of orange needles (53% yield), mp 168-170 °C; IR: ν 3439, 3215 (NH_2), 1689 (C=O) cm^{-1}; ^1H NMR (DMSO-d_6): δ 2.52 (3H, s, 4-CH_3), 2.76 (3H, s, 6-CH_3), 6.95 (2H, d, J = 1.2 Hz, 4-H of pyrazoline), 7.04 (1H, s, 5-H), 7.24 (1H, t, J = 3.0 Hz, 5-H of pyrazoline), 7.78-7.70 (5H, m, phenyl-H), 7.57 (1H, dd, J = 5.0, 5.0 Hz, 4-H of furyl), 7.67 (1H, d, J = 3.0 Hz, 3-H of furyl), 7.99 (1H, d, J = 6.9 Hz, 5-H of furyl); MS: 416 (M^+).

Anal. Calcd. for $C_{23}H_{20}N_4O_2S$: C, 66.33; H, 4.84; N, 13.45. Found: C, 66.21; H, 4.75; N, 13.66.

3-Amino-2-[(3-phenyl-5-thienylpyrazolin-1-yl)carbonyl]-4,6-dimethylthieno[2,3-*b*]pyridine (3c)

This compound was synthesized from carbohydrazide **1** (0.50 g, 2.1 mmol) and 1-phenyl-3-(2-thienyl)-2-propen-1-one (**2c**) (0.45 g, 2.1 mmol) in a manner similar to that described for the preparation of **3a**. It was recrystallized from glacial acetic acid/dioxane to give 0.48 g of reddish orange needles (53% yield), mp 205 °C; IR: ν 3446, 3316 (NH_2), 1640 (C=O) cm^{-1}; ^1H NMR (CF_3COOD): δ 2.94 (3H, s, 4-CH_3), 3.12 (3H, s, 6-CH_3), 7.35 (2H, d, J = 5.0 Hz, 4-H of pyrazoline), 7.55 (1H, s, 5-H), 7.64 (1H, t, J = 8.1 Hz, 5-H of pyrazoline), 7.77-7.71 (5H, m, phenyl-H), 7.90 (1H, dd, J = 1.4, 1.0 Hz, 4-H of thienyl), 8.05-7.33 (2H, m, 3-H, 5-H of thienyl); MS: 432 (M^+).

Anal. Calcd. for $C_{23}H_{20}N_4OS_2$: C, 63.86; H, 4.66; N, 12.95. Found: C, 63.76; H, 4.68; N, 12.97.

3-Amino-2-[(3,5-difurylpyrazolin-1-yl)carbonyl]-4,6-dimethylthieno[2,3-*b*]pyridine (3d)

This compound was synthesized from carbohydrazide **1** (0.50 g, 2.1 mmol) and 1,3 di(2-furyl)-2-propen-1-one (**2d**) (0.40 g, 2.1 mmol) in a manner similar to that described for the preparation of **3a**. It was recrystallized from glacial acetic acid/dioxane to give 0.61 g of orange needles (72% yield), mp 197-198 °C; IR: ν 3449, 3316 (NH_2), 1689 (C=O) cm^{-1}; ^1H NMR (DMSO-d_6): δ 2.48 (3H, s, 4-CH_3), 2.72 (3H, s, 6-CH_3), 6.62 (2H, d, J = 11.0 Hz, 4-H of pyrazoline), 7.00 (1H, s, 5-H), 7.14 (1H, t, J = 10.8 Hz, 5-H of pyrazoline), 8.03-6.73 (6H, m, furyl-H); MS: 406 (M^+).

Anal. Calcd. for $C_{21}H_{18}N_4O_3S$: C, 62.06; H, 4.46; N, 13.78. Found: C, 62.11; H, 4.24; N, 13.59.

3-Amino-2-carbocinnamylidenehydrazido-4,6-dimethylthieno[2,3-*b*]pyridine (3e)

This compound was synthesized from carbohydrazide **1** (0.50 g, 2.1 mmol) and cinnamaldehyde (**2e**) (0.28 g, 2.1 mmol) in a manner similar to that described for the preparation of **3a**. It was recrystallized from dioxane to give 0.55 g of orange needles (75% yield), mp 252-254 °C; IR: ν 3492, 3252 (NH_2), 1708 (C=O), 972 (CH=CH) cm^{-1}; ^1H NMR (DMSO-d_6): δ 2.53 (3H, s, 4-CH_3), 2.74 (3H, s, 6-CH_3), 7.00 (2H, br, NH_2), 7.01 (1H, s, 5-H), 7.34-7.29 (2H, m, CH_c-CH_b), 7.64-7.40 (5H, m, phenyl-H), 7.91 (1H, d, J = 2.5 Hz, CH_a), 11.29 (1H, br, CONH); MS: 350 (M^+).

Anal. Calcd. for $C_{19}H_{18}N_4OS$: C, 65.12; H, 5.18; N, 15.99. Found: C, 65.30; H, 5.22; N, 16.01.

***N,N*-Dimethyl-2-benzoylethylamine Hydrochloride (6a)**

A mixture of acetophenone (**5a**) (6.00 g, 0.05 mol), dimethylamine hydrochloride (5.30 g, 0.065 mol), and paraformaldehyde (0.022 mol) was refluxed for 2 h in 95% ethanol (8 mL) in the prescene of hydrochloric acid (1 mL). The reaction mixture was added acetone (40 mL), allowed to cool slowly to room temperature, and then chilled overnight in the refrigerator. The crystals were filtered and washed with acetone. It was recrystallized from 95% ethanol to yield 7.3 g of coloress needles (68% yield of **6a**), mp 156-157 °C; IR: ν 1766 (C=O) cm^{-1}; ^1H NMR (DMSO-d_6): δ 2.81 (6H, s, $N(CH_3)_2$), 3.45 (2H, t, J = 5.1 Hz, CH_2), 3.63 (2H, t, J = 5.1 Hz, CH_2), 7.92-7.73 (5H, m, phenyl-H); MS: 213.7 (M^+).

Anal. Calcd. for $C_{11}H_{16}NClO$: C, 61.82; H, 7.55; N, 6.55. Found: C, 61.78; H, 7.54; N, 6.50.

***N,N*-Dimethyl-2-furoylethylamine Hydrochloride (6b)**

This compound was synthesized from 2-acetylfuran (**5b**) (5.50 g, 0.05 mol), dimethylamine hydrochloride (5.30 g, 0.065 mol) and paraformaldehyde (0.022 mol) in a manner similar to that described for the preparation of **6a**. It was recrystallized from 95% ethanol to give 6.11 g of colorless needles (60% yield of **6b**), mp 180-181 °C; IR: ν 1756 (C=O) cm^{-1}; ^1H NMR (DMSO-d_6): δ 2.75 (6H, s, $N(CH_3)_2$),

3.35-3.29 (4H, m, CH_2CH_2), 6.64 (1H, dd, J = 5.1, 5.1 Hz, 4-H of furyl), 7.44 (1H, d, J = 1.1 Hz, 3-H of furyl), 7.82 (1H, d, J = 1.2 Hz, 5-H of furyl); MS: 203.7 (M^+).

Anal. Calcd. for $C_9H_{14}NClO_2$: C, 53.08; H, 6.93; N, 6.88. Found: C, 53.01; H, 7.01; N, 6.93.

3-Amino-2-[(3-phenylpyrazolin-1-yl)carbonyl]-4,6-dimethylthieno[2,3-*b*]pyridine (7a)

A mixture of carbohydrazide 1 (0.50 g, 2.1 mmol), *N,N*-dimethyl-2-benzoylethylamine hydrochloride (**6a**) (0.45 g, 2.1 mmol) and anhydrous potassium carbonate (0.44 g, 3.5 mmol) was refluxed in absolute ethanol (10 mL) for 8 h. The reaction mixture was cooled. The resulting solid product was collected by filtration and recrystallized from dioxane to give 0.35 g of pale yellow needles (46% yield of **7a**) mp 174-176 °C; IR: ν 3477, 3318 (NH_2), 1600 (C=O) cm^{-1}; ^1H NMR (CF_3COOD): δ 2.98 (3H, s, 4-CH_3), 3.17 (3H, s, 6-CH_3), 3.69 (2H, t, J = 3.4 Hz, 4-H of pyrazoline), 4.46 (2H, t, J = 3.1 Hz, 5-H of pyrazoline), 7.34 (1H, s, 5-H), 7.67-7.60 (5H, m, phenyl-H); MS: 350 (M^+).

Anal. Calcd. for $C_{19}H_{18}N_4OS$: C, 65.12; H, 5.18; N, 15.99. Found: C, 65.23; H, 5.21; N, 15.95.

3-Amino-2-[(3-furylpyrazolin-1-yl)carbonyl]-4,6-dimethylthieno[2,3-*b*]pyridine (7b)

This compound was synthesized from carbohydrazide 1 (0.50 g, 2.1 mmol) and *N,N*-dimethyl-2-furoylethylamine hydrochloride (**6b**) (0.43 g, 2.1 mmol) in a manner similar to that described for the preparation of **7a**. It was recrystallized from dioxane to give 0.42 g of yellow product (59% yield of **7b**), mp 300 °C; IR: ν 3477, 3313 (NH_2), 1600 (C=O) cm^{-1}; ^1H NMR ($CDCl_3$): δ 2.58 (3H, s, 4-CH_3), 2.75 (3H, s, 6-CH_3), 3.20 (2H, t, J = 6.8 Hz, 4-H of pyrazoline), 4.16 (2H, t, J = 7.0 Hz, 5-H of pyrazoline), 6.51 (1H, dd, J = 1.0, 1.0 Hz, 4-H of furyl), 6.75 (2H, br, NH_2), 6.81 (1H, s, 5-H), 6.90 (1H, d, J = 1.2 Hz, 3-H of furyl), 7.53 (1H, d, J = 1.2 Hz, 5-H of furyl); MS: 340 (M^+).

Anal. Calcd. for $C_{17}H_{16}N_4O_2S$: C, 59.98; H, 4.74; N, 16.46. Found: C, 59.93; H, 4.65; N, 16.58.

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Key Words

Pyrazolines; 3-Amino-2-thieno[2,3-*b*]pyridine carbohydrazide.

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