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Microwave-assisted synthesis of novel 8-chloro-[1,2,4]triazolo[4,3-a]pyridine derivatives

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Abstract: A series of novel 1,2,4-triazolo[4,3-a]pyridine derivatives were synthesized from 2,3-dichloropyridine and hydrazine hydrate as starting materials by multistep reactions under microwave assistance, and their structures were characterized by ^{1}H NMR, MS, and elemental analysis. This method provides several advantages such as high yields, facile work-up, and environmental friendliness.

Key words: 1,2,4-Triazolo[4,3-a]pyridine, urea, microwave-assisted synthesis

1. Introduction

At present, nitrogen-containing compounds are a hot topic due to their diverse function in the field of organic synthesis, medicinal chemistry, pesticide chemistry, and industrial chemistry.[1−3] 1,2,4-Triazole and pyridine derivatives often displayed broad and excellent activities.[4,5] On the other hand, fused heterocycles generally exhibit properties of the single heterocyclic. The incorporation of a pyridine ring into a triazole ring was proved to be a good way to produce novel active compounds.[6] Furthermore, various derivatives of ureas including simple ureas,[7] arylureas,[8] aminourea,[9] and thioureas[10] are considered privileged scaffolds in drug discovery with a wide array of biological activities.[11−13] In the literature, aminourea derivatives have been utilized for their herbicidal,[14] antibacterial,[15] and insecticidal activity.[16]

Generally, green chemistry has been attracting great interest from chemists because of its environmental benefits.[17] Many green methods, such as catalyst-free,[18] supercritical fluids,[19] ionic liquids,[20] solvent-free reactions,[21] and ultrasound[22] or microwave[23] irradiation as energy sources were applied in organic synthesis. Recently, microwave irradiation has become an effective tool in organic synthesis, because of its short reaction time and higher yields.

In view of all these facts and in continuation of our research on bioactive compounds,[24−26] a series of novel urea derivatives containing 1,2,4-triazolo[4,3-a]pyridine moiety were synthesized under microwave irradiation.

2. Results and discussion

2.1. Synthesis

Microwave technology was applied to the synthetic reaction to shorten the reaction time and increase the yields of urea derivatives **4**. The one pot synthesis of intermediate **1** under microwave irradiation was conducted, but

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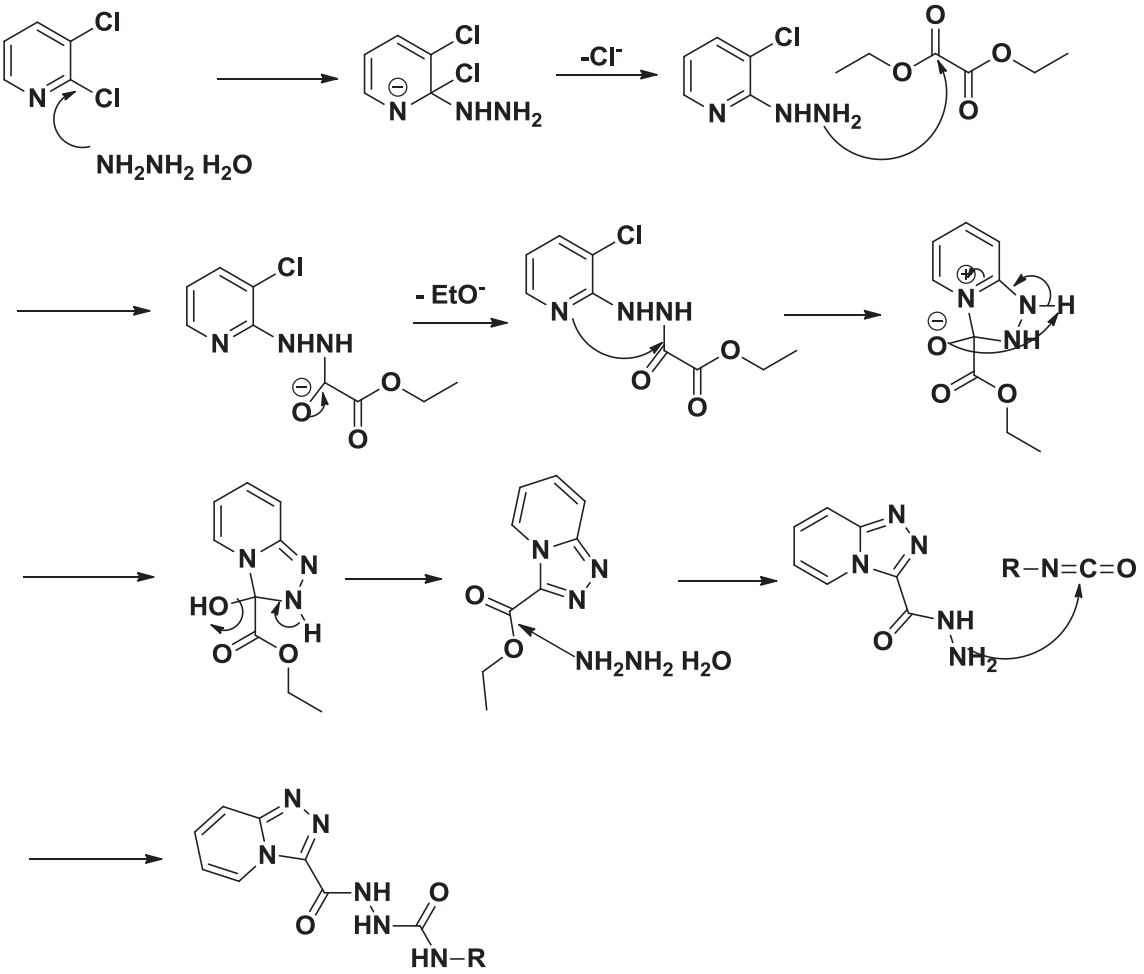
the result was not better than that of conventional conditions. Intermediate **1** and diethyl oxalate led to the intermediate **2** by nucleophile substitution reaction. All the reaction mechanisms are nucleophile substitution reactions (Scheme 1). The mixture of acylhydrazine and RNCO in different solvents was irradiated with microwaves. This reaction was completed with higher yields compared with the conventional mode of heating. The reaction parameters were optimized for the synthesis of the title compounds. Compound **4b** was chosen as a model reaction under different conditions. Several key reaction conditions were investigated including the reaction times, reaction temperatures with or without microwave irradiation, and the reaction solvent. Table 1 indicates that the microwave irradiation can accelerate the rate of the reaction. The effect of three different solvents was studied. The best solvent of this reaction is CH_3CN; the yield of **4b** was significantly higher than that of other solvents. The reaction temperature, reaction time, and reaction molar ratio were also studied. When they were reacted at 90 °C under a stoichiometric ratio of 1:1.3 for 1 min under microwave irradiation, the yield of compound **4b** was higher (Table 2).



Scheme 1. The reaction mechanism of the title compounds.

2.2. Spectrum

In the title compounds, there are three NH groups (Figure). The signals of NH protons were observed at around δ8.00∼10.41 ppm. The order of chemical shift of NH protons is δ(Hc)(10.10∼10.41) > δ(Ha) (8.15∼9.24)

$> \delta$(Hb) (8.00∼9.20). Of these, the chemical shift of Ha and Hb is difficult to distinguish, due to the hydrogen bonds that are formed between the two O atoms and NH (Figure). The ESI-MS spectrum showed that the m/z of molecular ion is in agreement with its molecular formula. The measured elemental analyses were also consistent with the corresponding calculated values.

Table 1. Comparison of yields of **4b** through methods with or without microwave irradiation.

Entry	Solvent	Method	Time	Temperature/°C	Yield/%
1	CH_3CN	No-MW	3 h	r.t.	90
2	CH_3CN	MW	1 min	90	91
3	CH_2Cl_2	MW	1 min	50	60
4	Toluene	MW	1 min	115	65
5	CH_3CN	MW	1 min	80	75
6	CH_3CN	MW	1 min	85	81
7	CH_3CN	MW	1 min	90	91
8	CH_3CN	MW	2 min	90	89

Table 2. Comparison of yields of **4b** in different stoichiometric ratio under microwave irradiation.

Entry	Solvent	Method	Time	Temperature/°C	Stoichiometric ratio	Yield/%
1	CH_3CN	MW	1 min	90	1:1.1	72
2	CH_3CN	MW	1 min	90	1:1.2	78
3	CH_3CN	MW	1 min	90	1:1.3	84
4	CH_3CN	MW	1 min	90	1:1.4	83

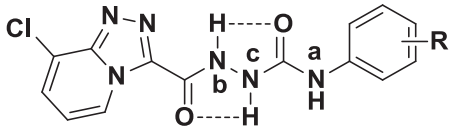


Figure. Plausible hydrogen bonding in 8-chloro-[1,2,4]triazolo[4,3-*a*]pyridine derivatives.

3. Experimental

3.1. Materials and methods

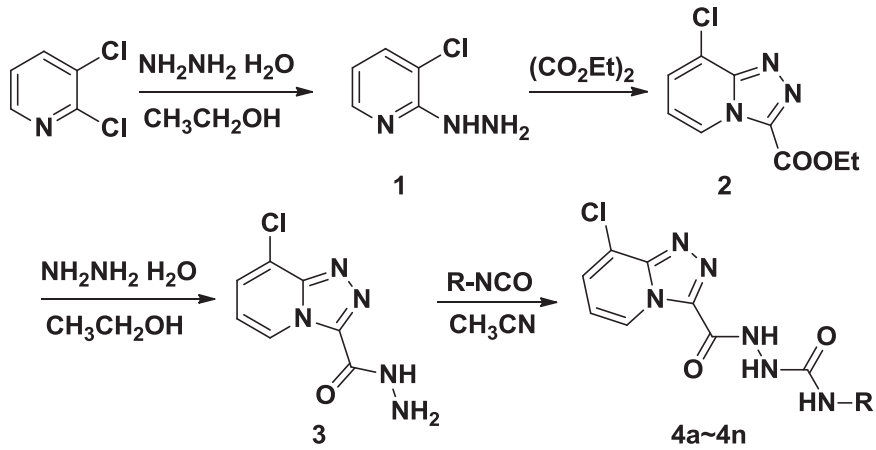
All reagents were analytical grade. Melting points were determined using an X-4 apparatus and were uncorrected. ^{1}H NMR spectra were measured on a Bruker Avance 400 MHz spectrometer using TMS as an internal standard and DMSO-d_6 as solvent. A CEM Discover Focused Synthesizer was used to carry out the microwave reaction. Elemental analysis was performed with a PerkinElmer 240C analyzer.

3.2. Synthesis

3.2.1. General procedure

2,3-Dichloropyridine (7.50 mmol) was dissolved in ethanol (300 mL); then hydrazine hydrate (30 mmol) was added dropwise under refluxing over 72 h to give 3-chloro-2-hydrazinylpyridine **1**. A CEM designed 10-mL pressure-rated vial was charged with 3-chloro-2-hydrazinylpyridine **1** (143 mg, 1 mmol) and diethyloxalate (1 mmol). The mixture was irradiated in a CEM Discover Focused Synthesizer (150 w, 140 °C, 200 psi, 15 min). The mixture was cooled to room temperature by passing compressed air through the microwave cavity for 2 min. It was poured into cold ice (40 mL) and the formed precipitate filtered. The crude solid was recrystallized

from ethanol to give the title compound **2**. Then compound **2** (20 mmol) was reacted with hydrazine hydrate (30 mmol) under microwave irradiation (150 W, 100 °C, 200 psi, 10 min), to afford compound **3**. Last, the title compound **4** was synthesized from compound **3** and isocyanate under microwave conditions. All the other compounds are synthesized according to the procedure (Scheme 2).



Scheme 2. **4a**: R = phenyl; **4b**: R = 2,5-dimethylphenyl; **4c**: R = 2-methoxyphenyl; **4d**: R = 3-chloro-2-methylphenyl; **4e**: R = 3-phenoxyphenyl; **4f**: R = m-tolyl; **4g**: R = 2,6-dichloro-4-(trifluoromethyl)phenyl; **4h**: R = 3,5-dichlorophenyl; **4i**: R = 2,3-dimethylphenyl; **4j**: R = 2-methyl-4-nitrophenyl; **4k**: R = 2-methyl-3-(trifluoromethyl)phenyl; **4l**: R = 2-(trifluoromethoxy)phenyl; **4m**: R = naphthalen-1-yl; **4n**: R = 2-chloro-5-(trifluoromethyl)phenyl.

1-(8-chloro-[1,2,4]triazolo[4,3-*a*]pyridine-3-carbonyl)-4-phenylsemicarbazide 4a: white crystal, yield 50.51%, mp > 300 °C; ^{1}H NMR (DMSO-d_6, 400 MHz), δ: 6.97 (d, J = 7.6 Hz, 1H, Ph-H), 7.26 (m, 5H, 1Py-H, 4Ph-H), 7.90 (d, J = 7.6 Hz, 1H, Py-H), 8.43 (s, 1H, NH), 8.80 (s, 1H, NH), 9.14 (d, J = 7.2 Hz, 1H, Py-H), 10.99 (s, 1H, NH). MS (ESI), m/z: 331 $(M+1)^+$. Elemental anal. (%), calculated: C, 50.84; H, 3.35; N, 25.41; found: C, 50.99; H, 3.44; N, 25.34.

1-(8-chloro-[1,2,4]triazolo[4,3-*a*]pyridine-3-carbonyl)-4-(2,5-dimethylphenyl)semicarbazide 4b: light yellow crystal, yield 78.21%, mp > 300 °C; ^{1}H NMR (DMSO-d_6, 400 MHz), δ: 2.18 (s, 3H, –CH_3), 2.24 (s, 3H, –CH_3), 6.80 (d, J = 7.6 Hz, 1H, Ph-H), 7.05 (d, J = 7.6 Hz, 1H, Ph-H), 7.25 (t, J = 7.2 Hz, 1H, Py-H), 7.50 (s, 1H, Ph-H), 7.83 (d, J = 7.6 Hz, 1H, Py-H), 8.06 (s, 1H, NH), 8.57 (s, 1H, NH), 9.14 (d, J = 7.2 Hz, 1H, Py-H), 11.02 (s, 1H, NH). MS (ESI), m/z: 359 $(M+1)^+$. Elemental anal. (%), calculated: C, 53.56; H, 4.21; N, 23.42; found: C, 53.65; H, 4.53; N, 23.65.

1-(8-chloro-[1,2,4]triazolo[4,3-*a*]pyridine-3-carbonyl)-4-(2-methoxyphenyl)semicarbazide 4c: white crystal, yield 59.25%, mp > 300 °C; ^{1}H NMR (DMSO-d_6, 400 MHz), δ: 3.87 (s, 3H, –CH_3), 6.85–7.26 (m, 4H, 1Py-H, 3Ph-H), 7.82–8.26 (m, 3H, 1Py-H, 1Ph-H, 1NH), 8.90–9.16 (m, 2H, 1NH, 1Py-H), 11.06 (s, 1H, NH). ^{13}C NMR (DMSO-d_6, 100 MHz) δ: 56.25, 111.27, 116.49, 118.81, 120.80, 121.01, 122.54, 125.60, 128.82, 129.26, 139.99, 148.19, 149.03, 155.17, 157.86. MS (ESI), m/z: 361 $(M+1)^+$. Elemental anal. (%), calculated: C, 49.94; H, 3.63; N, 23.30; found: C, 50.21; H, 3.76; N, 23.45.

4-(3-chloro-2-methylphenyl)-1-(8-chloro-[1,2,4]triazolo[4,3-*a*]pyridine-3-carbonyl)semicarbazide 4d: light yellow crystal, yield 75.83%, mp > 300 °C; ^{1}H NMR (DMSO-d_6, 400 MHz), δ: 2.27 (s, 3H, –CH_3), 7.17–7.26 (m, 3H, 1Py-H, 2Ph-H), 7.83 (d, J = 7.0 Hz, 1H, 1Py-H), 8.43 (s, 1H, NH), 8.66 (s, 1H,

NH), 9.14 (d, J = 6.8 Hz, 1H, 1Py-H), 11.08 (s, 1H, NH). MS (ESI), m/z: 379 $(M+1)^+$. Elemental anal. (%), calculated: C, 47.51; H, 3.19; N, 22.16; found: C, 47.55; H, 3.23; N, 22.32.

1-(8-chloro-[1,2,4]triazolo[4,3-*a*]pyridine-3-carbonyl)-4-(3-phenoxyphenyl)semicarbazide 4e: white crystal, yield 83.72%, mp 268–272 °C; ^{1}H NMR (DMSO-d_6, 400 MHz), δ: 6.96 (t, J = 6.8 Hz, 4H, Ph-H), 7.09 (t, J = 6.8 Hz, 1H, Ph-H), 7.25 (t, J = 7.2 Hz, 1H, Py-H), 7.36 (t, J = 8.0 Hz, 2H, Ph-H), 7.51 (d, J = 8.8 Hz, 2H, Ph-H), 7.84 (d, J = 7.2 Hz, 1H, Py-H), 8.45 (s, 1H, NH), 8.87 (s, 1H, NH), 9.14 (d, J = 6.8 Hz, 1H, Py-H), 11.01 (s, 1H, NH). MS (ESI), m/z: 423 $(M+1)^+$. Elemental anal. (%), calculated: C, 56.81; H, 3.58; N, 19.88; found: C, 56.75; H, 3.45; N, 20.01.

1-(8-chloro-[1,2,4]triazolo[4,3-*a*]pyridine-3-carbonyl)-4-m-tolylsemicarbazide 4f: white crystal, yield 84.00%, mp > 300 °C; ^{1}H NMR (DMSO-d_6, 400 MHz), δ: 2.25 (s, 3H, CH_3), 6.78 (d, J = 7.3 Hz, 1H, Ph-H), 7.13 (t, J = 8.5 Hz, 1H, Ph-H), 7.22–7.31 (m, 3H, 1Py-H, 2Ph-H), 7.83 (d, J = 7.3 Hz, 1H, Py-H), 8.40 (s, 1H, NH), 8.75 (s, 1H, NH), 9.13 (d, J = 6.8 Hz, 1H, Py-H), 10.99 (s, 1H, NH). MS (ESI), m/z: 345 $(M+1)^+$. Elemental anal. (%), calculated: C, 52.26; H, 3.80; N, 24.38; found: C, 52.34; H, 3.99; N, 24.45.

1-(8-chloro-[1,2,4]triazolo[4,3-*a*]pyridine-3-carbonyl)-4-(2,6-dichloro-4-(trifluoromethyl) phenyl)semicarbazide 4g: white crystal, yield 91.5%, mp > 300 °C; ^{1}H NMR (DMSO-d_6, 400 MHz), δ: 7.19 (t, J = 7.0 Hz, 1H, Py-H), 7.77 (m, 2H, 1Py-H, 1Ph-H), 8.01 (s, 2H, 1Ph-H, 1NH), 8.89 (s, 1H, NH), 9.15 (d, J = 7.0 Hz, 1H, Py-H), 10.56 (s, 1H, NH). MS (ESI), m/z: 489 $(M+Na)^-$. Elemental anal. (%), calculated: C, 38.53; H, 1.72; N, 17.97; found: C, 38.73; H, 1.97; N, 18.21.

1-(8-chloro-[1,2,4]triazolo[4,3-*a*]pyridine-3-carbonyl)-4-(3,5-dichlorophenyl)semicarbazide 4h: yellow green crystal, yield 72.03%, mp 260–265 °C; ^{1}H NMR (DMSO-d_6, 400 MHz), δ: 7.25 (t, J = 7.2 Hz, 1H, Py-H), 7.6 (m, 1H, Ph-H), 7.77–7.88 (m, 3H, 1Py-H, 2Ph-H), 8.56 (s, 1H, NH), 9.15 (d, J = 4.0 Hz, 1H, Py-H), 9.46 (s, 1H, NH), 11.11 (s, 1H, NH). MS (ESI), m/z: 398 $(M-1)^-$. Elemental anal. (%), calculated: C, 42.08; H, 2.27; N, 21.03; found: C, 42.22; H, 2.35; N, 21.22.

1-(8-chloro-[1,2,4]triazolo[4,3-*a*]pyridine-3-carbonyl)-4-(2,3-dimethylphenyl)semicarbazide 4i: light yellow crystal, yield 85.66%, mp > 300 °C; ^{1}H NMR (DMSO-d_6, 400 MHz), δ: 2.12 (s, 3H, CH_3), 2.25 (s, 3H, CH_3), 6.92 (m, 1H, Ph-H), 7.04 (t, J = 7.6 Hz, 1H, Ph-H), 7.25 (t, J = 7.2 Hz, 1H, 1Py-H), 7.83 (d, J = 7.3 Hz, 1H, Py-H), 8.05 (s, 1H, Ph-H), 8.15 (s, 1H, NH), 8.51 (s, 1H, NH), 9.15 (d, J = 7.0 Hz, 1H, Py-H), 11.01 (s, 1H, NH). MS (ESI), m/z: 359 $(M+1)^+$. Elemental anal. (%), calculated: C, 53.56; H, 4.21; N, 23.42; found: C, 53.45; H, 4.45; N, 23.65.

1-(8-chloro-[1,2,4]triazolo[4,3-*a*]pyridine-3-carbonyl)-4-(2-methyl-4-nitrophenyl)semicarbazide 4j: light yellow crystal, yield 94.82%, mp > 300 °C; ^{1}H NMR (DMSO-d_6, 400 MHz), δ: 2.38 (s, 3H, CH_3), 7.26 (t, J = 7.1 Hz, 1H, Py-H), 7.84 (d, J = 7.3 Hz, 1H, Py-H), 7.99 (m, 3H, Ph-H) 8.50 (s, 1H, NH), 9.05 (s, 1H, NH), 9.14 (d, J = 6.8 Hz, 1H, Py-H), 11.19 (s, 1H, NH). MS (ESI), m/z: 390 $(M+1)^+$. Elemental anal. (%), calculated: C, 46.22; H, 3.10; N, 25.16; found: C, 46.13; H, 3.43; N, 25.25.

1-(8-chloro-[1,2,4]triazolo[4,3-*a*]pyridine-3-carbonyl)-4-(2-methyl-3-(trifluoromethyl)phenyl) semicarbazide 4k: light yellow crystal, yield 89.00%, mp > 300 °C; ^{1}H NMR (DMSO-d_6, 400 MHz), δ: 2.32 (s, 3H, CH_3), 7.26 (t, J = 7.1 Hz, 1H, Py-H), 7.38 (t, J = 7.5 Hz, 1H, Ph-H), 7.48 (t, J = 7.8 Hz, 2H, Ph-H), 7.84 (d, J = 7.3 Hz, 1H, Py-H), 8.47 (s, 1H, NH), 8.72 (s, 1H, NH), 9.15 (d, J = 6.8 Hz, 1H, Py-H), 11.10 (s, 1H, NH). MS (ESI), m/z: 413 $(M+1)^+$. Elemental anal. (%), calculated: C, 46.56; H, 2.93; N, 20.36; found: C, 46.76; H, 2.79; N, 20.51.

1-(8-chloro-[1,2,4]triazolo[4,3-*a*]pyridine-3-carbonyl)-4-(2-(trifluoromethoxy)phenyl)semicarbazide 4l: white crystal, yield 86.96%, mp > 300 °C; ^{1}H NMR (DMSO-d_6, 400 MHz), δ: 7.12 (t, J = 7.1 Hz, 1H, Ph-H), 7.25 (t, J = 7.1 Hz, 1H, Py-H), 7.35 (m, 2H, Ph-H), 7.83 (d, J = 7.2 Hz, 1H, Py-H), 8.16 (d, J = 9.6 Hz, 1H, Ph-H), 8.58 (s, 1H, NH), 8.94 (s, 1H, NH), 9.14 (d, J = 6.9 Hz, 1H, Py-H), 11.13 (s, 1H, NH). MS (ESI), m/z: 415 $(M+1)^+$. Elemental anal. (%), calculated: C, 43.44; H, 2.43; N, 20.26; found: C, 43.65; H, 2.62; N, 20.34.

1-(8-chloro-[1,2,4]triazolo[4,3-*a*]pyridine-3-carbonyl)-4-(naphthalen-1-yl)semicarbazide 4m: light yellow crystal, yield 93.00%, mp > 300 °C; ^{1}H NMR (DMSO-d_6, 400 MHz), δ: 7.26 (t, J = 7.2 Hz, 1H, Py-H), 7.50 (m, 4H, Ph-H), 7.70 (t, J = 8.4 Hz, 1H, Ph-H), 7.83 (d, J = 7.3 Hz, 1H, Py-H), 7.93 (d, J = 8.2 Hz, 1H, Ph-H), 8.13 (d, J = 8.0 Hz, 1H, Ph-H), 8.67 (s, 1H, NH), 8.91 (s, 1H, NH), 9.17 (d, J = 7.2 Hz, 1H, Py-H), 11.15 (s, 1H, NH). MS (ESI), m/z: 381 $(M+1)^+$. Elemental anal. (%), calculated: C, 56.78; H, 3.44; N, 22.07; found: C, 56.96; H, 3.53; N, 22.25.

4-(2-chloro-5-(trifluoromethyl)phenyl)-1-(8-chloro-[1,2,4]triazolo[4,3-*a*]pyridine-3-carbonyl) semicarbazide 4n: light yellow crystal, yield 57.10%, mp 252–255 °C; ^{1}H NMR (DMSO-d_6, 400 MHz), δ: 4.72 (d, J = 6.4 Hz, 2H, NH), 7.20 (t, J = 7.2 Hz, 1H, Py-H), 7.46 (m, 1H, Ph-H), 7.77 (d, J = 7.2 Hz, 1H, Py-H), 8.54 (s, 1H, Ph-H), 9.15 (d, J = 7.2 Hz, 1H, Py-H), 9.50 (s, 1H, Py-H), 10.54 (s, 1H, NH). MS (ESI), m/z: 434 $(M+1)^+$. Elemental anal. (%), calculated: C, 41.59; H, 2.09; N, 19.40; found: C, 41.55; H, 2.23; N, 19.54.

Acknowledgments

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