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Synthesis of New Cyclopenta[*b*][1,7]phenanthroline Derivatives

A. B. Tereshko[a] and N. G. Kozlov[a]*

[a] *Institute of Physical Organic Chemistry, National Academy of Sciences of Belarus, Minsk, Belarus*
**e-mail: loc@ifoch.bas-net.by*

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Abstract—New 7-aryl(hetaryl)-9,10-dihydro-7*H*-cyclopenta[*b*][1,7]phenanthrolin-8(11*H*)-ones have been synthesized by three-component condensation of quinolin-5-amine with cyclopentane-1,3-dione and aldehydes of the aromatic and heteroaromatic series.

Keywords: condensation, quinolin-5-amine, cyclopentane-1,3-dione, aromatic aldehydes, cyclopenta[*b*][1,7]-phenanthroline derivatives.

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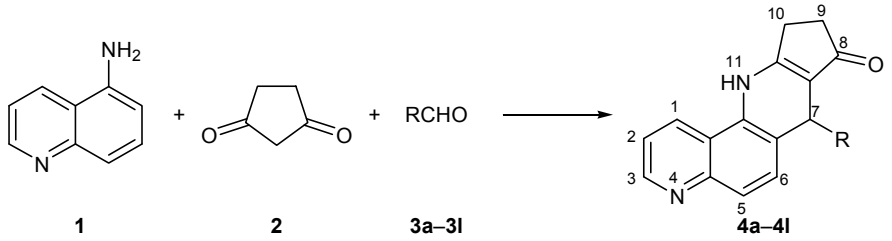
We previously showed that quinolin-6-amine reacts with aromatic aldehydes and methyl or methylene ketones (CH acids) to give 4,7-phenanthroline derivatives [1–4]. It was also found that cyclic 1,3-diketones were the most reactive CH acids [3, 4]. The condensation products, partially hydrogenated oxo derivatives of benzo[*b*][4,7]phenanthroline attract interest from the practical viewpoint as analogs of alkaloids, enzyme inhibitors, bactericidal agents, and antibiotics [5–8]. It is also known that organic compounds containing a cyclopenta[*b*]quinoline fragment often exhibit high biological activity [9, 10].

The present work was aimed at synthesizing previously unknown fused 1,7-phenanthroline derivatives that are isomeric to the above noted benzo[*b*][4,7]-phenanthrolines and are therefore expected to have a high biological potential [11]. For this purpose, we were the first to study the condensation of quinolin-5-amine (**1**) with cyclopentane-1,3-dione (**2**) and aromatic (heteroaromatic) aldehydes **3a**–**3l**. The condensation was accomplished by heating equimolar amounts of the reactants in butan-1-ol. Due to the high reactivity of cyclopentane-1,3-dione (**2**), it reacted with amine **1** and aldehydes **3** in alcoholic medium in the absence of a catalyst. The role of acid catalyst is played by protons released as a result of dissociation of the enol form of the β-diketone. The condensation selectively afforded 7-aryl(hetaryl)-9,10-dihydro-7*H*-cyclopenta[*b*][1,7]phenanthrolin-8(11*H*)-ones **4a**–**4l** which were isolated in 44–80% yield (Scheme 1).

By analogy with the data of Cortes et al. [12] who studied three-component condensation of naphthalen-1-amine (carbocyclic analog of quinolin-5-amine) with aromatic aldehydes and 5,5-dimethylcyclohexane-1,3-dione, the fused benzo[*b*][1,7]phenanthroline system can be formed via initial condensation of amine **1** with aldehyde **3** to give Schiff base **A**, addition of diketone **2** to the C=N bond of intermediate **A**, Hofmann–Martius-like rearrangement (migration of *N*-alkyl substituent in anilines to the aromatic ring [13]) of adduct **B**, and subsequent intramolecular cyclization of rearrangement product **C**. The transformation of intermediate **C** can also proceed through its hydramine fission into initial amine **1** and α,β-unsaturated ketone **D**. The C=C double bond of the latter is activated due to conjugation with two adjacent carbonyl group, and it reacts with amine **1** at the aromatic carbon atom in the α-position with respect to the amino group, which possesses the highest electron density. Amino diketone **B** thus formed undergoes dehydrocyclation to benzo-[*b*][1,7]phenanthrolinone **4** (Scheme 2). Alternatively, diketone **2** can react initially with aldehyde **3** to produce 2-arylmethylidenecyclopentane-1,3-dione **D** which then reacts with amine **1** as described above.

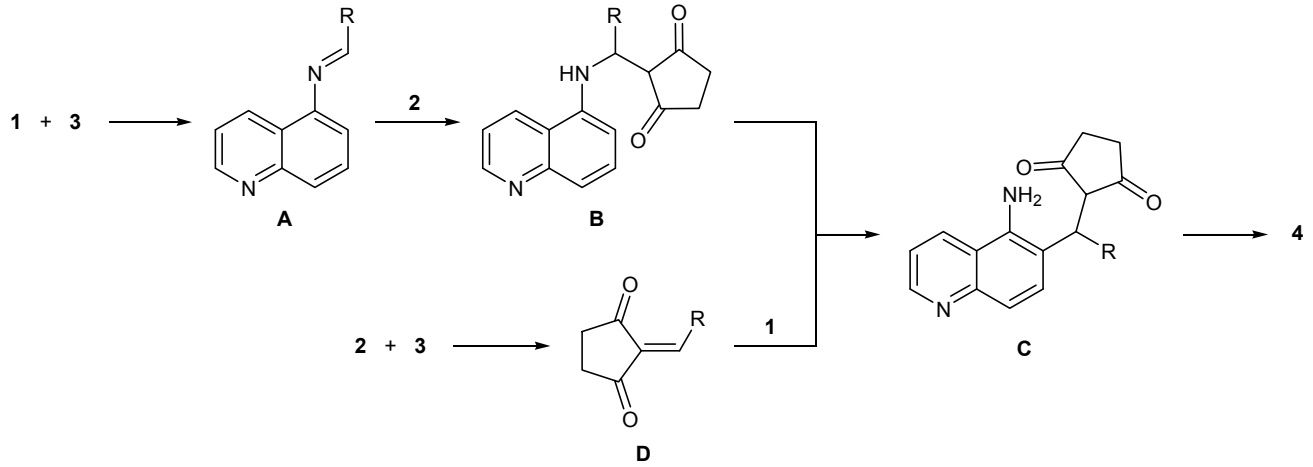
The yield of **4a**–**4l** depends on the R substituent. Benzaldehydes **3e**, **3h**, and **3i** containing hydroxy and alkoxy groups were converted to phenanthrolines **4e**, **4h**, and **4i**, respectively, in good yields 68–81%. Steric effect was observed in the reactions with 2-methylbenzaldehyde (**3a**) and 3-methylthiophene-2-carbalde-

Scheme 1.



R = 2-MeC_6H_4 (**a**), 4-MeC_6H_4 (**b**), 4-*i*-PrC_6H_4 (**c**), 2-IC_6H_4 (**d**), 3-HOC_6H_4 (**e**), 3,4,5-$(MeO)_3C_6H_2$ (**f**), 1,3-benzodioxol-5-yl (**g**), 4-$EtOC_6H_4$ (**h**), 4-$PrOC_6H_4$ (**i**), 4-$MeSC_6H_4$ (**j**), thiophen-2-yl (**k**), 3-methylthiophen-2-yl (**l**).

Scheme 2.



hyde (**3l**), and the yields of compounds **4a** and **4l** were lower (44–47%).

The structure of **4a**–**4l** was determined on the basis of their IR and ^{1}H NMR spectra. The IR spectra of **4a**–**4l** contained strong absorption bands at 1590 and 1525 cm^{-1}, which were assigned to vibrations of the vinylogous amide moiety. Strong bands at 3440 and 1620 cm^{-1} correspond to stretching and bending vibrations, respectively, of the secondary amino group. Stretching vibrations of aliphatic and cycloaliphatic C–H bonds appeared in the region 2960–2870 cm^{-1}, and aromatic C–H stretching bands were located at 3060–3030 cm^{-1}. Compounds **4f**–**4i** also displayed C–O–C stretchings at 1240–1230 cm^{-1}, and a strong C–S stretching band was observed in the spectrum of **4j** at 1125 cm^{-1}.

The ^{1}H NMR spectra of **4a**–**4l** were almost identical to those of isomeric 4,7-phenanthroline analogs [4]. The lack of spin–spin coupling between the NH proton and 7-H confirmed the assigned structure and the presence of a 1,4-dihydropyridine ring in their molecules. The NH and 7-H protons resonated as singlets at δ 9.14–9.58 and 5.21–5.66 ppm, respectively.

In summary, we have shown that the three-component condensation of quinolin-5-amine with cyclopentane-1,3-dione and aromatic (heteroaromatic) aldehydes provides an efficient and selective method of synthesis of new polynuclear heterocycles containing a pharmacophoric aryl or hetaryl substituent. The synthesized compounds are expected to exhibit a broad spectrum of biological activity.

EXPERIMENTAL

The IR spectra were recorded on a Nicolet Protégé-460 spectrometer with Fourier transform from samples prepared as KBr discs. The ^{1}H NMR spectra were recorded on Bruker AC 500 (500 MHz) and Tesla BS-567 (100 MHz) spectrometers using DMSO-d_6 as solvent and tetramethylsilane as internal standard. The melting points were measured on a Kofler hot stage.

7-Aryl(hetaryl)-9,10-dihydro-7*H*-cyclopenta[*b*]-[1,7]phenanthrolin-8(11*H*)-ones 4a–4l (*general procedure***).** A solution of 5 mmol of quinolin-5-amine (**1**), 5 mmol of cyclopentane-1,3-dione (**2**), and 5 mmol of aldehyde **3a**–**3l** in 20 mL of butan-1-ol was refluxed for 1–2 h. The mixture was cooled, and the

precipitate was filtered off, washed with diethyl ether, and recrystallized from ethanol–benzene (1:3).

7-(2-Methylphenyl)-9,10-dihydro-7*H*-cyclopenta-[*b*][1,7]phenanthrolin-8(11*H*)-one (4a). Yield 45%, mp 202–203°C. ^{1}H NMR spectrum, δ, ppm: 2.63 s (3H, Me), 3.27 m (2H, 10-H), 3.75 m (2H, 9-H), 5.43 s (1H, 7-H); 6.69 t, 6.86 d, 7.00 t, and 7.08 t (1H each, 3′-H–6′-H, $J_{3',4'}$ = 8.1, $J_{4',5'} = J_{5',6'}$ = 8.5 Hz); 7.38 d.d (1H, 2-H, $J_{2,1}$ = 8.9, $J_{2,3}$ = 4.1 Hz), 7.43 d (1H, 6-H, $J_{6,5}$ = 8.9 Hz), 7.57 d (1H, 5-H, $J_{5,6}$ = 8.9 Hz), 8.84 d (1H, 3-H, $J_{3,2}$ = 4.0 Hz), 8.90 d (1H, 1-H, $J_{1,2}$ = 8.8 Hz), 9.34 s (1H, NH). Found, %: C 80.88; H 5.48; N 8.49. $C_{22}H_{18}N_2O$. Calculated, %: C 80.96; H 5.56; N 8.58.

7-(4-Methylphenyl)-9,10-dihydro-7*H*-cyclopenta-[*b*][1,7]phenanthrolin-8(11*H*)-one (4b). Yield 51%, mp 207–208°C. ^{1}H NMR spectrum, δ, ppm: 2.48 s (3H, Me), 3.26 m (2H, 10-H), 3.74 m (2H, 9-H), 5.26 s (1H, 7-H), 7.09 d (2H, 2′-H, 6′-H, $J_{2',3'} = J_{6',5'}$ = 8.4 Hz), 7.26 d (2H, 3′-H, 5′-H, $J_{3',2'} = J_{5',6'}$ = 8.4 Hz), 7.33 d (1H, 6-H, $J_{6,5}$ = 9.0 Hz), 7.43 d.d (1H, 2-H, $J_{2,1}$ = 8.9, $J_{2,3}$ = 4.1 Hz), 7.52 d (1H, 5-H, $J_{5,6}$ = 9.0 Hz), 8.76 d (1H, 3-H, $J_{3,2}$ = 4.1 Hz), 8.83 d (1H, 1-H, $J_{1,2}$ = 8.9 Hz), 9.19 s (1H, NH). Found, %: C 80.89; H 5.47; N 8.47. $C_{22}H_{18}N_2O$. Calculated, %: C 80.96; H 5.56; N 8.58.

7-[4-(Propan-2-yl)phenyl]-9,10-dihydro-7*H*-cyclopenta[*b*][1,7]phenanthrolin-8(11*H*)-one (4c). Yield 74%, mp 214–215°C. ^{1}H NMR spectrum, δ, ppm: 2.73 m (7H, *i*-Pr), 3.28 m (2H, 10-H), 3.75 m (2H, 9-H), 5.28 s (1H, 7-H), 7.10 d (2H, 2′-H, 6′-H, $J_{2',3'} = J_{6',5'}$ = 8.4 Hz), 7.26 d (2H, 3′-H, 5′-H, $J_{3',2'} = J_{5',6'}$ = 8.4 Hz), 7.34 d (1H, 6-H, $J_{6,5}$ = 9.0 Hz), 7.42 d.d (1H, 2-H, $J_{2,1}$ = 8.9, $J_{2,3}$ = 4.1 Hz), 7.51 d (1H, 5-H, $J_{5,6}$ = 9.0 Hz), 8.77 d (1H, 3-H, $J_{3,2}$ = 4.1 Hz), 8.82 d (1H, 1-H, $J_{1,2}$ = 8.9 Hz), 9.20 s (1H, NH). Found, %: C 81.22; H 6.17; N 7.79. $C_{24}H_{22}N_2O$. Calculated, %: C 81.33; H 6.26; N 7.90.

7-(2-Iodophenyl)-9,10-dihydro-7*H*-cyclopenta-[*b*][1,7]phenanthrolin-8(11*H*)-one (4d). Yield 44%, mp 230–231°C. ^{1}H NMR spectrum, δ, ppm: 3.31 m (2H, 10-H), 3.81 m (2H, 9-H), 5.66 s (1H, 7-H); 6.96 t, 7.12 t, and 7.24 d (4H, 3′-H–6′-H, $J_{3',4'}$ = 8.1, $J_{4',5'} = J_{5',6'}$ = 8.5 Hz); 7.42 d.d (1H, 2-H, $J_{2,1}$ = 8.9, $J_{2,3}$ = 4.1 Hz), 7.46 d (1H, 6-H, $J_{6,5}$ = 8.9 Hz), 7.55 d (1H, 5-H, $J_{5,6}$ = 8.9 Hz), 8.76 d (1H, 3-H, $J_{3,2}$ = 4.0 Hz), 8.81 d (1H, 1-H, $J_{1,2}$ = 8.8 Hz), 9.19 s (1H, NH). Found, %: C 57.42; H 3.34; I 28.85; N 6.27. $C_{21}H_{15}IN_2O$. Calculated, %: C 57.55; H 3.45; I 28.96; N 6.39.

7-(3-Hydroxyphenyl)-9,10-dihydro-7*H*-cyclopenta[*b*][1,7]phenanthrolin-8(11*H*)-one (4e). Yield 68%, mp 265–266°C. ^{1}H NMR spectrum, δ, ppm: 3.37 m (2H, 10-H), 3.86 m (2H, 9-H), 5.30 s (1H, 7-H), 7.18 d (2H, 5′-H, 6′-H, $J_{5',6'} = J_{5',4'}$ = 8.8 Hz), 7.41 d.d (1H, 2-H, $J_{2,1}$ = 8.7, $J_{2,3}$ = 4.0 Hz), 7.52 d (1H, 6-H, $J_{6,5}$ = 8.9 Hz), 7.55 d (1H, 5-H, $J_{5,6}$ = 8.9 Hz), 8.19 d (1H, 4′-H, $J_{4',5'}$ = 8.8 Hz), 8.51 s (1H, 2′-H), 8.62 s (1H, OH), 8.73 d (1H, 3-H, $J_{3,2}$ = 4.0 Hz), 8.79 d (1H, 1-H, $J_{1,2}$ = 8.7 Hz), 9.11 s (1H, NH). Found, %: C 76.72; H 4.83; N 8.47. $C_{21}H_{16}N_2O_2$. Calculated, %: C 76.81; H 4.91; N 8.53.

7-(3,4,5-Trimethoxyphenyl)-9,10-dihydro-7*H*-cyclopenta[*b*][1,7]phenanthrolin-8(11*H*)-one (4f). Yield 71%, mp 210–211°C. ^{1}H NMR spectrum, δ, ppm: 3.38 m (2H, 10-H), 3.82 m (2H, 9-H), 3.61 s (6H, MeO), 3.71 s (3H, MeO), 5.35 s (1H, 7-H), 6.74 s (1H, 2′-H), 6.91 s (1H, 6′-H), 7.42 d.d (1H, 2-H, $J_{2,1}$ = 8.9, $J_{2,3}$ = 4.2 Hz), 7.51 d (1H, 6-H, $J_{6,5}$ = 8.9 Hz), 7.57 d (1H, 5-H, $J_{5,6}$ = 8.9 Hz), 8.82 d (1H, 3-H, $J_{3,2}$ = 4.2 Hz), 8.91 d (1H, 1-H, $J_{1,2}$ = 8.9 Hz), 9.26 s (1H, NH). Found, %: C 71.52; H 5.47; N 6.88. $C_{24}H_{22}N_2O_4$. Calculated, %: C 71.63; H 5.51; N 6.96.

7-(1,3-Benzodioxol-5-yl)-9,10-dihydro-7*H*-cyclopenta[*b*][1,7]phenanthrolin-8(11*H*)-one (4g). Yield 75%, mp 253–253°C. ^{1}H NMR spectrum, δ, ppm: 3.37 m (2H, 10-H), 3.87 m (2H, 9-H), 5.22 s (1H, 7-H), 5.83 s (2H, OCH_2O), 6.65 d (1H, 6′-H, $J_{6',5'}$ = 8.8 Hz), 6.71 d (1H, 5′-H, $J_{5',6'}$ = 8.8 Hz), 6.76 s (1H, 2′-H), 7.53 d (1H, 6-H, $J_{6,5}$ = 8.8 Hz), 7.55 d (1H, 5-H, $J_{5,6}$ = 8.8 Hz), 7.57 d.d (1H, 2-H, $J_{2,1}$ = 8.7, $J_{2,3}$ = 4.0 Hz), 8.84 d (1H, 3-H, $J_{3,2}$ = 4.0 Hz), 8.91 d (1H, 1-H, $J_{1,2}$ = 8.7 Hz), 9.41 s (1H, NH). Found, %: C 74.07; H 4.45; N 7.74. $C_{22}H_{16}N_2O_3$. Calculated, %: C 74.15; H 4.53; N 7.86.

7-(4-Ethoxyphenyl)-9,10-dihydro-7*H*-cyclopenta-[*b*][1,7]phenanthrolin-8(11*H*)-one (4h). Yield 81%, mp 223–224°C. ^{1}H NMR spectrum, δ, ppm: 1.27 t (3H) and 3.86 q (2H) (OEt), 3.45 m (2H, 10-H), 3.87 m (2H, 9-H), 5.21 s (1H, 7-H), 6.76 d (2H, 2′-H, 6′-H, $J_{2',3'} = J_{6',5'}$ = 8.5 Hz), 7.15 d (2H, 3′-H, 5′-H, $J_{3',2'} = J_{5',6'}$ = 8.5 Hz), 7.50 d.d (1H, 2-H, $J_{2,1}$ = 8.8, $J_{2,3}$ = 4.0 Hz), 7.52 d (1H, 6-H, $J_{6,5}$ = 8.9 Hz), 7.58 d (1H, 5-H, $J_{5,6}$ = 8.9 Hz), 8.82 d (1H, 3-H, $J_{3,2}$ = 4.0 Hz), 8.90 d (1H, 1-H, $J_{1,2}$ = 8.8 Hz), 9.31 s (1H, NH). Found, %: C 77.44; H 5.49; N 7.72. $C_{23}H_{20}N_2O_2$. Calculated, %: C 77.51; H 5.66; N 7.86.

7-(4-Propoxyphenyl)-9,10-dihydro-7*H*-cyclopenta[*b*][1,7]phenanthrolin-8(11*H*)-one (4i). Yield 77%, mp 293–294°C. ^{1}H NMR spectrum, δ, ppm:

0.96 t, 1.64 q, and 3.74 t (3H, OPr); 3.47 m (2H, 10-H), 3.86 m (2H, 9-H), 5.25 s (1H, 7-H), 6.75 d (2H, 2′-H, 6′-H, $J_{2',3'} = J_{6',5'}$ = 8.5 Hz), 7.14 d (2H, 3′-H, 5′-H, $J_{3',2'} = J_{5',6'}$ = 8.4 Hz), 7.50 d.d (1H, 2-H, $J_{2,1}$ = 8.7, $J_{2,3}$ = 4.0 Hz), 7.55 d (1H, 6-H, $J_{6,5}$ = 8.8 Hz), 7.58 d (1H, 5-H, $J_{5,6}$ = 8.9 Hz), 8.82 d (1H, 3-H, $J_{3,2}$ = 4.0 Hz), 8.90 d (1H, 1-H, $J_{1,2}$ = 8.8 Hz), 9.40 s (1H, NH). Found, %: C 77.69; H 5.84; N 7.45. $C_{24}H_{22}N_2O_2$. Calculated, %: C 77.81; H 5.99; N 7.56.

7-[4-(Methylsulfanyl)phenyl]-9,10-dihydro-7*H*-cyclopenta[*b*][1,7]phenanthrolin-8(11*H*)-one (4j). Yield 55%, mp 215–216°C. ^{1}H NMR spectrum, δ, ppm: 2.35 s (3H, SMe), 3.26 m (2H, 10-H), 3.87 m (2H, 9-H), 5.27 s (1H, 7-H), 6.74 d (2H, 2′-H, 6′-H, $J_{2',3'} = J_{6',5'}$ = 8.5 Hz), 7.14 d (2H, 3′-H, 5′-H, $J_{3',2'} = J_{5',6'}$ = 8.4 Hz), 7.51 d.d (1H, 2-H, $J_{2,1}$ = 8.8, $J_{2,3}$ = 4.1 Hz), 7.55 d (1H, 6-H, $J_{6,5}$ = 8.8 Hz), 7.58 d (1H, 5-H, $J_{5,6}$ = 8.9 Hz), 8.82 d (1H, 3-H, $J_{3,2}$ = 4.0 Hz), 8.90 d (1H, 1-H, $J_{1,2}$ = 8.8 Hz), 9.40 s (1H, NH). Found, %: C 73.60; H 4.93; N 7.69; S 8.79. $C_{22}H_{18}N_2OS$. Calculated, %: C 73.71; H 5.06; N 7.82; S 8.95.

7-(Thiophen-2-yl)-9,10-dihydro-7*H*-cyclopenta-[*b*][1,7]phenanthrolin-8(11*H*)-one (4k). Yield 54%, mp 243–244°C. ^{1}H NMR spectrum, δ, ppm: 3.26 m (2H, 10-H), 3.87 m (2H, 9-H), 5.60 s (1H, 7-H), 7.14 m (3H, H_{Th}), 7.47 d.d (1H, 2-H, $J_{2,1}$ = 8.7, $J_{2,3}$ = 4.0 Hz), 7.51 d (1H, 6-H, $J_{6,5}$ = 8.9 Hz), 7.57 d (1H, 5-H, $J_{5,6}$ = 8.9 Hz), 8.85 d (1H, 3-H, $J_{3,2}$ = 4.0 Hz), 8.91 d (1H, 1-H, $J_{1,2}$ = 8.7 Hz), 9.54 s (1H, NH). Found, %: C 71.55; H 4.29; N 8.68; S 9.93. $C_{19}H_{14}N_2OS$. Calculated, %: C 71.67; H 4.43; N 8.80; S 10.07.

7-(3-Methylthiophen-2-yl)-9,10-dihydro-7*H*-cyclopenta[*b*][1,7]phenanthrolin-8(11*H*)-one (4l). Yield 47%, mp 272–273°C. ^{1}H NMR spectrum, δ, ppm: 2.52 s (3H, Me), 3.27 m (2H, 10-H), 3.88 m (2H, 9-H), 5.59 s (1H, 7-H), 6.68 d (1H, 4′-H, $J_{4',5'}$ = 5.0 Hz), 7.04 d (1H, 5′-H, $J_{5',4'}$ = 5.0 Hz), 7.46 d.d (1H, 2-H, $J_{2,1}$ = 8.7, $J_{2,3}$ = 4.0 Hz), 7.51 d (1H, 6-H, $J_{6,5}$ = 8.9 Hz), 7.57 d (1H, 5-H, $J_{5,6}$ = 8.9 Hz), 8.86 d (1H, 3-H, $J_{3,2}$ = 4.0 Hz), 8.92 d (1H, 1-H, $J_{1,2}$ = 8.7 Hz), 9.55 s (1H, NH). Found, %: C 72.13; H 4.76; N 8.31; S 9.54. $C_{20}H_{16}N_2OS$. Calculated, %: C 72.26; H 4.85; N 8.43; S 9.65.

CONFLICT OF INTERESTS

The authors declare the absence of conflict of interests.

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