REACTIVITY OF 4-BROMOACETYL-1,2,3-TRIAZOLES TOWARDS AMINES AND PHENOLS: SYNTHESIS AND ANTIMICROBIAL ACTIVITY OF NOVEL HETEROCYCLES

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Abstract – The reactivity of 4-bromoacetyl-1H-1,2,3-triazoles towards amines and phenols was studied. Reaction of 4-bromoacetyl-1H-1,2,3-triazole (1a; R = H) with benzylamine (2) in the absence of any catalyst unexpectedly afforded heterocycle 2,5-bis(5-methyl-1-phenyl-1H-1,2,3-triazol-4-yl)pyrazine (4) in 50% yield. Additionally, the reaction of 1b (R = Br) with 4-bromoaniline and of 1c (R = Me) or 1d (R = NO₂) with 1H-benzotriazole in basic media gave the expected aminoketone products 8 and 9a or 9b in high yields. Furthermore, the reaction of 1c (R = Me) or 1e (R = Cl) with phenol or β-naphthol in basic media led to the production of keto-ethers 10 or 11, respectively in an excellent yield. Compound 10 showed the highest inhibitory effect against the growth of the tested pathogens.

INTRODUCTION

Pyrazines are a common class of heterocyclic compounds that have many applications.1 The compounds are important intermediates in the total synthesis of complex derivatives, and are used in medicinal chemistry, chemical biology, materials, imaging, and dyes.2-5 The synthesis of heterocycles containing pyrazine ring systems is therefore of wide interest.6-9 The traditional method for the synthesis of pyrazines involves the use of 1,2-diketones in the presence of ammonia to produce the corresponding diamines or
aminoketones followed by condensation and oxidation.\textsuperscript{10–13} They can also be produced using $\alpha$-halo- or $\alpha$-hydroxy-ketones,\textsuperscript{14,15} $2H$-azirines,\textsuperscript{16} and nitro epoxides.\textsuperscript{17} Aminoketones have been shown to display antibacterial, antifungal, and antiviral activities\textsuperscript{18–22} and are widely used as key intermediates in many organic synthesis transformations.\textsuperscript{23} In addition, $1,2,3$-triazole-containing heterocycles exhibit a variety of biological properties including antibacterial and antiviral activities.\textsuperscript{24–26} Consequently, in continuation of our previous related work,\textsuperscript{27,28} we have attempted the synthesis of novel heterocyclic compounds containing the $1,2,3$-triazole ring system.

RESULTS AND DISCUSSION

The reactivity of $4$-bromoacetyl-$1H$-$1,2,3$-triazoles $1a$–$e$ ($1a$: $R = H$, $1b$: $R = Br$, $1c$: $R = Me$, $1d$: $R = NO_2$, and $1e$: $R = Cl$) towards amines and phenols has been investigated. A catalyst-free reaction between $2$-bromo-$1$-(5-methyl-$1$-phenyl-$1H$-$1,2,3$-triazol-$4$-yl)ethan-$1$-one ($1a$) and benzylamine ($2$) in boiling ethanol (EtOH) afforded the unexpected product, $2,5$-bis(5-methyl-$1$-phenyl-$1H$-$1,2,3$-triazol-$4$-yl)pyrazine ($4$), in $50\%$ yield rather than the expected $2$-(benzylamino)-$1$-(5-methyl-$1$-phenyl-$1H$-$1,2,3$-triazol-$4$-yl)ethan-$1$-one ($3$; Scheme 1). Clearly, $2$ acted as a nitrogen source to produce $4$. It should be noted that reactions of other $1H$-$1,2,3$-triazoles $1b$–$e$ and $2$ failed to produce pure products.

\begin{center}
\includegraphics[width=\textwidth]{Scheme_1}
\end{center}

\textbf{Scheme 1.} Synthesis of $4$

The chemical structure of $4$ was established by NMR spectral data and single-crystal X-ray crystallography. The $^1H$ NMR spectrum of $4$ did not show the exchangeable singlet signal corresponding to the NH proton. Instead, it showed the presence of a characteristic singlet signal that appeared at $9.45$ ppm corresponding to the two pyrazine protons.

The molecule of $4$ is symmetrical in the crystal structure with an inversion center located in the middle of the pyrazine ring (Figure 1). In the crystal, the methyl-triazolyl and pyrazinyl groups are coplanar with a
twist angle of 10.2(1)° between adjacent rings. The phenyl group is twisted further, making an angle of 50.5 (5)° with the methyl-triazolyl group.

Figure 1. The molecule of compound 4 showing atomic displacement ellipsoids at 50% probability

A proposed mechanism for the formation of 4 is shown in Scheme 2. The formation of 4 could involve [3+3] cyclization. Dehydrobromination from 1 and 2 leads to the intermediate 5, which undergoes a dimerization reaction with the elimination of two moles of H2O to result in the formation of the intermediate 7. Aromatization of 7 through the elimination of two moles of benzyl fragments gives 4. The oxidative process of benzylic C–H bonds has been reported to take place under mild conditions.

Scheme 2. A proposed mechanism for the formation of 4

The reactivity of 1b (R = Br), 1c (R = Me) or 1d (R = NO2) towards 4-bromoaniline or 1H-benzotriazole in boiling absolute EtOH in the presence of triethylamine (Et3N) was also investigated (Scheme 3). While, the reaction between 1b and 4-bromoaniline gave 8 in 82% yield, reactions of 1c or 1d and 1H-benzotriazole under similar reaction conditions gave the corresponding 9a or 9b, respectively, in both 83% yields (Scheme 3).
Scheme 3. Synthetic routes to aminoketones 8 and 9

The $^1$H NMR spectrum of 8 showed a characteristic exchangeable singlet signal at 6.23 ppm corresponding to the NH proton. The protons of the CH$_2$ group in compound 9a appeared at 6.50 ppm in its $^1$H NMR spectrum. The NMR spectra of 8 and 9 showed the presence of the CH$_2$ protons and carbons.

The structures of 8, 9a, and 9b are shown in Figures 2, 3, and 4, respectively. The crystal structure of 8 contains two independent types of molecules with different conformations (Figure 2). In both molecules, one bromo-phenyl (Br1, C1–C6 or Br3, C18–C23), aminoacetaldehyde and methyl-triazolyl groups are almost coplanar. The twist angles between these bromo-phenyl and methyl-triazolyl groups are 13.6 (1)$^\circ$ for the first molecule and 18.1(2)$^\circ$ for the second. The second bromo-phenyl group (Br2, C12–C17 or Br4, C29–C34) of each molecule is twisted farther (by 43.7(1)$^\circ$ for the first molecule or 40.4(1)$^\circ$ for the second) from the methyl-triazolyl group.

Figure 2. The asymmetric unit of 8 showing atomic displacement ellipsoids at 50% probability
In the crystal structure of 9a, the acetaldehyde and methyl-triazolyl groups of the molecule are coplanar whereas the benzotriazolyl and methyl-phenyl groups are twisted from this plane (Figure 3). The angles between the latter two groups and the methyl-triazolyl groups are 70.9(1)° and 63.2(1)° respectively.

Figure 3. The molecule of 9a showing atomic displacement ellipsoids at 50% probability

In the crystal structure of 9b, the twist angle between the phenyl and nitro groups of the molecule is 10.0(2)°. Comparably to 9a, the acetaldehyde and methyl-triazolyl groups in the middle of the molecule are coplanar. The benzotriazolyl and methyl-phenyl groups are twisted from the plane of the triazolyl group by 86.6(1)° and 35.3(1)°, respectively (Figure 4).

Figure 4. The molecule of 9b showing atomic displacement ellipsoids at 50% probability
Finally, the reaction between 1c (R = Me) or 1e (R = Cl) and phenol or β-naphthol under reflux conditions in dry acetone containing anhydrous potassium carbonate (K₂CO₃) furnished the corresponding β-keto-ethers 10 or 11, respectively, in both 89% yields (Scheme 4).

Scheme 4. Synthetic routes to β-keto-esters 10 and 11

The ¹H NMR spectra of 10 and 11 showed characteristic singlet signals that appeared at 5.56 and 5.70 ppm, respectively corresponding to the CH₂ protons. Also, the CH₂ carbon in both compounds 10 and 11 appeared at 70.4 and 70.7 ppm, respectively in their ¹³C NMR spectra.

The X-ray crystal structures of 10 and 11 are shown in Figures 5 and 6, respectively. In the crystal of 10, the acetaldehyde and methyl-triazolyl groups in the middle of the molecule are also coplanar. The twist angles from the plane of the methyl-triazolyl by the phenyl-hydroxy and methyl-phenyl groups are 17.0(1)° and 35.1(1)° respectively (Figure 5).

Figure 5. The molecule of 10 showing atomic displacement ellipsoids at 50% probability
There are two independent types of molecules, with different conformations, in the crystal structure of molecule 11 (Figure 6). In the central part of both molecules, the acetaldehyde and methyl-triazolyl groups are coplanar. The twist angles between the chlorophenyl and methyl-triazolyl groups are 34.4(1)° and 40.7(1)° for the first and second molecules respectively. The twist angles between the oxy-naphthalene and methyl-triazolyl groups are 37.8(1)° and 10.7(1)° for the first and second molecules respectively.

![Figure 6. Asymmetric unit of 11 showing atomic displacement ellipsoids at 50% probability](image)

The antimicrobial activities of the synthesized heterocycles were determined against pathogens obtained from the American type culture collection (ATCC; Rockville, MD, USA). The organisms used in the tests were Staphylococcus aureus ATCC-47077 (S. aureus), Listeria monocytogenes ATCC-35152 (L. monocytogenes), Escherichia coli ATCC-25922 (E. coli), Salmonella typhi ATCC-15566 (S. typhi), and Candida albicans ATCC-10231 (C. albicans). Ampicillin and vancomycin were used as the reference antibiotics for comparison.

The results of antimicrobial activity are summarized in Table 1. All the compounds displayed at least moderate activity against the microorganisms (the exceptions were 4 and 8 which had limited activity against S. typhimurium). Compound 10 was the most effective heterocycle and against all the microorganisms tested. Particularly notable was the observation that compound 10 was more effective than ampicillin and vancomycin. Ampicillin and vancomycin are recognized as essential antibiotics. The
minimal inhibitory concentration (MIC) of the new heterocycles was also investigated. The results for compound 10 indicated that a concentration from 50–70 µg/mL was needed to kill all the pathogens.

Table 1. Antimicrobial activity (mm) of the synthesized heterocycles

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CONCLUSIONS

Reactions of 4-bromoacetyl-1H-1,2,3-triazoles towards amines and phenols provided novel heterocyclic compounds. Markedly, 2,5-bis(5-methyl-1-phenyl-1H-1,2,3-triazol-4-yl)pyrazine was unexpectedly produced from a catalyst free reaction of 2-bromo-1-(5-methyl-1-phenyl-1H-1,2,3-triazol-4-yl)ethan-1-one and benzylamine. Of all the synthesized heterocycles, compound 1-[5-methyl-1-(4-methylphenyl)-1H-1,2,3-triazol-4-yl]-2-phenoxyethan-1-one (10) showed the highest activity against the tested pathogens with a performance exceeding that of the reference drugs (ampicillin and vancomycin).

EXPERIMENTAL

General Melting points were determined using an Electrothermal melting point apparatus. A Bruker Tensor 27 FTIR Spectrometer was used to record the IR spectra. The NMR spectra were recorded on a JEOL NMR 500 MHz spectrometer. 1H (500 MHz) and 13C NMR (125 MHz) spectra were recorded in deuterated dimethyl sulfoxide (DMSO-d6) using tetramethylsilane as a standard. The chemical shift (δ) is reported in ppm and the chemical shift (J) is reported in Hz. Compounds 1a–e34 were prepared based on literature procedures.

Synthesis of 2,5-bis(5-methyl-1-phenyl-1H-1,2,3-triazol-4-yl)pyrazine (4). A mixture of 1a (0.56 g, 2.0 mmol) and 2 (0.43 g, 4.0 mmol) in dry EtOH (20 mL) was refluxed for 3 h to give 4 as a colorless solid. Crystallization from DMF by slow evaporation of solvent at rt gave crystals of pure 4. Yield 50%, mp 239–241 °C. IR (νmax, cm⁻¹): 3300 (NH), 2946 (CH), 1685 (C=O), 1593 (C=C). 1H NMR: 2.45 (s, 6H, 2 Me), 7.30 (s, 2H, Ar), 7.45–7.60 (m, 10 H, Ar). Anal. Calcd for C22H18N8 (394.16): C, 66.99; H, 4.60; N, 28.41; Found: C, 67.21, H, 4.67, N, 28.55%.
General procedure for the synthesis of 8–11. A mixture of 1 (2.0 mmol) and appropriate reagent (2 mmol; 4-bromoaniline for production of 8, 1H-benzotriazole for 9a,b, phenol for 10 or β-naphthol for 11) in dry EtOH in the cases of 8 or 9a,b or acetone in the cases of 10 or 11 (20 mL) containing anhydrous K$_2$CO$_3$ (4 mmol, 0.55 g) or Et$_3$N (4 mmol, 0.40 g) was refluxed for 8 h to give the corresponding products 8–11. The products were purified by crystallization using DMF by slow evaporation of solvent at rt.

1-[1-(4-Bromophenyl)-5-methyl-1H-1,2,3-triazol-4-yl]-2-[(4-bromophenyl)amino]ethan-1-one  (8)

Yield 82%, mp 193–195 °C. IR ($\nu_{\text{max}}, \text{cm}^{-1}$): 3392 (NH), 2920 (CH), 1687 (C=O), 1590 (C=C). $^1$H NMR: 2.40 (s, 3H, Me), 4.68 (s, 2H, CH$_2$), 6.23 (s, exch., 1H, NH), 6.57 (d, $J = 7.6$ Hz, 2H, Ar), 7.17 (d, $J = 7.6$ Hz, 2H, Ar), 7.60 (d, $J = 7.6$ Hz, 2H, Ar), 7.83 (d, $J = 7.6$ Hz, 2H, Ar). $^{13}$C NMR: 10.2, 50.7, 107.4, 114.8, 124.0, 127.9, 131.87, 133.3, 134.7, 138.8, 141.9, 148.1, 192.4. Anal. Calcd for C$_{17}$H$_{15}$Br$_2$N$_4$O (447.95): C, 45.36; H, 3.14; N, 12.45; Found: C, 45.50, H, 3.26, N, 12.57%.

2-(1H-Benzotriazol-1-yl)-1-[5-methyl-1-(4-methylphenyl)-1H-1,2,3-triazol-4-yl]ethan-1-one  (9a)

Yield 83%, mp 163–165 °C. IR ($\nu_{\text{max}}, \text{cm}^{-1}$): 2984 (CH), 1692 (C=O), 1587 (C=C). $^1$H NMR: 2.40 (s, 3H, Me), 2.46 (s, 3H, Me), 6.50 (s, 2H, CH$_2$), 7.41 (d, $J = 7.5$ Hz, 2H, Ar), 7.52 (d, $J = 7.5$ Hz, 2H, Ar), 7.82 (d, $J = 7.5$ Hz, 2H, Ar), 8.07 (d, $J = 7.5$ Hz, 2H, Ar). $^{13}$C NMR: 10.2, 21.3, 54.8, 111.5, 118.4, 119.6, 124.4, 125.8, 127.9, 130.8, 132.9, 134.6, 139.6, 140.8, 145.7, 187.7. Anal. Calcd for C$_{18}$H$_{16}$N$_6$O (332.14): C, 65.05; H, 4.85; N, 25.29; Found: C, 65.17, H, 4.92, N, 25.38%.

2-(1H-Benzotriazol-1-yl)-1-[5-methyl-1-(4-nitrophenyl)-1H-1,2,3-triazol-4-yl]ethan-1-one  (9b)

Yield 83%, mp 188–190 °C. IR ($\nu_{\text{max}}, \text{cm}^{-1}$): 2990 (CH), 1698 (C=O), 1588 (C=C). $^1$H NMR: 2.47 (s, 3H, Me), 6.52 (s, 2H, CH$_2$), 7.41 (t, $J = 7.5$, 1H, Ar), 7.81 (t, $J = 7.5$, 1H, Ar), 7.98 (d, $J = 7.5$, 1H, Ar), 8.01 (d, $J = 8.6$ Hz, 2H, Ar), 8.06 (d, $J = 7.5$ Hz, 1H, Ar), 8.50 (d, 2H, $J = 8.6$ Hz, Ar). $^{13}$C NMR: 10.3, 54.9, 111.5, 119.6, 124.5, 125.8, 127.1, 128.0, 134.6, 140.2, 141.5, 145.7, 148.7, 187.7. Anal. Calcd for C$_{18}$H$_{15}$N$_7$O$_3$ (363.11): C, 56.20; H, 3.61; N, 26.99; Found: C, 56.28, H, 3.70, N, 27.09%.

1-[5-Methyl-1-(4-methylphenyl)-1H-1,2,3-triazol-4-yl]-2-phenoxyethan-1-one  (10)

Yield 89%, mp 145–147 °C. IR ($\nu_{\text{max}}, \text{cm}^{-1}$): 2896 (CH), 1701 (C=O), 1601 (C=C). $^1$H NMR: 2.39 (s, 3H, Me), 2.49 (s, 3H, Me), 5.56 (s, 2H, CH$_2$), 6.92–6.94 (m, 3H, Ar), 7.26 (t, $J = 7.5$ Hz, 2H, Ar), 7.44 (d, $J = 7.7$ Hz, 2H, Ar), 7.50 (d, $J = 7.7$ Hz, 2H, Ar). $^{13}$C NMR: 10.1, 21.3, 70.4, 115.1, 121.5, 125.7, 130.0, 130.7, 132.9, 138.9, 140.7, 141.2, 158.5, 190.2. Anal. Calcd for C$_{18}$H$_{17}$N$_3$O$_2$ (307.13): C, 70.34; H, 5.58; N, 13.67; Found: C, 70.45, H, 5.69, N, 13.75%.

1-[1-(4-Chlorophenyl)-5-methyl-1H-1,2,3-triazol-4-yl]-2-(naphthalen-2-yloxy)ethan-1-one  (11)

Yield 89%, mp 155–157 °C. IR ($\nu_{\text{max}}, \text{cm}^{-1}$): 2901 (CH), 1702 (C=O), 1586 (C=C). $^1$H NMR: 2.47 (s, 3H, Me), 5.70 (s, 2H, CH$_2$), 7.34–7.50 (m, 4H, Ar), 7.69–7.83 (m, 7H, Ar). $^{13}$C NMR: 10.1, 70.7, 107.8, 119.0, 124.3,
126.9, 127.3, 127.7, 128.0, 129.3, 129.9, 130.4, 134.2, 134.768, 135.5, 139.1, 156.4, 189.8. Anal. Calcd for C_{21}H_{16}ClN_{3}O_{2} (377.09): C, 66.76; H, 4.27; N, 11.12; Found: C, 66.88, H, 4.38, N, 11.30%.

**Antimicrobial Activity.** The agar well diffusion procedure was used as a medium to investigate the antimicrobial activities of the newly synthesized heterocycles.\(^{35,36}\) Ampicillin and vancomycin were used as references drugs and their effects on the pathogens were recorded for comparison. Bacterial (70 µL) and yeast (106 CFU/mL) cells were spread on plates containing nutrient agar. The wells (6 mm diameter) were excavated on the injected agar plates, then each sample (200 mg) in DMSO (1 mL) was added. The reference antibiotics disks (10 and 30 µg/disk of ampicillin and vancomycin, respectively) were introduced on the surface of agar inoculated plates. The plates were kept at 4 °C for 2 h before incubation to permit the diffusion to occur. The plates were kept at 37 °C for 24 h except for yeast strains that were incubated at 28 °C for 24 h. The diameter of the inhibition zone (mm) was measured. The tests were replicated five times and the averages were calculated.

**Determination of Minimum Inhibition Concentration (MIC).** The MIC is the concentration of microorganisms that do not present visible growth with regard to the positive control. The MIC was determined for the synthesized heterocycles based on a reported procedure.\(^{36}\)

**Crystal Structure Determination.** Crystal and structure refinement parameters are shown in Table 2.

**Table 2.** Crystal data and structure refinement data

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<td>30099</td>
<td>28147</td>
<td>14807</td>
<td>14436</td>
</tr>
<tr>
<td>Ind. refs</td>
<td>C_{17}H_{16}N_{3}O_{2}</td>
<td>2362</td>
<td>8629</td>
<td>3444</td>
<td>4178</td>
<td>3920</td>
</tr>
<tr>
<td>R(int)</td>
<td>C_{17}H_{16}N_{3}O_{2}</td>
<td>0.0295</td>
<td>0.0456</td>
<td>0.0430</td>
<td>0.0249</td>
<td>0.0286</td>
</tr>
<tr>
<td>Parameters</td>
<td>C_{17}H_{16}N_{3}O_{2}</td>
<td>137</td>
<td>435</td>
<td>230</td>
<td>245</td>
<td>210</td>
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<tr>
<td>Goodness-of-fit</td>
<td>C_{17}H_{16}N_{3}O_{2}</td>
<td>1.145</td>
<td>1.029</td>
<td>1.066</td>
<td>1.076</td>
<td>1.028</td>
</tr>
<tr>
<td>R1 (I&gt;2σ(I))</td>
<td>C_{17}H_{16}N_{3}O_{2}</td>
<td>0.0603</td>
<td>0.0459</td>
<td>0.0511</td>
<td>0.0623</td>
<td>0.0545</td>
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</table>
Single-crystal XRD data were collected at rt on an Agilent SuperNova Dual Atlas diffractometer with a mirror monochromator using Mo or Cu radiation. The crystal structures were solved by SHELXS\textsuperscript{37} and refined using SHELXL.\textsuperscript{38} Non-hydrogen atoms were refined with anisotropic displacement parameters. Hydrogen atoms were inserted in idealized positions, and a riding model was used with Uiso set at 1.2 or 1.5 times the value of Ueq for the atom to which they are bonded. The crystal structures have been deposited in the Cambridge Structural Database under reference CCDC 2172761-2172766.

ACKNOWLEDGEMENTS

B. F. Abdel-Wahab and H. A. Mohamed; thank the National Research Center, Dokki, Giza, Egypt for support. G. A. El-Hiti thanks the Researchers Supporting Project number (RSP-2021/404), King Saud University, Riyadh, Saudi Arabia.

REFERENCES


