Arabian Journal of Chemistry (2014) 7, 604–608

King Saud University

Arabian Journal of Chemistry

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ORIGINAL ARTICLE

*1st Heterocyclic Update*

# Heterocyclic synthesis using nitrilimines: Part 19. Synthesis of novel 1,3,5-trisubstituted-1,2,4-triazoles

**Hany M.M. Dalloul**

*Department of Chemistry, Faculty of Applied Science, Alaqsa University of Gaza, P.O. Box 4051, Gaza 76888, Palestine*

Received 6 September 2011; accepted 28 February 2012
Available online 3 March 2012

**KEYWORDS**

Nitrilimine;
1,3-Dipolar cycloaddition;
Guanidine;
1,2,4-Triazoles

**Abstract** This paper describes the synthesis of a new series of 1,3,5-trisubstituted-1,2,4-triazoles by 1,3-dipolar cycloaddition reaction of *C*-phenyl-aminocarbonyl-*N*-arylnitrilimines with guanidine derivatives. The structures of the newly synthesized compounds were elucidated by spectral methods (IR, $^{1}$H NMR, $^{13}$C NMR and MS spectroscopy) and elemental analysis. The microbial features of the synthesized compounds were studied using well-established methods from the literature.


## 1. Introduction

A literature survey revealed azole derivatives belonging to an important group of heterocyclic compounds that have a long history in pharmaceutical and medicinal chemistry. In particular, triazoles represent a class of heterocyclic compounds with a wide variety of biological activities (Dogan et al., 2005; Amir and Kumar, 2007; Tozkoparan et al., 2007; Ezabadi et al., 2008; Küçükgüzel et al., 2008; Sakac et al., 2009; Sun et al., 2010; Jyothi et al., 2010; Rama et al., 2010). Furthermore, fused heterocyclic compounds containing a 1,2,4-triazole nucleus have a broad spectrum of pharmacological activities, including anti-inflammatory (Husain and Naseer, 2011; Aytac et al., 2009), analgesic (Aytac et al., 2009), ulcerogenic (Amir et al., 2008; Reddy et al., 2010), antimicrobial (Amir et al., 2008; Reddy et al., 2010; Sztanke et al., 2008), anticancer (Sztanke et al., 2008; Badr and Barwa, 2011), antiproliferative and apoptotic properties (Sztanke et al., 2008). The synthesis of compounds whose structure contains 1,2,4-triazole rings has attracted widespread attention. 1,3-Dipolar cycloaddition is one of the most versatile methods for the construction of five-membered heterocycles (Padwa, 1991). Recently, we have described a versatile and efficient one-pot synthesis of dispiroheterocycles containing 1,2,4-triazole moieties utilizing available keto oximes, hydrazones, and hydrazonoyl halides (Dalloul and Abu Samaha, 2010). Considering the promising opportunities that the synthesis of such heterocycles might open with regard to the production of biologically active nitrogen, the present study represents a continuation of our previous work in an attempt to search for and synthesize a biologically active nitrogen heterocycle (Yuksek et al., 1997;

E-mail address: hmdalloul60@yahoo.com
Peer review under responsibility of King Saud University.

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http://dx.doi.org/10.1016/j.arabjc.2012.02.011
1878-5352 

**Table 1** Physical data, molecular ion peaks and elemental analysis for compounds **5a–j**.

| Cpd. No. | Mol. Formula (MW) | Yield: mg (%) | Mp. ± 2 (°C) | Analysis (%), Calcd./Found | | | [M$^+$] |
|---|---|---|---|---|---|---|---|
| | | | | C | H | N | |
| **5a** | $C_{15}H_{13}N_5O$ (279.30) | 0.0021 (76) | 173 | 64.51 (64.75) | 4.69 (4.80) | 25.07 (24.95) | 279 |
| **5b** | $C_{15}H_{12}ClN_5O$ (313.75) | 0.0022 (72) | 184 | 57.42 (57.60) | 3.86 (3.70) | 22.32 (22.45) | 313/315 |
| **5c** | $C_{15}H_{12}BrN_5O$ (358.20) | 0.0028 (78) | 163 | 50.30 (50.55) | 3.38 (3.20) | 19.55 (19.65) | 358/360 |
| **5d** | $C_{15}H_{12}FN_5O$ (297.29) | 0.0021 (73) | 171 | 60.60 (60.35) | 4.07 (3.90) | 23.56 (23.40) | 297/299 |
| **5e** | $C_{16}H_{15}N_5O$ (293.33) | 0.0022 (75) | 189 | 65.52 (65.75) | 5.15 (5.00) | 23.88 (24.00) | 293 |
| **5f** | $C_{21}H_{17}N_5O$ (355.40) | 0.0025 (72) | 187 | 70.97 (70.75) | 4.82 (4.95) | 19.71 (19.55) | 355 |
| **5g** | $C_{21}H_{16}ClN_5O$ (389.85) | 0.0027 (71) | 212 | 64.70 (64.50) | 4.14 (4.30) | 17.96 (18.10) | 389/391 |
| **5h** | $C_{21}H_{16}BrN_5O$ (434.30) | 0.003 (70) | 193 | 58.08 (57.85) | 3.71 (3.60) | 16.13 (16.30) | 434/436 |
| **5i** | $C_{21}H_{16}FN_5O$ (373.39) | 0.0026 (69) | 188 | 67.55 (67.70) | 4.32 (4.20) | 18.76 (18.60) | 373/375 |
| **5j** | $C_{22}H_{19}N_5O$ (369.43) | 0.0023 (63) | 241 | 71.53 (71.30) | 5.18 (5.00) | 18.96 (19.15) | 369 |

Ikizler et al., 2000; Demirbas et al., 2002, 2005; Demirbas and Ugurluoglu, 2004; Bayrak et al., 2009). This paper reports on the synthesis of a series of some new substituted amino-1,2,4-triazoles via the reaction of *C*-phenylaminocarbonyl-*N*-arylnitrilimines with guanidine derivatives, and investigates the spectra of potential biological activities involved in the process.

## 2. Experimental

### *2.1. Material and instruments*

Melting points were taken in open capillary tubes on Gallenkamp apparatus and were uncorrected. Infrared spectra were obtained by means of a Pye Unicam SP-3000 infrared spectrophotometer using a KBr disk technique. The $^1$H NMR and $^{13}$C NMR spectra were measured on a Bruker AM 300 MHz spectrometer at room temperature in $CDCl_3$ or DMSO-$d_6$ solution using tetramethylsilane (TMS) as the internal reference. Chemical shifts were recorded as $\delta$ values in parts per million (ppm) downfield from internal TMS. Electron impact (EI) mass spectra were run on a Shimadzu GCMS-QP1000 EX spectrometer at 70 eV. Elemental analyses were performed at Cairo University, Egypt, and the results agreed with the calculated values within experimental errors. The hydrazonoyl halides **1**(Frohberg et al., 2002) were prepared according to well-established procedures in the literature. Guanidine and diphenylguanidine hydrochloride, tetrahydrofuran (THF), and triethylamine were purchased from Avocado Research Chemicals, England, and used without further purification.

### *2.2. Synthesis of 1,3,5-trisubstituted-1,2,4-triazoles (**5a–j**)*

To a stirred solution of the hydrazonoyl halides (10 mmol) in THF (50 mL) was added a solution of guanidine hydrochloride derivatives (15 mmol) in methanol (30 ml). To the resulting reaction mixture, cooled in an ice-salt bath (−5–0 °C), was dropwise added triethylamine (50 mmol). After addition was complete, stirring was continued for 1 h at 0 °C, and then at room temperature over night. The solvent was removed under reduced pressure, and the residue was washed with water (50 mL) to remove triethylammonium salt. The resulting crude solid product was collected and recrystallized from methanol or ethanol to give the desired good yields of the products **5a–j**. The physical and analytical data of the title compounds are given in Table 1.

#### *2.2.1. 5-Amino-3-carbanlino-1-phenyl-1,2,4-triazole (**5a**)*

IR ($\nu$/cm$^{-1}$): 3430, 3428, 3352 ($NH_2$ and NH), 1660 (C═O), 1622, 1618 (C═N); $^1$H NMR ($\delta$/ppm): 9.20 (s, 1H, NH amide), 8.23 (s, 2H, $NH_2$), 7.60–7.10 (m, 10H, Ar-H); $^{13}$C NMR ($\delta$/ppm): 161.7 (C═O), 152.2, 150.8 (C═N), 140.9, 136.9, 129.6, 129.2, 128.7, 127.2, 125.2, 119.7 (C═C, Ar).

#### *2.2.2. 5-Amino-3-carbanlino-1-(4-chlorophenyl)-1,2,4-triazole (**5b**)*

IR ($\nu$/cm$^{-1}$): 3426, 3424, 3351 ($NH_2$ and NH), 1665 (C═O), 1625, 1621 (C═N); $^1$H NMR ($\delta$/ppm): 9.20 (s, 1H, NH amide), 8.31 (s, 2H, $NH_2$), 7.89 (d, $J$ = 8.6 Hz, 2H, Ar-H), 7.90–7.21 (m, 5H, Ar-H), 7.44 (d, $J$ = 8.6 Hz, 2H, Ar-H); $^{13}$C NMR ($\delta$/ppm): 161.9 (C═O), 153.5, 151.3 (C═N), 140.5, 138.8, 136.4, 132.5, 129.3, 129.1, 126.2, 116.2 (C═C, Ar).

#### *2.2.3. 5-Amino-1-(4-bromophenyl)-3-carbanlino-1,2,4-triazole (**5c**)*

IR ($\nu$/cm$^{-1}$): 3434, 3431, 3348 ($NH_2$ and NH), 1663 (C═O), 1620, 1616 (C═N); $^1$H NMR ($\delta$/ppm): 9.20 (s, 1H, NH amide), 8.30 (s, 2H, $NH_2$), 7.84 (d, $J$ = 8.3 Hz, 2H, Ar-H), 7.74–7.16 (m, 5H, Ar-H) 7.50 (d, $J$ = 8.3 Hz, 2H, Ar-H); $^{13}$C NMR ($\delta$/ppm): 161.7 (C═O), 152.4, 150.9 (C═N), 140.3, 137.5, 131.2, 128.7, 126.6, 124.5, 120.8, 118.5 (C═C, Ar).

*2.2.4. 5-Amino-3-carbanlino-1-(4-fluorophenyl)-1,2,4-triazole (**5d**)*

IR ($\nu$/cm$^{-1}$): 3422, 3418, 3350 ($NH_2$ and NH), 1660 (C═O), 1626, 1622 (C═N); $^1$H NMR ($\delta$/ppm): 9.20 (s, 1H, NH amide), 8.38 (s, 2H, $NH_2$), 7.98 (d, $J$ = 8.7 Hz, 2H, Ar-H), 7.87–7.22 (m, 5H, Ar-H), 7.54 (d, $J$ = 8.7 Hz, 2H, Ar-H); $^{13}$C NMR ($\delta$/ppm): 162.3 (C═O), 153.6, 151.4 (C═N), 141.0, 139.9, 137.5, 136.8, 128.7, 126.5, 120.9, 115.7 (C═C, Ar).

*2.2.5. 5-Amino-3-carbanlino-1-(4-methylphenyl)-1,2,4-triazole (**5e**)*

IR ($\nu$/cm$^{-1}$): 3428, 3425, 3345 ($NH_2$ and NH), 1665 (C═O), 1621, 1612 (C═N); $^1$H NMR ($\delta$/ppm): 9.20 (s, 1H, NH amide), 8.20 (s, 2H, $NH_2$), 7.44 (d, $J$ = 7.6 Hz, 2H, Ar-H), 7.62–6.86 (m, 5H, Ar-H), 7.44 (d, $J$ = 7.6 Hz, 2H, Ar-H), 2.73 (s, 3H, $CH_3$); $^{13}$C NMR ($\delta$/ppm): 161.2 (C═O), 152.2, 150.1 (C═N), 140.9, 136.9, 129.5, 129.2, 128.8, 127.3, 125.1, 119.8 (C═C, Ar), 21.7 ($CH_3$).

*2.2.6. 3-Carbanlino-1-phenyl-5-phenylmino-1,2,4-triazole (**5f**)*

IR ($\nu$/cm$^{-1}$): 3420, 3349 (N–H), 1655 (C═O), 1615, 1608 (C═N); $^1$H NMR ($\delta$/ppm): 9.20 (s, 1H, NH amide), 8.50 (s, 1H, NH), 7.70–7.20 (m, 15H, Ar-H); $^{13}$C NMR ($\delta$/ppm): 161.6 (C═O), 153.4, 151.8 (C═N), 141.4, 138.6, 138.1, 136.3, 132.6, 129.9, 129.3, 128.9, 128.7, 127.3, 125.1, 119.8 (C═C, Ar).

*2.2.7. 3-Carbanlino-1-(4-chlorophenyl)-5-phenylmino-1,2,4-triazole (**5g**)*

IR ($\nu$/cm$^{-1}$): 3418, 3344 (N–H), 1652 (C═O), 1620, 1610 (C═N); $^1$H NMR ($\delta$/ppm): 9.20 (s, 1H, NH amide), 8.54 (s, 2H, NH), 7.83 (d, $J$ = 8.5 Hz, 2H, Ar-H), 7.78–7.61 (m, 10H, Ar-H), 7.42 (d, $J$ = 8.5 Hz, 2H, Ar-H); $^{13}$C NMR ($\delta$/ppm): 161.6 (C═O), 153.7, 151.6 (C═N), 140.7, 138.7, 138.1, 136.4, 132.6, 129.3, 129.1, 128.9, 127.6, 126.3, 125.1, 119.8 (C═C, Ar).

*2.2.8. 1-(4-Bromophenyl)-3-carbanlino-5-phenylmino-1,2,4-triazole (**5h**)*

IR ($\nu$/cm$^{-1}$): 3421, 3347 (N–H), 1657 (C═O), 1618, 1604 (C═N); $^1$H NMR ($\delta$/ppm): 9.20 (s, 1H, NH amide), 8.52 (s, 1H, NH), 7.85 (d, $J$ = 8.1 Hz, 2H, Ar-H), 7.66–7.16 (m, 10H, Ar-H), 7.52 (d, $J$ = 8.0 Hz, 2H, Ar-H); $^{13}$C NMR ($\delta$/ppm): 161.7 (C═O), 152.8, 150.7 (C═N), 140.6, 139.6, 137.5, 135.1, 131.3, 128.8, 128.6, 127.8, 126.6, 124.6, 120.8, 118.7 (C═C, Ar).

*2.2.9. 3-Carbanlino-1-(4-fluorophenyl)-5-phenylmino-1,2,4-triazole (**5i**)*

IR ($\nu$/cm$^{-1}$): 3424, 3345 (NH), 1655 (C═O), 1622, 1612 (C═N); $^1$H NMR ($\delta$/ppm): 9.20 (s, 1H, NH amide), 8.58 (s, 1H, NH), 7.90 (d, $J$ = 8.8 Hz, 2H, Ar-H), 7.86–7.26 (m, 10H, Ar-H), 7.56 (d, $J$ = 8.8 Hz, 2H, Ar-H); $^{13}$C NMR ($\delta$/ppm): 161.9 (C═O), 153.8, 151.9 (C═N), 140.9, 139.9, 137.5, 136.9, 128.8, 128.6, 128.2, 127.0, 126.6, 124.6, 121.0, 115.4 (C═C, Ar).

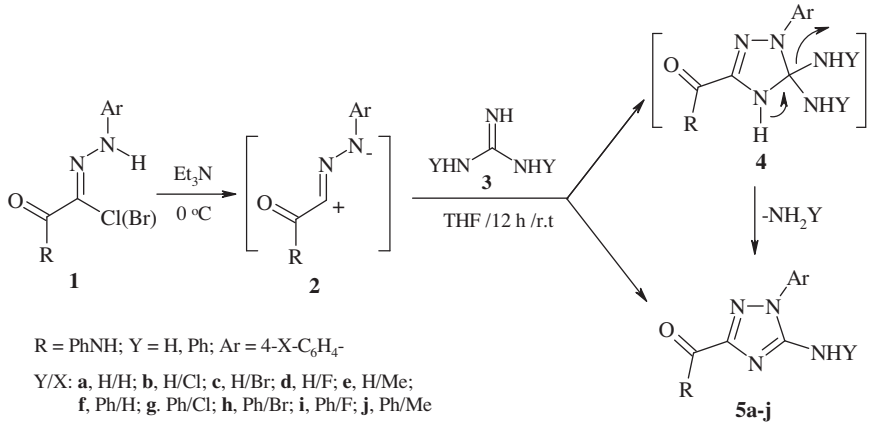
*2.2.10. 3-Carbanlino-1-(4-methylphenyl)-5-phenylmino-1,2,4-triazole (**5j**)*

IR ($\nu$/cm$^{-1}$): 3422, 3343 (NH), 1650 (C═O), 1617, 1608 (C═N); $^1$H NMR ($\delta$/ppm): 9.20 (s, 1H, NH amide), 8.55 (s, 1H, NH), 7.44 (d, $J$ = 7.4 Hz, 2H, Ar-H), 7.73–6.93 (m, 10H, Ar-H), 7.44 (d, $J$ = 7.4 Hz, 2H, Ar-H), 2.72 (s, 3H, $CH_3$); $^{13}$C NMR ($\delta$/ppm): 160.9 (C═O), 152.6, 150.5 (C═N), 141.1, 138.6, 138.1, 136.6, 132.6, 129.4, 129.3, 129.1, 128.7, 126.6, 125.1, 119.3 (C═C, Ar), 21.7 ($CH_3$).

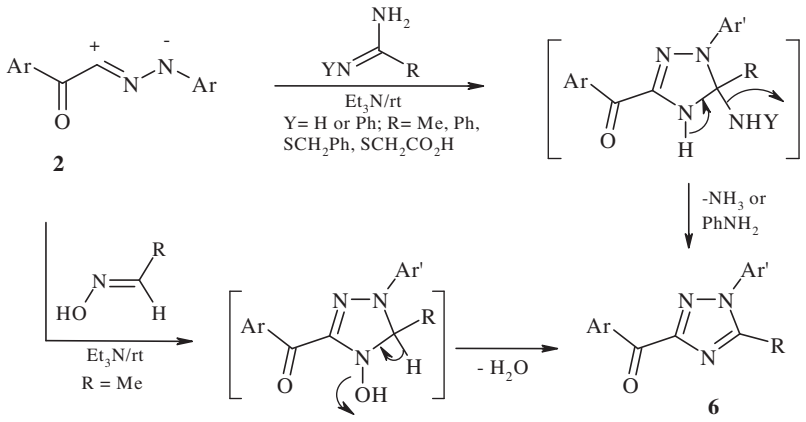
## 3. Results and discussion

The generation of the nitrilimine intermediate **2** was accomplished *in situ* by treatment of the corresponding hydrazonoyl chloride **1** with triethylamine in the presence of the dipolarophile, guanidine and diphenylguanidine hydrochloride **3**. The reaction led to the formation of 1,3,5-trisubstituted-1,2,4-triazole derivatives **5a–j** (Scheme 1). Both analytical and spectroscopic data (IR, $^1$H NMR, $^{13}$C NMR and MS) of the synthesized compounds were in full agreement with the proposed structures.

The formation of compounds (**5a–j**) is assumed to involve the formation of 5,5-diamino-1,2,4-triazoles **4** (Scheme 1), through nucleophilic addition of the electron pair of the imino group of guanidine then cyclization at the imine carbon, or by cycloaddition onto C═N of the guanidine moiety or (group).




**Scheme 1** Synthetic pathway for the preparation of compounds **5a–j**.




**Scheme 2** Synthetic pathway substituted triazoles **6**.

**Table 2** Antimicrobial screening results of the tested compounds.*

| Comp. No. | Antibacterial activity | | | Antifungal activity | |
|---|---|---|---|---|---|
| | *Euterococci* | *Escherichia coli* | *Staphylococcus aureus* | *Candida albicans* | *Aspergillus niger* |
| **5a** | 16 | 17 | 16 | 15 | 14 |
| **5b** | 17 | 18 | 16 | 17 | 18 |
| **5c** | 14 | 15 | 13 | 18 | 16 |
| **5d** | 19 | 18 | 17 | 19 | 12 |
| **5e** | 16 | 19 | 16 | 17 | 11 |
| **5g** | 15 | 12 | 14 | 16 | 9 |
| **5h** | 16 | 17 | 17 | 15 | 14 |
| **5i** | 19 | 18 | 19 | 17 | 18 |
| DMF | – | – | – | – | – |

* Zone of inhibition in mm.

The intermediate **4** cannot be isolated nor observed by TLC, ultimately undergoes the elimination of ammonia or aniline molecule yielding the aromatic 1,2,4-triazole derivatives **5a–j** as outlined in the Scheme 1.

It is worth mentioning that the same nitrilimines 2 are found to react with acetamidine, benzamidine, benzylthioformamidine, and acetaldoxime in cycloaddition processes to give 1,3,5-trisubstituted 1,2,4-triazoles **6** through the elimination of ammonia or water molecules as shown in Scheme 2 (Dalloul, 2009).

### *3.1. Spectral data analysis*

The assignment of structures of compounds **5a–j** is based on their analytical and spectroscopic data. Physical properties, molecular ion peaks and microanalysis are presented in Table 1. The electron impact (EI) mass spectra of these compounds **5a–j** displayed the correct molecular ions ($M^+$) in accordance with the suggested structures (Table 1). Their IR spectra showed strong absorption bands of NH in the region 3430–3340 $cm^{-1}$, in addition to, the characteristic band of amide C=O at about 1660–1650 $cm^{-1}$ and C=N of triazole ring in the region of 1620–1600 $cm^{-1}$. The $^1H$ NMR confirmed the formation of compounds **5a–j** their spectra showed, in addition to aromatic protons signals, a characteristic signal due to the NH proton at C-5 of the ring resonating as a singlet at 8.6–8.2 ppm and the amide NH appeared as a singlet in the range of 8.9–8.8 ppm. The structures of compounds **5a–j** were further confirmed by $^{13}C$ NMR spectra, which account for the different carbons of these triazoles. The signals at about 153–151 ppm were attributed to the C-3 and C-5 carbons of the triazole ring, and are in accordance with reported values of azo-methine carbons in five-membered heterocycles (Dalloul et al., 2008; Dalloul, 2009). The $^1H$ and $^{13}C$ NMR spectral data of the synthesized compounds are presented in the experimental part.

### *3.2. Antimicrobial activity*

Most of the newly synthesized compounds were tested for their antibacterial and antifungal activities in vitro against bacterial strains such as *Escherichia coli, Staphylococcus aureus*, *Pseudomonas aeruginosa* and *Aspergillus flavus*, and *Candida albicans* as fungi. The compounds tested at a concentration of 1–10 mg/mL in *N*,*N*-dimethylformamide (DMF) solution, using the nutrient agar disc diffusion method (Collins et al., 1989) and measuring the average diameter of the inhibition zone in mm. The results showed that all the tested compounds exhibited a marked degree of activity against bacteria and fungi compared with well known antibacterial and antifungal substances such as tetracycline and fluconazole. According to NCCLS (2004), zones of inhibition for tetracycline and fluconazole $< 14$ mm were considered resistant, between 15 and 18 mm were considered weakly sensitive and $> 19$ mm

were considered sensitive. Also, the results showed the degree of inhibition varied with the tested compounds (Table 2).

## 4. Conclusion

In conclusion, the reaction of nitrilimines with guanidine and diphenyl-guanidine hydrochloride leads to the formation of aromatic heterocyclic triazoles in one step, and some of them proved to have potent antibacterial and antifungal activities. The results confirm that the antimicrobial activity is strongly dependent on the nature of the substituents at the triazole ring.

## Acknowledgments

The author is thankful to the UAU, Supporting Box of Palestinian Universities, Amman, Jordan, for partial finance support and to Dr. A. S. Abu Samaha for providing the antimicrobial testing facility for the synthesized compounds.

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