Journal of Molecular Structure 1067 (2014) 83–87

Contents lists available at ScienceDirect

Journal of Molecular Structure

journal homepage: www.elsevier.com/locate/molstruc

Synthesis, spectroscopic and structural studies of 4-Amino-3-(ethyl)-5-(4-chlorophenyl)-4H-1,2,4-triazole and 4-Amino-3-cyclopropyl-5-oxo-4,5-dihydro-1,2,4-triazole-1-yl-acetic acid ethyl ester

Onur Şahin [a,*], Cihan Kantar [b], Selami Şaşmaz [b], Nurhan Gümrükçüoğlu [c], Orhan Büyükgüngör [d]

[a] Scientific and Technological Research Application and Research Center, Sinop University, 57010 Sinop, Turkey
[b] Deparment of Chemistry, Faculty of Arts and Sciences, Recep Tayyip Erdoğan University, 53150 Rize, Turkey
[c] Vocational School of Health Services, Karadeniz Technical University, 61080 Trabzon, Turkey
[d] Department of Physics, Faculty of Arts and Sciences, Ondokuz Mayıs University, 55139 Samsun, Turkey

HIGHLIGHTS

- We report the synthesis of two new triazole derivatives.
- The crystallographic calculations were performed by X-ray diffraction technique.
- The compounds have been characterized by IR, ^{1}H NMR and ^{13}C NMR.

ARTICLE INFO

Article history:
Received 31 December 2013
Received in revised form 13 March 2014
Accepted 13 March 2014
Available online 20 March 2014

Keywords:
Synthesis
Crystal structures
Triazole
Hydrogen bonds

ABSTRACT

The title compounds of 4-Amino-3-(ethyl)-5-(4-chlorophenyl)-4H-1,2,4-triazole (**I**, $C_{10}H_{11}ClN_4$) and 4-Amino-3-cyclopropyl-5-oxo-4,5-dihydro-1,2,4-triazol-1-yl-acetic acid ethyl ester (**II**, $C_9H_{14}N_4O_3$), have been determined using X-ray diffraction techniques and characterized by IR, ^{1}H NMR and ^{13}C NMR. X-ray study shows that the title compounds both have strong intermolecular N—H···N hydrogen bonds. The molecules of **I** are linked into a two-dimensional framework structure by N—H···N and C—H···N hydrogen bonds which produce $R_2^2(8)R_4^4(14)R_4^4(15)$ chain of rings, while in **II**, the combination of N—H···N, N—H···O and C—H···O hydrogen bonds along [100] generates a chain of edge-fused $R_2^2(10)R_2^2(16)R_4^2(14)$ $R_4^4(14)$ rings.

© 2014 Elsevier B.V. All rights reserved.

Introduction

The derivatives of 1,2,4-triazole have been used as starting materials for the synthesis of many heterocycles [1–4]. Triazole compounds have many applications in medicine and were reported to exhibit various pharmacological activities such as antiviral, antimicrobial, analgesic, anti-inflammatory, anticancer and antioxidant properties [5–8]. Some of them are also known to exhibit analgesic, anticonvulsant, tranquilizing, antidepressant, anxiolytic [9–12] or even antitumour activities [13] and are applied in therapy (e.g. Alprazolam, Estazolam, Triazolam and Adinazolam) [14]. In the present paper, we wish to report the synthesis, spectroscopic and structural studies of the two new triazole compounds (**I** and **II**) (see Table 1).

Experimental

Synthesis

Synthesis of Ethyl N-(4-chlorobenzoyl)propanhydrazonoate (3)

To the solution of ethyl iminoethylester hydrochloride (**1**) in absolute ethanol (10 mmol) was added the solution of 4-cholorobenzoicacid hydrazide (10 mmol) in absolute ethanol and the mixture was stirred at 0–5 °C for 6 h. Then the precipitated ammonium chloride was filtered off. After the solvent was evaporated at 35–40 °C under reduced pressure, a white solid appeared. This crude product was recrystallized from benzene–petroleum ether (1:2) to afford compound **3**. (Yield: 52%). mp 113–114 °C; IR (KBr) cm^{-1}: 3270 (ν_{NH}), 1668 ($\nu_{C=O}$), 1621 ($\nu_{C=N}$); ^{1}H NMR (DMSO-d6) δ ppm 1.28 (t, 3H, CH_3, J = 5.08 Hz), 1.40 (t, 3H, OCH_2—$\underline{CH_3}$, J = 6.96 Hz), 2.48 (q, 2H, CH_2, J = 5.08 Hz), 4.22 (q, 2H, OCH_2, J = 6.96 Hz), [ar-H:7.56 (d, 2H, J = 7.20 Hz), 8.06 (d, 2H, J = 7.20 Hz)], 10.28 (s, 1H, NH); ^{13}C NMR (DMSO-d6) δ ppm:

* Corresponding author. Tel.: +90 03682715644.
E-mail address: onurs@sinop.edu.tr (O. Şahin).

http://dx.doi.org/10.1016/j.molstruc.2014.03.022
0022-2860/© 2014 Elsevier B.V. All rights reserved.

Table 1
Parameters for data collection and structure refinement of **I** and **II**.

Crystal data	I	II
Empirical formula	$C_{10}H_{11}N_4Cl$	$C_9H_{14}N_4O_3$
Formula weight	222.68	226.24
Crystal system	orthorhombic	triclinic
Space group	*Pbca*	*P–1*
a (Å)	7.9240 (5)	6.2727 (4)
b (Å)	26.785 (2)	8.6161 (6)
c (Å)	10.3141 (7)	10.9899 (8)
α (°)	90.00	80.210 (6)
β (°)	90.00	83.224 (5)
γ (°)	90.00	73.387 (6)
V (Å^3)	2189.1 (3)	559.37 (7)
Z	8	2
D_c (g cm^{-3})	1.351	1.343
μ (mm^{-1})	0.32	0.10
θ range (°)	1.5–26.7	1.9–28.0
Measured refls.	16,644	9294
Independent refls.	2193	2545
R_{int}	0.073	0.099
S	1.04	1.06
R_1/wR_2	0.056/0.125	0.046/0.105
$\Delta\rho_{max}/\Delta\rho_{min}$ (e Å^{-3})	0.13/−0.18	0.27/−0.19

170.00 (C=O), 160.05 (C=N), ar-C: [135.43 (C), 134.05 (C), 128.00 (2CH), 127.52 (2CH)], 53.15 (OCH_2), 28.87 (CH_2), 20.05 (OCH_2—$\underline{CH_3}$), 13.86 (CH_3).

*Synthesis of 4-Amino-3-(ethyl)-5-(4-chlorophenyl)-4H-1,2,4-triazole (**I**)*

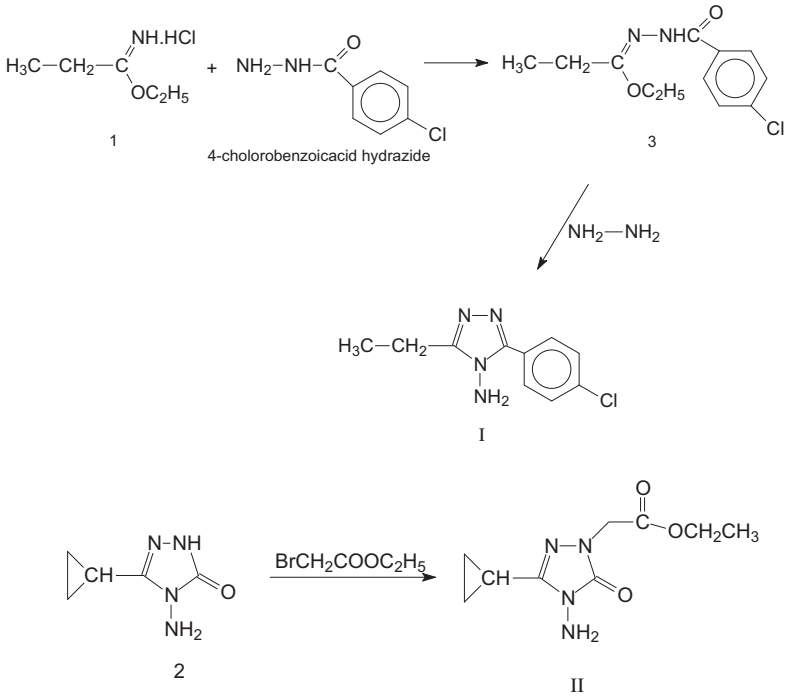
A solution of the corresponding compound **3** (10 mmol) in n-propanol was refluxed with hydrazine hydrate (25 mmol) for 24 h. After it was cooled to room temperature, a white solid appeared. This crude product was filtered off, washed with benzene 3 times, and recrystallized from an ethanol to afford the desired compound (**I**). Single crystals of compound (**I**) (Scheme 1) were obtained from ethyl acetate at room temperature by slow evaporation. (Yield: 64%). mp 191–192 °C; IR (KBr) cm^{-1}: 3320–3275 (νNH_2), 1652 (νC=N); ^{1}H NMR (DMSO-d6) δ ppm 1.38 (t, 3H, CH_3, *J* = 5.08 Hz), 2.80 (q, 2H, CH_2, *J* = 5.08 Hz), 5.22 (s, 2H, NH_2); [ar-H: 7.46 (d, 2H, *J* = 7.20 Hz), 8.55 (d, 2H, *J* = 7.20 Hz)], ^{13}C NMR (DMSO-d6) δ ppm 155.00 (triazole C_5), 153.00 (triazole C_3), ar-C: [149.90 (2CH), 136.05 (C), 134.00 (2CH), 129.32 (C)], 20.83 (CH_2), 12.68 (CH_3). MS: *m*/*z* 222.78 (M+1).

*Synthesis of 4-Amino-3-cyclopropyl-5-oxo-4,5-dihydro-1,2,4-triazol-1-yl-acetic acid ethyl ester (**II**)*

The corresponding 3-cyclopropyl-4-amino-5-oxo-4,5-dihydro-1,2,4-triazol (**2**) (0.01 mol) was refluxed with equivalent amount of sodium metal in absolute ethanol for 2 h. Then, ethylbromoacetate (0.01 mol) was added and the mixture was refluxed for additional 5 h. After evaporation at 35–40° in a vacuum, the resulting solid was recrystallized from an ethanol. Single crystals of compound (**II**) (Scheme 1) were obtained from ethyl acetate at room temperature by slow evaporation. (Yield: 61.20%). mp 155–156 °C; IR(KBr) cm^{-1}: 3298–3210 (νNH_2), 1750 (ester—C=O), 1706 (triazole—C=O), 1611 (νC=N), 1215 (νC—O); ^{1}H NMR (DMSO-d6) δ ppm: 1.16 (t, 3H, —$OCH_2\underline{CH_3}$, *J* = 6.96 Hz), 1.80–1.87 (m, 4H, CH_2), 3.43 (m, 1H, CH), 4.10 (q, 2H, —$O\underline{CH_2}CH_3$, *J* = 6.96 Hz), 4.52 (s, 2H, —NCH_2), 5.36 (s, 2H, —NH_2); ^{13}C NMR (DMSO-d6) δ ppm: 167.48 (exocyclic—C=O), 154.18 (triazole C_5), 146.04 (triazole C_3), 61.20 (—$O\underline{CH_2}CH_3$), 53.18 (CH), 46.67 (—NCH_2), 28.12 (CH_2), 28.08 (CH_2), 14.60 (—$OCH_2\underline{CH_3}$). MS: *m*/*z* 226.92 (M+1).

Instrumentation

Melting points were determined on a Gallenkampmelting point apparatus and are uncorrected. ^{1}H NMR and ^{13}C NMR spectra were recorded on a Varian-Mercury 200 MHz spectrometer. The IR spectra were measured as potassium bromide pellets using a Perkin–Elmer 1600 series FTIR spectrometer. Mass spectra were performed on Thermo TSQ Quantum acces max. All the chemicals were obtained from Fluka Chemie AG Buchs. Ethyl iminoethylester



Scheme 1. Synthesis of compounds **I**–**II**.

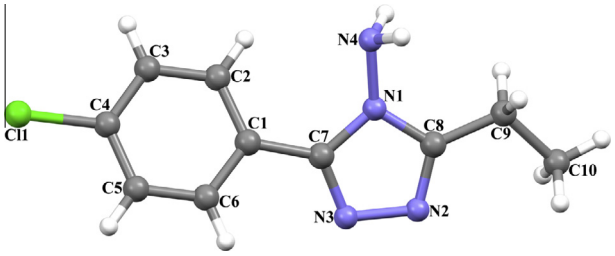


Fig. 1. Structure **I** showing the atom numbering scheme.

hydrochloride (**1**) and 3-cyclopropil-4-amino-5-oxo-4,5-dihydro-1,2,4-triazol (**2**) were obtained as reported earlier [15–17].

Crystal structure determination

Crystals of **I** and **II** suitable for data collection were selected and data collection was performed on a STOE IPDS II diffractometer with graphite monochromated Mo Kα radiation at 296 K. The structures were solved by direct-methods using SHELXS-97 and refined by full-matrix least-squares methods on F^2 using SHELXL-97 [18] from within the WINGX [19] suite of software. All non-hydrogen atoms were refined with anisotropic parameters. The H atoms of nitrogen atoms were located in difference maps and refined freely. Hydrogen atoms bonded to carbon were placed in calculated positions (C—H = 0.93–0.98 Å) and treated using a riding model with U = 1.2 times the U value of the parent atom for CH, CH_2 and CH_3. Molecular diagrams were created using MERCURY [20]. Geometric calculations were performed with PLATON [21].

Results and discussion

Description of the crystal structures

The molecular structure of **I** with the atom labeling is shown in Fig. 1. The dihedral angle between the benzene and triazole rings is 22.66(12)°. The N—N bond distances are 1.397(3) Å and 1.412(3) Å, respectively. These values are comparable to those observed in other similar triazole complexes [22–27]. The benzene and triazole rings plane are approximately planar, with maximum deviation from the least-squares plane being 0.0074(25) Å for atom C4 and 0.0045(14) Å for atom N1. Molecules of (**I**) are linked into sheets by a combination of N—H···N and C—H···N hydrogen bonds. Amino atom N4 in the molecule at (x,y,z) acts as hydrogen-bond donor, via atom H12, respectively, to atom N3 at $(x-1/2,y,-z+3/2)$, so forming a C(5) zig-zag chain running parallel to the [100] direction. Similarly, amino atom N4 in the molecule at (x,y,z) acts as hydrogen-bond donor, via atom H14, respectively, to atom N2 at $(x,-y+3/2,z-1/2)$, so forming a C(5) chain running parallel to the [001] direction. The combination of C—H···N hydrogen bonds and C(5) chains produce $R_2^2(8)R_4^4(14)R_4^4(15)$ chain of rings running parallel to the [101] direction (Fig. 2).

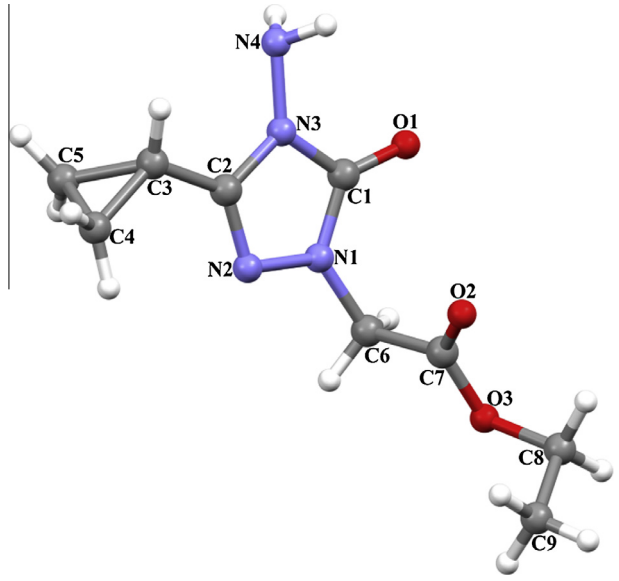


Fig. 3. The molecule of **II** showing the atom labeling scheme.

The molecular structure of **II** with the atom labeling is shown in Fig. 3. The dihedral angle between the cyclopropane and triazole rings is 76.46(8)°. The C1—O1, C7—O2 and N2—C2 bond lengths

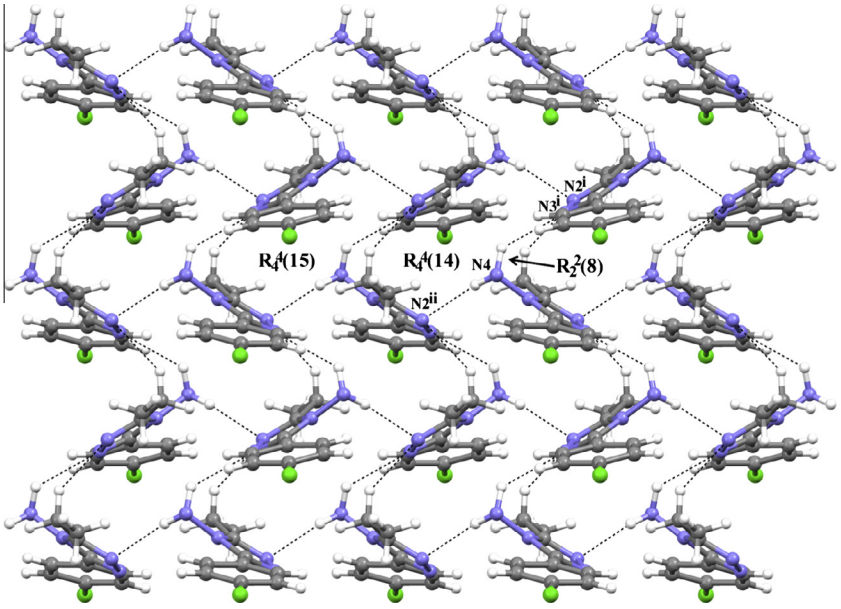


Fig. 2. The crystal structure of **I** showing the formation of $R_2^2(8)$, $R_4^4(14)$ and $R_4^4(15)$ rings along [101]. [Symmetry code as in Table 3].

Table 2
Selected bond lengths and angles (Å, °).

I			
Bond lengths (Å)			
C7—N3	1.311 (3)	C7—N1	1.367 (3)
C8—N2	1.305 (3)	C8—N1	1.361 (3)
N1—N4	1.412 (3)	N2—N3	1.397 (3)
Bond angles (°)			
C8—N1—C7	106.72 (19)	C8—N1—N4	126.9 (2)
C7—N1—N4	125.2 (2)	C8—N2—N3	107.6 (2)
C7—N3—N2	107.7 (2)		
II			
Bond lengths (Å)			
C1—O1	1.2310 (14)	C1—N1	1.3533 (14)
C1—N3	1.3779 (15)	C2—N2	1.2979 (14)
C2—N3	1.3747 (13)	C6—N1	1.4369 (14)
C7—O2	1.1861 (16)	C7—O3	1.3227 (14)
N1—N2	1.3939 (14)	N3—N4	1.3980 (13)
Bond angles (°)			
N1—C1—N3	102.92 (9)	N2—C2—N3	110.79 (10)
C1—N1—N2	112.65 (9)	C1—N1—C6	125.35 (10)
N2—N1—C6	121.34 (9)	C2—N2—N1	104.51 (9)
C2—N3—C1	109.02 (9)	C2—N3—N4	124.41 (9)
C1—N3—N4	126.53 (9)		

Table 3
Hydrogen-bond parameters (Å, °).

D—H···A	D—H	H···A	D···A	D—H···A
I				
N4—H12···N3^{i}	0.94 (4)	2.44 (4)	3.114 (4)	129 (3)
N4—H14···N2^{ii}	0.88 (3)	2.25 (3)	3.118 (4)	169 (3)
C9—H9A···N2^{i}	0.97	2.58	3.351 (3)	136
II				
N4—H4D···N2^{i}	0.91 (2)	2.355 (19)	3.1000 (14)	139.6 (16)
N4—H4C···O1^{ii}	0.95 (2)	2.02 (2)	2.9538 (16)	167.2 (16)
C3—H3···O2^{iii}	0.98	2.45	3.2382 (16)	138
C6—H6B···O1^{iv}	0.97	2.56	3.5084 (15)	166
C8—H8B···O1^{v}	0.97	2.59	3.4387 (18)	146

Symmetry codes: (i) $x-1/2, y, -z+3/2$; (ii) $x, -y+3/2, z-1/2$ for **I**; (i) $x+1, y, z$; (ii) $-x+2, -y, -z+1$; (iii) $-x+1, -y+1, -z+1$; (iv) $x-1, y, z$; (v) $-x+1, -y, -z+2$ for **II**.

are indicative of significant double-bond character (Table 2). In the cyclopropane ring, the C—C bond distances ranged between 1.476(2) and 1.5049(18) Å, while the C—C—C bond angles ranged between 58.94(9)° and 60.88(9)°. These values are comparable to those observed in other triazole complexes with cyclopropane ring [28,29]. The triazole ring plane is approximately planar, with maximum deviation from the least-squares plane being 0.0180(7) Å for atom N1. In **II**, molecules are linked into sheets by a combination of N—H···N, N—H···O and C—H···O hydrogen bonds. Amino atom N4 in the molecule at (x,y,z) acts as hydrogen-bond donor, via atom H4D, respectively, to atom N2 at $(x+1,y,z)$, so forming a C(5) chain running parallel to the [100] direction. Similarly, amino atom N4 in the molecule at (x,y,z) acts as hydrogen-bond donor, via atom H4C, respectively, to atom O1 at $(-x+2,-y,-z+1)$, so forming a centrosymmetric $R_2^2(10)$ ring centered at (1,0,1/2). The combination of N—H···N and N—H···O hydrogen bonds along [100] generates a chain of edge-fused $R_2^2(10)R_4^4(14)$ rings (Fig. 4). Atom C8 in the molecule at (x,y,z) acts as hydrogen-bond donor, via atom H8B, respectively, to atom O1 at $(-x+1,-y,-z+2)$, so forming a centrosymmetric $R_2^2(16)$ ring centered at $(n+1/2, 0, 1)$ (n = zero or integer). Atom C6 in the molecule at (x,y,z) acts as hydrogen-bond donor, via atom H6B, respectively, to atom O1 at $(x-1,y,z)$, so forming a C(5) chain running parallel to the [100] direction. The combination of C—H···O hydrogen bonds along [100] generates a chain of edge-fused $R_2^2(16)R_4^2(14)$ rings (Fig. 5).

Evaluation of spectroscopic results

In this study two new compounds **(I, II)** were synthesized. Their structures were established by IR, ^{1}H NMR, ^{13}C NMR and MS techniques.

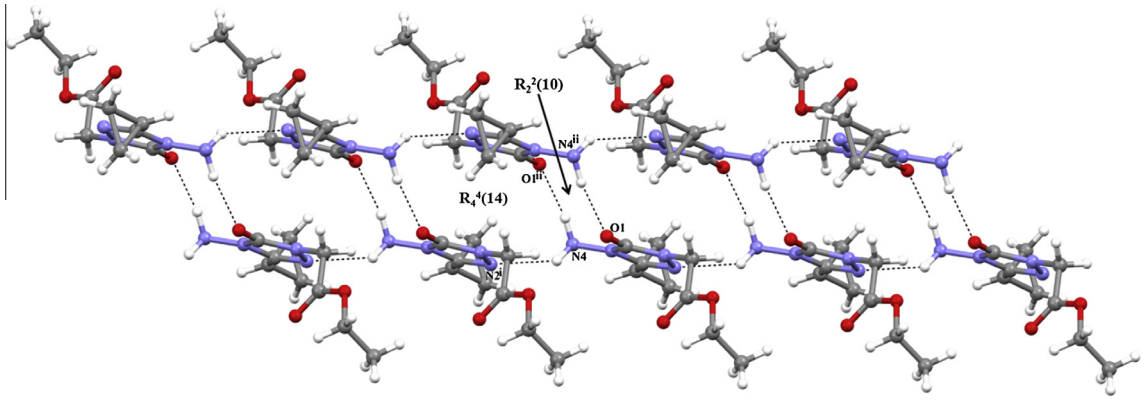


Fig. 4. The crystal structure of **II** showing the formation of an [100] chain of edge-fused $R_2^2(10)$ and $R_4^4(14)$ rings. [Symmetry code as in Table 3].

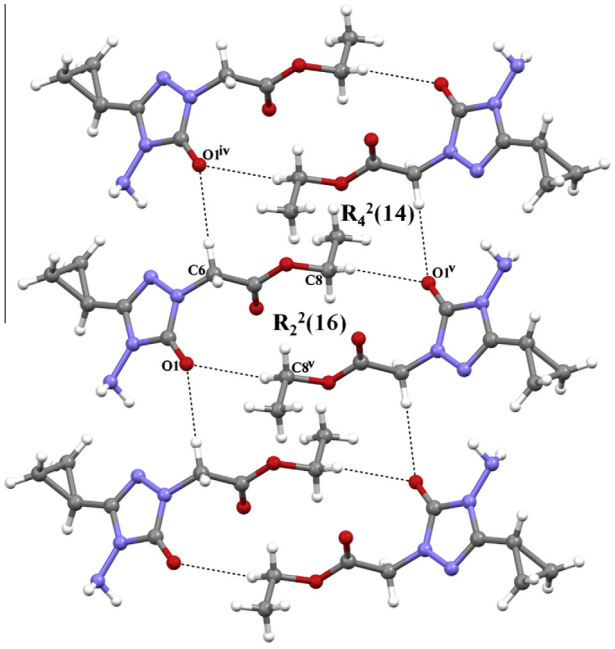


Fig. 5. The crystal structure of **II**, showing the formation of a chain rings along [100] generated by C—H···O hydrogen bonds. [Symmetry code as in Table 3].

In the first part of the study, compound **3** was synthesized from the reaction of corresponding iminoester hydrochloride **1** with acyl hydrazine which was obtained by a published method [1]. The formed acylhydrazone's structure was established by IR, ^{1}H NMR and ^{13}C NMR technigues. Compound **I** was obtained by treatment of compound **3** with hydrazine hydrate. The reaction was carried out in 1-propanol at refluxing temperature for 24 h and the desired 3-alkyl-4-amino-5-aryl-1,2-4-triazole was yielded.

The IR spectrum of compound **3** displayed no absorption derived from —NH_2 stretching. In the NMR spectra of this compound new signal originating from the —OEt group was recorded. In the IR spectra of compound **I**, the stretching band derived from the —NH_2 group was present. Beside this the signal due to the ethoxy group was absent in the NMR spectra of compound **I**, While the peak corresponding to the —NH_2 group was recorded at 5.22 ppm in the ^{1}H NMR spectra of this compound (exchangeable with D_2O).

In the second part of the study, compound **II** was synthesized via the nucleophylic attack of N-1 on 5-oxo-[1,2,4] triazole ring to bromine-bearing C atom of ethyl bromoacetate. This compound was obtained by a published method [7]. In the NMR spectra of this compound additional signals derived from ester group were observed at 4.52 (—NCH_2), 4.10 ppm —$O\underline{CH_2}CH_3$ and 1.16 ppm —$OCH_2\underline{CH_3}$ ppm integrating for two proton, two proton and three proton, respectively. ^{13}C NMR spectra of this compound, the signals belonging to the same groups were recorded at 46.67, 61.20, 14.60 ppm, respectively.

High resolution MS spectra (ESI) of compounds (**I–II**) provided a definitive proof for their characterization. Ionization took place in the methanol solution. Molecular ions peaks of compounds (**I–II**) were detected as expected. MS spectrum measurements confirmed unambiguously the molecular mass of compounds **(I)** (m/z = 222.78 M+1), **(II)** (m/z = 226.92 M+1).

Conclusions

Two new triazole derivatives were syhthesized and characterized by the IR, NMR, MS spectroscopy and X-ray diffraction method. All spectroscopic data of compounds show good agreement with the expected values. In addition the present study provides the bond angles, bond lengths and other crystallographic information of synthesized molecules. X-ray study shows that the compound **I** is extended into 2-D supramolecular network by N—H···N and C—H···N hydrogen bonds while the compound **II** is extended into 1-D supramolecular network by N—H···N, N—H···O and C—H···O hydrogen bonds.

Supplementary data

Crystallographic data for the structural analysis have been deposited with the Cambridge Crystallographic Data Centre, CCDC 929139 for compound **I** and 929140 for compound **II**. Copies of this information may be obtained free of charge from the Director, CCDC 12 Union Road, Cambridge CB2 1EZ, UK. (Fax: +44 1223 336 033 or e-mail: deposit@ccdc.cam.ac.uk).

References

[1] S.M. Desenko, Khim. Geterotsikl. Soedin. (Chem. Heterocycl. Comput.) (1995) 2–24.
[2] N. Gümrükçüoğlu, M. Serdar, E. Çelik, A. Sevim, N. Demirbaş, Turk. J. Chem. 31 (2007) 335–348.
[3] M. Serdar, N. Gümrükçüoğlu, Ş.A. Karaoğlu, N. Demirbaş, Turk. J. Chem. 31 (2007) 315–326.
[4] S. Fandaklı, S. Başoğlu, H. Bektaş, Turk. J. Chem. 36 (2012) 567–582.
[5] M. Amir, H. Kumar, S.A. Javed, Eur. J. Med. Chem. 43 (2008) 2056–2066.
[6] K. Sztanke, T. Tuzimski, J. Rzymowska, K. Pasternak, M. Kandefer-Szerszen, Eur. J. Med. Chem. 43 (2008) 404–419.
[7] V. Padmavathi, P. Thriveni, G.S. Reddy, D. Deepti, Eur. J. Med. Chem. 43 (2008) 917–924.
[8] M. Thenmozhi, T. Kavitha, B.P. Reddy, V. Vijayakumar, M.N. Ponnuswamy, Acta Cryst. E66 (2010) o558.
[9] J.M. Kane, M.W. Dudley, S.M. Sorensen, F.P. Miller, J. Med. Chem. 31 (1988) 1253–1258.
[10] J.K. Sughen, T. Yoloye, Pharm. Acta Helv. 58 (1978) 64–68.
[11] M.R. Stillings, A. Welbourn, D.S. Walter, J. Med. Chem. 29 (1986) 2280–2284.
[12] R.H. Bradbury, J.E. Rivett, J. Med. Chem. 34 (1991) 151–157.
[13] G.J. Hatheway, C. Hansch, K.H. Kim, S.R. Milstein, C.L. Schmidt, R.N. Smith, F.R. Quinn, J. Med. Chem. 21 (1978) 563–567.
[14] S. Budavari, M.J. O'Neil, A. Smith, P.E. Heckelman, J.F. Kinnearty (Eds.), The Merck Index, 12th ed., Entries 159, 320, 3744 and 9734, 1996.
[15] R. Un, A.A. Ikizler, Chem. Acta Turc. 7 (1979) 269–290.
[16] F. Malbec, R. Milcent, P. Vikard, J. Het. Chem. 21 (1984) 1769–1774.
[17] A.A. Ikizler, R. Un, Chem. Acta Turc. 7 (1979) 119–123.
[18] G.M. Sheldrick, Acta Cryst. A64 (2008) 112–122.
[19] L.J. Farrugia, J. Appl. Crystallogr. 32 (1999) 837–838.
[20] C.F. Macrae, I.J. Bruno, J.A. Chisholm, P.R. Edgington, P. McCabe, E. Pidcock, L. Rodriguez-Monge, R. Taylor, J. van de Streek, P.A. Wood, J. Appl. Crystallogr. 41 (2008) 466–470.
[21] A.L. Spek, PLATON – A multipurpose Crystallographic Tool, Utrecht University, Utrecht, 2005.
[22] O. Şahin, C. Kantar, O. Bekircan, S. Şaşmaz, O. Büyükgüngör, Chinese J. Struct. Chem. (Jiegou Huaxue) 30 (2011) 731–737.
[23] H.S. Yathirajan, B. Prabhuswamy, C.R. Raju, P. Nagaraja, M.N. Ponnuswamy, K. Palani, Anal. Sci. 21 (2005) x1–x2.
[24] J. Zachara, I. Madura, M. Wlostowski, Acta Cryst. C60 (2004) o57–o59.
[25] A. Aijaz, P. Lama, P.K. Bharadwaj, Inorg. Chem. 49 (2010) 5883–5889.
[26] O. Şahin, O. Büyükgüngör, S. Şaşmaz, N. Gümrükçüoğlu, C. Kantar, Acta Cryst. C62 (2006) o643–o646.
[27] G. Laus, V. Kahlenberg, D.M. Többens, R.K.R. Jetti, U.J. Griesser, J. Schütz, E. Kristeva, K. Wurst, H. Schottenberger, Cryst. Growth Des. 6 (2006) 404–410.
[28] K. Puviarasan, L. Govindasamy, S.S.S. Raj, D. Velmurugan, G. Jayanthi, H.K. Fun, Acta Cryst. C55 (1999) 1119–1121.
[29] A.U. Broceta, M.J. Pineda-de-las-Infantas, J.J.D. Mochon, R. Romagnoli, P.G. Baraldi, M.A. Gallo, A. Espinosa, J. Org. Chem. 70 (2005) 2878–2880.