SYNTHESIS AND X-RAY STRUCTURAL CHARACTERIZATION OF 1-(5-BROMOBENZOFURAN-2-YL)-2-MESITYLETHANONEOXIME AND 1-(5-BROMOBENZOFURAN-2-YL)-2-MESITYLETHANONE-O-(2- PHENYLACETYL)OXIME

GÜL YAKALI[a], ESRA BARIM[b], CUMHUR KIRILMIŞ[b,], MUHİTTİN AYGÜN[c,*]*

[a]*Department of Opticianry Program, Vocational School of Higher Education, Akdeniz University, 07070-Konyaaltı Antalya, Turkey.*
[b]*Department of Chemistry, Faculty of Science, Adıyaman University, 02040-Adıyaman, Turkey*
[c] *Department of Physics, Faculty of Sciences, Dokuz Eylül University, 35160-Buca, İzmir, Turkey*

ABSTRACT

The selected molecules of benzofuran derivatives were synthesized and their structures were studied using the X-ray crystallography and spectroscopic methods. The 1-(5-bromobenzofurane-2-yl)-2-mesitylethanoneoxime **(I)**, $C_{19}H_{18}BrNO_2$, and 1-(5-bromobenzofuran-2-yl)-2-mesitylethanone-*O*-(2-phenylacetyl) oxime **(II)**, $C_{27}H_{24}BrNO_3$, crystallize in the monoclinic crystal system in space group $P2_1/c$ with $Z = 4$ and in the triclinic system in space group P-1 with Z=2, respectively. The both compounds adopt Z conformation. The compound **(I)** consists of a dimeric arrangement of molecules around an inversion centre formed via a O-H…N intermolecular hydrogen bond linking the molecules – Along the *a* axis. This centrosymmetric hydrogen-bonded dimers are formed with an $R_2^2(6)$ ring motif. The compound **(II)** forms one dimensional infinite chain via C–H···O hydrogen bond along the *a* axis. Moreover, in the crystal structures of **(I), (II)** weak C-H..O, C-H…π and π…π interactions serve to organize formation of a two dimensional network. Moreover a short N1-N1 intermolecular contact [2.89 Å] is observed between inversion-related chains in the crystal structure of **(I)**. The two compounds have essentialy similar bond lengths and angles. The prominent discrepancy is observed for the fragment attaching benzofuran ring with mesityl group. Benzofuran groups and other ring groups are almost planar in the crystal structures of **(I)** and **(II)**.

Keywords: Benzofuran derivatives; Oxime derivatives; Crystal structure; X-ray; 2D Network.

1. INTRODUCTION

Benzofuran contains a ring system obtained by the fusion of a benzene nucleus with a furan ring. The benzofuran ring system, although by no means as common as indole, is incorporated into many natural products and into synthetic pharmaceuticals[1,2]. Oximes are belonging to the imines, with the general formula R_1R_2C=NOH, where R_1 is an organic side-chain and R_2 may be hydrogen, forming an aldoxime, or another organic group, forming a ketoxime. They exist as two geometric stereo isomers: a *syn* isomer and an *anti* isomer. Aldoximes, except for aromatic aldoximes, which exist only as *anti* isomers, and ketoximes can be separated almost completely and obtained as a *syn* isomer and an *anti* isomer[3].

The life threatening infections caused by pathogenic fungi have increased during the past two decades and there is need to develop new antifungals with good activity against variety of fungal species. Benzofurans and their derivates have physiological, pharmacological and toxic properties and find application as sedatives, hypnotics, agrochemicals, pharmaceuticals, cosmetics and as the building blocks of optical brighteners[4-5]. Oximes are a very effective antifungal agent so they became a matter of interest[6]. Aryl benzofuryl ketoxime sand their derivatives are highly effective compounds[7] and these some shows excellent anibacterialactivity[8].

Hydrogen-bond patterns in crystalline oximes have been analyzed by Bertolasi et al.[9], followed by a systematic examination of hydrogen-bonding in aromatic and aliphatic oximes by Bruton et al.[10] Depending upon the presence of diferent acceptors and donors in oximes, the intermolecular interactions between two or more oximes in the solid-state can be classified into four major categories: Dimers based on O−H···N hydrogen bonds (R_2^2 (6) motif), catemers directed by O−H···N inter-actions (C(3) chains), catemers governed by O−H···O hydrogen bonds (C(2) chains), and oximes in which the R′ group plays a dominant role by accepting a hydrogen-bond from the oxime moiety (C(6) catemeric chains)[11,12]. In this study, the both molecules are ketoxim structures and the molecule (1) forms a dimeric structure with O-H....N hydrogen bond ($R_2^2(6)$ motif).

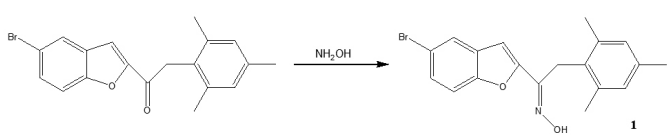
2. EXPERIMENTAL

2.1. Materials and Methods

All of there agents were obtained from commercial sources. Solvents were dried and purified with known conventional methods. Melting points (uncorrected) were determined on a StuartSMP40 automatic melting point apparatus. The IR spectra were measured on a Perkin Elmer FT-IR spectrophotometer (with ATR apparatus). The ^{1}H NMR spectra were recorded on a Varian-Gemini 200 MHz spectrometer and are reported in ppm (δ) relative to tetramethylsilane (TMS) as the internal standard. ^{13}C NMR spectra (50.34 MHz) is referenced to deutero-chloroform ($CDCl_3$). Elemental analyses were determined by a LECO CHNSO-932 auto elemental analysis apparatus.

2.2. Synthesis of 1-(5-Bromobenzofuran-2-yl)-2-mesitylethanoneoxime

To a 100 mL round-bottom flask, 1-(5-Bromobenzofuran-2-yl)-2-mesitylethanon (3.0 g, 10.22 mmol) (which was synthesized by the reaction of 5-Bromocalicylaldehyde, anhydrous K_2CO_3 and 1-Chloro-3-mesitylacetone in acetone) hydroxylamine hydrochloride (0.83 g, 12.00 mmol) and 25 mL of pyridine were respectively added, and the solution was refluxed about two hours. After this time, the mixture was cooled, precipitated in water, filtered, washed with excessive water. Compound **(I)** was dried and recrystallized from acetone respectively (Scheme 1). Yield %: 98. M.p. 189^{0}C. IR (KBr): 3221 cm^{-1} (-OH), 2973-2894 cm^{-1} (C-H), 1613 cm^{-1} (C=N), 1170-1044 cm^{-1} (C-O-C), 1000 cm^{-1} (N-O); ^{1}HNMR ($CDCl_3$) δ: 2.28 (s, 6H, C*H*$_3$), 2.31 (s, 3H, C*H*$_3$), 4.14 (s, 2H, C*H*$_2$), 6.00 (s, 1H, C-*H* furan ring), 6.92 (s 2H, C-*H*mesitylene), 7.34-7.55 (m, 2H, aromatic protons), 7.75 (s, 1H, Br-C-C*H*), 9.63 (s, 1H, -O*H*); ^{13}CNMR ($CDCl_3$) δ: 20.55, 21.20, 26.72, 113.09, 113.31, 124.43, 125.10, 128.72, 129.14, 129.50, 130.42, 136.81, 137.16, 137.41, 149.26, 153.22. Anal. Calcd. for $C_{19}H_{18}O_2BrN$: C, 61.30; H, 4.87; N, 3.76. Found: C, 61.22; H, 4.04; N, 2.96.



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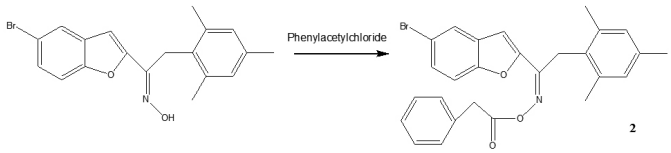
Scheme 1. The preparation of $C_{19}H_{18}BrNO_2$, **(I)**

2.3. Synthesis of 1-(5-Bromobenzofuran-2-yl)-2-mesitylethanone-*O*-(2-phenylacetyl)oxime

Oxime (1.0 g, 2.68 mmol) and dry acetone (50 mL) were placed in a 100 mL two-necked flask with a reflux condenser. The mixture was cooled to -5 ^{0}C and then phenylacetylchloride (0.45 g, 0.38 mL, 2.90 mmol) was added drop wise and the reaction was maintained at room temperature for 2 h. After the reaction mixture was cooled, it was neutralized with NH_3 solution. The resulting precipitate was filtrated and washed with water. Compound **(II)** was recrystallized from ethanol. The following product was obtained (Scheme 2). Yield %: 71. M.p. 135^{0}C. IR (KBr): 3145-2911 cm^{-1} (C-H), 1773 cm^{-1} (C=O),

e-mail addresses:gulyakali@akdeniz.edu.tr ckirilmis@adiyaman.edu.tr

1165-1100 cm^{-1} (C-O-C), 1045 cm^{-1} (N-O); ^{1}HNMR ($CDCl_3$) δ: 2.27 (s, 6H, CH_3), 2.32 (s, 3H, CH_3), 3.80 (s, 2H, Ph-CH_2), 4.15 (s, 2H, CH_2), 6.70 (s 2H, C-*H* mesitylene), 6.98 (s, 1H, C-*H* furan ring), 7.19-7.51 (m, 7H, aromatic protons), 7.65 (s, 1H, Br-C-C*H*); ^{13}CNMR ($CDCl_3$) δ: 22.44, 22.94, 34.24, 42.03, 111.36, 115.31, 116.95, 118.36, 126.50, 130.72, 131.00, 131.30, 131.84, 131.49, 131.72, 135.06, 138.84, 139.24, 152.23, 154.69, 158.44, 169.98.Anal. Calcd. for $C_{27}H_{24}O_3NBr$: C, 66.13; H, 4.93; N, 2.86. Found: C, 65.99; H, 4.98; N, 2.65.



Scheme 2. The preparation of $C_{27}H_{24}BrNO_3$, **(II)**.

2.4. X-ray Structure Determination

Single crystals having dimensions of for **(I)** and for **(II)** were selected for X-ray diffraction studies and were mounted on a glass fiber. X–ray diffraction intensity data for crystals **(I)** and **(II)** were measured on a Agilent Diffraction Xcalibur diffractometer equipped with an Eos-CCD detector, operated at 50 kV and 40 mA with graphite monochromated Mo-Kα (λ = 0.71073 Å) radiation at room temperature. The absorption corrections of the collected data of **(I)** and **(II)** were done analytically. Using Olex2[13], the both structures were solved with the SHELXS structure solution program using the Direct Methods and refined with the SHELXL using least squares minimization[14]. In the crystal structure of **(I),** all H atoms except H atoms bound to O2 atom were located in calculated positions and refined using pertinent riding models with C-H=0.96Å, Uiso(H)=1.5Ueq(C) for methyl H atoms and C-H=0.9 Å, Uiso(H)=1.2Ueq(C) for aromatic H atoms. H atom bound to O2 atom were located in a difference Fourier map and refined freely with O-H=0.782 Uiso(H)=1.2Ueq(O). In the crystal structure of **(II)**, H atoms bound to methyl were located in calculated positions and refined using a riding model with C-H=0.96Å, Uiso(H)=1.5Ueq(C), H atoms bound to aromatic group were located in a difference Fourier map and refined freely with C-H=0.97Å Uiso(H)=1.2Ueq(C) for methylene H atoms, C-H = 0.93other H atoms bound to aromatic group. In this compound, O3 atom was refined disorderly and its position was split into two alternative positions as suggested by SHELXL97[14] using PART instruction without any constraint. The occupancies of disordered groups are allowed to be refined freely. Occupancy of the O3A and O3B is a nearly ratio of 0.55:0.45, describing a 55 % to 45 % disorder, respectively. A summary of crystallographic data, experimental details, and refinement results for the complex are given in Table 1.

3. RESULTS AND DISCUSSIONS

3.1 Crystal Structure of Compounds

The molecular structures of the studied compounds are very similar. The both compounds adopt *Z* conformation and they are ketoxim structure. The selected bond lengths (**Å)** and angles (°) for the compounds are given in Table 2. There is a small geometric difference between the bond angles of -C-CH_2-C- bonds bonded to oxime group of **(I)** and oxime ester group of **(II)**. Their respective bond angles are obtained as 117.9(2)° and 117.0(2)°. In the molecule **(I)** and **(II),** phenyl ring and mesityl ring adopt trans conformation. So the torsion angles of **(II)** are O2-C20-C21-C22 18.3(4)° and C1-C2-N1-C17 12.8(4)°. Fig.1 shows an ORTEP plot[15] of the molecular structures of both compounds. Generally, the bond lengths and angles observed in similar chemical fragments of each structure are quite typical [N1-C9 1.280(3)Å for **(I)**, N1-C2 1.279 Å for **(II)**]. On the contrary, the torsion angles around the oxime group of (**I**) and oxime ester group of (**II**) and N1-O2 bond lengths are slightly different from each other as shown in Table 2. The reason for such discrepancies is the presence of bulky substituent of mesityl group in (**II**).

Table 1. Crystal data and structural refinement parameters for **(I)** and **(II).**

Crystal Data		
Compound	**(I)**	**(II)**
Empirical Formula	$C_{19}H_{18}BrNO_2$	$C_{27}H_{24}BrNO_3$
Formula Weight	372.25	490.38
Cell setting / Space group	monoclinic/$P2_1/c$	triclinic/P-1
Unit cell dimensions	β=102.088(5)^0	α=75.322(5) β=82.682(4) γ=86.746(4)
	a=9.6803(6)Å b=10.7302(8) Å c= 17.0775(8) Å	a=8.2079(3) Å b=10.9999(8) Å c= 13.5020(6) Å
Unit cell volume	1734.53(18) Å^3	1169.27(11) Å^3
Temperature (K)	293	293
Absorption coefficient	2.379 mm^1	1.786 mm^1
Z / Density [g/cm^3]	4/1.426	2/1.393
F(000)	760.0	504.0
Crystal size (mm^3)	0.68× 0.41× 027	0 .62 × 0.24 × 0.20
θ range (°)	2.94-26.37	3.11-26.37
h range		
k range		
l range		
Reflections collected / unique	6866/3538	9061/4779
Completeness to θ_{max}	99.99%	99.9%
Goodness-of-fit on F^2	1.010	1.016
Final R indices [I > 2σ(I)]	R_1 = 0.0441 wR_2 = 0.0827	R_1 = 0.0433 wR_2= 0.0883
R indicesall data	R_1 = 0.0984 wR_2 = 0.0986	R_1 = 0.0791 wR_2 = 0.1030
Max. diff. peakand hole	0.27/-0.33	0.42/-0.36

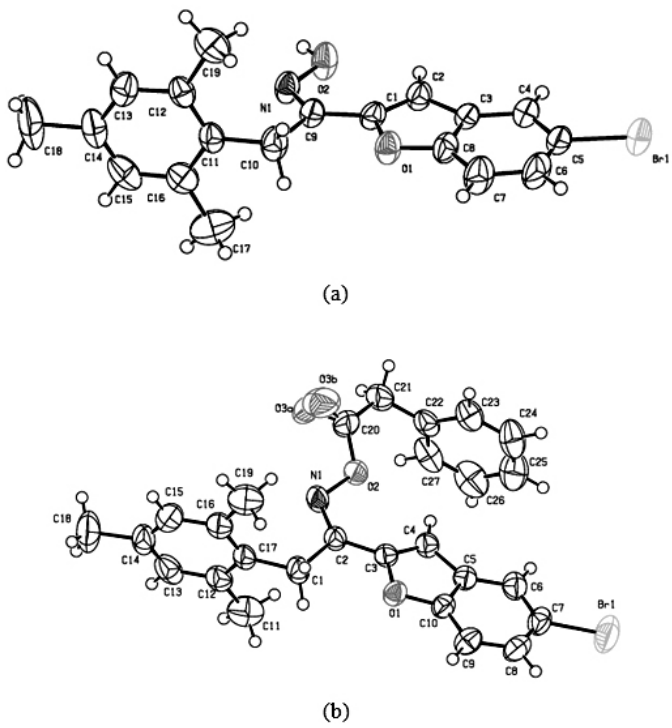


Fig.1. The molecular structure of the compounds, with the atom-numbering scheme. Displacement ellipsoids are drawn at the 50% probability level and H atoms are shown as small spheres of arbitrary radii.

Benzofuran groups and other ring groups almost planar in the crystal structures **(I), (II)**. The benzofuran moiety of **(I)** and **(II)** is essentially planar with a maximum deviation of 0.016(2)Å for C1, 0.029(2) Å for C4, respectively.

The mesityl group of **(I)** and **(II)** is planar with a maximum deviation from the plane of -0.057Å for C10, 0.015Å for C17 and makes an angle of 87.61(10)^0, 78.47(12)^0 with plane of the benzofuran system **(I)** and **(II)** respectively, differing from the values reported between the structures. These differences may be explained by the presence of the different substituents in these compounds**.** These both ring systems of **(I), (II)** are essentially orthogonal to each other. Other planar group in the **(II)** is phenyl group that is almost planar with a maximum deviation from the plane of 0.013Å for C(23). While the dihedral angles between the benzofurane ring system and the phenyl group is 80.23(11)^0, the dihedral angles between the mesityl group and the phenyl group is 3.26(15)^0. Indeed, the mesityl group and the phenyl group are almost in the same plane.

Table 2. Selected bond lengths (**Å)** and angles (°) for the compounds.

Distance (Å)			
Compound (I)		**Compound (II)**	
Br1-C5	1.893(3)	Br1- C7	1.890(3)
N1-C9	1.280(3)	N1-C2	1.279(3)
O1-C1	1.385(3)	O1-C3	1.383(3)
O2-N1	1.389(3)	O2-N1	1.436(3)
Angle (°)			
Compound (I)		**Compound (II)**	
O1-C1-C9	113.4(2)	O1-C3-C2	113.3(2)
C1-O1-C8	106.0(2)	C3-O1-C10	106.15(19)
O2-N1-C9	113.2(2)	O2-N1-C2	110.(2)
O1-C1-C2	110.6(2)	O1-C3-C4	110.8(2)
Br1-C5-C4	119.9(2)	Br1-C6-C7	119.8(2)
Br1-C5-C6	117.4(2)	Br1-C5-C6	117.8(2)
N1-C9-C1	125.8(2)	N1-C2-C3	127.8(2)
N1-C9-C10	116.4(2)	N1-C2-C1	115.2(2)
Br1-C5-C6-C7	-179.4(2)	Br1-C7- C8-C9	-178.9(2)
C19-C10-C11-N1	-2.6(4)	C1-C2-C17-N1	12.8(4)
C1-C9- N1-O2	-0.2 (4)	C2-C3-N1-O2	2.1(4)
O1-C1-C9-C10	0.9(4)	O1-C1-C2-C3	-12.2

An examination of the crystal structure of **(I)** reveals that hydrogen-bond donors (the –OH moieties) are involved in O−H···N intermolecular interactions with oxime nitrogen atoms (the C=N moiety) of neighboring molecules (O2···N1^a =2.791 Å, H2···N1^a =2.08(3) Å), thus forming dimers are show in Fig. 2a. Here the molecules are linked by paired O2-H2 ...N1^a hydrogen bonds in $R_2^2(6)$[16]. Also, there are weak intramolecular C2-H2A…O2 hydrogen bonds in the crystal structure **(I)**. A packing diagram of the molecule **(I)** viewed along the *a* axis by these hydrogen bonds is shown in Fig. 4a. In the crystal structure **(II),** intermolecular C19–H19C···O3B^b hydrogen bonds which form one dimensional infinite chain along the *a* axis is shown in Fig. 2b. A packing diagram of the molecule **(II)** emphasizes the role of intermolecular C19–H19C···O3B^b hydrogen bonds and intramolecular C19-H19C…O3B hydrogen bonds to stabilize the extended packing motif of (**II**) is shown in Fig 4b. Geometric details of intermolecular and intramolecular hydrogen bonds are given Table 3.

Table 3. Hydrogen-bond geometry(Å, °), O-H…π and C-H…π interactions for compound (I) and compound (II)

Bond	D – H	H…A	D…A	D – H…A
Compound (I)				
O2 -H2 ...N1^a	0.78	2.08	2.791(3)	151
O2 -H2… Cg(3)^a	0.78	2.88	3.291(2)	115

Symmetry codes: (a): 2-x,1-y,1-z

Bond	D – H	H…A	D…A	D – H…A
Compound (II)				
C19-H19C…O3B^b	0.96	2.47	3.32(3)	148
C4-H4…Cg4^c	0.93	2.76	3.646(3)	161
C11-H11A… Cg3^d	0.96	2.94	3.754(4)	143
C27-H27…Cg2^e	0.93	2.81	3.705(3)	162

Symmetry codes: (b) : -1+x,y,z; (c): x,y,z ; (d): 1-x, 2-y, 1-z; (e): 1+x, y,z

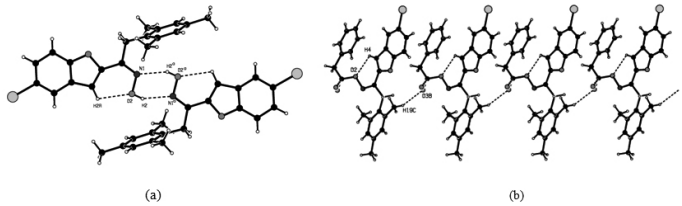


Fig.2. (a) A centrosymetric hydrogen-bonded dimer structure formed *via* intermolecular O-H…N hydrogen bonds around the inversion centre and intramolecular C-H…O bonds for **(I). (b)** One dimensional infinite chain formed *via* intermolecular C–H···O hydrogen bonds for **(II)**.

The stacking and O-H…π interactions are observed in the structure of compound **(I)** and similarly stacking and C-H…π interactions are observed in the structure of compound **(II)** (shown in Fig. 3). 2D supramolecular network **(I)** is formed by stacking [Cg1 …Cg3^f = 4.0336, where Cg1 are the centroids of the furan ring and Cg3 centroids of the mesityl ring ; symetry codes: (f) x,1/2-y,-1/2+z] and O-H- interactions [O2-H2…Cg3^a]. In the structure of compound (II), 2D supramolecular network is formed by two stacking [Cg1… Cg2^g =3.7118(15) Å, where Cg1 and Cg2 are the centroids of the furan ring and benzen ring in benzofuran; symetry codes: (g) 1-x,1-y,2-z, Cg3…Cg4^h = 4.3467(19) Å, where Cg3 and Cg4 are the centroids of the (C12/C17) and (C22/C27) benzen rings; symetry codes: (h) 2-x,1-y,1-z], two C-H…π interactions [C4-H4…Cg4^d and C11-H11A…Cg3^e].

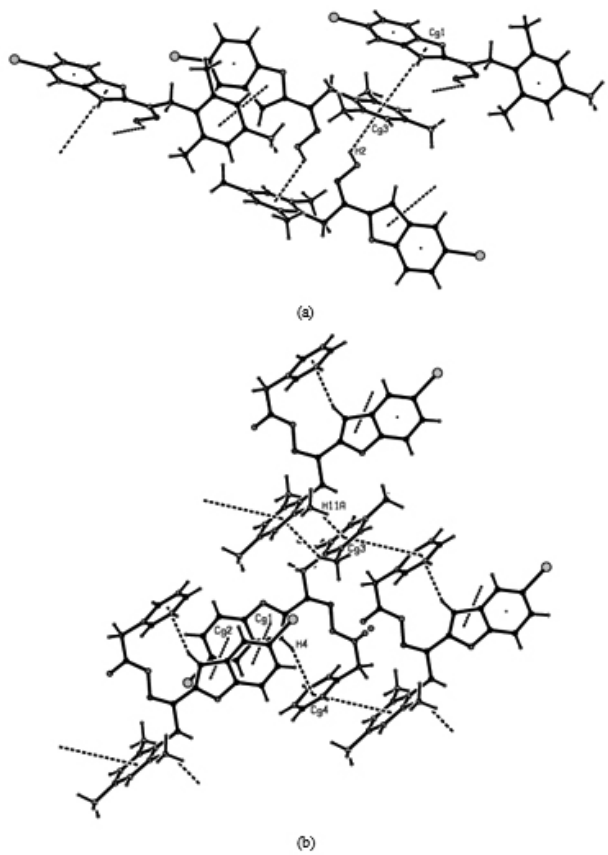


Fig. 3. (a) 2D supramolecular network formed by intermolecular and O-H…π interactions in the crystal structure of compound **(I)**. **(b)** 2D supramolecular network formed by intermolecular and C-Hinteractions in the crystal structure of compound **(II)**.

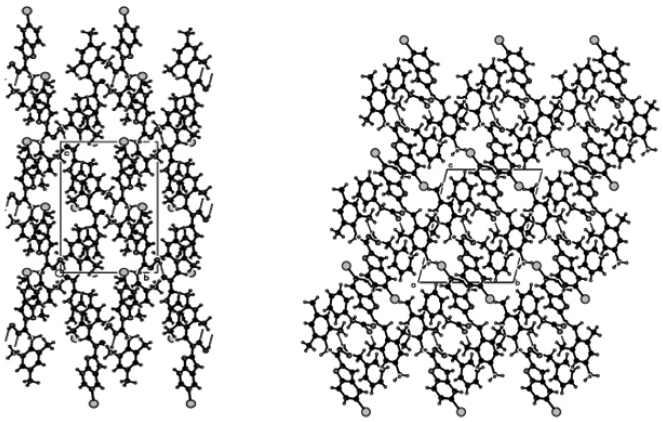


Fig. 4. (a), (b) A packing diagram of compound **(I)** and **(II) ,** viewed along the crystallographic *a* axis by intramolecular and intermolecular hydrogen bonds. The dashed lines indicated hydrogen bonds.

4. CONCLUSIONS

The single-crystal X-ray diffraction studies reported in this work have established a Z stereochemistry for compound **(I)** and compound **(II)** consist of oxime and oxime ester group respectively as derived from spectroscopy data. Also,it is defined that the both compounds are ketoxim structure. In the case of ketoximes, the higher charge on the oxime nitrogen atom, coupled with an inactive R′ group, clearly tilts the balance toward the formation of (6)(R_2^2(6) dimers. In this work we have shown that in the solid state H-bonds link two oxime molecules for compound **(I)**, which form centrosymmetric dimers with an (6)(R_2^2(6) graph-set motif whereas a chain formed by hydrogen bond is observed in the compound **(II)**. Furthermore, C-H…π, O-H…π and π…π interactions give rise to a 2D self-arrangement in the both compounds. Therefore oxime structures may provide expanded supramolecular synthetic opportunities compared to other hydrogen bonding moieties.

5. ACKNOWLEDGEMENTS

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6.Supplementary material

Crystallographic data as .cif files for the structures reported in this paper have been deposited at the Cambridge Crystallographic Data Center with CCDC 1404921 for compound **(I)** and CCDC 1404983 for compound **(II).** Copies of the data can be obtained free of charge at www.ccdc.cam.ac.uk/cont s/retrieving.html (or from the Cambridge Crystallographic Data Center, 12, Union Road, Cambridge CB2 1EZ, UK). Email: deposit@ccdc.cam.ac.uk.

7. REFERENCES

1. H. Khanam, Shamsuzzaman, **2015**, *European Journal of Medicinal Chemistry,* 97, 483-504.
2. T.L. Gilchrist, **1985**, *Heterocyclic Chemistry*, 174.
3. J. Kalia, R.T. Raines, **2008**, *Angew. Chem. Int. Ed.*, 47, 7523–7526.
4. M. Kamal, K. A. Shakya, T. Jawaıd, *International Journal of Medicaland Pharmaceutical Sciences,* **2011**, 1-3.
5. C. Kirilmis, M. Ahmedzade, S. Servi, M. Koca, A. Kizirgil, C. Kazaz, *European Journal of Medicinal Chemistry,* **2008,** 43, 300-308.
6. V. N. Telvekar, H. S. Kundaikar, K. N. Patel, H. K. Chaudhari, *QSAR Comb. Sci.,* **2008**, 27, 1193-1203.
7. N. G. Karaburun, K. Benkli, Y. Tunali, Ü. Uçucu, Ş. Demirayak, *European Journal of Medicinal Chemistry*, **2006**, 1, 651–656.
8. K. Mehnaz, A. K. Shakya, J. Talha , *International Jounal of Medical and Pharmaceutical Sciences*, **2011**, 13, 1-15.
9. V. Bertalosi, G. Gilli, A. Veronese, *Acta. Cryst*. B, **1982**, 38, 502−511.
10. E. A. Bruton, L. Brammer, F. C. Pigge, C. B. Aakeröy, D. S. Leinen, *New J. Chem.,* **2003**, 27, 1084−1094.
11. J. N. Low, L. M. N. B. F. Santos, C. F. R. A. C. Lima, P. Brandão, L. R. Gomes, Eur. *J. Chem*., **2010**, 1, 61−66.
12. C. B. Aakeröy, A. S. Sinha, K. N. Epa, P. D. Chopade, M. M. Smith, J. Desper, *Crystal Growth Design*, **2013**, 13, 2687-2695.
13. O.V. Dolomanov, L.J. Bourhis, R.J. Gildea, J.A.K. Howard, H. Puschmann, *J. Appl. Cryst.,* **2009**, *42*, 339-341.
14. G.M. Sheldrick, *Acta Cryst. A*, **2008**, *64*, 112.
15. M.N. Burnett, C.K. Johnson, ORTEPIII. Report ORNL-6895, Oak Ridge National Laboratory, TN, USA, **1996.**
16. J. Bernstein, R. E Davis, L. Shimoni, N. L. Chang, **1995**, *Angew. Chem. Int. Ed. Engl.,* 34, 1555–1573.
17. CrysAlis PRO and CrysAlis RED. Agilent Technologies, Yarnton, Oxfordshire, England, **2002**.
18. V. T. Yilmaz, C. Kazak, C. Kirilmis, M. Kocac and F.W. Heinemannd, *Acta Cryst.C,* **2005,** 61, o438-o441.
19. R. Koner, I. Goldberg, **2008**, *Acta Cryst. C,* 65,m37-m41.
20. H.D. Choi, P. J. Seo, U. Lee, **2013**, *Acta Cryst. E*, 69, o1135.
21. H.D. Choi, P.J. Seo, B.W. Sonb, U. Leeb, **2006**, *Acta Cryst. E*, 62, o2335–o2336.
22. C. Arıcı, D. Ülkü, M. Koca, M. Ahmedzade, **2004**, *Acta Cryst. E*, 60, o94-o942.
23. L. Chen, Q.B. Xu, G.L.Song, H.J. Zhu, **2009**, *Acta Cryst. E* 65, o1083
24. G. Anuradha, G. Vasuki, I. A. Khan, M.V. Kulkarni, **2012**, *Crystal Structure Theory and Applications*, 1, 107-113.
25. J. Sepencer, A.P. Mendham, B.Z. Chowdhry, R.A. Palmer, D.R.Lisgarten, B.M. Trost, N. Cramer, L. Male, S.J. Coles, **2014**, *Journal of Pharmaceutical Chemistry*, 1(3), 44-49.
26. A. Drzewiecka, A.E. Koziol, T.P. Ruiz, M. F. Gomez, M. Struga, J. Kossakowski, K. Ostrowska, T. Lis, **2012,** *Original Research***,** 23, 1617–1629.
27. R. Gowda, V.B. Jadhav, M. V. Kulkarni, K. V. A. Gowda, **2009,** *X-ray Structure Analysis Online*, VOL. 25.
28. J. Kossakowski, M. Krawiecka, B. Kuran, J. Stefańska, I. Wolska, **2010**, *Molecules, 15*, 4737-4749.
29. H. M. Titi, I. Goldberg, **2010,** *CrystEngComm,* 12, 3914–3922.
30. M. Krawiecka, B. Kuran, J. Kossakowski, I. Wolska, M. Kierzkowska, G. Meynarczyk, **2012**, *Polish Pharmaceutical Society,* Vol. 69 No.6 1055-1065.
31. W. W. Hanand, J.L. Wu, **2014**, *Z. Kristallogr. NCS,* 229, 27-28.
32. Y. Ooyama, T. Mamura, K. Yoshida, **2006**, *Chem. Eur. J*.**,** 12, 7827–7838.