Original Article

Illumınatıon of Sıngle Crystal Structure Contaınıng Bıthıazole and Methylcyclobutyl Groups By X-Ray Dıffractıon Method

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Abstract

In this thesis study, 4,4'-Bis(3-phenyl-3-methylcyclobutyl)-2,2'-bithiazole (C28H28N2S2) the single crystal structure consisting of some compound groups such as thiazole, and phenyl was synthesized and characterized by IR, 1H NMR, and 13C NMR techniques. The molecular structure of the single crystal structure consisting of compounds that have a common usage area was analyzed by X-ray diffraction technique. The compound C28H28N2S2 was found to be in the monoclinic crystal system and the P21/c space group. The unit cell parameters of the molecular structure were obtained at a= 5.8461(6)Å, b= 35.806(4) Å, c= 11.6538(11) Å, α=γ= 90° and β= 93.774(8)°, z =4, and (volume) V= 2434.2(4) Å3 from the X-ray diffraction results. It is seen that the molecular structure C28H28N2S2 in the asymmetric unit is in a symmetrical structure. When the bond parameters of the molecular structure were compared with similar studies, the bond parameters were found to be quite compatible. The molecular structure C28H28N2S2 classically does not contain an intermolecular or intramolecular hydrogen bond.. However, the C–H…*π* interactions have been observed between the phenyl carbon atoms and the phenyl ring centers, which allow the molecular structure to be stacked in space.

Keywords: Bithiazole, Cyclobutane, Single crystal, X-ray diffraction.

INTRODUCTION

Crystal structures are investigated by studying the diffraction pattern of the X-ray beam by the crystal. Diffraction occurs only in directions defined just as it does for light by diffraction gratings. Measurement of the diffraction directions and intensities of scattered rays may give the composition analysis more information. The crystal is responsible for this diffraction (Blake et al. 2008).

From the previous studies about crystal networks, it was found that the most prominent characteristic of the crystal is its enjoyment of transitional symmetry, and this requires that all crystalline properties be periodic, repeating themselves according to the different crystal directions defined by a vector in the crystal lattice. This study investigates the packing of the crystal structure and how to determine the crystal structure (Ladd and Palmer 1985).

Three types of beams are used in diffraction experiments: X-rays, neutron beams, and electron beams. The mathematical treatment of the three types is almost the same so that the examination in detail will be about only X-ray diffraction (Stout and Jensen 1989).

After a brief discussion about X-rays' generatation and properties, we will be given to a brief conclusion of Bragg's law of ray scattering by crystalline levels. This study will discuss the scattering of rays by the atom and by the crystal. Also, It will discuss the symmetrical crystal structure and various practical methods for studying crystal structure.

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Access this article online	
Quick Response Code:	**Website:** www.pnrjournal.com
	DOI: 10.47750/pnr.2022.13.04.126

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How to cite this article: Suzan Abdulkareem Abbas Kbah, Dr. Çiğdem Yüksektepe Ataol, Dr. İbrahim Yılmaz, Illumınatıon of Sıngle Crystal Structure Contaınıng Bıthıazole and Methylcyclobutyl Groups By X-Ray Dıffractıon Method, J PHARM NEGATIVE RESULTS 2022;13: 927-935.

In this thesis study, it is investigated to the structure of the compound containing functional groups such as bithiazole, and cyclobutane.

Bithiazole derivatives are of interest in the field of organic metals and as nonlinear optics materials. Solid bithiazoles have a planar or nearly planar structure. Due to the equilibrium between conjugative and steric intramolecular interactions, large deviations from planarity are possible in both solution and the free state. Thermochromic and solvatochromic research provides evidence of chain flexibility in solutions (Su et al. 2017).

The analysis of the single crystal structure will be carried out by using the X-ray diffraction method. The single crystal bond parameters are compared with the literature values. The crystal packing of the molecular structure in three-dimensional space was investigated, and hydrogen bonds contributing to the flocculation were determined.

MATERIALS AND METHOS

In this thesis, a single crystal structure with a closed formula of C28H28N2S2 was synthesized, characterized and its molecular structure was elucidated.

Material

In Figure 3.1, the crystal image of a single crystal with the closed formula C28H28N2S2 is given. The single crystal structure seen in Figure 3.1 has been studied in the thesis, and its structure has been investigated.

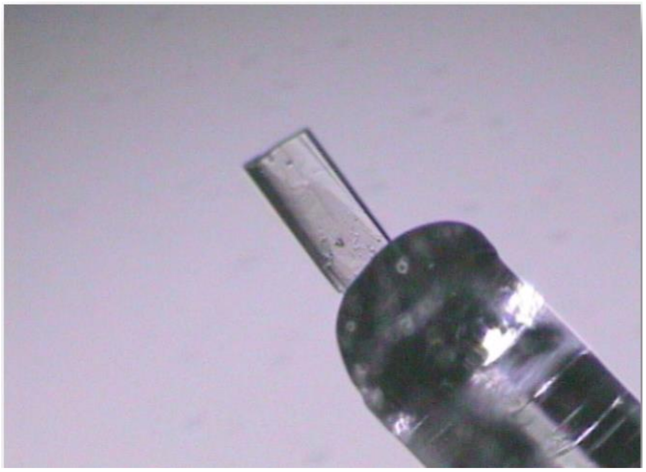

Figure 3.1 The crystal image of the single crystal C28H28N2S2

Synthesis of the single crystal (C28H28N2S2)

“The mixture of dithiooxamide (0.601 g, 5 mmoL) and 2-chloro-1-(3-methyl-3-phenylcyclobutyl) ethanone (2.2271 g, 10 mmoL) in 20 mL of absolute ethanol was stirred at room temperature for 2h (IR). The solution was cooled to room temperature and then made alkaline with an aqueous solution of NH3 (5%), and brilliant brown precipitate separated by suction, washed with aqueous NH3 solution several times and dried in air, and crystallized from ethanol. Yield: 65%, melting point: 446 K. The chemical diagram and the synthesis form of the molecular structure are given in Figures 3.2 and 3.3.”

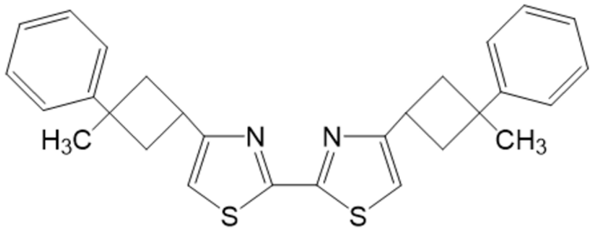

Figure 3.2 Chemical diagram of the C28H28N2S2 compound

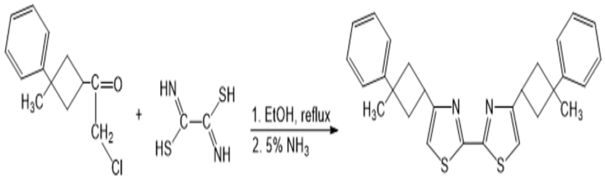

Figure 3.3 Synthetic route for the synthesis of the target compound”

Method

The single crystal structure in the thesis was characterized by IR (Infrared), 1H NMR (Nuclear Magnetic Resonance) and 13C NMR methods. Its structure was elucidated by the X-ray diffraction analysis. The experimental data belonging to the single crystal were collected with the aid of a STOE IPDS II single crystal diffractometer at 296 K utilizing a MoKα ($\lambda = 0.71073$Å) characteristic X-ray. With the help of these data, the space group of the crystal, unit cell parameters and the number of molecules in the component cell were found. All structures were solved with the help of direct methods (Sheldrick 2015). The intensity peaks of the heavy atoms in the Fourier map were named and the positions of all atoms except hydrogen were found. All non-hydrogen atoms were easily found from the difference Fourier map and refined anisotropically. All hydrogen atoms except formamide hydrogen atom were included using a riding model and refined isotropically with CH = 0.93 Å (CH = 0.98 Å for cyclobutene ring), CH3 = 0.96 Å, and CH2 = 0.97 Å, Uiso(H) = 1.2Ueq (1.5 for methyl group). After the initial phase was created and the structure solution process was completed, the refinement process was performed using the least squares method (Sheldrick 2015). Molecular figures were created with ORTEP-3 (Farrugia 2012) and PLATON (Spek 2009) programs. Experimental IR spectra were acquired from ATI Unicam-Mattson 1000 FTIR spectrometer (KBr). Experimental NMR spectra were obtained with the BerRUKER device.”

RESULTS AND DISCUSSION

Characterization of 4,4'-Bis(3-Phenyl-3-Methylcyclobutyl)-2,2'-Bithiazole

The single crystal structure in the thesis was characterized by IR, 1H NMR, and 13C NMR methods.

IR spectra of the C28H28N2S2 compound

Some vibration parameters of the molecular structure are observed at 3020 cm-1 (aromatics), 2961-2856 cm-1 (aliphatics), 1500 cm-1 (C=N azomethine), 1442 cm-1 (C=C), 702 cm-1 (C-S-C). by IR spectra. The IR spectra of the C28H28N2S2 compound is given in Figure 4.1.

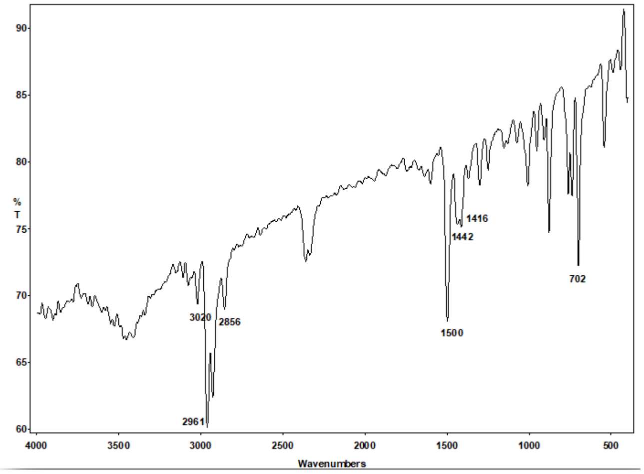

Figure 4.1 IR spectra of the C28H28N2S2 compound

1H NMR spectra of the C28H28N2S2 compound

Some characteristic 1H NMR shifts (CDCl3, δ, ppm) are observed at 1.65 (s, 6H, -CH3), 2.61-2.64 (m, 8H, -CH2- in cyclobutane ring), 3.91 (quint, j = 9.2 Hz, 2H, >CH- in cyclobutane ring), 6.99 (s, 2H, on thiazole ring), 7.24-7.26 (m, 6H aromatics), 7.34-7.36 (m, 4H aromatics). The 1H NMR spectra of the C28H28N2S2 compound is given in Figure 4.2.

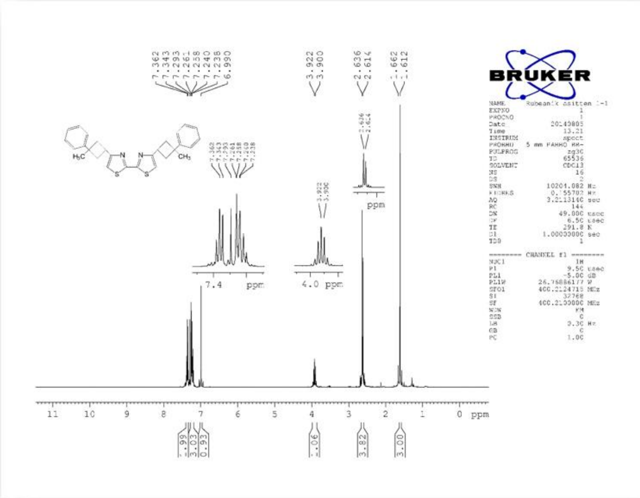

Figure 4.2 1H NMR spectra of the C28H28N2S2 compound

13C NMR spectra of C28H28N2S2 compound

Some characteristic 13C NMR shifts (CDCl3, δ, ppm) are observed at 162.48, 161.15, 152.17, 128.33, 125.68, 124.65, 114.20, 41.06, 39.00, 30.76, 30.17. The 13C NMR spectra of the C28H28N2S2 is in Figure 4.3.

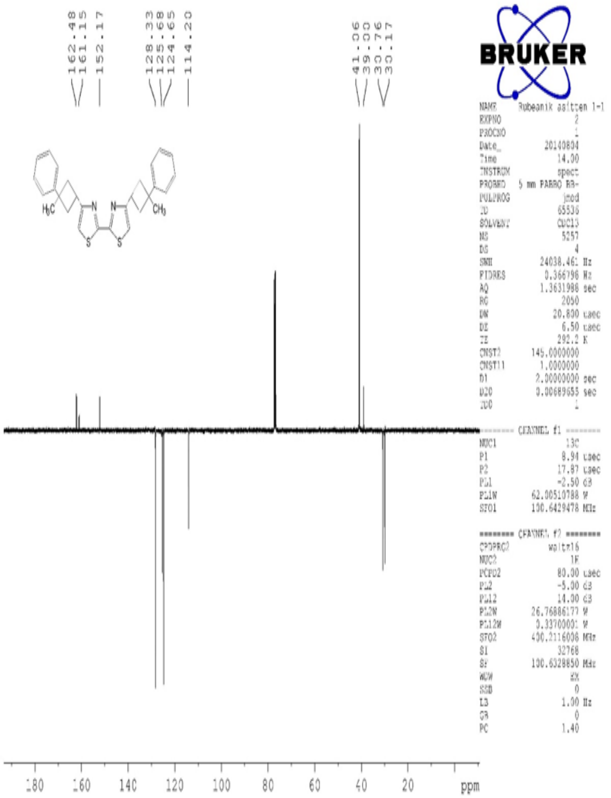

Figure 4.3 13C NMR spectra of the C28H28N2S2 compound

X-ray Structure Analysis of the C28H28N2S2 Single Crystal

It contains groups such as thiazole, phenyl, and cyclobutene rings in its structure and as we mentioned in the previous sections, it has many uses. The crystal structure of the compound with the formula C28H28N2S2 was elucidated using the XRD technique. The atomic coordinates of the molecular structure (x, y, z) are given in Appendix 1. Anisotropic vibration parameters of the molecular structure are given in Appendix 2.

The molecular structure of C28H28N2S2 using XRD data, it was found to be in the monoclinic crystal system and the P21/c space group. The unit cell parameters of the molecular structure a= 5.8461(6)Å, b= 35.806(4) Å, c= 11.6538(11) Å, α=γ= 90° and β= 93.774(8)°, z =4, and (volume) V= 2434.2(4) Å3 from the XRD analysis results. The crystallographic data of the molecular structure is given in

Table 4.1. Ortep view of the molecular structure is shown in Figure 4.4. As seen in Figure 4.4, the molecular structure has a symmetrical structure. The selected geometric parameters and the hydrogen bond interactions of the compound C28H28N2S2 are given in Table 4.2 and Table 4.3.

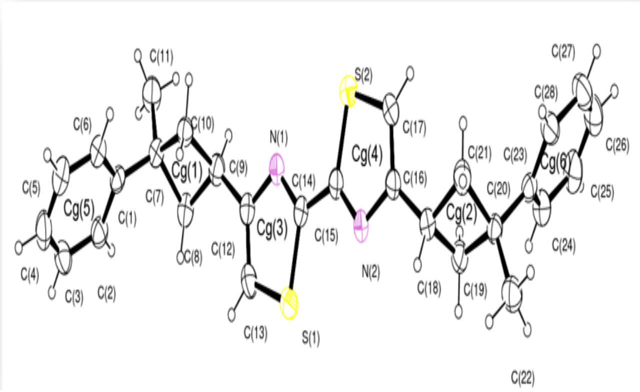

Figure 4.4 Ortep view of the C28H28N2S2 single crystal

“From the anisotropic vibration parameters given in Appendix 2. As can be seen in Figure 4.4, the definitions such as the cyclobutene rings Cg(1)(C(7)-C(8)-C(9)-C(10)), Cg(2)(C(18)-C(19)-C(20)-C(21)), thiazole rings Cg(3)(N(1)-C(13)-C(12)-S(1)-C(14)), Cg(4)(N(2)-C(16)-C(17)-S(2)-C(15)), phenyl rings Cg(5)(C(1)-C(2)-C(3)-C(4)-C(5)-C(6)), and Cg(6)(C(23)-C(24)-C(25)-C(26)-C(27)-C(28)) have been made. The rings Cg(3), Cg(4), Cg(5), and Cg(6) are planar with maximum deviations of 0.0023(19), -0.0056(21), 0.0057(23), and 0.0030(26) Å, respectively. The dihedral angles between the rings Cg(1), Cg(2), Cg(3), Cg(4), Cg(5), and Cg(6) are 5.92(24)° Cg(1)/Cg(2), 56.41(16)° Cg(1)/Cg(3), 59.17(15)° Cg(1)/Cg(4), 38.44(15)° Cg(1)/Cg(5), 45.32(15)° Cg(1)/Cg(6), 61.70(15)° Cg(2)/Cg(3), 64.40(15)° Cg(2)/Cg(4), 32.99(16)° Cg(2)/Cg(5), 39.84(16)° Cg(2)/Cg(6), 2.90(21)° Cg(3)/Cg(4), 84.62(09)° Cg(3)/Cg(5), 89.82(09)° Cg(3)/Cg(6), 86.61(09)° Cg(4)/Cg(5), 87.98(09)° Cg(4)/Cg(6). The cyclobutane rings Cg(1) and Cg(2), are non-planar, the dihedral angle between C(8)-C(9)-C(10) plane and C(8)-C(7)-C(10) plane with C(19)-C(20)-C(21) plane and C(19)-C(18)-C(21) plane are 27.49 (23)° and 21.08(24)°, respectively. “The value found in this study is in agreement with literature values” such as 24.02(22)° (Saraçoğlu and Cukurovalı 2012), 25.74(15)° (Yuksektepe et al. 2010) and 24.3° (Allen 1984).”

Table 4.1 The crystallographic data of the C28H28N2S2 compound

Chemical formula	$C_{28}H_{28}N_2S_2$
Molecular weight	456.64
Temperature, T (K)	296
Wavelength (Å)	0.71073
Crystal system	Monoclinic
Crystal size (mm^3)	0.02 x 0.32 x 0.65
Space group	$P\ 2_1/c$
a (Å)	5.8461(6)
b (Å)	35.806(4)
c (Å)	11.6538(11)
α (°)	90
β (°)	93.774(8)
γ (°)	90
Volume, V ($Å^3$)	2434.2(4)
Z	4
Calculated density ($Mg\ m^{-3}$)	1.246
θ range (º)	1.14– 26.27
Index range	h = -7→7, k = -43→43, l = -14→14
Measured reflections	17441
Independent reflections	4610
Observed reflections (I>2σ)	2252
GOOF, S	0.949
R_1 index (I>2σ)	0.0583
wR_2 index (I>2σ)	0.0734
$\Delta\rho_{min}$, $\Delta\rho_{max}$ ($e/Å^3$)	-0.170, 0.163
CCDC number	1584783

Table 4.2 The calculated and the experimental bond parameters of the C28H28N2S2 compound

	EXPERIMENTAL VALUES (Å, °)		**EXPERIMENTAL VALUES (Å, °)**
BOND LENGTHS (Å)		**BOND ANGLES (°)**	
C(1)–C(6)	1.382(4)	C(1)–C(7)–C(11)	109.9(2)
C(1)– C(2)	1.384(4)	C(1)–C(7)–C(10)	116.6(2)
C(1)–C(7)	1.500(4)	C(11)–C(7)–C(10)	112.5(2)
C(2)–C(3)	1.387(4)	C(1)–C(7)–C(8)	116.5(2)
C(3)–C(4)	1.373(5)	C(11)–C(7)–C(8)	113.0(3)
C(4)–C(5)	1.367(4)	C(10)–C(7)–C(8)	86.7(2)
C(5)–C(6)	1.375(4)	C(9)–C(8)–C(7)	89.2(2)
C(7)–C(11)	1.531(4)	C(12)–C(9)–C(8)	120.9(3)
C(7)–C(10)	1.550(4)	C(12)–C(9)–C(10)	116.9(3)
C(7)–C(8)	1.554(4)	C(8)–C(9)–C(10)	87.9(2)
C(8)–C(9)	1.534(4)	C(9)–C(10)–C(7)	89.4(2)
C(9)–C(12)	1.494(4)	C(13)–C(12)–N(1)	113.4(3)
C(9)–C(10)	1.536(4)	C(13)–C(12)–C(9)	128.2(3)
C(12)–C(13)	1.350(4)	N(1)–C(12)–C(9)	118.2(3)
C(12)–N(1)	1.377(3)	C(12)–C(13)–S(1)	111.9(2)
C(13)–S(1)	1.709(3)	N(1)–C(14)–C(15)	122.8(3)
C(14)–N(1)	1.312(4)	N(1)–C(14)–S(1)	115.0(2)
C(14)–C(15)	1.444(4)	C(15)–C(14)–S(1)	122.2(2)
C(14)–S(1)	1.708(3)	N(2)–C(15)–C(14)	123.6(3)
C(15)–N(2)	1.310(3)	N(2)–C(15)–S(2)	115.3(2)
C(15)–S(2)	1.717(3)	C(14)–C(15)–S(2)	121.1(2)
C(16)–C(17)	1.371(3)	C(17)–C(16)–N(2)	113.8(3)
C(16)–N(2)	1.355(4)	C(17)–C(16)–C(18)	127.2(3)
C(16) C(18)	1.489(4)	N(2)–C(16)–C(18)	118.9(3)
C(17)–S(2)	1.713(3)	C(16)–C(17)–S(2)	111.8(2)
C(18)–C(21)	1.536(4)	C(16)–C(18)–C(21)	120.3(3)
C(18)–C(19)	1.542(4)	C(16)–C(18)–C(19)	115.7(3)
	EXPERIMENTAL VALUES		**EXPERIMENTAL VALUES**
C(20)–C(23)	1.552(4)	C(21)–C(18)–C(19)	88.4(2)
C(19)–C(20)	1.506(4)	C(18)–C(19)–C(20)	89.9(2)
C(20)–C(22)	1.525(4)	C(23)–C(20)–C(22)	109.6(2)
C(20)–C(21)	1.551(4)	C(23)–C(20)–C(21)	116.4(2)
C(23)–C(28)	1.379(4)	C(22)–C(20)–C(21)	112.5(3)
C(23)–C(24)	1.386(4)	C(23)–C(20)–C(19)	117.1(2)

C(24)–C(25)	1.383(4)	C(22)–C(20)–C(19)	112.3(2)
C(25)–C(26)	1.361(5)	C(21)–C(20)–C(19)	87.5(2)
C(26)–C(27)	1.369(5)	C(18)–C(21)–C(20)	90.2(2)
C(27)–C(28)	1.384(4)	C(28)–C(23)–C(24)	117.6(3)
BOND ANGLES (°)		C(28)–C(23)–C(20)	120.4(3)
C(6)–C(1)–C(2)	117.2(3)	C(24)–C(23)–C(20)	121.9(3)
C(6)–C(1)–C(7)	121.9(3)	C(25)–C(24)–C(23)	121.0(3)
C(2)–C(1)–C(7)	120.9(3)	C(26)–C(25)–C(24)	120.6(4)
C(1)–C(2)–C(3)	121.3(3)	C(25)–C(26)–C(27)	119.2(3)
C(4)–C(3)–C(2)	120.0(3)	C(26)–C(27)–C(28)	120.6(3)
C(5)–C(4)–C(3)	119.5(3)	C(23)–C(28)–C(27)	121.0(3)
C(4)–C(5)–C(6)	120.3(3)	C(14)–N(1)–C(12)	111.1(3)
C(5)–C(6)–C(1)	121.8(3)	C(15)–N(2)–C(16)	110.9(2)
TORSION ANGLES (°)		C(14)–S(1)–C(13)	88.59(17)
C(6)–C(1)–C(2)–C(3)	0.8(4)	C(17)–S(2)–C(15)	88.19(16)
C(7)–C(1)–C(2)–C(3)	179.9(3)	**TORSION ANGLES (°)**	
C(1)–C(2)–C(3)–C(4)	-0.3(5)	C(7)–C(8)–C(9)–C(10)	-19.6(2)
C(2)–C(3)–C(4)–C(5)	-0.6(5)	C(12)–C(9)–C(10)–C(7)	143.5(3)
C(3)–C(4)–C(5)–C(6)	1.2(5)	C(8)–C(9)–C(10)–C(7)	19.6(2)
C(4)–C(5)–C(6)–C(1)	-0.7(5)	C(1)–C(7) –C(10)–C(9)	-137.5(3)
C(2)–C(1)–C(6)–C(5)	-0.2(4)	C(11)–C(7)–C(10)–C(9)	94.1(3)
C(7)–C(1)–C(6)–C(5)	-179.4(3)	C(8)–C(7)–C(10)–C(9)	-19.4(2)
	EXPERIMENTAL VALUES		**EXPERIMENTAL VALUES**
C(6)–C(1)–C(7)–C(11)	93.1(3)	C(8)–C(9)–C(12)–C(13)	7.9(5)
C(2)–C(1)–C(7)–C(11)	-86.0(3)	C(10)–C(9)–C(12)–C(13)	-97.0(4)
C(6)–C(1)–C(7)–C(10)	-36.4(4)	C(8)–C(9)–C(12)–N(1)	-176.8(3)
C(2)–C(1)–C(7)–C(10)	144.4(3)	C(10)–C(9)–C(12)–N(1)	78.4(4)
C(6)–C(1)–C(7)–C(8)	-136.7(3)	N(1)–C(12)–C(13)–S(1)	0.4(4)
C(2)–C(1)–C(7)–C(8)	44.1(4)	C(9)–C(12)–C(13)–S(1)	175.9(3)
C(1)–C(7)–C(8)–C(9)	137.6(3)	N(1)–C(14)–C(15)–N(2)	-177.9(3)
TORSION ANGLES (°)		S(1)–C(14)–C(15)–N(2)	3.0(4)
C(11)–C(7)–C(8)–C(9)	-93.7(3)	N(1)–C(14)–C(15)–S(2)	2.6(4)
C(10)–C(7)–C(8)–C(9)	19.4(2)	S(1)–C(14)–C(15)–S(2)	-176.49(18)
C(7)–C(8)–C(9)–C12	-140.0(3)	N(2)–C(16)–C(17)–S(2)	1.0(4)
C(18)–C(16)–C(17)–S(2)	-175.3(3)	C(20)–C(23)–C(24)–C(25)	178.5(3)
C(17)–C(16)–C(18)–C(21)	-14.1(5)	C(23)–C(24)–C(25)–C(26)	-0.6(5)
N(2)–C(16)–C(18)–C(21)	169.8(3)	C(24)–C(25)–C(26)–C(27)	0.6(6)
C(17)–C(16)–C(18)–C(19)	90.2(4)	C(25)–C(26)–C(27)–C(28)	-0.3(6)
N(2)–C(16)–C(18)–C(19)	-86.0(3)	C(24)–C(23)–C(28)–C(27)	0.1(5)

C(16)–C(18)–C(19)–C(20)	-138.2(3)	C(20)–C(23)–C(28) –C(27)	-178.2(3)
C(21)–C(18)–C(19)–C(20)	-15.0(2)	C(26)–C(27)–C(28)–C(23)	-0.1(5)
C(18)–C(19)–C(20)–C(23)	133.5(3)	C(15)–C(14)–N(1)–C(12)	-179.2(3)
C(18)–C(19)–C(20)–C(22)	-98.5(3)	S(1)–C(14)–N(1)–C(12)	-0.1(3)
C(18)–C(19)–C(20)–C(21)	14.9(2)	C(13)–C(12)–N(1)–C(14)	-0.2(4)
C(16)–C(18)–C(21)–C(20)	134.2(3)	C(9)–C(12)–N(1)–C(14)	-176.2(3)
C(19)–C(18)–C(21)–C(20)	15.1(2)	C(14)–C(15)–N(2)–C(16)	-178.9(3)
C(23)–C(20)–C(21)–C(18)	-134.1(3)	S(2)–C(15)–N(2)–C(16)	0.6(3)
C(22)–C(20)–C(21)–C(18)	98.2(3)	C(17)–C(16)–N(2)–C(15)	-1.0(4)
C(19)–C(20)–C(21)–C(18)	-15.0(2)	C(18)–C(16)–N(2)–C(15)	175.6(3)
C(22)–C(20)–C(23)–C(28)	84.1(3)	N(1)–C(14)–S(1)–C(13)	0.2(3)
C(21)–C(20)–C(23)–C(28)	-45.0(4)	C(15)–C(14)–S(1)–C(13)	179.4(3)
	EXPERIMENTAL VALUES		**EXPERIMENTAL VALUES**
C(19)–C(20)–C(23)–C(28)	-146.5(3)	C(12)–C(13)–S(1)–C(14)	-0.3(3)
C(22)–C(20)–C(23)–C(24)	-94.1(3)	C(16)–C(17)–S(2)–C(15)	-0.5(3)
C(21)–C(20)–C(23)–C(24)	136.8(3)	N(2)–C(15)–S(2)–C(17)	0.0(3)
C(19)–C(20)–C(23)–C(24)	35.3(4)	C(14)–C(15)–S(2)–C(17)	179.5(3)
C(28)–C(23)–C(24)–C(25)	0.2(5)		

As shown in Table 4.2, N(1)–C(14) and N(2)–C(15) (1.312(4)Å and 1.310(3)Å) bond lengths are shorter than N(1)–C(12) and N(2)–C(16) (1.377(3)Å and 1.355(4)Å) bond lengths in the thiazole rings. These bonds have a double bond character. We can say from Table 4.2 that the C(14)–C(15) bond distance connecting the two symmetrical molecular structures was found to be 1.444(4)Å. These bond distances in the thiazole ring were found to be compatible with the literature (Yuksektepe et al. 2010).

Table 4.3 The hydrogen bonding geometry of the compound C28H28N2S2 (Å, °)

HYDROGEN BONDS (Å, °)	**D–H**	**H...Cg**	**D...Cg**	**D–H...Cg**
C(4)−H(4)...Cg(6)^{i}	0.93	2.81	3.613(4)	145
C(26)−H(26)...Cg(5)^{ii}	0.93	2.95	3.691(4)	138

Symmetry codes: (i): 1-x, 1/2+y, 1/2-z; (ii): -1+x, 1/2-y, 1/2+z

There are no classical intramolecular or intermolecular hydrogen bonds in the molecular structure. However, π…ring interactions have been observed between the phenyl carbon atoms and the phenyl ring centers, which allow the molecular structure to be stacked in space. The C–H…Cg/π intermolecular hydrogen bonds are also effective in crystal packing, as shown in Figure 4.5 and Table 4.3. These hydrogen bonds are between the C(4)/C(26) donor carbon atoms in the phenyl rings of the molecular structure with the (x, y, z) symmetry code and the acceptor of another phenyl ring centers (π) of the molecular structure with (1-x, 1/2+y, 1/2-z) and (-1+x, 1/2-y,1/2+z) symmetry codes. The C(4)−H(4)...Cg(6) hydrogen bond repeats itself along the b direction of the unit cell, forming the chain structure.

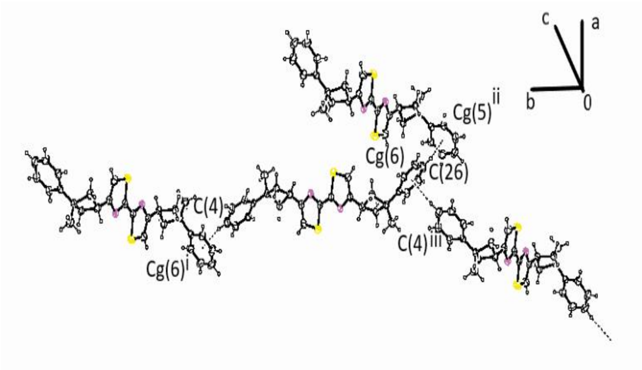

Figure 4.5 The crystal packing of the C28H28N2S2 compound

CONCLUSIONS AND RECOMMENDATION

Conclusions

4,4'-Bis(3-phenyl-3-methylcyclobutyl)-2,2'-bithiazole (C28H28N2S2) compound was synthesized and “its molecular structure was characterized. The molecular structure was characterized” by IR, 1H NMR, and 13C NMR techniques. In addition, the single crystal structure of the C28H28N2S2 molecular structure was analyzed by XRD. According to XRD results, the molecular structure crystallized in the monoclinic crystal system and the P21/c space group. The unit cell parameters of the molecular structure a= 5.8461(6)Å, b= 35.806(4) Å, c= 11.6538(11) Å, α=γ= 90° and β= 93.774(8)°, z =4, and (volume) V= 2434.2(4) Å3 from the XRD analysis results. The molecular structure creates a symmetrical molecular structure in an asymmetric unit with the (C(14)-C(15)) bond between the carbon atoms in the thiazole rings. Considering the dihedral angles between the thiazole, cyclobutane, and phenyl rings in the molecular structure, we can say that the molecular structure is not planar. The bending of 27.49(23)° and 21.08(24)° in the cyclobutane rings is similar when compared to the literature. In addition, the bond “parameters in the molecular structure were compared with similar studies in the literature and were found to be compatible. According to the hydrogen bond analysis of the molecular structure”, we can say about the existence of π…ring interaction between the carbon atoms and the phenyl ring centers. This hydrogen bond type interaction repeats itself in three-dimensional space and forms the crystal structure.

Recommendation

If the crystal structure data of the molecular structure in this thesis study is appropriate, it can be re-examined with possible different space groups. In addition, many physical and chemical properties of the molecular structure can be investigated with the help of theoretical calculations. Structure-enzyme studies can be investigated and explained by different methods. In addition, the biological activity of the molecular structure can be examined.

REFERENCES

1. Allen, F. H. 1984. The geometry of small rings VI: Geometry and bonding in cyclobutane and cyclobutene. Acta Cryst. B40: 64-72.
2. Blake, A. J., Cole, J. M., Evans, J. S., Main, P., Parsons, S., & Watkin, D. J. 2009. Crystal structure analysis: principles and practice (Vol. 13). OUP Oxford.
3. Bragg, W. H. 1915. IX. Bakerian Lecture.—X-rays and crystal structure. Philosophical Transactions of the Royal Society of London. Series A, Containing Papers of a Mathematical or Physical Character, 215(523-537): 253-274.
4. Bragg, W. H., & Bragg, W. L. 1913. The reflection of X-rays by crystals. Proceedings of the Royal Society of London. Series A, Containing Papers of a Mathematical and Physical Character, 88(605): 428-438.
5. Buerger, M. J. 1960. Crystal Structure Analysis, John Wiley and Sons Inc., New York, 110-115.
6. Charnes, A., Frome, E. L., & Yu, P. L. 1976. The equivalence of generalized least squares and maximum likelihood estimates in the exponential family. Journal of the American Statistical Association, 71(353): 169-171.
7. Crews, P., Kakou, Y., & Quinoa, E. 1988. Mycothiazole, a polyketide heterocycle from a marine sponge. J. Amer. Chem. Soc. 110(13): 4365–4369.
8. Cullity, B. D. 1956. X-Ray Diffraction Elements of X-ray Diffraction, Addison- Wesley, Pub.com, USA.
9. De Gelder, R., Israël, R., Lam, E. J., Beurskens, P. T., van Smaalen, S., Fu, Z. Q., & Fan, H. F. 1996. Direct methods for incommensurately modulated structures. On the applicability of normalized structure factors. Acta Crystallographica Section A: Foundations of Crystallography, 52(6): 947-954.
10. Farrugia, L. J. 2012. WinGX and ORTEP for Windows: an update. J. Appl. Cryst. 45: 849-854.
11. Förster, A., Brandstetter, S., & Schulze-Briese, C. 2019. Transforming X-ray detection with hybrid photon counting detectors. Philosophical Transactions of the Royal Society A, 377(2147): 20180241.
12. Giacovazzo, C., Monaco, H. L., Viterbo, D., Scordari, F., Gilli, G., Zanotti, G., & Catti M. 1995. Fundamentals of Crystallography International Union of Crystallography Oxford University Press.
13. Gullikson, E. M., Korde, R., Canfield, L. R., & Vest, R. E. 1996. Stable silicon photodiodes for absolute intensity measurements in the VUV and soft x-ray regions. Journal of Electron Spectroscopy and Related Phenomena, 80: 313-316.
14. Güneş, B. 2016. Kristalografi Ders Notları. http://bilalgunes.com (Erişim tarihi: 22/04/2016)
15. Hantzsch, A., & Weber, J. H. 1887. Ueber verbindungen des thiazols (pyridins der thiophenreihe). Berichte der deutschen chemischen Gesellschaft, 20(2): 3118-3132.
16. Henderson, R., & Moffat, J. K. 1971. The difference Fourier technique in protein crystallography: errors and their treatment. Acta Crystallographica Section B: Structural Crystallography and Crystal Chemistry, 27(7): 1414-1420.
17. Kasim, N. A. M., & Yamin, B. M. 2005. 5-Acetyl-4-methyl-2-phenylamino-1, 3-thiazole. Acta Crystallographica Section E: Structure Reports Online, 61(11): o3794-o3795.
18. Kittel, C., McEuen, P., & McEuen, P. 1996. Introduction to solid state physics (Vol. 8, pp. 105-130). New York: Wiley.
19. Kotadiya, M. 2017. Thiazoles: Past, present and future. International Journal of Advancement in Engineering Technology, Management and Applied Science, 4(1): 24-44.
20. Kumar, P. M., Badrinarayanan, S., & Sastry, M. 2000. Nanocrystalline TiO2 studied by optical, FTIR and X-ray photoelectron spectroscopy: correlation to presence of surface states. Thin solid films, 358(1-2): 122-130.
21. Ladd, M. F. C., & Palmer, R. A. 1985. Methods in X-Ray Structure Analysis. II. In Structure Determination by X-Ray Crystallography (pp. 213-294). Springer, Boston, MA.
22. Lukin, O. 2020. Explore Our Novel Cyclobutane Derivatives. Life Chemical Press.
23. Metzger, J. V. 1984. Thiazoles and their Benzo Derivatives, edited by K. T. Potts, Vol. 6. New York: Pergamon Press.
24. Muller, P., Herbst-Irmer, R., Spek, A., Schneider, T., & Sawaya, M. 2006. Crystal Structure Refinement: A Crystallographer's Guide to SHELXL (Vol. 8). OUP Oxford.
25. Roessle, M. 2012. Basics of X-ray Scattering. EMBO course, 2009.
26. Santiago, M. 2017. Introduction to X-ray Diffractionmeter. Melalui http://geology. uprm. edu/facilities/Class% 20Forms/xrd. pdf.
27. Saraçoğlu, H., & Cukurovali, A. 2012. An experimental and theoretical approach to the molecular structure of 3-{[4-(3-Methyl-3-phenyl-cyclobutyl)-thiazol-2-yl]-hydrazono}-1, 3-dihydro-indol-2-one. International Journal of Quantum Chemistry, 112(6): 1566-1578.
28. Schenk, H. 2022. Structure Solution. Department of Chemistry & Biochemistry Chemical Crystallography Laboratory.
29. Sheldrick, G. M. 1993. SHELXL93 Program for Refinement of Crystal Structures, University of Göttingen, Germany.
30. Sheldrick, G. M. 1997. SHELXL97-A Program Manual for the Refinement of Crystal Structures, University of Göttingen, Germany, 15-25.
31. Sheldrick, G. M. 2015. Crystal structure refinement with SHELXL. Acta Cryst. A71: 3-8.

32. Smart, 2015. Apex II User Manual Version, 2015.
33. Spek, A. L. 2009. Structure validation in chemical crystallography. Acta Cryst. D65: 148-155.
34. Stoe & Cie GmbH. 2014. Available on: https://www.stoe.com/pages/about.html
35. Stout, G. H. & Jensen, L. H. 1989. X-Ray Determination, John Wiley and Sons., New York, Chischester, Brisbone, Toronto, Singapere, 453-458.
36. Su, H. L., Sredojevic, D. N., Bronstein, H., Marks, T. J., Schroeder, B. C., & Al-Hashimi, M. 2017. Bithiazole: An Intriguing Electron-Deficient Building for Plastic Electronic Applications. Macromolecular Rapid Communications, 38(10): 1600610.
37. Wang, M., & Lu, P. 2018. Catalytic approaches to assemble cyclobutane motifs in natural product synthesis. Organic Chemistry Frontiers, 5(2): 254-259.
38. Whaites, E., & Drage, N. 2013. Essentials of dental radiography and radiology. Elsevier Health Sciences.
39. Yuksektepe, C., Saracoglu, H., Caliskan, N., Yilmaz, I., & Cukurovali, A. 2010. Experimental and ab initio Computational Studies on Dimethyl-(4-{4-{3-methyl-3-phenyl-cyclobutyl)-thiazol-2-yl]-hydrazonomethyl}-phenyl)-amine. Bulletin of the Korean Chemical Society, 31(12): 3553-3560.
40. Zink, F. E. 1997. X-ray tubes. Radiographics, 17(5): 1259-1268.