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Crystal and Molecular Structure Analysis of 1-Acryloyl-3-methyl-2,6-bis(3,4,5-trimethoxyphenyl)piperidine-4-one

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The title compound $C_{27}H_{33}NO_8$ crystallizes in the orthorhombic crystal class under the space group Pbca with cell parameters a = 13.775(4) Å, b = 16.389(2) Å, c = 23.085(6) Å, Z = 8, and V = 5212.6(3) Å^3. The piperidine ring in the structure adopts a twist boat conformation. The bond lengths and bond angles are in good agreement with the standard values. The structure exhibits intermolecular hydrogen bond of the type C–H···O. The synthesized molecule was characterized by FT-IR, ^{1}H-NMR, MS, and elemental analysis.

Keywords: antifungal and tranquilizers; antimicrobial; piperidone

INTRODUCTION

Piperdines are an important group of compounds in the field of medicinal chemistry owing to the fact that these can frequently be recognized in the structure of numerous naturally occurring alkaloid and synthetic compounds with interesting biological and pharmacological properties. This has been reviewed by Prostakov and Gaivoronskaya [1]. A good number of reports are there to prove that the piperidine derivatives have antimicrobial [2], analgesic [3], local anaesthetic [4], and anti-fungal activities [5]. Further, the 2,6-substituted piperdines are useful as tranquilizers and possess hypotensive activity. In view of the above, 1-acryloyl-3-methyl-1-propionyl-2,6-bis

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(3,4,5-trimethoxy phenyl)piperidine-4-one was synthesized, and the crystal structure is reported here.

EXPERIMENTAL

IR spectra were recorded in JASCO FT-IR 4100 spectrophotometer with KBr only significant absorption levels (reciprocal centimeter) are listed. ^{1}H-NMR spectra were recorded on Bruker AMX-400 MHz High Resolution Multinuclear FT-NMR Spectrometer in Dimethyl Sulfoxide (DMSO) using tetramethyl silane (TMS) as internal standard. Mass spectra were recorded on a Waters-Q-TOF Ultimaspectophotometer. Elemental analysis (C, H, and N) of the synthesized compound was carried out on Elementar Vario EL III CHN_2 analyzer. The crystallographic measurements were made on DIPLabo Imaging plate system with graphite monochromated radiation (MoK_α).

Synthesis of 1-Acryloyl-3-methyl-2,6-bis (3,4,5-Tri methoxy Phenyl) Piperidine-4-one

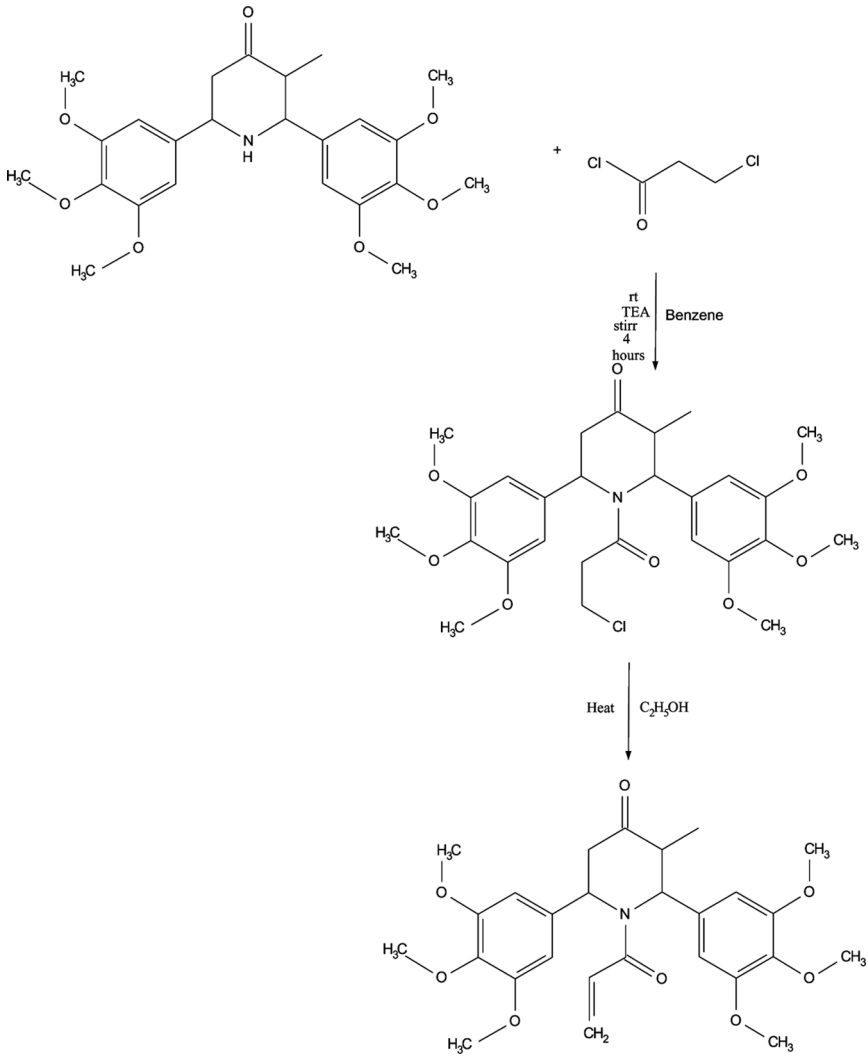
To a well-stirred solution of 3-methyl-2,6-bis(3,4,5-trimethoxy phenyl)piperidine-4-one (5 mmol) and triethylamine (5 mmol) in 30 ml of benzene, 3-chloropropanoyl chloride (5 mmol) in 20 ml benzene was added dropwise through the addition funnel for about an hour. The resulting mixture was stirred for about 4 hours under room condition. After the completion of the reaction the mixture was quenched in cold water, and the organic layer was extracted into ethyl acetate, washed with 5% sodium bicarbonate solution and dried over anhydrous sodium sulphate. This upon evaporation and recrystallization in alcohol yields 2,6-bis(3,4,5-trimethoxy phenyl) piperidine-4-one. Further, the sample was dissolved in ethanol (60 ml), refluxed for half an hour and allowed to crystallize by slow evaporation to form the title compound (Scheme 1). The schematic diagram of the molecule is shown in Figure 1.

IR (KBr) (cm^{-1}): 3184.2, 3076.0, 2996.0, 2888.0, 28321.1 (C–H stretching); 1724.9.6 (C=O stretching); 1643.9 (N–C=O stretching); 1586.4, 1508.8, 1454.5, 1426.6, 1323.4, 1122.2, 1036.1, 915.8, 840.2, 690.2.

^{1}H NMR (δ, ppm); 3.11 (t, CH, 2 H, C_{5a}, and C_3); 2.75 (dd, CH, 1 H, C_{5b}); 3.61 (d, CH_2, CH, CH_2); 2.556 (d, CH, H, CH–C=O); 3.66 and 3.78 (2 S, OCH_3, 18 H, 6 OCH_3 in benzene ring); 5.19 (s, CH, 1 H, C_2); 5.79 (s, CH, 1H, C_6) 6.53 (s Ar-H, 4 H, benzene ring); 1.15(d, CH_3, 3H, CH_3 at C_3).

Mass (m/z): 500.00.

Elemental Analysis: C, 64.892; H, 6.61; N, 2.83; O, 25.53.



SCHEME 1 The reaction scheme.

CRYSTAL STRUCTURE DETERMINATION

A single crystal of the title compound with dimensions $0.27 \times 0.25 \times 0.21$ mm was chosen for X-ray diffraction study. The data were collected on a DIPLabo Image Plate system equipped with a normal focus, 3 kW sealed X-ray source (graphite monochromated MoK_{α}).

The crystal to detector distance was fixed at 120 mm with a detector area of $441 \times 240\,mm^2$. Thirty six frames of data were collected at room temperature by the oscillation method. Each exposure of the image plate was set to a period of 400 s. Successive frames were scanned in steps of 5° per minute with an oscillation range of 5°. Image processing and data reduction were done using Denzo [6]. The reflections were merged with Scalepack [7]. All the frames could be indexed using a primitive orthorhombic lattice. Absorption correction was not applied. The structure was solved by direct methods using SHELXS-97 [8]. All the non-hydrogen atoms were revealed in the first Fourier map itself. Full matrix least squares refinement using SHELXL-97 [8] with isotropic displacement factors for all the non-hydrogen atoms converged the residual to 0.1865. Subsequent refinements were carried

TABLE 1 Crystal Data and Structure Refinement Table

CCDC Deposition Number	CCDC 725678
Empirical formula	$C_{27}H_{33}NO_8$
Formula weight	499.54
Temperature	293 (2) K
Wavelength	0.71073 Å
Crystal system	Orthorhombic
Space group	*Pbca*
Cell dimensions	$a = 13.775(4)$ Å
	$b = 16.389(2)$ Å
	$c = 23.085(6)$ Å
Volume	5212(2) Å^3
Z	4
Density (calculated)	1.273 Mg/m^3
Absorption coefficient	0.094 mm^{-1}
F_{000}	2128
Crystal Size	$0.27 \times 0.25 \times 0.21$ mm
Theta range for data collection	2.62° to 25.02°
Index ranges	$-14 \leq h \leq 14$
	$-18 \leq k \leq 17$
	$-27 \leq l \leq 27$
Reflections collected	6343
Independent reflections	3711 [R(int) = 0.0460]
Absorption correction	None
Refinement method	Full-matrix least-squares on F^2
Data/restraints/parameters	3711/0/333
Goodness-of-fit on F^2	1.079
Final R indices [$I > 2\sigma(I)$]	$R1 = 0.0694$, $wR2 = 0.1704$
R indices (all data)	$R1 = 0.1163$, $wR2 = 0.1984$
Extinction coefficient	0.0088(1)
Largest diff. peak and hole	0.249 and –0.210 eÅ^{-3}

out with anisotropic thermal parameters for non-hydrogen atoms and isotropic displacement factors for the hydrogen atoms which were placed at chemically acceptable positions. The residual finally converged to 0.0694. The highest peak and the deepest hole in the final difference map were 0.249 and $-0.210\ e^{-3}$, respectively.

TABLE 2 Atomic Coordinates and Equivalent Thermal Parameters of the Non-hydrogen Atoms

Atom	x	y	z	U_{eq}
N1	1.3983(2)	0.2512(2)	0.36674(1)	0.0552(8)
C2	1.3485(3)	0.2800(2)	0.31336(1)	0.0556(1)
C3	1.3657(3)	0.3714(3)	0.30317(2)	0.0688(1)
C4	1.4709(4)	0.3947(3)	0.31037(2)	0.0848(1)
C5	1.5239(3)	0.3555(3)	0.35940(2)	0.0780(1)
C6	1.4596(3)	0.3076(2)	0.40093(1)	0.0603(1)
O7	1.5117(3)	0.4414(3)	0.27818(2)	0.1421(2)
C8	1.3247(4)	0.3984(3)	0.2448(2)	0.1059(1)
C9	1.3990(3)	0.1711(3)	0.38239(1)	0.0615(1)
O10	1.4340(2)	0.14768(2)	0.42864(1)	0.0843(1)
C11	1.3584(3)	0.1091(3)	0.34193(2)	0.0668(1)
C12	1.3368(5)	0.0361(3)	0.3586(3)	0.125(2)
C13	1.2405(3)	0.2617(2)	0.31405(1)	0.0537(1)
C14	1.1823(3)	0.2879(2)	0.35969(1)	0.0554(1)
C15	1.0839(3)	0.2714(2)	0.35970(1)	0.0594(1)
C16	1.0422(3)	0.2278(2)	0.31460(1)	0.0588(1)
C17	1.0996(3)	0.2033(2)	0.26857(1)	0.0591(1)
C18	1.1980(3)	0.2203(2)	0.26813(1)	0.0581(1)
O19	1.02122(2)	0.29375(2)	0.40324(1)	0.0711(9)
C20	1.0584(3)	0.3431(3)	0.44871(2)	0.0794(1)
O21	0.9434(2)	0.21064(2)	0.31536(1)	0.0704(8)
C22	0.9208(3)	0.1283(3)	0.33177(2)	0.0896(1)
O23	1.0529(2)	0.16092(2)	0.22555(1)	0.0753(9)
C25	1.4005(3)	0.3568(3)	0.44499(1)	0.0591(1)
C24	1.1102(3)	0.1190(3)	0.18443(1)	0.0766(1)
C26	1.3989(3)	0.4411(3)	0.44670(1)	0.0673(1)
C27	1.3461(3)	0.4812(3)	0.48996(2)	0.0675(1)
C28	1.2937(3)	0.4372(2)	0.53147(1)	0.0623(1)
C29	1.2970(3)	0.3526(3)	0.52949(1)	0.0620(1)
C30	1.3492(3)	0.3120(2)	0.48667(1)	0.0591(1)
O31	1.3403(2)	0.56406(2)	0.49563(1)	0.0889(1)
C32	1.3888(4)	0.6128(3)	0.4545(2)	0.0996(1)
O33	1.2453(2)	0.47640(2)	0.57552(1)	0.0772(9)
C34	1.1515(4)	0.5062(3)	0.56022(2)	0.0913(16)
O35	1.2454(2)	0.31231(2)	0.57164(1)	0.0731(9)
C36	1.2662(4)	0.2278(3)	0.58071(2)	0.0807(1)

$U_{eq} = (1/3)\sum_i \sum_j U_{ij}\left(a_i^* a_j^*\right)(\mathbf{a_i} \cdot \mathbf{a_j})$.

RESULTS AND DISCUSSION

The details of crystal data and refinement are given in Table 1. Table 2 gives the list of atomic coordinates and equivalent thermal parameters of the non-hydrogen atoms. Table 3 gives the list of anisotropic thermal parameters of the non-hydrogen atoms. The bond lengths and bond angles of all the non-hydrogen atoms are given in Table 4. and are in good agreement with the standard values [9]. Figure 2

TABLE 3 Anisotropic Thermal Parameters of the Non-hydrogen Atoms

Atom	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
N1	0.056(2)	0.069(2)	0.0405(2)	0.0030(2)	0.0037(1)	−0.0029(1)
C2	0.060(3)	0.069(3)	0.0378(2)	0.001(2)	0.0080(2)	−0.0009(2)
C3	0.084(4)	0.071(3)	0.052(2)	0.003(2)	0.012(2)	0.0067(2)
C4	0.101(4)	0.086(4)	0.067(3)	−0.023(3)	0.024(3)	−0.008(2)
C5	0.068(3)	0.097(4)	0.069(3)	−0.011(2)	0.020(2)	−0.024(2)
C6	0.048(3)	0.079(3)	0.054(2)	−0.003(2)	0.0048(2)	−0.0134(2)
O7	0.166(4)	0.151(4)	0.110(3)	−0.075(3)	0.020(3)	0.032(3)
C8	0.148(5)	0.097(4)	0.073(3)	−0.002(3)	0.006(3)	0.028(3)
C9	0.059(3)	0.083(3)	0.042(2)	0.007(2)	−0.0002(2)	−0.001(2)
O10	0.107(3)	0.086(2)	0.0599(2)	0.0168(2)	−0.0182(2)	−0.0010(1)
C11	0.068(3)	0.073(3)	0.060(2)	0.004(2)	−0.006(2)	0.004(2)
C12	0.176(6)	0.089(4)	0.109(4)	−0.020(4)	−0.038(4)	0.014(3)
C13	0.053(3)	0.070(3)	0.0381(2)	0.0056(2)	0.0037(2)	0.0049(2)
C14	0.056(3)	0.072(3)	0.0382(2)	0.006(2)	0.0037(2)	0.0005(2)
C15	0.059(3)	0.083(3)	0.037(2)	0.014(2)	0.0073(2)	0.0031(2)
C16	0.050(3)	0.086(3)	0.040(2)	0.013(2)	0.0016(2)	0.0097(2)
C17	0.055(3)	0.086(3)	0.037(2)	0.006(2)	−0.0046(2)	0.0015(2)
C18	0.062(3)	0.078(3)	0.0336(2)	0.011(2)	0.0052(2)	−0.0007(2)
O19	0.059(2)	0.107(2)	0.0471(1)	0.0087(1)	0.0103(1)	−0.0106(1)
C20	0.083(4)	0.097(3)	0.058(3)	0.006(3)	0.019(2)	−0.020(2)
O21	0.054(2)	0.103(2)	0.0548(2)	0.0101(2)	−0.0015(1)	0.0034(1)
C22	0.068(3)	0.123(4)	0.077(3)	−0.004(3)	−0.007(2)	0.023(3)
O23	0.064(2)	0.118(3)	0.0436(1)	0.0070(2)	−0.0036(1)	−0.0170(1)
C25	0.056(3)	0.078(3)	0.043(2)	−0.002(2)	0.0008(2)	−0.0103(2)
C24	0.079(3)	0.094(3)	0.058(2)	0.008(2)	−0.005(2)	−0.017(2)
C26	0.078(3)	0.077(3)	0.047(2)	−0.007(2)	0.0098(2)	−0.0079(2)
C27	0.090(3)	0.063(3)	0.050(2)	−0.001(2)	0.004(2)	−0.0065(2)
C28	0.074(3)	0.067(3)	0.046(2)	0.005(2)	0.0061(2)	−0.0061(2)
C29	0.062(3)	0.081(3)	0.043(2)	−0.001(2)	0.0023(2)	−0.0003(2)
C30	0.067(3)	0.061(3)	0.050(2)	0.0016(2)	0.0021(2)	−0.0061(2)
O31	0.132(3)	0.066(2)	0.0690(2)	−0.0014(2)	0.0303(2)	−0.0039(1)
C32	0.148(5)	0.069(3)	0.081(3)	−0.014(3)	0.028(3)	0.002(2)
O33	0.097(3)	0.084(2)	0.0505(1)	0.0164(2)	0.0168(1)	−0.0083(1)
C34	0.094(4)	0.101(4)	0.078(3)	0.027(3)	0.027(3)	0.004(2)
O35	0.083(2)	0.080(2)	0.0562(2)	0.0051(1)	0.0165(1)	0.0043(1)
C36	0.089(4)	0.078(3)	0.074(3)	−0.001(3)	0.005(2)	0.008(2)

TABLE 4 Bond Lengths (Å) and Bond Angles (°)

Atoms	Length	Atoms	Length
N1-C9	1.361(5)	C16-C17	1.384(5)
N1-C6	1.481(5)	C16-O21	1.390(5)
N1-C2	1.487(4)	C17-O23	1.373(4)
C2-C13	1.518(5)	C17-C18	1.383(5)
C2-C3	1.535(5)	O19-C20	1.420(5)
C3-C4	1.508(6)	O21-C22	1.435(5)
C3-C8	1.528(6)	O23-C24	1.412(4)
C4-O7	1.206(5)	C25-C26	1.382(6)
C4-C5	1.492(6)	C25-C30	1.400(5)
C5-C6	1.523(5)	C26-C27	1.399(5)
C6-C25	1.532(5)	C27-031	1.366(5)
C9-O10	1.233(4)	C27-C28	1.400(5)
C9-C11	1.489(5)	C28-033	1.375(4)
C11-C12	1.292(6)	C28-C29	1.388(5)
C13-C18	1.388(5)	C29-035	1.374(4)
C13-C14	1.391(5)	C29-C30	1.391(5)
C14-C15	1.383(5)	O31-C32	1.408(5)
C15-O19	1.374(4)	O33-C34	1.426(5)
C15-C16	1.387(5)	O35-C36	1.430(5)
C9-N1-C6	117.2(3)	O19-C15-C16	115.2(4)
C9-N1-C2	121.9(3)	C17-C16-O21	120.8(3)
C6-N1-C2	120.4(3)	C15-C16-O21	120.0(3)
N1-C2-C13	112.4(3)	O23-C17-C18	123.8(3)
N1-C2-C3	111.4(3)	O23-C17-C16	115.7(4)
C15-O19-C20	117.8(3)	C16-O21-C22	113.9(3)
O7-C4-C5	120.9(5)	C17-O23-C24	118.1(3)
O7-C4-C3	122.7(5)	N1-C6-C5	108.6(3)
N1-C6-C25	112.3(3)	O31-C27-C26	124.5(3)
O31-C27-C28	114.6(3)	O10-C9-N1	122.2(3)
O10-C9-C11	118.5(4)	O33-C28-C29	120.5(3)
N1-C9-C11	119.3(3)	O33-C28-C27	121.0(4)
O35-C29-C28	116.1(3)	O35-C29-C30	122.8(4)
C15-C14-C13	120.3(3)	O19-C15-C14	124.3(3)

represents the OR-TEP [10] diagram of the molecule with thermal ellipsoids drawn at 50% probability. The piperidine ring is puckered. The ring-puckering analysis [11] of six-membered ring in the molecule indicates that the piperidine ring adopts twist boat conformation, with a puckering amplitude $Q = 0.650(4)$ Å, $\theta = 91.6(4)^\circ$, and $\phi = 111.9(4)^\circ$. Atoms C2 and C6 deviate from the plane (Cremer and Pople, 1975) defined by the atoms N1/C2/C3/C4/C5/C6 by $-0.224(4)$ Å and 0.379(4) Å, respectively. The piperdine ring in the molecule 1-acryloyl-3-methyl-1-propionyl-2,6-bis(3,4,5-trimethoxyphenyl)piperidine-4-one has a weighted average torsion angle of 33.26°. The substituent at

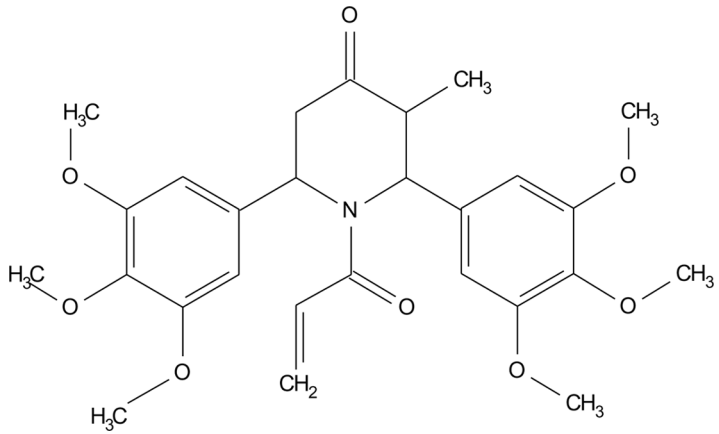


FIGURE 1 Schematic diagram of the title compound.

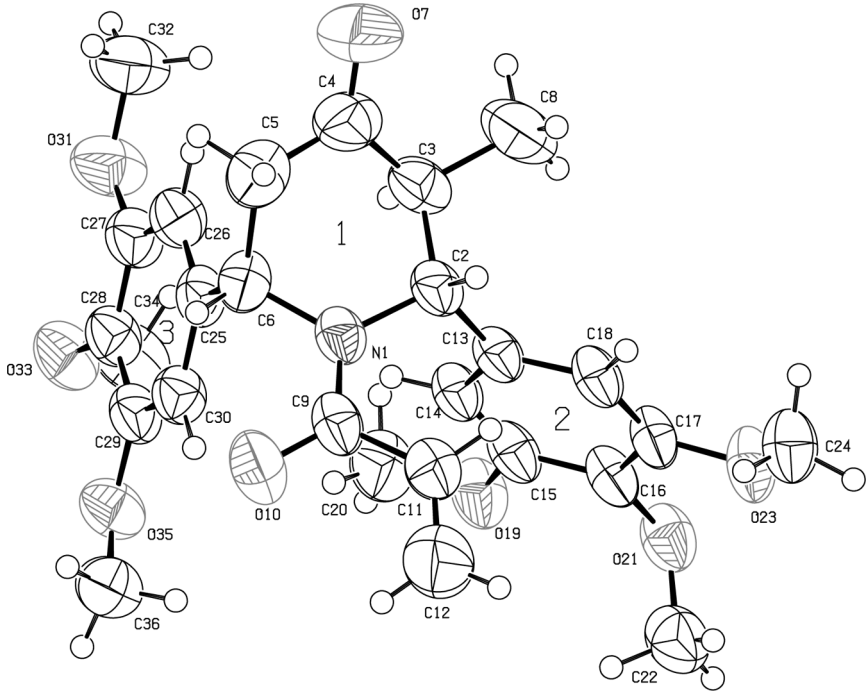


FIGURE 2 ORTEP of the molecule plot at 50% probability.

TABLE 5 Selected Torsion Angles (°)

Atoms	Angle
C2-N1-C9-O10	174.6(3)
C6-N1-C9-O10	12.9(5)
N1-C2-C9-C11	165.6(3)
N1-C2-C3-C8	173.3(3)
C28-C29-O35-C36	−164.2(3)
C28-C27-O31-C32	−178.2(4)
C24-O23-C17-C16	−166.1(3)
C20-O19-C15-C16	−176.0(4)
C27-C28-O33-C34	83.1(5)
C22-O21-C16-C17	78.8(4)

C6 exhibits equatorial conformation as indicated by the dihedral angle of 84.2(2)° between piperidine ring 1 and the phenyl ring 3 and the phenyl ring 2 and the piperidine ring 1 has dihedral angle of 71.68(2)°. The torsion angle value of 173.(3)° for N1-C6-C5-C8 indicates that methyl group substituted at C5 is oriented in *anti-periplanar* conformation. The methyl group substituted at the C5 position have been reported to act as active anitbacterial activity [12] against bacterial strains, i.e., Bacillus subtilis and Escherichia coli. The literature survey [12,13] shows that the presence of an alkyl group at the C5 position of the piperidone ring and also the attachment of methoxy group to the phenyl ring at C2 and C6 positions have reported to act as high antimicrobial activity [12]. The torsion angle value of 174.6(3)° for C2-N1-C9-O10 indicates that O10 is oriented in +*anti-periplanar* conformation. The acryloyl group substituted at N1 is oriented in +*syn-periplanr* conformation as indicated by the torsion angle value of 12.9(5)° for C6-N1-C9-C10. The methoxy groups substituted at C27, C29, C17, and C15 are nealry planar with the phenyl ring whereas the methoxy groups substitued at C28 exhibits orthogonal conformation as indicated by the torsion angles (Table 5). The molecules are linked into a linear one dimensional complex chains

TABLE 6 Hydrogen-bonding Geometry (Å)

D–H ··· A	D–H	H–A	D–A	D–H ··· A	Symmetry codes
C2-H2 ··· O21	0.98	2.48	3.440(4)	165	$1/2 + x, y, 1/2 - z$
C22-H22A ··· O7	0.96	2.59	3.432(6)	147	$5/2 - x, -1/2 + y, z$
C36-H36C ··· O19	0.96	2.59	3.550(5)	178	$1/2 + x, 1/2 - y, 1 - z$
C20-H20B ··· O10	0.96	2.41	3.313(5)	156	$-1/2 + x, 1/2 - y, 1 - z$

*The D–H and H–A distances are essentially standard values and are not derived from the experiment.

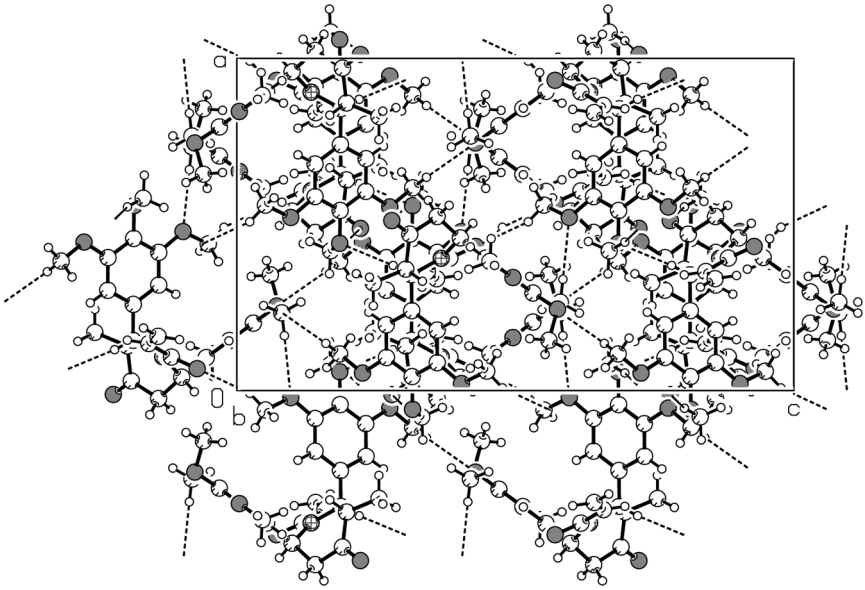


FIGURE 3 Packing of the molecules down *b* axis.

by weak intermolecular hydrogen bond of the type C–H · · · O as shown in Table 6.

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