

## Synthesis and Structural Analysis of 2-Amino-4-(4-hydroxy-3-methoxyphenyl)-7,9-dimethyl-5-oxo-4, 5, 6, 7-tetrahydropyrano [2, 3-d] pyrazolo [3, 4-b] pyridine-3-carbonitrile through X-ray Crystallography

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### Abstract

The crystal structure of the potential active 2-amino-4-(4-hydroxy-3-methoxyphenyl)-7, 9-dimethyl-5-oxo-4, 5, 6, 7-tetrahydropyrano [2, 3-d] pyrazolo [3, 4-b] pyridine-3-carbonitrile (C<sub>21</sub>H<sub>22</sub>N<sub>5</sub>O<sub>6</sub>S) has been determined from single crystal X-ray diffraction data. In the title compound crystallizes in the monoclinic space group P-1 with unit cell dimension a=8.1201(9)Å, b=12.2684(4)Å and c= 12.387(2)Å [ $\alpha=69.573^\circ$ ,  $\beta= 12.168^\circ$  and  $\gamma=76.060^\circ$ ]. In the structure the pyrazole, pyridine and pyran are almost coplanar each other. The crystal packing is stabilized by intermolecular C-H...O and N-H...O hydrogen bond interaction.

**Keywords:** Pyrazole; Pyran; Single Crystal Structure; X-ray Diffraction

### 1. Introduction

Pyrazole and its derivatives, a class of well known nitrogen containing heterocyclic compounds, occupy an important position in medicinal and pesticide chemistry with having a wide range of bioactivities such as antimicrobial<sup>[1]</sup>, anticancer<sup>[2]</sup>, anti-inflammatory, antidepressant, anticonvulsant, antihyperglycemic<sup>[3]</sup>, antipyretic, antifungal activities<sup>[4]</sup>, CNS regulators<sup>[5]</sup> and selective enzyme inhibitory activities.

It has been found that these compounds have hypoglycemic activity, and are also known as inhibitors and deactivators of liver alcohol dehydrogenase and oxidoreductases<sup>[6]</sup>. It has been shown in vivo that some of the pyrazole derivatives have appreciable antihypertensive activity.

The 1-phenylpyrazole motif is present in several drug candidates for treatment of various diseases such as cyclooxygenase-2 (Cox-2) inhibitors, IL-1 synthesis inhibitors, and protein kinase inhibitors etc. Similarly, some of the 1,5-diarylpyrazole derivatives have been shown to exhibit non-nucleoside HIV-1 reverse tran-

scriptase inhibitor activities along with Cox-2 inhibitor activity<sup>[7]</sup>.

Literature survey revealed that pyrazole derivatives possess diverse pharmacological activities. Pyrazole derivatives have a long history of application in agrochemicals and pharmaceutical industry as herbicides and active pharmaceuticals. The recent success of a pyrazole COX-2 inhibitor has further highlighted the importance of these heterocyclic rings in medicinal chemistry. The prevalence of pyrazole cores in biologically active molecules has stimulated the need for elegant and efficient ways to make these heterocyclic leads.

Based on the above facts, the X-ray crystal structures of pyrazole derivatives have been elucidated. X-ray crystallographic studies of the following one such compound have been carried out to obtain detailed information on the molecular conformation in the solid state. The IUPAC name and chemical diagram of the compounds are given in Fig. 1.

### 2. Material and Methods

With the collaboration of Chemistry Department at Pondicherry University, we obtained the title compound and crystallized by simple solvent slow evaporation method. Three round of crystallization trials, diffraction

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crystals. The diffraction quality crystals after screening its size and stability, X-ray diffraction data collection was done at Pondicherry University. The data was reduced with appropriate corrections at the facility and the error free data was taken for structure determination.

Using WinGx suite, structure determination was done using SHELXS97 with Direct Methods protocols. After manual inspections and corrections, Isotropic refinement followed by anisotropic refinements was carried out. With the satisfied model (agreeable R factor, Goodness of Fit and other) hydrogen atoms were geometrically fixed and after the final refinement the R factor is 8.0%.

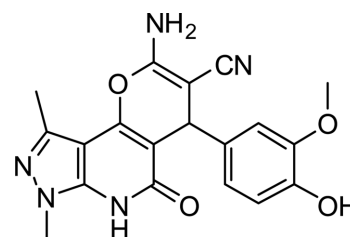
### 3. Experimental Section

#### 3.1. Synthesis of the Title Compound

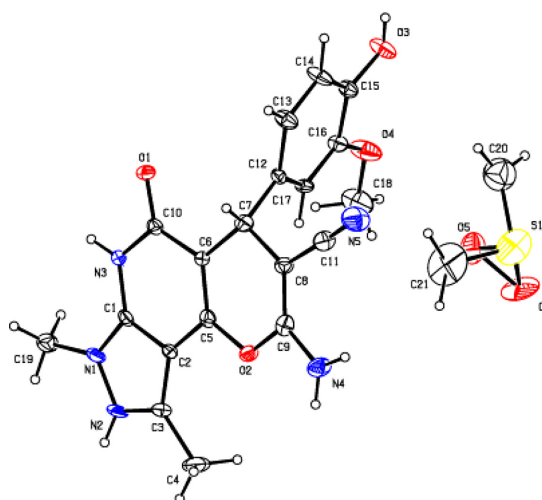
To an aqueous mixture of 4-hydroxy-1, 3-dimethyl-1H-pyrazolo [3,4-b]pyridin-6(7H)-one (2 mmol), 4-hydroxy-3-methoxy benzaldehyde (2 mmol), malononitrile (2 mmol) and piperidine (10 mol%) were added successively at room temperature under an open atmosphere and vigorously stirred for 10 mins. The progress of the reaction was monitored by thin layer chromatography. The precipitated solid was filtered, washed with water (10 mL) and ethanol (5 mL) and then with ether (3 mL). The product was dried under vacuum. Since the compound has not yield the diffraction quality crystals initially, the compound has been recrystallized with ethanol by slow evaporation method to get better quality single crystals.

#### 3.2. X-Ray Crystallography

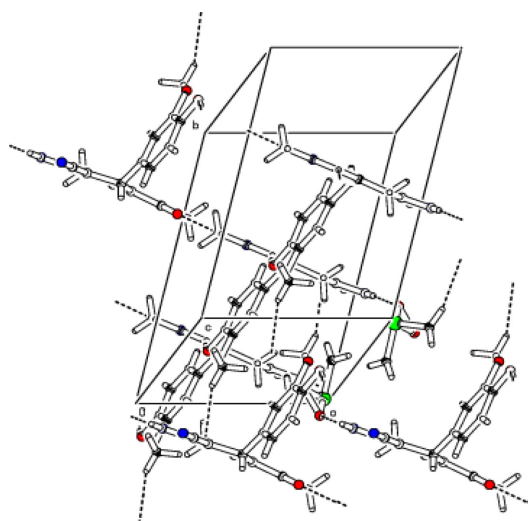
For the crystal structure determination, the single crystal of the compound  $C_{21}H_{22}N_5O_6S$  was used for data collection on a OXFORD diffractometer<sup>[8]</sup>. The  $MoK\alpha$  radiation of wavelength, ( $\lambda = 0.71073 \text{ \AA}$ ) and multi-scan technique for absorption correction were used for data collection. The lattice parameters were determined by the least-squares methods on the basis of all reflections with  $F_2 > 2\sigma(F_2)$ . The structures were solved by direct methods using SHELXS-97 and refined by a full-matrix least-squares procedure using the program SHELXL-97<sup>[9,10]</sup>. H atoms were positioned geometrically and refined using a riding model, fixing the aromatic C-H distances at  $0.93 \text{ \AA}$  [ $U_{iso}(H) = 1.2 U_{eq}(C)$ ]. The softwares used for Molecular graphics are ORTEP-3



**Fig. 1.** Chemical structure of 2-amino-4-(4-hydroxy-3-methoxyphenyl)-7,9-dimethyl-5-oxo-4,5,6,7-tetrahydropyrazolo [2,3-d] pyrazolo [3,4-b] pyridine-3-carbonitrile.



**Fig. 2.** Displacement ellipsoids are drawn at the 30% probability level.



**Fig. 3.** Crystal packing of the title compound, dashed line indicates the intermolecular interaction in the unit cell.

**Table 1.** Crystal data and structure refinement.

Empirical formula	C <sub>21</sub> H <sub>22</sub> N <sub>5</sub> O <sub>6</sub> S
Formula weight	472.50
Temperature	293(2) K
Wavelength	0.71073 Å
Crystal system, space group	Triclinic, P-1
Unit cell dimensions	a = 8.120 (9) Å, α = 69.573(1)° b = 12.268 (4) Å, β = 76.895(2)° c = 12.387 (2) Å, γ = 76.060(1)°
Volume	1108.5(2) Å <sup>3</sup>
Z, Calculated density	2, 1.416 Mg/m <sup>3</sup>
Absorption coefficient	0.195 mm <sup>-1</sup>
F(000)	494
Crystal size	0.35 × 0.20 × 0.25 mm
θ range for data collection	2.85 to 29.26°
Limiting indices	-8 ≤ h ≤ 10, -12 ≤ k ≤ 16, -12 ≤ l ≤ 17
Reflections collected / unique	1257 / 4110 [Rint = 0.024]
Refinement method	Full-matrix least-squares on F <sup>2</sup>
Data / restraints / parameters	4110 / 0 / 298
Goodness-of-fit on F <sup>2</sup>	0.807
Final R indices [I > 2σ(I)]	R1 = 0.0857, wR2 = 0.2218
R indices (all data)	R1 = 0.2650, wR2 = 0.2227
Largest diff. peak and hole	-0.607 and 0.42 e.Å <sup>-3</sup>

**Table 2.** Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å<sup>2</sup>)

	x	y	z	$U_{iso}^*/U_{eq}$
N5	0.0902 (8)	0.6193 (6)	0.8611 (5)	0.083 (2)
C19	0.9874 (8)	0.6389 (6)	0.1515 (5)	0.0543 (19)
H19A	0.9909	0.6684	0.0685	0.081*
H19B	1.0278	0.5551	0.1748	0.081*
H19C	1.0595	0.6767	0.1734	0.081*
C20	0.2409 (14)	1.0140 (10)	0.8507 (9)	0.129 (4)*
H20A	0.2647	0.9337	0.9024	0.155*
H20B	0.2805	1.0744	0.8669	0.155*
H7	0.486 (6)	0.541 (4)	0.696 (4)	0.000 (14)*
O2	0.3110 (5)	0.7111 (3)	0.4550 (3)	0.0382 (11)
N3	0.8347 (6)	0.5814 (4)	0.4150 (4)	0.0355 (12)
H3	0.9435	0.5555	0.4038	0.043*
O1	0.8243 (5)	0.5076 (3)	0.6101 (3)	0.0458 (12)
O3	0.7122 (6)	0.8354 (4)	0.9057 (3)	0.0619 (14)
H3A	0.7155	0.7944	0.9738	0.093*
C17	0.5872 (7)	0.7713 (5)	0.6758 (4)	0.0328 (15)
H17	0.5772	0.8095	0.5976	0.039*
S1	0.0750 (6)	1.0540 (4)	0.7717 (4)	0.1749 (17)
C8	0.2832 (8)	0.6417 (5)	0.6642 (5)	0.0380 (16)
C5	0.4841 (8)	0.6681 (5)	0.4490 (5)	0.0348 (15)
C6	0.5660 (7)	0.6132 (5)	0.5427 (4)	0.0295 (14)
C7	0.4757 (9)	0.6027 (6)	0.6640 (5)	0.0384 (18)
C10	0.7467 (8)	0.5649 (5)	0.5266 (5)	0.0336 (15)

**Table 2.** Continued

	x	y	z	$U_{iso}^*/U_{eq}$
N1	0.8121 (7)	0.6639 (4)	0.2087 (4)	0.0421 (13)
C2	0.5746 (8)	0.6846 (5)	0.3324 (5)	0.0333 (14)
O4	0.6885 (7)	0.9382 (4)	0.6885 (4)	0.0801 (18)
C9	0.2134 (8)	0.6921 (5)	0.5652 (6)	0.0413 (16)
C16	0.6447 (8)	0.8271 (5)	0.7343 (5)	0.0378 (16)
C12	0.5439 (7)	0.6618 (5)	0.7272 (4)	0.0290 (14)
C1	0.7497 (8)	0.6387 (5)	0.3219 (5)	0.0342 (15)
N2	0.6843 (7)	0.7214 (5)	0.1464 (4)	0.0487 (15)
H2	0.6949	0.7456	0.0715	0.058*
C3	0.5407 (9)	0.7346 (5)	0.2181 (5)	0.0432 (17)
C15	0.6596 (8)	0.7766 (5)	0.8489 (5)	0.0395 (16)
N4	0.0473 (7)	0.7343 (5)	0.5528 (4)	0.0567 (16)
H4A	-0.0304	0.7308	0.6134	0.068*
H4B	0.0190	0.7647	0.4842	0.068*
C13	0.5672 (9)	0.6065 (6)	0.8425 (5)	0.0515 (19)
H13	0.5431	0.5307	0.8802	0.062*
C11	0.1730 (9)	0.6301 (6)	0.7724 (6)	0.0524 (19)
C14	0.6257 (9)	0.6624 (6)	0.9027 (5)	0.060 (2)
H14	0.6425	0.6231	0.9796	0.072*
O5	0.2030 (8)	1.0381 (6)	0.7081 (6)	0.099 (2)
O6	0.0952 (9)	1.1502 (5)	0.6642 (5)	0.122 (3)
C18	0.6891 (13)	0.9990 (7)	0.5725 (7)	0.098 (3)
H18A	0.7244	1.0734	0.5550	0.148*
H18B	0.5757	1.0126	0.5542	0.148*
H18C	0.7677	0.9536	0.5268	0.148*
C4	0.3752 (9)	0.7982 (7)	0.1759 (6)	0.074 (2)
H4C	0.3927	0.8223	0.0922	0.111*
H4D	0.3344	0.8667	0.2016	0.111*
H4E	0.2920	0.7468	0.2066	0.111*
C21	0.0875 (16)	0.9226 (9)	0.7395 (12)	0.178 (6)
H21A	0.0217	0.9235	0.6829	0.213*
H21B	0.1221	0.8469	0.7960	0.213*

**Table 3.** Bond lengths [Å] and angles [°]

Bond length		Bond length	
N5—C11	1.132 (8)	C7—C12	1.492 (8)
C19—N1	1.452 (7)	C7—H7	0.71 (4)
C19—H19A	0.9600	N1—C1	1.329 (7)
C19—H19B	0.9600	N1—N2	1.342 (6)
C19—H19C	0.9600	C2—C1	1.392 (8)
C20—S1	1.719 (11)	C2—C3	1.394 (7)
C20—O5	1.771 (13)	O4—C18	1.368 (8)
C20—H20A	0.9700	O4—C16	1.382 (7)
C20—H20B	0.9700	C9—N4	1.346 (7)
O2—C5	1.372 (6)	C16—C15	1.357 (7)

**Table 3.** Continued

<b>Bond length</b>		<b>Bond length</b>	
O2—C9	1.386 (7)	C12—C13	1.386 (7)
N3—C1	1.364 (7)	N2—C3	1.310 (7)
N3—C10	1.375 (6)	N2—H2	0.8600
N3—H3	0.8600	C3—C4	1.489 (8)
O1—C10	1.239 (6)	C15—C14	1.391 (8)
O3—C15	1.354 (6)	N4—H4A	0.8600
O3—H3A	0.8200	N4—H4B	0.8600
C17—C16	1.366 (7)	C13—C14	1.388 (8)
C17—C12	1.365 (7)	C13—H13	0.9300
C17—H17	0.9300	C14—H14	0.9300
S1—O5	1.173 (6)	O5—O6	1.437 (8)
S1—O6	1.446 (6)	O5—C21	1.769 (11)
S1—C21	1.765 (11)	C18—H18A	0.9600
C8—C9	1.350 (8)	C18—H18B	0.9600
C8—C11	1.415 (9)	C18—H18C	0.9600
C8—C7	1.520 (8)	C4—H4C	0.9600
C5—C6	1.353 (7)	C4—H4D	0.9600
C5—C2	1.433 (7)	C4—H4E	0.9600
C6—C10	1.441 (7)	C21—H21A	0.9700
C6—C7	1.492 (8)	C21—H21B	0.9700
<b>Bond Angle</b>		<b>Bond Angle</b>	
N1—C19—H19A	109.5	C15—C16—C17	121.4 (5)
N1—C19—H19B	109.5	C15—C16—O4	112.7 (5)
H19A—C19—H19B	109.5	C17—C16—O4	125.9 (5)
N1—C19—H19C	109.5	C17—C12—C13	116.5 (5)
H19A—C19—H19C	109.5	C17—C12—C7	122.6 (5)
H19B—C19—H19C	109.5	C13—C12—C7	120.9 (5)
S1—C20—O5	39.2 (3)	N1—C1—N3	128.8 (6)
S1—C20—H20A	119.4	N1—C1—C2	107.6 (5)
O5—C20—H20A	119.4	N3—C1—C2	123.6 (5)
S1—C20—H20B	119.4	C3—N2—N1	108.9 (4)
O5—C20—H20B	119.4	C3—N2—H2	125.6
H20A—C20—H20B	117.1	N1—N2—H2	125.5
C5—O2—C9	117.2 (4)	N2—C3—C2	108.9 (5)
C1—N3—C10	120.1 (5)	N2—C3—C4	122.1 (5)
C1—N3—H3	120.0	C2—C3—C4	129.0 (6)
C10—N3—H3	120.0	O3—C15—C16	120.1 (5)
C15—O3—H3A	109.5	O3—C15—C14	122.3 (5)
C16—C17—C12	122.8 (5)	C16—C15—C14	117.5 (6)
C16—C17—H17	118.6	C9—N4—H4A	120.0
C12—C17—H17	118.6	C9—N4—H4B	120.0
O5—S1—O6	65.6 (5)	H4A—N4—H4B	120.0
O5—S1—C20	72.8 (6)	C12—C13—C14	121.2 (6)
O6—S1—C20	114.6 (6)	C12—C13—H13	119.4

**Table 3.** Continued

Bond Angle		Bond Angle	
O5—S1—C21	70.8 (6)	C14—C13—H13	119.4
O6—S1—C21	109.4 (6)	N5—C11—C8	177.4 (7)
C20—S1—C21	101.0 (6)	C15—C14—C13	120.6 (6)
C9—C8—C11	118.5 (6)	C15—C14—H14	119.7
C9—C8—C7	122.8 (6)	C13—C14—H14	119.7
C11—C8—C7	118.7 (5)	S1—O5—O6	66.4 (5)
C6—C5—O2	124.5 (5)	S1—O5—C20	68.0 (5)
C6—C5—C2	121.5 (6)	O6—O5—C20	112.1 (6)
O2—C5—C2	114.0 (5)	S1—O5—C21	70.5 (6)
C5—C6—C10	120.0 (5)	O6—O5—C21	109.7 (6)
C5—C6—C7	122.1 (6)	C20—O5—C21	98.8 (7)
C10—C6—C7	118.0 (5)	O5—O6—S1	48.0 (3)
C12—C7—C6	114.9 (5)	O4—C18—H18A	109.5
C12—C7—C8	112.6 (5)	O4—C18—H18B	109.5
C6—C7—C8	109.8 (5)	H18A—C18—H18B	109.5
C12—C7—H7	107 (4)	O4—C18—H18C	109.5
C6—C7—H7	107 (4)	H18A—C18—H18C	109.5
C8—C7—H7	105 (4)	H18B—C18—H18C	109.5
O1—C10—N3	119.2 (5)	C3—C4—H4C	109.5
O1—C10—C6	122.1 (5)	C3—C4—H4D	109.5
N3—C10—C6	118.8 (5)	H4C—C4—H4D	109.5
C1—N1—N2	109.5 (5)	C3—C4—H4E	109.5
C1—N1—C19	129.5 (5)	H4C—C4—H4E	109.5
N2—N1—C19	120.9 (5)	H4D—C4—H4E	109.5
C1—C2—C3	105.1 (5)	S1—C21—O5	38.8 (3)
C1—C2—C5	116.1 (5)	S1—C21—H21A	119.5
C3—C2—C5	138.9 (6)	O5—C21—H21A	119.5
C18—O4—C16	120.2 (6)	S1—C21—H21B	119.5
N4—C9—C8	128.9 (6)	O5—C21—H21B	119.5
N4—C9—O2	108.2 (5)	H21A—C21—H21B	117.1
C8—C9—O2	122.9 (6)		

**Table 4.** Anisotropic displacement parameters ( $\text{\AA}^2$ )

	$U^{11}$	$U^{22}$	$U^{33}$	$U^{12}$	$U^{13}$	$U^{23}$
N5	0.064 (5)	0.122 (6)	0.051 (4)	-0.018 (4)	0.005 (3)	-0.021 (4)
C19	0.044 (4)	0.078 (5)	0.040 (4)	-0.004 (4)	-0.006 (3)	-0.023 (3)
O2	0.031 (3)	0.044 (3)	0.037 (2)	-0.003 (2)	-0.0098 (19)	-0.0099 (19)
N3	0.037 (3)	0.039 (3)	0.032 (3)	0.008 (2)	-0.013 (2)	-0.018 (2)
O1	0.044 (3)	0.057 (3)	0.031 (2)	0.008 (2)	-0.016 (2)	-0.0127 (19)
O3	0.098 (4)	0.066 (3)	0.038 (2)	-0.037 (3)	-0.017 (2)	-0.016 (2)
C17	0.042 (4)	0.032 (4)	0.022 (3)	-0.007 (3)	-0.012 (3)	0.000 (3)
S1	0.162 (4)	0.156 (4)	0.195 (4)	0.018 (3)	-0.057 (3)	-0.052 (3)
C8	0.034 (4)	0.044 (4)	0.039 (4)	-0.007 (3)	-0.011 (3)	-0.013 (3)
C5	0.034 (4)	0.034 (4)	0.047 (4)	-0.010 (3)	-0.007 (3)	-0.022 (3)

**Table 4.** Continued

	$U^{11}$	$U^{22}$	$U^{33}$	$U^{12}$	$U^{13}$	$U^{23}$
C6	0.032 (4)	0.029 (3)	0.027 (3)	0.000 (3)	-0.008 (3)	-0.010 (2)
C7	0.054 (5)	0.025 (4)	0.033 (4)	-0.008 (4)	-0.012 (3)	-0.002 (3)
C10	0.042 (4)	0.030 (4)	0.034 (3)	-0.006 (3)	-0.011 (3)	-0.014 (3)
N1	0.043 (3)	0.062 (4)	0.028 (3)	-0.005 (3)	-0.011 (3)	-0.022 (2)
C2	0.040 (4)	0.032 (4)	0.037 (3)	-0.001 (3)	-0.017 (3)	-0.019 (3)
O4	0.137 (5)	0.064 (4)	0.052 (3)	-0.049 (3)	-0.040 (3)	0.002 (3)
C9	0.036 (4)	0.033 (4)	0.057 (4)	-0.014 (3)	-0.003 (3)	-0.015 (3)
C16	0.052 (4)	0.030 (4)	0.033 (3)	-0.015 (3)	-0.012 (3)	-0.003 (3)
C12	0.029 (3)	0.035 (4)	0.025 (3)	-0.007 (3)	-0.002 (2)	-0.013 (3)
C1	0.047 (4)	0.033 (4)	0.029 (3)	-0.006 (3)	-0.008 (3)	-0.017 (3)
N2	0.055 (4)	0.072 (4)	0.020 (3)	-0.003 (3)	-0.017 (3)	-0.015 (2)
C3	0.049 (4)	0.050 (4)	0.036 (3)	-0.002 (3)	-0.023 (3)	-0.015 (3)
C15	0.049 (4)	0.039 (4)	0.038 (4)	-0.016 (3)	-0.010 (3)	-0.013 (3)
N4	0.033 (3)	0.079 (4)	0.053 (3)	-0.007 (3)	-0.009 (3)	-0.015 (3)
C13	0.080 (5)	0.044 (4)	0.037 (4)	-0.027 (4)	-0.013 (3)	-0.008 (3)
C11	0.048 (5)	0.057 (5)	0.049 (4)	-0.009 (4)	-0.010 (4)	-0.011 (4)
C14	0.093 (6)	0.072 (5)	0.023 (3)	-0.036 (4)	-0.011 (3)	-0.012 (3)
O5	0.059 (4)	0.128 (6)	0.098 (5)	-0.002 (4)	0.019 (4)	-0.049 (4)
O6	0.173 (7)	0.062 (4)	0.102 (5)	-0.017 (4)	-0.080 (4)	0.044 (3)
C18	0.169 (10)	0.063 (6)	0.074 (6)	-0.045 (6)	-0.039 (6)	-0.006 (4)
C4	0.060 (5)	0.106 (6)	0.052 (4)	0.000 (5)	-0.032 (4)	-0.016 (4)
C21	0.212 (14)	0.061 (7)	0.302 (16)	-0.068 (8)	-0.086 (12)	-0.050 (8)

**Table 5.** Torsion angles [°]

Torsion Angle		Torsion Angle	
O5—C20—S1—O6	-51.8 (6)	N2—N1—C1—C2	-1.4 (6)
O5—C20—S1—C21	65.7 (6)	C19—N1—C1—C2	178.0 (5)
C9—O2—C5—C6	-2.8 (8)	C10—N3—C1—N1	179.3 (6)
C9—O2—C5—C2	176.3 (4)	C10—N3—C1—C2	0.9 (8)
O2—C5—C6—C10	176.1 (5)	C3—C2—C1—N1	1.5 (6)
C2—C5—C6—C10	-2.9 (8)	C5—C2—C1—N1	-179.0 (5)
O2—C5—C6—C7	-5.2 (9)	C3—C2—C1—N3	-179.8 (5)
C2—C5—C6—C7	175.7 (5)	C5—C2—C1—N3	-0.2 (8)
C5—C6—C7—C12	-118.3 (7)	C1—N1—N2—C3	0.7 (7)
C10—C6—C7—C12	60.4 (7)	C19—N1—N2—C3	-178.8 (5)
C5—C6—C7—C8	9.8 (8)	N1—N2—C3—C2	0.3 (7)
C10—C6—C7—C8	-171.5 (5)	N1—N2—C3—C4	178.3 (6)
C9—C8—C7—C12	121.4 (7)	C1—C2—C3—N2	-1.1 (7)
C11—C8—C7—C12	-55.3 (8)	C5—C2—C3—N2	179.5 (6)
C9—C8—C7—C6	-7.9 (9)	C1—C2—C3—C4	-178.9 (7)
C11—C8—C7—C6	175.4 (5)	C5—C2—C3—C4	1.7 (13)
C1—N3—C10—O1	176.5 (5)	C17—C16—C15—O3	-178.1 (6)
C1—N3—C10—C6	-2.4 (7)	O4—C16—C15—O3	0.2 (9)
C5—C6—C10—O1	-175.5 (6)	C17—C16—C15—C14	4.4 (10)

**Table 5.** Continued

Torsion Angle		Torsion Angle	
C7—C6—C10—O1	5.8 (8)	O4—C16—C15—C14	-177.3 (6)
C5—C6—C10—N3	3.5 (8)	C17—C12—C13—C14	2.2 (10)
C7—C6—C10—N3	-175.2 (5)	C7—C12—C13—C14	-178.1 (6)
C6—C5—C2—C1	1.3 (8)	C9—C8—C11—N5	-174 (16)
O2—C5—C2—C1	-177.8 (5)	C7—C8—C11—N5	3 (17)
C6—C5—C2—C3	-179.4 (7)	O3—C15—C14—C13	178.0 (6)
O2—C5—C2—C3	1.5 (10)	C16—C15—C14—C13	-4.5 (10)
C11—C8—C9—N4	-0.6 (10)	C12—C13—C14—C15	1.2 (11)
C7—C8—C9—N4	-177.3 (6)	C20—S1—O5—O6	-128.3 (6)
C11—C8—C9—O2	177.8 (5)	C21—S1—O5—O6	123.0 (6)
C7—C8—C9—O2	1.0 (10)	O6—S1—O5—C20	128.3 (6)
C5—O2—C9—N4	-176.5 (5)	C21—S1—O5—C20	-108.6 (6)
C5—O2—C9—C8	4.8 (8)	O6—S1—O5—C21	-123.0 (6)
C12—C17—C16—C15	-1.0 (10)	C20—S1—O5—C21	108.6 (7)
C12—C17—C16—O4	-179.1 (6)	S1—C20—O5—O6	50.9 (5)
C18—O4—C16—C15	176.6 (7)	S1—C20—O5—C21	-64.6 (6)
C18—O4—C16—C17	-5.2 (11)	C20—O5—O6—S1	-51.7 (6)
C16—C17—C12—C13	-2.4 (9)	C21—O5—O6—S1	57.0 (6)
C16—C17—C12—C7	177.9 (6)	C20—S1—O6—O5	55.5 (7)
C6—C7—C12—C17	43.0 (8)	C21—S1—O6—O5	-57.1 (6)
C8—C7—C12—C17	-83.7 (7)	O6—S1—C21—O5	54.0 (6)
C6—C7—C12—C13	-136.7 (6)	C20—S1—C21—O5	-67.2 (6)
C8—C7—C12—C13	96.6 (7)	O6—O5—C21—S1	-54.7 (6)
N2—N1—C1—N3	180.0 (6)	C20—O5—C21—S1	62.7 (5)
C19—N1—C1—N3	-0.6 (10)		

**Table 6.** Hydrogen-bond geometry (Å, °)

<i>D</i> —H... <i>A</i>	<i>D</i> —H(Å)	H... <i>A</i> (Å)	<i>D</i> ... <i>A</i> (Å)	<i>D</i> —H... <i>A</i> (°)
N2—H2...O3 <sup>i</sup>	0.86	1.95	2.798 (4)	167
N3—H3...O1 <sup>ii</sup>	0.86	1.85	2.710(5)	176
C18—H18A...O2 <sup>iii</sup>	0.86	2.55	3.452 (3)	156
C20—H20B...N2 <sup>iii</sup>	0.97	2.53	3.457(2)	159

**Symmetry codes:**(i) *x*, *y*, 1+*z*(ii) -*x*, 1-*y*, 1-*z*(iii) 1-*x*, -*y*, 1-*z*

for Windows<sup>[11]</sup> and PLATON<sup>[12]</sup>. The software used to prepare material for publication is WinGX publication routines<sup>[13]</sup>. Experimental data are listed in Table 1. Fig. 1 shows schematic diagram of the molecule and molecular structure of the title compound along with the atom numbering scheme is depicted in Fig. 2 and a packing diagram is shown in Fig. 3. Table 1 shows the crystal data and crystal refinement. Table 2 gives the

atomic coordinates, Table 3 describes the bond lengths and angles; Table 4 shows anisotropic displacement parameters, Table 5 shows the torsion angles and Table 6 shows hydrogen-bond geometry.

## 4. Results and Discussion

Title compound crystallizes in the triclinic centrosym-



metric space group P-1 with  $Z=2$ . The pyrazole, pyrimidine and pyran rings are attached one another. The methoxy phenyl ring is attached at the C7 position of the pyran ring. An isolated solvent  $C_2O_2SH_4$  is present in the unit cell. the pyrazole ring adopts planar conformation. The average C-N bond length of the pyrazole ring are shorter than a C-N single bond length of 1.424 Å, but longer than a double bond length of 1.31 Å<sup>[14]</sup>, indicating the possibility of electron delocalization. The sum of the bond angles at N1 of the pyrazole ring is 359.9(5)° in accordance with  $sp^2$  hybridized state<sup>[15]</sup>. The pyrazole, pyridine and pyran are almost planar one another but pyran ring attached methoxy phenyl ring are tilted with the dihedral angle of 79.92(1)°. Many of C-H...O and N-H...O type of hydrogen bonds plays a vital role for the stability of crystal packing. In this molecular structure, the C18-H8A...O2 (1-x, -y, 1-z), C20-H20B...N2 (1-x,-y,1-z) and N2-H2...O3 (-x,1-y,1-z) hydrogen bonds has stabilized the crystal packing of the title compound.

## 5. Conclusion

Crystal structure of a novel pyrazole based derivatives having a wide range of applications is described. The title compound is insoluble in millipore water and it is crystallized in ethanol by slow evaporation technique. The Pyrazole, Pyridine and pyran rings adopt a planar conformation. The title structure may be important from a medicinal point of view as well as their widespread biological significance. The structure may be useful for further investigation on the mechanism, potential activity, optimal reaction condition etc which will be further characterized as a future prospective of our project.

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