Synthesis and Structural Analysis of 2-Amino-4-(4-hydroxy-3-methoxyphenyl)-7,9-dimethyl-5-oxo-4, 5, 6, 7-tetrahydropyranono [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-tetrahydropyranono [2, 3-d] pyrazolo [3, 4-b] pyridine-3-carbonitrile (C₂₁H₂₂N₅O₆S) 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 [α=69.573°, β=12.168° and γ=76.060°]. 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 antimicrobial¹, anticancer², anti-inflammatory, antidepressant, anticonvulsant, antihyperglycemic³, antipyretic, antifungal activities⁴, CNS regulants⁵ 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 oxidoreductases⁶. It has been shown in vivo that some of the pyrazole derivatives have appreciable antihyperensive 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 transcriptase inhibitor activities along with Cox-2 inhibitor activity⁷.

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
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 (acceptable 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_{5}O_{6}S was used for data collection on an OXFORD diffractometer. The Mo Kα radiation of wavelength, (λ = 0.71073 Å) 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 F2>2σ(F2). The structures were solved by direct methods using SHELXS-97 and refined by a full-matrix least-squares procedure using the program SHELXL-97. H atoms were positioned geometrically and refined using a riding model, fixing the aromatic C-H distances at 0.93 Å [Uiso(H) = 1.2 Ueq (C)]. The softwares used for Molecular graphics are ORTEP-3.
Table 1. Crystal data and structure refinement.

| Property                                      | Value                      |
|-----------------------------------------------|----------------------------|
| Empirical formula                             | C₉H₂N₂O₂S                  |
| 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) Å³               |
| Z, Calculated density                         | 2, 1.416 Mg/m³             |
| Absorption coefficient                        | 0.195 mm⁻¹                 |
| 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 F2 |
| Data / restraints / parameters                | 4110 / 0 / 298             |
| Goodness-of-fit on F2                         | 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⁻³      |

Table 2. Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²)

| Atom  | x     | y     | z     | Ueq  |
|-------|-------|-------|-------|------|
| N5    | 0.0902 (8) | 0.6193 (6) | 0.8615 (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\textsubscript{eq}/U\textsubscript{eq} |
|-----|-----------|-----------|-----------|-------------------------------------|
| N1  | 0.8121 (7)| 0.6639 (4)| 0.2087 (4)| 0.0421 (13)                         |
| C2  | 0.5746 (8)| 0.6841 (5)| 0.3324 (5)| 0.0533 (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    | 0.7546    | 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)| 0.6085 (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)   |
| C19—N1      | 1.452 (7)   |
| C19—H19A    | 0.9600      |
| C19—H19B    | 0.9600      |
| C19—H19C    | 0.9600      |
| C20—S1      | 1.719 (11)  |
| C20—O5      | 1.771 (13)  |
| C20—H20A    | 0.9700      |
| O2—C5       | 1.372 (6)   |
| C7—C12      | 1.492 (8)   |
| C7—H7       | 0.71 (4)    |
| N1—C1       | 1.329 (7)   |
| N1—N2       | 1.342 (6)   |
| C2—C3       | 1.394 (7)   |
| O4—C18      | 1.368 (8)   |
| O4—C16      | 1.382 (7)   |
| C9—N4       | 1.346 (7)   |
| C16—C15     | 1.357 (7)   |

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Table 3. Continued

| Bond length | Bond length |
|-------------|-------------|
| O2—C9       | 1.386 (7)   |
| N3—C1       | 1.364 (7)   |
| N3—C10      | 1.375 (6)   |
| N3—H3       | 0.8600      |
| O1—C10      | 1.239 (6)   |
| O3—C15      | 1.354 (6)   |
| O3—H3A      | 0.8200      |
| C17—C16     | 1.366 (7)   |
| C17—C12     | 1.365 (7)   |
| C17—H17     | 0.9300      |
| S1—O5       | 1.173 (6)   |
| S1—C21      | 1.765 (11)  |
| C8—C9       | 1.350 (8)   |
| C8—C11      | 1.415 (9)   |
| C8—C7       | 1.520 (8)   |
| C5—C6       | 1.355 (7)   |
| C5—C2       | 1.433 (7)   |
| C6—C10      | 1.441 (7)   |
| C6—C7       | 1.492 (8)   |

| Bond Angle | Bond Angle |
|------------|------------|
| N1—C19—H19A | 109.5 |
| N1—C19—H19B | 109.5 |
| H19A—C19—H19B | 109.5 |
| N1—C19—H19C | 109.5 |
| H19A—C19—H19C | 109.5 |
| H19B—C19—H19C | 109.5 |
| S1—C20—O5   | 39.2 (3)   |
| S1—C20—H20A | 119.4   |
| O5—C20—H20A | 119.4   |
| S1—C20—H20B | 119.4   |
| O5—C20—H20B | 119.4   |
| H20A—C20—H20B | 117.1 |
| C5—O2—C9    | 117.2 (4)  |
| C1—N3—C10   | 120.1 (5)  |
| C1—N3—H3    | 120.0      |
| C10—N3—H3   | 120.0      |
| C15—O3—H3A  | 109.5      |
| C16—C17—C12 | 122.8 (5)  |
| C16—C17—H17 | 118.6     |
| C12—C17—H17 | 118.6     |
| O5—S1—O6    | 65.6 (5)   |
| O5—S1—C20   | 72.8 (6)   |
| O6—S1—C20   | 114.6 (6)  |

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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 (Å²)

|       | $U^{11}$ | $U^{22}$ | $U^{33}$ | $U^{12}$ | $U^{13}$ | $U^{23}$ |
|-------|----------|----------|----------|----------|----------|----------|
| 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)           |
| C5—C6—C10—N3     | 3.5 (8)           |
| C7—C6—C10—N3     | -175.2 (5)        |
| C6—C5—C2—C1      | 1.3 (8)           |
| O2—C5—C2—C1      | -177.8 (5)        |
| C6—C5—C2—C3      | -179.4 (7)        |
| O2—C5—C2—C3      | 1.5 (10)          |
| C11—C8—C9—N4     | -0.6 (10)         |
| C7—C8—C9—N4      | -177.3 (6)        |
| C10—C9—N3        | 3.5 (8)           |
| C7—C10—C12—O1    | -175.2 (5)        |
| O3—C15—C14—C13   | 178.0 (6)         |
| O4—C16—C15—C14   | -174 (16)         |
| O5—C16—C15—C13   | 3 (17)            |
| O6—C20—O5—C21    | 128.3 (6)         |
| C21—O5—O6—S1     | 50.9 (5)          |
| C20—S1—O5—O6     | -123.0 (6)        |
| S1—C21—O5—C20    | 57.0 (5)          |
| C20—S1—O5—C21    | -64.6 (6)         |
| S1—C21—O5—C20    | 57.0 (5)          |
| C21—O5—C20—S1    | -54.0 (6)         |
| C20—S1—O6—S2     | 57.1 (6)          |
| C21—O6—C20—S1    | -54.0 (6)         |
| S2—C21—O6—S3     | 57.1 (6)          |
| C21—O6—C20—S2    | -54.0 (6)         |
| S3—C21—O6—S1     | 57.0 (5)          |
| C21—O6—C20—S3    | -54.0 (6)         |
| S1—C21—O6—C20    | 57.1 (6)          |

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

| D—H···A   | D—H(Å) | H···A(Å) | D···A(Å) | D—H···A(°) |
|-----------|--------|----------|----------|-----------|
| N2—H2···O3   | 0.86   | 1.95     | 2.798 (4) | 167       |
| N3—H3···O1   | 0.86   | 1.85     | 2.710(5)  | 176       |
| C18—H18A···O2   | 0.86   | 2.55     | 3.452 (3) | 156       |
| C20—H20B···N2   | 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[11] and PLATON[12]. The software used to prepare material for publication is WinGX publication routines[11]. 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 C2O2SH4 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[14], indicating the possibility of electron delocalization. The sum of the bond angles at N1 of the pyrazole ring is 359.9(5)o in accordance with sp2 hybridized state[15]. 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)o. 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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