In the title compound C_{14}H_{17}N_{3}O_{2}, the dihedral angle between the rings is 16.68 (13)°. Although the compound crystallizes in the keto form, the possibility of keto-enamine–imine tautomeration is explained by a strong intramolecular N—H—O hydrogen bond.

Related literature
4-Acetylpyrazolones are good chelating ligands and also show antibacterial, antifungal, anti-inflammatory, carcinostatic and enzyme inhibitory activity, see: Patel et al. (2000, 2001); Chohan & Kausar (2000); Chohan, Jaffery & Supuran (2001); Chohan, Munawar & Supuran (2001); Chohan et al. (2002); Yang et al. (2000). For analgesic agents, see: Gursoy et al. (2000).

Experimental

Crystal data
C_{14}H_{17}N_{3}O_{2}
c = 18.065 (11) Å
Mo Kα radiation
\(\alpha = 22.4703 (10)\) Å
\(b = 7.0002 (4)\) Å
\(a = 2.2470 (3)\) Å

Data collection
Oxford Diffraction Xcalibur Eos diffractometer
4492 measured reflections
2353 independent reflections
\(R_{int} = 0.026\)

Refinement
Hydrogen-bond geometry (Å, °)

Key indicators: single-crystal X-ray study; \(\text{R} = 0.09\), \(w\text{R} = 0.20\); data-to-parameter ratio = 13.4.

Inorganic compounds

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176 parameters
2353 reflections
\(S = 0.98\)
\(\alpha = 0.03\) mm\(^{-1}\)
\(\mu = 0.35\) mm\(^{-1}\)
\(T = 273\) K

Refinement
Hydrogen atoms treated by a mixture of independent and constrained refinement
\(D-H \cdots A\) bond lengths (Å)

Data collection
Oxford Diffraction Xcalibur Eos diffractometer
4492 measured reflections
2353 independent reflections
\(R_{int} = 0.026\)

Refinement
Hydrogen-bond geometry (Å, °)

<table>
<thead>
<tr>
<th>D—H</th>
<th>A</th>
<th>D—H</th>
<th>H—A</th>
<th>D—A</th>
<th>D—H—A</th>
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<tr>
<td>N3—H</td>
<td>0.89 (3)</td>
<td>1.82 (3)</td>
<td>2.71 (3)</td>
<td>146 (3)</td>
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</tbody>
</table>

RI thanks the UGC, India, for the award of Rajiv Gandhi National Fellowship. GV thanks the UGC, India, and the DST-India (Green Chemistry open-ended project) for financial assistance and the DST-FIST for the single crystal X-ray facility at the Department of Chemistry, Pondicherry University, Pondicherry.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: DS2072).

References
Crystal Structure of (Z)-4-(1-(2-anilineimino)ethylidene)-3-methyl-1-phenyl-1H-pyrazol-5(4H)-one

Sharmila P.¹, Jagadeesan G.¹, Jayarajan R.¹, Vasuki G.², Aravindhan S.¹,*

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²Department of Chemistry, Pondicherry University, Pondicherry-605 011, India
*Authors contributed equally to this work
*Correspondence e-mail: aravindhanpresidency@gmail.com

The crystal structure of the title compound C₁₈H₁₈N₄O show all the three rings are tilted to one another. The molecular conformation and other geometrical parameters show the possibility of Pyrazole - Pyrazolone tautomerism. The molecules are stabilized by well defined hydrogen bonding network.

Received: 19 January 2011  Published: 8 April 2011

Experimental Details

X-ray Structure Determination

<table>
<thead>
<tr>
<th>Table 1. Crystallographic data and refinement details</th>
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<tr>
<td>Empirical formula</td>
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<tr>
<td>Formula weight (g/mol)</td>
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<td>Temperature (K)</td>
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<td>Wavelength (Å)</td>
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<tr>
<td>Space group</td>
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<td>Goodness-of-fit on F²</td>
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<td>R indices (I &gt; 2σ(I))</td>
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<td>R indices (all data)</td>
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<td>Δρ max/Δρ min (e Å⁻³)</td>
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</table>

A single crystal of the title compound with dimensions 0.2 x 0.2 x 0.25 mm³ was chosen for an X-ray diffraction study. Diffraction data were collected Xcalibur, Eos diffractometer with graphite fine-focus sealed tube. The structure was solved by Direct Methods. The programs Xcalibur, SAINT, SHELXS-97 and SHELXL-97 were used for data collection, data reduction, structure solution and structure refinement respectively. Molecular graphics like ORTEP-3[4] and PLATON [5] were used for visualization. All non-hydrogen atoms were refined anisotropically.

All the non-hydrogen atoms were revealed in the first Fourier map itself. Full-matrix least squares refinement was carried out using SHELXL-97 [3]. The hydrogen atoms were fixed at chemically acceptable positions and were allowed to ride on their parent atoms. 240 parameters were refined with 2314 unique reflections which converged the residuals to 0.029. The details of the crystallographic data and refinement are given in Table 1. Fig. 1 shows the schematic diagram of the molecule. Fig. 2 represents the ORTEP diagram of the molecule with thermal ellipsoids drawn at 30% probability.

Fig. 1. Schematic diagram of the molecule.

Fig. 2 The ORTEP diagram of molecule with thermal ellipsoids drawn at 30% probability.
Synthesis

Ethanolic solution of 3-methyl-1-phenyl-4-acetylpyrazolin-5-ol (0.432 g, 2 mM) and 2-aminoethanol (0.122 g, 2mM) was taken in a round bottom flask and refluxed for 4 h. The solid product was filtered and washed with cold ethanol. The product obtained was pure by TLC and NMR spectroscopy. However, the products were further purified by re-crystallization from ethanol and dried under vacuum. The compound was crystallized by slow evaporation technique using methanol as solvent at room temperature.

Structure Description

The ORTEP diagram of the molecule is given in Fig: 2. The phenyl ring and the middle pyrazole ring are tilted to an angle of 21.54° whereas the aniline ring tilted with the pyrazole ring with 61.48°. The terminal phenyl and aniline rings are oriented with one another by angle of 75.69°. The N3 hydrogen is planar to C13 & C15. This unusual planarity can be explained by strong N3—HN3···O hydrogen bond. The N3 is connected to aniline ring by 1.429 Å (2) whereas the same N3 is connected to C13 by 1.326Å which should be a partial double bond. The unusual geometry around N3 and its strong hydrogen bond (D···A=2.698Å and D–H···A=140.57°) with keto oxygen (O) shows the possibility of pyrazole-Pyrazolone tautomerism (6). The keto oxygen (O) of a molecule (x, y, z) accepts a hydrogen from aniline amine of another molecule (x + 1, y, z) and in turn aniline donates hydrogen to the keto oxygen of that molecule. By this way a defined dimer exists throughout the crystal. The other hydrogen of the aniline group is involving in making an inter molecular hydrogen bond (x, y, z; 3.283 (3) Å and 146 (2)°) network for stability of the crystal packing. The packing of the molecule when viewed down along b axis shows the layered arrangement of the molecules (Fig. 3).

Acknowledgement

R.J. thanks the UGC, India for the award of Rajiv Gandhi National Fellowship and GV thanks UGC, India and DST-India (Green Chemistry open ended project) for financial assistance and DST-FIST for Single crystal X-ray facility to Department of Chemistry, Pondicherry University, Pondicherry. S. A thanks the UGC, India for financial support.

Supporting Information

CIF files of this compound is given as supplementary material. The files CCDC No. 802878 contain the crystallographic data. These data can be obtained from the CCDC web site via http://www.ccdc.cam.ac.uk/conts/retrieving.html, or from the Cambridge Crystallographic Data Centre, 12 Union Road, Cambridge CB2 1EZ, UK; fax: +44(0)1223-336-033; or e-mail: deposit@ccdc.cam.ac.uk.

Notes and References

4. ORTEP-3 for Windows University of Glasgow, Scotland.
Crystal Structure of (Z)-4-(1-(2-hydroxyphenylimino)ethyl)-3-methyl-1-phenylpyrazol-5-ol


*Department of Physics, Presidency College, Chennai-600005, India
**Department of Chemistry, Pondicherry University, Pondicherry-605014, India

The crystal structure of the title compound C18H13N3O2 contains two molecules in the asymmetric unit show all the three rings are tilted to one another. The molecular conformation and other geometrical parameters show the possibility of pyrazole-pyrazolone tautomeration.

Received: 6 May 2011 Published: 4 November 2011

Experimental Details
X-ray Structure Determination

Table 1. Crystallographic data and refinement details

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<tr>
<th>Empirical formula</th>
<th>C18H13N3O2</th>
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<td>c (Å)</td>
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<td>R indices (all data)</td>
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<td>Δρmax / Δρmin (e.Å⁻³)</td>
<td>0.17 / -0.14</td>
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A single crystal of the title compound with dimensions 0.20 x 0.20 x 0.20 mm³ was chosen for an X-ray diffraction study. Diffraction data were collected Xcalibur, Eos oxford diffractometer [1] with graphite fine-focus sealed tube. The structure was solved by Direct Methods. The programs Xcalibur, SAINT, SHELXS-97 and SHELXL-97 were used for data collection, data reduction, structure solution and structure refinement respectively. Molecular graphics like ORTEP-3 [2] and PLATON [3] were used for visualization. All non-hydrogen atoms were refined anisotropically. All the non-hydrogen atoms were revealed in the first Fourier map itself. Full-matrix least squares refinement was carried out using SHELXL-97 [4]. The hydrogen atoms were fixed at chemically acceptable positions and were allowed to ride on their parent atoms. 435 parameters were refined with 2901 unique reflections which converged the residuals to 0.0641. The details of the crystallographic data and refinement are given in Table 1.

Figure 1. Schematic diagram of the molecule

Synthesis
Ethanolic solution of 3-methyl-1-phenyl-4-acetylpyrazol-5-ol (2mmol) and 2-aminophenol (2mmol) was taken in round bottom flask and refluxed for 8h. The solid product was filtered and washed with cold ethanol and recrystallized from aceto-nitrite and dried under vacuum. Pale yellow powder, Yield (70%); m.p. 220°C. temperature.

Figure: 2 represents the ORTEP diagram of the molecule with thermal ellipsoids drawn at 30% probability.
Structure Description

The ORTEP diagram of the molecule is given in Fig. 2. There are two molecules (A&B) in asymmetric unit. In molecule A, the phenyl ring and the middle pyrazole ring are tilted to an angle of 169.60° whereas the hydroxyl phenyl ring tilted with the pyrazole ring with 140.83°. The terminal phenyl and hydroxyl phenyl rings are oriented with one another by an angle of 135.79°. In molecule B, the corresponding values are 171.19°, 133.12° and 134.17° respectively.

The hydrogen on N3 maintains planarity with N3, C13 & C15. This unusual planarity can be explained by strong N3—H3N3···O1 hydrogen bond. The N3 is connected to phenyl ring by 1.425 Å (molecule B: 1.432Å) whereas the same N3A is connected to C13A by 1.390Å (molecule B: 1.333Å) which should be a partial double bond. The unusual geometry around N3A and its strong hydrogen bond (D—A=1.899Å (molecule B: 1.877 Å) and D—H—A=139.37° (molecule B: 139.03°) with keto oxygen (O) shows the possibility of pyrazole-Pyrazolone tautomerism. The molecules are stabilized by well defined hydrogen bonding network. Although the compound crystallizes in the keto form, the possibility of keto-enameine-ename-imine tautomerism [5, 6] is explained by a strong intramolecular N—H—O hydrogen bond.

Keto oxygen (O) of molecule B accepts hydrogen from phenolic oxygen (O2AH) of molecule A. Phenolic oxygen (O2BH) of molecule B donates hydrogen to pyrazole N2A of molecule A. These intermolecular interactions also stabilize crystal packing by well defined hydrogen bonding network. The packing of the molecule when viewed down along b-axis (Figure: 3) shows the hydrogen bond network.

Acknowledgments

R. J. thanks the UGC, India for the award of Rajiv Gandhi National Fellowship and G. V. thanks UGC, India and DST-India (Green Chemistry open ended project) for financial assistance and DST-FIST for Single crystal X-ray facility to Department of Chemistry, Pondicherry University, Pondicherry. S. A. thanks the UGC, India for financial support.

Supporting Information

CIF files of this compound are given as supplementary material. The files CCDC No 805771 contain the crystallographic data. These data can be obtained from the CCDC web site via, or from the Cambridge Crystallographic Data Centre, 12 Union Road, Cambridge CB2 1EZ, UK; fax: +44(0)1223-336-033; or e-mail: deposit@ccdc.cam.ac.uk.

Notes and References

2 ORTEP-3 for Windows. University of Glasgow, Scotland.
Methyl 8-bromo-3-[1-(4-methoxyphenyl)-4-oxo-3-phenylazetidin-2-yl]-1-methyl-1,2,3,3a,4,9b-hexahydrochromeno[4,3-b]pyrrole-3a-carboxylate

P. Sharmila, a G. Jagadeesan, a Rajesh Raju, b Raghunathan Raghavachary b and S. Aravindhan a*

aDepartment of Physics, Presidency College, Chennai 600 005, India, and
bDepartment of Organic Chemistry, University of Madras, Guindy Campus, Chennai 600 025, India

Correspondence e-mail: aravindhanpresidency@gmail.com

Received 21 June 2013; accepted 7 September 2013

Key indicators: single-crystal X-ray study; 28413 measured reflections; 6511 independent reflections; 5355 reflections with I > 2σ(I); R indices (all data): R = 0.037, wR2 = 0.065; data-to-parameter ratio = 19.0.

In the title compound, C30H29BrN2O5, the β-lactam ring is essentially planar, with the O atom displaced from this plane by 0.856 (9) Å, and forming dihedral angles of 24.35 (13) and 89.42 (14)° with the planes of the benzene substituent groups on this ring. The tetrahydropyran ring adopts an envelope conformation with the C atom bearing the β-lactam ring as the flap. In the crystal, weak C—H⋯O hydrogen bonds with carbonyl and tetrahydropyran O-atom acceptors give rise to a chain structure extending along the b-axis direction.

Related literature


Experimental

Crystal data

C30H29BrN2O5
M_r = 577.46
Monoclinic, P2_1
a = 10.904 (5) Å
b = 10.765 (5) Å
γ = 11.405 (5) Å

Data collection

Bruker APEX2 CCD diffractometer
28413 measured reflections
5355 independent reflections

Refinement

R[F^2 > 2σ(F^2)] = 0.030
wR2 = 0.065
S = 1.00

Table 1

Hydrogen-bond geometry (Å, °)

B—H—A
B—H—A

C21—H21B⋯O4ii
0.97
2.43
3.36 (3)
161
C14—H14⋯O4v
0.93
2.56
3.39 (3)
138

Symmetry codes: (ii) x, y−1/2, −z+1/2; (v) x, y−1/2, z+1/2.

Data collection: APEX2 (Bruker, 2008); cell refinement: APEX2 and SAINT (Bruker, 2008); data reduction: SAINT and XPREP (Bruker, 2008); program(s) used to solve structure: SHELX97 (Sheldrick, 2008); program(s) used to refine structure: SHEXL97 (Sheldrick, 2008); molecular graphics: ORTEP-3 for Windows (Farrugia, 2012); software used to prepare material for publication: PLATON (Spek, 2009).

SA thanks the UGC-India for financial support.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: ZS2268).

References


Acta Cryst. (2013). E69, o1570

01570 Sharmila et al.
Methyl 9-hydroxy-15-methyl-2-oxo-11-(pyren-1-yl)-10-oxa-15-azatetracyclo-
[7.6.0.1^1,12.03,8]pentadeca-3(8),4,6-triene-12-carboxylate

P. Sharmila, G. Jagadeesan, Rajesh Raju, Raghunathan Raghavachary and S. Aravindan

In the title compound, C_{32}H_{25}NO_{5}, the furan and pyrrole rings each adopt an envelope conformation, the respective flap atoms being the C atom bearing the pyrene substituent and the CH_{3} atom adjacent to the N atom. The molecular conformation is stabilized by an intramolecular O—H—N hydrogen bond. In the crystal, C—H···O contacts link the molecules, forming a two-dimensional network parallel to (001).

Related literature

For the solid-state structures of pyrenes, see: Robertson & White (1947); Camerman & Trotter (1965); Allmann (1970); Hazell et al. (1972); Kato et al. (1978). For a related structure, see: Gruber et al. (2010). For the use of pyrenes in fluorescence sensors, see: Bren (2001).
4-Bromo-2-(diethoxymethyl)phenyl benzoate

P. Sharmila, C. Suresh Kumar, S. Maheshwaran, S. Narasimhan and S. Aravindhan

Acta Cryst. (2013). E69, o553

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Crystallography Journals Online is available from journals.iucr.org
4-Bromo-2-(diethoxymethyl)phenyl benzoate

P. Sharmila,a C. Suresh Kumar,b S. Maheshwaran,b S. Narasimhanb and S. Aravindhana*

a Department of Physics, Presidency College, Chennai 600 005, India, and  
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Correspondence e-mail: aravindhanpresidency@gmail.com 
Received 17 February 2013; accepted 5 March 2013 

Key indicators: single-crystal X-ray study; T = 293 K; mean /C27 (C–C) = 0.003 Å; R factor = 0.034; wR factor = 0.085; data-to-parameter ratio = 21.5.

In the title compound, C18H19BrO4, the aromatic rings enclose a dihedral angle of 81.9 (7)°. There are no short directional contacts in the crystal structure.

Related literature

For the biological activity of ester derivatives, see: Bi et al. (2012); Bartzatt et al. (2004); Anadu et al. (2006).

Experimental

Crystal data

C18H19BrO4  
Mr = 379.24  
Triclinic, P  
a = 8.2662 (8) Å  
b = 9.6378 (10) Å  
c = 11.6224 (13) Å  
α = 99.927 (5)°  
β = 93.700 (5)°  
γ = 101.178 (5)°  
V = 999.18 (16) Å³  
Z = 2  
Mo Kα radiation  
µ = 2.33 mm⁻¹  
T = 293 K  
0.35 × 0.30 × 0.25 mm

Data collection

Bruker Kappa APEXII CCD  
diffractometer  
16152 measured reflections  
4493 independent reflections  
3037 reflections with I > 2σ(I)

Refinement

R[ I/2( F²)] = 0.034  
s( R[F²]) = 0.085  
R1 = 0.035  
W = 1.01

References


SA thanks the UGC, India, for financial support

SA thanks the UGC, India, for financial support

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: BT6492).

References


Diethyl 2,6-dimethyl-4-[4-(3-phenylacryloyloxy)phenyl]-1,4-dihydropyridine-3,5-dicarboxylate hemihydrate

P. Sharmila, C. Suresh Kumar, Karthik Ananth, S. Narasimhan and S. Aravindhan

*Acta Cryst. (2013). E69, o389*
Diethyl 2,6-dimethyl-4-[4-(3-phenyl-acryloyloxy)phenyl]-1,4-dihydropyridine-3,5-dicarboxylate hemihydrate

P. Sharmila, C. Suresh Kumar, Karthik Ananth, S. Narasimhanb and S. Aravindhan*

*Department of Physics, Presidency College, Chennai 600 005, India, and *Anagiri Herbal Research Foundation, Porur, Chennai 600 106, India

Correspondence e-mail: aravindhanpresidency@gmail.com

Received 27 December 2012; accepted 11 February 2013

Key indicators: single-crystal X-ray study; m = 293 K; mean

organic compounds

Table 1

Hydrogen-bond geometry (Å, °)

D—H—A D—H H—A D—A D—H—A

N1—H1N—O5 0.87 (2) 2.31 (2) 3.14 (2) 163 (2)

O1W—H1W1—O9s 1.04 1.91 2.93 (3) 168

O14—H14—O18w 0.93 2.59 3.40 (4) 162

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

Related literature

For the biological activity of ester derivatives, see: Bi et al. (2012); Bartzatt et al. (2004); Anadu et al. (2006).

Data collection

Bruker APEXII CCD diffractometer

Absorption correction: multi-scan

SADABS (Bruker, 2004)

Refinement

H atoms treated by a mixture of independent and constrained refinement

Δρmax = 0.18 e Å−3

Δρmin = −0.31 e Å−3

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: SU2546).

SA thanks the UGC, India, for financial support.