Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

7-Allyl-1,3-dimethyl-2,4-dioxo-2,3,4,7-tetrahydro-1*H*-pyrrolo[2,3-*d*]pyrimidine-6-carbaldehyde

B. K. Satis Kumar,[a] D. Gayathri,[b] D. Velmurugan,[b]* K. Ravikumar[c] and E. Ramesh[d]

[a]Department of Physics, Presidency College, Chennai 600 005, India, [b]Department of Crystallography and Biophysics, University of Madras, Guindy Campus, Chennai 600 025, India, [c]Laboratory of X-ray Crystallography, Indian Institute of Chemical Technology, Hyderabad 500 007, India, and [d]Department of Organic Chemistry, University of Madras, Guindy Campus, Chennai 600 025, India

Correspondence e-mail: d_velu@yahoo.com

Key indicators

Single-crystal X-ray study
T = 293 K
Mean σ(C–C) = 0.002 Å
R factor = 0.046
wR factor = 0.139
Data-to-parameter ratio = 17.0

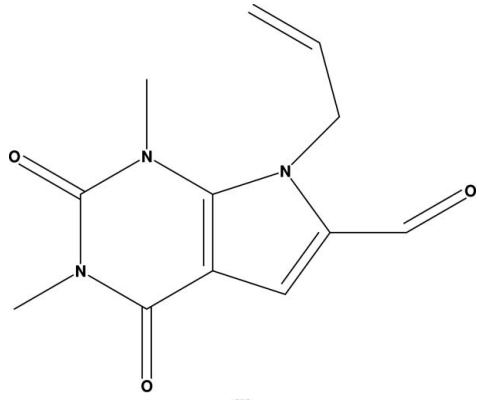
For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

Received 27 March 2007
Accepted 2 April 2007

The five-membered ring in the title compound, $C_{12}H_{13}N_3O_3$, is planar and the six-membered ring is slightly non-planar, with one N atom deviating by 0.075 (1) Å. The molecule is stabilized by C—H···O and C—H···N intramolecular interactions. The crystal packing is stabilized by C—H···O intermolecular interactions generating a centrosymmetric dimer with an $R_2^2(10)$ ring.

Comment

Pyrrolo[2,3-*d*]pyrimidines are known to possess considerable antitumour, anti-allergic, antiviral and anti-inflammatory activities (Hutzenlaub *et al.*, 1972; Smith *et al.*, 1972). Pyrrolo[2,3-*d*]pyrimidine nucleosides act as inhibitors of human cytomegalovirus (Turk *et al.*, 1987). It is reported that pyrrolo[2,3-*d*]pyrimidine-based antifolate inhibits multiple folate-requiring enzymes (Shih *et al.*, 1997). As the title compound, (I), is of high medicinal value, we have undertaken the three-dimensional structure determination by X-ray diffraction (Fig. 1).



(I)

The bond geometry of (I) is comparable with reported values (Allen *et al.*, 1987). The sums of the bond angles around atoms N1, N2 and N3 [360.0, 360.0 and 359.5°, respectively] indicate the sp^2 hydrization of all the N atoms in the structure. The five-membered ring in the structure is planar. The torsion angles around N3—C6—C7—O3 [0.0 (3)°] and C5—C6—C7—O3 [−180.0 (2)°] indicate that the aldehyde group lies in the plane of the five-membered ring. The six-membered ring in the structure is slightly non-planar, with atom N1 deviating by 0.075 (1) Å from the plane of the remaining atoms in the ring. Atoms O1 and C12 lie below and atoms O2 and C11 lie above the plane of C2/N2/C3/C4/C1 by 0.051 (1), 0.084 (2), 0.057 (1) and 0.269 (2) Å, respectively.

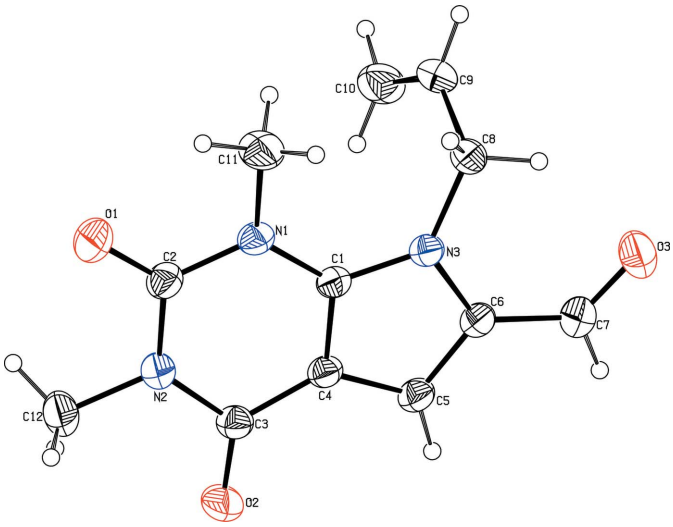


Figure 1
The molecular structure of compound (I), showing 30% probability displacement ellipsoids.

The molecule of (I) is stabilized by weak C—H···O and C—H···N intramolecular interactions. The crystal packing is stabilized by C—H···O intermolecular interactions (Table 1). Atom C5 acts as a donor to atom O2($1-x, -y, 1-z$), generating a centrosymmetric dimer with an $R_2^2(10)$ ring (Bernstein *et al.*, 1995) centred at ($\frac{1}{2}$, 0, $\frac{1}{2}$). These dimers run along the *bc* plane.

Experimental

2,3,4,7-Tetrahydro-1,3-dimethyl-2,4-dioxo-1*H*-pyrrolo[2,3-*d*]pyrimidine-6-carbaldehyde (1 equivalent) and allyl bromide (1.2 equivalents) were stirred with K_2CO_3 (2.5 equivalents) in dry dimethylformamide (25 ml) at room temperature for 12 h, and then the reaction mixture was poured into water and extracted with ethyl acetate. The organic layer was dried over Na_2SO_4 and evaporated to give a residue, which was purified by column chromatrography using hexane–ethyl acetate (3:1 *v*/*v*) as eluant. Pure compound (I) was crystallized from ethyl acetate by slow evaporation.

Crystal data

$C_{12}H_{13}N_3O_3$
M_r = 247.25
Monoclinic, $P2_1/n$
a = 6.7065 (4) Å
b = 13.1295 (9) Å
c = 13.6977 (9) Å
β = 99.613 (1)°
V = 1189.19 (13) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.10 mm^{-1}
T = 293 (2) K
0.24 × 0.22 × 0.21 mm

Data collection

Bruker SMART APEX CCD area-detector diffractometer
Absorption correction: none
13428 measured reflections
2810 independent reflections
2489 reflections with $I > 2\sigma(I)$
R_{int} = 0.017

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.046
$wR(F^2)$ = 0.139
S = 1.03
2810 reflections
165 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.25 e Å^{-3}
$\Delta\rho_{min}$ = −0.19 e Å^{-3}

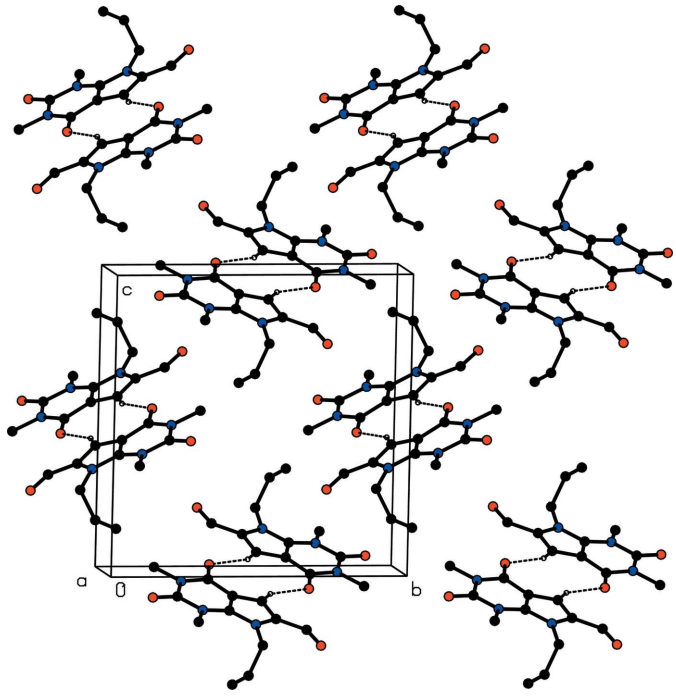


Figure 2
The molecular packing of (I), viewed approximately along the *a* axis. For clarity, H atoms not involved in hydrogen bonding (dashed lines) have been omitted.

Table 1
Hydrogen-bond geometry (Å, °).

D—H···A	D—H	H···A	D···A	D—H···A
C5—H5···O2^{i}	0.93	2.37	3.237 (2)	155
C8—H8*B*···O3	0.97	2.26	2.987 (2)	131
C10—H10*A*···N3	0.93	2.52	2.848 (2)	101
C11—H11*B*···O1	0.96	2.25	2.671 (2)	106
C12—H12*B*···O1	0.96	2.30	2.684 (2)	103

Symmetry code: (i) $-x+1, -y, -z+1$.

All H atoms were refined using a riding model, with C—H = 0.93–0.97 Å and U_{iso}(H) = 1.2U_{eq}(C) or 1.5U_{eq}(C).

Data collection: *SMART* (Bruker, 2001); cell refinement: *SAINT* (Bruker, 2001); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *PLATON* (Spek, 2003); software used to prepare material for publication: *SHELXL97* and *PARST* (Nardelli, 1995).

DG thanks the CSIR, India, for the award of a Senior Research Fellowship. DV thanks the DST, India, for a Major Research Project. The Department of Science and Technology (DST-FIST) and the University Grants Commission (UGC), Government of India, are acknowledged by DV for providing facilities to the department.

References

Allen, F. H., Kennard, O., Watson, D. G., Brammer, L., Orpen, A. G. & Taylor, R. (1987). *J. Chem. Soc. Perkin Trans. 2*, pp. S1–19.
Bernstein, J., Davis, R. E., Shimoni, L. & Chang, N.-L. (1995). *Angew. Chem. Int. Ed. Engl.* **34**, 1555–1573.

Bruker (2001). *SMART* (Version 5.625/NT/2000) and *SAINT* (Version 6.28a). Bruker AXS Inc., Madison, Wisconsin, USA.
Hutzenlaub, W., Tolman, R. L. & Robins, R. K. (1972). *J. Med. Chem.* **15**, 879–883.
Nardelli, M. (1995). *J. Appl. Cryst.* **28**, 659.
Sheldrick, G. M. (1997). *SHELXS97* and *SHELXL97*. University of Göttingen, Germany.
Shih, C., Chen, V. J., Gossett, L. S., Gates, S. B., MacKellar, W. C., Habeck, L. L., Shackelford, K. A., Mendelsohn, L. G., Soose, D. J., Patel, V. F., Andis, S. L., Bewley, J. R., Rayl, E. A., Moroson, B. A., Beardsley, G. P., Kohier, W., Ratnam, M. & Schult, R. M. (1997). *Cancer Res.* **57**, 1116–1123.
Smith, C. W., Sidwell, R. W., Robins, R. K. & Tolman, R. L. (1972). *J. Med. Chem.* **15**, 883–887.
Spek, A. L. (2003). *J. Appl. Cryst.* **36**, 7–13.
Turk, S. R., Shipman, C. Jr, Nassiri, R., Genzlinger, G., Krawczyk, S. H., Townsend, L. B. & Drach, J. C. (1987). *Antimicrob. Agents Chemother.* **31**, 544–550.