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2,4-Dioxo-1-(prop-2-ynyl)-1,2,3,4-tetrahydropyrimidine-5-carbaldehyde

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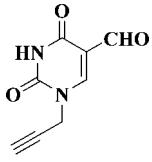
Key indicators: single-crystal X-ray study; T = 296 K; mean σ(C–C) = 0.002 Å; R factor = 0.040; wR factor = 0.123; data-to-parameter ratio = 12.9.

In the crystal structure of the title compound, $C_8H_6N_2O_3$, the molecules are linked by a pairs of intermolecular N—H···O hydrogen bonds, forming inversion dimers. The aldehyde group is in the same plane as the pyrimidine ring [with a maximum deviation of 0.083 (2) Å for the O atom), and the linear propargyl group [C—C—C = 178.99 (19)°] makes a dihedral angle of 74.36 (13)° with the ring.

Related literature

For applications of acyclic pyrimidine nucleosides, see: De Clercq (2009, 2010*a*,*b*); Fan *et al.* (2011).



Experimental

Crystal data

$C_8H_6N_2O_3$
M_r = 178.15
Monoclinic, $P2_1/n$
a = 5.1756 (7) Å
b = 8.4877 (12) Å
c = 18.565 (3) Å
β = 90.611 (2)°
V = 815.5 (2) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.11 mm^{-1}
T = 296 K
0.41 × 0.37 × 0.25 mm

Data collection

Bruker SMART CCD area-detector diffractometer
Absorption correction: multi-scan (*SADABS*; Bruker, 1997)
T_{min} = 0.955, T_{max} = 0.972
5826 measured reflections
1520 independent reflections
1261 reflections with $I > 2\sigma(I)$
R_{int} = 0.020

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.040
$wR(F^2)$ = 0.123
S = 1.08
1520 reflections
118 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.14 e Å^{-3}
$\Delta\rho_{min}$ = −0.23 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
N2—H2···O1^{i}	0.86	1.98	2.8329 (18)	174

Symmetry code: (i) $-x+2, -y+2, -z+1$.

Data collection: *SMART* (Bruker, 1997); cell refinement: *SAINT* (Bruker, 1997); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *SHELXTL* (Sheldrick, 2008); software used to prepare material for publication: *SHELXTL*.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: IS2760).

References

Bruker (1997). *SADABS*, *SMART* and *SAINT*. Bruker AXS Inc., Madison, Wisconsin, USA.
De Clercq, E. (2009). *Rev. Med. Virol.* **19**, 287–299.
De Clercq, E. (2010*a*). *J. Med. Chem.* **53**, 1438–1450.
De Clercq, E. (2010*b*). *Antiviral Res.* **85**, 19–24.
Fan, X.-S., Wang, Y.-Y., Qu, Y.-Y., Xu, H.-Y., He, Y., Zhang, X.-Y. & Wang, J.-J. (2011). *J. Org. Chem.* **76**, 982–985.
Sheldrick, G. M. (2008). *Acta Cryst.* A**64**, 112–122.

supporting information

Acta Cryst. (2011). E**67**, o2350 [doi:10.1107/S1600536811032272]

2,4-Dioxo-1-(prop-2-ynyl)-1,2,3,4-tetrahydropyrimidine-5-carbaldehyde

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S1. Comment

Acyclic pyrimidine nucleosides have drawn much attention because of their insteresting structures and broad utilizations as effective drugs for the treatment of diseases caused by herpes simplex virus (HSV) and varizella zoster (VZV) (De Clercq, 2009, 2010*a*,*b*). The title compound can be used as a powerful synthon for the preparation of acyclic pyrimidine nucleoside derivatives with potential biological activities due to the rich and extensive chemistry of the aldehyde carbonyl (Fan, 2011). Herein, we report the synthesis and crystal structure of the title compound.

In the title compound, $C_8H_6N_2O_3$, all the atoms in the pyrimidine ring, atoms connected directly with the pyrimidine ring and atoms in the aldehyde carbonyl group in the 5-position of the pyrimidine ring are in the same plane, which means there is a big conjugated system in the molecule. The linear structure of the propynyl group is connected with the big plane at an angle of 150.3°. In the crystal structure, the molecules are linked *via* intermolecular N—H···O hydrogen bond.

S2. Experimental

To a solution of $K_2S_2O_8$ (16.5 mmol) and $CuSO_4$ (3.2 mmol) in 30 ml H_2O was added a CH_3CN solution (25 ml) of 5-methyl-1-(prop-2-ynyl)pyrimidine-2,4(1*H*,3*H*)-dione (8 mmol) and 2,6-lutidine (3.2 ml). The mixture was stirred at 60 °C for 5 h. Upon completion, the mixture was concentrated to half of the initial volume, and the remaining solution was extracted with EtOAc. The organic layer was washed with H_2O. The aqueous layers were combined and back-extracted with $CHCl_3$. Then the organic layers were combined, dried over Na_2SO_4, and then concentrated. The residue was purified through silica gel column chromatography with a mixture of methylene chloride-methanol (60:1, *v*/*v*) as eluent to give 1,2,3,4-tetrahydro-2,4-dioxo-1-(prop-2-ynyl)- pyrimidine-5-carbaldehyde. Single crystals of the title compound were obtained by slow evaporation of the solvent from a methylene chloride-methanol (60:1 *v*/*v*) solution.

S3. Refinement

H atoms were positioned geometrically and refined using riding model, with C—H = 0.93 or 0.97 Å, and N—H = 0.86 Å, and with U_{iso}(H) = 1.2U_{eq}(C, N).

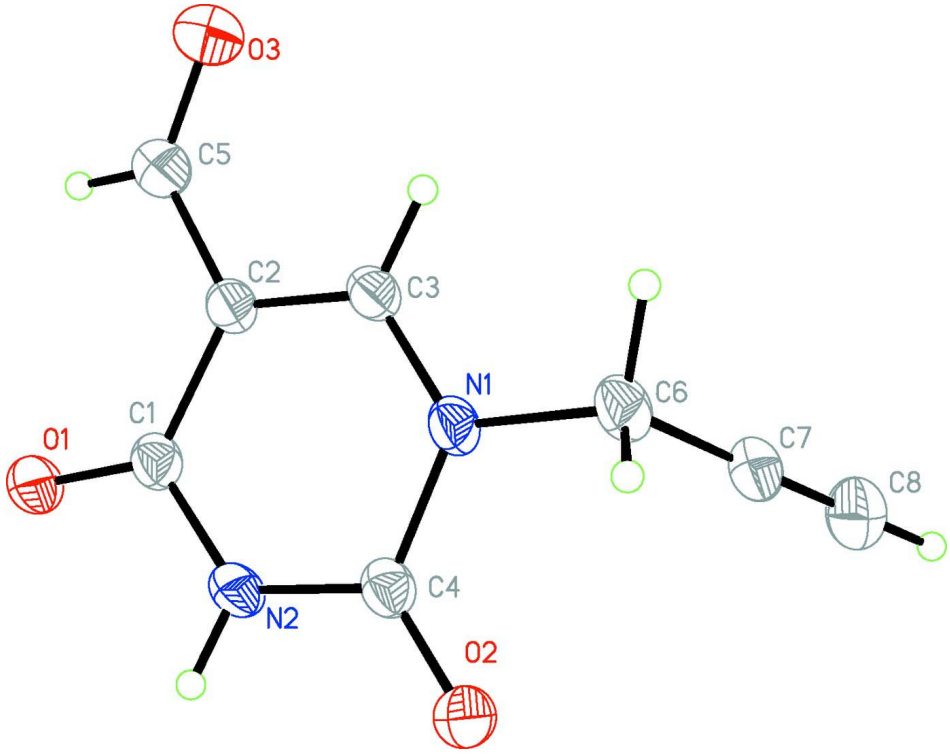

Figure 1

Molecular structure of the title compound, with displacement ellipsoids drawn at the 30% probability level.

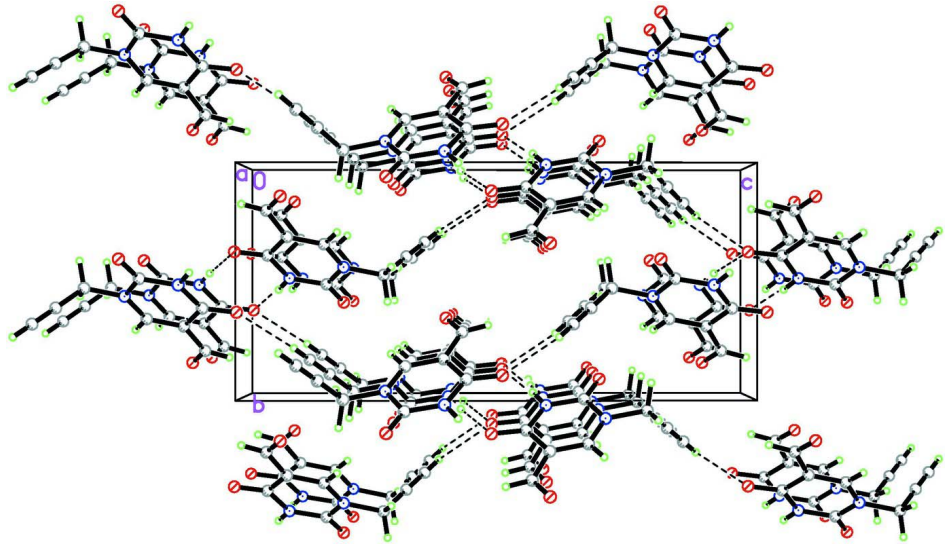

Figure 2

Crystal packing of the title compound with view along the *a* axis. Intermolecular N—H···O hydrogen bonds are shown as dashed lines.

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$b = 8.4877$ (12) Å
$c = 18.565$ (3) Å
$\beta = 90.611$ (2)°
$V = 815.5$ (2) Å^3
$Z = 4$
$F(000) = 368$
$D_x = 1.451$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 2188 reflections
$\theta = 2.6$–26.7°
$\mu = 0.11$ mm^{-1}
$T = 296$ K
Block, colourless
0.41 × 0.37 × 0.25 mm

Data collection

Bruker SMART CCD area-detector diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
phi and ω scans
Absorption correction: multi-scan (*SADABS*; Bruker, 1997)
$T_{min} = 0.955$, $T_{max} = 0.972$
5826 measured reflections
1520 independent reflections
1261 reflections with $I > 2\sigma(I)$
$R_{int} = 0.020$
$\theta_{max} = 25.5°$, $\theta_{min} = 2.6°$
$h = -6 \rightarrow 6$
$k = -10 \rightarrow 10$
$l = -22 \rightarrow 21$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.040$
$wR(F^2) = 0.123$
$S = 1.08$
1520 reflections
118 parameters
0 restraints
Primary atom site location: structure-invariant direct methods
Secondary atom site location: difference Fourier map
Hydrogen site location: inferred from neighbouring sites
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0694P)^2 + 0.1695P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.14$ e Å^{-3}
$\Delta\rho_{min} = -0.23$ e Å^{-3}

Special details

Geometry. All e.s.d.'s (except the e.s.d. in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell e.s.d.'s are taken into account individually in the estimation of e.s.d.'s in distances, angles and torsion angles; correlations between e.s.d.'s in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell e.s.d.'s is used for estimating e.s.d.'s involving l.s. planes.

Refinement. Refinement of F^2 against ALL reflections. The weighted R-factor wR and goodness of fit S are based on F^2, conventional R-factors R are based on F, with F set to zero for negative F^2. The threshold expression of $F^2 > \sigma(F^2)$ is used only for calculating R-factors(gt) *etc*. and is not relevant to the choice of reflections for refinement. R-factors based on F^2 are statistically about twice as large as those based on F, and R-factors based on ALL data will be even larger.

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

	x	*y*	*z*	U_{iso}*/U_{eq}
C1	0.7042 (3)	0.8893 (2)	0.44458 (8)	0.0374 (4)
C2	0.5046 (3)	0.8128 (2)	0.40196 (8)	0.0376 (4)
C3	0.4934 (3)	0.84333 (19)	0.33033 (8)	0.0372 (4)
H3	0.3631	0.7959	0.3031	0.045*
C4	0.8652 (3)	1.01190 (19)	0.33360 (8)	0.0368 (4)
C5	0.3180 (4)	0.7061 (2)	0.43522 (10)	0.0510 (5)
H5	0.3435	0.6787	0.4833	0.061*
C6	0.6377 (3)	0.9724 (2)	0.21870 (8)	0.0432 (4)
H6A	0.4685	0.9390	0.2017	0.052*
H6B	0.6512	1.0851	0.2110	0.052*
C7	0.8363 (4)	0.8923 (2)	0.17686 (9)	0.0464 (5)
C8	0.9931 (4)	0.8282 (3)	0.14242 (11)	0.0612 (6)
H8	1.1175	0.7773	0.1151	0.073*
N1	0.6616 (2)	0.93903 (17)	0.29646 (7)	0.0370 (4)
N2	0.8664 (3)	0.98582 (16)	0.40649 (7)	0.0398 (4)
H2	0.9818	1.0356	0.4312	0.048*
O1	0.7344 (2)	0.87340 (16)	0.51012 (6)	0.0490 (4)
O2	1.0262 (2)	1.09061 (15)	0.30334 (6)	0.0472 (4)
O3	0.1324 (3)	0.65141 (19)	0.40376 (8)	0.0685 (5)

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.0360 (8)	0.0454 (9)	0.0308 (8)	0.0027 (7)	−0.0031 (6)	−0.0023 (7)
C2	0.0353 (8)	0.0442 (9)	0.0333 (8)	0.0011 (7)	−0.0018 (6)	−0.0066 (7)
C3	0.0326 (8)	0.0439 (9)	0.0350 (8)	0.0021 (7)	−0.0041 (6)	−0.0089 (7)
C4	0.0365 (8)	0.0414 (9)	0.0323 (8)	0.0027 (7)	−0.0036 (7)	−0.0023 (7)
C5	0.0503 (10)	0.0600 (11)	0.0427 (10)	−0.0103 (9)	−0.0011 (8)	−0.0042 (8)
C6	0.0451 (10)	0.0551 (10)	0.0294 (8)	0.0008 (8)	−0.0085 (7)	0.0013 (7)
C7	0.0533 (11)	0.0548 (11)	0.0311 (8)	−0.0078 (9)	−0.0027 (8)	−0.0019 (8)
C8	0.0625 (13)	0.0738 (14)	0.0474 (11)	−0.0033 (11)	0.0076 (10)	−0.0125 (10)
N1	0.0366 (7)	0.0471 (8)	0.0272 (7)	0.0016 (6)	−0.0044 (5)	−0.0029 (6)
N2	0.0402 (8)	0.0494 (8)	0.0297 (7)	−0.0079 (6)	−0.0084 (5)	−0.0014 (6)
O1	0.0500 (7)	0.0683 (8)	0.0285 (6)	−0.0120 (6)	−0.0057 (5)	0.0017 (5)
O2	0.0470 (7)	0.0566 (8)	0.0378 (7)	−0.0094 (6)	−0.0019 (5)	0.0039 (5)
O3	0.0592 (9)	0.0805 (11)	0.0658 (10)	−0.0216 (7)	0.0004 (7)	−0.0139 (8)

Geometric parameters (Å, º)

C1—O1	1.2325 (19)	C5—O3	1.211 (2)
C1—N2	1.374 (2)	C5—H5	0.9300
C1—C2	1.449 (2)	C6—C7	1.462 (3)
C2—C3	1.356 (2)	C6—N1	1.475 (2)
C2—C5	1.465 (3)	C6—H6A	0.9700
C3—N1	1.351 (2)	C6—H6B	0.9700

C3—H3	0.9300	C7—C8	1.173 (3)
C4—O2	1.211 (2)	C8—H8	0.9300
C4—N2	1.371 (2)	N2—H2	0.8600
C4—N1	1.397 (2)		
O1—C1—N2	120.11 (14)	C7—C6—N1	112.24 (14)
O1—C1—C2	124.91 (15)	C7—C6—H6A	109.2
N2—C1—C2	114.98 (13)	N1—C6—H6A	109.2
C3—C2—C1	118.22 (15)	C7—C6—H6B	109.2
C3—C2—C5	120.68 (15)	N1—C6—H6B	109.2
C1—C2—C5	121.10 (15)	H6A—C6—H6B	107.9
N1—C3—C2	123.42 (14)	C8—C7—C6	178.99 (19)
N1—C3—H3	118.3	C7—C8—H8	180.0
C2—C3—H3	118.3	C3—N1—C4	121.52 (13)
O2—C4—N2	123.44 (14)	C3—N1—C6	121.56 (13)
O2—C4—N1	122.30 (14)	C4—N1—C6	116.91 (14)
N2—C4—N1	114.26 (14)	C4—N2—C1	127.43 (13)
O3—C5—C2	123.80 (18)	C4—N2—H2	116.3
O3—C5—H5	118.1	C1—N2—H2	116.3
C2—C5—H5	118.1		
O1—C1—C2—C3	179.16 (16)	O2—C4—N1—C3	175.63 (15)
N2—C1—C2—C3	−0.7 (2)	N2—C4—N1—C3	−4.1 (2)
O1—C1—C2—C5	−0.4 (3)	O2—C4—N1—C6	−4.8 (2)
N2—C1—C2—C5	179.81 (15)	N2—C4—N1—C6	175.44 (14)
C1—C2—C3—N1	1.3 (2)	C7—C6—N1—C3	−106.92 (18)
C5—C2—C3—N1	−179.19 (15)	C7—C6—N1—C4	73.53 (19)
C3—C2—C5—O3	−7.1 (3)	O2—C4—N2—C1	−174.72 (16)
C1—C2—C5—O3	172.38 (18)	N1—C4—N2—C1	5.0 (2)
C2—C3—N1—C4	1.3 (2)	O1—C1—N2—C4	177.47 (15)
C2—C3—N1—C6	−178.27 (15)	C2—C1—N2—C4	−2.7 (2)

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N2—H2···O1^{i}	0.86	1.98	2.8329 (18)	174

Symmetry code: (i) −*x*+2, −*y*+2, −*z*+1.