3-Acetyl-2-methyl-4*H*-pyrido[1,2-*a*]pyrimidin-4-one

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In the title compound, $C_{11}H_{10}N_2O_2$, the pyridopyrimidine moiety deviates slightly from planarity. In the crystal, molecules stack along the *a*-axis direction assisted by π–π stacking interactions and C—H···O hydrogen bonds. The stacks are associated in centrosymmetric pairs through C—H···N hydrogen bonds.

3D view
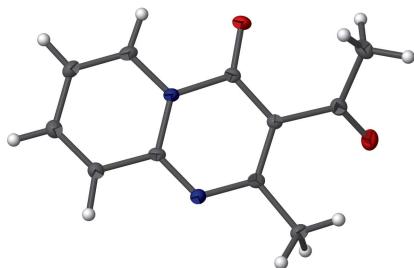
Chemical scheme
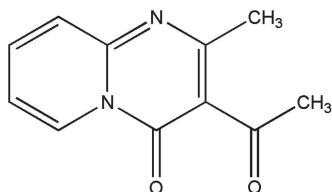
Structure description

Pyrido[1,2-*a*]pyrimidine derivatives exhibit a broad range of biological activities (Harriman *et al.*, 2003), including antibacterial (Nargund *et al.*, 1991) and antimalarial agents (Mane *et al.*, 2014). Also, the antiallergic agent ramastine (Awouters *et al.*, 1986), the antidepressant lusaperidone (Kennis *et al.*, 2000) and the tranquilizer pirenperone (Smith *et al.*, 1995) derive from such aza-bridgehead-fused heterocyclic compounds. As part of our ongoing studies (Lahmidi *et al.*, 2016) of pyridopyrimidine derivatives, we report here the synthesis and structure of the title compound.

The pyridopyrimidine unit deviates slightly from planarity as indicated by the dihedral angle of 2.76 (5)° between the two constituent rings (Fig. 1). The acetyl moiety is inclined to the pyrimidine ring by 41.21 (7)°. In the crystal, the molecules form stacks along the *a*-axis direction through head-to-head π–π stacking interactions [centroid···centroid = 3.7715 (9) Å], which are reinforced by C10—H10*A*···O1[i] [symmetry code: (i) *x* + 1, *y*, *z*] hydrogen bonds (Table 1 and Fig. 2). Pairs of stacks are associated through pairwise inversion-related C2—H2···N2[ii] [symmetry code: (ii) −*x* + 1, −*y* + 1, −*z* + 1] hydrogen bonds (Table 1 and Fig. 2).
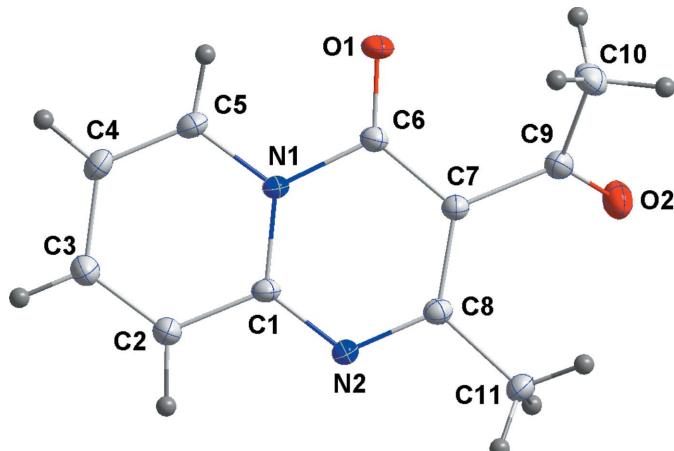
Figure 1
The molecular structure of the title compound, with displacement ellipsoids drawn at the 50% probability level.

Synthesis and crystallization

A mixture of 1-(2-methyl-4-oxo-4H-pyrido[1,2-a]pyrimidin-3-yl)butane-1,3-dione (0.7 g, 2.86 mmol) and o-phenylenedi-amine (0.93 g, 8.58 mmol) was stirred in acetic acid (20 ml) at room temperature for 8 h. After completion of the reaction as monitored by thin-layer chromatography, the reaction mixture was cooled and the excess of acetic acid removed under reduced pressure. The residue was re-dissolved in dichloro-methane and evaporated again. The solid obtained was filtered, washed with cold water and recrystallized from ethanol solution.
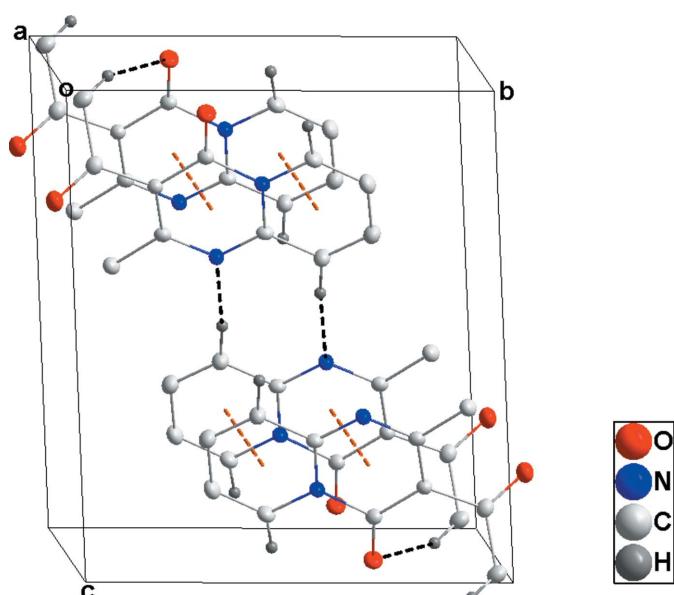

Figure 2
The crystal packing viewed approximately along the a-axis direction, with C—H⋯O and C—H⋯N hydrogen bonds shown as black dashed lines and π–π stacking interactions shown as orange dashed lines.

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

D—H⋯A	D—H	H⋯A	D⋯A	D—H⋯A
C2—H2⋯N2^i	0.997 (14)	2.494 (14)	3.4679 (15)	165.5 (11)
C5—H5⋯O1^{ii}	0.955 (15)	2.349 (16)	3.1710 (14)	143.9 (12)
C10—H10A⋯O1^{iii}	0.972 (16)	2.513 (17)	3.4153 (16)	154.5 (12)

Symmetry codes: (i) $-x+1, -y+1, -z+1$; (ii) $-x, -y+1, -z$; (iii) $x+1, y, z$.

Table 2
Experimental details.

Crystal data	
Chemical formula	$C_{11}H_{10}N_2O_2$
M_r	202.21
Crystal system, space group	Triclinic, $P\bar{1}$
Temperature (K)	100
a, b, c (Å)	3.7716 (6), 10.3959 (15), 12.3280 (18)
α, β, γ (°)	78.017 (2), 83.431 (2), 81.266 (2)
V (Å^3)	465.63 (12)
Z	2
Radiation type	Mo $K\alpha$
μ (mm^{-1})	0.10
Crystal size (mm)	$0.21 \times 0.20 \times 0.11$
Data collection	
Diffractometer	Bruker SMART APEX CCD
Absorption correction	Multi-scan (*SADABS*; Bruker, 2016)
T_{min}, T_{max}	0.88, 0.99
No. of measured, independent and observed [$I > 2\sigma(I)$] reflections	9022, 2470, 1901
R_{int}	0.027
$(\sin \theta/\lambda)_{max}$ (Å^{-1})	0.688
Refinement	
$R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, S	0.049, 0.136, 1.00
No. of reflections	2470
No. of parameters	176
H-atom treatment	All H-atom parameters refined
$\Delta\rho_{max}, \Delta\rho_{min}$ (e Å^{-3})	0.49, −0.23

Computer programs: *APEX3* and *SAINT* (Bruker, 2016), *SHELXT* (Sheldrick, 2015a), *SHELXL2014* (Sheldrick, 2015b), *DIAMOND* (Brandenburg & Putz, 2012) and *SHELXTL* (Sheldrick, 2008).

Refinement

Crystal data, data collection and structure refinement details are summarized in Table 2.

Acknowledgements

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full crystallographic data

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Crystal data

$C_{11}H_{10}N_2O_2$	$Z = 2$
$M_r = 202.21$	$F(000) = 212$
Triclinic, $P\bar{1}$	$D_x = 1.442$ Mg m^{-3}
$a = 3.7716$ (6) Å	Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
$b = 10.3959$ (15) Å	Cell parameters from 3681 reflections
$c = 12.3280$ (18) Å	$\theta = 2.4$–29.2°
$\alpha = 78.017$ (2)°	$\mu = 0.10$ mm^{-1}
$\beta = 83.431$ (2)°	$T = 100$ K
$\gamma = 81.266$ (2)°	Block, colourless
$V = 465.63$ (12) Å^3	$0.21 \times 0.20 \times 0.11$ mm

Data collection

Bruker SMART APEX CCD diffractometer	9022 measured reflections
Radiation source: fine-focus sealed tube	2470 independent reflections
Graphite monochromator	1901 reflections with $I > 2\sigma(I)$
Detector resolution: 8.3333 pixels mm^{-1}	$R_{int} = 0.027$
φ and ω scans	$\theta_{max} = 29.3°$, $\theta_{min} = 1.7°$
Absorption correction: multi-scan (*SADABS*; Bruker, 2016)	$h = -5\rightarrow5$
	$k = -14\rightarrow14$
$T_{min} = 0.88$, $T_{max} = 0.99$	$l = -16\rightarrow16$

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	
$R[F^2 > 2\sigma(F^2)] = 0.049$	Hydrogen site location: difference Fourier map
$wR(F^2) = 0.136$	All H-atom parameters refined
$S = 1.00$	$w = 1/[\sigma^2(F_o^2) + (0.0998P)^2]$
2470 reflections	where $P = (F_o^2 + 2F_c^2)/3$
176 parameters	$(\Delta/\sigma)_{max} < 0.001$
0 restraints	$\Delta\rho_{max} = 0.49$ e Å^{-3}
Primary atom site location: structure-invariant direct methods	$\Delta\rho_{min} = -0.23$ e Å^{-3}

Special details

Experimental. The diffraction data were obtained from 3 sets of 400 frames, each of width 0.5° in ω, colllected at $\varphi = 0.00$, 90.00 and 180.00° and 2 sets of 800 frames, each of width 0.45° in φ, collected at $\omega = -30.00$ and 210.00°. The scan time was 25 sec/frame.

Geometry. All esds (except the esd in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell esds are taken into account individually in the estimation of esds in distances, angles and torsion angles; correlations between esds in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell esds is used for estimating esds 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 $F^2 > 2\text{sigma}(F^2)$ 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 ($Å^2$)

	x	y	z	$U_{iso}*/U_{eq}$
O1	0.2926 (2)	0.34971 (8)	0.07890 (6)	0.0208 (2)
O2	0.5557 (2)	0.00837 (8)	0.28674 (7)	0.0249 (2)
N1	0.2518 (2)	0.47066 (9)	0.21721 (7)	0.0139 (2)
N2	0.4884 (3)	0.37592 (9)	0.39182 (7)	0.0152 (2)
C1	0.3175 (3)	0.47757 (10)	0.32396 (8)	0.0142 (2)
C2	0.1954 (3)	0.59856 (11)	0.36119 (9)	0.0182 (3)
H2	0.252 (4)	0.5959 (14)	0.4387 (12)	0.024 (3)*
C3	0.0194 (3)	0.70330 (12)	0.29381 (9)	0.0207 (3)
H3	−0.064 (4)	0.7835 (15)	0.3192 (13)	0.033 (4)*
C4	−0.0399 (3)	0.69249 (12)	0.18479 (9)	0.0196 (3)
H4	−0.161 (4)	0.7630 (15)	0.1353 (12)	0.028 (4)*
C5	0.0787 (3)	0.57829 (11)	0.14802 (9)	0.0172 (3)
H5	0.039 (4)	0.5650 (14)	0.0762 (13)	0.031 (4)*
C6	0.3588 (3)	0.34940 (10)	0.17422 (8)	0.0147 (2)
C7	0.5185 (3)	0.24174 (10)	0.25242 (8)	0.0140 (2)
C8	0.5827 (3)	0.25987 (11)	0.35758 (8)	0.0144 (2)
C9	0.6088 (3)	0.10916 (11)	0.21968 (9)	0.0174 (3)
C10	0.7731 (4)	0.10147 (12)	0.10354 (10)	0.0222 (3)
H10A	0.912 (4)	0.1737 (16)	0.0714 (13)	0.036 (4)*
H10B	0.933 (5)	0.0147 (18)	0.1094 (14)	0.048 (5)*
H10C	0.586 (5)	0.1028 (16)	0.0571 (13)	0.039 (4)*
C11	0.7671 (3)	0.15253 (11)	0.44132 (9)	0.0177 (3)
H11A	0.592 (4)	0.1061 (14)	0.4931 (12)	0.029 (4)*
H11B	0.899 (4)	0.1935 (14)	0.4844 (12)	0.026 (4)*
H11C	0.940 (4)	0.0891 (14)	0.4056 (11)	0.028 (4)*

Atomic displacement parameters ($Å^2$)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.0308 (5)	0.0203 (4)	0.0126 (4)	−0.0014 (3)	−0.0067 (3)	−0.0046 (3)
O2	0.0339 (5)	0.0155 (4)	0.0239 (4)	−0.0019 (4)	0.0002 (4)	−0.0030 (3)
N1	0.0176 (5)	0.0136 (5)	0.0107 (4)	−0.0011 (4)	−0.0036 (3)	−0.0020 (3)
N2	0.0189 (5)	0.0137 (5)	0.0127 (4)	−0.0002 (4)	−0.0033 (3)	−0.0022 (3)
C1	0.0157 (6)	0.0169 (5)	0.0102 (5)	−0.0019 (4)	−0.0024 (4)	−0.0027 (4)
C2	0.0228 (6)	0.0164 (5)	0.0155 (5)	0.0007 (4)	−0.0042 (4)	−0.0046 (4)
C3	0.0258 (7)	0.0177 (6)	0.0183 (6)	0.0015 (5)	−0.0026 (5)	−0.0054 (4)
C4	0.0222 (6)	0.0161 (5)	0.0183 (5)	0.0016 (4)	−0.0053 (4)	0.0003 (4)

C5	0.0202 (6)	0.0182 (6)	0.0125 (5)	−0.0014 (4)	−0.0046 (4)	−0.0006 (4)
C6	0.0172 (6)	0.0150 (5)	0.0125 (5)	−0.0025 (4)	−0.0021 (4)	−0.0035 (4)
C7	0.0160 (6)	0.0140 (5)	0.0120 (5)	−0.0012 (4)	−0.0015 (4)	−0.0025 (4)
C8	0.0146 (5)	0.0163 (5)	0.0118 (5)	−0.0020 (4)	−0.0006 (4)	−0.0020 (4)
C9	0.0180 (6)	0.0172 (5)	0.0176 (5)	−0.0004 (4)	−0.0036 (4)	−0.0051 (4)
C10	0.0268 (7)	0.0218 (6)	0.0189 (6)	−0.0020 (5)	0.0007 (5)	−0.0086 (4)
C11	0.0199 (6)	0.0171 (5)	0.0148 (5)	0.0012 (4)	−0.0038 (4)	−0.0012 (4)

Geometric parameters (Å, º)

O1—C6	1.2283 (13)	C4—H4	0.947 (16)
O2—C9	1.2197 (14)	C5—H5	0.955 (15)
N1—C5	1.3838 (14)	C6—C7	1.4229 (15)
N1—C1	1.3849 (13)	C7—C8	1.4007 (14)
N1—C6	1.4515 (13)	C7—C9	1.4952 (14)
N2—C1	1.3346 (14)	C8—C11	1.4984 (15)
N2—C8	1.3443 (13)	C9—C10	1.5066 (16)
C1—C2	1.4214 (14)	C10—H10A	0.972 (16)
C2—C3	1.3593 (16)	C10—H10B	1.001 (18)
C2—H2	0.997 (14)	C10—H10C	0.955 (18)
C3—C4	1.4171 (16)	C11—H11A	0.974 (15)
C3—H3	0.948 (15)	C11—H11B	0.964 (15)
C4—C5	1.3530 (15)	C11—H11C	0.990 (14)
C5—N1—C1	121.40 (9)	C8—C7—C6	120.56 (9)
C5—N1—C6	117.53 (8)	C8—C7—C9	121.33 (10)
C1—N1—C6	121.07 (9)	C6—C7—C9	118.11 (9)
C1—N2—C8	118.71 (9)	N2—C8—C7	122.90 (10)
N2—C1—N1	122.63 (9)	N2—C8—C11	113.96 (9)
N2—C1—C2	119.61 (9)	C7—C8—C11	123.13 (9)
N1—C1—C2	117.76 (9)	O2—C9—C7	120.39 (9)
C3—C2—C1	120.72 (10)	O2—C9—C10	120.45 (10)
C3—C2—H2	126.2 (8)	C7—C9—C10	119.15 (10)
C1—C2—H2	113.1 (8)	C9—C10—H10A	112.7 (9)
C2—C3—C4	119.75 (10)	C9—C10—H10B	106.3 (10)
C2—C3—H3	120.5 (9)	H10A—C10—H10B	109.8 (14)
C4—C3—H3	119.8 (9)	C9—C10—H10C	109.1 (9)
C5—C4—C3	120.04 (10)	H10A—C10—H10C	109.2 (13)
C5—C4—H4	117.5 (9)	H10B—C10—H10C	109.6 (14)
C3—C4—H4	122.4 (9)	C8—C11—H11A	110.8 (9)
C4—C5—N1	120.31 (10)	C8—C11—H11B	108.1 (9)
C4—C5—H5	123.4 (9)	H11A—C11—H11B	107.0 (12)
N1—C5—H5	116.3 (9)	C8—C11—H11C	112.1 (8)
O1—C6—C7	128.13 (10)	H11A—C11—H11C	110.6 (12)
O1—C6—N1	117.88 (9)	H11B—C11—H11C	108.0 (12)
C7—C6—N1	113.94 (9)		
C8—N2—C1—N1	−3.80 (17)	C1—N1—C6—C7	2.35 (15)

C8—N2—C1—C2	176.08 (9)	O1—C6—C7—C8	178.50 (11)
C5—N1—C1—N2	−179.02 (10)	N1—C6—C7—C8	−4.19 (16)
C6—N1—C1—N2	1.65 (17)	O1—C6—C7—C9	−2.57 (18)
C5—N1—C1—C2	1.10 (16)	N1—C6—C7—C9	174.74 (9)
C6—N1—C1—C2	−178.24 (9)	C1—N2—C8—C7	1.82 (17)
N2—C1—C2—C3	−179.70 (11)	C1—N2—C8—C11	−179.04 (9)
N1—C1—C2—C3	0.18 (17)	C6—C7—C8—N2	2.34 (17)
C1—C2—C3—C4	−0.70 (19)	C9—C7—C8—N2	−176.56 (9)
C2—C3—C4—C5	−0.05 (19)	C6—C7—C8—C11	−176.72 (10)
C3—C4—C5—N1	1.32 (18)	C9—C7—C8—C11	4.39 (17)
C1—N1—C5—C4	−1.87 (17)	C8—C7—C9—O2	39.27 (16)
C6—N1—C5—C4	177.49 (10)	C6—C7—C9—O2	−139.66 (11)
C5—N1—C6—O1	0.60 (16)	C8—C7—C9—C10	−139.53 (12)
C1—N1—C6—O1	179.95 (10)	C6—C7—C9—C10	41.55 (15)
C5—N1—C6—C7	−177.01 (9)		

Hydrogen-bond geometry (Å, º)

D—H···A	D—H	H···A	D···A	D—H···A
C2—H2···N2[i]	0.997 (14)	2.494 (14)	3.4679 (15)	165.5 (11)
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Symmetry codes: (i) −x+1, −y+1, −z+1; (ii) −x, −y+1, −z; (iii) x+1, y, z.