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Ethyl 2-(2-amino-5-methyl-1,2,4-triazolo-[1,5-*a*]pyrimidin-7-yl)acetate

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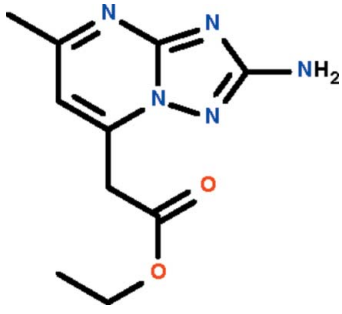
Key indicators: single-crystal X-ray study; T = 293 K; mean σ(C–C) = 0.002 Å; R factor = 0.050; wR factor = 0.138; data-to-parameter ratio = 28.3.

The nine-membered fused-ring of the title compound, $C_{10}H_{13}N_5O_2$, is approximately planar [maximum deviation = 0.012 (1) Å]; the bond angle at the methylene C atom is 111.33 (10)°. In the crystal, the amino group forms hydrogen bonds to the N atoms of the triazole rings of adjacent molecules, generating a ribbon running along the *a* axis.

Related literature

For a related molecule, see: Fettouhi *et al.* (1996).



Experimental

Crystal data

$C_{10}H_{13}N_5O_2$
M_r = 235.25
Monoclinic, $C2/c$
a = 22.9635 (4) Å
b = 7.7447 (1) Å
c = 14.7017 (3) Å
β = 124.574 (1)°
V = 2152.87 (6) Å^3
Z = 8
Mo $K\alpha$ radiation
μ = 0.11 mm^{-1}
T = 293 K
0.32 × 0.21 × 0.20 mm

Data collection

Bruker APEX DUO diffractometer
21320 measured reflections
4613 independent reflections
3058 reflections with $I > 2\sigma(I)$
R_{int} = 0.050

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.050
$wR(F^2)$ = 0.138
S = 1.02
4613 reflections
163 parameters
2 restraints
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.50 e Å^{-3}
$\Delta\rho_{min}$ = −0.32 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
N5—H1···N2^{i}	0.88 (1)	2.24 (1)	3.095 (1)	166 (2)
N5—H2···N3^{ii}	0.89 (1)	2.15 (1)	3.037 (2)	175 (2)

Symmetry codes: (i) $-x+\frac{3}{2}, y+\frac{1}{2}, -z+\frac{3}{2}$; (ii) $-x+\frac{3}{2}, y-\frac{1}{2}, -z+\frac{3}{2}$.

Data collection: *APEX2* (Bruker, 2010); cell refinement: *SAINT* (Bruker, 2010); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *X-SEED* (Barbour, 2001); software used to prepare material for publication: *publCIF* (Westrip, 2010).

We thank Université Mohammed V-Agdal and the University of Malaya for supporting this study.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: XU5412).

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supplementary materials

Acta Cryst. (2012). E**68**, o239 [**doi:10.1107/S1600536811054468**]

Ethyl 2-(2-amino-5-methyl-1,2,4-triazolo[1,5-*a*]pyrimidin-7-yl)acetate

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Comment

We reported the reaction of 3-diamino-1,2,4-triazole and 4-hydroxy-6-methyl-pyran-2-one to form ethyl 2-(2-amino-5-methyl-[1,2,4]triazolo[1,5-*a*]pyrimidin-7-yl)acetate (Fettouhi *et al.*, 1996), which is a member of a class of antiviral compounds. The use of 3,5-diamino-1,2,4-triazol with the pyrone gave the analogous amino-substituted compound (Scheme I). The nine-membered fused-ring of $C_{10}H_{13}N_5O_2$ is planar; the methylene unit connecting the fused-ring and the ethoxycarbonyl unit is slightly opened up to 111.33 (10)° (Fig. 1). The amino group forms hydrogen bonds to the N atoms of the triazole rings of adjacent molecules by a two-fold symmetry operation to generate a ribbon running along the *a*-axis of the monoclinic unit cell (Table 1).

Experimental

A solution of 3,5-diamino-1,2,4-triazole (1 g, 10 mmol) and 4-hydroxy-6-methyl-pyran-2-one (1.6g, 12.6 mmol) in ethanol (30 ml) was heated for 12 hours. The solvent was removed by evaporation and the residue recrystallized from ethanol to afford the colorless crystals.

Refinement

Carbon-bound H-atoms were placed in calculated positions (C–H 0.93–0.97 Å) and were included in the refinement in the riding model approximation, with *U*(H) set to 1.2–1.5*U*(C).

The amino H-atoms were located in a difference Fourier map, and were refined with a distance restraint of N–H 0.88±0.01 Å; their temperature factors were refined.

Figures

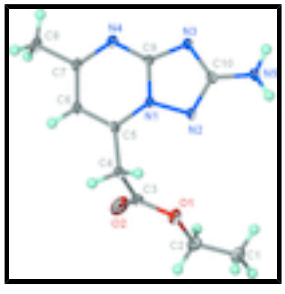

Fig. 1. Thermal ellipsoid plot (Barbour, 2001) of $C_{10}H_{13}N_5O_2$ at the 70% probability level; hydrogen atoms are drawn as spheres of arbitrary radius.

Ethyl 2-(2-amino-5-methyl-1,2,4-triazolo[1,5-*a*]pyrimidin-7-yl)acetate

Crystal data

$C_{10}H_{13}N_5O_2$	$F(000) = 992$
$M_r = 235.25$	$D_x = 1.452$ Mg m^{-3}
Monoclinic, *C*2/*c*	Mo *K*α radiation, λ = 0.71073 Å
Hall symbol: -C 2yc	Cell parameters from 3608 reflections
a = 22.9635 (4) Å	θ = 2.8–35.5°
b = 7.7447 (1) Å	μ = 0.11 mm^{-1}
c = 14.7017 (3) Å	*T* = 293 K
β = 124.574 (1)°	Prism, colorless
V = 2152.87 (6) Å^3	0.32 × 0.21 × 0.20 mm
Z = 8	

Data collection

Bruker APEX DUO diffractometer	3058 reflections with $I > 2\sigma(I)$
Radiation source: fine-focus sealed tube	$R_{int} = 0.050$
graphite	$\theta_{max} = 34.7°$, $\theta_{min} = 2.8°$
ω scans	$h = -35 \rightarrow 36$
21320 measured reflections	$k = -12 \rightarrow 9$
4613 independent reflections	$l = -23 \rightarrow 22$

Refinement

Refinement on F^2	Primary atom site location: structure-invariant direct methods
Least-squares matrix: full	Secondary atom site location: difference Fourier map
$R[F^2 > 2\sigma(F^2)] = 0.050$	Hydrogen site location: inferred from neighbouring sites
$wR(F^2) = 0.138$	H atoms treated by a mixture of independent and constrained refinement
$S = 1.02$	$w = 1/[\sigma^2(F_o^2) + (0.0666P)^2 + 0.6364P]$ where $P = (F_o^2 + 2F_c^2)/3$
4613 reflections	$(\Delta/\sigma)_{max} = 0.001$
163 parameters	$\Delta\rho_{max} = 0.50$ e Å^{-3}
2 restraints	$\Delta\rho_{min} = -0.32$ e Å^{-3}

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å^2)

	x	*y*	*z*	U_{iso}*/U_{eq}
O1	0.66634 (4)	0.58516 (12)	0.48590 (7)	0.01669 (18)
O2	0.59517 (5)	0.70201 (15)	0.31780 (7)	0.0279 (2)
N1	0.60826 (5)	0.99885 (13)	0.53229 (7)	0.01076 (18)
N2	0.67164 (5)	0.93485 (13)	0.61966 (7)	0.01190 (18)

N3	0.66734 (5)	1.23064 (13)	0.63055 (8)	0.01260 (19)
N4	0.55068 (5)	1.27125 (14)	0.46404 (8)	0.01363 (19)
N5	0.77145 (5)	1.08068 (14)	0.76545 (8)	0.0147 (2)
C1	0.77877 (6)	0.45604 (19)	0.56140 (11)	0.0200 (3)
H1A	0.8124	0.4183	0.5466	0.030*
H1B	0.7646	0.3596	0.5856	0.030*
H1C	0.8000	0.5427	0.6181	0.030*
C2	0.71486 (6)	0.53072 (18)	0.45759 (10)	0.0177 (2)
H2A	0.7284	0.6284	0.4321	0.021*
H2B	0.6928	0.4445	0.3995	0.021*
C3	0.60866 (6)	0.66849 (16)	0.40788 (9)	0.0151 (2)
C4	0.56130 (6)	0.71606 (16)	0.44533 (9)	0.0140 (2)
H4A	0.5150	0.6649	0.3955	0.017*
H4B	0.5811	0.6694	0.5186	0.017*
C5	0.55386 (5)	0.90755 (15)	0.44764 (9)	0.0118 (2)
C6	0.49644 (6)	1.00289 (16)	0.36969 (9)	0.0133 (2)
H6	0.4573	0.9479	0.3099	0.016*
C7	0.49650 (6)	1.18385 (16)	0.37998 (9)	0.0135 (2)
C8	0.43393 (6)	1.28736 (18)	0.29316 (10)	0.0188 (2)
H8A	0.4375	1.4029	0.3195	0.028*
H8B	0.3912	1.2349	0.2773	0.028*
H8C	0.4331	1.2904	0.2271	0.028*
C9	0.60642 (6)	1.17669 (15)	0.54011 (9)	0.0114 (2)
C10	0.70439 (6)	1.08132 (15)	0.67461 (9)	0.0115 (2)
H1	0.7866 (9)	1.1735 (16)	0.8069 (12)	0.027 (4)*
H2	0.7920 (8)	0.9812 (15)	0.7978 (13)	0.027 (4)*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.0130 (4)	0.0209 (5)	0.0142 (4)	0.0049 (3)	0.0066 (3)	0.0026 (3)
O2	0.0328 (5)	0.0336 (6)	0.0165 (4)	0.0168 (5)	0.0135 (4)	0.0082 (4)
N1	0.0087 (4)	0.0096 (4)	0.0101 (4)	0.0000 (3)	0.0030 (3)	−0.0006 (3)
N2	0.0087 (4)	0.0104 (5)	0.0101 (4)	0.0000 (3)	0.0015 (3)	0.0003 (3)
N3	0.0108 (4)	0.0102 (4)	0.0118 (4)	−0.0001 (3)	0.0034 (3)	−0.0002 (3)
N4	0.0108 (4)	0.0114 (5)	0.0145 (4)	0.0010 (3)	0.0047 (3)	0.0007 (4)
N5	0.0122 (4)	0.0110 (5)	0.0113 (4)	−0.0002 (4)	0.0009 (3)	−0.0004 (4)
C1	0.0147 (5)	0.0207 (6)	0.0225 (6)	0.0038 (4)	0.0094 (4)	0.0025 (5)
C2	0.0178 (5)	0.0181 (6)	0.0180 (5)	0.0034 (5)	0.0106 (4)	−0.0006 (5)
C3	0.0148 (5)	0.0116 (5)	0.0138 (5)	0.0009 (4)	0.0050 (4)	−0.0017 (4)
C4	0.0118 (4)	0.0102 (5)	0.0153 (5)	−0.0013 (4)	0.0049 (4)	−0.0020 (4)
C5	0.0093 (4)	0.0116 (5)	0.0116 (4)	−0.0015 (4)	0.0043 (4)	−0.0025 (4)
C6	0.0091 (4)	0.0140 (5)	0.0115 (4)	−0.0001 (4)	0.0028 (4)	−0.0016 (4)
C7	0.0100 (4)	0.0147 (6)	0.0125 (4)	0.0007 (4)	0.0044 (4)	0.0003 (4)
C8	0.0122 (5)	0.0170 (6)	0.0178 (5)	0.0039 (4)	0.0028 (4)	0.0027 (5)
C9	0.0113 (4)	0.0087 (5)	0.0124 (4)	0.0004 (4)	0.0057 (4)	0.0000 (4)
C10	0.0112 (4)	0.0107 (5)	0.0104 (4)	−0.0007 (4)	0.0048 (4)	0.0001 (4)

Geometric parameters (Å, °)

O1—C3	1.3276 (14)	C1—H1B	0.9600
O1—C2	1.4555 (15)	C1—H1C	0.9600
O2—C3	1.2049 (15)	C2—H2A	0.9700
N1—C5	1.3582 (14)	C2—H2B	0.9700
N1—N2	1.3755 (12)	C3—C4	1.5169 (17)
N1—C9	1.3847 (15)	C4—C5	1.4954 (17)
N2—C10	1.3474 (15)	C4—H4A	0.9700
N3—C9	1.3374 (14)	C4—H4B	0.9700
N3—C10	1.3621 (15)	C5—C6	1.3720 (15)
N4—C7	1.3373 (14)	C6—C7	1.4096 (17)
N4—C9	1.3419 (14)	C6—H6	0.9300
N5—C10	1.3494 (14)	C7—C8	1.5016 (16)
N5—H1	0.877 (9)	C8—H8A	0.9600
N5—H2	0.887 (9)	C8—H8B	0.9600
C1—C2	1.5085 (17)	C8—H8C	0.9600
C1—H1A	0.9600		
C3—O1—C2	116.37 (9)	C5—C4—H4A	109.4
C5—N1—N2	127.13 (10)	C3—C4—H4A	109.4
C5—N1—C9	122.57 (9)	C5—C4—H4B	109.4
N2—N1—C9	110.30 (9)	C3—C4—H4B	109.4
C10—N2—N1	101.03 (9)	H4A—C4—H4B	108.0
C9—N3—C10	103.08 (10)	N1—C5—C6	115.69 (11)
C7—N4—C9	116.18 (10)	N1—C5—C4	118.66 (9)
C10—N5—H1	117.7 (11)	C6—C5—C4	125.64 (10)
C10—N5—H2	119.6 (11)	C5—C6—C7	120.23 (10)
H1—N5—H2	117.3 (15)	C5—C6—H6	119.9
C2—C1—H1A	109.5	C7—C6—H6	119.9
C2—C1—H1B	109.5	N4—C7—C6	123.11 (10)
H1A—C1—H1B	109.5	N4—C7—C8	117.06 (11)
C2—C1—H1C	109.5	C6—C7—C8	119.83 (10)
H1A—C1—H1C	109.5	C7—C8—H8A	109.5
H1B—C1—H1C	109.5	C7—C8—H8B	109.5
O1—C2—C1	106.55 (10)	H8A—C8—H8B	109.5
O1—C2—H2A	110.4	C7—C8—H8C	109.5
C1—C2—H2A	110.4	H8A—C8—H8C	109.5
O1—C2—H2B	110.4	H8B—C8—H8C	109.5
C1—C2—H2B	110.4	N3—C9—N4	128.53 (11)
H2A—C2—H2B	108.6	N3—C9—N1	109.22 (9)
O2—C3—O1	124.23 (11)	N4—C9—N1	122.23 (10)
O2—C3—C4	123.83 (11)	N2—C10—N5	121.70 (10)
O1—C3—C4	111.95 (10)	N2—C10—N3	116.37 (9)
C5—C4—C3	111.33 (10)	N5—C10—N3	121.86 (10)
C5—N1—N2—C10	178.81 (11)	C9—N4—C7—C8	−179.26 (11)
C9—N1—N2—C10	−0.44 (12)	C5—C6—C7—N4	−0.35 (18)
C3—O1—C2—C1	174.51 (11)	C5—C6—C7—C8	178.90 (11)
C2—O1—C3—O2	−0.81 (19)	C10—N3—C9—N4	−178.56 (12)

C2—O1—C3—C4	178.92 (10)	C10—N3—C9—N1	0.13 (12)
O2—C3—C4—C5	−62.89 (16)	C7—N4—C9—N3	178.85 (11)
O1—C3—C4—C5	117.38 (11)	C7—N4—C9—N1	0.31 (16)
N2—N1—C5—C6	−179.22 (10)	C5—N1—C9—N3	−179.09 (10)
C9—N1—C5—C6	−0.05 (16)	N2—N1—C9—N3	0.20 (13)
N2—N1—C5—C4	−0.24 (17)	C5—N1—C9—N4	−0.30 (17)
C9—N1—C5—C4	178.93 (10)	N2—N1—C9—N4	178.99 (10)
C3—C4—C5—N1	−76.46 (13)	N1—N2—C10—N5	−176.45 (10)
C3—C4—C5—C6	102.41 (13)	N1—N2—C10—N3	0.56 (13)
N1—C5—C6—C7	0.35 (16)	C9—N3—C10—N2	−0.45 (13)
C4—C5—C6—C7	−178.55 (11)	C9—N3—C10—N5	176.55 (10)
C9—N4—C7—C6	0.00 (17)		

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N5—H1···N2^{i}	0.88 (1)	2.24 (1)	3.095 (1)	166 (2)
N5—H2···N3^{ii}	0.89 (1)	2.15 (1)	3.037 (2)	175 (2)

Symmetry codes: (i) −*x*+3/2, *y*+1/2, −*z*+3/2; (ii) −*x*+3/2, *y*−1/2, −*z*+3/2.

Fig. 1