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3,4-Dimethyl-1*H*-1,2,4-triazepino[2,3-*a*]-benzimidazol-2(3*H*)-one

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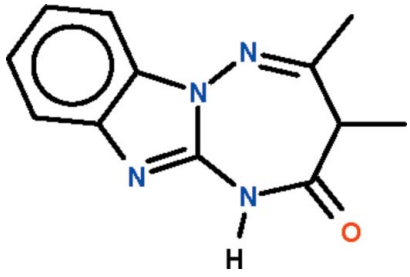
Key indicators: single-crystal X-ray study; T = 293 K; mean σ(C–C) = 0.003 Å; R factor = 0.049; wR factor = 0.157; data-to-parameter ratio = 19.3.

In the molecule of the title compound, $C_{12}H_{12}N_4O$, a C atom and an N atom of the benzimidazole fused-ring portion are part of a seven-membered ring; this ring adopts a boat-shaped conformation (with the fused-ring atoms representing the stern and the sp^3-hybridized C atom the prow). The amino group is a hydrogen-bond donor to the imidazole group of an inversion-related molecule, the pair of N—H···N hydrogen bonds giving rise to a hydrogen-bonded dimer.

Related literature

For the synthesis, see: Romano *et al.* (1988).



Experimental

Crystal data

$C_{12}H_{12}N_4O$
M_r = 228.26
Monoclinic, $P2_1/c$
a = 7.2899 (3) Å
b = 14.4888 (5) Å
c = 10.9932 (4) Å
β = 104.314 (1)°
V = 1125.08 (7) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.09 mm^{-1}
T = 293 K
0.21 × 0.19 × 0.16 mm

Data collection

Bruker X8 APEXII diffractometer
14670 measured reflections
3095 independent reflections
1823 reflections with $I > 2\sigma(I)$
R_{int} = 0.034

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.049
$wR(F^2)$ = 0.157
S = 1.01
3095 reflections
160 parameters
1 restraint
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.24 e Å^{-3}
$\Delta\rho_{min}$ = −0.19 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
N1—H1···N4^{i}	0.86 (1)	2.01 (1)	2.867 (2)	174 (2)

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

Data collection: *APEX2* (Bruker, 2008); cell refinement: *SAINT* (Bruker, 2008); 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: SI2255).

References

Barbour, L. J. (2001). *J. Supramol. Chem.* **1**, 189–191.
Bruker (2008). *APEX2* and *SAINT*. Bruker AXS Inc., Madison, Wisconsin, USA.
Romano, C., De la Cuesta, E., Avendano, C., Florencio, F. & Sainz-Aparicio, J. (1988). *Tetrahedron*, **44**, 7185–7192.
Sheldrick, G. M. (2008). *Acta Cryst.* A**64**, 112–122.
Westrip, S. P. (2010). *publCIF*. In preparation.

supporting information

Acta Cryst. (2010). E**66**, o1093 [https://doi.org/10.1107/S1600536810013498]

3,4-Dimethyl-1*H*-1,2,4-triazepino[2,3-*a*]benzimidazol-2(3*H*)-one

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S1. Comment

1,2-Diaminobenzimidazoles react with *β*-dicarbonyl compounds to form 1,2,4-triazepino[2,3-*a*]benzimidazoles (Romano *et al.*, 1988), a class of compounds used in the treatment of neuronal disorders. The title compound (Scheme I, Fig. 1) was synthesized from 2-aminobenzimidazole and ethyl 2-methylacetoacetate. A carbon atom and a nitrogen atom of the benzimidazole fused-ring portion are part of a seven-membered ring; this ring adopts a boat-shaped conformation (with the fused-ring atoms representing the stern and the sp^3-hybridized carbon atom the prow). Its methyl substitent occupies a quasi-equatorial position. The amino group is hydrogen-bond donor to the imidazole group (Table 1) of an inversion-related molecule, the pair of N—H···N hydrogen bonds (Fig. 1) giving rise to a hydrogen-bonded dimer.

S2. Experimental

2-Aminobenzimidazole (1 g, 6.75 mmol) and a slight excess of ethyl 2-methylacetoacetate (1.69 ml) were refluxed in xylene (10 ml) and acetic acid (0.5 ml) for 3 hours. The mixture was concentrated under reduced pressure and the resulting residue was recrystallized from ethanol. Brown crystals were isolated when the solvent was allowed to evaporate.

S3. Refinement

Carbon-bound H-atoms were placed in calculated positions (C—H 0.93–0.98 Å) and were included in the refinement in the riding model approximation, with U_{iso}(H) set to 1.2U_{eq}(C). The amino H-atom was located in a difference Fourier map, and was refined with a distance restraint of N–H 0.86 (1) Å.

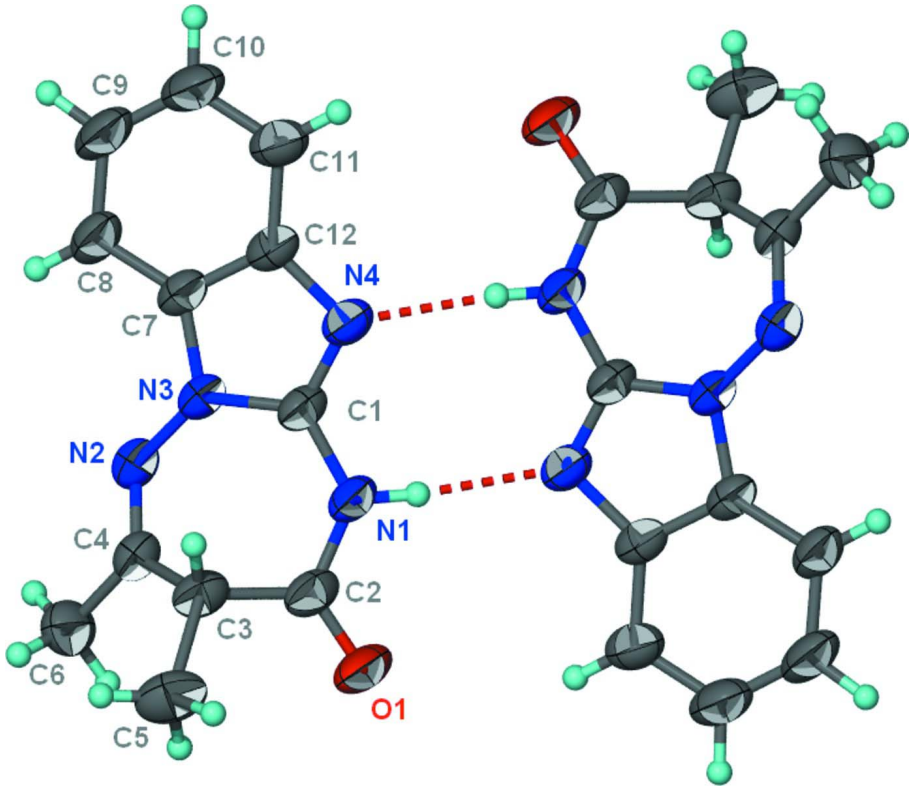

Figure 1

Thermal ellipsoid plot (Barbour, 2001) of the hydrogen-bonded dimeric structure of $C_{12}H_{12}N_4O$ at the 50% probability level; hydrogen atoms are drawn as arbitrary radius. Symmetry code for the inversion related molecule: 1 - *x*, 1 - *y*, 1 - *z*.

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Crystal data

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Monoclinic, $P2_1/c$
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$b = 14.4888$ (5) Å
$c = 10.9932$ (4) Å
$\beta = 104.314$ (1)°
$V = 1125.08$ (7) Å^3
$Z = 4$
$F(000) = 480$
$D_x = 1.348$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 3188 reflections
$\theta = 2.4$–24.8°
$\mu = 0.09$ mm^{-1}
$T = 293$ K
Prism, brown
0.21 × 0.19 × 0.16 mm

Data collection

Bruker X8 APEXII diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
φ and ω scans
14670 measured reflections
3095 independent reflections
1823 reflections with $I > 2\sigma(I)$
$R_{int} = 0.034$
$\theta_{max} = 29.4°$, $\theta_{min} = 2.4°$
$h = -9 \rightarrow 10$
$k = -20 \rightarrow 19$
$l = -15 \rightarrow 13$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.049$
$wR(F^2) = 0.157$
$S = 1.01$
3095 reflections
160 parameters
1 restraint
Primary atom site location: structure-invariant direct methods
Secondary atom site location: difference Fourier map
Hydrogen site location: inferred from neighbouring sites
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0742P)^2 + 0.1838P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.24$ e Å^{-3}
$\Delta\rho_{min} = -0.19$ e Å^{-3}

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

	x	*y*	*z*	U_{iso}*/U_{eq}
O1	0.40956 (19)	0.51793 (12)	0.14398 (13)	0.0762 (5)
N1	0.5626 (2)	0.53766 (12)	0.34574 (15)	0.0574 (4)
H1	0.486 (3)	0.5002 (13)	0.3696 (19)	0.075 (7)*
N2	0.95622 (19)	0.59291 (9)	0.32932 (14)	0.0508 (4)
N3	0.86966 (19)	0.60627 (9)	0.42759 (13)	0.0475 (4)
N4	0.6799 (2)	0.58224 (10)	0.55706 (13)	0.0552 (4)
C1	0.6977 (2)	0.57641 (11)	0.44172 (16)	0.0488 (4)
C2	0.5294 (2)	0.55903 (14)	0.22125 (17)	0.0569 (5)
C3	0.6491 (2)	0.63705 (13)	0.18892 (16)	0.0551 (5)
H3	0.6428	0.6886	0.2454	0.066*
C4	0.8529 (2)	0.60475 (11)	0.21815 (17)	0.0498 (4)
C5	0.5699 (3)	0.67076 (16)	0.05638 (19)	0.0797 (7)
H5A	0.6472	0.7202	0.0387	0.120*
H5B	0.4429	0.6926	0.0475	0.120*
H5C	0.5692	0.6210	−0.0014	0.120*
C6	0.9446 (3)	0.58710 (16)	0.11287 (19)	0.0694 (6)
H6A	1.0719	0.5659	0.1463	0.104*
H6B	0.9466	0.6432	0.0667	0.104*
H6C	0.8742	0.5409	0.0580	0.104*
C7	0.9748 (2)	0.62947 (10)	0.54769 (16)	0.0486 (4)
C8	1.1602 (3)	0.65870 (13)	0.59037 (19)	0.0636 (5)
H8	1.2399	0.6664	0.5369	0.076*
C9	1.2194 (3)	0.67573 (16)	0.7180 (2)	0.0790 (7)
H9	1.3432	0.6950	0.7517	0.095*
C10	1.0993 (4)	0.66492 (16)	0.7969 (2)	0.0780 (7)
H10	1.1444	0.6782	0.8819	0.094*
C11	0.9147 (3)	0.63502 (14)	0.75350 (18)	0.0653 (5)
H11	0.8348	0.6279	0.8070	0.078*
C12	0.8542 (3)	0.61603 (11)	0.62561 (16)	0.0516 (4)

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.0480 (8)	0.1131 (12)	0.0627 (9)	−0.0193 (7)	0.0049 (6)	−0.0313 (8)

N1	0.0401 (8)	0.0756 (10)	0.0538 (9)	−0.0152 (7)	0.0068 (7)	−0.0171 (7)
N2	0.0385 (7)	0.0536 (8)	0.0580 (9)	−0.0022 (6)	0.0074 (7)	−0.0016 (6)
N3	0.0368 (7)	0.0521 (8)	0.0487 (8)	−0.0038 (6)	0.0012 (6)	−0.0036 (6)
N4	0.0461 (8)	0.0639 (9)	0.0520 (9)	−0.0080 (7)	0.0055 (7)	−0.0121 (7)
C1	0.0378 (8)	0.0540 (9)	0.0505 (9)	−0.0027 (7)	0.0035 (7)	−0.0103 (7)
C2	0.0349 (9)	0.0763 (12)	0.0565 (10)	0.0011 (8)	0.0056 (8)	−0.0202 (9)
C3	0.0468 (10)	0.0591 (10)	0.0536 (10)	0.0051 (8)	0.0014 (8)	−0.0080 (8)
C4	0.0404 (9)	0.0497 (9)	0.0563 (10)	−0.0035 (7)	0.0066 (8)	−0.0038 (7)
C5	0.0758 (15)	0.0797 (14)	0.0688 (13)	0.0114 (11)	−0.0103 (11)	−0.0030 (11)
C6	0.0578 (12)	0.0891 (15)	0.0640 (12)	−0.0009 (10)	0.0202 (10)	−0.0015 (10)
C7	0.0439 (9)	0.0417 (8)	0.0517 (9)	−0.0036 (7)	−0.0045 (8)	0.0010 (7)
C8	0.0482 (10)	0.0662 (11)	0.0657 (11)	−0.0128 (8)	−0.0065 (9)	0.0088 (9)
C9	0.0628 (13)	0.0825 (14)	0.0724 (14)	−0.0247 (11)	−0.0200 (11)	0.0067 (11)
C10	0.0816 (16)	0.0786 (14)	0.0575 (12)	−0.0199 (11)	−0.0137 (11)	−0.0014 (10)
C11	0.0708 (13)	0.0655 (11)	0.0521 (10)	−0.0082 (10)	0.0009 (9)	−0.0061 (8)
C12	0.0501 (10)	0.0467 (9)	0.0507 (9)	−0.0036 (7)	−0.0012 (8)	−0.0047 (7)

Geometric parameters (Å, º)

O1—C2	1.213 (2)	C5—H5B	0.9600
N1—C2	1.365 (2)	C5—H5C	0.9600
N1—C1	1.373 (2)	C6—H6A	0.9600
N1—H1	0.864 (9)	C6—H6B	0.9600
N2—C4	1.279 (2)	C6—H6C	0.9600
N2—N3	1.393 (2)	C7—C8	1.383 (2)
N3—C1	1.371 (2)	C7—C12	1.385 (3)
N3—C7	1.394 (2)	C8—C9	1.384 (3)
N4—C1	1.309 (2)	C8—H8	0.9300
N4—C12	1.396 (2)	C9—C10	1.386 (3)
C2—C3	1.523 (3)	C9—H9	0.9300
C3—C5	1.510 (3)	C10—C11	1.382 (3)
C3—C4	1.515 (2)	C10—H10	0.9300
C3—H3	0.9800	C11—C12	1.393 (2)
C4—C6	1.495 (3)	C11—H11	0.9300
C5—H5A	0.9600		

C2—N1—C1	126.41 (17)	H5A—C5—H5C	109.5
C2—N1—H1	118.6 (14)	H5B—C5—H5C	109.5
C1—N1—H1	114.7 (14)	C4—C6—H6A	109.5
C4—N2—N3	116.74 (14)	C4—C6—H6B	109.5
C1—N3—N2	130.27 (13)	H6A—C6—H6B	109.5
C1—N3—C7	105.84 (14)	C4—C6—H6C	109.5
N2—N3—C7	121.30 (14)	H6A—C6—H6C	109.5
C1—N4—C12	104.53 (15)	H6B—C6—H6C	109.5
N4—C1—N3	113.68 (14)	C8—C7—C12	123.37 (17)
N4—C1—N1	123.18 (16)	C8—C7—N3	131.12 (19)
N3—C1—N1	122.98 (16)	C12—C7—N3	105.50 (14)
O1—C2—N1	120.60 (19)	C7—C8—C9	115.6 (2)

O1—C2—C3	123.76 (18)	C7—C8—H8	122.2
N1—C2—C3	115.63 (14)	C9—C8—H8	122.2
C5—C3—C4	115.20 (18)	C8—C9—C10	121.89 (19)
C5—C3—C2	111.16 (15)	C8—C9—H9	119.1
C4—C3—C2	108.05 (14)	C10—C9—H9	119.1
C5—C3—H3	107.4	C11—C10—C9	122.06 (19)
C4—C3—H3	107.4	C11—C10—H10	119.0
C2—C3—H3	107.4	C9—C10—H10	119.0
N2—C4—C6	116.53 (16)	C10—C11—C12	116.7 (2)
N2—C4—C3	124.01 (16)	C10—C11—H11	121.7
C6—C4—C3	119.45 (16)	C12—C11—H11	121.7
C3—C5—H5A	109.5	C7—C12—C11	120.37 (17)
C3—C5—H5B	109.5	C7—C12—N4	110.36 (15)
H5A—C5—H5B	109.5	C11—C12—N4	129.27 (19)
C3—C5—H5C	109.5		

C4—N2—N3—C1	45.3 (2)	C5—C3—C4—C6	−14.2 (2)
C4—N2—N3—C7	−155.63 (15)	C2—C3—C4—C6	110.73 (19)
C12—N4—C1—N3	−1.68 (19)	C1—N3—C7—C8	176.13 (18)
C12—N4—C1—N1	173.69 (17)	N2—N3—C7—C8	12.6 (3)
N2—N3—C1—N4	164.44 (16)	C1—N3—C7—C12	−2.84 (17)
C7—N3—C1—N4	2.94 (19)	N2—N3—C7—C12	−166.39 (14)
N2—N3—C1—N1	−10.9 (3)	C12—C7—C8—C9	−1.1 (3)
C7—N3—C1—N1	−172.44 (16)	N3—C7—C8—C9	−179.90 (18)
C2—N1—C1—N4	152.30 (18)	C7—C8—C9—C10	−0.6 (3)
C2—N1—C1—N3	−32.8 (3)	C8—C9—C10—C11	1.2 (4)
C1—N1—C2—O1	177.59 (18)	C9—C10—C11—C12	0.0 (3)
C1—N1—C2—C3	−3.3 (3)	C8—C7—C12—C11	2.3 (3)
O1—C2—C3—C5	13.2 (3)	N3—C7—C12—C11	−178.66 (16)
N1—C2—C3—C5	−165.91 (17)	C8—C7—C12—N4	−177.09 (16)
O1—C2—C3—C4	−114.2 (2)	N3—C7—C12—N4	1.99 (18)
N1—C2—C3—C4	66.8 (2)	C10—C11—C12—C7	−1.6 (3)
N3—N2—C4—C6	−177.91 (15)	C10—C11—C12—N4	177.58 (18)
N3—N2—C4—C3	3.5 (2)	C1—N4—C12—C7	−0.27 (19)
C5—C3—C4—N2	164.30 (17)	C1—N4—C12—C11	−179.55 (19)
C2—C3—C4—N2	−70.7 (2)		

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1—H1···N4^{i}	0.86 (1)	2.01 (1)	2.867 (2)	174 (2)

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