Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

3-Benzoyl-4-methyl-4,5,6,7-tetrahydro-1,2,3-triazolo[1,5-*a*]pyrimidine

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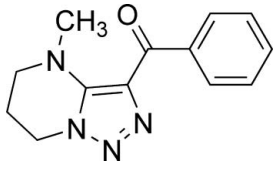
Received 19 May 2007; accepted 28 May 2007

Key indicators: single-crystal X-ray study; T = 294 K; mean σ(C–C) = 0.003 Å; R factor = 0.041; wR factor = 0.116; data-to-parameter ratio = 15.6.

In the title compound, $C_{13}H_{14}N_4O$, the dihedral angle between the mean planes of the C_2N_3 heterocyclic ring and the phenyl ring is 49.51 (9)°.

Related literature

For background, see: Huang & Wang (1992, 1994).



Experimental

Crystal data

$C_{13}H_{14}N_4O$
M_r = 242.28
Orthorhombic, *Pbca*
a = 14.644 (5) Å
b = 7.597 (3) Å
c = 22.711 (8) Å
V = 2526.7 (16) Å^3
Z = 8
Mo $K\alpha$ radiation
μ = 0.09 mm^{-1}
T = 294 (2) K
0.22 × 0.20 × 0.10 mm

Data collection

Bruker SMART CCD diffractometer
Absorption correction: multi-scan (*SADABS*; Bruker, 1997)
T_{min} = 0.982, T_{max} = 0.992
13257 measured reflections
2576 independent reflections
1602 reflections with $I > 2\sigma(I)$
R_{int} = 0.057

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.041
$wR(F^2)$ = 0.116
S = 1.01
2576 reflections
165 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.15 e Å^{-3}
$\Delta\rho_{min}$ = −0.17 e Å^{-3}

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

The authors thank Mr Wang Hong Geng of Nankai University for the X-ray crystallographic determination.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: HB2429).

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  doi:10.1107/S1600536807025998

supplementary materials

Acta Cryst. (2007). E**63**, o3078 [**doi:10.1107/S1600536807025998**]

3-Benzoyl-4-methyl-4,5,6,7-tetrahydro-1,2,3-triazolo[1,5-*a*]pyrimidine

C.-Y. Yu, X.-N. Yuan and Z.-T. Huang

Comment

Heterocyclic ketene aminals, also named cyclic 1,1-enediamines, are useful synthons in organic synthesis, especially for the synthesis of heterocycles (Huang & Wang, 1994). The title compound, (I) (Fig. 1), belongs to the family of 1, 2,3-triazole fused 1,3-diazoheterocles. These compounds are potentially useful in developing new pharmaceuticals and agrochemicals. The crystal structure was expected to provide unambiguous evidence for the determination of the chemical structure of the title compound.

In the structure of (I), the six-membered –N1—C2—C3—C4—N2—C5- ring takes on an envelope conformation with C3 in the flap position. The dihedral angle between the mean planes of the C_2N_3-heterocyclic ring and the pendant benzene ring is 49.51 (9)°.

Experimental

The title compound (I) was prepared according to the procedure reported by Huang & Wang (1992) and purified by recrystallization from ethyl acetate to obtain (I) as very pale green crystals in 75.6% yield; mp 376–377 K; FT—IR (KBr): 3396.03, 1640.16, 1583.27, 1473.35 cm^{-1}. ^1H NMR ($CDCl_3$, δp.p.m.): 8.00 (d, 2H, aryl H), 7.45–7.36 (m, 3H, aryl H), 4.24–4.20 (t, 2H, CH_2), 3.25–3.23 (t, 2H, CH_2), 3.16 (s, 3H, N—CH_3), 2.13–2.11 (m, 2H, CH_2); ^{13}C NMR ($CDCl_3$, δp.p.m.): 186.52, 145.92, 138.72, 132.11, 130.42, 129.31, 127.99, 49.24, 43.58, 40.53, 20.90; MS (EI) m/*z* (relative intensity, %): 243(16, $[M+H]^+$), 242(100, $[M]^+$), 213 (82), 186 (28), 185 (44). Anal. Calcd for $C_{13}H_{14}N_4O$: C, 64.45; H, 5.82; N, 23.13. Found: C, 64.33; H, 5.78; N, 23.23.

Refinement

All H atoms were placed in geometrically idealized positions (C—H = 0.95–1.00 Å) and refined as riding with U_{iso}(H) = 1.2U_{eq}(carrier) or U_{iso}(H) = 1.5U_{eq}(methyl carrier).

Figures

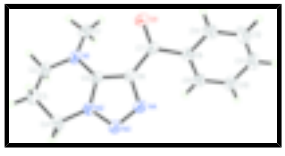

Fig. 1. Molecular structure of (I) showing 30% displacement ellipsoids (arbitrary spheres for the H atoms).

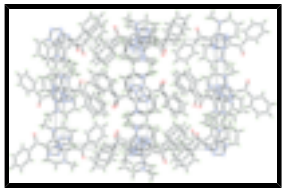

Fig. 2. Packing diagram of (I) viewed down the *b*-axis.

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Crystal data

$C_{13}H_{14}N_4O$	$D_x = 1.274$ Mg m^{-3}
$M_r = 242.28$	Melting point: 376-377 K
Orthorhombic, *Pbca*	Mo *K*α radiation λ = 0.71073 Å
$a = 14.644$ (5) Å	Cell parameters from 2603 reflections
$b = 7.597$ (3) Å	θ = 2.8–21.8º
$c = 22.711$ (8) Å	μ = 0.09 mm^{-1}
$V = 2526.7$ (16) Å^3	$T = 294$ (2) K
$Z = 8$	Prism, very pale green
$F_{000} = 1024$	0.22 × 0.20 × 0.10 mm

Data collection

Bruker SMART CCD diffractometer	2576 independent reflections
Radiation source: fine-focus sealed tube	1602 reflections with $I > 2\sigma(I)$
Monochromator: graphite	$R_{int} = 0.057$
$T = 294$(2) K	$\theta_{max} = 26.4$º
ω scans	$\theta_{min} = 1.8$º
Absorption correction: multi-scan (SADABS; Bruker, 1997)	$h = -17\rightarrow18$
$T_{min} = 0.982$, $T_{max} = 0.992$	$k = -8\rightarrow9$
13257 measured reflections	$l = -28\rightarrow13$

Refinement

Refinement on F^2	Hydrogen site location: inferred from neighbouring sites
Least-squares matrix: full	H-atom parameters constrained
$R[F^2 > 2\sigma(F^2)] = 0.041$	$w = 1/[\sigma^2(F_o^2) + (0.0529P)^2 + 0.2472P]$ where $P = (F_o^2 + 2F_c^2)/3$
$wR(F^2) = 0.116$	$(\Delta/\sigma)_{max} = 0.004$
$S = 1.01$	$\Delta\rho_{max} = 0.15$ e Å^{-3}
2576 reflections	$\Delta\rho_{min} = -0.17$ e Å^{-3}
165 parameters	Extinction correction: SHELXL97 (Sheldrick, 1997), $Fc^* = kFc[1+0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$

Primary atom site location: structure-invariant direct methods
Secondary atom site location: difference Fourier map
Extinction coefficient: 0.0109 (11)

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 $F^2 > 2\text{sigma}(F^2)$ 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	0.38429 (8)	0.38641 (18)	0.35194 (6)	0.0657 (4)
N1	0.33793 (9)	0.18589 (19)	0.47987 (6)	0.0504 (4)
N2	0.48321 (9)	0.23988 (18)	0.52259 (6)	0.0445 (4)
N3	0.56950 (10)	0.2989 (2)	0.50826 (7)	0.0521 (4)
N4	0.56915 (9)	0.33018 (19)	0.45181 (6)	0.0484 (4)
C1	0.29124 (12)	0.1074 (3)	0.42903 (9)	0.0663 (6)
H1A	0.2966	0.1845	0.3957	0.099*
H1B	0.2279	0.0903	0.4383	0.099*
H1C	0.3187	−0.0041	0.4199	0.099*
C2	0.30857 (12)	0.1198 (3)	0.53793 (9)	0.0605 (6)
H2A	0.3229	−0.0045	0.5408	0.073*
H2B	0.2429	0.1327	0.5415	0.073*
C3	0.35475 (14)	0.2177 (3)	0.58799 (8)	0.0635 (6)
H3A	0.3341	0.1704	0.6254	0.076*
H3B	0.3381	0.3412	0.5865	0.076*
C4	0.45787 (13)	0.1995 (2)	0.58340 (8)	0.0559 (5)
H4A	0.4877	0.2805	0.6102	0.067*
H4B	0.4764	0.0806	0.5934	0.067*
C5	0.42705 (11)	0.2381 (2)	0.47487 (7)	0.0396 (4)
C6	0.48354 (11)	0.2968 (2)	0.42818 (7)	0.0402 (4)
C7	0.46293 (11)	0.3477 (2)	0.36725 (7)	0.0447 (4)
C8	0.53960 (11)	0.3549 (2)	0.32287 (7)	0.0437 (4)
C9	0.61735 (12)	0.2493 (2)	0.32667 (8)	0.0519 (5)
H9	0.6254	0.1766	0.3592	0.062*
C10	0.68318 (13)	0.2516 (3)	0.28232 (8)	0.0629 (6)
H10	0.7344	0.1798	0.2852	0.075*
C11	0.67223 (15)	0.3606 (3)	0.23396 (9)	0.0721 (6)
H11	0.7162	0.3629	0.2044	0.087*
C12	0.59586 (17)	0.4655 (3)	0.23000 (9)	0.0830 (7)
H12	0.5886	0.5393	0.1977	0.100*

C13	0.52960 (14)	0.4623 (3)	0.27367 (8)	0.0667 (6)
H13	0.4780	0.5327	0.2700	0.080*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.0450 (8)	0.0844 (10)	0.0677 (9)	0.0147 (7)	−0.0028 (6)	0.0087 (7)
N1	0.0361 (8)	0.0526 (9)	0.0624 (10)	−0.0028 (7)	0.0089 (7)	0.0014 (8)
N2	0.0411 (8)	0.0449 (8)	0.0475 (9)	0.0026 (6)	0.0055 (7)	0.0017 (6)
N3	0.0411 (9)	0.0620 (10)	0.0532 (10)	−0.0024 (7)	0.0012 (7)	−0.0023 (7)
N4	0.0390 (9)	0.0558 (9)	0.0504 (9)	−0.0043 (7)	0.0021 (7)	−0.0024 (7)
C1	0.0427 (11)	0.0700 (13)	0.0861 (15)	−0.0115 (10)	−0.0008 (10)	−0.0117 (12)
C2	0.0456 (11)	0.0546 (12)	0.0811 (15)	0.0029 (9)	0.0196 (10)	0.0172 (11)
C3	0.0694 (13)	0.0589 (12)	0.0621 (13)	0.0159 (10)	0.0259 (11)	0.0127 (10)
C4	0.0680 (13)	0.0503 (11)	0.0496 (11)	0.0059 (9)	0.0084 (9)	0.0045 (9)
C5	0.0351 (9)	0.0332 (9)	0.0506 (10)	0.0041 (7)	0.0025 (8)	−0.0007 (7)
C6	0.0342 (9)	0.0389 (9)	0.0475 (10)	0.0001 (7)	0.0031 (8)	−0.0016 (7)
C7	0.0420 (10)	0.0385 (9)	0.0536 (11)	0.0028 (8)	0.0004 (8)	−0.0013 (8)
C8	0.0444 (10)	0.0433 (9)	0.0435 (10)	−0.0003 (8)	0.0011 (8)	−0.0019 (8)
C9	0.0506 (11)	0.0543 (11)	0.0510 (11)	0.0040 (9)	0.0035 (9)	0.0022 (8)
C10	0.0514 (12)	0.0749 (14)	0.0624 (13)	0.0048 (10)	0.0088 (10)	−0.0115 (11)
C11	0.0734 (15)	0.0853 (16)	0.0576 (14)	−0.0081 (12)	0.0215 (11)	−0.0015 (12)
C12	0.0946 (18)	0.0908 (17)	0.0637 (14)	0.0118 (14)	0.0208 (13)	0.0282 (12)
C13	0.0709 (14)	0.0689 (13)	0.0603 (12)	0.0157 (11)	0.0070 (10)	0.0172 (11)

Geometric parameters (Å, °)

O1—C7	1.2385 (19)	C3—H3B	0.9700
N1—C5	1.369 (2)	C4—H4A	0.9700
N1—C1	1.469 (2)	C4—H4B	0.9700
N1—C2	1.475 (2)	C5—C6	1.417 (2)
N2—C5	1.360 (2)	C6—C7	1.468 (2)
N2—N3	1.3798 (19)	C7—C8	1.510 (2)
N2—C4	1.463 (2)	C8—C13	1.391 (2)
N3—N4	1.3039 (19)	C8—C9	1.395 (2)
N4—C6	1.387 (2)	C9—C10	1.394 (2)
C1—H1A	0.9600	C9—H9	0.9300
C1—H1B	0.9600	C10—C11	1.385 (3)
C1—H1C	0.9600	C10—H10	0.9300
C2—C3	1.518 (3)	C11—C12	1.376 (3)
C2—H2A	0.9700	C11—H11	0.9300
C2—H2B	0.9700	C12—C13	1.388 (3)
C3—C4	1.520 (3)	C12—H12	0.9300
C3—H3A	0.9700	C13—H13	0.9300
C5—N1—C1	119.77 (14)	C3—C4—H4B	110.2
C5—N1—C2	116.80 (15)	H4A—C4—H4B	108.5
C1—N1—C2	115.39 (15)	N2—C5—N1	120.87 (15)
C5—N2—N3	111.66 (13)	N2—C5—C6	103.89 (14)

C5—N2—C4	126.63 (14)	N1—C5—C6	135.23 (16)
N3—N2—C4	121.55 (14)	N4—C6—C5	107.20 (14)
N4—N3—N2	106.70 (13)	N4—C6—C7	120.15 (14)
N3—N4—C6	110.53 (13)	C5—C6—C7	131.93 (15)
N1—C1—H1A	109.5	O1—C7—C6	121.22 (15)
N1—C1—H1B	109.5	O1—C7—C8	119.70 (15)
H1A—C1—H1B	109.5	C6—C7—C8	119.07 (14)
N1—C1—H1C	109.5	C13—C8—C9	118.21 (16)
H1A—C1—H1C	109.5	C13—C8—C7	118.63 (16)
H1B—C1—H1C	109.5	C9—C8—C7	123.00 (15)
N1—C2—C3	111.89 (15)	C8—C9—C10	120.83 (17)
N1—C2—H2A	109.2	C8—C9—H9	119.6
C3—C2—H2A	109.2	C10—C9—H9	119.6
N1—C2—H2B	109.2	C11—C10—C9	120.00 (19)
C3—C2—H2B	109.2	C11—C10—H10	120.0
H2A—C2—H2B	107.9	C9—C10—H10	120.0
C2—C3—C4	110.29 (15)	C12—C11—C10	119.49 (18)
C2—C3—H3A	109.6	C12—C11—H11	120.3
C4—C3—H3A	109.6	C10—C11—H11	120.3
C2—C3—H3B	109.6	C11—C12—C13	120.76 (19)
C4—C3—H3B	109.6	C11—C12—H12	119.6
H3A—C3—H3B	108.1	C13—C12—H12	119.6
N2—C4—C3	107.33 (15)	C12—C13—C8	120.69 (19)
N2—C4—H4A	110.2	C12—C13—H13	119.7
C3—C4—H4A	110.2	C8—C13—H13	119.7
N2—C4—H4B	110.2		
C5—N2—N3—N4	−1.57 (18)	N1—C5—C6—N4	−179.97 (17)
C4—N2—N3—N4	−177.20 (14)	N2—C5—C6—C7	−170.36 (16)
N2—N3—N4—C6	1.27 (18)	N1—C5—C6—C7	10.1 (3)
C5—N1—C2—C3	35.7 (2)	N4—C6—C7—O1	−150.75 (16)
C1—N1—C2—C3	−175.54 (15)	C5—C6—C7—O1	18.2 (3)
N1—C2—C3—C4	−58.9 (2)	N4—C6—C7—C8	28.4 (2)
C5—N2—C4—C3	−19.4 (2)	C5—C6—C7—C8	−162.64 (16)
N3—N2—C4—C3	155.57 (15)	O1—C7—C8—C13	23.3 (2)
C2—C3—C4—N2	48.38 (19)	C6—C7—C8—C13	−155.89 (16)
N3—N2—C5—N1	−179.16 (14)	O1—C7—C8—C9	−151.94 (17)
C4—N2—C5—N1	−3.8 (2)	C6—C7—C8—C9	28.8 (2)
N3—N2—C5—C6	1.18 (17)	C13—C8—C9—C10	0.0 (3)
C4—N2—C5—C6	176.55 (14)	C7—C8—C9—C10	175.28 (16)
C1—N1—C5—N2	−151.85 (16)	C8—C9—C10—C11	0.6 (3)
C2—N1—C5—N2	−4.5 (2)	C9—C10—C11—C12	−0.4 (3)
C1—N1—C5—C6	27.7 (3)	C10—C11—C12—C13	−0.4 (3)
C2—N1—C5—C6	175.05 (17)	C11—C12—C13—C8	0.9 (3)
N3—N4—C6—C5	−0.57 (18)	C9—C8—C13—C12	−0.7 (3)
N3—N4—C6—C7	170.82 (14)	C7—C8—C13—C12	−176.24 (19)
N2—C5—C6—N4	−0.39 (17)		

Fig. 1

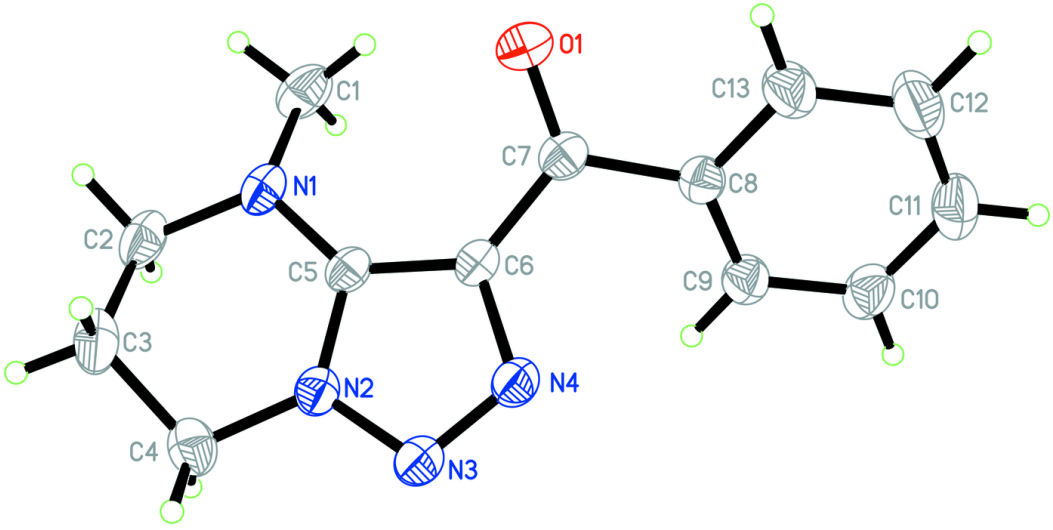

Fig. 2