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N-[(*E*)-1,3-Benzodioxol-5-ylmethylidene]-3,4-dimethyl-1,2-oxazol-5-amine

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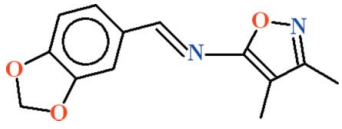
Key indicators: single-crystal X-ray study; T = 296 K; mean σ(C–C) = 0.003 Å; R factor = 0.045; wR factor = 0.132; data-to-parameter ratio = 12.6.

In the title compound, $C_{13}H_{12}N_2O_3$, the dihedral angle between the aromatic rings is 7.94 (12)°. In the crystal, inversion dimers linked by pairs of C—H···O hydrogen bonds generate $R_2^2(6)$ loops. Weak π–π [centroid–centroid separations = 3.7480 (13) and 3.9047 (13) Å] and C—H···π interactions help to consolidate the packing.

Related literature

For background to conjugated azo-methanes, see: Asiri & Khan (2010). For related structures, see: Asiri *et al.* (2010); Tahir *et al.* (2010). For graph-set notation, see: Bernstein *et al.* (1995).



Experimental

Crystal data

$C_{13}H_{12}N_2O_3$
M_r = 244.25
Monoclinic, $P2_1/n$
a = 7.5759 (5) Å
b = 10.6980 (9) Å
c = 14.6307 (12) Å
β = 102.607 (2)°
V = 1157.19 (16) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.10 mm^{-1}
T = 296 K
0.28 × 0.24 × 0.22 mm

Data collection

Bruker Kappa APEXII CCD diffractometer
Absorption correction: multi-scan (*SADABS*; Bruker, 2005)
T_{min} = 0.975, T_{max} = 0.980
8018 measured reflections
2087 independent reflections
1447 reflections with $I > 2\sigma(I)$
R_{int} = 0.032

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.045
$wR(F^2)$ = 0.132
S = 1.03
2087 reflections
165 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.17 e Å^{-3}
$\Delta\rho_{min}$ = −0.18 e Å^{-3}

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

*Cg*1 is the centroid of the C1–C6 benzene ring.

D—H···A	D—H	H···A	D···A	D—H···A
C7—H7*B*···O1^{i}	0.97	2.58	3.264 (3)	128
C12—H12*A*···*Cg*1^{ii}	0.96	2.95	3.763 (2)	143

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

Data collection: *APEX2* (Bruker, 2009); cell refinement: *SAINT* (Bruker, 2009); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *ORTEP-3 for Windows* (Farrugia, 1997) and *PLATON* (Spek, 2009); software used to prepare material for publication: *WinGX* (Farrugia, 1999) and *PLATON*.

The authors would like to thank the Chemistry Department, King Abdulaziz University, Jeddah, Saudi Arabia, for providing the research facilities.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: HB6330).

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Acta Cryst. (2011). E**67**, o2305 [doi:10.1107/S1600536811030327]

N-[(*E*)-1,3-Benzodioxol-5-ylmethylidene]-3,4-dimethyl-1,2-oxazol-5-amine

Abdullah M. Asiri, Salman A. Khan and M. Nawaz Tahir

S1. Comment

Donor-acceptor conjugated azo-methanes are used as substrates in the preparation of a large number of bioactive and industrial compounds *via* ring closure, cycloaddition, replacement reactions, *etc* (Asiri & Khan, 2010). The title compound (I, Fig. 1) has been prepared as a pharmaceutical intermediate.

The crystal structures of *N*-(4-chlorobenzylidene)-3,4-dimethylisoxazol -5-amine (Asiri *et al.*, 2010) and *N*-[(*E*)-1,3-benzodioxol -5-ylmethylidene]-4-methylaniline (Tahir *et al.*, 2010) have been published, which are related to (I).

In (I), the group A (C1–C8/O1/O2) of 1,3-benzodioxole-5-carbaldehyde moiety and the group B (C9—C12/N1/N2/O3) of 5-amino-3,4-dimethylisoxazole moiety are almost planar with r.m.s. deviations of 0.022 and 0.019 Å, respectively. The dihedral angle between A/B is 8.29 (3)°. The inter-molecular H-bondings of C—H···O type dimerize the molecules with $R_2^2(6)$ ring motif (Bernstein *et al.*, 1995) (Table 1, Fig. 2). In stabilizing the molecules π–π interactions [separation: 3.7480 (13), 3.9047 (13) Å] and C—H···π interaction (Table 1) play important role in stabilizing the molecules.

S2. Experimental

A mixture of 1,3-benzodioxole-5-carbaldehyde (0.50 g, 3.3 mmol) and 5-amino-3,4-dimethylisoxazole (3.3 mmol) in ethanol (15 ml) was heated for 3 h. The progress of the reaction was monitored by TLC. The solid that separated from the cooled mixture was collected and recrystallized from a methanol-chloroform mixture (8:2) to give the yellow prisms of the title compound (I).

Yield: 74%; m.p. 504–505 K.

S3. Refinement

The H-atoms were positioned geometrically (C–H = 0.93–0.97 Å) and refined as riding with U_{iso}(H) = xU_{eq}(C), where x = 1.5 for methyl and x = 1.2 for aryl H-atoms.

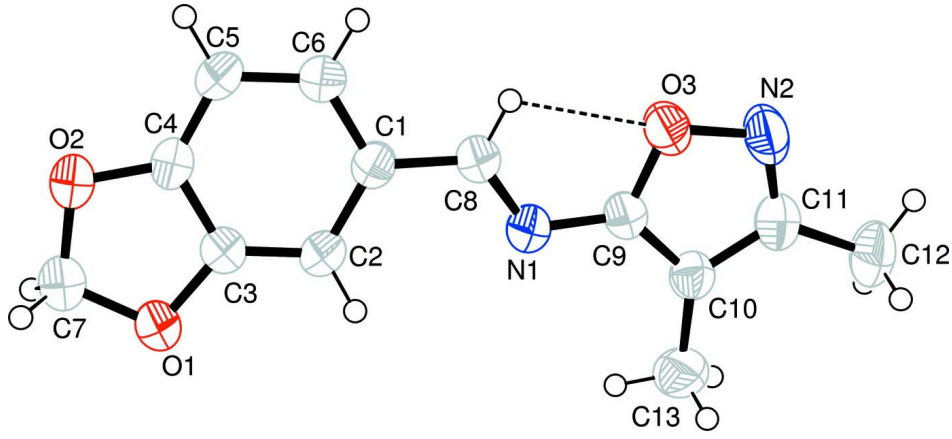

Figure 1

View of (I) with displacement ellipsoids drawn at the 50% probability level.

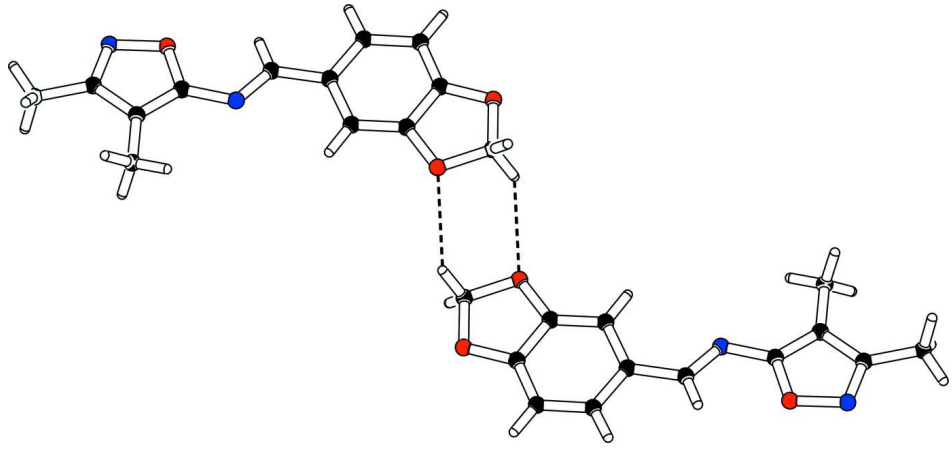

Figure 2

The partial packing for (I), which shows that molecules form dimers with $R_2^2(6)$ ring motif.

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$c = 14.6307\ (12)$ Å
$\beta = 102.607\ (2)°$
$V = 1157.19\ (16)$ Å^3
$Z = 4$
$F(000) = 512$
$D_x = 1.402$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 1447 reflections
$\theta = 3.4–25.3°$
$\mu = 0.10$ mm^{-1}
$T = 296$ K
Prism, yellow
0.28 × 0.24 × 0.22 mm

Data collection

Bruker Kappa APEXII CCD diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
Detector resolution: 8.10 pixels mm^{-1}
ω scans
Absorption correction: multi-scan (*SADABS*; Bruker, 2005)

T_{min} = 0.975, T_{max} = 0.980
8018 measured reflections
2087 independent reflections
1447 reflections with $I > 2\sigma(I)$
R_{int} = 0.032

θ_{max} = 25.3°, θ_{min} = 3.4°
h = −9→9
k = −12→12
l = −17→17

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.045
$wR(F^2)$ = 0.132
S = 1.03
2087 reflections
165 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
$w = 1/[\sigma^2(F_o^2) + (0.0693P)^2 + 0.1704P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.17 e Å^{-3}
$\Delta\rho_{min}$ = −0.18 e Å^{-3}

Special details

Geometry. Bond distances, angles *etc*. have been calculated using the rounded fractional coordinates. All su's are estimated from the variances of the (full) variance-covariance matrix. The cell e.s.d.'s are taken into account in the estimation of distances, angles and torsion angles

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}
O1	0.6073 (2)	0.09898 (13)	0.08565 (10)	0.0729 (6)
O2	0.6345 (2)	0.27941 (14)	0.00426 (10)	0.0702 (6)
O3	0.6207 (2)	0.38037 (12)	0.55753 (10)	0.0699 (6)
N1	0.6338 (2)	0.23824 (15)	0.43533 (11)	0.0487 (5)
N2	0.6192 (3)	0.37712 (18)	0.65383 (13)	0.0742 (8)
C1	0.6183 (2)	0.31798 (17)	0.28025 (13)	0.0457 (6)
C2	0.6088 (3)	0.20099 (17)	0.23618 (13)	0.0493 (6)
C3	0.6150 (3)	0.19982 (17)	0.14411 (14)	0.0490 (7)
C4	0.6313 (3)	0.30805 (19)	0.09481 (13)	0.0510 (7)
C5	0.6386 (3)	0.42247 (19)	0.13520 (15)	0.0618 (8)
C6	0.6302 (3)	0.42546 (18)	0.22912 (14)	0.0572 (7)
C7	0.6271 (3)	0.1475 (2)	−0.00210 (15)	0.0687 (8)
C8	0.6190 (2)	0.33074 (18)	0.37881 (13)	0.0497 (7)
C9	0.6331 (3)	0.26031 (18)	0.52782 (13)	0.0478 (7)
C10	0.6405 (2)	0.18039 (18)	0.60027 (13)	0.0477 (7)
C11	0.6292 (3)	0.2593 (2)	0.67649 (14)	0.0556 (7)
C12	0.6270 (3)	0.2203 (2)	0.77363 (15)	0.0716 (9)
C13	0.6585 (3)	0.0425 (2)	0.60257 (15)	0.0697 (9)
H2	0.59873	0.12747	0.26857	0.0591*
H5	0.64865	0.49515	0.10183	0.0741*
H6	0.63264	0.50254	0.25877	0.0686*

H7A	0.73721	0.11561	−0.01717	0.0824*
H7B	0.52555	0.12203	−0.05118	0.0824*
H8	0.60811	0.41048	0.40229	0.0596*
H12A	0.51285	0.18213	0.77471	0.1074*
H12B	0.64420	0.29227	0.81383	0.1074*
H12C	0.72268	0.16145	0.79518	0.1074*
H13A	0.69485	0.01447	0.54709	0.1045*
H13B	0.54443	0.00536	0.60531	0.1045*
H13C	0.74801	0.01816	0.65679	0.1045*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.1287 (13)	0.0486 (9)	0.0459 (9)	−0.0125 (8)	0.0292 (9)	−0.0072 (7)
O2	0.1149 (12)	0.0587 (10)	0.0411 (8)	−0.0105 (8)	0.0259 (8)	−0.0007 (7)
O3	0.1182 (12)	0.0479 (9)	0.0477 (9)	0.0126 (8)	0.0273 (8)	−0.0003 (7)
N1	0.0606 (10)	0.0462 (9)	0.0407 (9)	−0.0010 (7)	0.0143 (7)	0.0013 (7)
N2	0.1179 (16)	0.0650 (13)	0.0447 (11)	0.0151 (10)	0.0288 (10)	−0.0032 (9)
C1	0.0549 (11)	0.0427 (10)	0.0424 (10)	0.0009 (8)	0.0170 (8)	0.0031 (8)
C2	0.0648 (12)	0.0409 (10)	0.0445 (11)	−0.0023 (9)	0.0172 (9)	0.0050 (9)
C3	0.0631 (12)	0.0423 (11)	0.0432 (11)	−0.0035 (9)	0.0148 (9)	−0.0020 (9)
C4	0.0652 (12)	0.0526 (12)	0.0373 (10)	−0.0033 (9)	0.0157 (9)	0.0023 (9)
C5	0.0975 (16)	0.0443 (12)	0.0495 (12)	−0.0033 (10)	0.0291 (11)	0.0083 (9)
C6	0.0860 (15)	0.0407 (11)	0.0497 (12)	−0.0020 (10)	0.0253 (10)	−0.0009 (9)
C7	0.1030 (17)	0.0597 (14)	0.0456 (12)	−0.0082 (12)	0.0210 (11)	−0.0036 (10)
C8	0.0656 (13)	0.0431 (10)	0.0434 (11)	−0.0004 (9)	0.0186 (9)	−0.0022 (9)
C9	0.0591 (12)	0.0446 (11)	0.0410 (11)	0.0003 (8)	0.0140 (9)	−0.0023 (9)
C10	0.0517 (11)	0.0505 (12)	0.0404 (11)	−0.0020 (8)	0.0091 (8)	0.0009 (9)
C11	0.0582 (12)	0.0666 (14)	0.0426 (12)	0.0025 (10)	0.0122 (9)	0.0005 (10)
C12	0.0825 (16)	0.0934 (18)	0.0410 (12)	−0.0012 (13)	0.0179 (11)	0.0039 (12)
C13	0.0987 (17)	0.0543 (13)	0.0523 (14)	−0.0088 (11)	0.0083 (12)	0.0070 (10)

Geometric parameters (Å, º)

O1—C3	1.370 (2)	C9—C10	1.353 (3)
O1—C7	1.423 (3)	C10—C11	1.416 (3)
O2—C4	1.365 (2)	C10—C13	1.481 (3)
O2—C7	1.415 (3)	C11—C12	1.485 (3)
O3—N2	1.412 (2)	C2—H2	0.9300
O3—C9	1.366 (2)	C5—H5	0.9300
N1—C8	1.279 (2)	C6—H6	0.9300
N1—C9	1.375 (2)	C7—H7A	0.9700
N2—C11	1.301 (3)	C7—H7B	0.9700
C1—C2	1.403 (3)	C8—H8	0.9300
C1—C6	1.385 (3)	C12—H12A	0.9600
C1—C8	1.447 (3)	C12—H12B	0.9600
C2—C3	1.358 (3)	C12—H12C	0.9600
C3—C4	1.384 (3)	C13—H13A	0.9600

C4—C5	1.355 (3)	C13—H13B	0.9600
C5—C6	1.390 (3)	C13—H13C	0.9600

C3—O1—C7	106.12 (15)	C10—C11—C12	126.98 (19)
C4—O2—C7	106.20 (16)	C1—C2—H2	121.00
N2—O3—C9	108.09 (14)	C3—C2—H2	121.00
C8—N1—C9	119.01 (17)	C4—C5—H5	122.00
O3—N2—C11	105.39 (17)	C6—C5—H5	122.00
C2—C1—C6	119.69 (17)	C1—C6—H6	119.00
C2—C1—C8	122.06 (17)	C5—C6—H6	119.00
C6—C1—C8	118.24 (17)	O1—C7—H7A	110.00
C1—C2—C3	117.05 (17)	O1—C7—H7B	110.00
O1—C3—C2	128.35 (17)	O2—C7—H7A	110.00
O1—C3—C4	109.26 (17)	O2—C7—H7B	110.00
C2—C3—C4	122.39 (18)	H7A—C7—H7B	108.00
O2—C4—C3	109.91 (17)	N1—C8—H8	118.00
O2—C4—C5	128.19 (19)	C1—C8—H8	118.00
C3—C4—C5	121.89 (18)	C11—C12—H12A	109.00
C4—C5—C6	116.46 (19)	C11—C12—H12B	109.00
C1—C6—C5	122.49 (18)	C11—C12—H12C	109.00
O1—C7—O2	108.37 (16)	H12A—C12—H12B	109.00
N1—C8—C1	123.51 (17)	H12A—C12—H12C	110.00
O3—C9—N1	119.35 (16)	H12B—C12—H12C	109.00
O3—C9—C10	109.80 (16)	C10—C13—H13A	109.00
N1—C9—C10	130.84 (18)	C10—C13—H13B	110.00
C9—C10—C11	103.94 (17)	C10—C13—H13C	109.00
C9—C10—C13	129.41 (18)	H13A—C13—H13B	109.00
C11—C10—C13	126.65 (18)	H13A—C13—H13C	109.00
N2—C11—C10	112.77 (18)	H13B—C13—H13C	109.00
N2—C11—C12	120.26 (19)		

C7—O1—C3—C2	−177.5 (2)	C6—C1—C8—N1	−170.00 (18)
C7—O1—C3—C4	2.4 (2)	C1—C2—C3—O1	−179.6 (2)
C3—O1—C7—O2	−3.8 (2)	C1—C2—C3—C4	0.5 (3)
C7—O2—C4—C3	−2.4 (2)	O1—C3—C4—O2	0.0 (3)
C7—O2—C4—C5	178.9 (2)	O1—C3—C4—C5	178.8 (2)
C4—O2—C7—O1	3.8 (2)	C2—C3—C4—O2	179.9 (2)
C9—O3—N2—C11	−0.4 (2)	C2—C3—C4—C5	−1.3 (4)
N2—O3—C9—N1	178.87 (19)	O2—C4—C5—C6	179.1 (2)
N2—O3—C9—C10	−0.3 (2)	C3—C4—C5—C6	0.5 (3)
C9—N1—C8—C1	179.97 (18)	C4—C5—C6—C1	1.1 (3)
C8—N1—C9—O3	−1.4 (3)	O3—C9—C10—C11	0.8 (2)
C8—N1—C9—C10	177.5 (2)	O3—C9—C10—C13	−178.49 (18)
O3—N2—C11—C10	0.9 (3)	N1—C9—C10—C11	−178.2 (2)
O3—N2—C11—C12	−178.74 (19)	N1—C9—C10—C13	2.5 (4)
C6—C1—C2—C3	1.1 (3)	C9—C10—C11—N2	−1.1 (3)
C8—C1—C2—C3	−177.91 (18)	C9—C10—C11—C12	178.5 (2)
C2—C1—C6—C5	−2.0 (3)	C13—C10—C11—N2	178.2 (2)

C8—C1—C6—C5	177.09 (19)	C13—C10—C11—C12	−2.2 (3)
C2—C1—C8—N1	9.0 (3)		

Hydrogen-bond geometry (Å, º)

Cg1 is the centroid of the C1–C6 benzene ring.

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C7—H7*B*···O1^{i}	0.97	2.58	3.264 (3)	128
C12—H12*A*···*Cg*1^{ii}	0.96	2.95	3.763 (2)	143

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