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3,4-Dimethyl-*N*-[(*E*)-3-nitrobenzylidene]-1,2-oxazol-5-amine

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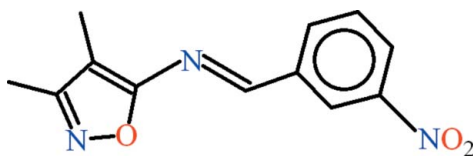
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Key indicators: single-crystal X-ray study; $T = 296$ K; mean $\sigma(\text{C}-\text{C}) = 0.006$ Å; R factor = 0.061; wR factor = 0.161; data-to-parameter ratio = 12.3.

In the title compound, $\text{C}_{12}\text{H}_{11}\text{N}_3\text{O}_3$, the dihedral angle between the 3-nitrobenzaldehyde and 5-amino-3,4-dimethyl-1,2-oxazole moieties is 2.46 (12°). The molecule is close to planar, the r.m.s. deviation for the non-H atoms being 0.028 Å. The packing only features van der Waals interactions between the molecules.

Related literature

For background and related crystal structures, see: Asiri *et al.* (2010*a,b,c,d*).



Experimental

Crystal data

$\text{C}_{12}\text{H}_{11}\text{N}_3\text{O}_3$
 $M_r = 245.24$

Monoclinic, $P2_1/c$
 $a = 12.602$ (2) Å

$b = 3.9267$ (6) Å
 $c = 23.366$ (4) Å
 $\beta = 94.791$ (9°)
 $V = 1152.3$ (3) Å³
 $Z = 4$

Mo $K\alpha$ radiation
 $\mu = 0.11$ mm⁻¹
 $T = 296$ K
 $0.22 \times 0.08 \times 0.06$ mm

Data collection

Bruker Kappa APEXII CCD diffractometer
Absorption correction: multi-scan (SADABS; Bruker, 2005)
 $T_{\min} = 0.992$, $T_{\max} = 0.995$

8616 measured reflections
2046 independent reflections
846 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.097$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.061$
 $wR(F^2) = 0.161$
 $S = 0.99$
2046 reflections

166 parameters
H-atom parameters constrained
 $\Delta\rho_{\max} = 0.16$ e Å⁻³
 $\Delta\rho_{\min} = -0.19$ e Å⁻³

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 (Farrugia, 1997) and PLATON (Spek, 2009); software used to prepare material for publication: WinGX (Farrugia, 1999) and PLATON.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: HB5634).

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supporting information

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3,4-Dimethyl-*N*-[(*E*)-3-nitrobenzylidene]-1,2-oxazol-5-amine

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S1. Comment

The title compound (I, Fig. 1) is being reported in continuation of our synthetic and structural studies of various Schiff bases of 5-amino-3,4-dimethylisoxazole (Asiri *et al.*, 2010*a, b, c, d*).

In (I), the 3-nitrobenzaldehyde moiety A (C1—C7/N1/O1/O2) and 5-amino-3,4-dimethylisoxazole moiety B (N2/C8—C12/N3/O3) are planar with r. m. s. deviation of 0.0124 and 0.0099 Å, respectively. The dihedral angle between A/B is 2.46 (12)°. All the heavy atoms (C1—C12/N1—N3/O1—O3) consituate plane with r. m. s. deviation of 0.0276 Å. In this plane, the methyl atom C12 deviates at the maximum with 0.0721 (33) Å. The title compound essentially consists of monomers. There exists no $\pi\cdots\pi$ interactions in the crystal.

S2. Experimental

A mixture of 4-nitrobenzaldehyde (0.33 g, 2.2 mmol) and 5-amino-3,4-dimethylisoxazole (0.24 g, 2.2 mmol) in ethanol (15 ml) was refluxed for 5 h with stirring to give a light yellow precipitate. This material was filtered off and washed with ethanol to give long thin needles of (I).

Yield: 56.45%; m.p. 463–464 K.

IR (KBr) ν_{\max} cm⁻¹: 3069 (C—H for CH₃), 2922 (C—H), 1568 (C=C), 1523 (C=N), 1162 (C—N).

S3. Refinement

The H-atoms were positioned geometrically (C—H = 0.93–0.96 Å) and refined as riding with $U_{\text{iso}}(\text{H}) = xU_{\text{eq}}(\text{C})$, where $x = 1.5$ for methyl and $x = 1.2$ for other H-atoms.

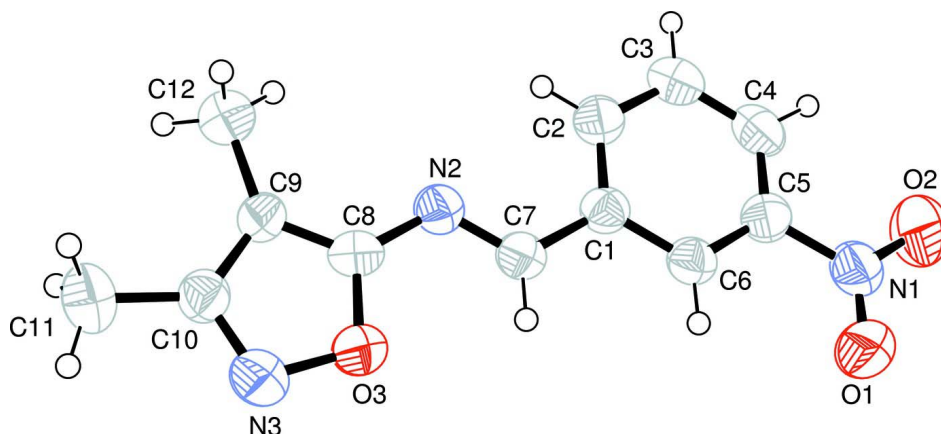


Figure 1

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

3,4-Dimethyl-*N*-[(*E*)-3-nitrobenzylidene]-1,2-oxazol-5-amine

Crystal data

C₁₂H₁₁N₃O₃ $M_r = 245.24$ Monoclinic, $P2_1/c$

Hall symbol: -P 2ybc

 $a = 12.602$ (2) Å $b = 3.9267$ (6) Å $c = 23.366$ (4) Å $\beta = 94.791$ (9)° $V = 1152.3$ (3) Å³ $Z = 4$ $F(000) = 512$ $D_x = 1.414$ Mg m⁻³Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å

Cell parameters from 846 reflections

 $\theta = 2.3$ – 25.0 ° $\mu = 0.11$ mm⁻¹ $T = 296$ K

Needle, colorless

 $0.22 \times 0.08 \times 0.06$ mm

Data collection

Bruker Kappa APEXII CCD

diffractometer

Radiation source: fine-focus sealed tube

Graphite monochromator

Detector resolution: 8.20 pixels mm⁻¹ ω scans

Absorption correction: multi-scan

(SADABS; Bruker, 2005)

 $T_{\min} = 0.992$, $T_{\max} = 0.995$

8616 measured reflections

2046 independent reflections

846 reflections with $I > 2\sigma(I)$ $R_{\text{int}} = 0.097$ $\theta_{\max} = 25.0$ °, $\theta_{\min} = 2.3$ ° $h = -15$ → 15 $k = -4$ → 4 $l = -27$ → 27

Refinement

Refinement on F^2

Least-squares matrix: full

 $R[F^2 > 2\sigma(F^2)] = 0.061$ $wR(F^2) = 0.161$ $S = 0.99$

2046 reflections

166 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

 $w = 1/[\sigma^2(F_o^2) + (0.0341P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$ $(\Delta/\sigma)_{\max} < 0.001$ $\Delta\rho_{\max} = 0.16$ e Å⁻³ $\Delta\rho_{\min} = -0.19$ 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)

	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{iso}}^*/U_{\text{eq}}$
O1	0.0661 (2)	0.9618 (9)	0.11316 (16)	0.0932 (16)
O2	0.0217 (3)	0.7756 (9)	0.19373 (16)	0.1103 (19)
O3	0.5961 (2)	0.7628 (6)	0.02581 (12)	0.0630 (11)
N1	0.0873 (3)	0.8153 (10)	0.15894 (19)	0.0708 (17)
N2	0.5490 (3)	0.5694 (8)	0.11691 (13)	0.0523 (12)

N3	0.6882 (3)	0.7702 (8)	-0.00509 (15)	0.0660 (17)
C1	0.3736 (3)	0.6218 (9)	0.14659 (17)	0.0475 (17)
C2	0.3985 (3)	0.4605 (10)	0.19866 (18)	0.0579 (17)
C3	0.3218 (4)	0.4146 (10)	0.23763 (18)	0.0629 (17)
C4	0.2194 (4)	0.5255 (10)	0.22380 (19)	0.0618 (17)
C5	0.1954 (3)	0.6856 (10)	0.17248 (19)	0.0534 (17)
C6	0.2697 (3)	0.7379 (9)	0.13294 (17)	0.0511 (17)
C7	0.4530 (3)	0.6720 (9)	0.10518 (17)	0.0527 (17)
C8	0.6232 (3)	0.6142 (10)	0.07766 (17)	0.0521 (17)
C9	0.7266 (3)	0.5238 (9)	0.08165 (17)	0.0485 (17)
C10	0.7629 (3)	0.6275 (9)	0.02938 (19)	0.0513 (17)
C11	0.8723 (3)	0.5881 (10)	0.00968 (19)	0.0727 (19)
C12	0.7889 (3)	0.3614 (10)	0.13104 (17)	0.0672 (17)
H2	0.46743	0.38159	0.20778	0.0695*
H3	0.33974	0.30952	0.27277	0.0758*
H4	0.16713	0.49195	0.24908	0.0742*
H6	0.25095	0.84733	0.09826	0.0611*
H7	0.43386	0.77846	0.07029	0.0629*
H11A	0.87321	0.67477	-0.02869	0.1088*
H11B	0.89153	0.35146	0.01024	0.1088*
H11C	0.92235	0.71279	0.03486	0.1088*
H12A	0.82773	0.53290	0.15333	0.1006*
H12B	0.83789	0.20017	0.11712	0.1006*
H12C	0.74137	0.24627	0.15460	0.1006*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.064 (2)	0.141 (3)	0.074 (3)	0.0185 (19)	0.003 (2)	0.011 (2)
O2	0.066 (3)	0.170 (4)	0.100 (3)	0.011 (2)	0.037 (2)	0.017 (2)
O3	0.0537 (19)	0.084 (2)	0.051 (2)	0.0055 (14)	0.0022 (15)	0.0092 (15)
N1	0.057 (3)	0.097 (3)	0.059 (3)	0.002 (2)	0.009 (2)	-0.011 (2)
N2	0.048 (2)	0.061 (2)	0.048 (2)	-0.0018 (17)	0.0047 (18)	-0.0012 (16)
N3	0.065 (3)	0.080 (3)	0.054 (3)	-0.001 (2)	0.011 (2)	0.0068 (19)
C1	0.050 (3)	0.047 (3)	0.045 (3)	-0.0016 (19)	0.001 (2)	-0.005 (2)
C2	0.052 (3)	0.068 (3)	0.053 (3)	-0.001 (2)	0.000 (2)	-0.010 (2)
C3	0.073 (3)	0.071 (3)	0.045 (3)	-0.003 (2)	0.006 (3)	-0.003 (2)
C4	0.065 (3)	0.073 (3)	0.049 (3)	-0.007 (2)	0.014 (2)	-0.010 (2)
C5	0.046 (3)	0.059 (3)	0.054 (3)	-0.006 (2)	-0.002 (2)	-0.013 (2)
C6	0.050 (3)	0.058 (3)	0.045 (3)	-0.0016 (19)	0.003 (2)	-0.005 (2)
C7	0.049 (3)	0.065 (3)	0.044 (3)	-0.001 (2)	0.004 (2)	0.000 (2)
C8	0.056 (3)	0.056 (3)	0.043 (3)	-0.001 (2)	-0.003 (2)	0.003 (2)
C9	0.042 (3)	0.055 (3)	0.048 (3)	0.001 (2)	0.001 (2)	-0.003 (2)
C10	0.049 (3)	0.053 (3)	0.052 (3)	0.001 (2)	0.004 (2)	-0.005 (2)
C11	0.063 (3)	0.079 (3)	0.079 (4)	0.000 (2)	0.024 (3)	0.000 (3)
C12	0.064 (3)	0.078 (3)	0.059 (3)	0.010 (2)	0.002 (2)	0.002 (2)

Geometric parameters (Å, °)

O1—N1	1.224 (6)	C8—C9	1.346 (5)
O2—N1	1.217 (6)	C9—C10	1.400 (6)
O3—N3	1.418 (5)	C9—C12	1.484 (5)
O3—C8	1.362 (5)	C10—C11	1.498 (5)
N1—C5	1.464 (5)	C2—H2	0.9300
N2—C7	1.283 (5)	C3—H3	0.9300
N2—C8	1.375 (5)	C4—H4	0.9300
N3—C10	1.312 (5)	C6—H6	0.9300
C1—C2	1.384 (6)	C7—H7	0.9300
C1—C6	1.398 (5)	C11—H11A	0.9600
C1—C7	1.462 (5)	C11—H11B	0.9600
C2—C3	1.394 (6)	C11—H11C	0.9600
C3—C4	1.375 (7)	C12—H12A	0.9600
C4—C5	1.365 (6)	C12—H12B	0.9600
C5—C6	1.385 (6)	C12—H12C	0.9600
N3—O3—C8	107.9 (3)	N3—C10—C11	119.2 (4)
O1—N1—O2	122.2 (4)	C9—C10—C11	127.9 (4)
O1—N1—C5	118.9 (4)	C1—C2—H2	120.00
O2—N1—C5	118.9 (4)	C3—C2—H2	120.00
C7—N2—C8	120.0 (3)	C2—C3—H3	120.00
O3—N3—C10	104.8 (3)	C4—C3—H3	120.00
C2—C1—C6	119.3 (4)	C3—C4—H4	120.00
C2—C1—C7	121.7 (3)	C5—C4—H4	120.00
C6—C1—C7	119.0 (3)	C1—C6—H6	121.00
C1—C2—C3	121.0 (4)	C5—C6—H6	121.00
C2—C3—C4	119.6 (4)	N2—C7—H7	120.00
C3—C4—C5	119.2 (4)	C1—C7—H7	120.00
N1—C5—C4	118.9 (4)	C10—C11—H11A	109.00
N1—C5—C6	118.2 (4)	C10—C11—H11B	109.00
C4—C5—C6	122.8 (4)	C10—C11—H11C	109.00
C1—C6—C5	118.2 (4)	H11A—C11—H11B	109.00
N2—C7—C1	120.1 (3)	H11A—C11—H11C	109.00
O3—C8—N2	120.9 (3)	H11B—C11—H11C	109.00
O3—C8—C9	110.1 (3)	C9—C12—H12A	109.00
N2—C8—C9	128.9 (4)	C9—C12—H12B	109.00
C8—C9—C10	104.4 (3)	C9—C12—H12C	109.00
C8—C9—C12	127.8 (4)	H12A—C12—H12B	109.00
C10—C9—C12	127.8 (3)	H12A—C12—H12C	109.00
N3—C10—C9	112.9 (3)	H12B—C12—H12C	109.00
C8—O3—N3—C10	0.2 (4)	C7—C1—C6—C5	179.5 (3)
N3—O3—C8—C9	-0.5 (4)	C2—C1—C7—N2	-0.9 (6)
N3—O3—C8—N2	-179.4 (3)	C1—C2—C3—C4	1.2 (6)
O2—N1—C5—C4	-0.3 (6)	C2—C3—C4—C5	-1.4 (6)
O2—N1—C5—C6	-178.9 (4)	C3—C4—C5—N1	-177.6 (4)

O1—N1—C5—C4	179.3 (4)	C3—C4—C5—C6	0.9 (6)
O1—N1—C5—C6	0.7 (6)	N1—C5—C6—C1	178.4 (3)
C7—N2—C8—C9	-179.6 (4)	C4—C5—C6—C1	-0.2 (6)
C8—N2—C7—C1	179.4 (3)	O3—C8—C9—C10	0.5 (4)
C7—N2—C8—O3	-0.9 (5)	N2—C8—C9—C12	-2.5 (7)
O3—N3—C10—C11	179.1 (3)	O3—C8—C9—C12	178.7 (3)
O3—N3—C10—C9	0.1 (4)	N2—C8—C9—C10	179.4 (4)
C6—C1—C2—C3	-0.4 (6)	C8—C9—C10—N3	-0.4 (4)
C7—C1—C2—C3	-180.0 (4)	C12—C9—C10—C11	2.5 (6)
C6—C1—C7—N2	179.6 (3)	C8—C9—C10—C11	-179.3 (4)
C2—C1—C6—C5	-0.1 (5)	C12—C9—C10—N3	-178.6 (4)
