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N-[4-(Dimethylamino)benzylidene]-3,4-dimethylisoxazol-5-amine

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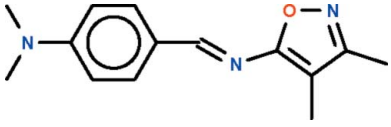
Key indicators: single-crystal X-ray study; T = 100 K; mean σ(C–C) = 0.002 Å; R factor = 0.039; wR factor = 0.113; data-to-parameter ratio = 17.1.

The aromatic rings attached to the azomethine double bond in the title compound, $C_{14}H_{17}N_3O$, are *trans* to each other [C—C═N—C torsion angle = 179.5 (1)°], and they are approximately coplanar [dihedral angle between the five- and six-membered rings = 13.7 (1)°].

Related literature

For the spectroscopic characterization of a related Schiff base, see: Asiri *et al.* (2010).



Experimental

Crystal data

$C_{14}H_{17}N_3O$
M_r = 243.31
Triclinic, $P\overline{1}$
a = 6.5772 (6) Å
b = 9.1246 (9) Å
c = 10.538 (1) Å
α = 92.995 (1)°
β = 95.183 (1)°
γ = 90.873 (1)°
V = 628.86 (10) Å^3
Z = 2
Mo $K\alpha$ radiation
μ = 0.08 mm^{-1}
T = 100 K
0.35 × 0.15 × 0.10 mm

Data collection

Bruker SMART APEX diffractometer
6092 measured reflections
2866 independent reflections
2401 reflections with $I > 2\sigma(I)$
R_{int} = 0.023

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.039
$wR(F^2)$ = 0.113
S = 1.04
2866 reflections
168 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.25 e Å^{-3}
$\Delta\rho_{min}$ = −0.24 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: *X-SEED* (Barbour, 2001); software used to prepare material for publication: *publCIF* (Westrip, 2010).

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: JH2170).

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supplementary materials

Acta Cryst. (2010). E**66**, o1783 [**doi:10.1107/S1600536810023780**]

N-[4-(Dimethylamino)benzylidene]-3,4-dimethylisoxazol-5-amine

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Comment

Although there is a large number of crystal structure studies of Schiff bases derived by condensing an aromatic aldehyde and an aromatic amine, there has not been any structural report on the condensation product involving 5-amino-3,4-dimethylisoxazole, a commerically available chemical. We have recently reported the spectroscopic characterization of the *N*-ethylcarbazole-3-aldehyde condensation product of this amine (Asiri *et al.*, 2010). The 4-dimethylaminobenzaldehyde condensation product (Scheme I, Fig. 1) features an azomethine double-bond whose aromatic substituents are located in *trans* positions. The rings are coplanar [C–C═N–C torsion angle 179.5 (1) °].

Experimental

5-Amino-3,4-dimethylisoxazole (0.36 g, 3.2 mol) and *N*,*N*-dimethylaminobenzaldehyde (0.5 g, 3.2 mol) were heated in methanol (15 ml) for 5 h. The solvent was removed and the solid material recrystallized from methanol to give the crystalline Schiff base.

Refinement

Carbon-bound H-atoms were placed in calculated positions [C–H 0.95 to 0.98 Å, *U*(H) 1.2 to 1.5U_{eq}(C)] and were included in the refinement in the riding model approximation.

Figures

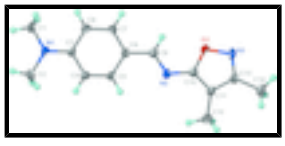

Fig. 1. Thermal ellipsoid plot (Barbour, 2001) of $C_{14}H_{13}N_3O$ at the 70% probability level; hydrogen atoms are drawn as spheres of arbitrary radius.

N-[4-(Dimethylamino)benzylidene]-3,4-dimethylisoxazol-5-amine

Crystal data

$C_{14}H_{17}N_3O$	$Z = 2$
$M_r = 243.31$	$F(000) = 260$
Triclinic, $P\overline{1}$	$D_x = 1.285$ Mg m^{-3}
Hall symbol: -P 1	Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
$a = 6.5772$ (6) Å	Cell parameters from 2610 reflections
$b = 9.1246$ (9) Å	$\theta = 2.2$–$28.3°$
$c = 10.538$ (1) Å	$\mu = 0.08$ mm^{-1}
$\alpha = 92.995$ (1)°	$T = 100$ K

β = 95.183 (1)°	Prism, yellow
γ = 90.873 (1)°	0.35 × 0.15 × 0.10 mm
V = 628.86 (10) Å^3	

Data collection

Bruker SMART APEX diffractometer	2401 reflections with $I > 2\sigma(I)$
Radiation source: fine-focus sealed tube	R_{int} = 0.023
graphite	θ_{max} = 27.5°, θ_{min} = 1.9°
ω scans	h = −8→8
6092 measured reflections	k = −11→11
2866 independent reflections	l = −13→13

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)]$ = 0.039	H-atom parameters constrained
$wR(F^2)$ = 0.113	$w = 1/[\sigma^2(F_o^2) + (0.0652P)^2 + 0.0819P]$ where $P = (F_o^2 + 2F_c^2)/3$
S = 1.04	$(\Delta/\sigma)_{max}$ = 0.001
2866 reflections	$\Delta\rho_{max}$ = 0.25 e Å^{-3}
168 parameters	$\Delta\rho_{min}$ = −0.24 e Å^{-3}
0 restraints	Extinction correction: *SHELXL97* (Sheldrick, 2008), $Fc^*=kFc[1+0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$
Primary atom site location: structure-invariant direct methods	Extinction coefficient: 0.024 (5)

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

	x	y	z	U_{iso}*/U_{eq}
O1	0.34307 (12)	0.64426 (8)	0.20359 (7)	0.0212 (2)
N2	0.46514 (14)	0.43648 (10)	0.31374 (9)	0.0188 (2)
N3	0.41200 (15)	0.73832 (10)	0.11248 (9)	0.0229 (2)
N1	0.13966 (15)	0.02064 (10)	0.73244 (9)	0.0214 (2)
C1	−0.06808 (18)	−0.00795 (14)	0.76262 (12)	0.0275 (3)
H1A	−0.1258	0.0831	0.7959	0.041*
H1B	−0.0673	−0.0816	0.8271	0.041*
H1C	−0.1511	−0.0446	0.6852	0.041*
C3	0.17610 (17)	0.11519 (11)	0.64040 (10)	0.0175 (2)
C2	0.29986 (19)	−0.07284 (12)	0.78465 (12)	0.0251 (3)
H2A	0.3270	−0.1499	0.7204	0.038*
H2B	0.2561	−0.1179	0.8604	0.038*
H2C	0.4245	−0.0138	0.8084	0.038*
C4	0.37269 (17)	0.13022 (12)	0.59705 (10)	0.0189 (2)
H4	0.4797	0.0699	0.6293	0.023*

C5	0.41094 (17)	0.23055 (12)	0.50920 (10)	0.0184 (2)
H5	0.5447	0.2391	0.4827	0.022*
C6	0.25736 (16)	0.32079 (12)	0.45762 (10)	0.0176 (2)
C7	0.06149 (17)	0.30343 (12)	0.49799 (11)	0.0194 (2)
H7	−0.0457	0.3622	0.4634	0.023*
C8	0.01988 (17)	0.20356 (12)	0.58643 (11)	0.0201 (2)
H8	−0.1148	0.1941	0.6113	0.024*
C9	0.29510 (17)	0.42739 (12)	0.36576 (10)	0.0185 (2)
H9	0.1903	0.4939	0.3424	0.022*
C10	0.49005 (17)	0.54187 (12)	0.22757 (10)	0.0180 (2)
C11	0.64745 (16)	0.56405 (11)	0.15557 (10)	0.0180 (2)
C12	0.83736 (17)	0.47749 (13)	0.14888 (11)	0.0237 (3)
H12A	0.8486	0.4097	0.2182	0.036*
H12B	0.9564	0.5444	0.1574	0.036*
H12C	0.8318	0.4215	0.0667	0.036*
C13	0.58994 (17)	0.68813 (12)	0.08652 (10)	0.0197 (2)
C14	0.71023 (19)	0.76199 (13)	−0.00676 (11)	0.0245 (3)
H14A	0.7154	0.6972	−0.0835	0.037*
H14B	0.8493	0.7833	0.0320	0.037*
H14C	0.6448	0.8538	−0.0299	0.037*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.0190 (4)	0.0238 (4)	0.0218 (4)	−0.0006 (3)	0.0047 (3)	0.0072 (3)
N2	0.0200 (5)	0.0196 (5)	0.0168 (5)	−0.0031 (4)	0.0033 (4)	0.0010 (3)
N3	0.0236 (5)	0.0256 (5)	0.0203 (5)	−0.0034 (4)	0.0033 (4)	0.0088 (4)
N1	0.0200 (5)	0.0231 (5)	0.0224 (5)	−0.0003 (4)	0.0047 (4)	0.0078 (4)
C1	0.0240 (6)	0.0296 (6)	0.0311 (7)	−0.0021 (5)	0.0093 (5)	0.0117 (5)
C3	0.0197 (6)	0.0169 (5)	0.0160 (5)	−0.0017 (4)	0.0026 (4)	0.0006 (4)
C2	0.0285 (6)	0.0216 (6)	0.0267 (6)	0.0039 (5)	0.0053 (5)	0.0092 (5)
C4	0.0176 (5)	0.0198 (5)	0.0193 (5)	0.0015 (4)	0.0022 (4)	0.0015 (4)
C5	0.0157 (5)	0.0208 (5)	0.0190 (5)	−0.0020 (4)	0.0039 (4)	−0.0001 (4)
C6	0.0179 (5)	0.0188 (5)	0.0159 (5)	−0.0020 (4)	0.0018 (4)	0.0001 (4)
C7	0.0165 (5)	0.0224 (5)	0.0197 (5)	0.0010 (4)	0.0015 (4)	0.0034 (4)
C8	0.0160 (5)	0.0241 (6)	0.0207 (6)	−0.0013 (4)	0.0038 (4)	0.0026 (4)
C9	0.0173 (5)	0.0211 (5)	0.0168 (5)	−0.0012 (4)	0.0007 (4)	0.0012 (4)
C10	0.0182 (5)	0.0189 (5)	0.0165 (5)	−0.0020 (4)	0.0000 (4)	0.0003 (4)
C11	0.0184 (5)	0.0206 (5)	0.0146 (5)	−0.0047 (4)	0.0010 (4)	0.0004 (4)
C12	0.0197 (6)	0.0280 (6)	0.0242 (6)	−0.0016 (4)	0.0045 (5)	0.0038 (5)
C13	0.0205 (6)	0.0227 (5)	0.0156 (5)	−0.0052 (4)	0.0003 (4)	0.0006 (4)
C14	0.0267 (6)	0.0273 (6)	0.0201 (6)	−0.0054 (5)	0.0039 (5)	0.0046 (5)

Geometric parameters (Å, °)

O1—C10	1.3709 (13)	C5—C6	1.4025 (15)
O1—N3	1.4201 (11)	C5—H5	0.9500
N2—C9	1.2926 (14)	C6—C7	1.4020 (15)
N2—C10	1.3745 (14)	C6—C9	1.4419 (15)

N3—C13	1.3097 (15)	C7—C8	1.3788 (15)
N1—C3	1.3659 (14)	C7—H7	0.9500
N1—C2	1.4533 (14)	C8—H8	0.9500
N1—C1	1.4534 (14)	C9—H9	0.9500
C1—H1A	0.9800	C10—C11	1.3565 (15)
C1—H1B	0.9800	C11—C13	1.4155 (15)
C1—H1C	0.9800	C11—C12	1.4930 (15)
C3—C8	1.4132 (15)	C12—H12A	0.9800
C3—C4	1.4168 (15)	C12—H12B	0.9800
C2—H2A	0.9800	C12—H12C	0.9800
C2—H2B	0.9800	C13—C14	1.4966 (15)
C2—H2C	0.9800	C14—H14A	0.9800
C4—C5	1.3718 (15)	C14—H14B	0.9800
C4—H4	0.9500	C14—H14C	0.9800
C10—O1—N3	107.86 (8)	C8—C7—C6	121.91 (10)
C9—N2—C10	119.52 (10)	C8—C7—H7	119.0
C13—N3—O1	105.28 (9)	C6—C7—H7	119.0
C3—N1—C2	120.76 (9)	C7—C8—C3	120.48 (10)
C3—N1—C1	120.12 (9)	C7—C8—H8	119.8
C2—N1—C1	118.15 (9)	C3—C8—H8	119.8
N1—C1—H1A	109.5	N2—C9—C6	122.97 (10)
N1—C1—H1B	109.5	N2—C9—H9	118.5
H1A—C1—H1B	109.5	C6—C9—H9	118.5
N1—C1—H1C	109.5	C11—C10—O1	109.95 (10)
H1A—C1—H1C	109.5	C11—C10—N2	129.32 (10)
H1B—C1—H1C	109.5	O1—C10—N2	120.73 (9)
N1—C3—C8	121.22 (10)	C10—C11—C13	104.10 (10)
N1—C3—C4	121.27 (10)	C10—C11—C12	128.36 (10)
C8—C3—C4	117.50 (10)	C13—C11—C12	127.54 (10)
N1—C2—H2A	109.5	C11—C12—H12A	109.5
N1—C2—H2B	109.5	C11—C12—H12B	109.5
H2A—C2—H2B	109.5	H12A—C12—H12B	109.5
N1—C2—H2C	109.5	C11—C12—H12C	109.5
H2A—C2—H2C	109.5	H12A—C12—H12C	109.5
H2B—C2—H2C	109.5	H12B—C12—H12C	109.5
C5—C4—C3	121.04 (10)	N3—C13—C11	112.81 (10)
C5—C4—H4	119.5	N3—C13—C14	120.31 (10)
C3—C4—H4	119.5	C11—C13—C14	126.88 (11)
C4—C5—C6	121.58 (10)	C13—C14—H14A	109.5
C4—C5—H5	119.2	C13—C14—H14B	109.5
C6—C5—H5	119.2	H14A—C14—H14B	109.5
C7—C6—C5	117.44 (10)	C13—C14—H14C	109.5
C7—C6—C9	120.23 (10)	H14A—C14—H14C	109.5
C5—C6—C9	122.33 (10)	H14B—C14—H14C	109.5
C10—O1—N3—C13	0.59 (11)	C7—C6—C9—N2	−171.57 (10)
C2—N1—C3—C8	178.20 (10)	C5—C6—C9—N2	7.97 (17)
C1—N1—C3—C8	9.66 (16)	N3—O1—C10—C11	−0.86 (11)
C2—N1—C3—C4	−2.91 (16)	N3—O1—C10—N2	179.38 (9)

C1—N1—C3—C4	−171.45 (10)	C9—N2—C10—C11	−174.71 (11)
N1—C3—C4—C5	−176.74 (10)	C9—N2—C10—O1	5.00 (15)
C8—C3—C4—C5	2.19 (16)	O1—C10—C11—C13	0.75 (12)
C3—C4—C5—C6	−0.80 (17)	N2—C10—C11—C13	−179.51 (10)
C4—C5—C6—C7	−0.81 (16)	O1—C10—C11—C12	−178.70 (10)
C4—C5—C6—C9	179.64 (9)	N2—C10—C11—C12	1.03 (19)
C5—C6—C7—C8	1.00 (16)	O1—N3—C13—C11	−0.13 (12)
C9—C6—C7—C8	−179.45 (10)	O1—N3—C13—C14	−179.61 (9)
C6—C7—C8—C3	0.44 (17)	C10—C11—C13—N3	−0.38 (13)
N1—C3—C8—C7	176.93 (10)	C12—C11—C13—N3	179.08 (10)
C4—C3—C8—C7	−2.01 (16)	C10—C11—C13—C14	179.06 (10)
C10—N2—C9—C6	−179.54 (9)	C12—C11—C13—C14	−1.47 (18)

Fig. 1