# N-(4-Bromobenzylidene)-3,4-dimethyl-isoxazol-5-amine

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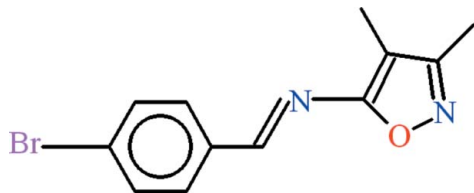Correspondence e-mail: dmntahir_uos@yahoo.com

In the title compound, $C_{12}H_{11}BrN_2O$, the 4-bromo-benzaldehyde and 5-amino-3,4-dimethylisoxazole units are oriented at a dihedral angle of 4.89 (8)°. In the crystal, weak $\pi$–$\pi$ interactions are present between the benzene rings at a centroid–centroid distance of 3.7862 (14) Å.

## Related literature

For related structures, see: Asiri *et al.* (2010): Fun *et al.* (2010*a,b*): Shad *et al.* (2008): Tahir *et al.* (2008). For graph-set notation, see: Bernstein *et al.* (1995).



## Experimental

### Crystal data

| | |
|---|---|
| $C_{12}H_{11}BrN_2O$ | $b$ = 8.8709 (5) Å |
| $M_r$ = 279.14 | $c$ = 9.1052 (5) Å |
| Triclinic, $P\bar{1}$ | $\alpha$ = 97.024 (2)° |
| $a$ = 7.6406 (4) Å | $\beta$ = 102.961 (1)° |
| $\gamma$ = 92.786 (2)° | $\mu$ = 3.43 mm$^{-1}$ |
| $V$ = 595.06 (6) Å$^3$ | $T$ = 296 K |
| $Z$ = 2 | 0.30 × 0.14 × 0.12 mm |
| Mo $K\alpha$ radiation | |

### Data collection

| | |
|---|---|
| Bruker Kappa APEXII CCD diffractometer | 8212 measured reflections |
| Absorption correction: multi-scan (*SADABS*; Bruker, 2005) | 2119 independent reflections |
| $T_{min}$ = 0.568, $T_{max}$ = 0.665 | 1643 reflections with $I > 2\sigma(I)$ |
| | $R_{int}$ = 0.022 |

### Refinement

| | |
|---|---|
| $R[F^2 > 2\sigma(F^2)]$ = 0.025 | 147 parameters |
| $wR(F^2)$ = 0.059 | H-atom parameters constrained |
| $S$ = 1.03 | $\Delta\rho_{max}$ = 0.20 e Å$^{-3}$ |
| 2119 reflections | $\Delta\rho_{min}$ = −0.17 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 for Windows* (Farrugia, 1997) and *PLATON* (Spek, 2009); software used to prepare material for publication: *WinGX* (Farrugia, 1999) and *PLATON*.

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## supporting information

## *N*-(4-Bromobenzylidene)-3,4-dimethylisoxazol-5-amine

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

### S1. Comment

Heterocycles such as nitrogen and oxygen containing compounds are abundant in nature and are of great significance to life. We herein report the synthesis and crystal structure of title compound (I, Fig. 1).

The crystal structures of 4-chloro-2- [(*E*)-({4-[*N*-(3,4-dimethylisoxazol-5-yl)sulfamoyl]phenyl}iminio) methyl]-phenolate (Shad *et al.*, 2008), 4-bromo-2-((*E*)-{4-[(3,4-dimethylisoxazol-5-yl)sulfamoyl]phenyl} iminiomethyl)phenolate (Tahir *et al.*, 2008), 2-[(*E*)-(3,4-dimethylisoxazol-5-yl)iminomethyl]phenol (Fun *et al.*, 2010*a*), 1-[(*E*)-(3,4-dimethyl-isoxazol-5-yl)iminomethyl]-2-naphthol (Fun *et al.*, 2010*b*) and *N*-[4-(dimethylamino)benzylidene]-3,4-dimethyl-isoxazol-5-amine (Asiri *et al.*, 2010) have been published previously, which contain the 5-amino-3,4-dimethylisoxazole moiety.

In (I), the 4-bromobenzaldehyde moiety A (C1—C7/BR1) and 5-amino-3,4-dimethylisoxazole moiety B (N1/C8—C12/N2/O1) are planar with r. m. s. deviations of 0.0119 Å and 0.0128 Å, respectively. The dihedral angle between A/B is 4.89 (8)°. The title compound essentially consists of monomers. Weak intramolecular H-bonding of C—H···O type (Table 1, Fig. 1) exists and complete an S(5) ring motif (Bernstein *et al.*, 1995). There exists also $\pi$–$\pi$ interaction between the centroids of phenyl rings at a distance of 3.7862 (14) Å [symmetry code: -*x*, 2 - *y*, 1 - *z*].

### S2. Experimental

A mixture of 4-bromobenzaldehyde (0.40 g, 0.0022 mol) and 5-amino-3,4-dimethylisoxazole (0.24 g, 0.0022 mol) in ethanol (15 ml) was refluxed for 5 h with stirring to give a light brown needles of title compound (I).

### S3. Refinement

The H-atoms were positioned geometrically (C–H = 0.93–0.96 Å) and refined as riding with $U_{iso}$(H) = x$U_{eq}$(C), where x = 1.5 for methyl and x = 1.2 for aryl H-atoms.
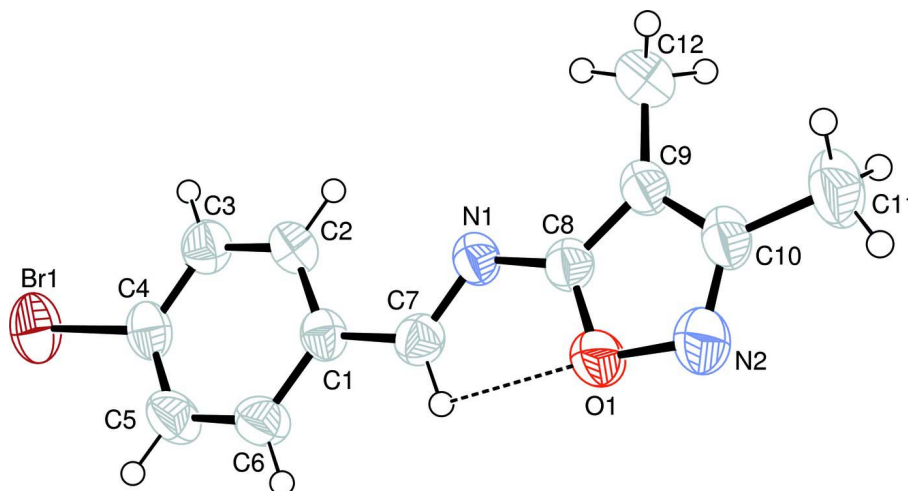
**Figure 1**

View of the title compound with the atom numbering scheme. The thermal ellipsoids are drawn at the 50% probability level. The dotted line indicate the intramolecular H-bond.

*N*-**(4-Bromobenzylidene)-3,4-dimethylisoxazol-5-amine**

*Crystal data*

| | |
|---|---|
| $C_{12}H_{11}BrN_2O$ | $Z = 2$ |
| $M_r = 279.14$ | $F(000) = 280$ |
| Triclinic, $P\overline{1}$ | $D_x = 1.558$ Mg m$^{-3}$ |
| Hall symbol: -P 1 | Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å |
| $a = 7.6406\ (4)$ Å | Cell parameters from 1643 reflections |
| $b = 8.8709\ (5)$ Å | $\theta = 2.3–25.3°$ |
| $c = 9.1052\ (5)$ Å | $\mu = 3.43$ mm$^{-1}$ |
| $\alpha = 97.024\ (2)°$ | $T = 296$ K |
| $\beta = 102.961\ (1)°$ | Needle, brown |
| $\gamma = 92.786\ (2)°$ | $0.30 \times 0.14 \times 0.12$ mm |
| $V = 595.06\ (6)$ Å$^3$ | |

*Data collection*

| | |
|---|---|
| Bruker Kappa APEXII CCD diffractometer | 8212 measured reflections |
| Radiation source: fine-focus sealed tube | 2119 independent reflections |
| Graphite monochromator | 1643 reflections with $I > 2\sigma(I)$ |
| Detector resolution: 8.10 pixels mm$^{-1}$ | $R_{int} = 0.022$ |
| $\omega$ scans | $\theta_{max} = 25.3°$, $\theta_{min} = 2.3°$ |
| Absorption correction: multi-scan (*SADABS*; Bruker, 2005) | $h = -9 \rightarrow 9$ |
| | $k = -10 \rightarrow 10$ |
| $T_{min} = 0.568$, $T_{max} = 0.665$ | $l = -10 \rightarrow 10$ |

*Refinement*

| | |
|---|---|
| Refinement on $F^2$ | 0 restraints |
| Least-squares matrix: full | Primary atom site location: structure-invariant direct methods |
| $R[F^2 > 2\sigma(F^2)] = 0.025$ | |
| $wR(F^2) = 0.059$ | Secondary atom site location: difference Fourier map |
| $S = 1.03$ | |
| 2119 reflections | Hydrogen site location: inferred from neighbouring sites |
| 147 parameters | |

| | |
|---|---|
| H-atom parameters constrained | $(\Delta/\sigma)_{max} = 0.001$ |
| $w = 1/[\sigma^2(F_o^2) + (0.0225P)^2 + 0.2246P]$ | $\Delta\rho_{max} = 0.20$ e Å$^{-3}$ |
| where $P = (F_o^2 + 2F_c^2)/3$ | $\Delta\rho_{min} = -0.17$ 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 (Å$^2$)*

| | *x* | *y* | *z* | $U_{iso}$*/$U_{eq}$ |
|---|---|---|---|---|
| Br1 | −0.32255 (4) | 1.00984 (3) | 0.72075 (3) | 0.0712 (1) |
| O1 | 0.4539 (2) | 0.51578 (18) | 0.30328 (18) | 0.0572 (6) |
| N1 | 0.1487 (3) | 0.55019 (19) | 0.3071 (2) | 0.0440 (6) |
| N2 | 0.5503 (3) | 0.4289 (3) | 0.2111 (3) | 0.0656 (8) |
| C1 | 0.0706 (3) | 0.7262 (2) | 0.4987 (2) | 0.0421 (8) |
| C2 | −0.1140 (3) | 0.7008 (2) | 0.4413 (3) | 0.0471 (8) |
| C3 | −0.2317 (3) | 0.7844 (3) | 0.5070 (3) | 0.0509 (8) |
| C4 | −0.1622 (3) | 0.8940 (2) | 0.6301 (3) | 0.0489 (9) |
| C5 | 0.0186 (4) | 0.9215 (3) | 0.6888 (3) | 0.0571 (9) |
| C6 | 0.1356 (3) | 0.8365 (3) | 0.6240 (3) | 0.0538 (9) |
| C7 | 0.1988 (3) | 0.6429 (2) | 0.4298 (3) | 0.0455 (8) |
| C8 | 0.2749 (3) | 0.4794 (2) | 0.2430 (3) | 0.0433 (8) |
| C9 | 0.2501 (3) | 0.3744 (2) | 0.1180 (3) | 0.0457 (8) |
| C10 | 0.4265 (3) | 0.3478 (3) | 0.1044 (3) | 0.0517 (9) |
| C11 | 0.4836 (4) | 0.2423 (3) | −0.0149 (3) | 0.0745 (11) |
| C12 | 0.0764 (4) | 0.3028 (3) | 0.0200 (3) | 0.0659 (10) |
| H2 | −0.15915 | 0.62688 | 0.35786 | 0.0565* |
| H3 | −0.35551 | 0.76687 | 0.46887 | 0.0611* |
| H5 | 0.06270 | 0.99657 | 0.77139 | 0.0686* |
| H6 | 0.25905 | 0.85335 | 0.66457 | 0.0646* |
| H7 | 0.32075 | 0.65805 | 0.47691 | 0.0546* |
| H11A | 0.61269 | 0.24881 | 0.00435 | 0.1118* |
| H11B | 0.43483 | 0.27080 | −0.11341 | 0.1118* |
| H11C | 0.43975 | 0.13964 | −0.01207 | 0.1118* |
| H12A | −0.02153 | 0.34332 | 0.05809 | 0.0989* |
| H12B | 0.07209 | 0.19462 | 0.02086 | 0.0989* |
| H12C | 0.06658 | 0.32433 | −0.08221 | 0.0989* |

*Atomic displacement parameters (Å$^2$)*

| | $U^{11}$ | $U^{22}$ | $U^{33}$ | $U^{12}$ | $U^{13}$ | $U^{23}$ |
|---|---|---|---|---|---|---|
| Br1 | 0.0760 (2) | 0.0641 (2) | 0.0851 (2) | 0.0188 (1) | 0.0448 (2) | 0.0012 (1) |
| O1 | 0.0487 (11) | 0.0650 (10) | 0.0542 (10) | 0.0090 (8) | 0.0134 (8) | −0.0109 (8) |

| | | | | | | |
|---|---|---|---|---|---|---|
| N1 | 0.0499 (12) | 0.0408 (10) | 0.0421 (11) | 0.0068 (8) | 0.0138 (9) | 0.0023 (9) |
| N2 | 0.0549 (14) | 0.0742 (14) | 0.0684 (15) | 0.0155 (11) | 0.0226 (12) | −0.0074 (12) |
| C1 | 0.0501 (15) | 0.0399 (12) | 0.0394 (13) | 0.0059 (10) | 0.0163 (10) | 0.0061 (10) |
| C2 | 0.0523 (16) | 0.0422 (12) | 0.0466 (14) | 0.0001 (10) | 0.0157 (11) | −0.0006 (10) |
| C3 | 0.0448 (15) | 0.0493 (13) | 0.0603 (16) | 0.0033 (10) | 0.0164 (12) | 0.0063 (12) |
| C4 | 0.0598 (17) | 0.0419 (12) | 0.0521 (15) | 0.0077 (10) | 0.0271 (12) | 0.0063 (11) |
| C5 | 0.0624 (19) | 0.0568 (15) | 0.0492 (15) | 0.0015 (12) | 0.0174 (12) | −0.0115 (12) |
| C6 | 0.0456 (15) | 0.0632 (15) | 0.0486 (15) | 0.0020 (11) | 0.0104 (11) | −0.0063 (12) |
| C7 | 0.0449 (14) | 0.0482 (13) | 0.0446 (14) | 0.0075 (10) | 0.0117 (10) | 0.0071 (11) |
| C8 | 0.0476 (15) | 0.0428 (12) | 0.0410 (13) | 0.0081 (10) | 0.0119 (10) | 0.0065 (10) |
| C9 | 0.0590 (16) | 0.0397 (12) | 0.0398 (13) | 0.0087 (10) | 0.0140 (11) | 0.0045 (10) |
| C10 | 0.0652 (17) | 0.0456 (13) | 0.0482 (15) | 0.0143 (12) | 0.0209 (13) | 0.0037 (11) |
| C11 | 0.089 (2) | 0.0725 (18) | 0.0698 (19) | 0.0249 (15) | 0.0379 (16) | −0.0036 (14) |
| C12 | 0.073 (2) | 0.0616 (16) | 0.0553 (16) | 0.0060 (13) | 0.0070 (14) | −0.0075 (13) |

*Geometric parameters (Å, º)*

| | | | |
|---|---|---|---|
| Br1—C4 | 1.899 (2) | C9—C10 | 1.409 (3) |
| O1—N2 | 1.420 (3) | C9—C12 | 1.486 (4) |
| O1—C8 | 1.361 (3) | C10—C11 | 1.500 (4) |
| N1—C7 | 1.274 (3) | C2—H2 | 0.9300 |
| N1—C8 | 1.374 (3) | C3—H3 | 0.9300 |
| N2—C10 | 1.307 (4) | C5—H5 | 0.9300 |
| C1—C2 | 1.387 (3) | C6—H6 | 0.9300 |
| C1—C6 | 1.390 (3) | C7—H7 | 0.9300 |
| C1—C7 | 1.460 (3) | C11—H11A | 0.9600 |
| C2—C3 | 1.384 (3) | C11—H11B | 0.9600 |
| C3—C4 | 1.378 (4) | C11—H11C | 0.9600 |
| C4—C5 | 1.363 (4) | C12—H12A | 0.9600 |
| C5—C6 | 1.382 (4) | C12—H12B | 0.9600 |
| C8—C9 | 1.351 (3) | C12—H12C | 0.9600 |
| | | | |
| Br1···C11[i] | 3.595 (3) | C4···C6[ii] | 3.553 (3) |
| Br1···C7[ii] | 3.687 (2) | C6···C4[ii] | 3.553 (3) |
| O1···C3[iii] | 3.355 (3) | C7···Br1[ii] | 3.687 (2) |
| O1···H3[iii] | 2.6900 | C7···C2[iv] | 3.481 (3) |
| O1···H7 | 2.3400 | C8···C3[iv] | 3.512 (3) |
| N1···C2[iv] | 3.426 (3) | C11···Br1[viii] | 3.595 (3) |
| N1···H2 | 2.6000 | H2···N1 | 2.6000 |
| N1···H12A | 2.7600 | H2···N2[vii] | 2.7400 |
| N1···H12C[v] | 2.7100 | H3···O1[vii] | 2.6900 |
| N2···H2[iii] | 2.7400 | H6···H7 | 2.4200 |
| N2···H11B[vi] | 2.9100 | H7···O1 | 2.3400 |
| C2···N1[iv] | 3.426 (3) | H7···H6 | 2.4200 |
| C2···C7[iv] | 3.481 (3) | H11B···N2[vi] | 2.9100 |
| C3···O1[vii] | 3.355 (3) | H12A···N1 | 2.7600 |
| C3···C8[iv] | 3.512 (3) | H12C···N1[v] | 2.7100 |

| | | | |
|---|---|---|---|
| N2—O1—C8 | 107.76 (18) | C1—C2—H2 | 120.00 |
| C7—N1—C8 | 119.9 (2) | C3—C2—H2 | 120.00 |
| O1—N2—C10 | 105.0 (2) | C2—C3—H3 | 121.00 |
| C2—C1—C6 | 118.9 (2) | C4—C3—H3 | 121.00 |
| C2—C1—C7 | 122.08 (18) | C4—C5—H5 | 120.00 |
| C6—C1—C7 | 119.0 (2) | C6—C5—H5 | 120.00 |
| C1—C2—C3 | 120.6 (2) | C1—C6—H6 | 120.00 |
| C2—C3—C4 | 118.8 (2) | C5—C6—H6 | 120.00 |
| Br1—C4—C3 | 119.17 (18) | N1—C7—H7 | 119.00 |
| Br1—C4—C5 | 119.02 (19) | C1—C7—H7 | 119.00 |
| C3—C4—C5 | 121.8 (2) | C10—C11—H11A | 109.00 |
| C4—C5—C6 | 119.2 (2) | C10—C11—H11B | 109.00 |
| C1—C6—C5 | 120.6 (2) | C10—C11—H11C | 109.00 |
| N1—C7—C1 | 122.0 (2) | H11A—C11—H11B | 109.00 |
| O1—C8—N1 | 120.5 (2) | H11A—C11—H11C | 109.00 |
| O1—C8—C9 | 110.4 (2) | H11B—C11—H11C | 109.00 |
| N1—C8—C9 | 129.2 (2) | C9—C12—H12A | 109.00 |
| C8—C9—C10 | 103.9 (2) | C9—C12—H12B | 109.00 |
| C8—C9—C12 | 127.6 (2) | C9—C12—H12C | 109.00 |
| C10—C9—C12 | 128.5 (2) | H12A—C12—H12B | 110.00 |
| N2—C10—C9 | 113.0 (2) | H12A—C12—H12C | 109.00 |
| N2—C10—C11 | 118.9 (2) | H12B—C12—H12C | 109.00 |
| C9—C10—C11 | 128.1 (2) | | |

| | | | |
|---|---|---|---|
| C8—O1—N2—C10 | −0.1 (3) | C1—C2—C3—C4 | 0.3 (4) |
| N2—O1—C8—C9 | 0.1 (2) | C2—C3—C4—Br1 | 179.81 (18) |
| N2—O1—C8—N1 | −178.1 (2) | C2—C3—C4—C5 | −0.3 (4) |
| C7—N1—C8—C9 | 177.3 (2) | Br1—C4—C5—C6 | 179.42 (19) |
| C8—N1—C7—C1 | 177.18 (18) | C3—C4—C5—C6 | −0.5 (4) |
| C7—N1—C8—O1 | −4.8 (3) | C4—C5—C6—C1 | 1.2 (4) |
| O1—N2—C10—C9 | 0.1 (3) | O1—C8—C9—C10 | 0.0 (3) |
| O1—N2—C10—C11 | 179.6 (2) | N1—C8—C9—C12 | −2.8 (4) |
| C2—C1—C6—C5 | −1.2 (3) | O1—C8—C9—C12 | 179.1 (2) |
| C6—C1—C2—C3 | 0.4 (3) | N1—C8—C9—C10 | 178.0 (2) |
| C7—C1—C2—C3 | −178.4 (2) | C8—C9—C10—N2 | −0.1 (3) |
| C7—C1—C6—C5 | 177.6 (2) | C12—C9—C10—C11 | 1.4 (4) |
| C2—C1—C7—N1 | 5.0 (3) | C8—C9—C10—C11 | −179.5 (3) |
| C6—C1—C7—N1 | −173.7 (2) | C12—C9—C10—N2 | −179.2 (2) |

Symmetry codes: (i) $x-1, y+1, z+1$; (ii) $-x, -y+2, -z+1$; (iii) $x+1, y, z$; (iv) $-x, -y+1, -z+1$; (v) $-x, -y+1, -z$; (vi) $-x+1, -y+1, -z$; (vii) $x-1, y, z$; (viii) $x+1, y-1, z-1$.

*Hydrogen-bond geometry (Å, °)*

| D—H···A | D—H | H···A | D···A | D—H···A |
|---|---|---|---|---|
| C7—H7···O1 | 0.9300 | 2.3400 | 2.702 (3) | 103.00 |