# organic compounds

# 3,5-Bis(4-methoxyphenyl)-4,5-dihydro-isoxazole

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In the title compound, $C_{17}H_{17}NO_3$, the five-membered isoxazoline ring adopts an envelope conformation with the chiral C atom at the flap position and 0.133 (2) Å out of the mean plane formed by the other four atoms. The two benzene rings form dihedral angles of 6.05 (5) and 81.52 (5)° with the C—C—N—O plane of the isoxazoline ring. The crystal structure is stabilized by weak C—H···O hydrogen bonds and C—H···$\pi$ interactions.

## Related literature

For medical uses of isoxazole derivatives, see: Sperry & Wright (2005). For their biological activity, see; Boyd (1991); Lang & Lin (1984). For related structures, see; Baktır *et al.* (2011*a,b*); Chopra *et al.* (2006); Dardouri *et al.* (2010); Fun *et al.* (2010*a,b*); Guo *et al.* (2006); Jasinski *et al.* (2010); Ko *et al.* (2011); Samshuddin *et al.* (2010). For ring puckering and asymmetry parameters, see: Cremer & Pople (1975); Nardelli (1983).
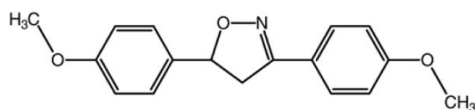


## Experimental

### Crystal data

| | |
|---|---|
| $C_{17}H_{17}NO_3$ | $V$ = 2883.9 (3) Å³ |
| $M_r$ = 283.32 | $Z$ = 8 |
| Orthorhombic, $Pbca$ | Mo $K\alpha$ radiation |
| $a$ = 10.5071 (7) Å | $\mu$ = 0.09 mm⁻¹ |
| $b$ = 8.4023 (5) Å | $T$ = 295 K |
| $c$ = 32.6662 (19) Å | 0.42 × 0.36 × 0.16 mm |

### Data collection

| | |
|---|---|
| Oxford Diffraction Xcalibur Ruby Gemini diffractometer | 13952 measured reflections |
| Absorption correction: multi-scan (*CrysAlis PRO*; Oxford Diffraction, 2007) | 4821 independent reflections |
| $T_{min}$ = 0.967, $T_{max}$ = 1.000 | 2738 reflections with $i > 2\sigma(i)$ |
| | $R_{int}$ = 0.046 |

### Refinement

| | |
|---|---|
| $R[F^2 > 2\sigma(F^2)]$ = 0.075 | 192 parameters |
| $wR(F^2)$ = 0.156 | H-atom parameters constrained |
| $S$ = 1.06 | $\Delta\rho_{max}$ = 0.23 e Å⁻³ |
| 4821 reflections | $\Delta\rho_{min}$ = −0.17 e Å⁻³ |

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

$Cg2$ and $Cg3$ are the centroids of the C1A–C6A and C1B–C6B benzene rings, respectively.

| $D$—H···$A$ | $D$—H | H···$A$ | $D$···$A$ | $D$—H···$A$ |
|---|---|---|---|---|
| C6A—H6AA···O1[i] | 0.93 | 2.52 | 3.324 (2) | 145 |
| C1—H1A···Cg3[ii] | 0.98 | 2.62 | 3.590 (2) | 170 |
| C6B—H6BA···Cg3[iii] | 0.93 | 3.00 | 3.724 (2) | 136 |
| C7B—H7BC···Cg2[iv] | 0.96 | 2.83 | 3.541 (3) | 132 |

Symmetry codes: (i) $-x+\frac{3}{2}, y+\frac{1}{2}, z$; (ii) $-x+1, -y+1, -z+1$; (iii) $-x+\frac{3}{2}, y-\frac{1}{2}, z$; (iv) $-x+1, -y+2, -z+1$.

Data collection: *CrysAlis PRO* (Oxford Diffraction, 2007); cell refinement: *CrysAlis PRO*; data reduction: *CrysAlis RED* (Oxford Diffraction, 2007); 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); software used to prepare material for publication: *WinGX* (Farrugia, 1999) and *PLATON* (Spek, 2009).

## References

Baktır, Z., Akkurt, M., Samshuddin, S., Narayana, B. & Yathirajan, H. S. (2011*a*). *Acta Cryst.* E**67**, o1262–o1263.
Baktır, Z., Akkurt, M., Samshuddin, S., Narayana, B. & Yathirajan, H. S. (2011*b*). *Acta Cryst.* E**67**, o1292–o1293.
Boyd, G. V. (1991). *Prog. Heterocyl. Chem.* **3**, 166–185.
Chopra, D., Mohan, T. P. & Vishalakshi, B. (2006). *Acta Cryst.* E**62**, o3547–o3548.
Cremer, D. & Pople, J. A. (1975). *J. Am. Chem. Soc.* **97**, 1354–1358.
Dardouri, R., Kandri Rodi, Y., Saffon, N., El Ammari, L. & Essassi, E. M. (2010). *Acta Cryst.* E**66**, o2983.
Farrugia, L. J. (1997). *J. Appl. Cryst.* **30**, 565.
Farrugia, L. J. (1999). *J. Appl. Cryst.* **32**, 837–838.
Fun, H.-K., Hemamalini, M., Samshuddin, S., Narayana, B. & Yathirajan, H. S. (2010*a*). *Acta Cryst.* E**66**, o582–o583.
Fun, H.-K., Hemamalini, M., Samshuddin, S., Narayana, B. & Yathirajan, H. S. (2010*b*). *Acta Cryst.* E**66**, o864–o865.
Guo, X.-H., Qi, X.-X. & Chang, J.-B. (2006). *Acta Cryst.* E**62**, o374–o375.
Jasinski, J. P., Guild, C. J., Samshuddin, S., Narayana, B. & Yathirajan, H. S. (2010). *Acta Cryst.* E**66**, o1948–o1949.

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Ko, Y. K., Ryu, J. W., Koo, D. W., Woo, J. C. & Kim, C.-H. (2011). *Acta Cryst.* E**67**, o1040.

Lang, A. & Lin, Y. (1984). *Comprehensive Heterocyclic Chemistry*, Vol. 6, edited by A. R. Katritzky, pp. 1–130. Oxford: Pergamon Press.

Nardelli, M. (1983). *Acta Cryst.* C**39**, 1141–1142.

Oxford Diffraction (2007). *CrysAlis PRO* and *CrysAlis RED*. Oxford Diffraction Ltd, Abingdon, England.

Samshuddin, S., Narayana, B., Yathirajan, H. S., Safwan, A. P. & Tiekink, E. R. T. (2010). *Acta Cryst.* E**66**, o1279–o1280.

Sheldrick, G. M. (2008). *Acta Cryst.* A**64**, 112–122.

Spek, A. L. (2009). *Acta Cryst.* D**65**, 148–155.

Sperry, J. & Wright, D. (2005). *Curr. Opin. Drug Discov. Dev.* **8**, 723–740.

**supplementary materials**

## 3,5-Bis(4-methoxyphenyl)-4,5-dihydroisoxazole

**S. Samshuddin, R. J. Butcher, M. Akkurt, B. Narayana** and **H. S. Yathirajan**

### Comment

Chalcones are convenient intermediates for the synthesis of various biodynamic cyclic derivatives such as isoxazoline, pyrazoline, benzodiazepine and cyclohexenone derivatives (Samshuddin *et al.*, 2010; Fun *et al.*, 2010*a,b*; Jasinski *et al.*, 2010; Baktır *et al.*, 2011*a,b*). Isoxazole derivatives represent a unique class of nitrogen- and oxygen-containing five-membered heterocycles. These are the components in a variety of natural products and medicinally useful compounds (Sperry *et al.*, 2005). Isoxazole derivatives bearing different substituents are known to have various biological activities in pharmaceutical and agricultural areas (Lang & Lin, 1984; Boyd, 1991).

The crystal structures of some 4,5-dihydroisoxazole derivatives *viz.*, 5-(4-fluoro-3-phenoxyphenyl)-3-(4-methylphenyl)-4,5-dihydroisoxazole (Chopra *et al.*, 2006), 5-(1*H*-indol-3-yl)-3-(4-methylphenyl)-4,5-dihydroisoxazoline (Guo *et al.*, 2006), 1,5-dimethyl-3-[(3-phenyl-4,5-dihydro-1,2-oxazol-5-yl)methyl]-1*H*-1,5- benzodiazepine-2,4(3*H*,5*H*)-dione (Dardouri *et al.*, 2010) and (*S*)-[5-methyl-3-(3-methylthiophen-2-yl)-4,5-dihydroisoxazol-5-yl] methanol solvate (Ko *et al.*, 2011) have been reported. In view of the importance of isoxazoles and in continuation of our work on synthesis of various derivatives of chalcones, the title compound (I) was prepared and its crystal structure is reported.

As shown in Fig. 1, the 4,5-dihydroisoxazole ring (O1/N1/C1–C3) of the title compound (I) has an envelope conformation with the chiral C1 atom at the flap position, displaced from the mean plane by -0.133 (2) Å; the puckering parameters (Cremer & Pople, 1975) are $Q(2) = 0.2156\ (17)$ Å, $\varphi(2) = 319.7\ (5)°$. Furthermore, one (O1B/C1B–C7B) of the methoxyphenyl ring systems makes a dihedral angle of 6.05 (5)°, whereas the other one (O1A/C1A–C7A) is almost orthogonal to the plane formed by the four atoms O1, N1, C2 and C3 of the five-membered isoxazoline ring, the dihedral angle being 81.52 (5)° (Nardelli, 1983). The dihedral angle between the methoxyphenyl ring systems (O1A/C1A–C7A and O1B/C1B–C7B) is 76.56 (4)°.

In the crystal structure, molecules are linked by weak C6A—H6AA···O1 (Table 1, Fig. 2) hydrogen bonds, and are further consolidated by C–H···π interactions (Table 1, Cg2 and Cg3 are the centroids of the C1A–C6A and C1B–C6B benzene rings, respectively).

### Experimental

A solution of (2*E*)-1,3-bis(4-methoxyphenyl)prop-2-en-1-one (2.68 g, 0.01 mol) and hydroxylamine hydrochloride (0.695 g, 0.01 mol) in 25 ml ethanol containing 3 ml of 10% sodium hydroxide solution was refluxed for 12 h. After cooling the mixture was poured into 50 ml ice-cold water. The resulting precipitate was collected by filtration and purified by recrystallization from ethanol. The single-crystal was grown from 2-propanol by slow evaporation of the solvent (yield: 59%; (m.p.: 407 K).

# supplementary materials

### Refinement

H atoms were located geometrically (aromatic C—H = 0.93 Å, methyl C—H = 0.96 Å, methylene C—H = 0.97 Å and methine C—H = 0.98 Å) and refined using the riding model approximation with fixed isotropic displacement parameters: $U_{iso}(H) = 1.5\ U_{eq}$(methyl-C) and $U_{iso}(H) = 1.2\ U_{eq}$(other C atoms).
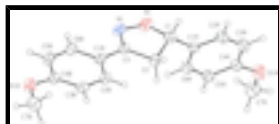
### Figures



Fig. 1. Molecular structure of the title compound with ellipsoids drawn at the 30% probability level and H atoms as spheres of arbitrary radius.
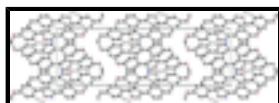


Fig. 2. Packing diagram of the title molecule, showing C—H···O interactions, viewed down the *b* axis. Hydrogen atoms not involved in H-bonding have been omitted for clarity.

### 3,5-Bis(4-methoxyphenyl)-4,5-dihydroisoxazole

*Crystal data*

| | |
|---|---|
| $C_{17}H_{17}NO_3$ | $F(000) = 1200$ |
| $M_r = 283.32$ | $D_x = 1.305$ Mg m$^{-3}$ |
| Orthorhombic, *Pbca* | Mo *K*α radiation, λ = 0.71073 Å |
| Hall symbol: -P 2ac 2ab | Cell parameters from 2593 reflections |
| $a = 10.5071\ (7)$ Å | θ = 5.2–32.6° |
| $b = 8.4023\ (5)$ Å | μ = 0.09 mm$^{-1}$ |
| $c = 32.6662\ (19)$ Å | $T = 295$ K |
| $V = 2883.9\ (3)$ Å$^3$ | Triangular plate, colourless |
| $Z = 8$ | 0.42 × 0.36 × 0.16 mm |

*Data collection*

| | |
|---|---|
| Oxford Diffraction Xcalibur Ruby Gemini diffractometer | 4821 independent reflections |
| Radiation source: Enhance (Mo) X-ray Source | 2738 reflections with i > 2σ(i) |
| graphite | $R_{int} = 0.046$ |
| Detector resolution: 10.5081 pixels mm$^{-1}$ | $\theta_{max} = 32.7°$, $\theta_{min} = 5.2°$ |
| ω scans | $h = -12 \rightarrow 15$ |
| Absorption correction: multi-scan (*CrysAlis PRO*; Oxford Diffraction, 2007) | $k = -10 \rightarrow 12$ |
| $T_{min} = 0.967$, $T_{max} = 1.000$ | $l = -29 \rightarrow 46$ |
| 13952 measured reflections | |

*Refinement*

| | |
|---|---|
| Refinement on $F^2$ | Primary atom site location: structure-invariant direct methods |
| Least-squares matrix: full | Secondary atom site location: difference Fourier map |
| $R[F^2 > 2\sigma(F^2)] = 0.075$ | Hydrogen site location: inferred from neighbouring sites |
| $wR(F^2) = 0.156$ | H-atom parameters constrained |
| $S = 1.06$ | $w = 1/[\sigma^2(F_o^2) + (0.0468P)^2 + 0.4739P]$ where $P = (F_o^2 + 2F_c^2)/3$ |
| 4821 reflections | $(\Delta/\sigma)_{max} = 0.001$ |
| 192 parameters | $\Delta\rho_{max} = 0.23$ e Å$^{-3}$ |
| 0 restraints | $\Delta\rho_{min} = -0.17$ 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 on $F^2$ for ALL reflections except those flagged by the user for potential systematic errors. Weighted $R$-factors $wR$ and all goodnesses 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 observed criterion of $F^2 > \sigma(F^2)$ is used only for calculating -$R$-factor-obs *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 | 0.70804 (13) | 0.51832 (16) | 0.43065 (4) | 0.0604 (5) |
| O1A | 0.63367 (15) | 0.77980 (18) | 0.25241 (4) | 0.0702 (5) |
| O1B | 0.62961 (12) | 0.83077 (16) | 0.65053 (4) | 0.0603 (5) |
| N1 | 0.72185 (15) | 0.54460 (19) | 0.47317 (4) | 0.0554 (5) |
| C1 | 0.58593 (17) | 0.5848 (2) | 0.41763 (5) | 0.0480 (6) |
| C1A | 0.59901 (16) | 0.6424 (2) | 0.37429 (5) | 0.0424 (5) |
| C1B | 0.63231 (14) | 0.69607 (18) | 0.52798 (5) | 0.0381 (5) |
| C2 | 0.55553 (17) | 0.7045 (2) | 0.45099 (5) | 0.0487 (6) |
| C2A | 0.51887 (17) | 0.5861 (2) | 0.34384 (6) | 0.0534 (6) |
| C2B | 0.53722 (15) | 0.7969 (2) | 0.54218 (5) | 0.0420 (5) |
| C3 | 0.63763 (14) | 0.64427 (19) | 0.48509 (5) | 0.0392 (5) |
| C3A | 0.53294 (18) | 0.6342 (2) | 0.30376 (6) | 0.0588 (7) |
| C3B | 0.53190 (15) | 0.8445 (2) | 0.58282 (5) | 0.0440 (5) |
| C4A | 0.62771 (17) | 0.7403 (2) | 0.29317 (5) | 0.0476 (6) |
| C4B | 0.62455 (15) | 0.7929 (2) | 0.60983 (5) | 0.0435 (5) |
| C5A | 0.70837 (17) | 0.7983 (2) | 0.32303 (5) | 0.0485 (6) |
| C5B | 0.71995 (16) | 0.6916 (2) | 0.59621 (5) | 0.0502 (6) |
| C6A | 0.69329 (16) | 0.7490 (2) | 0.36319 (5) | 0.0472 (5) |

| | | | |
|---|---|---|---|
| C6B | 0.72320 (15) | 0.6433 (2) | 0.55609 (5) | 0.0459 (6) |
| C7A | 0.7369 (2) | 0.8744 (3) | 0.23880 (6) | 0.0751 (9) |
| C7B | 0.53076 (19) | 0.9273 (3) | 0.66684 (6) | 0.0651 (8) |
| H2AA | 0.45450 | 0.51470 | 0.35060 | 0.0640* |
| H1A | 0.52180 | 0.50020 | 0.41830 | 0.0580* |
| H3AA | 0.47830 | 0.59510 | 0.28370 | 0.0710* |
| H2A | 0.57900 | 0.81170 | 0.44300 | 0.0580* |
| H2B | 0.46610 | 0.70220 | 0.45830 | 0.0580* |
| H5AA | 0.77240 | 0.87000 | 0.31620 | 0.0580* |
| H6AA | 0.74780 | 0.78840 | 0.38320 | 0.0570* |
| H7AA | 0.73380 | 0.88390 | 0.20950 | 0.1130* |
| H7AB | 0.81560 | 0.82520 | 0.24670 | 0.1130* |
| H7AC | 0.73120 | 0.97830 | 0.25090 | 0.1130* |
| H2BA | 0.47560 | 0.83330 | 0.52400 | 0.0500* |
| H3BA | 0.46660 | 0.91050 | 0.59180 | 0.0530* |
| H5BA | 0.78200 | 0.65630 | 0.61440 | 0.0600* |
| H6BA | 0.78700 | 0.57420 | 0.54750 | 0.0550* |
| H7BA | 0.54280 | 0.93930 | 0.69580 | 0.0980* |
| H7BB | 0.45000 | 0.87780 | 0.66170 | 0.0980* |
| H7BC | 0.53280 | 1.03010 | 0.65400 | 0.0980* |

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

| | $U^{11}$ | $U^{22}$ | $U^{33}$ | $U^{12}$ | $U^{13}$ | $U^{23}$ |
|---|---|---|---|---|---|---|
| O1 | 0.0765 (9) | 0.0589 (8) | 0.0457 (7) | 0.0266 (7) | 0.0012 (6) | −0.0037 (6) |
| O1A | 0.0859 (10) | 0.0801 (10) | 0.0445 (8) | −0.0125 (8) | −0.0025 (7) | 0.0053 (7) |
| O1B | 0.0668 (8) | 0.0680 (9) | 0.0461 (7) | 0.0106 (7) | −0.0018 (6) | −0.0049 (6) |
| N1 | 0.0640 (10) | 0.0565 (10) | 0.0458 (8) | 0.0209 (8) | 0.0000 (7) | 0.0006 (7) |
| C1 | 0.0512 (10) | 0.0445 (10) | 0.0484 (10) | −0.0028 (8) | −0.0005 (8) | 0.0025 (7) |
| C1A | 0.0459 (9) | 0.0381 (9) | 0.0433 (9) | 0.0013 (7) | 0.0011 (7) | −0.0018 (7) |
| C1B | 0.0353 (8) | 0.0358 (8) | 0.0431 (9) | −0.0013 (7) | 0.0019 (7) | 0.0057 (6) |
| C2 | 0.0467 (9) | 0.0551 (11) | 0.0442 (9) | 0.0109 (8) | 0.0026 (7) | 0.0033 (8) |
| C2A | 0.0503 (10) | 0.0533 (11) | 0.0565 (11) | −0.0148 (9) | −0.0028 (8) | 0.0009 (8) |
| C2B | 0.0387 (8) | 0.0410 (9) | 0.0463 (9) | 0.0046 (7) | −0.0007 (7) | 0.0084 (7) |
| C3 | 0.0371 (8) | 0.0354 (8) | 0.0452 (9) | −0.0005 (7) | 0.0028 (7) | 0.0051 (6) |
| C3A | 0.0607 (12) | 0.0630 (12) | 0.0528 (11) | −0.0120 (10) | −0.0144 (9) | −0.0034 (9) |
| C3B | 0.0428 (9) | 0.0387 (9) | 0.0504 (10) | 0.0047 (7) | 0.0067 (7) | 0.0044 (7) |
| C4A | 0.0534 (10) | 0.0480 (10) | 0.0413 (9) | 0.0038 (9) | 0.0011 (7) | −0.0012 (7) |
| C4B | 0.0462 (9) | 0.0427 (9) | 0.0417 (9) | −0.0033 (8) | 0.0020 (7) | 0.0031 (7) |
| C5A | 0.0493 (10) | 0.0458 (10) | 0.0505 (10) | −0.0062 (8) | 0.0029 (8) | 0.0004 (7) |
| C5B | 0.0439 (10) | 0.0568 (11) | 0.0500 (10) | 0.0083 (8) | −0.0081 (7) | 0.0044 (8) |
| C6A | 0.0482 (9) | 0.0480 (10) | 0.0454 (9) | −0.0055 (8) | −0.0050 (7) | −0.0055 (7) |
| C6B | 0.0384 (9) | 0.0483 (10) | 0.0511 (10) | 0.0087 (8) | 0.0017 (7) | 0.0024 (8) |
| C7A | 0.0860 (16) | 0.0809 (16) | 0.0584 (13) | −0.0076 (13) | 0.0103 (11) | 0.0144 (11) |
| C7B | 0.0696 (13) | 0.0732 (14) | 0.0524 (12) | 0.0015 (11) | 0.0105 (9) | −0.0100 (10) |

*Geometric parameters (Å, °)*

| | | | |
|---|---|---|---|
| O1—N1 | 1.4139 (19) | C4B—C5B | 1.388 (2) |

| | | | |
|---|---|---|---|
| O1—C1 | 1.463 (2) | C5A—C6A | 1.385 (2) |
| O1A—C4A | 1.374 (2) | C5B—C6B | 1.372 (2) |
| O1A—C7A | 1.416 (3) | C1—H1A | 0.9800 |
| O1B—C4B | 1.368 (2) | C2—H2A | 0.9700 |
| O1B—C7B | 1.421 (3) | C2—H2B | 0.9700 |
| N1—C3 | 1.279 (2) | C2A—H2AA | 0.9300 |
| C1—C1A | 1.503 (2) | C2B—H2BA | 0.9300 |
| C1—C2 | 1.517 (2) | C3A—H3AA | 0.9300 |
| C1A—C2A | 1.386 (2) | C3B—H3BA | 0.9300 |
| C1A—C6A | 1.384 (2) | C5A—H5AA | 0.9300 |
| C1B—C2B | 1.390 (2) | C5B—H5BA | 0.9300 |
| C1B—C3 | 1.468 (2) | C6A—H6AA | 0.9300 |
| C1B—C6B | 1.397 (2) | C6B—H6BA | 0.9300 |
| C2—C3 | 1.497 (2) | C7A—H7AA | 0.9600 |
| C2A—C3A | 1.378 (3) | C7A—H7AB | 0.9600 |
| C2B—C3B | 1.388 (2) | C7A—H7AC | 0.9600 |
| C3A—C4A | 1.381 (3) | C7B—H7BA | 0.9600 |
| C3B—C4B | 1.384 (2) | C7B—H7BB | 0.9600 |
| C4A—C5A | 1.381 (2) | C7B—H7BC | 0.9600 |
| | | | |
| N1—O1—C1 | 108.43 (13) | C1—C2—H2A | 112.00 |
| C4A—O1A—C7A | 118.35 (15) | C1—C2—H2B | 112.00 |
| C4B—O1B—C7B | 117.94 (14) | C3—C2—H2A | 112.00 |
| O1—N1—C3 | 109.29 (14) | C3—C2—H2B | 112.00 |
| O1—C1—C1A | 108.47 (14) | H2A—C2—H2B | 109.00 |
| O1—C1—C2 | 103.24 (13) | C1A—C2A—H2AA | 119.00 |
| C1A—C1—C2 | 118.86 (14) | C3A—C2A—H2AA | 119.00 |
| C1—C1A—C2A | 120.70 (15) | C1B—C2B—H2BA | 119.00 |
| C1—C1A—C6A | 121.39 (15) | C3B—C2B—H2BA | 119.00 |
| C2A—C1A—C6A | 117.88 (15) | C2A—C3A—H3AA | 120.00 |
| C2B—C1B—C3 | 121.78 (14) | C4A—C3A—H3AA | 120.00 |
| C2B—C1B—C6B | 117.75 (15) | C2B—C3B—H3BA | 120.00 |
| C3—C1B—C6B | 120.47 (14) | C4B—C3B—H3BA | 120.00 |
| C1—C2—C3 | 100.90 (13) | C4A—C5A—H5AA | 120.00 |
| C1A—C2A—C3A | 121.09 (16) | C6A—C5A—H5AA | 120.00 |
| C1B—C2B—C3B | 121.60 (15) | C4B—C5B—H5BA | 120.00 |
| N1—C3—C1B | 120.73 (14) | C6B—C5B—H5BA | 120.00 |
| N1—C3—C2 | 113.17 (14) | C1A—C6A—H6AA | 119.00 |
| C1B—C3—C2 | 126.01 (14) | C5A—C6A—H6AA | 119.00 |
| C2A—C3A—C4A | 120.32 (17) | C1B—C6B—H6BA | 120.00 |
| C2B—C3B—C4B | 119.44 (15) | C5B—C6B—H6BA | 119.00 |
| O1A—C4A—C3A | 115.58 (16) | O1A—C7A—H7AA | 109.00 |
| O1A—C4A—C5A | 124.85 (16) | O1A—C7A—H7AB | 109.00 |
| C3A—C4A—C5A | 119.57 (16) | O1A—C7A—H7AC | 109.00 |
| O1B—C4B—C3B | 125.04 (15) | H7AA—C7A—H7AB | 109.00 |
| O1B—C4B—C5B | 115.21 (14) | H7AA—C7A—H7AC | 109.00 |
| C3B—C4B—C5B | 119.72 (15) | H7AB—C7A—H7AC | 110.00 |
| C4A—C5A—C6A | 119.56 (16) | O1B—C7B—H7BA | 109.00 |
| C4B—C5B—C6B | 120.35 (15) | O1B—C7B—H7BB | 109.00 |
| C1A—C6A—C5A | 121.58 (16) | O1B—C7B—H7BC | 109.00 |

| | | | |
|---|---|---|---|
| C1B—C6B—C5B | 121.12 (15) | H7BA—C7B—H7BB | 110.00 |
| O1—C1—H1A | 109.00 | H7BA—C7B—H7BC | 109.00 |
| C1A—C1—H1A | 109.00 | H7BB—C7B—H7BC | 109.00 |
| C2—C1—H1A | 109.00 | | |
| C1—O1—N1—C3 | −12.91 (18) | C2B—C1B—C3—N1 | −176.15 (16) |
| N1—O1—C1—C1A | 148.33 (13) | C2B—C1B—C3—C2 | 7.6 (2) |
| N1—O1—C1—C2 | 21.37 (16) | C6B—C1B—C3—N1 | 3.2 (2) |
| C7A—O1A—C4A—C3A | −173.31 (17) | C6B—C1B—C3—C2 | −173.08 (15) |
| C7A—O1A—C4A—C5A | 6.5 (3) | C2B—C1B—C6B—C5B | −1.1 (2) |
| C7B—O1B—C4B—C3B | 1.7 (3) | C3—C1B—C6B—C5B | 179.54 (15) |
| C7B—O1B—C4B—C5B | −176.75 (16) | C1—C2—C3—N1 | 14.78 (19) |
| O1—N1—C3—C1B | −178.53 (14) | C1—C2—C3—C1B | −168.71 (15) |
| O1—N1—C3—C2 | −1.81 (19) | C1A—C2A—C3A—C4A | −0.1 (3) |
| O1—C1—C1A—C2A | 123.44 (17) | C1B—C2B—C3B—C4B | 1.2 (2) |
| O1—C1—C1A—C6A | −54.6 (2) | C2A—C3A—C4A—O1A | 179.75 (16) |
| C2—C1—C1A—C2A | −119.19 (18) | C2A—C3A—C4A—C5A | −0.1 (3) |
| C2—C1—C1A—C6A | 62.8 (2) | C2B—C3B—C4B—O1B | −179.72 (15) |
| O1—C1—C2—C3 | −20.68 (16) | C2B—C3B—C4B—C5B | −1.4 (2) |
| C1A—C1—C2—C3 | −140.76 (15) | O1A—C4A—C5A—C6A | −179.71 (16) |
| C1—C1A—C2A—C3A | −177.79 (16) | C3A—C4A—C5A—C6A | 0.1 (3) |
| C6A—C1A—C2A—C3A | 0.3 (3) | O1B—C4B—C5B—C6B | 178.89 (15) |
| C1—C1A—C6A—C5A | 177.80 (16) | C3B—C4B—C5B—C6B | 0.4 (3) |
| C2A—C1A—C6A—C5A | −0.2 (3) | C4A—C5A—C6A—C1A | 0.0 (3) |
| C3—C1B—C2B—C3B | 179.44 (15) | C4B—C5B—C6B—C1B | 0.9 (3) |
| C6B—C1B—C2B—C3B | 0.1 (2) | | |

*Hydrogen-bond geometry (Å, °)*

Cg2 and Cg3 are the centroids of the C1A–C6A and C1B–C6B benzene rings, respectively.

| D—H⋯A | D—H | H⋯A | D⋯A | D—H⋯A |
|---|---|---|---|---|
| C6A—H6AA⋯O1[i] | 0.93 | 2.52 | 3.324 (2) | 145 |
| C1—H1A⋯Cg3[ii] | 0.98 | 2.62 | 3.590 (2) | 170 |
| C6B—H6BA⋯Cg3[iii] | 0.93 | 3.00 | 3.724 (2) | 136 |
| C7B—H7BC⋯Cg2[iv] | 0.96 | 2.83 | 3.541 (3) | 132 |

Symmetry codes: (i) −x+3/2, y+1/2, z; (ii) −x+1, −y+1, −z+1; (iii) −x+3/2, y−1/2, z; (iv) −x+1, −y+2, −z+1.
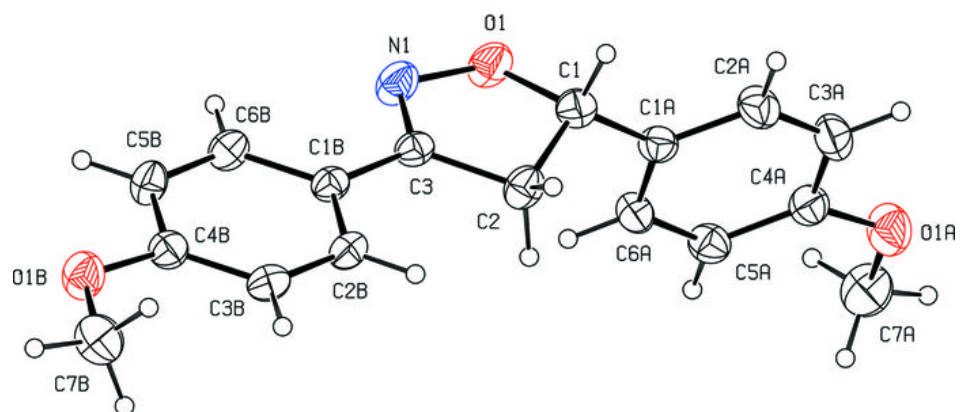
**Fig. 1**

**Fig. 2**