Acta Crystallographica Section E
**Structure Reports Online**
ISSN 1600-5368

# 5-Methoxy-1*H*-benzo[*d*]imidazole-2(3*H*)-thione

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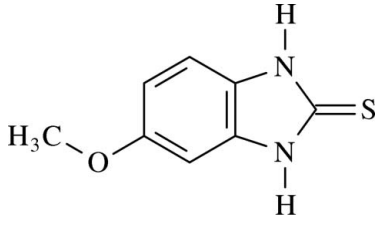
Received 4 June 2007; accepted 6 June 2007

Key indicators: single-crystal X-ray study; $T$ = 296 K; mean $\sigma$(C–C) = 0.003 Å; $R$ factor = 0.039; $wR$ factor = 0.106; data-to-parameter ratio = 11.8.

The title compound, $C_8H_8N_2OS$, is stabilized by intermolecular N—H···O and N—H···S hydrogen bonds and by $\pi$–$\pi$ interactions. The hydrogen bonds generate a two-dimensional network with edge-fused centrosymmetric $[R_2^2(8)R_4^4(20)R_2^2(8)]$ motifs, and these networks are connected by the $\pi$–$\pi$ interactions. These $\pi$–$\pi$ interactions occur between the homoaromatic rings of the molecules at $(x, y, z)$ and $(1-x, 1-y, 1-z)$; the centroid–centroid distance is 3.658 (1) Å and the plane–plane separation is 3.321 Å. The molecule is approximately planar, with a dihedral angle of 1.58 (13)° between the two rings.

## Related literature

For related structures, see: Ravikumar *et al.* (1995); Elerman & Kabak (1997); Swamy & Ravikumar (2005); Jian *et al.* (2006); Navarrete-Vázquez *et al.* (2006). For related literature, see: Bell *et al.* (1993); Skalitzky *et al.* (2003); Lalezari *et al.* (2002); Singh & Dash (1988); Sakemi *et al.* (2002); Wang (2001); Etter (1990).



## Experimental

### Crystal data

$C_8H_8N_2OS$
$M_r$ = 180.22
Triclinic, $P\overline{1}$
$a$ = 7.4922 (8) Å
$b$ = 7.6532 (8) Å
$c$ = 8.8403 (9) Å
$\alpha$ = 90.316 (8)°
$\beta$ = 114.148 (8)°
$\gamma$ = 118.516 (7)°
$V$ = 393.47 (9) Å$^3$
$Z$ = 2
Mo $K\alpha$ radiation
$\mu$ = 0.36 mm$^{-1}$
$T$ = 296 K
0.56 × 0.42 × 0.27 mm

### Data collection

Stoe IPDSII diffractometer
Absorption correction: integration (*X-RED32*; Stoe & Cie, 2002)
$T_{min}$ = 0.829, $T_{max}$ = 0.935
8166 measured reflections
1545 independent reflections
1445 reflections with $I > 2\sigma(I)$
$R_{int}$ = 0.084

### Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.039
$wR(F^2)$ = 0.106
$S$ = 1.08
1545 reflections
131 parameters
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.33 e Å$^{-3}$
$\Delta\rho_{min}$ = −0.38 e Å$^{-3}$

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

| $D$—H···$A$ | $D$—H | H···$A$ | $D$···$A$ | $D$—H···$A$ |
|---|---|---|---|---|
| N1—H1···S1$^{i}$ | 0.86 (3) | 2.43 (3) | 3.2853 (16) | 169 (2) |
| N2—H2···O1$^{ii}$ | 0.87 (3) | 2.15 (3) | 2.997 (2) | 165 (2) |

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

Data collection: *X-AREA* (Stoe & Cie, 2002); cell refinement: *X-AREA*; data reduction: *X-RED32* (Stoe & Cie, 2002); program(s) used to solve structure: *SHELXS97* (Sheldrick, 1990); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *ORTEP-3 for Windows* (Farrugia, 1997); software used to prepare material for publication: *WinGX* (Farrugia, 1999).

The authors acknowledge the Faculty of Arts and Sciences, Ondokuz Mayıs University, Turkey, for the use of the Stoe IPDS II diffractometer (purchased under grant F.279 of the University Research Fund). BN thanks Mangalore University for research facilities

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: AT2322).

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# supplementary materials

*Acta Cryst.* (2007). E**63**, o3199-o3200 [ **doi:10.1107/S1600536807027730** ]

# 5-Methoxy-1*H*-benzo[*d*]imidazole-2(3*H*)-thione

**M. Odabasoglu, O. Büyükgüngör, B. Narayana, A. M. Vijesh and H. S. Yathirajan**

## Comment

Benzimidazole derivatives are inhibitors of cyclin-dependent kinase and useful for inhibiting cell proliferation, in for the treatment of cancer and bis-benzimidazoles have potent activity against a number of microorganisms including those that lead to AIDS-related infections. These compounds bind to DNA in AT-rich sequences. Recently, benzimidazole derived drugs have received much attention owing to the fact that benzimidazole residue is a constituent of vitamin B12 which supports their potential use as therapeutics. The derivatives are also well known antioxidants used in the manufacture of rubber and anticorrosive agents for mild steel. In view of the importance of the title compound, (I), $C_8H_8N_2OS$, a crystal structure is reported (Fig. 1).

Compund (I) displays two moderate intermolecular hydrogen bond (Table 1) involving atoms O, S and N. In (I), the molecules are linked through an N—H···O and an N—H···S intramolecular hydrogen bonds and these hydrogen bonds generate edge-fussed centrosymmetric [$R_2^2(8)$ $R_4^4(20)R_2^2(8)$] ring motifs (Fig.2) (Etter, 1990) linked also by π···π interactions.

The intermolecular π···π interactions combine to stabilize the extended structure (Fig. 2). These π···π interactions occur between the C2—C7 rings of the molecules at ($x$, $y$, $z$) and ($1 - x$, $1 - y$, $1 - z$), with a centroid-to-centroid distance of 3.658 (1) Å and a plane-to-plane separation of 3.321 Å.

## Experimental

A pure sample of the compound was obtained from Strides Arco Labs, Mangalore, India and crystallized from DMF (m.p. 528–530 K).

## Refinement

All H atoms except the methyl's were located in difference Fourier map and refined freely. The H atoms of methyl were positioned at their positions [C—H = 0.96 Å, $U_{iso}$(H) = 1.5$U_{eq}$(C)] and allowed to ride and to rotate as well.

## Figures

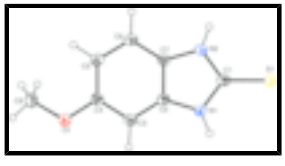

Fig. 1. A view of (I) showing the atomic numbering scheme with displacement ellipsoids drawn at the 30% probability level.

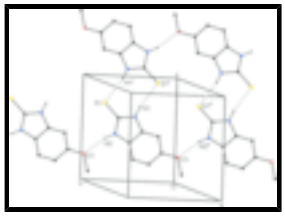

Fig. 2. Part of the crystal structure of (I). For the sake of clarity, H atoms not involved in the hydrogen bonding motifs shown have been omitted; hydrogen bonds are drawn as dashed lines. [Symmetry codes: (i) 2 − *x*, 2 − *y*, 2 − *z*; (ii) *x*, 1 − *y*, *z*; (iii) *x*, 1 + *y*, *z*].

**5-Methoxy-1*H*-benzo[*d*]imidazole-2(3*H*)-thione**

*Crystal data*

| | |
|---|---|
| $C_8H_8N_2OS$ | $Z = 2$ |
| $M_r = 180.22$ | $F_{000} = 188$ |
| Triclinic, $P\overline{1}$ | $D_x = 1.521$ Mg m$^{-3}$ |
| Hall symbol: -P 1 | Mo $K\alpha$ radiation<br>$\lambda = 0.71073$ Å |
| $a = 7.4922$ (8) Å | Cell parameters from 8166 reflections |
| $b = 7.6532$ (8) Å | $\theta = 3.1$–$28.8$º |
| $c = 8.8403$ (9) Å | $\mu = 0.36$ mm$^{-1}$ |
| $\alpha = 90.316$ (8)º | $T = 296$ K |
| $\beta = 114.148$ (8)º | Prism, colourless |
| $\gamma = 118.516$ (7)º | 0.56 × 0.42 × 0.27 mm |
| $V = 393.47$ (9) Å$^3$ | |

*Data collection*

| | |
|---|---|
| Stoe IPDS II diffractometer | 1545 independent reflections |
| Radiation source: sealed X-ray tube, 12 x 0.4 mm long-fine focus | 1445 reflections with $I > 2\sigma(I)$ |
| Monochromator: plane graphite | $R_{int} = 0.084$ |
| Detector resolution: 6.67 pixels mm$^{-1}$ | $\theta_{max} = 26.0$º |
| $T = 296$ K | $\theta_{min} = 3.1$º |
| ω scans | $h = -9 \rightarrow 9$ |
| Absorption correction: integration (X-RED32; Stoe & Cie, 2002) | $k = -9 \rightarrow 9$ |
| $T_{min} = 0.829$, $T_{max} = 0.935$ | $l = -10 \rightarrow 10$ |
| 8166 measured reflections | |

*Refinement*

| | |
|---|---|
| Refinement on $F^2$ | Hydrogen site location: inferred from neighbouring sites |
| Least-squares matrix: full | H atoms treated by a mixture of independent and constrained refinement |
| $R[F^2 > 2\sigma(F^2)] = 0.039$ | $w = 1/[\sigma^2(F_o^2) + (0.0648P)^2 + 0.0705P]$ where $P = (F_o^2 + 2F_c^2)/3$ |
| $wR(F^2) = 0.106$ | $(\Delta/\sigma)_{max} < 0.001$ |

| | |
|---|---|
| $S$ = 1.08 | $\Delta\rho_{max}$ = 0.33 e Å$^{-3}$ |
| 1545 reflections | $\Delta\rho_{min}$ = −0.38 e Å$^{-3}$ |
| 131 parameters | Extinction correction: SHELXL97 (Sheldrick, 1997), $Fc^*=kFc[1+0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$ |
| Primary atom site location: structure-invariant direct methods | Extinction coefficient: 0.081 (14) |
| Secondary atom site location: difference Fourier map | |

*Special details*

**Geometry**. All e.s.d.'s (except the e.s.d. in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell e.s.d.'s are taken into account individually in the estimation of e.s.d.'s in distances, angles and torsion angles; correlations between e.s.d.'s in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell e.s.d.'s is used for estimating e.s.d.'s involving l.s. planes.

**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 $F^2 > 2\text{sigma}(F^2)$ 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* | $U_{iso}$*/$U_{eq}$ |
|---|---|---|---|---|
| C1 | 0.8686 (3) | 1.0602 (3) | 0.7350 (2) | 0.0330 (4) |
| C2 | 0.8064 (3) | 0.7699 (2) | 0.6012 (2) | 0.0296 (4) |
| C3 | 0.7925 (3) | 0.5877 (2) | 0.5602 (2) | 0.0319 (4) |
| C4 | 0.6897 (3) | 0.4987 (2) | 0.3873 (2) | 0.0317 (4) |
| C5 | 0.6006 (3) | 0.5849 (3) | 0.2616 (2) | 0.0374 (4) |
| C6 | 0.6166 (3) | 0.7675 (3) | 0.3053 (2) | 0.0383 (4) |
| C7 | 0.7245 (3) | 0.8611 (3) | 0.4768 (2) | 0.0314 (4) |
| C8 | 0.6164 (4) | 0.2430 (3) | 0.1758 (3) | 0.0476 (5) |
| H8A | 0.4554 | 0.1883 | 0.1039 | 0.071* |
| H8B | 0.6494 | 0.1371 | 0.1699 | 0.071* |
| H8C | 0.7041 | 0.3540 | 0.1374 | 0.071* |
| N1 | 0.8937 (2) | 0.8967 (2) | 0.75761 (19) | 0.0330 (3) |
| N2 | 0.7669 (3) | 1.0393 (2) | 0.56429 (19) | 0.0346 (3) |
| O1 | 0.6761 (2) | 0.3167 (2) | 0.34818 (17) | 0.0419 (3) |
| S1 | 0.94575 (8) | 1.25049 (7) | 0.88771 (6) | 0.0405 (2) |
| H1 | 0.943 (4) | 0.875 (3) | 0.858 (3) | 0.043 (6)* |
| H2 | 0.742 (4) | 1.131 (4) | 0.519 (3) | 0.045 (6)* |
| H3 | 0.848 (4) | 0.520 (3) | 0.643 (3) | 0.041 (5)* |
| H5 | 0.518 (4) | 0.516 (4) | 0.137 (3) | 0.050 (6)* |
| H6 | 0.551 (4) | 0.831 (3) | 0.219 (3) | 0.041 (5)* |

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

| | $U^{11}$ | $U^{22}$ | $U^{33}$ | $U^{12}$ | $U^{13}$ | $U^{23}$ |
|---|---|---|---|---|---|---|
| C1 | 0.0352 (8) | 0.0329 (8) | 0.0326 (9) | 0.0182 (7) | 0.0169 (7) | 0.0127 (7) |

| | | | | | | |
|---|---|---|---|---|---|---|
| C2 | 0.0306 (7) | 0.0308 (8) | 0.0283 (8) | 0.0157 (6) | 0.0150 (6) | 0.0114 (6) |
| C3 | 0.0352 (8) | 0.0313 (8) | 0.0319 (9) | 0.0191 (7) | 0.0160 (7) | 0.0129 (7) |
| C4 | 0.0351 (8) | 0.0286 (8) | 0.0353 (9) | 0.0168 (6) | 0.0196 (7) | 0.0114 (7) |
| C5 | 0.0456 (9) | 0.0390 (9) | 0.0286 (9) | 0.0233 (8) | 0.0170 (7) | 0.0110 (7) |
| C6 | 0.0480 (9) | 0.0415 (9) | 0.0311 (9) | 0.0280 (8) | 0.0176 (8) | 0.0168 (7) |
| C7 | 0.0351 (8) | 0.0316 (8) | 0.0323 (8) | 0.0192 (6) | 0.0177 (7) | 0.0128 (7) |
| C8 | 0.0669 (12) | 0.0356 (10) | 0.0411 (10) | 0.0250 (9) | 0.0285 (10) | 0.0086 (8) |
| N1 | 0.0399 (7) | 0.0325 (7) | 0.0272 (7) | 0.0205 (6) | 0.0144 (6) | 0.0113 (6) |
| N2 | 0.0445 (8) | 0.0327 (7) | 0.0319 (8) | 0.0239 (6) | 0.0180 (6) | 0.0134 (6) |
| O1 | 0.0605 (8) | 0.0363 (7) | 0.0368 (7) | 0.0296 (6) | 0.0244 (6) | 0.0137 (6) |
| S1 | 0.0559 (3) | 0.0359 (3) | 0.0343 (3) | 0.0271 (2) | 0.0215 (2) | 0.0113 (2) |

*Geometric parameters (Å, °)*

| | | | |
|---|---|---|---|
| C1—N2 | 1.350 (2) | C5—H5 | 1.00 (3) |
| C1—N1 | 1.356 (2) | C6—C7 | 1.378 (3) |
| C1—S1 | 1.6775 (18) | C6—H6 | 0.99 (2) |
| C2—C3 | 1.382 (2) | C7—N2 | 1.390 (2) |
| C2—N1 | 1.386 (2) | C8—O1 | 1.426 (2) |
| C2—C7 | 1.392 (2) | C8—H8A | 0.9600 |
| C3—C4 | 1.385 (2) | C8—H8B | 0.9600 |
| C3—H3 | 0.97 (2) | C8—H8C | 0.9600 |
| C4—O1 | 1.379 (2) | N1—H1 | 0.86 (3) |
| C4—C5 | 1.392 (2) | N2—H2 | 0.87 (3) |
| C5—C6 | 1.383 (3) | | |
| N2—C1—N1 | 106.42 (15) | C5—C6—H6 | 122.9 (13) |
| N2—C1—S1 | 126.47 (14) | C6—C7—N2 | 132.91 (16) |
| N1—C1—S1 | 127.11 (14) | C6—C7—C2 | 120.72 (16) |
| C3—C2—N1 | 131.56 (15) | N2—C7—C2 | 106.30 (15) |
| C3—C2—C7 | 122.33 (16) | O1—C8—H8A | 109.5 |
| N1—C2—C7 | 106.11 (14) | O1—C8—H8B | 109.5 |
| C2—C3—C4 | 116.21 (15) | H8A—C8—H8B | 109.5 |
| C2—C3—H3 | 124.8 (13) | O1—C8—H8C | 109.5 |
| C4—C3—H3 | 119.0 (13) | H8A—C8—H8C | 109.5 |
| O1—C4—C3 | 115.58 (15) | H8B—C8—H8C | 109.5 |
| O1—C4—C5 | 122.35 (16) | C1—N1—C2 | 110.65 (14) |
| C3—C4—C5 | 122.04 (16) | C1—N1—H1 | 120.8 (15) |
| C6—C5—C4 | 120.86 (16) | C2—N1—H1 | 128.1 (15) |
| C6—C5—H5 | 117.2 (14) | C1—N2—C7 | 110.51 (15) |
| C4—C5—H5 | 121.9 (15) | C1—N2—H2 | 123.3 (17) |
| C7—C6—C5 | 117.78 (16) | C7—N2—H2 | 126.0 (17) |
| C7—C6—H6 | 119.3 (13) | C4—O1—C8 | 117.01 (15) |
| N1—C2—C3—C4 | 179.05 (15) | N1—C2—C7—N2 | 0.19 (16) |
| C7—C2—C3—C4 | −0.7 (2) | N2—C1—N1—C2 | −0.44 (18) |
| C2—C3—C4—O1 | −179.72 (13) | S1—C1—N1—C2 | 179.04 (12) |
| C2—C3—C4—C5 | −1.4 (2) | C3—C2—N1—C1 | −179.58 (16) |
| O1—C4—C5—C6 | 179.81 (16) | C7—C2—N1—C1 | 0.16 (17) |
| C3—C4—C5—C6 | 1.6 (3) | N1—C1—N2—C7 | 0.56 (19) |
| C4—C5—C6—C7 | 0.3 (3) | S1—C1—N2—C7 | −178.92 (12) |

| | | | |
|---|---|---|---|
| C5—C6—C7—N2 | −178.88 (17) | C6—C7—N2—C1 | 176.43 (18) |
| C5—C6—C7—C2 | −2.3 (3) | C2—C7—N2—C1 | −0.47 (18) |
| C3—C2—C7—C6 | 2.6 (2) | C3—C4—O1—C8 | −167.30 (16) |
| N1—C2—C7—C6 | −177.18 (15) | C5—C4—O1—C8 | 14.4 (2) |
| C3—C2—C7—N2 | 179.95 (14) | | |

*Hydrogen-bond geometry (Å, °)*

| *D*—H···*A* | *D*—H | H···*A* | *D*···*A* | *D*—H···*A* |
|---|---|---|---|---|
| N1—H1···S1$^{i}$ | 0.86 (3) | 2.43 (3) | 3.2853 (16) | 169 (2) |
| N2—H2···O1$^{ii}$ | 0.87 (3) | 2.15 (3) | 2.997 (2) | 165 (2) |

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

**Fig. 1**

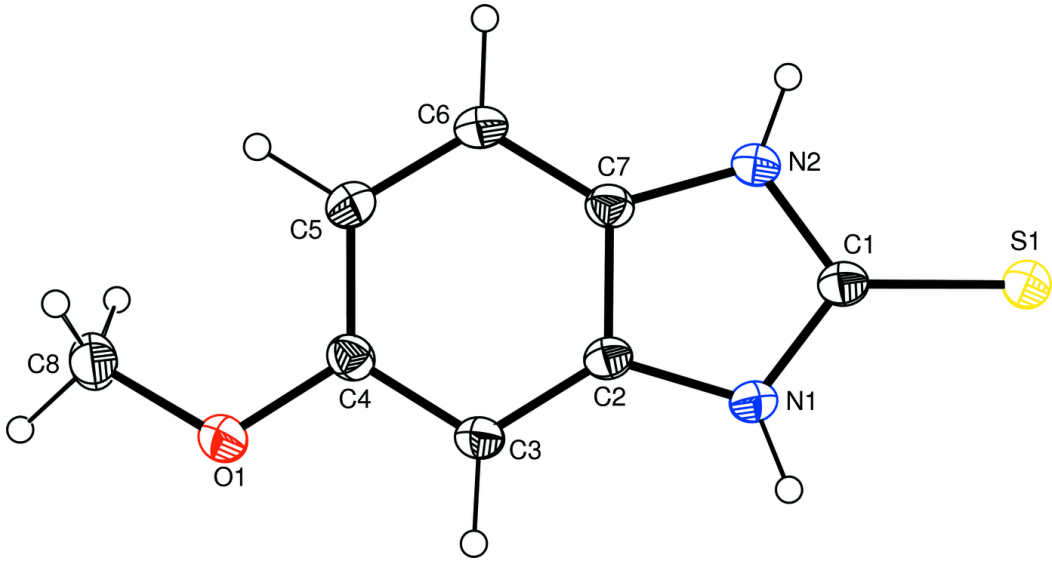

**Fig. 2**