Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

Methyl (1*H*-benzimidazol-2-ylsulfanyl)-acetate

Yvon Bibila Mayaya Bisseyou,[a]* A. Adohi-Krou,[a] Roger S. P. Zoakouma,[b] R. C. A. Yao-Kakou[a] and N'dédé Ebby[a]

[a]Laboratoire de Cristallographie et Physique Moléculaire, UFR SSMT Université de Cocody, 22 BP 582 Abidjan 22, Cote d'Ivoire, and [b]Laboratoire de Chimie Organique Structurale, UFR SSMT Université de Cocody, 22 BP 582 Abidjan 22, Cote d'Ivoire
Correspondence e-mail: bibilamayayabisseyou@yahoo.fr

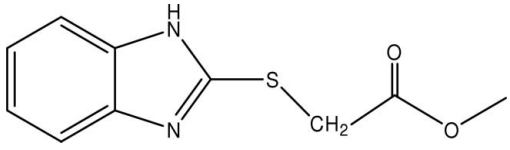
Received 29 June 2007; accepted 30 August 2007

Key indicators: single-crystal X-ray study; T = 295 K; mean σ(C–C) = 0.004 Å; R factor = 0.037; wR factor = 0.062; data-to-parameter ratio = 17.8.

In the molecule of the title compound, $C_{10}H_{10}N_2O_2S$, the benzimidazole ring system and the plane through the non-H atoms of the methyl mercaptoacetate group form a dihedral angle of 81.75 (1)°. The crystal structure is stabilized by N—H···N hydrogen bonds, giving infinite chains along the [001] direction.

Related literature

The crystal structures of some related 2-substituted benzimidazole derivatives have previously been reported (Langer *et al.*, 2006; Eltayeb *et al.*, 2007; Jian *et al.*, 2007; Yıldırım *et al.*, 2007). For related literature see: Tebbe *et al.* (1997); Tomio *et al.* (2006). For the refinement weighting scheme, see: Watkin (1994); Prince (1982).



Experimental

Crystal data

$C_{10}H_{10}N_2O_2S$
M_r = 222.27
Monoclinic, *Cc*
a = 9.337 (9) Å
b = 13.418 (15) Å
c = 9.667 (8) Å
β = 116.04 (7)°
V = 1088 (2) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.28 mm^{-1}
T = 295 K
0.40 × 0.20 × 0.15 mm

Data collection

Nonius KappaCCD diffractometer
Absorption correction: multi-scan (*SORTAV*; Blessing, 1995)
T_{min} = 0.88, T_{max} = 0.96
10772 measured reflections
2433 independent reflections
2248 reflections with $I > 2\sigma(I)$
R_{int} = 0.037

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.037
$wR(F^2)$ = 0.062
S = 0.92
2433 reflections
137 parameters
2 restraints
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.15 e Å^{-3}
$\Delta\rho_{min}$ = −0.13 e Å^{-3}
Absolute structure: Flack (1983), with 1156 Friedel pairs
Flack parameter: −0.01 (7)

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

D—H···A	D—H	H···A	D···A	D—H···A
N1—H1···N2^{i}	0.85	2.07	2.879 (2)	159

Symmetry code: (i) $x, -y, z+\frac{1}{2}$.

Data collection: *COLLECT* (Nonius, 2001); cell refinement: *DENZO* (Otwinowski & Minor, 1997) and *SORTAV* (Blessing, 1995); data reduction: *DENZO* and *SORTAV*; program(s) used to solve structure: *SIR92* (Altomare *et al.*, 1994); program(s) used to refine structure: *CRYSTALS* (Betteridge *et al.*, 2003); molecular graphics: *ORTEP3* (Farrugia, 1997) and *PLATON* (Spek, 2003); software used to prepare material for publication: *CRYSTALS*.

The authors thank the Laboratoire de Cristallographie et Modélization des Matériaux Minéraux et Biologiques of Henri Poincaré University, France, for the use of their Bruker Nonius KappaCCD area-detector diffractometer.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: CF2115).

References

Altomare, A., Cascarano, G., Giacovazzo, G., Guagliardi, A., Burla, M. C., Polidori, G. & Camalli, M. (1994). *J. Appl. Cryst.* **27**, 435.
Betteridge, P. W., Carruthers, J. R., Cooper, R. I., Prout, K. & Watkin, D. J. (2003). *J. Appl. Cryst.* **36**, 1487.
Blessing, R. H. (1995). *Acta Cryst.* A**51**, 33–38.
Eltayeb, N. E., Teoh, S. G., Teh, J. B.-J., Fun, H.-K. & Ibrahim, K. (2007). *Acta Cryst.* E**63**, o300–o302.
Farrugia, L. J. (1997). *J. Appl. Cryst.* **30**, 565.
Flack, H. D. (1983). *Acta Cryst.* A**39**, 876–881.
Jian, F.-F., Yu, H.-Q., Qiao, Y.-B., Liang, T.-L. & Zhao, P.-S. (2007). *Acta Cryst.* E**63**, o321–o322.
Langer, V., Mičová, J., Steiner, B. & Koóš, M. (2006). *Acta Cryst.* E**62**, o2138–o2140.
Nonius (2001). *COLLECT*. Nonius BV, Delft, The Netherlands.
Otwinowski, Z. & Minor, W. (1997). *Methods in Enzymology*, Vol. 276, *Macromolecular Crystallography*, Part A, edited by C. W. Carter Jr & R. M. Sweet, pp. 307–326. New York: Academic Press.
Prince, E. (1982). *Mathematical Techniques in Crystallography and Materials Science*. New York: Springer-Verlag.
Spek, A. L. (2003). *J. Appl. Cryst.* **36**, 7–13.
Tebbe, M. J., Spitzer, W. A., Victor, F., Miller, S. C., Lee, C. C., Sattelberg, T. R., Mckinney, E. & Tang, C. J. (1997). *J. Med. Chem.* **40**, 3937–3946.
Tomio, I., Takayoshi, S., Shintaro, H., Kenji, M., Atsuhito, Y., Izuru, A., Satoru, I., Tsuyoshi, A. & Hiromasa, H. (2006). *Bioorg. Med. Chem. Lett.* **16**, 1859–1863.
Watkin, D. (1994). *Acta Cryst.* A**50**, 411–437.
Yıldırım, S. Ö., Akkurt, M., Şireci, N., Küçükbay, H. & Kazak, C. (2007). *Acta Cryst.* E**63**, o2433.

 doi:10.1107/S1600536807042572

supplementary materials

Acta Cryst. (2007). E**63**, o3987 [**doi:10.1107/S1600536807042572**]

Methyl (1*H*-benzimidazol-2-ylsulfanyl)acetate

Y. Bibila Mayaya Bisseyou, A. Adohi-Krou, R. S. P. Zoakouma, R. C. A. Yao-Kakou and N. Ebby

Comment

The synthesis of new benzimidazole derivatives presents is of considerable interest in pharmacology because of their therapeutic benefits in many diseases. Several studies showed that benzimidazole derivatives possess versatile pharmacological properties, such as anthelmintic, fungicidal, antitumour, analgesic and antiviral activities. It was reported that benzimidazole ring systems are an important pharmacophore in the discovery and design of new drugs (Tebbe *et al.*, 1997). Furthermore, a recent study showed that 2-[(4-diarylmethoxy)phenyl]-benzimidazoles are potent inhibitors of the hepatitis C virus NS5B polymerase (Tomio *et al.*, 2006). The title compound (I) was synthesized and we have determined its crystal structure in conjunction with new investigations of 2-substituted benzimidazole derivative properties. The molecular structure of (I) and the atomic numbering scheme are shown in Fig. 1. The results are comparable to those obtained in recent studies related to 2-substitued benzimidazole derivatives (Langer *et al.*, 2006; Eltayeb *et al.*, 2007; Jian *et al.*, 2007; Yıldırım *et al.*, 2007). The benzimidazole ring system is essentially planar, with a maximum deviation of 0.051 (1) Å for atom C1. The dihedral angle between the benzimidazole ring system and the plane through S1/C8/C9/O1/O2/C10 is 81.75 (1)°. The crystal packing is stabilized by an intermolecular N—H···N hydrogen bond which leads to the formation of infinite molecular chains along the [001] direction (Fig. 2).

Experimental

2.25 ml of triethylamine (15.98 mmol, 1.2 eq) and 1.4 ml of methyl bromoacetate (14.65 mmol, 1.1 eq) were added to 2-mercaptobenzimidazole (13.32 mmol, 2 g) in 10 ml of anhydrous ethanol. The mixture was stirred for half an hour at ambient temperature, then was refluxed for 3 h. The solvent was removed and 20 ml of water was added to the residue. The organic residues were collected, then they were dried, filtered and concentrated with a rotary evaporator. The addition of hexane to the substrate led to a precipitate which was recrystallized from a mixture of dichloromethane/hexane 20/80 to obtain white crystals of the title compound with a yield of 75%; m.p. 393 K. ^{1}H-NMR (DMSO-d_6, 300 MHz), δ(p.p.m.): 3.66 (s, 3H, OCH_3), 4.24 (s, 2H, CH_2), 7.1O-7.51 (m, 4H, C_6H_4), 12.63 (1 s, 1H, NH). ^{13}C-NMR (DMSO-d_6, 300 MHz), δ(p.p.m.): 33.98 (CH_2), 52.89 (OCH_3), 113.35, 114.53, 124.59, 126.13, 126.68, 133.72 (C_6H_4), 149.51 (C═N), 168.15 (C═O).

Refinement

H atoms were located in a difference Fourier map, but those attached to C10 were geometrically repositioned. They were all initially refined with soft restraints on the bond lengths and angles to regularize their geometry (C—H in the range 0.90–0.98, N—H 0.85 Å and U_{iso}(H) in the range 1.1–1.5 times U_{eq} of the parent atom), after which their positions were refined with riding constraints.

Figures

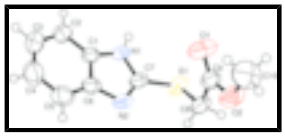


Fig. 1. The molecular structure of compound (I) and the atomic numbering scheme. Displacement ellipsoids are drawn at the 50% probability level. H atoms are shown as spheres of arbitary radius.

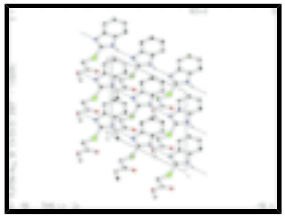


Fig. 2. Crystal packing of compound (I) viewed down the *b* axis, showing the hydrogen-bonded chains along the *c* axis. H atoms not involved in hydrogen bonds have been omitted for clarity. Dashed lines indicate N—H···N hydrogen bonds.

Methyl (1*H*-benzimidazol-2-ylsulfanyl)acetate

Crystal data

$C_{10}H_{10}N_2O_2S$	F_{000} = 464
M_r = 222.27	D_x = 1.357 Mg m^{-3}
Monoclinic, *Cc*	Melting point: 393 K
Hall symbol: C -2yc	Mo *K*α radiation λ = 0.71073 Å
a = 9.337 (9) Å	Cell parameters from 10772 reflections
b = 13.418 (15) Å	θ = 3–28º
c = 9.667 (8) Å	μ = 0.28 mm^{-1}
β = 116.04 (7)º	*T* = 295 K
V = 1088 (2) Å^3	Lozenge, white
Z = 4	0.40 × 0.20 × 0.15 mm

Data collection

Nonius KappaCCD diffractometer	2248 reflections with $I > 2\sigma(I)$
Monochromator: graphite	R_{int} = 0.037
T = 295 K	θ_{max} = 27.9º
φ scans	θ_{min} = 3.0º
Absorption correction: multi-scan (SORTAV; Blessing, 1995)	*h* = −12→12
T_{min} = 0.88, T_{max} = 0.96	*k* = −17→17
10772 measured reflections	*l* = −12→11
2433 independent reflections	

Refinement

Refinement on F^2	Hydrogen site location: inferred from neighbouring sites
Least-squares matrix: full	H-atom parameters constrained

$R[F^2 > 2\sigma(F^2)] = 0.037$	$w = [1-(F_o-F_c)^2/36\sigma^2(F)]^2/[349T_0(x) + 411T_1(x) + 249T_2(x) + 63.5T_3(x)]$ where T_i are Chebychev polynomials and x = F_c/F_{max} (*P*rince, 1982; Watkin, 1994)
$wR(F^2) = 0.062$	$(\Delta/\sigma)_{max} = 0.0002$
$S = 0.92$	$\Delta\rho_{max} = 0.15$ e Å^{-3}
2433 reflections	$\Delta\rho_{min} = -0.13$ e Å^{-3}
137 parameters	Extinction correction: None
2 restraints	Absolute structure: Flack (1983), 1156 Friedel pairs
Primary atom site location: structure-invariant direct methods	Flack parameter: −0.01 (7)

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

	x	*y*	*z*	U_{iso}*/U_{eq}
S1	0.13266 (12)	−0.07154 (4)	0.70877 (11)	0.0629
N1	0.3917 (2)	0.00112 (13)	0.95018 (18)	0.0512
N2	0.3656 (2)	0.05903 (13)	0.72407 (18)	0.0539
O1	−0.0254 (2)	0.11704 (13)	0.73910 (19)	0.0868
O2	−0.1561 (2)	0.14742 (14)	0.48884 (18)	0.0771
C1	0.5178 (2)	0.06469 (16)	0.9834 (2)	0.0481
C2	0.6407 (2)	0.09640 (19)	1.1223 (2)	0.0642
C3	0.7432 (3)	0.1657 (2)	1.1134 (3)	0.0746
C4	0.7264 (3)	0.2027 (2)	0.9725 (3)	0.0758
C5	0.6069 (3)	0.17041 (19)	0.8362 (3)	0.0684
C6	0.5009 (2)	0.10176 (16)	0.8420 (2)	0.0500
C7	0.3049 (2)	0.00213 (15)	0.7947 (2)	0.0484
C8	0.0081 (3)	0.01087 (17)	0.5564 (2)	0.0598
C9	−0.0568 (3)	0.09576 (15)	0.6092 (2)	0.0543
C10	−0.2304 (4)	0.2342 (2)	0.5187 (4)	0.0984
H101	−0.2889	0.2693	0.4257	0.1501*
H102	−0.3051	0.2101	0.5520	0.1500*
H103	−0.1562	0.2761	0.5931	0.1500*
H2	0.6509	0.0725	1.2184	0.0740*
H82	0.0693	0.0398	0.5053	0.0659*
H3	0.8243	0.1908	1.2060	0.0843*
H4	0.7915	0.2524	0.9723	0.0907*
H1	0.3683	−0.0280	1.0151	0.0619*
H81	−0.0736	−0.0295	0.4848	0.0681*
H5	0.5948	0.1940	0.7390	0.0841*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
S1	0.0686 (3)	0.0556 (3)	0.0471 (3)	−0.0007 (3)	0.0094 (2)	−0.0013 (3)
N1	0.0571 (9)	0.0653 (10)	0.0303 (7)	0.0007 (8)	0.0184 (7)	0.0052 (7)
N2	0.0656 (10)	0.0634 (10)	0.0304 (7)	0.0074 (8)	0.0190 (7)	0.0026 (7)
O1	0.1244 (15)	0.0847 (12)	0.0467 (9)	0.0190 (12)	0.0332 (9)	−0.0042 (8)

O2	0.0834 (11)	0.0879 (12)	0.0508 (9)	0.0263 (9)	0.0212 (8)	0.0110 (8)
C1	0.0476 (10)	0.0619 (12)	0.0346 (8)	0.0086 (9)	0.0177 (7)	0.0024 (8)
C2	0.0511 (11)	0.0960 (17)	0.0375 (10)	−0.0016 (11)	0.0121 (9)	0.0059 (10)
C3	0.0511 (12)	0.0985 (18)	0.0608 (14)	−0.0046 (13)	0.0121 (10)	−0.0025 (13)
C4	0.0605 (13)	0.0882 (16)	0.0754 (15)	−0.0070 (12)	0.0267 (11)	0.0150 (14)
C5	0.0697 (14)	0.0845 (16)	0.0543 (12)	0.0022 (12)	0.0304 (11)	0.0170 (11)
C6	0.0546 (10)	0.0593 (11)	0.0369 (9)	0.0107 (9)	0.0207 (8)	0.0056 (8)
C7	0.0561 (10)	0.0541 (11)	0.0313 (8)	0.0115 (9)	0.0160 (8)	0.0009 (8)
C8	0.0616 (12)	0.0709 (13)	0.0332 (9)	0.0035 (11)	0.0081 (9)	−0.0051 (9)
C9	0.0588 (11)	0.0604 (12)	0.0408 (10)	−0.0041 (9)	0.0194 (8)	0.0011 (8)
C10	0.112 (2)	0.0871 (19)	0.104 (2)	0.0331 (17)	0.054 (2)	0.0222 (16)

Geometric parameters (Å, °)

S1—C7	1.755 (3)	C2—H2	0.947
S1—C8	1.800 (2)	C3—C4	1.392 (4)
N1—C1	1.373 (3)	C3—H3	0.945
N1—C7	1.359 (3)	C4—C5	1.371 (4)
N1—H1	0.845	C4—H4	0.902
N2—C6	1.400 (3)	C5—C6	1.371 (3)
N2—C7	1.308 (3)	C5—H5	0.949
O1—C9	1.192 (2)	C8—C9	1.482 (3)
O2—C9	1.322 (3)	C8—H82	0.985
O2—C10	1.448 (3)	C8—H81	0.944
C1—C2	1.395 (3)	C10—H101	0.947
C1—C6	1.397 (3)	C10—H102	0.944
C2—C3	1.364 (3)	C10—H103	0.936
C7—S1—C8	99.78 (13)	N2—C6—C1	108.96 (19)
C1—N1—C7	106.67 (17)	N2—C6—C5	130.61 (19)
C1—N1—H1	126.0	C1—C6—C5	120.4 (2)
C7—N1—H1	127.1	S1—C7—N1	119.73 (16)
C6—N2—C7	104.87 (17)	S1—C7—N2	126.76 (15)
C9—O2—C10	117.3 (2)	N1—C7—N2	113.49 (18)
N1—C1—C2	132.32 (18)	S1—C8—C9	113.90 (16)
N1—C1—C6	105.98 (17)	S1—C8—H82	110.1
C2—C1—C6	121.6 (2)	C9—C8—H82	106.5
C1—C2—C3	116.8 (2)	S1—C8—H81	105.7
C1—C2—H2	121.8	C9—C8—H81	111.9
C3—C2—H2	121.4	H82—C8—H81	108.6
C2—C3—C4	121.6 (2)	C8—C9—O2	109.53 (17)
C2—C3—H3	118.4	C8—C9—O1	126.75 (19)
C4—C3—H3	119.9	O2—C9—O1	123.7 (2)
C3—C4—C5	121.5 (2)	O2—C10—H101	109.8
C3—C4—H4	118.6	O2—C10—H102	106.5
C5—C4—H4	119.8	H101—C10—H102	106.8
C4—C5—C6	118.1 (2)	O2—C10—H103	112.2
C4—C5—H5	122.6	H101—C10—H103	110.7
C6—C5—H5	119.3	H102—C10—H103	110.6

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1—H1···N2^{i}	0.85	2.07	2.879 (2)	159

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

Fig. 1

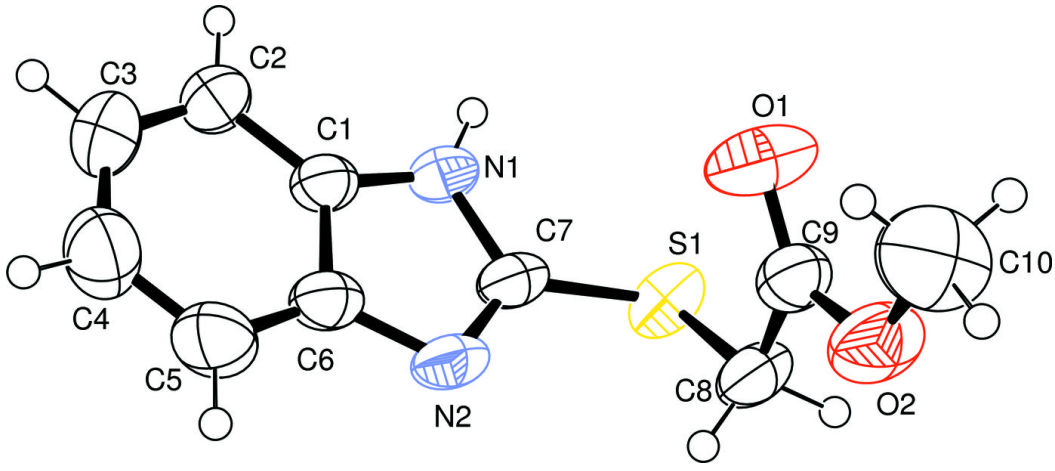
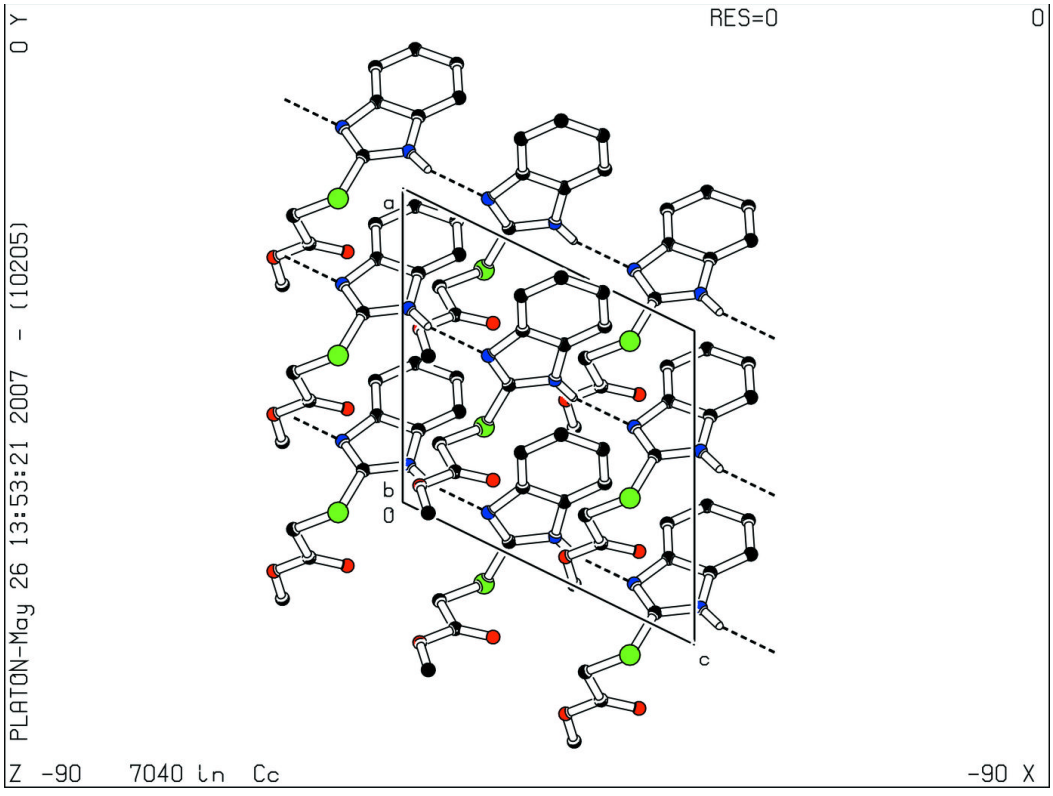


Fig. 2


RES=0
0
0 Y
PLATON-May 26 13:53:21 2007 - (10205)
a
b
0
c
Z -90
7040 ln Cc
-90 X