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Crystal structure of 1-[(2,2-dimethyl-1,3-dioxolan-4-yl)methyl]-2-(thiazol-4-yl)-1*H*-benzimidazole

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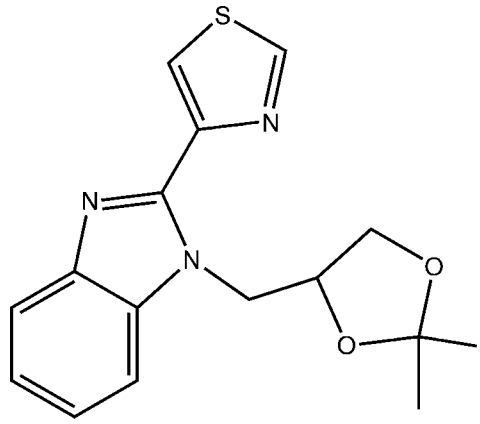
The benzimidazole ring in the title compound, $C_{16}H_{17}N_3O_2S$, is almost planar, with the greatest deviation from the mean plane being 0.032 (1) Å. The fused-ring system makes dihedral angles of 19.91 (7) and 24.51 (8)° with the best plane through each of the thiazol-4-yl and 1,3-dioxolan-4-yl rings, respectively; the latter exhibits an envelope conformation with the methylene C atom being the flap. Finally, the thiazol-4-yl ring makes a dihedral angle of 33.85 (9)° with the 1,3-dioxolan-4-yl ring. In the crystal, molecules are connected by a pair of C—H···π(imidazole) interactions to form centrosymmetric aggregates.

Keywords: crystal structure; benzimidazole; thiazol-4-yl; 1,3-dioxolan-4-yl.

CCDC reference: 1435046

1. Related literature

For the use of the title compound as an anthelmintic, see: Brown *et al.* (1961); Hennekeuser *et al.* (1969); as a food preservative and an agricultural fungicide, see: Arenas & Johnson (1994); for induction of aneuploidy and photogenotoxicity in bacteria and cultured human cells, see: Watanabe-Akanuma *et al.* (2005); as an anti-angiogenic, see: Cha *et al.* (2012); and as a ligand for transition metal ions, see: Gueddar *et al.* (2013).



2. Experimental

2.1. Crystal data

$C_{16}H_{17}N_3O_2S$
M_r = 315.38
Triclinic, $P\overline{1}$
a = 9.3177 (5) Å
b = 9.3786 (6) Å
c = 9.5418 (6) Å
α = 78.739 (4)°
β = 78.777 (3)°
γ = 73.632 (3)°
V = 775.95 (8) Å^3
Z = 2
Mo $K\alpha$ radiation
μ = 0.22 mm^{-1}
T = 296 K
0.36 × 0.31 × 0.26 mm

2.2. Data collection

Bruker X8 APEX diffractometer
Absorption correction: multi-scan (*SADABS*; Bruker, 2009)
T_{min} = 0.700, T_{max} = 0.747
19810 measured reflections
4740 independent reflections
2804 reflections with $I > 2\sigma(I)$
R_{int} = 0.037

2.3. Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.043
$wR(F^2)$ = 0.116
S = 1.03
4740 reflections
199 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.16 e Å^{-3}
$\Delta\rho_{min}$ = −0.32 e Å^{-3}

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

*Cg*1 is the centroid of the N2/N3/C4/C5/C10 ring.

D—H···A	D—H	H···A	D···A	D—H···A
C13—H13*B*···*Cg*1^{i}	0.97	2.83	3.7543 (18)	160

Symmetry code: (i) $-x+1, -y+1, -z+1$.

Data collection: *APEX2* (Bruker, 2009); cell refinement: *SAINT-Plus* (Bruker, 2009); data reduction: *SAINT-Plus*; program(s) used to solve structure: *SHELXS2014*/ (Sheldrick, 2008); program(s) used to refine structure: *SHELXL2014* (Sheldrick, 2015); molecular graphics: *ORTEP-3 for Windows* (Farrugia, 2012); software used to prepare material for publication: *PLATON* (Spek, 2009) and *publCIF* (Westrip,2010).

Acknowledgements

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Supporting information for this paper is available from the IUCr electronic archives (Reference: TK5404).

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supporting information

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Crystal structure of 1-[(2,2-dimethyl-1,3-dioxolan-4-yl)methyl]-2-(thiazol-4-yl)-1*H*-benzimidazole

Hicham Gueddar, Rachid Bouhfid, El Mokhtar Essassi, Mohamed Saadi and Lahcen El Ammari

S1. Comment

Thiabendazole [2-(4-thiazolyl)benzimidazole, TBZ], is used as a broad spectrum anthelmintic in various animals (Brown *et al.*, 1961) and in humans (Hennekeuser *et al.*, 1969). TBZ inhibits anaerobic respiration at the level of mitochondrial helminth-specific enzyme. This compound has also been used as a food preservative and an agricultural fungicide (Arenas and Johnson, 1994). Induction of aneuploidy and photogenotoxicity has been reported for TBZ in bacteria and cultured human cells (Watanabe-Akanuma *et al.*, 2005). TBZ has recently been verified to be vascular disrupting agent and thus as a potential complementary therapeutic for use in combination with current anti-angiogenic therapeutics (Cha *et al.*, 2012). TBZ is also an effective ligand to coordinate transition metal ions (Gueddar *et al.*, 2013). The title compound is synthesized by action of TBZ on tosylated solketal under phase transfer catalysis conditions.

The molecule of the title compound is build up from fused five- and six-membered rings linked to a thiazol-4-yl cycle and, *via* one —CH_2— link, to a 2,2-dimethyl-1,3-dioxolan-4-yl group as shown in Fig. 1. The benzimidazole ring is essentially planar with the maximum deviation from the mean plane being 0.032 (1)° at C4 and makes dihedral angles of 19.91 (7) and 24.51 (8)° with the mean plane through the thiazol-4-yl and the 3-dioxolan-4-yl rings, respectively. The dihedral angle between the thiazol-4-yl cycle and the 3-dioxolan-4-yl ring is of 33.85 (9)°. Furthermore, the five-membered ring (O1O2C12C13C14) adopts an envelope conformations on C13 as indicated by the total puckering amplitude Q2 = 0.381 (2) Å and spherical polar angle $\varphi 2$ = 72.2 (2)°. In the crystal, the molecules are held together by C13–H13B···π interactions involving the imidazole ring.

S2. Experimental

To a solution of thiabendazole (1 g, 5 × 10^{-3} mol.) dissolved in DMF (20 ml) was added potassium carbonate (0.83 g, 6 × 10^{-3} mol.), tetra-*n*-butylammonium bromide (0.13 g, 0.4 × 10^{-3} mol) and tosylated solketal (2.79 g, 10 × 10^{-3} mol). The mixture was heated for 48 h. After the completion of the reaction (as monitored by TLC), the inorganic material was filtered and the solvent was removed under reduced pressure. The residue obtained was recrystallized from ethanol to afford the title compound as colourless crystals.

S3. Refinement

The H atoms were located in a difference map and treated as riding with C—H = 0.93 Å (aromatic), C—H = 0.97 (methylene), C—H = 0.98 (methine) and C—H = 0.96 Å (methyl), and with U_{iso}(H) = 1.2 U_{eq}(aromatic, methine and methylene) and U_{iso}(H) = 1.5 U_{eq}(methyl). The reflections (0 1 0) and (1 0 0), which were affected by the beam-stop, were removed from the final cycles of refinement owing to poor agreement.

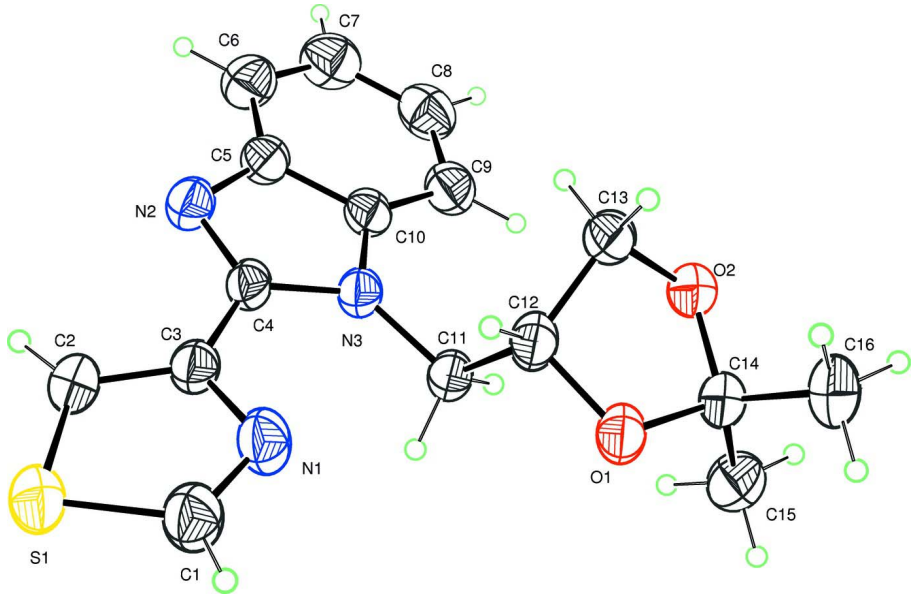

Figure 1

Molecular structure of the title compound with the atom-labelling scheme. Displacement ellipsoids are drawn at the 50% probability level. H atoms are represented as small circles.

1-[(2,2-Dimethyl-1,3-dioxolan-4-yl)methyl]-2-(thiazol-4-yl)-1*H*-benzimidazole

Crystal data

$C_{16}H_{17}N_3O_2S$
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Triclinic, $P\overline{1}$
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$\alpha = 78.739$ (4)°
$\beta = 78.777$ (3)°
$\gamma = 73.632$ (3)°
$V = 775.95$ (8) Å^3
$Z = 2$
$F(000) = 332$
$D_x = 1.350$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 4740 reflections
$\theta = 2.8$–30.5°
$\mu = 0.22$ mm^{-1}
$T = 296$ K
Block, colourless
0.36 × 0.31 × 0.26 mm

Data collection

Bruker X8 APEX diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Bruker, 2009)
$T_{min} = 0.700$, $T_{max} = 0.747$
19810 measured reflections
4740 independent reflections
2804 reflections with $I > 2\sigma(I)$
$R_{int} = 0.037$
$\theta_{max} = 30.5°$, $\theta_{min} = 2.8°$
$h = -13 \rightarrow 13$
$k = -13 \rightarrow 13$
$l = -13 \rightarrow 13$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.043$
$wR(F^2) = 0.116$
$S = 1.03$
4740 reflections
199 parameters
0 restraints
Hydrogen site location: inferred from neighbouring sites

H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0449P)^2 + 0.0871P]$ where $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.16$ e Å^{-3}
$\Delta\rho_{min} = -0.32$ e Å^{-3}

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.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²)

	x	*y*	*z*	U_{iso}*/U_{eq}
C1	0.9210 (2)	−0.0317 (2)	0.68651 (19)	0.0621 (5)
H1	1.0193	−0.0386	0.6391	0.074*
C2	0.69556 (19)	−0.05253 (19)	0.84926 (17)	0.0567 (4)
H2	0.6188	−0.0714	0.9230	0.068*
C3	0.67345 (17)	0.04071 (16)	0.72279 (15)	0.0455 (3)
C4	0.52432 (17)	0.11882 (16)	0.68306 (15)	0.0431 (3)
C5	0.28330 (18)	0.20058 (17)	0.69744 (17)	0.0480 (4)
C6	0.12767 (19)	0.2376 (2)	0.7404 (2)	0.0625 (5)
H6	0.0870	0.2227	0.8375	0.075*
C7	0.0354 (2)	0.2967 (2)	0.6362 (2)	0.0700 (5)
H7	−0.0690	0.3218	0.6636	0.084*
C8	0.0949 (2)	0.3197 (2)	0.4904 (2)	0.0672 (5)
H8	0.0292	0.3598	0.4225	0.081*
C9	0.2485 (2)	0.28454 (19)	0.44413 (19)	0.0569 (4)
H9	0.2883	0.2997	0.3467	0.068*
C10	0.34127 (17)	0.22519 (16)	0.55071 (16)	0.0450 (3)
C11	0.60240 (18)	0.16781 (16)	0.40757 (15)	0.0454 (3)
H11A	0.5501	0.1612	0.3315	0.055*
H11B	0.6843	0.0776	0.4179	0.055*
C12	0.66868 (18)	0.30373 (17)	0.36290 (16)	0.0477 (4)
H12	0.7195	0.3150	0.4394	0.057*
C13	0.55626 (19)	0.44806 (18)	0.31814 (17)	0.0535 (4)
H13A	0.4595	0.4562	0.3802	0.064*
H13B	0.5932	0.5348	0.3188	0.064*
C14	0.69603 (17)	0.36581 (17)	0.11179 (16)	0.0488 (4)
C15	0.6876 (2)	0.2593 (2)	0.01678 (19)	0.0673 (5)
H15A	0.7877	0.2135	−0.0270	0.101*
H15B	0.6277	0.3134	−0.0572	0.101*
H15C	0.6420	0.1827	0.0738	0.101*
C16	0.7744 (2)	0.4856 (2)	0.0327 (2)	0.0702 (5)
H16A	0.8749	0.4389	−0.0094	0.105*
H16B	0.7793	0.5480	0.0993	0.105*
H16C	0.7187	0.5459	−0.0422	0.105*
N1	0.80427 (15)	0.05273 (16)	0.62983 (15)	0.0575 (4)
N2	0.40020 (14)	0.13467 (15)	0.77840 (13)	0.0487 (3)
N3	0.49717 (14)	0.17320 (13)	0.54286 (12)	0.0428 (3)

O1	0.77265 (12)	0.28446 (12)	0.23223 (11)	0.0532 (3)
O2	0.54583 (12)	0.43257 (12)	0.17557 (11)	0.0528 (3)
S1	0.88238 (5)	−0.12939 (6)	0.85352 (5)	0.06612 (16)

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.0568 (10)	0.0661 (12)	0.0570 (10)	−0.0168 (9)	−0.0043 (8)	0.0032 (9)
C2	0.0634 (10)	0.0589 (10)	0.0394 (8)	−0.0103 (8)	−0.0008 (7)	−0.0019 (7)
C3	0.0575 (9)	0.0398 (8)	0.0378 (8)	−0.0142 (7)	−0.0019 (7)	−0.0050 (6)
C4	0.0582 (9)	0.0353 (7)	0.0353 (7)	−0.0151 (6)	−0.0030 (6)	−0.0028 (6)
C5	0.0571 (9)	0.0408 (8)	0.0460 (8)	−0.0155 (7)	−0.0027 (7)	−0.0067 (7)
C6	0.0574 (10)	0.0646 (11)	0.0609 (11)	−0.0161 (8)	0.0029 (8)	−0.0092 (9)
C7	0.0560 (10)	0.0694 (13)	0.0828 (14)	−0.0143 (9)	−0.0089 (10)	−0.0110 (11)
C8	0.0697 (12)	0.0584 (11)	0.0753 (13)	−0.0103 (9)	−0.0268 (10)	−0.0070 (9)
C9	0.0713 (11)	0.0475 (9)	0.0522 (10)	−0.0137 (8)	−0.0138 (8)	−0.0055 (7)
C10	0.0560 (9)	0.0344 (7)	0.0455 (8)	−0.0143 (6)	−0.0063 (7)	−0.0053 (6)
C11	0.0600 (9)	0.0392 (8)	0.0350 (7)	−0.0138 (7)	−0.0013 (6)	−0.0045 (6)
C12	0.0630 (9)	0.0460 (9)	0.0358 (7)	−0.0202 (7)	−0.0048 (7)	−0.0032 (6)
C13	0.0705 (10)	0.0408 (8)	0.0465 (9)	−0.0163 (8)	0.0016 (8)	−0.0071 (7)
C14	0.0540 (9)	0.0481 (9)	0.0383 (8)	−0.0113 (7)	−0.0030 (7)	0.0013 (7)
C15	0.0823 (13)	0.0676 (12)	0.0505 (10)	−0.0155 (10)	−0.0057 (9)	−0.0144 (9)
C16	0.0817 (13)	0.0655 (12)	0.0573 (11)	−0.0289 (10)	0.0011 (9)	0.0091 (9)
N1	0.0523 (8)	0.0596 (9)	0.0537 (8)	−0.0172 (7)	−0.0055 (6)	0.0095 (7)
N2	0.0546 (7)	0.0489 (7)	0.0405 (7)	−0.0155 (6)	−0.0004 (6)	−0.0048 (6)
N3	0.0534 (7)	0.0380 (7)	0.0354 (6)	−0.0131 (5)	−0.0029 (5)	−0.0029 (5)
O1	0.0508 (6)	0.0579 (7)	0.0422 (6)	−0.0102 (5)	−0.0041 (5)	0.0049 (5)
O2	0.0562 (6)	0.0514 (6)	0.0429 (6)	−0.0066 (5)	−0.0048 (5)	−0.0014 (5)
S1	0.0670 (3)	0.0732 (3)	0.0465 (3)	−0.0051 (2)	−0.0116 (2)	0.0032 (2)

Geometric parameters (Å, º)

C1—N1	1.295 (2)	C10—N3	1.3879 (19)
C1—S1	1.7030 (18)	C11—N3	1.4612 (18)
C1—H1	0.9300	C11—C12	1.522 (2)
C2—C3	1.359 (2)	C11—H11A	0.9700
C2—S1	1.6913 (18)	C11—H11B	0.9700
C2—H2	0.9300	C12—O1	1.4300 (17)
C3—N1	1.3794 (19)	C12—C13	1.504 (2)
C3—C4	1.461 (2)	C12—H12	0.9800
C4—N2	1.3167 (18)	C13—O2	1.4205 (19)
C4—N3	1.3807 (18)	C13—H13A	0.9700
C5—N2	1.388 (2)	C13—H13B	0.9700
C5—C6	1.390 (2)	C14—O2	1.4299 (18)
C5—C10	1.398 (2)	C14—O1	1.4431 (18)
C6—C7	1.372 (3)	C14—C15	1.501 (2)
C6—H6	0.9300	C14—C16	1.510 (2)
C7—C8	1.393 (3)	C15—H15A	0.9600

C7—H7	0.9300	C15—H15B	0.9600
C8—C9	1.378 (3)	C15—H15C	0.9600
C8—H8	0.9300	C16—H16A	0.9600
C9—C10	1.393 (2)	C16—H16B	0.9600
C9—H9	0.9300	C16—H16C	0.9600
N1—C1—S1	115.57 (13)	O1—C12—C11	108.49 (12)
N1—C1—H1	122.2	C13—C12—C11	114.15 (13)
S1—C1—H1	122.2	O1—C12—H12	110.6
C3—C2—S1	110.39 (12)	C13—C12—H12	110.6
C3—C2—H2	124.8	C11—C12—H12	110.6
S1—C2—H2	124.8	O2—C13—C12	101.61 (12)
C2—C3—N1	114.69 (14)	O2—C13—H13A	111.4
C2—C3—C4	123.77 (14)	C12—C13—H13A	111.4
N1—C3—C4	121.50 (13)	O2—C13—H13B	111.4
N2—C4—N3	113.24 (13)	C12—C13—H13B	111.4
N2—C4—C3	122.47 (13)	H13A—C13—H13B	109.3
N3—C4—C3	124.13 (13)	O2—C14—O1	104.86 (11)
N2—C5—C6	130.26 (15)	O2—C14—C15	108.45 (13)
N2—C5—C10	110.23 (13)	O1—C14—C15	110.33 (13)
C6—C5—C10	119.47 (16)	O2—C14—C16	110.57 (13)
C7—C6—C5	118.54 (17)	O1—C14—C16	109.05 (13)
C7—C6—H6	120.7	C15—C14—C16	113.24 (15)
C5—C6—H6	120.7	C14—C15—H15A	109.5
C6—C7—C8	121.29 (17)	C14—C15—H15B	109.5
C6—C7—H7	119.4	H15A—C15—H15B	109.5
C8—C7—H7	119.4	C14—C15—H15C	109.5
C9—C8—C7	121.69 (18)	H15A—C15—H15C	109.5
C9—C8—H8	119.2	H15B—C15—H15C	109.5
C7—C8—H8	119.2	C14—C16—H16A	109.5
C8—C9—C10	116.57 (17)	C14—C16—H16B	109.5
C8—C9—H9	121.7	H16A—C16—H16B	109.5
C10—C9—H9	121.7	C14—C16—H16C	109.5
N3—C10—C9	131.86 (14)	H16A—C16—H16C	109.5
N3—C10—C5	105.65 (13)	H16B—C16—H16C	109.5
C9—C10—C5	122.43 (15)	C1—N1—C3	109.91 (14)
N3—C11—C12	113.34 (12)	C4—N2—C5	104.91 (12)
N3—C11—H11A	108.9	C4—N3—C10	105.96 (12)
C12—C11—H11A	108.9	C4—N3—C11	129.77 (13)
N3—C11—H11B	108.9	C10—N3—C11	124.02 (12)
C12—C11—H11B	108.9	C12—O1—C14	108.64 (11)
H11A—C11—H11B	107.7	C13—O2—C14	106.22 (12)
O1—C12—C13	101.94 (12)	C2—S1—C1	89.43 (8)

Hydrogen-bond geometry (Å, º)

Cg1 is the centroid of the N2/N3/C4/C5/C10 ring.

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C13—H13*B*···*Cg*1^{i}	0.97	2.83	3.7543 (18)	160

Symmetry code: (i) −*x*+1, −*y*+1, −*z*+1.