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Methyl 2-(1,1,3-trioxo-2,3-dihydro-1,2-benzothiazol-2-yl)acetate: a monoclinic polymorph

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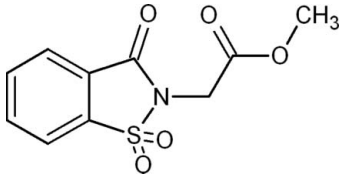
Key indicators: single-crystal X-ray study; T = 296 K; mean σ(C–C) = 0.003 Å; R factor = 0.039; wR factor = 0.115; data-to-parameter ratio = 18.0.

In the title compound, $C_{10}H_9NO_5S$, the fused ring system and the planar (r.m.s. deviation = 0.0037 Å) methoxycarbonylmethyl side chain form a dihedral angle of 84.67 (10)°. The crystal structure is stabilized by intermolecular C—H···O hydrogen bonds. A triclinic polymorph of the title compound is already known [Siddiqui *et al.* (2008). *Acta Cryst.* E**64**, o859].

Related literature

For the synthesis and biological activity of related compounds, see: Ahmad *et al.* (2010); Zia-ur-Rehman *et al.* (2005, 2006, 2007). For a related structure, see: Arshad *et al.* (2009). For the triclinic polymorph, see: Siddiqui *et al.* (2008).



Experimental

Crystal data

$C_{10}H_9NO_5S$
M_r = 255.24
Monoclinic, $P2_1/n$
a = 8.9418 (4) Å
b = 12.7595 (6) Å
c = 10.3145 (5) Å
β = 107.300 (1)°
V = 1123.57 (9) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.30 mm^{-1}
T = 296 K
0.43 × 0.41 × 0.13 mm

Data collection

Bruker APEXII CCD area-detector diffractometer
Absorption correction: multi-scan (*SADABS*; Sheldrick, 2004)
T_{min} = 0.883, T_{max} = 0.962
12621 measured reflections
2796 independent reflections
2022 reflections with $I > 2\sigma(I)$
R_{int} = 0.025

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.039
$wR(F^2)$ = 0.115
S = 1.03
2796 reflections
155 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.24 e Å^{-3}
$\Delta\rho_{min}$ = −0.32 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
C8—H8B···O1^{i}	0.97	2.46	3.371 (3)	156
C2—H2···O4^{ii}	0.93	2.59	3.455 (3)	155
C3—H3···O2^{ii}	0.93	2.50	3.331 (3)	148
C10—H10C···O2^{iii}	0.96	2.52	3.419 (3)	156

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

Data collection: *APEX2* (Bruker, 2007); cell refinement: *SAINT* (Bruker, 2007); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *PLATON* (Spek, 2009); software used to prepare material for publication: *WinGX* (Farrugia, 1999) and *PLATON*.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: BT5235).

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supporting information

Acta Cryst. (2010). E**66**, o1028 [https://doi.org/10.1107/S1600536810012006]

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Muhammad Zia-ur-Rehman, Muhammad Nadeem Arshad, Shafaq Mubarak and Islam Ullah Khan

S1. Comment

A triclinic polymorph (Siddiqui *et al.*, 2008) of the title compound, methyl (1,1-dioxido-3-oxo-1,2-benzisothiazol-2(3*H*)-yl)acetate (**I**), has already been reported. In continuation of our work on the synthesis (Zia-ur-Rehman *et al.*, 2006; Ahmad *et al.*, 2010), and crystal structures (Zia-ur-Rehman *et al.*, 2007; Arshad *et al.*, 2009) of various 1,2-benzothiazine-1,1-dioxides, we have been able to crystallize a monoclinic polymorph of the title compound. The methoxycarbonylmethyl side chain is oriented at a dihedral angle of 84.67 (10)° with respect to the fused ring system. The molecules are connected by intermolecular C–H···O interactions giving rise to a three dimentional network.

S2. Experimental

The title compound was prepared following the procedure reported earlier (Zia-ur-Rehman *et al.*, 2005). Crystals suitable for X-ray crystallography were grown in chloroform by slow evaporation at 313 K.

S3. Refinement

H-atoms were included in the refinement at geometrically idealized positions with aryl, methylene and methyl C—H distances 0.95, 0.99 and 0.98 Å, respectively, and U(H) = 1.2 times U_{eq}(C).

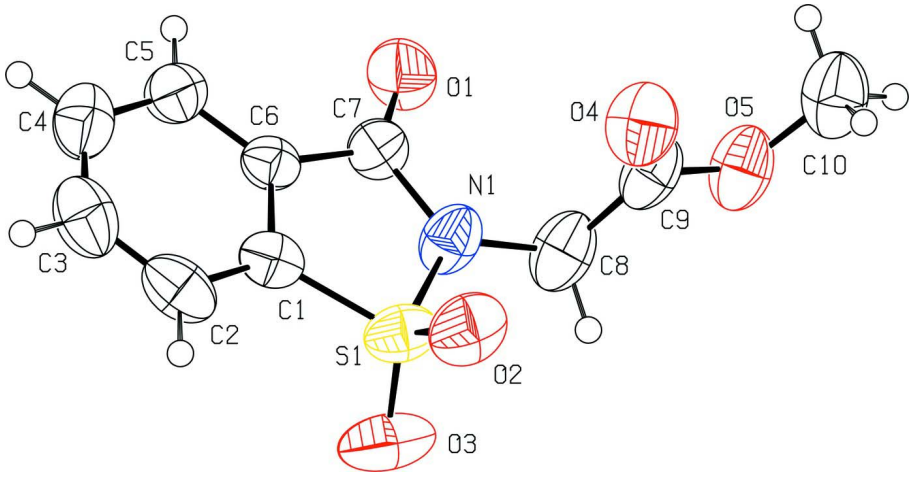

Figure 1

The title molecule with the displacement ellipsoids plotted at 50% probability level.

methyl 2-(1,1,3-trioxo-2,3-dihydro-1,2-benzothiazol-2-yl)acetate

Crystal data

$C_{10}H_9NO_5S$
$M_r = 255.24$
Monoclinic, $P2_1/n$
Hall symbol: -P 2yn
$a = 8.9418$ (4) Å
$b = 12.7595$ (6) Å
$c = 10.3145$ (5) Å
$\beta = 107.300$ (1)°
$V = 1123.57$ (9) Å^3
$Z = 4$
$F(000) = 528$
$D_x = 1.509$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 4707 reflections
$\theta = 2.7$–$26.7°$
$\mu = 0.30$ mm^{-1}
$T = 296$ K
Needles, colourless
0.43 × 0.41 × 0.13 mm

Data collection

Bruker APEXII CCD area-detector diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
phi and ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 2004)
$T_{min} = 0.883$, $T_{max} = 0.962$
12621 measured reflections
2796 independent reflections
2022 reflections with $I > 2\sigma(I)$
$R_{int} = 0.025$
$\theta_{max} = 28.4°$, $\theta_{min} = 2.6°$
$h = -11 \rightarrow 11$
$k = -16 \rightarrow 17$
$l = -13 \rightarrow 12$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.039$
$wR(F^2) = 0.115$
$S = 1.03$
2796 reflections
155 parameters
0 restraints
Primary atom site location: structure-invariant direct methods
Secondary atom site location: difference Fourier map
Hydrogen site location: inferred from neighbouring sites
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0523P)^2 + 0.2836P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.24$ e Å^{-3}
$\Delta\rho_{min} = -0.32$ e Å^{-3}

Special details

Geometry. All s.u.'s (except the s.u. in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell s.u.'s are taken into account individually in the estimation of s.u.'s in distances, angles and torsion angles; correlations between s.u.'s in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell s.u.'s is used for estimating s.u.'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 $F^2 > 2\sigma(F^2)$ 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}
S1	0.38220 (5)	0.08616 (4)	0.63435 (4)	0.05841 (18)
O1	0.16399 (18)	0.16167 (11)	0.27984 (13)	0.0700 (4)
O2	0.33381 (18)	0.13244 (13)	0.74048 (13)	0.0744 (4)
O3	0.54303 (16)	0.05974 (16)	0.66339 (16)	0.0892 (5)

O4	0.17124 (19)	0.34008 (12)	0.54605 (16)	0.0776 (4)
O5	0.33001 (19)	0.44091 (12)	0.46863 (15)	0.0729 (4)
N1	0.3269 (2)	0.16078 (13)	0.49608 (15)	0.0602 (4)
C1	0.25915 (19)	−0.01610 (14)	0.55496 (17)	0.0491 (4)
C2	0.2399 (3)	−0.11272 (17)	0.6095 (2)	0.0649 (5)
H2	0.2951	−0.1303	0.6984	0.078*
C3	0.1358 (3)	−0.18129 (17)	0.5268 (2)	0.0720 (6)
H3	0.1196	−0.2466	0.5605	0.086*
C4	0.0549 (3)	−0.15539 (16)	0.3951 (2)	0.0681 (6)
H4	−0.0144	−0.2036	0.3413	0.082*
C5	0.0746 (2)	−0.05929 (15)	0.3415 (2)	0.0556 (4)
H5	0.0201	−0.0423	0.2523	0.067*
C6	0.17697 (18)	0.01082 (13)	0.42324 (16)	0.0449 (4)
C7	0.2156 (2)	0.11722 (15)	0.38655 (17)	0.0508 (4)
C8	0.3914 (3)	0.26501 (17)	0.4912 (2)	0.0697 (6)
H8A	0.4143	0.2735	0.4056	0.084*
H8B	0.4892	0.2714	0.5636	0.084*
C9	0.2822 (2)	0.35100 (16)	0.50537 (18)	0.0603 (5)
C10	0.2369 (3)	0.53266 (18)	0.4757 (2)	0.0805 (7)
H10A	0.1314	0.5223	0.4189	0.121*
H10B	0.2809	0.5930	0.4450	0.121*
H10C	0.2368	0.5433	0.5678	0.121*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
S1	0.0530 (3)	0.0830 (4)	0.0369 (2)	0.0002 (2)	0.00972 (18)	−0.0025 (2)
O1	0.0958 (11)	0.0643 (9)	0.0439 (7)	0.0047 (7)	0.0117 (7)	0.0109 (6)
O2	0.0837 (10)	0.0975 (11)	0.0418 (7)	−0.0028 (8)	0.0183 (7)	−0.0136 (7)
O3	0.0478 (8)	0.1463 (16)	0.0655 (10)	0.0058 (9)	0.0046 (7)	−0.0070 (10)
O4	0.0842 (10)	0.0759 (10)	0.0847 (11)	−0.0175 (8)	0.0436 (9)	0.0019 (8)
O5	0.0924 (11)	0.0655 (9)	0.0701 (9)	−0.0261 (8)	0.0383 (8)	−0.0062 (7)
N1	0.0750 (10)	0.0620 (10)	0.0416 (8)	−0.0159 (8)	0.0140 (7)	−0.0033 (7)
C1	0.0490 (9)	0.0595 (10)	0.0414 (8)	0.0098 (8)	0.0176 (7)	0.0036 (7)
C2	0.0748 (13)	0.0683 (13)	0.0589 (12)	0.0215 (10)	0.0312 (10)	0.0200 (10)
C3	0.0869 (15)	0.0536 (12)	0.0892 (16)	0.0044 (11)	0.0471 (13)	0.0101 (11)
C4	0.0695 (13)	0.0585 (12)	0.0837 (15)	−0.0092 (10)	0.0340 (11)	−0.0112 (11)
C5	0.0519 (10)	0.0594 (11)	0.0543 (10)	0.0007 (8)	0.0139 (8)	−0.0039 (8)
C6	0.0437 (8)	0.0503 (9)	0.0416 (8)	0.0061 (7)	0.0140 (7)	0.0016 (7)
C7	0.0589 (10)	0.0548 (10)	0.0387 (9)	0.0022 (8)	0.0146 (8)	−0.0011 (7)
C8	0.0799 (14)	0.0725 (14)	0.0637 (12)	−0.0248 (11)	0.0319 (11)	−0.0117 (10)
C9	0.0734 (13)	0.0662 (12)	0.0434 (10)	−0.0248 (10)	0.0203 (9)	−0.0088 (8)
C10	0.1110 (19)	0.0673 (14)	0.0676 (14)	−0.0142 (13)	0.0335 (13)	−0.0033 (11)

Geometric parameters (Å, º)

S1—O3	1.4196 (15)	C3—C4	1.376 (3)
S1—O2	1.4199 (14)	C3—H3	0.9300

S1—N1	1.6630 (16)	C4—C5	1.378 (3)
S1—C1	1.7457 (19)	C4—H4	0.9300
O1—C7	1.202 (2)	C5—C6	1.375 (2)
O4—C9	1.194 (2)	C5—H5	0.9300
O5—C9	1.319 (2)	C6—C7	1.478 (3)
O5—C10	1.451 (3)	C8—C9	1.505 (3)
N1—C7	1.381 (2)	C8—H8A	0.9700
N1—C8	1.456 (3)	C8—H8B	0.9700
C1—C6	1.382 (2)	C10—H10A	0.9600
C1—C2	1.387 (3)	C10—H10B	0.9600
C2—C3	1.374 (3)	C10—H10C	0.9600
C2—H2	0.9300		

O3—S1—O2	117.25 (9)	C6—C5—H5	120.8
O3—S1—N1	109.94 (10)	C4—C5—H5	120.8
O2—S1—N1	110.00 (10)	C5—C6—C1	120.23 (17)
O3—S1—C1	112.28 (10)	C5—C6—C7	127.21 (16)
O2—S1—C1	112.24 (9)	C1—C6—C7	112.55 (15)
N1—S1—C1	92.32 (8)	O1—C7—N1	123.13 (17)
C9—O5—C10	116.58 (17)	O1—C7—C6	127.78 (17)
C7—N1—C8	122.39 (16)	N1—C7—C6	109.08 (15)
C7—N1—S1	115.42 (13)	N1—C8—C9	112.79 (16)
C8—N1—S1	122.19 (14)	N1—C8—H8A	109.0
C6—C1—C2	121.73 (18)	C9—C8—H8A	109.0
C6—C1—S1	110.62 (13)	N1—C8—H8B	109.0
C2—C1—S1	127.65 (15)	C9—C8—H8B	109.0
C3—C2—C1	117.17 (19)	H8A—C8—H8B	107.8
C3—C2—H2	121.4	O4—C9—O5	125.2 (2)
C1—C2—H2	121.4	O4—C9—C8	125.44 (19)
C2—C3—C4	121.4 (2)	O5—C9—C8	109.32 (17)
C2—C3—H3	119.3	O5—C10—H10A	109.5
C4—C3—H3	119.3	O5—C10—H10B	109.5
C3—C4—C5	121.2 (2)	H10A—C10—H10B	109.5
C3—C4—H4	119.4	O5—C10—H10C	109.5
C5—C4—H4	119.4	H10A—C10—H10C	109.5
C6—C5—C4	118.33 (19)	H10B—C10—H10C	109.5

O3—S1—N1—C7	115.77 (16)	C2—C1—C6—C5	−1.0 (3)
O2—S1—N1—C7	−113.66 (15)	S1—C1—C6—C5	179.06 (13)
C1—S1—N1—C7	1.03 (15)	C2—C1—C6—C7	179.97 (16)
O3—S1—N1—C8	−64.37 (18)	S1—C1—C6—C7	0.06 (18)
O2—S1—N1—C8	66.20 (18)	C8—N1—C7—O1	0.0 (3)
C1—S1—N1—C8	−179.10 (16)	S1—N1—C7—O1	179.84 (15)
O3—S1—C1—C6	−113.27 (13)	C8—N1—C7—C6	179.00 (16)
O2—S1—C1—C6	112.14 (13)	S1—N1—C7—C6	−1.14 (19)
N1—S1—C1—C6	−0.60 (13)	C5—C6—C7—O1	0.7 (3)
O3—S1—C1—C2	66.84 (19)	C1—C6—C7—O1	179.61 (18)
O2—S1—C1—C2	−67.76 (18)	C5—C6—C7—N1	−178.26 (17)

N1—S1—C1—C2	179.51 (17)	C1—C6—C7—N1	0.6 (2)
C6—C1—C2—C3	0.3 (3)	C7—N1—C8—C9	77.7 (2)
S1—C1—C2—C3	−179.82 (14)	S1—N1—C8—C9	−102.1 (2)
C1—C2—C3—C4	0.4 (3)	C10—O5—C9—O4	−1.6 (3)
C2—C3—C4—C5	−0.4 (3)	C10—O5—C9—C8	179.77 (17)
C3—C4—C5—C6	−0.4 (3)	N1—C8—C9—O4	16.0 (3)
C4—C5—C6—C1	1.0 (3)	N1—C8—C9—O5	−165.36 (16)
C4—C5—C6—C7	179.88 (17)		

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C8—H8*B*···O1^{i}	0.97	2.46	3.371 (3)	156
C2—H2···O4^{ii}	0.93	2.59	3.455 (3)	155
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