# organic compounds

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

## Muhammad Zia-ur-Rehman,[a]* Muhammad Nadeem Arshad,[b] Shafaq Mubarak[a] and Islam Ullah Khan[b]

[a]Applied Chemistry Research Centre, PCSIR Laboratories Complex, Lahore 54600, Pakistan, and [b]Department of Chemistry, Government College University, Lahore 54000, Pakistan
Correspondence e-mail: rehman_pcsir@hotmail.com

In the title compound, $C_{10}H_9NO_5S$, the fused ring system and the planar (r.m.s. deviation = 0.0037 Å) methoxycarbonyl-methyl 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).
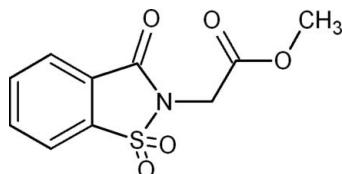


## Experimental

### Crystal data

| | |
|---|---|
| $C_{10}H_9NO_5S$ | $V = 1123.57$ (9) Å³ |
| $M_r = 255.24$ | $Z = 4$ |
| Monoclinic, $P2_1/n$ | Mo $K\alpha$ radiation |
| $a = 8.9418$ (4) Å | $\mu = 0.30$ mm⁻¹ |
| $b = 12.7595$ (6) Å | $T = 296$ K |
| $c = 10.3145$ (5) Å | $0.43 \times 0.41 \times 0.13$ mm |
| $\beta = 107.300$ (1)° | |

### Data collection

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

### Refinement

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

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

| $D$—H···$A$ | $D$—H | H···$A$ | $D$···$A$ | $D$—H···$A$ |
|---|---|---|---|---|
| C8—H8$B$···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—H10$C$···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*.

## References

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**supplementary materials**

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

**M. Zia-ur-Rehman, M. N. Arshad, S. Mubarak and I. U. Khan**

### 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.

### 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.

### 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).

### Figures
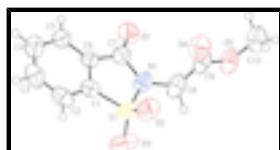


Fig. 1. The title molecule with the displacement ellipsoids plotted at 50% probability level.

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

*Crystal data*

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

$V = 1123.57\ (9)$ Å$^3$

$Z = 4$

$0.43 \times 0.41 \times 0.13$ mm

## Data collection

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

## Refinement

| | |
|---|---|
| Refinement on $F^2$ | Primary atom site location: structure-invariant direct methods |
| Least-squares matrix: full | Secondary atom site location: difference Fourier map |
| $R[F^2 > 2\sigma(F^2)] = 0.039$ | Hydrogen site location: inferred from neighbouring sites |
| $wR(F^2) = 0.115$ | H-atom parameters constrained |
| $S = 1.02$ | $w = 1/[\sigma^2(F_o^2) + (0.0523P)^2 + 0.2836P]$ where $P = (F_o^2 + 2F_c^2)/3$ |
| 2796 reflections | $(\Delta/\sigma)_{max} < 0.001$ |
| 155 parameters | $\Delta\rho_{max} = 0.24$ e Å$^{-3}$ |
| 0 restraints | $\Delta\rho_{min} = -0.32$ 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 (Å$^2$)*

| | $U^{11}$ | $U^{22}$ | $U^{33}$ | $U^{12}$ | $U^{13}$ | $U^{23}$ |
|---|---|---|---|---|---|---|
| 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, °)*

| | | | |
|---|---|---|---|
| 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, °)*

| *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) $x+1/2$, $-y+1/2$, $z+1/2$; (ii) $-x+1/2$, $y-1/2$, $-z+3/2$; (iii) $-x+1/2$, $y+1/2$, $-z+3/2$.

**Fig. 1**