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4-(5-Oxo-5H-1,2,4-dithiazol-3-yl)phenyl 4-methylbenzenesulfonate

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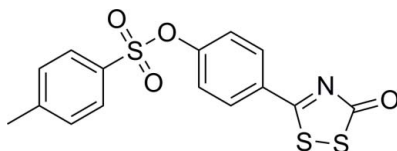
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Key indicators: single-crystal X-ray study; $T = 145$ K; mean $\sigma(\text{C}-\text{C}) = 0.003$ Å; R factor = 0.032; wR factor = 0.078; data-to-parameter ratio = 13.3.

In the molecular structure of the title compound, $\text{C}_{15}\text{H}_{11}\text{NO}_4\text{S}_3$, the 1,2,4-dithiazolone and central benzene rings are approximately coplanar, making a dihedral angle of 3.08 (7)°. The central benzene ring and the 4-methylbenzene ring subtend a dihedral angle of 57.47 (8)°. In the crystal, π - π stacking occurs between the central benzene ring and the 1,2,4-dithiazolone ring of adjacent molecules, which are aligned almost parallel, the centroid-centroid distance being 3.555 (7) Å.

Related literature

For the synthesis of related compounds, see: Cho *et al.* (2003); Chen *et al.* (1996). For their biological activity, see: Iwakawa *et al.* (1994).



Experimental

Crystal data

$\text{C}_{15}\text{H}_{11}\text{NO}_4\text{S}_3$	$V = 3144.36$ (18) Å ³
$M_r = 365.43$	$Z = 8$
Orthorhombic, <i>Pbcn</i>	Mo $K\alpha$ radiation
$a = 30.2449$ (9) Å	$\mu = 0.49$ mm ⁻¹
$b = 7.0841$ (3) Å	$T = 145$ K
$c = 14.6755$ (5) Å	$0.25 \times 0.20 \times 0.20$ mm

Data collection

Agilent Xcalibur Eos diffractometer	6725 measured reflections
Absorption correction: multi-scan (<i>CrysAlis PRO</i> ; Agilent, 2010)	2775 independent reflections
$T_{\min} = 0.952$, $T_{\max} = 1.000$	2355 reflections with $I > 2\sigma(I)$
	$R_{\text{int}} = 0.022$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.032$	209 parameters
$wR(F^2) = 0.078$	H-atom parameters constrained
$S = 1.04$	$\Delta\rho_{\max} = 0.41$ e Å ⁻³
2775 reflections	$\Delta\rho_{\min} = -0.29$ e Å ⁻³

Data collection: *CrysAlis PRO* (Agilent, 2010); cell refinement: *CrysAlis PRO*; data reduction: *CrysAlis PRO*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *OLEX2* (Dolomanov *et al.*, 2009); software used to prepare material for publication: *OLEX2*.

We thank the Analytical and Testing Center of Sichuan University for the X-ray measurements.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: IM2348).

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supplementary materials

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4-(5-Oxo-5H-1,2,4-dithiazol-3-yl)phenyl 4-methylbenzenesulfonate

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Comment

3H-1,2,4-dithiazol-3-one derivatives have been reported to possess antibacterial activity against a variety of bacteria and fungus strains including *Staphylococcus aureus*, *Pasteurella piscicida* and *Botlytis cinerea* (Iwakawa *et al.*, 1994).

The molecular structure of the title compound is shown in Fig. 1. The central benzene ring is twisted away from the planes of the 3H-1,2,4-dithiazol-3-one and 4-methylbenzene rings by 3.08 (7) and 57.47 (8)°, respectively. In the crystal structure, intermolecular π - π stacking between central benzene rings and 3H-1,2,4-dithiazol-3-one rings of adjacent molecules which are aligned almost parallel {centroid-centroid distance = 3.555 (7) Å} are observed (Fig. 2).

Experimental

To a solution of 4-hydroxybenzothioamide (19.10 mmol) in chloroform (20 ml) at 273 K was added pyridine (3.70 ml, 45.84 mmol) dropwise over a period of 20 min and then *p*-toluenesulfonyl chloride (22.92 mmol) in small portions. This reaction mixture was stirred at room temperature for 12 h and diluted with dichloromethane and then 10% aqueous HCl. The separated organic layer was washed with 10% aqueous HCl, water and saturated aqueous NaCl. The organic phase was then dried over Na₂SO₄ and concentrated *in vacuo*. Recrystallization from EtOAc afforded 4-carbamothioylphenyl 4-methylbenzenesulfonate. To the solution of 4-carbamothioylphenyl 4-methylbenzenesulfonate (2 mmol) in THF (10 ml) was added chlorocarbonyl sulfenyl chloride (350 μ L, 4 mmol). The mixture was stirred at ambient temperature for 16 h. The solvent was removed *in vacuo* and the residue was purified by silica-gel column chromatography using EtOAc/hexane as eluent solvent system to get the title compound. Crystals suitable for X-ray analysis were obtained by slow evaporation from a solution in EtOAc at room temperature.

Refinement

H atoms were positioned geometrically and refined using a riding model with C—H = 0.95 Å and $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{C})$ for aromatic H atoms and C—H = 0.98 Å and $U_{\text{iso}}(\text{H}) = 1.5U_{\text{eq}}(\text{C})$ for methyl H atoms.

Figures

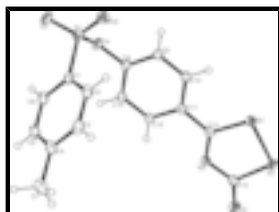


Fig. 1. Molecular structure of the title compound with displacement ellipsoids drawn at the 30% probability level.



Fig. 2. Crystal structure of the title compound.

4-(5-Oxo-5H-1,2,4-dithiazol-3-yl)phenyl 4-methylbenzenesulfonate

Crystal data

$C_{15}H_{11}NO_4S_3$	$D_x = 1.544 \text{ Mg m}^{-3}$
$M_r = 365.43$	Mo $K\alpha$ radiation, $\lambda = 0.7107 \text{ \AA}$
Orthorhombic, $Pbcn$	Cell parameters from 3356 reflections
$a = 30.2449 (9) \text{ \AA}$	$\theta = 2.9\text{--}28.8^\circ$
$b = 7.0841 (3) \text{ \AA}$	$\mu = 0.49 \text{ mm}^{-1}$
$c = 14.6755 (5) \text{ \AA}$	$T = 145 \text{ K}$
$V = 3144.36 (18) \text{ \AA}^3$	Block, colorless
$Z = 8$	$0.25 \times 0.20 \times 0.20 \text{ mm}$
$F(000) = 1504$	

Data collection

Agilent Xcalibur Eos diffractometer	2775 independent reflections
Radiation source: Enhance (Mo) X-ray Source graphite	2355 reflections with $I > 2\sigma(I)$
Detector resolution: $16.0874 \text{ pixels mm}^{-1}$	$R_{\text{int}} = 0.022$
ω scans	$\theta_{\text{max}} = 25.0^\circ$, $\theta_{\text{min}} = 3.0^\circ$
Absorption correction: multi-scan (CrysAlis PRO; Agilent, 2010)	$h = -33 \rightarrow 35$
$T_{\text{min}} = 0.952$, $T_{\text{max}} = 1.000$	$k = -6 \rightarrow 8$
6725 measured reflections	$l = -10 \rightarrow 17$

Refinement

Refinement on F^2	Primary atom site location: structure-invariant direct methods
Least-squares matrix: full	Secondary atom site location: difference Fourier map
$R[F^2 > 2\sigma(F^2)] = 0.032$	Hydrogen site location: inferred from neighbouring sites
$wR(F^2) = 0.078$	H-atom parameters constrained
$S = 1.04$	$w = 1/[\sigma^2(F_o^2) + (0.0338P)^2 + 1.4416P]$
2775 reflections	where $P = (F_o^2 + 2F_c^2)/3$
209 parameters	$(\Delta/\sigma)_{\text{max}} = 0.001$
0 restraints	$\Delta\rho_{\text{max}} = 0.41 \text{ e \AA}^{-3}$
	$\Delta\rho_{\text{min}} = -0.29 \text{ e \AA}^{-3}$

Special details

Experimental. CrysAlisPro, Agilent Technologies, Version 1.171.35.11 (release 16-05-2011 CrysAlis171 .NET) (compiled May 16 2011,17:55:39) Empirical absorption correction using spherical harmonics, implemented in SCALE3 ABSPACK scaling algorithm.

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 $F^2 > \sigma(F^2)$ 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 (\AA^2)

	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{iso}}^*/U_{\text{eq}}$
S1	-0.163728 (17)	0.52253 (8)	0.56985 (4)	0.03114 (16)
S2	0.060198 (17)	0.13631 (7)	0.67063 (4)	0.02683 (15)
S3	0.094456 (17)	-0.09859 (7)	0.70721 (4)	0.02913 (15)
O1	-0.20211 (5)	0.5638 (2)	0.51776 (12)	0.0445 (4)
O2	-0.13702 (5)	0.6722 (2)	0.60383 (12)	0.0418 (4)
O3	0.05762 (5)	-0.4287 (2)	0.68332 (12)	0.0405 (4)
O4	-0.13468 (5)	0.3962 (2)	0.50138 (10)	0.0308 (4)
N2	0.01393 (5)	-0.1767 (2)	0.63961 (12)	0.0252 (4)
C1	-0.17634 (6)	0.3654 (3)	0.65777 (15)	0.0255 (5)
C2	-0.15800 (7)	0.3921 (3)	0.74340 (16)	0.0312 (5)
H2	-0.1378	0.4924	0.7540	0.037*
C3	-0.16952 (7)	0.2713 (4)	0.81257 (16)	0.0379 (6)
H3	-0.1574	0.2903	0.8716	0.045*
C4	-0.19842 (7)	0.1220 (4)	0.79851 (17)	0.0388 (6)
C5	-0.21578 (7)	0.0968 (3)	0.71170 (18)	0.0402 (6)
H5	-0.2353	-0.0057	0.7007	0.048*
C6	-0.20530 (7)	0.2173 (3)	0.64109 (16)	0.0324 (5)
H6	-0.2177	0.1993	0.5822	0.039*
C7	-0.21083 (9)	-0.0087 (5)	0.8753 (2)	0.0632 (9)
H7A	-0.2430	-0.0261	0.8761	0.095*
H7B	-0.2012	0.0460	0.9333	0.095*
H7C	-0.1964	-0.1311	0.8664	0.095*
C8	-0.09673 (6)	0.3042 (3)	0.53602 (14)	0.0249 (5)
C9	-0.05942 (6)	0.4055 (3)	0.55942 (15)	0.0287 (5)
H9	-0.0588	0.5390	0.5535	0.034*
C10	-0.02302 (7)	0.3088 (3)	0.59164 (15)	0.0275 (5)
H10	0.0028	0.3762	0.6091	0.033*
C11	-0.02389 (6)	0.1121 (3)	0.59875 (13)	0.0218 (4)
C12	-0.06182 (6)	0.0141 (3)	0.57370 (14)	0.0245 (4)
H12	-0.0625	-0.1197	0.5780	0.029*

supplementary materials

C13	-0.09845 (7)	0.1102 (3)	0.54266 (14)	0.0264 (5)
H13	-0.1245	0.0437	0.5261	0.032*
C14	0.01431 (6)	0.0060 (3)	0.63420 (13)	0.0224 (4)
C15	0.05148 (7)	-0.2615 (3)	0.67444 (15)	0.0282 (5)

Atomic displacement parameters (\AA^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
S1	0.0248 (3)	0.0286 (3)	0.0401 (3)	0.0061 (2)	0.0028 (2)	0.0039 (3)
S2	0.0256 (3)	0.0214 (3)	0.0335 (3)	0.0013 (2)	-0.0051 (2)	-0.0017 (2)
S3	0.0264 (3)	0.0249 (3)	0.0361 (3)	0.0036 (2)	-0.0070 (2)	-0.0011 (2)
O1	0.0302 (8)	0.0534 (11)	0.0498 (11)	0.0164 (8)	-0.0020 (8)	0.0137 (9)
O2	0.0383 (9)	0.0240 (8)	0.0631 (11)	-0.0013 (7)	0.0100 (9)	-0.0006 (8)
O3	0.0388 (9)	0.0220 (8)	0.0607 (11)	0.0036 (7)	-0.0145 (9)	0.0032 (8)
O4	0.0254 (7)	0.0375 (9)	0.0297 (8)	0.0089 (6)	-0.0002 (7)	0.0033 (7)
N2	0.0254 (9)	0.0224 (9)	0.0279 (10)	0.0009 (7)	-0.0009 (8)	0.0009 (8)
C1	0.0181 (10)	0.0261 (11)	0.0322 (11)	0.0028 (8)	0.0013 (9)	-0.0050 (9)
C2	0.0237 (11)	0.0336 (12)	0.0362 (13)	-0.0027 (9)	-0.0021 (10)	-0.0091 (10)
C3	0.0297 (12)	0.0543 (16)	0.0296 (12)	0.0033 (11)	-0.0023 (11)	-0.0029 (11)
C4	0.0259 (12)	0.0476 (15)	0.0428 (14)	0.0008 (10)	0.0059 (11)	0.0102 (12)
C5	0.0270 (12)	0.0368 (13)	0.0566 (16)	-0.0108 (10)	0.0000 (11)	0.0002 (12)
C6	0.0255 (11)	0.0347 (12)	0.0369 (13)	-0.0022 (10)	-0.0040 (10)	-0.0060 (11)
C7	0.0435 (15)	0.081 (2)	0.065 (2)	-0.0094 (15)	0.0105 (15)	0.0295 (17)
C8	0.0214 (10)	0.0319 (12)	0.0215 (10)	0.0066 (9)	0.0007 (9)	0.0013 (9)
C9	0.0280 (11)	0.0229 (11)	0.0350 (12)	0.0024 (9)	0.0036 (10)	0.0027 (10)
C10	0.0221 (10)	0.0275 (11)	0.0329 (12)	-0.0001 (9)	-0.0001 (9)	-0.0009 (10)
C11	0.0230 (10)	0.0237 (11)	0.0186 (10)	0.0030 (8)	0.0034 (8)	-0.0004 (8)
C12	0.0259 (10)	0.0230 (11)	0.0247 (10)	-0.0002 (8)	0.0027 (9)	-0.0016 (9)
C13	0.0238 (10)	0.0289 (12)	0.0266 (11)	-0.0004 (9)	0.0013 (9)	-0.0017 (9)
C14	0.0223 (10)	0.0263 (11)	0.0185 (10)	0.0002 (8)	0.0030 (8)	-0.0025 (9)
C15	0.0293 (11)	0.0252 (12)	0.0300 (11)	0.0001 (9)	-0.0028 (10)	-0.0007 (9)

Geometric parameters (\AA , $^\circ$)

S1—O1	1.4204 (16)	C4—C7	1.506 (3)
S1—O2	1.4229 (16)	C5—H5	0.9500
S1—O4	1.6069 (15)	C5—C6	1.380 (3)
S1—C1	1.746 (2)	C6—H6	0.9500
S2—S3	2.0325 (7)	C7—H7A	0.9800
S2—C14	1.7506 (19)	C7—H7B	0.9800
S3—C15	1.803 (2)	C7—H7C	0.9800
O3—C15	1.206 (2)	C8—C9	1.381 (3)
O4—C8	1.414 (2)	C8—C13	1.379 (3)
N2—C14	1.297 (2)	C9—H9	0.9500
N2—C15	1.383 (3)	C9—C10	1.380 (3)
C1—C2	1.387 (3)	C10—H10	0.9500
C1—C6	1.388 (3)	C10—C11	1.398 (3)
C2—H2	0.9500	C11—C12	1.390 (3)
C2—C3	1.373 (3)	C11—C14	1.473 (3)

C3—H3	0.9500	C12—H12	0.9500
C3—C4	1.387 (3)	C12—C13	1.378 (3)
C4—C5	1.390 (3)	C13—H13	0.9500
O1—S1—O2	119.95 (10)	C4—C7—H7B	109.5
O1—S1—O4	103.00 (9)	C4—C7—H7C	109.5
O1—S1—C1	110.51 (10)	H7A—C7—H7B	109.5
O2—S1—O4	108.89 (9)	H7A—C7—H7C	109.5
O2—S1—C1	109.88 (10)	H7B—C7—H7C	109.5
O4—S1—C1	103.08 (9)	C9—C8—O4	120.89 (18)
C14—S2—S3	93.04 (7)	C13—C8—O4	116.98 (18)
C15—S3—S2	94.93 (7)	C13—C8—C9	122.11 (19)
C8—O4—S1	118.39 (13)	C8—C9—H9	120.7
C14—N2—C15	116.67 (17)	C10—C9—C8	118.61 (19)
C2—C1—S1	119.67 (16)	C10—C9—H9	120.7
C2—C1—C6	121.0 (2)	C9—C10—H10	119.8
C6—C1—S1	119.29 (17)	C9—C10—C11	120.38 (19)
C1—C2—H2	120.6	C11—C10—H10	119.8
C3—C2—C1	118.9 (2)	C10—C11—C14	121.35 (18)
C3—C2—H2	120.6	C12—C11—C10	119.56 (18)
C2—C3—H3	119.2	C12—C11—C14	119.07 (17)
C2—C3—C4	121.7 (2)	C11—C12—H12	119.9
C4—C3—H3	119.2	C13—C12—C11	120.30 (19)
C3—C4—C5	118.2 (2)	C13—C12—H12	119.9
C3—C4—C7	121.0 (2)	C8—C13—H13	120.5
C5—C4—C7	120.9 (2)	C12—C13—C8	119.03 (19)
C4—C5—H5	119.3	C12—C13—H13	120.5
C6—C5—C4	121.5 (2)	N2—C14—S2	120.98 (15)
C6—C5—H5	119.3	N2—C14—C11	121.59 (17)
C1—C6—H6	120.7	C11—C14—S2	117.42 (14)
C5—C6—C1	118.7 (2)	O3—C15—S3	119.25 (16)
C5—C6—H6	120.7	O3—C15—N2	126.36 (19)
C4—C7—H7A	109.5	N2—C15—S3	114.38 (15)

Fig. 1

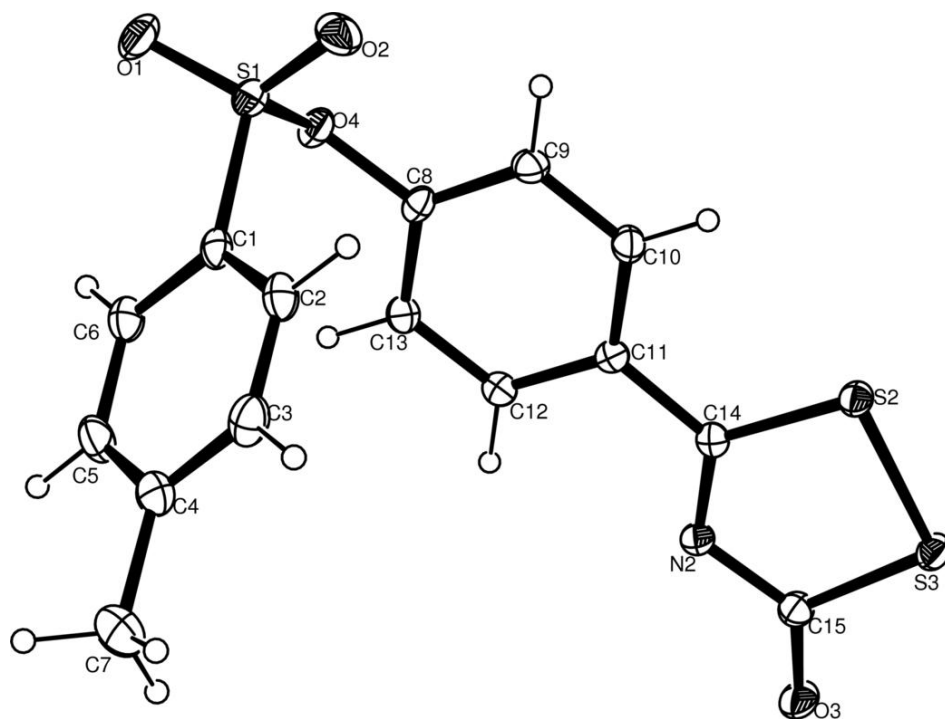


Fig. 2

