5-(2,6-Dimethoxyphenoxy)-2-methyl-sulfanylmethyl-2*H*-tetrazole

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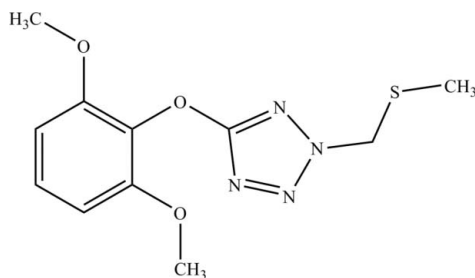
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In the title molecule, $C_{11}H_{14}N_4O_3S$, the tetrazole and benzene rings are nearly perpendicular to each other, forming a dihedral angle of 104.93 (14)°. The crystal packing exhibits weak intermolecular C—H···O hydrogen bonds.

Related literature

For a related crystal structure, see: Dabbagh *et al.* (2005).

Experimental

Crystal data

$C_{11}H_{14}N_4O_3S$	V = 1336.45 (9) Å^3
M_r = 282.32	Z = 4
Orthorhombic, $Pca2_1$	Mo $K\alpha$ radiation
a = 12.1795 (5) Å	μ = 0.25 mm^{-1}
b = 11.0809 (4) Å	T = 173 K
c = 9.9026 (4) Å	0.50 × 0.25 × 0.10 mm

Data collection

Bruker X8 APEXII diffractometer	7950 measured reflections
Absorption correction: multi-scan (*SADABS*; Bruker, 2008)	2873 independent reflections
T_{min} = 0.871, T_{max} = 0.975	2670 reflections with $I > 2\sigma(I)$
	R_{int} = 0.020

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.026	H-atom parameters constrained
$wR(F^2)$ = 0.067	$\Delta\rho_{max}$ = 0.18 e Å^{-3}
S = 1.08	$\Delta\rho_{min}$ = −0.15 e Å^{-3}
2873 reflections	Absolute structure: Flack (1983), 1220 Friedel pairs
175 parameters	Flack parameter: 0.06 (6)
1 restraint	

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

D—H···A	D—H	H···A	D···A	D—H···A
C10—H10B···O1^i	0.99	2.45	3.4277 (19)	170
C11—H11C···O3^i	0.98	2.45	3.421 (2)	172
C11—H11B···O1^{ii}	0.98	2.52	3.471 (2)	163
C10—H10A···O2^{iii}	0.99	2.52	3.312 (2)	137

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

Data collection: *APEX2* (Bruker, 2008); cell refinement: *APEX2*; data reduction: *SAINT* (Bruker, 2008); program(s) used to solve structure: *SIR* (Altomare *et al.*, 1999); program(s) used to refine structure: *SHELXTL* (Sheldrick, 2008); molecular graphics: *ORTEP-3* (Farrugia, 1997); software used to prepare material for publication: *SHELXTL*.

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supplementary materials

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Comment

In continuation of our structural study of tetrazole derivatives (Dabbagh *et al.*, 2005) we report herein the structure of the title compound, (I).

In (I) (Fig. 1), the methylsulfanylmethylation proceeded at N2 atom on tetrazole ring. Because of the conjugation of O3 with tetrazole ring the bond distance O3—C9 [1.3388 (17) Å] is obviously shorter than O3—C1 [1.4062 (15) Å]. A similar effect has been found in 5-(4-nitrophenoxy)-1-methylsulfanylmethyl-1*H*-tetrazole (Dabbagh *et al.*, 2005). Tetrazole ring in (I) is planar, and 2,6-dimethoxyphenoxy group deviates from the tetrazole ring plane so torsion angles C6—C1—O3—C9 and C1—O3—C9—N4 are -79.52 (17)° and -9.6 (2)°, respectively. The torsion angle O3—C1—C2—C3 of 174.02 (13)° implies steric interaction between the benzene and tetrazole rings. The S1—C10 bond [1.792 (2) Å] is slightly shorter than C11—S1 [1.798 (2) Å] in methylsulfanylmethyl group.

The crystal packing exhibits weak intermolecular C—H⋯O hydrogen bonds (Table 1).

Experimental

Dry DMSO, 5 ml, was added dropwise over a period of 30 min to a solution of 0.143 g of compound in 4 ml of acetic anhydride. The mixture was stirred for 40 h at 45–50°C, excess DMSO and acetic anhydride were removed under reduced pressure, and the residue was washed with several 2–3-ml portions of water. The precipitate was dissolved in 20 ml of methylene chloride, the solution was dried over calcium chloride and evaporated, and the residue was separated by column chromatography on silica gel to isolate compound. Yield 20%. IR spectrum (KBr), ν, cm^{-1}: 3050, 2975, 1590, 1530, 1390, 1370, 1300, 1260, 1180, 1110, 760. ^{1}H NMR (300 MHz, DMSO-d_6), δ: 7.14 (t, 1H, J = 10 Hz), 6.65 (d, 2H, J = 10 Hz), 5.48 (s, 2H), 3.71 (s, 6H), 2.17 (s, 3H). 13 C NMR (75 MHz, DMSO-d6), δ: 177.50, 152.29, 131.80, 126.74, 105.44, 78.88, 56.20, 56.03, 15.48. Mass spectrum (EI), *m/z* (Irel, %) 284 (0.6) $[M + 2]^+$, 282 (15) $[M]^+$, 281 (20), 236 (83), 151 (73), 140 (39), 107 (59), 43 (100).

Refinement

All H atoms were geometrically positioned (C—H 0.95–0.99 Å), and allowed to ride on their parent atoms, with $U_{iso}(H)$ = 1.2–1.5 $U_{eq}(C)$.
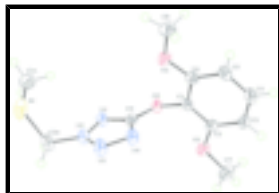
Figures

Fig. 1. The molecular structure of the title compound showing the atomic numbering and 50% probability displacement ellipsoids.

5-(2,6-Dimethoxyphenoxy)-2-methylsulfanylmethyl-2*H*-tetrazole

Crystal data

$C_{11}H_{14}N_4O_3S$	$F_{000} = 592$
$M_r = 282.32$	$D_x = 1.403$ Mg m^{-3}
Orthorhombic, $Pca2_1$	Mo $K\alpha$ radiation $\lambda = 0.71073$ Å
Hall symbol: P 2c -2ac	Cell parameters from 4562 reflections
$a = 12.1795 (5)$ Å	$\theta = 2.8–27.8º$
$b = 11.0809 (4)$ Å	$\mu = 0.25$ mm^{-1}
$c = 9.9026 (4)$ Å	$T = 173$ K
$V = 1336.45 (9)$ Å^3	Irregular, colourless
$Z = 4$	$0.50 \times 0.25 \times 0.10$ mm

Data collection

Bruker X8 APEXII diffractometer	2873 independent reflections
Radiation source: fine-focus sealed tube	2670 reflections with $I > 2\sigma(I)$
Monochromator: graphite	$R_{int} = 0.020$
$T = 173$ K	$\theta_{max} = 28.0º$
Area–detector scans	$\theta_{min} = 1.8º$
Absorption correction: multi-scan (SADABS; Bruker, 2008)	$h = -16 \rightarrow 15$
$T_{min} = 0.871$, $T_{max} = 0.975$	$k = -14 \rightarrow 14$
7950 measured reflections	$l = -12 \rightarrow 12$

Refinement

Refinement on F^2	Hydrogen site location: inferred from neighbouring sites
Least-squares matrix: full	H-atom parameters constrained
$R[F^2 > 2\sigma(F^2)] = 0.026$	$w = 1/[\sigma^2(F_o^2) + (0.0323P)^2 + 0.1529P]$ where $P = (F_o^2 + 2F_c^2)/3$
$wR(F^2) = 0.067$	$(\Delta/\sigma)_{max} = 0.002$
$S = 1.08$	$\Delta\rho_{max} = 0.18$ e Å^{-3}
2873 reflections	$\Delta\rho_{min} = -0.15$ e Å^{-3}

175 parameters	Extinction correction: none
1 restraint	Absolute structure: Flack (1983), 1220 Friedel pairs
Primary atom site location: structure-invariant direct methods	Flack parameter: 0.06 (6)
Secondary atom site location: difference Fourier map	

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.

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 ($Å^2$)

	x	y	z	U_{iso}*/U_{eq}
C1	0.56871 (11)	0.77249 (11)	0.88143 (15)	0.0219 (3)
C2	0.65815 (12)	0.75439 (12)	0.96546 (15)	0.0236 (3)
C3	0.74485 (12)	0.83700 (14)	0.96021 (18)	0.0282 (3)
H3	0.8075	0.8264	1.0161	0.034*
C4	0.73884 (12)	0.93401 (13)	0.87333 (17)	0.0296 (3)
H4	0.7984	0.9893	0.8700	0.035*
C5	0.64873 (13)	0.95357 (13)	0.79062 (17)	0.0279 (3)
H5	0.6460	1.0215	0.7322	0.033*
C6	0.56210 (12)	0.87112 (12)	0.79505 (15)	0.0244 (3)
C7	0.74036 (16)	0.64203 (15)	1.1436 (2)	0.0417 (4)
H7A	0.8086	0.6213	1.0966	0.063*
H7B	0.7216	0.5776	1.2073	0.063*
H7C	0.7503	0.7179	1.1930	0.063*
C8	0.45668 (14)	0.97946 (14)	0.6331 (2)	0.0388 (4)
H8A	0.4621	1.0539	0.6861	0.058*
H8B	0.3848	0.9761	0.5887	0.058*
H8C	0.5148	0.9782	0.5646	0.058*
C9	0.39202 (11)	0.70979 (11)	0.94053 (14)	0.0213 (3)
C10	0.12256 (13)	0.65157 (14)	1.0324 (2)	0.0334 (3)
H10A	0.0972	0.6804	1.1217	0.040*
H10B	0.1216	0.5622	1.0341	0.040*
C11	0.05905 (16)	0.59888 (17)	0.77160 (19)	0.0451 (4)
H11A	0.1360	0.6080	0.7445	0.068*
H11B	0.0113	0.6157	0.6941	0.068*
H11C	0.0464	0.5162	0.8031	0.068*
N1	0.30621 (9)	0.63680 (9)	0.92891 (14)	0.0250 (3)
N2	0.23531 (10)	0.69247 (10)	1.01082 (14)	0.0266 (3)
N3	0.27358 (11)	0.79161 (11)	1.06706 (16)	0.0328 (3)

N4	0.37580 (11)	0.80385 (11)	1.02308 (15)	0.0298 (3)
O1	0.65369 (8)	0.65558 (9)	1.04724 (12)	0.0297 (2)
O2	0.46880 (9)	0.87735 (9)	0.72061 (12)	0.0316 (3)
O3	0.48597 (8)	0.68410 (8)	0.87649 (10)	0.0231 (2)
S1	0.02856 (3)	0.70315 (4)	0.90562 (6)	0.04717 (14)

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.0180 (6)	0.0215 (6)	0.0261 (8)	−0.0025 (5)	0.0015 (6)	−0.0033 (5)
C2	0.0226 (7)	0.0245 (6)	0.0237 (8)	0.0029 (5)	0.0009 (6)	−0.0048 (5)
C3	0.0182 (7)	0.0354 (7)	0.0310 (9)	−0.0003 (5)	−0.0019 (6)	−0.0091 (6)
C4	0.0239 (7)	0.0294 (7)	0.0354 (9)	−0.0069 (5)	0.0059 (6)	−0.0086 (6)
C5	0.0292 (7)	0.0237 (6)	0.0308 (8)	−0.0041 (5)	0.0051 (6)	−0.0009 (6)
C6	0.0239 (7)	0.0249 (6)	0.0243 (8)	0.0007 (5)	0.0008 (6)	−0.0018 (5)
C7	0.0426 (10)	0.0425 (8)	0.0400 (10)	0.0014 (8)	−0.0177 (9)	0.0046 (8)
C8	0.0417 (9)	0.0323 (8)	0.0425 (10)	−0.0001 (7)	−0.0069 (9)	0.0140 (7)
C9	0.0203 (6)	0.0197 (5)	0.0238 (8)	0.0018 (5)	−0.0028 (6)	0.0026 (5)
C10	0.0221 (7)	0.0346 (7)	0.0436 (9)	−0.0046 (6)	0.0078 (7)	−0.0024 (7)
C11	0.0453 (11)	0.0475 (10)	0.0423 (11)	−0.0133 (8)	−0.0079 (9)	0.0117 (8)
N1	0.0201 (5)	0.0251 (5)	0.0298 (7)	−0.0018 (4)	0.0008 (5)	−0.0012 (5)
N2	0.0209 (6)	0.0257 (6)	0.0332 (8)	−0.0011 (5)	0.0028 (5)	−0.0010 (5)
N3	0.0267 (6)	0.0288 (6)	0.0430 (9)	−0.0028 (5)	0.0053 (6)	−0.0072 (5)
N4	0.0244 (6)	0.0267 (6)	0.0382 (8)	−0.0030 (5)	0.0022 (6)	−0.0059 (5)
O1	0.0279 (6)	0.0299 (5)	0.0312 (6)	0.0007 (4)	−0.0075 (5)	0.0025 (4)
O2	0.0296 (6)	0.0292 (5)	0.0361 (7)	−0.0045 (4)	−0.0102 (5)	0.0098 (4)
O3	0.0187 (5)	0.0204 (4)	0.0302 (6)	−0.0028 (3)	−0.0003 (4)	−0.0023 (4)
S1	0.02395 (18)	0.0424 (2)	0.0752 (4)	0.00636 (16)	−0.0048 (2)	0.0022 (2)

Geometric parameters (Å, °)

C1—O3	1.4062 (15)	C7—H7B	0.9800
C9—O3	1.3388 (17)	C7—H7C	0.9800
N2—C10	1.4618 (19)	C8—O2	1.4330 (18)
S1—C10	1.792 (2)	C8—H8A	0.9800
C6—O2	1.3562 (18)	C8—H8B	0.9800
C2—O1	1.3630 (17)	C8—H8C	0.9800
C1—C2	1.385 (2)	C9—N1	1.3265 (17)
C1—C6	1.390 (2)	C9—N4	1.3393 (18)
C2—C3	1.399 (2)	C10—H10A	0.9900
C3—C4	1.379 (2)	C10—H10B	0.9900
C3—H3	0.9500	C11—S1	1.798 (2)
C4—C5	1.386 (2)	C11—H11A	0.9800
C4—H4	0.9500	C11—H11B	0.9800
C5—C6	1.3964 (19)	C11—H11C	0.9800
C5—H5	0.9500	N1—N2	1.3357 (17)
C7—O1	1.431 (2)	N2—N3	1.3169 (17)
C7—H7A	0.9800	N3—N4	1.3260 (18)

C9—O3—C1	116.61 (10)	O2—C8—H8B	109.5
N2—C10—S1	113.52 (12)	H8A—C8—H8B	109.5
C10—S1—C11	100.37 (9)	O2—C8—H8C	109.5
C2—C1—C6	121.93 (12)	H8A—C8—H8C	109.5
C2—C1—O3	118.92 (12)	H8B—C8—H8C	109.5
C6—C1—O3	118.99 (12)	N1—C9—O3	120.17 (11)
O1—C2—C1	116.22 (12)	N1—C9—N4	114.28 (12)
O1—C2—C3	125.32 (13)	O3—C9—N4	125.50 (12)
C1—C2—C3	118.47 (13)	N2—C10—H10A	108.9
C4—C3—C2	119.57 (14)	S1—C10—H10A	108.9
C4—C3—H3	120.2	N2—C10—H10B	108.9
C2—C3—H3	120.2	S1—C10—H10B	108.9
C3—C4—C5	122.18 (13)	H10A—C10—H10B	107.7
C3—C4—H4	118.9	S1—C11—H11A	109.5
C5—C4—H4	118.9	S1—C11—H11B	109.5
C4—C5—C6	118.51 (14)	H11A—C11—H11B	109.5
C4—C5—H5	120.7	S1—C11—H11C	109.5
C6—C5—H5	120.7	H11A—C11—H11C	109.5
O2—C6—C1	115.02 (12)	H11B—C11—H11C	109.5
O2—C6—C5	125.64 (13)	C9—N1—N2	100.09 (11)
C1—C6—C5	119.33 (14)	N3—N2—N1	114.41 (12)
O1—C7—H7A	109.5	N3—N2—C10	121.96 (13)
O1—C7—H7B	109.5	N1—N2—C10	123.56 (12)
H7A—C7—H7B	109.5	N2—N3—N4	106.18 (12)
O1—C7—H7C	109.5	N3—N4—C9	105.04 (12)
H7A—C7—H7C	109.5	C2—O1—C7	116.81 (12)
H7B—C7—H7C	109.5	C6—O2—C8	117.09 (12)
O2—C8—H8A	109.5		

C6—C1—O3—C9	−79.52 (17)	N4—C9—N1—N2	−0.13 (16)
N4—C9—O3—C1	−9.6 (2)	C9—N1—N2—N3	0.54 (16)
C6—C1—C2—O1	178.73 (13)	C9—N1—N2—C10	177.63 (14)
O3—C1—C2—O1	−5.86 (18)	S1—C10—N2—N3	93.31 (17)
C6—C1—C2—C3	−1.4 (2)	S1—C10—N2—N1	−83.56 (15)
O3—C1—C2—C3	174.02 (13)	N1—N2—N3—N4	−0.76 (18)
O1—C2—C3—C4	−179.56 (14)	C10—N2—N3—N4	−177.90 (15)
C1—C2—C3—C4	0.6 (2)	N2—N3—N4—C9	0.60 (17)
C2—C3—C4—C5	0.5 (2)	N1—C9—N4—N3	−0.31 (17)
C3—C4—C5—C6	−0.7 (2)	O3—C9—N4—N3	−177.81 (14)
C2—C1—C6—O2	−179.07 (13)	C1—C2—O1—C7	−174.19 (14)
O3—C1—C6—O2	5.51 (18)	C3—C2—O1—C7	5.9 (2)
C2—C1—C6—C5	1.2 (2)	C1—C6—O2—C8	178.13 (14)
O3—C1—C6—C5	−174.26 (13)	C5—C6—O2—C8	−2.1 (2)
C4—C5—C6—O2	−179.84 (14)	N1—C9—O3—C1	173.03 (12)
C4—C5—C6—C1	−0.1 (2)	C2—C1—O3—C9	104.93 (14)
O3—C9—N1—N2	177.52 (12)	N2—C10—S1—C11	78.85 (13)

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*

C10—H10B···O1^{i}	0.99	2.45	3.4277 (19)	170
C11—H11C···O3^{i}	0.98	2.45	3.421 (2)	172
C11—H11B···O1^{ii}	0.98	2.52	3.471 (2)	163
C10—H10A···O2^{iii}	0.99	2.52	3.312 (2)	137

Symmetry codes: (i) $x-1/2$, $-y+1$, z; (ii) $-x+1/2$, y, $z-1/2$; (iii) $-x+1/2$, y, $z+1/2$.

Fig. 1