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3-(*p*-Anisyl)sydnone

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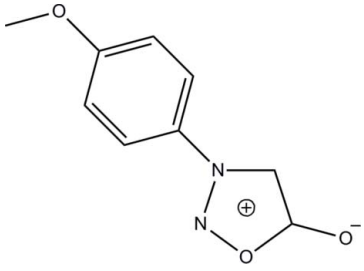
Key indicators: single-crystal X-ray study; T = 100 K; mean σ(C–C) = 0.002 Å; R factor = 0.037; wR factor = 0.100; data-to-parameter ratio = 11.1.

In the title sydnone compound [systematic name: 3-(4-methoxyphenyl)-1,2,3-oxadiazol-3-ium-5-olate], $C_9H_8N_2O_3$, the essentially planar oxadiazole ring [maximum deviation = 0.005 (1) Å] is inclined at a dihedral angle of 30.32 (8)° with respect to the benzene ring. In the crystal, adjacent molecules are interconnected by intermolecular C—H···O hydrogen bonds into sheets lying parallel to (100). Weak intermolecular π–π interactions [centroid–centroid distance = 3.5812 (8) Å] further stabilize the crystal packing.

Related literature

For general background to and applications of the title sydnone compound, see: Hegde *et al.* (2008); Kalluraya & Rahiman (1997); Kalluraya *et al.* (2002); Rai *et al.* (2008). For closely related sydnone structures, see: Goh *et al.* (2010*a*,*b*,*c*). For the stability of the temperature controller used in the data collection, see: Cosier & Glazer (1986).



Experimental

Crystal data

$C_9H_8N_2O_3$ M_r = 192.17
Orthorhombic, $P2_12_12_1$
a = 7.0505 (2) Å
b = 9.8220 (3) Å
c = 12.0934 (3) Å
V = 837.47 (4) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.12 mm^{-1}
T = 100 K
0.56 × 0.15 × 0.14 mm

Data collection

Bruker SMART APEXII CCD area-detector diffractometer
Absorption correction: multi-scan (*SADABS*; Bruker, 2009)
T_{min} = 0.938, T_{max} = 0.984
7384 measured reflections
1760 independent reflections
1577 reflections with $I > 2\sigma(I)$
R_{int} = 0.031

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.037
$wR(F^2)$ = 0.100
S = 1.06
1760 reflections
159 parameters
All H-atom parameters refined
$\Delta\rho_{max}$ = 0.32 e Å^{-3}
$\Delta\rho_{min}$ = −0.26 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
C1—H1A···O2^{i}	1.00 (2)	2.59 (2)	3.5441 (19)	159.1 (15)
C7—H7A···O3^{ii}	0.98 (2)	2.42 (2)	3.3814 (18)	165.7 (17)

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

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

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: RZ2524).

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3-(*p*-Anisyl)sydnone

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S1. Comment

Sydnones constitute a well-defined class of mesoionic compounds consisting of 1,2,3-oxadiazole ring system. The study of sydnones still remains a field of interest because of their electronic structure and also because of the varied types of biological activity displayed by some of them (Rai *et al.*, 2008). Sydnone derivatives were found to exhibit promising anti-microbial properties (Hegde *et al.*, 2008). Sydnones are synthesized by the cyclodehydration of *N*-nitroso-*N*-substituted amino acids using acetic anhydride. The sydnones unsubstituted in the 4-position readily undergo typical electrophilic substitution reaction namely formylation (Kalluraya & Rahiman, 1997) and acetylation (Kalluraya *et al.*, 2002).

In the title sydnone compound (Fig. 1), the 1,2,3-oxadiazole ring with atom sequence C7/C8/O1/N1/N2 is essentially planar, with a maximum deviation of 0.005 (1) Å at atom O1. The whole molecule is not planar, as indicated by the dihedral angle formed between the 1,2,3-oxadiazole and phenyl rings of 30.32 (8)°. Comparing with those previously reported structures with substitution at the 4-position of the sydnone moiety (Goh *et al.*, 2010*a*,*c*), the exocyclic C8—O2 bond length [1.2174 (8) Å] is longer than the respective values observed [1.193 (3) and 1.2089 (9) Å]. All other geometric parameters agree well with those observed in closely related sydnone structures (Goh *et al.*, 2010*a*,*b*,*c*).

In the crystal packing, intermolecular C1—H1A···O2 and C7—H7A···O3 hydrogen bonds (Table 1) link adjacent molecules into two-dimensional sheets lying parallel to the *bc* plane (Fig. 2). The crystal packing is further stabilized by weak intermolecular π–π interactions [*Cg*1···*Cg*2 = 3.5812 (8) Å; symmetry code: x-1/2, -y+3/2, -z+1] involving the 1,2,3-oxadiazole and phenyl rings.

S2. Experimental

N-Nitroso-*p*-methoxyanilinoacetic acid (0.01 mol) was heated with acetic acid anhydride (0.5 mol) on a water bath for 2–3 h. The reaction mixture was kept aside at room temperature for overnight. It was then poured into ice-cold water. The obtained solid was dried and crystallized from benzene. Single crystals suitable for X-ray analysis were obtained from ethanol by slow evaporation.

S3. Refinement

All H atoms were located from difference Fourier map and allowed to refine freely with range of C—H = 0.95 (2) – 1.00 (2) Å. In the absence of significant anomalous dispersion, 1266 Friedel pairs were merged in the final refinement.

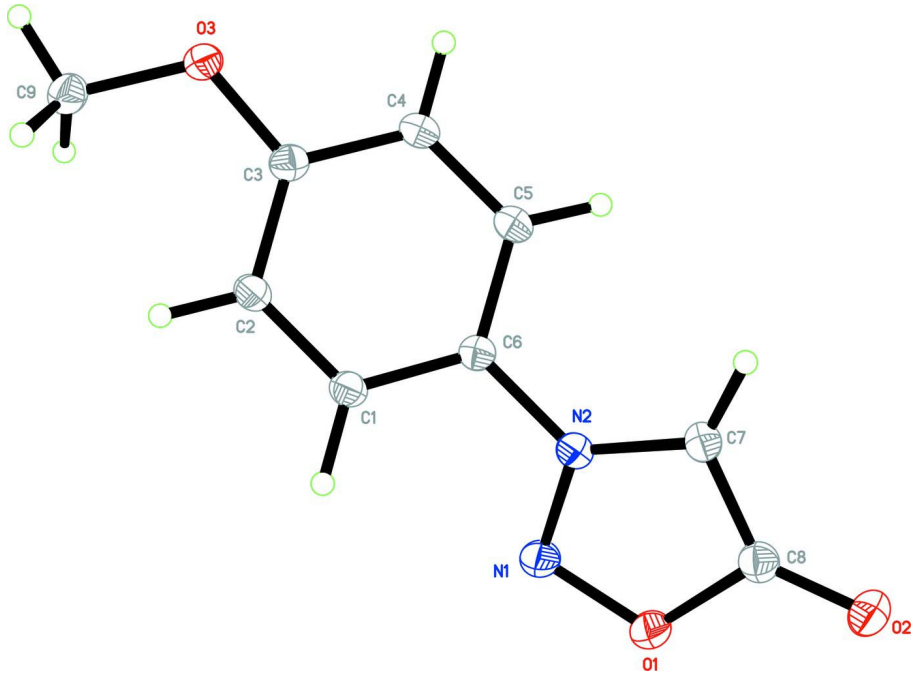

Figure 1

The molecular structure of the title sydnone compound, showing 50 % probability displacement ellipsoids for non-H atoms and the atom-numbering scheme.

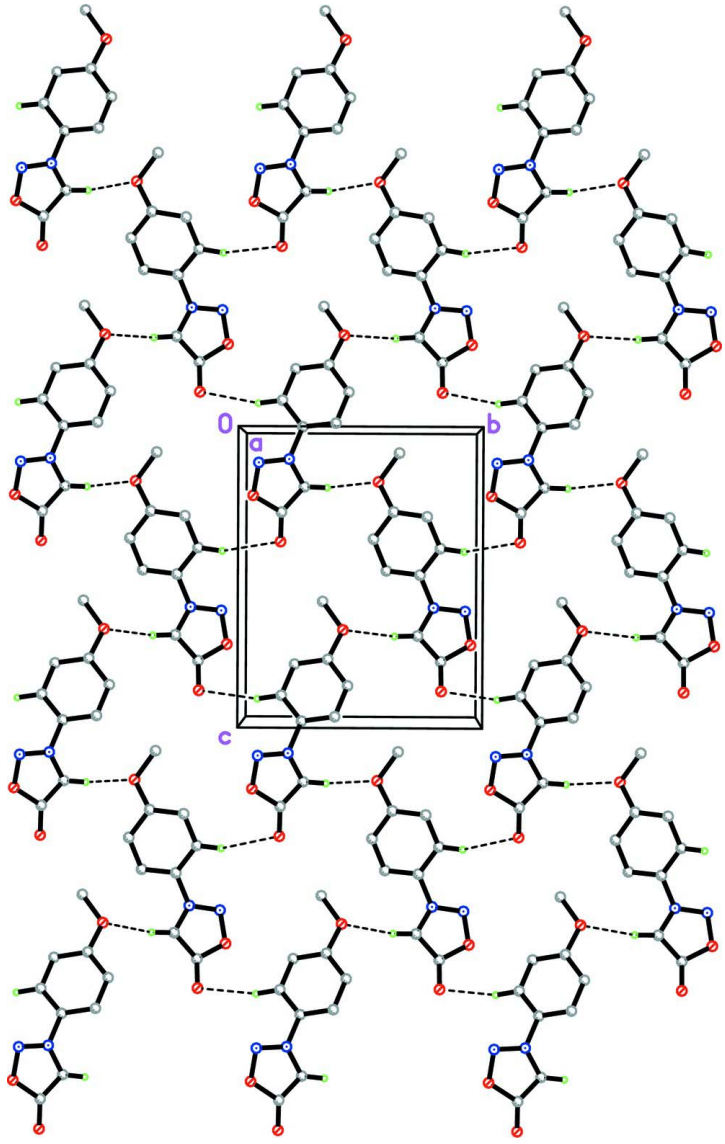

Figure 2

The crystal structure of the title compound, viewed along the *a* axis, showing a two-dimensional sheet parallel to the *bc* plane. H atoms not involved in intermolecular hydrogen bonds (dashed lines) have been omitted for clarity.

3-(4-methoxyphenyl)-1,2,3-oxadiazol-3-ium-5-olate

Crystal data

$C_9H_8N_2O_3$
M_r = 192.17
Orthorhombic, $P2_12_12_1$
Hall symbol: P 2ac 2ab
a = 7.0505 (2) Å
b = 9.8220 (3) Å
c = 12.0934 (3) Å
V = 837.47 (4) Å^3
Z = 4
$F(000)$ = 400
D_x = 1.524 Mg m^{-3}
Mo $K\alpha$ radiation, λ = 0.71073 Å
Cell parameters from 2851 reflections
θ = 3.4–32.4°
μ = 0.12 mm^{-1}
T = 100 K
Needle, yellow
0.56 × 0.15 × 0.14 mm

Data collection

Bruker SMART APEXII CCD area-detector diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Bruker, 2009)
T_{min} = 0.938, T_{max} = 0.984
7384 measured reflections
1760 independent reflections
1577 reflections with $I > 2\sigma(I)$
R_{int} = 0.031
θ_{max} = 32.7°, θ_{min} = 2.7°
h = −10→10
k = −14→14
l = −18→18

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.037
$wR(F^2)$ = 0.100
S = 1.06
1760 reflections
159 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
All H-atom parameters refined
$w = 1/[\sigma^2(F_o^2) + (0.0608P)^2 + 0.0718P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.32 e Å^{-3}
$\Delta\rho_{min}$ = −0.26 e Å^{-3}

Special details

Experimental. The crystal was placed in the cold stream of an Oxford Cryosystems Cobra open-flow nitrogen cryostat (Cosier & Glazer, 1986) operating at 100.0 (1)K.

Geometry. All esds (except the esd in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell esds are taken into account individually in the estimation of esds in distances, angles and torsion angles; correlations between esds in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell esds is used for estimating esds 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 (Å²)

	x	y	z	U_{iso}*/U_{eq}
O1	0.08268 (18)	0.95246 (11)	0.72517 (9)	0.0204 (2)
O2	0.15734 (19)	0.83477 (13)	0.88211 (9)	0.0252 (3)
O3	0.12932 (18)	0.57288 (10)	0.18145 (8)	0.0199 (2)
N1	0.0695 (2)	0.92968 (13)	0.61277 (11)	0.0199 (3)
N2	0.11891 (19)	0.80122 (12)	0.60181 (10)	0.0152 (2)
C1	0.1632 (2)	0.82919 (14)	0.40336 (11)	0.0157 (2)
C2	0.1665 (2)	0.77417 (15)	0.29712 (11)	0.0158 (3)
C3	0.1284 (2)	0.63583 (15)	0.28173 (12)	0.0160 (3)
C4	0.0868 (2)	0.55302 (15)	0.37253 (13)	0.0179 (3)
C5	0.0829 (2)	0.60641 (15)	0.47811 (12)	0.0165 (3)
C6	0.1211 (2)	0.74504 (16)	0.49222 (12)	0.0145 (2)
C7	0.1622 (2)	0.73661 (15)	0.69641 (12)	0.0173 (3)
C8	0.1394 (2)	0.83234 (16)	0.78201 (12)	0.0186 (3)
C9	0.1403 (3)	0.65840 (16)	0.08473 (12)	0.0205 (3)

H1A	0.192 (3)	0.928 (2)	0.4147 (15)	0.019 (5)*
H2A	0.194 (3)	0.831 (2)	0.2340 (16)	0.024 (5)*
H4A	0.070 (3)	0.457 (2)	0.3623 (16)	0.025 (5)*
H5A	0.047 (3)	0.5524 (19)	0.5411 (15)	0.015 (5)*
H7A	0.203 (3)	0.641 (2)	0.6995 (17)	0.026 (5)*
H9A	0.124 (3)	0.603 (2)	0.0190 (16)	0.020 (5)*
H9B	0.040 (3)	0.723 (2)	0.0852 (16)	0.023 (5)*
H9C	0.262 (4)	0.708 (3)	0.0800 (19)	0.041 (7)*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.0255 (5)	0.0180 (5)	0.0179 (5)	0.0014 (5)	0.0003 (5)	−0.0028 (4)
O2	0.0316 (6)	0.0267 (5)	0.0172 (5)	−0.0033 (6)	−0.0014 (5)	−0.0011 (4)
O3	0.0290 (6)	0.0155 (5)	0.0151 (5)	0.0016 (4)	−0.0015 (4)	−0.0013 (3)
N1	0.0258 (6)	0.0151 (5)	0.0188 (6)	0.0035 (5)	−0.0003 (5)	−0.0016 (4)
N2	0.0152 (5)	0.0141 (5)	0.0163 (5)	−0.0003 (4)	−0.0002 (4)	0.0006 (4)
C1	0.0168 (6)	0.0136 (5)	0.0167 (6)	−0.0008 (5)	−0.0016 (5)	0.0004 (4)
C2	0.0168 (6)	0.0136 (6)	0.0169 (6)	−0.0011 (5)	−0.0010 (5)	0.0016 (4)
C3	0.0154 (6)	0.0158 (6)	0.0169 (6)	0.0007 (5)	−0.0016 (5)	−0.0013 (4)
C4	0.0213 (7)	0.0130 (5)	0.0195 (6)	−0.0017 (5)	0.0001 (5)	0.0000 (5)
C5	0.0166 (6)	0.0138 (5)	0.0191 (6)	−0.0013 (5)	0.0004 (6)	0.0010 (5)
C6	0.0138 (6)	0.0142 (5)	0.0154 (5)	0.0001 (5)	0.0004 (5)	−0.0003 (4)
C7	0.0190 (6)	0.0167 (6)	0.0164 (6)	−0.0002 (5)	−0.0007 (5)	0.0016 (5)
C8	0.0176 (6)	0.0188 (6)	0.0192 (6)	−0.0014 (6)	−0.0003 (6)	0.0006 (5)
C9	0.0256 (7)	0.0200 (6)	0.0159 (6)	0.0019 (6)	−0.0014 (6)	−0.0002 (5)

Geometric parameters (Å, º)

O1—N1	1.3807 (16)	C2—H2A	0.97 (2)
O1—C8	1.4227 (19)	C3—C4	1.398 (2)
O2—C8	1.2174 (18)	C4—C5	1.381 (2)
O3—C3	1.3613 (17)	C4—H4A	0.95 (2)
O3—C9	1.4421 (18)	C5—C6	1.398 (2)
N1—N2	1.3158 (16)	C5—H5A	0.961 (19)
N2—C7	1.3434 (17)	C7—C8	1.408 (2)
N2—C6	1.4356 (18)	C7—H7A	0.98 (2)
C1—C6	1.388 (2)	C9—H9A	0.97 (2)
C1—C2	1.3940 (19)	C9—H9B	0.95 (2)
C1—H1A	1.00 (2)	C9—H9C	0.99 (3)
C2—C3	1.3976 (19)		
N1—O1—C8	111.11 (12)	C4—C5—C6	118.61 (13)
C3—O3—C9	117.27 (11)	C4—C5—H5A	121.9 (11)
N2—N1—O1	103.69 (12)	C6—C5—H5A	119.4 (11)
N1—N2—C7	115.30 (12)	C1—C6—C5	121.77 (14)
N1—N2—C6	117.68 (12)	C1—C6—N2	119.22 (13)
C7—N2—C6	127.02 (12)	C5—C6—N2	119.00 (13)

C6—C1—C2	119.11 (13)	N2—C7—C8	106.54 (13)
C6—C1—H1A	121.0 (11)	N2—C7—H7A	123.4 (12)
C2—C1—H1A	119.9 (11)	C8—C7—H7A	130.1 (12)
C1—C2—C3	119.76 (13)	O2—C8—C7	137.09 (16)
C1—C2—H2A	120.5 (13)	O2—C8—O1	119.56 (14)
C3—C2—H2A	119.8 (13)	C7—C8—O1	103.34 (12)
O3—C3—C4	115.89 (13)	O3—C9—H9A	109.4 (12)
O3—C3—C2	124.01 (13)	O3—C9—H9B	110.2 (12)
C4—C3—C2	120.10 (13)	H9A—C9—H9B	107.0 (17)
C5—C4—C3	120.65 (13)	O3—C9—H9C	112.3 (15)
C5—C4—H4A	119.4 (12)	H9A—C9—H9C	109.3 (18)
C3—C4—H4A	119.8 (12)	H9B—C9—H9C	108.5 (19)

C8—O1—N1—N2	−0.84 (16)	C4—C5—C6—C1	−0.2 (2)
O1—N1—N2—C7	0.46 (17)	C4—C5—C6—N2	−179.47 (14)
O1—N1—N2—C6	−179.58 (12)	N1—N2—C6—C1	30.90 (19)
C6—C1—C2—C3	−0.2 (2)	C7—N2—C6—C1	−149.14 (15)
C9—O3—C3—C4	169.99 (14)	N1—N2—C6—C5	−149.86 (15)
C9—O3—C3—C2	−10.3 (2)	C7—N2—C6—C5	30.1 (2)
C1—C2—C3—O3	−179.64 (13)	N1—N2—C7—C8	0.09 (18)
C1—C2—C3—C4	0.0 (2)	C6—N2—C7—C8	−179.87 (13)
O3—C3—C4—C5	179.72 (14)	N2—C7—C8—O2	−179.70 (19)
C2—C3—C4—C5	0.0 (2)	N2—C7—C8—O1	−0.58 (16)
C3—C4—C5—C6	0.1 (2)	N1—O1—C8—O2	−179.79 (14)
C2—C1—C6—C5	0.3 (2)	N1—O1—C8—C7	0.89 (17)
C2—C1—C6—N2	179.53 (13)		

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C1—H1*A*···O2^{i}	1.00 (2)	2.59 (2)	3.5441 (19)	159.1 (15)
C7—H7*A*···O3^{ii}	0.98 (2)	2.42 (2)	3.3814 (18)	165.7 (17)

Symmetry codes: (i) −*x*+1/2, −*y*+2, *z*−1/2; (ii) −*x*+1/2, −*y*+1, *z*+1/2.