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4-Acetyl-3-[2-(ethoxycarbonyl)phenyl]-sydnone

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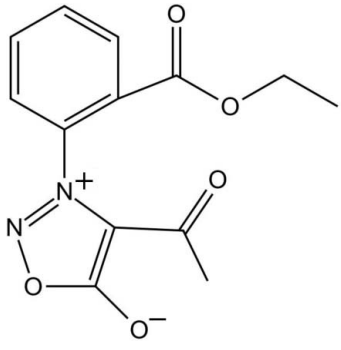
Key indicators: single-crystal X-ray study; T = 173 K; mean σ(C–C) = 0.002 Å; R factor = 0.050; wR factor = 0.121; data-to-parameter ratio = 20.5.

Sydnones, which contain a mesoionic five-membered heterocyclic ring, are more stable if synthesized with an aromatic substutuent at the N3 position. In the title compound {systematic name: 4-acetyl-3-[2-(ethoxycarbonyl)phenyl]-1,2,3-oxadiazol-3-ylium-5-olate}, $C_{13}H_{12}N_2O_5$, the aromatic substitutent is 2-(ethoxycarbonyl)phenyl. Intra- and intermolecular hydrogen bonds are observed. The interplanar angle between the sydnone and benzene rings is 71.94 (8)°. π-ring···carbonyl interactions of 3.2038 (16) Å arise between the sydnone ring and a symmetry-related C=O group.

Related literature

For more information on the sydnone family of compounds, see: Ohta & Kato (1969). For synthesis and structure information, see: Grossie & Turnbull (1992); Grossie *et al.* (2001, 2007); Hope & Thiessen (1969); Hodson & Turnbull (1985); Riddle *et al.* (2004*a*,*b*,*c*); Hanley *et al.* (1976). For stability of the temperature controller used for the data collection, see: Cosier & Glazer (1986).



Experimental

Crystal data

$C_{13}H_{12}N_2O_5$
M_r = 276.25
Monoclinic, $P2_1/n$
a = 11.353 (3) Å
b = 8.093 (2) Å
c = 14.607 (4) Å
β = 112.582 (4)°
V = 1239.1 (6) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.12 mm^{-1}
T = 173 K
0.22 × 0.20 × 0.17 mm

Data collection

Bruker Kappa APEXII diffractometer
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
T_{min} = 0.90, T_{max} = 0.98
13826 measured reflections
3718 independent reflections
2783 reflections with $I > 2\sigma(I)$
R_{int} = 0.038

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.050
$wR(F^2)$ = 0.121
S = 0.96
3718 reflections
181 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.49 e Å^{-3}
$\Delta\rho_{min}$ = −0.38 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
C36—H361···O5^{i}	0.95	2.51	3.253 (2)	136
C40—H401···O41^{ii}	0.97	2.46	3.116 (2)	124
C42—H423···O5	0.96	2.51	3.065 (2)	117

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

Data collection: *APEX2* (Bruker, 2006); cell refinement: *APEX2*; data reduction: *APEX2*; program(s) used to solve structure: *SIR92* (Altomare *et al.*, 1994); program(s) used to refine structure: *CRYSTALS* (Betteridge *et al.*, 2003); molecular graphics: *CAMERON* (Watkin *et al.*, 1996); software used to prepare material for publication: *CRYSTALS*.

The authors acknowledge the diffractometer time granted by A. Hunter, Youngstown State University, USA.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: GG2123).

References

Altomare, A., Cascarano, G., Giacovazzo, C., Guagliardi, A., Burla, M. C., Polidori, G. & Camalli, M. (1994). *J. Appl. Cryst.* **27**, 435.
Betteridge, P. W., Carruthers, J. R., Cooper, R. I., Prout, K. & Watkin, D. J. (2003). *J. Appl. Cryst.* **36**, 1487.
Bruker (2006). *APEX2*. Bruker AXS Inc., Madison, Wisconsin, USA.
Cosier, J. & Glazer, A. M. (1986). *J. Appl. Cryst.* **19**, 105–107.
Grossie, D. A., Sun, L. & Turnbull, K. (2007). *Acta Cryst.* E**63**, o2042–o2043.
Grossie, D. A. & Turnbull, K. (1992). *Acta Cryst.* C**48**, 377–379.
Grossie, D. A., Turnbull, K. & Krein, D. M. (2001). *Acta Cryst.* E**57**, o985–o987.
Hanley, R. N., Ollis, W. D. & Ramsden, C. A. (1976). *J. Chem. Soc. Chem. Commun.* **9**, 306–307.
Hodson, S. J. & Turnbull, K. (1985). *J. Heterocycl. Chem.* **22**, 1223–1227.
Hope, H. & Thiessen, W. E. (1969). *Acta Cryst.* B**25**, 1237–1247.
Ohta, M. & Kato, H. (1969). *Nonbenzenoid Aromatics*, edited by J. P. Snyder, pp. 117–248. New York: Academic Press.
Riddle, G. B., Grossie, D. A. & Turnbull, K. (2004*a*). *Acta Cryst.* E**60**, o977–o978.
Riddle, G. B., Grossie, D. A. & Turnbull, K. (2004*b*). *Acta Cryst.* E**60**, o1568–o1570.
Riddle, G. B., Grossie, D. A. & Turnbull, K. (2004*c*). *Acta Cryst.* E**60**, o258–o259.
Sheldrick, G. M. (1996). *SADABS*. University of Göttingen, Germany.
Watkin, D. J., Prout, C. K. & Pearce, L. J. (1996). *CAMERON*. Chemical Crystallography Laboratory, Oxford, England.

supporting information

Acta Cryst. (2013). E**69**, o1662 [doi:10.1107/S1600536813027207]

4-Acetyl-3-[2-(ethoxycarbonyl)phenyl]sydnone

David Grossie, Leanna Harrison and Kenneth Turnbull

S1. Comment

The bond distances and angles were within expected values. The sydnone ring (O1 – C5) and phenyl ring (C31 – C36) of the structure are planar as expected, with all deviations less than 0.1 Å. The angle between the planes of the sydnone (O1 – C5) and phenyl ring (C31 – C36) is 71.94 (8)°. π-atom interactions are seen between the sydnone ring and a symmetry-related O(5) with a distance of 3.2038 (16) Å. Numerous short intra and inter-molecular contacts are noted within the structure. The potential H bonds in the structure are tabulated below.

S2. Experimental

4-Acetyl-3-(2-ethoxycarbonylphenyl)sydnone was synthesized in 47% yield by heating 3-[2-(ethoxycarbonyl)phenyl]-sydnone, acetic anhydride (5 eq), bismuth trifluoromethanesulfonate (25 mole %), and lithium perchlorate (25 mole %) in acetonitrile (2 ml) in a sealed tube at 140°C for 5 h.

S3. Refinement

The H atoms were all located in a difference map, but those attached to carbon atoms were repositioned geometrically. The H atoms were initially refined with soft restraints on the bond lengths and angles to regularize their geometry (C—H in the range 0.93–0.98, N—H in the range 0.86–0.89 N—H to 0.86 O—H = 0.82 Å) and U_{iso}(H) (in the range 1.2–1.5 times U_{eq} of the parent atom), after which the positions were refined with riding constraints.

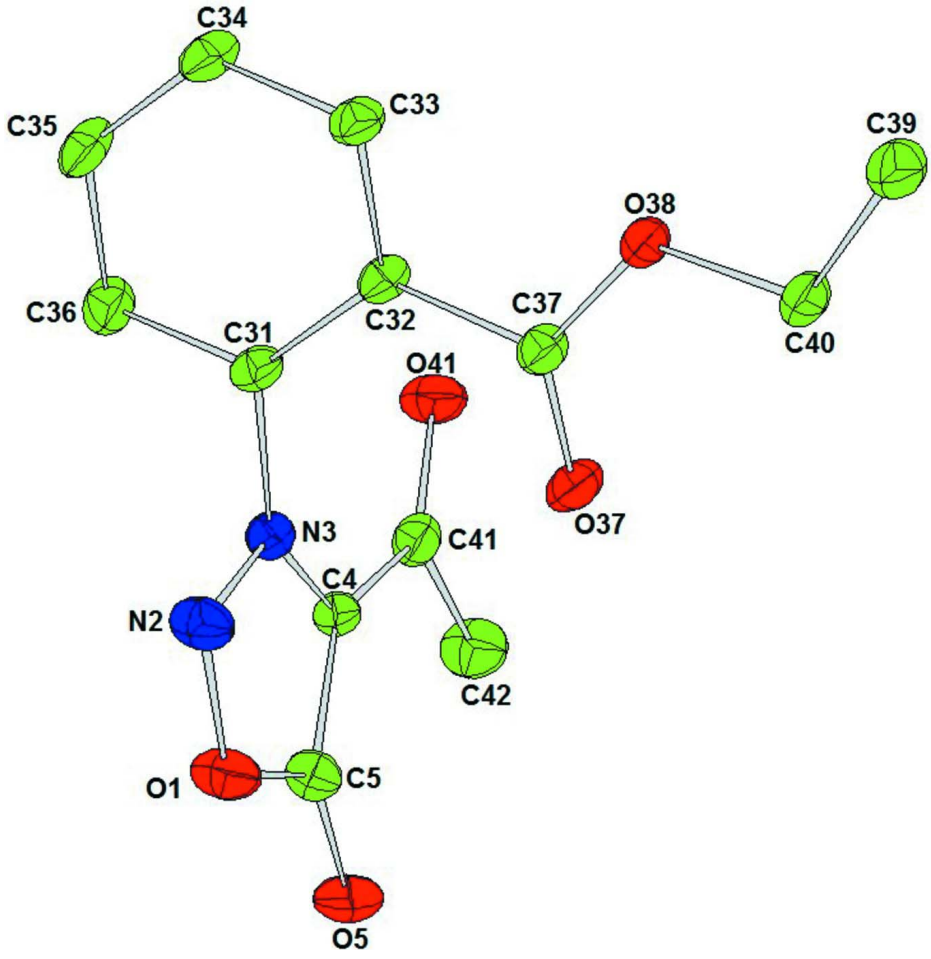


Figure 1

The title compound with displacement ellipsoids drawn at the 50% probability level. H atoms are shown as spheres of arbitary radius.

4-Acetyl-3-[2-(ethoxycarbonyl)phenyl]-1,2,3-oxadiazol-3-ylium-5-olate

Crystal data

$C_{13}H_{12}N_2O_5$
$M_r = 276.25$
Monoclinic, $P2_1/n$
Hall symbol: -P 2yn
$a = 11.353$ (3) Å
$b = 8.093$ (2) Å
$c = 14.607$ (4) Å
$\beta = 112.582$ (4)°
$V = 1239.1$ (6) Å^3
$Z = 4$
$F(000) = 576$
$D_x = 1.481$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 2783 reflections
$\theta = 6$–60°
$\mu = 0.12$ mm^{-1}
$T = 173$ K
Block, colourless
0.22 × 0.20 × 0.17 mm

Data collection

Bruker Kappa APEXII diffractometer
Graphite monochromator
ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)

T_{min} = 0.90, T_{max} = 0.98
13826 measured reflections
3718 independent reflections
2783 reflections with $I > 2\sigma(I)$
R_{int} = 0.038

θ_{max} = 30.6°, θ_{min} = 2.0°
h = −15→15
k = −11→11
l = −20→20

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.050
$wR(F^2)$ = 0.121
S = 0.96
3718 reflections
181 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
Method = Modified Sheldrick $w = 1/[\sigma^2(F^2) + (0.05P)^2 + 0.68P]$, where $P = (\max(F_o^2,0) + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.0001892
$\Delta\rho_{max}$ = 0.49 e Å^{-3}
$\Delta\rho_{min}$ = −0.38 e Å^{-3}

Special details

Experimental. The crystal was placed in the cold stream of an Oxford Cryosystems open-flow nitrogen cryostat (Cosier & Glazer, 1986) with a nominal stability of 0.1 K.

Geometry. Least-squares planes (x,y,z in crystal coordinates) and deviations from them (* indicates atom used to define plane)
Sydnone:O1—C5
0.6390 (6) x - 0.6043 (6) y - 0.4760 (7) z = -0.206 (6) (11)
* 0.005 (1) O1 * 0.003 (1) N2 * -0.010 (1) N3 * 0.012 (1) C4 *- 0.010 (2) C5
0.1671 (6) x - 0.0840 (6) y + 0.9824 (1) z = 5.3188 (17)
Attached phenyl ring: C31–36
* -0.003 (1) C31 * 0.004 (1) C32 * -0.002 (1) C33 * -0.001 (2) C34 * 0.002 (2) C35 * 0.000 (2) C36
Angle to previous plane (with approximate e.s.d.) = 71.94 (8)

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²)

	x	y	z	U_{iso}*/U_{eq}
O41	0.40593 (10)	0.21460 (14)	0.17007 (8)	0.0279
C41	0.49814 (14)	0.30225 (18)	0.18948 (11)	0.0208
C4	0.60889 (13)	0.28357 (17)	0.28115 (10)	0.0175
N3	0.62198 (11)	0.16639 (15)	0.35007 (9)	0.0189
N2	0.72926 (12)	0.16842 (18)	0.42560 (10)	0.0265
O1	0.79450 (10)	0.29896 (15)	0.40892 (9)	0.0296
C5	0.72170 (14)	0.37830 (19)	0.31878 (11)	0.0222
O5	0.76294 (11)	0.49824 (14)	0.29284 (9)	0.0293
C31	0.53744 (14)	0.03347 (17)	0.35081 (11)	0.0190
C36	0.57526 (15)	−0.12260 (18)	0.33667 (12)	0.0242
C35	0.50160 (16)	−0.25590 (18)	0.34078 (12)	0.0267
C34	0.39295 (15)	−0.23188 (19)	0.35861 (12)	0.0253
C33	0.35629 (14)	−0.07463 (18)	0.37287 (11)	0.0218
C32	0.42817 (14)	0.06187 (17)	0.36981 (10)	0.0191
C37	0.39015 (14)	0.23053 (18)	0.38796 (11)	0.0203
O38	0.28355 (10)	0.22771 (13)	0.40506 (8)	0.0243
C40	0.23638 (15)	0.38788 (19)	0.42045 (13)	0.0264

C39	0.12669 (18)	0.3580 (2)	0.45073 (15)	0.0354
O37	0.44855 (11)	0.35433 (13)	0.38822 (9)	0.0296
C42	0.50663 (16)	0.4341 (2)	0.12175 (12)	0.0296
H361	0.6494	−0.1359	0.3224	0.0288*
H351	0.5251	−0.3639	0.3321	0.0328*
H341	0.3441	−0.3213	0.3614	0.0302*
H331	0.2821	−0.0586	0.3855	0.0256*
H402	0.3053	0.4420	0.4736	0.0308*
H401	0.2089	0.4494	0.3586	0.0317*
H393	0.0942	0.4636	0.4613	0.0530*
H392	0.1546	0.2974	0.5114	0.0523*
H391	0.0588	0.2989	0.3991	0.0514*
H423	0.5461	0.5316	0.1577	0.0481*
H422	0.5591	0.3955	0.0874	0.0479*
H421	0.4258	0.4594	0.0745	0.0459*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O41	0.0226 (5)	0.0250 (6)	0.0288 (6)	−0.0055 (4)	0.0018 (4)	0.0058 (5)
C41	0.0232 (7)	0.0167 (6)	0.0234 (7)	0.0017 (5)	0.0099 (6)	0.0009 (5)
C4	0.0181 (6)	0.0150 (6)	0.0211 (6)	−0.0005 (5)	0.0094 (5)	0.0016 (5)
N3	0.0186 (5)	0.0172 (5)	0.0208 (6)	0.0000 (4)	0.0075 (5)	0.0015 (5)
N2	0.0213 (6)	0.0296 (7)	0.0245 (6)	−0.0027 (5)	0.0043 (5)	0.0058 (5)
O1	0.0226 (5)	0.0320 (6)	0.0296 (6)	−0.0068 (5)	0.0048 (5)	0.0055 (5)
C5	0.0211 (7)	0.0237 (7)	0.0231 (7)	−0.0016 (5)	0.0100 (6)	0.0006 (6)
O5	0.0289 (6)	0.0269 (6)	0.0348 (6)	−0.0090 (5)	0.0153 (5)	0.0016 (5)
C31	0.0221 (7)	0.0149 (6)	0.0195 (6)	−0.0020 (5)	0.0075 (5)	0.0025 (5)
C36	0.0276 (7)	0.0192 (7)	0.0280 (8)	0.0029 (6)	0.0130 (6)	0.0031 (6)
C35	0.0367 (9)	0.0134 (6)	0.0319 (8)	0.0022 (6)	0.0154 (7)	0.0020 (6)
C34	0.0322 (8)	0.0155 (7)	0.0297 (8)	−0.0040 (6)	0.0135 (7)	0.0011 (6)
C33	0.0252 (7)	0.0172 (6)	0.0249 (7)	−0.0023 (5)	0.0118 (6)	0.0020 (6)
C32	0.0239 (7)	0.0145 (6)	0.0194 (7)	−0.0010 (5)	0.0089 (6)	0.0014 (5)
C37	0.0245 (7)	0.0166 (6)	0.0221 (7)	−0.0004 (5)	0.0115 (6)	0.0003 (5)
O38	0.0268 (5)	0.0162 (5)	0.0356 (6)	−0.0010 (4)	0.0185 (5)	−0.0018 (4)
C40	0.0307 (8)	0.0163 (7)	0.0380 (9)	0.0002 (6)	0.0195 (7)	−0.0032 (6)
C39	0.0397 (10)	0.0244 (8)	0.0538 (11)	−0.0024 (7)	0.0311 (9)	−0.0041 (8)
O37	0.0375 (6)	0.0160 (5)	0.0455 (7)	−0.0042 (4)	0.0274 (6)	−0.0040 (5)
C42	0.0307 (8)	0.0287 (8)	0.0266 (8)	−0.0010 (6)	0.0081 (7)	0.0090 (6)

Geometric parameters (Å, º)

O41—C41	1.2047 (18)	C34—H341	0.922
C41—C4	1.450 (2)	C33—C32	1.3842 (19)
C41—C42	1.483 (2)	C33—H331	0.938
C4—N3	1.3490 (18)	C32—C37	1.486 (2)
C4—C5	1.410 (2)	C37—O38	1.3266 (18)
N3—N2	1.2918 (17)	C37—O37	1.2006 (18)

N3—C31	1.4445 (18)	O38—C40	1.4527 (18)
N2—O1	1.3648 (17)	C40—C39	1.493 (2)
O1—C5	1.4118 (19)	C40—H402	0.969
C5—O5	1.2006 (18)	C40—H401	0.972
C31—C36	1.375 (2)	C39—H393	0.967
C31—C32	1.390 (2)	C39—H392	0.955
C36—C35	1.380 (2)	C39—H391	0.972
C36—H361	0.948	C42—H423	0.958
C35—C34	1.369 (2)	C42—H422	0.966
C35—H351	0.937	C42—H421	0.935
C34—C33	1.379 (2)		

O41—C41—C4	121.51 (13)	C34—C33—H331	120.2
O41—C41—C42	122.68 (14)	C32—C33—H331	118.7
C4—C41—C42	115.81 (13)	C31—C32—C33	117.18 (13)
C41—C4—N3	124.76 (12)	C31—C32—C37	122.01 (12)
C41—C4—C5	129.68 (13)	C33—C32—C37	120.81 (13)
N3—C4—C5	105.55 (12)	C32—C37—O38	111.49 (12)
C4—N3—N2	115.20 (12)	C32—C37—O37	124.70 (13)
C4—N3—C31	130.25 (12)	O38—C37—O37	123.80 (13)
N2—N3—C31	114.47 (12)	C37—O38—C40	115.53 (11)
N3—N2—O1	104.81 (12)	O38—C40—C39	107.47 (13)
N2—O1—C5	110.80 (11)	O38—C40—H402	107.1
O1—C5—C4	103.58 (12)	C39—C40—H402	110.2
O1—C5—O5	120.17 (14)	O38—C40—H401	108.6
C4—C5—O5	136.25 (15)	C39—C40—H401	110.6
N3—C31—C36	115.86 (13)	H402—C40—H401	112.7
N3—C31—C32	121.65 (13)	C40—C39—H393	108.5
C36—C31—C32	122.40 (13)	C40—C39—H392	109.9
C31—C36—C35	118.82 (14)	H393—C39—H392	108.5
C31—C36—H361	119.5	C40—C39—H391	110.7
C35—C36—H361	121.7	H393—C39—H391	108.8
C36—C35—C34	120.17 (14)	H392—C39—H391	110.5
C36—C35—H351	120.9	C41—C42—H423	111.3
C34—C35—H351	118.9	C41—C42—H422	108.9
C35—C34—C33	120.39 (14)	H423—C42—H422	107.4
C35—C34—H341	119.9	C41—C42—H421	110.7
C33—C34—H341	119.7	H423—C42—H421	110.1
C34—C33—C32	121.04 (14)	H422—C42—H421	108.3

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C36—H361···O5^{i}	0.95	2.51	3.253 (2)	136
C40—H401···O41^{ii}	0.97	2.46	3.116 (2)	124

C33—H331···O38	0.94	2.33	2.681 (2)	101
C42—H423···O5	0.96	2.51	3.065 (2)	117

Symmetry codes: (i) $-x+3/2$, $y-1/2$, $-z+1/2$; (ii) $-x+1/2$, $y+1/2$, $-z+1/2$.