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4-(3-Nitrophenyl)-3-(phenylsulfonyl)but-3-en-2-one

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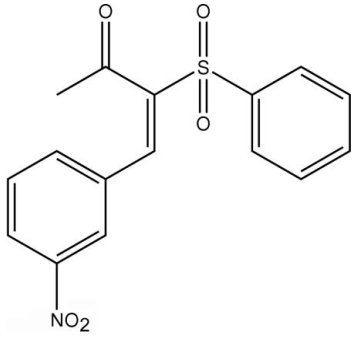
Key indicators: single-crystal X-ray study; T = 298 K; mean σ(C–C) = 0.002 Å; R factor = 0.039; wR factor = 0.107; data-to-parameter ratio = 16.7.

The C=C double bond in the title molecule, $C_{16}H_{13}NO_5S$, has an E configuration. The crystal structure is stabilized by C—H···O hydrogen bonds. There is also a weak C—H···π-ring interaction in the structure.

Related literature

For related literature, see: Pei (1998). For the chemical preparation, see: Wada *et al.* (1996).



Experimental

Crystal data

$C_{16}H_{13}NO_5S$
M_r = 331.34
Monoclinic, $P2_1/n$
a = 7.957 (2) Å
b = 10.580 (3) Å
c = 18.271 (6) Å
β = 95.976 (14)°
V = 1529.7 (8) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.24 mm^{-1}
T = 298 (1) K
0.25 × 0.20 × 0.20 mm

Data collection

Rigaku R-AXIS RAPID diffractometer
Absorption correction: multi-scan (*ABSCOR*; Higashi, 1995)
T_{min} = 0.923, T_{max} = 0.954
14836 measured reflections
3507 independent reflections
2890 reflections with $I > 2\sigma(I)$
R_{int} = 0.027

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.038
$wR(F^2)$ = 0.106
S = 1.05
3507 reflections
210 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.34 e Å^{-3}
$\Delta\rho_{min}$ = −0.40 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
C4—H4···O4^{i}	0.93	2.60	3.104 (2)	114
C8—H8···O5^{ii}	0.93	2.53	3.321 (2)	143
C10—H10···O3^{iii}	0.93	2.54	3.364 (2)	148
C13—H13···O5^{iii}	0.93	2.59	3.433 (2)	152
C12—H12···O4	0.93	2.52	2.903 (2)	105
C16—H16···O4^{i}	0.93	2.60	3.524 (2)	176
C9—H9···*Cg*1^{ii}	0.93	2.80	3.608 (2)	145

Symmetry codes: (i) $-x+\frac{3}{2}, y-\frac{1}{2}, -z+\frac{1}{2}$; (ii) $x-\frac{1}{2}, -y+\frac{1}{2}, z-\frac{1}{2}$; (iii) $x+1, y, z$. *Cg*1 is the centroid of the C11–C16 phenyl ring.

Data collection: *PROCESS-AUTO* (Rigaku, 1998); cell refinement: *PROCESS-AUTO*; data reduction: *CrystalStructure* (Rigaku/MSC, 2004); program(s) used to solve structure: *SIR97* (Altomare *et al.*, 1999); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *ORTEP-3 for Windows* (Farrugia, 1997); software used to prepare material for publication: *CrystalStructure*.

This work was supported by the Open Foundation of the Key Discipline of Industrial Catalysis.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: FB2081).

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supplementary materials

Acta Cryst. (2008). E**64**, o640 [**doi:10.1107/S1600536808005370**]

4-(3-Nitrophenyl)-3-(phenylsulfonyl)but-3-en-2-one

Y. Wang and W. Pei

Comment

Phenylsulfonyl-3-alken-2-ones have been used as asymmetric synthetic reagents (Wada *et al.*, 1996) as well as electron-deficient dienophiles. They have been applied in asymmetric Diels–Alder chemistry (Pei, 1998). The title compound has been prepared and studied in order to get a better understanding about the synthesis of phenylsulfonyl-3-alken-2-ones. The molecular structure, Fig. 1, shows that an *E*-configuration is present on the C=C double bond between the atoms C1 and C4. There is also present a C—H···π-electron ring interaction in the structure. It involves the phenyl ring C11–C16 with centroid *Cg*1 (Table 1).

Experimental

The title compound was prepared according to the procedure of Wada *et al.* (1996). Diffraction quality crystals were obtained by recrystallization from ethanol solution by slow evaporation at room temperature. The average dimension of the block crystals was about 0.2 mm.

Refinement

All the H atoms were discerned in the difference Fourier map. Nevertheless, the H atoms were positioned into the idealized positions and their parameters were constrained in riding-mode approximation. The constraints: C—$H_{aryl,alkenyl}$)=0.93 and C—H_{methyl}=0.96 Å. $U_{iso}(H_{aryl},H_{alkenyl})$= 1.2$U_{eq}(C_{aryl},C_{alkenyl})$ and $U_{iso}(H_{metyl})$=1.5$U_{eq}(C_{methyl})$.

Figures

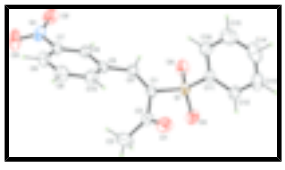


Fig. 1. View of the title molecule showing the atom labelling scheme. The displacement ellipsoids are drawn at the 40% probability level.

4-(3-Nitrophenyl)-3-(phenylsulfonyl)but-3-en-2-one

Crystal data

$C_{16}H_{13}NO_5S$	F_{000} = 688.00
M_r = 331.34	D_x = 1.439 Mg m^{-3}
Monoclinic, $P2_1/n$	Mo $K\alpha$ radiation λ = 0.71075 Å
Hall symbol: -P 2yn	Cell parameters from 12472 reflections
a = 7.957 (2) Å	θ = 3.2–27.5º

b = 10.580 (3) Å
c = 18.271 (6) Å
β = 95.976 (14)º
V = 1529.7 (8) Å^3
Z = 4

μ = 0.24 mm^{-1}
T = 298 (1) K
Block, colourless
0.25 × 0.20 × 0.20 mm

Data collection

Rigaku R-AXIS RAPID diffractometer
Monochromator: graphite
Detector resolution: 10.00 pixels mm^{-1}
T = 285 K
ω scans
Absorption correction: multi-scan (ABSCOR; Higashi, 1995)
T_{min} = 0.923, T_{max} = 0.954
14836 measured reflections

3507 independent reflections
2890 reflections with $I > 2\sigma(I)$
R_{int} = 0.027
θ_{max} = 27.5º
θ_{min} = 3.2º
h = −8→10
k = −13→13
l = −23→23

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.038
$wR(F^2)$ = 0.106
S = 1.05
3507 reflections
210 parameters
42 constraints
Primary atom site location: structure-invariant direct methods

Secondary atom site location: difference Fourier map
Hydrogen site location: difference Fourier map
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0516P)^2 + 0.4387P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 0.34 e Å^{-3}
$\Delta\rho_{min}$ = −0.40 e Å^{-3}
Extinction correction: SHELXL97 (Sheldrick, 2008)
Extinction coefficient: 0.0097 (17)

Special details

Geometry. ENTER SPECIAL DETAILS OF THE MOLECULAR GEOMETRY

Refinement. Refinement using all reflections. The weighted *R*-factor (*wR*) and goodness of fit (S) are based on F^2. *R*-factor (gt) are based on F. The threshold expression of F^2 > 2.0 sigma(F^2) is used only for calculating *R*-factor (gt).

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å^2)

	x	*y*	*z*	U_{iso}*/U_{eq}
S1	0.84122 (5)	0.43647 (4)	0.25330 (2)	0.03894 (14)
O1	0.99494 (16)	0.40871 (14)	0.10399 (8)	0.0607 (4)
O2	0.02708 (17)	0.12269 (16)	0.11304 (9)	0.0674 (4)
O3	−0.02286 (17)	0.14516 (19)	−0.00423 (9)	0.0806 (5)
O4	0.89564 (17)	0.56378 (11)	0.24210 (8)	0.0568 (4)

O5	0.72060 (15)	0.41361 (14)	0.30472 (7)	0.0566 (4)
N1	0.07214 (18)	0.14380 (15)	0.05295 (9)	0.0508 (4)
C1	0.75310 (18)	0.37984 (14)	0.16622 (8)	0.0349 (3)
C2	0.8455 (2)	0.42725 (15)	0.10363 (9)	0.0418 (4)
C3	0.7499 (3)	0.5076 (2)	0.04658 (14)	0.0770 (7)
H31	0.8105	0.5121	0.0039	0.116*
H32	0.7371	0.5910	0.0659	0.116*
H33	0.6404	0.4713	0.0332	0.116*
C4	0.61399 (18)	0.30895 (15)	0.16516 (8)	0.0370 (3)
H4	0.5739	0.2956	0.2106	0.044*
C5	0.51608 (18)	0.24949 (14)	0.10202 (8)	0.0357 (3)
C6	0.34611 (19)	0.22278 (15)	0.10721 (9)	0.0376 (3)
H6	0.2974	0.2406	0.1502	0.045*
C7	0.25178 (19)	0.16961 (15)	0.04761 (9)	0.0385 (3)
C8	0.3182 (2)	0.13919 (17)	−0.01672 (9)	0.0473 (4)
H8	0.2507	0.1052	−0.0565	0.057*
C9	0.4884 (2)	0.16088 (19)	−0.02025 (10)	0.0526 (4)
H9	0.5374	0.1386	−0.0625	0.063*
C10	0.5862 (2)	0.21504 (17)	0.03795 (10)	0.0462 (4)
H10	0.7005	0.2289	0.0346	0.055*
C11	1.02028 (18)	0.34291 (15)	0.28003 (8)	0.0369 (3)
C12	1.1754 (2)	0.40079 (18)	0.29545 (9)	0.0453 (4)
H12	1.1883	0.4867	0.2868	0.054*
C13	1.3113 (2)	0.3282 (2)	0.32406 (11)	0.0590 (5)
H13	1.4168	0.3654	0.3350	0.071*
C14	1.2909 (3)	0.2017 (2)	0.33632 (12)	0.0655 (6)
H14	1.3824	0.1538	0.3563	0.079*
C15	1.1369 (3)	0.1452 (2)	0.31942 (13)	0.0658 (6)
H15	1.1252	0.0589	0.3271	0.079*
C16	0.9995 (2)	0.21509 (17)	0.29120 (11)	0.0524 (4)
H16	0.8947	0.1770	0.2799	0.063*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
S1	0.0325 (2)	0.0432 (2)	0.0403 (2)	0.00241 (15)	−0.00025 (15)	−0.00634 (16)
O1	0.0424 (7)	0.0742 (9)	0.0676 (9)	−0.0004 (6)	0.0163 (6)	0.0104 (7)
O2	0.0468 (7)	0.0850 (11)	0.0725 (10)	−0.0159 (7)	0.0157 (7)	−0.0016 (8)
O3	0.0393 (7)	0.1266 (15)	0.0713 (10)	0.0022 (8)	−0.0154 (7)	−0.0245 (10)
O4	0.0583 (8)	0.0375 (6)	0.0717 (9)	0.0017 (5)	−0.0075 (7)	−0.0083 (6)
O5	0.0395 (6)	0.0887 (10)	0.0420 (7)	0.0056 (6)	0.0062 (5)	−0.0108 (6)
N1	0.0348 (7)	0.0560 (9)	0.0606 (10)	−0.0022 (6)	0.0003 (7)	−0.0121 (7)
C1	0.0321 (7)	0.0371 (7)	0.0346 (7)	0.0026 (6)	−0.0009 (6)	0.0016 (6)
C2	0.0411 (8)	0.0418 (8)	0.0423 (9)	−0.0043 (7)	0.0038 (7)	0.0017 (6)
C3	0.0709 (14)	0.0856 (16)	0.0746 (15)	0.0032 (12)	0.0080 (12)	0.0424 (13)
C4	0.0335 (7)	0.0425 (8)	0.0344 (7)	0.0013 (6)	0.0012 (6)	0.0035 (6)
C5	0.0325 (7)	0.0384 (7)	0.0355 (7)	0.0008 (6)	0.0004 (6)	0.0036 (6)
C6	0.0345 (7)	0.0437 (8)	0.0348 (8)	−0.0007 (6)	0.0047 (6)	0.0001 (6)

C7	0.0319 (7)	0.0412 (8)	0.0413 (8)	0.0020 (6)	−0.0006 (6)	0.0001 (6)
C8	0.0476 (9)	0.0549 (10)	0.0377 (8)	0.0027 (8)	−0.0039 (7)	−0.0071 (7)
C9	0.0516 (10)	0.0671 (12)	0.0406 (9)	0.0026 (9)	0.0128 (8)	−0.0096 (8)
C10	0.0356 (8)	0.0568 (10)	0.0472 (9)	0.0010 (7)	0.0086 (7)	−0.0031 (8)
C11	0.0317 (7)	0.0447 (8)	0.0336 (7)	0.0004 (6)	0.0004 (6)	−0.0030 (6)
C12	0.0347 (8)	0.0575 (10)	0.0435 (9)	−0.0072 (7)	0.0031 (7)	−0.0021 (7)
C13	0.0308 (8)	0.0930 (16)	0.0526 (11)	0.0023 (9)	0.0016 (7)	−0.0053 (10)
C14	0.0525 (11)	0.0838 (15)	0.0587 (12)	0.0304 (11)	−0.0011 (9)	−0.0044 (11)
C15	0.0724 (14)	0.0492 (11)	0.0744 (14)	0.0161 (10)	0.0006 (11)	−0.0012 (9)
C16	0.0471 (10)	0.0451 (9)	0.0633 (11)	−0.0017 (7)	−0.0020 (8)	−0.0047 (8)

Geometric parameters (Å, °)

S1—O5	1.4317 (13)	C6—H6	0.9300
S1—O4	1.4360 (13)	C7—C8	1.376 (2)
S1—C11	1.7617 (16)	C8—C9	1.382 (3)
S1—C1	1.7747 (16)	C8—H8	0.9300
O1—C2	1.204 (2)	C9—C10	1.375 (2)
O2—N1	1.211 (2)	C9—H9	0.9300
O3—N1	1.224 (2)	C10—H10	0.9300
N1—C7	1.468 (2)	C11—C16	1.380 (2)
C1—C4	1.335 (2)	C11—C12	1.381 (2)
C1—C2	1.508 (2)	C12—C13	1.384 (3)
C2—C3	1.491 (3)	C12—H12	0.9300
C3—H31	0.9600	C13—C14	1.369 (3)
C3—H32	0.9600	C13—H13	0.9300
C3—H33	0.9600	C14—C15	1.369 (3)
C4—C5	1.465 (2)	C14—H14	0.9300
C4—H4	0.9300	C15—C16	1.375 (3)
C5—C6	1.394 (2)	C15—H15	0.9300
C5—C10	1.396 (2)	C16—H16	0.9300
C6—C7	1.377 (2)		
O5—S1—O4	118.99 (9)	C8—C7—C6	123.02 (15)
O5—S1—C11	107.53 (8)	C8—C7—N1	118.44 (14)
O4—S1—C11	108.63 (8)	C6—C7—N1	118.54 (14)
O5—S1—C1	107.57 (8)	C7—C8—C9	117.81 (15)
O4—S1—C1	106.61 (8)	C7—C8—H8	121.1
C11—S1—C1	106.95 (7)	C9—C8—H8	121.1
O2—N1—O3	124.14 (16)	C10—C9—C8	120.74 (16)
O2—N1—C7	118.48 (15)	C10—C9—H9	119.6
O3—N1—C7	117.38 (16)	C8—C9—H9	119.6
C4—C1—C2	130.13 (14)	C9—C10—C5	120.92 (15)
C4—C1—S1	116.88 (12)	C9—C10—H10	119.5
C2—C1—S1	112.89 (11)	C5—C10—H10	119.5
O1—C2—C3	121.90 (17)	C16—C11—C12	121.53 (15)
O1—C2—C1	120.05 (15)	C16—C11—S1	119.05 (12)
C3—C2—C1	117.76 (16)	C12—C11—S1	119.18 (13)
C2—C3—H31	109.5	C11—C12—C13	118.54 (18)
C2—C3—H32	109.5	C11—C12—H12	120.7

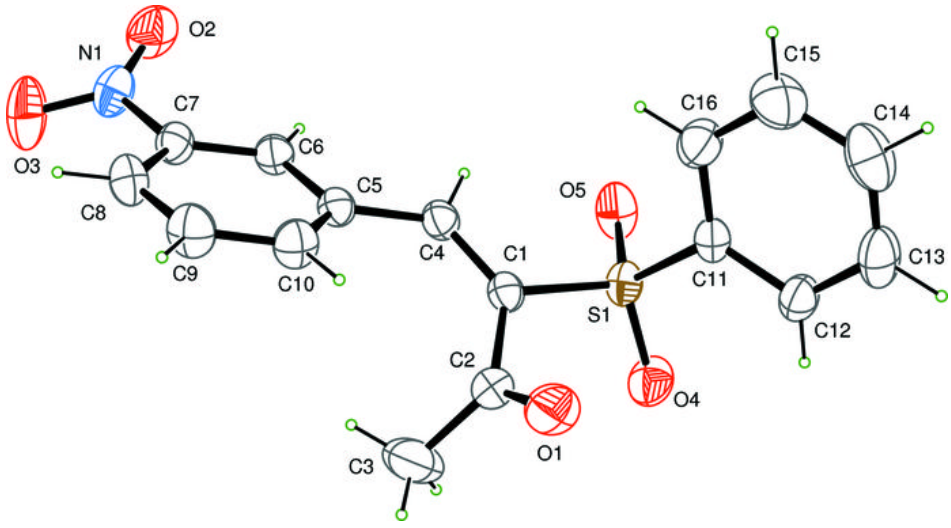
H31—C3—H32	109.5	C13—C12—H12	120.7
C2—C3—H33	109.5	C14—C13—C12	120.20 (18)
H31—C3—H33	109.5	C14—C13—H13	119.9
H32—C3—H33	109.5	C12—C13—H13	119.9
C1—C4—C5	128.65 (14)	C15—C14—C13	120.55 (18)
C1—C4—H4	115.7	C15—C14—H14	119.7
C5—C4—H4	115.7	C13—C14—H14	119.7
C6—C5—C10	118.61 (14)	C14—C15—C16	120.5 (2)
C6—C5—C4	118.26 (14)	C14—C15—H15	119.7
C10—C5—C4	123.10 (14)	C16—C15—H15	119.7
C7—C6—C5	118.80 (14)	C15—C16—C11	118.64 (18)
C7—C6—H6	120.6	C15—C16—H16	120.7
C5—C6—H6	120.6	C11—C16—H16	120.7
O4—S1—C1—C2	36.47 (12)	C4—C1—C2—C3	58.5 (2)
O4—S1—C1—C4	−140.16 (11)	C1—C4—C5—C6	−155.46 (15)
O4—S1—C11—C12	8.06 (15)	C1—C4—C5—C10	26.5 (2)
O4—S1—C11—C16	−177.55 (14)	C4—C5—C6—C7	178.54 (13)
O5—S1—C1—C2	165.12 (10)	C4—C5—C10—C9	−179.12 (15)
O5—S1—C1—C4	−11.50 (13)	C6—C5—C10—C9	2.9 (2)
O5—S1—C11—C12	−121.99 (13)	C10—C5—C6—C7	−3.4 (2)
O5—S1—C11—C16	52.59 (15)	C5—C6—C7—N1	−178.84 (13)
C1—S1—C11—C12	122.77 (13)	C5—C6—C7—C8	1.3 (2)
C1—S1—C11—C16	−62.81 (15)	N1—C7—C8—C9	−178.34 (14)
C11—S1—C1—C2	−79.59 (11)	C6—C7—C8—C9	1.6 (2)
C11—S1—C1—C4	103.79 (12)	C7—C8—C9—C10	−2.2 (2)
O2—N1—C7—C6	−28.2 (2)	C8—C9—C10—C5	−0.0 (2)
O2—N1—C7—C8	151.86 (15)	S1—C11—C12—C13	173.00 (13)
O3—N1—C7—C6	151.83 (16)	S1—C11—C16—C15	−173.37 (15)
O3—N1—C7—C8	−28.2 (2)	C12—C11—C16—C15	1.0 (2)
S1—C1—C2—O1	56.37 (18)	C16—C11—C12—C13	−1.3 (2)
S1—C1—C2—C3	−117.54 (15)	C11—C12—C13—C14	0.2 (2)
S1—C1—C4—C5	−179.53 (12)	C12—C13—C14—C15	1.0 (3)
C2—C1—C4—C5	4.5 (2)	C13—C14—C15—C16	−1.3 (3)
C4—C1—C2—O1	−127.56 (18)	C14—C15—C16—C11	0.3 (3)

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C4—H4···O4^{i}	0.93	2.60	3.104 (2)	114
C8—H8···O5^{ii}	0.93	2.53	3.321 (2)	143
C10—H10···O3^{iii}	0.93	2.54	3.364 (2)	148
C13—H13···O5^{iii}	0.93	2.59	3.433 (2)	152
C12—H12···O4	0.93	2.52	2.903 (2)	105
C16—H16···O4^{i}	0.93	2.60	3.524 (2)	176
C9—H9···Cg1^{ii}	0.93	2.80	3.608 (2)	145

Symmetry codes: (i) −*x*+3/2, *y*−1/2, −*z*+1/2; (ii) *x*−1/2, −*y*+1/2, *z*−1/2; (iii) *x*+1, *y*, *z*.

Fig. 1


O2
N1
C7
O3
C6
C8
C5
C9
C10
C4
C1
O5
S1
C11
C15
C16
C14
C13
C12
C2
O4
O1
C3