

4-[4-(Dimethylamino)phenyl]-1-(phenylsulfonyl)-
but-3-en-2-oneWen Pei,^{a*} Yong-Jiang Wang,^b
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Key indicators

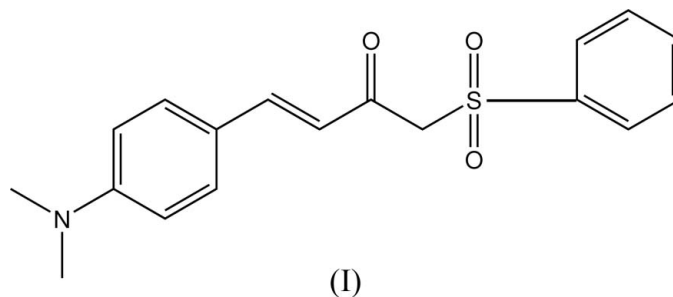
Single-crystal X-ray study
T = 296 K
Mean $\sigma(\text{C}-\text{C}) = 0.003 \text{ \AA}$
Disorder in main residue
R factor = 0.051
wR factor = 0.104
Data-to-parameter ratio = 16.5For details of how these key indicators were automatically derived from the article, see <http://journals.iucr.org/e>.An *E* configuration is found about the C=C double bond in the title molecule, C₁₈H₁₉NO₃S. Evidence is found for significant conjugation in the C₆H₄-NMe₂ residue.

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Comment

1-Phenylsulfonyl-3-alken-2-ones attract the attention of organic chemists owing to their utility as asymmetric synthetic reagents (Wada *et al.*, 1996), and as electron-deficient dienophiles, finding applications in asymmetric Diels–Alder chemistry (Pei, 1998). The title compound, (I), has been prepared and studied in order to obtain a better understanding of its stereochemistry. The molecular structure (Fig. 1 and Table 1) shows that an *E* configuration is found about the C3=C4 double bond. The torsion angles listed in Table 1 indicate that the dimethylamine group and its attached benzene ring are essentially coplanar. The N1–C8 bond distance of 1.359 (3) Å is considerably shorter than the normal single N–C bond of 1.43 Å (Allen *et al.*, 1987), indicating conjugation in this region of the molecule.

Experimental

The title compound was prepared according to the procedure of Wada *et al.* (1996). Diffraction quality crystals were obtained by slow evaporation of an ethanol solution of (I) at room temperature.

Crystal data

C₁₈H₁₉NO₃S
M_r = 329.41
Monoclinic, *P*2₁/*n*
a = 7.224 (2) Å
b = 19.712 (6) Å
c = 12.177 (5) Å
 β = 105.272 (14)°
V = 1672.7 (10) Å³
Z = 4*D_x* = 1.308 Mg m⁻³
Mo *K*α radiation
Cell parameters from 11803 reflections
 θ = 3.1–27.5°
 μ = 0.21 mm⁻¹
T = 296 (1) K
Block, yellow
0.35 × 0.18 × 0.16 mm

Data collection

Rigaku R-AXIS RAPID
diffractometer
 ω scans
Absorption correction: multi-scan
(*ABSCOR*; Higashi, 1995)
 $T_{\min} = 0.960$, $T_{\max} = 0.967$
15968 measured reflections

3834 independent reflections
2183 reflections with $F^2 > 2\sigma(F^2)$
 $R_{\text{int}} = 0.034$
 $\theta_{\text{max}} = 27.5^\circ$
 $h = -9 \rightarrow 9$
 $k = -25 \rightarrow 25$
 $l = -15 \rightarrow 15$

Refinement

Refinement on F^2
 $R[F^2 > 2\sigma(F^2)] = 0.051$
 $wR(F^2) = 0.104$
 $S = 1.01$
3834 reflections
233 parameters
H atoms treated by a mixture of
independent and constrained
refinement

$w = 1/[0.0001F_o^2 + 3\sigma(F_o^2) + 0.5]/(4F_o^2)$
 $(\Delta/\sigma)_{\text{max}} = 0.002$
 $\Delta\rho_{\text{max}} = 0.39 \text{ e } \text{\AA}^{-3}$
 $\Delta\rho_{\text{min}} = -0.48 \text{ e } \text{\AA}^{-3}$
Extinction correction:
(Larson, 1970)
Extinction coefficient: $2.8(3) \times 10^2$

Table 1

Selected geometric parameters (\AA , $^\circ$).

| | | | |
|--------------|-------------|--------------|------------|
| S1—O1 | 1.4301 (13) | N1—C8 | 1.359 (3) |
| S1—O2 | 1.4339 (16) | N1—C11 | 1.435 (4) |
| S1—C1 | 1.768 (2) | N1—C12 | 1.453 (3) |
| S1—C13 | 1.757 (2) | C31—C41 | 1.393 (7) |
| O3—C2 | 1.186 (4) | C32—C42 | 1.393 (7) |
| C8—N1—C11 | 121.3 (2) | C2—C31—C41 | 114.5 (4) |
| C8—N1—C12 | 121.5 (2) | C5—C41—C31 | 118.1 (4) |
| C11—N1—C12 | 117.1 (2) | | |
| C11—N1—C8—C7 | 1.2 (3) | C12—N1—C8—C7 | -175.2 (2) |
| C11—N1—C8—C9 | -177.8 (2) | C12—N1—C8—C9 | 5.7 (3) |

Atoms C3 and C4 were found to be disordered over two positions, *viz.* C31 and C41, and C32 and C42, and were refined anisotropically with 50% site occupancy factors and equal C—C bond distances of 1.393 (7) \AA . The H atoms associated with this group were located in a difference Fourier map and were refined freely [C—H = 0.82 (2)–1.02 (2) \AA]. The remaining H atoms were included in the riding-

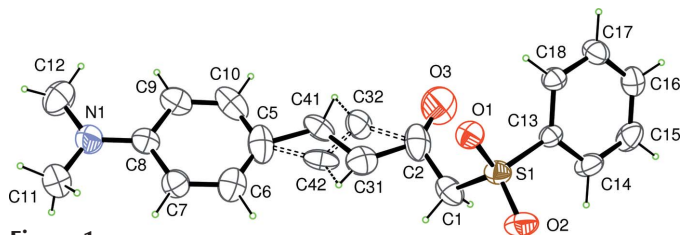


Figure 1

View of (I), showing the atom-labelling scheme. Displacement ellipsoids are drawn at the 40% probability level. Both disorder components are shown.

model approximation, with C—H(aromatic) = 0.98 \AA , C—H(methylene) = 0.97 \AA and C—H(methyl) = 0.96 \AA , and with $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{C})$.

Data collection: *PROCESS-AUTO* (Rigaku Corporation, 1998); cell refinement: *PROCESS-AUTO*; data reduction: *CrystalStructure* (Rigaku/MS, 2004); program(s) used to solve structure: *SIR97* (Altomare *et al.*, 1999); program(s) used to refine structure: *CRYSTALS* (Betteridge *et al.*, 2003); molecular graphics: *ORTEP-3 for Windows* (Farrugia, 1997); software used to prepare material for publication: *CrystalStructure*.

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