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3,5,5-Trimethyl-2-[2-(phenylsulfonyl)ethyl]-2-cyclohexen-1-one

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Key indicators

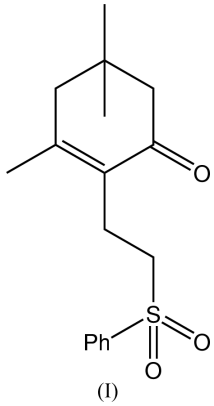
Single-crystal X-ray study
T = 173 K
Mean σ(C–C) = 0.004 Å
R factor = 0.040
wR factor = 0.129
Data-to-parameter ratio = 19.5

For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

The structure of title compound, $C_{17}H_{22}O_3S$, reveals conjugation between the double bond within the cyclohexene ring and the exocyclic carbonyl, and, as a result, the three substituents at C1—C3 occupy equatorial positions.

Comment

The structure determination of (I) (Fig. 1) shows the double bond to exist between the C2 and C3 atoms [1.347 (3) Å], and conjugation with the C1 carbonyl group [O1—C1 1.223 (3) Å and C1—C2 1.474 (3) Å]; the O1—C1—C2—C3 torsion angle is 174.6 (2)°. As a consequence, the substituents at C1, C2 and C3 project equatorially from the six-membered ring. The side chain has an extended conformation up to the sulfonyl group as evidenced by the torsion angle of 171.05 (16)° for C2—C21—C22—S22. The phenyl group folds back [C21—C22—S22—C23 is −77.54 (18)°] so as to lie on the same side of the molecule as the C3-methyl substituent.



(I)

The crystal structure is stabilized by C—H···O interactions. Thus, C22—H22a is separated by 2.35 Å from the O22a^{i} atom with C22···O22a^{i} being 3.312 (3) Å and the angle subtended at H22a being 163° [symmetry code: (i) $x, \frac{1}{2} - y, \frac{1}{2} + z$]. Similarly, C4—H4$a$ is 2.59 Å from O22b^{ii} so that C4···O22b^{ii} is 3.543 (3) Å and the angle at H4a is 161° [symmetry code: (ii) x, y, $1 + z$]. While not forming an intermolecular interaction, the O1 atom forms an intramolecular contact with H22b so that O1···H22b is 2.55 Å.

Experimental

The title compound was obtained as a by-product from the bicycloannulation reaction of isophorone and phenyl vinyl sulfone as described in the literature (Cory & Renneborg, 1984). Recrystallization was from dichloromethane/hexane

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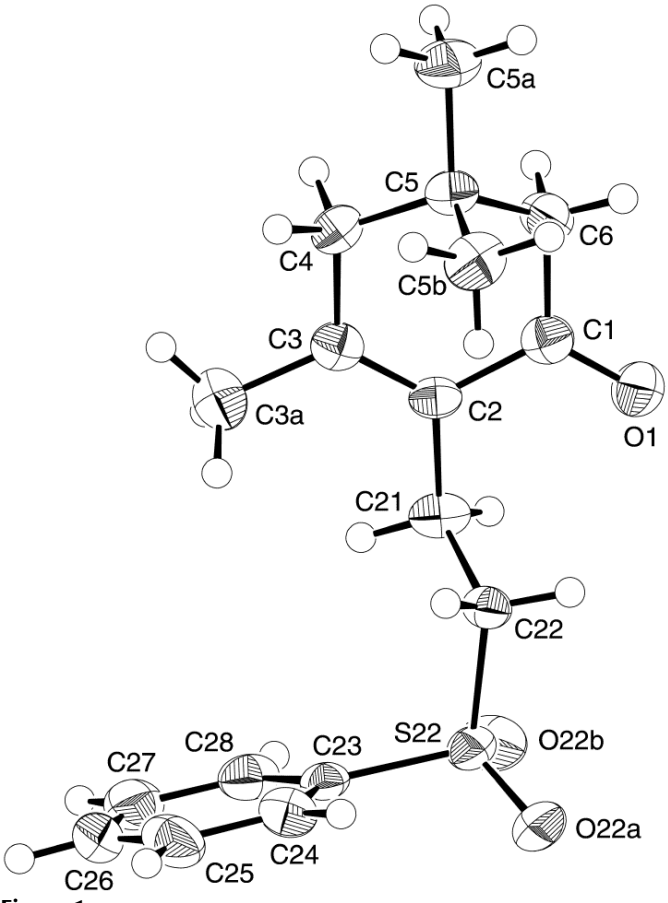

Figure 1
The molecular structure and crystallographic numbering scheme for (I). Displacement ellipsoids are shown at the 50% probability level (Johnson, 1976).

Crystal data

$C_{17}H_{22}O_3S$
M_r = 306.42
Orthorhombic, *Pbca*
a = 16.560 (5) Å
b = 20.69 (1) Å
c = 9.453 (2) Å
V = 3240 (2) Å^3
Z = 8
D_x = 1.256 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 22 reflections
θ = 7.3–9.5°
μ = 0.21 mm^{-1}
T = 173 K
Plate, colourless
0.37 × 0.37 × 0.11 mm

Data collection

Rigaku AFC-7*R* diffractometer
ω–2θ scans
3725 measured reflections
3725 independent reflections
1960 reflections with $I > 2\sigma(I)$
θ_{max} = 27.5°
$h = 0 \rightarrow 21$
$k = 0 \rightarrow 26$
$l = 0 \rightarrow 12$
3 standard reflections every 400 reflections intensity decay: −1.4%

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.040
$wR(F^2)$ = 0.129
S = 1.01
3725 reflections
191 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0508P)^2 + 0.9004P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.26 e Å^{-3}
$\Delta\rho_{min}$ = −0.29 e Å^{-3}

The C-bound H atoms were placed in geometrically calculated positions and included in the final refinement in the riding-model approximation with an overall displacement parameter, U_{iso}, for all phenyl-type H atoms, $1.25U_{iso}$ for methylene H atoms and $1.5U_{iso}$ for methyl H atoms.

Data collection: *MSC/AFC Diffractometer Control Software* (Molecular Structure Corporation, 1996); cell refinement: *MSC/AFC Diffractometer Control Software*; data reduction: *TEXSAN for Windows* (Molecular Structure Corporation, 1997); program(s) used to solve structure: *SIR*97 (Altomare *et al.*, 1994); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); software used to prepare material for publication: *SHELXL*97.

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