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Key indicators

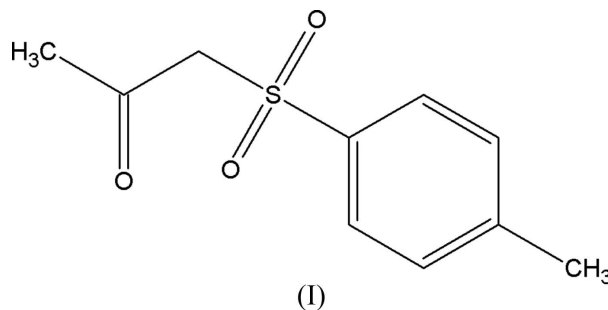
Single-crystal X-ray study
 $T = 294$ K
Mean $\sigma(\text{C}-\text{C}) = 0.004$ Å
 R factor = 0.039
 wR factor = 0.104
Data-to-parameter ratio = 17.1For details of how these key indicators were automatically derived from the article, see <http://journals.iucr.org/e>.1-(*p*-Tolylsulfonyl)propan-2-one

The asymmetric unit of the title compound, $\text{C}_{10}\text{H}_{12}\text{O}_3\text{S}$, contains two independent molecules having different conformations. In one conformation, the aromatic ring and the acetyl substituent are *gauche* to each other, while in the other they are *anti*.

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Comment

The title compound (I), prepared from chloroacetone and sodium *p*-toluenesulfinate, undergoes condensations with aldehydes in the presence of base (Mélot *et al.*, 1988; Roche *et al.*, 1998; Swenson *et al.*, 2002). Condensations with other electrophiles have not been reported, although simple base-mediated alkylations are known (Sato *et al.*, 1981; Shono *et al.*, 1988). We have used (I) in base-induced condensations with cyclic methylthioiminium salts to give vinylogous sulfonamides (Michael *et al.*, 2004). This type of intermediate is a precursor for bicyclic products, which were prepared as models in ongoing studies aimed at the total synthesis of indolizidine alkaloids (Michael *et al.*, 1999, 2005).



The title compound crystallizes with two independent molecules in the asymmetric unit. These two molecules, labelled *A* and *A'*, have different conformations (Fig. 1). In molecule *A*, the aromatic ring and the acetyl group are *gauche* to each other, with a torsion angle of $-54.9(2)^\circ$ for the $\text{C}2-\text{C}3-\text{S}1-\text{C}4$ segment. In addition, dipolar interactions between the carbonyl group and the $\text{S}=\text{O}$ bonds of the sulfone appear to be minimized, since the $\text{C}=\text{O}$ bond effectively points in the opposite direction to the resultant dipole of the two $\text{S}=\text{O}$ bonds. In molecule *A'*, by contrast, the aromatic ring and the acetyl group are *anti* to each other, the corresponding torsion angle being $-173.97(18)^\circ$. In this molecule, the carbonyl group lies parallel to the $\text{S}1'=\text{O}3'$ bond, and their dipoles point in the same direction. The carbon–oxygen ($\text{C}2'=\text{O}1'$) bond length of $1.201(3)$ Å and the adjacent $\text{C}2'-\text{C}3'$ bond length of $1.526(3)$ Å indicate that the former is a typical carbonyl double bond while the latter is clearly a single

bond, ruling out the hypothesis that the second conformation is a result of enolization of the ketone and subsequent formation of an intramolecular hydrogen bond of the type $O-H \cdots O_2S$.

Experimental

A mixture of sodium *p*-toluenesulfinate (5.00 g, 28.0 mmol) and chloroacetone (2.59 g, 28.0 mmol) in dimethyl sulfoxide (12.5 ml) was heated on a water bath at 363 K for 4 h. The light-brown reaction mixture was diluted with water (50 ml) and extracted with dichloromethane (3×30 ml). The organic fractions were combined, dried ($MgSO_4$) and concentrated *in vacuo*. Purification by column chromatography on silica gel with ethyl acetate–hexane (1: 1) as eluting solvent gave (I) as a yellow solid [yield 5.83 g, 98%; m.p. 323–324 K; literature m.p. 323–324.5 K (Crandall & Pradat, 1985)]. 1H NMR (200 MHz, $CDCl_3$): δ 7.78 (2H, *d*, $J = 8.2$ Hz, 5-H and 9-H), 7.38 (2H, *d*, $J = 8.2$ Hz, 6-H and 8-H), 4.17 (2H, *s*, CH_2), 2.44 (3H, *s*, $ArCH_3$) 2.37 (3H, *s*, $COCH_3$); ^{13}C NMR (50 MHz, $CDCl_3$): δ 196.01 (C=O), 145.29 (C7), 137.57 (C4), 129.82 and 128.00 (C5, C6, C8, C9), 67.54 (CH_2), 31.26 ($COCH_3$), 21.47 ($ArCH_3$). Crystals suitable for X-ray crystallography were obtained as colourless plates by slow evaporation of a diethyl ether solution.

Crystal data

$C_{10}H_{12}O_3S$	$Z = 4$
$M_r = 212.26$	$D_x = 1.315$ Mg m^{-3}
Monoclinic, $P2_1$	Mo $K\alpha$ radiation
$a = 14.5381$ (16) Å	$\mu = 0.28$ mm $^{-1}$
$b = 5.5394$ (6) Å	$T = 294$ (2) K
$c = 14.6879$ (16) Å	Cut plate, colourless
$\beta = 114.998$ (2)°	$0.4 \times 0.4 \times 0.12$ mm
$V = 1072.0$ (2) Å 3	

Data collection

Bruker SMART CCD area-detector diffractometer	7547 measured reflections
φ and ω scans	4419 independent reflections
Absorption correction: multi-scan (SADABS; Sheldrick, 1996)	3580 reflections with $I > 2\sigma(I)$
$T_{min} = 0.891$, $T_{max} = 0.967$	$R_{int} = 0.019$
	$\theta_{max} = 28.3^\circ$

Refinement

Refinement on F^2	$w = 1/[\sigma^2(F_o^2) + (0.0638P)^2]$
$R[F^2 > 2\sigma(F^2)] = 0.039$	where $P = (F_o^2 + 2F_c^2)/3$
$wR(F^2) = 0.104$	$(\Delta/\sigma)_{max} = 0.024$
$S = 1.03$	$\Delta\rho_{max} = 0.21$ e Å $^{-3}$
4419 reflections	$\Delta\rho_{min} = -0.23$ e Å $^{-3}$
258 parameters	Absolute structure: Flack (1983),
H-atom parameters constrained	1467 Friedel pairs
	Flack parameter: -0.03 (7)

H atoms were positioned geometrically and allowed to ride on their respective parent atoms, with C–H bond lengths of 0.93 (aromatic CH), 0.96 (CH_3) or 0.97 Å (CH_2), with $U_{iso}(H)$ 1.2 (CH and CH_2) or 1.5 (CH_3) times $U_{eq}(C)$.

Data collection: SMART-NT (Bruker, 1998); cell refinement: SAINT-Plus (Bruker, 1999); data reduction: XPREP (Bruker, 1999); program(s) used to solve structure: SHELXS97 (Sheldrick, 1997);

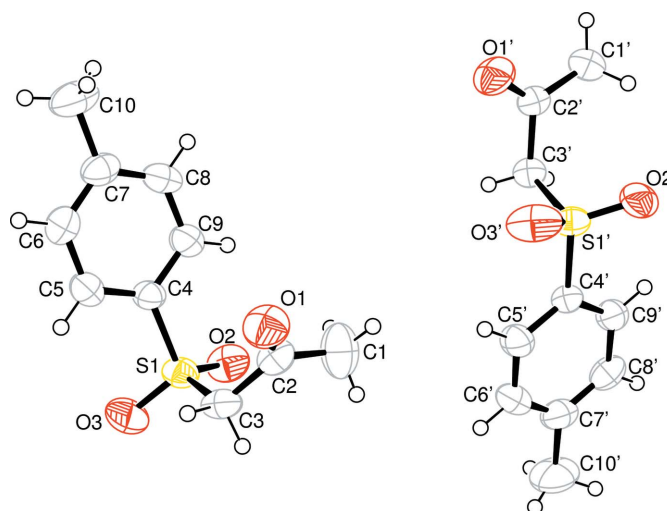


Figure 1

The molecular structures of the two independent molecules *A* (left) and *A'* (right) in the asymmetric unit of (I), showing the atomic numbering scheme. Displacement ellipsoids are drawn at the 50% probability level.

program(s) used to refine structure: SHELXL97 (Sheldrick, 1997); molecular graphics: ORTEP-3 for Windows (Farrugia, 1997); software used to prepare material for publication: WinGX (Farrugia, 1999) and PLATON (Spek, 2003).

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