1-(4-Bromophenyl)-2-(2-propenylsulfonyl)-ethanone

The title compound, $\text{C}_{11}\text{H}_{11}\text{BrO}_{3}\text{S}$, was prepared by reaction of sodium allylsulfinate with $p$-bromophenacyl bromide to confirm the identity of the former.

Comment

Thermal desulfination of allylic sulfinic acids is a synthetically useful procedure for the regio- and stereospecific synthesis of alkenes. In general, allylic sulfinic acids are unstable, with spontaneous retro-ene desulfination occurring readily at room temperatures with allylic transposition of the double bond (Braverman, 1990).

In our studies on the preparation of allyl sulfinic acid (4) by acid cleavage of the corresponding tributyltin allylic sulfinate, (3), the unstable acid was isolated as the sodium salt, (2), by immediate quenching with sodium bicarbonate (Hiscock et al., 1995). The products obtained in this reaction did not, however, give satisfactory combustion analysis and, to confirm its identity, it was derivatized with $p$-bromophenacyl bromide. Spectroscopic analysis ($^1\text{H}$ NMR and IR) suggested that this compound was a sulfone, (1), rather than the isomeric sulfinate ester, (5). The X-ray crystal structure determination of (1), reported here, confirmed this structural assignment. Compound (1) crystallizes as discrete molecular species with the molecule comprising the asymmetric unit (Fig. 1). The bond lengths and angles are in accord with conventional values (Allen et al., 1987). The $\text{CH}_{2}\text{COC}_{2}\text{H}_{5}\text{Br}$ fragment is essentially planar, with the C4—S1 bond lying nearly
perpendicular to this plane, with $S1—C4—C5—O3 = -91.4^\circ$.

**Experimental**

The title compound was prepared according to published procedures (Hiscock et al., 1995). Crystals suitable for X-ray diffraction studies were obtained as pale-yellow needles by recrystallization from ethanol (m.p. 401–403 K).

**Crystal data**

$C_{11}H_{11}BrO_{3}S$

$M_r = 303.17$

Triclinic, $\overline{P}$

$a = 5.2888 (13)$ Å

$b = 8.5968 (11)$ Å

$c = 13.878 (2)$ Å

$\alpha = 74.393 (10)^\circ$

$\beta = 89.598 (18)^\circ$

$\gamma = 88.870 (16)^\circ$

$V = 607.37 (19)$ Å$^3$

$Mr = 8.5968 (11)$ Å$^2$

$c = 13.878 (2)$ Å

$\alpha = 74.393 (10)^\circ$

$\beta = 89.598 (18)^\circ$

$\gamma = 88.870 (16)^\circ$

$V = 607.37 (19)$ Å$^3$

Data collection

Rigaku AFC-7R diffractometer

$\omega$–$2\theta$ scans

Absorption correction: $\psi$ scan

(North et al., 1968)

$T_{\text{min}} = 0.471$, $T_{\text{max}} = 0.871$

2298 measured reflections

2142 independent reflections

1185 reflections with $I > 2\sigma(I)$

Refinement

Refinement on $F^2$

$R[F^2 > 2\sigma(F)^2] = 0.034$

$wR(F)^2 = 0.088$

$S = 1.02$

2142 reflections

145 parameters

Table 1

Selected geometric parameters (Å, °).

<table>
<thead>
<tr>
<th>Bond</th>
<th>Distance (Å)</th>
<th>Angle (°)</th>
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<tbody>
<tr>
<td>Br1—C9</td>
<td>1.895 (4)</td>
<td>C1—C2—C3: 1.236 (11)</td>
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<tr>
<td>S1—O1</td>
<td>1.430 (4)</td>
<td>C2—C3: 1.492 (9)</td>
</tr>
<tr>
<td>S1—O2</td>
<td>1.435 (3)</td>
<td>C4—C5: 1.522 (6)</td>
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<tr>
<td>S1—C3</td>
<td>1.778 (5)</td>
<td>C5—C6: 1.485 (6)</td>
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<tr>
<td>S1—C1</td>
<td>1.774 (4)</td>
<td>C6—C7: 1.386 (6)</td>
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<tr>
<td>O3—C5</td>
<td>1.211 (5)</td>
<td></td>
</tr>
<tr>
<td>O1—S1—O2</td>
<td>117.4 (2)</td>
<td>C1—C2—C3: 112.8 (4)</td>
</tr>
<tr>
<td>O1—S1—C3</td>
<td>108.5 (2)</td>
<td>C1—C2—C3: 112.8 (4)</td>
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<tr>
<td>O1—S1—C4</td>
<td>108.7 (2)</td>
<td>C1—C2—C3: 112.8 (4)</td>
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<td>O2—S1—C3</td>
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<tr>
<td>O2—S1—C4</td>
<td>108.5 (2)</td>
<td>C1—C2—C3: 112.8 (4)</td>
</tr>
<tr>
<td>C3—S1—C4</td>
<td>104.0 (2)</td>
<td>C1—C2—C3: 112.8 (4)</td>
</tr>
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</table>

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References


