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2-Bromo-2-propyl phenyl sulfone

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Crystalline 2-bromo-2-propyl phenyl sulfone (α-bromoisopropyl phenyl sulfone), $C_9H_{11}BrO_2S$, exhibits no rotational disorder of its α-bromoisopropyl group, in contrast to the large disorder of the same group exhibited by crystalline 2-bromo-2-propyl mesityl sulfone.

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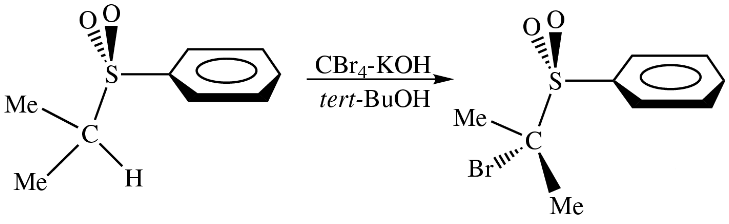
Key indicators

Single-crystal X-ray study
T = 296 K
Mean σ(C–C) = 0.012 Å
R factor = 0.034
wR factor = 0.094
Data-to-parameter ratio = 8.3

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

Comment

The unexpected large rotational disorder of the 2-bromo-2-propyl group that characterized crystalline 2-bromo-2-propyl mesityl sulfone (Chan-Yu-King *et al.*, 2001) prompted us to examine its less hindered analog, 2-bromo-2-propyl phenyl sulfone, (I).



(I)

The X-ray structure of (I), shown in Fig. 1 with its atom numbering, shows it to be a single rotamer, completely devoid of rotational disorder. The Br atom of this rotamer resides on the side of the sulfonyl group adjacent to O1. There is little steric interaction in (I), the most prominent being C6···C9 and Br···C2 which are, respectively, 0.07 Å greater than and 0.01 Å less than the sum of their van der Waals radii. The shortest intermolecular distances are O1···C9, O2···C8, and Br···C3 which are, respectively, −0.09 Å less than, 0.19 Å greater than, and 0.01 Å greater than the sum of their van der Waals radii. The shortest intra- and intermolecular contact distances are given in Table 1. The intermolecular packing is shown by Fig. 2.

Experimental

2-Bromo-2-propyl phenyl sulfone, (I), was prepared from phenyl 2-propyl sulfone (Matthews, 1972) by treatment with CBr_4–powdered KOH–*tert*-BuOH (Parady, 1977; see also, Meyers *et al.*, 1977; Meyers, 1978), recrystallized from hexanes and used in this study (m.p. 367–368 K). Similar reactions with $CBrCl_3$, CCl_2Br_2 or CBr_2F_2 instead of CBr_4 produced varying yields of (I) (Parady, 1977).

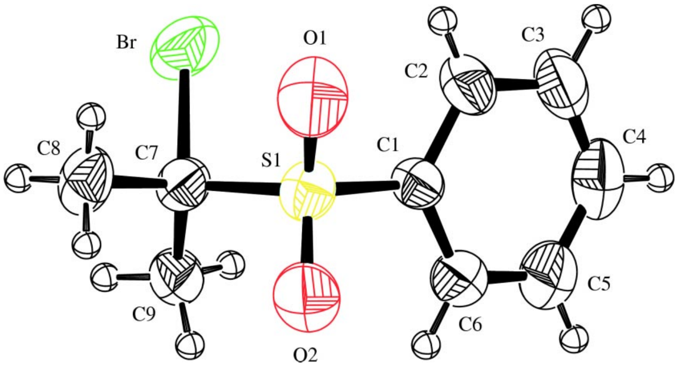


Figure 1
The molecular structure and atom numbering scheme for (I) with displacement ellipsoids at the 50% probability level.

Crystal data

$C_9H_{11}BrO_2S$
$M_r = 263.15$
Orthorhombic, $Pna2_1$
$a = 21.134\ (2)$ Å
$b = 6.2537\ (11)$ Å
$c = 7.979\ (2)$ Å
$V = 1054.6\ (4)$ Å^3
$Z = 4$
$D_x = 1.657$ Mg m^{-3}

Mo *Kα* radiation
Cell parameters from 25 reflections
$\theta = 12.5–14.6°$
$\mu = 4.06$ mm^{-1}
$T = 296$ K
Prism, colorless
0.36 × 0.20 × 0.15 mm

Data collection

Rigaku AFC-5*S* diffractometer
ω scans
Absorption correction: *ψ* scan (North *et al.*, 1968)
$T_{\min} = 0.398$, $T_{\max} = 0.544$
1917 measured reflections
1008 independent reflections
668 reflections with $I > 2\sigma(I)$

$R_{\rm int} = 0.043$
$\theta_{\max} = 25.0°$
$h = 0 \rightarrow 25$
$k = -7 \rightarrow 7$
$l = 0 \rightarrow 9$
3 standard reflections every 100 reflections intensity decay: 3.2%

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.034$
$wR(F^2) = 0.094$
$S = 1.01$
1008 reflections
121 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0492P)^2 + 0.1257P]$ where $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{\max} < 0.001$
$\Delta\rho_{\max} = 0.31$ e Å^{-3}
$\Delta\rho_{\min} = -0.27$ e Å^{-3}
Extinction correction: *SHELXL*97 (Sheldrick, 1997)
Extinction coefficient: 0.0094 (15)
Absolute structure: Flack (1983), 0 Friedel opposites, $wR(F^2)$ (inverse) = 0.097
Flack parameter = 0.09 (2)

Table 1
Contact geometry (Å).

C6···C9	3.469 (12)	O2···C8$^{\rm ii}$	3.414 (10)
Br···C2	3.539 (9)	Br···C3$^{\rm iii}$	3.555 (10)
O1···C9$^{\rm i}$	3.133 (10)		

Symmetry codes: (i) $x, y-1, z$; (ii) $\frac{1}{2}-x, \frac{1}{2}+y, z-\frac{1}{2}$; (iii) $1-x, -y, \frac{1}{2}+z$.

The non-centrosymmetric polar space group of (I) was unexpected and caused difficulty with respect to the data/parameter ratio which resulted in higher than normal standard uncertainties. Also, since Friedel pairs were not collected, we were unable to use the results of the Flack parameter to determine whether or not the orientation of the crystal on the diffractometer and of the model was identical. However, a comparison of the $wR(F^2)$ values for the model and its inverse gave values of 0.094 and 0.097, respectively, indicating that the selected model is most likely correct. The rotational orientation of the methyl groups were refined by the circular Fourier method available in *SHELXL*97 (Sheldrick, 1997). All H atoms are riding.

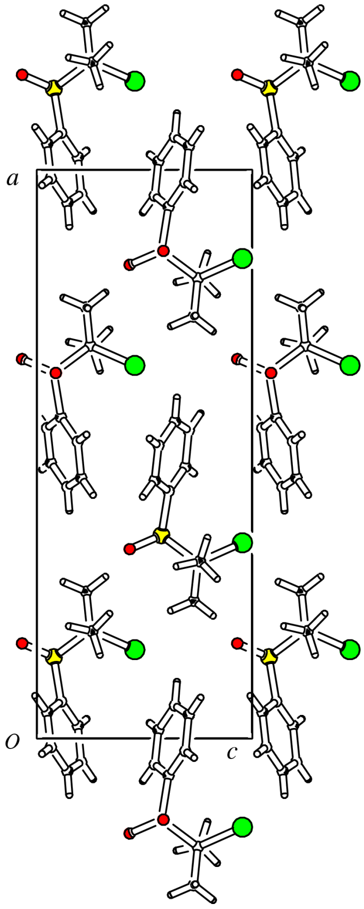


Figure 2
The molecular packing in (I) as viewed down the *b* axis. Color code: green = Br, red = O, yellow = S.

Data collection: *MSC/AFC Diffractometer Control Software* (Molecular Structure Corporation,1996); cell refinement: *MSC/AFC Diffractometer Control Software*; data reduction: *PROCESS* in *TEXSAN* (Molecular Structure Corporation, 1997); program(s) used to solve structure: *SIR*92 (Burla *et al.*, 1989); program(s) used to refine structure: *LS* in *TEXSAN* and *SHELXL*97 (Sheldrick, 1997); molecular graphics: *ORTEP* (Johnson, 1965) in *TEXSAN*; software used to prepare material for publication: *TEXSAN, SHELXL*97, and *PLATON* (Spek, 2000).

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