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(7-*endo*-Bromocamphorylsulfonyl)imine

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[3aS-(3aα,6α,7β)]-7-Bromo-4,5,6,7-tetrahydro-8,8-dimethyl-3*H*-3a,6-methano-2,1-benzisothiazole 2,2-dioxide, $C_{10}H_{14}BrNO_2S$, has the (−)-camphorsulfonimine structure, with the Br atom β-oriented. The C—Br distance is 1.941 (5) Å, the S—N distance is 1.687 (3) Å, and the C═N distance is 1.271 (6) Å.

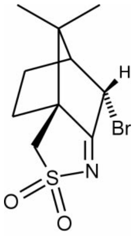
Key indicators

Single-crystal X-ray study
T = 296 K
Mean σ(C–C) = 0.007 Å
R factor = 0.057
wR factor = 0.162
Data-to-parameter ratio = 18.0

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

Comment

The title compound, (I), was described approximately 100 years ago by Armstrong & Lowry (1902). They correctly determined that its crystals are orthorhombic and obtained axial ratios within less than 0.1% of our values well before the discovery of X-ray diffraction. Their paper contains a beautiful drawing of a crystal and an extensive table of interfacial angles.



(I)

The structure of (I) is found to be identical to that of 3-ethylthiocamphorylsulfonimine (Barkley *et al.*, 1995), except for the *endo*-Br substituent instead of SCH_2CH_3 at the same position. These two compounds have the same space group and very similar cell dimensions. The crystal structure of (+)-camphorylsulfonylimine, which is unsubstituted and has the opposite absolute configuration, has also been reported (Chakraborty *et al.*, 1997). The title compound is one of three for which *ab initio* crystal structure predictions were recently carried out in blind tests (Motherwell, 2001).

Experimental

The synthesis of (I) has been described by Armstrong & Lowry (1902) and more recently by Davis *et al.* (1992). Crystals of (I) were grown by slow cooling of a toluene/2,2,5-trimethylpentane solution.

Crystal data

$C_{10}H_{14}BrNO_2S$
$M_r = 292.19$
Orthorhombic, $P2_12_12_1$
$a = 7.2643$ (4) Å
$b = 10.6393$ (6) Å
$c = 15.6331$ (13) Å
$V = 1208.24$ (14) Å^3
$Z = 4$
$D_x = 1.606$ Mg m^{-3}

Cu $K\alpha$ radiation
Cell parameters from 25 reflections
$\theta = 11.3$–$34.0°$
$\mu = 6.11$ mm^{-1}
$T = 296$ K
Fragment, colorless
0.23 × 0.22 × 0.15 mm

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Data collection

Enraf–Nonius CAD-4 diffractometer
$\theta/2\theta$ scans
Absorption correction: ψ scan (North *et al.*, 1968)
$T_{\min} = 0.267$, $T_{\max} = 0.399$
5380 measured reflections
2484 independent reflections
2348 reflections with $I > 2\sigma(I)$

$R_{\rm int} = 0.057$
$\theta_{\max} = 74.9°$
$h = 0 \rightarrow 9$
$k = -13 \rightarrow 13$
$l = -19 \rightarrow 19$
3 standard reflections
frequency: 120 min
intensity decay: 9.0%

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.057$
$wR(F^2) = 0.162$
$S = 1.15$
2484 reflections
138 parameters
H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.0977P)^2 + 0.6776P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\max} < 0.001$
$\Delta\rho_{\max} = 0.89$ e Å^{-3}
$\Delta\rho_{\min} = -0.81$ e Å^{-3}
Absolute structure: Flack (1983), 1033 Friedel pairs
Flack parameter = 0.02 (4)

Table 1
Selected geometric parameters (Å, °).

Br—C1	1.941 (5)	S—N	1.687 (3)
S—O2	1.408 (5)	S—C7	1.820 (6)
S—O1	1.418 (5)	N—C2	1.271 (6)
O2—S—O1	117.7 (4)	O1—S—C7	111.1 (2)
O2—S—N	107.7 (2)	N—S—C7	97.7 (2)
O1—S—N	107.8 (2)	C2—N—S	107.6 (3)
O2—S—C7	112.7 (4)	C3—C8—C6	93.0 (4)
C7—S—N—C2	−9.8 (3)	N—C2—C3—C7	14.9 (5)
S—N—C2—C3	−1.6 (5)	C2—C3—C7—S	−18.6 (4)
Br—C1—C2—N	54.8 (5)	N—S—C7—C3	17.4 (3)

H atoms were placed in calculated positions with C—H bond distances in the range 0.96—0.98 Å and thereafter treated as riding. A torsional parameter was refined for methyl groups. $U_{\rm iso} = 1.2U_{\rm eq}$ of the attached atom (1.5 for methyl groups).

Data collection: *CAD*-4 *EXPRESS* (Enraf–Nonius, 1994); cell refinement: *CAD*-4 *EXPRESS*; data reduction: *maXus* (Mackay *et al.*, 1999); program(s) used to solve structure: *SIR*97 (Altomare *et al.*, 1999); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); molecular graphics: *ORTEP*-3 *for Windows* (Farrugia, 1997); software used to prepare material for publication: *SHELXL*97.

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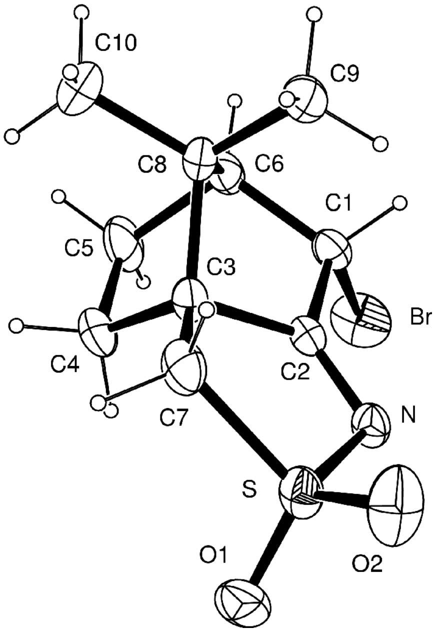

Figure 1
View of the title compound showing the numbering scheme and ellipsoids at the 30% probability level.

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