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Structure of 3-Bromo-λ^6-thietane 1,1-Dioxide, $C_3H_5BrO_2S$

BY JOSEPH F. CHIANG*

Laboratory for the Structure of Matter, Naval Research Laboratory, Washington, DC 20375, USA

Abstract. $M_r = 184 \cdot 9$, orthorhombic, *Pnma*, $a = 16 \cdot 664$ (10), $b = 6 \cdot 617$ (3), $c = 5 \cdot 103$ (4) Å, $Z = 4$, $V = 562 \cdot 78$ Å^3, $D_x = 2 \cdot 182$ Mg m^{-3}, λ(Mo $K\alpha$) = $0 \cdot 71069$ Å, $F(000) = 360$. Final $R = 8 \cdot 0\%$ for 404 observed reflections. The structure is isomorphous to 3-chlorothietane 1,1-dioxide reported earlier.

Introduction. The purpose of this study is to determine the geometry and the position of the Br atom with respect to the thietane ring. It is also our purpose to study the ring puckering. The sample was synthesized from λ^6-thietane 1,1-dioxide by bromination and recrystallized from chloroform–hexane.

Experimental. Sample supplied by Professor D. C. Dittmer of Syracuse University, nearly rectangular crystal, $0 \cdot 42 \times 0 \cdot 40 \times 0 \cdot 80$ mm; Nicolet *P3F* diffractometer, graphite monochromator, Mo $K\alpha$ radiation; 12 reflections used for measuring lattice parameters, no absorption correction applied, $2\theta_{max} = 45 \cdot 0°$, range of *hkl*: (0,0,−5) to (16,7,5), three reflections used as standards measured after intervals of 60 measurements, their intensities remained constant (within ±6%) during data collection; 406 reflections measured, 404 unique; structure solved by direct-phase determination using symbolic addition procedure and refined on all reflections by anisotropic full-matrix least squares (*ORFLS*, Busing, Martin & Levy, 1962) on heavy atoms with parameters for H atoms kept constant, positions of H atoms derived from a difference map; $R = 8 \cdot 0\%$ for the 404 reflections after six cycles of refinement (initial value of $R = 12\%$), $wR = 10 \cdot 27\%$, $S = 4 \cdot 56$, function minimized $\sum w(|F_o| - |F_c|)^2$ with weights, w, calculated according to Gilardi (1973); ratio of maximum least-squares shift-to-error = $0 \cdot 68$; atomic scattering factors from *International Tables for X-ray Crystallography* (1974).

* Present address: Department of Chemistry, State University of New York, College at Oneonta, Oneonta, New York 13820, USA.

Discussion. The structural parameters are shown in Fig. 1. (Note: the numbering system used here is different from the IUPAC nomenclature of the compound.) Coordinates and B_{eq} values for the non-H atoms are listed in Table 1.†

† Lists of structure factors, anisotropic thermal parameters and unrefined H-atom parameters have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 38296 (5 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.
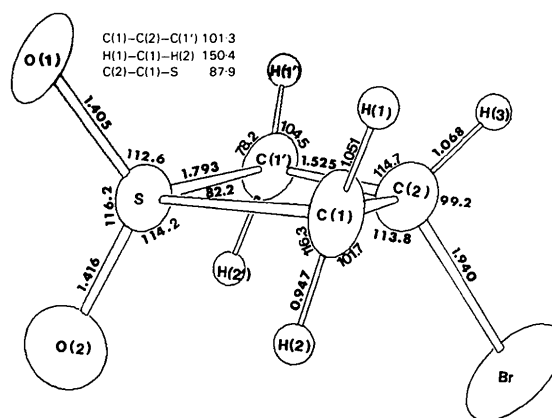

Fig. 1. The structure of 3-bromo-λ^6-thietane 1,1-dioxide. Bond distances in Å, angles in°. The e.s.d.'s for bond distances are in the range 0·01–0·02 Å; for bond angles, 0·6–1·4°.

Table 1. *Fractional coordinates and B_{eq} values for the non-H atoms of 3-bromo-λ^6-thietane 1,1-dioxide*

The e.s.d.'s for B_{eq} are near $0 \cdot 1$ Å^2. $B_{eq} = \frac{4}{3}\sum_i \sum_j \beta_{ij} \mathbf{a}_i \cdot \mathbf{a}_j$.

	x	y	z	B_{eq}(Å^2)
Br	0·2335 (1)	0·2500	0·2093 (6)	2·2
C(1)	0·3914 (6)	0·0718 (18)	0·2862 (24)	1·2
C(2)	0·3392 (10)	0·2500	0·3684 (36)	1·6
S	0·4587 (3)	0·2500	0·1386 (9)	0·9
O(1)	0·4540 (8)	0·2500	−0·1363 (26)	2·4
O(2)	0·5369 (6)	0·2500	0·2473 (28)	1·2

Table 2. *Comparison of the structure of 3-bromo-λ^6-thietane 1,1-dioxide with related molecules*

For title-compound e.s.d.'s, see Fig. 1.

	3-Bromo-λ^6-thietane 1,1-dioxide	3-Chloro-λ^6-thietane 1,1-dioxide	3-Hydroxy-λ^6-thietane 1,1-dioxide
C(1)–C(2)	1·525 Å	1·536 Å	1·532 Å
C(1)–S	1·793	1·791	1·788(1·782)
S–O(1)	1·405	1·426	1·435
S–O(2)	1·416	1·419	1·436
C–X	1·940	1·776	1·415
(X = OH, Cl, Br)			
O(1)–S–O(2)	116·2°	117·9°	117·5°
C(1)–S–C(1')	82·2	81·5	82·4
S–C(1)–C(2)	87·9	89·3	88·8

The molecule has m symmetry in the crystal. The position of the Br atom with respect to the thietane ring was determined. The dihedral angle between planes C(1)SC(1') and C(1)C(2)C(1') is 171·0° while that between planes SC(1)C(2) and SC(1')C(2) is 171·4°. In general, the structural parameters agree with the previously reported structures of 3-chloro-λ^6-thietane

1,1-dioxide (Andreetti, Bocelli & Sgarabotto, 1973a) and 3-hydroxy-λ^6-thietane 1,1-dioxide (Andreetti, Bocelli & Sgarabotto, 1973b). A comparison of their structures is shown in Table 2.

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3-Hydroxy-1,2,3-benzotriazin-4(3H)-one, C7H5N3O2

BY W. EDWARD HUNT* AND CARL H. SCHWALBE

Department of Pharmacy, The University of Aston in Birmingham, Gosta Green, Birmingham B4 7ET, England
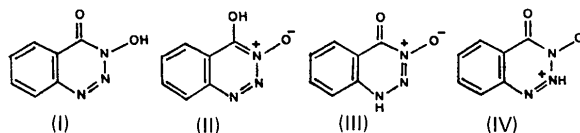
AND KEITH VAUGHAN

Department of Chemistry, Saint Mary's University, Halifax, Nova Scotia, Canada B3H 2J5

Abstract. $M_r = 163·14$, $P2_1/n$, $a = 15·04$ (1), $b = 4·29$ (2), $c = 10·95$ (1) Å, $\beta = 103·2$ (1)°, $U = 687·7$ Å^3, $Z = 4$, $D_x = 1·575$ Mg m^{-3}, λ(Mo $K\alpha$) = 0·71069 Å, $\mu = 0·130$ mm^{-1}, $F(000) = 336$, $T = 293$ K. Final $R = 0·109$ for 890 observed reflections. The ring N(1)–N(2) and carbonyl bond distances of 1·251 (7) and 1·221 (7) Å respectively show that these bonds are double, thus confirming the 3-hydroxy-4-one structure deduced chemically. A 2·589 (7) Å contact between O(3) and O(4) of an adjacent molecule related by the 2_1 axis is best explained by hydrogen bonding.

Introduction. The title compound has been assigned structure (I) in preference to the other feasible tauto-

mers (II) and (III) on the basis of chemical reactivity (Harrison & Smith, 1960) and spectral characteristics (Ahern, Navratil & Vaughan, 1977). The IR spectrum of a Nujol suspension showed a broad carbonyl band at relatively low frequency (1660 cm^{-1}) suggestive of intramolecular hydrogen bonding. Thermolysis leads to dimer formation minus the elements of N$_2$O, apparently *via* stepwise loss of O and N$_2$ (Ahern *et al.*, 1977). The crystal-structure determination reported here was undertaken in order to confirm the structure and to relate it to these spectroscopic and chemical properties.

(I)   (II)   (III)   (IV)

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* Present address: Department of Materials, Queen Mary College, Mile End Road, London E1 4NS, England.

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