

Table 2. Selected bond lengths (Å) and bond angles (°)

O(1)—S(1)	1.615 (4)	O(2)—S(1)	1.616 (4)
O(3)—S(1)	1.434 (4)	C(1)—O(1)	1.483 (4)
C(7)—O(2)	1.473 (4)	C(2)—C(1)	1.511 (5)
C(6)—C(1)	1.514 (6)	C(7)—C(1)	1.549 (5)
C(3)—C(2)	1.527 (6)	C(4)—C(3)	1.507 (6)
C(5)—C(4)	1.514 (7)	C(6)—C(5)	1.524 (6)
C(8)—C(7)	1.522 (5)	C(12)—C(7)	1.525 (6)
C(9)—C(8)	1.525 (6)	C(10)—C(9)	1.505 (7)
C(11)—C(10)	1.506 (7)	C(12)—C(11)	1.526 (6)
O(2)—S(1)—O(1)	94.2 (2)	O(3)—S(1)—O(1)	109.7 (2)
O(3)—S(1)—O(2)	106.1 (2)	C(1)—O(1)—S(1)	112.5 (2)
C(7)—O(2)—S(1)	113.4 (2)	C(2)—C(1)—O(1)	105.6 (3)
C(6)—C(1)—O(1)	109.0 (3)	C(6)—C(1)—C(2)	110.1 (3)
C(7)—C(1)—O(1)	101.2 (3)	C(7)—C(1)—C(2)	116.0 (3)
C(7)—C(1)—C(6)	114.0 (3)	C(3)—C(2)—C(1)	112.6 (3)
C(4)—C(3)—C(2)	112.2 (4)	C(5)—C(4)—C(3)	111.0 (4)
C(6)—C(5)—C(4)	111.1 (4)	C(5)—C(6)—C(1)	112.3 (3)
C(1)—C(7)—O(2)	102.5 (3)	C(8)—C(7)—O(2)	105.9 (3)
C(8)—C(7)—C(1)	115.1 (3)	C(12)—C(7)—O(2)	108.1 (3)
C(12)—C(7)—C(1)	114.5 (3)	C(12)—C(7)—C(8)	109.9 (3)
C(9)—C(8)—C(7)	112.6 (3)	C(10)—C(9)—C(8)	111.9 (4)
C(11)—C(10)—C(9)	110.7 (4)	C(12)—C(11)—C(10)	111.2 (4)
C(11)—C(12)—C(7)	112.0 (4)		

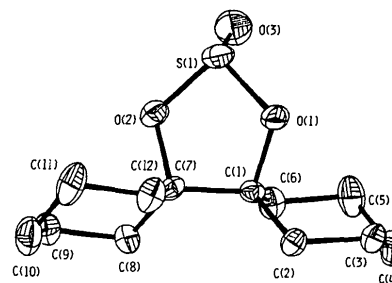


Fig. 1. Numbering of atoms and conformation of the molecule.

those found for 4-phenyl-1,3,2-dioxathiolane 2-oxide (25°) and *meso*-4,5-diphenyl-1,3,2-dioxathiolane 2-oxide (35°).

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Structure of *cis*-4,*trans*-6-Di-*tert*-butyl-5,5-dimethyl-1,3,2-dioxathiane 2-Oxide

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Abstract. C₁₃H₂₆O₃S, *M_r* = 262.407, monoclinic, *P*₂₁/*c*, *a* = 8.559 (3), *b* = 12.562 (3), *c* = 14.116 (6) Å, β = 102.18 (3)°, *V* = 1483.56 (4) Å³, *Z* = 4, *D_x* = 1.17, *D_m* = 1.2 g cm⁻³, λ(Mo Kα) = 0.71069 Å, μ = 2.05 cm⁻¹, *F*(000) = 576, *T* = 298 K, *R* = 0.0506 for 2601 unique reflections. The six-membered sulfite ring has an unusual twist conformation with the

exocyclic S=O group in an isoclinal position and the *tert*-butyl groups in pseudo-equatorial positions. Although most of the bond lengths and bond angles are as predicted, the S=O bond length of 1.392 (5) Å is considerably shorter than expected. The structure determined confirms the interpretations of IR and NMR spectra.

Table 1. Fractional atomic coordinates ($\times 10^4$) and equivalent isotropic thermal parameters ($\text{\AA}^2 \times 10^3$)
$$U_{\text{eq}} = (1/3) \sum_i \sum_j U_{ij} a_i^* a_j^* a_i \cdot a_j$$

	x	y	z	U_{eq}
S(1)	8537.1 (15)	1124.7 (11)	5229.7 (8)	65.0 (5)
O(1)	8543 (3)	487 (2)	6218 (2)	49 (1)
O(2)	7293 (3)	2029 (2)	5388 (2)	52 (1)
O(3)	7817 (5)	419 (3)	4508 (3)	95 (2)
C(1)	8540 (5)	1184 (3)	7046 (3)	45 (1)
C(2)	6795 (4)	1593 (3)	6977 (3)	45 (1)
C(3)	6033 (5)	1684 (3)	5881 (3)	46 (1)
C(4)	9554 (5)	587 (4)	7930 (3)	57 (2)
C(5)	4581 (5)	2410 (3)	5452 (3)	53 (2)
C(21)	5785 (7)	792 (5)	7414 (4)	64 (2)
C(22)	6878 (7)	2653 (4)	7500 (4)	68 (2)
C(41)	9025 (8)	-562 (5)	8011 (5)	75 (2)
C(42)	11284 (6)	572 (6)	7763 (4)	71 (2)
C(43)	9596 (7)	1186 (6)	8886 (4)	79 (2)
C(51)	3994 (8)	2054 (6)	4394 (4)	76 (2)
C(52)	5012 (9)	3587 (4)	5460 (6)	79 (3)
C(53)	3191 (6)	2247 (5)	5956 (5)	68 (2)

Table 2. Selected bond lengths (\AA) and bond angles ($^\circ$)

O(1)—S(1)	1.608 (5)	O(2)—S(1)	1.604 (5)
O(3)—S(1)	1.392 (5)	C(1)—O(1)	1.461 (5)
C(3)—O(2)	1.467 (5)	C(2)—C(1)	1.563 (7)
C(4)—C(1)	1.554 (8)	C(3)—C(2)	1.552 (7)
C(21)—C(2)	1.538 (8)	C(22)—C(2)	1.517 (8)
C(5)—C(3)	1.557 (8)	C(41)—C(4)	1.524 (9)
C(42)—C(4)	1.548 (8)	C(43)—C(4)	1.538 (9)
C(51)—C(5)	1.538 (9)	C(52)—C(5)	1.524 (9)
C(53)—C(5)	1.524 (8)		
O(2)—S(1)—O(1)	96.5 (2)	O(3)—S(1)—O(1)	103.8 (3)
O(3)—S(1)—O(2)	110.4 (3)	C(1)—O(1)—S(1)	113.3 (3)
C(3)—O(2)—S(1)	115.7 (3)	C(2)—C(1)—O(1)	108.1 (4)
C(4)—C(1)—O(1)	105.0 (4)	C(4)—C(1)—C(2)	125.0 (4)
C(3)—C(2)—C(1)	106.3 (4)	C(21)—C(2)—C(1)	111.8 (5)
C(21)—C(2)—C(3)	107.1 (5)	C(22)—C(2)—C(1)	108.2 (5)
C(22)—C(2)—C(3)	112.8 (5)	C(22)—C(2)—C(21)	110.6 (5)
C(2)—C(3)—O(2)	107.4 (4)	C(5)—C(3)—O(2)	104.4 (4)
C(5)—C(3)—C(2)	124.3 (4)	C(41)—C(4)—C(1)	113.1 (5)
C(42)—C(4)—C(1)	105.9 (5)	C(42)—C(4)—C(41)	108.0 (6)
C(43)—C(4)—C(1)	112.0 (5)	C(43)—C(4)—C(41)	110.5 (6)
C(43)—C(4)—C(42)	107.1 (5)	C(51)—C(5)—C(3)	106.1 (5)
C(52)—C(5)—C(3)	113.2 (5)	C(52)—C(5)—C(51)	108.6 (6)
C(53)—C(5)—C(3)	112.2 (5)	C(53)—C(5)—C(51)	106.7 (5)
C(53)—C(5)—C(52)	109.8 (5)		

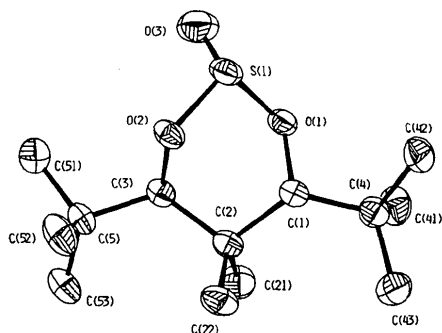


Fig. 1. Structure and conformation of the title compound with numbering of the atoms.

Table 3. Selected geometrical parameters (\AA , $^\circ$) for the chair forms, CA and CE, and TB (1) and (2)

	CA/CE range	(1)	(2)
S=O	1.434–1.456	1.392	1.439
S—O	1.599–1.630	1.604/1.606	1.605/1.616
C—O	1.453–1.493	1.464/1.467	1.467/1.479
C—C	1.512–1.548	1.552/1.563	1.524/1.551
O—S—O	97.5–108.2	96.5	97.5
S—O—C	111.4–122.7	113.3/115.7	115.6/116.5
O—C—C	107.8–113.3	105.0/108.1	106.2/106.8
C—C—C	107.1–116.9	106.3	111.2
O—S—O	103.4–109.1	103.8/110.4	102.2/109.8

Introduction. In the last 25 years the conformations of substituted 1,3,2-dioxathiane 2-oxides have been the subject of vigorous research (Hellier & Webb, 1977; Hellier & Liddy, 1988). It is now recognized that six-membered-ring sulfites can be divided into three distinct classes, characterized by the orientation of the S=O bond and the shape of the ring. The most common classes are chair forms with the S=O bond pointing in either an axial (CA) or equatorial direction (CE). The third type, the twist-boat conformation (TB), is found for only a small number of sulfites. Consequently, because of our interest in the stereochemistry of cyclic sulfites, we have prepared and isolated a sulfite (1) with a twist-boat conformation according to a previously described procedure (Liddy, 1988).

Experimental. Single crystals were grown by recrystallization from ether at 263 K, m.p. 311 K. Density measured by flotation. The X-ray measurements were made using an Enraf-Nonius CAD-4 diffractometer, operating in the $\omega/2\theta$ scan mode with graphite-monochromated Mo $K\alpha$ radiation ($\lambda = 0.71069 \text{ \AA}$). The lattice parameters were determined by least-squares refinement using setting angles for 25 reflections ($10 < \theta < 15^\circ$). Intensities were collected for $1.5 < \theta < 25^\circ$, the index range was $h - 10/10$, $k 0/14$, $l 0/16$, with scan rate $1.3\text{--}5.9^\circ \text{ min}^{-1}$, ω -scan width $0.85^\circ + 0.35^\circ \tan \theta$ and aperture setting 4 mm. Three intensity control reflections were monitored every hour. They showed $\sim 10\%$ decay which was corrected for during the data processing. 2950 reflections were measured, 2601 of which were unique, $R_{\text{int}} = 0.0138$. 1436 intensities [$F_o > 4\sigma(F_o)$] were used in refinement. The structure was solved by direct methods using SHELXS86 (Sheldrick, 1986).

All non-H atoms were located in the best E map. A subsequent difference map revealed the H-atom positions. An empirical absorption correction (DIFABS, Walker & Stuart, 1983) was applied with transmission factors in the range 0.995–0.953. Least-squares anisotropic refinement on F of non-H atoms and isotropic refinement of H atoms gave final $R = 0.0501$, $wR = 0.0567$ and $S = 1.75$, with $w = 1/[\sigma^2(F_o)]$.

+ 0.000835 F_o^2], giving acceptable agreement analyses; maximum and minimum $\Delta\rho = +0.64$ and $-0.21 \text{ e } \text{\AA}^{-3}$. Maximum shift/e.s.d. = 0.023 (*SHELX76*, Sheldrick 1976). The computations were made on a VAX 11/750 computer. Atomic scattering factors were taken from *International Tables for X-ray Crystallography* (1974).

Discussion. The refined atomic coordinates and isotropic temperature factors are presented in Table 1.* Bond distances and bond angles are given in Table 2. A *SNOOPI* (Davies, 1983) drawing of the molecular structure and the numbering of atoms are shown in Fig. 1. A number of structure determinations have been made on the two chair forms (CA and CE) of 1,3,2-dioxathiane 2-oxides and the results have been summarized by Geise, Lenstra, Petit, van Alsenoy & van Nuffel (1984). Interestingly, only one twist-boat conformation of a sulfite containing *tert*-butyl groups has been studied by X-ray analysis, namely, *cis*-4,*trans*-6-di-*tert*-butyl-5-chloro-1,3,2-dioxathiane 2-oxide (2) (Carbonnelle, Jeannin & Robert, 1978). The present X-ray structure determination of the title compound (1) was undertaken to enable comparison of geometrical differences arising from the substitution at C(5) changing from a chloro to dimethyl

* Lists of structure factors, anisotropic thermal parameters, H-atom parameters, and bond lengths and angles involving H atoms have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 53009 (13 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

groups. A selection of geometrical parameters is shown in Table 3.

Bond lengths and bond angles are quite similar for compounds (1) and (2), and most parameters lie within the range of values found for the CA/CE forms. The C—C bond length is apparently longer in twist-boat forms. One notable difference is the shortening of the S=O bond length in (1) compared with (2) and the CA/CE forms. The reason for this anomaly is not clear and with only two compounds to compare, further structural studies of related twist forms are required before any definite conclusions can be drawn.

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Acta Cryst. (1990). **C46**, 1915–1916

Ammonium Bismuth Chromate Dichromate Monohydrate

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Abstract. $\text{NH}_4\text{Bi}(\text{CrO}_4)(\text{Cr}_2\text{O}_7)\cdot\text{H}_2\text{O}$, $M_r = 577.01$, triclinic, $P\bar{1}$, $a = 6.916$ (2), $b = 7.249$ (3), $c = 10.798$ (3) \AA , $\alpha = 80.28$ (3), $\beta = 90.52$ (2), $\gamma =$

86.49 (3) $^\circ$, $V = 532.5$ (6) \AA^3 , $Z = 2$, $D_m = 3.58$ (1), $D_x = 3.598$ (4) Mg m^{-3} , $\text{Mo K}\alpha$, $\lambda = 0.71073$ \AA , $\mu = 19.938$ mm^{-1} , $F(000) = 528$, room temperature, $R =$