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The Crystal Structure of Trimethyloxosulfonium Perchlorate [(CH₃)₃SO]⁺ClO₄^{-*}

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Crystals of $[(CH_3)_3SO]^+ClO_4^-$ display tetragonal symmetry with unit cell dimensions $a = 11.66 \pm 0.01$ Å and $c = 5.99 \pm 0.01$ Å. The space group is $P\overline{42}_1m$ with Z = 4. A satisfactory trial structure was found by use of a three-dimensional Patterson summation and the refinement was carried out by means of three-dimensional difference syntheses and least-squares routine on the IBM 7090.

The $[(CH_3)_3SO]^+$ ion is required crystallographically to have the symmetry *m* but it approximates the symmetry 3*m*. The perchlorate ions are crystallographically of two types, one with required symmetry $\overline{4}$ and the other with required symmetry *mm*2. However, the perchlorate ions of the second type achieve the required symmetry in a statistical (disordered) manner.

Bond distances and angles in the trimethyloxosulfonium ion are:

 $S-C(1) = 1 \cdot 78 \pm 0 \cdot 01 \text{ Å}, \ S-C(2) = 1 \cdot 76 \pm 0 \cdot 01 \text{ Å}, \ S-O = 1 \cdot 45 \pm 0 \cdot 01 \text{ Å}, \ C(1)-S-C(1) = 105 \cdot 8 \pm 0 \cdot 7^{\circ}, \\ C(1)-S-C(2) = 105 \cdot 7 \pm 0 \cdot 5^{\circ}, \ C(1)-S-O = 112 \cdot 1 \pm 0 \cdot 5^{\circ}, \ C(2)-S-O = 114 \cdot 8 \pm 0 \cdot 6^{\circ}.$

In the ordered perchlorate ions: $Cl-O = 1.45 \pm 0.01$ Å, $O-Cl-O = 111.5 \pm 0.9^{\circ}$ and $108.8 \pm 0.9^{\circ}$.

Introduction

The syntheses and properties of members of two series of salts formed by the reaction of dimethyl sulfoxide with esters RX have been reported by Smith & Winstein (1958) and by Smith (1959). One series was reported to involve the ion $[R(CH_3)_2SO]^+$ in which all three alkyl groups are bonded directly to sulfur, and the other series the ion $[(RO)(CH_3)_2S]^+$ in which the R group is separated from sulfur by an oxygen atom. Structure assignments for the two series of salts were made on the basis of proton magnetic resonance studies as well as on chemical evidence. As a check on this structure assignment and in order to determine the bond distances and angles in the interesting $[(CH_3)_3SO]^+$ ion, the present study of the perchlorate was undertaken.

Experimental

Trimethyloxosulfonium perchlorate was prepared by Smith by the reaction of silver perchlorate on the iodide salt in aqueous solution. The substance was purified by recrystallization from water and elementary analyses for carbon, hydrogen, sulfur and chlorine gave results in close agreement with the formula $C_3H_6SClO_5$.

Crystals suitable for the X-ray study were grown by spontaneous evaporation of aqueous solutions. Precession and Weissenberg photographs about the

^{*} Contribution No. 1546 from the Department of Chemistry, University of California at Los Angeles.

a and c axes of the tetragonal unit were prepared by use of both Mo $K\alpha$ and Cu $K\alpha$ radiation. The lattice parameters were found to have the values a = $11\cdot66\pm0\cdot01$ and $c=5\cdot99\pm0\cdot01$ Å, based on Cu $K\alpha =$ $1\cdot5418$ Å. The flotation density of the crystals was found to be $1\cdot56$ g.cm⁻³ and that calculated for Z=4 is $1\cdot571$ g.cm⁻³. The photographs indicated a diffraction symmetry of 4/mmm and the only systematic extinctions in over 400 independent observed reflections are those for h00 with \hbar odd. The space groups consistent with these observations are $P42_1m$ and $P42_12$. Since a satisfactory trial structure based on $P42_1m$ was readily found, this space group was taken as the more probable one.

Complete sets of multiple-film Weissenberg intensity photographs about the *a* and *c* axes were prepared. The *a* axis photographs were prepared with $\operatorname{Cu} K\alpha$ radiation on a crystal with a cross section 0.16 mm square. The maximum value of μR for these photographs is 0.5. A crystal with a cross section 0.15 mmby 0.24 mm was used with Mo $K\alpha$ radiation for the c axis data. The corresponding maximum value of μR is about 0.08. Since the maximum difference in absorption correction was about 12%, no corrections were applied. The intensities were estimated visually by use of a calibrated intensity strip prepared with the same crystal and radiation used in the exposure of that film. The observed intensities were corrected and correlated in the usual manner to give a set of $k|F_o|$ values. Of some 600 possible reflections in the copper sphere, 414 were observed. Within the range covered by these observed reflections, some 85 possible reflections were below the minimum observable intensity.

Structure determination and refinement

The shape of the unit cell, the arrangement of symmetry elements in $P\bar{4}2_1m$ and the value Z=4 suggested a trial structure similar to that of CsCl with a unit cell about 6 Å on an edge. A three-dimensional Patterson summation not only confirmed this suggestion but furnished approximate positional parameters for sulfur, chlorine and some of the lighter atoms. This trial structure places the $[(CH_3)_3SO]^+$ ions on the mirror planes and requires that the ClO_4^- ions be of two types. The ions of the first type are in the twofold positions having symmetry $\overline{4}$ with Cl at $0, 0, 0; \frac{1}{2}, \frac{1}{2}, 0$. The ions of the second type are in the twofold positions having symmetry mm^2 with Cl at $0, \frac{1}{2}, z; \frac{1}{2}, 0, \overline{z}$ and with z approximately 0.9. With the positional parameters from the threedimensional Patterson summation as a basis, two- and three-dimensional Fourier and least-squares refinement procedures were employed. During this refinement, all atoms except the oxygen atoms of the perchlorate group of the second type behaved normally. The indicated atoms behaved anomalously with respect to both positional and vibrational parameters and the value of R leveled off at 20%. This behavior of the oxygen atoms suggested the possibility that this perchlorate ion is disordered. Accordingly a threedimensional difference Fourier, phased on sulfur and chlorine only, was computed. This summation contained maxima at reasonable positions for all carbon and oxygen atoms except those about Cl(2). The point group symmetry of mm^2 at this position would lead one to look for oxygen atoms in only two possible sets of positions; however, there were no maxima at either of these. Instead, maxima of one-half the expected height were distributed in a way which implied that this ClO_4^- ion utilizes only the point group symmetry m. The crystal apparently contains equal numbers of ClO_4^- of the second type in each of two positions related by the otherwise incompletely utilized mirror.

The final three-dimensional least-squares refinement and the structure factor calculations were performed on the IBM 7090 by means of ACA Computer Program No. 317 (UCLALS1) written by P. K. Gantzel, R. A. Sparks and K. N. Trueblood. This program minimizes the weighted sum of the squares of the quantity $(KF_o - G|F_c|)$ by a full-matrix routine, where K and G are scale factors. The program provides for several weighting options and for either isotropic or anisotropic temperature factors on the individual atoms. The options selected were the weighting scheme of Hughes (1941) and individual anisotropic temperature factors of the form

$$\exp\left[-(B_{11}h^2+B_{22}k^2+B_{33}l^2+B_{12}hk+B_{13}hl+B_{23}kl)\right].$$

Unobserved reflections were omitted from the refinement procedures and in computing the agreement index, $R = \Sigma ||F_o| - |F_c||/\Sigma |F_o|$, but were included in the final structure factor calculations. The standard deviations of the positional and vibrational parameters were estimated from the inverse matrix of the normal equations.

Table 1. Atomic positional parameters and their standard deviations

All values have been multiplied by 10^4

Atom	\boldsymbol{x}	$\sigma(x)$	y	$\sigma(y)$	z	$\sigma(z)$
s	2792	03	2208	03	6133	07
O(1)	2143	11	2857	11	4566	21
C(1)	2576	09	0735	08	5821	18
C(2)	2495	12	2505	12	8897	24
Cl(1)	0000	00	0000	00	0000	00
O(2)	0997	08	0116	10	1313	23
Cl(2)	0000	00	5000	00	9141	09
O(3)	0178	13	6198	16	9261	35
O(4)	0471	32	4529	32	7023	49
O(5)	0543	39	4457	39	0965	61

In the final three-dimensional least-squares refinement the disordered oxygen atoms were put in both positions with statistical weights of one-half. The refinement proceeded smoothly and R converged at 6.7%. The final positional and vibrational parameters, with their standard deviations, are given in Tables 1 Table 2. Vibrational parameters and their standard deviations

All values have been multiplied by 10^4

The temperature factor	has the form:	$\exp[-(B_{11})]$	$h^2 + B_{2}k^2 +$	$-B_{22}l^2 +$	$B_{10}hk + B_{10}hk$	$a_{12}hl + B_{22}$	<i>kl</i>)1
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									 10	20 . 2		
Atom	B_{11}	σ(11)	B_{22}	$\sigma(22)$	B_{33}	$\sigma(33)$	B_{12}	$\sigma(12)$	B_{13}	$\sigma(13)$	B_{23}	$\sigma(23)$
s	052	002	052	002	183	009	014	003	-015	011	015	011
O(1)	123	010	123	010	388	053	049	020	-156	041	156	041
C(1)	081	008	068	007	250	032	-027	012	-015	032	- 088	028
C(2)	075	009	075	009	214	036	-002	016	-020	046	020	046
Cl(1)	060	003	060	003	228	013	000	000	000	000	000	000
O(2)	165	010	150	010	668	046	079	019	061	056	375	037
Cl(2)	060	003	060	003	240	017	-022	006	000	000	000	000
O(3)	090	020	154	013	636	094	-044	026	021	086	101	059
O(4)	235	046	235	046	386	087	-083	071	340	116	- 340	116
O(5)	180	030	180	030	687	135	-008	054	- 349	126	349	126

Table 3. Comparison of observed and calculated structure factors for [(CH₃)₃SO]ClO₄

Within each group, the numbers are (left to right) h, $|F_0|$ and F_c . Those labeled U were observed to be less than $\sqrt{2}$ times the value given or $\sqrt{3}$ times for those with h, k or l=0. Those labeled E are believed to have been subject to extinction. Reflections labeled U or E were not used to obtain the parameters given in Tables 1 and 2

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and 2 respectively. A three-dimensional difference Fourier summation based on these values showed no significant maxima or minima. In Table 3, the calculated structure factors based on the final parameters

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are compared with the observed values. In these calculations, the following atomic scattering factors were used without correction for anomalous dispersion: Cl and S, Dawson (1960); oxygen, Hoerni & Ibers (1954) and carbon, McWeeny's values for diamond (1954).

Discussion of the structure

A projection of the structure of trimethyloxosulfonium perchlorate on (001) is shown in Fig. 1 while a projection on (110) is shown in Fig. 2. In these figures each ion of the disordered type is shown in only one of the two possible positions open to it. The bond distances and angles and the structurally significant nonbonded separations are given in Tables 4 and 5 respectively. The bonded distances and angles include correction for librational motion (Cruickshank, 1956, 1961). These corrections were applied successively in directions normal to each of the three principal axes of libration, a procedure in keeping with the more



Fig. 1. Projection of the structure of $[(CH_3)_3SO]^+ClO_4^-$ on (001). Alternate positions of the oxygen atoms in one of the disordered perchlorate ions are shown by means of dots. The $(CH_3)_3SO^+$ ions are shaded.



Fig. 2. Projection of the structure of $[(CH_3)_3SO]^+ClO_4^-$ on (110). Alternate positions of the oxygen atoms in one of the disordered perchlorate ions are shown by means of dots. The $[(CH_3)_2SO]^+$ ions and disordered ClO_4^- in the same mirror plane are shaded.

 Table 4. Bond distances and angles with their standard

 deviations and corrections for implied librational motion

		Uncorrected	Corrected
(a)	Trimethyloxosulfoni	um ion	
	S-O(1)	1.424 ± 0.013 Å	1·45 Å*
	S-C(1)	1.746 ± 0.011	1.80^{+}
	S-C(2)	1.727 ± 0.014	1.78†
	C(1) - S - C(1)	$105.8 \pm 0.7^{\circ}$	
	C(1)-S-C(2)	105.7 ± 0.5	
	C(1)-S-O(1)	$112 \cdot 1 \pm 0 \cdot 5$	
	C(2)-S-O(1)	114.8 ± 0.6	
<i></i>			
(0)	Perchlorate ion (1),	(symmetry 4)	
	Cl(1)-O(2)	$1.410 \pm 0.012 \text{ Å}$	1·45 Å
	(O-CI-O)(1)	$112 \cdot 1 + 0 \cdot 9^{\circ}$	
	(O-CI-O)(2)	$108 \cdot 1 \pm 0.9$	
		()	
(c)	Perchlorate ion (11),	(disordered)	
	Cl(2) - O(3)	1.414 + 0.020 Å	1·45 Å
	Cl(2) - O(4)	1.413 + 0.035	1.45
	Cl(2) - O(5)	1.488 ± 0.035	1.53
	O(3)-Cl(2)-O(3)	$106.7 \pm 1.6^{\circ}$	
	O(3)-Cl(2)-O(4)	110.7 ± 1.2	
	O(3)-Cl(2)-O(5)	109.7 ± 1.2	
	O(4) - Cl(2) - O(5)	109.2 ± 1.7	

* Based on vibrational parameters of S and O(1) only. † Based on assumption that librational motion of the trimethyloxosulfonium is same as that of perchlorate ions.

Table 5. Interionic packing distances in trimethyloxosulfonium perchlorate

$\begin{array}{c} C(1)-O(1) \\ C(1)-O(1') \\ C(1)-O(2) \\ C(1)-O(3) \\ C(1)-O(3') \\ C(1)-O(3') \end{array}$	3·38 Å 3·45 3·35 3·34 3·43 2·17	$\begin{array}{c} C(1)-O(5) \\ C(2)-O(2) \\ C(2)-O(3) \\ C(2)-O(3') \\ C(2)-O(4) \\ C(2)-O(4) \\ C(3) \\ O(5) \end{array}$	3·28 Å 3·58 3·30 3·47 3·49 2:45
C(1)–O(4)	3·17 O-O (min.) C-C (min.)	C(2)–O(5) 3·13 Å 4·52	3.42

Sum of van der Waals radii: CH₃-CH₃, 4.00 Å; CH₃-O, 3.40 Å; O-O, 2.80 Å

Table 6. Principal r.m.s. values of rigid body translational and librational amplitudes Standard deviations 0.01 Å and 1°

	(Å)	(Å)	(Å)	(°)	(°)	(°)
$(CH_3)_3SO^+$	0.20	0.19	0.18	10	10	10
ClO_4^{-} (I)	0.22	0.21	0.19	16	11	8
ClO_4^- (II)	0.21	0.21	0.19	15	15	9

* Based on vibrational parameters of S and O(1) only.

recent publication. For the perchlorate ions, r.m.s. librational amplitudes of 8° to 16° (Table 6) lead to an average increase of 0.04 Å in the Cl–O bond lengths. Within experimental error, a regular tetrahedron of oxygen atoms surrounds each chlorine atom, and the average Cl–O bond distance of 1.46 Å is in excellent agreement with the average value, 1.464 ± 0.007 Å, reported by Truter, Cruickshank & Jeffrey (1960) in

nitronium perchlorate and by Nordman (1962) in hydronium perchlorate.

The trimethyloxosulfonium ion approximates closely to the point group symmetry 3m. A rigid body analysis of thermal motion of this ion is unsatisfactory because of the great difference in the thermal parameters of the oxygen and carbon atoms. Librational corrections based on thermal motion exclusive of that of oxygen are insignificant; this would seem inconsistent with the large torsional oscillations of the perchlorate groups. After subtraction of the sulfur thermal parameters, the net thermal motion for the oxygen is exclusively normal to its bond to sulfur. implying librational r.m.s. amplitudes of 10° and 11°. If correction for this motion is applied to the sulfuroxygen bond length, the latter becomes 1.454 Å, but leaving the sulfur-carbon distances at their uncorrected values would imply that the motion of the oxygen atom is chiefly vibrational rather than librational, *i.e.* that the $(CH_3)_3SO^+$ system could not be regarded as a rigid body. However, if the carbon thermal parameters are too low and the $(CH_3)_3SO^+$ ion does undergo as large librations as the perchlorate ions, then the sulfur-carbon bond lengths should be increased by 0.05 Å to about 1.79 Å and there would no longer be a significant shortening of these bonds from the usual bond length of 1.80 Å. This possibility is accordingly quite attractive and is suggested tentatively as the cause of the observed anomaly,

The S–O bond distance in the $(CH_3)_3SO^+$ ion is of considerable interest. The observed value of 1.45 ± 0.02 Å (corrected, as indicated, for librational motion) is intermediate between the value of 1.411 ± 0.009 Å in sulfamide which resulted from further refinement of the data of Trueblood & Mayer (1956) by one of the present authors (P. K. G.), and the values of $1.471 \pm$ 0.008 Å and of 1.48 ± 0.01 Å in sulfate ion reported by Larson (1961) in Li₂SO₄. H₂O and by Atoji & Rundle (1958) in CaSO₄. 2H respectively.

The packing of the ions is closely related to that in CsCl with distortions caused by deviations of the ions from true spheres. All of the packing distances are reasonable and appear to be exclusivley between methyl groups and oxygen. The interionic C(1)-O(4)and C(1)-O(5) separations of $3\cdot17$ and $3\cdot28$ Å do appear somewhat shorter than the sum of the van der Waals radii for a methyl group and an oxygen atom, $2\cdot0+1\cdot4=3\cdot4$ Å. However, if the oxygen is considered to approach the methyl group in a plane bisecting one of the H-C-H bond angles, a distance of $3\cdot2$ Å is not unreasonable.

In an attempt to find the reasons for the disorder, consideration was given to possible ordered positions for the ClO_4^- ions of the second type which would conform to the required mm2 symmetry. In these considerations, the positions of the $(\text{CH}_3)_3\text{SO}^+$ ions and the ClO_4^- ions of the first type were regarded as fixed in their observed locations. The ClO_4^- groups of the second type were given the same dimensions as those of the first type but the z parameter of the Cl atom was permitted to vary from 0 to 1.00 with the ion as a whole in each of the two permitted orientations 90° apart. In one of the two orientations, the entire range of z parameters involves at least two O-C contacts less than 3.10 Å. In the other orientation there is one narrow region at about z=0.95 which comes close to being satisfactory. However, even this position involves four O-C contacts of 3.1 Å and two of 3.2 Å. Furthermore, this places the ClO_4^- ion in a position at a potential maximum with respect to rotation about an axis normal to one of the two mirror planes. Thus, even the best ordered position is unstable. Rotation of the ClO_4^- ion out of one of the mirrors and translation so that the z parameter of chlorine is at the observed value of 0.914 gives a position at a potential minimum with more satisfactory packing distances. Since the rotation may be made in either of two equivalent directions, the observed disorder results.

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Die Kristallstruktur von Äthyl-Lithium

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The crystal structure of ethyl-lithium has been determined from X-ray diffraction data. The space group is $Pcan(D_{2h}^{14})$, with a = 7.24, b = 8.27, c = 18.11 Å. The unit cell contains $16 C_2 H_5 Li$ units which form double layers (perpendicular to c) running through the whole crystal. The $C_2 H_5 Li$ molecules are combined in fours with their Li atoms in a nearly regular tetrahedral arrangement surrounded by the ethyl groups. The tetramer is built up of two dimers $(C_2 H_5 Li)_2$ which are strongly associated to each other.

A complex system of electron-deficient bonds for the structure is discussed.

Gang der Strukturbestimmung

Die Substanz kristallisiert in farblosen Blättchen, deren mechanische Eigenschaften glimmerähnlich sind. Abmessungen der Elementarzelle, sowie eine Projektion der Elektronendichte wurden bereits früher veröffentlicht (Dietrich, 1959) und dienten als Grundlage für die dreidimensionale Bearbeitung der Struktur: $a_0=7,24$, $b_0=8,27$, $c_0=18,11$ Å, V=1084 Å³, n=16 (Einheiten C₂H₅Li). Die tatsächliche Raumgruppe konnte erst gegen Ende der Strukturbestimmung ermittelt werden, da die Auslöschungsbedingungen* auf keine Raumgruppe ganz passten, also teilweise von einer Eigensymmetrie der Molekülassoziate herrühren mussten. Der grösste Teil der

^{*} In der vorläufigen Mitteilung (Dietrich, 1959) wurde die Auslöschungsbedingung, hk0 nur mit h+k=2n, nicht erwähnt, weil dieser Bedingung zunächst der Reflex 120 zu widersprechen schien. Eine genauere Untersuchung der betreffenden Schwärzungsflecken auf den Filmen (1kl) und (hk0) zeigte aber, dass es sich dabei nicht um den Reflex 120 handeln konnte, da die Flecken unter einem etwas grösseren Beugungswinkel entstanden waren, als dem Reflex 120 zukommt. Die Herkunft der fraglichen Beugungsflecken wurde nicht geklärt.