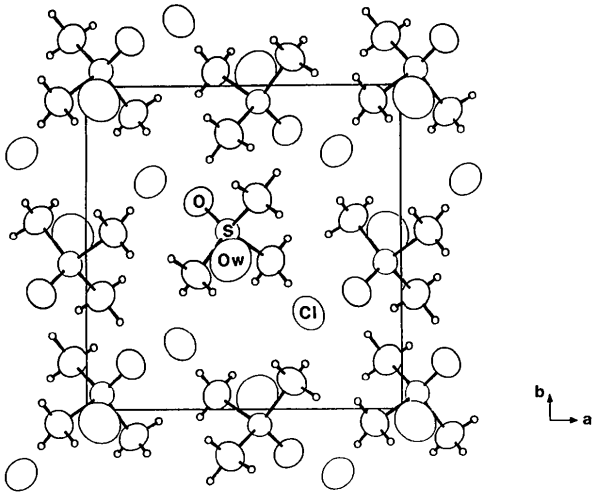

Fig. 1. Projection along the *c* axis of the atomic arrangement of $(CH_3)_3SOCl.H_2O$.

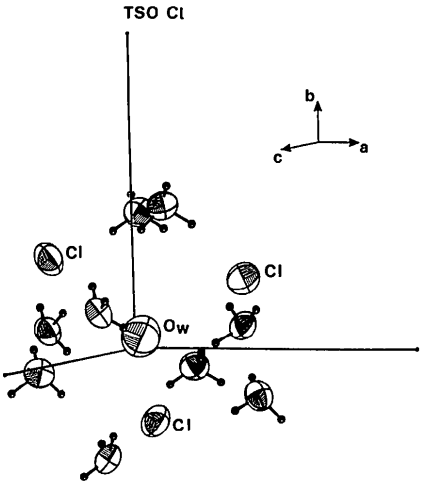

Fig. 2. The surroundings of the O(*w*) atom.

Fig. 1 and the environment of the O(*w*) atom is in Fig. 2.

Related literature. Very little is known about the structures of trimethyloxosulfonium salts, only the structures of the perchlorate (Coulder, Gantzel & McCullough, 1963) and the fluoroborate (Zimmermann, Barlow & McCullough, 1963) have been described at 293 K. In a previous paper, we reported the structure of the isotypic iodide and bromide salts (Jannin, Puget, de Brauer & Perret, 1991).

This work was carried out at the Centre de Diffractométrie de l'Université de Bourgogne.

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Structures of Trimethyloxosulfonium Salts. III. The Nitrate

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Abstract. $[(CH_3)_3SO]NO_3$, $M_r = 155{\cdot}7$, orthorhombic, *Pbca*, $a = 11{\cdot}048\ (2)$, $b = 11{\cdot}238\ (2)$, $c = 11{\cdot}549\ (2)$ Å, $V = 1434{\cdot}0\ (6)$ Å^3, $Z = 8$, $D_m = 1{\cdot}42\ (1)$, $D_x = 1{\cdot}437$ Mg m^{-3}, λ(Mo $K\alpha$) = 0·71073 Å, μ(Mo $K\alpha$) = 0·3852 mm^{-1}, $F(000) = 656$, $T = 293$ K, final $R = 0{\cdot}034$ and $wR = 0{\cdot}032$ for 683 independent observed reflections. The structure contains the planar anions NO_3^- and pyramidal cations $[(CH_3)_3OS]^+$, which closely approximate to symmetry 3*m*. The H atoms were located.

Experimental. The preparation of the crystals is described elsewhere (de Brauer & Perret, 1991). Prismatic colorless crystals, density measured by pycnometry in xylene, $D_m = 1{\cdot}42\ (1)$ Mg m^{-3}, crystal size 0·22 × 0·27 × 0·25 mm. Enraf–Nonius CAD-4

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diffractometer, monochromated Mo $K\alpha$. Systematic absences $hk0$ ($h = 2n$), $0kl$ ($k = 2n$), $h0l$ ($l = 2n$), space group *Pbca* (No. 61). Unit-cell constants from least-squares refinement of 25 reflections with $7 < \theta < 15°$. $\omega/2\theta$ scan, scan width 1·2°, $1 < \theta < 25°$, $-12 < h < 12$, $-13 < k < 13$, $-13 < l < 6$. Four orientation reference reflections measured every 200 scans (220, 202, 022, 040): no significative variation. Intensities of the same reference reflections recorded every 2 h: fading of 17·9% during 316·1 h; decay correction. 8700 measured reflections, 3827 reflections with $I > 3\sigma(I)$. Lorentz and polarization corrections. Absorption correction from ψ scan: relative transmission factor between 0·923 and 1·000. 696 independent reflections after averaging: $R_{int} = 0·027$.

Patterson function used for structure determination in *Pbca*. H atoms from difference Fourier synthesis. Anisotropic full-matrix least-squares refinement (on F) for non-H atoms, isotropic for H atoms. 119 parameters and unit weights. Final refinement with 683 reflections (five rejected as unobserved, eight rejected as bad): final $R = 0·034$, $wR = 0·032$, $S = 1·040$. Maximum and minimum heights in final difference Fourier synthesis 0·27 and $-0·22$ e Å^{-3}, maximum $(\Delta/\sigma) = 0$. Scattering factors for neutral atoms and f',f'' from *International Tables for X-ray Crystallography* (1974, Vol. IV). Enraf–Nonius (1977) *SDP* used for all calculations. Computer used: VAX 730.

Final atomic coordinates and equivalent isotropic thermal parameters are reported in Table 1; Table 2 gives the main interatomic distances and bond angles.* The NO_3 group is quasi-planar as usual; the N atom is surrounded by three O atoms, O(1), O(2) and O(3), making a quasi-equilateral triangle; it should be noted that these three O atoms are very mobile, specially O(3) with $B_{eq} = 12·2$ Å^2. In this compound, the $(CH_3)_3SO$ group has neither a symmetry plane nor axis; nevertheless, it closely approximates to the $3m$ symmetry assumed for the free cation. The projection of the atomic arrangement of $[(CH_3)_3SO]NO_3$ along the a axis is given in Fig. 1.

Related literature. As we have noted previously, very little is known about the structures of trimethyloxosulfonium salts. In this series of papers, the structures of some new salts are determined. We have already reported the iodide, bromide and chloride structures (Jannin, Puget, de Brauer & Perret, 1991*a*,*b*).

* Lists of structure factors, anisotropic thermal parameters, H-atom parameters and interatomic contacts have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 53845 (8 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Table 1. *Final atomic coordinates for* $[(CH_3)_3SO]NO_3$ *with e.s.d.'s in parentheses*

$B_{eq} = (4/3)\sum_i\sum_j \mathbf{a}_i\mathbf{a}_j\beta_{ij}$.

	x	y	z	B_{eq}(Å^2)
N	0·2687 (3)	0·5039 (3)	0·0209 (3)	4·17 (8)
O(1)	0·2817 (4)	0·5950 (3)	0·0731 (3)	8·2 (1)
O(2)	0·3301 (4)	0·4812 (2)	−0·0631 (3)	9·3 (1)
O(3)	0·1924 (4)	0·4343 (43)	0·0474 (4)	12·3 (1)
S	0·02809 (9)	0·22307 (8)	0·31329 (8)	2·91 (2)
O	−0·0437 (2)	0·2931 (2)	0·2352 (2)	4·48 (6)
C(1)	0·1826 (3)	0·2567 (4)	0·3033 (4)	4·08 (9)
C(2)	0·0146 (4)	0·0718 (4)	0·2860 (4)	4·6 (1)
C(3)	−0·0141 (4)	0·2466 (4)	0·4574 (3)	4·7 (1)

Table 2. *Main interatomic distances* (Å) *and bond angles* (°) *for* $[(CH_3)_3SO]NO_3$

Nitrate anion			
N—O(1)	1·198 (5)	O(1)—N—O(2)	121·1 (5)
N—O(2)	1·211 (5)	O(1)—N—O(3)	121·2 (4)
N—O(3)	1·189 (6)	O(2)—N—O(3)	117·7 (4)
O(1)—O(2)	2·097 (5)	O(2)—O(1)—O(3)	58·9 (3)
O(1)—O(3)	2·079 (7)	O(1)—O(2)—O(3)	60·1 (3)
O(2)—O(3)	2·055 (6)	O(1)—O(3)—O(2)	60·9 (2)
Trimethyloxosulfonium cation			
S—O	1·436 (3)	O—S—C(1)	112·3 (2)
S—C(1)	1·752 (4)	O—S—C(2)	112·0 (2)
S—C(2)	1·736 (5)	O—S—C(3)	111·6 (2)
S—C(3)	1·749 (4)	C(1)—S—C(2)	106·5 (2)
		C(1)—S—C(3)	106·8 (2)
		C(2)—S—C(3)	107·3 (2)

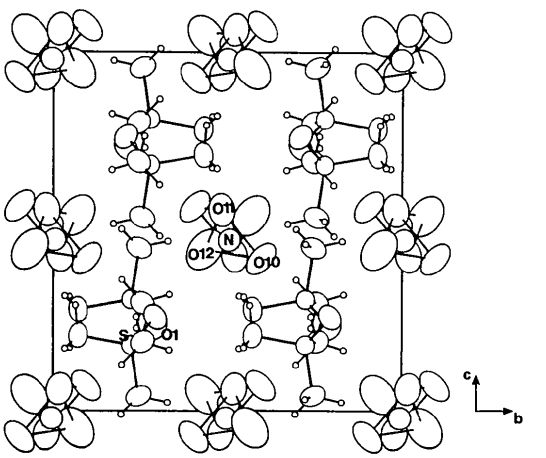

Fig. 1. Projection along the a axis of the atomic arrangement of $[(CH_3)_3SO]NO_3$.

This work was carried out at the Centre de Diffractométrie de l'Université de Bourgogne.

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