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Structure of Trimethylammoniosulphonate, $C_3H_9NO_3S$

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Abstract. $M_r = 139{\cdot}2$, rhombohedral, $R3m$, $a = 5{\cdot}73\,(1)$ Å, $\alpha = 107{\cdot}4\,(1)°$, $V = 154{\cdot}8$ Å^3, $Z = 1$, $D_m = 1{\cdot}50$, $D_c = 1{\cdot}49$ Mg m^{-3}, $\lambda(\text{Mo } K\alpha) = 0{\cdot}7107$ Å, $\mu(\text{Mo } K\alpha) = 0{\cdot}44$ mm^{-1}, $T = 293$ K, $R = 0{\cdot}029$ for 217 observed reflections. $(CH_3)_3N(SO_3)$ was made by reacting trimethylamine in chloroform with chlorosulphonic acid at 273 K. The molecule has trigonal symmetry with the N—S bond constrained to the crystallographic threefold axis. The N—S distance [1·844 (2) Å] is longer than that found in $H(CH_3)_2N(SO_3)$ [1·790 (6) Å] and $H_3N(SO_3)$ [1·76 (2) Å].

Introduction. As part of a comprehensive investigation into the chemistry of potassium salts of methyl-substituted sulphamic acids (Morris, Kennard, Hall & Smith, 1982), the X-ray structure of the title compound (Shimizu & McLachlan, 1953) was redetermined.

Experimental. Source: synthesized by reacting trimethylamine in chloroform at 273 K with chlorosulphonic acid and recrystallized from water to give colourless prismatic crystals; composition, found: C 26·0, H 6·7, N 10·0%; calculated for $C_3H_9NO_3S$: C 25·9, H 6·5, N 10·1%; D_m measured by flotation; X-ray data collected from one crystal (0·2 × 0·2 × 0·15 mm) for a hemisphere of the reciprocal lattice ($2\theta_{\max}$ 50°), analysis of equivalent reflections confirmed the reported space group (Shimizu & McLachlan, 1953); 10 high-angle reflections used to measure lattice parameters; of the 959 reflections, 217 with $I > 2{\cdot}5\sigma(I)$ were unique, and used in the structure determination; Syntex $P2_1$ four-circle diffractometer (2θ–ω scanning mode), graphite-monochromatized Mo $K\alpha$ radiation ($\lambda = 0{\cdot}7107$ Å); no absorption or extinction correction; with one molecule in the rhombohedral cell and S and N atoms lying on the threefold axis, a sharpened Patterson synthesis located the x coordinate for N, leaving the S at the origin; other atoms were located from a subsequent structure factor–Fourier calculation; full-matrix anisotropic refinement of non-hydrogen atoms (H isotropic, $U = 0{\cdot}05$ Å^2) reduced R from 0·43 to 0·029 and R_w to 0·032;* a final difference Fourier map showed no peaks greater than 0·13 e Å^{-3}; reflections were given the weights $w = 3{\cdot}1/[\sigma^2(F_o) + 0{\cdot}0006\,(F_o)^2]$; $F(000) = 74$; all computations completed using *SHELX* (Sheldrick, 1976); neutral-atom scattering factors for non-hydrogen atoms (Cromer & Mann, 1968) and H (Stewart, Davidson & Simpson, 1965).

Discussion. The N and S atoms in the $(CH_3)_3N(SO_3)$ molecule (Fig. 1; atomic parameters in Table 1) lie on the threefold axis of the rhombohedral cell, with methyl groups (about the N) and O atoms (about the S) related by this threefold rotational symmetry. The interatomic distances and angles (Table 2) indicate comparable results to those found for *N,N*-dimethylsulphamic acid (Morris, Kennard, Hall & Smith, 1982) and sulphamic acid (Sass, 1960) except for the N—S distance. The significant extension from 1·790 (6) [$H(CH_3)_2N(SO_3)$] to 1·844 (2) A [$(CH_3)_3N(SO_3)$] could be due to bulkiness of the third methyl group around the N. The previous investigation (Shimizu & McLachlan, 1953) reported a rhombohedral cell $a = 5{\cdot}71$ Å and $\alpha = 107{\cdot}66°$, space group $R3m$. Their trial-and-error determination based on four possible models suggested a stationary $N(CH_3)_3$ with the SO_3 group rotating about the molecular axis. No such possibility was found in this determination.

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* Lists of structure factors and anisotropic thermal parameters have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 38083 (3 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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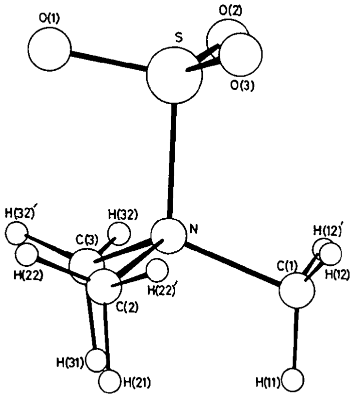

Fig. 1. Molecular stereochemistry and atom numbering. Atoms designated as (2) and (3) are related to (1) by crystallographic threefold rotational symmetry.

Table 1. *Atomic parameters of non-hydrogen atoms* ($\times 10^4$) *and hydrogen atoms* ($\times 10^3$) (*with estimated standard deviations in parentheses*)

U_{eq}($\text{Å}^2 \times 10^4$) is defined as $(U_{11}U_{22}U_{33})^{1/3}$.

	x	y	z	Wyckoff notation	U_{eq}
S	0	0	0	*a*	333 (4)
N	2931 (7)	2931 (7)	2931 (7)	*a*	278 (12)
O	−1317 (5)	−1317 (5)	1263 (7)	*b*	692 (15)
C	4616 (8)	4616 (8)	1977 (9)	*b*	474 (18)
H(11)	617 (8)	617 (8)	357 (9)	*b*	
H(12)	328 (7)	502 (8)	87 (8)	*c*	

Table 2. *Interatomic distances* (Å) *and angles* (°) *with estimated standard deviations in parentheses*

	$(CH_3)_3N(SO_3)^a$	$H(CH_3)_2N(SO_3)^b$ (mean results)	$H_3N(SO_3)^c$ (mean results)
N—S	1·844 (2)	1·790 (6)	1·76 (2)
N—C	1·496 (2)	1·504 (10)	—
S—O	1·405 (2)	1·430 (5)	1·44 (2)
C—N—C	109·1 (1)	111·5 (7)	—
S—N—C	109·8 (1)	112·3 (5)	—
N—S—O	101·8 (1)	102·1 (3)	103·2 (9)
O—S—O	115·9 (1)	115·8 (4)	115·1 (9)

References: (*a*) this work; (*b*) Morris, Kennard, Hall & Smith (1982); (*c*) Sass (1960).

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Structure of the Tetrapeptide L-Methionyl-L-α-glutamyl-L-histidyl-L-phenylalanine Monohydrate, $C_{25}H_{34}N_6O_7S.H_2O$

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Abstract. $M_r = 580{\cdot}68$, $C2$, $a = 23{\cdot}333\ (6)$, $b = 5{\cdot}4741\ (11)$, $c = 24{\cdot}783\ (5)$ Å, $\beta = 115{\cdot}03\ (2)°$, $U = 2868\ (2)$ Å^3, $Z = 4$, $D_x = 1{\cdot}344$ Mg m^{-3}, $\lambda(\text{Mo } K\bar{\alpha}) = 0{\cdot}71069$ Å, $\mu = 0{\cdot}169$ mm^{-1}, $F(000) = 1232$; 3494 independent reflections were measured at room temperature with Mo radiation on a diffractometer. The crystal structure was determined by a Patterson search based on dipeptide fragments with a large range of φ, ψ values. The conformation with the best 'measure of fit' was extended to an almost complete structure model by *DIRDIF*. Least-squares refinement gave $R = 0{\cdot}055$ for 3113 reflections with $w = 1$. The molecule is a double zwitterion with (terminal)NH_3^+, (terminal)CO_2^-, (Glu)CO_2^- and (imidazole)H^+ as charged groups. The backbone shows a helix-type bend at Glu and is almost fully extended at His with N—Cα(His)—C = 105·6 (4)°; NH and C=O of His are not involved in H bonding. The crystal contains regions with strong H