cube retains threefold symmetry with Nb and Li(3) lying on the threefold axis. Along [100], alternating zigzag chains of all Li atoms and chains containing one Nb atom per unit cell are apparent. Half the chains contain Li atoms exclusively; the other half contain one Nb atom and three Li atoms per unit cell. Each Nb atom is coordinated tetrahedrally by nitrogen at an average distance of 1.95 Å. This distance is much smaller than that found in the sixfold coordinate Nb in NbN (2.35 Å) (Christensen, 1977), but as expected for a lower coordination number, the Nb—N bond length is comparable to the W—N bond length (1.86 Å) in the $[WN_4]^{6-}$ tetrahedra found in Ba_3WN_4 (Gudat, Hohn, Kneip, Rabenau, 1991).

It is interesting to note that Li_7VN_4 (space group $P43n$, $a = 9.60$ Å) is not reportedly isostructural with the title phase (Juza, Giernn & Haug, 1959). Along [100] in the Nb phase, the closest Nb—Nb distance is 5.50 Å. In the vanadium phase, there is an irregular distribution of V atoms with the closest V—V distance (4.80 Å) equal to half the lattice constant. We find it questionable that the V atoms are so close, and because the structure of the vanadium phase was determined by powder methods in which Laue symmetries $m3m$ and $m3$ are not easily distinguished, we suggest that the reported positions may be incorrect and should be redetermined by a single-crystal struc-

tural analysis. Juza *et al.* (1959) also report that Li_7NbN_4 and Li_7TaN_4 are isostructural and are rhombohedrally or tetragonally distorted structures based on Li_7VN_4. Because we have determined that Li_7NbN_4 is cubic, we also suggest that the structure of the Li_7TaN_4 phase should be determined by single-crystal studies.

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Structure of the Sodium Salt of *m*-Xylenesulfonate Monohydrate

BY J. LEFEBVRE

Laboratoire de Dynamique et Structure des Matériaux Moléculaires (UA 801), UFR de Physique, Université de Lille I, 59655 Villeneuve d'Ascq CEDEX, France

AND A. MINIEWICZ AND R. KOWAL

Institute of Organic and Physical Chemistry, Technical University of Wroclaw, Wybrzeze Wyspianskiego 27, 50-370 Wroclaw, Poland

Abstract. Sodium 4-*m*-xylenesulfonate monohydrate, $Na^+.C_8H_9SO_3^-.H_2O$, $M_r = 226.2$, monoclinic, $P2_1/c$, $a = 15.750$ (5), $b = 8.783$ (2), $c = 7.625$ (2) Å, $\beta = 99.82$ (3)°, $V = 1039$ (1) Å^3, $Z = 4$, $D_x = 1.45$ g cm^{-3}, Mo $K\alpha$, $\lambda = 0.71073$ Å, $\mu = 3.23$ cm^{-1}, $F(000) = 472$, $T = 294$ K, $R = 0.036$, $wR = 0.036$ for 2434 observations. The structure is formed by alternative stacks of *m*-xylenesulfonate and water molecules along the c axis, with O—H···O hydrogen bonds. The Na ions are situated at the centres of octahedra formed with O atoms of the SO_3 group and water molecule.

Introduction. It has already been established (Marder, Perry & Schaefer, 1989) that noncentrosymmetric organic salts can be used in optical information processing owing to their second-order nonlinear optical properties (NLO) (Chemla & Zyss,

1987). Among various counterions used for preparation of these salts, the salts with p-$CH_3C_6H_4SO_3^-$ gave exceptionally large power second-harmonic generation of light (SHG) efficiencies (Marder *et al.*, 1989).

In order to obtain an NLO crystal of good optical quality we commenced preparation of salts containing the $(CH_3)_2C_6H_3SO_3^-$ anion. In this communication we present structural data for the sodium salt of *m*-xylenesulfonate monohydrate. As will be seen this compound crystallizes in a centrosymmetric point group and thus does not exhibit second-order nonlinear optical properties.

Experimental. 2,4-Dimethylbenzene was sulfonated with sulfuric acid. The product was treated subsequently with a diluted aqueous solution of NaOH or 10% aqueous solution of Na_2CO_3 in order to obtain the sodium salt. Large transparent single crystals of the sodium salt of *m*-xylenesulfonate (*m*-XSNa) were grown from an aqueous solution at 294 K.

A spherically shaped single crystal of *m*-XSNa (0.6 mm in diameter) was prepared for X-ray structure determination. Diffraction measurements were performed with a Philips PW 1100 automatic diffractometer, using monochromated Mo $K\alpha$ radiation. Lattice parameters were determined from 25 selected reflections in the range $16 < 2\theta < 25°$. ω scans were used with $2 < \theta < 30°$ ($0.05 < \sin\theta/\lambda < 0.70$ Å^{-1}), scan width amounted to 1.6° and scan speed was 0.025° s^{-1}. Background was counted for half of the total scan time on each side of the $K\alpha$ position. Every 2 h, three standard reflections ($\overline{1}02$, $\overline{2}22$ and $2\overline{2}2$) were monitored for correction purposes; however, no significant variations in intensity were observed. 3416 reflections were measured with $-22 \le h \le 22$, $0 \le k \le 12$ and $0 \le l \le 10$, corresponding to 2434 non-equivalent reflections with $I > 3\sigma(I)$. Data were corrected for Lorentz and polarization effects, but not for absorption ($\mu = 3.23$ cm^{-1}, $\mu R \approx 0.1$).

Systematic absences of $h0l$ reflections for $l = 2n + 1$ and $0k0$ reflections for $k = 2n + 1$ implied $P2_1/c$ as space group for the *m*-XSNa crystal. Initial coordinates of Na, S and O atoms of the SO_3 group and C atoms of the benzene ring were found by direct methods (*MULTAN*77; Main, Lessinger, Woolfson, Germain & Declercq, 1977). Refinement on F. Subsequent Fourier syntheses enabled determination of the positions of the C atoms of the methyl groups and the O atom of the water molecule. Further Fourier syntheses, made after several refinement cycles, enabled localization of the H atoms. In order to avoid extinction effects, 21 low-angle and high-intensity reflections were removed (in fact, all reflections were measured with an attenuator). Further

refinements using anisotropic temperature factors for heavy atoms and isotropic ones for H atoms gave $R = 0.036$ {$wR = 0.036$, with the following weighting scheme: $w = [\sigma^2(F) + 25 \times 10^{-6}F^2]^{-1}$}. The shifts in atomic parameters in the final refinement cycle were less than 0.1σ; $S = 0.047$.

The a_i, b_i and c_i coefficients used for the analytical approximation to the atomic scattering factors came from *International Tables for X-ray Crystallography* (1974, Vol. IV); anomalous-dispersion effects for Na and S were taken into account (Cromer & Liberman, 1970). A final difference synthesis showed peak values of 0.39 and -0.45 e Å^{-3}. All calculations were carried out with *SHELX*76 (Sheldrick, 1976) using a MicroVAX II computer.

Discussion. Atomic positional parameters and equivalent isotropic temperature factors for the non-H atoms are listed in Table 1; bond lengths and bond angles are presented in Table 2.* Numbering of the atoms is shown in Fig. 1 and the projection of the unit-cell contents along the b axis is given in Fig. 2. The packing of the crystal of sodium *m*-xylenesulfonate monohydrate is realized by *m*-xylenesulfonate (*m*-XS) and water molecules arranged alternately along the c axis. Contacts between an *m*-XS molecule and its neighbouring water molecules within a chain are not equivalent. For one of the water molecules, contact with the *m*-XS molecule is realized *via* a hydrogen bond: $O(3)\cdots HO(1) = 2.078$ (30), $O(3)\cdots O(4) = 2.757$ Å and $O(3)\cdots HO(1)\cdots O(4) = 172$ (3)°. For the other water molecule, there is a van der Waals contact with an *m*-XS molecule and the distance between HO(2) and the benzene ring is 2.54 Å.

There are also two possibilities of interactions between adjacent chains. In the first case, when *m*-XS molecules are related by a twofold screw axis, the contact is made by methyl groups, the dihedral angle between the mean planes of the *m*-XS being 54.6°. In the second case, where the symmetry element between the two neighbouring chains is the inversion centre, the two *m*-XS molecules are parallel and lie almost within the same plane. They are connected by sulfonate groups *via* an Na ion.

In fact, the Na ions are situated at the centre of an octahedron formed by six O atoms: two O(1) and two O(2) (each of them belonging to four different *m*-XS molecules) and also two O(4) atoms of water molecules. The Na$\cdots$O lengths and O$\cdots$Na$\cdots$O angles

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* Lists of structure factors, final atomic coordinates, anisotropic thermal parameters and bond lengths and angles involving the H atoms have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 54716 (21 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Table 1. *Positional parameters* (× 10⁴) *and equivalent isotropic temperature factors* (Å² × 10³) *for sodium 4-m-xylenesulfonate monohydrate*

U_{eq} is defined as one third of the trace of the orthogonalized U_{ij} tensor.

	x	y	z	U_{eq}
C(1)	7187 (1)	4595 (2)	3425 (2)	32 (1)
C(2)	7859 (1)	5640 (2)	3901 (2)	38 (1)
C(3)	8655 (1)	5281 (2)	3461 (2)	49 (2)
C(4)	8809 (1)	3956 (2)	2580 (2)	56 (2)
C(5)	8137 (1)	2948 (2)	2139 (2)	50 (2)
C(6)	7334 (1)	3255 (2)	2564 (2)	36 (1)
C(7)	7748 (2)	7120 (3)	4837 (4)	58 (3)
C(8)	9681 (2)	3639 (5)	2081 (5)	101 (4)
S	6143 (1)	4927 (1)	3935 (1)	38 (1)
O(1)	5621 (1)	3649 (1)	3165 (2)	58 (1)
O(2)	5844 (1)	6359 (1)	3150 (1)	44 (1)
O(3)	6253 (1)	4958 (2)	5855 (2)	93 (2)
Na	4870 (1)	2008 (1)	4897 (1)	48 (1)
O(4)	5940 (1)	98 (1)	4302 (2)	45 (1)

Table 2. *Bond lengths* (Å) *and bond angles* (°) *for sodium 4-m-xylenesulfonate monohydrate*

C(1)—C(2)	1.401 (2)	C(4)—C(5)	1.377 (2)
C(1)—C(6)	1.386 (2)	C(4)—C(8)	1.512 (4)
C(1)—S	1.776 (2)	C(5)—C(6)	1.384 (2)
C(2)—C(3)	1.388 (2)	S—O(1)	1.448 (2)
C(2)—C(7)	1.507 (3)	S—O(2)	1.445 (1)
C(3)—C(4)	1.386 (2)	S—O(3)	1.445 (2)
C(2)—C(1)—C(6)	119.9 (1)	C(5)—C(4)—C(8)	120.9 (2)
C(2)—C(1)—S	121.7 (1)	C(4)—C(5)—C(6)	120.9 (2)
C(6)—C(1)—S	118.3 (1)	C(1)—C(6)—C(5)	120.6 (2)
C(1)—C(2)—C(3)	117.5 (5)	C(1)—S—O(1)	105.8 (1)
C(1)—C(2)—C(7)	122.9 (2)	C(1)—S—O(2)	107.3 (1)
C(3)—C(2)—C(7)	119.6 (2)	C(1)—S—O(3)	105.7 (1)
C(2)—C(3)—C(4)	123.2 (2)	O(1)—S—O(2)	112.6 (1)
C(3)—C(4)—C(5)	117.9 (1)	O(1)—S—O(3)	113.1 (1)
C(3)—C(4)—C(8)	121.2 (2)	O(2)—S—O(3)	111.8 (1)

are listed in Table 3. One notes that the different Na⋯O lengths are approximately equivalent while some of the O⋯Na⋯O angles differ considerably from 90 and 180°, and this leads to the formation of distorted octahedra. Each O atom belongs to two octahedra and two neighbouring octahedra have a common edge. One should also note that the O(3) atoms involved in the hydrogen bonds do not belong to these octahedra.

The mean plane of the *m*-xylenesulfonate molecule has been calculated using a least-squares procedure (H and O atoms were removed). In the orthogonal (*a*, *b*, *c**) system, the equation of this plane is given by $-0.1167\,x + 0.4593\,y - 0.8806\,z - 1.6885 = 0$, with a deviation of 0.032 Å for C(8), but for the other atoms the deviation was lower than 0.010 Å. Within the benzene ring there are five C—C bonds of length between 1.384 and 1.388 Å, whereas the sixth one, the C(1)—C(2) bond, with atoms bonded to SO₃ and CH₃ groups, respectively, has a length of 1.401 Å. Similarly, the angles S—C(1)—C(2) and C(1)—C(2)—C(7) are significantly greater than 120°. These two facts permit an increase in the distance

between the two neighbouring groups. The smallest distances found between atoms of the SO₃ and CH₃ groups are O(2)⋯H(73) = 2.556 and O(3)⋯H(72) = 2.666 Å; atoms of these two groups are not inter-meshed, as might be thought, but they do face each other. Finally the C—C—C angles, with the central C atom connected to a methyl group, are smaller than 120°: C(1)—C(2)—C(3) = 117.9 and C(3)—C(4)—C(5) = 117.5°; this permits an increase in the distance between atoms of the methyl groups and H atoms of neighbouring C—H bonds.

In the molecule of water, the O(4)—HO(1) bond length is surprisingly small [0.685 (30) Å, HO(1) being the H atoms involved in hydrogen bonding].

A careful analysis of the final Fourier synthesis map showed that the residual electron density ranges between −0.45 and 0.39 e Å⁻³. Electron density peaks appear approximately in the middle of each C—C bond of the *m*-XS molecule, the mean value of these peaks being 0.20 e Å⁻³. There is also a peak of 0.32 e Å⁻³ in the middle of the C(1)—S bond. But the most important residual electron density is seen in the vicinity of the sulfonate group: there are peaks
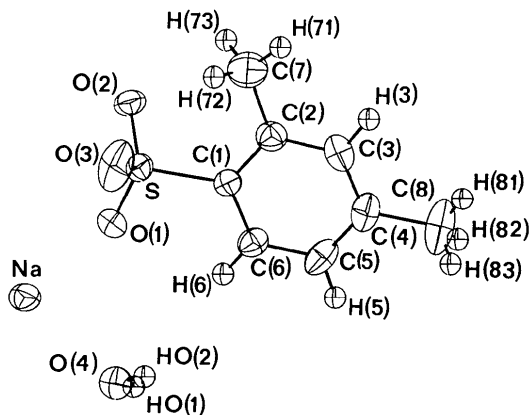

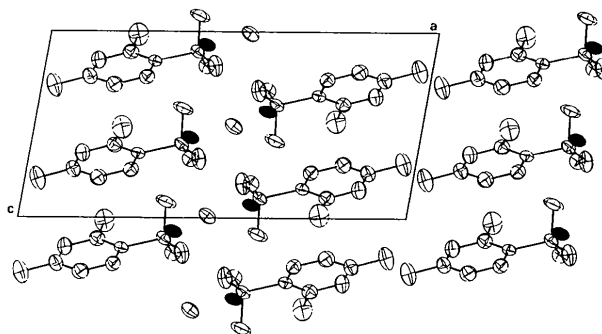Fig. 1. Atom-numbering scheme and thermal ellipsoids for *m*-XSNa.

Fig. 2. Packing projection along the *b* axis of sodium 4-*m*-xylenesulfonate monohydrate. (O atoms of water molecules are represented by filled ellipsoids.)

Table 3. *Lengths (Å) and angles (°) between* Na *and* O *neighbouring atoms*

Na···O(1)	2.399 (2)	Na···O(4)"	2.384 (2)
Na···O(4)	2.474 (2)	Na···O(1)"'	2.663 (2)
Na···O(2)'	2.472 (2)	Na···O(2)'ᵛ	2.469 (1)
O(1)···Na···O(4)	83.7 (1)	O(4)···Na···O(2)'ᵛ	83.5 (1)
O(1)···Na···O(2)'	107.5 (1)	O(2)'···Na···O(4)"	88.7 (1)
O(1)···Na···O(4)"	160.5 (1)	O(2)'···Na···O(1)"'	74.6 (1)
O(1)···Na···O(1)"'	117.9 (1)	O(2)'···Na···O(2)'ᵛ	121.3 (1)
O(1)···Na···O(2)'ᵛ	79.0 (1)	O(4)"···Na···O(1)"'	76.2 (1)
O(4)···Na···O(2)'	153.9 (1)	O(4)"···Na···O(2)'ᵛ	83.4 (1)
O(4)···Na···O(4)"	86.1 (1)	O(1)"'···Na···O(2)'ᵛ	154.0 (1)
O(4)···Na···O(1)"'	79.3 (1)		

Symmetry code: (i) $1-x$, $1-y$, $1-z$; (ii) $1-x$, $-y$, $1-z$; (iii) x, $\frac{1}{2}-y$, $\frac{1}{2}+z$; (iv) $1-x$, $-\frac{1}{2}-y$, $\frac{1}{2}-z$.

of 0.39 and 0.27 e Å^{-3} at a distance of about 0.8 Å from the S atom. These peaks and holes are located between the S—O bonds. In order to take this, and also the ionic character of Na$^+$ and SO$_3^-$, into account the refinements were made with the atomic scattering factors of Na$^+$ and SO$_3^-$. Several possibilities have been considered: first, the atomic scattering factors of neutral atoms were used, then electron partial charges were localized on different O atoms of the SO$_3^-$ ion. In all cases, however, no significant improvement in the reliability factor occurred. It seems that further improvements can be achieved by the use in calculations of non-spherical atomic scattering factors for atoms of the sulfonate ion.

An analysis of the anisotropic temperature factors (Schomaker & Trueblood, 1968), calculated from *SHELX*76 for heavy atoms of the *m*-XS molecule, shows that the whole molecule cannot be treated as a rigid body. This fact can be explained by the possibility of rotation of the SO$_3$ group around the C(1)—S axis, independently of the rest of the molecule. Such a first rigid-body analysis has been made

without O atoms and a second one with the C(1)—SO$_3$ group. Eigenvalues and eigenvectors are almost equal for the two calculations, except for the eigenvalues L_1: $T_1 = 0.0358$, $T_2 = 0.0255$, $T_3 = 0.0149$ Å^2, $L_1 = 44.3$, $L_2 = 14.2$ and $L_3 = 13.5$ deg^2 for the *m*-XS molecule without O atoms; for the C(1)—SO$_3$ group they are $T_1 = 0.0336$, $T_2 = 0.0230$, $T_3 = 0.0175$ Å^2, $L_1 = 65.2$, $L_2 = 15.7$ and $L_3 = 16.6$ deg^2. L_1 in both cases is the mean-square rotational vibration around the C(4)—C(1)—S axis and, as expected, this rotational vibration is greater for the sulfonate group (65.2 deg^2) than for the rest of the molecule (44.3 deg^2). This thermal rotational vibration, which corresponds to the 'easy' direction (*e.g.* the smallest inertia moment), is about three times greater than the rotational vibrations around the two other directions.

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Structure of a Zinc 3-Methylanthranilate Complex: (2-Amino-3-methylbenzoato)hydroxozinc(II)

BY SHARON M. BOUDREAU AND HELMUT M. HAENDLER

Department of Chemistry, University of New Hampshire, Durham, New Hampshire 03824-3598, USA

Abstract. [Zn(OH)(C$_8$H$_8$NO$_2$)], $M_r = 232.5$, monoclinic, $P2_1/c$, $a = 14.512$ (1), $b = 5.260$ (1), $c = 11.774$ (2) Å, $\beta = 111.01$ (2)°, $V = 839.0$ (4) Å^3, $Z = 4$, $D_x = 1.84$ Mg m^{-3}, λ(Cu $K\alpha$) = 1.54056 Å, $\mu =$ 3.9 mm^{-1}, $F(000) = 472$, $T = 293$ (1) K, $R = 0.037$ for 1053 reflections. The coordination of the Zn atom is tetrahedral. Each aminomethylbenzoate radical acts as a bridging ligand between two Zn