Tabelle 2 (*Fort.*)

C(4)	C(3)	O(31)	C(5)
C(3)	1,559 1,55	118,1	114,7
O(31)	117	1,206 1,28	126,9
C(5)	120	123	1,447 1,42

C(5)	P(1)	C(4)	C(32)
P(1)	1,711 1,71	116,6	120,9
C(4)	116	1,447 1,42	122,5
C(32)	119	125	1,463 1,47

mesomere Verteilung der Doppelbindung um das C(5) führt zu einer starken Verkürzung der C—C- bzw. P—C-Bindungen. Deutlicher noch als bei Saenger (1973) wird die 'beste Ebene' durch die Atome P(1), C(3), C(4), C(5), C(32), O(31) und O(33) bestimmt. Sie treten maximal nur 0,007 Å aus dieser heraus, das P(2) dagegen 0,04 Å in der Richtung, in der die Benzolringe liegen (zum Vergleich: maximale Abstände der C-Atome von den 'besten Ebenen' der Benzolringe: 0,005 Å).

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Sodium Anilinomethanesulphonate Monohydrate

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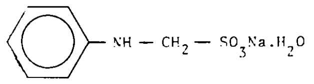
Abstract. $C_7H_8NNaO_3S.H_2O$, orthorhombic, $P2_12_12_1$, $a = 5{\cdot}342$ (2), $b = 30{\cdot}041$ (5), $c = 5{\cdot}938$ (2) Å, $M_r = 227{\cdot}22$, $Z = 4$, $D_m = 1{\cdot}575$ (flotation), $D_x = 1{\cdot}584$ Mg m^{-3}, μ(Mo $K\alpha$) = 0·37 mm^{-1}. The structure was solved by direct methods and refined by full-matrix least-squares calculations. The final R was 0·066 for 1672 measured reflections. Coordination of the Na atom is octahedral, involving four O atoms from sulphonate groups and two O atoms from water molecules. The Na—O distances are between 2·345 (2) and 2·700 (2) Å.

Introduction. Sodium anilinomethanesulphonate (I) and sodium *p*-bromoanilinomethanesulphonate (Lechat & Rosenstein, 1977) crystallize respectively as monohydrate and sesquihydrate. The degree of hydration of these compounds influences their spectra in the infrared region where a demultiplication of the O—H stretching band and a shift of the N—H stretching band are observed (De Groote, 1978). This investigation has been undertaken in order to establish a correlation between the behaviour of aminomethanesulphonate compounds in the IR region and their crystal structure. Further aminomethanesulphonate compounds in the aromatic and aliphatic series are being studied at present.



(I)

Crystals of (I) were kindly provided by Professor R. A. M. C. De Groote, of the Chemistry and Molecular Physics Department, Institute of Physics and Chemistry of São Carlos.

A crystal of dimensions 0·2 × 0·6 × 0·3 mm was used for the data collection on a Nonius CAD-4 diffractometer. The cell parameters were refined from the setting angles for 25 reflections. The three-dimensional intensity data were collected using graphite-monochromated Mo $K\alpha$ radiation up to $2\theta = 60°$. The ω–2θ scanning mode with varying interval was used. Of the 1672 recorded independent reflections, 1532 were considered observed above background [$I > 2\sigma(I)$, where σ was based on counting statistics]. The data were reduced to structure factors without absorption correction. The structure was solved by application of *MULTAN* (Germain, Main & Woolfson, 1971) to the 254 reflections with $E > 1{\cdot}40$. All the

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heavy atoms appeared clearly in the E map based on the set of phases giving the highest figures of merit. The H atoms of the anilinomethanesulphonate residue were placed at their calculated positions; the water molecule H atoms were located by a difference synthesis. The H atoms were included in the refinement, which was carried out by full-matrix least-squares calculations with anisotropic thermal parameters for the heavy atoms and isotropic thermal parameters equal to 6·0 Å^2 for the H atoms. The function minimized was

$$\sum w_i(K|F_o| - |F_c|)^2$$

where

$$w_i = [1\text{–}5 \times 10^{-2}K|F_o| + 10^{-3}(K|F_o|)^2]^{-1},$$

based on structure factor statistics, and $w_i = 0$ for the unobserved reflections.

The atomic scattering factors used were those of Cromer & Waber (1974) and the dispersion correction coefficients were those of Cromer & Ibers (1974). The final refinement cycle gave $R = 0·066$ for all 1672 reflections and $R = 0·052$ for the 1532 observed reflections.

The final atomic parameters are given in Table 1.*

Discussion. The Na atom is at the centre of a distorted octahedron whose vertices are occupied by O atoms belonging to suphonate groups and water molecules.*

Two O atoms, O(2) and O(4), belonging to the sulphonate group and the water molecule respectively, are shared by two Na atoms related by the twofold screw axis along the c direction. The distortion of the octahedron is due to the fact that O(2) and O(3), belonging to the same sulphonate residue, are coordinated to the same Na atom, and that O(2) is coordinated to two Na atoms. As a result, a very low value of the O(2)—Na—O(3) angle, 56·24 (6)°, as well as a long Na—O(2) distance, 2·700 (2) Å, is observed, the other angles

Table 1. *Fractional atomic coordinates* (×10^5, *for* H ×10^3) *with their e.s.d.'s*

	x	y	z
S	−11794 (17)	7275 (3)	58121 (16)
Na	44183 (34)	2358 (6)	79657 (30)
O(1)	15197 (56)	7130 (11)	61255 (58)
O(2)	−21491 (67)	3301 (10)	46632 (61)
O(3)	−25570 (67)	8181 (12)	78665 (58)
O(4)	16598 (69)	−3545 (11)	67466 (60)
N	−751 (84)	12038 (12)	21199 (64)
C(1)	17869 (81)	15309 (13)	19864 (71)
C(2)	32143 (104)	15597 (16)	52 (83)
C(3)	51159 (111)	18691 (17)	−1875 (95)
C(4)	56059 (107)	21635 (17)	15731 (112)
C(5)	42077 (106)	21425 (17)	35177 (101)
C(6)	23342 (99)	18239 (16)	37661 (85)
C(7)	−18074 (78)	11839 (14)	39438 (95)
H(1)	−59 (17)	112 (3)	96 (14)
H(2)	287 (19)	133 (2)	−141 (15)
H(3)	628 (16)	187 (3)	−166 (16)
H(4)	659 (17)	239 (3)	128 (14)
H(5)	461 (18)	234 (3)	477 (17)
H(6)	155 (17)	178 (3)	489 (17)
H(7)	−336 (18)	116 (3)	354 (15)
H(8)	−177 (18)	144 (3)	472 (16)
H(9)	295 (18)	58 (2)	186 (15)
H(10)	472 (18)	32 (3)	233 (19)

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

S—O(1)	1·455 (2)	C(1)—C(2)	1·405 (4)
S—O(2)	1·469 (2)	C(1)—C(6)	1·406 (3)
S—O(3)	1·450 (2)	C(2)—C(3)	1·382 (4)
S—C(7)	1·795 (3)	C(3)—C(4)	1·394 (5)
N—C(7)	1·426 (4)	C(4)—C(5)	1·374 (5)
N—C(1)	1·400 (3)	C(5)—C(6)	1·393 (4)
O(1)—S—O(2)	112·6 (1)	N—C(1)—C(6)	123·0 (2)
O(1)—S—O(3)	113·6 (1)	C(2)—C(1)—C(6)	118·6 (2)
O(1)—S—C(7)	106·7 (1)	C(1)—C(2)—C(3)	120·7 (3)
O(2)—S—O(3)	111·4 (1)	C(2)—C(3)—C(4)	120·2 (3)
O(2)—S—C(7)	105·5 (1)	C(3)—C(4)—C(5)	119·8 (3)
O(3)—S—C(7)	106·4 (1)	C(4)—C(5)—C(6)	120·9 (3)
C(1)—N—C(7)	122·2 (2)	C(1)—C(6)—C(5)	119·9 (3)
N—C(1)—C(2)	118·4 (2)	S—C(7)—N	112·3 (2)

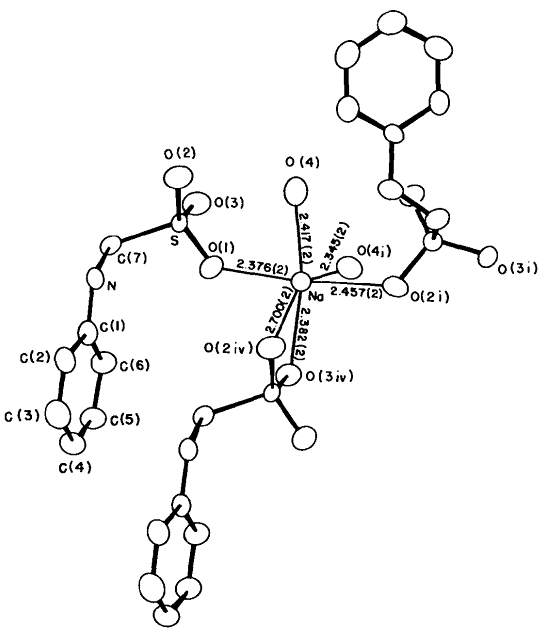

Fig. 1. Octahedral neighbourhood of the Na atom and atomic numbering in sodium anilinomethanesulphonate monohydrate.

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* Lists of structure factors, anisotropic thermal parameters and tables of bond lengths and angles have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 33894 (10 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

* See previous footnote.

being close to 90 or 180° and the other distances ranging from 2·345 (2) to 2·457 (2) Å. The octahedral neighbourhood of the Na atom is depicted in Fig. 1, where the numbering of the atoms of the structure and the Na—O distances are also given. The Na atoms distribute themselves around the $y = 0{\cdot}0$ and $y = 0{\cdot}5$ planes, their distances above and below those planes being 0·708 (1) Å. The shortest Na-atom contacts are 3·876 (1) Å between two atoms related by the twofold screw axis along the *c* direction who share the O(2) and O(4) atoms. The bond lengths and angles in the anilinomethanesulphonate residue are given in Table 2.

The hydrogen-bonding scheme is intermolecular and involves only the sulphonate residue O atoms as electro donors; the corresponding distances and angles are given in Table 3.

There is a close intramolecular approach of 2·925 (3) Å between N and O(1) but no hydrogen bond since the N—H bond points away from O(1). Water molecule O atoms show no close approach.*

All calculations were performed in this Institute on a PDP 11/45, with the Nonius SDP crystallographic programs.

Table 3. *Hydrogen-bonding distances* (Å) *and angles* (°) *with their standard deviations in parentheses*

i	*j*	*k*	D_{ij}	D_{jk}	D_{ik}	∠*ijk*
O(4)–	H(9)···	$O(1^{i})$	0·72 (5)	2·67 (6)	2·978 (3)	108 (5)
O(4)–	H(10)···	$O(2^{ii})$	0·82 (6)	2·17 (6)	2·968 (3)	163 (6)
N–	H(1)···	$O(3^{iii})$	0·79 (5)	2·30 (6)	3·079 (3)	172 (6)

Symmetry code

(i) $\frac{1}{2} - x, -y, \frac{1}{2} + z$
(ii) $-\frac{1}{2} - x, -y, \frac{1}{2} + z$
(iii) $x, y, -1 + z$
(iv) $1 + x, y, z$

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* See deposition footnote.

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Chlorhydrate d'Acébutolol [(Hydroxy-2 Isopropylamino-3 Propoxy)-2 Butyrylamino-5 Acétophénone Chlorhydraté]

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(*Reçu le* 27 *février* 1978, *accepté le* 5 *juillet* 1978)

Abstract. $C_{18}H_{29}N_2O_4^+.Cl^-$, monoclinic, *C*2/*c*, $a = 27{\cdot}552\,(3)$, $b = 4{\cdot}995\,(1)$, $c = 29{\cdot}122\,(4)$ Å, $\beta = 99{\cdot}55\,(1)°$, $Z = 8$. The structure was determined by the heavy-atom method and with the *MULTAN* program. The final *R* value is 0·029. The Cl^- anion is responsible for crystalline cohesion.

Introduction. Parmi l'arsenal des β-bloquants existant sur le marché pharmaceutique, l'acébutolol (commercialisé en France sous le nom de Sectral) occupe une place de choix: c'est en effet le seul médicament qui a une action spécifique *in vitro* et *in vivo* sur les récepteurs β_1 (cardiaques et intestinaux).

La détermination de la structure cristalline a été entreprise afin de comparer la conformation à celle des autres dérivés adrénolytiques β-bloquants de synthèse étudiés au laboratoire (Gadret, Goursolle, Leger & Colleter, 1975*a*,*b*,*c*,*d*, 1976; Gadret, Goursolle, Leger, Colleter & Carpy, 1976; Carpy, Colleter, Gadret, Goursolle & Leger, 1976; Leger, Gadret & Carpy, 1977; Gadret, Leger & Carpy, 1977; Gadret, Leger, Carpy & Berthod, 1978).

Le chlorhydrate d'acébutolol cristallise sous forme de prismes, par évaporation lente, à partir de solutions de Me_2SO. Les diagrammes de Bragg et de Weissenberg ont permis de déterminer la symétrie mono-

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