

Table 3. Bond distances (Å), strengths and orders (BO) for MoO<sub>6</sub> octahedra

(a) Mo—O distances			
Mo(1)—O(1)	2.416 (2)	Mo(2)—O(1)	2.282 (3)
—O(1')	1.972 (3)	—O(2)	2.274 (2)
—O(2)	1.867 (3)	—O(5)	1.982 (3)
—O(3)	1.756 (3)	—O(6)	1.947 (3)
—O(4)	2.145 (3)	—O(8)	1.694 (4)
—O(7)	1.704 (2)	—O(9)	1.702 (3)
Mo(3)—O(2)	2.266 (3)	Mo(4)—O(1)	2.191 (3)
—O(4)	2.068 (3)	—O(3)	2.402 (2)
—O(5)	1.887 (3)	—O(4)	1.927 (3)
—O(10)	1.699 (4)	—O(6)	1.913 (3)
O(11)	1.705 (3)	—O(12)	1.678 (3)
—O(14)	2.124 (3)	—O(13)	1.718 (3)
(b) Bond strengths and orders			
	<i>s</i>		<i>s</i>
Mo(1)—O(1)	0.203	Mo(2)—O(1)	0.315
—O(1')	0.756	—O(2)	0.321
—O(2)	1.049	—O(5)	0.733
—O(3)	1.516	—O(6)	0.816
—O(4)	0.456	—O(8)	1.880
—O(7)	1.815	—O(9)	1.828
BO	5.815		5.893
	<i>s</i>		<i>s</i>
Mo(3)—O(2)	0.328	Mo(4)—O(1)	0.402
—O(4)	0.568	—O(3)	0.231
—O(5)	0.984	—O(4)	0.868
—O(10)	1.847	—O(6)	0.907
—O(11)	1.809	—O(12)	1.991
—O(14)	1.484	—O(13)	1.728
BO	6.020		6.127

of the polyanions. Thus, the crystal is held together mainly by the three-dimensional hydrogen-bond system. On UV irradiation, the H atoms in appropriate positions form bonds with O atoms, converting Mo<sup>VI</sup>=O to Mo<sup>V</sup>—OH. This causes the colour change from colourless to reddish brown.

## References

- ADAMS, R. D., KLEMPERER, W. G. & LIU, R.-S. (1979). *J. Chem. Soc. Chem. Commun.* pp. 256–257.
- BART, J. C. J. & RAGAINI, V. (1979). *Proceedings of the Climax 3rd International Conference on the Chemistry and Uses of Molybdenum*, edited by H. F. BARRY & P. C. H. MITCHELL. Climax Molybdenum Co., Ann Arbor, MI, USA.
- CHEN, J., YOU, X. Z., HAN, S. Y. & SUI, Y. X. (1988). *Acta Chim. Sin.* No. 4, p. 30.
- International Tables for X-ray Crystallography* (1974). Vol. IV. Birmingham: Kynoch Press. (Present distributor Kluwer Academic Publishers, Dordrecht.)
- MACARRON, E. M. I., WHITNEY, J. F. & CHASE, D. B. (1984). *Inorg. Chem.*, **23**, 3275–3280.
- MAIN, P., FISKE, S. J., HULL, S. E., LESSINGER, L., GERMAIN, G., DECLERCQ, J.-P. & WOOLFSON, M. M. (1982). *MULTAN11/82. A System of Computer Programs for the Automatic Solution of Crystal Structures from X-ray Diffraction Data*. Univs. of York, England, and Louvain, Belgium.
- NORTH, A. C. T., PHILLIPS, D. C. & MATHEWS, F. S. (1968). *Acta Cryst.* **A24**, 351.

*Acta Cryst.* (1989). **C45**, 415–418

## Structure of the Orthorhombic Form of the Barium Salt of Lasalocid A

BY IL-HWAN SUH, KATSUYUKI AOKI\* AND HIROSHI YAMAZAKI

*The Institute of Physical and Chemical Research Wako-shi, Saitama 351-01, Japan*

(Received 20 June 1988; accepted 17 October 1988)

**Abstract.** Ba<sup>2+</sup>·(C<sub>34</sub>H<sub>53</sub>O<sub>8</sub>)<sub>2</sub><sup>2-</sup>·H<sub>2</sub>O, *M<sub>r</sub>* = 1334.94, orthorhombic, *P*2<sub>1</sub>2<sub>1</sub>2<sub>1</sub>, *a* = 23.192 (4), *b* = 18.775 (5), *c* = 16.893 (2) Å, *V* = 7356 (2) Å<sup>3</sup>, *Z* = 4, *D<sub>x</sub>* = 1.205 g cm<sup>-3</sup>, λ(Mo *K*α) = 0.71073 Å, μ = 5.62 cm<sup>-1</sup>, *F*(000) = 2832, *T* = 294 K, *R* = 0.047 for 4125 observed reflections. The salt, which was prepared from aqueous acetone, forms a 'head-to-tail' dimer having a pseudo twofold symmetry, with the polar oxygen groups directed towards the interior of the molecule to wrap the metal ion and the non-polar groups towards the exterior. The metal ion is ninefold coordinated by eight oxygen atoms of the two crystallographically independent ionophores and a water molecule, with the metal—O distances in the range 2.631 (5)–3.022 (5) Å.

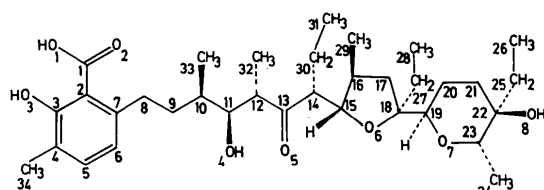
**Introduction.** Antibiotic lasalocid A (1) is a naturally occurring ionophore which has been most extensively studied by X-ray diffraction methods (Duesler & Paul, 1983). A feature of special interest in lasalocid A's structural chemistry is its propensity to form a variety of structures: monomers, 'head-to-tail' or 'head-to-head' dimers, or polymers. In order to reveal further the whole structural aspects of lasalocid A to explain the mechanisms of metal ion uptake and release by lasalocid A, we have tried to prepare variable metal complexes from solvents of different polarity, since the solvent conditions used for crystallization are important factors affecting lasalocid A structures (Duesler & Paul, 1983). We report here the structure of a barium salt crystallized in the orthorhombic form from aqueous acetone. The 'head-to-tail' dimeric structure with a pseudo twofold symmetry is essentially the same as that

\* To whom correspondence should be addressed.

of the monoclinic  $\text{Ba}^{2+}$  salt, which was prepared from aqueous ethanol (Johnson, Herrin, Liu & Paul, 1970).

Table 1. Fractional atomic coordinates ( $\times 10^5$  for Ba and  $\times 10^4$  for others) and equivalent isotropic temperature factors with e.s.d.'s in parentheses

$$B_{\text{eq}} = \frac{1}{3} \sum_i \beta_{ij} a_i \cdot a_j$$



(1)

**Experimental.** The complex was prepared by mixing lasalocid A sodium salt (61 mg, 0.1 mmol) dissolved in 10 mL of acetone and  $\text{BaCl}_2 \cdot 2\text{H}_2\text{O}$  (26 mg, 0.1 mmol) in 10 mL of water and allowing the solution to stand at room temperature. Colourless plates formed after a few days. Crystal  $0.36 \times 0.29 \times 0.28$  mm, Rigaku diffractometer, graphite-monochromated  $\text{Mo K}\alpha$  ( $\lambda = 0.71073 \text{ \AA}$ ) radiation; cell parameters by a least-squares fitting of diffractometer setting angles for 20 reflections ( $19 < 2\theta < 25^\circ$ ); reflection data  $2\theta_{\text{max}} = 45^\circ$  ( $h = 0$  to 24,  $k = 0$  to 29,  $l = 0$  to 18),  $\omega$ -scan for  $2\theta \leq 30^\circ$  and  $\omega - 2\theta$  scan for  $2\theta > 30^\circ$ , scan speed  $2^\circ \text{ min}^{-1}$  in  $2\theta$ ,  $\omega$ -scan width  $(1.6 + 0.5 \tan \theta)^\circ$ , background measured for 5 s on either side of the peak; intensity checks for three standard reflections showed little ( $-2.7 \rightarrow +1.1\%$ ) variation, 5416 independent reflections, 4125 observed with  $F_o > 3\sigma(F_o)$ ; Lp corrections, no absorption correction (variation in  $F_o$  of an axial reflection, 020, at  $\chi = 90^\circ$  with the spindle angle  $\phi$ , within 2.4% from the mean). Structure solved with *MULTAN78* (Main, Hull, Lessinger, Germain, Declercq & Woolfson, 1978) and refined with block-diagonal least squares, minimizing the function  $\sum w(F_o - |F_c|)^2$ . All 87 non-H atoms refined anisotropically, including the terminal C26 atom disordered at two positions with occupancy factors of 0.5 each for C26a and C26b, values estimated by the extent of their electron densities. All 107 H atoms attached to C atoms calculated from the known stereochemistry, and two H atoms attached to an OW and all five H atoms to O atoms except for O4', located from a difference Fourier map. H-atom positions not refined but included in the final cycles of the refinement with isotropic thermal parameters fixed at  $6 \text{ \AA}^2$ .  $R = 0.047$ ,  $wR = 0.026$ ,  $S = 1.86$  for 4125 observed reflections and 784 variables where  $w = \sigma(F_o)^{-2}$ ,  $(\Delta/\sigma)_{\text{max}} = 0.34$ , max. height in the final difference map  $0.41 \text{ e \AA}^{-3}$ . Final atomic coordinates with their e.s.d.'s are listed in Table 1.\* Atomic

	x	y	z	$B_{\text{eq}}(\text{\AA}^2)$
Ba	29229 (2)	23455 (3)	30485 (3)	3.9
OW	3553 (2)	3071 (3)	2033 (4)	5.7
O1	1841 (2)	4188 (3)	3096 (4)	5.9
O2	2748 (2)	3869 (3)	3126 (4)	5.4
O3	1536 (3)	5269 (3)	2370 (3)	6.0
O4	3479 (2)	3331 (3)	4217 (4)	5.2
O5	4113 (2)	2217 (3)	3271 (3)	5.1
O6	3327 (2)	1532 (3)	4484 (3)	4.3
O7	2165 (2)	1456 (3)	4029 (3)	4.2
O8	1948 (3)	2863 (3)	3735 (4)	6.9
C1	2368 (3)	4327 (4)	2997 (6)	5.0
C2	2530 (4)	5073 (5)	2691 (5)	4.5
C3	2069 (5)	5492 (4)	2381 (5)	5.4
C4	2213 (4)	6178 (4)	2064 (6)	5.7
C5	2771 (4)	6388 (4)	2089 (6)	6.2
C6	3221 (4)	5990 (5)	2368 (5)	6.1
C7	3108 (4)	5284 (5)	2675 (5)	5.8
C8	3625 (4)	4881 (4)	2971 (6)	5.4
C9	3681 (4)	4850 (4)	3867 (5)	5.1
C10	4146 (4)	4352 (5)	4173 (6)	4.9
C11	4029 (4)	3577 (5)	3931 (5)	4.2
C12	4477 (4)	3032 (5)	4221 (5)	4.6
C13	4308 (3)	2293 (5)	3933 (5)	4.0
C14	4371 (4)	1684 (5)	4480 (5)	4.9
C15	3814 (4)	1674 (4)	5001 (5)	4.4
C16	3783 (4)	1096 (5)	5628 (5)	5.8
C17	3144 (4)	1020 (5)	5739 (5)	5.2
C18	2912 (4)	1068 (4)	4889 (5)	4.4
C19	2310 (3)	1419 (4)	4844 (5)	4.3
C20	2284 (4)	2152 (4)	5222 (5)	5.1
C21	1697 (3)	2499 (5)	5047 (5)	6.0
C22	1522 (3)	2462 (5)	4175 (5)	6.2
C23	1594 (4)	1710 (5)	3865 (6)	5.3
C24	1149 (4)	1172 (5)	4150 (6)	7.1
C25	917 (4)	2759 (6)	4051 (6)	7.1
C26a	693 (8)	2786 (12)	3222 (15)	11.0
C26b	871 (9)	3525 (12)	4282 (16)	10.4
C27	2928 (4)	365 (4)	4452 (5)	5.2
C28	2496 (5)	-202 (5)	4731 (6)	7.4
C29	4110 (4)	1313 (6)	6403 (6)	7.5
C30	4502 (4)	990 (5)	4042 (6)	5.6
C31	5063 (4)	1023 (6)	3596 (6)	8.0
C32	5079 (4)	3189 (5)	3899 (6)	6.1
C33	4217 (4)	4452 (5)	5044 (6)	6.9
C34	1738 (4)	6620 (5)	1744 (6)	7.7
O1'	2367 (3)	954 (3)	2217 (4)	7.8
O2'	3272 (2)	1172 (3)	2168 (4)	6.0
O3'	1987 (3)	-210 (3)	2683 (3)	6.8
O4'	3886 (3)	1772 (3)	841 (4)	6.3
O5'	4787 (2)	2811 (3)	1703 (3)	5.6
O6'	3815 (2)	3618 (3)	527 (3)	5.4
O7'	2603 (2)	3694 (3)	1015 (3)	5.0
O8'	2353 (2)	2328 (3)	1582 (3)	6.9
C1'	2877 (4)	742 (4)	2215 (5)	5.5
C2'	2972 (4)	-57 (4)	2281 (4)	4.5
C3'	2493 (4)	-472 (5)	2548 (5)	5.6
C4'	2605 (4)	-1218 (5)	2704 (5)	6.2
C5'	3112 (4)	-1487 (5)	2545 (5)	7.2
C6'	3578 (4)	-1087 (5)	2270 (6)	7.4
C7'	3510 (4)	-339 (5)	2137 (6)	6.1
C8'	4037 (4)	57 (5)	1840 (6)	5.9
C9'	3993 (4)	230 (5)	967 (6)	5.4
C10'	4476 (4)	705 (5)	645 (6)	5.9
C11'	4434 (4)	1460 (5)	994 (5)	4.9
C12'	4891 (4)	1972 (5)	671 (5)	5.0
C13'	4816 (3)	2714 (5)	1001 (5)	4.4
C14'	4818 (4)	3315 (5)	406 (5)	5.1
C15'	4222 (4)	3326 (5)	-14 (5)	5.4
C16'	4176 (4)	3831 (6)	-735 (6)	6.8
C17'	3547 (4)	4001 (5)	-758 (6)	7.1
C18'	3339 (4)	3989 (5)	119 (5)	6.4
C19'	2792 (4)	3621 (5)	228 (5)	5.6
C20'	2783 (4)	2837 (5)	17 (5)	6.5
C21'	2197 (4)	2477 (5)	178 (5)	6.9
C22'	1982 (4)	2638 (5)	1001 (5)	6.0
C23'	2041 (4)	3440 (4)	1187 (5)	5.7
C24'	1578 (4)	3934 (5)	813 (6)	7.9
C25'	1362 (4)	2404 (5)	1164 (6)	8.2
C26'	1302 (6)	1632 (7)	1110 (8)	13.4
C27'	3353 (5)	4741 (5)	476 (6)	7.2

\*Lists of structure factors, anisotropic thermal parameters, H-atom positions, torsion angles, least-squares planes and close contacts have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 51506 (28 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Table 1 (cont.)

	x	y	z	$B_{eq}(\text{\AA}^2)$
C28'	2865 (5)	5239 (5)	206 (6)	8.7
C29'	4407 (5)	3490 (7)	-1467 (6)	9.9
C30'	4990 (4)	4006 (5)	796 (6)	6.9
C31'	5616 (5)	4024 (6)	1000 (7)	9.0
C32'	5500 (4)	1743 (5)	865 (7)	7.8
C33'	4446 (5)	701 (5)	-249 (6)	7.9
C34'	2107 (5)	-1639 (4)	3017 (6)	8.4

scattering factors from *International Tables for X-ray Crystallography* (1974). All calculations performed with the UNICS III program system (Sakurai & Kobayashi, 1979) on a FACOM 780 computer.

**Discussion.** Figs. 1 and 2 show the molecular structure and the crystal packing, respectively. Interatomic distances and angles are listed in Table 2.

The structure consists of the discrete dimer composed of one  $\text{Ba}^{2+}$  cation, two lasalocid A anions ('unprimed' and 'primed') deprotonated at the carboxyl group and a water molecule. The metal ion is nine-coordinated, six from one anion molecule ('unprimed'): carboxyl O2, hydroxyls O4 and O8, carbonyl O5 and ethers O6 and O7, two from another molecule ('primed'): carbonyl O2' and hydroxyl O8', and a water molecule, with the  $\text{Ba}^{2+}$ -O distances in the range of 2.631 (5)-3.022 (5) Å. Thus two antibiotic molecules are held together in a 'head-to-tail' fashion in which the 'head' phenyl ring of one anion is opposite the 'tail' ether ring of the other, by virtue of coordination to the metal ion. The water ligand helps to stabilize the dimer structure by forming hydrogen bonds with the less-complexed lasalocid anion ('primed'): H1(OH) to O5' and H2(OH) to both O6' and O7' (a bifurcated hydrogen bond). The conformation of the two ionophores is very similar despite their different environments. The molecule is folded into a pseudo-cyclic conformation stabilized by an intramolecular 'head-to-tail' hydrogen bond between the carboxylate O1 and the terminal hydroxyl O8 and further reinforced by an intramolecular hydrogen bond between the carboxylate O2 and the inner hydroxyl O4 and a 'salicylic acid-type' hydrogen bond between the hydroxyl O3 and the carboxylate O1. The polar oxygen groups are directed towards the interior of the molecule and the non-polar groups towards the exterior. The largest difference in torsion angles of the ionophore backbone between the two anions is only 14.5° for C15-O6-C18-C19. Both ionophores further include common structural features: the six-membered tetrahydropyran ring in a chair conformation, the five-membered furan ring in an envelope conformation with the C16 atom deviating from the plane of the remaining four atoms [torsion angle C15-O6-C18-C17 = 7.9 (8) and -7.7 (8)° for the 'unprimed' and the 'primed' anions respectively] and the carboxylate group nearly coplanar with the phenyl ring plane with the torsion angle

C3-C2-C1-O1 of -13 (1)° for the 'unprimed' anion and -16 (1)° for the 'primed' anion. A major difference in the two anions is found in the orientation of the C25-C26 ethyl substituent: in the 'unprimed' ionophore, the ethyl group is disordered into two positions in which the C25-C26a bond bisects the C23-C22-O8 angle ('type A' position designated by Duesler & Paul, 1983) with a C23-C22-C25-C26a angle of -58 (1)° while the C25-C26b bond (not shown in Figs. 1 and 2) bisects the C21-C22-O8 angle ('type B' position) with the C21-C22-C25-C26b angle 61 (1)°, while in the 'primed' molecule the C25'-C26' bond occupies only the 'type B' position with the C21'-C22'-C25'-C26' angle 64 (1)°.

The crystal packing is dominated by non-polar van der Waals contacts, with the closest contact of 3.46 (1) Å between C6' and C31' (at  $1-x, -\frac{1}{2}+y, \frac{1}{2}-z$ ).

The structure of the present  $\text{Ba}^{2+}$  salt is essentially identical to that of the monoclinic  $\text{Ba}^{2+}$  salt, including the water coordination mode and the hydrogen bond scheme. However, the arrangement of the corresponding C25-C26 ethyl group of the 'primed' anion differs, having the 'type B' position in the present salt

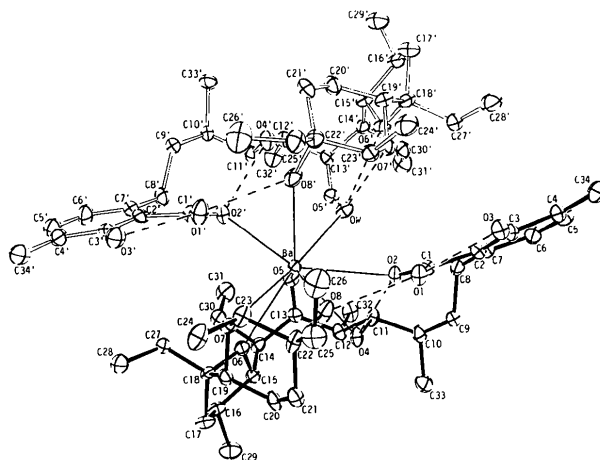


Fig. 1. A 'head-to-tail' dimeric structure of the  $\text{Ba}^{2+}$ (lasalocid A) $_2$ -(H $_2$ O) salt, which has a pseudo twofold symmetry. Broken lines denote hydrogen bonds.

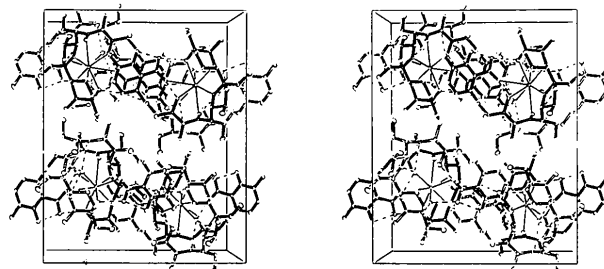


Fig. 2. A stereoscopic view showing the crystal packing, viewed down the  $c$  axis with the  $b$  axis horizontal and the  $a$  axis vertical. Broken lines denote hydrogen bonds.

Table 2. *Interatomic distances (Å) and angles (°) with e.s.d.'s in parentheses*

Coordination sphere			
Ba—O <sup>W</sup>	2.631 (5)	Ba—O7	2.928 (5)
Ba—O2	2.891 (5)	Ba—O8	2.719 (6)
Ba—O4	2.997 (6)	Ba—O2'	2.781 (5)
Ba—O5	2.795 (5)	Ba—O8'	2.811 (5)
Ba—O6	3.022 (5)		
O <sup>W</sup> —Ba—O2	66.5 (2)	O4—Ba—O8	82.0 (2)
O <sup>W</sup> —Ba—O4	82.7 (2)	O4—Ba—O2'	135.6 (2)
O <sup>W</sup> —Ba—O5	65.6 (2)	O4—Ba—O8'	142.5 (2)
O <sup>W</sup> —Ba—O6	127.9 (2)	O5—Ba—O6	62.6 (1)
O <sup>W</sup> —Ba—O7	173.5 (2)	O5—Ba—O7	117.6 (2)
O <sup>W</sup> —Ba—O8	123.7 (2)	O5—Ba—O8	142.6 (2)
O <sup>W</sup> —Ba—O2'	84.0 (2)	O5—Ba—O2'	73.4 (2)
O <sup>W</sup> —Ba—O8'	72.1 (2)	O5—Ba—O8'	125.6 (2)
O2—Ba—O4	54.5 (2)	O6—Ba—O7	56.3 (1)
O2—Ba—O5	102.7 (2)	O6—Ba—O8	95.6 (2)
O2—Ba—O6	120.1 (2)	O6—Ba—O2'	86.6 (2)
O2—Ba—O7	116.8 (2)	O6—Ba—O8'	148.1 (2)
O2—Ba—O8	60.6 (2)	O7—Ba—O8	57.7 (2)
O2—Ba—O2'	148.3 (2)	O7—Ba—O2'	91.5 (2)
O2—Ba—O8'	89.6 (2)	O7—Ba—O8'	102.0 (2)
O4—Ba—O5	62.5 (2)	O8—Ba—O2'	138.9 (2)
O4—Ba—O6	69.3 (1)	O8—Ba—O8'	89.4 (2)
O4—Ba—O7	103.8 (1)	O2'—Ba—O8'	69.7 (2)
Ba—O2—C1	140.9 (5)	Ba—O7—C19	115.8 (4)
Ba—O4—C11	111.0 (4)	Ba—O7—C23	105.0 (4)
Ba—O5—C13	118.9 (4)	Ba—O8—C22	126.8 (5)
Ba—O6—C15	129.7 (4)	Ba—O2'—C1'	105.6 (5)
Ba—O6—C18	118.2 (4)	Ba—O8'—C22'	151.1 (5)

## Lasalocid A anions

	'unprimed'	'primed'		'unprimed'	'primed'
C1—C2	1.53 (1)	1.51 (1)	C14—C30	1.54 (1)	1.52 (1)
C2—C3	1.41 (1)	1.42 (1)	C30—C31	1.51 (1)	1.50 (1)
C3—C4	1.43 (1)	1.44 (1)	C15—C16	1.50 (1)	1.53 (1)
C4—C5	1.34 (1)	1.32 (1)	C16—C17	1.51 (1)	1.49 (1)
C5—C6	1.38 (1)	1.40 (1)	C17—C18	1.52 (1)	1.53 (1)
C6—C7	1.44 (1)	1.42 (1)	C18—O6	1.468 (9)	1.47 (1)
C7—C2	1.41 (1)	1.40 (1)	O6—C15	1.43 (1)	1.42 (1)
C1—O1	1.265 (9)	1.24 (1)	C16—C29	1.58 (1)	1.52 (1)
C1—O2	1.243 (9)	1.23 (1)	C18—C19	1.55 (1)	1.49 (1)
C3—O3	1.33 (1)	1.30 (1)	C18—C27	1.51 (1)	1.53 (1)
C4—C34	1.49 (1)	1.48 (1)	C27—C28	1.53 (1)	1.55 (1)
C7—C8	1.51 (1)	1.52 (1)	C19—C20	1.51 (1)	1.50 (1)
C8—C9	1.51 (1)	1.50 (1)	C20—C21	1.55 (1)	1.53 (1)
C9—C10	1.52 (1)	1.54 (1)	C21—C22	1.52 (1)	1.53 (1)
C10—C11	1.54 (1)	1.54 (1)	C22—C23	1.51 (1)	1.54 (1)
C10—C33	1.49 (1)	1.49 (1)	C23—O7	1.44 (1)	1.41 (1)
C11—C12	1.56 (1)	1.54 (1)	O7—C19	1.430 (9)	1.42 (1)
C11—O4	1.43 (1)	1.43 (1)	C22—O8	1.45 (1)	1.42 (1)
C12—C13	1.52 (1)	1.51 (1)	C22—C25	1.52 (1)	1.54 (1)
C12—C32	1.53 (1)	1.51 (1)	C25—C26a	1.53 (3)	1.45 (2)
C13—C14	1.49 (1)	1.52 (1)	C25—C26b	1.51 (2)	—
C13—O5	1.206 (9)	1.198 (9)	C23—C24	1.52 (1)	1.57 (1)
C14—C15	1.57 (1)	1.55 (1)			
C1—C2—C3	117.7 (7)	117.4 (8)	C14—C30—C31	112.5 (7)	112.4 (8)
C2—C3—C4	118.3 (8)	118.3 (8)	C14—C15—C16	115.5 (7)	115.5 (7)
C3—C4—C5	118.3 (8)	118.2 (8)	C15—C16—C17	101.4 (7)	102.8 (7)
C4—C5—C6	125.3 (8)	124.4 (9)	C16—C17—C18	102.7 (7)	105.6 (7)
C5—C6—C7	118.5 (8)	119.6 (8)	C17—C18—O6	104.0 (6)	103.3 (7)
C6—C7—C2	116.9 (8)	116.8 (8)	C18—O6—C15	109.5 (6)	111.8 (6)
C7—C2—C1	119.5 (7)	119.8 (8)	O6—C15—C16	105.3 (6)	103.1 (7)
C7—C2—C3	122.6 (8)	122.6 (7)	O6—C15—C14	108.7 (6)	108.7 (7)

Table 2 (cont.)

	'unprimed'	'primed'		'unprimed'	'primed'
C2—C1—O1	117.0 (7)	117.0 (8)	C15—C16—C29	112.4 (7)	111.9 (8)
C2—C1—O2	122.1 (7)	123.2 (8)	C17—C16—C29	112.9 (7)	114.6 (8)
O1—C1—O2	120.9 (7)	119.8 (7)	C17—C18—C19	112.9 (6)	113.0 (7)
C2—C3—O3	122.1 (7)	123.0 (7)	O6—C18—C19	108.2 (6)	111.2 (7)
C4—C3—O3	119.6 (8)	118.7 (8)	C17—C18—C27	113.5 (6)	111.2 (8)
C3—C4—C34	118.2 (8)	117.9 (8)	O6—C18—C27	106.4 (6)	103.6 (7)
C5—C4—C34	123.5 (7)	123.9 (8)	C19—C18—C27	111.3 (7)	113.7 (8)
C2—C7—C8	127.4 (7)	126.2 (7)	C18—C27—C28	116.3 (7)	114.9 (8)
C6—C7—C8	115.7 (7)	116.9 (8)	C18—C19—C20	113.9 (6)	115.9 (8)
C7—C8—C9	114.5 (7)	112.2 (7)	C19—C20—C21	110.2 (7)	113.2 (7)
C8—C9—C10	114.9 (7)	115.1 (7)	C20—C21—C22	113.1 (7)	110.8 (7)
C9—C10—C11	111.3 (7)	110.3 (7)	C21—C22—C23	110.4 (7)	110.6 (7)
C9—C10—C33	109.8 (7)	109.1 (8)	C22—C23—O7	110.2 (6)	111.7 (7)
C11—C10—C33	113.7 (7)	113.6 (8)	C23—O7—C19	114.6 (6)	115.9 (6)
C10—C11—C12	114.7 (7)	113.0 (7)	O7—C19—C20	110.2 (6)	108.0 (6)
C10—C11—O4	112.2 (6)	111.4 (7)	O7—C19—C18	106.4 (6)	109.2 (7)
C12—C11—O4	105.7 (6)	106.9 (6)	C21—C22—O8	107.2 (6)	110.4 (7)
C11—C12—C13	108.6 (6)	111.0 (7)	C23—C22—O8	103.5 (6)	102.5 (6)
C11—C12—C32	111.8 (7)	112.6 (7)	C21—C22—C25	110.8 (7)	114.6 (7)
C13—C12—C32	107.3 (7)	106.9 (7)	C23—C22—C25	113.4 (7)	108.9 (7)
C12—C13—C14	117.6 (6)	115.1 (7)	O8—C22—C25	111.3 (7)	109.1 (7)
C12—C13—O5	120.5 (7)	121.5 (8)	C22—C25—C26a	118 (1)	111.5 (8)
C14—C13—O5	121.9 (8)	123.3 (8)	C22—C25—C26b	113 (1)	—
C13—C14—C15	106.3 (6)	108.5 (7)	C22—C23—C24	115.6 (7)	115.7 (7)
C13—C14—C30	111.2 (7)	110.5 (7)	O7—C23—C24	110.3 (7)	110.4 (7)
C15—C14—C30	114.7 (7)	113.9 (7)			
Hydrogen bonds					
O <sup>W</sup> ...O5'	2.957 (7)	O8...O1	2.724 (7)		
O <sup>W</sup> ...O6'	2.815 (8)	O3'...O1'	2.487 (8)		
O <sup>W</sup> ...O7'	3.022 (8)	O4'...O2'	2.879 (8)		
O3'...O1	2.479 (8)	O8'...O1'	2.782 (8)		
O4'...O2	2.696 (8)				

and the 'type A' position in the monoclinic salt. Another minor difference is the larger twisting of the carboxylate group from the phenyl plane (24–25°) in the monoclinic salt.

## References

- DUESLER, E. N. & PAUL, I. C. (1983). *Polyether Antibiotics: Naturally Occurring Acid Ionophores*, edited by J. M. WESTLEY, Vol. II, pp. 138–164. New York and Basel: Marcel Dekker.
- International Tables for X-ray Crystallography* (1974). Vol. IV. Birmingham: Kynoch Press. (Present distributor Kluwer Academic Publishers, Dordrecht.)
- JOHNSON, S. M., HERRIN, J., LIU, S. J. & PAUL, I. C. (1970). *J. Am. Chem. Soc.* **92**, 4428–4435.
- MAIN, P., HULL, S. E., LESSINGER, L., GERMAIN, G., DECLERCQ, J.-P. & WOOLFSON, M. M. (1978). *MULTAN78. A System of Computer Programs for the Automatic Solution of Crystal Structures from X-ray Diffraction Data*. Univ. of York, England, and Louvain, Belgium.
- SAKURAI, T. & KOBAYASHI, K. (1979). *Rikagaku Kenkyusho Hokoku* **55**, 69–77.