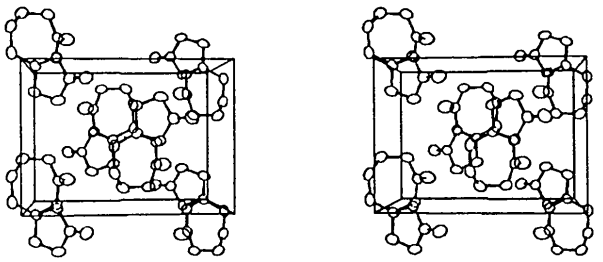

Fig. 2. Stereoscopic view of the molecular packing. The *a* axis points from left to right, the *b* axis upwards, and the *c* axis onto the plane of the paper; the H atoms are omitted.

H(11)···O(2)–C(10) 116·4 (5)°]. The sheets are stacked along **a** by van der Waals interactions with normal interatomic contacts.

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References

ASHIDA, T. (1973). *HBLS-V and DAPH. The Universal Crystallographic Computing System – Osaka.* The Computation Center, Osaka Univ., Japan.

BOCIAN, D. F. & STRAUSS, H. L. (1977). *J. Am. Chem. Soc.* **99**, 2876–2882.

FUJII, S. (1979). *MOLCON. The Universal Crystallographic Computing System – Osaka.* The Computation Center, Osaka Univ., Japan.

International Tables for X-ray Crystallography (1974). Vol. IV. Birmingham: Kynoch Press. (Present distributor D. Reidel, Dordrecht.)

JOHNSON, C. K. (1971). *ORTEP.* Report ORNL-3794, revised. Oak Ridge National Laboratory, Tennessee, USA.

KIMURA, M. & MOROSAWA, S. (1981). *J. Am. Chem. Soc.* **103**, 2433–2434.

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.* Univs. of York, England, and Louvain, Belgium.

OREN, J. & FUCHS, B. (1986). *J. Am. Chem. Soc.* **108**, 4881–4887.

ZIZUASHVILI, J., ABRAMSON, S., SHMUELI, U. & FUCHS, B. (1982). *J. Chem. Soc. Chem. Commun.* pp. 1375–1378.

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The Structure and Absolute Configuration of Acetomycin

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Abstract. $C_{10}H_{14}O_5$, $M_r = 214{\cdot}22$, orthorhombic, $P2_12_12_1$, $a = 14{\cdot}1084\ (6)$, $b = 10{\cdot}6443\ (3)$, $c = 7{\cdot}1970\ (1)$ Å, $V = 1080{\cdot}80\ (6)$ Å^3, $Z = 4$, $D_x = 1{\cdot}317$ Mg m^{-3}, Cu $K\alpha$, $\lambda = 1{\cdot}5418$ Å, $\mu = 0{\cdot}8571$ mm^{-1}, $F(000) = 456$, $T = 293$ K, $R = 0{\cdot}052$ for 816 observed [$3\sigma(I)$] Friedel pairs. The determined absolute configuration may be described as 3*S*,4*R*,5*R*, the five-membered ring having an envelope conformation, with the bulky substituents at *cis* positions. The bond lengths and angles are in agreement with those of the bromoacetoxy derivative.

Introduction. Acetomycin, (3*S*,4*S*,5*R*)-5-acetoxy-3-acetyl-3,4-dimethyldihydro-2(3*H*)-furanone, is an interesting antibiotic, both for its unique structure and for its biological properties. The correct molecular structure was established by Keller-Schierlein, Prelog and co-workers in a series of classical papers (Ettinger *et al.*, 1958; Keller-Schierlein, Mihailovic & Prelog, 1958; Bosshard, Goeckner & Keller-Schierlein, 1959; Bachmann, Gerlach, Prelog & Zahner, 1963). Even though it is not a first-line antibiotic, its broad activity, on bacteria, fungi (Ettinger *et al.*, 1958), mycobacteria (Uhr, Zeeck, Clegg, Egert, Fuher & Peter, 1985) and protozoa (Carrasco, 1984), makes it worthy of further study. The structural assessment and the relative and absolute configuration at the three chiral centres led us to undertake this study. Having finished our work, we received a preprint, from Professor Zeeck, with the absolute configuration of a bromoacetoxy derivative (Uhr *et al.*, 1985), both studies supporting the other's results.

Experimental. Samples were kindly supplied by Professors Prelog and Keller-Schierlein to whom we are indebted.* Cell constants obtained from a least-squares fit using 83 reflexions up to $\theta = 45°$ and Cu $K\alpha$

* The sample shows an optical rotation of $[\alpha]_D^{20\cdot C} = -157°$ (ethanol) as already described (Uhr *et al.*, 1985).

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radiation. A colourless polyhedron of dimensions ranging from 0·30 to 0·35 mm was used for the analysis on a Philips PW 1100 diffractometer, with Cu $K\alpha$ radiation, graphite monochromator, $\omega/2\theta$ scans, bisecting geometry, 1 min per reflexion, up to 65° in θ. Good stability for the sample checked every 90 min. After each measurement the Friedel pair was collected. 2234 data gave 1090 independent [h: 0–16; k: 0–12; l: 0–8]. No absorption correction was applied ($\mu r \sim 0{\cdot}3$).

The structure was solved by direct methods (Main *et al.*, 1980) and refined by one-block-matrix least-squares procedures on $|F_{obs}|$ (Stewart, Machin, Dickinson, Ammon, Heck & Flack, 1976). All the H atoms were located on a difference synthesis. An empirical weighting scheme, so as to give no trends in $\langle w\Delta^2F\rangle$ *vs* $\langle |F_o| \rangle$ and $\langle(\sin\theta)/\lambda\rangle$, was introduced. The final shift/e.s.d. was 0·03 with maximum peak in the final ΔF of 0·21 e Å^{-3}. The final R and wR values were 0·0519 and 0·0547 respectively with $S = 1{\cdot}164$. The enantiomeric structure gave R and wR values of 0·0520 and 0·0549 and so was rejected (Hamilton, 1965). η refinements (Rogers, 1981) gave, with $wR = 0{\cdot}0547$, an η of 0·937 (18) and confirm the proposed chirality, which was again confirmed by comparing Friedel pairs (Martinez-Ripoll & Fayos, 1980) and by the mentioned preprint (Uhr *et al.*, 1985).* All the calculations were performed on a VAX 11/750 computer. The atomic scattering factors were taken from *International Tables for X-ray Crystallography* (1974). Table 1 shows the final non-H atomic coordinates corresponding to Fig. 1 (Johnson, 1965).

Table 1. *Final atomic coordinates and thermal parameters* (Å^2 × 10^3)

$U_{eq} = \frac{1}{3}\sum[U_{ij}a_i^*a_j^*a_ia_j\cos(\mathbf{a}_i,\mathbf{a}_j)]$.

	x	y	z	U_{eq}
O1	0·2527 (2)	−0·0823 (2)	−0·3515 (4)	55 (1)
O2	0·1956 (2)	0·1101 (2)	−0·3067 (5)	76 (1)
O3	−0·0634 (2)	−0·0847 (4)	−0·2952 (4)	94 (1)
O4	0·2014 (1)	−0·2756 (2)	−0·2496 (4)	48 (1)
O5	0·3533 (2)	−0·3319 (3)	−0·2366 (5)	74 (1)
C1	0·1811 (3)	0·0033 (4)	−0·3532 (6)	51 (1)
C2	0·0891 (2)	−0·0533 (3)	−0·4225 (5)	39 (1)
C3	0·1256 (2)	−0·1773 (3)	−0·5102 (5)	42 (1)
C4	0·2187 (2)	−0·2023 (3)	−0·4113 (5)	46 (1)
C5	0·0194 (2)	−0·0763 (4)	−0·2585 (6)	50 (1)
C6	0·0535 (4)	−0·0880 (6)	−0·0642 (6)	61 (2)
C7	0·0418 (3)	0·0332 (4)	−0·5611 (7)	55 (1)
C8	0·0566 (3)	−0·2861 (4)	−0·5186 (8)	63 (2)
C9	0·2757 (2)	−0·3369 (3)	−0·1701 (6)	49 (1)
C10	0·2474 (4)	−0·4020 (5)	0·0004 (8)	66 (2)

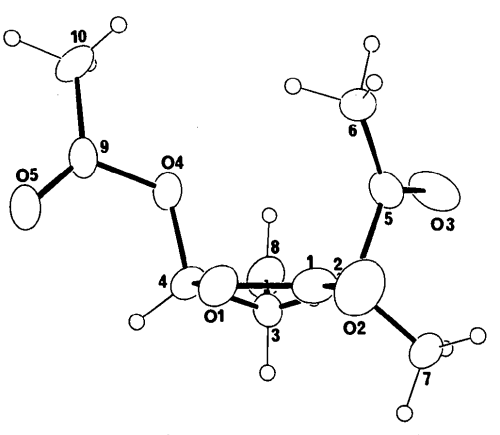

Fig. 1. An *ORTEP* view of the molecule showing the atomic numbering used in the present crystallographic work.

Table 2. *Selected geometrical parameters* (Å, °) *and main steric interactions* (H···O < 2·7 Å)

O1–C1	1·361 (5)	C2–C7	1·513 (6)
O1–C4	1·430 (4)	C3–C4	1·518 (5)
C1–O2	1·202 (5)	C3–C8	1·514 (6)
C1–C2	1·516 (5)	C4–O4	1·422 (5)
C2–C3	1·551 (5)	O4–C9	1·361 (4)
C2–C5	1·555 (5)	C9–O5	1·196 (4)
C5–O3	1·200 (4)	C9–C10	1·465 (7)
C5–C6	1·484 (6)		
C1–O1–C4	110·2 (3)	O1–C1–O2	120·3 (3)
O1–C1–C2	111·9 (3)	O2–C1–C2	127·9 (4)
C1–C2–C3	100·8 (3)	O1–C4–C3	106·0 (3)
C2–C3–C4	104·2 (3)	O4–C9–C10	111·7 (3)
O5–C9–C10	127·3 (4)		
O1–C1–C2–C3	−14·4 (4)	C1–C2–C3–C4	23·7 (3)
C2–C3–C4–O1	−25·9 (4)	C3–C4–O1–C1	17·7 (4)
C4–O1–C1–C2	−1·6 (4)	O4–C4–C3–C8	−40·5 (4)
C8–C3–C2–C5	35·3 (4)	C1–C2–C5–C6	−21·9 (5)
C1–C2–C5–O3	158·4 (4)	C8–C3–C4–O4	−40·5 (5)
C1–O1–C4–O4	−99·3 (3)	O1–C4–C3–C8	−156·2 (3)
C4–C3–C2–C5	−156·2 (3)	H8*A*–C8–C3–C4	53 (3)
H8*A*–C8–C3–H3	171 (4)	C2–C3–C4–H4	−145 (3)
H3···O1^{i}	2·61 (4)	H4···O1^{i}	2·05 (5)
H4···O4^{i}	2·08 (5)	H4···O5^{i}	2·42 (5)
H6*A*···O4^{i}	2·50 (5)	H6*C*···O3^{i}	2·45 (5)
H7*C*···O3^{i}	2·48 (4)	H8*A*···O4^{i}	2·47 (4)
H10*A*···O4^{i}	2·56 (9)	H10*B*···O4^{i}	2·62 (7)
H10*C*···O5^{i}	2·53 (7)	H3···O2^{ii}	2·68 (4)
H4···O2^{ii}	2·67 (5)	H7*B*···O1^{ii}	2·65 (5)
H6*C*···O5^{iii}	2·67 (5)	H7*C*···O5^{iv}	2·69 (4)
H8*B*···O5^{iv}	2·56 (4)	H10*C*···O3^{v}	2·56 (7)

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

Discussion. Table 2 presents the main geometrical parameters describing the acetomycin molecule as it appears in Fig. 1. Here, as in the bromoacetoxy derivative (Uhr *et al.*, 1985), the molecule presents an enlargement of the O2–C1–C2 and O5–C9–C10 angles and both have values of O1–C1–C2 and O4–C9–C10 which are quite similar. The five-membered ring adopts an envelope conformation, flap at C3, with all the substituents, apart from the

* Lists of anisotropic thermal parameters, observed and calculated structure amplitudes, with their Friedel pairs, H-atom parameters, all bond lengths and angles and a list of the 12 most significant reflexions used in the chirality determination have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 44742 (18 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

C7 methyl group, on one side. While the acetoxy substitution at C4 presents the same conformation as in the bromoacetoxy derivative (C9 *trans* to C3 and C10 *trans* to C4), the main differences between them are due to the substitution at O3 and are reflected by the torsion angles C1—C2—C5—C6 and C1—C2—C5—O3, −21·9 (5) and 158·4 (4)° *vs* 56·1 (3) and 175·1 (3)° in the derivative. The above conformation gives rise to some steric interactions in the packing as well as within the molecule (Table 2) (Vainshtein, Fridkin & Indenbom, 1982).

References

BACHMANN, E., GERLACH, H., PRELOG, V. & ZAHNER, H. (1963). *Helv. Chim. Acta*, **46**, 605–611.

BOSSHARD, E., GOECKNER, N. A. & KELLER-SCHIERLEIN, W. (1959). *Helv. Chim. Acta*, **42**, 2746–2750.

CARRASCO, L. (1984). Unpublished results. (Centro de Biologia Molecular, Univ. Autonoma de Madrid, Spain.)

ETTINGER, L., GAUMANN, E., HUTTER, R., KELLER-SCHIERLEIN, W., KRADOLFER, F., NEIPP, L., PRELOG, V. & ZAHNER, H. (1958). *Helv. Chim. Acta*, **41**, 216–219.

HAMILTON, W. (1965). *Acta Cryst.* **18**, 502–510.

International Tables for X-ray Crystallography (1974). Vol. IV. Birmingham: Kynoch Press. (Present distributor D. Reidel, Dordrecht.)

JOHNSON, C. K. (1965). *ORTEP*. Report ORNL-3794. Oak Ridge National Laboratory, Tennessee, USA.

KELLER-SCHIERLEIN, W., MIHAILOVIC, M. L. & PRELOG, V. (1958). *Helv. Chim. Acta*, **41**, 220–228.

MAIN, P., FISKE, S. J., HULL, S. E., LESSINGER, L., GERMAIN, G., DECLERCQ, J.-P. & WOOLFSON, M. M. (1980). *MULTAN80. A System of Computer Programs for the Automatic Solution of Crystal Structures from X-ray Diffraction Data*. Univs. of York, England, and Louvain, Belgium.

MARTINEZ-RIPOLL, M. & FAYOS, J. (1980). *Z. Kristallogr.* **152**, 189–194.

ROGERS, D. (1981). *Acta Cryst.* A**37**, 734–741.

STEWART, J. M., MACHIN, P. A., DICKINSON, C. W., AMMON, H. L., HECK, H. & FLACK, H. (1976). The *XRAY76* system. Tech. Rep. TR-446. Computer Science Center, Univ. of Maryland, College Park, Maryland, USA.

UHR, H., ZEECK, A., CLEGG, W., EGERT, E., FUHER, H. & PETER, H. H. (1985). *J. Antibiot.* **38**, 1684–1690.

VAINSHTEIN, B. K., FRIDKIN, V. M. & INDENBOM, V. L. (1982). *Modern Crystallography* II, p. 87. Berlin, Heidelberg, New York: Springer-Verlag.

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meso-α,α′-Diaminosuccinic Acid: An Example of Strong Intermolecular Hydrogen Bonding

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Abstract. $C_4H_8N_2O_4$, $M_r = 148{\cdot}12$, monoclinic, $P2_1/n$, $a = 7{\cdot}439$ (1), $b = 4{\cdot}758$ (1), $c = 8{\cdot}301$ (1) Å, $\beta = 102{\cdot}21$ (1)°, $V = 287{\cdot}2$ (1) Å^3, $Z = 2$, $D_x = 1{\cdot}713$ g cm^{-3}, λ(Cu $K\alpha$) = 1·54184 Å, $\mu = 12{\cdot}9$ cm^{-1}, $F(000) = 156$, $T = 295$ K. The structure has been refined to $R = 0{\cdot}034$ for 516 unique reflections with $I \geq 2{\cdot}5\sigma(I)$. The asymmetric unit of the double zwitterionic title compound consists of one half molecule. The NH_3^+ protons are involved in N—H···O intermolecular hydrogen bonds. Each molecule is connected to eight neighbouring molecules by 12 hydrogen bonds. Two unique hydrogen bonds connect molecules which lie in sheets parallel to **b** and one hydrogen bond serves as a link between these sheets.

Introduction. The lack of structural information on the class of α,α′-diaminodicarboxylic acids and our general interest in hydrogen bonding of α-substituted carboxylic acids led us to the structure analysis of the first member of this class, *meso*-α,α′-diaminosuccinic acid. For a long time this compound attracted our attention because of its high density (1·713 g cm^{-3}) and its low solubility in water and in a wide variety of organic solvents, which are indicative of strong intermolecular hydrogen bonding of this supposedly double zwitterionic compound. However, numerous attempts to obtain single crystals of sufficient size and quality applying conventional methods were unsuccessful over a period of years. By using an alternative crystallization procedure crystals of excellent quality were obtained and we now report the crystal-structure analysis.

Experimental. Because of the insolubility of the title compound no standard crystallization technique was useful. Therefore lithium and hydrogen chloride salts were prepared and crystallization attempts were made in acidified gels (Li salt) and in basic gels (HCl salt). This procedure only yielded twinned crystals in different silica and agarose gels. Finally single crystals

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