Data collection

Stoe IPDS diffractometer
Image plate scans
Absorption correction: integration (*XPREP*; Siemens, 1996*b*)
$T_{min} = 0.511$, $T_{max} = 0.906$
12 133 measured reflections
3116 independent reflections
2163 reflections with $I > 2\sigma(I)$
$R_{int} = 0.051$
$\theta_{max} = 28°$
$h = -18 \rightarrow 19$
$k = -9 \rightarrow 9$
$l = -16 \rightarrow 16$
Intensity decay: none

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.029$
$wR(F^2) = 0.059$
$S = 0.903$
3116 reflections
188 parameters
H atoms: see below
$w = 1/[\sigma^2(F_o^2) + (0.0271P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.393$ e Å^{-3}
$\Delta\rho_{min} = -0.390$ e Å^{-3}
Extinction correction: none
Scattering factors from *International Tables for Crystallography* (Vol. C)

Table 1. *Selected geometric parameters (Å, °)*

Cu—O2	1.9346 (15)	Cu—N1	1.9905 (17)
Cu—O1	1.9544 (16)	Cu—O5	2.3103 (16)
Cu—O4^i	1.9816 (14)	Cu—O6	3.0621 (18)
O2—Cu—O1	171.99 (7)	O4^i—Cu—O5	104.82 (6)
O2—Cu—O4^i	85.07 (6)	N1—Cu—O5	93.61 (6)
O1—Cu—O4^i	96.26 (6)	O2—Cu—O6	93.64 (6)
O2—Cu—N1	93.66 (7)	O1—Cu—O6	78.72 (6)
O1—Cu—N1	82.57 (7)	O4^i—Cu—O6	83.17 (5)
O4^i—Cu—N1	161.52 (7)	N1—Cu—O6	78.51 (6)
O2—Cu—O5	90.43 (6)	O5—Cu—O6	171.33 (5)
O1—Cu—O5	96.83 (6)		

Symmetry code: (i) $1 - x, y - \frac{1}{2}, \frac{1}{2} - z$.

Table 2. *Hydrogen-bonding geometry (Å, °)*

D—H$\cdots A$	D—H	H$\cdots A$	$D\cdots A$	D—H$\cdots A$
N2—H21$\cdots$O5^i	0.88 (2)	2.16 (2)	3.038 (3)	175 (3)
N2—H22$\cdots$O1^{ii}	0.89 (2)	2.05 (2)	2.940 (3)	174 (3)
N3—H31$\cdots$O3^{ii}	0.89 (1)	2.09 (2)	2.971 (3)	171 (2)
O5—H51$\cdots$O4^{iii}	0.89 (1)	2.22 (2)	3.094 (2)	167 (2)
O5—H51$\cdots$O2^{iv}	0.89 (1)	2.43 (2)	2.790 (2)	105 (2)
O5—H52$\cdots$O6^v	0.89 (1)	1.86 (1)	2.739 (2)	166 (2)

Symmetry codes: (i) $1 - x, \frac{1}{2} + y, \frac{1}{2} - z$; (ii) $x, 1 + y, z$; (iii) $x, y - 1, z$; (iv) $1 - x, y - \frac{1}{2}, \frac{1}{2} - z$; (v) $x, \frac{3}{2} - y, \frac{1}{2} + z$.

All H atoms except the hydrogen-bond donors were included at calculated positions using *SHELXL*97 (Sheldrick, 1997) and refined using a riding model. The U_{iso} values for the H atoms on CH_2 or CH_3 were taken as $1.2U_{eq}$ and $1.5U_{eq}$ of the carrier atoms, respectively. The H atoms of C3 were refined as a rigid group. Atoms H21, H22, H31, H51 and H52 were found from difference Fourier syntheses and refined with $U_{iso} = 1.2U_{eq}$ of the carrier atoms; O—H and N—H distances were restrained to be equal with an s.u. of 0.01 Å. Analytical absorption correction based on face indexing was carried out with the following faces and distances (mm): (010) 0.2503, (0$\bar{1}$0) 0.2118, (001) 0.0192, (00$\bar{1}$) 0.0385, (20$\bar{1}$) 0.0008 and ($\bar{2}$01) 0.1155. The data completeness to $\theta = 28°$ amounts to 98.4%.

Data collection: *EXPOSE* (Stoe, 1997*b*). Cell refinement: *CELL* (Stoe, 1997*a*). Data reduction: *INTEGRATE* (Stoe, 1997*c*). Program(s) used to solve structure: *SHELXS*97 (Sheldrick, 1990). Program(s) used to refine structure: *SHELXL*97. Molecular graphics: *XP* (Siemens, 1996*a*). Software used to prepare material for publication: *SHELXL*97.

I thank the Head of the Section for X-ray Diffraction, Professor Dr W. Massa, for making diffractometer time available, and Madame G. Geiseler for performing the measurements.

Supplementary data for this paper are available from the IUCr electronic archives (Reference: JZ1313). Services for accessing these data are described at the back of the journal.

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Acta Cryst. (1999). C**55**, 54–56

Micacocidin A

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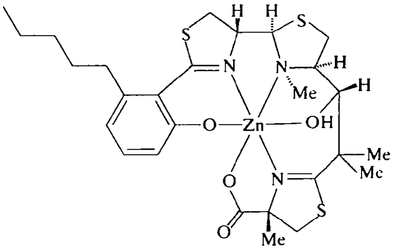
Abstract

In the title compound, (4,5-dihydro-2-{2-hydroxy-1,1-dimethyl-2-[2,3,4',5'-tetrahydro-3-methyl-2'-(2-oxido-6-pentylphenyl)-2,4'-bithiazolyl-4-yl-*N*,*N'*,*O*]ethyl-*O*}-4-methylthiazole-4-carboxylato-*N*,*O*)zinc(II), $[Zn(C_{27}H_{37}N_3O_4S_3)]$, the Zn atom is octahedrally coordinated by

three O atoms and three N atoms. There are two crystallographically independent but chemically equivalent molecules, *A* and *B*, in the asymmetric unit. They form a dimeric structure through two intermolecular O—H···O hydrogen bonds.

Comment

Micacocidin A has been isolated from the culture filtrate of *Pseudomonas* sp. No. 57-250. It has specific and excellent antibiotic activity against *Mycoplasma* species (Kobayashi *et al.*, 1998). The present study of the title compound, (I), was undertaken to elucidate the structure and absolute configuration. As a result of the crystal structure analysis, it was found that micacocidin A is a zinc complex with a molecule of $C_{27}H_{37}N_3O_4S_3$, as shown in Fig. 1.



(I)

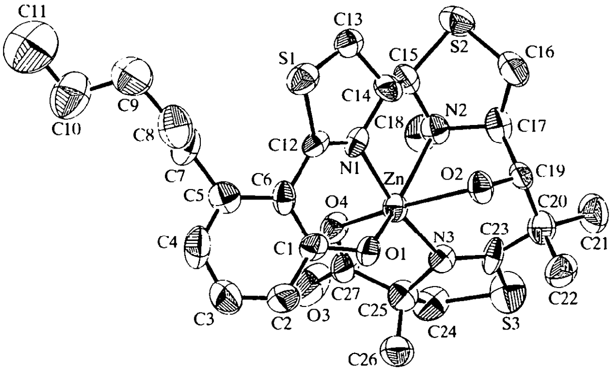


Fig. 1. An *ORTEP*II (Johnson, 1976) plot of molecule *A* showing 50% probability displacement ellipsoids. H atoms have been omitted for clarity.

The Zn atom is octahedrally coordinated by the O atoms of the phenolate and carboxylate anions and the aliphatic hydroxyl groups, and by the three N atoms. The two crystallographically independent molecules *A* and *B* form a dimeric structure through two intermolecular hydrogen bonds; O1*A*···H2—O2*B* and O1*B*···H1—O2*A* (Fig. 2). The geometries of the hydrogen bonds are listed in Table 2. The molecules *A* and *B* in the dimer are related by a pseudo-twofold rotation axis, which is perpendicular to the plane consisting of the four O atoms forming the intermolecular hydrogen bonds (O1*A*, O2*A*, O1*B* and O2*B*). The geometries of *A* and *B* are almost the same. The pentyl groups of *A* and *B* show considerable thermal vibrations and the bond distances between these atoms are therefore different from the standard ones. The bond distances C6*A*—C12*A* [1.49 (1) Å] and C6*B*—C12*B* [1.50 (1) Å] are extended with respect to the standard Csp^2—Csp^2 bond distance as a result of twisting between the Csp^2 planes [torsion angles N1*A*—C12*A*—C6*A*—C1*A* = 27 (1) and N1*B*—C12*B*—C6*B*—C1*B* = 29 (1)°]. The compound has the absolute configuration C14 *R*, C15 *R*, C17 *R*, C19 *S*, C25 *S*, and N2 *R*.

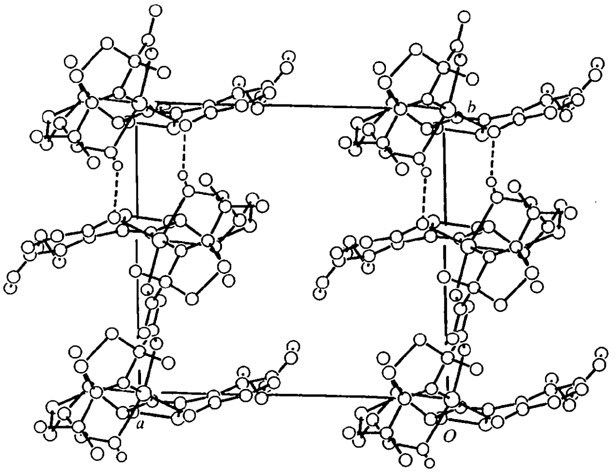


Fig. 2. A packing diagram viewed along the *c* axis (Lauher, 1989). H atoms have been omitted except for those of the OH groups. Intermolecular hydrogen bonds are represented by dashed lines. The upper right molecule is molecule *A* and the centre right one is molecule *B*.

Experimental

Crystals were grown from a methanol/ethyl acetate solution of the title compound.

Crystal data

$[Zn(C_{27}H_{37}N_3O_4S_3)]$
$M_r = 629.17$
Triclinic
$P1$
$a = 12.146 (3)$ Å
$b = 12.682 (6)$ Å
$c = 10.842 (8)$ Å
$\alpha = 115.26 (4)°$
$\beta = 93.17 (4)°$
$\gamma = 86.49 (3)°$
$V = 1506 (1)$ Å^3
$Z = 2$
$D_x = 1.387$ Mg m^{-3}
D_m not measured

Mo $K\alpha$ radiation
$\lambda = 0.7107$ Å
Cell parameters from 25 reflections
$\theta = 20.2–23.2°$
$\mu = 1.059$ mm^{-1}
$T = 295$ K
Rhombic plate
0.40 × 0.15 × 0.05 mm
Pale yellow

Data collection

Rigaku AFC-5*R* diffractometer
ω–2θ scans
Absorption correction: refined from ΔF (Walker & Stuart, 1983)
$T_{\min} = 0.817$, $T_{\max} = 0.948$
11 132 measured reflections
11 132 independent reflections
3266 reflections with $I > 2\sigma(I)$
$\theta_{\max} = 25.1°$
$h = -14 \rightarrow 14$
$k = -15 \rightarrow 15$
$l = -12 \rightarrow 12$
3 standard reflections every 150 reflections intensity decay: 1.57%

Refinement

Refinement on *F*
$R = 0.055$
$wR = 0.065$
$S = 1.000$
6967 reflections
683 parameters
H-atom parameters not refined
$w = 1/[\sigma^2(F_o) + 0.00164|F_o|^2]$
$(\Delta/\sigma)_{\max} = 0.005$
$\Delta\rho_{\max} = 0.46$ e Å^{-3}
$\Delta\rho_{\min} = -0.63$ e Å^{-3}
Extinction correction: none
Scattering factors from *International Tables for Crystallography* (Vol. C)
Absolute structure: Flack (1983)
Flack parameter = 0.01 (2)

Table 1. *Selected geometric parameters (Å, °)*

Zn*A*—O1*A*	2.020 (6)	Zn*B*—O1*B*	2.030 (6)
Zn*A*—O2*A*	2.245 (6)	Zn*B*—O2*B*	2.232 (6)
Zn*A*—O4*A*	2.015 (6)	Zn*B*—O4*B*	2.036 (6)
Zn*A*—N1*A*	2.036 (7)	Zn*B*—N1*B*	2.027 (7)
Zn*A*—N2*A*	2.389 (7)	Zn*B*—N2*B*	2.380 (7)
Zn*A*—N3*A*	2.070 (7)	Zn*B*—N3*B*	2.067 (7)
O1*A*—Zn*A*—O2*A*	93.7 (2)	O1*B*—Zn*B*—O2*B*	93.1 (2)
O1*A*—Zn*A*—O4*A*	101.2 (3)	O1*B*—Zn*B*—O4*B*	101.5 (3)
O1*A*—Zn*A*—N1*A*	84.5 (3)	O1*B*—Zn*B*—N1*B*	84.9 (3)
O1*A*—Zn*A*—N2*A*	155.8 (2)	O1*B*—Zn*B*—N2*B*	155.9 (2)
O1*A*—Zn*A*—N3*A*	109.1 (2)	O1*B*—Zn*B*—N3*B*	109.1 (2)
O2*A*—Zn*A*—O4*A*	160.9 (3)	O2*B*—Zn*B*—O4*B*	161.1 (3)
O2*A*—Zn*A*—N1*A*	92.2 (3)	O2*B*—Zn*B*—N1*B*	92.4 (3)
O2*A*—Zn*A*—N2*A*	71.1 (2)	O2*B*—Zn*B*—N2*B*	70.8 (2)
O2*A*—Zn*A*—N3*A*	84.6 (2)	O2*B*—Zn*B*—N3*B*	84.0 (2)
O4*A*—Zn*A*—N1*A*	100.8 (3)	O4*B*—Zn*B*—N1*B*	100.7 (3)
O4*A*—Zn*A*—N2*A*	98.1 (3)	O4*B*—Zn*B*—N2*B*	98.4 (3)
O4*A*—Zn*A*—N3*A*	79.4 (3)	O4*B*—Zn*B*—N3*B*	79.9 (3)
N1*A*—Zn*A*—N2*A*	77.6 (3)	N1*B*—Zn*B*—N2*B*	78.2 (3)
N1*A*—Zn*A*—N3*A*	166.2 (3)	N1*B*—Zn*B*—N3*B*	165.7 (3)
N2*A*—Zn*A*—N3*A*	88.7 (3)	N2*B*—Zn*B*—N3*B*	87.6 (3)

Table 2. *Hydrogen-bonding geometry (Å, °)*

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O2*A*—H1···O1*B*	0.80	1.89	2.562 (8)	141
O2*B*—H2···O1*A*	0.86	1.88	2.593 (8)	144

Data collection: *MSC/AFC Diffractometer Control Software* (Molecular Structure Corporation, 1991). Cell refinement: *MSC/AFC Diffractometer Control Software*. Data reduction: *TEXSAN* (Molecular Structure Corporation, 1992). Program(s) used to solve structure: *SHELXS*86 (Sheldrick, 1990) and *DIRDIF*92 (Beurskens *et al.*, 1992). Program(s) used to refine structure: *TEXSAN*. Software used to prepare material for publication: *TEXSAN*.

Supplementary data for this paper are available from the IUCr electronic archives (Reference: OA1049). Services for accessing these data are described at the back of the journal.

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Oxonium Λ-{*N*,*N*′-bis[2(*S*)-pyrrolidinylmethyl]ethane-1,2-diamine}-*cis*-α-dichlorocobalt(III) dichloride dihydrate

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Abstract

In the complex cation of the title compound, $(H_3O)[CoCl_2(C_{12}H_{26}N_4)]Cl_2\cdot2H_2O$, the two Cl ligands are in *cis* positions. The complex has a twofold axis and is nearly octahedral. The chiral C atoms possess the *SS* configuration. This configuration may require the secondary amines on the ligand to have the *SS* configuration also, and may be responsible for the ring conformations.

Comment

Stereospecific *N*,*N*′-bis[2(*S*)-pyrrolidinylmethyl]ethane-1,2-diamine tetrahydrochloride (*S*-epm·4HCl) has been