and refined by least-squares methods (*SHELX*76; Sheldrick, 1976). Molecular graphics were produced with the *SCHAKAL* program (Keller, 1988). Material for publication was prepared using *BONDLA* (Hall & Stewart, 1990) and *PARST* (Nardelli, 1983).

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Lists of structure factors, anisotropic displacement parameters, H-atom coordinates, complete geometry and torsion angles have been deposited with the IUCr (Reference: NA1087). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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Zinc(II) Complex of Urocanic Acid

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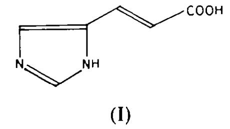
Abstract

In the title compound, tetraaquabis(4-imidazoleacrylato)zinc(II), $[Zn(C_6H_5N_2O_2)_2(H_2O)_4]$, the Zn atom has a quasi-octahedral coordination environment with four water O atoms in a square-planar arrangement and two N atoms of the imidazole ring in *trans* positions. The acrylic acid side chain is partially disordered. The imidazole ring and the carboxyl group have a *trans* configuration about the double bond and the carboxyl group is tilted towards the imidazole ring. The urocanic acid molecules are held together *via* hydrogen bonds involving all available H atoms.

Comment

Urocanic acid (I) is the first metabolic product of histidine. It has two geometrical isomers which display *trans* or *cis* configurations of the imidazole ring and the carboxyl group about the double bond of the acryl side chain. The biological activity of *trans*-urocanic acid (for example in the contraction of blood vessels or the lowering of blood pressure) is greater than that of *cis*-urocanic acid. Also, the major pathway of histidine metabolism is *via trans*-urocanic acid (Kurogochi, Fukui, Nakagawa & Yamamoto, 1957; Abrams & Borsook, 1952). The crystal structure of urocanic acid dihydrate was reported to compise molecules with the *trans* configuration (Hawkinson, 1977; Svinning & Sorum, 1979).



(I)

In order to investigate the interaction between imidazole compounds and zinc ions, zinc–urocanic acid complexes (1:2 and 1:1) were synthesized (Weitzel & Fretzdorff, 1956); tetrahedral coordination models were proposed for the *cis* configurations of each complex, but

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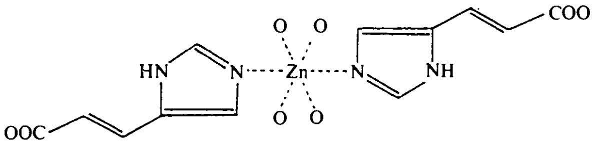
were not tested experimentally. In this study, we aim to clarify the interaction mode between the zinc cation and urocanic acid.



The crystal structure analysis of urocanic acid dihydrate (Hawkinson, 1977; Svinning & Sorum, 1979) shows that the molecule is nearly planar and the two water molecules are positioned along the acryl side chain. The crystal structure analysis of the zinc–urocanic acid complex (Fig. 1) shows that the urocanic acid molecule is essentially non-planar, with the carboxyl group tilted towards the imidazolium ring by 41.5 (5)°. Four water molecules are coordinated to the zinc ion; none is found near the acryl side chain. The atoms C(6*A*) and C(7*A*) of the acryl side chain are partially disordered with the corresponding atoms, denoted C(6*B*) and C(7*B*), in *trans* configurations. Each of these disordered C atoms was assumed to be equally distributed over the two sites and the occupancies of both sites were assumed to be 0.5. Analogous partially disordered structures near π bonds are observed for 1-ethyl-5-bromouracil (Tsukihara, Ashida & Kakudo, 1972), *trans*-stilbene (Hoekstra, Meertens & Vos, 1975) and betaxolol hydrochloride (Pascard, Huu Dau, Manoury & Mompon, 1984). The H atoms bound to C(6*B*) and C(7*B*) could not be located. Molecules are linked through a hydrogen-bonded network in which the water molecules and the imino groups of the imidazole rings act as proton donors and the O atoms of the carboxylate groups function as proton acceptors. All H atoms of these donor groups are hydrogen bonded to the acceptors. Details of the hydrogen-bonding geometry have been deposited.

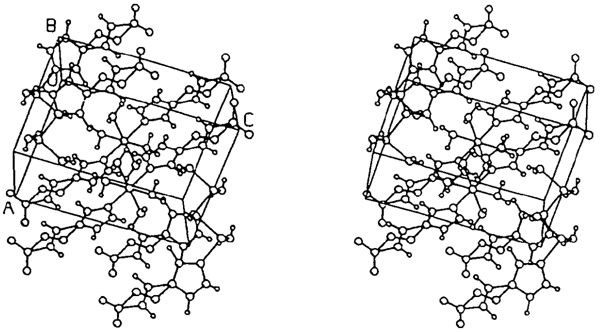


Fig. 2. Stereoscopic view (*PLUTO*; Motherwell & Clegg, 1978) of the packing of the title complex.

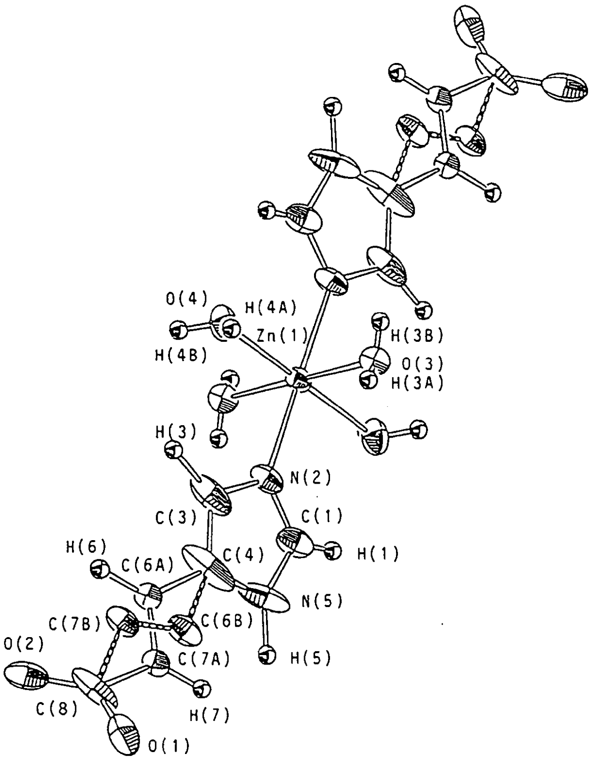


Fig. 1. A perspective view (*ORTEP*II; Johnson, 1976) of the molecular structure of the title complex with the atomic numbering scheme. Non-H atoms are shown as 50% probability ellipsoids.

Experimental

Crystals were obtained by the slow evaporation at room temperature of 20 ml of an aqueous solution containing 1 mmol of urocanic acid and 0.5 mmol of $Zn(OH)_2$ at pH 9.0.

Crystal data

$[Zn(C_6H_5N_2O_2)_2(H_2O)_4]$
$M_r = 411.68$
Monoclinic
$P2_1/c$
$a = 8.825$ (2) Å
$b = 7.203$ (2) Å
$c = 11.870$ (4) Å
$\beta = 92.38$ (2)°
$V = 753.9$ (3) Å^3
$Z = 2$
$D_x = 1.813$ Mg m^{-3}
$D_m = 1.810$ Mg m^{-3}

Mo $K\alpha$ radiation
$\lambda = 0.71073$ Å
Cell parameters from 25 reflections
$\theta = 21.2$–$24.3°$
$\mu = 3.315$ mm^{-1}
$T = 269$ K
Plate
0.3 × 0.2 × 0.1 mm
Colourless

Data collection

Rigaku AFC-5*R* diffractometer
$\omega/2\theta$ scans
Absorption correction: empirical (Walker & Stuart, 1983)
$T_{\min} = 0.93$, $T_{\max} = 1.05$
2001 measured reflections
1987 independent reflections
1403 observed reflections [$I > 3\sigma(I)$]

$R_{\rm int} = 0.017$
$\theta_{\max} = 27.5°$
$h = 0 \rightarrow 11$
$k = 0 \rightarrow 9$
$l = -15 \rightarrow 15$
3 standard reflections monitored every 150 reflections
intensity variation: 0.59% decline

Refinement

Refinement on F
$R = 0.032$
$wR = 0.042$
$S = 1.72$
1403 reflections
133 parameters
H-atom parameters not refined
$w = 4F_o^2/\sigma^2(F_o^2)$
$(\Delta/\sigma)_{\max} = 0.01$
$\Delta\rho_{\max} = 0.30$ e Å^{-3}
$\Delta\rho_{\min} = -0.46$ e Å^{-3}
Atomic scattering factors from *International Tables for X-ray Crystallography* (1974, Vol. IV)

Table 1. *Fractional atomic coordinates and equivalent isotropic displacement parameters* (Å^2)

$$B_{eq} = (8\pi^2/3)\Sigma_i\Sigma_j U_{ij}a_i^*a_j^*\mathbf{a}_i.\mathbf{a}_j.$$

	x	y	z	B_{eq}
Zn(1)	1/2	0	0	1.92 (2)
O(1)	−0.3655 (4)	0.0313 (3)	0.3422 (2)	3.9 (1)
O(2)	−0.1896 (2)	0.1038 (3)	0.4734 (2)	3.5 (1)
O(3)	0.4251 (2)	−0.2564 (3)	−0.0797 (2)	2.45 (8)
O(4)	0.5572 (3)	−0.1526 (3)	0.1498 (2)	2.9 (1)
N(2)	0.2861 (3)	0.0631 (3)	0.0572 (2)	2.4 (1)
N(5)	0.0563 (3)	0.1817 (4)	0.0575 (3)	4.3 (1)
C(1)	0.1816 (4)	0.1580 (5)	0.0005 (3)	3.0 (1)
C(3)	0.2257 (5)	0.0278 (5)	0.1606 (3)	4.0 (2)
C(4)	0.0836 (5)	0.0987 (5)	0.1620 (4)	5.2 (2)
C(6*A*)	−0.0022 (7)	0.0770 (8)	0.2717 (6)	2.0 (2)
C(6*B*)	−0.070 (1)	0.103 (1)	0.2118 (7)	2.8 (3)
C(7*A*)	−0.1512 (6)	0.1011 (8)	0.2643 (5)	2.0 (2)
C(7*B*)	−0.070 (1)	0.066 (1)	0.3192 (7)	2.4 (3)
C(8)	−0.2324 (5)	0.0721 (5)	0.3726 (4)	4.0 (2)

Table 2. *Selected geometric parameters* (Å, °)

Zn(1)—O(3)	2.167 (2)	N(5)—C(1)	1.331 (4)
Zn(1)—O(4)	2.132 (2)	N(5)—C(4)	1.389 (6)
Zn(1)—N(2)	2.082 (3)	C(3)—C(4)	1.354 (6)
O(1)—C(8)	1.250 (5)	C(4)—C(6*A*)	1.541 (8)
O(2)—C(8)	1.260 (5)	C(4)—C(6*B*)	1.501 (9)
N(2)—C(1)	1.310 (4)	C(6*A*)—C(7*A*)	1.325 (8)
N(2)—C(3)	1.382 (4)	C(6*B*)—C(7*B*)	1.30 (1)
C(7*A*)—C(8)	1.513 (7)	C(7*B*)—C(8)	1.59 (1)
O(3)—Zn(1)—O(4)	88.97 (8)	N(5)—C(4)—C(3)	106.1 (3)
O(3)—Zn(1)—N(2)	93.68 (8)	N(5)—C(4)—C(6*A*)	136.5 (4)
O(4)—Zn(1)—N(2)	91.5 (1)	N(5)—C(4)—C(6*B*)	102.5 (5)
Zn(1)—N(2)—C(1)	125.0 (2)	C(3)—C(4)—C(6*A*)	117.4 (5)
Zn(1)—N(2)—C(3)	129.6 (3)	C(3)—C(4)—C(6*B*)	149.8 (5)
C(1)—N(2)—C(3)	105.2 (3)	C(4)—C(6*A*)—C(7*A*)	116.9 (6)
C(1)—N(5)—C(4)	106.4 (3)	C(4)—C(6*B*)—C(7*B*)	114.6 (9)
N(2)—C(1)—N(5)	112.8 (3)	C(6*A*)—C(7*A*)—C(8)	115.4 (6)
N(2)—C(3)—C(4)	109.4 (4)	C(6*B*)—C(7*B*)—C(8)	114.9 (8)
O(1)—C(8)—C(7*A*)	105.1 (4)	O(1)—C(8)—O(2)	124.0 (3)
O(1)—C(8)—C(7*B*)	136.9 (4)	O(2)—C(8)—C(7*A*)	130.3 (4)
O(2)—C(8)—C(7*B*)	98.2 (4)		

Data collection and cell refinement: *Rigaku MSC/AFC Data Collection and Refinement Software* (Rigaku Corporation, 1988). The scan rate was 8.0° min^{-1} and the scan width was (1.31 + 0.30 tanθ)°. The ratio of peak counting time to background counting time was 2:1. Structure solution: *MITHRIL* (Gilmore, 1984). Structure refinement: *DIRDIF* (Beurskens, 1984). All calculations, including data reduction: *TEXSAN* (Molecular Structure Corporation, 1985). Refinement was by full-matrix least-squares methods with anisotropic displacement parameters for all non-H atoms. Molecular graphics: *ORTEP*II (Johnson, 1976), *PLUTO* (Motherwell & Clegg, 1978).

Lists of structure factors, anisotropic displacement parameters, H-atom coordinates, hydrogen-bonding geometry, complete geometry and torsion angles have been deposited with the IUCr (Reference: HR1000). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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Tris(triphenyltin)borate

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Abstract

The central BO_3 unit in $B(OSnPh_3)_3$ has essentially trigonal-planar geometry but the Sn atoms are located