Lists of structure factors, anisotropic displacement parameters and H-atom coordinates have been deposited with the IUCr (Reference: NA1105). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

References

André, D. & Michalowicz, A. (1991). *MACORTEP*. Laboratoire de Physicochimie Structurale, UFR de Sciences et Technologie, Créteil, France.

Caëtano, O., Lopez, M., Mahoui, A., Lapasset, J., Moret, J., Assih, T. & Saint Grégoire, P. (1993). *Ferroelectrics*. In the press.

Cromer, D. T. & Mann, J. B. (1968). *Acta Cryst.* A**24**, 321–324.

Johnson, C. K. (1976). *ORTEP*II. Report ORNL-5138. Oak Ridge National Laboratory, Tennessee, USA.

Morosin, B. & Lingafelter, E. C. (1959). *Acta Cryst.* **12**, 611–612.

Sawada, S., Shiroishi, Y., Takashige, M. & Matsuo, M. (1978). *Phys. Lett. A*, **67**, 56–58.

Sawada, S., Shiroishi, Y., Yamamoto, A., Takashige, M. & Matsuo, M. (1978). *J. Phys. Soc. Jpn*, **44**, 2.

Sheldrick, G. M. (1976). *SHELX*76. *Program for Crystal Structure Determination*. Univ. of Cambridge, England.

Sheldrick, G. M. (1985). *SHELXS*86. *Program for the Solution of Crystal Structures*. Univ. of Göttingen, Germany.

Stucky, G. D., Folkers, J. B. & Kistenmacher, T. J. (1967). *Acta Cryst.* **23**, 1064–1070.

Wolthuis, A. J., Huiskamp, W. J., Carlin, R. L. & De Jongh, L. J. (1986). *Physica*, **B142**, 301–319.

Acta Cryst. (1995). C**51**, 222–224

A Zinc(II) Complex of Creatinine

NOBUO OKABE, YOSHIKO KOHYAMA AND KAZUYUKI IKEDA

Faculty of Pharmaceutical Sciences, Kinki University, Kowakae 3-4-1, Higashiosaka, Osaka 577, Japan

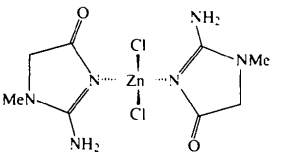
(*Received* 16 *September* 1993; *accepted* 1 *September* 1994)

Abstract

In the crystals of bis(2-amino-1,5-dihydro-1-methyl-4*H*-imidazol-4-one-N^3)dichlorozinc(II), $[ZnCl_2(C_4H_7N_3O)_2]$, the Zn atom is fourfold coordinated by the two N atoms situated at the 3 positions of the imidazole rings and by two Cl atoms. The coordination environment at the Zn atom is distorted tetrahedral. The amino and carbonyl groups participate in a hydrogen-bond network.

Comment

Creatinine (2-amino-1,5-dihydro-1-methyl-4*H*-imidazol-4-one) is a final metabolic product of arginine. It is one of the factors inducing chronic renal failure and/or uremic symptoms (Bell, Lee, Sadler, Wilkie & Woodham, 1991). Toxic effects in rats (Yokozawa, Mo & Oura, 1989) and determination by a flow-injection biosensor system (Rui, Sonomoto, Ogawa & Kato, 1993) or by an optical sensor using a host–guest system (Buhlmann, Badertscher & Simon, 1993) have been reported. To date, the crystal structures of creatinine (du Pré & Mendel, 1955) and its serotonin (Karle, Dragonette & Brenner, 1965), phenylmercury(II) (Canty, Chaichit & Gatehouse, 1979) and platinum(II) complexes (Bontchev *et al.*, 1987; Mitewa, Gencheva, Bontchev, Angelova & Maciček, 1988) have been reported. In order to obtain structural information on the mode of interaction between creatinine and biologically important metal ions, we thought it worthwhile to determine the crystal structure of the complex, (I), of creatinine with zinc(II) chloride.



(I)

The molecular structure of the title complex with atomic labelling is depicted in Fig. 1. A stereoview of the molecules in the unit cell is depicted in Fig. 2. The molecules are held together by intermolecular hydrogen bonds between amino and carbonyl groups: N(2)—H(1)···O(1)(x, y, $z-1$) 2.96 (1) and N(2′)—H(1′)···O(1′)(x, y, $1+z$) 2.797 (9) Å.

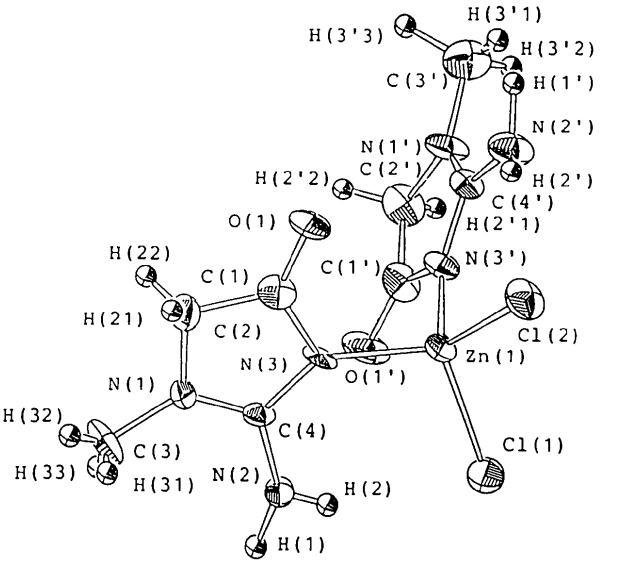


Fig. 1. Perspective view of the title compound with the atomic numbering. Ellipsoids are shown at 50% probability.

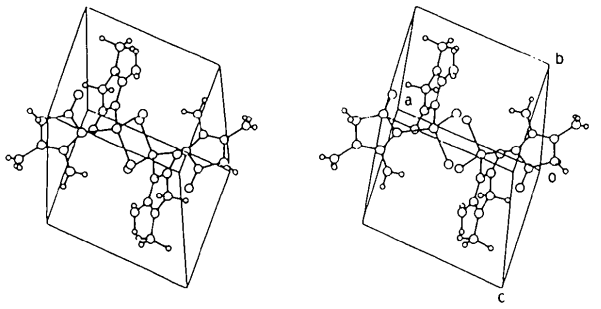


Fig. 2. Stereoview showing the packing in the unit cell viewed from the same direction as Fig. 1.

Experimental

Crystal data

$[ZnCl_2(C_4H_7N_3O)_2]$	Mo $K\alpha$ radiation
M_r = 362.52	λ = 0.71069 Å
Triclinic	Cell parameters from 25 reflections
$P\bar{1}$	θ = 10.3–19.0°
a = 8.184 (5) Å	μ = 2.195 mm^{-1}
b = 12.363 (4) Å	T = 296 K
c = 7.101 (2) Å	Needle
α = 94.93 (3)°	0.3 × 0.1 × 0.1 mm
β = 94.00 (4)°	Colourless
γ = 77.34 (4)°	Crystal source: crystallized by evaporation from H_2O
V = 697.5 (6) Å^3	
Z = 2	
D_x = 1.726 Mg m^{-3}	

Data collection

Rigaku AFC-5*R* diffractometer	1248 observed reflections [$I > 3\sigma(I)$]
ω–2θ scans	R_{int} = 0.076
Absorption correction: refined from ΔF (*DIFABS*; Walker & Stuart, 1983)	θ_{max} = 27.5°
	h = 1 → 10
	k = −14 → 15
	l = −9 → 9
T_{min} = 0.87, T_{max} = 1.07	3 standard reflections monitored every 150 reflections
3426 measured reflections	intensity decay: 1.40%
3200 independent reflections	

Refinement

Refinement on F	$w = 4F_o^2/\sigma^2(F_o^2)$
R = 0.049	$(\Delta/\sigma)_{max}$ = 0.01
wR = 0.049	$\Delta\rho_{max}$ = 0.52 e Å^{-3}
S = 1.25	$\Delta\rho_{min}$ = −0.46 e Å^{-3}
1248 reflections	Atomic scattering factors from *International Tables for X-ray Crystallography* (1974, Vol. IV)
172 parameters	
H-atom parameters not refined	

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

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

	x	y	z	B_{eq}
Zn(1)	0.7124 (2)	0.2489 (1)	0.2518 (2)	2.17 (6)
Cl(1)	0.5414 (3)	0.2581 (2)	0.4920 (3)	3.2 (1)
Cl(2)	0.6093 (3)	0.1688 (2)	−0.0130 (3)	3.3 (1)
O(1)	0.807 (1)	0.4548 (6)	0.5098 (8)	3.9 (4)
O(1′)	1.0512 (9)	0.1536 (6)	0.0391 (8)	3.6 (4)
N(1)	0.776 (1)	0.5484 (7)	0.055 (1)	2.9 (5)
N(1′)	1.178 (1)	0.1006 (7)	0.508 (1)	2.5 (4)
N(2)	0.687 (1)	0.4065 (7)	−0.132 (1)	3.3 (5)
N(2′)	0.927 (1)	0.1563 (7)	0.662 (1)	2.7 (4)
N(3)	0.734 (1)	0.4031 (6)	0.201 (1)	2.2 (4)
N(3′)	0.942 (1)	0.1666 (6)	0.3336 (9)	2.0 (4)
C(1)	0.784 (1)	0.4733 (8)	0.342 (1)	2.8 (5)
C(1′)	1.070 (1)	0.1390 (8)	0.210 (1)	2.5 (5)
C(2)	0.809 (1)	0.5743 (8)	0.256 (1)	3.2 (6)
C(2′)	1.234 (1)	0.0919 (9)	0.315 (1)	2.8 (5)
C(3)	0.781 (1)	0.6245 (9)	−0.089 (1)	3.9 (6)
C(3′)	1.295 (1)	0.0622 (9)	0.665 (1)	3.3 (6)
C(4)	0.733 (1)	0.4509 (8)	0.033 (1)	2.1 (5)
C(4′)	1.018 (1)	0.1396 (7)	0.506 (1)	1.7 (5)

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

Zn(1)—Cl(1)	2.261 (3)	N(1′)—C(3′)	1.46 (1)
Zn(1)—Cl(2)	2.249 (3)	N(1′)—C(4′)	1.29 (1)
Zn(1)—N(3)	2.022 (7)	N(2)—C(4)	1.32 (1)
Zn(1)—N(3′)	2.009 (7)	N(2′)—C(4′)	1.35 (1)
O(1)—C(1)	1.22 (1)	N(3)—C(1)	1.36 (1)
O(1′)—C(1′)	1.23 (1)	N(3)—C(4)	1.37 (1)
N(1)—C(2)	1.45 (1)	N(3′)—C(1′)	1.38 (1)
N(1)—C(3)	1.46 (1)	N(3′)—C(4′)	1.363 (9)
N(1)—C(4)	1.32 (1)	C(1)—C(2)	1.50 (1)
N(1′)—C(2′)	1.46 (1)	C(1′)—C(2′)	1.51 (1)
Cl(1)—Zn(1)—Cl(2)	110.5 (1)	Zn(1)—N(3)—C(4)	130.3 (6)
Cl(1)—Zn(1)—N(3)	110.0 (2)	C(1)—N(3)—C(4)	107.7 (8)
Cl(1)—Zn(1)—N(3′)	108.3 (2)	Zn(1)—N(3′)—C(1′)	121.4 (5)
Cl(2)—Zn(1)—N(3)	109.0 (2)	Zn(1)—N(3′)—C(4′)	133.2 (6)
Cl(2)—Zn(1)—N(3′)	112.6 (2)	C(1′)—N(3′)—C(4′)	104.9 (7)
N(3)—Zn(1)—N(3′)	106.5 (3)	O(1)—C(1)—N(3)	125.6 (9)
C(2)—N(1)—C(3)	123.1 (8)	O(1)—C(1)—C(2)	126.1 (9)
C(2)—N(1)—C(4)	108.5 (7)	N(3)—C(1)—C(2)	108.2 (8)
C(3)—N(1)—C(4)	128.3 (8)	O(1′)—C(1′)—N(3′)	124.0 (9)
C(2′)—N(1′)—C(3′)	121.0 (8)	O(1′)—C(1′)—C(2′)	125.8 (9)
C(2′)—N(1′)—C(4′)	109.1 (7)	N(3′)—C(1′)—C(2′)	110.2 (7)
C(3′)—N(1′)—C(4′)	129.8 (8)	N(1)—C(2)—C(1)	102.5 (8)
Zn(1)—N(3)—C(1)	121.1 (6)	N(1′)—C(2′)—C(1′)	100.2 (7)
N(1)—C(4)—N(2)	123.9 (8)	N(1′)—C(4′)—N(2′)	123.9 (8)
N(1)—C(4)—N(3)	113.0 (8)	N(1′)—C(4′)—N(3′)	115.6 (7)
N(2)—C(4)—N(3)	123.1 (9)	N(2′)—C(4′)—N(3′)	120.4 (8)

The scan rate was 8° min^{-1} in ω and the scan width was (1.63 + 0.30tanθ)°. The ratio of peak counting time to background counting time was 2:1. Refinement was by a full-matrix least-squares method.

Data collection and cell refinement: *Rigaku MSC/AFC Data Collection and Refinement Software* (Rigaku Corporation, 1988). Programs used to solve structure: *MITHRIL* (Gilmore, 1984) and *DIRDIF* (Beurskens, 1984). All calculations, including data reduction: *TEXSAN* (Molecular Structure Corporation, 1985).

Lists of structure factors, anisotropic displacement parameters, H-atom coordinates, complete geometry, including bond distances and angles involving H atoms, and torsion angles have been deposited with the IUCr (Reference: HU1081). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

References

Bell, J. D., Lee, J. A., Sadler, P. J., Wilkie, D. R. & Woodham, R. H. (1991). *Biochim. Biophys. Acta*, **1096**, 101–107.

Beurskens, P. T. (1984). *DIRDIF. Direct Methods for Difference Structures – an Automatic Procedure for Phase Extension and Refinement of Difference Structure Factors.* Technical Report 1984/1. Crystallography Laboratory, Toernooiveld, 6525 ED Nijmegen, The Netherlands.

Bontchev, P. R., Mitewa, M., Gencheva, G., Maciček, J., Angelova, O. & Nefed, V. I. (1987). *Proc. Conf. Coord. Chem.* **11**, 37–43.
Buhlmann, P., Badertscher, M. & Simon, W. (1993). *Tetrahedron*, **49**, 595–598.
Canty, A. J., Chaichit, N. & Gatehouse, B. M. (1979). *Acta Cryst.* B**35**, 592–596.
Gilmore, C. J. (1984). *J. Appl. Cryst.* **17**, 42–46.
Karle, I. L., Dragonette, K. S. & Brenner, S. A. (1965). *Acta Cryst.* **19**, 713–716.
Mitewa, M., Gencheva, G., Bontchev, P. R., Angelova, O. & Maciček, J. (1988). *Polyhedron*, **7**, 1273–1278.
Molecular Structure Corporation (1985). *TEXSAN. TEXRAY Structure Analysis Package*. MSC, 3200 Research Forest Drive, The Woodlands, TX 77381, USA.
Pré, S. du & Mendel, H. (1955). *Acta Cryst.* **8**, 311–313.
Rigaku Corporation (1988). *Rigaku MSC/AFC Data Collection and Refinement Software*. Rigaku Corporation, Nishishinjuk 4-15-3, Tokyo 160, Japan.
Rui, C.-S., Sonomoto, K., Ogawa, H. I. & Kato, Y. (1993). *Anal. Biochem.* **210**, 163–171.
Walker, N. & Stuart, D. (1983). *Acta Cryst.* A**39**, 159–166.
Yokozawa, T., Mo, Z. L. & Oura, H. (1989). *Nephron*, **51**, 388–392.

Acta Cryst. (1995). C**51**, 224–226

Dichlorobis(creatinine)cadmium(II)

NOBUO OKABE, KAZUYUKI IKEDA AND YOSHIKO KOHYAMA

Faculty of Pharmaceutical Sciences, Kinki University, Kowakae 3-4-1, Higashiosaka, Osaka 577, Japan

YOH SASAKI

Department of Science and Engineering, Kinki University, Kowakae 3-4-1, Higashiosaka, Osaka 577, Japan

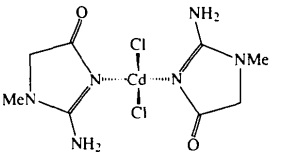
(*Received* 3 *November* 1993; *accepted* 1 *September* 1994)

Abstract

The structure of bis(2-amino-1,5-dihydro-1-methyl-4*H*-imidazol-4-one-N^3)dichlorocadmium(II), [$CdCl_2$-($C_4H_7N_3O)_2$], consists of four-coordinate molecular units with the metal centre bonded to two imidazole N(3) atoms and two Cl ions. The coordination about the Cd atom is pseudo-tetrahedral. The amino H atom is hydrogen bonded to the carbonyl O atom of an adjacent molecule.

Comment

Creatinine (2-amino-1,5-dihydro-1-methyl-4*H*-imidazol-4-one) is the anhydride form of creatine and is produced in muscle, by dehydration of creatine phosphate, as the final arginine metabolite (Rodwell, 1983). Normal blood contains 7–15 mg l^{-1} of creatinine, which is used as a diagnostic index reflecting kidney function (Martin, 1983). Creatinine is also known to be one of the factors inducing chronic renal failure and/or uremic symptoms (Bell, Lee, Sadler, Wilkie & Woodham, 1991). It is important to determine the precise mode of interaction between biological substances and biologically significant metal ions as this information is fundamental for understanding many biological and physiological phenomena involving metal ions. To date, the crystal structure of creatinine (du Pré & Mandel, 1955) and its phenylmercury(II) (Canty, Chaichit & Gatehouse, 1979) and platinum(II) complexes (Bontchev *et al.*, 1987; Mitewa, Gencheva, Bontchev, Angelova & Maciček, 1988) have been reported. In this study, we report the crystal structure of the chelate complex, (I), of creatinine with cadmium(II) chloride which is a significant toxic metal ion causing toxicosis, *e.g.* itai-itai disease.



(I)

The molecular structure of the title complex with labelling is shown in Fig. 1. A stereoview of the unit cell is presented in Fig. 2. The creatinine molecules are held together by intermolecular hydrogen bonds

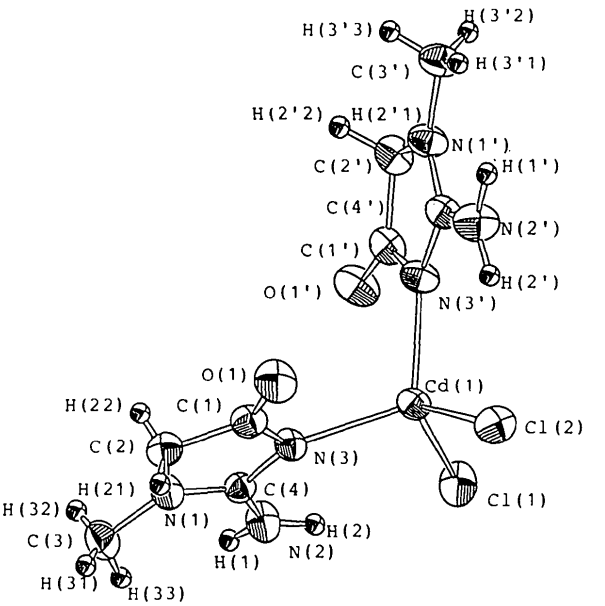


Fig. 1. Perspective view of the title compound with the atomic numbering. Ellipsoids are drawn at 50% probability.