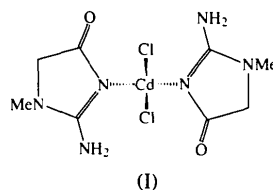


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final arginine metabolite (Rodwell, 1983). Normal blood contains 7–15 mg l<sup>-1</sup> of creatinine, which is used as a diagnostic index reflecting kidney function (Martin, 1983). Creatinine is also known to be one of the factors inducing chronic 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.



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## Dichlorobis(creatinine)cadmium(II)

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### Abstract

The structure of bis(2-amino-1,5-dihydro-1-methyl-4*H*-imidazol-4-one-*N*<sup>3</sup>)dichlorocadmium(II), [CdCl<sub>2</sub>·(C<sub>4</sub>H<sub>7</sub>N<sub>3</sub>O)<sub>2</sub>], 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

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

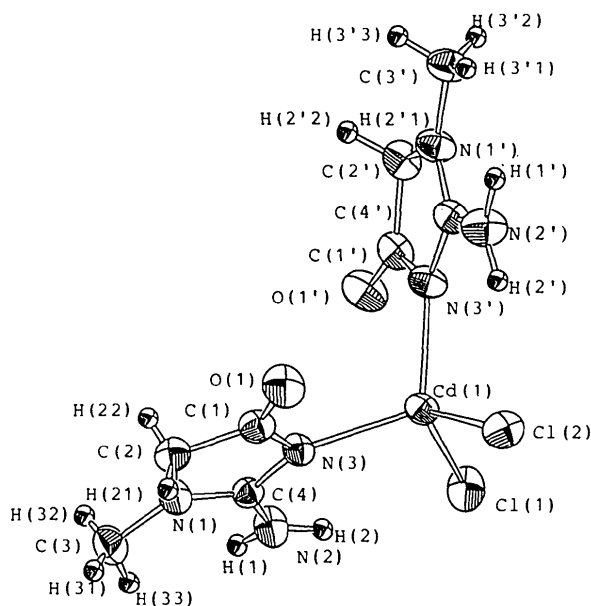


Fig. 1. Perspective view of the title compound with the atomic numbering. Ellipsoids are drawn at 50% probability.

involving the amino H atoms and the carbonyl O atoms: N(2)—H(1)···O(1)(x, y, 1 + z) 3.003 (3) and N(2')—H(1')···O(1')(x, y, z - 1) 2.967 (3) Å.

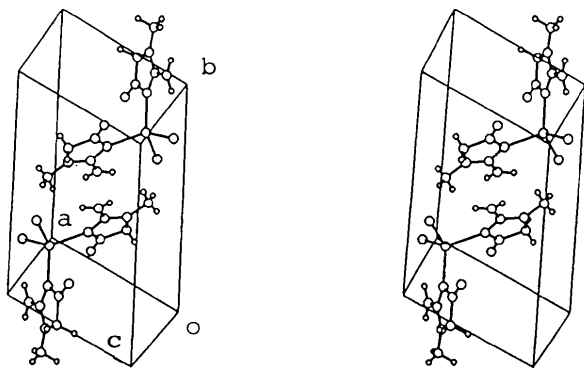


Fig. 2. Stereoview showing the packing in the unit cell viewed from the same direction as Fig. 1.

## Experimental

### Crystal data

[CdCl<sub>2</sub>(C<sub>4</sub>H<sub>7</sub>N<sub>3</sub>O)<sub>2</sub>]

*M<sub>r</sub>* = 409.55

Triclinic

*P* $\bar{1}$

*a* = 8.568 (2) Å

*b* = 12.697 (2) Å

*c* = 7.1759 (6) Å

$\alpha$  = 101.30 (1)°

$\beta$  = 107.17 (1)°

$\gamma$  = 73.30 (1)°

*V* = 709.2 (2) Å<sup>3</sup>

*Z* = 2

*D<sub>x</sub>* = 1.918 Mg m<sup>-3</sup>

*D<sub>m</sub>* = 1.909 (8) Mg m<sup>-3</sup>

### Data collection

Rigaku AFC-5R diffractometer

$\omega$ -2 $\theta$  scans

Absorption correction:

refined from  $\Delta F$

(DIFABS; Walker & Stuart, 1983)

*T<sub>min</sub>* = 0.97, *T<sub>max</sub>* = 1.04

3479 measured reflections

3263 independent reflections

### Refinement

Refinement on *F*

*R* = 0.022

*wR* = 0.030

*S* = 1.27

2924 reflections

172 parameters

H-atom parameters not refined

Mo *K* $\alpha$  radiation

$\lambda$  = 0.71069 Å

Cell parameters from 25 reflections

$\theta$  = 13.45–24.85°

$\mu$  = 1.921 mm<sup>-1</sup>

*T* = 296 K

Needle

0.45 × 0.20 × 0.15 mm

Colourless

Crystal source: crystallized by evaporation from H<sub>2</sub>O

2924 observed reflections

[*I* > 3 $\sigma$ (*I*)]

*R<sub>int</sub>* = 0.010

$\theta_{max}$  = 27.5°

*h* = 0 → 11

*k* = -15 → 15

*l* = -9 → 8

3 standard reflections

monitored every 150

reflections

intensity decay: none

$w = 4F_o^2/\sigma^2(F_o^2)$

( $\Delta/\sigma$ )<sub>max</sub> = 0.001

$\Delta\rho_{max}$  = 0.32 e Å<sup>-3</sup>

$\Delta\rho_{min}$  = -0.35 e Å<sup>-3</sup>

Atomic scattering factors

from *International Tables for X-ray Crystallography* (1974, Vol. IV)

Table 1. Fractional atomic coordinates and equivalent isotropic displacement parameters (Å<sup>2</sup>)

$$B_{eq} = (8\pi^2/3)\sum_i\sum_j U_{ij}a_i^*a_j^*a_i \cdot a_j$$

	<i>x</i>	<i>y</i>	<i>z</i>	<i>B<sub>eq</sub></i>
Cd(1)	0.23065 (2)	0.76897 (1)	0.23694 (2)	2.459 (8)
Cl(1)	0.05457 (9)	0.76988 (7)	0.4487 (1)	3.67 (3)
Cl(2)	0.1258 (1)	0.68753 (6)	-0.09785 (9)	3.41 (3)
O(1)	0.5943 (3)	0.6506 (2)	0.1291 (3)	4.0 (1)
O(1')	0.3022 (3)	0.9883 (2)	0.5328 (3)	3.9 (1)
N(1)	0.7052 (3)	0.5836 (2)	0.6079 (3)	2.65 (8)
N(1')	0.2762 (3)	1.0805 (2)	0.0951 (3)	2.9 (1)
N(2)	0.4477 (3)	0.6506 (2)	0.6938 (3)	3.0 (1)
N(2')	0.2130 (3)	0.9309 (2)	-0.1369 (3)	3.5 (1)
N(3)	0.4784 (3)	0.6674 (2)	0.3873 (3)	2.47 (8)
N(3')	0.2460 (3)	0.9340 (2)	0.2005 (3)	2.73 (9)
C(1)	0.6066 (3)	0.6342 (2)	0.2955 (4)	2.8 (1)
C(1')	0.2818 (3)	1.0065 (2)	0.3648 (3)	2.8 (1)
C(2)	0.7651 (3)	0.5773 (2)	0.4351 (4)	2.9 (1)
C(2')	0.2964 (4)	1.1097 (2)	0.3054 (3)	3.0 (1)
C(3)	0.8066 (3)	0.5304 (2)	0.7808 (4)	3.2 (1)
C(3')	0.2929 (4)	1.1509 (2)	-0.0306 (4)	3.5 (1)
C(4)	0.5426 (3)	0.6337 (2)	0.5699 (3)	2.21 (9)
C(4')	0.2431 (3)	0.9816 (2)	0.0441 (3)	2.5 (1)

Table 2. Selected geometric parameters (Å, °)

Cd(1)—Cl(1)	2.4334 (7)	N(1')—C(3')	1.449 (3)
Cd(1)—Cl(2)	2.4359 (7)	N(1')—C(4')	1.324 (3)
Cd(1)—N(3)	2.226 (2)	N(2)—C(4)	1.322 (3)
Cd(1)—N(3')	2.207 (2)	N(2')—C(4')	1.318 (3)
O(1)—C(1)	1.221 (3)	N(3)—C(1)	1.367 (3)
O(1')—C(1')	1.224 (3)	N(3)—C(4)	1.365 (3)
N(1)—C(2)	1.456 (3)	N(3')—C(1')	1.362 (3)
N(1)—C(3)	1.451 (3)	N(3')—C(4')	1.367 (3)
N(1)—C(4)	1.325 (3)	C(1)—C(2)	1.513 (4)
N(1')—C(2')	1.452 (3)	C(1')—C(2')	1.503 (4)
Cl(1)—Cd(1)—Cl(2)	111.95 (3)	Cd(1)—N(3)—C(4)	133.6 (2)
Cl(1)—Cd(1)—N(3)	101.76 (5)	C(1)—N(3)—C(4)	107.0 (2)
Cl(1)—Cd(1)—N(3')	115.10 (6)	Cd(1)—N(3')—C(1')	118.2 (2)
Cl(2)—Cd(1)—N(3)	112.33 (6)	Cd(1)—N(3')—C(4')	134.1 (2)
Cl(2)—Cd(1)—N(3')	103.76 (5)	C(1')—N(3')—C(4')	107.3 (2)
N(3)—Cd(1)—N(3')	112.28 (8)	O(1)—C(1)—N(3)	124.9 (2)
C(2)—N(1)—C(3)	123.7 (2)	O(1)—C(1)—C(2)	126.1 (2)
C(2)—N(1)—C(4)	108.8 (2)	N(3)—C(1)—C(2)	109.0 (2)
C(3)—N(1)—C(4)	127.1 (2)	O(1')—C(1')—N(3')	124.9 (2)
C(2')—N(1')—C(3')	123.5 (2)	O(1')—C(1')—C(2')	126.2 (2)
C(2')—N(1')—C(4')	108.7 (2)	N(3')—C(1')—C(2')	108.8 (2)
C(3')—N(1')—C(4')	127.8 (2)	N(1)—C(2)—C(1)	101.5 (2)
Cd(1)—N(3)—C(1)	119.2 (2)	N(1')—C(2')—C(1')	101.8 (2)
N(1)—C(4)—N(2)	124.5 (2)	N(1')—C(4')—N(2')	124.9 (2)
N(1)—C(4)—N(3)	113.7 (2)	N(1')—C(4')—N(3')	113.2 (2)
N(2)—C(4)—N(3)	121.8 (2)	N(2')—C(4')—N(3')	121.9 (2)

The scan rate was 32° min<sup>-1</sup> in  $\omega$  and the scan width was (1.57 + 0.30tan $\theta$ )°. 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: *SHELXS86* (Sheldrick, 1985) 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: HU1089). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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## Anhydrous 2,3,5,6-Tetramethylpyrazinium Tetraiodocadmate(II) and its Trihydrate, [C<sub>8</sub>H<sub>12</sub>N<sub>2</sub>(H)<sub>2</sub>][CdI<sub>4</sub>].xH<sub>2</sub>O (x = 0, 3)

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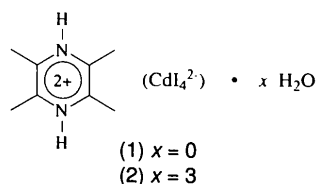
(Received 11 October 1994; accepted 12 May 1994)

## Abstract

The structures of diprotonated 2,3,5,6-tetramethylpyrazinium tetraiodocadmate(II) as an anhydrous salt, (C<sub>8</sub>H<sub>14</sub>N<sub>2</sub>)[CdI<sub>4</sub>], (1), and as a trihydrate, (C<sub>8</sub>H<sub>14</sub>N<sub>2</sub>)[CdI<sub>4</sub>].3H<sub>2</sub>O, (2), have been determined. In both compounds the CdI<sub>4</sub><sup>2-</sup> anion has slightly distorted tetrahedral geometry with Cd—I bonds ranging from 2.755 (2) to 2.793 (2) Å for (1) and from 2.751 (2) to 2.795 (2) Å for (2). In (1), the cations and anions are joined through hydrogen bonds to form layers in the *ab* plane. In (2) the cations are linked through hydrogen bonds to strings of three hydrogen-bonded water molecules to form infinite chains; there are no significant interactions involving the anions.

## Comment

We have shown recently that tetramethylpyrazinium cations crystallize with polyiodide anions to give a sheet structure, in which the polyiodide layers consist of a unique arrangement of interwoven branched chains of I<sub>3</sub><sup>-</sup> anions and I<sub>2</sub> molecules (Bailey & Pennington, 1995). In order to determine the extent to which this cation can influence the stoichiometry and structure of other anionic species, we have extended this work to iodocadmates. While not as structurally diverse as polyiodides (Tebbe, 1977; Coppens, 1982), the structures of iodocadmates have been shown to vary as a function of the counter cation: a tetrahedral CdI<sub>4</sub><sup>2-</sup> anion crystallizes with tetramethylammonium (Kallel, Bats & Daoud, 1981), methyltriphenylphosphonium (Couldwell & Prout, 1978) or alkali metal cations (Sjövall, 1989), and a Cd<sub>2</sub>I<sub>6</sub><sup>2-</sup> anion, composed of two edge-sharing CdI<sub>4</sub> tetrahedra, crystallizes with a [tris(2-dimethylaminoethyl)amine]iodocadmium(II) cation (Orioli & Ciampolini, 1972). In order to determine the effect of a tetramethylpyrazinium cation on the structure of iodocadmate anions, we have prepared the compounds [tetramethylpyrazine(H)<sub>2</sub>]CdI<sub>4</sub>, (1), and [tetramethylpyrazine(H)<sub>2</sub>]CdI<sub>4</sub>.3H<sub>2</sub>O, (2) (Fig. 1).



Both compounds crystallize with CdI<sub>4</sub><sup>2-</sup> anions. The Cd atom has distorted tetrahedral geometry [102.0 (1)–118.5 (1)°] in (1), while in (2) the range of the relevant angles is closer to the ideal [106.3 (1)–112.8 (1)°]. The Cd—I bond distances [2.755 (2)–2.793 (2) Å for (1); 2.751 (2)–2.795 (2) Å for (2)] lie within the range of those of known compounds.

H atoms could not be located, but the angles at the N atoms of the cations in both structures agree well with those of a protonated N atom [124.1 (5)°] in the tetramethylpyrazinium cation of a triiodide salt (Bailey & Pennington, 1995), indicating that the cations are protonated at both the N atoms of the pyrazine ring.

In the anhydrous compound, (1), the cations interact with the anions through bifurcated N—H···I hydrogen bonds; the N···I distances of 3.501 (6) and 3.540 (6) Å involving atom N(1) and those of 3.525 (6) and 3.534 (6) Å involving atom N(2) are significantly shorter than the nitrogen–iodine van der Waals distance of 3.65 Å (Pauling, 1960). Ion pairs participating in these interactions are related by 2<sub>1</sub> screw operations and by translation along the *a* and *b* axes, linking the ions into an extended two-dimensional network (Fig. 2). The strength of the Cd—I bond appears to be inversely related to the strength of N—H···I interaction, the I atom