Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the IUCr (Reference: BK1259). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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Acta Cryst. (1996). C**52**, 3043-3045

A Cadmium(II) Complex of Creatinine, $[Cd(C_2H_3O_2)_2(C_4H_7N_3O)_2].0.5H_2O$

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(*Received 30 April 1996; accepted 22 July 1996*)

Abstract

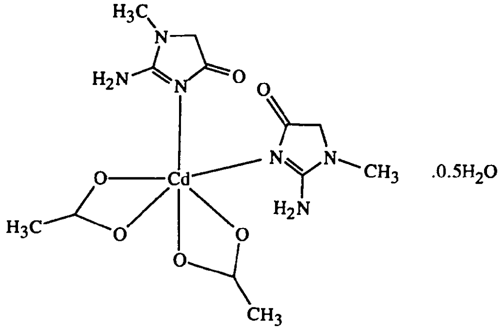
The crystal structure of bis(acetato-*O*,*O*′)bis(2-amino-1,5-dihydro-1-methyl-4*H*-imidazol-4-one-*N*^{3})cadmium(II) hemihydrate, $[Cd(CH_3CO_2)_2(creat)_2].0.5H_2O$, shows the coordination geometry around the Cd atom to be distorted octahedral involving two monodentate heterocyclic N-bound creatinine molecules and two *O*,*O*-bidentate acetate groups.

Comment

Creatinine is the final catabolic product of creatine. The concentration of creatinine in blood and urine is an important factor in assessing renal function. In order to probe the metabolic processes that occur with creatinine, many studies investigating the coordination chemistry of the ligand have been carried out and, more recently, comprehensively reviewed (Mitewa, 1995).

Structural studies involving creatinine include its phenylmercury(II) (Canty, Fyfe & Gatehouse, 1978; Canty, Chaichit & Gatehouse, 1979), silver(I) (Udupa & Krebs, 1981), tetrachlorocuprate(II) (Udupa & Krebs, 1979), platinum(II) (Mitewa, Gencheva, Bontchev, Angelova & Maciček, 1988; Maciček, Angelova, Gencheva, Mitewa & Bontchev, 1988; Beurskens, Perales, Martin-Gil & Martin-Gil, 1988; Beja, Paixao, Martin-Gil & Salgado, 1991; Gencheva *et al.*, 1992) and palladium(II) (Beurskens *et al.*, 1988) complexes.

As part of our studies (Chotalia, Hambley, Ridley & Turner, 1996) on the modes of coordination of creatinine with different metal ions, we have investigated the reactivity of the ligand with the Group 12 metal triad. At the time of the preparation of this communication, the structures of $[Zn(creat)_2Cl_2]$ (Okabe, Kohyama & Ikeda, 1995) and $[Cd(creat)_2Cl_2]$ (Okabe, Ikeda & Kohyama, 1995) had been reported. Both have four-coordinate tetrahedral metal centres coordinated by two imidazole N atoms and two Cl atoms. The title compound, $[Cd(creat)_2(CH_3CO_2)_2].0.5H_2O$, (I), was obtained upon mixing creatinine and $[Cd(CH_3CO_2)_2].2H_2O$ in ethanol–acetone (1:1).



(I)

The structure of (I) with the atom numbering is shown in Fig. 1. The metal ion is in a distorted octahedral environment, coordinated by two creatinine imidazole

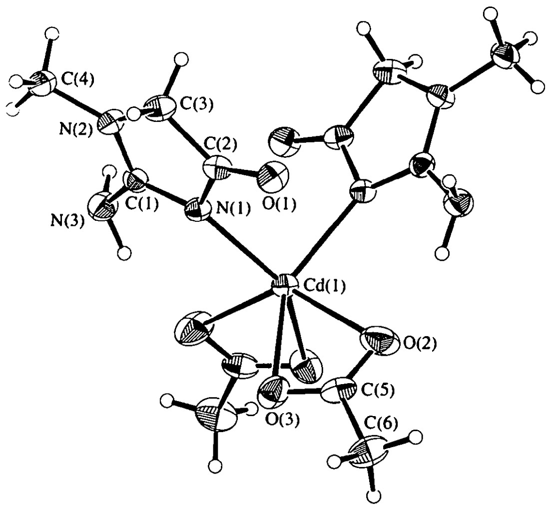


Fig. 1. A perspective view showing the structure of $[Cd(creat)_2(CH_3CO_2)_2]$ and the atom-numbering scheme. Displacement ellipsoids are shown at the 25% probability level.

Acta Crystallographica Section C
ISSN 0108-2701 © 1996

N atoms and two *cis*-related *O*,*O*-bidentate acetate ions. The Cd—N(1) distances of 2.255 (3) Å are typical of Cd—N distances within imidazole-type ligands (Prince, 1987). The complex also displays intramolecular hydrogen bonding between an N(3) proton and the O atom of an acetate ion, O(2). The water molecule, O(4), does not take part in hydrogen bonding.

Experimental

The title compound was obtained upon mixing creatinine and $[Cd(CH_3CO_2)_2].2H_2O$ in a 1:1 solution of ethanol and acetone.

Crystal data

$[Cd(C_2H_3O_2)_2(C_4H_7N_3O)_2]$.-$0.5H_2O$
$M_r = 465.76$
Tetragonal
$I4_1/a$
$a = 13.791$ (2) Å
$c = 20.313$ (4) Å
$V = 3863.4$ (9) $Å^3$
$Z = 8$
$D_x = 1.601$ Mg m^{-3}
D_m not measured

Mo $K\alpha$ radiation
$\lambda = 0.7107$ Å
Cell parameters from 25 reflections
$\theta = 19.26$–$24.16°$
$\mu = 1.174$ mm^{-1}
$T = 294$ K
Octahedron
0.50 × 0.30 × 0.30 mm
Colourless

Data collection

Enraf–Nonius CAD-4 diffractometer
$\omega/2\theta$ scans
Absorption correction: analytical (de Meulenaer & Tompa, 1965)
$T_{\min} = 0.730$, $T_{\max} = 0.749$
2395 measured reflections
1810 independent reflections

1472 observed reflections $[I > 2\sigma(I)]$
$R_{\rm int} = 0.017$
$\theta_{\max} = 24.95°$
$h = -1 \rightarrow 16$
$k = -1 \rightarrow 16$
$l = -1 \rightarrow 24$
3 standard reflections monitored every 60 min intensity decay: 0.46%

Refinement

Refinement on F
$R = 0.0305$
$wR = 0.0302$
$S = 2.798$
1472 reflections
118 parameters
H-atom parameters not refined
$w = 1/[\sigma^2(F_o)]$

$(\Delta/\sigma)_{\max} = 0.0002$
$\Delta\rho_{\max} = 0.72$ e $Å^{-3}$
$\Delta\rho_{\min} = -0.46$ e $Å^{-3}$
Extinction correction: none
Atomic scattering factors from *International Tables for Crystallography* (1992, Vol. C, Tables 6.1.1.1 and 6.1.1.2)

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

$$U_{\rm eq} = (1/3)\Sigma_i\Sigma_j U_{ij}a_i^*a_j^*\mathbf{a}_i.\mathbf{a}_j.$$

	x	y	z	$U_{\rm eq}$
Cd(1)	0	3/4	0.09463 (2)	0.0424 (1)
O(1)	−0.0702 (2)	0.6022 (2)	−0.0198 (1)	0.063 (1)
O(2)	−0.1585 (3)	0.7157 (3)	0.1393 (2)	0.090 (1)
O(3)	−0.0357 (3)	0.6380 (2)	0.1797 (2)	0.072 (1)
O(4)†	−0.460 (2)	0.726 (2)	0.1416 (10)	0.16 (1)
N(1)	0.0750 (2)	0.6601 (2)	0.0171 (1)	0.0413 (9)
N(2)	0.1731 (2)	0.6129 (2)	−0.0649 (2)	0.049 (1)
N(3)	0.2402 (3)	0.7026 (3)	0.0209 (2)	0.061 (1)
C(1)	0.1654 (3)	0.6598 (3)	−0.0079 (2)	0.044 (1)
C(2)	0.0170 (3)	0.6122 (3)	−0.0262 (2)	0.047 (1)
C(3)	0.0783 (3)	0.5760 (3)	−0.0825 (2)	0.056 (1)
C(4)	0.2609 (3)	0.5892 (3)	−0.1009 (2)	0.060 (1)
C(5)	−0.1242 (4)	0.6573 (4)	0.1785 (2)	0.061 (2)
C(6)	−0.1908 (4)	0.6097 (4)	0.2281 (2)	0.088 (2)

† O(4) has $U_{\rm iso}$ refined and an occupancy fixed at 0.25.

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

Cd(1)—O(2)	2.413 (4)	N(1)—C(2)	1.361 (5)
Cd(1)—O(3)	2.370 (3)	N(2)—C(1)	1.331 (5)
Cd(1)—N(1)	2.255 (3)	N(2)—C(3)	1.447 (5)
O(1)—C(2)	1.217 (5)	N(2)—C(4)	1.452 (5)
O(2)—C(5)	1.228 (5)	N(3)—C(1)	1.324 (5)
O(3)—C(5)	1.249 (5)	C(2)—C(3)	1.506 (5)
N(1)—C(1)	1.347 (5)	C(5)—C(6)	1.513 (6)
O(2)—Cd(1)—O(2$^{\rm i}$)	135.8 (2)	C(1)—N(1)—C(2)	107.4 (3)
O(2)—Cd(1)—O(3)	53.8 (1)	C(1)—N(2)—C(3)	108.3 (3)
O(2)—Cd(1)—O(3$^{\rm i}$)	92.4 (1)	C(1)—N(2)—C(4)	127.8 (4)
O(2)—Cd(1)—N(1)	124.8 (1)	C(3)—N(2)—C(4)	123.4 (3)
O(2)—Cd(1)—N(1$^{\rm i}$)	87.4 (1)	N(1)—C(1)—N(2)	113.8 (4)
O(2$^{\rm i}$)—Cd(1)—N(1)	87.4 (1)	N(1)—C(1)—N(3)	123.6 (4)
O(3)—Cd(1)—O(3$^{\rm i}$)	86.4 (2)	N(2)—C(1)—N(3)	122.6 (4)
O(3)—Cd(1)—N(1)	104.2 (1)	O(1)—C(2)—N(1)	124.5 (4)
O(3)—Cd(1)—N(1$^{\rm i}$)	140.6 (1)	O(1)—C(2)—C(3)	126.7 (4)
N(1)—Cd(1)—N(1$^{\rm i}$)	91.5 (2)	N(1)—C(2)—C(3)	108.7 (4)
Cd(1)—O(2)—C(5)	91.3 (3)	N(2)—C(3)—C(2)	101.8 (3)
Cd(1)—O(3)—C(5)	92.8 (3)	O(2)—C(5)—O(3)	122.0 (5)
Cd(1)—N(1)—C(1)	133.6 (3)	O(2)—C(5)—C(6)	118.9 (5)
Cd(1)—N(1)—C(2)	116.7 (3)	O(3)—C(5)—C(6)	119.2 (5)

Symmetry code: (i) $-x, \frac{3}{2} - y, z$.

Table 3. *Hydrogen-bonding geometry (Å, °)*

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N(3)—H(1N3)···O(2$^{\rm i}$)	0.97	1.94	2.885 (5)	162.9
N(3)—H(2N3)···O(3$^{\rm ii}$)	0.97	2.26	2.983 (5)	130.8

Symmetry codes: (i) $-x, \frac{3}{2} - y, z$; (ii) $y - \frac{1}{4}, \frac{3}{4} - x, z - \frac{1}{4}$.

The water O(4) atom is 0.72 Å away from the fourfold *a* axis. Its occupancy was initially refined and then fixed at 0.25, this being consistent with its proximity to a fourfold axis. H atoms were placed at calculated positions and given group displacement parameters. The H atoms of the water molecule were not included.

Data collection: *MSC/AFC Diffractometer Control Software* (Molecular Structure Corporation, 1993). Cell refinement: *MSC/AFC Diffractometer Control Software*. Data reduction: *TEXSAN* (Molecular Structure Corporation, 1995). Program(s) used to solve structure: *SHELXS*86 (Sheldrick, 1985). Program(s) used to refine structure: *TEXSAN*. Software used to prepare material for publication: *TEXSAN*.

The authors thank the Cooperative Research Centre for Molecular Engineering and Technology and the Australian Research Council for financial support.

Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the IUCr (Reference: TA1102). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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Acta Cryst. (1996). C**52**, 3045–3047

Bis(triethylsulfonium) Tetraiodocadmate(II)

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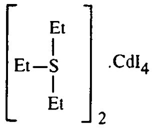
(*Received 2 February 1996; accepted 28 May 1996*)

Abstract

The reaction of CdI_2 with an acetone solution of $(Et_3S)I$ (molar ratio 1:2) gives the title compound. The structure of $[(C_2H_5)_3S]_2[CdI_4]$ contains discrete pyramidal Et_3S^+ and tetrahedral CdI_4^{2-} ions. The bond distances of the two independent anions, Cd1—I1 and Cd2—I2, are 2.772 (1) and 2.784 (1) Å, respectively. The cation, with approximate threefold symmetry, is disordered such that each of the middle methylene groups takes two positions with a ratio of *ca* 2:1.

Comment

The title compound, $(Et_3S)_2[CdI_4](s)$, (I), consists of discrete pyramidal Et_3S^+ cations and tetrahedral CdI_4^{2-} moieties.



$$\left[Et_3S\right]_2 . CdI_4$$

(I)

Intra- and intermolecular distances and angles are listed in Table 2. The cations with approximate threefold symmetry are disordered over two distinct orientations with populations of 0.65 (2) and 0.35 (2) (Fig. 1). The S and methyl C atoms coincide approximately in the two orientations, but the methylene C atoms are displaced between 1.34 (4) and 1.50 (5) Å. The S—C distances and S—C—C angles in the tetraiodocadmate(II) compound, as calculated from the disordered atom positions, differ somewhat from those obtained in the ordered $(Et_3S)[Hg_2I_6]_{1/2}$ (Bengtsson, Norén & Stegemann, 1995). The cation–anion arrangement is related to the antifluorite-type structure, but the 'cubes' of cations are heavily distorted. The triethylsulfonium cations are positioned so that the pyramidal S atoms are pointing towards the faces of the $Cd2I_4^{2-}$ tetrahedra. The 'back side' of the cations are centred over the I atoms of $Cd1I_4^{2-}$. A similar relationship between the cations and anions was found in $(Et_3S)[Hg_2I_6]_{1/2}$. Also, the I···S distances are of the same magnitude and indicate that the cation–anion interaction is predominantly of electrostatic and van der Waals types (Bengtsson *et al.*, 1995).

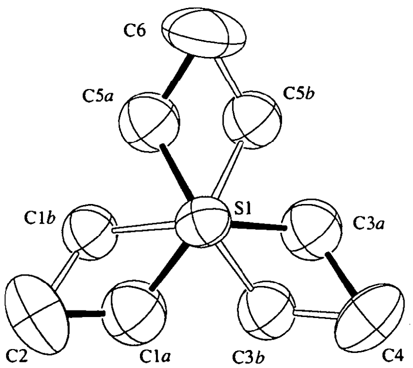


Fig. 1. The molecular structure of the Et_3S^+ cation showing 50% probability displacement ellipsoids. The major disorder components of the ethyl groups are shown with filled bonds.

Acta Crystallographica Section C
ISSN 0108-2701 © 1996