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A polymeric cadmium(II) complex of fusaric acid

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Key indicators

Single-crystal X-ray study
T = 296 K
Mean σ(C–C) = 0.016 Å
R factor = 0.047
wR factor = 0.188
Data-to-parameter ratio = 19.4

For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

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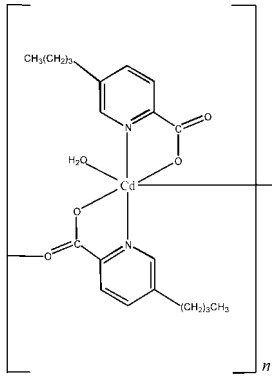
The title compound, *catena*-poly[[aqua(5-butyl-2-pyridine carboxylato-κ^2N,O)cadmium(II)]-μ-5-butyl-2-pyridinecarboxylato-$\kappa^3N,O:O'$], $[Cd^{II}(C_6H_4NO_2)_2(H_2O)]$, is a polymeric structure containing six-coordinate Cd^{II} in a distorted octahedral arrangement. The Cd^{II} atom is bonded to two N,O-bidentate ligands and one water O atom. The sixth coordination site is filled by a bridging Cd—O bond from a neighboring ligand.

Comment

Fusaric acid (5-butylpyridine-2-carboxylic acid or 5-butylpicolinic acid) is a fungal toxin, a mycotoxin produced by the *Fusarium* species which causes infections in cereal grains and agricultural commodities (Wang & Ng, 1999). It shows toxic activity towards some mammalian cell lines such as dog kidney fibroblast, rat hepatoma and Chinese hamster ovary (Wang & Ng, 1999). It and the related compound picolinic acid (2-pyridinecarboxylic acid) also induce apotosis in human promyelocytic leukemic HL-60 cells (Ogata *et al.*, 2001). On the other hand, the chelation of these compounds enhances the Fenton reaction which generates active oxygen species; the enhancement may be partly related to their biological activities, such as a marked growth-inhibitory action on rice seeding (Iwahashi *et al.*, 1999). For these reasons, it seems important to determine the structure of the chelate compounds of fusaric acid. DNA single-strand scission has been found in the kidneys and lungs of rats after parenteral administration of Cd^{II} and Ni^{II} (Kasprzak, 2002), and in the present study, the structure of the cadmium(II) complex, (I), has been determined.



(I)

This is the first report of the structure of a metal complex of fusaric acid. The molecular structure of (I) is shown in Fig. 1. It has a polymeric structure in which neighboring Cd^{II} ions are linked through carboxylate O atoms of a ligand molecule. One coordination unit consists of $[Cd^{II}(fusa)_2(H_2O)]$ (fusa is fusaric acid). The central Cd^{II} atom has a distorted octahedral

coordination geometry; it is bonded to three carboxylate O atoms, two N atoms of the two bidentate ligand molecules and one water O atom. Both carboxyl groups of the ligand molecules are ionized and essentially coplanar with the pyridine ring planes, as indicated by the relevant torsion angles [O1—C7—C2—N1 −171.5 (9)° and O4—C17—C12—N2 −2.2 (9)°]. Both butyl groups are positioned out of the planes of the pyridine rings, as indicated by the torsion angles C4—C5—C8—C9 of 76 (1)° and C14—C15—C18—C19 of 85 (1)°. The conformations of the butyl groups of the two ligand molecules are quite different from each other; one (C8—C9—C10—C11) has a *gauche–trans* conformation with respect to the C8—C9 bond [C5—C8—C9—C10 67 (1)°] and the C9—C10 bond [C8—C9—C10—C11 173 (1)°], respectively; the other (C18—C19—C20—C21) has a *trans–gauche* conformation with respect to the C18—C19 bond [C15—C18—C19—C20 175.8 (9)°] and C19—C20 bond [C18—C19—C20—C21 65 (1)°], respectively.

The distances between Cd^{II} and the carboxylate O atoms are shorter than those observed in the dinuclear cadmium(II) complexes of picolinic acid [Cd—O 2.313 (3)–2.332 (3) Å; Odoko *et al.*, 2001] and of pyridine-2,6-dicarboxylic acid [Cd—O 2.376–2.478 Å; Odoko *et al.*, 2002]. By contrast, the Cd—N distances are longer than those observed in those compounds [Cd—N 2.309–2.326 Å]. There is no stacking interaction between the pyridine rings in the crystal structure. The coordinated water O atom is hydrogen bonded to neighboring carboxylate O atoms [O5···O1^{i} 2.76 (1) Å and O5···O4^{ii} 2.66 (1) Å; symmetry codes as in Table 2]. The crystal structure is stabilized by its polymeric nature, van der Waals interactions and hydrogen-bond formation.

Experimental

The colorless plate-shaped crystal used for analysis was obtained by slow evaporation from a 50% ethanol–water solution of a mixture of fusaric acid and cadmium(II) chloride (4:1).

Crystal data

$[Cd(C_6H_4NO_2)_2(H_2O)]$	D_x = 1.506 Mg m^{-3}
M_r = 486.84	Mo $K\alpha$ radiation
Monoclinic, $P2_1/c$	Cell parameters from 25 reflections
a = 15.610 (3) Å	θ = 12.0–14.1°
b = 9.623 (4) Å	μ = 1.05 mm^{-1}
c = 15.923 (3) Å	T = 296.2 K
β = 116.17 (1)°	Plate, colorless
V = 2146.7 (11) Å^3	0.40 × 0.20 × 0.05 mm
Z = 4	

Data collection

Rigaku AFC-5*R* diffractometer	R_{int} = 0.065
ω–2θ scans	θ_{max} = 27.5°
Absorption correction: ψ scan (North *et al.*, 1968)	$h = 0 \rightarrow 20$
T_{min} = 0.777, T_{max} = 0.949	$k = 0 \rightarrow 12$
5402 measured reflections	$l = -20 \rightarrow 18$
4939 independent reflections	3 standard reflections
2200 reflections with $I > 2\sigma(I)$	every 150 reflections
	intensity decay: 1.3%

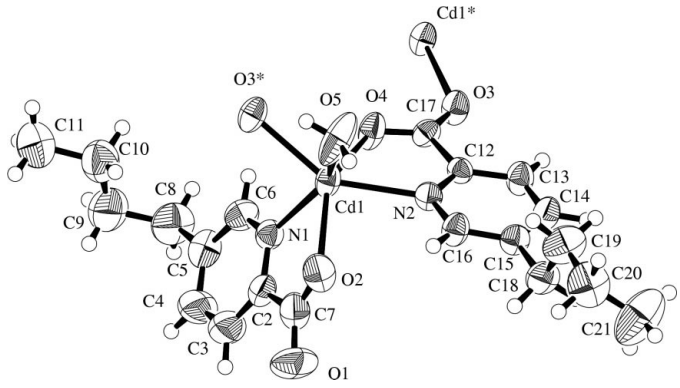


Figure 1
*ORTEP*II (Johnson, 1976) drawing of the title compound, with the atomic numbering scheme. Displacement ellipsoids for non-H atoms are drawn at the 50% probability level.

Refinement

Refinement on F^2	H-atom parameters constrained
$R[F^2 > 2\sigma(F^2)]$ = 0.047	$w = 1/[\sigma^2(F_o^2) + (0.1P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$wR(F^2)$ = 0.188	$(\Delta/\sigma)_{max} < 0.001$
S = 0.92	$\Delta\rho_{max}$ = 0.61 e Å^{-3}
4939 reflections	$\Delta\rho_{min}$ = −0.69 e Å^{-3}
255 parameters	

Table 1
Selected geometric parameters (Å, °).

Cd1—O2	2.262 (7)	O1—C7	1.22 (1)
Cd1—O3^{i}	2.308 (7)	O2—C7	1.25 (1)
Cd1—O4	2.301 (6)	O3—C17	1.245 (9)
Cd1—O5	2.274 (8)	O4—C17	1.26 (1)
Cd1—N1	2.370 (7)	C4—C5	1.40 (1)
Cd1—N2	2.333 (8)		
O2—Cd1—O3^{i}	125.8 (2)	O4—Cd1—N2	70.5 (3)
O2—Cd1—O4	139.6 (2)	O5—Cd1—N1	148.5 (3)
O2—Cd1—O5	94.9 (3)	O5—Cd1—N2	86.5 (3)
O2—Cd1—N1	71.8 (3)	N1—Cd1—N2	119.7 (2)
O2—Cd1—N2	86.2 (3)	Cd1—O2—C7	119.8 (8)
O3^{i}—Cd1—O4	85.3 (2)	Cd1^{ii}—O3—C17	127.6 (6)
O3^{i}—Cd1—O5	84.3 (3)	Cd1—O4—C17	119.3 (6)
O3^{i}—Cd1—N1	81.3 (2)	Cd1—N1—C2	113.0 (6)
O3^{i}—Cd1—N2	147.3 (2)	Cd1—N1—C6	127.8 (5)
O4—Cd1—O5	115.3 (2)	Cd1—N2—C12	116.6 (6)
O4—Cd1—N1	91.3 (2)	Cd1—N2—C16	124.2 (5)

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

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

D—H···A	D—H	H···A	D···A	D—H···A
O5—H5*WA*···O1^{i}	0.85	2.08	2.76 (1)	137
O5—H5*WB*···O4^{ii}	0.84	1.84	2.66 (1)	167

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

All H atoms were located from difference Fourier maps. However, the non-water H atoms were then placed at ideal positions and refined using a riding model; the water H atoms were not refined.

Data collection: *MSC/AFC Diffractometer Control Software* (Molecular Structure Corporation & Rigaku, 1999); cell refinement: *MSC/AFC Diffractometer Control Software*; data reduction: *TEXSAN* (Molecular Structure Corporation & Rigaku, 1999);

program(s) used to solve structure: *SIR*88 (Burla *et al.*, 1999) and *DIRDIF*94 (Beurskens *et al.*, 1992); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); molecular graphics: *ORTEP*II (Johnson, 1976); software used to prepare material for publication: *TEXSAN*.

References

Beurskens, P. T., Admiraal, G., Beurskens, G., Bosman, W. P., Garcia-Granda, S., Gould, R. O., Smits, J. M. M. & Smykalla, C. (1992). *The DIRDIF Program System*. Technical Report. Crystallography Laboratory, University of Nijmegen, The Netherlands.

Burla, M. C., Camalli, M., Cascarano, G., Giacovazzo, C., Polidori, G., Spagna, R. & Viterbo, D. (1989). *J. Appl. Cryst.* **22**, 389–393.

Iwahashi, H., Kawamori, H. & Fukushima, K. (1999). *Chem.-Biol. Interac.* **118**, 201–215.

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

Kasprzak, K. S. (2002). *Free Rad. Biol. Med.* **32**, 958–967.

Molecular Structure Corporation & Rigaku (1999). *MSC/AFC Diffractometer Control Software* and *TEXSAN* (Version 1.10). MSC, 3200 Research Forest Drive, The Woodlands, TX 77381, USA, and Rigaku Corporation, 3-9-12 Akishima, Tokyo, Japan.

North, A. C. T., Phillips, D. C. & Mathews, F. S. (1968). *Acta Cryst.* A**24**, 351–359.

Odoko, M., Isomoto, N. & Okabe, N. (2001). *Acta Cryst.* E**57**, m371–m372.

Odoko, M., Kusano, A. & Okabe, N. (2002). *Acta Cryst.* E**58**, m25–m27.

Ogata, S., Inoue, K., Okumura, K. & Taguchi, H. (2001). *Biosci. Biotech. Biochem.* **65**, 2337–2339.

Sheldrick, G. M. (1997). *SHELXL*97. University of Göttingen, Germany.

Wang, H. & Ng, T. B. (1999). *Life Sci.* **65**, 849–856.