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Tetraaquabis(3-carboxylatopyridine N-oxide- κO^3)cadmium(II)

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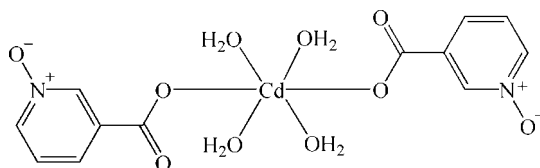
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 Key indicators: single-crystal X-ray study; $T = 293$ K; mean $\sigma(C-C) = 0.003$ Å; R factor = 0.017; wR factor = 0.047; data-to-parameter ratio = 11.9.

In the title complex, $[Cd(C_6H_4NO_3)_2(H_2O)_4]$, the Cd^{II} atom is situated on a crystallographic centre of inversion. The Cd^{II} atom shows a slightly distorted octahedral geometry and is coordinated by four O atoms from water molecules and two O atoms from deprotonated carboxyl groups of nicotinic acid N-oxide ligands. The mononuclear complex molecules are linked by $O-H \cdots O$ hydrogen bonds, forming a three-dimensional network structure.

Related literature

 For a related structure, see: Hilkka *et al.* (1983).


Experimental

Crystal data

 $[Cd(C_6H_4NO_3)_2(H_2O)_4]$
 $M_r = 460.67$

 Monoclinic, $P2_1/c$
 $a = 8.896$ (2) Å

 $b = 13.284$ (3) Å
 $c = 6.902$ (1) Å
 $\beta = 106.95$ (3)°
 $V = 780.2$ (3) Å³
 $Z = 2$

 Mo $K\alpha$ radiation
 $\mu = 1.46$ mm⁻¹
 $T = 293$ K
 $0.24 \times 0.24 \times 0.24$ mm

Data collection

 Bruker SMART CCD area-detector diffractometer
 Absorption correction: multi-scan (SADABS; Bruker, 1998)
 $T_{min} = 0.705$, $T_{max} = 0.712$

 3886 measured reflections
 1371 independent reflections
 1216 reflections with $I > 2\sigma(I)$
 $R_{int} = 0.013$

Refinement

 $R[F^2 > 2\sigma(F^2)] = 0.017$
 $wR(F^2) = 0.047$
 $S = 1.11$
 1371 reflections

 115 parameters
 H-atom parameters constrained
 $\Delta\rho_{max} = 0.26$ e Å⁻³
 $\Delta\rho_{min} = -0.27$ e Å⁻³

Table 1

Hydrogen-bond geometry (Å, °).

$D-H \cdots A$	$D-H$	$H \cdots A$	$D \cdots A$	$D-H \cdots A$
$O1W-H1WA \cdots O2^i$	0.85	1.90	2.678 (2)	151
$O1W-H1WB \cdots O3^{ii}$	0.85	1.86	2.697 (2)	165
$O2W-H2WA \cdots O3^{iii}$	0.86	1.86	2.716 (2)	175
$O2W-H2WB \cdots O2^{ii}$	0.86	1.93	2.787 (2)	173

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

Data collection: SMART (Bruker, 1998); cell refinement: SAINT (Bruker, 1998); data reduction: SAINT; program(s) used to solve structure: SHELXS97 (Sheldrick, 2008); program(s) used to refine structure: SHELXL97 (Sheldrick, 2008); molecular graphics: SHELXTL (Sheldrick, 2008); software used to prepare material for publication: SHELXTL.

This work was supported Beijing Municipal Natural Science Foundation (grant No. 2082004).

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: IM2121).

References

- Bruker (1998). SMART, SAINT and SADABS. Bruker AXS Inc., Madison, Wisconsin, USA.
 Hilkka, K., Univ, D. C. & Finland, J. J. (1983). *Acta Chem. Scand. Ser. A*, **37**, 697–702.
 Sheldrick, G. M. (2008). *Acta Cryst.* **A64**, 112–122.

supplementary materials

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Tetraaquabis(3-carboxylatopyridine *N*-oxide- κO^3)cadmium(II)

C.-Y. Zhang, Q. Gao, Y. Cui and Y.-B. Xie

Comment

The behaviour of nicotinic acid *N*-oxide ligand towards transition metals has been studied (Hilkka *et al.*, 1983). Herein, we prepared a new complex with the similar structure.

The title complex (Fig. 1) is made up of tetraaquametal cations and nicotinate *N*-oxide anion. The Cd^{II} centre shows a slightly distorted octahedral geometry and is six-coordinated by four O atoms from water molecules and two O atoms from deprotonated carboxylic groups of nicotinic acid *N*-oxide ligands. The O atoms of the *N*-oxide function bridge two water ligands of adjacent complex molecules *via* O—H \cdots O hydrogen bonds, forming infinite chains along *c* axis (Fig. 2). Otherwise, the chains are linked by additional O—H \cdots O hydrogen bonds observed between carboxyl O atoms and H atoms of coordinated water molecules. In conclusion, the mononuclear complexes are linked by O—H \cdots O hydrogen bonds, forming a three-dimensional network structure.

Experimental

A solution containing a 1 : 1 : 2 molar ratio of nicotinic acid *N*-oxide, LiOH \times H₂O and Cd(NO₃)₂ \times 4 H₂O in water was sealed in a 25 ml teflon reactor and kept at 140° for 3 days. The mixture was stepwise cooled to 40° with a rate of 10° per hour and was then allowed to cool to room temperature naturally. Colorless block-shaped crystals suitable for X-ray investigation were collected from the final mixture.

Refinement

All H atoms were fixed geometrically (C—H = 0.93 Å, O—H = 0.85–0.86 Å) and treated as riding with $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{carrier})$.

Figures

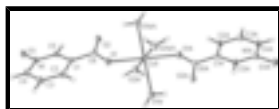


Fig. 1. The molecular structure of the title compound with displacement ellipsoids drawn at the 30% probability level for non-hydrogen atoms. Symmetry related atoms labelled A have the symmetry code $A = -x + 1, -y + 1, -z$.

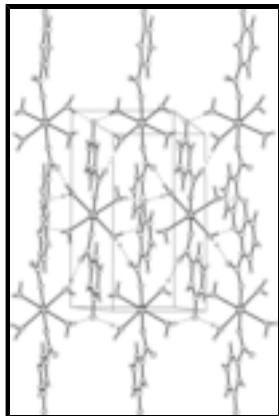


Fig. 2. Supramolecular structure of the title compound realized by O—H...O hydrogen bond.

Tetraaquabis(3-carboxylatopyridine N-oxide- κO^3)cadmium(II)

Crystal data

[Cd(C₆H₄NO₃)₂(H₂O)₄]

$M_r = 460.67$

Monoclinic, $P2_1/c$

Hall symbol: -P 2ybc

$a = 8.896$ (2) Å

$b = 13.284$ (3) Å

$c = 6.902$ (1) Å

$\beta = 106.95$ (3)°

$V = 780.2$ (3) Å³

$Z = 2$

$F_{000} = 460$

$D_x = 1.961$ Mg m⁻³

Mo $K\alpha$ radiation

$\lambda = 0.71073$ Å

Cell parameters from 2694 reflections

$\theta = 2.4$ – 30.8 °

$\mu = 1.46$ mm⁻¹

$T = 293$ K

Block, colorless

$0.24 \times 0.24 \times 0.24$ mm

Data collection

Bruker SMART CCD area-detector diffractometer

Radiation source: fine-focus sealed tube

Monochromator: graphite

$T = 293$ K

φ and ω scans

Absorption correction: Multi-Scan (SADABS; Bruker, 1998)

$T_{\min} = 0.705$, $T_{\max} = 0.712$

3886 measured reflections

1371 independent reflections

1216 reflections with $I > 2\sigma(I)$

$R_{\text{int}} = 0.013$

$\theta_{\text{max}} = 25.0$ °

$\theta_{\text{min}} = 2.4$ °

$h = -10 \rightarrow 9$

$k = -15 \rightarrow 13$

$l = -8 \rightarrow 8$

Refinement

Refinement on F^2

Least-squares matrix: full

$R[F^2 > 2\sigma(F^2)] = 0.017$

Secondary atom site location: difference Fourier map

Hydrogen site location: inferred from neighbouring sites

H-atom parameters constrained

$wR(F^2) = 0.047$	$w = 1/[\sigma^2(F_o^2) + (0.0239P)^2 + 0.3039P]$
$S = 1.11$	where $P = (F_o^2 + 2F_c^2)/3$
1371 reflections	$(\Delta/\sigma)_{\max} = 0.001$
115 parameters	$\Delta\rho_{\max} = 0.26 \text{ e } \text{\AA}^{-3}$
Primary atom site location: structure-invariant direct methods	$\Delta\rho_{\min} = -0.27 \text{ e } \text{\AA}^{-3}$
	Extinction correction: none

Special details

Geometry. All e.s.d.'s (except the e.s.d. in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell e.s.d.'s are taken into account individually in the estimation of e.s.d.'s in distances, angles and torsion angles; correlations between e.s.d.'s in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell e.s.d.'s is used for estimating e.s.d.'s involving l.s. planes.

Refinement. Refinement of F^2 against ALL reflections. The weighted R -factor wR and goodness of fit S are based on F^2 , conventional R -factors R are based on F , with F set to zero for negative F^2 . The threshold expression of $F^2 > \sigma(F^2)$ is used only for calculating R -factors(gt) *etc.* and is not relevant to the choice of reflections for refinement. R -factors based on F^2 are statistically about twice as large as those based on F , and R -factors based on ALL data will be even larger.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (\AA^2)

	x	y	z	$U_{\text{iso}}^*/U_{\text{eq}}$
Cd1	0.5000	0.5000	0.0000	0.02694 (9)
O1	0.69494 (17)	0.61434 (10)	0.0646 (2)	0.0322 (3)
O2	0.54243 (17)	0.73900 (11)	-0.1003 (2)	0.0356 (4)
O3	0.84106 (18)	1.04656 (11)	0.1475 (2)	0.0351 (4)
C5	0.7630 (2)	0.88034 (15)	0.0896 (3)	0.0258 (4)
H5A	0.6623	0.9017	0.0195	0.031*
C1	0.7954 (2)	0.77885 (14)	0.1108 (3)	0.0234 (4)
O1W	0.6079 (2)	0.42109 (12)	0.3026 (2)	0.0475 (5)
C6	0.6669 (2)	0.70560 (15)	0.0175 (3)	0.0257 (4)
C4	1.0221 (2)	0.92024 (16)	0.2703 (3)	0.0323 (5)
H4A	1.0988	0.9685	0.3232	0.039*
O2W	0.37122 (19)	0.60651 (11)	0.1620 (2)	0.0387 (4)
N1	0.8750 (2)	0.94858 (13)	0.1691 (3)	0.0263 (4)
C2	0.9455 (3)	0.74878 (15)	0.2168 (3)	0.0293 (5)
H2A	0.9699	0.6807	0.2349	0.035*
C3	1.0585 (2)	0.82017 (17)	0.2953 (3)	0.0349 (5)
H3A	1.1600	0.8004	0.3656	0.042*
H1WA	0.5831	0.3597	0.2735	0.042*
H1WB	0.6909	0.4234	0.4033	0.042*
H2WA	0.3009	0.5861	0.2151	0.042*
H2WB	0.4182	0.6543	0.2406	0.042*

Atomic displacement parameters (\AA^2)

U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
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supplementary materials

Cd1	0.02508 (13)	0.02246 (14)	0.02930 (14)	-0.00329 (8)	0.00169 (9)	-0.00103 (8)
O1	0.0296 (8)	0.0189 (7)	0.0431 (9)	-0.0038 (6)	0.0024 (7)	0.0023 (6)
O2	0.0315 (8)	0.0226 (8)	0.0428 (9)	-0.0029 (6)	-0.0046 (7)	0.0027 (7)
O3	0.0339 (8)	0.0176 (7)	0.0483 (9)	-0.0020 (6)	0.0034 (7)	-0.0013 (7)
C5	0.0213 (10)	0.0232 (10)	0.0301 (11)	-0.0017 (8)	0.0031 (8)	-0.0001 (8)
C1	0.0270 (10)	0.0196 (10)	0.0235 (10)	-0.0022 (8)	0.0072 (8)	-0.0001 (8)
O1W	0.0593 (11)	0.0263 (8)	0.0391 (9)	-0.0105 (8)	-0.0133 (8)	0.0035 (7)
C6	0.0279 (11)	0.0219 (11)	0.0269 (10)	-0.0035 (8)	0.0073 (9)	-0.0012 (8)
C4	0.0241 (11)	0.0301 (12)	0.0378 (12)	-0.0073 (9)	0.0015 (9)	-0.0026 (9)
O2W	0.0365 (9)	0.0331 (8)	0.0477 (10)	-0.0077 (7)	0.0143 (7)	-0.0101 (7)
N1	0.0269 (9)	0.0200 (9)	0.0300 (9)	-0.0021 (7)	0.0052 (7)	-0.0007 (7)
C2	0.0314 (11)	0.0217 (11)	0.0326 (12)	0.0014 (8)	0.0061 (9)	0.0013 (9)
C3	0.0239 (11)	0.0328 (12)	0.0420 (13)	0.0008 (9)	0.0006 (9)	0.0025 (10)

Geometric parameters (\AA , $^\circ$)

Cd1—O1 ⁱ	2.2499 (14)	C1—C2	1.382 (3)
Cd1—O1	2.2499 (14)	C1—C6	1.496 (3)
Cd1—O1W ⁱ	2.2836 (16)	O1W—H1WA	0.8537
Cd1—O1W	2.2836 (16)	O1W—H1WB	0.8538
Cd1—O2W	2.3045 (16)	C4—N1	1.345 (3)
Cd1—O2W ⁱ	2.3045 (16)	C4—C3	1.367 (3)
O1—C6	1.261 (2)	C4—H4A	0.9300
O2—C6	1.248 (2)	O2W—H2WA	0.8559
O3—N1	1.335 (2)	O2W—H2WB	0.8603
C5—N1	1.340 (3)	C2—C3	1.373 (3)
C5—C1	1.377 (3)	C2—H2A	0.9300
C5—H5A	0.9300	C3—H3A	0.9300
O1 ⁱ —Cd1—O1	180.0	Cd1—O1W—H1WA	102.3
O1 ⁱ —Cd1—O1W ⁱ	91.98 (6)	Cd1—O1W—H1WB	139.5
O1—Cd1—O1W ⁱ	88.02 (6)	H1WA—O1W—H1WB	109.2
O1 ⁱ —Cd1—O1W	88.02 (6)	O2—C6—O1	125.51 (19)
O1—Cd1—O1W	91.98 (6)	O2—C6—C1	118.05 (17)
O1W ⁱ —Cd1—O1W	180.00 (7)	O1—C6—C1	116.44 (18)
O1 ⁱ —Cd1—O2W	92.70 (6)	N1—C4—C3	119.74 (19)
O1—Cd1—O2W	87.30 (6)	N1—C4—H4A	120.1
O1W ⁱ —Cd1—O2W	91.49 (7)	C3—C4—H4A	120.1
O1W—Cd1—O2W	88.51 (7)	Cd1—O2W—H2WA	122.8
O1 ⁱ —Cd1—O2W ⁱ	87.30 (6)	Cd1—O2W—H2WB	122.7
O1—Cd1—O2W ⁱ	92.70 (6)	H2WA—O2W—H2WB	104.2
O1W ⁱ —Cd1—O2W ⁱ	88.51 (7)	O3—N1—C5	119.82 (16)
O1W—Cd1—O2W ⁱ	91.49 (7)	O3—N1—C4	119.00 (16)
O2W—Cd1—O2W ⁱ	180.0	C5—N1—C4	121.18 (18)
C6—O1—Cd1	120.98 (13)	C3—C2—C1	119.49 (19)
N1—C5—C1	120.73 (18)	C3—C2—H2A	120.3
N1—C5—H5A	119.6	C1—C2—H2A	120.3

C1—C5—H5A	119.6	C4—C3—C2	120.2 (2)
C5—C1—C2	118.64 (18)	C4—C3—H3A	119.9
C5—C1—C6	118.74 (18)	C2—C3—H3A	119.9
C2—C1—C6	122.62 (18)		
O1 ⁱ —Cd1—O1—C6	178 (100)	C5—C1—C6—O1	-170.21 (19)
O1W ⁱ —Cd1—O1—C6	39.82 (16)	C2—C1—C6—O1	10.0 (3)
O1W—Cd1—O1—C6	-140.18 (16)	C1—C5—N1—O3	-179.78 (18)
O2W—Cd1—O1—C6	-51.77 (16)	C1—C5—N1—C4	0.4 (3)
O2W ⁱ —Cd1—O1—C6	128.23 (16)	C3—C4—N1—O3	179.46 (19)
N1—C5—C1—C2	0.4 (3)	C3—C4—N1—C5	-0.7 (3)
N1—C5—C1—C6	-179.34 (18)	C5—C1—C2—C3	-1.0 (3)
Cd1—O1—C6—O2	-15.1 (3)	C6—C1—C2—C3	178.8 (2)
Cd1—O1—C6—C1	165.45 (13)	N1—C4—C3—C2	0.2 (4)
C5—C1—C6—O2	10.3 (3)	C1—C2—C3—C4	0.7 (3)
C2—C1—C6—O2	-169.5 (2)		

Symmetry codes: (i) $-x+1, -y+1, -z$.

Hydrogen-bond geometry ($\text{\AA}, ^\circ$)

<i>D</i> —H \cdots <i>A</i>	<i>D</i> —H	H \cdots <i>A</i>	<i>D</i> \cdots <i>A</i>	<i>D</i> —H \cdots <i>A</i>
O1W—H1WA \cdots O2 ⁱ	0.85	1.90	2.678 (2)	151
O1W—H1WB \cdots O3 ⁱⁱ	0.85	1.86	2.697 (2)	165
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O2W—H2WB \cdots O2 ⁱⁱ	0.86	1.93	2.787 (2)	173

Symmetry codes: (i) $-x+1, -y+1, -z$; (ii) $x, -y+3/2, z+1/2$; (iii) $-x+1, y-1/2, -z+1/2$.

Fig. 1

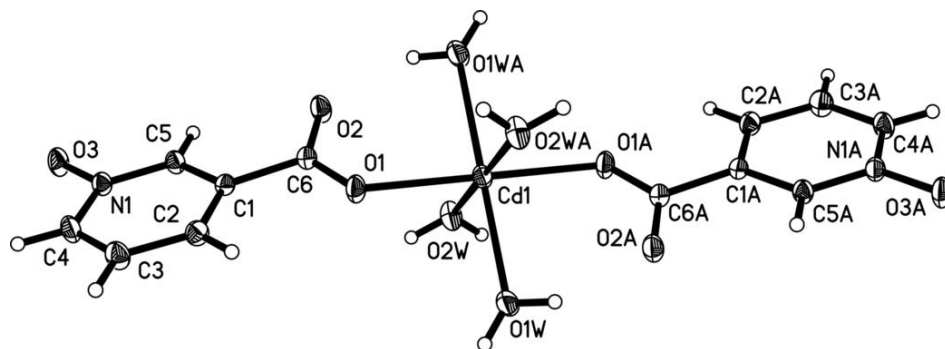


Fig. 2

