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catena-Poly[[bis(3-methyl-4-nitropyridine *N*-oxide- κ O)cadmium(II)]-di- μ -dicyanamido- $\kappa^4 N^1:N^5$]

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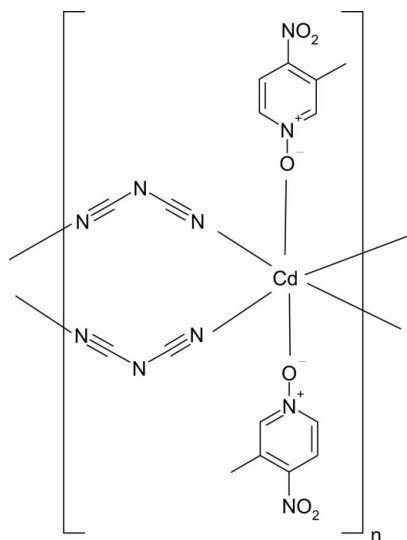
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Key indicators: single-crystal X-ray study; $T = 293$ K; mean $\sigma(\text{C}-\text{C}) = 0.004$ Å; R factor = 0.020; wR factor = 0.053; data-to-parameter ratio = 11.7.

In the title compound, $[\text{Cd}(\text{C}_2\text{N}_3)_2(\text{C}_6\text{H}_6\text{N}_2\text{O}_3)_2]_n$, the Cd^{II} ion (site symmetry $\bar{1}$) adopts a distorted trans- CdO_2N_4 octahedral environment, being coordinated by two O-bonded 3-methyl-4-nitropyridine *N*-oxide ligands and four dicyanamide (dca) anions. The bridging dca anions lead to a polymeric chain propagating in $[100]$.

Related literature

For related structures, see: Ghoshal *et al.* (2004); Wu *et al.* (2004); Schlueter *et al.* (2005).



Experimental

Crystal data

$[\text{Cd}(\text{C}_2\text{N}_3)_2(\text{C}_6\text{H}_6\text{N}_2\text{O}_3)_2]$
 $M_r = 552.76$
 Triclinic, $P\bar{1}$
 $a = 7.5472$ (8) Å
 $b = 7.5606$ (8) Å
 $c = 9.8352$ (10) Å
 $\alpha = 83.680$ (1)°
 $\beta = 68.528$ (1)°

$\gamma = 79.639$ (1)°
 $V = 513.14$ (9) Å³
 $Z = 1$
 Mo $K\alpha$ radiation
 $\mu = 1.12$ mm⁻¹
 $T = 293$ (2) K
 $0.32 \times 0.22 \times 0.18$ mm

Data collection

Bruker SMART CCD area-detector diffractometer
 Absorption correction: multi-scan (SADABS; Sheldrick, 1996)
 $T_{\min} = 0.692$, $T_{\max} = 0.817$

2770 measured reflections
 1780 independent reflections
 1764 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.015$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.020$
 $wR(F^2) = 0.053$
 $S = 1.00$
 1780 reflections

152 parameters
 H-atom parameters constrained
 $\Delta\rho_{\text{max}} = 0.34$ e Å⁻³
 $\Delta\rho_{\text{min}} = -0.38$ e Å⁻³

Table 1

Selected geometric parameters (Å, °).

Cd1—N3 ⁱ	2.288 (2)	Cd1—O3	2.3110 (19)
Cd1—N1	2.309 (2)		

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

Data collection: *SMART* (Bruker, 1998); cell refinement: *SAINTE* (Bruker, 1998); data reduction: *SAINTE*; 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*.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: HB2881).

References

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supporting information

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***catena*-Poly[[bis(3-methyl-4-nitropyridine *N*-oxide- κ O)cadmium(II)]-di- μ -dicyanamido- κ^4 N¹:N⁵]**

Rong-Min Wei

S1. Comment

The pseudohalide ligand dicyanamide (dca) has been used widely due to its polydentate character and bridging ability, yielding a variety of structures and interesting magnetic properties (Ghoshal *et al.*, 2004; Wu *et al.*, 2004; Schlueter *et al.*, 2005). As a further study of such complexes, the title Cd^{II} complex, (I), is reported in this paper (Fig. 1).

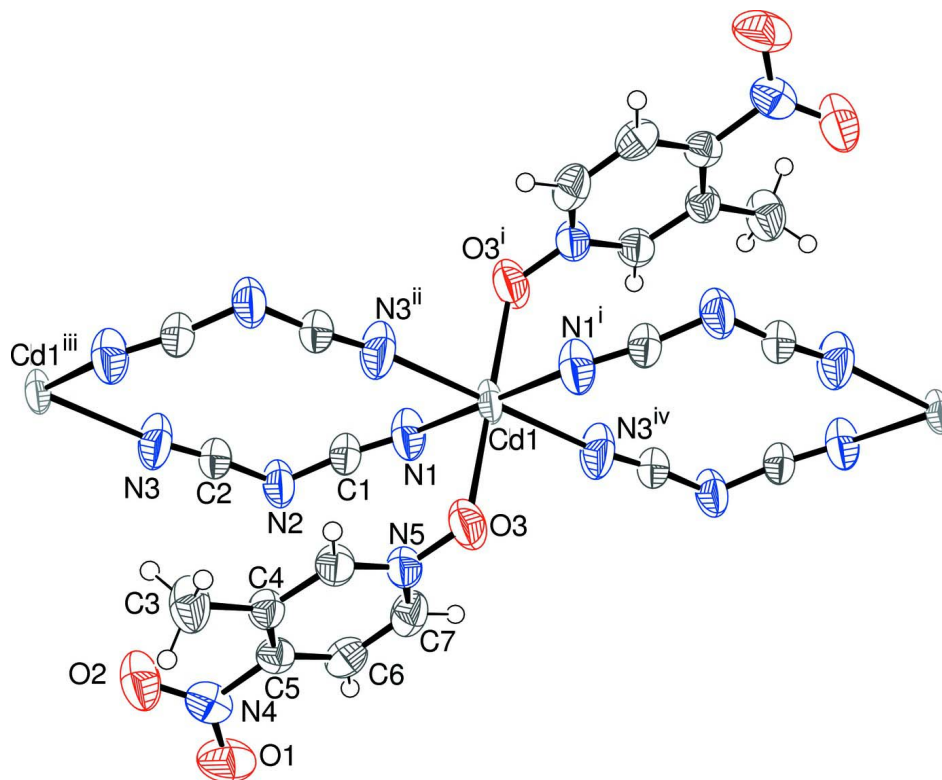
Each Cd^{II} atom exhibits a slightly distorted octahedral environment with four nitrogen atoms from dicyanamide groups in the equatorial plane, and two oxygen atoms from two *N*-oxide (pom) ligands at the axial positions (Table 1). Each Cd^{II} atom is coordinated to each other by the double bridging –NC—N—CN– ligands to form a one-dimensional chain structure, the Cd^{II}–Cd^{II} separation being equal to the value of the *a*-axis.

S2. Experimental

5 ml of a methanol solution of cadmium(II) chloride tetrahydrate (0.5 mmol, 128 mg) and 5 ml of a methanol solution of dicyanamide (1 mmol, 170 mg) were added to 10 ml of a methanol solution of POM (1 mmol, 154 mg). The mixture was stirred for 2 h and filtered. The filtrate was slowly evaporated at room temperature and red blocks of (I) were obtained after three weeks.

S3. Refinement

The hydrogen atoms were included in calculated positions (C—H = 0.93–0.96 Å) and refined as riding with $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{C})$ or $1.5U_{\text{eq}}(\text{methyl C})$.

**Figure 1**

Fragment of the infinite chain structure in (I) showing 50% displacement ellipsoids for the non-hydrogen atoms.

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

catena-Poly[[bis(3-methyl-4-nitropyridine *N*-oxide- κ O)cadmium(II)]-di- μ -dicyanamido- $\kappa^4 N^1:N^5$]

Crystal data

$[\text{Cd}(\text{C}_2\text{N}_3)_2(\text{C}_6\text{H}_6\text{N}_2\text{O}_3)_2]$

$M_r = 552.76$

Triclinic, $P\bar{1}$

Hall symbol: $-P\ 1$

$a = 7.5472$ (8) Å

$b = 7.5606$ (8) Å

$c = 9.8352$ (10) Å

$\alpha = 83.680$ (1)°

$\beta = 68.528$ (1)°

$\gamma = 79.639$ (1)°

$V = 513.14$ (9) Å³

$Z = 1$

$F(000) = 274$

$D_x = 1.789$ Mg m⁻³

$D_m = 1.789$ Mg m⁻³

D_m measured by not measured

Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å

Cell parameters from 2499 reflections

$\theta = 2.7\text{--}27.9^\circ$

$\mu = 1.12$ mm⁻¹

$T = 293$ K

Block, red

$0.32 \times 0.22 \times 0.18$ mm

Data collection

Bruker SMART CCD area-detector
diffractometer

Radiation source: fine-focus sealed tube

Graphite monochromator

φ and ω scans

Absorption correction: multi-scan

(*SADABS*; Sheldrick, 1996)

$T_{\min} = 0.692$, $T_{\max} = 0.817$

2770 measured reflections

1780 independent reflections

1764 reflections with $I > 2\sigma(I)$

$R_{\text{int}} = 0.015$

$\theta_{\max} = 25.0^\circ$, $\theta_{\min} = 2.2^\circ$

$h = -8 \rightarrow 5$

$k = -8 \rightarrow 8$

$l = -11 \rightarrow 11$

Refinement

Refinement on F^2
 Least-squares matrix: full
 $R[F^2 > 2\sigma(F^2)] = 0.020$
 $wR(F^2) = 0.053$
 $S = 1.00$
 1780 reflections
 152 parameters
 0 restraints
 Primary atom site location: structure-invariant
 direct methods

Secondary atom site location: difference Fourier
 map
 Hydrogen site location: inferred from
 neighbouring sites
 H-atom parameters constrained
 $w = 1/[\sigma^2(F_o^2) + (0.0283P)^2 + 0.2723P]$
 where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{\max} = 0.001$
 $\Delta\rho_{\max} = 0.34 \text{ e } \text{\AA}^{-3}$
 $\Delta\rho_{\min} = -0.38 \text{ e } \text{\AA}^{-3}$

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	0.5000	0.04339 (10)
O1	-0.1329 (4)	-0.2147 (3)	0.8956 (2)	0.0773 (6)
O2	-0.3397 (3)	-0.0003 (3)	0.8532 (3)	0.0777 (6)
O3	0.3605 (3)	0.3892 (3)	0.7372 (2)	0.0664 (5)
N1	0.4059 (3)	0.2818 (3)	0.4054 (3)	0.0602 (5)
N2	0.1066 (3)	0.1677 (3)	0.4552 (3)	0.0601 (6)
N3	-0.2102 (3)	0.3289 (3)	0.4816 (3)	0.0697 (7)
N4	-0.1834 (3)	-0.0583 (3)	0.8629 (2)	0.0528 (5)
N5	0.2298 (3)	0.2829 (3)	0.7670 (2)	0.0474 (4)
C1	0.2592 (3)	0.2383 (3)	0.4278 (2)	0.0417 (5)
C2	-0.0590 (3)	0.2619 (3)	0.4680 (2)	0.0424 (5)
C3	-0.3067 (4)	0.3407 (4)	0.9163 (4)	0.0644 (7)
H3A	-0.3083	0.4579	0.9460	0.097*
H3B	-0.3774	0.2705	0.9994	0.097*
H3C	-0.3652	0.3524	0.8433	0.097*
C4	-0.1028 (3)	0.2488 (3)	0.8542 (2)	0.0416 (5)
C5	-0.0425 (3)	0.0661 (3)	0.8317 (2)	0.0404 (5)
C6	0.1501 (4)	-0.0059 (3)	0.7798 (2)	0.0481 (5)
H6	0.1871	-0.1290	0.7677	0.058*
C7	0.2853 (4)	0.1047 (4)	0.7467 (3)	0.0529 (6)
H7	0.4155	0.0579	0.7101	0.063*
C8	0.0426 (3)	0.3523 (3)	0.8193 (3)	0.0466 (5)
H8	0.0092	0.4754	0.8326	0.056*

Atomic displacement parameters (\AA^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Cd1	0.02232 (12)	0.04541 (15)	0.06540 (16)	-0.00754 (9)	-0.01648 (10)	-0.00752 (10)
O1	0.1114 (18)	0.0440 (10)	0.0825 (14)	-0.0268 (11)	-0.0370 (13)	0.0064 (9)
O2	0.0531 (12)	0.0775 (14)	0.1087 (17)	-0.0215 (10)	-0.0273 (11)	-0.0157 (12)
O3	0.0540 (11)	0.0882 (14)	0.0684 (11)	-0.0361 (10)	-0.0250 (9)	0.0028 (10)
N1	0.0430 (12)	0.0582 (12)	0.0873 (16)	-0.0097 (10)	-0.0270 (11)	-0.0188 (11)
N2	0.0338 (11)	0.0471 (11)	0.1011 (17)	-0.0055 (9)	-0.0260 (11)	-0.0039 (11)
N3	0.0386 (13)	0.0663 (14)	0.109 (2)	0.0053 (11)	-0.0341 (12)	-0.0165 (13)
N4	0.0665 (15)	0.0482 (12)	0.0449 (10)	-0.0183 (10)	-0.0151 (9)	-0.0078 (8)
N5	0.0407 (11)	0.0577 (12)	0.0483 (10)	-0.0143 (9)	-0.0189 (8)	0.0006 (8)
C1	0.0333 (12)	0.0389 (11)	0.0559 (12)	-0.0019 (9)	-0.0185 (9)	-0.0114 (9)
C2	0.0361 (13)	0.0462 (11)	0.0487 (12)	-0.0063 (9)	-0.0181 (9)	-0.0071 (9)
C3	0.0403 (14)	0.0534 (14)	0.094 (2)	0.0019 (11)	-0.0176 (13)	-0.0192 (13)
C4	0.0370 (11)	0.0405 (11)	0.0470 (11)	-0.0040 (9)	-0.0147 (9)	-0.0043 (9)
C5	0.0450 (12)	0.0398 (11)	0.0375 (10)	-0.0078 (9)	-0.0152 (9)	-0.0018 (8)
C6	0.0527 (14)	0.0429 (12)	0.0475 (12)	0.0049 (10)	-0.0206 (10)	-0.0075 (9)
C7	0.0383 (12)	0.0652 (15)	0.0529 (13)	0.0042 (11)	-0.0174 (10)	-0.0087 (11)
C8	0.0446 (13)	0.0395 (11)	0.0573 (13)	-0.0061 (9)	-0.0201 (10)	-0.0028 (9)

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

Cd1—N3 ⁱ	2.288 (2)	N4—C5	1.472 (3)
Cd1—N3 ⁱⁱ	2.288 (2)	N5—C8	1.341 (3)
Cd1—N1	2.309 (2)	N5—C7	1.351 (3)
Cd1—N1 ⁱⁱⁱ	2.309 (2)	C3—C4	1.498 (3)
Cd1—O3 ⁱⁱⁱ	2.3110 (19)	C3—H3A	0.9600
Cd1—O3	2.3110 (19)	C3—H3B	0.9600
O1—N4	1.221 (3)	C3—H3C	0.9600
O2—N4	1.216 (3)	C4—C8	1.381 (3)
O3—N5	1.314 (3)	C4—C5	1.390 (3)
N1—C1	1.149 (3)	C5—C6	1.379 (3)
N2—C1	1.283 (3)	C6—C7	1.360 (4)
N2—C2	1.292 (3)	C6—H6	0.9300
N3—C2	1.128 (3)	C7—H7	0.9300
N3—Cd1 ^{iv}	2.288 (2)	C8—H8	0.9300
N3 ⁱ —Cd1—N3 ⁱⁱ	180.0	C8—N5—C7	120.7 (2)
N3 ⁱ —Cd1—N1	87.09 (8)	N1—C1—N2	172.1 (2)
N3 ⁱⁱ —Cd1—N1	92.91 (8)	N3—C2—N2	173.4 (3)
N3 ⁱ —Cd1—N1 ⁱⁱⁱ	92.91 (8)	C4—C3—H3A	109.5
N3 ⁱⁱ —Cd1—N1 ⁱⁱⁱ	87.09 (8)	C4—C3—H3B	109.5
N1—Cd1—N1 ⁱⁱⁱ	180.0	H3A—C3—H3B	109.5
N3 ⁱ —Cd1—O3 ⁱⁱⁱ	91.11 (9)	C4—C3—H3C	109.5
N3 ⁱⁱ —Cd1—O3 ⁱⁱⁱ	88.89 (9)	H3A—C3—H3C	109.5
N1—Cd1—O3 ⁱⁱⁱ	87.78 (8)	H3B—C3—H3C	109.5
N1 ⁱⁱⁱ —Cd1—O3 ⁱⁱⁱ	92.22 (8)	C8—C4—C5	115.5 (2)

N3 ⁱ —Cd1—O3	88.89 (9)	C8—C4—C3	117.9 (2)
N3 ⁱⁱ —Cd1—O3	91.11 (9)	C5—C4—C3	126.5 (2)
N1—Cd1—O3	92.22 (8)	C6—C5—C4	121.8 (2)
N1 ⁱⁱⁱ —Cd1—O3	87.78 (8)	C6—C5—N4	117.4 (2)
O3 ⁱⁱⁱ —Cd1—O3	180.0	C4—C5—N4	120.8 (2)
N5—O3—Cd1	119.76 (14)	C7—C6—C5	119.4 (2)
C1—N1—Cd1	132.98 (19)	C7—C6—H6	120.3
C1—N2—C2	123.0 (2)	C5—C6—H6	120.3
C2—N3—Cd1 ^{iv}	172.3 (2)	N5—C7—C6	119.8 (2)
O2—N4—O1	124.5 (2)	N5—C7—H7	120.1
O2—N4—C5	118.6 (2)	C6—C7—H7	120.1
O1—N4—C5	116.8 (2)	N5—C8—C4	122.8 (2)
O3—N5—C8	119.5 (2)	N5—C8—H8	118.6
O3—N5—C7	119.7 (2)	C4—C8—H8	118.6
N3 ⁱ —Cd1—O3—N5	68.54 (19)	C3—C4—C5—C6	-177.0 (2)
N3 ⁱⁱ —Cd1—O3—N5	-111.46 (19)	C8—C4—C5—N4	-179.23 (19)
N1—Cd1—O3—N5	-18.51 (19)	C3—C4—C5—N4	2.9 (4)
N1 ⁱⁱⁱ —Cd1—O3—N5	161.49 (19)	O2—N4—C5—C6	-151.9 (2)
O3 ⁱⁱⁱ —Cd1—O3—N5	-103 (100)	O1—N4—C5—C6	27.3 (3)
N3 ⁱ —Cd1—N1—C1	-34.0 (3)	O2—N4—C5—C4	28.2 (3)
N3 ⁱⁱ —Cd1—N1—C1	146.0 (3)	O1—N4—C5—C4	-152.6 (2)
N1 ⁱⁱⁱ —Cd1—N1—C1	139 (100)	C4—C5—C6—C7	-1.5 (3)
O3 ⁱⁱⁱ —Cd1—N1—C1	-125.2 (3)	N4—C5—C6—C7	178.7 (2)
O3—Cd1—N1—C1	54.8 (3)	O3—N5—C7—C6	178.9 (2)
Cd1—O3—N5—C8	-97.0 (2)	C8—N5—C7—C6	-0.2 (3)
Cd1—O3—N5—C7	84.0 (2)	C5—C6—C7—N5	1.1 (3)
Cd1—N1—C1—N2	-128.5 (19)	O3—N5—C8—C4	-179.4 (2)
C2—N2—C1—N1	174.7 (18)	C7—N5—C8—C4	-0.4 (3)
Cd1 ^{iv} —N3—C2—N2	-173.9 (15)	C5—C4—C8—N5	0.1 (3)
C1—N2—C2—N3	-180 (100)	C3—C4—C8—N5	178.1 (2)
C8—C4—C5—C6	0.9 (3)		

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