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

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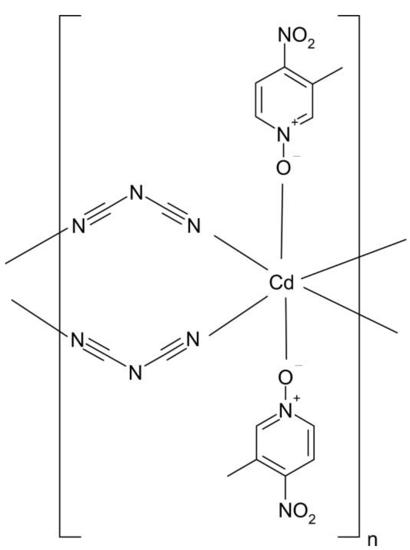
Key indicators: single-crystal X-ray study; T = 293 K; mean σ(C–C) = 0.004 Å; R factor = 0.020; wR factor = 0.053; data-to-parameter ratio = 11.7.

In the title compound, $[Cd(C_2N_3)_2(C_6H_6N_2O_3)_2]_n$, the Cd^{II} ion (site symmetry $\overline{1}$) 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

$[Cd(C_2N_3)_2(C_6H_6N_2O_3)_2]$
M_r = 552.76
Triclinic, $P\overline{1}$
a = 7.5472 (8) Å
b = 7.5606 (8) Å
c = 9.8352 (10) Å
α = 83.680 (1)°
β = 68.528 (1)°
γ = 79.639 (1)°
V = 513.14 (9) Å^3
Z = 1
Mo $K\alpha$ radiation
μ = 1.12 mm^{-1}
T = 293 (2) K
0.32 × 0.22 × 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_{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
$\Delta\rho_{max}$ = 0.34 e Å^{-3}
$\Delta\rho_{min}$ = −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: *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*.

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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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supplementary materials

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

R.-M. Wei

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···Cd separation being equal to the value of the a-axis.

Experimental

5 ml of a methanol solution of cadmium(II) chloride tetrahydrate (0.5 mmol, 128 mg) and 5 ml of a methanol sulution of dicyanamide (1 mmol, 170 mg) were aded 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 temperture and red blocks of (I) were obtained after three weeks.

Refinement

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

Figures

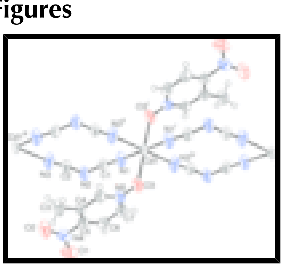


Fig. 1. Fagment of the infinite chain structure in (I) showing 50% displacement ellipsoids for the non-hydrogen atoms. Symmetry codes: (i) 1–x, 1–y, 1–z; (ii) –x, 1–y, 1–z; (iii) x–1, y, z; (iv) 1+x, y, z.

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

Crystal data

$[Cd(C_2N_3)_2(C_6H_6N_2O_3)_2]$	$Z = 1$

M_r = 552.76	F_{000} = 274
Triclinic, $P\bar{1}$	D_x = 1.789 Mg m^{-3} D_m = 1.789 Mg m^{-3} D_m measured by not measured
Hall symbol: -P 1	Mo $K\alpha$ radiation λ = 0.71073 Å
a = 7.5472 (8) Å	Cell parameters from 2499 reflections
b = 7.5606 (8) Å	θ = 2.7–27.9º
c = 9.8352 (10) Å	μ = 1.12 mm^{-1}
α = 83.680 (1)º	T = 293 (2) K
β = 68.528 (1)º	Block, red
γ = 79.639 (1)º	0.32 × 0.22 × 0.18 mm
V = 513.14 (9) Å^3	

Data collection

Bruker SMART CCD area-detector diffractometer	1780 independent reflections
Radiation source: fine-focus sealed tube	1764 reflections with $I > 2\sigma(I)$
Monochromator: graphite	R_{int} = 0.015
T = 293(2) K	θ_{max} = 25.0º
φ and ω scans	θ_{min} = 2.2º
Absorption correction: multi-scan (SADABS; Sheldrick, 1996)	$h = -8 \rightarrow 5$
T_{min} = 0.692, T_{max} = 0.817	$k = -8 \rightarrow 8$
2770 measured reflections	$l = -11 \rightarrow 11$

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)]$ = 0.020	H-atom parameters constrained
$wR(F^2)$ = 0.053	$w = 1/[\sigma^2(F_o^2) + (0.0283P)^2 + 0.2723P]$ where $P = (F_o^2 + 2F_c^2)/3$
S = 1.00	$(\Delta/\sigma)_{max}$ = 0.001
1780 reflections	$\Delta\rho_{max}$ = 0.34 e Å^{-3}
152 parameters	$\Delta\rho_{min}$ = −0.38 e Å^{-3}
Primary atom site location: structure-invariant direct methods	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 (Å^2)

	x	y	z	U_{iso}*/U_{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 (Å^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)

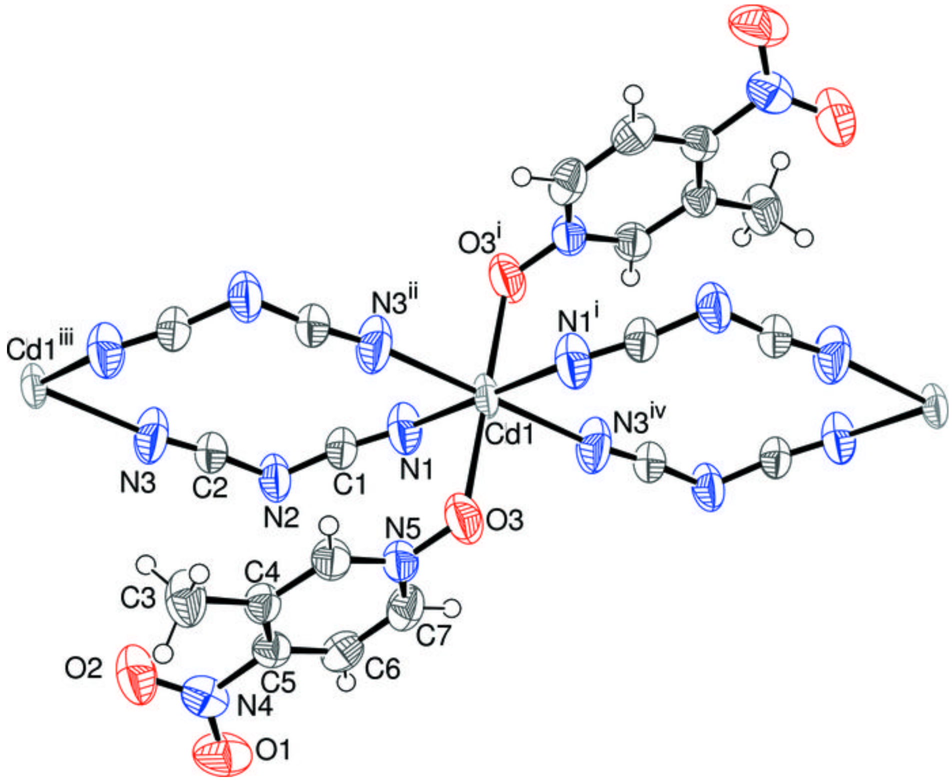
Geometric parameters (Å, °)

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.

Fig. 1


O3i
N3ii
N1i
Cd1iii
N3iv
Cd1
N3
C2
N2
C1
N1
N5
O3
C4
C3
C7
C6
C5
O2
N4
O1