

catena-Poly[[bis(dimethylformamide- κ O)-cadmium]-bis(μ -4-nitrophenylcyanamido- κ^2 N¹:N³)]

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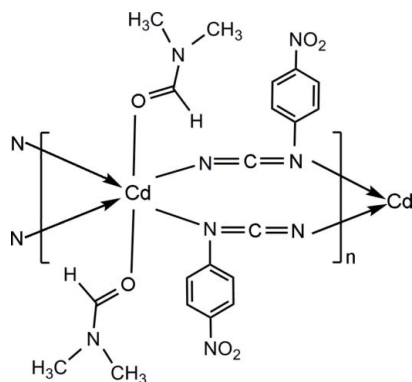
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Key indicators: single-crystal X-ray study; $T = 298$ K; mean $\sigma(\text{C}-\text{C}) = 0.005$ Å; R factor = 0.044; wR factor = 0.114; data-to-parameter ratio = 19.4.

In the title coordination polymer, $[\text{Cd}(\text{C}_7\text{H}_4\text{N}_3\text{O}_2)_2(\text{C}_3\text{H}_7\text{NO})_2]_n$, the Cd^{II} atom, lying on an inversion center, is six-coordinated in a distorted N_4O_2 octahedral geometry. The N atoms of the 4-nitrophenylcyanamide anions form the equatorial plane and the O atoms of the dimethylformamide molecules occupy the axial positions. The anions act as bridging ligands, connecting the Cd atoms into a one-dimensional coordination polymer along [100].

Related literature

For background to phenylcyanamide ligands and their complexes, see: Crutchley (2001). For polynuclear complexes of phenylcyanamide ligands, see: Ainscough *et al.* (1991); Chiniforoshan *et al.* (2009, 2010); Escuer *et al.* (2004). For the preparation of 4-nitro-phenylcyanamide used in the synthesis of the title compound, see: Crutchley & Naklicki (1989).



Experimental

Crystal data

$[\text{Cd}(\text{C}_7\text{H}_4\text{N}_3\text{O}_2)_2(\text{C}_3\text{H}_7\text{NO})_2]$	$\gamma = 84.28$ (3)°
$M_r = 582.87$	$V = 586.7$ (2) Å ³
Triclinic, $P\bar{1}$	$Z = 1$
$a = 5.6070$ (11) Å	Mo $K\alpha$ radiation
$b = 9.811$ (2) Å	$\mu = 0.98$ mm ⁻¹
$c = 11.679$ (2) Å	$T = 298$ K
$\alpha = 67.44$ (3)°	$0.45 \times 0.10 \times 0.08$ mm
$\beta = 81.93$ (3)°	

Data collection

Stoe IPDS 2T diffractometer	6589 measured reflections
Absorption correction: numerical (<i>X-SHAPE</i> and <i>X-RED32</i> ; Stoe & Cie, 2002)	3150 independent reflections
$T_{\min} = 0.887$, $T_{\max} = 0.923$	3038 reflections with $I > 2\sigma(I)$
	$R_{\text{int}} = 0.066$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.044$	162 parameters
$wR(F^2) = 0.114$	H-atom parameters constrained
$S = 1.11$	$\Delta\rho_{\text{max}} = 0.82$ e Å ⁻³
3150 reflections	$\Delta\rho_{\text{min}} = -0.89$ e Å ⁻³

Table 1

Selected bond lengths (Å).

Cd1—N1 ⁱ	2.287 (3)	Cd1—N2	2.383 (3)
Cd1—O3	2.347 (3)		

Symmetry code: (i) $-x + 1, -y + 2, -z$.

Data collection: *X-AREA* (Stoe & Cie, 2002); cell refinement: *X-AREA*; data reduction: *X-AREA*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *ORTEP-3* (Farrugia, 1997); software used to prepare material for publication: *WinGX* (Farrugia, 1999).

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: HY2496).

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supplementary materials

Acta Cryst. (2012). E68, m232 [doi:10.1107/S1600536812001924]

catena-Poly[[bis(dimethylformamide- κ O)cadmium]-bis(μ -4-nitrophenyl-cyanamido- κ^2 N¹:N³)]**Hossein Chiniforoshan, Mehdi Jazestani and Behrouz Notash****Comment**

Phenylcyanamide ligands can act as monodentate, bidentate and also as bridging ligands (Crutchley, 2001). In the bridging mode, the cyanamido group (NCN) is coordinated in an end-to-end mode, forming polynuclear complexes (Ainscough *et al.*, 1991; Chiniforoshan *et al.*, 2009, 2010; Escuer *et al.*, 2004).

Following our work with this family of ligands, we report here the synthesis and crystal structure of a cadmium(II) coordination polymer of 4-nitro-phenylcyanamide ligand (Crutchley & Naklicki, 1989). The asymmetric unit of the title compound is shown in Fig. 1. In the title compound, the Cd^{II} atom lies on an inversion center and has a distorted octahedral geometry (Fig. 2, Table 1). The coordination environment consists of four N atoms from 4-nitro-phenyl-cyanamide ligands in the equatorial plane and two O atoms from DMF molecules in the axial positions. The one-dimensional structure of the title compound is shown in Fig. 2.

Experimental

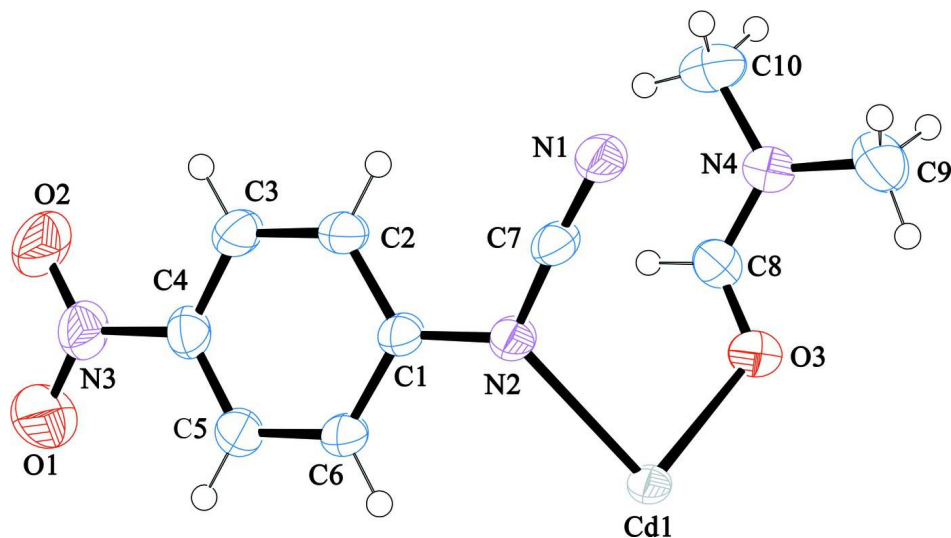
4-Nitrophenylcyanamide (Crutchley & Naklicki, 1989) (0.163 g, 1 mmol) was dissolved in methanol (25 ml) and was added slowly to a solution of cadmium(II) acetate (0.133 g, 0.5 mmol) in methanol (25 ml). The mixture was stirred for 3 hrs. The resulting solid was filtered off. Yellow needles of the title compound were obtained by n-hexane diffusion into a DMF solution of the title compound after 4 weeks.

Refinement

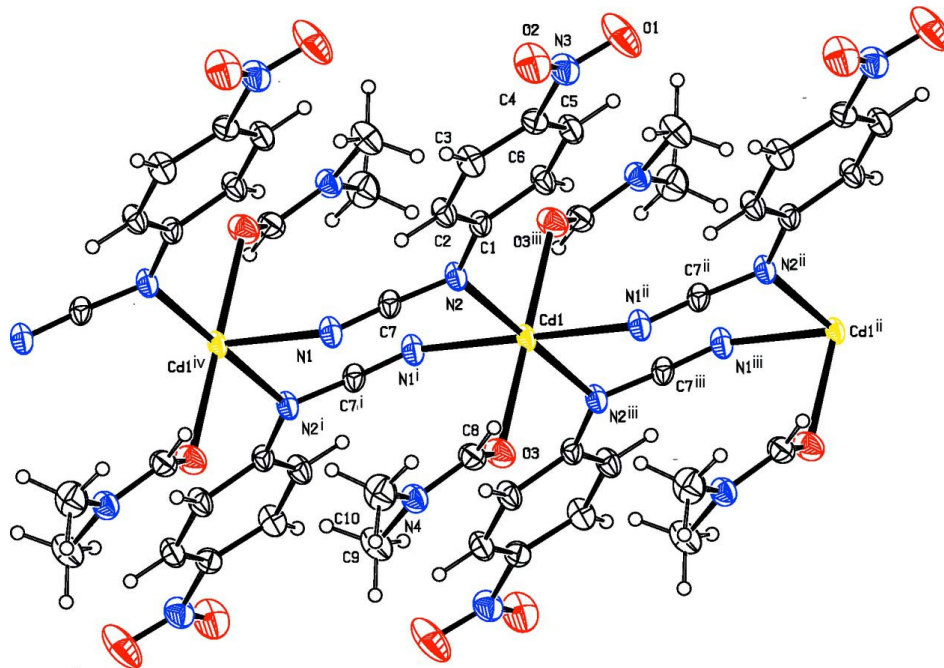
H atoms were positioned geometrically and refined as riding atoms, with C—H = 0.93 (CH) and 0.96 (CH₃) Å and with $U_{\text{iso}}(\text{H}) = 1.2(1.5 \text{ for methyl})U_{\text{eq}}(\text{C})$.

Computing details

Data collection: *X-AREA* (Stoe & Cie, 2002); cell refinement: *X-AREA* (Stoe & Cie, 2002); data reduction: *X-AREA* (Stoe & Cie, 2002); program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *ORTEP-3* (Farrugia, 1997); software used to prepare material for publication: *WinGX* (Farrugia, 1999).


Figure 1

The asymmetric unit of the title compound with displacement ellipsoids drawn at 50% probability level.


Figure 2

The one-dimensional polymeric structure of the title compound. [Symmetry codes: (i) 1-x, 2-y, -z; (ii) -1+x, y, z; (iii) -x, 2-y, -z; (iv) 1+x, y, z.]

catena-Poly[[bis(dimethylformamide- κ O)cadmium]-bis(μ -4-nitrophenylcyanamido- κ^2 N¹:N³)]

Crystal data

[Cd(C₇H₄N₃O₂)₂(C₃H₇NO)₂]

$M_r = 582.87$

Triclinic, $P\bar{1}$

Hall symbol: -P 1

$a = 5.6070$ (11) Å

$b = 9.811$ (2) Å

$c = 11.679$ (2) Å

$\alpha = 67.44$ (3)°

$\beta = 81.93 (3)^\circ$
 $\gamma = 84.28 (3)^\circ$
 $V = 586.7 (2) \text{ \AA}^3$
 $Z = 1$
 $F(000) = 294$
 $D_x = 1.650 \text{ Mg m}^{-3}$
 Mo $K\alpha$ radiation, $\lambda = 0.71073 \text{ \AA}$

Cell parameters from 3150 reflections
 $\theta = 2.3\text{--}29.2^\circ$
 $\mu = 0.98 \text{ mm}^{-1}$
 $T = 298 \text{ K}$
 Needle, yellow
 $0.45 \times 0.10 \times 0.08 \text{ mm}$

Data collection

Stoe IPDS 2T
 diffractometer
 Radiation source: fine-focus sealed tube
 Graphite monochromator
 ω scans
 Absorption correction: numerical
 (*X-SHAPE* and *X-RED32*; Stoe & Cie, 2002)
 $T_{\min} = 0.887$, $T_{\max} = 0.923$

6589 measured reflections
 3150 independent reflections
 3038 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.066$
 $\theta_{\max} = 29.2^\circ$, $\theta_{\min} = 2.3^\circ$
 $h = -7 \rightarrow 7$
 $k = -13 \rightarrow 13$
 $l = -15 \rightarrow 16$

Refinement

Refinement on F^2
 Least-squares matrix: full
 $R[F^2 > 2\sigma(F^2)] = 0.044$
 $wR(F^2) = 0.114$
 $S = 1.11$
 3150 reflections
 162 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
 $w = 1/[\sigma^2(F_o^2) + (0.0672P)^2 + 0.308P]$
 where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{\max} < 0.001$
 $\Delta\rho_{\max} = 0.82 \text{ e \AA}^{-3}$
 $\Delta\rho_{\min} = -0.89 \text{ e \AA}^{-3}$

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)

	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{iso}}^*/U_{\text{eq}}$
Cd1	0.0000	1.0000	0.0000	0.03092 (12)
O1	-0.1591 (9)	0.2034 (5)	0.4718 (4)	0.0897 (14)
O2	0.1354 (7)	0.1984 (4)	0.5719 (3)	0.0705 (10)
O3	0.0654 (5)	1.1593 (3)	0.0984 (3)	0.0493 (6)
N1	0.6524 (5)	0.9163 (3)	0.1262 (3)	0.0404 (6)
N2	0.2627 (5)	0.8176 (3)	0.1293 (3)	0.0362 (5)
N3	0.0180 (6)	0.2570 (4)	0.4842 (3)	0.0511 (8)
N4	0.3441 (5)	1.1798 (4)	0.2129 (3)	0.0417 (6)
C1	0.2102 (5)	0.6769 (3)	0.2158 (3)	0.0326 (6)
C2	0.3446 (6)	0.6018 (4)	0.3172 (3)	0.0392 (7)

H2	0.4765	0.6457	0.3264	0.047*
C3	0.2838 (7)	0.4640 (4)	0.4033 (3)	0.0437 (7)
H3	0.3748	0.4149	0.4694	0.052*
C4	0.0864 (6)	0.4002 (4)	0.3899 (3)	0.0387 (7)
C5	-0.0458 (6)	0.4682 (4)	0.2903 (4)	0.0446 (8)
H5	-0.1761	0.4225	0.2818	0.053*
C6	0.0172 (6)	0.6057 (4)	0.2026 (4)	0.0413 (7)
H6	-0.0697	0.6511	0.1344	0.050*
C7	0.4701 (5)	0.8656 (4)	0.1296 (3)	0.0338 (6)
C8	0.1668 (6)	1.1147 (4)	0.1947 (4)	0.0412 (7)
H8	0.1137	1.0288	0.2590	0.049*
C9	0.4414 (9)	1.3125 (5)	0.1153 (5)	0.0584 (10)
H9A	0.6050	1.2913	0.0873	0.088*
H9B	0.4371	1.3892	0.1476	0.088*
H9C	0.3461	1.3444	0.0466	0.088*
C10	0.4716 (9)	1.1095 (6)	0.3235 (5)	0.0598 (11)
H10A	0.3882	1.0246	0.3806	0.090*
H10B	0.4778	1.1784	0.3631	0.090*
H10C	0.6327	1.0793	0.2994	0.090*

Atomic displacement parameters (\AA^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Cd1	0.01969 (14)	0.03075 (16)	0.03821 (18)	-0.00441 (9)	-0.00930 (10)	-0.00556 (12)
O1	0.092 (3)	0.066 (2)	0.089 (3)	-0.048 (2)	-0.027 (2)	0.011 (2)
O2	0.073 (2)	0.0528 (18)	0.0584 (19)	-0.0116 (16)	-0.0145 (16)	0.0133 (15)
O3	0.0510 (14)	0.0441 (14)	0.0569 (16)	-0.0031 (11)	-0.0226 (12)	-0.0175 (12)
N1	0.0274 (11)	0.0406 (14)	0.0439 (15)	-0.0065 (10)	-0.0072 (10)	-0.0035 (12)
N2	0.0253 (11)	0.0335 (13)	0.0425 (14)	-0.0060 (9)	-0.0082 (10)	-0.0037 (11)
N3	0.0499 (17)	0.0391 (16)	0.0529 (18)	-0.0092 (13)	-0.0037 (14)	-0.0038 (14)
N4	0.0381 (13)	0.0435 (15)	0.0451 (15)	-0.0046 (11)	-0.0100 (11)	-0.0159 (13)
C1	0.0256 (12)	0.0321 (14)	0.0362 (14)	-0.0014 (10)	-0.0073 (10)	-0.0072 (11)
C2	0.0347 (14)	0.0401 (16)	0.0398 (16)	-0.0078 (12)	-0.0126 (12)	-0.0075 (13)
C3	0.0431 (17)	0.0400 (17)	0.0389 (17)	-0.0069 (13)	-0.0139 (13)	-0.0003 (13)
C4	0.0380 (15)	0.0302 (14)	0.0436 (17)	-0.0045 (11)	-0.0024 (13)	-0.0092 (13)
C5	0.0341 (15)	0.0359 (16)	0.059 (2)	-0.0078 (12)	-0.0133 (14)	-0.0081 (15)
C6	0.0332 (14)	0.0350 (15)	0.0507 (19)	-0.0046 (11)	-0.0181 (13)	-0.0052 (14)
C7	0.0256 (12)	0.0349 (14)	0.0340 (14)	0.0007 (10)	-0.0073 (10)	-0.0045 (11)
C8	0.0410 (16)	0.0386 (16)	0.0467 (18)	-0.0055 (13)	-0.0073 (13)	-0.0172 (14)
C9	0.059 (2)	0.054 (2)	0.060 (2)	-0.0214 (19)	-0.0045 (19)	-0.015 (2)
C10	0.056 (2)	0.072 (3)	0.056 (2)	0.001 (2)	-0.0242 (19)	-0.024 (2)

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

Cd1—N1 ⁱ	2.287 (3)	C2—C3	1.381 (5)
Cd1—O3	2.347 (3)	C2—H2	0.9300
Cd1—N2	2.383 (3)	C3—C4	1.380 (5)
O1—N3	1.219 (5)	C3—H3	0.9300
O2—N3	1.214 (5)	C4—C5	1.377 (5)
O3—C8	1.238 (5)	C5—C6	1.388 (5)

N1—C7	1.170 (4)	C5—H5	0.9300
N1—Cd1 ⁱⁱ	2.287 (3)	C6—H6	0.9300
N2—C7	1.299 (4)	C8—H8	0.9300
N2—C1	1.392 (4)	C9—H9A	0.9600
N3—C4	1.461 (4)	C9—H9B	0.9600
N4—C8	1.316 (4)	C9—H9C	0.9600
N4—C9	1.456 (5)	C10—H10A	0.9600
N4—C10	1.460 (5)	C10—H10B	0.9600
C1—C6	1.401 (4)	C10—H10C	0.9600
C1—C2	1.409 (4)		
N1 ⁱ —Cd1—N1 ⁱⁱⁱ	180.000 (1)	C3—C2—H2	119.5
N1 ⁱ —Cd1—O3	86.19 (12)	C1—C2—H2	119.5
N1 ⁱⁱⁱ —Cd1—O3	93.81 (12)	C4—C3—C2	119.2 (3)
N1 ⁱ —Cd1—O3 ^{iv}	93.81 (12)	C4—C3—H3	120.4
N1 ⁱⁱⁱ —Cd1—O3 ^{iv}	86.19 (12)	C2—C3—H3	120.4
O3—Cd1—O3 ^{iv}	180.00 (10)	C5—C4—C3	121.5 (3)
N1 ⁱ —Cd1—N2 ^{iv}	95.72 (10)	C5—C4—N3	119.8 (3)
N1 ⁱⁱⁱ —Cd1—N2 ^{iv}	84.28 (10)	C3—C4—N3	118.7 (3)
O3—Cd1—N2 ^{iv}	90.74 (10)	C4—C5—C6	119.4 (3)
O3 ^{iv} —Cd1—N2 ^{iv}	89.26 (10)	C4—C5—H5	120.3
N1 ⁱ —Cd1—N2	84.28 (10)	C6—C5—H5	120.3
N1 ⁱⁱⁱ —Cd1—N2	95.72 (10)	C5—C6—C1	120.8 (3)
O3—Cd1—N2	89.26 (10)	C5—C6—H6	119.6
O3 ^{iv} —Cd1—N2	90.74 (10)	C1—C6—H6	119.6
N2 ^{iv} —Cd1—N2	180.0	N1—C7—N2	176.4 (3)
C8—O3—Cd1	121.5 (2)	O3—C8—N4	124.9 (4)
C7—N1—Cd1 ⁱⁱ	142.5 (3)	O3—C8—H8	117.6
C7—N2—C1	116.9 (3)	N4—C8—H8	117.6
C7—N2—Cd1	113.9 (2)	N4—C9—H9A	109.5
C1—N2—Cd1	128.50 (19)	N4—C9—H9B	109.5
O2—N3—O1	123.1 (4)	H9A—C9—H9B	109.5
O2—N3—C4	119.1 (3)	N4—C9—H9C	109.5
O1—N3—C4	117.7 (4)	H9A—C9—H9C	109.5
C8—N4—C9	120.7 (3)	H9B—C9—H9C	109.5
C8—N4—C10	120.8 (4)	N4—C10—H10A	109.5
C9—N4—C10	117.9 (4)	N4—C10—H10B	109.5
N2—C1—C6	119.5 (3)	H10A—C10—H10B	109.5
N2—C1—C2	122.5 (3)	N4—C10—H10C	109.5
C6—C1—C2	118.0 (3)	H10A—C10—H10C	109.5
C3—C2—C1	121.1 (3)	H10B—C10—H10C	109.5
N1 ⁱ —Cd1—O3—C8	-96.1 (3)	C6—C1—C2—C3	-1.9 (5)
N1 ⁱⁱⁱ —Cd1—O3—C8	83.9 (3)	C1—C2—C3—C4	-0.7 (6)
N2 ^{iv} —Cd1—O3—C8	168.2 (3)	C2—C3—C4—C5	2.5 (6)
N2—Cd1—O3—C8	-11.8 (3)	C2—C3—C4—N3	-177.6 (4)
N1 ⁱ —Cd1—N2—C7	35.1 (3)	O2—N3—C4—C5	-179.7 (4)
N1 ⁱⁱⁱ —Cd1—N2—C7	-144.9 (3)	O1—N3—C4—C5	-1.2 (6)
O3—Cd1—N2—C7	-51.2 (3)	O2—N3—C4—C3	0.4 (6)

O3 ^{iv} —Cd1—N2—C7	128.8 (3)	O1—N3—C4—C3	178.9 (5)
N1 ⁱ —Cd1—N2—C1	-155.1 (3)	C3—C4—C5—C6	-1.5 (6)
N1 ⁱⁱⁱ —Cd1—N2—C1	24.9 (3)	N3—C4—C5—C6	178.6 (4)
O3—Cd1—N2—C1	118.6 (3)	C4—C5—C6—C1	-1.2 (6)
O3 ^{iv} —Cd1—N2—C1	-61.4 (3)	N2—C1—C6—C5	-177.2 (3)
C7—N2—C1—C6	-165.8 (3)	C2—C1—C6—C5	2.9 (5)
Cd1—N2—C1—C6	24.6 (5)	Cd1—O3—C8—N4	131.9 (3)
C7—N2—C1—C2	14.0 (5)	C9—N4—C8—O3	-1.7 (6)
Cd1—N2—C1—C2	-155.5 (3)	C10—N4—C8—O3	-172.6 (4)
N2—C1—C2—C3	178.2 (3)		

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