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Diaquabis(9-oxo-4,5-diazafluoren-3-olato- κ^2N^4,O^3)cadmium(II)

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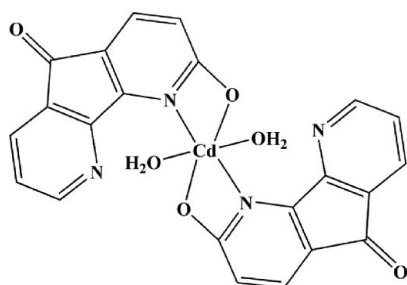
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Key indicators: single-crystal X-ray study; $T = 293$ K; mean $\sigma(C-C) = 0.004$ Å; R factor = 0.029; wR factor = 0.082; data-to-parameter ratio = 15.2.

The title compound, $[Cd(C_{11}H_5N_2O_2)_2(H_2O)_2]$, is a mononuclear complex consisting of a Cd^{II} atom, two 3-hydroxy-4,5-diazafluoren-9-one ligands and two coordinated water molecules. The Cd^{II} atom, lying on a twofold axis, displays a distorted octahedral coordination. Adjacent molecules are linked by $O-H\cdots O$ hydrogen bonds and $\pi-\pi$ interactions [centroid-centroid distance = $3.84(1)$ Å], leading to a one-dimensional chain. Weak $C-H\cdots O$ hydrogen bonds connect the chains into a two-dimensional supramolecular structure.

Related literature

For related literature, see: Li *et al.* (2006); Rillema *et al.* (2007); Terhshansy *et al.* (2006); Zhao *et al.* (2006).



Experimental

Crystal data

 $[Cd(C_{11}H_5N_2O_2)_2(H_2O)_2]$ $M_r = 542.77$ Monoclinic, $C2/c$ $a = 28.190(5)$ Å $b = 5.572(4)$ Å $c = 13.933(5)$ Å $\beta = 110.622(5)^\circ$ $V = 2048.3(17)$ Å³ $Z = 4$ Mo $K\alpha$ radiation $\mu = 1.12$ mm⁻¹ $T = 293(2)$ K $0.26 \times 0.21 \times 0.17$ mm

Data collection

Rigaku R-Axis RAPID

diffractometer

Absorption correction: multi-scan

(ABSCOR; Higashi, 1995)

 $T_{\min} = 0.760$, $T_{\max} = 0.833$

5972 measured reflections

2406 independent reflections

1965 reflections with $I > 2\sigma(I)$ $R_{\text{int}} = 0.021$

Refinement

 $R[F^2 > 2\sigma(F^2)] = 0.029$ $wR(F^2) = 0.082$ $S = 1.02$

2406 reflections

158 parameters

2 restraints

H atoms treated by a mixture of independent and constrained refinement

 $\Delta\rho_{\max} = 0.62$ e Å⁻³ $\Delta\rho_{\min} = -0.36$ e Å⁻³

Table 1

Selected geometric parameters (Å, °).

Cd1—O1	2.233 (2)	Cd1—O2	2.410 (2)
Cd1—N1	2.3217 (19)		
O1—Cd1—O1 ⁱ	101.59 (13)	O1 ⁱ —Cd1—O2	148.19 (7)
O1—Cd1—N1 ⁱ	92.77 (7)	N1 ⁱ —Cd1—O2	101.27 (7)
O1—Cd1—N1	105.03 (8)	N1—Cd1—O2	56.38 (7)
N1 ⁱ —Cd1—N1	151.82 (11)	O2—Cd1—O2 ⁱ	85.23 (11)
O1—Cd1—O2	94.62 (9)		

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

Table 2

Hydrogen-bond geometry (Å, °).

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
O1—H1A \cdots N2 ⁱ	0.82 (2)	2.02 (2)	2.832 (3)	172 (3)
O1—H1B \cdots O2 ⁱⁱ	0.83 (2)	1.86 (2)	2.686 (3)	170 (4)
C3—H3 \cdots O3 ⁱⁱⁱ	0.93	2.48	3.351 (5)	156

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

Data collection: *PROCESS-AUTO* (Rigaku, 1998); cell refinement: *PROCESS-AUTO*; data reduction: *PROCESS-AUTO*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *SHELXTL-Plus* (Siemens, 1990); software used to prepare material for publication: *SHELXL97*.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: HY2109).

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supplementary materials

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Diaquabis(9-oxo-4,5-diazafluoren-3-olato- κ^2N^4,O^3)cadmium(II)

Y.-H. Zhao, X. Yuan, W.-C. Qin, L.-X. Sheng and Y.-Z. Ding

Comment

The structures of 4,5-diazafluoren-9-one and its metal complexes have been reported (Li *et al.*, 2006; Rillema *et al.*, 2007; Tershansy *et al.*, 2006; Zhao *et al.*, 2006). However, no studies of its derivative, 3-hydroxyl-4,5-diazafluoren-9-one, and any metal complexes of the ligand are known to date. In this paper, we present the crystal structure of the title compound, $[\text{Cd}(\text{C}_{11}\text{H}_5\text{N}_2\text{O}_2)_2(\text{H}_2\text{O})_2]$.

The title compound is a neutral mononuclear complex. The Cd^{II} atom is six-coordinate and exhibits a distorted octahedral coordination geometry, defined by two N atoms and two O atoms from two 3-hydroxyl-4,5-diazafluoren-9-one ligands and two O atoms from two aqua ligands (Fig. 1). The Cd—O distances are in the range of 2.233 (2)–2.410 (2) Å and the Cd—N length is 2.322 (2) Å (Table 1). Adjacent molecules are linked by O—H \cdots O hydrogen bonds and π – π interactions [centroid–centroid distance 3.84 (1) Å], leading to a one-dimensional chain. Weak C—H \cdots O hydrogen bonds connect the chains into a two-dimensional supramolecular structure (Fig. 2; Table 2).

Experimental

Cadmium(II) acetate dihydrate (0.080 g, 0.3 mmol), 3-hydroxyl-4,5-diazafluoren-9-one (0.040 g, 0.2 mmol), sodium hydroxide (0.024 g, 0.4 mmol) and water (14 ml) were placed in a 23 ml Teflon-lined autoclave, which was heated at 423 K for 3 d. After cooling slowly to room temperature at a rate of 10 K h $^{-1}$, colorless crystals of the title compound were obtained. Analysis calculated for $\text{C}_{22}\text{H}_{14}\text{CdN}_4\text{O}_6$: C 48.64, H 2.58, N 10.32%; found: C 48.51, H 2.67, N 10.42%.

Refinement

H atoms on C atoms were positioned geometrically and refined as riding, with C—H = 0.93 Å and $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{C})$. H atoms of water molecule were located on a difference Fourier map and refined isotropically with a restraint of O—H = 0.82 (1) Å.

Figures

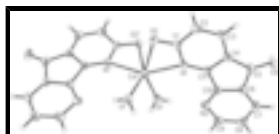


Fig. 1. Molecular structure of the title compound. Displacement ellipsoids are drawn at the 30% probability level. [Symmetry code: (i) $-x + 2, y, -z + 1/2$.]

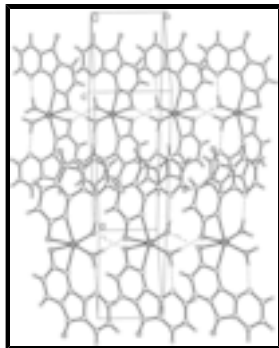


Fig. 2. A packing diagram for the two-dimensional supramolecular structure *via* hydrogen bonds (dashed lines).

Diaquabis(9-oxo-4,5-diazafluoren-3-olato- κ^2N^4,O^3)cadmium(II)

Crystal data

[Cd(C₁₁H₅N₂O₂)₂(H₂O)₂]

$M_r = 542.77$

Monoclinic, $C2/c$

Hall symbol: $-C\ 2yc$

$a = 28.190\ (5)\ \text{\AA}$

$b = 5.572\ (4)\ \text{\AA}$

$c = 13.933\ (5)\ \text{\AA}$

$\beta = 110.622\ (5)^\circ$

$V = 2048.3\ (17)\ \text{\AA}^3$

$Z = 4$

$F_{000} = 1080$

$D_x = 1.760\ \text{Mg m}^{-3}$

Mo $K\alpha$ radiation

$\lambda = 0.71073\ \text{\AA}$

Cell parameters from 4465 reflections

$\theta = 3.0\text{--}28.3^\circ$

$\mu = 1.12\ \text{mm}^{-1}$

$T = 293\ (2)\ \text{K}$

Block, colorless

$0.26 \times 0.21 \times 0.17\ \text{mm}$

Data collection

Rigaku R-AXIS RAPID
diffractometer

Radiation source: rotation anode

Monochromator: graphite

$T = 293\ (2)\ \text{K}$

ω scans

Absorption correction: multi-scan
(ABSCOR; Higashi, 1995)

$T_{\min} = 0.760$, $T_{\max} = 0.833$

5972 measured reflections

2406 independent reflections

1965 reflections with $I > 2\sigma(I)$

$R_{\text{int}} = 0.021$

$\theta_{\max} = 28.3^\circ$

$\theta_{\min} = 3.0^\circ$

$h = -26 \rightarrow 37$

$k = -7 \rightarrow 7$

$l = -18 \rightarrow 12$

Refinement

Refinement on F^2

Least-squares matrix: full

$R[F^2 > 2\sigma(F^2)] = 0.029$

$wR(F^2) = 0.082$

Secondary atom site location: difference Fourier map

Hydrogen site location: inferred from neighbouring sites

H atoms treated by a mixture of independent and constrained refinement

$w = 1/[\sigma^2(F_o^2) + (0.0433P)^2 + 1.5483P]$

$S = 1.02$

2406 reflections

158 parameters

2 restraints

Primary atom site location: structure-invariant direct methods

where $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{\max} < 0.001$

$\Delta\rho_{\max} = 0.62 \text{ e } \text{\AA}^{-3}$

$\Delta\rho_{\min} = -0.36 \text{ e } \text{\AA}^{-3}$

Extinction correction: none

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	1.0000	0.24267 (5)	0.2500	0.04153 (11)
N1	0.91692 (7)	0.1412 (4)	0.15373 (15)	0.0337 (4)
N2	0.90481 (8)	0.5376 (4)	-0.00770 (16)	0.0400 (5)
O2	0.95401 (7)	-0.0756 (3)	0.29552 (14)	0.0455 (4)
O3	0.75172 (7)	0.0883 (4)	-0.09790 (15)	0.0553 (5)
C1	0.91486 (9)	-0.0461 (4)	0.21600 (18)	0.0358 (5)
C2	0.87029 (11)	-0.1903 (5)	0.1904 (2)	0.0430 (6)
H2	0.8695	-0.3203	0.2317	0.052*
C3	0.82874 (10)	-0.1395 (5)	0.1061 (2)	0.0423 (6)
H3	0.7995	-0.2322	0.0897	0.051*
C4	0.83130 (9)	0.0555 (5)	0.04508 (18)	0.0366 (5)
C5	0.79449 (9)	0.1567 (5)	-0.0489 (2)	0.0404 (6)
C6	0.82108 (9)	0.3654 (5)	-0.07684 (18)	0.0377 (5)
C7	0.80569 (10)	0.5278 (5)	-0.1560 (2)	0.0470 (6)
H7	0.7731	0.5249	-0.2045	0.056*
C8	0.84113 (12)	0.6963 (5)	-0.1601 (2)	0.0501 (7)
H8	0.8325	0.8102	-0.2122	0.060*
C9	0.88918 (12)	0.6950 (5)	-0.0868 (2)	0.0460 (7)
H9	0.9122	0.8091	-0.0920	0.055*
C10	0.87021 (9)	0.3786 (4)	-0.00494 (17)	0.0334 (5)
C11	0.87612 (9)	0.1865 (4)	0.07194 (19)	0.0327 (5)
O1	0.99406 (8)	0.4960 (4)	0.36946 (16)	0.0505 (5)
H1A	1.0223 (8)	0.519 (6)	0.412 (2)	0.065 (10)*
H1B	0.9809 (13)	0.630 (4)	0.353 (3)	0.067 (11)*

Atomic displacement parameters (\AA^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Cd1	0.02980 (15)	0.03837 (17)	0.04841 (18)	0.000	0.00383 (11)	0.000
N1	0.0281 (10)	0.0314 (10)	0.0377 (11)	-0.0028 (8)	0.0066 (8)	0.0013 (9)
N2	0.0387 (11)	0.0401 (12)	0.0400 (11)	-0.0032 (9)	0.0125 (9)	0.0036 (9)
O2	0.0427 (10)	0.0407 (10)	0.0454 (10)	0.0028 (8)	0.0058 (8)	0.0076 (8)
O3	0.0343 (10)	0.0707 (14)	0.0495 (11)	-0.0142 (9)	0.0005 (8)	-0.0081 (10)
C1	0.0369 (12)	0.0317 (12)	0.0382 (13)	0.0023 (10)	0.0124 (10)	0.0002 (10)
C2	0.0489 (16)	0.0348 (13)	0.0490 (15)	-0.0056 (11)	0.0218 (13)	0.0017 (11)
C3	0.0385 (14)	0.0400 (14)	0.0494 (15)	-0.0123 (11)	0.0168 (12)	-0.0068 (12)
C4	0.0296 (11)	0.0403 (13)	0.0385 (13)	-0.0051 (10)	0.0103 (10)	-0.0061 (11)

supplementary materials

C5	0.0331 (13)	0.0464 (14)	0.0396 (13)	-0.0031 (11)	0.0100 (11)	-0.0089 (12)
C6	0.0317 (12)	0.0444 (15)	0.0345 (12)	-0.0003 (10)	0.0085 (10)	-0.0048 (11)
C7	0.0415 (14)	0.0581 (17)	0.0355 (13)	0.0076 (12)	0.0062 (11)	0.0035 (12)
C8	0.0597 (18)	0.0509 (16)	0.0383 (14)	0.0123 (14)	0.0154 (13)	0.0101 (12)
C9	0.0500 (16)	0.0423 (15)	0.0499 (16)	0.0011 (12)	0.0227 (13)	0.0083 (12)
C10	0.0318 (11)	0.0350 (13)	0.0312 (11)	-0.0004 (9)	0.0086 (9)	-0.0022 (10)
C11	0.0296 (12)	0.0320 (12)	0.0353 (12)	-0.0025 (9)	0.0097 (10)	-0.0030 (10)
O1	0.0448 (12)	0.0426 (11)	0.0532 (12)	0.0076 (9)	0.0039 (10)	-0.0025 (10)

Geometric parameters (Å, °)

Cd1—O1	2.233 (2)	C3—C4	1.397 (4)
Cd1—O1 ⁱ	2.233 (2)	C3—H3	0.9300
Cd1—N1 ⁱ	2.3217 (19)	C4—C11	1.391 (3)
Cd1—N1	2.3217 (19)	C4—C5	1.468 (4)
Cd1—O2	2.410 (2)	C5—C6	1.507 (4)
Cd1—O2 ⁱ	2.410 (2)	C6—C7	1.374 (4)
N1—C11	1.327 (3)	C6—C10	1.396 (3)
N1—C1	1.371 (3)	C7—C8	1.386 (4)
N2—C10	1.328 (3)	C7—H7	0.9300
N2—C9	1.355 (3)	C8—C9	1.380 (4)
O2—C1	1.269 (3)	C8—H8	0.9300
O3—C5	1.219 (3)	C9—H9	0.9300
C1—C2	1.427 (4)	C10—C11	1.482 (4)
C2—C3	1.365 (4)	O1—H1A	0.817 (18)
C2—H2	0.9300	O1—H1B	0.832 (19)
O1—Cd1—O1 ⁱ	101.59 (13)	C4—C3—H3	120.9
O1—Cd1—N1 ⁱ	92.77 (7)	C11—C4—C3	118.8 (2)
O1 ⁱ —Cd1—N1 ⁱ	105.03 (8)	C11—C4—C5	109.1 (2)
O1—Cd1—N1	105.03 (8)	C3—C4—C5	132.1 (2)
O1 ⁱ —Cd1—N1	92.77 (7)	O3—C5—C4	128.9 (3)
N1 ⁱ —Cd1—N1	151.82 (11)	O3—C5—C6	125.5 (3)
O1—Cd1—O2	94.62 (9)	C4—C5—C6	105.5 (2)
O1 ⁱ —Cd1—O2	148.19 (7)	C7—C6—C10	119.6 (2)
N1 ⁱ —Cd1—O2	101.27 (7)	C7—C6—C5	132.1 (2)
N1—Cd1—O2	56.38 (7)	C10—C6—C5	108.3 (2)
O1—Cd1—O2 ⁱ	148.19 (7)	C6—C7—C8	116.8 (2)
O1 ⁱ —Cd1—O2 ⁱ	94.62 (9)	C6—C7—H7	121.6
N1 ⁱ —Cd1—O2 ⁱ	56.38 (7)	C8—C7—H7	121.6
N1—Cd1—O2 ⁱ	101.27 (7)	C9—C8—C7	120.0 (3)
O2—Cd1—O2 ⁱ	85.23 (11)	C9—C8—H8	120.0
C11—N1—C1	118.1 (2)	C7—C8—H8	120.0
C11—N1—Cd1	147.41 (17)	N2—C9—C8	124.0 (3)
C1—N1—Cd1	94.51 (14)	N2—C9—H9	118.0
C10—N2—C9	115.1 (2)	C8—C9—H9	118.0
C1—O2—Cd1	93.31 (15)	N2—C10—C6	124.6 (2)

O2—C1—N1	115.7 (2)	N2—C10—C11	127.3 (2)
O2—C1—C2	124.3 (2)	C6—C10—C11	108.1 (2)
N1—C1—C2	120.0 (2)	N1—C11—C4	124.2 (2)
C3—C2—C1	120.8 (3)	N1—C11—C10	126.9 (2)
C3—C2—H2	119.6	C4—C11—C10	108.9 (2)
C1—C2—H2	119.6	Cd1—O1—H1A	109 (2)
C2—C3—C4	118.2 (2)	Cd1—O1—H1B	120 (2)
C2—C3—H3	120.9	H1A—O1—H1B	107 (3)
O1—Cd1—N1—C11	93.5 (3)	C3—C4—C5—C6	-179.4 (3)
O1 ⁱ —Cd1—N1—C11	-9.3 (3)	O3—C5—C6—C7	2.3 (5)
N1 ⁱ —Cd1—N1—C11	-139.1 (3)	C4—C5—C6—C7	-179.5 (3)
O2—Cd1—N1—C11	179.0 (3)	O3—C5—C6—C10	-176.9 (3)
O2 ⁱ —Cd1—N1—C11	-104.6 (3)	C4—C5—C6—C10	1.4 (3)
O1—Cd1—N1—C1	-87.51 (16)	C10—C6—C7—C8	0.9 (4)
O1 ⁱ —Cd1—N1—C1	169.70 (14)	C5—C6—C7—C8	-178.2 (3)
N1 ⁱ —Cd1—N1—C1	39.89 (13)	C6—C7—C8—C9	0.1 (4)
O2—Cd1—N1—C1	-2.01 (13)	C10—N2—C9—C8	0.3 (4)
O2 ⁱ —Cd1—N1—C1	74.44 (15)	C7—C8—C9—N2	-0.7 (5)
O1—Cd1—O2—C1	107.16 (15)	C9—N2—C10—C6	0.7 (4)
O1 ⁱ —Cd1—O2—C1	-13.7 (2)	C9—N2—C10—C11	-179.8 (2)
N1 ⁱ —Cd1—O2—C1	-159.08 (14)	C7—C6—C10—N2	-1.3 (4)
N1—Cd1—O2—C1	2.17 (14)	C5—C6—C10—N2	178.0 (2)
O2 ⁱ —Cd1—O2—C1	-104.75 (16)	C7—C6—C10—C11	179.0 (2)
Cd1—O2—C1—N1	-3.4 (2)	C5—C6—C10—C11	-1.7 (3)
Cd1—O2—C1—C2	177.7 (2)	C1—N1—C11—C4	-0.2 (4)
C11—N1—C1—O2	-177.1 (2)	Cd1—N1—C11—C4	178.7 (2)
Cd1—N1—C1—O2	3.5 (2)	C1—N1—C11—C10	-180.0 (2)
C11—N1—C1—C2	1.9 (3)	Cd1—N1—C11—C10	-1.1 (5)
Cd1—N1—C1—C2	-177.5 (2)	C3—C4—C11—N1	-1.3 (4)
O2—C1—C2—C3	176.7 (2)	C5—C4—C11—N1	179.7 (2)
N1—C1—C2—C3	-2.2 (4)	C3—C4—C11—C10	178.6 (2)
C1—C2—C3—C4	0.7 (4)	C5—C4—C11—C10	-0.5 (3)
C2—C3—C4—C11	0.9 (4)	N2—C10—C11—N1	1.6 (4)
C2—C3—C4—C5	179.7 (3)	C6—C10—C11—N1	-178.8 (2)
C11—C4—C5—O3	177.7 (3)	N2—C10—C11—C4	-178.3 (2)
C3—C4—C5—O3	-1.2 (5)	C6—C10—C11—C4	1.4 (3)
C11—C4—C5—C6	-0.5 (3)		

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

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>
O1—H1A \cdots N2 ⁱ	0.82 (2)	2.02 (2)	2.832 (3)	172 (3)
O1—H1B \cdots O2 ⁱⁱ	0.83 (2)	1.86 (2)	2.686 (3)	170 (4)
C3—H3 \cdots O3 ⁱⁱⁱ	0.93	2.48	3.351 (5)	156

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

Fig. 1

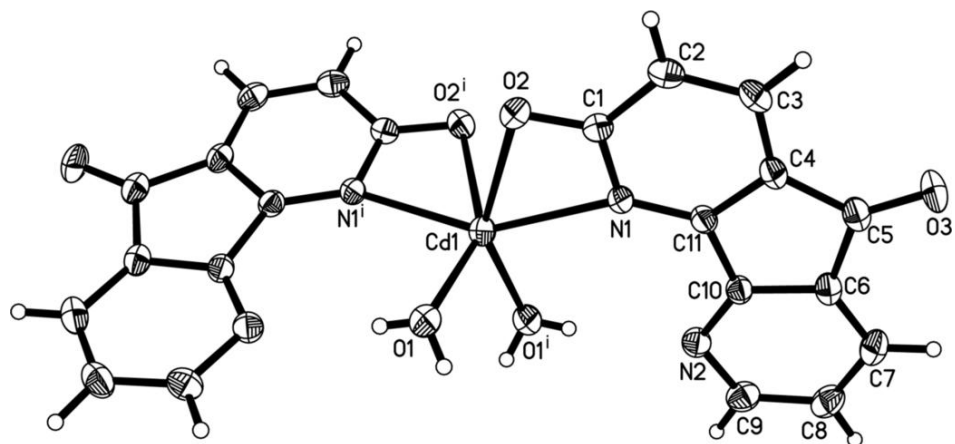


Fig. 2

