STRUCTURAL
CHEMISTRY

Volume 77 (2021)

Supporting information for article:

A new two-dimensional folding sheet-like coordination polymer assembled from cadmium(II) and (*S*)-2-(benzylamino)succinic acid: synthesis, structure and properties

Yan Liu and Hong-Tao Zhang

1.1.1. Synthesis of (S)-2-(benzylamino)succinic acid (N-benzyl-(*S*)-aspartic acid)

All commercially available chemicals were of reagent grade and used as received without further purification. The IR spectra were taken on a Shimadzu IR Prestige-21 spectrometer. The ^{1}H NMR and ^{13}C NMR spectra were recorded with a Bruker AVANCE III HD 500 MHz NMR spectrometer by using D_2O solvent.

To a solution of *S*-aspartic acid (3.99 g, 0.03 mol) and NaOH (2.40 g, 0.06 mol) in 30 ml of water was added excess salicylaldehyde (6.45 g, 0.06 mol) in 30 ml of EtOH. The light yellow solution was stirred for 1 h at room temperature prior to cooling in an ice bath. Then excess solid $NaBH_4$ (2.65 g, 0.07 mol) was added to the intermediate Schiff base solution in portions with gentle and stirring while the light yellow colour slowly discharged. After 2 h the solvent was completely removed and extracted with 200ml MeOH. Then the solution was acidified with concentrated HCl to a pH of 6.0~7.0. The resulting colourless solid was filtered off, washed with MeOH and Et2O. The crude product was purified by recrystallization from H2O/EtOH (1:10) and dried in oven at 120°C. Yield: 3.73 g (56%). m.p. 201–203 °C.

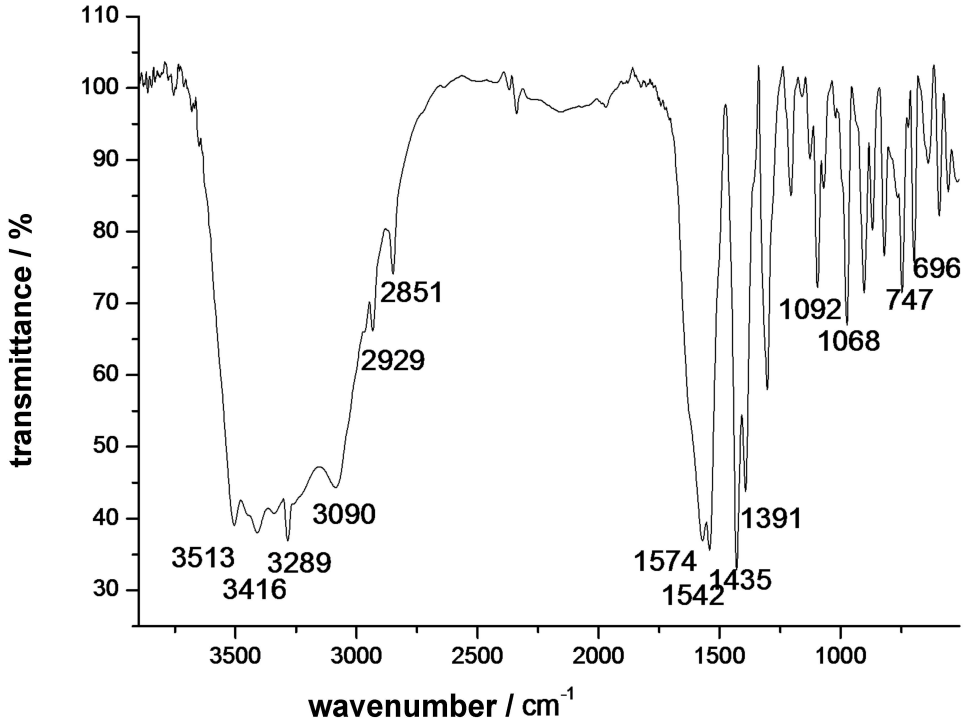

Figure S1 The IR spectrum of the title complex (I).

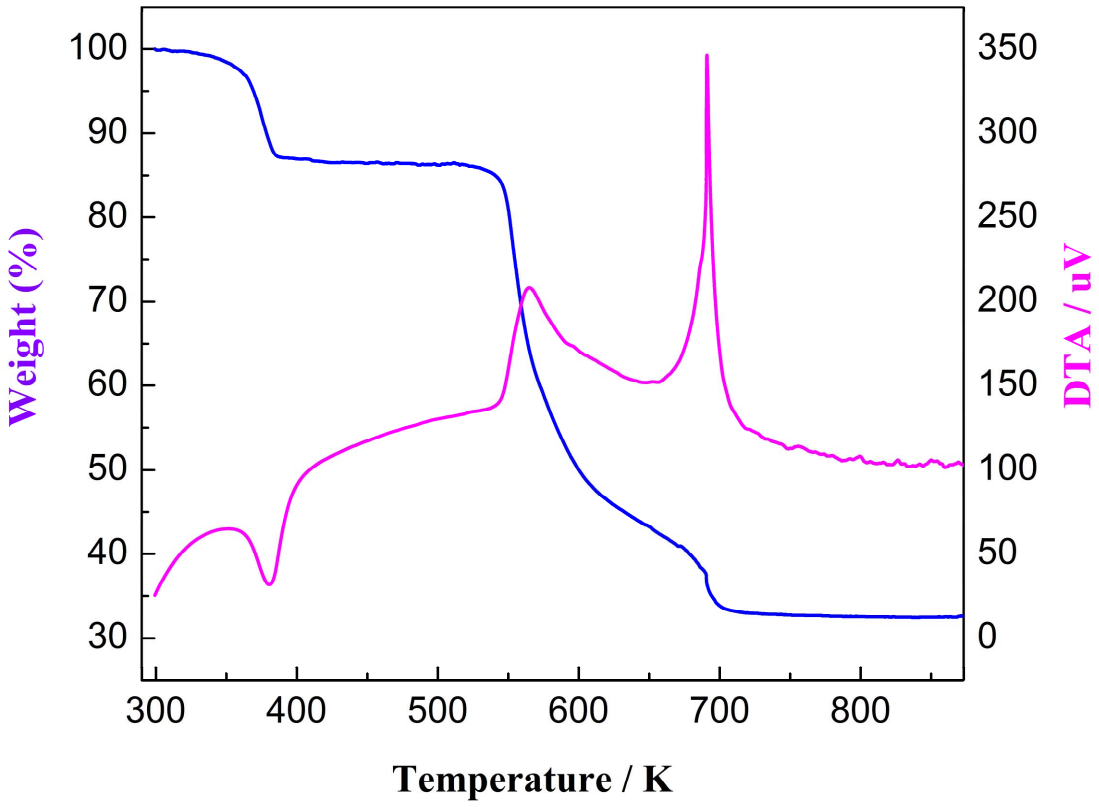

Figure S2. The TG and DTA curves for the title complex (I).

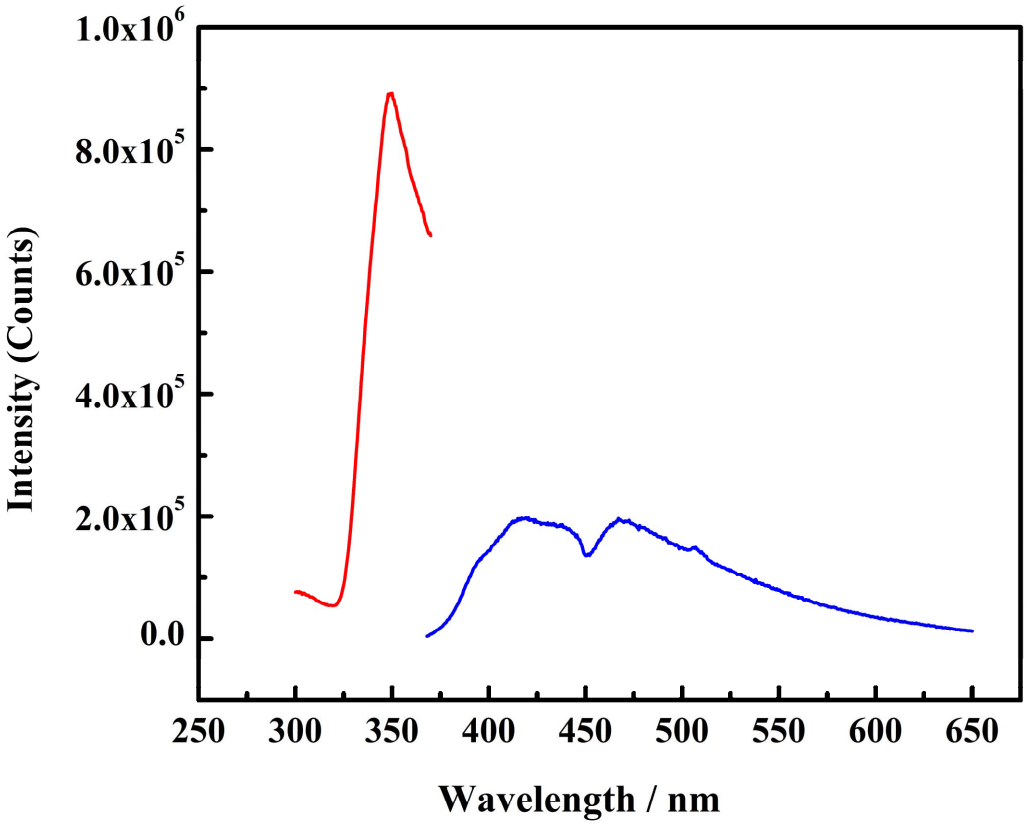

Figure S3. The Excitation (red) and emission (blue) spectra of the coordination polymer (I).

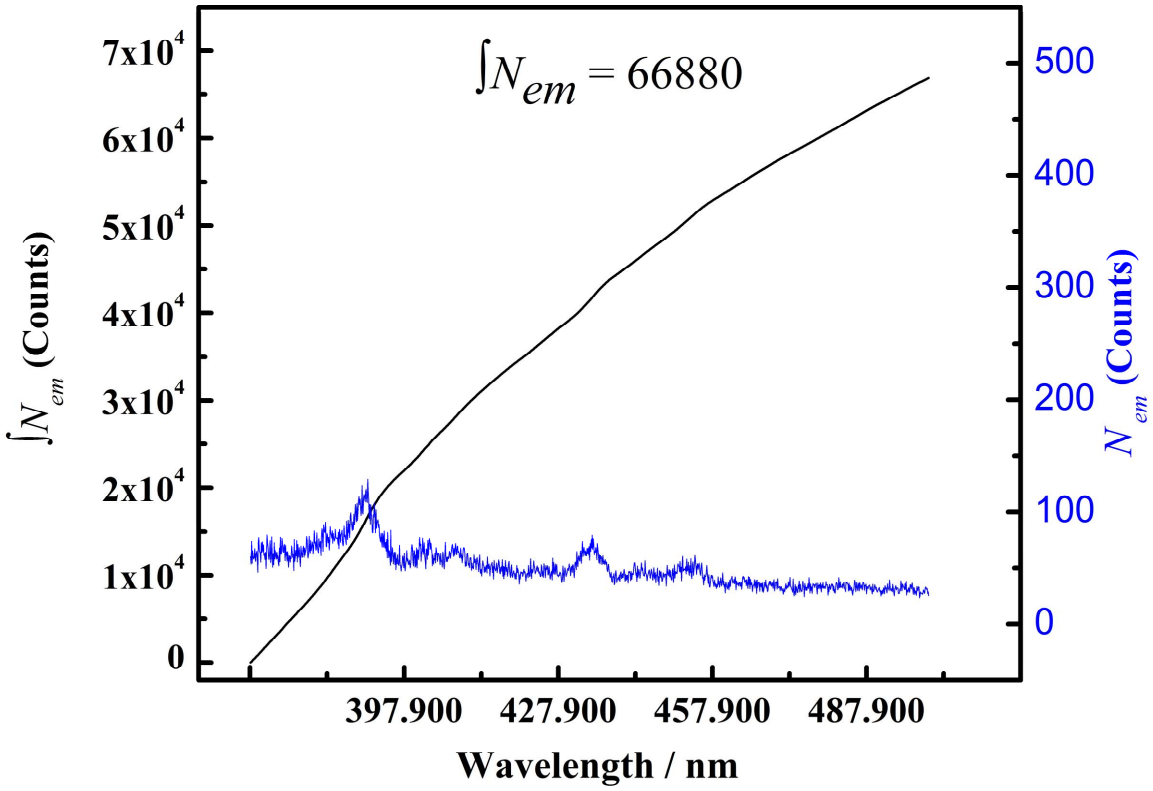

Figure S4. The intergration of the corrected emission for (I).

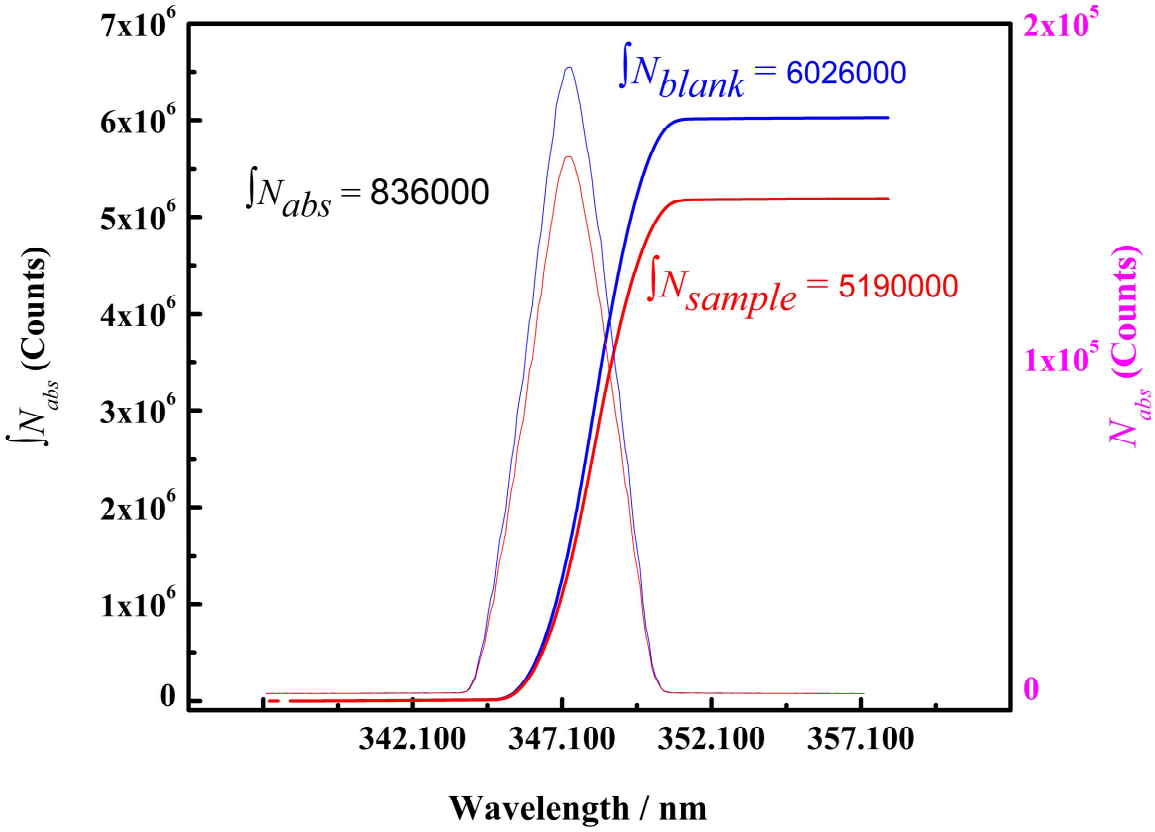

Figure S5. The intergration of the corrected absorption of (I) at the excitation wavelength (348nm).

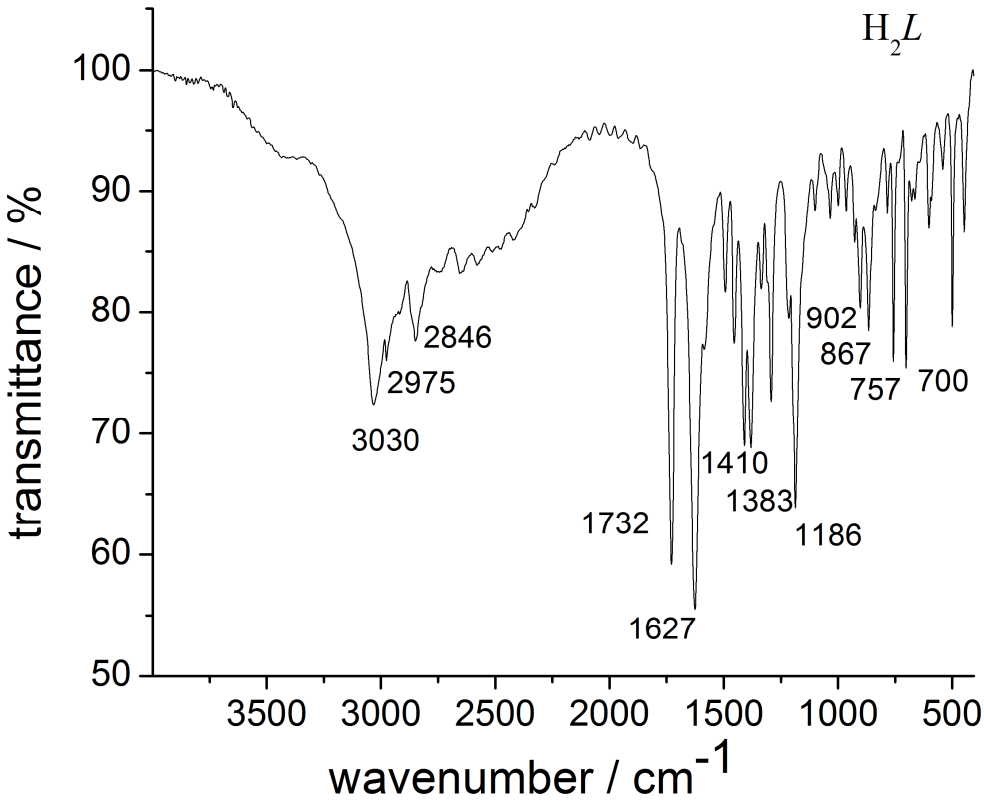

Figure S6. The IR spectrum of N-benzyl-(*S*)-aspartic acid.

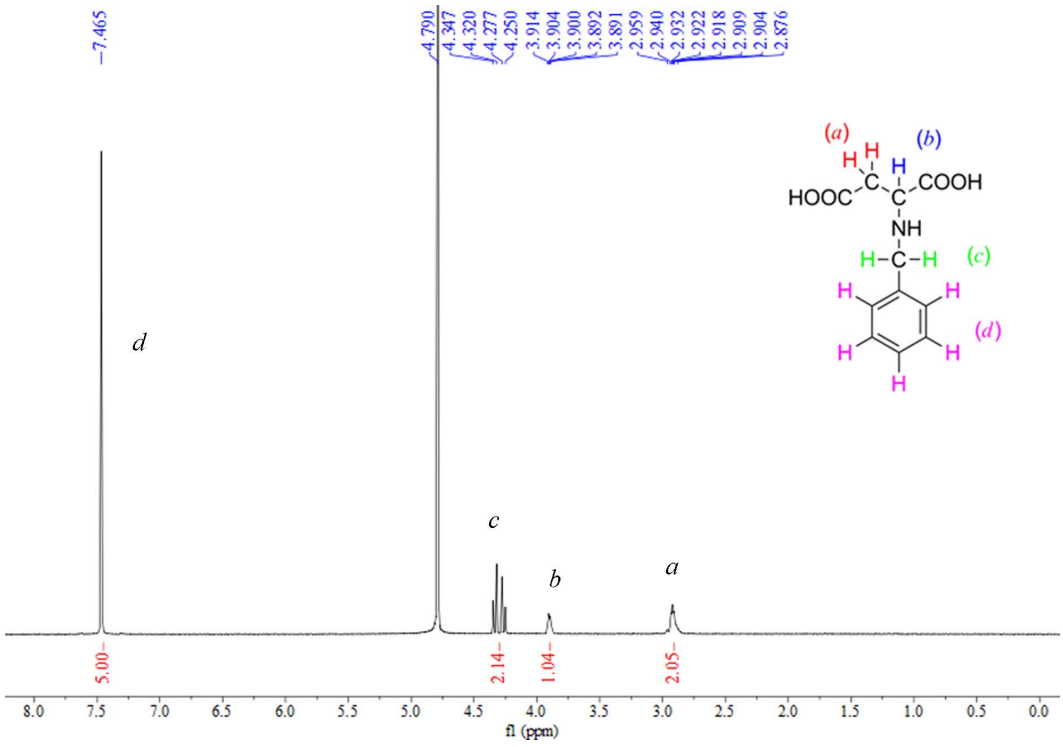

Figure S7. The ^{1}H NMR spectrum of N-benzyl-(*S*)-aspartic acid.

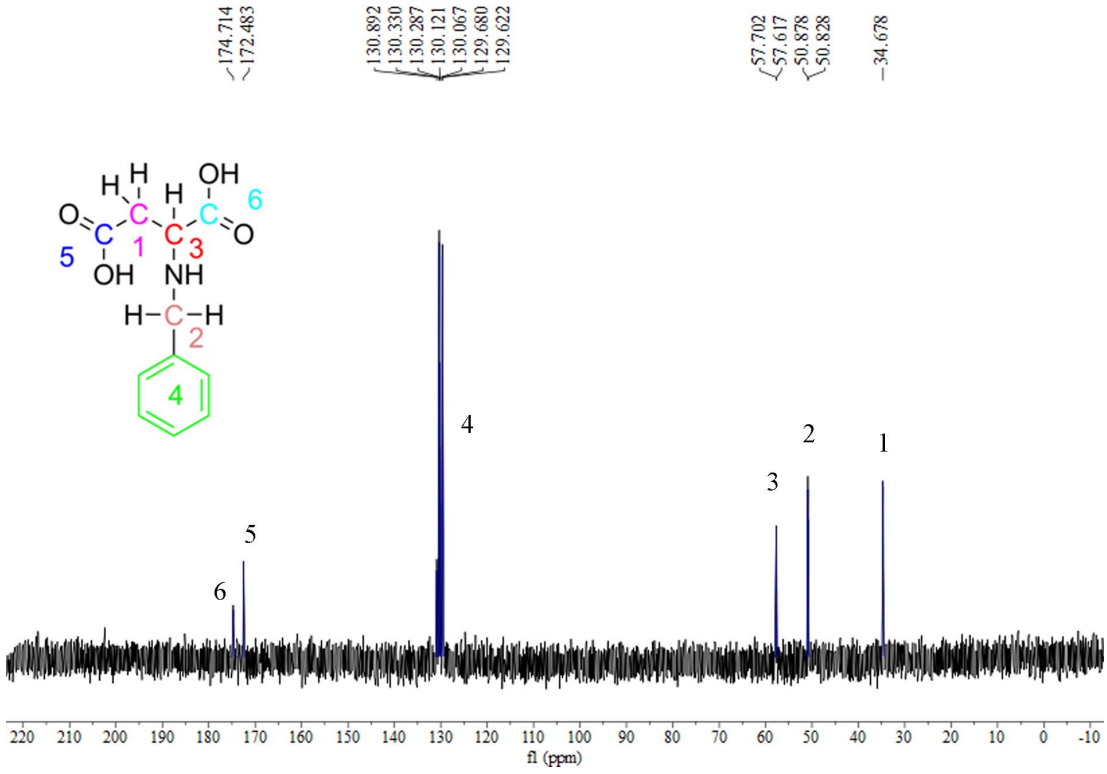

Figure S8. The ^{13}C NMR spectrum of N-benzyl-(*S*)-aspartic acid.

Table 1 Experimental details

	140530_0m
Crystal data	
Chemical formula	$C_{11}H_{15}CdNO_6 \cdot H_2O$
M_r	387.65
Crystal system, space group	Monoclinic, $P2_1$
Temperature (K)	298
a, *b*, *c* (Å)	5.8737 (9), 8.3842 (13), 13.788 (2)
β (°)	95.025 (2)
V (Å^3)	676.39 (18)
Z	2
Radiation type	Mo *K*α
μ (mm^{-1})	1.65
Crystal size (mm)	0.19 × 0.08 × 0.06
Data collection	
Diffractometer	CCD area detector
Absorption correction	Multi-scan *SADABS* (Bruker, 2004)
T_{min}, T_{max}	0.765, 0.908

No. of measured, independent and observed [$I > 2\sigma(I)$] reflections	5888, 2952, 2618
R_{int}	0.034
$(\sin \theta/\lambda)_{max}$ (Å^{-1})	0.652
Refinement	
$R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, S	0.030, 0.064, 0.94
No. of reflections	2952
No. of parameters	181
No. of restraints	163
H-atom treatment	H-atom parameters constrained
$\Delta\rho_{max}$, $\Delta\rho_{min}$ (e Å^{-3})	0.49, −0.73
Absolute structure	Flack x determined using 1034 quotients [(I+)-(I-)]/[(I+)+(I-)] (Parsons, Flack and Wagner, Acta Cryst. B69 (2013) 249-259).
Absolute structure parameter	0.02 (3)

Computer programs: *APEX2* (Bruker, 2004), *SAINT* (Bruker, 2004), *SHELXS97* (Sheldrick, 2008), *SHELXL2014*/7 (Sheldrick, 2015), *DIAMOND* (Brandenburg, 2008).

Table 2 Selected geometric parameters (Å, º)

Cd1—O1^{i}	2.267 (3)	Cd1—O5	2.390 (4)
Cd1—O2^{ii}	2.289 (4)	N1—Cd1^{iii}	2.374 (5)
Cd1—O6	2.298 (4)	O1—Cd1^{iv}	2.267 (3)
Cd1—O3	2.312 (4)	O2—Cd1^{iii}	2.289 (4)
Cd1—N1^{ii}	2.374 (5)		
O1^{i}—Cd1—O2^{ii}	81.6 (2)	O3—Cd1—N1^{ii}	98.42 (16)
O1^{i}—Cd1—O3	94.07 (15)	O6—Cd1—O5	78.83 (16)
O2^{ii}—Cd1—N1^{ii}	82.33 (15)	N1^{ii}—Cd1—O5	82.23 (16)
O6—Cd1—N1^{ii}	79.72 (16)		
O1—C1—C2—C3	143.6 (5)	N1—C3—C4—O3	18.9 (7)
O2—C1—C2—C3	−36.5 (8)	C2—C3—N1—C5	85.2 (6)
C1—C2—C3—N1	80.6 (6)	C4—C3—N1—C5	−148.4 (5)

C1—C2—C3—C4	−45.4 (7)	O2—C1—O1—Cd1^{iv}	−13.5 (7)
O1—C1—O2—Cd1^{iii}	148.0 (4)		

Symmetry code(s): (i) −*x*+1, *y*−1/2, −*z*+2; (ii) *x*+1, *y*, *z*; (iii) *x*−1, *y*, *z*; (iv) −*x*+1, *y*+1/2, −*z*+2.

Table 3 Selected hydrogen-bond parameters

D—H···*A*	*D*—H (Å)	H···*A* (Å)	*D*···*A* (Å)	*D*—H···*A* (°)
C5—H5B···O6^{i}	0.97	2.51	3.182 (8)	126.7
C2—H2B···O4^{i}	0.97	2.56	3.256 (8)	128.8
C2—H2A···O7^{ii}	0.97	2.55	3.345 (8)	139.4
O7—H7B···O4^{iii}	0.85	2.39	2.865 (7)	116.2
O7—H7B···O1^{iii}	0.85	2.51	3.149 (7)	132.3
O7—H7A···O5^{i}	0.85	2.36	2.945 (7)	126.6
O6—H6B···O2^{iv}	0.85	2.52	3.055 (6)	121.9
O6—H6B···O1^{v}	0.85	2.10	2.858 (8)	147.7
O6—H6A···O7	0.86	1.79	2.651 (7)	171.7
O5—H5D···O4^{vi}	0.84	1.92	2.706 (7)	154.4
O5—H5C···O3^{vii}	0.85	2.13	2.829 (6)	139.0
N1—H1···O3	0.98	2.21	2.712 (6)	110.1

Symmetry code(s): (i) *x*−1, *y*, *z*; (ii) *x*, *y*+1, *z*; (iii) *x*, *y*−1, *z*; (iv) −*x*+1, *y*−1/2, −*z*+2; (v) *x*+1, *y*−1, *z*; (vi) −*x*+2, *y*−1/2, −*z*+2; (vii) *x*+1, *y*, *z*.