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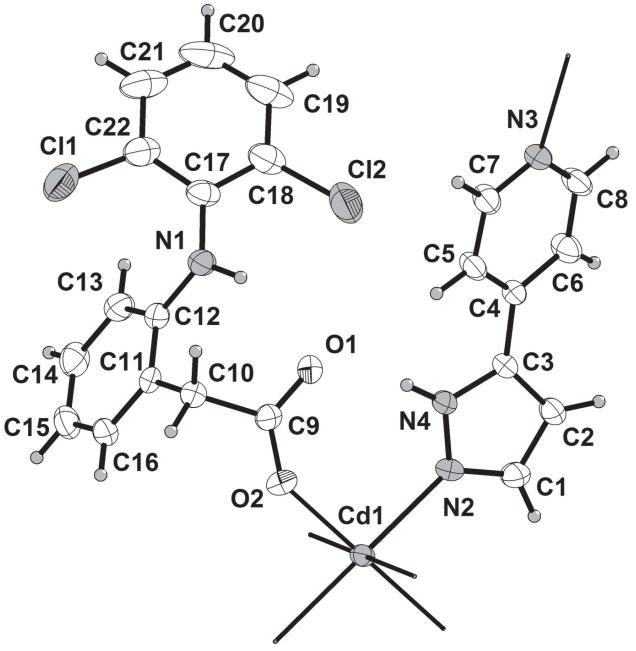
Crystal structure of bis(4-(1*H*-pyrazol-5-yl)pyridine-κ²*N*:*N*′)-bis(2-(2-((2,6-difluorophenyl)amino)phenyl)acetate-κO)cadmium(II), $C_{44}H_{34}N_8CdCl_4O_4$



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Abstract

$C_{44}H_{34}N_8CdCl_4O_4$, monoclinic, $P2_1/c$ (no. 14), $a = 9.7056(2)$ Å, $b = 14.3244(3)$ Å, $c = 15.3626(3)$ Å, $\beta = 99.7540(10)°$, $V = 2104.94(7)$ Å^3, $Z = 2$, $R_{gt}(F) = 0.0271$, $wR_{ref}(F^2) = 0.0717$, $T = 296(2)$ K.

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The crystal structure is shown in the figure. Tables 1 and 2 contain details on crystal structure and measurement conditions and a list of the atoms including atomic coordinates and displacement parameters.

Source of material

A mixture of $Cd(OAc)_2{\cdot}2H_2O$ (0.1 mmol, 0.0267 g), [2-(2,6-Dichloro-phenylamino)-phenyl]-acetic acid (0.1 mmol, 891 mg), 4-(1*H*-pyrazol-5-yl)pyridine (0.1 mmol, 55 mg) and distilled water (10 mL) was heated in a 25 mL stainless steel reactor with a Teflon liner at 403 K for 40 h, followed by slow cooling to room temperature. Colourless crystals of the title compound formed.

Table 1: Data collection and handling.

Crystal:	Block, colourless
Size:	0.41 × 0.32 × 0.28 mm
Wavelength:	Mo $K\alpha$ radiation (0.71073 Å)
μ:	90.83 mm^{-1}
Diffractometer, scan mode:	Bruker SMART, φ and ω-scans
$2\theta_{max}$, completeness:	27.4°, >99%
$N(hkl)_{measured}$, $N(hkl)_{unique}$, R_{int}:	15541, 3916, 0.036
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\,\sigma(I_{obs})$, 3305
$N(param)_{refined}$:	281
Programs:	Bruker programs [1], SHELX [2]

Experimental details

The hydrogen atoms were placed geometrically and refined using a riding model were set to $1.2U_{eq}$ (C, N).

Discussion

The design and construction of metal-organic frameworks (MOFs) with well-regulated network structures has been provoked significant interest on materials with properties in the areas of electrochemistry, photophysics, catalysis, adsorption and separation [3–6]. The organic ligands, which have their differences in the size, the flexibility, the coordination ability, the number of the carboxylate groups, the positions of the carboxylate groups and so on, play crucial role in the design and construction of desirable frameworks with interesting properties [7–9]. In addition, N-containing auxiliary ligands are known to be ideal connectors between metal atoms for the propagation of coordination networks.

The asymmetric unit of the title structure contains one half of a Cd(II) ion, one 4-(1*H*-Pyrazol-5-yl)-pyridine and one [2-(2,6-Dichloro-phenylamino)-phenyl]-acetate ligands to construct a coordination polymer. The cadmium atom is six-coordinated [CdN_4O_2] in a distorted octahedral manner by two oxygen atoms and by four nitrogen atoms. The Cd—O bond length is 2.3028(16) Å [10]. The Cd—N bond lengths are 2.347(2) Å and 2.373(2) Å respectively. The C(17)—C(22)—Cl(1) angle is 120.2(2)°, the C(17)—C(18)—Cl(2) angle is 118.2(2)°, the C(17)—N(1)—C(12) angle is 123.7(2)°. The hydrogen bonding

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Table 2: Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å^2).

Atom	*x*	*y*	*z*	U_{iso}*/U_{eq}
Cd1	0.5000	0.5000	0.0000	0.02525(8)
Cl1	−0.01106(9)	1.02161(5)	−0.13655(5)	0.0604(2)
Cl2	0.43714(10)	0.94438(6)	0.11272(5)	0.0698(2)
N1	0.2092(2)	0.89303(14)	−0.03446(14)	0.0378(5)
N2	0.6388(2)	0.59542(12)	0.10400(12)	0.0327(4)
N3	0.5709(2)	0.91377(12)	0.38330(12)	0.0323(4)
N4	0.58459(19)	0.66741(12)	0.14308(11)	0.0289(4)
H4	0.5010	0.6883	0.1271	0.035*
O1	0.33851(18)	0.72262(11)	0.04983(11)	0.0419(4)
O2	0.29393(17)	0.58216(10)	−0.01035(11)	0.0380(4)
C1	0.7677(3)	0.58519(16)	0.14839(16)	0.0389(6)
H1	0.8301	0.5397	0.1362	0.047*
C2	0.7976(3)	0.65099(16)	0.21537(16)	0.0387(6)
H2	0.8807	0.6584	0.2549	0.046*
C3	0.6770(2)	0.70289(14)	0.21022(14)	0.0282(5)
C4	0.6406(2)	0.77903(14)	0.26620(14)	0.0281(5)
C5	0.5038(3)	0.79654(16)	0.27378(15)	0.0356(6)
H5	0.4318	0.7632	0.2397	0.043*
C6	0.7424(3)	0.83333(17)	0.31681(16)	0.0399(6)
H6	0.8360	0.8258	0.3121	0.048*
C7	0.4740(3)	0.86353(17)	0.33197(15)	0.0375(6)
H7	0.3807	0.8744	0.3357	0.045*
C8	0.7036(3)	0.89851(17)	0.37418(16)	0.0395(6)
H8	0.7733	0.9336	0.4082	0.047*
C9	0.2635(2)	0.66551(14)	0.00247(14)	0.0284(5)
C10	0.1234(2)	0.70103(15)	−0.04653(15)	0.0322(5)
H10A	0.0622	0.6486	−0.0652	0.039*
H10B	0.0791	0.7398	−0.0075	0.039*
C11	0.1463(2)	0.75718(15)	−0.12630(14)	0.0290(5)
C12	0.1924(2)	0.84984(15)	−0.11815(14)	0.0320(5)
C13	0.2205(3)	0.89681(17)	−0.19272(16)	0.0396(6)
H13	0.2532	0.9579	−0.1875	0.048*
C14	0.2003(3)	0.85349(18)	−0.27415(16)	0.0430(6)
H14	0.2181	0.8859	−0.3235	0.052*
C15	0.1541(3)	0.76290(18)	−0.28274(16)	0.0419(6)
H15	0.1402	0.7338	−0.3377	0.050*
C16	0.1287(2)	0.71549(17)	−0.20907(16)	0.0359(6)
H16	0.0989	0.6538	−0.2149	0.043*
C17	0.2297(3)	0.98808(15)	−0.02138(16)	0.0391(6)
C18	0.3365(3)	1.02311(18)	0.04314(18)	0.0497(7)
C19	0.3631(4)	1.1166(2)	0.0549(2)	0.0705(10)
H19	0.4352	1.1371	0.0984	0.085*
C20	0.2815(5)	1.1801(2)	0.0015(2)	0.0775(12)
H20	0.3023	1.2434	0.0065	0.093*
C21	0.1707(4)	1.14997(19)	−0.0584(2)	0.0641(9)
H21	0.1141	1.1930	−0.0930	0.077*
C22	0.1419(3)	1.05543(17)	−0.06793(17)	0.0468(7)
H1D	0.254(3)	0.8617(17)	0.0036(16)	0.037(8)*

between one of the oxygen atoms and the NH group is present (d(N···O): 2.943(3) Å and N—H···O is 149(2) Å). Weak interactions result in the infinite three dimensional architecture.

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