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# Synthesis, crystal structure and thermal properties of *catena*-poly[[[bis(2,3-dimethylpyrazine- $\kappa$ N<sup>1</sup>)-cadmium(II)]-di- $\mu$ -chlorido] monohydrate]

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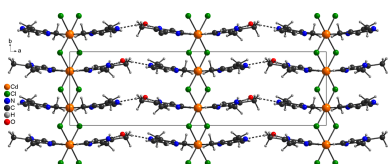
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The title compound,  $\{[\text{CdCl}_2(\text{C}_6\text{H}_8\text{N}_2)_2] \cdot \text{H}_2\text{O}\}_n$  ( $\text{C}_6\text{H}_8\text{N}_2$  = 2,3-dimethylpyrazine) was prepared by the reaction of cadmium chloride with 2,3-dimethylpyrazine in water. The asymmetric unit consists of two crystallographically independent cadmium cations that are situated on a twofold rotation axis, two crystallographically independent chloride anions and 2,3-dimethylpyrazine ligands as well as one water molecule that are all located in general positions. The cadmium cations are octahedrally coordinated by two terminally N-bonded 2,3-dimethylpyrazine ligands and four chloride anions and are linked by pairs of  $\mu$ -1,1 bridging chloride anions into chains that propagate in the crystallographic *b*-axis direction. These chains are connected into layers *via* the water molecule of crystallization by interchain O—H...N hydrogen bonding. Powder X-ray diffraction (PXRD) indicates that a pure phase has been obtained. Thermogravimetry (TG) coupled to differential thermal analysis (DTA) indicates that the water molecules are removed in the first step and that the 2,3-dimethylpyrazine ligands are lost in the subsequent two steps, leading to a compound with the composition  $[\text{CdCl}_2(\text{C}_6\text{H}_8\text{N}_2)]$ .

## 1. Chemical context

The crystal structures of transition-metal halide and pseudo-halide coordination compounds are characterized by a large structural variability because the metal cations can be linked by bridging halide anions into metal-halide substructures of different dimensionality (Kromp & Sheldrick, 1999; Peng *et al.*, 2010; Li *et al.*, 2005; Näther *et al.*, 2002). This also leads to compounds of different stoichiometry in which the ratio between the metal halide and the coligand is different. In this context, thermal decomposition of coligand-rich compounds are useful to detect possible coligand-deficient compounds as intermediates because in several cases the coligands were removed in separate steps before a transformation into the pure metal halides is observed (Näther *et al.*, 2001; Näther & Jess, 2001).

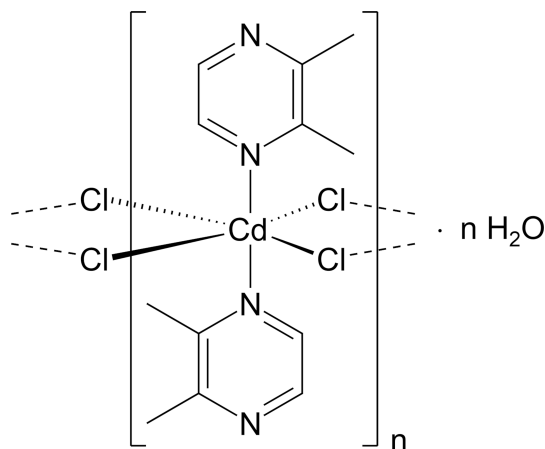
In the course of our systematic work, we reported new cadmium halide coordination compounds with 2,3-dimethylpyrazine ( $\text{C}_6\text{H}_8\text{N}_2$ ) as ligand. With  $\text{CdI}_2$  we obtained two compounds with the compositions  $\text{CdI}_2(\text{C}_6\text{H}_8\text{N}_2)_2$  and  $\text{CdI}_2(\text{C}_6\text{H}_8\text{N}_2)$  (Näther, 2026*a*). In both compounds the cadmium cations are tetrahedrally coordinated, leading to discrete complexes in the 2,3-dimethylpyrazine-rich compound and which are linked into chains by the coligands in the 2,3-dimethylpyrazine-deficient compound. This is surprising because, in most cases, cadmium prefers an octahedral coordination and in this case one would expect the formation of  $\text{CdI}_2$  chains built up of  $\text{CdI}_4\text{N}_2$  octahedra linked



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by common iodide edges. This is the case, for example, in  $\text{CdI}_2(\text{pyrazine})$ , in which the cadmium cations are linked by pairs of  $\mu$ -1,1 bridging halide anions into chains that are further connected into layers by the pyrazine ligands [CSD refcodes RINSOW, (Bailey & Pennington, 1997) and RINSOW01 (Pickardt & Staub, 1997)]. Starting from these results, we prepared the corresponding compounds with cadmium bromide and obtained a compound with the composition  $\text{CdBr}_2(\text{C}_6\text{H}_8\text{N}_2)(\text{H}_2\text{O}) \cdot \text{C}_6\text{H}_8\text{N}_2 \cdot 0.5\text{H}_2\text{O}$  that consists of octahedrally coordinated cadmium cations that are linked into chains *via* pairs of bromide anions (Näther, 2026*b*). In further work,  $\text{CdBr}_2(\text{C}_6\text{H}_8\text{N}_2)$  was structurally characterized. This compound exhibits the same ratio between the cadmium halide and the coligand as  $\text{CdI}_2(\text{C}_6\text{H}_8\text{N}_2)$  (Näther, 2026*c*). In contrast to the latter compound, where the cadmium cations adopt a tetrahedral coordination, for the bromide compound an octahedral coordination with a chain structure was observed. To complete these investigations, we became interested in the corresponding compounds with  $\text{CdCl}_2$  and obtained crystals of the title compound,  $\text{CdCl}_2(\text{C}_6\text{H}_8\text{N}_2)_2 \cdot \text{H}_2\text{O}$  (**I**), which were characterized by single-crystal X-ray diffraction.



## 2. Structural commentary

The asymmetric unit of (**I**) is built up of two crystallographically independent cadmium cations that are both located on crystallographic twofold rotation axes, two crystallographically independent chloride anions and two distinct 2,3-dimethylpyrazine ligands as well as one water molecule, all of them located in general positions. The cadmium cations are surrounded by four chloride anions and two terminally bonded 2,3-dimethylpyrazine ligands (Fig. 1). The bond angles show small deviations from the ideal octahedral values and there are no significant structural differences between the two crystallographically independent Cd centers (Table 1). The cadmium cations are linked by pairs of  $\mu$ -1,1 bridging chloride anions into linear chains that propagate in the *b*-axis direction (Fig. 2).

This structure is therefore similar to that of  $\text{CdBr}_2(\text{C}_6\text{H}_8\text{N}_2)$ , in which linear  $\text{CdX}$  chains are also found (Näther, 2026*c*). The major difference is that the 2,3-di-

**Table 1**  
Selected geometric parameters ( $\text{\AA}$ ,  $^\circ$ ).

|                                                                |             |                                                                  |            |
|----------------------------------------------------------------|-------------|------------------------------------------------------------------|------------|
| $\text{Cd1} - \text{N1}$                                       | 2.493 (3)   | $\text{Cd2} - \text{N11}$                                        | 2.491 (3)  |
| $\text{Cd1} - \text{Cl1}$                                      | 2.5773 (9)  | $\text{Cd2} - \text{Cl2}$                                        | 2.5815 (9) |
| $\text{Cd1} - \text{Cl2}^{\text{i}}$                           | 2.5958 (9)  | $\text{Cd2} - \text{Cl1}$                                        | 2.5930 (9) |
| $\text{Cd1} - \text{Cl2}^{\text{ii}}$                          | 2.5958 (9)  |                                                                  |            |
| $\text{N1} - \text{Cd1} - \text{N1}^{\text{iii}}$              | 176.45 (11) | $\text{N11} - \text{Cd2} - \text{Cl2}$                           | 88.11 (7)  |
| $\text{N1} - \text{Cd1} - \text{Cl1}^{\text{iii}}$             | 95.65 (7)   | $\text{N11}^{\text{iii}} - \text{Cd2} - \text{Cl2}$              | 94.93 (7)  |
| $\text{N1} - \text{Cd1} - \text{Cl1}$                          | 86.91 (7)   | $\text{Cl2} - \text{Cd2} - \text{Cl2}^{\text{iii}}$              | 88.64 (4)  |
| $\text{Cl1}^{\text{iii}} - \text{Cd1} - \text{Cl1}$            | 88.14 (4)   | $\text{N11} - \text{Cd2} - \text{Cl1}$                           | 89.88 (7)  |
| $\text{N1} - \text{Cd1} - \text{Cl2}^{\text{i}}$               | 86.33 (7)   | $\text{N11}^{\text{iii}} - \text{Cd2} - \text{Cl1}$              | 87.06 (7)  |
| $\text{N1}^{\text{iii}} - \text{Cd1} - \text{Cl2}^{\text{i}}$  | 91.11 (7)   | $\text{Cl2} - \text{Cd2} - \text{Cl1}$                           | 177.93 (2) |
| $\text{Cl1}^{\text{iii}} - \text{Cd1} - \text{Cl2}^{\text{i}}$ | 178.02 (2)  | $\text{Cl2}^{\text{iii}} - \text{Cd2} - \text{Cl1}$              | 91.98 (3)  |
| $\text{Cl1} - \text{Cd1} - \text{Cl2}^{\text{i}}$              | 91.95 (3)   | $\text{Cl2}^{\text{iii}} - \text{Cd2} - \text{Cl1}^{\text{iii}}$ | 177.93 (2) |
| $\text{Cl1} - \text{Cd1} - \text{Cl2}^{\text{ii}}$             | 178.02 (2)  | $\text{Cl1} - \text{Cd2} - \text{Cl1}^{\text{iii}}$              | 87.47 (4)  |
| $\text{Cl2}^{\text{i}} - \text{Cd1} - \text{Cl2}^{\text{ii}}$  | 88.03 (4)   | $\text{Cd1} - \text{Cl1} - \text{Cd2}$                           | 92.20 (3)  |
| $\text{N11} - \text{Cd2} - \text{N11}^{\text{iii}}$            | 175.76 (11) | $\text{Cd2} - \text{Cl2} - \text{Cd1}^{\text{iv}}$               | 91.66 (3)  |

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

methylpyrazine ligands are only terminally bonded in the title compound, whereas they act as bridging ligands in the bromide compound and this is also reflected in the different ratio between  $\text{CdX}_2$  and the 2,3-dimethylpyrazine coligand,

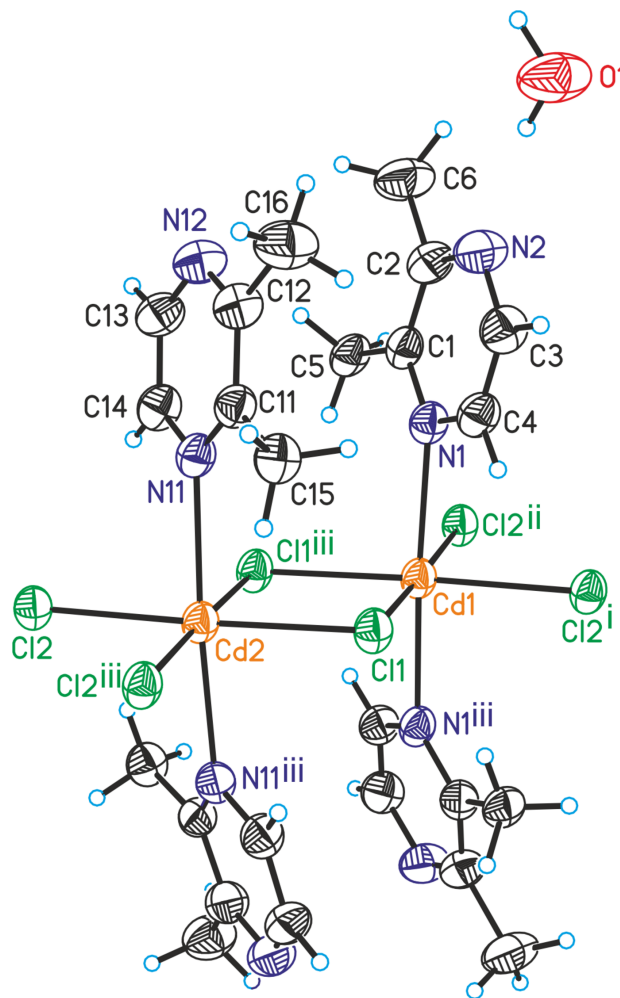


**Figure 1**  
Crystal structure of (**I**) with labeling and displacement ellipsoids drawn at the 50% probability level. Symmetry codes: (i)  $-x + 1, y + 1, -z + \frac{1}{2}$ ; (ii)  $x, y + 1, z$ ; (iii)  $-x + 1, y, -z + \frac{1}{2}$ .

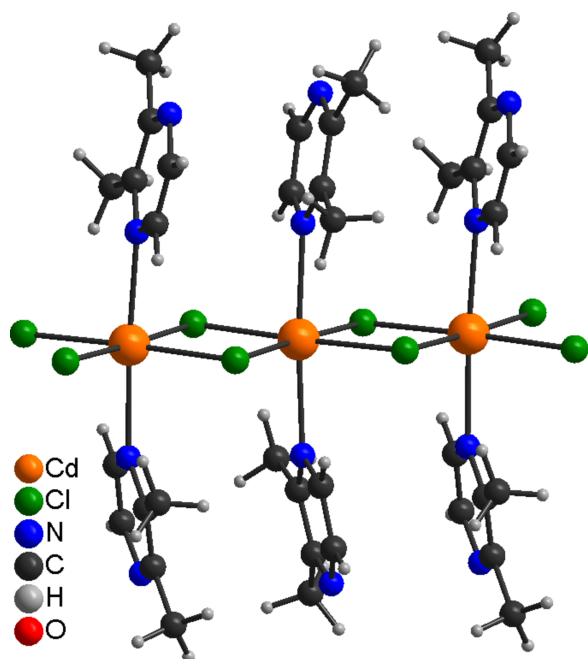


**Figure 2**  
View of a part of a chain in the extended structure of (I).

which is 1:2 in the title compound and 1:1 in the bromide compound. Moreover, these findings also show that in cadmium halide coordination compounds with 2,3-dimethylpyrazine, chloride and bromide anions behave in a similar way with an octahedral Cd coordination and the presence of chains, whereas for the corresponding iodide compounds a tetrahedral coordination is observed (Näther, 2026a). At first glance one would assume that the reason why the iodide compound shows a tetrahedral coordination might originate from steric repulsion between the larger iodide anions, which is lower in a tetrahedral coordination, but this would not explain why in  $\text{CdI}_2(\text{pyrazine})$  an octahedral coordination is observed. One may therefore suggest that repulsion between the large iodide anions and the methyl group that is adjacent to the coordinating N atom is responsible for all these findings.

### 3. Supramolecular features

In the extended structure of (I), the chains are linked *via* the water molecules of crystallization into layers by  $\text{O} \cdots \text{H} \cdots \text{N}$

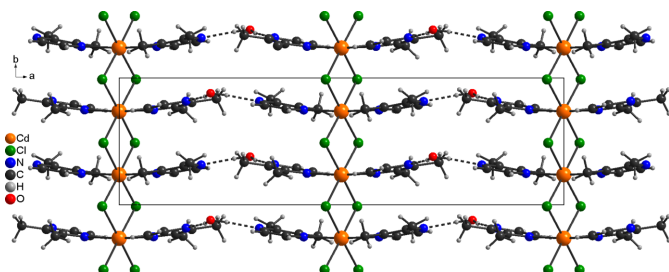


**Figure 3**  
Crystal structure of (I) in a view along the crystallographic *a*-axis direction.

**Table 2**  
Hydrogen-bond geometry ( $\text{\AA}$ ,  $^\circ$ ).

| $D\cdots H\cdots A$                                   | $D\cdots H$ | $H\cdots A$ | $D\cdots A$ | $D-H\cdots A$ |
|-------------------------------------------------------|-------------|-------------|-------------|---------------|
| $\text{O1}-\text{H1A}\cdots\text{N12}^{\text{v}}$     | 1.00 (7)    | 2.00 (7)    | 2.965 (4)   | 164 (5)       |
| $\text{O1}-\text{H1B}\cdots\text{N2}$                 | 0.94 (6)    | 2.02 (7)    | 2.939 (5)   | 165 (5)       |
| $\text{C3}-\text{H3}\cdots\text{O1}^{\text{vi}}$      | 0.95        | 2.61        | 3.282 (5)   | 128           |
| $\text{C4}-\text{H4}\cdots\text{Cl1}$                 | 0.95        | 2.93        | 3.490 (4)   | 119           |
| $\text{C4}-\text{H4}\cdots\text{Cl2}^{\text{i}}$      | 0.95        | 2.97        | 3.420 (4)   | 110           |
| $\text{C5}-\text{H5B}\cdots\text{Cl1}^{\text{vii}}$   | 0.98        | 2.88        | 3.747 (3)   | 147           |
| $\text{C5}-\text{H5C}\cdots\text{Cl1}^{\text{iii}}$   | 0.98        | 2.71        | 3.650 (4)   | 160           |
| $\text{C5}-\text{H5C}\cdots\text{Cl2}^{\text{ii}}$    | 0.98        | 2.97        | 3.451 (4)   | 112           |
| $\text{C13}-\text{H13}\cdots\text{Cl1}^{\text{vii}}$  | 0.95        | 2.98        | 3.786 (4)   | 143           |
| $\text{C14}-\text{H14}\cdots\text{Cl1}^{\text{iii}}$  | 0.95        | 2.95        | 3.419 (4)   | 112           |
| $\text{C14}-\text{H14}\cdots\text{Cl2}$               | 0.95        | 2.94        | 3.510 (4)   | 120           |
| $\text{C15}-\text{H15A}\cdots\text{Cl1}$              | 0.98        | 2.90        | 3.470 (4)   | 118           |
| $\text{C15}-\text{H15A}\cdots\text{Cl2}^{\text{iii}}$ | 0.98        | 2.76        | 3.670 (4)   | 154           |

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

hydrogen bonds to the second N atom of the 2,3-dimethylpyrazine ligand that is not involved in the metal coordination (Fig. 3). These layers lie parallel to the *ab* plane (Fig. 3). The  $\text{O}-\text{H}\cdots\text{N}$  bond angles are close to linear, indicating that this is a relatively strong interaction (Table 2). There are also a number of  $\text{C}-\text{H}\cdots\text{Cl}$  contacts, but most of them exhibit long distances and angles far from  $180^\circ$ , indicating only very weak interactions (Table 2). However, two of these contacts are relatively short with angles of  $154$  and  $160^\circ$ , indicating intra-chain hydrogen bonding (Table 2).

### 4. Additional characterization

The purity of the title compound was investigated by powder X-ray diffraction (PXRD) and comparison of the experimental PXRD pattern with that calculated from the results of the single crystal structure determination indicate that a pure phase has been obtained. However, some reflections are slightly shifted to higher Bragg angles in the calculated pattern, because of the different temperature and the pattern shows unusually strong preferred orientation (Fig. 4).

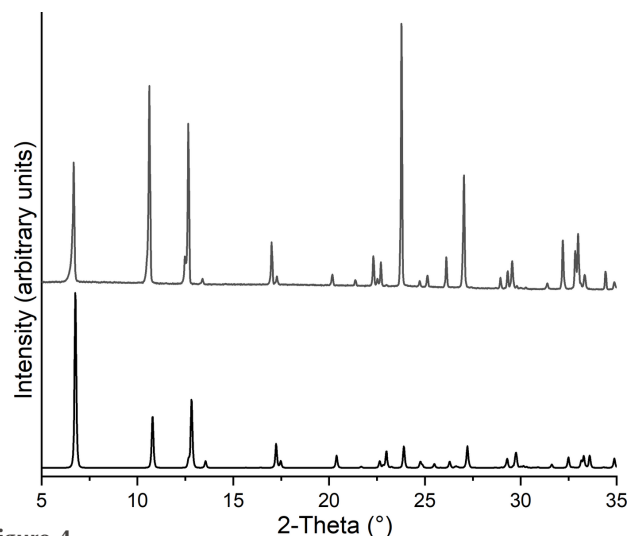


**Figure 4**  
Experimental (top) and calculated powder patterns (bottom) for (I).

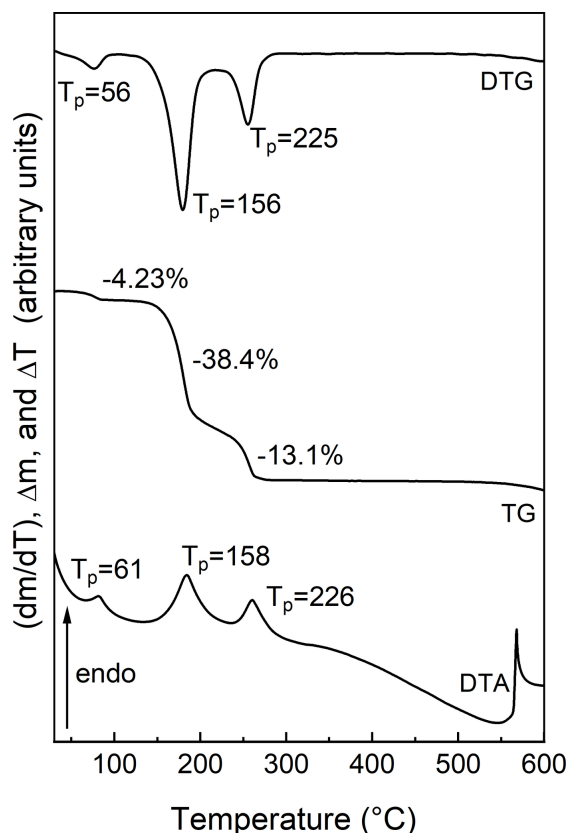


**Figure 5**  
DTG, TG and DTA curves for (I).

The thermal behavior of the title compound was investigated by thermogravimetry (TG) coupled to differential thermal analysis (DTA) to check if 2,3-dimethylpyrazine-deficient phases can be detected, as is frequently observed in such compounds. Upon heating three mass losses are observed, which are accompanied by endothermic events in the DTA curve (Fig. 5). The experimental mass loss in the first step of 4.23% is in very good agreement with that calculated for the removal of the water molecules (4.31%) and the mass loss in the second step corresponds to the removal of 1.5 of the 2,3-dimethylpyrazine molecules ( $\Delta m_{\text{calc.}} = 38.8\%$ ). Based on these results, one can expect that in the first step, a compound with the composition  $\text{CdCl}_2(\text{C}_6\text{H}_8\text{N}_2)_2$  is formed, which transforms into  $(\text{CdCl}_2)_2(\text{C}_6\text{H}_8\text{N}_2)$  upon further heating.

The residues obtained after the first and second mass losses were investigated by PXRD. Comparison of the pattern of the residue with that of the pristine compound shows only very minor differences in the intensity and practically no shift in the reflection positions (Fig. S1). Two such experiments were performed with identical results. At first glance one would assume that this reaction might be topotactic, but at least some shift in peak positions would be expected. It is very unlikely that the title compound immediately decomposes on storage and that the anhydrate was measured because several TG-DTA experiments were performed using the same batch and in all of them the mass loss associated with removal of the water molecules remain constant. On the other hand, we cannot exclude the possibility that the anhydrate is extremely

hygroscopic and immediately transforms back to the title compound. The powder pattern of the residue obtained after the second mass loss is completely different from that of the title compound and shows that a powder of low crystallinity has formed (Fig. S2). The composition assumed for this residue seems to be a bit unusual and points to a more condensed  $\text{CdCl}_2$  substructure. In this context it is noted that, for the corresponding copper(I) chloride-2,3-dimethylpyrazine coordination compounds, a number of different compounds with varying ratios between  $\text{CuCl}$  and 2,3-dimethylpyrazine have been reported. These include  $(\text{CuCl})_4(\text{C}_6\text{H}_8\text{N}_2)_9$  with a ratio of 4:9,  $\text{CuCl}(\text{C}_6\text{H}_8\text{N}_2)$  with a ratio of 1:1,  $(\text{CuCl})_3(\text{C}_6\text{H}_8\text{N}_2)_2$  with a ratio of 3:2 and  $(\text{CuCl})_2(\text{C}_6\text{H}_8\text{N}_2)$  with a ratio of 2:1 (Jess & Näther, 2006).

## 5. Database survey

As mentioned above, several compounds with cadmium halides and 2,3-dimethylpyrazine have already been reported and this formed the basis for this work. This includes the iodide compounds  $\text{CdI}_2(\text{C}_6\text{H}_8\text{N}_2)_2$  and  $\text{CdI}_2(\text{C}_6\text{H}_8\text{N}_2)$  (Näther, 2026a), as well as the bromide compounds  $\text{CdBr}_2(\text{C}_6\text{H}_8\text{N}_2)(\text{H}_2\text{O}) \cdot \text{C}_6\text{H}_8\text{N}_2 \cdot 0.5\text{H}_2\text{O}$  and  $\text{CdBr}_2(\text{C}_6\text{H}_8\text{N}_2)$  (Näther, 2026b,c).

A search in the CSD (Version 5.43, 2025; Groom *et al.*, 2016) using CONQUEST (Bruno *et al.*, 2002) revealed that in several other compounds with pyrazine or pyridine and its derivatives, similar  $\text{CdCl}_2$  chains and an octahedral coordination is found. This is the case, for example, in  $\text{CdCl}_2(\text{pyridine})_2$ , which shows the same chain structure as the title compound but contains no water of crystallization [CSD refcodes CDPYCL01 (Paulus, 1969), CDPYCL03 (Satoh *et al.*, 2001) and CDPYCL04 (Hu *et al.*, 2003)]. The same structure is also found in  $\text{CdCl}_2(4\text{-methylpyridine})_2$  (BASQUI; Satoh *et al.*, 2001),  $\text{CdCl}_2(3\text{-methylpyridine})_2$  [BASQOC (Satoh *et al.*, 2001), BASQOC01 (Hu *et al.*, 2003)].  $\text{CdCl}_2(\text{pyrazine})$  [TISSUJ (Pickardt & Staub, 1996), TISSUJ01 (Bailey & Pennington, 1997)] is an additional example but in contrast to the pyridine compounds, the  $\text{CdCl}_2$  chains are linked into layers.

## 6. Synthesis, crystallization and experimental details

Cadmium chloride and 2,3-dimethylpyrazine were purchased from Sigma-Aldrich. To prepare (I), 1.00 mmol (183.3 mg) of  $\text{CdCl}_2$  and 1.00 mmol (108.2 mg) of 2,3-dimethylpyrazine were reacted in 2 ml of water for 3 d at room temperature, which led to the formation of crystals suitable for single-crystal structure analysis.

The PXRD measurements were performed with  $\text{Cu } K\alpha_1$  radiation ( $\lambda = 1.540598 \text{ \AA}$ ) using a Stoe Transmission Powder Diffraction System (STADI P) equipped with a MYTHEN 1K detector and a Johansson-type Ge(111) monochromator.

Thermogravimetry and differential thermal analysis (TG-DTA) measurements were performed in a dynamic air atmosphere in  $\text{Al}_2\text{O}_3$  crucibles using a STA-PT 1000 ther-

mobalance from Linseis. The instrument was calibrated using standard reference materials.

## 7. Refinement

Crystal data, data collection and structure refinement details are summarized in Table 3. The C—H hydrogen atoms were positioned with idealized geometry (methyl H atoms allowed to rotate but not to tip) and were refined isotropically with  $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{C})$  (1.5 for methyl H atoms). The O-bound H atoms were located in difference maps and refined isotropically without restraints.

## Acknowledgements

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**Table 3**

Experimental details.

|                                                                                                                                                                                                                                                               |                                                                              |
|---------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------|------------------------------------------------------------------------------|
| Crystal data                                                                                                                                                                                                                                                  |                                                                              |
| Chemical formula                                                                                                                                                                                                                                              | $[\text{CdCl}_2(\text{C}_6\text{H}_8\text{N}_2)_2] \cdot \text{H}_2\text{O}$ |
| $M_r$                                                                                                                                                                                                                                                         | 417.60                                                                       |
| Crystal system, space group                                                                                                                                                                                                                                   | Monoclinic, $C2/c$                                                           |
| Temperature (K)                                                                                                                                                                                                                                               | 170                                                                          |
| $a, b, c$ (Å)                                                                                                                                                                                                                                                 | 30.6316 (15), 7.4391 (3),<br>16.3857 (8)                                     |
| $\beta$ (°)                                                                                                                                                                                                                                                   | 121.538 (3)                                                                  |
| $V$ (Å <sup>3</sup> )                                                                                                                                                                                                                                         | 3182.3 (3)                                                                   |
| $Z$                                                                                                                                                                                                                                                           | 8                                                                            |
| Radiation type                                                                                                                                                                                                                                                | Mo $K\alpha$                                                                 |
| $\mu$ (mm <sup>−1</sup> )                                                                                                                                                                                                                                     | 1.71                                                                         |
| Crystal size (mm)                                                                                                                                                                                                                                             | 0.14 × 0.12 × 0.09                                                           |
| Data collection                                                                                                                                                                                                                                               |                                                                              |
| Diffractometer                                                                                                                                                                                                                                                | IPDS2                                                                        |
| Absorption correction                                                                                                                                                                                                                                         | Numerical ( <i>X-RED</i> and<br><i>X-SHAPE</i> ; Stoe, 2002)                 |
| $T_{\text{min}}, T_{\text{max}}$                                                                                                                                                                                                                              | 0.719, 0.812                                                                 |
| No. of measured, independent and<br>observed [ $I > 2\sigma(I)$ ] reflections                                                                                                                                                                                 | 24424, 3836, 2792                                                            |
| $R_{\text{int}}$                                                                                                                                                                                                                                              | 0.111                                                                        |
| $(\sin \theta/\lambda)_{\text{max}}$ (Å <sup>−1</sup> )                                                                                                                                                                                                       | 0.661                                                                        |
| Refinement                                                                                                                                                                                                                                                    |                                                                              |
| $R[F^2 > 2\sigma(F^2)], wR(F^2), S$                                                                                                                                                                                                                           | 0.047, 0.143, 1.03                                                           |
| No. of reflections                                                                                                                                                                                                                                            | 3836                                                                         |
| No. of parameters                                                                                                                                                                                                                                             | 195                                                                          |
| H-atom treatment                                                                                                                                                                                                                                              | H atoms treated by a mixture of<br>independent and constrained<br>refinement |
| $\Delta\rho_{\text{max}}, \Delta\rho_{\text{min}}$ (e Å <sup>−3</sup> )                                                                                                                                                                                       | 1.03, −1.76                                                                  |
| Computer programs: <i>X-AREA</i> (Stoe, 2002), <i>SHELXT2014/4</i> (Sheldrick, 2015a),<br><i>SHELXL2016/6</i> (Sheldrick, 2015b), <i>DIAMOND</i> (Brandenburg, 1999), <i>XP</i> in<br><i>SHELXTL-PC</i> (Sheldrick, 2008) and <i>publCIF</i> (Westrip, 2010). |                                                                              |
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## supporting information

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## Synthesis, crystal structure and thermal properties of *catena*-poly[[[bis(2,3-dimethylpyrazine- $\kappa N^1$ )cadmium(II)]-di- $\mu$ -chlorido] monohydrate]

Christian Näther

### Computing details

*catena*-Poly[[[bis(2,3-dimethylpyrazine- $\kappa N^1$ )cadmium(II)]-di- $\mu$ -chlorido] monohydrate]

#### Crystal data

[CdCl<sub>2</sub>(C<sub>6</sub>H<sub>8</sub>N<sub>2</sub>)<sub>2</sub>] $\cdot$ H<sub>2</sub>O

$M_r = 417.60$

Monoclinic,  $C2/c$

$a = 30.6316(15)$  Å

$b = 7.4391(3)$  Å

$c = 16.3857(8)$  Å

$\beta = 121.538(3)^\circ$

$V = 3182.3(3)$  Å<sup>3</sup>

$Z = 8$

$F(000) = 1664$

$D_x = 1.743$  Mg m<sup>-3</sup>

Mo  $K\alpha$  radiation,  $\lambda = 0.71073$  Å

Cell parameters from 22970 reflections

$\theta = 1.5\text{--}28.9^\circ$

$\mu = 1.71$  mm<sup>-1</sup>

$T = 170$  K

Block, colorless

$0.14 \times 0.12 \times 0.09$  mm

#### Data collection

IPDS-2

diffractometer

$\omega$  scans

Absorption correction: numerical

(X-Red and X-Shape; Stoe, 2002)

$T_{\min} = 0.719$ ,  $T_{\max} = 0.812$

24424 measured reflections

3836 independent reflections

2792 reflections with  $I > 2\sigma(I)$

$R_{\text{int}} = 0.111$

$\theta_{\max} = 28.0^\circ$ ,  $\theta_{\min} = 1.6^\circ$

$h = -40 \rightarrow 40$

$k = -8 \rightarrow 9$

$l = -21 \rightarrow 21$

#### Refinement

Refinement on  $F^2$

Least-squares matrix: full

$R[F^2 > 2\sigma(F^2)] = 0.047$

$wR(F^2) = 0.143$

$S = 1.03$

3836 reflections

195 parameters

0 restraints

Hydrogen site location: mixed

H atoms treated by a mixture of independent and constrained refinement

$w = 1/[\sigma^2(F_o^2) + (0.0824P)^2]$

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

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

$\Delta\rho_{\max} = 1.03$  e Å<sup>-3</sup>

$\Delta\rho_{\min} = -1.76$  e Å<sup>-3</sup>

Extinction correction: *SHELXL2016/6*

(Sheldrick, 2015b),

$F_c^* = kFc[1 + 0.001x Fc^2 \lambda^3 / \sin(2\theta)]^{-1/4}$

Extinction coefficient: 0.00097 (15)

*Special details*

**Geometry.** All esds (except the esd in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell esds are taken into account individually in the estimation of esds in distances, angles and torsion angles; correlations between esds in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell esds is used for estimating esds involving l.s. planes.

*Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters ( $\text{\AA}^2$ )*

|      | <i>x</i>     | <i>y</i>      | <i>z</i>    | $U_{\text{iso}}^*/U_{\text{eq}}$ |
|------|--------------|---------------|-------------|----------------------------------|
| Cd1  | 0.500000     | 0.73731 (5)   | 0.250000    | 0.03384 (15)                     |
| Cd2  | 0.500000     | 0.23652 (5)   | 0.250000    | 0.03428 (15)                     |
| Cl1  | 0.53794 (4)  | 0.48838 (10)  | 0.19592 (7) | 0.0374 (2)                       |
| Cl2  | 0.46474 (4)  | −0.01175 (10) | 0.31017 (7) | 0.0374 (2)                       |
| N1   | 0.58483 (13) | 0.7477 (3)    | 0.4026 (2)  | 0.0375 (7)                       |
| C1   | 0.59557 (14) | 0.7695 (4)    | 0.4922 (2)  | 0.0373 (7)                       |
| C2   | 0.64592 (14) | 0.8044 (6)    | 0.5672 (2)  | 0.0467 (8)                       |
| N2   | 0.68450 (12) | 0.8149 (5)    | 0.5519 (2)  | 0.0536 (8)                       |
| C3   | 0.67300 (14) | 0.7877 (5)    | 0.4618 (3)  | 0.0469 (8)                       |
| H3   | 0.699816     | 0.790312      | 0.448839    | 0.056*                           |
| C4   | 0.62419 (15) | 0.7566 (4)    | 0.3882 (3)  | 0.0413 (8)                       |
| H4   | 0.617810     | 0.740743      | 0.325322    | 0.050*                           |
| C5   | 0.55300 (15) | 0.7540 (4)    | 0.5117 (3)  | 0.0417 (9)                       |
| H5A  | 0.545233     | 0.873110      | 0.526423    | 0.063*                           |
| H5B  | 0.563519     | 0.673542      | 0.566231    | 0.063*                           |
| H5C  | 0.522444     | 0.705258      | 0.455015    | 0.063*                           |
| C6   | 0.65882 (17) | 0.8313 (7)    | 0.6686 (3)  | 0.0669 (12)                      |
| H6A  | 0.660295     | 0.714319      | 0.697424    | 0.100*                           |
| H6B  | 0.632439     | 0.905913      | 0.668492    | 0.100*                           |
| H6C  | 0.692140     | 0.891136      | 0.705752    | 0.100*                           |
| N11  | 0.57755 (12) | 0.2489 (3)    | 0.4142 (2)  | 0.0368 (7)                       |
| C11  | 0.62713 (14) | 0.2707 (4)    | 0.4435 (2)  | 0.0384 (8)                       |
| C12  | 0.66333 (13) | 0.3077 (6)    | 0.5407 (2)  | 0.0449 (8)                       |
| N12  | 0.65036 (12) | 0.3168 (5)    | 0.6062 (2)  | 0.0503 (7)                       |
| C13  | 0.60089 (15) | 0.2888 (5)    | 0.5756 (2)  | 0.0470 (8)                       |
| H13  | 0.590506     | 0.289731      | 0.620968    | 0.056*                           |
| C14  | 0.56478 (16) | 0.2589 (5)    | 0.4813 (3)  | 0.0431 (9)                       |
| H14  | 0.529946     | 0.244861      | 0.462766    | 0.052*                           |
| C15  | 0.64336 (14) | 0.2545 (4)    | 0.3723 (3)  | 0.0423 (8)                       |
| H15A | 0.614010     | 0.217636      | 0.310243    | 0.064*                           |
| H15B | 0.670571     | 0.164260      | 0.394365    | 0.064*                           |
| H15C | 0.656145     | 0.370793      | 0.365481    | 0.064*                           |
| C16  | 0.71882 (14) | 0.3366 (7)    | 0.5745 (3)  | 0.0619 (11)                      |
| H16A | 0.734999     | 0.408587      | 0.633216    | 0.093*                           |
| H16B | 0.721521     | 0.399934      | 0.524842    | 0.093*                           |
| H16C | 0.736137     | 0.220025      | 0.587352    | 0.093*                           |
| O1   | 0.79443 (13) | 0.8800 (6)    | 0.6842 (2)  | 0.0899 (13)                      |
| H1A  | 0.806 (2)    | 0.853 (9)     | 0.752 (5)   | 0.12 (2)*                        |
| H1B  | 0.759 (2)    | 0.864 (8)     | 0.651 (4)   | 0.108 (19)*                      |

*Atomic displacement parameters ( $\text{\AA}^2$ )*

|     | $U^{11}$    | $U^{22}$    | $U^{33}$    | $U^{12}$     | $U^{13}$     | $U^{23}$     |
|-----|-------------|-------------|-------------|--------------|--------------|--------------|
| Cd1 | 0.0430 (2)  | 0.0296 (2)  | 0.0362 (2)  | 0.000        | 0.02573 (18) | 0.000        |
| Cd2 | 0.0436 (3)  | 0.0299 (2)  | 0.0362 (2)  | 0.000        | 0.02563 (19) | 0.000        |
| Cl1 | 0.0490 (5)  | 0.0325 (5)  | 0.0419 (5)  | −0.0010 (3)  | 0.0315 (4)   | −0.0015 (3)  |
| Cl2 | 0.0484 (5)  | 0.0323 (5)  | 0.0417 (5)  | −0.0006 (3)  | 0.0307 (4)   | −0.0013 (3)  |
| N1  | 0.0498 (18) | 0.0335 (17) | 0.0373 (16) | 0.0001 (11)  | 0.0284 (14)  | −0.0009 (10) |
| C1  | 0.0463 (19) | 0.0342 (19) | 0.0381 (17) | 0.0016 (13)  | 0.0267 (16)  | 0.0032 (12)  |
| C2  | 0.053 (2)   | 0.053 (2)   | 0.0357 (16) | 0.0010 (17)  | 0.0245 (15)  | 0.0019 (16)  |
| N2  | 0.0452 (16) | 0.072 (2)   | 0.0395 (15) | −0.0004 (17) | 0.0194 (13)  | 0.0003 (16)  |
| C3  | 0.0452 (19) | 0.054 (2)   | 0.0474 (19) | 0.0022 (16)  | 0.0285 (17)  | 0.0023 (16)  |
| C4  | 0.049 (2)   | 0.044 (2)   | 0.0389 (18) | 0.0022 (13)  | 0.0281 (17)  | 0.0020 (13)  |
| C5  | 0.055 (2)   | 0.046 (2)   | 0.0363 (17) | 0.0021 (14)  | 0.0320 (17)  | 0.0028 (12)  |
| C6  | 0.066 (3)   | 0.094 (4)   | 0.0358 (18) | −0.001 (2)   | 0.0232 (18)  | −0.005 (2)   |
| N11 | 0.0425 (16) | 0.0355 (18) | 0.0387 (16) | 0.0018 (10)  | 0.0257 (14)  | 0.0027 (10)  |
| C11 | 0.0457 (19) | 0.0344 (19) | 0.0364 (17) | 0.0019 (13)  | 0.0225 (15)  | 0.0010 (12)  |
| C12 | 0.0425 (18) | 0.050 (2)   | 0.0426 (17) | 0.0039 (16)  | 0.0223 (15)  | 0.0005 (16)  |
| N12 | 0.0491 (16) | 0.062 (2)   | 0.0381 (15) | 0.0037 (15)  | 0.0213 (13)  | −0.0010 (15) |
| C13 | 0.054 (2)   | 0.055 (2)   | 0.0370 (17) | 0.0032 (17)  | 0.0274 (16)  | −0.0001 (15) |
| C14 | 0.048 (2)   | 0.045 (2)   | 0.044 (2)   | 0.0037 (14)  | 0.0300 (18)  | 0.0020 (13)  |
| C15 | 0.0422 (19) | 0.045 (2)   | 0.048 (2)   | 0.0013 (13)  | 0.0294 (18)  | 0.0007 (13)  |
| C16 | 0.045 (2)   | 0.086 (3)   | 0.049 (2)   | 0.000 (2)    | 0.0210 (17)  | −0.005 (2)   |
| O1  | 0.0568 (19) | 0.168 (4)   | 0.0464 (16) | −0.017 (2)   | 0.0282 (15)  | 0.002 (2)    |

*Geometric parameters ( $\text{\AA}$ ,  $^\circ$ )*

|                        |            |          |           |
|------------------------|------------|----------|-----------|
| Cd1—N1                 | 2.493 (3)  | C5—H5B   | 0.9800    |
| Cd1—N1 <sup>i</sup>    | 2.493 (3)  | C5—H5C   | 0.9800    |
| Cd1—Cl1 <sup>i</sup>   | 2.5773 (9) | C6—H6A   | 0.9800    |
| Cd1—Cl1                | 2.5773 (9) | C6—H6B   | 0.9800    |
| Cd1—Cl2 <sup>ii</sup>  | 2.5958 (9) | C6—H6C   | 0.9800    |
| Cd1—Cl2 <sup>iii</sup> | 2.5958 (9) | N11—C11  | 1.342 (5) |
| Cd2—N11                | 2.491 (3)  | N11—C14  | 1.348 (5) |
| Cd2—N11 <sup>i</sup>   | 2.491 (3)  | C11—C12  | 1.412 (5) |
| Cd2—Cl2                | 2.5815 (9) | C11—C15  | 1.494 (5) |
| Cd2—Cl2 <sup>i</sup>   | 2.5815 (9) | C12—N12  | 1.328 (4) |
| Cd2—Cl1                | 2.5930 (9) | C12—C16  | 1.503 (5) |
| Cd2—Cl1 <sup>i</sup>   | 2.5930 (9) | N12—C13  | 1.340 (5) |
| N1—C1                  | 1.337 (4)  | C13—C14  | 1.367 (5) |
| N1—C4                  | 1.346 (5)  | C13—H13  | 0.9500    |
| C1—C2                  | 1.404 (5)  | C14—H14  | 0.9500    |
| C1—C5                  | 1.500 (5)  | C15—H15A | 0.9800    |
| C2—N2                  | 1.332 (5)  | C15—H15B | 0.9800    |
| C2—C6                  | 1.506 (4)  | C15—H15C | 0.9800    |
| N2—C3                  | 1.342 (5)  | C16—H16A | 0.9800    |
| C3—C4                  | 1.363 (5)  | C16—H16B | 0.9800    |
| C3—H3                  | 0.9500     | C16—H16C | 0.9800    |

|                                           |             |               |           |
|-------------------------------------------|-------------|---------------|-----------|
| C4—H4                                     | 0.9500      | O1—H1A        | 1.00 (7)  |
| C5—H5A                                    | 0.9800      | O1—H1B        | 0.94 (6)  |
| N1—Cd1—N1 <sup>i</sup>                    | 176.45 (11) | N1—C4—C3      | 121.5 (3) |
| N1—Cd1—Cl1 <sup>i</sup>                   | 95.65 (7)   | N1—C4—H4      | 119.3     |
| N1 <sup>i</sup> —Cd1—Cl1 <sup>i</sup>     | 86.91 (7)   | C3—C4—H4      | 119.3     |
| N1—Cd1—Cl1                                | 86.91 (7)   | C1—C5—H5A     | 109.5     |
| N1 <sup>i</sup> —Cd1—Cl1                  | 95.65 (7)   | C1—C5—H5B     | 109.5     |
| Cl1 <sup>i</sup> —Cd1—Cl1                 | 88.14 (4)   | H5A—C5—H5B    | 109.5     |
| N1—Cd1—Cl2 <sup>ii</sup>                  | 86.33 (7)   | C1—C5—H5C     | 109.5     |
| N1 <sup>i</sup> —Cd1—Cl2 <sup>ii</sup>    | 91.11 (7)   | H5A—C5—H5C    | 109.5     |
| Cl1 <sup>i</sup> —Cd1—Cl2 <sup>ii</sup>   | 178.02 (2)  | H5B—C5—H5C    | 109.5     |
| Cl1—Cd1—Cl2 <sup>ii</sup>                 | 91.95 (3)   | C2—C6—H6A     | 109.5     |
| N1—Cd1—Cl2 <sup>iii</sup>                 | 91.11 (7)   | C2—C6—H6B     | 109.5     |
| N1 <sup>i</sup> —Cd1—Cl2 <sup>iii</sup>   | 86.33 (7)   | H6A—C6—H6B    | 109.5     |
| Cl1 <sup>i</sup> —Cd1—Cl2 <sup>iii</sup>  | 91.95 (3)   | C2—C6—H6C     | 109.5     |
| Cl1—Cd1—Cl2 <sup>iii</sup>                | 178.02 (2)  | H6A—C6—H6C    | 109.5     |
| Cl2 <sup>ii</sup> —Cd1—Cl2 <sup>iii</sup> | 88.03 (4)   | H6B—C6—H6C    | 109.5     |
| N11—Cd2—N11 <sup>i</sup>                  | 175.76 (11) | C11—N11—C14   | 117.3 (3) |
| N11—Cd2—Cl2                               | 88.11 (7)   | C11—N11—Cd2   | 130.7 (2) |
| N11 <sup>i</sup> —Cd2—Cl2                 | 94.93 (7)   | C14—N11—Cd2   | 111.3 (3) |
| N11—Cd2—Cl2 <sup>i</sup>                  | 94.93 (7)   | N11—C11—C12   | 120.1 (3) |
| N11 <sup>i</sup> —Cd2—Cl2 <sup>i</sup>    | 88.11 (7)   | N11—C11—C15   | 119.2 (3) |
| Cl2—Cd2—Cl2 <sup>i</sup>                  | 88.64 (4)   | C12—C11—C15   | 120.7 (3) |
| N11—Cd2—Cl1                               | 89.88 (7)   | N12—C12—C11   | 122.1 (3) |
| N11 <sup>i</sup> —Cd2—Cl1                 | 87.06 (7)   | N12—C12—C16   | 117.0 (3) |
| Cl2—Cd2—Cl1                               | 177.93 (2)  | C11—C12—C16   | 120.9 (3) |
| Cl2 <sup>i</sup> —Cd2—Cl1                 | 91.98 (3)   | C12—N12—C13   | 116.5 (3) |
| N11—Cd2—Cl1 <sup>i</sup>                  | 87.06 (7)   | N12—C13—C14   | 122.5 (3) |
| N11 <sup>i</sup> —Cd2—Cl1 <sup>i</sup>    | 89.87 (7)   | N12—C13—H13   | 118.8     |
| Cl2—Cd2—Cl1 <sup>i</sup>                  | 91.98 (3)   | C14—C13—H13   | 118.8     |
| Cl2 <sup>i</sup> —Cd2—Cl1 <sup>i</sup>    | 177.93 (2)  | N11—C14—C13   | 121.4 (4) |
| Cl1—Cd2—Cl1 <sup>i</sup>                  | 87.47 (4)   | N11—C14—H14   | 119.3     |
| Cd1—Cl1—Cd2                               | 92.20 (3)   | C13—C14—H14   | 119.3     |
| Cd2—Cl2—Cd1 <sup>iv</sup>                 | 91.66 (3)   | C11—C15—H15A  | 109.5     |
| C1—N1—C4                                  | 117.3 (3)   | C11—C15—H15B  | 109.5     |
| C1—N1—Cd1                                 | 129.4 (3)   | H15A—C15—H15B | 109.5     |
| C4—N1—Cd1                                 | 112.7 (2)   | C11—C15—H15C  | 109.5     |
| N1—C1—C2                                  | 120.6 (3)   | H15A—C15—H15C | 109.5     |
| N1—C1—C5                                  | 118.8 (3)   | H15B—C15—H15C | 109.5     |
| C2—C1—C5                                  | 120.5 (3)   | C12—C16—H16A  | 109.5     |
| N2—C2—C1                                  | 121.5 (3)   | C12—C16—H16B  | 109.5     |
| N2—C2—C6                                  | 116.9 (3)   | H16A—C16—H16B | 109.5     |
| C1—C2—C6                                  | 121.6 (3)   | C12—C16—H16C  | 109.5     |
| C2—N2—C3                                  | 116.7 (3)   | H16A—C16—H16C | 109.5     |
| N2—C3—C4                                  | 122.3 (3)   | H16B—C16—H16C | 109.5     |
| N2—C3—H3                                  | 118.8       | H1A—O1—H1B    | 104 (4)   |
| C4—C3—H3                                  | 118.8       |               |           |

|              |            |                 |            |
|--------------|------------|-----------------|------------|
| C4—N1—C1—C2  | 1.4 (5)    | C14—N11—C11—C12 | −1.8 (5)   |
| Cd1—N1—C1—C2 | −168.3 (3) | Cd2—N11—C11—C12 | 167.8 (2)  |
| C4—N1—C1—C5  | −177.9 (3) | C14—N11—C11—C15 | 177.9 (3)  |
| Cd1—N1—C1—C5 | 12.4 (4)   | Cd2—N11—C11—C15 | −12.5 (4)  |
| N1—C1—C2—N2  | −0.7 (6)   | N11—C11—C12—N12 | 2.0 (6)    |
| C5—C1—C2—N2  | 178.5 (4)  | C15—C11—C12—N12 | −177.6 (4) |
| N1—C1—C2—C6  | 179.4 (4)  | N11—C11—C12—C16 | −178.6 (4) |
| C5—C1—C2—C6  | −1.3 (6)   | C15—C11—C12—C16 | 1.7 (6)    |
| C1—C2—N2—C3  | −1.0 (6)   | C11—C12—N12—C13 | 0.1 (6)    |
| C6—C2—N2—C3  | 178.9 (4)  | C16—C12—N12—C13 | −179.2 (4) |
| C2—N2—C3—C4  | 2.1 (6)    | C12—N12—C13—C14 | −2.4 (6)   |
| C1—N1—C4—C3  | −0.4 (5)   | C11—N11—C14—C13 | −0.4 (5)   |
| Cd1—N1—C4—C3 | 171.0 (3)  | Cd2—N11—C14—C13 | −172.0 (3) |
| N2—C3—C4—N1  | −1.4 (6)   | N12—C13—C14—N11 | 2.7 (6)    |

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

### Hydrogen-bond geometry ( $\text{\AA}$ , $^\circ$ )

| $D-H\cdots A$                       | $D-H$    | $H\cdots A$ | $D\cdots A$ | $D-H\cdots A$ |
|-------------------------------------|----------|-------------|-------------|---------------|
| O1—H1A $\cdots$ N12 <sup>v</sup>    | 1.00 (7) | 2.00 (7)    | 2.965 (4)   | 164 (5)       |
| O1—H1B $\cdots$ N2                  | 0.94 (6) | 2.02 (7)    | 2.939 (5)   | 165 (5)       |
| C3—H3 $\cdots$ O1 <sup>vi</sup>     | 0.95     | 2.61        | 3.282 (5)   | 128           |
| C4—H4 $\cdots$ Cl1                  | 0.95     | 2.93        | 3.490 (4)   | 119           |
| C4—H4 $\cdots$ Cl2 <sup>ii</sup>    | 0.95     | 2.97        | 3.420 (4)   | 110           |
| C5—H5B $\cdots$ Cl1 <sup>vii</sup>  | 0.98     | 2.88        | 3.747 (3)   | 147           |
| C5—H5C $\cdots$ Cl1 <sup>i</sup>    | 0.98     | 2.71        | 3.650 (4)   | 160           |
| C5—H5C $\cdots$ Cl2 <sup>iii</sup>  | 0.98     | 2.97        | 3.451 (4)   | 112           |
| C13—H13 $\cdots$ Cl1 <sup>vii</sup> | 0.95     | 2.98        | 3.786 (4)   | 143           |
| C14—H14 $\cdots$ Cl1 <sup>i</sup>   | 0.95     | 2.95        | 3.419 (4)   | 112           |
| C14—H14 $\cdots$ Cl2                | 0.95     | 2.94        | 3.510 (4)   | 120           |
| C15—H15A $\cdots$ Cl1               | 0.98     | 2.90        | 3.470 (4)   | 118           |
| C15—H15A $\cdots$ Cl2 <sup>i</sup>  | 0.98     | 2.76        | 3.670 (4)   | 154           |

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