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# Metal halide coordination compounds with quinazolin-4(3*H*)-one

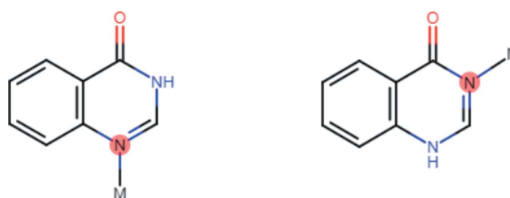
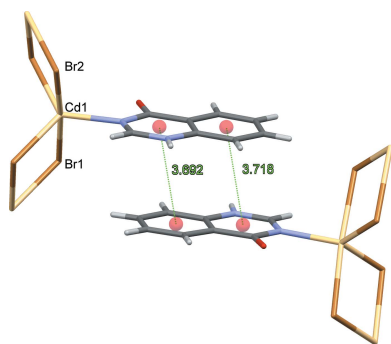
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Three coordination compounds of quinazolin-4(3*H*)-one (**quinoz**; C<sub>8</sub>H<sub>6</sub>N<sub>2</sub>O) with divalent group 12 halides are reported. In all complexes, coordination occurs *via* the nitrogen atom *ortho* to the quinazolinone carbonyl group. In the two chain polymers with composition [MX<sub>2</sub>(**quinoz**)], *viz.* (*M* = Cd, *X* = Br), *catena*-poly[[[quinazolin-4(3*H*)-one- $\kappa$ N<sup>3</sup>]cadmium(II)]-di- $\mu$ -bromido], [CdBr<sub>2</sub>(C<sub>8</sub>H<sub>6</sub>N<sub>2</sub>O)]<sub>n</sub> (**I**), and *M* = Hg, *X* = Cl, *catena*-poly[[[quinazolin-4(3*H*)-one- $\kappa$ N<sup>3</sup>]mercury(II)]-di- $\mu$ -chlorido], [HgCl<sub>2</sub>(C<sub>8</sub>H<sub>6</sub>N<sub>2</sub>O)]<sub>n</sub> (**II**), the divalent cations are five-coordinate, with four bridging halide and one terminal **quinoz** ligand. The Cd<sup>II</sup> atom in (**I**) has an almost trigonal-bipyramidal coordination environment, whereas the Hg<sup>II</sup> atom in (**II**) has a more distorted coordination environment. Likewise, the halide bridges in (**II**) are significantly more asymmetric than in (**I**). In both (**I**) and (**II**), **quinoz** ligands at adjacent cations along each strand are oriented in opposite directions, and the organic ligands of neighboring strands interdigitate with resulting  $\pi$ - $\pi$  interactions. In contrast to the halide-bridged chain polymers (**I**) and (**II**), the adduct of **quinoz** with CdI<sub>2</sub> is the tetrahedral complex [CdI<sub>2</sub>(**quinoz**)<sub>2</sub>], diiodidobis[quinazolin-4(3*H*)-one- $\kappa$ N<sup>3</sup>]cadmium(II), [CdI<sub>2</sub>(C<sub>16</sub>H<sub>12</sub>N<sub>4</sub>O<sub>2</sub>)], (**III**). The Cd<sup>II</sup> atom in this discrete complex is located on a twofold rotation axis. Disorder in (**III**) is reflected in an alternative minority orientation of the molecules for which the iodine sites closely match the position of the majority orientation. In view of the low site occupancy of only 0.0318 (8) Å, only the Cd<sup>II</sup> position for this alternative orientation was taken into account during refinement. In all three compounds, classical N—H···O hydrogen bonds with donor-acceptor distances of *ca* 2.9 Å occur; they link the polymer chains in (**I**) and (**II**) into di-periodic networks and connect adjacent discrete complexes in (**III**) to mono-periodic strands.

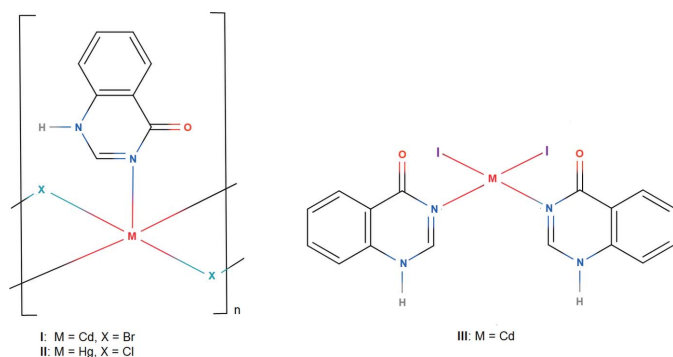
## 1. Chemical context

4(3*H*)-Quinazolinone (**quinoz**) can act as ligand for metal ions in different coordination modes. Both coordination through the nitrogen atom *para* (mode 1) and, after tautomerization, *via* the nitrogen atom *ortho* to the quinazolinone carbonyl group (mode 2) have been observed (Fig. 1). An Ag<sup>I</sup> coord-



**Figure 1** mode 1 mode 2  
The two possible types of coordination modes for the quinazolin-4-one ligand in metal complexes.

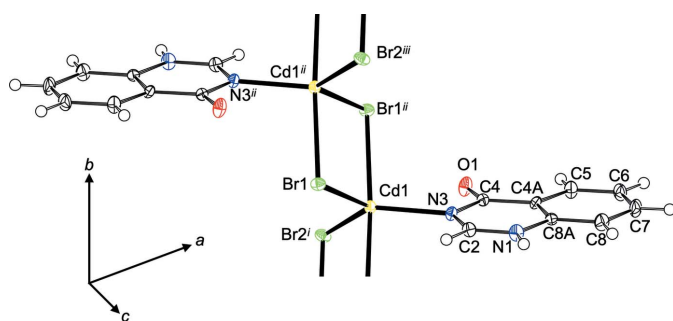
ination compound (Li *et al.*, 2015) provides an example for the co-existence of both binding modes in the same crystal structure. Earlier studies on the reaction products of cadmium chloride or bromide with quinazolin-4(3*H*)-one have shown that the **quinoz** ligand may interact with Cd<sup>II</sup> cations *via* the *para* nitrogen atom, *i.e.* according to mode 1. Four bridging halides in the equatorial plane and two **quinoz** ligands in a *trans*-axial arrangement give rise to a pseudo-octahedral coordination environment around the metal cation (Turgunov & Englert, 2010; Turgunov *et al.*, 2010; Shomurotova *et al.*, 2012; Đaković *et al.*, 2018).



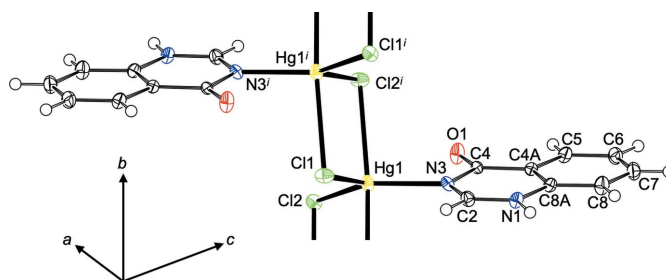
We here report three other examples for coordination according to mode 2, namely the adducts of quinoz with CdBr<sub>2</sub> (**I**), HgCl<sub>2</sub> (**II**) and CdI<sub>2</sub> (**III**). The influence of different halide ligands on the coordination environment of divalent cations with N-donor co-ligands has been discussed in detail (Hu & Englert, 2001, 2002; Hu *et al.*, 2003).

## 2. Structural commentary

The asymmetric unit of (**I**) consists of a Cd<sup>II</sup> cation, two Br<sup>-</sup> ligands and one **quinoz** ligand attached in mode 2 (Fig. 2). The cation adopts a coordination number of 5 and is characterized by a  $\tau_5$  descriptor (Addison *et al.*, 1984) of 0.80. In an alternative description (Holmes, 1984), its shape corresponds to only 5% distortion along a hypothetical pathway from *D*<sub>3h</sub> to *C*<sub>4v</sub>. Both qualifiers consistently assign this shape as trigonal

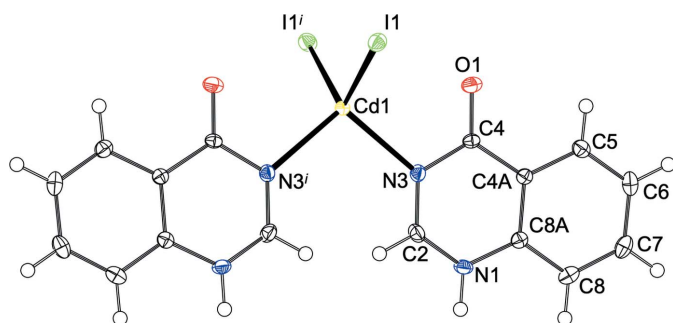


**Figure 2**  
Section of polymer (**I**) with the atom-numbering scheme. Displacement ellipsoids are drawn at the 50% probability level, H atoms are shown as spheres of arbitrary radius. [Symmetry codes: (i)  $-x, 1 - y, 1 - z$ ; (ii)  $-x, 2 - y, 1 - z$ ; (iii)  $x, 1 + y, z$ ].



**Figure 3**  
Section of polymer (**II**) with the atom-numbering scheme. Displacement ellipsoids are drawn at the 50% probability level, H atoms are shown as spheres of arbitrary radius. [Symmetry code: (i)  $2 - x, 1 - y, -z$ ].

bipyramidal, with the **quinoz** ligand in an equatorial position. The equatorial plane defined by Cd1, Br1, Br2<sup>i</sup> [symmetry code: (i)  $-x, 1 - y, 1 - z$ ] and N3 and the least-squares plane through the **quinoz** ligand subtend a dihedral angle of 38.32 (13)°. The bromido ligands act as rather symmetric bridges between neighboring cations, thus giving rise to a chain polymer extending along [010]. Additional details concerning the crystal structure of (**I**) are best discussed together with the related derivative (**II**) (Fig. 3). Both compounds share the same composition [MX<sub>2</sub>(**quinoz**)], with bridging halide ligands between neighboring divalent group 12 cations at a distance slightly less than 4 Å. The mercury compound (**II**) shows a considerably more distorted coordination environment than its cadmium congener (**I**): On the one hand, the coordination environment about the cation is less regular; both the  $\tau_5$  (0.56) and the Holmes descriptor (23%) assign a shape in-between trigonal bipyramidal and square pyramidal. On the other hand, the chlorido bridges in (**II**) are significantly more asymmetric than the bromido linkers in (**I**). Even more asymmetric halide bridges have been observed in the bis adduct of 1,2,3,9-tetrahydro-pyrrolo[2,1-*b*]quinazolin-9-one to HgCl<sub>2</sub> (Turgunov *et al.*, 2011). Both chain polymers (**I**) and (**II**) fit well into the wider context of halide-bridged chain polymers. The adducts of donor ligands to CdBr<sub>2</sub> or HgCl<sub>2</sub> mostly display coordination numbers of 5 or 6 and have bridging halide ligands. For such bromido-bridged Cd<sup>II</sup> strands, similar Cd–Cd separations as in (**I**) [Cd1<sup>i</sup>···Cd1<sup>ii</sup> = 3.8667 (10) and Cd1<sup>i</sup>···Cd1<sup>iii</sup> = 3.9051 (10) Å; symmetry codes: (i)  $-x, 2 - y, 1 - z$ ; (ii)  $-x, 1 - y, 1 - z$ ] have been reported (Hu & Englert, 2002; Merckens *et al.*, 2014; Hu *et al.*, 2003). The Hg–Hg separations [Hg1<sup>i</sup>···Hg1<sup>i</sup> = 3.7881 (6) and Hg1<sup>i</sup>···Hg1<sup>ii</sup> = 3.8827 (6) Å, symmetry codes: (i)  $2 - x, 1 - y, -z$ ; (ii)  $2 - x, -y, -z$ ] in (**II**) are comparable to those encountered in related chlorido-bridged polymers (Hu *et al.*, 2007; Truong *et al.*, 2017; van Terwingen *et al.*, 2021; Merckens *et al.*, 2010). A different situation arises for (**III**) (Fig. 4): for the bis(ligand) adduct of CdI<sub>2</sub>, a discrete complex may be expected and is indeed encountered. The CSD database (Groom *et al.*, 2016) contains only a few structures for six-coordinated Cd with four iodido and two additional arbitrary ligands, for example a di-periodic structure with bipyridyl ligands in one and iodido bridges in a second direction (Hu *et al.*, 2003). In contrast, more than 600 hits for tetrahedrally

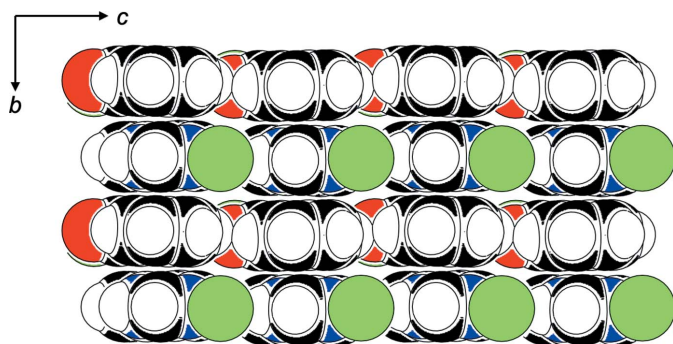

**Figure 4**

Molecular structure of **(III)** with the atom-numbering scheme; the minor disorder of the Cd site is shown in Fig. 9 and has been omitted here. Displacement ellipsoids are drawn at the 50% probability level, H atoms are shown as spheres of arbitrary radius. [Symmetry code: (i)  $-x, y, \frac{1}{2} - z$ ]

coordinated Cd<sup>II</sup> with two iodido and two additional ligands have been documented, and **(III)** falls into this category. The crystal structure of **(III)** has been previously reported by Đaković *et al.* (2018). Our present report takes a minor disorder into account, which explains an otherwise unaccounted high residual electron density; details are provided in the *Refinement* section. In **(III)**, the cation resides on a twofold rotation axis of space group *C2/c*, Wyckoff position 4e. Its coordination environment is characterized by a  $\tau_4$  descriptor (Yang *et al.*, 2007) of 0.93, corresponding to an almost undistorted tetrahedron.

### 3. Supramolecular features

Classical N–H···O hydrogen bonds exist in structures **(I)**–**(III)**. They link the NH group to the carbonyl oxygen atom of a neighboring **quinox** ligand [parallel to [001] for **(I)** and **(II)**, and parallel to [010] for **(III)**], and involve donor–acceptor distances around 2.9 Å. Numerical details of the hydrogen-bonding interactions are compiled in Tables 1–3. In the coordination polymers **(I)** and **(II)**, **quinox** ligands of adjacent strands interdigitate. The distances between neighboring coplanar organic ligands amount to one half of the lattice parameter *b*, *i.e.* 3.5–3.6 Å and suggest  $\pi$ – $\pi$  stacking. As an


**Figure 5**

Space-filling model for **(I)** (*PLUTO*; Spek 2009) as viewed along [100]; Br atoms have been omitted. Color code: Cd green, C black, O red, N blue, H white.

**Table 1**

Hydrogen-bond geometry (Å, °) for **(I)**.

<i>D</i> –H··· <i>A</i>	<i>D</i> –H	H··· <i>A</i>	<i>D</i> ··· <i>A</i>	<i>D</i> –H··· <i>A</i>
N1–H1···O1 <sup>i</sup>	0.87 (3)	2.10 (3)	2.917 (6)	155 (6)

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

**Table 2**

Hydrogen-bond geometry (Å, °) for **(II)**.

<i>D</i> –H··· <i>A</i>	<i>D</i> –H	H··· <i>A</i>	<i>D</i> ··· <i>A</i>	<i>D</i> –H··· <i>A</i>
N1–H1···O1 <sup>i</sup>	0.89 (4)	2.11 (4)	2.935 (8)	155 (7)

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

**Table 3**

Hydrogen-bond geometry (Å, °) for **(III)**.

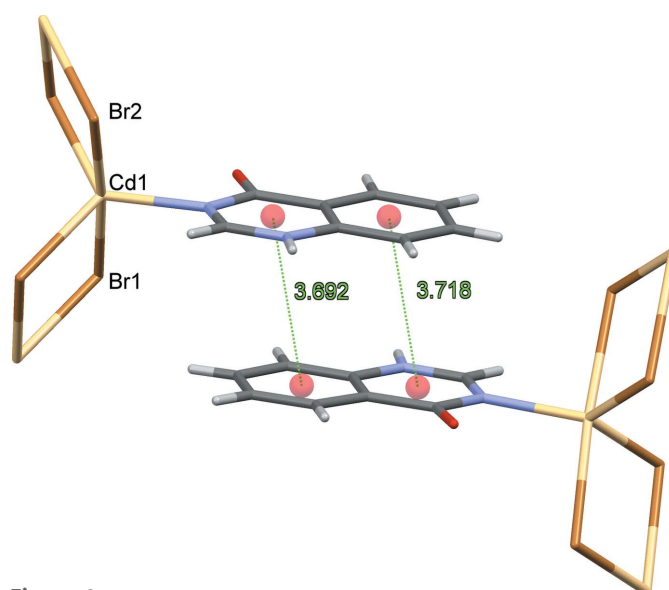
<i>D</i> –H··· <i>A</i>	<i>D</i> –H	H··· <i>A</i>	<i>D</i> ··· <i>A</i>	<i>D</i> –H··· <i>A</i>
N1–H1A···O1 <sup>i</sup>	0.93 (3)	1.97 (3)	2.893 (3)	168 (4)

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

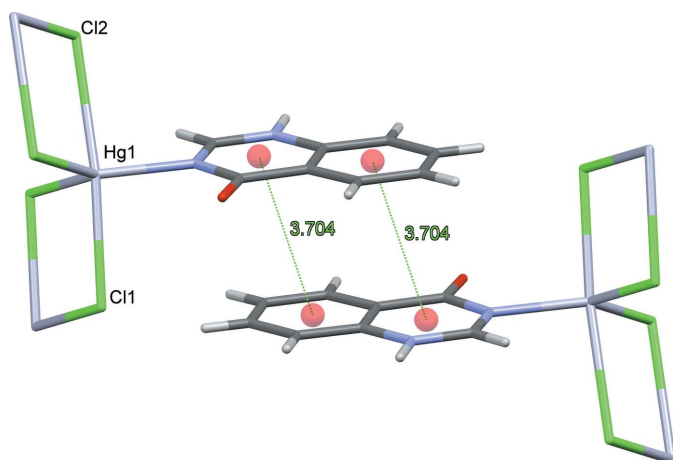
example, a space-filling model for **(I)** (Fig. 5) shows the close approach between organic **quinox** ligands on neighboring strands. An analysis with *PLATON* (Spek, 2020) gives numerical values of  $\pi$ – $\pi$  stacking interactions observed between two parallel **quinox** ligands for crystals of **(I)**–**(III)**: Cg(pyrimidine ring)···Cg(benzene ring) distances are 3.6923 (3) Å (slippage 0.843 Å) and 3.718 (3) Å (0.906 Å) in **(I)**, 3.7042 (4) Å (1.003 Å) in **(II)** and 3.5578 (14) Å (1.185 Å) in **(III)** (Figs. 6–8).

### 4. Synthesis and crystallization

Compound **(I)**. 70 mg (0.2 mmol) of cadmium bromide tetrahydrate were dissolved in a mixture of 4 ml of ethanol and 1 ml of water. 60 mg (0.4 mmol) of quinoxalin-4(3*H*)-one


**Figure 6**

The relevant  $\pi$ – $\pi$  interactions in the crystal structure of **(I)**.



**Figure 7**  
The relevant  $\pi$ - $\pi$  interactions in the crystal structure of **(II)**.

dissolved in 5 ml of ethanol were added to the cadmium bromide solution. Crystals started to precipitate after a few minutes, and colorless prismatic crystals suitable for single-crystal X-ray diffraction analysis formed within 2–3 h.

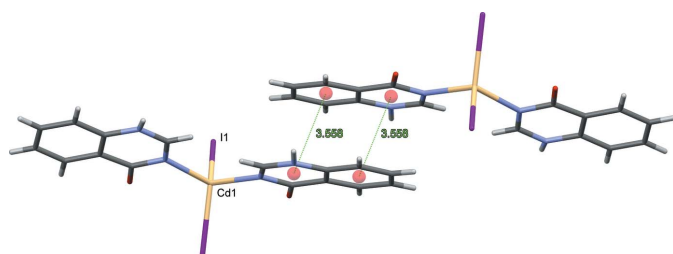
**Compound (II)**. 54.3 mg (0.2 mmol) of  $\text{HgCl}_2$  were dissolved in  $\sim 3$  ml acetone. 30 mg (0.2 mmol) of quinazolin-4(3*H*)-one were dissolved in 3 ml of acetone under mild heating, and the resulting solution was added to the  $\text{HgCl}_2$  solution. Colorless prismatic crystals suitable for X-ray diffraction analysis formed within seconds.

**Compound (III)**: 73 mg (0.2 mmol) of  $\text{CdI}_2$  were dissolved in 1 ml of ethanol. 60 mg (0.4 mmol) of the ligand were dissolved in 4 ml of ethanol under mild heating, and the resulting solution was added to the  $\text{CdI}_2$  solution. After slow evaporation of the solvent at ambient temperature for several days, colorless single crystals suitable for X-ray diffraction analysis were obtained.

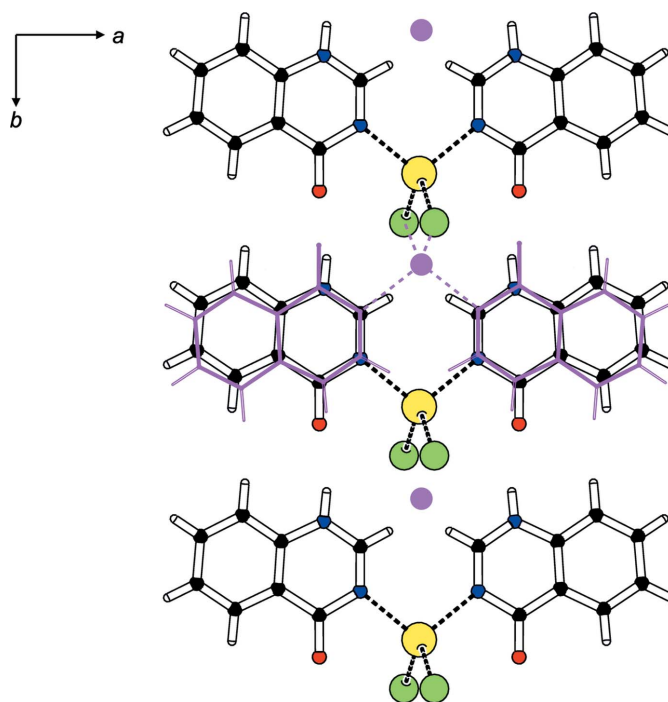
## 5. Refinement

Crystal data, data collection and structure refinement details are summarized in Table 4. Positional parameters for H atoms attached to N atoms were refined, H atoms bonded to carbon were introduced in calculated positions and treated as riding on their parent atoms.

Several crystals of **(I)** were tested and proved to be twinned; two domains of roughly equal volume are related by a  $180^\circ$  rotation about the *c* axis. The specimen selected for intensity



**Figure 8**  
The relevant  $\pi$ - $\pi$  interactions in the crystal structure of **(III)**.



**Figure 9**  
Disorder in **(III)**. The alternative Cd site ( $\text{Cd}_2$ ) is shown as a magenta-colored sphere. For clarity, the alternative ligand orientations are also shown in magenta. However, they have not been revealed experimentally and were not taken into account during refinement.

data collection showed *ca* 12000 overlapped out of a total of 65000 reflections. Final refined component fractions amounted to 0.5569 (8):0.4431 (8). Crystals of **(II)** were also twinned by non-merohedry. Here, two domains of roughly equal volume are related by a  $180^\circ$  rotation about the *b* axis. In the selected crystal, two domains contributed to *ca* 2000 overlapped out of a total of *ca* 14000 reflections. Final refined component fractions amounted to 0.5178 (9):0.4822 (9). The crystal selected for intensity data collection for **(III)** was a single crystal. After completion of the structure model, a difference-Fourier map showed a local density maximum of *ca* 5 electrons/ $\text{\AA}^3$  not associated with any atom site. This position subtended distances to the iodine atoms similar to  $\text{Cd1}-\text{I1}$ . We suggest that this residual electron density represents an alternative Cd site. In the final refinement, the sum of the site occupancies for the positionally disordered Cd sites was constrained to unity, and both sites were constrained to share the same anisotropic displacement parameters. Fig. 9 explains the arrangement of the molecules in both alternative orientations; the minority orientation is depicted in magenta. As the minority Cd site refined to an occupancy of only 0.0318 (8) and the iodine ligands for both orientations closely overlap, no attempt was made to detect and refine the alternative sites for the light atoms associated with the **quinoz** ligand. Interestingly, the authors of the previous crystal-structure determination of **(III)** (Đaković *et al.*, 2018) encountered the same local density maximum (but without modeling the disorder). Hence, the disorder appears to be a feature of the crystal structure and not of the individual crystal chosen for the data collection.

**Table 4**  
Experimental details.

	(I)	(II)	(III)
Crystal data			
Chemical formula	[CdBr <sub>2</sub> (C <sub>8</sub> H <sub>6</sub> N <sub>2</sub> O)]	[HgCl <sub>2</sub> (C <sub>8</sub> H <sub>6</sub> N <sub>2</sub> O)]	[CdI <sub>2</sub> (C <sub>16</sub> H <sub>12</sub> N <sub>4</sub> O <sub>2</sub> )]
<i>M<sub>r</sub></i>	418.37	417.64	658.50
Crystal system, space group	Monoclinic, <i>P</i> 2 <sub>1</sub> / <i>c</i>	Triclinic, <i>P</i> $\bar{1}$	Monoclinic, <i>C</i> 2/ <i>c</i>
Temperature (K)	100	100	100
<i>a</i> , <i>b</i> , <i>c</i> (Å)	10.7930 (11), 7.2019 (7), 13.7605 (14)	6.8191 (8), 7.0735 (8), 10.4659 (12)	22.242 (3), 6.8450 (9), 13.3702 (17)
$\alpha$ , $\beta$ , $\gamma$ (°)	90, 100.4705 (18), 90	85.718 (2), 80.7887 (19), 89.152 (2)	90, 118.8220 (16), 90
<i>V</i> (Å <sup>3</sup> )	1051.79 (18)	496.92 (10)	1783.4 (4)
<i>Z</i>	4	2	4
Radiation type	Mo <i>K</i> $\alpha$	Mo <i>K</i> $\alpha$	Mo <i>K</i> $\alpha$
$\mu$ (mm <sup>-1</sup> )	9.64	15.99	4.70
Crystal size (mm)	0.25 × 0.10 × 0.05	0.04 × 0.03 × 0.03	0.12 × 0.10 × 0.04
Data collection			
Diffractometer	Bruker D8 goniometer with APEX CCD detector	Bruker D8 goniometer with APEX CCD detector	Bruker D8 goniometer with APEX CCD detector
Absorption correction	Multi-scan ( <i>TWINABS</i> ; Bruker, 2014)	Multi-scan ( <i>TWINABS</i> ; Bruker, 2014)	Multi-scan ( <i>SADABS</i> ; Bruker, 2014)
<i>T</i> <sub>min</sub> , <i>T</i> <sub>max</sub>	0.446, 0.746	0.302, 0.433	0.544, 0.746
No. of measured, independent and observed [ <i>I</i> > 2 $\sigma$ ( <i>I</i> )] reflections	65521, 5868, 4717	14434, 5037, 4693	13210, 2687, 2519
<i>R</i> <sub>int</sub> ( <i>sin</i> $\theta$ / $\lambda$ ) <sub>max</sub> (Å <sup>-1</sup> )	0.089 0.718	0.050 0.709	0.024 0.717
Refinement			
<i>R</i> [ <i>F</i> <sup>2</sup> > 2 $\sigma$ ( <i>F</i> <sup>2</sup> )], <i>wR</i> ( <i>F</i> <sup>2</sup> ), <i>S</i>	0.039, 0.076, 1.04	0.036, 0.076, 1.08	0.026, 0.063, 1.12
No. of reflections	5868	5037	2687
No. of parameters	132	132	119
No. of restraints	1	1	2
H-atom treatment	H atoms treated by a mixture of independent and constrained refinement	H atoms treated by a mixture of independent and constrained refinement	H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{\max}$ , $\Delta\rho_{\min}$ (e Å <sup>-3</sup> )	1.13, -0.96	1.54, -1.49	3.17, -0.50

Computer programs: *APEX2* and *SAINT* (Bruker, 2014), *SHELXT* (Sheldrick, 2015a), *SHELXL* (Sheldrick, 2015b), *PLATON* (Spek, 2020) and *pubCIF* (Westrip, 2010).

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## supporting information

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Metal halide coordination compounds with quinazolin-4(3*H*)-one

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## Computing details

For all structures, data collection: *APEX2* (Bruker, 2014); cell refinement: *S SAINT* (Bruker, 2014); data reduction: *S SAINT* (Bruker, 2014); program(s) used to solve structure: *SHELXT* (Sheldrick, 2015a); program(s) used to refine structure: *SHELXL* (Sheldrick, 2015b); molecular graphics: *PLATON* (Spek, 2020); software used to prepare material for publication: *publCIF* (Westrip, 2010).

*catena*-Poly[[[quinazolin-4(3*H*)-one- $\kappa$ N<sup>3</sup>]cadmium(II)]-di- $\mu$ -bromido] (I)

## Crystal data

[CdBr<sub>2</sub>(C<sub>8</sub>H<sub>6</sub>N<sub>2</sub>O)]

$M_r = 418.37$

Monoclinic, *P2<sub>1</sub>/c*

$a = 10.7930$  (11) Å

$b = 7.2019$  (7) Å

$c = 13.7605$  (14) Å

$\beta = 100.4705$  (18)°

$V = 1051.79$  (18) Å<sup>3</sup>

$Z = 4$

$F(000) = 776$

$D_x = 2.642$  Mg m<sup>-3</sup>

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

Cell parameters from 3537 reflections

$\theta = 3.0$ – $26.0$ °

$\mu = 9.64$  mm<sup>-1</sup>

$T = 100$  K

Rod, colourless

$0.25 \times 0.10 \times 0.05$  mm

## Data collection

Bruker D8 goniometer with APEX CCD detector diffractometer

Radiation source: Incoatec microsource

Multilayer optics monochromator

$\omega$  scans

Absorption correction: multi-scan

(*TWINABS*; Bruker, 2014)

$T_{\min} = 0.446$ ,  $T_{\max} = 0.746$

65521 measured reflections

5868 independent reflections

4717 reflections with  $I > 2\sigma(I)$

$R_{\text{int}} = 0.089$

$\theta_{\max} = 30.7$ °,  $\theta_{\min} = 3.0$ °

$h = -15$ → $14$

$k = 0$ → $10$

$l = 0$ → $19$

## Refinement

Refinement on  $F^2$

Least-squares matrix: full

$R[F^2 > 2\sigma(F^2)] = 0.039$

$wR(F^2) = 0.076$

$S = 1.04$

5868 reflections

132 parameters

1 restraint

Primary atom site location: structure-invariant direct methods

Hydrogen site location: mixed

H atoms treated by a mixture of independent and constrained refinement

$w = 1/[\sigma^2(F_o^2) + (0.026P)^2 + 1.8P]$

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

$(\Delta/\sigma)_{\max} = 0.002$

$\Delta\rho_{\max} = 1.13$  e Å<sup>-3</sup>

$\Delta\rho_{\min} = -0.96$  e Å<sup>-3</sup>

*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.

**Refinement.** Refined as a 2-component twin.

*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	0.06536 (3)	0.75144 (5)	0.52545 (2)	0.01158 (8)
Br1	-0.12368 (4)	0.94278 (7)	0.57455 (4)	0.01378 (11)
Br2	-0.01900 (5)	0.45101 (7)	0.63243 (4)	0.01525 (11)
O1	0.3587 (3)	0.7297 (5)	0.5233 (3)	0.0182 (8)
N1	0.3312 (4)	0.7386 (6)	0.8091 (3)	0.0145 (8)
H1	0.315 (6)	0.738 (8)	0.869 (2)	0.029 (17)*
C2	0.2323 (4)	0.7393 (7)	0.7372 (4)	0.0138 (9)
H2	0.151110	0.739229	0.754639	0.017*
N3	0.2391 (4)	0.7401 (6)	0.6423 (3)	0.0122 (8)
C4	0.3551 (4)	0.7349 (7)	0.6125 (4)	0.0126 (9)
C4A	0.4672 (4)	0.7350 (7)	0.6901 (4)	0.0117 (9)
C5	0.5890 (5)	0.7309 (7)	0.6692 (4)	0.0175 (10)
H5	0.600677	0.726430	0.602429	0.021*
C6	0.6918 (5)	0.7333 (8)	0.7441 (4)	0.0197 (11)
H6	0.774211	0.731509	0.728953	0.024*
C7	0.6761 (5)	0.7385 (8)	0.8425 (4)	0.0199 (11)
H7	0.747907	0.741545	0.893885	0.024*
C8	0.5571 (5)	0.7391 (8)	0.8656 (4)	0.0187 (11)
H8	0.546255	0.739904	0.932618	0.022*
C8A	0.4523 (4)	0.7384 (7)	0.7886 (3)	0.0122 (9)

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

	$U^{11}$	$U^{22}$	$U^{33}$	$U^{12}$	$U^{13}$	$U^{23}$
Cd1	0.01016 (15)	0.01311 (15)	0.01124 (16)	0.00003 (14)	0.00135 (12)	0.00022 (13)
Br1	0.0145 (2)	0.0135 (2)	0.0149 (2)	0.00045 (19)	0.00676 (19)	0.00145 (19)
Br2	0.0203 (2)	0.0149 (2)	0.0113 (2)	-0.0055 (2)	0.00477 (19)	-0.00073 (19)
O1	0.0152 (17)	0.031 (2)	0.0085 (17)	0.0037 (16)	0.0025 (14)	0.0009 (15)
N1	0.0140 (19)	0.024 (2)	0.0074 (19)	0.0003 (18)	0.0071 (16)	-0.0001 (19)
C2	0.012 (2)	0.016 (2)	0.014 (2)	0.001 (2)	0.0053 (18)	-0.001 (2)
N3	0.0101 (18)	0.0153 (19)	0.011 (2)	0.0016 (17)	0.0014 (15)	0.0012 (17)
C4	0.011 (2)	0.013 (2)	0.014 (2)	-0.0006 (19)	0.0013 (18)	0.002 (2)
C4A	0.010 (2)	0.013 (2)	0.011 (2)	0.0027 (18)	0.0011 (18)	0.003 (2)
C5	0.013 (2)	0.028 (3)	0.012 (2)	-0.001 (2)	0.003 (2)	0.003 (2)
C6	0.010 (2)	0.028 (3)	0.021 (3)	-0.002 (2)	0.0029 (19)	0.005 (2)
C7	0.016 (2)	0.030 (3)	0.012 (2)	-0.002 (2)	-0.0035 (19)	0.004 (2)
C8	0.019 (3)	0.027 (3)	0.009 (2)	-0.002 (2)	0.0016 (19)	0.002 (2)
C8A	0.012 (2)	0.015 (2)	0.010 (2)	-0.0025 (19)	0.0021 (18)	0.004 (2)

## Geometric parameters (Å, °)

Cd1—N3	2.238 (4)	C4—C4A	1.460 (6)
Cd1—Br2 <sup>i</sup>	2.5886 (6)	C4A—C8A	1.394 (6)
Cd1—Br1	2.6494 (6)	C4A—C5	1.396 (6)
Cd1—Br1 <sup>ii</sup>	2.7302 (6)	C5—C6	1.371 (7)
Cd1—Br2	2.8574 (6)	C5—H5	0.9500
O1—C4	1.236 (5)	C6—C7	1.395 (7)
N1—C2	1.317 (6)	C6—H6	0.9500
N1—C8A	1.386 (6)	C7—C8	1.378 (7)
N1—H1	0.88 (2)	C7—H7	0.9500
C2—N3	1.321 (6)	C8—C8A	1.403 (7)
C2—H2	0.9500	C8—H8	0.9500
N3—C4	1.388 (6)		
N3—Cd1—Br2 <sup>i</sup>	126.23 (10)	O1—C4—N3	119.2 (4)
N3—Cd1—Br1	114.78 (10)	O1—C4—C4A	123.7 (4)
Br2 <sup>i</sup> —Cd1—Br1	117.82 (2)	N3—C4—C4A	117.1 (4)
N3—Cd1—Br1 <sup>ii</sup>	98.72 (11)	C8A—C4A—C5	118.7 (4)
Br2 <sup>i</sup> —Cd1—Br1 <sup>ii</sup>	93.35 (2)	C8A—C4A—C4	119.0 (4)
Br1—Cd1—Br1 <sup>ii</sup>	88.152 (19)	C5—C4A—C4	122.3 (4)
N3—Cd1—Br2	84.61 (11)	C6—C5—C4A	120.6 (5)
Br2 <sup>i</sup> —Cd1—Br2	88.56 (2)	C6—C5—H5	119.7
Br1—Cd1—Br2	85.999 (18)	C4A—C5—H5	119.7
Br1 <sup>ii</sup> —Cd1—Br2	174.08 (2)	C5—C6—C7	120.4 (5)
Cd1—Br1—Cd1 <sup>ii</sup>	91.846 (19)	C5—C6—H6	119.8
Cd1 <sup>i</sup> —Br2—Cd1	91.44 (2)	C7—C6—H6	119.8
C2—N1—C8A	120.8 (4)	C8—C7—C6	120.5 (5)
C2—N1—H1	116 (4)	C8—C7—H7	119.8
C8A—N1—H1	124 (4)	C6—C7—H7	119.8
N1—C2—N3	124.1 (4)	C7—C8—C8A	118.9 (5)
N1—C2—H2	118.0	C7—C8—H8	120.6
N3—C2—H2	118.0	C8A—C8—H8	120.6
C2—N3—C4	120.4 (4)	N1—C8A—C4A	118.5 (4)
C2—N3—Cd1	121.4 (3)	N1—C8A—C8	120.4 (4)
C4—N3—Cd1	118.2 (3)	C4A—C8A—C8	121.0 (4)
C8A—N1—C2—N3	−0.1 (8)	C4—C4A—C5—C6	179.2 (5)
N1—C2—N3—C4	1.8 (8)	C4A—C5—C6—C7	0.5 (8)
N1—C2—N3—Cd1	−177.8 (4)	C5—C6—C7—C8	0.7 (9)
C2—N3—C4—O1	177.7 (5)	C6—C7—C8—C8A	−1.3 (8)
Cd1—N3—C4—O1	−2.7 (6)	C2—N1—C8A—C4A	−1.3 (7)
C2—N3—C4—C4A	−2.0 (7)	C2—N1—C8A—C8	179.5 (5)
Cd1—N3—C4—C4A	177.7 (3)	C5—C4A—C8A—N1	−178.7 (5)
O1—C4—C4A—C8A	−179.1 (5)	C4—C4A—C8A—N1	1.1 (7)
N3—C4—C4A—C8A	0.6 (7)	C5—C4A—C8A—C8	0.4 (8)
O1—C4—C4A—C5	0.7 (8)	C4—C4A—C8A—C8	−179.8 (5)



N3—C4—C4A—C5	-179.7 (5)	C7—C8—C8A—N1	179.9 (5)
C8A—C4A—C5—C6	-1.0 (8)	C7—C8—C8A—C4A	0.8 (8)

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

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

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
N1—H1 $\cdots$ O1 <sup>iii</sup>	0.87 (3)	2.10 (3)	2.917 (6)	155 (6)

Symmetry code: (iii)  $x, -y+3/2, z+1/2$ .

### catena-Poly[[[quinazolin-4(3H)-one- $\kappa N^3$ ]mercury(II)]-di- $\mu$ -chlorido] (II)

#### Crystal data

[HgCl<sub>2</sub>(C<sub>8</sub>H<sub>6</sub>N<sub>2</sub>O)]

$M_r = 417.64$

Triclinic,  $P\bar{1}$

$a = 6.8191$  (8)  $\text{\AA}$

$b = 7.0735$  (8)  $\text{\AA}$

$c = 10.4659$  (12)  $\text{\AA}$

$\alpha = 85.718$  (2) $^\circ$

$\beta = 80.7887$  (19) $^\circ$

$\gamma = 89.152$  (2) $^\circ$

$V = 496.92$  (10)  $\text{\AA}^3$

$Z = 2$

$F(000) = 380$

$D_x = 2.791$  Mg m<sup>-3</sup>

Mo  $K\alpha$  radiation,  $\lambda = 0.71073$   $\text{\AA}$

Cell parameters from 3121 reflections

$\theta = 3.0\text{--}29.9^\circ$

$\mu = 15.99$  mm<sup>-1</sup>

$T = 100$  K

Prism, colourless

$0.04 \times 0.03 \times 0.03$  mm

#### Data collection

Bruker D8 goniometer with APEX CCD detector diffractometer

Radiation source: Incoatec microsource

Multilayer optics monochromator

$\omega$  scans

Absorption correction: multi-scan

(*TWINABS*; Bruker, 2014)

$T_{\min} = 0.302$ ,  $T_{\max} = 0.433$

14434 measured reflections

5037 independent reflections

4693 reflections with  $I > 2\sigma(I)$

$R_{\text{int}} = 0.050$

$\theta_{\max} = 30.3^\circ$ ,  $\theta_{\min} = 2.9^\circ$

$h = -9 \rightarrow 9$

$k = -9 \rightarrow 10$

$l = 0 \rightarrow 14$

#### Refinement

Refinement on  $F^2$

Least-squares matrix: full

$R[F^2 > 2\sigma(F^2)] = 0.036$

$wR(F^2) = 0.076$

$S = 1.08$

5037 reflections

132 parameters

1 restraint

Primary atom site location: structure-invariant direct methods

Hydrogen site location: mixed

H atoms treated by a mixture of independent and constrained refinement

$w = 1/[\sigma^2(F_o^2) + (0.020P)^2 + 2.5P]$

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

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

$\Delta\rho_{\max} = 1.54$  e  $\text{\AA}^{-3}$

$\Delta\rho_{\min} = -1.49$  e  $\text{\AA}^{-3}$

#### 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.

**Refinement.** Refined as a 2-component twin.

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

	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{iso}}^*/U_{\text{eq}}$
Hg1	0.96071 (4)	0.24641 (4)	0.07042 (3)	0.01548 (9)
Cl2	1.2589 (3)	0.0702 (3)	0.01773 (19)	0.0173 (4)
Cl1	0.8430 (3)	0.4314 (2)	-0.1209 (2)	0.0188 (4)
O1	0.9528 (7)	0.2176 (8)	0.3605 (5)	0.0212 (11)
N1	0.3826 (9)	0.2857 (9)	0.3209 (6)	0.0138 (12)
H1	0.255 (5)	0.293 (12)	0.313 (8)	0.03 (2)*
C2	0.5279 (11)	0.2835 (10)	0.2211 (7)	0.0161 (15)
H2	0.494018	0.298894	0.136314	0.019*
N3	0.7156 (9)	0.2615 (8)	0.2317 (6)	0.0129 (12)
C4	0.7779 (10)	0.2372 (10)	0.3539 (7)	0.0113 (13)
C4A	0.6195 (11)	0.2370 (10)	0.4663 (7)	0.0134 (14)
C5	0.6608 (11)	0.2103 (11)	0.5938 (7)	0.0161 (15)
H5	0.793612	0.189228	0.608450	0.019*
C6	0.5093 (12)	0.2146 (11)	0.6969 (8)	0.0203 (16)
H6	0.537995	0.197419	0.782925	0.024*
C7	0.3144 (12)	0.2440 (11)	0.6770 (8)	0.0202 (16)
H7	0.211185	0.246517	0.749509	0.024*
C8	0.2688 (11)	0.2694 (11)	0.5539 (7)	0.0193 (16)
H8	0.135319	0.290262	0.540841	0.023*
C8A	0.4223 (10)	0.2640 (10)	0.4475 (7)	0.0130 (14)

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

	$U^{11}$	$U^{22}$	$U^{33}$	$U^{12}$	$U^{13}$	$U^{23}$
Hg1	0.01190 (13)	0.02113 (15)	0.01333 (14)	0.00139 (11)	-0.00131 (9)	-0.00250 (11)
Cl2	0.0123 (9)	0.0204 (9)	0.0201 (10)	0.0022 (7)	-0.0039 (7)	-0.0058 (7)
Cl1	0.0219 (10)	0.0179 (8)	0.0194 (10)	-0.0013 (7)	-0.0120 (8)	-0.0003 (7)
O1	0.013 (3)	0.034 (3)	0.015 (3)	0.002 (2)	-0.003 (2)	0.002 (3)
N1	0.007 (3)	0.022 (3)	0.013 (3)	-0.001 (2)	-0.005 (2)	0.001 (2)
C2	0.014 (4)	0.019 (4)	0.015 (4)	0.001 (3)	-0.002 (3)	-0.003 (3)
N3	0.014 (3)	0.015 (3)	0.011 (3)	0.003 (2)	-0.003 (2)	-0.003 (2)
C4	0.013 (3)	0.013 (3)	0.008 (3)	-0.001 (3)	-0.003 (3)	-0.001 (3)
C4A	0.014 (3)	0.014 (3)	0.012 (3)	-0.002 (3)	-0.001 (3)	-0.001 (3)
C5	0.013 (3)	0.023 (4)	0.013 (4)	-0.004 (3)	-0.004 (3)	0.001 (3)
C6	0.023 (4)	0.025 (4)	0.012 (4)	-0.004 (3)	-0.002 (3)	-0.001 (3)
C7	0.018 (4)	0.025 (4)	0.016 (4)	0.000 (3)	0.002 (3)	-0.003 (3)
C8	0.014 (4)	0.028 (4)	0.016 (4)	0.000 (3)	-0.003 (3)	0.000 (3)
C8A	0.010 (3)	0.015 (3)	0.014 (3)	0.000 (3)	-0.002 (3)	0.000 (3)

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

Hg1—N3	2.185 (6)	C4—C4A	1.464 (10)
Hg1—Cl2	2.3791 (18)	C4A—C8A	1.398 (10)
Hg1—Cl1	2.5416 (19)	C4A—C5	1.406 (10)
Hg1—Cl1 <sup>i</sup>	2.7861 (18)	C5—C6	1.372 (10)

O1—C4	1.211 (8)	C5—H5	0.9500
N1—C2	1.320 (9)	C6—C7	1.389 (11)
N1—C8A	1.392 (9)	C6—H6	0.9500
N1—H1	0.89 (3)	C7—C8	1.371 (10)
C2—N3	1.309 (9)	C7—H7	0.9500
C2—H2	0.9500	C8—C8A	1.403 (10)
N3—C4	1.409 (9)	C8—H8	0.9500
N3—Hg1—Cl2	138.49 (16)	C8A—C4A—C5	118.8 (7)
N3—Hg1—Cl1	105.30 (16)	C8A—C4A—C4	119.7 (7)
Cl2—Hg1—Cl1	114.98 (7)	C5—C4A—C4	121.4 (7)
N3—Hg1—Cl1 <sup>i</sup>	96.08 (16)	C6—C5—C4A	119.9 (7)
Cl2—Hg1—Cl1 <sup>i</sup>	93.94 (6)	C6—C5—H5	120.0
Cl1—Hg1—Cl1 <sup>i</sup>	89.50 (6)	C4A—C5—H5	120.0
Hg1—Cl1—Hg1 <sup>i</sup>	90.50 (6)	C5—C6—C7	120.7 (7)
C2—N1—C8A	120.8 (6)	C5—C6—H6	119.7
C2—N1—H1	123 (6)	C7—C6—H6	119.7
C8A—N1—H1	116 (6)	C8—C7—C6	120.9 (7)
N3—C2—N1	124.0 (7)	C8—C7—H7	119.6
N3—C2—H2	118.0	C6—C7—H7	119.6
N1—C2—H2	118.0	C7—C8—C8A	119.0 (7)
C2—N3—C4	121.5 (6)	C7—C8—H8	120.5
C2—N3—Hg1	125.6 (5)	C8A—C8—H8	120.5
C4—N3—Hg1	112.9 (4)	N1—C8A—C4A	118.3 (6)
O1—C4—N3	119.9 (6)	N1—C8A—C8	121.0 (7)
O1—C4—C4A	124.5 (6)	C4A—C8A—C8	120.6 (7)
N3—C4—C4A	115.6 (6)		
C8A—N1—C2—N3	0.0 (11)	C4—C4A—C5—C6	178.7 (7)
N1—C2—N3—C4	0.2 (11)	C4A—C5—C6—C7	0.4 (12)
N1—C2—N3—Hg1	177.6 (5)	C5—C6—C7—C8	0.0 (12)
C2—N3—C4—O1	-179.8 (7)	C6—C7—C8—C8A	0.4 (12)
Hg1—N3—C4—O1	2.4 (8)	C2—N1—C8A—C4A	-1.0 (10)
C2—N3—C4—C4A	0.5 (10)	C2—N1—C8A—C8	178.9 (7)
Hg1—N3—C4—C4A	-177.3 (5)	C5—C4A—C8A—N1	-178.5 (6)
O1—C4—C4A—C8A	178.9 (7)	C4—C4A—C8A—N1	1.6 (10)
N3—C4—C4A—C8A	-1.4 (9)	C5—C4A—C8A—C8	1.6 (10)
O1—C4—C4A—C5	-1.0 (11)	C4—C4A—C8A—C8	-178.3 (7)
N3—C4—C4A—C5	178.7 (6)	C7—C8—C8A—N1	178.8 (7)
C8A—C4A—C5—C6	-1.2 (11)	C7—C8—C8A—C4A	-1.2 (11)

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

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

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
N1—H1 $\cdots$ O1 <sup>ii</sup>	0.89 (4)	2.11 (4)	2.935 (8)	155 (7)

Symmetry code: (ii)  $x-1, y, z$ .

Diiodidobis[quinazolin-4(3*H*)-one- $\kappa$ N<sup>3</sup>]cadmium(II) (III)

## Crystal data

[CdI<sub>2</sub>(C<sub>16</sub>H<sub>12</sub>N<sub>4</sub>O<sub>2</sub>)]  
*M<sub>r</sub>* = 658.50  
 Monoclinic, *C*2/*c*  
*a* = 22.242 (3) Å  
*b* = 6.8450 (9) Å  
*c* = 13.3702 (17) Å  
 $\beta$  = 118.8220 (16)°  
*V* = 1783.4 (4) Å<sup>3</sup>  
*Z* = 4

*F*(000) = 1224  
*D<sub>x</sub>* = 2.453 Mg m<sup>-3</sup>  
 Mo *K* $\alpha$  radiation,  $\lambda$  = 0.71073 Å  
 Cell parameters from 7294 reflections  
 $\theta$  = 3.1–30.6°  
 $\mu$  = 4.70 mm<sup>-1</sup>  
*T* = 100 K  
 Plate, colourless  
 0.12 × 0.10 × 0.04 mm

## Data collection

Bruker D8 goniometer with APEX CCD detector  
 diffractometer  
 Radiation source: Incoatec microsource  
 Multilayer optics monochromator  
 $\omega$  scans  
 Absorption correction: multi-scan  
 (*SADABS*; Bruker, 2014)  
*T<sub>min</sub>* = 0.544, *T<sub>max</sub>* = 0.746

13210 measured reflections  
 2687 independent reflections  
 2519 reflections with *I* > 2 $\sigma$ (*I*)  
*R<sub>int</sub>* = 0.024  
 $\theta_{\max}$  = 30.7°,  $\theta_{\min}$  = 3.1°  
*h* = -31→31  
*k* = -9→9  
*l* = -18→18

## Refinement

Refinement on *F*<sup>2</sup>  
 Least-squares matrix: full  
*R*[*F*<sup>2</sup> > 2 $\sigma$ (*F*<sup>2</sup>)] = 0.026  
*wR*(*F*<sup>2</sup>) = 0.063  
*S* = 1.12  
 2687 reflections  
 119 parameters  
 2 restraints

Primary atom site location: structure-invariant  
 direct methods  
 Hydrogen site location: mixed  
 H atoms treated by a mixture of independent  
 and constrained refinement  
 $w = 1/[\sigma^2(F_o^2) + (0.0331P)^2 + 1.7P]$   
 where  $P = (F_o^2 + 2F_c^2)/3$   
 $(\Delta/\sigma)_{\max} = 0.001$   
 $\Delta\rho_{\max} = 3.17 \text{ e } \text{Å}^{-3}$   
 $\Delta\rho_{\min} = -0.49 \text{ e } \text{Å}^{-3}$

## 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 (Å<sup>2</sup>)

	<i>x</i>	<i>y</i>	<i>z</i>	<i>U<sub>iso</sub></i> <sup>*</sup> / <i>U<sub>eq</sub></i>	Occ. (<1)
I1	-0.02284 (2)	0.27011 (3)	0.06205 (2)	0.01738 (7)	
Cd1	0.000000	0.06009 (4)	0.250000	0.01209 (8)	0.9682 (8)
Cd2	0.000000	-0.5534 (10)	0.250000	0.01209 (8)	0.0318 (8)
O1	-0.15047 (11)	0.1341 (3)	0.16751 (18)	0.0192 (4)	
N1	-0.14386 (12)	-0.4477 (3)	0.2024 (2)	0.0156 (5)	
H1A	-0.1400 (18)	-0.583 (5)	0.200 (3)	0.019*	
C2	-0.08880 (14)	-0.3397 (4)	0.2268 (2)	0.0163 (5)	
H2A	-0.046045	-0.405307	0.254856	0.020*	
N3	-0.08910 (11)	-0.1482 (3)	0.21497 (19)	0.0135 (4)	

C4	-0.15069 (13)	-0.0452 (4)	0.1747 (2)	0.0117 (5)
C4A	-0.21284 (13)	-0.1587 (4)	0.1429 (2)	0.0120 (5)
C5	-0.27777 (14)	-0.0702 (4)	0.0976 (2)	0.0161 (5)
H5A	-0.282158	0.066631	0.083981	0.019*
C6	-0.33542 (15)	-0.1813 (5)	0.0726 (2)	0.0188 (6)
H6A	-0.379070	-0.120238	0.042601	0.023*
C7	-0.32946 (15)	-0.3836 (5)	0.0915 (2)	0.0191 (6)
H7A	-0.369040	-0.458717	0.075289	0.023*
C8	-0.26645 (15)	-0.4744 (4)	0.1334 (2)	0.0172 (5)
H8A	-0.262633	-0.611862	0.144689	0.021*
C8A	-0.20823 (13)	-0.3616 (4)	0.1592 (2)	0.0131 (5)

*Atomic displacement parameters (Å<sup>2</sup>)*

	$U^{11}$	$U^{22}$	$U^{33}$	$U^{12}$	$U^{13}$	$U^{23}$
I1	0.01805 (11)	0.01668 (11)	0.01536 (10)	-0.00240 (6)	0.00642 (8)	0.00158 (6)
Cd1	0.01073 (13)	0.00997 (13)	0.01466 (14)	0.000	0.00539 (11)	0.000
Cd2	0.01073 (13)	0.00997 (13)	0.01466 (14)	0.000	0.00539 (11)	0.000
O1	0.0209 (10)	0.0124 (10)	0.0246 (11)	-0.0019 (8)	0.0112 (9)	-0.0008 (8)
N1	0.0173 (11)	0.0099 (11)	0.0214 (12)	0.0013 (8)	0.0108 (10)	0.0023 (9)
C2	0.0129 (12)	0.0168 (13)	0.0194 (13)	0.0036 (10)	0.0080 (11)	0.0029 (11)
N3	0.0115 (10)	0.0147 (11)	0.0142 (10)	-0.0005 (8)	0.0060 (9)	0.0006 (9)
C4	0.0151 (12)	0.0102 (12)	0.0105 (11)	-0.0007 (9)	0.0067 (10)	-0.0012 (9)
C4A	0.0134 (12)	0.0124 (12)	0.0105 (11)	0.0001 (9)	0.0059 (10)	-0.0011 (9)
C5	0.0166 (12)	0.0156 (13)	0.0154 (13)	0.0020 (10)	0.0072 (11)	0.0001 (10)
C6	0.0124 (12)	0.0289 (16)	0.0140 (13)	0.0016 (11)	0.0055 (10)	-0.0019 (11)
C7	0.0165 (13)	0.0259 (15)	0.0159 (13)	-0.0066 (11)	0.0087 (11)	-0.0028 (11)
C8	0.0212 (14)	0.0156 (13)	0.0167 (13)	-0.0050 (11)	0.0108 (11)	-0.0026 (10)
C8A	0.0146 (12)	0.0136 (13)	0.0111 (11)	-0.0009 (10)	0.0061 (10)	-0.0005 (9)

*Geometric parameters (Å, °)*

I1—Cd2 <sup>i</sup>	2.608 (3)	N3—C4	1.397 (3)
I1—Cd1	2.7219 (4)	C4—C4A	1.459 (4)
Cd1—N3 <sup>ii</sup>	2.299 (2)	C4A—C8A	1.402 (4)
Cd1—N3	2.299 (2)	C4A—C5	1.406 (4)
Cd2—C2 <sup>ii</sup>	2.355 (5)	C5—C6	1.386 (4)
Cd2—C2	2.355 (5)	C5—H5A	0.9500
O1—C4	1.232 (3)	C6—C7	1.403 (5)
N1—C2	1.329 (4)	C6—H6A	0.9500
N1—C8A	1.391 (4)	C7—C8	1.381 (4)
N1—H1A	0.93 (4)	C7—H7A	0.9500
C2—N3	1.320 (4)	C8—C8A	1.401 (4)
C2—H2A	0.9500	C8—H8A	0.9500
N3 <sup>ii</sup> —Cd1—N3	103.32 (12)	C2—N3—Cd1	129.61 (17)
N3 <sup>ii</sup> —Cd1—I1	105.94 (6)	C4—N3—Cd1	110.74 (17)
N3—Cd1—I1	112.37 (6)	O1—C4—N3	119.5 (2)

N3 <sup>ii</sup> —Cd1—I1 <sup>ii</sup>	112.37 (6)	O1—C4—C4A	123.2 (2)
N3—Cd1—I1 <sup>ii</sup>	105.94 (6)	N3—C4—C4A	117.3 (2)
I1—Cd1—I1 <sup>ii</sup>	116.237 (16)	C8A—C4A—C5	118.5 (2)
C2 <sup>ii</sup> —Cd2—C2	103.2 (3)	C8A—C4A—C4	119.6 (2)
C2 <sup>ii</sup> —Cd2—I1 <sup>iii</sup>	100.14 (7)	C5—C4A—C4	121.9 (2)
C2—Cd2—I1 <sup>iii</sup>	113.53 (8)	C6—C5—C4A	120.4 (3)
C2 <sup>ii</sup> —Cd2—I1 <sup>iv</sup>	113.53 (8)	C6—C5—H5A	119.8
C2—Cd2—I1 <sup>iv</sup>	100.14 (7)	C4A—C5—H5A	119.8
I1 <sup>iii</sup> —Cd2—I1 <sup>iv</sup>	124.8 (3)	C5—C6—C7	120.1 (3)
C2—Cd2—H2A <sup>ii</sup>	97.1 (4)	C5—C6—H6A	119.9
I1 <sup>iii</sup> —Cd2—H2A <sup>ii</sup>	95.37 (11)	C7—C6—H6A	119.9
I1 <sup>iv</sup> —Cd2—H2A <sup>ii</sup>	123.21 (12)	C8—C7—C6	120.5 (3)
C2—N1—C8A	120.6 (2)	C8—C7—H7A	119.8
C2—N1—H1A	118 (2)	C6—C7—H7A	119.8
C8A—N1—H1A	120 (2)	C7—C8—C8A	119.2 (3)
N3—C2—N1	124.9 (2)	C7—C8—H8A	120.4
N3—C2—Cd2	126.0 (2)	C8A—C8—H8A	120.4
N1—C2—Cd2	107.6 (2)	N1—C8A—C8	120.9 (3)
N3—C2—H2A	117.6	N1—C8A—C4A	117.9 (2)
N1—C2—H2A	117.6	C8—C8A—C4A	121.2 (2)
C2—N3—C4	119.6 (2)		
C8A—N1—C2—N3	1.1 (4)	C8A—C4A—C5—C6	1.7 (4)
C8A—N1—C2—Cd2	-165.5 (2)	C4—C4A—C5—C6	-177.4 (3)
N1—C2—N3—C4	0.4 (4)	C4A—C5—C6—C7	-0.6 (4)
Cd2—C2—N3—C4	164.55 (19)	C5—C6—C7—C8	-1.0 (4)
N1—C2—N3—Cd1	-179.0 (2)	C6—C7—C8—C8A	1.3 (4)
C2—N3—C4—O1	177.5 (2)	C2—N1—C8A—C8	-179.9 (3)
Cd1—N3—C4—O1	-3.0 (3)	C2—N1—C8A—C4A	-0.2 (4)
C2—N3—C4—C4A	-2.6 (4)	C7—C8—C8A—N1	179.5 (3)
Cd1—N3—C4—C4A	176.94 (17)	C7—C8—C8A—C4A	-0.1 (4)
O1—C4—C4A—C8A	-176.8 (2)	C5—C4A—C8A—N1	179.0 (2)
N3—C4—C4A—C8A	3.3 (3)	C4—C4A—C8A—N1	-1.9 (4)
O1—C4—C4A—C5	2.3 (4)	C5—C4A—C8A—C8	-1.4 (4)
N3—C4—C4A—C5	-177.6 (2)	C4—C4A—C8A—C8	177.7 (2)

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

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

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
N1—H1A $\cdots$ O1 <sup>iii</sup>	0.93 (3)	1.97 (3)	2.893 (3)	168 (4)

Symmetry code: (iii)  $x, y-1, z$ .