Acta Cryst E
CRYSTALLOGRAPHIC COMMUNICATIONS

ISSN 2056-9890

# Crystal structures of $Zn(cyclam)I_2$ (second monoclinic polymorph) and $Zn(cyclam)I(I_3)$

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Received 14 October 2021
Accepted 24 October 2021

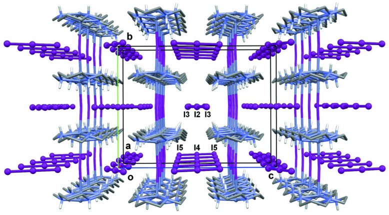
Edited by W. T. A. Harrison, University of Aberdeen, Scotland

**Keywords:** crystal structure; polymorph; cyclam; zinc; iodide; triiodide; hydrogen bonds.

**CCDC references**: 2115387; 2115388

**Supporting information**: this article has supporting information at journals.iucr.org/e

The asymmetric unit of the first title compound iodido(1,4,8,11-tetraazacyclotetradecane-$\kappa^4N^1,N^4,N^8,N^{11}$)zinc(II) iodide, $[ZnI(C_{10}H_{24}N_4)]I$, **I**, consists of the zinc–cyclam macrocyclic cation with one iodide anion coordinated to the metal ion [Zn—I = 2.6619 (5) Å] and the second present as a counter-ion. The asymmetric unit of the second title compound iodido(1,4,8,11-tetraazacyclotetradecane-$\kappa^4N^1,N^4,N^8,N^{11}$)zinc(II) triiodide, $[ZnI(C_{10}H_{24}N_4)]I_3$, **II**, consists of half of the centrosymmetric macrocyclic cation, in which the $Zn^{II}$ ion coordinated to an iodide anion [Zn—I = 2.766 (2) Å] is disordered over two positions [Zn$\cdots$Zn = 0.810 (3) Å], and of the two halves of the crystallographically non-equivalent, non-coordinated, centrosymmetric triiodide anions. In both compounds, the *N*,*N*,*N*,*N*-tetradentate macrocyclic ligand is present in the most energetically favored *trans*-III conformation. In the crystals of **I**, the $[Zn(C_{10}H_{24}N_4)I]^+$ cations and the non-coordinated iodide anions are linked by N—H$\cdots$I and bifurcated N—H$\cdots$(I,I) hydrogen bonds, resulting in the formation of two-dimensional networks lying parallel to the (001) and (101) planes. In contrast, the crystals of **II** are built up from infinite chains of the five-coordinate macrocyclic units arranged along the *b*-axis direction and perpendicular sheets formed of the triiodide counter-ions without significant hydrogen bonding between them.



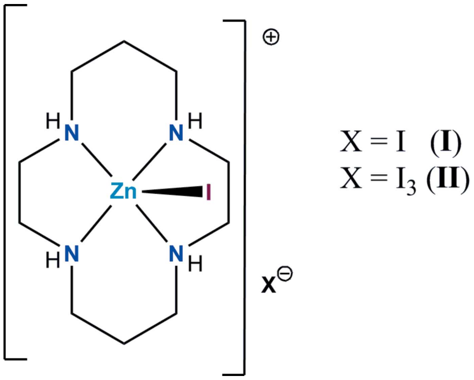
## 1. Chemical context

The 14-membered tetraazamacrocycle 1,4,8,11-tetraazacyclotetradecane ($C_{10}H_{24}N_4$, cyclam, *L*) is one of the most useful and widely studied ligands because of a number of unique properties, such as exceptionally high thermodynamic stability, kinetic inertness and unusual redox properties inherent to its complexes with transition-metal ions (Melson, 1979; Yatsimirskii & Lampeka, 1985). Typically, cyclam coordinates to the metal ion by its four N atoms in a planar manner, leaving two vacant *trans* binding sites in the coordination sphere for additional ligands, including halide anions as an important class. To date, a number of complexes of $[M(L)]^{2+}$ cations ($M$ = $Cu^{II}$, $Ni^{II}$, $Zn^{II}$) with halides $X^-$ ($X$ = Cl, Br, I) have been reported (Ito *et al.*, 1984; Adam *et al.*, 1991; Porai-Koshits *et al.*, 1994; Chen *et al.*, 1996; Makhaev *et al.*, 1996; Ha, 2017; Horii *et al.*, 2020).

Typically, the compounds under consideration are prepared by the direct reaction of $MX_2$ salts with *L*. We were interested in the development of alternative methods of synthesizing zinc(II) iodide compounds by anion exchange, starting from the initially formed acetate or nitrate species. It was found in the course of this investigation that precipitation of $Zn(L)I_2$ from the *in situ* formed acetate complex by potassium iodide

https://doi.org/10.1107/S2056989021011166

in methanol solution occurs slowly (over several days) and results in the formation of the colorless compound **I**, the structure of which is different from that described previously (Porai-Koshits *et al.*, 1994). When the metathesis reaction was carried out in aqueous solution, a small amount of the iodide/triiodide salt (compound **II**) was obtained in the form of intensely colored brown crystals. The lattice parameters for this compound were reported by Heinlein & Tebbe (1985) in an alternate setting of the unit cell (see *Database Survey*) but no atomic coordinates were established. Here, we report the crystal structures of these two compounds, namely, iodido-(1,4,8,11-tetraazacyclotetradecane-$\kappa^4N^1N^4N^8N^{11}$)zinc(II) iodide, [ZnI($L$)]I, **I** and iodido-(1,4,8,11-tetraazacyclotetradecane-$\kappa^4N^1N^4N^8N^{11}$)zinc(II) triiodide, [ZnI($L$)]$I_3$, **II**.



## 2. Structural commentary

The molecular structure of **I** is shown in Fig. 1. It represents the square-pyramidal macrocyclic [Zn($L$)I]$^+$ cation with one iodide anion coordinated in the axial position of the zinc(II) ion, while the second iodide anion acts as a counter-ion.

**Table 1**
Selected geometrical parameters (Å, °) of the complex cations.

| **I** | | **II** | |
|---|---|---|---|
| Zn1—N1 | 2.101 (3) | Zn1—N1 | 2.014 (10) |
| Zn1—N2 | 2.121 (3) | Zn1—N2 | 2.014 (10) |
| Zn1—N3 | 2.121 (3) | Zn1—N1i | 2.179 (10) |
| Zn1—N4 | 2.110 (3) | Zn1—N2i | 2.210 (10) |
| Zn1—I1 | 2.6619 (5) | Zn1—I1 | 2.766 (2) |
| N1—Zn1—N4 | 95.77 (11) | N1—Zn1—N2 | 98.9 (5) |
| N2—Zn1—N3 | 88.94 (11) | N1$^i$—Zn1—N2$^i$ | 88.4 (4) |
| N1—Zn1—N2 | 82.64 (11) | N1$^i$—Zn1—N2 | 82.9 (5) |
| N3—Zn1—N4 | 82.61 (11) | N1—Zn1—N2$^i$ | 82.2 (5) |

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

Thus, **I** belongs to a rather limited family of [Zn($L$)] compounds in which the Zn$^{II}$ ion is five-coordinated. Other distinct examples are complexes with thiolate (Notni *et al.*, 2006) and hexacyanoferrate(3–) (Colacio *et al.*, 2001) axial ligands. In the majority of compounds, the Zn$^{II}$ ion is six-coordinated. Analogously to these complexes, the macrocyclic ligand in **I** adopts the most energetically favorable *trans*-III ($R$,$R$,$S$,$S$) conformation (Bosnich *et al.*, 1965).

The coordination polyhedron of the [Zn($L$)I]$^+$ cation in **I** is characterized by a large deviation [0.4412 (14) Å] of the metal ion from the mean $N_4$ plane of donor atoms toward the coordinated iodide ion and this results in conformational peculiarities, distinguishing it from planar tetra- or hexa-coordinated species. In particular, this deviation results in non-equivalence of the six-membered chelate rings in *chair* conformations with *syn* and *anti* directivity of the NH-hydrogen atoms with respect to the displacement of the metal ion. In the first case, the ring becomes more flattened at the Zn side, and in the second more puckered. Simultaneously, the five-membered rings in **I** adopt *gauche–envelope* conforma-

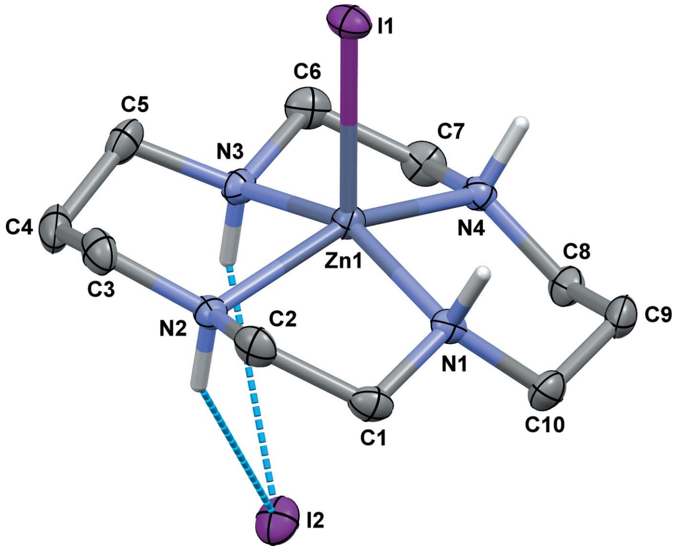


**Figure 1**
View of the molecular structure of **I** showing the atom-labeling scheme with displacement ellipsoids drawn at the 30% probability level. C-bound H atoms are omitted for clarity. Hydrogen-bonding interactions are shown as dashed lines.

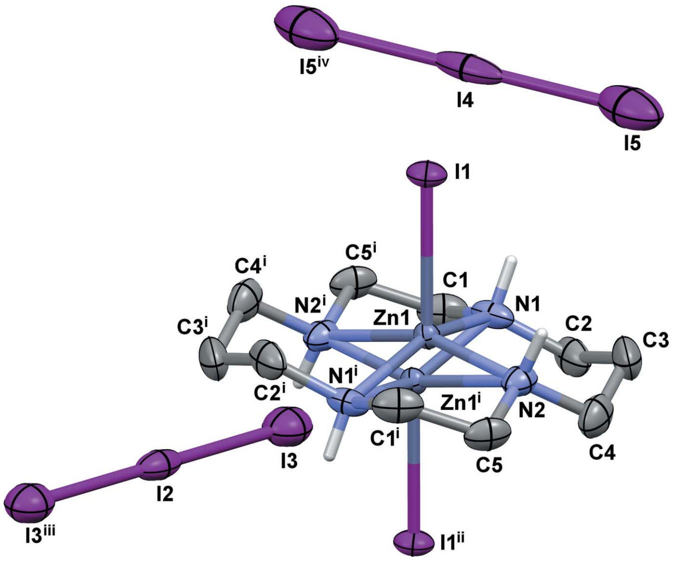


**Figure 2**
View of the molecular structure of **II** showing the atom-labeling scheme with displacement ellipsoids drawn at the 30% probability level. C-bound H atoms are omitted for clarity. Symmetry codes: (i) $-x + \frac{3}{2}, -y + \frac{3}{2}, -z + \frac{1}{2}$; (ii) $-x + \frac{3}{2}, y - \frac{1}{2}, -z + \frac{1}{2}$; (iii) $-x + 2, y, -z + 1$; (iv) $-x + 1, y, -z + 1$.]

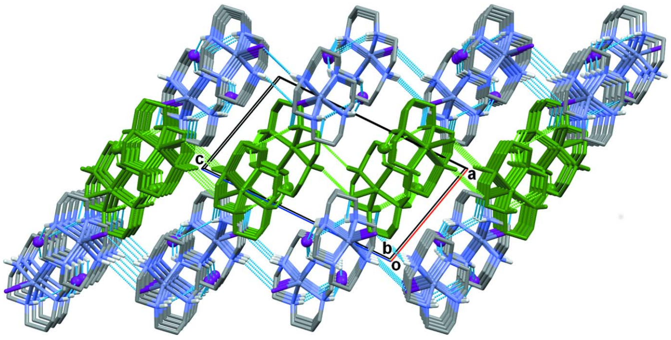


**Figure 3**
The packing in **I** viewed down the *b*-axis direction. Hydrogen-bonding interactions are shown as dashed lines.

tions (one of the carbon atoms lies almost in the N—Zn—N plane) in contrast to the symmetric *gauche* conformations in planar structures.

As expected, the bite angles in the five-membered chelate rings in **I** (*ca* 82.6°, Table 1) are reduced compared to the typical value of *ca* 85° in planar structures. At the same time, a considerable decrease in the bite angle occurs only in the '*anti*' six-membered chelate ring [88.94 (11)° *versus ca* 95° in planar structures].

The molecular structure of compound **II** is shown in Fig. 2. In this case the [Zn($L$)] unit is centrosymmetric but the zinc(II) ion is disordered over two positions with site occupancies of 50% constrained by symmetry with a Zn1$\cdots$Zn1$^{i}$ distance of 0.810 (3) Å [symmetry code: (i) $-x + \frac{3}{2}, -y + \frac{3}{2}, -z + \frac{1}{2}$]. Two crystallographically non-equivalent, non-coordinated centrosymmetric triiodide anions serve as counter-ions, with I2 and I4 occupying the inversion centers.

The structural characteristics of the [Zn($L$)I]$^+$ unit in **II** are in general agreement with those described above for **I**, with the deviation of the zinc(II) ion from the mean N$_4$ plane being 0.381 (2) Å. The '*syn*' and '*anti*' six-membered chelate rings are characterized by even higher divergences in their bite angles as compared to **I** (10.5° and 6.8°, respectively, Table 1). The five-membered rings in **II** are also present in *gauche–envelope* conformations. A notable distinction in **II** is the considerable difference of the Zn—N bond lengths in the '*syn*' and '*anti*' six-membered chelate rings [average values = 2.01 (1) and 2.20 (2) Å, respectively], while in **I** this difference is only 0.015 Å.

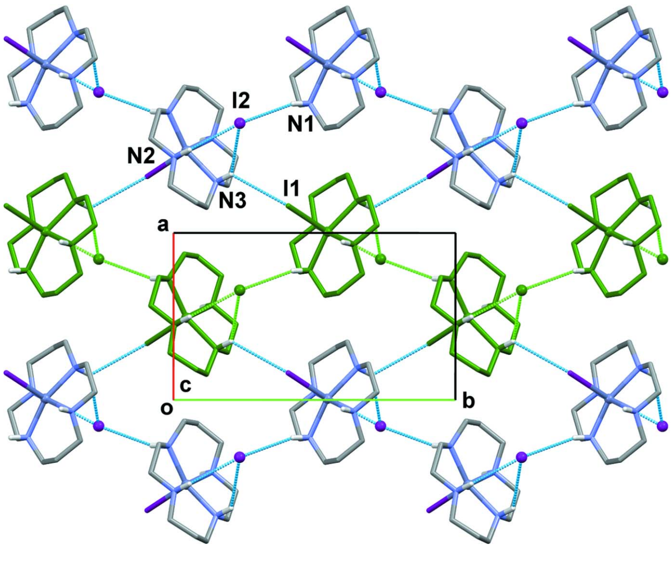


**Figure 4**
The structure of the hydrogen-bonded layer parallel to the *ab* plane in **I**. Hydrogen-bonding interactions are shown as dashed lines.

**Table 2**
Hydrogen-bond geometry (Å, °) for **I**.

| $D$—H$\cdots$$A$ | $D$—H | H$\cdots$$A$ | $D\cdots A$ | $D$—H$\cdots$$A$ |
|---|---|---|---|---|
| N1—H1$\cdots$I2$^{i}$ | 0.98 | 2.82 | 3.708 (3) | 151 |
| N2—H2$\cdots$I2 | 0.98 | 2.78 | 3.634 (3) | 146 |
| N3—H3$\cdots$I1$^{ii}$ | 0.98 | 3.20 | 3.819 (3) | 123 |
| N3—H3$\cdots$I2 | 0.98 | 3.13 | 3.897 (3) | 137 |
| N4—H4$\cdots$I1$^{iii}$ | 0.98 | 2.86 | 3.680 (3) | 142 |

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

## 3. Supramolecular features

The crystals of **I** have dual lamellar structure. The layers parallel to the *ab* plane are readily discernible (Fig. 3). They are composed of zigzag chains propagating along the *b*-axis direction, in which the links between the [Zn($L$)I]$^+$ units occur *via* N—H$\cdots$I hydrogen bonds between the secondary amino groups of the macrocyclic ligands (N1—H1, N2—H2 and N3—H3) as the donors and the non-coordinated I2 anions as the acceptors (Table 2). These chains are linked in the perpendicular (*a*-axis) direction through weak N3—H3$\cdots$ I1 bonds (Fig. 4). At the same time, paired hydrogen-bond contacts involving the coordinated iodide anions I1 and the N4–H4 groups of neighboring macrocycles lead to the formation of another two-dimensional network (Fig. 5). Since

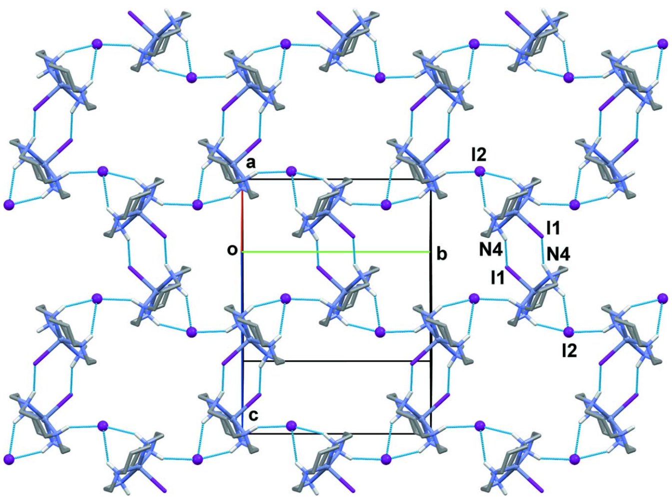


**Figure 5**
The structure of the hydrogen-bonded layer parallel to the (101) plane in **I**. Hydrogen-bonding interactions are shown as dashed lines.

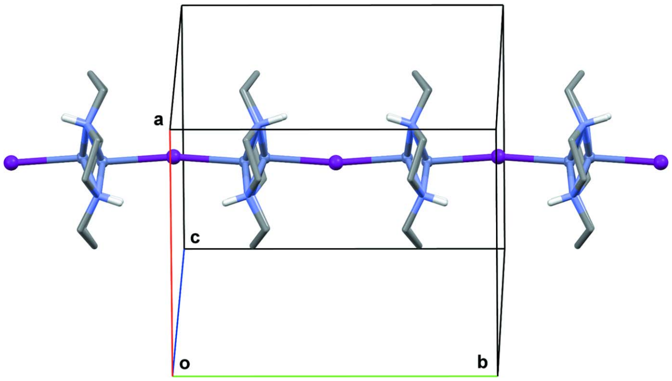


**Figure 6**
The arrangement of $[Zn(L)I]^+$ cations along the *b*-axis direction in **II**. C-bound H atoms are omitted for clarity.

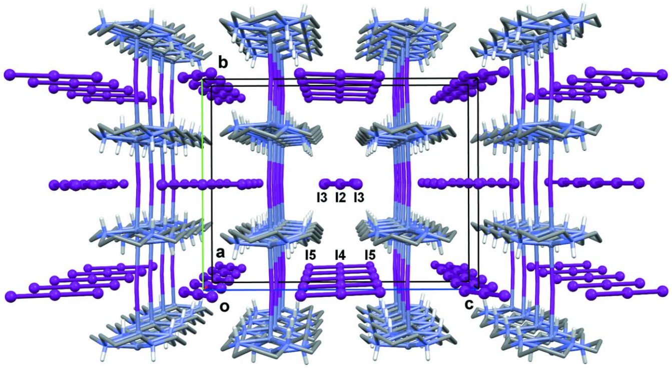


**Figure 7**
The packing in **II** viewed down the *a*-axis direction. C-bound H atoms are omitted for clarity.

the existence of such hydrogen-bonded layers parallel to the (101) plane is not so evident, one of these sheets in Figs. 3 and 4 is highlighted in dark green.

The disordered $[Zn(L)I]^+$ cations in the crystal of **II** are arranged in parallel chains running along the *b*-axis direction (Fig. 6). The peculiarity of this structure is that all of the iodine atoms, both coordinated (I1) and those of the triiodide anions [I3/I2/I3$^i$ and I5/I4/I5$^{ii}$; symmetry codes (i) $-x + 2, y, -z + 1$; (ii) $-x + 1, y, -z + 1$] lie strictly in crystallographic planes parallel to the *ac* plane, thus forming 'purely iodide' layers separated by half of the *b* unit-cell length (Fig. 7). As can be seen, all of the I3—I2—I3 triiodide anions are parallel, as well as the I5—I4—I5 ones, and they form an angle of 71.5 (3)° to each other. The shortest distance between the coordinated iodide and the triiodide anion is 4.803 (3) Å (I1···I5), while the shortest distance between triiodide anions is 4.949 (3) Å [I3···I3$^{iii}$; symmetry code: (iii) $-x + 1, y, -z + 1$]. Surprisingly, there are no hydrogen-bonding interactions in the crystal of **II** so its three-dimensional structure is based on weak ionic and van der Waals interactions.

## 4. Database survey

A search of the Cambridge Structural Database (CSD, version 5.40, last update February 2019; Groom *et al.*, 2016) indicated that a number of compounds of the composition $[M(L)]X_2$ have been characterized structurally. They include complexes of nickel(II) [refcodes TAZDNC01 (Ito *et al.*, 1984); TAZDNC02–08 (Horii *et al.*, 2020); RAPKAX (Ha, 2017); JIZTUH (Adam *et al.*, 1991); JIZTUH01–04 (Horii *et al.*, 2020)], copper(II) [TEGPOK (Chen *et al.*, 1996); TUCQEN (Makhaev *et al.*, 1996)] and zinc(II) [VUSDUI10, HEGNEM and HEGNOW (Porai-Koshits *et al.*, 1994)] cyclam cations with the full series (except for $CuLCl_2$) of halide anions ($X$ = Cl, Br, I).

In the overwhelming majority of cases, these complexes form monoclinic (space group $P2_1/c$ or $P2_1/n$) molecular crystals with the same structural motif: the complex moieties form infinite chains, in which they are joined by the pairs of N—H···*X* hydrogen bonds between the NH group of the macrocycle and the coordinated halide ion. On the other hand, in the case of the nickel(II), two other polymorphs of the iodide salt are known. These are also chain structures; however, one of the iodide anions is not coordinated [CAFHUM (Prasad & McAuley, 1983) and JIZTUH05–08 (Horii *et al.*, 2020)]. The peculiarity, characteristic only of zinc(II) complexes, is that quite similar to the situation observed in **II**, the metal ion is disordered over two positions. It should also be noted that a degree of pyramidalization of the $Zn(N_4)$ chromophore progressively increases on going from Cl to I (the deviation of the $Zn^{II}$ ion from the mean $N_4$ plane is 0.237, 0.322 and 0.385 Å, respectively) and the conformations of the chelate rings and their bite angles demonstrate systematic trends consistent with this variation. The structure of the complex $[Zn(L)I]I_3$ is also mentioned (DEHVOB; Heinlein & Tebbe, 1985), but without atomic coordinates. The reported unit-cell parameters (space group $C2/m$; $a$ = 19.189, $b$ = 12.615, $c$ = 10.072 Å; $\beta$ = 120.65°) represent an alternative setting of the $I2/m$ unit cell found here for **II**: the matrix 0 0 1 / 0 1 0 / −1 0 1 transforms the DEHVOB cell to that of **II**.

## 5. Synthesis and crystallization

All chemicals and solvents used in this work were purchased from Sigma–Aldrich and were used without further purification.

To prepare **I**, a solution of 48 mg (0.240 mmol) of cyclam in 2 ml of MeOH was added to a solution of 50 mg (0.228 mmol) of $Zn(CH_3CO_2)_2·2H_2O$ in 2 ml of MeOH and the mixture was heated at *ca* 333 K for 10 h. After cooling, a solution of 0.6 g of KI in 4 ml of MeOH was added and the mixture was left at room temperature. After one week, colorless prismatic crystals formed were filtered off, washed with MeOH and dried in air. Yield: 79 mg (67%). Analysis calculated for $C_{10}H_{24}N_4Zn_1I_2$: C 23.12; H 4.66; N 10.78%. Found: C 22.98; H 4.72; N 10.63%. Single crystals of **I** in the form of colorless prisms suitable for X-ray diffraction analysis were picked from the sample resulting from the synthesis.

Crystals of **II** were obtained in an experiment when the precipitation of the product was attempted in aqueous solu-

Table 3
Experimental details.

| | I | II |
|---|---|---|
| Crystal data | | |
| Chemical formula | $[ZnI(C_{10}H_{24}N_4)]I$ | $[ZnI(C_{10}H_{24}N_4)]I_3$ |
| $M_r$ | 519.50 | 773.30 |
| Crystal system, space group | Monoclinic, $P2_1/n$ | Monoclinic, $I2/m$ |
| Temperature (K) | 293 | 293 |
| $a, b, c$ (Å) | 8.3837 (3), 13.7570 (4), 14.6478 (5) | 10.0629 (12), 12.6263 (12), 16.5133 (16) |
| $\beta$ (°) | 103.852 (3) | 90.921 (10) |
| $V$ (Å$^3$) | 1640.25 (9) | 2097.9 (4) |
| $Z$ | 4 | 4 |
| Radiation type | Mo $K\alpha$ | Mo $K\alpha$ |
| $\mu$ (mm$^{-1}$) | 5.25 | 7.05 |
| Crystal size (mm) | 0.2 × 0.2 × 0.15 | 0.18 × 0.18 × 0.10 |
| Data collection | | |
| Diffractometer | Xcalibur, Eos | Xcalibur, Eos |
| Absorption correction | Multi-scan (*CrysAlis PRO*; Rigaku OD, 2019) | Multi-scan (*CrysAlis PRO*; Rigaku OD, 2019) |
| $T_{min}, T_{max}$ | 0.650, 1.000 | 0.563, 1.000 |
| No. of measured, independent and observed [$I > 2\sigma(I)$] reflections | 10691, 3785, 2982 | 1931, 1931, 1531 |
| $R_{int}$ | 0.031 | 0.065 |
| $(\sin \theta/\lambda)_{max}$ (Å$^{-1}$) | 0.684 | 0.595 |
| Refinement | | |
| $R[F^2 > 2\sigma(F^2)], wR(F^2), S$ | 0.033, 0.053, 1.02 | 0.065, 0.209, 1.02 |
| No. of reflections | 3785 | 1931 |
| No. of parameters | 155 | 100 |
| H-atom treatment | H-atom parameters constrained | H-atom parameters constrained |
| $\Delta\rho_{max}, \Delta\rho_{min}$ (e Å$^{-3}$) | 0.57, −0.83 | 1.86, −2.21 |

Computer programs: *CrysAlis PRO* (Rigaku OD, 2019), *SHELXT2018/2* (Sheldrick, 2015*a*), *SHELXL2018/3* (Sheldrick, 2015*b*), *Mercury* (Macrae *et al.*, 2020) and *publCIF* (Westrip, 2010).

tion. After addition of the solution of 0.5 g of KI in 0.5 ml of $H_2O$ to the solution of the nitrate salt of the macrocyclic cation [obtained *in situ* from 50 mg (0.25 mmol) of cyclam and 75 mg (0.25 mmol) of $Zn(NO_3)_2{\cdot}6H_2O$] in 2 ml of $H_2O$, a white precipitate formed (*ca* 92 mg), which was filtered off and the mother liquor was left exposed to the air. After several days, a small quantity of brown crystals of **II** had formed, which were picked for crystallographic investigation.

## 6. Refinement

Crystal data, data collection and structure refinement details are summarized in Table 3. All of the H atoms in **I** and **II** were placed in geometrically idealized positions and constrained to ride on their parent atoms, with C—H = 0.97 Å and N—H = 0.98 Å with $U_{iso}(H)$ values of $1.2U_{eq}$ of the parent atoms.

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# supporting information

*Acta Cryst.* (2021). E**77**, 1185-1189    [https://doi.org/10.1107/S2056989021011166]

# Crystal structures of Zn(cyclam)$I_2$ (second monoclinic polymorph) and Zn(cyclam)I($I_3$)

**Sergey P. Gavrish, Sergiu Shova and Yaroslaw D. Lampeka**

## Computing details

For both structures, data collection: *CrysAlis PRO* (Rigaku OD, 2019); cell refinement: *CrysAlis PRO* (Rigaku OD, 2019); data reduction: *CrysAlis PRO* (Rigaku OD, 2019); program(s) used to solve structure: *SHELXT2018/2* (Sheldrick, 2015a); program(s) used to refine structure: *SHELXL2018/3* (Sheldrick, 2015b); molecular graphics: *Mercury* (Macrae *et al.*, 2020); software used to prepare material for publication: *publCIF* (Westrip, 2010).

## Iodido(1,4,8,11-tetraazacyclotetradecane-$\kappa^4N^1,N^4,N^8,N^{11}$)zinc(II) iodide (I)

### *Crystal data*

[ZnI($C_{10}H_{24}N_4$)]I
$M_r$ = 519.50
Monoclinic, $P2_1/n$
$a$ = 8.3837 (3) Å
$b$ = 13.7570 (4) Å
$c$ = 14.6478 (5) Å
$\beta$ = 103.852 (3)°
$V$ = 1640.25 (9) Å$^3$
$Z$ = 4

$F(000)$ = 992
$D_x$ = 2.104 Mg m$^{-3}$
Mo $K\alpha$ radiation, $\lambda$ = 0.71073 Å
Cell parameters from 3942 reflections
$\theta$ = 2.1–27.3°
$\mu$ = 5.25 mm$^{-1}$
$T$ = 293 K
Prism, clear light colourless
0.2 × 0.2 × 0.15 mm

### *Data collection*

Xcalibur, Eos diffractometer
Radiation source: fine-focus sealed X-ray tube, Enhance (Mo) X-ray Source
Graphite monochromator
Detector resolution: 16.1593 pixels mm$^{-1}$
$\omega$ scans
Absorption correction: multi-scan (CrysAlisPro; Rigaku OD, 2019)

$T_{\min}$ = 0.650, $T_{\max}$ = 1.000
10691 measured reflections
3785 independent reflections
2982 reflections with $I > 2\sigma(I)$
$R_{\text{int}}$ = 0.031
$\theta_{\max}$ = 29.1°, $\theta_{\min}$ = 2.1°
$h = -10 \rightarrow 10$
$k = -17 \rightarrow 11$
$l = -18 \rightarrow 19$

### *Refinement*

Refinement on $F^2$
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.033
$wR(F^2)$ = 0.053
$S$ = 1.02
3785 reflections
155 parameters
0 restraints

Primary atom site location: structure-invariant direct methods
Hydrogen site location: inferred from neighbouring sites
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0145P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\max}$ = 0.001
$\Delta\rho_{\max}$ = 0.57 e Å$^{-3}$

$\Delta\rho_{\min} = -0.83$ e Å$^{-3}$

Extinction correction: *SHELXL2018/3* (Sheldrick 2015b), $Fc^{*}=kFc[1+0.001xFc^{2}\lambda^{3}/\sin(2\theta)]^{-1/4}$
Extinction coefficient: 0.00134 (7)

*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$^2$)*

| | *x* | *y* | *z* | $U_{\text{iso}}$*/$U_{\text{eq}}$ |
|---|---|---|---|---|
| I1 | 0.16199 (3) | 0.40650 (2) | 0.15018 (2) | 0.03711 (9) |
| Zn1 | 0.00746 (5) | 0.54540 (3) | 0.22192 (3) | 0.02665 (12) |
| N1 | −0.2241 (3) | 0.48210 (19) | 0.2122 (2) | 0.0272 (7) |
| H1 | −0.226473 | 0.423178 | 0.174250 | 0.033* |
| N2 | 0.0454 (3) | 0.5033 (2) | 0.36481 (19) | 0.0292 (7) |
| H2 | 0.003393 | 0.556139 | 0.397386 | 0.035* |
| N3 | 0.1933 (3) | 0.65217 (19) | 0.2632 (2) | 0.0282 (7) |
| H3 | 0.149659 | 0.704337 | 0.295758 | 0.034* |
| N4 | −0.0687 (3) | 0.63817 (19) | 0.1048 (2) | 0.0288 (7) |
| H4 | −0.054780 | 0.601213 | 0.050069 | 0.035* |
| C1 | −0.2327 (5) | 0.4485 (3) | 0.3069 (3) | 0.0371 (10) |
| H1A | −0.306512 | 0.393370 | 0.301155 | 0.044* |
| H1B | −0.275717 | 0.500177 | 0.339202 | 0.044* |
| C2 | −0.0644 (5) | 0.4196 (3) | 0.3632 (3) | 0.0384 (10) |
| H2A | −0.068776 | 0.402191 | 0.426776 | 0.046* |
| H2B | −0.024607 | 0.363962 | 0.334543 | 0.046* |
| C3 | 0.2174 (5) | 0.4875 (3) | 0.4148 (3) | 0.0410 (10) |
| H3A | 0.264794 | 0.437386 | 0.382861 | 0.049* |
| H3B | 0.221722 | 0.465027 | 0.478124 | 0.049* |
| C4 | 0.3177 (5) | 0.5806 (3) | 0.4193 (3) | 0.0463 (11) |
| H4A | 0.262635 | 0.631620 | 0.445670 | 0.056* |
| H4B | 0.423918 | 0.570130 | 0.462252 | 0.056* |
| C5 | 0.3457 (4) | 0.6162 (3) | 0.3260 (3) | 0.0415 (10) |
| H5A | 0.426638 | 0.667933 | 0.337714 | 0.050* |
| H5B | 0.389197 | 0.563309 | 0.295420 | 0.050* |
| C6 | 0.2140 (5) | 0.6907 (3) | 0.1731 (3) | 0.0389 (10) |
| H6A | 0.260339 | 0.641125 | 0.139970 | 0.047* |
| H6B | 0.288015 | 0.745919 | 0.183961 | 0.047* |
| C7 | 0.0475 (5) | 0.7213 (3) | 0.1152 (3) | 0.0389 (10) |
| H7A | 0.006315 | 0.775097 | 0.145756 | 0.047* |
| H7B | 0.056870 | 0.742876 | 0.053664 | 0.047* |
| C8 | −0.2429 (4) | 0.6695 (3) | 0.0832 (3) | 0.0365 (9) |
| H8A | −0.267305 | 0.706063 | 0.024857 | 0.044* |
| H8B | −0.259879 | 0.712074 | 0.132732 | 0.044* |
| C9 | −0.3590 (4) | 0.5841 (3) | 0.0740 (3) | 0.0393 (10) |

| | | | | |
|---|---|---|---|---|
| H9A | −0.323413 | 0.534611 | 0.036061 | 0.047* |
| H9B | −0.467479 | 0.605410 | 0.040333 | 0.047* |
| C10 | −0.3724 (4) | 0.5385 (3) | 0.1661 (3) | 0.0391 (10) |
| H10A | −0.389967 | 0.589381 | 0.208462 | 0.047* |
| H10B | −0.467193 | 0.495836 | 0.154407 | 0.047* |
| I2 | −0.15695 (4) | 0.73451 (2) | 0.37919 (2) | 0.04638 (10) |

*Atomic displacement parameters (Å$^2$)*

| | $U^{11}$ | $U^{22}$ | $U^{33}$ | $U^{12}$ | $U^{13}$ | $U^{23}$ |
|---|---|---|---|---|---|---|
| I1 | 0.04629 (18) | 0.03129 (16) | 0.03725 (17) | 0.00582 (11) | 0.01687 (13) | −0.00334 (11) |
| Zn1 | 0.0257 (2) | 0.0263 (2) | 0.0277 (3) | −0.00164 (17) | 0.00594 (19) | 0.00302 (18) |
| N1 | 0.0295 (18) | 0.0244 (16) | 0.0288 (18) | −0.0045 (13) | 0.0095 (14) | −0.0032 (13) |
| N2 | 0.0333 (18) | 0.0282 (17) | 0.0252 (18) | 0.0054 (13) | 0.0054 (14) | −0.0004 (13) |
| N3 | 0.0252 (17) | 0.0267 (17) | 0.0320 (19) | −0.0028 (13) | 0.0054 (14) | −0.0032 (13) |
| N4 | 0.0294 (17) | 0.0297 (17) | 0.0276 (18) | −0.0024 (13) | 0.0073 (14) | −0.0020 (13) |
| C1 | 0.039 (2) | 0.037 (2) | 0.039 (3) | −0.0065 (18) | 0.019 (2) | −0.0012 (19) |
| C2 | 0.048 (3) | 0.037 (2) | 0.032 (2) | −0.0008 (19) | 0.014 (2) | 0.0108 (18) |
| C3 | 0.050 (3) | 0.039 (3) | 0.030 (2) | 0.0088 (19) | 0.001 (2) | 0.0068 (18) |
| C4 | 0.039 (3) | 0.051 (3) | 0.039 (3) | 0.009 (2) | −0.010 (2) | −0.007 (2) |
| C5 | 0.025 (2) | 0.040 (2) | 0.054 (3) | −0.0029 (17) | −0.002 (2) | −0.005 (2) |
| C6 | 0.042 (3) | 0.029 (2) | 0.048 (3) | −0.0098 (18) | 0.016 (2) | −0.0040 (19) |
| C7 | 0.050 (3) | 0.026 (2) | 0.042 (3) | −0.0054 (18) | 0.014 (2) | 0.0059 (18) |
| C8 | 0.037 (2) | 0.036 (2) | 0.034 (2) | 0.0092 (17) | 0.0049 (18) | 0.0057 (18) |
| C9 | 0.027 (2) | 0.048 (3) | 0.037 (3) | 0.0015 (18) | −0.0034 (18) | −0.0012 (19) |
| C10 | 0.025 (2) | 0.047 (3) | 0.044 (3) | −0.0047 (18) | 0.0070 (19) | −0.003 (2) |
| I2 | 0.04667 (19) | 0.03273 (17) | 0.0554 (2) | 0.00644 (12) | 0.00368 (14) | 0.00011 (13) |

*Geometric parameters (Å, º)*

| | | | |
|---|---|---|---|
| I1—Zn1 | 2.6619 (5) | C3—H3A | 0.9700 |
| Zn1—N1 | 2.101 (3) | C3—H3B | 0.9700 |
| Zn1—N2 | 2.121 (3) | C3—C4 | 1.525 (5) |
| Zn1—N3 | 2.121 (3) | C4—H4A | 0.9700 |
| Zn1—N4 | 2.110 (3) | C4—H4B | 0.9700 |
| N1—H1 | 0.9800 | C4—C5 | 1.522 (6) |
| N1—C1 | 1.481 (4) | C5—H5A | 0.9700 |
| N1—C10 | 1.483 (4) | C5—H5B | 0.9700 |
| N2—H2 | 0.9800 | C6—H6A | 0.9700 |
| N2—C2 | 1.471 (4) | C6—H6B | 0.9700 |
| N2—C3 | 1.468 (4) | C6—C7 | 1.509 (5) |
| N3—H3 | 0.9800 | C7—H7A | 0.9700 |
| N3—C5 | 1.471 (4) | C7—H7B | 0.9700 |
| N3—C6 | 1.470 (4) | C8—H8A | 0.9700 |
| N4—H4 | 0.9800 | C8—H8B | 0.9700 |
| N4—C7 | 1.486 (4) | C8—C9 | 1.510 (5) |
| N4—C8 | 1.482 (4) | C9—H9A | 0.9700 |
| C1—H1A | 0.9700 | C9—H9B | 0.9700 |

| | | | |
|---|---|---|---|
| C1—H1B | 0.9700 | C9—C10 | 1.516 (5) |
| C1—C2 | 1.507 (5) | C10—H10A | 0.9700 |
| C2—H2A | 0.9700 | C10—H10B | 0.9700 |
| C2—H2B | 0.9700 | | |

| | | | |
|---|---|---|---|
| N1—Zn1—I1 | 101.81 (8) | N2—C3—H3B | 109.4 |
| N1—Zn1—N2 | 82.64 (11) | N2—C3—C4 | 111.3 (3) |
| N1—Zn1—N3 | 155.51 (11) | H3A—C3—H3B | 108.0 |
| N1—Zn1—N4 | 95.77 (11) | C4—C3—H3A | 109.4 |
| N2—Zn1—I1 | 102.79 (8) | C4—C3—H3B | 109.4 |
| N2—Zn1—N3 | 88.94 (11) | C3—C4—H4A | 108.4 |
| N3—Zn1—I1 | 102.47 (8) | C3—C4—H4B | 108.4 |
| N4—Zn1—I1 | 101.21 (8) | H4A—C4—H4B | 107.4 |
| N4—Zn1—N2 | 155.76 (11) | C5—C4—C3 | 115.7 (3) |
| N4—Zn1—N3 | 82.61 (11) | C5—C4—H4A | 108.4 |
| Zn1—N1—H1 | 105.9 | C5—C4—H4B | 108.4 |
| C1—N1—Zn1 | 108.5 (2) | N3—C5—C4 | 111.9 (3) |
| C1—N1—H1 | 105.9 | N3—C5—H5A | 109.2 |
| C1—N1—C10 | 111.5 (3) | N3—C5—H5B | 109.2 |
| C10—N1—Zn1 | 118.3 (2) | C4—C5—H5A | 109.2 |
| C10—N1—H1 | 105.9 | C4—C5—H5B | 109.2 |
| Zn1—N2—H2 | 107.0 | H5A—C5—H5B | 107.9 |
| C2—N2—Zn1 | 104.6 (2) | N3—C6—H6A | 110.1 |
| C2—N2—H2 | 107.0 | N3—C6—H6B | 110.1 |
| C3—N2—Zn1 | 115.5 (2) | N3—C6—C7 | 108.2 (3) |
| C3—N2—H2 | 107.0 | H6A—C6—H6B | 108.4 |
| C3—N2—C2 | 115.3 (3) | C7—C6—H6A | 110.1 |
| Zn1—N3—H3 | 108.1 | C7—C6—H6B | 110.1 |
| C5—N3—Zn1 | 114.5 (2) | N4—C7—C6 | 109.7 (3) |
| C5—N3—H3 | 108.1 | N4—C7—H7A | 109.7 |
| C6—N3—Zn1 | 103.3 (2) | N4—C7—H7B | 109.7 |
| C6—N3—H3 | 108.1 | C6—C7—H7A | 109.7 |
| C6—N3—C5 | 114.3 (3) | C6—C7—H7B | 109.7 |
| Zn1—N4—H4 | 106.2 | H7A—C7—H7B | 108.2 |
| C7—N4—Zn1 | 108.6 (2) | N4—C8—H8A | 109.2 |
| C7—N4—H4 | 106.2 | N4—C8—H8B | 109.2 |
| C8—N4—Zn1 | 116.1 (2) | N4—C8—C9 | 112.0 (3) |
| C8—N4—H4 | 106.2 | H8A—C8—H8B | 107.9 |
| C8—N4—C7 | 112.7 (3) | C9—C8—H8A | 109.2 |
| N1—C1—H1A | 109.6 | C9—C8—H8B | 109.2 |
| N1—C1—H1B | 109.6 | C8—C9—H9A | 108.5 |
| N1—C1—C2 | 110.3 (3) | C8—C9—H9B | 108.5 |
| H1A—C1—H1B | 108.1 | C8—C9—C10 | 115.2 (3) |
| C2—C1—H1A | 109.6 | H9A—C9—H9B | 107.5 |
| C2—C1—H1B | 109.6 | C10—C9—H9A | 108.5 |
| N2—C2—C1 | 107.6 (3) | C10—C9—H9B | 108.5 |
| N2—C2—H2A | 110.2 | N1—C10—C9 | 112.9 (3) |
| N2—C2—H2B | 110.2 | N1—C10—H10A | 109.0 |

| | | | |
|---|---|---|---|
| C1—C2—H2A | 110.2 | N1—C10—H10B | 109.0 |
| C1—C2—H2B | 110.2 | C9—C10—H10A | 109.0 |
| H2A—C2—H2B | 108.5 | C9—C10—H10B | 109.0 |
| N2—C3—H3A | 109.4 | H10A—C10—H10B | 107.8 |

| | | | |
|---|---|---|---|
| Zn1—N1—C1—C2 | −30.6 (3) | N4—C8—C9—C10 | 76.8 (4) |
| Zn1—N1—C10—C9 | 46.4 (4) | C1—N1—C10—C9 | 173.2 (3) |
| Zn1—N2—C2—C1 | −50.4 (3) | C2—N2—C3—C4 | −175.4 (3) |
| Zn1—N2—C3—C4 | 62.3 (4) | C3—N2—C2—C1 | −178.3 (3) |
| Zn1—N3—C5—C4 | −63.1 (4) | C3—C4—C5—N3 | 69.4 (4) |
| Zn1—N3—C6—C7 | 53.0 (3) | C5—N3—C6—C7 | 178.0 (3) |
| Zn1—N4—C7—C6 | 27.4 (3) | C6—N3—C5—C4 | 178.0 (3) |
| Zn1—N4—C8—C9 | −53.1 (4) | C7—N4—C8—C9 | −179.3 (3) |
| N1—C1—C2—N2 | 55.6 (4) | C8—N4—C7—C6 | 157.5 (3) |
| N2—C3—C4—C5 | −68.4 (4) | C8—C9—C10—N1 | −72.4 (4) |
| N3—C6—C7—N4 | −55.5 (4) | C10—N1—C1—C2 | −162.5 (3) |

*Hydrogen-bond geometry (Å, º)*

| *D*—H···*A* | *D*—H | H···*A* | *D*···*A* | *D*—H···*A* |
|---|---|---|---|---|
| N1—H1···I2$^{i}$ | 0.98 | 2.82 | 3.708 (3) | 151 |
| N2—H2···I2 | 0.98 | 2.78 | 3.634 (3) | 146 |
| N3—H3···I1$^{ii}$ | 0.98 | 3.20 | 3.819 (3) | 123 |
| N3—H3···I2 | 0.98 | 3.13 | 3.897 (3) | 137 |
| N4—H4···I1$^{iii}$ | 0.98 | 2.86 | 3.680 (3) | 142 |

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

## Iodido(1,4,8,11-tetraazacyclotetradecane-$\kappa^4N^1,N^4,N^8,N^{11}$)zinc(II) triiodide (II)

*Crystal data*

$[\mathrm{ZnI(C_{10}H_{24}N_4)]I_3}$
$M_r = 773.30$
Monoclinic, $I2/m$
$a = 10.0629\ (12)$ Å
$b = 12.6263\ (12)$ Å
$c = 16.5133\ (16)$ Å
$\beta = 90.921\ (10)°$
$V = 2097.9\ (4)$ Å$^3$
$Z = 4$
$F(000) = 1416$
$D_x = 2.448$ Mg m$^{-3}$
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 2913 reflections
$\theta = 2.0–26.7°$
$\mu = 7.05$ mm$^{-1}$
$T = 293$ K
Prism, clear light red
0.18 × 0.18 × 0.10 mm

*Data collection*

Xcalibur, Eos diffractometer
Radiation source: fine-focus sealed X-ray tube, Enhance (Mo) X-ray Source
Graphite monochromator
Detector resolution: 16.1593 pixels mm$^{-1}$
$\omega$ scans
Absorption correction: multi-scan (CrysAlisPro; Rigaku OD, 2019)
$T_{\min} = 0.563$, $T_{\max} = 1.000$
1931 measured reflections
1931 independent reflections
1531 reflections with $I > 2\sigma(I)$
$R_{\mathrm{int}} = 0.065$
$\theta_{\max} = 25.0°$, $\theta_{\min} = 2.0°$
$h = -11 \rightarrow 11$
$k = -14 \rightarrow 15$
$l = -19 \rightarrow 19$

*Refinement*

| | |
|---|---|
| Refinement on $F^2$ | Primary atom site location: dual |
| Least-squares matrix: full | Hydrogen site location: mixed |
| $R[F^2 > 2\sigma(F^2)] = 0.065$ | H-atom parameters constrained |
| $wR(F^2) = 0.209$ | $w = 1/[\sigma^2(F_o^2) + (0.1207P)^2 + 24.1639P]$ where $P = (F_o^2 + 2F_c^2)/3$ |
| $S = 1.02$ | $(\Delta/\sigma)_{max} < 0.001$ |
| 1931 reflections | $\Delta\rho_{max} = 1.86$ e Å$^{-3}$ |
| 100 parameters | $\Delta\rho_{min} = -2.20$ e Å$^{-3}$ |
| 0 restraints | |

*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 (Å$^2$)*

| | *x* | *y* | *z* | $U_{iso}$*/$U_{eq}$ | Occ. (<1) |
|---|---|---|---|---|---|
| I1 | 0.75984 (13) | 1.000000 | 0.25422 (8) | 0.0546 (4) | |
| Zn1 | 0.7426 (3) | 0.78147 (17) | 0.24844 (16) | 0.0313 (6) | 0.5 |
| N1 | 0.5759 (11) | 0.7637 (8) | 0.3129 (7) | 0.054 (3) | |
| H1 | 0.547169 | 0.831842 | 0.316695 | 0.065* | |
| N2 | 0.6627 (11) | 0.7691 (9) | 0.1362 (6) | 0.052 (2) | |
| H2 | 0.638693 | 0.836861 | 0.124367 | 0.062* | |
| C1 | 0.5986 (19) | 0.7286 (12) | 0.3953 (8) | 0.071 (4) | |
| H1A | 0.592616 | 0.652075 | 0.398272 | 0.085* | |
| H1B | 0.532274 | 0.758792 | 0.430502 | 0.085* | |
| C2 | 0.4593 (13) | 0.7157 (13) | 0.2742 (11) | 0.071 (4) | |
| H2A | 0.382036 | 0.730667 | 0.306679 | 0.085* | |
| H2B | 0.471137 | 0.639494 | 0.272985 | 0.085* | |
| C3 | 0.4330 (15) | 0.7558 (16) | 0.1869 (12) | 0.088 (6) | |
| H3A | 0.344086 | 0.734576 | 0.170108 | 0.106* | |
| H3B | 0.435683 | 0.832586 | 0.187081 | 0.106* | |
| C4 | 0.5303 (16) | 0.7154 (15) | 0.1257 (9) | 0.076 (5) | |
| H4A | 0.541286 | 0.639577 | 0.132177 | 0.091* | |
| H4B | 0.495758 | 0.728671 | 0.071478 | 0.091* | |
| C5 | 0.7596 (16) | 0.7338 (12) | 0.0779 (7) | 0.063 (4) | |
| H5A | 0.737529 | 0.762622 | 0.024916 | 0.076* | |
| H5B | 0.757810 | 0.657158 | 0.074099 | 0.076* | |
| I2 | 1.000000 | 0.500000 | 0.500000 | 0.0780 (7) | |
| I3 | 0.7276 (2) | 0.500000 | 0.55899 (13) | 0.0992 (7) | |
| I4 | 0.500000 | 1.000000 | 0.500000 | 0.1262 (15) | |
| I5 | 0.3190 (3) | 1.000000 | 0.3615 (2) | 0.1519 (13) | |

*Atomic displacement parameters (Å$^2$)*

| | $U^{11}$ | $U^{22}$ | $U^{33}$ | $U^{12}$ | $U^{13}$ | $U^{23}$ |
|---|---|---|---|---|---|---|
| I1 | 0.0706 (8) | 0.0283 (6) | 0.0650 (8) | 0.000 | 0.0056 (6) | 0.000 |

| | | | | | | |
|---|---|---|---|---|---|---|
| Zn1 | 0.0299 (11) | 0.0357 (16) | 0.0285 (10) | 0.0018 (15) | 0.0024 (8) | 0.0004 (15) |
| N1 | 0.061 (6) | 0.032 (5) | 0.070 (7) | 0.005 (5) | 0.028 (5) | 0.001 (5) |
| N2 | 0.067 (6) | 0.041 (6) | 0.048 (5) | −0.005 (5) | −0.008 (5) | 0.002 (4) |
| C1 | 0.109 (12) | 0.056 (8) | 0.049 (7) | −0.003 (9) | 0.028 (8) | 0.002 (6) |
| C2 | 0.036 (6) | 0.062 (9) | 0.115 (13) | −0.002 (6) | 0.017 (7) | 0.003 (9) |
| C3 | 0.049 (8) | 0.084 (13) | 0.131 (16) | 0.014 (8) | −0.031 (10) | −0.002 (11) |
| C4 | 0.084 (10) | 0.078 (11) | 0.064 (9) | −0.008 (9) | −0.034 (8) | 0.009 (8) |
| C5 | 0.104 (11) | 0.056 (9) | 0.030 (6) | −0.004 (8) | 0.010 (7) | −0.004 (5) |
| I2 | 0.1383 (19) | 0.0391 (10) | 0.0555 (10) | 0.000 | −0.0321 (11) | 0.000 |
| I3 | 0.1264 (15) | 0.0671 (11) | 0.1039 (13) | 0.000 | −0.0103 (11) | 0.000 |
| I4 | 0.124 (2) | 0.0324 (10) | 0.227 (4) | 0.000 | 0.123 (2) | 0.000 |
| I5 | 0.1283 (19) | 0.0718 (13) | 0.258 (4) | 0.000 | 0.062 (2) | 0.000 |

*Geometric parameters (Å, º)*

| | | | |
|---|---|---|---|
| I1—Zn1$^{i}$ | 2.766 (2) | N2—C4 | 1.502 (19) |
| I1—Zn1 | 2.766 (2) | N2—C5 | 1.451 (17) |
| Zn1—Zn1$^{ii}$ | 0.810 (4) | C1—C5$^{ii}$ | 1.56 (2) |
| Zn1—N1$^{ii}$ | 2.179 (10) | C2—C3 | 1.55 (2) |
| Zn1—N1 | 2.014 (10) | C3—C4 | 1.51 (3) |
| Zn1—N2$^{ii}$ | 2.210 (10) | I2—I3$^{iii}$ | 2.924 (2) |
| Zn1—N2 | 2.014 (10) | I2—I3 | 2.924 (2) |
| N1—C1 | 1.444 (19) | I4—I5$^{iv}$ | 2.901 (4) |
| N1—C2 | 1.458 (19) | I4—I5 | 2.901 (4) |
| | | | |
| Zn1$^{i}$—I1—Zn1 | 171.86 (12) | C1—N1—Zn1$^{ii}$ | 103.7 (9) |
| Zn1$^{ii}$—Zn1—I1 | 164.9 (4) | C1—N1—C2 | 113.5 (12) |
| Zn1$^{ii}$—Zn1—N1$^{ii}$ | 67.6 (4) | C2—N1—Zn1$^{ii}$ | 111.2 (9) |
| Zn1$^{ii}$—Zn1—N1 | 90.6 (5) | C2—N1—Zn1 | 119.1 (9) |
| Zn1$^{ii}$—Zn1—N2 | 93.0 (5) | Zn1—N2—Zn1$^{ii}$ | 21.47 (16) |
| Zn1$^{ii}$—Zn1—N2$^{ii}$ | 65.5 (4) | C4—N2—Zn1 | 118.8 (9) |
| N1$^{ii}$—Zn1—I1 | 103.0 (3) | C4—N2—Zn1$^{ii}$ | 109.8 (8) |
| N1—Zn1—I1 | 98.3 (3) | C5—N2—Zn1$^{ii}$ | 101.5 (8) |
| N1—Zn1—N1$^{ii}$ | 158.18 (16) | C5—N2—Zn1 | 111.8 (8) |
| N1—Zn1—N2$^{ii}$ | 82.2 (5) | C5—N2—C4 | 112.9 (12) |
| N1$^{ii}$—Zn1—N2$^{ii}$ | 88.4 (4) | N1—C1—C5$^{ii}$ | 107.7 (11) |
| N2$^{ii}$—Zn1—I1 | 103.4 (3) | N1—C2—C3 | 113.4 (13) |
| N2—Zn1—I1 | 97.6 (3) | C4—C3—C2 | 114.3 (13) |
| N2—Zn1—N1 | 98.9 (5) | N2—C4—C3 | 110.8 (13) |
| N2—Zn1—N1$^{ii}$ | 82.9 (5) | N2—C5—C1$^{ii}$ | 109.8 (10) |
| N2—Zn1—N2$^{ii}$ | 158.53 (16) | I3$^{iii}$—I2—I3 | 180.0 |
| Zn1—N1—Zn1$^{ii}$ | 21.82 (16) | I5$^{iv}$—I4—I5 | 180.0 |
| C1—N1—Zn1 | 114.3 (10) | | |

Symmetry codes: (i) *x*, −*y*+2, *z*; (ii) −*x*+3/2, −*y*+3/2, −*z*+1/2; (iii) −*x*+2, −*y*+1, −*z*+1; (iv) −*x*+1, −*y*+2, −*z*+1.