Crystal structures of three zinc(II) halide coordination complexes with quinoline N-oxide

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The reaction of one equivalent of zinc(II) halide with two equivalents of quinoline N-oxide (QNO) in methanol yields compounds as ZnX$_2$(QNO)$_2$, where X = Cl (I), Br (II) and I (III), namely, dichloridobis(quinoline N-oxide-κO)zinc(II), [ZnCl$_2$(C$_9$H$_7$NO)$_2$], dibromidobis(quinoline N-oxide-κO)-zinc(II), [ZnBr$_2$(C$_9$H$_7$NO)$_2$], and diiodidobis(quinoline N-oxide-κO)zinc(II) [ZnI$_2$(C$_9$H$_7$NO)$_2$]. In all three complexes, Zn cations are coordinated by two QNO ligands bound through the oxygen atoms and two halide atoms, with X—Zn—X bond angles ca 20° wider than the O—Zn—O, giving rise to a distorted tetrahedral geometry. Crystals of (II) and (III) are isostructural and both show pairwise π-stacking of QNO ligands and weak C—H···Cl hydrogen bonds, while (I) packs differently, with a shorter C—H···Cl bond and without π-stacking.

1. Chemical context

N-oxide complexes have a rich history in organic transformations, including utility with transition metals in oxotransformations [see, for example, Eppenson (2003) and Moustafa et al. (2014)]. These transition-metal N-oxide complexes highlight the strong Lewis acid/Lewis base properties of the zinc(II) ion and N-oxides, respectively. Aromatic N-oxides are strong Lewis base ligands and form transition-metal complexes that are prevalent in the literature and highlight the strong transition metal interactions with the lone pair on the N-oxide oxygen atom. Examples of such complexes include a 4-methylpyridine N-oxide (MePyNO) derivative CuCl$_2$·2MePyNO (CMPYUC; Watson & Johnson, 1971) and pyridine N-oxide (6PyNO) derivatives Ni(BF$_4$)$_2$·6PyNO (PYNONE; van Ingen Schenau et al., 1974) or Au(CF$_3$)$_3$·PyNO (NEPVOW; Pérez-Bitrián et al., 2017). Previous reports of zinc(II) complexes with aromatic N-oxides include dibromo(4-methoxypridined N-oxide-κO)zinc(II) (GAWHW; Shi et al. 2005a), diaquabis(picolinato N-oxide-κ^2O, O')zinc(II) (XISBOR; Li et al., 2008) and dichloridobis(pyridine N-oxide)zinc(II) (QQQBX01; McConnell et al., 1986), all of which are mononuclear complexes.

Herein we report the crystal structures of three complexes of quinoline N-oxide (QNO) with zinc(II) chloride, bromide and iodide. All three were obtained by 1:2 stoichiometric reaction of the zinc(II) halide with QNO in methanol and found to be mononuclear ZnX$_2$(QNO)$_2$ complexes with a distorted tetrahedral environment around the zinc ion.
2. Structural commentary

Compound (I) crystallizes in the monoclinic space group P2₁ (Fig. 1), whereas compounds (II) (Fig. 2) and (III) (Fig. 3) both crystallize in the monoclinic space group P2₁/c. Each structure contains one symmetrically independent molecule, the coordination sphere around each Zn atom being a distorted tetrahedron. Selected bond lengths and angles in these complexes are shown in Table 1. Compounds (II) and (III) are isostructural in both the molecular conformation and crystal packing, while (I) differs in both aspects, as illustrated by an overlay of molecules (I) and (II) (Fig. 4a) on one hand, and molecules (II) and (III) on the other (Fig. 4b). Most notably, (I) differs in the orientation of the QNO rings relative to each other, the C2—N1—N2—C11 torsion angles being −16.9 (5)° in (I) versus −113.9 (3)° in (II) and −111.6 (3)° in (III).

3. Supramolecular features

Figs. 5, 6 and 7 show the packing of compounds (I), (II) and (III), respectively. In the crystal structures, the packing is stabilized by van der Waals interactions and, in (II) and (III), by similar systems of pairwise π–π stacking interactions. Quinoline moieties Cg1–Cg3 and Cg2–Cg4 (see Figs. 6 and 7) are stacked each against its own inversion-related equivalent, with the separations between their (parallel) mean planes equaling 3.483 (5) and 3.402 (5) Å, respectively, for (II), 3.466 (5) and 3.436 (5) Å for (III). The structure of (I) has no π-stacking. Besides, all three structures are characterized by C—H⋯X hydrogen bonds (X = halogen), see below.

Table 1

| Compound (I) | Compound (II) | Compound (III) |
|--------------|---------------|----------------|
| Zn1—Cl1     | 2.215 (2)     | Zn1—I1        | 2.5534 (8)     |
| Zn1—Cl2     | 2.211 (2)     | Zn1—I2        | 2.5475 (9)     |
| Zn1—O1      | 1.991 (5)     | Zn1—I1        | 2.5534 (8)     |
| Zn1—O2      | 1.995 (5)     | Zn1—I2        | 2.5475 (9)     |
| C1—Zn1—O2   | 117.80 (9)    | Br1—Zn1—I1−O2 | 123.45 (4)     |
| O1—Zn1—O2   | 99.4 (2)      | O1—I1−Zn1—I2  | 122.34 (3)     |

Figure 1

A view of compound (I), showing the atom labeling. Displacement ellipsoids are drawn at the 50% probability level.

Figure 2

A view of compound (II), showing the atom labeling. Displacement ellipsoids are drawn at the 50% probability level.

Figure 3

A view of compound (III), showing the atom labeling. Displacement ellipsoids are drawn at the 50% probability level.
4. Hirshfeld surface analysis

The intermolecular interactions were further investigated by quantitative analysis of the Hirshfeld surface, and visualized with Crystal Explorer 21 (Spackman et al., 2021) and the two-dimensional fingerprint plots (McKinnon et al., 2007). Figs. 8, 9 and 10 show Hirshfeld surfaces of molecules (I) to (III) mapped with the function $d_{\text{norm}}$, the sum of the distances from a surface point to the nearest interior ($d_i$) and exterior ($d_e$) atoms, normalized by the van der Waals (vdW) radii of the corresponding atoms ($r_{\text{vdW}}$). Contacts shorter than the sums of vdW radii are shown in red, those longer in blue, and those approximately equal to vdW as white spots.

For (I), the most intense red spots correspond to the intermolecular contacts O1⋯C9(1 − x, y − $\frac{1}{2}$, 1 − z) [3.048 (9) Å] and the hydrogen bond C18⋯H18/C1/C1/C1 Cl2(x, y + 1, z). The latter has the distances H⋯Cl = 2.53 Å (for the C—H distance normalized to 1.083 Å) and C⋯Cl = 3.416 (9) Å within the previously observed range but shorter than the average values of 2.64 and 3.66 Å, respectively (Steiner, 1998). The other chloride ligand, Cl2, forms four H⋯Cl contacts of 2.83–2.98 Å, more typical for van der Waals interactions (Rowland & Taylor, 1996). For (II) and (III), the red spots correspond to C—H⋯X interactions, viz. C18⋯H18⋯X1, C5—H5⋯X1, C16—H16⋯X2, and C9—H9⋯X2, which can be also regarded as weak hydrogen bonds (Steiner, 1998). The H⋯X distances in (II) ($X = \text{Br}$) are 2.85, 2.88, 2.88 and 2.89 Å, respectively, while in (III) ($X = I$) they are 3.03, 3.12, 3.03 and 2.96 Å, respectively.
Analysis of the two-dimensional fingerprint plots (Table 2) indicates that H···H contacts are the most common in all three structures. X···H contacts make the second highest contribution, which increases in the succession (I) < (II) < (III), together with the size of the halogen atoms and hence their share of the molecular surface (16.9, 18.5 and 20.6%, respectively). Interestingly, π-stacking in the structures of (II) and (III) gives only a modest increase of C···C contacts compared to (I), probably because it is counterbalanced by an overall decrease of carbon atoms’ share of the surface (21.4 > 19.5 > 18.3%). No halogen···halogen contacts are observed in any of the three structures.

5. Database survey

A search in the Cambridge Structural Database (CSD, version 5.42, update of February 2021; Groom et al., 2016) for aromatic N-oxides and halogen ligands bound to zinc returned 21 unique entries, the majority (15) of which contain pyridine N-oxide and its derivatives. Of these, the most closely related are pyridine N-oxide complexes, dichlorobis(pyridine N-oxide)zinc(II) (QQQBXG; McConnell et al., 1986), dibromorobis(pyridine N-oxide)zinc(II) (QQQBXJ, QQQBXM), for which only unit-cell parameters were determined (Kidd et al., 1967), and finally, diiodobis(4-methylypyridine N-oxide)zinc(II) (SANRUV; Shi et al., 2005b). There is one known structure of a quinoline N-oxide derivative, dichlorobis(2-methylquinoline N-oxide)zinc(II) (AFUSEZ; Ivashevskaja et al., 2002).

6. Synthesis and crystallization

The water content of QNO and ZnBr\textsubscript{2} have been determined by Thermal Gravimetric Analysis. The formulation for each was found to be QNO\textsubscript{0.28}H\textsubscript{2}O (MW = 150.21 g mol\textsuperscript{-1}) and ZnBr\textsubscript{2}\textsubscript{0.86}H\textsubscript{2}O (MW = 240.69 g mol\textsuperscript{-1}).

The title compounds were all synthesized in a similar manner. Compound (I) was synthesized by dissolving 0.0986 g of QNO\textsubscript{0.28}H\textsubscript{2}O (0.656 mmol, purchased from Aldrich) in 33 mL of methanol to which 0.0440 g of ZnCl\textsubscript{2} (0.176 mmol, purchased from Strem Chemicals) were added at 295 K. The solution was covered with parafilm then allowed to sit; X-ray quality crystals were grown by slow evaporation at 295 K. Yield, 0.0822 g (60.2%). Selected IR bands (ATR–IR, cm\textsuperscript{-1}): 3107 (w), 3083 (w), 3057 (w), 1579 (m), 1513 (m), 1447 (m), 1402 (s), 1269 (s), 1227 (m), 1203 (s), 1179 (m), 1144 (m), 1089 (s), 1050 (m), 883 (s), 800 (s), 768 (s), 723 (m), 584 (m), 559 (m), 542 (m).

Table 2

| Compound                   | (I)   | (II)   | (III)  |
|----------------------------|-------|--------|--------|
| H···H                      | 32.0  | 36.7   | 36.5   |
| H···X···X···H               | 24.4  | 28.4   | 30.0   |
| C···H···C                  | 22.7  | 18.5   | 18.0   |
| C···C                      | 5.4   | 7.1    | 6.4    |
| O···O···H···O               | 6.0   | 4.0    | 3.7    |

Figure 8
Hirshfeld surface for (I) mapped over d\textsuperscript{norm}.

Figure 9
Hirshfeld surface for (II) mapped over d\textsuperscript{norm}.

Figure 10
Hirshfeld surface for (III) mapped over d\textsuperscript{norm}.

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*Padgett et al.* · [ZnCl\textsubscript{2}(C\textsubscript{9}H\textsubscript{7}NO\textsubscript{2})\textsubscript{2}], [ZnBr\textsubscript{2}(C\textsubscript{9}H\textsubscript{7}NO\textsubscript{2})\textsubscript{2}] and [ZnI\textsubscript{2}(C\textsubscript{9}H\textsubscript{7}NO\textsubscript{2})\textsubscript{2}]

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Table 3
Experimental details.

|                  | (I)                  | (II)                  | (III)                  |
|------------------|----------------------|-----------------------|------------------------|
| Crystal data     | [ZnCl₂(C₉H₇NO)₂]     | [ZnBr₂(C₉H₇NO)₂]     | [ZnI₂(C₉H₇NO)₂]        |
| Mₒ                | 426.58               | 515.50                | 609.48                 |
| Crystal system, space group | Monoclinic, P₂₁ | Monoclinic, P₂₁/c | Monoclinic, P₂₁/c |
| Temperature (K)  | 298                  | 298                   | 297                    |
| β (°)             | 94.890 (5)           | 878.94 (8)            |                        |
| V (Å³)           | 3169                 | 3415                  | 3668                   |
| Z                 | 4                    | 4                     | 4                      |
| Radiation type   | Mo Kα                | Mo Kα                 | Mo Kα                  |
| μ (mm⁻¹)         | 1.72                 | 5.62                  | 4.32                   |
| Crystal size (mm) | 0.1 x 0.1 x 0.03     | 0.15 x 0.08 x 0.03    | 0.3 x 0.3 x 0.3        |

Data collection

|                    | Rigaku XtaLAB mini | XtaLAB Mini (ROW)    | Rigaku XtaLAB mini    |
|--------------------|--------------------|----------------------|-----------------------|
| Diffractometer     | Multi-scan         | Multi-scan           | Multi-scan            |
| Absorption correction | (CrysAlis PRO: Rigaku OD, 2019) | (CrysAlis PRO: Rigaku OD, 2019) | (CrysAlis PRO: Rigaku OD, 2019) |
| T_min, T_max       | 0.968, 1.000       | 0.833, 1.000         | 0.896, 1.000          |
| No. of measured reflections | 5308, 3169, 2456 | 7207, 3415, 2095     | 11510, 3668, 2748     |
| R int (sinθ/λ) max ( Å⁻¹) | 0.036             | 0.043                | 0.032                  |

Refinement

|                   | R[F² > 2σ(F²)], wR(F²), S | 0.044, 0.077, 1.03 | 0.042, 0.090, 1.02 |
|-------------------|---------------------------|--------------------|--------------------|
| No. of reflections|                           | 3169               | 3415               |
| No. of parameters |                           | 226                | 227                |
| No. of restraints |                           | 1                  | 0                  |
| H-atom treatment  |                           | H-atom parameters constrained | H-atom parameters constrained |
| Δρ_max, Δρ_min (e Å⁻³) | 0.42, -0.35 | 0.55, -0.35         | 0.80, -0.81        |
| Absolute structure |                           | Flack x determined using 810 quotients [(I')-(I)][(I)+(I')] | (Parsons et al., 2013). |
| Absolute structure parameter | -0.006 (15) | - | - |

Compound (II) was synthesized by dissolving 0.0983 g of QNO-OH₂ (0.654 mmol), in 40 mL of methanol to which 0.0778 g of ZnBr₂·0.86H₂O (0.323 mmol, purchased from Alfa Aesar) were added at 295 K. The solution was covered with paraffin then allowed to sit; X-ray quality crystals were grown by slow evaporation at 295 K. Yield, 0.0866 g (46.7%). Selected IR bands (ATR–IR, cm⁻¹): 3106 (w), 3075 (w), 3061 (w), 3016 (w), 1580 (m), 1510 (s), 1455 (m), 1270 (s), 1227 (m), 1214 (s), 1204 (s), 1173 (m), 1138 (m), 1086 (s), 1048 (m), 877 (m), 800 (s), 767 (s), 720 (s), 581 (m), 563 (m), 500 (m).

Compound (III) was synthesized by dissolving 0.0517 g of QNO-OH₂ (0.352 mmol) in approximately 36 mL of methanol to which 0.0524 g of ZnI₂ (0.164 mmol, purchased from Aldrich) were added at 295 K. The solution was covered with paraffin then allowed to sit; X-ray quality crystals were grown by slow evaporation at 295 K. Yield, 0.0910 g (52.3%). Selected IR Bands (ATR–IR, cm⁻¹): 3100 (w), 3090 (w), 2076 (w), 3059 (w), 3027 (w), 1580 (s), 1507 (s), 1382 (s), 1267 (m), 1225 (m), 1207 (s), 1169 (m), 1141 (m), 1044 (m), 880 (s), 807 (s), 769 (s), 720 (m), 580 (m), 562 (m), 499 (m).

Infrared spectroscopy confirms the presence of the QNO ligand in all three complexes. Characteristic IR bands include weak νC–H aromatic stretches observed from 3020–3107 cm⁻¹ and νN–O stretches of the bound N-oxide in the range 1350–1150 cm⁻¹; notably, a medium band observed in the ligand at 1311 cm⁻¹, appears at between 1225–1227 cm⁻¹ in the three metal complexes. Finally, a broad absorbance in the free ligand from 3100–3500 cm⁻¹ (assigned to the water νO–H stretch) is absent in all of the metal complexes (Mautner et al., 2016).

7. Refinement

Crystal data, data collection and structure refinement details are summarized in Table 3. All carbon-bound H atoms were positioned geometrically and refined as riding: C—H = 0.95–0.98 Å with Uiso(H) = 1.2Ueq(C).

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*research communications*
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Computing details
For all 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: SHELXT2014/5 (Sheldrick, 2015a); program(s) used to refine structure: SHELXL2018/1 (Sheldrick, 2015b); molecular graphics: OLEX2 (Dolomanov et al., 2009); software used to prepare material for publication: OLEX2 (Dolomanov et al., 2009).

Dichloridobis(quinoline N-oxide-κO)zinc(II) (I)

Crystal data

\[ \text{[ZnCl}_2\text{(C}_9\text{H}_7\text{NO)}_2] } \]

\( M_r = 426.58 \)

Monoclinic, \( P2_1 \)

\( a = 8.5167 (4) \text{ Å} \)

\( b = 7.8697 (4) \text{ Å} \)

\( c = 13.1617 (7) \text{ Å} \)

\( \beta = 94.890 (5)° \)

\( V = 878.94 (8) \text{ Å}^3 \)

\( Z = 2 \)

Data collection

Rigaku XtaLAB Mini diffractometer

Radiation source: fine-focus sealed X-ray tube, Rigaku (Mo) X-ray Source

Graphite monochromator

\( \omega \) scans

Absorption correction: multi-scan

(CrysAlisPro; Rigaku OD, 2019)

\( T_{\text{min}} = 0.968, T_{\text{max}} = 1.000 \)

Refinement

Refinement on \( F^2 \)

Least-squares matrix: full

\( R[F^2 > 2\sigma(F^2)] = 0.044 \)

\( wR(F^2) = 0.077 \)

\( S = 1.03 \)

3169 reflections

226 parameters

1 restraint

Primary atom site location: dual

Hydrogen site location: inferred from \( \text{neighbouring sites} \)

\( \text{H-atom parameters constrained} \)

\( w = 1/[\sigma^2(F_c) + (0.0183P)^2] \)

where \( P = (F_c^2 + 2F_c\tilde{e})/3 \)

\( \Delta \sigma_{\text{max}} < 0.001 \)

\( \Delta \rho_{\text{max}} = 0.42 \text{ e Å}^{-3} \)

\( \Delta \rho_{\text{min}} = -0.35 \text{ e Å}^{-3} \)
Absolute structure: Flack $x$ determined using 810 quotients $[(I^-)/(I^+)]/[((I^-)+(I^+))$ (Parsons et al., 2013).
Absolute structure parameter: $-0.006 (15)$

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 ($\AA^2$)

|    | $x$    | $y$    | $z$    | $U_{eq}$ |
|----|--------|--------|--------|----------|
| Zn1 | 0.60832 (9) | 0.40878 (9) | 0.69325 (6) | 0.0481 (2) |
| Cl1 | 0.8131 (2)  | 0.5260 (3)  | 0.78185 (18) | 0.0717 (6)  |
| Cl2 | 0.5724 (2)  | 0.1322 (2)  | 0.71012 (17) | 0.0668 (6)  |
| O1  | 0.6087 (5)  | 0.4435 (7)  | 0.5459 (4)   | 0.0660 (17) |
| O2  | 0.4152 (6)  | 0.5393 (6)  | 0.7184 (4)   | 0.0543 (13) |
| N1  | 0.6919 (7)  | 0.5702 (8)  | 0.5068 (4)   | 0.0472 (15) |
| N2  | 0.3927 (6)  | 0.6163 (7)  | 0.8067 (4)   | 0.0461 (14) |
| C1  | 0.7938 (8)  | 0.5254 (9)  | 0.4342 (5)   | 0.0418 (17) |
| C2  | 0.8061 (9)  | 0.3562 (9)  | 0.4045 (6)   | 0.052 (2)   |
| H2  | 0.745778    | 0.272501    | 0.432433     | 0.063*      |
| C3  | 0.9086 (10) | 0.3150 (11) | 0.3332 (6)   | 0.065 (2)   |
| H3  | 0.916168    | 0.203042    | 0.311636     | 0.077*      |
| C4  | 1.0011 (10) | 0.4398 (14) | 0.2932 (6)   | 0.071 (3)   |
| H4  | 1.072298    | 0.409606    | 0.246598     | 0.085*      |
| C5  | 0.9891 (9)  | 0.6041 (11) | 0.3210 (6)   | 0.061 (2)   |
| H5  | 1.051065    | 0.685676    | 0.292553     | 0.074*      |
| C6  | 0.8835 (8)  | 0.6538 (9)  | 0.3931 (5)   | 0.0469 (18) |
| C7  | 0.8623 (9)  | 0.8234 (8)  | 0.4243 (6)   | 0.056 (2)   |
| H7  | 0.920737    | 0.910057    | 0.397750     | 0.067*      |
| C8  | 0.7577 (10) | 0.8601 (9)  | 0.4927 (6)   | 0.063 (2)   |
| H8  | 0.742052    | 0.972081    | 0.511924     | 0.075*      |
| C9  | 0.6733 (9)  | 0.7293 (10) | 0.5342 (6)   | 0.056 (2)   |
| H9  | 0.602718    | 0.754824    | 0.582143     | 0.068*      |
| C10 | 0.3113 (8)  | 0.5307 (9)  | 0.8777 (6)   | 0.0441 (18) |
| C11 | 0.2654 (9)  | 0.3621 (9)  | 0.8595 (6)   | 0.059 (2)   |
| H11 | 0.289239    | 0.306487    | 0.800371     | 0.071*      |
| C12 | 0.1846 (10) | 0.2810 (12) | 0.9306 (7)   | 0.073 (2)   |
| H12 | 0.154890    | 0.168062    | 0.920740     | 0.087*      |
| C13 | 0.1458 (11) | 0.3686 (13) | 1.0195 (7)   | 0.081 (3)   |
| H13 | 0.089040    | 0.312853    | 1.066778     | 0.097*      |
| C14 | 0.1899 (10) | 0.5309 (12) | 1.0360 (7)   | 0.069 (3)   |
| H14 | 0.163818    | 0.585631    | 1.094912     | 0.082*      |
| C15 | 0.2745 (8)  | 0.6187 (10) | 0.9661 (5)   | 0.0508 (19) |
| C16 | 0.3245 (9)  | 0.7899 (11) | 0.9803 (6)   | 0.065 (2)   |
| H16 | 0.300458    | 0.850485    | 1.037580     | 0.078*      |
### Atomic displacement parameters (Å²)

|     | U¹¹ | U¹² | U¹³ | U²² | U²³ | U³³ |
|-----|-----|-----|-----|-----|-----|-----|
| Zn1 | 0.0494 (4) | 0.0451 (5) | 0.0519 (5) | −0.0013 (5) | 0.0168 (4) | 0.0023 (5) |
| Cl1 | 0.0656 (13) | 0.0678 (14) | 0.0812 (16) | −0.0073 (11) | 0.0034 (12) | −0.0053 (12) |
| Cl2 | 0.0710 (14) | 0.0421 (11) | 0.0875 (16) | −0.0013 (10) | 0.0087 (12) | 0.0054 (10) |
| O1  | 0.066 (3) | 0.083 (5) | 0.052 (3) | −0.033 (3) | 0.025 (3) | 0.006 (3) |
| O2  | 0.059 (3) | 0.063 (3) | 0.043 (3) | 0.011 (3) | 0.017 (3) | −0.010 (3) |
| N1  | 0.045 (3) | 0.057 (4) | 0.040 (4) | −0.004 (3) | 0.006 (3) | 0.002 (3) |
| N2  | 0.042 (3) | 0.050 (4) | 0.046 (4) | 0.007 (3) | 0.004 (3) | −0.003 (3) |
| C1  | 0.041 (4) | 0.047 (4) | 0.037 (4) | −0.003 (4) | 0.000 (3) | 0.010 (4) |
| C2  | 0.052 (5) | 0.056 (5) | 0.048 (5) | −0.008 (4) | 0.005 (4) | 0.003 (3) |
| C3  | 0.071 (6) | 0.064 (6) | 0.060 (5) | 0.005 (5) | 0.012 (5) | −0.003 (4) |
| C4  | 0.065 (5) | 0.097 (8) | 0.053 (5) | 0.012 (6) | 0.016 (4) | 0.008 (6) |
| C5  | 0.047 (5) | 0.078 (6) | 0.061 (6) | −0.006 (5) | 0.018 (4) | 0.027 (5) |
| C6  | 0.044 (4) | 0.052 (5) | 0.045 (4) | −0.008 (4) | 0.004 (4) | 0.010 (4) |
| C7  | 0.058 (5) | 0.043 (5) | 0.062 (5) | −0.012 (4) | −0.013 (4) | 0.019 (4) |
| C8  | 0.076 (6) | 0.042 (5) | 0.068 (5) | 0.006 (4) | −0.009 (5) | 0.001 (4) |
| C9  | 0.059 (5) | 0.065 (6) | 0.046 (4) | 0.013 (4) | 0.010 (4) | −0.004 (4) |
| C10 | 0.039 (4) | 0.043 (4) | 0.050 (5) | 0.009 (4) | 0.004 (4) | 0.008 (4) |
| C11 | 0.055 (5) | 0.061 (6) | 0.062 (5) | −0.003 (4) | 0.011 (4) | −0.004 (4) |
| C12 | 0.076 (6) | 0.056 (5) | 0.086 (7) | −0.007 (5) | 0.015 (6) | 0.002 (5) |
| C13 | 0.073 (6) | 0.097 (10) | 0.075 (6) | −0.004 (6) | 0.021 (5) | 0.022 (6) |
| C14 | 0.062 (6) | 0.085 (7) | 0.060 (6) | 0.004 (5) | 0.012 (5) | 0.001 (5) |
| C15 | 0.047 (4) | 0.059 (5) | 0.046 (5) | 0.008 (4) | 0.004 (4) | 0.002 (4) |
| C16 | 0.066 (6) | 0.065 (6) | 0.063 (5) | 0.010 (5) | 0.001 (5) | −0.021 (5) |
| C17 | 0.070 (6) | 0.053 (6) | 0.078 (6) | −0.004 (4) | 0.001 (5) | −0.011 (4) |
| C18 | 0.072 (6) | 0.039 (4) | 0.073 (6) | −0.005 (4) | 0.012 (5) | −0.004 (5) |

### Geometric parameters (Å, °)

|     |     |     |     |     |     |
|-----|-----|-----|-----|-----|-----|
| Zn1—Cl1  | 2.215 (2) | C7—H7  | 0.9300 |
| Zn1—Cl2  | 2.211 (2) | C7—C8  | 1.350 (11) |
| Zn1—O1   | 1.959 (5) | C8—H8  | 0.9300 |
| Zn1—O2   | 1.991 (4) | C8—C9  | 1.393 (10) |
| O1—N1    | 1.351 (7) | C9—H9  | 0.9300 |
| O2—N2    | 1.339 (6) | C10—C11 | 1.398 (10) |
| N1—C1    | 1.389 (8) | C10—C15 | 1.412 (10) |
| N1—C9    | 1.316 (9) | C11—H11 | 0.9300 |
| N2—C10   | 1.385 (8) | C11—C12 | 1.366 (10) |
| N2—C18   | 1.324 (9) | C12—H12 | 0.9300 |
| C1—C2    | 1.395 (9) | C12—C13 | 1.421 (12) |
| C1—C6    | 1.403 (9) | C13—H13 | 0.9300 |

*sup-3*
C2—H2 0.9300  C13—C14 1.344 (12)
C2—C3 1.373 (10)  C14—H14 0.9300
C3—H3 0.9300  C14—C15 1.399 (10)
C3—C4 1.390 (11)  C15—C16 1.420 (11)
C4—H4 0.9300  C16—H16 0.9300
C4—C5 1.350 (12)  C16—C17 1.362 (11)
C5—H5 0.9300  C17—H17 0.9300
C5—C6 1.417 (10)  C17—C18 1.373 (10)
C6—C7 1.412 (10)  C18—H18 0.9300

Cl2—Zn1—Cl1 117.80 (9)  C8—C7—H7 119.9
O1—Zn1—Cl1 113.30 (15)  C7—C8—H8 120.2
O1—Zn1—Cl2 104.40 (18)  C7—C8—C9 119.7 (7)
O1—Zn1—O2 99.4 (2)  C9—C8—H8 120.2
O2—Zn1—Cl1 108.81 (16)  N1—C9—C8 121.1 (7)
O2—Zn1—Cl2 111.57 (16)  N1—C9—H9 119.4
N1—O1—Zn1 121.7 (4)  C9—N1—O1 121.2 (6)
N2—O2—Zn1 124.0 (4)  N2—C10—C11 119.6 (7)
O1—N1—C1 117.0 (6)  N2—C10—C15 118.5 (7)
C9—N1—O1 121.2 (6)  C11—C10—C15 121.9 (7)
C9—N1—C1 121.8 (6)  C11—C10—C15 120.8
N2—O2—Zn1 110.81 (16)  C12—C11—C15 123.2 (8)
C8—N2—O2 110.81 (16)  C12—C11—C15 117.6 (8)
C18—N2—C10 118.2 (7)  C13—C14—C15 121.0 (9)
C18—N2—C10 118.2 (7)  C14—C15—C16 117.6 (7)
N1—C1—C2 120.5  C14—C15—C16 117.6 (7)
N1—C1—C6 118.3 (7)  C15—C16—H16 120.6
C2—C1—C6 121.6 (7)  C16—C17—C18 122.1 (8)
C1—C2—H2 120.5  C17—C18—H18 118.9
C3—C2—H2 118.9 (7)  N2—C18—H18 118.9
C3—C2—C1 120.5  C18—C19—C20 120.6
C3—C2—H2 118.9 (7)  C19—C20—C21 119.8
C3—C2—H2 118.9 (7)  C19—C20—C21 119.8
C2—C3—C4 120.4 (8)  C20—C21—C22 118.8 (7)
C2—C3—H3 119.8  C21—C22—C23 118.8 (7)
C2—C3—C4 120.4 (8)  C22—C23—C24 118.8 (7)
C2—C3—H3 119.8  C23—C24—C25 118.8 (7)
C3—C4—C5 119.5  C24—C25—C26 118.8 (7)
C3—C4—H4 119.5  C25—C26—C27 118.8 (7)
C5—C4—C3 121.0 (8)  C26—C27—C28 118.8 (7)
C5—C4—H4 119.5  C27—C28—C29 118.8 (7)
C5—C5—H5 119.6  C28—C29—C30 118.8 (7)
C5—C5—H5 119.6  C29—C30—C31 118.8 (7)
C6—C5—H5 120.9 (8)  C30—C31—C32 118.8 (7)
C6—C5—H5 120.9 (8)  C31—C32—C33 118.8 (7)
C1—C6—C5 117.2 (7)  C32—C33—C34 118.8 (7)
C1—C6—C7 118.8 (7)  C33—C34—C35 118.8 (7)
C7—C6—C5 124.0 (7)  C34—C35—C36 118.8 (7)
C6—C7—H7 119.9  C35—C36—C37 118.8 (7)
C8—C7—H7 120.3 (7)  C36—C37—C38 118.8 (7)

Zn1—O1—N1—C1 127.4 (5)  C4—C5—C6—C1 0.5 (11)
Zn1—O1—N1—C9 −54.6 (8)  C4—C5—C6—C7 −178.7 (8)
Zn1—O2—N2—C10 −94.8 (6)  C5—C6—C7—C8 179.3 (7)
supporting information

Dibromidobis(quinoline N-oxide-κO)zinc(II) (II)

Crystal data

-ZnBr₂(C₉H₇NO)₂-

Mr = 515.50

Monoclinic, P₂₁/c

a = 16.3922 (11) Å

b = 7.3527 (6) Å

C = 15.5809 (10) Å

β = 97.113 (6)°

V = 1863.5 (2) Å³

Z = 4

F(000) = 1008

Dx = 1.837 Mg m⁻³

Mo Kα radiation, λ = 0.71073 Å

Cell parameters from 1219 reflections

θ = 2.6–22.0°

µ = 5.62 mm⁻¹

T = 298 K

Irregular, clear colourless

0.15 × 0.08 × 0.03 mm

Data collection

XtaLAB Mini (ROW) diffractometer

Radiation source: fine-focus sealed X-ray tube, Rigaku (Mo) X-ray Source

Graphite monochromator

ω scans

Absorption correction: multi-scan

(CrysAlisPro; Rigaku OD, 2019)

Tmin = 0.833, Tmax = 1.000

7207 measured reflections

3415 independent reflections

2095 reflections with I > 2σ(I)

Rint = 0.043

θmax = 25.4°, θmin = 2.5°

h = −16→19

k = −8→8

l = −18→18

Refinement

Refinement on F²

Least-squares matrix: full

R[F² > 2σ(F²)] = 0.042

wR(F²) = 0.090

S = 1.01

3415 reflections

226 parameters

Primary atom site location: dual

Hydrogen site location: inferred from neighbouring sites

H-atom parameters constrained

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sup-5
\[ w = \frac{1}{\sigma^2(F_o^2) + (0.0258 \cdot P)^2} \]
\[ \Delta \rho_{\text{max}} = 0.55 \text{ e Å}^{-3} \]
\[ \Delta \rho_{\text{min}} = -0.35 \text{ e Å}^{-3} \]
\[ (\Delta/\sigma)_{\text{max}} < 0.001 \]

**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 (Å²)**

| Atom | x    | y    | z    | Uiso */Ueq |
|------|------|------|------|------------|
| Zn1  | 0.25508 (4) | 0.26213 (9) | 0.37264 (4) | 0.0514 (2) |
| Br2  | 0.22409 (4) | −0.03623 (9) | 0.41131 (4) | 0.0695 (2) |
| Br1  | 0.26119 (4) | 0.35514 (10) | 0.22878 (4) | 0.0698 (2) |
| O1   | 0.3616 (2) | 0.3196 (6) | 0.4411 (2) | 0.0662 (11) |
| O2   | 0.1778 (2) | 0.4332 (5) | 0.4197 (2) | 0.0597 (10) |
| N2   | 0.1157 (3) | 0.3586 (5) | 0.4557 (3) | 0.0445 (11) |
| N1   | 0.4115 (3) | 0.4386 (7) | 0.4079 (3) | 0.0543 (12) |
| C10  | 0.0394 (3) | 0.3446 (7) | 0.4065 (3) | 0.0415 (12) |
| C7   | 0.5136 (4) | 0.1490 (10) | 0.4098 (4) | 0.093 (2) |
| C8   | 0.607681 | 0.030702 | 0.424267 | 0.099* |
| C5   | 0.6244 (4) | 0.4382 (12) | 0.3568 (4) | 0.085 (2) |
### Atomic displacement parameters (Å²)

|       | U¹¹ | U¹² | U¹³ | U¹² | U¹³ | U¹³ |
|-------|-----|-----|-----|-----|-----|-----|
| Zn1   | 0.0345 (4) | 0.0688 (5) | 0.0519 (4) | -0.0003 (3) | 0.0089 (3) | -0.0017 (3) |
| Br2   | 0.0598 (4) | 0.0637 (4) | 0.0855 (5) | 0.0040 (3) | 0.0113 (3) | 0.0048 (4) |
| Br1   | 0.0663 (4) | 0.0960 (5) | 0.0476 (3) | -0.0052 (4) | 0.0090 (3) | -0.0030 (4) |
| O1    | 0.037 (2) | 0.102 (3) | 0.059 (2) | -0.016 (2) | 0.0020 (18) | 0.010 (2) |
| O2    | 0.046 (2) | 0.055 (2) | 0.083 (3) | -0.005 (2) | 0.0278 (19) | 0.002 (2) |
| N2    | 0.040 (3) | 0.042 (3) | 0.054 (3) | 0.007 (2) | 0.014 (2) | -0.005 (2) |
| N1    | 0.043 (3) | 0.072 (4) | 0.045 (3) | -0.005 (3) | -0.007 (2) | -0.012 (3) |
| C10   | 0.043 (3) | 0.039 (3) | 0.043 (3) | 0.007 (3) | 0.011 (2) | -0.006 (3) |
| C1    | 0.039 (3) | 0.062 (4) | 0.037 (3) | -0.005 (3) | -0.002 (2) | -0.011 (3) |
| C15   | 0.041 (3) | 0.048 (3) | 0.054 (3) | 0.003 (3) | 0.015 (3) | -0.003 (3) |
| C16   | 0.051 (4) | 0.060 (4) | 0.057 (4) | -0.001 (3) | 0.020 (3) | 0.006 (3) |
| C18   | 0.053 (4) | 0.056 (4) | 0.047 (3) | 0.009 (3) | -0.003 (3) | -0.004 (3) |
| C17   | 0.063 (4) | 0.062 (4) | 0.046 (3) | 0.007 (3) | 0.017 (3) | 0.008 (3) |
| C11   | 0.064 (4) | 0.063 (4) | 0.046 (3) | 0.008 (3) | 0.015 (3) | -0.003 (3) |
| C6    | 0.051 (4) | 0.076 (5) | 0.049 (3) | -0.016 (4) | 0.001 (3) | -0.015 (3) |
| C2    | 0.053 (4) | 0.072 (5) | 0.059 (4) | -0.003 (3) | -0.007 (3) | -0.003 (3) |
| C14   | 0.042 (4) | 0.079 (5) | 0.081 (5) | -0.008 (3) | 0.008 (3) | 0.000 (4) |
| C13   | 0.055 (4) | 0.092 (5) | 0.072 (4) | 0.002 (4) | -0.007 (3) | -0.006 (4) |
| C12   | 0.085 (5) | 0.094 (5) | 0.037 (3) | 0.016 (4) | 0.002 (3) | -0.002 (3) |
| C9    | 0.052 (4) | 0.088 (5) | 0.075 (4) | 0.009 (4) | -0.010 (3) | -0.028 (4) |
| C7    | 0.086 (6) | 0.074 (5) | 0.071 (4) | -0.029 (4) | 0.002 (4) | -0.003 (4) |
| C3    | 0.064 (5) | 0.079 (5) | 0.101 (5) | 0.010 (4) | -0.007 (4) | -0.016 (4) |
| C8    | 0.098 (6) | 0.053 (4) | 0.087 (5) | -0.003 (5) | -0.028 (5) | 0.000 (4) |
| C5    | 0.056 (5) | 0.122 (7) | 0.079 (5) | -0.035 (5) | 0.022 (4) | -0.023 (5) |
| C4    | 0.050 (5) | 0.130 (7) | 0.104 (6) | 0.006 (5) | 0.008 (4) | -0.029 (6) |

### Geometric parameters (Å, °)

|       |        |        |        |        |        |
|-------|--------|--------|--------|--------|--------|
| Zn1—Br2 | 2.3472 (10) | C11—H11 | 0.9300 |
| Zn1—Br1 | 2.3575 (8) | C11—C12 | 1.364 (8) |
| Zn1—O1 | 1.975 (3) | C6—C7 | 1.395 (8) |
| Zn1—O2 | 1.989 (4) | C6—C5 | 1.422 (9) |
| O1—N1 | 1.345 (5) | C2—H2 | 0.9300 |
| O2—N2 | 1.339 (5) | C2—C3 | 1.356 (8) |
| N2—C10 | 1.388 (6) | C14—H14 | 0.9300 |
| N2—C18 | 1.323 (6) | C14—C13 | 1.352 (8) |
| N1—C1 | 1.386 (6) | C13—H13 | 0.9300 |
| N1—C9 | 1.313 (7) | C13—C12 | 1.392 (8) |
| C10—C15 | 1.406 (7) | C12—H12 | 0.9300 |
| C10—C11 | 1.402 (7) | C9—H9 | 0.9300 |
| C1—C6 | 1.410 (7) | C9—C8 | 1.400 (9) |
| Bond          | Distance (Å) | Bond          | Distance (Å) | Angle (°)  |
|--------------|--------------|--------------|--------------|-----------|
| C1—C2        | 1.380 (7)    | C7—H7        | 0.9300       |           |
| C15—C16      | 1.408 (7)    | C7—C8        | 1.354 (9)    |           |
| C15—C14      | 1.405 (7)    | C3—H3        | 0.9300       |           |
| C16—H16      | 0.9300       | C3—C4        | 1.378 (10)   |           |
| C16—C17      | 1.346 (7)    | C8—H8        | 0.9300       |           |
| C18—H18      | 0.9300       | C5—H5        | 0.9300       |           |
| C18—C17      | 1.387 (7)    | C5—C4        | 1.338 (9)    |           |
| C17—H17      | 0.9300       | C4—H4        | 0.9300       |           |

| Bond                  | Distance (Å) | Bond                  | Distance (Å) | Angle (°)  |
|-----------------------|--------------|-----------------------|--------------|-----------|
| Br2—Zn1—Br1          | 123.45 (4)   | C12—C11—H11          | 121.0        |           |
| O1—Zn1—Br2           | 105.44 (12)  | C1—C6—C5            | 116.5 (6)    |           |
| O1—Zn1—Br1           | 108.21 (11)  | C7—C6—C1            | 119.2 (6)    |           |
| O1—Zn1—O2            | 103.10 (16)  | C7—C6—C5            | 124.2 (7)    |           |
| O2—Zn1—Br2           | 109.17 (11)  | C1—C2—H2           | 120.4        |           |
| O2—Zn1—Br1           | 105.72 (11)  | C3—C2—C1            | 119.3 (6)    |           |
| N1—O1—Zn1            | 118.1 (3)    | C3—C2—H2            | 120.4        |           |
| N2—O2—Zn1            | 116.6 (3)    | C15—C14—H14         | 119.6        |           |
| O2—N2—C10            | 118.5 (4)    | C13—C14—C15         | 120.8 (6)    |           |
| C18—N2—O2            | 120.1 (4)    | C13—C14—H14         | 119.6        |           |
| C18—N2—C10           | 121.4 (5)    | C14—C13—H13         | 119.9        |           |
| O1—N1—C1             | 117.1 (5)    | C14—C13—C12         | 120.2 (6)    |           |
| C9—N1—O1             | 120.5 (5)    | C12—C13—H13         | 119.9        |           |
| C9—N1—C1             | 122.4 (6)    | C11—C12—C13         | 121.8 (6)    |           |
| N2—C10—C15           | 118.6 (5)    | C11—C12—H12         | 119.1        |           |
| N2—C10—C11           | 120.1 (5)    | C13—C12—H12        | 119.1        |           |
| C11—C10—C15          | 121.3 (5)    | N1—C9—H9           | 119.9        |           |
| N1—C1—C6             | 118.0 (6)    | N1—C9—C8           | 120.2 (6)    |           |
| C2—C1—N1             | 120.5 (5)    | C8—C9—H9           | 119.9        |           |
| C2—C1—C6             | 121.5 (6)    | C6—C7—H7           | 120.0        |           |
| C10—C15—C16          | 118.6 (5)    | C8—C7—C6           | 119.9 (7)    |           |
| C14—C15—C10          | 117.9 (5)    | C8—C7—H7           | 120.0        |           |
| C14—C15—C16          | 123.6 (5)    | C2—C3—H3           | 119.7        |           |
| C15—C16—H16          | 119.8        | C2—C3—C4           | 120.7 (7)    |           |
| C17—C16—C15          | 120.3 (5)    | C4—C3—H3           | 119.7        |           |
| C17—C16—H16          | 119.8        | C9—C8—H8           | 119.9        |           |
| N2—C18—H18           | 119.4        | C7—C8—C9           | 120.2 (7)    |           |
| N2—C18—C17           | 121.1 (5)    | C7—C8—H8           | 119.9        |           |
| C17—C18—H18          | 119.4        | C6—C5—H5           | 119.7        |           |
| C16—C17—C18          | 120.0 (5)    | C4—C5—C6           | 120.5 (7)    |           |
| C16—C17—H17          | 120.0        | C4—C5—H5           | 119.7        |           |
| C18—C17—H17          | 120.0        | C3—C4—H4           | 119.3        |           |
| C10—C11—H11          | 121.0        | C5—C4—C3           | 121.5 (7)    |           |
| C12—C11—C10          | 118.0 (6)    | C5—C4—H4           | 119.3        |           |

| Angle                  |           | Angle                  |           |
|-----------------------|-----------|-----------------------|-----------|
| Zn1—O1—N1—C1         | −122.3 (4) | C1—C6—C7—C8         | 1.1 (9)   |
| Zn1—O1—N1—C9         | 57.8 (6)   | C1—C6—C5—C4        | 0.7 (9)   |
| Zn1—O2—N2—C10        | −97.8 (4)  | C1—C2—C3—C4        | 0.2 (9)   |
| Zn1—O2—N2—C18        | 83.4 (5)   | C15—C10—C11—C12     | −2.0 (8)  |
O1—N1—C1—C6 −178.5 (4) C15—C16—C17—C18 0.9 (9)
O1—N1—C1—C2 0.7 (7) C15—C14—C13—C12 0.3 (10)
O1—N1—C9—C8 179.3 (5) C16—C15—C14—C13 178.8 (6)
O2—N2—C10—C15 −178.1 (4) C18—N2—C10—C15 0.6 (7)
O2—N2—C10—C11 −0.3 (7) C18—N2—C10—C11 178.4 (5)
O2—N2—C18—C17 178.4 (5) C11—C10—C15—C14 2.6 (8)
N2—C10—C11—C12 −179.7 (5) C6—C1—C2—C3 1.1 (8)
N2—C10—C11—C12 −0.5 (8) C6—C7—C8—C9 −0.3 (10)
N1—C9—C8—C7 0.1 (10) C14—C15—C16—C17 178.9 (6)
C10—N2—C18—C17 −0.3 (8) C14—C13—C12—C11 0.4 (10)
C10—C15—C14—C13 −1.7 (9) C9—N1—C1—C6 1.4 (7)
C10—C11—C12—C13 0.5 (9) C9—N1—C1—C2 −179.4 (5)
C1—N1—C9—C8 −0.6 (8) C7—C6—C5—C4 179.9 (6)

Diiododobis(quinoline N-oxide-κO)zinc(II) (III)

Crystal data

[ZnI2(C9H7NO)2]

F(000) = 1152

Mr = 609.48

Monoclinic, P21/c

a = 16.7231 (7) Å

b = 7.6155 (4) Å

c = 15.8689 (7) Å

β = 97.192 (4)°

V = 2005.08 (16) Å³

Z = 4

Data collection

Rigaku XtaLAB mini
diffractometer
3668 independent reflections

ω scans
2748 reflections with I > 2σ(I)

Absorption correction: multi-scan
θmax = 25.4°, θmin = 2.5°

(CrysAlisPro; Rigaku OD, 2019)

Tmin = 0.896, Tmax = 1.000

11510 measured reflections

Refinement

Refinement on F²

R[F² > 2σ(F²)] = 0.035

wR(F²) = 0.085

S = 1.07

3668 reflections

227 parameters

0 restraints

Hydrogen site location: inferred from neighbouring sites

H-atom parameters constrained

w = 1/[σ²(F³) + (0.0249P)² + 3.8317P]

where P = (F³² + 2Fc²)/3

(Δ/σ)max < 0.001

Δρmax = 0.80 e Å⁻³

Δρmin = −0.81 e Å⁻³

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sup-9
Extinction correction: SHELXL-2018/1 (Sheldrick 2015a),

\[ F_c^* = kF_c[1 + 0.001xF_c^2\lambda^3/sin(2\theta)]^{-1/4} \]

Extinction coefficient: 0.00071 (11)

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 (Å²)

|    | x          | y          | z          | Uiso*/Ueq |
|----|------------|------------|------------|-----------|
| I1 | 0.26214 (2) | 0.37499 (6) | 0.22453 (2) | 0.07021 (17) |
| I2 | 0.22463 (3) | −0.02923 (6) | 0.41988 (3) | 0.07442 (17) |
| Zn1| 0.25578 (3) | 0.28426 (10) | 0.37845 (4) | 0.0553 (2) |
| O1 | 0.3601 (2)  | 0.3426 (7)  | 0.4449 (3)  | 0.0780 (13) |
| O2 | 0.1777 (2)  | 0.4466 (5)  | 0.4234 (3)  | 0.0627 (10) |
| N1 | 0.4094 (3)  | 0.4544 (7)  | 0.4109 (3)  | 0.0592 (12) |
| N2 | 0.1160 (3)  | 0.3728 (6)  | 0.4566 (3)  | 0.0509 (11) |
| C1 | 0.4847 (3)  | 0.3925 (8)  | 0.3984 (3)  | 0.0545 (14) |
| C2 | 0.5069 (4)  | 0.2175 (9)  | 0.4180 (4)  | 0.0707 (17) |
| H2 | 0.470599    | 0.140389    | 0.438341    | 0.085*     |
| C3 | 0.5817 (5)  | 0.1635 (11) | 0.4068 (5)  | 0.095 (2)  |
| H3 | 0.596812    | 0.048098    | 0.419687    | 0.114*     |
| C4 | 0.6360 (5)  | 0.2760 (13) | 0.3768 (6)  | 0.105 (3)  |
| H4 | 0.687500    | 0.235837    | 0.370683    | 0.126*     |
| C5 | 0.6159 (4)  | 0.4433 (12) | 0.3560 (5)  | 0.088 (2)  |
| H5 | 0.653406    | 0.516920    | 0.335438    | 0.106*     |
| C6 | 0.5378 (4)  | 0.5077 (9)  | 0.3652 (4)  | 0.0641 (16) |
| C7 | 0.5127 (5)  | 0.6797 (10) | 0.3444 (5)  | 0.082 (2)  |
| H7 | 0.547198    | 0.757244    | 0.321637    | 0.099*     |
| C8 | 0.4374 (5)  | 0.7327 (10) | 0.3576 (5)  | 0.085 (2)  |
| H8 | 0.419938    | 0.846144    | 0.343511    | 0.102*     |
| C9 | 0.3871 (4)  | 0.6157 (10) | 0.3923 (4)  | 0.0758 (19) |
| H9 | 0.336222    | 0.652818    | 0.402601    | 0.091*     |
| C10| 0.0431 (3)  | 0.3575 (7)  | 0.4054 (3)  | 0.0487 (12) |
| C11| 0.0343 (4)  | 0.4227 (8)  | 0.3219 (4)  | 0.0638 (16) |
| H11| 0.077050    | 0.475911    | 0.299604    | 0.077*     |
| C12| −0.0388 (5) | 0.4051 (10) | 0.2751 (4)  | 0.081 (2)  |
| H12| −0.046101   | 0.448351    | 0.219880    | 0.098*     |
| C13| −0.1039 (4) | 0.3237 (11) | 0.3073 (5)  | 0.088 (2)  |
| H13| −0.153042   | 0.311918    | 0.273148    | 0.106*     |
| C14| −0.0955 (4) | 0.2624 (9)  | 0.3879 (5)  | 0.0755 (19) |
| H14| −0.138783   | 0.208570    | 0.408920    | 0.091*     |
| C15| −0.0218 (3) | 0.2796 (7)  | 0.4398 (4)  | 0.0534 (13) |
| C16| −0.0094 (4) | 0.2205 (8)  | 0.5249 (4)  | 0.0632 (16) |
| H16| −0.051513   | 0.168071    | 0.548760    | 0.076*     |
|   | \( \text{C17} \) | \( \text{H17} \) | \( \text{C18} \) | \( \text{H18} \) |
|---|---|---|---|---|
|   | 0.0633 (4) | 0.2398 (9) | 0.5717 (4) | 0.0666 (17) |
|   | 0.071247 | 0.201324 | 0.627730 | 0.080* |
|   | 0.1257 (4) | 0.3171 (8) | 0.5358 (4) | 0.0589 (15) |
|   | 0.175593 | 0.330122 | 0.568251 | 0.071* |

### Atomic displacement parameters (Å²)

|   | \( U^{11} \) | \( U^{22} \) | \( U^{33} \) | \( U^{12} \) | \( U^{13} \) | \( U^{23} \) |
|---|---|---|---|---|---|---|
| I1 | 0.0640 (3) | 0.0961 (4) | 0.0508 (2) | −0.0078 (2) | 0.00801 (19) | −0.0018 (2) |
| I2 | 0.0652 (3) | 0.0668 (3) | 0.0928 (3) | 0.0106 (2) | 0.0157 (2) | 0.0090 (2) |
| Zn1 | 0.0377 (3) | 0.0742 (5) | 0.0542 (4) | −0.0006 (3) | 0.0070 (3) | −0.0005 (3) |
| O1 | 0.048 (2) | 0.122 (4) | 0.063 (3) | −0.014 (2) | 0.002 (2) | 0.006 (3) |
| O2 | 0.055 (2) | 0.059 (3) | 0.078 (3) | −0.0007 (19) | 0.024 (2) | 0.003 (2) |
| N1 | 0.043 (3) | 0.079 (4) | 0.052 (3) | −0.001 (3) | −0.003 (2) | −0.012 (3) |
| N2 | 0.047 (2) | 0.052 (3) | 0.057 (3) | 0.007 (2) | 0.016 (2) | −0.001 (2) |
| C1 | 0.042 (3) | 0.075 (4) | 0.044 (3) | −0.001 (3) | −0.005 (2) | −0.014 (3) |
| C2 | 0.066 (4) | 0.069 (5) | 0.073 (4) | −0.001 (3) | −0.007 (3) | −0.004 (3) |
| C3 | 0.073 (5) | 0.086 (6) | 0.120 (7) | 0.013 (4) | −0.009 (5) | −0.026 (5) |
| C4 | 0.064 (5) | 0.114 (7) | 0.136 (8) | 0.012 (5) | 0.011 (5) | −0.054 (6) |
| C5 | 0.061 (4) | 0.110 (7) | 0.097 (6) | −0.016 (4) | 0.021 (4) | −0.026 (5) |
| C6 | 0.053 (3) | 0.073 (5) | 0.066 (4) | −0.011 (3) | 0.006 (3) | −0.017 (3) |
| C7 | 0.083 (5) | 0.076 (5) | 0.086 (5) | −0.022 (4) | 0.001 (4) | −0.008 (4) |
| C8 | 0.087 (5) | 0.065 (5) | 0.095 (5) | 0.004 (4) | −0.018 (4) | −0.013 (4) |
| C9 | 0.063 (4) | 0.084 (5) | 0.076 (4) | 0.008 (4) | −0.011 (4) | −0.024 (4) |
| C10 | 0.051 (3) | 0.047 (3) | 0.049 (3) | 0.007 (2) | 0.013 (3) | 0.000 (2) |
| C11 | 0.070 (4) | 0.071 (4) | 0.051 (3) | 0.005 (3) | 0.012 (3) | 0.003 (3) |
| C12 | 0.092 (5) | 0.099 (6) | 0.052 (4) | 0.014 (4) | 0.002 (4) | −0.001 (4) |
| C13 | 0.065 (4) | 0.110 (6) | 0.085 (5) | 0.005 (4) | −0.015 (4) | −0.006 (5) |
| C14 | 0.056 (4) | 0.083 (5) | 0.086 (5) | −0.008 (3) | 0.004 (4) | −0.010 (4) |
| C15 | 0.050 (3) | 0.053 (3) | 0.058 (3) | 0.004 (3) | 0.011 (3) | −0.003 (3) |
| C16 | 0.062 (4) | 0.060 (4) | 0.071 (4) | 0.005 (3) | 0.025 (3) | 0.013 (3) |
| C17 | 0.067 (4) | 0.080 (5) | 0.056 (4) | 0.014 (3) | 0.016 (3) | 0.011 (3) |
| C18 | 0.056 (3) | 0.069 (4) | 0.051 (3) | 0.009 (3) | 0.005 (3) | 0.000 (3) |

### Geometric parameters (Å, °)

|   | \( \text{I1—Zn1} \) | \( \text{I2—Zn1} \) | \( \text{Zn1—O1} \) | \( \text{Zn1—O2} \) | \( \text{O1—N1} \) | \( \text{O2—N2} \) | \( \text{N1—C1} \) | \( \text{N1—C9} \) | \( \text{N2—C10} \) | \( \text{N2—C18} \) | \( \text{C1—C2} \) | \( \text{C1—C6} \) |
|---|---|---|---|---|---|---|---|---|---|---|---|---|
|   | 2.5534 (8) | 2.5473 (9) | 1.973 (4) | 1.994 (4) | 1.345 (6) | 1.339 (5) | 1.381 (7) | 1.307 (8) | 1.383 (7) | 1.317 (7) | 1.408 (9) | 1.397 (8) |
|   | C7—H7 | C7—C8 | C8—H8 | C8—C9 | C9—H9 | C10—C11 | C10—C15 | C11—H11 | C11—C12 | C12—H12 | C12—C13 | C13—H13 |
C2—H2 0.9300 C13—C14 1.352 (10)
C2—C3 1.350 (9) C14—H14 0.9300
C3—H3 0.9300 C15—C16 1.414 (8)
C3—C4 1.377 (12) C16—H16 0.9300
C4—H4 0.9300 C17—H17 0.9300
C4—C5 1.348 (12) C17—C18 1.382 (8)
C5—H5 0.9300 C18—H18 0.9300
C5—C6 1.420 (9)
C6—C7 1.402 (10)

I2—Zn1—I1 122.33 (3) C8—C7—H7 120.2
O1—Zn1—I1 108.10 (13) C7—C8—H8 120.4
O1—Zn1—I2 105.60 (15) C7—C8—C9 119.3 (7)
O1—Zn1—O2 104.13 (19) C9—C8—H8 120.4
O2—Zn1—I1 106.36 (12) C7—C8—C9 121.6 (7)
O2—Zn1—I2 108.93 (12) C9—C8—C10 119.2
N1—O1—Zn1 118.3 (3) C8—C9—H9 119.2
N2—O2—Zn1 116.9 (3) N2—C10—C11 120.3 (5)
O1—N1—C1 117.2 (5) N2—C10—C15 118.3 (5)
C9—N1—O1 120.9 (5) C15—C10—C11 121.4 (5)
C9—N1—C1 121.9 (6) C10—C11—H11 121.2
O2—N2—C10 118.0 (4) C12—C11—C10 117.6 (6)
C18—N2—C10 120.2 (5) C12—C11—H11 121.2
C18—N2—C10 121.8 (5) C11—C12—C13 118.9
N1—C1—C2 120.7 (6) C11—C12—H12 122.1 (6)
N1—C1—C6 118.3 (6) C13—C12—H12 118.9
C6—C1—C2 121.0 (6) C12—C13—C12 119.9
C1—C2—H2 120.6 C12—C13—H13 120.2 (7)
C3—C2—C1 118.8 (7) C13—C14—C15 119.9
C3—C2—H2 120.6 C14—C13—H13 119.9
C2—C3—H3 119.4 C13—C14—H14 119.8
C2—C3—C4 121.3 (8) C14—C15—C16 118.5 (5)
C4—C3—H3 119.4 C14—C15—C10 118.4 (5)
C3—C4—H4 119.4 C15—C16—C17 120.3 (6)
C5—C4—C3 121.2 (8) C15—C16—H16 119.9
C5—C4—H4 119.4 C16—C17—H17 120.2
C4—C5—H5 119.8 C17—C16—C15 120.3 (6)
C4—C5—C6 120.4 (8) C17—C16—H16 119.9
C6—C5—H5 119.8 C16—C17—C18 119.6 (6)
C1—C6—C5 117.3 (7) C16—C17—H17 120.2
C1—C6—C7 119.3 (6) C18—C17—H17 120.2
C7—C6—C5 123.4 (7) N2—C18—C17 121.5 (6)
C6—C7—H7 120.2 N2—C18—H18 119.3
C8—C7—C6 119.7 (7) C17—C18—H18 119.3

Zn1—O1—N1—C1 −119.9 (4) C4—C5—C6—C1 1.6 (10)
Zn1—O1—N1—C9 61.5 (6) C4—C5—C6—C7 −179.6 (7)
Zn1—O2—N2—C10 −96.9 (5) C5—C6—C7—C8 −178.0 (7)
| Bond                  | Angle (deg) (E) | Bond                  | Angle (deg) (E) | Bond                  | Angle (deg) (E) |
|----------------------|-----------------|----------------------|-----------------|----------------------|-----------------|
| Zn1—O2—N2—C18       | 83.1 (5)        | C6—C1—C2—C3         | 2.1 (9)         |                       |                 |
| O1—N1—C1—C2         | 2.2 (7)         | C6—C7—C8—C9         | 0.6 (11)        |                       |                 |
| O1—N1—C1—C6         | −178.3 (5)      | C7—C8—C9—N1         | −1.6 (11)       |                       |                 |
| O1—N1—C9—C8         | 179.7 (5)       | C9—N1—C1—C2         | −179.2 (6)      |                       |                 |
| O2—N2—C10—C11       | −1.7 (7)        | C9—N1—C1—C6         | 0.3 (8)         |                       |                 |
| O2—N2—C10—C15       | 179.7 (5)       | C10—N2—C18—C17      | 0.3 (9)         |                       |                 |
| O2—N2—C18—C17       | −179.7 (5)      | C10—C11—C12—C13     | −0.6 (11)       |                       |                 |
| N1—C1—C2—C3         | −178.4 (6)      | C10—C15—C16—C17     | 0.3 (9)         |                       |                 |
| N1—C1—C6—C5         | 177.6 (5)       | C11—C10—C15—C14     | 2.1 (9)         |                       |                 |
| N1—C1—C6—C7         | −1.2 (8)        | C11—C10—C15—C16     | −178.5 (5)      |                       |                 |
| N2—C10—C11—C12      | −179.5 (6)      | C11—C12—C13—C14     | 1.2 (12)        |                       |                 |
| N2—C10—C15—C14      | −179.4 (5)      | C12—C13—C14—C15     | 0.0 (12)        |                       |                 |
| N2—C10—C15—C16      | 0.0 (8)         | C13—C14—C15—C10     | −1.6 (10)       |                       |                 |
| C1—N1—C9—C8         | 1.1 (9)         | C13—C14—C15—C16     | 179.1 (7)       |                       |                 |
| C1—C2—C3—C4         | −0.1 (11)       | C14—C15—C16—C17     | 179.6 (6)       |                       |                 |
| C1—C6—C7—C8         | 0.7 (10)        | C15—C10—C11—C12     | −1.0 (9)        |                       |                 |
| C2—C1—C6—C5         | −2.9 (9)        | C15—C16—C17—C18     | −0.3 (10)       |                       |                 |
| C2—C1—C6—C7         | 178.3 (6)       | C16—C17—C18—N2      | 0.0 (10)        |                       |                 |
| C2—C3—C4—C5         | −1.2 (13)       | C18—N2—C10—C11      | 178.3 (5)       |                       |                 |
| C3—C4—C5—C6         | 0.4 (13)        | C18—N2—C10—C15      | −0.2 (8)        |                       |                 |