Synthesis and crystal structure of a new chiral \( \alpha \)-aminooxime nickel(II) complex

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A dinuclear nickel complex with (S)-limonene based aminooxime ligand has been isolated and its crystal structure determined. The resolved structure of dichloridobis\{(2\(S\),5\(R\)-2-methyl-5-(prop-1-en-2-yl)-2-[(pyridin-2-yl)methylamino]cyclohexan-1-one oxime\}dinickel(II), \([\text{Ni}_2\text{Cl}_2\text{C}_{16}\text{H}_{23}\text{ClN}_3\text{O}_2]\), at 100 K has monoclinic (\(P2_1\)) symmetry. The two Ni\(^{II}\) ions in the dinuclear complex are each coordinated in a distorted octahedral environment by three nitrogen atoms, a terminal chloride and two \(\mu\)-chloro bridging chlorides. Each oxime ligand is coordinated to nickel(II) by the three nitrogen atoms, leading to two five-membered chelate rings, each displaying an envelope conformation. In the crystal, numerous intermolecular and intramolecular hydrogen bonds lead to the formation of a three-dimensional network structure.

1. Chemical context

Asymmetric synthesis allows the preparation of enantiomerically enriched compounds either by using a chiral auxiliary, which will be temporarily introduced, or by using catalytic procedures (Gawley & Aubé, 2012). This latter method is particularly attractive as it contributes to the development of green chemistry, which maximizes efficiency and minimizes hazardous effects on human health and the environment (Anastas & Zimmerman, 2013). Thus, asymmetric catalysis avoids synthetic steps and only catalytic amounts of the optically pure auxiliary are needed (Ojima, 2010). As part of the development of this chemistry, the synthesis of new chiral organometallic complexes is always challenging. The pivotal point is then the synthesis of optically pure ligands, which will be coordinated to the metal center. In terms of sustainable chemistry, using the chiral pool to develop new ligands is most interesting (Elalami \textit{et al.}, 2015). Coordination metal complexes containing terpenoid fragments are widely used in the pharmaceutical field and in catalysis. We have therefore developed ligands based on terpenes such as pinene and limonene (El Alami \textit{et al.}, 2009, 2015; Chahboun \textit{et al.}, 2012). In particular, the synthesis of optically pure aminooxime ligands has been performed successfully from \(\alpha\)-limonene (El Alami \textit{et al.}, 2012). These compounds possess structures with two or three nitrogen atoms as donor heteroatoms that could coordinate to the metal center. They have advantageously replaced phosphine ligands, which are generally unstable under air. Ruthenium (Benabdelouahab \textit{et al.}, 2015) and palladium (de la Cueva-Alique \textit{et al.}, 2019)
complexes have already been synthesized with these ligands. Here we report the first synthesis of a limonene-based α-aminooxime nickel complex and its crystal structure. In the dinuclear title complex, each nickel ion is coordinated by \((1\text{S},4\text{R})\)-1-picolylamino-p-menth-8-en-2-one oxime. The ligand was first synthesized from \((R)\)-limonene through the addition of nitrosyl chloride, NOCl, to a picolylamine moiety, allowing the formation of the oxime moiety.

2. Structural commentary

The title compound (Fig. 1) crystallizes in the monoclinic space group \(P2_1\) with two chiral molecules per unit cell. The two Ni\(^{II}\) ions in the dinuclear complex are each coordinated by three nitrogen atoms, a terminal chloride and two \(\mu\) bridging chlorides. The environment around each metal center can then be described as a distorted octahedron with Ni—Ni—Cl angles of 79.91 (13) and 91.99 (4)\(^\circ\), respectively, together with Cl1—Ni1—N2 and Cl2—Ni1—N1 angles of 165.04 (11) and 88.69 (10)\(^\circ\), respectively. A similar arrangement can be found around the Ni2 atom \([\text{Ni4—Ni2—N5, Cl2—Ni2—Cl4, Cl4—Ni2—N5 and Cl4—Ni2—N4} = 79.7 (2), 99.38 (4), 166.04 (12) and 93.24 (16)\(^\circ\), respectively].

Each aminooxime ligand is coordinated to nickel(II) by the three nitrogen atoms, leading to two five-membered chelate rings, each displaying an envelope conformation (with N2 as the flap for Ni1/N1/C5/C6/N2 and N5 for Ni2/N4/C21/C22/N5). The six-membered carbocycles of the limonene units adopt a chair conformation. The lengths of the Ni1—N1, Ni1—N2 and Ni1—N3 bonds are 2.077 (3), 2.126 (4) and 2.041 (3) Å, respectively, while Ni2—N4, Ni2—N5 and Ni2—N6 are 2.095 (4), 2.103 (4) and 2.027 (3) Å from the metal centers Ni1 and Ni2, respectively. The two metal centers are linked by two bridging Cl atoms with an average Ni—Cl distance of 2.42 Å, which is normal for these bond lengths. All these values compare well with literature values. The two nickel ions are separated by a distance of 3.5198 (7) Å, which is similar to average values (Zheng et al., 2010; Cheng et al., 2012).

3. Supramolecular features

The crystal structure is stabilized by numerous intermolecular and intramolecular hydrogen bonds (Table 1), which link the component into a three-dimensional network (Figs. 2 and 3).

![Figure 1](Image)

**Figure 1**
Displacement ellipsoid plot at the 50% probability level for Ni\(_2\)(aminooxime)\(_2\)Cl\(_4\). H atoms are omitted for clarity.

![Figure 2](Image)

**Figure 2**
Intermolecular and intramolecular hydrogen bonds in the structure, shown as dashed lines.

| D—H···A | D—H | H···A | D···A | D—H···A |
|---------|------|-------|-------|---------|
| O1—H1···Cl1 | 0.85 (7) | 2.32 (6) | 3.009 (4) | 139 (6) |
| N2—H2···Cl4 | 0.77 (5) | 2.46 (5) | 3.209 (4) | 166 (5) |
| O2—H2···Cl4 | 0.76 (8) | 2.31 (7) | 2.978 (4) | 147 (7) |
| C3—H3···O1' | 0.95 | 2.58 | 3.432 (5) | 149 |
| C1—H1···Cl1 | 0.95 | 2.75 | 3.369 (5) | 124 |
| C6—H6A···Cl2 | 0.99 | 2.76 | 3.309 (5) | 115 |
| C11—H11B···Cl3 | 0.99 | 2.64 | 3.573 (5) | 156 |
| C17—H17···Cl4 | 0.95 | 2.69 | 3.327 (6) | 125 |
| C26—H26···O2iii | 1.00 | 2.56 | 3.489 (6) | 154 |
| C22—H22B···Cl2 | 0.99 | 2.81 | 3.352 (6) | 115 |
| C19—H19···Cliv | 0.95 | 2.64 | 3.570 (7) | 167 |

Symmetry codes: (i) \(-x + 2, y - \frac{1}{2}, -z + 1\); (ii) \(-x + 1, y - \frac{1}{2}, -z + 1\); (iii) \(-x, y + \frac{1}{2}, -z\); (iv) \(-x + 1, y - \frac{1}{2}, z\).
In particular, the two \([\text{Ni(aminooxime)}\mu\text{-Cl}]{\text{Cl}}\) units are slightly asymmetrical with the existence of a hydrogen-bonding interaction between the amine N2—H2 linked to Ni1 and the chlorine atom Cl4 linked to Ni2. In addition, the two oxygen atoms O1 and O2 of the oxime groups are involved in intramolecular O1—H1/C1/C1/Cl1 and O2—H2/A/C1/C1/Cl4 hydrogen bonds and in intermolecular C3—H3/C1/C1/C1 and C26—H26/C1/C1/C1 interactions.

4. Database survey

The aminooxime ligand used in this study was previously reacted with palladium and platinum precursors, generating three N-coordinated cationic complexes as enantiopure compounds (de la Cueva-Alique et al., 2019). A heteronuclear TiIV/PdII complex has also been described. The compounds were studied to assess their potential biological activity, a high anticancer activity (de la Cueva-Alique et al., 2019).

5. Synthesis and crystallization

To a solution of NiII chloride ethylene glycol dimethyl ether (0.15 g, 1.48 mmol) in MeOH (5 mL) was added (1S,4R)-1-picolylamino-p-menth-8-en-2-one-oxime (0.101 g, 0.36 mmol) dissolved in MeOH (3 mL). The solution turned green. The mixture was stirred overnight at room temperature during which time the mixture changed color to blue–green. The solvent was then evaporated to produce a crude solid that was washed with diethyl ether before crystallization. Single crystals were grown by slow diffusion at room temperature of diethyl ether into a dichloromethane solution. Elemental analysis calculated for C32H46Cl4N6Ni2O2: C, 46.33; H, 5.54; N, 9.65. Found: C, 46.35; H, 5.672; N, 9.77.

6. Refinement

Crystal data, data collection and structure refinement details are summarized in Table 2. N- and O-bound atoms were refined with the restraint \(U_{iso}(H) = 1.2U_{eq}(N)\) or \(1.5U_{eq}(O)\). H atoms were positioned geometrically (C—H = 0.95–1.00 Å) and refined as riding with \(U_{iso} = 1.2U_{eq}(C)\) or \(1.5U_{eq}(C\text{-methyl})\).

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We would like to thank Céline Delabre for the elemental analysis.

Table 2

| Crystal data | [NiCl2(C16H23ClN3O)2] |
|--------------|----------------------|
| Chemical formula | NiCl2(C16H23ClN3O)2 |
| Mass (Mr) | 805.97 |
| Crystal system, space group | Monoclinic, P21 |
| Temperature (K) | 100 |
| No. of measured, independent and observed \(|F| > 2\sigma(|F|)\) reflections | 42747, 10769, 9436 |
| \(R_{w}||F||F||F\rangle\), \(wR_{2}\), | 0.043, 0.109, 1.05 |
| No. of parameters | 431 |
| No. of restraints | 13 |
| H-atom treatment | H atoms treated by a mixture of independent and constrained refinement |
| \(\Delta \rho_{\text{max}}, \Delta \rho_{\text{min}} \text{ (e Å}^{-3})\) | 1.50, −1.19 |
| Absolute structure parameter | −0.009 (4) |

Computer programs: APEX2 and SAINT (Bruker, 2019), SHELXT (Sheldrick, 2015a), SHELXL (Sheldrick, 2015b) and OLEX2 (Dolomanov et al., 2009).

Figure 3

Packing diagram.
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Synthesis and crystal structure of a new chiral α-aminooxime nickel(II) complex

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Computing details

Data collection: APEX2 (Bruker, 2019); cell refinement: SAINT (Bruker, 2019); data reduction: SAINT (Bruker, 2019); program(s) used to solve structure: ShelXT (Sheldrick, 2015a); program(s) used to refine structure: SHELXL (Sheldrick, 2015b); molecular graphics: OLEX2 (Dolomanov et al., 2009); software used to prepare material for publication: OLEX2 (Dolomanov et al., 2009).

Dichloridobis((2S,5R)-2-methyl-5-(prop-1-en-2-yl)-2-[(pyridin-2-yl)methylamino]cyclohexan-1-one oxime)dinickel(II)

Crystal data

\[\text{[Ni}_2\text{Cl}_4(\text{C}_{19}\text{H}_{24}\text{ClN}_5\text{O})_2]}\]
\[M_r = 805.97\]
Monoclinic, \(P2_1\)
\(a = 13.3729\) (9) \(\text{Å}\)
\(b = 8.9363\) (7) \(\text{Å}\)
\(c = 16.4248\) (16) \(\text{Å}\)
\(\beta = 114.014\) (2)°
\(V = 1792.9\) (3) \(\text{Å}^3\)
\(Z = 2\)

Data collection

Bruker APEXII CCD diffractometer
Radiation source: microfocus sealed X-ray tube
\(\varphi\) and \(\omega\) scans
Absorption correction: multi-scan (SADABS; Krause et al., 2015)
\(T_{\text{min}} = 0.669, T_{\text{max}} = 0.746\)
42747 measured reflections
10769 independent reflections
9436 reflections with \(I > 2\sigma(I)\)

Refinement

Refinement on \(F^2\)
Least-squares matrix: full
\(R[F^2 > 2\sigma(F^2)] = 0.043\)
\(wR(F^2) = 0.109\)
\(S = 1.05\)
10769 reflections
431 parameters
13 restraints
Primary atom site location: dual
Secondary atom site location: difference Fourier map
Hydrogen site location: mixed
\(H\) atoms treated by a mixture of independent and constrained refinement
\(w = 1/[\sigma^2(F_c^2) + (0.0581P)^2 + 0.9636P]\)
where \(P = (F_c^2 + 2F_s^2)/3\)
\((\Delta/\sigma)_{\text{max}} = 0.001\)
\(\Delta\rho_{\text{max}} = 1.50\ \text{e} \ \text{Å}^{-3}\)
Δρ_{min} = −1.18 e Å⁻³

Absolute structure: Flack x determined using
3850 quotients [(I⁺)−(I⁻)]/[(I⁺)+(I⁻)] (Parsons et al., 2013)
Absolute structure parameter: −0.009 (4)

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     | U_{iso}/*U_{eq} |
|---|-------|-------|-------|-----------------|
| Ni1 | 0.66455 (4) | 0.50226 (6) | 0.35327 (3) | 0.01526 (11) |
| Ni2 | 0.41917 (4) | 0.48960 (6) | 0.15985 (3) | 0.01813 (12) |
| Cl2 | 0.61155 (8) | 0.45928 (14) | 0.19208 (7) | 0.0259 (2) |
| Cl3 | 0.48342 (7) | 0.60270 (12) | 0.30567 (7) | 0.0212 (2) |
| Cl1 | 0.74487 (8) | 0.75170 (12) | 0.37010 (7) | 0.0249 (2) |
| Cl4 | 0.38501 (9) | 0.25191 (13) | 0.21270 (9) | 0.0336 (3) |
| O1  | 0.7388 (3) | 0.6301 (4) | 0.3539 (2) | 0.0244 (7) |
| H1  | 0.740 (5) | 0.705 (7) | 0.507 (4) | 0.037* |
| N1  | 0.8111 (3) | 0.3926 (4) | 0.3786 (2) | 0.0182 (7) |
| N3  | 0.6939 (2) | 0.5043 (5) | 0.4853 (2) | 0.0183 (6) |
| N2  | 0.6216 (3) | 0.2811 (4) | 0.3745 (2) | 0.0183 (7) |
| H2  | 0.563 (4) | 0.289 (6) | 0.339 (3) | 0.022* |
| O2  | 0.1908 (3) | 0.4533 (5) | 0.1497 (2) | 0.0339 (9) |
| H2A | 0.226 (6) | 0.389 (9) | 0.176 (5) | 0.051* |
| C8  | 0.6846 (3) | 0.3877 (5) | 0.5258 (3) | 0.0203 (8) |
| C5  | 0.8008 (3) | 0.2431 (5) | 0.3699 (3) | 0.0202 (8) |
| N6  | 0.2619 (3) | 0.5471 (4) | 0.1318 (2) | 0.0212 (8) |
| N5  | 0.4081 (3) | 0.6943 (5) | 0.0936 (3) | 0.0394 (12) |
| H5  | 0.468 (5) | 0.736 (8) | 0.140 (4) | 0.047* |
| C12 | 0.7001 (3) | 0.1123 (6) | 0.5107 (3) | 0.0250 (9) |
| H12A| 0.660298 | 0.022653 | 0.478112 | 0.030* |
| H12B| 0.770464 | 0.117488 | 0.504159 | 0.030* |
| C14 | 0.9033 (3) | 0.2356 (6) | 0.6758 (3) | 0.0257 (9) |
| C3  | 0.9925 (4) | 0.2136 (6) | 0.4131 (3) | 0.0301 (11) |
| H3  | 1.054899 | 0.151943 | 0.425362 | 0.036* |
| C9  | 0.7192 (4) | 0.3765 (6) | 0.6253 (3) | 0.0279 (10) |
| H9A | 0.653362 | 0.378843 | 0.638523 | 0.034* |
| H9B | 0.764996 | 0.464264 | 0.654444 | 0.034* |
| C1  | 0.9109 (3) | 0.4531 (5) | 0.4044 (3) | 0.0220 (9) |
| H1A | 0.918464 | 0.558677 | 0.410777 | 0.026* |
| C30 | 0.0369 (4) | 0.6416 (7) | −0.1114 (4) | 0.0357 (12) |
| N4  | 0.3669 (3) | 0.4143 (6) | 0.0282 (3) | 0.0361 (11) |
| C2  | 1.0041 (3) | 0.3660 (6) | 0.4222 (3) | 0.0277 (10) |
| H2B | 1.074096 | 0.411370 | 0.440161 | 0.033* |
| C25 | 0.1029 (4) | 0.7072 (7) | 0.0546 (4) | 0.0384 (13) |
|    | X    | Y    | Z    |    |
|----|------|------|------|----|
| H25A | 0.095090 | 0.786671 | 0.093629 | 0.046* |
| H25B | 0.059972 | 0.619493 | 0.058673 | 0.046* |
| C7  | 0.6314 (3) | 0.2540 (5) | 0.4678 (3) | 0.0206 (8) |
| C4  | 0.8897 (4) | 0.1499 (6) | 0.3860 (3) | 0.0278 (10) |
| H4  | 0.880183 | 0.044699 | 0.378533 | 0.033* |
| C24 | 0.2208 (4) | 0.6641 (5) | 0.0869 (3) | 0.0258 (10) |
| C6  | 0.6855 (3) | 0.1848 (5) | 0.3402 (3) | 0.0218 (8) |
| H6A | 0.649955 | 0.182503 | 0.274260 | 0.026* |
| H6B | 0.687313 | 0.081400 | 0.362311 | 0.026* |
| C10 | 0.7844 (4) | 0.2319 (6) | 0.6644 (3) | 0.0281 (10) |
| H10 | 0.785669 | 0.219265 | 0.725250 | 0.034* |
| C23 | 0.2999 (4) | 0.7724 (5) | 0.0732 (4) | 0.0365 (12) |
| C15 | 0.9419 (4) | 0.3220 (6) | 0.6296 (3) | 0.0301 (10) |
| H15A| 1.016603 | 0.314817 | 0.638711 | 0.036* |
| H15B| 0.894916 | 0.391186 | 0.587469 | 0.036* |
| C31 | 0.0663 (3) | 0.5006 (7) | −0.0931 (3) | 0.0322 (10) |
| H31A| 0.053020 | 0.430999 | −0.140077 | 0.039* |
| C31B| 0.100801 | 0.468485 | −0.032826 | 0.039* |
| C13 | 0.5139 (3) | 0.2394 (7) | 0.4628 (3) | 0.0314 (11) |
| H13A| 0.471161 | 0.327732 | 0.433110 | 0.047* |
| H13B| 0.516636 | 0.231714 | 0.523147 | 0.047* |
| H13C| 0.479378 | 0.149469 | 0.428806 | 0.047* |
| C11 | 0.7236 (4) | 0.0951 (6) | 0.6094 (3) | 0.0306 (11) |
| H11A| 0.768517 | 0.004325 | 0.633040 | 0.037* |
| H11B| 0.653697 | 0.081821 | 0.615749 | 0.037* |
| C16 | 0.9776 (4) | 0.1259 (6) | 0.7427 (3) | 0.0313 (11) |
| H16A| 0.980940 | 0.149758 | 0.802043 | 0.047* |
| H16B| 1.051172 | 0.132186 | 0.743621 | 0.047* |
| H16C| 0.948965 | 0.024270 | 0.726130 | 0.047* |
| C18 | 0.2741 (6) | 0.2441 (10) | −0.0937 (4) | 0.0582 (18) |
| H18 | 0.234602 | 0.153747 | −0.115022 | 0.070* |
| C17 | 0.3151 (5) | 0.2832 (8) | −0.0055 (4) | 0.0493 (16) |
| H17 | 0.306660 | 0.213712 | 0.035033 | 0.059* |
| C26 | 0.0565 (4) | 0.7638 (7) | −0.0421 (4) | 0.0423 (14) |
| H26 | −0.016160 | 0.810447 | −0.054167 | 0.051* |
| C21 | 0.3804 (4) | 0.5139 (10) | −0.0254 (4) | 0.0504 (17) |
| C28 | 0.2479 (5) | 0.8323 (7) | −0.0237 (4) | 0.0486 (16) |
| H28A| 0.293285 | 0.915184 | −0.030027 | 0.058* |
| H28B| 0.247256 | 0.751483 | −0.065083 | 0.058* |
| C32 | −0.0176 (7) | 0.6926 (9) | −0.2067 (4) | 0.068 (2) |
| H32A| 0.032890 | 0.756035 | −0.220891 | 0.101* |
| H32B| −0.083660 | 0.749858 | −0.215141 | 0.101* |
| H32C| −0.037751 | 0.605256 | −0.246168 | 0.101* |
| C22 | 0.4373 (5) | 0.6511 (10) | 0.0162 (4) | 0.064 (2) |
| H22A| 0.416191 | 0.732765 | −0.028357 | 0.077* |
| H22B| 0.517365 | 0.635850 | 0.038173 | 0.077* |
| C27 | 0.1312 (6) | 0.8883 (7) | −0.0493 (4) | 0.0552 (18) |
| H27A| 0.101814 | 0.926492 | −0.111198 | 0.066* |
### Atomic displacement parameters (Å²)

|         | \( U_{11} \)        | \( U_{22} \)        | \( U_{33} \)        | \( U_{12} \)        | \( U_{13} \)        | \( U_{23} \)        |
|---------|---------------------|---------------------|---------------------|---------------------|---------------------|---------------------|
| Ni1     | 0.0139 (19)         | 0.0171 (2)          | 0.0136 (2)          | 0.0000 (2)          | 0.00431 (17)        | 0.0017 (2)          |
| Ni2     | 0.0158 (2)          | 0.0210 (3)          | 0.0153 (2)          | −0.0019 (2)         | 0.00406 (17)        | −0.0011 (2)         |
| Cl2     | 0.0180 (4)          | 0.0431 (7)          | 0.0155 (4)          | 0.0164 (4)          | 0.0057 (3)          | 0.0037 (4)          |
| Cl3     | 0.0178 (4)          | 0.0225 (5)          | 0.0194 (5)          | 0.0017 (4)          | 0.0034 (4)          | −0.0031 (4)         |
| Cl1     | 0.0240 (5)          | 0.0198 (5)          | 0.0244 (5)          | −0.0048 (4)         | 0.0031 (4)          | 0.0052 (4)          |
| Cl4     | 0.0301 (5)          | 0.0180 (5)          | 0.0367 (7)          | −0.0042 (4)         | −0.0027 (5)         | 0.0021 (5)          |
| O1      | 0.0275 (15)         | 0.0233 (17)         | 0.0205 (16)         | 0.0014 (13)         | 0.0077 (13)         | −0.0051 (13)        |
| N1      | 0.0159 (14)         | 0.0249 (19)         | 0.0133 (16)         | 0.0017 (13)         | 0.0054 (13)         | 0.0017 (14)         |
| N3      | 0.0163 (13)         | 0.0206 (17)         | 0.0170 (15)         | 0.0001 (15)         | 0.0056 (12)         | 0.0000 (16)         |
| N2      | 0.0122 (13)         | 0.0204 (19)         | 0.0200 (18)         | 0.0019 (13)         | 0.0044 (13)         | 0.0019 (14)         |
| O2      | 0.0186 (14)         | 0.052 (3)           | 0.0312 (19)         | −0.0003 (14)        | 0.0099 (13)         | 0.0128 (17)         |
| C8      | 0.0184 (18)         | 0.026 (2)           | 0.020 (2)           | 0.0079 (16)         | 0.0113 (16)         | 0.0058 (17)         |
| C5      | 0.0221 (18)         | 0.025 (2)           | 0.0146 (19)         | 0.0034 (17)         | 0.0086 (15)         | 0.0006 (17)         |
| N6      | 0.0182 (15)         | 0.028 (2)           | 0.0163 (17)         | 0.0011 (13)         | 0.0055 (14)         | −0.0007 (14)        |
| N5      | 0.029 (2)           | 0.040 (3)           | 0.033 (2)           | −0.0178 (19)        | −0.0040 (18)        | 0.020 (2)           |
| C12     | 0.0200 (18)         | 0.025 (2)           | 0.027 (2)           | −0.0009 (17)        | 0.0064 (17)         | 0.0088 (19)         |
| C14     | 0.0249 (19)         | 0.033 (3)           | 0.016 (2)           | 0.0063 (18)         | 0.0045 (16)         | 0.0044 (19)         |
| C3      | 0.025 (2)           | 0.036 (3)           | 0.029 (3)           | 0.0125 (19)         | 0.0111 (19)         | 0.004 (2)           |
| C9      | 0.032 (2)           | 0.037 (3)           | 0.018 (2)           | 0.011 (2)           | 0.0145 (18)         | 0.006 (2)           |
| C1      | 0.0194 (17)         | 0.028 (2)           | 0.019 (2)           | 0.0006 (16)         | 0.0077 (15)         | 0.0024 (17)         |
| C30     | 0.035 (2)           | 0.041 (3)           | 0.033 (3)           | 0.006 (2)           | 0.015 (2)           | 0.000 (2)           |
| N4      | 0.0223 (18)         | 0.061 (3)           | 0.021 (2)           | 0.0157 (19)         | 0.0044 (16)         | −0.005 (2)          |
| C2      | 0.0149 (17)         | 0.041 (3)           | 0.028 (2)           | 0.0019 (18)         | 0.0097 (17)         | 0.007 (2)           |
| C25     | 0.033 (2)           | 0.047 (3)           | 0.031 (3)           | 0.022 (2)           | 0.009 (2)           | −0.001 (2)          |
| C7      | 0.0174 (16)         | 0.025 (2)           | 0.022 (2)           | 0.0014 (16)         | 0.0105 (15)         | 0.0077 (18)         |
| C4      | 0.025 (2)           | 0.032 (3)           | 0.027 (2)           | 0.0106 (18)         | 0.0114 (18)         | 0.002 (2)           |
| C24     | 0.029 (2)           | 0.025 (2)           | 0.017 (2)           | 0.0080 (18)         | 0.0041 (18)         | −0.0052 (18)        |
| C6      | 0.0243 (19)         | 0.017 (2)           | 0.024 (2)           | 0.0046 (16)         | 0.0094 (17)         | −0.0026 (17)        |
| C10     | 0.030 (2)           | 0.034 (3)           | 0.024 (2)           | 0.007 (2)           | 0.0142 (18)         | 0.012 (2)           |
| C23     | 0.048 (3)           | 0.015 (2)           | 0.032 (3)           | −0.003 (2)          | 0.001 (2)           | 0.006 (2)           |
| C15     | 0.0204 (19)         | 0.036 (3)           | 0.028 (2)           | 0.0014 (18)         | 0.0032 (18)         | 0.008 (2)           |
| C31     | 0.0267 (19)         | 0.033 (2)           | 0.032 (2)           | −0.005 (2)          | 0.0058 (18)         | 0.001 (3)           |
| C13     | 0.0198 (19)         | 0.040 (3)           | 0.034 (3)           | −0.001 (2)          | 0.0108 (18)         | 0.010 (2)           |
| C11     | 0.026 (2)           | 0.033 (3)           | 0.035 (3)           | 0.0008 (19)         | 0.015 (2)           | 0.018 (2)           |
| C16     | 0.033 (2)           | 0.037 (3)           | 0.024 (2)           | 0.009 (2)           | 0.0120 (19)         | 0.011 (2)           |
### Geometric parameters (Å, °)

| Bond/Distance     | Value (Å)  | Bond/Distance     | Value (Å)  | Bond/Distance     | Value (Å)  |
|-------------------|------------|-------------------|------------|-------------------|------------|
| Ni1—C12           | 2.4762 (11)| C2—H2B            | 0.950      | Ni1—C13           | 2.3964 (10) |
| Ni1—Cl2           | 2.4408 (12)| C25—H25A          | 0.9900     | Ni1—Cl3           | 2.4128 (12) |
| Ni1—Cl3           | 2.077 (3)  | C25—H25B          | 0.9900     | Ni1—Cl4           | 2.4077 (14) |
| Ni1—Cl1           | 2.041 (3)  | C25—C24           | 1.495 (6)  | Ni2—N6            | 2.027 (3)   |
| Ni1—N1            | 2.126 (4)  | C25—C26           | 1.536 (8)  | Ni2—N5            | 2.103 (4)   |
| Ni1—N3            | 2.126 (4)  | C7—C13            | 1.545 (5)  | Ni2—N4            | 2.095 (4)   |
| Ni1—N2            | 2.4216 (10)| C4—H4             | 0.9500     | O1—H1             | 0.85 (7)    |
| Ni2—Cl2           | 2.4216 (10)| C24—C23           | 1.516 (7)  | O1—N3             | 1.403 (5)   |
| Ni2—Cl3           | 2.4128 (12)| C24—C23           | 0.9900     | N1—C5             | 1.345 (6)   |
| Ni2—Cl4           | 2.4077 (14)| C6—H6A            | 0.9900     | N1—C1             | 1.338 (5)   |
| Ni2—Cl1           | 2.027 (3)  | C6—H6B            | 0.9900     | N3—C8             | 1.269 (6)   |
| Ni2—N6            | 2.103 (4)  | C10—H10           | 1.0000     | N2—H2             | 0.77 (5)    |
| Ni2—N5            | 2.095 (4)  | C10—C11           | 1.540 (8)  | N2—C7             | 1.503 (5)   |
| Ni2—N4            | 2.103 (4)  | C13—H13A          | 0.9800     | N2—C6             | 1.477 (5)   |
| O1—H1             | 0.85 (7)   | C13—H13B          | 0.9800     | O2—H2A            | 0.76 (8)    |
| N1—C5             | 1.345 (6)  | C15—H15A          | 0.9500     | N6—N2             | 1.385 (5)   |
| N1—C1             | 1.338 (5)  | C15—H15B          | 0.9500     | C8—C9             | 1.509 (6)   |
| N3—C8             | 1.269 (6)  | C31—H31A          | 0.9500     | C8—C7             | 1.513 (7)   |
| N2—H2             | 0.77 (5)   | C31—H31B          | 0.9500     | C5—C4             | 1.385 (6)   |
| N2—C7             | 1.503 (5)  | C13—H13A          | 0.9800     | C5—C6             | 1.508 (6)   |
| N2—C6             | 1.477 (5)  | C13—H13B          | 0.9800     | N6—C24            | 1.269 (6)   |
| O2—H2A            | 0.76 (8)   | C13—H13C          | 0.9800     | N5—H5             | 0.93 (7)    |
| O2—N6             | 1.385 (5)  | C11—H11A          | 0.9900     | N5—C23            | 1.517 (7)   |
| C8—C9             | 1.509 (6)  | C11—H11B          | 0.9900     | N5—C22            | 1.524 (9)   |
| C8—C7             | 1.513 (7)  | C16—H16A          | 0.9800     | N5—H12A           | 0.9900     |
| C5—C4             | 1.385 (6)  | C16—H16B          | 0.9800     | C12—H12A          | 0.9900     |
| C5—C6             | 1.508 (6)  | C16—H16C          | 0.9800     | C12—H12B          | 0.9900     |
| N6—C24            | 1.269 (6)  | C18—H18           | 0.9500     | C12—C7            | 1.555 (6)   |
| N5—H5             | 0.93 (7)   | C18—C17           | 1.370 (8)  | C12—C11           | 1.528 (7)   |
| N5—C23            | 1.517 (7)  | C18—C19           | 1.318 (12) |                  |            |
| N5—C22            | 1.524 (9)  | C17—H17           | 0.9500     |                  |            |
| C12—H12A          | 0.9900     | C26—H26           | 1.0000     |                  |            |
| C12—H12B          | 0.9900     | C26—C27           | 1.532 (10) |                  |            |
| C12—C7            | 1.555 (6)  | C21—C22           | 1.457 (12) |                  |            |
| C12—C11           | 1.528 (7)  | C21—C20           | 1.465 (10) |                  |            |
| Bond                  | Distance (Å) | Bond                  | Distance (Å) |
|----------------------|--------------|----------------------|--------------|
| C14—C10              | 1.523 (6)    | C28—H28A             | 0.9900       |
| C14—C15              | 1.326 (7)    | C28—H28B             | 0.9900       |
| C14—C16              | 1.506 (7)    | C28—C27              | 1.525 (9)    |
| C3—H3                | 0.9500       | C32—H32A             | 0.9800       |
| C3—C2                | 1.373 (8)    | C32—H32B             | 0.9800       |
| C3—C4                | 1.384 (7)    | C32—H32C             | 0.9800       |
| C9—H9A               | 0.9900       | C22—H22A             | 0.9900       |
| C9—H9B               | 0.9900       | C22—H22B             | 0.9900       |
| C9—C10               | 1.545 (7)    | C27—H27A             | 0.9900       |
| C1—H1A               | 0.9500       | C27—H27B             | 0.9900       |
| C1—C2                | 1.396 (6)    | C29—H29A             | 0.9800       |
| C30—C31              | 1.318 (8)    | C29—H29B             | 0.9800       |
| C30—C26              | 1.521 (8)    | C29—H29C             | 0.9800       |
| C30—C32              | 1.503 (8)    | C19—H19              | 0.9500       |
| N4—C17               | 1.359 (8)    | C19—C20              | 1.360 (13)   |
| N4—C21               | 1.314 (8)    | C20—H20              | 0.9500       |
| C13—Ni1—Cl2          | 84.13 (4)    | C8—C7—C13            | 108.0 (4)    |
| C13—Ni1—Cl1          | 91.99 (4)    | C13—C7—C12           | 110.9 (4)    |
| C11—Ni1—Cl2          | 100.61 (4)   | C5—C4—H4             | 120.7        |
| N1—Ni1—Cl2           | 88.69 (10)   | C3—C4—C5             | 118.5 (5)    |
| N1—Ni1—Cl3           | 171.31 (10)  | C3—C4—H4             | 120.7        |
| N1—Ni1—Cl1           | 94.14 (11)   | N6—C24—C25           | 124.3 (5)    |
| N1—Ni1—N2            | 79.91 (13)   | N6—C24—C23           | 116.7 (4)    |
| N3—Ni1—Cl2           | 170.10 (12)  | C25—C24—C23          | 118.8 (4)    |
| N3—Ni1—Cl3           | 94.30 (9)    | N2—C6—C5             | 110.5 (4)    |
| N3—Ni1—Cl1           | 89.21 (12)   | N2—C6—H6A            | 109.6        |
| N3—Ni1—N1            | 91.94 (13)   | N2—C6—H6B            | 109.6        |
| N3—Ni1—N2            | 77.38 (15)   | C5—C6—H6A            | 109.6        |
| N2—Ni1—Cl2           | 93.02 (10)   | C5—C6—H6B            | 109.6        |
| N2—Ni1—Cl3           | 95.56 (9)    | H6A—C6—H6B           | 108.1        |
| N2—Ni1—Cl1           | 165.04 (11)  | C14—C10—C9           | 114.7 (4)    |
| C13—Ni2—Cl2          | 84.97 (4)    | C14—C10—H10          | 106.7        |
| C14—Ni2—Cl2          | 99.38 (4)    | C14—C10—C11          | 111.4 (4)    |
| C14—Ni2—Cl3          | 93.14 (5)    | C9—C10—H10           | 106.7        |
| N6—Ni2—Cl2           | 171.72 (12)  | C11—C10—C9           | 110.3 (4)    |
| N6—Ni2—Cl3           | 92.13 (11)   | C11—C10—H10          | 106.7        |
| N6—Ni2—Cl14          | 88.51 (11)   | N5—C23—C28           | 112.0 (5)    |
| N6—Ni2—N5            | 79.29 (16)   | C24—C23—N5           | 109.5 (4)    |
| N6—Ni2—N4            | 88.11 (15)   | C24—C23—C28          | 108.9 (4)    |
| N5—Ni2—Cl2           | 93.15 (13)   | C29—C23—N5           | 104.7 (5)    |
| N5—Ni2—Cl3           | 94.06 (15)   | C29—C23—C24          | 109.9 (5)    |
| N5—Ni2—Cl4           | 166.04 (12)  | C29—C23—C28          | 111.8 (5)    |
| N4—Ni2—Cl2           | 93.92 (11)   | C14—C15—H15A         | 120.0        |
| N4—Ni2—Cl3           | 173.62 (15)  | C14—C15—H15B         | 120.0        |
| N4—Ni2—Cl4           | 93.24 (16)   | H15A—C15—H15B        | 120.0        |
| N4—Ni2—N5            | 79.7 (2)     | C30—C31—H31A         | 120.0        |
| Ni2—Cl2—Ni1          | 91.88 (4)    | C30—C31—H31B         | 120.0        |
Ni1—Cl3—Ni2  94.09 (4)  H31A—C31—H31B  120.0
N3—O1—H1   111 (4)  C7—C13—H13A  109.5
C5—N1—Ni1   113.6 (3) C7—C13—H13B  109.5
C1—N1—Ni1   127.6 (3) C7—C13—H13C  109.5
C1—N1—C5    118.8 (4) H13A—C13—H13B  109.5
O1—N3—Ni1   121.4 (3) H13A—C13—H13C  109.5
C8—N3—Ni1   122.2 (3) H13B—C13—H13C  109.5
C8—N3—O1    115.9 (3) C12—C11—C10  112.0 (4)
Ni1—N2—H2   93 (4)  C12—C11—H11A  109.2
C7—N2—Ni1   113.6 (3) C12—C11—H11B  109.2
C7—N2—H2    116 (4)  C10—C11—H11A  109.2
C6—N2—Ni1   104.0 (2) C10—C11—H11B  109.2
C6—N2—H2    109 (4)  H11A—C11—H11B  107.9
C6—N2—C7    118.1 (3) C14—C16—H16A  109.5
N6—O2—H2A   105 (5)  C14—C16—H16B  109.5
N3—C8—C9    124.7 (4) C14—C16—H16C  109.5
N3—C8—C7    116.1 (4) H16A—C16—H16B  109.5
C9—C8—C7    119.2 (4) H16A—C16—H16C  109.5
N1—C5—C6    122.3 (4) H16B—C16—H16C  109.5
N1—C5—C4    115.1 (4) C17—C18—H18  121.9
C4—C5—C6    122.6 (4) C19—C18—H18  121.9
O2—N6—Ni2   122.4 (3) C19—C18—C17  116.1 (8)
C24—N6—Ni2  120.3 (3) N4—C17—C18  124.7 (7)
C24—N6—O2   116.7 (4) N4—C17—H17  117.7
Ni2—N5—H5   94 (4)  C18—C17—H17  117.7
C23—N5—Ni2  112.0 (3) C30—C26—C25  114.3 (5)
C23—N5—H5   115 (4)  C30—C26—H26  107.1
C23—N5—C22  118.6 (4) C30—C26—C27  112.5 (5)
C22—N5—Ni2  102.9 (4) C25—C26—H26  107.1
C22—N5—H5   110 (4)  C27—C26—C25  108.5 (5)
H12A—C12—H12B  107.8  C27—C26—H26  107.1
C7—C12—H12A 109.0  N4—C21—C22  116.4 (5)
C7—C12—H12B 109.0  N4—C21—C20  117.2 (8)
C11—C12—H12A 109.0  C22—C21—C20  126.3 (7)
C11—C12—H12B 109.0  C23—C28—H28A  109.2
C11—C12—C7   113.0 (4) C23—C28—H28B  109.2
C15—C14—C10  124.9 (4) H28A—C28—H28B  107.9
C15—C14—C16  120.2 (4) C27—C28—C23  112.2 (6)
C16—C14—C10  114.9 (4) C27—C28—H28A  109.2
C2—C3—H3    120.1  C27—C28—H28B  109.2
C2—C3—C4    119.7 (4) C30—C32—H32A  109.5
C4—C3—H3    120.1  C30—C32—H32B  109.5
C8—C9—H9A   109.2  C30—C32—H32C  109.5
C8—C9—H9B   109.2  H32A—C32—H32B  109.5
C8—C9—C10   112.2 (4) H32A—C32—H32C  109.5
H9A—C9—H9B  107.9  H32B—C32—H32C  109.5
C10—C9—H9A  109.2  N5—C22—H22A  109.7
C10—C9—H9B  109.2  N5—C22—H22B  109.7
| Bond                  | Angle (°) | Bond                  | Angle (°) |
|----------------------|-----------|----------------------|-----------|
| N1—C1—H1A           | 119.0     | C21—C22—N5           | 110.0     |
| N1—C1—C2            | 122.1     | C21—C22—H22A         | 109.7     |
| C2—C1—H1A           | 119.0     | C21—C22—H22B         | 109.7     |
| C31—C30—C26         | 124.8     | H22A—C22—H22B        | 108.2     |
| C31—C30—C32         | 120.1     | C26—C27—H27A         | 109.3     |
| C32—C30—C26         | 115.1     | C26—C27—H27B         | 109.3     |
| C17—N4—Ni2          | 126.7     | C28—C27—C26          | 111.5     |
| C21—N4—Ni2          | 113.2     | C28—C27—H27A         | 109.3     |
| C21—N4—C17          | 119.9     | C28—C27—H27B         | 109.3     |
| C3—C2—C1            | 118.6     | H27A—C27—H27B        | 108.0     |
| C3—C2—H2B           | 120.7     | C23—C29—H29A         | 109.5     |
| C1—C2—H2B           | 120.7     | C23—C29—H29B         | 109.5     |
| H25A—C25—H25B       | 107.9     | C23—C29—H29C         | 109.5     |
| C24—C25—H25A        | 109.2     | H29A—C29—H29B        | 109.5     |
| C24—C25—H25B        | 109.2     | H29A—C29—H29C        | 109.5     |
| C26—C25—H25A        | 112.1     | H29B—C29—H29C        | 109.5     |
| C26—C25—H25B        | 109.2     | H27A—C27—H27B        | 108.0     |
| N2—C7—C8            | 110.8     | C20—C19—H19          | 118.5     |
| N2—C7—C12           | 112.5     | C21—C20—H20          | 120.5     |
| N2—C7—C13           | 107.1     | C19—C20—C21          | 119.0     |
| C8—C7—C12           | 108.5     | C19—C20—H20          | 120.5     |

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\begin{align*}
\text{Ni1—N1—C5—C4} & \quad -178.5 \quad \text{N4—C21—C20—C19} \quad 2.4 \\
\text{Ni1—N1—C5—C6} & \quad 2.4 \quad \text{C2—C3—C4—C5} \quad -0.9 \\
\text{Ni1—N1—C1—C2} & \quad 177.8 \quad \text{C25—C24—C23—N5} \quad -168.7 \\
\text{Ni1—N3—C8—C9} & \quad 171.5 \quad \text{C25—C24—C23—C28} \quad -45.9 \\
\text{Ni1—N3—C8—C7} & \quad -10.6 \quad \text{C25—C24—C23—C29} \quad 76.9 \\
\text{Ni1—N2—C7—C8} & \quad -7.7 \quad \text{C25—C26—C27—C28} \quad 59.6 \\
\text{Ni1—N2—C7—C12} & \quad -128.6 \quad \text{C7—N2—C6—C5} \quad -83.5 \\
\text{Ni1—N2—C7—C13} & \quad 109.3 \quad \text{C7—C8—C9—C10} \quad 47.9 \\
\text{Ni1—N2—C6—C5} & \quad 43.4 \quad \text{C7—C12—C11—C10} \quad -57.9 \\
\text{Ni2—N6—C24—C25} & \quad 173.2 \quad \text{C4—C5—C6—N2} \quad 148.6 \\
\text{Ni2—N6—C24—C23} & \quad -12.5 \quad \text{C4—C3—C2—C1} \quad 0.6 \\
\text{Ni2—N5—C23—C24} & \quad -13.2 \quad \text{C24—C25—C26—C30} \quad 73.6 \\
\text{Ni2—N5—C23—C28} & \quad -134.2 \quad \text{C24—C25—C26—C27} \quad -52.8 \\
\text{Ni2—N5—C23—C29} & \quad 104.5 \quad \text{C24—C23—C28—C27} \quad 49.5 \\
\text{Ni2—N5—C22—C21} & \quad 44.6 \quad \text{C6—N2—C7—C8} \quad 114.5 \\
\text{Ni2—N4—C17—C18} & \quad 174.2 \quad \text{C6—N2—C7—C12} \quad -6.5 \\
\text{Ni2—N4—C21—C22} & \quad 5.4 \quad \text{C6—N2—C7—C13} \quad -128.6 \\
\text{Ni2—N4—C21—C20} & \quad -178.0 \quad \text{C6—C5—C4—C3} \quad 180.0 \\
\text{O1—N3—C8—C9} & \quad -0.7 \quad \text{C23—N5—C22—C21} \quad -79.6 \\
\text{O1—N3—C8—C7} & \quad 177.3 \quad \text{C23—C28—C27—C26} \quad -59.7 \\
\text{N1—C5—C4—C3} & \quad 1.0 \quad \text{C15—C14—C10—C9} \quad -24.5 \\
\text{N1—C5—C6—N2} & \quad -32.3 \quad \text{C15—C14—C10—C11} \quad 101.6 \\
\text{N1—C1—C2—C3} & \quad -0.2 \quad \text{C31—C30—C26—C25} \quad -6.7 \\
\text{N3—C8—C9—C10} & \quad -134.2 \quad \text{C31—C30—C26—C27} \quad 117.5 \\
\text{N3—C8—C7—N2} & \quad 11.5 \quad \text{C11—C12—C7—N2} \quad 172.3 \\
\end{align*}

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[sup-8]
Hydrogen-bond geometry (Å, º)

| D—H···A  | D—H  | H···A  | D···A  | D—H···A |
|----------|-------|--------|--------|----------|
| O1—H1···Cl1 | 0.85 (7) | 2.32 (6) | 3.009 (4) | 139 (6) |
| N2—H2···Cl4  | 0.77 (5) | 2.46 (5) | 3.209 (4) | 166 (5) |
| O2—H2A···Cl4 | 0.76 (8) | 2.31 (7) | 2.978 (4) | 147 (7) |
| C3—H3···O1i  | 0.95 | 2.58 | 3.432 (5) | 149 |
| C1—H1A···Cl1 | 0.95 | 2.75 | 3.369 (5) | 124 |
| C6—H6A···Cl2 | 0.99 | 2.76 | 3.309 (5) | 115 |
| C11—H11B···Cl3ii | 0.99 | 2.64 | 3.573 (5) | 156 |
| C17—H17···Cl4 | 0.95 | 2.69 | 3.327 (6) | 125 |
| C26—H26···O2iii | 1.00 | 2.56 | 3.489 (6) | 154 |
| C22—H22A···Cl2 | 0.99 | 2.81 | 3.352 (6) | 115 |
| C19—H19···Cl1iv | 0.95 | 2.64 | 3.570 (7) | 167 |

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