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catena-Poly[[nickel(II)- μ_3 -1,1-dicyanoethene-2,2-dithiolato- κ^4 S,S':N:N'-bis[(15-crown-5)magnesium(II)]- μ_3 -1,1-dicyanoethene-2,2-dithiolato- κ^4 N:N':S,S'] dichloride]

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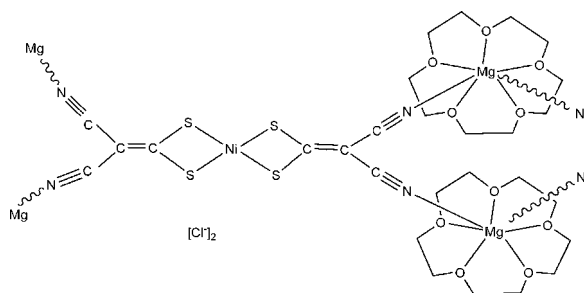
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Key indicators: single-crystal X-ray study; $T = 298$ K; mean $\sigma(\text{C}-\text{C}) = 0.026$ Å;
R factor = 0.064; wR factor = 0.192; data-to-parameter ratio = 8.2.

The reaction of MgCl_2 , NiCl_2 , and $\text{Na}_2(\text{i-mnt})$ (i-mnt is 1,1-dicyanoethene-2,2-dithiolate) with 15-crown-5 (15-C-5) leads to an infinite chain polymer, $[\{\text{NiMg}_2(\text{C}_4\text{N}_2\text{S}_2)_2(\text{C}_{10}\text{H}_{20}\text{O}_5)_2\}\text{Cl}_2]_n$ or $[\{\text{Mg}(15\text{-C-5})_2[\text{Ni}(\text{i-mnt})_2]\text{Cl}_2\}]_n$, which consists of two $[\text{Mg}(15\text{-C-5})]^{2+}$ complex cations, one $[\text{Ni}(\text{i-mnt})_2]^{2-}$ complex anion and two Cl^- ions per formula unit. In the $[\text{Ni}(\text{i-mnt})_2]^{2-}$ complex anion, Ni^{2+} is located on a crystallographic mirror plane with a slightly distorted square-planar coordination by four S atoms. In the $[\text{Mg}(15\text{-C-5})]^{2+}$ complex cations, the Mg and one O atom of the crown lie on mirror planes and the Mg atoms are in sevenfold coordination environments of five O atoms from the crown and two N atoms from two i-mnt anions. The bridging of the two complexes *via* the Mg—N bonds leads to the formation of one-dimensional chains along the *a* axis.

Related literature

For studies on crown ether complexes of alkaline earth metals, see: Junk & Steed (1999). For comparative data on Ni—S bonds, see: Gao *et al.* (2005). For comparative data on Mg—O bonds, see: Chadwick *et al.* (1999).



Experimental

Crystal data

$[\text{NiMg}_2(\text{C}_4\text{N}_2\text{S}_2)_2(\text{C}_{10}\text{H}_{20}\text{O}_5)_2]\text{Cl}_2$
 $M_r = 899.11$
Orthorhombic, $Cmc2_1$
 $a = 13.6227$ (16) Å
 $b = 20.591$ (3) Å
 $c = 15.148$ (2) Å
 $V = 4249$ (1) Å³
 $Z = 4$
Mo $K\alpha$ radiation
 $\mu = 0.86$ mm⁻¹
 $T = 298$ (2) K
 $0.41 \times 0.32 \times 0.30$ mm

Data collection

Bruker SMART CCD area-detector diffractometer
Absorption correction: multi-scan (SADABS; Sheldrick, 1996)
 $T_{\min} = 0.719$, $T_{\max} = 0.783$
10632 measured reflections
1979 independent reflections
1651 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.033$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.065$
 $wR(F^2) = 0.192$
 $S = 1.06$
1979 reflections
242 parameters
1 restraint
H-atom parameters constrained
 $\Delta\rho_{\max} = 0.92$ e Å⁻³
 $\Delta\rho_{\min} = -0.47$ e Å⁻³
Absolute structure: Flack (1983),
1979 Friedel pairs
Flack parameter: 0.02 (5)

Data collection: SMART (Bruker, 1997); cell refinement: SAINT (Bruker, 1997); data reduction: SAINT; program(s) used to solve structure: SHELXS97 (Sheldrick, 1997a); program(s) used to refine structure: SHELXL97 (Sheldrick, 1997a); molecular graphics: SHELXTL (Sheldrick, 1997b); software used to prepare material for publication: SHELXTL.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: SQ2001).

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supplementary materials

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***catena*-Poly[[nickel(II)- μ_3 -1,1-dicyanoethene-2,2-dithiolato- κ^4 S,S':N:N'-bis[(15-crown-5)magnesium(II)]- μ_3 -1,1-dicyanoethene-2,2-dithiolato- κ^4 N:N':S,S'] dichloride]**

J. Yang, C. Li, D. Li and D. Wang

Comment

Crown ethers have gained special attention due to their coordination abilities with not only alkali metal ions, but also alkaline earth ions (Junk & Steed, 1999). In this work, we report the synthesis and structure of a crown ether complex of Mg^{2+} networked with a dithiolate Ni^{II} complex. As shown in Fig. 1, the asymmetric unit of title complex is made up of two half $[\text{Mg}(15\text{---C-5})]^{2+}$ complex cations, one half $[\text{Ni}(\text{i-mnt})_2]^{2-}$ complex anion and two dissociative Cl^- ions on mirror planes. For the two crystallographically independent $[\text{Mg}(15\text{---C-5})]^{2+}$ complex cations, each Mg^{2+} is coordinated by five O atoms of the crown ether with the average Mg–O distance of 2.566 (12) Å, which is far longer than the value in the complex $[\text{Mg}(15\text{---C-5})(\text{SCPh}_3)_2](2.177 \text{ \AA})$ (Chadwick *et al.*, 1999). The additional coordination sites of Mg^{2+} are occupied by two N atoms from cyano groups of the neighboring complex anions $[\text{Ni}(\text{i-mnt})_2]^{2-}$, with the average Mg–N bond length of 2.531 (12) Å. For the complex anion, the Ni^{2+} is coordinated by four S atoms of two (i-mnt)²⁻ anions in a square planar geometry. The Ni–S bond lengths are in the range of 2.207 (3) to 2.212 (3) Å, which is in perfect agreement with the values (average 2.215 Å) reported in the complex $[\text{Na}(\text{N15---C-5})_2][\text{Ni}(\text{i-mnt})_2]$ (Gao *et al.*, 2005). Fig. 2 shows that the title complex is assembled into a one-dimensional polymer by the Mg–N bonds between the adjacent $[\text{Mg}(15\text{---C-5})]^{2+}$ complex cations and the $[\text{Ni}(\text{i-mnt})_2]^{2-}$ complex anions along the *a* axis. This motif is similar to what is found in the complex $[\text{Na}(\text{N15---C-5})_2][\text{Ni}(\text{i-mnt})_2]$, which is also assembled into a one-dimensional structure by the Na–N bonds between the complex cations and the complex anions. $[\text{Na}(\text{N15---C-5})_2][\text{Ni}(\text{i-mnt})_2]$ further exhibits a two-dimensional supramolecular structure resulting from π - π stacking interactions between the naphthylene moieties of N15–C-5, which is not observed in the title complex.

Experimental

A solution of NiCl_2 (0.2377 g, 0.1 mmol), $\text{Na}_2(\text{i-mnt})$ and MgCl_2 in methanol (10 ml), was added to a solution of 15-C-5 (0.44 g, 2 mmol) in CH_2Cl_2 (10 ml). The mixture was stirred for 3 hrs at room temperature, and then separated. The underlayer was recrystallized in a mixture of CH_2Cl_2 and ether, and crystals suitable for X-ray diffraction were obtained after two weeks (m.p. 471–473 K). Analysis calc. for $\text{C}_{28}\text{H}_{40}\text{Cl}_2\text{Mg}_2\text{N}_4\text{O}_{10}\text{S}_4$: C 37.37, H 4.49, N 6.23%; found: C 37.29, H 4.40, N 6.32%.

Refinement

All H atoms were placed in geometrically idealized positions (C–H 0.97 Å) and treated as riding on their parent atoms, with $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{C})$. 1979 Friedel pairs were used to determine the Flack parameter.

Figures

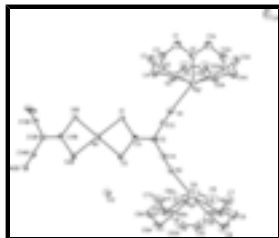


Fig. 1. The molecular structure of (I), with atom labels and 20% probability displacement ellipsoids for non-H atoms. Symmetry codes: (A) 1 - x , + y , + z ; (B) - x , + y , + z .



Fig. 2. One-dimensional chain-like structure of the title complex (Cl^- ions are omitted).

catena-Poly[[nickel(II)- μ_3 -1,1-dicyanoethene-2,2-dithiolato- κ^4 S,S':N:N']-bis[(15-crown-5)magnesium(II)]- μ_3 -1,1-dicyanoethene-2,2-dithiolato- κ^4 N:N':S,S'] dichloride]

Crystal data

$[\text{NiMg}_2(\text{C}_4\text{N}_2\text{S}_2)_2(\text{C}_{10}\text{H}_{20}\text{O}_5)_2]\text{Cl}_2$

$M_r = 899.11$

Orthorhombic, $Cmc2_1$

Hall symbol: C 2c -2

$a = 13.6227$ (16) Å

$b = 20.591$ (3) Å

$c = 15.148$ (2) Å

$V = 4249$ (1) Å³

$Z = 4$

$F_{000} = 1864$

$D_x = 1.406$ Mg m⁻³

Mo $K\alpha$ radiation

$\lambda = 0.71073$ Å

Cell parameters from 4296 reflections

$\theta = 2.2$ – 24.9°

$\mu = 0.86$ mm⁻¹

$T = 298$ (2) K

Block, brown

$0.41 \times 0.32 \times 0.30$ mm

Data collection

Bruker SMART CCD area-detector diffractometer

Radiation source: fine-focus sealed tube

Monochromator: graphite

$T = 298$ (2) K

φ and ω scans

Absorption correction: multi-scan (SADABS; Sheldrick, 1996)

$T_{\min} = 0.719$, $T_{\max} = 0.783$

10632 measured reflections

1979 independent reflections

1651 reflections with $I > 2\sigma(I)$

$R_{\text{int}} = 0.033$

$\theta_{\text{max}} = 25.0^\circ$

$\theta_{\text{min}} = 2.4^\circ$

$h = -16 \rightarrow 16$

$k = -24 \rightarrow 23$

$l = -9 \rightarrow 18$

Refinement

Refinement on F^2

Hydrogen site location: inferred from neighbouring sites

| | |
|--|--|
| Least-squares matrix: full | H-atom parameters constrained |
| $R[F^2 > 2\sigma(F^2)] = 0.065$ | $w = 1/[\sigma^2(F_o^2) + (0.1309P)^2 + 12.7693P]$ |
| $wR(F^2) = 0.192$ | where $P = (F_o^2 + 2F_c^2)/3$ |
| $S = 1.06$ | $(\Delta/\sigma)_{\max} = 0.001$ |
| 1979 reflections | $\Delta\rho_{\max} = 0.92 \text{ e } \text{\AA}^{-3}$ |
| 242 parameters | $\Delta\rho_{\min} = -0.46 \text{ e } \text{\AA}^{-3}$ |
| 1 restraint | Extinction correction: none |
| Primary atom site location: structure-invariant direct methods | Absolute structure: Flack (1983) |
| Secondary atom site location: difference Fourier map | Flack parameter: 0.02 (5) |

Special details

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

Refinement. Refinement of F^2 against ALL reflections. The weighted R -factor wR and goodness of fit S are based on F^2 , conventional R -factors R are based on F , with F set to zero for negative F^2 . The threshold expression of $F^2 > \sigma(F^2)$ is used only for calculating R -factors(gt) *etc.* and is not relevant to the choice of reflections for refinement. R -factors based on F^2 are statistically about twice as large as those based on F , and R -factors based on ALL data will be even larger.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (\AA^2)

| | x | y | z | $U_{\text{iso}}^*/U_{\text{eq}}$ |
|-----|--------------|--------------|--------------|----------------------------------|
| Ni1 | 0.0000 | 0.12793 (7) | 0.9803 (2) | 0.0492 (4) |
| Mg1 | 0.5000 | 0.3176 (3) | 1.0866 (4) | 0.0713 (14) |
| Mg2 | 0.5000 | -0.0308 (2) | 0.8135 (3) | 0.0565 (11) |
| N1 | 0.3850 (8) | 0.2476 (5) | 0.9910 (9) | 0.093 (4) |
| N2 | 0.3863 (6) | 0.0587 (5) | 0.8547 (8) | 0.074 (3) |
| O1 | 0.5000 | 0.4399 (6) | 1.1122 (9) | 0.084 (3) |
| O2 | 0.3268 (7) | 0.3721 (4) | 1.1116 (7) | 0.084 (2) |
| O3 | 0.3963 (10) | 0.2655 (6) | 1.2030 (8) | 0.112 (4) |
| O4 | 0.5000 | -0.0908 (6) | 0.9581 (9) | 0.109 (5) |
| O5 | 0.6412 (9) | -0.1054 (5) | 0.8323 (8) | 0.103 (3) |
| O6 | 0.5988 (12) | -0.0446 (7) | 0.6826 (9) | 0.131 (4) |
| Cl1 | 0.0000 | 0.9064 (4) | 0.8312 (9) | 0.159 (4) |
| Cl2 | 0.0000 | 0.2859 (4) | 0.2265 (8) | 0.149 (3) |
| S1 | 0.12480 (17) | 0.18894 (12) | 1.02320 (19) | 0.0571 (6) |
| S2 | 0.12522 (16) | 0.07215 (11) | 0.9274 (2) | 0.0564 (6) |
| C1 | 0.4140 (11) | 0.4707 (7) | 1.1444 (12) | 0.095 (4) |
| H1A | 0.4158 | 0.5167 | 1.1309 | 0.114* |
| H1B | 0.4095 | 0.4655 | 1.2079 | 0.114* |
| C2 | 0.3302 (12) | 0.4407 (6) | 1.1020 (12) | 0.098 (4) |
| H2A | 0.2706 | 0.4591 | 1.1265 | 0.118* |
| H2B | 0.3316 | 0.4512 | 1.0395 | 0.118* |

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| | | | | |
|------|-------------|--------------|-------------|-------------|
| C3 | 0.2781 (12) | 0.3557 (9) | 1.1909 (12) | 0.100 (4) |
| H3A | 0.2084 | 0.3647 | 1.1863 | 0.120* |
| H3B | 0.3047 | 0.3803 | 1.2401 | 0.120* |
| C4 | 0.2959 (14) | 0.2843 (9) | 1.2035 (13) | 0.108 (5) |
| H4A | 0.2670 | 0.2712 | 1.2592 | 0.129* |
| H4B | 0.2620 | 0.2609 | 1.1569 | 0.129* |
| C5 | 0.4508 (14) | 0.2588 (10) | 1.2820 (11) | 0.116 (6) |
| H5A | 0.4301 | 0.2936 | 1.3210 | 0.140* |
| H5B | 0.4301 | 0.2185 | 1.3093 | 0.140* |
| C6 | 0.589 (2) | -0.1249 (9) | 0.9749 (16) | 0.132 (7) |
| H6A | 0.5784 | -0.1707 | 0.9635 | 0.159* |
| H6B | 0.6055 | -0.1202 | 1.0368 | 0.159* |
| C7 | 0.674 (2) | -0.1019 (10) | 0.9200 (15) | 0.130 (7) |
| H7A | 0.6922 | -0.0577 | 0.9353 | 0.156* |
| H7B | 0.7311 | -0.1297 | 0.9287 | 0.156* |
| C8 | 0.7145 (16) | -0.0869 (10) | 0.7714 (16) | 0.123 (6) |
| H8A | 0.7715 | -0.1148 | 0.7775 | 0.147* |
| H8B | 0.7347 | -0.0425 | 0.7823 | 0.147* |
| C9 | 0.6722 (17) | -0.0931 (10) | 0.6793 (15) | 0.127 (7) |
| H9A | 0.7209 | -0.0838 | 0.6343 | 0.152* |
| H9B | 0.6445 | -0.1358 | 0.6691 | 0.152* |
| C10 | 0.552 (2) | -0.0474 (13) | 0.600 (2) | 0.172 (11) |
| H10A | 0.5743 | -0.0863 | 0.5698 | 0.206* |
| H10B | 0.5743 | -0.0105 | 0.5652 | 0.206* |
| C11 | 0.1961 (6) | 0.1356 (4) | 0.9620 (6) | 0.051 (2) |
| C12 | 0.2935 (7) | 0.1459 (4) | 0.9403 (7) | 0.0521 (19) |
| C13 | 0.3435 (7) | 0.2023 (5) | 0.9693 (8) | 0.061 (2) |
| C14 | 0.3456 (7) | 0.0975 (5) | 0.8923 (8) | 0.057 (2) |

Atomic displacement parameters (\AA^2)

| | U^{11} | U^{22} | U^{33} | U^{12} | U^{13} | U^{23} |
|-----|-------------|-------------|-------------|------------|-------------|--------------|
| Ni1 | 0.0437 (7) | 0.0506 (8) | 0.0532 (8) | 0.000 | 0.000 | -0.0009 (7) |
| Mg1 | 0.077 (3) | 0.067 (3) | 0.069 (3) | 0.000 | 0.000 | -0.006 (3) |
| Mg2 | 0.057 (2) | 0.058 (2) | 0.055 (2) | 0.000 | 0.000 | -0.009 (2) |
| N1 | 0.075 (6) | 0.090 (7) | 0.116 (9) | -0.019 (5) | 0.025 (6) | -0.051 (7) |
| N2 | 0.044 (4) | 0.075 (6) | 0.104 (7) | 0.008 (4) | 0.006 (5) | -0.033 (6) |
| O1 | 0.080 (8) | 0.071 (7) | 0.101 (9) | 0.000 | 0.000 | -0.023 (6) |
| O2 | 0.078 (5) | 0.086 (5) | 0.089 (6) | 0.005 (4) | 0.003 (5) | -0.012 (5) |
| O3 | 0.121 (9) | 0.138 (9) | 0.077 (6) | 0.016 (7) | 0.026 (6) | 0.007 (6) |
| O4 | 0.185 (16) | 0.072 (7) | 0.069 (8) | 0.000 | 0.000 | 0.007 (6) |
| O5 | 0.126 (9) | 0.080 (6) | 0.103 (8) | 0.020 (6) | -0.031 (7) | -0.013 (6) |
| O6 | 0.173 (12) | 0.117 (9) | 0.103 (9) | 0.033 (9) | 0.043 (9) | 0.004 (7) |
| Cl1 | 0.130 (6) | 0.122 (5) | 0.225 (11) | 0.000 | 0.000 | 0.018 (7) |
| Cl2 | 0.115 (5) | 0.136 (5) | 0.197 (9) | 0.000 | 0.000 | 0.046 (6) |
| S1 | 0.0443 (12) | 0.0618 (13) | 0.0652 (14) | 0.0027 (9) | 0.0003 (11) | -0.0202 (12) |
| S2 | 0.0471 (12) | 0.0464 (11) | 0.0757 (15) | 0.0005 (8) | 0.0012 (12) | -0.0051 (11) |
| C1 | 0.092 (9) | 0.084 (8) | 0.108 (11) | 0.010 (7) | -0.009 (9) | -0.006 (8) |

| | | | | | | |
|-----|------------|------------|------------|-------------|-------------|-------------|
| C2 | 0.096 (10) | 0.081 (8) | 0.117 (11) | 0.016 (7) | -0.011 (9) | -0.009 (8) |
| C3 | 0.085 (9) | 0.116 (12) | 0.100 (11) | -0.005 (8) | 0.004 (8) | -0.006 (9) |
| C4 | 0.103 (11) | 0.120 (12) | 0.101 (11) | -0.014 (9) | 0.026 (9) | -0.002 (10) |
| C5 | 0.132 (13) | 0.136 (14) | 0.081 (9) | -0.036 (10) | 0.006 (9) | 0.012 (9) |
| C6 | 0.21 (2) | 0.098 (11) | 0.085 (9) | 0.031 (14) | -0.032 (16) | 0.003 (9) |
| C7 | 0.161 (19) | 0.104 (12) | 0.126 (16) | 0.037 (12) | -0.043 (15) | -0.029 (12) |
| C8 | 0.121 (15) | 0.108 (12) | 0.140 (16) | 0.038 (11) | 0.015 (14) | -0.022 (12) |
| C9 | 0.138 (16) | 0.110 (13) | 0.132 (16) | 0.022 (12) | 0.044 (14) | -0.013 (11) |
| C10 | 0.20 (3) | 0.18 (2) | 0.135 (18) | 0.064 (19) | 0.041 (18) | -0.006 (17) |
| C11 | 0.046 (4) | 0.052 (4) | 0.055 (6) | 0.007 (4) | -0.005 (4) | 0.001 (4) |
| C12 | 0.052 (5) | 0.049 (4) | 0.055 (5) | 0.006 (4) | -0.002 (4) | -0.005 (4) |
| C13 | 0.053 (5) | 0.062 (5) | 0.066 (6) | -0.002 (4) | 0.004 (5) | -0.017 (5) |
| C14 | 0.048 (5) | 0.052 (5) | 0.072 (6) | 0.000 (4) | -0.003 (5) | -0.006 (5) |

Geometric parameters (Å, °)

| | | | |
|-------------------------|-------------|------------|------------|
| Ni1—S2 ⁱ | 2.207 (3) | O6—C10 | 1.41 (3) |
| Ni1—S2 | 2.207 (3) | O6—C9 | 1.41 (2) |
| Ni1—S1 | 2.212 (3) | S1—C11 | 1.734 (9) |
| Ni1—S1 ⁱ | 2.212 (3) | S2—C11 | 1.708 (9) |
| Mg1—O3 | 2.502 (13) | C1—C2 | 1.45 (2) |
| Mg1—O3 ⁱⁱ | 2.502 (13) | C1—H1A | 0.9700 |
| Mg1—O1 | 2.548 (13) | C1—H1B | 0.9700 |
| Mg1—N1 | 2.575 (12) | C2—H2A | 0.9700 |
| Mg1—N1 ⁱⁱ | 2.575 (12) | C2—H2B | 0.9700 |
| Mg1—O2 ⁱⁱ | 2.640 (10) | C3—C4 | 1.50 (2) |
| Mg1—O2 | 2.640 (10) | C3—H3A | 0.9700 |
| Mg2—O6 ⁱⁱ | 2.414 (12) | C3—H3B | 0.9700 |
| Mg2—O6 | 2.414 (12) | C4—H4A | 0.9700 |
| Mg2—O5 ⁱⁱ | 2.478 (11) | C4—H4B | 0.9700 |
| Mg2—O5 | 2.478 (11) | C5—H5A | 0.9700 |
| Mg2—N2 ⁱⁱ | 2.487 (10) | C5—H5B | 0.9700 |
| Mg2—N2 | 2.487 (9) | C6—C7 | 1.51 (3) |
| Mg2—O4 | 2.514 (14) | C6—H6A | 0.9700 |
| N1—C13 | 1.140 (13) | C6—H6B | 0.9700 |
| N2—C14 | 1.128 (13) | C7—H7A | 0.9700 |
| O1—C1 ⁱⁱ | 1.418 (16) | C7—H7B | 0.9700 |
| O1—C1 | 1.418 (16) | C8—C9 | 1.51 (3) |
| O2—C3 | 1.41 (2) | C8—H8A | 0.9700 |
| O2—C2 | 1.420 (15) | C8—H8B | 0.9700 |
| O3—C5 | 1.41 (2) | C9—H9A | 0.9700 |
| O3—C4 | 1.42 (2) | C9—H9B | 0.9700 |
| O4—C6 | 1.42 (2) | C10—H10A | 0.9700 |
| O4—C6 ⁱⁱ | 1.42 (2) | C10—H10B | 0.9700 |
| O5—C7 | 1.40 (2) | C11—C12 | 1.383 (13) |
| O5—C8 | 1.41 (2) | | |
| S2 ⁱ —Ni1—S2 | 101.24 (16) | C11—S2—Ni1 | 85.8 (3) |

supplementary materials

| | | | |
|--|-------------|--------------------------|------------|
| S2 ⁱ —Ni1—S1 | 175.2 (2) | O1—C1—C2 | 108.0 (12) |
| S2—Ni1—S1 | 78.94 (8) | O1—C1—H1A | 110.1 |
| S2 ⁱ —Ni1—S1 ⁱ | 78.94 (8) | C2—C1—H1A | 110.1 |
| S2—Ni1—S1 ⁱ | 175.2 (2) | O1—C1—H1B | 110.1 |
| S1—Ni1—S1 ⁱ | 100.48 (15) | C2—C1—H1B | 110.1 |
| O3—Mg1—O3 ⁱⁱ | 68.8 (6) | H1A—C1—H1B | 108.4 |
| O3—Mg1—O1 | 108.5 (4) | O2—C2—C1 | 113.8 (12) |
| O3 ⁱⁱ —Mg1—O1 | 108.5 (4) | O2—C2—H2A | 108.8 |
| O3—Mg1—N1 | 79.2 (4) | C1—C2—H2A | 108.8 |
| O3 ⁱⁱ —Mg1—N1 | 120.0 (5) | O2—C2—H2B | 108.8 |
| O1—Mg1—N1 | 129.7 (4) | C1—C2—H2B | 108.8 |
| O3—Mg1—N1 ⁱⁱ | 120.0 (5) | H2A—C2—H2B | 107.7 |
| O3 ⁱⁱ —Mg1—N1 ⁱⁱ | 79.2 (4) | O2—C3—C4 | 105.4 (13) |
| O1—Mg1—N1 ⁱⁱ | 129.7 (4) | O2—C3—H3A | 110.7 |
| N1—Mg1—N1 ⁱⁱ | 74.9 (5) | C4—C3—H3A | 110.7 |
| O3—Mg1—O2 ⁱⁱ | 125.9 (4) | O2—C3—H3B | 110.7 |
| O3 ⁱⁱ —Mg1—O2 ⁱⁱ | 64.9 (4) | C4—C3—H3B | 110.7 |
| O1—Mg1—O2 ⁱⁱ | 63.8 (2) | H3A—C3—H3B | 108.8 |
| N1—Mg1—O2 ⁱⁱ | 149.6 (4) | O3—C4—C3 | 114.9 (14) |
| N1 ⁱⁱ —Mg1—O2 ⁱⁱ | 77.0 (3) | O3—C4—H4A | 108.5 |
| O3—Mg1—O2 | 64.9 (4) | C3—C4—H4A | 108.5 |
| O3 ⁱⁱ —Mg1—O2 | 125.9 (4) | O3—C4—H4B | 108.5 |
| O1—Mg1—O2 | 63.8 (2) | C3—C4—H4B | 108.5 |
| N1—Mg1—O2 | 77.0 (3) | H4A—C4—H4B | 107.5 |
| N1 ⁱⁱ —Mg1—O2 | 149.6 (4) | C5 ⁱⁱ —C5—O3 | 121.6 (8) |
| O2 ⁱⁱ —Mg1—O2 | 126.7 (4) | C5 ⁱⁱ —C5—H5A | 106.9 |
| O6 ⁱⁱ —Mg2—O6 | 67.8 (9) | O3—C5—H5A | 106.9 |
| O6 ⁱⁱ —Mg2—O5 ⁱⁱ | 65.7 (5) | C5 ⁱⁱ —C5—H5B | 106.9 |
| O6—Mg2—O5 ⁱⁱ | 117.0 (5) | O3—C5—H5B | 106.9 |
| O6 ⁱⁱ —Mg2—O5 | 117.0 (5) | H5A—C5—H5B | 106.7 |
| O6—Mg2—O5 | 65.7 (5) | O4—C6—C7 | 113.9 (16) |
| O5 ⁱⁱ —Mg2—O5 | 101.9 (6) | O4—C6—H6A | 108.8 |
| O6 ⁱⁱ —Mg2—N2 ⁱⁱ | 129.8 (5) | C7—C6—H6A | 108.8 |
| O6—Mg2—N2 ⁱⁱ | 86.9 (5) | O4—C6—H6B | 108.8 |
| O5 ⁱⁱ —Mg2—N2 ⁱⁱ | 156.1 (4) | C7—C6—H6B | 108.8 |
| O5—Mg2—N2 ⁱⁱ | 87.0 (3) | H6A—C6—H6B | 107.7 |
| O6 ⁱⁱ —Mg2—N2 | 86.9 (5) | O5—C7—C6 | 104.8 (19) |
| O6—Mg2—N2 | 129.8 (5) | O5—C7—H7A | 110.8 |
| O5 ⁱⁱ —Mg2—N2 | 87.0 (3) | C6—C7—H7A | 110.8 |
| O5—Mg2—N2 | 156.1 (4) | O5—C7—H7B | 110.8 |
| N2 ⁱⁱ —Mg2—N2 | 77.0 (4) | C6—C7—H7B | 110.8 |
| O6 ⁱⁱ —Mg2—O4 | 131.1 (4) | H7A—C7—H7B | 108.9 |

| | | | |
|--------------------------|------------|-----------------------------|------------|
| O6—Mg2—O4 | 131.1 (4) | O5—C8—C9 | 108.1 (17) |
| O5 ⁱⁱ —Mg2—O4 | 66.1 (4) | O5—C8—H8A | 110.1 |
| O5—Mg2—O4 | 66.1 (4) | C9—C8—H8A | 110.1 |
| N2 ⁱⁱ —Mg2—O4 | 98.4 (4) | O5—C8—H8B | 110.1 |
| N2—Mg2—O4 | 98.4 (4) | C9—C8—H8B | 110.1 |
| C13—N1—Mg1 | 157.3 (12) | H8A—C8—H8B | 108.4 |
| C14—N2—Mg2 | 163.5 (10) | O6—C9—C8 | 100.3 (15) |
| C1 ⁱⁱ —O1—C1 | 111.5 (14) | O6—C9—H9A | 111.7 |
| C1 ⁱⁱ —O1—Mg1 | 119.6 (7) | C8—C9—H9A | 111.7 |
| C1—O1—Mg1 | 119.6 (8) | O6—C9—H9B | 111.7 |
| C3—O2—C2 | 109.9 (13) | C8—C9—H9B | 111.7 |
| C3—O2—Mg1 | 116.1 (9) | H9A—C9—H9B | 109.5 |
| C2—O2—Mg1 | 112.2 (9) | O6—C10—C10 ⁱⁱ | 116.9 (12) |
| C5—O3—C4 | 121.8 (13) | O6—C10—H10A | 108.1 |
| C5—O3—Mg1 | 110.0 (10) | C10 ⁱⁱ —C10—H10A | 108.1 |
| C4—O3—Mg1 | 115.4 (10) | O6—C10—H10B | 108.1 |
| C6—O4—C6 ⁱⁱ | 117 (2) | C10 ⁱⁱ —C10—H10B | 108.1 |
| C6—O4—Mg2 | 113.4 (13) | H10A—C10—H10B | 107.3 |
| C6 ⁱⁱ —O4—Mg2 | 113.4 (13) | C12—C11—S2 | 125.9 (7) |
| C7—O5—C8 | 112.1 (18) | C12—C11—S1 | 124.6 (7) |
| C7—O5—Mg2 | 109.1 (11) | S2—C11—S1 | 109.4 (5) |
| C8—O5—Mg2 | 107.9 (10) | C11—C12—C13 | 120.9 (8) |
| C10—O6—C9 | 105.1 (16) | C11—C12—C14 | 119.5 (8) |
| C10—O6—Mg2 | 119.0 (14) | C13—C12—C14 | 119.5 (8) |
| C9—O6—Mg2 | 120.5 (12) | N1—C13—C12 | 178.5 (12) |
| C11—S1—Ni1 | 85.1 (3) | N2—C14—C12 | 179.3 (13) |

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

Fig. 2

