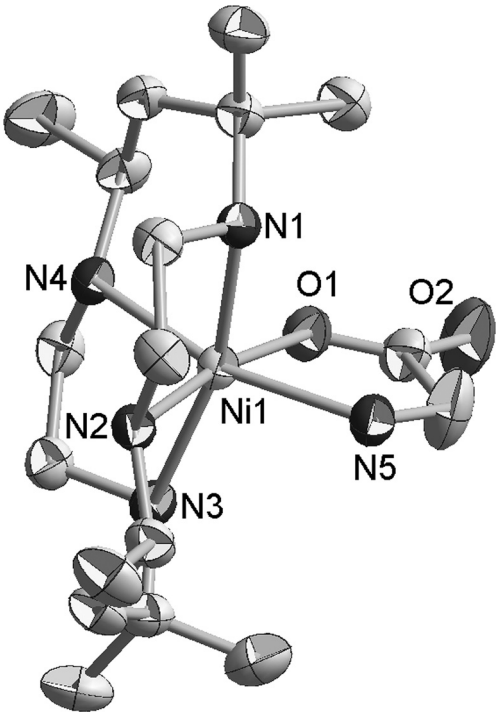
Zu-Jiao Zhang and Guang-Chuan Ou*

Crystal structure of (glycinto-κ^2O,N')-[5,5,7,12,12,14-hexamethyl-1,4,8,11-tetraazacyclotetradecane-κ^4N,N',N'',N'''] nickel(II) perchlorate monohydrate $C_{18}H_{42}ClN_5NiO_7$



https://doi.org/10.1515/ncrs-2023-0131
Received March 19, 2023; accepted April 14, 2023;
published online April 25, 2023

Abstract

$C_{18}H_{42}ClN_5NiO_7$, orthorhombic, $Pca2_1$ (no. 29), a = 14.270(3) Å, b = 9.918(2) Å, c = 17.603(4) Å, V = 2491.4(10) Å^3, Z = 4, $R_{gt}(F)$ = 0.0382, $wR_{ref}(F^2)$ = 0.0861, T = 173(2) K.

CCDC no.: 2256322

***Corresponding author: Guang-Chuan Ou**, Department of Biology and Chemistry, Hunan University of Science and Engineering, Yongzhou, Hunan 425199, P.R. China, E-mail: ogcouguangchuan@163.com. https://orcid.org/0000-0002-1005-8519

Zu-Jiao Zhang, Department of Biology and Chemistry, Hunan University of Science and Engineering, Yongzhou, Hunan 425199, P.R. China

Table 1: Data collection and handling.

Crystal:	Blue block
Size:	0.46 × 0.42 × 0.15 mm
Wavelength:	Mo *K*α radiation (0.71073 Å)
μ:	0.93 mm^{-1}
Diffractometer, scan mode:	φ and ω
θ_{max}, completeness:	27.1°, >99 %
$N(hkl)_{measured}$, $N(hkl)_{unique}$, R_{int}:	12,902, 5229, 0.041
Criterion for I_{obs}, $N(hkl)_{gt}$:	I_{obs} > 2 σ(I_{obs}), 3834
$N(param)_{refined}$:	301
Programs:	Bruker [1], Shelx [2, 3], Diamond [4]

The molecular structure is shown in the figure. Table 1 contains crystallographic data and Table 2 contains the list of the atoms including atomic coordinates and displacement parameters.

1 Source of material

A water solution (20 mL) of glycine (Gly, 0.150 g, 2 mmol) and NaOH (0.08 g, 2 mmol) was added to an acetonitrile solution of [Ni(rac-L)] $(ClO_4)_2$ (0.108 g, 2 mmol). The resulting solution was evaporated slowly at room temperature and blue crystals of the title compound were obtained after 2 weeks.

2 Experimental details

The structure was solved using Direct Methods, which yielded the positions of all non-hydrogen atoms. All hydrogen atoms of the ligands were placed in calculated positions with fixed isotropic thermal parameters and included in the structure factor calculations in the final stage of full-matrix least-squares refinement. The U_{iso} values of the hydrogen atoms of methyl groups were set to

Table 2: Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å^2).

Atom	*x*	*y*	*z*	U_{iso}*/U_{eq}
C1	1.1261 (4)	0.7738 (5)	0.6581 (3)	0.0423 (12)
H1A	1.116957	0.872696	0.659376	0.051*
H1B	1.184886	0.754598	0.630204	0.051*
C2	1.1329 (4)	0.7200 (5)	0.7376(3)	0.0432 (13)
H2A	1.140334	0.620750	0.736132	0.052*
H2B	1.188475	0.758754	0.763131	0.052*
C3	1.0416(4)	0.6899 (5)	0.8561 (3)	0.0448 (13)
H3A	1.025718	0.592680	0.848124	0.054*
C4	1.1334 (4)	0.6964 (7)	0.9009 (3)	0.0630 (18)
H4A	1.180657	0.640025	0.875861	0.095*
H4B	1.122873	0.663371	0.952660	0.095*
H4C	1.155549	0.789835	0.902800	0.095*
C5	0.9631 (4)	0.7532 (6)	0.9027 (3)	0.0489 (14)
H5C	0.972783	0.852046	0.902042	0.059*
H5D	0.971345	0.723339	0.955971	0.059*
C6	0.8607 (4)	0.7269 (5)	0.8810 (3)	0.0450 (12)
C7	0.7982 (4)	0.8034 (7)	0.9369 (3)	0.0620 (16)
H7A	0.732852	0.798119	0.920081	0.093*
H7B	0.817753	0.898074	0.938960	0.093*
H7C	0.804018	0.763105	0.987519	0.093*
C8	0.8368 (4)	0.5791 (6)	0.8862 (3)	0.0653 (16)
H8A	0.772521	0.564715	0.868473	0.098*
H8B	0.842299	0.549313	0.939115	0.098*
H8C	0.880224	0.527224	0.854467	0.098*
C9	0.8329 (4)	0.9174 (5)	0.7913 (3)	0.0449 (12)
H9A	0.888680	0.963050	0.812612	0.054*
H9B	0.777145	0.948494	0.819855	0.054*
C10	0.8224 (3)	0.9540 (4)	0.7088 (3)	0.0432 (10)
H10A	0.766826	0.908022	0.687305	0.052*
H10B	0.813094	1.052475	0.703779	0.052*
C11	0.8952 (4)	0.9239 (5)	0.5828 (3)	0.0438 (12)
H11	0.849838	0.852990	0.566207	0.053*
C12	0.8567 (5)	1.0613 (6)	0.5583 (3)	0.0716 (18)
H12A	0.794387	1.074633	0.580496	0.107*
H12B	0.852142	1.064472	0.502775	0.107*
H12C	0.898862	1.132733	0.575930	0.107*
C13	0.9874 (4)	0.8998 (5)	0.5418 (3)	0.0432 (12)
H13A	1.034770	0.960197	0.564872	0.052*
H13B	0.979134	0.929293	0.488479	0.052*
C14	1.0290 (4)	0.7583 (5)	0.5401 (3)	0.0411 (12)
C15	0.9633 (4)	0.6570 (6)	0.5021 (3)	0.0529 (15)
H15A	0.991451	0.566967	0.503921	0.079*
H15B	0.953132	0.683231	0.449067	0.079*
H15C	0.903149	0.655936	0.528986	0.079*
C16	1.1207 (4)	0.7625 (7)	0.4939 (3)	0.0539 (15)
H16A	1.159920	0.836973	0.512022	0.081*
H16B	1.106098	0.776070	0.440021	0.081*
H16C	1.154447	0.677181	0.500225	0.081*
C17	0.7978 (4)	0.5358 (6)	0.6404 (3)	0.0511 (13)
C18	0.8629 (5)	0.4376 (6)	0.6781(4)	0.076(2)
H18A	0.827684	0.387780	0.717621	0.091*
H18B	0.884186	0.371264	0.639797	0.091*
Cl1	0.55698(9)	0.82599 (14)	0.78208 (8)	0.0515 (3)
N1	1.0460(3)	0.7087 (4)	0.6191 (2)	0.0347 (10)
H1	1.063540	0.611572	0.613710	0.042*
N2	1.0481 (3)	0.7548 (4)	0.7804 (2)	0.0366 (9)
H2	1.049338	0.854481	0.788900	0.044*
N3	0.8430 (3)	0.7703(4)	0.7998 (2)	0.0375 (9)
H3	0.780262	0.732113	0.786401	0.045*
N4	0.9068(3)	0.9131(4)	0.6669(2)	0.0357(9)
H4	0.959818	0.972803	0.682684	0.043*
N5	0.9449 (2)	0.4994 (4)	0.7131 (3)	0.0433 (9)
H5A	0.997703	0.470661	0.689052	0.052*
H5B	0.948608	0.474284	0.762698	0.052*
Ni1	0.93531 (3)	0.71420 (5)	0.70552 (3)	0.03245 (15)
O1	0.8167 (2)	0.6586 (3)	0.64791 (18)	0.0452 (8)
O1W	0.5746 (4)	0.6362 (7)	0.5983 (3)	0.0824 (17)
H1WA	0.583 (7)	0.704 (10)	0.619 (6)	0.124*
H1WB	0.626 (6)	0.603 (9)	0.602 (5)	0.124*
O2	0.7305 (3)	0.4889 (4)	0.6070 (3)	0.0816 (14)
O3	0.6161(3)	0.7138 (4)	0.7918 (3)	0.0764 (13)
O4	0.5809 (3)	0.9320 (5)	0.8335 (3)	0.0914 (15)
O5	0.5668 (3)	0.8754 (5)	0.7072 (3)	0.0931 (13)
O6	0.4639 (3)	0.7875 (4)	0.7941 (3)	0.0919 (16)

$1.5U_{eq}$(C) and the U_{iso} values of all other hydrogen atoms were set to $1.2U_{eq}$(C, N).

3 Comment

Similar to the title compound some structures have been reported [5, 6].

X-ray crystal structural analysis reveals that the title compound consists of one $[Ni(rac\text{-}L)(Gly)]^+$ cation, one ClO_4^- anion and one water molecule. The Ni(II) atom displays a six-coordinate octahedral geometry by coordination with four nitrogen atoms from the macrocycle ligand L in a folded conformation and one carboxylate oxygen atom and one nitrogen atom from Gly^- in *cis*-position. The $[Ni(rac\text{-}L)(Gly)]^+$ and lattice water molecules are linked by N–H···O and O–H···O hydrogen bonds into a one-dimensional chain.

Author contributions: All the authors have accepted responsibility for the entire content of this submitted manuscript and approved submission.

Research funding: This work was financially supported by the scientific research fund of Hunan provincial education department (22B0804) and the program of applied characteristic discipline in Hunan province.

Conflict of interest statement: The authors declare no conflicts of interest regarding this article.

References

1. Bruker. *Apex3, Saint–Plus, Xprep;* Bruker AXS Inc.: Madison, Wisconsin, USA, 2016.
2. Sheldrick G. M. *SHELXTL* – integrated space-group and crystal-structure determination. *Acta Crystallogr.* 2015, *A71*, 3–8.
3. Sheldrick G. M. Crystal structure refinement with SHELXL. *Acta Crystallogr.* 2015, *C71*, 3–8.
4. Brandenburg K. Diamond. Visual Crystal Structure Information System. Ver. 4.0; Crystal Impact: Bonn, Germany, 2015.
5. Ou G. C., Li Z. Z., Zhang M., Yuan X. Y. Chiral resolution of L- and D-alanine and a racemic macrocyclic nickel(II) complex: synthesis and crystal structures. *Transition Met. Chem.* 2014, *39*, 135–140.
6. Skoulika S., Michaelides A., Aubry A. Two ternary NiII complexes: bisglycinato(1,10-phenanthroline)nickel(II) trihydrate and bisglycinato(2,2′-bi-pyridine)nickel(II) trihydrate. *Acta Crystallogr.* 1995, *C51*, 843–846.