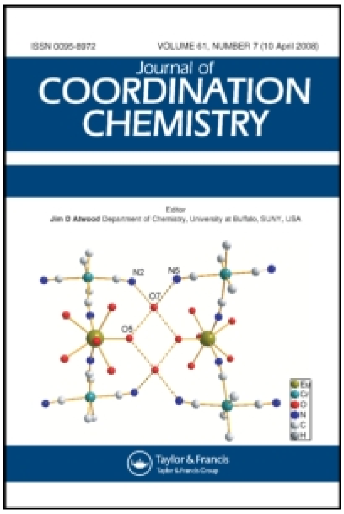

Journal of Coordination Chemistry
Publication details, including instructions for authors and subscription information:
http://www.informaworld.com/smpp/title~content=t713455674

Synthesis and Crystal Structure of $[Ni(H_2O)_6][Ni(H_2O)_2(C_4H_2O_4)]\cdot4H_2O$

Yue-Qing Zheng[a]; Zu-Ping Kong[a]
[a] Municipal Key Laboratory of Inorganic Materials Chemistry, Institute for Solid State Chemistry, Ningbo University, Ningbo, P.R. China

To cite this Article Zheng, Yue-Qing and Kong, Zu-Ping(2003) 'Synthesis and Crystal Structure of $[Ni(H_2O)_6][Ni(H_2O)_2(C_4H_2O_4)]\cdot4H_2O$', Journal of Coordination Chemistry, 56: 11, 967 — 973
To link to this Article: DOI: 10.1080/0095897031000135360
URL: http://dx.doi.org/10.1080/0095897031000135360

J. Coord. Chem. Vol. 56, No. 11, 20 July 2003, pp. 967–973

SYNTHESIS AND CRYSTAL STRUCTURE OF $[Ni(H_2O)_6][Ni(H_2O)_2(C_4H_2O_4)] \cdot 4H_2O$

YUE-QING ZHENG* and ZU-PING KONG

Municipal Key Laboratory of Inorganic Materials Chemistry,
Institute for Solid State Chemistry, Ningbo University, Ningbo, 315211 P.R. China

(Received 13 January 2003; In final form 03 April 2003)

Reaction of a freshly prepared $Ni(OH)_{2-2x}(CO_3)_x \cdot yH_2O$ with maleic acid in H_2O at room temperature afforded $[Ni(H_2O)_6][Ni(H_2O)_2(C_4H_2O_4)] \cdot 4H_2O$, which consists of $[Ni(H_2O)_6]^{2+}$ cations, $[Ni(H_2O)_2(C_4H_2O_4)]^{2-}$ anions and lattice H_2O molecules. Ni atoms in cations are octahedrally coordinated and Ni atoms in anions are each octahedrally coordinated by bidentate chelating maleato ligands and two water molecules at trans positions. Cations and anions are interlinked by hydrogen bonds to form 1D chains, which are hexagonally arranged and connected by the lattice water molecules. When heated in a flowing argon stream, the compound decomposes, with complete dehydration being followed by dissociation of nickel maleate into NiO and maleic anhydride.

Keywords: Ni(II); Maleato complex; Synthesis; Crystal structure; Thermal analysis

INTRODUCTION

Use of dicarboxylate spacer ligands is of current interest in construction of coordination polymers [1–3]. Over recent years, rational design and syntheses of coordination polymers based on saturated α,ω-dicarboxylate anions have attracted enormous attention [4–14]. As a continuing part of our research on self-assembly of transition metal cations with dicarboxylic acids [15], we have extended our investigation to the exploitation of unsaturated α,ω-dicarboxylic acids. As one of the lower members in the family of unsaturated α,ω-dicarboxylates, maleic acid is of particular interest. The partially deprotonated hydrogen maleato ligands can be viewed as a rigid bridging spacer [16] and fully deprotonated maleato ligands exhibit versatile coordination modes [17–24]. Up to now, however, no example has been reported of maleate anions acting as bidentate chelating ligands. Here, we report a new maleato Ni(II) complex $[Ni(H_2O)_6][Ni(H_2O)_2(C_4H_2O_4)] \cdot 4H_2O$, where bidentate maleate anions chelate Ni to form a seven-membered ring.

---

*Author for correspondence. E-mail: zhengcm@nbu.edu.cn

ISSN 0095-8972 print: ISSN 1029-0389 online
DOI: 10.1080/0095897031000135360

EXPERIMENTAL

Physical Measurements

C and H microanalyses were performed with a Heraeus Rapid–CHNO instrument. FT-IR spectra were recorded using KBr pellets in the range 4000–400 cm^{-1} on a Shimadzu FTIR-8900 spectrophotometer. Thermogravimetric measurements were carried out from room temperature to 600°C in a flowing argon stream using a Seiko Exstar6000 TG/DTA6300 apparatus with a heating rate of 10°C/min.

Synthesis

All chemicals of p.a. grade were purchased from Shanghai Chemicals Company and used without further purification. For optimal synthesis, freshly prepared $Ni(OH)_{2-2x}(CO_3)_x \cdot yH_2O$ was necessary, obtained by dropwise addition of 6.0 cm^3 of 1 M Na_2CO_3 to a stirred aqueous solution of $NiSO_4 \cdot 6H_2O$ (1.31 g, 5.0 mmol) in 5.0 cm^3 of H_2O, followed by separation through centrifugation and washing with doubly-distilled water until no SO_4^{2-} anions were detectable. The pale-green precipitate was then added to an aqueous solution of maleic acid (0.58 g, 5.0 mmol) in 30 cm^3 of H_2O. The mixture was stirred vigorously until the precipitate was completely dissolved. The resulting green solution (pH = 5.70) was allowed to stand at room temperature and well-shaped green crystals grew by slow evaporation over two weeks. Yield: 85%. Anal. Calc. for $C_8H_{28}Ni_2O_{20}$ (%): C, 17.11; H, 5.02. Found: C, 17.35; H, 5.13. IR (cm^{-1}): 3209s (br), 2259vw, 1551s, 1418s (sh), 1339s (sh), 1234m (sh), 995m (sh), 847w, 746w, 673w, 608w (br: broad; sh: sharp).

X-ray Crystallography

A crystal of approximate dimensions 0.57 × 0.48 × 0.39 mm was selected under a polarizing microscope and glued to a fine glass fibre with epoxy cement. Unit cell parameters and the orientation matrix were determined by least-squares refinement using the setting angles of 25 centred reflections ($10 \leq 2\vartheta \leq 25°$) measured on a Bruker P4 diffractometer with graphite-monochromatized Mo Kα radiation ($\lambda = 0.71073$ Å). Intensity data were collected at 293 K using a ϑ–2ϑ scan technique up to a maximum 2ϑ value of 55°. Three standard reflections were monitored for crystal and instrument stability after every 97 data measurements. No significant variation was observed. Out of 2774 measured reflections ($h \rightarrow -9$ to 1, $k \rightarrow -9$ to 10, $l \rightarrow -12$ to 12), 2238 reflections were independent ($R_{int} = 0.0290$) and 1901 reflections with $I \geq 2\sigma(I)$ were considered as observed and used for the structure determination. The intensity data were corrected for Lorentz and polarization effects and empirical absorption. SHELXS-97 and SHELXL-97 programs were used for the structure determination and refinement [25, 26]. The structure was solved using direct methods. Subsequent difference Fourier syntheses enabled all heavier atoms to be located. After several cycles of refinement, the hydrogen atoms associated with C atoms were added geometrically and aqua hydrogen atoms were located from the successive difference Fourier syntheses. The hydrogen and non-hydrogen atoms were refined with isotropic and anisotropic displacement parameters, respectively. Final refinement based on full-matrix least-squares technique converged very smoothly to agreement factor

TABLE I Crystal data and structure refinement details

Compound	$[Ni(H_2O)_6][Ni(H_2\ O)_2(CC_4H_2O_4]\cdot 4H_2O$
Empirical formula	$C_8H_{28}Ni_2O_{20}$
Colour/shape	green/plate-like
Size (mm)	$0.57 \times 0.48 \times 0.39$
Formula weight	561.72
Temperature	293 K
Crystal system, space group	triclinic, $P\bar{1}$(no. 2)
a (Å)	7.186(1)
b (Å)	8.659(2)
c (Å)	9.613(2)
α (°)	113.34(2)
β (°)	95.95(1)
γ (°)	105.53(2)
Volume (Å^3), Z	514.1(2), 1
$D_{\text{calcd.}}$ (g cm^{-3})	1.814
μ (Mo Kα) (cm^{-1})	19.26
F(000)	292
Diffractometer	Bruker P4
Radiation	Graphite monochromated Mo Kα ($\lambda = 0.71073$ Å)
Scan type	$\vartheta - 2\vartheta$
No. of measured reflections	Total: 2774; unique: 2238 ($R_{\text{int}} = 0.0290$)
Correction	Semi-empirical based on psi-scan
Structure solution	Direct methods (SHELXL-97)[25]
Refinement	Full-matrix least-squares (SHELXL-97)[26]
Refinement method	Full-matrix least-squares on F^2
Treatment of H atoms	Diff. Map and refined isotropically
No. of observed reflections $[I \geq 2\sigma(I)]$	1901
No. of variables	189
Goodness-of-fit on F^2	1.028
Final R indices $[I \geq 2\sigma(I)]$	$R1 = 0.0285$, $wR2 = 0.0704$
R indices (all data)[a]	$R1 = 0.0360$, $wR2 = 0.0742$
A, B values in weighting scheme[b]	0.0374, 0.1275
Extinction coefficient	0.007(2)
Max. and min. peak in final diff. map	0.409 and −0.376 e/Å^3

[a] $wR2 = \left[\sum w(F_0^2 - F_c^2)^2 / \sum w(F_0^2)^2\right]^{1/2}$; [b] $w = \left[\sigma^2(F_0^2) + (AP)^2 + BP\right]^{-1}$ with $P = (F_0^2 + 2F_c^2)/3$.

$R1 = 0.0285$ and $wR2 = 0.0704$. Atomic scattering factors were supplied by the SHELX programs [25,26]. Crystal data, intensity collection and structure refinement details are summarized in Table I, the final atomic positional parameters, selected interatomic distances and bond angles are given in Tables II and III, respectively. Crystallographic data (excluding structure factors) reported in this article have been deposited with Cambridge Crystallographic Data Centre as supplementary publication no. CCDC 215215 ($C_8H_{28}Ni_2O_{20}$). Copies of the data can be obtained free of charge on application to CCDC, 12 Union Road, Cambridge CB2 1EZ, UK, (fax: (+44)1223-336-033; e-mail: deposit@ccdc.cam.ac.uk).

RESULTS AND DISCUSSION

Synthesis and IR Spectrum

$[Ni(H_2O)_6][Ni(H_2O)_2(C_4H_2O_4]\cdot 4H_2O$ resulted from reaction of freshly prepared $Ni(OH)_{2-2x}(CO_3)_x \cdot yH_2O$ with maleic acid in H_2O at room temperature. The IR spectrum indicated that water molecules causes an intense broad band at 3209 cm^{-1}. Strong

TABLE II Fractional atomic coordinates and equivalent isotropic displacement parameters (Å^2) for non-hydrogen atoms. U_{eq} is defined as one third of the trace of the orthogonalized U_{ij} tensor

Atom	x/a	y/b	z/c	U_{eq}
Ni(1)	0	0.5000	0	0.0186(1)
Ni(2)	0.5000	1	0.5000	0.0209(1)
O(1)	−0.1713(2)	0.4875(2)	−0.1892(2)	0.0253(3)
O(2)	−0.2570(3)	0.5188(2)	−0.4017(2)	0.0351(4)
O(3)	0.2645(3)	1.0453(2)	0.1275(2)	0.0347(4)
O(4)	0.1079(2)	0.7721(2)	0.0962(2)	0.0262(3)
O(5)	0.2358(2)	0.4675(2)	−0.1050(2)	0.0256(3)
O(6)	0.4856(3)	1.2017(2)	0.6982(2)	0.0272(3)
O(7)	0.5394(3)	1.1713(3)	0.3996(2)	0.0320(4)
O(8)	0.1978(2)	0.9278(2)	0.4173(2)	0.0273(4)
O(9)	0.1529(3)	0.2990(3)	0.5747(2)	0.0326(4)
O(10)	0.4081(3)	0.2559(2)	0.9871(2)	0.0309(4)
C(1)	−0.1332(3)	0.5585(3)	−0.2808(2)	0.0221(4)
C(2)	0.0659(3)	0.6897(3)	−0.2541(2)	0.0252(5)
C(3)	0.1928(3)	0.8190(3)	−0.1242(2)	0.0254(5)
C(4)	0.1851(3)	0.8810(3)	0.0434(2)	0.0231(4)

TABLE III Selected interatomic distances (Å) and bond angles (°)

Ni(1)–O(1)	2.035(1)	Ni(2)–O(6)$^{\#2}$	2.047(2)	C(1)–C(2)	1.491(3)
Ni(1)–O(1)$^{\#1}$	2.035(1)	Ni(2)–O(7)	2.044(2)	C(2)–C(3)	1.324(3)
Ni(1)–O(4)	2.038(2)	Ni(2)–O(7)$^{\#2}$	2.044(2)	C(3)–C(4)	1.495(3)
Ni(1)–O(4)$^{\#1}$	2.038(2)	Ni(2)–O(8)	2.064(2)	C(4)–O(3)	1.250(3)
Ni(1)–O(5)	2.087(2)	Ni(2)–O(8)$^{\#2}$	2.064(2)	C(4)–O(4)	1.270(3)
Ni(1)–O(5)$^{\#1}$	2.087(2)	C(1)–O(1)	1.272(2)		
Ni(1)–O(6)	2.047(2)	C(1)–O(2)	1.252(3)		
O(1)–Ni(1)–O(1)$^{\#1}$	180	O(4)–Ni(1)–O(5)$^{\#1}$	85.55(7)	O(6)$^{\#2}$–Ni(2)–O(8)	90.81(7)
O(1)–Ni(1)–O(4)	91.25(6)	O(4)$^{\#1}$–Ni(1)–O(5)	85.55(7)	O(6)$^{\#2}$–Ni(2)–O(8)$^{\#2}$	89.19(7)
O(1)–Ni(1)–O(4)$^{\#1}$	88.75(6)	O(4)$^{\#1}$–Ni(1)–O(5)$^{\#1}$	94.45(7)	O(7)–Ni(2)–O(7)$^{\#2}$	180
O(1)–Ni(1)–O(5)	94.30(6)	O(5)–Ni(1)–O(5)$^{\#1}$	180	O(7)–Ni(2)–O(8)	87.70(8)
O(1)–Ni(1)–O(5)$^{\#1}$	85.70(6)	O(6)–Ni(2)–O(6)$^{\#2}$	180	O(7)–Ni(2)–O(8)$^{\#2}$	92.30(8)
O(1)$^{\#1}$–Ni(1)–O(4)	88.75(6)	O(6)–Ni(2)–O(7)	88.45(8)	O(7)$^{\#2}$—Ni(2)–O(8)	92.30(8)
O(1)$^{\#1}$–Ni(1)–O(4)$^{\#1}$	91.25(6)	O(6)–Ni(2)–O(7)$^{\#2}$	91.55(8)	O(7)$^{\#2}$–Ni(2)–O(8)$^{\#2}$	87.70(8)
O(1)$^{\#1}$–Ni(1)–O(5)	85.70(6)	O(6)–Ni(2)–O(8)	89.19(7)	O(8)–Ni(2)–O(8)$^{\#2}$	180
O(1)$^{\#1}$–Ni(1)–O(5)$^{\#1}$	94.30(6)	O(6)–Ni(2)–O(8)$^{\#2}$	90.81(7)	O(1)–C(1)–O(2)	122.5(2)
O(4)–Ni(1)–O(4)$^{\#1}$	180	O(6)$^{\#2}$–Ni(2)–O(7)	91.55(8)	O(3)–C(4)–O(4)	122.8(2)
O(4)–Ni(1)–O(5)	94.45(7)	O(6)$^{\#2}$–Ni(2)–O(7)$^{\#2}$	88.45(8)		

Hydrogen bonding contacts

D–H$\cdots$A	D–H	H$\cdots$A	D$\cdots$A	D–H$\cdots$A
O(5)–H(5a)$\cdots$O(10)$^{\#3}$	0.91	1.86	2.745	163
O(5)–H(5b)$\cdots$O(9)$^{\#4}$	0.81	1.95	2.750	172
O(6)–H(6a)$\cdots$O(1)$^{\#5}$	0.80	1.94	2.726	171
O(6)–H(6b)$\cdots$O(10)$^{\#6}$	0.82	1.99	2.770	161
O(7)–H(7a)$\cdots$O(2)$^{\#5}$	0.84	1.92	2.711	157
O(7)–H(7b)$\cdots$O(3)	0.67	2.08	2.729	166
O(8)–H(8a)$\cdots$O(9)$^{\#7}$	0.73	2.11	2.779	154
O(8)–H(8b)$\cdots$O(4)	0.84	1.92	2.754	173
O(9)–H(9a)$\cdots$O(8)$^{\#8}$	0.81	2.33	3.087	156
O(9)–H(9b)$\cdots$O(2)$^{\#1}$	0.79	1.96	2.742	172
O(10)–H(10a)$\cdots$O(5)$^{\#9}$	0.78	2.10	2.819	153
O(10)–H(10b)$\cdots$O(3)$^{\#10}$	0.81	1.94	2.728	166

Symmetry transformations used to generate equivalent atoms: #1 $= -x, -y+1, -z$; #2 $= -x+1, -y+2, -z+1$; #3 $= -x+1, -y+1, -z+1$; #4 $= x, y, z-1$; #5 $= x+1, y+1, z+1$; #6 $= x, y+1, z$; #7 $= -x, -y+1, -z+1$; #8 $= x, y-1, z$; #9 $= x, y, z+1$; #10 $= x, y-1, z+1$.

absorption at 1551 cm^{-1} could be assigned to the asymmetric COO stretching vibration while the strong, sharp absorption at 1418 cm^{-1} is attributed to the symmetric COO stretching vibration.

Thermal Analyses

When heated in flowing argon, the complex decomposes in two steps. Weight loss of 38.3% at 60–235°C corresponds to the calculated value of 38.5% for liberation of twelve moles of H_2O molecules per formula unit. Over the temperature range 235–410°C, the compound loses additional weight (35.4%), in agreement with a calculated value of 34.9% for sublimation of two moles of maleic anhydride ($C_4H_2O_3$). The pale-green residue collected at 600°C was identified as NiO by powder X-ray diffraction.

Structural Description

As shown in Fig. 1, the structure of the complex consists of $[Ni(H_2O)_6]^{2+}$ cations, $[Ni(H_2O)_2(C_4H_2O_4)]^{2-}$ anions and lattice H_2O molecules. Cations and anions are centred at crystallographic 1*f* and 1*c* positions, respectively. Within the anions (Fig. 2), the Ni atoms are coordinated by two H_2O molecules and two chelating maleate anions to complete a slightly elongated octahedron with the four carboxylate O atoms in the equatorial plane and two aqua O atoms at apical positions. Being practically identical within experimental error, the four equatorial Ni–O bond lengths average 2.037(2) Å, slightly shorter than those (2.087(2) Å) of the aqua O atoms. Ni atoms in the cations (Fig. 2) are each surrounded by six aqua O atoms at the corners of a tri-

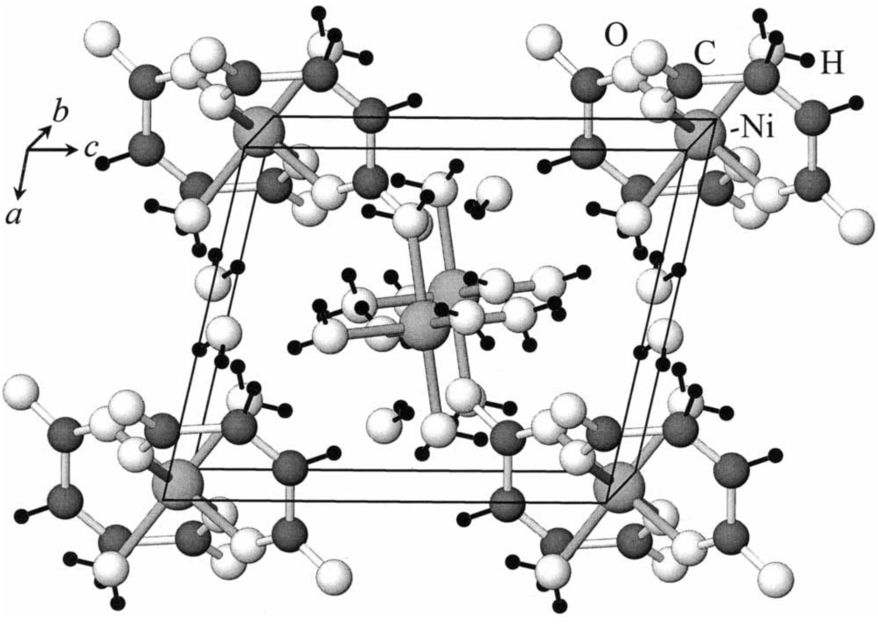

FIGURE 1 Crystal structure of $[Ni(H_2O)_6][Ni(H_2O)_2(C_4H_2O_4)]\cdot 4H_2O$.

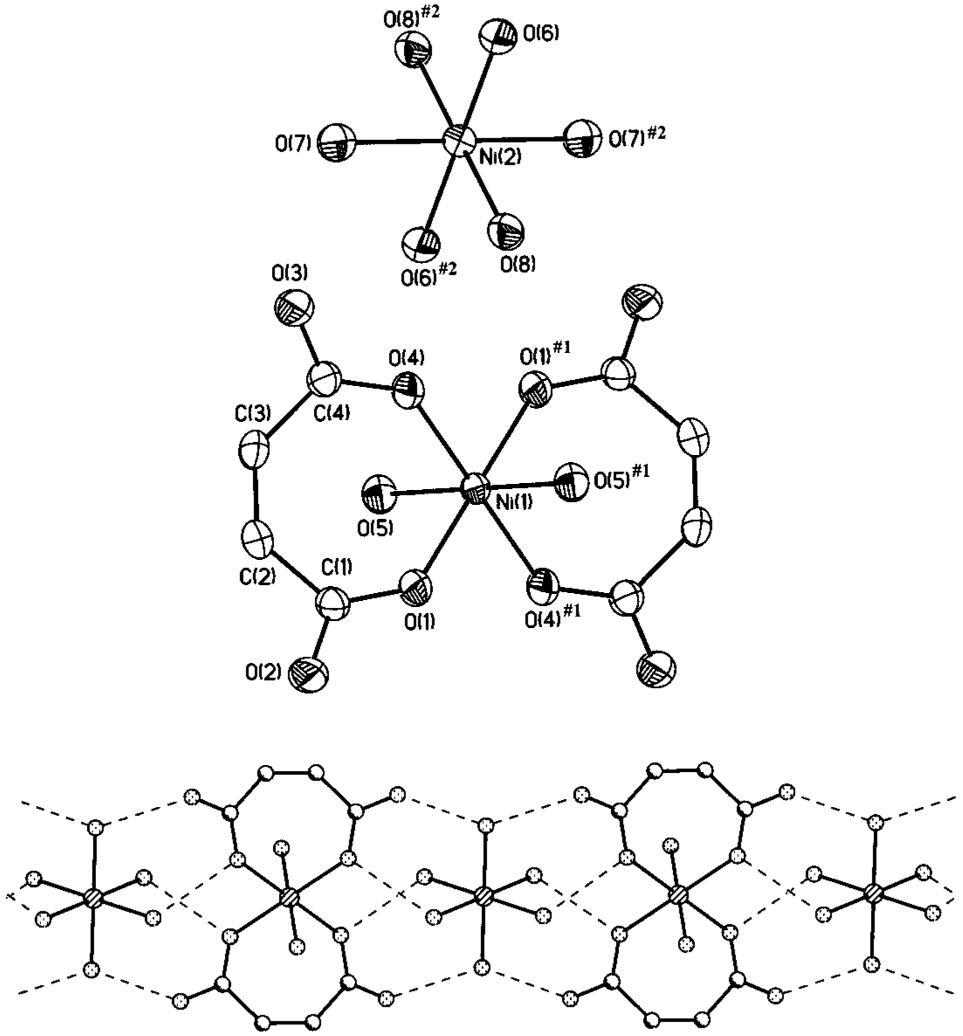

FIGURE 2 (Top) ORTEP view of $[Ni(H_2O)_6]^{2+}$ and $[Ni(H_2O)_2(C_4H_2O_4)]^{2-}$ with the atom numbering scheme. Thermal ellipsoids are drawn at 60% probability level. (Bottom) 1D chain generated from the $[Ni(H_2O)_6]^{2+}$ and $[Ni(H_2O)_2(C_4H_2O_4)]^{2-}$ via hydrogen bonds.

vially distorted octahedron with Ni–O bond distances in the range 2.044–2.064 Å (Table III). The *cisoid* O–Ni–O angles in both cation and anion exhibit slight deviations from 90°, while the *transoid* O–Ni–O angles are exactly equal to 180° owing to crystallographically imposed symmetry.

The complex offers a rare example in which the maleate anions function as a bidentate ligand to chelate Ni atom to form a seven-membered ring. Terminal carboxylate groups are rotated by 35.7(1) and 36.3(2)°, respectively, away from the plane defined by the four skeleton carbon atoms. As expected, C–O bond lengths for coordinating O atoms (1.271(1) Å) are significantly longer than those (1.251(1) Å) of the uncoordinated carboxyl groups.

Of the three crystallographically distinct H_2O molecules in the cations, (O(7)) donates hydrogen atoms to the uncoordinated carboxyl O atoms [$O(2)^{\#5}$, O(3)] to form hydrogen bonds with $d(O \cdots O) = 2.711, 2.729\,Å$; the other two, O(6) and O(8), are hydrogen bonded to the coordinating carboxylate $O(1)^{\#1}$ and O(4) atoms with $d(O \cdots O) = 2.726, 2.754\,Å$. Though these hydrogen bonds, cations and anions are linked to generate 1D chains (Fig. 2) extending along the [111] direction. The chains are hexagonally arranged and connected by lattice H_2O molecules, which serve as hydrogen bonding donors and acceptors (Table III).

Acknowledgments

The project was financially supported by the National Natural Science Foundation (20072022), the Excellent Young Teachers Program of MOE, P.R. China (C982302), the Zhejiang Provincial Natural Science Foundation (RC99034), Ningbo Municipal Key Doctor's Funds (2003A61014) and the Ningbo Municipal Natural Science Foundation (01J20130-1). The authors are grateful to Mr. Jian-Li Lin for X-ray diffraction data collection.

References

[1] S.S.-Y. Chui, S.M.-F. Lo, J.P.H. Charmant, A.G. Orpen and I.D. Williams, *Science*, **283**, 1148 (1999).
[2] H. Li, M. Eddaoudl, M. O'Keeffe and O.M. Yaghi, *Nature*, **402**, 276 (1999).
[3] C. Serre, F. Millange, C. Thouvenot, M. Noguès, G. Marsolier, D. Louër and G. Fèrey, *J. Am. Chem. Soc.*, **124**, 13519 (2002).
[4] P.M. Forster and A.K. Cheetham, *Angew. Chem. Int. Ed.*, **41**, 547 (2002).
[5] E.W. Lee, Y.J. Kim and D.-Y. Jung, *Inorg. Chem.*, **41**, 501 (2002).
[6] C. Livage, C. Egger and G. Férey, *Chem. Mater.*, **13**, 410 (2001).
[7] C. Livage, C. Egger and G. Férey, *C.R. Acad. Sci Ser. Iic*: *Chim.*, **4**, 221 (2001).
[8] Y.J. Kim, E.W. Lee and D.-Y Jung, *Chem. Mater.*, **13**, 2684 (2001).
[9] Y.J. Kim and D.-Y. Jung, *Inorg. Chem.*, **39**, 1470 (2000).
[10] Y.J. Kim and D.-Y. Jung, *Bull. Korean Chem. Soc.*, **21**, 656 (2000).
[11] C. Livage, C. Egger and G. Férey, *Chem. Mater.*, **11**, 1546 (1999).
[12] Y.J. Kim and D.-Y. Jung, *Bull. Korean Chem. Soc.*, **20**, 827 (1999).
[13] C. Livage, C. Egger and G. Férey, *J. Mater. Chem.*, **8**, 2743 (1998).
[14] A. Michaelides, V. Kiritsis, S. Skoulika and A. Aubry, *Angew. Chem. Int. Ed. Engl.*, **32**, 1495 (1993).
[15] Y.-Q. Zheng and Z.-P. Kong, *J. Solid State Chem.*, **166**, 279 (2000), and references therein.
[16] A. Sequeira, H. Rajagopal and M.P. Gupta, *Acta Cryst.*, **C48**, 1192 (1992).
[17] G. Smith, D.S. Sagatys, C. Dahlgren, D.E. Lynch, R.C. Bott, K.A. Byriel and C.H.L. Kennard, *Z. Kristallogr.*, **210**, 44 (1995).
[18] N.P. Porollo, Z.G. Aliev, G.I. Dzhardimalieva, I.N. Ivleva, I.E. Uflyand, A.D. Pomogailo and N.S. Ovanesyan, *Russia Chem. Bull.*, **46**, 362 (1997).
[19] C.-G. Zhang, D. Wu, K.-B. Yu and D.-Y. Yan, *J. Coord. Chem.*, **51**, 93 (2000).
[20] Z. Shi, L.-R. Zhang, S. Gao, G.-Y. Yang, J. Hua, L. Gao and S.-H. Feng, *Inorg. Chem.*, **39**, 1990 (2000).
[21] C.-G. Zhang, J.-X. Zhou, B. Liu and Z.-F. Ma, *J. Shanghai Jiaotong University*, **34**, 550 (2000).
[22] Y.-Q. Zheng and Z.-P. Kong, *J. Coord. Chem.*, **55**, 1241 (2001).
[23] Y.-Q. Zheng, J. Sun and J.-L. Lin, *Z. Anorg. Allg. Chem.*, **628**, 1397 (2002).
[24] Y.-Q. Zheng, J.-L. Lin, Z.-P. Kong and B.-Y. Chen, *J. Chem. Crystallogr.*, **32**, 399 (2002).
[25] G.M. Sheldrick, *SHELXS-97*, *Programm zur Lösung von Kristallsturkturen*, (University of Göttingen, Göttingen, 1997).
[26] G.M. Sheldrick, *SHELXS-97*, *Programm zur Verfeinerung von Kristallstrukturen*, (University of Göttingen, Göttingen, 1997).