Acta Crystallographica Section E
Structure Reports
Online

ISSN 1600-5368

Dipotassium diaquabis(malonato-κ^2O,O')-nickelate(II) dihydrate

Xunwen Xiao,[a,b] Wei Xu,[a] Yan Li,[c] Bin Zhang[a]* and Daoben Zhu[a]

[a]106 group, Organic Solid Laboratory, Institute of Chemistry, The Chinese Academy of Science, Zhongguacun, Beijing 100080, People's Republic of China, [b]Graduate School of Chinese Academy of Science, Beijing 100039, People's Republic of China, and [c]Centre for Physiochemical Analysis and Measurement, Institute of Chemistry, The Chinese Academy of Science, Zhongguacun, Beijing 100080, People's Republic of China

Correspondence e-mail: zhangbin@iccas.ac.cn

Key indicators

Single-crystal X-ray study
T = 293 K
Mean σ(C–C) = 0.002 Å
R factor = 0.019
wR factor = 0.051
Data-to-parameter ratio = 12.8

For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

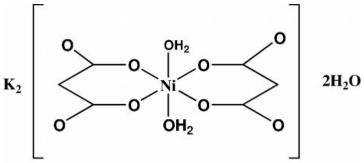
© 2004 International Union of Crystallography
Printed in Great Britain – all rights reserved

Received 17 November 2003
Accepted 1 December 2003
Online 12 December 2003

The anion of the title compound, $K_2[Ni(C_3H_2O_4)_2(H_2O)_2]\cdot 2H_2O$, possesses C_{2h} symmetry. The Ni atom is coordinated by two water molecules and two malonate ligands, with an elongated octahedral environment. Hydrogen bonds between the complex anion and the water molecules of crystallization produce an NaCl-type framework.

Comment

In recent years, significant research efforts have concentrated on carboxylate ligands, due to the fact that they are good candidates for the investigation of exchange coupling interactions between adjacent metal ions. Among the different carboxylates studied, the versatility of the malonate dianion as a ligand has attracted interest for two reasons. Firstly, there are two carboxylate groups which can adopt coordination modes *syn–syn*, *syn–anti* or *anti–anti*, and secondly, it is one of the best building blocks to form molecular magnets (Rodríguez-Martín *et al.*, 2002). It has been possible to obtain new molecular magnets, even porous magnets, with the flexible malonate dianion (Sain *et al.*, 2003; Delgado *et al.*, 2003; Harlow & Pfluger, 1973).



(I)

The title compound, (I), is an isomer of $2\,K^+[Zn(mal)_2(H_2O)]^{2-}\cdot H_2O$ (Kiosse *et al.*, 1989), and the anion possesses C_{2h} symmetry (Fig. 1). The Ni atom, which is coordinated by two malonate dianions and two water molecules, has an elongated octahedral environment. All the Ni—O bond lengths are in the same range as reported for other Ni^{II}—O compounds (Shi *et al.*, 1999). There are four Ni—O(malonate) bonds of 2.0195 (9) Å lying in a plane, and two Ni—O(water) bonds of 2.082 (1) Å in the *trans* axial position.

In the crystal structure there are two hydrogen bonds (Table 2 and Fig. 2), one involving the coordinated water molecule and the malonate dianions, and the other involving the water molecule of crystallization and the malonate dianions. The hydrogen bonds form an NaCl-type framework.

Experimental

$Ni(NO)_3\cdot 6H_2O$ (290 mg), KOH (112 mg) and $HOOCCH_2COOH$ (104 mg) were mixed in a solution of CH_3OH/H_2O. After 10 d, the title compound was obtained in a yield of 93% (193 mg). Analysis

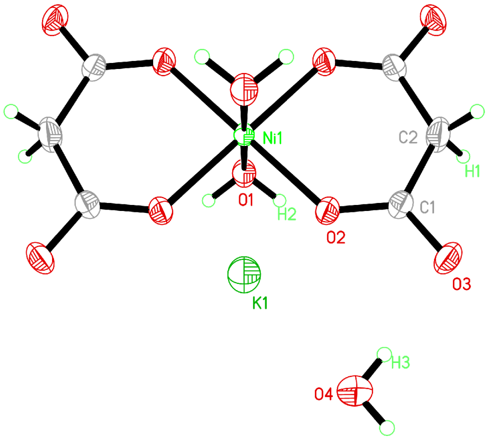


Figure 1
View of the title compound, (I), showing the atom-labelling scheme and displacement ellipsoids at the 50% probability. One K^+ ion and one water molecule of crystallization, symmetry-equivalent to those shown here, have been omitted.

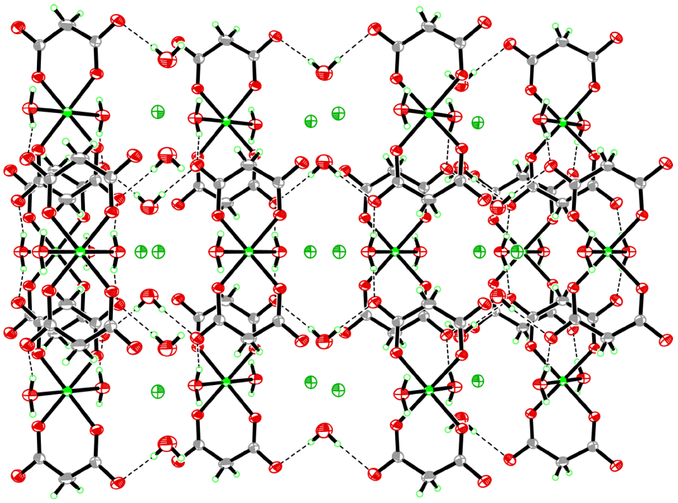


Figure 2
The crystal packing of (I), viewed along the *a* axis.

calculated for $C_6H_{12}K_2NiO_{12}$: C 17.43, H 2.90%; found: C 17.36, H 2.92%. IR(KBr)/ cm^{-1}: 3344 (*s*), 3261 (*s*), 1687 (*m*), 1585 (*s*), 1269 (*s*), 958 (*m*)

Crystal data

$K_2[Ni(C_3H_2O_4)_2(H_2O)_2]\cdot 2H_2O$
$M_r = 413.05$
Monoclinic, $C2/m$
$a = 9.3973$ (5) Å
$b = 10.8760$ (5) Å
$c = 7.6283$ (4) Å
$\beta = 115.026$ (2)°
$V = 706.45$ (6) Å^3
$Z = 2$

$D_x = 1.942$ Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 3628 reflections
$\theta = 3.0$–27.4°
$\mu = 2.02$ mm^{-1}
$T = 293$ (2) K
Block, blue
0.30 × 0.20 × 0.20 mm

Data collection

Rigaku R-AXIS RAPID IP diffractometer
φ scans
Absorption correction: multi-scan (*ABSCOR*; Higashi, 1995)
$T_{min} = 0.622$, $T_{max} = 0.668$
3183 measured reflections

859 independent reflections
799 reflections with $I > 2\sigma(I)$
$R_{int} = 0.019$
$\theta_{max} = 27.5°$
$h = -11 \rightarrow 12$
$k = -14 \rightarrow 14$
$l = -9 \rightarrow 8$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.019$
$wR(F^2) = 0.051$
$S = 1.04$
859 reflections
67 parameters
All H-atom parameters refined

$w = 1/[\sigma^2(F_o^2) + (0.0306P)^2 + 0.266P]$
where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.34$ e Å^{-3}
$\Delta\rho_{min} = -0.22$ e Å^{-3}
Extinction correction: *SHELXL97*
Extinction coefficient: 0.0071 (11)

Table 1
Selected geometric parameters (Å, °).

Ni1—O2	2.0195 (9)	O3—C1	1.2548 (16)
Ni1—O1	2.0818 (14)	C1—C2	1.5123 (16)
O2—C1	1.2540 (16)		
O2—Ni1—O2^i	180	O2—C1—O3	122.57 (13)
O2—Ni1—O2^{ii}	89.85 (5)	O2—C1—C2	122.36 (13)
O2^i—Ni1—O2^{ii}	90.15 (5)	O3—C1—C2	115.07 (13)
O2—Ni1—O1	92.77 (4)	C1^{ii}—C2—C1	123.65 (19)
O2^i—Ni1—O1	87.23 (4)		

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

Table 2
Hydrogen-bonding geometry (Å, °).

D—H···A	D—H	H···A	D···A	D—H···A
O1—H2···O3^{iii}	0.86 (2)	1.85 (2)	2.6932 (14)	166 (2)
O4—H3···O3	0.79 (2)	2.03 (2)	2.8161 (15)	175 (2)

Symmetry code: (iii) $\frac{1}{2} - x, \frac{1}{2} - y, -z$.

The refined C—H distance is 1.03 Å.

Data collection: *RAPID-AUTO* (Rigaku, 2001); cell refinement: *RAPID-AUTO*; data reduction: *RAPID-AUTO*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *XP* (Bruker, 1998); software used to prepare material for publication: *SHELXTL* (Bruker, 1998).

This work was supported by The Chinese Academy of Sciences (No. CMS-CX200314).

References

Bruker (1998). *XP* and *SHEXLTL*. Bruker AXS Inc., Madison, Wisconsin, USA.
Delgado, F. S., Sanchiz, J., Ruiz-Pérez, C., Lloret, F. & Julve, M. (2003). *Inorg. Chem.* **42**, 5938–5948.
Harlow, R. L. & Pfluger, C. E. (1973). *J. Coord. Chem.* **2**, 289–294.
Higashi, T. (1995). *ABSCOR*. Rigaku Corporation, Tokyo, Japan.
Kiosse, G. A., Filippova, I. G., Leontev, A. Y., Popovich, G. A., Malinovski, T. I., Yabokon, Y. V. & Gerbeleu, N. V. (1989). *Dokl. Akad. Nauk SSSR*, **304**, 607–610.
Rigaku (2001). *RAPID-AUTO*. Rigaku Corporation, Tokyo, Japan.
Rodríguez-Martín, Y., Hernández-Molina, M., Delgado, F. S., Pasán, J., Ruiz-Pérez, C., Sanchiz, J., Lloret, F. & Julve, M. (2002). *CrystEngComm*, **4**, 522–535.
Sain S., Maji T. K., Mostafa G., Lu T. H. & Chaudhuri N. R. (2003). *Inorg. Chim. Acta*, **351**, 12–20.
Sheldrick, G. M. (1997). *SHELXS97* and *SHELXL97*. University of Göttingen, Germany.
Shi, Z., Zhang, L. R., Zhu, G. S., Yang, G. Y., Hua, J., Ding, H. & Feng, S. H. (1999). *Chem. Mater.* **11**, 3565–3570.

Acta Cryst. (2004). E**60**, m48–m49 Xunwen Xiao *et al.* • $K_2[Ni(C_3H_2O_4)_2(H_2O)_2]\cdot 2H_2O$ **m49**