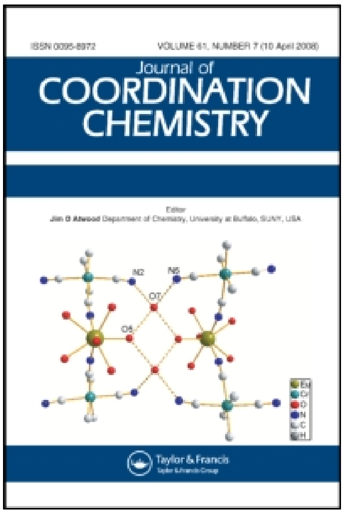

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Synthesis and crystal structure of $[Zn(H_2O)_4(C_4H_2O_4)]\cdot H_2O$

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SYNTHESIS AND CRYSTAL STRUCTURE OF $[Zn(H_2O)_4(C_4H_2O_4)] \cdot H_2O$

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Reaction of freshly prepared $Zn(OH)_{2-2x}(CO_3)_x \cdot yH_2O$ with fumaric acid in CH_3OH/H_2O (1:1 v/v) at room temperature afforded $[Zn(H_2O)_4(C_4H_2O_4)] \cdot H_2O$. An X-ray structure determination indicates that the title compound consists of hydrogen bonded H_2O molecules and polymeric chains, $^1_\infty[Zn(H_2O)_4(C_4H_2O_4)_{2/2}]$, resulting from Zn atoms bridged by *bis*-monodentate fumarate anions. The Zn atoms are octahedrally coordinated by six oxygen atoms of four aqua ligands and two different fumarate anions with the carboxylato oxygen atoms in *cis* positions. Supramolecular assembly of the polymeric chains is due to extensive hydrogen bonding interactions. Thermal analysis shows that $[Zn(H_2O)_4(C_4H_2O_4)] \cdot H_2O$ decomposes in two steps upon heating; initial endothermic dehydration over 45–80°C is followed by decomposition of anhydrous zinc fumarate at 360–825°C.

Keywords: Zn(II); Fumarato complex; Synthesis; Crystal structure; Thermal analysis

INTRODUCTION

Over the past decade, enormous effort has been dedicated to the rational design of supramolecular polymeric architectures, and exploitation of carboxylate anions as linkers is of growing interest in construction of supramolecular systems [1]. In recent years, interest in our laboratory has focussed on aliphatic dicarboxylate ligands [2] since the flexibility and conformational freedom of such multifunctional ligands promise great possibilities for generating novel metal–organic frameworks [2–9]. In comparison to the succinato ligand, the fumarato ligand displays less conformational freedom owing to incorporation of the C=C bond. Hitherto, however, fewer coordination polymers have been synthesized using the fumarato ligands [10–14]. Thus, we report the synthesis and structural characterization of a new polymeric zinc fumarate, $[Zn(H_2O)_4(C_4H_2O_4)] \cdot H_2O$.

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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. TG–DTA curves were recorded from room temperature to 825°C on a preweighed sample in an 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 the Shanghai Chemical Company and used without further purification. Addition of 6.0 cm^3 of 1 M Na_2CO_3 to an aqueous solution of 0.72 g (2.50 mmol) of $ZnSO_4 \cdot 7H_2O$ in 4.0 cm^3 of H_2O yielded a white precipitate of $Zn(OH)_{2-2x}(CO_3)_x \cdot yH_2O$, which was centrifuged and washed with doubly distilled water until there were no detectable SO_4^{2-} and then added to a stirred aqueous methanolic solution of 0.27 g (2.50 mmol) of fumaric acid in 50 cm^3 of CH_3OH/H_2O (1:1 v/v). Insoluble solids were filtered off. The resulting colourless filtrate (pH = 5.78) was allowed to stand at room temperature and colourless crystals grew overnight. Yield: 52%. Anal. Calc. for $C_4H_{12}O_9Zn$(%): C, 17.83; H, 4.49. Found: C, 17.98; H, 4.62. IR (cm^{-1}): 3061 s (broad), 2742 vw, 2650 w, 2538 w, 1728 s (shoulder), 1691 vs (sharp), 1416 vs (sharp), 1310 vs (sharp), 1202 vs (sharp), 1176 s (sharp), 908 s, 893 s, 802 m (sharp), 636 s (sharp), 582 w (sharp), 544 w (sharp), 442 w.

X-ray Crystallography

A crystal of approximate dimensions 0.44 × 0.22 × 0.22 mm was selected under a polarizing microscope and glued to a 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 the $\vartheta - 2\vartheta$ scan technique up to a maximum 2ϑ value of 55°. The standard reflections were monitored for crystal and instrument stability after every 97 data measurements. No significant variation was observed. Of 1520 measured reflections (h −1 to 6, k −16 to 1, l −17 to 17), 1062 reflections were independent ($R_{int} = 0.0246$) and 1026 reflections with $I \geq 2\sigma(I)$ were considered to be observed and used for the structure determination. The intensity data were corrected for Lorentz and polarization effects and absorption. SHELXS-97 and SHELXL-97 programs [15,16] were used for the structure determination and refinement. The structure was solved using direct methods. Subsequent difference Fourier syntheses enabled all heavy atoms to be located. After several cycles of refinement, hydrogen atoms were located from 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 techniques converged very smoothly to agreement factors $R1 = 0.0235$ and $wR2 = 0.0637$. Atomic scattering factors were supplied by the SHELX programs [15,16]. Crystal data, collection and structure refinement details are summarized in Table I. Final atomic positional parameters, selected interatomic distances and bond angles are given in Tables II and III,

TABLE I Crystal data and structure refinement details

Compound	$[Zn(H_2O)_4(C_4H_2O_4)] \cdot H_2O$
Empirical formula	$C_4H_{12}O_9Zn$
Colour/shape	colourless/lump-like
Size (mm)	$0.44 \times 0.22 \times 0.22$
Formula weight	269.51
Temperature (K)	293
Crystal system, Space group	monoclinic, $C2/c$
a (Å)	5.294(1)
b (Å)	13.196(3)
c (Å)	13.294(3)
β (°)	95.22(3)
Volume (Å^3), Z	924.9(3), 4
D_{calcd} (g cm^{-3})	1.936
μ(Mo Kα) (cm^{-1})	26.84
F(000)	552
Diffractometer	Bruker P4
Radiation	Graphite monochromated Mo Kα ($\lambda = 0.71073$ Å)
Scan type	$\vartheta - 2\vartheta$
No. of measured reflections	Total: 1520; unique: 1062 ($R_{int} = 0.0246$)
Correction	Semi-empirical based on ψ-scan
Structure solution	Direct methods (SHELXS-97)
Refinement	Full-matrix least-squares (SHELXL-97)
Refinement method	Full-matrix least-squares on F^2
Treatment of H atoms	Diff. map and refined isotropically
No. of observed reflections [$I \geq 2\sigma(I)$]	1026
No. of variables	90
Goodness-of-fit on F^2	1.238
Final R indices [$I \geq 2\sigma(I)$]^a	$R1 = 0.0235$, $wR2 = 0.0637$
R indices (all data)^a	$R1 = 0.0242$, $wR2 = 0.0641$
A, B values in weighting scheme^b	0.0249, 0.6811
Extinction coefficient	0.018(1)
Max. and min. peak in final diff. map	0.687, -0.524 e$^-$/Å^3

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

TABLE II Fractional atomic coordinates and equivalent isotropic displacement parameters (Å^2) for non-hydrogen atoms

Atom	x/a	y/b	z/c	U_{eq}
Zn	0	0.48153(2)	1/4	0.0197(1)
O(1)	$-0.2944(3)$	0.4743(1)	0.3421(1)	0.0314(4)
O(2)	0.2086(3)	0.3712(1)	0.3405(1)	0.0248(3)
O(3)	0.1675(3)	0.5907(1)	0.3486(1)	0.0271(3)
O(4)	$-0.1084(3)$	0.6214(1)	0.4624(1)	0.0288(3)
O(5)	1/2	0.7030(2)	1/4	0.0294(4)
C(1)	0.0977(3)	0.6356(1)	0.4262(1)	0.0211(3)
C(2)	0.2843(4)	0.7108(1)	0.4735(1)	0.0230(4)

U_{eq} is defined as one third of the trace of the orthogonalized U_{ij} tensor.

respectively. Crystallographic data have been deposited with Cambridge Crystallographic Data Centre as supplementary publication CCDC 214526 ($C_4H_{12}O_9Zn$). 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).

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

Zn–O(1)	2.070(2)	Zn–O(3)	2.090(1)	C(1)–O(4)	1.246(2)
Zn–O(1)$^{\#1}$	2.070(2)	Zn–O(3)$^{\#1}$	2.090(1)	C(1)–C(2)	1.497(2)
Zn–O(2)	2.132(1)	C(1)–O(3)	1.274(2)	C(2)–C(2)$^{\#2}$	1.322(4)
Zn–O(2)$^{\#1}$	2.132(1)				
O(1)–Zn–O(1)$^{\#1}$	174.69(9)	O(1)$^{\#1}$–Zn–O(3)	95.94(6)	O(3)$^{\#1}$–Zn–O(2)	173.26(5)
O(1)–Zn–O(2)	90.79(6)	O(1)$^{\#1}$–Zn–O(3)$^{\#1}$	87.73(6)	O(3)$^{\#1}$–Zn–O(2)$^{\#1}$	87.05(6)
O(1)–Zn–O(2)$^{\#1}$	85.58(6)	O(2)$^{\#1}$–Zn–O(2)	93.85(8)	C(2)$^{\#2}$–C(2)–C(1)	122.7(2)
O(1)–Zn–O(3)	87.73(6)	O(3)–Zn–O(2)	87.05(6)	O(3)–C(1)–C(2)	114.8(2)
O(1)–Zn–O(3)$^{\#1}$	95.94(6)	O(3)–Zn–O(2)$^{\#1}$	173.26(5)	O(4)–C(1)–O(3)	124.9(2)
O(1)$^{\#1}$–Zn–O(2)	85.58(6)	O(3)–Zn–O(2)$^{\#1}$	92.84(9)	O(4)–C(1)–C(2)	120.3(2)
O(1)$^{\#1}$–Zn–O(2)$^{\#1}$	90.79(6)				
Hydrogen bonding contacts					
D–H···A	D–H	H···A	D···A	D–H···A	
O(1)–H(1a)···O(4)	0.71	1.99	2.648	155	
O(1)–H(1b)···O(2)$^{\#3}$	0.79	2.17	2.960	174	
O(2)–H(2a)···O(4)$^{\#4}$	0.83	1.89	2.722	172	
O(2)–H(2b)···O(5)$^{\#5}$	0.76	2.00	2.712	162	
O(5)–H(5)···O(3)$^{\#6}$	0.69	2.04	2.726	175	

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

RESULTS AND DISCUSSION

IR spectra of the complex indicate the characteristic absorption of H_2O molecules at 3061 and 1728 cm^{-1}. Two strong, sharp bands at 1416 and 1310 cm^{-1} can be attributed to asymmetric and symmetric $-CO_2$ stretching vibrations, respectively. The difference of 106 cm^{-1} is remarkably less than values ($\Delta \gg 160\,\text{cm}^{-1}$) expected for monodentate carboxylate groups, but is similar to those ($\Delta \leq 160\,\text{cm}^{-1}$) of the bidentate carboxylate groups [17]. This may be because the uncoordinating carboxylate O atoms are involved in the strong intra-chain hydrogen bonds (see below). Additionally, the strong sharp absorption at 1691 cm^{-1} confirms the presence of C=C bonds.

Upon slight heating, the sample undergoes dehydration, endothermic dehydration ending at 80°C with a mass loss of 32.9% corresponding well to a value of 33.39% for five mols of H_2O molecules per formula unit. The anhydrous intermediate "$Zn(C_4H_2O_4)$" is stable up to 360°C. With further heating, a sharp endothermic decomposition is followed by gradual mass loss of 36.2% up to 825°C, in good agreement with the calculated value of 36.36% for liberation of one mol of fumaric anhydride $C_4H_2O_3$. The product is ZnO.

The compound consists of lattice H_2O molecules and polymeric chains formulated as ${}^{1}_{\infty}[Zn(H_2O)_4(C_4H_2O_4)_{2/2}]$ (Fig. 1). The Zn atoms are octahedrally coordinated by six oxygen atoms of four aqua ligands and two *bis*-monodentate fumarate anions with the carboxylate oxygen atoms in *cis* positions. Zn–O bond distances vary from 2.070 to 2.132 Å and the cisoid and transoid O–Zn–O bond angles fall in the regions 85.58–95.94° and 173.26–174.69°, respectively, suggesting significant distortion of coordination geometry from that of an ideal octahedron. The fumarate anions, with expected bond distances and angles [18–20] are centred at crystallographic 4*d* positions and form bis-monodentate bridges between Zn atoms to generate 1D zigzag chains, ${}^{1}_{\infty}[Zn(H_2O)_4(C_4H_2O_4)_{2/2}]$, extending along [101]. Of two crystallographi-

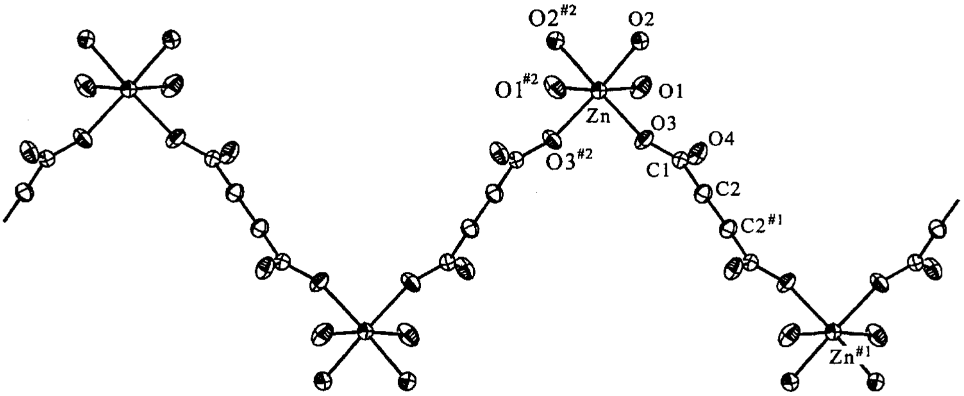

FIGURE 1 A fragment of a polymeric $^{1}_{\infty}[Zn(H_2O)_4(C_4H_2O_4)]$ chain. Thermal ellipsoids are drawn at the 50% probability level.

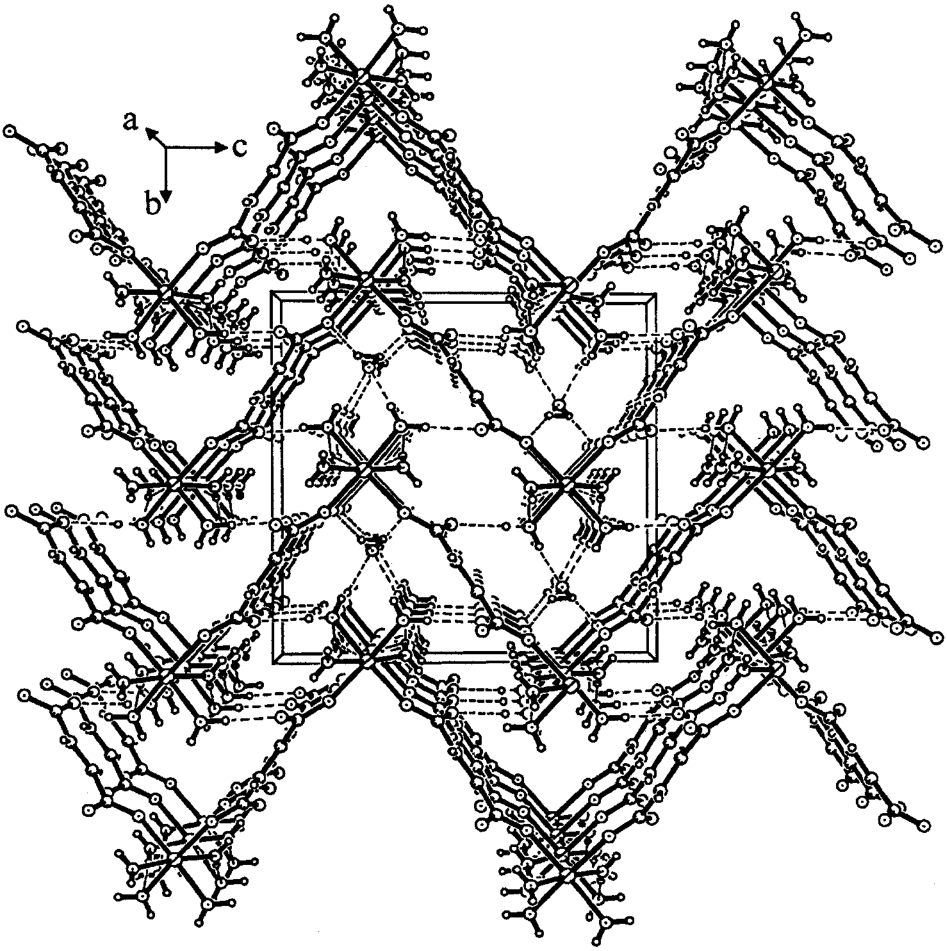

FIGURE 2 Perspective view of the crystal structure of $[Zn(H_2O)_4(C_4H_2O_4)] \cdot H_2O$ with the hydrogen bonds indicated by dashed lines (hydrogen atoms omitted for clarity).

cally distinct aqua ligands, one forms a hydrogen bond to the lattice H_2O molecule with $d[\mathrm{O}(2)\cdots\mathrm{O}(5)^{\#5}] = 2.712\,\text{Å}$ and $\angle[\mathrm{O}(2)\text{–}\mathrm{H}\cdots\mathrm{O}(5)^{\#5}] = 162^\circ$ and an inter-chain hydrogen bond to a non-coordinating carboxylate $\mathrm{O}(4)^{\#4}$ atom with $d[\mathrm{O}(2)\cdots\mathrm{O}(4)^{\#4}] = 2.722\,\text{Å}$ and $\angle[\mathrm{O}(2)\text{–}\mathrm{H}\cdots\mathrm{O}(4)^{\#4}] = 172^\circ$. The other is involved in an inter-chain hydrogen bond to the former aqua oxygen $\mathrm{O}(2)^{\#3}$ atom with $d[\mathrm{O}(1)\cdots\mathrm{O}(2)^{\#3}] = 2.960\,\text{Å}$ and $3\angle[\mathrm{O}(1)\text{–}\mathrm{H}\cdots\mathrm{O}(2)^{\#3}] = 174^\circ$ and a relatively strong intra-chain hydrogen bond to a non-coordinating carboxylate O(4) with $d[\mathrm{O}(1)\cdots\mathrm{O}(4)] = 2.648\,\text{Å}$ and $\angle[\mathrm{O}(1)\text{–}\mathrm{H}\cdots\mathrm{O}(4)] = 155^\circ$. The lattice H_2O molecule, in turn, is hydrogen bonded to a coordinating carboxylato $\mathrm{O}(3)^{\#6}$ atom with $d[\mathrm{O}(5)\cdots\mathrm{O}(3)^{\#6}] = 2.726\,\text{Å}$ and $\angle[\mathrm{O}(5)\text{–}\mathrm{H}\cdots\mathrm{O}(3)^{\#6}] = 175^\circ$. The extensive hydrogen bonding interactions play an important role in the supramolecular assembly of the polymeric chains into a 3D network (Fig. 2).

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