Acta Crystallographica Section E
Structure Reports
Online

ISSN 1600-5368

Poly[dimethylammonium [tris(μ_2-formato-κ^2O:O')cadmate(II)]]

Shan Gao[a] and Seik Weng Ng[b]*

[a]College of Chemistry and Materials Science, Heilongjiang University, Harbin 150080, People's Republic of China, and [b]Department of Chemistry, University of Malaya, 50603 Kuala Lumpur, Malaysia
Correspondence e-mail: seikweng@um.edu.my

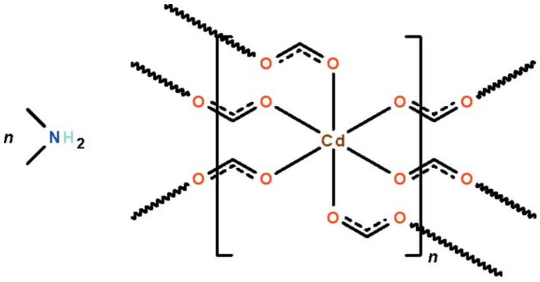
Received 9 November 2010; accepted 12 November 2010

Key indicators: single-crystal X-ray study; T = 293 K; mean σ(O–C) = 0.001 Å; disorder in main residue; R factor = 0.022; wR factor = 0.055; data-to-parameter ratio = 11.2.

In the coordination polymer, $\{(C_2H_8N)[Cd(CHO_2)_3]\}_n$, the Cd^{II} atom lies on a special position of $\overline{3}$ site symmetry in an octahedron of O atoms. The formate unit bridges the metal atoms, generating a three-dimensional polyanionic framework. The disordered cations occupy the cavities within the framework, and are N—H⋯O hydrogen-bonded to the framework.

Related literature

For the tris(formato)zincate cation, see; Fortier & Creber (1985); Marsh (1986). Tris(formato)cadmate is not isotypic to the aforementioned Zn structures.



Experimental

Crystal data

$(C_2H_8N)[Cd(CHO_2)_3]$
M_r = 293.55
Trigonal, $R\overline{3}c$
a = 8.5121 (4) Å
c = 23.0022 (9) Å
V = 1443.36 (9) Å^3
Z = 6
Mo $K\alpha$ radiation
μ = 2.27 mm^{-1}
T = 293 K
0.22 × 0.19 × 0.15 mm

Data collection

Rigaku R-AXIS RAPID diffractometer
Absorption correction: multi-scan (*ABSCOR*; Higashi, 1995)
T_{min} = 0.635, T_{max} = 0.727
4250 measured reflections
370 independent reflections
352 reflections with $I > 2\sigma(I)$
R_{int} = 0.024

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.022
$wR(F^2)$ = 0.055
S = 1.09
370 reflections
33 parameters
9 restraints
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.73 e Å^{-3}
$\Delta\rho_{min}$ = −0.36 e Å^{-3}

Table 1
Hydrogen-bond geometry (Å, °).

D—H⋯A	D—H	H⋯A	D⋯A	D—H⋯A
N1—H1⋯O1	0.88	1.99	2.84 (7)	163

Data collection: *RAPID-AUTO* (Rigaku, 1998); cell refinement: *RAPID-AUTO*; data reduction: *CrystalStructure* (Rigaku/MSC, 2002); program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *X-SEED* (Barbour, 2001); software used to prepare material for publication: *publCIF* (Westrip, 2010).

We thank the Key Project of the Natural Science Foundation of Heilongjiang Province (No. ZD200903) and the Innovation Team of the Education Bureau of Heilongjiang Province (No. 2010 t d03), Heilongjiang University and the University of Malaya for supporting this study.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: HG2747).

References

Barbour, L. J. (2001). *J. Supramol. Chem.* **1**, 189–191.
Fortier, S. & Creber, K. A. M. (1985). *Acta Cryst.* C**41**, 1763–1765.
Higashi, T. (1995). *ABSCOR*. Program for Absorption Correction, Tokyo, Japan.
Marsh, R. E. (1986). *Acta Cryst.* C**42**, 1327–1328.
Rigaku (1998). *RAPID-AUTO*. Rigaku Corporation, Tokyo, Japan.
Rigaku/MSC (2002). *CrystalStructure*. Rigaku/MSC, The Woodlands, Texas, USA.
Sheldrick, G. M. (2008). *Acta Cryst.* A**64**, 112–122.
Westrip, S. P. (2010). *J. Appl. Cryst.* **43**, 920–925.

supporting information

Acta Cryst. (2010). E**66**, m1599   [https://doi.org/10.1107/S1600536810046830]

Poly[dimethylammonium [tris(μ_2-formato-κ^2O:O')cadmate(II)]]

Shan Gao and Seik Weng Ng

S1. Comment

For some hydrothermal syntheses involving carboxylic acids, the *N*,*N*-dimethylformamide that is used as solvent is partially converted to the dimethylammonium cation, whose charge is balanced by the carboxylate ion. In the present study, the attempt to synthesize a coordination compound of a cadmium carboxylate yielded the tris(formato)cadmate anion (Scheme I). In the salt (Fig. 1), the cadmium atom lies on a special position of $\overline{3}$ site symmetry in an octahedron of O atoms. The formate unit bridges the metal atoms to generate a three-dimensional polyanionic framework, whose cavities are occupied by disordered cations.

A similar tris(formato)zincate(II) has been reported; the compound was synthesized directly from a zinc salt and formic acid in DMF medium (Fortier & Creber, 1985; Marsh, 1986). The later study has assumed the cation to be the formamidine cation, $(NH_2)CH(NH_2)^+$. Possibly, the cation is the dimethylammonium cation.

S2. Experimental

N,*N*-Dimethylformamide (10 ml), water (1 ml), ethanol (1 ml), formic acid (0.1 ml), cadmium nitrate (5 mmol), 1,10-phenanthroline (5 mmol) and benzoic acid (5 mmol) were heated in a 23-ml Teflon-lined autoclave at 383 K for 3 days. After slow cooling the autoclave to room temperature, colorless crystals were obtained.

S3. Refinement

Hydrogen atoms were placed in calculated positions (C–H 0.93, N–H 0.88 Å) and were included in the refinement in the riding model approximation, with U(H) set to 1.2–1.5U_{eq}(C,N).

The dimethylammonium cation was allowed to refine off the special position; the two N–C distances were restrained to 1.50±0.01 Å and the C···C distance to 2.35±0.01 Å. The anisotropic temperature factors of the carbon atoms were restrained to be nearly isotropic.

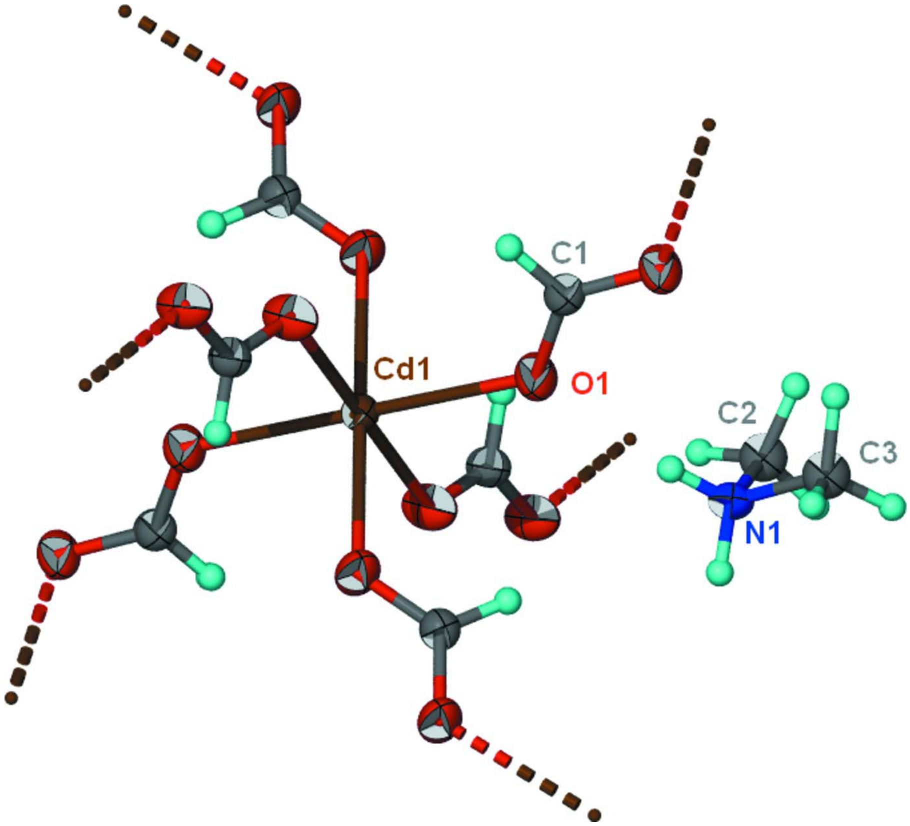


Figure 1

Thermal ellipsoid plot (Barbour, 2001) of a portion of poly[dimethylammonium tris(formato)cadmate] at the 50% probability level; hydrogen atoms are drawn as spheres of arbitrary radius.

Poly[dimethylammonium [tris(μ_2-formato-κ^2O:O')cadmate(II)]]

Crystal data

$(C_2H_8N)[Cd(CHO_2)_3]$	$D_x = 2.026$ Mg m^{-3}
$M_r = 293.55$	Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Trigonal, $R\bar{3}c$	Cell parameters from 3921 reflections
Hall symbol: -R 3 2" c	$\theta = 3.3–37.5°$
$a = 8.5121$ (4) Å	$\mu = 2.27$ mm^{-1}
$c = 23.0022$ (9) Å	$T = 293$ K
$V = 1443.36$ (9) Å^3	Prism, colorless
$Z = 6$	0.22 × 0.19 × 0.15 mm
$F(000) = 864$	

Data collection

Rigaku R-AXIS RAPID diffractometer	4250 measured reflections
Radiation source: fine-focus sealed tube	370 independent reflections
Graphite monochromator	352 reflections with $I > 2\sigma(I)$
Detector resolution: 10.000 pixels mm^{-1}	$R_{int} = 0.024$
ω scans	$\theta_{max} = 27.5°$, $\theta_{min} = 3.3°$
Absorption correction: multi-scan (*ABSCOR*; Higashi, 1995)	$h = -11 \rightarrow 11$
$T_{min} = 0.635$, $T_{max} = 0.727$	$k = -11 \rightarrow 9$
	$l = -29 \rightarrow 29$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.022$
$wR(F^2) = 0.055$
$S = 1.09$
370 reflections
33 parameters
9 restraints
Primary atom site location: structure-invariant direct methods
Secondary atom site location: difference Fourier map
Hydrogen site location: inferred from neighbouring sites
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0419P)^2 + 0.5666P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.001$
$\Delta\rho_{max} = 0.73$ e Å^{-3}
$\Delta\rho_{min} = -0.36$ e Å^{-3}

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

	x	y	z	U_{iso}*/U_{eq}	Occ. (<1)
Cd1	0.0000	0.0000	0.0000	0.02669 (17)	
O1	0.23112 (15)	0.21016 (15)	0.05612 (5)	0.0452 (3)	
C1	0.2265 (2)	0.3333	0.0833	0.0328 (4)	
H1A	0.1173	0.3333	0.0833	0.039*	
N1	0.578 (6)	0.252 (5)	0.0797 (17)	0.040 (4)	0.167
H1B	0.5788	0.1489	0.0798	0.048*	0.167
H1	0.4646	0.2277	0.0792	0.048*	0.167
C2	0.680 (5)	0.365 (4)	0.0282 (14)	0.041 (4)*	0.167
H2A	0.6061	0.3181	−0.0061	0.062*	0.167
H2B	0.7899	0.3619	0.0232	0.062*	0.167
H2C	0.7079	0.4881	0.0344	0.062*	0.167
C3	0.676 (7)	0.364 (4)	0.1320 (15)	0.041 (4)*	0.167
H3A	0.6210	0.2961	0.1667	0.062*	0.167
H3B	0.6687	0.4732	0.1322	0.062*	0.167
H3C	0.8010	0.3952	0.1306	0.062*	0.167

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Cd1	0.02630 (19)	0.02630 (19)	0.0275 (2)	0.01315 (10)	0.000	0.000
O1	0.0397 (6)	0.0420 (6)	0.0554 (7)	0.0215 (5)	−0.0121 (5)	−0.0206 (5)
C1	0.0301 (8)	0.0326 (11)	0.0367 (10)	0.0163 (5)	−0.0006 (4)	−0.0011 (8)
N1	0.040 (8)	0.028 (6)	0.055 (8)	0.019 (4)	−0.006 (7)	0.002 (7)

Geometric parameters (Å, º)

Cd1—O1^{i}	2.2841 (10)	N1—C3	1.505 (10)
Cd1—O1	2.2841 (10)	N1—H1B	0.8800
Cd1—O1^{ii}	2.2841 (10)	N1—H1	0.8800
Cd1—O1^{iii}	2.2841 (10)	C2—H2A	0.9600
Cd1—O1^{iv}	2.2841 (10)	C2—H2B	0.9600
Cd1—O1^{v}	2.2841 (10)	C2—H2C	0.9600
O1—C1	1.2384 (14)	C3—H3A	0.9600
C1—O1^{vi}	1.2383 (14)	C3—H3B	0.9600

C1—H1A	0.9300	C3—H3C	0.9600
N1—C2	1.499 (10)		

O1^{i}—Cd1—O1	180.00 (5)	C2—N1—C3	105.3 (8)
O1^{i}—Cd1—O1^{ii}	91.20 (4)	C2—N1—H1B	110.7
O1—Cd1—O1^{ii}	88.80 (4)	C3—N1—H1B	110.7
O1^{i}—Cd1—O1^{iii}	91.20 (4)	C2—N1—H1	110.7
O1—Cd1—O1^{iii}	88.80 (4)	C3—N1—H1	110.7
O1^{ii}—Cd1—O1^{iii}	91.20 (4)	H1B—N1—H1	108.8
O1^{i}—Cd1—O1^{iv}	88.80 (4)	N1—C2—H2A	109.5
O1—Cd1—O1^{iv}	91.20 (4)	N1—C2—H2B	109.5
O1^{ii}—Cd1—O1^{iv}	88.80 (4)	H2A—C2—H2B	109.5
O1^{iii}—Cd1—O1^{iv}	180.00 (7)	N1—C2—H2C	109.5
O1^{i}—Cd1—O1^{v}	88.80 (4)	H2A—C2—H2C	109.5
O1—Cd1—O1^{v}	91.20 (4)	H2B—C2—H2C	109.5
O1^{ii}—Cd1—O1^{v}	180.00 (5)	N1—C3—H3A	109.5
O1^{iii}—Cd1—O1^{v}	88.80 (4)	N1—C3—H3B	109.5
O1^{iv}—Cd1—O1^{v}	91.20 (4)	H3A—C3—H3B	109.5
C1—O1—Cd1	124.71 (11)	N1—C3—H3C	109.5
O1^{vi}—C1—O1	125.90 (19)	H3A—C3—H3C	109.5
O1^{vi}—C1—H1A	117.1	H3B—C3—H3C	109.5
O1—C1—H1A	117.1		

O1^{ii}—Cd1—O1—C1	151.34 (11)	O1^{v}—Cd1—O1—C1	−28.66 (11)
O1^{iii}—Cd1—O1—C1	60.11 (7)	Cd1—O1—C1—O1^{vi}	−174.40 (11)
O1^{iv}—Cd1—O1—C1	−119.89 (7)		

Symmetry codes: (i) $-x, -y, -z$; (ii) $y, -x+y, -z$; (iii) $x-y, x, -z$; (iv) $-x+y, -x, z$; (v) $-y, x-y, z$; (vi) $x-y+1/3, -y+2/3, -z+1/6$.

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1—H1···O1	0.88	1.99	2.84 (7)	163