

Poly[(μ_6 -benzene-1,3,5-tricarboxylato- $\kappa^6O^1:O^1':O^3:O^3':O^5:O^5'$)tris(N,N -dimethylformamide- κO)tris(μ_3 -formato- $\kappa^2O:O'$)trimagnesium(II)]

Chun-Ting Yeh, Hsin-Kuan Liu, Chia-Jing Lin and Chia-Her Lin*

Department of Chemistry, Chung-Yuan Christian University, Chung-Li 320, Taiwan
Correspondence e-mail: chiaher@cycu.edu.tw

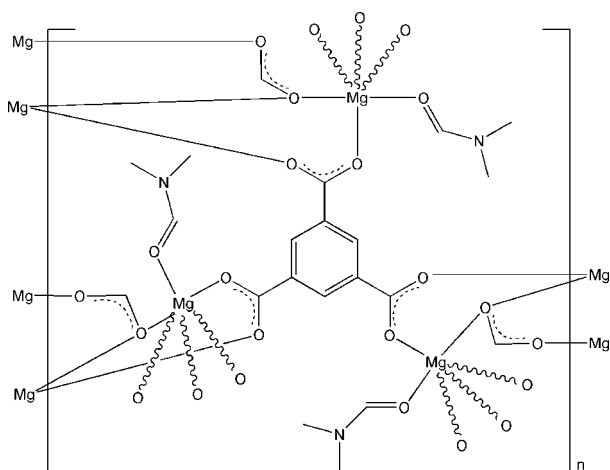
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Key indicators: single-crystal X-ray study; $T = 295$ K; mean $\sigma(C-C) = 0.006$ Å; R factor = 0.078; wR factor = 0.214; data-to-parameter ratio = 17.7.

The title complex, $[Mg_3(CHO_2)_3(C_9H_3O_6)(C_3H_7NO)_3]_n$, exhibits a two-dimensional structure parallel to (001), which is built up from the Mg^{II} atoms and bridging carboxylate ligands (3 symmetry). The Mg^{II} atom is six-coordinated by one O atom from a dimethylformamide molecule, two O atoms from two μ_6 -benzene-1,3,5-tricarboxylate ligands and three O atoms from three μ_3 -formate ligands in a distorted octahedral geometry.

Related literature

For general background to the synthesis and structures of coordination polymers, see: Kitagawa *et al.* (2004); Liu *et al.* (2009). For an isotopic structure, see: He *et al.* (2006).



Experimental

Crystal data

$[Mg_3(CHO_2)_3(C_9H_3O_6)(C_3H_7NO)_3]$ $Z = 6$
 $M_r = 211.46$ $Mo\ K\alpha$ radiation
 Trigonal, $P\bar{3}$ $\mu = 0.19\ mm^{-1}$
 $a = 13.9739$ (2) Å $T = 295\ K$
 $c = 8.1188$ (1) Å $0.25 \times 0.25 \times 0.15\ mm$
 $V = 1372.96$ (3) Å³

Data collection

Bruker APEXII CCD 12089 measured reflections
 diffractometer 2278 independent reflections
 Absorption correction: multi-scan 2212 reflections with $I > 2\sigma(I)$
 (*SADABS*; Bruker, 2001) $R_{int} = 0.032$
 $T_{min} = 0.954$, $T_{max} = 0.972$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.078$ 129 parameters
 $wR(F^2) = 0.214$ H-atom parameters constrained
 $S = 1.40$ $\Delta\rho_{max} = 0.47\ e\ \text{Å}^{-3}$
 2278 reflections $\Delta\rho_{min} = -0.35\ e\ \text{Å}^{-3}$

Table 1

Selected bond lengths (Å).

Mg1—O1	2.008 (3)	Mg1—O3	2.047 (3)
Mg1—O2	2.132 (3)	Mg1—O4 ⁱⁱ	2.080 (3)
Mg1—O2 ⁱ	2.135 (3)	Mg1—O1S	2.115 (4)

Symmetry codes: (i) $x - y, x - 1, -z + 1$; (ii) $-y + 1, x - y - 1, z$.

Data collection: *APEX2* (Bruker, 2007); cell refinement: *SAINT* (Bruker, 2007); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *SHELXTL* (Sheldrick, 2008) and *DIAMOND* (Brandenburg, 1999); software used to prepare material for publication: *SHELXTL*.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: HY2348).

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supplementary materials

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Poly[(μ_6 -benzene-1,3,5-tricarboxylato- $\kappa^6 O^1:O^1':O^3:O^3':O^5:O^5'$)tris(*N,N*-dimethylformamide- κO)tris(μ_3 -formato- $\kappa^2 O:O'$)trimagnesium(II)]

C.-T. Yeh, H.-K. Liu, C.-J. Lin and C.-H. Lin

Comment

The synthesis of coordination polymers, or so-called metal-organic frameworks (MOFs), has been a subject of intense research owing to their interesting structural chemistry and potential applications in gas storage, separation, catalysis, magnetism, luminescence. A large number of these materials have been synthesized by solvothermal reactions with organic carboxyl acids (Kitagawa *et al.*, 2004; Liu *et al.*, 2009). They commonly adopt three-, two- or one-dimensional structures *via* employed metal ions as connectors and rigid or flexible organic ligands as linkers. As a further study of such complexes, we report the title compound (Fig. 1), which is isotypic with $[\text{Co}_6(\text{C}_9\text{H}_3\text{O}_6)_2(\text{CHO}_2)_6(\text{C}_3\text{H}_7\text{NO})_6]_n$ (He *et al.*, 2006) and has a two-dimensional polymeric network (Fig. 2). All the geometric parameters are within normal ranges (Table 1). The Mg^{II} atom is six-coordinated by one O atoms from an *N,N*-dimethylformamide molecule and five O atoms from the carboxylate ligands, giving a distorted octahedral geometry.

Experimental

Solvothermal reactions were carried out at 423 K for 2 d in a Teflon-lined acid digestion bomb with an internal volume of 23 ml followed by slow cooling at 6 K h⁻¹ to room temperature. A single-phase product consisting of transparent colorless crystals was obtained from a mixture of $\text{Mg}(\text{NO}_3)_2 \cdot 6\text{H}_2\text{O}$ (0.051 g, 0.2 mmol), benzene-1,3,5-tricarboxylic acid (0.021 g, 0.1 mmol) and formic acid (0.5 ml, 0.4 M) and *N,N*-dimethylformamide (5.0 ml).

Refinement

H atoms were constrained to ideal geometries, with C—H = 0.93 (CH) and 0.96 (CH₃) Å and $U_{\text{iso}}(\text{H}) = 1.2(1.5 \text{ for methyl})U_{\text{eq}}(\text{C})$.

Figures

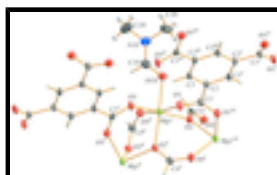


Fig. 1. Part of the polymeric structure of the title compound, showing 50% probability displacement ellipsoids. [Symmetry codes: (i) $x-y, -1+x, 1-z$; (ii) $1-y, -1+x-y, z$; (iii) $1-x+y, 1-x, z$; (iv) $1-x, -y, 1-z$; (v) $1-y, x-y, z$; (vi) $x-y, x, 1-z$; (vii) $1+y, 1-x+y, 1-z$.]

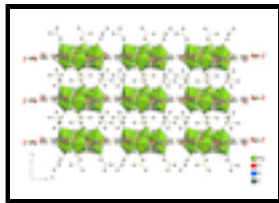


Fig. 2. Crystal packing diagram of the title compound along the *a* axis.

Poly[(μ_6 -benzene-1,3,5-tricarboxylato- $\kappa^6O^1:O^1':O^3:O^3':O^5:O^5'$)tris(*N,N*-dimethylformamide- κO)tris(μ_3 -formato- $\kappa^2O:O'$) trimagnesium(II)]

Crystal data

[Mg₃(CHO₂)₃(C₉H₃O₆)(C₃H₇NO)₃]

M_r = 211.46

Trigonal, *P* $\bar{3}$

Hall symbol: -P 3

a = 13.9739 (2) Å

c = 8.1188 (1) Å

V = 1372.96 (3) Å³

Z = 6

F(000) = 660

D_x = 1.535 Mg m⁻³

Mo *K*α radiation, λ = 0.71073 Å

Cell parameters from 9292 reflections

θ = 2.9–28.3°

μ = 0.19 mm⁻¹

T = 295 K

Columnar, colorless

0.25 × 0.25 × 0.15 mm

Data collection

Bruker APEXII CCD
diffractometer

Radiation source: fine-focus sealed tube
graphite

Detector resolution: 8.3333 pixels mm⁻¹
φ and ω scans

Absorption correction: multi-scan
(*SADABS*; Bruker, 2001)

T_{min} = 0.954, *T_{max}* = 0.972

12089 measured reflections

2278 independent reflections

2212 reflections with *I* > 2σ(*I*)

R_{int} = 0.032

θ_{max} = 28.3°, θ_{min} = 1.7°

h = -18→17

k = -17→18

l = -10→10

Refinement

Refinement on *F*²

Least-squares matrix: full

R[*F*² > 2σ(*F*²)] = 0.078

wR(*F*²) = 0.214

S = 1.40

2278 reflections

129 parameters

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/[σ²(*F_o*²) + (0.0309*P*)² + 6.185*P*]

where *P* = (*F_o*² + 2*F_c*²)/3

(Δ/σ)_{max} < 0.001

Δρ_{max} = 0.47 e Å⁻³

0 restraints

$$\Delta\rho_{\min} = -0.35 \text{ e } \text{\AA}^{-3}$$

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

	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{iso}}^*/U_{\text{eq}}$
Mg1	0.77826 (11)	0.01439 (11)	0.39889 (18)	0.0184 (3)
O1	0.7487 (3)	0.1240 (2)	0.5146 (4)	0.0261 (7)
O2	0.9488 (2)	0.1369 (2)	0.3855 (4)	0.0220 (6)
O3	0.6145 (2)	-0.1017 (2)	0.4220 (4)	0.0256 (7)
O1S	0.7459 (3)	0.0733 (3)	0.1779 (4)	0.0327 (8)
C2	0.7301 (3)	0.2815 (3)	0.5313 (5)	0.0181 (8)
C3	0.6154 (3)	0.2191 (3)	0.5310 (5)	0.0188 (8)
H3	0.5809	0.1423	0.5307	0.023*
C4	1.0034 (3)	0.1485 (3)	0.2561 (5)	0.0223 (8)
H4A	0.9631	0.1231	0.1590	0.027*
C1	0.7986 (3)	0.2259 (3)	0.5408 (5)	0.0190 (8)
N1S	0.6441 (4)	0.0408 (4)	-0.0535 (5)	0.0363 (10)
C1S	0.6876 (4)	0.0111 (4)	0.0655 (6)	0.0328 (10)
H1S	0.6738	-0.0612	0.0656	0.039*
C2S	0.5833 (5)	-0.0326 (6)	-0.1892 (7)	0.0506 (16)
H2S1	0.5853	-0.1000	-0.1790	0.076*
H2S2	0.6165	0.0025	-0.2918	0.076*
H2S3	0.5079	-0.0488	-0.1864	0.076*
C3S	0.6642 (6)	0.1535 (6)	-0.0614 (8)	0.0498 (15)
H3S1	0.6850	0.1869	0.0456	0.075*
H3S2	0.5982	0.1525	-0.0968	0.075*
H3S3	0.7227	0.1954	-0.1383	0.075*
O4	1.1048 (3)	0.1900 (3)	0.2443 (4)	0.0261 (7)

Atomic displacement parameters (\AA^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Mg1	0.0125 (6)	0.0126 (6)	0.0301 (7)	0.0064 (5)	0.0002 (5)	0.0000 (5)
O1	0.0203 (14)	0.0149 (14)	0.0459 (19)	0.0108 (12)	0.0012 (13)	-0.0037 (13)
O2	0.0148 (13)	0.0184 (14)	0.0301 (16)	0.0063 (11)	0.0005 (11)	-0.0004 (11)
O3	0.0123 (13)	0.0136 (13)	0.0477 (19)	0.0041 (11)	0.0005 (12)	0.0017 (13)
O1S	0.0328 (18)	0.0305 (17)	0.0361 (18)	0.0169 (15)	-0.0057 (14)	0.0031 (14)
C2	0.0127 (17)	0.0148 (17)	0.029 (2)	0.0085 (14)	-0.0008 (14)	-0.0002 (14)
C3	0.0139 (17)	0.0114 (16)	0.030 (2)	0.0056 (14)	-0.0003 (14)	-0.0002 (14)
C4	0.0185 (19)	0.0194 (19)	0.027 (2)	0.0081 (16)	-0.0016 (15)	0.0009 (15)
C1	0.0179 (18)	0.0160 (17)	0.027 (2)	0.0112 (15)	0.0021 (15)	-0.0001 (15)
N1S	0.033 (2)	0.044 (3)	0.032 (2)	0.019 (2)	-0.0002 (17)	0.0045 (19)
C1S	0.028 (2)	0.033 (2)	0.039 (3)	0.016 (2)	0.003 (2)	0.006 (2)
C2S	0.039 (3)	0.067 (4)	0.034 (3)	0.018 (3)	-0.001 (2)	-0.001 (3)
C3S	0.064 (4)	0.054 (4)	0.044 (3)	0.039 (3)	-0.005 (3)	0.010 (3)
O4	0.0182 (14)	0.0281 (16)	0.0316 (17)	0.0112 (13)	0.0011 (12)	0.0005 (13)

supplementary materials

Geometric parameters (\AA , $^\circ$)

Mg1—O1	2.008 (3)	C3—H3	0.9300
Mg1—O2	2.132 (3)	C4—O4	1.238 (5)
Mg1—O2 ⁱ	2.135 (3)	C4—H4A	0.9300
Mg1—O3	2.047 (3)	C1—O3 ^v	1.248 (5)
Mg1—O4 ⁱⁱ	2.080 (3)	N1S—C1S	1.314 (6)
Mg1—O1S	2.115 (4)	N1S—C3S	1.456 (8)
O1—C1	1.251 (5)	N1S—C2S	1.455 (7)
O2—C4	1.260 (5)	C1S—H1S	0.9300
O3—C1 ⁱ	1.248 (5)	C2S—H2S1	0.9600
O1S—C1S	1.243 (6)	C2S—H2S2	0.9600
C2—C3	1.390 (5)	C2S—H2S3	0.9600
C2—C3 ⁱⁱⁱ	1.392 (5)	C3S—H3S1	0.9600
C2—C1	1.507 (5)	C3S—H3S2	0.9600
C3—C2 ^{iv}	1.392 (5)	C3S—H3S3	0.9600
O1—Mg1—O3	89.26 (13)	C1S—O1S—Mg1	122.9 (3)
O1—Mg1—O4 ⁱⁱ	170.66 (15)	C3—C2—C3 ⁱⁱⁱ	119.2 (4)
O3—Mg1—O4 ⁱⁱ	93.03 (14)	C3—C2—C1	120.4 (4)
O1—Mg1—O1S	86.23 (15)	C3 ⁱⁱⁱ —C2—C1	120.3 (3)
O3—Mg1—O1S	90.84 (15)	C2—C3—C2 ^{iv}	120.8 (4)
O4 ⁱⁱ —Mg1—O1S	84.69 (14)	C2—C3—H3	119.6
O1—Mg1—O2	89.14 (13)	C2 ^{iv} —C3—H3	119.6
O3—Mg1—O2	177.59 (15)	O4—C4—O2	127.0 (4)
O4 ⁱⁱ —Mg1—O2	88.83 (13)	O4—C4—H4A	116.5
O1S—Mg1—O2	90.85 (14)	O2—C4—H4A	116.5
O1—Mg1—O2 ⁱ	96.73 (14)	O3 ^v —C1—O1	125.8 (4)
O3—Mg1—O2 ⁱ	89.05 (13)	O3 ^v —C1—C2	118.0 (3)
O4 ⁱⁱ —Mg1—O2 ⁱ	92.36 (13)	O1—C1—C2	116.2 (4)
O1S—Mg1—O2 ⁱ	177.04 (15)	C1S—N1S—C3S	120.3 (5)
O2—Mg1—O2 ⁱ	89.35 (15)	C1S—N1S—C2S	122.2 (5)
O1—Mg1—Mg1 ⁱ	123.18 (13)	C3S—N1S—C2S	117.3 (5)
O3—Mg1—Mg1 ⁱ	72.76 (10)	O1S—C1S—N1S	124.4 (5)
O4 ⁱⁱ —Mg1—Mg1 ⁱ	66.10 (9)	O1S—C1S—H1S	117.8
O1S—Mg1—Mg1 ⁱ	144.98 (12)	N1S—C1S—H1S	117.8
O2—Mg1—Mg1 ⁱ	106.68 (9)	N1S—C2S—H2S1	109.5
O2 ⁱ —Mg1—Mg1 ⁱ	32.42 (8)	N1S—C2S—H2S2	109.5
O1—Mg1—Mg1 ^v	65.14 (11)	H2S1—C2S—H2S2	109.5
O3—Mg1—Mg1 ^v	145.14 (13)	N1S—C2S—H2S3	109.5
O4 ⁱⁱ —Mg1—Mg1 ^v	116.15 (10)	H2S1—C2S—H2S3	109.5
O1S—Mg1—Mg1 ^v	109.51 (11)	H2S2—C2S—H2S3	109.5
O2—Mg1—Mg1 ^v	32.48 (8)	N1S—C3S—H3S1	109.5

O2 ⁱ —Mg1—Mg1 ^v	72.03 (9)	N1S—C3S—H3S2	109.5
Mg1 ⁱ —Mg1—Mg1 ^v	100.84 (5)	H3S1—C3S—H3S2	109.5
C1—O1—Mg1	137.5 (3)	N1S—C3S—H3S3	109.5
C4—O2—Mg1	120.5 (3)	H3S1—C3S—H3S3	109.5
C4—O2—Mg1 ^v	124.4 (3)	H3S2—C3S—H3S3	109.5
Mg1—O2—Mg1 ^v	115.10 (14)	C4—O4—Mg1 ^{vi}	137.3 (3)
C1 ⁱ —O3—Mg1	128.3 (3)		

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

Fig. 1

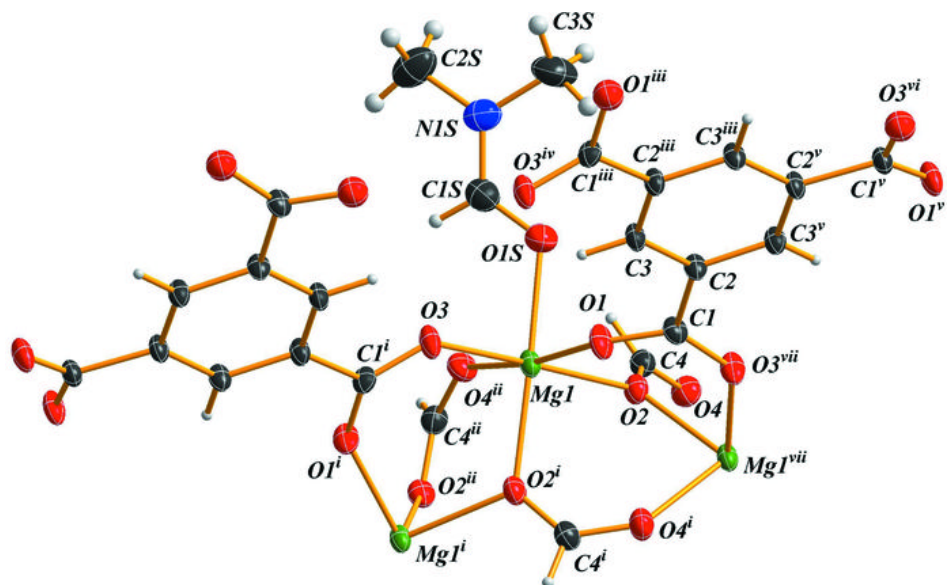


Fig. 2

