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# Poly[($\mu_6$-benzene-1,3,5-tricarboxylato-$\kappa^6O^1:O^{1'}:O^3:O^{3'}:O^5:O^{5'}$)tris(*N*,*N*-dimethylformamide-$\kappa O$)tris($\mu_3$-formato-$\kappa^2O:O'$)trimagnesium(II)]

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

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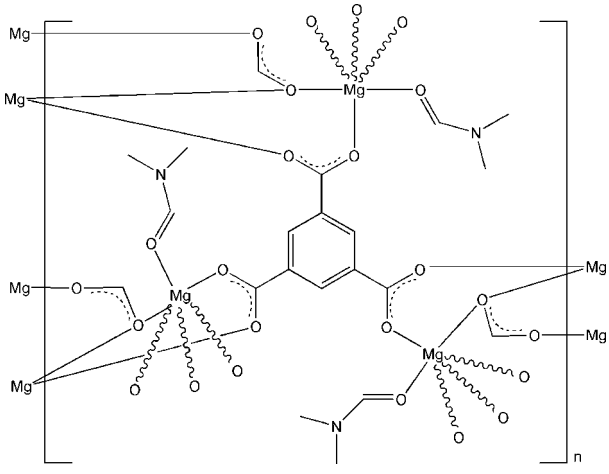
Key indicators: single-crystal X-ray study; $T$ = 295 K; mean $\sigma$(C–C) = 0.006 Å; $R$ factor = 0.078; $wR$ factor = 0.214; data-to-parameter ratio = 17.7.

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 $\mu_6$-benzene-1,3,5-tricarboxylate ligands and three O atoms from three $\mu_3$-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 isotypic structure, see: He *et al.* (2006).




## Experimental

### Crystal data

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

### Data collection

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

### Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.078
$wR(F^2)$ = 0.214
$S$ = 1.40
2278 reflections
129 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.47 e Å$^{-3}$
$\Delta\rho_{min}$ = −0.35 e 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—O1*S* | 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).

## References

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# supporting information

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# Poly[($\mu_6$-benzene-1,3,5-tricarboxylato-$\kappa^6O^1:O^{1'}:O^3:O^{3'}:O^5:O^{5'}$)tris(*N*,*N*-dimethylformamide-$\kappa O$)tris($\mu_3$-formato-$\kappa^2O:O'$)trimagnesium(II)]

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## S1. 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 $[Co_6(C_9H_3O_6)_2(CHO_2)_6(C_3H_7NO)_6]_n$ (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.

## S2. 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 $Mg(NO_3)_2.6H_2O$ (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).

## S3. Refinement

H atoms were constrained to ideal geometries, with C—H = 0.93 (CH) and 0.96 ($CH_3$) Å and $U_{iso}$(H) = 1.2(1.5 for methyl)$U_{eq}$(C).

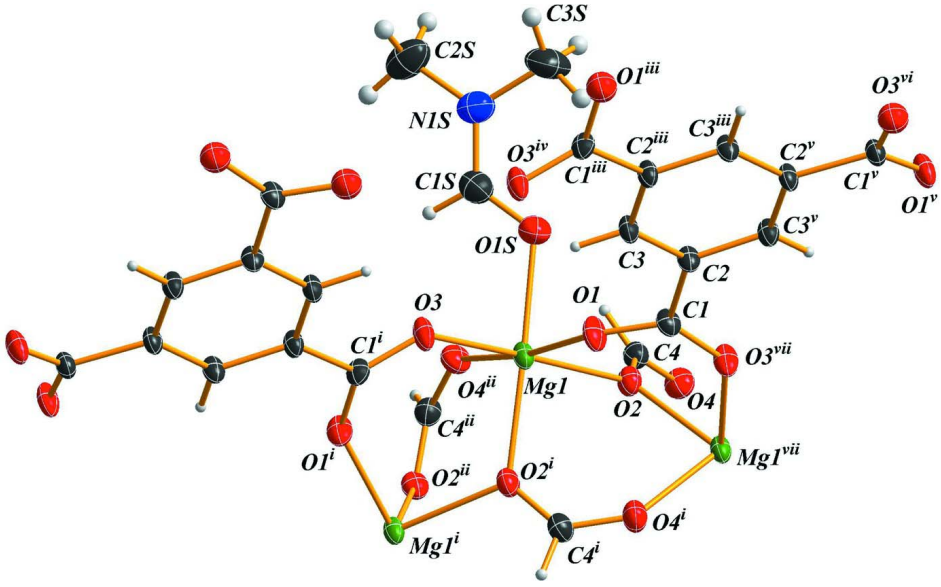


**Figure 1**

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.]

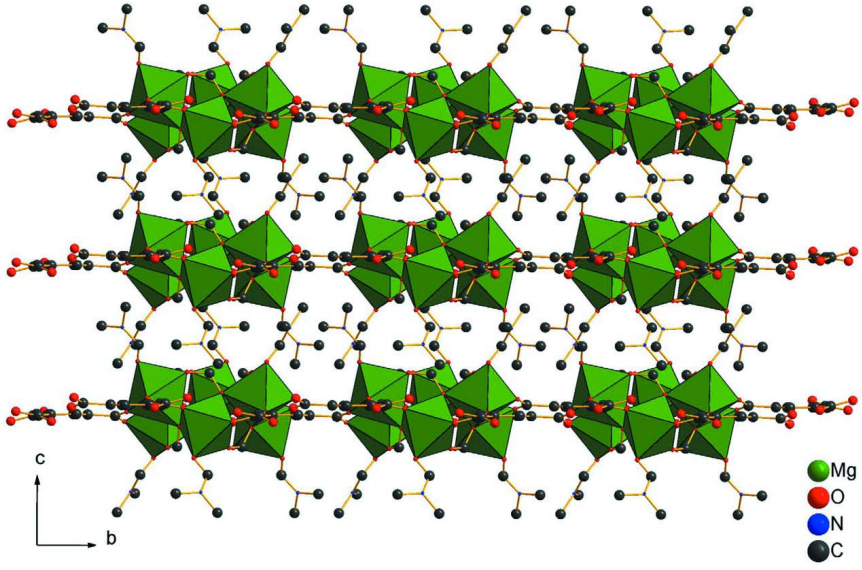


**Figure 2**

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

**Poly[($\mu_6$-benzene-1,3,5-tricarboxylato-$\kappa^6O^1:O^{1'}:O^3:O^{3'}:O^5:O^{5'}$)tris(*N*,*N*- dimethylformamide-$\kappa O$)tris($\mu_3$-formato-$\kappa^2O:O'$) trimagnesium(II)]**

*Crystal data*

$[Mg_3(CHO_2)_3(C_9H_3O_6)(C_3H_7NO)_3]$
$M_r$ = 211.46
Trigonal, $P\overline{3}$
Hall symbol: -P 3
$a$ = 13.9739 (2) Å
$c$ = 8.1188 (1) Å
$V$ = 1372.96 (3) Å$^3$
$Z$ = 6
$F(000)$ = 660
$D_x$ = 1.535 Mg m$^{-3}$
Mo $K\alpha$ radiation, $\lambda$ = 0.71073 Å
Cell parameters from 9292 reflections
$\theta$ = 2.9–28.3°
$\mu$ = 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 monochromator
Detector resolution: 8.3333 pixels mm$^{-1}$
$\varphi$ and $\omega$ 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 $I > 2\sigma(I)$
$R_{int}$ = 0.032
$\theta_{max}$ = 28.3°, $\theta_{min}$ = 1.7°
$h$ = −18→17
$k$ = −17→18
$l$ = −10→10

*Refinement*

Refinement on $F^2$
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.078
$wR(F^2)$ = 0.214
$S$ = 1.40
2278 reflections
129 parameters
0 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
$w = 1/[\sigma^2(F_o^2) + (0.0309P)^2 + 6.185P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.47 e Å$^{-3}$
$\Delta\rho_{min}$ = −0.35 e Å$^{-3}$

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

| | *x* | *y* | *z* | $U_{iso}$*/$U_{eq}$ |
|---|---|---|---|---|
| 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 (Å²)*

| | $U^{11}$ | $U^{22}$ | $U^{33}$ | $U^{12}$ | $U^{13}$ | $U^{23}$ |
|---|---|---|---|---|---|---|
| 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) |

*Geometric parameters (Å, º)*

| | | | |
|---|---|---|---|
| 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) $-x+y+2$, $-x+1$, $z$.