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Hexakis(*N*,*N*-dimethylformamide-κ*O*)-magnesium tetrachloromagnesate

Bobby L. Barker, David Aubry, Frank R. Fronczek, Steven F. Watkins* and George G. Stanley

Department of Chemistry, Louisiana State University, Baton Rouge, LA 70803, USA

Correspondence e-mail: swatkins@lsu.edu

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The crystallographically independent unit of the title compound, $[Mg(C_3H_7NO)_6][MgCl_4]$, consists of two six-coordinate magnesium dications and two four-coordinate magnesium dianions. The two cations are quasi-octahedral and statistically equivalent [average Mg—O = 2.07 (3) Å] and the anions are quasi-tetrahedral and statistically equivalent [average Mg—Cl = 2.334 (11) Å].

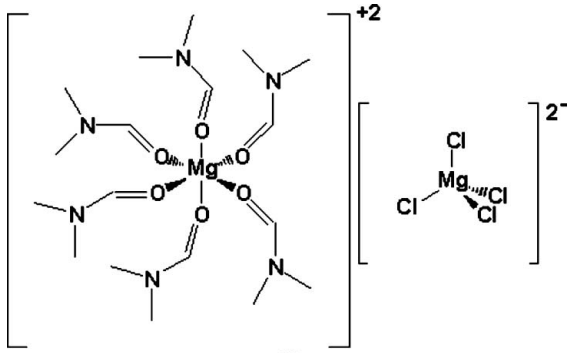
Key indicators

Single-crystal X-ray study
T = 120 K
Mean σ(l–Mg) = 0.001 Å
R factor = 0.035
wR factor = 0.068
Data-to-parameter ratio = 11.4

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

Comment

The asymmetric unit of the title compound consists of two cations and two anions. Equivalent bond lengths and bond angles of the metal-bonded *N*,*N*-dimethylformamide (DMFA) ligands are statistically equal to one another and are consistent with values reported for the $[(DMFA)_6Mg]^{2+}$ dication by Krautscheid & Vielsack (1999). Other DMFA–Mg complexes have been reported by Hollander *et al.* (1973) and Adams *et al.* (2005). The bond lengths and angles in the $[MgCl_4]^{2-}$ dianion do not differ significantly from the values reported by Sobota *et al.* (1986), Sobota & Szafert (1996), and Pavanello *et al.* (1994).



(I)

Experimental

Crystals of the title compound were grown from dimethylformamide solution. Their preparation is discussed by Barker (2005).

Crystal data

$[Mg(C_3H_7NO)_6][MgCl_4]$
M_r = 629
Monoclinic, $P2_1$
a = 13.905 (2) Å
b = 12.108 (3) Å
c = 19.079 (3) Å
β = 90.717 (15)°
V = 3211.9 (10) Å^3
Z = 4
D_x = 1.301 Mg m^{-3}
Mo *Kα* radiation
Cell parameters from 7510 reflections
θ = 2.5–27.5°
μ = 0.45 mm^{-1}
T = 120 K
Fragment, colorless
0.35 × 0.25 × 0.1 mm

Data collection

Nonius KappaCCD diffractometer with an Oxford Cryosystems Cryostream cooler
ω scans with κ offsets
Absorption correction: multi-scan (*SCALEPACK*; Otwinowski & Minor 1997)
T_{min} = 0.929, T_{max} = 0.956
45159 measured reflections
7677 independent reflections
5986 reflections with $I > 2\sigma(I)$
R_{int} = 0.036
θ_{max} = 27.5°
$h = -18 \rightarrow 18$
$k = -15 \rightarrow 15$
$l = -24 \rightarrow 24$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.035
$wR(F^2)$ = 0.068
S = 1.02
7677 reflections
673 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0276P)^2 + 0.1249P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 0.26 e Å^{-3}
$\Delta\rho_{min}$ = −0.23 e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

Cl1—Mg3	2.3392 (12)	Mg1—O1	2.047 (2)
Cl2—Mg3	2.3167 (14)	Mg1—O3	2.058 (2)
Cl3—Mg3	2.3374 (14)	Mg1—O5	2.060 (2)
Cl4—Mg3	2.3274 (13)	Mg1—O4	2.080 (2)
Cl5—Mg4	2.3467 (13)	Mg2—O12	2.046 (2)
Cl6—Mg4	2.3248 (15)	Mg2—O11	2.054 (2)
Cl7—Mg4	2.3330 (13)	Mg2—O9	2.057 (2)
Cl8—Mg4	2.3460 (13)	Mg2—O7	2.080 (2)
Mg1—O2	2.035 (2)	Mg2—O8	2.098 (2)
Mg1—O6	2.042 (2)	Mg2—O10	2.123 (2)
O2—Mg1—O6	91.03 (10)	O12—Mg2—O8	91.20 (9)
O2—Mg1—O1	91.54 (9)	O11—Mg2—O8	88.33 (9)
O6—Mg1—O1	91.79 (9)	O9—Mg2—O8	95.95 (9)
O2—Mg1—O3	87.90 (9)	O7—Mg2—O8	87.38 (9)
O6—Mg1—O3	91.77 (9)	O12—Mg2—O10	89.81 (9)
O1—Mg1—O3	176.40 (11)	O11—Mg2—O10	90.87 (9)
O2—Mg1—O5	94.07 (10)	O9—Mg2—O10	89.10 (9)
O6—Mg1—O5	174.78 (11)	O7—Mg2—O10	87.58 (9)
O1—Mg1—O5	86.96 (9)	O8—Mg2—O10	174.87 (10)
O3—Mg1—O5	89.53 (9)	Cl2—Mg3—Cl4	109.97 (5)
O2—Mg1—O4	174.92 (9)	Cl2—Mg3—Cl3	107.74 (5)
O6—Mg1—O4	87.90 (10)	Cl4—Mg3—Cl3	109.80 (5)
O1—Mg1—O4	93.46 (9)	Cl2—Mg3—Cl1	109.47 (5)
O3—Mg1—O4	87.17 (9)	Cl4—Mg3—Cl1	108.84 (5)
O5—Mg1—O4	87.12 (9)	Cl3—Mg3—Cl1	111.01 (5)
O12—Mg2—O11	177.49 (10)	Cl6—Mg4—Cl7	111.91 (5)
O12—Mg2—O9	88.60 (9)	Cl6—Mg4—Cl8	108.60 (5)
O11—Mg2—O9	89.00 (9)	Cl7—Mg4—Cl8	108.18 (5)
O12—Mg2—O7	90.82 (9)	Cl6—Mg4—Cl5	109.11 (5)
O11—Mg2—O7	91.62 (9)	Cl7—Mg4—Cl5	109.12 (5)
O9—Mg2—O7	176.63 (9)	Cl8—Mg4—Cl5	109.91 (5)

Refinement of the Flack (1983) parameter with 6646 Friedel pairs led to a value of 0.49 (3); the crystal was thus assumed to be an inversion twin with equal components. Friedel pairs were averaged in the final refinement, and the absolute structure chosen was arbitrary. All H atoms were placed in calculated positions, with C—H distances of 0.93 Å and $U_{iso} = 1.2U_{eq}$ of the attached C atom, and thereafter treated as riding. A torsional parameter was refined for each methyl group.

Data collection: *COLLECT* (Nonius, 2000); cell refinement: *DENZO* and *SCALEPACK* (Otwinowski & Minor, 1997); data reduction: *DENZO* and *SCALEPACK*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *ORTEP-3* (Farrugia, 1997); software used to prepare material for publication: *SHELXL97*.

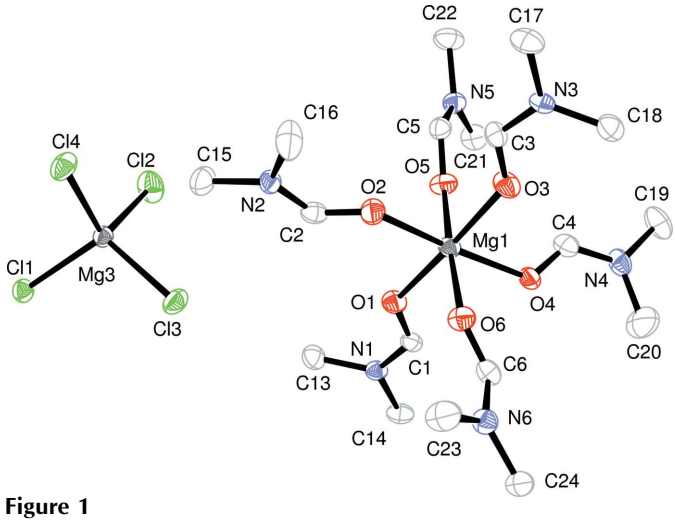


Figure 1
The atom-numbering scheme for cation–anion pair 1, with displacement ellipsoids shown at the 50% probability level. H atoms are not shown.

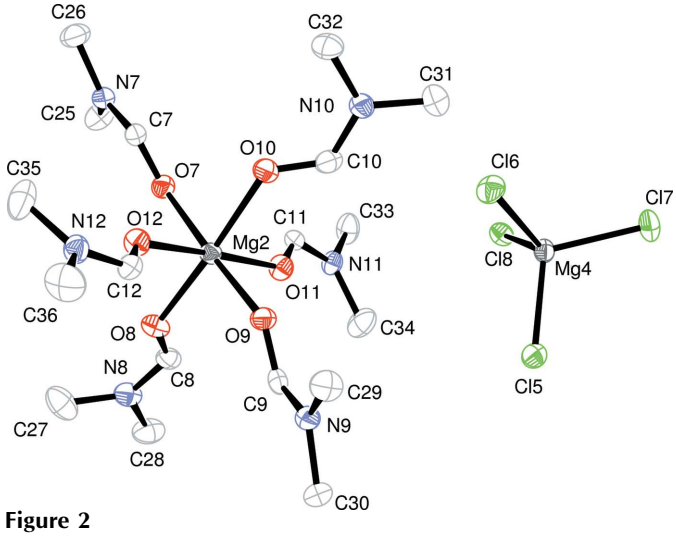


Figure 2
The atom-numbering scheme for cation–anion pair 2, with displacement ellipsoids shown at the 50% probability level. H atoms are not shown.

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