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Diaqua[2,13-dimethyl-3,12,18-triaza-6,9-dioxabicyclo[12.3.1]octadeca-1(18),2,12,14,16-pentaene]magnesium(II) Diperchlorate

A. HAMID OTHMAN

Department of Chemistry, National University of Malaysia, 43600 Bangi, Malaysia

SEIK WENG NG

Institute of Advanced Studies, University of Malaya, 59100 Kuala Lumpur, Malaysia

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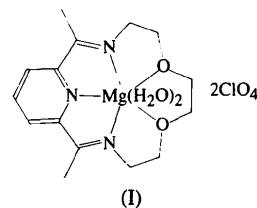
Abstract

The O and N atoms of the macrocyclic ligand occupy the pentagonal sites and the aqua O atoms the apical sites of the pentagonal bipyramidal geometry at the seven-coordinate Mg atom of the title compound, $[\text{Mg}(\text{C}_{15}\text{H}_{21}\text{N}_3\text{O}_2)(\text{H}_2\text{O})_2](\text{ClO}_4)_2$. Each H_2O molecule is involved in hydrogen bonding with the two ClO_4^- ions [2.797 (7) and 2.963 (9) Å].

Comment

The study of the title complex, diaqua[2,13-dimethyl-3,12,18-triaza-6,9-dioxabicyclo[12.3.1]octadeca-1(18),-2,12,14,16-pentaene]magnesium(II) diperchlorate, (I), was undertaken as an extension of a study of the effect of metal ions in promoting the synthesis of 2,13-dimethyl-3,12,18-triaza-6,9-dioxabicyclo[12.3.1]octadeca-1(18),2,12,14,16-pentaene, a relatively

rigid macrocycle that can be isolated only as its metal complexes (Drew, Othman, McFall, McIlroy & Nelson, 1977).



The magnesium complex of the macrocycle was assigned pentagonal bipyramidal geometry (Cook, Fenton, Drew, McFall & Nelson, 1977) and this has been confirmed in the present study. The cationic complex has exact twofold symmetry with the Mg, N and C3 atoms lying on the twofold axis that relates one half of the cation to the other.

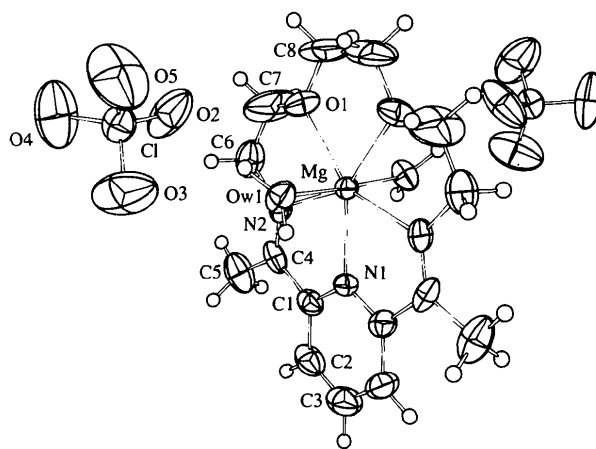


Fig. 1. ORTEPII (Johnson, 1976) plot of the title complex with displacement ellipsoids plotted at the 40% probability level.

Experimental

The title compound was synthesized from magnesium perchlorate, 2,6-diacetylpyridine and 1,8-diamino-3,6-dioxaoctane, according to the method of Cook, Fenton, Drew, McFall & Nelson (1977). Recrystallization from methanol gave yellow octahedral crystals.

Crystal data

$[\text{Mg}(\text{C}_{15}\text{H}_{21}\text{N}_3\text{O}_2)(\text{H}_2\text{O})_2](\text{ClO}_4)_2$
 $M_r = 534.59$
 Monoclinic
 $C2/c$
 $a = 15.964 (3) \text{ \AA}$
 $b = 13.603 (1) \text{ \AA}$
 $c = 10.835 (2) \text{ \AA}$
 $\beta = 97.000 (7)^\circ$
 $V = 2335.5 (6) \text{ \AA}^3$
 $Z = 4$
 $D_x = 1.520 \text{ Mg m}^{-3}$

Mo $K\alpha$ radiation
 $\lambda = 0.71073 \text{ \AA}$
 Cell parameters from 25 reflections
 $\theta = 1-25^\circ$
 $\mu = 0.370 \text{ mm}^{-1}$
 $T = 298 \text{ K}$
 Octahedral
 $0.29 \times 0.29 \times 0.29 \text{ mm}$
 Yellow

Data collection

Enraf–Nonius CAD-4 diffractometer	$R_{\text{int}} = 0.0181$
ω -2 θ scans	$\theta_{\text{max}} = 22.45^\circ$
Absorption correction: none	$h = 0 \rightarrow 17$
1589 measured reflections	$k = 0 \rightarrow 14$
1526 independent reflections	$l = -11 \rightarrow 11$
1075 observed reflections [$I > 2\sigma(I)$]	3 standard reflections
	frequency: 60 min
	intensity decay: none

Refinement

Refinement on F^2	$(\Delta/\sigma)_{\text{max}} < 0.001$
$R(F) = 0.0675$	$\Delta\rho_{\text{max}} = 0.457 \text{ e } \text{\AA}^{-3}$
$wR(F^2) = 0.1194$	$\Delta\rho_{\text{min}} = -0.342 \text{ e } \text{\AA}^{-3}$
$S = 1.062$	Atomic scattering factors
1526 reflections	from <i>International Tables for Crystallography</i> (1992),
157 parameters	Vol. C, Tables 4.2.6.8 and 6.1.1.4)
$w = 1/[\sigma^2(F_o^2) + (0.1011P)^2 + 6.6075P]$	
where $P = (F_o^2 + 2F_c^2)/3$	

Table 1. Fractional atomic coordinates and equivalent isotropic displacement parameters (\AA^2)

$$U_{\text{eq}} = (1/3)\sum_i\sum_j U_{ij}a_i^*a_j$$

	x	y	z	U_{eq}
Cl	0.31683 (10)	0.84307 (13)	0.31915 (15)	0.0561 (6)
Mg	1/2	0.7231 (2)	3/4	0.0358 (7)
OW1	0.3988 (3)	0.7266 (3)	0.6112 (4)	0.0546 (12)
O1	0.5487 (3)	0.8514 (3)	0.6615 (4)	0.0638 (13)
O2	0.3896 (4)	0.8505 (5)	0.4039 (6)	0.132 (3)
O3	0.2782 (4)	0.7527 (5)	0.3360 (9)	0.145 (3)
O4	0.3389 (5)	0.8529 (7)	0.2031 (6)	0.157 (3)
O5	0.2579 (5)	0.9145 (6)	0.3318 (8)	0.155 (3)
N1	1/2	0.5628 (5)	3/4	0.039 (2)
N2	0.5862 (3)	0.6692 (4)	0.6174 (4)	0.0480 (13)
C1	0.5493 (4)	0.5133 (4)	0.6802 (5)	0.047 (2)
C2	0.5509 (5)	0.4111 (5)	0.6790 (7)	0.070 (2)
C3	1/2	0.3615 (8)	3/4	0.080 (3)
C4	0.5990 (4)	0.5781 (5)	0.6059 (6)	0.052 (2)
C5	0.6583 (5)	0.5315 (7)	0.5249 (7)	0.084 (3)
C6	0.6275 (5)	0.7440 (7)	0.5495 (7)	0.077 (2)
C7	0.6165 (9)	0.8395 (8)	0.5988 (14)	0.160 (6)
C8	0.5296 (8)	0.9411 (6)	0.7103 (13)	0.145 (5)

Table 2. Selected geometric parameters (\AA , $^\circ$)

Mg—OW1	2.069 (4)	Mg—N1	2.182 (7)
		Mg—N2	2.231 (5)
OW1 ⁱ —Mg—OW1	177.4 (3)	O1—Mg—N2	72.5 (2)
OW1—Mg—O1 ⁱ	90.7 (2)	N1—Mg—N2	70.8 (2)
OW1—Mg—O1	87.2 (2)	OW1—Mg—N2 ⁱ	89.3 (2)
OW1—Mg—N1	91.3 (2)	O1—Mg—N2 ⁱ	145.9 (2)
O1—Mg—N1	143.2 (1)	N2—Mg—N2 ⁱ	141.6 (3)
OW1—Mg—N2	91.6 (2)		

Symmetry code: (i) $1 - x, y, \frac{3}{2} - z$.

H atoms were allowed to ride on their parent atoms with displacement factors 1.5 times those of their parent C atoms. The H atom of the water molecule was located and refined with U fixed at 0.10 \AA^2 .

Data collection: CAD-4 VAX/PC Fortran System (Enraf–Nonius, 1988). Cell refinement: CAD-4 VAX/PC Fortran System. Data reduction: Xtal3.0 (Hall & Stewart, 1990). Program(s) used to solve structure: MULTAN80 (Main, Fiske, Hull, Lessinger, Germain, Declercq & Woolfson, 1980). Pro-

gram(s) used to refine structure: SHELXL93 (Sheldrick, 1993). Molecular graphics: ORTEPII (Johnson, 1976). Software used to prepare material for publication: SHELXL93.

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Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the IUCr (Reference: TA1014). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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Dimorphism in Actinide Phosphides: Tetrakis[bis(2-dimethylphosphinoethyl)-phosphido]thorium(IV)

SIMON J. COLES, PETER G. EDWARDS,
MICHAEL B. HURSTHOUSE AND PAUL W. READ

Department of Chemistry, University of Wales, Cardiff,
PO Box 912, Cardiff CF1 3TB, Wales

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Abstract

The first example of dimorphism in actinide dialkylphosphides has been observed in the title complex, [Th(C₈H₂₀P₃)₄]. The Th atom is eight-coordinate with coordination geometry which may be described as a bi-capped trigonal prism, and is bonded to four phosphido