metal-organic papers

Bis(triphenylphosphine)iminium μ-acetato-di-μ-acetylacetonato-bis[acetylacetonato-magnesate(II)] monohydrate

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Key indicators

Single-crystal X-ray study
$T = 133$ K
Mean σ(C–C) = 0.004 Å
R factor = 0.045
wR factor = 0.105
Data-to-parameter ratio = 14.4

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

In the title compound, $(C_{36}H_{30}NP_2)[Mg_2(C_2H_3O_2)(C_5H_7O_2)_4] \cdot H_2O$, the anion involves two magnesium centres, one bridging acetate, two chelating acetylacetonates and two acetylacetonates each with one bridging and one non-bridging oxygen donor. The water molecule acts as an additional bridge *via* hydrogen bonds to two acetylacetonate O atoms.
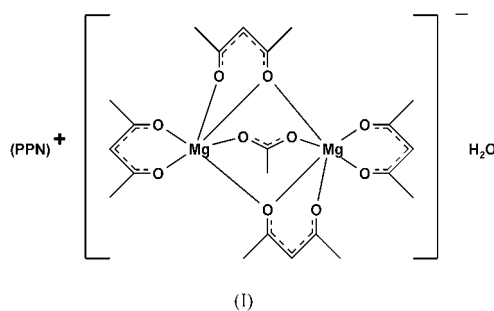
Comment

The title compound, (I), was obtained in small quantities as an unexpected crystalline product during the study of gold–acetylide complexes (see *Experimental*).

(I)

The anion (Fig. 1) involves two magnesium centres with distorted octahedral coordination. The acetate ligand bridges the metal centres. Of the four acetylacetonate ligands, two are
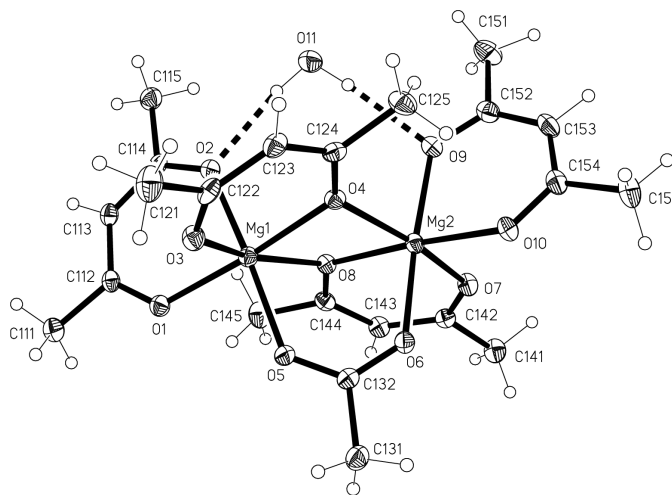

Figure 1
The anion of the title compound in the crystal. Ellipsoids are drawn at the 30% probability level. H-atom radii are arbitrary. Hydrogen bonds are indicated as dashed lines.

chelating (one at each Mg); the other two each have one oxygen (O4 and O8) that bridges both Mg centres and one (O2 and O7) that coordinates to only one Mg. The water molecule bridges the acetylacetonate atoms O2 and O9 by hydrogen bonding. The Mg⋯Mg distance is 3.1925 (12) Å.

The related structures of hexakis(acetylacetonato)-trimagnesium(II) and hexakis(ethylacetoacetato)trimagnesium(II) have been described by Weiß *et al.* (1985) and Petrov *et al.* (1992).

Experimental

An equimolar mixture of 1,2-diethynylbenzene and (PPN)[Au-(acac)$_2$] [PPN is bis(triphenylphosphine)iminium] in acetone was stirred at room temperature for 3.5 h and then filtered to remove traces of gold. The resulting solution was concentrated to dryness under reduced pressure; the residue was washed with diethyl ether (4 × 20 ml), filtered and air dried. Recrystallization from dichloromethane and diethyl ether yielded the expected organometallic gold polymer (24% yield), which was characterized by NMR and IR spectroscopy. However, attempts to grow single crystals of this product from a 1:1 mixture of dichloromethane and acetone (as solvent) and diethyl ether (as precipitant) yielded a crystal of the unexpected title compound (as well as amorphous material). Presumably the magnesium came from solvent drying agents. The acetate could have arisen from acac or acetone.

Crystal data

$(C_{36}H_{30}NP_2)[Mg_2(C_2H_3O_2)\text{-}$
$(C_5H_7O_2)_4]\cdot H_2O$
$M_r = 1060.65$
Monoclinic, $P2_1/n$
$a = 19.945 (2)$ Å
$b = 14.069 (2)$ Å
$c = 20.762 (3)$ Å
$\beta = 106.209 (6)°$
$V = 5594.6 (13)$ Å^3
$Z = 4$

$D_x = 1.259$ Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 7821 reflections
$\theta = 2.5–25°$
$\mu = 0.16$ mm^{-1}
$T = 133 (2)$ K
Plate, colourless
$0.32 \times 0.18 \times 0.03$ mm

Data collection

Bruker SMART 1000 CCD diffractometer
ω and φ scans
Absorption correction: none
57401 measured reflections
9872 independent reflections

5934 reflections with $I > 2\sigma(I)$
$R_{int} = 0.087$
$\theta_{max} = 25.0°$
$h = -23 \to 23$
$k = -16 \to 16$
$l = -24 \to 24$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.045$
$wR(F^2) = 0.105$
$S = 0.92$
9872 reflections
684 parameters

H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.05P)^2]$
   where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.001$
$\Delta\rho_{max} = 0.47$ e Å^{-3}
$\Delta\rho_{min} = -0.30$ e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

Mg1—O5	2.0220 (19)	Mg2—O10	2.0243 (19)
Mg1—O3	2.0445 (19)	Mg2—O7	2.0320 (18)
Mg1—O1	2.0501 (19)	Mg2—O6	2.0588 (19)
Mg1—O2	2.0560 (19)	Mg2—O9	2.0692 (19)
Mg1—O4	2.0926 (18)	Mg2—O8	2.0904 (18)
Mg1—O8	2.1926 (18)	Mg2—O4	2.1069 (18)
O5—Mg1—O3	93.44 (8)	O7—Mg2—O8	87.49 (7)
O5—Mg1—O1	87.83 (8)	O6—Mg2—O8	89.35 (7)
O3—Mg1—O1	88.78 (8)	O9—Mg2—O8	96.18 (8)
O5—Mg1—O2	168.66 (8)	O10—Mg2—O4	102.78 (7)
O3—Mg1—O2	95.62 (8)	O7—Mg2—O4	166.88 (8)
O1—Mg1—O2	85.55 (8)	O6—Mg2—O4	90.07 (7)
O5—Mg1—O4	94.69 (7)	O9—Mg2—O4	90.11 (7)
O3—Mg1—O4	85.87 (7)	O8—Mg2—O4	79.56 (7)
O1—Mg1—O4	174.21 (8)	C112—O1—Mg1	130.25 (17)
O2—Mg1—O4	92.77 (7)	C114—O2—Mg1	129.52 (17)
O5—Mg1—O8	84.64 (7)	C122—O3—Mg1	129.24 (17)
O3—Mg1—O8	163.10 (8)	C124—O4—Mg1	126.47 (16)
O1—Mg1—O8	107.88 (7)	C124—O4—Mg2	134.34 (16)
O2—Mg1—O8	88.64 (7)	C132—O5—Mg1	131.24 (17)
O4—Mg1—O8	77.58 (7)	Mg1—O4—Mg2	98.97 (7)
O10—Mg2—O7	90.26 (8)	C142—O6—Mg2	129.89 (17)
O10—Mg2—O6	87.71 (8)	C142—O7—Mg2	128.14 (17)
O7—Mg2—O6	91.88 (7)	C144—O8—Mg2	125.75 (16)
O10—Mg2—O9	86.79 (8)	C144—O8—Mg1	128.52 (16)
O7—Mg2—O9	89.21 (7)	Mg2—O8—Mg1	96.35 (7)
O6—Mg2—O9	174.40 (8)	C152—O9—Mg2	127.05 (18)
O10—Mg2—O8	176.24 (8)	C154—O10—Mg2	128.98 (18)

Table 2
Hydrogen-bonding geometry (Å, °).

$D—H\cdots A$	$D—H$	$H\cdots A$	$D\cdots A$	$D—H\cdots A$
O11—H01⋯O2	0.87 (2)	1.98 (2)	2.851 (3)	172 (3)
O11—H02⋯O9	0.87 (2)	2.03 (2)	2.883 (3)	169 (3)
C62—H62⋯O1^i	0.95	2.60	3.362 (3)	138
C63—H63⋯O3^i	0.95	2.65	3.566 (3)	163
C25—H25⋯O6^{ii}	0.95	2.64	3.428 (3)	140
C54—H54⋯O11^{iii}	0.95	2.67	3.433 (4)	138

Symmetry codes: (i) $\frac{3}{2} - x, \frac{1}{2} + y, \frac{1}{2} - z$; (ii) $\frac{3}{2} - x, y - \frac{1}{2}, \frac{1}{2} - z$; (iii) $1 - x, 2 - y, -z$.

Water H atoms were refined freely, but with O—H distances restrained to be equal. Methyl H atoms were located in difference syntheses, idealized (C—H = 0.98 Å and H—C—H 109.5°) and refined on the basis of rigid groups allowed to rotate but not tip. Other H atoms were included using a riding model with fixed C—H bond lengths of 0.95 Å; U_{iso}(H) values were fixed at $1.2U_{eq}$ of the parent atom. To improve refinement stability, restraints (DELU and SIMU) were applied to the displacement parameters of all non-H atoms.

Data collection: *SMART* (Bruker, 1998); cell refinement: *SAINT* (Bruker, 1998); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS*97 (Sheldrick, 1990); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); molecular graphics: *XP* (Siemens, 1994); software used to prepare material for publication: *SHELXL*97.

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