metal-organic papers

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Key indicators

Single-crystal X-ray study
$T = 115$ K
Mean σ(O–C) = 0.003 Å
R factor = 0.036
wR factor = 0.085
Data-to-parameter ratio = 19.4

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

Hexaaquanickel(II) bis(dimethyl phosphate)

In the title compound, $[Ni(H_2O)_6][O_2P(OCH_3)_2]_2$, the hexaaquanickel(II) dication (Ni site symmetry $\bar{1}$) and the organic anion interact by way of intermolecular O—H$\cdots$O hydrogen bonds.

Comment

Recently, we reported the syntheses and crystal structures of $[Cu_2(DMP)_4(DMSO)]_n$ (Rafizadeh *et al.*, 2005) and $[UO_2(\mu\text{-}DMP)_4(DMSO)]_n$ (Rafizadeh *et al.*, 2006), where DMP is dimethyl phosphate, $[O_2P(OCH_3)_2]^-$, and DMSO is dimethyl sulfoxide. In these complexes, DMP acts as an O-atom donor ligand, forming coordination polymers in the solid state. As an extension of these studies, we now report the synthesis and structure of the title compound, (I), in which the DMP does not bond to the metal.

(I)
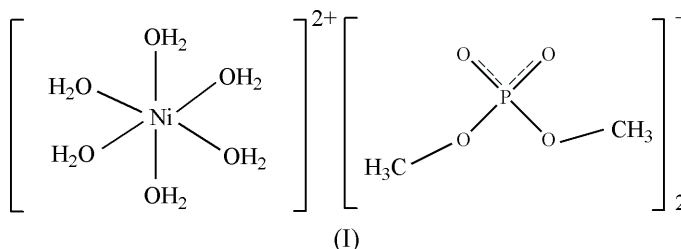
In the title complex, the Ni[II] cation (site symmetry $\bar{1}$) has an octahedral coordination comprising six water O atoms. The P atom of the anion adopts a disorted tetrahedral geometry (Fig. 1). The almost equal P1—O1 and P1—O2 bond lengths (Table 1) imply delocalization of the negative charge. The Ni—O and P—O bond lengths (Table 1) are in agreement with the corresponding bond lengths in $[Ni(H_2O)_6](H_2PO_2)_2$
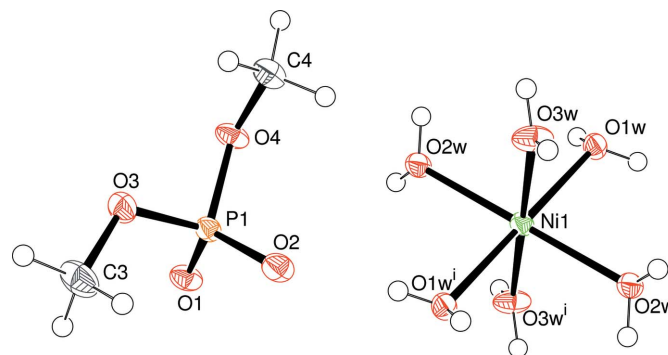

Figure 1
The structure of (I). Displacement ellipsoids are drawn at the 50% probability level (arbitrary spheres for the H atoms). [Symmetry code: (i) $1 - x$, $-y$, $1 - z$.]
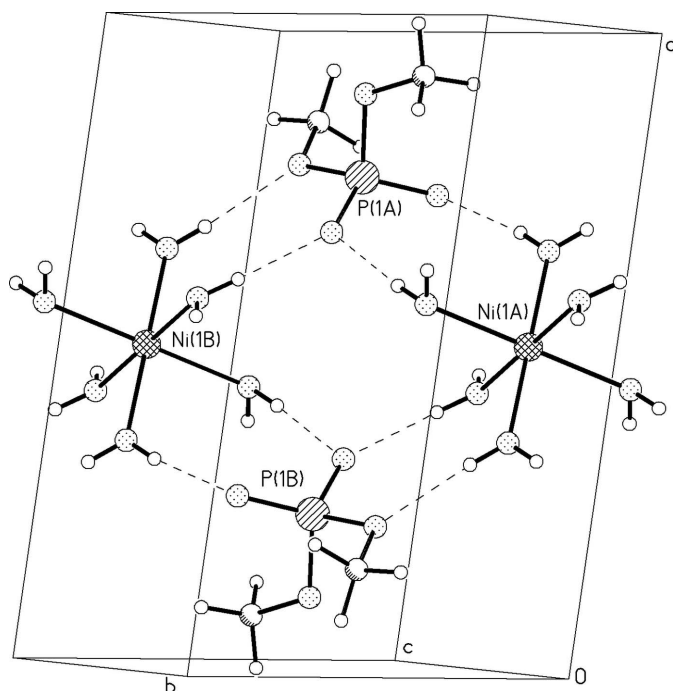
Figure 2
Packing diagram for (I). Hydrogen bonds are shown as dashed lines.

(Kuratieva *et al.*, 2003). The six water H atoms all participate in hydrogen bonds (Table 2): five of the acceptors are O atoms of the DMP anions and one is another water molecule (Fig. 2).

Experimental

Trimethyl phosphate (0.97 g, 0.8 ml, 6.72 mmol) was added to a solution of NiCl$_2$·6H$_2$O (0.81 g, 3.36 mmol) in tetrahydrofuran (30 ml) and the resulting green solution was refluxed at 313 K for 1 h under a nitrogen atmosphere. This solution was left to evaporate slowly at room temperature. After three weeks, green prismatic crystals of (I) were isolated (yield 1.02 g, 72.8%, m.p. 375 K).

Crystal data

[Ni(H$_2$O)$_6$](C$_2$H$_6$O$_4$P)$_2$	$Z = 2$
$M_r = 416.88$	$D_x = 1.724$ Mg m^{-3}
Monoclinic, $P2_1/c$	Mo $K\alpha$ radiation
$a = 12.450$ (2) Å	$\mu = 1.47$ mm^{-1}
$b = 7.1208$ (14) Å	$T = 115$ (2) K
$c = 9.7342$ (19) Å	Prism, green
$\beta = 111.492$ (4)°	$0.20 \times 0.10 \times 0.05$ mm
$V = 803.0$ (3) Å^3	

Data collection

Bruker SMART1000 CCD diffractometer	5155 measured reflections
	1881 independent reflections
ω scans	1346 reflections with $I > 2\sigma(I)$
Absorption correction: multi-scan ($SADABS$; Sheldrick, 1998)	$R_{int} = 0.028$
	$\theta_{max} = 28.0°$
$T_{min} = 0.745$, $T_{max} = 0.928$	

Refinement

Refinement on F^2	H-atom parameters constrained
$R[F^2 > 2\sigma(F^2)] = 0.036$	$w = 1/[\sigma^2(F_o^2) + (0.0425P)^2]$
$wR(F^2) = 0.085$	where $P = (F_o^2 + 2F_c^2)/3$
$S = 1.09$	$(\Delta/\sigma)_{max} < 0.001$
1881 reflections	$\Delta\rho_{max} = 0.96$ e Å^{-3}
97 parameters	$\Delta\rho_{min} = -0.33$ e Å^{-3}

Table 1
Selected bond lengths (Å).

Ni1—O2W	2.0313 (15)	P1—O2	1.4982 (16)
Ni1—O3W	2.0351 (17)	P1—O4	1.5862 (16)
Ni1—O1W	2.0734 (15)	P1—O3	1.5908 (18)
P1—O1	1.4920 (17)		

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

D—H$\cdots A$	D—H	H$\cdots A$	$D\cdots A$	D—H$\cdots A$
O1W—H2$\cdots$O1^i	0.83	1.88	2.708 (2)	172
O1W—H3$\cdots$O2^{ii}	0.77	1.93	2.691 (2)	172
O2W—H4$\cdots$O1W^{iii}	0.70	2.16	2.857 (3)	176
O2W—H5$\cdots$O1^{iv}	0.80	1.87	2.668 (2)	178
O3W—H6$\cdots$O4^{iv}	0.77	2.01	2.781 (2)	174
O3W—H7$\cdots$O2^v	0.78	1.92	2.694 (2)	170

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

The O-bound H atoms were located in difference maps and refined as riding in their as-found relative positions, with U_{iso}(H) = $1.2U_{eq}$(O). The C-bound H atoms were positioned geometrically (C—H = 0.98 Å) and refined using a riding-model approximation, with U_{iso}(H) = $1.5U_{eq}$(C).

Data collection: *SMART* (Bruker, 1998); cell refinement: *SAINT* (Bruker, 1998); data reduction: *SAINT*; program(s) used to solve structure: *SHELXTL* (Sheldrick, 1998); program(s) used to refine structure: *SHELXTL*; molecular graphics: *SHELXTL*; software used to prepare material for publication: *SHELXTL*.

References

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