S(2)	−0.05930 (5)	0.11870 (8)	0.09050 (5)	3.14 (3)
O(1)	0.1088 (1)	0.5576 (2)	0.1334 (1)	3.7 (1)
O(2)	−0.1284 (1)	−0.1172 (2)	0.0877 (1)	4.3 (1)
N(1)	−0.0036 (1)	0.4579 (2)	0.1391 (2)	2.9 (1)
N(2)	−0.0985 (1)	−0.0124 (2)	0.2216 (2)	2.9 (1)
C(1)	−0.0448 (3)	0.5732 (4)	0.0911 (3)	4.1 (2)
C(2)	0.0720 (2)	0.4571 (3)	0.1605 (2)	2.8 (1)
C(3)	0.1926 (2)	0.5561 (4)	0.1448 (3)	4.0 (2)
C(4)	0.2091 (3)	0.6628 (4)	0.0880 (3)	4.3 (2)
C(5)	−0.1254 (3)	−0.1309 (4)	0.2577 (3)	4.2 (2)
C(6)	−0.0987 (2)	−0.0118 (3)	0.1384 (2)	2.9 (1)
C(7)	−0.1334 (3)	−0.1248 (4)	−0.0075 (2)	4.4 (2)
C(8)	−0.2107 (3)	−0.0794 (6)	−0.0631 (3)	6.8 (3)

Table 2. *Selected geometric parameters* (Å, °)

Starred atoms are related to the corresponding unstarred atoms by a twofold axis.

Cu(1)—Cu(1*)	2.8270 (7)	S(2)—C(6)	1.761 (3)
Cu(1)—Cu(2)	2.5974 (5)	N(1)—C(1)	1.468 (4)
Cu(1)—Cu(2*)	2.6344 (5)	N(1)—C(2)	1.282 (3)
Cu(1)—S(1*)	2.2953 (8)	N(2)—C(5)	1.467 (4)
Cu(1)—S(2)	2.2706 (9)	N(2)—C(6)	1.283 (3)
Cu(1)—N(1)	1.982 (2)	O(1)—C(2)	1.344 (3)
Cu(2)—Cu(2*)	2.8628 (7)	O(1)—C(3)	1.444 (4)
Cu(2)—S(1*)	2.2680 (9)	O(2)—C(6)	1.345 (3)
Cu(2)—S(2*)	2.2974 (9)	O(2)—C(7)	1.450 (4)
Cu(2)—N(2)	1.983 (2)	C(3)—C(4)	1.479 (5)
S(1)—C(2)	1.766 (3)	C(7)—C(8)	1.467 (6)
N(1)—Cu(1)—S(2)	116.44 (7)	O(1)—C(2)—S(1)	119.0 (2)
N(1)—Cu(1)—S(1*)	119.33 (7)	C(2)—O(1)—C(3)	121.8 (2)
S(2)—Cu(1)—S(1*)	124.22 (3)	Cu(2)—Cu(1)—Cu(2*)	66.34 (2)
N(2)—Cu(2)—S(1*)	119.19 (7)	Cu(2)—Cu(1)—Cu(1*)	57.93 (1)
N(2)—Cu(2)—S(2*)	117.23 (7)	Cu(2)—Cu(1*)—Cu(1)	56.66 (1)
S(1*)—Cu(2)—S(2*)	123.57 (3)	Cu(1)—Cu(2)—Cu(1*)	65.41 (2)
C(2)—N(1)—C(1)	117.8 (3)	Cu(1)—Cu(2)—Cu(2*)	57.45 (1)
C(1)—N(1)—Cu(1)	121.6 (2)	Cu(1)—Cu(2*)—Cu(2)	56.21 (1)
C(2)—N(1)—Cu(1)	120.6 (2)	Cu(1*)—S(1)—Cu(2*)	69.39 (3)
N(1)—C(2)—O(1)	118.5 (3)	Cu(1)—S(2)—Cu(2*)	70.44 (3)
N(1)—C(2)—S(1)	122.5 (2)		
Cu(2)—Cu(1)—Cu(1*)—Cu(2*)	81.11 (2)		
Cu(1)—Cu(1*)—Cu(2)—Cu(2*)	66.06 (2)		
Cu(1)—Cu(2)—Cu(2*)—Cu(1*)	80.40 (2)		
Cu(2)—Cu(1)—Cu(2*)—Cu(1*)	64.31 (2)		
Cu(1)—N(1)—C(2)—O(1)	175.2 (2)		
S(1)—C(2)—N(1)—C(1)	175.0 (3)		
Cu(1)—N(1)—C(2)—S(1)	−6.0 (3)		
Cu(2)—N(2)—C(6)—O(2)	−178.4 (2)		
S(2)—C(6)—N(2)—C(5)	−173.9 (3)		
Cu(2)—N(2)—C(6)—S(2)	3.4 (3)		

The unit cell was found to be *C*-centred monoclinic. Based on the systematic absences (*hkl*, $h + k = 2n$; *h*0*l*, $l = 2n$), packing considerations, a statistical analysis of intensity distribution and the successful solution and refinement of the structure, the space group was determined to be *C*2/*c*. The structure was solved by direct methods (Gilmore, 1990) and expanded using Fourier techniques (Beurskens *et al.*, 1992).

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Lists of structure factors, anisotropic displacement parameters and H-atom coordinates have been deposited with the IUCr (Reference: OH1057). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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[{PtCl(dmso)}$_2$(μ-OH)$_2$]

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Abstract

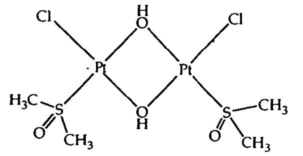
The crystal structure of di-μ-hydroxo-bis[chloro(dimethyl sulfoxide)platinum(II)], $[Pt_2Cl_2(\mu\text{-}OH)_2(C_2H_6OS)_2]$, has been determined from X-ray intensity data. The compound contains a hydroxo-bridged dinuclear *cis* complex, *i.e.* the Cl atoms are in *cis* positions in $[Cl(dmso)Pt(OH)_2Pt(dmso)Cl]$. Each Pt atom has pseudo square-planar coordination composed of one dmso molecule, one Cl atom and two hydroxo groups. The complexes are linked by a hydrogen-bond network parallel to the *ab* plane.

Comment

The title compound (I) was obtained in an attempt to synthesize $[PtCl_2(dmso)(H_2O)]$ in aqueous solution by abstracting one Cl^- in $K[PtCl_3(dmso)]$ by AgF in the following way: 0.03 g AgF in 5 ml water was added to 0.17 g K[PtCl$_3$(dmso)] in 15 ml water (molar ratio 1:1); the solution was stirred for 5 h and precipitated AgCl was filtered off. The beaker was covered by parafilm and after a few weeks crystals were obtained. Suggested reactions are:

$$[\mathrm{PtCl_3(dmso)}]^- + \mathrm{H_2O} + \mathrm{Ag}^+ \rightarrow [\mathrm{PtCl_2(dmso)(H_2O)}] + \mathrm{AgCl}(s)$$

$$2[\mathrm{PtCl_2(dmso)(H_2O)}] + 2\mathrm{F}^- \rightarrow [\{\mathrm{PtCl(dmso)}\}_2(\mu\text{-OH})_2] + 2\mathrm{HF} + 2\mathrm{Cl}^-$$



(I)

The synthesis was carried out with both AgF and AgNO$_3$, but only AgF gave crystals of the title compound. In order to exclude the possibility of the presence of an F atom in the compound it was dissolved in dimethylformamide for an ^{19}F NMR experiment. A scan was made between −325 and 565 p.p.m. with respect to CFCl$_3$, which was used as a reference for the ^{19}F signal. No ^{19}F peak was found in this interval. The title compound may also be synthesized by treating an aqueous solution of K[Pt(dmso)Cl$_3$] with diethylamine or triethylamine (Kukushkin, Pakhomova & Kalyukokova, 1974). Both F$^-$ and amines are weak bases and thus promote the formation of hydroxo species in solution, *i.e.* a hydroxo-bridged complex.

The compound is composed of neutral hydroxo-bridged dinuclear complexes, [{PtCl(dmso)}$_2$(μ-OH)$_2$], with the Cl atoms in *cis* positions (Fig. 1).

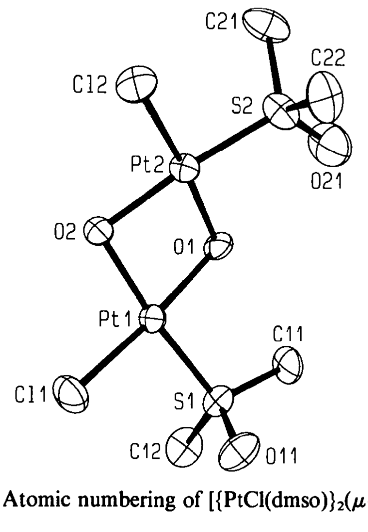


Fig. 1. Atomic numbering of [{PtCl(dmso)}$_2$(μ-OH)$_2$].

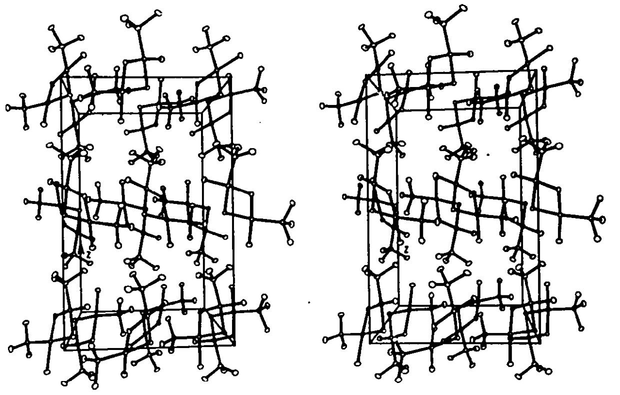


Fig. 2. Stereoscopic view of the crystal packing of [{PtCl(dmso)}$_2$(μ-OH)$_2$].

Table 1. *Fractional atomic coordinates and equivalent isotropic displacement parameters* (Å^2)

$$U_{\mathrm{eq}} = (1/3)\Sigma_i\Sigma_j U_{ij}a_i^*a_j^*\mathbf{a}_i.\mathbf{a}_j.$$

	x	y	z	U_{eq}
Pt(1)	0.39283 (6)	0.14494 (5)	0.03570 (3)	0.0216 (2)
Pt(2)	0.35819 (6)	0.00699 (5)	−0.09738 (3)	0.0238 (2)
O(1)	0.4731 (10)	0.1228 (8)	−0.0598 (6)	0.026 (4)
O(2)	0.3357 (10)	−0.0123 (8)	0.0103 (5)	0.024 (4)
Cl(1)	0.2961 (5)	0.1543 (4)	0.1402 (2)	0.040 (2)
Cl(2)	0.2148 (4)	−0.1165 (4)	−0.1278 (3)	0.035 (2)
S(1)	0.4607 (4)	0.3118 (3)	0.0490 (2)	0.025 (1)
S(2)	0.4019 (4)	0.0429 (4)	−0.2065 (2)	0.033 (2)
O(11)	0.3760 (11)	0.4002 (9)	0.0581 (7)	0.039 (5)
C(11)	0.5522 (20)	0.3405 (15)	−0.0226 (9)	0.041 (7)
C(12)	0.5556 (17)	0.3217 (14)	0.1207 (10)	0.036 (7)
O(21)	0.4974 (14)	0.1243 (12)	−0.2165 (8)	0.058 (6)
C(21)	0.4403 (18)	−0.0808 (16)	−0.2530 (10)	0.043 (7)
C(22)	0.2785 (18)	0.0928 (16)	−0.2527 (10)	0.045 (8)

Table 2. *Selected geometric parameters* (Å, °)

Pt(1)—O(1)	2.049 (11)	Pt(2)—O(1)	2.050 (11)
Pt(1)—O(2)	2.065 (10)	Pt(2)—O(2)	2.072 (10)
Pt(1)—S(1)	2.174 (4)	Pt(2)—S(2)	2.174 (4)
Pt(1)—Cl(1)	2.274 (5)	Pt(2)—Cl(2)	2.295 (5)
S(1)—O(11)	1.45 (1)	S(2)—O(21)	1.47 (1)
S(1)—C(11)	1.75 (2)	S(2)—C(21)	1.78 (2)
S(1)—C(12)	1.75 (2)	S(2)—C(22)	1.77 (2)
Pt(1)···Pt(2)	3.0496 (9)	O(1)···O(2)	2.63 (2)
Pt(1)···Pt(2$^{\mathrm{i}}$)	3.593 (1)		
Cl(1)—Pt(1)—S(1)	91.6 (2)	Cl(2)—Pt(2)—S(2)	93.2 (2)
S(1)—Pt(1)—O(1)	93.5 (3)	S(2)—Pt(2)—O(1)	92.7 (3)
O(1)—Pt(1)—O(2)	79.5 (4)	O(1)—Pt(2)—O(2)	79.3 (4)
O(2)—Pt(1)—Cl(1)	95.4 (3)	O(2)—Pt(1)—Cl(2)	94.9 (3)
Cl(1)—Pt(1)—O(1)	174.8 (3)	Cl(2)—Pt(1)—O(1)	172.8 (3)
S(1)—Pt—O(2)	172.9 (3)	S(2)—Pt—O(2)	171.9 (3)
Pt(1)—S(1)—O(11)	116.9 (5)	Pt(2)—S(2)—O(21)	115.1 (6)
Pt(1)—S(1)—C(11)	108.0 (6)	Pt(2)—S(2)—C(21)	111.1 (7)
Pt(1)—S(1)—C(12)	112.1 (6)	Pt(2)—S(2)—C(22)	110.7 (7)
O(11)—S(1)—C(11)	110.4 (9)	O(21)—S(2)—C(21)	107.9 (9)
O(11)—S(1)—C(12)	106.0 (8)	O(21)—S(2)—C(22)	107.6 (9)
C(11)—S(1)—C(12)	102 (1)	C(21)—S(2)—C(22)	103.7 (9)
Pt(1)—O(1)—Pt(2)	96.1 (5)	Pt(1)—O(2)—Pt(2)	95.0 (4)
Hydrogen bonds			
O(1)···O(2$^{\mathrm{i}}$)	2.735 (17)	O(2)···O(11$^{\mathrm{ii}}$)	2.801 (18)
O(1$^{\mathrm{i}}$)···O(2)···O(11$^{\mathrm{ii}}$)	113.7 (5)		

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

Table 3. *Comparison of structural data* (Å, °) *for some hydroxo-bridged complexes*

	Symmetry	Pt···Pt	Pt—O	*trans*-partner		O—Pt—O	Pt—O—Pt	Reference
$[\{Pt(NH_3)_2\}_2(\mu\text{-}OH)_2](NO_3)_2$	$\bar{1}$	3.085 (1)	2.03 (1)	N	NH_3	81.3 (4)	98.7 (4)	(*a*)
			2.03	N	NH_3			
$K_2[\{Pt(NO_2)_2\}_2(\mu\text{-}OH)_2].1\frac{1}{2}H_2O$	1	3.167 (1)	2.02 (2)	N	NO_2	81 (1)	99 (1)	(*b*)
			2.05 (2)	N	NO_2			
			2.06 (2)	N	NO_2	80 (1)	99 (1)	
			2.04 (2)	N	NO_2			
$[\{Pt(C_4H_8SO)_2\}_2(\mu\text{-}OH)_2](NO_3)_2$	$\bar{1}$	3.137 (3)	2.045 (5)	S	tmso	80.0 (2)	100.0 (3)	(*c*)
			2.050 (5)	S	tmso			
$[\{Pt(dmso)_2\}_2(\mu\text{-}OH)_2](ClO_4)_2$	$\bar{1}$	3.145 (3)	2.031 (9)	S	dmso	79.6 (4)	100.4 (4)	(*d*)
			2.064 (8)	S	dmso			
$[\{Pt(NH_3)_2\}_2(\mu\text{-}OH)_2](CO_3)_2.2H_2O$	1	3.104 (1)	2.02 (1)	N	NH_3	81.9 (5)	98.8 (5)	(*e*)
			2.04 (1)	N	NH_3			
			2.06 (1)	N	NH_3	80.6 (5)	98.5 (5)	
			2.07 (1)	N	NH_3			
$[\{Pt(dmso)_2\}_2(\mu\text{-}OH)_2](BF_4)_2$	$\bar{1}$	3.144 (1)	2.052 (14)	S	dmso	80.0 (6)	100.0 (6)	(*f*)
			2.054 (13)	S	dmso			
$[\{PtCl(dmso)\}_2(\mu\text{-}OH)_2]$	1	3.0496 (9)	2.049 (11)	Cl		79.5 (4)	96.1 (5)	(*g*)
			2.065 (10)	S	dmso			
			2.050 (11)	Cl		79.3 (4)	95.0 (4)	
			2.072 (10)	S	dmso			
$[\{Pt(dmso)_2\}_2(\mu\text{-}OH)_2](CF_3SO_3)_2$	$\bar{1}$	3.1417 (9)	2.063 (9)	S	dmso	81.0 (4)	99.0 (4)	(*f*)
			2.068 (9)	S	dmso			
$[NBu_4]_2[\{Pt(C_6F_5)_2\}_2(\mu\text{-}OH)_2]$	$\bar{1}$	3.211 (1)	2.07 (2)	C	C_6F_5	78.6 (5)	101.4 (6)	(*h*)
			2.08 (2)	C	C_6F_5			
$[\{Pt(PMe_3)_2\}_2(\mu\text{-}OH)_2](NO_3)_2$	$\bar{1}$	3.261 (1)	2.08 (1)	P	PMe_3	75.9 (4)	104.1 (4)	(*i*)
			2.06 (1)	P	PMe_3			
$[\{Pt(dppf)\}_2(\mu\text{-}OH)_2](BF_4)_2.2CH_2Cl_2.2H_2O$	$\bar{1}$	3.227 (1)	2.100 (11)	P	dppf	79.4 (4)	100.6 (7)	(*j*)
			2.092 (11)	P	dppf			
$[\{Pt(PEt_3)_2\}_2(\mu\text{-}OH)_2](BF_4)_2$	1	3.121 (1)	2.166 (13)	P	PEt_3	79.4 (6)	92 (2)	(*k*)
			2.097 (13)	P	PEt_3			
			2.167 (14)	P	PEt_3	79.3 (6)	96 (2)	
			2.101 (13)	P	PEt_3			

References: (*a*) Faggiani, Lippert, Lock & Rosenberg (1977); (*b*) Min, Larsen, Emerson & Aboot (1990); (*c*) Rochon & Guay (1987); (*d*) Rochon, Kong & Melanson (1985); (*e*) Lippert, Lock, Rosenberg & Zvagulis (1978); (*f*) Ericson, Lövqvist, Norén & Oskarsson (1992); (*g*) this work; (*h*) López, Ruiz, Garciá, Vincente, Martí, Hermoso, Vegas & Martínez-Ripoll (1992); (*i*) Trovó, Bandoli, Casellato, Corain, Nicolini & Longato (1990); (*j*) Longato, Pilloni, Valle & Corain (1988); (*k*) Bushnell (1978).

The coordination geometry around each Pt atom is distorted square planar with one chlorine, one dmso bonded *via* the S atom, and two bridging-hydroxo groups. The least-squares planes were calculated with unit weights. The deviations from the two $SClPtO_2$ planes are: Pt(1) 0.0131 (6), S(1) −0.012 (4), Cl(1) 0.006 (5), O(1) 0.01 (1), O(2) −0.01 (1) Å; Pt(2) 0.0259 (6), S(2) −0.082 (5), Cl(2) 0.028 (5), O(1) −0.11 (1), O(2) 0.14 (1) Å. The angle between the two $SClPtO_2$ least-squares coordination planes (33°) is about the same as that observed in $[Pt_2(OH)_2(PEt_3)_4](BF_4)_2$ [36° (Bushnell, 1978)]. Comparative structural data for the title compound and other bis(μ-hydroxo)platinum(II) complexes are reported in Table 3. Out of the 12 complexes, eight exhibit a center of symmetry, *i.e.* the four-membered ring PtO_2Pt is planar due to symmetry. The Pt···Pt distance in the title compound [3.050 (1) Å] is the shortest observed so far for this type of compound, but it is too long to be regarded as a metal–metal bond. The short Pt···Pt distance is a consequence of the large tilt angle between the two coordination planes, the small Pt—O—Pt angles and the fairly short Pt—O distances (Table 3). These distances are 2.05 (1) (*trans* Cl) and 2.07 (1) Å (*trans* dmso). The Pt—O bond *trans* to dmso is somewhat longer than the one *trans* to Cl; it could be argued that this is caused by a larger *trans* influence of the sulfoxide as compared to Cl, but the difference is hardly significant. However, as expected, it is shown in Table 3 that phosphines have a somewhat larger *trans* influence than Cl^- and ligands with S and N as donor atoms.

The Pt—S distances [2.174 (4) Å] are smaller than those (2.189–2.223 Å) found in hydroxo-bridged complexes with two dmso molecules coordinated to each Pt atom (Ericson, Lövqvist, Norén & Oskarsson, 1992; Rochon, Kong & Melanson 1985); this is probably due to a larger *cis* influence of S, compared to Cl, on the Pt—S bond length.

The dinuclear $[\{PtCl(dmso)\}_2(\mu\text{-}OH)_2]$ complexes are most probably hydrogen bonded into pairs by hydrogen bonds between the hydroxo groups $[O(1)^{i}\cdots O(2) = 2.74\ (2)$ Å (Table 2)]. These centrosymmetric pairs are further linked by hydrogen bonds between one of the hydroxo groups and an O atom in the dmso molecule $[O(2)\cdots O(11)^{ii} = 2.80\ (2)$ Å], forming layers parallel to the *ab* plane (Fig. 2). The presence of hydrogen bonds of this strength (~2.7–2.8 Å) is supported by an IR spectrum, which shows a broad intense peak at 3200 cm^{-1} (Hamilton & Ibers, 1968).

Experimental

Crystal data

$[Pt_2Cl_2(OH)_2(C_2H_6OS)_2]$	Mo $K\alpha$ radiation
$M_r = 651.34$	$\lambda = 0.7107$ Å

Orthorhombic
Pbca
$a = 11.487\ (2)$ Å
$b = 12.067\ (1)$ Å
$c = 18.967\ (3)$ Å
$V = 2629.0\ (5)$ Å^3
$Z = 8$
$D_x = 3.29$ Mg m^{-3}

Cell parameters from 25 reflections
$\theta = 6.4$–$22.6°$
$\mu = 22.19$ mm^{-1}
Room temperature
Prism
0.2 × 0.18 × 0.12 mm
Yellow

Data collection

Enraf-Nonius CAD-4 diffractometer
ω-2θ scans
Absorption correction: by integration from crystal shape
$T_{\min} = 0.098$, $T_{\max} = 0.218$
2571 measured reflections
2511 independent reflections

1497 observed reflections $[I > 2\sigma(I)]$
$R_{\rm int} = 0.0082$ (based on standard reflections)
$\theta_{\max} = 28°$
$h = 0 \rightarrow 14$
$k = 0 \rightarrow 14$
$l = 0 \rightarrow 23$
2 standard reflections
frequency: 120 min
intensity variation: <2.5%

Refinement

Refinement on F
$R = 0.038$
$wR = 0.048$
$S = 1.09$
1497 reflections
118 parameters
H-atom parameters not refined
$w = 1/[\sigma^2(F)/4|F_o|^2 + 0.03|F_o|^2 + 8.0]$
$(\Delta/\sigma)_{\max} = 0.06$

$\Delta\rho_{\max} = 2.27$ e Å^{-3}
$\Delta\rho_{\min} = -1.89$ e Å^{-3}
Extinction correction: Becker & Coppens (1975)
Extinction coefficient: 0.1013×10^5
Atomic scattering factors from *International Tables for X-ray Crystallography* (1974, Vol. IV)

The structure was solved by direct and difference Fourier methods and refined by full-matrix least-squares calculations employing programs amended by Lundgren (1982). The H atoms could not be located in the difference Fourier maps and they were not included in the structure-factor calculations.

Financial support from the Swedish Natural Science Research Council is gratefully acknowledged. We are much obliged to Dr Lars A. Bengtsson for help with the NMR experiment.

Lists of structure factors, anisotropic displacement parameters and complete geometry have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 71796 (27 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: AB1105]

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Hexakis(*tert*-butylammonium) Heptamolybdate(VI)–Water (1/7)

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Abstract

The asymmetric unit of $[(CH_3)_3CNH_3]_6[Mo_7O_{24}].7H_2O$ consists of a heptamolybdate anion, six *tert*-butylammonium cations and seven water molecules. The crystal packing is stabilized by electrostatic interactions and an extensive network of hydrogen contacts involving the O atoms from the polyanion, the water molecules and the N atoms, and some C atoms of the cations. Some of these hydrogen bonds may play an important role in the UV-induced photochromism of this compound.

Comment

This work was undertaken in order to obtain a deeper insight into the effect of the cation on the crystal packing, the strength of the intermolecular contacts and the chemical properties of organoammonium polyoxomolybdates. Several studies on polyoxometallates of V, Mo and W have been carried out in recent years because of their applications in photochemistry, catalysis and biomedicine

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