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Synthesis and Structure of $[H(DMSO)_2]_2[OsX_6]$ (X = Cl, Br)

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Abstract—The reactions of $H_2OsX_6 \cdot nH_2O$ (X = Cl, Br) with dimethyl sulfoxide (DMSO) or DMSO-d$_6$ in acetone afford the osmium(IV) complexes: $[H(DMSO\text{-}O)_2]_2[OsCl_6]$ (**I**), $[H(DMSO\text{-}O)_2]_2[OsBr_6]$ (**II**), $[H(DMSO\text{-}d_6)_2]_2[OsCl_6]$ (**Ia**), and $[H(DMSO\text{-}d_6)_2]_2[OsBr_6]$ (**IIa**). According to the X-ray diffraction data (CIF files CCDC 997574 (**I**) and 997575 (**II**)), the crystals of compounds **I** and **II** are isostructural, triclinic, space group $P\bar{1}$, $a = 7.7732(2)$, $b = 8.9209(3)$, $c = 9.8176(3)$ Å, $\alpha = 69.0120(10)°$, $\beta = 72.2170(10)°$, $\gamma = 67.4330(10)°$, $Z = 1$ (**I**); $a = 7.9241(4)$, $b = 9.1701(5)$, $c = 10.2644(5)$ Å, $\alpha = 67.5250(10)°$, $\beta = 71.0670(10)°$, $\gamma = 66.5990(10)°$, $Z = 1$ (**II**). The compounds consist of cations $[H(DMSO)_2]^+$ with a very strong hydrogen bond O···H···O (O···O 2.441 Å in **I** and 2.457 Å in **II**; the hydrogen atom is predominantly localized at one oxygen atom) and anions $[OsX_6]^{2-}$, being somewhat distorted octahedra. The synthesized complexes are characterized by IR spectroscopy, UV/Vis spectroscopy, NMR spectroscopy, and potentiometry. In DMSO solutions, cations $[H(DMSO)_2]^+$ dissociate and DMSO enters the internal sphere of the anions to form the DMSO complexes of osmium(III) and osmium(II).

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INTRODUCTION

Dimethyl sulfoxide $(CH_3)_2SO$ is a widely used aprotic solvent and an ambidentate ligand that can be coordinated by oxygen (DMSO-O) or sulfur (DMSO-S) atoms, depending on the "hardness" or "softness" of the reagent according to Pearson's theory [1]. DMSO is a fairly strong base and is easily protonated in an acidic medium. Since the hydrogen ion is a "hard" acid, the DMSO molecule is protonated at the oxygen atom and dimeric cation $[H(DMSO)_2]^+$ is usually formed in solutions [2–4].

The compounds were synthesized in which $[H(DMSO)_2]^+$ is a counterion of the complex anion containing metal-coordinated DMSO, for example, $[H(DMSO)_2][M(DMSO\text{-}S)_2X_4]$, where X = Cl, M = Ru, Os, Ir, and Rh [5, 6]; X = Br, M = Ru and Os [7, 8]. The interaction of DMSO with acids H_2MCl_6, where $[H(DMSO)_2][M(DMSO\text{-}S)_2X_4]$, where X = Cl, M = Pt, Ir, and Os, results in the isolation of compounds $[H(DMSO)_2]_2[MCl_6]$ in the solid phase [9–12]. Due to the high solubility in many solvents, $[H(DMSO)_2]_2[OsCl_6]$ has already found use as the starting substance for the synthesis of osmium complexes with both DMSO and other organic ligands [12–14]. Bromo complexes of platinum metals with protonated DMSO as a cation are almost unknown.

The methods for syntheses and the characteristics of new osmium complexes, namely, bromo complex $[H(DMSO)_2]_2[OsBr_6]$ and deuterated derivatives $[H(DMSO\text{-}d_6)_2]_2[OsX_6]$ (X = Cl, Br), and the X-ray diffraction data for $[H(DMSO\text{-}O)_2]_2[OsCl_6]$ and $[H(DMSO\text{-}O)_2]_2[OsBr_6]$ are presented in this work.

EXPERIMENTAL

Acids $H_2OsX_6 \cdot nH_2O$ (X = Cl, Br) were obtained from OsO_4 by the interaction with concentrated HX in the presence of ethanol [15]. DMSO (reagent grade) was used without additional purification.

Synthesis of $[H(DMSO)_2]_2[OsCl_6]$ (I) was carried out by the reaction of $H_2OsCl_6 \cdot nH_2O$ with DMSO in ethanol according to a described procedure [12]. The substance was identified by the data of elemental analysis, UV/Vis spectroscopy, and IR and NMR spectroscopy. The obtained characteristics coincide with the published data [9, 12, 16].

Synthesis of $[H(DMSO\text{-}d_6)_2]_2[OsCl_6]$ (Ia). A weighed sample of $H_2OsCl_6 \cdot nH_2O$ (0.15 g) was dissolved in ethanol (10 mL), DMSO-d$_6$ (0.5 mL) was added, the mixture was evaporated (60°C) to ~2 mL, and acetone (20 mL) was added. The formed yellow precipitate was filtered off and washed with acetone. The yield was 0.14 g (~80% based on osmium).

For $[H(C_2D_6SO)_2]_2[OsCl_6]$	
anal. calcd., %:	Os, 25.65.
Found, %:	Os, 25.6.

UV/Vis spectra, λ_{max}, nm (ε), mol^{-1} L cm^{-1}): in DMSO 420 sh (1810), 381 (7980), 346 (11 000), 309 sh (1820), 280 (1590); in HCl (0.5 mol/L) 413 sh (1670), 370 (7730), 334 (8310), 302 sh (1410), 276 sh (1040), 254 (2250).

Synthesis of $[H(DMSO)_2]_2[OsBr_6]$ (II). A weighed sample (0.25 g) of $H_2OsBr_6 \cdot nH_2O$ was dissolved in acetone (25 mL), and DMSO (1 mL) was added. Dark red plates began to precipitate almost immediately. In 1 h, the precipitate was filtered off and washed with acetone. The yield was 0.17 g (~50% based on osmium).

For $[H(C_2H_6SO)_2]_2[OsBr_6]$

anal. calcd., %:	Os, 19.33.
Found, %:	Os, 19.3.

UV/Vis spectra, λ_{max}, nm (ε), mol^{-1} L cm^{-1}): in DMSO 580 (1140), 535 sh (4010), 515 sh (6090), 501 (7740), 458 (12 100), 424 (7460), 352 sh (1540), 285 (11 200); in H_2O 569 (980), 489 (6150), 445 (8120), 419 (4940), 399 (5260), 346 (970), 279 (9570), 245 (19400); in HBr (0.5 mol/L) 568 sh (1120), 489 (6580), 444 (8830), 419 (5550), 398 (5820), 346 (1290), 279 (10 500), 244 (21000).

NMR spectra, δ, ppm: ^1H 2.54; ^{13}C 40.42.

Synthesis of $[H(DMSO\text{-}d_6)_2]_2[OsBr_6]$ (IIa). A weighed sample (0.10 g) of $H_2OsBr_6 \cdot nH_2O$ was dissolved in acetone (10 mL), DMSO (0.5 mL) was added, and a precipitate began to form almost immediately. In 1 h, the precipitate was filtered off and washed with acetone. The yield was 0.074 g (~50% based on osmium).

For $[H(C_2H_6SO)_2]_2[OsBr_6]$

anal. calcd., %:	Os, 18.87.
Found, %:	Os, 18.8.

UV/Vis spectra, λ_{max}, nm (ε), mol^{-1} L cm^{-1}): in DMSO 580 (1050), 535 sh (3930), 515 sh (5950), 501 (7610), 458 (12 200), 424 (7480), 352 sh (1410), 285 (11000); in HBr (0.5 mol/L) 568 sh (1010), 489 (6540), 444 (8700), 419 (5310), 398 (5690), 346 (990), 279 (10300), 244 (20800).

An analysis to osmium was carried out by spectrophotometry using the reaction with thiocarbamide in an acidic medium.

UV/Vis spectra of solutions were recorded on a Varian CARY 50 spectrophotometer. IR spectra were measured on a Nicolet 6700 FT-IR spectrometer (Shared Research and Educational Center of Physico-Chemical Studies of New Materials, Substances and Catalytic Systems, Peoples' Friendship University of Russia). Samples were prepared as KBr pellets (absorption in a range of 4000–400 cm^{-1}) and as powders (attenuated total internal reflectance, 600–150 cm^{-1}). ^1H and ^{13}C NMR spectra were recorded on a Bruker Avance II 300 in DMSO-d_6 solutions, the resonance frequency being 300.2 MHz on protons and 75.5 MHz on ^{13}C nuclei. The spectra were calibrated by the residual signal from the solvent: 2.50 (^1H) and 39.5 ppm (^{13}C).

The X-ray diffraction analyses for crystals **I** and **II** were carried out on a Bruker APEX II CCD diffractometer (MoK_α radiation, $\lambda = 0.71072$ Å, ω scan mode). Single crystals with sizes of 0.25 × 0.20 × 0.15 mm (**I**) and 0.16 × 0.14 × 0.08 mm (**II**) were taken from the overall mixture. The structures were solved by a direct method and refined by full-matrix least squares for F^2 in the anisotropic–isotropic approximation. The positions of the H(C) hydrogen atoms were calculated geometrically. The hydrogen atoms at the oxygen atoms were localized from the difference Fourier syntheses of electron density. All hydrogen atoms were refined in the isotropic approximation in the riding model. Since the H(O) atom is disordered in structure **II**, the refinement was performed for two models: the freely refined occupancy of positions of this atom and its fixed occupancy; in both cases, the coordinates of the atom were not fixed. All calculations were performed using the SHELXTL PLUS 5.0.11 program package [17]. The crystallographic data and the main refinement parameters are presented in Table 1.

The full X-ray diffraction data are available from the Cambridge Crystallographic Data Centre (CCDC 997574 (**I**) and 997575 (**II**); deposit@ccdc.cam.ac.uk or http://www.ccdc.cam.ac.uk).

RESULTS AND DISCUSSION

It is known that halogenosmates(IV) are kinetically inert and the first product of the reaction of H_2OsCl_6 with DMSO is $[H(DMSO)_2]_2[OsCl_6]$ (**I**), which is readily isolated in the solid phase from solutions in DMSO, ethanol, acetone [12], or isopropanol [9]. It was difficult to isolate complex $[H(DMSO)_2]_2[OsBr_6]$ (**II**) to the solid phase from solutions containing $[H(DMSO)_2]^+$ and $[OsBr_6]^{2-}$ ions. The reaction of H_2OsBr_6 with DMSO without a diluent gave no precipitate, most likely, because of the high solubility of the compound. The improvement of the synthesis procedure made it possible to reveal the conditions for the formation of a precipitate with the composition $[H(DMSO)_2]_2[OsBr_6]$: (a) acetone as a solvent, (b) ratio DMSO : acetone = 1 : 20, and (c) osmium concentration in the range from 1×10^{-3} to 0.02 mol/L. At $c_{Os} > 0.02$ mol/L, dimethyl sulfide complexes are formed along with compound **II**, and no precipitate is formed at $c_{Os} < 1 \times 10^{-3}$ mol/L.

The UV/Vis spectra of the synthesized compounds (see Experimental) correspond to the absorption of ions $[OsX_6]^{2-}$ (X = Cl, Br) [18, 19]. The spectra of DMSO solutions of compounds **I** (Fig. 1) and **II**, as well as solutions of $K_2[OsX_6]$ [19], change very slowly and indicate the stepwise reduction of osmium to form

Table 1. Crystallographic data and the main refinement parameters for structures $[H(DMSO)_2]_2[OsX_6]$ (X = Cl (**I**), Br (**II**))

Parameter	Value	
	I	**II**
Empirical formula	$C_8H_{26}O_4S_4Cl_6Os$	$C_8H_{26}O_4S_4Br_6Os$
Molecular weight	717.43	984.19
T, K	120	120
Crystal system	Triclinic	Triclinic
Space group	$P\bar{1}$	$P\bar{1}$
Z	1	1
a, Å	7.7732(2)	7.9241(4)
b, Å	8.9209(3)	9.1701(5)
c, Å	9.8176(3)	10.2644(5)
α, deg	69.0120(10)	67.5250(10)
β, deg	72.2170(10)	71.0670(10)
γ, deg	67.4330(10)	66.5990(10)
V, Å^3	575.71(3)	619.28(6)
ρ_{calcd}, g/cm^3	2.069	2.639
μ, cm^{-1}	66.06	151.74
$F(000)$	348	456
$2\theta_{max}$, deg	60	58
Number of measured reflections	10206	6324
Number of independent reflections	3347	3253
Number of reflections with $I > 2\sigma(I)$	3347	2872
Number of refined parameters	114	111
R_1	0.0140	0.0211
wR_2	0.0339	0.0488
GOOF	1.005	0.969
$\Delta\rho_{max}/\Delta\rho_{min}$, e Å^{-3}	1.043/–0.956	1.302/–0.752

trans-$[Os^{III}(DMSO\text{-}S)_2X_4]^-$ and, finally, the dimethyl sulfoxide complexes of osmium(II).

The IR absorption spectra of compounds **I** and **II** exhibit broad bands characteristic of hydrogen bonds S=O···H···O=S in a region of 700–900 cm^{-1} with maxima at ~840 cm^{-1}, diffuse bands at 1100–1700 cm^{-1}, intense ν(SO) bands of DMSO bound to the proton through oxygen at 945 cm^{-1}, and bands of the methyl groups of the DMSO molecules (Table 2). The IR spectra of compounds **I** and **II** in a range of 400–4000 cm^{-1} are nearly identical, indicating the same structures of the compounds.

The IR absorption spectra of deuterated derivatives **Ia** and **IIa**, as compared to the spectra of compounds **I** and **II**, exhibit the shift of the absorption bands of the methyl groups with the isotopic substitution coefficients (k) 1.22–1.38, and the ν(SO) and ν(S=O···H···O=S) bands slightly change their positions (Table 2).

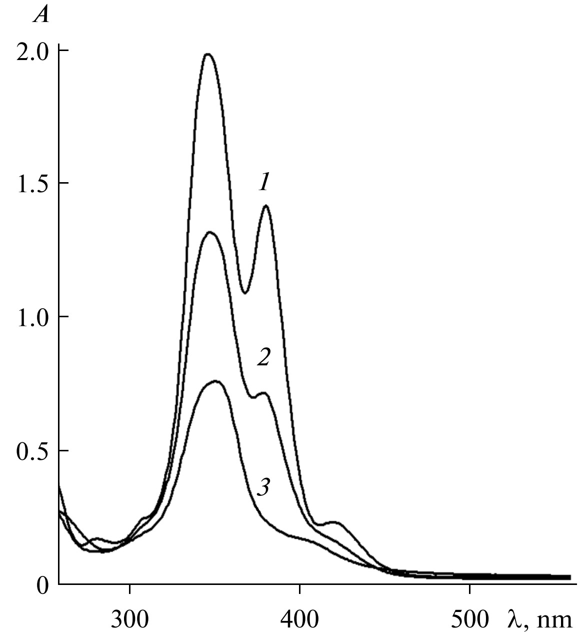

Fig. 1. UV/Vis spectra of (*1*) a freshly prepared solution of compound **I** in DMSO and this solution stored for (*2*) 3 and (*3*) 5 years. Curve *3* corresponds to the absorption of *trans*-$[Os(DMSO\text{-}S)_2Cl_4]^-$.

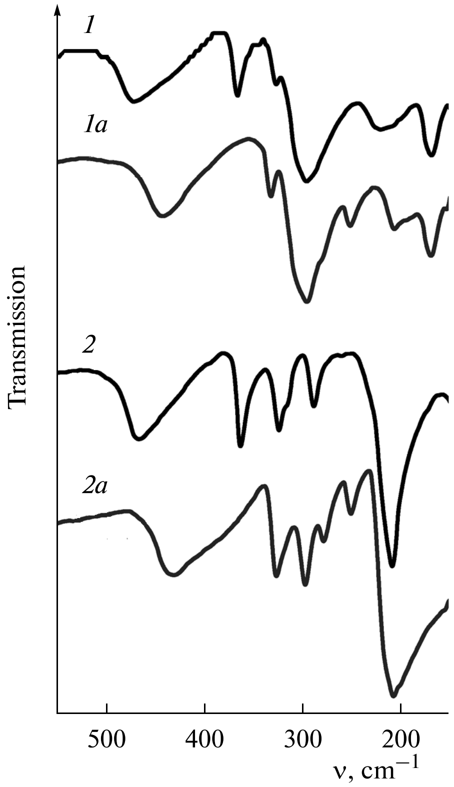

Fig. 2. Far IR spectra (ATIR) of $[H(DMSO)_2]_2[OsX_6]$ (X = (*1*) Cl and (*2*) Br) and $[H(DMSO\text{-}d_6)_2]_2[OsX_6]$ (X = (*1a*) Cl and (*2a*) Br).

The positions of the bands corresponding to vibrations of the Os–Cl bonds (ν_3 = 294 and ν_4 = 167 cm^{-1}) in compounds **I** and **Ia** and Os–Br bonds (ν_3 = 207–206 cm^{-1}) in the Far IR spectra of compounds **II** and **IIa** remain unchanged upon deuteration. The maxima of these bands are shifted to the low-frequency range compared to the spectra of $K_2[OsX_6]$ (315, 172 cm^{-1} for X = Cl; 220 cm^{-1} for X = Br) and nearly coincide with similar bands in the IR spectra of $(Bu_4N)_2[OsX_6]$ (296, 166 cm^{-1} for X = Cl; 209 cm^{-1} for X = Br). Other bands in the spectra of compounds **Ia** and **IIa** undergo a low-frequency shift (k = 1.07–1.15), which indicates a significant contribution from vibrations of the CH bonds to these bands (Fig. 2, Table 2). The assignment in the Far IR range was made on the basis of a comparative analysis of the spectra using the calculation of the vibrational spectrum of DMSO [20].

Different techniques of measuring the IR spectra in the Far region (attenuated total internal reflectance (ATIR), powder) and in the medium region (absorption, KBr pellets) should be mentioned. The ATIR spectra of $K_2[OsX_6]$ are fairly well consistent with the published IR absorption spectra of $K_2[OsCl_6]$ (314, 177 cm^{-1}) and $K_2[OsBr_6]$ (227 cm^{-1}) [21]. The δ(CSO) bands of compound **I** are observed in the ATIR and absorption spectra at 469 and 473 cm^{-1}, while those for compound **II** are observed at 440 and 444 cm^{-1} (Table 2).

The NMR spectra of a solution of compound **II** in DMSO-d_6, as well as the spectra of a solution of compound **I** [16], contain signals from free DMSO at 2.54 ppm (^1H) and 40.42 ppm (^{13}C). Therefore, ions $[H(DMSO)_2]^+$ dissociate in solutions.

According to the data of potentiometric measurements, in freshly prepared solutions (1.0×10^{-3} M) of compounds **I** and **II**, the proton concentration is 1.7×10^{-3} and 2.0×10^{-3} mol/L, respectively. This indicates an almost complete dissociation of ions $[H(DMSO)_2]^+$ in aqueous solutions.

The X-ray diffraction analysis confirmed the structures of compounds **I** and **II**. The crystals of compounds **I** and **II** are isostructural to each other and to $[H(DMSO)_2]_2[PtCl_6]$ [10].

The external spheres of both compounds include dimethylsulfonium cations (Figs. 3, 4) with the very strong hydrogen bond O(1)···H···O(2) (O(1)···O(2) 2.441 and 2.457 Å, angles OHO 175° and 163° in compounds **I** and **II**, respectively).

In compound **II**, the hydrogen atom involved in the strong hydrogen bond is disordered over two positions corresponding to the O(1)–H···O(2) and O(1)···H–O(2) forms with site occupancies of 70 and 30%. The distribution of the formally bonding O–H distances in both structures indicates the predominant localization of the hydrogen atom rigidly at one oxygen atom, which corresponds to the nonsymmetrical two-well

Table 2. IR spectra of the osmium complexes in the range 4000–400 cm^{-1} (absorption, KBr pellets) and in the range 600–150 cm^{-1} (ATIR, powder)

I	Ia	*k**	II	IIa	*k**	Assignment
Absorption (range 4000–400 cm^{-1})						
3012 m	2263 w	1.33	3006 m	2259 w	1.33	CH_3 (CD_3)
2999 m	2252 w	1.33	2993 m	2247 w	1.33	″
2915 m	2128 w	1.37	2910 w	2123 w	1.37	″
1406 m	1017 m	1.38	1403 m	1016 m	1.38	″
1030 m	830 s	1.24	1028 s	828 s	1.24	″
1001 s	750 s	1.33	998 s	750 s	1.33	″
965 w	791 w	1.22	962 w	788 w	1.22	″
	769 w			766 w		
945 m	958 m		943 m	957 m		SO
841 s.br	852 s		840 s.br	851 s		S=O···H···O=S
733 w	667 w	1.10	731 w	655 w	1.11	SC
719 w	634 w	1.13	715 w	634 w	1.13	SC
473 m	444 m	1.07	470 m	438 m	1.07	CSO
ATIR (range 600–150 cm^{-1})						
469 br	440 br	1.07	464 br	429 br	1.08	CSO + CH_3 (CD_3)
364 m	330 m	1.10	361 m	325 m	1.11	″
325 w	**		322 m	295 m	1.09	″
**	278 w		314 w	276 w	1.14	CSC + CSO + CH_3 (CD_3)
294 s	294 s					OsCl
**	249 w		287 w	249 w	1.15	CSO + CH_3 (CD_3)
219 w	205 w	1.07	**	198 w		CSO + CH_3 (CD_3)
			207 s	206 s		OsBr
167 m	167 m					OsCl

* *k* is the overlapping coefficient.

** Overlapping with the OsX band.

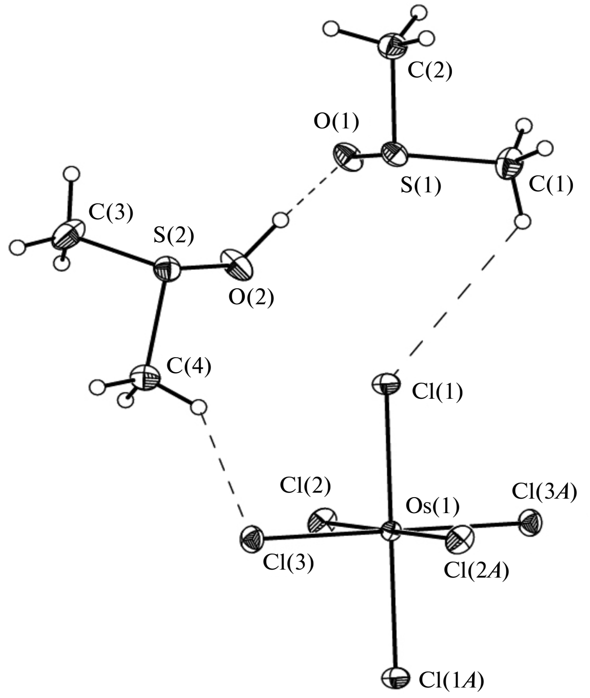

Fig. 3. Structures of cation $[H(DMSO)_2]^+$ and anion $[OsCl_6]^{2-}$ in complex **I** (non-hydrogen atoms are presented by probability ellipsoids of atomic vibrations (50%). The short strong O–H···O hydrogen bond and weak C–H···Cl contacts are shown).

potential of the strong hydrogen bond. For comparison, the position of the hydrogen atom is more centered in the dimethylsulfonium cation bound to $[PtCl_6]^{2-}$ (Table 3).

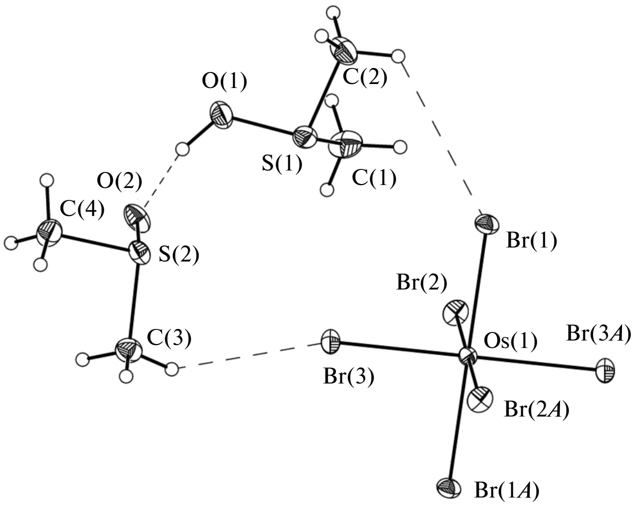

Fig. 4. Structures of cation $[H(DMSO)_2]^+$ and anion $[OsBr_6]^{2-}$ in complex **II** (non-hydrogen atoms are presented by probability ellipsoids of atomic vibrations (50%). The short strong O–H···O hydrogen bond and weak C–H C–H···Br contacts are shown).

The S–O bonds in the cations are elongated compared to those of uncoordinated DMSO (1.495 Å [22]), whereas the S–O bond is substantially longer in DMSO more strongly bound to the proton (Table 3). These results correlate with the data for a similar platinum compound (Table 3). Note that the nonequivalence of the S–O bonds is manifested in the IR spectra of compounds **I** and **II** by a complicated nonsymmetrical ν(SO) band.

Table 3. X-ray diffraction data for $[H(DMSO)_2]_2[MX_6]$

M	X	S–O, Å	O–H, Å	O–O, Å	Angle OHO, deg	M–Cl, Å	References
Os	Cl	1.5704(15)	1.09(5)	2.441(2)	175	2.3397(4)	This work
		1.5405(14)	1.36(5)			2.3437(4)	
						2.3519(4)	
Os	Br	1.579(3)	0.98 1.561*	2.457(5)	163 146*	2.4849(3)	This work
		1.539(3)	1.506 1.002*			2.4914(3)	
						2.5043(3)	
Pt	Cl	1.587(14)	1.219	2.420	166.5	2.316(4)	[10]
		1.53(2)	1.267			2.316(4)	
						2.324(4)	

* Occupancy 30%.

The $[OsX_6]^{2-}$ anions (X = Cl, Br) in compounds **I** and **II** have somewhat distorted octahedral structures with insignificantly elongated Os–X distances for two ligands in the position X(1)–Os–X(1*A*) (by ~0.01 Å), which is obviously related to the static Jahn–Teller effect. Note than in $K_2[OsX_6]$ all Os–X distances are the same and equal to 2.36 Å (X = Cl) and 2.51 Å (X = Br). However, the determination accuracy of structural parameters is low [23].

The possibility of using $[H(DMSO)_2]_2[OsX_6]$ for the syntheses of the dimethyl sulfoxide complexes and dimethyl sulfide derivatives formed due to the deoxygenation of DMSO upon thermolysis is presently studied.

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