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The Crystal Structures of Bis(8-hydroxyquinolinium) Oxoperoxotetrafluorotungstate(VI) Trihydrate, $(C_9H_8NO)_2[WO(O_2)F_4].3H_2O$, and Bis(8-hydroxyquinolinium) Peroxopentafluoroniobate(V) Trihydrate, $(C_9H_8NO)_2[Nb(O_2)F_5].3H_2O$

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Crystals of the title compounds are isomorphous and crystallize in space group $P2_1/c$ with $a = 6{\cdot}792$, $b = 24{\cdot}610$, $c = 13{\cdot}749$ Å, $\beta = 103{\cdot}0°$, $Z = 4$ for the W compound and $a = 6{\cdot}771$, $b = 24{\cdot}785$, $c = 13{\cdot}768$ Å, $\beta = 103{\cdot}3°$, $Z = 4$ for the Nb compound. The crystals are sensitive to X-rays. The structures were determined from film data by direct methods and Fourier syntheses. The W structure was refined to $R = 0{\cdot}071$ and the Nb structure to 0·091. Both metal atoms exhibit octahedral coordination with one corner of the polyhedron at the centre of the peroxo bond. The mean W–F length is 1·96 and W–O_{peroxo} 1·98 Å. The value of 1·75 (2) Å for W–O_{oxo} indicates double-bond character. The mean Nb–F length is 1·93 and Nb–O_{peroxo} 1·93 Å. The two structures consist of discrete octahedra, 8-hydroxyquinolinium cations and water molecules connected by O–H···F, O–H···O, N–H···F and N–H···O hydrogen bonds.

Introduction

These complexes were prepared and their IR spectra recorded by Dr N. Vuletić (unpublished). The spectra show the characteristic strong bands in the region 885–760 cm^{-1} originating from the coordinated peroxo group. The spectrum of the Nb complex shows no band in the region 1000–900 cm^{-1} that can be assigned to Nb=O, but for the W complex the W=O band appears at 985, 951 cm^{-1}.

Experimental

The space groups were determined from Weissenberg photographs recorded with Cu $K\alpha$ radiation. The diffraction symmetry and systematic absences uniquely determined $P2_1/c$. The density was measured at 25 °C pycnometrically with decalin.

Equi-inclination Weissenberg photographs were taken with filtered Cu $K\alpha$ radiation and the multiple-film technique. 1312 observed reflexions for the W compound and 1635 for the Nb compound were recorded and used in all calculations. Their intensities were measured with a microdensitometer. Crystals were shaped into spheres and had the radii 0·0036 cm for $0kl$–$3kl$ for the W compound and 0·0068 cm for $0kl$–$4kl$ for the Nb compound. The intensities were corrected for absorption (Bond, 1967). Scale factors were determined for each layer line separately and further improved in the course of refinement.

Table 1. *Crystal data*

Numbers in parentheses here and throughout this paper are the estimated standard deviations in the least significant digit.

	$(C_9H_8NO)_2[WO(O_2)F_4].3H_2O$	$(C_9H_8NO)_2[Nb(O_2)F_5].3H_2O$
a	6·792 (2) Å	6·771 (2) Å
b	24·610 (5)	24·785 (5)
c	13·749 (3)	13·768 (3)
β	103·0 (2)°	103·3 (3)°
V	2239·6 Å^3	2248·6 Å^3
Z	4	4
D_m	1·953 g cm^{-3}	1·686 g cm^{-3}
D_c	1·934	1·666
μ(Cu $K\alpha$)	105·48 cm^{-1}	54·02 cm^{-1}

Crystal data

Both compounds are sensitive to X-rays. The similar cell parameters and chemical formulae indicate isomorphism (Table 1).

Structure determination and refinement

The positions of the W, four F atoms and oxo O atom were determined with *MULTAN* (Declercq, Germain, Main & Woolfson, 1973). An overall temperature factor ($B = 2{\cdot}35$ Å^2) and a scale factor were determined (Wilson, 1942) and used to compute normalized structure amplitudes by the routine *NORMAL*. The solution was based on 300 reflexions with $|E| > 1{\cdot}2$. A difference synthesis revealed the positions of one peroxo group, three water molecules and two 8-hydroxyquinolinium cations. The locations of 12 H atoms (excluding four atoms attached to N and O) in both cations were calculated on stereochemical grounds. With isomorphism assumed between the W and Nb structures, the position of the W atom was used to calculate a Fourier synthesis with the Nb data. The first synthesis located five F atoms and three water molecules. The positions of the peroxo group and two 8-hydroxyquinolinium cations were recognized in a difference synthesis. In the Fourier syntheses of the W and Nb compounds a large difference in the peak heights of the peroxo O atoms (3 : 1) was observed. In the final cycles of refinement the population parameters of the peroxo O atoms were varied; for one the population parameter kept its normal value (1) but for the other this value was reduced to 0·3. Decomposition of these compounds under the influence of X-rays is manifested in escaping peroxides.

Both structures were refined by full-matrix least squares minimizing $\Sigma w(|F_o| - |F_c|)^2$ with unit weights. All atoms were included in the calculation of structure factors. The scale factors, non-hydrogen atomic positions and isotropic temperature factors together with anisotropic thermal parameters for F atoms and oxo O (for the W structure) were refined.

The final $R = \Sigma||F_o| - |F_c||/\Sigma|F_o| = 0{\cdot}071$ and 0·091 for the W and Nb structures respectively. Scattering factors given by Cromer & Mann (1968) were used. An anomalous dispersion correction was included for W and Nb (Cromer & Liberman, 1970). The calculations were made on a Univac 1110 computer at the University Computing Centre in Zagreb with the X-RAY 72 system (Stewart, Kruger, Ammon, Dickinson & Hall, 1972). Positional and thermal parameters for both structures are listed in Tables 2 and 3.*

* A list of structure factors has been deposited with the British Library Lending Division as Supplementary Publication No. SUP 32089 (26 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 13 White Friars, Chester CH1 1NZ, England.

Table 2. *Final atomic* (× 10^4) *and thermal* (Å^2 × 10^3) *parameters for the* W *compound*

$T = \exp[-2\pi^2(U_{11}h^2a^{*2} + U_{22}k^2b^{*2} + U_{33}l^2c^{*2} + 2U_{12}hka^*b^* + 2U_{13}hla^*c^* + 2U_{23}klb^*c^*)]$.
O(X) and O(X′) belong to OH groups attached to 8-hydroxyquinolinium cations.

	x	y	z	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
W	4190 (3)	1145 (0)	2476 (1)	83 (8)	37 (1)	42 (1)	−2 (1)	14 (1)	−1 (1)
F(1)	2841 (44)	1690 (7)	1501 (14)	175 (36)	56 (12)	72 (13)	−35 (15)	−29 (15)	18 (10)
F(2)	2389 (44)	1442 (10)	3267 (16)	109 (0)	154 (0)	98 (0)	17 (0)	72 (0)	−46 (0)
F(3)	5618 (57)	772 (10)	3594 (20)	254 (40)	104 (19)	133 (22)	36 (23)	−8 (24)	81 (17)
F(4)	5918 (37)	1814 (6)	3050 (12)	130 (30)	39 (9)	51 (10)	−17 (12)	−20 (12)	−7 (8)
O	2388 (53)	666 (8)	1926 (18)	157 (42)	53 (14)	92 (18)	−84 (19)	23 (20)	−30 (13)

	x	y	z	U		x	y	z	U
O(1)	6433 (136)	710 (30)	2089 (54)	70 (0)	C(8)	6510 (62)	3949 (12)	3900 (20)	49 (9)
O(2)	5894 (59)	1088 (15)	1543 (23)	99 (17)	C(9)	6373 (57)	3409 (11)	3609 (19)	39 (8)
O(*W*1)	8716 (46)	1779 (9)	1051 (17)	72 (8)	C(1′)	1259 (66)	4856 (12)	3324 (23)	57 (10)
O(*W*2)	9606 (45)	1674 (9)	4276 (16)	70 (7)	C(2′)	1766 (76)	4640 (16)	4260 (29)	82 (13)
O(*W*3)	−51 (64)	254 (14)	4006 (26)	148 (14)	C(3′)	1738 (66)	4107 (13)	4373 (23)	57 (10)
O(X)	6662 (39)	2963 (7)	4181 (13)	43 (5)	C(4′)	1401 (60)	3743 (11)	3543 (20)	41 (9)
O(X′)	26 (39)	3930 (8)	823 (13)	46 (6)	C(5′)	938 (63)	3985 (9)	2587 (20)	34 (7)
C(1)	5229 (64)	2670 (12)	1259 (22)	52 (9)	C(6′)	1432 (66)	3184 (13)	3618 (23)	57 (10)
C(2)	4918 (66)	3118 (12)	498 (21)	50 (9)	C(7′)	1065 (61)	2878 (10)	2768 (18)	37 (8)
C(3)	5089 (66)	3631 (12)	850 (22)	53 (9)	C(8′)	580 (59)	3106 (10)	1788 (19)	36 (7)
C(4)	5566 (56)	3735 (9)	1872 (17)	31 (7)	C(9′)	549 (61)	3653 (10)	1732 (20)	38 (8)
C(5)	5825 (67)	3312 (10)	2570 (22)	41 (7)	N	5668 (47)	2782 (8)	2205 (14)	31 (6)
C(6)	5687 (60)	4295 (10)	2238 (18)	37 (8)	N′	880 (55)	4533 (8)	2492 (18)	40 (6)
C(7)	6240 (66)	4358 (12)	3259 (23)	52 (9)					

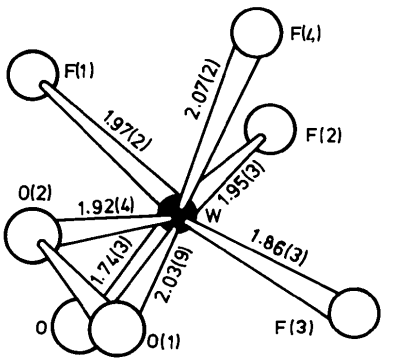

Fig. 1. The W coordination sphere.

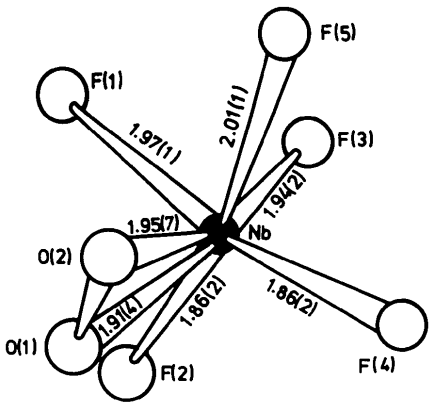

Fig. 2. The Nb coordination sphere.

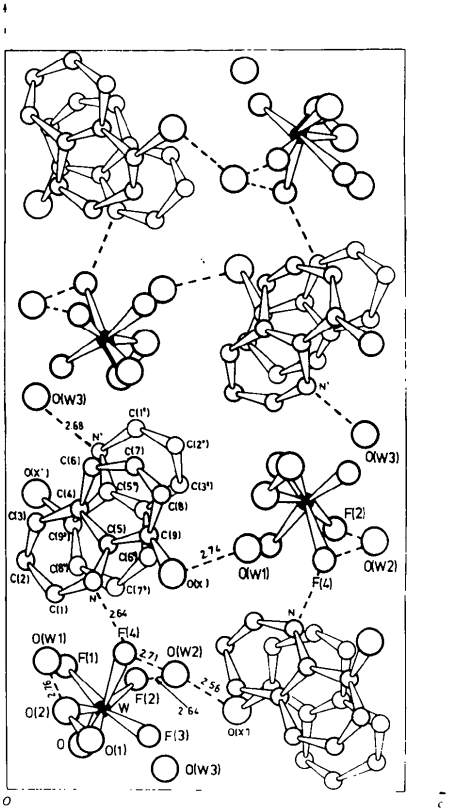

Fig. 3. The crystal structure with the hydrogen bonds viewed along **a**.

Table 3. *Final atomic* ($\times 10^4$) *and thermal* ($Å^2 \times 10^3$) *parameters for the* Nb *compound*

	x	y	z	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
Nb	4165 (3)	1169 (1)	2490 (1)	43 (5)	31 (1)	33 (1)	−2 (1)	9 (1)	−2 (1)
F(1)	2788 (22)	1686 (4)	1465 (8)	84 (14)	54 (6)	52 (7)	−6 (7)	−3 (7)	10 (5)
F(2)	2173 (30)	680 (6)	1876 (13)	110 (20)	102 (11)	159 (15)	−84 (12)	−3 (13)	−9 (11)
F(3)	2366 (26)	1452 (7)	3276 (11)	93 (18)	158 (14)	108 (12)	32 (12)	80 (11)	−22 (10)
F(4)	5604 (33)	787 (7)	3593 (12)	186 (24)	169 (17)	111 (12)	117 (16)	65 (13)	90 (12)
F(5)	5869 (23)	1818 (4)	3012 (9)	86 (14)	52 (6)	80 (8)	−12 (8)	−34 (8)	4 (6)
O(1)	6159 (56)	1055 (16)	1724 (23)	89 (28)	120 (26)	127 (20)	7 (23)	80 (18)	−24 (19)
O(2)	4997 (134)	701 (34)	1512 (33)	51 (71)	90 (53)	77 (32)	35 (44)	11 (29)	−48 (27)

	x	y	z	U		x	y	z	U
O($W1$)	8713 (28)	1761 (6)	1024 (11)	106 (5)	C(8)	6507 (36)	3952 (7)	3876 (13)	44 (5)
O($W2$)	9626 (29)	1710 (6)	4301 (12)	80 (5)	C(9)	6428 (35)	3420 (7)	3570 (13)	40 (5)
O($W3$)	−329 (41)	244 (9)	3981 (17)	121 (8)	C(1′)	1344 (41)	4838 (8)	3324 (16)	61 (6)
O(X)	6667 (23)	2986 (5)	4163 (9)	57 (4)	C(2′)	1837 (43)	4618 (9)	4260 (18)	70 (7)
O(X′)	90 (24)	3940 (5)	831 (9)	36 (4)	C(3′)	1815 (43)	4086 (9)	4391 (17)	70 (7)
C(1)	5221 (37)	2676 (7)	1218 (14)	48 (5)	C(4′)	1465 (37)	3727 (7)	3560 (13)	43 (5)
C(2)	4973 (39)	3101 (8)	471 (15)	54 (6)	C(5′)	1039 (36)	3972 (6)	2599 (13)	35 (4)
C(3)	5151 (39)	3604 (8)	795 (14)	51 (6)	C(6′)	1443 (41)	3174 (9)	3593 (16)	59 (6)
C(4)	5614 (36)	3744 (7)	1834 (13)	40 (5)	C(7′)	1036 (38)	2870 (7)	2764 (14)	47 (5)
C(5)	5883 (37)	3318 (6)	2540 (14)	37 (4)	C(8′)	576 (37)	3103 (7)	1791 (14)	45 (5)
C(6)	5797 (37)	4284 (8)	2189 (13)	47 (5)	C(9′)	528 (36)	3656 (6)	1711 (13)	36 (5)
C(7)	6291 (41)	4384 (9)	3216 (16)	60 (6)	N	5695 (27)	2791 (5)	2191 (9)	34 (4)
					N′	1006 (32)	4525 (6)	2497 (12)	47 (4)

Table 4. *Interatomic distances* (Å) *and angles* (°)

Within the W polyhedron

W–F(1)	1·97 (2)	F(1)–W–F(2)	82 (1)
W–F(2)	1·95 (3)	F(1)–W–F(3)	166 (1)
W–F(3)	1·86 (3)	F(1)–W–F(4)	82 (1)
W–F(4)	2·07 (2)	F(1)–W–O	89 (1)
W–O	1·74 (3)	F(1)–W–O(1)	116 (2)
W–O(1)	2·03 (9)	F(1)–W–O(2)	81 (1)
W–O(2)	1·92 (4)	F(2)–W–F(3)	90 (1)
		F(2)–W–F(4)	82 (1)
F(1)···F(2)	2·59 (3)	F(2)–W–O	92 (1)
F(1)···F(4)	2·64 (3)	F(2)–W–O(1)	161 (2)
F(1)···O	2·62 (3)	F(2)–W–O(2)	161 (1)
F(2)···F(3)	2·70 (4)	F(3)–W–F(4)	86 (1)
F(2)···F(4)	2·64 (4)	F(3)–W–O	102 (1)
F(2)···O	2·66 (3)	F(3)–W–O(1)	71 (2)
F(3)···F(4)	2·69 (3)	F(3)–W–O(2)	103 (1)
F(3)···O	2·81 (4)	F(4)–W–O	170 (1)
		F(4)–W–O(1)	97 (2)
Within the peroxo group		F(4)–W–O(2)	86 (1)
O(1)–O(2)	1·20 (8)	O–W–O(1)	91 (2)
		O–W–O(2)	97 (1)

Within the Nb polyhedron

Nb–F(1)	1·97 (1)	F(1)–Nb–F(2)	86 (1)
Nb–F(2)	1·86 (2)	F(1)–Nb–F(3)	85 (1)
Nb–F(3)	1·94 (2)	F(1)–Nb–F(4)	170 (1)
Nb–F(4)	1·86 (2)	F(1)–Nb–F(5)	83 (1)
Nb–F(5)	2·01 (1)	F(1)–Nb–O(1)	89 (1)
Nb–O(1)	1·91 (4)	F(1)–Nb–O(2)	93 (2)
Nb–O(2)	1·95 (7)	F(2)–Nb–F(3)	90 (1)
		F(2)–Nb–F(4)	103 (1)
F(1)···F(2)	2·61 (2)	F(2)–Nb–F(5)	167 (1)
F(1)···F(3)	2·64 (2)	F(2)–Nb–O(1)	100 (1)
F(1)···F(5)	2·64 (2)	F(2)–Nb–O(2)	66 (3)
F(2)···F(3)	2·70 (2)	F(3)–Nb–F(4)	90 (1)
F(2)···F(4)	2·92 (2)	F(3)–Nb–F(5)	84 (1)
F(3)···F(4)	2·70 (3)	F(3)–Nb–O(1)	167 (1)
F(3)···F(5)	2·64 (2)	F(3)–Nb–O(2)	156 (3)
F(4)···F(5)	2·69 (2)	F(4)–Nb–F(5)	88 (1)
		F(4)–Nb–O(1)	94 (1)
Within the peroxo group		F(4)–Nb–O(2)	95 (2)
O(1)–O(2)	1·17 (9)	F(5)–Nb–O(1)	100 (1)
		F(5)–Nb–O(2)	119 (2)

Description and discussion of the structures

Both W and Nb atoms exhibit octahedral coordination with one corner of the polyhedron at the centre of the peroxo bond (Figs. 1 and 2). The structures are composed of discrete octahedra, 8-hydroxyquinolinium cations and crystalline water molecules connected by O–H···F, O–H···O, N–H···F and N–H···O hydrogen bonds (Fig. 3). Interatomic distances and hydrogen bonds are given in Tables 4 and 5.

The W atom is coordinated by four F atoms, an oxo O atom and a peroxo group. The W–F distances range from 1·86 (3) to 2·07 (2) Å. The value of 1·75 (2) Å for W–O_{oxo} indicates double-bond character. The W–O_{peroxo} distances are 2·03 (9) and 1·92 (4) Å. The Nb atom is surrounded by five F atoms and a peroxo group. The Nb–F distances range from 1·86 (2) to 2·01 (1) Å. The Nb–O_{peroxo} distances are 1·91 (4) and 1·95 (7) Å. The O–O lengths in the peroxide group, 1·20 (8) for the W and 1·17 (9) Å for the Nb structures, are extremely short. As a consequence of peroxide decomposition large errors in these bond lengths can be expected.

The mean value of the C–C distances in the 8-hydroxyquinolinium cations is 1·38 Å (W) and 1·39 Å (Nb) (Table 4). The C–C–C and C–C–N angles are in the usual range.

Molecular packing is realized through hydrogen bonds O–H···F, O–H···O, N–H···F and N–H···O (Fig. 3 and Table 5). The polyhedra are joined by O(*W*1)–H···F(1), 2·74 (4), O(*W*2)–H···F(2), 2·64 (4), O(*W*2)–H···F(4), 2·71 (3), and O(*W*1)–H···O(2), 2·76 (5) Å, hydrogen bonds acting between the crystalline water molecules and octahedra. Polyhedra are also connected to 8-hydroxyquinolinium cations by N^+–H···F(4) bonds, 2·64 (2) Å. The crystalline water molecules are involved in hydrogen

Table 4 (*cont.*)

Within the 8-hydroxyquinolinium cations

	W compound	Nb compound		W compound	Nb compound
C(1)–C(2)	1·50 (4)	1·46 (3)	C(2)–C(1)–N	120 (2)	121 (2)
C(1)–N	1·30 (3)	1·33 (2)	C(1)–C(2)–C(3)	117 (3)	117 (2)
C(2)–C(3)	1·35 (4)	1·32 (3)	C(2)–C(3)–C(4)	121 (2)	123 (2)
C(3)–C(4)	1·39 (4)	1·43 (3)	C(3)–C(4)–C(5)	121 (2)	117 (1)
C(4)–C(5)	1·40 (3)	1·42 (2)	C(5)–C(4)–C(6)	118 (2)	119 (1)
C(4)–C(6)	1·46 (3)	1·42 (2)	C(4)–C(5)–C(9)	122 (2)	121 (1)
C(5)–C(9)	1·41 (4)	1·40 (2)	C(4)–C(5)–N	117 (2)	118 (1)
C(5)–N	1·39 (3)	1·39 (2)	C(4)–C(6)–C(7)	116 (2)	120 (2)
C(6)–C(7)	1·38 (4)	1·40 (3)	C(6)–C(7)–C(8)	124 (3)	119 (2)
C(7)–C(8)	1·32 (4)	1·39 (3)	C(7)–C(8)–C(9)	123 (3)	123 (2)
C(8)–C(9)	1·38 (4)	1·38 (3)	C(5)–C(9)–C(8)	116 (2)	118 (1)
C(9)–O(X)	1·34 (3)	1·34 (2)	C(1)–N–C(5)	123 (2)	122 (1)
C(1′)–C(2′)	1·36 (5)	1·37 (3)	C(2′)–C(1′)–N′	121 (3)	121 (2)
C(1′)–N′	1·37 (4)	1·35 (3)	C(1′)–C(2′)–C(3′)	119 (3)	121 (2)
C(2′)–C(3′)	1·32 (5)	1·33 (5)	C(2′)–C(3′)–C(4′)	122 (3)	121 (2)

Table 4 (*cont.*)

C(3′)–C(4′)	1·43 (4)	1·42 (3)	C(3′)–C(4′)–C(5′)	116 (2)	116 (2)
C(4′)–C(5′)	1·41 (4)	1·42 (2)	C(5′)–C(4′)–C(6′)	119 (2)	117 (2)
C(4′)–C(6′)	1·38 (4)	1·37 (3)	C(4′)–C(5′)–C(9′)	120 (2)	121 (1)
C(5′)–C(9′)	1·40 (4)	1·43 (2)	C(4′)–C(5′)–N′	120 (2)	121 (1)
C(5′)–N′	1·35 (3)	1·38 (2)	C(4′)–C(6′)–C(7′)	119 (3)	122 (2)
C(6′)–C(7′)	1·36 (4)	1·34 (3)	C(6′)–C(7′)–C(8′)	123 (2)	122 (2)
C(7′)–C(8′)	1·43 (3)	1·43 (3)	C(7′)–C(8′)–C(9′)	116 (2)	118 (2)
C(8′)–C(9′)	1·35 (4)	1·37 (2)	C(5′)–C(9′)–C(8′)	122 (2)	119 (1)
C(9′)–O(X′)	1·40 (3)	1·37 (2)	C(1′)–N′–C(5′)	120 (2)	119 (2)

Table 5. *Hydrogen bonds*

W structure		Nb structure		Symmetry operations
O(*W*1)–H···F(1)	2·74 (4)	O(*W*1)–H···F(1)	2·69 (2)	x,y,z; $1+x,y,z$
O(*W*1)–H···O(2)	2·76 (5)	O(*W*1)–H···O(1)	2·78 (4)	
O(*W*2)–H···F(2)	2·64 (4)	O(*W*2)–H···F(3)	2·65 (3)	x,y,z; $1+x,y,z$
O(*W*2)–H···F(4)	2·71 (3)	O(*W*2)–H···F(5)	2·76 (2)	
N–H···F(4)	2·64 (2)	N–H···F(5)	2·65 (2)	
O(X)–H···O(*W*1)	2·71 (3)	O(X)–H···O(*W*1)	2·69 (2)	x,y,z; $x,\frac{1}{2}-y,\frac{1}{2}+z$
O(X′)–H···O(*W*2)	2·56 (3)	O(X′)–H···O(*W*2)	2·61 (2)	x,y,z; $-1+x,\frac{1}{2}-y,-\frac{1}{2}+z$
O(*W*3)···N′	2·68 (4)	O(*W*3)···N′	2·66 (3)	x,y,z; $\bar{x},-\frac{1}{2}+y,\frac{1}{2}-z$

bonds between two 8-hydroxyquinolinium cations. In two cases water molecules act as acceptors: O(X)–H···O(*W*1), 2·71 (3), and O(X′)–H···O(*W*2), 2·56 (3) Å. The donor–acceptor mechanism in the O(*W*3)···N′, 2·68 (4) Å, hydrogen bond is not obvious. According to Pimentel & McClellan (1960) for about 40 compounds in which N^+–H···O bonding occurs, the N···O distances are in the range 2·8–2·9 Å. In $UO_2(C_9H_6NO)_2C_9H_6NOH$ (Corsini, Abraham & Thompson, 1967) the N···O contact of 2·71 Å was assigned as a N^+–H···O hydrogen bond. A very short N^+–H···O(carboxylic), 2·696 Å, hydrogen bond was observed in α-benzylpenilloic acid (Kojić-Prodić & Ružić-Toroš, to be published). The same hydrogen-bonding scheme occurs in the Nb structure (Table 5).

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