




Article

Hydroxyplumbopyrochlore, $(\text{Pb}_{1.5}\square_{0.5})\text{Nb}_2\text{O}_6(\text{OH})$, a new member of the pyrochlore group from Jabal Sayid, Saudi Arabia

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ABSTRACT

A new mineral species of the pyrochlore supergroup, hydroxyplumbopyrochlore (IMA2018-145), $(\text{Pb}_{1.5}\square_{0.5})\text{Nb}_2\text{O}_6(\text{OH})$, has been discovered in the Jabal Sayid peralkaline granitic complex of the Arabian Shield, Saudi Arabia. It is associated with quartz, microcline, 'biotite', rutile, zircon, calcite, rhodochrosite, columbite-(Fe), goethite, thorite, bastnäsitate-(Ce), xenotime-(Y), samarskite-(Y), euxenite-(Y), hydroxyrochlore and fluornatropyrochlore. Hydroxyplumbopyrochlore usually shows euhedral octahedra, slightly rhombic dodecahedra and cubes or their combination (0.01–0.06 mm). The mineral is pale yellow to pale brown, transparent with white streak, and has adamantine to transparent lustre. It is brittle with conchoidal fracture. No cleavage or parting are observed. It is isotropic and non-fluorescent. The average microhardness is 463.4 kg mm⁻². The calculated density is 6.474 g cm⁻³.

Hydroxyplumbopyrochlore belongs to the cubic crystal system and exhibits the space group $Fd\bar{3}m$ with unit-cell parameters $a = 10.5456(6)$ Å, $V = 1172.8(2)$ Å³ and $Z = 8$. Electron microprobe analysis gave (6-point average composition, wt.%): CaO 0.32, SrO 0.16, FeO 0.17, Ce₂O₃ 0.07, Pr₂O₃ 0.02, PbO 51.69, Nb₂O₅ 40.06, SiO₂ 0.05, TiO₂ 1.68, Ta₂O₅ 4.74, H₂O_{calc} 0.95, total 99.90, yielding the empirical formula $(\text{Pb}_{1.34}\text{Ca}_{0.03}\text{Fe}_{0.01}\text{Sr}_{0.01}\square_{0.61})_{\Sigma 2}(\text{Nb}_{1.75}\text{Ti}_{0.12}\text{Ta}_{0.12}\text{Si}_{0.01})_{\Sigma 2}\text{O}_6(\text{OH}_{0.53}\text{O}_{0.08}\square_{0.39})_{\Sigma 1}$, where \square = vacancy. The Raman spectrum of hydroxyplumbopyrochlore contains the characteristic bands of O–H vibrations and no bands for H₂O vibrations.

Keywords: hydroxyplumbopyrochlore, pyrochlore supergroup, new mineral, crystal structure, Saudi Arabia

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Introduction

A new mineral species, hydroxyplumbopyrochlore, ideal formula, $(\text{Pb}_{1.5}\square_{0.5})\text{Nb}_2\text{O}_6(\text{OH})$, was discovered in a sample from the Jabal Sayid peralkaline granitic complex in Arabian Shield, Saudi Arabia. The new mineral and its name have been approved by the International Mineralogical Association (IMA) Commission on New Minerals, Nomenclature and Classification (CNMNC) as IMA2018-145 (Li *et al.*, 2020). The holotype specimen is deposited in the Geological Museum of China, Beijing, China (catalogue number: M13239).

There are ten pyrochlore-group minerals approved by IMA–CNMNC (see Table 1). However, no Pb predominant member has been approved. According to the nomenclature of the pyrochlore supergroup (Atencio *et al.*, 2010, p. 688), the correspondence between old and new names on the pyrochlore supergroup of minerals is cited as:

“plumbopyrochlore of Skorobogatova *et al.* (1966), Kartashov *et al.* (1992), Voloshin *et al.* (1993), Kovalenko *et al.* (1995), and Xie *et al.* (2006) is “plumbopyrochlore”. Plumbopyrochlore of Chakhmouradian & Mitchell (2002), Wang *et al.* (2003), and

Beurlen *et al.* (2005) is zero-valent-dominant pyrochlore. Plumbopyrochlore of Voloshin & Pakhomovskiy (1986) corresponds to oxyplumbopyrochlore [their anal. 1, Table 3.1], kenoplumbopyrochlore [their anal. 20, Table 3.1] and “plumbopyrochlore” [several compositions]”.

Instead of an O²⁻ anion and a vacancy, Hydroxyplumbopyrochlore has the dominant OH⁻ anion at the Y site, and therefore differs from ‘oxyplumbopyrochlore’ and ‘kenoplumbopyrochlore’. This paper describes the physical and chemical properties of hydroxyplumbopyrochlore and the characterisation of the structure based on single-crystal X-ray diffraction data.

Occurrence

Hydroxyplumbopyrochlore grains were collected from the pegmatite–aplite mineralised rocks from the Jabal Sayid peralkaline granitic complex in the Arabian Shield, Saudi Arabia (23°49′28.72″N, 40°56′30.93″E). Its associated minerals are quartz, microcline, 'biotite', rutile, zircon, calcite, rhodochrosite, columbite-(Fe), goethite, thorite, bastnäsitate-(Ce), xenotime-(Y), samarskite-(Y), euxenite-(Y), hydroxyrochlore and fluornatropyrochlore (Fig. 1). The hydroxyplumbopyrochlore grains intergrow closely with other rare earth elements (REE) and U–Th–Nb-bearing minerals, which are all hosted within the pegmatite or aplite in this area, signifying their genetic relationship. The U–Th–REE–Nb mineralisation is hosted in the peralkaline granitic pegmatite with hydrothermal superposition in

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Table 1. Current members of the pyrochlore group.

Dominant at the A site	Dominant species of the dominant-valence group at the Y site					
	OH	F	O	H ₂ O	□	Cs
Na	1	2	-	-	-	-
Ca	3	4	5	-	-	-
Sn ²⁺	-	-	-	-	-	-
Sr	-	-	-	-	-	-
Pb ²⁺	*	-	-	-	-	-
Sb ³⁺	-	-	-	-	-	-
Y	-	-	-	-	-	-
U ⁴⁺	-	-	-	-	-	-
H ₂ O	-	-	-	6	-	-
□	7	-	-	8	-	9
Mn ²⁺	10	-	-	-	-	-

*: Hydroxyplumbopyrochlore, (Pb_{1.5}□_{0.5})Nb₂O₆(OH), (this study)

1: Hydroxynatropyrochlore, (Na,Ca,Ce)₂Nb₂O₆(OH), (Ivanyuk *et al.*, 2017)

2: Fluornatropyrochlore, (Na,Pb,Ca,REE,U)₂Nb₂O₆F, (Yin *et al.*, 2015)

3: Hydroxycalcipyrochlore, (Ca,Na,U,□)₂(Nb,Ti)₂O₆(OH), (Yang *et al.*, 2014)

4: Fluorcalcipyrochlore, (Ca,Na)₂(Nb,Ti)₂O₆F, (Li *et al.*, 2016)

5: Oxycalcipyrochlore, Ca₂Nb₂O₆O, (Atencio *et al.*, 2010)

6: Hydropyrochlore, (H₂O,□)₂Nb₂(O,OH)₆(H₂O), (Atencio *et al.*, 2010)

7: Hydroxykenopyrochlore, (□,Ce,Ba)₂(Nb,Ti)₂O₆(OH,F), (Miyawaki *et al.*, 2017)

8: Hydrokenopyrochlore, (□,Sb³⁺,Na)₂Nb₂O₆·H₂O, (Biagioni *et al.*, 2017)

9: Cesiokenopyrochlore, □Nb₂(O,OH)₆Cs_{1-x}, (Agakhanov *et al.*, 2017)

10: Hydroxymanganopyrochlore, (Mn,Th,Na,Ca,REE)₂(Nb,Ti)₂O₆(OH), (Chukanov *et al.*, 2013)

the study areas. The similar geological settings were reported in other areas (Gysi and Williams-Jones, 2013; Dostal *et al.*, 2014; Estrade *et al.*, 2014; Harris and Marriner, 1980). According to the mineral assemblages, the hydroxyplumbopyrochlore is associated originally with the peralkaline granitic pegmatite.

Appearance, physical and optical properties

Hydroxyplumbopyrochlore occurs mostly as well-shaped euhedral octahedra, slightly rhombic dodecahedra and cubes or their combination (Fig. 2). The dispersed grain size is generally in the range of 0.01–0.06 mm. Hydroxyplumbopyrochlore is pale yellow to pale brown, transparent with white streak and has adamantine to transparent lustre. It is brittle with conchoidal fracture. No cleavage or parting are observed. The sample is isotropic and non-fluorescent. The microhardness was measured by a Wolpert-401MVD microhardness tester and ranges from 440.6 to 490.4 kg mm⁻², with the average of 463.4 kg mm⁻², corresponding to a Mohs hardness ≈ 5½. The density is calculated at 6.474 g cm⁻³ on the basis of empirical formula and unit-cell volume refined from single-crystal X-ray diffraction data. Optically, hydroxyplumbopyrochlore is isotropic, $n_{\text{cal}} = 2.26(3)$ (Mandarino, 1979). The mineral is insoluble in cold HCl and HNO₃.

Experimental methods and results

Composition

The composition of hydroxyplumbopyrochlore was determined at Beijing Research Institute of Uranium Geology (BRIUG) with a JEOL JXA-8100 electron microprobe at 20 kV and 10 nA with a 1 μm beam diameter. The standards include bustamite for Ca; celestite for Sr; hematite for Fe; monazite for Ce and Pr; crocoite for Pb; Nb(metal) for Nb; albite for Si; Ta(metal) for Ta; and rutile for Ti.

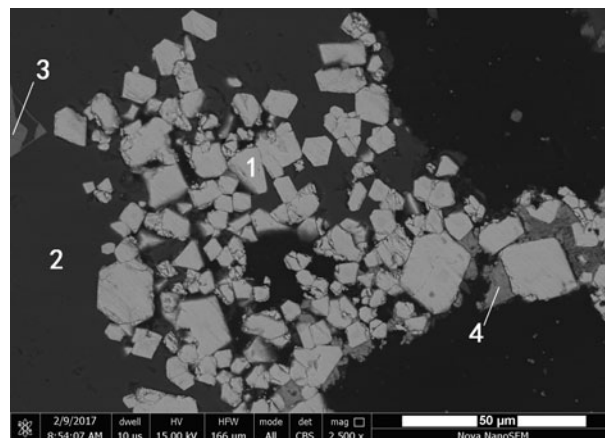


Fig. 1. Back-scattered electron image of hydroxyplumbopyrochlore: 1 – hydroxyplumbopyrochlore; 2 – quartz; 3 – zircon; and 4 – goethite.



Fig. 2. The crystal forms of hydroxyplumbopyrochlore.

The 6-point average composition of hydroxyplumbopyrochlore by Electron probe microanalysis (EPMA) is given in Table 2. Due to the paucity of material, direct determination of H₂O was not possible. H₂O was calculated on the basis of structure refinement, whereas the presence of OH groups and absence of molecular water were confirmed by the Raman spectroscopy.

Raman spectrum

The Raman spectrum of hydroxyplumbopyrochlore was obtained using a Horiba Jobin-Yvon Lab Raman HR Evolution spectrometer with a laser excitation wavelength of 473 nm and measured in the range of 50–4000 cm⁻¹. This experiment was performed at the lab of Horiba Scientific (Shanghai, China).

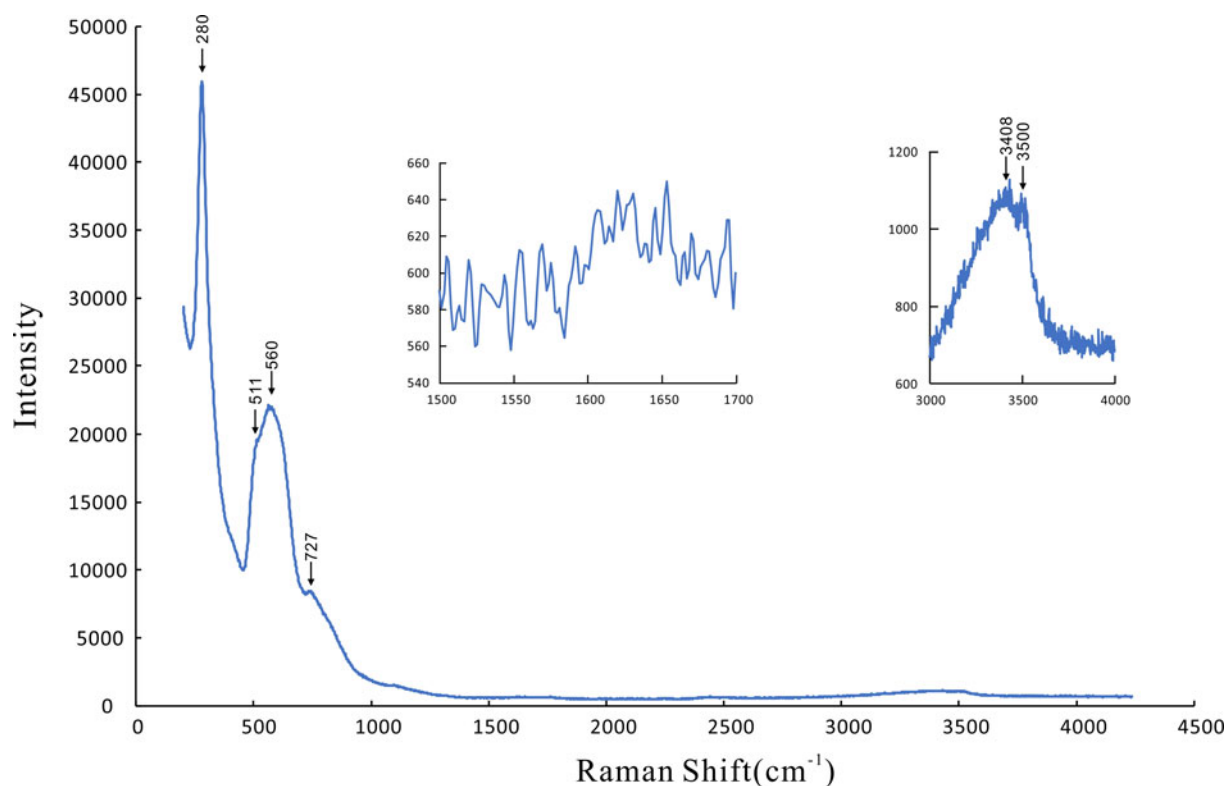
The Raman spectrum of hydroxyplumbopyrochlore is shown in Fig. 3. The assignments of absorption bands have been made by analogy with a pyrochlore-like compound (Ivanyuk *et al.*, 2017; Bahfenne and Frost 2010; RRUFF no. R060980 [<https://rruff.info/hydroxyrochlore/>]). The bands at 3408 cm⁻¹ and 3500 cm⁻¹ can be attributed to O–H stretching vibrations. Bands at 727 cm⁻¹ are caused by (Nb,Ti)–O stretching vibrations and those at 560 cm⁻¹ and 511 cm⁻¹ are interpreted as (Nb,Ti)–O₆ stretching vibrations. A sharp band at 280 cm⁻¹ is due to the (Nb,Ti)–O₆ bending

Table 2. Chemical data (in wt.%) for hydroxyplumbopyrochlore.

Constituent	Mean	Range	S.D. (σ)	Reference material	Element	Apfu	Sum	
CaO	0.32	0.23–0.42	0.07	Bustamite	Ca	0.03		
SrO	0.16	0.08–0.28	0.09	Celestite	Sr	0.01		
FeO	0.17	0.15–0.2	0.02	Hematite	Fe	0.01		
Ce ₂ O ₃	0.07	0–0.17	0.05	Monazite	Ce	–		
Pr ₂ O ₃	0.02	0–0.04	0.01	Monazite	Pr	–		
PbO	51.69	50.22–52.4	0.80	Crocoite	Pb	1.34	ΣA site	1.39
Nb ₂ O ₅	40.06	38.71–41.13	0.87	Nb, metal	Nb	1.75		
SiO ₂	0.05	0–0.11	0.02	Albite	Si	0.01		
TiO ₂	1.68	1.56–1.76	0.10	Rutile	Ti	0.12		
Ta ₂ O ₅	4.74	4.01–5.43	0.57	Ta, metal	Ta	0.12	ΣB site	2.00
H ₂ O ⁺ *	0.95				OH	0.53		
F	b.d.l.				O	0.08		
Total	99.90				\square	0.39	ΣY site	1.00

b.d.l. – below detection limit; S.D. – standard deviation; Apfu – atoms per formula unit.

*Calculated according to the structure refinement.

**Fig. 3.** Raman spectrum of hydroxyplumbopyrochlore.

vibrations. There is no characteristic band of the H₂O vibrations at 1600 cm⁻¹.

X-ray crystallography

Both powder and single-crystal X-ray diffraction of hydroxyplumbopyrochlore were collected at BRIUG using a Bruker D8 QUEST diffractometer equipped with a CMOS monochromated MoK α radiation (0.71073 Å) from a sealed microfocus tube.

The powder-diffraction data are reported in Fig. 4 and Table 3, which were integrated using Bruker APEX3 software and refined using TOPAS software. Unit-cell parameters refined from the powder

diffraction data are as follows: $Fd\bar{3}m$ (#227), $a = 10.5669$ (8) Å, $V = 1179.8$ (9) Å³ and $Z = 8$, which are in good agreement with the single-crystal data.

The single-crystal diffraction data were collected from a single crystal (0.08 mm × 0.10 mm × 0.11 mm). The integration of the data using a cubic unit cell yielded a total of 10,876 reflections to a maximum θ angle of 47.78° (0.48 Å resolution). Data were corrected for absorption effects using the Multi-Scan method (SADABS, Bruker). The final cell constants were $a = 10.5456$ (6) Å and $V = 1172.8$ (2) Å³. The structure was solved and refined using the space group $Fd\bar{3}m$, with $Z = 8$ for the formula unit Pb_{1.34}Ca_{0.06}Nb₂O_{6.61}. The final anisotropic full-matrix least-squares

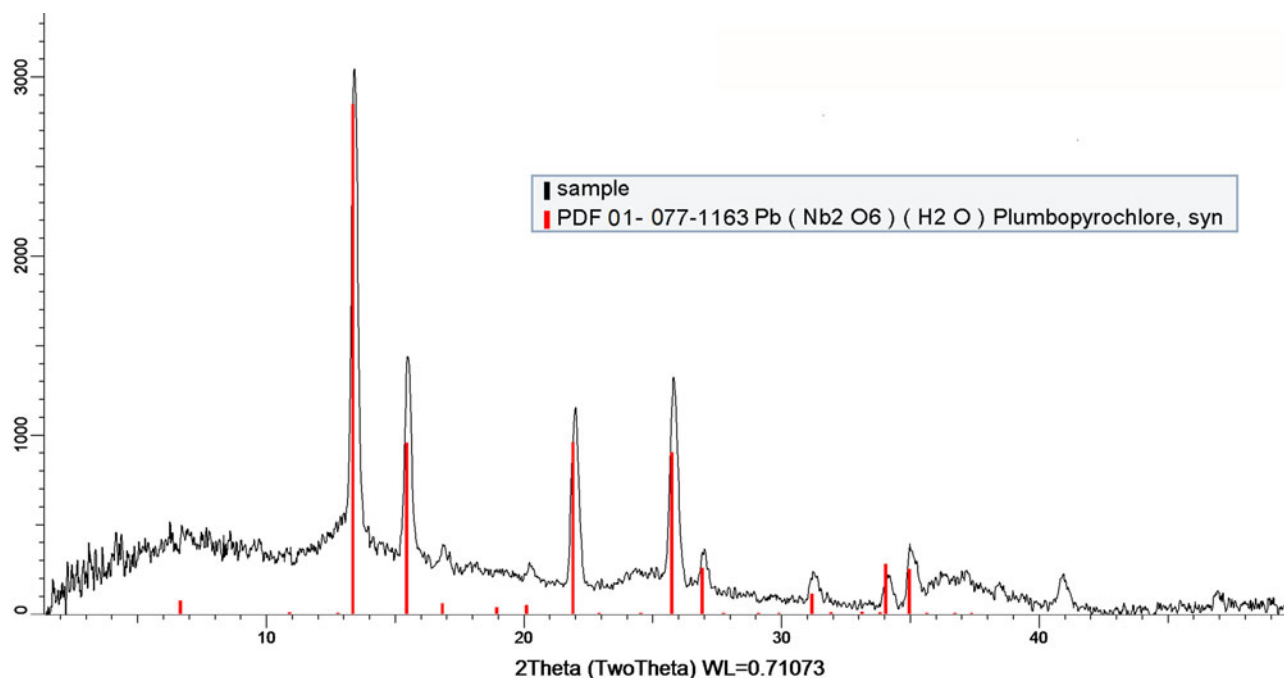


Fig. 4. Powder X-ray diffraction pattern for hydroxyplumbopyrochlore.

Table 3. Powder X-ray diffraction data (d in Å) for hydroxyplumbopyrochlore.

I/I_{100}	I_{calc}/I_{100}	d_{obs}	d_{calc}	$h\ k\ l$
8	1	6.051	6.101	1 1 1
100	52	3.043	3.050	2 2 2
42	29	2.636	2.642	4 0 0
5	2	2.422	2.424	3 3 1
3	1	2.025	2.034	5 1 1
36	49	1.862	1.868	4 4 0
43	100	1.591	1.593	6 2 2
8	19	1.521	1.525	4 4 4
6	25	1.320	1.321	8 0 0
6	30	1.210	1.212	6 6 2
12	69	1.183	1.181	8 4 0

The eight strongest lines are in bold.

refinement on F^2 with 10 variables converged at $R_1 = 3.17\%$ for the observed data and $wR_2 = 13.49\%$ for all data. The goodness-of-fit is 1.136 (Table 4). Final atom coordinates, occupancies, isotropic and anisotropic displacement parameters are given in Table 5. Selected interatomic distances are given in Table 6. The crystallographic information files have been deposited with the Principal Editor of *Mineralogical Magazine* and are available as Supplementary material (see below). The single-crystal structure was solved using direct methods, and the refinement conducted with *SHELXTL* (Sheldrick, 2015).

Discussion

The ideal formula of pyrochlore supergroup is $A_2B_2X_6Y_1$ (Atencio et al., 2010). On the basis of $(\text{Nb} + \text{Ti} + \text{Ta} + \text{Si}) = 2$ atoms per formula unit (B site), the empirical formula of hydroxyplumbopyrochlore is $(\text{Pb}_{1.34}\text{Ca}_{0.03}\text{Fe}_{0.01}\text{Sr}_{0.01}\square_{0.61})_{\Sigma 2}(\text{Nb}_{1.75}\text{Ti}_{0.12}\text{Ta}_{0.12}\text{Si}_{0.01})_{\Sigma 2}\text{O}_6\text{Y}\text{O}^*_{0.345}$ where O^* denotes the amount of charge needed to balance

Table 4. Data collection information and structure-refinement parameters for hydroxyplumbopyrochlore.

Crystal data	
Effective structural formula	$(\text{Pb}_{1.34}\text{Ca}_{0.06})\text{Nb}_2\text{O}_{6.61}$
Crystal dimensions (mm)	$0.082 \times 0.100 \times 0.112$
Temperature (K)	296(2)
Crystal system, Space group	Cubic, $Fd\bar{3}m$
a (Å)	10.5456(6)
V (Å ³)	1172.8(2)
Z	8
Calculated density (g cm ⁻³)	6.474
Absorption coefficient (mm ⁻¹)	42.221
$F(000)$	1967
Date collection	
Theta range for data collection (°)	3.35 to 47.78
Index ranges	$-21 \leq h \leq 21, -21 \leq k \leq 21, -21 \leq l \leq 21$
Coverage of independent reflections	99.3%
Reflections collected	10,876
Independent reflections	303
Absorption correction	Multi-scan
Refinement	
Structure solution technique	Direct methods
Refinement method	Full-matrix least-squares on F^2
R_{int}	0.0432
Data / restraints / parameters	303/0/10
Goodness-of-fit on F^2	1.136
$R_1 [I > 2\sigma(I)], R_1(\text{all})$	0.0317, 0.0422
$wR_2 [I > 2\sigma(I)], wR_2(\text{all})$	0.1234, 0.1349
Largest diff. peak and hole (e ⁻ Å ⁻³)	0.819, -1.139

Weighting scheme: $w = 1/[\sigma^2(F_o^2) + (0.0609P)^2 + 8.8260P]$ where $P = (F_o^2 + 2F_c^2)/3$

the formula recalculated into O atoms. Therefore, the X site has a charge of -12 and the Y site should have a charge of -0.69 .

Because the structure data and composition data are collected from the same particle, we fixed the occupancy of the A and B sites on the basis of the empirical formula. The B site is fully occupied by Nb instead of $(\text{Nb}_{1.75}\text{Ti}_{0.12}\text{Ta}_{0.12}\text{Si}_{0.01})$ for the process of

Table 5. Coordinates and displacement parameters for hydroxyplumbopyrochlore.

Site	Wyk.	s.o.f.	<i>x/a</i>	<i>y/b</i>	<i>z/c</i>	<i>U_{eq}*</i>	<i>U¹¹</i>	<i>U²²</i>	<i>U³³</i>	<i>U²³</i>	<i>U¹³</i>	<i>U¹²</i>
Pb	16 <i>d</i>	Pb _{0.67} Ca _{0.03}	¼	½	¼	0.0291(2)	0.0291(2)	0.0291(2)	0.0291(2)	0.00328(9)	-0.00328(9)	0.00328(9)
Nb	16 <i>c</i>	Nb _{1.00}	½	¾	¼	0.01494(17)	0.01494(17)	0.01494(17)	0.01494(17)	-0.00070(9)	0.00070(9)	0.00070(9)
O1	8 <i>b</i>	O _{0.61}	0.375	0.375	0.375	0.06						
O2	48 <i>f</i>	O _{1.00}	0.4362(3)	0.625	0.125	0.0198(5)	0.0195(14)	0.0199(9)	0.0199(9)	-0.0050(10)	0	0

**U_{eq}* is defined as one third of the trace of the orthogonalised *U_{ij}* tensor. The anisotropic atomic displacement factor exponent takes the form: $-2\pi^2[h^2a^{*2}U_{11} + \dots + 2hka^*b^*U_{12}]$
Wyk. = Wyckoff symbol

Table 6. Selected interatomic distances (Å) for hydroxyplumbopyrochlore.

Pb–O1	2.28319(13) ×2	Nb–O2	1.9819(12) ×6
Pb–O2	2.708(2) ×6		
<Pb–O>	2.602		

Table 7. Site occupancy factors (s.o.f.), *R*₁ and difference-Fourier values for the Y site under different *U_{iso}* values.

<i>U_{iso}</i>	s.o.f.	<i>R</i> ₁	Difference Fourier
0.03	0.48	0.0324	-2.05 e ⁻
0.04	0.53	0.0320	-1.57 e ⁻
0.05	0.57	0.0318	-1.0 e ⁻
0.06	0.61	0.0317	-0.5 e ⁻

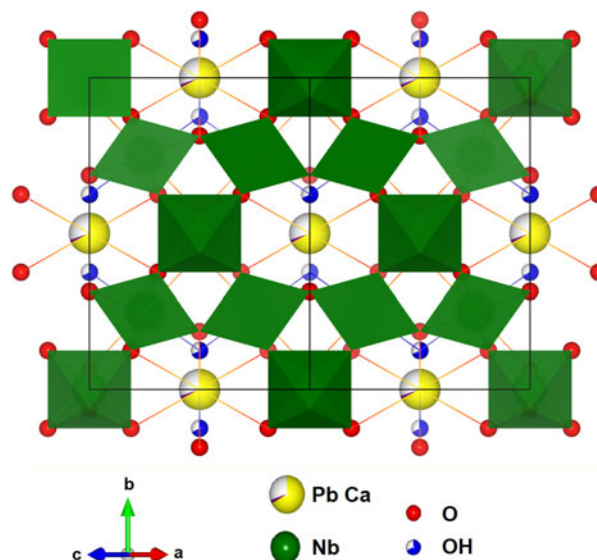
Table 8. Bond-valence analysis for hydroxyplumbopyrochlore.

	Pb	Ca	Nb	Sum
O1	0.261 ^{×21} ×4→	0.078 ^{×21} ×4→		1.08
O2	0.134 ^{×61} ×2→	0.040 ^{×61} ×2→	0.825 ^{×61} ×2→	1.93
Sum	1.33	0.04		
	1.37		4.95	

Multiplicity is indicated by × → and ↓.

refinement. The refinement results of the Y site are a site occupancy factor (s.o.f.) of 0.68 and an associated *U_{eq}* of 0.09 Å², if we allow the site occupancy and displacement parameters for the O1 atom to vary at the same time. However, the *U_{eq}* of O1 is too high for this sample, leading to an inappropriate Y site occupancy. In order to determine the nature of the occupation of the critical Y site, we refined the structure as follows: (1) a refinement with the O1 atom omitted results in an *R*₁ = 0.039 and the largest peak in the difference Fourier is 4.39 e⁻ at the Y site, which suggests an oxygen occupancy of a little over 50%; (2) using a range of reasonable fixed *U_{iso}* values, i.e. 0.03 Å², 0.04 Å², 0.05 Å² and 0.06 Å², refinement of the O1 occupancy then gives the s.o.f., *R*₁ and difference-Fourier values as shown in Table 7; (3) the *U_{iso}* value of 0.06 is the most appropriate for this partially filled site, given the lowest *R*₁ value and the flattest difference map, which suggests this modelling appears to be the best; and (4) after the above steps, (Pb_{1.34}Ca_{0.06}□_{0.6})_{Σ2}Nb₂O₆Y_{O_{0.61}} is therefore indicated, where O[#] denotes the amount of occupation needed to balance the formula charge recalculated into O atoms.

The empirical formula from the EPMA indicates that the Y site requires a charge of -0.69, thus 0.53 OH⁻, 0.08 O²⁻ and 0.39 □ (vacancy) are assigned to the Y site. According to the Raman spectrum H₂O is not present in hydroxyplumbopyrochlore. In

**Fig. 5.** The crystal structure of hydroxyplumbopyrochlore along [101].

summary, based on experiments and analyses, the chemical formula is given as (Pb_{1.34}Ca_{0.03}Fe_{0.01}Sr_{0.01}□_{0.61})_{Σ2}(Nb_{1.75}Ti_{0.12}Ta_{0.12}Si_{0.01})_{Σ2}O₆(OH_{0.53}O_{0.08}□_{0.39})_{Σ1}. The simplified formula is (Pb_{1.5}□_{0.5})Nb₂O₆(OH), which requires PbO 54.92%, Nb₂O₅ 43.60%, H₂O 1.48%, total 100%.

The bond valences were calculated using the parameters of Brown and Altermatt (1985) with the site-occupancy factors given in Table 5. The bond-valence analysis for A and B positions are 1.37 valence units (vu) and 4.95 vu, respectively. The value for O1 is -1.08 vu in site Y (Table 8), which indicates that OH⁻ exists in this site. No direct proof is given for Fe²⁺ or Fe³⁺ because of its 0.17% low chemical contents. We used both Fe²⁺ and Fe³⁺ for structure refinement separately. No matter what element combinations are assigned, the Y site is occupied predominately by OH.

Hydroxyplumbopyrochlore possesses the pyrochlore structure. The A site is dominated by Pb, the B site by Nb and the Y site by OH. The crystal structure of hydroxyplumbopyrochlore is shown in Fig. 5. The structure can be described as a three-dimensional octahedral framework of corner-sharing NbO₆ octahedra with Pb cations and OH groups in the interstices.

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Supplementary material. To view supplementary material for this article, please visit <https://doi.org/10.1180/mgm.2020.69>

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