

# Rumseyite, $[\text{Pb}_2\text{OF}]\text{Cl}$ , the first naturally occurring fluoroxychloride mineral with the parent crystal structure for layered lead oxychlorides

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## ABSTRACT

Rumseyite, ideally  $[\text{Pb}_2\text{OF}]\text{Cl}$ , is a new mineral species which is associated with calcite, cerussite, diaboiteite, hydrocerussite and undifferentiated Mn oxides in a small cavity in 'hydrocerussite' from a manganese pod at Merehead quarry, Somerset, England. Rumseyite is tetragonal,  $I4/mmm$ ,  $a = 4.065(1)$ ,  $c = 12.631(7)$  Å,  $V = 208.7(1)$  Å<sup>3</sup>,  $Z = 2$ . The mineral is translucent pale orange-brown with a white streak and vitreous lustre. It is brittle with perfect  $\{100\}$  cleavage;  $D_{\text{calc}} = 7.71$  g cm<sup>-3</sup> (for the ideal formula,  $[\text{Pb}_2\text{OF}]\text{Cl}$ ). The mean refractive index in air at 589 nm is 2.15. The six strongest reflections in the X-ray powder-diffraction pattern [ $d_{\text{meas}}$  in Å, ( $I_{\text{rel}}$ ), ( $hkl$ )] are as follows: 2.923(100)(013), 2.875(68)(110), 3.848(41)(011), 6.306(17)(002), 1.680(14)(123), 2.110(12)(006). The crystal structure of rumseyite is based on alternating  $[\text{OFPb}_2]$  and Cl layers. Rumseyite is related to other layered Pb oxyhalides. Fluorine and oxygen are statistically disordered over one crystallographic site. Rumseyite is named in honour of Michael Scott (Mike) Rumsey (1980–), Curator and Collections Manager at the NHM (London), who discovered the mineral. The mineral and name have been approved by the IMA Commission on New Mineral Names and Classification (IMA 2011-091). The holotype specimen is in the collections of the Natural History Museum, London (specimen number BM1970,110).

**KEYWORDS:** rumseyite, Merehead quarry, Mendip Hills, layered lead oxychlorides, lead oxyhalides, new mineral species.

## Introduction

FOR some time we have been investigating the mineralogy of the Mendip Hills with particular reference to the assemblages of Pb oxyhalides and other Pb secondary minerals in the manganese pods that occur there; we have also investigated similar oxyhalide minerals from elsewhere in the world (e.g. Turner, 2006; Rumsey, 2008; Siidra and Krivovichev, 2008; Siidra *et al.*, 2008a,b;

Krivovichev *et al.*, 2009; Turner and Rumsey, 2010; Rumsey *et al.*, 2011; Turner *et al.*, 2012). In the course of this research, specimens in the collections of the Natural History Museum (NHM), London were examined, and one was found to contain an unusual looking phase, which subsequently proved to be the new mineral rumseyite. The mineral and name have been approved by the IMA Commission on New Mineral Names and Classification (IMA 2011-091). The holotype specimen is stored in the collections of the NHM under catalogue number BM1970,110. Rumseyite is named in

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honour of the discoverer, Michael Scott (Mike) Rumsey (1980– ), Curator and Collections Manager at the Natural History Museum. Mike is noted for his work on the mineralogy of the Mendip Hills and Kombat mine (Namibia), and in particular for studies of Pb oxychlorides and related Pb secondary minerals, which have already led to the description of several new minerals (Rumsey *et al.*, 2011; Turner *et al.*, 2012).

## Occurrence

Rumseyite is an extremely rare constituent of a lead-dominated mineral assemblage which occurs in manganese oxide pods irregularly distributed in Carboniferous Limestone at the Merehead (also known as Torr Works) quarry, East Cranmore, Somerset, England.

The origin of the manganese oxide pods and the lead oxychloride phases that they enclose has been debated for many years. Turner (2006) suggested that the lead oxychlorides formed when galena was locally exposed to the action of seawater. Surface oxidation of the galena initiated the deposition of manganese minerals, which adsorbed heavy metals from the seawater and the local environment. A later hydrothermal event caused the galena to decompose, a variety of secondary minerals to form and the conversion of the manganate phases to manganese oxides. These oxides formed an impervious layer around a lead-rich core and generated a series of closed systems (the manganese pods). The chemistry of these systems is dependent on local starting conditions, and there is considerable variation in mineralogy from one manganese pod to another.

Associated minerals are calcite, cerussite, diableite, 'hydrocerussite' and undifferentiated Mn oxides, typically a mixture of manganite and pyrolusite (Turner, 2006). It has been shown that the so-called 'hydrocerussite' from this locality is made up of a number of different phases, which at the time of this submission still require further characterization (Turner and Rumsey, 2010); the term hydrocerussite is used *sensu lato* in the following text.

Rumseyite is in direct contact only with hydrocerussite on the type specimen, although there are a small number of minute diableite crystals nearby. The hydrocerussite mass containing the rumseyite is coated with a rind of cerussite, and lies within a cavity in a typical pyrolusite-, manganite- and calcite-rich pod.

## Appearance

Rumseyite is currently known from a single cavity,  $\sim 1.5 \times 1 \times 1$  mm in size, on the holotype specimen, where it is enclosed in a platy mass of hydrocerussite (Figs 1–3); it is possible that other examples will be identified as similar hydrocerussite specimens were abundant at the time that this specimen was collected and added to the NHM collection. They can be identified by their characteristic foliated appearance, off-white colour with a distinct greenish tinge, and pearly lustre. Rumseyite does not completely fill the cavity but is nucleated into one main crystalline mass, with a few other small crystals and crystalline fragments adhering to the cavity edges and nearby fractures in the hydrocerussite crystal. In the unfilled portion of the cavity, platy sheaves of hydrocerussite coat rumseyite and the cavity walls. Sparse diableite occurs but only in direct contact with hydrocerussite.

Rumseyite forms an irregular crystalline mass (Fig. 2) which scanning electron microscope (SEM) examination shows to be composed of aggregates of minute tabular to prismatic crystallites with pyramidal terminations (Fig. 3). No twinning has been observed. The small size of the micro crystals precludes direct measurement but they show forms that appear to be  $\{100\}$  prisms, with  $\{111\}$  pyramid and rarely, small  $\{001\}$  pinacoid terminations.



FIG. 1. The whole of the holotype specimen of rumseyite (BM 1970,110), showing platy white hydrocerussite on a typical manganese oxide matrix. The specimen is  $\sim 40$  mm across. Rumseyite is the small square pinkish brown patch just above and right of centre.



FIG. 2. Part of the holotype specimen (BM 1970,110) of rumseyite showing the approximately cuboidal mass of rumseyite in hydrocerussite. Small pale blue crystals of diaboiteite are also present.

Terminated rumseyite crystals in the main aggregate are generally no larger than about  $50 \times 50 \times 100 \mu\text{m}$ , and most are much smaller, typically  $\sim 15 \times 15 \times 30 \mu\text{m}$ . Broken surfaces within the aggregate suggest that it might be composed of a small number of larger intergrown but heavily fractured and poorly formed crystals without terminations, in which case the largest crystal size would have been in the range 100 to  $500 \mu\text{m}$ . A single, well-formed, possibly double terminated crystal,  $250 \times 80 \times 80 \mu\text{m}$  in size, that was not attached to the main mass, was removed and used for single-crystal and powder-diffraction analysis.



FIG. 3. Scanning electron microscope showing minute tetragonal crystals of rumseyite with conspicuous prism and pyramid faces and small rare pinacoids.

## Physical properties

Rumseyite is translucent pale orange-brown with a white streak and vitreous lustre. It is brittle with perfect  $\{100\}$  cleavage, no observed parting and a conchoidal fracture. Hardness and density could not be measured due to lack of material. The calculated density for the ideal formula  $[\text{Pb}_2\text{OF}]\text{Cl}$  is  $7.72 \text{ g cm}^{-3}$ .

## Optical properties

Rumseyite is non-metallic, but has the high refractive index (RI) typical of lead oxychlorides. Its optical properties were studied using the methods common for metallic minerals to avoid using toxic RI liquids. In reflected light rumseyite is grey with abundant pale yellow internal reflections; it is not pleochroic and has no bireflectance. Any anisotropy is masked by the internal reflections. Reflectance values are listed in Table 1. The mean RI in air at 589 nm calculated from the Fresnel Equation (assuming  $k = 0$ ) is 2.15, which is similar to other Pb oxychloride minerals.

## Chemical composition

Twenty-five chemical analyses were obtained using a Cameca SX100 electron microprobe operating in wavelength-dispersive spectrometry (WDS) mode at 20 kV and 20 nA with a  $1 \mu\text{m}$  beam diameter. Analytical results and standards are listed in Table 2; Mo, As, Cr, V, P and Mg were sought, but they were below detection limits. No other elements were detected by energy-dispersive spectrometry (EDS) on a Jeol 5900LV variable pressure scanning electron microscope. Insufficient material exists for the determination of  $\text{H}_2\text{O}$  or  $\text{CO}_2$  by CHN analysis.

The possible presence of borate or carbonate groups, which are known in some Pb oxychloride minerals from this area (e.g. Welch *et al.*, 1998; Krivovichev *et al.*, 2009) was ruled out by the structure determination. The absence of carbonate was also indicated by a preliminary uncoated EDX analysis.

In the data reduction, O was determined by difference rather than by stoichiometry. The reason for this is the presence of F and Cl, which would have resulted in an excess of O of  $\sim 3 \text{ wt.}\%$  in the PAP matrix correction. This excess O would result in an overcorrection, particularly to the F data, due to the large

TABLE 1. Reflectance values on an area of  $10 \times 10 \mu\text{m}$  of an unoriented section of rumseyite with no internal reflections (using a SiC standard, measured in air).

$\lambda$ (nm)	$R$ (%)
400	15.6
420	15.3
440	15.0
460	14.7
<b>470 (COM)</b>	<b>14.6</b>
480	14.4
500	14.1
520	13.9
540	13.7
<b>546 (COM)</b>	<b>13.6</b>
560	13.5
580	13.4
<b>589 (COM)</b>	<b>13.4</b>
600	13.3
620	13.2
640	13.2
<b>650 (COM)</b>	<b>13.2</b>
660	13.2
680	13.3
700	13.5

The COM wavelengths and reflectances, listed in bold, are those recommended by the Commission on Ore Microscopy.

absorption correction made to F in the presence of O. The O value was subsequently calculated by stoichiometry, and this value is quoted in Table 2. The O wt.% value used in the matrix correction is thus  $((100 - \text{total}) + \text{stoichiometric O wt.\%})$  which produces an overestimation of O in the matrix correction of less than 1 wt.%.

The possibility that hydroxyl groups might partly substitute for O and F was ruled out on the basis of structural and crystal-chemical considerations. Any OH group present would either have to replace the central  $X$  atom in an anion-centred  $X\text{Pb}_4$  ( $X = \text{O}, \text{F}$ ) tetrahedron, which is not chemically possible; or to replace Cl in chlorine sites in the interlayer; although this is possible, these sites were found to be fully occupied by Cl in the structure refinement.

The empirical charge-balanced formula for rumseyite, based on the data in Table 2, is  $\text{Pb}_{2.039}\text{O}_{1.00}\text{F}_{0.992}\text{Cl}_{1.001}$ , and the ideal formula is  $[\text{Pb}_2\text{OF}]\text{Cl}$ , which requires PbO 92.07, Cl 7.31, F 3.92, O = F + Cl - 3.30; total 100.00 wt.%.

### X-ray diffraction data

A fragment of the  $250 \times 80 \times 80 \mu\text{m}$  crystal that was removed from the specimen was used for single-crystal structure analysis. It was mounted on a Stoe IPDS II diffractometer operated at 50 kV and 40 mA at the Department of Crystallography, St Petersburg State University, Russia. More than a hemisphere of three-dimensional data was collected using monochromatic  $\text{MoK}\alpha$  radiation, with frame widths of  $2^\circ$  in  $\omega$ , and a 600 s count for each frame. Unit-cell parameters (Table 3) were refined using least-squares techniques. The analysis revealed tetragonal symmetry,  $I4/mmm$ ,  $a = 4.065(1)$ ,  $c = 12.631(7) \text{ \AA}$ ,  $V = 208.7(1) \text{ \AA}^3$ ,  $Z = 2$ . The intensity data were integrated and corrected for Lorentz, polarization and background effects using the Stoe program *X-Area*. An analytical absorption correction based on the indexed faces was applied. The structure of rumseyite was refined to  $R_1 = 0.072$  for 64 unique reflections with  $F^2 \geq 4\sigma(F^2)$ , using the simplified formula, indicating the lack of defects in the Pb–O–F sheet. Every relevant atom position is filled either

TABLE 2. Analytical data for rumseyite.

Constituent	Mean (wt.%)	Range	SD	Standard
PbO	91.63	90.73–92.44	0.37	Vanadinite
F	3.53	3.05–3.75	0.17	Topaz
Cl	7.15	6.89–7.33	0.10	Vanadinite
O = F + Cl	–3.23			
Total	99.08			

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TABLE 3. Crystallographic data and refinement parameters for rumseyite.

<b>Crystal data</b>	
Temperature (K)	293
Radiation, wavelength (Å)	MoK $\alpha$ , 0.71073
Crystal system	tetragonal
Space group	<i>I4/mmm</i>
Unit-cell dimensions <i>a</i> , <i>c</i> (Å)	4.065(1), 12.631(7)
Unit-cell volume (Å <sup>3</sup> )	208.7(1)
<i>Z</i>	2
Calculated density (g cm <sup>-3</sup> )	7.72
Absorption coefficient (mm <sup>-1</sup> )	81.038
<b>Data collection</b>	
$\theta$ range (°)	6.98–27.83
<i>h</i> , <i>k</i> , <i>l</i> ranges	0 $\rightarrow$ 3, 0 $\rightarrow$ 5, 0 $\rightarrow$ 16
Total reflections collected	93
Unique reflections $F > 4\sigma(F)$	64
<b>Structure refinement</b>	
Refinement method	Full-matrix least-squares on $F^2$
Weighting coefficients <i>a</i> , <i>b</i>	0.1323, 0.0
Extinction coefficient	0.003(5)
Data/restraints/parameters	873/0/47
$R_1 [F > 4\sigma(F)]$ , $wR_2 [F > 4\sigma(F)]$ ,	0.071, 0.202
$R_1$ (all), $wR_2$ (all)	0.099, 0.215
Goodness-of-fit on $F^2$	1.107
Largest diff. peak and hole (e Å <sup>-3</sup> )	2.90, -2.37

with O or F, and there are no vacancies in the sheet. This contrasts with every other natural litharge-derived Pb-oxyhalide.

The poor quality of the crystal resulted in relatively a high  $R_1$  factor and high displacement parameters (Table 4). These are typical of Pb-oxyhalide minerals, which do not commonly form crystals of the perfection required for high resolution single-crystal X-ray studies (e.g. Cooper and Hawthorne, 1994; Welch *et al.*, 1998; Pasero and Vacchiano, 2000). The best possible crystal fragment was chosen for examina-

tion from the tiny amount of material that was available. Final atom coordinates and anisotropic-displacement parameters are given in Table 4. A list of observed and calculated structure factors has been deposited with *Mineralogical Magazine* and can be downloaded from [http://www.minersoc.org/pages/e\\_journals/dep\\_mat\\_mm.html](http://www.minersoc.org/pages/e_journals/dep_mat_mm.html).

X-ray powder diffraction data were obtained using a STOE STADI P diffractometer at the Department of Crystallography, St Petersburg State University, Russia after crushing the crystal fragment used for the single crystal

TABLE 4. Atom coordinates, site occupancies and displacement parameters (Å<sup>2</sup>) for rumseyite.

Site	Wyckoff position	Occupancy	<i>x/a</i>	<i>y/a</i>	<i>z/c</i>	$U_{eq}$	$U_{11}$	$U_{22}$	$U_{33}$
Pb	4 <i>e</i>	1	0	0	0.1469(3)	0.061(2)	0.065(2)	0.065(2)	0.052(2)
Cl	2 <i>b</i>	1	½	½	0	0.056(7)	0.062(11)	0.062(11)	0.043(14)
OF	4 <i>d</i>	O <sub>0.5</sub> F <sub>0.5</sub>	½	0	¼	0.049(11)	0.049(17)	0.049(17)	0.05(2)

The off-axis tensor components,  $U_{12}$ ,  $U_{13}$  and  $U_{23} = 0$ .

analysis (there being insufficient additional material for another sample, and it also being necessary to preserve as much as possible of the holotype specimen for future study). These data are listed in Table 5. Refinement in the tetragonal space group  $I4/mmm$  produced  $a = 4.07(1) \text{ \AA}$ ,  $c = 12.61(1) \text{ \AA}$ ,  $V = 208.4(1) \text{ \AA}^3$ , with  $Z = 2$ , which is in excellent agreement with the single-crystal data.

### Crystal structure

The structure of rumseyite contains one crystallographically unique Pb position. The  $\text{Pb}^{2+}$  cation is coordinated by four O/F atoms and four Cl atoms. The  $\text{Pb}^{2+}$  cations in rumseyite are coordinated by short Pb–O/F bonds (2.41 Å) in one coordination hemisphere (Fig. 4); in the opposite hemisphere there are four long Pb–Cl bonds (3.42 Å). This distortion, which is a general feature of Pb-oxychlorides, is usually interpreted as being due to the influence of the stereoactivity of  $s^2$  lone electron pairs on the  $\text{Pb}^{2+}$  cations. The coordination geometry of the Pb atom in rumseyite can be described as a distorted square antiprism. The Cl atoms have cubic coordination, whereas the O/F atoms have tetrahedral coordination, thus being central for anion-centred (O/F) $\text{Pb}_4$  tetrahedra. The presence of fluorine in the crystal structure of rumseyite is confirmed by the Pb–(O/F) bond length of 2.41 Å. The mean bond length for ideal  $\text{OPb}_4$  tetrahedra is 2.33 Å (Krivovichev and Filatov, 1999). Anion-centred

$\text{FPb}_4$  tetrahedra are present in grandreefite,  $\text{Pb}_2\text{F}_2(\text{SO}_4)$  (Kampf, 1991). The average Pb–F bond length in this structure is 2.55 Å, with individual bond lengths in the range 2.39–2.71 Å.

The crystal structure of rumseyite is based on an alternation (Fig. 5) of continuous  $[\text{OFPb}_2]$  layers of edge-sharing  $X\text{Pb}_4$  tetrahedra ( $X = \text{O}, \text{F}$ ; Fig. 6a) and sheets of chloride ions (Fig. 6c). The  $[\text{OFPb}_2]$  layers are of the type observed in the structure of tetragonal PbO (Dickinson and Friauf, 1924). Note that the unit-cell parameters and the structural motif are very similar to those of a related synthetic lead oxychloride described by Gasperin (1964).

### Discussion

The rumseyite structure is closely related to a number of other Pb-oxihalide minerals with litharge-like sheets. The PbO-like sheets in all of these minerals are made up of edge- and corner-sharing  $\text{OPb}_4$  oxocentred tetrahedra and/or halide layers; however, all of the other minerals have different types of vacancies in the sheets. For instance, the defects in the PbO-like layers in the structure of symesite have a square form (Fig. 6b). In mereheadite, additional Pb atoms are inserted in the chloride layer (Fig. 6d). This leads us to conclude that the crystal structure of rumseyite is a defect-free parent of all of the minerals in the growing class of layered lead oxychlorides. Interestingly, the structure of rumseyite is the same as that of perite (Gillberg, 1960) and nadorite (Giuseppetti and Tadani, 1973), but the oxocentred layers in perite and nadorite are made up of  $\text{OPb}_2\text{A}_2$  ( $A = \text{Bi}, \text{Sb}$ ) heterometallic tetrahedra (Siidra *et al.*, 2008a). This makes rumseyite very different from these

TABLE 5. Powder X-ray data for rumseyite.

$I_{\text{rel}}$ (%)	$d_{\text{meas}}$ (Å)	$d_{\text{calc}}$ (Å)	$h$	$k$	$l$
17	6.306	6.316	0	0	2
41	3.848	3.869	0	1	1
4	3.149	3.158	0	0	4
100	2.923	2.924	0	1	3
68	2.875	2.874	1	1	0
12	2.110	2.105	0	0	6
10	2.049	2.032	0	2	0
9	1.719	1.709	0	2	4
14	1.680	1.669	1	2	3
3	1.646	1.649	0	1	7
2	1.440	1.437	2	2	0
3	1.281	1.281	1	2	7

Data acquired using MoK $\alpha$  radiation.

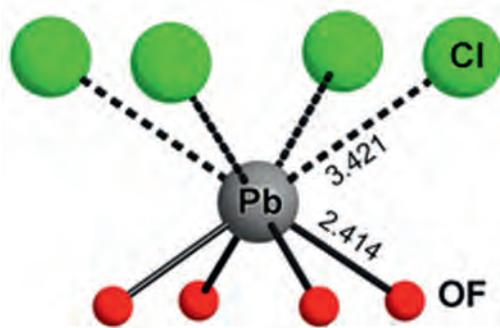


FIG. 4. Coordination of the  $\text{Pb}^{2+}$  cation in rumseyite.

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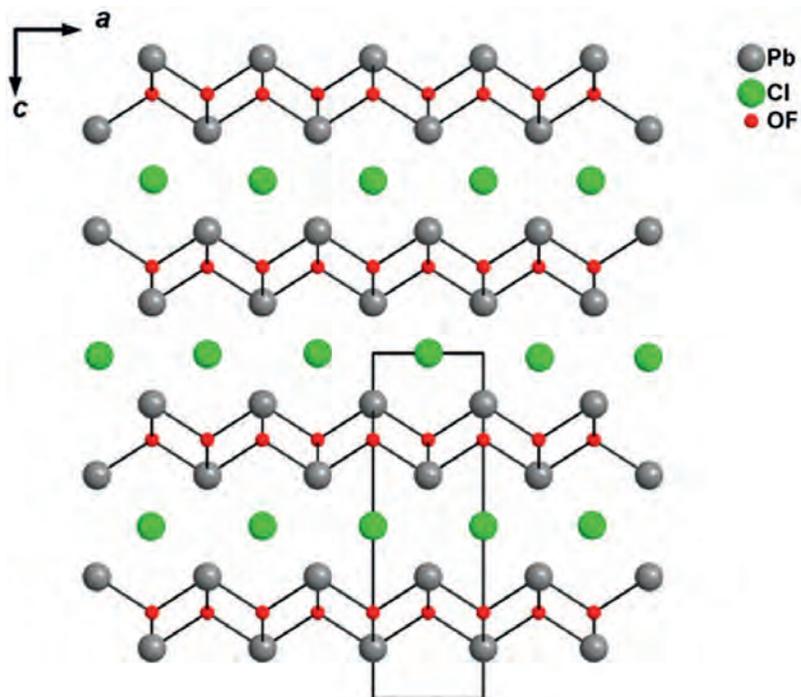


FIG. 5. Projection of the crystal structure of rumseyite along the *b* axis. The Pb–Cl bonds are omitted for clarity.

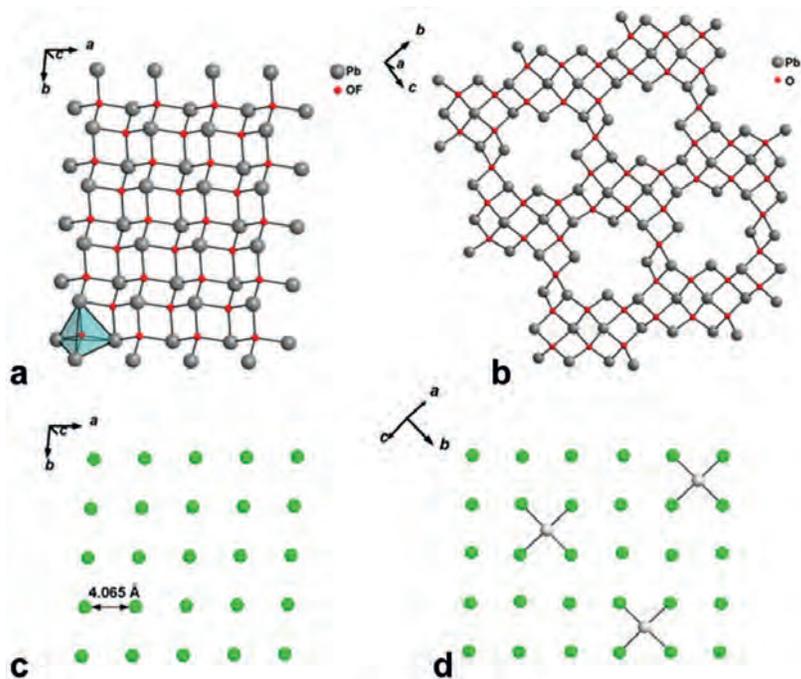


FIG. 6. (a) Projection of the  $[OFPb_2]$  layer and (c) the chloride layer in rumseyite. (b) The PbO-like sheet with square vacancies in the crystal structure of symesite and (d) Pb atoms inserted into the halide sheet in mereheadite.

minerals; it is the first Pb oxychloride mineral containing defect-free halide and PbO-like layers. Lead oxychlorides with layered defect-free PbO-derived blocks were first described in synthetic compounds by Aurivillius (1982, 1983) but rumseyite is the first example of mineral with this type of atomic arrangement.

Rumseyite is also the first example of a mineral crystal structure containing mixed O/F anion-centred tetrahedra. The fluoride anion plays an important role as a charge compensating agent within the O–Pb sheets. As F and O have similar electron densities, they are difficult to distinguish by single-crystal XRD; an attempt was made to refine the occupancy of unique sites for oxygen and fluorine in order to assign rumseyite to a lower symmetry space group. However, no indication of lower symmetry was found. The data were also checked using the *PLATON* computer program to verify these findings.

Rumseyite appears to have formed in a highly unusual environment as fluorine is otherwise absent from the manganese pod mineral assemblages. The only fluorine-bearing mineral present in the Mendips is fluorite, which is widespread but scarce in the original primary galena-bearing veins. It is possible that a small trapped fluorite crystal was the original source of the fluorine in rumseyite.

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