

Somersetite, $\text{Pb}_8\text{O}(\text{OH})_4(\text{CO}_3)_5$, a new complex hydrocerussite-related mineral from the Mendip Hills, England

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ABSTRACT

The new mineral somersetite, has been found at Torr Works ('Merehead quarry') in Somerset, England, United Kingdom. Somersetite is green or white (typically it is similar visually to hydrocerussite-like minerals but with a mint-green tint), forms plates and subhedral grains up to 5 mm across and up to 2 mm thick. In bi-coloured crystals it forms very thin intergrowths with plumbonacrite. The empirical formula of somersetite is $\text{Pb}_{8.00}\text{C}_{5.00}\text{H}_{4.00}\text{O}_{20}$. The simplified formula is $\text{Pb}_8\text{O}(\text{OH})_4(\text{CO}_3)_5$, which requires: $\text{PbO} = 87.46$, $\text{CO}_2 = 10.78$, $\text{H}_2\text{O} = 1.76$, total 100.00 wt.%.

The infrared spectrum of somersetite is similar to that of plumbonacrite and, to a lesser degree, hydrocerussite. Somersetite is hexagonal, $P6_3/mmc$, $a = 5.2427(7)$, $c = 40.624(6)$ Å, $V = 967.0(3)$ Å³ and $Z = 2$. The eight strongest reflections of the powder X-ray diffraction (XRD) pattern [$d, \text{Å}(I)(hkl)$] are: 4.308(33) (103), 4.148(25)(104), 3.581(40)(107), 3.390(100)(108), 3.206(55)(109), 2.625(78)(110), 2.544(98)(0.0.16) and 2.119(27)(1.0.17). The crystal structure was solved from single-crystal XRD data giving $R_1 = 0.031$. The structure of somersetite is unique and consists of the alternation of the electroneutral plumbonacrite-type $[\text{Pb}_5\text{O}(\text{OH})_2(\text{CO}_3)_3]^{0-}$ and hydrocerussite-type $[\text{Pb}_3(\text{OH})_2(\text{CO}_3)_2]^{0-}$ blocks separated by stereochemically active lone electron pairs on Pb^{2+} . There are two blocks of each type per unit cell in the structure, which corresponds to the formula $[\text{Pb}_5\text{O}(\text{OH})_2(\text{CO}_3)_3][\text{Pb}_3(\text{OH})_2(\text{CO}_3)_2]$ or $\text{Pb}_8\text{O}(\text{OH})_4(\text{CO}_3)_5$ in a simplified representation. The 2D blocks are held together by weak Pb–O bonds and weak interactions between lone pairs.

KEYWORDS: somersetite, hydrocerussite, plumbonacrite, new mineral, lead, carbonate, layered structure, Merehead quarry, Torr works, lone electron pair.

Introduction

MEREHEAD quarry (now called, Torr Works quarry by its current owners) is well known for its suite of

rare Pb minerals found within so called 'manganese pods' (Turner and Rumsey, 2010). It is the type locality for six Pb-oxychlorides and the neotype locality for the redefined lead basic carbonate, plumbonacrite (Rumsey *et al.*, 2012).

The new Pb basic carbonate mineral somersetite was discovered in a sample that was collected

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during a Russell Society field trip to Merehead in summer 2006, but has since been visually identified on specimens collected in the 1980s. Somersetite (Cyrillic: сомeрсетит) is named for the locality in the county of Somerset in South West England. The mineral and mineral name have been approved by the International Mineralogical Association Commission on New Minerals and Mineral Names (IMA2017-024). Type material is deposited in the collections of the Department of Mineralogy, Saint-Petersburg State University, Saint-Petersburg, Russia, under the catalogue number 1/19661. A number of other visually identical samples from the 2006 find and earlier collecting trips are housed in the extensive collection of Merehead quarry specimens at the Natural History Museum in London.

Occurrence and association

Torr Works quarry is located near the town of Cranmore in Somerset, England, in the Mendip Hills. Worked for aggregate, it is well known for its suite of rare Pb minerals (Turner and Rumsey, 2010). The quarry works Carboniferous limestone strata, which at a simplified regional level form a plunging anticline, having a roughly E–W axis and plunging towards the East. Folding, regional uplift, and subsidence caused the limestone to fracture, and therefore there are now numerous joints, as well as sub-parallel fractures and faults of all sizes, all roughly aligned with the axis of the anticline.

The regional uplift, surface erosion and subsequent subsidence and marine transgression has led to the Carboniferous strata being overlain discontinuously by Jurassic Upper Oolites. The discontinuity represents some 170 M.y. of erosion, and apart from rare and localized preserved wash features preserved in the palaeosurface, the entire Permo–Triassic succession has been removed by erosion. The basement consists of Silurian volcanics, mostly andesites, which are exposed locally at Moons Hill quarry, for example.

Across the Mendip Hills, this structural geology has controlled mineralization, including the emplacement of Mississippian Pb ore veins bearing galena, fluorite, calcite and baryte into the Carboniferous limestone, resulting in a large area being irregularly mineralized with sub-parallel ore veins. After their deposition, regional uplift, erosion and subsequent marine transgression allowed the ingress of seawater into many of these Pb veins, where auto-catalysis then caused

deposition of manganate minerals (Turner, 2006). Much later, multiple hydrothermal events occurred, and hot hydrothermal fluids followed the same structural features. Hot enough to silicify the limestone wallrocks in places, they caused irregular thermal alteration of existing Pb veins, eventually leading to formation of the ‘manganese pods’ for which Merehead is famous. These hydrothermal events also deposited quartz and other minerals, and must have led to the remobilization of Pb into solution as hydrocerussite is (locally) common in these hydrothermal veins, even where there are no other Pb minerals present. Crude crystals of hydrocerussite up to 50 mm in size and aggregates to 150 mm have been found in this environment. The new mineral somersetite was found in one such vein. Associated with symesite, calcite, aragonite and quartz (in well-formed crystals up to ~50 mm – exceptionally large for the Mendips) were masses of what appeared to be hydrocerussite of an unusual pale green colour. Up to ~25 mm across, these crystals have proved to contain somersetite and plumbonacrite. Further somersetite samples at the Natural History Museum from earlier finds have a similar mineralogical assemblage and are associated largely with calcite within manganese oxide, however, a few have been identified that show other minerals in close association; directly adjacent to orange-yellow mereheadite, thin coatings of cerussite and tiny flecks of blue diabolite, and in proximity to pale-yellow mimetite and in one example, well-formed large crystals of baryte.

Physical properties and optical data

In the samples studied, somersetite forms plates and subhedral grains (Fig. 1a) up to 5 mm across and up to 2 mm thick. In bi-coloured crystals (Fig. 1b) it forms intergrowths with plumbonacrite (Fig. 2). Note that in the samples investigated, hydrocerussite does not associate directly with somersetite, which may indicate different stability fields of these minerals. Somersetite is green or white (most commonly, its visually similar to hydrocerussite-like minerals but with a mint-green tint), with white streak and adamantine lustre. It is brittle with a perfect cleavage on (001). Parting was not observed, and its fracture is uneven across the cleavage. Density could not be measured due to a lack of material and the absence of heavy liquids with densities higher than 7 g cm^{-3} . Density calculated using the empirical formula is equal to 7.01 g cm^{-3} . Somersetite has a Vickers Hardness



FIG. 1. (a) Somerssetite crystal (green subhedral grain in the vug), field of view = 1 cm and (b) multi-coloured somerssetite crystal (2 cm across) with inclusions of plumbonacrite and colourless transparent cerussite in the rim. The green zone is enriched in somerssetite, but plumbonacrite was also determined from this area, whereas the white zone is enriched in plumbonacrite. All of these minerals can be identified reliably, by X-ray techniques.

Number (VHN20) of 140.4 kg/mm² ($n = 3$, range 111–167 kg/mm²), which corresponds to a Mohs hardness of ~3.

Somerssetite is optically negative. Despite the transparency of somerssetite, its optical properties were investigated in reflected light because of the high values of refractive indices. A calculated mean refractive index is 2.00 and $K_C = 0.1451$ was obtained from the chemical data in Table 1. Reflectance measurements were made using a SiC standard in air in the range 400–700 nm (Table 2). In reflected light, somerssetite is grey. It is non-pleochroic, with white internal reflections and a very weak bireflectance. The measured anisotropism is $\Delta R_{589} = 1\%$; under the optical microscope,

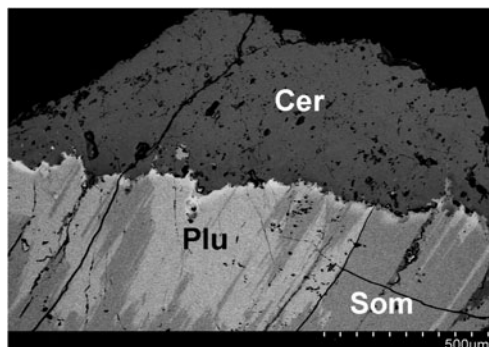


FIG. 2. Scanning electron microscopy image showing mutual intergrowths of somerssetite (dark grey) with plumbonacrite (lighter grey) and cerussite (very dark) in the rim.

the relatively weak anisotropy is masked by the abundant internal reflections of white and blue with yellowish tints.

Chemical composition

Two samples of somerssetite have been studied, the first is a 15 µm × 10 µm crystal, which was used for single-crystal analysis, and the second is a large sample (ca. 7 mm × 5 mm × 7 mm) consisting of somerssetite, plumbonacrite, cerussite, symesite and an unidentified Pb-Cl-C(?) mineral (Fig. 2). The composition of somerssetite (Table 1) was determined by wavelength-dispersive spectrometry (WDS) using a Cameca SX-100 electron microprobe (Natural History Museum, London, UK) with an acceleration voltage of 20 kV, beam current of 20 nA and a variable electron-beam diameter of 1, 5 and 10 µm. The mineral is stable under the electron beam and WDS produced consistent compositional data. Full spectrometer scans were obtained using the following diffracting crystals: LPCO ($d = 45 \text{ \AA}$), TAP ($d = 25.745 \text{ \AA}$), LPET (8.742 Å) and LLIF (4.027 Å). Only peaks originating from Pb and O were observed in the scans. Spectrometer scans were not obtained from a larger 'd' spacing crystal as the sample was carbon coated. Vanadinite was used as the standard for the determination of Pb. Calcium, Fe, Mn, Co, Zn, V, Mo and As were included in the analytical protocol, but all determined values were below the detection limit. Fluorine, Cl and S were also found to be below the detection limit (530, 720 and 260 ppm respectively). Corrections for peak interferences (Pb/S, Pb/Si and Pb/Mo) were applied from empirical measurements taken from standards. These elements were also determined to be below the

TABLE 1. Chemical composition of somersetite.

Constituent	Wt.%	Range	S.D.	Probe standard
Crystal 1				
PbO	86.77	86.54–87.12	0.23	Vanadinite
CO ₂ *	10.70			
H ₂ O*	1.75			
Total	99.22			
Crystal 2				
PbO	88.12	87.47–88.82	0.49	Vanadinite
CO ₂ *	10.85			
H ₂ O*	1.78			
Total	100.75			

*Calculated values.

S.D. – Standard deviation.

detection limit. CO₂ was initially calculated by difference to the analytical total prior to PAP matrix correction (Pouchou and Pichoir, 1991). Stoichiometric CO₂ and H₂O were then derived by calculation from the crystal structure (5 (CO₃)²⁻ and 4 (OH)⁻ groups).

The content of PbO within somersetite from two analysed crystals is slightly different (average values are 86.77 wt.% for the first sample and 88.12 for the second). The empirical formulas of the mineral (Table 1), calculated on the basis of 20 O atoms per formula unit, are identical Pb_{8.00}C_{5.00}H_{4.00}O₂₀. The simplified formula is Pb₈O(OH)₄(CO₃)₅, which requires: PbO = 87.46, CO₂ = 10.78, H₂O = 1.76, total 100.00 wt.%.

TABLE 2. Reflectance values of an unoriented section free of internal reflections on a measured area of 20 μm × 20 μm (SiC standard, measured in air) for somersetite.

λ, nm	R _{min} /R _{max} , %	λ, nm	R _{min} /R _{max} , %
400	12.98/14.06	560	12.13/13.15
420	12.82/13.92	580	12.05/13.06
440	12.70/13.77	589	12.02/13.02
460	12.59/13.65	600	11.98/12.98
470	12.54/13.58	620	11.90/12.89
480	12.49/13.52	640	11.82/12.80
500	12.39/13.41	650	11.78/12.75
520	12.30/13.31	660	11.73/12.70
540	12.20/13.23	680	11.64/12.62
546	12.18/13.21	700	11.56/12.52

Note: The values required by the Commission on Ore Mineralogy are given in bold.

Infrared spectroscopy

In order to obtain infrared (IR) absorption spectra, powdered samples of crushed single crystals previously checked by single-crystal XRD were mixed with anhydrous KBr, pelletized, and analysed using an ALPHA FTIR spectrometer (Bruker Optics) with the resolution of 4 cm⁻¹; 16 scans were obtained. The IR spectrum of analogous pellet of pure KBr was used as a reference.

The IR spectrum of somersetite is similar to that of plumbonacrite and, to a lesser degree, hydrocerussite (Fig. 3). The wavenumbers of absorption bands in the IR spectrum of somersetite and their assignments are (cm⁻¹; s – strong band, w – weak band, sh – shoulder): 3549, 3280w (O–H-stretching vibrations); 2524w (combination mode), 1734w (overtone of in-plane bending vibrations of CO₃²⁻ anions); 1403s (asymmetric C–O-stretching vibrations of CO₃²⁻ anions); 1217 (possibly, combination mode involving Pb–O-stretching and O–C–O bending vibrations); 1046w (symmetric C–O-stretching vibrations of CO₃²⁻ anions); 849 (out-of-plane bending vibrations of CO₃²⁻ anions); 738w, 690sh, 683s (in-plane bending vibrations of CO₃²⁻ anions); 615w (Pb···O–H bending vibrations), 507w, 391 (Pb–O-stretching vibrations). The absence of absorption bands in the range from 1550 to 1700 cm⁻¹ indicates the absence of H₂O molecules in somersetite.

Powder XRD data

Powder XRD data (Britvin *et al.* 2017) were obtained after single-crystal XRD from crushed fragments of 1/19661 using a Rigaku RAXIS

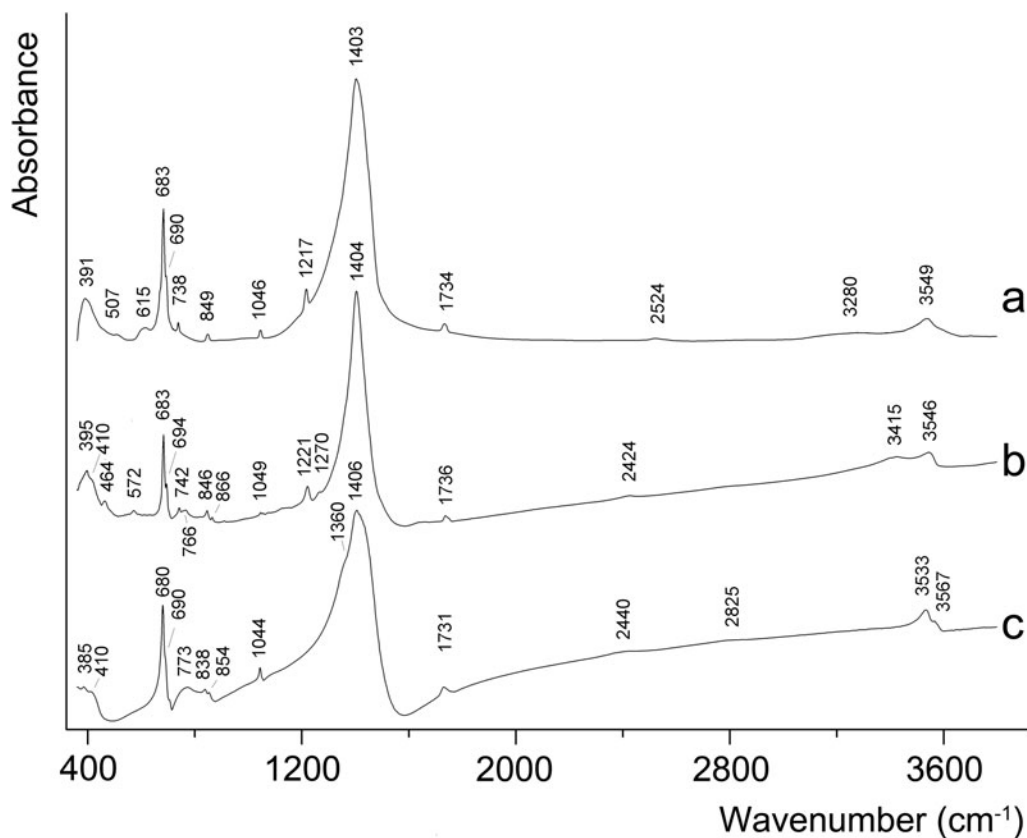


FIG. 3. IR spectra of (a) somersetite; (b) plumbonacrite from Långban, Sweden and (c) hydrocerussite from Merehead quarry, England.

Rapid II single-crystal diffractometer (CoK α) equipped with a cylindrical image plate detector using Debye-Scherrer geometry (with $d = 127.4$ mm). Data are given in Table 3. The unit-cell parameters were refined for a hexagonal unit cell, space group $P6_3/mmc$, $a = 5.249(1)$, $c = 40.679(2)$ Å, $V = 970.89(55)$ Å³ and $Z = 2$.

Crystal structure

Experiment

A crystal of somersetite from sample 1/19661 was mounted on a thin glass fibre and mounted on a Bruker DUO APEX II CCD four-circle diffractometer with a Mo- λ uS micro-focus tube at 50 kV and 40 mA. More than a hemisphere of XRD data was collected with frame widths of 0.5° in ω , and with 90 s spent counting for each frame. The data were integrated and corrected for absorption using

an empirical ellipsoidal model using the *APEX* and *XPREP* Bruker programs. The observed systematic absences for somersetite were consistent with the space group $P\bar{3}1c$. In this space group, light atoms (C, O) could not be refined anisotropically. The obtained structure model was transformed to the space group $P6_3/mmc$ using the *ADDSYM* algorithm incorporated in the *PLATON* program package (Le Page, 1987; Spek, 2003). The structure was refined successfully with the use of *SHELX* (Sheldrick, 2015). Structure refinement resulted in the crystallographic agreement index $R_1 = 0.031$ (Table 4). The final atomic coordinates and anisotropic displacement parameters are given in Table 5 and selected interatomic distances in Table 6. Hydrogen atom positions were not localized. The structural model involves disorder of several intrablock Pb positions. Attempts to eliminate the disorder by using different space groups were not successful, indicating that disorder is

TABLE 3. Powder XRD data for somersetite*.

<i>h k l</i>	Calculated powder pattern		Experimental powder pattern	
	<i>d</i> Å	<i>I</i> %	<i>d</i> Å	<i>I</i> %
1 0 1	4.512	8	4.515	7
1 0 2	4.431	9	4.445	24
1 0 3	4.305	31	4.308	33
1 0 4	4.145	22	4.148	25
1 0 6	3.771	16	3.772	21
1 0 7	3.576	30	3.581	40
1 0 8	3.385	100	3.390	100
1 0 9	3.201	54	3.206	55
0 0 14	2.902	2	2.909	12
1 0 11	2.865	0	2.859	6
1 0 12	2.714	8	2.715	8
1 1 0	2.621	62	2.625	78
0 0 16	2.539	32	2.544	98
1 1 6	2.445	7	2.448	7
2 0 3	2.239	14	2.239	22
2 0 4	2.215	13	2.220	25
2 0 6	2.152	8	2.154	15
1 0 17	2.115	9	2.119	27
2 0 8	2.072	10	2.075	15
2 0 9	2.028	10	2.031	17
1 0 19	1.934	2	1.938	6
2 0 12	1.885	8	1.889	8
1 0 20	1.854	1	1.857	3
1 1 16	1.824	25	1.827	18
2 1 1	1.715	1	1.716	4

*The eight strongest lines are shown in bold.

TABLE 4. Crystallographic data and structure refinement details for somersetite.

Crystal data	
Crystal system	Hexagonal
Space group	$P6_3/mmc$
Unit-cell dimensions	
<i>a</i> (Å)	5.2427(7)
<i>c</i> (Å)	40.624(6)
Unit-cell volume (Å ³)	967.0(3)
<i>Z</i>	2
Calculated density (g·cm ⁻³)	7.01
Absorption coefficient (mm ⁻¹)	69.48
Crystal size (mm)	0.10×0.10×0.03
Data collection	
Temperature (K)	150(2)
Radiation, wavelength (Å)	MoK α (0.71073)
<i>F</i> (000)	1700
θ range (°)	2.01–27.93
Index ranges	$-6 \leq h \leq 5$ $-6 \leq k \leq 6$ $-53 \leq l \leq 53$
Total reflections collected	9431
Unique reflections (R_{int})	518 (0.09)
Unique reflections $F > 4\sigma(F)$	509
Structure refinement	
Refinement method	Full-matrix least-squares on F^2
Weighting coefficients <i>a</i> , <i>b</i>	0.009700, 28.653200
Data/restraints/parameters	518/57/6
R_1 [$F > 4\sigma(F)$], wR_2 [$F > 4\sigma(F)$]	0.031, 0.032
R_2 all, wR_2 all	0.073, 0.074
Gof on F^2	1.232

characteristic of the structure. This feature is typical for the other Pb basic carbonate minerals structurally related to hydrocerussite (Krivovichev and Burns, 2000; Martinetto *et al.*, 2002; Siidra *et al.*, 2018a,b,c). The crystallographic information files have been deposited with the Principal Editor of *Mineralogical Magazine* and are available as Supplementary material (see below).

Single-crystal XRD is a reliable method for the determination of Pb basic carbonate minerals related to hydrocerussite. However, in some cases confusion may occur during the preliminary check of the unit-cell parameters. Somersetite and plumbonacrite form very fine mutual intergrowths (Fig. 2) in the aggregates from the Torr Works quarry. These intergrowths may mimic a good quality 'single crystal'. The latter results in the wrong determination of the unit-cell parameters as $a = 9.0929(5)$ Å and $c = 40.660(6)$ Å. The *a* parameter is identical with plumbonacrite, whereas the *c* value is the same as in

somersetite (Table 7). 'Crystals' with this pseudo unit-cell were checked by powder XRD and expectedly provided patterns of the somersetite with plumbonacrite mixture. We suggest that some of the 'hydrocerussite'-like phases from Torr Works quarry, studied previously at the Natural History Museum, London and described in Turner and Rumsey (2010) also correspond to the mixtures of layered Pb basic carbonate minerals. As different stackings of the electroneutral sheets described below may result in the other unusual minerals, the structural architectures related to hydrocerussite and all hydrocerussite occurrences should be evaluated carefully.

Cation and anion coordination

The structure of somersetite contains five symmetrically unique Pb positions (Table 5). Coordination

TABLE 5. Atomic coordinates and displacement parameters (\AA^2) for somersetite.

Atom	BVS [†]	<i>x/a</i>	<i>y/b</i>	<i>z/c</i>	<i>U</i> _{eq}	<i>U</i> ¹¹	<i>U</i> ²²	<i>U</i> ³³	<i>U</i> ²³	<i>U</i> ¹³	<i>U</i> ¹²
Hydrocerussite block [Pb ₃ (OH) ₂ (CO ₃) ₂] ⁰											
Pb1	2.23	2/3	1/3	0.06809(2)	0.0167(2)	0.0167(3)	0.0167(3)	0.0166(4)	0.000	0.000	0.00836(15)
Pb2*	1.80	0.0834(3)	0.1668(6)	0.99947(9)	0.0264(8)	0.0346(15)	0.0182(14)	0.0210(11)	−0.0019(16)	−0.0010(8)	0.0091(7)
C1	3.90	1/3	2/3	0.0570(4)	0.005(2)	0.005(2)	0.005(2)	0.004(3)	0.000	0.000	0.0025(10)
OH1	1.46	2/3	1/3	0.0138(4)	0.039(5)	0.051(8)	0.051(8)	0.016(8)	0.000	0.000	0.026(4)
O1	2.16	0.6182(17)	0.8091(8)	0.0577(2)	0.0183(17)	0.004(4)	0.017(3)	0.029(4)	0.0013(16)	0.003(3)	0.0018(19)
Plumbonacrite block [Pb ₅ O(OH) ₂ (CO ₃) ₃] ⁰											
Pb3	2.20	0	0	0.12475(2)	0.0217(3)	0.0259(3)	0.0259(3)	0.0132(4)	0.000	0.000	0.01293(17)
Pb4**	2.08	1/3	2/3	0.23911(4)	0.0154(4)	0.0152(5)	0.0152(5)	0.0156(6)	0.000	0.000	0.0076(2)
Pb5***	1.88	0.58135(15)	0.41865(15)	0.19068(3)	0.0209(3)	0.0275(6)	0.0275(6)	0.0173(6)	0.0011(3)	−0.0011(3)	0.0209(6)
C2	3.93	1/3	2/3	0.1374(6)	0.018(4)	0.005(5)	0.005(5)	0.045(13)	0.000	0.000	0.002(3)
C3	4.05	0	0	1/4	0.014(5)	0.018(9)	0.018(9)	0.006(11)	0.000	0.000	0.009(4)
OH2	1.18	0	0	0.1797(4)	0.053(6)	0.079(10)	0.079(10)	0.001(6)	0.000	0.000	0.039(5)
O2	2.15	0.8593(12)	0.1407(12)	0.2500	0.019(2)	0.016(4)	0.016(4)	0.029(6)	0.000	0.000	0.011(5)
O3	2.24	0.4752(9)	−0.0496(17)	0.3617(2)	0.0238(19)	0.019(3)	0.006(4)	0.042(5)	0.001(4)	0.0005(18)	0.003(2)
O4**	1.99	1/3	2/3	0.2072(8)	0.028(7)	0.029(11)	0.029(11)	0.027(17)	0.000	0.000	0.015(6)

*Site occupancy factor (SOF) = 1/6; ** SOF = 1/2; *** SOF = 1/3. † BVS – bond-valence sums calculated using bond-valence parameters from Krivovichev and Brown (2001) for the Pb²⁺–O bonds and from Brese and O’Keeffe (1991) for the C–O bonds.

TABLE 6. Selected interatomic distances in the crystal structure of somersetite.

Hydrocerussite block [Pb ₃ (OH) ₂ (CO ₃) ₂] ⁰		Plumbonacrite block [Pb ₅ O(OH) ₂ (CO ₃) ₃] ⁰	
Pb1–OH1	2.21(1)	Pb3–OH2	2.23(1)
Pb1–O1	2.664(1) × 6	Pb3–O3	2.688(2) × 6
Pb1–O3	3.339(9) × 3	Pb3–O1	3.229(8) × 3
Pb2–Pb2	0.759(3) × 2	Pb4–Pb4	0.885(3)
Pb2–Pb2	1.312(5) × 2	Pb4–O4	2.18(3)
Pb2–Pb2	1.516 (6)	Pb4–O2	2.6688(11) × 6
Pb2–OH1	2.332(5)		
Pb2–O1	2.558(9)	Pb5–Pb5	1.342(2) × 2
Pb2–O1	2.767(8) × 2	Pb5–O3	2.337(9)
Pb2–OH1	2.789(4) × 2	Pb5–O4	2.350(10)
Pb2–O1	3.238(9) × 2	Pb5–OH2	2.760(3) × 2
Pb2–O1	3.405(9)	Pb5–O2	2.848(6) × 2
Pb2–OH1	3.510(3)	Pb5–O3	3.083(8) × 2
		Pb5–O2	3.490(8)
C1–O1	1.294(8) × 3	C2–O3	1.289(8) × 3
		C3–O2	1.278(11) × 3

spheres of the Pb²⁺ cations are different due to the different degree of the stereochemical activity of 6s² lone electron pairs. In one coordination hemisphere, Pb1 and Pb3 atoms are coordinated by

seven oxygen atoms at rather short Pb–O distances <3.0 Å. The resulting PbO₇/PbO₆OH [1 + 6] configuration is complemented by three additional long Pb–O bonds in another coordination hemisphere. The half occupied split Pb4 site is coordinated rather symmetrically by six Pb4–O2 bonds of 2.6688(11) Å each in plane and two additional apical Pb4–O4 bonds of 2.18(3) Å each. Coordination spheres of the split Pb2 site (SOF = ½) and Pb5 site (SOF = ⅓), despite the strong disorder, also demonstrate a rather uniform distribution of short and long bonds, which suggests that these atoms are not as stereochemically active as the other Pb sites. Very similar disorder for the Pb2 site was observed in the structure of the synthetic analogue of hydrocerussite: Pb₃(OH)₂(CO₃)₂ (Martinetto *et al.*, 2002), determined by synchrotron XRD and hydrocerussite from Merehead quarry, England (Siidra *et al.*, 2018a).

The carbonate triangles are very similar and show typical bond lengths, with a <C–O> of 1.294(8), 1.289(8) and 1.278(11) Å for C1-, C2- and C3-centred triangles, respectively.

The OH1 site attributed to hydroxyl group forms one short OH1–Pb1 bond (0.61 valence units) and three weaker OH1–Pb2 bonds. Coordination of the OH1 site is identical to ones in hydrocerussite from Merehead quarry (Siidra *et al.*, 2018a) and synthetic powder material (Martinetto *et al.*, 2002). In the synthetic analogue of hydrocerussite,

TABLE 7. Comparative data of somersetite, an unnamed slag phase, hydrocerussite and plumbonacrite.

Mineral/ compound	Somersetite	Unnamed slag phase	Hydrocerussite	Plumbonacrite
Formula	Pb ₈ O(OH) ₄ (CO ₃) ₅ = [Pb ₃ (OH) ₂ (CO ₃) ₂] [Pb ₅ O(OH) ₂ (CO ₃) ₃]	NaPb ₅ (CO ₃) ₄ (OH) ₃ = [Pb ₃ (OH) ₂ (CO ₃) ₂] [NaPb ₂ (OH)(CO ₃) ₂]	Pb ₃ (OH) ₂ (CO ₃) ₂	Pb ₅ O(OH) ₂ (CO ₃) ₃
Crystal system	Hexagonal	Hexagonal	Trigonal	Trigonal
Space group	<i>P6₃/mmc</i>	<i>P6₃/mmc</i>	<i>R3m</i>	<i>P3c1</i>
<i>a</i> (Å)	5.2427(7)	5.2533(11)	5.2475(1)	9.0891–9.0921
<i>c</i> (Å)	40.624(6)	29.425(6)	23.6795(7)	24.832–24.923
<i>V</i> (Å ³)	967.0(3)	703.3(3)	564.69(1)	1776.6–1784.3
<i>R</i> ₁ (%)	3.05	4.71	2.08	4.90
<i>D</i> (g cm ⁻³)	7.01 (calc.)	6.41 (calc.)	6.80–6.82 (meas.)* 6.873 (calc.)	7.07 (meas.) 7.07 (calc.)
Sources	This work	Siidra <i>et al.</i> (2018b)	Siidra <i>et al.</i> (2018a); *Palache <i>et al.</i> (1951); Anthony <i>et al.</i> (2003);	Palache <i>et al.</i> (1951); Olby (1966); Krivovichev and Burns (2000); Rumsey <i>et al.</i> (2012)

*Palache *et al.* (1951) and Anthony *et al.* (2003) do not provide structural data.

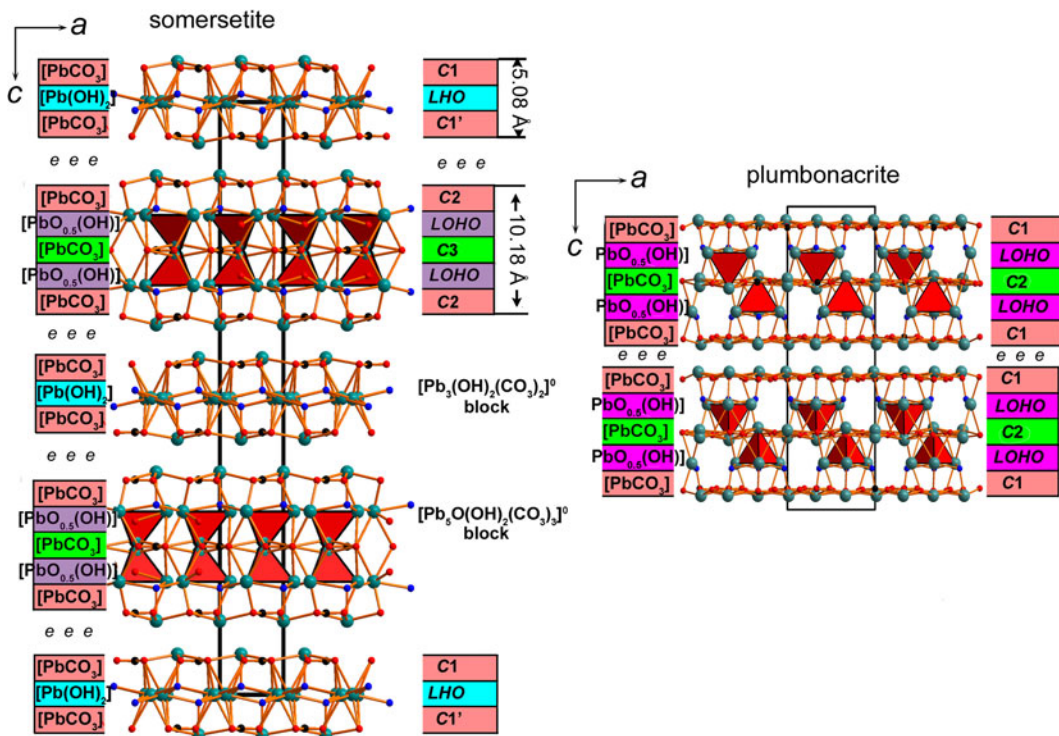


FIG. 4. General projections of the crystal structures of somersetite and plumbonacrite (only strong Pb–O bonds $< 3.0 \text{ \AA}$ are shown). Identical types of sheets are marked by the same colours. The crystal structure of somersetite is based on 2D blocks of two types: (i) $[\text{Pb}_3(\text{OH})_2(\text{CO}_3)_2]_0^0$ formed by three sheets and identical in topology and composition to those in hydrocerussite and (ii) $[\text{Pb}_5\text{O}(\text{OH})_2(\text{CO}_3)_3]_0^0$ similar to those in plumbonacrite. OPb_4 oxocentred tetrahedra are ordered in $[\text{Pb}_5\text{O}(\text{OH})_2(\text{CO}_3)_3]_0^0$ blocks in the structure of plumbonacrite. Lone pairs on Pb^{2+} cations between the blocks in the structure of somersetite and plumbonacrite are symbolized by 'e'. OPb_4 tetrahedra are marked by red. See text for details.

$\text{Pb}_3(\text{OH})_2(\text{CO}_3)_2$, the OH1 site demonstrates additional static disorder, whereas this is absent in somersetite and hydrocerussite (Siidra *et al.*, 2018a), as well as in $\text{NaPb}_5(\text{CO}_3)_4(\text{OH})_3$ (Siidra *et al.*, 2018b). The OH2 site is coordinated similarly by one Pb3 atom at $2.23(1) \text{ \AA}$ and three Pb5 atoms at $2.760(3) \text{ \AA}$ each. The O4 atom is coordinated tetrahedrally by four Pb atoms, which results in the formation of oxocentred OPb_4 tetrahedra (Krivovichev *et al.*, 2013; Siidra *et al.*, 2008a) in somersetite. The disorder of the O4 atom ($\text{SOF} = 1/2$) correlates with the disorder of the Pb4 ($\text{SOF} = 1/2$) site.

Bond-valence sums for the cations and anions were calculated using the structure are listed in Table 5. They are in general agreement with the expected oxidation states, taking into account the strong disorder of several Pb sites.

Structure description

Polyhedra PbO_n (with Pb–O bonds $< 3.1 \text{ \AA}$) share common oxygen atoms with CO_3 triangles thus forming two types of the two-dimensional (2D) blocks shown in Fig. 4. The first block is formed by Pb1, Pb2, C1, O1 and OH1 atoms, whereas Pb3, Pb4, Pb5, C2, C3, O2, O3, O4 and OH2 atoms build the second block. Each of the blocks can be split into separate sheets (Fig. 5). The outer ordered sheets in each block are topologically identical and correspond to the $[\text{PbCO}_3]_0^0$ composition. Each $[\text{PbCO}_3]_0^0$ sheet consists of Pb^{2+} cations, coordinated by three CO_3 triangles. The $[\text{PbCO}_3]_0^0$ cerussite-type (denoted as C) sheets can be cut from the framework structure of cerussite PbCO_3 . The stereochemically active lone electron pairs on Pb^{2+} cations in the Pb1 and Pb3 sites are accommodated between the layers

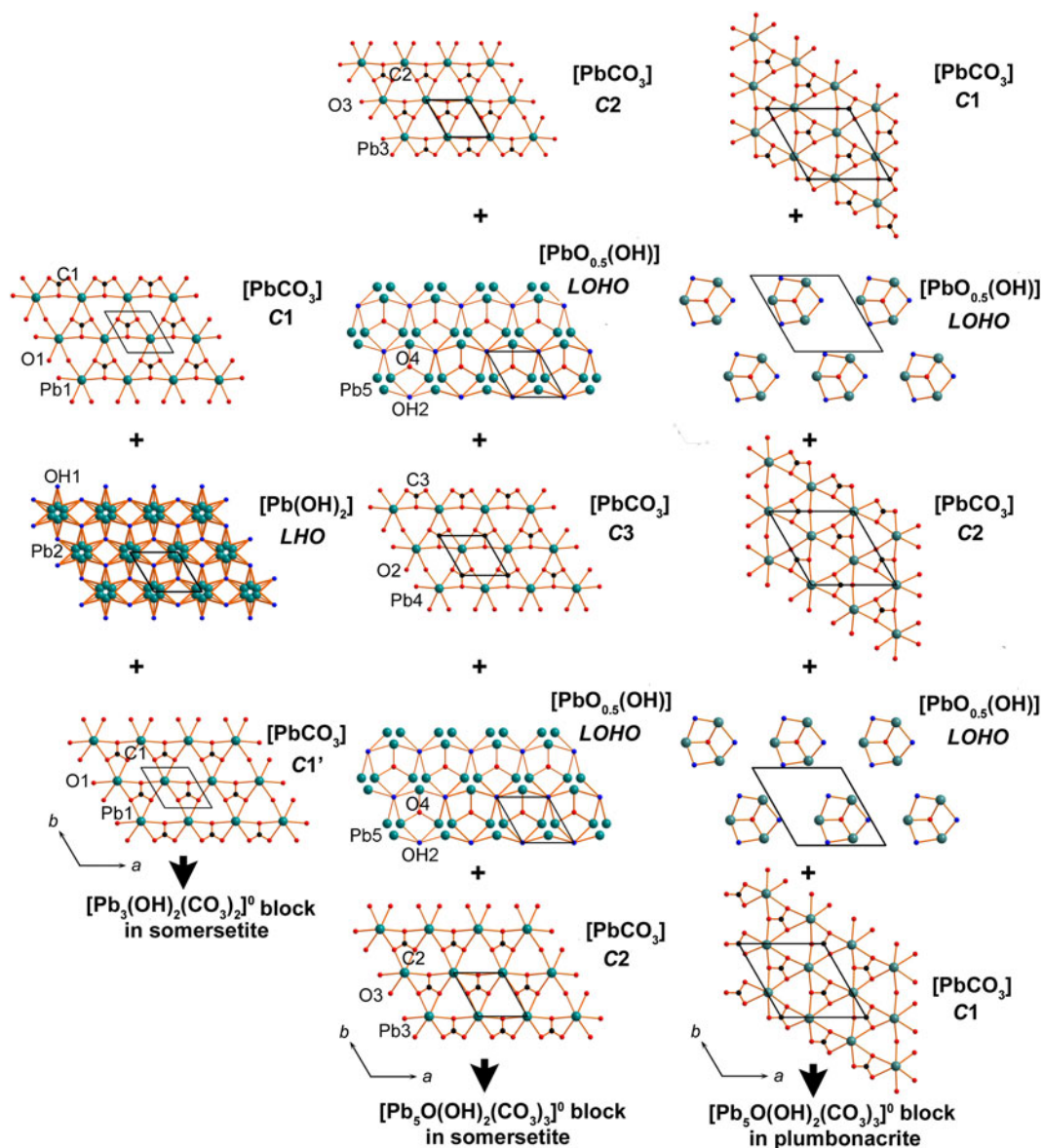


FIG. 5. Stackings of sheets in 2D blocks in the structures of somersetite and plumbonacrite. See text for details.

and point to each other (Fig. 4). The $[\text{Pb(OH)}_2]_0$ lead hydroxide (denoted as *LHO*) sheet is sandwiched between the two $[\text{PbCO}_3]_0$ sheets, shifted one relative to the other and denoted as C1 and C1' in Figs 4 and 5. The resulting $[\text{Pb}_3(\text{OH})_2(\text{CO}_3)_2]_0$ block with a 2:1 ($2[\text{PbCO}_3] : 1[\text{Pb(OH)}_2]$) sheet structure and the disorder of Pb atoms in *LHO* sheet is very similar to that in the structure of

hydrocerussite $\text{Pb}_3(\text{OH})_2(\text{CO}_3)_2$ (Siidra *et al.*, 2018a; Martinetto *et al.*, 2002) and the hydrocerussite-type block in the $\text{NaPb}_5(\text{CO}_3)_4(\text{OH})_3$ slag phase (Siidra *et al.*, 2018b).

The number of sheets in the second block in somersetite is five, identical to that in plumbonacrite (Figs 4 and 5). C2 sheets (in somersetite) and C1 sheets (in plumbonacrite) are not shifted

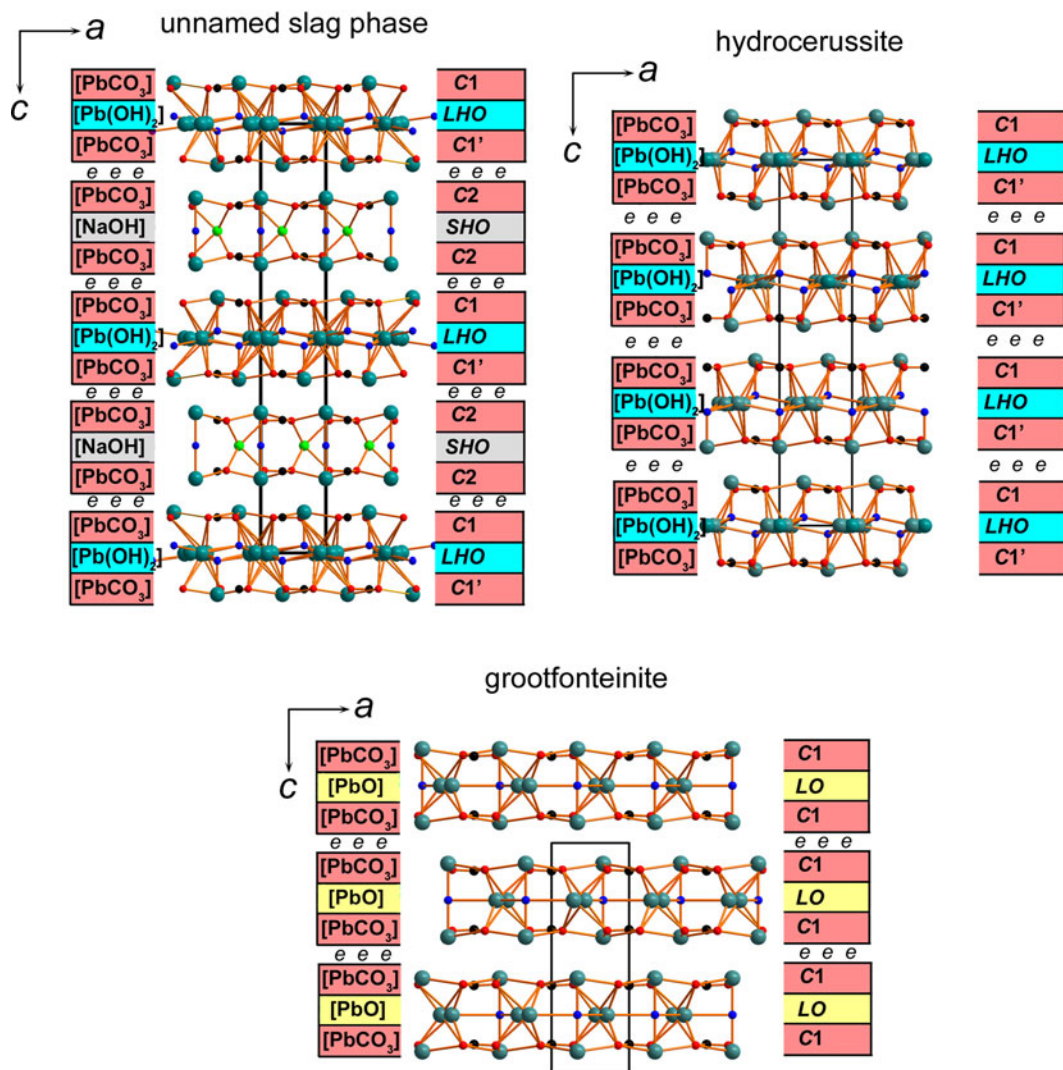


FIG. 6. General projections of the crystal structures of $\text{NaPb}_5(\text{CO}_3)_4(\text{OH})_3$ unnamed phase from Lavrion (Siidra *et al.*, 2018a); hydrocerussite (Siidra *et al.*, 2018b); and grootfonteinite (Siidra *et al.*, 2018c). The structural architecture of $\text{NaPb}_5(\text{CO}_3)_4(\text{OH})_3$ is similar to somersetite based on 2D blocks of two types: (i) $[\text{Pb}_3(\text{OH})_2(\text{CO}_3)_2]^0$ hydrocerussite type and (ii) $[\text{NaPb}_2(\text{OH})(\text{CO}_3)_2]^0$ abellaite type. Lone pairs on Pb^{2+} cations are symbolized by 'e' between the blocks in the structures of Pb basic carbonates. See text for details.

relative to each other. Also, similarly to plumbonacrite, an additional $[\text{PbCO}_3]^0$ cerussite-type C3 sheet with the disordered Pb4 site is inserted in the middle of the $[\text{Pb}_5\text{O}(\text{OH})_2(\text{CO}_3)_3]^0$ block in somersetite. The composition of the $[\text{PbO}_{0.5}(\text{OH})]^0$ lead oxide hydroxide (denoted as LOHO) sheets in somersetite is identical to that in plumbonacrite. However, LOHO sheets in somersetite demonstrate strong disorder not observed in plumbonacrite

(Fig. 7). Taking into account $\frac{1}{2}$ occupancy of the central O4 atoms in OPb_4 tetrahedra and disorder of Pb5 in somersetite, the arrangement of isolated oxocentred tetrahedra is very similar to those in plumbonacrite.

The structure of somersetite consists of the alternation of the $[\text{Pb}_5\text{O}(\text{OH})_2(\text{CO}_3)_3]^0$ and $[\text{Pb}_3(\text{OH})_2(\text{CO}_3)_2]^0$ blocks separated by stereochemically active lone electron pairs on Pb^{2+} . There are

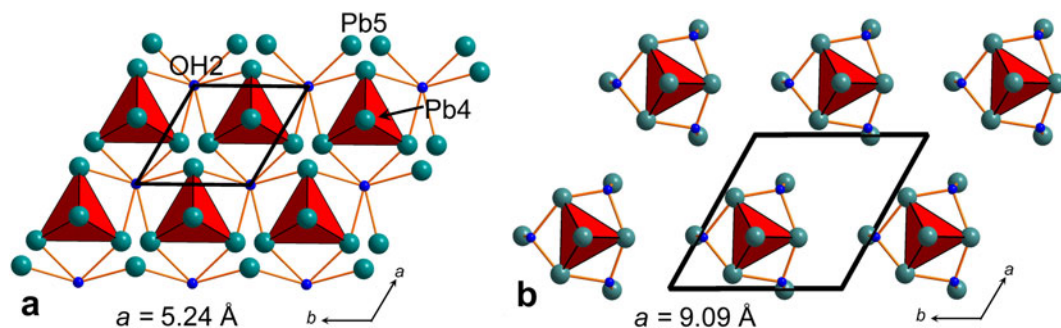


FIG. 7. Arrangement of O4-centred OPb_4 oxocentred tetrahedra in the structure of somersetite (a). Site occupation factors of atoms are listed in Table 6. OPb_4 oxocentred tetrahedra are ordered in plumbonacrite (b).

two electroneutral blocks of each type per unit cell, which corresponds to the formula $[\text{Pb}_5\text{O}(\text{OH})_2(\text{CO}_3)_3][\text{Pb}_3(\text{OH})_2(\text{CO}_3)_2]$ or $\text{Pb}_8\text{O}(\text{OH})_4(\text{CO}_3)_5$ in a simplified representation.

Discussion

Our study demonstrated the ‘green hydrocerussite’ from the Mendip Hills to be a new mineral formed by both the hydrocerussite-type $[\text{Pb}_3(\text{OH})_2(\text{CO}_3)_2]^0$ and plumbonacrite-type $[\text{Pb}_5\text{O}(\text{OH})_2(\text{CO}_3)_3]^0$ blocks. Somersetite is related directly to hydrocerussite and plumbonacrite and its structure type is new for both minerals and inorganic compounds. In its crystal structure (Fig. 4), stereochemically active $6s^2$ lone electron pairs on Pb^{2+} cations act as ‘chemical scissors’ and are responsible for the formation of well-defined electroneutral structural blocks, similar to those in other hydrocerussite-related mineral species (Fig. 6). The block size in these structures can vary and may consist of either three or five sheets. The thickness of the $[\text{Pb}_5\text{O}(\text{OH})_2(\text{CO}_3)_3]^0$ blocks in somersetite and plumbonacrite is nearly identical and corresponds to 10.18 Å and 10.14 Å, respectively. Hydrocerussite-type blocks in somersetite are half the size (5.08 Å) of the plumbonacrite type and are very similar to abellaite-type and grootfontein-type blocks. Directional lone electron pairs of the Pb^{2+} cations fill the volume between the blocks, which effectively reduces the dimensionality of the system from 3D in cerussite PbCO_3 to 2D in hydrocerussite-related minerals. The 2D blocks are held together only by very weak Pb–O bonds and weak interactions between lone pairs (Pyykö, 1997). This feature is characteristic for the other Pb basic carbonate minerals including: hydrocerussite $\text{Pb}_3(\text{OH})_2(\text{CO}_3)_2$ (Siidra *et al.*, 2018a; Martinetto

et al., 2002), plumbonacrite $\text{Pb}_5\text{O}(\text{OH})_2(\text{CO}_3)_3$ (Krivovichev and Burns, 2000; Rumsey *et al.*, 2012), abellaite $\text{NaPb}_2(\text{OH})(\text{CO}_3)_2$ (Ibáñez-Insa *et al.*, 2017), unnamed slag phase $\text{NaPb}_5(\text{CO}_3)_4(\text{OH})_3$ (Siidra *et al.*, 2018b), grootfontein-type $\text{Pb}_3\text{O}(\text{CO}_3)_2$ (Siidra *et al.*, 2018c) and leadhillite polymorphs $\text{Pb}_4(\text{SO}_4)(\text{CO}_3)_2(\text{OH})_2$ (Giuseppetti *et al.*, 1990; Steele *et al.*, 1998; Steele *et al.*, 1999). Electroneutral 2D blocks of only one topology per structure are observed in all of these minerals except for the slag phase $\text{NaPb}_5(\text{CO}_3)_4(\text{OH})_3$. The structural architecture in somersetite is the same as in $\text{NaPb}_5(\text{CO}_3)_4(\text{OH})_3$, where hydrocerussite-type $[\text{Pb}_3(\text{OH})_2(\text{CO}_3)_2]^0$ blocks alternate with abellaite-type $[\text{NaPb}_2(\text{OH})(\text{CO}_3)_2]^0$ (Fig. 6). It is also similar in this sense to the layered crystal structure of synthetic $[\text{Ti}_5^+(\text{SiO}_4)(\text{OH})_2][\text{Ti}_6^+(\text{SO}_4)(\text{OH})_4] = \text{Ti}_{16}^+(\text{SiO}_4)_2(\text{SO}_4)(\text{OH})_6$ (Siidra *et al.*, 2014).

The presence of $[\text{Pb}_5\text{O}(\text{OH})_2(\text{CO}_3)_3]^0$ blocks in somersetite reveals structural relationships with the closely associated plumbonacrite. OPb_4 tetrahedra are a typical feature of the various Pb oxychloride minerals found in association with hydrocerussite-type minerals at Torr Works: mereheadite $\text{Pb}_{47}\text{O}_{24}(\text{OH})_{13}\text{Cl}_{25}(\text{BO}_3)_2(\text{CO}_3)$ (Krivovichev *et al.*, 2009); chloroxiphite $\text{Pb}_3\text{CuO}_2(\text{OH})_2\text{Cl}_2$ (Siidra *et al.*, 2008b); $\text{Pb}_2\text{O}(\text{OH})\text{Cl}$ yeomanite (Turner *et al.*, 2015); rickturnerite $\text{Pb}_7\text{O}_4[\text{Mg}(\text{OH})_4](\text{OH})\text{Cl}_3$ (Rumsey *et al.*, 2012); rumseyite $[\text{Pb}_2\text{OF}]\text{Cl}$ (Turner *et al.*, 2012); symesite $\text{Pb}_{10}\text{O}_7\text{SO}_4\text{Cl}_4(\text{H}_2\text{O})$ (Welch *et al.*, 2000); parkinsonite $(\text{Pb},\text{Mo},\square)_8\text{O}_8\text{Cl}_2$ (Symes *et al.*, 1994); and mendipite $\text{Pb}_3\text{O}_2\text{Cl}_2$ (Turner and Rumsey, 2010).

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Supplementary material

To view supplementary material for this article, please visit <https://doi.org/10.1180/minmag.2017.081.087>

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