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RESEARCH ARTICLE

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Key Points:

- The Basque Lakes in British Columbia, Canada, are an analog for both “cold and wet” and “warm and wet” Mg-sulfate lakes on Mars
- Mineralogy varies based on season with hydrated sulfates forming in the winter pools and multi-cation salts in the fall efflorescent crusts
- Coupled mixed hydration-state Mg-sulfates may be indicative of surficial fluids interacting with a fluctuating atmosphere on Earth and Mars

Supporting Information:

Supporting Information may be found in the online version of this article.

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Seasonally Dependent Formation of Sulfates in the Basque Lakes, British Columbia, as Analogous to “Cold and Wet” and “Warm and Wet” Mars

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Abstract Saline lakes are expected to have been extensively present on ancient Mars, particularly as the planet dried or cooled. Such lakes likely deposited sulfate salts, as these salts have been widely identified from orbital and in situ Mars data. However, the relationship between martian sulfates and the environmental conditions that formed them (including whether conditions were warm, cool, drying, or freezing), remains under-characterized. To evaluate the relationship between sulfates and climate, we investigated the hypersaline, sulfate-bearing Basque Lakes in British Columbia, Canada, which serve as an analog for both “cold and wet” and “warm and wet” early Mars. We use the rover and orbiter relevant instrument techniques of Raman, Near Infrared, and X-Ray Fluorescence spectroscopy to evaluate seasonal lake mineralogy. We find that temperature-dependent, multi-cation salts form widely within the Basque Lakes' efflorescent crusts in the fall, which transform to meridianiite, mirabilite, a metastable Na-sulfate 7-hydrate, and epsomite in the wintertime. In both seasons, salt assemblages are metastable and persist beyond expected thermodynamic stability fields, suggesting ongoing climatic changes can prevent saline systems from settling into equilibrium phases. Coupled with sedimentological evidence, intimately mixed Mg-sulfates of different hydration states could be an indicator of surface fluids that interacted with an atmosphere, while formation of Na-sulfates could be evidence for brine freezing. Curiosity's exploration of the Gale crater sulfate-bearing unit and Perseverance's exploration of Jezero crater on Mars offer excellent chances to investigate the influence of climate on Mg-sulfate formation.

Plain Language Summary Early Mars was once home to lakes. These lakes either froze or evaporated over time, and concentrated waters formed salts like sulfates. Whether most of these lakes on Mars were present under cold conditions with an ice cover, or whether they were open to the environment, remains a major question. Here we investigate the Basque Lakes in British Columbia, Canada as analogs for both frozen and open lakes on early Mars, to determine how mineralogy changes based on climate, and how we can detect changes using Mars-relevant instruments. We find that in the Basque Lakes, temperature-dependent sulfates form in crusts in the fall, which melt by the winter. These crusts likely do not get preserved in the geologic record. A useful climate indicator may be the winter formation of highly hydrated Mg- and Na-sulfates in brine pools. If cold brines seep into sediment on Mars, such brines might form highly hydrated sulfates that would lose water over time, but still be detectable based on mineralogy and sedimentology. Finding sulfates that formed in cold environments would be useful for understanding Mars's ancient climate. Rovers like Perseverance and Curiosity are equipped to evaluate evidence of these cold climate salts.

1. Introduction

Paleolakes are an intriguing aspect of Mars's geologic record. Their existence attests to the presence of ancient liquid water on the surface of the planet, and the preservation of materials associated with such aqueous processes. The provenance of Mars paleolakes is diverse, but many show evidence for inflow and outflow channels, sedimentary structures, and mineral precipitation as the lakes dried or cooled (Bristow et al., 2018; Goudge et al., 2016, 2018; Grotzinger et al., 2014; Horgan et al., 2020; Stack et al., 2020; Tutolo et al., 2025). Magnesium sulfate salts in particular are considered evidence of saline fluids associated with paleolakes (e.g., Rapin et al., 2019; Wray et al., 2011). Yet major unknowns remain regarding the martian paleolake record, including their duration and climatic conditions, general trends in fluid source, and their fate as Mars underwent

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environmental transitions. These lakes may have been intermittently stable via punctuated periods of warming (Halevy & Head, 2014; Ramirez et al., 2014, 2020; Wordsworth et al., 2017, 2021), or may have persisted under broadly warmer, clement conditions (Hu & Thomas, 2022; Kite et al., 2021; Ramirez & Craddock, 2018). Paleolakes may have also existed in relatively cold and wet conditions, forming through glacial meltwaters and insulated by overlying layers of ice (Fastook & Head, 2015). On Earth, analogs like perennially ice-covered lakes may provide insight into the sedimentation and mineral formation in lakes present at times of cooling on Mars, or in a “cold and wet” early Mars scenario (McKay & Davis, 1991; Newsom et al., 1996; Rivera-Hernandez et al., 2019; Wharton et al., 1995). Throughout the history of Mars, any number of these scenarios may have been, at times, applicable.

Evaluating the geologic history of paleolakes on Mars relies on geomorphic, sedimentary, and mineralogical analyses. Earth analog lakes can be useful for recognizing the evidence for cold-climate environments, by studying lake response to climatic forcings, and how such changes affect the sedimentologic structure, mineralogy, geochemistry, and habitability of the system. However, lakes with Mars-relevant compositions—in particular, lakes with high magnesium and sulfate—are rare on Earth (Eugster & Hardie, 1978). Most lakes on Earth are freshwater rather than saline, and any saline lakes are more frequently enriched in sodium and calcium rather than magnesium (Eugster & Hardie, 1978), due to weathering of Earth's largely granitic and sedimentary source rocks, versus Mars's primarily basaltic crust (e.g., Nesbitt & Markovics, 1980; Tosca et al., 2005; White & Brantley, 2003). Studying the few examples of analog Mg-sulfate lakes on Earth is crucial for evaluating Mars-relevant geochemical and mineralogical cycling, and determining how Mg-sulfate lakes respond to environmental changes, as a potential proxy for variable climatic conditions on early Mars. Here, we investigate one such analog Mg-sulfate system: the Basque Lakes in the Cariboo Plateau of British Columbia, Canada.

The Basque Lakes are small (10 s of m² in area) endorheic lakes that likely formed initially via glacial meltwater (Renaut & Long, 1989). These lakes are rich in Mg²⁺, Na⁺ and SO₄^{2−}, with only minor contributions of Ca²⁺, CO₃^{2−} and Cl[−]. The Basque Lakes have been studied previously for their unique geochemical provenance (Cummings, 1940; Eugster & Hardie, 1978; Jenkins, 1918; Nesbitt, 1990; Renaut & Long, 1989) and relevance to Mars (Buffo et al., 2022; Nichols et al., 2023) but a systematic study of the mineralogy of these lakes using Mars rover-relevant instrumentation has not yet been published.

In this study, we evaluate the mineralogy and geochemistry of the Basque Lakes over six seasons, in the fall of 2022, 2023, and 2024, and the winters of 2023, 2024 and 2025. These seasons represent the driest and coldest periods in an annual cycle, respectively, and provide the greatest windows into how Mg-sulfate lakes change under freezing versus evaporative conditions. In the wintertime, the Basque Lakes freeze over entirely, cryoconcentrating—in other words, removing liquid water from a brine via freezing—the underlying brines. Over the summertime into the fall, the lakes evaporate, frequently to completion. We focus here on seasonal mineralogical evolution of the Basque Lakes as a proxy for “warm and wet” and “cold and wet” endmember environments on early Mars, in which Mg-sulfates may have formed in lakes. We investigate the changes in mineralogy and geochemistry with season over 3 years, with the aim of determining whether and what climatic forcings and concentration mechanisms lead to changes in mineralogy on an annual basis, and whether such changes are identifiable using rover-relevant instrumentation (Raman, near infrared (NIR), and X-ray fluorescence (XRF) spectroscopy). We also evaluate the timescales of mineralogical changes in response to climate forcings and metastability of minerals, with the aim of determining how hydration states of magnesium sulfates might help distinguish formation environment. Finally, we speculate on preservation potential of the mineralogical systems of these lakes and consider the geologic relevance to environments on Mars. We consider our results in the context of rover identifications of Mg-sulfates, acknowledging that the depositional settings may differ between our field site and stratigraphy investigated by rovers. Despite this caveat, we find that results from the Basque Lakes are informative for understanding sulfate formation and deposition on Mars.

2. Mars Relevance

Magnesium-sulfates are common on the surface of Mars (e.g., Table 1), indicating Mars was home to ancient magnesium-rich fluids, likely brines (defined generally as solutions with concentrations of solutes higher than that of Earth's seawater). Saline lakes precipitating Mg-sulfates have been inferred from orbital data (e.g., Wray et al., 2011) and in situ data from rovers like Opportunity, Perseverance and Curiosity (e.g., McLennan et al., 2005; Phua et al., 2024; Rapin et al., 2019; Siljeström et al., 2024). Extensive polyhydrated sulfates and

Table 1
Mineral Names and Formula of Relevance to This Work

Mineral name	Formula	Reference for Mars I.D.
Kieserite ^a	MgSO ₄ •1H ₂ O	Pandey et al. (2025); Chipera et al. (2023)
Starkeyite ^a	MgSO ₄ •4H ₂ O	Pandey et al. (2025); Chipera et al. (2023)
Pentahydrate ^b	MgSO ₄ •5H ₂ O	Sheppard et al. (2021)
Hexahydrate ^b	MgSO ₄ •6H ₂ O	Sheppard et al. (2021)
Epsomite ^b	MgSO ₄ •7H ₂ O	Sheppard et al. (2021)
Meridianiite ^b	MgSO ₄ •11H ₂ O	Peterson and Wang (2006)
Amorphous Mg-sulfate ^b	MgSO ₄ • <i>n</i> H ₂ O	Phua et al. (2024); Siljeström et al. (2024)
Bloedite ^b	Mg(Na ₂) (SO ₄) ₂ •4H ₂ O	Hughes et al. (2024)
Konyaite	Mg(Na ₂) (SO ₄) ₂ •5H ₂ O	N/A
Schoenite (Picromerite)	Mg(K ₂) (SO ₄) ₂ •6H ₂ O	N/A
Polyhalite	K ₂ Ca ₂ Mg(SO ₄) ₄ •2(H ₂ O)	N/A
Thenardite ^b	Na ₂ SO ₄	Gasda et al. (2022); B. C. Clark (2023)
Mirabilite	Na ₂ SO ₄ •10H ₂ O	N/A
Gypsum ^a	CaSO ₄ •2H ₂ O	Vaniman et al. (2024); Chipera et al. (2023); Pandey et al. (2025)
Anhydrite ^a	CaSO ₄	Vaniman et al. (2024); Pandey et al. (2025)
Halite ^a	NaCl	Vaniman et al. (2024); Pandey et al. (2025)
Sylvite ^b	KCl	Osterloo et al. (2008)
Magnesite ^b	MgCO ₃	Zastrow and Glotch (2021)
Alunite ^b	KAl ₃ (SO ₄) ₂ (OH) ₆	Leask et al. (2024)
Gibbsite	Al(OH) ₃	N/A

Note. References serve as examples of detections, and do not represent an exhaustive list. ^aMinerals definitively identified on Mars (via X-Ray diffraction from the CheMin instrument on board Curiosity) are identified. ^bMinerals inferred or possibly present on Mars are identified.

kieserite (MgSO₄•1H₂O, Table 1) have been identified from orbit throughout Meridiani Planum (Arvidson et al., 2005; Griffes et al., 2007; Poulet et al., 2008) and, based on Opportunity investigations, such polyhydrated sulfates are likely to be Mg-rich (Glotch et al., 2006). Observations of polyhydrated sulfates from orbit in the Gale crater sulfate-bearing unit have also been discovered to be largely Mg-sulfate rich from in situ data (Chipera et al., 2023), including the polyhydrated crystalline mineral starkeyite (MgSO₄•4H₂O). Meanwhile, the Perseverance rover has revealed Mg-sulfates of mixed hydration states present in sedimentary and igneous rocks in Jezero crater (Phua et al., 2024; Siljeström et al., 2024). Such results suggest a significant record of Mg-sulfate formation, associated with ancient paleolakes like Gale and Jezero crater, is present on Mars.

Magnesium sulfates are of particular interest in the context of Mars environmental reconstruction because the Mg-sulfate system is temperature and humidity dependent, with six different hydration states that will be discussed in this work, among additional stable or metastable hydration states (e.g., Chipera & Vaniman, 2007; Chou et al., 2013; Wang et al., 2006). For example, the Mg-sulfate kieserite—discovered across Mars from infrared absorption features and X-ray diffraction data from Curiosity's CheMin instrument—typically forms at high temperatures (>69°C from a solution, or >50°C from dehydration; e.g., Chipera & Vaniman, 2007; Chou & Seal, 2003; Grevel et al., 2012; Wang et al., 2011; Wang et al., 2009). Meanwhile, meridianiite—first identified on Earth in the Basque Lakes by Peterson et al. (2007)—is a cold-climate (≤2°C) Mg-sulfate named for Meridiani Planum on Mars due to the presence of crystal molds that might indicate dehydration from meridianiite (Peterson & Wang, 2006). That multiple hydration states of Mg-sulfates co-occur on modern Mars constitutes one of the more interesting outstanding mysteries of Mars water activity and temperature through time.

Such a mystery might be better understood when we consider that any Mg-sulfate bearing lakes, or indeed paleolakes more broadly, may also have persisted under variable climatic conditions. Lakes may have been ice-

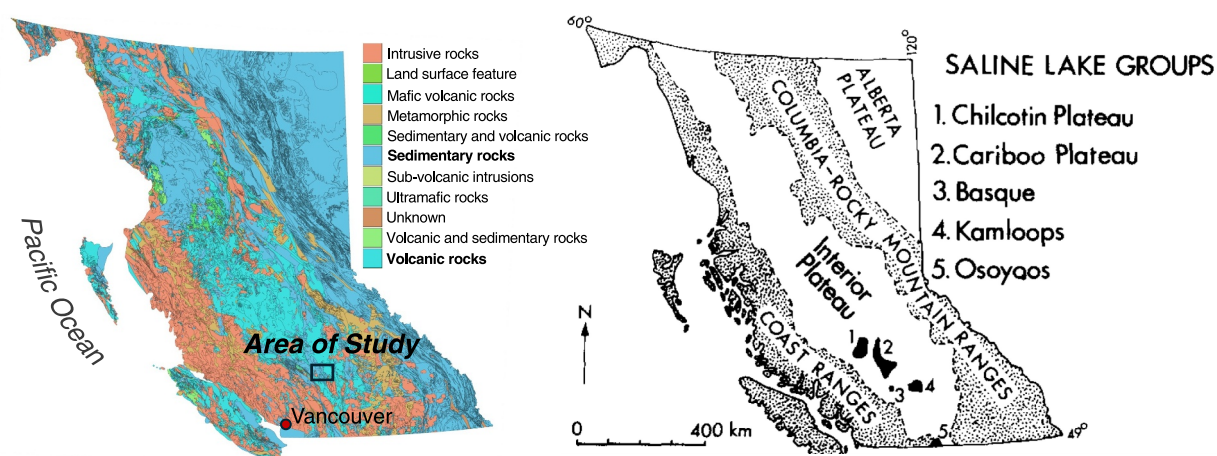


Figure 1. Left: British Columbia Geologic Survey map of rock types (Cui et al., 2017) with the area of study outlined and relevant rock types highlighted. The Basque Lakes are flanked on the west by the broadly sedimentary Cache Creek Complex, and to the east by the primarily volcanic Venables Valley assemblage. Right: Map of British Columbia adapted from (Renaut & Long, 1989) highlighting the location of the Basque Lake saline lake group, among other regional saline lakes.

covered allowing for cryoconcentration of saline fluids, open to atmospheric exchange, or a combination of both (McKay & Davis, 1991; Newsom et al., 1996; Rivera-Hernandez et al., 2019). Differences in concentration mechanism, and overall temperature and water availability of a system, lead to different stable salt hydrates (e.g., Herrero et al., 2015). Thus, the provenance of Mg-sulfate bearing lakes on Mars—such as whether these lakes were ice-covered or open—is relevant to the variability in Mg-sulfate hydrates on Mars, raising a need for further study of these relationships.

3. Geologic Setting

The Basque Lakes are located within the Interior Plateau of British Columbia, flanked on the western side by the Coastal Range and on the east by the Columbia-Rocky mountain range (Figure 1). The Interior and its surrounding mountain ranges span almost the entire southern region of British Columbia. Within this region are a large number of endorheic lake basins of varying sizes, depths, and compositions, including those within the Cariboo Plateau (Renaut & Long, 1989) and the Basque Lake group (Figure 1). The lakes within this region are some of the most diverse in the Interior, and likely in the world (Hammer, 1986). Their compositions range from broadly alkaline with Na-CO₃-SO₄-Cl systems, to highly Mg-Na-SO₄ rich. Lakes of the latter compositional endmember are the primary subject of this work.

This region is also home to the Marble Range, a mountain range primarily composed of thick to massive beds of greenstone, chert, limestone and argillite (Campbell & Tipper, 1971; Renaut & Long, 1989). The Marble Range lies to the west of the Basque Lakes, and outcrops immediately adjacent to Basque Lakes 1 and 2 (Figure 2). Basque Lakes 3 and 4 lie within the Venables Valley Assemblage (Figure 2): an Upper Permian to Middle Triassic volcanic unit composed of basalts, schists, rhyolite and local silicic volcanic materials (Childe et al., 1996). Throughout the region are 23 mya to 2.6 mya (Miocene- and Pliocene-aged) basaltic lava flows (Renaut & Long, 1989). The entire region has been heavily glaciated, contributing to the preponderance of lakes. The last glacial ice appears to have moved from the northwest to the northeast along the marble range (Campbell & Tipper, 1971), and meltwater channels are present near the northeast side of the Marble Range, indicating the regional influence of glacial melt on the hydrology. Ice retreated at minimum 9500 years ago (Clague, 1980; Fulton, 1984); the meltwater eroded glacial deposits and deposited sand- and gravel-sized sediment regionally (Tipper, 1971). The Basque lakes are broadly located within the glacial drift region.

The lakes discussed in this work are Basque Lakes 1–4. There are five Basque Lakes within 2 km, though only four of them—Basque Lake 1, or “BL1,” Basque Lake 2, or “BL2,” Basque Lake 3, or “BL3,” and Basque Lake 4, or “BL4”—are accessible via road, and therefore we do not discuss Basque Lake 5 here. While there are additional lakes near the Basque Lake group aside from those discussed in this work, these lakes are either highly dry mudflats without obvious brine pools or are difficult to access due to lack of roads. Previous studies suggest that

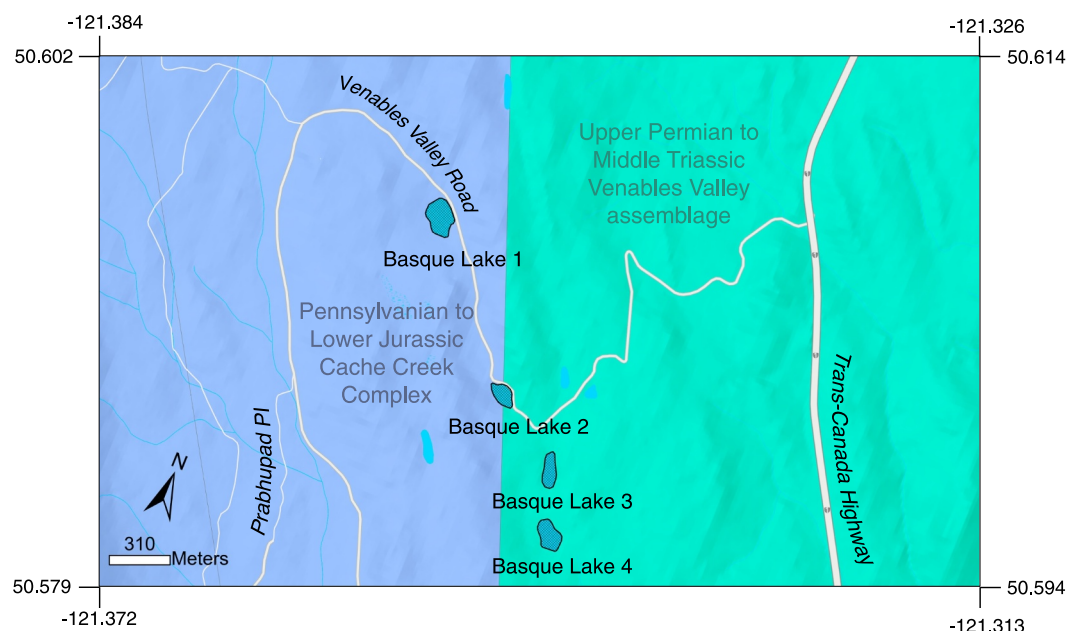


Figure 2. Map of the Basque Lakes with British Columbia Geologic Survey (BCGS) data, highlighting the Cache Creek Complex in blue (which includes the Marble Range) and Venables Valley Assemblage in turquoise. Map projection is Canadian Lambert Conformal Conic. Image Credit: BCGS, Esri Canada and Esri Community Maps Contributors.

some of the lakes surrounding Basque 1–4 are rich in mirabilite and hydromagnesite, suggesting either different watersheds, weathering sources, and/or the existence of local solubility gradients (Eugster & Hardie, 1978).

The Basque Lakes are primarily groundwater and precipitation fed, as there is no apparent source of surface inflow. Fluid leaching of serpentines, or igneous silicates like olivine and pyroxene in the Marble Range has been proposed as the fluid source for the Mg-rich brines in the Basque Lakes (e.g., Eugster & Hardie, 1978; Nesbitt, 1990). The Carboniferous Lower Cache Creek Group of the Marble Range, which is rich in dolomitic limestone, outcrops adjacent to the Basque Lake pools (Eugster & Hardie, 1978; Figure 2 of this work) and possibly also provides a source of magnesium and calcium. The brines may have become rich in sulfate via oxidation of leached sulfide, present in pyrite as an accessory mineral in the local argillites and ultramafic rocks (Renaut, 1990). Carbonate formation and zonation was identified by Nesbitt (1990), associated with an underlying ash horizon rather than within the lakes themselves. Carbonates associated with this horizon vary from primarily calcite upslope from Basque Lake 2, to magnesite-dominated closer to the lakes, indicating a precipitation gradient based on solubility and cation availability. This magnesite leaves the groundwater before collecting in the brine pools; the lake waters themselves have low alkalinity (Table 2).

The Basque Lakes brines are broadly reported as Mg-Na-SO₄ rich. Brine chemistries reported by Goudge (1924), Cummings (1940), Nesbitt (1990) and Buffo et al. (2022) all broadly agree despite nearly a century between the earliest and most recent measurements, with some potential seasonal variability and variation between lakes. Compositions measured by Buffo et al. (2022) were obtained when the lakes were frozen over, and therefore might capture differences in composition as moderated by changing precipitation sequences. We consider the fluid chemistries reported below in the context of the geochemical and mineralogical evolution of the Basque Lakes.

3.1. Lake Structure

The Basque Lakes have a “spotted” internal structure, which is uncommon for evaporative basins (Figure 3; Figure 4). BL1 and BL2 contain dozens of small, semi-circular pools of salt or brine lying within the bounds of the lake. These pools are flanked by mud-enriched raised ridges that are often covered in large amounts of salt crusts, particularly in the summer and fall. The origin of these “spotted” features—features shared by other lakes regionally, including Spotted Lake and Last Chance Lake (Pontefract et al., 2017; Renaut & Long, 1989)—

Table 2

Composition of the Basque Lakes as Measured by Goudge (1924), Cummings (1940), Nesbitt (1990) and Buffo et al. (2022)

Solute	Basque Lake 1 (Livingston, 1963; after Cummings, 1940) (g/L)	Basque Lake 2, site 3 (Nesbitt, 1990) (g/L)	Basque Lake 2, site 24 (Goudge, 1924) (g/L)	Basque Lake 1, site 1, brine (Buffo et al., 2022) (g/kg)	Basque Lake 2, site 2, brine (Buffo et al., 2022) (g/kg)	Basque Lake 4, site 1, brine (Buffo et al., 2022) (g/kg)
Mg	42.4	41.60	42.52	31.41	38.59	12.85
Na	13.66	38.70	30.99	11.40	9.69	9.25
K	1.57	4.97	6.40	1.15	0.63	0.44
Ca	–	0.88	n.a.	0.49	0.31	0.29
HCO ₃	3.02	2.82	2.96	n.a.	n.a.	n.a.
SO ₄	195.71	243.01	232.92	147.19	172.49	71.57
Cl	1.69	3.52	5.52	1.43	0.55	0.46
CO ₃ (g/L)	n.a.	n.a.	n.a.	1.37	0.23	0.34
pH	n.a.	8.10	n.a.	10.2	9.45	9.86

Note. Compositions are reported in the units defined within the first row for each column, except in the case of CO₃ which is exclusively reported as grams per liter across all columns. Compositions acquired by Buffo et al. (2022) were obtained in February of 2019 and 2020, and represent the endmember lake compositions while ice-covered. Symbology: “–” indicates below detection limit; “n.a.” means that the ion was not reported.

remains enigmatic. Density-driven feedback between salt formation and sinking, such that muds are pushed to the perimeters of the pools as the heavier salts sink, is plausible. Other proposed formation mechanisms include dendritic, upward growth of pools from the base of the lake (Jenkins, 1918) or a shallow-subsurface freeze-thaw process similar to that of patterned ground formation (Renaut & Long, 1989). The number of lakes with spotted

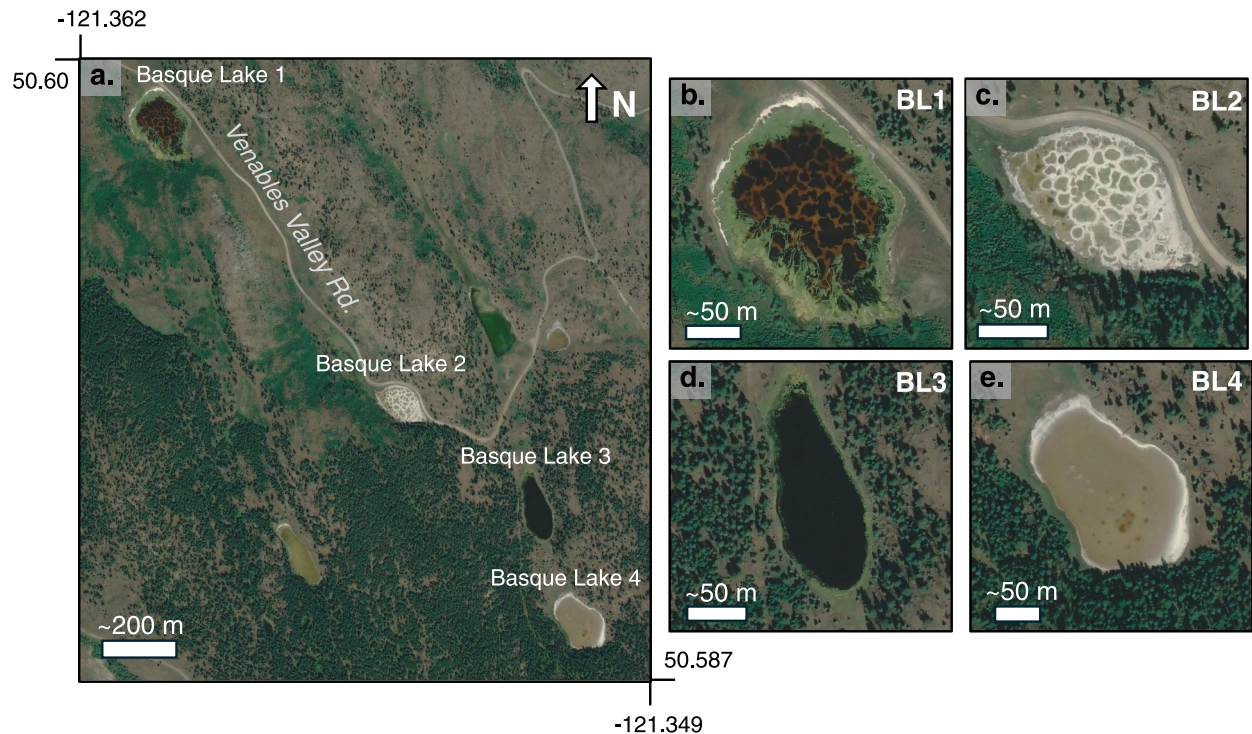


Figure 3. (a) Locations of Basque Lakes via orbital visible imagery. Image acquired in August 2018. (b) Basque Lake 1, demonstrating lake-fill with irregular internal structures. (c) Basque Lake 2, demonstrating semi-circular ridges topped with salt crusts, which surround pools of salts. (d) Basque Lake 3, entirely filled with brine, and no observable structures. (e) Basque Lake 4, with irregular internal salt pools identifiable by the darker spots. Image credit: CNES/Airbus/Maxar.

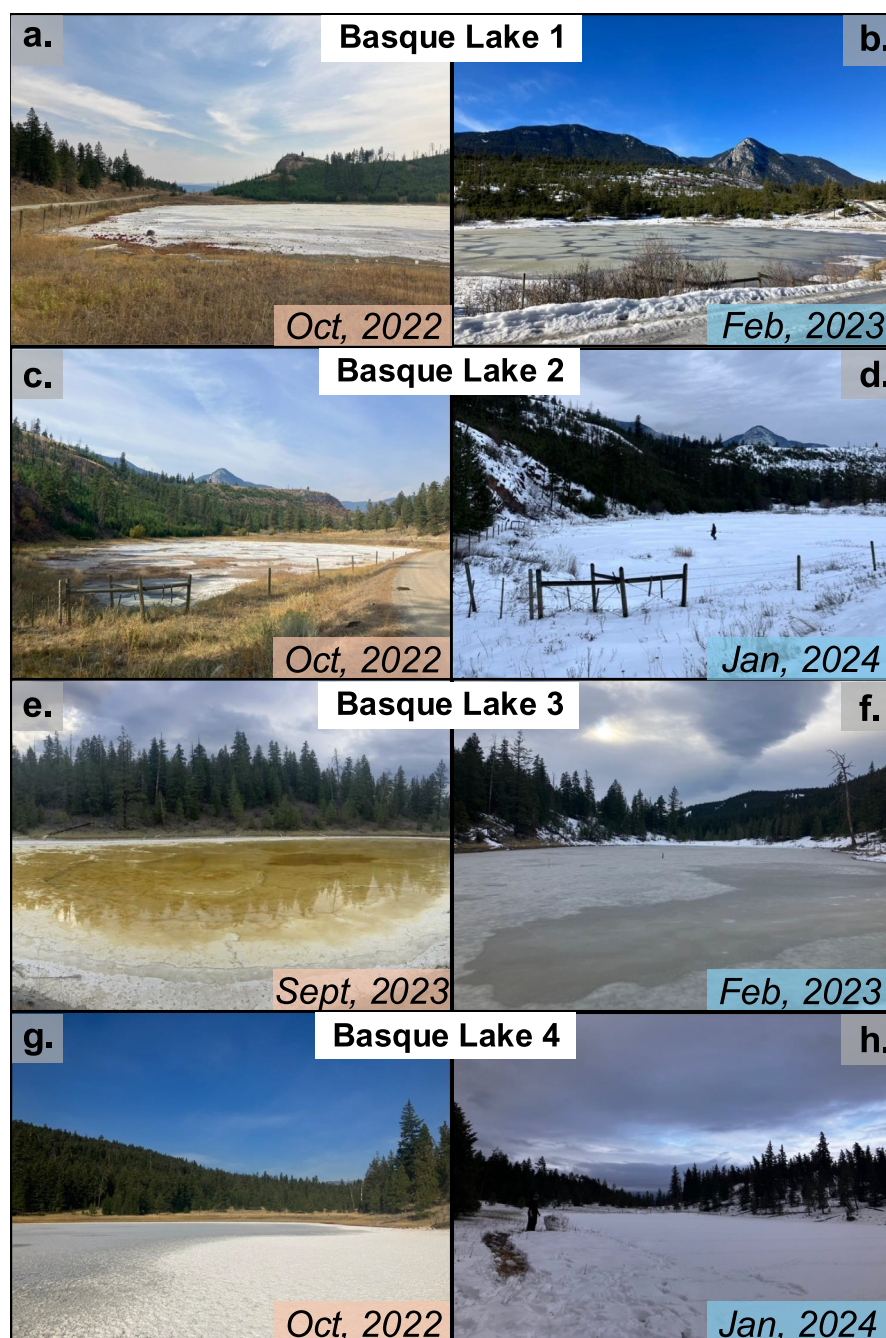


Figure 4. Basque Lakes 1–4 in the fall and winter field seasons. (a) BL1 in fall. (b) BL1 in winter with minimal snow-cover. (c) BL2 in fall. (d) BL2 in winter with snow cover. (e) BL3 in fall, demonstrating raised salt ridges. (f) BL3 in winter with complete ice cover. (g) BL4 in fall. (h) BL4 in winter with snow cover.

internal features in the high desert of British Columbia despite compositional differences suggests regional environmental influences on the geomorphology of the lakes.

The pool interiors are typically filled with crystalline salts, often under a layer of thin brine. In the summertime and fall, the ridges are muddy and have buried salt crystals. Ridges in the Basque Lakes also frequently have thick crusts of botryoidal salts (Figure 4). Occasional salts with clearly crystalline habits can be identified embedded within the ridges, though most precipitates appear to take a botryoidal form. BL3 typically has a thicker brine layer than BL1, BL2, and BL4, and typically had internal ridges composed of thick Mg-sulfate crystals (as

opposed to muds—Figure 4e). Basque Lake 4 appears to be much more mud-enriched, with fewer pools of salts and no obvious raised ridges (Figures 4g and 4h).

3.2. Basque Lake Climate

Air temperature can vary substantially on a seasonal basis, reaching an average of about 15°C in the summertime and fluctuating to the mid to high 30 s (°C). In the winter, temperatures can drop to lows of −45°C during the nighttime (Buffo et al., 2022) and tend to average around −1°C to −4°C during the day. Such vast differences in temperature allow for seasonally variable brine concentration mechanisms. Through the summertime, low rainfall and higher temperatures lead to evaporation of the Basque Lake brines, intermittently producing entirely dry lake surfaces that rehydrate with higher humidities, rain, and/or cooling. Lakes are typically at their driest in the fall due to the ongoing evaporation over the summer. Meanwhile in the wintertime, brine temperatures can drop below the eutectic temperature for Mg-sulfate brines (around −3.9°C, Himawan et al., 2006) allowing for precipitation of cryogenic hydrated Mg-sulfate. Brine temperatures tend to be much more stable than air temperatures in the winter due to ice insulation; between 2019 and 2020, logger data showed that brines in Basque Lake 2 reached a low temperature of around −6°C in December and then stabilized between −1°C and −3°C in the following winter months, before beginning to warm up in the spring (Buffo et al., 2022). Winter brines therefore persistently stay below the maximum stable temperature for meridianiite ($\text{MgSO}_4 \cdot 11\text{H}_2\text{O}$) of 2°C (Peterson et al., 2007), while surface salts experience more fluctuation in temperature. A layer of ice covers the brine pools during the winter, intermittently thickening and thinning depending on temperature and precipitation (Buffo et al., 2022), thereby cryoconcentrating the sub-surface brine. These ice layers can contain trapped salts, and may have small brine channels/conduits (Buffo et al., 2022).

4. Methods

4.1. Field Methods

We visited Basque Lakes 1–4 during six field expeditions: 4–10 October 2022; 22–27 September 2023; 12–17 September 2024; 4–8 February 2023; 26–29 January 2024; and 7–10 January 2025. These six field seasons were intended to target the lakes at their most evaporated in the fall, and at their highest volume of ice cover in the winter. We selected samples for characterization and collection that best represent the variable surface materials at each lake, and span the edge to the center of each of the lakes. We defined three broad categories of surfaces materials based on in situ observations, which we describe as “pools,” “crusts” and “muds” (Figure 5). Pools are defined as the higher density and lower topography regions of the lakes that precipitate salts through the evaporation or cryoconcentration of standing brines (Figures 5a and 5b). Pools typically contain dense crystalline salts at their base (or surface, when the pools are evaporated to completion). Crusts are defined as efflorescent, raised, often “popcorn-textured” salts that typically form on the edges of the lakes, on the ridges surrounding the pools, or on materials like logs that may be present at lake edges (Figures 5c and 5d). These crusts form in contact with the air (rather than at the base of a precipitating brine), through upward wicking of brine. Muds are defined as fine (<63 μm) sediment that may have intermixed salts and other organics, often darker in appearance, and are frequently ridge-forming (Figures 5e and 5f). Unlike crusts, muds are not primarily composed of salts, though muds do frequently underlie crusts, indicating that brine wicks through the muds as well as the crusts and interstitial salts may have been deposited. For all sampled locations, geospatial coordinates were obtained using either a Bad Elf Flex GPS unit (Bad Elf, West Hartford, CT, 06107) with a horizontal accuracy down to 0.3 m and a maximum vertical accuracy of 0.6 m, or a Emlid Reach RS2+ Real-Time Kinematic unit (Emlid, Budapest, Hungary) with ~ 2 cm accuracy. We characterized relative humidity (RH) and temperature using a Protomex (Protech international Group, Guangdong, China) PT6508 digital temperature and humidity meter. Water activity measurements were made for a brine extracted from Basque Lake 4 in January 2025, using an AquaLab (Addium, Pullman, Washington) 4TE Chilled Dewpoint Water Activity Meter. Measurements were made at 15°C and 25°C, though when sampled, the brine was near freezing, such that the water activity range we indicate represents the lowest bound for possible water activity for the samples.

In some cases, we were unable to obtain coupled geochemical and mineralogical in situ analyses at all regions of interest for each lake across seasons (Figure 6). For Basque Lake 4, for example, we did not obtain any coupled geochemical and mineralogical in situ data in the fall, though we did obtain spectral data uncoupled to geochemical data. Basque Lake 3 has only a single set of measurements in the wintertime with coupled

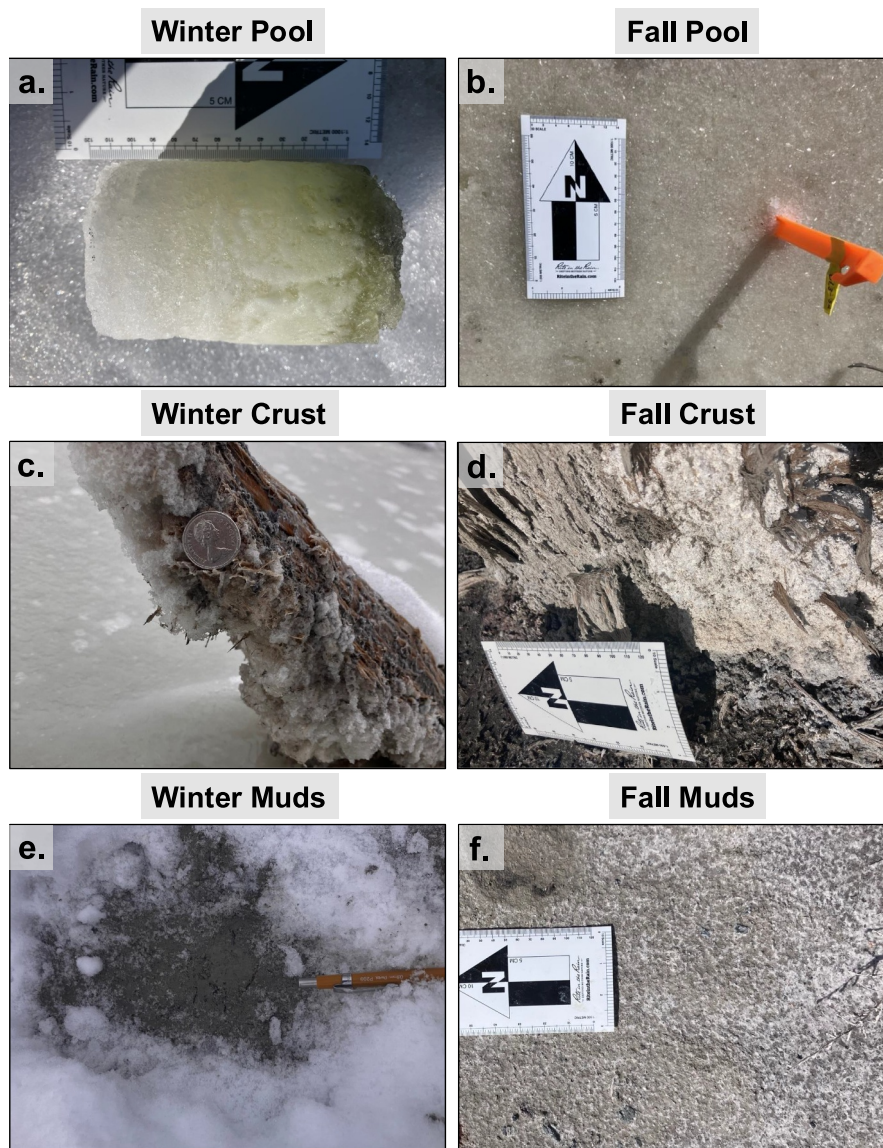


Figure 5. (a) A salt core extracted from a BL2 pool. (b) Salt crystals from a BL3 salt pool (c) Winter crusts from the edge of the BL4 lake (d) Fall crusts from BL2 (e) Winter muds from BL2 (f) Fall muds from BL2.

geochemical and mineralogical data. Both occasions were due to access issues: Basque Lake 4 has high amounts of mud and was difficult to traverse in the non-winter season, while Basque Lake 3 has a weak ice-cover in the wintertime such that traversing it would have been precarious.

We primarily focused our analyses on in situ analytical techniques, given these salts change rapidly in changing environmental conditions (temperature and humidity), such that returning samples to the laboratory can result in changed mineralogy. We relied on three in situ analytical techniques: XRF spectroscopy for elemental data and Raman and NIR spectroscopy for mineralogical data.

4.2. XRF Calibration and Methods

Elemental composition in weight percent (wt.%) was obtained for geologic samples using a SciAps X-250 model XRF field spectrometer (SciAps, Andover, Massachusetts, 01810). The X-250 obtains quantitative data for 12 elements (Al, Ca, Cr, Fe, K, Mg, Mn, Ni, P, S, Si, Ti) using a 6–50 kV and 200 μ A rhodium anode X-ray tube for the X-ray excitation source, and a 20 mm silicon drift detector. The spot size is 4 mm. We used 30 and/or 60 s

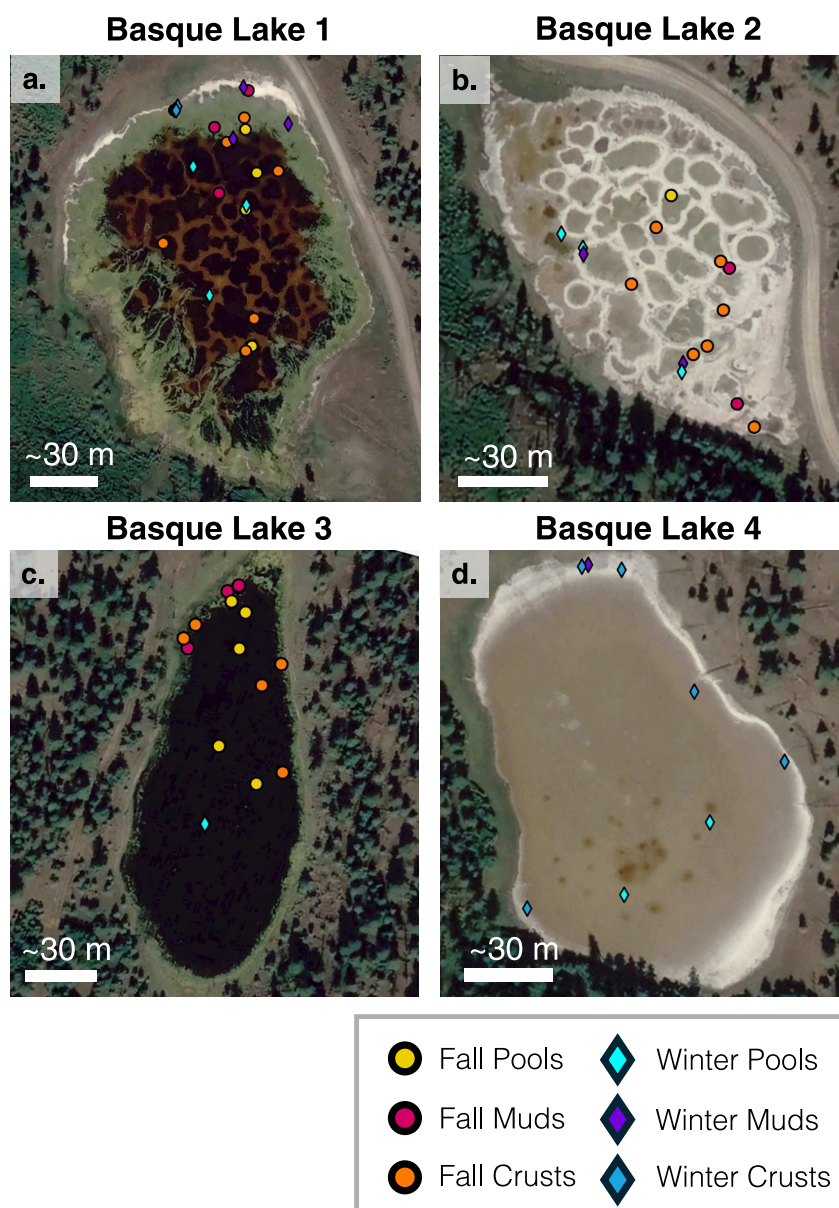


Figure 6. (a) Locations of in situ analyses in fall (circles) and winter (diamonds) for BL1. (b) In situ analytical locales for BL2. (c) In situ analytical locales for BL3. (d) In situ analytical locales for BL4.

integration times for each target, which provide a robust analytical result. In the field, we obtain XRF data using the “mining mode” calibration model, which is suited for industrial mining materials such as metals but requires an additional calibration for heterogeneous mineral mixtures. We developed an additional derivative calibration model to further refine the results obtained in mining mode, using a set of known standards with a range of values for all elements of interest. We also included salt standards mixed with varying percentages of basalt. These materials are provided as powders, which we press into pellets before acquiring the mining mode geochemical result using a 60 s integration time, with three scans per standard sample. We then derive a regression relationship between the oxide abundance obtained in mining mode and the standard composition, which we analyze using the SciAps XRF analytical software ProfileBuilder. The slope of the line for each element constitutes the correction we apply to the data acquired in mining mode. We provide the standards and their chemistry, as well as the derived calibration coefficient, in the data repository for this work (Hughes et al., 2025).

All elements lighter than magnesium are not quantifiable using the X-250 XRF. These elements (which include Na, an element of interest for this work, as well as H, C, and O) are included in a catch-all variable that we refer to as the “Light Elements” variable. This variable encompasses higher contributions from these elements, and frequently means overall higher hydration.

4.3. In Situ Mineralogical Methods

Near Infrared (NIR) spectral data were obtained using a PSR-3500 field portable spectroradiometer from Spectral Evolution (Haverhill, Massachusetts, 01835 USA). The PSR-3500 integrates over 0.35–2.5 μm and converts radiance spectra to reflectance spectra, with a spectral resolution of $\sim 0.005 \mu\text{m}$. The bare optical fiber field of view is 25° and the contact probe has a typical spot size of 10 mm. We rely on a contact probe for protection of the optical fiber and an internal light source from the spectrometer, to mitigate for effects of changing ambient light in the field. We obtained a “reference” spectrum of a portable 99% Spectralon panel before each in situ spectrum, and we obtained three in situ spectra per target. VNIR spectra were compared to reference spectra, mostly from the United States Geologic Survey mineral spectral library (Kokaly et al., 2017). The spectrum for hexahydrite ($\text{MgSO}_4 \cdot 6\text{H}_2\text{O}$) was obtained from Crowley (1991); the spectrum for meridianiite ($\text{MgSO}_4 \cdot 11\text{H}_2\text{O}$) was obtained via a personal communication with Dr. Edward Cloutis; the konyaite ($\text{Na}_2\text{Mg}(\text{SO}_4)_2 \cdot 5\text{H}_2\text{O}$) spectrum is from Shi et al. (2024); the spectra for schoenite ($\text{MgK}_2(\text{SO}_4)_2 \cdot 6\text{H}_2\text{O}$) and polyhalite ($\text{K}_2\text{Ca}_2\text{Mg}(\text{SO}_4)_4 \cdot 2\text{H}_2\text{O}$) were collected from mineral samples in the Joe Webb Peoples Museum of Wesleyan University using the spectrometer set-up described by Hughes et al. (2023). We present exclusively NIR data between 1 and 2.5 μm , because the diffuse light in an outdoor field setting led to artifacts in the visible spectral range for many targets, rendering this portion of the spectrum less useful. For interpreting saline mineralogy, the visible spectral region also contains much less information than the NIR.

We additionally conducted in situ Raman analysis using a Bruker Bravo Raman field-portable spectrometer (Bruker Scientific LLC, Billerica, MA). The Bravo Raman spectrometer covers a wavelength range of 300–3,200 cm^{-1} with a spectral resolution of 2 cm^{-1} and uses two laser sources with wavelengths of 785 and 852 nm. The Bravo spectrometer also uses sequentially shifted excitation Raman spectroscopy for fluorescence mitigation, such that the resulting spectrum contains only Raman information. The spot size for Raman analyses is $\sim 1 \text{ mm}$. The spectra were exported and analyzed using the OPUS 8.7.31 software suite. We compared Raman spectra of our materials to reference spectra in the RRUFF library (<https://rruff.info>) and to the spectrum for meridianiite provided in a personal communication with Dr. Alian Wang.

Our mineral interpretations are generally based on the $\text{SO}_4 \nu_1$ peak position, as our Raman spectra do not carry information about hydration state, due to the laser excitation wavelengths of the instrument. For Mg-sulfates, the $\text{SO}_4 \nu_1$ typically shifts from higher to lower wavenumbers as the hydration state of the Mg-sulfate increases (e.g., Wang et al., 2006, 2011). The position of the peak is moderated by the bonding of the SO_4 tetrahedra to the $\text{MgO}_n(\text{OH}_2)_{6-n}$ octahedra as well as the amount of hydrogen bonding (Wang et al., 2006). As the polymerization increases (higher polymerization occurs with lower Mg-sulfate hydration states), the ν_1 peak shifts to higher wavenumbers. However, in the case of Mg-sulfates with little to no polymerization and more free water, including hexahydrite and epsomite, this free water theoretically should not affect the position of the $\text{SO}_4 \nu_1$ peak, and therefore hexahydrite and epsomite both have $\text{SO}_4 \nu_1$ peaks around 984 cm^{-1} . We therefore take care not to distinguish between epsomite and hexahydrite Raman spectra. Meridianiite has a peak at 990 cm^{-1} and is therefore distinct from the lower hydrate Mg-sulfates (Hamilton & Menzies, 2010), though Genceli et al. (2009) report a mirabilite peak at 990 cm^{-1} as well, making distinction between meridianiite and mirabilite via Raman difficult. Our mirabilite reference spectrum, collected by Hughes et al. (2023) has a different major peak (a 1,004 cm^{-1} major peak and a shoulder around 990 cm^{-1}), which we include as a point of reference, but we assume the spectrum provided by Genceli et al. (2009) is accurate and do not attempt to distinguish between mirabilite and meridianiite based on the 990 cm^{-1} peak. A Na-sulfate heptahydrate was identified by Hamilton and Menzies (2010) as well, with a major peak at 987.6 cm^{-1} . We do not include a reference spectrum for this material, but we do consider it in the context of our mineral identifications.

4.4. Geochemical Modeling

We modeled brine evolution using the low temperature aqueous geochemical modeling program FREZCHEM (Version 13). FREZCHEM is a thermodynamic model relying on a Pitzer parameterization to predict activity

coefficients, osmotic coefficients, and mineral precipitation for solutions of high ionic strength. FREZCHEM has been applied in a variety of planetary and Earth settings, notably deriving a range of Mars-relevant brine compositions (Marion et al., 2010; Toner et al., 2014; Tosca et al., 2011) as well as brine evolution for outer solar system bodies like Titan, Enceladus, and Ceres (Castillo-Rogez et al., 2018; Marion et al., 2012). For freezing and evaporating models, we used fractional crystallization with a closed carbon system. We ran our evaporative models at 293.15 K (“Warm Evap 1”) and 303.15 K (“Warm Evap 2”), and our freezing model began at 273.15 K and iterated to 255.15 K in 1 kelvin temperature steps, with a starting H₂O abundance of 1,000 g in all cases. We included a partial pressure of 0.0004 bars of CO₂, and 0.21 bars of O₂. We allowed the system to proceed until it could no longer converge (i.e., convergence failure), meaning the system failed to identify the convergence criteria after 1,000 iterative attempts to solve the equilibrium system state (Marion et al., 2008; Tosca et al., 2011). This is common for FREZCHEM modeling and was previously reported on by Tosca et al. (2011). Here we report on the final values before the system no longer successfully converged, and for completeness include the remaining number of grams of water in the solution before the model run terminated. We ran the model for brine compositions reported in Table 2 and compiled from work by Goudge (1924), Cummings (1940), Nesbitt (1990) and Buffo et al. (2022). FREZCHEM requires molality (mol/kg) input parameters, while all except for Buffo et al. report their values in mmol/L, requiring a conversion from molarity or molality. We estimate brine density from an average of two modern brine samples from Basque Lake 2 acquired in the fall of 2022. We make the assumption that this density is applicable to data collected by Nesbitt, Goudge, and Cummings et al. because (a) Nesbitt (1990) note the remarkable similarity in brine composition between the data collected by Goudge (1924) and their own, despite 50 years’ difference, (b) the compositions reported by Goudge (1924), Cummings (1940), and Nesbitt (1990) are indeed similar, and (c) all data sets appear to have been acquired in the summer or early fall, aligning with the collection time for our data. The density value we used was 1.22 kg/L to convert molarity to molality, for which we subtracted the mass fraction of ions to ensure only the solvent (water) is included. In all cases for which pH is not reported, we specify an initial pH of 7. We convert the provided data to mol/kg and in some cases the data were not charge balanced upon conversion. As FREZCHEM relies on a charge balanced solution to properly run, we added the appropriate amount of either the Mg cation or the SO₄ anion to properly charge the solution, under the assumption that these two ions have the highest concentration and therefore charge adjustments for these ions will result in the least deviation from real-world values.

4.5. Spectral Band Analyses

Spectral band analysis and “band math” are techniques commonly employed for differentiation of hydrated phases and mineral mapping, particularly with data from Mars such as Compact Reconnaissance Imaging Spectrometer for Mars (CRISM) instrument data, or Earth observing spectrometers with multispectral resolution, such as Landsat data (e.g., Radwin & Bowen, 2021; Viviano et al., 2014). Here we apply a similar technique to data acquired using our NIR spectrometer. We focus our efforts on the wavelength region between 1.53 and 1.86 μm , as it can be used for differentiating the materials of interest in this study (e.g., Cloutis et al., 2006; Crowley, 1991; Shi et al., 2024). All major minerals of interest for this study have features in this spectral window, due to the S–O bond, OH combination or overtones, and H₂O combination or overtones (Figure 7; Cloutis et al., 2006). We begin by removing all spectra whose reflectance are too low (below 4% reflectance near the 1.4 μm feature and the 1.9 μm feature) from our analyses, and visually verifying all remaining spectra have features within this region, to avoid interpreting spectra saturated by water. We then truncate all field and reference mineral spectral data to the window of 1.53 and 1.86 μm to capture absorption features between shoulders (Figure 7). We then perform a continuum remove function available in the ENVI software suite, which relies on the Segmented Upper Hull technique (R. N. Clark & King, 1987). We then determine the wavelength position of the band minimum in this region. We also develop a “band parameter” for minerals of interest in this work, intended to highlight the similarity between reference spectra and field spectra concerning the structure of absorption features in this window.

Band parameters are suited to evaluate mineral presence based on the relative reflectance and absorbance at points in the spectrum. Here we focus on a “bloedite band parameter” (BBP) as a general proxy for change in the abundance of seasonal double salts. Double salts, salts with two or more different cations or anions, for example Mg and Na, often have seasonal dependencies. Bloedite for example, has seasonal dependency (Marion & Farnen, 1999), with a stability field between $\sim 7^\circ\text{C}$ and 60°C (Warren, 2006). Bloedite and the pentahydrate Mg–Na–sulfate konyaite (stable at slightly cooler temperatures than bloedite, Onac et al., 2001) exhibit absorption features

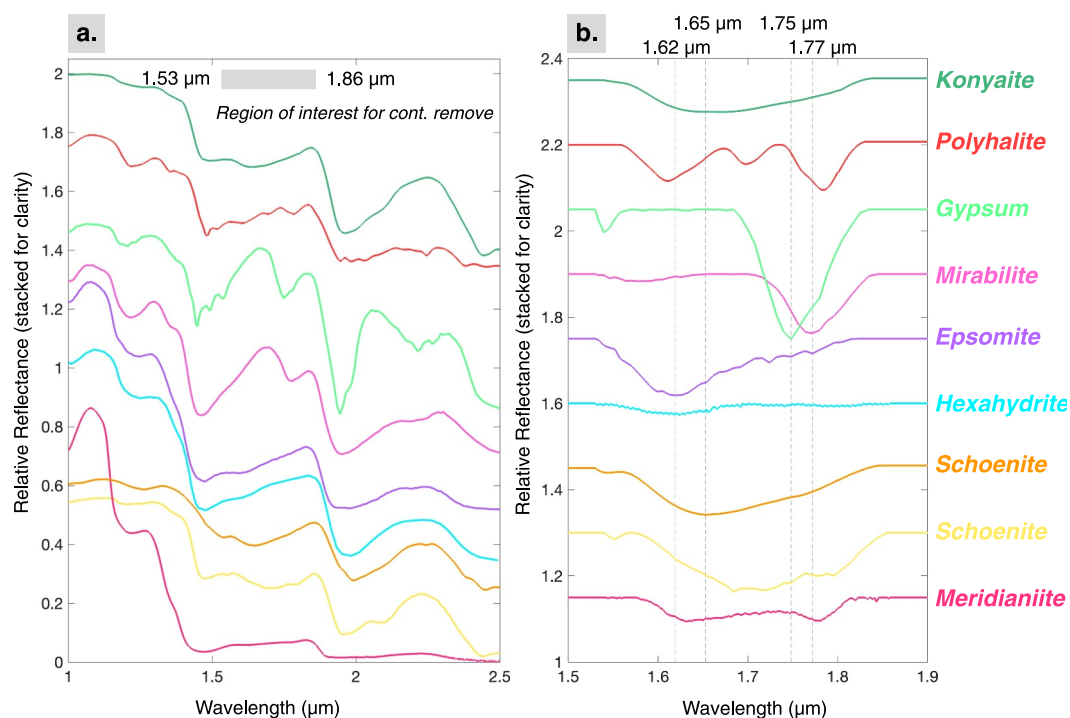


Figure 7. (a) Reference spectra of the minerals of interest for this work (see legend on the far right for disambiguation). The window used for band parameter and band minima analysis is outlined at the top of the plot. (b) Continuum-removed reference spectra within the window of interest. Wavelengths for the minima for epsomite, schoenite, gypsum and mirabilite, respectively, are labeled.

between 1.65–1.69 μm, distinct from absorption features of the mono-cation salts mirabilite and epsomite. Our band parameter is suited for evaluating the presence of absorption features around ~1.686 and ~1.714 μm (bloedite absorption features), and a lack of absorption feature around ~1.617 and ~1.771 μm, where epsomite and mirabilite have absorptions, respectively. This means our band parameter distinguishes minerals likely present during colder seasons, when bloedite lacks a stability field, from warm-temperature minerals. The BBP is:

$$\text{Band Parameter}_{\text{Bloedite}} = \frac{((1 - R_{1.686}) + (1 - R_{1.714}))}{((1 - R_{1.617}) + (1 - R_{1.771}))}$$

where R represents the reflectance of the material spectrum at that wavelength notated in the subscript, after a continuum remove function. We use this band parameter to evaluate the relative likelihood of bloedite presence by season, but note that other minerals will have characteristic values at these wavelengths, so it is possible to use this parameter to distinguish different mineral species in addition to bloedite. In other words, double salts like schoenite ($\text{MgK}_2(\text{SO}_4)_2 \cdot 6\text{H}_2\text{O}$) and konyaite have stronger BBP values than mono-cation salts, and when coupled with band minima, the BBP helpfully distinguishes these species as well.

We do not include magnesite (MgCO_3) or thenardite (Na_2SO_4) in our band minima and parameter analyses, because these salts have extremely mild to absent spectral features in this wavelength range (Chung et al., 2020; Crowley, 1991; Harner & Gilmore, 2015), so band parameter analysis would not yield meaningful results for these minerals.

5. Results

5.1. Modeling and In Situ Stability

We modeled lake compositions reported by Goudge (1924), Cummings (1940), Nesbitt (1990), and Buffo et al. (2022) until convergence failure under freezing conditions from 275.15 (0°C)–255.15 K (–20°C), and under evaporative conditions at 293.15 K (20°C) and 303.15 K (30°C). We call these models “Freezing Model,” “Warm

Evap 1” and “Warm Evap 2” respectively. The BL1-S1 brine from Buffo et al. (2022) failed to converge via evaporative models and BL2-S2 failed to converge in the freezing model after only a few iterations/steps, so very few precipitates were modeled. We therefore do not report the results of these simulations.

Results represent a range of possible salt mineral assemblages forming at the Basque Lakes under variable conditions, but do not represent complete evaporative assemblage, as some water remained in the simulation for each model run (Table 3). All evaporative simulations predicted in the precipitation of epsomite ($\text{MgSO}_4 \cdot 7\text{H}_2\text{O}$), and most predicted bloedite (Table 3). Mirabilite ($\text{Na}_2\text{SO}_4 \cdot 10\text{H}_2\text{O}$) is predicted in many cases in the Warm Evap 1 evaporative simulation but is entirely absent in the Warm Evap 2 model; Na is primarily in the bloedite phase under warmer temperatures, in addition to minor thenardite (Na_2SO_4) presence. Minor amounts of schoenite, magnesite, and gypsum ($\text{CaSO}_4 \cdot 2\text{H}_2\text{O}$) are frequently predicted to form from evaporative models, though not consistently across all starting brine compositions. Dissolved aluminum values were reported by Nesbitt (1990), and minor gibbsite ($\text{Al}(\text{OH})_3$) and alunite ($\text{KAl}_3(\text{SO}_4)_2(\text{OH})_6$) are predicted for this brine composition.

In a freezing simulation, the model predicts meridianiite ($\text{MgSO}_4 \cdot 11\text{H}_2\text{O}$) and mirabilite across all brine compositions included in this study. Minor amounts of schoenite, gypsum and magnesite are also predicted to form under freezing conditions. Bloedite is not predicted during the freezing simulations. Minor epsomite precipitation was predicted for Goudge (1924) and Nesbitt (1990) brines, forming early in the freezing run before meridianiite became the stable Mg-sulfate.

Thermodynamic modeling considers the effect of temperature on mineral stability, but RH also plays a role in mineral stability. We obtained in situ RH and temperature measurements at the Basque Lakes, which we compare to Mg-sulfate, Na-sulfate and Ca-sulfate stability data (Figure 8). Our meridianiite stability field is from Chou et al. (2013), as is the gypsum-anhydrite (CaSO_4) stability field. The remaining stability fields for the Mg-sulfate system are from Chipera et al. (2023), and the mirabilite-thenardite stability field is from Herrero et al. (2015). RH and temperature measurements are primarily relevant to materials in direct contact with the air (muds and crusts) rather than submerged salts. Likewise, as daily temperature/RH values vary, individual RH/temperature measurements are less relevant than the span of RH/temperature values for each season. We observe no overlap in RH/temperature values between fall and winter data, and we observe that mineralogy accordingly should vary (Figure 8). RH and air temperature data from the winter suggest the stable Na-sulfate and Mg-sulfate hydrates are mirabilite and epsomite (Figure 8). The thermodynamic model primarily predicts meridianiite from freezing, but epsomite is also stable at low temperatures (particularly with low relative humidities) and so may form under cold conditions. In situ environmental surface data suggests epsomite should broadly be present in the winter for salts in contact with the air. Meanwhile, within the brines insulated by ice, meridianiite may be the stable phase: water activity (related to RH in that water activity is simply the $\text{RH}_l/100$ of a liquid phase) appears to be very high from January 2025 measurements, and temperatures are below freezing for BL4 brines measured in February 2019 by Buffo et al. (2022). From these measurements, we provide a general Basque Lake brine temperature/activity window. In the winter Basque Lake brines, meridianiite is the stable Mg-sulfate hydrate, mirabilite would remain the stable Na-sulfate, and gypsum is the stable Ca-sulfate. For salts in contact with air, epsomite is the stable Mg-sulfate. Meanwhile in the summer, air temperature rises and RH drops, such that hexahydrate ($\text{MgSO}_4 \cdot 6\text{H}_2\text{O}$) and starkeyite ($\text{MgSO}_4 \cdot 4\text{H}_2\text{O}$) are predicted to be stable on the surface. Thenardite is expected to be the stable Na-sulfate phase, though we do not plot the stability field for bloedite which is also favored in warmer temperatures, and so Na-sulfates might be present in the fall in either phase. Anhydrite should be the stable Ca-sulfate.

Cold-temperature meridianiite and mirabilite are predicted to form via FREZCHEM, consistent with the brine temperature and water activity measurements also suggesting that meridianiite and mirabilite should be the stable hydrates (Table 4). While epsomite is minimally predicted from freezing modeling, it's possible epsomite would form on the surface from cold-temperature evaporation. Our Warm Evap 1 and Warm Evap 2 models predict epsomite to form, which is inconsistent with the climatological data indicating the less hydrated Mg-sulfates hexahydrate and starkeyite should be stable at the surface (Table 4). We explore such discrepancies in relation to observed data in the results and discussion section of this work.

5.2. Geochemical XRF Analyses

Geochemical analyses from the Basque Lakes in the winter and fall seasons reveal that most data from the Basque Lakes plot along a Mg-sulfate mixing trendline (Figure 9a). XRF-derived abundances of Mg and S are higher on average for data acquired in the fall than data acquired in the winter, but almost all data plot on the Mg-sulfate

Table 3
Results From FREZCHEM Modeling of Basque Lake Brine Chemistries

Evaporation simulation at 293 K (Warm Evap 1)					
Salt	BL2-S2-brine (Buffo et al., 2022)	Buffo-BL4-S1Br (Buffo et al., 2022)	BL1 brine (Cummings, 1940)	BL2 site 24 (Goudge, 1924)	BL2 site 3 (Nesbitt, 1990)
Halite	–	–	0.03	0.67	–
Sylvite	–	–	–	0.12	–
Gypsum	0.32	9.36	–	–	0.74
Thenardite	–	–	–	–	0.85
Schoenite	–	–	1.37	5.80	3.92
Bloedite	0.41	–	7.54	40.91	40.97
Epsomite	86.76	87.16	79.13	50.24	47.51
Mirabilite	12.06	–	11.52	1.83	4.76
Magnesite	0.45	3.48	0.42	0.43	–
Gibbsite	–	–	–	–	0.46
Alunite	–	–	–	–	0.78
H ₂ O remaining (g)	31.77	194.51	7.61	13.80	33.74
Evaporation simulation at 303 K (Warm Evap 2)					
Salt	BL2-S2-brine (Buffo et al., 2022)	Buffo-BL4-S1Br (Buffo et al., 2022)	BL1 brine (Cummings, 1940)	BL2 site 24 (Goudge, 1924)	BL2 site 3 (Nesbitt, 1990)
Halite	–	–	–	0.06	–
Gypsum	0.35	1.17	–	–	0.84
Schoenite	0.03	–	1.35	5.86	3.14
Bloedite	16.49	46.73	21.38	45.56	52.47
Epsomite	82.65	51.68	76.80	48.07	41.75
Magnesite	0.48	0.42	0.46	0.44	0.68
Gibbsite	–	–	–	–	0.88
Alunite	–	–	–	–	0.25
H ₂ O remaining (g)	18.82	63.14	12.97	27.38	74.47
Freezing simulation					
Salt	BL1-S1-brine (Buffo et al., 2022)	Buffo-BL4-S1Br (Buffo et al., 2022)	BL1 brine (Cummings, 1940)	BL2 site 24 (Goudge, 1924)	BL2 site 3 (Nesbitt, 1990)
Gypsum	0.42	0.52	–	–	0.47
Schoenite	0.81	–	0.90	3.37	2.34
Epsomite	–	–	–	0.90	3.30
Meridianiite	82.21	69.01	84.26	67.42	62.04
Mirabilite	16.17	30.24	14.52	28.03	31.03
Magnesite	0.39	0.23	0.32	0.28	–
Gibbsite	–	–	–	–	0.30
Alunite	–	–	–	–	0.52
H ₂ O remaining (g)	10.08	87.22	12.40	42.91	36.82

Note. Results are reported in wt.% assuming the total precipitate abundance sums to 100 wt.%. We additionally report remaining H₂O in the solution assuming a starting abundance of 1,000 g.

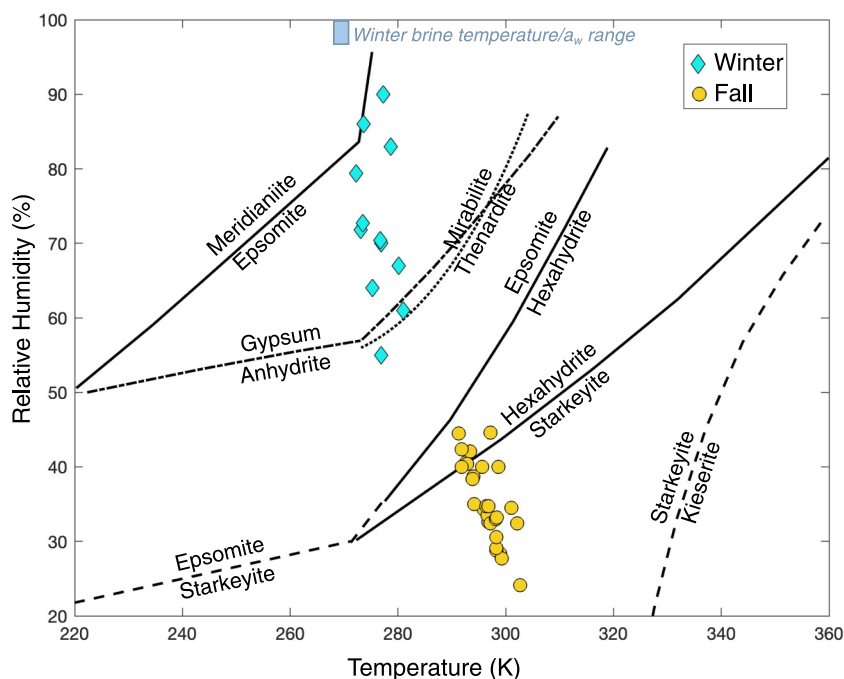


Figure 8. In situ relative humidity data acquired at the Basque Lakes acquired in the winter and fall seasons, compared to Mg-sulfate, Na-sulfate and Ca-sulfate stability curves adapted from Chipera et al. (2023), Chou et al. (2013) and Herrero et al. (2015). The blue box represents the range of measured water activities (reported in this study) and temperatures (reported by Buffo et al. (2022)) for BL4 brines.

mixing line in both seasons (Figure 9a). Most data acquired in the winter are lower on the Mg-sulfate mixing line, consistent with a higher proportional “Light Element” elemental contribution. This contribution is likely a combination of mineralogically structural water and interstitial ice. Muds have the lowest proportional Mg and S in the winter data, and indeed muds frequently exhibit interstitial ice crystals in the winter, which is likely responsible for the lower values. The winter pools show the highest proportional Mg and S of winter data, suggesting either proportionally lower ice or structural H₂O contributions.

Data from the fall on Figure 9 plot with proportionally higher Mg and S, suggesting the presence of lower hydration state Mg-sulfates in the fall, such as epsomite, hexahydrite, and starkeyite. As in the winter, data from fall pools plot with the highest amount of Mg and S relative to other fall data, indicating stronger contributions from relatively pure Mg-sulfates. Many data points plot with more Mg contribution than the Mg-sulfate mixing line, indicating a larger complement of Mg than is characteristic for pure Mg-sulfates. The associated anion for this excess Mg is unclear. Because C is not characterized by the XRF, it is difficult to know whether magnesite or another Mg-bearing salt might be responsible.

Meanwhile, the Ca-sulfate mixing line demonstrates likely contributions from a Ca-sulfate in the fall crust data for some data points, and some mixed Ca-bearing materials with excess sulfur, such that the latter points do not plot along the Ca-sulfate mixing line (Figure 9b). Data from fall and winter pools show almost no contribution from Ca-sulfate. Data from the winter crusts likewise show minimal contributions from Ca-sulfates, while winter muds do show some minor evidence for Ca-sulfates at the low end of the Ca-sulfate mixing line (Figure 9b). Trends based on lake (Figures S1 and S2 in Supporting Information S1) indicate higher contributions of Ca in Basque Lakes 1 and 2 than in Basque Lake 3. We were unable to obtain XRF data from Basque Lake 4 in the fall, so cannot compare trends for this lake.

We additionally investigated the trends of other relevant oxides based on surface material and season (Figure 10). CaO demonstrates a wide range of values not clearly coupled to SiO₂ (Figure 10a). The primary minor oxide trends for fall and winter muds include variable K₂O, broadly correlated Al₂O₃ and SiO₂, and broadly correlated FeO_T and SiO₂ (Figures 10b–10d). Fall and winter muds show relatively high amounts of SiO₂. Fall crusts also exhibit broadly correlated Al₂O₃ and SiO₂, and FeO_T and SiO₂, and relatively high SiO₂ (in some cases, up to 10

Table 4
Mineral Modeling and Stability in the Fall and Winter for the Basque Lakes

Mineral name	Formula	Winter		Fall	
		Modeling	RH/Temp stability	Modeling	RH/Temp stability
Kieserite	$\text{MgSO}_4 \cdot \text{H}_2\text{O}$	–	–	–	–
Starkeyite	$\text{MgSO}_4 \cdot 4\text{H}_2\text{O}$	–	–	–	Yes
Pentahydrate	$\text{MgSO}_4 \cdot 5\text{H}_2\text{O}$	–	–	–	n.a.
Hexahydrate	$\text{MgSO}_4 \cdot 6\text{H}_2\text{O}$	–	–	–	Yes
Epsomite	$\text{MgSO}_4 \cdot 7\text{H}_2\text{O}$	Yes	Yes	Yes	–
Meridianiite	$\text{MgSO}_4 \cdot 11\text{H}_2\text{O}$	Yes	Yes	–	–
Bloedite	$\text{Mg}(\text{Na}_2)(\text{SO}_4)_2 \cdot 4\text{H}_2\text{O}$	–	n.a.	Yes	n.a.
Schoenite (Picromerite)	$\text{Mg}(\text{K}_2)(\text{SO}_4)_2 \cdot 6\text{H}_2\text{O}$	Yes	n.a.	Yes	n.a.
Thenardite	Na_2SO_4	–	Yes	Yes	Yes
Mirabilite	$\text{Na}_2\text{SO}_4 \cdot 10\text{H}_2\text{O}$	Yes	Yes	Yes	–
Gypsum	$\text{CaSO}_4 \cdot 2\text{H}_2\text{O}$	Yes	Yes	Yes	–
Anhydrite	CaSO_4	–	Yes	–	Yes
Halite	NaCl	–	n.a.	Yes	n.a.
Sylvite	KCl	–	n.a.	Yes	n.a.
Magnesite	MgCO_3	Yes	n.a.	Yes	n.a.
Alunite	$\text{KAl}_3(\text{SO}_4)_2(\text{OH})_6$	Yes	n.a.	Yes	n.a.
Gibbsite	$\text{Al}(\text{OH})_3$	Yes	n.a.	Yes	n.a.

Note. “Yes” means the mineral was either modeled or was predicted to be stable. “–” means the mineral was not modeled or is not expected to be stable. “n.a.” means stability data for this mineral was not included or was not available.

wt.%) while winter crusts do not show strong correlations or enrichments of these materials. Winter crusts, instead, show relatively high Al_2O_3 contributions, uncoupled to anions characterized using the XRF technique (Figure 10b).

The pools also exhibit trends around K_2O , Al_2O_3 and SiO_2 based on season, observable in both the oxide bivariate plots and ternary analysis. In the fall, there appears to be a contribution from K_2O to the pools (up to ~4 wt.%) that is higher than contributions of K_2O to the pools in the winter (Figure 10c). This K_2O enrichment in fall pools is coupled with minor Al_2O_3 and SiO_2 contributions. The relative proportion of K_2O , while lower in the wintertime,

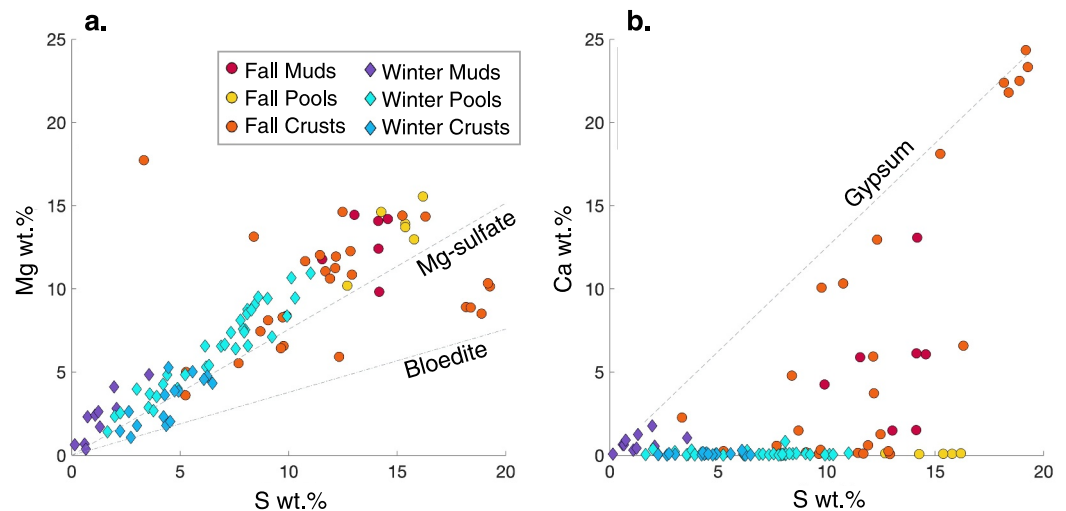


Figure 9. (a) Mg versus S data for fall (circles) and winter (diamonds). (b) Ca versus S for fall (circles) and winter (diamonds).

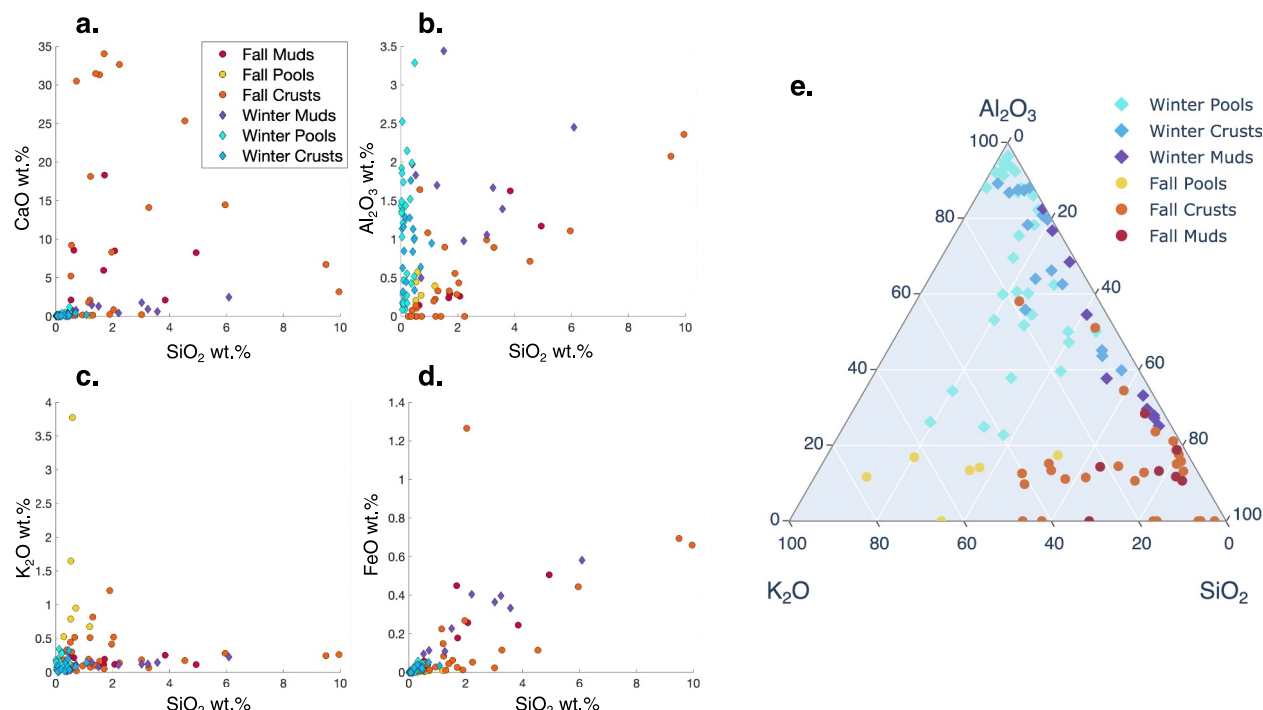


Figure 10. (a–d) Oxide bivariate plots for materials of interest across all lakes, sorted by material type. (e) Al₂O₃-K₂O-SiO₂ ternary analysis of material types. Ternary plotting software available via (Essenfeld & Morris, 2025).

is still higher in the pools than other types of materials based on our ternary analyses (Figure 10e). This suggests some K₂O contribution to the pools in both seasons.

Meanwhile winter pool data also exhibit contributions from Al₂O₃ that are relatively high and appear to be uncoupled from SiO₂. Indeed, winter pool data show notable Al₂O₃ contributions (up to ~3.5 wt.%) without obvious associated anions (Figure 10b). In contrast, data from the fall pools show much lower Al₂O₃ contributions both in terms of bulk weight percent (Figure 10b) and in terms of proportional contribution from ternary analysis (Figure 10e).

Both winter and fall data show lower SiO₂ in the pools than in the crusts and the muds. Generally, SiO₂ appears to be lower in both weight percent and relative abundance in the winter data across all kinds of surface materials; only muds show some contribution from SiO₂.

5.3. NIR Spectral Analyses

NIR spectral data reveal a diversity of sulfate-bearing materials present across lakes in the winter and fall seasons. Here we focus on select examples of spectra from pools, muds, and crusts across the Basque Lakes, as exemplars of mineral diversity based on season and surface features. We then present results from band parameter and band minima analyses to evaluate general trends in mineralogy, using techniques applicable to data from Mars orbiters and rovers. All NIR spectra and disambiguation of plot nomenclature are available in our data repository (Hughes et al., 2025).

5.3.1. Pools

Spectral data from salt pools were obtained across all lakes. Particularly in the fall, many pools contained minor amounts of brine as either thin films or interstitially between salt crystals. When such brine was present, salt NIR signals were near-totally swamped with water, leaving little to no possibility of mineralogical interpretation. For this reason, we present only two observations from pools in our NIR plots, for which we do not provide interpretations of mineralogy. Figure 11a showcases these two spectra from BL1 and BL2 obtained in situ from pools, with minimal spectral features, consistent with brine that likely swamped the signal.

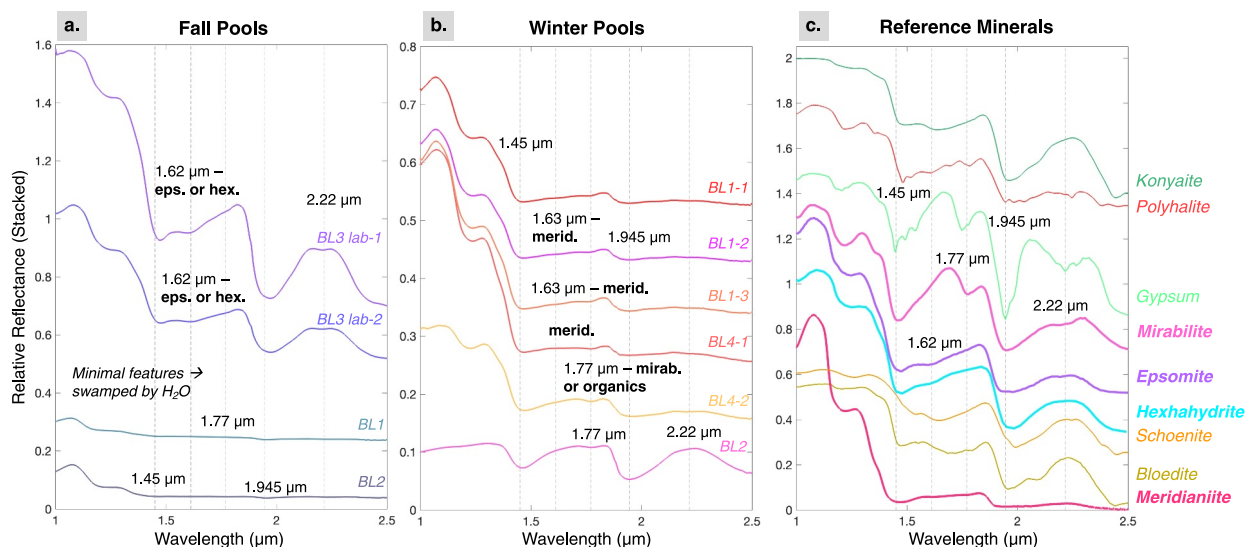


Figure 11. Spectra acquired from precipitated salts in pools in the winter and fall. Spectra are labeled according to the lake from which they were acquired, in numbered order if more than one spectrum is presented for a given lake. Names of likely minerals are identified at or near relevant absorptions. “eps.” refers to epsomite; “hex.” to hexahydrate; “merid.” to meridianiite and “mirab.” to mirabilite. Dotted lines represent general positions of major absorption features for relevant salt species and are labeled accordingly. All spectra are stacked for clarity. (a) Data acquired in situ from fall pools (BL1 and BL2) and pool samples returned to the lab and desiccated (BL3 lab-1 and BL3 lab-2). (b) Spectra of salt cores extracted from pools in the wintertime. (c) Reference spectra of relevant minerals, with the minerals likely identified in the fall and winter spectra in bold.

We returned salt samples extracted from Basque Lake 3 to the laboratory and desiccated them before obtaining additional NIR data. Such data (presented in Figure 11a) do not represent the hydration state of salts present in the brine pools in the fall, but can be used to understand the general mineralogy (e.g., Mg-sulfate rich, Na-Mg-sulfate rich, or Ca-sulfate rich). These spectra indicate, via absorptions near 1.62 and 2.21 μm , the presence of epsomite or hexahydrate. The spectrum for BL3 lab-1 has an upturn around 1.8 μm consistent with epsomite, while the spectrum of BL3 lab-2 may be consistent with epsomite or hexahydrate given a slightly less pronounced upturn feature.

We acquired winter pool spectral data from extracted salt cores, which underwent brine drainage, in turn providing a better salt NIR signal. While it remains possible some features are due to brine hydration, we expect NIR features for the spectra selected here are primarily due to the presence of salts. Spectra from BL1 (1–3) are consistent with meridianiite, with a relatively flat spectrum between 1.45 and 1.9 μm , but a small upturn/shoulder before the 1.9 μm hydration feature (Figures 11b and 11c). Minor features at ~ 1.63 and ~ 1.77 μm for BL1 (1–3) are also aligned with meridianiite. BL4-1 has a feature around ~ 1.77 μm that is consistent with meridianiite, but no observable feature at ~ 1.63 μm ; however the overall shape of this spectrum does suggest meridianiite presence. BL4-2 shows red-sloping (higher reflectance at longer wavelengths) from 1.45 to 1.77 μm , as well as some structure around 2.22 μm , which is more consistent with mirabilite. The spectrum from BL2 appears similar to mirabilite, but this spectrum was acquired from a salt core also rich in interstitial muds. The C-H stretch common to organic material and plant matter has an overtone absorption around ~ 1.7 μm (e.g., R. N. Clark et al., 2009), so we cannot definitively assign mirabilite as the material present.

5.3.2. Crusts

Spectral data from crusts indicate a diversity of minerals observable with NIR spectroscopy in the winter and fall (Figure 12). Type spectra of crusts in the fall indicate the presence of epsomite as well as double salts including schoenite, konyaite, and/or bloedite (Figure 12b). The spectrum from BL3-1 is consistent with epsomite, based on the structure, ~ 1.62 μm feature, and position of the hydration feature. A feature at ~ 1.77 μm may be consistent with minor mirabilite contribution. The spectrum of BL3-2 has a broad absorption with a minimum around 1.65 μm , which is consistent with schoenite. BL1-1 is consistent with epsomite or hexahydrate, with a sharp ~ 1.4 μm hydration feature, an absorption feature near 1.62 μm , and a minor feature near 2.22 μm . BL1-2 likewise is consistent with epsomite, though a minor feature at 1.77 μm might be consistent with mirabilite. The spectrum

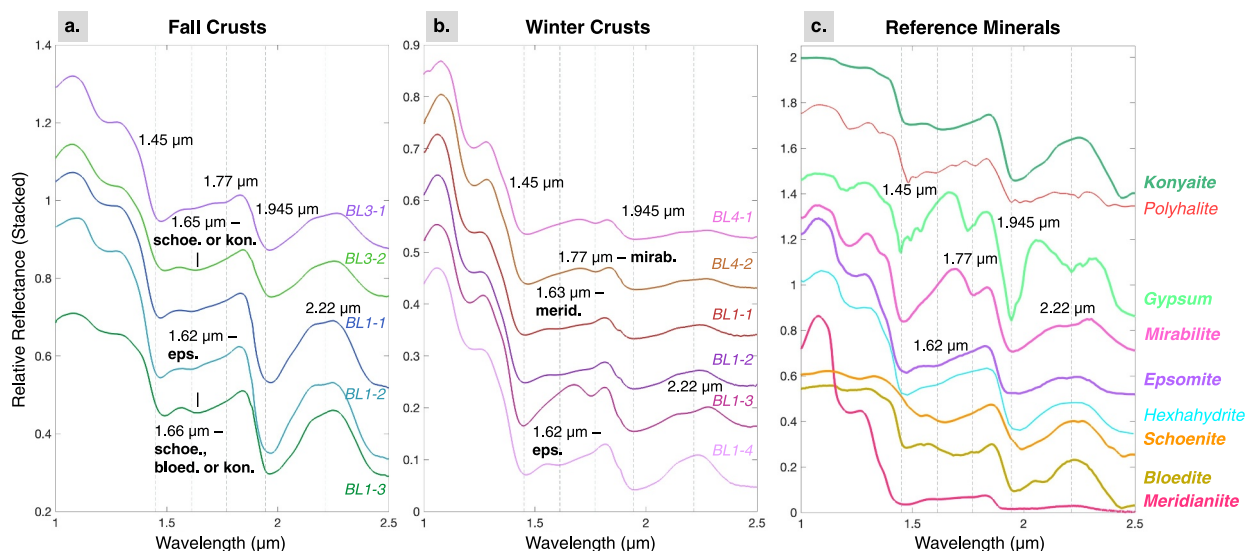


Figure 12. Spectra acquired from salts formed as crusts in the winter and fall. Spectra are labeled according to the lake from which they were acquired, in numbered order if more than one spectrum is presented for a given lake. Names of likely minerals are identified at or near relevant absorptions. “schoe.” refers to schoenite; “kon.” to konyaite; “eps.” to epsomite; “blood.” to bloedite; “merid.” to meridianiite and “mirab.” to mirabilite. Dotted lines represent general positions of major absorption features for salt species and are labeled. All spectra are stacked for clarity. (a) Spectra acquired in situ from fall crusts. (b) Spectra acquired in situ from winter crusts. (c) Reference spectra of relevant minerals, with the minerals likely identified in the fall and winter spectra in bold.

BL1-3 has a relatively broad absorption feature around 1.66 μm consistent with schoenite, konyaite, or bloedite. The structure of the ~1.4 μm water feature is more consistent with epsomite than any double salt for this spectrum. BL1-3 also lacks a strong ~2.1 μm feature, indicating it may be less likely to be bloedite than schoenite or konyaite, and may be a mixture of epsomite and schoenite or konyaite.

Spectra acquired from crusts in the winter indicate a loss of these double salts. All NIR spectra acquired from winter crusts exhibit band minima at or near 1.63 and 1.77 μm (if any) in the 1.53–1.86 μm spectral range, and while minima closer to 1.65–1.68 μm are entirely absent. BL4-1 and BL4-2 showcase a red slope feature between 1.45 and ~1.6 μm, similar to the spectrum of mirabilite (Figure 12b), coupled with absorption features near 1.77 μm. The spectra BL1-1 and BL1-2 have features around ~1.63 μm, which are similar to meridianiite; alternatively this may indicate epsomite, which has a slightly shorter-wave absorption feature at 1.62 μm. Additional absorption features at 1.77 μm for BL1-1 and BL1-2 suggest mirabilite mixing. The spectrum BL1-3 shows a similar structure to mirabilite, particularly the shape of the ~1.4 μm water absorption feature, and the ~2.2 μm feature; however, the position of the 1.7 μm absorption is closer to 1.75 μm, which is more consistent with gypsum than mirabilite. The BL1-4 spectrum is consistent with epsomite, particularly based on the feature around 1.62 μm and the upturn near 1.8 μm.

5.3.3. Muds

Muds present in the winter and fall are visually different from one another, with icier muds—lacking clearly efflorescing salts—present in the wintertime, and dryer, saltier muds, in the fall (e.g., Figures 5e and 5f). Fall data indicate that such salts might be a combination of Mg- and Ca-sulfates (Figure 13). BL3-1 has few spectral features that can be clearly linked to salts, but an absorption minimum at 1.67 μm may be consistent with either konyaite or bloedite (Figure 13a). The spectrum from BL1 is likewise difficult to assign clear mineralogy, but an absorption feature around 2.22 μm may be from gypsum, with shallower absorption features overall indicating some spectral mixing. Minor absorption features appear at about 1.75 μm (consistent with gypsum) and 1.81 μm, which does not match to any of our minerals; this spectrum remains somewhat elusive. BL3-2 exhibits features consistent with epsomite (notably a 1.62 μm feature) but a 1.75 μm feature is perhaps a closer match for gypsum, suggesting some spectral mixing between these phases. BL2 and BL3-3 show unambiguous evidence for gypsum in the spectra, with some indications of triplet features around the 1.4 μm H₂O band, a clear 1.75 μm absorption feature, and a doublet feature near 2.22 μm.

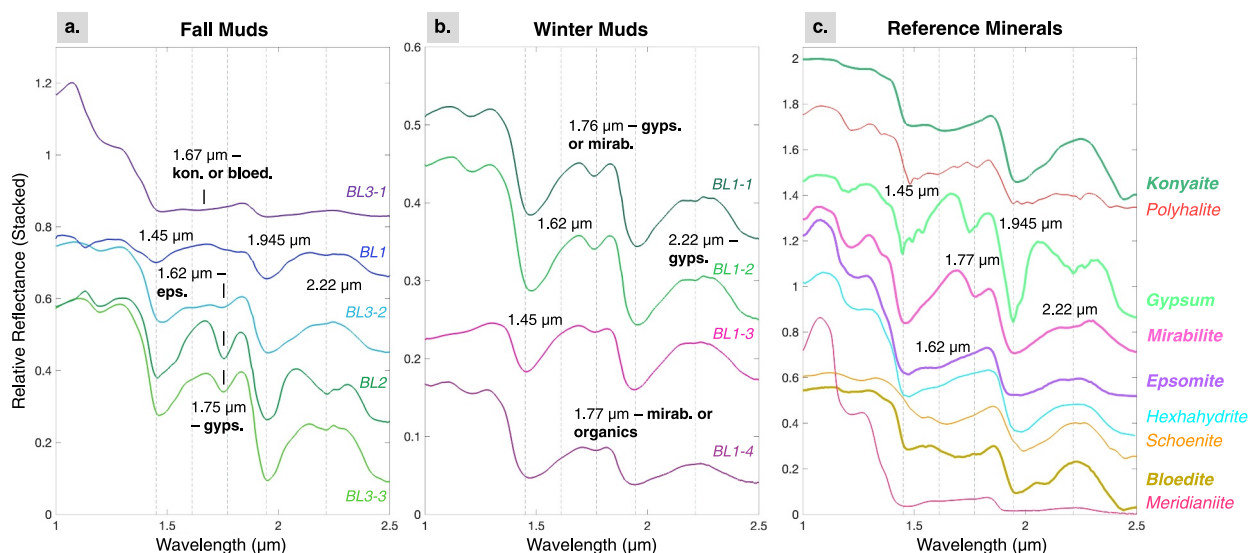


Figure 13. Spectra acquired from muds in the winter and fall. Spectra are labeled according to the lake from which they were acquired, in numbered order if more than one spectrum is presented for a given lake. Names of likely minerals are identified at or near relevant absorptions. “kon.” refers to konyaite; “bloed.” to bloedite; “eps.” to epsomite; “gygs.” to gypsum; and “mirab.” to mirabilite. Dotted lines represent general positions of major absorption features for salt species and are labeled. All spectra are stacked for clarity. (a) Spectra acquired in situ from fall muds. (b) Spectra acquired in situ of winter muds. (c) Reference spectra of relevant minerals, with the minerals likely identified in the fall and winter spectra in bold.

Spectra acquired in the wintertime are a bit more homogeneous, with some indication of mirabilite and gypsum (Figures 13b and 13c), but possible influence of organic materials as well. BL1-1 and BL1-2 indicate gypsum presence, given 1.76 μm features—possibly a blend of gypsum and mirabilite—and features at ~ 2.22 and ~ 2.28 μm , consistent with gypsum's doublet in this region. BL1-3 also shows a possible doublet in this region, but more dampened absorptions, indicating this spectrum might be consistent with mirabilite or possibly hydrated and organic material. BL1-4 appears to be the most consistent with mirabilite of the group, given a 1.77 μm absorption feature and the absence of a clear doublet around 2.2 μm .

5.3.4. Band Parameter Analyses

To understand general trends in mineralogy based on season and surface type, we employ band parameter analysis targeting bloedite, and investigate band minima variability between 1.54 and 1.86 μm . We do not include data from fall pools in this investigation, as the presence of interstitial or surface brine frequently lead to noisy or featureless spectra in this range, making their mineralogy uninterpretable.

Spectra from winter and fall muds and crusts, clustered into two distinct groups in our band parameter analysis (Figure 14), one group with broadly stronger BBP values and band minima between 1.6 and 1.7 μm , and one with weaker BBP values and band minima between 1.75 and 1.8 μm . Most spectra of fall crusts are most consistent with double salts and moderately hydrated salts like epsomite and hexahydrite. Fall crust spectra make up the majority of points clustering with stronger BBP values and shorter band minima wavelengths. Within this region of the plot, fall crust spectra plot close to the schoenite reference mineral position, with some variability in band minimum approaching konyaite, and some spectral data approaching the meridianiite, epsomite, and hexahydrite reference mineral positions (Figure 14). A few additional fall crust data plot with longer-wave band minima, nearing the mirabilite reference mineral position. Still other data from fall crusts plot with BBP values around 0.6 but band minima closer to mirabilite. This latter group of points likely represents mixed mirabilite/epsomite samples, as the BBP values for these spectra matches that of epsomite, but the band minima are a closer match for mirabilite (Figure S3 in Supporting Information S1). Spectrum BL3-1 from Figure 12a is an excellent example of this candidate material.

Meanwhile, fall muds plot in a much more tightly clustered region, near the gypsum band minimum. These data have a slightly lower value for the BBP than gypsum, and band minima around 1.76 μm . This suggests possible mixing with another material, which may be mirabilite (drawing down the BBP and the absorption feature

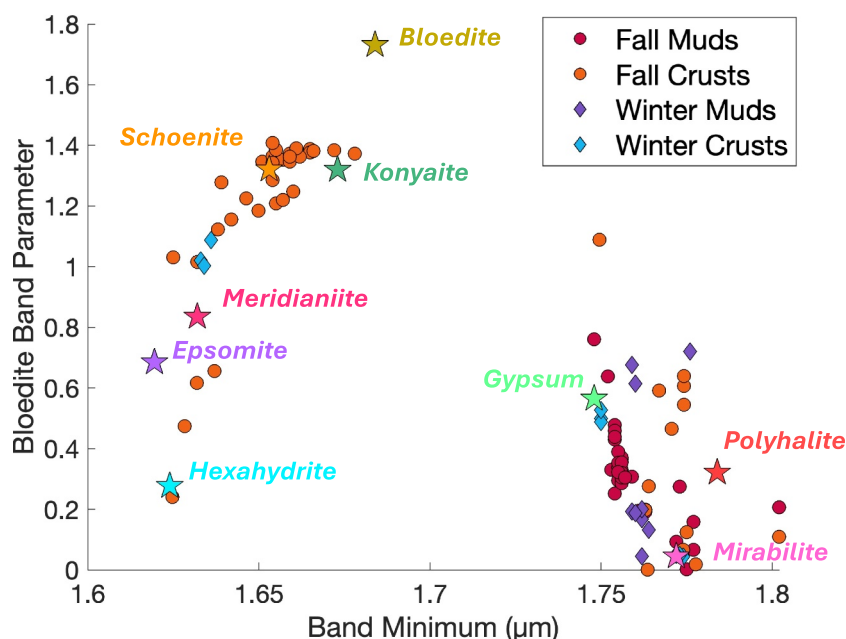


Figure 14. Bloedite band parameter values for all reference minerals (stars) and for in situ spectral data from winter and fall muds and winter and fall crusts.

towards longer wavelengths). A few fall muds plot with higher bloedite band parameters than gypsum, or values closer to mirabilite, suggesting additional mineralogical mixing.

Nine winter crust spectra are analyzed here, all of which plot in distinct regions in Figure 14. One set of spectra plot extremely close to the mirabilite reference point; these spectra are from Basque Lake 4 (BL4-1 and BL4-2 on Figure 12b) and indeed are close matches for a mirabilite spectrum. Three additional spectra taken from BL1 (one of which is plotted as BL1-3 on Figure 12b) plot closely to the gypsum reference mineral position. Overall their spectra are closer matches for mirabilite (see: results regarding spectrum BL1-3), but the position of the 1.75 μm band nevertheless suggests gypsum. Three spectra also from BL1 (BL1-1 and BL1-2 on Figure 12b) plot closer to the meridianiite spectral reference position, and indeed match meridianiite well spectrally.

Winter muds exclusively have band minima between 1.75 and 1.78 μm . These are largely between the gypsum and mirabilite band minima, with a cluster closer to mirabilite's BBP value. Additional points from winter muds plot with stronger BBP values; these points might represent a mixture of gypsum and Mg-sulfate (e.g., epsomite) present in the muds in the winter.

We additionally plot band minima in histogram form for the two seasons and three material types (neglecting fall pools, for the reasons delineated above) but we do include winter pools (Figure 15). We observe clear seasonal changes when we plot the data in this manner. Fall muds (Figure 15a) exhibit band minima distributed around 1.75 μm , consistent with gypsum, and around 1.77 μm , consistent with mirabilite. The relative abundance of minima consistent with gypsum is fewer for fall crusts than muds, while absorption features similar to mirabilite (1.77 μm) are common in both surface materials (Figure 15b). Meanwhile, absorptions near 1.62–1.64 μm are observable for fall crusts, which are consistent with epsomite, and band minima between 1.64 and 1.68 μm are also observable for fall crusts (Figure 15b). Such features between 1.64 and 1.68 μm are consistent with schoenite, konyaite, and bloedite. These band minima are nearly completely absent across all other kinds of material, and entirely absent in winter crusts.

Winter muds have band minima suggesting mixed gypsum/mirabilite/organic materials (Figure 15c) and have a narrower distribution of absorption features than fall muds. Winter crusts lack evidence for absorption features between 1.64 and 1.68 μm , indicating the disappearance of double salts. Instead, winter crusts show features consistent with meridianiite, epsomite, mirabilite, and gypsum (Figure 15d). Meridianiite has a band minimum

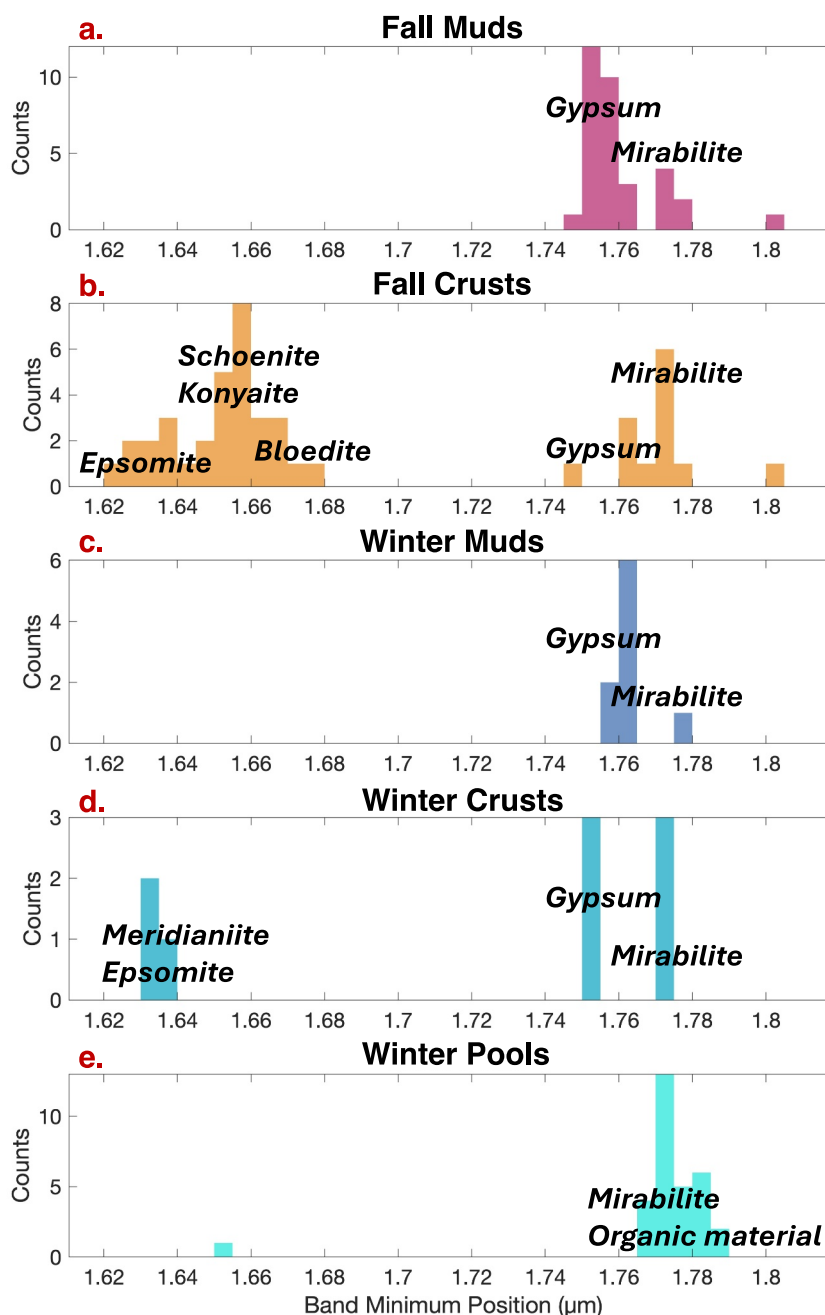


Figure 15. Histogram counts of band minima between 1.53 and 1.86 μm , based on season. Approximate positions of band minima for reference minerals are labeled. (a) spectral band minima for fall muds; (b) for fall crusts; (c) for winter muds; (d) for winter crusts; and (e) for winter pools.

feature similar to epsomite, and features for these spectra are similar to both meridianiite (e.g., spectrum BL1-2 from Figure 12b) and epsomite (e.g., spectrum BL1-4 from Figure 12b). Winter pools primarily have features around 1.77–1.79 μm , consistent with either mirabilite or organic material, and one outlier with a feature closer to 1.65 μm (Figure 15e).

5.4. Raman Analyses

Raman spectra of materials of interest were obtained in winter 2024 and 2025 and fall 2024, and primarily reveal a diversity of Mg-sulfate hydration states across lakes and seasons, as well as some evidence for gypsum in muds

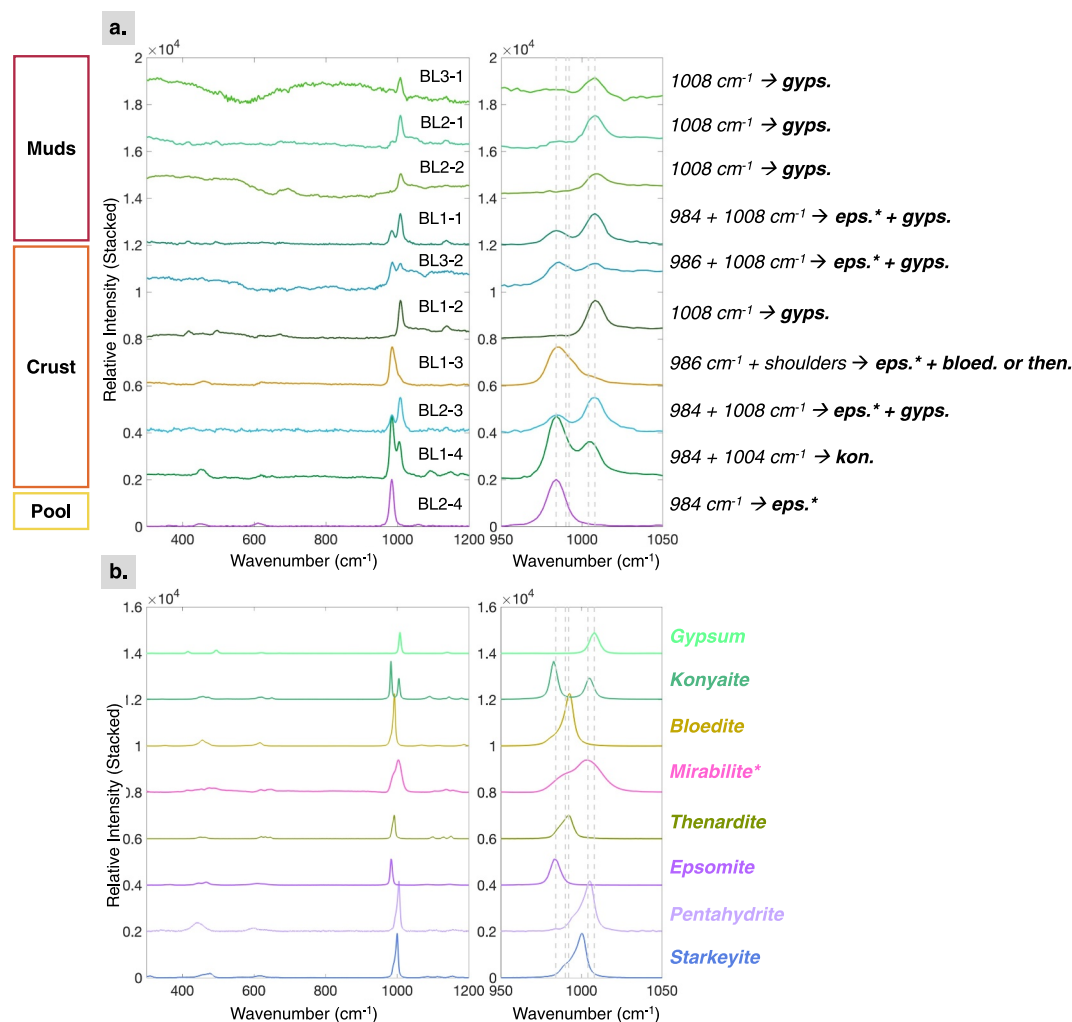


Figure 16. Stacked Raman spectra from fall 2024 and reference materials. The gray dashed lines on the right panels cross 984 cm^{-1} , 990 cm^{-1} , 992 cm^{-1} , $1,004\text{ cm}^{-1}$, $1,008\text{ cm}^{-1}$ from left to right. (a) In situ Raman spectra collected of different materials in fall, 2024. Material type is indicated by the boxes on the left. Major peaks, and mineral assignment based on such peaks, are noted on the right side of the plot. “eps.*” is used as a catch-all referring to epsomite or hexahydrate; “gyps.” refers to gypsum; “bloed.” to bloedite; “then.” to thenardite, and “kon.” to konyaite. Spectra have been magnified for clarity as needed. (b) Raman spectra of relevant reference materials, with the same gray dashed line positions as (a). Spectra have been magnified for clarity as needed. Mirabilite is starred (*) because available reference spectra for mirabilite differ slightly.

and crusts. Here we evaluate fall and winter Raman spectra from different material types, and discuss representative Raman spectra of each material type. We focus our analyses primarily on the $\sim 1,000\text{ cm}^{-1}$ peak, associated with the $\text{SO}_4\text{ } \nu_1$ vibrational mode, as this is the most intense peak for our Raman spectra and the most reliable diagnostic of different materials of interest for this work. All Raman spectra, disambiguation of plot nomenclature, and Raman peak assignments are available in our data repository (Hughes et al., 2025).

5.4.1. Fall Raman Data

Fall muds primarily indicate the presence of gypsum from Raman peaks at $1,008\text{ cm}^{-1}$ (Figures 16a and 16b). BL2-1 has a slight shoulder around 984 cm^{-1} that might be consistent with a Mg-sulfate contribution, but the feature is not clear. One spectrum of muddy material—BL1-1 in Figure 16a—has a clear additional peak at 984 cm^{-1} suggesting the presence of epsomite or hexahydrate, and gypsum.

Fall crusts are somewhat more diverse. BL3-2 has peaks at 986 cm^{-1} and $1,008\text{ cm}^{-1}$, indicating the presence of a Mg-sulfate (possibly epsomite) and gypsum. BL1-3 has a peak at 986 cm^{-1} , one shoulder near 992 cm^{-1} , and

another shoulder near $1,004\text{--}1,008\text{ cm}^{-1}$. This spectrum appears consistent with contribution from epsomite or another Mg-sulfate given the 986 cm^{-1} peak, but may have contributions from additional materials such as bloedite or thenardite, which have peaks at 992 cm^{-1} and 991 cm^{-1} respectively, and/or mirabilite, which has a $1,004\text{ cm}^{-1}$ peak with another peak around 990 cm^{-1} . Mirabilite may be a good contender to explain both peaks, with the caveat that the mirabilite spectra from Hughes et al. (2023) and Hamilton and Menzies (2010) differ (the latter lacks a $1,004\text{ cm}^{-1}$ feature). BL2-3 is consistent with a strong contribution from gypsum at $1,008\text{ cm}^{-1}$, and a contribution from epsomite or hexahydrate as well, based on the 984 cm^{-1} Raman peak.

BL1-4 is an interesting case: this spectrum has clear peaks at 984 and $1,004\text{ cm}^{-1}$, which is consistent with konyaite, which has major peaks at 983 and $1,005\text{ cm}^{-1}$. Our spectrometer has a 2 cm^{-1} resolution, such that peaks at 983 and $1,005\text{ cm}^{-1}$ could appear at 984 and $1,004\text{ cm}^{-1}$; meanwhile, konyaite's peaks at $1,090$, $1,145$, and $1,180\text{ cm}^{-1}$ are shared by the BL1-4 spectrum (Figures 16a and 16b), indicating konyaite is an excellent match for this material. The alternative is a possible mix of epsomite and pentahydrate (which have peaks at 984 and $1,004\text{ cm}^{-1}$, respectively); however we do not prefer this interpretation because the relative intensities and minor peaks are a more parsimonious match for konyaite.

Only one Raman spectrum for pool salts was successfully obtained during the fall; all additional attempts resulted in signal that was too low, suggesting brine swamped the signal. The single spectrum we did obtain from a pool has a peak at 984 cm^{-1} , consistent with epsomite or hexahydrate.

No material appears to match starkeyite's $\sim 1,000\text{ cm}^{-1}$ peak, indicating starkeyite is not present from Raman data in the fall.

In summation, in the fall, we observe evidence in our Raman data for gypsum and epsomite within muds, epsomite, gypsum, konyaite, and possible bloedite or thenardite in crusts, and epsomite in pools.

5.4.2. Winter Raman Data

Raman acquired in the winter primarily indicate highly hydrated Mg- and Na-sulfates, possible metastable Na-sulfate heptahydrate, and gypsum (Figures 17a and 17b). Winter muds are consistent with gypsum, via Raman peaks at $1,008\text{ cm}^{-1}$. Raman spectra of crusts often show peaks at 988 cm^{-1} . This peak is most likely associated with a metastable Na-sulfate heptahydrate mineral, which has a peak at 987.6 cm^{-1} (Hamilton & Menzies, 2010) (Figure 17a). Detailed analysis of the SO_4 ν_1 , ν_2 , and ν_4 peaks for spectra containing the 988 cm^{-1} peak indicates that ν_2 and ν_4 peaks, when interpretable, are often closer matches for mirabilite than the Na-sulfate heptahydrate peaks (Figure S4 in Supporting Information S1). This suggests a possible mix of Na-sulfate hydration states. With that noted however, we assign these spectra in our analyses to Na-sulfate heptahydrate (rather than mirabilite) based on the strength of the 988 cm^{-1} peak. Raman analyses of crusts BL1-3, BL4-1, BL4-2 and BL1-5 all have peaks at 988 cm^{-1} , while BL1-4 has a peak at 984 cm^{-1} , consistent with epsomite or hexahydrate. Data from pools show a range of peak positions. One pool spectrum, BL2-1, has a peak at 992 cm^{-1} . The 992 cm^{-1} spectral feature matches bloedite particularly well, and we observe peaks at ~ 620 , $\sim 1,070$ and $\sim 1,190\text{ cm}^{-1}$ that are close to bloedite's 617 , $1,070$ and $1,188\text{ cm}^{-1}$ peaks (Figure S5 in Supporting Information S1); however our spectrum is missing a minor $1,114\text{ cm}^{-1}$ peak associated with bloedite's $\nu_3\text{ SO}_4$ vibrational mode (Shi et al., 2024). Other than bloedite, it's possible this spectrum may have resulted from convolution between meridianiite and another phase, leading to a slightly shifted 990 cm^{-1} meridianiite feature due to such convolution, or may be influenced by the spectral resolution of the spectrometer (e.g., the peak position may be closer to 993 or 991 cm^{-1}). An alternative material responsible for the $1,070$ and $1,190\text{ cm}^{-1}$ peaks other than bloedite, however, is not clear, so we favor bloedite. A second pool spectrum (BL3-1) once again has a peak at 988 cm^{-1} , indicating either the metastable Na-sulfate heptahydrate or a mix of epsomite and meridianiite. BL4-3 has a peak at 984 cm^{-1} , indicating epsomite or hexahydrate. Finally, BL4-4 has a peak at 990 cm^{-1} , indicating the presence of meridianiite or mirabilite, which has a peak at 990 cm^{-1} (Genceli et al., 2009; Hamilton & Menzies, 2010).

In summary, winter Raman spectra suggest contributions of gypsum to muds, metastable Na-sulfates and epsomite or hexahydrate to spectra from crusts, and a mix of meridianiite or mirabilite, epsomite, hexahydrate, possible bloedite and possible metastable Na-sulfates in the pools.

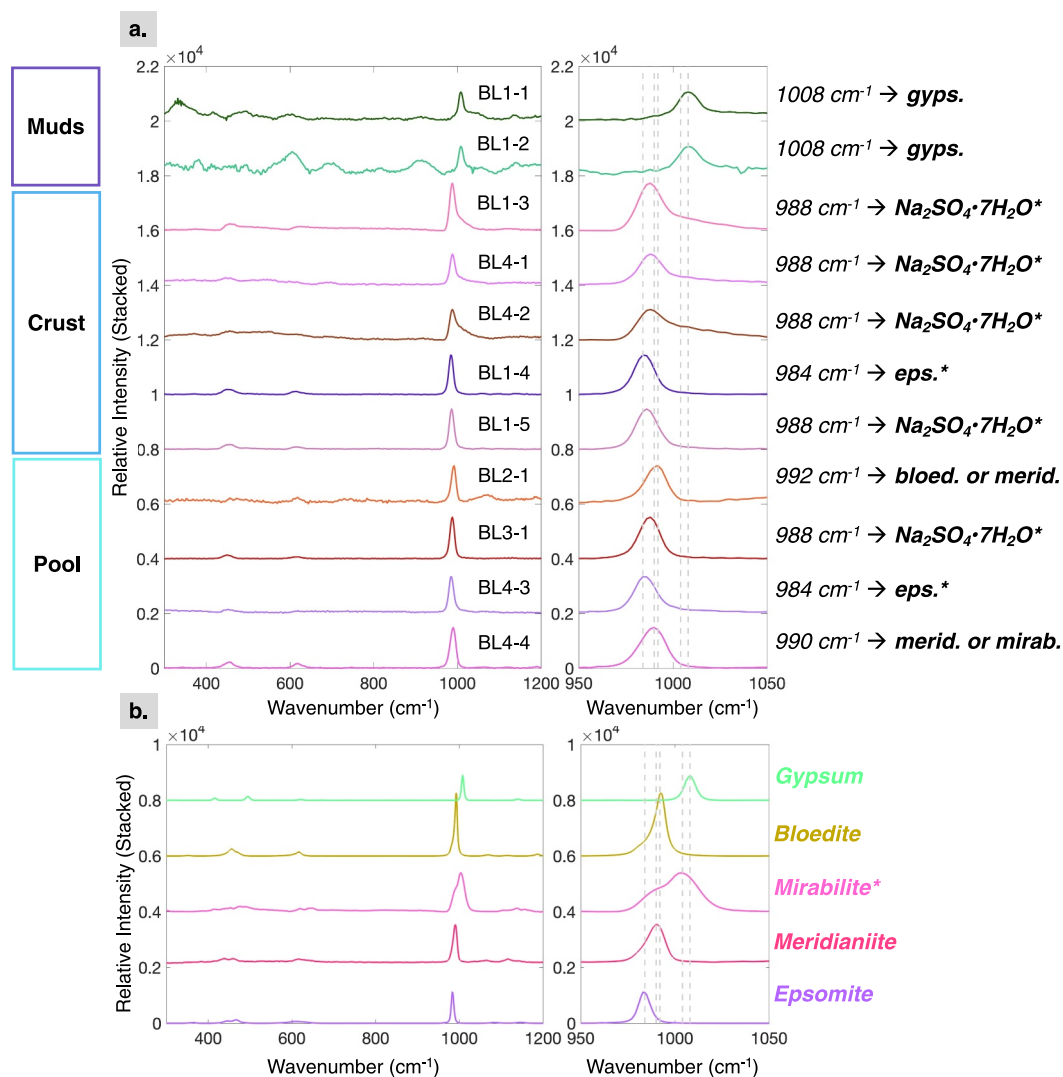


Figure 17. Stacked Raman spectra from winter 2024 and 2025 and reference materials. The gray dashed lines on the right panels cross 984 cm⁻¹, 990 cm⁻¹, 992 cm⁻¹, 1,004 cm⁻¹, 1,008 cm⁻¹ from left to right. (a) In situ Raman spectra collected of different materials in winter, 2024 and 2025. Material type is indicated by the boxes on the left. Major peaks, and mineral assignment based on such peaks, are noted on the right side of the plot. “eps.*” is a catch-all referring to epsomite or hexahydrate, “bloed.” refers to bloedite, “merid.” to meridianiite and “mirab.” to mirabilite. We include Na₂SO₄·7H₂O as a possible contender for the 988 cm⁻¹ peak, but do not have an available reference spectrum for this material so do not make a definitive assignment. Spectra have been magnified for clarity as needed. (b) Raman spectra of relevant reference materials, with the same gray dashed line positions as (a). Spectra have been magnified for clarity as needed. Mirabilite is starred (*) because available reference spectra for mirabilite differ slightly.

6. Discussion

6.1. Distribution of Minerals Within Lakes and Between Lakes

We summarize our observations, in the context of previous literature, in Figure 18. We suggest three general models for lake evolution, one for BL1 and BL2, one for BL3, and one for BL4. Nesbitt (1990) identified a sub-surface groundwater system for Basque Lake 2 with ongoing interactions between groundwaters and basin muds. Such a system is likely also present for BL1, BL3 and BL4, given there is no clear source of surface inflow for these lakes. The groundwater table is likely responsible for depositing crystalline gypsum interstitially within the muds, present in both fall and winter. Gypsum likely forms in muds due to its lower solubility than other sulfates, and precipitation before fluids concentrate in pools. This groundwater table may also precipitate mirabilite in the muds (Table S1 in Supporting Information S2). Perennial basement salts, which we did not analyze, appear to be

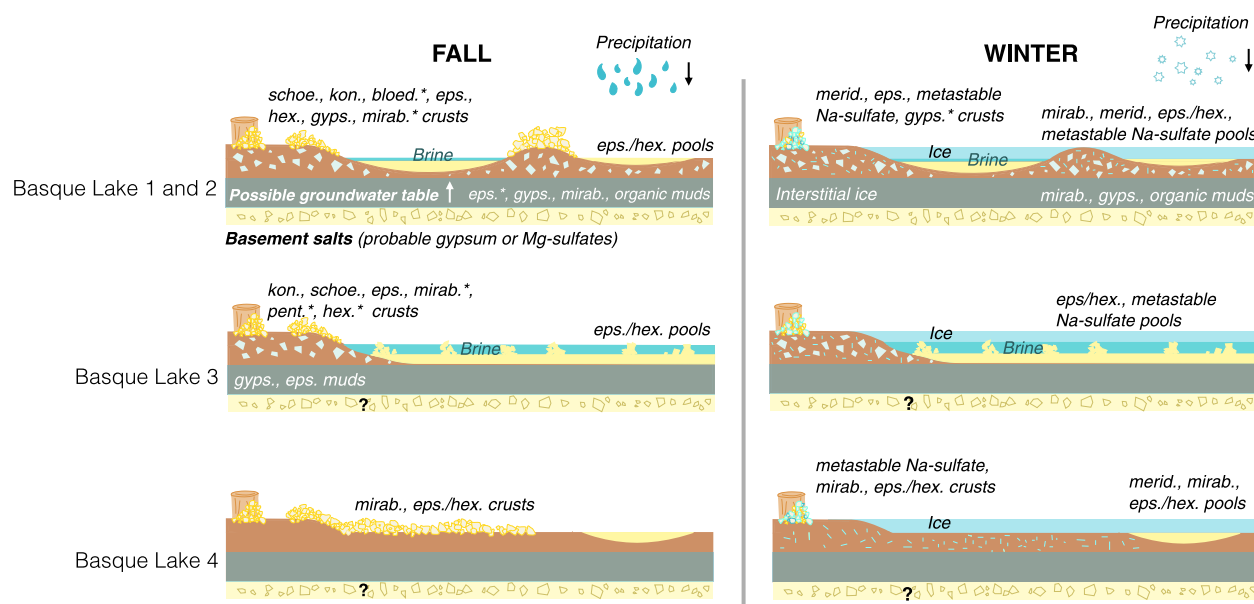


Figure 18. Idealized mineralogical distribution of materials in the pools, muds and crusts for the Basque Lakes, in both the fall and winter. A groundwater table might be in part responsible for the deposition of gypsum and other salts in the muds. Precipitation (rain and snow) is the other intermittent source of inflow. Brine pools sit at the surface and primarily form Mg-sulfates in the fall, while Na- and Mg-sulfates form in the winter. Mg-Na-K-SO₄ salts form in the fall from surface brines or possible groundwater brines, or both. In the winter, double salts melt/transform and mono-cation salts form, like meridianiite, and Na-sulfate heptahydrate. The “schoe.” refers to schoenite; “kon.” to konyaite, “bloed.” to bloedite, “eps.” to epsomite, “hex.” to hexahydrate, “gyps.” to gypsum, “mirab.” to mirabilite, “merid.” to meridianiite. The * indicates possible but not definitive mineral detection.

present at the base of at least one Basque Lake (Jenkins, 1918), possibly composed of Mg- and Ca-sulfates. Brine pools concentrate salts like epsomite and hexahydrate in the fall. In the winter, lakes freeze over, cryoconcentrating brines and forming meridianiite, a metastable Na-sulfate heptahydrate, and epsomite. Seasonal salt crusts may form from the wider hydrological brine or from concentrated surface brines leaching into the muds and efflorescing (Figure 18). The presence of mixed double salts in fall crusts suggests that brine waters saturated within sediments and concentrated in Na, Mg, K, and SO₄ “wick” to the surface and deposit salts like epsomite, konyaite and schoenite. In the winter, these crusts transform into metastable Na-sulfate heptahydrate, meridianiite, mirabilite, and epsomite (Figure 18).

6.2. Modeled Salts Versus In Situ Identifications

The distribution of salt hydrates is moderated by the thermodynamics and kinetics of the system. Eugster and Hardie (1978) for example proposed the evolution of the Basque Lakes followed a progression of magnesite and calcite precipitation, followed by gypsum, while fluids concentrate in Mg²⁺, SO₄²⁻ and Cl⁻. Our study did not reveal magnesite or calcite, which likely precipitated out of groundwater fluids early in the fluid evolution (e.g., Nesbitt, 1990), or are obscured spectrally by other salts. Magnesite might also account for excess Mg higher than expected for Mg-sulfates (Figure 9), but without modeled carbon, cannot be verified with XRF. Gypsum clearly forms in the muds, following the predicted chemical evolution pathway. We investigated the evolution of these solutions as they continue to concentrate via FREZCHEM and in situ analysis, and find that models are largely successful at predicting major salt species forming in pools, but are more limited at characterizing the mineralogical variability of crusts for the Basque Lakes (Table S2 in Supporting Information S2).

In surface muds and crusts, Mg-sulfates appear to be holding onto more water than is thermodynamically expected in the fall. Air temperature data indicate that hexahydrate and starkeyite should be the stable or metastable Mg-sulfate phases in the fall in crusts and muds (Figure 8). Hexahydrate may be present, but is difficult to distinguish from epsomite in NIR and Raman data. However, we see no evidence for starkeyite; the 1,000 cm⁻¹ SO₄ ν₁ starkeyite peak is notably absent in Raman spectra. The finding that salts appear to be more hydrated than favored, particularly in crusts, aligns with previous research establishing the dehydration of epsomite or

hexahydrate to starkeyite as extremely slow (Wang et al., 2016), and suggests that salts in the fall persist as metastable or thermodynamically unfavored species.

In brines however, we find the model aligns better with in situ findings with respect to Mg-sulfate hydration state. Epsomite is thermodynamically favored, and is indeed well-represented by our in situ data from pools. This implies that the longer crystallization time in pools, and possibly more stable temperatures than air temperature, allows for thermodynamically favored Mg-sulfate phase to form.

Gypsum was modeled to form at minor amounts in both seasons, and was indeed identified clearly via Raman and NIR analysis in both seasons. As the models rely on brine compositions rather than groundwater fluid compositions, the Ca abundance in the simulations is very low; gypsum within the muds likely precipitated out of solution prior to concentration in the pools, so the model would not be expected to simulate the formation of copious gypsum. However, gypsum is clearly the stable Ca-sulfate phase in both the fall and winter, despite a transformation between gypsum and anhydrite at temperatures relevant to the Basque Lakes (Chou et al., 2013; Figure 8)—i.e., gypsum should be stable in the winter at RH values above 50% and temperatures below or near 0°C, and anhydrite should form in the fall at <40% humidity conditions. Gypsum crystals appear to be resistant to transformation, however, likely in part due to their size and structure inhibiting dissolution, and possibly higher water availability in muds than in the air or in crusts. Like starkeyite, anhydrite can also have extremely slow crystallization kinetics even when thermodynamically favored (Charola et al., 2007).

Fall Na-sulfates varied in species and distribution, often present in thermodynamically unfavored phases. Bloedite was commonly predicted to be the stable Na-sulfate in the fall, particularly for models run at 303.15 K (Warm Evap 2). Mirabilite was also predicted to form in warmer temperatures (primarily in Warm Evap 1 models), and indeed, mirabilite has a stability field at 25°C (Marion & Farren, 1999), suggesting it is thermodynamically viable at these temperatures even as bloedite is also favored. Neither were identified in fall pools, suggesting Na⁺ typically remained concentrating in solution while Mg-sulfates precipitated. Mirabilite was frequently inferred via NIR for muds, crusts and pools in the fall. We never directly confirmed bloedite via NIR analysis. Bloedite's lack of clear identification in the field may be explained by the detection of a different salt: konyaite, the 5-hydrate Na-Mg-sulfate phase, which was likely identified via NIR analysis and confirmed via Raman analysis. Konyaite's stability range in real-world environments is likely colder and higher humidity than the stability field for bloedite. Konyaite formation has been observed in caves with near 100% RH and temperatures between 5.8 and 8°C (Onac et al., 2001), while in caves with RH values between 80% and 85%, and temperatures above 12°C, bloedite appears to be the stable phase (Freeman et al., 1973). Fall temperatures for the Basque Lakes are higher than these caves, and RH is lower, suggesting bloedite rather than konyaite should be the stable phase. We did however firmly detect konyaite, suggesting konyaite might have a wider temperature range of stability than predicted, that konyaite might be favored in multicomponent environments (rather than simple Na-Mg-SO₄ systems), or that konyaite might have formed earlier in the season, but persisted through spring, summer and fall. Konyaite is known to persist in efflorescent crusts for extended periods (Keller et al., 1986), which indicates transformation to bloedite might be sluggish, and may permit supersaturation of solutions with respect to mirabilite, epsomite and thenardite (Keller et al., 1986). It may be that konyaite's resistance to alteration is in part responsible for the metastability of salts in the Basque Lakes, by permitting Na-sulfates to remain in otherwise supersaturated solutions.

Overall we find even with high temperatures and low humidities, salts do not dehydrate to the thermodynamically expected anhydrous phases, likely due to fluctuating atmospheric humidity, higher available water in the muds and crusts, and sluggish kinetics.

In the wintertime, thermodynamic modeling accurately predicted the formation of meridianiite exclusively under cold temperatures. Water activity and temperature conditions, as evaluated in 2019, indicated that meridianiite should be the stable salt phase in the winter brines, which we validate via NIR. The model also predicted epsomite might form from freezing conditions, though in low mass fraction, which also aligns with our frequent detections of epsomite in the winter, including in brine pools and crusts (BL4-3 in Figure 17). The frequency of our detections of epsomite in Raman spectra suggests more common precipitation than might be expected from the modeling. In the case of the winter crust epsomite detections, such observations might be readily explained via the air temperature and humidity data. In the winter, within the air, epsomite is exclusively the thermodynamically favored Mg-sulfate hydrate for each of our winter field seasons (Figure 8). Epsomite that formed in the pools in

the winter may have formed in warmer environments and persisted into the winter, or may in fact be forming in the winter, in spite of its thermodynamic unfavorability.

In addition to the meridianiite and epsomite detections, material with a 988 cm^{-1} Raman peak was present in crusts and pools in the winter and was entirely absent in the fall, which is likely Na-sulfate heptahydrate (Hamilton & Menzies, 2010). The presence of material with a 988 cm^{-1} peak in both crusts and pools suggests that the differences in environmental conditions between crusts and pools—e.g., water availability, surface temperatures versus brine temperatures, and temperature variability or stability—did not preclude the putative metastable Na-sulfate heptahydrate in either condition. Indeed, its presence in the brine pools—which have stable temperatures due to ice insulation (Buffo et al., 2022)—would indicate that it likely formed through cryoconcentration of the brine. Likewise, the identification of mirabilite in winter salt cores via NIR suggests that additional Na-sulfates are likely forming from cryoconcentration of solution. Previous work has established that mirabilite should crystallize early from solution via freezing of Na-SO₄ bearing brines (Herrero et al., 2015). Our modeling echoes this; in models targeting BL1 and BL2 (for which we have the most in situ brine data) FREZCHEM predicts that mirabilite precipitates after about 5% of the solution has frozen, before meridianiite precipitates, in two of the four freezing models (Figure S6 in Supporting Information S1). In another freezing model, mirabilite and meridianiite form at the same fluid concentration. In Warm Evap 1 models however we find that Na-sulfates invariably precipitate after epsomite for BL1 and BL2 brine compositions, once solutions have concentrated by about 50%–90% (Figure S7 in Supporting Information S1). It is therefore likely that a changing solubility at low temperatures permits metastable Na-sulfate heptahydrate and mirabilite to form early from solution, along with meridianiite, which is consistent between modeling and field observations. The Na-sulfate heptahydrate (a phase not included in FREZCHEM) appears to be forming within both crusts and pools, indicating a stability field overlapping with mirabilite.

Winter modeling predicted no bloedite precipitation, and predicted that mirabilite should be the second most common sulfate phase in the winter (Table 3). Bloedite lacks a stability field close to 0°C, though it is typically stable down to ~7°C (Warren, 2006) and has been found in cold environments at temperatures near 4.5°C precipitating from fluids rich in K and Cl (Keys, 1979). The Basque Lake fluids are not enriched in K and Cl, suggesting cold-temperature bloedite should not form in this environment. However, a possible bloedite detection was identified in a pool in BL2 (Figure S5 in Supporting Information S1), from a salt core extracted in –0.9°C air temperatures and likely colder brine temperatures. We therefore speculate that bloedite might have a cold-temperature stability field, but the conditions permitting such a field remain enigmatic.

Other salts modeled to form in the winter, such as schoenite and magnesite, were not identified, likely due to their low mass fraction, spectral obscuration, or due to kinetic inhibition.

In summation, we observe changes in mineralogy contingent upon environmental factors and depending on season, which notably include formation of meridianiite, mirabilite, and a metastable Na-sulfate heptahydrate in the winter, and various double salts in the fall. Complex Mg-sulfate, Na-sulfate, and Na-Mg-sulfate phases all persist in different hydration states in the Basque Lakes, suggesting salts of variable hydration states and stabilities can co-occur in disequilibrium.

Our results can be useful for the application of thermodynamic modeling to both terrestrial and planetary systems. Current available thermodynamic models rarely consider konyaite, which appears to form extensively in the Basque Lakes at temperatures that overlap with modeled bloedite stability. Konyaite may form elsewhere in the solar system on Mars and icy moons, but is currently largely ignored in the context of planetary brines. Inclusion of this Na-Mg-sulfate hydrate in future modeling databases may therefore improve results and applications. Our results also indicate that thermodynamic modeling alone cannot account for real-world assemblages (e.g., metastable salts or salts persisting beyond thermodynamic boundaries). Long-term kinetic studies of brine precipitates under arid, Mars-like conditions may improve upon our initial interpretations, and help contextualize the application of thermodynamic modeling to Mars. While the Na-sulfate heptahydrate appears to be metastable, reducing its suitability for inclusion in thermodynamic models, evaluating the precipitation kinetics of this material may also aid in considering sulfate formation on other planetary bodies and Earth, and in particular Na-sulfates as low temperature indicators. With these caveats, thermodynamic modeling successfully predicted the major constituents of brine pools in the fall (epsomite) and winter (meridianiite), suggesting that modeling may be useful for predicting some precipitates from Mars fluids.

6.3. Geochemical Variability

The Basque Lakes see various geochemical trends based on seasons, relating to changing concentrations of Mg, S, Ca, K, and Al. In the winter, interstitial ice seeps into muds and salt deposits, such that abundances of elements decrease proportionally with our detection method. Higher hydration states of salts may also contribute to this change. We therefore note that XRF is not a definitive technique for establishing Mg-sulfate hydration state variability seasonally, due to the influence of ice.

Ca-sulfate salts appeared to decrease substantially in mass fraction in the winter season. A simple explanation is that muds tend to have the highest amount of interstitial ice, and the lowest sums of oxides for characterized elements by the XRF in the winter. The lowered detectability of Ca-sulfate with XRF is not echoed with Raman, which was able to detect gypsum even at very low mass fractions in winter muds, consistent with the fact that gypsum is highly Raman active.

K₂O appears to be present in both winter and fall pools from XRF data, yet its associated salt species is not clear. Schoenite, which has a Raman peak is at 994 cm⁻¹ based on Hughes et al. (2023), was not observed in pools, crusts or muds via Raman analysis. Schoenite was also not identified in pools via VNIR analysis for fall or winter. The source of such seasonally present K₂O in pools, therefore, is not clear.

The change in seasonal aluminum abundance, decoupled from silica in the winter, is intriguing. These results suggest aluminum without silica might be a useful tracer of cold-climate brines. Nesbitt (1990) note that X-ray amorphous Si- and Al-bearing phases appear to be present in the muds and solutions, but that while minor Al in groundwater increases in concentration within the pools, Si appears to decrease as the brines are more concentrated (Table 1 in Nesbitt, 1990). It is possible that warmer seasons permits such aluminum to remain in solution, but Al-bearing materials precipitate out in the winter. One might consider Al-sulfate precipitation, such as alunite, a candidate for such a process, but Al-sulfates form in much lower pH environments than those of the Basque Lakes (e.g., Long et al., 1992; Nordstrom, 1982). At pH conditions relevant to the Basque Lakes, gibbsite would be expected to be the favored Al-bearing mineral (Nordstrom, 1982). Gibbsite is highly insoluble, suggesting it is less likely to be stable in brine solutions, but it can form rapidly (Barnes, 1975). It is also possible that a metastable Al-phase forms at low temperatures from brine pools, as metastable Al-bearing materials with higher solubility than gibbsite have been known to form rapidly from solutions (Wesolowski, 1992), and Nesbitt did not identify X-ray crystalline Al-bearing materials. We therefore favor the possibility that in the winter, X-ray amorphous Al-oxides tend to form, which may act as a tracer of colder temperatures, but note that further research into temperature dependent Al-salt precipitation in brine pools is needed to confirm this.

6.4. Implications for Mars

6.4.1. Mixed-Hydration Mg-Sulfates on Mars and Earth

Mg-sulfates have been identified on Mars via in situ analysis, but the rover-identified Mg-sulfates in Gale and Jezero crater are not present in the form of primary salt crusts, nor preserved, lithified playa deposits. In Gale crater, sedimentary Mg-sulfates are deposited in fluvio-lacustrine environments as likely diagenetic materials from seeping brines (e.g., Rapin et al., 2019), as well diagenetic sulfates in eolian bedrock (Chipera et al., 2023; Tutolo et al., 2025). In Jezero, mixed-hydration Mg-sulfates have been identified in igneous units on the crater floor and in sedimentary deposits in the western fan (Phua et al., 2024). Most sulfates identified by these rovers are in the form of cements and diagenetic features (e.g., veins and nodules). Detrital sulfates may also be present in these environments and in others, such as Meridiani Planum (McLennan et al., 2005).

These depositional environments explored by rovers on Mars therefore differ from those of the Basque Lakes, which are firmly hypersaline lacustrine environments. The Basque Lakes are most relevant to any saline lakes responsible for fluid infiltration and cementation of underlying bedrock on Mars (e.g., Rapin et al., 2019), or detrital sulfate grains transported by wind, rather than sulfate diagenesis of silicate bedrock, directly. We therefore consider how the metastable, mixed hydration state Mg-sulfates that form in the Basque Lakes may be relevant to any ancient evaporative lakes on Mars, which may have sourced the brines that cemented aeolian and fluvial bedrock, or altered volcanic rocks. We also consider the formation of mixed sulfates from brines and how these sulfates relate to fluid conditions and temperatures.

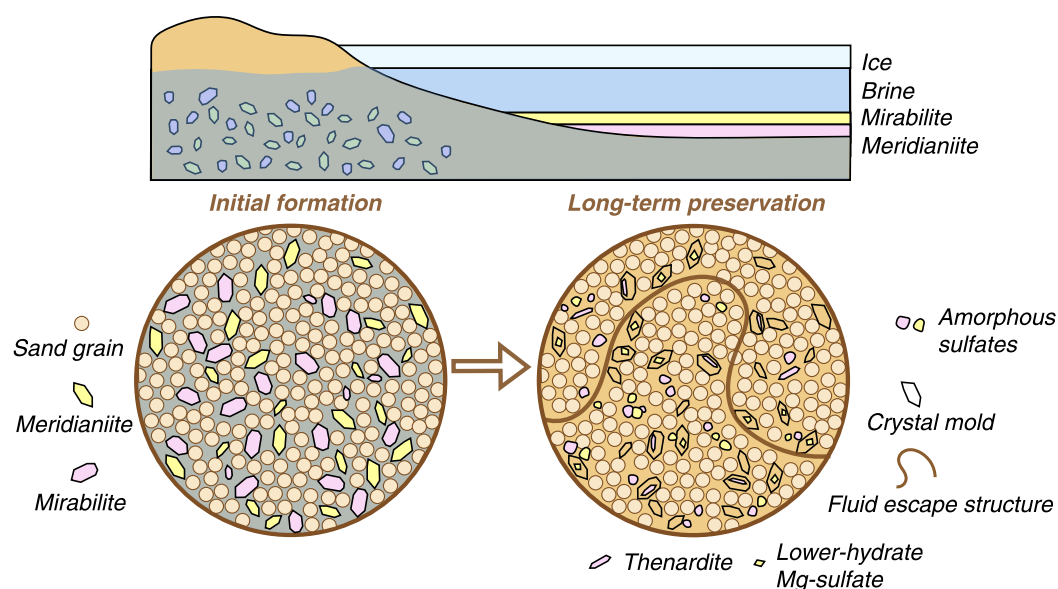


Figure 19. Model for cold-climate salts on Mars and their preservation. In a Na-Mg-SO₄-Cl rich fluid, meridianiite and mirabilite will precipitate via freezing. Fluids may seep into sediment and lead to precipitation of these salts. Over time, these salts are no longer stable due to long-term drying conditions. Volumetric water loss will lead to fluid escape structures, crystal molds, secondary minerals with lower hydration states (e.g., thenardite) and amorphous dehydration products.

Sulfates in multiple hydration states are clearly present across the Basque Lakes in both the fall and winter season. Salts vary seasonally—demonstrated by the appearance of meridianiite in the winter and the appearance of konyaite in the fall—but sulfates of multiple hydration states co-occur in each season. These results indicate that the Basque Lakes constitute a continually evolving metastable system, with ongoing transformations between salt phases, and sluggish kinetics preventing complete transformation into stable phases. Few studies have explored cold-temperature (near- or sub-freezing) sulfate-dominated efflorescent crust compositions, but low winter temperatures might stall kinetic transformations further, permitting persistence of epsomite or meridianiite in unfavorable environmental conditions. Sluggish kinetics might lead to the formation of a metastable Na-sulfate heptahydrate, which precipitates out of brine solutions due to cryoconcentration, and may form via the melting of konyaite and other double salts in crusts. In general, the Basque Lake system does not settle into equilibrium mineral assemblages on an annual cycle.

The reason for such metastability in the Basque Lake system is likely fluctuating atmospheric conditions, which affect the stability of salts in contact with the changing atmosphere. On Mars, both the diurnal and annual cycles of surface temperature have larger amplitudes than those of Earth (e.g., Rummel et al., 2014). Salts forming above the sub-surface skin depth or in contact with Mars's atmosphere may therefore have undergone frequent climatological forcings. Martian brine in contact with a changing atmosphere that seeped into sediment, or fluids with fluctuating temperatures on yearly bases, may produce assemblages of mixed, metastable phases. Such a system might lead to structural features indicative of volumetric water loss through time, or complex transformations, such as dewatering structures or pseudomorphic textures (Figure 19). Alternatively, systems with stable temperatures for long periods of time (e.g., deep groundwater systems, below the martian skin depth) might see fewer of these features.

We can apply these considerations to Gale and Jezero crater. Gale crater offers an example of Mg-sulfates that alternate in hydration state spatially, while Jezero crater hosts co-occurring Mg-sulfates of mixed hydration states. The CheMin instrument on board the Curiosity rover has thus far only detected alternating Mg-sulfate hydration states between drill holes in Gale's sulfate-bearing unit, and never two crystalline Mg-sulfates of different hydration states within the same drill (Chipera et al., 2023; Pandey et al., 2025). The variability between starkeyite and kieserite in Gale crater may be due to intervals of changing bulk fluid compositions, including possibly other salts buffering or changing stability, as is observed in the Basque Lakes. It's also possible and perhaps more likely that a groundwater table unaffected by atmospheric temperature fluctuations—as is sedimentologically possible

for the Gale crater sulfate-bearing unit (Roberts et al., 2025)—permitted the formation of equilibrated, stable salt phases from solution, rather than metastable phases that underwent ongoing transformation. This is consistent with a lack of reported pseudomorphic textures, crystal molds, or dewatering structures in this segment of the rover traverse.

In Jezero Crater, however, intimate mixtures of Mg-sulfates of differing hydration states ($\text{MgSO}_4 \cdot [3-5]\text{H}_2\text{O}$ and $\text{MgSO}_4 \cdot \text{H}_2\text{O}$) have been identified (Siljeström et al., 2024), possibly from lower-temperature (i.e., non-hydrothermal) fluids with moderate pHs. The reason for co-occurring Mg-sulfates of mixed hydration in Jezero crater, absent in Gale crater, is presently unknown. Phua et al. (2024) offer the possibilities that CheMin analytical methods influence Mg-sulfate decomposition, Ca-sulfates may buffer the formation of certain Mg-sulfate hydrates, and/or variable mixes of crystalline and amorphous salts may influence these differences. Results from the Basque Lakes suggest a fourth possibility: that the mixed hydration Mg-sulfate salts in Jezero crater might have formed in fluids affected by fluctuating temperatures, relatively close to the surface, permitting the formation and transformation of metastable phases. Meanwhile the Mg-sulfates in Gale crater may have formed from warm groundwater fluids disconnected from the atmosphere. Alternative explanations remain, but the duration of exposure time of the Gale crater sulfate-bearing unit salts and the Jezero crater salts may be comparable, as both regions are broadly Noachian to Hesperian in age, potentially forming in a window between 3.45–3.8 Ga (Goudge et al., 2012; Stack et al., 2020; Thomson et al., 2011). Such comparable ages limit the possibility that dehydration-induced amorphization is the sole culprit, and hint at different fluid sources or processes. Further examination of chemical/mineralogical rover data in sedimentological context is crucial for the evaluation of this possibility.

The hydration states of Mg-sulfates on Mars are consistently lower than those identified in the Basque Lakes. Crystalline kieserite and starkeyite have been confirmed in Gale crater, and various hydrated Mg-sulfates (with up to 5 molecular waters) have been detected in Jezero crater (Chipera et al., 2023; Pandey et al., 2025; Phua et al., 2024; Siljeström et al., 2024), but epsomite, hexahydrite, and meridianiite have only ever been inferred (Peterson et al., 2007; Peterson & Wang, 2006; Sheppard et al., 2021). These differences suggest either that the climates permitting the formation of Mg-sulfates early on Mars may have been similar to the Basque Lakes, but long-term drying conditions stripped water molecules from Mg-sulfates, or that fluids that formed the Mg-sulfates on Mars were mostly warm to hot with low water activity/humidity (Chipera et al., 2023). These differences place limits on the applicability of the Basque Lakes to Mars climates. However such differences may also indicate the possibility that fluids infiltrating Mars bedrock and forming Mg-sulfates may have been warmer (i.e., $>50^\circ\text{C}$) than those of the Basque Lakes.

6.4.2. Applications and Limits of Salts as Climate Indicators

The most robust evidence of climate-dependent salt transformations come in the form of double salts appearing in efflorescent crusts in the fall, and the formation of highly hydrated Na- and Mg-sulfates in crusts and pools in the winter. We confirm that double salts have clear seasonal dependency, including possibly schoenite, in spite of its stability field at 0°C (Marion & Farren, 1999). One possible bloedite identification in the winter indicates that bloedite may have a stability field at cold temperatures, but the lack of any other detections of bloedite in the winter suggests that on the whole, double salts act as robust paleoclimate indicators, particularly via NIR reflectance spectra. NIR analysis firmly distinguished seasonal formation of these materials, while Raman was less definitive.

These results suggest that the identification of double salts on Mars—very doable with NIR spectroscopy—would provide some constraints on the climate of formation. Konyaite in particular may be a mineral of interest for Mars, given its formation in temperatures between $6^\circ\text{--}8^\circ\text{C}$ and high humidity based on previous work (Onac et al., 2001), and our konyaite identification at 24°C . Our findings suggest konyaite might have a wider stability range than previously assumed, particularly as a metastable salt. However, the double salts schoenite, bloedite, and konyaite have never been identified on Mars, though bloedite has been inferred in Gale crater (Hughes et al., 2024) and some of these salts might be responsible for the polyhydrated Mg-sulfate signatures observed from orbit (e.g., Roach et al., 2009). Bloedite, schoenite and konyaite all have clear NIR signatures, but the $1.65\text{ }\mu\text{m}$ filter boundary (Murchie et al., 2009) for CRISM instrument data may make identification based on the spectral region of interest for this work difficult, as well as CRISM's higher noise than our field NIR spectrometer. The broadly dehydrating surface of Mars also may impact the legibility of features in the $1.56\text{--}1.86\text{ }\mu\text{m}$ region. Experimental work evaluating double salts such as these in Mars-relevant environments, and obtaining spectra

with CRISM-relevant wavelengths and noise, may help determine how identifiable these minerals might be using orbital data. Meanwhile, the SuperCam instrument on Perseverance is certainly equipped to detect double salts such as these, given CO₂ correction techniques, lower noise, and much closer target range (Reess et al., 2018). SuperCam also has the capacity to couple Raman and NIR detections, and we note here that SuperCam may observe different materials via each detection method, with NIR more sensitive to double salts.

The lack of identification of double salts thus far on Mars initially appears to indicate broadly colder temperatures of formation for martian materials. However, the lack of clear evidence for double salts may have another explanation. Warm-temperature transformation to double salts appears to be most common in the efflorescent crusts, rather than in pools or interstitial muds, in the Basque Lakes. Efflorescent crusts likely have the lowest long-term preservation potential of materials in and around lakes, as they can very easily dissolve with rainfall or snowfall. This makes them excellent short-term climate indicators but less useful on geologic timescales. Any double salts forming adjacent to ancient martian lakes undergoing climatic transformations may not be extensively preserved, whereas saline fluids infiltrating into sediment perhaps through groundwater—rather than wicking up through sediment—might have higher preservation potential, but may produce fewer double salts based on our observations from the Basque Lakes.

Via remote sensing techniques, the most useful indicator of paleoclimate for salts forming in pools (as opposed to crusts and muds) might therefore be the formation of Na-sulfates from a freezing fluid. Salts within Basque Lake pools—which have higher preservation potential than efflorescent crusts—do change seasonally, primarily via the appearance of meridianiite and/or mirabilite, and a metastable Na-sulfate heptahydrate, in the wintertime, and the loss of such materials through the fall. Given these observations we might expect that on Mars, if such materials are deposited and cemented rapidly, then Na-sulfate formation would be responsive to climatic variables on relatively short timescales (e.g., Figure 19). Na-sulfates are infrequently observed on Mars, however, and have never been definitively found via mineralogical techniques, only inferred via chemical techniques (e.g., B. C. Clark, 2023; Gasda et al., 2022). This implies possibly limited low-temperature/freezing fluid processes forming sulfates, consistent with the low Mg-sulfate hydration states identified via Mars rover instrumentation. Investigating evidence for hydrated Na- and Mg-sulfates that may have altered, coupled with other sedimentological evidence, will verify the presence of ancient freezing fluids on Mars. If such evidence remains scant, this may imply broadly warmer-climate or warmer-fluid deposition of salts on Mars.

6.4.3. Gypsum Detectability in High-Mg Systems

Gypsum has been identified extensively on Mars via remote observations (e.g., Das et al., 2022; Fishbaugh et al., 2007; Wray et al., 2009) and in situ observations (Nachon et al., 2014; Vaniman et al., 2018, 2024). In Gale crater, recent observations suggest Ca-sulfate is present extensively within the Mg-sulfate-bearing unit (Gasnault et al., 2024; Pandey et al., 2025), indicating gypsum formation in a Mg-rich environment at Gale crater. In Jezero crater, intimate mixtures of Mg- and Ca-sulfates have also been confirmed (Bosak et al., 2024; Corpolongo et al., 2023; Phua et al., 2024; Siljeström et al., 2024). The Basque Lakes are therefore highly relevant to Mars with respect to coupled Ca-sulfate and Mg-sulfate formation and preservation. We observe clear crystalline gypsum within the ridge-forming muds and muds at the edges of the Basque Lakes. Our identifications of gypsum in the Basque Lakes via Raman and NIR are definitive, even with low XRF-modeled abundances of Ca and S. This indicates that gypsum is highly NIR and Raman active, even in mixed materials, as has also been established in previous work (Hughes et al., 2023). Gypsum in intimate mixtures and possibly bedrock deposits therefore may be highly Raman active on Mars, and should be easily detectable via the SuperCam and SHERLOC (Scanning Habitable Environments with Raman and Luminescence for Organics and Chemicals) Raman instruments, even at low mass fractions.

At our field site, this gypsum is also commonly associated with epsomite in individual Raman spectra, suggesting gypsum and Mg-sulfates mix intimately on the sub-mm scale. While any coatings or adsorbed materials might have lower preservation potential than crystalline materials, it's possible that in high Mg systems, Mg- and Ca-sulfates can be highly mixed. This suggests that gypsum forming in high Mg environments on Mars may show some association between Mg and Ca. We suggest that any gypsum identified using chemical techniques on Mars be investigated for such linkages, to determine whether the fluid depositing gypsum might have been high in Mg²⁺. If gypsum and Mg-sulfates are not closely linked, this might suggest gypsum-unique fluid events, or time-steps between gypsum deposition and any Mg-sulfate deposition for an evolving fluid.

6.4.4. Evaluation of Intimate Salt Mixtures on Mars

Raman and NIR data revealed, on the whole, complementary but firmly different sets of saline minerals in the Basque Lakes. NIR data indicated the presence of double salts that appear in the fall but transform in the winter, via the presence in the fall of absorption features between 1.65 and 1.68 μm . Detection of double salts in Raman data, however, was limited to a possible bloedite detection from a winter brine and a konyaite detection in a fall crust. In high Mg-sulfate systems, Raman therefore might be less robust at evaluating the formation of double salts, as Raman signal appears to be swamped by mono-cation sulfates. It's also likely that the much smaller spot size of the Raman instrument reveals different salts than that of NIR analysis, which can detect intimately mixed salts on a mm- and cm-scale. Yet in spite of a larger spot size, NIR spectroscopy was insufficient for evaluating the presence of mixed epsomite-gypsum deposits in muds and crusts, which Raman analyses captured quite firmly. Likewise, NIR spectra were often swamped by hydration features when we targeted salts that formed in brine pools, while Raman was well-equipped to characterize these materials. We therefore recommend coupled analysis using both instrument techniques for the characterization of mixed salt assemblages on Mars. Ideally, such analyses would also be paired with geochemical analyses, in order to determine, for example, whether any Ca-sulfates also have high associated Mg in an intimate mix. The SuperCam instrument suite on board Perseverance therefore offers an ideal set of analyses for any mixed Mars salt assemblages.

7. Conclusions

We investigated the mineralogical and geochemical variability of the Basque Lakes in British Columbia, Canada, using Mars-relevant instrumentation, to constrain the influence of environment on mineral formation. The Basque Lakes represent a metastable system, with sulfates of variable hydration states in disequilibrium phases present simultaneously, in both the winter and the fall. Such metastability is likely a function of ongoing transformations with annual and seasonal environmental changes. The clearest evidence for seasonal change is observed via near-infrared spectroscopy of efflorescent crusts, which have high mineral diversity in both seasons, but show formation of double salts in the fall that firmly disappear by the end of winter. Efflorescent salts, however, likely have low preservation potential, and double salts are not as clearly nor extensively forming in brine pools and muds, which have higher preservation potential. Therefore, the most robust climate indicators with the highest preservation potential might be hydrated Na- and Mg-sulfates forming within brine pools in the winter that are absent in the fall. Raman spectroscopy is suited to evaluate the hydration state of such Na-sulfates. On Mars, the lack of identification of Mg-sulfate double salts may be due to loss or alteration of these materials over long timescales, considering they appear more likely to form as transient crusts than stable crystalline salts. Meanwhile, any Mg-sulfates found co-occurring in different hydration states on Mars need not have formed through long-term transformations, but might have formed concurrently in a metastable environment, though we note that the long geologic timescales of Mars would likely influence the long-term hydration of Mg-sulfates. Finally, if gypsum is intimately associated with high amounts of Mg-sulfate, this might indicate such gypsum formed rapidly from a Mg-rich brine, while gypsum without associated Mg might have formed independently from Mg-sulfates or over longer brine evaporation timescales. These results have implications for sulfates identified by the SHERLOC and SuperCam instruments in Jezero Crater, and the Mg- and Ca-sulfates identified by the Curiosity rover in Gale crater. In Gale crater, searching for evidence for mineral water loss or alteration (e.g., crystal molds, pseudomorphs, or dewatering structures) may illuminate whether salts experienced fluctuating temperature and/or atmospheric conditions. Salts formed from fluids largely disconnected from such fluctuations (e.g., a relatively deep groundwater table) may show minimal evidence for mineralogical change. In Jezero crater, intimately mixed Mg-sulfates of variable hydration states might have formed from surface fluids connected to a fluctuating atmosphere. Band analysis with SuperCam NIR spectra may be helpful for evaluating the presence or absence of double salts, though we predict few double salts may be identified due to low preservation potential. We confirm a combination of Raman, NIR and chemical analyses provide a valuable suite of tools for evaluating saline mineralogy on Earth and Mars.

Conflict of Interest

The authors declare no conflicts of interest relevant to this study.

Data Availability Statement

All data acquired for this work (Raman, NIR and XRF derived data) are available in the main text, supplement, and the data repository (Hughes et al., 2025).

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