

1 **Partially-Saturated Brines Within Basal Ice or Sediments can Explain the Bright Basal**
2 **Reflections in the South Polar Layered Deposits**

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20 Key points:

- 21 • Brines within ice and icy sediments can explain the bright basal reflection in the south polar
22 layered deposits
- 23 • Salt enhancements of 5–16 times the salt-regolith concentration at the Phoenix landing site is
24 needed in brine-sediment mixtures
- 25 • Calcium perchlorate brines are stable above their eutectic temperature, which is
26 experimentally measured at 197.3 ± 0.2 K

27 **Abstract**

28 Strong radar reflections have been previously mapped at the base of the Martian South Polar
29 Layered Deposits (SPLD). Here, we analyze laboratory measurements of dry and briny samples to
30 determine the cause of this radar return. We find that liquid vein networks consisting of brines at
31 the grain boundaries of ice crystals can greatly enhance the electrical conductivity, thereby causing
32 strong radar reflection. A liquid brine concentration of 2.7–6.0 vol% in ice is sufficient to match
33 the electrical properties of the basal reflection as observed by MARSIS. When brine is mixed with
34 sediments, we find that the brine-ice mixture in the pores must be 2–5 times more concentrated in
35 salt, increasing the brine concentration to 6.3–29 vol%. Thus, our best fit of the median observed
36 MARSIS value suggests a salt-bulk sample concentration of ~6 wt%, which is ~8 larger than that
37 of the Phoenix landing site. To form brine, the basal reflector must reach a temperature greater
38 than the eutectic temperature of calcium perchlorate of 197.3 ± 0.2 K. Colder metastable brines are
39 possible, but it is unclear if brines can remain metastable for millions of years. Additionally, grey
40 hematite with a concentration of 33.2–59.0 vol% possess electrical properties that could cause the
41 observed radar returns. However, such concentrations are 2–3 times larger than anywhere
42 currently mapped on Mars. We also demonstrate that brines mixed with high-surface-area
43 sediments, or dry red hematite, jarosite, and ilmenite cannot create the observed radar returns at
44 low temperatures.

45

46 **Plain-Speak Abstract**

47 Previously research has shown that strong radar reflections emanate from the interface between
48 Mars' southern ice cap and their underlying sediments over a region with an area of 20×30 km and
49 1.5 kms beneath the surface. Radar reflections are caused by changes in electrical properties. Here,
50 we analyze electrical property laboratory measurements of materials under Mars-like conditions.
51 We find that a small amount (~3–6%) of brine in ice samples could create strong radar reflections
52 similar to those that are observed. A greater concentration of salt is needed in sediment ice
53 mixtures, such that a salt enhancement of ~10 would be needed compared to the rest of Mars.
54 Additionally, a calcium perchlorate brine could only form if the temperature was greater than 197.3
55 K. Colder metastable brines are possible, but it is unclear if brines can remain metastable for
56 millions of years. Additionally, dry grey hematite could also cause the observed radar returns, but
57 the concentration of grey hematite would have to be 2–3 times larger than anywhere else on Mars.

58 We can also rule out any brines mixed with clays, or dry red hematite, jarosite, and ilmenite as
59 they cannot create the observed radar returns at low temperature.

60

61 1. Introduction

62 The Mars Advanced Radar for Subsurface and Ionospheric Sounding (MARSIS) instrument
63 on Mars Express has detected strong subsurface radar reflections in the region of Ultimi Scopuli
64 (81°S, 193°E), within the South Polar Layered Deposits (SPLD) (*Orosei et al.*, 2018; *Lauro et al.*,
65 2021). These subsurface reflections are ~10 dB greater in power than the surrounding reflections
66 and ~3 dB greater than the reflections from the surface (*Orosei et al.*, 2018). The reflecting unit is
67 located at the base of the SPLD, approximately 1.5 km below the topographic surface. Because
68 data acquired by MARSIS do not separate the real (ϵ') and imaginary (ϵ'') parts of the complex
69 permittivity of reflectors, the apparent permittivity (ϵ_a), a single parameter accounting for both ϵ'
70 and ϵ'' (*Mattei et al.*, 2022a; ref. to §2.2) is commonly reported. In their investigation of the SPLD,
71 *Orosei et al.* (2018) obtained reflectivities characterized by a median ϵ_a value of 33 ± 1 at 4 MHz.
72 Using a threshold value of $\epsilon_a = 15$, the largest reflective zone is ~20×30 km (*Lauro et al.*, 2021).
73 Furthermore, *Lauro et al.* (2021) also detected at least three other smaller locations, about 10 km
74 across, within 120 km of the largest anomaly.

75 The high reflectivity values observed by MARSIS (*Orosei et al.*, 2018) were found to have a
76 1st and 3rd quartile values ϵ_a of 16 and 91, respectfully. Such high values of ϵ_a significantly limit
77 the number materials that could cause such reflections. Three categories of materials have thus far
78 been proposed to explain the high reflectivity values: clays with adsorbed water (*Smith et al.*,
79 2021); dry minerals and rocks with high iron content (*Bierson et al.*, 2021; *Grima et al.*, 2022);
80 and saline ice (*Bierson et al.*, 2021).

81 *Smith et al.* (2021) suggested that the dielectric relaxation of clay with adsorbed water at the
82 base of the SPLD could result in high ϵ_a values at the presumed basal temperature in Ultimi
83 Scopuli. However, *Smith et al.*'s (2021) data were measured at 230 K and are inconsistent with
84 similar types of measurements as reported in the literature (*Moore and Maeno*, 1993; *Stillman et*
85 *al.*, 2010; *Stillman and Grimm*, 2011b; *Lorek and Wagner*, 2013; *Cunje et al.*, 2018; *Kulacz and*
86 *Orzechowski*, 2019; *Mattei et al.*, 2022a).

87 *Bierson et al.* (2021) suggested that red hematite is a possible source for the bright reflection,
88 because it has an $\epsilon' = 18.1$ (*Robinson and Friedman*, 2003). In fact, *Robinson and Friedman*
89 (2003) found that a soil sample of red hematite with 50.5% porosity, likely a reasonable analog for
90 Martian soils, has a bulk $\epsilon' = 4.5$, which is consistent with measurements reported by *Stillman and*

91 *Olhoeft* (2008). The $\epsilon' = 18.1$ used by *Bierson et al.* (2021) was the estimate of the grain or solid
92 permittivity, which assumes no porosity. Assuming minimal porosity of 10% for a hematite-rich
93 rock, using *Robinson and Friedman's* (2003) mixing models we estimate a bulk ϵ' value of 10.8,
94 which is lower than the observed 1st quartile value. As an alternative, the same authors also
95 suggested jarosite as a possible material, based on its proposed high value of ϵ' . This is however
96 inconsistent with dry soil measurements of permittivity for this mineral, as published in the
97 literature (ref. to figure 1 in *Stillman and Olhoeft*, 2008).

98 *Grima et al.* (2022) suggested that measurements of bulk $\epsilon' > 16$ have been reported for ilmenite
99 (FeTiO_4) and basalts (*Parkamento*, 1967; *Shmulevich et al.*, 1971; *Rust et al.*, 1999). We addressed
100 this specific interpretation later on in this paper, where we present and discuss the results of our
101 measurements on dry ilmenite.

102 *Bierson et al.* (2021) suggested that the conductivity of saline ice, i.e., ice with a bulk salt mass
103 concentration range of 0.1–3.5 wt%, could return ϵ_a values observed at Ultimi Scopuli. Liquid
104 brines have high ϵ' (~80) and are highly conductive; thus, brines in ice or sediments can plausibly
105 create ϵ_a values within range of those observed by MARSIS (*Mattei et al.*, 2022a).

106 Here, we report on our experimental investigation of the compositional and thermal range of
107 stability of Martian-consistent brines, which is the principal purpose of this study. We also address
108 the dry material interpretation through our laboratory experiments on selected dry materials. The
109 paper is structured as follows: (i) we briefly provide a background into the MARSIS instrument,
110 electrical properties of materials, formation of brines, and estimated basal temperatures in Ultimi
111 Scopuli (§2); (ii) we then present our measurement methodology (§3); (iii) we follow with a
112 discussion of our results (§4); (iv) we then proceed to interpret our results in the context of
113 published hypotheses on the nature of the reflectors (§5); and (v) our principal findings are
114 summarized in the conclusions (§6).

115

116 **2. Background**

117 *2.1 MARSIS*

118 MARSIS acquires data on four channels, with center frequencies of 1.8, 3, 4, and 5 MHz, each
119 with a bandwidth of 1 MHz (e.g., *Picardi et al.*, 2005). The MARSIS dataset used by *Orosei et al.*
120 (2018) and *Lauro et al.* (2021) was acquired without onboard synthetic-aperture radar processing
121 to ensure the amplitudes were as accurate as possible (*Lauro et al.*, 2021). These MARSIS results

122 rely on the 4 MHz center frequency data as it is the most complete data set around the Ultimi
 123 Scopuli region (*Lauro et al.*, 2021). The radius of the first Fresnel zone is ~3.5-6 km depending
 124 on satellite altitude (300-800 km) (*Orosei et al.*, 2018), while the more conservative pulse limited
 125 criterion, has a diameter of 7.5 km assuming a satellite altitude of 400 km (*Lauro et al.*, 2021).
 126 Thus, the anomalous areas are larger than the pulse limited criterion. Lastly, the vertical resolution
 127 is ~55 m, after range compression and Hanning windowing when assuming an $\epsilon' = 3.1$ of pure ice
 128 (*Lauro et al.*, 2021).

129

130 2.2 Electrical Properties

131 The electrical properties of the material control the speed at which radar waves propagate, the
 132 amount of energy that is attenuated, and how much energy is reflected at discontinuities. Here and
 133 in the following discussion, we ignore the effects of the magnetic permeability, given its small
 134 variability (*Stillman and Olhoeft*, 2008). The real part of the relative dielectric permittivity (ϵ')
 135 primarily controls the speed at which radar waves propagate through the material. In most dry
 136 rocks at radar frequencies, ϵ' can be estimated using the following equation:

$$137 \quad \epsilon' = (1.93 \pm 0.17)^\rho, \quad (\text{Eq. 1})$$

138 where ρ is the bulk density of the rock or soil in g cm^{-3} (*Olhoeft and Strangway*, 1975). The
 139 imaginary part of the relative dielectric permittivity (ϵ'') primarily controls the attenuation of radar
 140 waves through the material and is a summation of losses due to dielectric relaxations (ϵ''_p) and
 141 Direct Current (DC) electrical conductivity (σ_{DC}),

$$142 \quad \epsilon'' = \epsilon''_p + \frac{\sigma_{DC}}{2\pi f \epsilon_0}, \quad (\text{Eq. 2})$$

143 where f is the frequency of the radar energy and ϵ_0 is the dielectric permittivity in vacuum (8.854
 144 $\times 10^{-12} \text{ F m}^{-1}$).

145 Next, we convert ϵ' and ϵ'' into ϵ_a (*Mattei et al.*, 2022a) using

$$146 \quad \epsilon_a = \epsilon_1 \frac{\epsilon_1 + |\epsilon_2| + \sqrt{\epsilon_1^2 + |\epsilon_2|^2 - 2\epsilon_1\epsilon_2'}}{\epsilon_1 + |\epsilon_2| - \sqrt{\epsilon_1^2 + |\epsilon_2|^2 - 2\epsilon_1\epsilon_2'}}, \quad (\text{Eq. 3})$$

147 where ϵ_1 is the ϵ' of the SPLD assumed to be 3.5, ϵ'' of the SPLD is assumed to be zero, and
 148 magnitude of the permittivity of the basal reflector is

$$149 \quad |\epsilon_2| = \sqrt{\epsilon_2'^2 + \epsilon_2''^2}. \quad (\text{Eq. 4})$$

150 In **Figure 1**, the combination of the reflector's ε'_2 and ε''_2 that would create ε_a similar to that
151 observed in the MARSIS data at 4 MHz (Lauro *et al.*, 2021) is shown: minimum values of ε'_2 and
152 ε''_2 of 13.5 and 6, respectively, could allow for an $\varepsilon_a \geq 16$, i.e., the observed 1st quartile value. If
153 both the real and imaginary part values of a material are less than these minimum values, then ε_a
154 cannot reach the MARSIS threshold values observed.

155 Electrical properties are frequency dependent because different polarization mechanisms
156 separate charges at different speeds (e.g., Stillman and Olhoeft, 2008; Stillman *et al.*, 2010). To
157 model the material's frequency dependence, we use the Cole-Cole equation (Cole and Cole, 1941,
158 Stillman and Olhoeft, 2008). Frequency dependent, materials are also temperature dependent,
159 because the speed at which charge separates is temperature dependent: this behavior is modeled
160 with a Boltzmann temperature dependence (e.g., Stillman and Olhoeft, 2008, and refs. therein).
161 These models allow us to extrapolate and interpolate electrical properties in frequency and
162 temperature. Thus, ε' and ε'' measured to 1 MHz can be extrapolated to comprise the MARSIS
163 frequency range of 2 – 5 MHz (e.g. Stillman and Grimm, 2011a). Additionally, as the frequency
164 dependence shifts to lower frequency at colder temperatures, we can test whether the extrapolation
165 is valid (Stillman and Grimm, 2011a).

166

167 2.3 Formation of Brines

168 Salt-H₂O mixtures are characterized by four distinct phases (**Fig 2A**):

- 169 (1) Brine: when the temperature of the mixture is above the melting temperature;
- 170 (2) Brine + ice: when the temperature of a sub-eutectic concentration (<49.8 wt% for
171 Ca(ClO₄)₂) is below the melting temperature, but above the eutectic temperature;
- 172 (3) Brine + hydrate: when the temperature of a super-eutectic concentration is below the
173 melting temperature, but above the eutectic temperature; and
- 174 (4) Ice + hydrate: when the temperature of a mixture is below the eutectic temperature.

175 Here, we focus on the brine + ice phase for sub-eutectic concentrations. As a sub-eutectic
176 concentration (<49.8 wt% for Ca(ClO₄)₂) sample begins to freeze below the melting point, H₂O is
177 fractionated out of the system as pure ice (or nearly pure ice if Cl⁻, F⁻, or NH₄⁺; for more see Gross
178 *et al.*, 1977; Petrenko and Whitworth, 1999; Stillman *et al.*, 2013ab) and the salt-H₂O mass
179 concentration increases in the liquid brine until it reaches a eutectic concentration at the eutectic
180 temperature. The amount of liquid brine present at the eutectic depends on its initial concentration

181 and salt type (**Fig. 2**). For very low salt concentrations in the mixture, the melt accumulates at the
182 boundaries of the ice grains. Even low salinity brines (e.g., ~3 mM, ~0.03 mass%, or ~0.06 vol%;
183 *Grimm et al.*, 2008) are electrically conductive, they form electrically connected networks across
184 the ice matrix, defined as along Liquid Vein Networks (LVNs). Thus, salt-H₂O mixtures display
185 high electrical conductivity even when most of the ice is frozen (*Grimm et al.*, 2008; *Stillman et*
186 *al.*, 2010). The electrical conductivity of the mixture drops significantly below the eutectic
187 temperature, when the entire system is frozen.

188

189 2.4 Estimated Basal SPLD Temperature

190 Estimates of the values of SPLD basal temperatures in Ultimi Scopuli have been recently
191 published (*Sori and Bramson*, 2019; *Ojha et al.*, 2019; *Egea-González et al.*, 2022), and are heavily
192 dependent upon the heat flow from the planetary interior, and the thermal conductivity of the
193 SPLD, both of which are not fully constrained. Assuming a temperature-dependent thermal
194 conductivity of pure H₂O ice for the SPLD, a heat flow >84 mW/m² is required to allow liquid Ca-
195 perchlorate brine to form. For a more realistic heat flow range of 14-25 mW m⁻² (*Parro et al.*,
196 2017), a basal temperature of ~171-176 K is obtained (*Sori and Bramson*, 2019). *Egea-González*
197 *et al.* (2022) found a basal temperature range of 175-187 K using the maximum surface heat flow
198 (32 mW m⁻²) by assuming a dust proportion of 15%. We speculate that inclusion of CO₂ ice into
199 H₂O ice layers or as pure CO₂ layers within the SPLD could further enhance basal temperatures
200 (*Wiezorek*, 2008), as CO₂ ice has a thermal conductivity that is 5-6 times lower than that of H₂O
201 ice (e.g., *Mellon*, 1996).

202

203 3. Methods

204 This section describes the methodology of electrical property measurements of salt-H₂O
205 mixtures and salt-H₂O mixtures with Martian analogs. Note, new measurements are complemented
206 by data obtained in previous published (*Grimm et al.*, 2008; *Stillman et al.*, 2010; *Stillman and*
207 *Grimm*, 2011ab) and unpublished work. Low-frequency measurements were conducted at
208 Southwest Research Institute in Boulder Colorado. Solutions of salt-H₂O mixtures were freshly
209 prepared before each measurement. For measurements of salt-H₂O mixtures, the liquid solution
210 was poured into the three-electrode sample holder with a Teflon cup (Solartron 12962A and
211 12964A). For measurements of salt-H₂O mixtures with Martian analogs, the analogs (e.g., clays,

212 sands, ilmenite) were dried in a vacuum at ~ 1 mbar and ~ 383 K until their mass changed by less
213 than 0.5% over a ~ 12 -hour period. The granular analog material was then spooned into the Teflon
214 cup sample holder with a spatula, then the liquid solution was poured into the granular material.
215 Lastly, the sample was intimately mixed with a spatula to ensure a homogenous sample was
216 created. The sample was 3.5 cm in diameter and ~ 0.6 cm thick. The sample holder was then placed
217 into the sample chamber.

218 The sample chamber has been previously described (*Grimm et al.*, 2008; *Stillman et al.*, 2010).
219 For measurements made after Sept 2019, we used a custom vacuum chamber that is placed in the
220 Ultra-Low freezer (So-Low C85-9). Only dry materials were measured in vacuum of ~ 1 mbar,
221 samples with ice were measured at atmospheric pressure. The sample holder sits on a liquid
222 nitrogen cold plate so that temperatures below 180 K can be obtained. Lake Shore Cryotronics
223 Silicon diode (DT-670) temperature sensors are used to measure the sample temperature on the
224 Teflon cup and a second sensor controls two 25 W cartridge heaters (Lake Shore HTR-25-100)
225 via a Lake Shore Cryotronics temperature controller (331). The sample is then connected via BNC
226 cables to a Solartron 1260A impedance analyzer and a Solartron 1296A dielectric interface. The
227 sample temperature was then lowered to reach a temperature that is 2 K below its freezing point
228 for 1 hour to initialize crystallization. Subsequently, the temperature is further decreased to 10 K
229 below the freezing point for an additional 2 hours to ensure crystallization of any metastable water.
230 Reheating of the sample is carried out by raising the temperature back to 2 K below the freezing
231 point for 3 hours. This process guarantees formation of stable LVNs for the entire duration of the
232 experiment, reducing the rate of cracking in the sample, and avoiding the effect of rapid
233 recrystallization. The temperature of the sample is then drastically lowered at a rate that is lower or
234 equal to < 1 K min^{-1} to the sample's lowest temperature. Electrical property measurements are
235 conducted non-stop as soon as the sample obtains properties that allow it to be measured easily,
236 typically this occurs when $\sigma_{DC} < 0.1$ S/m. After obtaining the lowest temperature value the sample
237 is slowly heated. To increase temperature accuracy, the target temperature is held to within 0.2 K
238 for at least 20 min. Typically, measurements are made over the range of $1-10^6$ Hz, which takes ~ 6
239 minutes to acquire. However, as σ_{DC} increases the lower frequency limit is also increased, which
240 reduces the time required to measure a spectrum. Additionally, once the sample reaches the
241 frequency at which σ_{DC} dominates the electrical properties the uncertainty of ϵ' increases greatly.

242 The electrical property measurements output complex impedance as a function of frequency,
243 which is converted into complex dielectric permittivity using the electrode geometry. Cole-Cole
244 parameters of each spectrum were modeled by nonlinear curve fitting (*Stillman and Olhoeft, 2008*;
245 *Grimm et al., 2008*; *Stillman and Grimm, 2011ab*). The Cole-Cole parameters were then used to
246 find ϵ_a at 4 MHz as a function of temperature.

247

248 **4. Results and Interpretations**

249 *4.1 Dry Ilmenite*

250 Dry granular ilmenite (purchased from Ward's Scientific) with 35.5% porosity and a bulk
251 density of 3.07 g cm^{-3} possesses a significant σ_{DC} and a polarization mechanism that increases ϵ'
252 and ϵ'' at MARSIS frequencies (**Fig. 3**). The observed relaxation mechanism is known as an
253 anomalous low-frequency dispersion (e.g., *Jonscher 1978*; *1999*; *Shahihi et al., 1975*; *Stillman et*
254 *al., 2010*). Previously reported data indicate that ilmenite reaches a value of $\epsilon' > 33$ at room
255 temperature (*Parkmenko, 1967*). In our experiments, at $\sim 273 \text{ K}$, the ϵ_a of ilmenite does not
256 approach the observed median, dropping below the observed 1st quartile value at a temperature of
257 $\sim 252.8 \text{ K}$. Therefore, we conclude that ilmenite is not a plausible candidate as the cause of the
258 strong basal reflections at Ultimi Scopuli.

259

260 *4.2 Chloride Salt-H₂O Mixtures*

261 Chloride brines in ice were previously shown to obtain high values of σ_{DC} and ϵ'' at
262 temperatures above their eutectic temperatures (*Grimm et al., 2008*). However, NaCl (*Grimm et*
263 *al., 2008*) and MgCl₂ (*Primm et al., 2020*) have eutectic temperatures of 251 and 240 K,
264 respectively; thus, they are too high to be considered in the context of the SPLD. CaCl₂ has a much
265 lower eutectic temperature of 223 K (*Stillman et al., 2010*). The ϵ_a data versus temperature and
266 concentration show that a 100 mM (1.1 wt%) of CaCl₂ at the eutectic temperature has an ϵ_a
267 equivalent to the observed 1st quartile value (**Fig. 4**). The ϵ_a of the 1,000 mM (10 wt%) of CaCl₂
268 is larger than the observed 3rd quartile value (**Fig. 4**), indicating a lower concentration is need to
269 match the 3rd quartile value.

270

271

272

273 4.3 Perchlorate Salt-H₂O Mixtures

274 Because of their low eutectic temperatures of 198 and 216 K (Toner *et al.*, 2014) respectively,
275 calcium and magnesium perchlorate brines have been proposed as most plausible source for the
276 bright basal reflections in Ultimi Scopuli (Lauro *et al.*, 2021). The ϵ_a versus temperature and
277 concentration show that a concentration of 100 mM (2.2 wt%) of Mg(ClO₄)₂ is just below the
278 observed 1st quartile value (Fig. 5), while 500 mM (10 wt%) of Mg(ClO₄)₂ is greater than the
279 observed 3rd quartile value (Fig. 5).

280 For the 300 mM (6.9 wt%) of Ca(ClO₄)₂, the selected measurements of ϵ' (Fig. 6A) and ϵ''
281 (Fig. 6B) display a strong ice relaxation below the eutectic temperature. Above the eutectic
282 temperature, σ_{DC} increases to large values (Fig. 6C) so that ϵ' can no longer be measured accurately
283 due to so much energy being dissipated by ϵ'' . The observed dependence of ϵ_a with temperature
284 demonstrates that concentrations of 300 mM (6.9 wt%) of Ca(ClO₄)₂ are sufficient to obtain the
285 observed 3rd quartile value at the measured eutectic temperature of 197.3±0.2 K (Fig. 7). The brine
286 in the 200 mM (4.7 wt%) of Ca(ClO₄)₂ never frozen into hydrate and ice even though the
287 temperature was dropped to 161.9 K. As will be shown in the next section and Figure 8, ϵ_a has the
288 same value at a given temperature when above the eutectic temperature no matter whether the
289 temperature is warming or cooling. Thus, ϵ_a at the eutectic temperature for the 200 mM Ca(ClO₄)₂
290 sample shows it is near the median observed value, making this a likely material for the base of
291 the SPLD in Ultimi Scopuli. Meanwhile, the 100 mM (2.4 wt%) Ca(ClO₄)₂ sample shows it is
292 below the 3rd quartile value.

293

294 4.4 Metastability of Salts-H₂O Mixtures

295 The results of the experiments conducted to explore the metastability of perchlorate brines are
296 shown in Figure 8. For the 300 mM (6.7 wt%) Ca(ClO₄)₂, the brine remained in a metastable form
297 down to a temperature of 187.9±0.2 K, i.e., 9.4 K below the eutectic temperature, although the
298 measured value of ϵ_a is below the observed 1st quartile. The decreasing value of ϵ_a with decreasing
299 temperature is attributed to a decreasing volume of the liquid brine combined. Thus we conclude
300 that, to record values of apparent permittivity consistent with the bright MARSIS reflections,
301 metastable brines must form from concentrations above that in this experiment. We were able to
302 hold the brine sample in a metastable form for 6.9 hours before decreasing the temperature to a
303 lower temperature. Metastability of other salts has been previously reported to last at least 72 hours

304 when held at a temperature a few degrees below the eutectic temperature (*Primm et al.*, 2020).
305 Other $\text{Ca}(\text{ClO}_4)_2$ samples displayed in **Figures 7 & 9** never froze even though they were taken to
306 temperatures below 173 K.

307

308 *4.5 Salt-H₂O Mixtures in Low-Surface Area Sediments*

309 Previous measurements (*Stillman et al.*, 2010) have shown that brines in low-specific-surface
310 ($<1 \text{ m}^2/\text{g}$) area sediments also show distinct increases of σ_{DC} at the eutectic temperature that
311 indicate that LVNs still dominate conduction in icy sediment mixtures. Our experiments were
312 conducted with fine-grained (mean grain size of 110 μm) sand with a porosity of $\sim 40\%$. We
313 focused on $\text{Ca}(\text{ClO}_4)_2$ salt-H₂O mixture with sand. We found that a 37.8 vol% of 700 mM (15.1
314 wt%) $\text{Ca}(\text{ClO}_4)_2$ mixed with sand is near the observed ε_a values for the 1st quartile (**Fig. 9**).
315 Similarly, a 40.4 vol% of 1.5 mM (29.1 wt%) $\text{Ca}(\text{ClO}_4)_2$ mixed with sand is near the observed ε_a
316 values for the median observed values (**Fig. 9**), making this a likely material for the base of the
317 SPLD in Ultimi Scopuli.

318

319 *4.6 Salt-H₂O Mixtures in High-Surface Area Sediments*

320 Clay samples from the Clay Mineral Society were previously measured at Mars-like
321 temperatures (*Stillman et al.*, 2010; *Stillman and Grimm*, 2011ab). A compilation of multiple
322 samples of STx-1 (Texas Calcium Montmorillonite) measured dry and with slight additions of
323 water at a temperature of $193.2 \pm 0.2 \text{ K}$ are shown in **Figure 10**. STx-1 possesses a large H₂O
324 surface area of $217 \text{ m}^2/\text{g}$ (*Jänchen et al.*, 2009), where 3 and 7 monolayers (ML) represent ~ 14
325 and $\sim 32 \text{ wt}\%$ water, respectively. (Note a ML of water was assumed to be 0.3 nm thick.) None of
326 the data plotted approach the ε_a threshold for the 1st quartile observations. Furthermore, the full
327 temperature dataset for 7 ML of 100 mM CaCl_2 indicates that, once the temperature drops below
328 233 K, the electrical properties are not compatible with the 1st quartile MARSIS observations (**Fig.**
329 **11**).

330 For this work, we measured clay specimen SAz-1 (“Cheto” from Arizona with an N_2 surface
331 area of $97.4 \text{ m}^2/\text{g}$) saturated with 54.2 wt% of 500 mM $\text{Ca}(\text{ClO}_4)_2$ brine (**Fig. 12**). While the sample
332 displays σ_{DC} and strong polarization mechanisms, these are not of sufficient magnitude to approach
333 the range of ε_a values observed by MARSIS. Thus, our new experiment corroborates the
334 conclusion that salt-H₂O mixtures in high-surface area sediments do have increased values of σ_{DC}

335 above the eutectic temperature (*Stillman et al.*, 2010; *Mattei et al.*, 2022ab). While absorbed/bound
336 water does conduct electrical current below and above the eutectic temperature, the ions in
337 adsorbed/bound water are less mobile and follow a much more tortuous path compared to brines
338 in sand or ice (*Stillman et al.*, 2010; 2019).

339 JSC Mars-1, which consists of hematite and titano-magnetite, has been measured by many
340 researchers (e.g., *Williams and Greeley*, 2004; *Stillman and Olhoeft*, 2008). When measured dry,
341 it does not approach the 1st quartile values. JSC Mars-1 has a large H₂O surface area (106 m²/g;
342 *Pommerol et al.*, 2009) due to a palagonitic surface. However, even with 7 ML (~18 wt%) it still
343 does not obtain large electrical property values approaching the range of ϵ_a values of that observed
344 by MARSIS until temperatures larger than 273 K (**Fig. 13**). In conclusion, salt-H₂O mixtures in
345 high-surface area (>1 m²/g) sediments do not possess a large enough σ_{DC} to provide an ϵ_a value at
346 SPLD-like temperatures that is similar to the bright reflector measured by MARSIS.

347

348 **5. Discussion**

349 *5.1 Dry Minerals*

350 A discussion on red hematite and jarosite is not necessary as previous measurements in the
351 literature can eliminate these minerals, as was discussed in §2.5. While dry ilmenite can have very
352 high ϵ_a values at room temperature (*Parkmenko*, 1967; *Boivin et al.*, under review), temperature
353 dependence significantly lowers ϵ_a values (**Fig. 3**). Thus, ilmenite has an ϵ_a value near ~12 that is
354 lower than the observed 1st quartile value at the approximate temperature of the base of the SPLD.

355 Intrusive and extrusive igneous rocks were measured by *Shmulevich et al.* (1971) of which 12
356 of 89 had $\epsilon' > 13.5$ at 500 MHz, which could have an $\epsilon_a > 16$ depending on their ϵ'' , which were
357 not published. There is little data regarding the methodology of the *Shmulevich et al.* (1971)
358 measurements. Leaving many important questions: what temperature were the samples measured
359 at, where the samples were acquired, and were the samples vacuum or thermally dried or measured
360 as collected? Additionally, frequency and temperature dependence are also omitted. Thus, it is
361 impossible to extrapolate these measurements to SPLD temperatures and MARSIS frequencies
362 given the lack of details given in *Shmulevich et al.* (1971).

363 Many other measurements (*Campbell and Ulrichs*, 1969; *Chung et al.*, 1970; *Gold et al.*, 1970;
364 *Adams et al.*, 1996; *Russell and Stasiuk*, 1997) of volcanic rocks all measured $\epsilon' < 12$ at radar
365 frequencies. While *Rust et al.* (1999) found two of 34 basaltic samples, with an $\epsilon' > 13.5$ at 4 MHz

366 and at room temperature. Only the MB1 sample has an $\varepsilon_a > 16$ at 4 MHz at room temperature (**Fig.**
367 **S1**). *Rust et al.* (1999) does comments that this sample hosts thin cracks, thus a layer of absorbed
368 water could be increasing ε' as has been measured by many other (e.g., *Jonscher* 1978; 1999;
369 *Shahidi et al.*, 1975; *Knight and Endres*, 1990; *Stillman et al.*, 2010). Thus, we are skeptical that
370 this single basalt measurement could produce an ε_a value due to adsorbed/bound water that is larger
371 than clay or JSC Mars-1.

372 Grey hematite possesses a radio-frequency relaxation with a small activation energy of 0.1 eV
373 (*Stillman and Olhoeft*, 2008). A dry sample of 59.0 vol% grey hematite with a porosity of 41.0%
374 can obtain the median MARSIS ε_a value at 4 MHz over possible SPLD-like temperatures (**Fig.**
375 **14**). This is a significant amount of grey hematite and about four times greater than the maximum
376 grey hematite value measured at the surface of 15% at Aram Chaos and Meridiani Planum (*Glotch*
377 *and Christensen*, 2005). Additionally, the grey hematite in Meridiani Planum is a lag deposit and
378 thus concentrated compared to its *in-situ* formation concentration (*Hynek*, 2004). This is likely
379 why no large increases in surface reflectivity have been measured at Aram Chaos or Meridiani
380 Planum even though they have a high surficial concentration of grey hematite.

381 To further explore the possibilities of grey hematite, we perform dielectric mixing models.
382 Using a Lickenecker power law mixing formula (*Stillman and Olhoeft*, 2008), we estimate the
383 solid grain ε_a of grey hematite to be 137.6 at 4 MHz at 200 K, assuming a grain density δ_G of 5.26
384 g cm^{-3} . We then used *Eq. 1* to estimate an ε_a value of ultramafic and mafic grain assuming a δ_G of
385 3.8 and 3.0 g cm^{-3} , respectively. We assume that any porosity is filled with ice (no brine) with ε_a
386 of 3.15 as ground ice is stable below the SPLD. We then created four mixing models with grey
387 hematite (**Fig. 15; Table 1**) using a power-law mixing model with an exponent of 2.65 (*Shabtaie*
388 *and Bentley*, 1994; *Stillman et al.*, 2010). First, we mix grey hematite with ice that produces grey
389 hematite volume concentrations of 26.8-45.2-81.0% for the observed 1st quartile-median-3rd
390 quartile values. The replacement of the porosity with ice reduces the volume concentration of the
391 median value from 59.0% (**Fig. 15**) to 45.2%, however this is still three times larger than the largest
392 values detected on the surface of Mars. We then assume an ice-filled porosity of 19% and 10%,
393 while mixing the ultramafic grains with grey hematite. This produces smaller grey hematite
394 concentrations of 12.4-35.6-81.0% and 10.0-33.2-78.6% for ice concentrations of 19% and 10%,
395 respectively, for the observed 1st quartile-median-3rd values. These values of grey hematite are
396 only about two times larger than those detected on the surface, however the bulk density δ_B of

397 these mixtures are higher than any typical volcanic rocks measured on Earth (*Olhoeft and Johnson,*
398 1984; *Shmulevich et al., 1971; Kiefer et al., 2012; Rust et al., 1999*). To reduce δ_B , we then mixed
399 a 10% ice with grey hematite and mafic grains to produce grey hematite concentrations of 18.5-
400 39.2-79.8%. Such a reduction in δ_B leads to an increase in grey hematite concentration, and would
401 likely still produce a measurable gravity anomaly. *Li et al. (2012)* displays no gravity anomaly is
402 present at the Ultimi Scopuli site, albeit greater gravity resolution is needed before this could be
403 totally ruled out. Overall, we find that large amounts of grey hematite can create ε_a values that
404 match the MARSIS observations. However, the concentration of grey hematite (median values
405 from 33.2-59.0 vol% and 3rd quartile value from 78.6-81.0 vol%) and extent (20-30 km ellipse) of
406 the deposits needed rival that of terrestrial banded iron formation, such formations are not expected
407 on Mars.

408

409 5.2 Metastability and Premelting

410 Measurements made while cooling brine mixtures can show significant hysteresis as well as
411 long-term (of at least a few days) stability (**Fig. 8**; *Primm et al., 2020*). Unlike the measurements
412 of MgCl_2 (*Primm et al., 2020*), we could not reproduce the metastability of $\text{Ca}(\text{ClO}_4)_2$. One
413 experiment froze as low as 188 K, while many other experiments it did not freeze down to 173 K
414 and in one case down to 163 K. Recall, *Toner et al. (2014)* measured two $\text{Ca}(\text{ClO}_4)_2$ samples,
415 which did not freeze until 153 K. Without freezing no hysteresis was measured. It is far from
416 demonstrated that the longevity of metastable brines shown in laboratory settings extends over
417 geologic timescales. Therefore, below we pursued hypotheses that do not require metastability
418 until future improved temperature models can fully eliminate the possibility of SPLD basal
419 temperatures of 197.3 K.

420 **Figures 5, 6, & 7** show an increase in ε_a and σ_{DC} before the eutectic temperature is reached.
421 This parabolic behavior is known as premelting (e.g., *Rempel et al., 2004; Stillman et al., 2010;*
422 2019; *Rempel, 2012*) and is typically seen in terrestrial permafrost samples as they approach their
423 melting temperature near 273 K (*Grimm and Stillman, 2015*). This premelting effect allows very
424 high salt concentrations to reach the observed MARSIS ε_a values a degree or two below the salt's
425 eutectic temperature.

426

427

428 5.4 Brine Mixtures

429 Partially-saturated brines are capable of increasing ε_a to the observed values, if the temperature
430 is greater than the eutectic temperature of the salt (**Figs 4, 5, & 7**). The significant increase in ε_a is
431 due to the increase in ε'' via σ_{DC} . Our $\text{Ca}(\text{ClO}_4)_2\text{-H}_2\text{O}$ measurements show that 1st and 3rd quartile
432 values are reached at the eutectic temperature with a $\text{Ca}(\text{ClO}_4)_2$ concentration of 140 mM (3.3
433 wt%) and 310 mM (7.1 wt%), respectively (**Fig. 16**). Note, the lower end of this range overlaps
434 with the upper range of saline ice (0.1–3.5 wt%) range, as suggested by *Bierson et al.* (2021). The
435 bulk brine concentrations of these samples at the 1st and 3rd quartile values are 2.7 and 6.0 vol%
436 brine at a eutectic temperature (**Fig. 2**).

437 In salt-H₂O-sediment mixtures, the salt-H₂O just occupies the pore space. Thus, a greater
438 concentration of salt is needed when mixed with sediment to match the MARSIS observations
439 compared to the salt-H₂O mixtures without sediment. At the eutectic temperature, we find that for
440 a mixture that is ~60 vol% sand and 40 vol% $\text{Ca}(\text{ClO}_4)_2\text{-H}_2\text{O}$ the observed ε_a values for the 1st and
441 3rd quartile values would have a $\text{Ca}(\text{ClO}_4)_2$ concentration of 770 mM (16.4 wt%) and 2.5 M (43.4
442 wt%), respectively (**Fig. 16**). We then modeled the amount of brine in the bulk sample to calculate
443 a brine concentration of 6.3 and 28.7 vol% at the 1st and 3rd quartile values, respectively. Thus, we
444 find that the brine concentration in fine sand must be 2-5 times larger than when the brine is just
445 in ice. This occurs because of increased tortuosity of the LVNs, which leads to a lower σ_{DC} . This
446 drop in σ_{DC} is much larger when the pores and pore throats are even smaller. Therefore, high-
447 specific surface area sediments (i.e., clays and JSC Mars-1) possess LVNs, but with high tortuosity
448 and small σ_{DC} (*Stillman et al.*, 2010; 2019; *Stillman and Grimm*, 2011ab; **Figs. 10-13**) Therefore,
449 these salt-H₂O-sediment mixtures cannot obtain the MARSIS observed ε_a values.

450

451 5.5 Salt Enhancement

452 The salt-regolith concentration discovered at the Phoenix landing site was 0.7 mass% (*Hecht*
453 *et al.*, 2009). Our 1st quartile, median, and 3rd quartile salt-H₂O-sediment values have a salt-bulk
454 sample concentration of 3.6, 5.7, and 11.3 mass%, respectively. Thus, the sediment at the basal
455 layer of the SPLD would have to be enhanced by a factor of ~5–16 compared to the Phoenix
456 landing site. Below we discuss four salt enhancement mechanisms:

457 (1) Drainage of an interconnected system of LVNs could drain concentrate brine down through
458 the SPLD. Similar physics occurs within sea ice and has been hypothesized to occur within the

459 Europa ice shell (e.g., *Hesse et al.*, 2022). LVNs could have drained to the base of the SPLD or
460 have drained into regolith/rock sediments below the SPLD.

461 (2) The sublimation of CO₂/H₂O snow from a previous SPLD-like construct would have left
462 deposited salt on the remaining sediment or dust upon which the current SPLD now rests. Similar
463 processes occur when snow with small amount of salt blows into the upper dry valleys in
464 Antarctica. This snow then sublimates away, leaving the sediment highly enriched in salt (*Levy et*
465 *al.*, 2012).

466 (3) The sublimation of ice due to a change in orbital parameters can concentrate the amount of
467 dust and by proxy the amount of salt mixed into the dust. This process has occurred in the SPLD
468 as images at the edge of the SPLD show layers with varying amount of dust (some likely with
469 more than 10%; *Milkovich and Plaut*, 2008). Note that ~80% of the ice would have to sublimate
470 here to concentrate the ice to our estimated values, as such the resulting layer would be more
471 similar to a salt-H₂O mixture in sediments.

472 (4) Salt could also be concentrated via the accumulation of water in a paleolake, which then
473 dried up, concentrating salts in the paleolake sediments. Similar processes are thought to have
474 formed the chloride deposits found throughout the southern highlands (*Osterloo et al.*, 2008). Note
475 that these previously identified deposits have a maximum area of 25 km² compared to the strong
476 reflector that has an area of ~600 km².

477

478 **6. Conclusions**

479 Here, we have compiled new and old measurements of electrical properties of Martian analog
480 materials at SPLD basal temperatures. This paper has shown that of all the materials proposed as
481 the source of the bright reflections at Ultimi Scopuli, only brine-rich ice mixtures are viable
482 options. Ca(ClO₄)₂ brines are the most likely brine as they have the lowest eutectic temperature of
483 any common Martian salt. The concentration of Ca(ClO₄)₂ to H₂O needed is 140 – 310 mM (3.3 –
484 7.1 mass%) to obtain the observed 1st and 3rd quartile values. Likewise if liquid brines exist in
485 sediment with ~40% porosity, a larger concentration of 0.77 – 2.5 M (16.4 – 43.4 mass%) of
486 Ca(ClO₄)₂ is required to obtain the observed 1st and 3rd quartile values. Thus, the sediments require
487 a factor of ~5-16 concentration in salt enhancement compared to that measured at the Phoenix
488 landing site. However, the large increase by Ca(ClO₄)₂ brines is only stable above its measured
489 eutectic temperature of 197.3±0.2 K, which is too large estimates of simple SPLD thermal models.

490 We suggest that layers of CO₂ ice or CO₂ mixed in with H₂O could increase the basal temperatures
491 above the eutectic temperature of Ca(ClO₄)₂. Additionally, the volume of brine is estimated to be
492 2.7-6.0 vol% and 6.3-28.7 vol% in ice mixtures and sand mixed ice, respectively. Thus, partially-
493 saturated ice or ice sediment mixtures (not briny lakes) are required to produce the observed
494 reflection.

495 If thermal modeling cannot increase temperatures to the eutectic temperature of Ca(ClO₄)₂, then
496 metastable brines are necessary to drop the range at which brines could exist. However, additional
497 research is needed constrain the stability/lifetime of metastable brines, as well as a larger
498 concentration of salt. We also demonstrated that grey hematite is the only known mineral on Mars
499 to possess a large ϵ_a when dry and at SPLD-like temperatures. However, concentrations 2–4 times
500 (33.2–59.0 vol%) than any currently known are needed to match the MARSIS SPLD observations.
501 Such large concentrations of grey hematite would produce a gravity anomaly that could be used to
502 test this hypothesis.

503

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511

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