

# Crystal molds on Mars: Melting of a possible new mineral species to create Martian chaotic terrain

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## ABSTRACT

Images sent back by the Mars Exploration Rover *Opportunity* from the Meridiani Planum show sulfate-rich rocks containing plate-shaped voids with tapered edges that are interpreted as crystal molds formed after a late-stage evaporite mineral has been removed. Experimental studies of the  $\text{MgSO}_4\text{-H}_2\text{O}$  system at low temperatures reveal that the triclinic phase  $\text{MgSO}_4\cdot 11\text{H}_2\text{O}$  exhibits a crystal morphology that matches the shapes of these molds.  $\text{MgSO}_4\cdot 11\text{H}_2\text{O}$  melts incongruently above 2 °C to a mixture of 70% epsomite ( $\text{MgSO}_4\cdot 7\text{H}_2\text{O}$ ) and 30%  $\text{H}_2\text{O}$  by volume. When this occurs while crystals are encased in sediment, plate-shaped voids remain. The existence of ice, low surface temperatures, and the high sulfate content of surface rocks and soil on Mars makes  $\text{MgSO}_4\cdot 11\text{H}_2\text{O}$  a possible mineral species near the surface at high latitudes or elsewhere in the subsurface. If an evaporite layer contained a significant amount of this phase, incongruent melting would result in a rapid release of a large volume of water and could explain some of the landform features on Mars that are interpreted as outflow channels.  $\text{MgSO}_4\cdot 11\text{H}_2\text{O}$  would not survive a sample return mission unless extraordinary precautions were taken.

**Keywords:** sulfate, epsomite, Mars, mineralogy, incongruent melting.

## INTRODUCTION

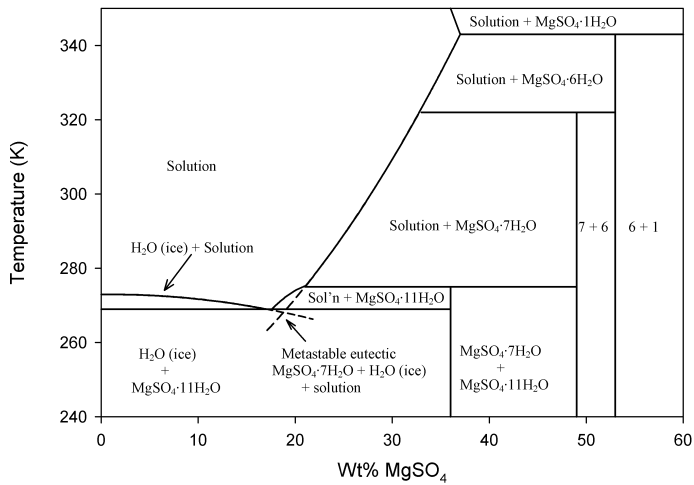
Hydrated sulfate minerals are suspected to exist on the surface of Mars. The evidence for hydrate sulfates comes from several instruments both on rovers on the surface and orbiters above the planet. The chemical analyses provided by measurements with the alpha particle X-ray spectrometer on the Mars Exploration Rover *Opportunity* show that rocks on the Meridiani Planum contain as much as 25 wt%  $\text{SO}_3$  (Rieder et al., 2004). The thermal emission spectra of outcrops identify 15–35 vol% sulfate minerals (Christensen et al., 2004). Mössbauer spectra indicate the presence of the sulfate mineral jarosite (Klingelhofer et al., 2004). Kieserite (Arvidson et al., 2005) and gypsum (Langevin et al., 2005) have been identified on the surface at the Terra Meridiani landing site as well as other areas of the planet by the OMEGA/Mars Express instrument. Exposed ice has been observed, at high latitudes, by the OMEGA spectrometer on the Mars Express Orbiter (Bibring et al., 2005) and high-resolution visible near-infrared data from the OMEGA spectrometer indicate that the upper 1–2 m of soil may contain as much as 6–10 wt% water in some areas (Milliken et al., 2005). Epsomite and hexahydrite have been proposed to explain the measured abundance of water-equivalent hydrogen in equatorial areas (Chipera et al., 2005; Feldman et al., 2004). Based on observations of sedimentary textures made by the Mars Exploration Rover *Opportunity*, it has been suggested that acidic salt solutions once existed on Mars (Squyres and Knoll, 2005). The combination of acidic sulfate solutions saturated with metals and subzero temperatures creates ideal conditions for the formation of hydrate metal sulfates such as  $\text{FeSO}_4\cdot 7\text{H}_2\text{O}$  (melanterite) and  $\text{MgSO}_4\cdot 11\text{H}_2\text{O}$ .

The experimentally determined phase diagram for the system  $\text{MgSO}_4\text{-H}_2\text{O}$  has been summarized (Hogenboom et al., 1995) and is presented in Figure 1. Fritzsche (1837) described the synthesis of crystals, at temperatures below 0 °C, of  $\text{MgSO}_4\cdot n\text{H}_2\text{O}$ , for which he concluded that  $n = 12$ . Fritzsche obtained small milky crystals that on close inspection were seen to consist of even smaller clear crystals.

Fritzsche measured the water content of crystals separated from solution and found, based on 3 measurements, that the water content was 62.43 wt%. Possible hydration states are therefore 11 (ideally 62.2 wt%  $\text{H}_2\text{O}$ ) or 12 (ideally 64.23%  $\text{H}_2\text{O}$ ). Fritzsche concluded that the formula was  $\text{MgSO}_4\cdot 12\text{H}_2\text{O}$  rather than  $\text{MgSO}_4\cdot 11\text{H}_2\text{O}$ , presumably because he observed that the material had become slightly cloudy from handling and he assumed a small amount of water loss had occurred. The atomic structure determination described in the following indicates that crystals grown by the method outlined by Fritzsche are  $\text{MgSO}_4\cdot 11\text{H}_2\text{O}$ .

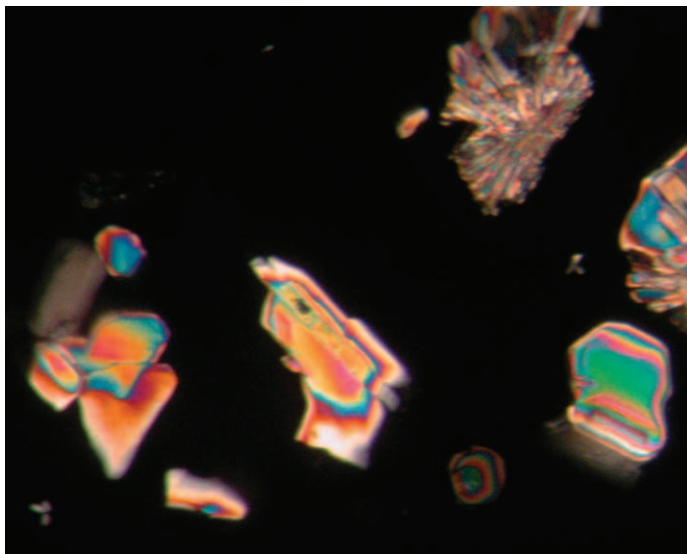
## EXPERIMENTAL METHODS

In this study  $\text{MgSO}_4\cdot 11\text{H}_2\text{O}$  was synthesized from a solution of 30 wt%  $\text{MgSO}_4$ –70 wt%  $\text{H}_2\text{O}$ . This solution was placed in an open Petri dish and left for several days at temperatures that ranged from –0.3 to –2.3 °C at a relative humidity of between 66% and 58%. The synthesis was conducted in an unheated garage and the temperature range was the result of diurnal variation in southeastern Ontario in late December. Acicular crystals of epsomite,  $\text{MgSO}_4\cdot 7\text{H}_2\text{O}$ , began to form, metastably, after 24 h. At 48 h clear colorless euhedral crystals of  $\text{MgSO}_4\cdot 11\text{H}_2\text{O}$  with a platy morphology and tapered edges began to form (Fig. 2). The crystals nucleate and grow separately but can also be closely associated with the metastable epsomite. Above 2 °C  $\text{MgSO}_4\cdot 11\text{H}_2\text{O}$  incongruently melts to  $\text{MgSO}_4\cdot 7\text{H}_2\text{O}$  and 4 $\text{H}_2\text{O}$  (Fig. 1). Clear single crystals transform very quickly to a mixture of fine epsomite needles and solution. This rapid melting can be observed under cross-polarized light (Fig. 2). Placing a finger near an  $\text{MgSO}_4\cdot 11\text{H}_2\text{O}$  crystal for just a moment results in a slight increase in temperature and the formation of a splay of needles of epsomite inside the crystal of  $\text{MgSO}_4\cdot 11\text{H}_2\text{O}$ . The needles emanate from a point closest to the heat source and the final result is a slurry of shards of epsomite and solution. The speed at which this melting occurs is much faster than the melting rate for an equivalent-sized crystal of water ice, indicating a much lower heat of fusion of  $\text{MgSO}_4\cdot 11\text{H}_2\text{O}$ .

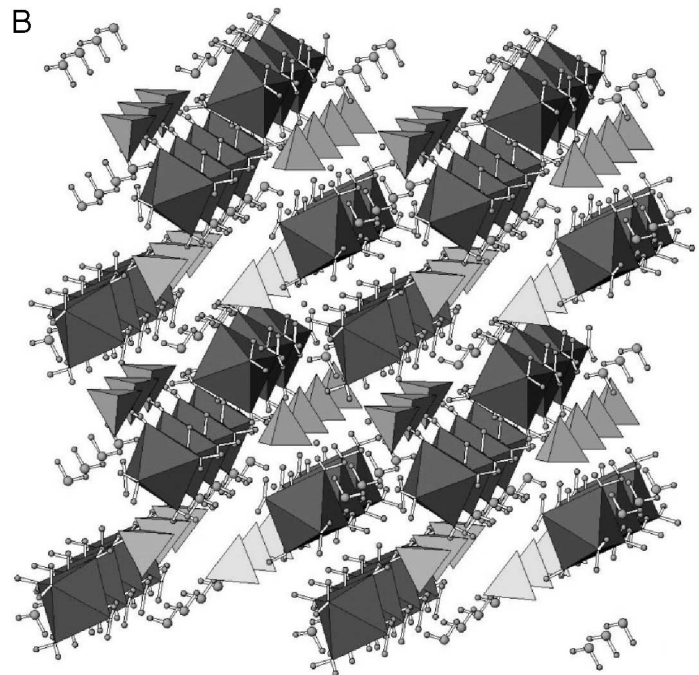
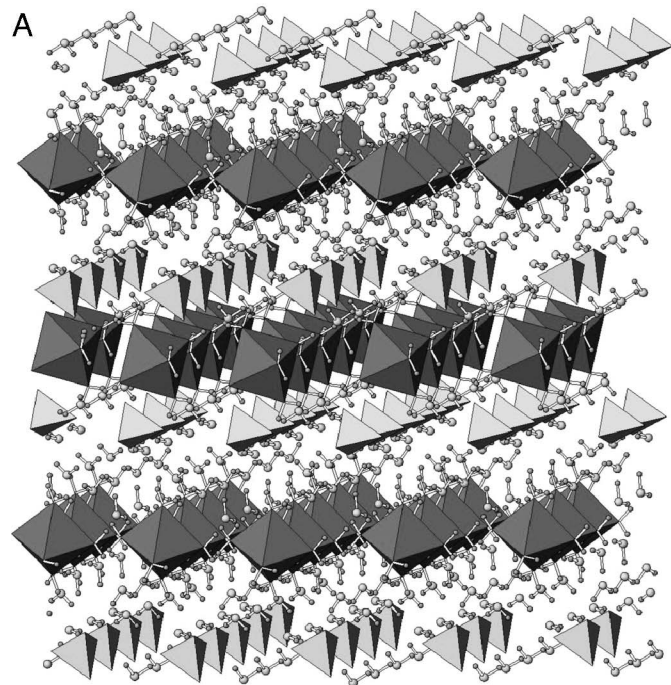


**Figure 1.** Phase diagram for  $\text{MgSO}_4\text{-H}_2\text{O}$  system (after Hogenboom et al., 1995). Field of  $\text{MgSO}_4\cdot 12\text{H}_2\text{O}$  in original reference has been relabeled as  $\text{MgSO}_4\cdot 11\text{H}_2\text{O}$  based on this work.  $\text{MgSO}_4\cdot 11\text{H}_2\text{O}$  melts incongruently to epsomite and saturated solution at  $2^\circ\text{C}$  ( $275\text{K}$ ) in this binary system.

During the course of this study the crystal structure of  $\text{MgSO}_4\cdot 11\text{H}_2\text{O}$  was determined. A colorless platy crystal,  $0.30 \times 0.30 \times 0.20\text{ mm}$ , was sealed in a capillary with mineral oil at  $0^\circ\text{C}$ . The capillary containing the crystal was mounted on a goniometer head and the capillary was covered with a chilled thick-walled 8-mm-diameter Pyrex tube to provide thermal insulation. The goniometer was then surrounded with snow and packed in an insulated container. Once at the diffractometer, the goniometer was quickly placed on the instrument and the crystal was cooled to  $-93^\circ\text{C}$  in a stream of nitrogen gas controlled with a Cryostream Controller 700. The space group was found to be P-1 with  $a = 6.7322(6)\text{ \AA}$ ,  $b = 6.7915(7)\text{ \AA}$ ,  $c = 17.2930(17)\text{ \AA}$ ,  $\alpha = 88.220(2)^\circ$ ,  $\beta = 89.494(2)^\circ$ ,  $\gamma = 62.664(2)^\circ$ , and  $Z = 2$ . Details of the X-ray diffraction data collection, crystallographic data, atomic coordinates and equivalent isotropic displacement parameters, bond lengths and angles, anisotropic displacement parameters, hydrogen coordinates and isotropic displacement parameters, and hy-



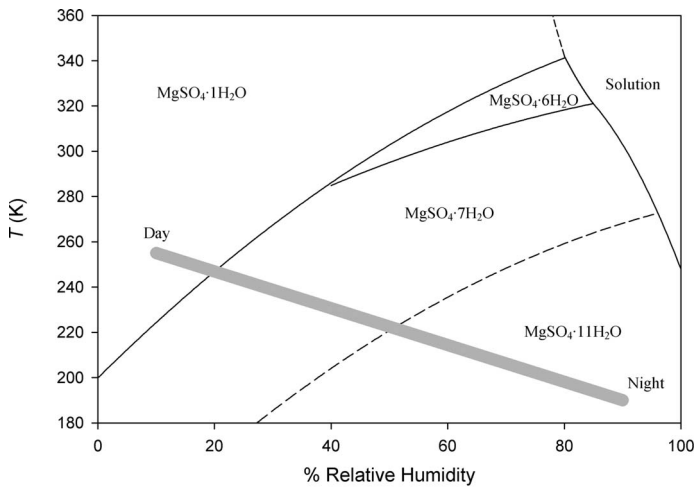
**Figure 2.** Crystals of  $\text{MgSO}_4\cdot 11\text{H}_2\text{O}$  in cross-polarized light. Tapered morphology of crystals is indicated by gradual reduction in birefringence toward edges of each crystal. Crystal that has incongruently melted to splay of epsomite needles and solution is visible at upper right corner. Field of view =  $1\text{ mm}$ .



**Figure 3.** Comparison of crystal structure of  $\text{MgSO}_4\cdot 11\text{H}_2\text{O}$  (A) and epsomite  $\text{MgSO}_4\cdot 7\text{H}_2\text{O}$  (B): 11 hydrate structure consists of layers of magnesium containing octahedra and layers of mixture of magnesium octahedra and sulfate tetrahedra, separated by layer composed of molecules of  $\text{H}_2\text{O}$ . Epsomite has much less water and magnesium octahedra and sulfate tetrahedra are more closely spaced. Linear arrangements of  $\text{Mg}(\text{H}_2\text{O})_6\text{-SO}_4$  groups exist in both structures.

drogen bond information are available in the GSA Data Repository.<sup>1</sup> The crystal structure (Fig. 3) consists of  $\text{Mg}(\text{H}_2\text{O})_6$  octahedra that are linked into chains by hydrogen bonds to sulfate tetrahedra. These

<sup>1</sup>GSA Data Repository item 2006212, appendix and data tables DR1-DR6, is available online at [www.geosociety.org/pubs/ft2006.htm](http://www.geosociety.org/pubs/ft2006.htm), or on request from [editing@geosociety.org](mailto:editing@geosociety.org) or Documents Secretary, GSA, P.O. Box 9140, Boulder, CO 80301, USA.

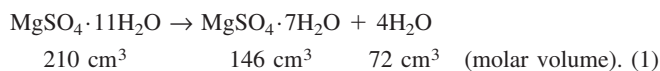


**Figure 4.** Phase diagram illustrating hydration-dehydration relationships of  $\text{MgSO}_4$  as function of temperature ( $T$ ) and relative humidity (Chou and Seal, 2003). Gray line indicates conditions at Viking Lander 1 site in summer (Savijarvi, 1995). Possible boundary for epsomite- $\text{MgSO}_4 \cdot 11\text{H}_2\text{O}$  as function of relative humidity is given as dashed line by assuming it to be parallel to kieserite-epsomite dehydration boundary. Dehydration-hydration rates are slow at these temperatures. Reactions would not reverse on daily basis, but in general temperature and relative humidity conditions at or below surface at Viking site are such that either epsomite or  $\text{MgSO}_4 \cdot 11\text{H}_2\text{O}$  will be stable phase.

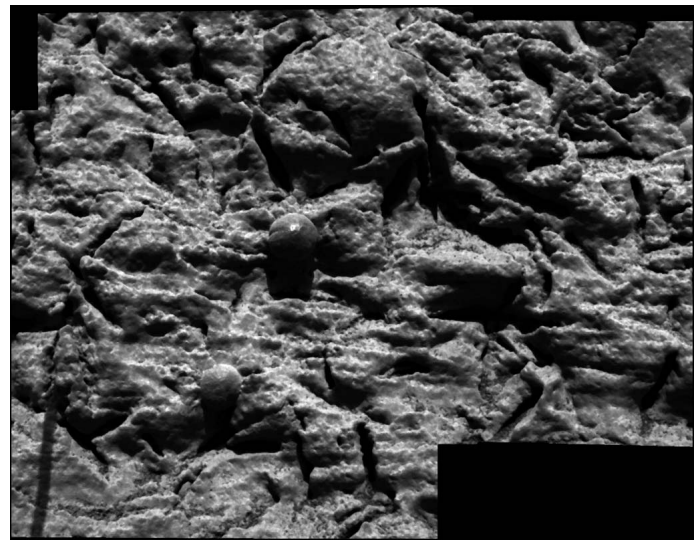
chains are then linked together by hydrogen bonds involving additional water molecules to form sheets. A second set of  $\text{Mg}(\text{H}_2\text{O})_6$  octahedra is linked by hydrogen bonds to form a second sheet. These two sheets are held together by hydrogen bonding to water molecules found between the layers. The difference between the structure of  $\text{MgSO}_4 \cdot 11\text{H}_2\text{O}$  and epsomite ( $\text{MgSO}_4 \cdot 7\text{H}_2\text{O}$ ) is clear in Figure 3. The additional water molecules contained in  $\text{MgSO}_4 \cdot 11\text{H}_2\text{O}$  create a very open hydrogen-bonded structure with a layer arrangement, whereas the epsomite structure consists of closely packed chains. Two apices of a sulfate tetrahedron link octahedra in epsomite to form a strong chain. In  $\text{MgSO}_4 \cdot 11\text{H}_2\text{O}$  only one apex of a sulfate tetrahedron is hydrogen bonded to two magnesium octahedra to form a weaker chain link.

## RESULTS AND DISCUSSION

A significant amount of water is produced by the incongruent melting of  $\text{MgSO}_4 \cdot 11\text{H}_2\text{O}$ ; 1 mol of  $\text{MgSO}_4 \cdot 11\text{H}_2\text{O}$  will yield 1 mol of epsomite and 4 mol of water. A small amount of the resulting epsomite will dissolve in the 4 mol of the evolved water.



At reduced humidity and temperatures lower than 0 °C, in the absence of solution or ice,  $\text{MgSO}_4 \cdot 11\text{H}_2\text{O}$  will slowly dehydrate to epsomite through diffusion-limited dehydration (Fig. 4). This boundary is not well constrained and an in situ diffraction experiment is planned to study this reaction. The transformation from epsomite to the lower hydrates takes place at low temperatures by diffusion-limited dehydration to vapor and a lower hydrate. The hydrated magnesium sulfate minerals that have been observed as minerals on Earth are kieserite ( $\text{MgSO}_4 \cdot 1\text{H}_2\text{O}$ ), sanderite ( $\text{MgSO}_4 \cdot 2\text{H}_2\text{O}$ ), starkeyite ( $\text{MgSO}_4 \cdot 4\text{H}_2\text{O}$ ), pentahydrate ( $\text{MgSO}_4 \cdot 5\text{H}_2\text{O}$ ), hexahydrate ( $\text{MgSO}_4 \cdot 6\text{H}_2\text{O}$ ), and epsomite ( $\text{MgSO}_4 \cdot 7\text{H}_2\text{O}$ ). The epsomite-hexahydrate phase boundary has previously been studied as a function of temperature and relative humidity between 25 and 45 °C and 56 and 80% relative humidity at 0.1 MPa total pressure (Chou and Seal, 2003).



**Figure 5.** Mosaic image taken by microscopic imager on Mars Exploration Rover *Opportunity* showing portion of rock outcrop at Meridiani Planum, Mars, dubbed "Guadalupe." Plate-shaped features are ~1 mm across and as long as 8 mm and have tapered edges. In other rocks nearby, molds continue into rock for at least as deep as rock abrasion tool was able to grind (~5 mm) and often increase in dimension with depth (Herkenhoff et al., 2004, 2006) Field of view is 4.8 cm across. Nasa/JPL image 16-jg-02-mi1-B035R1.br2.

There are conditions on Earth where  $\text{MgSO}_4 \cdot 11\text{H}_2\text{O}$  would form and thus be a new mineral species. However, the upper stability limit of 2 °C makes collection and analysis difficult. Epsomite crystallizes from saturated solutions in a variety of occurrences (Gaines et al., 1997), and some of these localities are below freezing at times during the year. Crystallization of these solutions below 2 °C would result in a naturally formed mineral with composition  $\text{MgSO}_4 \cdot 11\text{H}_2\text{O}$ . The confirmation of  $\text{MgSO}_4 \cdot 11\text{H}_2\text{O}$  on the surface of Mars would allow the designation of a new mineral species on another planet. However, it would be difficult for a mission to return samples of  $\text{MgSO}_4 \cdot 11\text{H}_2\text{O}$  for study without degradation (Vaniman et al., 2004). Epsomite and the lower hydrates would survive the trip from Mars if packed in filled sealed containers or immersed in mineral oil and kept cool.  $\text{MgSO}_4 \cdot 11\text{H}_2\text{O}$  will transform to epsomite and solution unless it is kept below 2 °C.

During Sol 28 the Mars Exploration Rover *Opportunity* investigated a rock on Meridiani Planum, named "Guadalupe," that exhibited elongate cavities and hematite spheres. The plate-shaped cavities in the rock have been described as "crystal-shaped moulds" (McLennan et al., 2005, p. 111). The cavities are 0.5–1.5 mm wide and 2–8 mm long with tapered edges (Fig. 5). Large crystals of a late-stage evaporate mineral can grow in or at the sediment surface and if they subsequently dissolve or melt they can leave behind cavities that are the shape of the original crystals. The fact that the immediate surroundings of these molds have not undergone obvious dissolution suggests that the material that once filled the molds was very soluble.  $\text{MgSO}_4 \cdot 11\text{H}_2\text{O}$  is a possible candidate for this material; the crystals exhibit a plate-shaped morphology that thins toward the margins (Fig. 2). On warming, crystals of  $\text{MgSO}_4 \cdot 11\text{H}_2\text{O}$  melt incongruently to epsomite and solution and, if enclosed in a sediment, would immediately create a euhedral mold of the original crystal that would be partially filled with epsomite. Over time, at reduced relative humidity conditions, the epsomite in the crystal molds would slowly dehydrate to hexahydrate and finally kieserite. The solids remaining in the crystal mold would then be 25% of the volume (Table 1) of the original crystal of  $\text{MgSO}_4 \cdot 11\text{H}_2\text{O}$  and would be very fine grained. If the enthalpy of fusion of  $\text{MgSO}_4 \cdot 11\text{H}_2\text{O}$  is low, as suggested by the rapid melting seen for the synthetic material, in-

TABLE 1. COMPARISON OF CELL VOLUMES OF  $\text{MgSO}_4 \cdot n\text{H}_2\text{O}$ 

Phase	Unit cell volume $\text{\AA}^3$ normalized to $Z = 4$	Difference (%)
$\text{MgSO}_4 \cdot 11\text{H}_2\text{O}$	1,404.04 (12)	
Epsomite <sup>a</sup> $\text{MgSO}_4 \cdot 7\text{H}_2\text{O}^*$	976 (2)	30.5
Hexahydrate <sup>b</sup> $\text{MgSO}_4 \cdot 6\text{H}_2\text{O}^*$	880.6	37.3
Kieserite <sup>c</sup> $\text{MgSO}_4 \cdot 1\text{H}_2\text{O}^*$	355.6 (2)	74.7

Note: Values in parentheses are experimental errors in the cell volume.

\*Sources of data; <sup>a</sup>Bauer (1964); <sup>b</sup>Zalkin et al. (1964); <sup>c</sup>Hawthorne et al. (1987).

congruent melting would occur quickly. When the abundance of  $\text{MgSO}_4 \cdot 11\text{H}_2\text{O}$  in the sediment is high, such that the crystals are well connected, the rapid release of fluid could explain some of the Martian topography that has been postulated to be the result of a rapid outwash of fluid. The fluid would be saturated with  $\text{MgSO}_4$  and would leave a residue of magnesium sulfate in the surface sediments as it evaporated.

It has been proposed (Montgomery and Gillespie, 2005) that dehydration of evaporite beds could cause the observed outwash channels on Mars. The model they proposed involves an increase in the geothermal gradient, which causes dehydration of buried hydrous phases such as gypsum, epsomite, or mirabilite. The reactions, such as the dehydration of gypsum or epsomite, would result in the production of liquid water only if there is significant overpressure from overlying deposits.

Other sulfate minerals that may have formed the molds are melanterite, epsomite, or gypsum (Squyres and Knoll, 2005). All of these would slowly dehydrate under Martian conditions with no possibility of melting. Melanterite most commonly exhibits a prismatic habit (Gaines et al., 1997). Melanterite requires ferrous iron, which could exist in strongly acidic solutions. Gypsum can exhibit a platy morphology (Gaines et al., 1997), but is much less soluble than other evaporite minerals.

The interaction of sulfate brines with ice and rocks on the surface of Mars is a reasonable model to explain the spectroscopic observations made by orbiters and the visual and chemical observations made by landers and surface rovers. The phase  $\text{MgSO}_4 \cdot 11\text{H}_2\text{O}$  is expected under these conditions and may be an important sulfate on Mars. If confirmed,  $\text{MgSO}_4 \cdot 11\text{H}_2\text{O}$  would be the first new mineral species found on Mars.

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