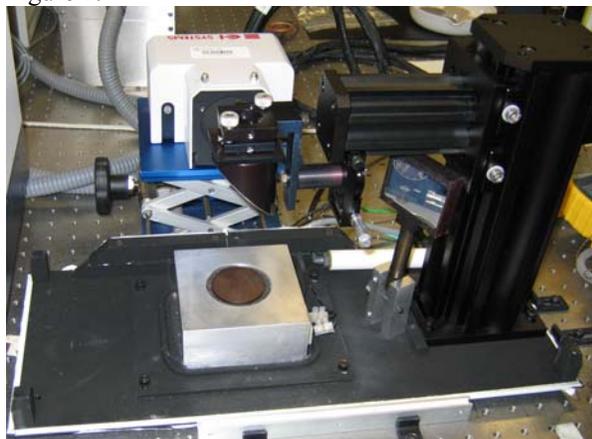


**DEHYDRATION OF PHYLLOSILICATES UNDER LOW TEMPERATURES: AN APPLICATION TO MARS.** A. Maturilli, J. Helbert, and M. D'Amore, Institute for Planetary Research, DLR, Rutherfordstrasse 2, 12489, Berlin, Germany (alessandro.maturilli@dlr.de)

**Introduction:** Hydrous minerals as phyllosilicates class have been detected on Mars by the OMEGA instrument, onboard the ESA Mars Express mission [1, 2]. These minerals can keep the record of the interaction of martian pristine rocks with water. The OMEGA analysis found a limited range of mineralogy (Fe/Mg and Al smectite), located only in ancient terrains [3]. The very recent measurements of the CRISM instrument, onboard the NASA MRO mission, show that the phyllosilicate mineralogy on Mars is wider, giving evidence of kaolinite, chlorite, illite or muscovite, and hydrated silica. Furthermore, nontronite, saponite, and in less amount, chlorite are the most common smectites [3].

**Laboratory set-up:** The instrumentation is located in the Planetary Emissivity Laboratory (PEL) at the Institute for Planetary Research (PF) of the German Aerospace Center (DLR) in Berlin, Germany. It consists of an air purged spectrometer Bruker IFS88 attached to an external emissivity device. The 3 to 20  $\mu\text{m}$  spectral range is covered using a liquid nitrogen cooled MTC detector, and a KBr beam-splitter.

The emissivity device is composed of the sample chamber, a double-walled box with three apertures: a 15 cm squared door used to insert the cup in the box, a 5 cm rounded opening through which the beam is directed to the spectrometer and a 5 cm opening facing the attached blackbody unit. A heater is placed in the chamber and is used to heat the cup with samples from the bottom. The thermal radiation emitted normal to the surface by the sample or the blackbody is collected by an Au-coated parabolic off-axis mirror and reflected to the entrance port of the spectrometer, as in Figure 1.



**Figure 1:** Emissivity chamber without cover.

A pump circulates water at a constant temperature in the volume between the inner and outer walls of the chamber. The surfaces of the box are painted with black high emissivity paint. The chamber is purged with dry air to remove particulates, water vapour and  $\text{CO}_2$ . Further details can be found in [5, 6].

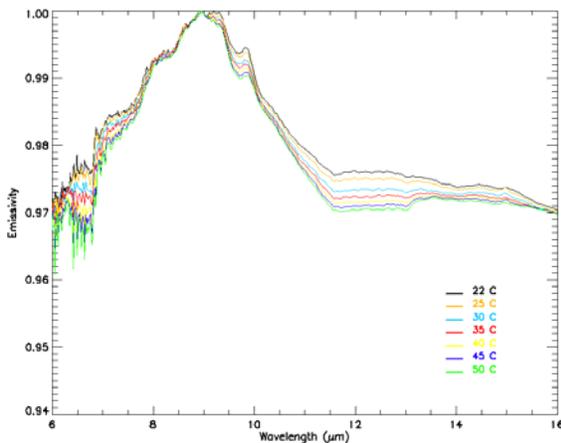
**Sample preparation:** The standard Martian analogue material JSC Mars-1 (a palagonitic tephra from Hawaii, USA) was chosen to carry on the experiment. To prevent any influence of the used material's grain size on the measured quantities, we choose the raw, unsieved sample available in our laboratory for the experiment. Immediately after filling, the cup was weighted with a high precision balance. Then the sample was as fast as possible transferred in the emissivity chamber, on the sample heater. Figure 2 shows the JSC Mars-1 sample, poured in the 2 cm high rim cup.



**Figure 2:** Cup with the JSC Mars-1 unsieved sample.

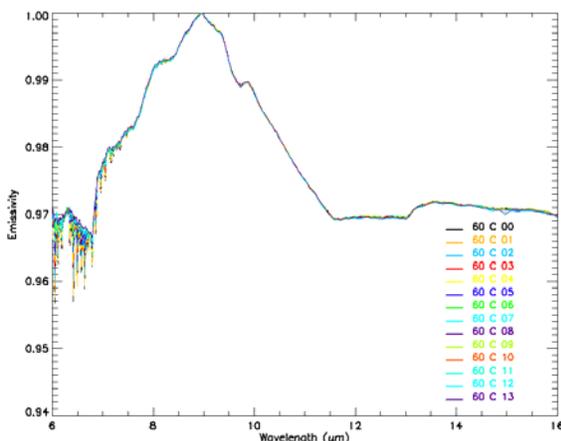
The first measurement was taken 7 minutes later, with the heater switched off, thus the sample was at room temperature ( $22^\circ\text{C}$ ). To decrease the influence of the ambient (emissivity chamber) on the measurements, the circulating water temperature was set to  $10^\circ\text{C}$  for this experiment. The temperature of the sample emitting layer was estimated to be  $18^\circ\text{C}$ . Five minutes later the sample was measured with the heater set at  $25^\circ\text{C}$ , and then to  $30^\circ$  after successive 5 minutes. Then, the relaxation interval was set to 10 minutes and the sample was measured with a  $5^\circ\text{C}$  increase each time, until  $50^\circ\text{C}$  on the heater display. Figure 3 shows the calibrated emissivity for this first set of measurements. The small variations in the curve continuous remains well below 1%, while a typical error level for emissivity measurements is around 2% [5]. On the other side, it is clearly visible that the numerous water

bands between 6 and 8  $\mu\text{m}$  increase with the temperature, that indicates a de-hydration of the mineral. In fact, what we see in the spectra, is water vapour dispersed from the heated mineral in the atmosphere inside the emissivity cup. The short relaxation time used is so small that the purging system can't get rid of this gas.



**Figure 3:** Emissivity of JSC Mars-1, part 1.

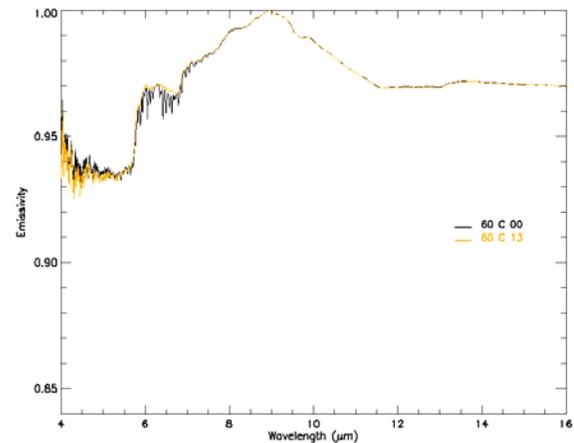
Then the heater temperature was set to 60° C and after the usual 10 minutes relaxation time, a serie of repeted measurements (each 10 minutes) was taken. The estimated temperature of the sample emitting layer was 44.5° in this case. The second set of calibrated spectra is shown in Figure 4.



**Figure 4:** Emissivity of JSC Mars-1, part 2.

In this picture, we can see that the water bands start to decrease in intensity, because the losing rate of adsorbed water slowed or even all the available water was already evaporated, and the purging inside the chamber was starting to be effective. Figure 5 shows the first and last spectra of this second set of measurements only. After 2 hours at 60° C (44.5° C in the surface proximity) the water bands observed in the region between 6 and 8  $\mu\text{m}$  almost disappeared, meaning that all or most of the available adsorbed water was lost to

the environment. After the last measurement was completed, the cup was immediately weighted, recording a loss of 6% in weight. It's presumable that the mass lost during the experiment was indeed the water released to the chamber, lost from the heated mineral.



**Figure 5:** Emissivity of JSC Mars-1, part 3.

**Conclusion and Outlook:** The described experiment was proposed to investigate the effect on TIR emissivity spectra of the loss in adsorbed water for a standard Martian soil simulant (JSC Mars-1), at low temperatures, typical for a Martian day. On the other hand, the results found confirm that the equipment in the Planetary Emissivity Laboratory (PEL) is suitable for emissivity measurements at low temperatures. The sample was heated starting from room temperatures to slowly and gradually reach the maximum temperature of 45° C on its surface. A loss of 6% in weight, attributable to loss water, was recorded at the end of the experiment. Spectral signature of this process will be recorded, confirming the result. Future work will extend the number of examined samples and the observing conditions.

**References:** [1] Bibring J. P. et al. (2005) *Science*, 307, 1576-1581. [2] Poulet F. et al. (2005) *Nature*, 438, 623-627. [3] Mustard J. F. et al. (2008) *Nature*, doi:10.1038/nature07097. [4] Helbert J. et al. (2007) *ASR*, 40, 272-279. [5] Maturilli A. et al. (2006) *PSS*, 54, 1057-1064. [6] Maturilli A. et al. (2008) *PSS*, 56, 420-425. [7] Koeppen W. C. and Hamilton V. E. (2005) *JGR*, doi:10.1029/2005JE002474. [8] Glotch T. D. et al. (2007) *Icarus*, 192, 605-622.