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Design, Development and Testing of an Environmental P-T Cell for InfraRed Spectroscopy Measurements.

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Introduction. Infrared spectra of minerals and rocks acquired in the laboratory are often dominated by the presence of absorption features due to adsorbed water. Frequently H₂O molecules from ambient water vapor, at standard P-T conditions, are subject to physical adsorption: the molecules quickly attach between the surface grains of the materials (especially powders, but also slabs) both during the preparation, storage and measuring phases. Fundamental absorption features due to OH-stretching and H-O-H bending transitions in water occur in the 3-6- μm spectral region, while overtones and combinations occur in the 0.9-2.5- μm region. These absorptions can often mask diagnostic bands characteristic of the host materials, and this can also be problematic when using laboratory spectra for comparison and interpretation of data from planetary missions. In order to overcome these issues, we designed and developed an environmental cell to acquire infrared reflectance spectra of materials under controlled pressure and temperature.

Cell Design. The vacuum chamber was realized custom by Vacom, while other parts were realized custom at INAF-IAPS. The sample to be measured is placed in a copper sample holder, which is in contact with a ceramic heater, that allows to warm up to 400 °C. The temperature control is performed thanks to a PID controller. The cell consists of a UHV stainless steel cube: a primary diaphragm membrane pump and a secondary turbomolecular pump in the current version permit to reach a vacuum of about 10^{-5} mbar within the cell. A 3-mm thick transparent CaF₂ window allows acquiring spectra in the visible-infrared range. Both the sample holder and ceramic heater are 25.4 mm in diameter, while the viewport diameter is 40 mm. The total height of the cell is about 50 mm.

Preliminary tests. We performed numerous tests with various types of powder samples, mostly phyllosilicates with different hydration states, using a FieldSpec Pro spectro-photometer in the range 0.35-2.5 μm and a QTH lamp. Test spectra were acquired in continuously-running mode by placing a sample within the cell, then pumping from ambient pressure down to 10^{-4} - 10^{-5} mbar, and keeping fixed the temperature at room values.

In the pumping phase the sample lost almost all the adsorbed water. We start the heating phase once that the limit pressure is reached. Further spectra were acquired at different temperatures in the range 20-300 °C. Most of the samples lost all their water above 150-

200 °C. At higher temperatures ($T > 240 \div 250$ °C) we register the dehydroxylation process for a few phyllosilicates, in accordance with the TGA data for these minerals.

Example 1: Hectorite. The spectra of hectorite (SHCa_1) acquired during the initial pumping phase from 10^4 down to 10^{-4} mbar are shown in figure 1. The water absorptions located at 1.4 and 1.9 μm almost disappear during the pumping. The absorption band near 2.3 μm , likely due to Mg-OH, remains quite unaltered. The spectra of hectorite acquired in vacuum (10^{-4} mbar) during the subsequent heating phase are shown in figure 2. The room P-T spectrum (red) is shown for comparison. Spectra have been acquired during the temperature ramp 50-250 °C. The mineral experiences dehydroxylation for $T > 240$ °C, as evidenced by the net change in spectral shape and the disappearing of hydration bands.

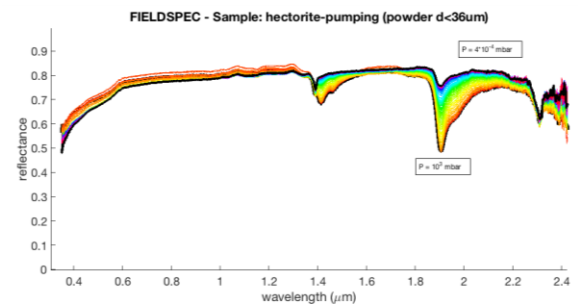


Fig. 1. Spectra of hectorite, acquired with FieldSpec Pro during the pumping phase, from 10^4 to 10^{-4} mbar. The water absorptions at 1.4 and 1.9 μm almost disappear during the pumping.

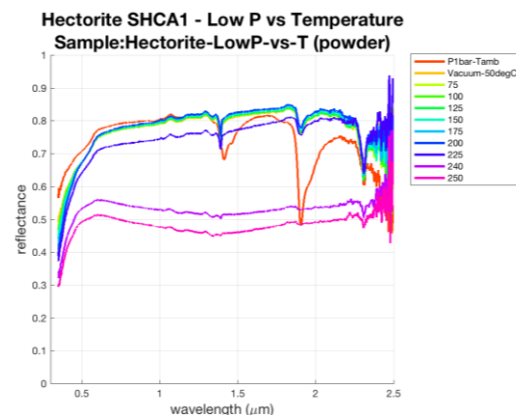


Fig. 2. Spectra of hectorite acquired with FieldSpec Pro in vacuum, during the heating phase. The starting spectrum (room P-T) is in red, for comparison. Spectra are acquired during the temperature ramp 50-250 °C.

Example 2: NH₄-Nontronite. As a second example we consider ammonium nontronite (NH₄-NAU1). Spectra acquired during the pumping phase (10^4 to 10^{-4} mbar) are shown in figure 3. We can see that the absorption features due to adsorbed water located at 1.4 and 1.9 μm nearly disappear during the pumping stage. Two features due to NH₄⁺ ions occurring near 2.01 and 2.12 μm are not affected by the pumping, as well as the band near 2.25 μm , likely due to Al-OH. In figure 4 the spectra acquired in vacuum (10^{-4} mbar) during the heating phase are shown. The spectra remain quite stable for temperatures up to 200 °C. Above that temperature the absorption bands due to OH⁻, H₂O and NH₄⁺ tend to disappear. At T=250 °C the dehydroxylation occurs, and also the Al-OH almost disappears.

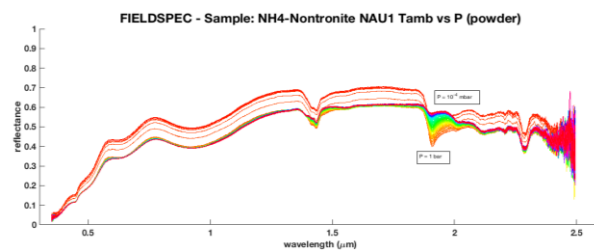


Fig. 3. Spectra of NH₄-nontronite, acquired during the pumping phase from ambient pressure to 10^{-4} mbar.

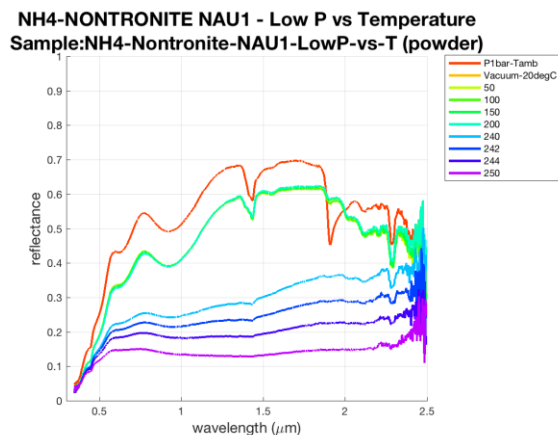


Fig. 4. Spectra of NH₄-nontronite, acquired in vacuum during the heating phase. The room P-T spectrum (red) is shown for comparison.

Conclusions and future work. In the next future efforts will be done to further reduce the lower limit pressure and in parallel to further increase the upper limit temperature. Moreover the cell will be coupled with an imaging spectrometer, to be used in the range 0.2-5.1 μm .

References: [1] Farmer, V. C.: *The vibrations of protons in minerals: hydroxyl, water, and ammonium*, in *The infrared spectra of minerals*, edited by V. C. Farmer, pp. 137-181, Mineralogical Society, London, 1974. [2] Clark, R.N., et al: *High spectral resolution reflectance spectroscopy of minerals*. Journal of Geophysical Research 95 (B8), 12653–12680, 1990

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