LABORATORY SPECTROSCOPY MEASUREMENTS OF MOON-MARS ANALOGUE SAMPLES.

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Introduction: Analysis of sampled material derived from Moon-Mars analogue locations was used to create a database of spectra that could function as a reference for future field measurements. Multiple spectroscopy techniques were tested to gain insight into the applicability of the spectrometers in the field. Measurements were conducted with a variety of instruments, namely a UV-VIS and NIR reflectance as well as a Fourier Transform IR (FTIR), an X-Ray Fluorescence (XRF) and Raman laser spectrometer.

The main focus of this research is the calibration of the UV-VIS and NIR spectrometers in order to bridge the gap between laboratory and in situ measurements of Moon-Mars analogue samples. In our survey, the portable spectrometers (UV-VIS, NIR and Raman) were used in combination with the ExoGeoLab lander [1] and conducted spectral analysis in the field, during campaigns in the Eifel, Germany in November 2015 and February 2016. With the stationary laboratory spectrometers samples collected during the campaigns were analysed, so that the obtained spectra could act as reference spectra.

Methodology: The test bench (fig. 1) on which the experiments were conducted consists of a sampling platform, an optical fiber holder, a light source and the spectrometers. The highly reflecting material Teflon was used for calibration and to acquire a reference spectrum. Multiple factors play a role in the calibration of the spectrometers, in pursuance of obtaining the best spectral signal. Calibrations in the set-up are e.g. those concerning the type of light source and the type of optical fibers used during the experiments as well as softwarematic calibrations and data processing, such as selecting suitable exposure times and applying calculations to the raw data.

In case of the UV-VIS reflectance spectroscopy daylight functioned as a light source, since this covers all wavelengths in the visible range. The set-up was similar for the NIR reflectance measurement, except that a "white" light source was used during less bright conditions. The optical fibers used during the experiments must allow enough flux to pass through in order to create an acceptable signal as well as prevent too much noise towards the edges of the range in wavelengths covered by each spectrometer.

Fibers compatible with the UV-VIS and NIR spectrometer are fiber patch cords with an SMA 905 connector covering wavelength ranges of 180 – 800 nm and 1100 – 2400 nm respectively. To acquire the best spectra the exposure time has to be adjusted so that the most accurate signal can be obtained without leading to an oversaturated signal. This was done e.g. in NIR by collecting a dark spectrum around 2000 counts and a reference spectrum around 4000 counts, resulting in an intensity signal of the sample with a maximum of 6000 counts. Typical integration times varied between 1 – 18 s for the UV-VIS measurements, where intergration times varying from 100 – 200 ms were used for the NIR measurements. During NIR experiments 400 – 1000 spectra were collected and averaged to eliminate statistical noise.

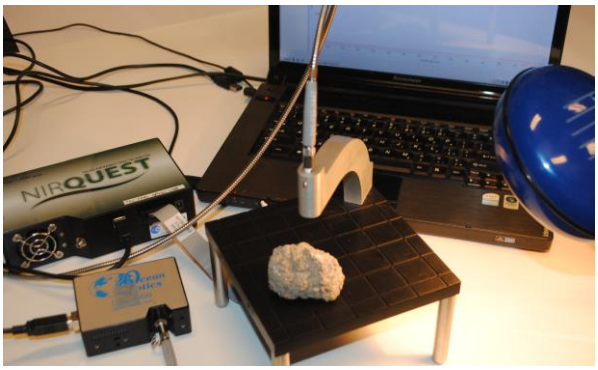

Figure 1. Test bench with UV-VIS and NIR reflectance spectrometers, optical fibers, sample platform, light source and sample derived from the Eifel campaign in November 2015.

For uniform measurements each sample was measured under similar conditions, where the distance towards the fiber was kept equal. Also each sample was measured under three different angles to collect an average reflectance spectrum taking into account the diffuse origin of the signal. Frequent recalibration of the set-up is required, due to the fact that the devices heat up during use, which results in changes in the spectra. For this purpose a methodical approach is required where reference and dark spectra are obtained before and after each sample measurement, so that the exact conditions during the experiment are known. After acquiring the sample spectra, separate calculations can be made in order to obtain the most accurate signal by taking into account the average of the collected dark and reference spectra.

Data: Volcanic samples derived from the previous Eifel campaign [1], Utah MDRS [2, 3], campaigns in Iceland [4] and the La Réunion campaign in France [5], were analysed together with the latest collection of

Mars-Moon analogue samples from the Eifel campaigns in 2015 and 2016.. The Mars-Moon analogue samples from VU University, Amsterdam, The Netherlands and standard lunar analogue reference minerals were also used for the calibration of both reflectance spectrometers.

The FTIR infrared absorption spectra were obtained within a wavelength range of 1 - 2.5 μm thanks to tests of samples carried out at Leiden University, The Netherlands. The XRF spectrometer, available at the University of Utrecht, The Netherlands, provided insight into the elemental composition of the samples. The Raman laser spectroscopy measurements were performed in the laboratory set-up at ESA/ESTEC as well as during the Eifel field campaigns in 2015 and 2016, resulting in the molecular identification and thereby mineralogical characterisation of the analogue samples. UV-VIS and NIR reflectance spectroscopy measurements resulted in reflectance spectra covering a wavelength range from 200 – 850 nm and 900 – 2500 nm, respectively, obtained during laboratory as well as field experiments.

Results: In the UV-VIS region of the electromagnetic spectrum molecules undergo electronic transitions. The reflectance in this range directly affects the colour of involved chemicals as it is shown in Figure 2, where the reflectance spectrum of an olivine reference sample is depicted. The green colour of olivine reflectance is higher in the corresponding wavelengths between 495 – 570 nm. Moreover, we searched for transitions in Fe state that are visible with a band at 700 nm.

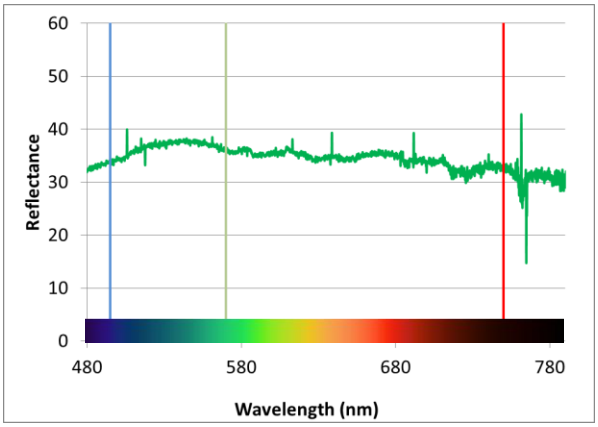

Figure 2. UV-VIS reflectance spectrum of olivine reference mineral before calibrations. Acquired with integration time of 18 s and 5 scans to average.

Additionally, the mineralogy of the sample (here olivine) is determined by characteristic bands in the NIR spectrum (fig. 3), which is is based on molecular overtone and combination vibrations.

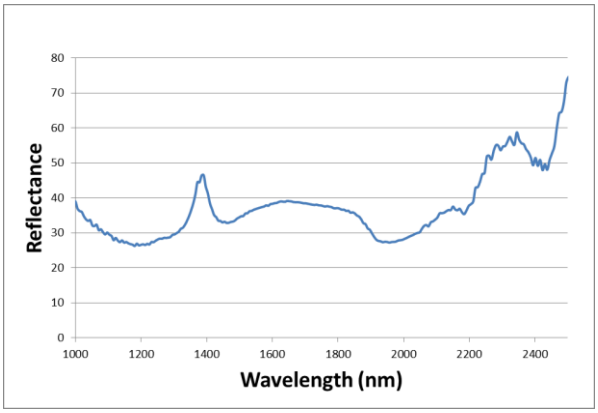

Figure 3. NIR spectrum of olivine before calibrations, showing characteristic band at 1 micron. Integration time: 196 ms, scans to average: 400.

Discussion & conclusion: To obtain the best results the set-up with the UV-VIS and NIR reflectance spectrometers needs to be calibrated between each sample measurement. The most suitable integration time for the UV-VIS spectrometer varies, depending on the availability of sunlight. For the NIR spectrometer the optimal integration time is dependent on the amount of light passing through the optical fiber, taking into account that longer exposure times might lead to oversaturation. Noise in the spectrum towards the edges of the covered wavelengths can be reduced by dividing the signal by a reference spectrum. Optical fibers covering a wider range of wavelengths should result in a clearer signal. Artefacts in the signal embedded in the NIR spectrometer are filtered out afterwards. The spectra obtained with the reflectance spectrometers is checked for accuracy with the use of the FTIR and Raman laser spectrometer.

Laboratory measurements of Moon-Mars analogue samples with UV-VIS and NIR spectrometers require specific set-up calibrations in order to attain the clearest spectra. Apart from selecting the optimal light source and optical fibers, new dark and reference spectra need to be acquired frequently in order to account for shifts in the signals caused by and during the use of the devices. An integration time to pick up a non saturated signal as well as performing enough scans to average the signal results in the clearest, noise-free spectra that can function as reference for future campaigns.

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References: [1] Foing B. et al, (2010) *LPSC*, 1701. [2] Foing B. et al, (2011) *IJA*, *10*, 137-139. [3] Foing B. et al, (2011) *IJA*, *10*, 141-160. [4] Calzada A., Foing B., Gazeas K., Tondeur A., Iceland Campaign, private communication. [5] Pignolet et al, (2010), *GLUC*, *3*, 6, 12.