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DEMONSTRATION OF VOLATILES EXTRACTION FROM NU-LHT-2M WITH THE PROSPA INSTRUMENT BREADBOARD. P. Reiss¹, L. Grill¹, and S. Barber², ¹Institute of Astronautics, Technical University of Munich, Boltzmannstr. 15, 85748 Garching, Germany, (p.reiss@tum.de), ²The Open University, Milton Keynes, MK7 6AA, UK.

Introduction: The European Space Agency (ESA) currently develops the Package for Resource Observation and in-Situ Prospecting for Exploration, Commercial exploitation and Transportation (PROSPECT) as a payload contribution to the planned Russian Luna-27 mission to the lunar polar regions. The package contains the sample analysis instrument ProSPA (PROSPECT Sample Processing and Analysis) to investigate volatile compounds that are extracted by heating regolith samples delivered by a drill [1-4]. In support of the instrument development, the Institute of Astronautics at Technical University of Munich (TUM) has established a breadboard to study the extraction of volatiles from hydrated lunar regolith analogues in a Moon-like environment [5]. This abstract describes the results from a test campaign that was dedicated to the Volatiles Extraction Demonstration (VED) mode of ProSPA.

Breadboard: The ProSPA laboratory breadboard at TUM consists of a sample conditioning system and a volatiles extraction system briefly described in the following, based on [6].

Sample conditioning system. The preparation of lunar regolith simulants for VED is done in a stainless steel glovebox with purge gas feed, humidification/dehumidification system, and an attached vacuum airlock with a separate heater setup for sample bake-out. The glovebox has an internal volume of ~ 0.8 m³ and automatically maintains an overpressure to avoid inward leakage from the laboratory atmosphere. The humidification/dehumidification system uses dry nitrogen as a purge gas, fed either directly into the glovebox for dehumidification or through a bubbler with distilled water before entering the glovebox for humidification. Temperature as well as relative humidity and dew point are recorded by a sensor. A precision balance is installed in the glovebox for sample weighing. The airlock is equipped with a pressure gauge, evacuation line, and a feedthrough for thermocouple and power supply to power a heater for sample bake-out.

Volatiles extraction system. The experimental setup for the thermal extraction of volatiles from the sample consists of an instrumented vacuum system with two sections (Figure 1). The lower part of the system can be evacuated to 10^{-5} mbar, and includes the sample holder, a purge gas feed, a Penningvac (Leybold PTR 90) pressure gauge, and a cooling/heating system that is applied externally to the sample holder. The upper part can be evacuated to 10^{-8} mbar, and contains a quadrupole mass

spectrometer (Stanford Research Systems RGA 200) and a second Penningvac pressure gauge. Both sections are connected via a manual dosing valve, which acts as an orifice to restrict the mass flow so that the RGA filament can be operated below its maximum gas pressure of 10^{-4} mbar. The entire system is evacuated using a turbomolecular pump and an oil-free scroll pump. The vacuum system is heated to 120 °C using heating wires wrapped around the tubes to enable a full bake-out and avoid condensation of the released water during operation. The sample holder is a modified stainless steel blind fitting to accommodate a sample that is 2.8 mm in diameter and 4.5 mm in height, according to the ProSPA baseline sample size.

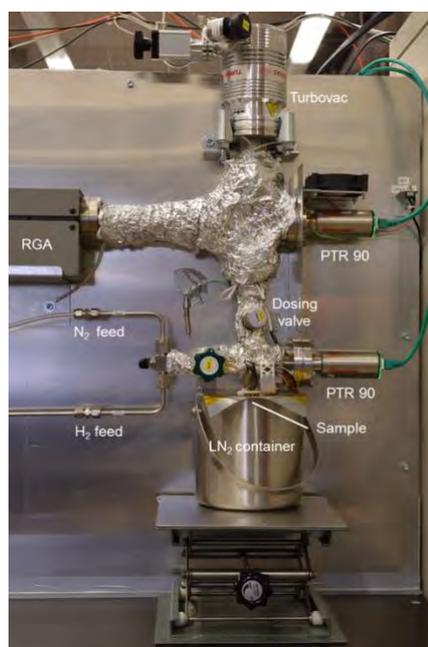


Figure 1: Detail view of the VED breadboard [6]

Procedure. Several grams of the lunar highland type regolith simulant NU-LHT-2M were placed inside the glovebox airlock and baked-out at 100-200 °C for 2 h in a medium vacuum ($\sim 10^{-1}$ mbar). The sample was then stored in the glovebox for at least 48 h to adsorb water from the humidified glovebox atmosphere. After that, a subsample of 33-36 mg (results in the ProSPA baseline sample size) was packed into the sample holder and transferred to the volatiles extraction system. After installation of the sample holder, it was cooled to -196 °C using a liquid nitrogen bath. The lower section of the vacuum system was then evacuated until the sample section reached values below 10^{-5} mbar. At this time the

sample heating was started with a controlled ramp of 6 °C/min from -150 °C to 800 °C. The evolved volatiles were constantly measured with the RGA, either by repeated analogue scans of m/z 1-50 or by tracking the partial pressures of selected species. Different sample conditions and test parameters were evaluated, as described in Table 1.

Table 1: Parameters varied for the VED tests

Parameter	Value
Relative humidity for sample conditioning	1 %, 10 %, 30 %, 50 %, 70 %, saturated
Sample mass	33-36 mg, 66-72 mg, 0 mg
Particle size	70-80 µm, 100-110 µm, none
Bulk density	loose (~1.3 g/cm ³), compacted (~2.0 g/cm ³), none
Heating rate	6 °C/min, 4 °C/min

Test Results: The following describes a brief summary of the key findings from the experiments, discussed in more detail in [6]. Figure 2 shows an exemplary outgassing profile for a 33-36 mg, 70-80 µm, loose sample, conditioned at 30 % relative humidity, and heated at a rate of 6 °C/min. The outgassing of volatiles from the sample can be divided into three distinct phases, as highlighted in Figure 2:

- (1) Initial outgassing of the sample holder surface at temperatures below -50 °C (peak 'A'),
- (2) Outgassing of volatile species from the sample between -50 °C and 300 °C (peak 'B'), and
- (3) Release of mineral-bound volatiles through decomposition above 300 °C (peak 'C').

The predominant species in the gas mixture is water (m/z 18). Hydrogen (m/z 2) is present as a residual gas from the atmosphere, carbon dioxide (m/z 44) and nitrogen or carbon monoxide (m/z 28) are mainly released at higher temperatures in the third temperature phase. Fragmentation through the RGA and the present mixture of gases however means that not all of the existing species can uniquely be identified. The release of m/z 44 and 28 is due to the decomposition of carbon-bearing minerals in NU-LHT-2M, most likely calcite (CaCO₃), above 650 °C. The signal of m/z 32 is attributed to sulphur-

bearing species that are released at 550 °C, most likely due to the decomposition of pyrite (FeS₂).

The effect of different humidity levels during the storage of the samples was visible at low relative humidity between 1 % and 10 %. Above that no further increase in the partial pressure of water was detected. The smaller particle fraction as well as the larger sample mass and the higher heating rate produced a generally higher amplitude in the partial pressures. The latter was also predicted by computer simulations of the heat and mass transfer in the sample and is further explained in [7].

Lessons Learned for ProSPA: The feasibility of VED was demonstrated with an instrument-like setup under relevant ambient conditions. It was found that:

- (1) A sufficient (measurable) amount of volatiles can be released from the sample,
- (2) Volatiles reliably arrive at the gas analysis instrument,
- (3) Reproducible outgassing profiles can be created,
- (4) Qualitative differences can be seen for different sample conditions.
- (5) Mineral decomposition is detected at temperatures above 300 °C.

Furthermore, it was found that NU-LHT-2M contains a noticeable amount of carbonates, which needs to be considered when comparing the results to real lunar regolith that most likely contains no carbonates. NU-LHT-2M also releases sulphur from pyrite, while in the case of lunar regolith a possible source of sulphur would be troilite (FeS). As a basis for future studies on VED with ProSPA, reference outgassing profiles were recorded for different combinations of boundary conditions and sample preparation methods, which can be used for interpretation of future measurements.

References: [1] Barber S. J. et al. (2017) *ELS*. [2] Barber S. J. et al. (2017) *LPSC*. [3] Carpenter J. et al. (2014) *LEAG*. [4] Fisackerly R. et al. (2015) *ASTRA*. [5] Reiss P. (2017), *ELS*. [6] Reiss P. (2018), Dissertation, *TUM*. [7] Reiss, P. (2018), *Icarus*, 306C 1-15.

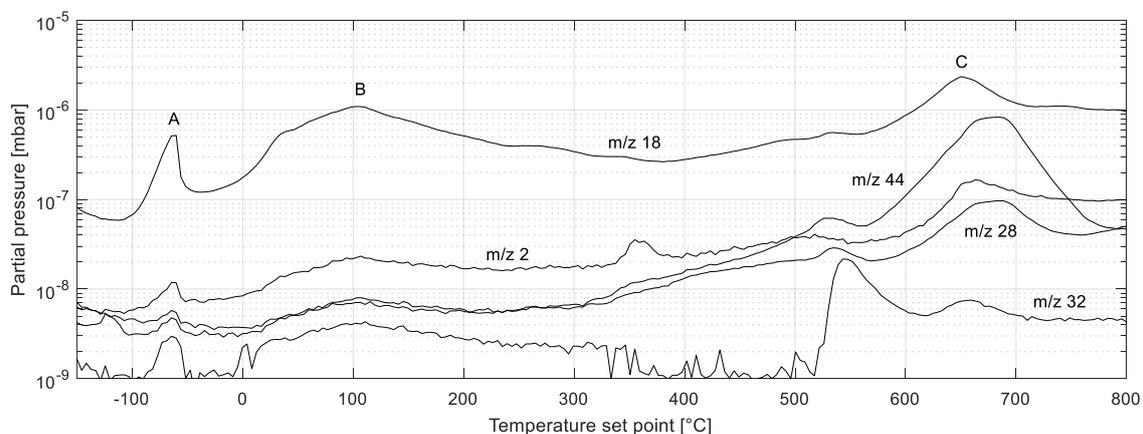


Figure 2: Exemplary partial pressure curves of selected m/z for the extraction of volatiles with 6 °C/min heating rate and a 33-36 mg, 70-80 µm, loose sample, conditioned at 30 % relative humidity [6]