

SOLID SPU: A TRL 5-6 SAMPLE PREPARATION INSTRUMENT FOR WET CHEMISTRY ANALYSIS ON MARS. J. M. Manchado¹, E. Sebastian¹, J. Romeral¹, J. Sobrado-Vallecillo¹, P. L. Herrero², C. Compostizo², J. Gómez-Elvira¹ and V. Parro¹, ¹ Centro de Astrobiología (CAB, INTA-CSIC), Torrejón de Ardoz, 28850 Madrid, Spain, ²SENER Ingeniería y Sistemas SA, Las Arenas, Spain. parrogv@cab.inta-csic.es.

Introduction: Sample preparation for downstream wet chemistry analysis in planetary exploration is challenging and not well established. This is one of the main reasons for the very few wet experiments performed on the surface of Mars so far. The proposed Icebreaker mission to Mars [1] which seeks to find organic biosignatures in ice-rich soils of the northern plains, is an excellent opportunity for the analysis of biomolecules in liquid extracts. We have developed SOLID (Signs Of Life Detector), an immunoassay-based instrument for biomarker detection in planetary exploration [2]. Current SOLID3.1 consists of two functional units, the Sample Preparation Unit (SPU), for the extraction of organics into a liquid solution, and the Sample Analysis Unit (SAU), for immunological assays in antibody microarray format. The new SPU (160x170x230 mm and 4.25 kg), consisting of a single extraction cell, can process up to 30 x 0.5 g powder or soil samples so that it renders ca. 2 mL of liquid extract per sample to feed downstream analytical instruments. After sample loading, the procedure involves: i) addition of liquid solvent into the extraction cell (EC); ii) homogenization and extraction of organic material into the solvent by ultrasonication; iii) evacuation of whole sample (solids plus solvent) into a deposit with cylindrical micrometer pore size walls; and iv) injection of filtered liquid extract into downstream analytical instruments (e.g. the SAU). The process of sample preparation may take 20 minutes and consumes 4.3 Wh of energy. In the case of SOLID, the liquid extract is directly incubated with an antibody microarray in the SAU, designed for detection of molecular biomarkers. Both units, SPU and SAU, require a top hierarchical unit to manage automatically the entire assay.

We have extensively tested the performance of the SPU-SEC under Martian environmental conditions, including electronics, fluidics (pump, valves, pipes and frozen liquids), ultrasonicator, and temperature and pressure sensors. Although not all components were space-qualified, the results showed that the whole system worked under Martian-like conditions and allow us to identify some critical aspects to be solved for the flight model.

SPU Technical Description: SPU processes the solid samples in order to extract the potential organics and biomolecules into a liquid solution or suspension for further analysis. This process consists of several steps: i) collecting a sample of soil or powdered rock;

ii) tightly sealing the chamber where the sample is collected (the Extraction Cell or EC); iii) addition of water-based liquid extraction buffer; iv) sonication the sample, that is, transmit to the sample ultrasonic vibrations that disgregate and homogenize it, extracting organics into the liquid and eventually lysing cells to release the intracellular content; v) moving the whole sample into a deposit/filter device where coarse material is retained and liquid extract with organics is released for further analysis to SAU.

Figure 1 shows a functional diagram of SPU, with valves, motors, sonicator, pump and temperature and pressure sensors in several locations of the unit.

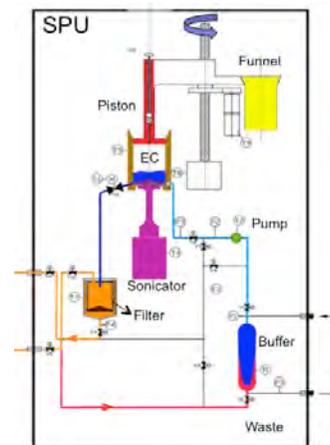


Figure 1. SPU functional diagram

A specific software controls the SPU, and interfaces between the user and the instrument. This software communicates via serial commands to SOLID to execute them sequentially. It can monitor and plot the periodically received measurements from the SPU-SEC, these data can be stored in files for further studies.

The SPU consists of four modules (Figure 2): i) The motion module, consisting of motors, encoders, sensors, limit switches; ii) The fluidics module, consisting of valves, pump, reservoir, filter, manifold and pressure sensors; iii) Sonication module, consisting of the extraction cell, the teflon membrane, and the piezoelectric probe that translates electrical energy into mechanical energy as vibrations that reach the sample via the membrane; and iv) Electronics module, which is the electronic board that controls all other modules. They are controlled by a processor that receives commands from a PC and sends the measurements of all

sensors distributed along the instrument. Besides all the components mentioned above, SPU also measures the signal of 11 RTD temperature sensors.

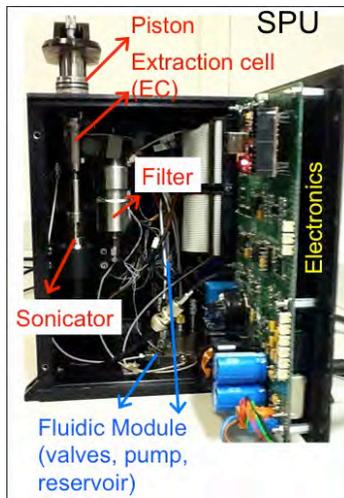


Figure 2. SPU showing the main elements

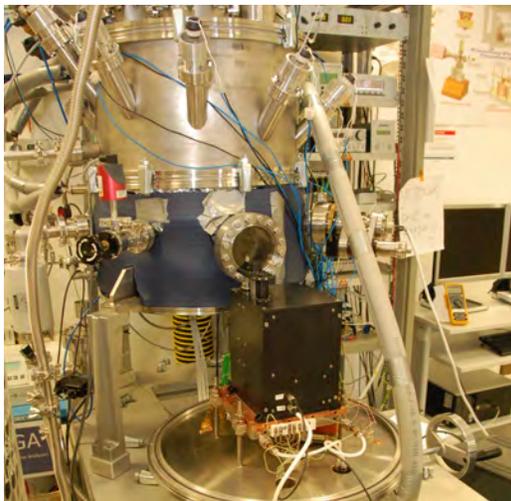


Figure 3. SOLID SPU tests in a Mars simulation chamber at CAB.

SPU Tests under Mars relevant conditions: To verify that all subsystem works properly, we have performed a comprehensive process with the SPU to visualize and review the results and measurements obtained. These tests were performed in a Mars simulation chamber [3] (Figure 3) under relevant temperatures and pressures. The set of tests are contained in three equal cycles (Figure 4):

1. At -20°C we tested the electronics and motion module performance, including the position, temperature, and pressure sensors, as well as motors.

2. At -40 °C, only tested the instrument storage, in off during the test.

3-4. The third and fourth trial is to test the sonication, and the fluidics modules. To test the fluidics we have to ensure that the liquid is not frozen. And to prove sonication we must be sure about working at minimum pressure.

5. The last trial of each cycle was at maximum temperature. In this case we only tested the reading and data sending by electronics.

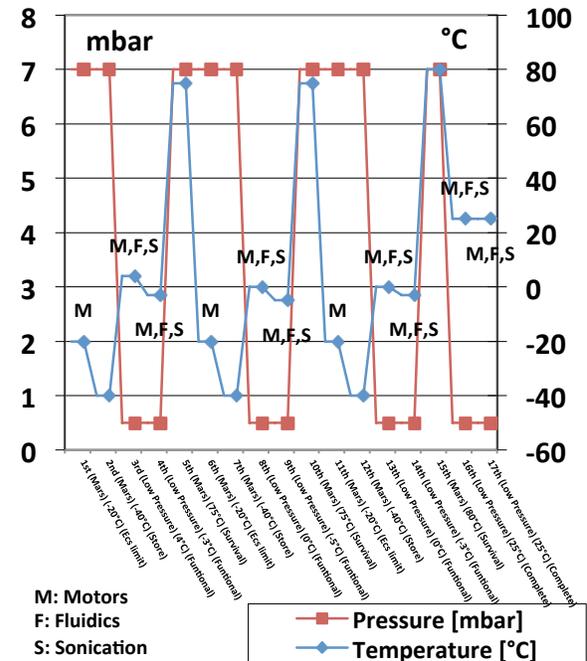


Figure 4. Testing SPU under Mars relevant conditions. At least 3 cycles were achieved to verify different components, functions, and sensors.

Conclusion: We have tested the subsystems and the whole SOLID SPU system under Mars relevant conditions. All the systems and components survived at least 3 cycles with temperatures from -40 °C to + 80 °C. Additionally, we successfully performed the functional tests below 1 mbar and -5°C including liquid pumping, ultrasonication, and filtering (Figure 4),

References: [1] McKay et al., (2013) *Astrobiology*, 13, 334-353. [2] Parro, V. et al. (2011) *Astrobiology* 11: 15-28. [3] Sobrado-Vallecillo, J. M. et al. (2014) *American Institute of Physics: Review of Scientific Instruments* 85: 035111.

Additional Information: Funded by Spanish Ministry MINECO No. AYA2011-24803.