VOLATILE ANALYSIS BY PYROLYSIS OF REGOLITH (VAPOR) FOR PLANETARY RESOURCE PROSPECTING.

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Introduction: Measuring the chemical composition of planetary bodies and their atmospheres is key to understanding the formation of the Solar System and the evolution of the planets and their moons. *In situ* volatile measurements enable a ground-truth assessment of the distribution and abundance of resources such as water-ice and oxygen, important for a sustained human presence on the Moon and beyond. The Volatile Analysis by Pyrolysis of Regolith (VAPoR) instrument is a compact pyrolysis mass spectrometer designed to detect volatiles released from solid samples that are heated to elevated temperatures and is one technique that should be considered for resource prospecting on the Moon, Mars, and asteroids.

There are several key lunar science and exploration measurement objectives that can be achieved by the VAPoR instrument: (1) Measure the abundance of water that can be released from lunar regolith for *in situ* resource utilization (ISRU) technology development, (2) .Measure the isotope ratios of carbon, hydrogen, oxygen, and nitrogen (CHON)-containing volatiles including water in polar regolith to establish their origin, and (3) Understand the processes by which terrestrial organic compounds are dispersed and/or destroyed on the surface of the Moon to prepare for future human exploration and life detection on Mars.

Here we will describe the VAPoR instrument concept that is based on the Sample Analysis at Mars (SAM) instrument on Curiosity currently operating on Mars and discuss the key science and resource prospecting measurement objectives enabled by evolved gas analysis.

Evolved Gas Analysis on Mars: The SAM instrument has been an essential tool in understanding the nature, abundance, and isotopic composition of volatiles and organics in Gale crater sediments [1]. The SAM instrument receives scooped or drilled soils or sediments from the rover sample acquisition system delivered to individual quartz cups. The cup is placed inside a pyrolysis oven under He flow at 25 mbar and heated at a rate of 35ºC/min up to ~860ºC. Evolved gases (e.g., H_2O, CO_2, O_2, SO_2) released from the sample are detected by a quadrupole mass spectrometer and their abundances determined from their characteristic masses and an estimate of the weight of the sample delivered to the oven. The SAM evolved gas analysis of eolian drift material called Rocknest have reported the detection of evolved water, SO_2, CO_2, O_2 and other trace volatiles (Fig. 1). The water abundances measured by SAM in Rocknest fines correspond to ~ 2.0 ± 0.5 wt.% [2]. Under the SAM heating conditions adsorbed water is released below 200ºC while bound water from amorphous phases, phyllosilicates, and hydrated salts and oxides are released at higher temperatures. Carbonate decomposition and oxidation of organics are the most likely sources of CO_2. Iron sulfates and oxidation of iron sulfides are possible sources of SO_2 and the oxygen releases are consistent with the presence of perchlorate and chlorate phases. Although SAM has a gas chromatography (GC) system, the GC was not required to determine the abundances of the major volatiles released from the sample.

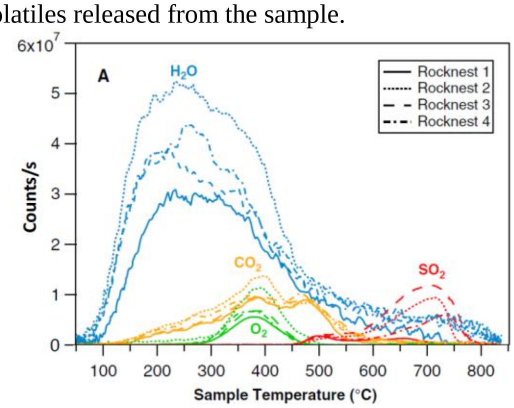

Fig. 1. SAM evolved gas analysis of four individual portions (50 ± 8 mg) of the Rocknest soil showing the major gases released as a function of temperature [2].

VAPoR Instrument: The VAPoR flight instrument concept based on SAM is shown in Fig. 2 and combines a sample carousel of up to six individually heated pyrolysis ovens with a time of flight mass spectrometer. The VAPoR gas processing system includes two gas manifolds, heated transfer lines, and two separate gas reservoirs for calibration of the mass spectrometer and oxygen for combustion experiments. One of the benefits of making evolved gas measurements on an airless body such as the Moon or an asteroid is that a vacuum pump is not required like it is on Earth or on Mars. Powdered rock or soil samples collected from a rover or lander drill or scoop and delivered through the solid sample tube to one of the VAPoR ovens can then be heated by a controlled ramp from ambient to temperatures up to 1400ºC to release the volatile constituents for direct measurement by the mass spectrometer. Two independent units have been built and tested to understand the performance of the different instrument components.

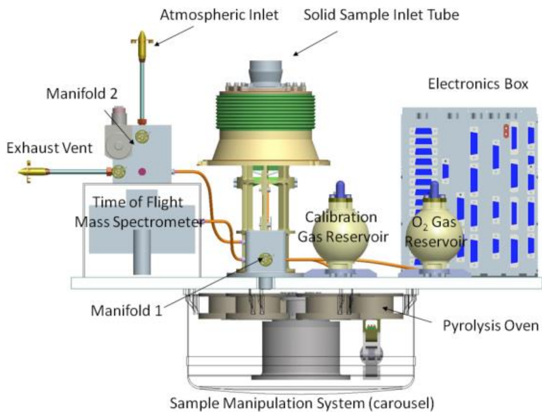

Fig. 2. Cross sectional view of the preliminary VAPoR flight instrument concept which combines a sample carousel containing six separate pyrolysis ovens integrated to a reflectron time of flight mass spectrometer for volatile analyses on the surface of planetary bodies throughout the Solar System.

A laboratory breadboard was developed to test, optimize, and calibrate the reflectron time of flight mass spectrometer (TOF-MS) component of VAPoR inside a separate vacuum chamber and is discussed in more and elsewhere [3]. A separate portable field unit (Fig. 3) consisting of a custom made pyrolysis oven coupled to a commercial RGA quadrupole mass spectrometer, vacuum manifold and turbomolecular pumping station, was built to demonstrate the feasibility of conducting vacuum pyrolysis evolved gas measurements on Apollo samples and meteorites in the laboratory as well as analyses in the field [4,5].

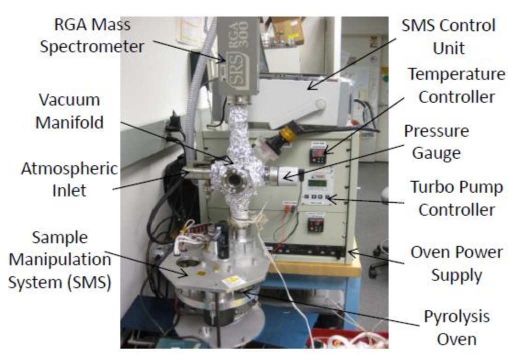

Fig. 3. The VAPoR laboratory and field instrument, which includes a new sample manipulation system and high temperature pyrolysis oven for evolved gas analysis of powdered solid samples.

The instrument is set up to enable direct line of sight from the heated sample in an oven to the ionization region of the RGA mounted at the top of the manifold. The RGA was setup to scan over the mass range 1-100 amu during the pyrolysis heating experiment. The current VAPoR field instrument does not include the TOF-MS, which is being tested and optimized separately in a larger vacuum chamber at NASA GSFC.

The custom VAPoR ovens are designed to bring solid samples up to a maximum temperature of 1400° C in order to release high temperature volatiles such as O_2 from the breakdown of silicates, SO_2 from the decomposition of gypsum, and high temperature noble gas releases of Ar, Kr, and Xe. The oven design was based off of the design of the two flight pyrolysis ovens in the SAM instrument suite, but unlike the SAM oven design where a cup containing solid powder is raised up into the oven, sample can be dropped directly inside the VAPoR alumina crucible which enables direct heating of the sample and lower power to achieve the desired temperature.

Conclusions: *In situ* pyrolysis evolved gas measurements of the lunar regolith and other airless bodies including asteroids are needed to characterize and determine the nature, distribution and abundance of volatiles, particularly, water, an important resource for future human exploration. SAM has demonstrated that *in situ* abundance measurements of water and other major volatiles released from martian surface samples by heating can be made by evolved gas analysis. Using the VAPoR instrument during NASA's 2011 Desert RATS field campaign, we successfully demonstrated that high temperature vacuum pyrolysis of solid samples to temperatures exceeding 1300°C coupled with line of sight detection of volatiles by mass spectrometry can be used for the identification of resources including water and oxygen in powdered rock samples [4]. The inclusion of volatile analysis capability in the field and continued testing of instruments such as VAPoR in future field tests will be critical to the success of future robotic and human planetary resource exploration missions.

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