

Volatile Analysis by Pyrolysis of Regolith in the 2011 D-RATS Field Test. C.A. Malespin^{1,2}, D.P. Glavin¹, I.L. ten Kate³, H. B. Franz^{1,4}, E. Mumm⁵, J.E. Bleacher¹, and J.W. Rice¹, ¹NASA Goddard Space Flight Center, 8800 Greenbelt Rd, Greenbelt, MD 20771, (daniel.p.glavin@nasa.gov), ²Universities Space Research Association, 10211 Wincopin Circle, Columbia, MD 21044, ³University of Oslo, Sem Saeland vei 24, NO-0316 Oslo, Norway, ⁴Center for Research and Exploration in Space Science and Technology, 5523 Research Park Drive, University of Maryland Baltimore County, Baltimore, MD 21228, ⁵Honeybee Robotics, 460 34th Street, New York, NY 10001

Introduction: The Volatile Analysis by Pyrolysis of Regolith (VAPoR) instrument is a compact vacuum pyrolysis mass spectrometer designed to detect volatiles such as water and organic compounds released from rock and soil samples that are heated to temperatures over 1300°C. VAPoR was used to analyze crushed rocks and soils collected by a crew of astronauts and geologists during the Desert RATS field operations near Black Point Lava flow in Arizona. This evolved gas data from VAPoR can then be used to select the most volatile-rich samples for large scale *in situ* resource utilization (ISRU).

Instrument description: The VAPoR field unit, shown in Fig. 1, uses a six-port stainless steel vacuum manifold connected to a six-position sample manipulation system (SMS, designed by honeybee robotics). The SMS contains two custom made high temperature pyrolysis ovens developed at the NASA Goddard Space Flight Center.

The vacuum manifold is connected to an atmospheric inlet, cold cathode ion pressure gauge and a commercial residual gas analyzer (Stanford Research Systems, RGA 300), all of which are actively pumped using a turbo molecular pumping system.

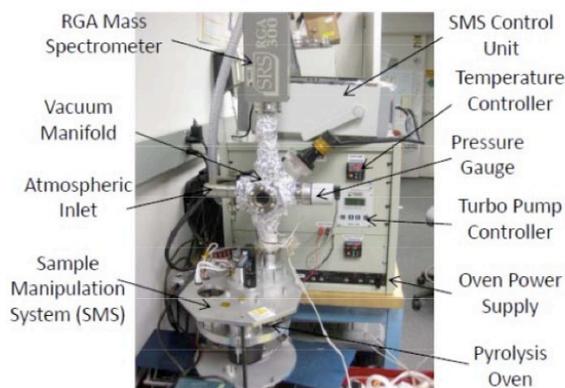


Figure 1. The VAPoR field instrument. Includes an SMS and high temperature pyrolysis oven for evolved gas analysis of powdered solid samples

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

O₂ and the noble gases Ar, Kr, and Xe [1,2]. The oven design was based on the design of the two flight pyrolysis ovens in the Sample Analysis at Mars (SAM) instrument suite currently on the Mars Science Laboratory, 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 required to achieve the desired sample temperature.

Procedure: Before each solid sample was pyrolyzed under vacuum, a procedural blank of fused silica inside an empty quartz sample tube with quartz wool was measured. The empty tube was heated from ambient temperature up to 1300° C at 30° C/min. This provided a background for each sample run, as seen in Figure 2.

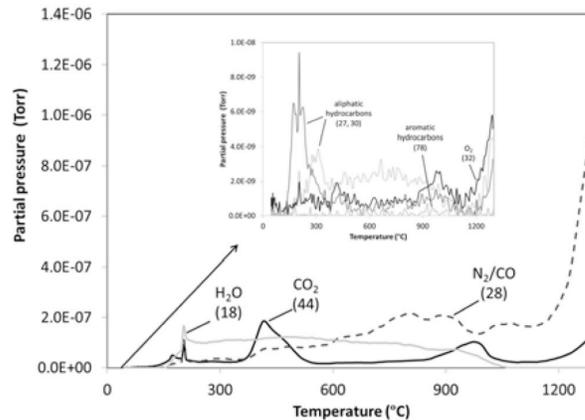


Figure 2. Procedural Blank. Evolved gas profile of empty quartz sample holder heated to 1300° C. m/z values are in parentheses N₂ and CO₂ cannot be separated by the RGA used and are therefore plotted together

Samples were collected by the DRATS field team consisting of a crew of astronauts and geologists, using clean metal tongs and wrapped in ultrahigh vacuum foil prior to bagging in polyethylene bags in order to minimize hydrocarbon contamination during collection. Each sample was then crushed, sieved to < 150 μm, and portioned into 10-20 mg aliquots to be trans-

ferred to a clean quartz tube for analysis. Once the sample is loaded in the pyrolysis oven, the SMS rotates the oven directly underneath the mass spectrometer and lifts the oven vertically to form a vacuum tight knife edge seal against the vacuum manifold and then the turbopump is turned on to pump down the manifold to a pressure of $< 10^{-7}$ mbar.

During the DRATS field test, the VAPoR oven was ramped from ambient to 1100° C at a rate of 20° C/min while the RGA monitored the volatiles evolved from the sample using continuous unit mass scans of m/z 2-100. Individual masses were subsequently plotted as a function of temperature to obtain an evolved gas plot for analysis of the sample.

Results: Here we show the results of a selected sample collected during the DRATS field test. DRATS sample 0212 was a basalt containing up to 3 mm diameter vesicles. A portion of this sample was chipped off for VAPoR analysis concurrently with the other instruments which participated in the DRATS test.

Figure 3 shows selected inorganic and organic volatiles released from sample 0212 as a function of temperature. The primary volatile released between 300-600° C was CO₂ (m/z 44) which is from the breakdown of both organic compounds and carbonate minerals. The low decomposition temperature of carbonate corresponding to the peak CO₂ around 450° C is due to the fact that VAPoR is operating at lower pressure than atmosphere, a finding noted by Kabanov [3].

Hydrocarbons were released from the sample at temperatures above 200° C, consisting primarily of alkanes as indicated by the m/z 27 and m/z 30 characteristic alkane hydrocarbon mass fragments. Some aromatic hydrocarbons, such as benzene (m/z 78), were seen in trace amounts.

Several water peaks at m/z 18, indicate the presence of multiple hydrated mineral phases, possibly hydrous oxides or phyllosilicates, as well as hydrated sulfate minerals as evidenced by the high temperature SO₂ peak seen at 960° C.

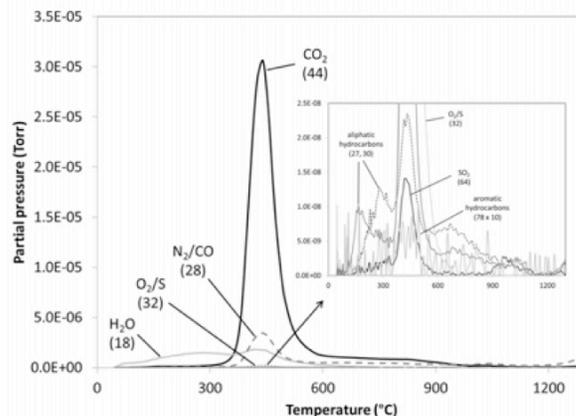


Figure 2. VAPoR evolved gas analysis of DRATS sample 0212, a vesicular basalt. m/z values are in parentheses.

Future Work: In order to identify trace volatiles of interest with similar masses and to make isotopic measurements we are currently developing a miniature reflectron time of flight mass spectrometer (TOF-MS) for VAPoR. that will replace the commercial RGA currently used. We are also developing an EGA library of mineral standards and analogs, will be used in conjunction with the new TOF-MS for future field tests.

References: [1] Lightner B. D. et al, presented at the Proceedings of the Fifth Lunar Science Conference, 1974. [2] Bogard D. et al, presented at the Proceedings of the Sixth Lunar Science Conference, 1975. [3] Kabanov A. (2006) *Russian Journal of Applied Chemistry*, 1070-4272, 711-714.