

THERMAL AND EVOLVED GAS ANALYSIS OF GEOLOGIC SAMPLES CONTAINING ORGANIC MATERIALS: IMPLICATIONS FOR THE 2007 MARS PHOENIX SCOUT MISSION. H. V. Lauer Jr.¹, D. W. Ming², D. C. Golden³, and W. V. Boynton⁴; ¹ESCG/Barrios Technology, Houston, TX 77258 (howard.v.lauer1@jsc.nasa.gov); ²ARES NASA/JSC, Houston, TX 77058 (douglas.w.ming@nasa.gov); ³ESCG/Hamilton Sundstrand., Houston, TX 77258, ⁴University of Arizona, Tuscon, AZ 85721

Introduction: The Thermal “and” Evolved Gas Analyzer (TEGA) instrument scheduled to fly on-board the 2007 Mars Phoenix Scout Mission will perform differential scanning calorimetry (DSC) and evolved gas analysis (EGA) of soil samples and ice collected from the surface and subsurface at a northern landing site on Mars. We have been developing a sample characterization data library using a laboratory DSC integrated with a quadrupole mass spectrometer to support the interpretations of TEGA data returned during the mission. The laboratory TEGA test-bed instrument has been modified to operate under conditions similar to TEGA, i.e., reduced pressure (e.g., 100 torr) and reduced carrier gas flow rates [1,2]. We have previously developed a TEGA data library for a variety of volatile-bearing mineral phases, including Fe-oxyhydroxides, phyllosilicates, carbonates, and sulfates [1].

Here we examine the thermal and evolved gas properties of samples that contain organics. One of the primary objectives of the Phoenix Scout Mission is to search for habitable zones by assessing organic or biologically interesting materials in icy soil. Nitrogen is currently the carrier gas that will be used for TEGA. In this study, we examine two possible modes of detecting organics in geologic samples; i.e., pyrolysis using N₂ as the carrier gas and combustion using O₂ as the carrier gas.

Materials and Methods: Green River Shale (SGR-1, U.S. Geological Survey) was used as a geologic standard that contains organics. SGR-1 contains about 28 wt % total C (~3 wt % inorganic C in the form of carbonates and 25 wt % organic C in the form of kerogen). Samples for thermal and evolved gas analyses (TA/EGA) were prepared by diluting the SGR-1 with ultra pure silica powder that had first been heated in air to 1000°C to combust any organics to formulate samples containing 10 wt % SGR-1 (2.5 wt. % organics).

We have modified our normal TEGA test-bed [1] by removing the DSC and replacing it with a closed system quartz chamber inserted in a vertical tube furnace that will reach 1100°C (laboratory DSC only obtains temperatures to 725°C). With this setup we are able to more closely reproduce the flight TEGA geometry and operating conditions with the exception of heat flow measurements. All analysis were pre-

formed at 100 torr pressure using either O₂ or N₂ as the carrier gas. In preliminary experiments, the quadrupole mass spectrometer was set to scan all mass fragments from 1 to 200 amu to determine organic fragments evolved during either pyrolysis or combustion. Based upon these preliminary experiments, 10 individual mass fragments were selected and monitored during EGA experiments.

Results: As would be expected, there are significant differences in the evolved gas behaviors for samples run under combustion and pyrolysis conditions (Figs. 1 & 2). In combustion experiments, organic C in the form of CO₂ and H₂ evolved between 300-600°C (Fig. 1); whereas, CO₂ and H₂ evolved between 450-600°C in pyrolysis experiments (Fig. 2). CO₂ that evolved between 650-750°C is primarily due to the thermal decomposition of dolomite, although, some of the CO₂ evolved in this range for the pyrolysis experiment may be due to decomposition of the kerogen (see below). Nearly all of the kerogen was “cracked” into CO₂, H₂, and H₂O in the combustion experiments with the exception of a mass 41 fragment that we have assigned to C₃H₅ and mass 15 that we assigned to methane (Figs. 3 & 4). In addition a small amount of mass 55, 67 and 70 was detected but not included because of space limitations. The methane peak for the combustion experiment has a high sloping background because O₂ is also detected at mass 15 from a small mass 16 drift in our quadrupole mass spectrometer [4].

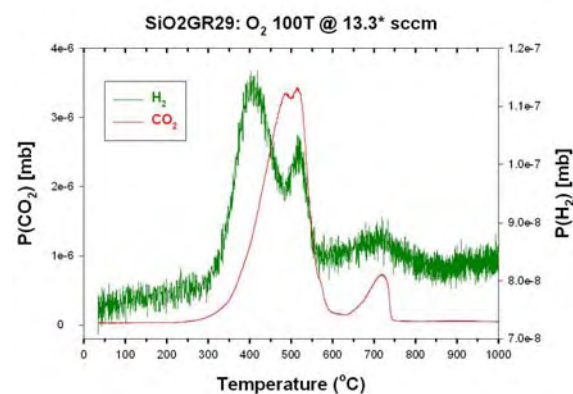


Figure 1. Evolved CO₂ and H₂ for a combustion experiment (O₂ carrier gas) from a sample containing 10 wt % SGR-1.

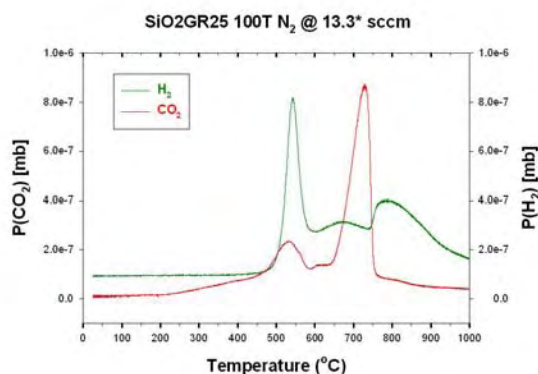


Figure 2. Evolved CO_2 and H_2 for a pyrolysis experiment (N_2 carrier gas) from a sample containing 10 wt % SGR-1.

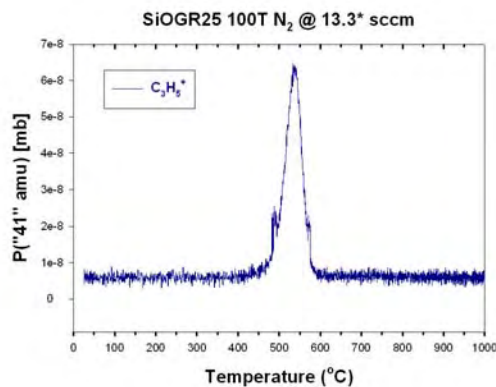


Figure 5 Evolved mass 41 molecule during pyrolysis experiments was assigned as C_3H_5 .

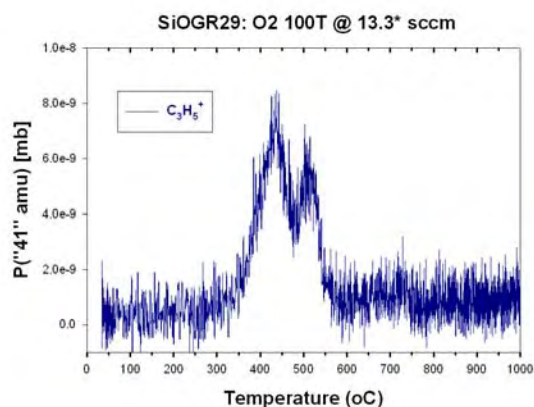


Figure 3. Evolved mass 41 molecule during combustion experiments that was assigned as C_3H_5 .

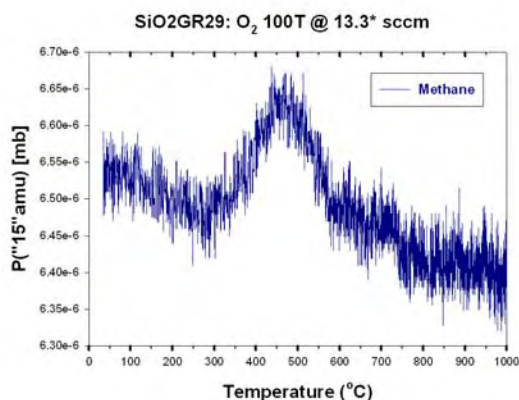


Figure 4. Evolved mass 15 molecule that was assigned as methane in combustion experiments.

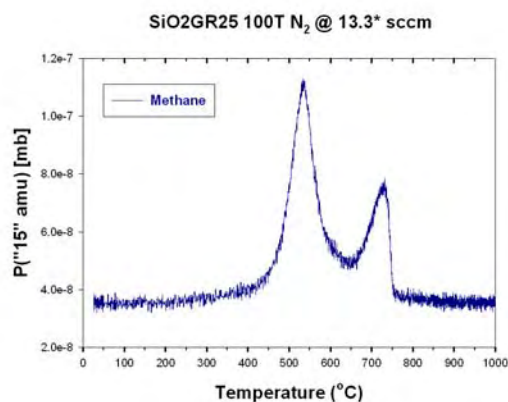


Figure 6. Evolved mass 15 molecule that was assigned as methane in pyrolysis experiments.

There were very well-defined releases of C_3H_5 and methane during the pyrolysis experiments that used N_2 as the carrier gas (Figs. 5 & 6). No doubt, H_2O and some O_2 released during the pyrolysis experiments that will result in some combustion of the kerogen material in SGR-1. However, the N_2 carrier gas “preserves” some of the fragments compared to the O_2 carrier gas that promotes combustion.

Implications for 2007 Mars Phoenix Mission.

The N_2 carrier gas that is base-lined for TEGA on Phoenix should aid in “preserving” organics (i.e., fragments) that may occur in the icy soil. Additional experiments are underway to look at these systems with trace amounts of organics (e.g., 25 ppm organic C) as well as experiments using mixtures of N_2 and trace reducing gases (e.g., CO) that may further “preserve” organics that fragment during pyrolysis.

References: [1] Lauer Jr. H. V. et al. (2000). *LPSC XXXI*, Abst. # 1990, (CD-ROM). [2] Golden D. C. et al. (1999) *LPSC XXX*, Abst. # 2027 (CD-ROM). [3] Vasgaard J. L. (2005) person. comm.