Using stable isotope geochemistry to investigate the source(s) of volatiles in the lunar regolith

Conference Item

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1: Introduction:

- Previous laboratory analyses of lunar soil samples have documented a range of volatile species present within the regolith that blankets the lunar surface.
- Thermal gas release studies (heating soil samples at rates of 4 °C/min up to temperatures just exceeding their initial melting points) released solid gases, CH4, CO, CO2, H2O, H2, and subliming species like Na, K, and Ba. The temperatures at which these gases are released can be used to tentatively identify their sources, for example, solar wind-deriv ed hydrogen and helium release is well established at 200-300 °C [2].
- Acid and deuterium dissolution studies (heating soil samples with HCl and D2O) released several gasses of ‘volatile’ methane (CH4) and water produced CO2, in situ from hydrocarbon sources (C60) is carbon associated with microcrystalline pyrolysis (H2) contributed by remnant impacts and solar wind interactions [6]. Such mechanisms and/or compounds are concentrated in the fine fractions of lunar soils (less than 10 μm diameter) located on the submicrocrystalline layer of grains [8].
- Isotopic studies have also been conducted, isotopic values are expressed using the delta notation, where:
  \[ \delta^13C = \frac{R_{sample}}{R_{standard}} - 1 \times 10^3 \]
- These studies have revealed that total C δ13C values for lunar soils range from -30 ‰ to +30 ‰. Given that the values for possible terrestrial contaminations range between -30 ‰ to +30 ‰ and that end member solar wind carbon has a calculated δ13C value of -15 ‰ to -15 ‰, it was suggested that lunar soil carbon is most likely from a meteoritic/cometary source [9]. However, this does not take into account carbon contamination, or the individual isotopic variations of carbon-bearing gasses in lunar soils.
- Therefore, focusing on CH4 or CO2, the overall aim of this project is to measure its carbon isotopic signature as a means of fingerprinting the likely source(s) of carbon.
- Given that methane in lunar soil is calculated to make up only 5% to 25% of the total carbon in lunar soils [4], such isotopic measurements will need to be performed on highly sensitive instrumentation, capable of handling relatively small amounts of gas sample.

2: IonCam 2020 Gas Analysis Mass Spectrometer

- The IonCam 2020 mass spectrometer was purchased from OI Analytical (of Alabama, USA) in the autumn of 2012.
- It is a miniature, transportable, non-scanning mass spectrometer allowing for simultaneous detection of all masses present in gas samples.
- The VSCCP detector (Charge Coupled Device) within the machine is described as pressure independent, and highly linear, suggesting that it should function well with small amounts of gas (ideal for lunar soil sampling).
- A linear response here would mean that gas flow (measured as gas intensity at the detector) should be proportional to pressure squared.
- It is capable of performing full analyses of samples, which allow transient processes to be monitored and recorded.

4: Stability

- In order to make reliable, precise ion mass spectrometers, the response seen on the detector needs to be as stable as possible over the time it takes to make a measurement (in this case, over 5 minutes).
- Given that δ13C values from previous studies of lunar soils range from -30 ‰ to +30 ‰, it is essential to distinguish between different potential sources of carbon, a variation over measurement time of ± 3 ‰ would be ideal.
- To ensure the stability of the measurements, the IonCam makes data collected at the detector’s reference level, which displays peaks at masses 44, 45, and 46 as a mass spectrum.
- Data was collected over several weeks, in both ‘raw’ form (measured intensity in each pixel on the CCD array) and ‘detected’ form (measured intensity at the detector). This allows for comparison of the two forms to check for any changes in the machine’s performance over time.

5: Suitability

- Using these results to make some rough calculations, based on the expected yield of volatiles containing carbon from lunar soils, the IonCam would only need around 30-60 minutes mass sample in the region of hundreds of micromoles per measurement to make one ion mass spectrometry measurement that shows high precision established mass spectrometry measurements used in the open literature. However, the amount of sample gas needed to build up enough pressure to 50 Torr (to make such an ion mass spectrometry measurement) must be taken into account, which takes the amount of carbon needed up to twice as much, for example, for a 50 Torr measurement, which would be expected to be an acceptable amount for such a measurement.
- Further, in this case, the δ13C results are calculated using ion mass spectrometry measurements with a variation of ± 3 ‰. Knowing that the whole range of valid values for possible solar system sources of carbon is only ± 30 ‰, it would be impossible to distinguish between different sources for the carbon found in lunar soils, using the IonCam in its current performance.
- There is also a number of other issues surrounding the use of the IonCam instrument for the IonCam work, one of which is poor resolution between peaks of similar masses, leading to tailing peaks which interfere with the measured isotopic ratios of neighboring peaks. Another problem is the occasional instability of a ‘dead’ or ‘stabilized’ response from the detector; this is a problem for the gas inlet pipes and the gas inlet itself. This requires further investigation.
- To conclude, in its current configuration, the IonCam 2020 instrument is not suitable for use with lunar soil samples. Making some fairly easy changes, such as reducing the initial volume of the reference gas line, and making mass measurements over a shorter time period would reduce the amount of sample needed by a factor of 100; for example, but to fully resolve the machine’s various issues, more in depth modifications (perhaps a high-quality gas chromatographic column, or replacing the pump with a more stable detector) are necessary. Such an instrument would need to be calibrated or checked for other issues before being used at the Open University in the future, although the uptake of the lunar geochemistry project does not.

6: Other Instrumentation

- There are several well-established instruments at the Open University that are capable of making highly precise isotopic measurements on small sample sizes, which are the next possibilities to explore.
- The first of these is ‘Rusker’, a custom-built instrument which incorporates an X-ray photoelectron spectrometer to determine elemental concentrations of solids for identification of rock samples.
- The second instrument suitable for use with lunar soil samples is the Thermo MAT 253 isotope ratio monitoring gas chromatographic mass spectrometer (IRMS-GCMS). Although with this machine, the initial proportion of the lunar soil samples can be difficult.
- Further experiments are made before the IonCam, the IonCam can be used to measure its sensitivity for this particular project.

7: References