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

Conference or Workshop 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.
- Thermo gas release studies (heating soil samples at rates of 4 °C/min up to temperatures just exceeding their initial melting points) revealed solid gasses, CH₄, CO, CO₂, H₂O, HD, and sublimed-bearing species Na₂S and SiO₂. The temperatures at which these gasses are released can be used to tentatively identify their source, for example, solar-wind-derived hydrogens and helium are released before 300–500 °C.[2]
- Add and subtracted null isolation studies (heating lunar soil samples with He, CO, and CO₂) released several gasses of low isotopic purity. CH₄ and reaction-produced CO₂, both from hydrocarbon-bearing carbon (C₀ in carbon associated with nanophase Fe₃O₄) contributed by anastomosis and other wind derived hydrogen and helium are released between 30–80 °C.[3, 2]
- Isotopic studies of these samples have also been carried out, and isotopic values are expressed using the delta notation, where:

\[
\delta = \frac{R_{sample} - R_{standard}}{R_{standard}} \times 10^3
\]

where \( R_{sample} \) and \( R_{standard} \) are the ratios of two isotopes of a given element.

- Some of these studies have revealed that total C-4 isotopes for lunar soils range from -30 ‰ to +30 ‰.[5] Given that the values for possible terrestrial contaminations range between -30 ‰ to +30 ‰,[5] and that end member solar wind gasses have a calculated 4°C value of -85 ‰ to 75 ‰,[7] it was suggested that lunar soil carbon is most likely derived from a meteoritic/terrestrial source.[4]
- Therefore, focusing on CH₄ as a lunar soil, the overall aim of this project is to measure its carbon isotopic signature as a means of fingerprinting the likely sources of carbon.
- Given that methane in lunar soil is calculated to make up only around 1–5% 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 samples.

In that end, this paper describes initial research work carried out on the Ioncam 2020 mass spectrometer at the Open University, to assess its suitability for use with lunar samples.

2: Ioncam 2020 Gas Analysis Mass Spectrometer

- The Ioncam 2020 mass spectrometer was purchased from O.H. (Analysts of Alabama, USA) in the autumn of 2012.
- It is a miniaturised, transportable, non-scanning mass spectrometer, allowing for simultaneous detection of all masses present in a gas sample.
- The VnCDP detector (Charge-Coupled Device) within the device is described as pressure independent and highly linear,[8] suggesting that it should function well with small amounts of gas (ideal for lunar soil samples).[4] A linear response here would mean that gas flow (measured in gas density at the detector) should be proportional to pressure.
- It is capable of performing fast analyses of samples, which allow transient processes to be monitored and recorded in real-time.

- The sample inlet system for the Ioncam 2020 mass spectrometer is designed to deliver a continuous flow of reference gases, via an inlet capillary, and then the spectrometer, where the mass spectrometer is housed.
- The inlet capillary is set at a flow rate of 100 μl/min, which is sufficient to carry the gas sample into the mass spectrometer at different times. Initial test runs (using pure 100% methane) conducted recently have shown a variation in the measurements of ± 2%.[5]
- Gas samples containing methane, carbon dioxide, helium, and hydrogen are released from the sample source at rates of 4 μl/min to the mass spectrometer at the Open University, Milton Keynes, using a gas delivery system that requires further investigation.

3: Detection Limit

- For purposes of making an isotope measurement, the detection limit of the machine is taken to be the smallest amount of gas needed to generate a stable, measurable peak in the mass spectrometer for the least abundant isotope, in this case 4°C, 85‰.
- To build up mass spectra at a range of different pressures, 20 Torr pressure was used. A high pressure of 20 Torr was selected to ensure that the peak for mass 46 disappears at around 20 Torr, while the peak for mass 44 remains visible for comparison purposes.[6]
- The standard reference gas used to characterise the machine’s performance was carbon dioxide (CO₂), which was shown to be highly sensitive and capable of detecting small amounts of gas (ideal for lunar soil samples).[4]

Given that δ4°C values from previous studies of lunar soils range from -30 ‰ to +30 ‰,[5] it is essential to distinguish between different potential sources of carbon, a variation over measurement time of + 2% would be acceptable.

4: Stability

- To determine the machine’s sensitivity to small changes in the flow rate of volatile species, several dynamic tests were carried out to assess the device’s sensitivity to small changes in pressure, which is greatest for volatile species such as CO₂, CH₄, and CO.[4]

5: Suitability

- Using these results to make some rough calculations, based on the expected yield of volatile species containing carbon from lunar soils, it is calculated that the Ioncam 2020 mass spectrometer should have high precision documented at the Open University, Milton Keynes, to ensure that the amount of gas needed to build up a stable 4°C peak in the mass spectrometer can be taken into account, this takes the amount of carbon needed up to 100 μl/min for a few less than 4°C.[4]

6: Other Instrumentation

- There are several well-established instruments at the Open University that are capable of making highly precise isotopic measurements on small samples, such as those on the list below:

7: References