

In-Situ Reaction Monitoring for Lunar Applications Utilizing a Single Quadrupole Residual Gas Analyzer

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Project Objective

To develop a calibration methodology for carbon monoxide (CO) and carbon dioxide (CO₂) utilizing a commercial off-the-shelf (COTS) residual gas analyzer (RGA) to quantitate reaction products extracted from lunar regolith via carbothermal reduction reactor. Qualitative data will also be collected to evaluate the presence of solar implanted volatiles from in-situ regolith samples on the lunar surface.

Introduction

With a revived focus to create a permanent presence on the moon and in preparation for future Mars exploration, it is imperative that all resources are utilized to their fullest potential.[1] In-Situ Resource Utilization (ISRU) will be critical for future mission success as it would enable independent operation of missions while minimally relying on the complex supply chain created between the Earth, moon and Mars.

One of the most critical resources that have been identified for ISRU is the creation of liquid oxygen (LO₂) for not only breathable air, but also for rocket propellant. On the moon and potentially Mars, one of the most O₂ rich resources is in the presence of regolith.[2],[3] Although there are numerous minerals within regolith and various processes to extract O₂, this is not the scope of this poster.

Problem Statement

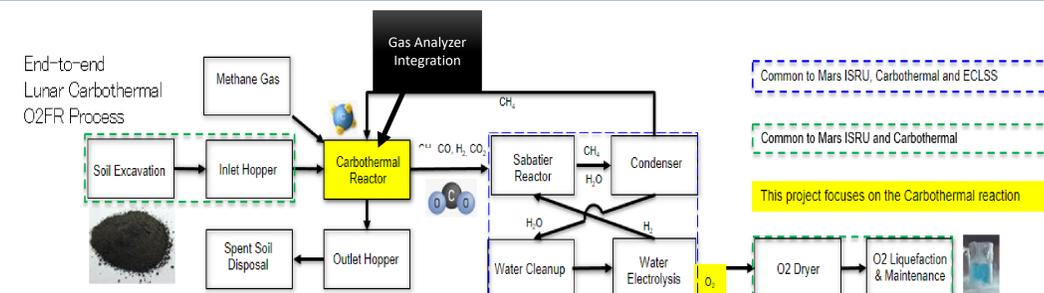
Solar wind implanted volatiles and various other compounds present in lunar regolith, such as nitrogen (N₂), helium (He), and deuterium (D₂), present a problem when analyzing using mass spectroscopy alone. Deconvolution and quantification of isobaric compounds becomes quite difficult when target compounds have similar fragmentation patterns as other species.

Specifically, the presence of N₂ makes quantification of CO, an oxygenated precursor, difficult due to both compounds having a peak at m/z 28. Furthermore, because CO₂ and other gases like ethane, can have fragmentation products at m/z 28 it becomes evident that a method distinguishing the parent compound needs to be established. Initially, the solution to overcome isobaric overlap was to use isotopic ratios, particularly between carbon-13 and nitrogen-15. Although limited literature discuss isotopic composition and attempt to quantify the ratios, variation is particularly high and limited to only a few lunar regolith samples. [3]

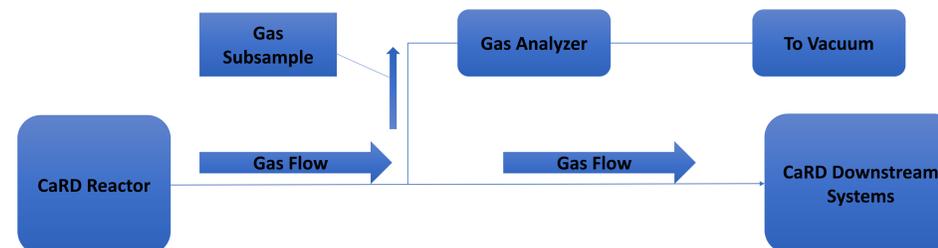
System Overview

Since the O₂ extraction method can vary, a critical standardized analytical method is needed to verify O₂ (or oxygenated precursor compounds) for extraction efficiency and purity. Our group utilizes a modified consumer off-the-shelf (COTS), Residual Gas Analyzer (RGA) Single Quadrupole Mass Spectrometer (QMS). Due to the current field mission requirements, the gas analysis module will need to rely only on mass-to-charge ratios (m/z) and peak intensities to differentiate and quantitate targeted gas-phase reactants or products, i.e., carbon monoxide (CO), and carbon dioxide (CO₂).

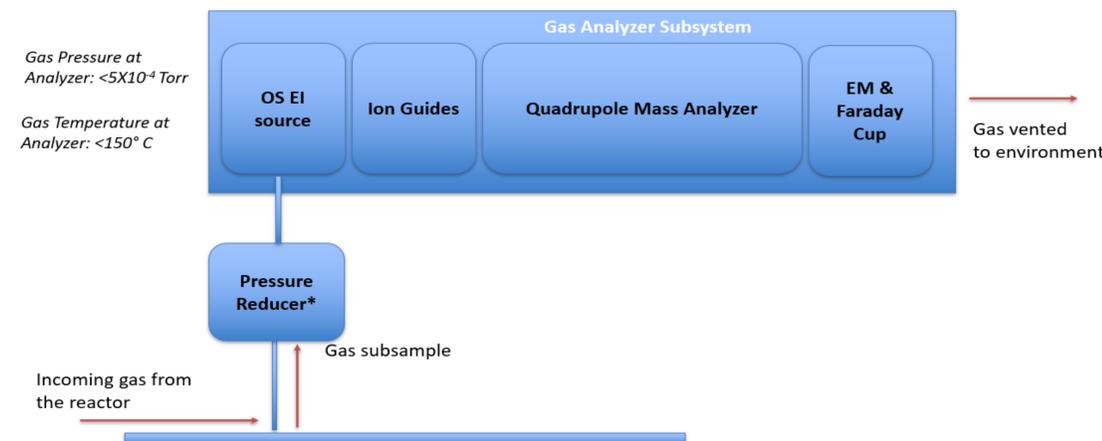
Concept of Operation (ConOps)



➤ End-to-End Carbothermal Process: Residual Gas Analyzer (gas analyzer) would be integrated in at the reactor output to analyze gases as close to the reactor as possible



- The process gas flow is outlined in the schematic above: the gas analyzer will “sip” a subsample as the bulk gas flows to other downstream systems
- Gas then will be analyzed by the gas analyzer and any remaining subsample will be vented out of the system to vacuum



- Since the process gas would be at relatively high pressure compared to the operating pressure of the residual gas analyzer, which is $< 5 \times 10^{-4}$ Torr, a pressure reducer will need to be utilized to ensure safe operation of the RGA.
- The pressure reducer will consist of vary sized tubing and/or frit capable of reducing the pressure by creating a calibrated leak through a precision cut orifice.

Quantification Approaches

Approaches to differentiate isobaric components

Ionization Energy Variation

CO and N₂ both have peaks at m/z 28, but only N₂ can produce a peak at m/z 7. [4] While a N₂ peak at m/z 7 is generally rare, instrument parameters can be optimized in order to maximize the peak intensity. Typically, a standard ionization energy of 70 eV is used to monitor a batch process and would produce reliable results for most of the reactants or products that we are interested in quantifying. However, to analyze the rare molecular fragment of N₂ (N₂⁺ at m/z 7), a different ionization energy may be beneficial. Ionization Energy Variation will be explored to optimize the N₂⁺ peak at m/z 7 by varying parameters such as ionization energy and pressure. Therefore, a quantitative value at m/z 7 will be used to determine the product gas concentration of N₂, which will be subtracted from the total concentration determined at m/z 28 enabling us to approximate the amount of CO.

Secondary Detector (infrared gas detector, IR).

Another approach to quantitate and differentiate between isobaric components is to leverage alternative modes of detection. Unlike mass spectrometry, which generally uses a separation step prior to analysis (i.e., gas chromatography), IR produces energy that is specifically absorbed depending on the bond and configuration of the molecule. This technique could be leveraged since it is generally only active on non-symmetric molecules, i.e., CO. [5] Utilizing concentrations of CO determined from another detector could help correct concentrations of CO obtained from the QMS. However, the inclusion of another detector adds additional mission requirements, such as power, software changes and avionics updates that add additional cost and schedule requirements.

Other Approaches being explored

Multivariate analysis could potentially be used to differentiate between isobaric species as demonstrated by Binninger, et al. [7]

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