

**Contract No:**

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Overview & Relevance

- Develop a novel, rugged, highly sensitive interferometric spectrometer that will support a real-time, and chemically-specific imaging technique to improve optical process analysis capability.
- The Spatial Heterodyne Raman Spectrometer (SHRS) is a recently developed technology for optical spectroscopy that promises enhanced sensitivity and new opportunities for process and field measurements compared to conventional spectrometers. Sensitivity gains of 10-100x are obtained through light collection over a wide field of view and measurement across a two-dimensional detector array. With no moving parts, a SHS can support the use of typically delicate laboratory instrumentation in a field environment in small, more rugged package (Fig.1).
  - The SHRS offers real-time imaging capability for all sample types and is amenable to wide area detection for process analysis, such as fugitive emissions from industrial processes or online process analysis.
    - Saltstone disposal unit 6 (SDU6, Fig.2) requires controls to ensure the flammable gas concentration limit for a list of chemicals is not exceeded
    - Calculated estimates of gas concentrations are very conservative and experimental measurements are needed to provide accurate gas concentrations to fill the tank with the appropriate amount of waste without exceeding the concentration limits.
    - In-situ measurements ideal; current tests require pulling a sample and sampling loop does not work as intended.
  - Responsive to ES interest in advanced techniques for characterizing conditions or contaminants of concern and/or for treating in-situ in hazardous or difficult-to-access environments.
  - FY18 Call focus area: Advanced Imaging Technologies

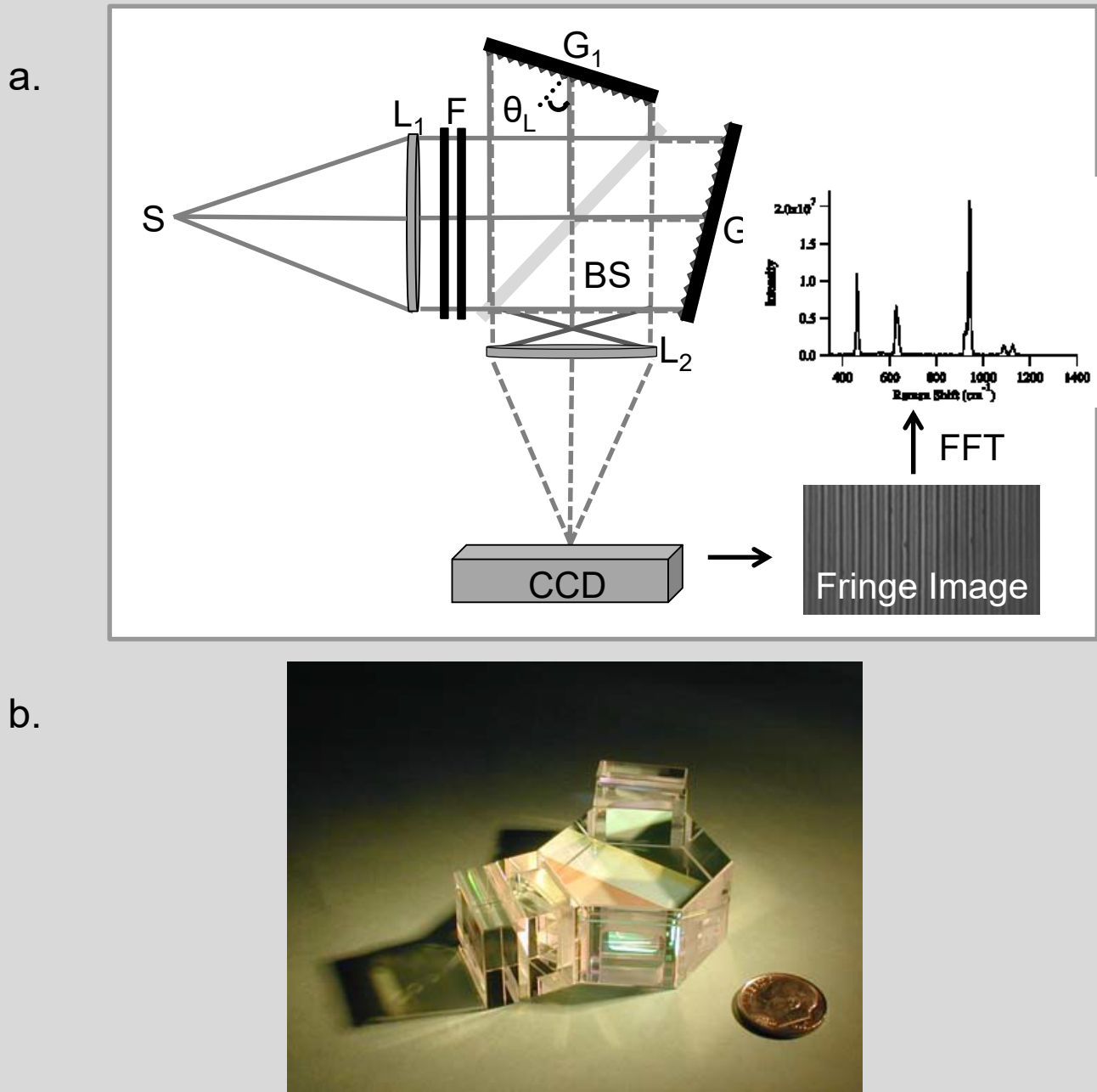


Figure 1: a) SHRS schematic; fringe image, and Raman spectrum from fast Fourier transform of fringe image. b) Image of SHIMMER, a SHS deployed on a satellite.

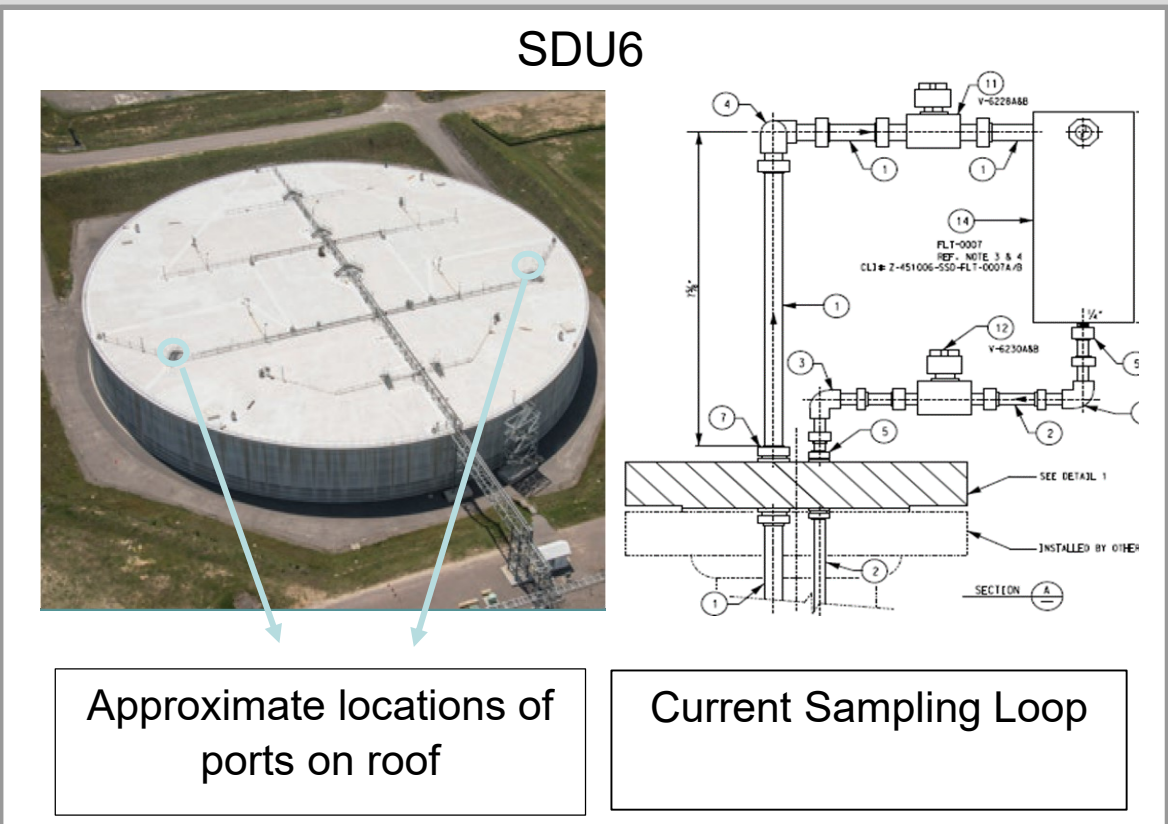


Figure 2: Saltstone Disposal Unit 6 and Sampling Loop

Project start date: 10/01/2017  
Project end date: end of FY19  
Percent complete: 80%  
Partners: University of South Carolina

- Budget:
- Total project funding: 460k
  - Funding received in FY17: NA
  - Funding received in FY18: 230k
  - Total funding planned for FY19: 305k

Highlight statement

Approach

- Overall Objectives:**  
Gas analysis is the most challenging Raman application due to low sample density and narrow spectral resolution, which can potentially be addressed using the SHRS.
- Objective 1: Instrumentation assembly and demonstration for Raman gas analysis**
    - Determine and procure (as necessary) components needed to analyze SDU-relevant compounds.
    - Design and build a gas sample cell (Fig. 3).
    - Demonstrate ability to measure gas samples with a SHRS and compare results to a conventional Raman spectrometer.
  - Objective 2: Optimization of SHRS system for SDU application and demonstration of SDU lab-scale measurements**
    - Lab measurements to demonstrate Raman as a useful technique for SDU samples and to identify potential implementation issues and solutions.
    - Streamlining data acquisition and developing data processing.
  - Objective 3: Adaptation of system for hyperspectral measurements and optimization**
    - Determine the components and arrangement needed to allow for hyperspectral measurements
    - Demonstrate ability to collect hyperspectral Raman images with a SHRS

- Objectives in this reporting period:**
- Objective 1: Instrumentation assembly and demonstration for Raman gas analysis

	Milestones, Deliverables, or Go/No-Go Decision	Progress Notes	% Complete
System Construction	1. Obtain system components and assemble. (March 18)	1. All system components procured and assembled. Final alignment tweaking in progress.	1. 98%
	2. Design and assemble a gas sampling apparatus. (April 18)	2. Gas cell sampling apparatus design and procured. Still awaiting parts delivery.	2. 95%
System Demonstration	3. Measure H <sub>2</sub> and compare to a conventional Raman spectrometer. (September 18)	3. Pending completion of Milestones 1 & 2 above.	3. 0%

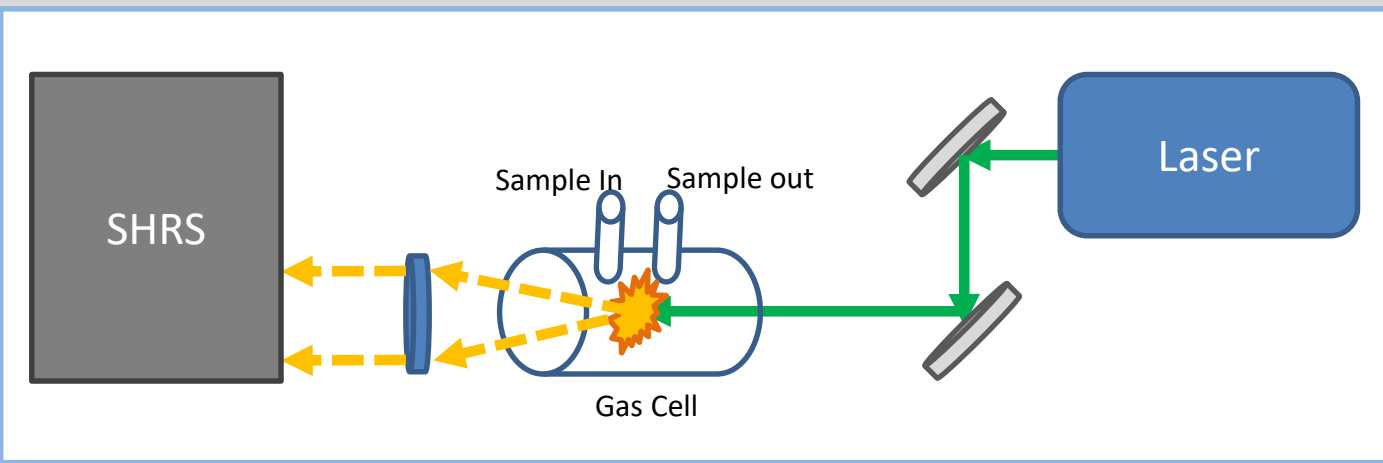


Figure 4: Raman gas sampling set-up schematic

Technical Progress (Accomplishments)

System Construction

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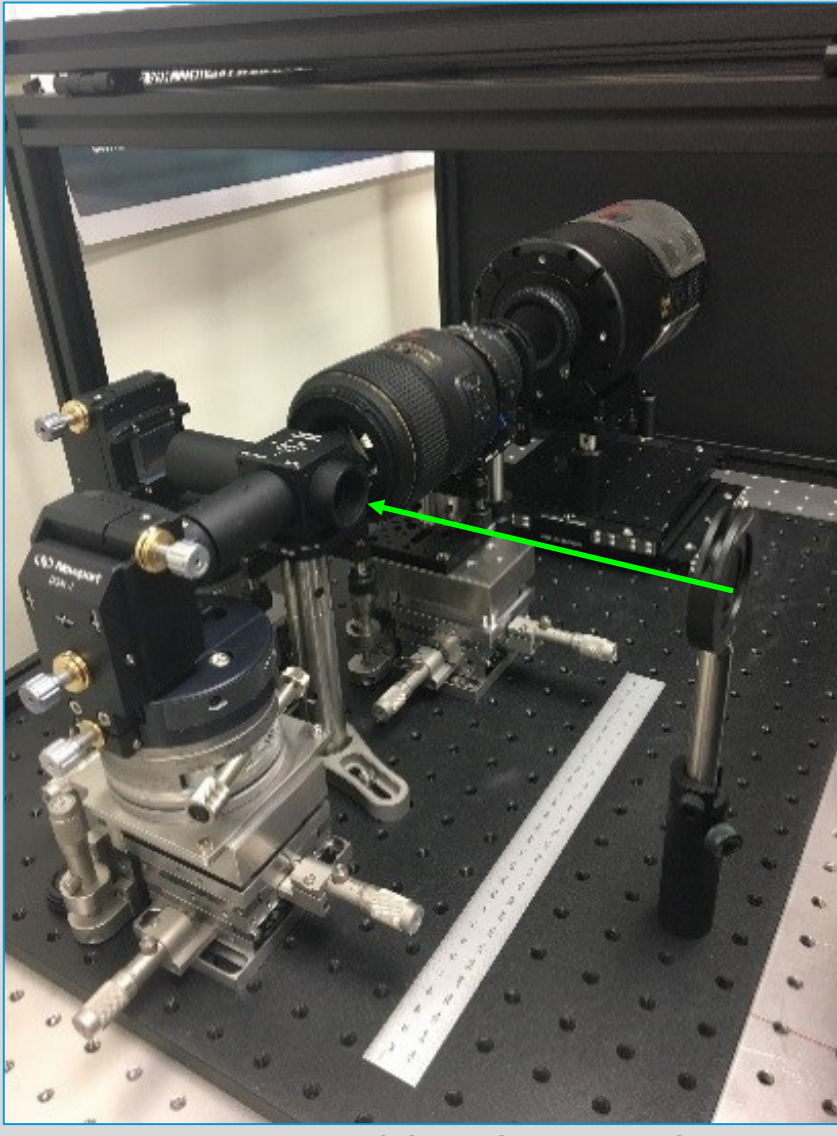


Figure 3: Image of SHRS built at SRNL. Green arrow indicates incoming light.

Gas Sampling Apparatus

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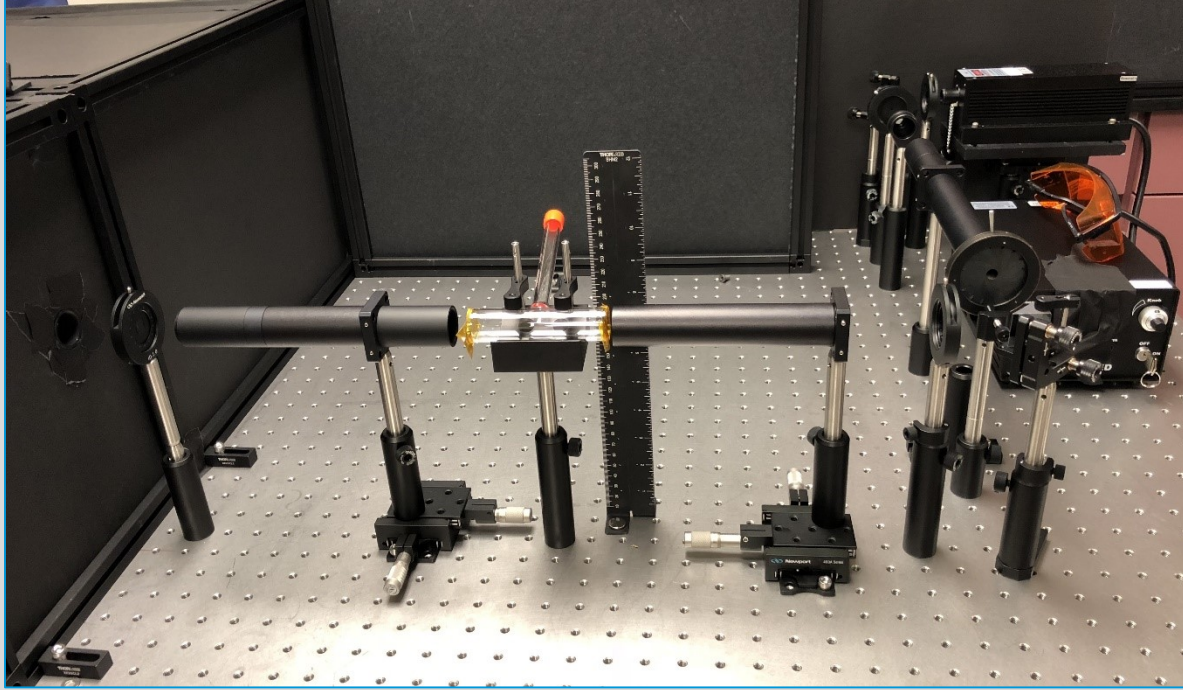
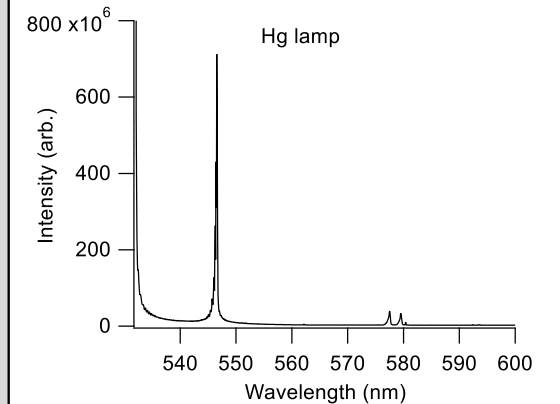


Figure 4: Raman gas sampling set-up.

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Intensity (a.u.)  
Wavelength (nm)

Collaborations

- Savannah River National Laboratory
- Analytical R&D Program & Materials Characterization
  - Energy Materials
- University of South Carolina
- Initiating a subcontract with Prof. S. Michael Angel
  - Scientific/technical expertise
  - Laboratory space to perform experiments

Remaining Challenges and Barriers

- Ground-? With traditional Raman measurements
- Deployment in relevant setting (e.g. Saltstone)

Proposed Future Work

- Task 1:
- Finalize Raman sampling apparatus.
  - Measure H<sub>2</sub> samples to determine instrument performance in the relevant wavelength range.
  - Set up Holospec f/1.8 spectrometer and run comparison study using H<sub>2</sub> to determine the improved sensitivity for Raman measurements using the SHS.
- Task 2:
- Obtain hyperspectral imaging components and assemble.
  - Optimize system for SDU application and run experiments with SDU lab testing.
  - Demonstrate hyperspectral imaging of relevant samples and develop data analysis/processing algorithms.

Project Summary

SHRS has been constructed. All major components have been purchased and received. Instrument has been assembled and aligned. Identified a potential application that would take advantage of the improved performance of SHRS for Raman spectroscopy and tailored the development activities to the specifics of that problem. Currently setting up Raman sampling apparatus and preparing to take initial Raman measurements.