<u>Project Title</u>: Infrared Instrument Development for In-Situ Organic Detection <u>Grant number: NNX08AV85A</u> <u>PI:</u> Dr. Patricia Hynes, pahynes@nmsu.edu, 575-646-6414 <u>Science PI:</u> Dr. Nancy Chanover, nchanove@nmsu.edu, 575-646-2567 <u>Award Institution:</u> New Mexico State University (NMSU) <u>Award date:</u> 7/3/08, Completion date : 7/2/2012 (after 1-year, no-cost extension) <u>Progress Report:</u> Due date to NASA May 1 annually <u>Technical Officer:</u> Diane DeTroye, diane.d.detroye@nasa.gov <u>Technical Monitor:</u> Jonathan Rall, jonathan.a.rall@nasa.gov

NASA EPSCoR Year 3 Progress Report

This annual report documents program activities over the Year 3 period of performance of the grant, and overall progress towards program objectives, as outlined below.

1. Research Accomplishments Measured Against the Proposed Goals & Objectives_

According to the work plan detailed in our submitted proposal, Year 3 activities were described as follows:

"The full system will be integrated at GSFC using facility supplies and controllers. Preliminary full-instrument testing will use a small set of pre-calibrated samples from Task 2 (the survey measurements using the breadboard instrument), with specific criteria for limits of detection of key compounds that will serve as objective measures of performance. The instrument then will be optimized for science relevance on potential missions. This activity includes modifications to the design or the operational protocols of the breadboard hardware necessary to achieve required sensitivity, mass resolution, throughput, limit of detection, and selectivity of the combined system using relevant planetary sample materials."

Our proposed project timeline for Year 3 focused on the tasks of completing the measurements with the breadboard instrument in the laboratory and integrating the AOTF and LDMS components at GSFC. Specific task elements were:

- opto-mechanical integration and testing of the prototype AOTF-LDMS instrument
- making AOTF hardware refinements as needed
- making corroborative sample measurements at GSFC and at NM field sites

We made progress in the above areas between April 2010 and April 2011, and continued work from our Year 2 task list that had not yet been completed. These accomplishments are detailed below.

• <u>Spectrometer Calibration</u>: We developed an algorithm for placing our measured sample reflectivity onto an absolute reflectance scale. We converted our sample measurements to absolute reflectance by ratioing each result to an Infragold reflectance standard with

reflectance > 0.97 over our range of wavelength coverage, after taking into account the number of coadditions and electronic gain. Wavelength calibration is accomplished by first computing a tuning model for the spectrometer, which depends on the orientation of the AOTF crystal relative to the optical axis and also on the acoustic wave direction. Residual wavelength error after this calibration step is generally less than 0.005 nm.

Sample measurements using the breadboard are carried out in ambient air. Even under lowhumidity conditions, the spectra contain vapor-phase H₂O absorption bands that exceed 10% in depth near 2.8 µm as a result of the high intrinsic spectral resolution of the AOTF ($\lambda/\Delta\lambda$ = 350-400). We correct for H₂O absorption by first creating a grid of gas cell transmittance models at standard temperature and pressure, based on the HITRAN 2008 spectral data base. Each measurement is then divided by a model that minimizes the H₂O signature. Leftover artifacts are smoothed by convolving with a Gaussian function of width ~0.015 µm. The resulting effective spectral resolution ($\lambda/\Delta\lambda$ ~ 200) was found to be adequate to fully resolve mineralogical features. An example of this approach, applied to an Infragold reflectance spectrum, is shown in Figure 1. The smoothed result was offset by 0.1 V in this sample plot for clarity.



Figure 1. Example showing the procedure for canceling ambient H_2O features during reflectance measurements.

• <u>Present Status of the Breadboard and Evaluation Work:</u> A breadboard version of the AOTF point spectrometer is shown in Figure 2. Narrow-band light of a single polarization exits the AOTF exit face where abrupt diffraction occurs. A combination of sample and detector mirrors (90° off-axis parabolic reflectors) illuminates a ~2 mm region of the sample and then guides the reflected light back to the detector at ~f/1. The AOTF spectrometer is currently being optimized in several ways, including the implementation of miniaturized RF drive components

and compact mounting of the optical components. The present InAs detector is being replaced by HgCdTe for wider wavelength response. Control of the system with a microcomputer allows the wavelength of the illuminating light to be quickly scanned to obtain the sample reflectance spectrum. A single scan through the full wavelength range of the AOTF breadboard spectrometer is accomplished in roughly 0.1 sec and we typically co-add multiple scans (~64) to improve the signal-to-noise ratio.



Figure 2. Breadboard version of the AOTF point spectrometer.

Our final design of the AOTF assemblies that will be installed in the LDTOF vacuum chamber is shown in Figure 3. We are now having these assemblies manufactured and will populate the electronics and optics decks once completed.

• <u>Ion extraction assembly (IEA) redesign/build</u>: The ion extraction lens (IEL) is essentially a thin tube extending to a few mm above the sample in vacuo, with electrostatic lens elements set to voltages that accelerate positive or negative ions into the tube, where they are collimated for analysis in the LDTOF-MS. In this hybrid AOTF-LDTOF instrument, we made several modifications to the IEL design compared to existing prototypes, which (1) accommodate the incidence and collection of light for the AOTF, (2) permit off-axis laser incidence one side of the sample region that is not occluded by AOTF components, and (3) are consistent with expected flight implementations and restrictions, under various mission scenarios.

Following a preliminary analysis of alternative geometries and ion optical designs for the IEL, a high-fidelity machine design of a "replacement" assembly was developed, involving the mounting of extraction lenses in a highly-tapered housing to maximize solid angle for collection of light for the AOTF. The final overall design of the IEL is shown in Figure 4 (left). Whereas in

previous instruments the laser illuminated the sample through a coaxial bore-hole in the analyzer, the IEL modification makes the sample accessible to the laser from an angle, thereby providing for scanning capability and promoting a more compact instrument package. To provide accommodation of the AOTF spectrometer and to allow angle incidence of the laser, we built a new vacuum housing to replace the previous instrument housing. The newly designed vacuum chamber is shown in Figure 4 (right).



Figure 3. Optical layout of the AOTF point spectrometer that will be paired with the GSFC LDTOF.



Figure 4. Left: The modified LDTOF analyzer incorporates a tapered ion extraction lens (IEL) with an opening half angle of less than 12 degrees, permitting maximal aperture for AOTF illumination and detection of reflectance from the FOV centered under the IEL. Right: The new vacuum chamber built to accommodate the integrated LDTOF and AOTF instruments. The 8" diameter is sufficiently large to house the AOTF prototype within the body of the chamber, and the LDTOF will be mounted into the vertical tube shown. The LDTOF laser will pass through the angled viewport to illuminate the sample surface.

• <u>Sample measurements</u>: Spectral detection of biological materials on mineral surfaces first requires a thorough characterization of the uninhabited host minerals, measured using the same instrument. We acquired near-IR spectra of a collection of field samples using both the AOTF spectrometer breadboard and the LDMS spectrometer at GSFC. The sample suite includes evaporites (sulfates, carbonates), clays, and iron oxides, all of which can be linked to aqueous environments and are therefore of high astrobiological interest. Examples of the AOTF and LDMS sample measurements of carbonates are shown in Fig. 5.



Figure 5. Left: Measurements of carbonates made with the AOTF breadboard. Absorptions due to the presence of carbonates are clearly seen. Right: Measurements of carbonates made with the GSFC LDMS. Sodium and potassium salts are seen, as are Fe, Mg, Fe, Al, and oxides. We can infer mineralogy of this sample from carbonates seen in AOTF data and cations seen in the LDMS data.

We also used both instruments to record the spectrum of a black desert varnish coating on a fractured sample of weathered rock obtained at the Luis Lopez mine site near Socorro, NM. The uncoated side appeared to have been fractured from a larger rock, and thus has shorter exposure age. The composition of desert varnish layers consists mostly of fine-grain clay minerals, and the dark color results from a high concentration of oxidized manganese, which can result from either biotic or abiotic processes. However, strong evidence suggests that oxidized Mn in rock varnish is produced by mixotrophic microorganisms in locations that lack abundant organic acids. Our desert varnish measurements are shown in Fig. 6.



Figure 6. Left: Measurements of rock coated with desert varnish made with the AOTF breadboard. We see water of hydration features and absorptions due to clay minerals, with a few features that still remain to be identified. Right: Measurements of the same desert varnish coated samples made with the GSFC LDMS. Salts (Na, K) are seen as usual, and Al, Ca, Fe, Ti, and oxides are also observed, suggesting mineralogy.

Finally, we used both the AOTF and LDMS instruments to make measurements of basalt samples that were both "neat" and doped with pyrene, a polycyclic aromatic hydrocarbon, in order to determine whether we could detect the presence of hydrocarbons in the rock. We see important differences in the spectra between the neat and doped basalts, which we are currently investigating further.

Our future strategy for measuring samples with both the AOTF and LDMS will be to increase the complexity of the samples. We will examine more field samples, samples that have been doped with more complex hydrocarbons, and ultimately samples that are known to be hosts to living biological organisms.

• <u>Test Suite Archive</u>: Co-I P. Boston (NM Tech) has led the effort to curate and archive our sample suite. She has provided a uniform set of test samples to both the NMSU and GSFC teams, and has provided key oversight regarding the handling, packaging, and testing of all samples. She will continue to guide our sample selections, acquisition, and testing in Year 4.

• <u>Cryo-ice laboratory measurements</u>: We have not yet moved the instrument to GSFC to be tested using Collaborator Moore's cryogenic samples. These activities will likely be taking place in the first quarter of Year 4.

2. Research Success of Individual Investigators

A. Journal Articles and Conference Proceedings

The instrument design details are currently being summarized in a manuscript that is being prepared for publication. The EE graduate student funded by this program will be the lead author on this paper:

Tawalbeh et al. (2011), Acousto-Optic Tunable Filter-based Infrared Point Spectrometer, to be submitted to *Optical Engineering* or *Applied Optics*, June 2011.

A description of the instrument was published in the peer-reviewed conference proceedings of the IEEE Aerospace Conference:

Chanover et al. (2011), An AOTF-LDTOF Spectrometer Suite for In Situ Organic Detection and Characterization, IEEE Aerospace Conference, Big Sky, MT, March 2011.

B. Meeting Presentations

Science PI Chanover presented the instrument concept at two meetings over the past year:

• **Poster Presentation:** American Astronomical Society Division for Planetary Science Conference, October 2010, Pasadena, CA: Chanover et al., "An AOTF-LDTOF Spectrometer Suite for In Situ Organic Detection and Characterization," Bull. Amer. Astron. Soc. 42, 1005. • **Oral Presentation:** IEEE Aerospace Conference, March 2011, Big Sky, MT: Chanover et al., "An AOTF-LDTOF Spectrometer Suite for In Situ Organic Detection and Characterization," Presentation # 2.13.

C. Patents

None.

D. Follow-on Grant Proposals

(i) In the summer of 2010 we were asked to participate in a proposal to NASA's Astrobiology Science and Technology for Exploring Planets (ASTEP) program led by scientists and engineers at the SETI Institute and NASA/Ames Research Center. The proposal was to field-demonstrate an autonomous rover in cave environments. The rover would be outfitted with several instruments, including an AOTF point spectrometer such as the one developed under this program. The proposal was submitted in July 2010 and we have still not learned the outcome.

(ii) In March 2011 we submitted a proposal to NMSU's Interdisciplinary Research Grant (IRG) program. We requested ~ \$50k to build a small, portable, "suitcase" version of our AOTF-based point spectrometer for use in field investigations of astrobiologically interesting sites within New Mexico. The outcome of this proposal is not known at the time of this writing.

E. Improvements in Jurisdiction Research and Development Infrastructure

None.

3. Systemic Change

Our EPSCoR program is contributing to systemic change at NMSU that is aimed at fostering interdisciplinary research collaborations. Our EPSCoR program is a model for intra-department collaboration, one that even spans multiple academic colleges within NMSU, and is frequently highlighted as a major interdisciplinary research activity at NMSU. Our success motivated other researchers at NMSU and NMT to pursue similar collaborations for opportunities such as the 2009 EPSCoR program, which was successful.

4. Examples of Successful Transfer of Technology to the Private Sector

None.

5. Evolution of Collaborations

Several collaborations have evolved as a result of our EPSCoR program:

• Glenar continued his close relationship with our team by coming out for several week-long collaborative visits during Year 3 (in June 2010, February 2011). His involvement has been critical to the success of our laboratory development efforts at NMSU.

• As a result of the oral presentation describing our instrument at the IEEE Aerospace Conference in March 2011, we were invited to bring our AOTF spectrometer and participate in a field expedition to the Arctic in August 2011. The principal investigator of this project is Dr. Brian Glass at NASA/Ames Research Center, and we are continuing discussions with him concerning this possibility. If we are unable to field an instrument by August 2011, Dr. Glass has agreed to provide us with astrobiologically interesting samples that he collects in the Arctic.

• Our collaborators at NASA/GSFC, Drs. Brinckerhoff, Mahaffy, Simon-Miller, and Moore, are integral team members of this project. We conduct regular telecons for the entire collaboration (all NM and GSFC team members who are available) to discuss progress, pitfalls, and near-term action items. This ensures that all team members remain informed about all aspects of the project. We also maintain a wiki, which is a collection of web pages that all team members can access and contribute to or modify content. We use the wiki for recording meeting minutes and agendas, sharing instrument design specifications, and uploading reference materials.

6. Furthering of Jurisdiction Priorities

As stated above in the discussions of Systemic Change and the Evolution of Collaborations, our EPSCoR activities have motivated new interdisciplinary collaborations within the state of New Mexico. These collaborations have resulted in several new funding proposals.

7. Interactions with the Jurisdiction Space Grant Program

Team members Voelz and Chanover attended the annual New Mexico NASA EPSCoR and Space Grant meeting in May 2010. The meeting provided an excellent opportunity for our team members to meet other EPSCoR funding recipients within New Mexico.

8. Demographic Information on Participants

Pls/Cols:

Patricia Hynes, New Mexico State University (White, female) Nancy Chanover, New Mexico State University (White, female) David Voelz, New Mexico State University (White, male) David Glenar, New Mexico State University (White, male) Penelope Boston, New Mexico Institute of Mining and Technology (White, female)

Students:

Rula Tawalbeh, New Mexico State University (Middle Eastern, female) Adam McKay, New Mexico State University (White, male) Ariel Boston, New Mexico Institute of Mining and Technology (White, female)

9. Budget for the Next 12-month Period & Schedule of Program Activities

No additional funds from NASA are anticipated during Year 4, which is a one-year, no-cost extension of our nominal 3-year award.

The focus of our Year 3 activities was the preliminary laboratory measurements of natural samples and analogs of astrobiological interest. Year 4 will focus on the integration with the GSFC-built laser desorption time-of-flight mass spectrometer and the operational demonstration of our instrument. A quarterly schedule for Year 4, which spans July 2011 - June 2012, is shown below.

Activity	Q1 (Jul-Sep)	Q2 (Oct-Dec)	Q3 (Jan-Mar)	Q4 (Apr-Jun)
submit ASTEP proposal				
sample measurements (NMSU lab)				
AOTF+LDMS unit assembly, testing				
cryo-ice sample meas. (GSFC lab)				
DPS or AGU meeting presentation				
LPSC meeting presentation				
sample meas. at NM field sites				