Extractive Sampling Methods and Analysis of Particulates

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SITB

Engineering Directorate Concept-to-Flight

http://engineering.larc.nasa.gov/

Introduction

- **Undergrad: University of Maine, School of Engineering (Orono, ME)**
  - B.S. Chemical Engineering
  - Minor: Mathematics, Biology

- **Undergrad Research: Developing value added products and biofuels from marine macro algae**

- **OMNOVA Solutions – Intern**

Next:

- **Ames Research Center: ECLSS Air Revitalization group**

Background: contamination control

Contamination control and planetary protection

- Concerned contamination of organic residuals and biological organisms on hardware and instrumentation
- Research materials compatibility with other hardware and the space environment

Contamination:

- Any unwanted matter which could be detrimental to the required operation, reliability, or performance of a system

Types of Contamination

- Can be from the system itself or introduced from external sources
  Other possible contaminants:
  - Biologicals (e.g. skin)
  - Metal shavings
  - Paper fibers
  - Adhesive from tape
  - Payloads

Contamination residuals on MEDLI2 Supersonic Tubing Assembly hardware

Particulate contamination overview

- **Predominantly sensitive devices:**
  - Mirrors, thermal control surfaces, solar arrays, and optical elements

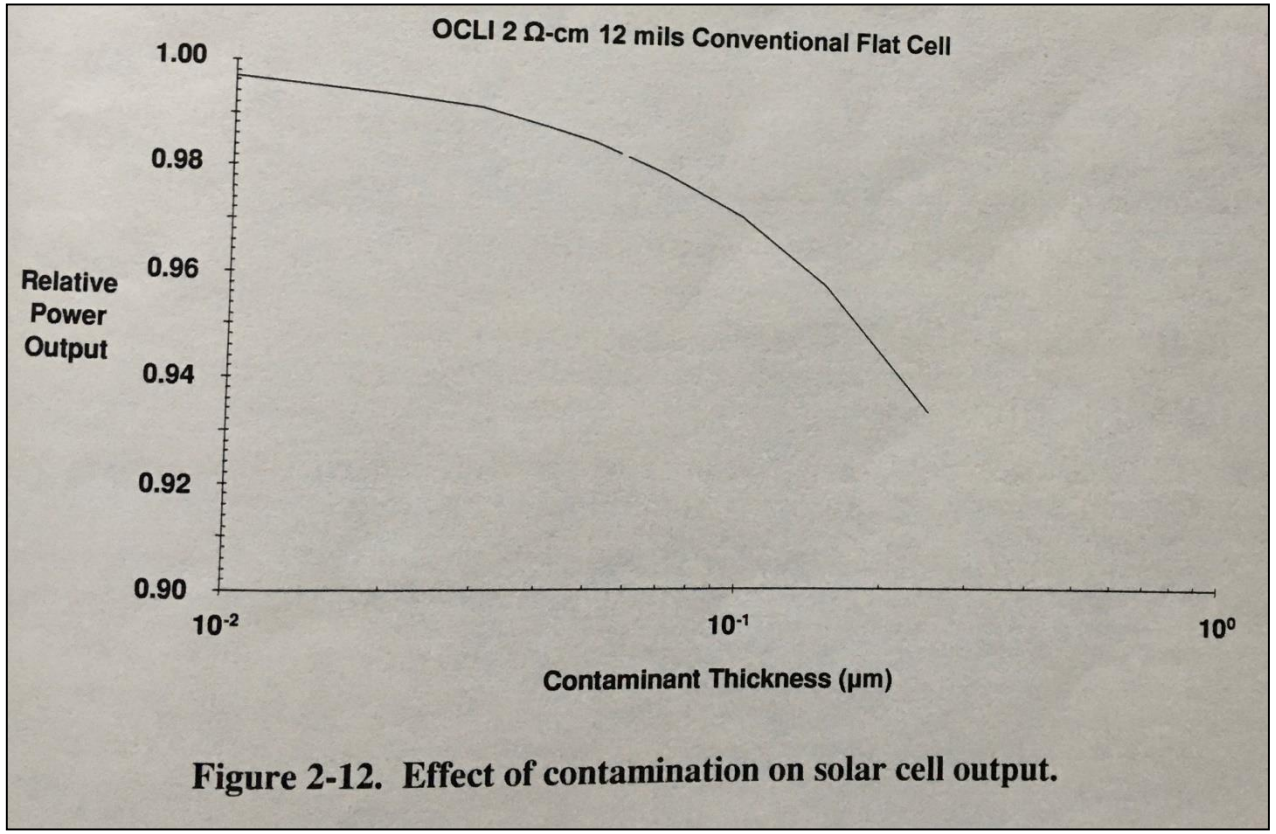


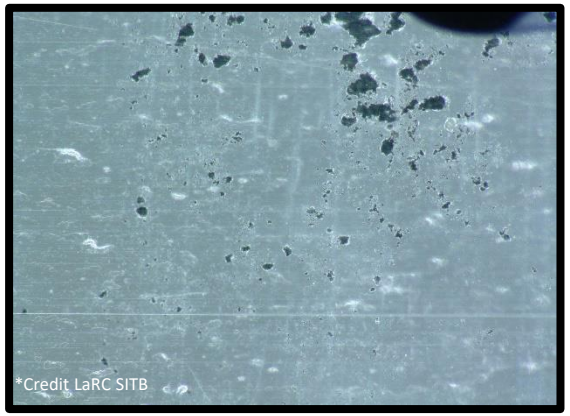
Figure 2-12. Effect of contamination on solar cell output.

From *Tribble, Allan (2000)

Issues:

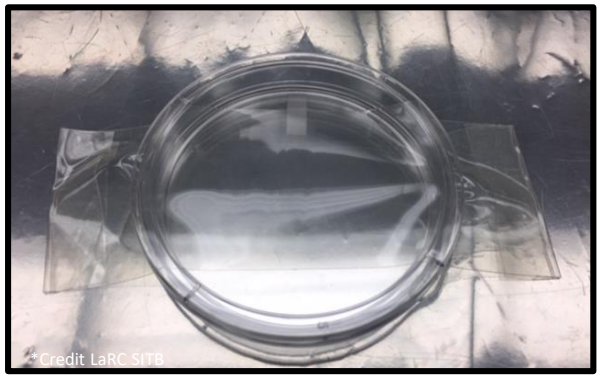
- Surface obscuration
- Light absorption
- Scattering of light / reflectance and transmittance

Particulate contamination overview



<u>Particulate contamination</u>: Deposition of visible conglomerations of matter

<u>Molecular contamination</u>: Build up of individual molecules

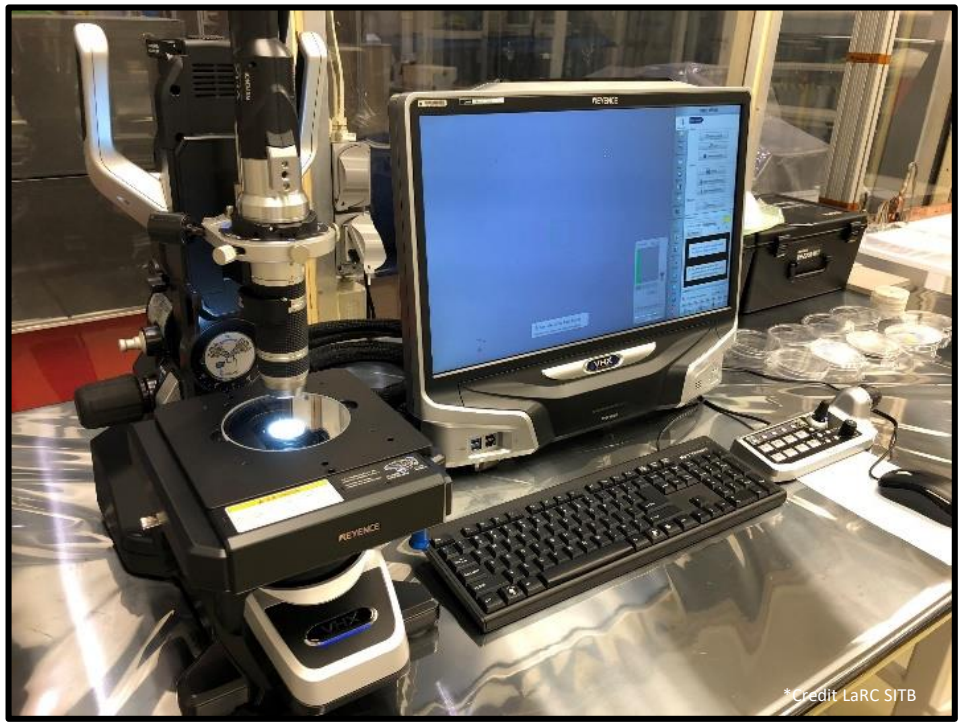


Physical extraction removal method

Solvent removal method

Particle analysis background

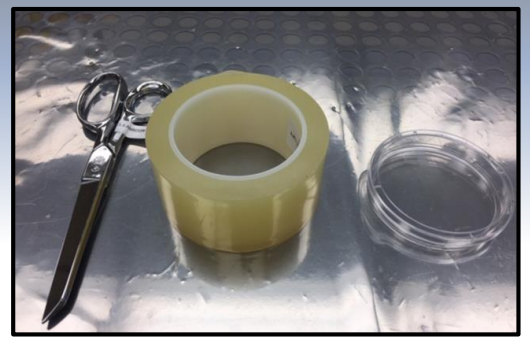
- Extractive sampling and analysis methods for particulate contamination
- Tape lift methods have been the historical choice for extractive sampling



Keyence Digital Microscope

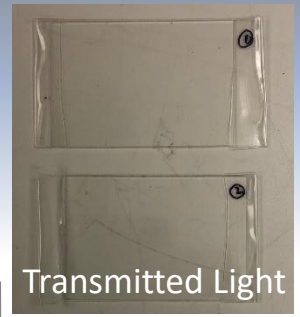
Particle analysis sampling – previous method

Sample Preparation



Sample Collection

Sample Preparation



Optical Microscopy

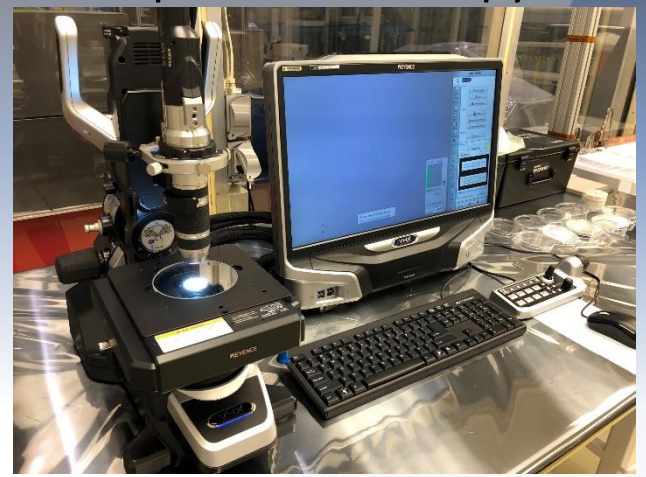


Image Processing

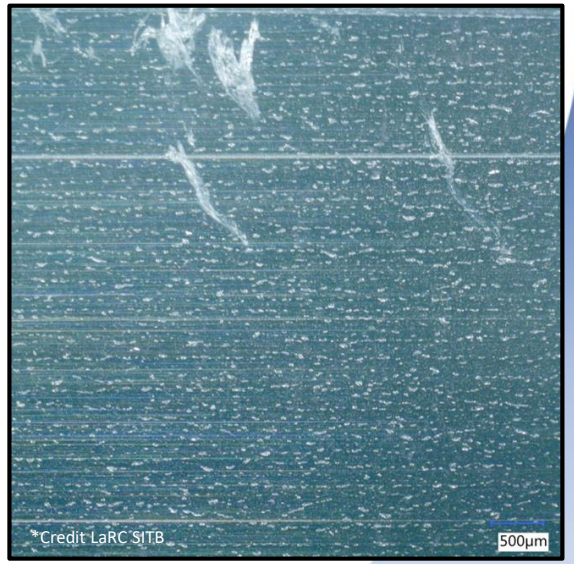
Data Analysis

	A	B	C	D	E	F
1	Total Particle Counts					
2	Sample ID	>100 um	50 um - 100 um	25 um - 50 um	15 um - 25 um	5 um - 15 um
3	TL1	6	21	58	247	5303
4	TL2	2	19	243	333	3211
5	TL3	4	84	333	580	4467
6						
7						
8	Average	4	42	211	387	4327
9						
10						
11	PAC Values					
12	Size Range	Particles per 0.1 m^2	Factor	PAC	Level	
13	5 um - 15 um	172860	4.18E-08	7.22E-03	FAILS ALL	
14	15 um - 25 um	13914	2.09E-07	2.91E-03	FAILS ALL	
15	25 um - 50 um	8820	6.25E-07	5.51E-03	FAILS ALL	
16	50 um - 100 um	1734	2.19E-06	3.80E-03	FAILS ALL	
17	>100 um	176	5.52E-06	9.73E-04	FAILS ALL	

Particle analysis sampling – previous method

Problems with this method:

- **Lots of interaction between the analyst and the tape - construction of tape lifts and preparation for analysis**
  - The more interaction a person has with the sample, the more likely that sample will be contaminated with particles not from the hardware
  - Tape is likely to fold up or unintentionally touch another surface which will ruin the sample
  - When the tape if removed from the roll, uneven tension will cause the surface of the adhesive to be uneven in certain areas
- **Unreliable analysis**
  - Identification of particles is user dependent and therefore, count and sizing of particles is inconsistent
  - Unable to easily distinguish particles from air bubbles in the tape or imperfections on the microscope stage
  - Difficult to get tape flat on petri dish which introduces additional factors and inconsistencies
  - Area on tape where hardware was sampled is not marked – analyst does not know where sample is on the lift
  - Time consuming analysis!



Tape lift under Keyence magnification

New method

- **Extractive sampling and analysis methods for particulate contamination**

Objectives:

- Establish test article and sampling method
- Improve imaging method
- Develop automated analytical method and report generator
- Analyze the capabilities/ viability of method
- Generate a comprehensive guide of the analytical methodology research

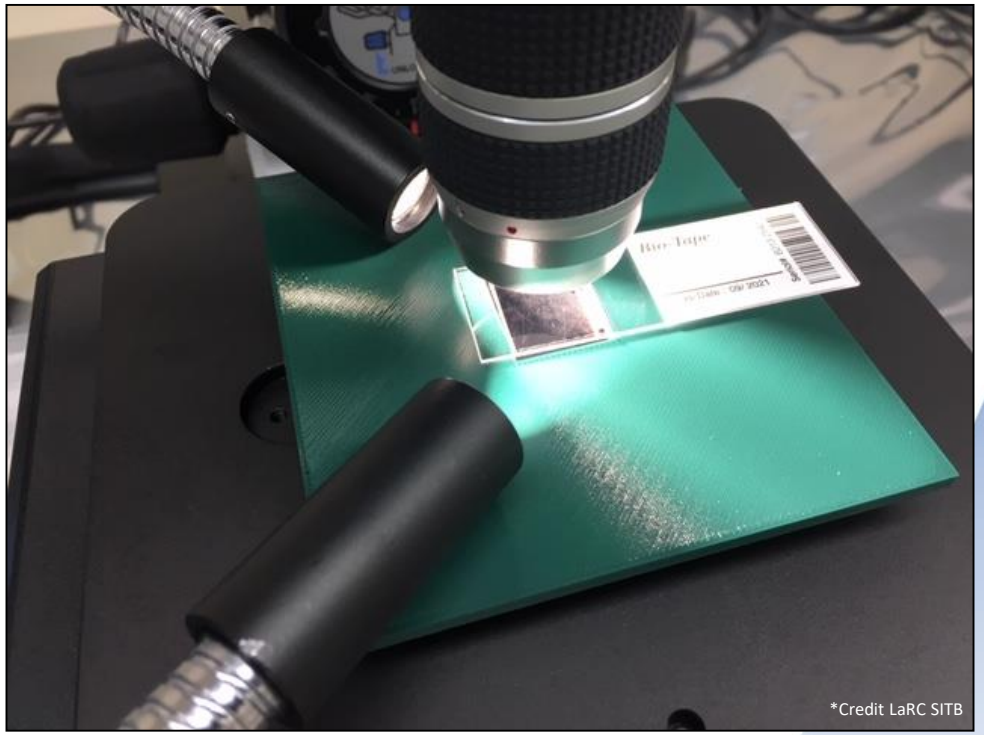
Improve test article and sampling method

- **New method involves using a commercial biological tape lift slide for contamination analysis**
  - Slides come prepackaged in a re-sealable case
  - Slides have a liner to cover the adhesive
  - Slide has predetermined and defined area for sampling and analysis
  - Slides can be easily labeled and even scanned into an electronic database due to bar code
  - Slides can be easily transported and reliably reanalyzed

Unused commercial biological tape lift slide inside case

Improve imaging method

- **Method replicates a more traditional biological microscope**
  - Slides are elevated above the stage
    - Imperfections in the stage's surface are not seen in the image
  - Side lighting is used
    - Removes visibility of the air bubbles in the tape and only shows particulates on the surface

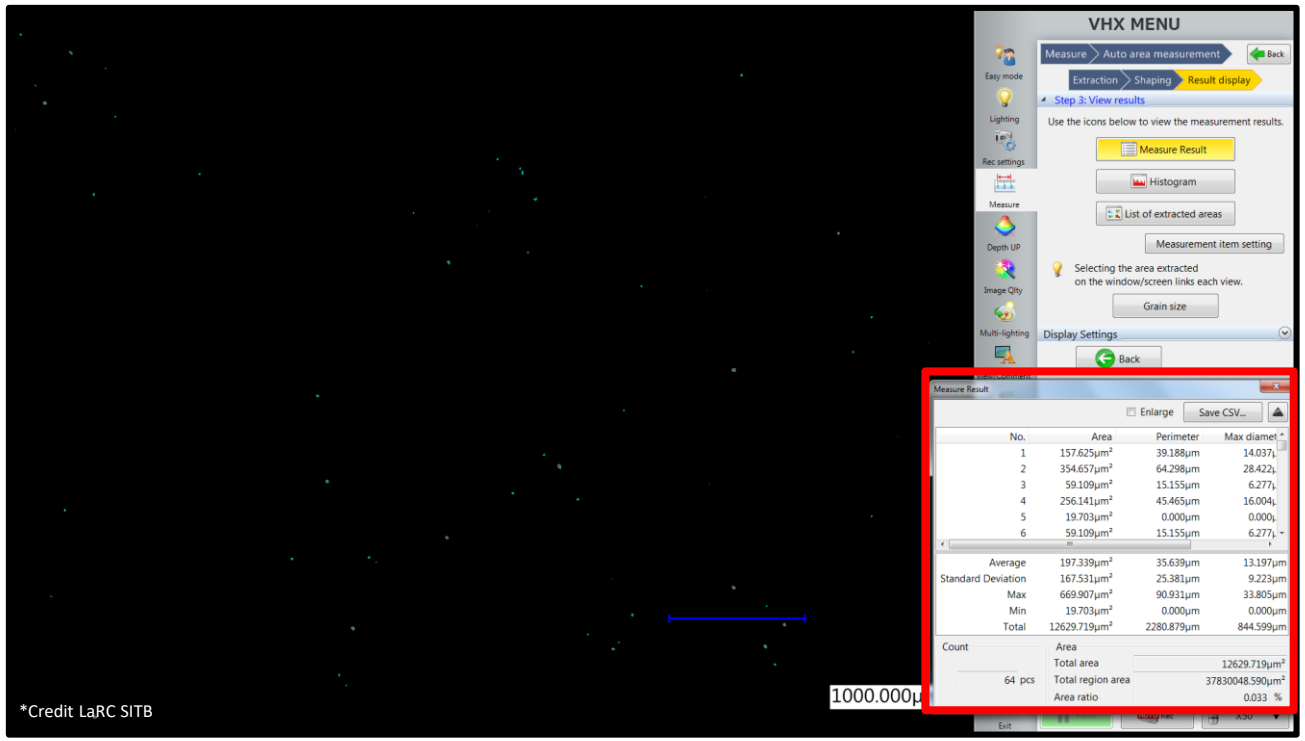


Elevated tape lift slide with side lighting analyzed under the Keyence microscope

Keyence imaging

- This form of analysis completely automates the counting and sizing of particles on a sample
- The results can be saved in a CSV file and then imported into the Excel analytical tool

Image of article sample surface



Save CSV

Particulate contamination overview

Measurements used to quantify particulate contamination:

- **Surface cleanliness value/particulate cleanliness level**
- **Percent area coverage (PAC)**

A few of the notable assumptions for calculations:

- **No particle overlap**
- **Randomly oriented**
- **Hemispherically capped cylindrical geometry**
- **Analysis using standardized size ranges**

Table 1. Cleanliness levels based on IEST-STD-CC1246E

Particle Size Range		Particulate Cleanliness Levels								
min (um)	*max (um)*	*25*	*50*	*100*	*200*	*300*	*400*	*500*	*750*	*1000*
5	15	19	141	1519						
15	25	2	17	186	2949					
25	50	1	6	67	1069	6433				
50	100	0	1	9	154	926	3583	10716		
100	250	0	0	1	15	92	359	1073	8704	
250	500	0	0	0	0	2	8	25	205	983
500	750	0	0	0	0	0	0	1	7	33
750	1000	0	0	0	0	0	0	0	1	3
1000	1250	0	0	0	0	0	0	0	0	1

Standards from IEST-STD-CC1246-E for particulate cleanliness level

Excel analytical tool demonstration

Next steps

Viability of method

- Development of imaging standard
- Validate lower limit of detection for this method
- Validation of new sample tape lift article with IEST-STD-1246-E
- Validate consistent results between analysts

Analytical tool

- Add statistical data analysis

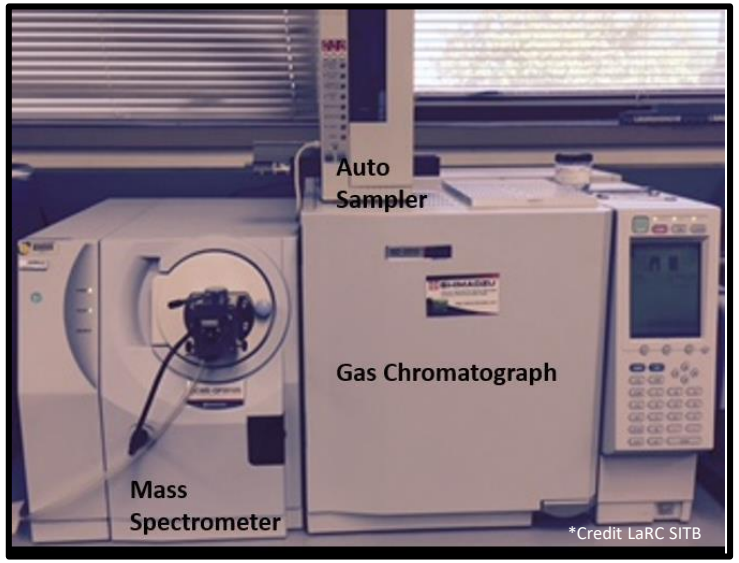
Semester overview

Branch duties typically focused in one of the following areas:

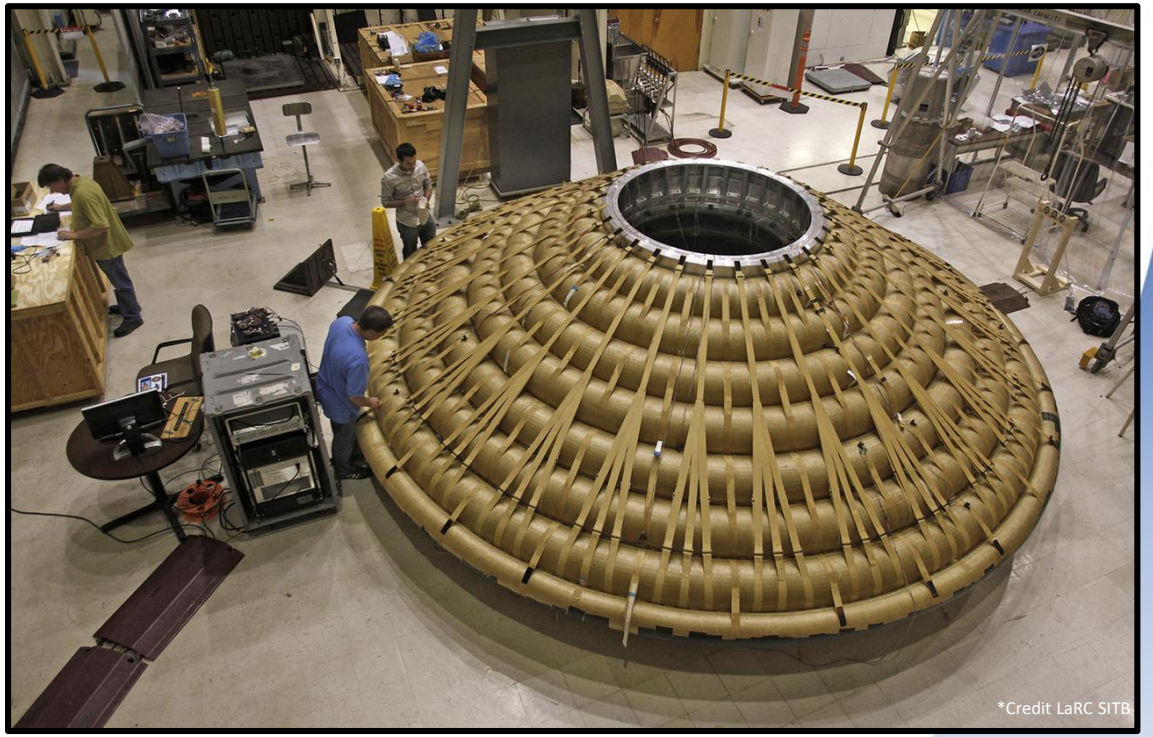
- **Analytical methodology research**
- **Direct project assistance**
- **Facility efficiency**
- **Training**

Analytical methodology research

- **Quantitative analytical methods for silicones**
  - Support of LOFTID
  - GC-MS demonstrated high sensitivity to DC-704
  - FTIR methods to be evaluated further



GC-MS Chromatograph Equipment Setup in B1293-C



LOFTID Inflatable structure

Direct project assistance

- **Conducted hardware inspections and sampling**
- **Led chemical analysis on project samples**
  - Completed data analysis
  - Generated reports, summaries, and presentations on completed project analysis
- **Provided project support in contamination control investigations as needed**

Portion of project samples

Facility efficiency

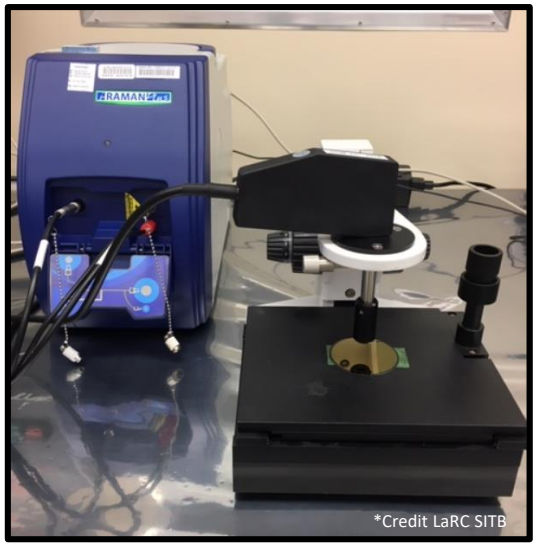
- **Cleanroom certifications and reports**
  - B1250
  - B1202
  - B1188
- **Cleanroom certification templates and database**
- **Automation of a remote monitoring process for cleanroom facilities**

Clean tent in B1250

Training

- **Makerspace facility tutorial write-up**
  - Capabilities
  - General use

- **Methods of sampling, chemical analysis, and data analysis**
  - Gravimetric analysis; Raman and FTIR instrument and software demonstration
    - Methods of sample preparation
    - Use of instruments / best practices
  - Tutorial on spectra analysis and use of analytical tools



Raman Spectroscopy Laboratory Set-up

FTIR Laboratory Set-up in B1293-C

Acknowledgements

Mentors

- Gugu Rutherford: NASA – Contamination Control and Planetary Protection Lead
- David Taylor: NASA – Contamination Control Engineer
- Joey Sparrow: NASA – Contamination Control Technician

Summary:

- **Extractive sampling methods and particulate analysis**
  - Background
  - Previous method
  - New sampling method
  - New imaging method
  - New analysis method
  - Next steps
- **Analytical methodology research**
- **Direct project assistance**
- **Facility efficiency**
- **Training**

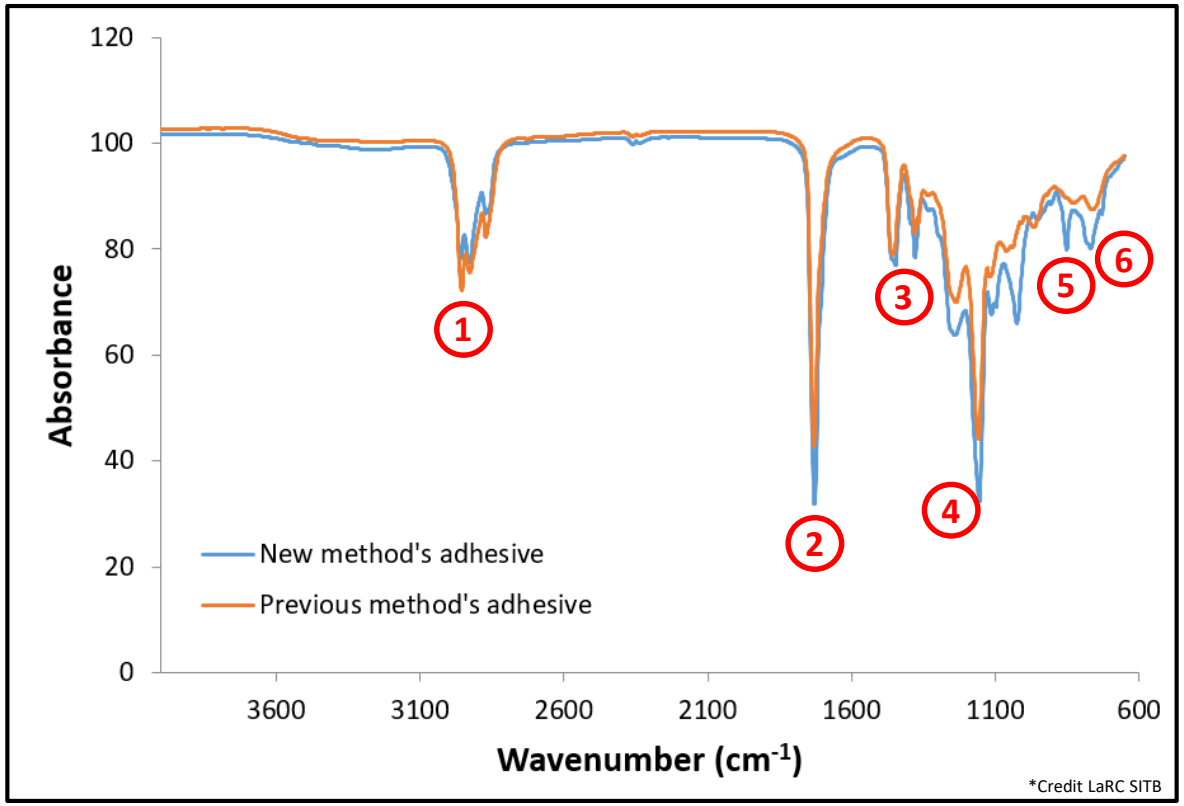
Citations

- **References and Reports:**
- **Contractor Report 4740** Contamination Control Engineering Design Guidelines for the Aerospace Community
- **TM-2017-219591** Method for the Collection, Gravimetric and Chemical Analysis of NVR on Surfaces
- **ECSS-Q-ST-70-50C** Particles Contamination Monitoring for Spacecraft Systems and Cleanrooms
- **IEST-STD-CC1246E** Product Cleanliness Levels – Applications, Requirements, and Determination
- **Kimes, Katerina. (2018)** Development of Particulate Contamination Analysis Methods
- **Ma, P., Fing, M., Lee, A. (1989)** Surface Particle Obscuration and BRDF Predictions
- **Tribble, Alan (2000)** Fundamentals of Contamination Control

BACKUP SLIDES

New tape vs previous tape adhesive

- **Spectra are indicative of an acrylic adhesive**



FTIR spectra comparing the bio tape adhesive with the adhesive from the previous method's tape

Table 2. Characteristic absorption bands of polymers visible in IS spectra of acrylic adhesives

Location Index	Wave number (cm^{-1})	Functional Group Description
1	2960, 2926, 2866	C-H stretching vibrations
2	1728	– C=O stretching vibration
3	1446, 1377	CH_3 deformation vibrations
4	1250, 1159	–C-O-C stretching asymmetrical vibrations
5	852	=C-H deformation vibrations
6	766, 729	C H deformation vibrations in ring