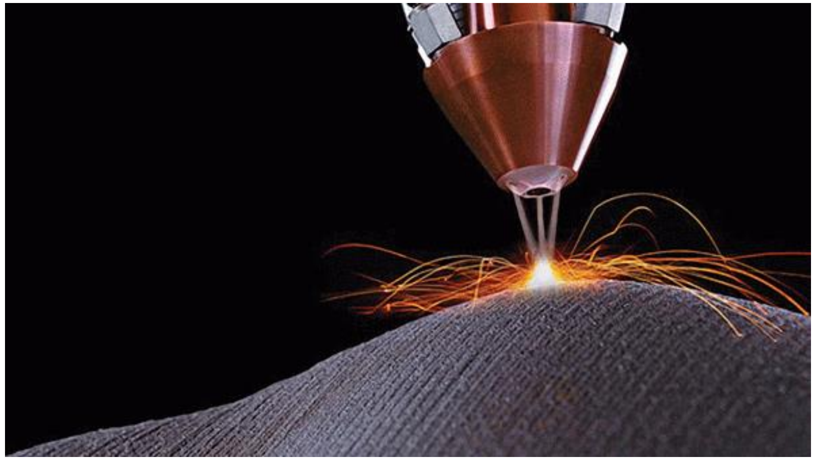
Cleaning and Cleanliness Measurement of Additive Manufactured Parts



Roger W. Welker

Sr. Contamination Control Engineer

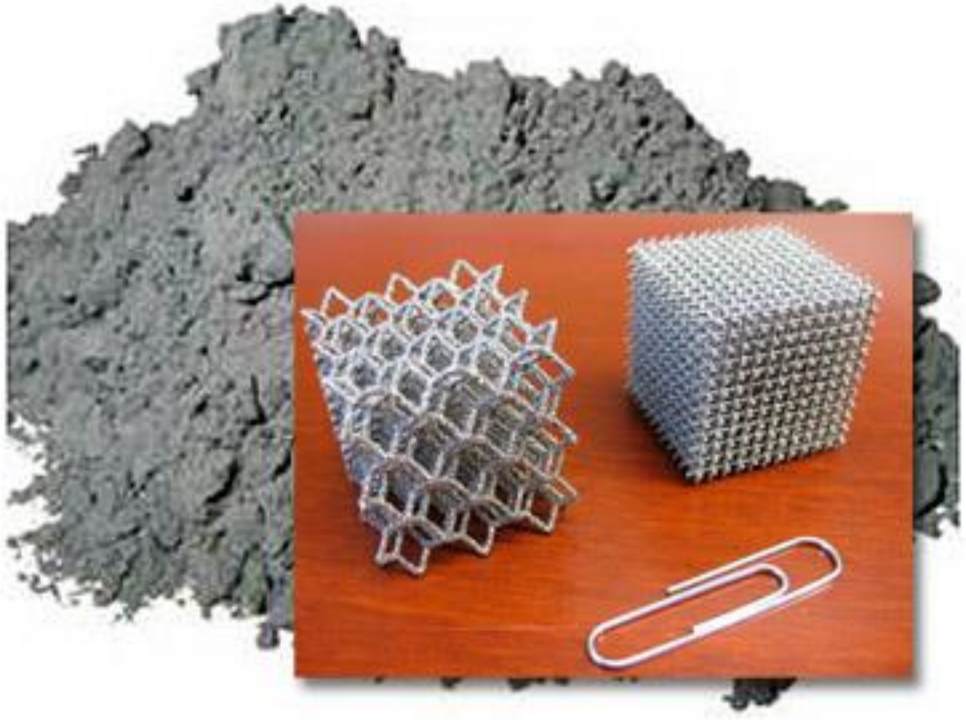
Jacobs ESSSA/Marshall Space Flight Center

Mark A. Mitchell

Project Lead

EM50

NASA/Marshall Space Flight Center



Cleaning and Cleanliness Measurement of Additive Manufactured Parts

- **Rationale for technical approach:**
  - Alloys for liquid fuel rocket engines are expected to be relatively hard, high melting point, corrosion and ultrasonic cavitation erosion resistant alloys: Ultrasonic [u/s] immersion cleaning should be a viable method
  - Selective laser melting process avoids use of organic materials, like cutting fluids, mold release compounds, etc.: This makes aqueous [H_2O] based cleaning a viable candidate
  - Move toward an objective means of assessing cleanliness rather than visual inspection: turbidimetry of extracted contamination
  - Multiple u/s extraction [MUX] has been successfully used to develop cleaning processes for similar materials

Cleaning and Cleanliness Measurement of Additive Manufactured Parts

Current effort:

- Cleanliness measurements
  - u/s extraction in detergent/H_2O solution followed by turbidimetry
  - Turbidimetry of aqueous ultrasonic (u/s) extracts is a well accepted method for indirect cleanliness measurements of metal parts
  - All equipment needed for measurements is already available
- Cleaning methods development
  - Prior work shows Inconel 718 exhibits well behaved multiple ultrasonic extraction[1]: Other alloys for liquid fuel engines expected to be the same
- Cleaning method candidates
  - H_2O: u/s immersion, spray, u/s immersion + spray
  - Solstice PF: u/s immersion/vapor degrease, u/s immersion/spray/vapor degrease

[1] JANNAF Liquid Propulsion Subcommittee and Advanced Materials Panel TIM, Huntsville, AL, 3 - 5 September 2014

Experimental Procedures

Turbidity measurements[1]:

1. Clean 300 ml Berzelius (high form) beaker and fill w/ 250 ml filtered distilled water containing approximately 0.02 % by volume Joy detergent
2. Sonicate 1 minute in Branson 5810 u/s tank filled w/ room temp. water
3. Fill vial with water/detergent mixture,
   u/s degas and measure turbidity in Hach – blank turbidity
4. Sonicate part for 1 minute.
5. Repeat step 3 – part turbidity
6. Subtract result 3 from result 5 to get net turbidity
   increase due to contamination extracted from the part

[1] When water ultrasonic immersion cleaning experiments are done, the turbidity of the water detergent is measured before and after cleaning, as well as extracting the parts after cleaning

Equipment Used

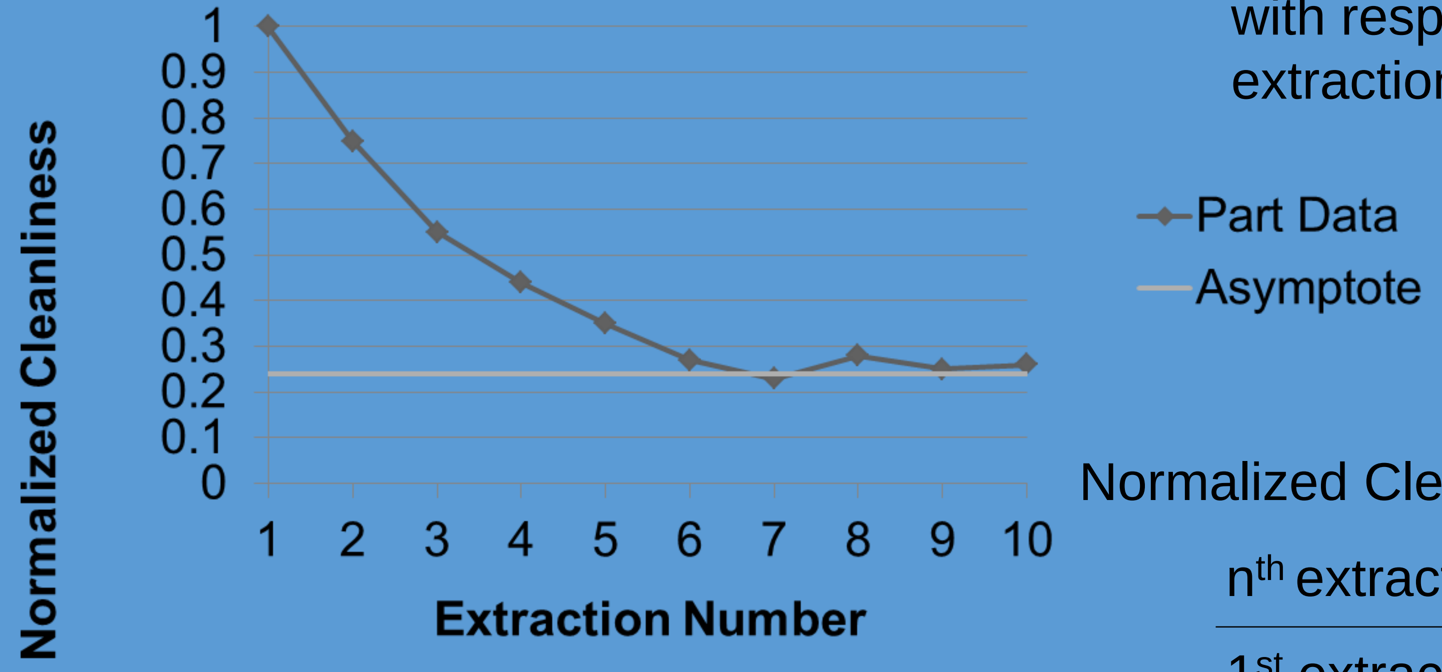
Working fluid: 250 ml filtered distilled H_2O in 300 ml beaker, 200 ppm (0.02%) Joy detergent, centered in tank, 1.0 minute extraction

Branson 8510 5.5 gallon (22 liter)
42 kHz, 250 watt (nominal – not measured)
Coupling fluid: room temp (22°C) tap H_2O, degassed 5 minutes

Hach 2100N Ratio Turbidimeter
0.001 NTU resolution (gage capability for this test not yet determined)

Multiple Extractions

A Well Behaved Multiple Ultrasonic Extraction

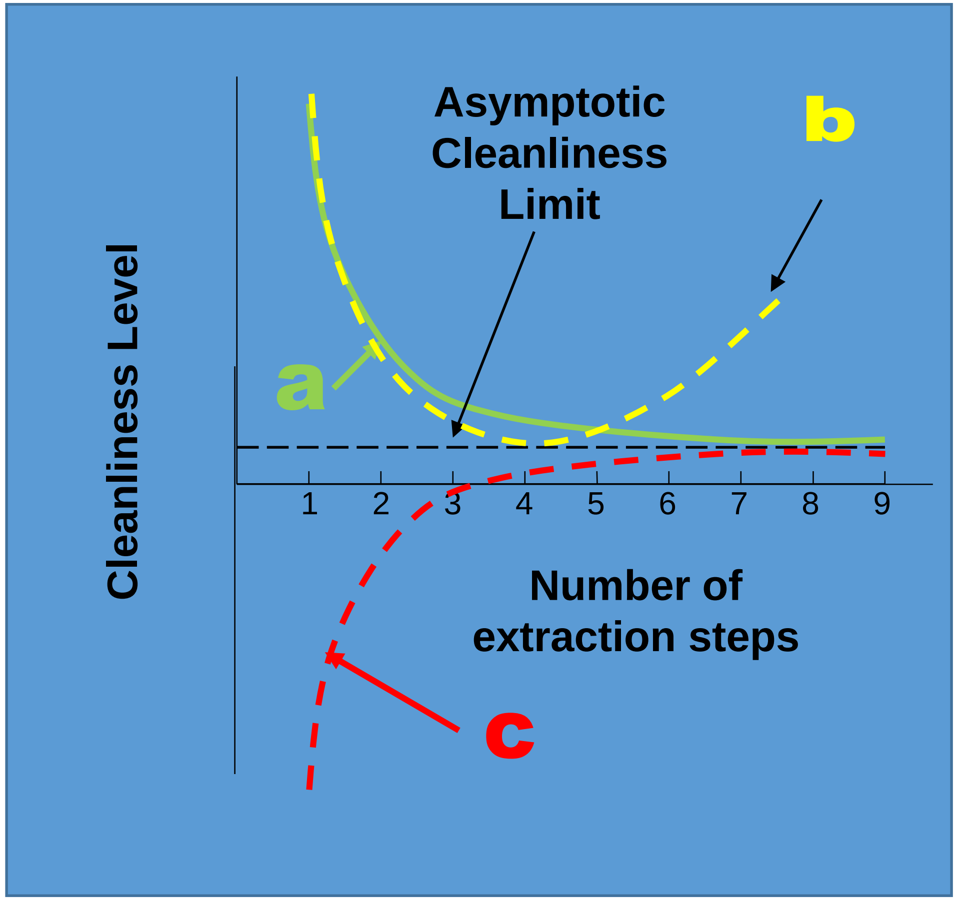


- Data is normalized with respect to 1st extraction

$$\text{Normalized Cleanliness} = \frac{n^{th}\ \text{extraction}}{1^{st}\ \text{extraction}}$$

Multiple Extractions

Three different types of ultrasonic cleanability curves



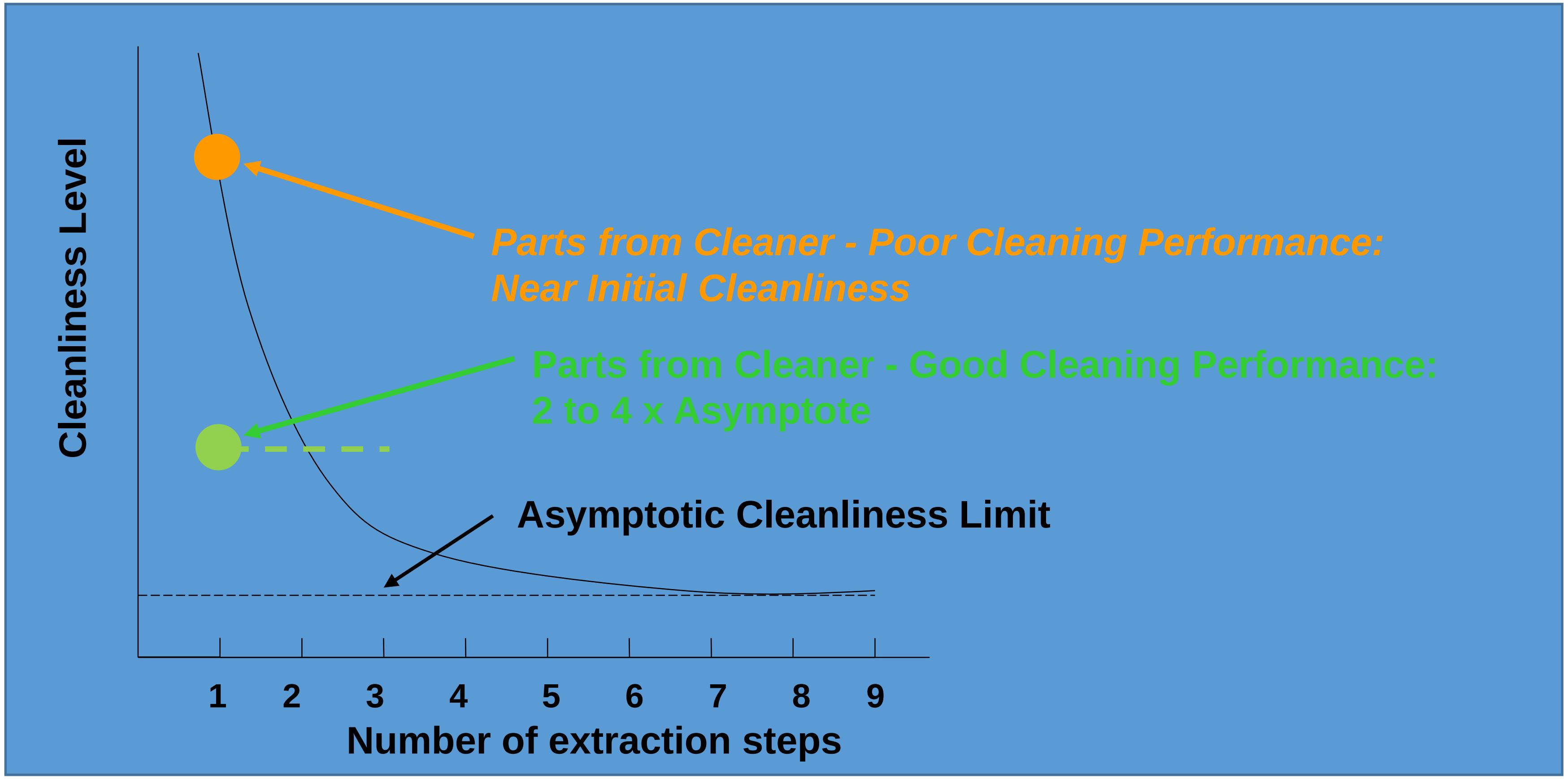
a) Well behaved

b) Erosion sensitive
  - *Ultrasonic cleaning must be done with care*
  - *Ultrasonic extraction for verification still useful*

c) Extremely erosion sensitive
  - *Neither ultrasonic cleaning nor ultrasonic extraction are suitable*

Multiple Extractions

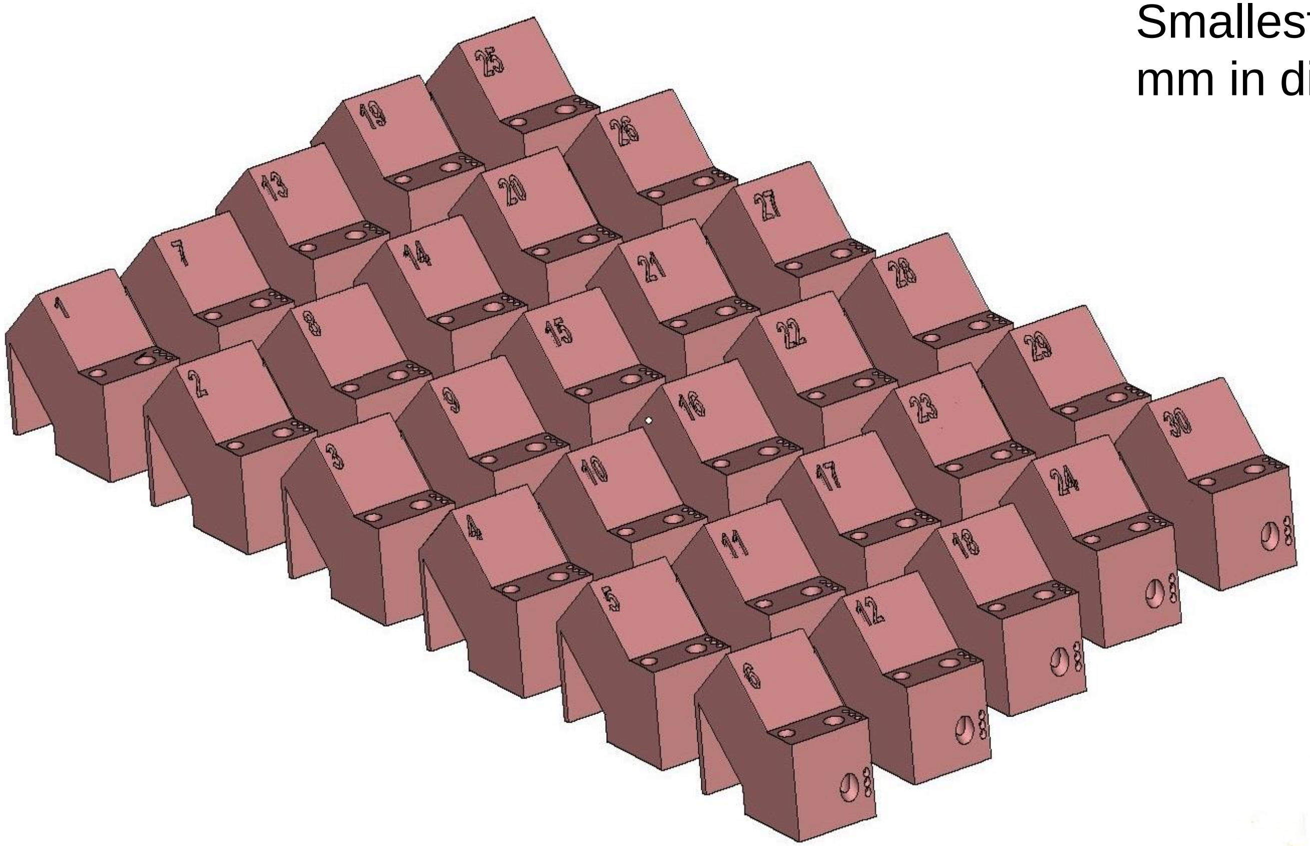
Using the multiple ultrasonic extraction cleanability curve to assess cleanliness, cleanability and cleaner performance

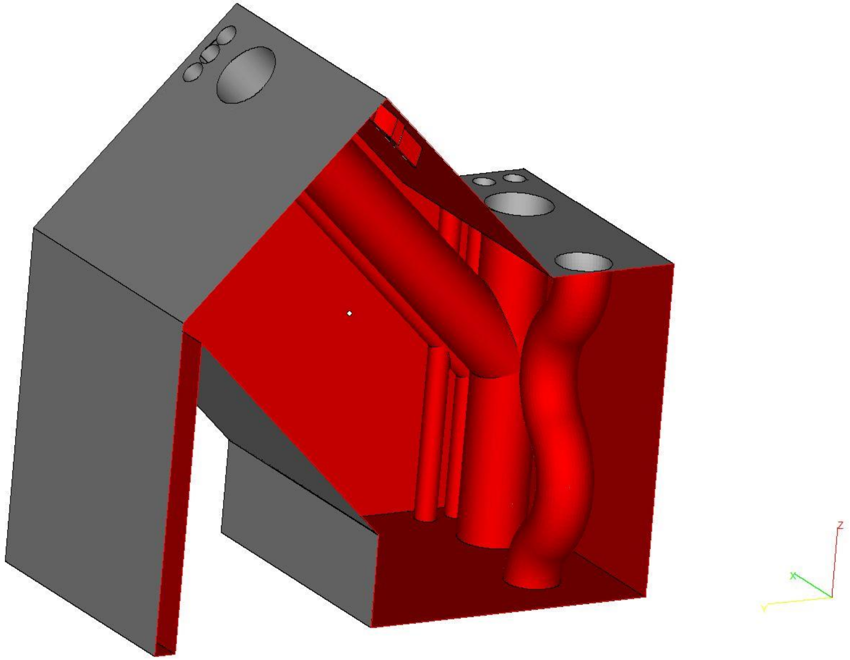


The Test Specimen

Surface area = 2831.6 mm^2

Smallest holes are approximately 0.5 mm in diameter





Cleaning Test Matrix

Test	Description	Purpose
1	5 parts MUX	1. Determine the variability of u/s cleanability and cleanliness level
		2. Determine a preliminary u/s immersion cleaning process time
2	5 parts u/s cleaned in detergent/H_2O solution	Determine preliminary cleanliness capability for US immersion cleaning in detergent/H_2O solution
3	5 parts spray cleaned using with DI/H_2O only	Measure spray cleaning process capability
4	5 parts US washed in detergent/H_2O solution followed by Millipore FilterJet spray rinse with H_2O only.	1. Measure u/s wash + spray rinse process.
		2. Does adding spray rinse improve cleanliness?
5	4 parts 3 minute u/s wash + vapor degrease	Measure Solstice PF performance in u/s vapor degreaser
6	5 parts 3 min u/s wash + 1 minute cold spray rinse + vapor degrease	Does adding cold Solstice PF spray rinse improve cleanliness?
7 - 11	2 parts from test 2 - 6 be cryo-shocked. 2 other parts from test 2 - 6 to be measured as control	Does cryo-shock degrade cleanliness of cleaned parts?

Additional Tests

- Parts were weighed before and after cleaning to supplement turbidity measurements
  - Parts highly variable in appearance: document by mass variability
  - Needed data for drying tests
- Parts were reused after cleaning for drying experiments
  - Compressed air blow-off plus room temperature air dry overnight
  - Blow-off plus oven baking
  - Blow-off plus vacuum oven baking
  - Blow-off plus solvent displacement drying

Variation in Appearance and Weight

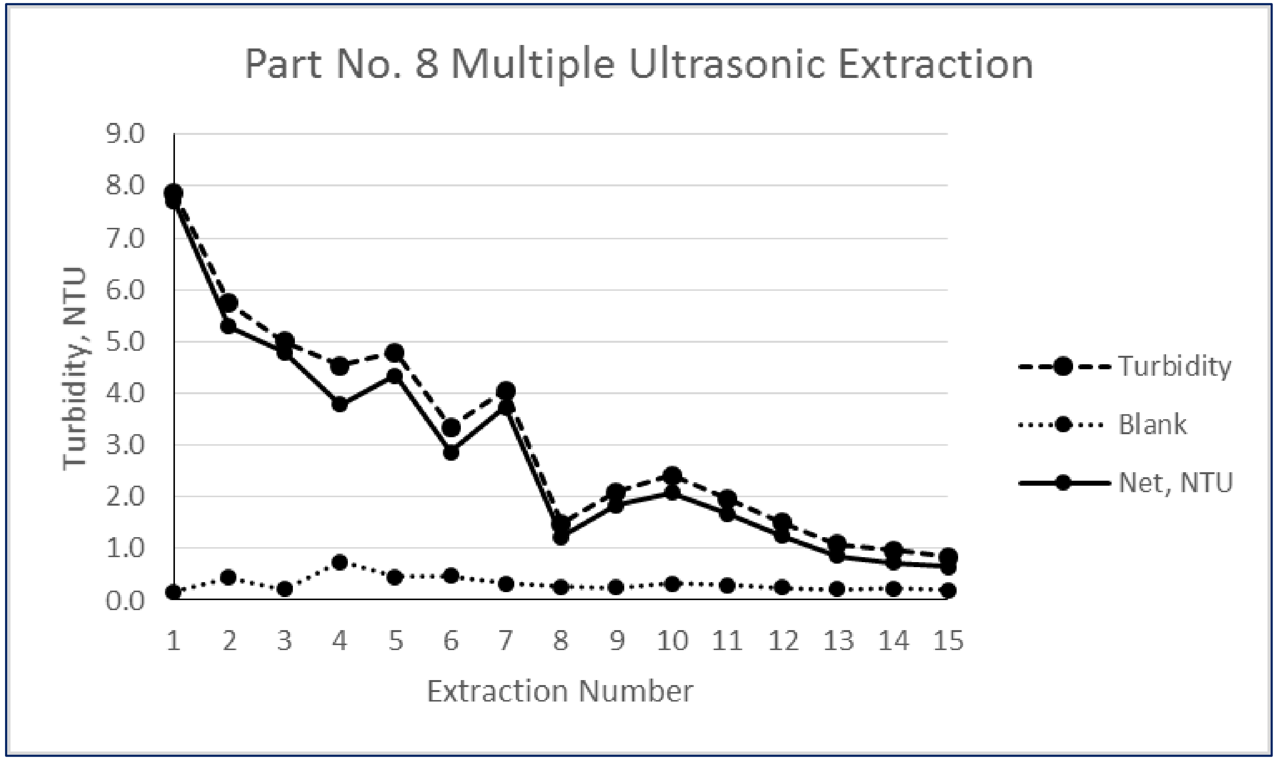
Weights vary from 31.3622 grams to 33.2450 grams

Experimental Procedures

- Parts are handled with washed, dry gloves or clean dry forceps (no bare hands) for all tests
- Condition the parts to room temperature before weighing
- Parts weighed with calibrated electronic balance to +/- 0.0001 gm

Multiple Ultrasonic Extraction Results

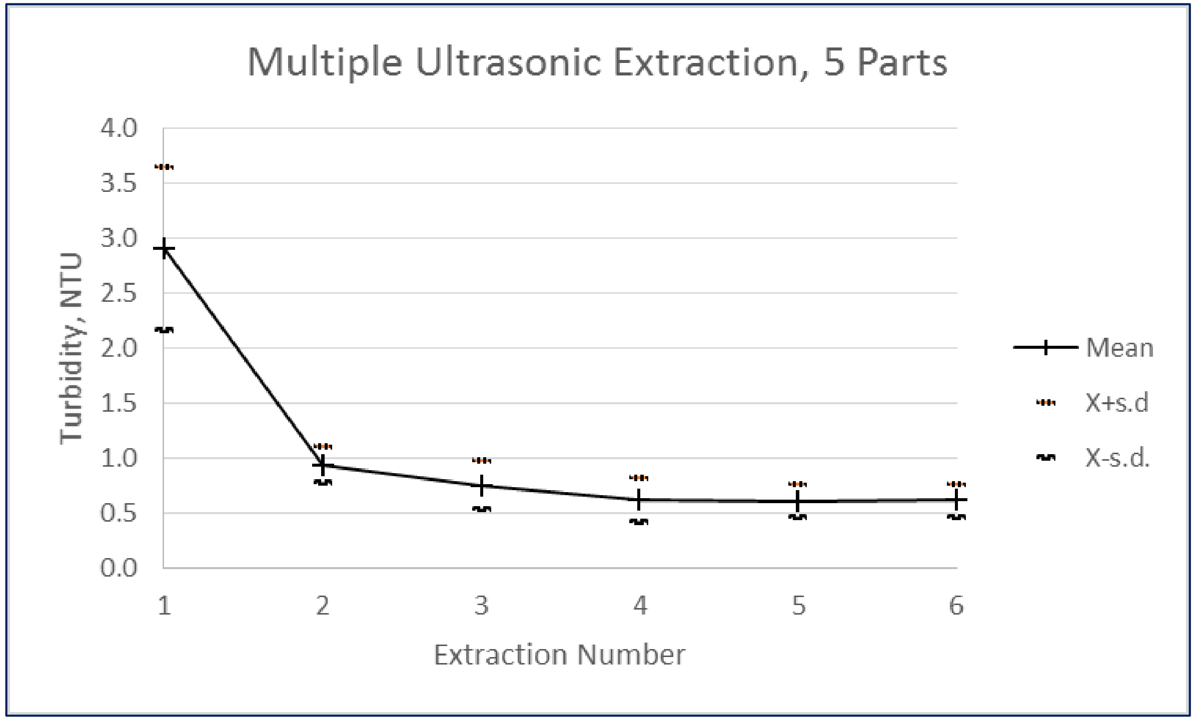
- A single part was subject to multiple u/s extraction (MUX) that produced an anomalous result
- Part was tested immediately after receipt, 2-9-2015
- No additional parts were tested until 3/26/2015 (wait for task order release)
- Anomalous result:
  - Initial cleanliness = 7.72 NTU
  - Asymptote = 0.65 NTU



Multiple Ultrasonic Extraction Results

- 5 additional parts were later tested to determine MUX curve

Part No.	Test Date	Initial NTU	Asym. NTU
21	3/26	4.21	0.83
28	3/30	2.46	0.75
9	4/01	2.65	0.54
1	4/02	2.70	0.49
4	4/03	2.49	0.56



- **Initial cleanliness significantly different from no. 8, but consistent within the group. Asymptote the same for all 6 MUX.**
- **Predict 3 minute ultrasonic cleaning time**

3 Minute Detergent/H_2O Ultrasonic Cleaning

Part No.	3 Minute Clean			Measurement		
	Blank, NTU	after 3 min, NTU	Net[1], NTU	Blank, NTU	Measure, NTU	Net[2], NTU
20	0.23	3.41	3.18	0.23	1.05	0.82
5	0.16	2.39	2.23	0.29	1.01	0.72
26	0.36	5.33	4.97	0.17	1.12	0.95
11	0.52	2.81	2.29	0.23	0.91	0.68
13	0.30	4.18	3.88	0.26	0.79	0.53

[1] Left in the beaker after cleaning. After 3 min. clean, the net NTU should be comparable to the sum of the net NTU in the MUX at 1 + 2 + 3 min.

[2] After 1 min. extraction The measured cleanliness should be comparable to the asymptotic cleanliness from the MUX

3 Minute Detergent/H_2O Ultrasonic Cleaning

Statistical analysis:

After 3 minute clean, the net NTU should be comparable to the sum of the net NTU in the MUX at 1 + 2 + 3 minutes

	3 min. clean, NTU	MUX (1+2+3), NTU
Mean	3.31	4.59
St. Dev.	1.152	0.855
N	5	5

P =0.0843
Not significantly different

The measured cleanliness should be comparable to the asymptotic cleanliness from the MUX

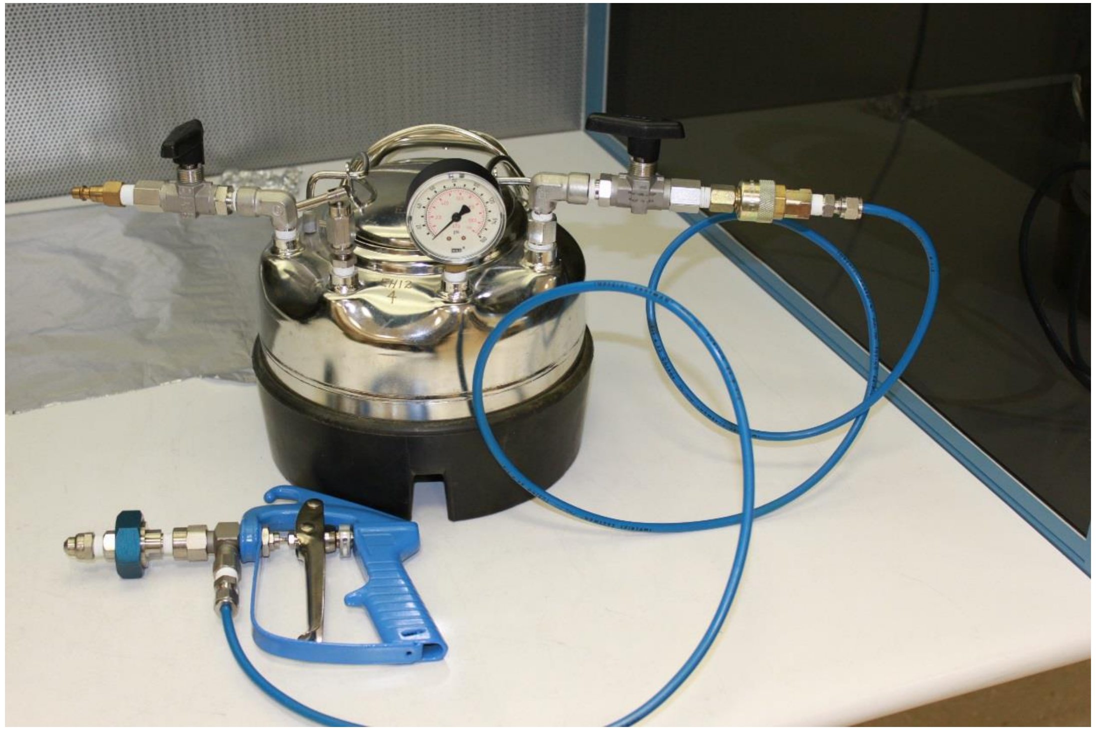
	After 3 min. u/s clean	Asymptote, MUX
Mean	0.74	0.62
St. Dev.	0.157	0.156
N	5	5

P =0.143
Not significantly different

3 Minute High Velocity H_2O Spray

- Millipore Filterjet w/ 5 µm filter at 50 ± 3 psig produces a finely collimated solid stream of liquid flowing at approximately 17.5 m/sec.

- **Parts were hand held and sprayed for 3 minutes with filtered distilled H_2O (no detergent)**
- **Parts were not allowed to dry, but were immediately extracted and turbidity measured**



3 Minute High Velocity H_2O Spray

Results:

Part No.	Blank, NTU	Measure, NTU	Net, NTU
27	0.46	1.88	1.42
7	0.09	1.48	1.39
2	0.20	2.27	2.07
23	0.19	2.27	2.08
18	0.23	0.81	0.58

Statistical analysis:

	After 3 min. spray clean	Asymptote, MUX
Mean	1.51	0.62
St. Dev.	0.616	0.156
N	5	5

P =0.0153
Significantly different

Conclusion: high velocity spray clean alone does not reach the asymptote and should not be considered a stand-alone cleaning process

3 Min. Ultrasonic in Detergent/H_2O + 1 Min. H_2O Spray

Results:

Part No.	After 3 Min. u/s Clean			After 1 Min. Spray		
	Blank, NTU	after 3 min, NTU	Net[1], NTU	Blank, NTU	Measure, NTU	Net[2], NTU
24	0.31	1.32	1.01	0.24	0.43	0.20
17	0.08	1.88	1.80	0.35	0.89	0.54
6	0.25	2.32	2.07	0.30	0.63	0.33
10	0.26	2.14	1.88	0.21	0.90	0.68
12	0.27	3.60	3.34	0.27	0.68	0.41

[1] Left in the beaker after 3 min. clean [2] After 1 min. extraction

- After 3 u/s min. clean, the net NTU should be comparable to the nets of previous 3 min. u/s clean.
- Measurement after spray rinse should compare to MUX asymptote
- Does spray rinse improve results of u/s wash alone?

3 Min. Ultrasonic in Detergent/H_2O + 1 Min. H_2O Spray

Statistical analysis:

	After 3 min. u/s clean	Prior 3 min. u/s clean
Mean	2.02	3.31
St. Dev.	0.839	1.15
N	5	5

P =0.0773
Not significantly different

	After 3 min. u/s + 1 min. spray	Asymptote, MUX
Mean	0.43	0.62
St. Dev.	0.189	0.156
N	5	5

P =0.1212
Not significantly different

	After 3 min. u/s + 1 min. spray	After 3 min. u/s clean
Mean	0.43	0.74
St. Dev.	0.189	0.157
N	5	5

P =0.0225, significantly different

- **Conclusion: Adding spray rinse improves cleanliness vs. 3 min. u/s clean alone**

3 Min. Ultrasonic Vapor Degrease in Solstice PF

- Solstice PF boils @ 19 °C (66 °F)
- Auxiliary cooling coil added to u/s tank to chill solvent below boiling point to ~12 °C (~54 °F)
- Two cleaning processes tested:
    - u/s clean for 3 min., then hold in vapor zone until condensation stops
    - u/s clean for 3 min., spray rinse with cold Solstice PF for 1 minute, then hold in vapor zone until condensation stops

3 Min. Ultrasonic Vapor Degrease in Solstice PF

u/s clean for 3 min., then hold in vapor zone until condensation stops

Results:

Part No.	Blank, NTU	Measure, NTU	Net, NTU
16	0.541	3.81	3.269
14	0.168	3.38	3.212
15	0.233	3.35	3.117
30	0.186	3.73	3.544

Statistical analysis:

	After 3 min. u/s + 1 min. spray	Asymptote, MUX
Mean	3.29	0.62
St. Dev.	0.183	0.156
N	4	5

P =0.0001
Extremely significantly different

3 Min Ultrasonic Vapor Degrease + 1 min Spray Rinse in Solstice PF

Results:

Part No.	Blank, NTU	Measure, NTU	Net, NTU
29	0.29	3.91	3.62
25	0.203	2.71	2.50
19	0.326	2.38	2.05
3	0.23	1.95	1.72
22	0.309	3.46	3.15

Statistical analysis:

	After 3 min. u/s + 1 min. spray	Asymptote, MUX
Mean	2.61	0.62
St. Dev.	0.779	0.156
N	5	5

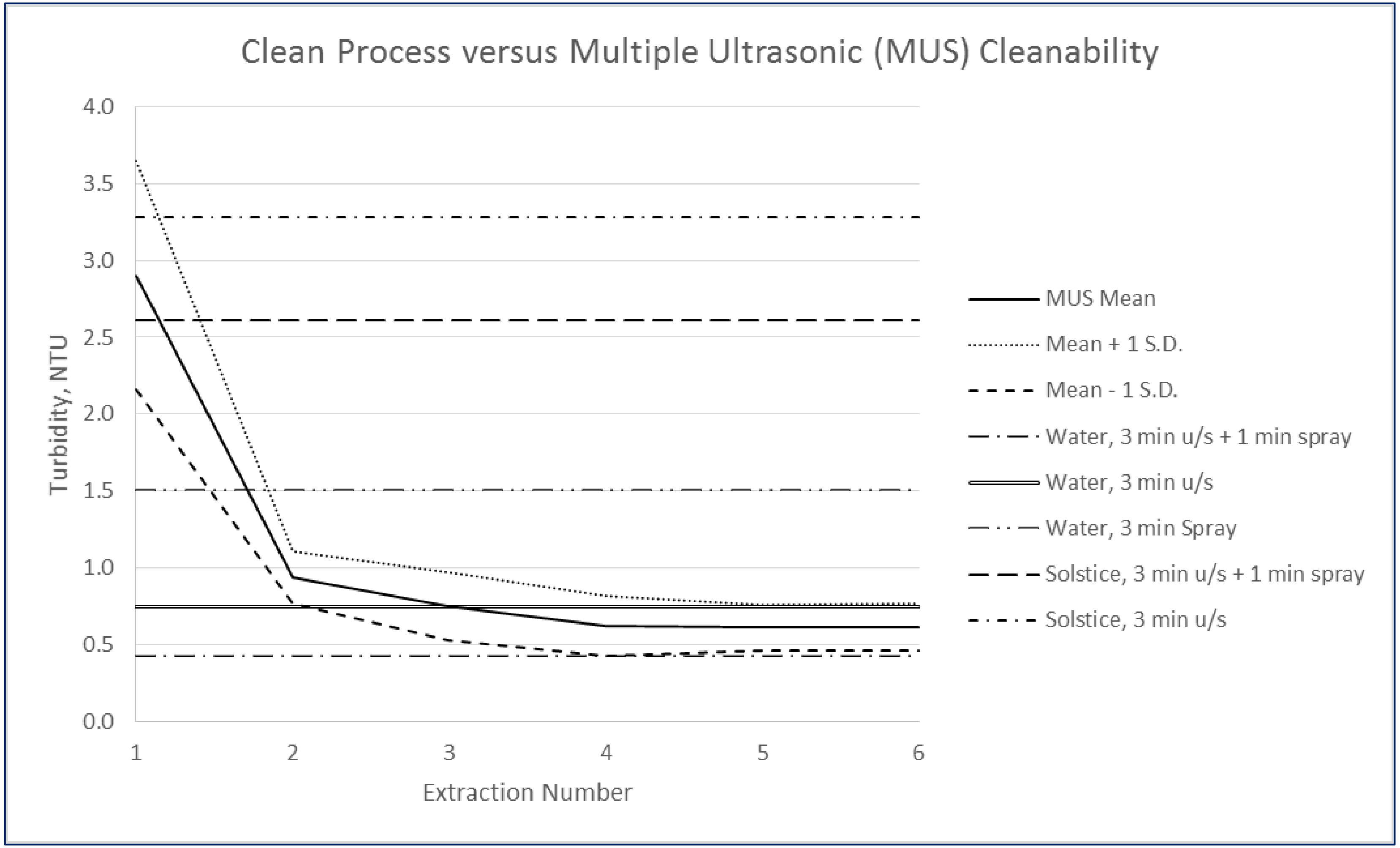
P =0.0001, Extremely significantly different

	After 3 min. u/s + 1 min. spray	After 3 min. u/s clean
Mean	2.61	3.29
St. Dev.	0.779	0.183
N	5	4

P =0.1355, Not significantly different

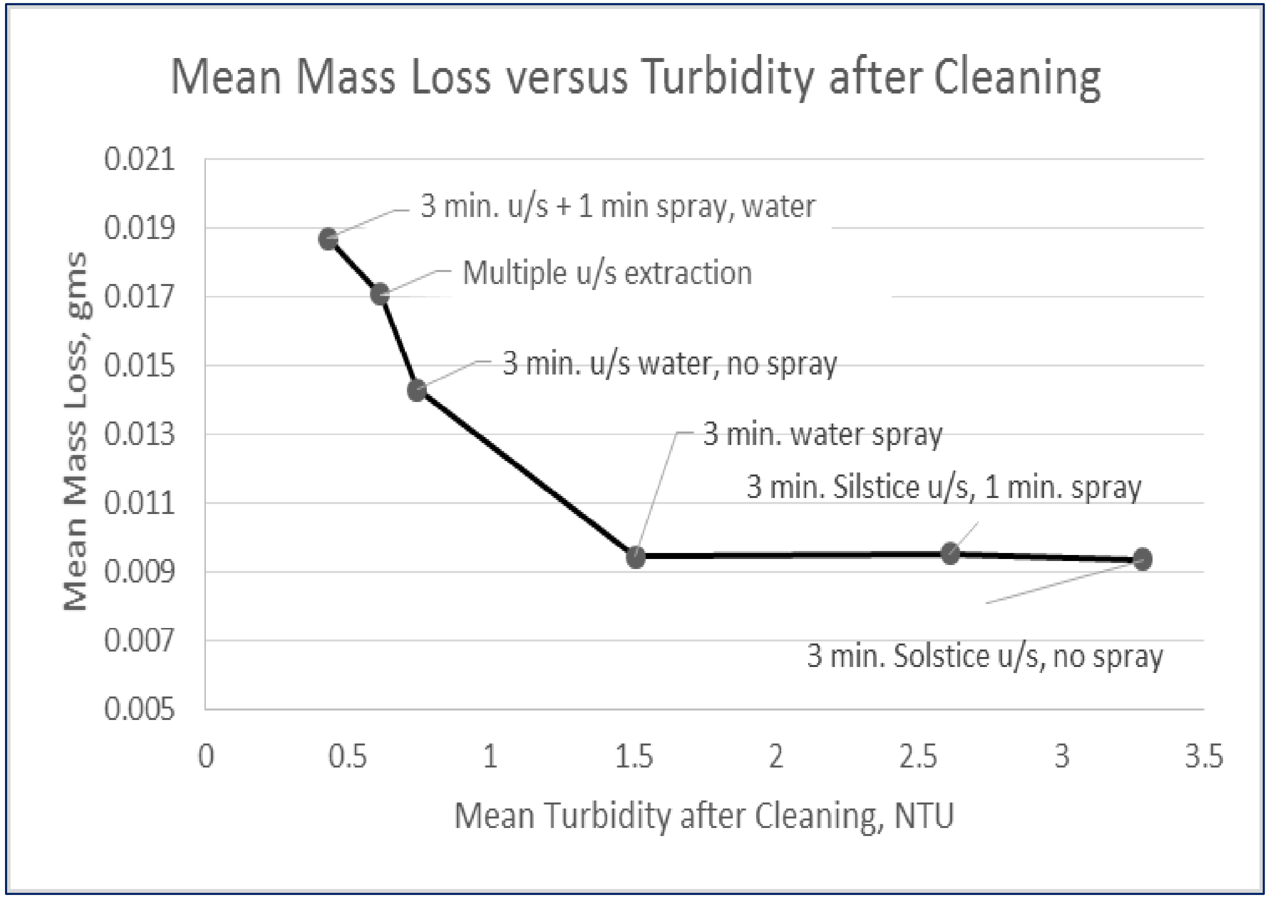
Graphic Representation of Cleaning Results

- **MUX shown as mean, mean plus 1 st.dev. and mean minus 1 st.dev. versus extraction number.**
- **Clean process shown as horizontal lines of mean values to show intercept versus initial cleanliness and asymptote of MUX**



Cleaning Results versus Mass Loss

- **Mass loss is negatively correlated with turbidity after cleaning (i.e., if little cleaning is done, most of the mass is left on the part and turbidity is high)**



Further Discussion – H_2O Based Cleaning

- H_2O based cleaning, based on residual turbidity after cleaning is in the order:
  - **Best: Detergent/H_2O u/s wash + high velocity spray rinse**
  - **Good: Detergent/H_2O u/s wash**
  - **Poor: High velocity H_2O spray clean**
- Detergent/H_2O u/s wash + spray rinse is significantly better than u/s wash alone (no rinse): This is not unexpected.
  - MUX shows that cavitation erosion occurs, though not catastrophic
  - Not rinsing with high velocity spray after u/s clean leaves the erosion debris on the parts, increasing measured turbidity after cleaning
  - This effect is seen as a lower net NTU for u/s + spray than the MUX asymptote

Further Discussion – Solstice PF

- Note similarity of turbidity of 3 min. u/s degrease in Solstice PF and 3 min. u/s degrease + 1 min. spray rinse in Solstice PF versus 1st extraction in MUX

	MUX, 1st Extract	Solstice Clean
Mean	2.90	2.91
St. Dev.	0.738	0.665
N	5	9

P =0.9838
Not statistically different

- This suggests very little cleaning is accomplished in u/s or u/s plus spray in the Solstice PF in the vapor degreaser.
- This result in not unexpected, as very low boiling point, low surface tension solvents don’t perform well in u/s cleaning of particles

Drying Experiments

- H_2O based cleaning introduces the problem of part drying
- Procedure:
  1. Weigh the parts to ± 0.0001 g (0.1 mg)
  2. Wet the parts with distilled water
  3. Blow off the parts, one at a time, with compressed air at 50 psig until sensibly dry

     3a. If heat dried, dry in the oven or vacuum oven

     3b. If heat dried, allow the parts to cool to room temperature before weighing

     3c. If using solvent displacement drying, blow dry the parts again and allow the parts to return to room temperature before weighing (solvent evaporation chills the parts)
  4. Weigh the parts to ± 0.0001 g (0.1 mg)
- For statistical analysis use a paired t-test (each part is weighed B4 and after)

Drying Experiments

- 50 psig Compressed Air Blow Off + room temperature over-night

	Dry Mass, g	**After Blow-Off, g**
Mean	32.4310	32.4334
St. Dev	0.6122	0.6124
N	21	21

P =0.0002, Extremely statistically significant

- Leaves an average 2.4 mg of water on the parts
- Not surprising, considering the fine-internal passageways in the part

Drying Experiments

- Blow-off plus 60 °C Convection Oven Dry

	Dry Mass, g	After 60 C Oven Dry, g
Mean	32.4310	32.4315
St. Dev	0.6122	0.6124
N	21	21

P =0.2498, Not statistically significant

- Blow-off plus 60 °C convection oven dry leaves an average of 0.5 mg of water on the parts.
- While not statistically significant on average, 7 of the 21 parts showed incomplete drying, with an average of 3.8 mg per part.

Drying Experiments

- 60 °C, Vacuum Oven, 25” Hg, No Preheat, Rapid Vacuum

	Dry Mass, g	After Blow-Off, g
Mean	32.4310	32.4319
St. Dev	0.6122	0.6125
N	21	21

P =0.1712, Not statistically significant

- Using the vacuum oven without preheating the parts and without controlling the rate the vacuum is applied leaves an average 0.9 mg of water on the parts
- This is caused by too rapid application of vacuum to a water wet part that has not been preheated, causing water to freeze in narrow internal passages.

Drying Experiments

- 60 °C, Vacuum Oven, 25 “ Hg, Preheat 10 min., Gradual Vacuum

	Dry Mass, g	After Blow-Off, g
Mean	32.4310	32.4309
St. Dev	0.6122	0.6127
N	21	21

P =0.4727, Not statistically significant

- Preheating the parts on a 60 °C surface and gradually reduces the vacuum dries the parts. The difference in mass, 0.1 mg, is within the measurement tolerance of the balance.

Cryo-shock Experiments

Procedure:

1. Prepare parts
    1. Select 4 parts from each test clean procedure group
    2. Measure turbidity in detergent H_2O solution
    3. Rinse with EtOH to remove detergent and solvent dry, oven bake @ 60 °C for 30 min.
    4. Cool to room temp. in desiccator and weigh
2. Select 2 parts from each test clean procedure group and immerse in LN_2 until boiling stops
3. Measure turbidity and reweigh all 4 parts
4. Determine if there is a difference between cryo-shocked parts and control parts that were not cryo-shocked

Cryo-shock Experiments

Results:

- 20 parts tested: 10 cryo-shock treatment and 10 handling controls
- No significant difference between cryo-shocked and not cryo-shocked (control) parts.

Statistical analysis (delta turbidity)

	Control	Cryo-shock
Mean	0.327	0.271
St. Dev.	0.239	0.278
N	10	10

P =0.766, Not significantly different

LN2 cryo-shock does not have a significant effect on part cleanliness

Thank you for your attention!

Questions?

Interested in Collaborating on Cleaning Processes?
Please contact:

Mark Mitchell
NASA/MSFC/EM50
mark.a.mitchell@nasa.gov