ULTRA PURE WATER CLEANING BASELINE STUDY ON NASA JSC ASTROMATERIAL CURATION GLOVEBOXES. M. J. Calaway[1], P. J. Burkett[1], J. H. Allton[2], and C. C. Allen[2]. [1] Jacobs Technology (ESCG) at NASA Johnson Space Center, Astromaterial Acquisition and Curation Office, Houston, TX; [2] NASA Johnson Space Center, Astromaterial Acquisition and Curation Office, Houston, TX; michael.calaway@nasa.gov.

Introduction: Future sample return missions will require strict protocols and procedures for reducing inorganic and organic contamination in isolation containment systems. In 2012, a baseline study was orchestrated to establish the current state of organic cleanliness in gloveboxes used by NASA JSC astromaterials curation labs [1, 2]. As part of this in-depth organic study, the current curatorial technical support procedure (TSP) 23 was used for cleaning the gloveboxes with ultra pure water (UPW) [3-5]. Particle counts and identification were obtained that could be used as a benchmark for future mission designs that require glovebox decontamination.

Background: Historically, the Lunar sample curatorial facility cleaning procedures for contamination control JSC 03243 in 1981 for cabinet (glovebox) nominal cleaning (similar to the 1971 procedure) used:

- Acid wash 2% nitric acid solution with distilled water (when required)
- 1% Oakite Liquidet detergent solution with DI water
- Mechanical scrubbing with nylon brushes, scouring pads, Scotch Brite pads and stainless steel toothbrushes
- Rinses with distilled water
- Isopropyl alcohol (IPA) rinse and gaseous nitrogen (GN_2) dry
- Blacklight inspection
- 1% Liquidet solution with distilled water (Millipore can at 85 psig)
- IPA rinse (Millipore can at 85 psig)
- Vacuum flask for liquid pickup and squeegee with GN_2 dry
- Acid Wash 2% nitric acid solution with distilled water (when required)
- Freon 113 (Millipore can at 85 psig)
- Perform particle counts, total hydrocarbon counts (THC), and non-volatile residue (NVR) analysis

A separate degreasing procedure for gloveboxes mainly used high pressurized Freon 113 and 2% nitric acid wash along with mechanical scrubbing. In 1994, UPW replaced Freon cleaning in JSC curation.

UPW cleaning: The 2012 TSP 23 glovebox cleaning uses pressurized UPW with resistivity >18 MΩ, total organic carbon (TOC) < 5 ppb, and heated to 70°C. Two nitrogen enriched gloveboxes were cleaned: Lunar Curation Glovebox (LCG), Apollo 11 processing cabinet #307-41 and the Advanced Curation Glovebox (ACG). The LCG was manufactured with 316L stainless steel, glass and polycarbonate (Lexan) windows, viton gaskets, and six neoprene gloves. The ACG was manufactured with 304 stainless steel (electropolished), glass viewing windows, viton gasket seals, and four hypalon gloves. For this cleaning, the UPW was set at 52°C and GN_2 was flowing in the glovebox at 50 scfh. PFA tubing and nozzle were used for rinsing through glove ports covered with Teflon film. At least three rinses were done with rinse water being collected after each rinse with 47 mm diameter 0.8 µm Millipore filter pad composed of a mixed cellulose ester (MCE) for particle counting and cleaning verification. In addition, the LCG used two 185 mm diameter Whatman 41 ashless 20µm cellulose filters were used to trap lunar material from the initial rinses. After the final rinse, the Millipore filters were observed under an optical stereomicroscope to meet military standard (MIL-STD) 1246C cleanliness Level 50. TSP 23 procedure states that glovebox rinses will continue "until no further significant decrease in particles is attained and there are no more than 50 particles >10 microns."

Test Results: After TSP 23 procedure deemed gloveboxes were cleaned, all UPW collected from each rinse was analyzed on a HIAC liquid syringe sampler and particle counter in the Genesis Advanced Precision Cleaning Laboratory ISO class 4 cleanroom. Table 1 shows the final rinse particle counts compared with MIL-STD Level 50. The observation of particles < 15 µm are difficult to resolve with the optical stereomicroscope used in TSP 23. The liquid particle counts show that at 25 µm diameter particle size, each glovebox passes MIL-STD Level 50 cleanliness standards. However, at 1 µm particle size, both gloveboxes have large particle loads for stainless steel. Based on these results, the LCG is cleaner than the ACG. This is probably a result of the early Lunar degreasing and cleaning procedure while ACG was never degreased.

Particle Size (µm)	Average UPW Control (Count per 10 ml)	Lunar Glovebox Final Rinse Average	Advanced Curation Glovebox Final Rinse Average	MIL-STD Level 50 (Count per 10 ml)
1	19.5	1246.6	13289.75	
3	1	128.8	1806.75	
5	0.25	40.2	707.75	5.3
10	0.25	6.2	26.75	
25	0	0.6	0.5	0.34
50	0	0.4	0.25	
100	0	0.4	0.25	
150	0	0.4	0.25	

Table 1: Final rinse liquid particle counts

Possible Identified Material Groups	Particle %	Size Range (µm)	Average Size (µm)
Aluminum	1.12%	25	25.0
Aluminum Oxide	1.12%	12	12.0
Iron Oxide	1.12%	5	5.0
Stainless Steel	25.84%	2 to 10	4.7
Lunar Geologic Material	26.97%	2 to 15	6.1
Lunar Geologic Ilmenite	7.87%	3 to 8	7.5
Organic Compound	30.34%	2 to 100	16.4
Hydrocarbon	1.12%	10	10.0
Silicone	4.49%	4 to 8	6.3

Table 2: LCG Millipore filter with 89 particles counted

Possible Identified Material Groups	Particle %	Size Range (µm)	Average Size (µm)
Aluminum	1.63%	3 to 10	6.0
Calcium Carbonate	2.44%	1 to 5	2.5
Copper Sulfate	1.63%	1 to 5	3.8
Iron Oxide	6.91%	3 to 30	8.9
Iron Phosphate	0.81%	3 to 10	2.0
Iron Sulfide	0.81%	2 to 2	6.5
Potassium carbonate	0.41%	5	5.0
Geologic Silicate Mineral	20.73%	1 to 25	7.1
Silver Cadmium Oxide	1.22%	5 to 10	8.3
Stainless Steel	6.50%	0.5 to 10	4.5
Fluorosilicone	4.07%	1 to 25	6.0
Hydrocarbon	2.03%	5 to 50	24.0
Organic Compound	38.62%	0.5 to 100	17.8
Silicone	12.20%	1 to 25	6.3

Table 3: ACG Millipore filter with 246 particles counted

Particle identification was completed by analyzing both Millipore and Whatman filters on a JEOL JSM-7600F field emission scanning electron microscope (FE-SEM). 392 particles were surveyed with over 450 spot analyses with a low angled backscatter electron (LABe) detector. Table 2 and 3 show possible material groups from the Millipore filter final rinses. Fig. 1 provides a more generalized grouping of material identification.

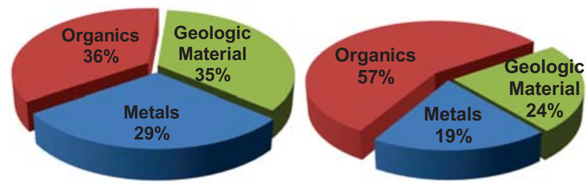

Fig. 1: LCG (left) and ACG (right) general particle groups identified after final rinse

Discussion: The particle counts and identification results provide a baseline for inorganic and organic contamination load on a cleaned glovebox using TSP 23. The elevated organic load in the SEM results of ACG when compared to the LCG is further evidence that the ACG was never degreased. In addition, particles maybe trapped at the interface of window and port seals. Both gloveboxes were not cleaned in laminar flow cleanrooms and cross-contamination may have occurred that elevated the levels. During microscopic inspection of cleanliness, human error could be mitigated by the use of a liquid particle counter and other inspection tools to provide quality assurance of cleanliness.

Summary: The UPW baseline study demonstrates that TSP 23 works well for gloveboxes that have been thoroughly degreased. However, TSP 23 could be augmented to provide even better glovebox decontamination. JSC 03243 could be used as a starting point for further investigating optimal cleaning techniques and procedures. DuPont Vertrel XF or other chemical substitutes to replace Freon-113, mechanical scrubbing, and newer technology could be used to enhance glovebox cleanliness in addition to high purity UPW final rinsing. Future sample return missions will significantly benefit from further cleaning studies to reduce inorganic and organic contamination.

Reference: [1] Calaway M.J. et al. (2012) *LPS LXXV*, this volume; [2] Allen C.C. et al. (2011) *Chemie der Erde Geochem.* 71(1), 1-20; [3] Allton J.H. et al. (2006) *LPS XXXVII*, 2324; [4] Allton J.H. et al. (2007) *LPS XXXVIII*, 2138; [5] Calaway M.J. et al. (2009) *LPS XL*, 1183.