CLEANING THE ANGSA COLD CURATION APOLLO-ERA GLOVEBOX – TRAINING THE NEXT GENERATION

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Introduction: The goal of the Apollo Next Generation Sample Analysis (ANGSA) Program is to study “specially curated samples,” that is, unstudied Apollo samples that have been preserved under unique conditions. A subset of the funded ANGSA work includes the study of Apollo samples stored under cold conditions since approximately one month after their return from the Moon. Ongoing ANGSA processing and future returned samples require cold curation strategies to preserve the integrity of organic and other temperature-sensitive constituents within those samples.

Standard Apollo curation cleaning processes and procedures are designed to protect lunar samples from particulate and general atmospheric and anthropogenic contamination. This abstract details the modifications to existing cleaning procedures in preparation for the first cold curation sample processing at Johnson Space Center. Future publications will provide further details on the low-temperature materials and operational procedures used for this effort. This work not only supports Apollo but also future cold sample return missions [1].

Background: Processing cold Apollo samples requires a reassessment of the materials used in sample cabinetry (glovebox), instrumentation, and cleaning procedures. Initial efforts in 2018 and 2019 focused on adapting an Apollo-era wire-saw glovebox into a cold-compatible ANGSA sample processing facility while complying with Apollo curation cleanliness and materials restrictions [2]. In 2020, the glovebox was cleaned in preparation for installation into a walk-in freezer to allow for sample processing at -20°C. The standard cleaning procedure was updated to comply with the new environmental regulations and laboratory requirements.

Revised cleaning process: The ANGSA glovebox was originally cleaned following standard procedures decades ago. However, the glovebox was then exposed to significant contamination while stored outside of the clean laboratories. This led to a full precision cleaning as if the glovebox were brand new, straight from a manufacturer. Updated glovebox cleaning procedures included the following steps: 1) nitric acid to remove trace lead from the interior glovebox floor, 2) chemical degreaser scrubbing and wash, 3) hydrogen peroxide disinfectant wash, 4) ultrapure water (UPW) rinse until the particle count in a sample collected from a drain port reached an acceptable level.

The current cleaning procedure on the Apollo processing gloveboxes only utilizes hot UPW washes because they have not left the clean environment of the Apollo sample laboratory. Therefore, the benefits of revisiting the precision cleaning process are two-fold: development of a refined procedure and training the next generation of technicians in preparation for future sample returns.

ANGSA cold sample processing requires the glovebox to meet Apollo particulate cleanliness levels, minimize organic chemical use and off-gassing materials, and ensure sterility to the greatest extent possible. The glovebox was bio-swabbed before and after each major step in the cleaning process to monitor sterility.

Nitric acid wash: The purpose of a nitric acid wash is to dissolve any residual lead on the glovebox floor. No current cleaning technician has performed a nitric acid wash, therefore we performed a trial run with water before executing the acid wash. The nitric acid washes were completed with the window and glove ports intact to protect the technicians from chemical exposure.

The washes involved two 30-minute sessions where the floor of the cabinet was filled with 0.5-inch depth 2% nitric acid (approximately 5-6 L). Every 15 minutes, the solution was mixed with a stainless steel paddle. The end of the glovebox opposite the drain port was raised 3-4 inches to facilitate drainage from the main chamber between washes. After the second wash, the glovebox was rinsed with UPW until waste stream reached a neutral pH.

After the nitric acid washes, the glovebox was completely disassembled. All nuts, bolts, washers, ports, etc. were cleaned following standard Apollo practices. The ANGSA team then reassembled and leak-tested the glovebox following the procedure developed in 2019 [2]. The glovebox was continuously purged with curation-grade N2 gas until the next step in the cleaning process.
**Isopropanol wash:** Original curation cleaning procedures called for a now obsolete degreasing chemical followed by an isopropanol scrub. For the purposes of this project, two isopropanol washes served as the only degreasing step. Technicians filled material-approved spray bottles with isopropanol and thoroughly coated all interior surfaces of the antechamber and main portion of the glovebox. Then, the interior surfaces were scrubbed with cleaned polypropylene brushes. Curation-grade nitrogen gas purged the glovebox for 5-6 days before the hydrogen peroxide disinfectant wash.

**Hydrogen peroxide wash:** Microbial swabs up to this point in the cleaning process showed some microbial growth. Analytical reagent-grade 30% hydrogen peroxide was diluted to 7.5%. Cleaned spray bottles filled with the diluted solution were used to coat all interior surfaces. The cleaning technicians then wiped the interior with particle-free polyester wipes saturated with hydrogen peroxide. After 30-60 minutes [3], the glovebox was dried with dry polyester wipes. Microbial swabbing inside the glovebox after the hydrogen peroxide wash showed no evidence of colony formation. As with the other washes, the glovebox was purged with curation-grade nitrogen gas for multiple days before the UPW wash.

**Ultrapure water (UPW) wash:** All actively used curation gloveboxes undergo routine UPW washes. The purpose of UPW washes is to remove any particulate matter generated by sample processing activities. This step is considered final “precision cleaning.” At this point, the lead has been dissolved from the floor of the cabinet and the interior surfaces have been degreased and disinfected; all that remained were the particles generated from the assembly and initial cleaning processes. A glovebox is considered ‘clean’ when the particle count in a collected drain sample is JPR 5322.1H Level 50 clean or better. [4]

Most curation gloveboxes are made of only stainless steel, which is able to withstand >120°F ultrapure water. However, the back panel and antechamber door hinge for the ANGSA glovebox are made of aluminum. Aluminum readily oxidizes when exposed to hot UPW. Therefore, the UPW used for this precision cleaning was not heated.

The baseline particle count was established by collecting a sample directly from the UPW wand, filtering the water sample through 0.8-μm filter, and particles counted via microscope. Particle counts were collected every 10 minutes during the UPW wash. After five sample collections, the particle count returned to the baseline value. Figure 1 shows one of the cleaning technicians using the UPW wand to rinse the interior of the glovebox.

Once the glovebox surpassed cleanliness Level 50, all of the UPW was dried using curation-grade nitrogen gas. Clean Apollo-standard gloves were attached to the glove ports. Since the completion of the UPW wash, the ANGSA glovebox was draped with one layer of nylon plastic wrap and has been continuously purged with nitrogen, see Figure 2.

**Results and Discussion:** The transition from a room-temperature Apollo-era glovebox into a modern, cold, sample processing facility has presented both challenges and opportunities. Revitalizing the heritage equipment and cleaning procedures into an operational cold curation facility while maintaining the established cleanliness and materials requirements has been accomplished. Extensive knowledge gained from this project will facilitate future cold sample curation efforts.

**References:**


