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# ONE SMALL STEP FOR MAN, ONE GIANT LEAP FOR CONSERVATION

## PAUL MARDIKIAN, CLAUDIA CHEMELLO, AND JERRAD ALEXANDER

This article describes the technical challenges of stabilizing and conserving 25,000 lb. (12.5 tons) of Apollo-era (1969–1972) Saturn V rocket engine parts that were recovered off the coast of Florida in 2013. After more than 40 years on the floor of the Atlantic Ocean, the engines had suffered extreme levels of deterioration and corrosion. Treatment was accomplished by taking a deliberate, archaeological object–based treatment approach for these composite objects that are both modern technological marvels and marine archaeological artifacts. The article describes the archaeological treatment approach adopted and the results of selected analytical work undertaken to understand the materials of construction and the deterioration processes.

KEYWORDS: Conservation, Marine, Archaeology, Aerospace heritage, NASA Apollo 11, Superalloys, Characterization, Corrosion, Stabilization, Composite materials, Corrosion inhibitors

# 1. INTRODUCTION

In March 2013, 25,000 lb. (12.5 tons) of Apollo-era (1969–1972) *Saturn V* rocket engine parts from several Apollo missions were recovered off the coast of Florida after more than 40 years on the floor of the Atlantic Ocean. With the support of the National Aeronautics and Space Administration (NASA), Jeff Bezos, Amazon's founder and chief executive, sponsored the expedition with the objective of finding the engines from Apollo 11. The engine parts were recovered from a debris field of 300 mi.<sup>2</sup> at a depth of approximately 14,000 ft. (4,300 m) using deep-sea sonar and remotely operated vehicles (ROVs). After recovery, the artifacts were transported to the Kansas Cosmosphere and Space Center in Hutchinson, Kansas, to undergo excavation, identification, documentation, and conservation in a custom-designed facility. The recovery initiated exceptional press coverage and launched an unprecedented conservation project to preserve the most powerful, yet disposable, liquid-fueled engines that were ever made.

One of the chief difficulties of the project was planning conservation strategies for modern marine artifacts whose size, composition, and state of preservation were virtually unknown prior to their arrival in the lab. These oversize, complex artifacts are made from a combination of superalloys and other modern materials, many of which are unfamiliar to conservators. A superalloy is a high-performance alloy that exhibits several critical characteristics at high temperature, including outstanding corrosion resistance, high strength, excellent surface stability, and resistance to creep (Reed 2006). Identifying what materials were present was surprisingly difficult due to a scarcity of information about the composition of the engines and a veil of secrecy that still prevails after half a century.

The size and weight of the engine parts presented additional practical challenges for treatment, as did the extraordinary levels of deformation and corrosion alongside almost completely pristine metal. Addressing immediate stabilization concerns, such as keeping the artifacts wet at all times to prevent deterioration, added to the complexity and required a creative and flexible workspace and conservation approach.

Developing stabilization treatments that were compatible with multimetal artifacts propelled the general principles applicable to the conservation of marine archaeological artifacts to a completely different level. The decision to separate the different components for treatment or adopt a more holistic preservation philosophy was decided on a case-by-case basis. A successful multidisciplinary dialogue ensued and prompted an effective integration of conservation theory and practice to understand the level of deterioration of the engines and to determine how to best preserve the collection.

# 2. BACKGROUND AND HISTORY

# 2.1 THE APOLLO PROGRAM 1969-1972.

The Apollo program was NASA's third human spaceflight program, with the explicit goal of a manned lunar landing. Six of the missions (Apollo 11, 12, 14, 15, 16, and 17) achieved this goal. The Apollo missions began in 1961 and concluded in 1972, with a total of 12 missions flown. The lunar missions continued until Apollo 17 in 1972. The Apollo missions used the *Saturn V* rocket, an enormous multistage vehicle with its first stage powered by five F-1 engines as launch vehicles. The launch of Skylab in May 1973 marked the final flight of the gigantic *Saturn V* rocket and its F-1 engines. By the end of the Apollo program, a total of 65 F-1 engines were flown and lost at sea.

## 2.2 THE F-1 ENGINE

The development of the F-1 began with the United States Air Force in 1955 using materials and methods that were cutting-edge at the time. Once the Apollo program was under way, the F-1 was selected to power the first stage, the S1-C, of the *Saturn V* rockets that would carry mankind to the moon. The first test flight of the engines came in 1967 on the unmanned Apollo 4 mission. Two years later, on July 16, 1969, Apollo 11 lifted off, powered by five F-1 engines reaching an altitude of 65 km and speeds in excess of Mach 7 (8,643 kph) in less than two and a half minutes. Explosive bolts then fired, separating the first stage from the rest of the rocket, which plunged into the Atlantic Ocean.

The F-1 engines are a marvel of modern engineering and ingenuity. More than four decades after they were last used, they still hold the title of the most powerful liquid-fueled engines ever created. Each engine essentially consisted of a thrust chamber, exhaust nozzle, turbopump, injector assembly, turbine, and heat exchanger. A fully assembled F-1 was nearly 6 m tall, 4 m wide, and weighed more than 8,000 kg (fig. 1). Each engine generated a staggering 700,000 kg of thrust, consuming a mixture of 976 L of highly

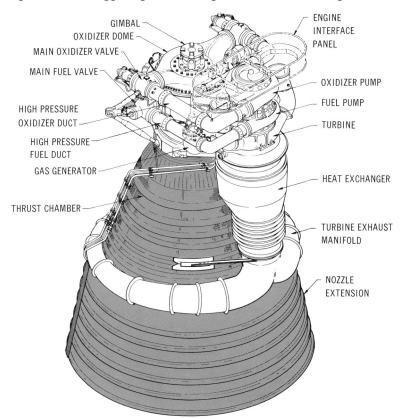


Fig. 1. An illustration of the early production F-1 engine (Courtesy of Wikipedia Commons)

refined kerosene and 1,565 L of liquid oxygen per second. By comparison, the space shuttle's three main engines collectively generated 544,000 kg of thrust. The thrust chamber and exhaust nozzle were constructed of a nickel-based superalloy called *Inconel X-750* to withstand the 3,300°C temperatures generated by the combustion process. The fuel and liquid oxygen were supplied via a turbopump driven by a 53,000-horsepower turbine. The fuels were kept separated until they reached the injector, which meters the mixture ratio and pressure to the thrust chamber. The heat exchanger acts as a sort of radiator, cooling exhaust gases from the turbine before they are introduced into the main exhaust nozzle.

## 2.3 RECOVERY

In March 2011, Jeff Bezos announced plans to find and recover the engines from Apollo 11, the historical mission that took man to the moon in 1969. The first step was to locate 5 engines out of 65 launched on *Saturn V* during the Apollo era from 1969 to 1972, 14,000 ft. (4,300 m) below sea level. A survey mission was planned with a 200-ft. vessel, the *Ocean Stalwart*, outfitted with specialized navigation and computer systems, and a 6-ton towfish containing state-of-the-art Synthetic Aperture Sonar capable of capturing high-resolution details at distances of more than 1,000 m.

Storms in the Atlantic caused difficulties and delays on the survey mission. Additionally, NASA's calculated trajectories for the S-1C stages did not account for the rockets breaking up on impact with the ocean surface and subsequent drifting on their decent to the seafloor. An initial 100 mi.<sup>2</sup> search area turned in to nearly 200 mi.<sup>2</sup> with hundreds of pieces of debris strewn across vast areas rather than the highly localized groupings the team expected (Capone 2013, 2014).

The recovery itself required its own set of specialized crew and technologically advanced equipment. The recovery mission employed the *Seabed Worker*, a six story tall, 300 ft. long recovery ship that utilizes sophisticated station-keeping and GPS systems to maintain precise positioning on the ocean surface. It carried two ROVs capable of working at depths of 5,000 m to video, photograph, dredge, and attach recovery slings and cables to the artifacts (fig. 2). A massive winch was used to hoist the recovered



Fig. 2. ROV during the excavation of a thrust chamber (still of video capture) (Courtesy of Bezos 2014)

engine components, safely contained in large recovery cradles, to the surface, often requiring lifting tensions in excess of 9 tons.

Nearly 25,000 lb. of engine components were recovered in approximately three weeks and brought safely to shore at Cape Canaveral, Florida, close to where they had originally been launched. While aboard the ship, the engine components were rinsed intermittently with fresh water. Recovered artifacts included five thrust chambers, three liquid oxygen (LOX) domes, three injector assemblies, two turbopumps, one exhaust nozzle, two heat exchangers, four turbines, three turbine manifolds, and one gas generator from various Apollo missions. Once offloaded from the ship, the engine parts were cocooned in moistened cotton towels and shrink-wrapped before being transferred to flatbed trailers for their journey to the Kansas Cosmosphere and Space Center.

# 3. PLANNING FOR THE ARRIVAL OF THE ENGINES 3.1 TREATMENT FACILITY

One of the main challenges faced by conservators as the project began to unfold was the initial lack of information about the number of engines that would be recovered and their state of preservation. Due to an estimated speed between 400 and 500 mph (644–805 kph) at the time they hit the surface of the water, partial or complete breakage was anticipated. In contrast, expectations of the recovery team suggested that complete engines might be recovered. A contingency plan to accommodate whole engines measuring 18.5 ft. in height and 12.2 ft. in diameter, and weighing 18,500 lb. (8,400 kg), had to be in place at the time of recovery—particularly to address the size of the recovery baskets to be constructed and the lab space needed to accommodate them.

Identifying a suitable facility that could adequately accommodate the conservation of an unknown number of engines in whatever conditions they were received, whole or disarticulated, was critical to the success of the project. Fortunately, the Kansas Cosmosphere and Space Center had a 5,000 ft.<sup>2</sup> building available. The building was retrofitted from an artifact storage space to a turnkey conservation lab in less than two months. The building had several important key features, such as large overhead doors allowing access for large trucks or forklifts, a ceiling height of 35 ft., and a flat continuous concrete floor slab throughout, allowing the use of a mobile 5-ton gantry crane, pallet jacks, and forklifts. The building was equipped with a powerful HVAC system, deionized water filtration system, eye wash station, compressed air, a powerful light system, Internet access, 24/7 security access, and surveillance.

The concrete slab extended outside the lab to a large fenced area where wet treatment and cleaning could be carried out. In addition, an observation gallery independent from the lab space was constructed to allow access to visitors without interfering with the conservation work. The conservation facility was also adjacent to a large and fully equipped workshop where supports, lifting devices, and other custom-made tools could be produced as needed by the metal fabricators on staff.

Keeping the entire collection wet while conservation plans were developed was achieved with a "showering system" installed around two 16 ft. × 23 ft. × 25 in. shallow custom-fabricated basins lined with a waterproof membrane covered with thick rubber pads to avoid puncture. The showering system used city water, and the artifacts were manually sprayed once per day with a 0.1% solution of FlashCorr, an anionic surfactant and multimetal corrosion inhibitor made by Cortec Corporation. Sump pumps were used for filtration and recirculation inside the basins. This flexible system was chosen for initial receipt of the engine parts in lieu of individual tanks due to the unknown size and shape of the objects. The objects remained in the showering system for up to five months, until they were ready for individual treatment. During that time, they were easily accessible for initial cataloguing, documentation, examination, and cleaning (figs. 3a, 3b). For subsequent treatment of individual engine parts, custom-made fiberglass tanks, as used in the oil industry, replaced the two large basins.





Fig. 3a. View of the lab with recently recovered artifacts in custom-fabricated basins; 3b. Detailed view of basin #1 with turbopumps in the foreground (Courtesy of Terra Mare Conservation LLC)

## 3.2 DEVELOPING A CONSERVATION STRATEGY

The challenge presented by the conservation of the Apollo engines was immediately apparent: how to comprehend and preserve a large collection of 20th century aerospace heritage after burial in an aggressive marine environment? It was obvious to the authors that a treatment strategy for these modern materials, many of which are unknown in the conservation sphere, would require a plan that was guided by archaeological conservation standards to ensure a logical work plan that respected the context of the objects, as well as their unique materials, and ensured long-term preservation.

Conservators of archaeological materials face difficult and unique challenges in the preservation of objects from buried contexts whether wet or dry, particularly for metal and composite objects. Among these challenges is the often rapid and irreversible deterioration that can result from recovery and exposure to air, particularly for objects from a marine or wet environment. Acute fragility or structural collapse upon excavation, extreme deterioration due to corrosion and burial position, particularly if chloride ions are present in the burial context, accumulated dirt, and sediment or concretions overlying the original surface obscuring detail and rendering the object illegible are typical issues faced by conservators of archaeological materials.

An additional requirement when dealing with archaeological material is to retain as much contextual information as possible, particularly from the objects' use and history. Archaeological objects are often transformed by their environment, creating difficulties of interpretation. Evidence of the firing of the F-1 engines had left subtle surface traces, some of which were ephemeral. Preserving evidence of use was critical and one of the most important components of the archaeological approach adopted for treatment (figs. 4a, 4b, 5).



Fig. 4a. Example of a stenciled number on a thrust chamber only visible due to the presence of rust surrounding the numbers (Courtesy of Terra Mare Conservation LLC)



Fig. 4b. Original unit number on Apollo 11 thrust chamber only visible under ultraviolet light (Courtesy of Kansas Cosmosphere and Space Center)

The deformation of the engines upon impact with the ocean also formed an important part of each object's history and needed to be preserved. The level of deformation was quite extensive in some cases, as shown on thrust chamber F1-2013-0005 from Apollo 11 (fig. 6).

The engines' short life history, followed by burial for more than four decades on the bottom of the ocean, left us wondering what kind of information, if any, would be preserved, and how we would reveal and preserve that information for objects that had a fairly secretive life history, followed by a life span of only a few minutes. The following sequential approach was taken in response to these difficult questions:

- 1. Document and assess the collection as rapidly as possible while keeping the objects wet at all times.
- 2. Initial cleaning to remove compacted sediment, followed by rinsing and daily tracking of chloride levels, conductivity, pH, and other metals in solution, particularly copper.
- 3. Separate engine components when feasible and document the process.
- 4. Rotate the objects when possible and continue systematic cleaning to remove accumulated sediment and corrosion products.
- 5. Fabricate engine mounts to support the most fragile artifacts or modify their angle to facilitate stabilization.
- 6. Mission identification.
- 7. Transfer each artifact into individual tanks for stabilization treatment—chemical cleaning and chloride removal.



Fig. 5. Soot deposition on the interior of the heat exchanger F1-2013-0015 from Apollo 11 (Courtesy of Terra Mare Conservation LLC)



Fig. 6. Ruptured jacket from a thrust chamber belonging to engine #5 from Apollo 11 (Courtesy of Terra Mare Conservation LLC)

- 8. Extended rinsing to remove treatment chemicals in warmed deionized water.
- 9. Mechanical cleaning as required in conjunction with pressure washing.
- 10. Apply a suitable corrosion inhibitor for multimetal objects.
- 11. Dry the objects both internally and externally with compressed air or molecular sieve.
- 12. Final finishing and coating, if needed.

## 4. DOCUMENTATION

Initial documentation of the artifacts upon their arrival in the lab was based on the archaeological numbers assigned during the survey and recovery operations at sea. Smaller objects and those removed for treatment were numbered with an expanding alphanumerical system based on these target designations. This facilitated the tracking of each major component to its original recovery position, as well as tracking each sub-component to its parent during dismantling and treatment.

Each object was also given individual treatment tracking documents that were created based on a standard United States Air Force aircraft maintenance form, AF Form 791A. Parameters such as weight, dimensions, treatment protocols, and actions taken could be readily documented and included the initials of the person responsible. These details were then transcribed into a custom computer database built using FileMaker Pro 12. The database entries included materials identification along with technical descriptions, condition reports, identifying markings, and photographs.

## 5. MISSION IDENTIFICATION

Matching the engine components to the specific mission on which they flew would prove to be one of the most difficult tasks of the project for several reasons. First, the recovery area was vast, and the engines were heavily damaged, corroded, embedded in sediment, and covered with stains and layers of corrosion products, all of which obscured the markings necessary to identify the components. Second, detailed documentation including individual part and serial numbers was not available. Rocketdyne, the company that produced the engines, did have a knowledge-retention program in place at the time of the F-1's development. However, due to the proprietary nature of the documents, four decades of corporate leadership changes, and government classification, much of the hard copy data has been lost or is not readily retrievable. In addition, Rocketdyne, while still in operation, was purchased and sold several times by various parent corporations during the decades since the Apollo missions, which has led to the loss of much of the original documentation. Evidence of part swapping and the reallocation of components were also discovered while researching the available data, supported by the discovery that some of the components had multiple serial numbers. It is also worth noting that the atmosphere in general during the Apollo era was one of "hurry up and get it done" (Clarke, pers. comm.). Finally, some of the methods of serializing the components, such as the painted stencils, rarely survived burial on the ocean floor. Others, such as stamped or etched numbering on the metal surface, proved to be an ideal location for corrosion to form, altering the markings.

To decisively establish the provenance of each object, conservators looked for specific identifying marks, called *unit numbers*, which were placed on several of the major engine components by various means in different locations with no discernible standardization. These unit numbers were assigned upon the completion of each engine and are only four digits, with the first two digits ("20") being identical for each. Thus, the only means by which to identify a specific mission were the final two digits, making the task much more difficult. Fortunately, through several means including visual inspection, mold casting, and using ultraviolet light in conjunction with digital photography,

components from the Apollo 11, 12, 14, and 16 missions were positively identified. An example of a stenciled area revealed under ultraviolet light on a thrust chamber belonging to engine #5 from Apollo 11 is given in figure 4b.

## 6. MATERIALS IDENTIFICATION

Prior to the objects arriving in the lab, research was undertaken to develop a list of metals and other materials likely to be found on the F-1 engines. Several original Rocketdyne documents were obtained, including a familiarization manual and an illustrated parts breakdown, which proved to be invaluable resources containing detailed schematics and diagrams (Rocketdyne 1970, 1972). However, they did not provide a precise bill of materials. Prior published research in the book *Saturn V* by Alan Lawrie (2005) provided an additional means of materials identification through manufacturing records. Later on, this book would prove helpful in identifying on which specific Apollo mission each component flew, as well as offer further insight into the difficulty of mission identification.

As the project progressed, the need to more precisely determine the alloys used on the F-1 engines became critical for several reasons: to elaborate a suitable conservation plan based on the materials present; to understand the corrosion processes affecting the most fragile and unstable objects, particularly galvanic corrosion; and to identify possible harmful materials that may have caused health issues for the conservators, particularly asbestos and beryllium.

Analysis was performed in several phases to assist conservators in implementing a treatment plan specific to each type of material present, and to help clarify specific questions concerning corrosion products and more precisely undertake materials and metallurgical investigation as the project developed.

The following analytical program was carried out:

- X-ray fluorescence (XRF) was performed by Bruce Kaiser of Bruker Elemental, Tim Foecke and Adam Creuziger of the National Institute of Standards and Technology (NIST), and Gregory Dale Smith of the Indianapolis Museum of Art.
- Scanning electron microscopy with energy dispersive spectroscopy (SEM/EDS) was undertaken at NIST.
- Raman spectroscopy and Fourier transform infrared spectroscopy (FTIR) were performed by Greg Smith.
- X-ray diffraction was carried out by Joe Swider of McCrone Associates Inc.
- X-radiography was carried out by Chris Watters and Mike Alton at Newco Inc.
- Metallurgical investigation of the corroded turbine blades was performed by NIST (not reported here)

Detailed results and discussion of the analytical results will be presented elsewhere. The following summary briefly describes analyses and results that were helpful to the conservators during treatment of the engines.

#### 6.1 MATERIALS AND METHODS OF MATERIALS IDENTIFICATION

Samples for analyses were removed mechanically from discreet areas on specific objects or were obtained from loose debris not attached to, but associated with, the particular object.

The initial XRF survey was performed in situ on 73 discreet areas on the surface of 12 objects using a Bruker Tracer III SD handheld XRF. Nine samples were obtained and further analyzed with XRF by NIST, using a portable XRF analyzer (model Olympic DS-4000-CC). Six samples were analyzed by Greg Smith using a Bruker Tracer III-V handheld XRF with rhodium tube, silicon-pin detector, and polymer window ( $-3 \times 5$  mm oval spot).

In tandem with the XRF analysis undertaken by NIST, six samples were obtained from four objects for more detailed compositional analysis using SEM/EDS. Three of these samples were analyzed by XRF, and three were measured with compositional mapping to show the spatial distribution of elements. Two different scanning electron microscopes (SEMs) with energy dispersive spectroscopy (EDS) detectors were used to analyze the samples. The first SEM was a JEOL 6400 with an Evex SiLi EDS detector and Revolution analysis software. Beam energy of 20 keV was used, and the current was chosen such that the detector dead time was between 20% and 35%. Energy spectra were recorded over a section of the sample magnified at 200X (approximately 500  $\mu$ m  $\times$  400  $\mu$ m, or 0.2 mm²).

The second SEM system used was a JEOL 7100 with an 80 mm<sup>2</sup> X-max<sup>N</sup> SDD detector with Oxford AZtec analysis software. Beam energy of 20 keV was used, and the current was chosen such that the detector deadtime was approximately 50%.

Six samples from four objects were characterized by Raman spectroscopy for phase identification, including copper corrosion products from one of the injector assemblies. Raman spectra were acquired using a Bruker Senterra microspectrometer on a Z-axis gantry. The spectrometer utilizes three selectable excitation lasers (532, 633, and 785 nm), an AndorPeltier-cooled CCD detector, and a 50-mm confocal pinhole. Laser power at the sample was generally below 3 mW. The spectra are the result of 10-second integrations with 20 to 30 coadditions. A 50X ultra-long working distance objective was used to focus on select particles. The analysis spot size was on the order of 1 mm, and the spectral resolution was in the range of 9 to 18 cm<sup>-1</sup>. OPUS software allowed for automated cosmic spike removal, peak shape correction, and spectral calibration.

Six samples were analyzed using FTIR spectroscopy using a SpectraTech Smart Orbit diamond ATR attachment coupled to a Nicolet 6700 spectrometer with a mid-IR DTGS detector. The instrument was purged with dry, CO<sub>2</sub>-free air. The spectra are the sum of 64 coadditions at 4 cm<sup>-1</sup> spectral resolution. Sample identification was performed using the Infrared and Raman Users Group (IRUG) reference spectral library.

Eight corrosion samples from six objects were obtained for phase identification by x-ray diffraction. The XRD instrument is a Rigaku RAPID x-ray diffraction system operated at 40 kV and 20 mA. Particles are typically run for 15 minutes and processed using Rigaku imaging software and MDI JADE software for data analysis. The resulting patterns were compared to references in the International Centre for Diffraction Data (ICDD) database of more than 300,000 references.

# 6.1.1 Results and Discussion of Materials Analysis

The XRF and EDS analyses revealed that the common materials present were alloys of nickel, aluminum (cast and wrought), and iron (various grades of stainless steel), as well as copper, brass, carbon steel, chrome and nickel plating, and other materials like polyurethane foam. The common nickel-chromium-ferrous alloys contain major quantities of nickel and chromium, and they could most readily be differentiated by the presence of alloying elements molybdenum, tungsten, niobium, and/or titanium (Creuziger and Foecke 2014). However, the often small or trace amounts of certain elements detected with XRF and EDS made precise identification difficult. The nickel-based alloys are most likely Hastelloy C-276, Inconel alloy X-750, Inconel 625, Inconel 718, Haynes 556, Rene 41, and Incoloy A-286. The results likely characterize a 6xxx wrought aluminum alloy and possibly a 6061 alloy. Investigation via compositional mapping further suggested that a 40x.x or 41x.x aluminum alloy was present, specifically on the turbopumps. The stainless steel alloys identified are made of 3xx series stainless steel based on the major iron-chromium-nickel signals from XRF and EDS. The trace amounts of niobium and titanium make distinguishing between 3xx series alloys difficult.

Of the samples analyzed, many deviated from the known composition of that alloy, raising the possibility that a non-standard alloy was used. This may help explain why some of the objects, such as the turbine blades, presented advanced levels of deterioration.

The Raman and FTIR spectra results identified solid matches for paratacamite on one of the fuel injectors, a corrosion mineral suspected from visual examination of all three injectors. This result was supported by the results of XRD analysis, which identified clinoatacamite, atacamite, and paratacamite, and copper chloride hydroxide as major, minor, and trace phases, in addition to cuprite and malachite. The presence of this problematic chloride-containing mineral species directly influenced the treatment protocol chosen for the three fuel injectors described further in section 7.4.

#### 6.2 X-RADIOGRAPHY

Seven turbine blades were imaged with radiography to determine the extent of corrosion that was visible on the blades, and noted by their loss of weight. A selection of four extremely corroded and lightweight turbine blades, two from stage 1 and two from stage 2, were imaged in the first group. In a subsequent group, three additional blades were imaged to compare results to group 1. The second set of blades were heavier in weight than the first, and it was presumed that this meant that the blades were in better condition with more metal remaining. The blades were imaged by Newco Inc. with a General Electric DXR250w Direct Radiography panel at 130 kV and 1.0 mA for 3.2 seconds.

The images from both groups confirmed the severe corrosion, most of which was not visible to the eye, with a more acute result noted in group 1 blades (not pictured here) that appeared to have lost the greatest density, judging by their weight. Images from group 2 revealed that these blades were also extremely corroded, although possibly the corrosion was not quite as advanced as that seen on the first set of blades, supported by the heavier weight of these blades (fig. 7). The x-ray images reveal that

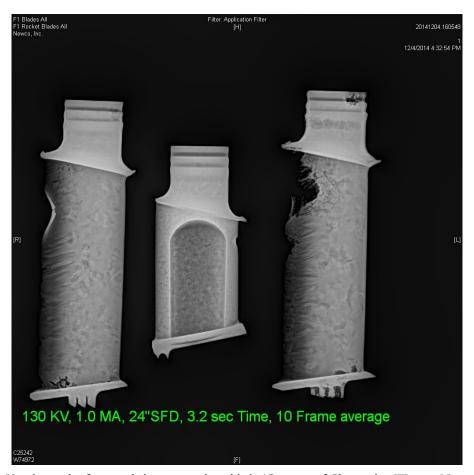


Fig. 7. X-radiograph of a corroded stage-2 turbine blade (Courtesy of Christopher Watters, Newco Inc.)

corrosion had penetrated the blade surface from small pits, most probably initiated by the stagnant conditions on the seafloor. This information proved valuable when a metallographic investigation was undertaken on samples of the turbine blades, and the full extent of the corrosion was revealed. It was also critical in deciding that consolidation of all detached turbine blades was needed to provide structural stability.

#### 7. TREATMENT

The engine components were treated in several phases, following the strategy previously outlined. These stages often overlapped, and the progress of the treatment was far from linear. Many of the most problematic objects went through multiple phases of the same treatment stage, as issues arose concerning access to all parts of the object.

# 7.1 MOVING THE OBJECTS

One of the biggest obstacles we faced during treatment was moving heavy, fragile, wet, and unstable objects weighing up to 3,000 lb. (1.3 tons). Moving the objects in and out of the treatment tanks, changing their position, or temporarily bracing, raising, or suspending them for examination or treatment had to be done in a careful and methodical manner to ensure safety for both the object and the team. Each move was thoroughly researched and planned by the team to ensure that the center of gravity was correctly located to accurately balance the object during moving. The combined experience of the team, encompassing engineering and technical knowledge, was invaluable at this time. Use of an overhead 5-ton gantry mobile along with a forklift and several pallet jacks allowed ease of movement. Lifting required innovative and customized modifications to allow the engines themselves to be picked up or maneuvered. Several methods were used for moving the engines. Where possible, engines were rigged with synthetic slings and lifted by their strongest component. Many of the heaviest parts were rigged and lifted with reinforced plastic pallets attached to evenly distribute the weight and avoid point load as much as possible. For some objects, the original rigging points were preserved, and, where feasible, these were fitted with new, custom lifting devices, spreader bars, connecting devices, and adaptors from which the part could then be rigged and safely lifted (fig. 8).

# 7.2 INITIAL TREATMENT: REMOVAL OF SEDIMENT AND IRON STAINING

Most of the artifacts were caked with very compacted grey sediment and massive corrosion products, in some cases totally obscuring the surface. Some of the worst-affected objects were the aluminum turbopumps (see fig. 3b) and the stainless steel fuel manifolds. The electric components and wire bundles still attached to some of the thrust chambers were held in place, embedded in hard sediment and iron corrosion products.

Complete removal of all sediment was necessary to enable access to the metal surface for stabilization (particularly inside the engines), to fully reveal surface details, and to improve the aesthetic appearance of the metal. The sediment, often mixed in with gelatinous corrosion products, was present in every possible cavity (even the tiniest cavities), inside the numerous instrumentation control lines, and within the cooling tubes that line the interior of the thrust chambers. The sediment was extremely stubborn, did not easily dissolve in water or other liquids, and had to be manually removed by a combination of hand cleaning and pressure washing. Multiple campaigns of cleaning were necessary, as most of the objects had extremely complex geometry that impeded access. As all sides of the objects

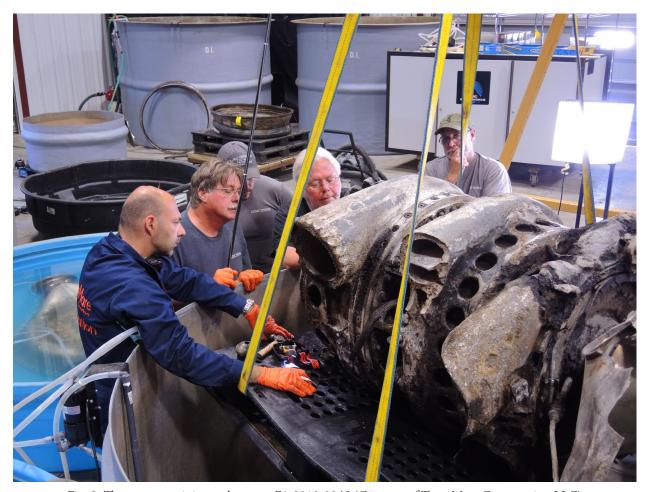


Fig. 8. The team examining turbopump F1-2013-0045 (Courtesy of Terra Mare Conservation LLC)

were gradually accessed, new pockets of sediment were revealed, which allowed for more in-depth cleaning (fig. 9).

Many of the objects were heavily stained with iron corrosion products from steel components that had preferentially corroded or completely disintegrated, causing widespread rust-colored stains. In some cases, the stains gave us valuable information about the steel components that had disappeared and clues about the position of the objects in the burial sediment. The objects worst affected by stains were the thrust chambers, the LOX domes, and the heat exchangers. The steel outriggers, originally welded to the exterior of the thrust chambers, and steel gimbals, the original attachment point of each engine to the rocket, were the cause of most, if not all, of the staining. None of the outriggers were recovered, and only one partial gimbal survived still attached to its LOX dome. The gimbal was identified as belonging to Apollo 11 and is shown in figure 10 before and after treatment.

The most severe rust stains were reduced or eliminated by immersion of the objects in recirculated deionized water with a 0.1% solution of FlashCorr, an anionic surfactant and multi-metal corrosion inhibitor made by Cortec Corporation. It was also observed that the chloride extraction process using this family of surface-active molecules was enhanced compared to that of deionized water, particularly on aluminum alloys. This was assessed by measuring the chloride levels of the treatment tanks on a weekly basis. This may be due to the ability of this type of anionic compound to displace adsorbed chloride ions from the metal (Monticelli et al. 1991; Malik 2011; Balbo et al. 2013). This treatment was



Fig. 9. Conservator Claudia Chemello working on heat exchanger F1-2013-0046 from Apollo 16 (Courtesy of Terra Mare Conservation LLC)

combined with periodic pressure washing and manual brushing of the surface. The treatment time varied depending on the extent of the staining and took up to two months for certain objects.

After removal of sediment and surface staining, hard, white deposits were still visible on certain parts of the engines. These were assumed to be calcium carbonate from the burial environment. These deposits were tenacious, generally unaffected by the chelating properties of FlashCorr, and were reduced or eliminated with dry ice cleaning after stabilization.

# 7.3 PRESERVING EPHEMERAL INFORMATION

The firing of the engines left numerous traces and marks on the surface of the objects. Preserving evidence of use was critical and one of the most important goals of the archaeological approach adopted for treatment. Indications of engine use included layers of soot from the burning of the propellants and bluing of the metal due to extreme heat. Examples of ephemeral traces include partial stenciled numbers that were identified on some of the thrust chambers using visible or ultraviolet light, adhesive remains that had secured a data plate to a thrust chambers with legible numbers visible in the adhesive, and a paper identification label adhered to a hypergol manifold. All treatments involving mechanical action avoided areas where ephemeral traces remained on the surface. In some instances, areas were specifically protected with physical barriers during activities such as chemical cleaning, pressure washing, or dry ice cleaning. For chemical treatments, the painted stencils were protected with cyclododecane during treatment.





Fig. 10a. Lead Technician Jerrad Alexander (left) and Conservator Paul Mardikian (right) removing the steel gimbal from Apollo 11's LOX dome F1-2013-0005-1 for conservation; 10b. The same LOX dome and gimbal after conservation (Courtesy of Terra Mare Conservation LLC)

## 7.4 CHEMICAL STABILIZATION

After several decades on the bottom of the Atlantic Ocean, salts (or more specifically, the chloride ions from these salts) had caused severe corrosion problems on some of the objects. Chloride ions permeate metals during burial and can cause active corrosion after recovery. The main goal of the stabilization phase was to ensure that entrapped chloride ions were reduced to the lowest achievable level to enhance long-term stability.

The chloride release from all objects was tracked from the beginning of the project, from the time rinsing first began in the basins. Chloride detection was carried out with various methods according to the stage of the project: QuanTab test strips for chloride, titration using the argentometric method (Rice 2015), and a chloride-specific electrode. Following the removal of sediment and staining, it was clear that a more interventive approach was necessary to further reduce chloride levels in certain objects. A stabilization strategy was designed for each type of object based on its materials and degree of corrosion. Some objects, such as the Inconel thrust chambers and turbine assemblies, required less stabilization, as the chloride levels had already been drastically reduced in successive baths of deionized water and a short immersion in FlashCorr for rust stain removal. The presence of stencils on the thrust chambers also meant that the use of more aggressive chemicals was too risky.

Some artifacts were extremely unstable and required a custom stabilization treatment, including the turbopumps and the fuel injectors. The turbopumps were constructed from at least 10 different alloys. One of the turbopumps was relatively intact, whereas the other had suffered extensive corrosion with major loss to its outer aluminum casing, as shown in figure 11. The presence of a nickel-plated steel

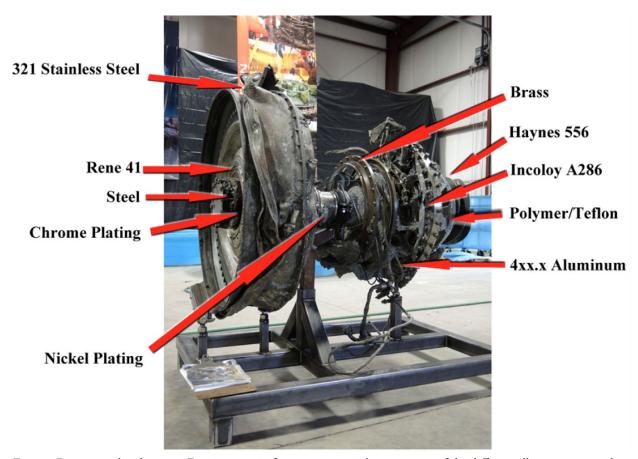


Fig. 11. Deteriorated turbopump F1-2013-0012 after conservation showing some of the different alloys present on this object (Courtesy of Terra Mare Conservation LLC)

drive shaft, steel bearings, and a deteriorated aluminum casing, in addition to numerous other metals in contact with each other, had created extremely complex corrosion issues. Dismantling of the turbopumps was briefly considered but deemed too unsafe for the objects. The high precision with which these objects were assembled and the difficulty of achieving a precise and accurate reassembly meant that certain objects could not be taken apart.

Active corrosion was visible on the drive shaft, particularly where the nickel plating was missing, and on the steel bearings. Stabilizing the steel components of the turbopumps was the main focus of the treatment. The treatment was complicated further because the treatment solution had to be alkaline enough to stabilize the steel without corroding the aluminum, which is highly susceptible to corrosion in an alkaline environment.

Stabilization was achieved using a 0.5% solution of sodium metasilicate in deionized water, a corrosion inhibitor used in industry for inhibition of aluminum, and successfully employed for the treatment of artifacts made from aluminum and steel recovered from freshwater sites (Degrigny 1995). The object was placed under electrolysis to facilitate chloride extraction at a cathodic potential of -1 V versus Ag/AgCl<sub>2</sub> reference electrode (-0.8 V vs. standard hydrogen electrode). Chloride extraction was tracked on a weekly basis and required six months to achieve a consistent chloride reading under 2.5 ppm in solution. The solution was then renewed and the object was maintained in treatment for an additional two months. During treatment, more detailed cleaning occurred, particularly to the heavily corroded shaft bearings. Conserved turbopumps are shown in figures 11 and 12.



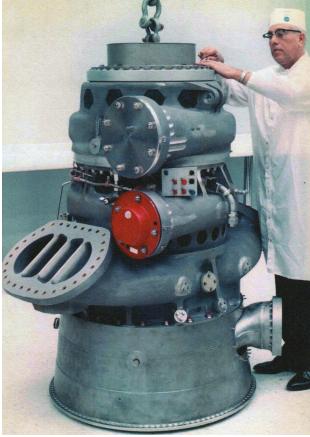


Fig. 12a. Turbopump F1-2013-0045 after conservation (Courtesy Terra Mare Conservation LLC); 12b. Original photo showing a similar turbopump on the assembly line (Courtesy Rocketdyne Harold C. Hall Collection)

The three fuel injectors, made primarily from copper alloy and 3xx series stainless steel, most likely 304, were found in their original position, sandwiched between an Inconel LOX dome and Inconel thrust chamber. The copper alloy side of the injector was heavily corroded, with bright green corrosion covering the surface, almost certainly due to severe galvanic corrosion. The stainless steel side was also heavily corroded and pitted. The choice of stabilization treatment had to be compatible with both materials, as the object was constructed as one unit and could not be dismantled.

The fuel injectors were initially stabilized in a recirculated solution of 1% sodium sesquicarbonate in deionized water to begin chloride extraction, whereas analysis with Raman spectroscopy and x-ray diffraction was undertaken to determine what corrosion products were present. The copper chloride species atacamite and paratacamite were identified, confirming the need for a more interventive treatment to arrest corrosion and stabilize the objects. This was accomplished using a modified version of alkaline Rochelle salts, a treatment familiar to conservators treating archaeological copper alloys. A 0.1M solution (3.9 g/L of sodium hydroxide and 28.2 g/L of sodium potassium tartrate) in 200 L of deionized water was used, and the chloride and copper levels were tracked in the treatment solution. Once the green corrosion products were reduced, the fuel injectors were thoroughly rinsed with a pressure washer, mechanically cleaned to remove corrosion inside the fuel holes, and returned to a 1% solution of sodium sesquicarbonate for final chloride removal. When the chloride levels remained consistently under 2.5 ppm for two months, the rinsing process began. Fuel injector F1-2013-0005-2 is shown before and after conservation in figure 13.

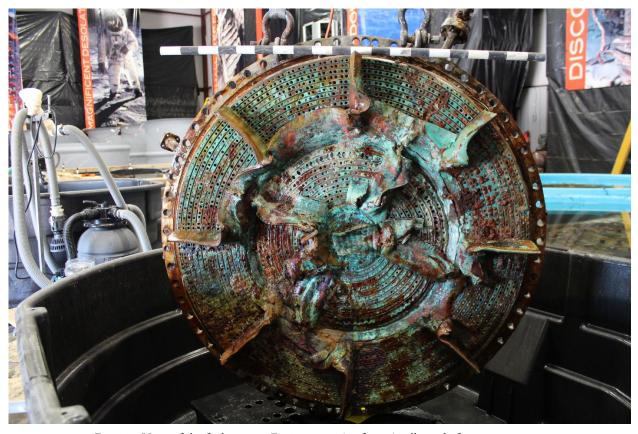


Fig. 13a. View of the fuel injector F1-2013-0005-2 from Apollo 11 before conservation



 $Fig.\ 13b.\ The\ fuel\ injector\ after\ conservation\ (Courtesy\ of\ Terra\ Mare\ Conservation\ LLC)$ 

## 7.5 RINSING

After chemical stabilization was complete, an extended period of rinsing, often lasting several months, commenced to remove traces of the chemicals used during stabilization. Rinsing was carried out with recirculated deionized water at a temperature of approximately 40°C. The pH and conductivity of the water was monitored until it reached that of the deionized water. The turbopumps treated with sodium metasilicate were rinsed under cathodic protection to avoid flash rusting of the steel components.

## 7.6 CORROSION INHIBITION

The complex materials of the engines, particularly their intricate internal geometry, prompted a decision to apply a corrosion inhibitor to the most problematic and unstable objects in the collection, namely the fuel injectors and the turbopumps. These objects had been through an extensive treatment regime for active corrosion and had numerous and complex internal cavities, posing a risk for long-term stability. However, the long-term environmental conditions that will prevail for these objects are unknown at the time of writing.

Several tests were undertaken to assess VpCI-377 and VpCI-316, vapor phase corrosion inhibitors made by Cortec Corporation, designed for indoor protection. Both are water-based multimetal corrosion inhibitors. Tests were performed on metal coupons at 2.5%, 5%, and 10% concentrations in deionized water, assessing the inhibitors' visual appearance, tackiness, viscosity, and corrosion protection. Inhibitor 377 at 2.5% proved to be a better choice, was easy to apply by dipping, and was almost invisible. However, the long-term behavior and protectiveness of vapor phase corrosion inhibitors is not totally understood on cultural heritage materials, and more research and evaluation are needed. It is our hope to be able to follow their long-term effect on this collection.

# 7.7 FINAL SURFACE FINISHING

Final surface cleaning was performed using dry ice blasting with a KG30 machine supplied by Continental Carbonic (fig. 14). This method was far superior at removing unwanted surface accretions and hard calcium deposits that simply could not be removed by any other method. Overall surface cleaning of the majority of the objects was accomplished using 35 to 45 pounds per hour (pph) of dry ice at a pressure of 25 to 50 psi with a medium-size splitter and a 2 in. wide nozzle attachment. This broad cleaning allowed for the identification of those areas in need of more aggressive treatment. For these areas, the wide nozzle was replaced with the 7-mm "standard" nozzle. This somewhat shorter, smaller attachment gave a greater degree of access to areas of the object that were difficult to reach, as well as a much more targeted cleaning. Machine settings for areas on objects requiring the removal of denser accretions and hard deposits were between 45 and 55 pph and 50 and 75 psi. Areas with the thickest encrustations of what appeared to be calcium deposits required the most aggressive settings, 60 to 65 pph and 75 to 100 psi, using the smallest (5-mm) nozzle. Working distance and dwell time were adjusted according to what was necessary to achieve the desired cleaning level at each phase (fig.14). A view of the lab at the end of the project is shown in figure 15.

#### 7.8 ARTIFACT DISPOSITION

Several museums expressed interest in acquiring objects from this collection, in agreement with NASA, who had oversight for the objects' disposition. These institutions included the Smithsonian National Air and Space Museum, who will receive the Apollo 11 objects; the Museum of Flight in Seattle, Washington; the US Space and Rocket Center in Huntsville, Alabama; the Kansas Cosmosphere and Space Center; and the Space Museum in Bonne Terre, Missouri. It is unclear whether the objects will be placed immediately on display; however, we believe that the intention of the receiving institutions is to



Fig. 14. Conservator Claudia Chemello cleans turbopump F1-2013-0012 with dry ice (Courtesy of Terra Mare Conservation LLC)

organize new exhibits around the engine components. The remainder of the collection is currently housed at the Kansas Cosmosphere and Space Center in the climate-controlled lab where they were treated but are not accessible to the public.

# 8. CONCLUSIONS

The conservation of Apollo-era F-1 engines provided an extraordinary opportunity to conserve objects made of materials that are rarely encountered in the conservation community. To add to the challenge of conserving a group of objects of such significance, the engines had been buried in the ocean for more than four decades and exhibited extreme levels of corrosion and alteration. Treatment was accomplished by taking a deliberate, archaeological object—based approach for these modern archaeological artifacts. Given the importance and irreplaceable nature of these objects, this approach has proved to be successful. The project was completed in two and a half years, a tour de force in the world of large-scale maritime conservation projects. The key steps to its success include the collaborative structure of the project, bridging the world of conservation, aerospace engineering, fabrication, restoration, and materials science together; advanced planning and logistical support; and a holistic approach to preserving the context and history behind these objects as much as the materials themselves.



Fig. 15. General view of the lab with some of the artifacts after conservation (Courtesy of Terra Mare Conservation LLC)

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# **REFERENCES**

Balbo, A., A. Frignani, V. Grassi, and F. Zucchi. 2013. Corrosion inhibition by anionic surfactants of AA2198 Li-containing aluminum alloy in chloride solutions. *Corrosion Science* 73: 80–88.

Bezos, J. 2014. F-1 Engine Recovery. Bezos Expeditions. March 16. <a href="http://www.bezosexpeditions.com/updates.html">http://www.bezosexpeditions.com/updates.html</a>

Capone, V. 2013. How do you recover an Apollo rocket engine from 3 miles beneath the Bermuda Triangle? Science and Adventure with Vince Capone. August 16. <a href="http://www.blacklaserlearning.com/adventure/how-do-you-recover-an-apollo-rocket-engine-from-over-2-miles-beneath-the-bermuda-triangle/">http://www.blacklaserlearning.com/adventure/how-do-you-recover-an-apollo-rocket-engine-from-over-2-miles-beneath-the-bermuda-triangle/</a>

——2014. Apollo F-1 engine returns to Cape Canaveral. Science and Adventure with Vince Capone. March 14. <a href="http://www.blacklaserlearning.com/adventure/apollo-f-1-engine-returns-to-cape-canaveral/">http://www.blacklaserlearning.com/adventure/apollo-f-1-engine-returns-to-cape-canaveral/</a>

Clarke, L. October 11, 2013. Personal communication by former Rocketdyne engineer.

Creuziger, A., and T. Foecke. 2014. Compositional analysis of various metal samples recovered from Apollo F-1 engine to aid in conservation efforts, preliminary report. Unpublished manuscript, National Institute of Standards and Technology, Gaithersburg, MD.

Degrigny, C. 1995. Stabilisation de moteurs d'avions immerges. Studies in Conservation 40 (1): 10-18.

Lawrie, A. 2005. Saturn V: The complete manufacturing and test records: Plus supplemental material. 2nd ed. Burlington, Ontario: Apogee.

Malik, M. A., M. A. Hashim, F. Nabi, S. A. Al-Thabaiti, and Z. Khan. 2011. Anti-corrosion ability of surfactants: A review. *International Journal of Electrochemical Science* 6: 1927–1948.

Monticelli, C., G. Brunoro, A. Frignani, and F. Zucchi. 1991. Surface-active substances as inhibitors of localized corrosion of the aluminium alloy AA 6351. *Corrosion Science* 32 (7): 693–705.

Reed, Roger C. 2006. The superalloys. Fundamentals and applications. New York: Cambridge University Press.

Rice, E., R. B. Baird, A. D. Eaton, and L.S. Clesceri. 2015. *Standard methods for the examination of water & wastewater*. 22nd ed. Washington, DC: American Public Health Association.

Rocketdyne. 1970. *R-2896-1 F-1 engine familiarization and training manual*. Roswell, GA: Rocket Science Institute.

——1972. R-3896-4 F-1 engine illustrated parts breakdown. Roswell, GA: Rocket Science Institute.

Young, A. 2009. The Saturn VF-1 engine: Powering Apollo into history. Berlin: Praxis Publishing Ltd.

## **FURTHER READING**

Cayless, R. B. C. 1990. Alloy and temper designation systems for aluminum and aluminum alloys. In *ASM handbook volume 2, Properties and selection: Nonferrous alloys and special-purpose materials*. 10th ed. Novelty, OH: ASM International.

Davis, J. R. (ed). 1993. ASM specialty handbook: Aluminum and aluminum alloys. Novelty, OH: ASM International.

Mankins, W. L., and S. Lamb. 1990. Nickel and nickel alloys. In *ASM handbook volume 2, Properties and selection: Nonferrous alloys and special-purpose materials.* 10th ed. Novelty, OH: ASM International.

Rodgers, K. A. 1993. Routine identification of aluminium hydroxide polymorphs with the laser Raman microprobe. *Clay Minerals* 40 (28): 85–99.

Special Metals Inc. 2004. Inconel 706 alloy specification sheet. Accessed March 20, 2016. <a href="http://www.specialmetals.com/documents/Inconel%20alloy%20706.pdf">http://www.specialmetals.com/documents/Inconel%20alloy%20706.pdf</a>

Washko, S. D., and G. Aggen. 1990. Wrought stainless steels. In *ASM handbook volume 1: Properties and selection: Irons, steels, and high-performance alloys.* 10th ed. Novelty, OH: ASM International.

# **SOURCES OF MATERIALS**

Sodium Metasilicate (Cat. No. 191382) and Sodium Potassium Tartrate Tetrahydrate (Cat. No. 150156) MP Biomedical LLC 29525 Fountain Parkway Solon, OH 44139 800-854-0530

FlashCorr, VpCI-377, and VpCI-316 Packnet Limited 2950 Lexington Ave. S Eagan, MN 55122 952-944-9124

Double Junction Reference Electrode Thermo Scientific 166 Cummings Center Beverly, MA 01915 978-232-6000

PAUL MARDIKIAN is co-founder and Senior Conservator of Terra Mare Conservation LLC, a firm specializing in the conservation of archaeological, industrial, fine art, and architectural objects. He has an undergraduate and graduate degree in art history and archaeology from the School of the Louvre and a graduate degree in conservation from Sorbonne University, Paris. He is a Fellow member of the AIC and Assistant Coordinator for the ICOM-CC Metal Working Group. Address: 14 Marbel Ln., Terra Mare Conservation LLC, Charleston, SC. E-mail: paul@terramareconservation.com

CLAUDIA CHEMELLO is co-founder and Senior Conservator of Terra Mare Conservation LLC, a firm specializing in the conservation of archaeological, industrial, fine art, and architectural objects. Prior to working in private practice, she was Senior Conservator at the Kelsey Museum of Archaeology at the University of Michigan and a Conservator for the Agora Excavations, American School of Classical Studies at Athens. Claudia has an undergraduate degree in archaeology and

Egyptology from Macquarie University, Sydney, and a graduate degree in conservation from the University of Western Sydney, Australia. She is a Fellow of the AIC and a Coordinator for the ICOM-CC Metal Working Group. Address: as for Mardikian. E-mail: <a href="mailto:claudia@terramareconservation.com">claudia@terramareconservation.com</a>

JERRAD ALEXANDER is a graduate student at the University of Melbourne in the Master of Cultural Materials Conservation program. He was the Lead Conservation Technician on the Apollo F-1 Engine Conservation project for the Kansas Cosmosphere and Space Center. He served as a United States Air Force Staff Sergeant, Aircraft Weapons Systems Specialist, and Flight Engineer. He earned an associate's degree in aircraft systems technologies from the Community College of the Air Force and a BA in organizational management from Ashford University. His training and experience focus on the care of aerospace and military museum collections. E-mail: <a href="mailto:jalexanderconsult@gmail.com">jalexanderconsult@gmail.com</a>