

# HYDRAZINE CATALYST PRODUCTION – SUSTAINING S-405 TECHNOLOGY

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## ABSTRACT

The development of the iridium-based Shell 405 catalyst for spontaneous decomposition of hydrazine was one of the key enabling technologies for today's spacecraft and launch vehicles. To ensure that this crucial technology was not lost when Shell elected to exit the business, Aerojet, supported by NASA, has developed a dedicated catalyst production facility that will supply catalyst for future spacecraft and launch vehicle requirements.

We have undertaken a program to transfer catalyst production from Shell Chemical USA (Houston, TX) to Aerojet's Redmond, WA location. This technology transition was aided by Aerojet's 30 years of catalyst manufacturing experience and NASA diligence and support in sustaining essential technologies. The facility has produced and tested S-405 catalyst to existing Shell 405 specifications and standards.

Our presentation will describe the technology transition effort including development of the manufacturing facility, capture of the manufacturing process, test equipment validation, initial batch build and final testing.

## INTRODUCTION

In the early 1960's, Shell Chemical Company and NASA-JPL originally teamed up to develop a catalyst for the spontaneous ignition of hydrazine. This effort culminated in the development of Shell 405 catalyst in 1964<sup>1</sup>. The invention was awarded patent 4,124,538 in 1978. The Shell Chemical Company conducted catalyst manufacturing initially at their Emeryville, CA laboratory. This production was moved to the Westhollow Technology Center (WTC) in Houston, Texas in the early 1980's and has now been moved to

Aerojet in Redmond, Washington. Current applications include monopropellant thrusters for NASA, USAF and commercial spacecraft, and auxiliary power generation for the US Space Shuttle.

The granular catalyst material is extraordinarily active for the decomposition of hydrazine and reacts in milliseconds at bed temperatures as low as 0°C. This ability to rapidly and reliably ignite hydrazine has led to widespread use of the catalyst in thrusters for control of spacecraft and launch vehicles. The catalyst is unique among heterogeneous catalysts in several respects. Shell 405 has an extremely high metal loading of 31 – 33 % (wt) iridium. In contrast, most commercial catalysts have 0.5 – 5% active metal loading. Also, Shell 405 is designed to be used at the highest possible reaction temperature – up to 1150°C – in order to maintain high thruster performance. Conventional catalysts are usually employed to reduce reaction temperatures and are usually limited to 700°C or less. Follow-on efforts sponsored by USAF-AFRPL looked at using the catalyst for higher performance hydrazine propellants<sup>2</sup> and increasing the catalyst life<sup>3</sup> and in fact Shell 405 is currently used in the development of reduced toxicity, or "green" monopropellants.

In response to changes in its business focus, Shell decided to exit the Shell 405 catalyst manufacturing business and entered into negotiations with Aerojet. Ultimately, with the support of NASA, a manufacturing license was assigned to Aerojet to provide S-405 catalyst for

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\* The licensing agreements do not include the use of the "Shell" name. Hence catalyst produced by Aerojet in Redmond, WA is designated "S-405". References to "Shell 405" in this document identify

the spacecraft and launch vehicle applications. The agreements were concluded in March of 2002. Transition of the manufacturing process from Houston to the Aerojet facility at Redmond, WA was begun immediately. The purpose of this report is to document the transition of the manufacturing process and to demonstrate that Shell 405 and S-405 spontaneous catalyst are equivalent by providing details of the transition process, explanations of minor differences in the test apparatus and process, and details of the results.

### **TRANSITION PROCESS**

To ensure that the Redmond-produced S-405 product was identical to the Houston-produced Shell 405, we identified a transition strategy that focussed on closely reproducing the Houston production and test facility in Redmond. Since the actual equipment used in Houston was not available for installation in Redmond, we procured or fabricated new equipment to match the equipment in Houston.

Along with the process equipment, we planned to produce catalyst according to the identical procedures and processes that were in use by Shell personnel at Houston. This extended beyond simple adoption of the Shell process paperwork. Hands-on training by Houston personnel was planned in order to ensure that we understood how the written processing documents were actually carried out.

Test equipment and test procedures were also developed to match the Houston facility. Test equipment verification was also performed where Houston-produced Shell 405 catalyst was tested on Redmond equipment with Redmond test procedures. The corroboration of test results from the two sets of measurements served to verify the new test equipment and methods. Luckily, prior collaboration between Aerojet and Shell had already led to significant alignment of laboratory test methods between the two sites.

The goal of the transition process was to produce a batch of S-405 catalyst in Redmond and show that it was identical to catalyst produced in Houston. This initial 3-kg (6.6 lb) batch of catalyst was manufactured and tested under full production controls so that it is "flight quality". This material is ready for further use by NASA for testing or flight application.

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catalyst which was produced by Shell Chemical Company at either Emeryville, CA or Houston, TX.

The transition process thus consisted of five areas:

- Procurement of Raw Materials and Equipment
- Process Documentation and Training
- Initial Production
- Test Equipment Fabrication and Verification
- Testing of Initial Production

### **PROCUREMENT OF RAW MATERIALS AND EQUIPMENT**

#### **Raw Material**

The general approach was to duplicate as closely as possible the materials and process equipment used by Shell in Houston. There are two major raw material components in S-405 catalyst – carrier and active material. Seven drums of carrier were provided by Shell. This consisted of three drums (net 360lb each) of nuggets and four drums (ca. 1000lb) of "in process" material that had undergone preliminary grinding and screening.

The other major raw material is an iridium salt which was purchased from Johnson-Matthey. Both Shell and Aerojet have consistently used this raw material from this vendor over many years of catalyst processing.

Other chemicals used in catalyst processing were purchased from standard laboratory supply sources. Generally ACS Reagent or USP grade materials were used which reflected the practice at Shell.

#### **Process Equipment**

New equipment was purchased for this program. Effort was made to purchase equipment identical to or very similar to the equipment used by Shell in Houston. Equipment used in the early stages of processing was generally commercially available. The equipment used in the latter soak/dry processing and the reduction processing had been custom fabricated by Shell. In this situation Aerojet purchased components and fabricated custom equipment to parallel the Shell Houston equipment.

Carrier Preparation Equipment. Carrier grinding is the initial step in making S-405 catalyst. Further processing yields the rounded, smooth, tough granules needed for catalyst preparation and eliminates the undesirable weak or defective granules. All of the carrier processing equipment was available for purchase and is very similar to the equipment originally used by Shell.

Catalyst Soak/Dry Processing Equipment. Once the carrier is prepared, the catalyst is fabricated by repeated soak/dry cycles. In this process it is critical that the active metal salt be well dispersed and anchored to the carrier before the next coating of metal salt is applied. The drying time and temperature is key to this dispersion and anchoring. Shell used custom equipment for the soak/dry process.

Sample equipment was provided by Shell and served as a model for preparing identical fixtures. The drying system was assembled using the same model of heater used at Houston.

After assembling the equipment and adjusting the processing parameters we achieved process temperatures and times identical to those observed at Shell. The weight changes for in-process material also followed the pattern seen for material processed by Shell in Houston.

Catalyst Reduction and Oxidation. The final processing step entails activating the catalyst by reduction. Precise process temperatures must be maintained to achieve the best catalyst activity. Shell WTC used a customized furnace for this processing. Aerojet was able to procure the same furnace from the same vendor according to the original Shell specifications. For our installation, we elected to use electronic mass flow controllers and solenoid regulated valves for control of process gases.

The last step in catalyst processing involves careful air passivation of the highly active, reduced material. The equipment for this process is simple and is equivalent to the configuration used by Shell.

## **PROCESS DOCUMENTATION AND TRAINING**

### **Documents Provided by Shell.**

Shell Chemical provided both paper and electronic copies of all process documents. This documentation ranged from general policy to very specific detailed documents that described how to mix solutions or prepare catalyst. In addition, a video was provided which showed the final soak process, reduction processing and cold-start rocket engine testing. A complete set of these documents as well as copies of the video have been incorporated into the Aerojet document archives.

Not all of the documents provided by Shell are needed for the production of the currently used ABSG type catalysts. Also, some of the documents provided by Shell address topics such

as configuration control, quality data, calibration and records handling. Since Aerojet is an ISO 9001 and AS 9100A certified manufacturing facility and in addition is a FAA qualified repair station, we already have systems in place which cover these topics. Aerojet did not adopt these Shell documents.

### **Incorporation of Shell Documents into Aerojet System.**

Twenty documents provided by Shell were incorporated into the Aerojet's documentation control system. Raw material specifications were incorporated without revision. The final product specification was initially incorporated without revision. Subsequent test results however, indicated that one of the final acceptance parameters required revision. This revision is discussed later in the Rocket Engine Test Verification section.

Laboratory test procedures were incorporated without revision. The rocket engine test procedure has been revised and updated so that it describes the new test facility fabricated by Aerojet. For further information, please see Rocket Engine Test Equipment, later in this report.

Fabrication process documents were adopted as Aerojet "Process Instructions". These documents are general in nature, do not reference part numbers and have not been updated or reformatted. These Shell documents served as the basis for the Aerojet "Work Instruction" packets that are issued for each part number for each manufacturing lot. The Aerojet work instructions are part number specific.

### **Training.**

Originally training had been planned to take place at the Shell Houston facility. Unfortunately, the production facility was closed and all manufacturing equipment was gone before work could begin on this project. However, the Shell facility and processes had been extensively videotaped. EJ Wucherer visited the Shell facility briefly after it was dismantled and discussed the processing with the Shell personnel. In addition, Tim Cook of Aerojet was able to observe a rocket engine test series conducted at Shell in Houston just before that part of the facility was dismantled.

The revised training plan called for key Shell personnel to train Aerojet personnel at the Redmond facility and to observe initial production. Shell personnel were also very responsive in

answering telephone and e-mail requests for information.

The training program consisted of daily reviews of the Shell and Aerojet process instructions. Individual process steps were then identified for "dry runs" using non-production materials. The equipment was then cleaned. Any adjustments or "redlines" were noted on the production work instructions and finally the production material was processed. This close coupling of tutoring, training and production processing reinforced the learning of critical process techniques and enabled us to capture first hand the actual "as built" processing that the Shell personnel had been performing.

In all, Shell personnel spent four weeks at the Aerojet Redmond facility training and assisting with the initial catalyst production and testing.

### **INITIAL PRODUCTION**

The initial production run of 14/18 mesh catalyst produced 3 kg.

#### **Carrier Preparation.**

The carrier raw material requires extensive grinding and sieving. This helps eliminate weak or friable granules. To further improve the strength and crush resistance of the carrier, the granules are rounded to remove sharp edges and smooth the surface. Finally, the carrier is rinsed repeatedly, dried and sieved again.

#### **Active Metal Loading.**

The carrier is dried and prepared for active metal loading. The active metal, iridium, in the form of a salt is prepared in aqueous solution. The carrier is then impregnated with the active metal solution and the product dried to "fix" the metal on the carrier. This process is then repeated until the carrier has taken up the required amount of active metal.

In this process it is important that the active metal solution is properly prepared and conditioned. Likewise, the drying temperature and time must "fix" the active metal on the carrier or the subsequent soak cycle will re-dissolve the metal.

#### **Reduction.**

The impregnated carrier requires reduction of the oxidized metal salt to the active metallic state. This one-time process requires strict control of process temperatures and gas flows. The reduction furnace is fitted with a steel liner to help assure uniform heating. Only one-half of the 3 kg catalyst batch can be reduced during one run. Thus each

production run results in two "half batches" which are normally combined in the final air passivation step.

After initial set-up and training, the first "half batch" was loaded into the reduction furnace. The reduction heat ramp took much longer than expected – roughly three times longer. Once the final processing temperature had been achieved, the rest of the reduction and cool down proceeded as expected.

Examination of the gas flow and temperature data obtained from the first "half batch" indicated a contradiction between the written process instructions and the expected reaction times from the experience of the Shell personnel. This contradiction was resolved when it was discovered that the H<sub>2</sub> gas flow meter at Shell had been calibrated with N<sub>2</sub> gas while the H<sub>2</sub> gas flow controller at Aerojet had been calibrated with H<sub>2</sub> gas. The hydrogen gas flow system was adjusted to match Shell's calibration method and the second "half batch" of carrier processed. The observed heating and reaction rates were much faster and fit the expected pattern based on Shell/Houston experience.

After the two "half batch" reductions, normal processing would have been to re-combine the "half batches" for the air passivation step. However, since our processing of the first "half batch" had not been optimal (low H<sub>2</sub> flow), we elected to maintain and test the two "half batches" separately. This strategy proved unnecessary since both "half batches" have independently passed all acceptance test requirements.

After reduction the catalyst is extremely active and retains a substantial amount of absorbed hydrogen. Rapid air exposure could cause a fire and certainly could damage the catalyst. The last processing step therefore allows air to slowly reach the catalyst under a nitrogen blanket.

Based on the Shell identification method the designations 9-ATRE-403-A (a.k.a. "half batch A") and 9-ATRE-403-B (AKA "half batch B") were assigned to the two "half batches". (RE was utilized to designate production at Redmond WA).

### **TEST EQUIPMENT FABRICATION AND VERIFICATION**

Our approach to testing and test equipment was to closely match our new Redmond facilities to those in use by Shell at Houston. In addition we sought to "verify" the functioning of the test equipment by

testing catalyst produced (and initially tested) in Houston on the Redmond equipment.

#### **Laboratory Test Equipment.**

Custom-made test equipment was used by Shell in Houston for the crush and H<sub>2</sub> chemisorption tests. Fortunately, Aerojet already had custom designed crush and H<sub>2</sub> chemisorption test equipment based on previous technical collaborations with Shell. This meant that we simply had to adopt the Aerojet equipment to the exact test article dimensions and test methods that Shell used. The other major laboratory test is BET and pore volume. This testing was performed on commercially available test equipment – both at Shell and Aerojet.

The crush test consists of simply crushing a sieved, weighed amount of material in a piston that has been loaded to a specific weight. The post-crush material is recovered, re-sieved and re-weighed. The specification indicates the mass of material which must survive the crushing. The diameter and volume of the crush chamber are important characteristics of the test rig. The Aerojet test tool matches the dimensions of the Shell test rig.

BET and pore volume measurements are performed by dosing a sample of material with measured aliquots of gas at low temperature. Gas pressure above the sample is then measured. Repeated dosing and pressure measurement results in a pressure/volume curve which is characteristic of the test material. Analysis of this curve by the Braunauer, Emmett and Teller<sup>4</sup> (BET) algorithm results in a measure of the physical surface area of the test material. This data collection and evaluation is conducted using commercially available test equipment. Aerojet was able to purchase this test equipment from the same manufacturer as Shell. The equipment installed at Aerojet was calibrated and certified by the manufacturer using a NIST standard material for (BET) surface area.

The H<sub>2</sub> chemisorption test equipment at Houston was a custom design for dynamic measurement. The dynamic H<sub>2</sub> chemisorption test method measures the catalyst's ability to absorb H<sub>2</sub> from a flowing gas stream. The depletion of H<sub>2</sub> from the flowing gas is detected and integrated. Based on prior collaborations with Shell, Aerojet already had a similar test rig available. Here we simply had to adopt the Shell test method for use on the Aerojet test rig.

#### **Laboratory Test Verification.**

Verification of the crush tester could not be carried out on an original sample of Houston-processed RA-1 carrier since Shell did not have samples of carrier traceable to specific test results. Samples of the carrier prepared as part of our initial production were tested and the results are very similar to the results reported by Shell for carrier. Shell has not established a specification for crush testing of finished catalyst.

Redmond BET and pore volume testing of carrier materials yielded results within 93% of the values reported by Shell. This discrepancy is explained by test rig differences between when the Shell data was collected in 1983 and the current automated test equipment used at Redmond. BET testing of Shell-produced catalyst at Aerojet reproduced the reported BET values within 5%.

H<sub>2</sub> chemisorption testing of Shell-produced catalyst on the Aerojet test rig using the Shell test method matched the values originally reported by Shell within 1%.

Based on these data comparisons we can say that the Aerojet laboratory test equipment is equivalent to the Shell/Houston laboratory test equipment.

#### **Laboratory Test Results of Initial Production.**

Carrier crush, carrier BET, carrier pore volume, catalyst BET and catalyst H<sub>2</sub> chemisorption tests were measured on the initial production materials. All parameters were within specification and were very similar to the values measured by Aerojet for Shell 405 materials (see above).

The laboratory test results indicate that the S-405 produced at Redmond meets the product specification and matches well with the Shell 405 data family. In addition, the test results for the two "half batches" (from reduction) were very similar and indicated that the low H<sub>2</sub> flow experienced during the processing of the first "half batch A" did not result in diminished performance or degraded product. (Rocket engine testing also supports this conclusion, see below).

#### **Rocket Engine Test Equipment**

**Houston Configuration.** The Shell 405 rocket firing test was developed in the early 1960s for the purpose of verifying the ability of the catalyst to decompose propellant grade (98%) hydrazine<sup>1</sup>. During the past 40 years there has been little or no changes to the test equipment or procedure.

The Shell test reactor in Houston consisted of a 5 lbf headspace injector design which was modified to accommodate a smaller bed size. The nozzle

throat and injector were also modified to accommodate a reduced flow rate.

Shell constructed a custom propellant feed system to provide cold propellant to a cold reactor. A 1000 ml jacketed feed tank was used to cool the propellant prior to testing. Between the feed tank and the firing valve was a water-jacketed feed line. Both the line and tank were insulated with Styrofoam pipe insulation and cooled with ice cold water. At the end of the feed line was the firing valve assembly. The last section of propellant feed line (~6 inches), the valve, and the line between the valve and the reactor were not thermally conditioned. The reactor was thermally conditioned by packing with ice. Finally, the reactor outlet connected to an exhaust duct which discharged outside the test facility.

A typical acceptance test firing included the following steps: The reactor was first cleaned and inspected. The reactor was then packed with the catalyst sample and then installed in the feed system. Propellant was loaded into the tank and cooled with circulating ice water. The reactor exterior was packed with ice. Once the propellant and reactor bed reached the prescribed temperature, the ice was removed from the reactor. After firing, the firing valve was closed and the reactor prepared for the next test sequence. After the last test sequence, the reactor was removed and disassembled in order to recover the catalyst. The recovered catalyst was then sieved and weighed.

**Redmond Configuration.** Aerojet's test system was designed with the intent of duplicating Shell's configuration while allowing for changes to accommodate simplicity and improved safety. Modern equipment was installed as necessary to substitute for components that were no longer available. Figure 1 shows a schematic of the test firing setup.

Aerojet's test system is located in a temperature-controlled chamber that conditions the entire test system to the required temperature. Outside the conditioning chamber are pressure regulators and valves that provide GN<sub>2</sub> to the system.

The propellant run tank supplies propellant through a turbine flow meter (FM) for direct measurement of the flow rate. Downstream of the flow meter are the feed pressure transducer (Pfeed) and the thruster control valve (TCV).

Aerojet's reactor is functionally identical to Shell's reactor. This reactor was designed using information from Shell's report<sup>1</sup> and inspection of

an old test unit which Shell provided for measurement.

The injector head spacer, catalyst bed and screens are identical in design and size to Shell's reactor. The Type K thermocouples and pressure transducers ports are also in the same location as on Shell's reactor.

Aerojet's instrumentation consists of digital computers, pressure transducers, thermocouples, thruster valve voltage and current lines, and a flow meter. One computer operates the test and triggers the second computer to record the data. The recording computer records each of the signals at 1000 Hz per channel. These signals are the pressures, temperatures, thruster valve voltage and current, and the flow rate.

Acceptance testing at Aerojet's facility is done in the following manner. The reactor is cleaned and the nozzle throat is measured. The reactor is then packed with the catalyst and inert material. The reactor is then leak checked and installed in the test fixture. The propellant tank is filled with propellant and then connected to the feed system. The various pressure transducers and instrumentation are then connected to the setup.

The temperature controlled oven conditions all of the components to the required temperature range. Once the fuel and reactor reach the correct temperature the digital recorder is started and the test firing initiated. After firing, the thruster valve is closed and the reactor is cooled for the next test.

After firing the required number of sequences the reactor is removed from test facility and disassembled. The catalyst is then sieved and weighed.

After each test sequence the digital data is analyzed to determine the downstream chamber pressure, bed outlet temperature, flow rate, run duration, fuel temperature and ignition delay. All of these values except ignition delay are simply read directly from the digital data. The ignition delay is calculated from the digital trace as the time interval between the valve open signal and the first increase in the downstream chamber pressure by 1 psig. From this time the system fill time is then subtracted to give the corrected ignition delay. The system fill time is measured from the recovery time of the Pf pressure transducer.

Significant differences exist between Shell's facility in Houston and Aerojet's facility in Redmond. These significant differences include:

- Aerojet's setup includes a flow meter

- Aerojet's cooling of the reactor and test facility is accomplished using an environmental chamber. This results in a very thorough and uniform thermal conditioning
- Aerojet uses a fast acting solenoid valve and measures the propellant flow delay for each test sequence.
- The injector nozzle used by Aerojet is the one called out in the Shell documentation. Inspection of the equipment provided by Shell suggested that they were not actually using the injector nozzle noted in the documents.

### **Rocket Engine Test Verification**

**Houston Data And Specification.** Each batch of catalyst made by Shell was engine tested in order to measure the catalyst's performance. The Shell 405 specification calls out eight acceptance requirements for the engine test. These are as follows:

- Packed Bed Volume: This is the volume of catalyst packed into the reactor for the test.
- Propellant Feed Rate.
- Reactor Throat Internal Diameter
- Number of Test Sequences. Requirement depends on mesh size of catalyst.
- Ignition Delay. The time from valve opening to initial indication of reactor pressure rise corrected for propellant fill time.
- Loss + Fines: After firing, the reactor was disassembled and the catalyst re-sieved. The mass of fines and lost material was recorded.
- Bed Outlet Temperature
- Outlet Pressure: The end of run downstream chamber pressure

Shell personnel at Shell's Westhollow Technology Center in Houston tested Aerojet's initial production batch of S-405. Batches A and B were tested separately to investigate possible differences in the catalyst performance due to changes in the reduction processing. No differences were found. All of the parameters met Shell's acceptance criteria and fell within the normal family of data for Shell 405. Figure 2 shows a sample S-405 Visicorder trace. All of the S-405 traces are indistinguishable from traces of Shell 405.

**Redmond Data And Revised Specification.** After fabrication and assembly of Aerojet's catalyst

test facility, tests were conducted to demonstrate proper operation. During these tests several minor problems were uncovered and resolved. A baseline configuration was selected which reliably provided adequate test results. This baseline configuration is referenced to as the "Standard Conditions" and consists of the following items:

Twelve test series were conducted under these standard conditions, three of which were done using Shell 405 manufactured in Houston (Lot # 2-ASHO-403-2).

Shown below in Figures 3 - 5 are typical pressure and temperature traces of tests performed in Aerojet's test facility. (The catalyst used in these tests was Shell 405).

As a result of the tests conducted under standard conditions, Aerojet has established S-405 acceptance criteria. These acceptance criteria are identical to the Shell 405 criteria for all parameters except for ignition delay. It was Aerojet's original intent to adopt, without change, all of the Shell 405 requirements. Now that more information is known about the limitations of Shell's ignition measurements and about the differences in the facilities, Aerojet feels that it is wise to adjust the ignition delay limit according to the current facility. The specifics of the S-405 ignition delay are as follows.

- Ignition Delay. Increased Shell 405 limit by 14ms. Ignition delays in Aerojet's facility are consistently longer than the delays in Shell's facility for both S-405 and Shell 405. As a result, Aerojet has adjusted the limit accordingly. Using data collected from the test series conducted under standard test conditions for both Shell 405 and S-405, Aerojet has selected a limit based on the Upper Natural Process Limit of the Rocket Engine Validation Testing.

### **Rocket Engine Test Results for Initial Production.**

Aerojet's initial batch of S-405 was acceptance tested in Redmond on January 9, 2003. All required parameters met the S-405 acceptance criteria

Shown in the Figures 6 - 8 are pressure and temperature traces for the tests. These traces are identical to traces of Shell 405.

### **RECOMMENDATIONS**

The experience of transitioning the production and testing of Shell 405 catalyst from Houston to our

facility in Redmond has provided us with several opportunities to "improve" the production and testing over what was carried out at Houston. In general this temptation has been largely resisted since we felt very strongly about maintaining all aspects of the heritage of this product – good or bad. At the outset we did not feel we had sufficient experience with all aspects of the production and testing to simply "jump in" and start making changes.

Over the course of the transition process we have learned a lot about the rocket engine test. We have also learned its history and its limitations. In reviewing our progress and setbacks with regard to the rocket engine test we have discussed internally and with our NASA transition team ways to improve the test. There are two lines of reasoning.

One approach on the rocket engine test is to drop the requirement entirely. According to Shell personnel, they have never failed a batch of catalyst based on the rocket engine test. The test does not simulate the current application environment. Shell and Aerojet have relied for many years on the H<sub>2</sub> chemisorption test as a reliable indicator of catalyst activity. Catalyst with diminished H<sub>2</sub> chemisorption activity shows poor performance when tested in rocket engines. When catalyst is recovered from engines that have shown diminished performance, the H<sub>2</sub> chemisorption value is low. Aerojet's experience over 25 years of in-house LCH catalyst production has never identified a catalyst that passed the H<sub>2</sub> chemisorption test, but subsequently failed rocket engine firing. This experience seems to hold true for Shell as well. The rocket engine test could readily be eliminated from the production process and would result in faster and less costly catalyst production. Aerojet recommends this approach.

An alternative suggestion would be to update the rocket engine test hardware and test conditions. The original test reactor design is not representative of current flight hardware. In addition, the original (current) rocket engine test is conducted at low temperature and atmospheric pressure. This also does not reflect current applications for the catalyst. An updated rocket engine test would entail a slight modification of a current flight-qualified design to enable repeated filling and recovery of catalyst (convert to bolt-up design). Test fixture improvements would be to conduct the testing with a flight-quality firing valve in a vacuum chamber. These conditions would eliminate many of the variables and bring the

acceptance test much closer to actual current applications.

A second area of discussion regards the need to "qualify" or "delta-qual" Aerojet's S-405 material for use in hardware that had previously been designed and qualified with Shell 405 (Shell production). As indicated earlier in this report, one of the major tenets of this technology transition effort was to maintain the heritage of the Shell 405 production. Equipment, documentation and testing were targeted to be identical to the Houston production facility. With the one minor exception of the ignition delay time specification of the rocket engine testing, we conclude that we have duplicated not only the process, but also the testing and the product as well. The testing of the S-405 product indicates it is identical to the "form, fit and function" of Shell 405 produced in Houston and the two products are equivalent and interchangeable. S-405 and Shell 405 exhibit identical beginning of life characteristics. In Aerojet's opinion, re-qualification of hardware for use with S-405 is unnecessary. However, the product has not been tested for life capability and end-of-life characteristics. It will be up to the individual users to assess the need to perform qualification and life testing as desired.

#### **SUMMARY AND CONCLUSION**

Shell Chemical Co. manufactured Shell 405 catalyst for many years. This catalyst is a key, "enabling" technology for spacecraft and launch vehicles. Shell Chemical Co. has exited the Shell 405 catalyst manufacturing business and, based on years of catalyst preparation experience, Aerojet has elected to assume this manufacturing responsibility.

This program was initiated to maintain the S-405 catalyst manufacturing capability. Aerojet (with substantial partial funding from NASA) has assembled manufacturing and test facilities at the company's Redmond, WA location. The Aerojet facility, manufacturing process and test equipment closely matches the installation successfully used for many years by Shell Chemical Co.

The manufacturing and testing of S-405 catalyst has been successfully transferred to the Aerojet facility at Redmond, WA. The transition preserves the production heritage established by Shell at their original Emeryville, CA facility and their recent production at the Houston, TX facility. The transition process has captured the original raw materials, documentation and testing used by Shell. We have included as part of the transition,

improvements in the manufacturing and test process documentation to bring them up to current standards for aerospace materials. Aerojet has a qualified S-405 production process in place and is now a qualified S-405 supplier. S-405 is ready for use in spacecraft and launch vehicles or for additional testing. Aerojet has demonstrated equivalent beginning of life characteristics for Shell 405 and S-405. Additional qualification and life tests were outside the scope of this transition effort and, if desired, will be the responsibility of the end-user to perform. Aerojet is available to support such efforts and is willing to enter contracts to perform additional catalyst testing and to act as a clearinghouse to share non-proprietary testing information with the user community.

**ACKNOWLEDGMENTS**

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**ADDENDUM**

Since the completion of the joint effort described in this report, Aerojet has successfully completed the manufacture of two further batches of S-405 catalyst - one batch of 14/18 mesh and one of 25/30 mesh. Processing of both batches went smoothly and all test parameters were nominal and within specification.

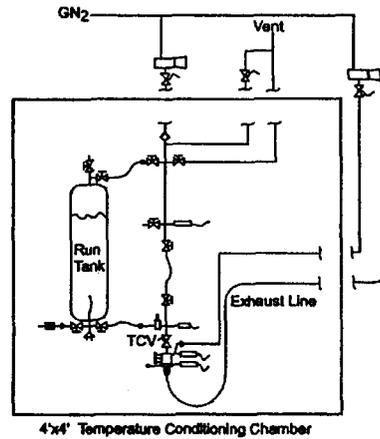


Figure 1: Schematic of Aerojet's S-405 Test Facility

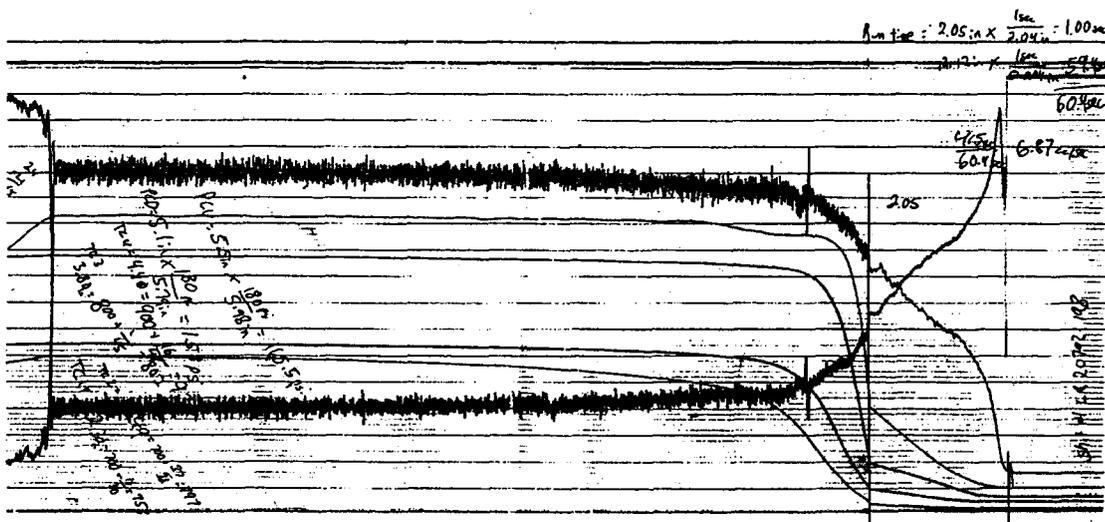


Figure 2: Visicorder Trace Recorded During Testing of S-405 in Houston.

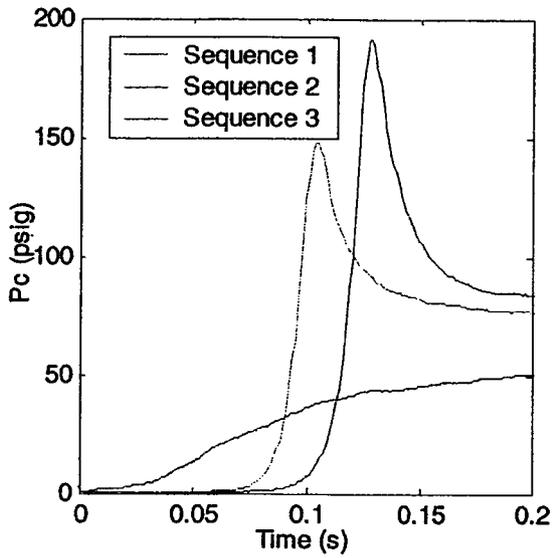


Figure 3: Ignition response of Shell 405.

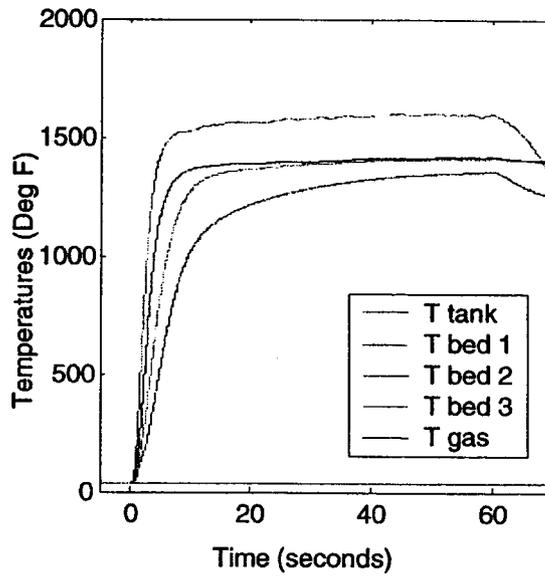


Figure 5: Typical Temperatures - Shell 405

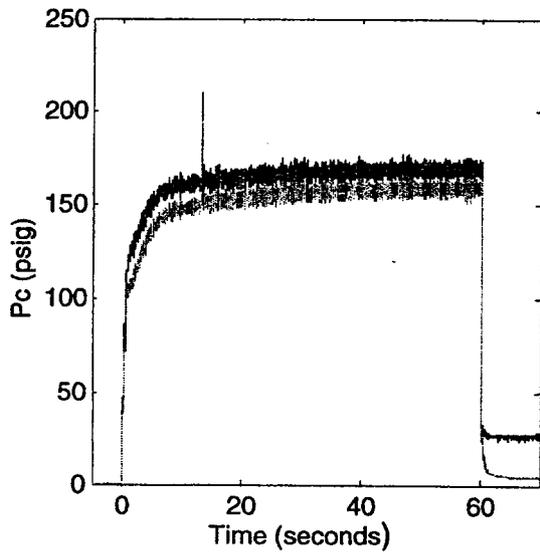


Figure 4: Typical Chamber Pressure - Shell 405.

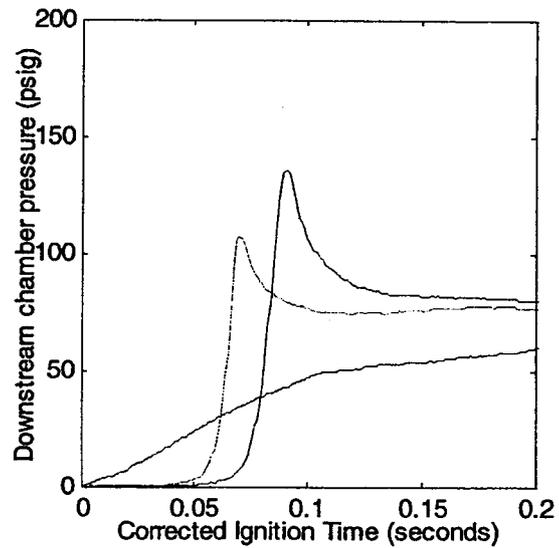


Figure 6: Ignition Response of S-405 from the Acceptance Test

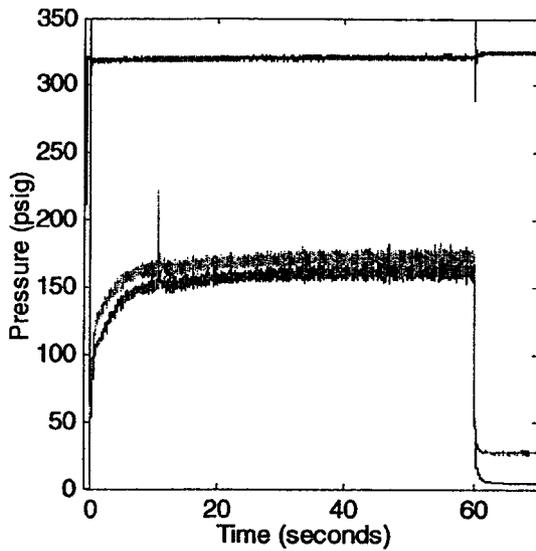


Figure 7: Chamber Pressures During S-405 Acceptance Test

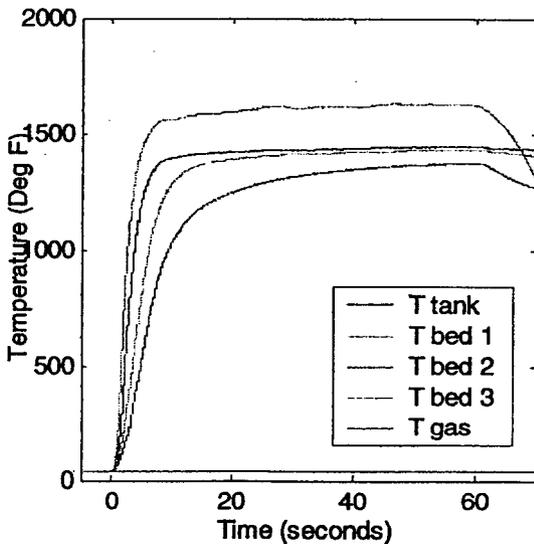


Figure 8: Temperatures During S-405 Acceptance Test.

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