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EVALUATION OF THE IMPACT OF KEROJET™ AQUARIUS WATER SCAVENGER ADDITIVE ON THE THERMAL STABILITY OF JET A FUELS

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Interim Report

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14. ABSTRACT
In a total of 11 advanced, reduced-scale fuel system simulator (ARSFSS) runs and several quartz crystal microbalance (QCM) and jet fuel thermal oxidation tester (JFTOT) tests, In a total of 11 ARSFSS Runs and several QCM and JFTOT tests, the data shows beyond reasonable doubt that for this program, the additive demonstrated no discernable negative impact on the thermal stability characteristics of the fuel used based on the testing performed. At the anticipated commercial use dosage, the additive results in thermal stability performance characteristics indiscernible from the baseline fuel. Testing also showed that under simulated aircraft fuel system conditions, the 4X dosage rate actually improved fuel thermal stability characteristics by reducing deposition in fuel wetted components and reducing hysteresis in servo valve and flow divider valve performance.

Based on the testing performed, Aquarius Water Scavenger Additive has no apparent or discernible negative impact on fuel thermal stability characteristics.

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List of Acronyms

ACRONYM LIST	
Acronym	**Definition**
AFPA	Air Force Petroleum Agency's Wright-Patterson Aerospace Fuels Laboratory
AFRL	Air Force Research Laboratory
AFTSTU	Aviation Fuel Thermal Stability Test Unit
ARSFSS	Advanced Reduced Scale Fuel System Simulator
BFA	Burner Feed Arm
CI/LI	Corrosion Inhibitor/Lubricity Improver
CONUS	Continental United States
EDTST	Extended Duration Thermal Stability Test
FAME	Fatty Acid Methyl Ester
FCOC	Fuel-Cooled Oil Cooler
FDV	Flow Divider Valve
FSII	Fuel System Icing Inhibitor
GDTC	Generic Durability Test Cycle
HP	High Pressure
ICP	Inductively Coupled Plasma
JFTOT	Jet Fuel Thermal Oxidation Tester
mg	milligrams
ppm	Parts per Million (by Volume unless otherwise stated)
QCM	Quartz Crystal Microbalance
RQTF	Fuels and Energy Branch, AFRL
SDA	Static Dissipator Additive
SEM	Scanning Electron Microscope
SV	Servo Valve
WWT	Wetted-Wall Temperature
XRD	X-Ray Diffraction
XRF	X-Ray Fluorescence
XRM	X-Ray Mapping (or Dot Mapping)
WWT	Wetted-Wall Temperature
µg	Micro-grams

1.0 EXECUTIVE SUMMARY

Aquarius Water Scavenger Additive was evaluated for its impact on the thermal stability characteristics of Jet A aviation turbine fuel using a variety of test devices – Quartz Crystal Microbalance (QCM), Jet Fuel Thermal Oxidation Tester (JFTOT) and the Advanced Reduced Scale Fuel System Simulator (ARSFSS). The additive was evaluated in these devices at the anticipated commercial usage concentration of 250 ppm by volume and at four times (4X) this concentration, i.e. 1000 ppm.

A total of 11 ARSFSS Runs and several QCM and JFTOT tests, the data shows that the additive demonstrated no discernible negative impact on the thermal stability characteristics of the fuel used based on the testing performed. At the anticipated commercial use dosage of 250 ppm, the thermal stability performance characteristics were indiscernible from the baseline fuel on all three platforms. Testing also showed that under simulated aircraft fuel system conditions, the 4X dosage rate appeared to improve fuel thermal stability characteristics by reducing deposition in fuel wetted components and reducing hysteresis in Servo Valve and Flow Divider Valve performance.

Based on these results, the Aquarius Water Scavenger Additive has no apparent or discernible negative impact on fuel thermal stability or other characteristics.

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2.0 INTRODUCTION AND BACKGROUND

For safety of flight and maintenance reasons, it is highly desirable to minimize or eliminate water from all flight aircraft fuel systems and ground support systems.

EI 1581 or 1583 Filter/Separator devices can remove free water down to less than 15ppm. However, these devices and the procedures that accompany them do not remove dissolved water. For example, a fully fueled wide-bodied aircraft with approximately 200,000 liters (about 53,000 gallons) leaving Dubai at 40C and 100% humidity will potentially produce 20 liters (a little over 5 gallons) free water during the flight even if the aircraft fuel system is 'clean and dry' upon takeoff. Considering the altitudes at which these type of aircraft fly and the duration of the flight, the threat of fuel flow interruption due to the formation of ice resulting from the presence of this free water is substantial. While aircraft fuel systems are designed to minimize this threat, a system devoid of free-water is very desirable.

Over the past few years, significant effort has been invested in the development of a fuel additive which can 'scavenge' free water in fuel systems and eliminate the potential free water formation by keeping the water 'dissolved' in the fuel. However, as with anything that is proposed to be added to fuel, safety-of-flight concerns dictate that any such addition be scrupulously evaluated. In order to assure that fuel specification, fit-for-purpose, safety-of-flight, system performance and maintenance characteristics remain unchanged, rigorous testing is undertaken to evaluate the impact of any new additive on these characteristics. For the additive which is the subject of this evaluation, Aquarius (referred to in the remaining portion of this report as "Aquarius"), a significant amount of testing has already been successfully accomplished. However, while all of the testing accomplished so far indicates that the Aquarius will perform adequately and safely in aircraft systems, this testing has all been accomplished using bench-scale tests. While these tests attempt to predict the chemistry and relative performance impact of the additive, it is still desirable to evaluate the additive in as close to a 'real world' aircraft fuel system as possible. Successful passing of such an evaluation can give operators and maintainers a great deal of confidence that the additive will perform safely and adequately in flight systems.

Since the mid-1980's, the U.S. Air Force at the Air Force Research Laboratory located at Wright-Patterson Air Force Base, Ohio, has been operating a Advanced Reduced Scale Fuel

System Simulator (ARSFSS). As the name suggests, this test rig simulates the engine and airframe fuel systems for an advanced aircraft. In its current state, the ARSFSS is configured to simulate an advanced military fighter-type aircraft. However this configuration is easily altered to simulate more conventional commercial-type aircraft.

The ARSFSS is a unique system that permits evaluation of fuels and additives under near- real-world aircraft operational conditions. It can be operated in two different modes – a mode which simulates real-world mission conditions and a steady-state mode.

2.1 General ARSFSS Description and Operations

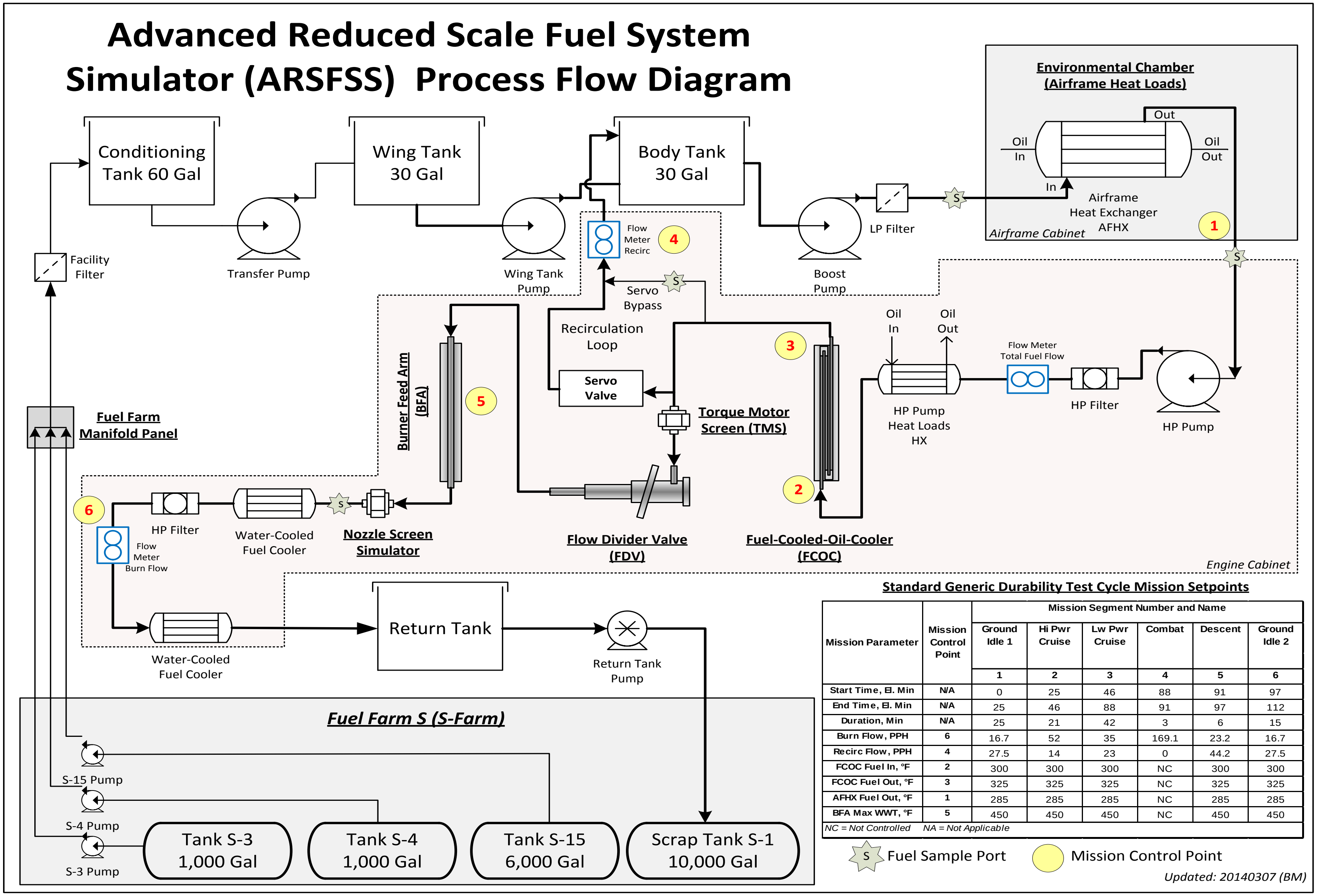
The Advanced Reduced Scale Fuel System Simulator (ARSFSS) is a thermal stability evaluation device that more closely represents and replicates military aircraft fuel system operating conditions than any other sub-aircraft scale test device in the world. Designed as a joint effort between AFRL, Boeing and Rolls Royce (UK) in the mid-1980s, the ARSFSS has been used extensively to evaluate fuels and additives under realistic aircraft fuel system conditions for almost three decades. The ARSFSS is used by AFRL as the last test before releasing a fuel or additive for engine- and component-scale testing and evaluation, or for use in the field. Not only is the ARSFSS capable of realistically simulating the flow, temperature, pressure and residence time profiles for a real aircraft fuel system, but it is capable of imposing these conditions on system hardware in real time with changes to flow, pressure and temperature conditions following a pre-established mission profile. In this way, the ARSFSS can 'fly' missions sequentially over time. An ARSFSS test run typically consists of between 65 and 150 missions executed sequentially operating 24 hours per day, 7 days a week. The ARSFSS control system is sophisticated enough to allow the test to operate unattended for days at a time.

The ARSFSS rig itself consists of three major subsystems: a Fuel Conditioning System, an Airframe Fuel System Simulator, and an Engine Fuel System Simulator

A schematic of the ARSFSS is shown in Figure 1. Figure 2 shows the Conditioning and Wing Tanks which comprise the fuel conditioning system and part of the airframe simulator.

Figure 3 shows the Body Tank which is also part of the Airframe Simulator. Figure 4 shows the Environmental Chamber, which is also part of the Airframe Simulator and is where heat loads associated with environmental systems and other airframe subsystems are imposed upon the fuel. This chamber is represented by the ‘Airframe Heat Loads’ element in the Figure 1 schematic. The remaining elements of the ARSFSS are all encompassed in the Engine Simulator. A front view of the Engine Simulator cabinet is shown in Figure 5.

The ARSFSS is configured to simulate an advanced aircraft with an advanced engine. Rig scaling is based on 1/3 scale of a single nozzle (the full-scale engine has 24 nozzles) – making the ARSFSS scaled overall at 1/72nd scale of the advanced engine. Total fuel required for each ARSFSS test is between 900 and 1500 gallons – depending on the mission profile used for the testing. For this program, a modified Generic Durability Test Cycle (GDTC) mission profile was used. Sixty-Five (65) mission cycles were executed for each test run requiring approximately 900 gallons of fuel.



Standard Generic Durability Test Cycle Mission Setpoints

Mission Parameter	Mission Control Point	Mission Segment Number and Name: Ground Idle 1 (1)	Hi Pwr Cruise (2)	Lw Pwr Cruise (3)	Combat (4)	Descent (5)	Ground Idle 2 (6)
Start Time, El. Min	N/A	0	25	46	88	91	97
End Time, El. Min	N/A	25	46	88	91	97	112
Duration, Min	N/A	25	21	42	3	6	15
Burn Flow, PPH	6	16.7	52	35	169.1	23.2	16.7
Recirc Flow, PPH	4	27.5	14	23	0	44.2	27.5
FCOC Fuel In, °F	2	300	300	300	NC	300	300
FCOC Fuel Out, °F	3	325	325	325	NC	325	325
AFHX Fuel Out, °F	1	285	285	285	NC	285	285
BFA Max WWT, °F	5	450	450	450	NC	450	450

NC = Not Controlled NA = Not Applicable

Figure 1 - ARSFSS Flow Schematic

Figure 2 - Conditioning Tank (Left) and Wing Tank (Right)

Figure 3 - Body Tank

Figure 4 - Environmental Chamber

Figure 5 - Front View of Engine Simulator Module

2.2 Servo Valve (SV)

For the ARSFSS, the Servo Valve component (Figure 6) is the second stage or hydraulic portion of an Electro-Hydraulic Servo Valve (EHSV) commonly found in advanced engines. This particular valve has a diametrical clearance of 0.00010 – 0.00020 inches and a total stroke of +/- 0.032 inches. In an EHSV, the first stage of the control is an electrical servo mechanism that responds to an input current or voltage. Increasing current or voltage results in a small movement of the electrical servo components. The electrical servo components are coupled to a hydraulic component – the second stage of the control of the valve. The hydraulic portion of the valve consists of a spool and sleeve arrangement where a specially designed spool moves within a sleeve. Movement of the spool causes clearances within the spool/sleeve assembly to change and thus, control flow through the valve. Because the hydraulic portion of the valve is driven by pressures within the fuel system, the small forces generated by electrically positioning the electrical-servo portion of the valve are amplified by system hydraulic pressures resulting in a substantial moving force being applied to a hydraulic component. These combined electrical and hydraulic components give engine manufactures the ability to exert substantial hydraulic forces upon the fuel system control using small electrical forces. However, since the hydraulic portion of the valve sees the fuel flow at bulk fuel system temperatures, coking and fouling can occur in these components.

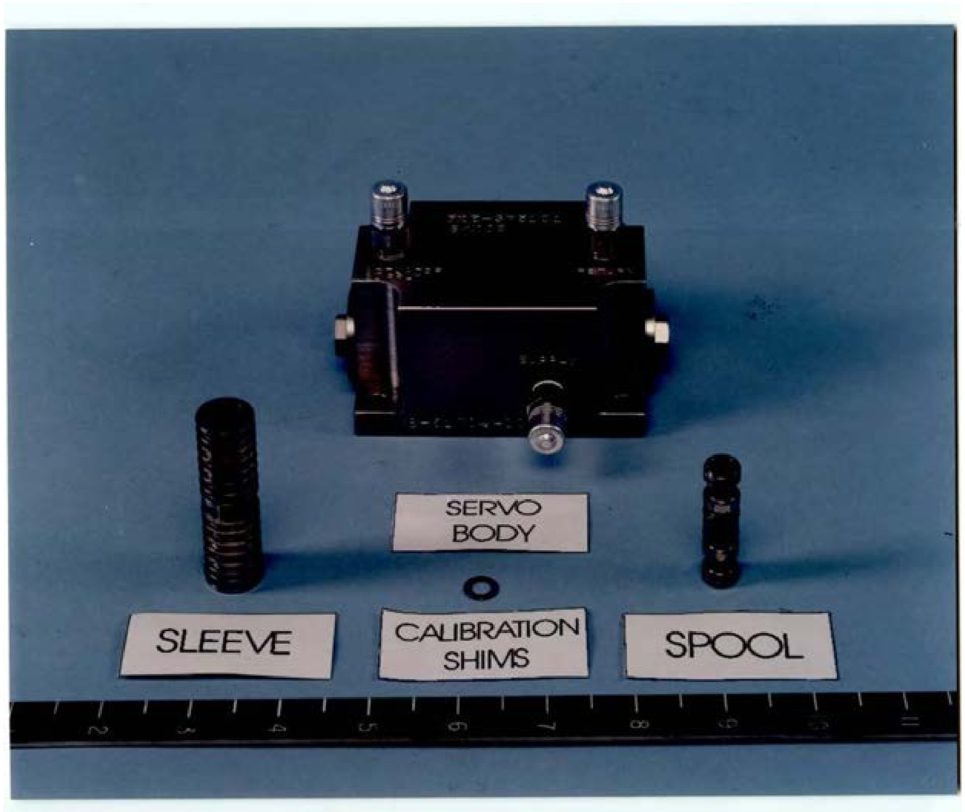


Figure 6 - Servo Valve Module

Since the ability of the EHSV to regulate fuel flow is dependent upon the unrestricted movement of the spool and sleeve valves that make up the hydraulic portion of the valve, even the slightest amount of deposition occurring in this valve can impact valve performance by causing hysteresis in the valve. Hysteresis in a valve can basically be described as the tendency of the performance of the valve (in terms of valve flow and pressure) to be dependent on its previous position along with whether the change in pressure to cause a change in valve flow is increasing or decreasing when reacting to an external control signal. Hysteresis leads to varying degrees of inaccuracy relative to valve actuation and operating forces and can drastically affect the performance of an engine fuel system. Under the best of circumstances, a well-designed and well-functioning control valve has little or no hysteresis thereby allowing the control algorithms that predict and impose control movements to reliably and predictably position the valve for stable system control. As hysteresis increases, control algorithms may not properly compensate and system control can become unstable.

For all ARSFSS testing, SV hysteresis is measure pre- and post-test and is defined by relating differential pressure (DP) across the SV to flow rate (F) through the valve. To generate this SV Differential Pressure (DP) vs. Flow data curve, the ARSFSS HP Engine Pump is operated at a fixed high RPM to generate fuel pressures necessary to actuate the SV. Fuel flow from the pump is regulated by a control valve (FCV801) starting with the control valve set to about 75% which applies pressure to the SV and forces it to a ‘closed’ position. Since the SV is not a ‘shut-off’ valve, there is usually some small measure of flow through the valve. With FCV801 at 75% (SV essentially closed), a flow measurement is made once it is determined that the flow through the valve is stabilized. Once that measurement is taken, FCV801 is set to 70% open and another measurement of flow is made. This stepwise closing of FCV801/opening of the SV continues in 5% increments until the SV is essentially full open (which is about 30% on FCV801). Once the final flow measurement is made at this condition, FCV801 is changed again, in 5% increments until FCV801 is back at the starting position of 75%. Flow measurements are made at each of these incremental positions and the results tabulated.

These measurements are made on the SV while it is installed in the ARSFSS both pre-test and post-test. The cyclic measurement process is executed a minimum of two and a maximum of three times and the data collected and tabulated. The cyclic measurement process is repeated because it is common for the first sequence of measurements to be ‘off’ slightly as a result of the valve ‘seating’ itself and getting fully wetted and lubricated with fuel. The second measurement series tends to be more representative of the SV in operational mode. The third and final series tends to virtually duplicate the second series so it is most times not performed. In the post-test mode, the third series is only performed if there are too many anomalies evident in the first two series because valve movement tends to remove deposition from the valve thereby returning the valve to a near-pre-test condition and thus eliminating the ability to assess the impact of coking on valve performance.

In addition to the hysteresis measurements made on the Servo Valve, at the end of each test run the Servo Valve is disassembled and photographed to document the amount and nature of the fuel deposits inside and on the valve components. This deposition, along with Servo Valve hysteresis measurements, documents the condition of the valve at the end of each test.

The very nature of the EHSV tends to minimize the impact of hysteresis naturally so no firm value for hysteresis in this component has been established as an acceptable amount. Instead, SV performance is generally evaluated as a ‘do no harm’ criteria. Post-test SV hysteresis behavior is determined generally to be acceptable as long as the hysteresis is not significantly different from pre-test measurements. This causes the data obtained on the SV performance to be somewhat subjective rather than analytical.

2.3 Flow Divider Valve (FDV)

Perhaps even more critical than the EHSV, valve hysteresis is a significant issue in the combustor nozzle Flow Divider Valve (FDV) shown in Figure 7. The engine simulator part of the ARSFSS was designed around an advanced engine using 24 combustor nozzles. Each of the 24 combustor fuel nozzles for this design contains two fuel flow paths to the injector nozzle – a Primary and a Secondary. The Primary path typically handles fuel flow in the ‘low’ power or low fuel flow regime - for example, engine starting and ground idle and idle descent and conditions. Once the engine requires fuel flows outside of this ‘low flow’ regime, a Secondary ‘high flow’ path is opened up to deliver the necessary flow to the engine. This ‘dividing’ of the fuel flow is accomplished using a pressure-driven ‘Flow Divider Valve’ (FDV). This valve is physically positioned upstream of the fuel nozzle face and is located outside of the combustor in the compressor bypass or fan air flow path. Since this air flow can reach high temperatures, the FDV is subject to occurrence of coking. As with any other valve that is used to regulate flow, any coking or fouling of the FDV can result in significant valve hysteresis. Unlike the EHSV, the FDV is driven only by inlet fuel pressure and does not have the benefit of multiplied hydraulic forces to overcome hysteresis. Any hysteresis in this valve can therefore change fuel flow characteristics and thus affect the combustor radial temperature profile and the critical Turbine Inlet Temperature profile. Adverse changes in this profile can have profound adverse effects on engine hardware.

In the ARSFSS, an actual FDV from an advanced engine is used. The flow slot has been modified by narrowing its width so that the typical stroke of the valve in the ARSFSS’ reduced flow environment is essentially the same as for the full flow in the engine. Figure 7 shows the various components of the FDV as well as an assembly view of the FDV itself.

Figure 7 - Flow Divider Valve Assembly

The normal acceptability criteria for FDV hysteresis would be 7% or less. According to design engineers, hysteresis values beyond 7% could adversely impact the fuel flow to the nozzles and thus change the combustor temperature profile in the engine. An altered combustor temperature profile can have serious and deleterious impact on engine performance, reliability and safety.

Hysteresis measurements on the FDV are determined in much the same way as for the SV. As with the Servo Valve, in addition to determining FDV valve hysteresis, the FDV is disassembled and photographed at the end of each ARSFSS run to document the degree and nature of the deposition that occurred in and on the valve components. These components include the FDV valve body, valve stem and strainer screen that surrounds the entire assembled valve and protects it from large pieces of debris.

2.4 Fuel-Cooled Oil Cooler (FCOC)

Aircraft fuel is used for cooling as well as propulsion. One area where fuel is used as a cooling medium is in the cooling of engine lubrication oil. In most systems, this involves simply exchanging heat between the engine oil and the fuel in a simple heat exchanger device – a Fuel-

Cooled Oil Cooler (FCOC). The FCOC is based on a shell-and-tube heat exchanger design where fuel passes through the exchanger on one side of the tube and engine lubrication oil passes on the other side. The number of tubes used in the FCOC depends upon the engine design and the amount of heat dissipation required. Normally, accepted engine design criteria dictates that bulk fuel temperature out of the FCOC should never exceed 325 °F (163 °C) which is the limit for oil operability in the engine. Obviously, at these temperatures, fuel can foul and coke can be deposited on the inside of the tubes of the FCOC. As with any heat exchanger, any fouling, either on the inside or the outside of the tubes, is detrimental to FCOC performance and can result in engine oil temperatures exceeding design limits. In the ARSFSS, the device simulating the engine FCOC is designed with three 3/8-inch diameter 0.035-inch thick walled stainless steel tubes. The tubes are connected via manifolds at either end of the FCOC device so that the fuel sees three complete end-to-end passes within the FCOC before emerging. The tube that is used for the final pass is removed at the end of each test and cut into 2-inch segments. A LECO Carbon Analyzer is used to measure the amount of carbon deposition that has occurred inside this tube. This carbon deposition data is plotted as part of the data for the ARSFSS run. No firm quantitative acceptance criteria has been established for this device. Acceptance is based on the deposition for the fuel under test being not more than the deposition for the baseline fuel. Figure 8 shows the FCOC as disassembled and as installed in the ARSFSS rig.

Figure 8 - Fuel-Cooled Oil Cooler

2.5 Burner Feed Arm (BFA)

In the engine that was used as a model for the ARSFSS simulator, each combustor nozzle is made up of an assembly of three components – the FDV (which was discussed in a previous Section), the tubular pathways connecting the FDV to the nozzle (often referred to as the 'Burner Feed Arm' (BFA)) and either a pressure-atomizing or air-blast nozzle. The FDV regulates fuel flow to the Primary and Secondary fuel flow paths which transport fuel through the flow tubes (Burner Feed Arms) to the nozzle. In the actual nozzle assembly, since this portion of the nozzle assembly is subjected to high temperature compressor discharge air, these paths are contained within a complex shroud assembly designed for thermal isolation and protection. As previously described, the performance of the combustor fuel nozzle is critical to engine performance and control. This performance and control is not only impacted by the performance of the FDV in each combustor nozzle assembly, but it is impacted by the ability of the BFA flow paths to deliver unrestricted fuel flow to the nozzle. Significant coke deposits can, however, develop inside these tubes which can restrict fuel flow to the nozzle and therefore impact nozzle assembly overall performance - even though these paths are shrouded for thermal protection.

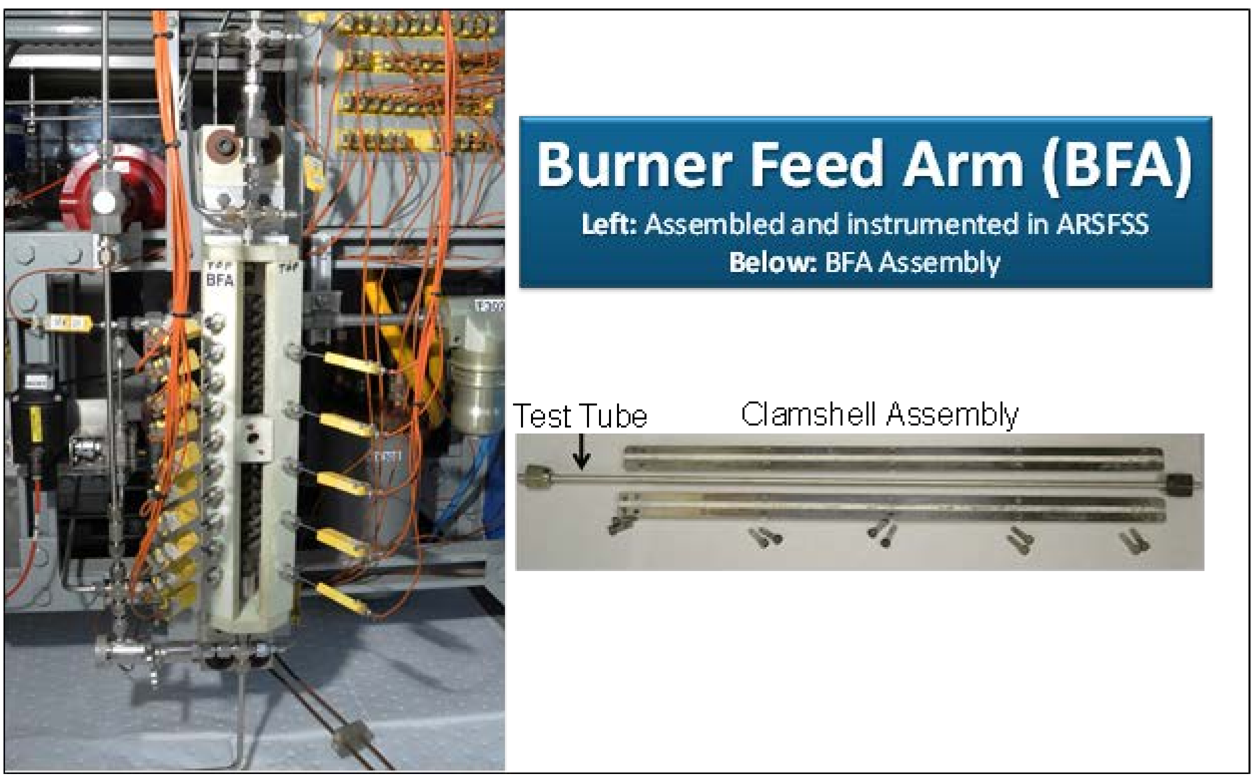


Figure 9 - Burner Feed Arm Assembly

Figure 9 shows the design of the BFA and its implementation in the ARSFSS. The BFA is constructed of two pieces. The first piece, the test article tube, is made of a 316 stainless steel tube 0.125 inches O.D, 0.085 inches I.D. with a 0.020 inch wall cut to 13.25 inches in length. This test article tube is placed inside a clamshell of 316 stainless steel that is 0.5 inches overall diameter and cut to 10.5 inches long. The clamshell is split in half so that is bolted around the test article tube. The clamshell is drilled through with ten thermocouple holes placed as shown in Figure 10. These allow thermocouples to be inserted through the clamshell to touch the external wall of the test article tube. These thermocouples measure the approximate internal fuel-wetted wall temperature along the test article tube and are designated TE317 through TE326.

The clamshell is also prepared with divots on its external surface along its length as described in Figure 10. As the assembled unit is installed in the holder on the ARSFSS (Figure 9), thermocouples are positioned in these divots to measure the external wall temperature of the clamshell itself. These thermocouples are installed via spring-loaded thermocouple holders that apply pressure to keep the thermocouple in direct contact with the clamshell surface inside the divot. Only five thermocouples are used even though there are 10 divots available. These thermocouples are designated TE327 through TE331.

The test article tube is placed inside the clamshell with 0.75 inches exposed at the fuel exit and 2 inches exposed at the fuel entrance. These sections allow assembly of the BFA assembly into the ARSFSS rig. A heat-conducting silicone compound similar to that used in mounting microprocessors to metal heat sinks is used to assure good thermal contact between the clamshell and the test article tube. The clamshell is then screwed together around the test article tube with 5 pairs of screws assuring both good thermal contact between test article tube and the clamshell and rigid construction of the test article assembly.

Once assembled, the BFA assembly is mounted vertically in a specially designed holder for the test. During the test, fuel flows into the bottom and out the top of the BFA assembly. Figure 10 also shows the ‘section’ designations used for the test article tube with Section 1 being the fuel inlet and Section 10 being the fuel outlet.

After the test is completed, the entire clamshell assembly is removed from the rig and disassembled. Those sections marked as “DISCARD” in Figure 10 are cut off of the test article tube and discarded. Any residual thermal heat sink compound is removed from the external of the test article tube. The test article is then cut into the Sections shown in Figure 10. Each section is gently rinsed with hexane to remove any remaining liquid fuel residue and then dried overnight in a vacuum oven at 110 °F. Once these sections are dried, they are subjected to destructive carbon analysis by LECO Carbon Analyzer the following day.

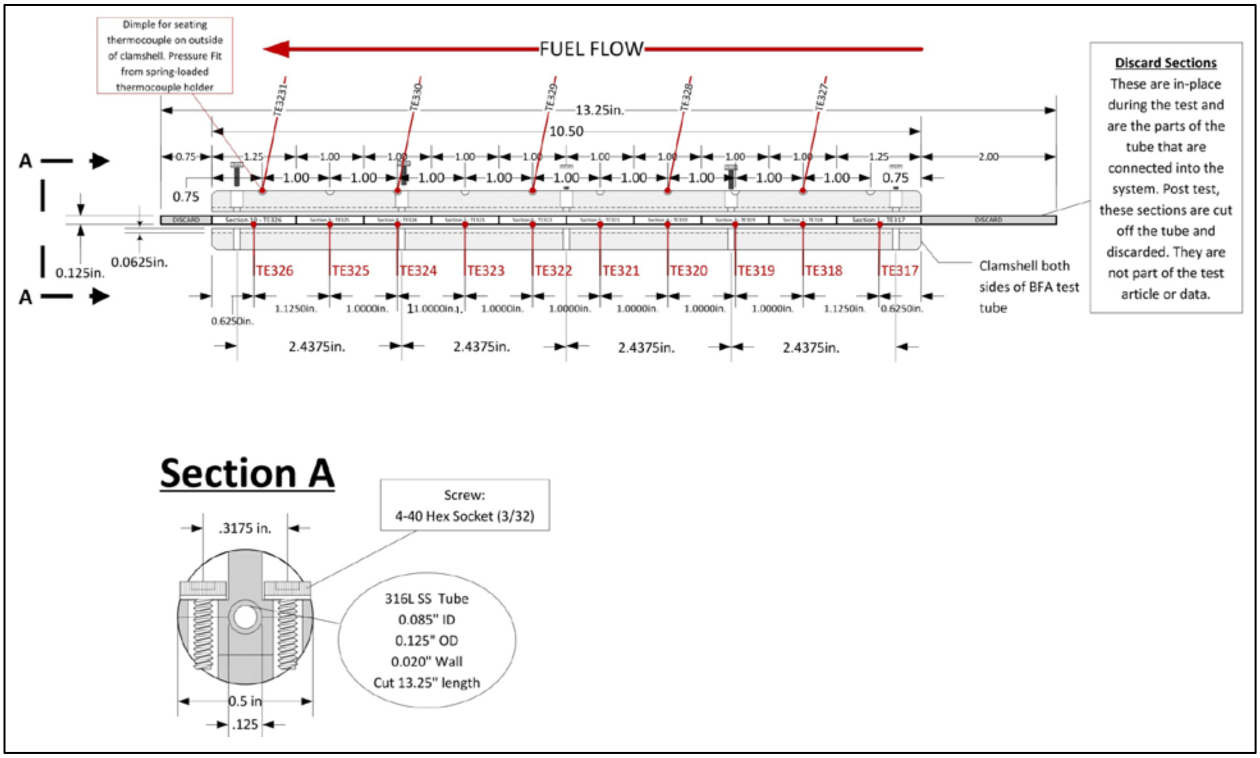


Figure 10 - Burner Feed Arm Design and Thermocouple Placement

2.6 Torque Motor Screen Module

In prior programs, AFRL has worked closely with the University of Sheffield (UK) and Rolls-Royce (UK) to establish operational condition links between the ARSFSS operated by AFRL and the AFTSTU operated by the University of Sheffield. At the request of Rolls-Royce, a new module, the Torque Motor Screen (TMS), was integrated into the ARSFSS configuration. In real-world hardware, the TMS is used to protect sensitive system components from large debris that might interfere with system operation. It is often referred to as a ‘last chance’ filter. This module is used by the University of Sheffield in the AFTSTU unit to look at bulk fuel deposition occurring in areas where the bulk temperature of the fuel is high. The particular screen used for this module is manufactured by Pall as Part No. 20020-250-70 with a part name of “Fluid

Filtering Disk”. It is integrated into the ARSFSS system using a specially-designed holder designed by the University of Sheffield. The University of Sheffield kindly provided design information for the local fabrication of the holder assembly and the assembly of the module for integration into the ARSFSS. Figure 11 shows the TMS module assembled while Figure 12 shows an exploded view of the TMS.

Figure 11 - Torque Motor Screen, Assembled

Figure 12 - Torque Motor Screen Module, Exploded View

2.7 Operation in Real-World Mission Cycle Mode (GDTC)

Since the design and development of the ARSFSS in the mid-1980s, the rig has typically been operated in a mode where mission conditions were established (fuel flow rate, temperatures, pressures) in ‘real time’ and executed in sequence. With the assistance of Pratt & Whitney, a standard mission configuration was developed based on their Generic Durability Test Cycle (GDTC) mission. The GDTC mission was a set of conditions that would be representative of what an advanced aircraft would see in the real world over its functional lifetime. This GDTC profile was modified slightly for research testing on the ARSFSS and it has become the standard operating test mission for the ARSFSS. It consists of 6 mission elements with a total elapsed mission time of 112 minutes. Table 1 shows the conditions for each mission segment as well as the duration of each segment. In GDTC mode, the ARSFSS is typically operated for 65 mission cycles. These cycles are performed back-to-back 24/7. The total duration of a test consisting of 65 mission cycles is approximately 7 days and consumes approximately 900 gallons of fuel.

Figure 13 shows the 'core' and 'recirculation' fuel flow rates for the standard GDTC mission. 'Core' flow is the fuel flow that goes to the combustor and is used for propulsion. 'Recirculation' flow represents the fuel flow that is typically used for thermal management processes.

Table 1 – Typical Generic Durability Test Cycle Mission as Modified for ARSFSS Use

Generic Durability Test Cycle (GDTC) Modified Standard Operating Conditions							
Mission Parameter	**Mission Control Point**	**Mission Segment Number and Name**					
		Ground Idle 1	**Hi Pwr Cruise**	**Lw Pwr Cruise**	**Combat**	**Descent**	**Ground Idle 2**
		1	**2**	**3**	**4**	**5**	**6**
Start Time, El. Min	**N/A**	0	25	46	88	91	97
End Time, El. Min	**N/A**	25	46	88	91	97	112
Duration, Min	**N/A**	25	21	42	3	6	15
Burn Flow, PPH	**4**	16.7	52	35	169.1	23.2	16.7
Recirc Flow, PPH	**3**	27.5	14	23	0	44.2	27.5
FCOC Fuel In, °F	**2**	300	300	300	NC	300	300
FCOC Fuel Out, °F	**3**	325	325	325	NC	325	325
AFHX Fuel Out, °F	**1**	285	285	285	NC	285	285
BFA Max WWT, °F	**4**	500	500	500	NC	500	500
NC = Not Controlled NA = Not Applicable							

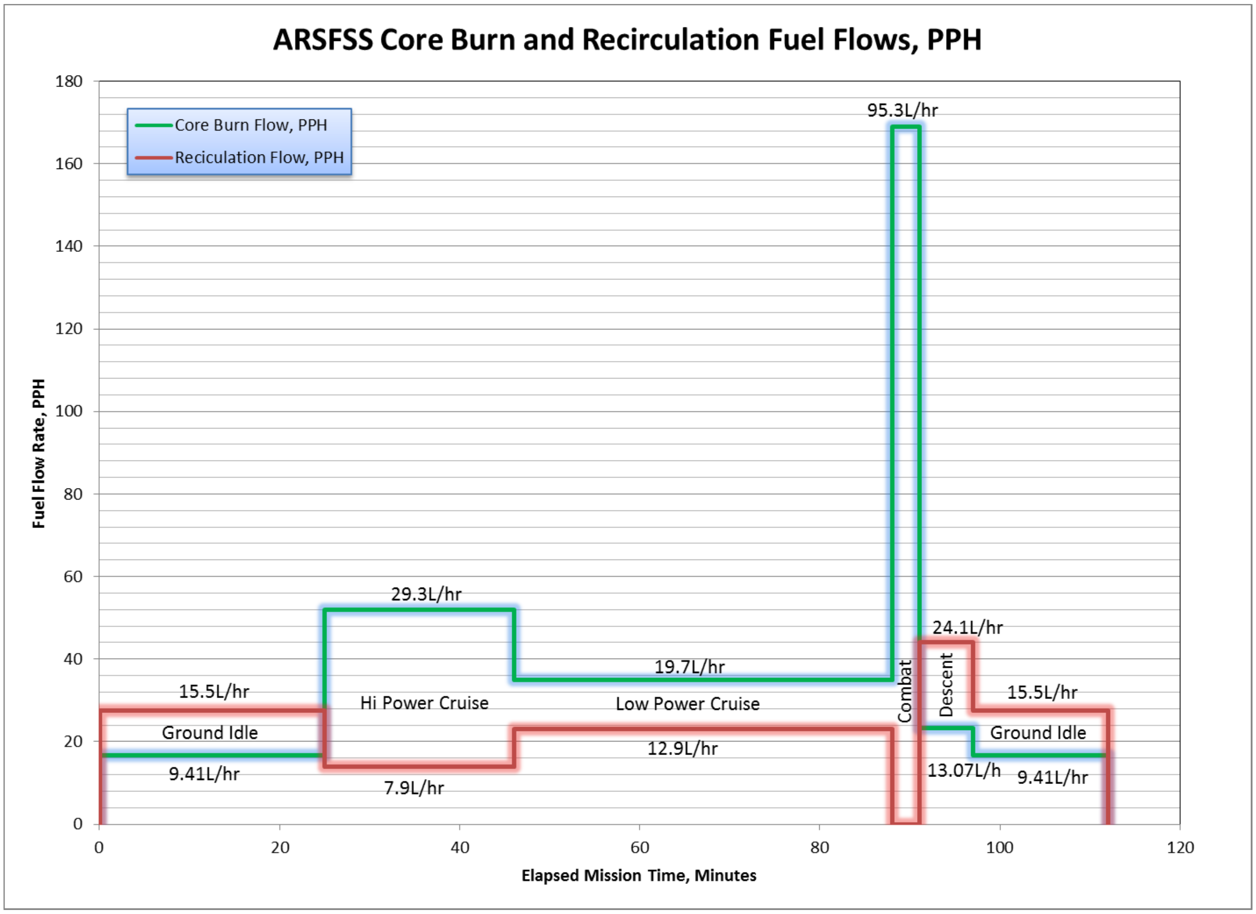


Figure 13 - Recirculation and Burn Flow Rates, Generic Durability Test Cycle Mission

2.8 Operation in Extended Duration Thermal Stability Test (EDTST) Mode

Recently, AFRL has developed a steady-state operational mode that allows for reduced fuel consumption and reduced test times. This mode is adapted from the procedure that was used on a test rig that was operated in the past by AFRL called the Extended Duration Thermal Stability Test. In that test, a specially designed rig was operated at a flow rate of 1 gallon per hour with 1 gallon per hour recirculation. The test duration was 4 days and 100 gallons of fuel was consumed. Many thousands of hours of operation were logged with this test rig but circa 2006 the rig was decommissioned due to a need for facility floor space. Recently, this test mode was re-implemented using the ARSFSS. It has a normal duration of 72 hours and consumes approximately 200 gallons of fuel. It is operated on the ARSFSS in standard ARSFSS configuration at the conditions shown in Table 2. This mode is much more suited to parametric-type studies due to the reduced test time and the reduced fuel consumption.

Table 2 – Standard EDTST-Mode Operating Conditions

EDTST-Mode Test Conditions	
Main 'Burn' Fuel Flow	16.7 pph
Recirculation Flow	27.5 pph
Fuel-Cooled-Oil Cooler Bulk Fuel Inlet Temperature	325 °F (163 °C)
Burner Feed Arm Inlet Bulk Fuel Temperature	375 °F (190 °C)
Burner Feed Arm Maximum Wetted Wall Temperature	510 °F (265 °C)

As testing proceeded in this program, due to the nature of the fuel used in this program, the EDTST-mode operating conditions were modified slightly to lower the temperatures in the FCOC and BFA. Table 3 represents the final EDTST-mode operating conditions established for this program. See Section 5.2.1 for additional details.

Table 3 – Revised EDTST-Mode Operating Conditions

Revised EDTST-Mode Test Conditions	
Main 'Burn' Fuel Flow	16.7 pph
Recirculation Flow	27.5 pph
Fuel-Cooled-Oil Cooler Bulk Fuel Inlet Temperature	300 °F
Burner Feed Arm Inlet Bulk Fuel Temperature	325 °F
Burner Feed Arm Maximum Wetted Wall Temperature	510 °F (265 °C)

2.9 Data Obtained From The ARSFSS and It's Subcomponents

The ARSFSS test rig is a complex rig that simulates the entire fuel system for an advanced aircraft system – both engine and airframe. Unlike small bench-scale tests, results obtained from the ARSFSS are not limited to one specific value or finding. The ARSFSS can be compared to an assemblage of smaller test articles. This means that assessing the results of a single test run involves assessing the data obtained from these various system test articles and then formulating an assessment of the test overall from these individual assessments.

There are several main subcomponents in the ARSFSS that are evaluated for each Run. Table 4 shows a listing of the subcomponents evaluated and the data that is collected from each subcomponent.

Table 4 – Data Collection and ARSRFSS Subcomponents

ARSFSS Components and Data Collection/Analsys Performed	
Component	**Data Collected**
Fuel-Cooled Oil Cooler	Maximum Wetted Wall Temperature Trend
	Wetted Wall Temperature Profile Along Tube
	Carbon Deposition (LECO)
	Pressure Drop
Servo Valve (Recycle)	Flow Hysteresis
	Visual Inspection of Internal Components
Burner Feed Arm	Maximum Wetted Wall Temperature Trend
	Wetted Wall Temperature Profile Along Tube
	Carbon Deposition (LECO)
	Pressure Drop
Flow Divider Valve	Flow Hysteresis
	Visual Inspection of Internal Components
Torque Motor Screen	Total Accumulated Mass
	Carbon Deposition (LECO)
	Pressure Drop

2.10 ARSFSS Behavior During an Emergency or Unscheduled Shutdown

The ARSFSS is programmed to operate 24/7 with minimal Operator intervention. It operates unattended 16 hours each week day and 24 hours each day on weekends. During extended test programs, it is not uncommon for some anomaly to occur which results in a shutdown of some sort. This inevitability was accounted for in the overall design and implementation of a control strategy for the ARSFSS. As a result, four potential overall shutdown conditions were anticipated and therefore four different shutdown sequences were developed. Each sequence is specifically designed to protect test integrity by reducing, as quickly as possible, the test article temperatures while maintaining some critical fuel flows. The four shutdown modes are Normal Shutdown, End-of-Cycle Shutdown, Mid-Cycle Shutdown, and Emergency Shutdown. Each shutdown mode is triggered by specific events.

A Normal Shutdown is executed at the end of a programmed sequence of mission cycles. This involves a controlled shutdown involving turning off heaters, maintaining fuel flows until temperatures in critical areas of the rig (those areas most likely to affect thermal stability determinations) are below established limits and then a final power-off.

An End-of-Cycle Shutdown is executed when the rig detects an anomaly that is not immediately detrimental to rig or test integrity but still needs Operator intervention prior to continuing the overall program. In the End-Of-Cycle Shutdown, the rig is allowed to complete the currently active mission normally and then at the end of that mission, a Normal Shutdown is executed. If at any time during the balance of the continued mission the rig detects further anomalies that would be more critical than those that triggered the End-Of-Cycle Shutdown initially, a Mid-Cycle Shutdown is immediately executed.

A Mid-cycle shutdown is executed when the rig detects an anomaly that could potentially impact the overall integrity of the rig and data if not immediately dealt with. In the Mid-Cycle Shutdown, no matter what part of the mission cycle is being executed, the mission is terminated in a controlled fashion by immediately executing a Normal Shutdown.

An Emergency Shutdown is executed only rarely and is triggered by the detection of no or very low flow anywhere on the rig. An Emergency Shutdown can be executed at any time – either during a mission or during one of the other shutdown sequences. In an Emergency Shutdown, the rig is immediately powered down no matter what the mission cycle or mission condition. This shutdown is executed when there is a risk that low or no fuel flow can irrevocably damage major rig hardware such as gear pumps.

There is always the possibility of a shutdown due to a utility-based power failure. In this case, the rig will shut down immediately. No control is possible during this type of shutdown but the hardware on the rig is designed so that in the event of a power outage, all rig components fail safe. Since no control is possible during this type of shutdown, it is possible that the test integrity will be compromised. To determine if the test is compromised, battery backup is provided on critical computer and I/O systems. This allows data to be collected during the shutdown and for this data to later be assessed to determine if the test integrity was indeed compromised.

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3.0 PROGRAM GOALS AND OBJECTIVES

The overall goal of this program is to evaluate the Aquarius additive using the Advanced Reduced Scale Fuel System Simulator (ARSFSS) to determine if there is any impact on the thermal stability characteristics of a typical Jet A under actual aircraft fuel system conditions. Jet A will be evaluated in this program because it is the fuel used by commercial and private aircraft. However, the US Air Force is currently transitioning from JP-8 (NATO F-34) to Jet A with FSII, CI/LI and SDA (NATO F-24) as the standard fuel for flight and ground systems operation in CONUS. Therefore the data obtained in this study will be of interest to military as well as commercial and private aircraft operators.

In this program, the Aquarius additive will be evaluated in the ARSFSS using both Extended Duration Thermal Stability Test (EDTST) and Generic Durability Test Cycle protocols. The EDTST protocol is a simplified protocol where a fixed set of steady-state conditions is imposed on the ARSFSS at a fixed fuel flow rate. The test is run at these steady state conditions for 72 hours.

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4.0 EXPERIMENTAL

4.1 Test Plan Overview

The overall test plan is summarized in Table 5. It is divided into three phases - Phase I (Runs AQ-1 through AQ-4), Phase II (Runs AQ-5 through AQ-8) and Post Test (AQ-9 – AQ-10). In addition to these three Phases, some pre-test work was accomplished. Initially, only Runs AQ-1 through AQ-10 (Runs 125-134) were planned. As the program proceeded, an additional Run was added to accommodate questions for engine OEMs during the program. That additional Run is shown in Table 5 as Run AQ-11 (Run 135).

Table 5 - Test Plan Run Details

AQUARIUS ADDITIVE EVALUATION TEST PLAN TABLE																						
	Run No.		Test Description	Test Type	Missions[1]	Fuel Type	Fuel Qty (gal)	AQUARIUS ml/m³	FCOC Bulk Inlet °F	BFA Bulk Inlet °F	BFA Max WWT °F	JFTOT Breakpoint		QCM		Spec Test		ICP-MS		Karl Fischer Water		Notes
												BL	Add	BL	Add	BL	Add	BL	Add	BL	Add	
Basic Effect of Aquarius Additive																						
PRE-TEST	PreTest		Baseline Jet A as Received									1		2		1		1		1		Evaluate baseline and additized fuel to 1) verify base fuel as received meets spec and 2) to establish baseline thermal stability characteristics for both baseline and additized fuels
PRE-TEST	PreTest		Baseline Jet A As Received + 4X Aquarius										1		2		1		1		1	
PHASE I	AQ-1	125	Baseline Jet A	ED	N/A	Jet A	275	0	300	325	510									2		See Note 2
PHASE I	AQ-2	126	Baseline Jet A	ED	N/A	Jet A	275	0	300	325	510									2		See Note 2
PHASE I	AQ-3	127	Jet A + 4X Aquarius	ED	N/A	Jet A	275	1000	300	325	510										2	See Note 2
PHASE I	AQ-4	128	Jet A + 4X Aquarius	ED	N/A	Jet A	275	1000	300	325	510	1	1	1	1			1	1		2	QCM, ICP-MS and JFTOT on BOTH baseline and Aquarius-additized fuels; See Note 2
Review			*Review data before proceeding to additive testing. Telecon review of data*																			
PHASE II	AQ-5	129	Run Baseline Jet A	GDTC	65	Jet A	900	0	300	325	500									2		See Note 2
PHASE II	AQ-6	130	Run Baseline Jet A	GDTC	65	Jet A	900	0	300	325	500									2		See Note 2
PHASE II	AQ-7	131	Run Jet A + 4X Aquarius	GDTC	65	Jet A	900	1000	300	325	500										2	See Note 2
PHASE II	AQ-8	132	Run Jet A + 4X Aquarius	GDTC	65	Jet A	900	1000	300	325	500										2	See Note 2
POST-TEST	AQ-9	133	Jet A + 4X Aquarius Retest	ED	N/A	Jet A	275	1000	300	325	510		1		1			1	1		2	Re-evaluate Baseline and Additized fuel in EDTST mode to determine magnitude (if any) of changes over time.
POST-TEST	AQ-10	134	Baseline Jet A Retest	ED	N/A	Jet A	275	0	300	325	510	1		1		1				2		See Note 2 QCM, ICP-MS and JFTOT on BOTH baseline and Aquarius-additized fuels.
	AQ-11	135	Jet A + 250 ppm Aquarius + 200 ppm total water	ED	N/A	Jet A	275	250	300	325	510		1		1		1		1		1	Evaluate AQ-additized fuel at 250 ppm Aquarius with 200 ppm total dissolved water to simulate full operational experience.
	Review		*Review data and assess impact of Aquarius.*																			
			Minimum Fuel Required ===>				5,525					2	3	3	4	1	1	3	3	9	11	
	BL = Baseline Fuel; ADD = Aquarius-Additized Fuel										TOTALS	5		7		2		6		20		

Notes:
1. Typically, 65 missions will be used based on the Generic Durability Test Cycle mission profile normally used for the ARSFSS. However, the number of missions may be increased as needed.
2. Water by Karl Fischer: 2 samples each Run - one from fuel farm tank being used and one sampled off-ARSFSS (downstream of BFA, towards end of Run) for comparison

4.2 Fuel Tankage Preparation

The ARSFSS was operated from Tank S-3 when running baseline fuel tests without Aquarius additive. For runs requiring additized fuel, Tank S-4 was used. In preparation for this program, Tank S-3 was rinsed with the program Jet A fuel and sumped to remove any residual fuel and water. Tank entry and manual cleaning was not required because this tank contained only a typical Jet A from a previous program. However, tank S-4 had potential contamination from a prior program so it was cleaned by a professional tank cleaning company. Tank S-4 was cleaned by spraying the inside of the tank with hot water/steam. The tank was entered and the walls were 'squeegeed' and wiped dry using lint-free cloths. The lines to and from the tank were cleaned using hot water. These line were then air-blown dry. This is the standard procedure used for all AFRL fuels work requiring clean tanks.

4.3 Fuel Requirements and Preparation

Fuel requirements for this sequence of testing were estimated to be a minimum of 5525 gallons based on the Table 1 test plan sequence. However, past experience has shown significant merit to having some extra fuel on hand so 7,100 gallons (26,876 liters) were obtained for this testing. The baseline Jet A for this program was acquired by UDRI from World Fuel Services from a pipeline terminal in Cleveland, Ohio (Sunoco Logistics). The source of the fuel was the Toledo Refining Company, Toledo, Ohio. According to the terminal operator, this fuel was clay-filtered using a 3-stage process before being loaded into the truck. The fuel was delivered to the S-Farm in one truck-load. Certificates of Analysis and shipping/receipt documentation are provided in Appendix A. The fuel sample code assigned to this bulk fuel sample was POSF-11769.

4.4 Impact of Clay Filtration on the Baseline Fuel Selection

It is important to note that clay-treatment is used extensively in the US to deal mainly with jet fuels derived from a multi-product pipeline. They appear at every break-out terminal where the fuels are separated into their respective storage tanks. Clay treatment is needed to assure that additives from other fuels do not make it through to the dedicated jet supply system downstream of the break-out location. Thus, clay treatment is an integral part of the supply chain of US pipeline jet fuels.

Clay is particularly good at adsorbing (removing) polar organic molecules such as Corrosion Inhibitor – Lubricity Improver (CILI), Static Dissipator Additive (SDA), and other additives. It is ineffective at removing inorganic ions - only water can do that. For this reason the ICP analysis of a fuel pre- and post-clay filtration commonly indicates no change in the load of elements in the fuel - ICP would be unchanged if you compared upstream and downstream samples across a clay filter. For further information see EI1550, Annex F - also CRC Aviation Fuels Handbook, IATA Guidance, JIG.

4.5 Pre-Test Analyses

Upon delivery, the fuel was loaded into three tanks on the S-Farm – S-3 (850 gallons), S-4 (850 gallons) and S-16 (5,400 gallons). The fuel lines routing fuel from S-16 to S-3 and S-4 were cleared with compressed air to make sure all fuel from previous programs was removed. Two 5-gallon retainer samples were collected from the truck at the time of delivery. Another two 5-gallon retainer samples were pulled from the in-ground tank into which the delivered fuel was loaded (tank S-16). Tanks S-3 and S-4 were not sampled. Another two-5-gallon samples were drawn from S-16 and this fuel was distributed to UDRI and AFPA for analysis (full specification testing, JFTOT Breakpoint, QCM, ICP-MS and Water (by Karl Fischer, W_{KF}). See Table 7 for specific specification test results.

In addition to normal visual tube deposit rating techniques and ratings, the tubes from these initial tests were subjected to Ellipsometer measurements. Table 6 and Figures 14 through 17 show the data from these evaluations.

Table 6 - Ellipsmeter Ratings of JFTOT Tubes

Date	S/N	Fuel Desc.	POSF No.	D3241 Temp (C)	VTR	Max Avg Deposit Thickness (nm)
7/1/2014	LHAAD170	Jet A	11769	275	<3	98
7/1/2014	LHEAD114	Jet A	11769	280	1A	62
7/1/2014	LHCAD238	Jet A	11769	285	2A	58
7/18/2014	LEaAC169	Jet A	11769	270	1	8
7/18/2014	LCKAA023	Jet A	11769	275	1	14
7/18/2014	LCKAA005	Jet A	11769	280	1	24
7/18/2014	LEaAC351	Jet A	11769	285	2A	68
7/1/2014	LHBAD243	Jet A w/ Aquarius	11770	280	1	11
7/1/2014	LGbAD239	Jet A w/ Aquarius	11770	285	2A	65

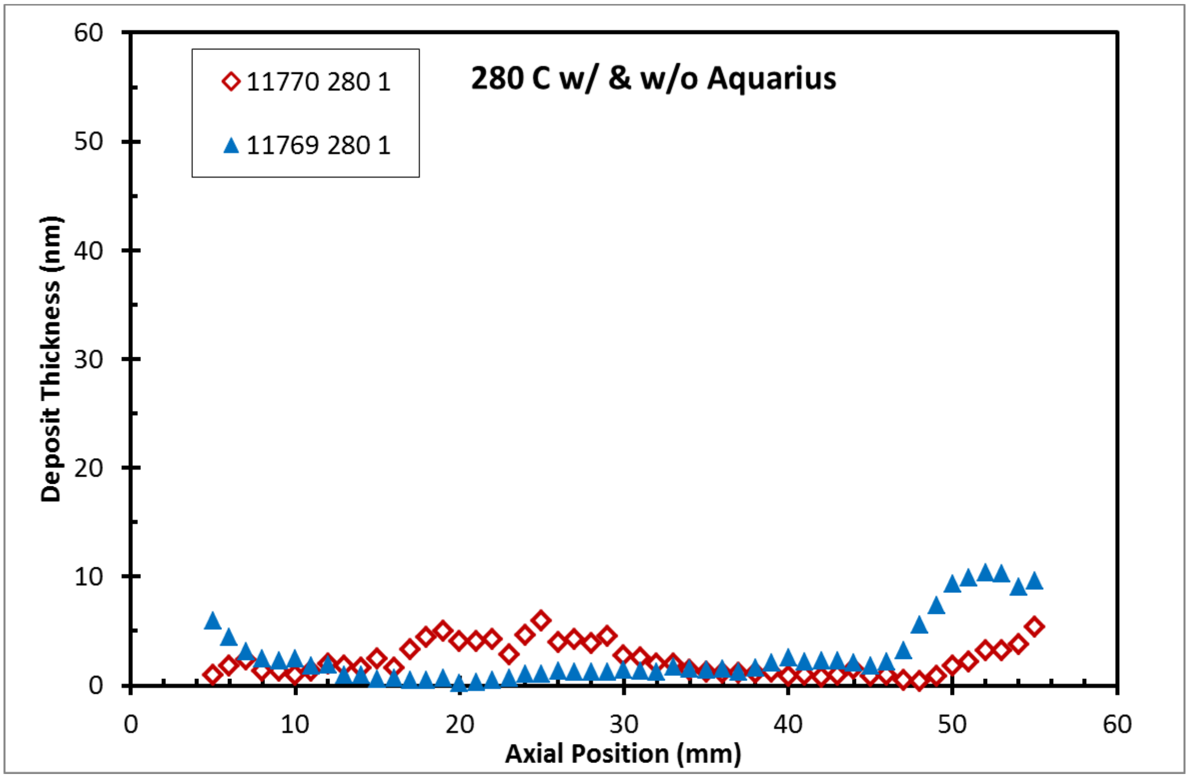


Figure 14 – Ellipsometer Data – 280 °C With and Without Aquarius Additive

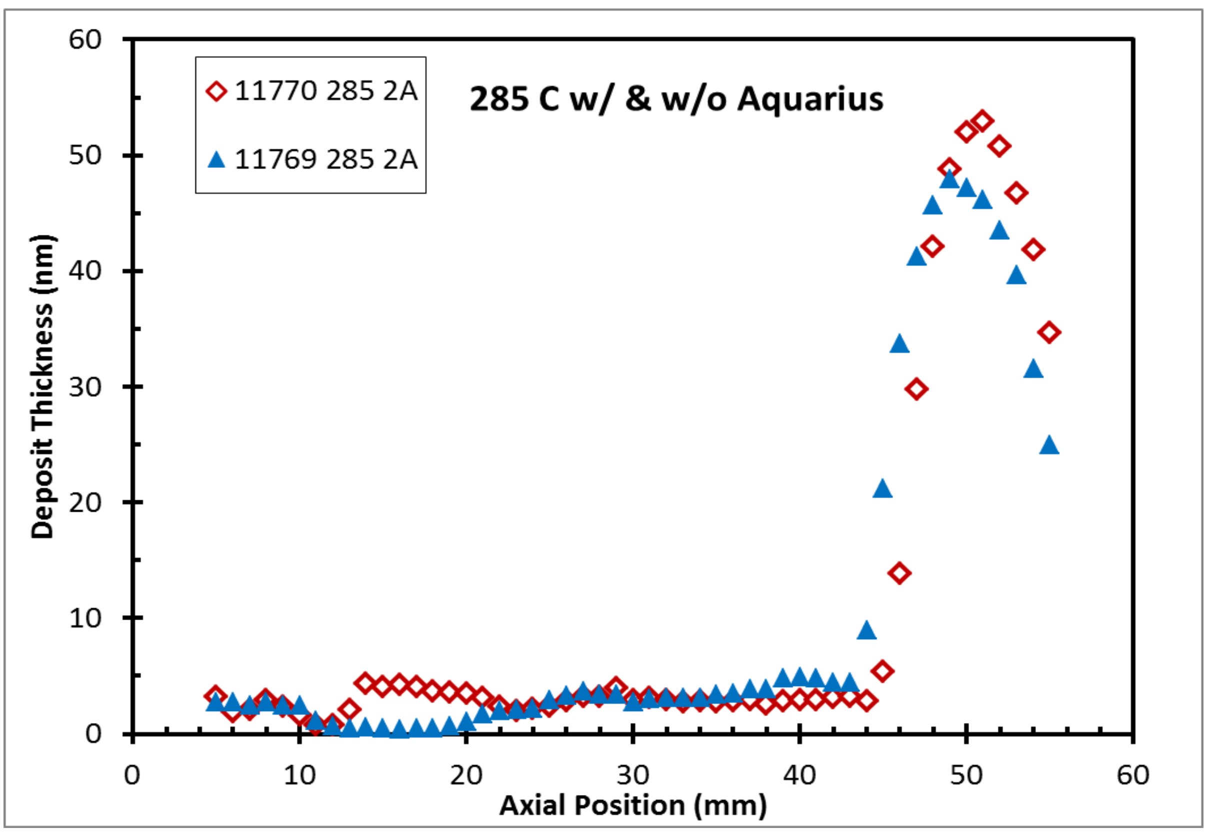


Figure 15 – Ellipsometer Data – 280 °C With and Without Aquarius Additive

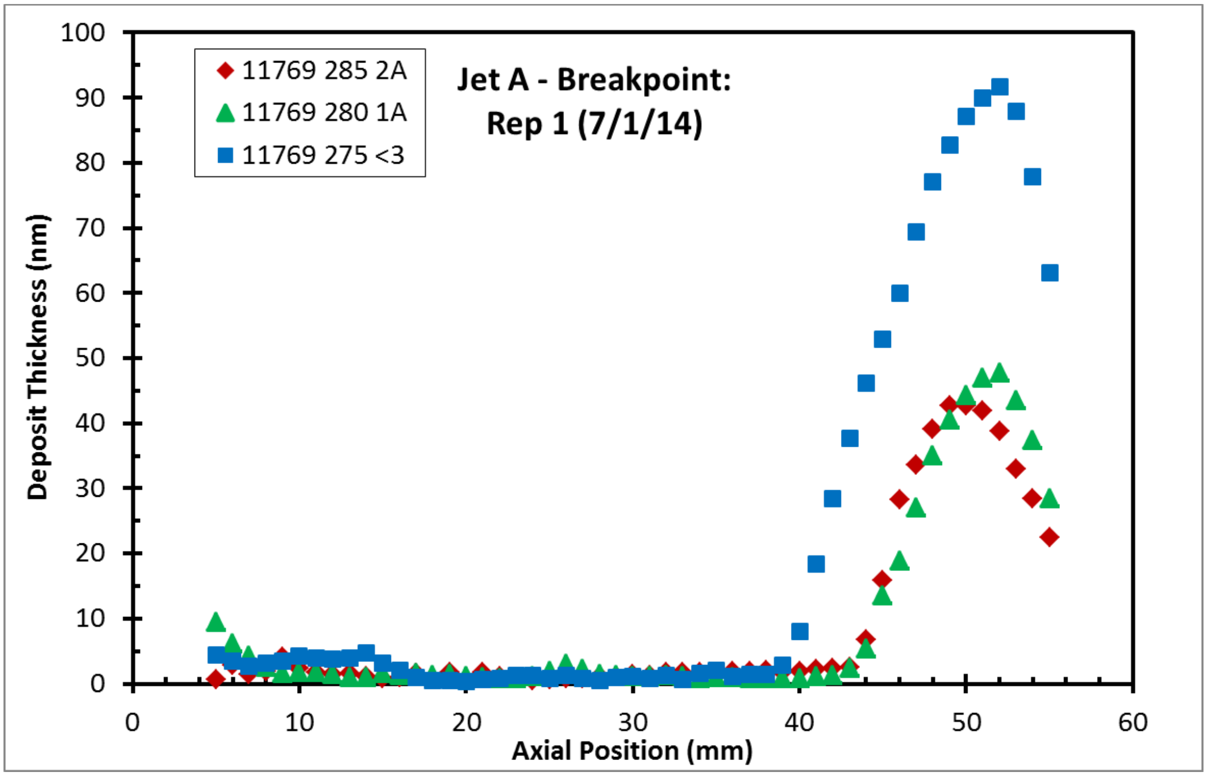


Figure 16 – Jet A Breakpoint Determination (7/1/14)

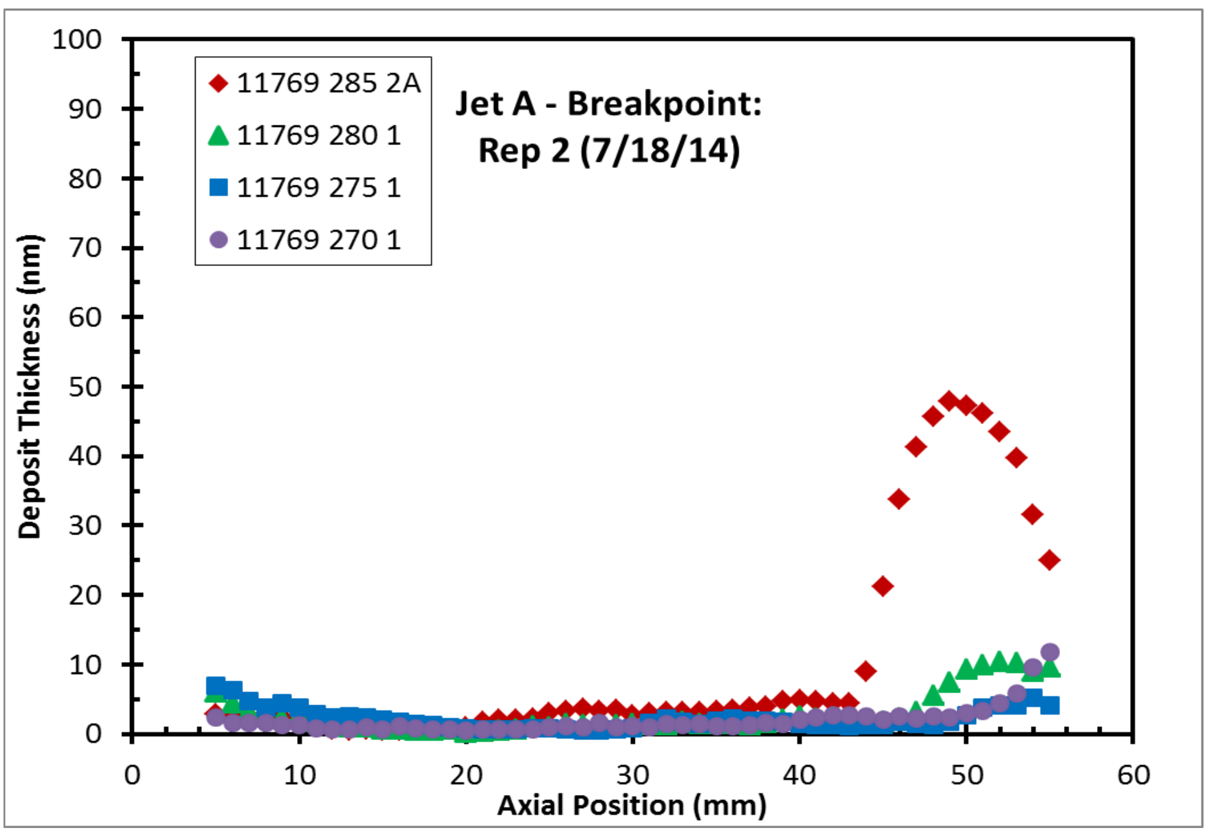


Figure 17 – Jet A Breakpoint Determination (7/18/14)

4.5.1 Fuel Thermal Stability:

It was known that the testing for this program would take several weeks and there was some concern that the Jet A thermal stability might change over that time because Jet A is not typically treated with an antioxidant additive[1]. This concern was born out of the experience of researchers at the University of Sheffield using their Aviation Fuel Thermal Stability Test Unit (AFTSTU) as well as the Air Force's own experience in a similar program where Jet A was used over a period of months and experienced a 10°C drop in breakpoint temperature. Therefore, throughout the program JFTOT and QCM testing was performed periodically to track any change in fuel thermal stability (See the test plan in Table 5). These results are documented in various places in this report. However, Table 8 shows the results of JFTOT Breakpoint evaluation of the fuel throughout the program. JFTOT testing indicates a breakpoint of 280 °C for the baseline fuel and as high as 300 °C for the Aquarius additized fuel. Figures 18 through 20 in this section document the QCM results obtained during this program.

[1] *Antioxidant (AO) "shall be added to a fuel (or component) which has been hydroprocessed (i.e. manufactured using a catalytic hydrogen process such as hydrotreating, hydrofining, hydrocracking, etc)…" (Ministry of Defense, Defense Standard 91-91, Issue 7 Publication Date 18 February 2011 (Note: Amendment 2 Implementation date 01 March 2013))*

Table 7 - Fuel Analyses Results

Method	Test	Min	Max	POSF-11769		POSF-11769		POSF-11769		POSF-11769		POSF-11770		POSF-11770		POSF-11770	
				14-Jun-14		30-Jun-14		14-Oct-14		14-Oct-14		1-Jul-14		29-Jul-14		20-Oct-14	
				Baseline Jet A		Baseline Jet A		Baseline Jet A		Baseline Jet A		1000 ppm AQ		1000 ppm AQ		1000 ppm AQ	
				Cert of Analysis		S-16		S-16, Top		S-16, Bottom		Tank S-4		Tank S-4		Tank S-4 Run 133	
				Result	Fail	Result	Fail	Result	Fail	Result	Fail	Result	Fail	Result	Fail	Result	Fail
MIL-STD-3004C(1)	Appearance			1				Pass		Pass						Pass	
ASTM D 3242 - 11	Total Acid Number (mg KOH/g)		0.1	0		0.001		0.001		0.001		0.001		0.002		0.001	
ASTM D 1319 - 13	Aromatics (% vol)		25	20				20.6		22.7				21		21	
ASTM D 3227 - 04a	Mercaptan Sulfur (% mass)		0.003	0		0		0		0		0		0		0	
ASTM D 4294 - 10	Total Sulfur (% mass)		0.3	0				0.01		0.01				0.01		0.01	
ASTM D 86 - 12	Distillation																
	10% Recovered (°C) 205 176		205	170		172		172		171		171		171		171	
	20% Recovered (°C) Report Only 184	Report		178.9		180		179		180		179		179		179	
	50% Recovered (°C) Report Only 205	Report		200		202		201		201		202		200		201	
	90% Recovered (°C) Report Only 244	Report		237.2		243		242		242		243		240		242	
	End Point (°C) 300 269		300	259.4		265		266		266		266		264		264	
	Residue (% vol) 1.5 1.2		1.5	1		1.4		1.2		1.2		1.4		1.2		1.4	
	Loss (% vol) 1.5 0.2		1.5	0.5		1.1		0.6		0.7		0.9		0.6		0.4	
ASTM D 56 - 05	Flash Point (°C)	38		45		45		45		46		43		44		43	
ASTM D 4052 - 11	Density @ 15°C (kg/m³)	775	840			808		812		812		808		812		812	
ASTM D 5972 - 05e1	Freezing Point (°C)		-40	-55.3		-55.4		-56		-56		-54.9		-41 (Note A)		-55	
ASTM D 445 - 12	Viscosity @ -20°C (mm²/s)		8	4.1*				4.6		4.2				4.2		4.3	
ASTM D 3338 - 08	Net Heat of Combustion (MJ/kg)	42.8		18535**				43.1		43.1				43.1		43.1	
ASTM D 1322 - 12e1	Smoke Point																
	Smoke Point (w/allowable Naphthalenes) (mm)	18		20				21		19				22		20	
ASTM D 1840 - 07	Naphthalenes (% vol)		3.0	0.7				0.7		0.7				0.7		0.7	
ASTM D 130 - 12	Copper Strip Corrosion (2 h @ 100°C)		1	1A		1a		1a		1a		1a		1a		1a	
ASTM D 3241 - 13	Thermal Stability @ 260°C																
	Change in Pressure (mmHg)		25					0		0							
	Tube Deposit Rating, Visual		<3 (Max)					1		1							
ASTM D 381 - 12	Existent Gum (mg/100 mL)		7	0				<1		<1				3		2	
ASTM D 1094 - 07	Water Reaction Interface Rating 1b (Max)		1b	1		1						4		4		2	
ASTM D 3948 - 11	WSIM	70						91		93				0		0	
ASTM D 5006 - 11	FSII (% vol) Report Only	Report				0		0		0		0.02		0.02		0	
ASTM D 2624 - 09	Conductivity (pS/m)	50	600	0		4		0		0		4		0		0	
ASTM D 5001 - 10	Lubricity Test (BOCLE) Wear Scar (mm) Report Only	Report						0.66						0.58		0.57	
MIL-DTL-83133H Amnd2	Filtration Time (min)			6										16			
ASTM D 7171 - 05	Hydrogen Content by NMR (% mass)															13.6	
ASTM D 1319 - 13	Olefins (% vol)																
ASTM D 4809 - 13	Particulate Matter ((mg/L)			0										1.1			
ASTM D 2532 - 03	Viscosity @ -40 °C																
	@ 35 min (cSt)	Report Only															
	@ 3 hrs (cSt)	Report Only															
	@ 72 hrs (cSt)	Report Only															
ASTM D 445 - 12	Viscosity @ -40 °C (cSt)																
	Total Water, Karl Fischer, ppm			58		61.5											

N/M = Not Measured | * @ -4 °F, Cs; ** BTU/lb;

Notes:

A. This data value believe to be in error based on technical issues with the test method

Table 8 - JFTOT Breakpoint Results on Program Fuel Samples

JFTOT Breakpoint Determinations				
Sample Date	Fuel Code	Baseline Fuel BP, °C	Additized Fuel BP, °C	Source
30-Jun-14	11769	280		S-16
1-Jul-14	11770		280	Lab Sample
18-Jul-14	11769	280		S-16
29-Jul-14	11770		300	S-4

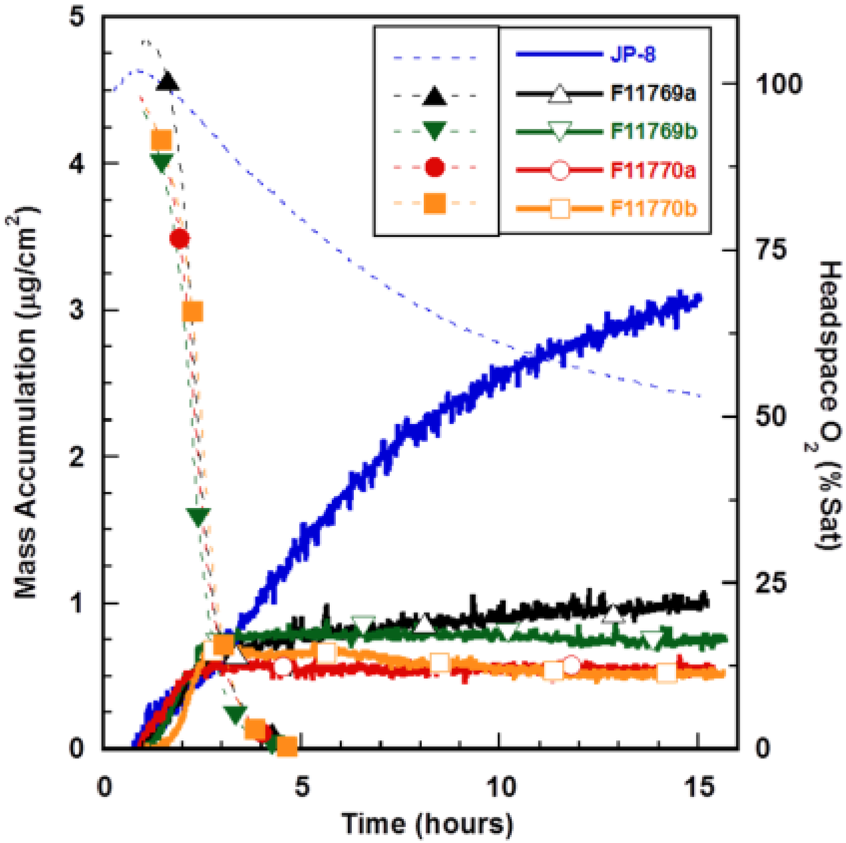


Figure 18 - Results of QCM Analysis

The Thermal stability characteristic of the as-delivered fuel, 11769, was assessed with and without the addition of Aquarius additive using a quartz crystal microbalance (QCM) apparatus. The experiment was conducted by placing 60 mL of sample into a batch reactor. The samples were air saturated under room conditions, The reactor was then closed and heated to 140°C. Measurements of headspace oxygen, temperature, pressure, and mass accumulation were recorded, while the sample was reacted isothermally for 15 hours. The objective was to investigate the oxidation and mass deposition characteristics of the sample under typical QCM conditions in an effort to identify any abnormalities in thermal stability behavior. Figure 18 shows the headspace oxygen (dotted lines) and mass accumulation profiles (solid lines) of replicate runs of Jet A sample F11769 and the Jet A with addition of the Aquarius additive at 0.1%v (sample F11770); a typical JP-8 fuel is shown for comparison (Blue). These tests show good repeatability between two identical runs, giving confidence in the measured values. The Jet A samples all exhibit fast oxidation, with oxygen being completely consumed after about 4 hours of thermal stress duration. Total deposition levels are very low, <1.0 µg/cm2, for all Jet A sample replicates. These data show no negative impact on thermal stability, under the experimental conditions, due to the addition of Aquarius additive at 0.1%v.

Since the deposition and oxidation curves (Figure 18) for the 11769 baseline fuel were so significantly different than for typical JP-8, the baseline fuel was additized with the standard military package of additives, effectively converting 11769 into NATO F-24. The military package of additives included FSII, SDA, & CI/LI. The sample ID for this additized fuel was designated as 11779. The fuel was rerun in the QCM and the results are presented in Figure 19. As Figure 19 shows, the addition of the military additives had no effect on either oxidation or deposition.

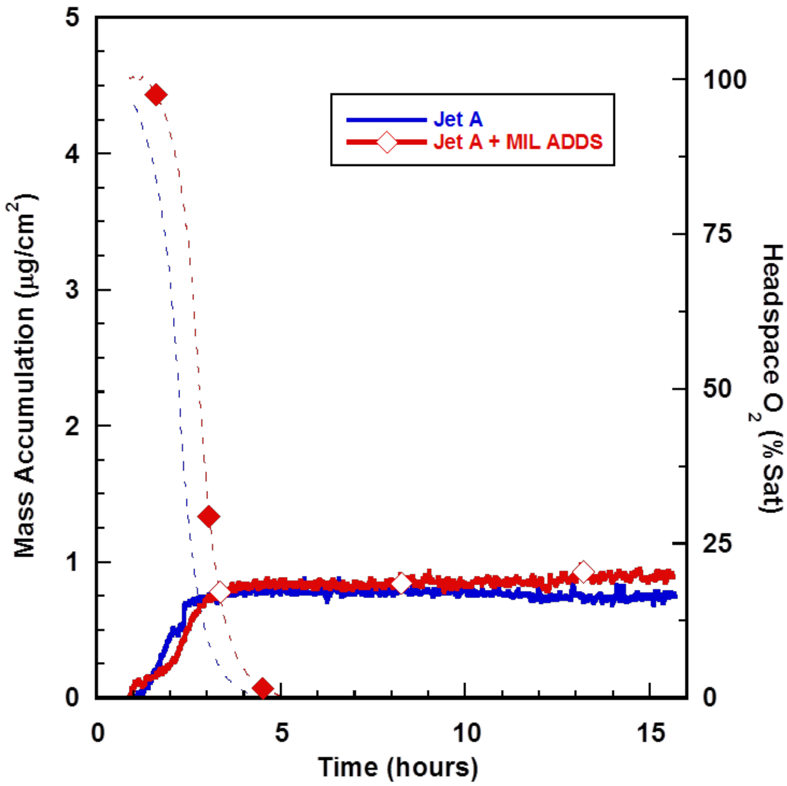


Figure 19 - QCM profiles of deposit (solid lines, open markers) and headspace oxygen (dashed lines, closed markers) at 140°C.

As described in later sections of this report, QCM tests were performed on these same baseline and additized fuel at the close of the program to evaluate any deterioration of the fuel thermal stability characteristics from the start to the end of the program that might skew the data and findings. The results of this final QCM assessment are presented in Figure 20. This figure shows QCM profiles of deposit (solid lines, open markers) and headspace oxygen (dashed lines, closed markers) at 140°C for Jet A fuel samples with and without Aquarius additive at various concentrations; fuel samples from October-2014. Also shown in this figure are the result for a fuel (F11829) that contained 250 ppm Aquarius and a total of 200 ppm dissolved water. This fuel sample is more fully described in Section 5.2.12 later in this report.

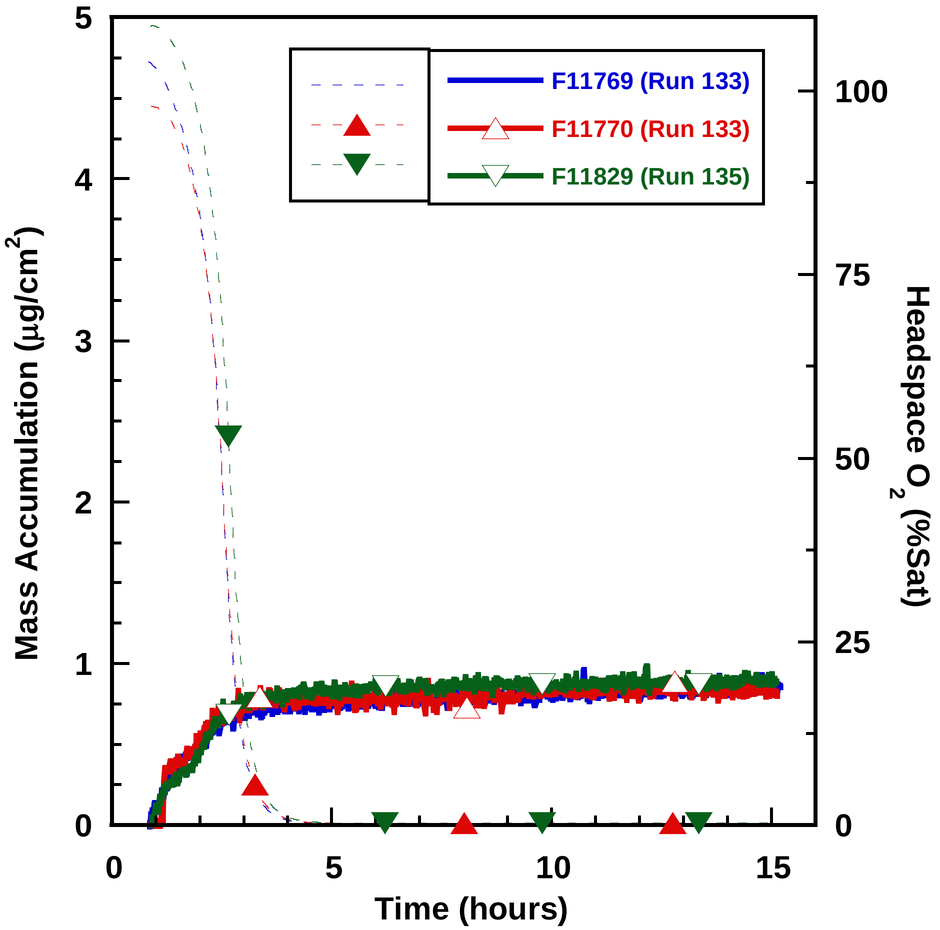


Figure 20 - End of Program QCM Evaluation of Program Fuels

This figure coupled with earlier figures clearly demonstrate that the fuel thermal stability characteristics did not deteriorate or otherwise change over the course of the program.

4.5.2 Quantifying the Amount of FAME in the Fuel

Since the program fuel was delivered by pipeline, analyses were undertaken to see if there had been any FAME contamination by the pipeline. Quantifying the amount of FAME in the program fuel was performed via the use of an Agilent 7890/5975 gas chromatograph/ mass spectrometer (GC/MS). The GC column was a 30-meter DB-5MS capillary column (0.25mm ID and 0.25um film). The GC temperature program employed an initial temperature of 40°C (0.5-minute hold) followed by ramping (20°C/min) to 300°C (5-minute hold). A constant column flow rate of 1 mL/min, and splitless 1-uL injections were used. The GC injector temperature was 275°C, and the Agilent Model 5975 mass spectrometer transfer line was held at a temperature of

280°C. The mass selective detector was operated in selected ion monitoring mode (SIM), and was only turned on where the compounds of interest eluted to protect the detector from the high concentrations of other fuel components. Mass spectral data was recorded for characteristic masses of the compounds of interest (i.e. 74, 67, and 55 for palmitic, linoleic, oleic, and stearic acid methyl esters; and 85 for the tetracosane internal standard).

A minimum of four standard solutions containing FAME and the internal standard (tetracosane – C24H50) were prepared in FAME-free Jet A (POSF-9326) fuel diluted at the same ratio in hexanes as the samples (1 to 5) and analyzed. The standard concentrations at 0, 5, 8, 16, and 20 ppm, rather narrowly bracketed the expected sample concentration in order to more accurately quantify in the low concentration range. The instrument was calibrated from the extracted ion area responses obtained for the four major FAME components and internal standard at each calibration level. Samples of fuel were diluted 1 to 5 with hexanes and the tetracosane internal standard was added. The FAME concentration in each sample was quantified using the extracted ion responses for the four FAME components, and tetracosane in the sample. Results of the analysis showed the program fuel to contain less than 3ppm FAME. It should be noted that the method employed will give a result of <3 ppm FAME even for a sample actually containing ZERO ppm FAME due to the accuracy of the method.

4.5.3 <u>Approval Of The Baseline Fuel</u>

Once the quality of the baseline fuel was documented, the analysis data was collected and reviewed with the Program Sponsor and the major engine OEMs in a teleconference on 10 July 2014. Data was reviewed and AFRL's recommendation to proceed with the program using this fuel was reviewed and accepted.

After the 10 July 14 conference and subsequent discovery that the baseline fuel had been clay filtered at the terminal, this information was relayed to the OEMs with a rationale and recommendation to continue with the program with this fuel (see Section 4.4 above). The general consensus of the OEMs was in agreement with the Aquarius team's assessment and the Program Sponsor authorized continuation of the program as proposed.

4.5.4 *Additized Fuel Preparation*

Having obtained Program Sponsor and engine OEM approval to use the fuel, the fuel in Tank S-4 was additized with the Aquarius additive to the requisite 1,000 ppm. Prior to adding the Aquarius additive, the fuel in S-4 was sampled and water measured (W_{KF}). Results indicated total water in the fuel was 61 ppm. After adding the Aquarius material, fuel in the tank was recirculated for 2 hours to assure the additive was well mixed into the fuel. Two 5-gallon samples were then drawn and submitted for analysis (full specification testing, JFTOT Breakpoint, QCM, ICP-MS and Water (by Karl Fischer,W_{KF}).

4.5.5 *Dissolved Water Measurements*

Throughout the testing in this program, dissolved water measurements were made on the fuel samples taken at various times and locations at the direction of the Air Force Program Manager. This data is presented in Table 9.

Table 9 - Summary of all Dissolved Water Measurements

Water Analysis (by Karl Fischer) By Sample Location					
Sample Date	Run No.	Aquarius Additive ppm	Tank S-16 ppm	Run Tank (S-3 or S-4) ppm	Off-Rig Sample ppm
14-Jul-14	124	0	N/A	N/A	70.2
21-Jul-14	125	0	62.9	55.3	63.4
29-Jul-14	126	0	65.9	69.2	52.1
6-Aug-14	127	1000	59.7	74.2	54.9
14-Aug-14	128	1000	57.4	63.9	48.4
24-Aug-14	129	0	65.1	64.9	52.3
2-Sep-14	130	0	62.8	62	53.2
10-Sep-14	131	1000	45.3	49.9	42.5
10-Sep-14	132	1000	47.6	47.6	41.5
3-Oct-14	133	1000	56.2	58.5	44.2
9-Oct-14	134	0	42.4	44.4	49.6
23-Oct-14	135	250 (+200 ppm H2O)	35.1	102*	33.7

* 102 ppm for the water content of Run 135. This is lower than the target of 200 ppm total dissolved water. Laboratory experimentation with the Fuel/Aquarius/Water blend resulted in Water measurements close to 200 ppm leading to the conlusion that our mixing in the tank was not vigorous enough to completely solubilize the water into the Aquarius.

4.6 Phase I Activities

In Phase I of the testing, the ARSFSS was operated in the Extended Duration Thermal Stability Test (EDTST) mode. All EDTST-mode testing was accomplished at one temperature profile with the target conditions of 300 °F (149 °C) bulk fuel temperature to the inlet of the fuel-cooled oil cooler (FCOC), 325 °F (163 °C) bulk fuel temperature out of the FCOC and into the Burner

Feed Arm (BFA). The fuel wetted-wall temperature in the BFA was 510 °F (266 °C). Selection of this mode and the temperature conditions was based on past results of evaluations of fuel contaminants where this mode and temperature profile had proven effective in thermal stability evaluations. Duplicate Jet A baseline and additized fuel Runs were performed.

During Phase I testing, fuel samples were taken at various times and from various places on the ARSFSS system and the amount of Aquarius additive present in the fuel was measured using the quick test procedure provided by the Program Sponsor.

4.7 Phase II Activities

Following the EDTST runs, four (4) GDTC mission tests were performed at a temperature profile condition of 325 °F (163 °C) bulk fuel temperature to the inlet of the fuel-cooled oil cooler (FCOC), 375 °F (190 °C) bulk fuel temperature out of the FCOC and into the Burner Feed Arm (BFA) and 500 °F (260 °C) wetted-wall temperature in the BFA. Each GDTC Run consisted of 65 missions. Duplicate Jet A Baseline and Jet A + 4X Aquarius Runs were performed.

During Phase II testing, fuel samples were taken at various times and at various places on the ARSFSS system and the amount of Aquarius additive was measured using the quick test procedure provided by the Program Sponsor.

At the end of Phase II, two additional EDTST-mode Runs were performed – one on the baseline Jet A and one on the baseline Jet A with 4X (1000 ppm) Aquarius. These tests were performed to determine if any degradation had occurred in the thermal stability characteristics of either the baseline fuel or the baseline fuel with the Aquarius additive. Repeat specification, JFTOT Breakpoint and QCM testing were also accomplished to determine if any fuel degradation has taken place that might influence the overall outcome of the program.

4.8 Post-Program Activities

As testing progressed, an additional EDTST-mode test was performed in response to some questions posed by an engine OEM. This run was intended to replicate the condition where Aquarius additive was in the fuel at normal concentration (250 ppm) and sufficient water was present in the fuel to theoretically tie up all of the available Aquarius additive. This Run received the designation Run 135 (AQ-11). These results are presented in Section 5.0.

4.9 Additive Usage and Preparation:

The Aquarius additive was provided to the Air Force for this program. It was delivered to RQTF on February 18, 2014 in nine 2.2L containers. The additive was logged into the RQTF sample database under **POSF-11712**. Per the additive OEM, this additive was stored at a temperature NOT LOWER THAN 32 °F (0 °C). Failure to store properly would have damaged the additive and invalidated this test series. Therefore, upon delivery the additive was stored in Rm 148 of the test facility where ambient temperatures typically do not get lower than 50 °F even during the coldest winter weather.

Per ASTM D4054 guidelines for additive assessment and approval, the Aquarius additive must be blended into a commercially available Jet A fuel at a dosage rate of four times the anticipated normal dosage. In this case, with a planned dosage rate for Aquarius of 250 ppm, the dosage rate for this program was 1,000 ml additive/m^3 of fuel. This meant that approximately 0.9 gallons of Aquarius additive was mixed with 900 gallons of fuel in Tank S-4. Once additive was placed in S-4, the tank was recirculated for 2 hours to make sure the additive was thoroughly blended into the fuel.

4.10 Test Rig Preparation:

The ARSFSS test rig was cleaned prior to introduction of the Aquarius additive to make sure any residual materials from previous testing were removed from the system. The cleaning procedure involved circulating a water/cleaner solution[2] throughout the ARSFSS followed by a thorough water rinse and fuel flush. Fuel used for the fuel flush was a commercial specification Jet A available on the S-Farm. Selected areas of the ARSFSS rig were opened or disassembled to assure that all cleaning and flushing fluids were removed from the system.

After all cleaning and maintenance activities were completed, the ARSFSS was run for two shake-down runs. With all rig parameters showing stable operation and dissolved water measurements showing no additional dissolved water, the ARSFSS rig was declared ready for the test program.

[2] *A solution of 4 parts soft water and 1 part Blue Gold cleaner. This cleaning combo is also typically used in preparing ARSFSS test modules for each Run. See Appendix D for an MSDS describing this cleaner.*

4.11 Fuel Sampling:

Each time fuel/additive blending operations were performed, the additized fuel was analyzed to determine the concentration of the additive in fuel using the test methods and materials provided for that purpose. Dissolved water measurements were also made. This information was collected for inclusion into the final report for this program and is part of Table 9.

A specialized test strip-based method was provided by the additive manufacturer to measure the relative presence of the Aquarius additive in the fuel. This test strip method is a color-comparative method and such methods can be somewhat subjective. Figure 21 shows the test strips from the additized fuel runs. All strips are from samples drawn off the ARSFSS rig toward the end of each test run.

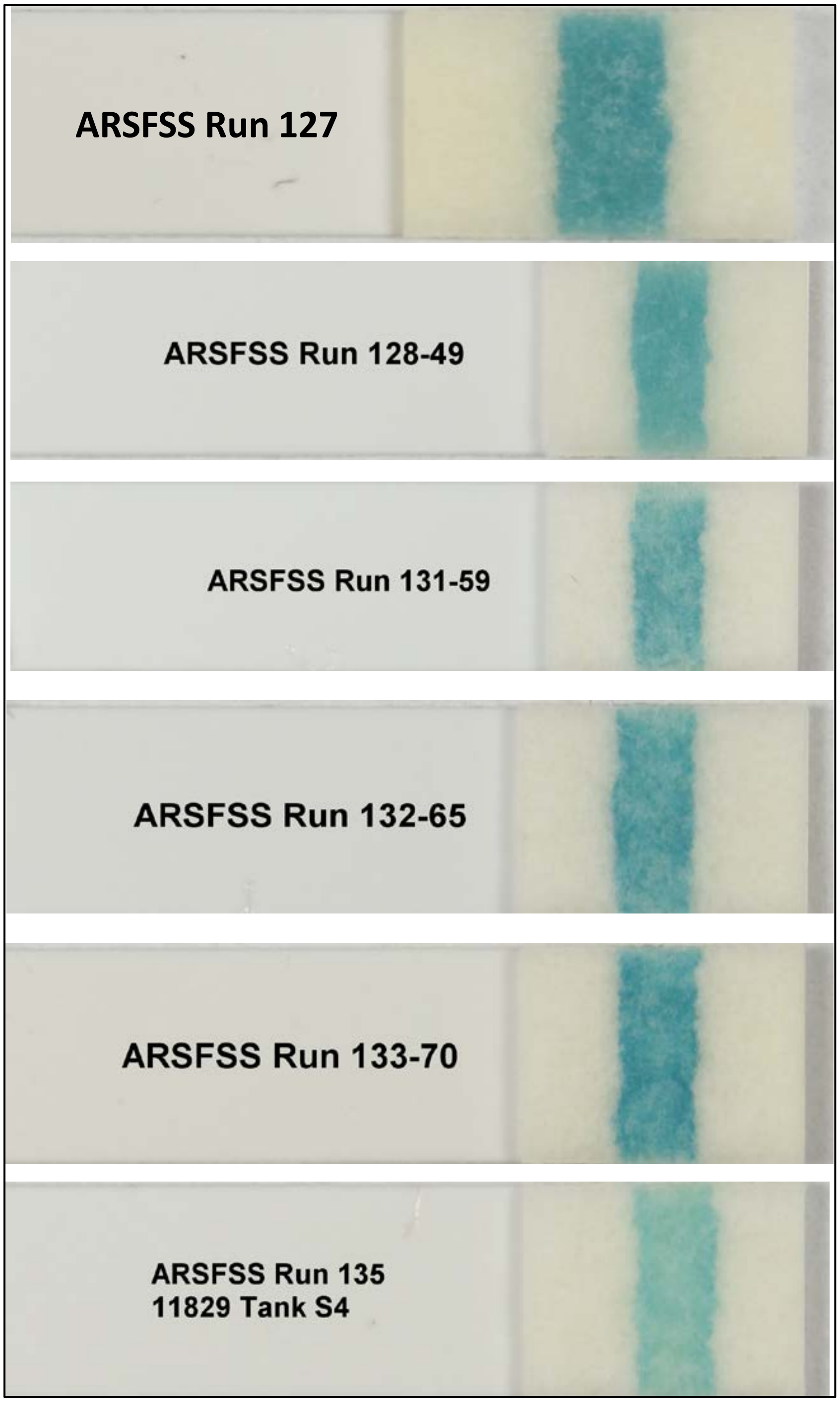


Figure 21 - Test Strips from Additized ARSFSS Runs

5.0 Results and Data-Specific Discussions

5.1 Fuel Approval

Upon receipt of the fuel from the Cleveland, Ohio fuel terminal and subsequent filling of S-Farm tanks S-3, S-4 and S-16, samples were taken for analysis. Two 5-gallon samples were taken from Tank S-16 after the tank had been recirculated for 2 hours at an approximate flow rate of 60 GPM. One 5-gallon sample was submitted for analysis[3]. In a teleconference on 10 July 2014, the Aquarius team came to a consensus that the fuel 11769 was acceptable for use in this program.

5.2 ARSFSS Run Execution and Run-Specific Narratives

5.2.1 Run 124 Pre-Program Condition Verification

Run 124 was initiated on 14 July 2014 and was configured for EDTST-mode operations with standard EDTST-mode conditions shown in Table 2

The fuel for this run was POSF-11769 – the baseline Jet A. The Run was completed on 17 July 2014. Data from this Run are presented in Section 5.3.

At the start of the first ARSFSS run, a sample was drawn off the ARSFSS downstream of the Burner Feed Arm (BFA) and water content was measured on this sample. The total dissolved water content was 70.2 ppm. Water bottoms in tank S-3 were also checked using a tank stick and water-detecting paste. At the start of testing, tank S-3 contained no free water bottoms in the tank. A summary table of all dissolved water measurements for all test runs is located in Table 9.

At about six hours into this first Run, the flow control valve regulating our 'burn flow' test parameter (fuel flow through the TMS and BFA) began to exhibit problems. At the start of the test, the valve was operating at approximately 17% open to maintain the burn flow of 16.7 pph. By the end of about 8 test hours, the valve was operating at 77% open to maintain fuel flow. This behavior is indicative of the valve plugging. As a corrective measure, the valve was repeatedly stroked between its full operating points. The issue was not resolved. The test would not be able to continue until this was fixed because control over the flow rate would be lost.

[3] *Unless specifically stated otherwise, when a fuel sample is submitted for general analysis, such general analysis consists of a full specification analysis in accordance with the Jet A specification, JFTOT Breakpoint, QCM and Water by Karl Fischer (W_{KF}).*

The test was paused in a controlled manner to preserve test integrity. The control valve was removed for repair or replacement. Upon disassembly of the valve, the valve components were coated with a thick gummy material. The valve was cleaned and restored to operation and the test was restarted. Within an hour, the control valve began exhibiting a similar behavior with burn flow control starting at 17% open quickly rising to almost 30%.

In a quick caucus, the research team determined the most likely cause of this behavior was the high temperature conditions under which the test was operating. These conditions were established in a prior program with a fuel of excellent thermal stability. For the 11769 fuel used in this current program, the fuel is a rapid oxidizer in the QCM (which is a closed system test where oxygen is depleted during the test, see Figure 18). In the ARSFSS, where the fuel is constantly replenished with oxygen, it was believed that this rapid oxidation tendency was resulting in a drastic increase in gums in the fuel which were accumulating in the fine orifice of the control valve and restricting fuel flow. The solution was to lower the temperature conditions incrementally until this gum formation and control valve problem were resolved. These conditions would then be adopted as the standard operating conditions for the remainder of the program for EDTST-mode operations.

Upon resumption of the Run (with the conditions of 325 °F bulk fuel temperature at the FCOC inlet, 350 °F bulk fuel temperature at the BFA inlet and 510 °F maximum BFA wetted Wall temperature [325/350/510]) , plugging of the flow control valve continued although at a much lower rate. At about 3 hours into the resumed Run, temperature conditions were reduced to 300/325/510.

After an overnight operation at the 300/325/510 conditions, the flow control performance remained stable indicating that these conditions were the correct conditions for this fuel. See Table 3 in Section 2.8 for the final EDTST-mode operating conditions.

It should be noted that after over a total of 26 hours of operation, the BFA maximum wetted wall temperature remained constant indicating little or no deposition on-going in the BFA. This was a little unusual so the decision was made to run at least another 24 hours to see if any temperature rise would be noted. A slight temperature rise was observed so these conditions were adopted as the default EDTST-mode operating conditions for this program.

Based on the performance of this Run and the changing conditions, this Run was re-designated a 'pre-test Run' for the purpose of finding the correct operating conditions for this program. Data compiled from this run will not be a part of the comparisons to additized fuel.

*5.2.2 **Run 125 (AQ-1)***

Run 125 (AQ-1) was initiated on 21 July 2014 after determining the proper operating conditions (Run 124) and was completed on 24 July 2014 without incident. The ARSFSS was configured for EDTST-mode operations with revised standard EDTST-mode conditions (see Table 3). These revised conditions were used for all EDTST-mode testing for the duration of the program. The fuel for this run was POSF-11769 – the baseline Jet A.

At the completion of the Run, the percentage open value for FCV303 (control valve that controls fuel flow through the TMS and BFA) had increased from about 17% to 29% indicating that bulk fuel deposition was occurring. During that same time, the BFA wetted wall temperature increased from 510 °F to 515 °F indicating that hot surface deposition was occurring in the BFA. All test parameters remained within allowable limits.

Dissolved water measurements were made at the start of the Run on the fuel feed tank (S-3), the bulk fuel storage tank (S-16) and on a fuel sample drawn off the ARSFSS downstream of the BFA. Dissolved water values were 55.3 ppm, 62.9 ppm and 63.4 ppm respectively. Data from this Run are presented in Section 5.3.

*5.2.3 **Run 126 (AQ-2)***

Run 126 (AQ-2) was initiated 28 July 2014 and was configured for EDTST-mode. The fuel for this run was POSF-11769 – the baseline Jet A. This Run was a duplicate of Run 125, AQ-1 performed to establish test repeatability.

At just under 13 hours into the Run, the oscillator tube on the RF heater for the BFA failed resulting in loss of heat to the BFA and a leak in the RF heater cooling system. The coolant leak resulted in the oscillator tube in the FCOC RF heater overheating as well. The system initiated a Mid-Cycle shutdown triggered by loss of coolant pressure. A Mid-cycle shutdown is designed to preserve test integrity in the event of a component failure.

The source of the problem was isolated and repaired. The oscillator tubes were repaired and tested. Run 126 was resumed the morning of 31 July 14 and completed on Saturday, Aug 2nd. Data from this Run are presented in Section 5.3

5.2.4 <u>Run 127 (AQ-3)</u>

Run 127 (AQ-3) was initiated on Tuesday, 5 Aug 2014 using the revised standard EDTST-mode operating conditions. All test parameters during operation were nominal with no test anomalies noted. During the Run, specific data was monitored at various critical points in the system. All data indicated that there was little or no deposition going on in the ARSFSS rig. Data from this Run are presented in Section 5.3

5.2.5 <u>Run 128 (AQ-4)</u>

Run 128 (AQ-4) was initiated on Monday, 11 Aug 2014 using the revised standard EDTST-mode operating conditions. Within a few hours of start, there was an issue with low fuel farm pump pressure due to air in the lines from some fuel farm repair work. The secondary line didn't bleed out in-time and the system detected low pressure. This system detected this anomaly and executed an Emergency Shutdown. The problem was resolved quickly and the test was restarted. Only about an hour was lost.

During the Run, specific data was monitored at various critical points in the system. All data indicated that there was little or no deposition going on in the ARSFSS rig. The Run completed on Thursday morning, 14 Aug 14 without further incident. All test parameters during operation were nominal with no test anomalies noted. Data from this Run are presented in Section 5.3

This completed the first phase of the program, the EDTST-mode ARSFSS Runs.

5.2.6 <u>Run 129 (AQ-5)</u>

In accordance with the test plan, Runs 129-132 were accomplished using GDTC mission cycle mode testing using the conditions in Table 1. The Mission Control Points in Table 1 refer to points in the ARSFSS Flow Diagram Schematic, Figure 1. Run 129 (AQ-5) was started on Wednesday Aug 20th. It should be noted that the conditions in Table 1 are based on standard ARSFSS test conditions for evaluating conventional petroleum-derived JP-8 **WITH THE EXCEPTION OF** the BFA Max Wetted Wall Temperature. True standard JP-8 conditions would have this value be 450 °F instead of 500 °F. However, for this program, 500 °F was selected in an attempt to operate closer to the University of Sheffield's AFTSTU Rig conditions without significantly compromising real-world aircraft fuel system relevance. These are similar conditions to those chosen for prior programs, including the JP-8+100 development program and the recently completed FAME evaluation.

Run 129 (AQ-5) was successfully completed on Tuesday, 26 August 2014. The test ran normally but did experience higher differential pressure across the TMS during the mission segments that were high fuel flow conditions (see Table 1for a chart showing ARSFSS typical flow conditions during mission segments). This is logical as during these conditions, we are attempting to flow 169 pounds per hour of fuel through the TMS. During this condition, the screen itself will offer significant Delta-P. A screen that has collected even moderate deposits may be even more sensitive to a high differential pressure at these high flow conditions.

*5.2.7 **Run 130 (AQ-6)***

Run 130 (AQ-6) was started on Thursday, 28 Aug 2014 as a duplicate baseline fuel run. During the 11th mission, the differential pressure across the TMS exceeded our set high limit and the ARSFSS initiated a controlled, end-of-mission shutdown. Data was reviewed and an assessment made of the high limit for the TMS. The high limit was reset to 10 PSID and the test was resumed with Mission 12. As we approached 40 missions, it was obvious that differential pressure across the TMS was increasing so we removed the TMS DP Sensor from the critical alarms that would shut down the rig. As of mission 56, the differential pressure across the TMS exceeded the measuring limit of the sensor so we no longer were able to track the actual differential pressure across the TMS.

Run 130 completed 65 mission cycles on Wednesday, 3 Sep 14 without further incident.

*5.2.8 **Run 131 (AQ-7)***

Run 131 (AQ-7) was started on Thursday, 4 Sep 14 and was the first mission cycle run on Aquarius-additized fuel. During this run with one minute remaining in Mission 59, a Uninterruptable Power Supply failed (I guess it wasn't as 'uninterruptable' as we had hoped…) and caused a hard power shutdown of the control system resulting in a rig shutdown. No test data was lost and the rig did not suffer any damage. The power supply was replaced and the test restarted. Run 31 completed on Wednesday, 10 September 14 without further incident.

5.2.9 Run 132 (AQ-8)

Run 132, the repeat run with Aquarius-additized fuel started Thursday, 11 Sep 14. Within the first mission, it was noted that the temperature above the normal hot spot on the BFA was indicating temperatures 10-16 °F higher than the normal TE324 hot spot. We suspected a defective thermocouple or thermocouple channel. After troubleshooting, it was determined that the thermocouple and the channel were not the problem so the issue had to be with the build of the BFA assembly. After the normal completion of the first mission the test was stopped and the BFA assembly was replaced. The test was restarted with Mission 1 on Friday, 12 Sep 14. Temperatures were normal except that the hot spot temperature appeared to float between TE324 (Section 8 of the BFA) and TE325 (Section 9 of the BFA) but we were not experiencing significantly high temperature anomalies. This 'floating' occurs sometimes and we believe it is generally due to slight deformations in the BFA test tube itself. This behavior normally has no effect on the data profile for carbon deposition so we do not make other adjustments except to monitor the temperatures closely to make sure the hot spot does not significantly change in temperature or further migration of location. Such was the case in this test. The hot spot showed no further migration and there were no temperature anomalies. The Run completed on Thursday 18 Sep 14 without further incident.

5.2.10 Run 133 (AQ-9)

Runs 133 (AQ-9) and 134 (AQ-10) were planned so that the condition of the baseline and additized fuel could be checked for any degradation that might have occurred over the course of the program. If the data from these two tests did not deviate significantly from Runs 125-128 then it could be assumed that no significant degradation in the thermal stability performance of the fuel had occurred resulting increased confidence in the testing results.

Run 133 (AQ-9) was started on Monday, September 29th 2014 using EDTST-mode configuration and conditions. The fuel used was Jet A additized with 1000 ppm Aquarius additive. Run 133 (AQ-9) was successfully completed on Thursday, October 2nd 2014. The test ran normally except for two brief shutdowns due to low pump pressure from the fuel farm tank pump. These shutdowns occurred in controlled manner and the test was not adversely affected.

5.2.11 Run 134 (AQ-10)

Run 134 (AQ-10) was started on Monday, October 6th, 2014. Fuel used for this test was baseline Jet A with no Aquarius additive. The run completed on Thursday, October 9th, 2014 without incident.

5.2.12 Run 135 (AQ-11)

As testing proceeded and results were obtained, a question arose regarding additive performance under conditions where the additive was confronted with a large amount of water in the fuel. Therefore another test was added to the plan to evaluate the additive at the standard anticipated dosage rate (250 ppm) in the presence of 200 ppm total dissolved water (Total dissolved water is the water that is normally dissolved in fuel, 40-60 ppm, with enough water added to bring the total to 200 ppm).

The test was run in EDTST-mode using standard EDTST-mode conditions. The fuel/additive/water blend was prepared in accordance with the following procedure.

1. *Prepare additized fuel with normal blending procedures. Additive concentration for Run 135 (AQ-11) will be 250 ppm instead of the normal 1000 ppm).*
    a. *Measure concentration of additive in the fuel using the litmus strips provided.*
    b. *Photograph the witness strips as evidence the fuel has the right amount of additive.*
2. *Within 12 hours of the test start, blend water into the fuel with the following procedure:*
    a. *Measure existing water content of the fuel (by KF) in the tank to be used and record this amount*
    b. *Prepare a quantity of water to add to the fuel to bring the TOTAL DISSOLVED WATER CONTENT OF THE FUEL TO 200 PPM. For example, if the additized fuel contains 50 ppm water, add an additional 150 ppm water. Water used shall be RO or distilled water.*
    c. *Start circulating the fuel in the tank. Circulate for 30 minutes to assure on-going turbulence in the tank.*
    d. *Add the determined amount of water to the tank in 25% increments while the tank is circulating. Circulate the fuel in the tank continuously during water addition. After adding the first 25% or the water, allow the tank to circulate at least 30*

minutes before adding more water. Do this repeatedly until all the water has been added. Once all of the water has been added, continue to circulate the tank for another 30 minutes.

Once prepared, this fuel was used for Run 135 (AQ-11). The Run was started on Monday, October 20th, 2014 and ended normally Thursday October 23rd, 2014 without incident.

5.3 Data Analysis - Runs 125 through 135

5.3.1 Fuel-cooled Oil Cooler (FCOC) and Burner Feed Arm (BFA) Carbon Deposition

Figure 22 and Figure 23 show carbon deposition (micro-grams per square centimeter) in the BFA and FCOC (respectively) for Runs 125 through 135. In these charts, black lines (solid or dashed) represent testing done in EDTST mode. Blue lines (solid or dashed) represent testing done in the GDTC mode. Solid lines of either color represent tests with non-additized baseline fuel. Dashed lines of either color represent tests with Aquarius additized fuel. The green dashed line is unique in that it represents additized fuel at 250 ppm in EDTST mode (Run 135).

In the BFA, EDTST-mode and GDTC-mode results show moderate (based on our historical rig experience) deposition that appears to be independent of the operational mode. However, additized fuel shows a much reduced, nearly zero, deposition. Again, these results appear to be independent of run mode. These results show that fuel containing the Aquarius additive has much less a tendency to form deposition than the baseline fuel. However from Run 135 data, when the amount of Aquarius additive is reduced to normal target dosage values, the single test indicates that deposition is more characteristic of the baseline fuel. This is an indication that the Aquarius additive, when present in target dosage amounts, has no detrimental effect upon fuel thermal stability and carbon deposition tendencies.

In the FCOC, which experiences a slightly lower wetted wall temperature than the BFA, deposition is virtually the same for both baseline and additized fuel. Once again, indicating that fuel containing the Aquarius additive has no impact on fuel thermal stability performance and deposition tendency.

The data shows that there is significantly less deposition in the BFA and FCOC using the additized fuel compared to the baseline fuel.

Overall, the distinct difference in deposition of baseline and additized fuel in the BFA leaves little room for doubt that the Aquarius additive has no detrimental impact on fuel deposition.

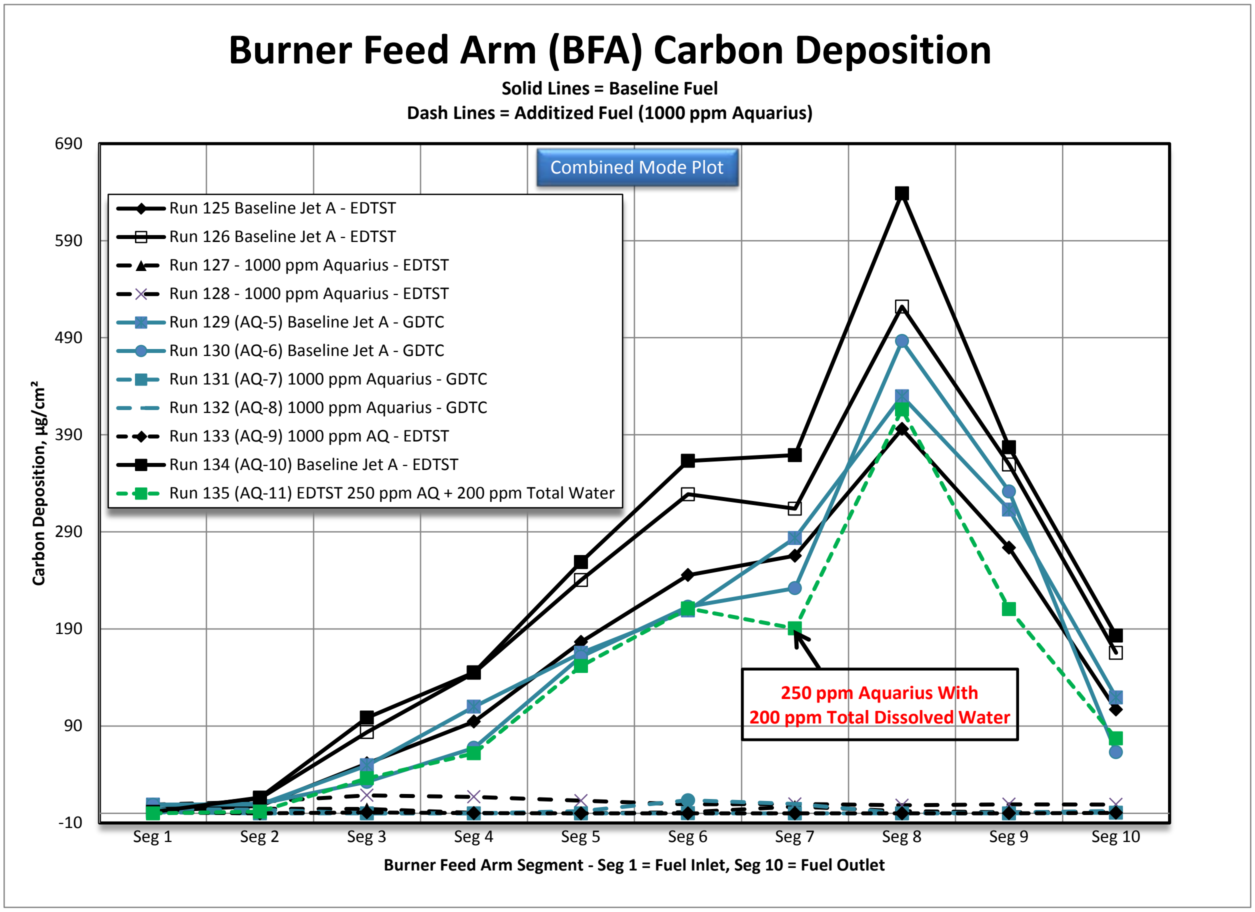


Figure 22 - BFA Deposition, Runs 125 (AQ-1) Through Run 135(AQ-11)

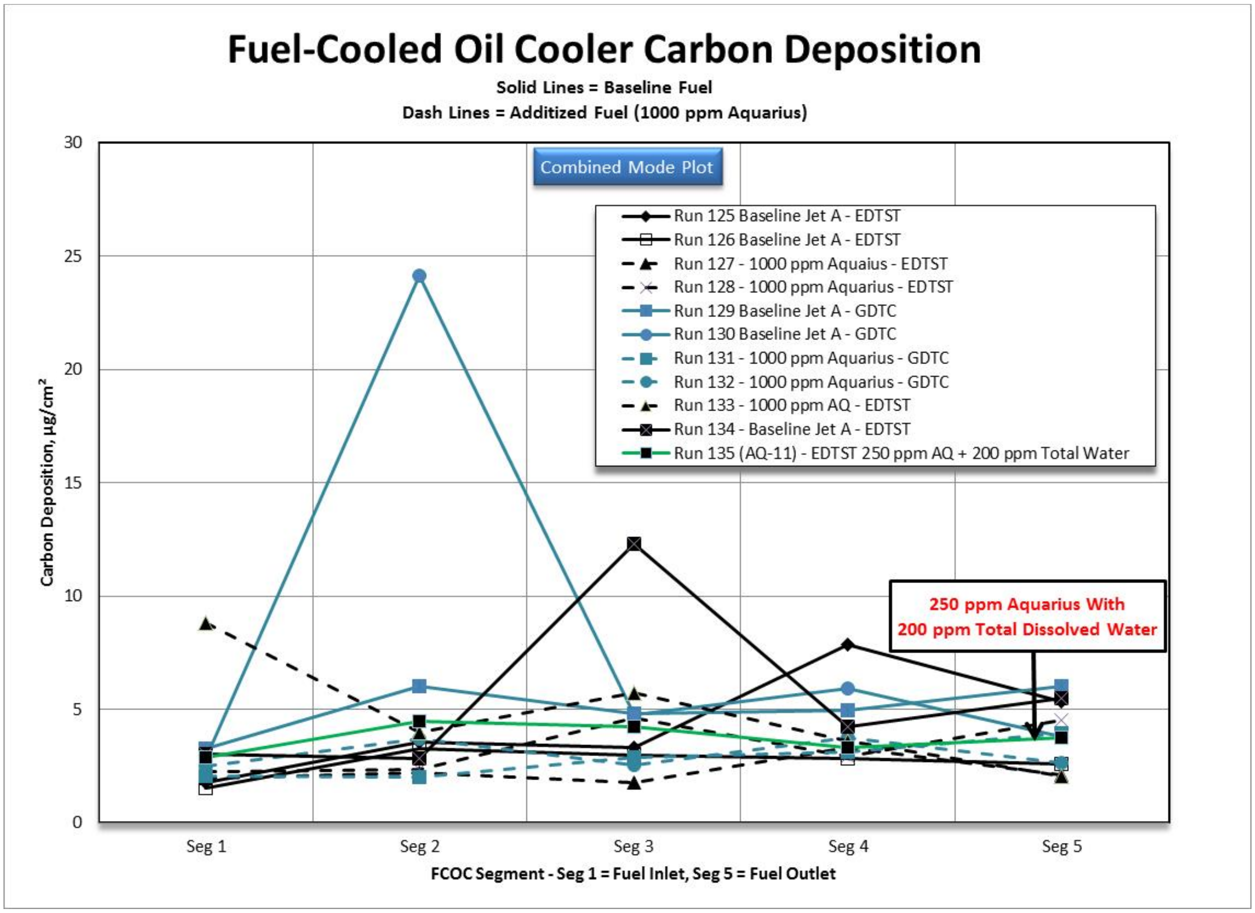
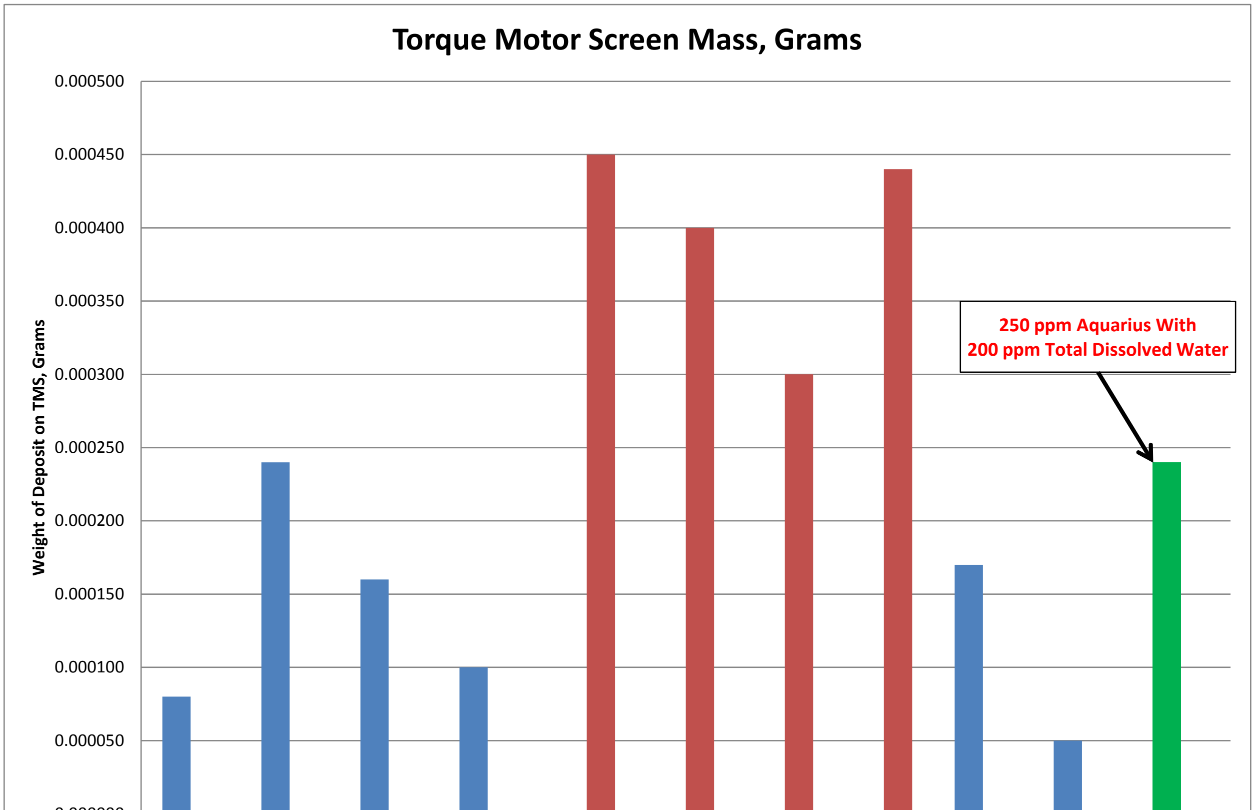


Figure 23 - FCOC Deposition, Runs 125 (AQ-1) Through Run 135(AQ-11)

5.3.2 Torque Motor Screen (TMS) Carbon Deposition and Visual Appearance

5.3.2.1 Torque Motor Screen Mass and Carbon Deposition

Two types of deposition measurements were made on the TMS – total mass accumulation (which would include carbon and non-carbon materials) and total effective carbon (carbon only) as determined by LECO Carbon Analyzer. Total Effective Carbon is the difference between the total measured carbon when the TMS is analyzed less the normal background carbon measure from a clean unused screen. Figure 24 shows the total weight of the deposition on the TMS for each Run. Blue bars represent mass accumulation during EDTST-mode Runs and red bars represent mass accumulation for GDCT-mode runs. There is significantly more mass accumulation for GDTC-mode runs than for EDTST-mode runs. This is expected and is due to the fact that a GDCT run consumes around four times the amount of fuel that an EDTST-mode run consumes. The mass accumulation based on fuel type does not show an apparent correlation in this data leading to the conclusion that total mass accumulation is not a function of fuel type, additized or non-additized.



	125	126	127	128	129	130	131	132	133	134	135
EDTST	0.000080	0.000240	0.000160	0.000100					0.000170	0.000050	0.000240
GDTC					0.000450	0.000400	0.000300	0.000440			

Figure 24 - Torque Motor Screen (TMS) Mass Accumulation

Figure 25 shows data similar to total mass accumulation but it is for total effective carbon as measured by Leco Carbon Analyzer. As with mass accumulation data, the magnitude of effective carbon deposition for GDTC-mode runs is greater than for EDTST-mode runs – again due to the difference in fuel quantities used. There is no readily apparent correlation between run mode type or fuel type and carbon deposition leading to the conclusion that the presence or absence of the Aquarius additive has no discernible negative affect on the data.

Table 10 shows a calculation of non-carbon mass for the TMS from each Run based on Total TMS Mass and Total Effective Carbon (as measured by the LECO Carbon Analyzer). This table shows a distinct reduction in Total Effective Carbon as well as Non-Carbon mass when the Aquarius additive is used.

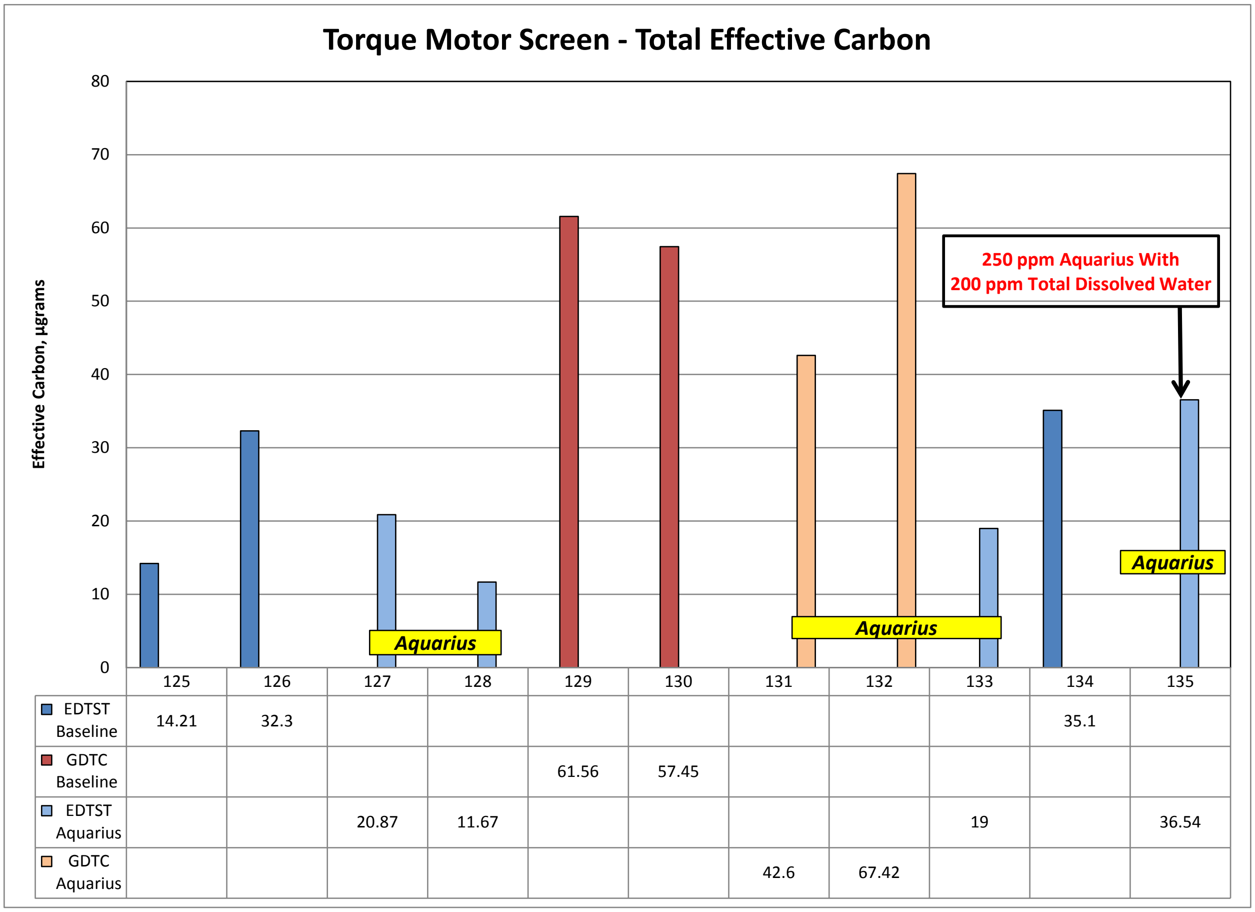
It should be noted that the total mass deposition on the TMS for Run 134 is substantially lower than for the previous baseline fuel Runs in EDTST mode. This data point may be correct or it may be in error – it is not possible to prove for certainty. However, given that the value is much lower than any of the test runs regardless of mode or fuel type, it is very likely that this value is an anomaly and not the correct value.

Table 10 – Tabulation of Total Mass, Effective Carbon Mass and Effective Non-Carbon Mass for the TMS

Run No.	Run Description	Total TMS Mass		Total Effective Carbon		Non-Carbon Mass
		Gross mass of deposit before C-burn off, µg	Av. Gross mass, µg	Effective C from LECO analyser, µg	Av. C mass µg	Estimated non-C deposit, µg
125	EDTST Baseline	80	160	14.21	23.26	136.75
126		240		32.3		
127	EDTST Aquarius	160	130	20.87	16.27	113.73
128		100		11.67		
129	GDTC Baseline	450	425	61.56	59.51	365.50
130		400		57.45		
131	GDTC Aquarius	300	370	42.6	55.01	314.99
132		440		67.42		
133	ED Aq	170	170	19	19.00	151.00
134	ED Base	50	50	35.1	35.10	14.90
135	ED Aq 250	240	240	36.54	36.54	203.46

5.3.2.2 Torque Motor Screen Visual Appearance

Figures 26 and 27 show a comparison of the appearance of deposition on the TMS in both EDTST and GDTC modes. These photographs show that for EDTST-mode testing, there is essentially no degradation in appearance of deposition between baseline and additized fuel. However, for GDTC-mode testing, there appears to be a slight improvement in the appearance of the TMS screens for additized fuel versus the baseline fuel. This is consistent with other results obtained in this program.



	125	126	127	128	129	130	131	132	133	134	135
EDTST Baseline	14.21	32.3								35.1	
GDTC Baseline					61.56	57.45					
EDTST Aquarius			20.87	11.67					19		36.54
GDTC Aquarius							42.6	67.42			

Figure 25- Torque Motor Screen (TMS) Effective Carbon Deposition by LECO Carbon Analyzer

Torque Motor Screen – EDTST Mode

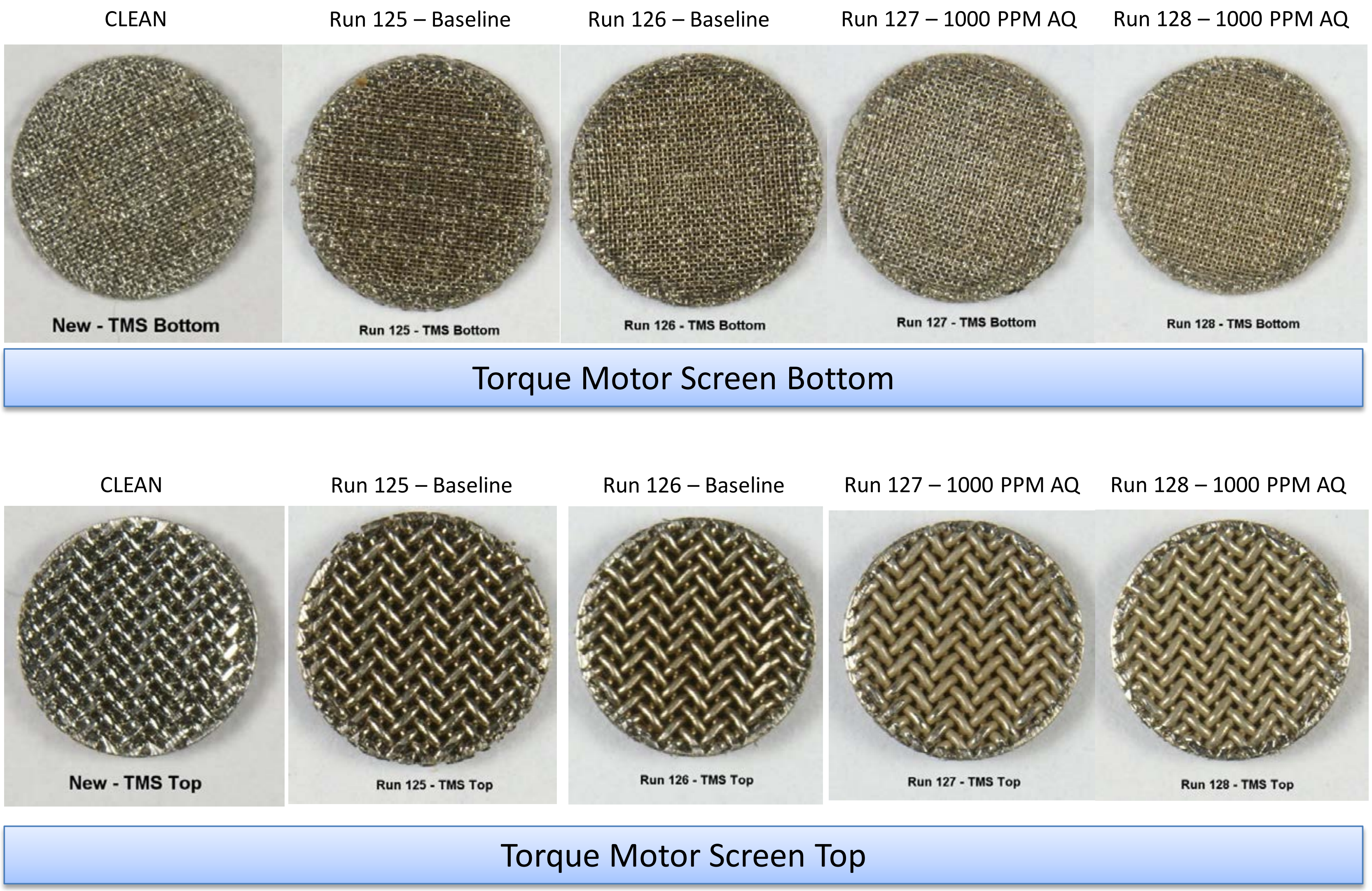


Figure 26 – Deposition on TMS, EDTST-Mode, Baseline and Additized Fuel Comparison

Torque Motor Screen – GDTC Mode

Figure 27 – Deposition on TMS, GDTC-Mode, Baseline and Additized Fuel Comparison

5.3.3 Servo Valve (SV) and Flow Divider Valve (FDV) Hysteresis

Figure 28 shows hysteresis plots for both the Servo Valve for Runs 125 through 128. These are EDTST-mode Runs with both baseline and additized fuels. With the exception of a significant hysteresis spread in Run 125 (Baseline Jet A), hysteresis is not markedly degraded in either baseline or additized test runs. However, when comparing the hysteresis spread of baseline fuel runs to the additized fuel runs, the hysteresis performance of the Servo Valve appears to be slightly better for additized fuel than for the baseline fuel.

Figure 29 shows hysteresis plots for both the Flow Divider Valve for Runs 125 through 128. These are EDTST-mode Runs with both baseline and additized fuels. As with the Servo Valve, generally the magnitude of the hysteresis performance degradation seems to be consistent for both additized and baseline fuels. However, looking at the detailed shapes and shift of the curves, it appears that the shift and spread of the curves for baseline fuel is slightly greater than for the additized fuel indicating that while there is no overtly detrimental impact of either fuel on the performance of the FDV, the additized fuel tends to give slightly better performance in the FDV than the baseline fuel.

Figure 30 shows the hysteresis plots for both the Servo Valve for Runs 129 through 132. These are GDTC-mode Runs with both baseline and additized fuels. For the baseline fuel Runs (129 and 130) there was SUBSTANTIAL hysteresis in the Servo Valves of both Runs. Hysteresis was so severe that the valves became essentially non-functional. In an aircraft, this valve performance degradation would have likely resulted in a control failure of some form. However, with the use of the Aquarius additive, these servo valves retained their fundamental operational characteristics even though there was substantial hysteresis in the valve for Run 132. Again, this is clearly indicative that the presence of the Aquarius additive reduces valve deposition and helps to maintain valve performance.

Figure 31 shows the hysteresis plots for the Flow Divider Valve for Runs 129 through 132. In the baseline fuel runs, (129 and 130), both valves exhibit not only a substantial degradation in valve performance due to hysteresis but the fundamental performance curve for these valves has significantly shifted. For runs 131 and 132 with the Aquarius additive present, the shift in hysteresis is significantly reduced and the spread in hysteresis performance is only slight.

In accordance with the test plan (Table 5), after the completion of Runs 129-131, two additional EDTST-mode runs were planned – one on baseline fuel and one on additized fuel. The purpose of these Runs was to provide cross-check data to determine if the overall fuel had changed during program. Figures 32 through 35 show comparative hysteresis plots for the Servo Valve for baseline and additized fuel at the start of the program to the end of the program. These figures demonstrate that, based on these hysteresis characteristics, there was no overall change in performance in either the baseline or additized fuel from the start of the program to the end of the program. This indicates that the fuel thermal stability remained consistent throughout the program.

Figures 36 and 37 show the results of Run 135 Servo and Flow Divider Valve hysteresis measurements (see Section 5.2.12 for a full description of Run 135 and the rational for performing this test). These figures show that hysteresis for both of these valves is consistent with the unadditized baseline fuel runs indicating that the presence of the additive with water is not detrimental to valve performance.

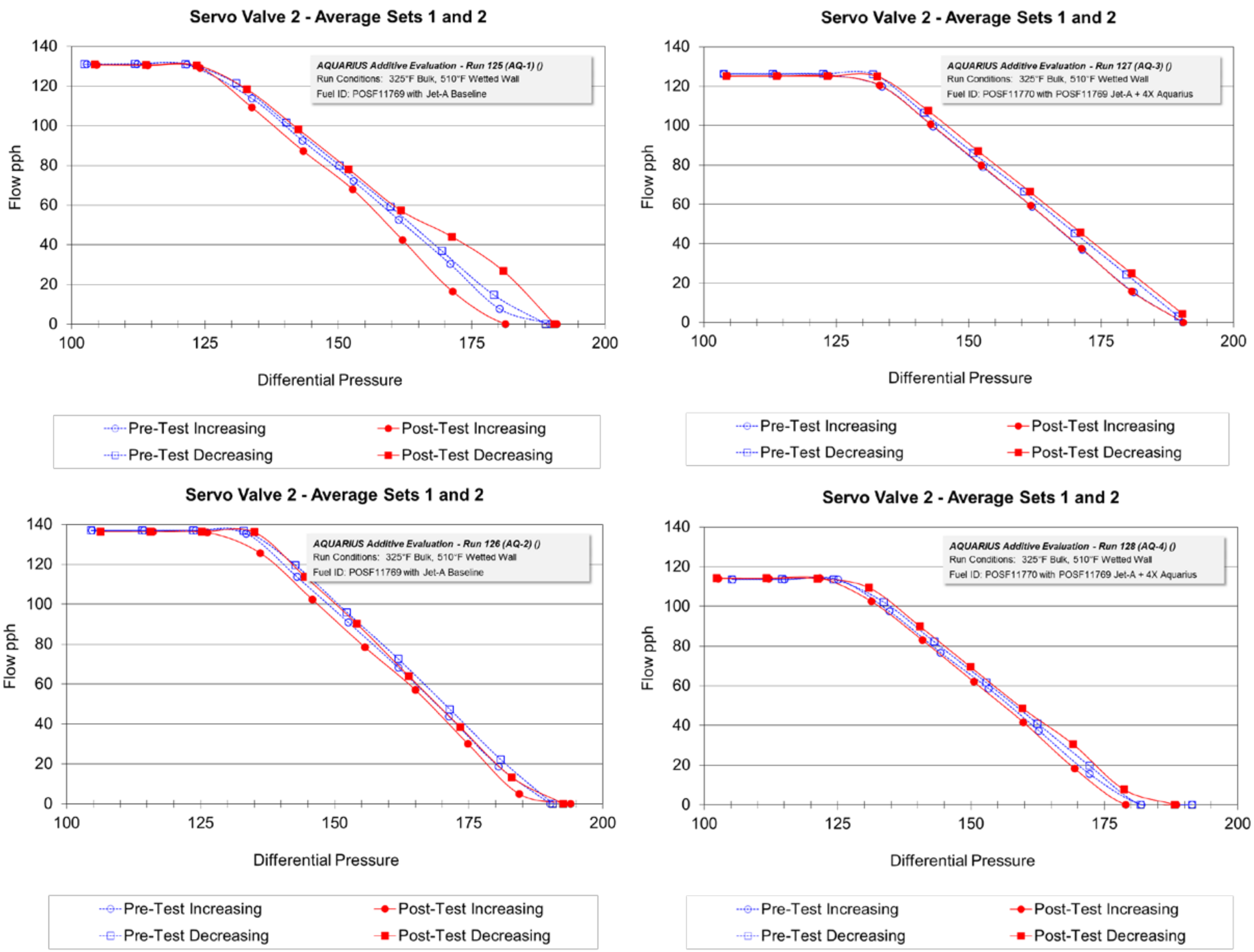


Figure 28- Servo Valve Hystersis Comparison, EDTST-Mode, Baseline and Additized Fuels

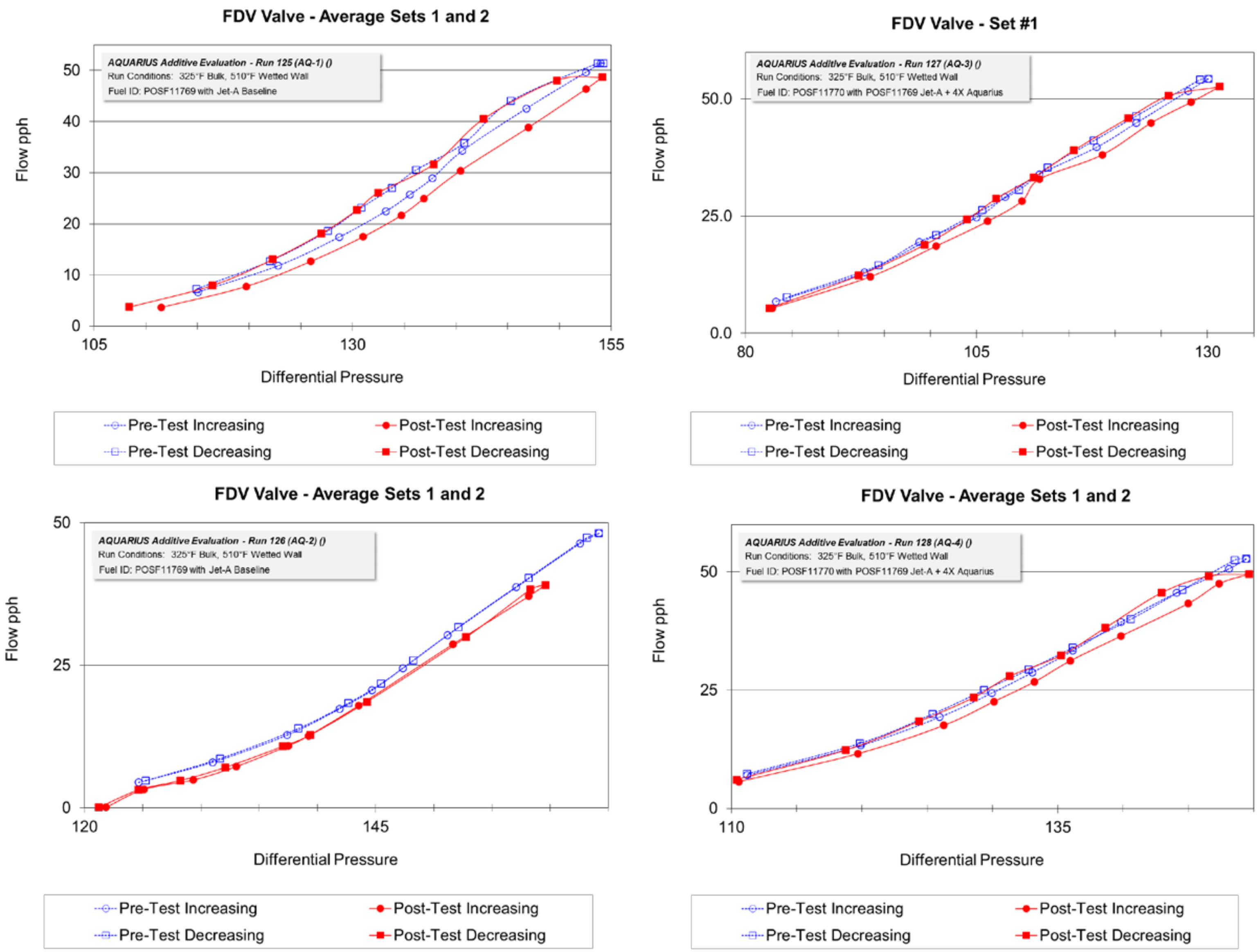


Figure 29- Flow Divider Valve Hystersis Comparison, EDTST-Mode, Baseline and Additized Fuels

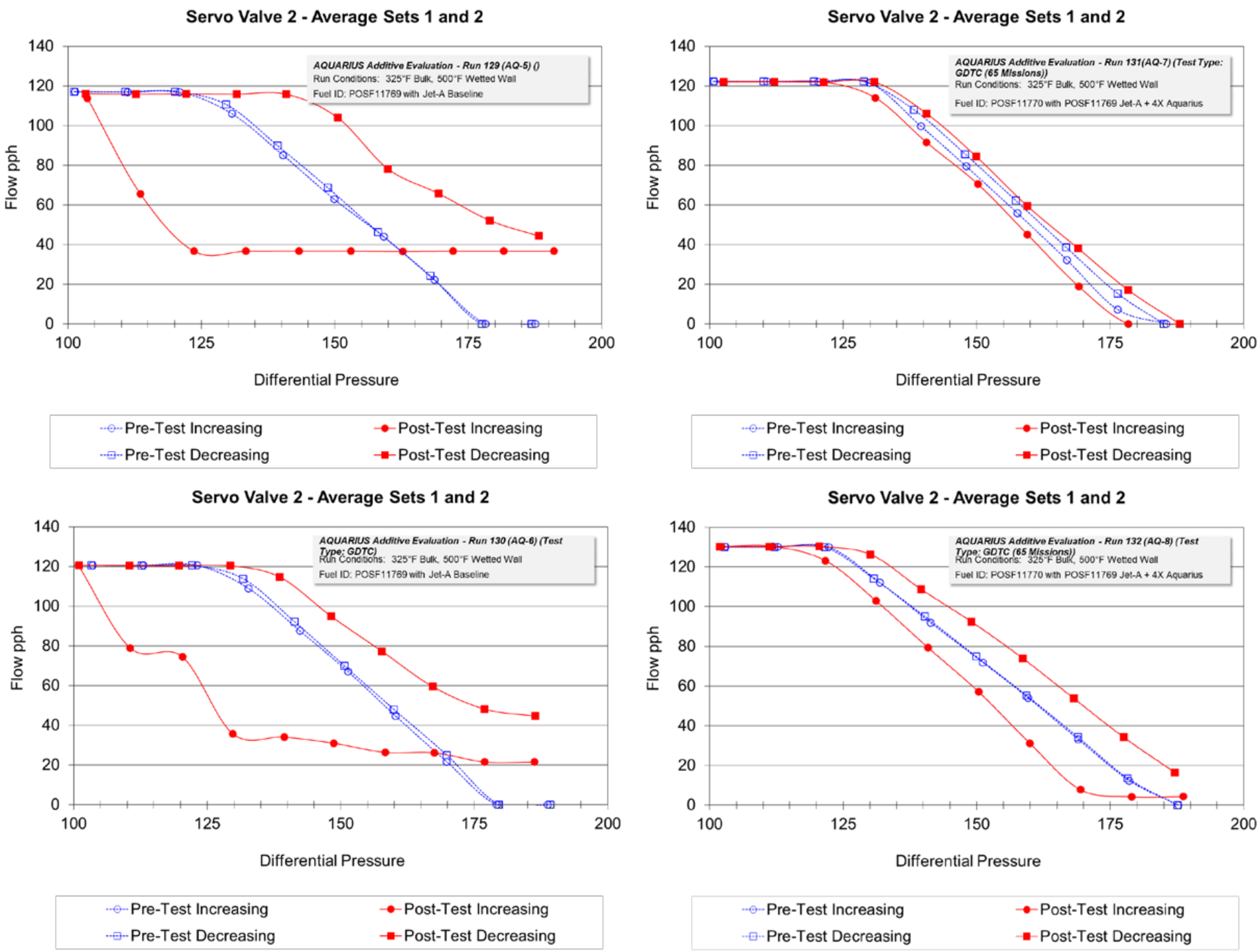


Figure 30- Servo Valve Hystersis Comparison, GDTC-Mode, Baseline and Additized Fuels

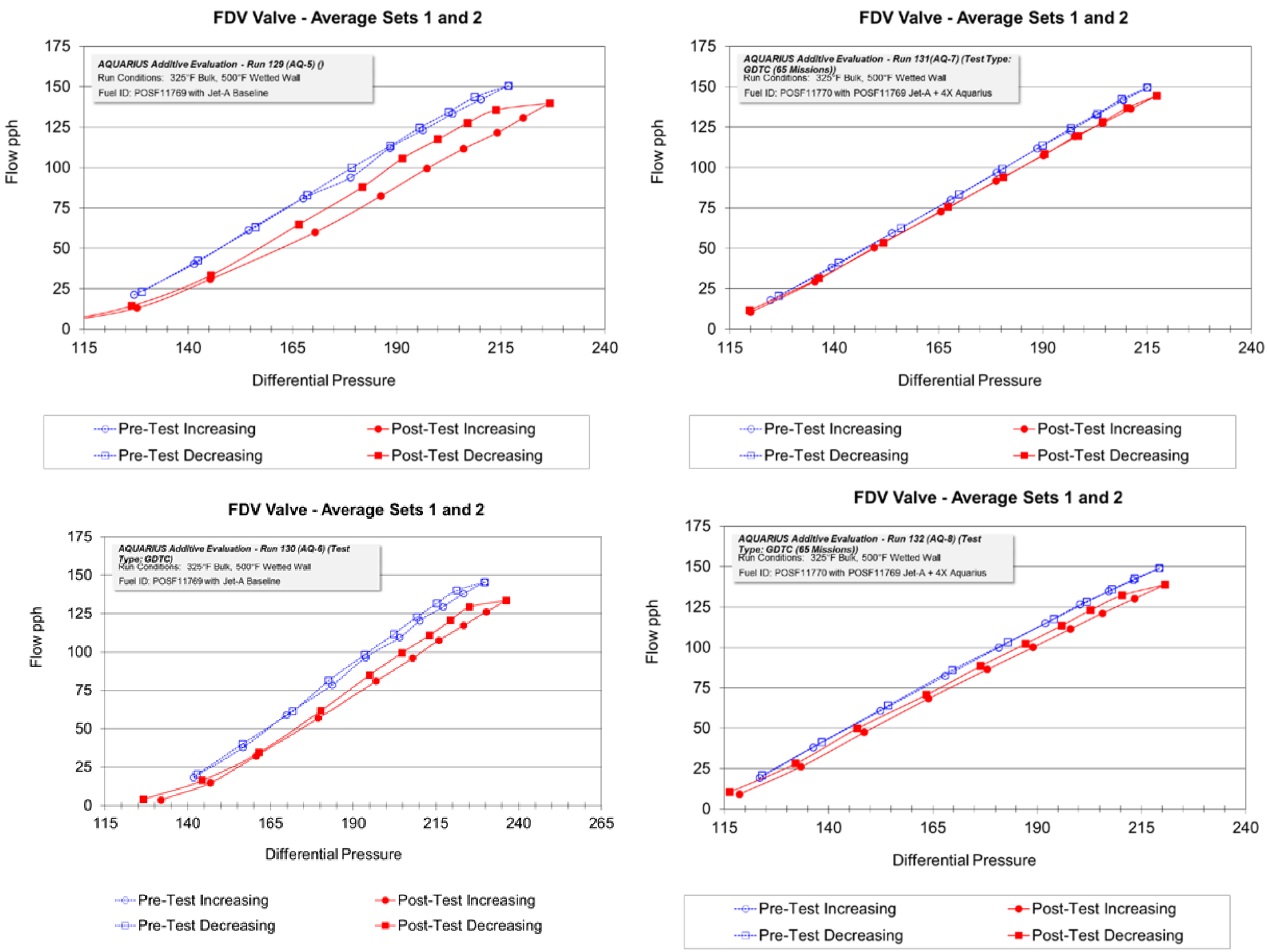
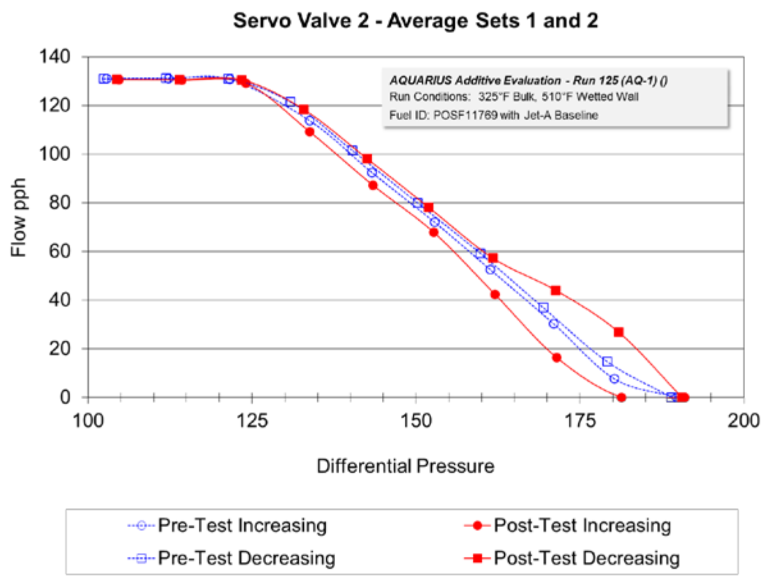
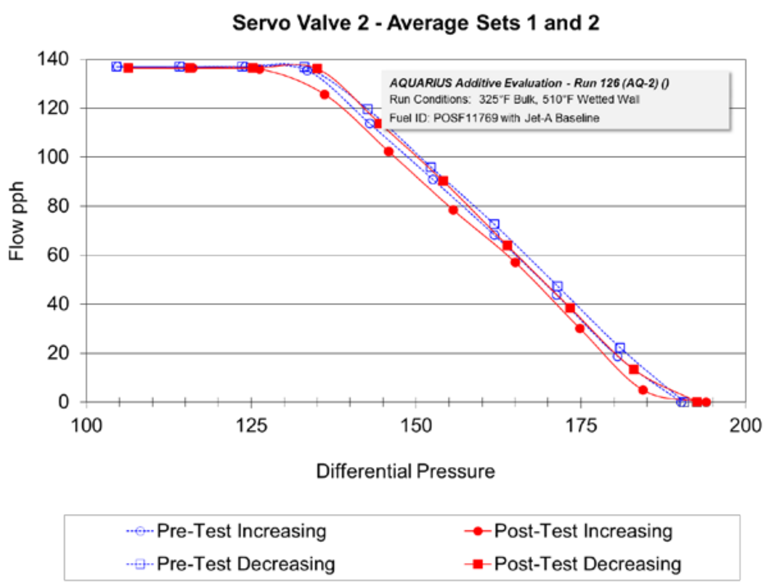


Figure 31- Flow Divider Valve Hystersis Comparison, GDTC-Mode, Baseline and Additized Fuels





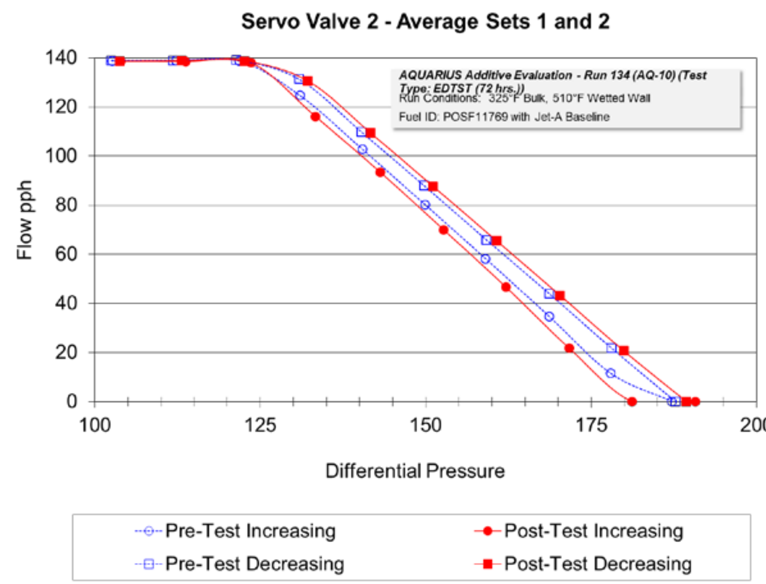


Figure 32 - Servo Valve Hysteresis, GDTC-Mode, Baseline Fuel, Start of Program and End of Program

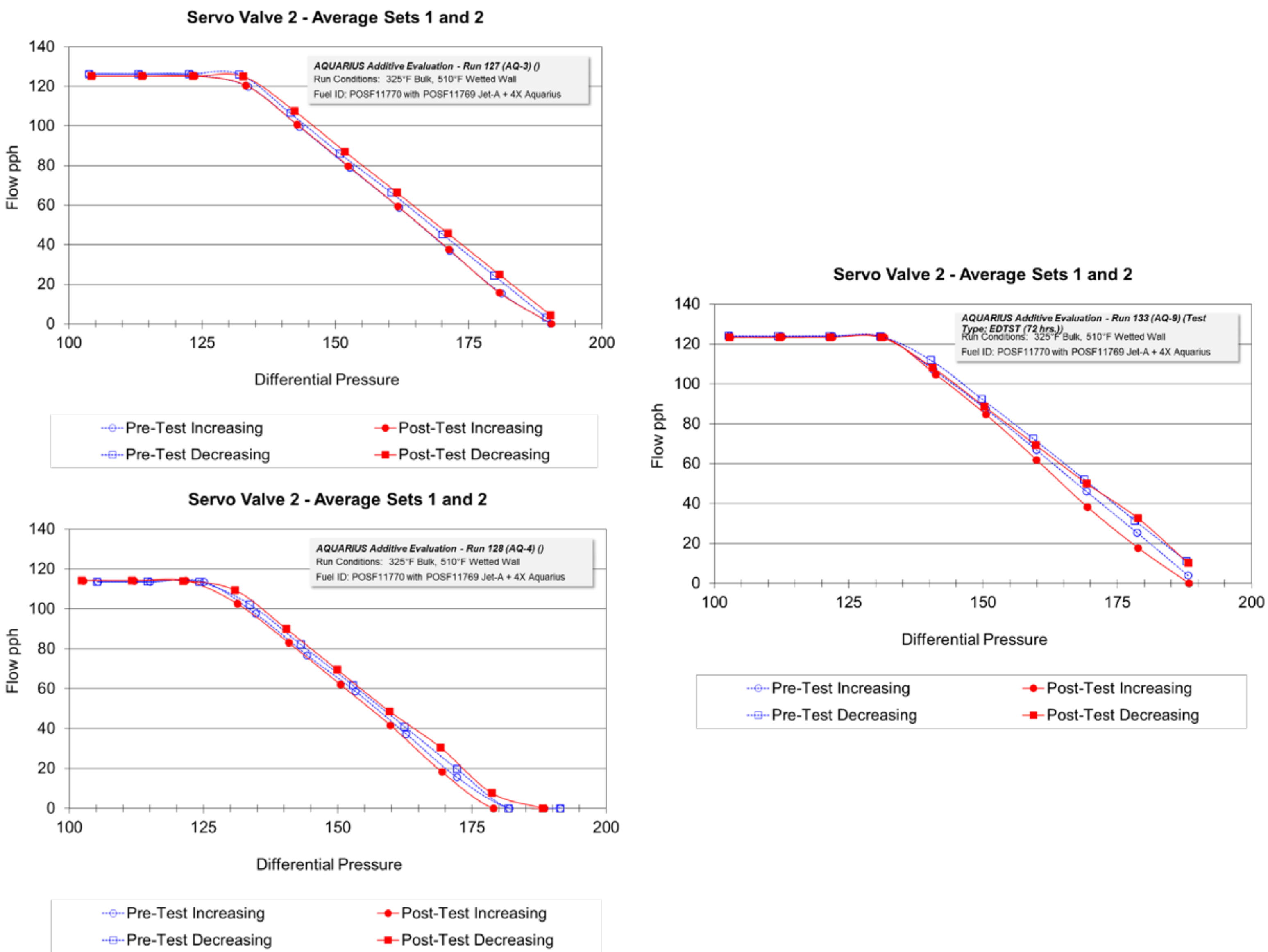


Figure 33 - Servo Valve Hysteresis, GDTC-Mode, Additized Fuel, Start of Program and End of Program

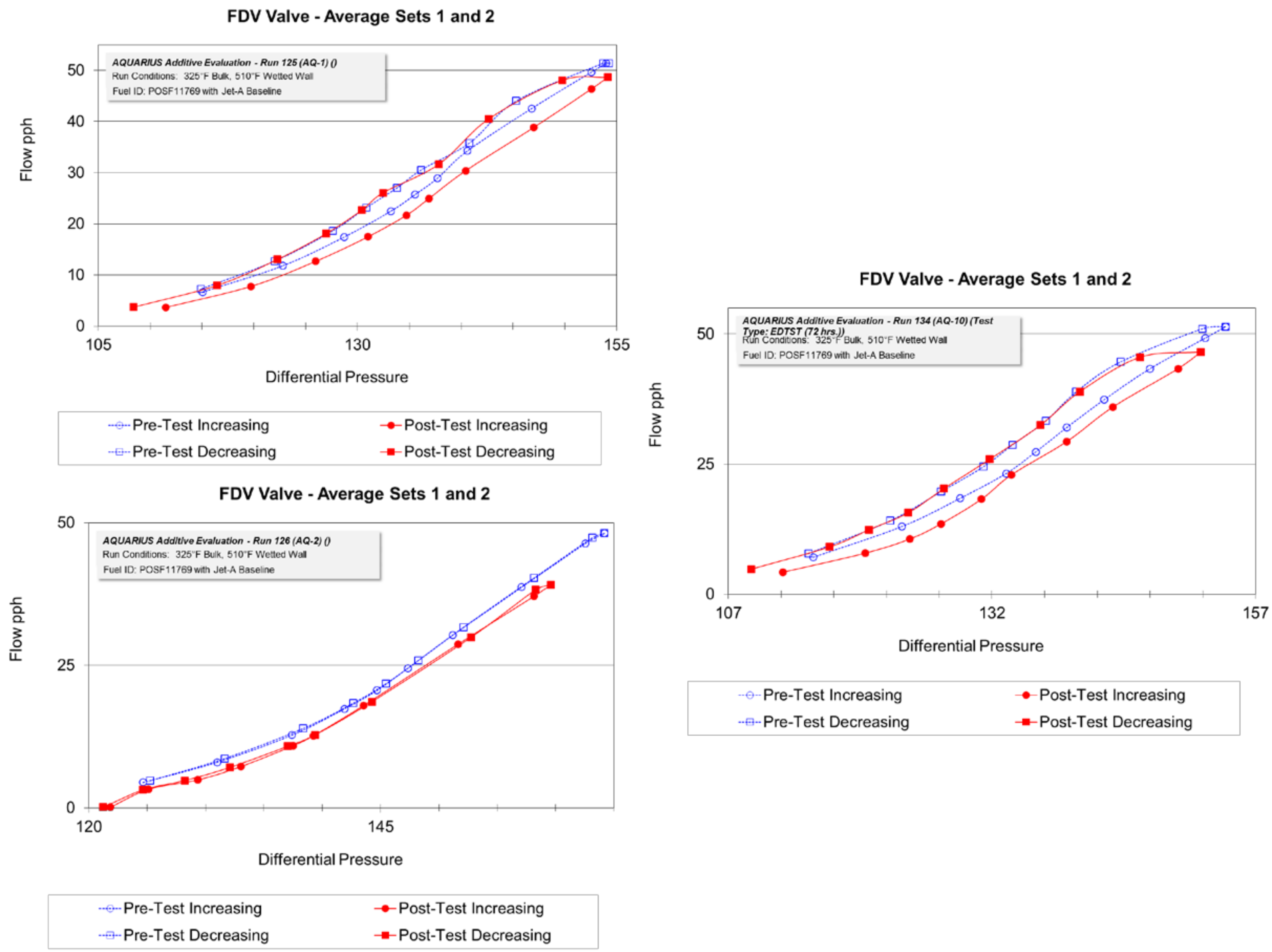


Figure 34 – Flow Divider Valve Hysteresis, GDTC-Mode, Baseline Fuel, Start of Program and End of Program

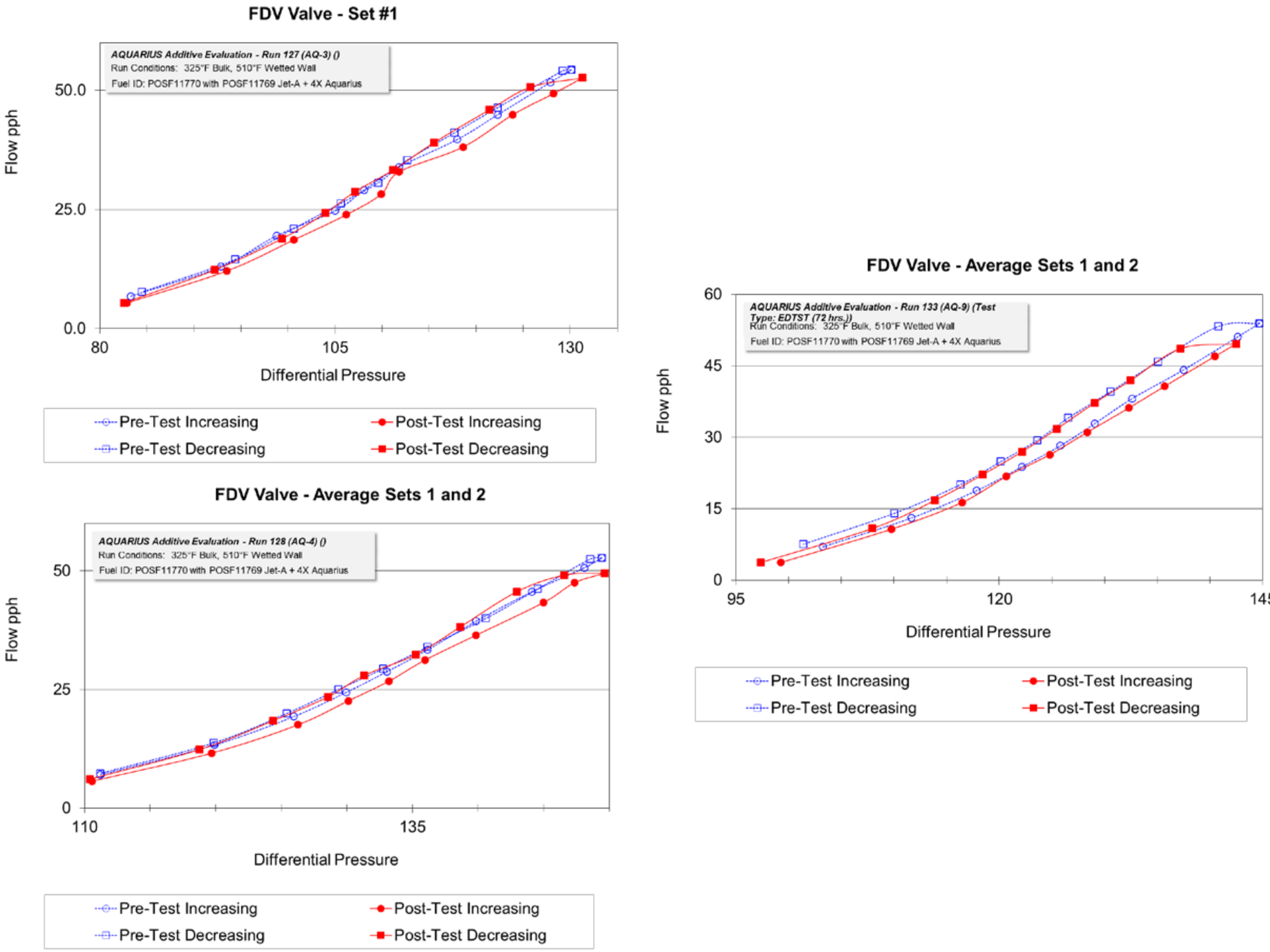


Figure 35 – Flow Divider Valve Hysteresis, GDTC-Mode, Additized Fuel, Start of Program and End of Program

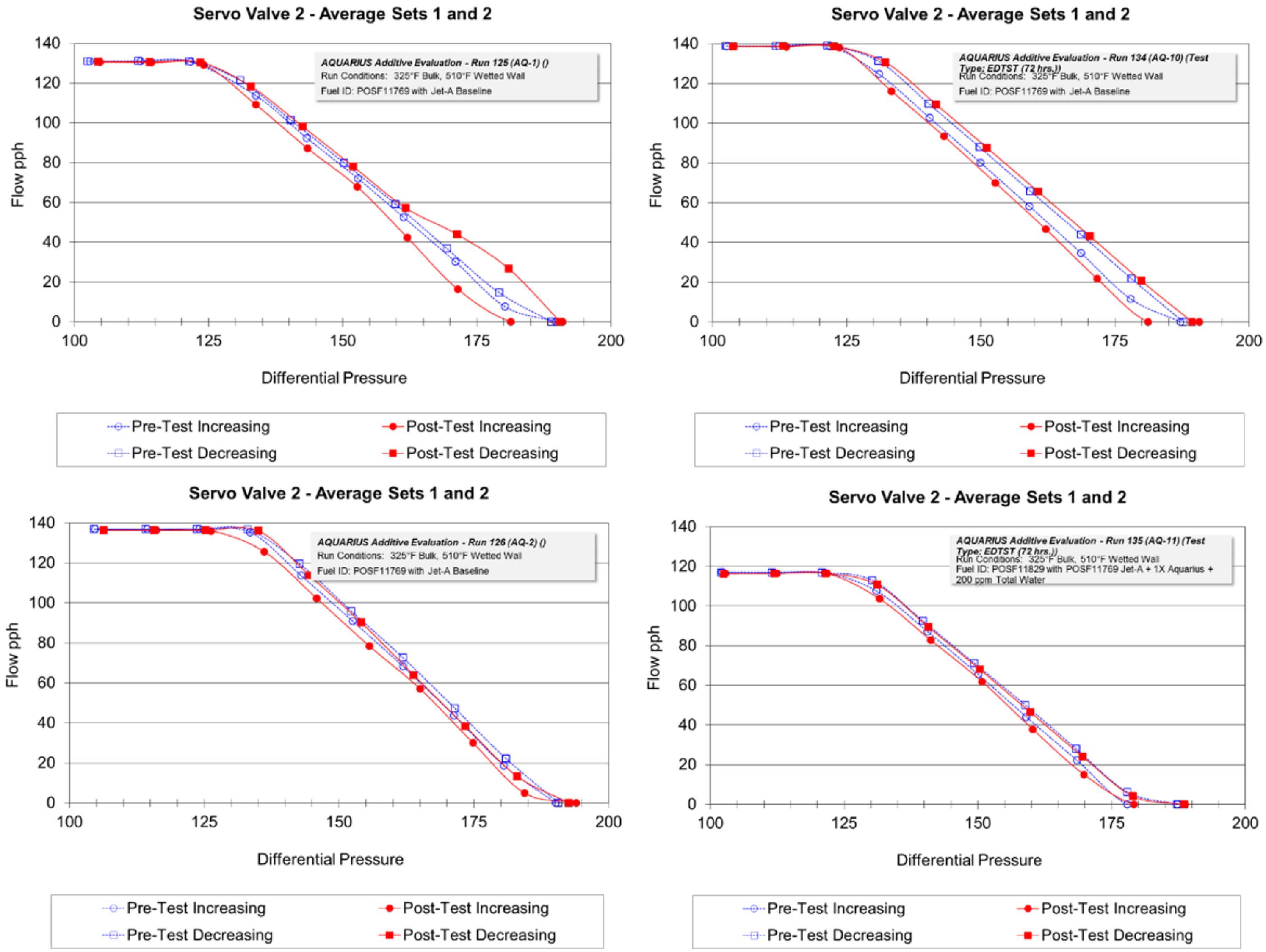


Figure 36 – Comparison of Servo Valve Hysteresis for Run 135 (250 ppm Aquarius + 200 ppm Total Dissolved Water) to Previous Baseline Test Fuel Runs

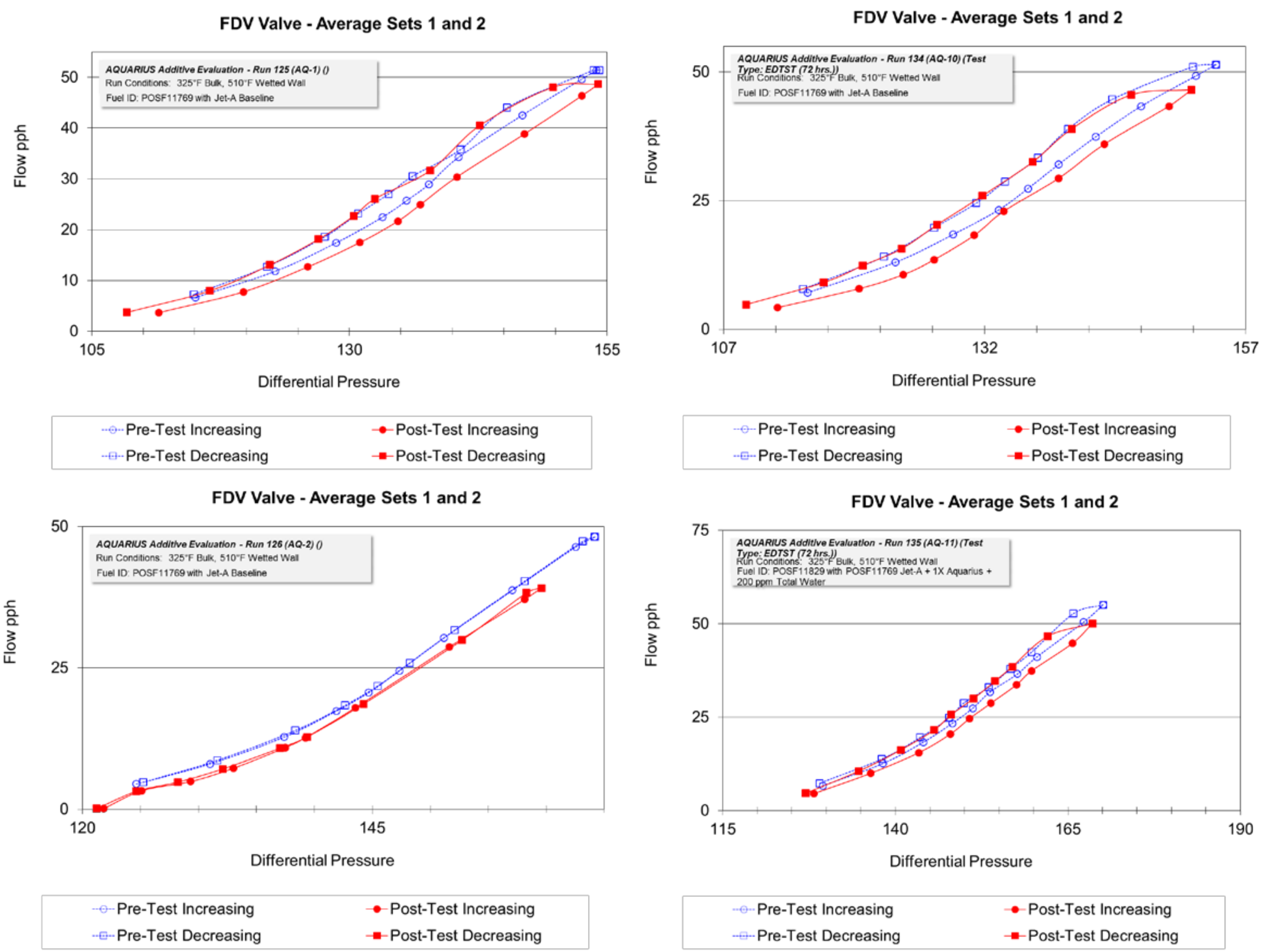


Figure 37 – Comparison of Flow Divider Valve Hysteresis for Run 135 (250 ppm Aquarius + 200 ppm Total Dissolved Water) to Previous Baseline Test Fuel Runs

Figures 38 through 41 show a comparison in the appearance of simulator components for EDTST-mode testing for both baseline and additized fuels. Consistently, the visual appearance of deposition on these components shows more deposition for baseline fuel runs than for additized fuel runs indicating that the Aquarius additive seems to reduce deposition in these components.

Figures 42 through 45 show a comparison in the appearance of simulator components for GDTC-mode testing for both baseline and additized fuels. As with components from the EDTST-mode testing the visual appearance of deposition on these components shows more deposition for baseline fuel runs than for additized fuel runs indicating that the Aquarius additive seems to reduce deposition in these components.

As described earlier in this report, at the end of the program, two EDTST-mode runs were repeated using the baseline and the additized fuel. The purpose of these two runs was to determine if the overall thermal stability characteristics of the fuel had changed over the course of the program. In previous sections, it has been described that no such degradation appeared to occur based on data from the Servo Valve and Flow Divider Valve hysteresis measurements (see Section 5.3.3). Figures 46 through 50 show a comparison of simulator components for start of program runs and end of program runs. Consistent with Section 5.3.3 findings, these figures show, firstly, a remarkable consistency between start of program results and end of program results and secondly, that the deposition for components exposed to Aquarius additive seems to be reduced compared to baseline fuel results. This is consistent with other findings in this program.

Figures 51 through 55 show the results of Run 135 (see Section 5.2.12 for a full description of Run 135 and the rational for performing this test). In these figures, images for simulator components are compared to images from previous baseline and additized runs. The reader will recall that the additive dosage rate for these comparisons was 1000 ppm, not 250 ppm. These figures show that for all simulator components except the Torque Motor Screen, the appearance of deposition for Run 135 is virtually identical to the baseline fuel deposition. In the Torque Motor Screen, there appears to be more deposition than in any of the previous baseline or additized fuel runs. However, in Figures 24 and 25 the accumulated mass and total effective carbon measurements are entirely consistent with measurements made on baseline fuel. It is likely that the presence of 200 ppm water lead to a darker appearance of the deposit yet no significant increase in the actual amount of deposition on the screen. It is therefore concluded that even though the appearance of the deposit on the TMS is darker for Run 135, the results remain consistent with previous baseline tests based on total deposit mass and total effective carbon measurements. This is further evidenced by looking at Figure 48 which shows that the TMS deposition for Run 134 (a baseline fuel run accomplished immediately prior to Run 135). This figure shows the TMS deposition to be darker than previous baseline fuel tests. Even though all other data points to no degradation in baseline fuel thermal stability performance, a subtle change may have occurred which was manifest only in the nature and appearance of the deposition in the TMS. In fact, in that same figure, for Run 133 (an additized fuel test with additive at 1000 ppm), the appearance of deposition in the TMS appears to be slightly more pronounced than for previous additized runs. From this data, it is concluded that there was a subtle shift in the thermal stability performance of the fuel that manifests itself only in the TMS environment and is not discernible by looking at other components. This is an indication that the TMS module is a valuable and discerning addition to the ARSFSS component slate.

Servo Valve Components – EDTST Mode

Run 125 – Baseline

Run 127 – 1000 PPM AQ

Run 126 – Baseline

Run 128 – 1000 PPM AQ

CLEAN SPOOL

Figure 38 – Comparison of Servo Valve Spools, EDTST-Mode, Baseline and Additized Fuels

Nozzle Simulator Components – EDTST Mode

Run 125 – Baseline

Run 127 – 1000 PPM AQ

Run 126 – Baseline

Run 128 – 1000 PPM AQ

CLEAN

Figure 39 – Comparison of Nozzle Simulator Components, EDTST-Mode, Baseline and Additized Fuels

Flow Divider Valve Screen & Body – EDTST Mode

CLEAN	Run 125 – Baseline	Run 126 – Baseline	Run 127 – 1000 PPM AQ	Run 128 – 1000 PPM AQ

Flow Divider Valve Screen

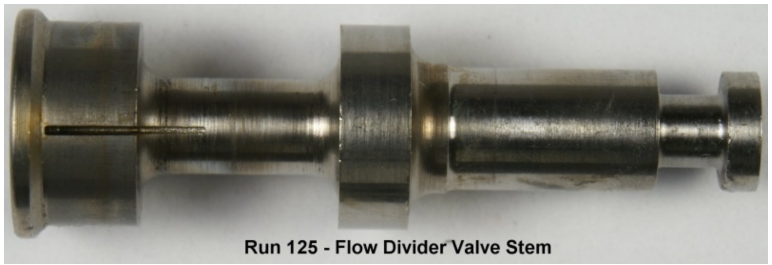
CLEAN	Run 125 – Baseline	Run 126 – Baseline	Run 127 – 1000 PPM AQ	Run 128 – 1000 PPM AQ

Flow Divider Valve Body

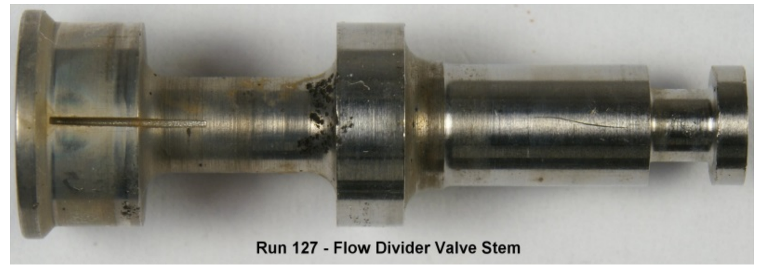
Figure 40 – Comparison of Flow Divider Valve Screen and Body, EDTST-Mode, Baseline and Additized Fuels

Flow Divider Valve Spool – EDTST Mode

Run 125 Baseline



Run 127+1000 PPM AQ



Run 126 Baseline

Run 128+1000 PPM AQ

CLEAN

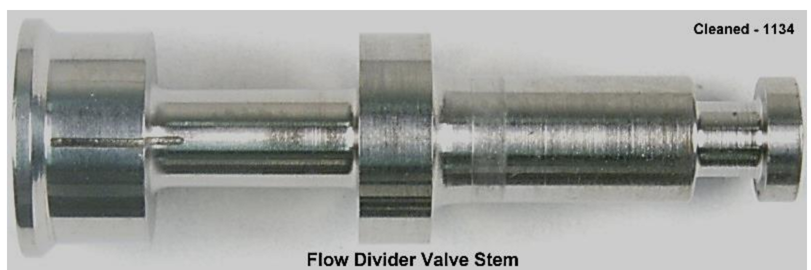


Figure 41 – Comparison of Flow Divider Valve Stem, EDTST-Mode, Baseline and Additized Fuels

Servo Valve Components – GDTC Mode

Run 129 – Baseline

Run 131 – 1000 PPM AQ

Run 130 – Baseline

Run 132 – 1000 PPM AQ

CLEAN SPOOL

Figure 42 – Comparison of Servo Valve Spools, GDTC-Mode, Baseline and Additized Fuels

Nozzle Simulator Components – GDTC Mode

Run 129 – Baseline

Run 131 – 1000 PPM AQ

Run 130 – Baseline

Run 132 – 1000 PPM AQ

CLEAN

Figure 43 – Comparison of Nozzle Simulator Components, GDTC-Mode, Baseline and Additized Fuels

Flow Divider Valve Screen & Body – GDTC Mode

CLEAN | Run 129 – Baseline | Run 130 – Baseline | Run 131 – 1000 PPM AQ | Run 132 – 1000 PPM AQ

Flow Divider Valve Screen

CLEAN | Run 129 – Baseline | Run 130 – Baseline | Run 131 – 1000 PPM AQ | Run 132 – 1000 PPM AQ

Flow Divider Valve Body

Figure 44 – Comparison of Flow Divider Valve Screens and Bodies, GDTC-Mode, Baseline and Additized Fuels

Flow Divider Valve Spool – GDTC Mode

Run 129 Baseline

Run 131+1000 PPM AQ

Run 130 Baseline

Run 132+1000 PPM AQ

CLEAN

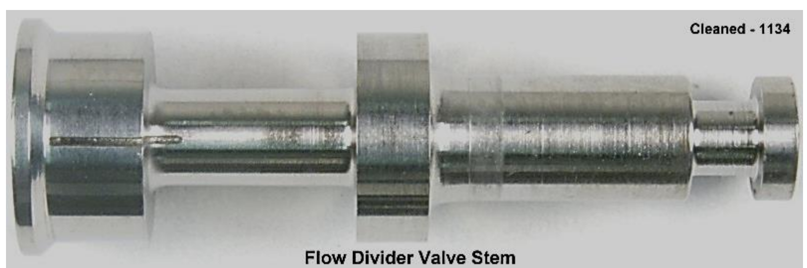


Figure 45 – Comparison of Flow Divider Valve Stems, GDTC-Mode, Baseline and Additized Fuels

Servo Valve Components – EDTST Mode

Run 125 – Baseline

Run 127 – 1000 PPM AQ

Run 126 – Baseline

Run 128 – 1000 PPM AQ

Run 134 – Baseline

Run 133 – 1000 PPM AQ

Figure 46 – Comparison of Start and End Program Servo Valve Spools, EDTST-Mode, Baseline and Additized Fuels

Run 125 – Baseline

Run 127 – 1000 PPM AQ

Run 126 – Baseline

Run 128 – 1000 PPM AQ

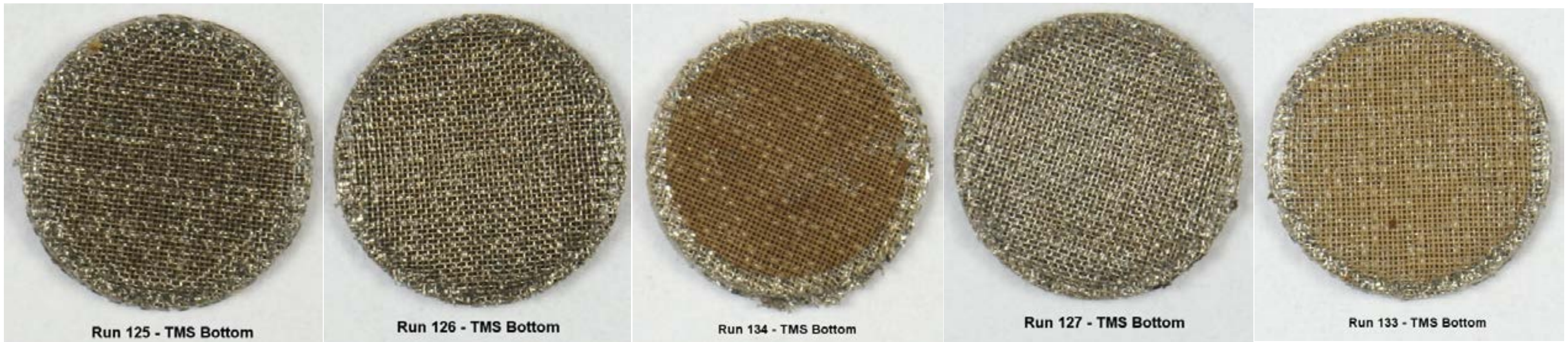
Run 134 – Baseline

Run 133 – 1000 PPM AQ

Figure 47 – Comparison of Start and End Program Nozzle Simulator Components, EDTST-Mode, Baseline and Additized Fuels

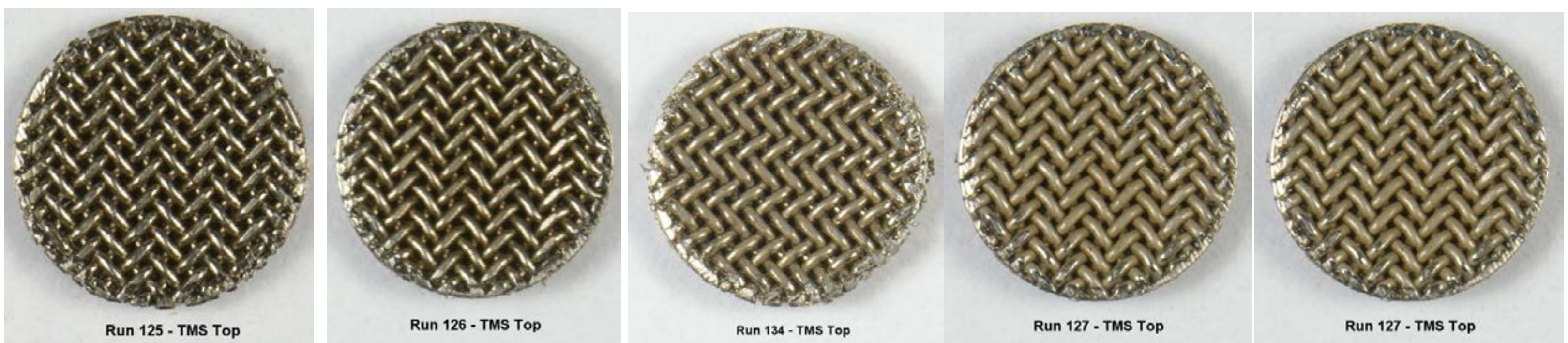
Torque Motor Screen – EDTST Mode

Run 125 – Baseline | Run 126 – Baseline | Run 134 – Baseline | Run 127 – 1000 PPM AQ | Run 133 – 1000 PPM AQ



Torque Motor Screen Bottom

Run 125 – Baseline | Run 126 – Baseline | Run 134 – Baseline | Run 127 – 1000 PPM AQ | Run 133 – 1000 PPM AQ



Torque Motor Screen Top

Figure 48 – Comparison of Start and End Program Torque Motor Screens, EDTST-Mode, Baseline and Additized Fuels

Flow Divider Valve Screen & Body – EDTST Mode

Run 125 – Baseline | Run 126 – Baseline | Run 134 – Baseline | Run 127 – 1000 PPM AQ | Run 133 – 1000 PPM AQ

Flow Divider Valve Screen

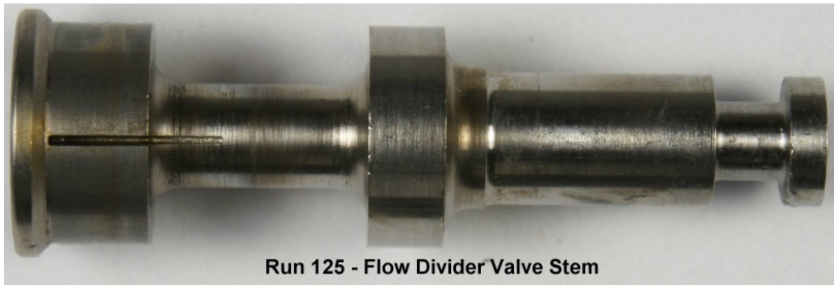
Run 125 – Baseline | Run 126 – Baseline | Run 134 – Baseline | Run 127 – 1000 PPM AQ | Run 133 – 1000 PPM AQ

Flow Divider Valve Body

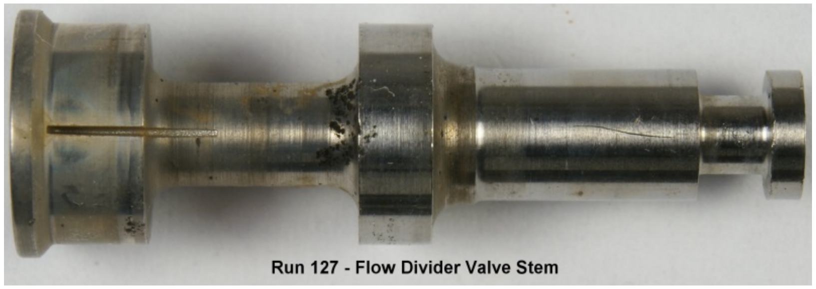
Figure 49 – Comparison of Start and End Program Flow Divider Valve Screens and Bodies, EDTST-Mode, Baseline and Additized Fuels

Flow Divider Valve Spool – EDTST Mode

Run 125 Baseline

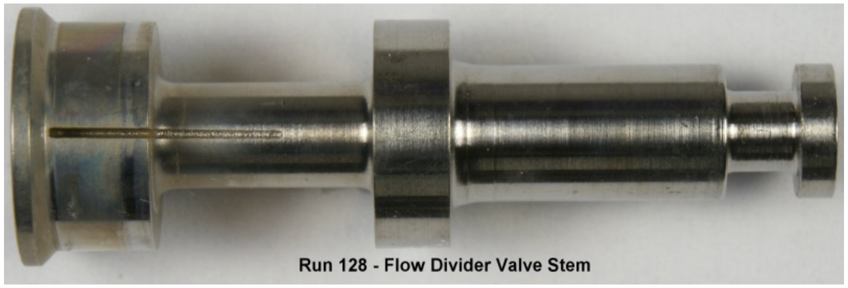


Run 127+1000 PPM AQ



Run 126 Baseline

Run 128+1000 PPM AQ



Run 134 Baseline

Run 133+1000 PPM AQ

Figure 50 – Comparison of Start and End Program Flow Divider Valve Stems, EDTST-Mode, Baseline and Additized Fuels

Servo Valve Components – EDTST Mode

Run 125 – Baseline

Run 127 – 1000 PPM AQ

Run 126 – Baseline

Run 128 – 1000 PPM AQ

Run 135 – 250 ppm AQ + 200 ppm Total Dissolved Water

Figure 51 – Comparison of Run 135 (250 ppm Aquarius with 200 ppm Total Dissolved Water) Servo Valve Spools To Baseline and 4X Additized Fuel

Nozzle Simulator Components – EDTST Mode

Run 125 – Baseline

Run 127 – 1000 PPM AQ

Run 126 – Baseline

Run 128 – 1000 PPM AQ

Run 135 – 250 ppm AQ + 200 ppm Total Dissolved Water

Figure 52 – Comparison of Run 135 (250 ppm Aquarius with 200 ppm Total Dissolved Water) Nozzle Simulator Components To Baseline and 4X Additized Fuel

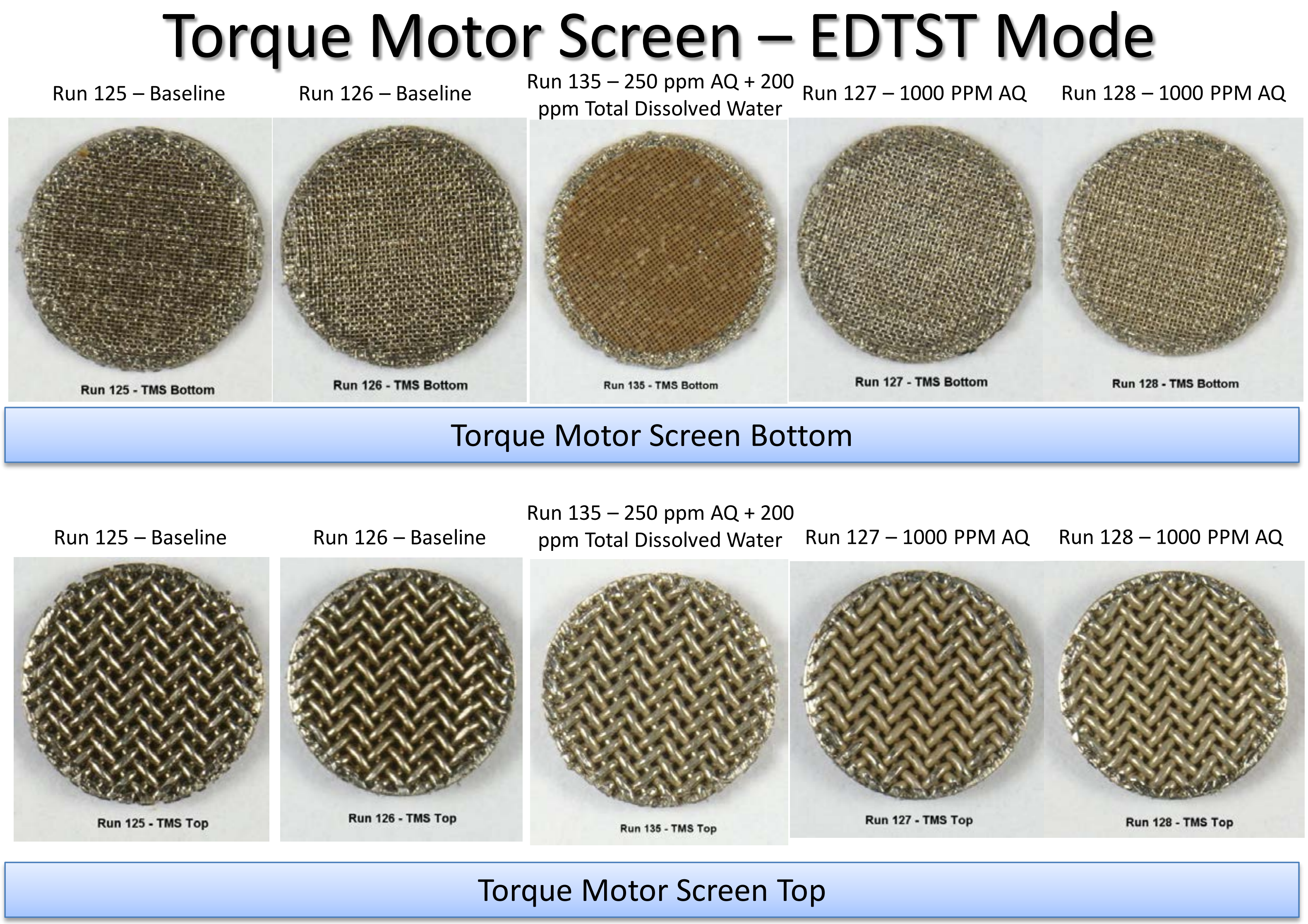


Figure 53 – Comparison of Run 135 (250 ppm Aquarius with 200 ppm Total Dissolved Water) Torque Motor Screens To Baseline and 4X Additized Fuel

Flow Divider Valve Screen & Body – EDTST Mode

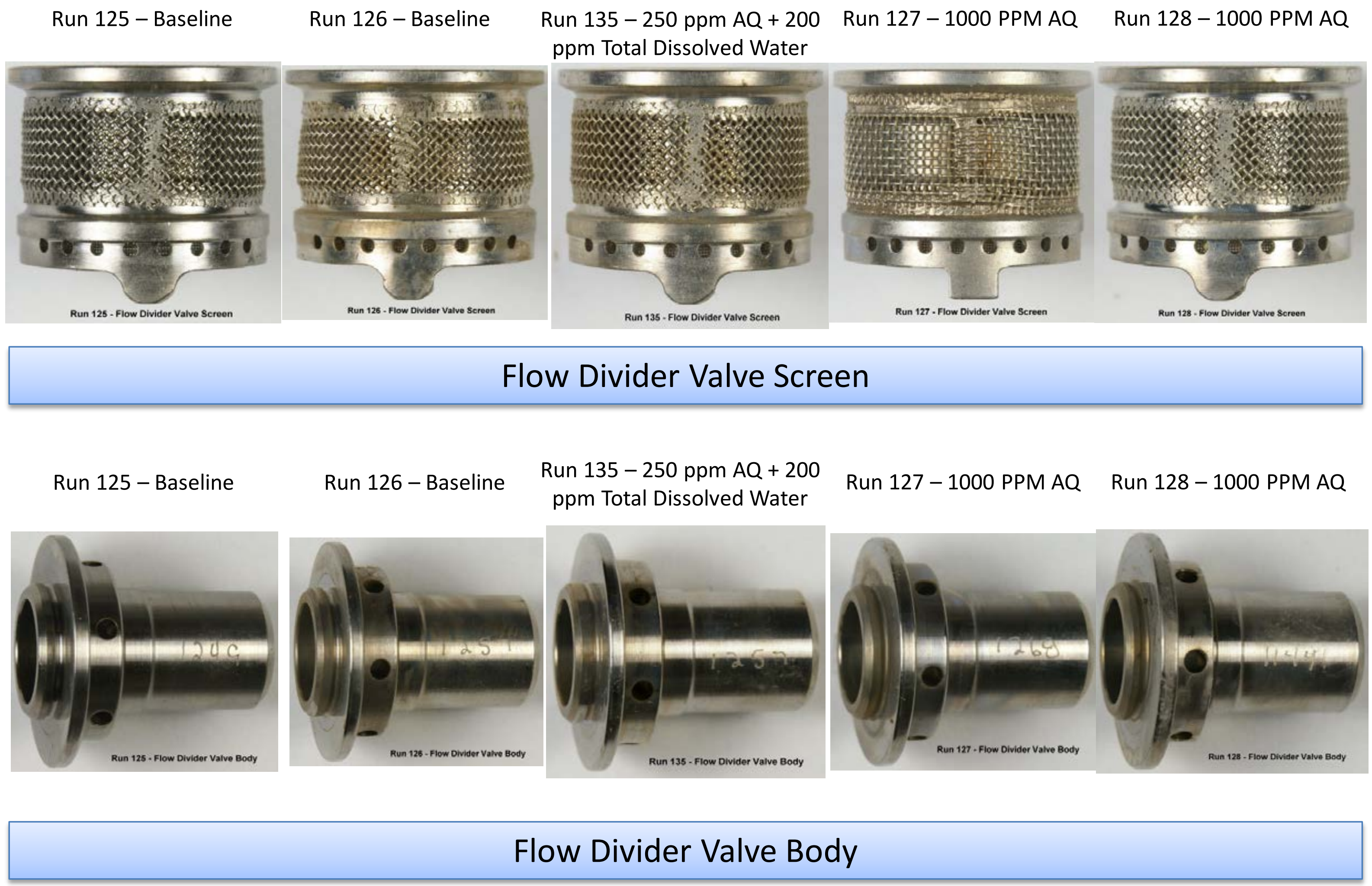
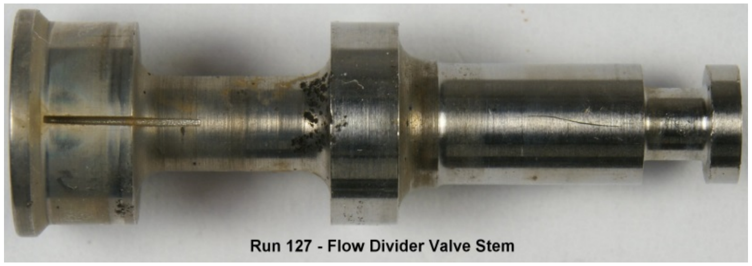


Figure 54 – Comparison of Run 135 (250 ppm Aquarius with 200 ppm Total Dissolved Water) Flow Divider Valve Screens and Bodies To Baseline and 4X Additized Fuel

Flow Divider Valve Spool – EDTST Mode

Run 125 Baseline

Run 127+1000 PPM AQ



Run 126 Baseline

Run 128+1000 PPM AQ

Run 135 – 250 ppm AQ + 200 ppm Total Dissolved Water

Figure 55 – Comparison of Run 135 (250 ppm Aquarius with 200 ppm Total Dissolved Water) Flow Divider Valve Stems To Baseline and 4X Additized Fuel

5.4 Summary of SEM Evaluation of the Torque Motor Screen

TMS screens from fuel tests were analyzed with a JEOL JSM-6460LV scanning electron microscope (SEM) equipped with an IXRF Systems Model 550i. In this work, the SEM was used in three different modes:

1. High Magnification Imaging (HMI) for the structural and morphological assaying of deposits on the TMS
2. X-Ray Mapping (XRM) or "Dot Mapping" to determine the distribution and frequency of individual elements using their characteristic X-ray emissions
3. X-Ray Fluorescence (XRF) to determine the overall elemental distribution of elements

SEM was used to detect material deposited on the screens during fuel testing as well as indicating the surface morphology of the deposits. The IXRF system was then used to identify the elemental make-up of these deposits. It should be noted that SEM is a surface analysis technique so the elemental analyses performed relate only to the makeup of the first few atomic layers of the surface. There are numerous "dot maps" of the deposits on the screens that show the distributions of the elements over the surface being analyzed. The dot maps are extremely useful since it reveals if certain elements are universally distributed or are concentrated in certain areas that can reveal features like oxides or unique particles or concentrations with-in the deposits themselves. However, it should be noted that more advanced methods such as X-Ray Diffraction (XRD) would be needed to identify specific compounds. Inductively Coupled Plasma (ICP) analysis of fuel samples was also used to determine which metals would likely be deposited on the TMS. The results of these analyses are discussed in Section 5.5.

Appendix B shows a series of SEM pictures and elemental analyses from the TMS for Runs 129 through 135. Run 131 is not included because a miscommunication resulted in the TMS from that run being subjected to LECO carbon burn-off before it could be evaluated by SEM.

5.4.1 <u>High Magnification Imaging</u>

Of particular interest amongst these pictures are Figures 83 through 85. Figure 83 shows the condition of a new screen compared to screens from Run 129 (Baseline Jet A) and Run 131 (Jet A + 1000 ppm Aquarius). Both of these runs were in GDTC (missions) mode. Also included is a picture of a post-burn-off screen. The magnification of these pictures is between 18X and 30X.

These pictures show a slightly cleaner screen when the Aquarius additive is present compared to the baseline non-additized fuel as evidenced by the sharper cleaner edges on the wires for the additized fuel. To get a better look, Figure 84 shows screens from Runs 129 and 133 (baseline and additized fuels respectively) at 200X magnification. It can be seen that the clean, post-burn-off and Aquarius additized screens appear very similar while the baseline screen shows a heavier deposition. This heavier deposition interpretation is based on the lack of white areas on the wires. These white areas are where very little organic deposition is located revealing bare metal (the bare metal edges have a tendency to show up as white 'glare' in the pictures. The lack of these 'glare' areas in the Run 129 picture indicate that deposition is heavier on the wire surface. Figure 85 is similar to Figure 84 except that the screen for Run 135 (baseline Jet A+250ppm Aquarius+200 ppm water) is shown. Again, the presence of the 'glare' white areas in the picture indicates bare metal exposure.

5.4.2 X-Ray Mapping

In Figure 56, the SEM image shows the field of view for the XRM procedure. The metal wire mesh is clearly visible. The high intensity Fe, Cr, Mn and Ni X-ray distributions are clearly associated with the metal mesh and indicate at least the surface metallurgical composition. Note that there is some S associated with the surface of the screen, evenly distributed but also more intense when associated with the particle captured by the screen. Cu is also faintly visible.

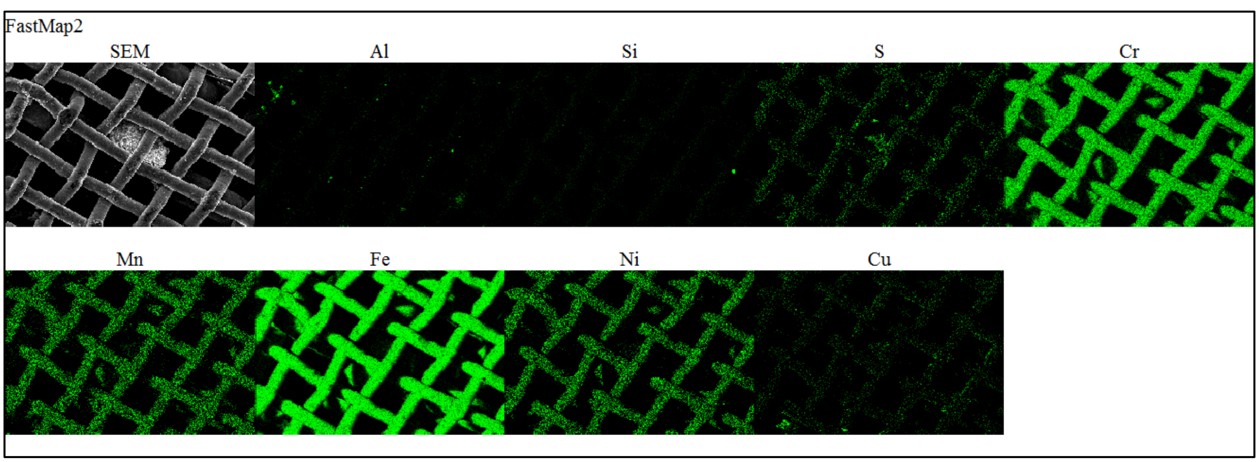


Figure 56 - Clean TMS Typical Post Burn-off SEM Back Scatter Image (Image Intensity = Element Frequency)

In Figures and58, XRM post- Aquarius testing reveals similar Fe, Cr, Mn, Ni and S distributions as in the post-burn-off baseline fuel example (Figure 56). In Figure 57, there are some Al hot spots, one Si hot-spot and a very faint distribution of K associated with the screen metallurgy. At a much higher magnification (Figure 58), the Fe/Cr association remains, the Si can be seen to be associated with a particle sitting on the mesh but the K has almost completely disappeared indicating its trace nature.

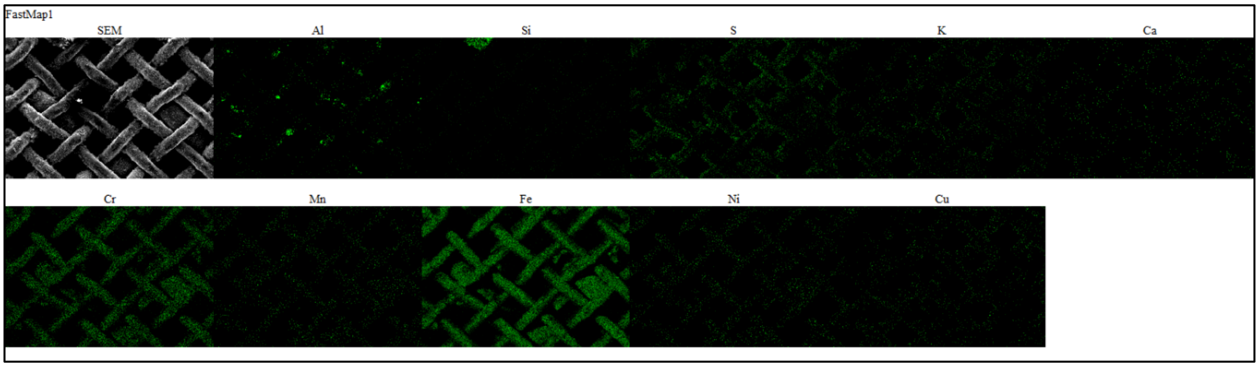


Figure 57 - SEM of Hexane-Rinsed TMS (Back Side), Run 133 (Jet A+1000 ppm Aquarius) at 200X

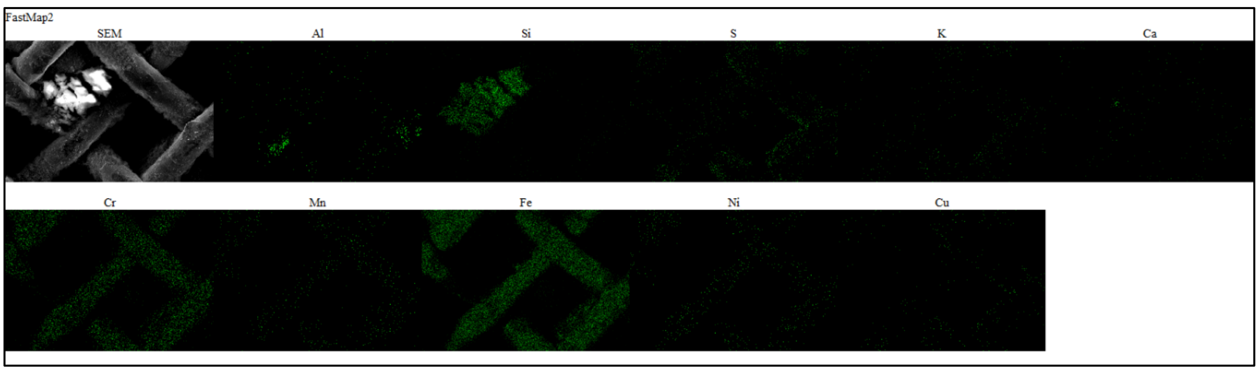


Figure 58 - SEM of Hexane-Rinsed TMS (Back Side), Run 133 (Jet A+1000 ppm Aquarius) at 500X

5.4.3 <u>X-Ray Fluorescence</u>

Table 11 and Table 12 summarize the elemental analysis data from Appendix B. Note that in estimating the actual masses of elements, no account has been taken of the non-detectable elements such as O or H. Consequently there may be some distortion in the data where, for instance, the element was present as an oxide or a halide, a sulphate or a carbonate. This will affect the analysis accuracy.

The more typical baseline fuel result produced a total of 365.5 µg of non-carbon deposit compared to 314 µg when the Aquarius additive is in use in this baseline fuel. Runs 129 and 131/132 provide the best back-to-back comparisons produced in this work. There are some very small differences in elemental make-up and taken together with the XRM results overall conclusions would be that non-C elemental deposition is essentially similar in both baseline and additized fuels.

Table 11 – Elemental Analysis Summary, Runs 129, 131 and 132 (GDTC Mode)

ELEMENT	Run 129, GDTC Baseline Jet A		Run 131/132, GDTC 1000	
	Avg Concen. In Deposit (Wt%)	Actual Mass, µg 365.50	Avg Concen. In Deposit (Wt%)	Actual Mass, µg 314.99
Al	1.303	4.76	1.069	3.37
Si				
P				
S	4.271	15.61	12.08	38.05
Cl				
K			7.923	24.96
Ca			0.415	1.31
Cr	17.003	62.15	12.318	38.80
Mn			0.951	3.00
Fe	61.657	225.35	41.778	131.60
Co				
Ni	8.225	30.06	5.249	16.53
Cu	7.541	27.56	17.902	56.39
TOTAL		365.50		314.00

Table 12 shows a similar elemental analysis summary for Runs 133 through 135. As previously mentioned, *it should be noted that the total mass deposition on the TMS for Run 134 is substantially lower than for the previous baseline fuel runs in EDTST mode. This data point may be correct or it may be in error – it is not possible to prove for certainty. However, given that the value is much lower than any of the test runs regardless of mode or fuel type, it is very likely that this value is an anomaly and not the correct value.*

The differences between Baseline and Aquarius test runs are interesting. For the elements that appear in both test runs there is a randomness in terms of frequency. In the Aquarius test run only, K, Ca and Mn appear as measurable masses. This is somewhat contradictory to the XRM data that showed almost no K in either test run and the prevalence of Mn in the Baseline test run. With such small amounts present it can be concluded that despite the peak intensities shown in Figures 57 and 58, the actual levels are low and of random distribution.

Table 12 – Elemental Analysis Summary, Runs 133-135 (EDTST Mode)

ELEMENT	Run 133, EDTST 1000 ppm AQ		Run 134, Baseline Jet A		Run 135, 250 ppm AQ + 200 ppm Water	
	Avg Concen. In Deposit (Wt%)	Actual Mass, µg 151.00	Avg Concen. In Deposit (Wt%)	Actual Mass, µg 14.90	Avg Concen. In Deposit (Wt%)	Actual Mass, µg 203.46
Al	1.5395	2.32	1.09	0.16	2.38	4.84
Si	2.935	4.43	0.47	0.07	1.11	2.26
P					1.52	3.09
S	4.42	6.67	4.67	0.70	6.58	13.39
Cl						
K	1.521	2.30			2.44	4.96
Ca	0.164	0.25				
Cr	16.0425	24.22	16.57	2.47	15.24	31.01
Mn	1.1715	1.77	1.3	0.19	1.11	2.26
Fe	58.468	88.29	60.11	8.96	55.85	113.63
Co					0.25	0.51
Ni	7.317	11.05	7.67	1.14	6.47	13.16
Cu	6.421	9.70	8.55	1.27	7.05	14.34
TOTAL		151.00		14.96		203.46

The data in this table shows once again a random variable and very low level of metallic deposition. P appears in the 250 ppm Aquarius run but not in any others. This may originate from the water that was used in this run but again might just be "noise". K appears to be associated with the Aquarius runs but the levels do not correlate with the actual levels of additive used. These are very low levels and again, might just be "noise". Ca appeared in the 1,000 ppm Aquarius test run only and Co in the 250 ppm Aquarius test run.

Note: Any metallic species bound up within the bulk carbonaceous deposit will not be assayed during the carbon burn-off analysis as this method detects CO_2 only.

5.5 Summary of ICP Analysis

ICP yields the relative contributions of individual elements to the overall population of elements present in a sample. When used with suitable standard solutions, the method can be highly quantitative. In this study, the method was used only semi-quantitatively. Appendix C provides ICP analysis data as provided directly from BASF. Three samples of fuels typically used in this program were sent to BASF. Sample 117690-1 was a sample drawn from Tank S-3 and represents the typical baseline neat fuel used in this program. Sample 11769-2 was drawn from Tank S-16 which was the holding tank for the baseline fuel for this program. Recall that fuel from S-16 was used to replenish Tanks S-3 and S-4 during ARSFSS operations. Sample 11770 represents the Aquarius-additized fuel typically used for this program. Aquarius concentration in this sample was 1000 ppm (4X).

In all three of these samples, the most noticeable elements present in ppm vs ppb levels were

1. Ca at greater than 2 ppm
2. Si at less than 1 ppm
3. Relatively high amounts of Al, Mg, Ni, Ti and Zn (baseline fuel only)

Further ICP analysis was carried out on three “post-test” samples of Baseline and Additive (1,000ppm and 250ppm) treated fuels. These were taken from the Burner Feed Arm (the hottest part of the rig). In all three samples high levels of Ca and Si are still present, as well as Sb, Ni and Sn. These last elements are more than likely contaminants from sample cans used in sample transport. Both Ca and Si are observed in the Aquarius treated fuel runs where there is less C deposited on the TMS in agreement with the SEM data.

Overall, from Tables 11 and 12 as well as all the SEM and ICP data, it seems that there are contaminants in the fuel mainly at ppb levels, which are caught by the TMS screen regardless of which fuel is being tested. These are more easily detected with the Aquarius runs due to the relatively lower Carbon deposition allowing the metals to be seen more easily.

5.6 Is There Evidence of Aquarius Having Cleaning Effect on Components?

The short answer is “No.” During the post-program data review and results evaluation for this program, it was asked if the data collected could be used to determine if the apparent reduced deposition in tests using 4X Aquarius additive was truly reducing deposition or perhaps performing some cleaning action on deposits after they were attached to the deposition surface.

To answer this question, plots for wetted wall temperature in the Burner Feed Arm for each additive run were prepared. As deposition is laid down in the BFA, it acts as an insulator to heat transfer from the outer tube surface to the fuel. When this insulation is present, a corresponding rise in wetted wall temperature in the area of the deposit is observed due to the insulating effect of the deposition. The effect then is to observe a slight wetted wall temperature rise as the test run proceeds and more deposit is laid down. Heavier the deposition results in a larger the temperature rise at the wetted wall surface.

EDTST-Mode runs are time-based so data for thermocouple TE324 (the typical hot spot in the BFA) was plotted against EDTST-mode run time. Figure 59 shows this plot. The data for each EDTST-mode Run (Runs 125-128) was trended with a linear trend line to determine the overall temperature rise at the TE324 hot spot (Section 8 on the BFA Tube) for that Run. This overall temperature rise was related to the Total Effective BFA Carbon Deposition (the total amount of carbon deposited in the BFA across its entire length, adjusted for background carbon inherent in the tube itself). The Effective Carbon Deposition at the hot spot was also determined. Table 13 shows a tabulation of this data.

GDTC-Mode runs are mission-based so data for thermocouple TE324 (the typical hot spot in the BFA) at each mission during the Low Power Cruise Mission Segment (LPCruise) was plotted against GDTC-mode Mission number. Figure 60 shows this plot. The data for each GDTC-mode Run (Runs 129-132) was trended with a linear trend line to determine the overall temperature rise at the TE324 hot spot (Section 8 on the BFA Tube) for that Run. This overall temperature rise was related to the Total Effective BFA Carbon Deposition (the total amount of carbon deposited in the BFA across its entire length, adjusted for background carbon inherent in the tube itself). The Effective Carbon Deposition at the hot spot was also determined. Table 14 shows a tabulation of this data.

Table 13 - Comparison of Wetted Wall Temperature Rise and Deposition for Runs 125 – 128, EDTST-Mode Runs, Baseline and 4X Aquarius

Run	Run Type	Max WWT Rise °F	Total Effective BFA Carbon, µg	BFA Carbon Hot Spot, µg/cm²
125	Baseline	3	2837	396
126	Baseline	9.4	3822	522
127	4X Aquarius	1.4	44	7
128	4X Aquarius	-1	206	8.3

Table 14 – Comparison of Wetted Wall Temperature Rise and Deposition for Runs 129 – 132, GDTC-Mode Runs, Baseline and 4X Aquarius

Run	Run Type	WWT Rise °F	Total Effective BFA Carbon, µg	BFA Carbon Hot Spot, µg/cm²
129	Baseline	7.6	2979	430
130	Baseline	3.3	2798	487
131	4X Aquarius	2.5	4	0.1
132	4X Aquarius	4.3	67	13.6

Based on these tables and plots, the temperature rise in the BFA due to deposition is less for Aquarius-additized fuel than for the baseline fuel. Total Effective BFA Carbon and Effective BFA Hot Spot Carbon for Aquarius-additized fuel is also substantially less than for baseline fuels. The data lines in the plots do not exhibit any evidence of deposition occurring on the tube as this would be evidenced by large temperature swings during the run. Instead, the temperature changes that are observed during the Runs are similar between baseline and additized fuel runs and are consistent with subtle changes in fuel flow through the BFA tube itself.

In addition to the temperature and deposition data, there is nothing in the additive chemistry that would lead to the conclusion that the additive has a ‘cleaning’ effect on deposition.

Based on these plots and an understanding of the chemistry of the Aquarius additive, it is not likely that the additive is causing cleaning or removal of deposition. It is more likely that the presence of the additive is either reducing the amount of deposition generated or preventing the deposit from adhering to wetted surfaces.

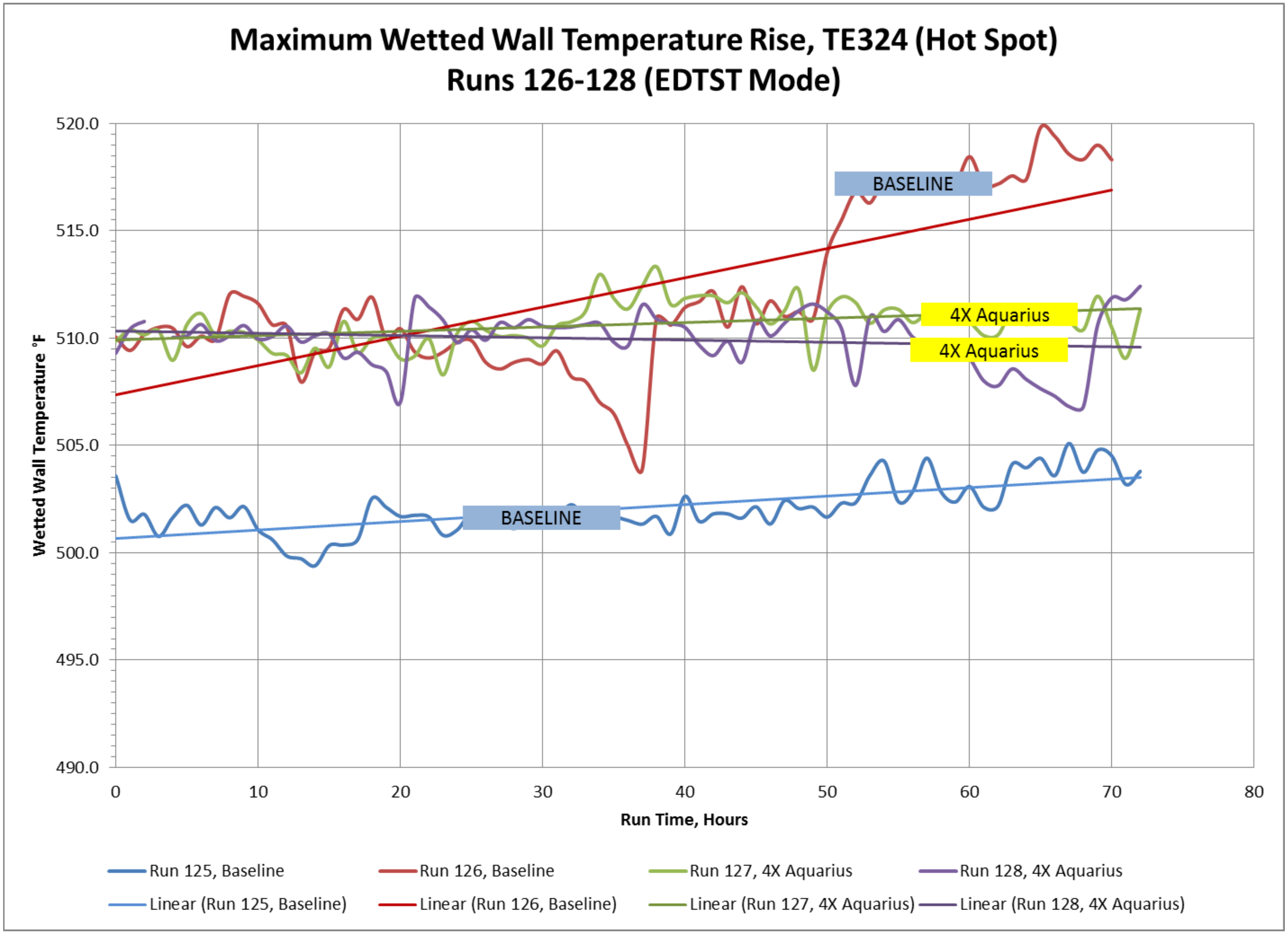


Figure 59 - BFA WWT Temperature Rise and Carbon Deposition, EDTST-Mode

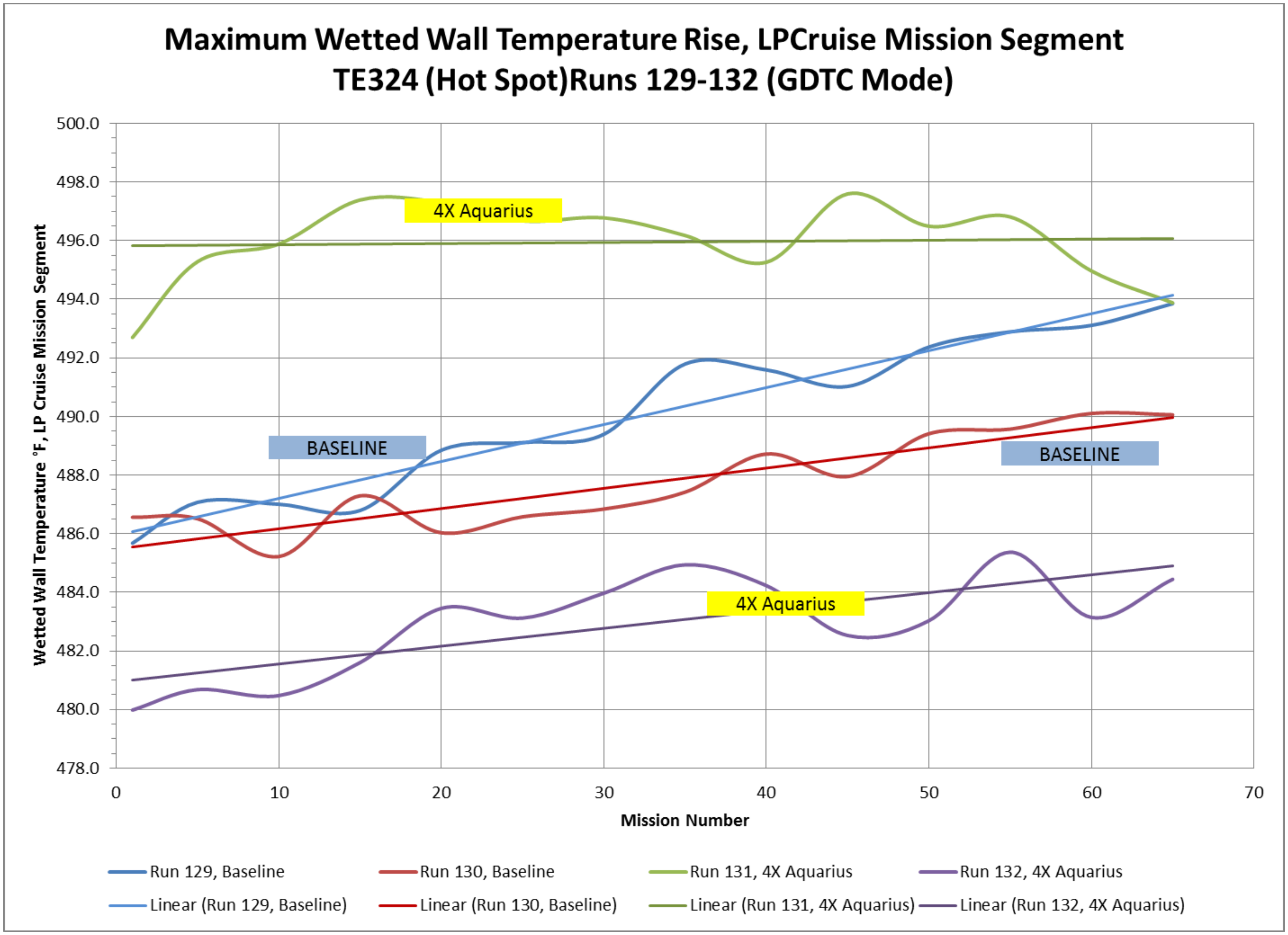


Figure 60 - BFA WWT Temperature Rise and Carbon Deposition, GDTC-Mode

6.0 Conclusions and Recommendations

Aquarius Water Scavenger Additive was evaluated for its impact on the thermal stability characteristics of Jet A aviation turbine fuel using a variety of test devices – Quartz Crystal Microbalance (QCM), Jet Fuel Thermal Oxidation Tester (JFTOT) and the Advanced Reduced Scale Fuel System Simulator (ARSFSS). The additive was evaluated in these devices at the anticipated commercial usage concentration of 250 ppm by volume and at four times (4X) this concentration, i.e. 1000 ppm. A synopsis of the results of this testing are:

1. In a total of 10 ARSFSS runs in both EDTST and GDTC modes, when compared to the baseline non-additized fuel, fuel additized with Aquarius at 4X (1000 ppm) demonstrated:
    a. Reduced Servo Valve hysteresis resulting in improved valve operational characteristics
    b. Reduced Flow Divider Valve hysteresis resulting in improved valve operational characteristics
    c. Reduced deposition in the Burner Feed Arm
    d. Reduced apparent deposition (visual appearance) in Servo Valve, Flow Divider Valve and TMS components
    e. Reduced hot-spot temperature rise in the BFA
2. In QCM tests, Aquarius-additized fuel demonstrated:
    a. No change in deposition formation compared to the baseline neat fuel.
    b. No change in oxygen consumption characteristics compared to the baseline fuel
    c. No change in either oxygen consumption or deposit formation when compared to NATO F-24 fuel (containing FSII, CI/LI and SDA).
3. In JFTOT tests the Aquarius-additized fuel demonstrated no reduction in JFTOT Breakpoint temperature.
4. In specification test analyses performed, the Aquarius-additized fuel showed no differences in measured properties (except WSIM and Water Reaction Interface Rating) when compare to the non-additized neat baseline fuel. Changes in WSIM and Water Reaction Interface Rating are expected by the very nature of the additive and how it works.

5. SEM and ICP/MS evaluations of Aquarius-additized fuel showed only inconsequential changes in some elements.

Based on the testing performed, Aquarius Water Scavenger Additive has no apparent or discernible negative impact on fuel thermal stability or other characteristics.

Appendix A – Fuel Certificates of Analysis and Shipping/Receiving Documents

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Jun. 18. 2014 9:33AM No. 5329 P. 1/2

Toledo Refining Company

Toledo Refinery Laboratory
P.O. Box 920
Toledo, Ohio 43697

Phone: (419) 698-6827
Fax: (419) 698-6948

Cleveland Sunoco Logistics
Tank 64

6-14-14

CERTIFICATE OF ANALYSIS

PRODUCT	JET A	CERT. NUMBER:	14-0763
TANK NUMBER:	T437	BATCH NUMBER:	T437-0800-061314
SAMPLE DATE:	06/13/14 08:00		

TEST	ASTM METHOD	RESULTS	
Gravity, API	D4052		
Low Value		42.7	42.7
High Value		42.7	
Average Value		42.7	
Distillation, F:	D86		
10% recovered		338	
20% recovered		354	
50% recovered		392	
90% recovered		459	
End Point		499	
Residue %		1.0	
Loss %		0.5	
Flash Point, F	D56		
Low Value		112	112
High Value		115	
Average Value		113	
H2O Reaction	D1094		
Interface rating		1	
Water Separation Index	D3948	100	97
Electrical Conductivity, Picosiemens/meter @ 70 F	D2624	0	
Corrosion @ 212F-2 Hrs	D130	1A	1A
Smoke Point, mm	D1322	20	
Thermal Stability	D3241		
Pressure Drop, mm/Hg	Run @	0	
Tube Deposit	275 C	<1	
Freeze Point, F	D5972	-70	-67.6
Viscosity @ -4 F, Cs	D445	4.1	
Existent gum, mg/100 ml	D381	0	
Total Sulfur, wt.%	D2622	0.00	
Mercaptan sulfur, wt. %	D3227	0.000	
Total acidity, mg KOH/g	D3242	0.00	
Aromatics, vol. %	D1319	20	
Naphthalenes, vol. %	D1840	0.7	
Net Heat of Combustion, BTU/LB.	D3338	18535	
Saybolt Color	D6045	30	
Appearance @ 77 F, procedure 2	D4176	1	0
Particulate matter, mg/l	D5452	0.0	
Filtration Time (minutes)		6	OK
Water content, ppm	D4377	58	
Visual Color Tint (rating)		WATER WHITE	

Comments:

* Antistatic to be added to Terminals.

Meets ASTM D1655 Standard for Aviation Turbine Fuels.

Kathy McCabe (signature)

Kathy McCabe
Fuels Quality Control

7712

Sunoco Logistics

SXL CLEVELAND - 20501
3200 Independence Road
Cleveland OH 44105
(216)883-6105 TCN: T340H3160
EPA FN: 986481410

PAGE 1 of 1

For Product Emergency – Spill, Leak, Fire or Exposure. Call CHEMTREC 800-424-9300 Day or Night

Date: 06/30/14
BOL #: 0278787

See Reverse Side for Hazard Warnings, Initial Emergency Response Guide, and Other Information

Bill To:
0000000055
Husky Energy Jet Accounts

Ship To:
1256571374
Ascent Aviation - OH

VARIOUS
OH

P.O.# :
Order #: 0000000000

Supplier:
0000000035
Husky Marketing & Supply Co
1150 South Metcalf St.
Lima
OH 45804

Terminal #: 0020501
Folio #: 06/030

Freight Terms: COLLECT

PRODUCT	MATERIALS DESCRIPTION	API	UOM	GROSS	TEMP	GRAV	NET
	UN1863, Fuel Aviation, Turbine Engine 3, PG III, ERG#128, 1 T/T						
402100	JET A TURBINE FUEL		gls	7099	75.1	42.7	7047

This product has been certified to meet all ASTM D 1655 specification requirements.

Meter(s): 050101
Tank(s): 0064

0278787

Carrier: HLXT HILLTRUX TANK LINES, INC.
200 Rosemont Rd., P.O. Box 696
North Jackson
OH 44451
3305383700

Tractor #:
Trailer #: 0045841

Seal(s):

Load Start: 06/30/14 07:07 Stop: 06/30/14 07:27

Vehicle Insurance Expires: 12/01/14

Vapor Tightness Test Due: 01/02/15

Liability Insurance Expires: 12/01/14

~~Workers Comp. Expires: 08/31/14~~

Carrier has loaded and accepted the above-named materials and certifies that the container supplied for this shipment is a proper container for the transportation of this commodity under DOT regulations and driver acknowledges Emergency Response Guide Information received on reverse side of this document. We, the undersigned company, are in compliance with the requirements specified in 49CFR172, and Specifically the new requirements outlined in the Federal Register, Vol. 68, No. 57 dated Tuesday, March 25, 2003 regarding security and related employee training. We further understand that compliance with the Department of Transportation security regulations is mandatory for continued access to Sunoco Partners Marketing and Terminals L.P. properties.

This is to certify that the herein named materials are properly classified, described, packaged, marked and labeled, and are in proper condition for transportation according to the applicable regulations of the department of transportation.

Signature: Jared Houston Date:

Driver: JARED HOUSTON

Driver #: 00026242

Sun-42069-20-B

Appendix B – SEM Analysis

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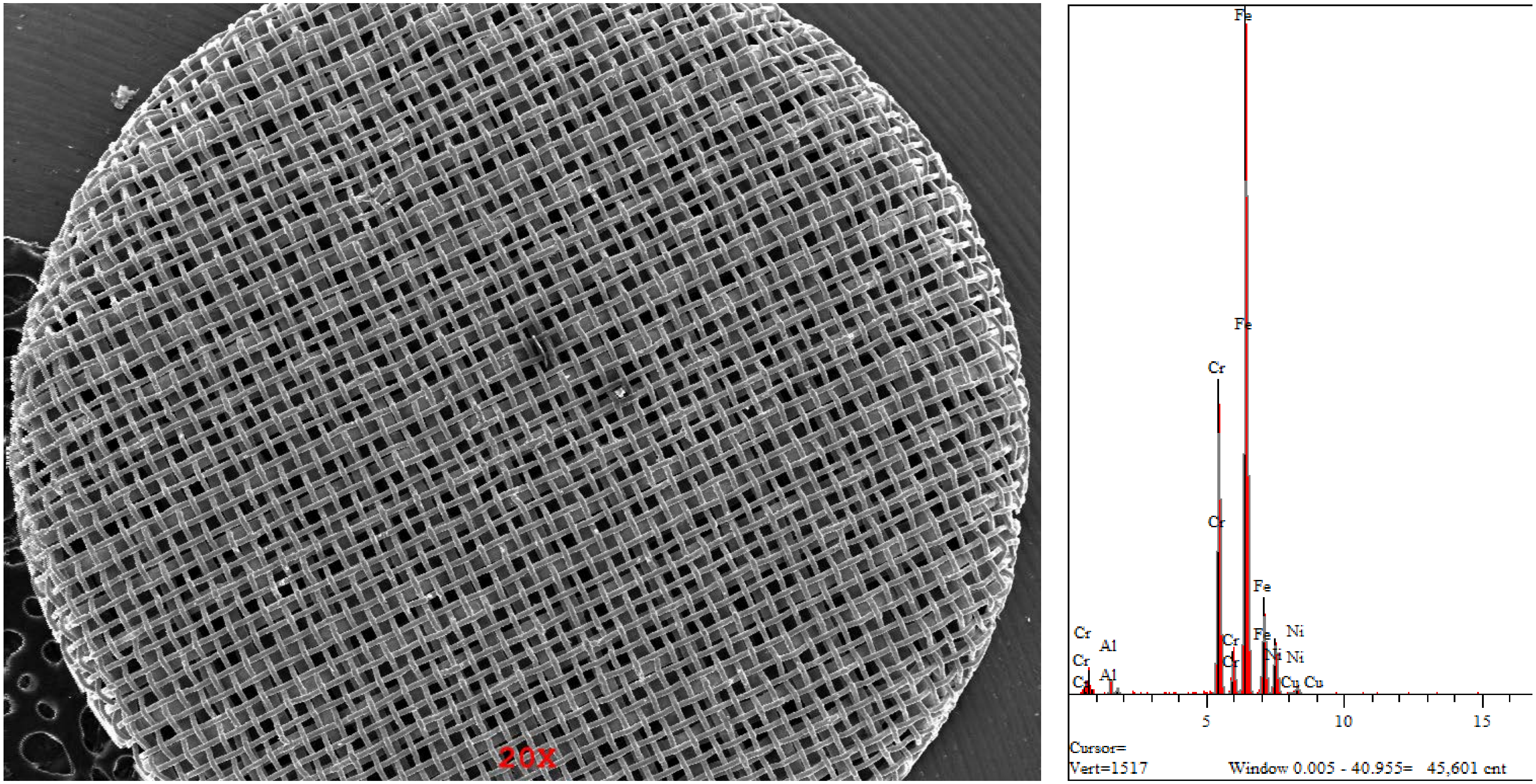
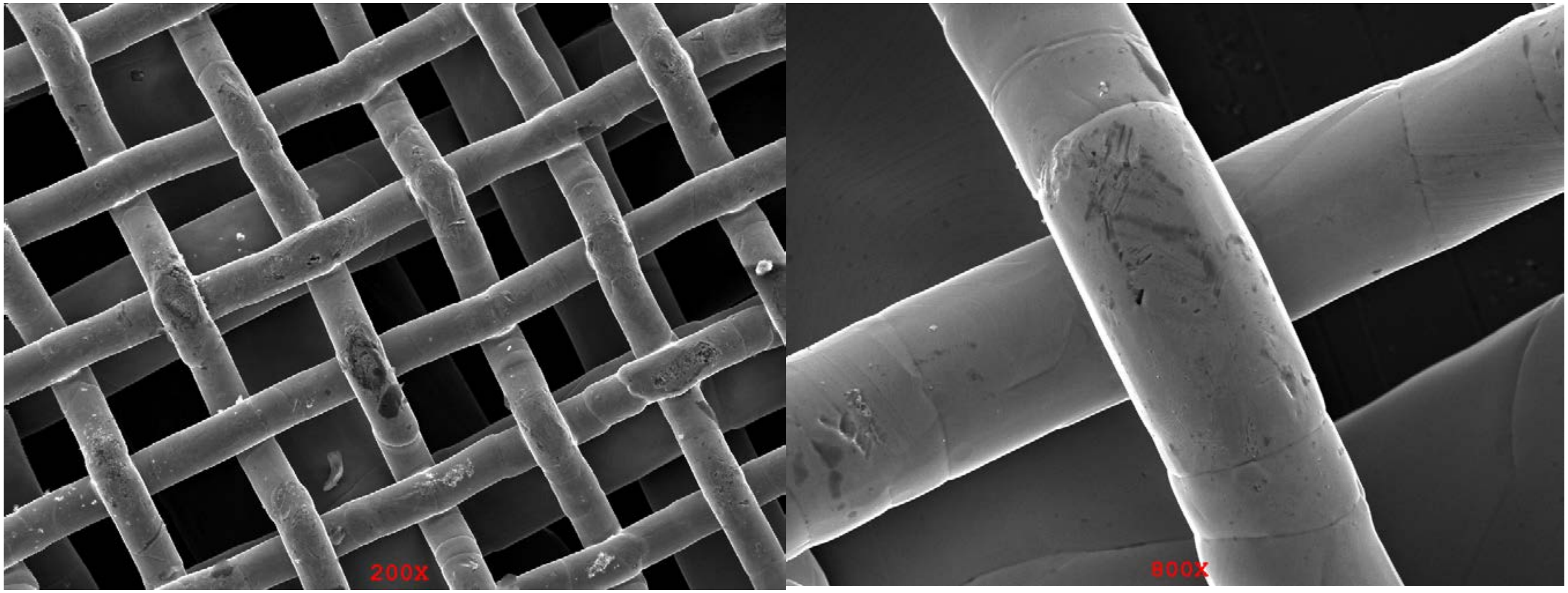


Figure 61 – SEM (20X) and Elemental Analysis of a Clean, Unused Torque Motor Screen (Typical)



Elt.	Line	Intensity (c/s)	Conc	Units	Error 2-sig	MDL 3-sig	
Al	Ka	6.65	1.563	wt.%	0.249	0.297	
Si	Ka	2.65	0.476	wt.%	0.177	0.246	
Cr	Ka	148.12	19.120	wt.%	0.430	0.217	
Fe	Ka	331.11	68.545	wt.%	0.998	0.342	
Ni	Ka	31.82	9.630	wt.%	0.508	0.385	
Cu	Ka	1.87	0.665	wt.%	0.299	0.415	
			100.000	wt.%			Total

Figure 62 - SEM (200X and 800X) of the Clean, Unused Torque Motor Screen (Figure 61)

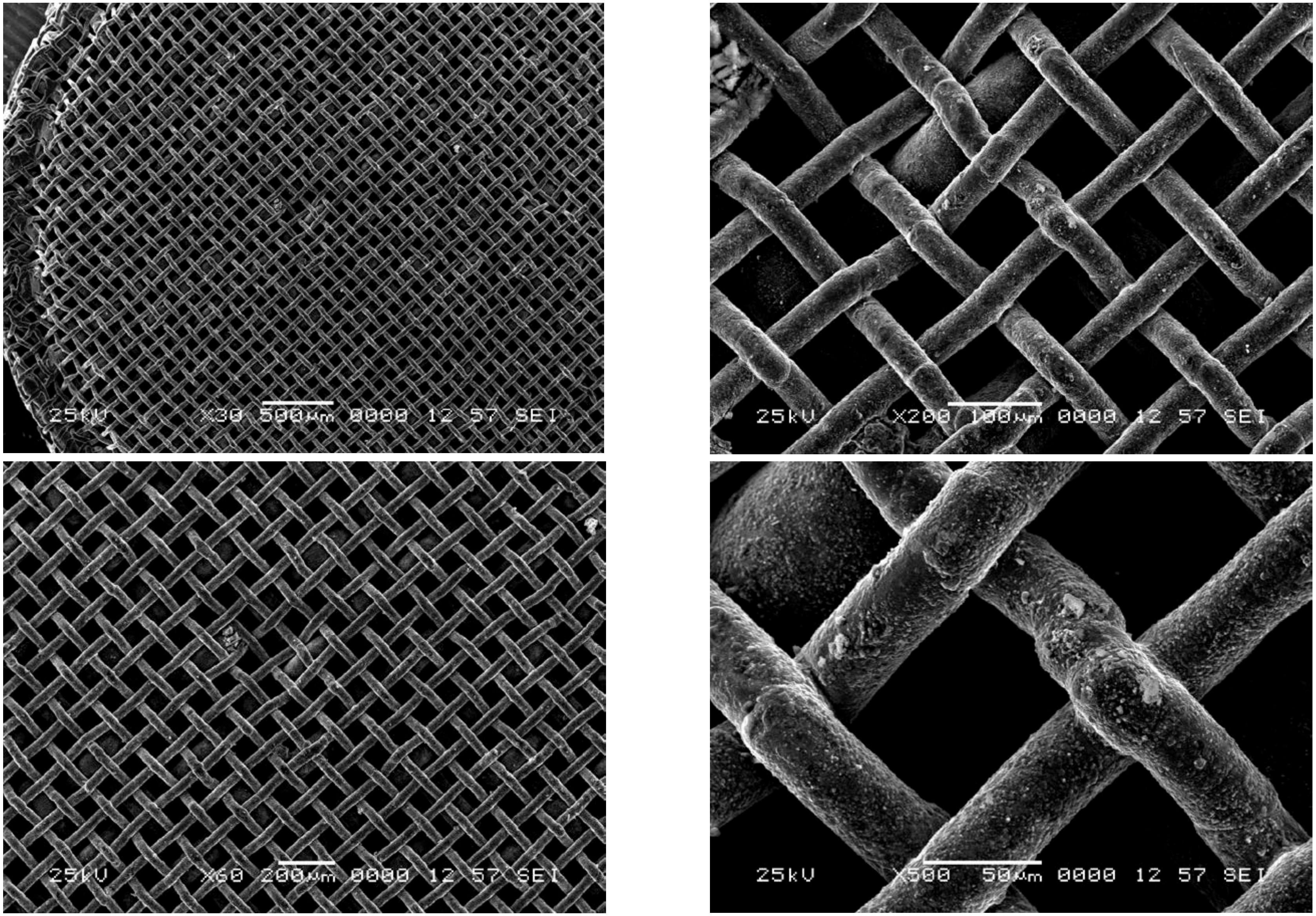


Figure 63 - Typical TMS, Post Carbon Burn-Off, Hexane Rinsed

Elt.	Line	Intensity (c/s)	Conc	Units	Error 2-sig	MDL 3-sig	
Al	Ka	2.07	0.606	wt.%	0.174	0.232	
Si	Ka	1.66	0.369	wt.%	0.138	0.193	
S	Ka	3.10	0.479	wt.%	0.113	0.152	
Cr	Ka	114.12	18.310	wt.%	0.364	0.186	
Mn	Ka	5.79	1.245	wt.%	0.203	0.267	
Fe	Ka	270.46	69.640	wt.%	0.871	0.315	
Ni	Ka	20.34	7.682	wt.%	0.417	0.367	
Cu	Ka	3.76	1.668	wt.%	0.294	0.365	
			100.000	wt.%			Total

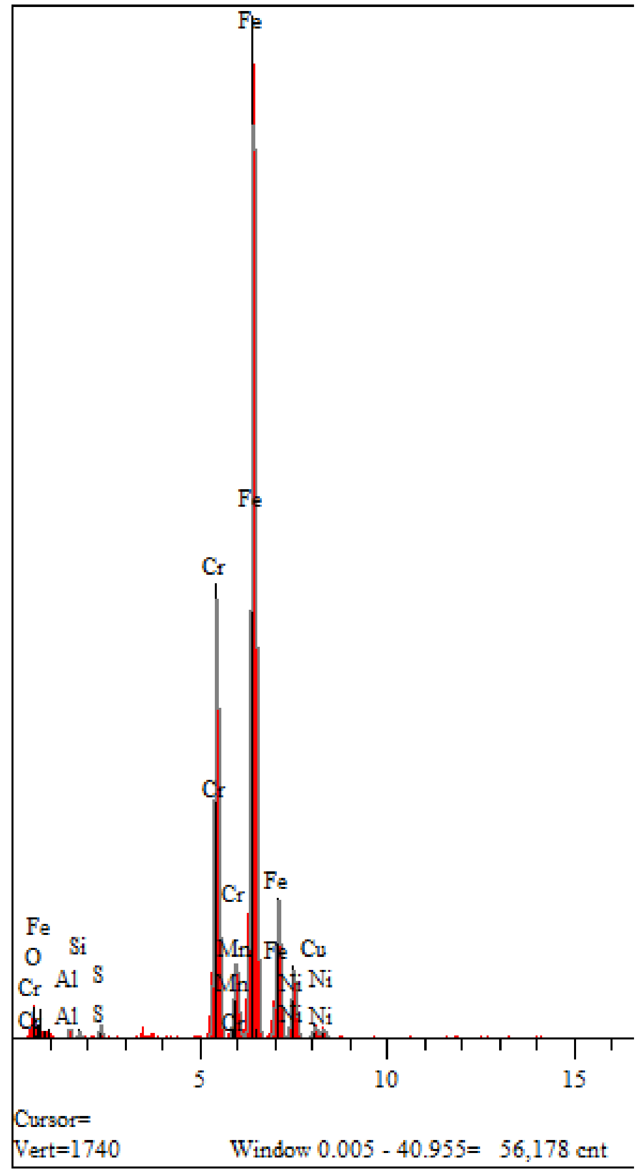


Figure 64 - Elemental Analysis, Typical TMS Post-Test, Post Carbon Burn-off, Hexane rinsed

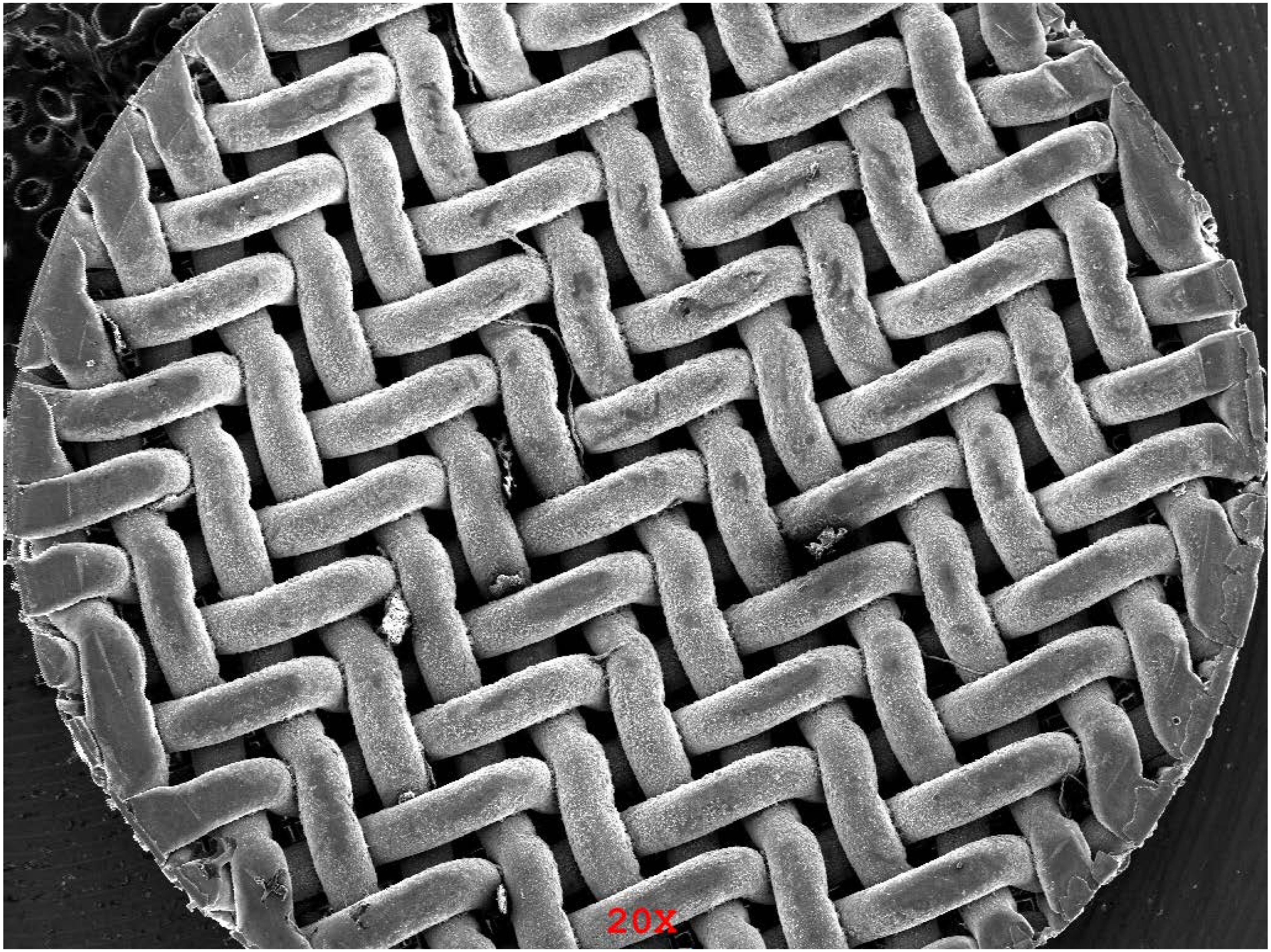
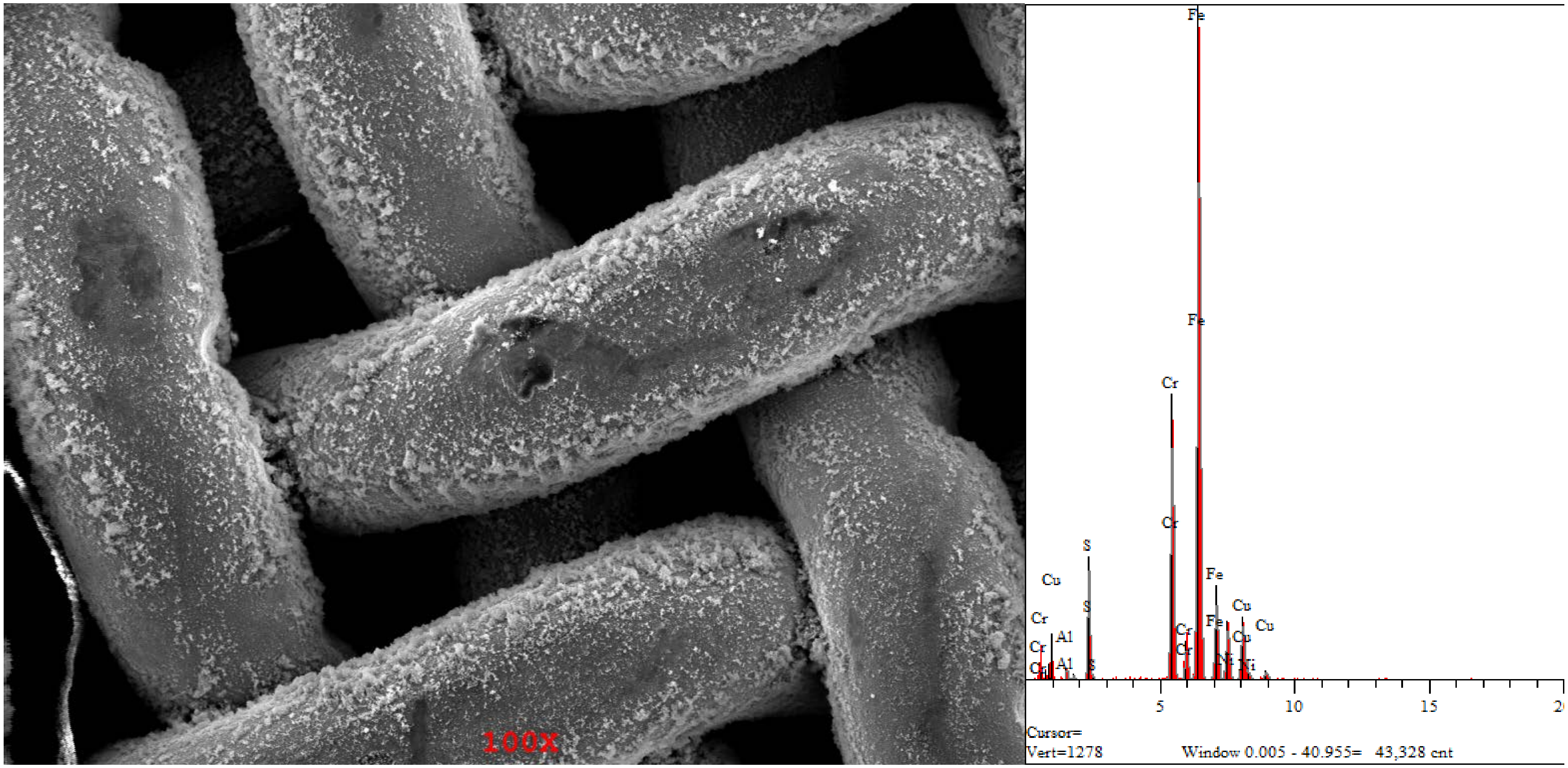
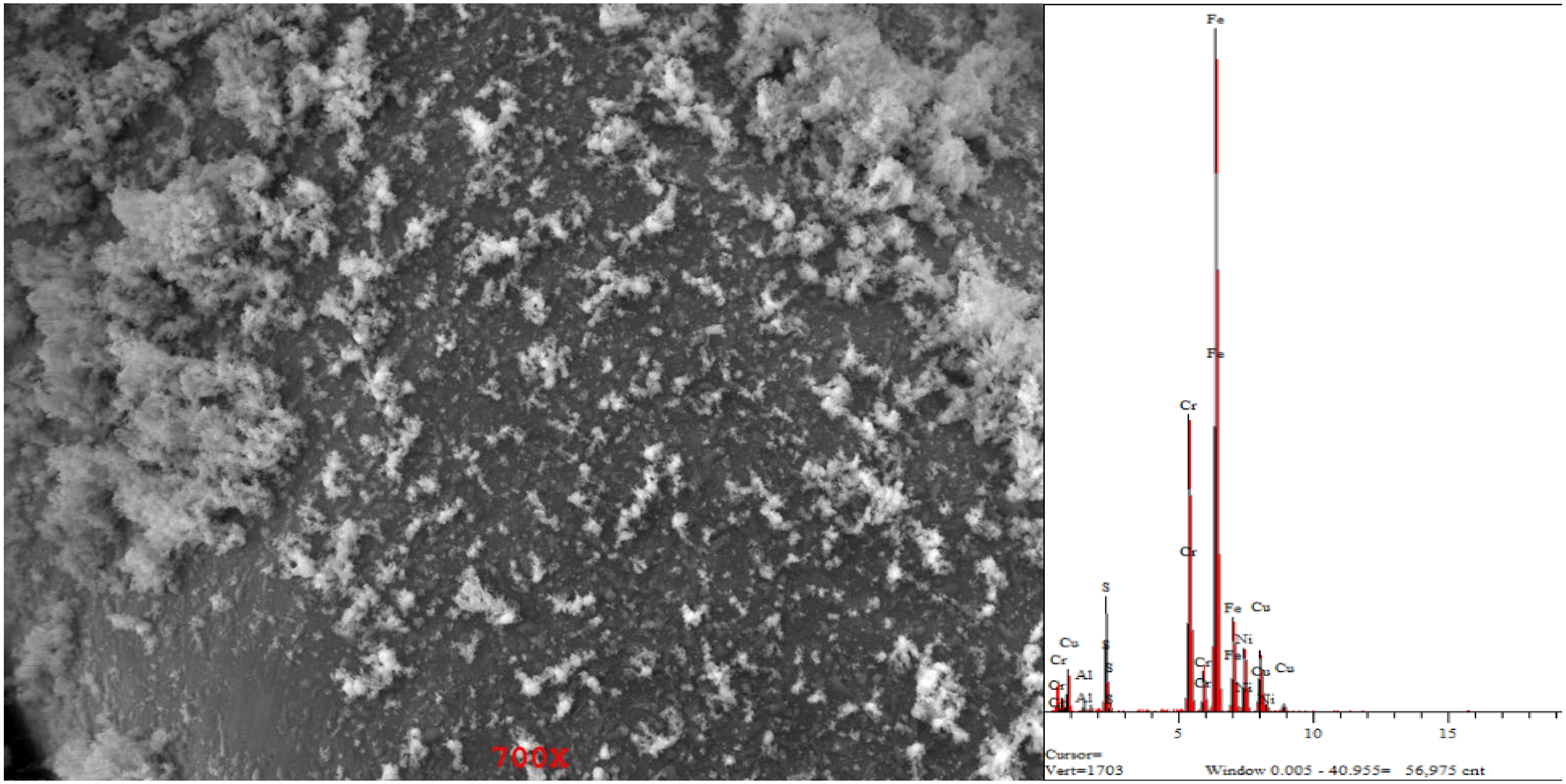


Figure 65 – SEM (20X) of TMS, Baseline Jet A, Run 129 (GDTC), Hexane-Rinsed Prior to Burn-Off



Elt.	Line	Intensity (c/s)	Conc	Units	Error 2-sig	MDL 3-sig	
Al	Ka	3.72	0.910	wt.%	0.240	0.317	
Si	Ka	1.64	0.306	wt.%	0.183	0.264	
S	Ka	41.19	5.357	wt.%	0.256	0.211	
Cr	Ka	112.96	15.603	wt.%	0.407	0.225	
Fe	Ka	275.72	58.355	wt.%	0.932	0.324	
Ni	Ka	27.54	8.451	wt.%	0.501	0.427	
Cu	Ka	30.38	11.019	wt.%	0.601	0.470	
			100.000	wt.%			Total

Figure 66 – SEM (100X) and Elemental Analysis of TMS, Baseline Jet A, Run 129 (GDTC), Hexane-Rinsed Prior to Burn-Off



Elt.	Line	Intensity (c/s)	Conc	Units	Error 2-sig	MDL 3-sig	
Al	Ka	4.32	0.803	wt.%	0.208	0.279	
Si	Ka	3.38	0.477	wt.%	0.165	0.230	
S	Ka	50.03	4.952	wt.%	0.216	0.182	
Cr	Ka	151.18	15.872	wt.%	0.357	0.194	
Fe	Ka	360.39	57.905	wt.%	0.812	0.300	
Ni	Ka	40.08	9.333	wt.%	0.452	0.373	
Cu	Ka	38.71	10.657	wt.%	0.524	0.429	
			100.000	wt.%			Total

Figure 67 – SEM (700X) and Elemental Analysis of TMS, Baseline Jet A, Run 129 (GDTC), Hexane-Rinsed Prior to Burn-Off

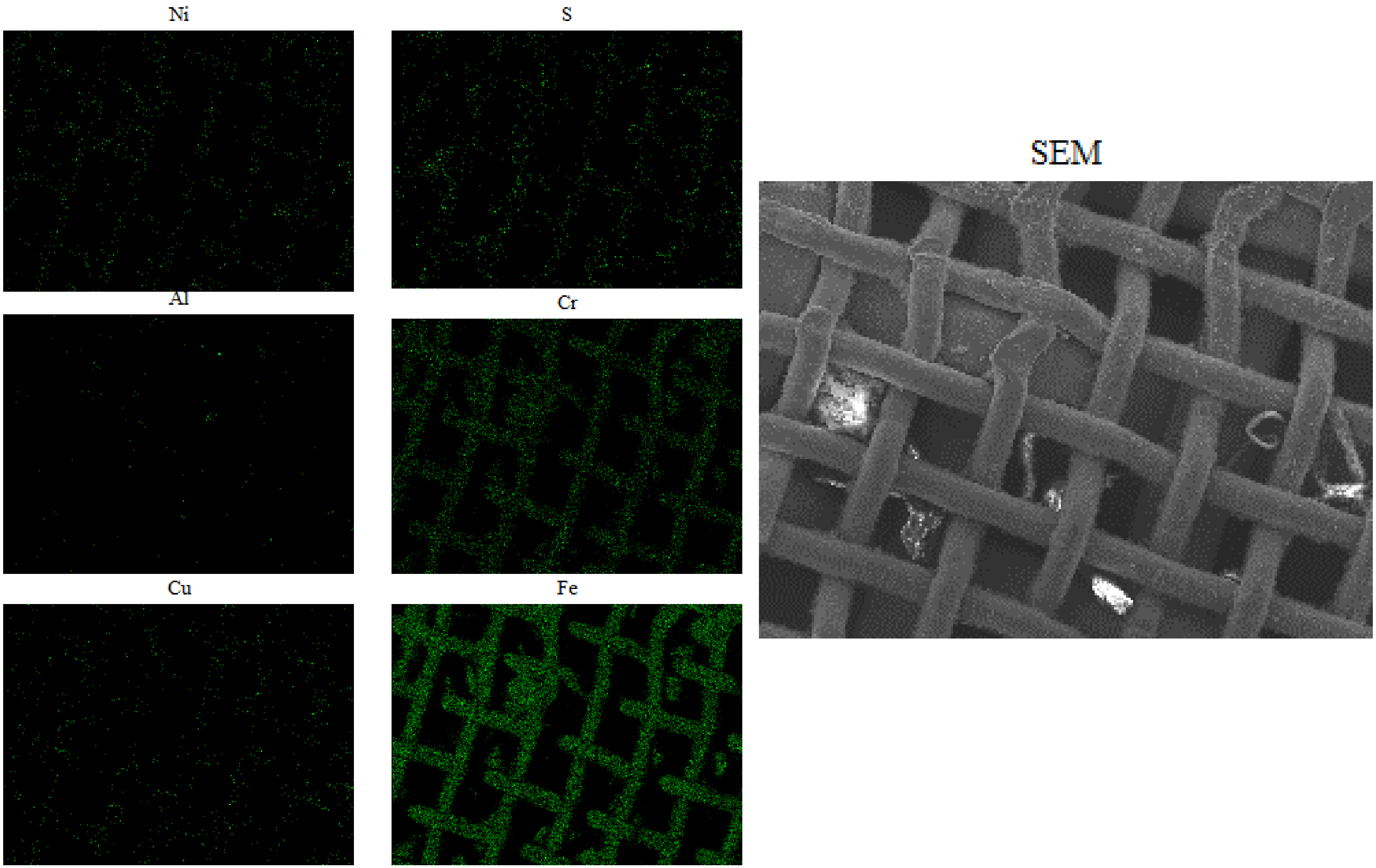
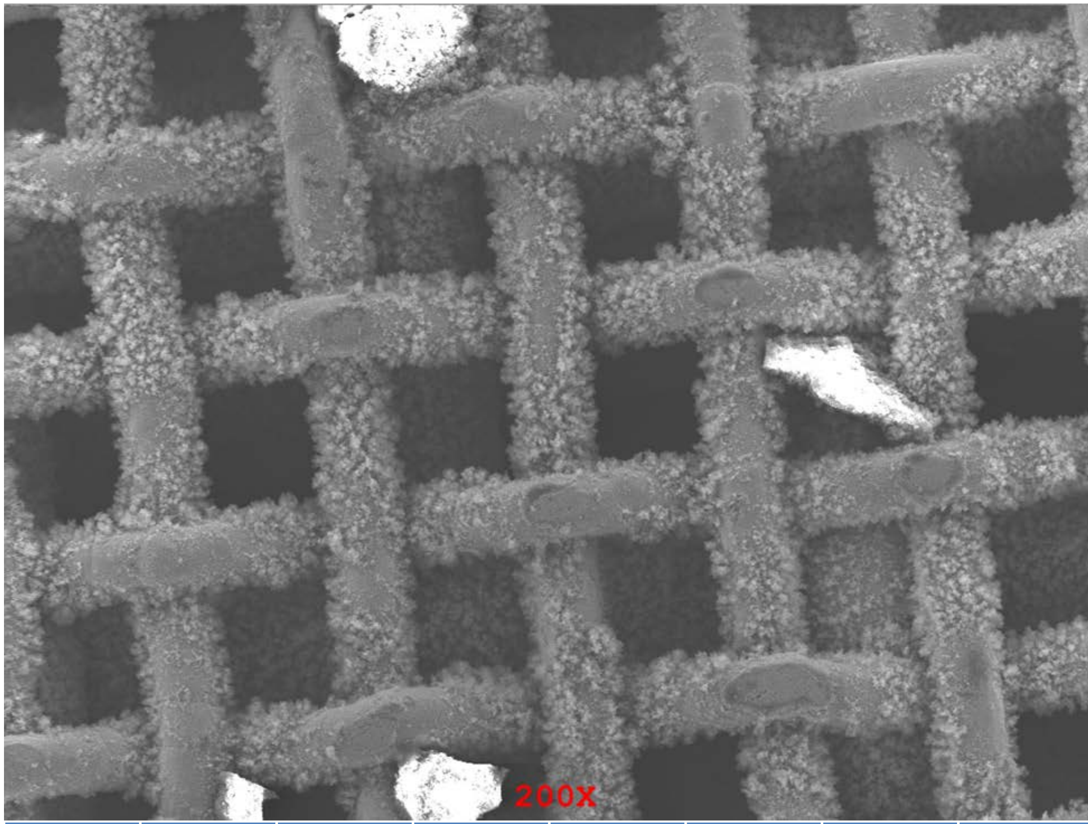


Figure 68 – SEM Backscatter Elemental Analysis of TMS, Baseline Jet A, Run 129 (GDTC), Hexane-Rinsed Prior to Burn-Off



Elt.	Line	Intensity (c/s)	Conc	Units	Error 2-sig	MDL 3-sig	
O	Ka	0.00	0.000	wt.%	0.000	0.000	
Al	Ka	2.08	2.044	wt.%	0.307	0.356	
S	Ka	15.40	9.809	wt.%	0.395	0.271	
K	Ka	7.78	5.205	wt.%	0.323	0.286	
Cr	Ka	13.95	13.602	wt.%	0.565	0.357	
Mn	Ka	0.77	0.951	wt.%	0.334	0.455	
Fe	Ka	32.44	47.240	wt.%	1.224	0.536	
Ni	Ka	2.59	5.549	wt.%	0.650	0.660	
Cu	Ka	5.86	15.600	wt.%	1.040	0.768	
			100.000	wt.%			Total

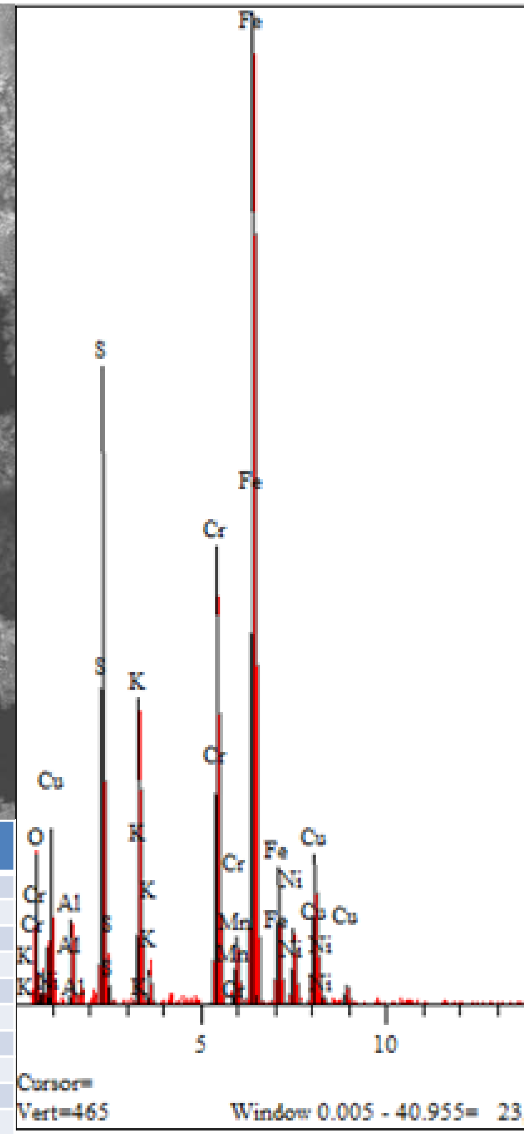
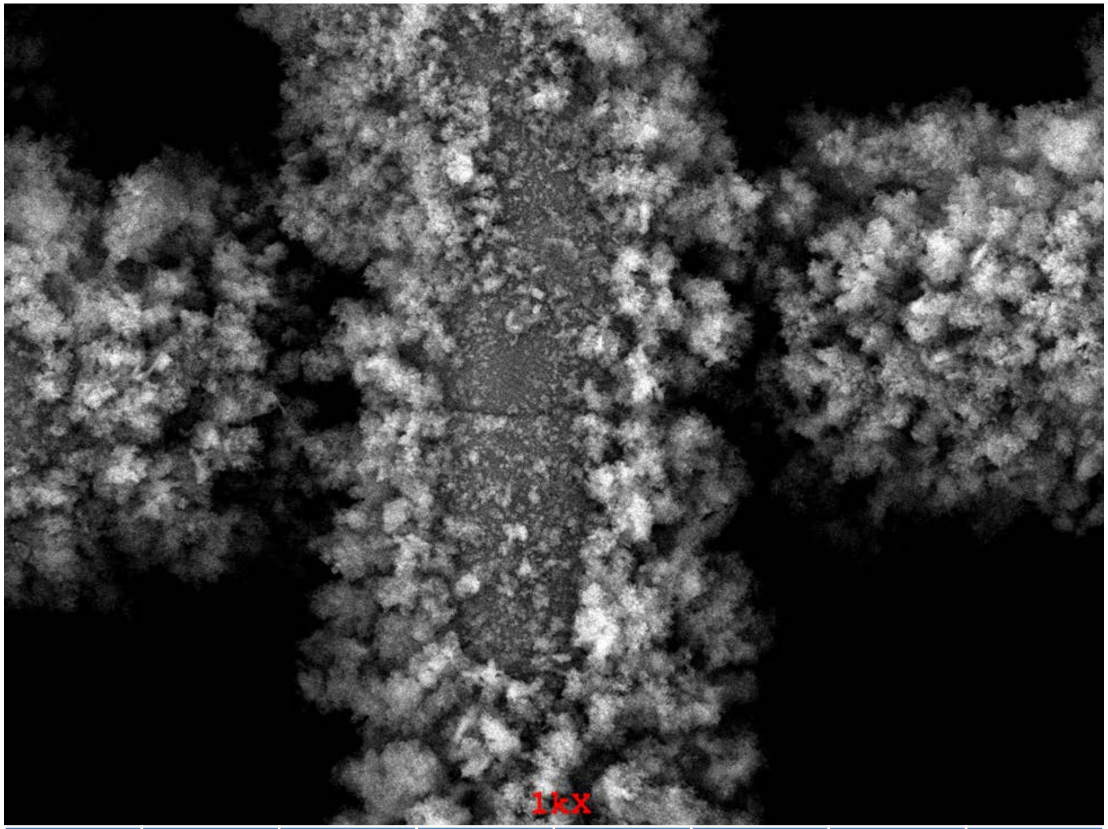


Figure 69 – SEM (200X) and Elemental Analysis of TMS, Baseline Jet A+ 1000 ppm Aquarius, Run 131 (GDTC), Hexane-Rinsed Prior to Burn-Off



Elt.	Line	Intensity (c/s)	Conc	Units	Error 2-sig	MDL 3-sig	
O	Ka	0.00	0.000	wt.%	0.000	0.000	
Al	Ka	2.08	2.044	wt.%	0.307	0.356	
S	Ka	15.40	9.809	wt.%	0.395	0.271	
K	Ka	7.78	5.205	wt.%	0.323	0.286	
Cr	Ka	13.95	13.602	wt.%	0.565	0.357	
Mn	Ka	0.77	0.951	wt.%	0.334	0.455	
Fe	Ka	32.44	47.240	wt.%	1.224	0.536	
Ni	Ka	2.59	5.549	wt.%	0.650	0.660	
Cu	Ka	5.86	15.600	wt.%	1.040	0.768	
			100.000	wt.%			Total

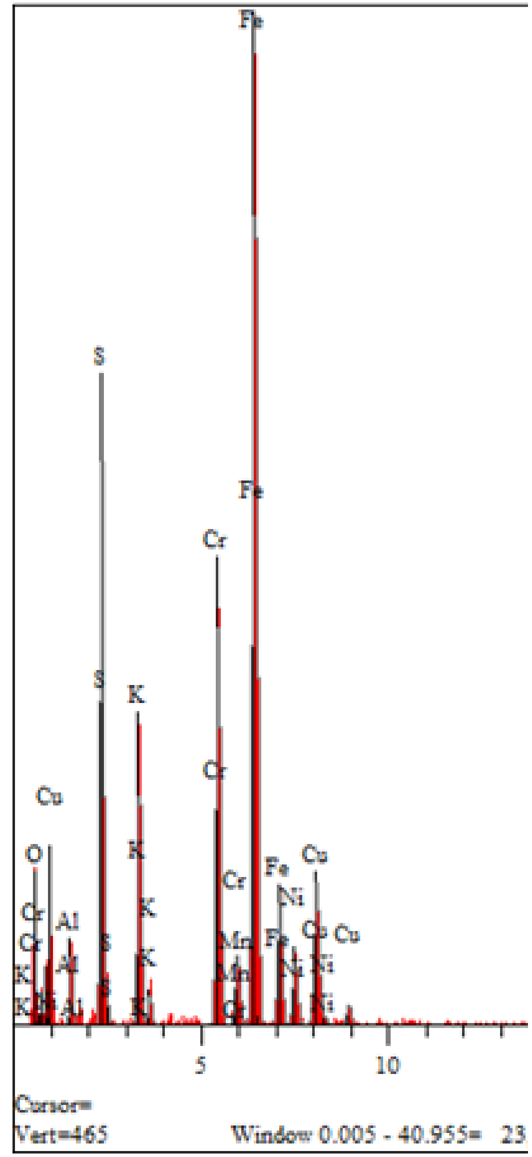


Figure 70 – SEM (1000X) and Elemental Analysis of TMS, Baseline Jet A+ 1000 ppm Aquarius, Run 131 (GDTC), Hexane-Rinsed Prior to Burn-Off

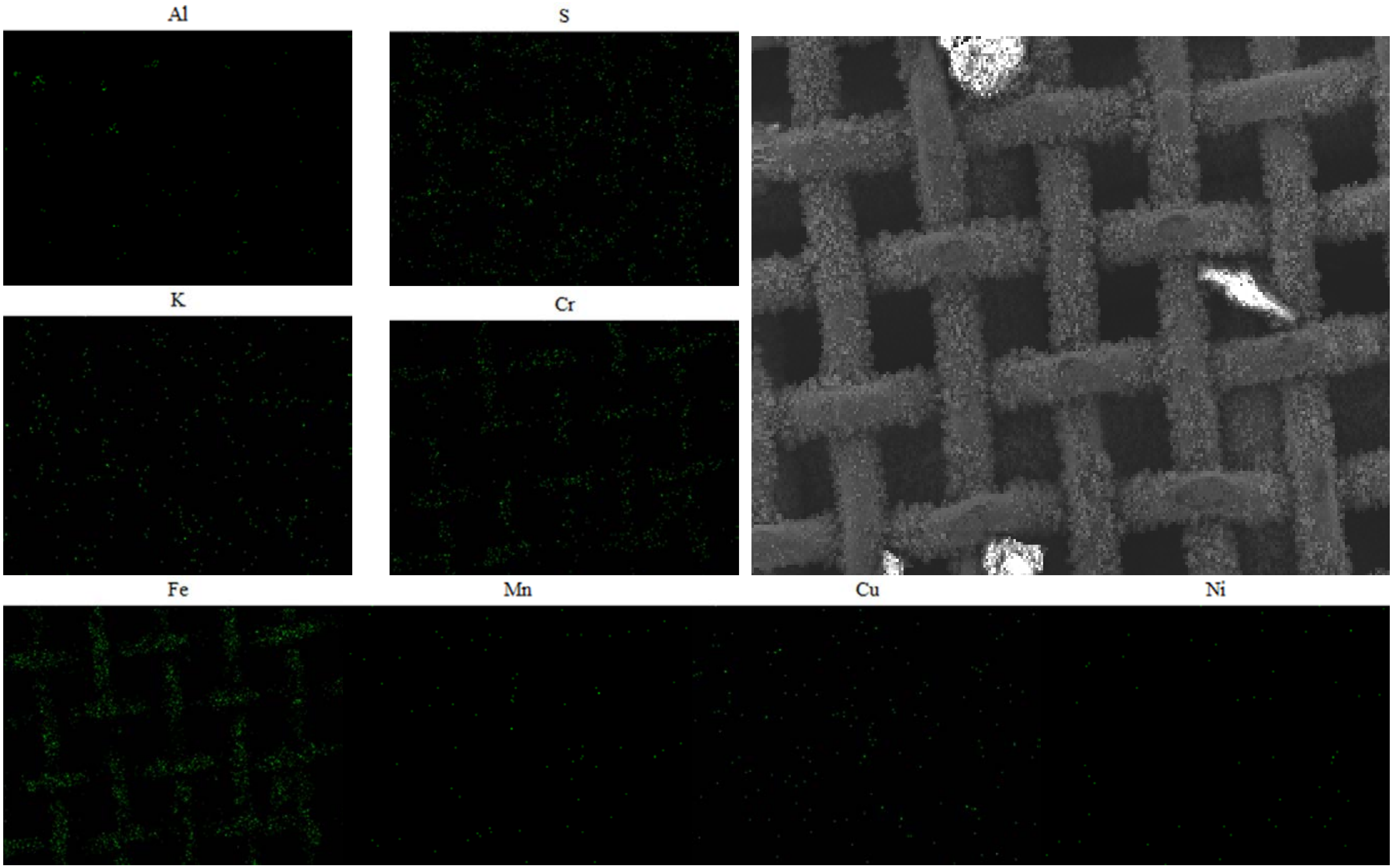
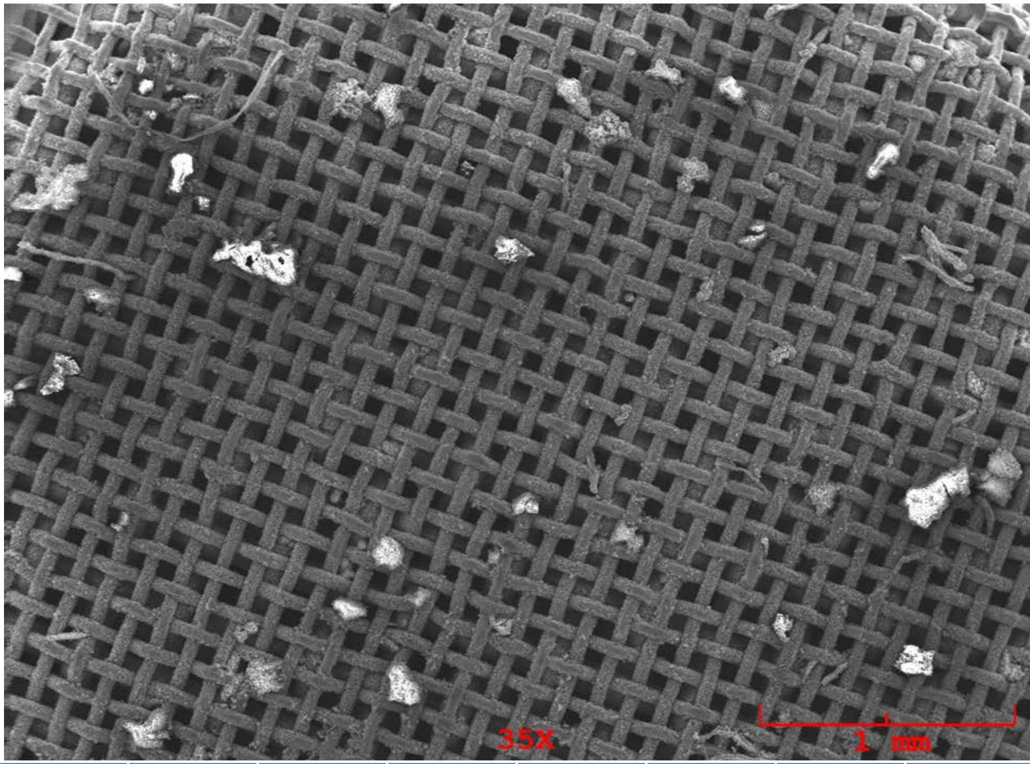


Figure 71 – SEM Backscatter Elemental Analysis of TMS, Baseline Jet A+1000 ppm Aquarius, Run 131 (GDTC), Hexane-Rinsed Prior to Burn-Off



Elt.	Line	Intensity (c/s)	Conc	Units	Error 2-sig	MDL 3-sig	
Al	Ka	3.00	0.588	wt.%	0.132	0.184	
S	Ka	109.64	14.092	wt.%	0.240	0.150	
Cl	Ka	3.90	0.555	wt.%	0.117	0.164	
K	Ka	70.04	9.865	wt.%	0.218	0.157	
Ca	Ka	2.71	0.406	wt.%	0.110	0.156	
Cr	Ka	54.10	11.362	wt.%	0.284	0.200	
Fe	Ka	126.25	37.923	wt.%	0.579	0.276	
Ni	Ka	10.89	4.760	wt.%	0.332	0.358	
Cu	Ka	37.49	20.449	wt.%	0.614	0.433	
			100.000	wt.%			Total

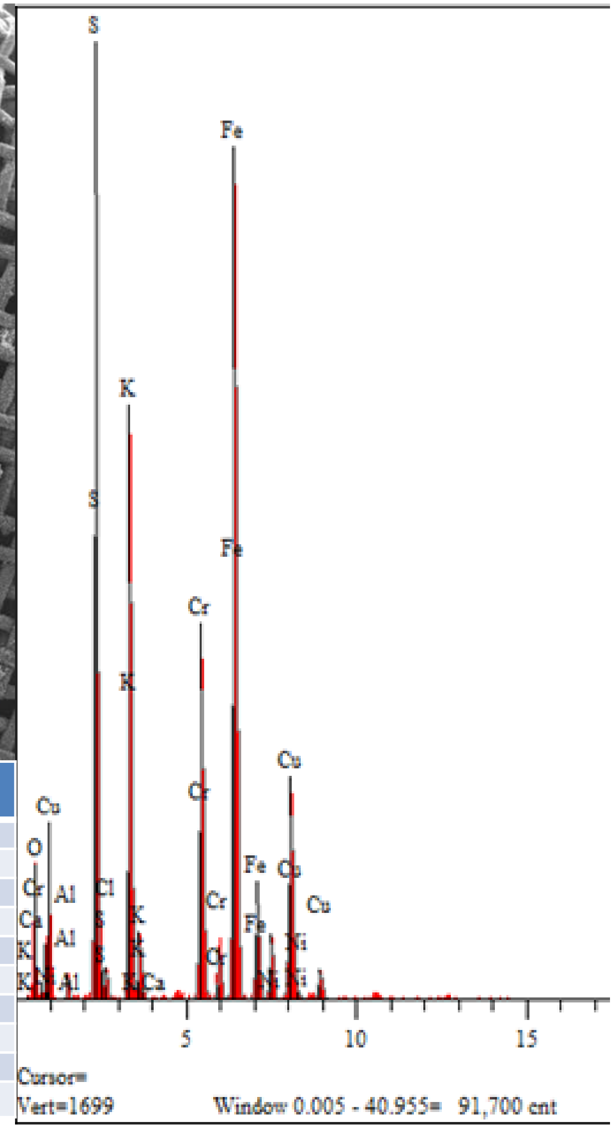
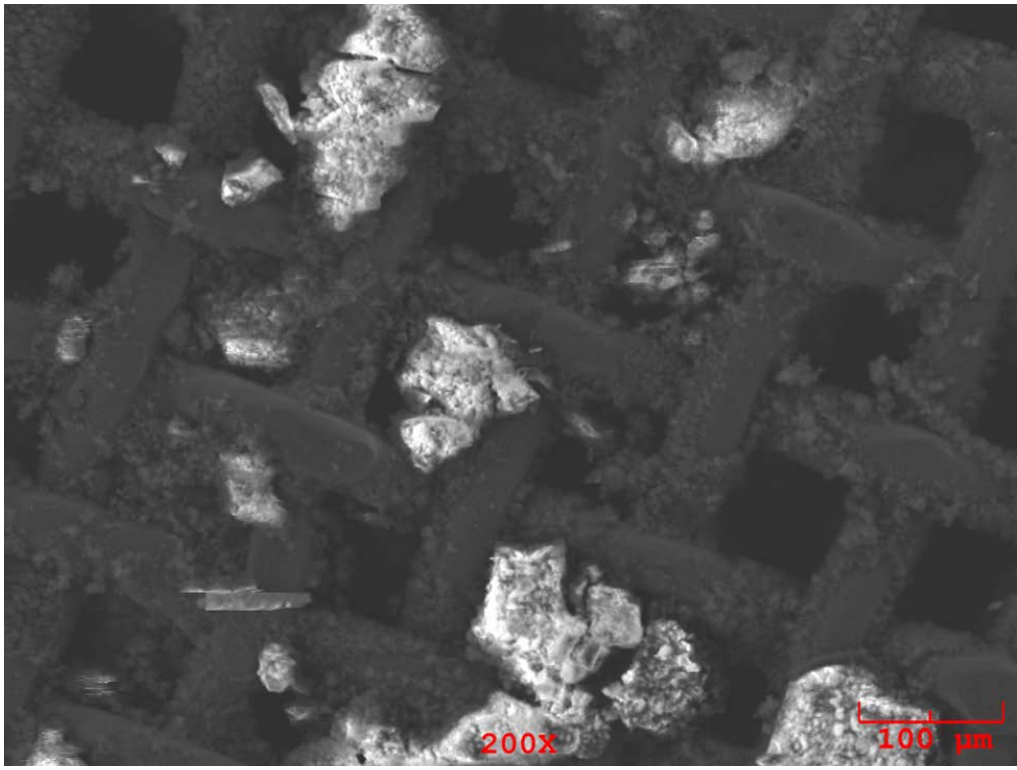
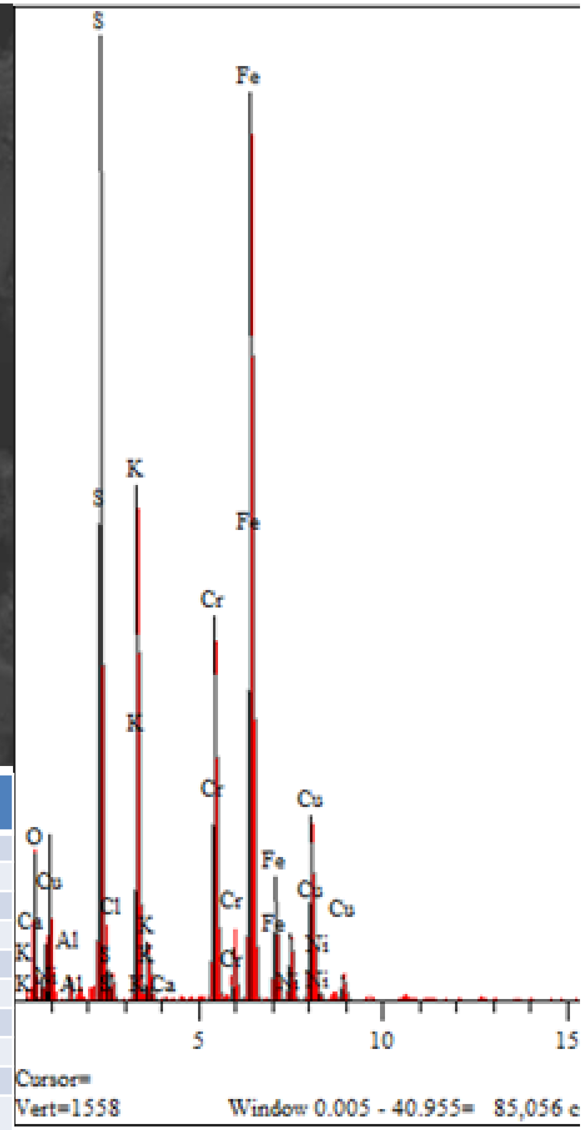


Figure 72 – SEM (35X) and Elemental Analysis of TMS, Baseline Jet A+ 1000 ppm Aquarius, Run 132 (GDTC), Hexane-Rinsed Prior to Burn-Off





Elt.	Line	Intensity (c/s)	Conc	Units	Error 2-sig	MDL 3-sig	
Al	Ka	4.04	0.576	wt.%	0.138	0.192	
S	Ka	155.35	14.524	wt.%	0.255	0.158	
Cl	Ka	4.99	0.519	wt.%	0.122	0.173	
K	Ka	84.78	8.698	wt.%	0.218	0.167	
Ca	Ka	3.91	0.424	wt.%	0.116	0.164	
Cr	Ka	78.79	11.991	wt.%	0.303	0.209	
Fe	Ka	182.88	40.172	wt.%	0.624	0.293	
Ni	Ka	17.01	5.439	wt.%	0.365	0.387	
Cu	Ka	44.29	17.658	wt.%	0.607	0.451	
			100.000	wt.%			Total

Figure 73 – SEM (200X) and Elemental Analysis of TMS, Baseline Jet A+ 1000 ppm Aquarius, Run 132 (GDTC), Hexane-Rinsed Prior to Burn-Off

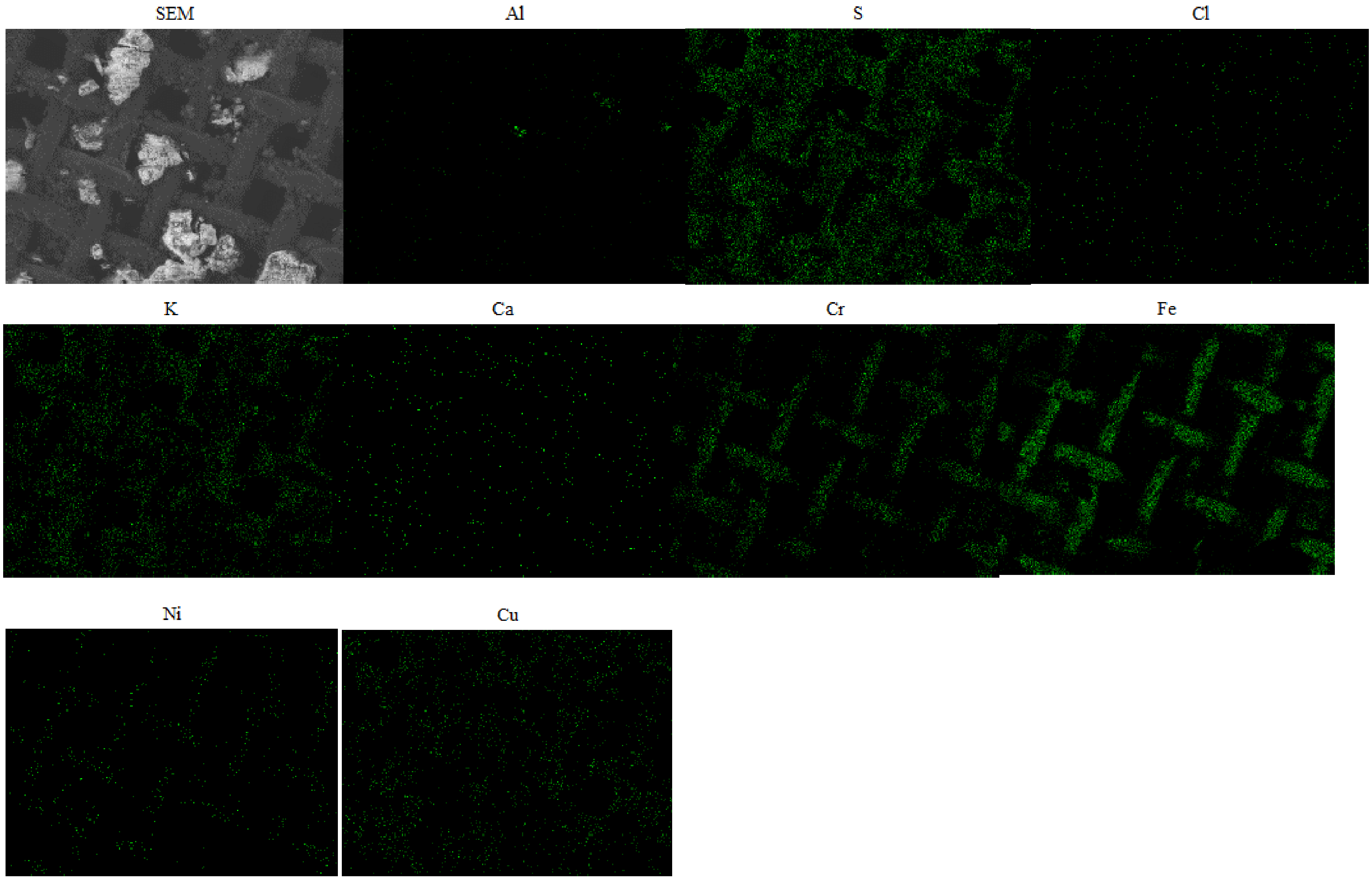
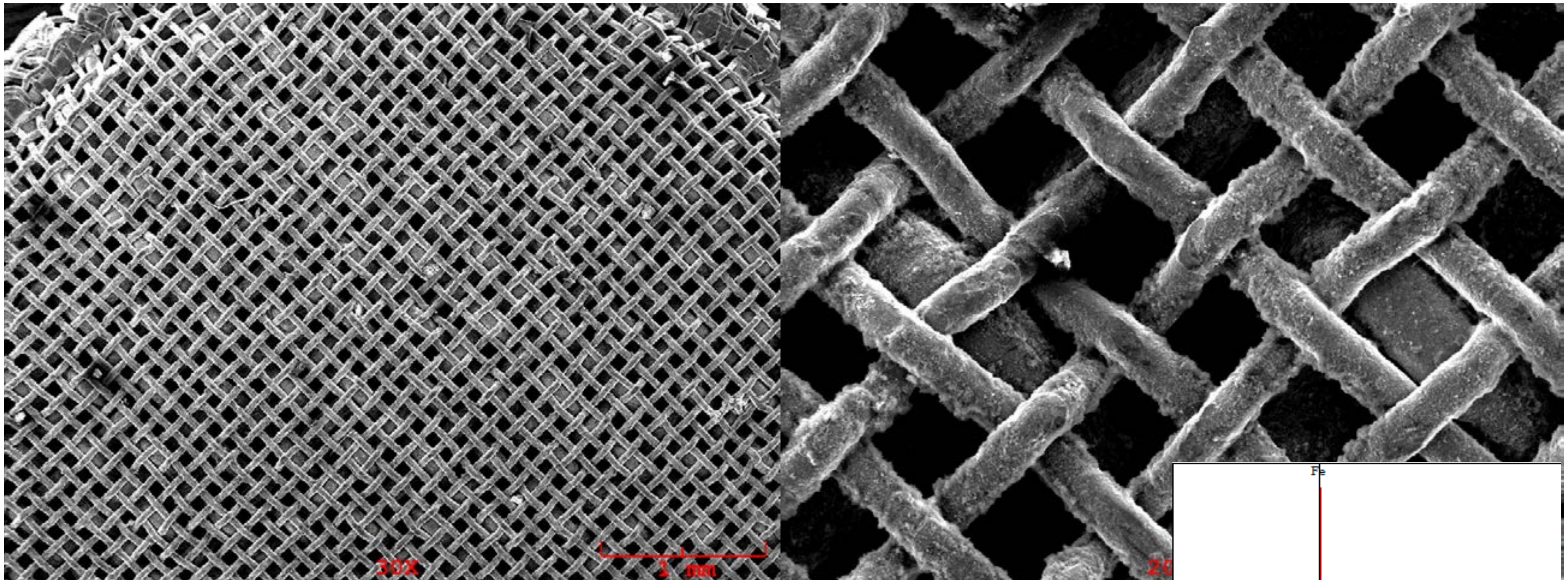
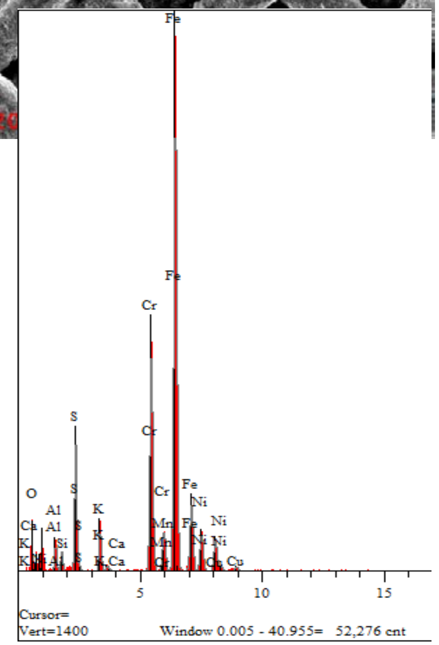


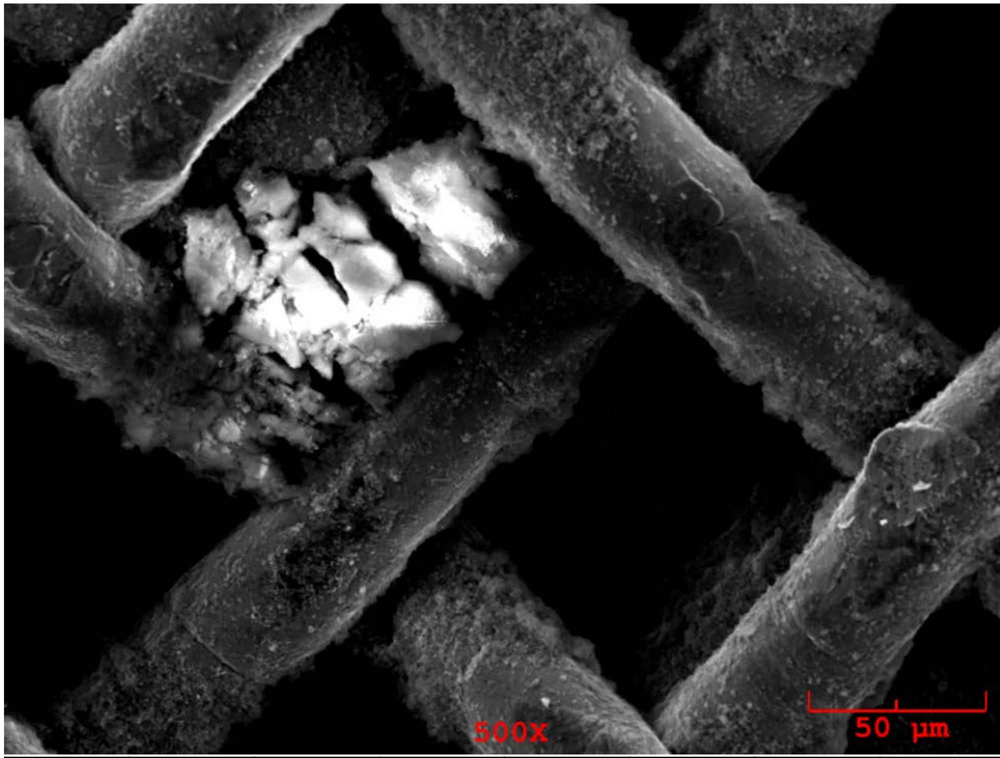
Figure 74 – SEM Backscatter Elemental Analysis of TMS, Baseline Jet A+1000 ppm Aquarius, Run 132 (GDTC), Hexane-Rinsed Prior to Burn-Off





Elt.	Line	Intensity (c/s)	Conc	Units	Error 2-sig	MDL 3-sig	
Al	Ka	7.80	1.633	wt.%	0.181	0.210	
Si	Ka	5.60	0.956	wt.%	0.145	0.183	
S	Ka	37.82	5.106	wt.%	0.197	0.162	
K	Ka	13.73	1.876	wt.%	0.150	0.169	
Ca	Ka	0.93	0.129	wt.%	0.112	0.167	
Cr	Ka	81.73	15.999	wt.%	0.383	0.222	
Mn	Ka	5.02	1.292	wt.%	0.221	0.289	
Fe	Ka	189.92	58.244	wt.%	0.874	0.339	
Ni	Ka	15.91	7.228	wt.%	0.462	0.438	
Cu	Ka	13.40	7.537	wt.%	0.528	0.507	
			100.000	wt.%			Total

Figure 75 – SEM (10X) and Elemental Analysis of TMS, Baseline Jet A+ 1000 ppm Aquarius, Run 133 (EDTST), Hexane-Rinsed Prior to Burn-Off



Elt.	Line	Intensity (c/s)	Conc	Units	Error 2-sig	MDL 3-sig	
Al	Ka	6.92	1.446	wt.%	0.178	0.213	
Si	Ka	28.62	4.914	wt.%	0.222	0.189	
S	Ka	26.64	3.734	wt.%	0.182	0.170	
K	Ka	8.34	1.166	wt.%	0.137	0.169	
Ca	Ka	1.41	0.199	wt.%	0.114	0.167	
Cr	Ka	80.56	16.086	wt.%	0.387	0.225	
Mn	Ka	3.99	1.051	wt.%	0.220	0.296	
Fe	Ka	186.94	58.692	wt.%	0.887	0.342	
Ni	Ka	15.94	7.406	wt.%	0.466	0.432	
Cu	Ka	9.22	5.305	wt.%	0.470	0.481	
			100.000	wt.%			Total

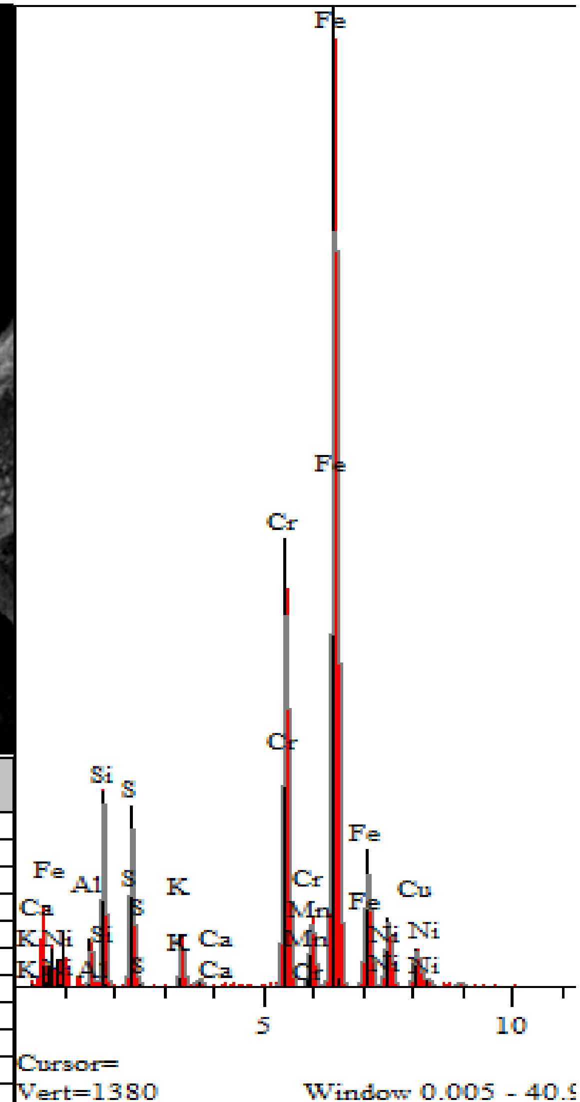
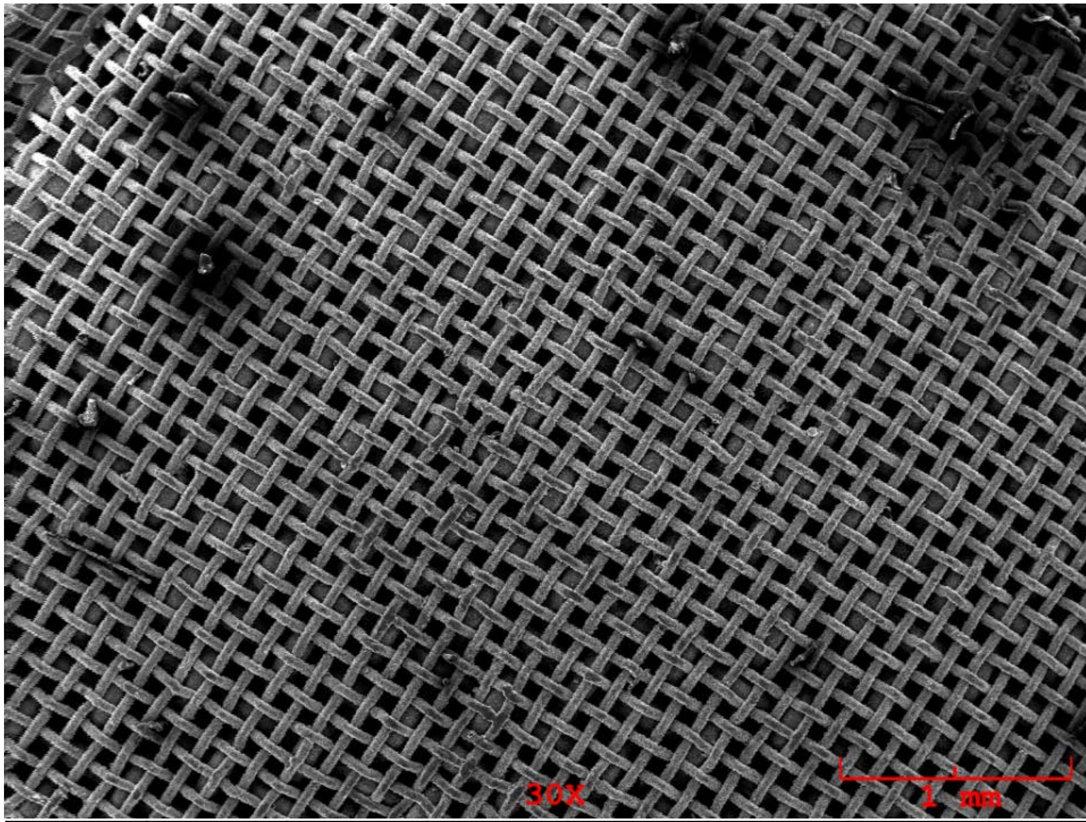


Figure 76 – SEM (500X) and Elemental Analysis of TMS, Baseline Jet A+ 1000 ppm Aquarius, Run 133 (EDTST), Hexane-Rinsed Prior to Burn-Off



Elt.	Line	Intensity (c/s)	Conc	Units	Error 2-sig	MDL 3-sig	
Al	Ka	7.16	1.181	wt.%	0.155	0.195	
Si	Ka	2.53	0.338	wt.%	0.119	0.170	
S	Ka	44.90	4.693	wt.%	0.170	0.147	
Cr	Ka	107.79	15.936	wt.%	0.334	0.201	
Mn	Ka	7.04	1.382	wt.%	0.202	0.264	
Fe	Ka	253.56	59.416	wt.%	0.773	0.307	
Ni	Ka	23.09	8.058	wt.%	0.411	0.363	
Cu	Ka	20.82	8.996	wt.%	0.493	0.454	
			100.000	wt.%			Total

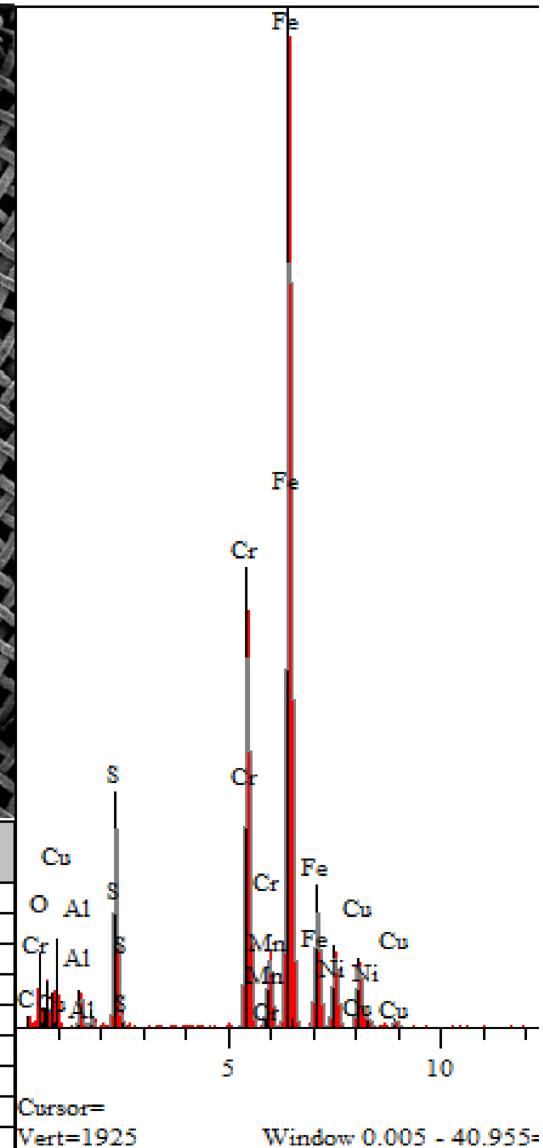
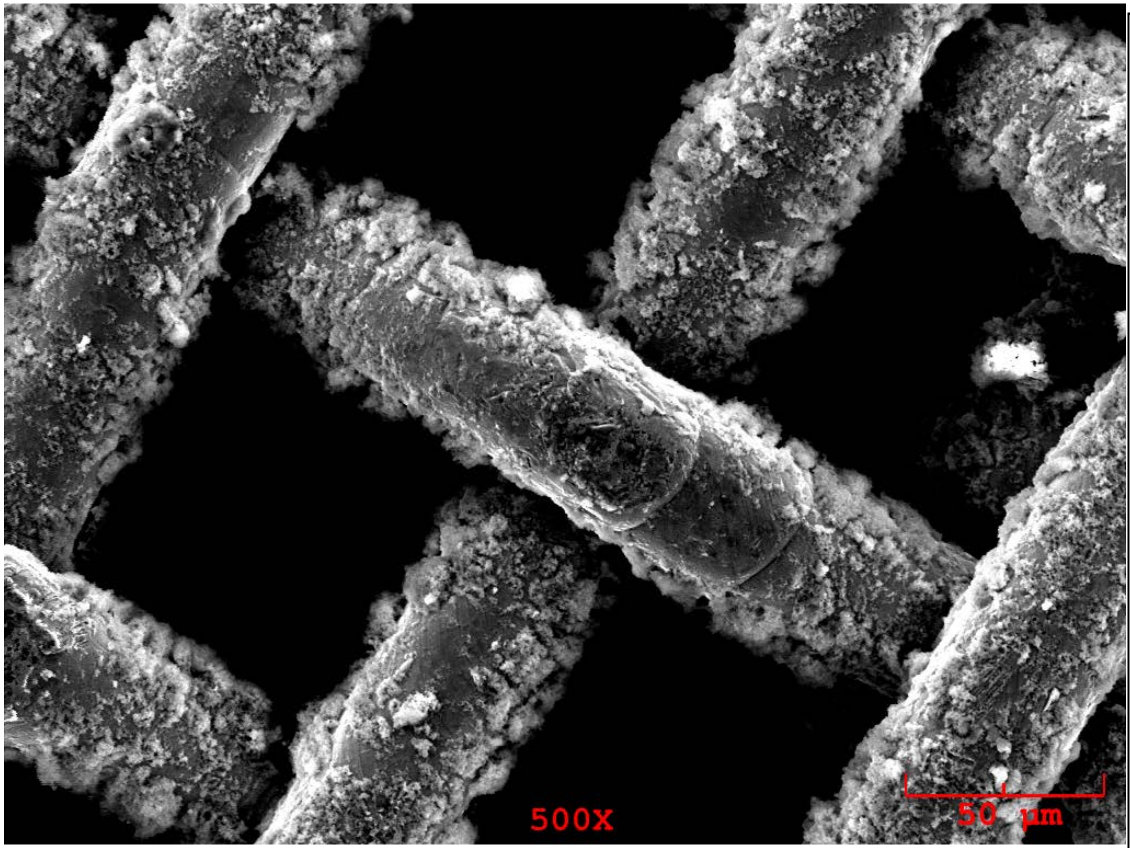


Figure 77 – SEM (30X) and Elemental Analysis of TMS, Baseline Jet A, Run 134 (EDTST), Hexane-Rinsed Prior to Burn-Off



Elt.	Line	Intensity (c/s)	Conc	Units	Error 2-sig	MDL 3-sig	
Al	Ka	8.35	1.323	wt.%	0.154	0.190	
Si	Ka	4.76	0.612	wt.%	0.121	0.164	
S	Ka	43.40	4.377	wt.%	0.164	0.146	
Cr	Ka	116.17	16.589	wt.%	0.332	0.190	
Mn	Ka	6.45	1.223	wt.%	0.188	0.248	
Fe	Ka	264.28	59.909	wt.%	0.761	0.290	
Ni	Ka	22.76	7.671	wt.%	0.404	0.374	
Cu	Ka	19.88	8.295	wt.%	0.472	0.445	
			100.000	wt.%			Total

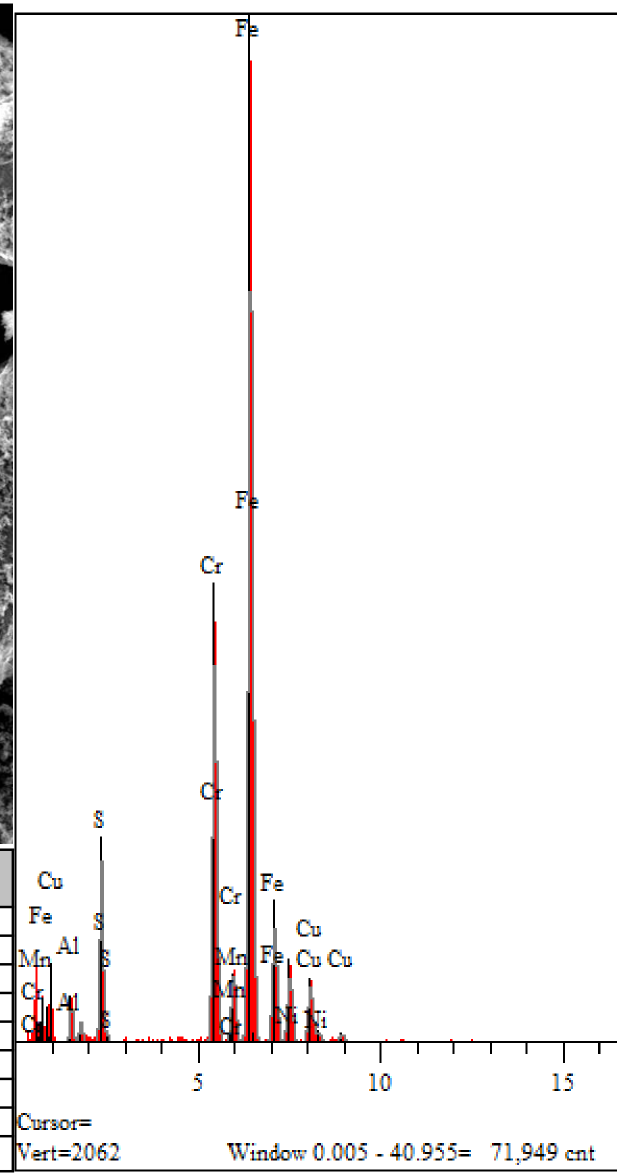
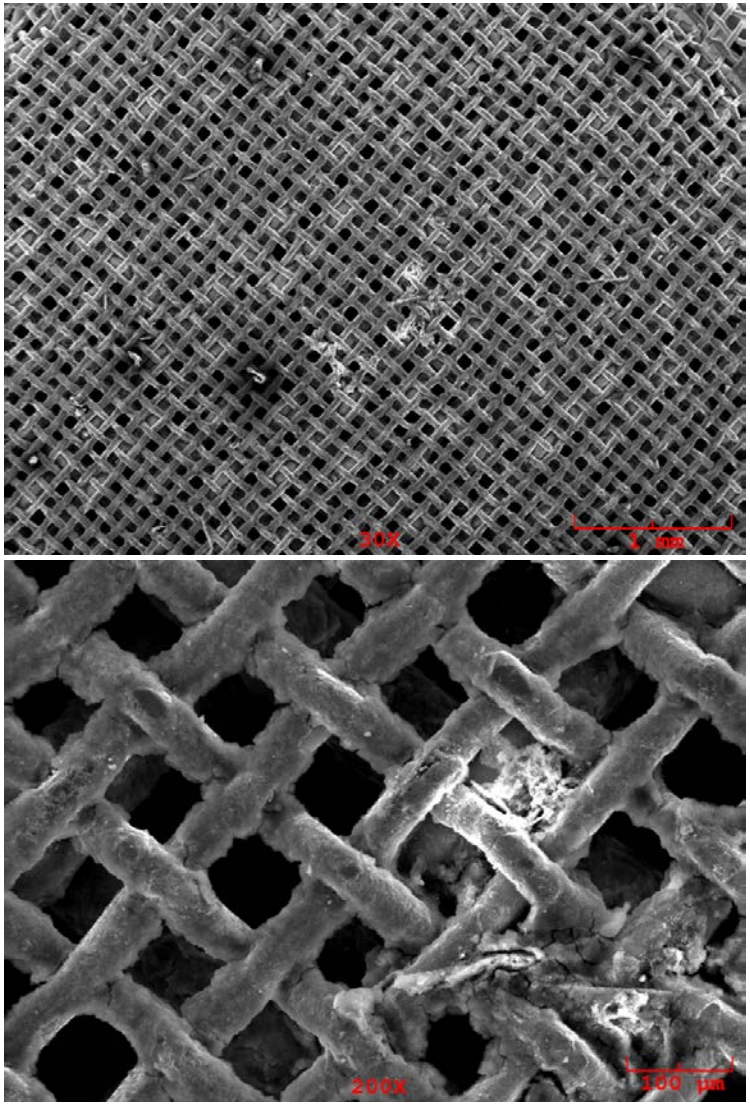
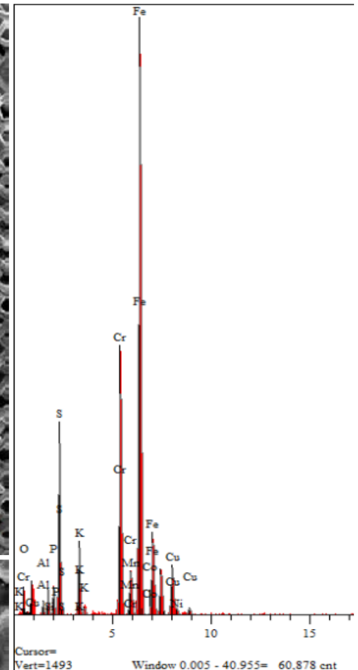


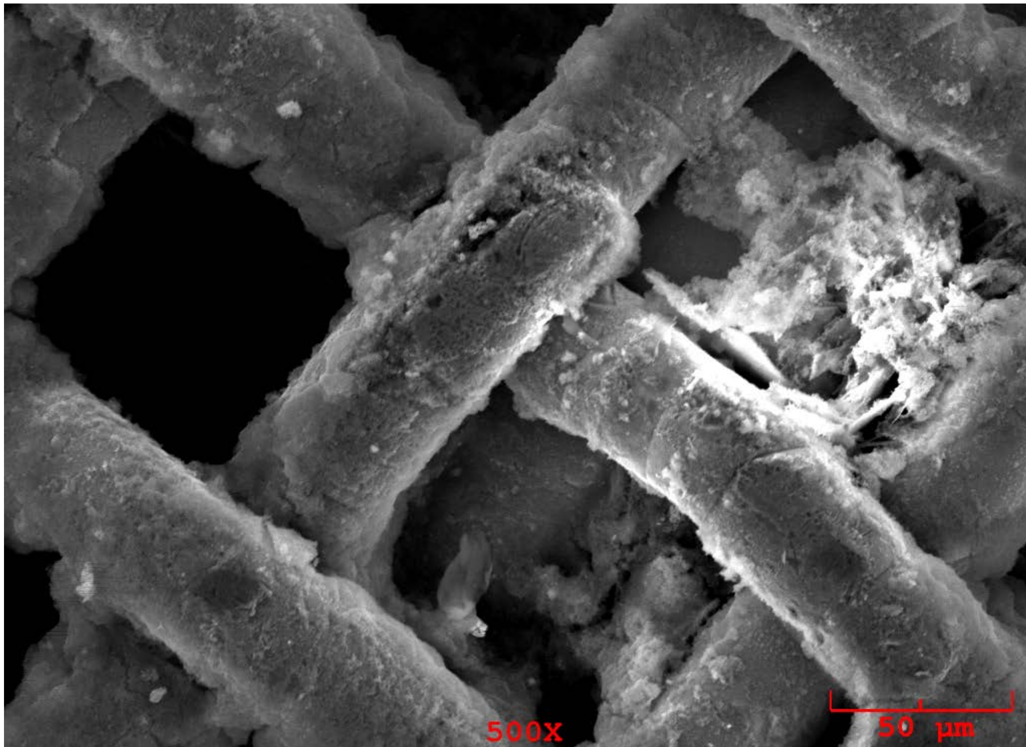
Figure 78 – SEM (500X) and Elemental Analysis of TMS, Baseline Jet A, Run 134 (EDTST), Hexane-Rinsed Prior to Burn-Off





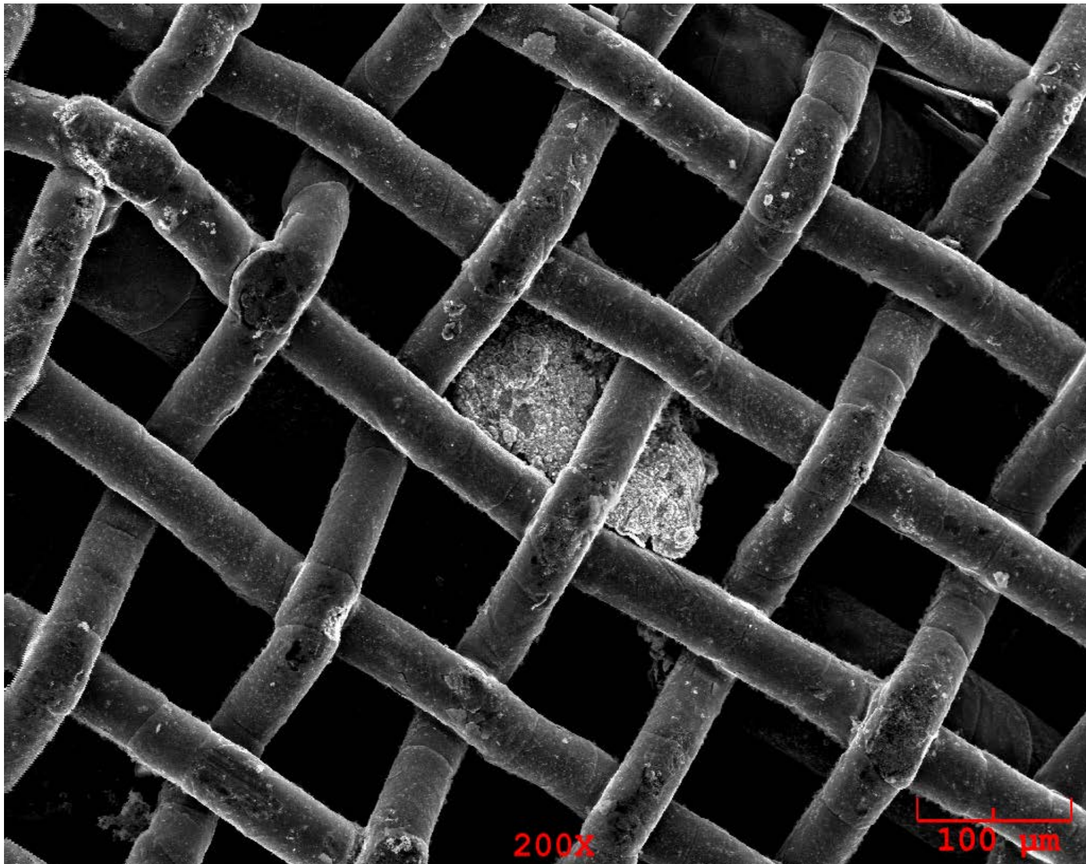
Elt.	Line	Intensity (c/s)	Conc	Units	Error 2-sig	MDL 3-sig	
Al	Ka	3.66	0.954	wt.%	0.186	0.241	
Si	Ka	6.89	1.378	wt.%	0.172	0.208	
P	Ka	7.69	1.324	wt.%	0.163	0.201	
S	Ka	52.68	7.695	wt.%	0.241	0.174	
K	Ka	21.98	2.940	wt.%	0.163	0.160	
Cr	Ka	91.72	15.078	wt.%	0.339	0.191	
Mn	Ka	5.43	1.135	wt.%	0.190	0.249	
Fe	Ka	223.08	54.412	wt.%	0.754	0.294	
Co	Ka	0.60	0.168	wt.%	0.201	0.300	
Ni	Ka	18.40	6.421	wt.%	0.371	0.334	
Cu	Ka	20.58	8.495	wt.%	0.458	0.402	
			100.000	wt.%			Total

Figure 79 – SEM (30X/200X) and Elemental Analysis of TMS, Baseline Jet A + 250 ppm Aquarius + 200 ppm Total Dissolved Water, Run 135 (EDTST), Hexane-Rinsed Prior to Burn-Off



Elt.	Line	Intensity (c/s)	Conc	Units	Error 2-sig	MDL 3-sig	
Al	Ka	4.57	1.219	wt.%	0.193	0.238	
Si	Ka	5.71	1.172	wt.%	0.165	0.204	
P	Ka	14.35	2.537	wt.%	0.184	0.192	
S	Ka	45.79	6.949	wt.%	0.232	0.165	
K	Ka	16.87	2.322	wt.%	0.154	0.159	
Cr	Ka	91.69	15.443	wt.%	0.346	0.191	
Mn	Ka	4.84	1.042	wt.%	0.188	0.249	
Fe	Ka	222.99	56.191	wt.%	0.776	0.287	
Co	Ka	0.60	0.173	wt.%	0.196	0.292	
Ni	Ka	17.71	6.393	wt.%	0.380	0.349	
Cu	Ka	15.38	6.561	wt.%	0.422	0.393	
			100.000	wt.%			Total

Figure 80 – SEM (500X) and Elemental Analysis of TMS, Baseline Jet A + 250 ppm Aquarius + 200 ppm Total Dissolved Water, Run 135 (EDTST), Hexane-Rinsed Prior to Burn-Off



Elt.	Line	Intensity (c/s)	Conc	Units	Error 2-sig	MDL 3-sig	
Al	Ka	2.07	0.606	wt.%	0.174	0.232	
Si	Ka	1.66	0.369	wt.%	0.138	0.193	
S	Ka	3.10	0.479	wt.%	0.113	0.152	
Cr	Ka	114.12	18.310	wt.%	0.364	0.186	
Mn	Ka	5.79	1.245	wt.%	0.203	0.267	
Fe	Ka	270.46	69.640	wt.%	0.871	0.315	
Ni	Ka	20.34	7.682	wt.%	0.417	0.367	
Cu	Ka	3.76	1.668	wt.%	0.294	0.365	
			100.000	wt.%			Total

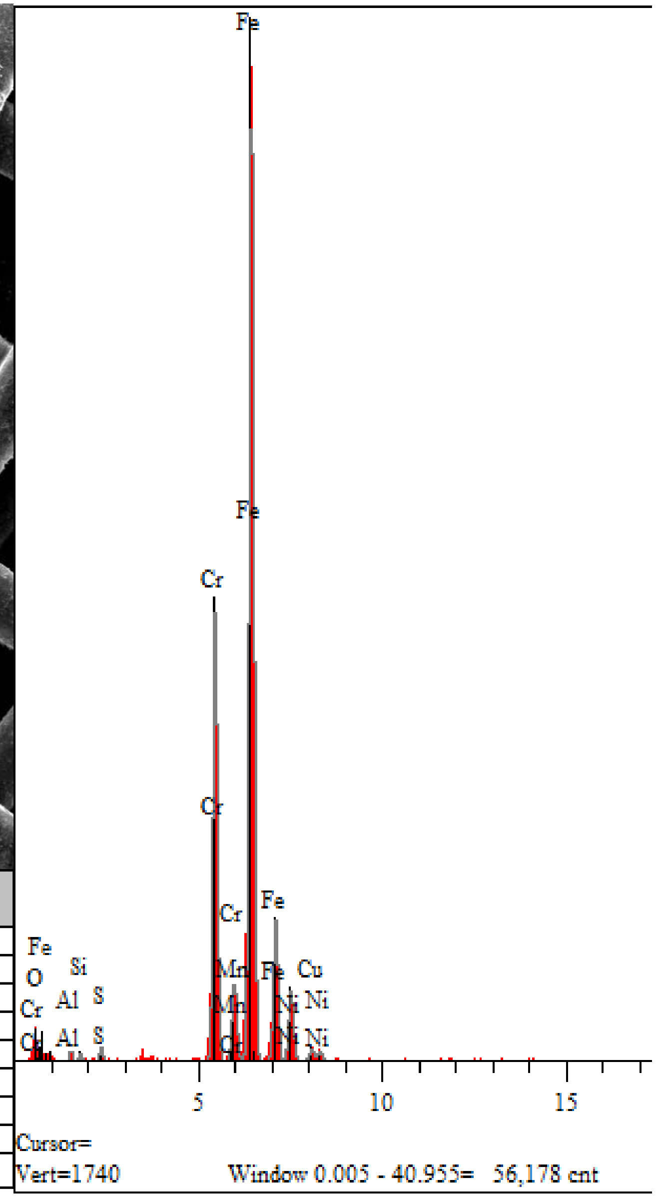


Figure 81 – SEM (200X) and Elemental Analysis of TMS, Post-Burn-off (Typical)

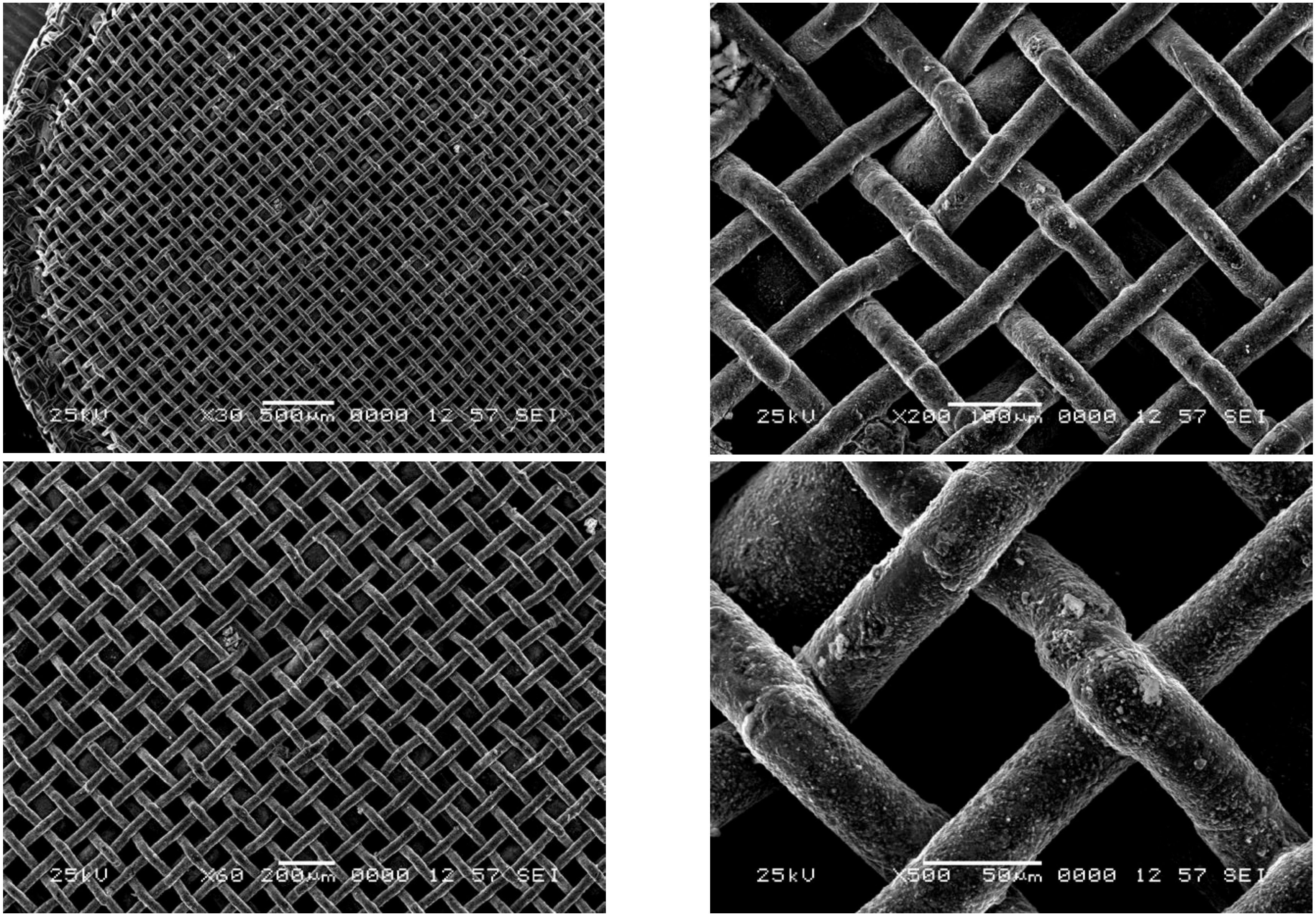
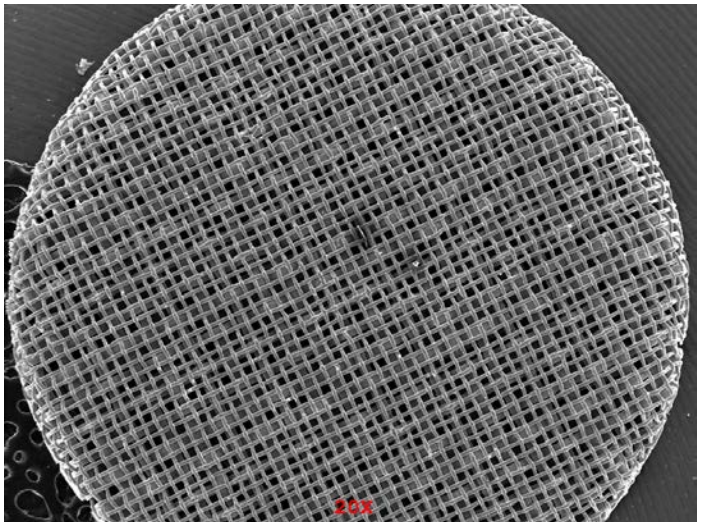
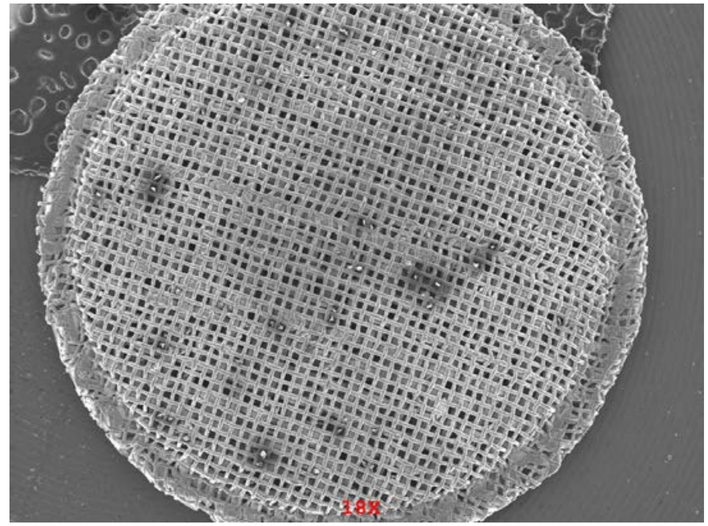


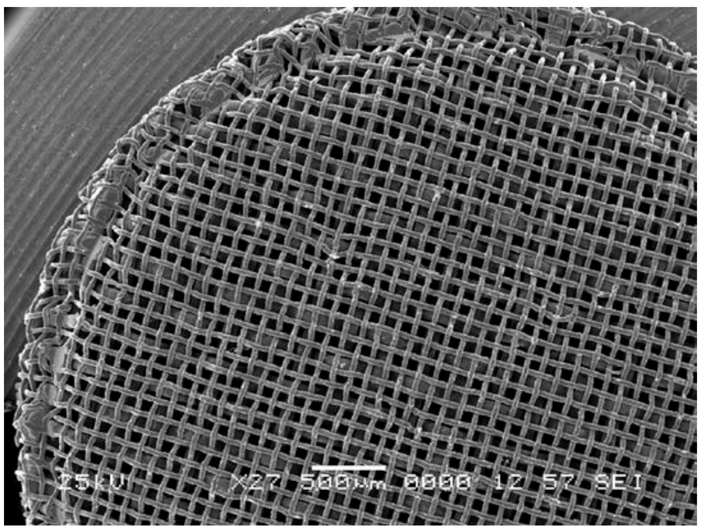
Figure 82 – SEM (30X/60X/200X/500X) and Elemental Analysis of TMS, Post-Burn-off (Typical)



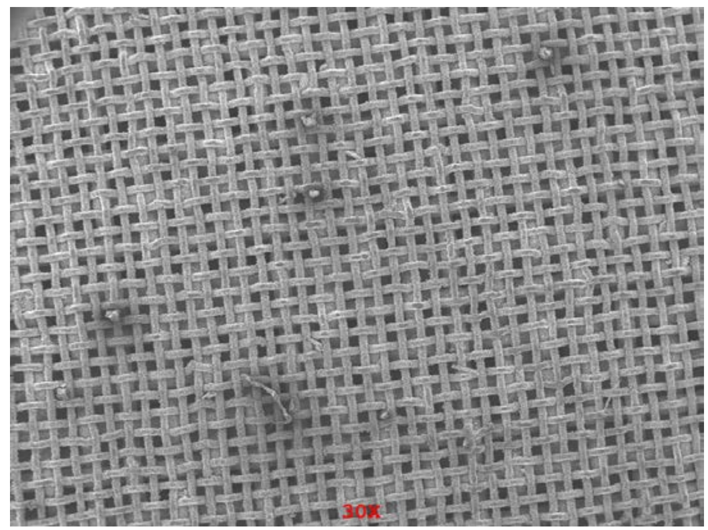
Clean New TMS



Run 129 Baseline Jet A, GDTC

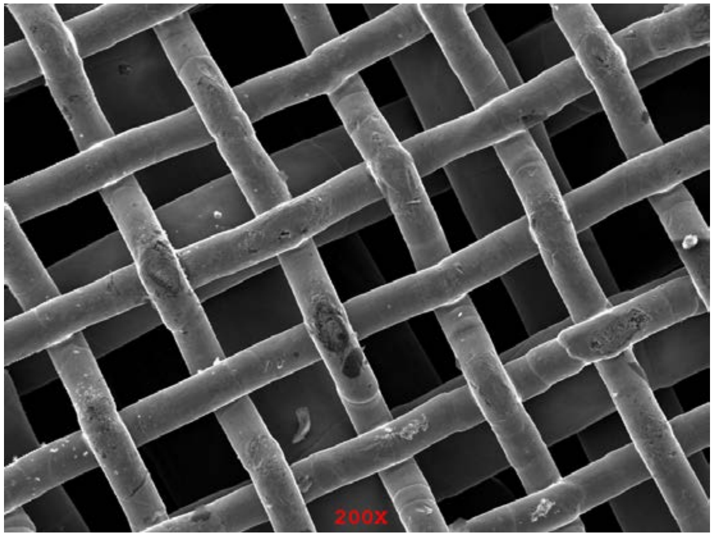


Post Burn-off TMS

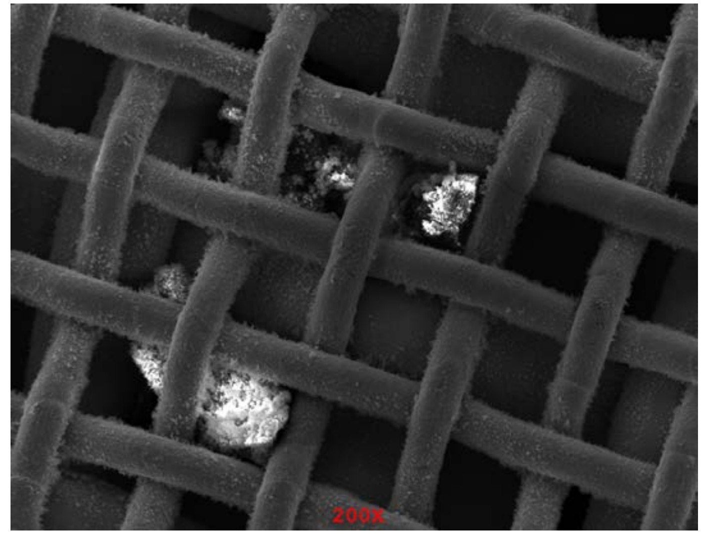


Run 131 Jet A+1000 ppm AQ, GDTC

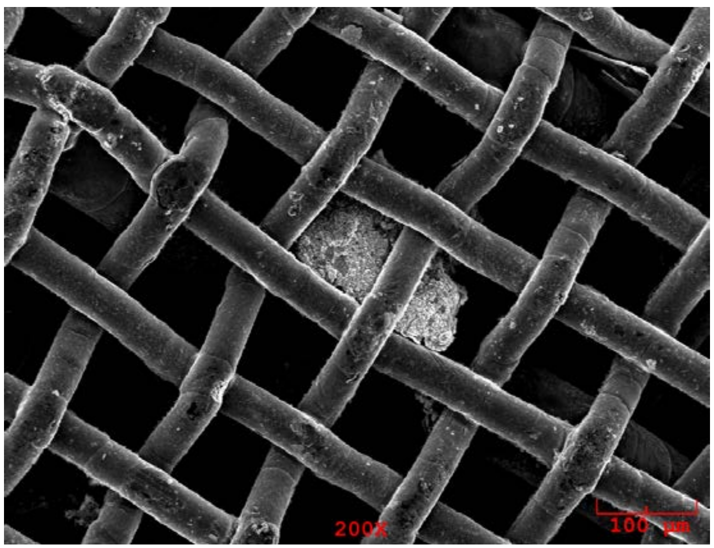
Figure 83 - SEM of TMS at Apprximately 20X - Clean New, Post -Burnoff and Runs 129 and 131



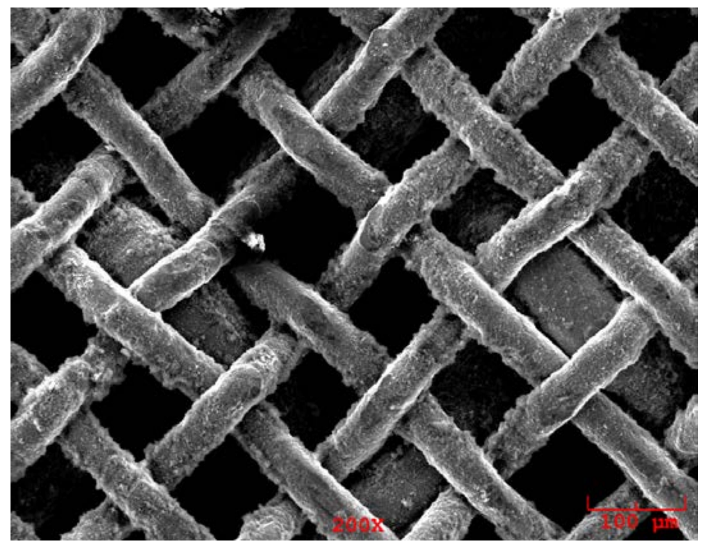
Clean New TMS



Run 129 Baseline Jet A, GDTC

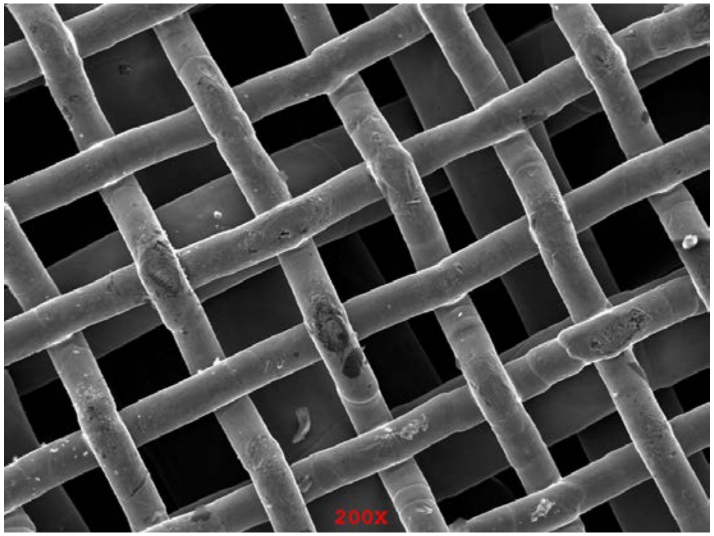


Post Burn-off TMS

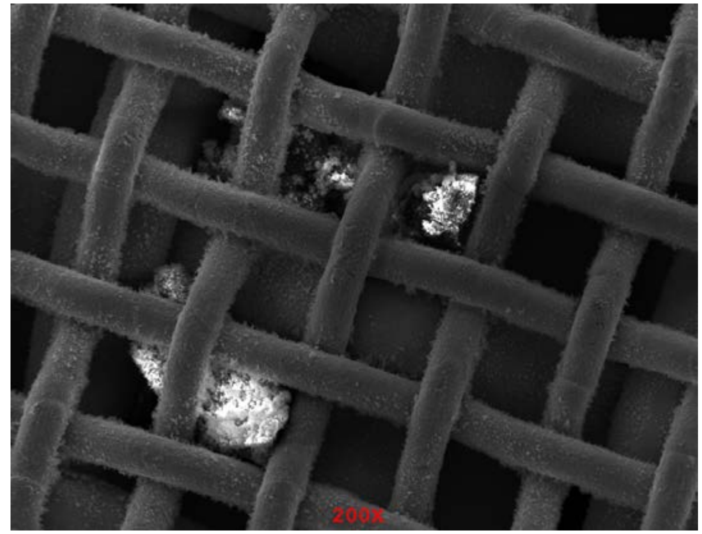


Run 133 Jet A+1000 ppm AQ, GDTC

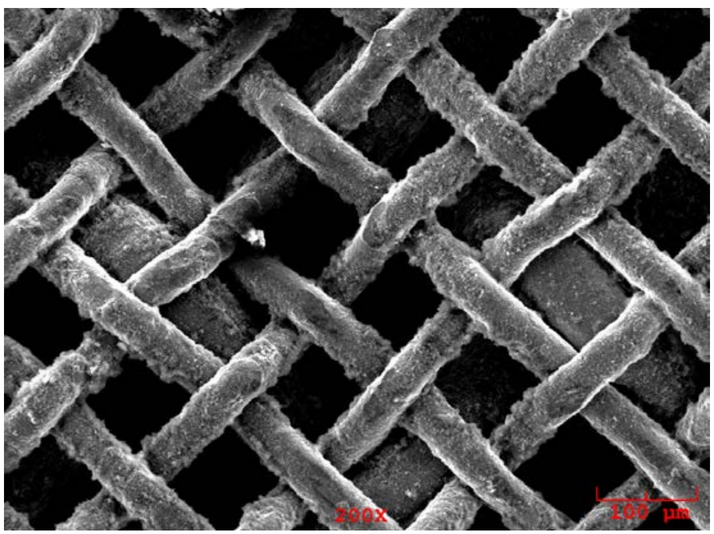
Figure 84 - SEM of TMS at Apprximately 200X - Clean New, Post -Burnoff and Runs 129 and 133



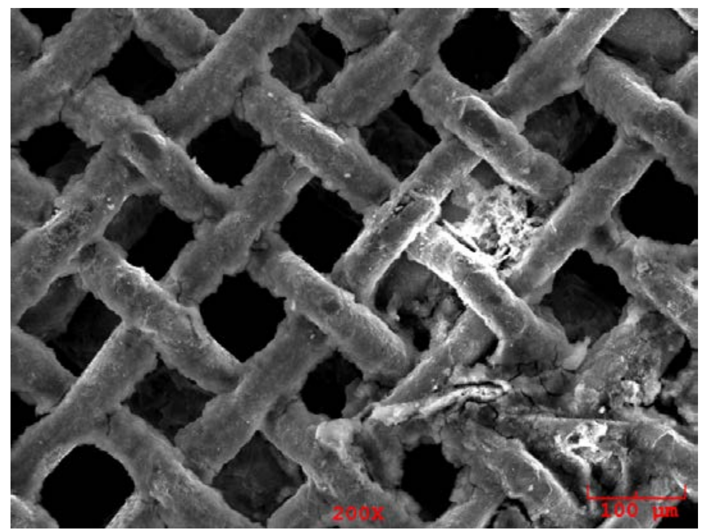
Clean New TMS



Run 129 Baseline Jet A, GDTC



Run 133 Jet A+1000 ppm AQ, GDTC



Run 135, Jet A+250 ppm AQ+200 ppm Water, EDTST

Figure 85 - SEM of TMS at Apprximately 200X - Clean New, Post -Burnoff and Runs 129, 133 and 135

Appendix C – ICP Analysis by BASF

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BASF
The Chemical Company

Analytical North America

Analytical Report	**Date Printed: 12/01/14**
Request: 2014002615	**Project: 60015260**
Description: Jet Fuel Samples for ICP/MS	
Submitter: Zeld-Stephen	
Coordinator: Yee-Ed	
Entry Date: 10/1/2014	**Completed Date: 10/15/2014**
Copies To: Dietmar PosselT	

For information about Analytical NA's services: Final Report Electronically
http://iwww.basf-corp.com/NP/ACCE/AnalyticalNorthAmerica/index.htm Transmitted ☒

INSTRUCTIONS

Jet fuel samples including some containing our Aquarius additive were analyzed for the US Air Force by an outside lab using ICP/MS. Significant discrepancies between samples were found for a number of elements. Please run ICP/MS on the three submitted fuel samples to determine if more consistent results can be obtained for the following elements: Al, Sb, As, Ba, Be, Bi, B, Cd, Ca, Cr, Co, Cu, Fe, Pb, Li, Mg, Mn, Hg, Mo, Ni, P, K, Se, Si, Ag, Na, Sr, Sn, Ti, V, Zn, Zr

Sample Information:
11769-1 (Tank S-3 NEAT)
11769-2 (Tank S-16 NEAT FUEL BASELINE STOCK)
117770 (Tank S-4 ADDITIZED)

Note that 11769-1 and -2 are not additized.

RESULTS

Elemental Analysis – Denise Grimsley and Keqing Fa

Background

Three fuel samples were received for trace element analysis. A data sheet with results from SGS was attached. The SGS data were considered inconsistent, particularly for B, V, Cu, and Mn. Results between samples were found to be significantly different, while it was believed that the elemental levels should have been similar. For example, the B concentrations from the SGS data sheet are <5 ppb and 848 ppb in two different samples.

The SGS data show excellent detection limit values, with most of them at 5 ppb. Such detection limits suggest that the samples were examined on a high resolution ICP MS or ICP MS/MS. To achieve a 5 ppb detection limit, many elements would have to be examined at around a 50 ppt level, which is challenging in the presence of interferences. However, it is difficult to understand the inconsistency of the B, V, Cu and Mn results if such powerful instruments were used.

Initially the WY lab's task was to resolve the inconsistencies in the above mentioned elements. Later over 30 elements were requested to be tested with ICP MS. The Elemental Analysis Lab at WY is equipped with a Perkin Elmer NexION 300D spectrometer with collision cell and reaction cell to minimize/eliminate interference. It is neither a high resolution ICP MS nor an ICP MS/MS. Certain elements can pose significant difficulties due to interferences. They are not very effectively measured on the NexION 300D. These elements include Ca, K, P, and Si.

Approval:________________ **Reviewed:** ________

Experimental

Samples were first prepared in nitric acid on a microwave-assisted digestion unit. However, once the samples were diluted by water and allowed to cool, the solutions became cloudy. Additional nitric acid and hydrochloric acid were then added, and the solutions were further cooked on a hotblock unit at 120°C to eliminate the cloudiness. The clarified solutions were then analyzed with ICP MS.

The ICP MS was tuned prior to use, which included torch alignment, mass calibration, deflection, and Ar gas. Detection methods utilized standard mode, KED (collision cell), and DRC (reaction cell) to understand, minimize, or eliminate interferences. Many elements were detected at multiple available masses if possible.

Calibration curves were established using blank, 5 ppb, 20 ppb, and 40 ppb standards. Elements were separated into three groups based on the availability of standards and matrix considerations.

Results

Results are given in Table 1. Estimated method detection limit values were also provided in Table 1 for reference.

Each sample was examined multiple times. It is difficult to measure certain elements such as Ca, Fe, K, P, and Si because of the presence of interferences. The data consistency will be affected by these interferences. The elements with the "*" note in Table 1 were considered difficult in this study. The lab needs more time for method development if better results for these elements are desired. One improvement would be sample preparation. The lab currently has no super clean crucibles for use in ashing. It might also be a good idea to do dry ashing and then acid digestion to remove as much carbon as possible to minimize or eliminate the interferences arising from carbon-containing species.

It was also found that the consistency between samples for elements B, Mn, Cu, and V can be controlled so that reproducible results are obtained.

The samples were also analyzed with ICP AES. Please see Table 2 for results. Values that are less than the method detection limits (MDL) are reported as not detected (ND). Values that are greater than the MDL but less than the practical quantification limit (PQL) are reported as "<PQL".

It is worthwhile to point out that acid digestion might not be effective for aluminum, silicon, and titanium oxides. In the Wyandotte lab samples generally need to be prepared by a fusion method in order to measure Al, Si, and Ti.

Note 1: If results between duplicate don't agree well, individual values were provided in Tables.

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Request: 2014002615 **Project: 60015260**

Table 1 Elemental analysis results from ICP MS and ICP AES

Element	Tank S-3 NEAT (ppb)	Tank S-16 NEAT (ppb)	Tank S-4 ADDITIZED (ppb)	MDL (ppb)	Note
Aluminum Al	213	208	449	25	*
Antimony, Sb	<15	<15	<15	15	
Arsenic, As	<25	<25	<25	25	
Barium, Ba	<15	<15	<15	15	
Beryllium, Be	<15	<15	<15	15	
Bismuth, Bi	<15	<15	<15	15	
Boron, B	<25	<25	<25	25	
Cadmium, Cd	<15	<15	<15	15	
Calcium, Ca	2.3 ppm	2.8 ppm	2.4 ppm	25	*
Chromium, Cr	<25	<25	<25	25	
Cobalt, Co	68	65	415	25	*
Copper, Cu	48	27	45	25	
Iron, Fe	966	55	75	25	*
Lead, Pb	<15	<15	<15	15	
Lithium, Li	<15	19	19	15	
Magnesium, Mg	56	58	60	25	*
Manganese, Mn	<25	<25	<25	25	
Mercury, Hg	<15	<15	<15	15	
Molybdenum, Mo	<25	<25	<25	25	
Nickel, Ni	<15, 122	44, 136	<15, 330	15	
Phosphorus, P	<500	<500	<500	500	*
Potassium, K	<25	<25	<25	25	*
Selenium, Se	<25	<25	<25	25	
Silicon, Si	<1 ppm	<1ppm	<1ppm	25	*
Silver, Ag	<15	<15	<15	15	
Sodium, Na	<25	<25	<25	25	
Strontium, Sr	17	<15	<15	15	
Tin, Sn	<15	<15	<15	15	
Titanium, Ti	80	60	90	15	
Vanadium, V	<15	<15	<15	15	
Zinc, Zn	360	183	<25	25	*
Zirconium, Zr	<25	<25	<25	25	

Table 2. Elemental Analysis Results by ICP AES in ppm.

Element	S-3 (ppm)	S-16 (ppm)	S-4 (ppm)	Estimated MDL
Al	ND	ND	ND	0.5
As	ND	ND	ND	4.0
B	ND	ND	ND	4.0
Ba	ND	ND	ND	0.1
Be	ND	ND	ND	0.2
Ca	3	ND, 4	2, 4	0.2
Cd	ND	ND	ND	0.5
Co	ND	ND	<1.2, ND	0.5
Cr	ND	ND	ND	0.4
Cu	ND	ND	<0.3	0.1
Fe	3	3, 0.7	2.7, <0.6	0.2
K	ND	ND	ND	1.0
Li	ND	ND	ND	0.1
Mg	ND	ND, <0.3	ND	0.1
Mn	ND	ND	ND	0.1
Mo	ND	ND	ND	4.0
Na	ND	ND	ND	0.4
Ni	ND	ND	ND	1.0
P	ND	ND	ND	4.0
Pb	ND	ND	ND	3.0
S	44	37	37	4.9
Sb	ND	ND	ND	2.5
Se	ND	ND	ND	4.9
Si	<3	ND	ND	1.0
Sn	ND	ND	ND	4.9
Sr	ND	ND	ND	0.1
Ti	ND	ND	ND	0.3
V	ND	ND	ND	0.3
Zn	ND	ND, 1	<0.6	0.2

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Analytical Report		Date Printed: 12/01/14
Request: 2014002901	Project: 60015260	
Description: Jet Fuel Samples (Aquarius Additve)		
Submitter: Zeld-Stephen		
Coordinator: Yee-Ed		
Entry Date: 10/29/2014	Completed Date: 11/19/2014	
Copies To:		

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INSTRUCTIONS

Additional jet fuel samples have been submitted for analysis with ICP/MS using the procedures developed in request 2014002615. Please report the same elements as in this earlier request.

Sample Information:

1. 11770 RUN 133 BFA - Jet A + 1000 ppm AQ taken off the rig, at the rig exit
2. 11770 RUN 134 BFA - Jet A baseline fuel taken off the rig, at the rig exit
3. 11829 RUN 135 BFA - Jet A + 250 ppm AQ+ 200 ppm total water. Sample taken off the rig at the rig exit.

RESULTS

Elemental Analysis – Denise Grimsley, Keqing Fa

Background

As part of the continuing effort to support the Aquarius Additive project, three fuel samples were received for the same trace element analysis as was performed in request 2014002615. A procedure similar to that previously used was requested to test the new samples.

Experimental

A similar procedure was used to test these three samples. Samples were first prepared in nitric acid on a microwave assisted digestion unit. After transfer of the digests to plastic sample tubes using water, additional nitric acid and hydrochloric acid were added, and the solutions were further cooked on a hotblock unit at 120 °C. The solutions were then analyzed with ICP MS.

The lab made efforts to understand the effects of cleanness on the results. Both bare quartz tubes and quartz tubes + plastic liners were used for the sample preparation. Therefore each sample was actually tested four times.

ICP MS detection parameters for each isotope were optimized for greater effectiveness in removing interferences and promoting sensitivities. In particular, the reaction gas flow and RPq parameters in DRC cell technology were optimized, and the KED cell gas flow was selected based on the sample matrix.

The ICP MS was tuned prior to use, including torch alignment, mass calibration, deflection, and Ar gas. Detection methods utilized the standard mode, KED (collision cell), and

Approval:____________________ **Reviewed:** __________

DRC (reaction cell) to understand, minimize, or eliminate interferences. Many elements were detected at various available masses if possible.

Calibration curves were established using blank, 5 ppb, 20 ppb, and 40 ppb standards. Elements were separated into three groups based on the availability of standards and matrix considerations.

The leftover samples were examined by ICP AES to confirm the results from ICP MS.

Results

The results of the ICP MS analysis are given in Table 1. Estimated method detection limit values are also provided in Table 1 for reference. If the results from multiple runs do not agree well, individual values are provided instead of averages.

Difficult elements, such as Al, Ca, Fe, K, P, and Si, etc., are denoted as "*" in Table 1 because of the presence of interferences that affect data consistency. In addition, acid digestion might not be effective on some oxides of Al, Si, and Ti. Their results need to be verified by other sample preparation techniques.

Variations for Sb, Sn, and Ni are observed in Table 1. Initially there was a concern about the possible contamination from the quartz tubes. However, it is also conceivable that the samples contain microscopic particles, possibly originating from the test rig. The variation in the results would then arise because the particles are sampled in one replicate but not in another. One run for sample 11829 RUN 135 BFA found a high level of Fe, Co, Cr, Si, and other elements, as shown in the last column of Table 2. Although the ICP MS results are in agreement with the ICP AES results for this sample, the values for this sample in Table 1 do not include the results from this run.

Results from ICP AES are shown in Table 2. Values that are less than the method detection limits (MDL) are reported as not detected (ND). Values that are greater than the MDL but less than the practical quantification limit (PQL) are reported as "<PQL".

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Table 1 Elemental Analysis Results from ICP MS in ppb

Element	11770 RUN 133 BFA (ppb)	11770 RUN 134 BFA (ppb)	11829 RUN 135 BFA (ppb)	MDL (ppb)	Note
Aluminum Al	87	133	215	25	*
Antimony, Sb	184, <25	<25	71, <25	25	
Arsenic, As	<25	<25	<25	25	
Barium, Ba	<15	<15	<15	15	
Beryllium, Be	<15	<15	<15	15	
Bismuth, Bi	<15	<15	<15	15	
Boron, B	<25	<25	<25	25	
Cadmium, Cd	<15	<15	<15	15	
Calcium, Ca	62	242	411	25	*
Chromium, Cr	<25	<25	<25	25	
Cobalt, Co	<25	<25	<25	25	
Copper, Cu	<25	<25	<25	25	
Iron, Fe	<25	<25	<25	25	*
Lead, Pb	<15	<15	<15	15	
Lithium, Li	<15	<15	<15	15	
Magnesium, Mg	<25	<25	<25	25	*
Manganese, Mn	<25	<25	<25	25	
Mercury, Hg	<15	<15	<15	15	
Molybdenum, Mo	<25	<25	<25	25	
Nickel, Ni	<15, 88, 173	<15, 62	<15, 52	15	
Phosphorus, P	<500	<500	<500	500	*
Potassium, K	<25	<25	<25	25	*
Selenium, Se	<25	<25	<25	25	
Silicon, Si	NA	NA	NA	25	* See ICPAES
Silver, Ag	<15	<15	<15	15	
Sodium, Na	<25	<25	104, <25	25	
Strontium, Sr	<15	<15	<15	15	
Tin, Sn	107, 68, <15	132, 69, <15	171, 21, <15	15	
Titanium, Ti	<15	<15	<15	15	*
Vanadium, V	<15	<15	<15	15	
Zinc, Zn	<25	<25	<25	25	*
Zirconium, Zr	<25	<25	<25	25	

*Elements that are difficult to analyze

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Table 2. Elemental Analysis Results by ICP AES in ppm.

Element	11770 RUN 133 BFA (ppm)	11770 RUN 134 BFA (ppm)	11829 RUN 135 BFA (ppm)	Estimated MDL	A "dirty" 135 BFA (ppm)
Al	ND	ND	ND	0.4	<1.2
As	ND	ND	ND	3.4	ND
B	ND	ND	ND	0.3	ND
Ba	ND	ND	ND	0.1	ND
Be	ND	ND	ND	0.2	ND
Bi	ND	ND	ND	0.9	ND
Ca	ND	ND	ND	0.2	3
Cd	ND	ND	ND	0.4	ND
Co	ND	ND	ND	0.4	13
Cr	ND	ND	ND	0.3	4
Cu	ND	ND, <0.3	<0.3	0.1	<0.3
Fe	ND	ND	ND	0.2	25
K	ND	ND	ND	0.9	ND
Li	ND	ND	ND	0.1	ND
Mg	ND	ND	ND	0.1	<0.3
Mn	ND	ND	ND	0.1	<0.3
Mo	ND	ND	ND	0.9	ND
Na	ND	ND	<1	0.3	<1
Ni	ND	ND	ND	0.9	<3
P	ND	ND	ND	3.4	ND
Pb	ND	ND	<5	2.6	ND
S	43	39	43	4.3	41
Sb	ND	ND	ND	2.1	ND
Se	ND	ND	ND	4.3	ND
Si	ND	4, 8	ND	0.9	4
Sn	ND	ND	ND	4.3	ND
Sr	ND	ND	ND	0.1	ND
Ti	ND	ND	<1	0.3	<1
V	ND	ND	ND	0.3	ND
Zn	ND	ND	ND	0.2	ND
Zr	ND	ND	ND	0.2	ND

Appendix D – MSDS Blue Gold Cleaner

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FILE
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modern chemical, inc.
P.O. BOX 368, JACKSONVILLE, AR 72078

TELEPHONE: (501) 988-1311
FAX: (501) 988-2229
www.bluegoldcleaner.com
E-mail: bluegold_cleaners@email.com

MATERIAL SAFETY DATA SHEET

REVISION DATE: 06/23/2011 PREPARED DATE: 01/01/2013

DISTRIBUTED BY: Modern Chemical & Authorized Distributors, 1-501-988-1311

MANUFACTURED BY: Abernathy Company, 3800 Abernathy Drive, Texarkana, AR 71854

EMERGENCY PHONE: **1-800-535-5053 (INFOTRAC)**

SECTION 1 – PRODUCT

Name: Blue Gold Industrial Cleaner
Product Code 109 (360-MC)
Product Type Hard Surface Cleaner/Degreaser

*DGIS

Health	Flammability	Reactivity	PPE
1	0	0	1
Health Haz	Fire Haz	Reactivity	Personal Protection
0=minimal	0=will not burn	0=none	0=not necessary
1=slightly haz	1=FP>141F	1=mild	1=goggles
2=hazardous	2=FP=>73F=<141F	2=strong	2=goggles, gloves
3=serious haz	3=FP<73F		3=goggles, gloves, protective clothes
4=severe haz	4=BP<95F		4=goggles, gloves, & respirator
	FP by PMCC		

SECTION 2 – HAZARDOUS INGREDIENTS

NONE

SECTION 3 – HEALTH HAZARD & FIRST AID

1. Acute Health Effect — None
2. Chronic Heath Effect — None
3. Carcinogen — No
4. Primary Entry Routes:
   a) Skin & Eyes: Repeated contact with the skin may be irritating. Eye contact slightly irritating.
   b) Ingestion: May be harmful.
   c) Inhalation: Inhalation of vapor or mist may be irritating.
5. First Aid:
   a) Skin: Remove contaminated clothing-wash skin with soap and water. If irritation persists get medical attention.
   b) Eyes: Wash eyes with large volumes of water for at least 15 minutes while lifting the upper and lower eyelids and rotating the eyeball. Get medical attention if irritation persists.
   c) Ingestion: Give large volumes of water. Do not induce vomiting. Get medical attention.
   d) Inhalation: Move to fresh air. If symptoms persists seek medical attention.

SECTION 4 – PHYSICAL & CHEMICAL CHARACTERISTICS

1. Physical State	Liquid
2. Color	Blue
3. Odor	Peppermint
4. Solubility in water	Complete
5. Specific Gravity (H2O=1.0)	1.07
6. pH	13.0
7. Freezing Point	N/A
8. Flash Point	None (Will Not Burn)
9. Vapor Pressure	N/A
10. VOC	0.5% at 5% use dilution rate 5 grams per liter of VOC in 5% dilution

SECTION 5 – FIRE AND EXPLOSION HAZARD

1. Flash Point	None (Will Not Burn)
2. Extinguishing Media	N/A
3. Special Fire Fighting Procedures	N/A
4. Unusual Fire & Explosion Hazard	Fire fighters should observe all precautions that apply to any fire where chemicals are stored.

SECTION 6 – REACTIVITY DATA

1. Stability	Stable
2. Conditions to Avoid	None Known

SECTION 7 – SPILL OR LEAK PROCEDURES

1. If product leaks or spills – Flood area with water – mop up dispose to sanitary sewer.
2. Abide by Federal, State, and Local regulations.

SECTION 8 – PERSONAL PROTECTION

1. Wear goggles.

SECTION 9 – SPECIAL PRECAUTIONS

1. Store containers tightly closed and in an upright position.
2. Do not destroy or deface the label.

SECTION 10 – SECTION 313 SUPPLIER NOTIFICATION (SARA)

This product contains the following toxic chemicals subject to the reporting requirements of Section 313 of the Emergency Planning and Community Right-To-Know Act of 1986 and of 40 CFR 372:

2-(2-butoxyethoxy) ethanol	CAS# 112-34-5	Wt%=9.0

SECTION 11 – TOXICOLOGICAL INFORMATION

Oral Toxicity Rats	LD >5050 mg/kg
Dermal Irritation Rabbit Protocol S9-FF81-5.M21	No observed irritation
Acute Dermal Toxicity Rabbit	LD50 >2020 mg/kg
Eye Irritation Rabbit Protocol S9-FF81-4.M21	Moderate Eye Irritant

The above test results indicate that Blue Gold Industrial Cleaner is not corrosive or toxic through skin contact or inhalation.

SECTION 12 – ECOLOGICAL INFORMATION

Results of Aerobic Aquatic Biodegradation conducted according to 40CFR796.3100 – based on dissolved organic carbon analysis Blue Gold Industrial Cleaner is 89.8% biodegradable in 28 days.

SECTION 13 – DISPOSAL CONSIDERATIONS

1. See section 7 above

SECTION 14 – DOT TRANSPORT INFORMATION

1. This product is not regulated

SECTION 15 – OTHER REGULATORY INFORMATION

All ingredients appear on the TSCA Inventory List

SECTION 16 – OTHER INFORMATION

1. N/A = Not Applicable
2. *Dangerous Goods Identification System (DGIS)
3. PMCC = Pensky Martin Closed Cup
4. RB Chemical Company believes that the information given here is accurate. The suggested procedures are based on experience and common sense and are not necessarily all-inclusive of every conceivable circumstance.