Storage Stability Test

PAFI-RTP-004

PAFI Rig #4 Storage Test

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None	12/22/20	All	Initial Release
A	11/29/22	Multiple	General update for organizational changes and current formatting. Includes edits to allow for public release of the document. Added disclaimer and updated preface on pages 2/3. Changed references of "test fuels" to the test fuel. Added safety notes to section 1.1.
A	6/13/25	Appendix A	Updated Appendix A with pre and post fuel testing

PREFACE

This test plan specifies the requirements for conducting comparative materials compatibility screening tests under the FAA's piston aviation fuels initiative (PAFI). This document is derived from extensive FAA and industry experience and best practices for evaluating the suitability of aviation fuels for use in the piston powered general aviation fleet. This document is used by the FAA to compare and evaluate new aviation fuels relative to the performance of 100LL aviation gasoline that meets ASTM D910.

In addition to the candidate unleaded fuel, a high aromatic ASTM D910 fuel formulation is required for the screening tests under this plan.

This document is not intended to provide a method to obtain data to serve as a showing of compliance to regulatory requirements for the certification of aircraft or aircraft engines.

Furthermore, the material in this document does not change or permit deviations from existing regulatory requirements. This material is neither mandatory nor regulatory in nature. Use of this document or any derivative, in an attempt to obtain an FAA authorization of an alternative aviation fuel, should be pre-coordinated and approved by the appropriate FAA office(s) as part of an approved qualification program.

Successful completion of this test plan provides data that may support both FAA Fleet authorization and development of an ASTM production specification for PAFI Fuels. It also allows the collection of data to be used to generate performance and detonation performance values in support of other candidate fuel types being investigated under various FAA Research & Development projects.

References made to the Alternative Fuels Program Office or AFPO, refer to the group within the FAA that is responsible for the PAFI program. At the time of this revision, that group is the AIR-650 organization. Please see https://www.faa.gov/about/initiatives/avgas/org_info.

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Materials Test Plan
PAFI-RTP-004
Storage Stability Test

1. SCOPE

This test plan specifies the requirements and procedures for conducting comparative storage stability testing of an unleaded fuel to generate data required for participating in the FAA Piston Aviation Fuels Initiative (PAFI) test program.

This data will be used for the evaluation of an unleaded fuel being tested in the PAFI test program. The data will be used as part of the final evaluation of data for fleet authorization.

Specific objectives of this plan are stated as follows:

1) Execute long-term (12 week or longer) storage stability testing under hot, humid, ambient conditions, and collect fuel performance data to assess fuel propensity for separation, degradation, collection of water, or other anomalous behavior with time.

2) Execute elevated temperature (accelerated) storage stability testing at 43°C (110°F) and collect fuel performance data to assess fuel degradation due to changes in oxidative and thermal stability, and in total acid number.

The test plan includes the requirements for confirming baseline condition, test set-up, instrumentation, specimen collection and supporting documentation such as fuel certification documents as prerequisites to the testing identified.

The long-term, storage test is to be performed at a location that provides warm/hot, humid ambient conditions, for example at a southern U.S., coastal location, over a 12-week (3 month) minimum period. Testing involves collecting samples and inspecting the fuel every three weeks for a minimum of 12 weeks. Data to be collected include: formation of water bottoms and increasing moisture content, bulk evaporation, deposition formation, and microbial growth within the test rig. In addition, one-gallon samples will be collected and analyzed for changes in existent gums, Saybolt color, motor octane number, total acid number, water reaction, and copper strip corrosion. Other tests include vapor pressure, electrical conductivity, hydrogen content, sulfur content, heat of combustion, and freeze point. The fuel remaining after 12 weeks shall have a full certificate of analysis performed.

Note: The location for the testing must be identified and submitted to the FAA Alternative Fuels Program Office (AFPO) for approval prior to beginning testing. The location must have a historical record of elevated temperatures and humidity for the majority of a 12-week period.

The accelerated storage test is to be performed at 43 °C (110 °F) using sealed containers. Testing involves collecting samples every three weeks for 12 weeks

and replenishing the available oxygen within the test vessel. Each sample shall be analyzed for moisture content, filterable solids, and adherent insolubles. The fuel after 12 weeks (3 months) shall have a full certificate of analysis performed.

Requirements for confirming test equipment configuration, test instrumentation, and supporting documentation such as fuel certification and equipment calibration/validation are pre-requisites to the testing identified.

1.1. PROVISOS

The following identifies significant assumptions, requirements, and provisions associated with the testing prescribed within this test plan. Testing performed pursuant to this test plan provides data supporting FAA fleet authorization for PAFI unleaded fuels. Testing performed to this test plan may be witnessed by an identified designated representative of the FAA. This may be achieved through the use of a Designated Engineering Representative (DER) or other representative approved by the AFPO staff prior to executing testing.

NOTE: In the event testing is conducted outside the FAA's William J. Hughes Technical Center, testing performed to this test plan must be witnessed by an identified designated representative of the FAA. This may be achieved through the use of a Designated Engineering Representative (DER) or other representative approved by the FAA AFPO or PAFI Advisory Committee prior to executing testing.

NOTE: *Testing performed pursuant to this test plan provides data supporting 1) FAA fleet authorization for each PAFI unleaded fuel, and 2) the development of an ASTM International aviation gasoline production specification for each fuel.*

2. OBJECTIVES

The objective of the Storage Stability Test Plan comparative fuel performance tests is defined as follows.

1) *Determine the stability of unleaded aviation fuel under warm, humid conditions and compare its stability to leaded aviation fuel.*
2) *Determine the accelerated stability of unleaded aviation fuel under controlled elevated temperature conditions (43 °C) and compare its stability to leaded aviation fuel.*

3. BACKGROUND

Bulk fuels stored for long periods of time can suffer from fuel storage degradation and contamination, resulting in serious fuel instability problems when eventually used. Problems encountered include polymerization and oxidation. The formation of filterable solids and gums can result in operational problems when the fuel is used. Depending on the location, aviation gasoline storage tanks may be smaller

(500 to 2500 gallons) with lower turnover rates than experienced with jet fuel storage tanks.

The fuel tanks on aircraft are susceptible to the same concerns, however, have a smaller mass than a bulk tank and may be more susceptible to degradation. No industry accepted storage stability test specific to smaller tanks such as those on aircraft is available. These properties relate to the thermal stability, oxidation stability, and acid number of the fuel.

Historically aviation gasoline has displayed an observably stable nature. This is due primarily to the chemical composition of aviation gasoline produced in compliance with ASTM D910, and the presence of lead which provides an antimicrobial function. In order to blend fuels which have the necessary detonation resistance, components with different properties with respect to both thermal and oxidative stability, and with respect to its resistance to microbial growth, may be used.

Currently no standard industry test for evaluating the long-term storage stability of aviation gasoline under ambient conditions exists. An industry accepted method, ASTM D4625, exists for evaluating accelerated bulk storage stability of jet fuel at 43 °C (110 °F), but not for gasoline.

The accelerated test method is being applied to the candidate unleaded fuel under PAFI. However, due to the higher evaporation rates of the aviation gasoline test fuel; this existing jet fuel test method has been modified. The existing ASTM testing involves the use of vented glass jars stored at 43 °C for the duration of the test. Specimens are collected at timed intervals and tested for the formation of soluble and insoluble gums created by oxidation of the fuel. This vented system is not practical for the gasoline test fluids due to their higher evaporation rates. The modified method involves sealed containers with routine introduction of air to replenish the oxygen in the system. The modifications to the test method (sealing of the individual specimens) are based on existing industry research (SwRI gasoline studies). In all other ways, the interval testing is performed in compliance with the ASTM D4625 test method.

Accordingly, this test plan prepared for the FAA PAFI Rig screening test, provides for the development of a common set of validation data supporting compliance with both ASTM and FAA requirements. Currently there are no published regulations for thermal or oxidative stability properties. ASTM D7826 provides guidance in evaluating the comparative behaviors of fuels and the handling of non-comparative outcomes.

The scope and extent of the FAA PAFI Storage Stability Test plan is defined by TAC-PROJ-015, latest revision, which integrates the findings and recommendations of the PAFI Technical Team meeting of October 9, 2014.

4. PAFI RIG SCREENING TEST PLANS

The matrix of rig screening test plans supporting the test protocols identified in TAC-PRJ-008, latest revision, is presented as follows. The test plans will follow a common format with general procedures, processes, and test instrumentation being common to all. The current plan is highlighted in the list below.

RIG SCREENING TEST PLANS

PAFI-RTP-001, Cold Flow Hardware Test
PAFI-RTP-002, Carburetor Icing Test (Removed)
PAFI-RTP-003, Dynamic/Operational Hardware Test (Removed)
PAFI-RTP-004, Long Term Storage Test, Ambient and Elevated
PAFI-RTP-005, Cold Storage Stability Test
PAFI-RTP-006, Hot Surface Ignition Temperature Test (Removed)
PAFI-RTP-007, Full Scale Engine Emissions Test (Not Released)
PAFI-RTP-008, Comingling Evaluation with 100LL (Not Released)
PAFI-RTP-009, Dynamic Diaphragm Test
PAFI-RTP-010, Fuel Sump Valve Test
PAFI-RTP-011, Vented Drum Storage Test

Test plans prepared in support of the FAA PAFI test program for unleaded fuels are subject to the following review and approval protocol.

1) PAFI rig test plans follow a format and content consistent with established practice for FAA compliance projects.
2) Draft test plans are furnished to the FAA AFPO office for review and approval prior to release to the TAC.
3) Upon concurrence from the FAA AFPO, the test plan will be formally furnished to the PAFI TAC for review and concurrence preceded by FAA Contracts review and authorization for release to the TAC.
4) A final test plan will be released incorporating changes resulting from the TAC review.
5) All changes and referrals to this test plan should reference the latest revision level and date.

5. APPLICABLE FAA, SAE & ASTM REQUIREMENTS

The following is a listing of the applicable FAA, SAE, and ASTM documents which contain information relevant to the testing specified by this test plan.

5.1. FAA REGULATIONS

There are no FAA Regulations applicable to the testing.

5.2. FAA ADVISORY CIRCULARS & ORDERS

There are no FAA Advisory circulars or orders applicable to the testing.

5.3. SAE STANDARDS

There are no SAE standards applicable to the testing.

5.4. ASTM GUIDANCE & PRACTICES

ASTM guidance and standards applicable to the testing identified are listed as follows. Data provided by this test plan will support the development of an ASTM production specification for the unleaded fuel.

1) ASTM D4625 Standard Test Method for Middle Distillate Fuel Storage Stability at 43 °C (110 °F)

2) ASTM D7826-19a Standard Guide for Evaluation of New Aviation Gasolines and New Aviation Gasoline Additives

6. APPLICABLE OEM DOCUMENTS

There are no applicable OEM documents required.

7. DESCRIPTION OF TEST ARTICLES

7.1. AMBIENT STORAGE STABILITY

This test plan covers the evaluation of changes to both the fuel and the fuel tank environment as a result of changes in the fuel. Multiple test articles are required within each individual task. In addition, the fuel itself, collected over the duration of the test, also represents a test article.

7.1.1. GLASS JARS

In order to assess the propensity of a fuel to draw and settle water from the ambient air, it is necessary to observe the fuel tank bottoms. It is not practical for the researcher to inspect the bottoms of the ambient tank rig, so a means of collecting the bottoms and bringing them to the researcher is required. The use of glass jars placed at the bottom of the storage tank permits the extraction of fuel tank “bottoms” without unacceptable mixing. The glass jars shall have a minimum volume of one quart (one liter). While one-quart, wide mouth canning jars have been successfully used in previous testing, any glass jar with the necessary volume and a wide mouth is acceptable. Five jars per fuel sample are required.

7.1.2. GLASS RODS

To assess the propensity for the formation of sediment, deposits, or microbial growth, it is necessary to provide a surface for collection of any deposits which are subsequently inspected. Standard 1.25 to 2.5 cm (½ to 1 inch) diameter borosilicate glass rods with a length similar to the height of the fuel tank rig are required. Five rods per fuel sample are required.

7.1.3. WOODEN DOWELS

In an attempt to assess how quickly fuel is likely to evaporate from a fuel tank over time, a means of positively measuring fuel volume is required. Because the diameter of the fuel tank rig is constant, the fuel volume can be determined by measuring the fuel depth. Because one gallon fuel samples will be collected at each inspection, it is necessary to measure the fuel depth after all sampling is completed during the inspection, and to compare this value to the fuel depth after the following three-week exposure time. Previous testing has found that standard 1 to 2.5 cm (½ to 1 inch) diameter untreated hardwood wooden dowels can be successfully used. The use of pine dowels is not recommended. These dowels also provide a support for the water indicating paste used to inspect the tank bottoms for free water at each inspection. Five dowels per fuel sample are required.

7.1.4. AIRCRAFT FUEL FILLER CAP

As part of the material compatibility testing, the long-term storage stability testing is best suited for testing fuel filler caps for fuel compatibility. A standard aircraft fuel cap shall be installed into the fuel tank rig aluminum lid (Figure 1). One fuel cap per fuel sample is required.

Figure 1 - Fuel Filler Cap Installed in Ambient Storage Rig Lid

7.2. ACCELERATED (OVEN) STORAGE STABILITY

This test plan covers the evaluation of changes to the fuel over time under elevated temperature. The fuel itself is the only test article.

8. DEFINITIONS & TERMINOLOGY

Definitions, terminology, and abbreviations specific to this document and to FAA nomenclature are summarized in the following section.

8.1. ADHERENT INSOLUBLES

Adherent insoluble are gums formed during the storage test and are adherent to the specimen container after the test fuel is removed. They are not soluble in the fuel and remain in the container.

8.2. EXPOSURE

Within this document, the term exposure is the execution of a test on a test fluid at a given temperature for a given amount of time.

8.3. FILTERABLE INSOLUBLES

Filterable insolubles are solids formed during the storage test which remain in the fuel and can be removed by filtering the fuel. They are not solubilized by the fuel but are carried in the fuel.

8.4. CALIBRATION

Calibration is a set of actions that establishes that the instrument being calibrated is equal to, or reports values equal to a standard. The unknown, the instrument being calibrated, will be checked against a known, the standard, at several different points and adjustments made to the unknown to bring it into alignment with the standard.

8.5. SAMPLE

Within this document the following concept is meant by the term “sample”. A sample is the specific material to be prepared as specimens and exposed to test fluids. It is also the individual fuel. For example, 100LL AvGas is a sample, and unleaded test fuel is a second sample.

8.6. SAMPLE SET

A sample set is all the individual specimens and all of the test fluids comprising a set of samples to generate a set of data. For example, fluid collected from the 100LL on week 3 and fluid collected from the unleaded fuel sample on Week 3 comprise the “Ambient Exposure, Week 3 Sample Set”.

8.7. SPECIMEN

Within this document the following concept is meant by the term “specimen”. Specimens are the individual pieces/aliquots collected from the sample materials. For example the individual fuel volumes collected from the test sample (the fuel) at each time interval are the test specimens for the ambient storage exposure. Similarly, the individual glass rods are specimens.

8.8. VALIDATION

As opposed to calibration, validation is the act of documenting that an instrument will actually and consistently give expected results. This may be done by measuring a specimen with a known value and confirming the instrument reports that value within the repeatability of the instrument. If the expected value does not result, validation has failed and some additional action must be taken to repair or adjust (calibrate) the instrument.

9. FUEL CONFORMITY

9.1. TEST FUEL

Each PAFI test fuel shall be shown to conform to the complete specification approved by the AFPO, as documented by a CoA furnished with each fuel batch or bulk volume delivered by the fuel supplier to the test facility. In addition, representative data must be generated by an independent 3rd party laboratory for specific pre- and post-test criteria. Pre-test samples should be from fuel collected from the original shipping containers 'as received' by the Test Facility. Analysis must be performed on the fuel samples in accordance with Appendix A. Deviations from the fuel specification shall require approval of AFPO. The full Test Fuel CoA as well as the pre and post-test analysis specified in Appendix A must be included in the test results report.

NOTE: A five-gallon retained sample of the test fuel 'as received' at the test facility must be collected and stored until completion of the PAFI test program. See PAFI-FHP-001.

10. SAFETY PROCEDURES

This document does not purport to address all safety considerations. This document does not replace or supersede individual instrument documentation, Federal, State and local regulations, site procedures, or general industry practices. Appropriate safety precautions are the responsibility of the Test Facility conducting the test. The Test Facility shall establish and ensure compliance with the applicable safety and health standards. The Test Facility shall establish and ensure compliance with the applicable safety and health standards. Prior to initiation of test, each test set-up shall be inspected for safe practices. Consideration for personal protective gear (PPE) is the responsibility of the Test Facility.

10.1. SAMPLE FILL (REFERENCE ASTM D4057-12, SECTION 5.2)

For safety and protection of the integrity of the samples, during transportation and storage, the use of a sample carrier is recommended. A safe fill of between 70-85% volume is recommended unless otherwise instructed. Do not fill over 85% of the container volume.

10.2. STATIC CHARGE (REFERENCE ASTM D4057-12)

10.2.1. GROUNDING (D4057-12, SECTION 5.4.1)

Fires and explosions have occurred as a result of hydrocarbon vapors being ignited by static electricity. If electrical charges are not grounded, they are unable to dissipate and become “static.” This static electric charge can accumulate and freely migrate to a single point on the sample container by a difference in electrical potential, then jump off as a high-energy spark discharge to a nearby less charged surface. The spark may be hot and prolonged enough to ignite nearby hydrocarbon vapors present above the lower explosive limit (LEL). This potential shall be managed by safely dissipating static charges, and through proper grounding, when handling flammable products. This includes grounding the ambient storage test rigs and providing grounding during fuel filtering as part of the laboratory tests.

10.2.2. PERSONAL ATTIRE (D4057-12, SECTION 4.5.2)

Footwear or clothing, capable of causing sparks, should not be worn during fuel handling activities in which flammable vapors are likely to be present. Sampling should not be carried out during periods of atmospheric electric disturbance or hailstorms. To ground any static charge on their body, the individual performing the handling should touch a grounded structure prior to handling fuel.

10.3. VAPOR HAZARDS

All testing must be done in compliance with site and local fire safety protocols as well as applicable NFPA regulations. Appropriate venting of vapors generated during the elevated storage stability test shall be addressed.

10.4. PERSONAL PROTECTIVE EQUIPMENT (PPE)

This document does not replace or supersede any Federal, State, local or site specific requirements related to personal protective equipment. Technicians and test engineers are advised to familiarize themselves with the safety data sheets (SDS) for all of the fluids under test. Recognize that exposure at elevated temperatures may result in the concentration of blend components and applicable PPE should be considered. Individuals are recommended to use at a minimum safety glasses or goggles, petroleum resistant gloves, and lab coats or aprons. Note that fire rated lab coats MAY NOT also be chemical resistant. Closed toed, non-charge isolating footwear is also recommended.

11. PRE-TEST REQUIREMENTS

11.1. SPECIMEN IDENTIFICATION

Specimens in this test plan are comprised of liquid fuels collected from the bottom of the ambient storage stability fuel tank rig, samples collected from the elevated temperature storage stability rig, and samples collected during laboratory testing.

In addition, glass rods and wooden dowels will be collected from the ambient storage stability fuel tank rig.

It is recommended that specimen containers to be used to collect the individual specimens be marked with unique specimen identifiers which are permanently affixed to the specimen containers. The selected means of marking must not interfere with the closure of the specimen container, and must not be removed by accidental contact with the sample material. Means of marking that have proven problematic in previous testing include labeling the glass surface of the container with permanent marker (i.e. Sharpies™), as the test fluids including aviation gasoline will remove the markings. The use of taped labels has been successful as long as the marks were made prior to sampling and the tape was placed on the container prior to use. Other successful methods include the attachment of a pre-marked metal tag wired to the container as long as the attaching wire was of a gage that did not interfere with the cap.

The glass rods and wooden dowels have been successfully identified by using tubes constructed from threaded PVC pipes and caps and attaching permanent specimen identification to the tubes.

11.2. PRE-EXPOSURE FUEL PREPARATION

11.2.1. AMBIENT STORAGE STABILITY TESTING

No special fuel preparation is required for the ambient storage stability testing.

11.2.2. ELEVATED STORAGE STABILITY TESTING

Prior to testing, all of the fuel to be used for preparing the individual 400mL specimens shall be filtered using 0.8 µm filters. Fuel to be used to prepare the five-gallon sample for post-exposure CoA testing does not require filtration.

Cleaning Glassware and Equipment

1) Wash all glassware to be used in filtering the fuel and executing the exposure. Wash the equipment using the alkaline washing procedure described in ASTM D4625. A summary of the process is provided below.
    a. Soak the clean equipment overnight in an alkaline detergent. Only touch the equipment after this point with laboratory forceps and clean gloves.
    b. After the overnight soak, rinse the glassware with distilled water for a minimum of three rinses.
    c. Allow the glassware to dry thoroughly in a laboratory oven at 100°C (212°F).
    d. If glassware is not to be used immediately, cover the glassware and segregate from other laboratory glassware.

Filter the solvents

1) Filter each of the laboratory solvents (iso-octane, iso-propanol, and the 1:1:1 methanol:toluene:acetone flushing fluid) through 0.45-micron filter membranes, using cleaned glassware, see Figure 2.

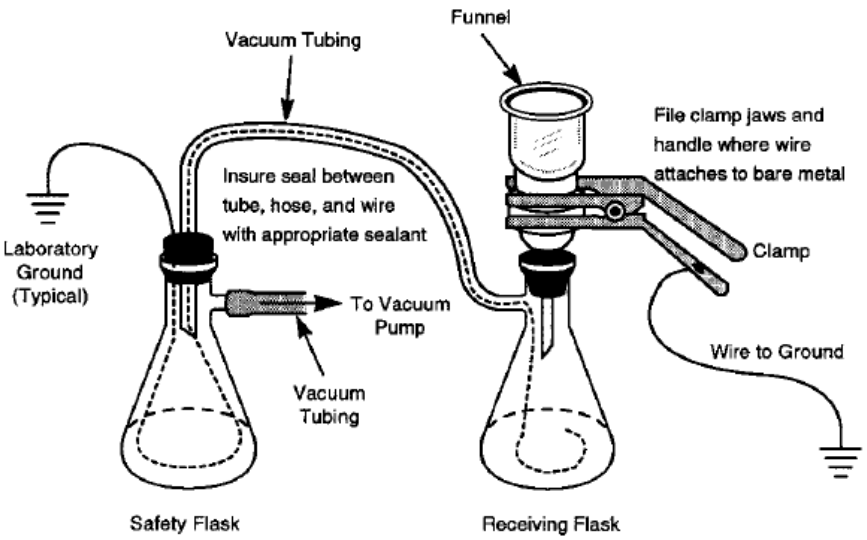
2) Use an aliquot of the filtered solvent to prepare the storage bottles for each solvent.

    a. Rinse the concomitant 4L bottle including the bottle threads and the lid.
    b. Discard the rinsate.
    c. The filtered solvent can now be stored in the rinsed bottle.
    d. Label the bottle as “Filtered” and segregate from other laboratory chemicals. Store in compliance with local safety protocols.

<u>Filter the fuel for testing, (100LL and any test fuels)</u>

1) When ready to filter the fuel samples, procure clean, dry filtration apparatus components and rinse with filtered iso-propanol, followed by filtered flushing fluid. Allow the filtration apparatus to dry and cover to protect from ambient contamination. The apparatus includes the flask stoppers, the receiving flask, the filter support, and the fuel funnel.

2) Assemble the filtration apparatus as shown in Figure 2 using a 0.8-micron membrane filter.



[1]Figure 2 - ASTM D4625 Filtration Apparatus

3) Carefully pour an aliquot of the fuel into the fuel funnel and slowly turn on the vacuum. The filtered fuel is collected in the clean receiving flask.

[1] Reprinted, with permission, from ASTM D4625-21, Standard Test Method for Middle Distillate Fuel Storage Stability at 43 °C (110 °F), copyright ASTM International. A copy of the complete standard may be obtained from www.astm.org.

a. Continue to slowly add fuel to the fuel funnel until sufficient fuel has been filtered to fill all of the sample jars for the 400 mL fuel exposure tests. This will be a minimum of 3 liters of fuel; 400 mL x 7 samples (Week 0, 3, 6, 9, 12 and two spares).

4) Transfer 400 mL to each clean 500 mL sample jar as the fuel is filtered to minimize potential contamination. These jars shall be sealed to prevent evaporation. Testing shall commence within 48 hours of the filtration.

5) Repeat steps 1 through 4 for each additional fuel sample to be tested.

11.3. PRE-EXPOSURE BASELINE DATA

11.3.1. AMBIENT STORAGE STABILITY TESTING

1) At the time of testing, collect a specimen of the fuel "as is." Measure the ambient water content of the test fuels by Karl Fischer Coulometric per ASTM D6304 and record the values. Because of the temperature dependence of water content, this specimen shall not be allowed to warm or cool prior to the analysis. Immediate testing is recommended. This will be used for comparative analysis.

11.3.2. ELEVATED STORAGE STABILITY TESTING

1) Collect pre-exposure insolubles and adherent data per ASTM D4625 on the T=0 filtered fuel.

2) Collect pre-exposure total acid number data on the filtered or unfiltered fuel.

3) If the supplied CoA does not include the data, collect vapor pressure, net heat of combustion, sulfur and hydrogen content, and oxidative stability data on unfiltered fuel.

11.4. PRE-EXPOSURE PHOTOGRAPHS

11.4.1. AMBIENT STORAGE STABILITY TESTING

Exemplar photographs of the ambient testing glass rod and glass jar shall be taken. It is recommended that the set up for photographs include a repeatable backdrop/surface (white or blue), repeatable lighting, and the ability to repeat camera location. It is recommended that a printed identification label be included in the photo that includes sample identification, and that it is pre-exposure.

Note: Consider how the photos will be collected at the test rig when selecting the photography conditions.

11.4.2. ELEVATED STORAGE STABILITY TESTING

Exemplar photographs of the fuel within the glass jar and the pressure vessel shall be taken.

Note: Depending on the color of the test fuel, it may be necessary to place a clean glass stir rod into the fuel to provide a focal point for the camera.

11.5. LABORATORY NOTEBOOK

Either a bound notebook or digital notebook shall be maintained for any exposure testing. Technicians shall record each individual specimen's unique identification, any lot or certification information, and all corresponding pre-exposure data. Each test and test method, as well as any equipment identification shall be recorded. The test results report shall document the manufacturer and model of any analytical equipment used. Information related to the instrument calibration or validation shall be recorded.

This notebook shall also be used to record any start times and dates, test conditions such as temperature, visual observations, and the time and date of test terminations.

Anomalies, issues, troubleshooting, unplanned events, and any other observations shall be recorded in the notebook. Final test data shall be recorded and positively connected to the appropriate pre-exposure data.

12. TEST FACILITY & EQUIPMENT

12.1. FUEL DRUM TRANSFER VALVING

It is assumed that the fuel samples will be stored and transferred using stainless steel drums. In order to dispense the fuel from the drums, the use of dedicated valves and drum vents shall be used. Each valve and vent shall be positively identified and only used with the specific test fuel. Regardless of the receiving and storage methods employed, segregation of the fuels shall be maintained throughout the fuel transfer and handling.

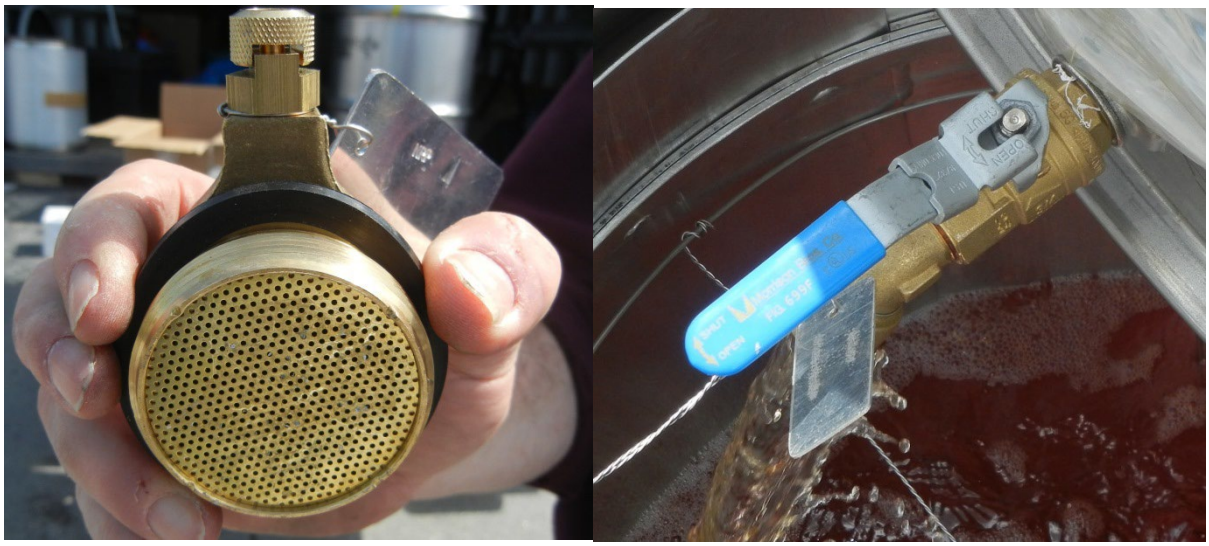


Figure 3 - Dedicated Drum Vent and Valve (Notice Metal ID Tag)

12.2. AMBIENT STORAGE TESTING

With no existing industry accepted ambient storage test procedure, it is necessary to construct a test rig and execute a protocol developed during Phase 1 PAFI testing. This rig is comprised of a vented exterior drum which holds an internal aluminum test tank rig. In this aluminum tank, a collection of jars and glass rods are submersed in the test fuel for a total of 12 weeks with sampling occurring every three weeks. The ambient storage rig successfully designed and constructed for the FAA PAFI testing is shown schematically in Figure 4.

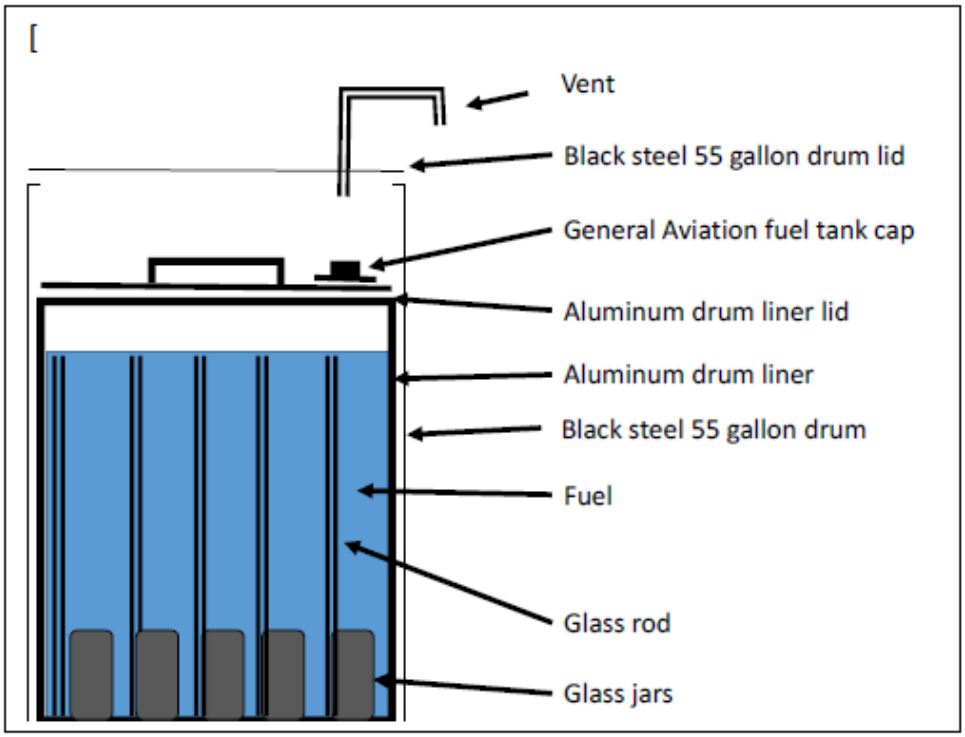


Figure 4 - Schematic of Ambient Storage Stability Test Rig (Courtesy of Purdue University)

12.2.1. EXTERIOR STORAGE DRUMS

The external rig containment is comprised of a 55-gallon black, steel drum fitted with a vent, see Figure 5. The drum is black to maximize solar heating. The drum is an open head drum with a fitted lid clamped into place with a split ring. It is recommended cutting the split ring 180° from the split. Add an additional bolt fixture so the ring can be released on both sides and opened as two halves. This facilitates opening the drum during routine inspections, see Figure 6.

Figure 5 - Exterior Storage Drum with Vent

Figure 6 - Cut to Split Ring and Additional Bolting Fixture

The drumhead shall be fitted with a vent. This vent may be made using aircraft tubing with a flared end and standard aircraft fittings. The vent shall have a bend so that the open end of the flare is pointing downward. This allows the test rig to "breathe" but prevents water such as rain from running directly into the rig.

A protective cover, such as a black trash bag, may be used to minimize water accumulation on top of the drumhead, making opening the drum easier to open during the routine inspections. The protective cover shall not be airtight and the vent shall protrude through the cover.

The external 55-gallon drum provides structure and protection to the aluminum test tank which will be placed inside of the exterior drum.

12.2.2. ALUMINUM OPEN HEAD DRUM WITH COVER

The aluminum fuel tank is represented with the use of an aluminum open head drum with a loose-fitting lid (Figure 7) which is fitted into the external 55-gallon steel drum.

The drum is constructed to fit into the 55-gallon steel drum. The aluminum drum shall be fitted with a supporting ring on the inside diameter on which the aluminum lid will rest. The aluminum lid shall be constructed with a means of lifting the lid, such as loop handles. The lid is to be lifted off the aluminum tank rig at each routine inspection.

The aluminum lid shall be fitted with a representative aircraft fuel filler cap which acts as a material compatibility test article.

Figure 7 - Interior Rig Test Tank with Lid

12.2.3. INNER STORAGE RACK

Throughout the testing, representative specimens of glass rods and jars of fuel will be collected and inspected. In order to support these specimens, an internal rack system capable of holding the minimum five glass rods and the minimum five wide mouth jars in a stable orientation and locations is required. An example is shown in Figure 8. In Figure 9, the loops at the top of the rack structure that will support the glass rods can be seen.

Figure 8 - Support Rack Interior to Test Rig Tank

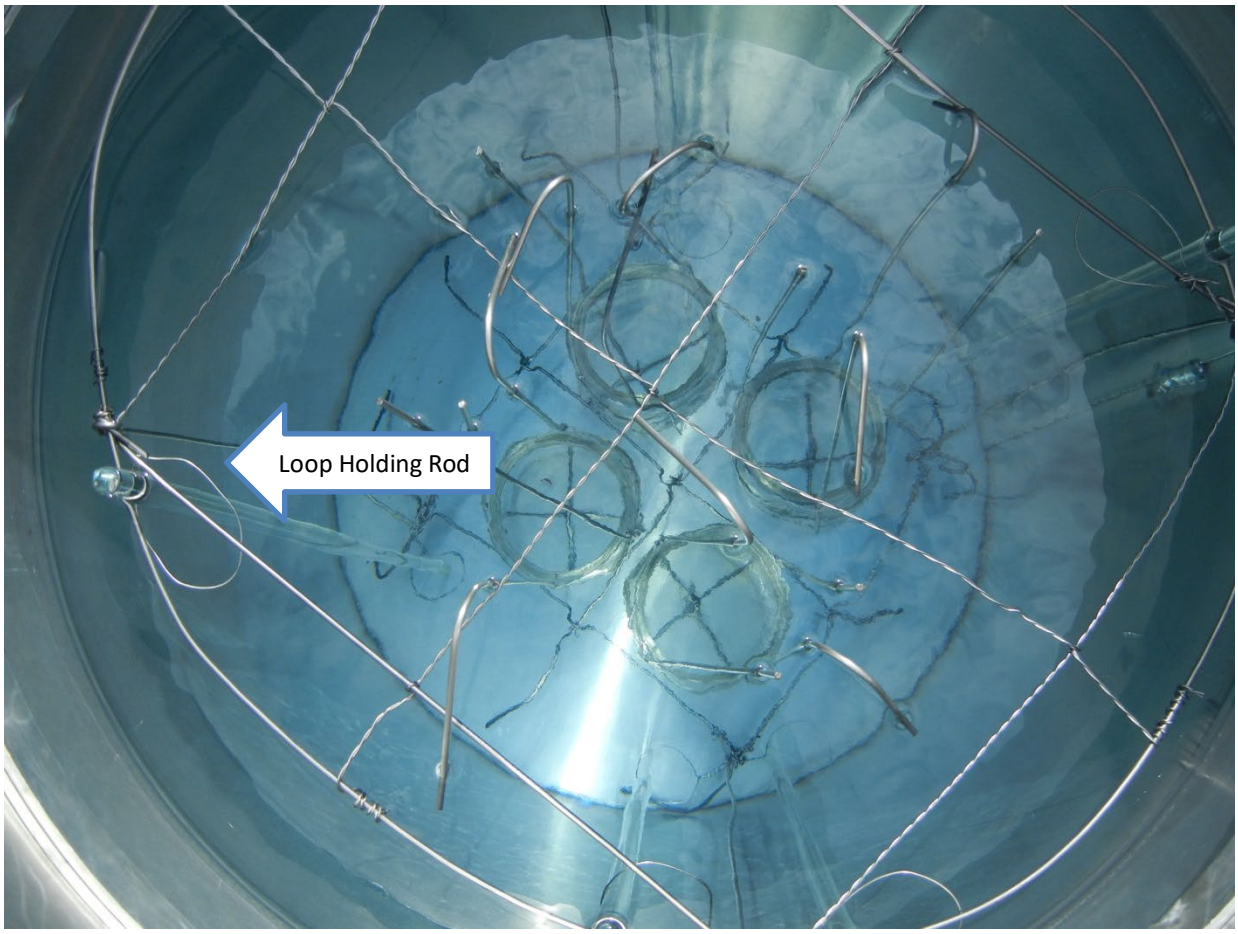


Figure 9 - Loops to Support Glass Rods

12.2.4. STORAGE CONTAINMENT FOR COLLECTED GLASS RODS AND DOWELS

At each routine sampling, one glass rod is removed and inspected. Following the inspection, the glass rod is to be collected and stored. One means of holding and protecting, as well as providing positive specimen identification of the glass rod is through the use of threaded PVC pipes with caps (Figure 10). Positively identifying the tubes with metal tags and bundling them into sample sets, i.e. all

five 100LL glass rod tubes for the entire exposure, has been successfully used to improve the ease of handling and storage of the tubes.

Similarly, a set of tubes for the wooden dowels used to track the fuel volume is recommended. The length of the tubes is determined by the length of the rods and dowels.

Figure 10 - PVC Threaded Pipes with Caps Bundled in Sample Sets

12.2.5. SPECIMEN JAR LIFTING RIG

In order to collect fuel bottoms, each installed specimen jar shall be lifted straight up and out of the test rig tank. This has been successfully accomplished by attaching a ring around the mouth of the jar and attaching stiff uprights which come above the surface of the fuel and can be grasped to lift the jar (Figure 11). The top of the lifting rig support wires are bent over to provide structure to lift given the likely slipperiness of the wires. These “hooks” may also be used to secure the jar via the lifting rig to the interior support structure. This will help prevent the jars being knocked over or upset due to inadvertent movement of the test rigs or due to movement caused during routine sample collection.

Figure 11 - Wide Mouth Jar Installed in Lifting Rig and Hooked to Interior Support Rack

12.2.6. DRUM STABILIZING STRUCTURE (FEET)

In order to provide positive stability for the entire rig, it is recommended a three-legged structure, into which the drum can be positively clamped in place, be constructed for each rig assembly. It is also recommended that this structure be positively attached to ground, for example bolted into the concrete. This will help prevent inadvertent movement of the drums during testing and will provide a point for grounding the drums. The structure may act as the ground point as long as there is a continuous path from the drums, to the feet, and to the grounding rod.

Figure 12 shows an example of the stabilizing feet sitting upright. The circle represents the location of the drum when installed in the structure. Figure 13 shows the drum rig installed into the stabilizing feet.

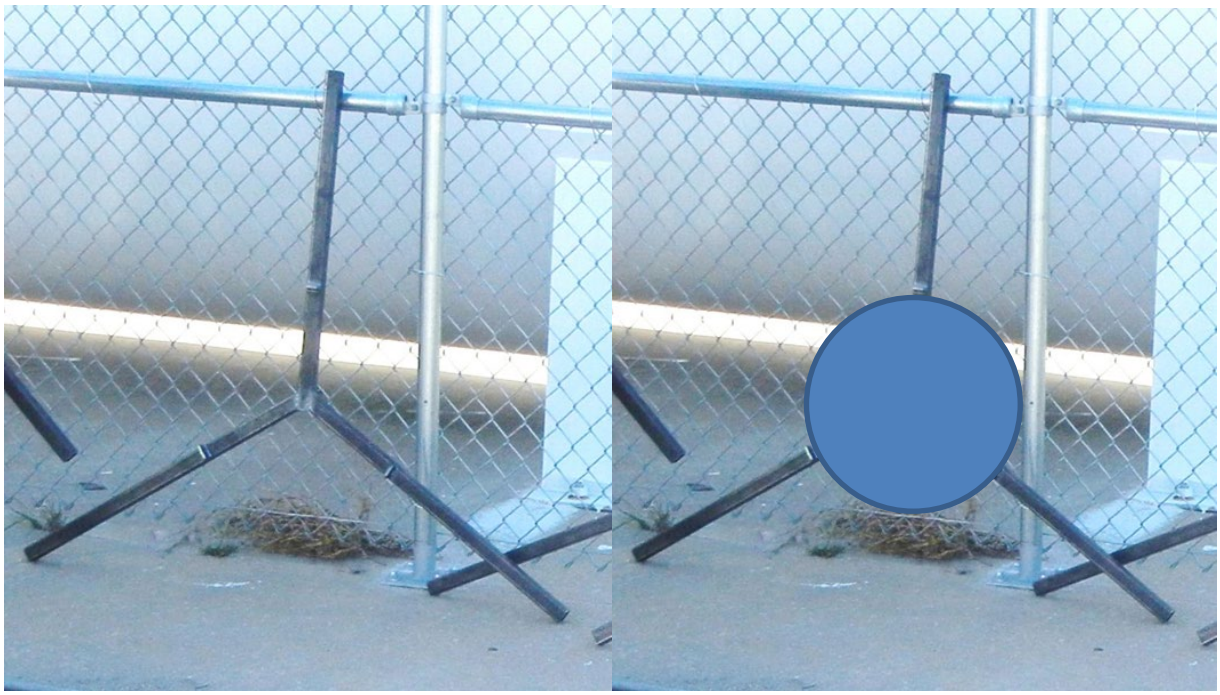


Figure 12 - Stabilizing Feet (Graphic Indicates Location of Drum within the Feet).

Figure 13 - Drum Installed into Stabilizing Feet

12.2.7. CONTROLLED ACCESS SPACE

In order to meet the objectives of the ambient testing, a location which receives sun for the majority of the day is required. The location shall provide positive control on access to the drums to prevent interruptions, unauthorized access, or movement of drums. This control shall be in place for the entire 12-week (3 month) period.

Space shall be sufficient to allow orientation of the drums such that the shadows from one drum onto another are minimized.

12.2.8. AMBIENT WEATHER MONITORING EQUIPMENT

A means of measuring and recording the daily ambient weather conditions is required. The data collected shall include: high and low air temperatures, high and low barometric pressure, wind speed and direction, high and low relative humidity, and amount of precipitation. The weather data shall be collected at the location of the testing. If the testing is performed at or immediately adjacent to an airport location, the daily NOAA data for that airport is acceptable.

12.3. ELEVATED (ACCELERATED) STORAGE TESTING

12.3.1. ELEVATED TEMPERATURE CONTROLLED AREAS – OVENS

The tests in this document require temperature-controlled environments capable of maintaining temperatures to ±3 °C.

Any elevated temperature spaces used for heating samples shall not have exposed heating elements or ignition sources. Considerations shall include ventilation of hazardous or flammable vapors. Temperature recorders are not required but a means of monitoring actual temperatures through a test is required.

Local fire safety regulations may limit the total volume of flammable liquids that may be stored in a space, especially at elevated temperatures or in personnel occupied spaces. Elevated testing requires approximately six gallons total volume of each fuel to be heated.

12.3.2. ASME STAINLESS STEEL PRESSURE VESSELS WITH FLUOROCARBON SEALS

Previous testing successfully used ASME wide-mouth, stainless steel pressure vessels fitted with special order fluorocarbon (Viton™) seals to perform the elevated temperature storage stability testing. A large volume pressure vessel is required to produce the required 5 gallons of aged fuel. To maintain a 5:4 fuel:air ratio required for the exposure and to produce the required 5 gallons requires a one-gallon and a five-gallon pressure vessel for each fuel. The pressure vessel may be fitted with multiple ports, but all ports must be capped.

Figure 14 - One Gallon ASME Pressure Vessel

12.3.3. 500mL (1 pint) glass Jars

In order to produce the aged specimens to be tested over the 12-week (3 month) elevated temperature storage stability testing, 500mL (1 pint) jars are required. The jars are filled with 400mL of sample. Previous testing successfully used wide mouth, pint canning jars. When filled with 400mL of sample, the resulting fuel:air ratio was calculated to be 5:4. Any glass jar with compatible lid may be used, however it may be necessary to recalculate the fuel:air ratio and duplicate it in the stainless steel pressure vessels used to prepare 5 gallons of aged fuel.

12.4. Karl Fischer Moisture Meter – Coulometric

A Karl Fischer titrator and concomitant chemicals are required to determine the moisture content of the samples. Testing is to be performed per ASTM D6304. Depending on the chemistry of the test fuel, reagents for aldehyde/ketone samples, or an additional oil dryer accessory, see D6304, Procedure C, for amine containing fuels may be required.

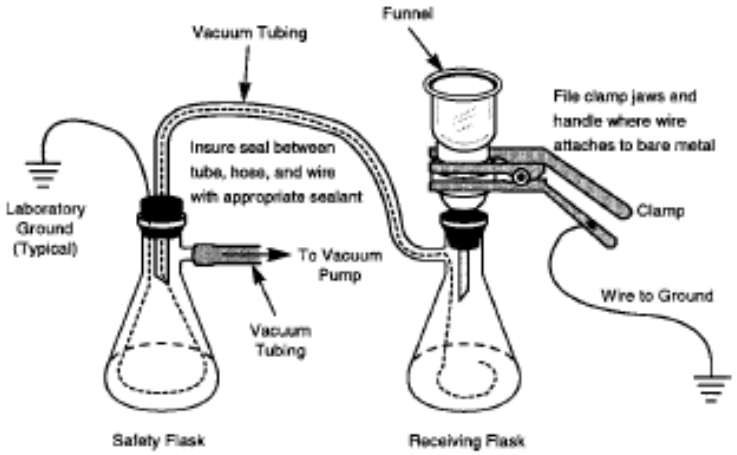
12.5. Calibrated / Validated Laboratory Measuring Equipment

Testing and measuring require standard laboratory equipment which has been calibrated or validated as appropriate. This list of equipment does not exclude others. Any equipment which has calibration shall have the date of the latest calibration recorded. For instruments using validation measurements, the results of the validation shall be recorded and the means of rectifying any non-compliant equipment documented.

Instrument	Report	Reference
Thermometer/thermocouples	Calibration	-
Electronic Balance	Calibration	
Weather station	Calibration	

12.6. ASTM D4625/D5452 Vacuum Filter Apparatus

In order to properly filter the fuel and solvents used in the ASTM D4625 Fuel Storage Stability testing, as well as determining filterable solids, and particulate contamination, a vacuum filter apparatus is required. This includes a filter funnel, a metal clamp, a filter support assembly, and a laboratory vacuum.



[2]**Figure 15 - ASTM D4625-14 Vacuum Filter Apparatus**

12.7. Other Specialty Equipment

Other support materials required include but are not limited to:

1) Sar-Gel Water Indicating Paste

2) Glass Stir Rods

3) Reagent grade laboratory chemicals filtered with 0.8 *μm* filter paper
   a. Isooctane
   b. Isopropanol
   c. Toluene
   d. Acetone

[2] Reprinted, with permission, from ASTM D4625-21, Standard Test Method for Middle Distillate Fuel Storage Stability at 43 °C (110 °F), copyright ASTM International. A copy of the complete standard may be obtained from www.astm.org.

e. Methanol

4) Nylon filter membranes; 0.8 *µm*

5) One-gallon stainless steel cans, a total of five for each test fuel

6) Five-gallon stainless steel cans, a total of one for each test fuel

13. TEST WITNESSING

Test witnessing is required for the collection and reporting of data collected in compliance with this test plan. This data is to be used by the FAA as part of their evaluation for fleet wide authorization. Fuel formulators shall submit a test plan for approval by the FAA before executing testing. Part of the submitted test plan must be a means of test witnessing by an FAA authorized representative.

Testing performed in accordance with an approved test plan shall be witnessed by a representative of the FAA AFPO or their designated representative. The extent of witnessing is at the discretion of the FAA; however, at a minimum the representative shall verify and witness the following segments.

1) Verify certificates of analysis are available for supporting samples as appropriate and for all test fuels
2) Verify instrument calibration/validation as appropriate
3) Inspect test set up (jars, specimen arrays, rigs, ovens, etc.
4) Verify pre-exposure data, record keeping and specimen control
5) Review test plan with test personnel
6) Witness test initiation
7) Witness fuel exchanges at the FAA's discretion and as appropriate
8) Witness test conclusion
9) Witness post exposure testing at the FAA's discretion and as appropriate
10) Verify testing in accordance with test plan

14. TEST PROCEDURE – AMBIENT STORAGE STABILITY

14.1. MATERIALS

1) ASTM D910 compliant aviation gasoline
2) Candidate fuel(s)
3) Sar-Gel Water Indicating Paste

14.2. RIG ASSEMBLY

The rig set up shall be at an open location which has been approved by the FAA AFPO. The following is a description based on previous FAA testing and provides an example. Modifications to the following are permitted with prior approval by the FAA AFPO.

The test area shall be prepared such that a controlled entry area is maintained where the storage stability rigs may be assembled and sit undisturbed except for sampling, the entire 12 weeks. The area shall be located to maintain uniformity in sun exposure. Consider shadows from other structures in the area as well as from the individual barrels. The area shall be large enough to facilitate setting up a work space sufficient to lay out the sampling supplies and the components of the rig. This space shall permit the sampling to occur without risk of cross contamination of components during the testing.

In most cases, the installation of the rigs will take long enough that it is recommended that the actual testing begin the next day to ensure all testing can be completed before the heat of the day. While this is not likely an issue during the setup, sampling at time zero, or at week three, it is likely to become an issue during week 9 and 12. It is desirous that all testing occur at roughly the same time of day for each sampling.

Figure 16 - Example of Controlled space for Ambient Storage Test

1) Using a stabilizing rig, such as that described in Section 11.2.6, install the rigs into the final position, confirming there is enough space around the drum to access the bolts on the split sealing ring once the barrels are installed. Bolt the stabilizing rig to the ground. This will facilitate maintaining the position of the drum and will prevent accidental upset of the drum (Figure 17).

Figure 17 - Example of Stabilizing Rig (Feet) Bolted to the Ground

2) Once the stabilizing feet are in place, install the 55-gallon steel drums into the stabilizing feet and clamp the drum in place.

Figure 18 - Drum Clamped into Place in Stabilizing Rig

3) Install a grounding post and connect the drums to the ground. A single grounding wire may be used to connect all the ambient storage test rigs. This grounding wire will also provide a connection point for personnel ground straps for use during testing.

4) If not already installed, for example for transport, set the aluminum test tank into the 55-gallon drum, and place the wire rack into the aluminum tank.

5) If not already installed, attach the external vent to the 55-gallon steel drum lid (Figure 19).

Figure 19 - External Vent Attached to the Exterior Drum Lid

6) Confirm each drum has a permanent sample identification label attached where it can be inspected during testing. It is permissible to use a more visible marking, such as an adhesive label, however the permanent tag will be the final arbitrator in cases of ambiguity, such as an adhesive label coming loose.

7) If testing is not to begin at this time, cover the drums with black plastic bags, such as large trash bags. Confirm the vent protrudes through the bag. The use of a weight to hold the bag in place is recommended. The use of the bags will facilitate the removal of any moisture that gathers on the top of the steel drum.

Figure 20 - Protective Cover in Place over Drum

14.3. TEST START

In the following discussions, only one test rig and concomitant testing equipment shall be in use at one time to prevent cross contamination. Finish all activities on one fuel, close the drum, and put all support equipment away before beginning a second fuel.

14.3.1. DRUM FILL

At the time of the test start, fill each labeled drum with the matching fuel sample. It is recommended that the fuel be transferred to the testing location in stainless steel drums fitted with dedicated valve equipment. For example, there shall be one set of fuel drum valve and vent for the 100LL aviation gasoline and one each for each test fuel. The valves and vents shall be positively identified and only used for the specific fuel sample.

1) Fill the test rig aluminum drum with 20 gallons of the fuel.
2) Place five glass rods into the wire rack holders. Rods shall be installed so they are upright/vertical.
3) Install the five-quart sample jars into their respective locations in the wire rack. The jars shall be sitting flat on the bottom of the aluminum test drum.

Figure 21 - Filled Test Rig with Jars and Glass Rods in Place

14.4. SPECIMEN AND DATA COLLECTION

The following specimen and data collection shall be followed for each sample time, including time zero through week 12. The procedure for Week 12 is slightly different and differences are found below in Section 14.6.

14.4.1. RESTRICTIONS AND RULES

1) A grounding strap (ESD wrist strap) is to be worn at all times when dealing with the fuels. Connect ESD strap to drum before starting the procedure.

2) Only one drum is to be open at any time.

3) All operations must take place during daylight hours

4) All operations must take place during calm weather conditions.

    a. No visible moisture (rain, fog, hail, etc.)
    b. No considerable wind
    c. Avoid exposure of the fuel, lids, and drums to all outside factors such as dirt and dust

5) Weather conditions will be recorded in the logbook.

    a. Temperature
    b. Relative humidity
    c. Barometric pressure
    d. Wind direction and speed

6) All samples must be taken within a maximum margin of one day from the scheduled sample days if at all possible.

    a. The only reason to not sample on the scheduled day is poor weather.

    b. If samples are taken on any day other than the scheduled day this must be specifically noted in the logbook.

    c. If inclement weather is forecast or occurs for greater than the margin of one day, notify the FAA so the deviation can be acknowledged.

7) Date and time of the sample is recorded in the logbook with technicians' legible signature(s).

8) Cover the working table and secure the table covering. Use a new covering after each sample.

9) All personnel shall wear a lab coat or other protective garment and wash the lab coats after each testing.

10) Use new nitrile gloves or dedicated PVA gloves for each drum and whenever the gloves are used.

    a. Change gloves when the gloves become soiled or torn even within a single sample.

11) Beginning in Week 6, every person working with fuels should wear a hat or bandana to absorb perspiration.

12) Application of sunscreen is recommended. Note the application shall occur outside of the testing sphere to avoid contamination by the sunscreen.

14.4.2. OPENING DRUM

1) Take a picture of the fuel farm.

2) Set up a working table. Cover the working table and secure the table covering.

3) Use new nitrile gloves for each drum and whenever the gloves have been soiled. This is to prevent cross-contamination of the fuels.

4) Take a picture of the drum.

5) Remove the plastic trash bag from the drum and discard.

    a. Take a picture of the drum uncovered

6) Match the drum number to the sample collection tubes and boxes.

7) Inspect the lid of the drum. If any water or debris is on the lid, clean the lid with disposable towels.

    a. Do not tip drum to remove water and debris from lid.
    b. Discard the used towels.
    c. Take a photo of the drum.

8) To unbolt the drum cover, carefully unbolt the 15/16” bolt and the 3/4" bolt. Remove the bolts from the sealing ring and place on the worktable.

9) Pull both halves of sealing ring away from each other to remove and set on the worktable.

10) Be sure there is nothing on the lid. Lift lid straight up and pull away without tipping to avoid dropping anything from on top of the lid into the fuel. Place the lid on the worktable upside down. Take care to avoid damaging the vent.

    a. Take a picture of the open drum

11) To remove the inner cover, lift with both handles. When lifting lid, do not lift straight up. Lift at an angle, with the alignment side lifting up first. Place the inner lid on the worktable with the handles down.

    a. Do not remove the fuel cap.
    b. Take a picture of the drum and lid.

12) Take multiple pictures of the fuel inside the drum showing the fuel level and condition.

14.4.3. PROCEDURE FOR WOODEN DOWEL

1) Prepare the water detection paste for application to the dowel.

2) The wooden dowels are recommended to be stored in the white PVC containers. Match the numbers on the PVC to the fuel ID tag. The longer PVC containers are for the wooden dowels.

3) Using the 'Week X' wooden dowel, mark the top of the dowel with the word 'Top'. The top of the dowel has a sticker and is marked with colored ink.

4) Place a thin layer of water detecting paste on the bottom three inches of the dowel.

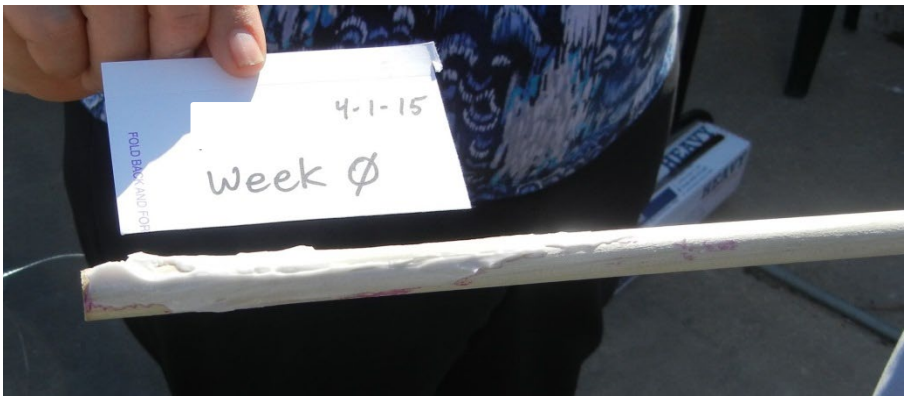


Figure 22 - Water Indicating Paste Applied to Dowel

5) Take a picture with an identification label of the wooden dowel and water paste before putting the dowel into the fuel.

6) Put the wooden dowel straight down into the fuel drum and ensure that it touches the bottom of the drum but does not enter a glass jar. Make sure that the dowel is vertical when placed inside the drum. Hold for 3-5 seconds on the bottom of the drum.

7) Carefully remove the dowel and place on the worktable.

8) Take a picture with an identification label of the wooden dowel and water paste immediately after putting the dowel into the fuel.

9) Make a notch on the dowel with a hacksaw blade or razor knife indicating the water level if any. It is indicated by the paste changing color.

    a. The paste will also continue to change color over time, especially in humid weather. Only the immediate color change at the bottom of the dowel is probative.

10) Make a notch on the dowel indicating the fuel level with a hacksaw blade or razor knife.

11) Take measurements, from the bottom of the dowel, to the top of any water level and to the fuel level. Record levels in the logbook.

12) Take pictures of dowel with tape measure in the picture.

    a. Make sure to take the measurements the same way every time. Typical tape measures are only accurate when the tape is pulled tight from the metal tab.

13) After the pictures, remove the paste using a clean towel.

14) Place the wooden dowel inside the white PVC tubing with the tag “Week X” (as matches the week in question).

15) Ensure that the tube number matches the drum number and week number.

    a. Place Kimwipes inside the tube to stabilize the dowel during shipment.
    b. Screw in the top, hand tighten, and then ¼ turn to tighten.

14.4.4. PROCEDURE FOR THE GLASS ROD

1) One glass rod will be removed at each sample period. The glass rods should be located inside the outer ring of the stainless-steel structure depending on construction.

2) Match the numbers on the PVC container to the fuel ID tag. Use the shorter PVC containers for the glass rods.

3) Remove the glass rod from the drum slowly so as not to disturb sediment on the glass rod or other materials in the drum.

4) Carefully place the glass rod on the worktable.

5) Take pictures of the glass rod and close-ups of any sediment, discolorations, or deposits. Record observations in the logbook.

    a. Do not reinsert the glass rod back into the test drum after it has been removed.

6) Place the glass rod inside the white PVC tubing with the “Week 0” tag (or as matches the week in question).

    a. Ensure that the tube number matches the drum number.
    b. Place Kimwipes inside the tube to stabilize the glass during shipment.
    c. Screw the top, hand tighten, and then ¼ turn to tighten.

14.4.5. PROCEDURE FOR FUEL SPECIMEN COLLECTION

1) Using a clean disposable pipette, extract 10 mL of fuel from a depth of 6 to 10 inches from the top of the fuel level in the drum. Put the collected fuel in 10 mL sample vial which matches the fuel ID and “Week 0” (or the week in question).

    a. Cap vial.
    b. Record the sample number of the vial in the record log.
    c. Place sample vial in zip locking bag and put in a container with secondary absorbent material. If shipping to the testing facility, use an appropriate shipping box.

2) To remove the jars for inspection, hold two of the stainless-steel supports and slowly raise the jar as to not disturb any sediment in the jar. By lifting slowly and straight up, the fuel and any sediment that were at the bottom of the drum and thus the bottom of the jar will remain at the bottom of the jar. It is critical the lifting be slow and completely level to avoid turbulent flow causing the fuel to “mix” or to “swirl” into jar and remove any sediment. This process should take up to 30 seconds.

3) Place the jar on the worktable and continue to hold the stainless-steel rods to support the jar. **Depending on the construction of the lifting rig, the jar will not stand unsupported**.

4) Photograph the jar including an identifying label. A second person may be required.

    a. Observe the jar for visible water, color, particulates, and cloudiness.
    b. Take multiple pictures of the fuel in the jar. The use of an ASTM haze chart or similar placed behind the jar can facilitate photography.
    c. Record all observations in the logbook.

5) At this point it is permissible to remove the wire lifting rig to facilitate pouring the fuel into a labeled one-gallon container. Confirm the fuel ID from the test rig matches the ID on the one-gallon container.

6) Examine empty jar for residue build up or solids in the jar that may not have been visible while filled.

    a. Take multiple pictures and record any observations in the logbook.

7) Take a dedicated dipping jar and dip one quart of fuel from the barrel. (First rinse)

   a. Submerge the jar six inches below the fluid level
   b. Remove the jar from the fuel.
   c. Pour off enough fuel from the jar back into the barrel, to reach the one quart, or 800 mL, level on the jar.
   d. Pour one pint, 400 mL, of the fuel from the dipping jar into the original sample jar collected above.
   e. Swirl the 400 mL of fuel in the sample jar to rinse any deposits from the sides of the jar and pour the 400 mL into the stainless-steel container.
   f. Pour the second pint, 400 mL, of the fuel from the dipping jar into the original sample jar.
   g. Swirl the second 400 mL of fuel in the sample jar to rinse any deposits from the sides of the jar and pour the second 400 mL into the stainless-steel container.

8) Take the dedicated dipping jar and dip one quart of fuel from the barrel. (second rinse), repeating a – g above.

9) Take a dipping jar and dip one quart of fuel from the barrel. (third rinse), repeating a – g above.

10) Verify there is one gallon of fuel in the one-gallon container. If there is not one gallon, dip additional fuel to achieve a total of one gallon.

11) Cap the one-gallon container and verify the specimen identification label.

12) Cover the original glass sample jar using a sealing lid. The lid should be snug or hand tight. Do not overtighten, especially plastic lids.

13) Attach an identification tag to the jar. Collect all jars until the end of the test.

14.5. PREPARING FOR CONTINUED EXPOSURE

14.5.1. MARK FUEL LEVEL

1) Take the wooden dowel from the container marked for the next testing day (3 weeks hence).

   a. Insert the wooden dowel into the barrel until the end of the wooden dowel touches the bottom of the barrel. Avoid inserting the dowel into the glass jars.
   b. Withdraw the wooden dowel and notch the dowel with hacksaw blade or razor knife.
   c. Measure the depth of the fuel and note fuel level in record log.

d. Photograph dowel with date and fuel level on written card in photograph.
e. Reinsert wooden dowel into its original container.

14.5.2. CLOSE DRUMS

Each fuel is covered by two lids. The inner lid fits loosely within the aluminum barrel and does not provide a water-proof seal. The outer lid fits over the steel barrel and is secured with two pieces of a barrel ring held together by bolts on opposite sides. A rubber gasket is held between the steel barrel rim and the underside of the outer lid forming an air-tight seal. Over time, swelling of the rubber gasket may make installing the lid and closing the split ring difficult. If swelling becomes too great, the gasket size (circumference) may be reduced by cutting out a section. Use a scarf cut (at an angle) so the two ends of the gasket may be overlapped, maintaining an airtight seal.

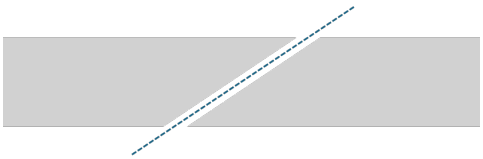


Figure 23 - Example of a scarf cut

1) To close the aluminum drum after samples have been taken, inspect the inner lid to ensure it has no contaminants on it. Then re-cover the inner drum by placing the lid down at an angle once again, with the same side being placed down first.

2) To seal the outer drum, place the lid on the drum and place both rings over the lip of the lid and drum.

3) Position the two sealing ring halves so that the space between the two ends meant for the ¾" bolt are separated by 1/2" (Figure 24).

Figure 24 - Gap on the Split Ring

4) Screw in the ¾" bolt first, retaining that 1/2" space between the two ring halves. Continue to screw until the bolt is just past flush with the nut.

5) Screw in the 15/16" bolt.

    a. Once the larger bolt is connected to both halves you can continue to screw in both bolts till the lid is secured down and ensures that it is waterproof.

6) Cover the drum with a new plastic trash bag, making sure the vent is protruding out of the cover. Place a weight on the bag.

7) Ensure area is secure when leaving.

14.6. WEEK 12

14.6.1. DOWEL AND GLASS ROD

1) Repeat the same procedure for the wooden dowel and the glass rod as described above.

14.6.2. FUEL SPECIMEN COLLECTION

1) Using a clean disposable pipette, extract 10 mL of fuel from a depth of 6 to 10 inches from the top of the fuel level in the drum as described above. Put the collected fuel in 10 mL sample vial which matches fuel ID and week 12.

2) Remove the jar for inspection, as described above.

3) Place the jar on the worktable and continue to hold the stainless-steel rods to support the jar.

4) Photograph as before.

5) At this point it is permissible to remove the wire lifting rig to facilitate pouring the fuel into a labeled gallon container. Confirm the fuel ID from the test rig matches the ID on the gallon container.

6) Examine empty jar for residue build up or solids in the jar that may not have been visible while filled.

7) Repeat the process for filling and rinsing the 12-week sample jar as described in 14.4.5, Items 7 – 9.

8) Remove all the wire frames from the inside of the barrel and place them in a plastic trash bag, and tie wrap the bag closed.

9) Stir the fuel remaining in the aluminum tank using a clean wooden stick to stir up any sediment in the bottom of the barrel. Agitate the remaining fluid in the barrel for two minutes.

10) Using the dipping jar appropriate for this barrel submerge the jar 6 to 10 inches below the surface and collect a full jar of fuel (between 800 mL and a full jar).

11) Pour the collected fuel into a stainless steel **five** gallon can.

12) Collect a total of one gallon of fuel

13) Re-stir the fuel in the barrel for 30 seconds to one minute and repeat to collect a second gallon.

14) Repeat 14.4.5, items 7 – 9 above until the five gallon can has been filled with a quantity of five gallons.

15) Record this collection in the record log

16) Install the cap on the five-gallon stainless-steel can.

17) By either dipping or pouring or a combination of both (the barrel may be removed from its stabilizing frame at this point), pour any remaining fuel from the test barrels into black, epoxy lined PLAIN STEEL cans, and close these cans.

18) Cover the original sample glass jar using a sealing lid. The lid should be snug or hand tight. Do not overtighten, especially plastic lids.

19) Attach an identification tag to the jar.

20) Collect all sample jars and hold for 60 days for any follow-up questions.

14.7. POST EXPOSURE MEASUREMENTS

Depending on the test facility, all or a part of the fuel tests may be performed at the test facility or may be performed by a contract laboratory facility. Adjustments to the following shall be documented and submitted to the FAA AFPO as part of the submitted test protocol. All standard industry tests are performed in compliance with the referenced standard as written unless otherwise indicated.

14.7.1. EVAPORATIVE LOSSES

No further measurements are required after the data collection over the twelve weeks. Proceed to calculation to determine the evaporative losses over the 12-week exposure. See Appendix B for an example data chart.

14.7.2. DEPOSIT FORMATION

Deposits are detected visually by evaluating the appearance of the fuel inside of the barrel as well as the glass rod and the sample jar which are removed during each test. Photographs document the overall appearance of the fuels in the barrels, the glass rods and the sample jars. Deposits can form through processes

that are biological (e.g., microbial growth) or chemical (e.g., precipitation). No further measurements are required after the data collection over the twelve weeks.

14.7.3. MOISTURE CONTENT BY KARL FISCHER – D6304

Using the vial sample of fuel, measure the ambient water content of the test fuels by Karl Fischer Coulometric per ASTM D6304 and record the values. Because of the temperature dependence of water content, it is desirable to have the vial come to the same temperature as it was when collected. Do not open the vial until ready to test the moisture content.

The following tests are to be run using the one-gallon sample collected every three weeks, including week 12.

14.7.4. COPPER STRIP CORROSION – D130

Determine the copper strip corrosion rating by ASTM D130 for the 100LL and the unleaded test fuel at each sample point. Compare the change in the corrosion rating over time for the 100LL, to the change in corrosion rating over time for the unleaded test fuel.

14.7.5. COLOR - D156 AND D1500

Determine the color of the test fuels by Saybolt (D156) and ASTM Color (D1500) for the 100LL and the unleaded test fuel per company test method at each sample point. Compare the change in color over time in the 100LL, to the change in color over time in the unleaded test fuel.

14.7.6. EXISTENT GUM CONTENT – D381

Determine the existent gum content by ASTM D381 air-jet method for aviation gasoline for the 100LL and the unleaded test fuel at each sample point. Compare the change in existent gum content over time in the 100LL, to the change in existent gum content over time in the unleaded test fuel.

14.7.7. TOTAL ACID NUMBER – D664

Determine the acid number in mg KOH/g by ASTM D664 for the 100LL and the unleaded test fuel at each sample point. Compare the change in acid number over time in the 100LL, to the change in acid number over time in the unleaded test fuel.

14.7.8. WATER REACTION TEST – D1094

Determine the water reaction by ASTM D1094 for the 100LL and the unleaded test fuel at each sample point. Compare the change in rating over time in the 100LL, to the change in rating over time in the unleaded test fuel.

14.7.9. MOTOR OCTANE NUMBER – D2700

Determine the motor octane number by ASTM D2700 for the 100LL and the unleaded test fuel at each sample point. Compare the change in motor octane number over time in the 100LL, to the change in motor octane number over time in the unleaded test fuel.

14.7.10. DENSITY – D4052

Determine the density kg/m^3 by ASTM D4052 densitometer for the 100LL and the unleaded test fuel at each sample point. Compare the change in density over time in the 100LL, to the change in density over time in the unleaded test fuel.

14.7.11. FREEZE POINT – D2386

Determine the freeze point in °C by ASTM D2386 for the 100LL and the unleaded test fuel at each sample point. Compare the change in freeze point over time in the 100LL, to the change in freeze point over time in the unleaded test fuel.

Note that aviation gasoline may have a freeze point below the lowest temperature the test apparatus is capable of reaching. In this case, report the value to be < -50 °C or the lowest temperature achievable.

14.7.12. ELECTRICAL CONDUCTIVITY – D2624

Determine the electrical conductivity value at the selected measurement temperature (record the temperature) in pS/M. Values are specific to the temperature at which they are measured and cannot be directly converted to a different temperature. Because of this limitation, it is recommended that all tests be run at the same temperature to facilitate observing changes in the electrical conductivity of the fuels over time. If testing is contracted, the test house may have a preferred temperature. Identify this temperature and assure this test temperature is used on every sample. It is also recommended the same test house be contracted for the entire program.

Compare the change in conductivity over time in the 100LL, to the change in conductivity over time in the unleaded test fuel.

14.7.13. HYDROGEN PEROXIDE CONTENT – D3703

Determine the hydroperoxide number by ASTM D3703 for the 100LL and the unleaded test fuel at each sample point. Compare the change in hydroperoxide number over time of the 100LL, to the change in hydroperoxide number over time of the unleaded test fuel.

14.7.14. NET HEAT OF COMBUSTION – D4809

Determine the net heat of combustion in MJ/kg (mass) by ASTM D4809 for the 100LL and the unleaded test fuel at each sample point. Compare the change in heat of combustion over time of the 100LL, to the change in heat of combustion over time of the unleaded test fuel. To run net heat of combustion, the mass percent of hydrogen in the sample is required.

14.7.15. VAPOR PRESSURE – D5191

Determine the vapor pressure at 38 °C in kPa by ASTM D5191 for the 100LL and the unleaded test fuel at each sample point. Compare the change in vapor pressure over time in the 100LL, to the change in vapor pressure over time in the unleaded test fuel.

14.7.16. PARTICLE CONTAMINATION – D5452

Determine the particle contamination in mg/L by ASTM D5452 for the 100LL and the unleaded test fuel at each sample point. This test method requires the same petroleum filtration rig as ASTM D4625. Compare the change in contamination over time in the 100LL, to the change in contamination over time in the unleaded test fuel.

14.7.17. HYDROGEN (D7171) AND SULFUR (D2622) CONTENT

Determine the hydrogen content in mass % by ASTM D7171 NMR and the sulfur content in mass % by ASTM D2622 XRF, for the 100LL and the unleaded test fuel at each sample point. Compare the change in contents over time for the 100LL to the change in contents over time in the unleaded test fuel. The hydrogen content is also required to determine the net heat of combustion.

At the end of the 12-week exposure test, all of the above tests shall be run on the week 12 fuel collected in the one-gallon container. In addition to the above tests, the samples shall also be analyzed for Distillation, Research Octane number and composition by GC-MS, and shall have a complete Certificate of Analysis repeat analysis performed using the five-gallon sample.

14.7.18. DISTILLATION – D86

Determine the distillation curve for the test fuels using, ASTM D86 for the 100LL and the unleaded test fuel at the end of the exposure test (Week 12).

Compare the change in distillation curve from the original CoA in the 100LL, to the change in distillation curve from the original CoA in the unleaded test fuel.

14.7.19. RESEARCH OCTANE NUMBER – D2699

Determine the Research Octane Number (RON) by ASTM D2699 Procedure A or C for the 100LL and the unleaded test fuel at the end of the exposure test (week 12). Compare the change in RON from time zero to week 12 for the 100LL, to the change in RON for the unleaded test fuel.

14.7.20. GAS CHROMATOGRAPH AND MASS-SPECTROSCOPY (GC-MS)

GC-MS is one means of determining compositional changes, for example ASTM D6733 Standard Test Method for Determination of Individual Components in Spark Ignition Engine Fuels by 50-Metre Capillary High Resolution Gas Chromatography and transferring the effluent directly into a mass spectrometer. It is also

acceptable to run GC X GC methods, i.e. UOP990 Organic Analysis of Distillate by Comprehensive Two-Dimensional Gas Chromatography with Flame Ionization Detection. No matter what method is used, the researcher shall fully document all of the testing parameters used and shall use the same testing parameters as appropriate for all specimens. The GC-MS testing is only required on the final 12 sample and may be taken from the vial, the one gallon can or the five-gallon can.

Past testing successfully utilized traditional GC/MS analyses. The following is one example of analyzing specimens for compositional differences.

1) Set up the two instruments per specific instrument guidance or the Table 1 Operating Conditions from ASTM D6733

2) Gently agitate the vial specimen. No other pre-injection separations or filtration are necessary.

3) Inject into the GC-MS with appropriate syringe and using a petroleum compatible column. A non-polar GC column is recommended. One type successfully used is a 50 meter Restek RTX-DHA, 0.2 mm inner diameter column optimized for hydrocarbon analysis with a film thickness of 0.5µm.

4) It is recommended that a column evaluation be performed before running the samples. Follow the evaluation in ASTM D6733. A system performance mixture of equal amounts by mass comprised of n-pentane, n-heptane, n-octane, n-dodecane, 2-methylheptane, 4-methylheptane, and toluene that has been diluted to a concentration of 2% by mass in n-hexane is available commercially.

    a. Perform an evaluation for efficiency, resolution, and polarity of the column using the following parameters.

Initial temperature	35 °C
Hold time	50 min
Final temperature	220 °C
Hold time	20 min
Rate	3 °C/min

5) Collect GC-MS data.

6) Repeat analysis for each of the three specimens in each sample set.

7) Compare the resulting chromatographs and concomitant mass spectra to each other and to the ambient baseline spectrum. Note any peak shifts, area changes or other differences in composition between the data. Because D6733 is for GC/FID concentration calculation, run the GC/MS/FID for the specimen and collect data from both the MS and FID detectors and combine them.

Using the software provided by the GC manufacturer that uses the MS data to identify the components, the response factors provided in D6733 which are optimized for the FID detector and the method conditions described above, identify the components, and find the individual concentrations.

14.8. CALCULATIONS

14.8.1. EVAPORATION

Fuel levels are measured at the beginning and conclusion of each test for each fuel. Comparing the final fuel volume of a fuel during a previous test to the initial fuel volume during the next test shows the amount of fuel that evaporated during a three-week period. The depth of fuel in the barrel is measured using the dowel and marking the fuel level with a hacksaw blade or razor knife. The distance from the bottom of the dowel to the mark is then measured to the nearest 1/8 inch. The diameter of the aluminum barrels is measured and recorded. Assuming a tank diameter of 17.5" for this example, the volume of fuel in the barrels is 1.04 gallons per inch of depth.

$$V = \pi r^2 h$$

Where:

V = volume of a cylinder
r = radius of the cylinder
h = height of the cylinder

$V = 3.14 \times (17.5/2)^2 \times 1 = 240.53$ in^3 or 1.04 gallons per inch of depth. To determine the volume loss, subtract the new height of fuel from the starting height of fuel. Multiply the height lost by 1.04 gallons per inch to determine how much fuel evaporated during the three-week period.

With a depth error of ± 1/8 inch, the error in volume is ± 0.13 gallons.

Generate a table showing the initial and final fuel volumes based on depth measurements and determine the evaporative losses for the 100LL and the unleaded test fuel. An example table is shown in Appendix B.

14.8.2. DEPOSITION

No calculations required for deposition. Report the observations in a tabular form, noting specifically any deposition or sediment formation. Compare the results between the fuels.

14.8.3. KARL FISCHER – D6304

No calculations are required.

Report the observations in a tabular form, noting specifically any changes in water content and if there is any trend to the change. Compare the results between the

fuels. It is possible for there to be changes observed that do not trend. These observations should be specifically noted.

14.8.4. DISTILLATION – D86

When performing ASTM D86, distillation, a number of calculations are required to determine the percentages at each point and the percent recovered. See ASTM D86, §12. The data may then be processed arithmetically or graphically.

Data may be reported either in tabular form or graphically, noting specifically any changes in the distillation curve over time. Note any significant shifts in recovery point temperatures.

Due to differences in the chemical compositions of the fuels, distillation curves may be different between the 100LL and the unleaded test fuel. Comparisons between the fuels shall be related to observable changes in the distillation curves over time.

14.8.5. COPPER STRIP CORROSION – D130

No calculations are required. A visual interpretation of the sample condition is required.

Report the observations in a tabular form, noting specifically any changes in rating and if there is any trend to the change. Compare the results between the fuels. It is possible for there to be changes observed that do not trend. These observations should be specifically noted.

14.8.6. VISCOSITY – D445

When performing ASTM D445 Kinematic Viscosity, the viscosity is calculated according to the following equation, see D445, §14.1.

$$\nu_{1,2} = C\, t_{1,2}$$

Where:

$\nu_{1,2}$ = determined kinematic viscosity values for v_1 and v_2, respectively, mm^2/s
C = calibration constant of the viscometer, mm^2/s^2
$t_{1,2}$ = measured flow times for t_1 and t_2, respectively, seconds

After the data is collected for all twelve weeks, perform a regression analysis to determine whether there is a trending change in viscosity over time. Report the data in tabular form. Compare the trends between the 100LL and the unleaded test fuel. It is possible for there to be changes observed that do not trend. These observations should be specifically noted.

14.8.7. COLOR – D156 AND D1500

No calculations are required to determine the Saybolt color. No calculations are required to determine the ASTM color.

Report the observations in a tabular form, noting specifically any changes in color and if there is any trend to the change. Compare the results between the fuels. It is possible for there to be changes observed that do not trend. These observations should be specifically noted.

14.8.8. EXISTENT GUM CONTENT – D381

When performing ASTM D381 Existent Gum Content for aviation fuels, not turbine, the existent gum is calculated according to the following equation, see D381, §12.1.

$$A = 2000\ (B - D + X - Y)$$

Where:

A = existent gum content, mg/100 mL
B = mass recorded for sample beaker and residue
D = mass for empty sample beaker
X = mass recorded for empty tare beaker
Y = mass recorded for tare beaker after exposure

After the data is collected for all twelve weeks, perform a regression analysis to determine whether there is a trending change in gum content over time. Compare the trends between the 100LL and the unleaded test fuel. Note that due to differences in additives, there may be a difference in the original gum content of the fuels. It is possible for there to be changes observed that do not trend. These observations should be specifically noted.

14.8.9. TOTAL ACID NUMBER – D664

When performing ASTM D664 Total Acid Number, the acid number is calculated according to the following equation, see D664, §19.2.

$$Acid\ number, mg\frac{KOH}{g} = (A - B)x\ M\ x\ 56.1/W$$

Where:

A = Volume of alcoholic KOH solution used to titrate sample to last inflection end point, mL
B = Volume corresponding to A for blank titration, mL
M = Concentration of alcoholic KOH solution, mol/L
W = sample, mass, g

After the data is collected for all twelve weeks, perform a regression analysis to determine whether there is a trending change in total acid over time. Report the data in tabular form. Compare the trends between the 100LL and the unleaded test fuel. Note that due to differences in additives, there may be a difference in the original total acid number of the fuels. It is possible for there to be changes observed that do not trend. These observations should be specifically noted.

14.8.10. WATER REACTION TEST - D1094

No further calculations are required. Results are a rating based on a visual inspection.

Report the observations in a tabular form, noting specifically any changes in rating and if there is any trend to the change. Compare the results between the fuels. It is possible for there to be changes observed that do not trend. These observations should be specifically noted.

14.8.11. MOTOR OCTANE NUMBER – D2700

When performing ASTM D2700 Motor Octane Number, the MON is calculated according to the following equation, see D2700, §18.2.

$$O.N._S = O.N._{LRF} + \left(\frac{K.I._{LRF} - K.I._S}{K.I._{LRF} - K.I._{HRF}} \right) (O.N._{HRF} - O.N._{LRF}) \quad (4)$$

where:

$O.N._S$ = octane number of the sample fuel,
$O.N._{LRF}$ = octane number of the low PRF,
$O.N._{HRF}$ = octane number of the high PRF,
$K.I._S$ = knock intensity (knockmeter reading) of the sample fuel,
$K.I._{LRF}$ = knock intensity of the low PRF, and
$K.I._{HRF}$ = knock intensity of the high PRF.

After the data is collected for all twelve weeks, perform a regression analysis to determine whether there is a trending change in MON over time. Compare any trends between the 100LL and the unleaded test fuel. It is possible for there to be changes observed that do not trend. These observations should be specifically noted.

14.8.12. DENSITY – D4052

The recorded or printed value from most density machines is the final result. No further calculations are required.

After the data is collected for all twelve weeks, perform a regression analysis to determine whether there is a trending change in density over time. Compare any trends between the 100LL and the unleaded test fuel. It is possible for there to be changes observed that do not trend. These observations should be specifically noted.

14.8.13. FREEZE POINT – D2386

With the exception of applying any relevant thermometer corrections, no calculations are required.

After the data is collected for all twelve weeks, perform a regression analysis to determine whether there is a trending change in freeze point over time. Compare any trends between the 100LL and the unleaded test fuel. It is possible for there to be changes observed that do not trend. These observations should be specifically noted.

14.8.14. ELECTRICAL CONDUCTIVITY – D2624

No calculations are required.

After the data is collected for all twelve weeks, perform a regression analysis to determine whether there is a trending change in density over time. Compare any trends between the 100LL and the unleaded test fuel. It is possible for there to be changes observed that do not trend. These observations should be specifically noted.

14.8.15. HYDROGEN PEROXIDE CONTENT – D3703

When performing ASTM D3703, the peroxide number is calculated according to the following equation, see D3708, §11.1.

$$Hydroperoxide\ number, X({}^{mg}/_{kg}) = \frac{[(A-B)N \text{ x } 1000 \text{ x } 8]}{S}$$

Where:

X = measured hydroperoxide number
A = milliliters of $Na_2S_2O_3$ solution required for titration of the sample
B = milliliters of $Na_2S_2O_3$ solution required for titration of the blank
N = normality of the $Na_2S_2O_3$ solution
8 = milliequivalent of hydroperoxide number
S = grams of sample used, accurate to 0.001 g

After the data is collected for all twelve weeks, perform a regression analysis to determine whether there is a trending change in hydrogen peroxide content over time. Compare any trends between the 100LL and the unleaded test fuel. It is possible for there to be changes observed that do not trend. These observations should be specifically noted.

14.8.16. NET HEAT OF COMBUSTION – D4809

When performing ASTM D4809, the net heat of combustion is calculated according to the following equation, see D4809, §3.1.2.

$$Q_n(net, 25\,°C) = Q_g(gross, 25\,°C) - 0.2122 \times H$$

Where:

Q_n(net, 25 °C) = net heat of combustion at constant pressure, MJ/kg
Q_g(gross, 25 °C) = gross heat of combustion at constant volume, MJ/kg
H = mass % of hydrogen in the sample

After the data is collected for all twelve weeks, perform a regression analysis to determine whether there is a trending change in net heat over time. Compare any trends between the 100LL and the unleaded test fuel. It is possible for there to be changes observed that do not trend. These observations should be specifically noted.

14.8.17. VAPOR PRESSURE – D5191

When performing ASTM D5191, the Dry Vapor Pressure Equivalent (DVPE) is calculated according to the following equation, see D5191, §14.2.

$$DVPE, kPa\ (psi) = (0.965\ X) - A$$

Where:

X = measured total vapor pressure in kPa (psi)
A = 3.78 kPa or 0.548 psi

After the data is collected for all twelve weeks, perform a regression analysis to determine whether there is a trending change in vapor pressure over time. Compare any trends between the 100LL and the unleaded test fuel. It is possible for there to be changes observed that do not trend. These observations should be specifically noted

14.8.18. PARTICLE CONTAMINATION – D5452

When performing ASTM D5452, the particle contamination is calculated according to the following equation, see D5452 §12.3.

$$Total\ contaminant\ \left({}^{mg}/_{L}\right) = \frac{(W_2 - W_1) - (W_4 - W_3}{(L)}$$

Where:

W_1 = initial test membrane mass (mg)
W_2 = final test membrane mass (mg)
W_3 = initial control membrane mass (mg)
W_4 = final control membrane mass (mg)
L = the volume filtered reported in liters

After the data is collected for all twelve weeks, perform a regression analysis to determine whether there is a trending change in particle contamination over time. Compare any trends between the 100LL and the unleaded test fuel. It is possible for there to be changes observed that do not trend. These observations should be specifically noted.

14.8.19. HYDROGEN (D7171) AND SULFUR (D2622) CONTENT

When performing hydrogen content by D7171 NMR, and sulfur content by XRF, the test instruments have software which reports the data in mass %. Note: the hydrogen content is also required to determine the net heat of combustion.

After the data is collected for all twelve weeks, perform a regression analysis to determine whether there is a trending change in element content over time. Compare any trends between the 100LL and the unleaded test fuel. It is possible for there to be changes observed that do not trend. These observations should be specifically noted.

14.8.20. GAS CHROMATOGRAPHY

When using the ASTM D6733 method, the calculations of % (m/m) of each compound without co-elution and not corrected for co-elutions are calculated according to the following equation, see D6733 §13.1.

$$C_i = \frac{A_i B_i}{\sum_{i=0}^{i=n} \sum (A_i B_i + A_{int} B_{int}} \ 100$$

Where:

C_i = % (m/m) of compound i without co-elution and no correction of coelutions,
A_i = peak area of compound i without co-elution (benzene, toluene, and oxygenates)

A_{int} = peak area of compounds col-eluting (benzene, toluene, and oxygenates)
B_i = response factor for components
B_{int} = response factor for components co-eluted with benzene, toluene, and oxygenates

This analysis may be performed using instrument software.

Prepare a report which correlates any visual observations with the appearance of any shifts in the chemical composition of the fuel samples with respect to sample depth shall be noted.

While NDA's may prohibit specific attempts to determine the chemical composition of a candidate fuel, reasonable identification of components whose ratio varies between layers is appropriate.

15. TEST PROCEDURE – ELEVATED STORAGE STABILITY

15.1. MATERIALS

1) ASTM D910 compliant aviation gasoline.

2) Candidate fuel(s)

    a. Fuel for 400 mL testing shall be filtered through 0.8-micron membranes prior to testing
    b. Fuel for five-gallon development is not filtered

15.2. RIG ASSEMBLY

15.2.1. PREPARATION OF PRESSURE VESSELS FOR FIVE-GALLON SAMPLE

1) Wash all pressure vessels to be used in executing the exposure of the 12 week five-gallon sample. Wash the equipment using an alkaline washing procedure such as described in ASTM D4625. A summary of the process is provided below.

    a. Soak the clean vessels, including the lids and seals, overnight in an alkaline detergent. Only touch the equipment after this point with laboratory forceps and clean gloves.
    b. After the overnight soak, rinse the pressure vessel parts with distilled water for a minimum of three rinses.
    c. Allow the parts to dry thoroughly, preferably in a laboratory oven at room temperature.
    d. Reassemble the pressure vessel and close vessel.

2) Wash all sample jars using an alkaline washing procedure such as described in ASTM D4625.

15.3. TEST START

When ready to begin, fill the sample containers to their prescribed fill levels which will result in a consistent fuel:air ratio between the smaller 500 mL containers and the larger pressure vessels. The following instructions are based on the use of 500 mL, pint glass jars and a pair of one- and five-gallon ASME pressure vessels giving a ratio of 5:4. While the ASTM test method uses sample containers fitted with breather tubes, the high volatility of the aviation gasoline precludes its use. Instead, to provide continued oxygen content to the samples, the samples will be open and aerated throughout the test.

15.3.1. 400 mL SAMPLES

1) Fill each of six, labeled one-pint jars with 400 mL of filtered fuel.

2) Place a double layer of foil over the mouth of the jar and smooth it over the rim. Tighten the lid in place.

    a. Note: if using laboratory jars with Teflon™ lined lids, do not use foil. Tighten sufficiently to seal the jar but avoid overtightening which may crack the lids.

3) Place sample jars into appropriate safety containment which can contain any fuel spillage, and which can be appropriately vented from the oven (See Figure 25).

Figure 25 - 400 mL Samples in Vented Safety Containment

4) Place the samples into a heated space / oven set to 43 °C (110 °F). This space shall be the same space used for the five-gallon samples if possible.

15.3.2. PRESSURE VESSEL – FIVE GALLON SAMPLES

In order to prepare aged five-gallon samples with the same fuel:air ratio as experienced by the 400 mL samples, it is necessary to use a pair of a one-gallon and a five-gallon pressure vessel. The fill volumes below are specific to these vessels. Recalculate if alternative vessels are used.

1) All vents on the pressure vessels shall be capped / plugged.

2) Using clean, alkaline washed glassware, fill the labeled one-gallon pressure vessel with 4,240 mL of the fuel to be tested.

3) Using clean, alkaline washed glassware, fill the labeled five-gallon pressure vessel with 16,000 mL (16 L) of the fuel to be tested.

Figure 26 - Filling One and Five-Gallon Pressure Vessels

4) Seal the pressure vessel, making sure the specially purchased Viton™ seal is appropriately positioned.

5) Place the samples into a heated space / oven set to 43 °C (110 °F). This space shall be the same space as the 400 mL samples if possible.

Figure 27 - Example Pressure Vessels in Heated Space (showing five fuels)

15.4. SPECIMEN AND DATA COLLECTION

At each sample point (Week 3, 6, 9, and 12) samples will be collected. In addition, at Week 3, 6, and 9 all samples will be opened and aerated.

15.4.1. 400 mL SAMPLES

1) Remove all sample jars from the heated space and allow them to cool sufficiently to safely handle the jars.

2) Collect one of the unopened 500 mL jars for analysis.

3) If it is necessary to transport the sample to the testing location,

    a. Confirm the lid is still tight/sealed.
    b. Wrap container with protective, absorbent material.
    c. Place in a plastic bag.
    d. Place in a box or other container for safe transportation.

4) Aerate the remaining samples.

    a. Carefully open each sample jar.
    b. Visually inspect and photograph the fuel. Especially note the remaining volume, color change or the formation of deposits. If any of the jars display observable sample evaporation, pay special attention to resealing the jar. No additional fuel may be added, and this sample should be considered one of the backup samples and avoid using.
    c. Using a clean, alkaline washed stir rod, stir the fuel in the jar in a manner to clearly aerate the fuel but taking care to avoid splashing / spilling the fuel from the jar.
    d. Stir for 10 seconds.

Figure 28 - Showing Aeration by Stirring

5) Reseal the jars and return the remaining samples to the safety containment vessel.
6) Return to the oven and continue aging.

15.4.2. PRESSURE VESSEL – FIVE GALLON SAMPLES

1) Remove all pressure vessels from the heated space and allow them to cool sufficiently to safely handle the vessels.

2) No samples are collected until the end of the exposure.

3) Vent the vessels to release any accumulated pressure.

4) Remove the lids, being careful to avoid immersing the fuel seals in the fuel.

5) Aerate the samples.

   a. Visually inspect and photograph the fuel. Especially note the remaining volume, color change or the formation of deposits. No additional fuel may be added.
   b. Using a clean, alkaline washed stir rod, stir the fuel in the vessels in a manner to clearly aerate the fuel but taking care to avoid splashing / spilling the fuel from the vessel.
   c. Care must also be taken to avoid breaking stir rods on any internal venting structures in the vessels.
   d. Stir for 10 seconds.

Figure 29 - Example of Aerating Large Samples by Stirring

6) Reseal the pressure vessels, making sure the Viton™ seal is appropriately placed.

7) Return to the oven and continue aging.

15.5. WEEK 12

At the end of the twelve-week exposure, all samples will be collected.

1) Perform the visual inspection and photography of each sample as in prior weeks at the conclusion of the test period.

2) If necessary, package the samples for transportation to the site of the testing.

3) Assuming the five-gallon fuel sample will be provided to an outside test facility for repeat Certification of Analysis testing, carefully transfer the five gallons of fuel from the one and the five-gallon pressure vessels to a clean, labeled stainless-steel five-gallon drum.

15.6. POST EXPOSURE MEASUREMENTS

15.6.1. FILTERABLE INSOLUBLES – ASTM D4625

Note that the fuel filtered to determine the filterable insolubles must be kept uncontaminated and be collected from the receiving flask for subsequent analyses. Remove the filtered fuel from the receiving flask BEFORE rinsing the fuel funnel.

Using the 400 mL samples which have cooled to room temperature, measure the filterable insoluble content of the test fuels by ASTM D4625. A summary of the process is provided below.

1) Assemble the alkaline washed vacuum filtration apparatus including the matched set of pre-weighed membrane filters.

2) Turn on the laboratory vacuum.

3) Pour fuel from the sample container, either directly or by using alkaline cleaned glassware, into the fuel filter funnel. Continue transferring fuel until all of the fuel (400 mL) is filtered or it takes longer than 10 minutes to filter 100 milliliters.

4) Slowly turn off the vacuum and carefully remove the filter assembly from the receiving flask. Measure the amount of fuel filtered using a clean graduated cylinder. Collect the filtered fuel into a clean, labeled container for subsequent analyses.

    a. If there is still fuel in the 500 mL container to be filtered, reassemble the filtration apparatus with a second set of prepared filter membranes and finish filtering the fuel. Repeat the collection and measurement of the fuel volume.

5) Re-attach the now **empty** receiving flask to the filter assembly, re-attach the vacuum, and rinse down the inside of the fuel filter and the joint between

the fuel funnel and filter support with filtered iso-octane. See ASTM D4625, §11.2.2.1 for full description of technique. Once all of the fuel has been filtered, also rinse the interior of the sample container with filtered iso-octane and filter this rinse.

6) Carefully remove the filter membranes, dry, and reweigh them, being careful not to disturb any particulate on the membrane filter.

7) Record the test membrane and the control membrane mass to the nearest 0.1 mg.

15.6.2. ADHERENT INSOLUBLES - ASTM D4625

Using the now empty 500 mL sample container,

1) Rinse the container with two washings using 30 – 35 mL each of the filtered adherent insolubles solvent (1:1:1 solvent).

2) Collect the washings in a 100 mL beaker weighed to the nearest 0.1 mg.

3) Evaporate the solvent from the beaker at 160 °C (320 °F) by the air jet method in accordance with ASTM D381. A weighed tare beaker shall be used for moisture correcting the adherent insolubles.

4) Cool the beakers to room temperature in a desiccator that has no desiccant.

5) Weigh the beakers to the nearest 0.1 mg.

Determine the adherent insoluble content for the 100LL and the unleaded test fuel for each sample point (3, 6, 9 and 12 weeks). Compare the change in adherent insolubles over time in the 100LL, to the change in adherent insolubles over time in the unleaded test fuel.

15.6.3. POTENTIAL GUM AND PRECIPITATE – ASTM D873

Using the filtered fuel collected following determination of filterable insoluble, determine the potential gum (mg/100 mL) and precipitate content (mg/100 mL) by ASTM D873 Oxidation Stability for the 100LL and the unleaded test fuel for each sample point (3, 6, 9 and 12 weeks). Compare the change in potential gum and precipitate over time in the 100LL, to the change in gum and precipitate over time in the unleaded test fuel.

15.6.4. NET HEAT OF COMBUSTION – D4809

Determine the net heat of combustion in MJ/kg (mass) by ASTM D4809 for the 100LL and the unleaded test fuel at each sample point. Compare the change in heat of combustion over time of the 100LL, to the change in heat of combustion over time of the unleaded test fuel. To run net heat of combustion, the mass percent of hydrogen in the sample is required.

15.6.5. VAPOR PRESSURE – D5191

Determine the vapor pressure at 38 °C in kPa by ASTM D5191 for the 100LL and the unleaded test fuel at each sample point. Compare the change in vapor pressure over time in the 100LL, to the change in vapor pressure over time in the unleaded test fuel.

15.6.6. LEAD (D5059), HYDROGEN (D7171) AND SULFUR (D2622) CONTENT

Determine the lead content in mass % by ASTM D5059 X-ray Spectroscopy, hydrogen content in mass % by ASTM D7171 NMR and the sulfur content in mass % by ASTM D2622 XRF, for the 100LL and the unleaded test fuel at each sample point. Compare the change in contents over time for the 100LL to the change in contents over time in the unleaded test fuel. The hydrogen content is also required to determine the net heat of combustion.

At the end of the exposure test, all of the above tests shall be run on the week 12 fuel collected in the 400ml container and the five-gallon sample. In addition to the above tests, the samples shall also be analyzed for composition by GC-MS and shall have a complete Certificate of Analysis repeat analysis performed on the five-gallon sample.

15.7. CALCULATIONS

15.7.1. MOISTURE CONTENT BY KARL FISCHER – D6304

No calculations are required.

Report the observations in a tabular form, noting specifically any changes in water content and if there is any trend to the change. Compare the results between the fuels. It is possible for there to be changes observed that do not trend. These observations should be specifically noted.

15.7.2. FILTERABLE INSOLUBLES – ASTM D4625

Note that the fuel filtered to determine the filterable insolubles must be kept uncontaminated and collected from the receiving flask for subsequent analyses. Remove the filtered fuel from the receiving flask BEFORE rinsing the fuel funnel.

No calculations are required. Filterable insoluble are a direct measure of mass.

Report the observations in a tabular form, noting specifically any changes in filterable insoluble content and if there is any trend to the change. Compare the results between the fuels. It is possible for there to be changes observed that do not trend. These observations should be specifically noted.

15.7.3. ADHERENT INSOLUBLES

$$Corrected\ adherent\ insolubles = (B - A) - (Y - X)$$

Where:

A = mass of empty test beaker, mg
B = mass of test beaker + residue after evaporation, mg
X = mass of empty tare beaker, mg
Y = mass of tare beaker after evaporation, mg

Report the observations in a tabular form, noting specifically any changes in adherent insoluble content and if there is any trend to the change. Compare the results between the fuels. It is possible for there to be changes observed that do not trend. These observations should be specifically noted.

15.7.4. POTENTIAL GUM AND PRECIPITATE – ASTM D873

When performing ASTM D873, the potential gum is calculated according to the following equation, see D873, §10.7.

$$C = 1000 \text{ x } ((D - E) + (F - G) + 2(X - Y))$$

Where:

C = soluble gum, mg /100 mL
D = mass of test specimen beaker 1 + residue, grams
E = mass of test specimen beaker 1, grams
F = mass of test specimen beaker 2 + residue, grams
G = mass of test specimen beaker 2, grams
X = mass of tare beaker before, grams
Y = mass of tare beaker after, grams

The precipitate is directly measured and no further calculation is required.

After the data is collected for all twelve weeks, perform a regression analysis to determine whether there is a trending change in potential gum or precipitate over time. Compare any trends between the 100LL and the unleaded test fuel. It is possible for there to be changes observed that do not trend. These observations should be specifically noted.

15.7.5. NET HEAT OF COMBUSTION – D4809

When performing ASTM D4809, the net heat of combustion is calculated according to the following equation, see D4809, §3.1.2.

$$Q_n(net, 25\,°C) = Q_g(gross, 25\,°C) - 0.2122 \times H$$

Where:

Q_n (net, 25 °C) = net heat of combustion at constant pressure, MJ/kg
Q_g (gross, 25 °C) = gross heat of combustion at constant volume, MJ/kg
H = mass % of hydrogen in the sample

After the data is collected for all twelve weeks, perform a regression analysis to determine whether there is a trending change in Net Heat over time. Compare any trends between the 100LL and the unleaded test fuel. It is possible for there to be changes observed that do not trend. These observations should be specifically noted.

15.7.6. VAPOR PRESSURE – D5191

When performing ASTM D5191, the Dry Vapor Pressure Equivalent (DVPE) is calculated according to the following equation, see D5191 §14.2.

$$DVPE, kPa\ (psi) = (0.965\ X) - A$$

Where:

X = measured total vapor pressure in kPa (psi)

A = 3.78 kPa or 0.548 psi

After the data is collected for all twelve weeks, perform a regression analysis to determine whether there is a trending change in vapor pressure over time. Compare any trends between the 100LL and the unleaded test fuel. It is possible for there to be changes observed that do not trend. These observations should be specifically noted.

15.7.7. LEAD (D5059), HYDROGEN (D7171) AND SULFUR (D2622) CONTENT

When performing lead content by D5059 by X-ray spectroscopy, a calibration chart, for which the slope is a determination of grams of lead per US gallon. It is possible to calibrate the instrument to determine grams of lead per liter. Convert

the grams of lead per US gallon (liter) to mL of TEL per liter. There are 1.06 grams of lead per mL of TEL.

For determining hydrogen content by D7171 NMR, and sulfur content by XRF, the test instruments have software which reports the data in mass %. Note: the hydrogen content is also required to determine the net heat of combustion.

After the data is collected for all twelve weeks, perform a regression analysis to determine whether there is a trending change in elemental contents over time. Compare any trends between the 100LL and the unleaded test fuel. It is possible for there to be changes observed that do not trend. These observations should be specifically noted.

16. PASS/FAIL CRITERIA

There are no published pass/fail criteria for storage stability of aviation gasoline. However, experience suggests that aviation gasoline is storage stable over time. Should there be observable differences in the measured physical properties over time, especially to being out of specification limits where values exist, and similar shifts are not observed in the 100LL fuel, this would raise concerns to the stability of the test fuel.

Actual separation or visible degradation of fuel components or failure of physical properties not observed in 100LL could be considered grounds for failure.

17. TEST PERSONNEL

The testing source shall identify a single POC for conduct of this testing. This POC shall be equivalent to a Test Director who shall have the authority for oversight and supervision of those conducting the test. The identified POC shall have responsibility during the test for decisions regarding executing and integrity of the test. This local POC for the testing shall have the FAA point of contact clearly identified and contact information available.

The local designated POC is not a FAA designated representative and does not have authority to witness testing for the FAA. While the FAA may choose to identify this person as a designated representative, this identification as such is solely at the discretion of the FAA. Any identification of the POC as the designated FAA representative for the purposes of witnessing tests must be made prior to any testing occurring.

18. TEST RESULTS REPORT

Objective of the testing specified is to generate data supporting a fuel formulator's participation in the FAA PAFI testing program with the intent of demonstrating the

fuel formulations remain within acceptable limits for the use of the offered fuels. The report documenting the test results pursuant to this Test Plan shall include the supporting data, observations, test articles, and fuel certifications. It shall also include the identification of test equipment used, and evidence of equipment calibration or validation as appropriate. A table showing graphically the test results, compliance to the pass/fail criteria, and the actual test results shall be provided.

19. REFERENCES

In addition to the reference material in Section 5, the following references are related to the testing described.

1) ASTM D86, Standard Test Method for Distillation of Petroleum Products at Atmospheric Pressure

2) ASTM D130, Standard Test Method for Corrosiveness to Copper from Petroleum Products by Copper Strip Test

3) ASTM D156, Standard Test Method for Saybolt Color of Petroleum Products (Saybolt Chromometer Method)

4) ASTM D445, Standard Test Method for Kinematic Viscosity of Transparent and Opaque Liquids (and Calculation of Dynamic Viscosity)

5) ASTM D664, Standard Test Method for Acid Number of Petroleum Products by Potentiometric Titration

6) ASTM D909, Standard Test Method for Supercharge Rating of Spark-Ignition Aviation Gasoline

7) ASTM D910, Standard Specification for Aviation Gasoline

8) ASTM D974, Standard Test Method for Acid and Base Number by Color-Indicator Titration

9) ASTM D1094, Standard Test Method for Water Reaction of Aviation Fuels

10) ASTM D2386, Standard Test Method for Freezing Point of Aviation Fuels

11) ASTM D2622, Standard Test Method for Sulfur in Petroleum Products by Wavelength Dispersive X-ray Fluorescence Spectrometry

12) ASTM D2624, Standard Test Methods for Electrical Conductivity of Aviation and Distillate Fuels

13) ASTM D2699, Standard Test Method for Research Octane Number of Spark-Ignition Engine Fuel

14) ASTM D3339 Standard Test Method for Acid Number of Petroleum Products by Semi-Micro Color Indicator Titration

15) ASTM D3606, Standard Test Method for Determination of Benzene and Toluene in Finished Motor and Aviation Gasoline by Gas Chromatography

16) ASTM D3703, Standard Test Method for Hydroperoxide Number of Aviation Turbine Fuels, Gasoline and Diesel Fuels

17) ASTM D4052, Standard Test Method for Density, Relative Density, and API Gravity of Liquids by Digital Density Meter

18) ASTM D4809, Standard Test Method for Heat of Combustion of Liquid Hydrocarbon Fuels by Bomb Calorimeter (Precision Method)

19) ASTM D5059, Standard Test Methods for Lead in Gasoline by X-Ray Spectroscopy

20) ASTM D5191, Standard Test Method for Vapor Pressure of Petroleum Products (Mini Method)

21) ASTM D5453, Standard Test Method for Determination of Total Sulfur in Light Hydrocarbons, Spark Ignition Engine Fuel, Diesel Engine Fuel, and Engine Oil by Ultraviolet Fluorescence

22) ASTM D6304 Standard Test Method for Determination of Water in Petroleum Products, Lubricating Oils, and Additives by Coulometric Karl Fischer Titration

23) ASTM D7171, Standard Test Method for Hydrogen Content of Middle Distillate Petroleum Products by Low-Resolution Pulsed Nuclear Magnetic Resonance Spectroscopy

24) ASTM E659, Standard Test Method for Autoignition Temperature of Liquid Chemicals

25) PAFI TAC-PROJ-015

26) FAA UAT ARC Final Report, dated 17 February, 2012.
http://www.faa.gov/about/initiatives/avgas/archive/2012-10-05

– END OF TEXT –

APPENDIX A
TEST FUEL COA REQUIREMENTS

TEST FUEL FULL COA REQUIREMENTS PRELIMINARY (PRE-TEST)

Analyze the fuel for ASTM D910 Table 1 properties, plus any additional parameters in the fuel company specification as well as those in the Table below that are beyond D910. If there are multiple methods allowed in D910, use any specific methods referenced below. Substitute any test method(s) specified by the fuel producer that vary from D910 methods (e.g. color), after coordination with, and approval from the AFPO.

Highlighted entries are not required Elevated Temperature Testing

PROPERTY	TEST METHOD
Moisture Content by Karl Fischer	D6304
MON	D2700
SC PN	D909
Lead content	D5059
Color	D156
	D1500
Density	D4052
Distillation	D86
Vapor Pressure	D5191
Freeze Point	D2386
Sulfur Content	D2622
	D5453
Net heat of combustion	D4809
Hydrogen content (Required for Net Heat of Combustion)	D7171
Corrosion, Copper strip	D130
Potential Gum and Precipitate	D873
Water Reaction	D1094
Electrical Conductivity	D2624
Water Solubility	E203
Corrosion, acidity, alkalinity	D974
	D2896
Total Acid Number	D664
Particle Contamination	D5452
Existent Gum	D381

Peroxides	D3703
Oxidation Stability	D525
Viscosity	D445
Filterable Insolubles & Adherent Insolubles	D4625
Qualitative Gas Chromatography / Mass Spectrometry	D6733

POST TEST FUEL TEST
AMBIENT AND ELEVATED STORAGE (5 GALLON) - WEEK 12

Analyze the fuel for ASTM D910 Table 1 properties, plus any additional parameters in the fuel company specification as well as those in the Table below that are beyond D910. If there are multiple methods allowed in D910, use any specific methods referenced below. Substitute any test method(s) specified by the fuel producer that vary from D910 methods (e.g., color), after coordination with, and approval from the AFPO.

Highlighted entries are not required Elevated Temperature Testing

PROPERTY	TEST METHOD
Moisture Content by Karl Fischer	D6304
MON	D2700
SC PN	D909
RON	D2699
Lead content	D5059
Color	D156
	D1500
Density	D4052
Distillation	D86
Vapor Pressure	D5191
Freeze Point	D2386
Sulfur Content	D2622
	D5453
Net heat of combustion	D4809
Hydrogen content (Required for Net heat of combustion)	D7171

Corrosion, Copper strip	D130
Potential Gum and Precipitate	D873
Water Reaction	D1094
Electrical Conductivity	D2624
Water Solubility	E203
Corrosion, acidity, alkalinity	D974
	D2896
Total Acid Number	D664
Particle Contamination	D5452
Existent Gum	D381
Peroxides	D3703
Oxidation Stability	D525
Viscosity	D445
Filterable Insolubles & Adherent Insolubles	D4625
Qualitative Gas Chromatography / Mass Spectrometry	D6733

AMBIENT AND ELEVATED EXPOSURE POST TEST FUEL TEST WEEKS 3, 6, 9, AND 12 (500 ML)

Highlighted entries not required for Elevated Exposure Test

PROPERTY	TEST METHOD
Moisture Content by Karl Fischer	D6304
Color	D156
	D1500
Vapor Pressure	D5191
Sulfur Content	D2622
	D5453
Lead content	D5059
Net heat of combustion	D4809
Hydrogen content (Required for Net heat of combustion)	D7171
Potential Gum and Precipitate	D873
Filterable Insolubles & Adherent Insolubles	D4625
Qualitative Gas Chromatography / Mass Spectrometry	D6733

APPENDIX B
EXAMPLE BULK EVAPORATION TABLE

	Week	Initial Volume (gal)	Final Volume (gal)	Fuel Loss (gal)	Total Fuel Loss (gal)
Baseline 100LL Sample	0	22.62	21.32		
	3	21.19	19.63	0.13	
	6	19.37	17.81	0.26	
	9	17.55	15.99	0.26	
	12	15.73	8.58	0.26	0.91
Test Fuel	0				
	3				
	6				
	9				
	12				

– END OF APPENDICES –