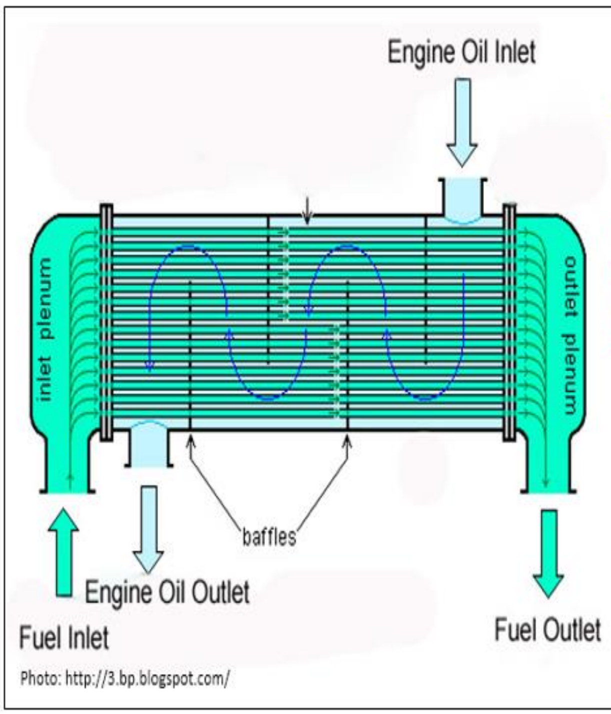
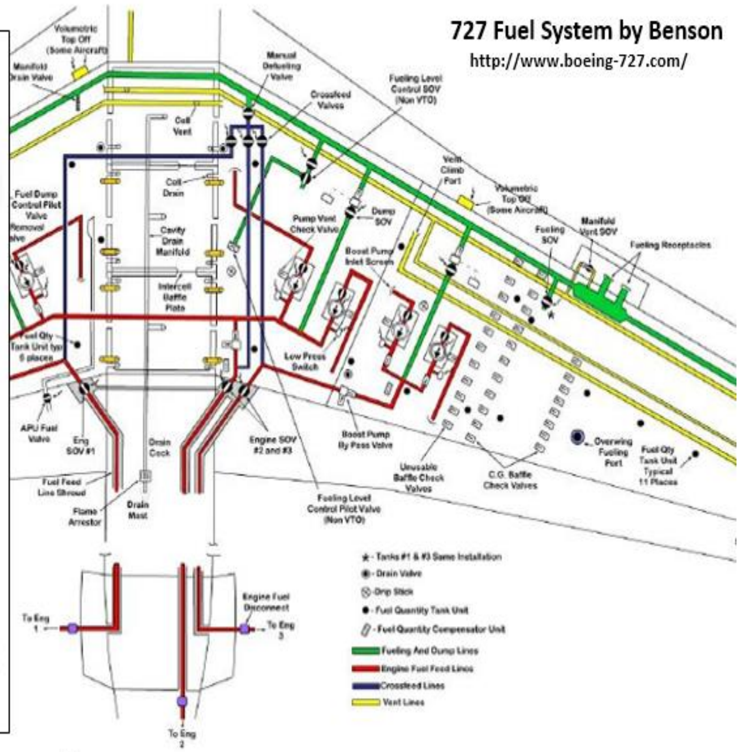
Technical Notes
#40

We're Going Global

ASTM D3241 Thermal Oxidation Stability using Spectroscopic Ellipsometry

In flight, cold jet fuel and hot engine oil pass each other in a heat exchanger. This transfer warms up the jet fuel and cools the engine oil and hot parts that would otherwise overheat at the high temperatures encountered. The thermal stresses experienced in modern jet engines can lead to the formation of undesirable and possibly harmful insoluble materials, such as lacquers on heat exchangers and control surfaces. Thus, jet fuel must have a high thermal stability and must not break down and deposit coke and varnishes in the fuel system passages.





Thermal Oxidation Stability of Aviation Turbine Fuels

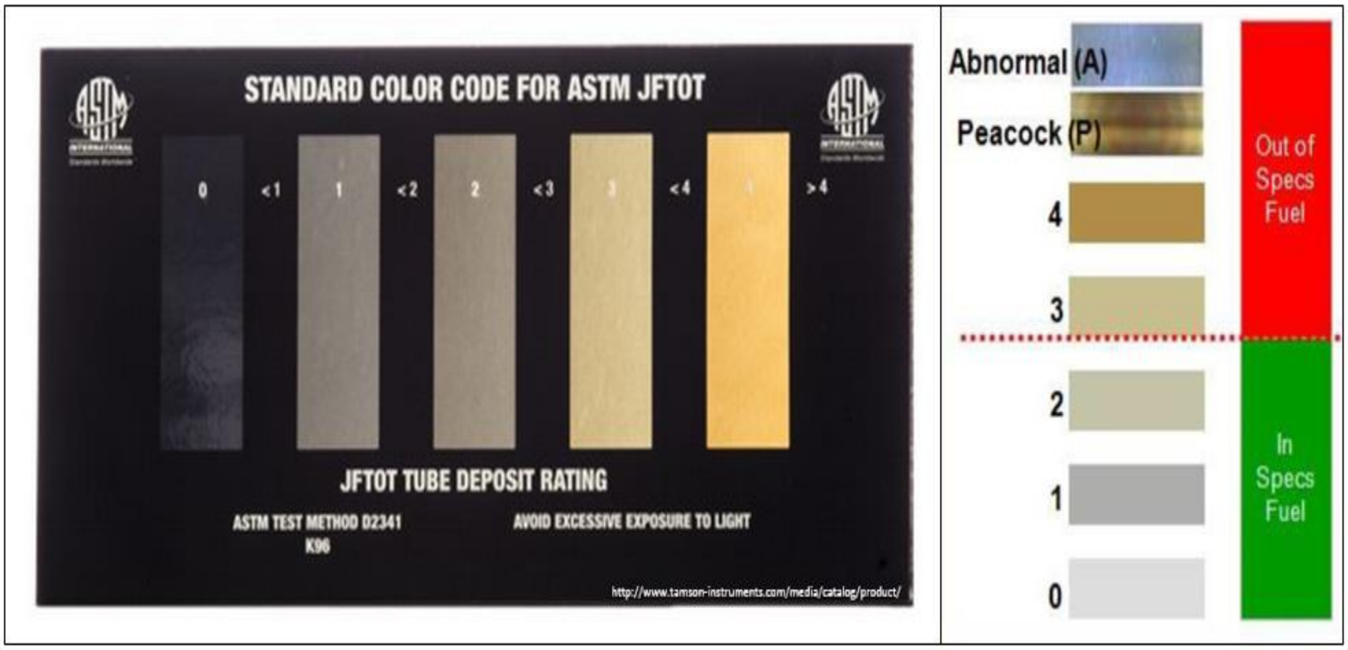
As discussed in the AmSpec TechTalk # 027, the test used to check jet fuel stability is ASTM D3241 (IP 323) Thermal Oxidation Stability of Aviation Turbine Fuels.

This test rates the tendency of gas turbine fuels to deposit decomposition products within the fuel system. The test results are indicative of fuel performance during gas turbine operation and can be used to assess the level of deposits that form when liquid fuel contacts a heated surface.

During the test, fuel is pumped over a heated aluminum alloy tube and through a very fine, heated stainless steel screen at a constant flow rate. After contacting the tube, the fuel is filtered to collect any solid decomposition products. The pressure drop across the filter is monitored during the test. At the end of the test, the tube is removed and visually examined for any stain or discoloration.

The operator utilizes a standardized Visual Tube Rater (VTR) to determine the color rating. The VTR is an internally lit black box with three 30 W incandescent bulbs and the color chart seen below on the left. The operator rates the tube deposits on a scale of 0 to 4.

Ratings using the Visual Tube Rater can be <u>subjective</u> between technicians and laboratories.



The industry specifications for on jet fuel thermal oxidation stability are a maximum pressure drop of 25 mm/Hg, a maximum tube deposit less than Code 3 and no peacock or abnormal color deposits.

Aviation Fuel Thermal Stability Characterization through Application of Ellipsometry

A NASA research team at the Glenn Research Center in Cleveland, Ohio developed a method to eliminate operator subjectivity. They found that a quantitative assessment of the physical characteristics of oxidative fuel deposits provides a more powerful method for comparing the thermal oxidation stability characteristics of fuels, especially in a research setting. They employed a Spectroscopic Ellipsometer to determine the film thickness and profile of oxidative fuel deposits on the heater tubes.

Once samples have been created through the ASTM D3241 procedure, film thickness can be attained using an ellipsometer. Inside the ellipsometer, the sample tube is positioned under optical sensors. Once positioned here, the ellipsometer obtains optical data on the full length of the tube at 1 mm intervals. From this, raw information regarding the film's optical properties are gathered and the data is plugged into a dispersion model to determine the actual film thickness.

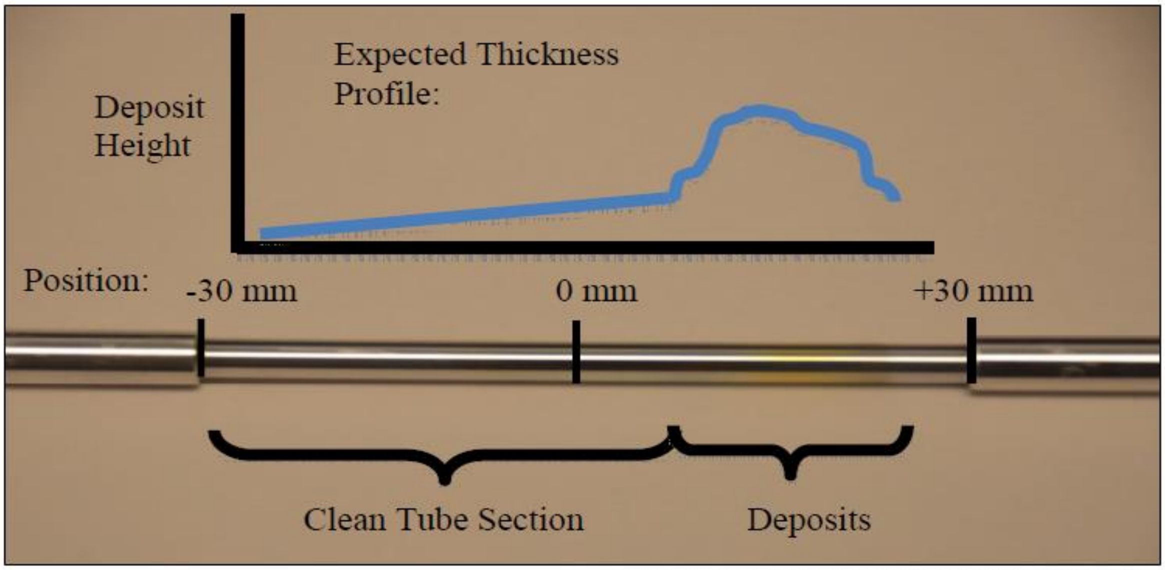


Photo: http://ntrs.nasa.gov/archive/nasa/casi.ntrs.nasa.gov/20120005439.pdf

The full NASA research paper and findings can be found at:
http://ntrs.nasa.gov/archive/nasa/casi.ntrs.nasa.gov/20120005439.pdf

ASTM D3241-14b A3 Rating of D3241 Heater Tubes - Ellipsometric Method

A3.1 Scope

A3.1.1 This annex describes a procedure for the ellipsometric rating of heater tubes produced by Test Method D3241—Thermal Oxidation Stability of Aviation Turbine Fuels.

A3.1.2 The final result from this rating procedure is an absolute measurement of the deposit thickness on the heater tube that can provide a basis for judging the thermal oxidative stability of the fuel sample. For aircraft fuel systems performance, deposit thickness is a useful parameter.

A3.3 Summary of Test Method

A3.3.1 An ellipsometric apparatus is used to rate the deposits on the D3241 heater tube. The computer-driven software analyses the ellipsometric data and the maximum deposit thickness of an average 2.5 ^{mm2} is derived and displayed.

A3.4 Significance and Use

A3.4.1 The final tube rating is a direct measure of the thickness of the degraded fuel deposited on the tube. This rating is a basis for judging the thermal oxidative stability of the fuel sample.

A3.10 Procedure

A3.10.1 Run the instrument in accordance with the manufacturer's operating instructions.

A3.10.2 At the end of the measurement procedure, record the maximum average 2.5 mm^2 deposit thickness in nanometers, nm. When using the test resolution of 24 × 50 points, the 2.5 mm^2 area is defined 6 points in a row or any set of 3 consecutive pairs of points. The manufacturer includes an algorithm that automatically identifies all 6 point combinations, calculates the average thickness of each of these combinations, selects the maximum average 6 point combination, displays the maximum average and shows the location of the maximum on the deposit graph.

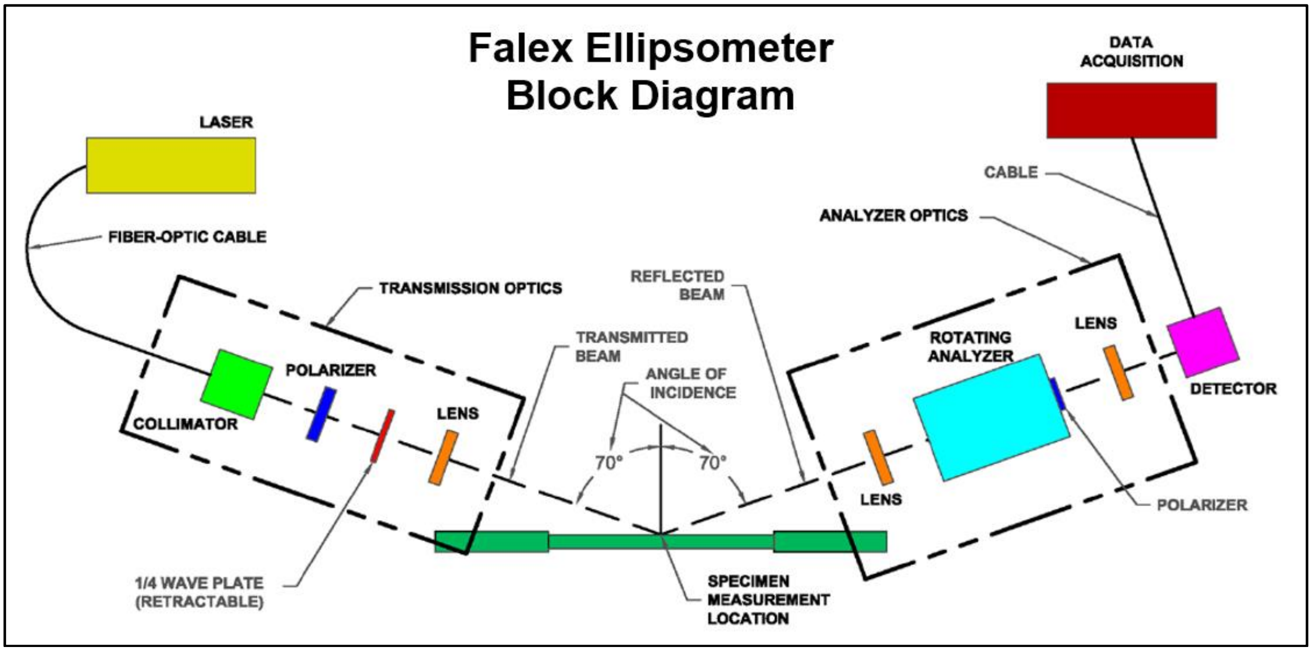
The Falex Ellipsometer at AmSpec Boston

The Falex Ellipsometer at our AmSpec Boston laboratory offers a consistent and precise analytical measurement of heater tube deposits in accordance with the ASTM D3241-14b A3 procedure. It was developed by Falex in cooperation with leading manufacturers and consumers of jet fuel. It is a fully automated, laser light source, optical detection system for measuring the film thickness of ASTM D3241 Heater Tube.

The test is reported as the maximum average $2.5mm^2$ deposit thickness in nanometers and the ASTM D1655.15 specification is 85nm maximum.

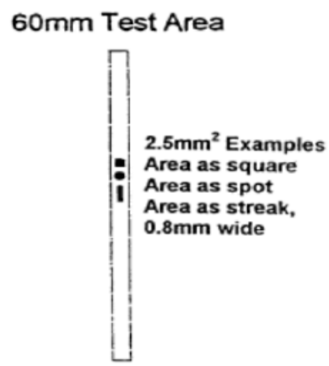
1-Test required by ASTM D3241 Appendix 3.
24 steps by 50 scans – Average length observed: 1.5-3hrs.

2-Quick check, not approved under D3241 A.3
24 steps by 25 scans – Average length observed: 0.5-1.5hrs.



PASS / FAIL
2.5 mm^2 Average Film Thickness Determination

- D3241 Darkest 2.5 mm^2 Rating Color on Strip
- Ellipsometer Uses Equivalent Area Evaluations
- Software Configured for ASTM D3241 Testing
- 5 mm to 55 mm along heater tube
- Historical Ellipsometer Data 85 nm = VTR Value 3
- Software Determination Max Thickness & Location
- PASS or FAIL Notification (Graphic Color Coded)
- Test Report Data and Test Information



For more information on **ASTM D3241 Heater Tube deposits using the Falex Ellipsometer**, please contact **AmSpec** at:

Phone: (617) 389-7272 or
Email: Boston@AmSpecGroup.com