AEROSPACE RECOMMENDED PRACTICE

SAE ARP5996

REV. A

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Superseding ARP5996

Evaluation of Coking Propensity of Aviation Lubricants Using the Hot Liquid Process Simulator (HLPS) Single Phase Flow Technique

1. SCOPE:

This method is designed to evaluate the coking propensity of synthetic ester-based aviation lubricants under single phase flow conditions found in certain parts of gas turbine engines, for instance in bearing feed tubes. This method is applicable to lubricants with a coking propensity, as determined by this method, falling in the range 0.01 to 3.00 mg.

2. APPLICABLE DOCUMENTS:

The following publications form a part of this document to the extent specified herein. The latest issue of SAE publications shall apply. The applicable issue of other publications shall be the issue in effect on the date of the purchase order. In the event of conflict between the text of this document and references cited herein, the text of this document takes precedence. Nothing in this document, however, supersedes applicable laws and regulations unless a specific exemption has been obtained.

2.1 Alcor, Hot Liquid Process Simulator, User's Manual.

3. WARNING:

This document may involve hazardous materials, operations, and equipment. This document does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of this document to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.

4. OUTLINE OF METHOD:

4.1 A measured volume of sample is placed in the apparatus. The apparatus is pressurized with air and the sample is then pumped through the system over a resistance-heated, tube-in-shell, heat exchanger for a period of 20 hours. The weight of deposit formed on the tube is then determined.

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4.2 This method assumes a degree of familiarity with the HLPS equipment. Users must, therefore, familiarize themselves with the apparatus, and the user's manual, before attempting to use this method.

5. APPARATUS:

5.1 Alcor, Hot Liquid Process Simulator (HLPS) 330, 320 or equivalent. It is recommended that the instrument be operated in a temperature-controlled environment to reduce the impact that room temperature fluctuations may have on method precision.

5.2 Oven capable of maintaining a temperature of 100 °C ± 5 °C.

5.3 Desiccator, dedicated solely for this test, filled with a suitable desiccant. It is recommended that the desiccator is operated in a temperature controlled environment to reduce the impact that room temperature fluctuations may have on method precision.

5.4 Three-channel chart recorder or other data storage device suitable for recording the output from type J thermocouples (not applicable for HLPS 400 series).

5.5 Laboratory balance capable of weighing heater tubes to 0.01 mg. It is recommended that the balance is operated in a temperature controlled environment to reduce the impact that room temperature fluctuations may have on method precision.

5.6 Measuring cylinder, 250 ml capacity.

5.7 Boiling tube, of dimensions such that the top of the center section of the heater tube is at least 30 mm below the top of the boiling tube, or other suitable receptacle, in which to soak the heater tubes in petroleum spirit.

6. REAGENTS AND/OR MATERIALS:

6.1 Stainless steel (grade 316) short heater tubes.

6.2 Acetone GPR.

6.3 Petroleum spirit (any type between 40 to 60 °C and 80 to 100 °C) GPR.

6.4 Trisolvent (equal quantities of acetone, propan-2-ol and toluene; all GPR grade).

6.5 Lead, 99% minimum purity, for thermocouple performance verification.

6.6 Tin, 99% minimum purity, for thermocouple performance verification.

6.7 Pre-weighed Filter papers, Teflon 5 m (suggest Millipore Cat no LSWP04700) (see Appendix C).

6.8 Reference Heater Tube:

A heater tube complying with the requirements of 6.1 of known mass. This tube is used solely as a control for detecting inconsistencies in the method weighing process and is not to be used as a sample analysis tube.

6.9 Compressed air supply, clean and oil free, capable of pressurizing the sample reservoir to 1380 kPa (200 psi).

7. SYSTEM VERIFICATION:

NOTE: Apart from the daily tube thermocouple calibration (7.1.2), it is not necessary to perform all of the system verification tests indicated below before every test. They should be conducted as often as is necessary to provide the user with confidence that the system is functioning correctly and consistently. It is recommended, however, that the temperature profile obtained during the test procedure (clause 9.10) be compared with that obtained during the last system verification check to detect any inconsistencies

7.1 Heater Tube Temperature Control Thermocouple:

7.1.1 The thermocouples should be checked in accordance with the method stated in the HLPS User's Manual. However, in addition to using the eutectic temperature of tin (232 °C), the performance should also be evaluated using a lead standard which has a eutectic point of 327 °C. The temperature and power levels quoted in the manual have to be adjusted to accommodate the eutectic temperature of the lead. Some users may find it more convenient to use a central or external calibration service in lieu of the above. This is permissible provided the service results are in a degree of calibration equivalent to that provided by the above method. In cases of dispute, the tin and lead eutectic method shall be the referee method. Calibration should be carried out every 6 months as a minimum.

7.1.2 The values obtained for the eutectic temperatures of lead and tin should be within ±3 °C of the quoted eutectic temperature. If this is not the case then the thermocouple in question should be replaced and the verification step (7.1.1) repeated.

7.1.3 Whichever calibration scheme is used, any deviation from the true melting point temperature values of more than 1 °C shall be compensated for when setting control temperatures, and when taking temperature measurements, by applying an appropriate correction to the setting or measurement. The degree of correction required may not be constant across the temperature range being used. Therefore, the extent of the calibration should be sufficient to determine the corrections necessary for the critical temperature settings.

7.1.4 Before the test commences check the heater tube thermocouple for proper position. Raise the slide assembly so that the top is even with the reference line on the scale (top of scale). The thermocouple tip must be flush with the top of the heater tube and upper bus bar end cap.

7.2 Pump Flow Rate:

7.2.1 Set the pump to obtain a flow rate of 1 ml min^{-1}. It has been determined that a flow rate of 1 ml min^{-1} will yield 20 drops (including a count of drop zero) in 30 seconds from the reservoir return tube. The appropriate flow controller setting can be determined by this method.

7.3 Power Setting:

7.3.1 The temperature of the tube is measured via a height adjustable thermocouple positioned within the HLPS tube. The test requires that the specified maximum tube temperature be achieved at position 10 as measured on the thermocouple (right hand) height scale. The power level required to achieve the specified temperature will depend on the type of power controller employed. Reference should be made to the user instructions for the controller employed.

7.3.2 To ascertain the power level required, set up the test as defined in the procedure section. Align the top of the thermocouple slide with the hottest pre-determined point 'A' (i.e., 10 position on the right hand side of the thermocouple height scale). Run the test and monitor the temperature. Adjust the power level until the required temperature is achieved (not applicable for the HLPS 400 series).

7.4 Tube Temperature Profile:

7.4.1 One of the most important stages in setting up the HLPS apparatus for this test is to determine the position of the hottest point on the tube, and the tube temperature profile. Appendix A explains how to do this, shows a typical profile and discusses the factors that can affect both the hottest point and the general profile. Users who are unfamiliar with these critical issues should refer to Appendix A before proceeding any further.

7.4.2 The procedure in Appendix A should also be performed following any significant changes to the system such as replacement of heating system parts, or after thermocouple calibration. In such cases it is acceptable to start the verification process using the previously known position A.

7.4.3 Determination of the tube temperature profile is also part of the test procedure. Profiles obtained at the beginning of test runs should be compared with those obtained at 7.4.1 and 7.4.2. Such comparisons can be used to ensure that the system has remained stable. If the comparison shows a deviation then the causes should be investigated (see Appendix A). If the hottest point on the tube has moved and it is not possible to return it to the previous position A following the investigation, then this must become the new position A.

8. PREPARATION OF APPARATUS:

8.1 Clean the pump by passing through a minimum of 150 mls acetone followed by 150 mls of the lubricant to be examined. This is operator specific but may be done by first attaching specially designed cleaning tubes. Discard the lubricant used to clean the pump (see Appendix B).

8.2 Once the last of the 150 mls test lubricant has passed through the pump, continue to allow the pump to run until the majority of lubricant has been removed from the pump.

8.3 Any remaining oil pipes and oil-wetted parts that were not cleaned at 8.1 should be flushed with the acetone and blow dried with a clean, oil-free air supply. All O-rings related to the thermocouples and pipework should be changed for each test. The two large seals in the reservoir may be reused and should be cleaned with the petroleum spirit and not be exposed to the acetone, which will cause them to swell.

8.4 To clean out the reservoir remove both the top and bottom of the vessel.

8.5 Clean the heater test section with a nylon brush and acetone. Blow dry with a clean, oil-free air supply. If deposits remain following this procedure then the use of Trisolvent is recommended.

8.6 Take a new heater tube and rinse the outside and inside with the acetone. Dry the tube in the oven at 100 °C ± 5 °C for 30 minutes and cool in a desiccator filled with a suitable desiccant for at least 30 minutes. Perform the same process with the reference heater tube. Following cleaning these tubes should be handled with PTFE coated or silicone tipped forceps. Touching the narrow center section of the tube must be avoided.

8.7 Weigh and record the masses of the cleaned tubes to an accuracy of 0.01 mg. Return the reference heater tube to the dedicated desiccator until required for the post test weighing. The other heater tube is to be used for the oil sample test.

8.8 Mount the test heater tube within the test section such that the groove on the test piece is at the top and the shoulders may be seen centrally positioned through the side arms.

8.8.1 Assemble the HLPS lines and fittings as specified in the HLPS manual for the standard test without filters or the differential pressure cell. The stirrer and holder should be removed from the reservoir and the system configured to allow recirculation of the lubricant under test.

8.9 Charge the reservoir using a measuring cylinder with 100 ml ± 2 ml of the lubricant to be tested. Bolt the reservoir lid into place. Place the reservoir heater jacket around the reservoir.

8.10 Align the top of the thermocouple slide surface with the hottest pre-determined point (i.e., position A on the right hand scale) (see Appendix A).

8.11 If used, couple the chart recorder or other data storage device to the HLPS apparatus according to the manufacturer's instructions and the HLPS operating manual.

9. PROCEDURE:

9.1 Turn the pump on. Initially set the flow rate controller to the 999 setting for 2 minutes and then reduce to the setting determined previously to approximately yield a flow rate of 1 ml/min.

9.2 Set the heater jacket controller to 150 °C. Turn the heater jacket power supply on.

9.3 Allow the system to equilibrate for 30 minutes.

9.4 Slowly pressurize the system with 1380 kPa (200 psi) of clean, oil free air while checking for any leaks or abnormalities.

9.5 Check that the flow rate is running at 1 ml/min. This is consistent with 20 drops (starting with count zero) in 30 seconds ± 2 seconds. Adjust the flow rate if necessary.

9.6 Repeat 9.5 three times and record the times.

9.7 Program the HLPS tube temperature controller to achieve the following time/temperature profile:

Ramp tube temperature from ambient to X °C over a 10 minute period.

Hold the tube temperature at X °C for Y hours.

At the end of Y hours, return to ambient over a 10 minute period.

Where:

X °C = the specified tube temperature
Y hours = the total specified test duration

Turn the power switch on and set the controller to run in automatic mode using the above parameters.

9.8 Run the test continuously until the end of the required test duration.

NOTE: From here on in to the method all reference to numbers on the scale are for reference only and should be adjusted according to the hottest pre-determined point (see Appendix A).

9.9 After the instrument has maintained the desired temperature for 30 minutes place the controller in manual mode. Obtain a tube temperature profile by moving the thermocouple and measuring the temperature at the following positions on the height scale: A-4, A, A+4, A+10, A+14, A+20, A+30, A+40, A+50 where A = the hottest point on the test heater tube (see NOTE at 9.8). Record the profile, the inlet temperature, the outlet temperature, the reservoir temperature, the power level and pressure.

NOTE: Before entering the manual mode, monitor the power level or tube temperature for a few seconds to ensure that it is stable. Erroneous profile readings may result if the manual mode is activated while the power level/temperature is fluctuating.

9.10 Return the thermocouple to ‘A’, the hottest pre-determined point (i.e., 10 position), place the system back in automatic mode and allow the controller to stabilize.

9.11 Place the controller in manual mode again, record the temperature at A+40 (i.e., 50 position), only and then repeat 9.10.

9.12 Place the controller in manual mode for a third time, and record the temperature at A+40 (i.e., 50 position) only. Leave the thermocouple in the A+40 position.

9.13 Calculate the mean temperature at A+40 (i.e., 50 position) from the three values obtained in 9.10, 9.11 and 9.12.

9.14 The time taken to record the temperature profile and establish the mean A+40 position temperature should ideally take between 10 and 20 minutes. However, up to 30 minutes is allowed for this process. If more than 30 minutes is required then this should be considered abnormal and the causes investigated.

9.15 Program the instrument to maintain the mean temperature calculated in 9.13. Return the controller to automatic and leave the thermocouple at A+40 (i.e., 50 position) for the remaining duration of the test.

9.16 Record the tube temperature, inlet temperature, outlet temperature, reservoir temperature and the power level at regular intervals. It is advisable to obtain a tube temperature profile in the last hour of the test for comparison and potential trouble shooting. If required, further tube temperature profiles may be obtained during the test, although it is advisable to keep these to a minimum to avoid disturbing equipment stability. In each case the operator must ensure that the thermocouple is returned to A+40 (i.e., 50 position), and that no changes are made to the temperature settings.

NOTE: Profiles obtained later in the test may reveal that the maximum tube temperature has risen above X °C due to the insulating effect of deposits formed. Such an occurrence is considered to be a normal process of the test and so the instrument should not be adjusted to compensate for it.

9.17 At the end of the test, turn off the power to the heater jacket and remove the jacket from around the reservoir.

9.18 Turn off the power to the heater tube.

9.19 Allow the system to cool for at least 10 minutes.

9.20 Turn off the pump and slowly vent the system pressure.

9.21 Carefully remove the test section from the HLPS.

9.22 Working over a small beaker, gently remove the test heater tube from the test section taking care not to disturb any deposit formed. If any deposit is collected in the beaker then set the beaker in order to recover that deposit, as stated in Appendix C.

9.23 Place the test heater tube into the boiling tube and fill with petroleum spirit, to a level at least 10 mm above the top of the center section of the test heater tube. Allow the tube to soak for at least 10 minutes and then gently agitate the solution. Remove the test heater tube from the petroleum spirit and rinse through the center of the tube with the petroleum spirit. Perform the whole of the above cleaning process with the reference heater tube.

9.24 The inside of the test section should be inspected for any dislodged deposit. Any such deposit should be washed from the heater section, with the petroleum spirit, into the boiling tube.

9.25 Wash any dislodged deposit with the petroleum spirit.

9.26 Any dislodged deposit found in the boiling tube and beaker should be recovered using the method stated in Appendix C.

9.27 Dry the tubes in the oven at 100 °C ± 5 °C for 30 minutes and cool in the desiccator for at least 30 minutes.

9.28 Determine and record the masses (to the nearest 0.01 mg) of the test and reference heater tubes.

9.29 Calculate the total mass of deposit formed by adding the mass of deposit formed on the test heater tube, to the mass of any dislodged deposit recovered in 9.26.

9.30 Calculate the change in mass of the reference heater tube from that determined at 8.7.

9.31 Repeat procedures 9.23, and 9.27 to 9.30 until:

9.31.1 two consecutive determinations of total deposit mass agree within ±0.02 mg or 5% of each other, whichever is the greater

9.31.2 and two consecutive determinations of the mass of the reference heater tube agree within ±0.05 mg of the mass obtained at 8.7. If the mass of the reference heater tube has changed, from that obtained at 8.7, by more than 0.05 mg then the tube cleaning and weighing process should be considered suspect and the test repeated.

9.32 If during 9.31 further deposit is found in the boiling tube then using a new filter repeat 9.26.

9.33 Transfer the test heater tube to a labelled container.

9.34 If required, record the appearance and nature of the deposit on the test heater tube. Comparing the test heater tube to a tube, pre-marked with height graduations, may help with determining where deposit is actually forming.

9.35 Transfer the used lubricant to a labelled bottle or flask.

9.36 Additional physical and chemical testing may be performed on the used sample as required.

NOTE: The coking propensity of a lubricant sample should ideally be determined in duplicate. As a minimum requirement, the total deposit mass generated by replicate tests should agree within the repeatability as stated in clause 11 of this method.

10. REPORTING:

10.1 Report the total deposit mass to the nearest 0.01 mg.

10.2 If required, report the appearance and nature of the deposit on the test heater tube.

10.3 If required, report the results of the additional physical and chemical tests performed on the used sample.

11. PRECISION:

This is an interim statement of the precision of this method based on the analysis of four lubricants, two medium and two low coking propensity. The data used to compile this precision statement was generated by seven laboratories analyzing the four samples in duplicate.

Repeatability: 26%
Reproducibility: 65%

The test conditions used were:

Maximum tube temperature (X °C) = 375 °C
Test duration (Y hours) =20 hours

Analysis of further pairs of samples will continue until sufficient data has been accumulated to enable a final precision statement to be generated.

PREPARED UNDER THE JURISDICTION OF
SAE COMMITTEE E-34, PROPULSION LUBRICANTS

APPENDIX A
DISCUSSION OF FACTORS THAT MAY INFLUENCE METHOD PRECISION

A.1 DETERMINATION OF THE HOTTEST POINT ON THE TUBE, AND THE TUBE TEMPERATURE PROFILE.

A.1.1 Assemble and run the apparatus in accordance with Sections 8 and 9, although preparation and weighing of the test tube is not necessary for purely setting up the system and determining the temperature profile. To start with the tube temperature thermocouple should be set at the 10 position.

A.1.2 Once the required temperature at the 10 position has been achieved, allow the system to equilibrate for 30 minutes before determining the tube temperature profile as described below.

A.1.3 Place the controller in manual mode. Obtain a tube temperature profile by moving the thermocouple and measuring the temperature at the following positions on the height scale, 6, 10, 14, 20, 24, 30, 40, 50, 60. Record the profile and plot a graph of thermocouple position versus temperature. The resulting temperature profile, when presented in graphical form, should be similar in appearance to that shown in Figure A1, with the maximum temperature at, or near, the 10 position. The hottest point on the tube is referred to as position “A”. If A is not at the 10 position then the thermocouple must be moved to position A, the system allowed to equilibrate at the desired temperature and a new profile determined. This time the measurements should be taken at the following positions: A-4, A, A+4, A+10, A+14, A+20, A+30, A+40, A+50. A new profile graph should be constructed to confirm that position A has not moved again. This process should be repeated until a consistent position A is obtained. If position A deviates from the 10 position by more that a few millimeters, or if the profile is significantly different in appearance to that shown in Appendix A then it is likely that the system is not set up correctly and the cause should be investigated. Potential causes and solutions are discussed in Section A.2.

A.1.4 Data collected during the round robin exercise to determine the precision of this method has shown that Figure A1 represents a typical tube temperature profile, with the maximum tube temperature at the 10 position.

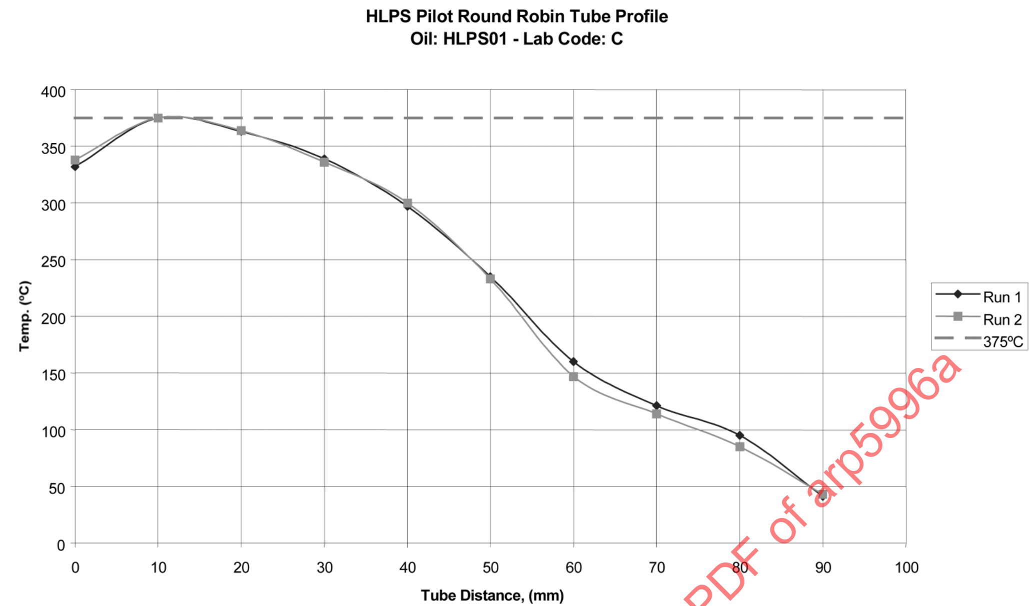

FIGURE A1

A.2 FACTORS INFLUENCING THE SHAPE OF THE TUBE PROFILE

A.2.1 There are six criteria that may effect the maximum tube temperature, its location on the tube, and the appearance of the tube profile.

1. Tube power level.
2. Sample flow rate.
3. Sample inlet temperature.
4. Cooling water flow rate.
5. Cooling water inlet temperature.
6. Laboratory temperature.

If the tube profile obtained is significantly different to that shown above, or the profile has shifted such that the hottest part of the tube is not at the 10 position, then the various parameters shown above should be investigated as potential causes.

A.2.2 The critical element of the tube temperature profile is the maximum tube temperature, rather than its position. The aim should be to achieve a profile similar to Figure A1 with the maximum tube temperature at the 10 position. However if, after investigating all of the above parameters, it is not possible to achieve this then it is important to at least ensure that no point on the tube exceeds X °C. Under such circumstances efforts should be made to bring the maximum tube temperature as close to the 10 position as possible. Achieving this may be a matter of trial and error, but determining the position of the hottest point on the tube is critical to the correct operation of this test method. Once determined the tube profiling positions must be amended accordingly as described in Section A.1 (i.e., A-4, A, A+4, A+10, A+14, A+20, A+30, A+40, A+50 where A=the hottest pre-determined point on the heater tube).

A.2.3 For a stable system the location of A should not change significantly over time. This should be determined during validation checks (7.4) and the initial profile determination during a test (9.9). A significant variation in the position of A should be cause for investigation.

A.2.4 Profiles obtained later in a test may reveal that the maximum tube temperature has risen above X °C due to the insulating effect of deposits formed. Such an occurrence is considered to be a normal process of the test and should not be cause for concern.

A.2.5 It is recommended that the HLPS apparatus is operated in a temperature controlled environment to reduce the impact that room temperature fluctuations during the test may have on method precision.

A.2.6 The cooling water supply for the instrument should be checked for temperature stability. If it has a tendency to fluctuate during tests then a temperature controlled supply should be used.