# AEROSPACE RECOMMENDED PRACTICE

| ARP5996™ | REV. C |
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## Evaluation of Coking Propensity of Aviation Lubricants Using the Single Phase Flow Technique

RATIONALE

The tendency of a lubricant to form deposits in an engine oil system is a critical factor that can influence engine design, performance and maintenance intervals. Therefore it is important to have an understanding of the coking propensity of the lubricant. The ARP has been revised to make the method more applicable to the use of more modern instruments and to introduce a procedure for extending the test for a further 20 hour period.

## 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 5.00 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 (HLPS), or Falex Thermal Fouling Tester (FT2), user's Manual appropriate to the type and series of instrument being used.

## 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.





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## 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 specified period (normally 20 or 40 hours). The weight of deposit formed on the tube is then determined.

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

## 5. APPARATUS

5.1 Any of the instruments listed below are considered suitable for this method. 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.

5.1.1 Alcor, Hot Liquid Process Simulator (HLPS) 300 series.

5.1.2 Alcor, Hot Liquid Process Simulator (HLPS) 400 Series.

5.1.3 Falex Thermal Fouling Tester (FT2).

5.2 Data recording/storage device suitable for recording the output from the various instrument thermocouples.

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

5.4 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.

5.5 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 cylinders, 100 ml and 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. A diagram showing the tube dimensions is shown in Appendix G. The following heater tubes have been found to be satisfactory:

PAC/Alcor part number 91747

Falex part number 400-560-003

6.2 Acetone GPR/LG.

NOTE: GPR/LG = general purpose reagent grade / laboratory grade.

6.3 Petroleum spirit (any type between BP 40 to 60 °C and BP 80 to 100 °C) GPR/LG. Heptane may be used as an alternative.

6.4 Trisolvent (equal quantities of acetone, propan-2-ol and toluene; all GPR/LG 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 µm (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, dry and oil free, capable of pressurizing the sample reservoir to 1380 kPa ± 140 kPa (200 psi ± 20 psi).

7. SYSTEM VERIFICATION

NOTE: Apart from the daily tube thermocouple calibration (7.1.1), 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 (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 instrument user's manual. If the manual states to use the eutectic temperature of tin (232 °C), the performance should also be evaluated using a lead standard which has a eutectic point of 327.5 °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 Check the heater tube thermocouple for proper position. Raise the thermocouple so that the tip is flush with the top of the heater tube and upper bus bar end cap. The corresponding position on the thermocouple height scale must be 51 mm above the zero point on the scale.

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. For instruments with a window at the top of the reservoir the appropriate flow controller setting can be determined by this method. The flow should be set to obtain 20 drops in 30 seconds ± 1 second.

7.3 Power Setting

7.3.1 The temperature of the tube is measured via a height adjustable thermocouple positioned within the heater tube. The test requires that the specified maximum tube temperature be achieved at the hottest pre-determined position "A" as measured on the thermocouple (right hand) height scale. If position A is not known see 7.4.1 and Appendix A.

7.3.2 The power level on more modern instruments is internally controlled on a feedback control loop to keep the temperature at the required set point. However, on older instruments the power level must be set manually to achieve the required temperature and the power level required will depend on the type of power controller employed. Reference should be made to the user instructions for the controller employed.

7.3.3 For manually controlled systems, 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' (start with the 10 position on the thermocouple height scale if point A is not known). Run the test and monitor the temperature. Adjust the power level until the required temperature is achieved.

7.4 Tube Temperature Profile

7.4.1 One of the most important stages in setting up the 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 of acetone followed by 150 mls of the lubricant to be examined. It is recommended that this be done by first attaching specially designed cleaning tubes (see Appendix B). Discard the lubricant used to clean the pump.

NOTE: If the equipment manufacturer recommends the use of solvents other than acetone then alternative solvents are permissible provided they completely remove the previous sample from the pump.

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 large seal(s) 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 the top and (if removable) the 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 (see Appendix B).

8.6 Take a new heater tube. Inspect the thermocouple hole and ensure there is no debris present. If in doubt, pass a probe through the hole to ensure any debris is removed. Rinse the outside and inside of the tube with pet spirit and then with the acetone. If there is any concern that rinsing alone will not remove all contaminants (such as a temporary protective coating) then it is recommended that the tube be wiped with lens tissue soaked in pet spirit and then in acetone before finally rinsing the tube with the solvents. 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 must be handled with clean gloved hands (powder free latex, PVC or other impervious materials, not cotton) or 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. Because of the high degree of accuracy required it is recommended that the tubes are weighed several times and the average of these weighings taken as the initial tube weight. 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 PAC tubes, or the company logo on Falex tubes, is at the top and the shoulders may be seen centrally positioned through the side arms.

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

8.10 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.

8.11 Insert the thermocouple into the test heater tube and adjust the height so that it is positioned at the hottest pre-determined point, position A (see Appendix A).

8.12 If used, couple the recorder or other data storage device to the apparatus according to the manufacturer's instructions and the instrument operating manual. More modern computer controlled instruments that automatically log the information during the run do not require this.



## 9. PROCEDURE

9.1 Whichever type of instrument or programming method is used it is critical to ensure that the following parameters are adhered to:

9.1.1 The test tube shall be held at the specified test temperature (X °C, usually 375 °C) for the specified test period (Y hours, usually 20 hours). The 10 minute period ramping up to the test temperature at the beginning of the test and the 10 minute period ramping down to ambient at the end of the test shall not be included in the test period (Y hours).

9.1.2 The oil flow rate during the specified test period shall be 1 ml/min.

9.1.3 The system shall be pressurized with 1380 kPa ± 140 kPa (200 psi ± 20 psi) of clean, oil free air.

9.2 Turn the pump on. Initially set the flow rate to approximately 3 ml/min until a constant stream of drops is visible in the reservoir window. Then reduce to the setting determined previously to approximately yield a flow rate of 1 ml/min.

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

9.4 Turn on the cooling water to the instrument and check that the flow rate is correct.

9.5 Allow the system to equilibrate for 30 minutes.

9.6 Slowly pressurize the system with 1380 kPa (200 psi) of clean, oil free air while checking for any leaks or abnormalities. (It is recommended to use a regulated supply, set to 300 psi / 20 bar.)

9.7 Check that the oil flow rate is running at 1 ml $min^{-1}$. This is consistent with 20 drops (starting with count zero) in 30 seconds ± 1 second. Adjust the flow rate if necessary.

9.8 Repeat 9.7 three times and record the times.

9.9 Ensure that the tube temperature controller/program is configured 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 (usually 375 °C)
Y hours = the total specified test duration (usually 20 or 40 hours)

9.10 Start the above program and 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 (point A, see Appendix A).

9.11 After the instrument has maintained the desired temperature for 30 minutes re-check the flow rate and adjust if necessary in accordance with 9.7. A tube temperature profile must then be obtained. During this process it is imperative that the temperature of the tube be maintained at X °C at the A position. However, the temperature profile process requires the temperature control thermocouple to be moved up and down within the test tube. This means that, during the profiling process, the temperature of the tube must be controlled and maintained independently of the thermocouple being used for the profiling. This may be achieved by switching the instrument to a manual control mode, or by monitoring the temperature of the oil at the outlet of the test section. Before doing this the operator must ensure the alternative method of control will provide a stable tube temperature.

9.12 Switch the instrument to control the tube temperature independently of the thermocouple as described above. 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 9.10). Record the profile, the inlet temperature, the outlet temperature, the reservoir temperature, the power level and pressure.

9.13 Return the thermocouple to 'A', the hottest pre-determined point (e.g., 10 position). If possible switch the instrument back to control the tube temperature via the tube thermocouple and allow the instrument to stabilize.

9.14 Switch the instrument to control the tube temperature independently of the thermocouple again, record the temperature at A+40 position (e.g., 50) only and then repeat 9.13.

9.15 Switch the instrument to control the tube temperature independently of the thermocouple for a third time, and record the temperature at A+40 position only. Leave the thermocouple in the A+40 position.

9.16 Calculate the mean temperature at A+40 from the three values obtained in 9.12, 9.14, and 9.15.

9.17 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.

9.18 Program the instrument to maintain the mean temperature calculated in 9.16. Return the controller to automatic and leave the thermocouple at A+40 for the remaining duration of the test.

NOTE: Instruments that have the capability to determine the temperature profile and A+40 position temperature automatically shall essentially follow the same procedure as in 9.12 to 9.18.

9.19 If the instrument has the appropriate data logging equipment, 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 troubleshooting. 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 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.

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

9.21 Turn off the power to the heater tube.

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

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

9.24 Carefully remove the test section from the instrument.

9.25 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 to one side in order to recover that deposit, as stated in Appendix C.

NOTE: During performance of 9.25 to 9.29 and 9.34 the operator is required to inspect all rinses, washes, etc., for the presence of loose coke deposits. Loose coke deposits are defined as chunks or flakes and must be visible to the naked eye. Any visible, dislodged deposits found in the boiling tube and beaker should be recovered using the method stated in Appendix C. The Appendix C procedure is not required if no loose coke deposits are generated or observed during the tube cleaning procedure.

9.26 Carefully rinse the heater tube with a stream of the petroleum spirit over a beaker and then place into a boiling tube with the petroleum spirit and allow to soak for sufficient time to remove all of the oil. If oil residue is visible in the pet spirit washings then it is recommended that this portion be tipped away and a fresh portion placed in the boiling tube. Perform the same cleaning process with the reference heater tube.

NOTE: If a residue is left on the heater tube by the O-rings, this may be removed with a gloved thumb/finger nail. If this does not remove all the of the O-ring deposit then the deposit may be removed with a piece of lens tissue soaked in acetone. Care should be taken not to disturb any deposits on the centre (narrow) section of the tube.

9.27 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 beaker used at 9.25.

9.28 Wash any dislodged deposit with the petroleum spirit.

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

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

9.31 Determine and record the masses (to the nearest 0.01 mg) of the test and reference heater tubes. Weigh the tubes multiple times until at least 3 consecutive weights, or three out of four consecutive weights, are obtained that are within 0.02 mg of each other. The three weights that differ by no more than 0.02 mg from each other are then averaged.

9.32 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 9.29.

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

9.34 If there is any reasonable doubt that all of the oil has been removed from the deposit then repeat procedures 9.26, and 9.29 to 9.33 until:

9.34.1 two consecutive determinations of total deposit mass agree within ±0.05 mg or 10% of each other, whichever is the greater

9.34.2 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.

NOTE: The need for multiple washing, drying and weighing of the heater tube should be balanced against the potential loss of deposit through this process as this may have a greater impact on the variation of deposit weight.

9.35 Transfer the test heater tube to a labelled container.

9.36 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.

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

9.38 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 Section 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. This precision statement is only applicable to lubricants that produce a deposit in the range 0.01 to 3.0 mg.

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.

## 12. NOTES

12.1 A change bar (l) located in the left margin is for the convenience of the user in locating areas where technical revisions, not editorial changes, have been made to the previous issue of this document. An (R) symbol to the left of the document title indicates a complete revision of the document, including technical revisions. Change bars and (R) are not used in original publications nor in documents that contain editorial changes only.

PREPARED BY 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 and the system has equilibrated for 30 minutes the temperature control will swap over to the outlet thermocouple in order to determine the tube temperature profile as described below.

A.1.3 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'.

A.1.4 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. Note that the actual hot spot may be as much as 4 mm from the actual 10 mm position and can be several mm long. Care should be taken to ensure the thermocouple is in the center of that hotspot. 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.1.5 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.

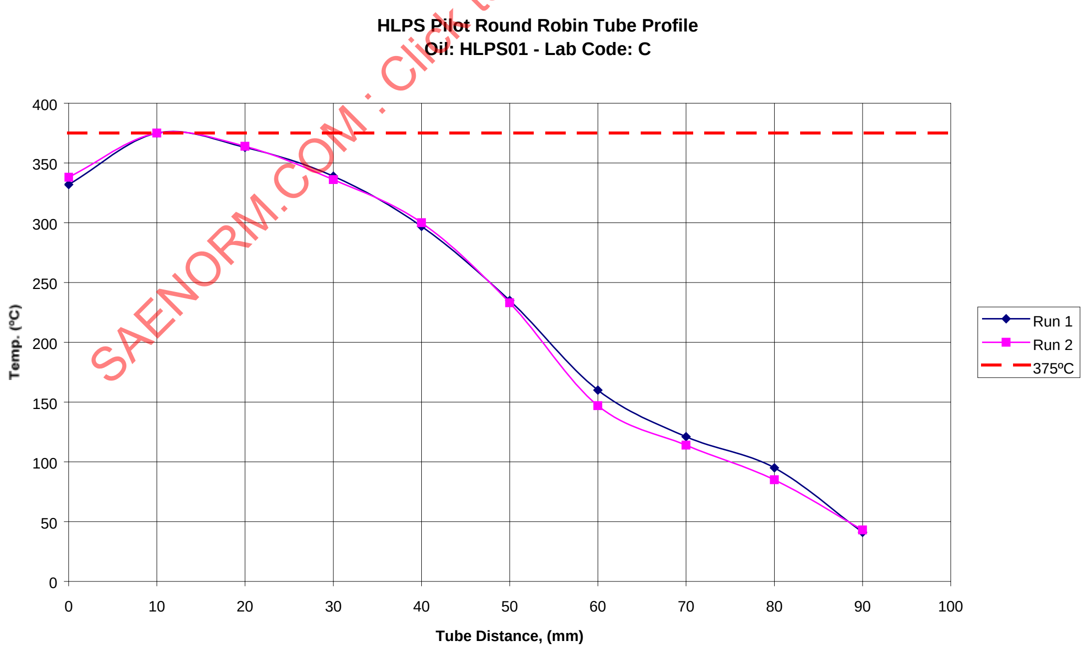



*Figure A1*

A.2 FACTORS INFLUENCING THE SHAPE OF THE TUBE PROFILE

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

- Tube power level.
- Sample flow rate.
- Sample inlet temperature.
- Cooling water flow rate.
- Cooling water inlet temperature.
- Laboratory temperature.

A.2.2 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.2.3 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.2.4 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 (9.1.6 or 9.2.9). A significant variation in the position of 'A' should be cause for investigation.

A.2.5 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.2.6 It is recommended that the HLPS apparatus be operated in a temperature controlled environment to reduce the impact that room temperature fluctuations during the test may have on method precision.

A.2.7 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.

## APPENDIX B - ADDITIONAL CLEANING PROCEDURES. THIS IS A RECOMMENDATION ONLY AS EACH OPERATOR HAS THEIR OWN CLEANING SYSTEM

B.1 Bending of system pipework during solvent and sample flushing of the pump should be avoided. Instead separate pipes can be manufactured specifically to facilitate the cleaning of the pump.

B.2 For 400 Series models to flush with acetone followed by test oil; whilst in “maintenance mode” set the flow-rate to 10 (its maximum) and press “Start”.

B.3 Periodically it is advisable to place all pipe-work, and the heater test section through a sonification process, for example in an ultra-sonic bath, in a mild detergent mix (such as de-con 90), followed by a thorough sonification with distilled water to remove any detergent, and finally with acetone. Alternatively Tri-solvent can be used for the sonification process.

## APPENDIX C - FILTERING ANY LOOSE DEPOSIT

### C.1 APPARATUS REQUIRED

C.1.1 Teflon membrane filters, having a mean pore size of 5 µm and diameter of 47 mm. (Millipore Cat. no. LSWP04700).

C.1.2 Filter holder apparatus consisting of the following (Millipore Cat. No. XX10 047 30 is suitable).

C.1.3 Borosilicate glass 300 ml funnel with ground glass seal.

C.1.4 Borosilicate glass base with ground glass seal.

C.1.5 Stainless steel screen filter support.

C.1.6 PTFE gasket for filter support.

C.1.7 Anodized aluminum spring clamp. (WARNING: The clamp shall be earthed in accordance with safe practices associated with the filtration of solvents.)

C.1.8 1 liter side arm flask with an opening comparable with the stopper of the filter holder apparatus (Millipore Cat. No. XX1004705 is suitable).

C.1.9 Covered petri dish.

C.1.10 Vacuum equipment such as vacuum pump or compressor system. (WARNING: A suitable solvent trap should be fitted to the vacuum line to prolong the life of the pump or compressor system elastomeric seals.)

### C.2 PROCEDURE FOR FILTERING DEPOSIT

C.2.1 Using flat-headed, un-serrated forceps, place the filter into a clean Petri dish. Place the Petri dish, the lid slightly ajar, in an oven set at 100 °C for 30 minutes.

C.2.2 Remove the Petri dish from the oven and allow to cool in the desiccator for 30 minutes.

C.2.3 Weigh the filter and record weight to the nearest 0.01 mg.

C.2.4 Using the forceps, place the filter paper on the stainless steel screen of the filter holder and install and clamp the holder.

C.2.5 Pour the contents of the boiling tube (9.25) into the prepared filter unit and apply vacuum. Keep the top of the filter holder covered with the foil lid to prevent ingress of dust.

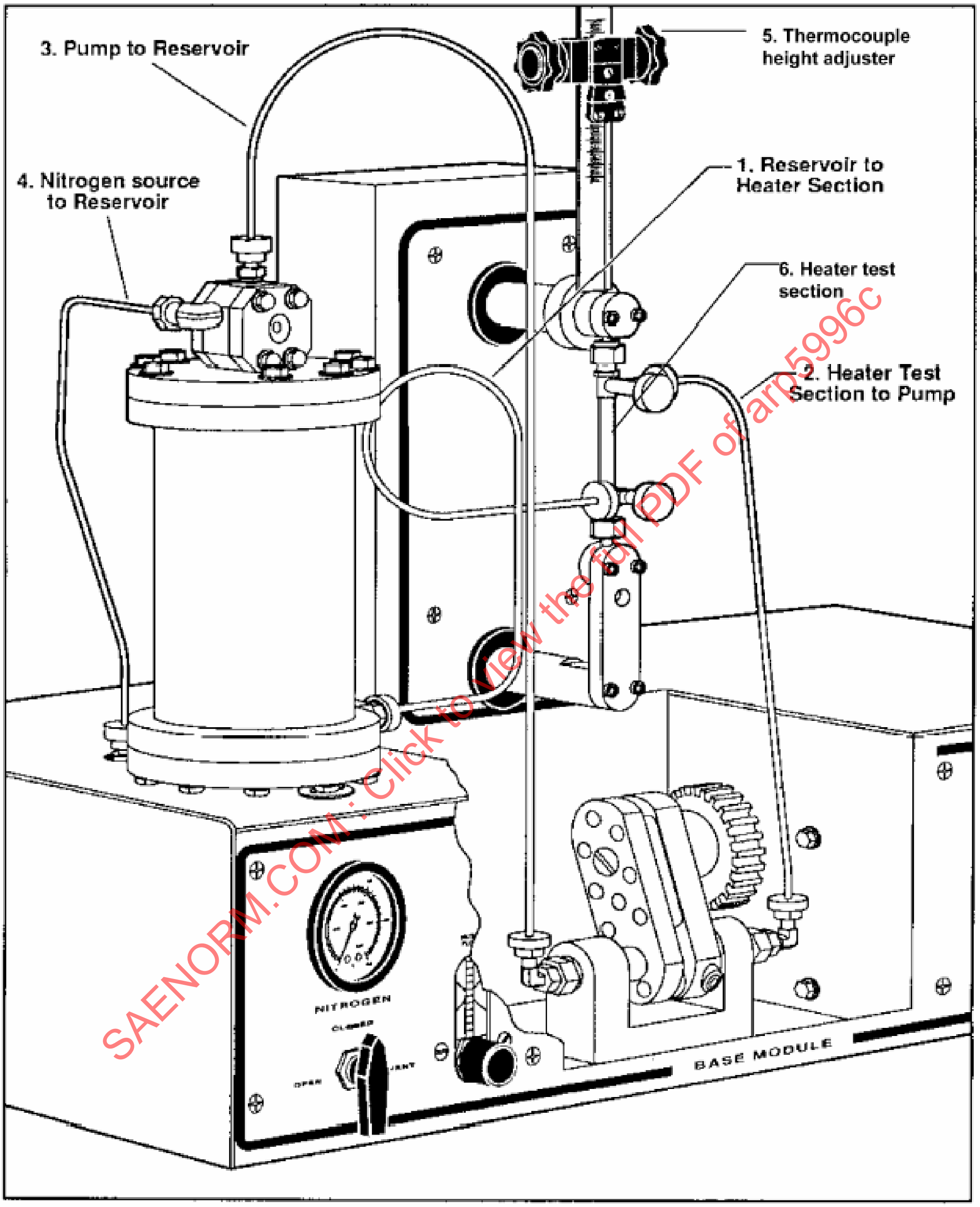
C.2.6 Rinse inside of boiling tube with pet spirit (6.3) and transfer into the filter unit to ensure all deposit has been recovered. With the vacuum still applied, carefully rinse around the inside of the filter funnel. Ensure all excess Pet Spirit has been drawn through before disconnecting the vacuum.

NOTE: If pet spirit alone is found to be ineffective at washing oil entrained in the filter then sequential washings with acetone (6.2) and pet spirit may be used.

C.2.7 Using forceps, carefully remove the filter and place into the covered Petri dish.

C.2.8 Follow procedure in C.2.1 to C.2.3 and repeat until two consecutive determinations of total deposit mass agree within ±0.05 mg or 10% of each other, whichever is the greater.

APPENDIX D - SCHEMATIC OF HLPS APPARATUS
(Reproduced with permission of Alcor)






NOTE: This method requires air to be used in place of nitrogen.

***Figure D1 - Schematic of HLPS apparatus***

APPENDIX E - PROGRAMMING A TEST ON A HLPS 400 INSTRUMENT

E.1 The process detailed in E.1 below is a basic method for programming a standard 20-hour test using the Alcor software provided with the HLPS-400 series rig.

E.1.1 Load the software package: ALCOR'S CE HLPS-400 Control Program.

E.1.2 From opening screen select tab "Setup a New Test".

E.1.3 Give the test a name; e.g., 20 hours at 375 C - 0.52 mls.

E.1.4 A screen titled "Setup Overall Test Configuration" will appear.

E.1.5 Change # Segments in Test to 4.

E.1.6 Change Tube Type to; STD - SS316.

E.1.7 The rest of the settings can be left as they are.

E.1.8 Click on the tab at the bottom of the screen "Done Overall Test Configuration Setup".

E.1.9 A screen titled "Test Segment Setup Menu" will appear. Test Seg# 01 will be displayed in a box.

E.1.10 Set the "Purpose of Segment" to Ramp and Soak.

E.1.11 Set the Ramp Time to 10 minutes.

E.1.12 Set the Soak Time to 30 minutes.

E.1.13 Set the Pump Speed to 0.5. (Required value is 0.52 but the program will only set to one decimal place.)

E.1.14 Click on the tab "Setup Bus Heater" and enter the following values:

***Table E1***

| Field Title | Value to be entered |
|---|---|
| 1 - Select Thermocouple | T3 |
| 2 - Use new Control Thermocouple | No |
| 3 - High Set Point Alarm | 25 |
| 4 - High Set Point Warning | 15 |
| 5 - High Set Point Control Limit | 10 |
| 6 - CONTROL TEMPERATURE SET POINT (for heater tube) | 375 |
| 7 - High Set Point Control Limit | -10 |
| 8 - High Set Point Warning | -15 |
| 9 - High Set Point Alarm | -25 |

E.1.15 Click on the tab “Setup System Pressure” and enter the following values:

***Table E2***

| Field Title | Value to be entered |
|---|---|
| 1 - System Pressure Used | Yes |
| 2 - High Set Point Alarm | 50 |
| 3 – High Set Point Warning | 25 |
| 4 - High Set Point Control Limit | 20 |
| 5 - SYSTEM PRESSURE SET POINT | 210 |
| 6 - High Set Point Control Limit | -20 |
| 7- High Set Point Warning | -25 |
| 8 - High Set Point Alarm | -50 |

E.1.16 Click on the tab “Setup Res#1 Heater” and enter the following values:



***Table E3***

| Field Title | Value to be entered |
|---|---|
| 1 - Reservoir Heater #1 Used | Yes |
| 2 - High Set Point Alarm | 40 |
| 3 - High Set Point Warning | 20 |
| 4 - High Set Point Control Limit | 15 |
| 5 - CONTROL TEMPERATURE SET POINT (for reservoir) | 150 |
| 6 - High Set Point Control Limit | -15 |
| 7 - High Set Point Warning | -20 |
| 8 - High Set Point Alarm | -40 |

E.1.17 Select the tab at the bottom of the screen “Done With Test Segment”.

E.1.18 A screen titled “Test Segment Setup Menu” will appear. Test Seg# 02 will be displayed in a box.

E.1.19 Set the “Purpose of Segment” to Soak. This Segment will be the initial temperature profiling segment.

E.1.20 Set the Soak Time to 10 minutes.

E.1.21 All other settings for this segment remain the same except for the following. In the tab “Setup Bus Heater” change the following:

***Table E4***

| Field Title | Value to be entered |
|---|---|
| 1 - Select Thermocouple | T2 |
| 2 - Use new Control Thermocouple | Yes |

E.1.22 Select the tab at the bottom of the screen “Done With Test Segment”.

E.1.23 Screen titled “Test Segment Setup Menu” will appear. Test Seg# 03 will be displayed in a box.

E.1.24 Set the “Purpose of Segment” to Soak. This Segment will be the main segment of the test.

E.1.25 The Soak Time to be entered at this point will depend upon the length of the test; e.g., 20, 40, or 100 hours. See table below for timings.

***Table E5***

| Length of Test | Minutes to be entered |
|---|---|
| 20 Hours | 1150 |
| 40 Hours | 2350 |
| 100 Hours | 5950 |

E.1.26 All other settings for this segment remain as per segment 01 except for the following. In the tab “Setup Bus Heater” change the following:

***Table E6***

| Field Title | Value to be entered |
|---|---|
| 1 - Select Thermocouple | T3 |
| 2 - Use new Control Thermocouple | Yes |



E.1.27 Select the tab at the bottom of the screen “Done With Test Segment”.

E.1.28 A screen titled “Test Segment Setup Menu” will appear. Test Seg# 04 will be displayed in a box.

E.1.29 Set the “Purpose of Segment” to Soak. This Segment will be the final temperature profiling segment.

E.1.30 Set the Soak Time to 10 minutes.

E.1.31 This segment should be programmed exactly the same as segment 02.

E.1.32 Select the tab at the bottom of the screen “Done With Test Segment”.

E.1.33 The program will now save the test and return to the original screen.

E.1.34 To load the required test select the tab “Load Test From Disk” and select the test from where you saved it, click open. This will communicate the test to the HLPS-400 series rig.

IMPORTANT NOTE: This method of programming may not be suited to each Lab running the HLPS 400 Series. For two alternative methods of programming see Section E.2.

E.2 ALTERNATIVE METHOD OF PROGRAMMING THE HLPS 400 RIG

E.2.1 The process detailed below is an alternative method for programming a standard 20-hour test using the Alcor software provided with the HLPS-400 series rig.