



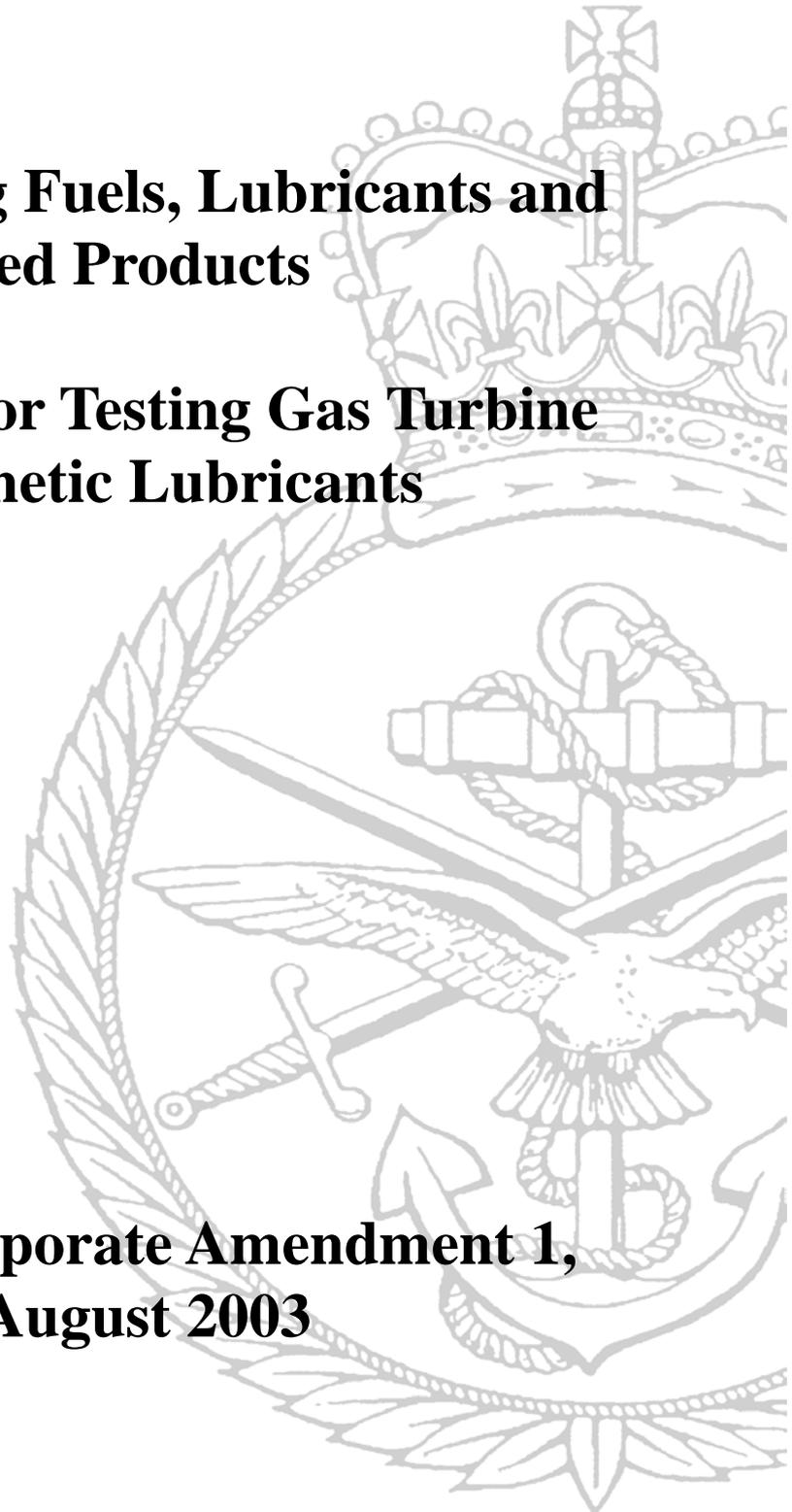
**Ministry of Defence  
Defence Standard 05-50(Part 61)**

**Issue 2 Publication Date 25 April 2003**

**Methods for Testing Fuels, Lubricants and  
Associated Products**

**Part 61: Methods for Testing Gas Turbine  
Engine Synthetic Lubricants**

**Reprinted to incorporate Amendment 1,  
dated 1 August 2003**



**AMENDMENT RECORD**

<b>Amd No</b>	<b>Date</b>	<b>Text Affected</b>	<b>Signature and Date</b>
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**REVISION NOTE**

This Defence Standard has been revised to incorporate Amendment 1, dated 1 August 2003.

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**PREFACE**

**Standards for Defence**

**Methods for Testing Gas Turbine Engine Synthetic Lubricants**

- a.** This Part of the Defence Standard covers the special test requirements of Defence Standard Gas Turbine Synthetic Lubricant Specifications for which no other suitable established methods exist. Those Defence Standard Test Methods which are based on Rolls-Royce originals have been established as joint Defence Standard/RR test methods and are designated as such individually.
- b.** The Technical Authority of this Defence Standard is the Aviation Fuels Technical Manager (AFTM), Defence Fuels Group (DFG), West Moors, Wimborne, Dorset, BH21 6QS.
- c.** This standard has been produced for the Standards Advisory Group (SAG), by the Aviation Propulsion Fuels and Lubricants Aviation Group (APFLAG), on behalf of the Defence Fuels and Lubricants Committee (DF&LC).
- d.** This standard has been agreed by the authorities concerned with its use and is intended to be used whenever relevant in all future designs, contracts, orders etc. and whenever practicable by amendment to those already in existence. If any difficulty arises which prevents application of the Defence Standard, UK Defence Standardization (DStan) shall be informed so that a remedy may be sought.
- e.** Any enquiries regarding this standard in relation to an invitation to tender or a contract in which it is incorporated are to be addressed to the responsible technical or supervising authority named in the invitation to tender or contract.
- f.** Compliance with this Defence Standard shall not in itself relieve any person from any legal obligations imposed upon them.
- g.** This standard has been devised solely for the use of the Ministry of Defence (MOD) and its contractors in the execution of contracts for the MOD. To the extent permitted by law, the MOD hereby excludes all liability whatsoever and howsoever arising (including, but without limitation, liability resulting from negligence) for any loss or damage however caused when the standard is used for any other purpose.

**TEXT**

**Standards for Defence**

**Methods for Testing Gas Turbine Engine Synthetic Lubricants**

**Method 1: Confined Heating Stability**

**1 SCOPE**

This method describes a procedure for the determination of the confined heat stability of synthetic gas turbine lubricants in terms of volatilisation loss, viscosity, acidity and insolubles increase brought about by a period of heating in a stainless steel, vented, vessel. This method is based on Rolls Royce lubricant approval methodology.

**2 WARNING**

The Ministry of Defence (MOD), like its contractors, is subject to both United Kingdom and European laws regarding Health and Safety at Work, without exemption. All Defence Standards either directly or indirectly invoke the use of processes and procedures that could be injurious to health if adequate precautions are not taken. Defence Standards or their use in no way absolves users from complying with statutory and legal requirements relating to Health and Safety at Work.

**3 RELATED DOCUMENTS**

**3.1** The publications referred to in the text of this standard are listed at **Annex C**. Publications are grouped and listed in alphanumeric order.

**3.2** Reference in this standard to any related document means in any invitation to tender or contract the edition and all amendments current at the date of such tender or contract unless a specific edition is indicated.

**3.3** In consideration of **3.2** above, users shall be fully aware of the issue and amendment status of all related documents, particularly when forming part of an invitation to tender or contract. Responsibility for the correct application of standards rests with users.

**3.4** DStan can advise regarding where related documents are obtained from. Requests for such information can be made to the DStan Helpdesk. How to contact the Helpdesk is shown on the outside rear cover of this standard.

## 4 OUTLINE OF METHOD

**4.1** The sample is heated under specified conditions in a vented cylindrical stainless steel vessel, in which it occupies approximately 75% of the volume. An air condenser fitted to the lid of the vessel restricts volatilisation and oxidation.

**4.2** A series of such tests varying either in respect to temperature or test duration are conducted. The deterioration in terms of the properties, referred to in the scope, are plotted against either temperature or time for the purpose of determining specific lubricant deterioration criteria as defined in **Appendix A**.

## 5 APPARATUS

**5.1** Heat Transfer apparatus as described in **Annex A, A.1** and **A.2**.

**5.2** Test vessel and air condenser as shown in **Annex B, Fig. 3**.

**5.3** 100 ml measuring cylinder BS 604

**5.4** Nickel spatula

**5.5** Wire brush

**5.6** Balance with an accuracy of 0.1 g

**5.7** Oven, capable of maintaining  $(190 \pm 5)^{\circ}\text{C}$

**5.8** Solid steel rod, suitable for cleaning the condenser tube

## 6 MATERIALS

**6.1** Dry film lubricant, graphite type. The use of PTFE tape to replace the dry film lubricant is permitted.

**6.2** Paper masking tape

**6.3** Acetone, laboratory reagent grade

**6.4** 40-60 mesh grit or bead

**6.5** Whatman 541 filter paper or 200 mesh filter

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**METHOD 1**

**7 TIME/TEMPERATURE GUIDELINES**

**7.1** The actual test programme will be determined by the evaluation objective but as a guideline tests are carried out in accordance with the following:

**7.1.1** Determine the temperature parameters as required. The parameters are defined in Appendix A. Put on tests at set times of 5, 25 and/or 192 hours at different temperatures (e.g. 120°C to 450°C at 10°C and 5°C intervals dependent on test duration and temperature) as required for the parameter being determined.

**7.1.2** The viscosity stability is measured by conducting 25 hour tests at different temperatures to establish a plot as shown in **Appendix A, Fig. 1.1**.

**7.1.3** From the graph produced the temperature ( $\theta$ ) for maximum viscosity decrease is obtained to the nearest 5°C by interpolation.

**7.1.4** Further tests are carried out for different test durations at  $\theta$ °C sufficient to establish a plot as shown in **Appendix A, Fig. 1.2**.

**8 PREPARATION OF APPARATUS**

**8.1** Cover the threads on both the test vessel and lid with the masking tape.

**8.2** Using a 40-60 mesh grit or bead wet grit blast or bead blast the test vessel and lid. Ensure the inside of the condenser is also grit or bead blasted.

**8.3** Remove the masking tape and brush the threads clean with the wire brush.

**8.4** Cover the threads with PTFE tape or alternatively apply a single coat of dry film lubricant and stove for one hour at  $(190 \pm 5)$ °C in a suitable air circulating oven.

**NOTE:** In vessels which have been previously used, and upon which the dry film lubricant is visually intact, reapplication of dry film lubricant is unnecessary. In vessels or lids where the dry film lubricant is not visually intact, remove the coating by vapour blasting, brush the threads clean with the wire brush and repeat **8.4**.

**9 PROCEDURE**

**9.1** Weigh the prepared assembly to the nearest 0.1 g.

**9.2** Introduce 85 ml of the oil sample from a BS604, 100 ml measuring cylinder and allow a 30 second draining period.

**9.3** Replace the lid and condenser and screw the lid firmly into position.

**9.4** Reweigh the whole assembly including test oil to the nearest 0.1 g.

**9.5** Place assembly into the heat transfer test block.

- 9.6** Inspect the apparatus frequently and ensure that the condenser has not become blocked by oil degradation products, by passing the solid steel rod through the condenser every 24 hours, (ensure that there is no loss to the system and that the rod does not enter the oil).
- 9.7** During the test period check the temperature daily to monitor that no fluctuation has taken place.
- 9.8** Reject tests where the temperature has fluctuated by more than  $\pm 2.5^{\circ}\text{C}$ .
- 9.9** Remove the test assembly from the heating block at the end of the prescribed test period and quench in cold water to room temperature ( $20 \pm 5^{\circ}\text{C}$ ). Ensure the level of the water used to quench the vessel is below the level of the screw threads to prevent ingress of water.
- 9.10** Examine the assembly for evidence of oil leakage past the threaded portion, and if leakage has occurred, reject the test.
- 9.11** Ensure the exterior of the assembly is clean.
- 9.12** Reweigh the assembly and contents to the nearest 0.1 g before removing the lid and attached condenser.
- 9.13** Thoroughly scrape the walls of the vessel and the lid free of deposit and disperse the deposit in the oil. This can be best achieved with the aid of the nickel spatula.
- 9.14** Remove a representative sample of the oil and measure the deterioration in accordance with the following:
- (i) Toluene insolubles content, Method 12 and (ii) Total Acidity, SAE ARP5088.
- 9.15** Remove a representative sample of the oil and filter through Whatman 541 filter paper or 200 mesh filter before determining the viscosity at  $100^{\circ}\text{C}$  in accordance with ASTM D445/IP 71 (BS 2000: Part 71).
- 9.16** Carry out sufficient tests at various duration's and / or temperatures to enable the required temperature parameters (as defined in **Appendix A**) to be determined.
- 9.17** Set heating block at  $\theta$  temperature (**Appendix A, Fig. 1.1**) and carry out tests of varying duration up to and including 192 hours.
- 9.18** Determine the kinematic viscosity at  $100^{\circ}\text{C}$  as described in **9.15**.
- 9.19** Determine new oil values for insolubles content, acidity and viscosity at  $100^{\circ}\text{C}$  using the same test methods as **9.14** and **9.15**.

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**METHOD 1**

**10 CALCULATION**

**10.1** Calculate the volatilisation loss in accordance with the following formula.

$$\text{Volatilisation loss, percent weight} = \frac{(W2 - W3)}{(W2 - W1)} \times 100$$

Where      W1 = mass of assembly  
              W2 = mass of assembly + sample before testing  
              W3 = mass of assembly + sample after testing

**10.2** The acidity change shall be calculated as the difference between the test sample and the new oil value.

**10.3** The kinematic viscosity (KV) change shall be calculated as a percentage change of the new oil value as follows:

$$\text{Viscosity change (\% at 100 °C)} = \left( \frac{\text{KV sample}}{\text{KV new}} - 1 \right) \times 100$$

**10.4** The viscosity stability shall be calculated by using the viscosity changes derived from the 25 hour duration test to construct a graph of the type depicted in **Appendix A, Fig. 1.2**.

**10.5** Use the graph to determine  $\theta$  °C.

**10.6** The toluene insolubles increase shall be calculated as the difference between the test sample and new oil values.

NOTE: **9.14** and **10.5** are inapplicable where no decrease in viscosity is recorded.

**11 REPORTING**

**11.1** Data is normally presented in graphical form. There are many curve fitting software packages available to do this.

## 11.2 Deterioration Parameters

11.2.1 Where it is required to quote the deterioration brought about by a particular time/temperature condition, the result shall be quoted to the following precision:

Volatilisation loss	-	nearest 1 per cent
Viscosity increase	-	nearest 1 per cent
Acidity increase	-	nearest 0.1 mg KOH g <sup>-1</sup>
Insolubles increase	-	nearest 0.1 mg g <sup>-1</sup>

## 11.3 Temperature Parameters

11.3.1 Determine the temperature parameters, as defined in appendix A, by interpolation from the best line of fit plot obtained when the deterioration parameters are plotted against temperature for constant time.

NOTE: Some oils possess as many as three S<sup>D</sup> temperatures as indicated in **Appendix A, Fig. 1.3** and care must be taken to ensure that only the S<sub>3</sub><sup>D</sup> temperature is selected for the assessment level. In order to confirm that the S<sub>3</sub><sup>D</sup> temperature has been obtained further tests at +5°C and -5°C should be conducted.

11.3.2 Report the temperature parameters to the nearest 5°C.

## 11.4 Viscosity Stability

11.4.1 The viscosity stability criteria as defined in **Appendix A** shall be obtained from the graphs produced in accordance with **Appendix A, Figs. 1.1 and 1.2**.

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METHOD 1**

**12 PRECISION**

A formal precision exercise has not been carried out. However, prior experience has shown that the following precision levels can be achieved:

**Table A Precision Levels**

PROPERTY			REPEATABILITY	REPRODUCIBILITY
Temperature Parameters (°C)		A <sup>D</sup>	±7½	±10
		B <sup>D</sup>	±5	±7½
		E <sup>D</sup>	±5	±7½
		V <sup>D</sup>	±5	±7½
		Z <sup>D</sup>	±5	±7½
Viscosity Stability	%	v	±2	±5
	°C	θ <sub>v</sub>	±5	±7½
	hrs	D <sub>v</sub>	Up to 5 hrs ± ¼	±¼
			5 to 25 hrs ± ½	±1
			25 to 100 hrs ± 2	±5

APPENDIX A

DEGRADATION MODES DEFINITION OF TEST PARAMETERS

**A.1 Temperature Parameters**

- A.1.1** E<sup>D</sup> temperature - the lowest constant temperature, for a test duration of D hours, at which the volatilisation loss is 15 % by weight.
- A.1.2** A<sup>D</sup> temperature - the lowest constant temperature, for a test duration of D hours, at which the total acidity number increases by 4.0 mg KOH g<sup>-1</sup>.
- A.1.3** B<sup>D</sup> temperature - the lowest constant temperature for a test duration of D hours, at which the toluene insoluble matter increases by 0.05 % by weight.
- A.1.4** V<sup>D</sup> temperature - the lowest constant temperature, for a test duration of D hours, at which the viscosity, measured at 100°C increases by 15 %.
- A.1.5** S<sup>D</sup> temperature - the highest constant temperature for a test duration of D hours at which the toluene insoluble matter increases by 0.50 % by weight.
- A.1.6** Z<sup>D</sup> temperature - the lowest constant temperature, for a test duration of D hours at which the residue is visually solid.
- A.1.7** VS Confined heating - the minimum change in 100°C kinematic viscosity (%) that can be achieved under optimum conditions of test duration and temperature.
- A.1.8** θ<sub>v</sub> temperature - the temperature (°C) at which the change in viscosity attains the minimum value (%).
- A.1.9** D<sub>v</sub> - the duration (hours) at which the change in viscosity attains a minimum value (%).

A.2 Figures

Fig. 1.1 Determination of  $\theta_v$

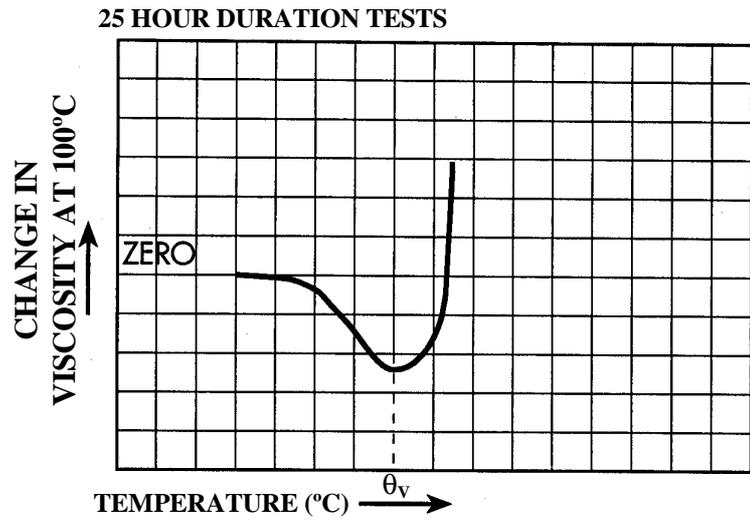


Fig. 1.2 Determination of  $D_v$  &  $V_S$

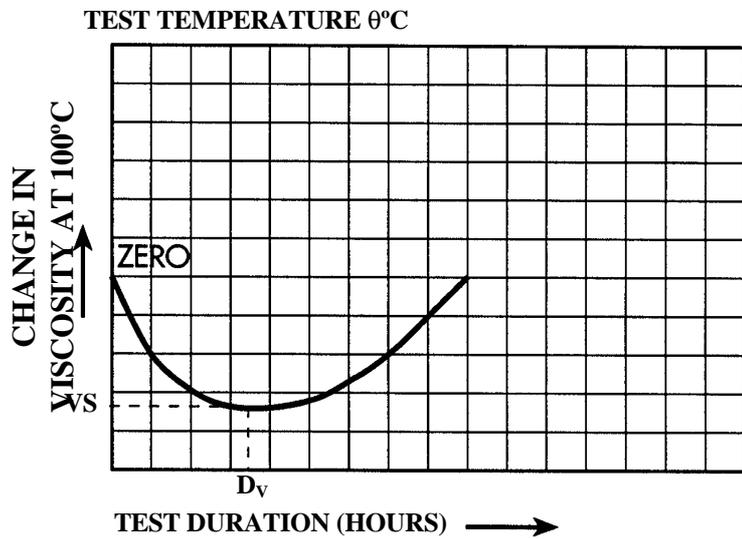
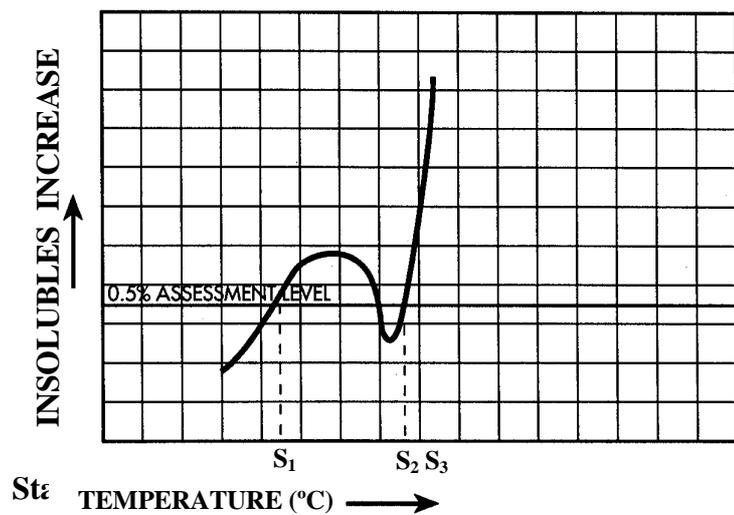


Fig. 1.3 Determination of S Temp



## Standards for Defence

### Methods for Testing Gas Turbine Engine Synthetic Lubricants

#### Method 3 : Corrosivity

## 1 SCOPE

**1.1** This method is intended for the determination of the corrosivity of synthetic gas turbine lubricants towards various materials constituting typical gas turbine engine lubricant systems.

**1.2** The method may be used to evaluate the corrosive effect that various forms of deterioration impart to lubricants. This method is based on Rolls-Royce lubricant approval methodology.

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**METHOD 3**

**4 OUTLINE OF METHOD**

**4.1** Metal specimens of standard dimensions are assembled on a stainless steel rod and completely immersed in the test lubricant under specified conditions of time and temperature.

**4.2** The assessment of the degree of corrosion is based on the change in mass and/or changes in appearance of the test specimens.

**4.3** Six test procedures are described which differ in respect to the materials of the test specimens used, the test temperature and duration. Procedures **1** and **2** are intended for all grades of lubricant and cover the main structural materials of the engine oil system and plating materials respectively. Procedures **3**, **4**, **5** and **6** are intended for oils designed for use in higher temperature environments; that is, those oils possessing a B<sup>192</sup> temperature (as defined in **Method 1, Appendix A**) greater than 200°C. Procedure **3** covers constructional materials, **4** covers plating materials, **5** covers unprotected magnesium alloy casing material, and **6** covers protected magnesium alloy casing material. .

**5 APPARATUS**

**5.1** Boiling tube.

**5.1.1** Procedure **1, 2**

As shown in **Annex B, Fig. 6** but fitted with a cork stopper in which there is a small vent in place of the condenser.

**5.1.2** Procedures **3, 4, 5, 6**

As shown in **Annex B, Fig. 6**.

**5.2** Condenser (Procedures **3, 4, 5, 6**)

As shown in annex **B, Fig. 6**.

**5.3** Test specimen assembly (all procedures)

Stainless steel stem and spacing collars as defined in **Annex B, Fig. 7**, and suitable stainless steel spring washers and locking nut.

#### 5.4 Test Specimens

The specimens shall be produced in the manner described in appendix A and shall conform dimensionally to **Annex B, Fig. 7**. The type and number of specimens required for each procedure is indicated by **Annex B, Figs. 8 and 9**. Test specimens are standardized and supplied by Rolls-Royce plc, PO Box 31, Derby, DE2 8BJ, England or QinetiQ FLC, Aviation Lubricants, Building 442, Cody Technology Park, Farnborough, Hants., GU14 0LX..

5.5 Heat Transfer apparatus as described in **Annex A, A.1 and A.2**.

5.6 Balance, capable of determining mass up to a total of 20 g to 5 decimal places.

5.7 Magnifying glass or microscope, x10 magnification.

5.8 Brush, short bristled, stiff (must be compatible with the tri-solvent)

### 6 MATERIALS

6.1 Tri-solvent (equal quantities of acetone, propan-2-ol and toluene; all GPR grade)

6.2 Silicon carbide paper, grade 00 and coarser grades (150, 240 and 600 are suggested).

6.3 Steel wool, size 00 (degreased).

6.4 Petroleum ether, analytical reagent grade, boiling range (100 - 120) °C and boiling range (40 - 60) °C.

6.5 Lint-free cloth.

6.6 Toluene, analytical reagent grade.

6.7 Acetone, analytical reagent grade.

6.8 Glassware cleaning fluid. Any suitable laboratory glass cleaning fluid may be used providing it removes all evidence of staining and deposits from previous tests.

6.9 Distilled/Deionised Water, ISO 3696 grade 3

### 7 TEST CONDITIONS

7.1 The following conditions of time and temperature have been standardised (**Annex C, Figs. 10 and 11** indicates the specimen materials used in each Procedure):

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METHOD 3**

**Table B Time and Temperature Conditions**

PROCEDURE	TEST SPECIMENS	DURATION (h)	TEMP (°C)	LUBRICANT TYPE
<b>1</b>	Constructional	192 ± 0.5	150	ALL
<b>2</b>	Plating			
<b>3</b>	Constructional	192 ± 0.5	200	Increased thermal stability oils possessing a B <sup>192</sup> temperature greater than 200 °C (see Method 1 appendix A)
<b>4</b>	Plating			
<b>5</b>	Magnesium alloy			
<b>6</b>	Protected magnesium alloy	192 ± 0.5	200	

**8 PROCEDURE**

**8.1** Chemically clean the glass boiling tube and condenser as follows:

**8.1.1** Immerse in the glassware cleaning fluid in accordance with laboratory procedures and safety instructions.

**8.1.2** Thoroughly rinse glassware in distilled/deionised water and dry in an air-circulating oven.

**8.2** Preparation of test specimens

**8.2.1** Annex B, Figs. 8 and 9, indicates those test specimens required for each procedure (1-6).

**8.2.2** With the exception of lead, protected magnesium, and soldered copper, prepare the specimens by polishing both faces and the outer edge with suitable grades of silicon carbide paper in order to remove any surface blemishes that may exist. Wet each grade of paper used in this process with the petroleum ether (boiling range 100 - 120 °C) and remove all marks made by the previous grade. This may most conveniently be achieved by turning the specimen by 90°, through an axis normal to the paper between each grade of paper used. Finish each specimen with 00 grade paper. The polishing process may most easily be carried out by clamping the abrasive paper sheet on a flat surface and rubbing the specimen against the paper through a single plane. The specimens should be held by the edge only. Conduct the final polish with a sheet of grade 00 paper reserved only for that particular material.

**8.2.3** Clean and debur the centre hole in each specimen, where necessary, with the aid either of a fine stone mounted on a revolving shaft, or with grade 0 paper wrapped around a rod of suitable size.

**8.2.4** Immediately after polishing, rinse the specimens in the petroleum ether (boiling range 100 - 120 °C) and immerse in the petroleum ether (boiling range 40 - 60 °C) until a complete set of specimens have been prepared.

**8.2.5** Prepare the lead and soldered copper specimens by rubbing with the steel wool, to produce a clean surface free from blemishes. Wash each specimen and store in a similar manner to the above specimens.

**8.2.6** Prepare the protected magnesium alloy specimens by careful wiping with a clean, lint-free cloth before weighing. Do not store these specimens in solvent.

**8.2.7** Repeated Use of Test Specimens

**8.2.7.1** The protected magnesium alloy specimens may be reused provided that the protective coating is renewed on each occasion.

**8.2.7.2** All the remaining specimens may be reused providing all evidence of previous corrosion is removed and that, in so doing, the surface condition of the specimen remains unaltered. Therefore, in the case of the nickel-chromium case-hardened steel specimen, do not use the specimen after the removal of a total depth of 0.25 mm (0.01 in) of metal.

**8.3** Procedures 1 and 2

**8.3.1** Remove the test specimens from the petroleum ether (boiling range 40 - 60 °C), rinse in the acetone, and wipe each carefully with a clean, dry lint-free cloth. Determine the mass of each specimen to the nearest 0.01 mg, taking care to avoid direct contact with the fingers. Record this mass,  $m_0$ .

**8.3.2** Prepare the test assembly support rod by cleaning with suitable grade steel wool to remove extraneous deposits from previous tests. Wash the rod, spring washer, locking nut and sufficient stainless steel spacers in the acetone and air dry.

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**METHOD 3**

**8.3.3** Place one spacer on the support rod followed by the first test specimen. Add another spacer, followed by the second test specimen, and so forth until all the specimens are assembled in the order indicated in **Annex B, Figs. 8 and 9**. Position sufficient spacers after the last specimen to completely cover the unthreaded section of the support rod. Place the spring washer on the rod and finally the locking nut, which should be screwed finger-tight.

**8.3.4** Position the cork stopper at the top of the support rod and insert the complete assembly into the boiling tube which contains  $(50 \pm 1)$  ml of the test lubricant. Position the assembly such that it is approximately 6 mm (0.25 in) clear of the bottom of the boiling tube. Insert the apparatus in to the heat transfer block at the prescribed temperature for the duration of the test.

**8.3.5** At 24 hour intervals, agitate the test lubricant by raising and lowering the support rod three times, ensuring that on each occasion all of the test specimens are lifted clear of the lubricant.

**8.3.6** On completion of the test, remove and dismantle the test assembly.

**8.3.7** Wash each specimen individually by successive 2 minute immersions in boiling toluene, and boiling acetone. Wipe each specimen with a clean, dry lint-free cloth to remove any loose powdery deposit or flakes. Determine the mass of each specimen to the nearest 0.01 mg. Record this mass,  $m_1$ .

**8.3.8** Note and record the appearance of the specimens under a magnification of x10 in respect to the following and with reference to **9.3**.

**8.3.8.1** Presence and nature of deposit.

**8.3.8.2** Extent and degree of discoloration of the metal.

**8.3.8.3** Extent and type of attack (ie, pitting and/or etching).

**8.4** Procedure **3**

**8.4.1** Carry out the procedure as per **8.3.1** to **8.3.3**, assembling the specimens in the order indicated in **Annex B, Figs. 8** and/or **9**.

**8.4.2** Attach a length of 33 SWG stainless steel wire by means of the hole at the top of the support rod. Insert the assembly into the boiling tube containing  $(50 \pm 1)$  ml of the test lubricant. Fit the condenser with the wire passing through its centre. Raise the test assembly approximately 6mm (0.25 in) clear of the bottom of the boiling tube and support this by crimping the wire round the top lip of the condenser. Insert the apparatus into the heat transfer block at the prescribed temperature for the duration of the test.

**8.4.3** Proceed as per **8.3.5** to **8.3.7** except that the test assembly is lifted by means of the stainless steel wire.

**8.4.4** Some specimens may have tenacious deposits remaining. If this is the case, clean the specimens (except the protected magnesium alloy) by immersion in the tri-solvent, followed by scrubbing with the brush until all deposits have been removed. Rinse with acetone and dry in a current of air.

**8.4.5** After cleaning, wipe each specimen with a clean, dry, lint-free cloth and re-determine the mass to the nearest 0.01 mg. Record this mass,  $m_2$ .

**8.4.6** Note and record the appearance of the specimens, under a magnification of  $\times 10$ , in respect to the following:-

**8.4.6.1** Presence and nature of any remaining deposit.

**8.4.6.2** Extent and degree of discoloration of the metal.

**8.4.6.3** Extent and type of attack (ie, pitting and/or etching).

**8.5** Procedures **4**, **5** and **6**

Carry out the procedure as per **8.4.1** to **8.4.5**

## **9 CALCULATION AND REPORTING**

**9.1** Calculation of specimen mass change

**9.1.1** Evaluate the metal mass change in the manner prescribed below. Where more than one specimen of the same material are employed in a procedure, calculate the arithmetic mean of each individual result.

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**9.1.2** Nickel-chromium case hardened steel, high speed steel and high carbon chromium steel.

These specimens are assumed to possess a surface area of 9 cm<sup>2</sup>. Therefore, calculate the specimen mass change per unit area as follows:

$$M = \frac{m_o - m_n}{0.009}$$

where;

M = change in mass of specimen per unit area (mg cm<sup>-2</sup>),

m<sub>o</sub> = mass of specimen prior to test (g),

m<sub>n</sub> = mass of specimen after test (g).

n = 1 where there are no tenacious deposits or

2 where further cleaning is required to remove tenacious deposits.

**9.1.3** Other Specimens

Specimens other than the nickel-chromium case hardened steel, high speed steel and high carbon chromium steel are assumed to possess a surface area of 7 cm<sup>2</sup>. Therefore, calculate the specimen mass change per unit area as follows:

$$M = \frac{m_o - m_n}{0.007}$$

where;

M = change in mass of specimen per unit area (mg cm<sup>-2</sup>),

m<sub>o</sub> = mass of specimen prior to test (g),

m<sub>n</sub> = mass of specimen after test (g).

n = 1 where there are no tenacious deposits or

2 where further cleaning is required to remove tenacious deposits.

**9.1.4** It should be noted that a negative value of M is indicative of loss of mass during the test, and vice versa.

**9.2** Report the specimen mass change to the nearest  $0.1 \text{ mg cm}^{-2}$ .

**9.3** Reporting specimen appearance

**9.3.1** Where required report the appearance using the following abbreviated classifications, localities and severities:

Classification;	D	discoloration
	E	etching
	P	pitting
Locality	l	localised
	g	generalised
Severity	(s)	slight
	(m)	moderate
	(x)	excessive

**9.3.2** Locality

Localised corrosion applies to 10 per cent or less of the specimen surface.

**9.3.3** Severity

**9.3.2.1** Slight corrosion applies to effects that are barely visible to the naked eye.

**9.3.2.2** Moderate corrosion applies to effects which are visible to the naked eye but which do not possess sufficient severity to remove or interrupt the lines produced upon the specimen by the final polish.

**9.3.2.3** Excessive corrosion applies to effects that remove or interrupt the lines produced upon the specimen by the final polish.

**9.3.2.4** In the case of discoloration, pale straw discoloration is regarded as slight, straw through to light brown is moderate, and brown through to black is excessive. Similar remarks apply to other colours such as blue or grey.

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METHOD 3**

**10 PRECISION**

**10.1** The precision of this test method has not been determined. However, it is recommended that in the case of the mass change, the accuracy given in **9.2** should be used. That is, for two separate mass change results,  $M_1$  and  $M_2$ , obtained using the same test specimen and test lubricant, the results should be considered suspect if they do not comply with the following:

$$(M_1 - 0.05) \leq M_2 < (M_1 + 0.05).$$

**10.2** In the case of specimen appearance, it is suggested that two separate results for the same test specimen and for the same test lubricant be considered suspect if they possess different classifications of surface appearance as defined in **9.3.1**.

**APPENDIX A**

**TABLE C TEST SPECIMEN MATERIALS AND METHOD OF MANUFACTURE**

TEST SPECIMEN	SPECIFICATION	PROCEDURE	METHOD OF MANUFACTURE
Aluminium alloy	MSRR8009	1, 3	Parted off from bar fully heat treated to MSRR8009
Brass	BS EN 12167	1	Parted off from bar, annealed 5 mins at 400 °C, air cooled
Chromated magnesium alloy	MSRR8013/ JDS/4511.01	6	Parted off from bar, fully heat treated to MSRR8013, chromated to JDS4511.01
Chromated magnesium alloy, phenolic resin coated	MSRR8013/ JDS/4511.01	6	Parted off from bar, fully heat treated to MSRR8013, chromated to JDS4511.01, phenolic resin coated to MSRR632
Copper	BS EN 13601	1, 2, 3, 4	Parted off from bar, annealed 5 mins at 400 °C, air cooled
Copper-nickel-silicon alloy	MSRR8501	3, 6	Parted off from bar, no heat treatment
High carbon chromium steel	RR MS 210A	3	Parted off from bar, fully heat treated to RR MS 210A
High speed steel	RR MS 258	3	Parted off from bar, fully heat treated to RR MS 258
Lead	99 % pure	1	Parted off from bar

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**METHOD 3**

TEST SPECIMEN	SPECIFICATION	PROCEDURE	METHOD OF MANUFACTURE
Lead bronze	MSRR9504	3	Cast into bar form, water quenched, parted off, no heat treatment
Leaded brass	BS EN 12167	1, 3	Parted off from bar, no heat treatment
Low alloy steel	MSRR6001	3, 6	Non-nitrided, parted off from bar, fully heat treated to MSRR6001
Magnesium alloy	MSRR8013	1, 5	Parted off from bar, fully heat treated to MSRR8013
Mild steel	MSRR6050	1, 3	Parted off from bar, no heat treatment
Nickel	99 % pure	2, 4	Parted off from bar
Nickel alloy	MSRR7137	3	Parted off from bar, fully heat treated to MSRR7137
Nickel-chromium case-hardened steel	MSRR6009	3	Parted off from bar, carburised to RPS371, delay quenched/frozen to RPS360 tempered at 150 °C
Silver	99 % pure	2, 4	Parted off from bar
Soldered copper	BS EN 13601/ BS EN 29453 grade C	1	See Appendix B
Tin	99 % pure	2	Parted off from bar

## APPENDIX B

### PREPARATION OF SOLDERED COPPER TEST SPECIMENS

- B.1** Copper test specimens shall be used for the purpose and shall have the centre hole opened up to 3.5 mm (0.14 ins) diameter. The specimens shall then be individually treated in accordance with the following sequence.
- B.2** Support the specimen by threading it onto a borosilicate glass rod having a slightly bulged end.
- B.3** Immerse the specimen completely into a bath of flux consisting of a saturated solution of zinc chloride in hydrochloric acid of which Baker's Fluid is a typical commercial example.
- B.4** Transfer to a bath containing not less than 1 kg of BS EN 29453 Grade C solder maintained at (240 -260) °C.
- B.5** Remove after approximately 5 seconds and, if a continuous uniform layer of solder has formed, cool and rinse thoroughly in cold running water and dry by immersion in hot water. Repeat operations **B.2** and **B.3** if the layer is not completely formed or is particularly uneven, and then wash.
- B.6** Renew the solder bath after treatment of 50 specimens.
- B.7** In the case of used specimens, their re-use shall only be permitted providing that they are stripped and resoldered.

**Standards for Defence**

**Methods for Testing Gas Turbine Engine Synthetic Lubricants**

**Method 6 : Hydrolytic Stability**

**1 SCOPE**

This method describes a procedure for determining the stability of synthetic gas turbine lubricants in contact with water at 90°C, in terms of a specified increase in acidity. This method is based on Rolls Royce lubricant approval methodology.

**2 WARNING**

The Ministry of Defence (MOD), like its contractors, is subject to both United Kingdom and European laws regarding Health and Safety at Work, without exemption. All Defence Standards either directly or indirectly invoke the use of processes and procedures that could be injurious to health if adequate precautions are not taken. Defence Standards or their use in no way absolves users from complying with statutory and legal requirements relating to Health and Safety at Work.

**3 RELATED DOCUMENTS**

**3.1** The publications referred to in the text of this standard are listed at **Annex C**. Publications are grouped and listed in alphanumeric order.

**3.2** Reference in this standard to any related document means in any invitation to tender or contract the edition and all amendments current at the date of such tender or contract unless a specific edition is indicated.

**3.3** In consideration of **3.2** above, users shall be fully aware of the issue and amendment status of all related documents, particularly when forming part of an invitation to tender or contract. Responsibility for the correct application of standards rests with users.

**3.4** DStan can advise regarding where related documents are obtained from. Requests for such information can be made to the DStan Helpdesk. How to contact the Helpdesk is shown on the outside rear cover of this standard.

## 4 OUTLINE OF METHOD

**4.1** A sample of oil, with excess water, is maintained at a temperature of 90°C. Samples are taken periodically so as to determine the time taken for the total acidity of the oil to increase by the equivalent of 1.5 mg KOH g<sup>-1</sup>. The time taken to achieve this is known as D<sub>1.5</sub><sup>90</sup> hours. The condition of the oil at this acidity level is examined in respect to water hold up and water solubility after a period of standing at room temperature.

## 5 APPARATUS

**5.1** Reaction Flask: Conical flask, 500 ml, alkali-resistant glass fitted with B24 glass stopper, **Annex B, Fig. 4**.

**5.2** Absorber Unit: Heat resistant glass with two U tube traps connected by a B7 ground glass joint as shown in **Annex B, Fig. 4**. The capacity of each bulb is (5 ± 1) ml.

**5.3** Conical Separating Funnel, 500 ml alkali-resistant glass.

**5.4** Fan-assisted oven, Capable of maintaining a temperature of (90 ± 2.5) °C.

**5.5** Measuring cylinders, BS604 (2 ml, 25 ml and 250 ml).

**5.6** Pipette, 15 ml.

## 6 MATERIALS

**6.1** Filter Paper, Whatman, grade 41.

**6.2** Distilled/deionized water.

**6.3** Acetone, Laboratory reagent grade.

**6.4** Glassware cleaning fluid. Any suitable non-surfactant laboratory glass cleaning fluid may be used providing it removes all evidence of staining and deposits from previous tests.

**6.5** Distilled/Deionised Water, ISO 3696 grade 3

## 7 PREPARATION OF APPARATUS

**7.1** Chemically clean the flask and absorber unit as follows:

**7.1.1** Immerse in the glassware cleaning fluid in accordance with laboratory procedures and safety instructions.

**7.1.2** Thoroughly rinse glassware in distilled/deionised water and dry in an air-circulating oven.

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**8 PROCEDURE**

- 8.1** Using the 2 ml measuring cylinder, place 2 ml distilled/deionized water in each bulb of the absorber unit.
- 8.2** Using the measuring cylinders, introduce  $(250 \pm 5)$  ml of sample and  $(25 \pm 1)$  ml of water into the reaction flask. Insert the stopper and shake the contents vigorously by hand for 30 seconds. Immediately remove the stopper and replace with the absorber unit.
- 8.3** Place the assembly in the oven maintained at  $90^{\circ}\text{C}$ .
- 8.4** At intervals of 24 hours remove the assembly from the oven and draw off a 15 ml sample by pipette into a 50 ml beaker. Shake the remaining sample in the reaction flask as in clause **8.2**, top up the distilled water in the absorber unit, reassemble and return to the oven.
- 8.5** Where the rate of acidity increase is high, the sampling period should be reduced to allow interpolation of the time to achieve an acidity increase of  $1.5 \text{ mg KOH.g}^{-1}$ . All other conditions of test remain the same.
- 8.6** Dry the 15 ml sample by passing through successive dry filter papers until it becomes clear.
- 8.7** Determine total acidity in accordance with SAE ARP 5088.
- 8.8** Determine the total acidity of the new oil by the same method.
- 8.9** Terminate the test when the total acidity has increased by more than  $1.5 \text{ mg KOH g}^{-1}$  from the original oil.
- 8.10** Shake the contents of the flask as **8.2** and then transfer to a 500 ml capacity conical separating funnel.
- 8.11** Stand undisturbed for 24 hours at room temperature.
- 8.12** Draw off the separated water together with approximately 5 ml of the oil.
- 8.13** Record the appearance of the remaining oil in terms descriptive of colour and cloudiness.
- 8.14** Take a representative 50 ml sample of the oil and determine the total water content in accordance with IP74 / ASTM D95 or by Karl Fischer titration.
- 8.15** Filter the remaining oil through successive layers of the filter paper, previously wetted with the original oil, until a clear filtrate is obtained.
- 8.16** Take a representative 50 ml sample of the filtrate and determine the dissolved water content in accordance with IP74 / ASTM D95 or by Karl Fischer titration.

## 9 CALCULATION

9.1 On a graph of test duration against acidity increase, interpolate the time in hours to achieve an acidity increase of  $1.5 \text{ mg KOH g}^{-1}$ . Record this time as  $D_{1.5}^{90}$  hrs.

## 10 REPORTING

10.1 Report  $D_{1.5}^{90}$  to the nearest hour below 50 hours and to the nearest 5 hours above 50 hours.

10.2 Report the following:

10.2.1 The appearance of the oil after standing 24 hours following the termination of the test.

10.2.2 The free water hold-up, per cent as the difference between the total and the dissolved water contents following the 24 hours standing period to two significant figures.

10.2.3 The dissolved water content.

## 11 PRECISION

A formal precision exercise has not been carried out. However, prior experience has shown that the following precision levels can be achieved:

- ± 2 hours below 50 hours
- ± 5 hours above 50 and below 100 hours
- ± 10 hours above 100 hours

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METHOD 9**

**Standards for Defence**

**Methods for Testing Gas Turbine Engine Synthetic Lubricants**

**Method 9: Resistance to Oxidation and Thermal Decomposition**

**1 SCOPE**

This method describes procedures for the determination of the oxidation and thermal stability of synthetic gas turbine lubricants in terms of volatilisation loss and increases in viscosity, acidity and insolubles. This is brought about by a period of heating during which air is bubbled through the sample. This method is based on Rolls Royce lubricant approval methodology.

**2 WARNING**

The Ministry of Defence (MOD), like its contractors, is subject to both United Kingdom and European laws regarding Health and Safety at Work, without exemption. All Defence Standards either directly or indirectly invoke the use of processes and procedures that could be injurious to health if adequate precautions are not taken. Defence Standards or their use in no way absolves users from complying with statutory and legal requirements relating to Health and Safety at Work.

**3 RELATED DOCUMENTS**

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## 4 OUTLINE OF METHOD

**4.1** A weighed volume of lubricant contained in an all glass boiling tube and blowing assembly is inserted in a special heat transfer test apparatus. Air is bubbled through the sample, which is maintained at constant temperature for a specified duration. After measurement of solidus formation and volatilisation loss, the sample is restored to its original weight by addition of fresh lubricant and then examined in respect to viscosity, acidity and insolubles content.

**4.2** A series of such tests varying either in respect to temperature or test duration are conducted and the deterioration in terms of the properties, referred to at **4.1**, are plotted against either temperature or time for the purpose of determining specific lubricant deterioration criteria. These are defined in **Appendix A** to this method.

## 5 APPARATUS

**5.1** Heat Transfer apparatus as described in **Annex A, A.1** and **A.2**.

**5.2** Glassware as depicted in **Annex B, Fig. 2**.

**5.2.1** When selecting the boiling tube and air blowing tube ensure that when fitted together the internal clearance of  $5.0 \pm 1.0$  mm, as depicted in **Fig. 2** is achieved.

**5.3** Oven, air circulating, capable of maintaining  $(150 \pm 5)^\circ\text{C}$ .

**5.4** Air Flowmeters. A separate flowmeter shall be provided for each test assembly such that an airflow rate of  $(250 \pm 5)$  ml per minute can be measured.

## 6 MATERIALS

**6.1** Acetone, laboratory reagent grade.

**6.2** Toluene, laboratory reagent grade.

**6.3** Air supply, dry and oil free

**6.4** Nitrogen, white spot grade.

**6.5** Whatman 541 filter paper.

**6.6** Glassware cleaning fluid. Any suitable laboratory glass cleaning fluid may be used providing it removes all evidence of staining and deposits from previous tests.

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**7 TIME/TEMPERATURE GUIDELINES**

**7.1** The actual test programme will be determined by the evaluation objectives but as a guideline tests are carried out in accordance with the following:

**7.1.1** Constant time varying temperature (eg 192 hours, (100 to 260)°C at 10°C or 5°C intervals) is used to determine temperature parameters as defined in **A.1** and **A.2** of **Appendix A**.

**7.1.2** Constant temperature varying time (eg 210°C for 5, 25, 50, 100 hours etc) is used to determine effective life parameters as defined in **A.1** and **A.3** of **Appendix A**.

**8 PROCEDURE**

**8.1** Chemically clean the glass blowing assembly. This procedure may be waived in the case of new glassware or used glassware which does not exhibit staining. In such cases wash the apparatus in acetone and dry in a current of air.

**8.2** Weigh the assembly to the nearest 0.01 g and record the weight as W1.

**8.3** Remove the gas delivery tube and introduce 50 ml sample into the boiling tube taking care not to splash the sample on the wall of the tube above the 50 ml mark.

**8.4** Reweigh the whole assembly to the nearest 0.01 g and record the weight as W2.

**8.5** Insert the assembly into the heating block set at the required temperature and connect the air supply.

**8.6** Adjust the airflow to  $(250 \pm 5)$  ml per minute.

**8.7** During the test period check the temperature and airflow at least twice daily to monitor that no fluctuation takes place.

**8.8** Reject tests where the temperature has fluctuated by more than  $\pm 2.5^\circ\text{C}$  or the airflow by more than  $\pm 25$  ml during a 24 hour period.

**8.9** At the end of the required test period disconnect the air supply and remove the assembly from the block. Immediately assess if the oil has formed a solidus in accordance with appendix A clause **A.5** and then allow the assembly to cool to room temperature.

**8.10** Weigh the assembly and contents to the nearest 0.01 g and record the weight as W3.

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**8.11** If a solidus has formed, the procedure is terminated at this point. In the absence of solidus restore the assembly and sample to the original weight  $\pm 0.01$  g (as determined at clause **8.4**) by the addition of fresh oil.

**8.12** Return the assembly to the test block or bath and connect a nitrogen supply.

**8.13** Set the flow of nitrogen to approximately  $20 \text{ ml}\cdot\text{min}^{-1}$  for a period of 30 minutes to ensure complete mixing.

**8.14** Remove the assembly and allow to cool to room temperature.

**8.15** Thoroughly scrape the inside of the boiling tube until free of deposit. This can best be achieved with the aid of wire gauze attached to a metal rod.

**8.16** Remove a representative sample of the oil and measure the deterioration in accordance with the following:

Toluene insolubles content – Method 12  
Total acidity – SAE ARP5088

**8.17** Remove a representative sample of the oil and filter through Whatman 541 filter paper or 200 mesh filter before determining the viscosity at  $100^{\circ}\text{C}$  in accordance with ASTM D445/IP 71 (BS 2000: Part 71).

**8.18** Determine new oil values for insolubles content, acidity and viscosity at  $100^{\circ}\text{C}$  using the same test methods as at **8.16** and **8.17**.

## **9 CALCULATION AND REPORTING**

### **9.1 Deterioration Parameters**

**9.1.1** Calculate the required deterioration parameters as defined in **Appendix A, A.1** in accordance with the following:

#### **9.1.1.1 Volatilisation Loss**

Calculate using the following formula:

$$\text{Volatilisation loss, percent weight} = \frac{(W2 - W3)}{(W2 - W1)} \times 100$$

Where  $W1$  = mass of assembly  
 $W2$  = mass of assembly + sample before testing  
 $W3$  = mass of assembly + sample after testing.

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**9.1.1.2** Acidity Change

Calculate as the difference between the test sample and new oil value.

**9.1.1.3** Kinematic Viscosity (KV) Change

Calculate as a percentage of the new oil value as follows:

$$\text{Viscosity change (\% at 100 °C)} = \left( \frac{\text{KV sample}}{\text{KV new}} - 1 \right) \times 100$$

**9.1.1.4** Insolubles Increase

Calculate as the difference between the test sample and new oil value.

**9.1.2** Report the required deterioration parameters to the following accuracy:

Volatilisation loss	=	nearest 1 per cent
Viscosity change	=	nearest 1 per cent
Acidity change	=	nearest 0.1mg KOH g <sup>-1</sup>
Insolubles increase	=	nearest 0.1 mg g <sup>-1</sup>

**9.2** Temperature Parameters - Oxidative Stability

**9.2.1** Determine the required temperature parameters as defined in **Appendix A, A.2** by interpolation from the best line of fit obtained when the deterioration parameters are plotted against temperature for constant time.

**9.2.2** Report the temperature parameters to the nearest 5°C.

**9.3** Effective Life - Oxidative Stability

**9.3.1** Determine the effective lives as defined in **Appendix A, A.3** by interpolation from the best line of fit obtained when the deterioration parameters are plotted against time for constant temperatures.

**9.3.2** Effective lives for temperatures at which no deterioration parameters have been determined can be determined by interpolation of the temperature on the least mean square plot (Arrhenius plot). This can be obtained using the procedure given in **Appendix A, A.4**.

**9.3.3** Report the effective life parameters to the following accuracy:

- |                 |   |                        |
|-----------------|---|------------------------|
| Up to 5 hours   | - | to the nearest ½ hour  |
| 5 to 25 hours   | - | to the nearest 1 hour  |
| 25 to 100 hours | - | to the nearest 2 hours |
| above 100 hours | - | to the nearest 5 hours |

**9.4** Activation Energy and Effective Collision Frequency - Oxidative Stability

**9.4.1** If required, calculate the apparent activation energy ( $E_a$ ) and the logarithm of effective collision frequency ( $\log_{10}m$ ) using the procedure given in **Appendix A, A.4.2**.

**9.4.2** Report  $E_a$  and  $\log_{10}m$ , where required, to three significant figures.

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**10 PRECISION**

A formal precision exercise has not been carried out. However, prior experience has shown that the following precision levels can be achieved.

**Table D Precision Levels**

PROPERTY		REPEATABILITY	REPRODUCIBILITY
Temperature Parameters °C	A <sup>D</sup>	±5	±7½
	B <sup>D</sup>	±5	±7½
	E <sup>D</sup>	±5	±7½
	V <sup>D</sup>	±5	±7½
	Z <sup>D</sup>	±5	±7½
Effective Life (hrs)	Up to 5 hrs	±½	±½
	5-25 hrs	±1	±1
	25-100 hrs	±2	±5
	>100 hrs	±5	±10
Deterioration Parameter (25 hrs air blowing at 190°C) Acidity Increase (mg KOH g <sup>-1</sup> )		±0.1	±0.2

NOTE: Precision data for E<sub>a</sub> and log<sub>10</sub>m are not given as these are derived from the above results.

## APPENDIX A

### SPECIFIC LUBRICANT DETERIORATION CRITERIA

#### A.1 Deterioration Parameters

The deterioration parameters are defined as; volatilisation loss, solidus formation, increase in acidity, viscosity and insolubles content. There are two protocols for examining the lubricant degradation process; Temperature Parameters uses a constant time whilst temperature is varied; Effective Life uses a constant temperature whilst time is varied. In each case the deterioration parameters are measured and then plotted on a graph, against temperature in the case of temperature parameters, and against time in the case of effective life. The graphs are then used to determine the points at which the various deterioration parameters reach certain pre-defined assessment levels. These assessment levels are defined in clauses A.2 and A.3.

#### A.2 Temperatures Parameters Assessment Levels

- |                            |   |  |
|----------------------------|---|--|
| A <sup>D</sup> temperature | - | the lowest constant temperature, for a test duration of D hours, at which the total acid number increases by 1.0 mg KOH g <sup>-1</sup> .              |
| B <sup>D</sup> temperature | - | the lowest constant temperature for a test duration of D hours, at which the toluene insoluble matter increases by 0.05 % by weight.                   |
| E <sup>D</sup> temperature | - | the lowest constant temperature, for a test duration of D hours, at which the volatilisation loss is 15 % by weight.                                   |
| V <sup>D</sup> temperature | - | the lowest constant temperature, for a test duration of D hours, at which the viscosity, measured at 100°C increases by 15 %.                          |
| Z <sup>D</sup> temperature | - | the lowest constant temperature, for a test duration of D hours, which produces the "solidus" condition defined in clause <b>A.5</b> of this appendix. |

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**A.3 Effective Life Assessment Levels**

- |                     |   |  |
|---------------------|---|--|
| Volatilisation Loss | - | the duration at a specified constant temperature which produces a volatilisation loss of 15 % by weight.                     |
| Acidity Increase    | - | the duration at a specified constant temperature which produces an increase in total acidity of 1.0 mg KOH g <sup>-1</sup>   |
| Viscosity Increase  | - | the duration at a specified constant temperature which produces an increase in viscosity, measured at 100 °C, of 15 %.       |
| Insolubles Increase | - | the duration at a specified constant temperature which produces an increase in toluene insoluble matter of 0.05 % by weight. |

**A.4 Effective Life and Temperature Relationship**

**A.4.1** The relationship between effective-life and temperature is given by the equation:

$$D = \frac{1}{m} \cdot \exp\left(\frac{E_a}{R\theta}\right) \quad \text{----- equation 1}$$

- where;
- |                |   |  |
|----------------|---|--|
| D              | = | effective life (hrs)   |
| θ              | = | absolute temperature K(= T °C + 273.15)  |
| R              | = | molar gas constant = 1.98719 calmol <sup>-1</sup> K <sup>-1</sup>  |
| E <sub>a</sub> | = | apparent activation energy (cal)   |
| m              | = | effective collision frequency peculiar to the species participating in the oxidation reaction (hrs <sup>-1</sup> ) |

**A.4.2** The following logarithmic form of equation 1 is more convenient in practice;

$$\log_{10} D = \frac{E_a}{R\theta \log_e 10} - \log_{10} m \quad \text{----- equation 2}$$

**A.4.3** Equation 2 represents a linear relationship between logarithm of effective life and reciprocal absolute temperature. The gradient will be proportional to  $E_a$  and the y-intercept will represent  $\log_{10}m$ .

**A.4.4** Calculation of Apparent Activation Energy  $E_a$  and Effective Collision Frequency  $\log_{10} m$ .

**A.4.4.1** Simple linear regression mathematics may therefore be employed to calculate the values  $E_a$  and  $\log_{10}m$ . The data points used in the calculation are the assessment level temperatures and effective life parameters as defined in this appendix at clauses **A.2** and **A.3** respectively. For the purpose of the calculation of  $E_a$  and  $\log_{10}m$ , the assessment level temperatures should be interpolated to the nearest 1°C and the effective lives should be interpolated to the nearest ½ hour.

**A.4.4.2** The following equation is used to calculate  $E_a$ ;

$$E_a = R \cdot \log_e 10 \left( \frac{n \cdot \sum_{i=1}^n \frac{\log_{10} D_i}{\theta_i} \cdot \sum_{i=1}^n \frac{1}{\theta_i} \cdot \sum_{i=1}^n \log_{10} D_i}{n \cdot \sum_{i=1}^n \frac{1}{\theta_i^2} - \sum_{i=1}^n \frac{1}{\theta_i}} \right) \quad \text{----- equation 3}$$

where;       $n$  =      number of data points available for particular mode of deterioration (i.e. number of assessment level temperatures plus number of effective lives determined).

**A.4.4.3** The following equation is used to calculate  $\log_{10} m$ ;

$$\log_{10} m = \left( \frac{1}{-n} \right) \cdot \sum_{i=1}^n \log_{10} D_i - \left( \frac{E_a}{R \cdot \log_e 10} \right) \cdot \sum_{i=1}^n \frac{1}{\theta_i} \quad \text{----- equation 4}$$

**A.4.5** Arrhenius Plot

An Arrhenius plot showing the relationship obtained between the effective life and temperature for the particular deterioration parameter may be produced using equation 2 to generate two suitable data points which will exist on the best fit straight line. This line should be drawn on the graph together with the original data points obtained in this appendix at clauses **A.2** and **A.3**.

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**METHOD 9**

**A.4.6** Calculation of Effective Life

Effective lives for temperatures at which no deterioration parameters have been determined by testing can be obtained by either of two methods. For approximate determinations, the effective life may be determined by interpolation or extrapolation of the Arrhenius Plot for the particular deterioration parameter. For accurate determinations (i.e. for reporting or calculation purposes), the effective life should be determined by substituting into equation 2 the values of  $E_a$  and  $\log_{10}m$  obtained above for the particular deterioration parameter and the temperature at which the effective life is required.

**A.5** Determination of a Solidus

**A.5.1** Disconnect the gas supply from the delivery tube.

**A.5.2** Remove the delivery tube from the boiling tube.

**A.5.3** Observe the oil sample in the boiling tube and on the delivery tube for evidence of flow.

**A.5.4** If no flow is observed within a 2 minute time period, record the formation of a solidus.

## Standards for Defence

### Methods for Testing Gas Turbine Engine Synthetic Lubricants

#### Method 12 : Determination of Toluene Insoluble Matter

## 1 SCOPE

This method describes a procedure for the determination of the amount of toluene insoluble matter present in synthetic gas turbine lubricants either when new or after being subject to other test procedures. This method is based on Rolls Royce lubricant approval methodology.

## 2 WARNING

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## 4 OUTLINE OF METHOD

A known weight of oil is mixed with toluene and passed through a PTFE membrane filter of 5.0 µm mean pore size. The filter shall be weighed before and after the oil is passed through it and the difference in weight is used to calculate the amount of insoluble matter present per gram of oil filtered.

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**METHOD 12**

**5 APPARATUS**

- 5.1** Conical glass flask, 500 ml capacity.
- 5.2** Plastic wash bottle for dispensing filtered (through 5 µm membrane) toluene.
- 5.3** PVC 'Policeman'. The PVC should be substantially free from plasticisers which may be readily leached out by toluene.
- 5.4** PTFE membrane filters, having a mean pore size of 5 µm and diameter of 47 mm (Millipore Cat No LSWP 047 00 is suitable).
- 5.5** Filter holder apparatus consisting of the following (Millipore Cat No XX10 047 30 is suitable);
- 5.5.1** borosilicate glass 300 ml funnel with ground glass seal
  - 5.5.2** borosilicate glass base with ground glass seal
  - 5.5.3** Stainless steel screen filter support
  - 5.5.4** PTFE gasket for stainless steel filter support
  - 5.5.5** anodised aluminium spring clamp (WARNING: The clamp shall be earthed in accordance with safe practices associated with the filtration of solvents).
  - 5.5.6** rubber stopper compatible with glass base
- 5.6** 1 litre side arm flask with an opening compatible with the stopper of the filter holder apparatus (Millipore Cat No XX10 047 05 is suitable).
- 5.7** Aluminium foil lid to cover filter holder.
- 5.8** Petri dish, approximately 100 mm in diameter.
- 5.9** Tweezers, flat headed with unserrated non-pointed tips.
- 5.10** Oven, electric, flash proof, controlled at  $(100 \pm 5)^{\circ}\text{C}$ .
- 5.11** Measuring cylinder, BS604, 100 ml capacity.

**5.12** Vacuum equipment such as a 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. The receiving flask, solvent trap and any tubing connecting them shall be earthed in accordance with safe practices associated with the filtration of solvents.)

## **6 MATERIALS**

**6.1** Acetone, analytical reagent grade.

**6.2** Toluene, analytical reagent grade pre-filtered through a 5 µm filter as identified in **5.4**.

## **7 PREPARATION OF APPARATUS**

**7.1** Clean all glassware and filtration equipment by thoroughly washing first in warm water containing detergent, then clean warm water, demineralised water, acetone and finally filtered toluene.

**7.2** Dry the petri dish, with its lid slightly ajar, in an oven set at 100°C for 30 minutes.

**7.3** Preparation of filter.

**7.3.1** Using the tweezers, place a filter in the cleaned petri dish. Place the petri dish with the lid slightly ajar, in an oven set at 100°C for 30 minutes. Remove from the oven and allow sufficient time (30 minutes) for the filter to equilibrate with ambient temperature and humidity; allow the petri dish cover to remain very slightly ajar but still protecting the filter from contamination. Weigh the filter and record the weight to the nearest 0.1 mg.

**7.3.2** Repeat **7.3.1**, until the difference between two successive determinations of filter weight is less than or equal to 0.1 mg.

**7.3.3** Using the tweezers, place the weighed filter centrally on the stainless steel screen of the filter holder and install and clamp the holder. Wash with filtered toluene and protect the funnel with the aluminium foil lid until required for use.

## **8 PROCEDURE**

**8.1** Thoroughly mix the oil sample by shaking, or stirring with a glass rod.

**8.2** Weigh, as appropriate, approximately 100 grams to the nearest 0.01 g of new oil or approximately 10 g to the nearest 0.001 g of degraded oil into the conical flask. For highly degraded oil the sample mass may be reduced to avoid filter blockage.

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**METHOD 12**

**8.3** Add (100 ± 1) ml of the toluene using the measuring cylinder and swirl for 30 seconds.

**8.4** Pour the toluene/oil sample mix 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.

**8.5** Rinse the conical flask thoroughly with filtered toluene, loosening any solid material adhering to the walls with the aid of the PVC policeman and transfer to the filter.

**8.6** Using filtered toluene, wash down the inside of the filter funnel. Repeat this operation six times. With the vacuum still applied, carefully remove the clamp and funnel. Wash the periphery of the Millipore filter by directing a gentle stream of filtered toluene from the edge to the centre, taking care not to wash any solid matter from the filter. After the final washing disconnect vacuum immediately after the excess toluene has been drawn through.

**8.7** With clean tweezers, carefully remove the filter from the screen and place in a clean petri dish and cover. Follow the procedure in **7.3.1** and **7.3.2**.

**9. CALCULATION AND REPORTING**

**9.1** Calculate Toluene Insoluble Matter, % mass, or mg g<sup>-1</sup> according to the following formulae:

$$\frac{100 \cdot (W2 - W1)}{w} = \% \text{ mass}$$

$$\frac{1000 \cdot (W2 - W1)}{w} = \text{mg g}^{-1}$$

where: W1 = Mass of filter in grams  
W2 = Mass of filter + toluene insoluble matter in grams  
w = Mass of oil sample used in grams

**9.2** Report to nearest 0.01 % or 0.1 mg g<sup>-1</sup>.

**10 PRECISION**

A formal precision exercise has not been carried out but prior experience has shown that the following precision levels can be achieved.

**Table E Precision Levels**

INSOLUBLES LEVEL	REPEATABILITY		REPRODUCIBILITY	
	%	mg g <sup>-1</sup>	%	mg g <sup>-1</sup>
<0.05% / 0.5 mg	0.01	0.1	0.02	0.2
0.1% / 1.0 mg	0.02	0.2	0.04	0.4
0.5% / 5.0 mg	0.2	2.0	0.4	4.0
10% / 10 mg	0.4	4.0	0.8	8.0

**Standards for Defence**

**Methods for Testing Gas Turbine Engine Synthetic Lubricants**

**Method 14 : Catalytic Oxidation Test**

**1 SCOPE**

This method describes a procedure for the determination of the influence of metals on the high temperature oxidative stability of synthetic gas turbine lubricants in respect to the specified degradation modes and for test durations of 192 hours and 6 hours. This Method is based on Rolls-Royce lubricant approval methodology.

**2 WARNING**

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## 4 OUTLINE OF METHOD

**4.1** Method 14 comprises a modification of **Method 9**, the only difference in apparatus being substitution of the glass air delivery tube by a metal tube of identical bore, but having brazed to its lower detachable head alternate vanes of copper and mild steel. Oil sample volume and air flow rate remain unaltered so that the results furnished by the method, when compared with those obtained by the glass delivery tube (**Method 9**) under the same conditions of test temperature and duration, will reflect the combined influence of copper and iron on the oxidative stability of the lubricant.

**4.2**  $A^D$ ,  $V^D$ ,  $E^D$ , and  $Z^D$  (**Method 9, Appendix A, A.1 and A.2**) are first determined by conducting tests in accordance with Method 9 at different temperatures, each for the same test duration (D) of 192 hours or 6 hours.

**4.3** The shifts in  $A^D$ ,  $V^D$ ,  $E^D$ , and  $Z^D$  produced by the presence of copper and iron are symbolised by  $\Delta A^D$ ,  $\Delta V^D$ ,  $\Delta E^D$  and  $\Delta Z^D$  respectively.

## 5 APPARATUS

**5.1** Heat Transfer apparatus as described in **Annex A, A.1 and A.2**.

**5.2** Glassware and metal air blowing assembly as depicted in **Annex B, Fig. 5**.

**5.3** Balance with an accuracy of 0.01 g.

**5.4** Air-Circulating Oven capable of maintaining a temperature of  $(150 \pm 2)^\circ\text{C}$ .

**5.5** 600 ml tall form beaker and watch glass to cover.

**5.6** Air Flowmeters. A separate flowmeter shall be provided for each test assembly such that an airflow rate of  $(250 \pm 5)$  ml per minute can be measured.

**5.7** Brush, short bristled, stiff (must be compatible with the tri-solvent).

## 6 MATERIALS

**6.1** Metal Vanes Air Blowing Assembly Cleaning Fluid. Tri-solvent (equal quantities of acetone, propan-2-ol and toluene; all GPR grade) is suitable.

**6.2** Glassware Cleaning Fluid. Any suitable laboratory glass cleaning fluid may be used providing it removes all evidence of staining and deposits from previous tests.

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METHOD 14**

- 6.3** Distilled/Deionised Water, ISO 3696 grade 3
- 6.4** Alumina abrasive, 400 Grade.
- 6.5** Acetone, laboratory reagent grade.
- 6.6** Whatman 541 filter paper or 200 mesh filter.
- 6.7** Toluene, laboratory reagent grade.
- 6.8** Air Supply, dry and oil free
- 6.9** Nitrogen, white spot
- 6.10** Metals for construction of the metal vanes air blower tube assembly are as follows:
- |                            |             |
|----------------------------|-------------|
| Upper stainless steel tube | BS 2T 66    |
| Lower copper tube          | BS EN 12451 |
| Copper vanes               | BS EN 13599 |
| Mild Steel vanes           | BS 2S 510   |
| Brazing brass              | BS 1845     |

**7 APPARATUS CLEANING**

- 7.1** Chemically clean the glass boiling tube and glass head as follows:
- 7.1.1** Immerse in the glassware cleaning fluid in accordance with laboratory procedures and safety instructions.
- 7.1.2** Thoroughly rinse glassware in distilled/deionised water and dry in an air-circulating oven.
- 7.2** Clean the metal blower tube assembly as follows:
- 7.2.1** Wet-blast with alumina abrasive 400 grade.
- 7.2.2** Wash thoroughly with warm water containing detergent.
- 7.2.3** Rinse thoroughly with warm tap water followed by rinses in demineralised water, toluene and acetone.
- 7.2.4** Dry with clean dry compressed air.

## 8 PROCEDURE

- 8.1** Test temperatures are selected such that a plot of deterioration versus temperature can be constructed for 192 hours and 6 hours test duration. By reference to similar plots for tests carried out in accordance with Method 9 in the absence of metals, the shift in degradation temperature can be quantified.
- 8.2** Weigh the detachable metal head assembly to the nearest 0.01 g.
- 8.3** Assemble the metal blower tube, connect to the glass head by means of silicone rubber tubing and insert into the boiling tube.
- 8.4** Weigh the whole assembly to the nearest 0.01 g.
- 8.5** Remove the metal delivery tube and introduce 50 ml sample into the boiling tube taking care not to splash the sample on the wall of the tube above the 50 ml mark.
- 8.6** Reweigh the whole assembly to the nearest 0.01 g.
- 8.7** Insert the assembly into the heating block and connect the air supply.
- 8.8** Adjust the airflow to  $(250 \pm 5)$  ml per minute.
- 8.9** During the 192 hour test period check the temperature and flow at least twice daily to monitor that no fluctuation takes place.
- 8.10** Reject tests where the temperature has fluctuated by more than  $\pm 2.5^{\circ}\text{C}$  or the flow by more than  $\pm 25$  ml during a 24 hour period.
- 8.11** At the end of the test period of either 192 hours or 6 hours disconnect the air supply and remove the assembly from the heating block. Immediately assess if the oil has formed a solidus in accordance with **Method 9, Appendix A, A.2** and allow the assembly to cool to room temperature.
- 8.12** Weigh the assembly and contents to the nearest 0.01 g.
- 8.13** If the only parameters required are solidus and/or volatility, or if the oil has formed a solidus, the procedure is terminated at this point. If any further deterioration parameters are required proceed as in **8.14**
- 8.14** Restore the assembly and contents to the original weight  $\pm 0.01$  g (as determined at clause **8.6**) by addition of fresh oil.
- 8.15** Return the assembly to the heating block/bath and connect a nitrogen supply.

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**METHOD 14**

- 8.16** Set the flow of nitrogen to approximately  $20 \text{ ml}\cdot\text{min}^{-1}$  for a period of 30 minutes to ensure complete mixing.
- 8.17** Remove the assembly, and allow to cool to room temperature.
- 8.18** Remove the metal assembly and allow surface oil to drain into the blowing tube.
- 8.19** Remove a representative sample of the oil and measure the total acidity in accordance with SAE ARP5088.
- 8.20** Remove a representative sample of the oil and filter through Whatman 541 filter paper or 200 mesh filter before determining the viscosity at  $100^{\circ}\text{C}$  in accordance with ASTM D445/IP 71(BS 2000: Part 71).
- 8.21** Determine new oil values for acidity and viscosity at  $100^{\circ}\text{C}$  using the same test methods at **8.19** and **8.20**.
- 8.22** Remove a representative sample of the oil and determine the dissolved copper and iron content by any suitable method.
- 8.23** Wash the detachable metal assembly head with acetone to remove oil and dry in a current of air.
- 8.24** Some vanes may have tenacious deposits remaining. If this is the case, clean the metal assembly head by immersion in the tri-solvent, followed by scrubbing with the brush until all deposits have been removed.
- 8.25** Rinse with acetone and dry in a current of clean dry compressed air.
- 8.26** Reweigh to the nearest 0.01 g.

**9 REPORTING**

- 9.1** Comparing the results obtained in this procedure with those obtained with method 9, report the shift in the degradation temperature parameters in respect to acidity increase  $A^{\text{D}}$ , viscosity increase  $V^{\text{D}}$ , volatilisation loss  $E^{\text{D}}$ , and solidus temperature  $Z^{\text{D}}$ .
- 9.2** Report the metal weight change of the detachable assembly head.
- 9.3** Report the weights of copper and iron dissolved in the oil.

**10 PRECISION**

A formal precision exercise has not been carried out. However, previous experience has shown that the following precision levels can be achieved:

**Table F Precision Levels**

PROPERTY		REPEATABILITY	REPRODUCIBILITY
Temperature Parameters °C	A <sup>D</sup>	±2.5	±5
	V <sup>D</sup>	±2.5	±5
	E <sup>D</sup>	±2.5	±5
	Z <sup>D</sup>	±2.5	±5

**Standards for Defence**

**Methods for Testing Gas Turbine Engine Synthetic Lubricants**

**Method 22: Assessment of the Compatibility of Gas Turbine Lubricants with Elastomers -  
Gravimetric Method**

**1 SCOPE**

**1.1** This method describes the procedure for assessing the compatibility of synthetic gas turbine lubricants with elastomers in terms of weight gain after ageing at elevated temperature. Resistance to embrittlement is assessed by determining the time for cracks to appear following ageing in a similar manner.

**1.2** Gas turbine lubricants comprise of a variety of additives, which either enhance or impart desirable properties to the base oil. These additives frequently contain materials that have pronounced detrimental effects on the physical properties of elastomeric seals, which are in contact with the lubricant in service.

**2 WARNING**

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**4 OUTLINE OF METHOD**

**4.1** An elastomer specimen completely immersed in the test lubricant, open to the atmosphere, is maintained at constant temperature for the period of the test, at the end of which it is cooled by immersion in the cold test lubricant.

**4.2** The change in mass of the elastomer is then measured followed by a test to assess the embrittlement characteristics of the elastomer.

**5 APPARATUS**

**5.1** Heat Transfer apparatus as described in **Annex A, A.1** and **A.2**.

**5.2** Oven, air circulating and capable of being controlled to  $\pm 2^{\circ}\text{C}$  at test temperatures of  $100^{\circ}\text{C}$ ,  $130^{\circ}\text{C}$ ,  $150^{\circ}\text{C}$ ,  $175^{\circ}\text{C}$  and  $200^{\circ}\text{C}$ .

**5.3** Test apparatus described in Table G and illustrated in **Annex B, Fig. 10**.

**Table G Test Apparatus**

Item	Comments	Number Required
Glass tube	50 ml level marked	7
Glass rod	shaped into hook at one end	4
Cork Stopper	size to fit neck of tube. hole bored through centre to accommodate glass rod. V shaped groove cut into tapered surface to act as vent.	4

**5.4** A microscope or magnifying lens that enables x 10 magnification.

**6 MATERIALS**

**6.1** Glassware Cleaning Fluid: Any suitable laboratory glass cleaning fluid may be used providing it removes all evidence of staining and deposits from previous tests.

**6.2** Distilled/Deionised Water, ISO 3696 grade 3.

**6.3** Petroleum Ether (reagent grade), boiling range 40/60 or 30/60.

**6.4** Test pieces: British Standard M48 (or SAE AS568) size 113 O-rings (four required per lubricant / elastomer combination, see **8.1** and **8.6.3**). Materials required are as shown in **Table H**.

**DEF STAN 05-50 (PART 61)/2**  
**METHOD 22**

**Table H Elastomer Materials**

ELASTOMER MATERIAL	ROLLS-ROYCE MATERIAL SPECIFICATION	HARDNESS, IRHD VALUE
Nitrile	MSRR9495	70 - 80
Silicone	MSRR9492	65 - 75
Fluorocarbon	MSSR9450	70 - 80
Low Compression Set Fluorocarbon	MSRR9482	75 - 85

**6.5** Test lubricant, (350 ml required per lubricant / elastomer combination.)

**7 PREPARATION OF APPARATUS**

**7.1** Chemically clean the glass boiling tube and glass head as follows:

**7.1.1** Immerse in the glassware cleaning fluid in accordance with laboratory procedures and safety instructions.

**7.1.2** Thoroughly rinse glassware in distilled/deionised water and dry in an air-circulating oven.

**7.2** Prepare each O-ring as follows:

**7.2.1** Thoroughly wash the O-ring with petroleum ether.

**7.2.3** Set an air-circulating oven to the relevant temperature for the material being tested (see Table I below) and allow it to stabilise. Place the O-ring on a heated flat tray in the oven for  $60 \pm 5$  minutes.

**7.2.4** Remove O-ring from oven, place in a desiccator and store away from bright sunlight. Allow O-ring to cool for a minimum of 3 hours.

**Table I Elastomer Temperature Data**

ELASTOMER TYPE	TEMPERATURE (°C)	LUBRICANTS
Nitrile	130 ± 2	All lubricants
Silicone	100 ± 2	For OX-26 to Def Stan 91-100
Silicone	175 ± 2	All lubricants except OX-26
Fluorocarbon and LCS Fluorocarbon	200 ± 2	All lubricants

## **8 PROCEDURE**

**8.1** For each combination of lubricant and elastomer material, three O-ring test pieces will be tested. **8.2** to **8.5** describe the procedure for each test piece. Follow these instructions for all three.

### **Weight Change Determination**

**8.2** Initial Weight Measurement

**8.2.1** Prepare each test piece as described in **7.2**.

**8.2.2** Weigh in air to the nearest 0.0001g and record the weight ( $W_o$ ).

**8.3** Procedure to begin test.

**8.3.1** Set the heating apparatus to the required test temperature shown in **clause 7, Table H**, and allow it to reach equilibrium.

**8.3.2** Insert a glass rod through the hole in a cork such that when the cork is located in the neck of the glass tube, the hook is approximately midway between the 50 ml line and the bottom of the tube.

**WARNING:** Care should be taken when inserting the rod into the cork, to avoid breakage.

**8.3.3** Remove the cork and glass rod from the glass tube and fill to the 50 ml mark with a fresh sample of test lubricant.

**8.3.4** Place the O-ring test piece on the hook of the glass rod and lower it into the oil slowly to ensure that it is not dislodged from the hook. Locate the cork firmly in the neck of the glass tube.

**DEF STAN 05-50 (PART 61)/2**  
**METHOD 22**

- 8.3.5** Place the tube in the heating apparatus and start the test period.
- 8.4** Interim Weight Measurement (24 hours)
- 8.4.1** Fill a glass tube to the 50ml mark with a fresh sample of test lubricant and maintain it at room temperature. This is used to cool the O-ring at **8.4.3**.
- 8.4.2** After 24 hours has passed, remove the tube from the heating apparatus.
- 8.4.3** Immediately transfer the cork/glass rod and O-ring test piece to the glass tube prepared at **8.4.1** and leave for  $(30 \pm 5)$  minutes.
- 8.4.4** Remove the O-ring test piece from the glass tube and rinse thoroughly in petroleum ether. Dry with a short exposure to a slow flow of room temperature air and/or blot with absorbent paper or fabric that does not deposit lint.
- 8.4.5** Weigh the O-ring test piece in air to the nearest 0.0001 g and record the weight ( $W_i$ ).
- 8.4.6** Replace the O-ring test piece on the hook and return it to the tube of test lubricant removed from the heating apparatus at **8.4.2**.
- 8.4.7** Replace the tube in the heating apparatus and continue the test period.
- 8.4.8** Store the tube prepared at **8.4.1** in a dark environment.
- 8.5** Final Weight Measurement
- 8.5.1** After 120 hours cumulative test time has elapsed remove the assembly from the heating apparatus and repeat **8.4.3** and **8.4.4**. Weigh the test piece in air and record the weight to the nearest 0.0001 g ( $W_f$ ).

**Embrittlement Procedure**

- 8.6** Take one of the O-ring test pieces weighed at **8.5.1** and assess it in the following manner.
- 8.6.1** Twist the O-ring test piece into a figure of eight and maintain for 5 seconds under a lens that gives x 10 magnification. Inspect for cracks against the criteria detailed below.

PASS - No cracks.

FAIL - Cracks visible or test piece breaks

**8.6.2** If the O-ring test piece meets the PASS criterion replace the O-ring on the hook, place the glass rod and cork in the tube and return it to the heating apparatus. Carry out the procedure in **8.7** with this O-ring and record that five days have already elapsed.

**8.6.3** If the test piece meets the FAIL criterion, prepare a new O-ring in accordance with **7.2**, and carry out the procedures in **8.3** and **8.7**.

**8.7** On a daily basis (every 24 hours) remove the tube from the heating apparatus.

**8.7.1** Remove the cork/glass rod and O-ring test piece from the tube. Dry the O-ring with absorbent paper or fabric that does not deposit lint. Allow the O-ring to cool to room temperature, this could be hastened by exposure to a slow flow of room temperature air.

**8.7.2** Carry out the procedure in **8.6.1**.

**8.7.3** If the O-ring test piece meets the FAIL criterion, note the number of days since the test began (see **10.3**).

**8.7.4** If the O-ring test piece meets the PASS criterion and it is not day 8 (see **8.7.5**) replace the O-ring on the hook and slowly lower the glass rod into the tube. Locate the cork firmly in the neck of the glass tube and return it to the heating apparatus.

**8.7.5** If the O-ring test piece passes on day 8 discard the tube of lubricant. Using a clean glass tube follow the procedure in **8.3.2** to **8.3.4**. Replace the tube in the heating apparatus and continue to carry out the procedure described in **8.7.1** to **8.7.3** on a daily basis.

**8.8** Repeat **8.7** until the test piece meets the FAIL criterion or day 15 is reached.

**8.9** If the O-ring test piece passes on day 15 the test is ended and the time to failure shall be reported as greater than 15 days.

## **9 CALCULATION**

Calculate the change in weight after immersion for 24 hours and 120 hours. Calculate the average result for the three O-ring test pieces used with each sample.

$$24 \text{ hours (\%)} = \left( \frac{W_i}{W_o} - 1 \right) \times 100$$

$$120 \text{ hours (\%)} = \left( \frac{W_f}{W_o} - 1 \right) \times 100$$

**DEF STAN 05-50 (PART 61)/2  
METHOD 22**

**10 REPORTING**

**10.1** Report the lubricant/elastomer combination tested.

**10.2** Weight Change: Report the weight change at 24 and 120 hours to the nearest 0.5 % units.

**10.3** Embrittlement Characteristics: Report the time taken, in days, for the O-ring test piece to display the embrittlement fail criterion specified in **8.6.1** (e.g. if the O-ring fails on the 14<sup>th</sup> day, report the result as a day 14). Report the result as >15 if failure has not occurred by day 15.

**11 PRECISION**

**11.1** Repeatability

The results obtained by two separate tests, on identical samples, conducted within the same laboratory (i.e. by the same operator) shall not be considered suspect unless they differ by more than 2 % units.

**11.2** Reproducibility

The results obtained by two separate tests, on identical samples, conducted by two different laboratories, should not be considered suspect unless they differ by more than 2 % units.

Standards for Defence

Methods for Testing Gas Turbine Engine Synthetic Lubricants

Method 24: Assessment of the Compatibility of Mixtures of Gas Turbine  
Lubricating Oils

**1 SCOPE**

This method is used for determining the compatibility of aircraft turbine lubricants with specific referee lubricants, as specified in the relevant lubricant specifications. This method is based on Federal Test Method 791C Method 3403, incorporating the modifications called for in MIL-PRF-23699F.

**2 WARNING**

The Ministry of Defence (MOD), like its contractors, is subject to both United Kingdom and European laws regarding Health and Safety at Work, without exemption. All Defence Standards either directly or indirectly invoke the use of processes and procedures that could be injurious to health if adequate precautions are not taken. Defence Standards or their use in no way absolves users from complying with statutory and legal requirements relating to Health and Safety at Work.

**3 RELATED DOCUMENTS**

**3.1** The publications referred to in the text of this standard are listed at **Annex C**. Publications are grouped and listed in alphanumeric order.

**3.2** Reference in this standard to any related document means in any invitation to tender or contract the edition and all amendments current at the date of such tender or contract unless a specific edition is indicated.

**3.3** In consideration of **3.2** above, users shall be fully aware of the issue and amendment status of all related documents, particularly when forming part of an invitation to tender or contract. Responsibility for the correct application of standards rests with users.

**3.4** DStan can advise regarding where related documents are obtained from. Requests for such information can be made to the DStan Helpdesk. How to contact the Helpdesk is shown on the outside rear cover of this standard.

**DEF STAN 05-50 (PART 61)/2**  
**METHOD 24**

**4 OUTLINE OF METHOD**

The oil under test is blended with referee lubricants as specified by the relevant product specification. Three mixtures of varying concentration are prepared for each referee lubricant. The mixtures are heated in an oven at a fixed temperature for seven days, and then stored in a dark environment at room temperature for 21 days. When this has elapsed the mixtures are assessed visually and then the solid particle contamination is determined in accordance with FED-STD-791 Method 3010. This involves filtering a known volume of each mixture through a pre-weighed filter and determining the increase in filter weight.

**5 APPARATUS**

**5.1** Oven, air circulating and capable of being controlled at  $105 \pm 1^\circ\text{C}$  ( $221 \pm 2^\circ\text{F}$ ) (to accommodate three 250 ml flasks).

**5.2** Glassware. 3 Erlenmeyer flasks, Pyrex, 250 ml with glass stoppers.

**5.3** Refer to FED-STD-791, method 3010 for relevant apparatus.

**6 MATERIALS**

**6.1** Sample lubricant 300 ml.

**6.2** Referee lubricant 300 ml.

**6.3** Solvent: any one of the following is suitable; Petroleum Ether (reagent grade), boiling range 40/60 or 30/60, n-heptane or hexane.

**6.4** Glassware cleaning fluid. Any suitable laboratory glass cleaning fluid may be used providing it removes all evidence of staining and deposits from previous tests.

**6.5** Distilled/Deionised Water, ISO 3696 grade 3

**7 PREPARATION OF APPARATUS**

**7.1** Clean the flasks and stoppers as follows;

**7.1.1** Thoroughly rinse with the solvent described at **6.3** and allow to air dry in a suitable fume extraction system.

**7.1.2** Immerse the glassware in the laboratory glass cleaning fluid.

**7.1.3** Rinse first with tap water, then with distilled water, and dry in an oven set at  $105^\circ\text{C}$  ( $220^\circ\text{F}$ ).

**8 PROCEDURE**

**8.1** Prepare three 200 ml mixtures, in 250 ml Erlenmeyer flasks, using the proportions shown in Table J (clearly mark each flask to identify the mixture it contains);

**Table J Composition of Lubricant Mixtures**

Mixture Number	Volume of Sample Lubricant	Volume of Referee lubricant
1	180	20
2	100	100
3	20	180

**8.2** Close each flask with a glass stopper and manually shake them vigorously for one minute.

**8.3** Loosen the stoppers and heat the flasks in an oven for 168 hours at  $105 \pm 3^{\circ}\text{C}$  ( $221 \pm 5^{\circ}\text{F}$ ).

**8.4** Upon completion of 168 hours, remove the flasks from the oven and store them in a dark environment at room temperature,  $24 \pm 5^{\circ}\text{C}$  ( $75 \pm 9^{\circ}\text{F}$ ) for 21 days.

**8.5** When 21 days has elapsed visually inspect the mixtures for evidence of turbidity.

**8.6** Determine the sediment content in accordance with FED-STD-791, method 3010 "Solid Particle Contamination in Aircraft Turbine Engine Lubricants (Gravimetric Procedure)".

**NOTE:** It is not necessary to determine the quantity of non-combustible contamination (ash) as described in FED-STD 791, Method 3010.

## **9 CALCULATION**

Perform the calculation detailed in FED-STD 791, Method 3010.

## **10 REPORTING**

**10.1** Report if there was any evidence of turbidity.

**10.2** Report the quantity of particulate formed, as  $\text{mg l}^{-1}$  of oil mixture.

## **11 PRECISION**

The precision of this method has not been established.

## ANNEX A

## TEST EQUIPMENT DETAILS

**A.1 HEAT TRANSFER TEST BLOCK**

**A.1.1** The apparatus required consists of an electrically heated aluminium block controlled by an electronic heater controlling device. The apparatus should contain holes for housing the test apparatus for **Methods 1, 3, 9, 14** and **22**. Any apparatus, with suitable holes, that can meet the requirements of **A.2.1** may be used although the heating block described at **A.1.2** is recommended.

**A.1.2** Grant Instruments (Cambridge) Ltd manufacture a suitable heating block and controller.

**Table K Recommended Heat Transfer Apparatus**

Part	Description
SP BLOCK	Special Block, as serial number 003995002, previously supplied to Rolls-Royce and QinetiQ FLC.
SP BLOCK CONTROLLER	Special Block heater control and alarm unit with interconnecting cables.

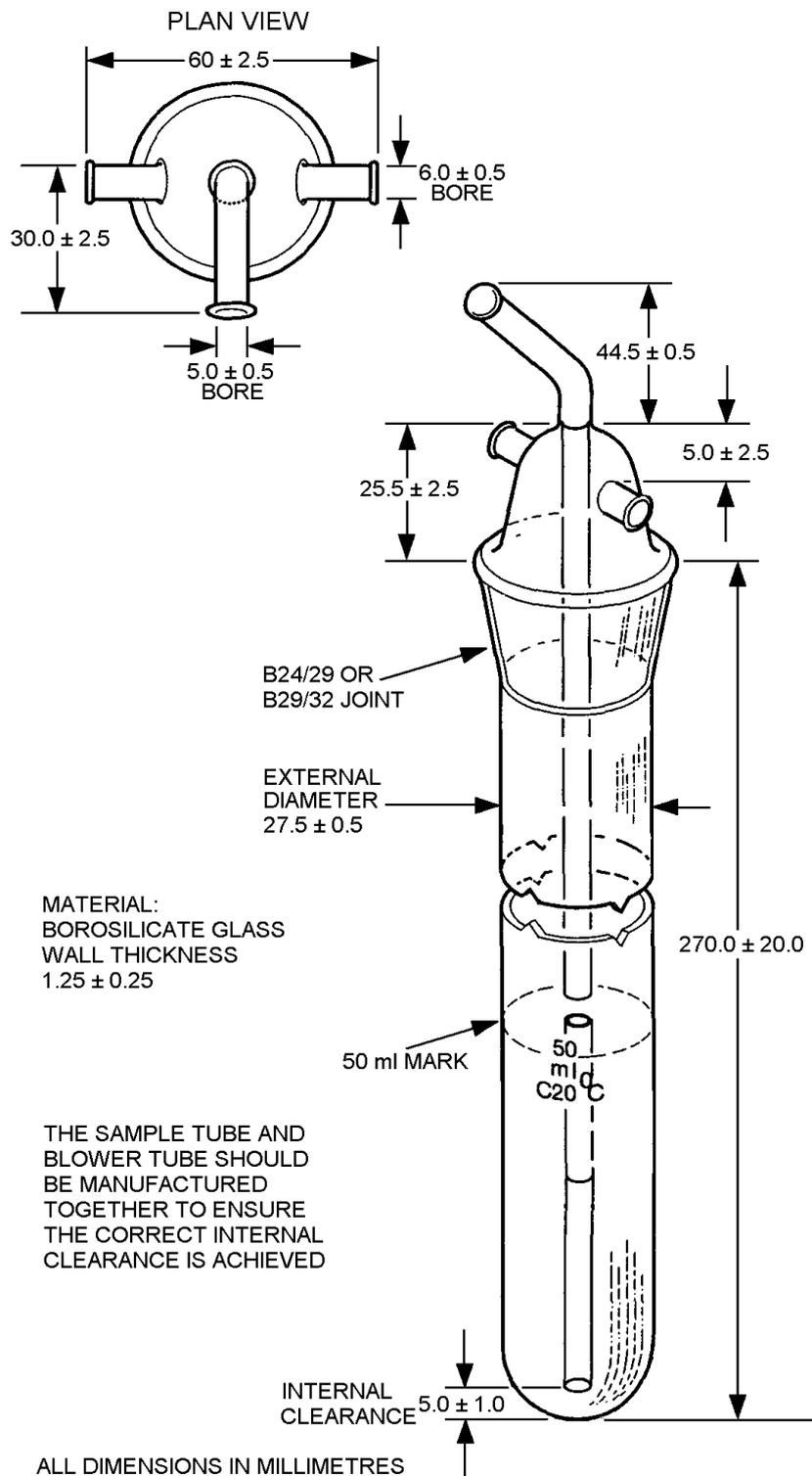
**A.1.3** Contact details for Grant Instruments (Cambridge) Ltd, Shepreth, Cambridgeshire, SG8 6GB, Tel +44 (0)1763 260811, [www.grant.co.uk](http://www.grant.co.uk).

**A.2 TEMPERATURE RECORDING AND CONTROL**

**A.2.1** A temperature controller, an electronic device, capable of maintaining the sample at the specified temperature to  $\pm 1.0$  °C and the temperature difference between any two samples to within 2.0 °C.

**A.2.2** A suitable data logging system should be used to monitor the temperature during tests.

ANNEX B  
DIAGRAMS

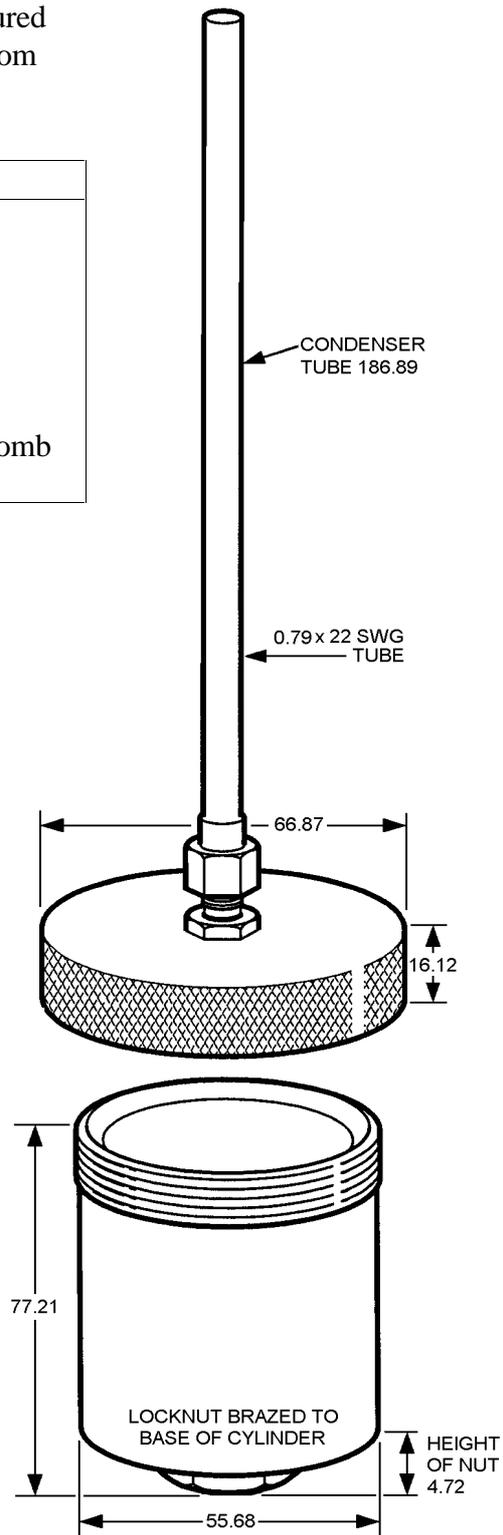


**Fig. 2 Oxidation Apparatus Blowing Tube Assembly**

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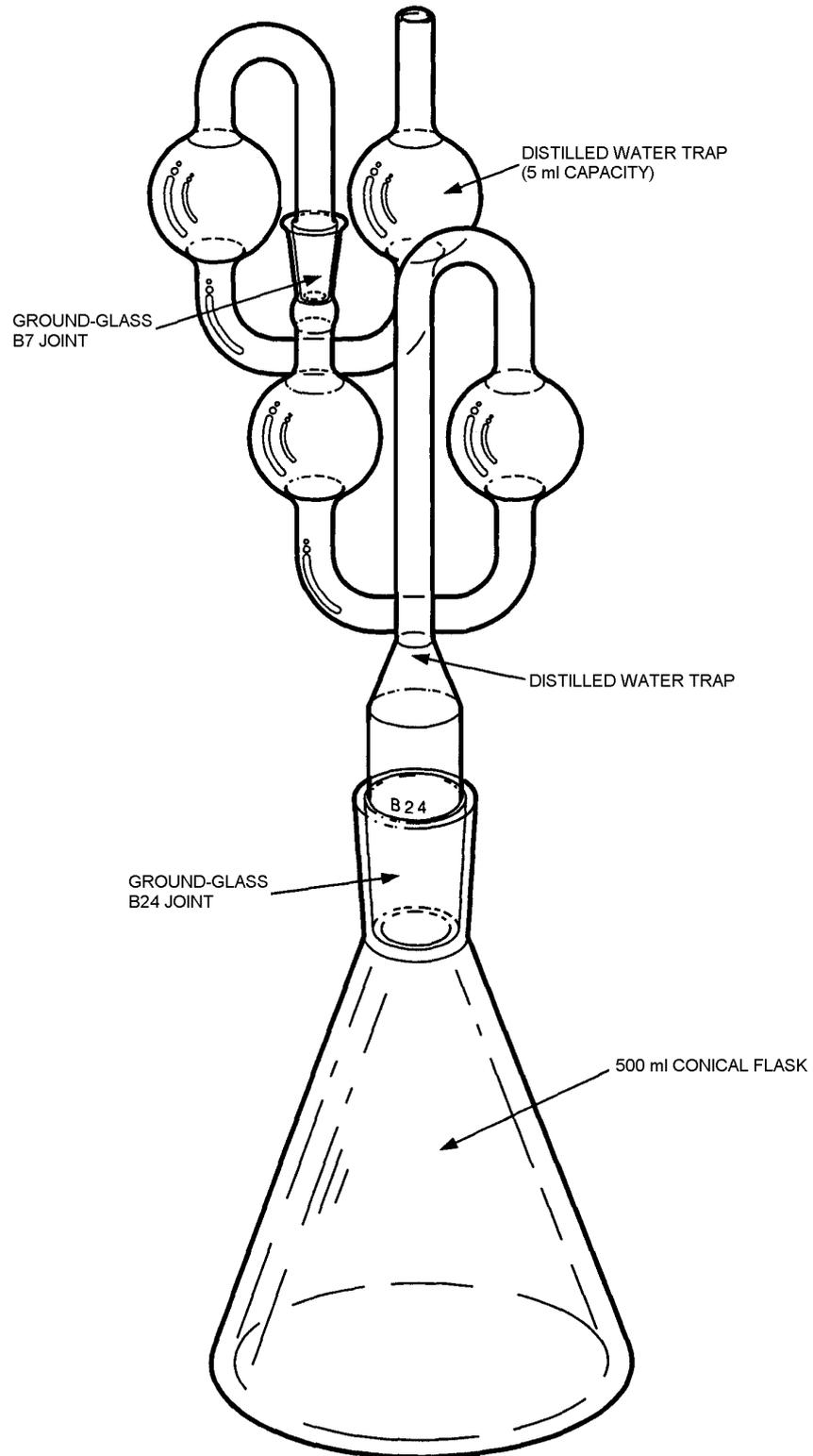
This equipment should only be manufactured according to master drawings available from Rolls Royce PO Box 31 DE2 8BJ

DRAWING NO.	ITEM
LAB 3636	Condenser Tube
LAB 3637	Pipe Nipple
LAB 3638	Single Ended Union
LAB 3639	Union Nut
LAB 3790	Cap
LAB 3641	Container
LAB 3792	Confined Heat Test Bomb General Arrangement

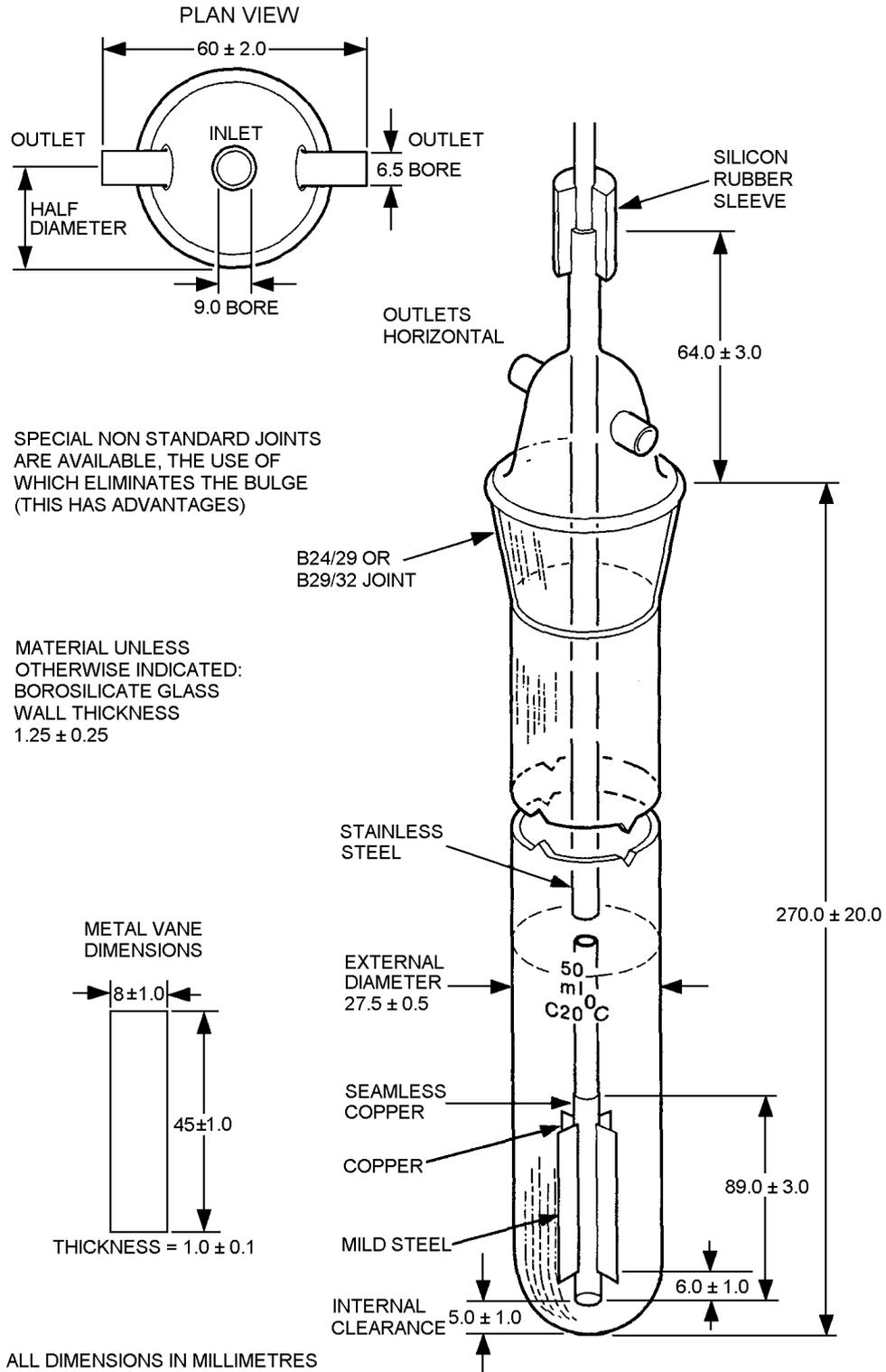


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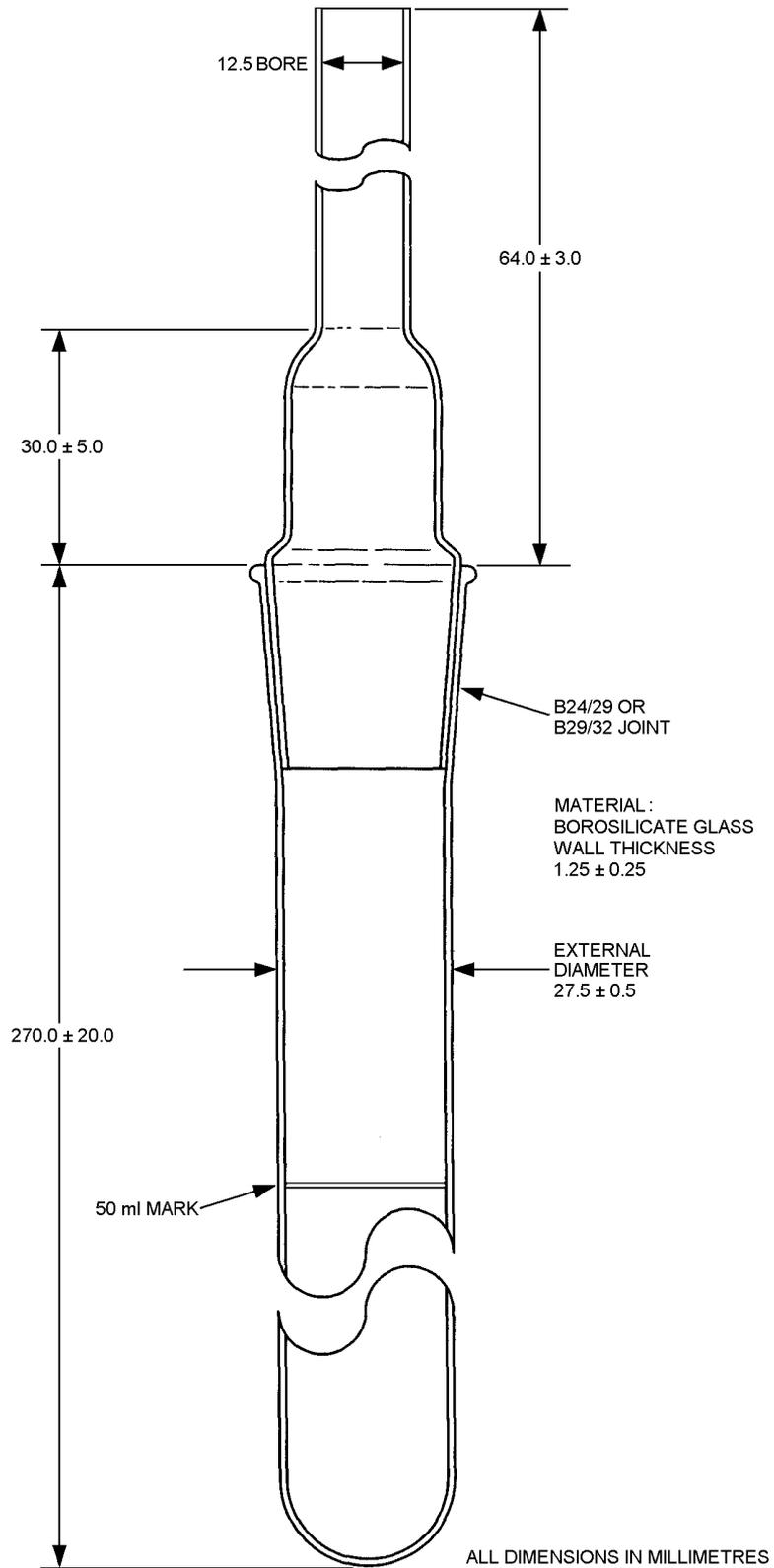
**Fig. 3 Confined Heating Test Vessel and Air Condenser Apparatus**



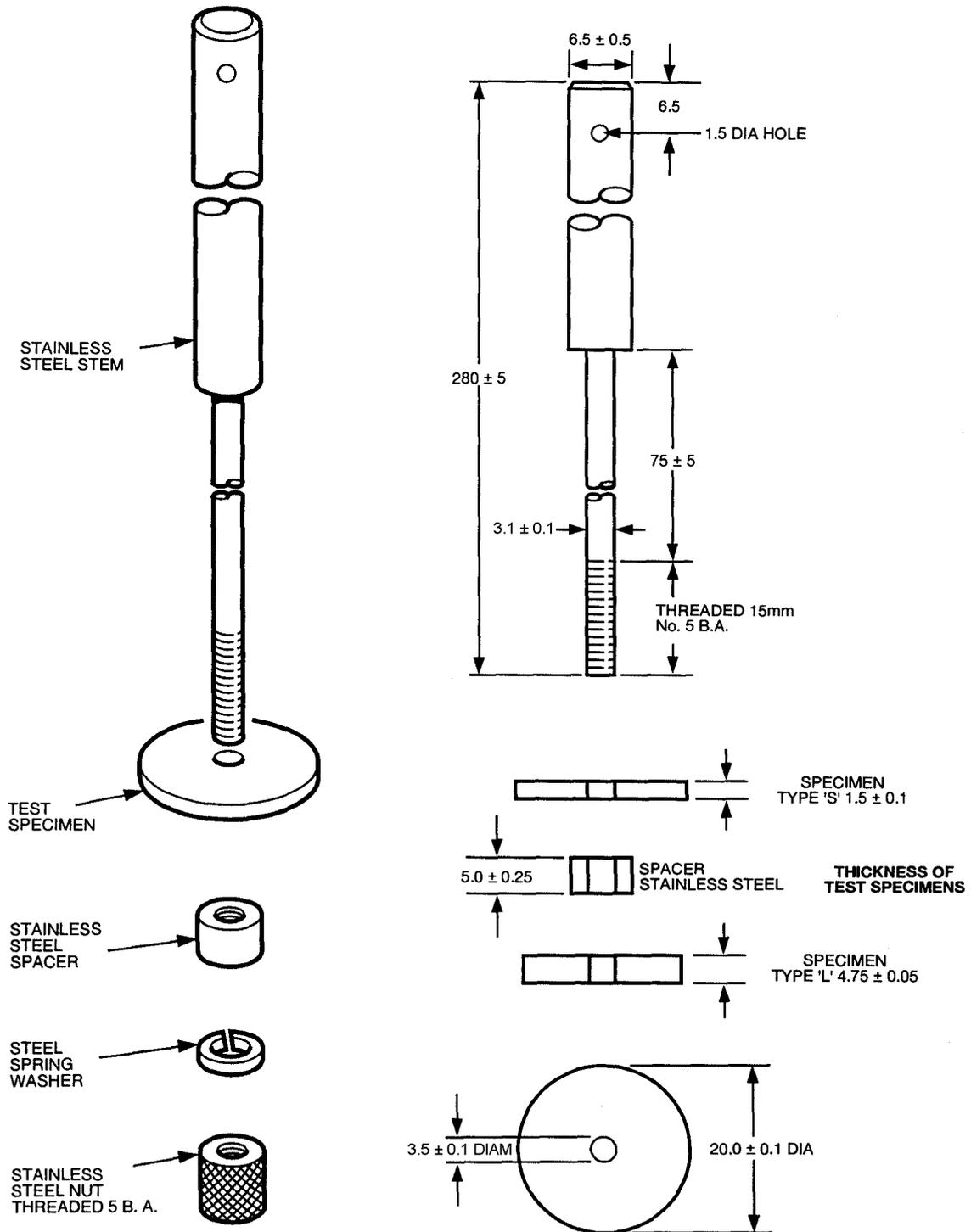
**Fig. 4 Hydrolytic Stability Apparatus**



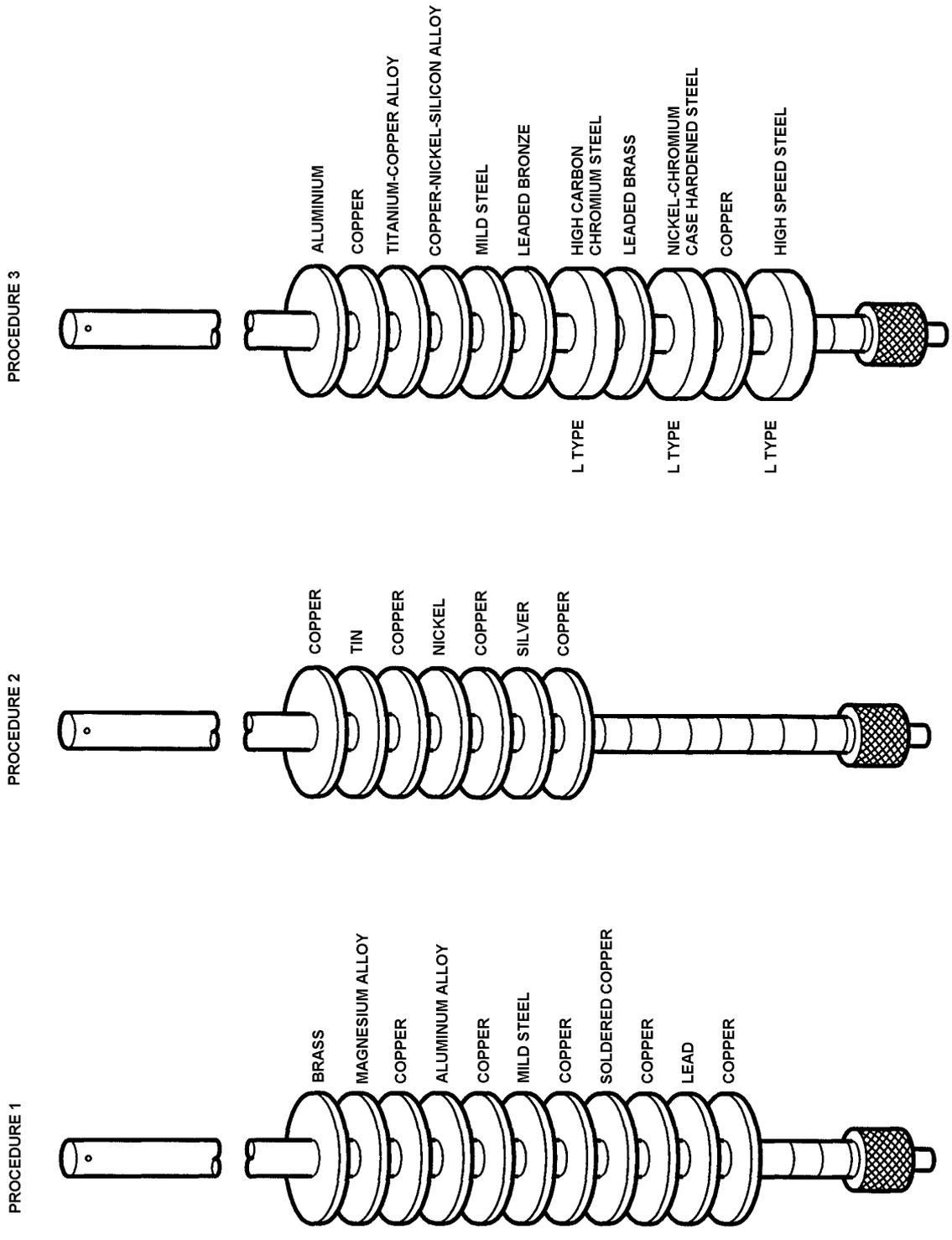
**Fig. 5 Catalytic Oxidation Blowing Tube Assembly**



**Fig. 6 Corrosivity Blowing Tube and Condenser**

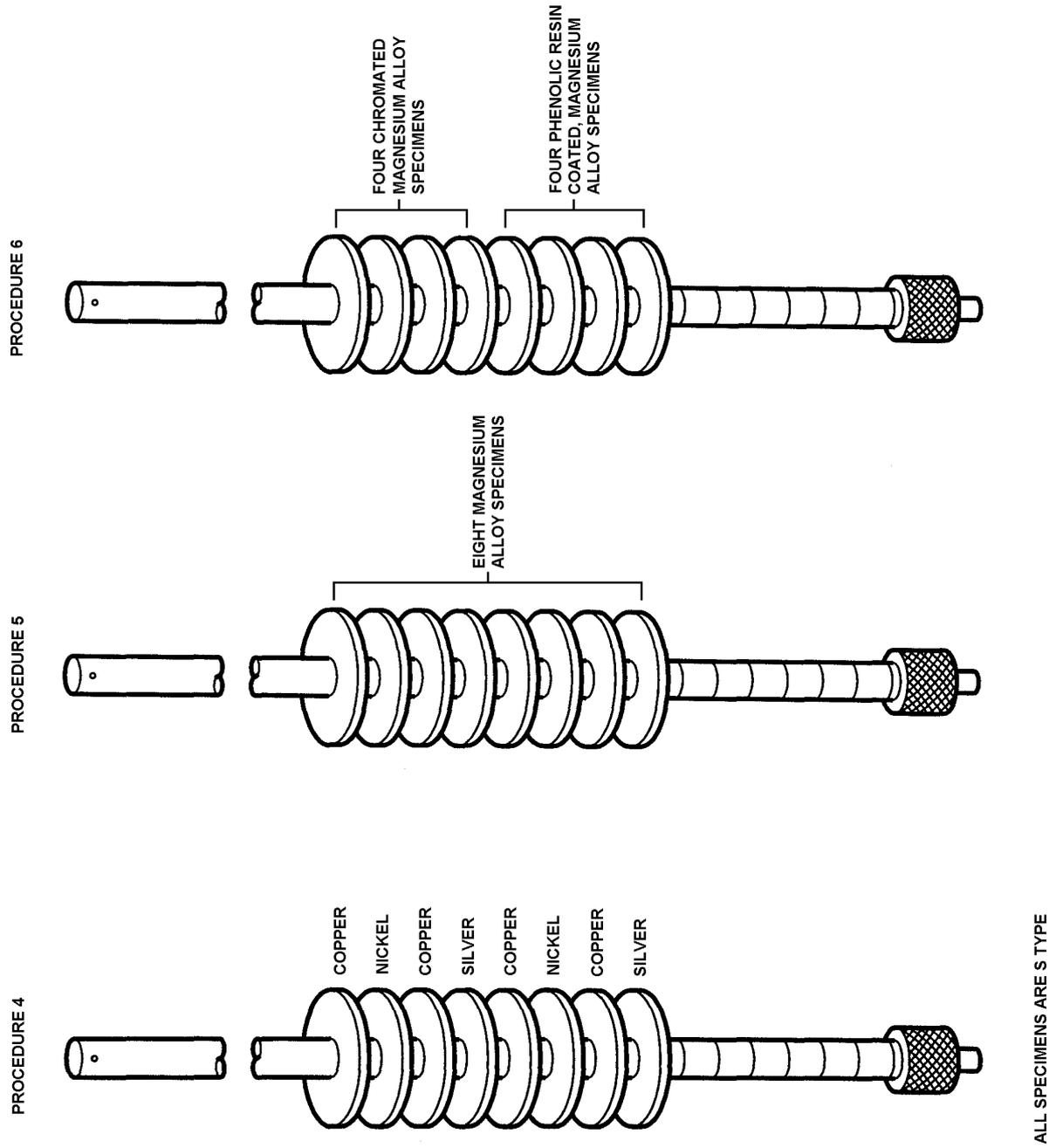


**Fig. 7 Corrosivity Test Assembly, Specimen Holder and Dimensions**

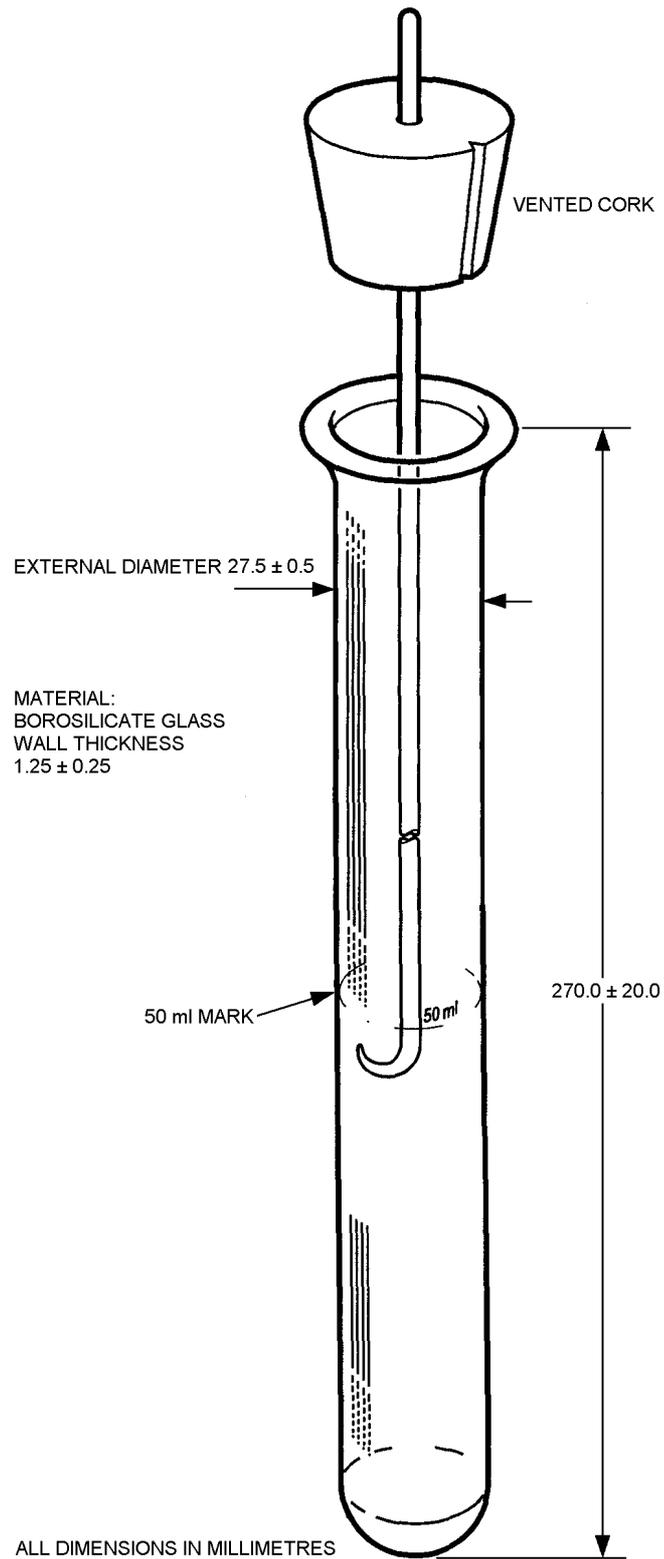


ALL SPECIMENS ARE S TYPE UNLESS OTHERWISE SPECIFIED

**Fig. 8 Corrosivity Test Assembly, Procedures 1, 2, and 3**



**Fig. 9 Corrosivity Test Assembly, Procedures 4, 5 and 6**



**Fig. 10 Elastomer Compatibility Test Tube Apparatus**

ANNEX C

RELATED DOCUMENTS LIST

Designation	Title
ISO 3696	Water for Analytical Laboratory Use. Specifications and Test Methods
BS EN 12167	Copper and copper alloys. Profiles and rectangular bar for general purposes.
BS EN 29453	Soft Solders Alloys. Chemical Compositions and Forms
BS 604	Specification for Graduated Glass Measuring Cylinders
BS EN 13601	Specification for Copper for Electrical Purposes. Rod and Bar
BS 1845	Specification for Filler Metals for Brazing
BS 2000-71.1	Determination of Kinematic Viscosity and Calculation of Dynamic Viscosity
BS 2000-74	Determination Of Water. Distillation Method.
BS EN 12451	Copper and Copper Alloys. Seamless, Round Tubes For Heat Exchangers.
BS EN 13599	Copper And Copper Alloys. Copper Plate, Sheet And Strip For Electrical Purposes.
BS 2S 510	Specification for Carbon Steel Sheet and Strip (430/540 MN/m <sup>2</sup> ) (Suitable for Welding)
BS 2T 66	Specification for 18/10 Chromium-Nickel Corrosion-Resisting Steel Tube (Niobium Stabilized: 550 Mpa) (Weldable)
BS M48	Specification For Dimensions Of Elastomeric Toroidal Sealing Rings For Aerospace Use – Inch Series.
Def Stan 91-100	Lubricating Oil, Gas Turbine, Synthetic Grade 5cSt NATO Code: O-160 Joint Service Designation: OX-26.
ASTM D95	Standard Test Method for Water in Petroleum Products and Bituminous Materials by Distillation
ASTM D445	Standard Test Method for Kinematic Viscosity of Transparent and Opaque Liquids
ASTM D892	Standard Test Method for Foaming Characteristics of Lubricating Oils
ASTM D1091	Standard Test Method for Phosphorus in Lubricating Oils and Additives
IP 71	Kinematic Viscosity and Calculation of Dynamic Viscosity
IP 74	Water Content of Petroleum Products - Distillation Method
SAE AS568	Aerospace Size Standard for O-Rings
SAE ARP 5088	Test Method for the Determination of Total Acidity in Polyol Ester and Diester Gas Turbine Lubricants by Automatic Potentiometric Titration

MSRR 6001	Low alloy steel
MSRR 6009	Nickel-chromium case hardened steel
MSRR 6050	Mild steel
MSRR 7137	Nickel alloy
MSRR 8009	Aluminium alloy
MSRR 8013	Magnesium alloy
MSRR 8501	Copper-nickel-silicon alloy
MSRR 8603	Titanium-copper alloy
MSRR 9450	Fluorocarbon elastomer
MSRR 9482	Low Compression Set Fluorocarbon elastomer
MSRR 9492	Silicone elastomer
MSRR 9495	Nitrile elastomer
MSRR 9504	Lead bronze
Federal Test Method Standard Number 791C	Method 3403 - Compatibility of Turbine Lubricating Oils
Federal Test Method Standard Number 791C	Method 3010 - Solid Particle Contamination in Aircraft Turbine Engine Lubricants (Gravimetric Procedure)
MIL-PRF-23699F	Lubricating Oil, Aircraft Turbine Engine, Synthetic Base, NATO Code Number O-156

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65 Brown Street  
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**DStan Helpdesk**

Tel 0141 224 2531/2  
Fax 0141 224 2503  
Internet e-mail [enquiries@dstan.mod.uk](mailto:enquiries@dstan.mod.uk)

**File Reference**

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