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## **OBSOLESCENCE NOTICE**

All DTD specifications were declared obsolescent from 1<sup>st</sup> April 1999. All DTD 900 series approvals also lapsed at that time. The standards will no longer be updated but will be retained as obsolescent documents to provide for the servicing of existing equipment.

### **Further Guidance**

The aim in declaring the specifications obsolescent is to recognise that the documents are not being updated and thus should be used with care by both purchaser and supplier. For example, a specification could contain valid technical information but may also contain type approval clauses that contradict procurement policy and/or use materials that do not comply with environmental legislation. The obsolescent specification can still be used as a basis for a purchase provided that the supplier and purchaser agree suitable changes to the specification within the purchase order/contract.

For the DTD 900 system, each specification has provided an MoD approved material and process. For these items, the declaration of obsolescence will constitute the termination of both the extant MoD approval and the continuing MoD assessment that had underpinned those approvals. Again, the technical content of the document remains valid and can be used by both purchaser and supplier as a basis for a contract but an acceptable (to the parties) approval/assessment procedure would be required.

**PROCUREMENT EXECUTIVE  
MINISTRY OF DEFENCE**

**D.T.D. 5628**

*Amendment No 1  
January 1984*

*Aerospace Materials Specifications*

**TEST METHODS AND PROCUREMENT PROCEDURES FOR ONE PART  
ANAEROBIC POLYMERISABLE COMPOUNDS FOR LOCKING, SEALING AND  
RETAINING.**

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**Section 1**

**Scope**      *Add new paragraph*

Anaerobic adhesives complying with this specification can cause severe solvent stress crazing in some stressed thermoplastic components and aircraft organic transparency materials, leading to a catastrophic failure.

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## Aerospace Material Specification

### TEST METHODS AND PROCUREMENT PROCEDURES FOR ONE PART ANAEROBIC POLYMERISABLE COMPOUNDS FOR LOCKING, SEALING AND RETAINING

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*NOTE 1 This specification is one of a series issued by the Procurement Executive, Ministry of Defence, either to meet a limited requirement not covered by an existing British Standard for aerospace material, or to serve as a basis for inspection of material, the properties and uses of which are not sufficiently developed to warrant submission to the British Standards Institution for standardization.*

*NOTE 2 The tests employed in this specification are chosen for their reproducibility and ability to control the properties of the material. They are not intended to be simulated service tests, which, because of variability of test conditions, may be unsatisfactory for control purposes.*

*NOTE 3 This specification calls for the use of substances and/or test procedures that may be injurious to health if adequate precautions are not taken. It refers only to technical suitability and in no way absolves either the supplier or the user from statutory obligations related to health and safety at any stage of manufacture or use.*

*This specification has been devised for the use of the Ministry of Defence and its contractors in the execution of contracts for the Ministry and, subject to the Unfair Contract Terms Act 1977, the Ministry will not be liable in any way whatever (including but without limitation negligence on the part of the Ministry, its Servants or agents) where the specification is used for other purposes.*

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## SECTION 1

### Scope

- 1 This specification defines the test methods and procurement procedures for a range of single component liquid compounds which polymerise upon the exclusion of oxygen and activation by a metal surface.

When the compounds are used on metallic surfaces the prior use of activator shall not normally be required. The compounds shall respond to the use of an activator if its use is required by prevailing circumstances, eg on non-metallic surfaces.

General applications of these materials include locking and sealing threaded fasteners and assembling slip-fitted parts. The materials should not normally be used on electrical connections where the assembled connection is required to carry electrical currents. Some of the compounds are compatible with certain explosives and propellants, but the appropriate Design Authority Approval must be obtained before use in proximity to such materials.

## SECTION 2

### Definitions

Activator:	A substance used in small proportions to increase the reaction rate of chemical systems.
Anaerobic polymerisable compound:	A polymerisable composition intended to polymerise spontaneously in the absence of oxygen and the presence of metal ions.
Batch:	The manufactured unit or a blend of two or more units of the same formulation and processing.
Cure:	To increase the cohesive strength by chemical changes.
Fluorescence:	The property of absorbing light of one wavelength and emitting light of another wavelength. The phenomenon ceases when the source of light is cut off.
Shear Strength:	The force necessary to bring an adhesive joint to the point of failure by means of forces applied in a shear mode.
Thixotropy:	A decrease of the apparent viscosity under shear stress followed by a gradual recovery when the stress is removed. The effect is time-dependent.
Thixotropic Index:	The ratio of viscosity measured at one shear rate to that at another shear rate. The shear rates, by convention, differ by a factor of 10; this is achieved by changing the rotational speed of the viscometer by a factor of 10.
Torque Strength:	The force necessary to rotate a nut on a bolt where engaged threads are coated with cured compound. The maximum torque is the highest force recorded during the first revolution after initial movement of a nut on a bolt coated with cured compound. Standard nuts and bolts are used.
Viscosity:	The property of a material to resist deformation increasingly with increasing rate of deformation.

## SECTION 3

### Related Documents

Reference is made in this specification to the following documents:

BS 188	Methods for determination of the viscosities of liquids.
BS 872	Abrasive papers and cloths (technical products).
BS 970	Wrought steels in the form of blooms, billets, bars and forgings.
BS 3643	ISO metric screw threads.
BS 3692	ISO metric precision hexagonal bolts, screws and nuts.
BS 5214	Testing machines for rubbers and plastics.
BS 5350	Methods of test for adhesives.

The related documents listed are those applicable at the date of publication of this specification. Their current applicability must be confirmed by all users of the specification. The Quality Assurance Authority named in the contract will supply, on request, information concerning any changes that may be necessary due to cancellation, replacement, supersession or amendment of any related document.

## SECTION 4

### General Requirements

#### 4.1 Composition

The compounds shall consist essentially of acrylic esters of a polyhydroxy compound. Generally they shall be clear homogenous liquids free from foreign matter and particulate solids. Certain formulations however are suspensions and low viscosity materials containing solids in suspension cannot be prevented from separating over a period of time. For these formulations an instruction to shake vigorously before use must be marked on the container.

#### 4.2 Colour and Visual Appearance

The compounds shall be substantially uniform in colour and shall be tested in accordance with Appendix A; where fluorescence is a requirement compounds shall be tested in accordance with Appendix B. The compounds shall be substantially free from contamination, foreign matter or other defects, and shall be homogeneous.

#### 4.3 Viscosity

Viscosity of the compounds shall be determined in accordance with Appendix C.  
The thixotropic index shall be determined in accordance with Appendix C.

#### 4.4 Torque Strength

- 4.4.1 Torque Strength at  $23 \pm 2^\circ\text{C}$  shall be determined in accordance with Appendix D.
- 4.4.2 Torque strength after 1000 hours at  $100^\circ$  and  $150^\circ\text{C}$  shall be determined in accordance with Appendix E.
- 4.4.3 Torque strength after 168 hours in boiling water shall be determined in accordance with Appendix F.
- 4.4.4 Torque strength at  $-55^\circ\text{C}$  shall be determined in accordance with Appendix G.

#### 4.5 Static Shear Strength

- 4.5.1 Static shear strength at  $23 \pm 2^\circ\text{C}$  shall be determined in accordance with Appendix H.
- 4.5.2 Static shear strength after 1000 hours at  $100^\circ\text{C}$  and  $150^\circ\text{C}$  shall be determined in accordance with Appendix I.

#### 4.6 Stability at $100^\circ\text{C}$ shall be determined in accordance with Appendix J.

#### 4.7 Excessive Cure Rate

The test for excessive cure rate shall be determined in accordance with Appendix K.

#### 4.8 Ability to set on Cadmium

The ability of the material to set on cadmium shall be determined in accordance with Appendix L.

## SECTION 5

### Type Approval

- 5.1 Before any compound is supplied as complying with the requirements of this specification and the relevant technical specification, the supplier must obtain Type Approval. The authority for type approval is The Director of Materials Quality Assurance, HQ Building, Royal Arsenal East, Woolwich, London SE18 6TD.
- 5.2 In applying for type approval the supplier shall submit:
- i a request for approval to a particular specification,
  - ii samples of the compound for which approval is sought,
  - iii test results for the properties listed in the specification for the same batch of material from which the samples were obtained,
  - iv full instructions for use,
  - v details of the composition; these will be regarded as confidential.
- 5.3 All the tests listed in the specification to which approval is sought shall be carried out on the samples provided. Each property of the material, when determined by the method stipulated in the specification, shall comply with the requirement given in the specification.
- 5.4 Any proposed changes whatsoever in the formulation of the compound shall be notified to DMQA, and if considered significant by DMQA, the compound shall be treated as a new product requiring specific Type Approval.

Any proposed changes whatsoever in the processing instructions shall be notified to DMQA, and if considered significant by DMQA the compound shall be treated as a new material requiring specific Type Approval using the new processing instructions.

## SECTION 6

### Shelf Life

When stored in the original unopened containers the material shall comply with the requirements of the specification to which the material is released for a period of at least six months in tropical climates or at least twelve months in temperate climates from the date of despatch by the manufacturer.

Materials stored for periods in excess of these shall be re-tested according to the requirements of clause 4.4.1 and the relevant specification. The storage life of material found to be satisfactory may be extended by 6 months (temperate) or 3 months (tropical). Further extensions may be granted on the same basis.

## SECTION 7

### Packaging

- 7.1 The compound shall be packed in sound, clean and dry containers which after packing shall hold an agreed quantity.
- 7.2 The nature of the container and the ullage after packing shall be such as to prevent premature solidification of the material during storage.
- 7.3 Each container and its matching carton shall be legibly and durably marked as called for by statutory requirements and in addition with the following details:

Container:

Type and grade reference number  
Contractor's initials or recognised trade mark  
Distinctive batch number or identification mark

Carton:

Description viz: COMPOUND, LOCKING AND RETAINING, ONE PART, ANAEROBIC  
Type and grade reference number  
Specification number viz: DTD . . . .  
Distinctive batch number  
Date of despatch  
Contract or order number  
Contractor's initials or recognised trade mark  
Manufacturer's reference for appropriate accelerator  
Additional markings called for in the contract or order  
Where the material contains a suspension which is liable to settle upon standing, instructions for shaking of the container shall be included.

*NOTE: Small bottles (10 cc and smaller) need only have the type, grade and batch numbers on the bottle - all other markings to be on the carton.*

## APPENDIX A

### Test for Colour

#### 1 Procedure

Place one drop of the compound on clean white paper or a clean glazed white tile and examine it visually in normal daylight.

## APPENDIX B

### Test for Fluorescence

#### 1 Apparatus

- a. One ultraviolet lamp
- b. One piece of aluminium strip or sheet approx 5 cm x 2 cm
- c. One microscope slide

#### 2 Procedure

Apply one or two drops of compound to one end of the aluminium strip and spread over the entire surface using the microscope slide. Illuminate the prepared strip with the ultraviolet lamp. The compound shall be seen to fluoresce.

## APPENDIX C

### Determination of Viscosity and Thixotropic Index

#### 1 Procedure

In all cases the viscosity shall be measured at  $25^{\circ}\text{C} \pm 0.1^{\circ}$ .

- 1.1 For Newtonian materials the viscosity shall be measured in a calibrated U-tube viscometer in accordance with BS 188.
- 1.2 For thixotropic materials the viscosity shall be determined by a procedure based on Method 1 of BS 5350 Part B8 but using the small sample adaptor with spindle SC4-27/13 at 20 rpm and 2 rpm. The viscometer shall be allowed to run for  $10 \pm 1$  minutes before the measurement. The ratio of the viscosity at 2 rpm to that at 20 rpm shall be stated as the thixotropic index.

Other methods and apparatus may be used with the agreement of the quality assurance authority.

## APPENDIX D

### Determination of Torque Strength

#### 1. Apparatus

- a. Six nuts and bolts to specification in Appendix M.
- b. Atmosphere thermostatically controlled at  $23 \pm 2^{\circ}\text{C}$ .
- c. Suitable means for unscrewing a bonded nut and bolt set at a substantially constant speed of 12 rpm and for indicating the applied torque.
- d. Petri dish.

#### 2 Procedure

Choose 6 nuts and 6 bolts with undamaged threads, and not having excessively tight or loose fits.

Rinse the nuts and bolts 3 times in cold 1, 1, 1, -trichloroethane and allow to dry. Alternatively, vapour degreasing may be used and the nuts and bolts allowed to cool to  $23 \pm 2^{\circ}\text{C}$ .

Avoid subsequent contamination of the nuts and bolts by fingers etc.

Fill a suitable size Petri dish to a depth of about 10 mm with the compound and completely immerse the nuts. Coat the entire thread of each bolt with the compound and immediately screw into one of the immersed nuts until the threads of the nut are fully engaged. Remove the assembly from the Petri dish and continue screwing the bolt into the nut until at least 3 but not more than 5 complete turns of the bolt thread protrude beyond the edge of the nut.

Place the 6 assemblies horizontally on a sheet of polyethylene ensuring that individual sets are not in contact. Maintain at  $23 \pm 2^{\circ}\text{C}$  for  $24 \pm 1$  hours.

At the end of this period unscrew the nuts from the bolts at a substantially constant speed of 12 rpm. Allow at least 1½ rotations of the nut to occur. Record the highest torque attained during the first revolution. The mean of the 6 results for each measurement shall be reported as the maximum torque.

## APPENDIX E

### Determination of Retention of Torque Strength After 1000 Hours at 100 and 150°C

#### 1 Apparatus

As at Appendix D plus an oven thermostatically controlled at  $100 \pm 1^\circ\text{C}$  or  $150 \pm 2^\circ\text{C}$ .

#### 2 Procedure

Prepare 6 nut and bolt assemblies as at Appendix D for each test temperature.

At the end of the  $24 \pm 1$  hour period at  $23 \pm 2^\circ\text{C}$  remove any surplus uncured compound from the assemblies and place them in the oven at the required temperature for 1000 hours. At the end of this period remove the assemblies from the oven, Allow to cool at  $23 \pm 2^\circ\text{C}$  for  $2 \pm \frac{1}{2}$  hours and test as at Appendix D.

Record the results as at Appendix D and express the mean value for maximum torque as a percentage of the corresponding value obtained in the test at Appendix D.

## APPENDIX F

### Determination of Retention of Torque Strength After 168 hours in Boiling Water

#### 1 Apparatus

As at Appendix D plus suitable apparatus to maintain distilled water boiling under reflux at normal atmospheric pressure for the required period.

#### 2 Procedure

Prepare 6 nut and bolt assemblies as at Appendix D.

At the end of the  $24 \pm 1$  hour period at  $23 \pm 2^\circ\text{C}$  remove any surplus uncured compound from the assemblies and immerse them in the boiling distilled water for  $168 \pm 2$  hours. Ensure that the temperature of the boiling distilled water does not fall below  $95^\circ\text{C}$ . At the end of this period remove the assemblies from the water and allow them to cool in air at  $23 \pm 2^\circ\text{C}$  for  $2 \pm \frac{1}{2}$  hours.

Test the assemblies and record the results as at Appendix D and express the mean value for maximum torque as a percentage of the corresponding value obtained in the test at Appendix D.

## APPENDIX G

### Determination of Retention of Torque Strength at $-55^\circ\text{C}$

#### 1 Apparatus

As at Appendix D plus a refrigerator or other suitable means of maintaining the assemblies at  $-55 \pm 2^\circ\text{C}$  for  $120 \pm 10$  minutes.

#### 2 Procedure

Prepare 6 nut and bolt assemblies as at Appendix D.

At the end of the  $24 \pm 1$  hour period at  $23 \pm 2^\circ\text{C}$  remove any surplus uncured compound from the assemblies and place them in the refrigerator at  $-55 \pm 2^\circ\text{C}$  for  $120 \pm 10$  minutes.

At the end of this period remove the assemblies from the refrigerator individually and test them as at Appendix D within 30 seconds of removal from the refrigerator. Express the mean value for maximum torque as a percentage of the corresponding value obtained in the test at Appendix D.

## APPENDIX H

### Determination of Static Shear Strength at $23 \pm 2^\circ\text{C}$

#### 1 Apparatus

- a. Six collar and pin sets complying with the requirements of Appendix N and having 0.025 - 0.05 mm diametrical clearance.
- b. Atmosphere thermostatically controlled at  $23 \pm 2^\circ\text{C}$ .
- c. Petri dish.
- d. A tensile test machine complying with the requirements of BS 5214: Part 1, grade B, suitably equipped to carry out compression testing.

**2 Procedure**

Rinse the collars and pins 3 times in 1, 1, 1-trichloroethane and allow to dry.

Avoid subsequent contamination of the collars and pins by fingers etc.

Alternatively vapour degreasing may be used, with subsequent cooling to  $23 \pm 2^\circ\text{C}$ .

Fill a suitable size Petri dish to a depth of about 10 mm with the compound. Coat the entire surface of each pin and each collar with the compound. Immediately insert a pin into each collar. Place the 6 assemblies vertically on a sheet of polyethylene using a suitable means (eg 2 strips of polyethylene about 6.5 mm thick) to support the collars approximately at the mid-points of the pins. Maintain the assemblies at  $23 \pm 2^\circ\text{C}$  for  $24 \pm 1$  hours and then remove surplus uncured material.

At the end of this period use the test machine and compression jig to push the pin out of the collar. The rate of loading shall be constant and such that the assembly fails 20-90 seconds after the commencement of loading. Record the maximum load achieved.

**3 Calculation**

Divide the mean of the 6 results by the internal surface area of the collars and report as the static shear strength in MPa at  $23 \pm 2^\circ\text{C}$ .

**APPENDIX I****Determination of Retention of Static Shear Strength  
After 1000 Hours at 100 and 150°C****1 Apparatus**

As at Appendix H plus an oven thermostatically controlled at  $100 \pm 1^\circ\text{C}$  or  $150 \pm 2^\circ\text{C}$ .

**2 Procedure**

Prepare 6 collar and pin assemblies as at Appendix H.

At the end of the  $24 \pm 1$  hour period at  $23 \pm 2^\circ\text{C}$  remove any surplus uncured compound from the assemblies and place them in the oven at the required temperature for 1000 hours.

At the end of this period remove the assemblies from the oven, allow to cool at  $23 \pm 2^\circ\text{C}$  for  $2 \pm \frac{1}{2}$  hrs and test as at Appendix H.

**3 Calculation**

Divide the mean of the 6 results by the internal surface area of the collars to obtain the static shear strength in kPa after ageing at 100 or 150°C.

**Reporting**

Express this result (para 3) as a percentage of the result obtained for the same compound in the test as  $23 \pm 2^\circ\text{C}$  (see Appendix H).

**APPENDIX J****Determination of Stability at 100°C****1 Procedure**

Place about 5g of material in a test tube of approximately 25 mm internal diameter. Place the tube in boiling water and stir the contents gently and continuously with a glass rod.

Record the time in minutes from the immersion of the tube for the material to gel or for lumps to form. If polymerization has not occurred after 10 minutes discontinue the test and record the stability time as greater than 10 minutes. Perform the determination in duplicate and report the lower of the 2 results as the stability time at 100°C.



**APPENDIX K****Test for Excessive Cure Rate****1 Apparatus**

- a. Nuts and bolts - M8. bright mild steel, bar turned, to specification in Appendix M.
- b. Atmosphere thermostatically controlled at  $23 \pm 2^\circ\text{C}$ .
- c. Petri dish.
- d. Tweezers.

**2 Procedure**

Choose 6 nuts and 6 bolts with undamaged threads, and not having excessively tight or loose fits.

Rinse the nuts and bolts 3 times in cold 1,1,1-trichloroethane and allow to dry. Alternatively, vapour degreasing may be used with subsequent cooling to  $23 \pm 2^\circ\text{I}$ .

Avoid subsequent contamination of the nuts and bolts by fingers etc. Place the nuts and bolts on polyethylene sheet and stabilise at  $23 \pm 2^\circ\text{C}$  for 30 minutes.

Fill a suitable size petri dish with sufficient adhesive to completely immerse the nuts. Coat the entire thread of each bolt with adhesive by immersing it in the adhesive and then immediately screw it into one of the nuts until at least 3 but not more than 5 complete turns of the bolt thread protrude beyond the edge of the nut. Place the 6 specimens horizontally on polyethylene sheet and ensure that individual sets are not in contact. Maintain at  $23 \pm 2^\circ\text{C}$  for the time specified.

At the end of this period examine the assembled components. All the nuts should still be free.

**APPENDIX L****Determination of Ability to Set on Cadmium Surfaces****1 Apparatus**

- a. Two blocks, cadmium (99.99%), approximately 100 x 25 x 25 mm.
- b. Atmosphere thermostatically controlled at  $23 \pm 2^\circ\text{C}$ .
- c. Stop Clock.
- d. Solvent - methanol or IPA analytical reagent quality.
- e. Abrasive paper, silicon carbide, waterproof, Grade 600 complying with BS 872.
- f. Balance, spring, 50N accurate to  $\pm 0.5\text{N}$ .

**2 Procedure**

Place a sheet of the silicon carbide paper on a sheet of glass and wet thoroughly with distilled water.hone one 100 x 25 mm surface of each of the cadmium blocks by moving back and forth on the abrasive surface, applying no downward pressure other than the weight of the block. Ensure that the paper is constantly wet and continue abrading until the metal surface is free from scratches and foreign material. Wash the blocks thoroughly in distilled water, wipe off excess water with clean tissues and rinse in the solvent. Finally polish the prepared surfaces with clean tissues.

Place the prepared blocks in the atmosphere at  $23 \pm 2^\circ\text{C}$  for approximately 30 minutes, polished surfaces uppermost. Apply 2 drops of the material under test to the centre of one polished surface. Gently apply the second block, prepared surface downwards onto the material and at right angles across the first block to form a cross. Start the stop clock. Maintain the assembly at  $23 \pm 2^\circ\text{C}$  for the time specified. At the end of this period separate the blocks by means of the following procedure. Grip one block in a vice or similar type of clamp. Apply a tensile force to the other block via a spring balance or other force measuring device; the force is in the plane of the cross and at right angles to the clamped block.

Note the applied force at which the blocks separate. If this force is greater than 25 N the material shall be reported as having set upon a cadmium surface within the number of minutes for which the material was cured.

*NOTE: As an alternative to blocks of cadmium, compound blocks made by facing steel blocks with cadmium strip of minimum thickness 0.25 mm may be used. These should have the cadmium surfaced face of the specified dimensions ie 100 mm x 2.5 mm, and the depth should be such that the blocks are approximately the same mass as blocks of cadmium. The honed and polished surface must be the cadmium surface.*

**APPENDIX M****Specification for Nuts and Bolts**

- Material: Mild Steel 220M07 (lead) complying with BS 970: Part 1.
- Cutting Oil: An oil which is soluble in 1, 1, 1-trichloroethane and does not contain lanolin. (For example Syncut 35 and Finasol 9B2186 are suitable oils.)
- Bolts: 25 mm M8 x 1.25-6 g (BS 3643: Part 2) bright mild steel bar turned sets.
- Nuts: M8 x 1.25-6H (BS 3643 : Part 2) double chamfer bright mild steel bar turned full nuts (BS 3692).
- NOTE: Experience has shown that the chemical nature of the surfaces of the nuts and bolts and the method of manufacture influence the rate of cure and ultimate performance of anaerobic compounds. Therefore, in order to obtain maximum practical reproducibility the method of manufacture and the materials are specified. However, for routine testing commercially available nuts and bolts may be used at the suppliers discretion. Nuts and bolts to the above specification will be used for type approval and in cases of dispute.*

**APPENDIX N****Specification for Collars and Pins**

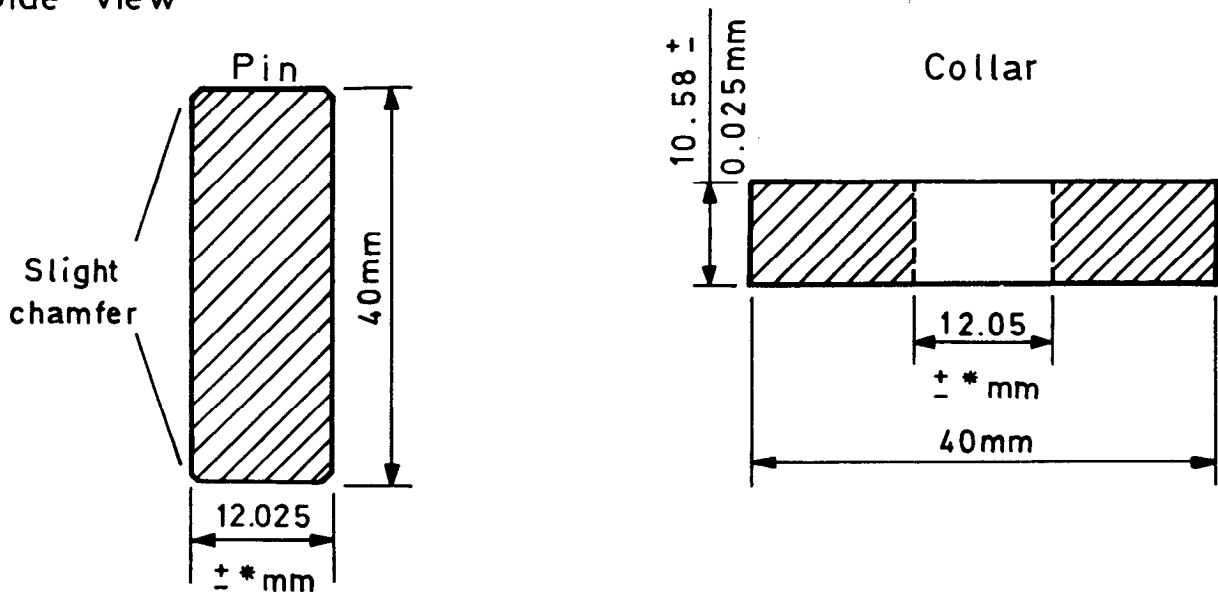
- Material: Mild steel 220M07 complying with BS 970, Part 1.
- Cutting Oil: An oil which is soluble in 1, 1, 1-trichloroethane and does not contain lanolin. (For example Syncut 35 and Finasol 9B2186 are suitable oils.)
- Dimensions: See Fig 1.
- Finish: See Fig 1.
- Note: Re-usability. Except for type approval, when new sets of collars and pins shall be used, collars and pins may be used up to three times provided the surface remains satisfactory.*

Approved for issue:

D. K. THOMAS

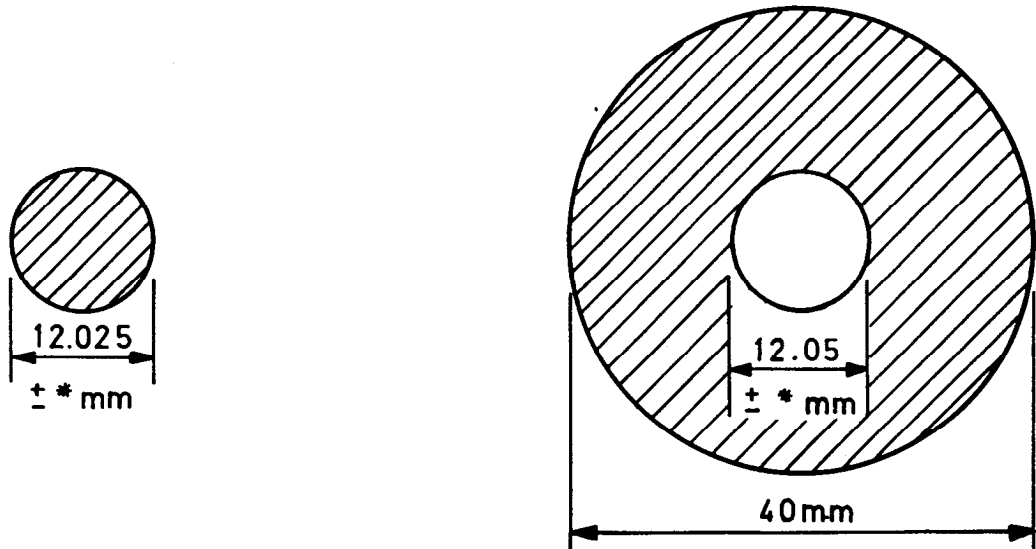
Head of Materials Department,  
Royal Aircraft Establishment, Farnborough, Hants.

Side view



Not to scale

End view



All dimensions in mm

- \* Collar and pins should be made in pairs such that the diametrical clearance is 0.025 to 0.05mm

N.B. 1 Where no tolerance is given, this dimension is not critical

- 2 The outside surface of the pin and inside hole of the collar should have a surface finish of 0.4 to 0.8 $\mu$ m (16-32 $\mu$ in)

Fig 1 Collar and pin test specimens

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