# D.T.D.911C

Ministry of Defence Defence Procurement Agency, ADRP2 Abbey Wood Bristol BS34 8JH

# **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.

# **D.T.D.** 911C

(Superseding D.T.D. 911B) May, 1963 Reprinted March, 1969

### **Process Specification**

## **PROTECTION OF MAGNESIUM-RICH ALLOYS** AGAINST CORROSION

#### Section I. Scope and general observations

#### 1.1. General

1.1.1 All magnesium-rich alloy parts shall be prepared and given a complete protective treatment of cleaning, chromate treatment, sealing and painting in accordance with this specification. Other methods may be used subject to prior approval by the Director of Materials Research and Development/Aviation.

1.1.2. The complete treatment involves operations on the material in its semi-finished form as a casting, forging, extrusion or sheet, with further operations after all forming and machining operations have been completed.

1.1.3. The standards of organic finishing laid down are minima. Extra coats of paint and/or erosionresisting coatings should be applied on parts subject to severe exposure to corrosion or mechanical damage.

#### 1.2. Safety precautions

Some of the chemicals used in the treatments described in this specification, notably hydrofluoric acid, ammonium bifluoride, chromic acid and selenious acid, are dangerous to health, and suitable precautions must be taken.

#### 1.3. General factors affecting corrosion resistance

In order that magnesium alloys shall show the highest resistance to corrosion it is important that:

- (a) The alloy itself should contain no entrapped flux and should have only a low content of impurities; (b) The surfaces of parts shall not be contaminated by flux, sand, or other residues from the casting process, or by metallic or other impurities which may be introduced during working processes such as die casting, rolling or forging or during abrasive blasting. Precautions shall be taken during machining to avoid smearing other metals, e.g., from machining residues in cutting fluids, into the surface;
- (c) No edges or asperities on which the thickness of the subsequently applied organic coatings would be less than required shall be present on the parts. Rough surfaces shall be made smooth, and sharp edges shall be radiused, preferably to 0.030 in or more. The edges of sheet shall be completely rounded.

#### Section II. Cleaning, chromate treatment and surface sealing

All parts shall be treated as described below : \_\_\_\_

#### 2.1. Castings, as cast

2.1.1. Castings other than certain die castings (see Clause 2.1.2.) shall be rough cleaned by blasting with steel or non-metallic grit, or by machining or scurfing, or by chemical milling or by immersion in acid pickling solutions.

2.1.2. Die castings which are not contaminated with sand may not need to be rough cleaned as required by Clause 2.1.1.

2.1.3. With the exception of castings which are to be machined all over, all castings shall then be

2.1.5. With the exception of castings which are to be machined all over, all castings shall then be cleaned by fluoride anodising by the method described in Appendix I. 2.1.4. The fluoride film left by the cleaning process shall then be removed by immersing the parts for up to 15 minutes in a boiling 10 to 15 per cent (w/v) solution of chromic acid ( $CrO_3$ ) followed by immersion either for 10 minutes in a boiling 5 per cent (w/v) solution of caustic soda or for 5 minutes in a cold 15 to 20 per cent (w/v) solution of hydrofluoric acid. Removal of the fluoride film is not, however, necessary prior to treatment in the acid chromate bath, Bath (iv) of Appendix II. 2.1.5. The castings shall then be chromate treated by one of the methods described in Appendix II.

2.1.6. Castings which are to be machined all over shall, after machining, be treated in accordance with Clause 2.2. Application of an approved temporary protective may be necessary for protection during delivery and storage prior to machining. 2.1.7. Castings other than those which are to be machined all over shall be given a surface sealing the treatment to DTD. DTD. 5562 or her other matched are to D.

treatment to D.T.D. 935 using resins to D.T.D. 5562, or by other methods approved by D. Mat/ Aviation.

2.1.8. Castings which are not to be machined at all shall then be painted as described in Section III.

NOTE: Impregnation of porous castings must be carried out prior to the final surface sealing described in Clause 2.2.3. The resin used must be compatible with prior and subsequent surface sealants.

#### 2.2. Castings, finish machined

2.2.1. Castings shall be thoroughly degreased by immersion in a suitable alkaline metal cleaning solution, preceded if necessary by treatment with an organic solvent such as trichloroethylene.

2.2.2. Castings shall then be chromate treated by one of the methods described in Appendix II. The cleaning and chromate treatment will not affect the stoved sealing resin remaining on unmachined areas.

2.2.3. Castings shall then be given a further surface sealing treatment, preferably all over, but at least over machined faces and adjacent areas. The treatment should preferably be the full treatment to D.T.D. 935 using resins complying with D.T.D. 5562. If, however, the full treatment would adversely affect tolerances, the treatment may be reduced to two or, in exceptional circumstances, to one coat of resin to D.T.D. 5562 applied accordingly to D.T.D. 935, or to one coat of resin approved by D. Mat./Aviation which is suitable for stoving at a selected lower temperature. 2.2.4. Castings shall then be painted as described in Section III.

### 2.3. Forgings

2.3.1. With the exception of parts which are to be machined all over, all forgings shall be cleaned by fluoride anodising, the fluoride film removed, and the parts chromate treated and given a surface sealing treatment as in Clauses 2.1.3. to 2.1.7.

2.3.2. After machining operations have been carried out, forgings shall be treated in the same way as castings, finished machined, according to Clauses 2.2.1. to 2.2.4.

#### 2.4. Sheet and extrusions

2.4.1. Sheet and extrusions shall be degreased by immersion in a suitable alkaline metal cleaning solution, preceded if necessary by treatment with an organic solvent such as trichloroethylene, and shall then be cleaned, preferably by fluoride anodising followed by removal of the fluoride film as in Clauses 2.1.3. and 2.1.4., or by acid pickling or by scouring with pumice powder applied with a moist rag.

2.4.2. The sheet and extrusions shall then be chromate treated by one of the methods described in Appendix II.

2.4.3. Sheet and extrusions shall then be given a surface sealing treatment to D.T.D. 935 using resins to D.T.D. 5562.

2.4.4. After having been machined (machining includes cutting, drilling, and radiusing of edges), sheet and extrusions shall be treated in the same way as Castings, finished machined, as in Clauses 2.2.1. to 2.2.4.

2.4.5. For hot-formed sheet, a single sequence of degreasing, cleaning, chromate treatment and (full) surface sealing may be given to sheet after hot forming and machining.

2.4.6. The requirements given in Clause 3.3. for touching up during assembly will be particularly applicable to sheet.

#### Section III. Painting and assembly

#### 3.1. General requirements

3.1.1. All parts shall, after final surface sealing, be degreased if necessary and may be lightly abraded and shall then be painted as described in Clause 3.2. It is preferable that one or more coats of primer be applied prior to assembly, due regard being paid to tolerances on mating surfaces. Whatever the sequence, a coating consisting of at least one coat of primer and one of finish shall be applied overall after assembly.

#### 3.2. Standard paint schemes

3.2.1. The paint scheme shall consist of not less than one coat of chromated primer and not less than one finishing coat to a specification allowed by the Design Requirements for the parts. An all-epoxy cold curing scheme to D.T.D. 5555 is preferred, but on parts containing inserts of aluminium alloy and/or cadmium plated metals a paint scheme based on an etch primer is equally recommended. *NOTE:* Stoving will improve the performance of the D.T.D. 5555 scheme, but will render it very diffi-

*NOTE:* Stoving will improve the performance of the D.T.D. 5555 scheme, but will render it very difficult to remove later, should this need to be done.

3.2.2. The minimum thickness of the total organic coating shall be 0.004 in; a thicker coating may be required by the Approving Authority.

3.2.3. The primer, besides conforming to the appropriate paint scheme specification, shall contain in the dried film not less than 15 per cent by weight of a chromate pigment which shall be either zinc chrome to B.S. 389: 1963, Type 2 or pure strontium chromate or pure zinc monoxy chromate. (The strontium chromate may contain up to 10 per cent of pure barium chromate if desired.) Zinc tetroxy chromate is not acceptable except in the etch primer.

3.2.4. The primer shall be free from compounds of mercury and lead. Pigments used in conjunction with the chromate shall be chosen from among the following :— lithopone, barium sulphate, barium chromate, titania, zinc oxide, kaolin, green oxide of chromium, magnesium carbonate, asbestine, talc, mica, silica and aluminium stearate. These pigments, and any lakes, dyes, etc., shall be free from soluble salts. Other pigments may be used with the prior approval of D. Mat/Aviation.

#### 3.3. Local treatment of exposed metal

3.3.1. Parts on which the protective coating is damaged during assembly should preferably be returned for chromate treatment, sealing and painting as in Clauses 2.2.1. to 2.2.4. Where this is not possible the exposed bare metal shall be treated as follows: 3.3.2. Where there is no danger of entrapment of treatment chemicals, the exposed area shall be treated as in Appendix III, "Repair of chromate films", followed by a full painting scheme as in Clause 3.2.1., but with not less than two coats of primer.

3.3.3. Where there is danger of entrapment of treatment chemicals, the bare metal shall be treated as follows :

- (i) *Mating surfaces.* These shall be given no treatment, but shall be wet-assembled and over painted after assembly with not less than one coat of primer and two coats of finish overall.
- (ii) *Non-mating surfaces.* These shall be painted with an etch primer or epoxy primer followed by the coats comprising the main paint scheme for the parts.
- *NOTE.* The procedures given in Clauses 3.3.2. and 3.3.3. omit sealing with a stoved resin and give substandard protection. Where these procedures cannot be avoided, every effort must be made to improve the standard of protection by the use of multiple coats of paint.

#### 3.4. Assembly

3.4.1. It is particularly important that the general requirement for wet assembly with approved jointing compound shall be rigorously met. The compound selected must not crack at any temperature the components will meet, either during any light stoving treatment given to the subsequent paint coating, or during service. Curing of the compound and curing of subsequently applied paint must not interfere with one another.

3.4.2. It is also particularly important that the appropriate requirements for the application of sacrificial metal coatings, usually cadmium plating, to carbon and low alloy steels, corrosion-resisting steels, copper and its alloys and titanium and its alloys shall be met where these materials are in contact with magnesium alloys.

3.4.3. The faces of all external joints should be caulked or filleted after assembly so as to fill any recesses or crevices where moisture might collect, e.g., round the heads of bolts and round nuts. The caulking compound should contain a chromate pigment and must be adherent and remain flexible throughout its life.

#### Section IV. Reclamation of corroded parts

### 4.1 Superficial corrosion, local repair in situ

4.1.1. Where the corrosion is of a superficial nature and occurs in a few isolated spots only, loose or damaged paint shall be scraped away from the immediate vicinity of the spot and, if possible, the corrosion removed by brushing with a stiff non-metallic brush (e.g., nylon) or by light blasting with non metallic grit.

4.1.2. Where there is no danger of entrapment of treatment chemicals, the corroded area shall then be cleaned chemically by swabbing with a solution containing 10 per cent by weight of chromic acid ( $CrO_3$ ) and 0.1 per cent by volume of sulphuric acid (sp.gr. 1.84) followed by rinsing with several changes of clean water. The process shall be repeated until no corrosion product remains. The cleaned spot shall then be treated as in Appendix III, and painted as in Clause 3.2.1., but with not less than two coats of primer.

4.1.3. Where there is danger of entrapment of treatment chemicals, the area shall be painted with an etch primer or epoxy primer followed by the coats comprising the main paint scheme for the parts.

#### 4.2. General retreatment

4.2.1. The parts shall be dis-assembled and wherever possible all metals other than magnesium alloy removed. The paint shall then be removed under conditions which do not substantially affect the surface sealant. The parts shall then be cleaned and treated by one or more of the following methods. 4.2.2. *Complete retreatment (inserts removed).* Parts shall be cleaned free from deteriorated surface sealing resin and corrosion product by blasting with non-metallic grit, machining or scurfing. Uncorroded close tolerance areas may be stopped-off during these processes. The parts shall then be degreased, fluoride anodised. chromate treated, surface sealed generally or locally and painted overall as in Clauses 2.1.3., 2.1.4. and 2.2.2. to 2.2.4.

### 4.2.3. Inserts present.

- (i) Mechanical cleaning. In this method, parts shall be cleaned and re-protected as in Clause 4.2.2. but omitting fluoride anodising.
- (ii) Chemical cleaning. In this method, corrosion product but not sealing resin is removed by immersing the parts in one or more of the following chromate solutions:
  - (a) cold 5 to 10 per cent ammonium chromate through which a stream of air is blown, for a period of hours or overnight.
  - (b) boiling 5 to 10 per cent ammonium chromate for about half an hour;
  - (c) boiling 10 per cent sodium, potassium or ammonium dichromate for about a quarter of an hour.
  - (d) parts from which most of the corrosion product has already been removed mechanically or by one of the above chemical methods, may be immersed for a maximum of  $1\frac{1}{2}$  minutes in a boiling 10 per cent solution of chromic acid.

After cleaning and washing, all corroded areas shall be clean and bright. The parts shall then be chromate treated, surface sealed and painted as in Clauses 2.2.2. to 2.2.4.

#### **APPENDIX I**

#### Method of cleaning magnesium alloy parts by the fluoride anodising process\*

#### General

This process is intended preferably to supersede abrasive blasting as a method of cleaning magnesium alloy castings, or as a method of substantially restoring the corrosion resistance lost as a result of abrasive blasting. The process can be applied to all magnesium alloys in all forms.

### Pre-cleaning

Loosely held particles of foundry sand should be removed by tapping or brushing. On a part which is being reconditioned, grease or paint should be removed by solvent degreasing and/or immersion in a suitable alkaline metal cleaning solution.

#### **Composition of solution** 3.

The bath shall consist of a solution in water having the following composition:-

Ammonium bifluoride ( $NH_4HF_2$ ) 15 to 25 per cent w/v. (150-250 lb/100 gal). The solution may be con-tained in a vessel lined with hard rubber or suitable plastic material resistant to acid fluoride solution. The inner wall of the vessel must not conduct electricity.

#### **Operation and control**

- (i) The composition of the bath shall be maintained between 15 per cent and 25 per cent ammonium bifluoride by the addition of fresh quantities of this salt. The bath must be discarded if it has become contaminated with foreign metals, acid radicles other than fluoride, or organic matter.
- (ii) The temperature of the bath must not exceed 30°C. The bath should be stirred with a pole of wood, ebonite or magnesium alloy.
- (iii) Parts are normally treated in pairs of approximately equal surface area using alternating current. The parts shall be fixed in good electrical contact and suspended not less than 9 inches below the surface of the solution. All parts of the fixing clamps which extend into the solution shall be made of magnesium alloy other than magnesium-manganese alloy. Alternating current is applied and the voltage raised to 90 to 120 volts. Current flow is usually heavy at first, but diminishes rapidly as surface impurities are removed and an unbroken magnesium fluoride film is formed.

The treatment shall be continued for 10 to 15 minutes or until the current falls to below 5 amp per sq ft of the smaller electrode. The current should then be switched off and the parts removed from the bath, washed and examined.

#### 5. **Requirements for acceptance**

The parts shall have a uniform clean white or pearly grey appearance and be free from foundry sand. Dark areas in hollows may indicate entrapment of gas during treatment. Very thin semi-transparent films indicate treatment at too low a voltage; only on machined surfaces or on wrought surfaces in good condition is such a film acceptable. An etched appearance indicates a bath too low in ammonium fluoride, too hot, or operated at too high a voltage. A thick film may indicate the presence of acid radicles other than fluoride. Surface pitting may be caused by chloride in the bath.

#### Safety precautions 6.

Because of the high voltage used, access to the bath must be prevented while the current is switched on. Spray from the bath is poisonous, and suitable ventilation must be provided.

#### **APPENDIX II**

#### **Chromate treatment**

After having been cleaned and degreased, the parts shall be washed free from cleaning solution and transferred without drying to one of the following chromate treatment baths.

#### Hot half hour chromate bath, Bath (iii) 1.

1.1. General This bath is suitable for the treatment of all types of magnesium-base alloy, but fairly close control is required for the treatment of alloys of the magnesium-manganese and magnesium-zirconium classes. The treatment is recommended where a high degree of protection is required with negligible dimensional change.

1.2. Composition of bath. The freshly prepared bath shall consist of a solution in water having the following composition :-

Ammonium sulphate	3.0 per cent w/v	(30 lb/100 gal)
Ammonium dichromate	1.5 per cent w/v	(15 lb/100 gal)
Potassium dichromate	1.5 per cent w/v	(15 lb/100 gal)
Ammonium solution	0.27 <sup>to</sup> 0.43 per	(43  to  69  fl oz/100  gal)
(sp. gr. 0.880)	cent v/v	
	(see Table I below)	

The potassium dichromate may be replaced by the same weight of sodium dichromate (Na<sub>2</sub>Cr<sub>2</sub>O<sub>7</sub>.

 $2H_2O$ ). The solution may be contained in a mild steel or, preferably, an aluminium vessel. 1.3. *Operation of bath.* The cleaned parts shall be immersed in the boiling solution for 30 minutes. Evaporation losses shall be made up from time to time. After treatment the parts shall be immediately washed in warm water (not above 50°C).

<sup>\*</sup> This process is covered by British Patent 721,445.

1.4. *Control of bath composition*. The pH value of the solution to give a film in accordance with para. 1.5. should be between 4.8 and 6.2. The pH values which will give good treatment in a freshly prepared bath are indicated in Table I.

Alloy	pH range	Percentage of 0.880 ammonia required to bring pH of bath to start of range
Magnesium-manganese alloysD.T.D. 118D.T.D. 142Magnesium-aluminium alloysMagnesium-zirconium alloys, all types	6.05-6.13 6.13-6.18 5.90-6.05 5.90-6.00	0.37 0.43 0.27 0.27

Table I

As the bath ages (due to accumulation of magnesium salts) it will be necessary to lower the pH of the solution to produce a satisfactory film. The pH value should be as high as possible consistent with the production of a film of good appearance, and must be adjusted by the addition of ammonia (to replace loss by evaporation), chromic acid or sulphuric acid as required.

- *NOTE 1.* Addition of ammonia (sp. gr. 0.880) at the rate of 8 fluid oz per 100 gallons (0.05 per cent v/v) of chromate bath raises the pH value of the bath by approximately 0.1. The ammonia solution should be diluted with at least its own volume of water before addition to the hot solution.
- *NOTE 2.* Addition of chromic acid at the rate of 10 oz per 100 gallons (0.063 per cent w/v) or sulphuric acid (sp. gr. 1.84) at the rate of 5 oz (2.7 fluid oz) per 100 gallons (0.17 per cent v/v) reduces the pH value of the bath by approximately 0.1. The sulphuric acid should be diluted with at least twice its own volume of water before addition to the hot solution.

The chloride content of the bath shall not exceed 0.5 per cent calculated as sodium chloride.

Details of methods of analysis of the bath are given in para. 1.7.

- 1.5. Requirements for acceptance of treated parts.
- (i) The chromate finish on machined, sheet or extruded surfaces shall be adherent and free from bloom. The adhesion can be tested conveniently by rubbing with white paper; no more than the faintest stain shall be shown on the paper, and the film shall not show signs of damage.
- (ii) On magnesium-manganese alloys (D.T.D. 118, D.T.D. 142) the film shall be of a uniform straw to light brown colour without blemishes. Chocolate coloured films, which result from treatment in too acid a bath, are brittle and non-adherent. Patchy and uneven films are sometimes caused by surface impurities, and should be reported to the manufacturer of the material.
- (iii) On magnesium-aluminium alloys the colour of the film on machined surfaces will vary from brown-black through jet black to grey-black with increasing content of aluminium in the alloy from 4 per cent upwards. The quality of the chromate treatment on castings should be judged by the appearance on machined surfaces.
- (iv) On magnesium-zirconium alloys with or without additions of zinc, rare earths and thorium, the film on machined or wrought surfaces shall be of a brown to black colour.

1.6. Alternative composition. A modification of Bath (iii), known as the de Havilland 167 bath, may be used. The solution contains 3 per cent ammonium dichromate, 1 per cent ammonium sulphate and 0.10 per cent ammonia (sp. gr. 0880). The bath is worked at a pH somewhat lower than that of Bath (iii). Requirements for acceptance are as in para. 1.5.

1.7. Method for the control of the composition of Bath (iii).

1.7.1. *Measurement of pH value*. The pH value of the bath may be controlled by any convenient method, but care must be taken to ensure that the measuring instrument used gives true pH values or that the readings given by it can be converted into true values, since it is these that are quoted in the text.

1.7.2. *Methods of analysis.* The analysis must be carried out on a clear sample of the bath solution. The sample should, if turbid, be filtered through a paper which should not be pre-wetted.

(i) Estimation of chromate. Acidify 5 ml of the bath solution with sulphuric acid, add excess of N/10 ferrous ammonium sulphate and back titrate the excess with N/10 potassium permanganate solution.

1 ml N/10 ferrous ammonium sulphate = 0.0667 per cent CrO<sub>3</sub>.

(ii) *Estimation of sulphute.* Acidify 5 ml of the bath solution with hydrochloric acid and, after warming, add gradually a few ml of alcohol until all the chromate is reduced as shown by a change of colour to a deep green. Dilute the solution to approximately 100 ml, heat to boiling and add 20 ml of a 10 per cent solution of barium chloride. After standing overnight filter off the barium sulphate, wash, ignite and weigh.

0.1g BaSO = 0.84 per cent H<sub>2</sub>SO<sub>4</sub>.

(iii) *Estimation of chloride*. Reduce 50 ml of the bath solution with sulphur dioxide. Boil off the excess of  $SO_2$  and add 5 ml of nitric acid (sp. gr. 1.42) followed by an excess of approximately N/10 silver nitrate solution. Boil the solution until the precipitate of silver chloride coagulates. Filter off the precipitate on a weighed sintered glass crucible, wash with hot water, dry and re weigh.

$$1.00g \text{ AgCl} = 0.815 \text{ per cent NaCl}.$$

#### Acid chromate bath, Bath (iv) 2.

2.1. General. This bath is particularly suitable for the treatment of magnesium-manganese alloy parts or generally for the treatment of unmachined parts to be kept in store. It is not recommended for the treatment of parts machined to fine tolerances. Parts previously treated in the acid chromate bath may be retreated directly in the same bath.

2.2. Cleaning prior to treatment. To avoid contamination of the bath, the parts should be free from any grease before treatment.

2.3. Composition of bath. The bath shall consist of a solution in water having the following composition :-

Sodium dichromate (Na<sub>2</sub>Cr<sub>2</sub>O<sub>7</sub>. 2H<sub>2</sub>O)

or Potassium dichromate  $(K_2Cr_2O_7)$ Nitric acid (sp. gr. 1.42)

15 per cent w/v (150 lb/100 gall)

20 to 25 per cent v/v (20 to 25 gal/ 100 gal)

Earthenware, slate, aluminium or stainless steel tanks may be used to contain the solution.

2.4 Operation and control of bath.

- (i) The parts shall be immersed in the solution at room temperature for a time varying from 10 seconds for a fresh bath to 2 minutes for a solution in which the nitric acid is nearing exhaustion. The parts shall be allowed to drain for a period of 5 to 15 seconds after removal from the solution. They shall then be washed in water at a temperature of not more than 50°C.
- (ii) Where large structures are being chromate treated, handling difficulties may require that the times of immersion be longer than those given above. In such instances the nitric acid content of the bath may be reduced to lessen the activity of the bath. The treatment time in this modified bath shall be from 15 seconds upwards.
- (iii) The bath is best controlled by means of a hydrometer. The freshly prepared solution has a specific gravity of 1.20. When the specific gravity has risen to 1.22 the solution is spent. A spent solution may be rejuvenated by small additions of nitric acid (6 per cent to 8 per cent at a time). The rejuvenated solution should be discarded when the specific gravity reaches 1.24.

2.5. *Requirements for acceptance of treated parts.* 

The chromate film shall be adherent and free from bloom (see para. 1.5 (i)). On magnesium-alu-minium alloys the film shall be of a light grey colour with a reddish iridescence. On other alloys the film shall be of a clear brassy yellow colour.

#### Chrome-manganese bath, Bath (v) 3

3.1. General. This bath is suitable for the treatment of all types of magnesium alloy where a high degree of protection is required with negligible dimensional change.

3.2. Composition of bath. The bath shall consist of a solution in water having the following composition :

Sodium dichromate (Na<sub>2</sub>Cr<sub>2</sub>O<sub>7</sub>. 2H<sub>2</sub>O) 10 per cent w/v (100 lb/100 gal)

 $\begin{array}{l} \text{Manganese sulphate (MnSO_4. 5H_2O)} & \text{for per cent w/v (100 lb/100 gal)} \\ \text{Magnesium sulphate (MgSO_4. 7H_2O)} & \text{5 per cent w/v (50 lb/100 gal)} \\ \end{array}$ 

The solution may be contained in a steel or aluminium vessel.

3.3. Operation and control of bath. The bath can be operated at any temperature up to boiling point. The following durations at different temperatures are given as a guide:  $-1\frac{1}{2}$  hours at 20°C to 30°C, 30 minutes at 50°C to 60°C, 15 minutes at 70°C to 80°C, 3 to 10 minutes at boiling point. The acidity of the bath varies from about pH4 when freshly prepared to about pH6 when exhausted. An exhausted solution may be rejuvenated by addition of 5 per cent w/v of manganese sulphate. (MnSO<sub>4</sub>.  $5H_2O$ ). Alternatively, sulphuric acid, or a mixture of equal weights of sulphuric acid and chromic acid, may be added until the pH value is reduced to not less than 4.

3.4. *Requirements for acceptance of treated parts.* The chromate film on machined, sheet or extruded surfaces shall be adherent, free from bloom, (see para. 1.5. (i)) and of a dark brown to black colour. Light golden films are not acceptable except on magnesium-manganese alloy sheet to D.T.D. 118.

# **APPENDIX III**

### **Repair of chromate films**

Where it is necessary to repair chromate films which have been damaged locally, one of the methods 1. described below shall be used. Neither method is intended for use over a substantial area; in such instances the components shall be given the full immersion treatment described in Appendix II. Neither method shall be used on assemblies where there is danger of entrapment of treatment chemicals. Areas to be treated shall first be degreased if required.

2. Acid chromate method. In this method the acid chromate bath solution, Bath (iv) of Appendix II, is applied by lightly swabbing with glass wool until a permanent golden colour is developed on the metal surface. The metal surface shall then be washed copiously and dabbed or blown dry. The method is suitable prior to the application of any primer for magnesium alloys.

3. Selenious acid method. In this method a solution containing 10 per cent w/v selenious acid ( $H_2SeO_3$ ) is applied by swabbing with cotton wool or soft rag until a permanent dark brown colour is developed on the metal surface. The metal surface shall then be washed copiously and dabbed or blown dry. This method is not suitable for use prior to the application of an etch primer.

4. Small in situ repairs of assemblies where there is danger of entrapment of treatment chemicals shall be carried out by application of etch primer followed by a full appropriate paint scheme.

*NOTE.* The acid chromate bath solution and the selenious acid solution must not be allowed to come in contact with the skin.

Approved for issue,

E. W. RUSSELL,

Director of Materials Research and Development/Aviation.