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AA 067 36 16: PROCESS FOR MANGANESE PHOSPHATING OF FERROUS SURFACES BY IMMERSION PROCESS

PAGE 1 OF 7; Cl 3.0 COMPLIANCE WITH NATIONAL STANDARDS

Year of IS reference is modified as follows:

IS:3618-66 (Reaffirmed 1997)

Please see Instructions on the reverse.

Ref : Cl; 31.11.19 of MOM of MRC-C	Amd No. 03	Approved MRC- C	Issued CORP. R&D	Date 15.10.2002	Cum.Sr.No. A 3170

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1) **PAGE 5.7; Cl 8.3:**
Ferrous ION given in the title is modified as "**ferrous iron**"

2) **PAGE 6.7; Cl 8.3.3:**
Ferrous ION given in the first sentence is modified as "**ferrous iron**"

Please see Instructions on the reverse.

Ref : Cl. No. 28.16.19 of MOM of 28th MRC(C)	Amd No. 02	Approved MRC (C)	Issued CORP. R&D	Date 15.10.2000	Cum.Sr.No. A 2868

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Cl. 6.1.3 " 9 Mmersion Time " is replaced by "Immersion Time"

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2.1 Cl. 8.2 The existing matter is replaced by the following:

"The concentration of bath solution shall be regularly checked depending upon the use of bath and maintained as detailed below".

2.2 Cl. 8.3 The title is replaced by the following:

"Ferrous ion concentration maximum 0.5%".

2.2 Cl 8.3.2, "The existing matter is replaced by the following" No. of ml of 0.1 N Potassium Permanganate consumed x 0.056 = Percentage of Ferrous Iron.

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CL. 8.3.3 In the I st sentence "when ferrous ION concentration reaches...." is replaced by the following:

" when ferrous ion concentration reaches...."

Please see Instructions on the reverse.

Ref :	Amd. No.	Approved	Issued	Date	Cum.Sl.No.
Cl 27.4.3 of MOM of MRC (C)	01	MRC (C)	CORP. R&D	15-08-99	A 2587

PROCESS FOR MANGANESE PHOSPHATING OF FERROUS SURFACES BY IMMERSION PROCESS

1. GENERAL:

This standard details the process for producing a black, non-metallic crystalline, antifriction, coating of manganese - iron phosphate on steel and iron surfaces and its subsequent treatment in oil and varnish.

2. APPLICATION:

This corrosion resistant coating reduces wear on moving parts such as piston, piston rings, gears, liners, bolts, nuts, tools, camshafts, compressor shafts, lubrication boxes, cylinders and all types of machine parts where ever wear is a constant factor to be considered.

3. COMPLIANCE WITH NATIONAL STANDARDS:

This standard has reference to the following national standards in respect of surface condition and quality of deposits.

IS: 3618-1966 (Reaffrimed 1991) : Phosophate treatment of iron and steel for protection against corrosin.

4. MATERIALS:

Material	CPS.No.	IS No.	Avialable From
4.1 Insulating Oil (Low viscosity)	AA 27101	(IS : 335)	
4.2 Chromic Acid	AA 54104	(IS : 330)	
4.3 Rusto-proof pc-19	AA 55608		M/s.Peddington Chemical Industry, Bombay.
4.4 Trichloroethylene (Technical)	AA 56706	(IS : 245)	

4.5 Sufix MN - 641 : M/s.Grauwer & Weil (I) LTD., Bombay.

4.6 Kemfix Mn 741 : M/s.Artek Surfin Chemicals (P) Ltd. Bombay.

4.7 Black Stain Shellac Varnish : M/s.C.I.T. BHEL, Bhopal and M/s.Shalimar Paints, Bombay.

5. EQUIPMENT:

5.1 Phosphating Tank:

Mild steel tank preferably lined with hard rubber or propylene and fitted with a water supply, an over flow and a drain system. Thermostatically controlled heating arrangement shall be provided in the tank.

5.2 Chromic Acid Tank:

Mild steel tank preferably lined with hard rubber and fitted with a water supply, an over flow and a drain. Thermostatically controlled heating arrangement must be provided in the tank.

5.3 Rinsing Tank:

Mild steel tank provided with a water supply, an overflow and a drain.

5.4 Air Blowing:

A high pressure of cool air supply may be provided for initial drying.

5.5 Staining Tank:

Mild steel tank fitted with a mild steel lid and a drain cock.

5.6 Oil Tank:

Mild steel tank fitted with a mild steel lid and a drain cock.

5.7 Jigs And Racks:

Jigs, brackets and suspension hooks must be made of mild steel, stainless steel or bakelite.

6. PREPARATION OF BATH SOLUTIONS AND OPERATING CONDITIONS:

6.1 Phosphating Solution:

6.1.1 Rusto-Proof PC 19 : 5% (V/V)

Operating Temperature : 95 to 99 °C.
Immersion Time : 10 to 30 minutes.

6.1.2 Surfix MN-641 : 7% (V/V)

Operating Temperature : 95 to 99 °C.
Immersion Time : 10 to 30 minutes.

6.1.3 Kemfix MN - 741 : 7% (V/V)
Operating Temperature : 95 to 99 °C
9 Mmersion Time : 10 to 30 minutes

6.1.4 The phosphating tank must be thoroughly cleaned before making up the solution.

6.1.5 The clean phosphating tank shall be half filled with clean water and then add the necessary quantity of RUSTO-PROOF PC-19 or SURFIX MN-641 or Kemfix MN 741 to the bath according to the clause 6.1.1, or 6.1.2, or 6.1.3. Bring the solution to working level by adding more water and mix well by stirring and heat to 65 to 70°C.

6.1.5 The bath shall be aged by introducing preferably 50 to 100 gm of cleaned steel wool or scrap iron pieces per 100 litres of bath solution for 30 to 50 minutes. The steel wool shall then be removed and the bath is heated to operating temperature.

6.2 Chromic Acid Solution:

6.2.1 Chromic acid : 0.05% (W/V)
Operating temperature : 85 to 90°C
Immersion time : 0.5 to 1 minute.

6.3 Black Stain Shellac Varnish:

The varnish shall be supplied ready for use at room temperature.

6.4 Insulating Oil:

The oil shall be supplied ready for use at room temperature.

7. **PROCESS:**

7.1 **Cleaning:**

The articles that are to be phosphated shall be free from oil, grease, rust, scale etc. For manganese phosphating rust and scales shall be preferably removed by shot blasting. In case shot blasting is not possible, cleaning shall be done as per Corporate Standard AA 067 36 01.

7.1.1 All articles shall be placed in a basket or jig or otherwise suitably suspended and the dipped in trichloroethylene for few seconds before immersing it in the phosphating bath.

7.2 **Phosphating:**

All articles shall then be phosphated in the specified operating conditions as mentioned in clause 6.1 and rinsed in clean running water for 15 to 30 seconds.

7.3 **Passivation:**

After rinsing articles shall be dipped in the Chromic Acid passivation solution for 0.5 to 1 minute.

7.4 **Drying:**

The articles shall then be dried at high pressure of cool air supply.

7.5 **Staining:**

Where necessary, after cooling but within two hours of air drying as above, the articles, shall be immersed in black stain shellac varnish for 3 to 5 seconds, removed and allowed to drain and dry in air for atleast 30 minutes.

7.6 **Oiling:**

After staining, the articles shall be immersed in low viscosity insulating oil for 3 to 5 seconds at room temperature. It shall then be removed from oil and allowed to drain.

8. **TESTING & MAINTENANCE :**

8.1 **Testing of Phosphating Solution:**

The solution shall be tested at suitable intervals by the following procedure:

8.1.1 Clean water shall first be added to the solution, if necessary, to restore the latter to the correct working level, followed by stirring to ensure complete mixing.

8.1.2 10 ml. of the working solution shall then be transferred to the 250 ml conical flask. Two or three drops of Alcoholic 1% Phenol-Phthalein indicator must then be added, with shaking.

8.1.3 Sodium Hydroxide 0.10 N shall then be added in with occational shaking or stirring until a permanent colour change (to pink) is just obtained.

8.1.4 The volume of 0.10 N NaOH in ml required shall be noted. The volume in ml is pointage.

8.2 Maintenance Of Solution Strength/Pointage:

The concentration of bath solution shall be regularly checked depending upon the use of bath and maintaince as detailed below.

8.2.1 If the solution is at the correct working strength/pointage the volume obtained in clause 8.1.4 will lie between 30 and 35 ml. for RUSTO-PROOF PC-19 solution and between 40 & 50 for SURFIX MN- 641,and Kemfix MN-741 solution. If the above tests show any deviation from this range, the strength of the solution shall be adjusted as follows:-

8.2.1.1 If the volume is greater than 35 ml. for RUSTO-PROOF PC-19 or greater than 50 ml for SURFIX MN-641 or Kemfix MN-741 sufficient quantity of the solution shall be removed from the tank and replaced by clean water to reduce the volume within the working range.

8.2.1.2 For Rusto-proof PC-19:

If the volume is less than 30 ml., then for each ml.(pointage) below 30, add 2 litres of PC-19 solution per 1000 litres of bath solution.

For Surfix Mn-641 OR KemFix MN-741:

If the volume is less than 40 ml., then for each ml.(pointage) below 40, add 1.75 litre of Surfix -Mn-641 solution per 1000 litres of bath solution.

8.3 Ferrous ION Concentration Maximum 0.5%:

The following procedure shall be followed for testing.

8.3.1 10 ml. of bath solution shal be taken into a 250ml. conical flask, add 1-2 ml. of 50 % H_2SO_4 solution to it. Titrate against 0.1 N Potassium Permanganate till colour changes from colourless to pink, persisting for a least 15 seconds.

8.3.2 No.of ml of 0.1 N Potassium Permanganate consumed X 0.056 = Percentage of Ferrous ION.

8.3.3 When ferrous ION concentration reaches above 0.5% (i.e. consumption of 0.1 N Potassium permanganate is 9 ml.) the phosphate bath should be partially replaced with fresh solution or if required completely discarded.

8.4 Sludge Removal:
The sludge formed during processing shall be removed from the tank and the heating coils after every month. After such cleaning, the solution strength shall be tested as in 8.1 and necessary adjustments made.

8.5 Chromic Acid Solution:
This solution shall be replaced each week.

8.6 Black Stain Shellac Varnish:
The viscosity of varnish at 27° C ± 2 in cup-4 to IS:3944 shall be 30 ± 5 seconds and to be tested each week.

8.7 Insulating Oil (Low Viscosity):
The working level of the oil shall be maintained by periodic addition of new oil.

8.8 Speed of Phosphate Coating:

5 to 7 microns of phosphate coating will be deposited in 30 minutes.

9. INSPECTION AND QUALITY OF DEPOSIT:

When tested in accordance with the test methods shown against each, the deposit shall conform to the norms specified below:

9.1 Sampling:

A minimum of 1% of each batch/load or part thereof shall be taken at random for testings. When the components are big and can not be subjected to any of the specified test, a test panel of suitable size of the same basis metal shall be phosphated along with component under identical condition for the purpose of test (approximate size of test panel 5 cm X 10 cm).

9.2 Freedom from Defects: (IS : 3618)

Phosphated surface shall be of mouse black / dark grey crystalline appearance. They shall be free from untreated patches and from flaky and uneven deposits, some time caused by excessive sludge in the bath. They shall be free from scratches, pits and residues of the processing solution as it may initiate deterioration of the organic coating or premature corrosion.

9.3 Weight of coating: (IS : 3618)

7.5 gm/m^2 minimum.

10. Referred Standards: (Latest Publication Including Amendments)

1) AA 27101 2) AA 54104 3) AA 56706 4) AA 55608
5) AA 0673601 6) IS : 330 7) IS : 335 8) IS : 245
9) IS : 3618