Pre-Treatment Solutions
The Print Works 35a Dane Road
Coventry CV2 4JR
United Kingdom.
Tel +44(0)2475 090 133 Fax +44(0)2475 090 139
sales@pretreatmentsolutions.com
www.pretreatmentsolutions.com
PHOS-PREP®
Experts in non Chrome Preparations

PHOS-PREP® PP 940 A YELLOW CHROME

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1. GENERAL

In order to enhance the performance of powder paint coatings on aluminium or zinc and give greatly improved corrosion resistance, it is necessary to passivate the surface of the metal using a conversion coating. Our PHOS-PREP® PP 940A system provides a yellow chromate conversion coating designed to meet the requirements of current standards.

The BS EN 12206-1:2004 for architectural powder organic coatings on aluminium specifies for conventional pre-treatment the application of a chromate or a chromate/ phosphate conversion coating. In compliance with the standard we set the operating conditions to produce a conversion coating weight of 0.4 to 0.8g/m^2.

2. SPECIFICATION

PHOS-PREP® PP 940A is a two pack system with PHOS-PREP® PP 940A providing the chromate ions and the free acidity of the bath and the PHOS-PREP® PP YMR providing the upkeep and added activators needed as the bath solution ages.

However, a new bath would typically be made up at 3-5% vol/vol PHOS-PREP® PP940A – fill tank with water adjust the pH with 60% Nitric to lower the pH to 4.5 / 5 pH (to condition the water then add between 3/5% by volume the PHOS-PREP® PP 940A) mix working solution and take pH reading adjust if required to 1.8 / 2.3 with 60% Nitric Acid

3. BATH MAKE-UP

The make up of a new PHOS-PREP® PP 940 bath will be dependent on the process time required and take into account the effluent system. Typically a new bath would be made up at 3-5% vol/vol PHOS-PREP® PP 940A for a chrome pointage of between 5.5mls (3%) to 9mls (5%). The operating pH of a PHOS-PREP® PP 940A bath will be 1.8pH to 2.3pH, should the operating bath pH rise to 2.6pH titrate the bath and add the correct amount of PHOS-PREP® PP 940A to the bath – in addition if the pH is still above 2.3pH adjust the pH down with 60% Nitric acid – (750mls of 60% Nitric acid per 100 ltrs working volume will reduce the pH by 0.2/0.4) allow 30 minutes after adjustment before re testing.

4. PROCESSING CONDITIONS

For immersion treatment the process times on a plant will vary depending on the type of cranes in operation.

Whereas immersion times of between 1 and 3 minutes are quite common, the concentration of the bath and the operating temperature can be varied to give longer or shorter process times.

Our recommended operating temperature range is 18°C to 24°C with the processing times varied accordingly.

When operating times and temperatures have been set for the plant, the solution concentrations are controlled to produce the characteristic yellow \ golden brown coating indicative of an optimum coating weight. The control parameters are normally between 5.5 and 9 points for the chrome pointage.

5. LABORATORY CONTROL

Equipment	**Reagents**
5ml pipette.	N/10 Sodium Thiosulphate
250ml conical flask.	10% Sulphuric Acid.
25/50ml burette	Stabilised Starch & Potassium Iodide crystal
100ml Volumetric Flask	

Methods.

PHOS-PREP® PP 940A Pointage.

Pipette a 5ml sample of the bath into a titration flask and add 50 ml of de-ionised water.

Add to the flask 5 ml of 10% Sulphuric acid and 2 grms Potassium Iodide crystal The solution turns a deep violet / black colour. Add 5mls or 2 grms Stabilised Starch Indicator

Titrate the solution with N/10 Sodium Thiosulphate, to a clear end point.

Titre = Bath Pointage.

% of the working bath = Pointage x 0.56%

Should the % / pointage not within specification make additions with either PP 940A or PP YMR depending the pH of working bath.

Method for Chromate Coating Weight Determination

A) STRIPPING SOLUTION

The solution to be used for stripping the test panels is made by diluting 70% w/w Nitric Acid with an equal volume of water.

B) PROCEDURE

Measure the m^2 surface area of the test panel and process the panel through the line under the standard processing conditions. Take the panel off the line before the drying oven and allow it to air-dry. The coating weight measurement should be carried out within three hours of pre-treatment.

Weigh the dry chromated panel on a 4 decimal place analytical balance to the nearest 0.1mg. Then immerse the test piece in the stripping solution for 1 minute at room temperature. Rinse the test piece immediately in clean running water and then in distilled water, dry rapidly and re-weigh.

C) CALCULATION

The difference between the weight of the test panel before and after stripping expressed in grams, divided by the surface are expressed in m^2 give the coating weight as g/m^2.

If the tests have been carried out in duplicate or triplicate then the mean result should be recorded.

D) SPECIFICATION

Chromate coating weight shall be between 0.4 g/m^2 & 0.8 g/m^2

5. EFFLUENT CONTROL

Hexavalent chromium is subject to quite rigorous control by the water authorities. The limits for its control in the effluent water varies can range from zero to 2 p.p.m, but fortunately quite simple procedures can be introduced to adhere to such limits.

The PHOS-PREP® PP940A bath itself is a NEVER-DUMP bath and therefore it is only the carry over from this bath to a running rinse that requires effluent treatment. For this reason the treatment of the chromate rinses is separated from the rest of the effluent treatment.

Initially the rinse waters would be diverted to a holding tank in order that it can be treated in batches: the first step is to dose with a reducing agent such as sodium metabisulphite in a reaction vessel. The sodium metabisulphite will reduce the chrome $^{6+}$ to chrome $^{3+}$, and its addition rate would be controlled by a REDOX probe.

For efficient reduction of the chromium by the sodium metabisulphite, the solution should be maintained at a pH value of no more than 2.5 (preferably pH 2 by use of pH controllers), and it is recommended that the solution is acidified with sulphuric acid before entry to the mixing tank. Continuous acid dosing of the solution in the mixing tank is also required because the bisulphite reduction of chrome^{6+} consumes acid by the following overall reaction:

$$\mathbf{Cr_2O_7{}^{2-} + 3HSO_3{}^{2-} + 8H^{1+} = 2Cr^{3+} + 3HSO_4{}^{2-} + 4H_2O}$$

In a separate tank the rinse waters now containing chromium in the trivalent state are neutralised to precipitate chromium hydroxide from solution. Neutralisation using either caustic soda or slaked lime to a pH greater that 6.8 reduces the solubility of chromium to less than 0.5 mg/l if the solution is held above pH 6.8 for at least 2 hours.

This is the basis for recommending that the water treatment is done in batches. After the initial precipitation of chromium hydroxide the pH of the solution will decrease but dosing with either caustic soda or slaked lime, to hold the pH of the solution above pH 6.8 for this length of time, maximises the chrome precipitation.

The use of calcium hydroxide slurry over the cheaper and more manageable liquid caustic soda for neutralisation has proved to be advantageous in two respects. The volume of the sediment formed using slaked lime is smaller than that using caustic soda. The maximum precipitation of the chromium hydroxide using caustic soda for neutralisation occurs between pH 7 and 8 whereas slaked lime neutralisation because of the presence of calcium ions gives a maximum precipitation range of between pH 7 and 10.

The presence of the calcium ions from the slaked lime is also beneficial in reducing the fluoride concentration of the effluent water. Above pH 7.0 the fluoride ions are precipitated as the sparingly soluble calcium fluoride. At pH 8.0 we would expect to find that the fluoride concentration in solution has been lowered to less than 60 mg/l and at pH 11.5 to be less than 8 mg/l although then we would be outside the pH consent limits.

Best practice we think would be the use of caustic soda for neutralisation, supplemented with a continuous feed of calcium hydroxide slurry at a rate based on reducing the fluoride concentration to an acceptable level. The control range for the neutralisation should be as near to pH 9 as possible.

The waters containing the precipitates are then best dealt with by passing them into settlement tanks for the settled solids to be separated by the use of a filter press for specialist collection and disposal of the sludge.

At pH 9 the clarified waters can have a total Chromium concentration of less than 0.5 mg/l and a fluoride concentration of less than 15 mg/l and suitable for discharge or selective re-cycling.