

CONTROL TESTS FOR PHOSPHATE COATING PROCESS

Application

These test methods are used where applicable to check the concentration of the solutions used in the phosphate coating process described in the Material Standards PL 53-07, PL 53-09, and PL 53-32.

The analytical techniques listed which utilize a "point system" for the titration of solutions is designed to provide for simple conversion of results in points to concentration of a solution. This procedure eliminates the need for relatively complicated manipulations of gram equivalent weights, specific gravity, etc. If it is necessary to express concentrations in actual percentage concentrations this may be accomplished by substituting into the following conversion formula.

$$\% \text{ unknown} = \frac{\text{mL titer used} \times \text{normality} \times \text{factor} \times 100}{\text{wt. sample}}$$

Solutions Required

See Ford Laboratory Test Method AZ 2-1 for preparation of solutions and indicators.

Standard Sodium Hydroxide – 0.1 N	Reagent No. 1
Standard Hydrochloric Acid – 0.1 N	Reagent No. 4
Standard Potassium Permanganate – 0.042 N	Reagent No. 9
Use 1.34 g and dilute to 1 liter or take 420 mL of 0.1 N solution and dilute to 1 liter.	

$$\text{titration factor} = \frac{\text{normality}}{0.042}$$

Standard Potassium Ferrocyanide	Reagent No. 12
Standard Ferrous Sulfate Solution – 0.1 N	Reagent No. 10
Standard Sodium Thiosulfate Solution – 0.1 N	Reagent No. 14
Standard Potassium Dichromate Solution – 0.1 N	Reagent No. 15

Indicators

Phenolphthalein	Reagent No. 101
Brom Cresol Green	Reagent No. 108
Brom Phenol Blue	Reagent No. 109
Diphenylamine	Reagent No. 110
Iron Test Paper	Reagent No. 111
Starch Solution	Reagent No. 113
Orthophenanthroline	Reagent No. 114

Date	Action	Revisions
2001 01 31	Revised	Editorial – no technical change A. Cockman
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Titration and Indicator Solutions – Supply List

Prepared solutions required for control tests may be obtained from the Amchem Products, Inc. or Parker Rust Proof Co. when ordered by their respective numbers as shown:

Standard Solutions	Amchem	Parker
Sodium Hydroxide – 0.1 N	11	11
Hydrochloric Acid – 0.1 N	20	--
Potassium Permanganate – 0.042 N	15	15
Sulphuric Acid – 0.1 N	--	20
Potassium Ferrocyanide (1 g = 0.015 g/Zn)	16	--
Sodium Thiosulfate – 0.1 N	31	--
Ferrous Sulfate – 0.1 N	--	31
Potassium Dichromate – 0.1 N	--	30
Indicators	Amchem	Parker
Phenolphthalein	3	3
Brom Cresol Green	2	2
Brom Phenol Blue	10	11
Diphenylamine	7	7
Indicator Paper – Iron	2005	17
Orthophenanthroline	--	12

Conditioning and Test Conditions

All test values indicated herein are based on material conditioned in a controlled atmosphere of 23 +/- 2 °C and 50 +/- % relative humidity for not less than 24 h prior to testing and tested under the same conditions unless otherwise specified.

Procedure

Test A – Cleaner Control

Free Alkali

1. Pipet 10 mL of sample into a 150 mL Erlenmeyer flask.
2. Add 5 drops of phenolphthalein indicator.
3. Titrate with 0.1 N HCl until solution changes from pink to colorless.
4. mL of HCl used = free alkali value.

Total Alkali

1. Pipet 10 mL of sample into a 150 mL Erlenmeyer flask.
2. Add 10 drops of brom cresol green indicator.
3. Titrate with 0.1 N HCl until color changes from purple red to yellow.
4. mL of 0.1 N HCl indicates total alkali value.

Test B – Rinse Water Control (After Alkaline Cleaning)

Total Alkali

1. Pipet 10 mL of the rinse water into a 150 mL Erlenmeyer flask.
2. Add 5 drops of brom cresol green indicator.
3. Titrate with 0.1 N HCl to yellow end point. Report mL of HCl as total alkali value
4. If solution turns yellow after addition of indicator, discard sample and proceed as follows: (See Note)
 - (a) Pipet 10 mL of sample into a 150 mL Erlenmeyer flask.
 - (b) Add 5 drops of phenolphthalein indicator.
 - (c) Titrate with 0.1 N NaOH to a pink color.
 - (d) mL of 0.1 N NaOH = points total acid

Note: This is an abnormal condition and indicates that phosphate solution is getting into the rinse water, possibly through a misdirected nozzle.

Test C – Phosphate Coating Control – M1178C or M3B1 with M13844 Accelerator

Total Acid

1. Pipet 10 mL of sample into a 150 mL Erlenmeyer flask.
2. Add 5 drops of phenolphthalein indicator.
3. Titrate with 0.1 N NaOH to the development of a pink color.
4. mL of 0.1 N NaOH = points total acid.