

DETERMINATION OF PERCENT ZINC IN WELDING PRIMERS**Application**

Part 1 of this procedure is used to separate the pigment from vehicle, binders and solvents in paints. Part 2 describes the procedure for the determination of percent zinc in welding primers. An information page is attached to this procedure which lists common pigments and extraction mixtures which may be used for separation of these pigments.

Equipment Required**Centrifuge*****Centrifuge Tubes**

Pear shaped - 100 mL capacity

Laboratory Oven

Recirculating - 250 F capacity

Sieve No. 80**Analytical Balance**

Sensitivity .001 g

*Some pigments are so finely dispersed that a super centrifuge of extremely high velocity is required. These pigments cannot be separated by this method.

Chemicals Required**Extraction Mixture "A"**

10 volumes ethyl ether
6 volumes benzol
4 volumes methyl alcohol
1 volume acetone

Extraction Mixture "B"

5 volumes benzol
4 volumes methyl alcohol
1 volume acetone

Extraction Mixture "C"

1 volume toluene
1 volume acetone

Date	Action	Revisions
2000 08 07		Editorial – no technical change A. Cockman
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Reagents Required

Standard Potassium Permanganate (0.1 N KMnO_4)

(Standardize per Ford Laboratory Test Method AZ 2-1, Reagent 9)

Sodium Acetate Solution (200 g/liter)

Dissolve 200 g of $\text{NaC}_2\text{H}_3\text{O}_2$ or 332 g of $\text{NaC}_2\text{H}_3\text{O}_2 \cdot 3\text{H}_2\text{O}$ and dilute to 1 liter.

Ferric Chloride Solution

Prepare a solution containing 20 g of $\text{FeCl}_3 \cdot 6\text{H}_2\text{O}$ and 20 mL of the $\text{NaC}_2\text{H}_3\text{O}_2$ solution per 100 mL (one day supply only).

Zimmerman Reinhardt Solution

Prepare a solution containing 67 g of $\text{MnSO}_4 \cdot 4\text{H}_2\text{O}$, 130 mL of H_2SO_4 (sp. gr. 1.84) and 138 mL of H_3PO_4 (85 %) per liter.

Conditioning and Test Conditions

All test values indicated herein are based on material conditioned in a controlled atmosphere of 23 $\pm$ 2 $^\circ\text{C}$ and 50 $\pm$ 5 % relative humidity for not less than 24 h prior to testing and tested under the same conditions unless otherwise specified.

Procedure - Part 1 - Pigment Separation

1. Place approximately 15 g of the well agitated sample into a centrifuge tube and add 20 to 30 mL of a suitable extraction mixture.

Note: It may be necessary to try several of the listed extraction materials, depending on the type of resins in the paint formulation.
2. Stir well with a glass rod and wash the rod with a quantity of the extraction mixture to result in 60 mL of solution in the tube.
3. Place the tube in the centrifuge, counterbalance and spin for 15 minutes at moderate speed or until the sample is well settled.
4. Decant the clear supernatant liquid and repeat the extraction twice with 40 mL of the extraction mixture and once with 40 mL of ether.
5. After drawing off the ether, hold the tube in a nearly horizontal position, tap the side of the tube to dislodge the pigment cake from the bottom of the glass tube and set the tube in a beaker of water at 23 $\pm$ 2 $^\circ\text{C}$.
6. Gradually raise the temperature by placing the beaker on top of a warm oven for 10 minutes. Pour off water; then place the beaker in the oven at 105 – 110 $^\circ\text{C}$ for 2 h.