STANDARD METHOD OF TEST

No. X 135-57

MATERIAL Pigment Mixtures

DETERMINATIONS Identification of M-50 Pigment by means of the Microscope

Purpose

This method is to identify M-50 in mixed pigment paint systems, particularly to differentiate M-50 particles from those of red lead, lead chromate and iron oxide.

Apparatus and Acessories

Microscope, polarizing equipped to give 600-700X. A binocular stand is preferred. Objectives: 40X and 60X dry. Eyepieces: 15X. Eyepiece micrometer disc, American Optical Company #1409. 10 mm square ruled into 0.5 mm squares. Substage condenser with diaphragm, 1.25 - 1.40 N.A.

Microscope lamp. Bausch & Lomb Research Microscope Illuminator 31-33-35-11, 6 Volts 108 Watts ribbon filament prefocus base lamp; transformer for 115 Volts 60 cycle A.C.

Microscope slides 50 x 75 mm Fisher Catalogue 12-550.

Micro cover glasses: 19 x 19 mm #1 Fisher Catalogue 12-524.

Spatula and lens paper.

Mounting Mediae: a) Acrylic Resin Solution - Acryloid F-10 manufactured by The Resinous Products and Chemical Company, Philadelphia, Pa. Mix 35 ml of Acryloid F-10 solution with 65 ml of Xylol and keep in a well stoppered dropping bottle.

b) Xylol.

c) Shillaber's Immersion Oil, low viscosity.

0.3 Normal Hydrochloric Acid saturated with hydrogen sulfide.

1:1 Hydrochloric Acid with distilled water.

Concentrated solution of ammonium acetate in distilled water.

Procedure

Staining Technique

Place a small amount of pigment on a clean microscope slide, and add to it a drop of 35% acrylic resin solution. Using a finger, incorporate the pigment into the resin solution and spread it out on the slide into a thin uniform film. This action must be fast and the finger withdrawn from the slide before the film sets, to avoid objectionable streaking. Allow the xylol to be completely evaporated from the film. This can be achieved by blowing a blast of hot air from a hair drier over the preparation or holding the slide over a hot plate. When the film is dry, place a drop of 0.3 normal hydrochloric acid saturated with hydrogen sulfide on the slide and cover with a cover glass. Next to it, place a drop of Shillaber's oil and cover with a cover glass. The oil is used as a blank. Place the slide on the microscope stage and first observe the hydrogen sulfide preparation.

Paints, in most cases, may be mounted in a similar fashion by placing a small drop of the well mixed paint and following the exact procedure described above.

Appearance of M-50 Particles Stained with Hydrogen Sulfide

The particles of the M-50 pigment are easily recognized as they rapidly assume a brownish black color. The original orange color imparts a warm tone of buff or beige in the first stages of the reaction with hydrogen sulfide.

In a mixture, following this procedure, only the particles of M-50 pigment would stain. Free silica particles would stand out

as colorless entities. The red lead does not stain. The iron oxide does not appear to change color. Basic lead chromate will change to solid black. However, its particle character and polarizing qualities would distinguish it from M-50 particles.

Removal of M-50 Coating

A quantity of M-50 pigment is treated in a beaker with 1:1 hydrochloric acid. It is best to bring the mixture to a boil and continue boiling for about 15 minutes. The preparation is allowed to cool, settle and is decanted. After five decantations, most of the hydrochloric acid and chlorides are removed. The sediment is treated with concentrated ammonium acetate and is boiled for about 5 - 10 minutes. The preparation may be filtered or washed by decantation with hot distilled water. The residue is silica from which the active coating was removed. See attached photographs. To verify this, mount a small portion of the dry residue in acrylic resin and stain with 0.3 normal hydrochloric acid saturated with hydrogen sulfide, as described above under "Staining". Under these conditions, the particles of free silica will remain perfectly clear and colorless. They do not stain.

Distinguishing of M-50 Particles in Mixtures by Means of the Microscope With Bright Field Illumination

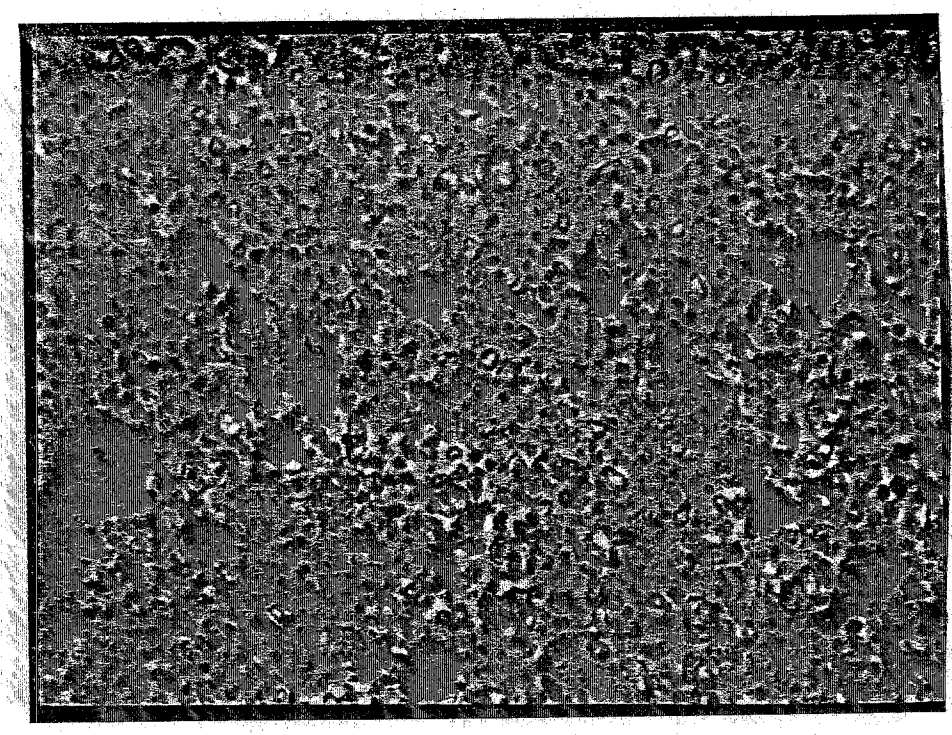
Dry powders or mixed paint systems in which M-50 pigment is one of the ingredients, can be best studied under the microscope when mounted in acrylic resin. The M-50 particles have the shape of ground silica. The average particle size of a typical M-50 pigment is about 8.0 microns. The particles are translucent, have

a yellowish to orange color and are angular in character. The particles of red lead, iron oxide and basic lead chromate vary according to the type material used, however, none of them would have the shape of M-50 pigment.

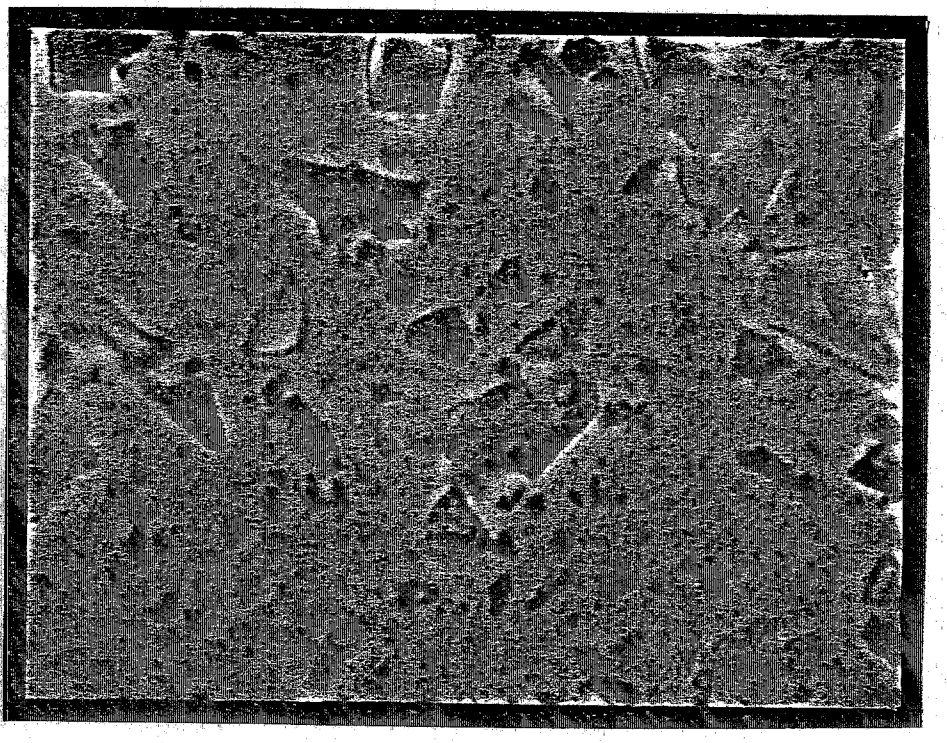
Distinction of M-50 Particles by Means of the Polarizing Microscope

The same slide which was used to study a mixture under the bright field microscope, can be used for the polarizing microscope. The particles are brought into sharp focus, and the nicol prisms crossed. The stage is rotated and any changes that might take place in the character of the particles noted. Under these conditions, the particles of M-50 pigment remain dark; the particles of red lead would extinct in one position and assume an intense green color at positions 90° from that of extinction; the basic lead chromate would alternately assume an orange-red color; and, the iron oxide emits alternately a yellowish color while the uncoated silica particles will assume alternately a pale white color.

PHOTOMICROGRAPHS IN COLOR AT 1200X



Basic Lead Silico Chromate M-50 Pigment



M-50 Pigment After Treatment With Hydrochloric Acid and Ammonium Acetate. Silica Particles are Stripped of Active Material.

Chromium trioxide, CrO_3, %	5.10	5.60	X 131
Coarse particles (+325 mesh), %	--	0.10	X 112
Ignition loss, %	--	0.20	X 117
Lead monoxide, PbO, %	46.2	48.7	X 175
Oil absorption, g per 100 g	10	18	X 111
Silica, SiO_2, %	45.5	48.5	X 127

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