

**DEPARTMENT OF TRANSPORTATION**  
**DIVISION OF ENGINEERING SERVICES**  
Transportation Laboratory  
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## METHOD OF TEST FOR IDENTIFICATION OF PIGMENTS AND EXTENDERS IN PAINTS AND COATINGS

**CAUTION:** Prior to handling test materials, performing equipment setups, and/or conducting this method, testers are required to read “**SAFETY AND HEALTH**” in Section E of this method. It is the responsibility of the user of this method to consult and use Departmental safety and health practices and determine the applicability of regulatory limitations before any testing is performed.

### A. SCOPE

This method describes the X-ray diffraction procedure used for identification of paint pigments. Perform testing in accordance with ASTM Designation: D 5380 except as indicated in this test method.

### B. APPARATUS

1. X-ray diffractometer as described in ASTM Designation: D 5380.
2. Leneta Opacity charts have been found to be satisfactory for use as substrates for the paint films. They are available from the Leneta Company.
3. Film applicator blade, 15-mil gap.

### C. SPECIMEN PREPARATION

1. Liquid Samples:
  - a. Draw down a uniform film of the sample on the glazed side of the Leneta chart and allow to dry thoroughly. The dry film thickness should be adequate to cover the substrate so that x-rays do not penetrate and record the clays in the Leneta chart.
  - b. After the coating has dried, a suitably sized specimen may be cut from the chart to fit into the X-ray diffractometer.
2. Dry Pigments and/or Extender Pigments:
  - a. Extract pigments as specified in California Test 402. Pass powdered pigments and extender pigments through a standard No. 50 sieve.
  - b. Extracted pigments or powders may need to be ground or pulverized to reduce particle size. The quality of the resultant spectra is dependant on the particle size of the sample.
  - b. Prepare a “backpack” sample with the appropriate holder supplied by the manufacturer of the X-ray equipment. Place the holder on a ground glass plate. Heap the powder

into the holder and using a glass microscope slide pack the powder so that it stays in place when lifted up and placed in the instrument.

#### **D. PROCEDURE**

1. Test for and identify compounds in accordance with ASTM Designation: D 5380.
2. When a pigment or extender appears to be absent or not in the specified quantity by X-ray diffraction, it is necessary to confirm the absence by X-ray emission, wet chemical, or petrographic analysis. Petrographic analysis may be used for confirming the presence of diatoms from the extender pigment, diatomaceous silica - when there is not sufficient crystallinity to give an X-ray diffraction pattern.

#### **E. SAFETY AND HEALTH**

This method may involve hazardous materials, operations, and equipment. This method does not purport to address all the safety problems associated with its use. It is the responsibility of whoever uses this method to consult and establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.

X-ray producing equipment can be dangerous to both the operator and persons in the immediate vicinity unless safety precautions are strictly observed. Refer to the manufacturer's instruction manual. Exposure to excessive quantities of X-radiation may be injurious to health. Therefore, users should avoid exposing any parts of their bodies, not only to the direct beam, but also to secondary or scattered radiation that occurs when an X-ray beam strikes or has passed through any material. It is strongly recommended that users check the degree of exposure by film carried on them or by the use of dosimeters and that blood counts be made periodically. Before utilizing the equipment, all persons designated or authorized to operate X-ray instrumentation or supervise its operation, should have a full understanding of its nature and should also become familiar with established safe exposure factors by a careful study of the National Institute of Standards and Technology Handbook "X-ray Recommendations of the International Roentgen Ray Committee on X-ray Protection," and other standard publications on the subject. Inquiries should be made of state agencies as to existing requirements.

Place colorful signs displaying the international radiation symbol near the X-ray equipment.

Use a portable counter periodically to test for leakage of X-rays from equipment. Lead or lead glass shielding is sometimes needed. X-rays of shorter wavelength require more caution.

Prior to handling, testing or disposing of any waste materials, testers are required to read the Caltrans Laboratory Safety Manual. Users of this method do so at their own risk.

**REFERENCES:**  
**ASTM Designation: D 5380**  
**California Test 402**  
**Caltrans Laboratory Safety Manual**

**End of Text**  
**(California Test 421 contains 2 pages)**