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Standard Test Method for Identification of Crystalline Pigments and Extenders in Paint by X-Ray Diffraction Analysis[1]

This standard is issued under the fixed designation D5380; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ε) indicates an editorial change since the last revision or reapproval.

1. Scope

1.1 This test method covers the identification of crystalline pigments and extenders in liquid paint and dry paint film. It is applicable to both water-reducible and solvent-reducible paint. It also may be used to identify pigment and extender in grind paste or alone as dry powder. It is not applicable to amorphous components such as carbon black, amorphous silica, or highly processed clay.

1.2 The values stated in SI units are to be regarded as standard. No other units of measurement are included in this standard.

1.3 *This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.* Specific hazard statements are given in Section 6.

2. Referenced Documents

2.1 *ASTM Standards:*[2]

D3925 Practice for Sampling Liquid Paints and Related Pigmented Coatings

3. Summary of Test Method

3.1 Every crystalline substance, in this case pigment or extender, has a characteristic X-ray diffraction pattern. Whether the substance is present alone or in a mixture, it produces its pattern independently and can be identified by it. An X-ray diffraction pattern of a sample is recorded. Each crystalline substance in the sample is identified by the Hanawalt or Fink method or other systematic procedure based on comparison of the diffraction pattern of the sample with reference patterns of standards.[3,4,5]

4. Significance and Use

4.1 The choice of pigments and extenders influences the appearance, durability, cost, and other properties of paint. This test method is a convenient way, and probably the most reliable, to identify pigments in paint.

5. Apparatus

5.1 *X-ray Diffractometer,* suitable for collecting intensity *versus* two theta (2θ) angle diffraction patterns in the range from 5° to 65° 2θ. It is preferred that the diffractometer be equipped with a copper target X-ray tube and a monochromator that passes only copper K-alpha radiation. If a monochromator is not available, then a suitable filter may be used to remove copper K-beta radiation from the diffracted X-ray beam. A nickel filter may be used for this purpose when a copper target tube is employed.

NOTE 1—Follow the recommendations of the manufacturer of the diffractometer used.

5.2 *Liquid Paint or Grind Paste:*

5.2.1 *Paint Shaker.*

5.2.2 *Film Applicator,* that will produce a 3- to 10-mil (75- to 250 µm) wet film thickness.

5.2.3 *Plastic Sheet,* such as polyester film, that contains no crystalline components that would produce interfering X-ray diffraction peaks and is not attacked by paint solvent.

5.2.4 *Perforated Suction Plate* or other flat surface.

5.3 *Paint Chips or Pigment Powder:*

5.3.1 *Miniature Reciprocating Ball Mill.*

5.3.2 *Stainless Steel Vials,* with agitator for ball mill.

5.3.3 *Powder Specimen Holders,* for X-ray diffractometer.

6. Hazards

6.1 *Precaution*—As exposure to excessive quantities of X-radiation is injurious to health, X-ray producing equipment

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[1] This test method is under the jurisdiction of ASTM Committee D01 on Paint and Related Coatings, Materials, and Applications and is the direct responsibility of Subcommittee D01.21 on Chemical Analysis of Paints and Paint Materials.

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[2] For referenced ASTM standards, visit the ASTM website, www.astm.org, or contact ASTM Customer Service at service@astm.org. For *Annual Book of ASTM Standards* volume information, refer to the standard's Document Summary page on the ASTM website.

[3] *Search Manual, (Hanawalt),* "Inorganic Phases," International Centre for Diffraction Data, (ICDD), Catalog No. HA 42, Newtown Square Corporate Campus, 12 Campus Blvd., Newtown Square, PA 19073-3273.

[4] *Search Manual, (Fink),* "Inorganic Phases," ICDD.

[5] "*Powder Diffraction File, Inorganic,*" ICDD.

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can be dangerous to both the operator and persons in the immediate vicinity unless safety precautions are strictly observed. 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 passes 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 NIST handbook,[6] "X-ray Recommendations of the International Roentgen Ray Committee on X-ray Protection," the manufacturer's instruction manual, and other standard publications on the subject. Inquiries should be made of state agencies as to existing requirements.

7. Specimen Preparation

7.1 Prepare a specimen from the sample using one of the following methods:

7.1.1 *Liquid Paint or Pigment Paste*—Thoroughly mix and sample the paint or paste in accordance with Practice D3925. Place a plastic sheet on the perforated suction plate. On the sheet make a drawdown of the sample. A wet film thickness of 3.0 to 10.0 mil (75 to 250 µm) is suggested. Cut from the drawdown on the plastic sheet a specimen of shape and size suitable for the mounting in the specimen holder of the diffractometer.

7.1.2 *Paint Chip*—Using a ball mill, grind to a powder the paint chip or, if it contains more than one layer, the part of the chip of interest. Prepare sufficient specimen to fill the specimen holder or to satisfy the requirements of the preparation technique to be used. In cases where the chip has a planar surface and uniform thickness, it may be possible to cut the chip to the required dimensions and mount it directly in the specimen holder with no preparation.

NOTE 2—X-ray diffraction patterns collected for specimens consisting of whole chips will reveal the composition of all paint layers probed by the X-rays. The depth probed will depend upon the wavelength of X-ray used, the composition of the paint layer or layers through which the X-rays pass, the composition of the deepest layer probed, the two-theta angle, and other factors. When a copper X-ray tube is used, the depth probed may range from as little as approximately 2 mils (75 µm) in the case of very high density coatings to as much as 197 mils (0.5 cm) in the case of unpigmented resin.

7.1.3 *Paint Film on Panel*—Cut from the panel a specimen of dimensions suitable for mounting in the specimen holder. As an alternative, it may be convenient to cut a piece of specimen to the same dimensions as the specimen holder and use the specimen without the holder.

NOTE 3—An X-ray diffraction pattern collected for coating on a panel will be a superposition of the diffraction patterns of the crystalline components in the coating on the panel and, to the extent that X-rays diffracted from the substrate reach the detector, the diffraction pattern of the substrate.

7.2 Mount the specimen in the specimen holder, taking care to insure that the surface of the specimen is flush with the optical plane of the holder.

NOTE 4—Failure to place the specimen surface in the optical plane illuminated by the X-ray source and viewed by the detector will result in a systematic shift from the correct position of peaks in the observed diffractogram. The greater the shift, the greater the difficulty in using the observed diffractogram to identify components in the specimen.

8. Procedure

8.1 *Experimental:*

8.1.1 Turn on the diffractometer and allow it to stabilize thoroughly before beginning collection of results. With the aid of the manufacturer's literature, select instrument operation conditions that permit collection of X-ray diffractograms spanning the two-theta range from 5 to 65°. The conditions chosen must be suitable for qualitative analysis of a multicomponent mixture assuming the presence of a minor component present at approximately one percent by weight. Results collected must be available as the *d*-spacing and intensity, preferably the integrated intensity, of each peak. In the case of diffractometers not equipped with a computer, the two-theta and intensity data must be measured manually from plotted diffraction patterns and the *d*-spacings then calculated from the two-theta angles of the peaks. In the latter case, the diffraction patterns must be plotted with two-theta scale expansion that permits the angle of each peak to be measured to the nearest 0.01 Å for peaks in the range above 3.5 Å and to the nearest 0.001 Å for peaks below approximately 3.5 Å.

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NOTE 5—If not already established, then make sure that the two-theta calibration of the goniometer of the diffractometer is verified at two or more angles in accordance with recommendations of the manufacturer and corrected as needed. The procedure will entail comparison of the observed and expected *d*-spacing of diffraction peaks of highly crystalline reference materials at least at one high and one low two-theta angle.

8.1.2 For each sample correct the diffraction pattern spanning at least from 5 to 65° two-theta.

9. Identification

9.1 Prepare a list of all diffraction peaks exhibited by the sample including both the *d*-spacing and intensity (preferably integrated intensity) of each peak, listed in decreasing order of *d*-spacing. The *d*-spacing of each peak should be stated at the nearest hundredth of an angstrom for *d*-spacings greater than 3.5 Å and stated to the nearest thousandth of an angstrom for *d*-values of less than about 3.5 Å.

9.2 Identify the crystalline phase or phases that account for all peaks in the diffraction pattern of the sample under analysis, using the Hanawalt Method, Fink Method, or other systematic procedure. Full identification of the crystalline components may be speeded by use of information about components known to be or suspected of being in the sample (that is, preconceived composition) or by use of the list of common pigments and extenders appended (see Table 1).

9.2.1 *Verification of Preconceived Components:*

9.2.1.1 Prepare a list of components known to be present or suspected of being present on the basis of other information, if

[6] *NIST Handbook, X-Ray Recommendations for the International Roentgen Ray Committee on X-Ray Protection*, NIST, Gaithersburg, MD, 20899.