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General Procedure References

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General Procedure Reference - 1 - 02

PMD 325 MESH SIEVING

Revised 8/03

SCOPE

The method is for the determination of the weight percent or ppm of particles to be found in an untreated, non-hydrophobic powder via a wet (water-washed) procedure.

METHODS

ASTM E 11	Wire Cloth Sieve Specifications
ASTM E1638	Standard Terminology Relating to Sieve Analysis
ASTM C430	Standard Method for Fineness of Hydraulic Cement by the 45 um (No. 325) Sieve
ASTM D718	Test Method of the Analysis of Aluminum Silicate Pigment
ASTM D185	Standard Method for Coarse particles in Pigments, Pastes, and Paints
TAPPI T681 cm-83	Screen Residue of Paper Coating Clays and Related Pigments

REFERENCE MATERIALS

Portland Cement: NIST SRM 114

KEY POINTS

- Specification of sieve: U.S. Standard Sieve Series or other, free of defects (tears, holes, warping)
- Calibration of sieves: use of NIST SRM 114
- Sampling procedure
- Sample size
- Sample preparation: use of dispersants, blenders
- Water pressure/filtration of water to remove oversize/length (time) of water wash
- Water temperature: warm water may contribute to dispersion/dissolution of particles and result in lower retains
- Drying temperature of sieves: mfg. recommendations are generally 80° C or less
- Drying of sample/residue: oven type, temperature, on sieve or removed prior to drying
- Collection/weight back of residue: into porcelain dishes, onto filter membranes, etc.
- Calculations/reporting: % or ppm

COMMENTS

October 1999: Particles greater than 325 mesh (45 microns) may be determined in surface treated, hydrophobic powder samples with the use of an appropriate solvent; in the case of stearic acid/stearate treatments, alcohols and dishwashing liquids have been found useful. Ultrasonic and/or high speed mixing of agglomerations formed in the dispersion process may be appropriate and should be agreed upon in any method to determine these values in such products.

General Procedure Reference - 2 - 02

PMD **pH**

Revised 8/03

SCOPE

The method is for the determination of pH in aqueous liquids or aqueous dispersions of powdered mineral particles/samples.

METHODS

ASTM D1208	Test Methods for Common Properties of Certain Pigments
ASTM E70	Test Method of Aqueous Solutions with Glass Electrode (By reference in DI 208)
TAPPI T667 cm-90	pH of Fillers and Pigment Slurries

REFERENCE MATERIALS

Commercially available pH 3, 7, 10 buffer solutions, either as ready to use or from dilutable vials.

KEY POINTS

- Instrument type/temperature compensation capability (manual or automatic)
- Electrode type: pH and reference electrodes, combination electrodes
- Calibration: single point, multi point; single point could lead to error if the analyte pH does not approximate that of the standard.
- Sample concentration
- Sample preparation: stirring, freshly boiled/cooled water for dry pigments
- Measurement conditions: temperature, stirred or not (effect of stirring during analysis)
- Report: to 0.1 pH units or other with agreement and appropriate method

General Procedure Reference – 3A – 02

PMD **SEDIGRAPH 5100**

Revised 8/03, page 1/2

SCOPE

The method is for the determination of particle size distribution (equivalent spherical diameter - ESD - vs. weight percent) using a Micromeritics SediGraph, Model 5100 (X-ray sedimentation technique) and is applicable to particles from 100 microns (maximum) to 0.18 microns ESD.

METHODS

ASTM E 1617	Standard Practice for Reporting Particle Size Characterization Data
ASTM D 1199	Standard Specification for CaCO ₃ Pigments (Section 3.3.5.1 note:the SediGraph, manufactured by Micromeritics, has been found suitable for this purpose.)
TAPPI UM655	Particle Size Distribution of Pigments and Fillers
TAPPI T649 cm-90	Particle-size Distribution of Coating Clay (A hydrometer method with Section 3 note: ...use of procedures such as determining the change in X-ray absorbence... for measuring changes in slurry concentration are recommended for the fine particle-size range.)

REFERENCE MATERIALS *See Comments*

- Fine Particle Size CaCO₃ Reference Material, NIST Traceable: Micromeritics part Number 004/16835/00 *Comment: Material must be dispersed using ultrasonic equipment; not readily dispersed using high speed/shear mixers. Approximately 0.7 micron median diameter.*
- Garnet Reference Material: Micromeritics part Number 004/16810/00
- Medium Particle Size Reference Material (Garnet), NIST Traceable: Micromeritics Part Number 004-16837-00 *Comment: Sample preparation with stirring and ultrasonic. Approximately 3.8 micron median diameter.*
- Silicon Nitride: NIST SRM 659
- Quartz 0.35 - 3.50 microns: BCR CRM 066
- Quartz 2.30 - 32.00 microns: BCR CRM 067
- Quartz 1.20 - 20.00 microns: BCR CRM 070

KEY POINTS

- Equipment: Instrument Model and software (ex: MicMOS *SEE COMMENTS* or Windows software/versions, Baseline/Full Scale Option, High Speed Analysis Option, MasterTech autosampler use), large bore/small bore cell/tubing, tubing type (Tygon, PharMed, LFL)
- Calibration: use of Reference Materials (above) to verify instrument performance, frequency of calibration/verification *SEE COMMENTS*
- Sample Preparation: Baseline/dispersing liquid (water, water/surfactant, SediSpense liquids), sample concentration, dispersant type/amount, dispersion (mixer/speeds/times, ultrasonic baths/probes, times/power, MasterTech ultrasonic/mixer times)
- Analysis Conditions: Sample Material Properties Options (Specific gravity, starting/ending diameter)
- Analysis Liquid Options (SediGraph default options for various liquids)
- Analysis Options (Stirrer times/speeds, sonication times, pump on during analysis, pump speed, bubble detection, stop at selected mass percent, rinse - auto/manual and number)
- Report Options: Distribution/plot types

COMMENTS

October 1999: Micromeritics is no longer offering new versions of the MicMOS software. All new SediGraph 5100 instruments are shipped with Windows software. October 1999: For users of Micromeritics Service contracts, the use of either of their reference materials (Fine Particle Size CaCO₃ or Garnet) may be specified in that contract to verify performance of instrument at time of service.

August 2003: Surface treated products may be analyzed on the SediGraph. There are two analytical schemes:

- Decoating the sample: Organic coatings (e.g. - fatty acids, such as stearic acid) may be removed in a furnace. Temperature, time, crucible size, and sample amount are variables to consider. It may also be possible to solvent strip the product.
- Dispersion of the treated product: Some coatings may not be readily removed thermally. It may be possible to disperse the sample. Dispersant type(s)/amount(s), solvent vs. aqueous dispersion, and dispersion method (mixing, ultrasonification, time) are some variables to consider.

Subsequent sample preparation (as noted in Key Points above) for presentation to the instrument and analysis conditions should then be agreed upon. In any analytical scheme, method development should optimally include analysis of both untreated feed and decoated (or dispersed) product samples to verify the agreement of data between the two. The particle size distribution of the treated product (by any scheme) should agree closely with that of the feed run by a standard method.

General Procedure Reference – 3B - 02

PMD

CILAS 920

Revised 8/03

SCOPE

This method describes a procedure for determining the particle size distribution of ground limestones in the range of 0.3 - 400 micrometers. This method uses automatic laser light diffraction principles to size particles. The Fraunhofer diffraction theory is used to determine and present data on a volume basis as equivalent spherical diameter.

METHODS

A sample of ground limestone dispersed in water is circulated through the path of a laser beam. The suspended particles scatter the laser light beam, which form a diffraction pattern. The light fluctuations are converted to electrical signals that are transmitted to a microprocessor for calculation and data presentation.

REFERENCE MATERIALS

None

KEY POINTS

- Equipment: Cilas Laser Model 920, computer, mouse, balance (accurate to ± 0.01 gram), water (filtered min 5 micron)
- Procedure: Turn power on (Cilas and computer), go into particle *size* program. Click on "calibers" to initiate background check. Click on "sample measurement" to initiate stirrer and ultra sonic. Add sufficient amount of limestone to obtain a 70-150% concentration level. Test results will appear on the screen when testing complete.
- Report Options: Record data from screen or print out graph and data.

General Procedure Reference – 3C - 02

PMD

CILAS 715

Revised 8/03

The CILAS Model 715 is no longer supported by the manufacturer. Parts for the instrument are increasingly harder to find. It is recommended that users evaluate other laser particle sizing instruments that are available. There may be several instrument models and/or automated sampling accessories available from each manufacturer. Manufacturers of such instrumentation include but are not limited to:

- CILAS
- Coulter Corporation
- Leeds and Northrup Malvern Instruments
- Micromeritics

There may be several instrument models available from each manufacturer. Users are encouraged to determine the capabilities of the different instruments and which one(s) will meet the criteria for the particular analysis. There are also other particle size instruments based upon techniques other than laser scattering that may meet the requirements of the analysis.

General Procedure Reference – 5 - 02

PMD **Brightness**

Revised 8/03, page 1/2

SCOPE

The method is for the determination of the brightness/color/reflectance of a powder mineral sample. It involves critical specification of sample preparation, instrument geometry, calibration standard, and color system.

METHODS

ASTM E805	Standard Practice of Identification of Instrumental Methods of Color or Color-Difference Measurement of Materials
ASTM E179	Standard Practice for Selection of Geometric Conditions for Measurement of Reflection and Transmission Properties of Materials
ASTM E259	Standard Practice for Preparation of Pressed Powder White Reflectance Factor Transfer Standards for Hemispherical Geometry
ASTM E1331	Standard Test Method for Reflectance Factor and Color by Spectrophotometry Using Hemispherical Geometry
ASTM E 1164	Standard Practice for Obtaining Spectrophotometric Data for Object-Color Evaluation
TAPPI T525 om-92	Diffuse Brightness of Pulp (d/0°)
TAPPI T534 om-86	Brightness of Clay and Other Mineral Pigments (Diffuse Blue Reflectance)
TAPPI T646 om-86	Brightness of Clay and Other Mineral Pigments (45°/0°)

REFERENCE MATERIALS *See Comments*

- Barium Sulfate: DIN 5033
- Barium Sulfate: Eastman Kodak White Reflectance Standard 6091
- Opal Glass: BCR CRM 406A (50 mm diameter) and 406B (100 mm diameter) Opal Glass: NIST SRM 2015
- Manufacturers' standards

KEY POINTS

- Equipment: Instrument geometry (Directional, i.e. - 45°/0°, or Integrating, i.e. - diffuse/0° or diffuse/6° or 8°) and model are critical, Color System, powder mills (hammer mills, rotor mills, coffee grinders and their set-up/use), sample presses *SEE COMMENTS for sample preparation.*
- Calibration: Use of Reference Materials (above) to verify instrument performance, preparation of Reference Materials, frequency of calibration/verification *SEE COMMENTS*
- Sample Preparation: drying/dryness of sample, grinding prior to sample plaque press-out *SEE COMMENTS*, sample weight/volume, pressure applied/time, plaque press glass surface (glossy side or matte/frosted side)
- Analysis Conditions: measurement aperture diameter (where applicable), number of measurements (where applicable), UV filter in/out (where applicable)
- Report Options: numerous Color Systems are available to report brightness/color values, including but not limited to Reflectance (i.e. - Rx, Ry, Rz), TAPPI R457, Hunter L,a,b, CIE L,a,b)

COMMENTS

October 1999: The instrument manufacturer does or should include appropriate Reference Materials for calibration of the instrument zero and reflectance values.

October, 1999: Per ASTM E259, suitable plaque presses are available several manufacturers: Carl Zeiss Canada, Ltd., Don Mills, ON, and Technidyne Corporation, New Albany, IN.

October 1999: The reflectivity of pressed powder plaques has been found to be influenced by the degree of agglomeration of the sample powder. Thus, the degree of de-agglomeration/dis-aggregation provided by the powder mill is critical to the reproducibility of intra- and inter-laboratory agreement.

General Procedure Reference – 7 - 02

PMD FINENESS OF GRIND

Revised 8/03

SCOPE

The method is for the determination of fineness of grind (a measure of the coarsest particles not dispersible in the vehicle) in Hegman (NS or North Scale) and/or micron units.

METHODS

ASTM D 1210 Standard Test Method for Fineness of Dispersion of Pigment Vehicle Systems

REFERENCE MATERIALS

None known/none referenced in ASTM D1210

KEY POINTS

- Equipment: Gage type (single/double path) calibrated in Hegman and micron units (or other scale with agreement), scraper free of nicks or wear (replace/recondition), mixer type
- Sample Preparation: As used for pigment-vehicle systems, follow ASTM D 1210. For dry pigment evaluations, agree upon sample weight, vehicle type (alkyd resins, nondrying oils *SEE COMMENTS*) and weight, mixing procedure (mixer type, speed, time)
- Analysis Conditions: Read drawdown within 10 seconds.
- Report: Number of replicates, Hegman value to nearest 1/4 unit, microns to 5, average value.

COMMENTS

October, 1999: *Per ASTM D1210, section 1.2 This standard may involve hazardous materials ... and does purport to address all of the safety problems associated with its use. It is the responsibility of the user to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.* The use and proper disposal of alkyd resin vehicles in this analysis is just such a problem. This analysis may be done reliably and safely using widely available vegetable oils with a minor amount of method development.

General Procedure Reference – 9 - 02

PMD MOISTURE

Revised 8/03

SCOPE

The method is for the thermogravimetric determination of "moisture" (any compounds volatile at the measurement temperature) using an oven and laboratory balance.

METHODS

ASTM D280 Method A: Test method for Hygroscopic Moisture in Pigments TAPPI T671
cm-85 Free Moisture in Fillers and Pigments

REFERENCE MATERIALS

None

KEY POINTS

- Equipment: Oven (convection/forced air), balance, analysis vessel type/size/cover *SEE COMMENTS*
- Calibration: Temperature calibration/frequency for oven, balance tolerances and calibration/frequency
- Analysis Conditions: Sample size, temperature, time
- Report: to % or ppm

COMMENTS

October 1999: The points discussed refer to a simple "oven method" for determination of moisture; it involves the use of relatively inexpensive and readily available laboratory equipment. There are numerous instruments on the market today that may be referred to collectively as "moisture balances." While this instrumentation may be a dedicated analyzer and not always available to all parties involved in standardization, these instruments have been shown in capability/reproducibility studies to provide results superior (i.e. - more sensitive and lower standard deviations) to the more manual "oven method." If the levels of moisture are particularly low (<0.10%) and/or critical to product use, these instruments should be evaluated. Manufacturers of such instrumentation include but are not limited to:

- Arizona Instruments
- Denver Instruments
- Mettler Toledo
- Ohaus
- Omnimark Instruments
- Sartorius

There may be several instrument models available from each manufacturer. Users are encouraged to determine the capabilities of the different instruments and which one(s) will meet the criteria for the particular analysis.

General Procedure Reference – 10 - 02

PMD SURFACE TREATMENT

Revised 8/03

SCOPE

The method is for the determination of the weight percent of a functional surface treatment applied to a powder product using (a) an oven method to determine oven-dry weight, (b) a furnace method to determine weight loss for the untreated product (blank), (c) a furnace method to determine gross loss of treatment, and a calculation to determine the net percent surface treatment.

METHODS

PMD – 5 Test Procedures & References for The Evaluation of Calcium Carbonate and Ground Limestone

REFERENCE MATERIALS

None

KEY POINTS

- Equipment: Oven (convection/forced air), furnace, balance, analysis vessels/size/cover *SEE COMMENTS*
- Calibration: Temperature calibration/frequency for oven/furnace, balance tolerances and calibration/frequency
- Analysis Conditions: Sample size, oven-drying temperature/time, furnace temperature/time (temperature should be high enough to remove treatment compound but not high enough to decompose the product itself.)
- Calculations/Report: Calculation of moisture, blank, and gross treatment values to determine net treatment, significant figures for % reported

COMMENTS

October, 1999: As noted for the referenced moisture method, the points discussed refer to a simple "oven/furnace method" for the determination of surface treatment using relatively inexpensive, readily available laboratory equipment and techniques." There are Thermogravimetric Analyzers (TGAs) available that, while more expensive to purchase, are much more sensitive and may be applicable to specialized determinations of very low surface treatments or where very small sample amounts are available.

General Procedure Reference – 11 - 02

PMD SURFACE AREA ANALYSIS

Revised 8/03

SCOPE

The method is for the determination of the specific surface area (surface area per unit weight) of a given sample of pulverized mineral.

METHODS

ASTM C1069-86 (1997)	STM for Specific Surface Area of Alumina or Quartz by Nitrogen Adsorption
ASTM D3663-99	STM for Surface Area of Catalysts and Catalyst Carriers
ASTM D5604-96	STM for Precipitated Silica-Surface Area by Single Point B.E.T. Nitrogen Adsorption
ASTM D1993-91 (1997)	STM for Precipitated Silica-Surface Area by Multipoint BET Nitrogen Adsorption
ASTM D4780-95	STM for Determination of Low Surface Area of Catalysts by Multipoint Krypton Adsorption

REFERENCE MATERIALS

- Silicon Nitride Powder NIST SRM 1899
- Silicon Nitride Powder NIST SRM 1900
- Calcined Kaolin NIST RM 8570

KEY POINTS

- Analytical Device
- Dryness of sample (sample preparation)
- Dispersion of sample (sample preparation)
- Sample size (for adequate total surface area)
- Cleanliness of cell and sample holder
- Purity of gases used
- Offgasing temperature

COMMENTS

Most mineral samples will have adequate total surface area to use nitrogen (single point or multi-point BET) adsorption techniques. However, some very coarse materials may require the use of a krypton gas blend. Cleanliness of sample is vital due to the large amount of error added when airborne particles are allowed to settle into and blend with an open sample.

GLOSSARY OF TERMS

ASTM	American Society for Testing and Materials Philadelphia, PA
BCR	Community Bureau of Reference European Commission Directorate General for Science, Research and Development Brussels, Belgium
CRM	Certified Reference Material
NIST	National Institute of Standards and Technology Gaithersburg, MD
SRM	Standard Reference Material

The inclusion of any Reference Material in the following methods notes is for member use and reference. It is not intended to be an exhaustive/complete compilation of the materials available from all manufacturers of such products. Nor does it constitute a recommendation that these materials are the only products that may be used when evaluating these methods of analysis. It has been compiled from materials referenced in the recognized standards organization methods and/or from member company use.