Designation: D6602 – 13 (Reapproved 2022)ε1

Standard Practice for Sampling and Testing of Possible Carbon Black Fugitive Emissions or Other Environmental Particulate, or Both[1]

This standard is issued under the fixed designation D6602; 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.

ε1 NOTE—Corrected 6.2.1 editorially in February 2023.

1. Scope

1.1 This practice covers sampling and testing for distinguishing ASTM type carbon black, in the N100 to N900 series, from other environmental particulates.

1.2 This practice requires some degree of expertise on the part of the microscopist. For this reason, the microscopist must have adequate training and on-the-job experience in identifying the morphological parameters of carbon black and general knowledge of other particles that may be found in the environment. In support of this analysis, Donnet's book[2] is highly recommended to be used as a technical reference for recognizing and understanding the microstructure of carbon black.

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

1.4 *This standard may involve hazardous materials, operations, and equipment. 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, health, and environmental practices and determine the applicability of regulatory limitations prior to use.*

1.5 *This international standard was developed in accordance with internationally recognized principles on standardization established in the Decision on Principles for the Development of International Standards, Guides and Recommendations issued by the World Trade Organization Technical Barriers to Trade (TBT) Committee.*

2. Referenced Documents

2.1 *ASTM Standards:*[3]

D1619 Test Methods for Carbon Black—Sulfur Content

D3053 Terminology Relating to Carbon Black

D3849 Test Method for Carbon Black—Morphological Characterization of Carbon Black Using Electron Microscopy

3. Terminology

3.1 *Definitions of Terms Specific to This Standard:*

3.1.1 *aciniform*—shaped like a cluster of grapes.

3.1.1.1 *Discussion*—The spheroidal primary particles of carbon black are fused into aggregates of colloidal dimension forming an acinoform morphology.

3.1.2 *aciniform carbon*—colloidal carbon having a morphology consisting of spheroidal primary particles (nodules) fused together in aggregates of colloidal dimension in a shape having grape-like clusters or open branch-like structures

3.1.3 *carbon black, n*—an engineered material, primarily composed of elemental carbon, obtained from the partial combustion or thermal decomposition of hydrocarbons, existing in the form of aggregates of aciniform morphology which are composed of spheroidal primary particles characterized by uniformity of primary particle sizes within a given aggregate and turbostratic layering within the primary particles.

3.1.3.1 *Discussion*—Particle size and aggregate size (number of particles per aggregate) are distributional properties and vary depending on the carbon black grade. Transmission electron micrographs shown in Annex A2 demonstrate that while particle and aggregate sizes vary greatly within a given grade of carbon black, the primary particle size is essentially uniform within an individual aggregate.

3.1.4 *chain of custody*—a document describing the condition of a sample during its collection, analysis, and disposal.

[1] This practice is under the jurisdiction of ASTM Committee D24 on Carbon Black and is the direct responsibility of Subcommittee D24.66 on Environment, Health, and Safety.

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[2] Hess, W.M. and Herd, C.R., *Carbon Black Science and Technology*, Edited by Donnet, J.B., Bansal, R.C., and Wang, M.J., Marcel Dekker, Inc., New York, NY, 1993, pp. 89–173.

[3] 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.

3.1.5 *char*—a particulate larger than 1 µm made by incomplete combustion which may not deagglomerate or disperse by ordinary techniques, may contain material which is not black, and may contain some of the original material's cell structure, minerals, ash, cinders, and so forth.

3.1.6 *fugitive dust*—transitory, fleeting material comprised of particulates foreign to the surface of deposition.

3.1.7 *fungus, sooty mold, mildew, biofilm*—particulates from a superficial growth that grows on living and decaying organic matter.

3.1.8 *mineral dust*—naturally occurring inorganic particulates inherent to the area such as soil minerals.

3.1.9 *pollen*—particulates from a mass of microspores in a seed plant.

3.1.10 *rubber dust*—finely divided soft particulates abraded from rubber.

3.1.11 *sample*—a small fractional part of a material or a specified number of objects that are selected for testing, inspection, or specific observations of particular characteristics.

3.1.12 *soot*—a submicron black powder generally produced as an unwanted by-product of combustion or pyrolysis. It consists of various quantities of carbonaceous and inorganic solids in conjunction with adsorbed and occluded organic tars and resins.

3.1.12.1 *Discussion*—The carbonaceous portion also is colloidal and often has the aciniform morphology. Soot may have several carbon morphologies. Examples of soot are carbon residues from diesel and gasoline engines, industrial flares, sludge pits, burning tires, and so forth.

3.1.13 *sticky tape*—a section of tape with a sticky, solvent-soluble adhesive used in the collection of particles from surfaces.

3.1.14 *surface*—the outer surface, facing, or exterior boundary of an object capable of supporting carbon and other fugitive and natural occurring dusts and particulates.

3.1.15 *turbostratic*—a type of graphitic crystallographic structure in which there is no symmetry along the z-axis.

3.2 *Acronyms:*

3.2.1 *EDS*—energy dispersive spectroscopy associated with SEM and TEM for the identification of elemental composition,

3.2.2 *LM*—light microscope,

3.2.3 *PLM*—polarizing light microscope,

3.2.4 *SEM*—scanning electron microscope,

3.2.5 *TEM*—transmission electron microscope.

3.2.6 *WDS*—wavelength dispersive spectroscopy associated with SEM and TEM for the identification of elemental composition.

NOTE 1—Standard terminology relating to carbon black can be found in Terminology D3053.

4. Summary of Practice

4.1 This practice describes the procedures and protocols to follow in order to collect fugitive emission/environmental samples and identify the classes of particulate present including materials consistent or inconsistent with manufactured carbon black (referred to simply as carbon black). A semi-quantitative estimate of the percentage of each type of surface particulate component is determined using polarized light microscopy (PLM). However, PLM analysis cannot differentiate between carbon black and soots (black carbons) that may come from many sources in the environment. Therefore, transmission electron microscopy (TEM) analysis is mandatory in determining whether a sample contains carbon black. Because the preparation steps for the TEM analysis eliminates certain types of particles and concentrates only the fine (small) particles from the sample, the TEM analysis alone cannot be used to estimate the amount of carbon black or other particle type in the whole sample. Either the PLM or TEM analysis may be done first.

4.2 Section 6 provides guidelines for proper sampling and handling of fugitive emission/environmental samples. Sections 8 and 9 describe the analysis of the sample using polarized light microscopy (PLM) and transmission electron microscopy (TEM). The TEM analysis is critical in determining if the collected sample is consistent or inconsistent with carbon black. Use of the TEM analysis is mandatory in determining whether a sample is positive for carbon black. The use of the PLM analysis is not mandatory when the TEM analysis finds no aciniform aggregates resembling carbon black. Section 9 describes additional ancillary techniques that may be included in a sample analysis for purposes of providing supporting information as to the nature of the sample material. These are situation-dependent methods and can provide critical identification information in certain cases.

4.3 A block diagram is presented in Fig. 1 to give a possible scheme to follow in performing this analysis. However, it should be noted that this diagram is a suggestion, not a requirement. Either the PLM or TEM analysis may be performed first.

5. Significance and Use

5.1 There are a variety of darkening agents that contribute to air and surface contamination in industrial, urban and rural environments. Biofilms (fungal and algal), soil minerals, plant fragments, rubber fragments, metal corrosion and soot are common darkening agents. Soot is formed as an unwanted by-product of combustion and consequently varies widely with the type of fuel and combustion conditions. Carbon black, on the other hand, is purposely produced under a controlled set of conditions. Therefore, it is important to be able to distinguish carbon black from soot, as well as other environmental contaminants.

6. Sampling

6.1 The area to be sampled should be representative of the contaminated area. For sampling, choose an area that appears to contain black particulates. In some situations, the same general surface can be used for gathering all test samples for each property site location or area.

6.2 *Equipment:*

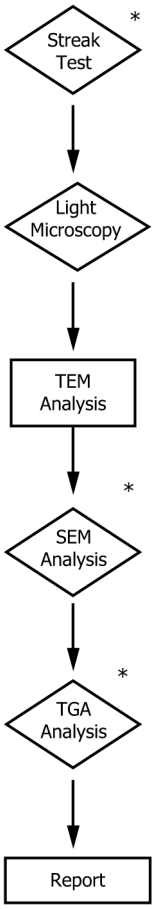

FIG. 1 Block Diagram of Suggested Analysis Scheme for Samples

6.2.1 *Polyester Wipes* (Texwipe Alphasat synthetic fiber wipes in 70 % alcohol/30 % DI water or equivalent).

6.2.2 *Sticky tape* (Scotch Crystal Clear Tape, No. 25 or equivalent).

6.2.3 *Polyethylene Ziploc Bags.*

6.2.4 *Standard Glass Microscope Slides.*

6.3 Samples are to be collected by the following two techniques (wipe and tape) in accordance with 6.3.1 and 6.3.2. Precautions should be taken to carefully collect, handle, and transport samples in a manner that will not cause further contamination.

6.3.1 *Technique I: Wipe Sampling*—Collect the wipe sample by wiping the surface to be sampled with a polyester wipe to remove surface particulates and solids. Light pressure on the wipe should be sufficient. Make sure that enough of an area has been wiped to load the surface of the wipe. Place the exposed wipe in a ziploc bag and label.

6.3.2 *Technique II: Tape Sampling*—Prepare a tape-lift slide by applying an appropriate length of tape to a clean glass microscope slide, leaving a tab for easy removal of the tape. Remove particulates and solids from surfaces by removing the tape from the prepared slide and applying it to the surface to be sampled. Carefully remove the tape and place back across the glass microscope slide. Take care not to overload the tape.

6.3.3 All collected samples must be clearly identified at the time of collection.

6.4 At the time of sample collection, complete a sampling record (Table 1) and also complete a chain of custody record (Table 2).

6.5 This practice does not preclude examination of samples collected by other means than the preceding, such as polyethylene glove wipes, filter paper, samples of clothing, material scraped directly from the surface of interest, and so forth, or a large sample taken in other containers at a spill site. However, these samples always require thorough identification taken at the time of sample collection.

6.6 It is advisable in the case of repeated incidents to clean the surface between sampling.

7. Examination by Light Microscopy

7.1 *Summary of Test Method*—This method of examination is a screening test method that provides an overview of the bulk composition of the sample through examination under a light microscope. This portion of the method is mandatory except in cases where TEM examination gives no positive results for aciniform aggregates resembling carbon black and there is no

TABLE 1 Example Sampling Record

Sample Identification Number: _____

Sample Location: _____

Date of Sampling: _____

Comments:

_____
_____
_____
_____
_____
_____
_____
_____
_____.