



Standard Test Method for Gravimetric Determination of Carbon Black in Nylon Materials (PA)¹

This standard is issued under the fixed designation D 6260; 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 is applicable to the determination of carbon black in a specific class of nylon materials, weather stable, listed in the property tables of Classification System D 4066.

1.2 The values stated in SI units are to be regarded as the 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.* See Note 2 and Note 3 for specific hazard statements.

NOTE 1—There is no similar or equivalent ISO standard.

2. Referenced Documents

2.1 ASTM Standards:

D 833 Terminology Relating to Plastics²

D 1898 Practice for Sampling of Plastics²

D 4066 Classification System for Nylon Materials³

3. Terminology

3.1 *Definitions*—Terms applicable to this test method are defined in Terminology D 833.

4. Significance and Use

4.1 Carbon black is an additive in a specific class of nylon materials, weather stable, listed in the property tables of Classification System D 4066.

4.2 This test method determines the presence and level of carbon black to ensure the suitability of the nylon material in weatherable applications.

5. Apparatus

5.1 *Normal Laboratory Apparatus.*

5.2 *Filtering Apparatus*, capable of containing fine particles of carbon black. Systems that have been used successfully include the following:

5.2.1 *Millipore Filter Holder, Glass Microanalysis System B*, Catalog Number XX100253, with Nucleopore Polycarbonate Filters, Type PC, 0.2 μm , 25 mm diameter.

5.2.2 *Good Crucible with Glass Microfiber Filters*, Whatman Catalog Number 1825-024, 2.4 cm GF/F.

5.3 *Drying Oven*, capable of minimum 70°C temperature.

6. Reagents

6.1 Acids for the hydrolysis step should be chosen by the user for the specific nylon under test.

6.1.1 *For Nylon 6 and 66*—1 + 1 hydrochloric acid. Prepare in a hood by slowly mixing one part reagent grade HCl into one part deionized water.

6.1.2 *For Nylon 612*—1 + 1 nitric acid. Prepare as in 6.1.1.

NOTE 2—Use caution. Never mix water into an acid. Continually mix, slowly, in an open flask, when adding acid to water.

NOTE 3—Hydrochloric and nitric acids are corrosive liquids. Wear rubber gloves, rubber apron, and chemical goggles when handling the acids and their solutions.

7. Test Specimens

7.1 Sample the material in accordance with Practice D 1898

7.2 Specimens should contain less than 0.3 % moisture to avoid weighing errors.

8. Procedure

8.1 Perform the hydrolysis step in a hood.

8.2 Into a clean, dry, 250 ml Erlenmeyer flask, transfer a sample (weighed to the nearest 0.0001 g) that will yield 20 to 100 mg of carbon black. Record the sample weight.

8.3 Add, by graduated cylinder, 100 mL of the appropriate acid solution and attach a water-cooled condenser.

8.4 Apply heat until the sample solution just boils. Continue slight boiling for 2 to 3 h.

¹ This test method is under the jurisdiction of Committee D-20 on Plastics and is the direct responsibility of Subcommittee D20.15 on Nylon Materials. Current edition approved May 10, 1998. Published April 1999.

² *Annual Book of ASTM Standards*, Vol 08.01.

³ *Annual Book of ASTM Standards*, Vol 08.02.

8.5 Remove the flask from the condenser without disturbing any material that may have collected in the condenser.

8.6 Rinse the material from the condenser with methanol and discard.

8.7 Prior to the next step, heat to a boil 200 to 400 mL of the acid solution; this will be used to rinse the sample following filtration.

8.8 Dry the filter (see 5.2) to a constant weight, record to the nearest 0.0001 g, and place in its holder. Record the weight. Wet the filter with a few milliliters of the acid solution.

8.9 Transfer the filter and holder to a vacuum flask.

8.10 Carefully filter the hydrolyzed sample solution, under slight vacuum. Immediately after the sample solution has completed filtering, begin rinsing with the heated acid solution (must be kept hot during filtration, to avoid erroneous high results).

8.11 At the completion of the filtration, and rinses if needed, transfer the filter to a dried, tared (record weight to nearest 0.0001 g) petri dish.

8.12 Dry the dish and filter in a vacuum oven for 1 h at 70°C. Cool in a desiccator for 15 min and record the weight to the nearest 0.0001 g.

NOTE 4—If there is no presence of mineral or glass (fiber) fillers suspected conclude the test here and calculate carbon black (see 9.1).

8.13 Place the dish and filter in an oxygen plasma low-temperature asher until all organic matter has been destroyed (5 to 6 h).

8.14 Remove and cool in a desiccator. Record weight of dish and residue.

9. Calculations

9.1 For sample with no fillers, calculate the carbon black content as follows:

$$\text{carbon black, \%} = \frac{E - (B + D)}{A} \times 100 \quad (1)$$

where:

A = sample weight (see 8.2),

E = weight of dish, filter, and sample (see 8.12),

B = weight of filter (see 8.8), and

D = weight of dish (see 8.11).

9.2 For sample containing fillers (fiber glass, mineral, etc.), calculate the carbon black content as follows:

$$\text{carbon black, \%} = \frac{E - (F + B)}{A} \times 100 \quad (2)$$

where:

F = weight of dish and filler after ashing (see 8.14).

10. Report

10.1 Report the following information:

10.1.1 Source and identity of sample,

10.1.2 Test results, to nearest 0.01 %,

10.1.3 Test deviances, if any, and statement of expected on results.

11. Precision and Bias

11.1 A round robin to establish the precision and bias of this test method is not available at this time. The repeatability standard deviation has been determined to be the following:

n	
2.01	average = 2.09
2.00	
2.06	standard deviation = 0.08
2.14	
2.24	95 % C. I. = 0.0701

11.2 A reproducibility study will be made at the earliest possible time and when sufficient laboratories are located.

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