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Standard Practice for
Carbon Black—Evaluation of an Industry Reference Black[1]

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~~ϵ^1 NOTE—Editorial changes were made throughout in January 1997.~~

1. Scope

1.1 This practice offers guidelines for the production and testing for uniformity of a lot of carbon black to be used as an Industry Reference Black (IRB).

1.2 The values stated in SI units are to be regarded as the standard. The values given in parentheses are for information only.

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.*

2. Referenced Documents

2.1 *ASTM Standards:*

D 412 Test Methods for Vulcanized Rubber and Thermoplastic ~~Rubbers and Thermoplastic Elastomers— Tension~~ Elastomers—Tension[2]

D 1506 Test Methods for Carbon Black—Ash Content[2]

D 1508 Test Method for Carbon Black, Pelleted~~—~~ Fines ~~Co~~ and At~~en~~trition[2]

D 1509 Test Methods for Carbon Black—Heating Loss[2]

D 1510 Test Method for Carbon Black—Iodine Adsorption Number[2]

D 1513 Test Method for Carbon Black, Pelleted—Pour Density[2]

D 1514 Test Method for Carbon Black—Sieve Residue[2]

D 1618 Test Method for Carbon ~~Extractables—Toluene Discoloration~~ Extractables—Transmittance of Toluene Extract[2]

D 1765 Classification System for Carbon Blacks Used in Rubber Products[2]

D 2414 Test Method for Carbon ~~Black—*n*-Dibutyl Phthalate~~ Black—Oil Absorption Number[2]

D 3191 Test Methods for Carbon Black Evaluation in SBR (Styrene-Butadiene Rubber)—Recipe and Evaluation Procedures[2]

D 3192 Test Methods for Carbon Black Evaluation in NR (Natural Rubber)[2]

D 3265 Test Method for Carbon Black—Tint Strength[2]

D 3493 Test Method for Carbon ~~Black—Dibutyl Phthalate~~ Black—Oil Absorption Number of Compressed Sample[2]

D ~~3765 Test~~ 5230 Test Method for Carbon ~~Black—CTAB (Cetyltrimethyl-ammonium Bromide) Surface Area~~ Black—Automated Individual Pellet Hardness[2]

D ~~4324 Test Method for Carbon Black, Pelleted—Pellet Attrition[2]~~

~~D 4820 Test Method for Carbon Black—Surface Area by Multipoint B. E. T. Nitrogen Adsorption[2]~~

~~D 5230 Test Method for Carbon Black Automated Individual Pellet Crush Strength[2]~~

~~D 5816 Test~~ 6556 Test Methods for Carbon Black—Total and External Surface Area by ~~Multipoint~~ Nitrogen Adsorption[2]

3. Significance and Use

3.1 These guidelines are intended to ensure that IRBs are evaluated by a standard procedure.

3.2 These guidelines are to be used to establish the average physicochemical and physical rubber properties of a lot of carbon black to be used as an IRB.

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[1] This practice is under the jurisdiction of ASTM Committee D-24 on Carbon Black and is the direct responsibility of Subcommittee D24.61 on Carbon Black Sampling and Statistical Analysis.

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[2] *Annual Book of ASTM Standards*, Vol 09.01.

4. Production, Quality Control, and Quality Assurance

4.1 It is assumed that the manufacturer of the IRB will use state-of-the-art techniques to ensure maximum uniformity throughout the entire production ~~run of at least 270 000 kg (600 000 lb).~~ run. The production should be made in one continuous production lot run. The testing called for in this practice is not intended to be a substitute for in-process quality ~~control.~~

~~4.2 It is assumed that the manufacturer of the IRB will use blending as needed to ensure uniformity throughout the entire production lot.~~ control. This interlaboratory study is only adequate to verify the quality of a homogeneous lot.

4.2 The size of the lot is determined by historical records on the rate of use. The lot should have an expected life of 8 to 10 years at the most recent rate of use.

4.3 The black should be bagged in 50–lb polyethylene bags to reduce moisture incursion. Each pallet of bagged black should be wrapped in plastic to reduce environmental exposure. The bagged black will be segregated into at least twelve ~~23 000-kg (50 000-lb) lots (20 skids). Partial lots of less than 23 000 kg (50 000 lb) will be discarded.~~ equal sized sublots for uniformity testing.

5. Sampling

5.1 After a suitable time to allow the black to ~~stabilize (30 to 45 days),~~ stabilize, a bag will be selected from the approximate middle of each of the ~~23 000-kg (50 000-lb) lots;~~ sublots; the bags selected will be numbered from one through *n*, where *n* is the total number of sublots, in order to represent the corresponding ~~23 000-kg (50 000-lb) lots.~~ production lot.

5.2 *n* 4-dm^3 (1-gal) samples, numbered from one through *n*, and taken from the corresponding bags, will be sent to each participant in the interlaboratory study to evaluate the new IRB.

5.3 Additionally, a 4-dm^3 (1-gal) sample of the *previous* IRB taken from a common blended source will also be sent to each participant.

6. Procedure

6.1 Test, in order, one of the *n* samples on each of the one to *n* days. These days shall be as near to consecutive as possible.

6.2 Each day a sample is tested, subject it to all of the test methods described in 6.3, 6.4, and 6.5.

6.3 *Physicochemical Tests*:

6.3.1 Perform the following physicochemical tests on both the new and previous IRB:

6.3.1.1 *Iodine Adsorption Number* (*Test Method D 1510*)—Report the result obtained from an individual determination in grams of iodine per kilogram to the nearest 0.1 unit.

6.3.1.2 *~~Multipoint B.E.T.~~Total and External NSA (Test Method ~~D 4820~~ D 6556)*—Report Total and External NSA from a single determination in 10^3 m^2/kg (m^2/g) to the nearest 0.1 unit.

6.3.1.3 *~~DBP~~Oil Absorption Number (Test Method D 2414)*—Report the result obtained from an individual determination in 10^{-5} m^{3}kg (cm^3/100 g) to the nearest 0.1 unit.

6.3.1.4 *~~Compressed DBP~~Oil Absorption Number of Compressed Sample (Test Method D 3493)*—Report the result obtained from an individual determination in 10^{-5} m^{3}kg (cm^3/100 g) to the nearest 0.1 unit.

6.3.1.5 *Tint Strength (Test Method D 3265)*—Report the result obtained from an individual determination in percent of ITRB to the nearest 0.1 unit.

6.3.1.6 *Pour Density (Test Method D 1513)*—Report the result obtained from an individual determination in kg/m^3 (lb/ft^3) to the nearest whole unit.

~~6.3.1.7 *Statistical Thickness Surface Area (STSA) (Test Methods D 5816)*— Report single determination in 10^3 m^2/kg (m^2/g) to the nearest 0.1 unit.~~

6.3.2 Record data on Table 1.

6.4 *Rubber Physical Tests*:

6.4.1 Perform the following physical tests in rubber on both the new and previous IRB. Test samples mixed in accordance with Test Methods D 3191 and cure for ~~35 and~~ 50 min at 145°C as well as samples mixed in accordance with Test Methods D 3192, Test Method A, and cure for 30 min at 145°C.

6.4.1.1 In accordance with Test Methods D 412, Test Method A, test five dumbbells from each cured sheet and determine the median values of tensile stress at 300 % elongation, tensile strength, and ultimate elongation.

6.4.1.2 Record data in absolute numbers (not as differences from IRB) on Table 1, reporting tensile stress and tensile strength to the nearest 0.1 MPa and ultimate elongation to the nearest 5 %.

6.5 *Informational Physicochemical Tests*:

6.5.1 Perform the following physicochemical tests on the new IRB:

6.5.1.1 *Ash Content (Test Method D 1506)*—Report results obtained from a single determination to the nearest 0.01 %.

6.5.1.2 *Fines ~~Content~~ and Attrition (Test Method D 1508)*—Report results obtained from a single determination to the nearest 0.1 %.

6.5.1.3 *Heating Loss (Test Method D 1509)*—Report results obtained from a single determination to the nearest 0.1 %.

6.5.1.4 *Sieve Residue, 325 Mesh (Test Method D 1514)*—Report results obtained from a single determination to the nearest mg/kg (ppm).

6.5.1.5 *~~Toluene Discoloration~~Transmittance of Toluene Extract (Test Method D 1618)*—Report results obtained from a single

TABLE 1 Industry Reference Black Test Data

Laboratory Number __

Day of Mixing and Date	Sample No.	Tensile Strength, MPa D 3191		Tensile Stress at 300 %, MPa D 3191	Elongation, % D 3191	Tensile Strength, MPa D 3192A		300 % Modulus, MPa D 3192A	Elongation, % D 3192A	Iodine No., g/kg D 1510	STSA 10^3 m^2/kg D 65~~81~~56	NSA Multipoint Adsorption 10^3 m^2/kg D ~~4820~~6556	~~DBP~~Oil Absorption, No., 10^{-5} mg^3/kg ($cm^3/100$ g) D 2414	Compressed ~~DBP~~ Oil No., 10^{-5} mg^3/kg ($cm^3/100$ g) D 3493	Tint Strength, D 3265	Pour Density, kg/m^3 (lb/ft^3) D 1513
Day No.__		50′				30′										
Date ____	Prev. IRB	50′				30′										
Day No.__		50′				30′										
Date ____	Prev. IRB	50′				30′										
Day No.__		50′				30′										
Date ____	Prev. IRB	50′				30′										
Day No.__		50′				30′										
Date ____	Prev. IRB	50′				30′										
Day No.__		50′				30′										
Date ____	Prev. IRB	50′				30′										
Day No.__		50′				30′										
Date ____	Prev. IRB	50′				30′										
Day No.__		50′				30′										
Date ____	Prev. IRB	50′				30′										
Day No.__		50′				30′										
Date ____	Prev. IRB	50′				30′										
Day No.__		50′				30′										
Date ____	Prev. IRB	50′				30′										
Day No.__		50′				30′										
Date ____	Prev. IRB	50′				30′										
Day No.__		50′				30′										
Date ____	Prev. IRB	50′				30′										
Day No.__		50′				30′										
Date ____	Prev. IRB	50′				30′										

determination to the nearest 0.1 % transmittance.

6.5.1.6 ~~*CTAB Surface Area*~~*Pellet Hardness* (*Test Method* ~~*D 3765*~~ *D 5230)*~~—Report a single determination in $10^3 m^2/kg$ (m^2/g) to the nearest 0.1 unit.~~

~~6.5.1.7 *Pellet Attrition (Test Method D 4324)*—Report results obtained from a single determination to the nearest 0.1 %.~~

~~6.5.1.8 *Pellet Crush Strength (Test Method D 5230)*—~~Report results obtained from a single determination to the nearest whole number for the maximum and average.

7. Statistical Analysis

7.1 For each test in Table 1, enter the results from each laboratory for each sample into the form shown in Table 2. Then

TABLE 2 Statistical Analysis Form

NOTE 1—Experience so far has shown that neither a laboratory's test values nor a sample's test values are random values about the grand average, but tend to reflect a persistent bias typified by the average value for the laboratory or the sample. Consequently, it is not appropriate to divide the reproducibility by the square root of L or N as might otherwise be the case when comparing averages of L or N values to the grand average of $L \times N$ values.

Test Method: ASTM D _ _ _								
Sample No. ↓	Laboratory No. →	1	2	...	i	...	L	$\bar{X}_R$
1								
2								
...								
j								
...								
N								
$\bar{X}_C$								$\bar{\bar{X}}=$

Row average $\bar{X}_R = \Sigma_i X/L$

Grand average test value $\bar{\bar{X}} = \Sigma_j \bar{X}_R/N$

Upper and lower control limits for row averages = $\bar{\bar{X}} \pm$ reproducibility of the test method.

Column average $\bar{X}_C = \Sigma_j X/N$

Upper and lower control limits for column averages = $\bar{\bar{X}} \pm$ reproducibility of the test method.

calculate the statistics defined in Table 2.

NOTE 1—Rubber physical test data are to be entered as differences from the previous IRB. For example:

$$Difference = X_1 - X_2 \tag{1}$$

where:

X_1 = measured value for new IRB, and
X_2 = measured value for previous IRB.

7.2 If any row average test result falls outside the interval defined by the upper and lower control limits shown in Table 2, this will indicate that the ~~23 000-kg (50 000-lb) lot~~ sublot of IRB represented by that row average may be rejected by Committee D-24 as being a nonhomogeneous portion of the production lot.

7.3 If any laboratory average test result (column average) falls outside the upper and lower control limits shown in Table 2, then that laboratory's data for that test should be deleted and Table 2 should be recalculated excluding that laboratory. Such data indicates that ~~that~~ the laboratory has a significant reproducibility problem, which needs corrective action.

7.4 After deleting data, the remaining data for each test method can be averaged to provide typical values for tabulation in Classification D 1765 and average differences between the new IRB and the previous one.

7.5 For each test in Table 3, enter the results from each laboratory for each sample in the form shown in Table 2. Then calculate the statistics defined in Table 2. The results are for information only and not to determine the uniformity of the lot.

8. Acceptance

8.1 All ~~23 000-kg (50 000-lb) lots~~ sublots tested as homogeneous by this practice will be considered acceptable by Committee D-24 for use as the new IRB. The average values will be published in Classification D 1765.

9. Keywords

9.1 blending; industry reference blacks (IRBs); lot size; physical properties; physicochemical properties; statistical analysis form; table for IRB test data; uniformity guidelines for production and testing

TABLE 3 Industry Reference Black Informational Test Data

Laboratory Number__

Day of Mixing and Date	Sample No.	Ash Content D 1506	Fines Content D 1508	Heating Loss D 1509	Sieve Residue, 325 Mesh D 1514	Transmittance of Toluene ~~Discolo~~ Extrac~~tion~~ ~~D1618~~	~~CTAB 10^3 m^2/kg~~ D~~37~~ 16~~5~~18	Pellet Attrition D~~4324~~ 1508	Average ~~C~~ Pellet Har~~u~~dnes~~h~~s ~~Strength~~ D 5230	Maximum ~~C~~Pellet Har~~u~~dne ~~Strength~~ D 5230
Day No.____ Date ____	___									
Day No.____ Date ____	___									
Day No.____ Date ____	___									
Day No.____ Date ____	___									
Day No.____ Date ____	___									
Day No.____ Date ____	___									
Day No.____ Date ____	___									
Day No.____ Date ____	___									
Day No.____ Date ____	___									
Day No.____ Date ____	___									
Day No.____ Date ____	___									
Day No.____ Date ____	___									

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