Documentation Sheet
Industry Reference Black – Lot 9
(IRB9 prepared according to ASTM D4122)
(Evaluated per ASTM D4122)
Approved by D24.61: December 4, 2018[1]
Supersedes: September 25, 2018

Introduction

The first Industry Reference Black (IRB) was produced in 1959 and approved for use at the December 1959 D24 meeting. The purpose of using the IRB is to equilibrate the testing variation caused by test conditions and material properties in rubber testing so that any change in the test result can be attributed to the grade of carbon black mixed with a rubber material. Test results are reported as the difference between a rubber property using the carbon black of interest and the same rubber property using the IRB.

Subsequent lots were numbered consecutively so this is the ninth such lot. All of the IRB lots have been an N330 grade of carbon black. The production of the IRB lots has been shared among the various carbon black producers. The quantity of the lots has varied with the consumption rate of the IRB. Because the properties of the carbon black affects the rubber test properties, it generally has been necessary for the industry to adjust specifications with the introduction of each new IRB lot. To minimize the need to change specifications, the goal has been to produce enough IRB to last eight to ten years at the expected annual consumption rate. A history of the IRB lots can be found in Annex 1 of ASTM D4122.

While IRB has been used as a reference in other tests, such as tint strength (D3265) and the now withdrawn CTAB test method, the two tests methods of primary interest are ASTM D3191 and ASTM D3192. The only reference values for IRB are for these two test methods. Any other property values, such as colloidal properties, in this document or D4122 are for information only and are not to be considered as reference values. The rubber material used in D3191 is Styrene-Butadiene Rubber (SBR) while Natural Rubber (NR) is the rubber material used in D3192. The properties of interest in both test methods are Tensile Stress at 300% Elongation (a.k.a. Modulus), Tensile Strength (Tensile), and Ultimate Elongation (Elongation). Each test method has three methods for the various equipment that can be used for mixing the rubber, carbon black, and other chemicals. Method A uses a mill mix, Method B uses an internal mixer, and Method C uses a miniature internal mixer. Due to the mixing efficiency that affects the incorporation of the carbon black and other chemicals in the rubber material and the work imparted to the rubber, the three methods give similar but not exactly the same results.

Due to the high cost of the equipment, labor, and materials needed to run the tests, few laboratories perform the D3191 or D3192 tests. Therefore, this IRB lot was tested by a limited number of laboratories that have the capability to run the D3191 and D3192 tests. The testing was performed using the current test method version as of the time of testing.

[1] The current version of this document is available from Laboratory Standards and Technologies, Inc., 227 Somerset Street, Borger, TX, 79007, www.carbonstandard.com.

D24 attempts to produce enough of each IRB lot so that it will last about 8 to 10 years at the current rate of consumption. One reason for doing this is because of the high cost and time it takes to produce, package, and validate a new IRB lot. A second reason involves the properties of the IRB carbon black of any specific lot. While every IRB lot is a N330 type carbon black and is produced to the same target parameters of iodine number and OAN, the typical properties can vary due to factors such as equipment design and feedstock oil properties. These different typical properties can result in changes in the in-rubber performance of the IRB. Because of these changes in rubber performance, it was usually necessary for the industry to issue new specifications for rubber properties every time a new IRB lot was introduced up through IRB7. The difference between IRB7 and IRB8 was less than the testing error so they were statistically equivalent and it was not necessary for the industry to issue new specifications. The difference between IRB8 and IRB9 is also less than the testing error so they too are statistically equivalent and it will not be necessary for the industry to issue new specifications. The history of the various IRB lots can be found in ASTM D4122 Annex Table A1.1.

Properties of the IRB9 Lot

Table 1 shows the test values for various carbon black tests performed on the IRB9 lot. These values are given as information only and are not reference values.

Tables 2 and 3 show the "IRB9 difference from IRB8" values for D3191 and D3192 in-rubber testing, respectively, for various rubber properties.

Special consideration for bias: When no absolute reference standard exists, such as is the case with in-rubber testing of carbon black, a laboratory's bias can be defined as the difference between its results and the mean result from an ITP involving many laboratories. Every laboratory can be expected to have some level of bias due to the unique combination of testing conditions (equipment, materials, manpower, environment, etc.) that exists within a given laboratory. The level of bias for a given laboratory may or may not be significant. Unfortunately, there were not enough laboratories participating in the evaluation of the IRB9 lot's in-rubber properties (See Table 4) to allow for any evaluation of laboratory bias.

TABLE 1 IRB9 Properties

Property	IRB9
Tint Strength, D3265, % ITRB	105.9
Iodine Absorption No., D1510, g/kg (mg/g)	82.1
NSA, D6556, 103 m2/kg (m2/g)	78.1
STSA, D6556, 103 m2/kg (m2/g)	77.1
OAN, D2414, 10-5 m3/kg	98.9
COAN, D3493, 10-5 m3/kg	90.1
Pour Density, D1513, kg/m3 (lb/ft3)	400 (25.0) [A]
Fines Content, D1508, %	4.4 [A]
Heating Loss, D1509, %	0.5[A]
Sieve Residue, D1514, mg/kg (ppm)	73
Transmittance of Toluene Extract, D1618, %	95
Mean Pellet Hardness, D5230, cN (gf)	41.7 (42.6)
Maximum Pellet Hardness, D5230, cN (gf)	77.5 (79.1)

[A]Test results from producer

Table 2 D3191 IRB9 Difference from IRB8

	Method A	Method B	Method C
Modulus, Mpa (psi)	-0.18 (-26)	0.07 (10)	0.18 (26)
Tensile, Mpa (psi)	-0.35 (-51)	-0.33 (-48)	0.35 (51)
Elongation, %	-3.56	-4.88	0.52

Table 3 D3192 IRB9 Difference from IRB8

	Method A	Method B	Method C
Modulus, Mpa (psi)	-0.28 (-41)	-0.08 (-12)	-0.35 (-51)
Tensile, Mpa (psi)	-0.54 (-78)	-0.03 (-4)	-0.42 (-61)
Elongation, %	-0.53	3.30	0.83

Table 4 Number of Laboratories for D3191 and D3192

	Method A	Method B	Method C
D3191	2	3	3
D3192	2	4	2

Background and Interlaboratory Test Program Details:
IRB9 Lot

Background - Industry Reference Black (IRB), used for a number of test methods under the jurisdiction of ASTM Committee D24, is prepared according to D4122, "Evaluation of an Industry Reference Black" with evaluation, statistical analysis, and acceptance as described in that document.

Evaluation of the IRB9 Lot – The production of the various IRB lots is rotated among the various carbon black producers. See D4122 Annex Table A1.1 for a list of the producers of the various IRB lots. Each lot is evaluated for uniformity by the producer. That uniformity data is reviewed with the chairman of subcommittee D24.61 and the final material selected to give the best uniformity possible for the rubber tests of interest.

Most of the values listed in Table 1 were obtained through D24's LPRS program with the testing being performed in March 2016 involving more than seventy laboratories. In this program a single blind sample is distributed to the participating laboratories. Two samples of different materials are tested each year about six months apart. (Materials other than those in the IRB lots are also tested in the LPRS program.) Each laboratory selects two technicians to perform the testing (the same two technicians may not have performed all the tests, depending on how the laboratory is staffed and organized) and each technician performs the testing once on two different days for a total of four test results. A few of the tests listed in Table 1 were not part of the LPRS program at the time of the testing of the IRB9 lot. The value for these tests were obtained from the producer of the material, as noted. All the values in Table 1 are for information only and are not reference values.

In addition to the test results, the LPRS program also collects information on the test conditions when the testing was performed for each sample. A questionnaire is distributed along with the data form asking about the equipment, materials, methods, and testing conditions used when testing each sample. This information is used to help identify sources of variation and bias between laboratories to help improve testing proficiency in the industry. Recently, a testing instruction sheet that specifies the testing conditions to be used for a given sample has been included with the data file because it has been determined that some laboratories were not using the correct conditions when performing the LPRS testing. With this knowledge, D24 logically concluded that only data from those laboratories reporting that they performed the testing per the conditions specified in each test method as included in the testing conditions instructions would be used to determine the mean values as shown in Tables 2 and 3.

Interlaboratory Test Program (ITP) – The data was analyzed per D4122. See Tables 2 and 3 for the "difference from IRB" values for the D3191 and D3192 test methods and test properties, respectively. See Table 4 for a list of how many laboratories participated in the testing for each test method.

Using the IRB9 Lot – Since the purpose of an IRB is the elimination of the major part of laboratory-to-laboratory variation in rubber testing, it is strongly recommended that it be used as a reference material within each laboratory to correct actual measured rubber property values in that laboratory's testing. As a minimum, an IRB mix should be included every day that rubber

testing is performed. If more than one cure will be performed during the day, it is recommended that an IRB mix should be included in each cure to help reduce the variation introduced by changes in the mixing, curing time, curing pressure, and curing temperature uniformity, etc.

To report corrections or request changes to this document, contact Laboratory Standards and Technologies or the chairman of ASTM subcommittee D24.61.