Documentation Sheet

Standard Reference Black – Set 8 (SRB8 prepared according to ASTM D6915) (Evaluated per ASTM D4483-99) Approved by D24.61: April 29, 2020¹

Supersedes: June 13, 2017

Introduction

The first Standard Reference Blacks (SRB) were produced in the early 1960s for the purpose of normalizing the test results from the recently developed oil absorptometer (See ASTM D2414). This initial set consisted of four grades of carbon black.

Subsequent sets were numbered consecutively so this is the eighth such set. Besides the oil absorption test, these later sets have been used to normalize the test results of other test methods. The number of materials and the grades have evolved as needed to cover the range of test results for all the tests for which the SRBs are used

The materials selected for a given SRB set are intended to span as much as possible the range of test results of the selected tests. The SRB8 set consists of seven grades of carbon black; three tread, three carcass, and one thermal. They are available for use to normalize the test results from the following carbon black ASTM test methods: D2414 – Oil Absorption Number; D3265 – Tint Strength; D3493 – Oil Absorption Number of Compressed Sample; D6556 – Total and External Surface Area by Nitrogen Adsorption and D7854 – Void Volume at Mean Pressure. Values have also been developed for D1510 – Iodine Number test method but these values are NOT to be used to normalize the Iodine Number results. A special set of materials (HT or INR) have been developed for this purpose and only those materials should be used to normalize the Iodine Number test results. Information on the HT and INR reference materials is available from Balentine Enterprises (See footnote 1 for contact information). When performing normalization of test results using the SRB materials, it is necessary to use at least three (3) of the appropriate materials from the same SRB set. For example, when normalizing OAN test results for a tread grade of carbon black, use the A, B, and C materials from the same SRB set.

This SRB set was initially tested by a limited number of laboratories for the properties and test methods as listed above using the current test method version at the time of testing. The test results were normalized to the previous SRB set, thus maintaining backwards compatibility for the current set. Subsequent to the initial testing, each of the materials has been tested in the Carbon Black (CB) Laboratory Proficiency Rating System (LPRS) program operated by OkStats, Inc. for the benefit of D24. The CB LPRS program includes a large number of laboratories (typically 60 or more) that represent the testing proficiency of the industry. The initial values and limits have been adjusted, as needed, to the values obtained from the LPRS program. The values and limits listed below are the most current as of the date of this publication. See page 6 of this Documentation Sheet for additional background on this SRB set.

¹ The current version of this document is available from Balentine Enterprises, Inc., 227 Somerset Street, Borger, TX, 79007, www.carbonstandard.com.

While D24 attempts to produce enough of each SRB material so that they will all be depleted at about the same time and a new SRB set produced, variability in the consumption rate of each material has thwarted this goal. Rather than waste the value of the remaining materials when the first material is depleted, D24 has adopted a program of producing supplemental lots of any depleted material. The quantity of the supplemental lots is set to be depleted when the last material is depleted, thus extending the life of the set. Every effort is made to have the supplemental lots produced by the same producer location that produced the initial lot. (See ASTM D7849 for information on the nomenclature used for D24 reference materials that identifies these supplemental lots.)

Shelf Life

Per ASTM D6915, Standard Practice for Carbon Black—Evaluation of Standard Reference Blacks, the shelf life of the Standard Reference Black (SRB) carbon blacks is indefinite when properly stored in a manner that protects it from exposure to sources of moisture, such as precipitation, other sources of liquid water, or high humidity environments. (See ASTM D8043, Standard Guide for Carbon Black—Shelf Life for similar information on other carbon blacks.)

Properties for the SRB8 Set

The testing conducted as part of the SRB8 set development evaluation using ASTM D4483-99, generated accepted reference values (means), AR-values, for the given tests as defined below and 2 and 3 standard deviation limits on these values or on individual daily values as obtained by any laboratory using the SRB8 set. The 2 and 3 standard deviation limits apply to a single measurement of the listed test properties. Two times the 2 or 3 standard deviation limit equals the total 4 or 6 standard deviation range, respectively.

ASTM D4483-99 uses a one-sided k test to identify outliers having high variability. The CB LPRS program uses a two-sided k test to identify outliers having high and low variability. This approach is thought to better represent expected variability in real-world testing and helps to offset memory-bias from an individual's repeated testing of the same material(s).

- 'Accepted Reference Value' or AR-value; this is the average (mean), for the various tests and materials listed below in Tables 1 through 7, obtained in an interlaboratory test program (ITP), i.e., the LPRS program, for a large group of typical laboratories using samples taken from the various material lots. See page 7 for more details on the ITPs.
- 'Within Typical Laboratory' 2 and 3 standard deviation value; this is the within laboratory \pm 2 and \pm 3 standard deviation (Sr) value (for single measurements) on the SRB8 set AR-values for the various tests, as obtained from the same group of typical ITP laboratories.
- 'Between Typical Laboratory' 2 and 3 standard deviation value); this is the between laboratory ± 2 and ± 3 standard deviation (SR) value (for single measurements) on the SRB8 set AR-values for the various tests, as obtained from the same group of typical ITP laboratories.
- **2 standard deviation versus 3 standard deviation use considerations:** Most carbon black test properties (with the exception of pellet hardness maximum) have an acceptable approximation to a normal distribution. With a normal distribution, 95.5% of all the test values are expected to fall

within the limits of mean \pm 2 standard deviation and 99.7% will fall within the limits of mean \pm 3 standard deviation. This means that with only random variation present, approximately 1 in 20 results will fall outside the 2 standard deviation limits and 3 in 1000 will fall outside the 3 standard deviation limits. This means that when using 2 standard deviation limits the laboratory will be looking for a problem 1 in 20 test results when there is no problem to be found. This is a waste of valuable resources. On the other hand, when using 3 standard deviation limits the laboratory will be looking for a problem when there is not a problem only 3 in 1000 test results. However, if the consequences of allowing a problem to go undetected for a long time are too high, using 3 standard deviation limits may not give adequate warning in sufficient time to implement timely corrective action. Using 2 standard deviation limits will give an earlier warning of the presence of a problem. It is up to the user to balance the costs of untimely warnings versus the costs of searching for problems that do not exist.

Special consideration for bias: When no absolute reference standard exists, such as is the case with carbon black testing, a laboratory's bias can be defined as the difference between its results and the mean result from an ITP involving many laboratories, such as the CB LPRS program. 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. A laboratory that did not participate in the ITP may find that it cannot maintain control within the control limits due to factors unique to that laboratory causing bias in its values, increased variation, or both. The laboratory should conduct an investigation to identify the presence and cause(s) of the bias and variation and eliminate them so that it is aligned with the ITP data. Participation in a multi-laboratory precision study, such as the CB LPRS program, may help to identify the unique sources of bias and variation. The SRB8 set can be used to assist a laboratory in determining the presence and magnitude of bias and variation using the values given in the Tables 1 to 7 below. See ASTM D4821 for specific instructions on how to use these values to evaluate a laboratory's testing proficiency (bias and variability).

Table 1 Mean (AR-value) and Limit Values for D1510, Iodine Number Methods A & B										
Units	g/kg	Wit	hin Labora	tory	Betwo	Between Laboratories				
Material	Mean (AR-value)	Sr	2 x Sr	3 x Sr	SR	2 x SR	3 x SR			
SRB-8B2 (N134)	146.3	0.57	1.14	1.70	1.70	3.40	5.09			
SRB-8C (HS Tread)	138.8	0.68	1.36	2.04	2.11	4.21	6.32			
SRB-8B (N134)	135.6	0.68	1.35	2.03	1.93	3.86	5.80			
SRB-8A (N326)	80.5	0.36	0.73	1.09	0.88	1.76	2.64			
SRB-8A2 (N326)	78.1	0.88	1.76	2.64	1.33	2.67	4.00			
SRB-8F2 (N683)	36.1	0.41	0.81	1.22	0.65	1.29	1.94			
SRB-8F (N683)	35.9	0.32	0.63	0.95	0.57	1.14	1.70			
SRB-8E (N660)	35.8	0.32	0.64	0.97	0.60	1.21	1.81			
SRB-8D (LS Carcass)	21.7	0.28	0.57	0.85	0.55	1.09	1.64			

Table 2 Mean (Al	R-value) and Limit V	alues for						
Units	$10^3 \text{ m}^2/\text{kg} (\text{m}^2/\text{g})$	Wit	hin Labora	tory	Between Laboratories			
Material	Mean (AR-value)	Sr	2 x Sr	3 x Sr	SR	2 x SR	3 x SR	
SRB-8B (N134)	142.0	0.47	0.95	1.42	1.44	2.87	4.31	
SRB-8B2 (N134)	138.0	0.31	0.62	0.93	0.79	1.58	2.37	
SRB-8C (HS Tread)	126.4	0.44	0.88	1.32	1.07	2.13	3.20	
SRB-8A (N326)	76.5	0.33	0.67	1.00	0.84	1.68	2.53	
SRB-8A2 (N326)	75.9	0.29	0.57	0.86	0.70	1.40	2.10	
SRB-8E (N660)	36.7	0.23	0.46	0.69	0.53	1.05	1.58	
SRB-8F (N683)	36.7	0.21	0.41	0.62	0.38	0.77	1.15	
SRB-8F2 (N683)	32.4	0.19	0.38	0.57	0.55	1.11	1.66	
SRB-8D (LS Carcass)	21.6	0.18	0.36	0.55	0.30	0.60	0.90	
SRB-8G (N990)	9.1	0.12	0.24	0.36	N/A	N/A	N/A	

Table 3 Mean (AF	R-value) and Limit V	alues for	D6556, ST	ΓSA					
Units	$10^3 \text{ m}^2/\text{kg} (\text{m}^2/\text{g})$	Wit	hin Labora	tory	Betwe	Between Laboratories			
Material	Mean (AR-value)	Sr	2 x Sr	3 x Sr	SR	2 x SR	3 x SR		
SRB-8B (N134)	133.1	0.71	1.41	2.12	1.39	2.77	4.16		
SRB-8B2 (N134)	126.7	0.56	1.11	1.67	2.02	4.05	6.07		
SRB-8C (HS Tread)	115.8	0.48	0.95	1.43	1.06	2.12	3.19		
SRB-8A (N326)	77.2	0.41	0.83	1.24	1.15	2.30	3.45		
SRB-8A2 (N326)	76.0	0.47	0.94	1.40	1.23	2.46	3.70		
SRB-8E (N660)	35.8	0.34	0.68	1.02	0.71	1.43	2.14		
SRB-8F (N683)	35.4	0.33	0.65	0.98	0.69	1.38	2.06		
SRB-8F2 (N683)	32.1	0.26	0.52	0.78	0.65	1.30	1.96		
SRB-8D (LS Carcass)	21.2	0.26	0.52	0.78	0.54	1.07	1.61		
SRB-8G (N990)	8.4	0.20	0.40	0.60	N/A	N/A	N/A		

Table 4 Mean (AF	R-value) and Limit Va	alues for l	D2414, O	AN				
Units	$10^{-5} \mathrm{m}^3/\mathrm{kg} (\mathrm{cm}^3/100 \mathrm{g})$	Wit	hin Labora	tory	Between Laboratories			
Material	Mean (AR-value)	Sr	2 x Sr	3 x Sr	SR	2 x SR	3 x SR	
SRB-8C (HS Tread)	174.9	0.50	1.00	1.49	1.08	2.15	3.23	
SRB-8B2 (N134)	125.2	0.42	0.84	1.26	0.97	1.94	2.90	
SRB-8B (N134)	123.5	0.45	0.89	1.34	0.91	1.81	2.72	
SRB-8A2 (N326)	71.5	0.46	0.93	1.39	1.56	3.12	4.68	
SRB-8A (N326)	70.9	0.46	0.92	1.38	0.93	1.86	2.79	
SRB-8F2 (N683)	134.5	0.54	1.08	1.62	1.65	3.30	4.95	
SRB-8F (N683)	132.0	0.41	0.82	1.23	0.91	1.83	2.74	
SRB-8E (N660)	87.8	0.36	0.72	1.08	1.30	2.60	3.90	
SRB-8D (LS Carcass)	36.9	0.26	0.52	0.78	1.09	2.18	3.28	
SRB-8G (N990)	36.2	0.25	0.50	0.75	N/A	N/A	N/A	

Table 5 Mean (AF	R-value) and Limit Va	alues for l	D3493, C0	OAN				
Units	$10^{-5} \mathrm{m}^3/\mathrm{kg} (\mathrm{cm}^3/100 \mathrm{g})$	Wit	hin Labora	in Laboratory Between Laboratories				
Material	Mean (AR-value)	Sr	2 x Sr	3 x Sr	SR	2 x SR	3 x SR	
SRB-8C (HS Tread)	130.6	0.54	1.07	1.61	1.47	2.94	4.42	
SRB-8B2 (N134)	103.1	0.50	1.00	1.51	1.03	2.06	3.09	
SRB-8B (N134)	99.4	0.47	0.93	1.40	1.03	2.06	3.09	
SRB-8A2 (N326)	67.5	0.35	0.69	1.04	1.08	2.16	3.24	
SRB-8A (N326)	66.7	0.42	0.85	1.27	0.87	1.74	2.61	
SRB-8F (N683)	88.6	0.40	0.79	1.19	0.91	1.82	2.73	
SRB-8F2 (N683)	85.5	0.35	0.70	1.05	1.17	2.35	3.52	
SRB-8E (N660)	74.7	0.36	0.71	1.07	0.99	1.99	2.98	
SRB-8D (LS Carcass)	36.9	0.26	0.52	0.78	0.96	1.92	2.89	

Table 6 Mean (AR-value) and Limit Values for D3265, Tint Strength ¹ , ZnO Lot 11, Greenflex 7170, ITRB2										
Units	Tint Strength		hin Labora							
Material	Mean (AR-value)	Sr	2 x Sr	3 x Sr	SR	2 x SR	3 x SR			
SRB-8B (N134)	131.7	1.19	2.38	3.57	1.78	3.56	5.34			
SRB-8B2 (N134)	130.7	1.19	2.38	3.57	1.47	2.94	4.41			
SRB-8C (HS Tread)	110.2	0.71	1.42	2.13	1.22	2.44	3.66			
SRB-8A2 (N326)	109.7	0.75	1.50	2.25	1.18	2.36	3.54			
SRB-8A (N326)	109.4	0.76	1.52	2.28	1.10	2.20	3.30			
SRB-8E (N660)	60.0	0.53	1.06	1.59	0.70	1.40	2.10			
SRB-8F (N683)	51.1	0.37	0.74	1.11	0.60	1.20	1.80			
SRB-8F2 (N683)	46.6	0.39	0.78	1.17	0.66	1.32	1.98			
SRB-8D (LS Carcass)	41.6	0.42	0.84	1.26	0.62	1.24	1.86			

¹These values were determined using zinc oxide lot #11, Greenflex 7170 ESO, and ITRB2. Testing using other materials will give different values.

Table 7	Mean	(AR	-value)	and Limit	Values for	r D7854 <u>,</u>	Void V	Volume	at 50 MPa	Mean P	ressure

Units	$10^{-5} \text{ m}^3/\text{kg} (\text{cm}^3/100 \text{ g})$	Wit	hin Labora	tory	Betw	Between Laboratories		
Material	Mean (AR-value)	Sr	2 x Sr	3 x Sr	SR	2 x SR	3 x SR	
SRB-8C (HS Tread)	72.8	0.27	0.54	0.81	0.26	0.52	0.78	
SRB-8B (N134)	69.7	0.26	0.52	0.78	0.32	0.64	0.96	
SRB-8B2 (N134)	67.6	0.26	0.52	0.78	0.35	0.70	1.05	
SRB-8A2 (N326)	46.6	0.15	0.30	0.45	0.17	0.34	0.51	
SRB-8A (N326)	45.5	0.15	0.30	0.45	0.25	0.50	0.75	
SRB-8F (N683)	60.3	0.21	0.42	0.63	0.33	0.66	0.99	
SRB-8F2 (N683)	54.5	0.16	0.32	0.48	0.23	0.46	0.69	
SRB-8E (N660)	49.4	0.16	0.32	0.48	0.22	0.44	0.66	
SRB-8D (LS Carcass)	23.9	0.13	0.26	0.39	0.17	0.34	0.51	

Background and Interlaboratory Test Program Details: SRB8 Set

<u>Background</u> - Standard Reference Blacks (SRBs), used for a number of test methods under the jurisdiction of ASTM Committee D24, are prepared according to D6915, "Evaluation of Standard Reference Blacks" with statistical analysis per D4483-99, "Standard Practice for Evaluating Precision for Test Method Standards in the Rubber and Carbon Black Manufacturing Industries". The objective of D24 is to select reference materials that will have values across the range of possible test results for any given test. Even better would be to select materials whose values are evenly spaced across the test range. Unfortunately, due to the realities of carbon black production and the interdependence of some properties, it may not be possible to select materials that cover the test range for all tests, much less be evenly spaced across that range. The materials selected for the SRB8 set represents D24's best attempt to satisfy these objectives.

<u>Evaluation of the SRB8 Set</u> – The production of the various SRB8 materials is shared among the various carbon black producers. See Table 8 for a list of the producers of the various SRB8 materials. Each lot is evaluated for uniformity by the producer. That uniformity data is reviewed with the chair of subcommittee D24.61 and the final material selected to give the best uniformity possible for all the tests of interest as listed in Tables 1 to 7.

The values listed in Tables 1 to 5 and 7 were obtained through the CB LPRS program. 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 SRB8 set are also tested in the CB LPRS program.) The tint values in Table 6 were obtained in a special ITP conducted in February/March 2016 to determine the SRB8 tint values using the zinc oxide lot #11, Greenflex 7170 ESO, and ITRB2 materials. For the CB LPRS and the special tint ITP, 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.

In addition to the test results, the CB LPRS program also collects information on the test conditions existing at the time 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 CB LPRS testing. With this knowledge, D24 can be in compliance with ASTM's requirement 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 w\should be used to determine the mean (AR-value) and limit values for a given material. That is the reason for the rather substantial drop from previous test rounds in the number of laboratories (See Table 8) whose data was used for the calculations of the SRB-8B2 material because only data from those laboratories that reported they followed the test condition instructions was used. (This knowledge is not available for materials tested before 2014.) Future CB LPRS testing and the use of the knowledge of whether

a laboratory's testing conforms per the ASTM Test Method requirements may result in refinement of some of the material values.

<u>Interlaboratory Test Program (ITP)</u> – See Table 8 for a list of when the various SRB8 materials were tested. Table 8 also shows how many laboratories participated in the testing for each test and how many were removed as outliers for mean (M), high variation (H), or low variation (L). The data was analyzed per D4483-99.

Table 8 SRB	8 Informa	tion								
]	Number of La	boratories	(M/H/L)		
SRB8 Material	Grade	Producer	Test Period	D1510	D6556 NSA	D6556 STSA	D2414	D3493	D3265 ¹	D7854 ²
SRB-8A	N326	Continental	March 2008	61(0/2/1)	58(0/1/0)	54(1/2/0)	64(1/0/0)	59(1/1/0)	27(1/1/3)	12
SRB-8A2	N326	Continental	March 2013	71(1/1/2)	67(1/2/0)	63(1/2/0)	72(0/1/1)	65(0/1/0)	27(0/0/0)	11
SRB-8B	N134	Cabot	June 2009	66(1/2/2)	65(0/3/1)	62(2/2/2)	66(0/0/0)	61(0/0/0)	27(0/1/1)	11
SRB-8B2	N134	Cabot	March/April 2014	39(3/3/4)	11(1/1/0)	15(1/1/0)	40(3/4/3)	39(2/2/2)	27(1/1/1)	11
SRB-8C	HS Tread	Columbian	September 2010	64(1/2/5)	63(1/2/0)	58(2/3/0)	66(2/1/0)	63(0/1/0)	27(0/1/0)	9
SRB-8D	LS Carcass	Cabot	March 2009	67(1/2/2)	64(1/2/2)	60(1/2/0)	67(0/2/0)	60(1/2/0)	27(0/0/0)	12
SRB-8E	N660	Orion	September 2008	57(0/3/0)	54(1/1/1)	51(1/1/0)	57(1/0/0)	51(0/1/1)	27(0/0/0)	11
SRB-8F	N683	Orion	March 2010	68(1/1/0)	67(2/2/0)	64(1/1/0)	67(1/1/0)	63(1/0/0)	27(0/1/1)	12
SRB-8F2	N683	Orion	March 2015	60(1/2/2)	30(0/1/0)	44(2/1/0)	62(1/0/0)	55(1/1/0)	27(0/1/0)	11
SRB-8G ³	N990	Cancarb	Last half of 1996	N/A	Unknown	Unknown	Unknown	N/A	N/A	N/A
1The ITP to det	ermine D32	65 values was	s conducted in Febru	ary/March	2016.					
² The ITP to dete	ermine D785	54 values was	conducted in 2017.							
³ SRB-8G was p	roduced and	approved in t	the last half of 1996	as SRB-50	and has conti	inued to be inc	luded in the	current SR	B sets	
since that time.	At the time	it was produc	ed and approved it v	vas D24's p	ractice to only	publish the wi	ithin-labora	itory standa	rd	
deviation, Sr, ar	nd associated	l limits. The b	etween-laboratory	standard de	viation, SR, wa	as never publisl	hed and sin	ce the data i	is no	
longer available	it is not pos	sible to calc	ulate or publish the	SR values a	nd correspond	ling limits. The	SRB G ma	terial was o	nly	
tested for NSA	STSA, and C	DAN per the t	est method version	available in	1996.					

<u>Using the SRB8 Set</u> – For the test methods listed in Tables 1 to 7 it is strongly recommended that laboratories determine if they are operating in an "in control" manner, by the use of the \pm 2 or \pm 3 standard deviation within-laboratory limits as the laboratory may choose to use. Despite rigorous analysis of the ITP data for the AR-value(s) and associated standard deviation(s), the group of laboratories in this (and any) ITP do not represent a typical "in statistical control system" to which the usual 6 standard deviation limits are applied. All the assignable causes of variation that are typically eliminated to attain 'statistical control' have not and cannot be, eliminated for the AR testing.

When performing normalization of test results using the SRB8 materials, it is necessary to use at least three (3) of the appropriate materials from the SRB8 set. For example, when normalizing OAN test results for a tread grade of carbon black, use the 8A (or 8A2), 8B (or 8B2), and 8C materials.

See ASTM D4821 for detailed instructions on using the SRB8 set values to monitor a given laboratory's testing proficiency (bias and variability) for the test methods listed in Tables 1 to 7. D4821 also includes instructions on evaluating testing proficiency (bias and variability) between laboratories.

To report corrections or request changes to this document, contact Balentine Enterprises or the chair of ASTM subcommittee D24.61.