D1510
Iodine Adsorption Number

It was noted that in the preparation of iodine solution (0.4728N), iodine solution (0.0473N), potassium iodide (KI) and potassium iodate/iodide solution (0.0394N), all need to add KI. However, KI is only required to be dried in the preparation of potassium iodate/iodide solution (0.0394N). The purpose of drying KI is to remove the water and iodine derived from the oxidation of KI. So in order to improve the concentration dried KI should be used in all solutions. The procedure will be changed to using dried KI in all solutions requiring KI and then balloted.

Columbian (G. Joyce) presented comparison data between the Brinkman automated system and the Mettler auto-titrators. The conclusion was that the Brinkman system gave statistically equivalent results using normalized values and that the use of actual calculated normality versus 0.0473 makes only slight improvement in the results.

Cabot (E. Khmelonitsjaia) presented data showing pellet size makes a difference when comparing automated versus manual methods of D1510. Difference are apparently due to the shaking versu the stirring of samples.

D6556
Total Surface Area by Nitrogen Adsorption

Columbian (G. Joyce) presented data on SRB A-8 (N326) which showed STSA values and STSA variances are affected by differences in sample lots. The conclusions were that the published A-8 STSA mean is incorrect and that the tolerances for SRB 8 NSA/STSA are too tight. This will be further discussed within the NSA/STSA Task Group and D24.61.

D24.31 Non-Carbon Black Components of Carbon Black
Chairman: Jeffery A. Melsom
Michelin Americas Research & Development Co.
(864) 422-4079
jeff.melsom@us.michelin.com

D1514
Sieve Residue

A task group is reviewing this standard and will make suggestions on ways to improve the procedure to reduce the repeatability and reproducibility.

D24.41 Carbon Black Nomenclature and Terminology
Chairman: Ricky Magee
Columbian Chemicals Co.
(770) 792-9472
rmagee@columbianchemicals.com

D3053
Standard Terminology Relating to Carbon Black

Definition for Void Volume will be updated to align with D6086 Carbon Black – Void Volume.

D24.45 USA Committee to ISO
Chairman: John A. Bailey, Jr.
Continential Carbon Co.
(281) 647-3851
john@okstats.com

ISO/TC45/SC3/WG3 met in Jeju, Republic of Korea during the week of October 26th.

Details and results from the meeting will be presented at the December ASTM D24.45 meeting in Miami, FL.

D24.51 Carbon Black Pellet Properties
Chairman: Pierre Hirtt
Hitec Luxemburg
(352) 49-847-8740
pierre.hirtt@hitec.lu

D5230
Carbon Black - Automated Individual Pellet Crush Strength

The question was raised if users were actually selecting the "most spherical pellets" before testing as stated in sections 3.1, 8.2, 8.3 and 8.3.1. The general response was that most labs disregard this step and use any and all that pellets that are on the No. 14 sieve. Only one company stated they did select the "most spherical pellets" for testing but internal studies did not show an advantage to selecting spherical pellets. The standard will be ballot for the removal of all references to "most spherical pellets".

D24.61 Carbon Black Sampling and Statistical Analysis
Chairman: John A. Bailey, Jr.
Continental Carbon Company
(281) 647-3851
john@okstats.com

Laboratory Proficiency Rating System (LPRS)

The latest LPRS data was distributed to all participating labs. In this report an attempt was made to show the effect of the various equipment, materials, and methods. Several interesting trends were noted but additional data is needed to reach any firm conclusions. The committee agreed that for the remaining SRB 8s this information is necessary to establish how the uniformity can be affected by the equipment, materials, and methods.

The next LPRS will be accomplished using SRB 8E. Equipment data will also be collected as well as 5' fines and attrition. In conjunction with this next LPRS the data form will be revised to remove ambiguities with respect to equipment identification.

Standards Inventory

SRB 7 materials are now fully depleted and no longer available. IRB 7 has approximately 18 – 24 months of inventory remaining. Continental Carbon has agreed to manufacture the next IRB (IRB 8). The SRB HT standards have sufficient material to last another six years. At that time a second lot of material will be validated (Laboratory Standards has already purchased 1000 lbs of each of the HT materials and will hold the material until the current SRB HT is almost depleted).

ASTM D24 Carbon Black Standards are available from:

Ballentine Enterprises, Inc.
227 Somerset Street
Borger, Texas 79007
(800) 742-7671 US Only
(806) 273-3006 phone/fax
email: jwbalen@arn.net
Web address: www.carbonstandard.com

D24.71 Carbon Black Rubber Testing
Chairman: Charles D. Leonard
Lion Copolymer
(225) 267-3575
charlie.leonard@lioncopolymer.com

Entech Polymer Consultants (R. S. Ramkumar) presented their work on nano particle carbon blacks. They are working with a local Indian university, Hi-Tec Carbons, and a tire company. At the heart of the study is to conserve oil by developing compounds that have a longer life than current formulas.

The study is focused on nano particles of carbon black and zinc oxide using ASTM formulations and test methods (D1646, D6204, D2240, and D412). Initially a sample of zinc oxide and carbon black was taken and nano particles were made by reducing the size of the particles to 3 - 7 microns using a technique develoed by IIT, Chennai. The experiments are expected to be continued as part of the research to determine optimum results for the various level of micro sized nano carbon black added to ASTM formulations. Additional information will be presented at future ASTM D24 meetings.

D24.81 Carbon Black Microscopy and Morphology
Chairman: Bonnie Mcwade-Furtado
Cabot Corporation
(978) 670-7069
bonnie_mcwade@cabot-corp.com

D3849-02
Primary Aggregate Dimensions from Electron Microscope Image Analysis

A task group is reviewing whether a manual method for determining particle size would be an acceptable means of measurement.

THE NEWSLETTER OF ASTM INTERNATIONAL COMMITTEE D24 ON CARBON BLACK

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the Carbon Aggregate

ASTM INTERNATIONAL
Standards Worldwide

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Aligning Carbon Black Quality between Carbon Black Manufacturers and their Customers

BY JEFFERY A. MELSOM
(Michelin Americas Research & Development Co.)

It is important when conducting business between a manufacturer (supplier) and their customers that the level of quality be well defined before the start of any business transactions. Aside from the normal business issues (price, packaging, delivery terms, payment, etc.) the level of quality must be properly and thoroughly addressed. If the level of quality (properties) and how that quality is measured is not thoroughly discussed and defined then the basic foundation for conducting open and fair business will be hampered. In the area of carbon black it is important that the manufacturer be able to consistently produce carbon blacks that meet defined quality standards (identifying the critical properties and assigning target values and ranges) and that they be controlled and measured. It is also just as important for the customer to be able to validate the quality of any carbon black received based on the agreed upon level of quality. It is this alignment between a manufacturer and their customers that must be established and continually maintained.

For any alignment program between a supplier and customer, where quality is at the forefront of the business, to be successful it must have four main ingredients: consensus on colloidal and physical properties (defined specifications); acceptable methods of analysis to measure those properties; standards to validate the methods of analysis; and a laboratory proficiency program to guarantee labs maintain their testing proficiency.

Consensus on Colloidal and Physical Properties

Consensus on colloidal and physical properties starts with the manufacturer's definition of the carbon black. This will normally be in the form of a technical bulletin or specification sheet. In some instances typical properties for commodity blacks have been defined in international standards. For instance ASTM International's *D1765 – Carbon Black Used in Rubber Products* defines the target and typical values for 43 rubber grade carbon blacks. Within this standard target properties (Iodine Adsorption Number and Oil Absorption Number) and typical properties (Oil Absorption Number of Compressed Sample, NSA Multipoint, STSA, Tint Strength, and Pour Density) have been defined and accepted in the global carbon black industry. Regardless of where the specifications originate they must be accepted by both the manufacturer and customer. If they are not then it becomes difficult to establish a strong business relationship.

Acceptable Methods of Analysis

Once the colloidal and physical properties have been defined and agreed upon it becomes necessary to guarantee those properties. Properties are guaranteed though acceptable analytical methods. In the area of carbon blacks most methods have been jointly developed by carbon black manufacturers, their customers, instrument manufactures, and other individuals

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or organizations having an interest in carbon black. These were primarily developed thorough standard institutions for the betterment of the industry. Most of the current carbon black methods have been developed via consensus between all interested companies and organizations working through ASTM International's technical committee D24 on Carbon Black. Through the work of D24 most of the carbon black methods have been accepted internationally and have become the basis for other countries' national standards or actually used as the reference in national and international standards.

Regardless of where the standard originated, globally speaking, the carbon black industry has a set of well established and internationally accepted methods of analysis. To date there are over 30 methods available to characterize both the colloidal properties and physical properties (in rubber compounds) of carbon black. Since these are readily available through ASTM International, ISO, AFNOR, BSI, GB, DIN, JSA, and etc. it is not necessary to try and define acceptable methods because they already exist, internationally accepted, and currently in use throughout the world.

The Carbon Aggregate
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The Carbon Aggregate is published semi-annually by ASTM to provideinformation about Committee D24 to the carbon black community.

Standards to Validate the Methods of Measurement[1]

Once the physical characteristics have been established and methods have been defined and accepted then it becomes necessary to continually validate a method through the use of reference materials or standards. ASTM International has developed a number of Standard Reference Blacks (SRBs) to continuously monitor the precision of those carbon black test methods for which standard values have been established. SRBs are used primarily for two reasons: (1) to monitor testing performance to ensure that no systematic error or bias is affecting the test results, or (2) to establish a statistical calibration when the correction of assignable causes does not yield in-control test results.

Currently there are five primary reference materials available. They are: IRB 7 (Industrial Reference Black – used to determine mix properties), ITRB (Industrial Tint Reference Black – used to calibrate Tint testing), SRB HT Iodine Standard (Heat Treated) Adsorption Testing, SRB 8 (Standard Reference Black – Series 8 – used in many of the tests for determining colloidal properties), and Sulfur Blacks (used in calibrating for % sulfur in carbon black). Using these reference materials will assist each lab to continually guarantee their lab's measurement over time. Other reference materials may be used once they have been properly qualified as a reference.

Laboratory Proficiency Program to Guarantee Labs Maintain their Testing Proficiency[2]

After specifications, methods and reference materials have been established the final key to aligning carbon black quality between carbon black manufactures and customers is the establishment of a laboratory proficiency program. This would be a program that continuously monitors the ability of each participating lab to maintain their testing proficiency. The breath and frequency of the program would be defined between the manufacturer, customers, instrument manufacturers and any other interested participants. An item of concern is how to organize and manage such a program so as to make it as inclusive as possible and relevant to a particular carbon black sector (inks, paints, mechanical rubber goods, tires, etc.), geographic zone (Asia, Europe, North America, etc.) or a defined region (Africa, China, European Union, Japan, North America, South America, Southeast Asia, etc.).

Many carbon black producers and large consumers having multiple laboratories have had in-house programs to measure testing proficiency within their organizations. In some cases these programs encompassed both manufacturer and a few customers because most customers did not have the resources to conduct a program on their own. Regardless of how well-run these in-house programs may have been, they still reached only a limited number of laboratories and provided little, if any, insight on how any one program compared to all the similar programs.

Primarily at issue was the availability of a homogeneous reference material, distribution of the reference samples, a sufficient number of participating laboratories, timing when a laboratory may have resources available to conduct the testing, and costs to manage and process the round robin data. The time and effort to organize and participate in an interlaboratory testing program (ITP), commonly referred to as a "round robin," discouraged many potential participants. Most saw the ITP as a lot of work with little tangible benefit to the participants. Usually, few laboratories would agree to participate in an IPT.

ASTM International's Laboratory Proficiency Rating System (LPRS)

Globally speaking there is only one comprehensive program currently in existence today. This program lies within ASTM International's technical committee D24 on Carbon Black. The members of D24 (producers, users, and instrument manufacturers) faced similar challenges in maintaining testing proficiency in their laboratories and knowing how well their laboratories perform compared to others in conducting those tests. D24's program is conducted twice a year and covers primarily carbon blacks used in the production of tires. There is a small annual fee of $150 paid to cover the expense of collecting the data and generating the reports.

Once objectives of the LPRS have been defined and the carbon black selected, samples are sent from a central distributor (Laboratory Standards and Technologies, LLC) to each of the participating laboratories. After testing has been completed all results are returned to John Bailey, chairman of subcommittee 24.61 on Sampling and Statistics.

A report is sent to each laboratory which consists of several tables and graphs. Two tables summarize the statistical data. There is also a set of graphs provided which show the performance of all laboratories for each test with each laboratory being identified only by its code (individual labs are identified only by a code to protect the confidentiality of the labs and is not shared amongst the labs). Also each laboratory receives graphs showing their performance for all tests.

The following information is provided in each report:

h-factor graph (Graph 1): measures how well a given laboratory's mean value matches the mean value of all the laboratories reporting data for any particular test.

h(crit)-factor: is the value used for selecting the outlier laboratories based on Student's t distribution and is a function of the number of laboratories reporting data and the confidence level (95%).

k-factor (Graph 2): measures the variability within a laboratory compared to the pooled variability for all laboratories.

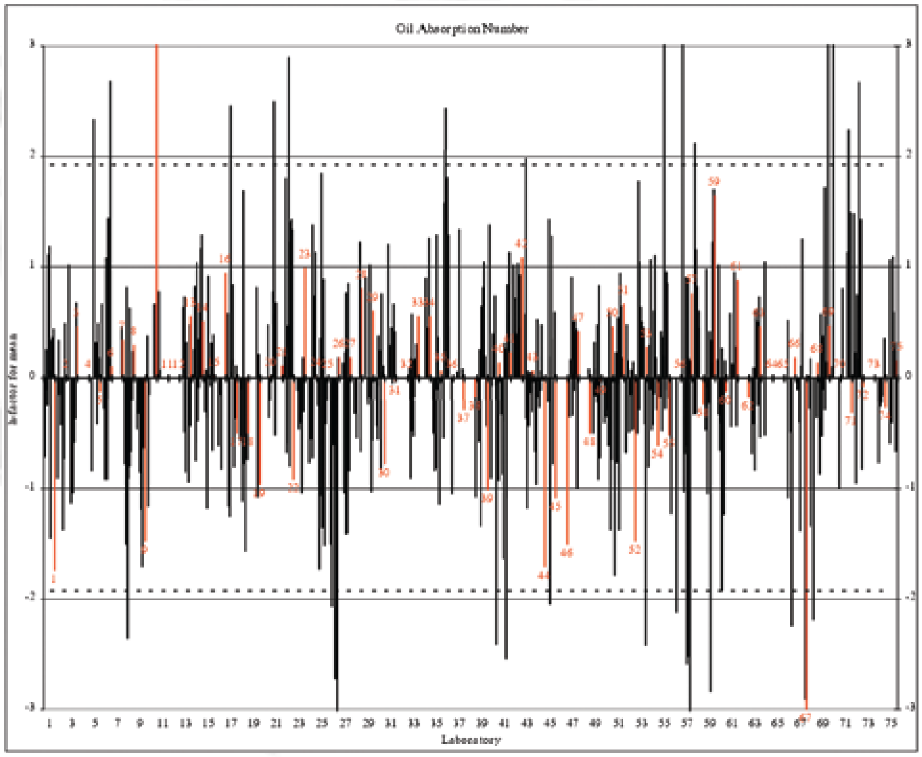
k(crit)-factor: is the value used for selecting the outlier laboratories based on the F-ratio and is a function of the number of laboratories reporting data and the confidence level (95%).

Variability Summary Table (Table 1): lists each test, measurement units, precision type (absolute or relative), and currently published repeatability [r or (r)] and reproducibility [R or (R)] are shown.

Statistics Summary Table (Table 2): lists each test, rows showing the nominal test period (month and year), mean value, standard deviation (Std. Dev.), and pooled standard deviation (Pool SD).

The LPRS results have been used to quantify each lab's testing proficiency for each test completed in the program. The data has also been used to: prepare new precision and bias statements for 12 base test methods, validate the uniformity of certain SRB and IRB materials, publish new target and acceptable range values

Graph 1. h-factor Graph of OAN



Graph 2. k-factor Graph of NSA

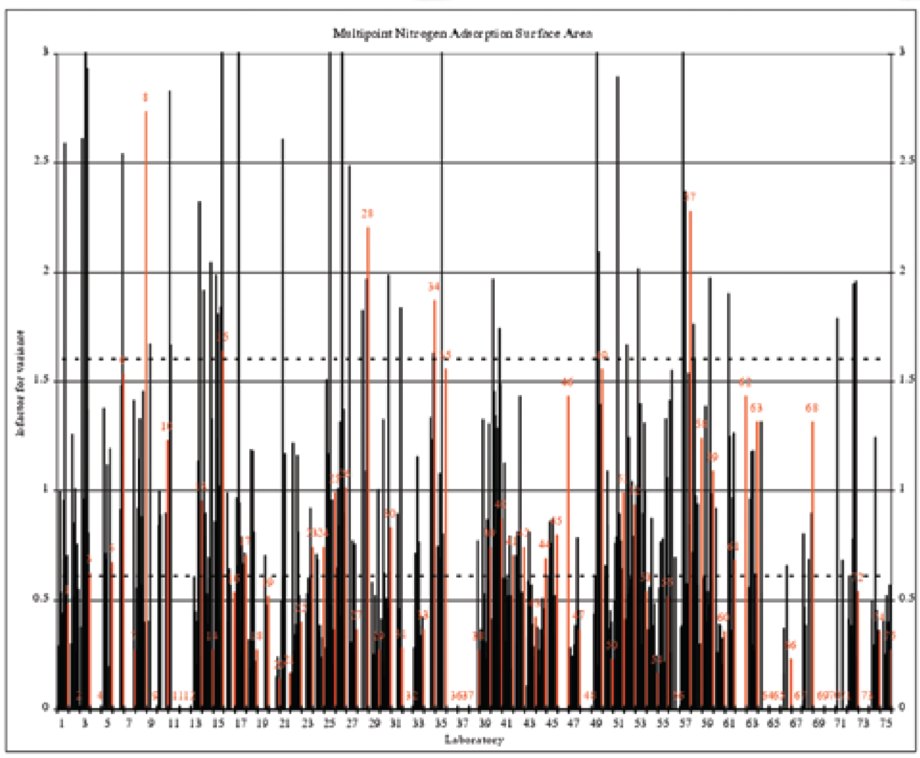


Table 1. Variability Summary of OAN and NSA

r'T = Within technician repeatability, r' = Within laboratory repeatability, R' = Between laboratories reproducibility

Material	SRB F5A	N339	N774	SRB C6	HS Tread	LS Carcass	SRB A6	N772	N299	SRB D7	N234	SRB8A	
OAN	Units:	cm3/100 g	Precision: Absolute		r:	1.51	R:	3.55					
	Sep 01	Mar 02	Sep 02	Mar 03	Sep 03	Mar 04	Sep 04	Mar 05	Mar 06	Sep 06	Sep 07	Mar '08	Average
Mean	130.10	119.02	72.20	70.90	175.79	35.39	124.14	63.30	123.15	38.34	124.70	70.96	95.67
r'T	1.71	0.98	1.08	0.97	2.01	0.93	1.19	1.12	1.08	0.91	1.47	1.12	1.26
r'	1.92	1.08	1.09	1.01	2.20	1.18	1.47	1.12	1.24	0.96	1.55	1.31	1.39
R'	5.67	2.43	7.50	3.23	7.07	4.24	2.99	4.94	2.74	4.58	3.06	3.23	4.60
NSA	Units:	m2/g		Precision: Relative		(r):	1.92	(R):	4.38				
	Sep 01	Mar 02	Sep 02	Mar 03	Sep 03	Mar 04	Sep 04	Mar 05	Mar 06	Sep 06	Sep 07	Mar '08	Average
Mean	38.17	91.75	30.20	78.64	126.54	20.60	143.94	29.53	101.36	20.97	118.43	76.49	73.05
r'T, %	2.53	1.38	2.39	1.92	0.91	2.61	1.38	2.23	1.33	3.20	0.92	0.97	1.55
r', %	2.53	1.45	2.91	2.08	1.11	4.41	1.56	2.38	1.78	3.25	1.25	1.31	1.83
R', %	4.85	3.45	9.11	3.29	4.21	11.81	2.85	10.59	3.16	7.51	2.90	3.14	4.31

Table 2. Statistical Summary of OAN and NSA

OAN	SRB F5A	N339	N774	SRB C6	HS Tread	LS Carcass	SRB A6	N772	N299	SRB D7	N234	SRB8A
Period	Sep 01	Mar 02	Sep 02	Mar 03	Sep 03	Mar 04	Sep 04	Mar 05	Mar 06	Sep 06	Sep 07	Mar '08
Mean	130.10	119.02	72.20	70.90	175.79	35.39	124.14	63.30	123.15	38.34	124.70	70.96
Std. Dev.	1.92	0.79	2.63	1.10	2.41	1.45	0.96	1.71	0.89	1.59	0.97	1.07
Pool SD	0.68	0.38	0.39	0.36	0.78	0.42	0.52	0.40	0.44	0.34	0.55	0.46
NSA	SRB F5A	N339	N774	SRB C6	HS Tread	LS Carcass	SRB A6	N772	N299	SRB D7	N234	SRB8A
Period	Sep 01	Mar 02	Sep 02	Mar 03	Sep 03	Mar 04	Sep 04	Mar 05	Mar 06	Sep 06	Sep 07	Mar '08
Mean	38.17	91.75	30.20	78.64	126.54	20.60	143.94	29.53	101.36	20.97	118.43	76.49
Std. Dev.	0.58	1.04	0.93	0.76	1.83	0.81	1.28	1.08	0.99	0.52	1.13	0.79
Pool SD	0.34	0.47	0.31	0.58	0.50	0.32	0.79	0.25	0.64	0.24	0.52	0.35

for the SRBs, document the equivalency of results for paraffin oil and n-dibutyl phthalate use in D 2414, and to investigate SRB candidate materials.

To date, 75 laboratories from 23 countries, 21 of which are not in North America, have participated in ASTM International's LPRS. Every continent and region of the world is represented, making this a truly international program. In some cases the precision and bias data coming from LPRS shows wider limits than were previously published. Due to the much larger number of laboratories participating and the international representation of those laboratories, it is generally felt that the precision and bias numbers more accurately reflect the state of testing proficiency in the carbon black industry.

The establishment of an LPRS program between carbon black manufacturers, their customers, instrument manufactures, and other organizations can improve a laboratory's testing proficiency and thus alignment between laboratories. The greatest challenge is establishing, organizing and managing a LPRS program. The benefits are great for the industry and address the need for guaranteeing the continual alignment of laboratories between companies thus a continual and successful business relationship.

Conclusion

It is important when conducting business between a manufacturer (supplier) and their customers that the level of quality be well defined before the start of any business transactions. Quality is defined by having well defined specifications, measured using standardized methods of analysis, guaranteed using accepted reference materials to assure measurement compliance and a robust LPRS program to assure lab alignment is maintained.

FOOTNOTES

1. Many of the comments in this section are direct quotes from ASTM International D4821-06, "Standard Guide for Carbon Black – Validation of Test Method Precision and Bias", 2008.

2. Many of the comments in this section are direct quotes from: John Bailey and Jeffery Melsom, "Laboratory Proficiency Rating System for Carbon Black," ASTM Standardization News, Vol. 33, No. 5, p. 46, October 2006.

Ricky Magee receives ASTM International Award of Merit

Ricky Magee, Columbian Chemicals Co., has been recognized by his peers and ASTM International by receiving ASTM's highest award, the Award of Merit. This award recognizes his exceptional contributions to Committee D24 on Carbon Black in the development of new analytical standards, for improvement of existing standards, and for strong leadership in developing international standards for the global market.

Ricky received his B.S. degree in Chemistry in 1982 and his M.S. degree in Chemistry in 1984 from Northeast Louisiana University. After a short stay with Morton-Thiokol, Ricky joined Columbian Chemical Co. (CCC) as an analytical chemist in their Research and Development center in Swartz, LA. In 1988 he became the manager of Columbia's Colloidal and Oil laboratories. In 2000 Ricky was named Director of Analytical Services for CCC and responsible for supporting Technical Services, R&D efforts and quality assurance activities.

Ricky has been actively involved in all facets of Technical Committee D24 on Carbon Black since July 1990. He initially became involved in improving D1510 Iodine Adsorption Number by studying the preparation and constituents of the iodine solution, stability of the iodine solution, effect of shake time on the repeatability and reproducibility.

Additionally, Ricky's work led to the development of a new standard measurement for the external surface area (D6556 Total and External Surface Area by Nitrogen Adsorption). This new method which incorporated the NSA procedure, replaced another ASTM method (D3765 CTAB (Cetyltrimethylammonium Bromide) Surface Area)) as the standard means to determine the external surface area of carbon blacks. As an additional benefit the classification system was

updated based on NSA. This improved system was then incorporated into D1765 Standard Classification System for Carbon Blacks used in Rubber Products.

Ricky is currently working on the development of a quick and easy method to determine structure of carbon black. He is instrumental in advancing the dynamic void volume method which could replace the traditional oil absorption method (D2414). This improved technology would allow structure determinations to be completed in minutes with little cleanup afterward. As work continues in this area Ricky has taken the lead to assure the continued development and implementation of this new technology. Already a vendor has produced a prototype instrument based on the work and recommendations by Ricky. The instrument is now available commercially and is beginning to be adopted in the carbon black field.

There isn't a standard within ASTM's Technical Committee D24 on Carbon Black that Ricky hasn't been involved. Aside from those already mentioned he was instrumental in the development of D6602 Sampling and Testing of Possible Carbon Black Fugitive Emissions and Other Environmental Particulate, or Both; D3265 Tint Strength; D1508 Pellet Fines and Attrition; D4122 Evaluation of Industrial Reference Black; and etc.

ASTM's Carbon Black standards are globally accepted as the reference for international use. Also D24's work in developing Standard Reference Blacks (SRBs), which are accepted and used internationally, establishes ASTM as the leader in the carbon black arena (rubber grades). The success of ASTM in this area is due to the dedicated contributions of individuals like Ricky Magee. No individual has contributed so widely across all of D24's standards than Ricky. It is through his hard work and perseverance that ASTM is able to see continuous improvement and development of new methods for the carbon black industry.

D24 Meeting Schedule

December 8-10, 2008
Miami Beach, FL

June 15-17, 2009
Vancouver, B.C., CA

October 5-7, 2009
Prague, CZ

June 7-9, 2010
St. Louis, MO

All who have an interest in carbon black are invited to attend.

Fully Automated Method for Carbon Black – Iodine Adsorption Number

(By George A. Joyce, Columbian Chemicals Research & Development)

The D24 carbon black technical committee recently approved a major revision to the Standard Test Method for Carbon Black – Iodine Adsorption Number. This revision to the standard, ASTM D1510-08c, was approved at the June 2008 meeting in Denver, CO. The standard was revised with a new method of analysis which utilizes an automated sample processor and titrator system available from Metrohm USA. This automated method has been utilized for a number of years in the carbon black industry. Columbian recently demonstrated the automated method by providing supporting data used to gain acceptance for inclusion in D1510.

The standard method of analysis for Carbon Black – Iodine Adsorption Number utilizes manual sample preparation followed by either manual or automated titration. This well-known method is now referred to as Method A. Manual sample preparation or processing starts by introducing specific amounts of both carbon black and 0.0473N iodine solution into a bottle. The bottle is capped and shaken for a one minute period typically using a mechanical shaker. This processing step results in an equilibrium adsorption of iodine on the surface of most carbon blacks. The next processing step is to centrifuge the carbon black – iodine slurry in order to obtain an aliquot of the iodine solution for analysis. The excess aqueous iodine, a mixture of elemental iodine, potassium iodide, and water is back-titrated with 0.0394N aqueous sodium thiosulfate. The following simplified equation is representative of the iodine reduction using sodium thiosulfate.

$$2\,S_2O_3^{2-}(aq) + I_2(aq) \rightarrow S_4O_6^{2-}(aq) + 2\,I^-(aq)$$

The reduction of iodine to iodide using the thiosulfate anion can be performed manually using a glass buret and a starch indicator. However, many laboratories today automate the analysis step with a potentiometric titrator and platinum-ring electrode to reach an equivalence end-point. Both methods of titration can yield excellent results. Depending on the number of incoming samples, Method A can consume 100 percent of an analyst's time in the laboratory.

The automated system, Method B, couples automatic sample processing with automatic titration providing a fast, precise method which frees the analyst to perform other tasks once the sample changer is loaded. The system is shown in Figure 1. The system was designed to use the same solutions as Method A, and also the same ratios of iodine to sample mass.

Basic components of the automated iodine adsorption system include:

- Windows PC running Tiamo software
- Analytical Balance with PC interface
- USB Robotic Sample Processor with 12-station sample changer
- Titrando Potentiometric Titrator
- 50 mL Dosino Dosing Pumps
- Reaction Vessel
- Pumps for automatic rinsing of reaction vessel

The Metrohm system components are off-the-shelf products used in numerous applications of chemistry. The Tiamo software contains detailed method settings and sequences for each of the system components along with a user-interface for testing samples and a database for storage and retrieval of results. The software facilitates automation scripts used to provide optional automatic normalization of data with the SRB HT Iodine standards.

The first step to performing an analysis with the Metrohm system is to select a new analysis in the Tiamo software, place an empty 100 mL Pyrex beaker onto the analytical balance, tare the beaker, then add the sample into the beaker using the sample mass prescribed in D1510-08c section 11.3.2. The 12-station sample changer is shown in Figure 2.

Sample processing is initiated with a mouse click and the next sample can be weighed while the previous sample is dosed with iodine and stirred. The next step is to allow settling of the carbon black. At this time the reaction vessel is cleaned, and numerous steps are taken to carefully purge all the pumps and lines prior to the analysis. Next an aliquot of the first sample is pumped through a disposable filter then into the reaction vessel for

Figure 1. Metrohm Iodine System

Figure 2. 12-Station Sample Changer

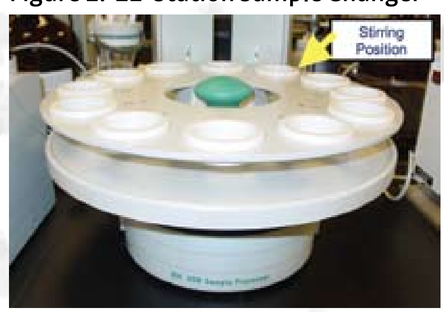


Figure 3. Reaction Vessel

analysis. A picture of the reaction vessel is shown in Figure 3.

Once a potentiometric titration is completed the waste solutions are automatically removed and the vessel cleaned for the next titration. This process can be repeated for a total of 12 sample analyses unattended.

Since Method B utilizes a different sample preparation process than Method A, slight differences in the measured results are possible, but most information presented to the D24 technical committee has indicated both methods provide equivalent results. The Metrohm software has the capability of performing automatic SRB HT normalization of measurements ensuring the results are always consistent with Method A. Method A and B data presented to the D24 committee is shown in Table 1.

Although the estimation of carbon black surface area from iodine adsorption has known weaknesses, the D1510 method remains important for specifications in the manufacture of carbon black products primarily due to very rapid analysis time, good precision and low cost. Like most other laboratory innovations, this fully automated iodine system is currently only available from one source – Metrohm. However, competition from other instrument manufacturers may result in availability of similar systems in the future.

Table 1. Measured Values from Methods A an B, Iodine Adsorption Number, D1510

IODINE NUMBER, mg/g							
Product		ID	Mass, g	Method B	Method A	Δ	% Diff
1	N326	A-8	1	79.7	79.4	0.3	0.4
2	N700 LS	D-8	1	20.8	20.9	-0.1	-0.6
3	N660	E-8	1	35.8	35.2	0.6	1.6
4	N683	F-8	1	35.4	35.7	-0.2	-0.6
5	N550	A-48411	1	39.4	39.3	0.1	0.2
6	N650	A-49503	1	35.6	35.6	0.0	0.0
7	N234	A-50143	1	117.2	117.7	-0.5	-0.4
8	N772	A-50142	1	25.2	25.6	-0.4	-1.5
9	N330	A-50145	1	82.3	82.0	0.3	0.3
10	N121	A-49769	1	119.7	119.2	0.5	0.4
11	R1000	A-11215	1	95.8	94.9	0.9	0.9
12	N134	B-8	0.5	131.4	133.2	-1.8	-1.3
13	N100 HS	C-8	0.5	139.0	138.7	0.3	0.2
14	N115	A-30584	0.5	155.2	154.3	0.9	0.6
15	CD-Product	A-44046	0.5	187.2	187.1	0.1	0.0
16	R1500	C71754	0.5	180.3	179.2	1.1	0.6
17	R2000	C71752	0.5	191.5	190.1	1.4	0.7
18	R3500	C71757	0.25	301.7	303.3	-1.5	-0.5
19	R7000	C71889	0.25	448.8	451.8	-3.0	-0.7
20	CD-Product	A-44047	0.125	570.8	573.9	-3.1	-0.5
		AVERAGE		149.6	149.9	-0.2	0.0

D24 Activities

D24.11 Carbon Black Structure
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D2414
Oil Absorption Number

Hitec (P. Hirtt) presented customer OAN data showing many laboratories were observing that SRB E-8 was consistently measuring higher than the published target value. Columbian Chemicals (G. Joyce) also had a lab that showed the same issue. A task group was formed to review the SRB E-8 OAN issue. Columbian Chemicals will analyze SRB 7 series and 8 series using a Dynamic Void Volume Analyzer (DVVA) OAN prediction model and compare the predicted values to target values.

D6086
Void Volume (VV)

Columbian Chemicals (G.Joyce) presented an update on their implementation of two Micromeritics DVVAs located in two of their production facilities. Initial data collection and subsequent modeling indicated models within the production facilities exhibit a low standard error less than or equal to the repeatability of the OAN test. Also the new modeling features incorporated by Micromeritics allow the user to input specific prediction models. Discussions of DVVA modeling and OAN problems suggests that OAN testing has led to product variation from producer-to-producer or plant-to-plant since OAN is not an intrinsic characteristic of the carbon black. A much more detailed paper will be presented at the ACS Rubber Division's Mini Expo in Louisville, KY and will then be presented at the next ASTM meeting in December in Miami, FL.

Columbian Chemicals will release the data after the ACS meeting in Louisville.

Hitec (P. Hirtt) presented initial compressed void volume data based on an experimental instrument with a 1 inch diameter cylinder, 2 gram sample, and a 170 kN compressive force. Hitec has evaluated both compression and decompression data of the SRB series and indicated a universal COAN model was possible from the decompression curve. They also confirmed some of Columbian's earlier research from 2005 which indicated the log pressure model and inverse variable root model provided good data fits for the compression curves.

D24.21 Carbon Black Surface Area and Related Properties
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